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SOLUBILITY DATA SERIES

Volume 63

METALS IN LIQUID ALKALI METALS
PART I: Be to Os

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CONTENTS

INTRODUCTION TO THE SOLUBILITY DATA SERIES

SOLUBILITY OF SOLIDS IN LIQUIDS

NATURE OF THE PROJECT

The Solubility Data project (SDP) has as its aim a comprehensive review of published data for solubilities of gases, liquids and solids in liquids or solids. Data of suitable precision are compiled for each publication on data sheets in a uniform format. The data for each system are evaluated and, where data from independent sources agree sufficiently, recommended values are proposed. The evaluation sheets, recommended values, and compiled data sheets are published on consecutive pages.

COMPILATIONS AND EVALUATIONS

The formats for the compilations and critical evaluations have been standardized for all volumes. A description of these formats follows.

Compilations

The format used for the compilations is, for the most part, self-explanatory. Normally, a compilation sheet is divided into boxes, with detailed contents described below.

Components: Each component is listed according to IUPAC name, formula, and Chemical Abstracts (CA) Registry Number. The Chemical Abstracts name is also included if this differs from the IUPAC name, as are trivial names if appropriate. IUPAC and common names are cross-referenced to Chemical Abstracts names in the System Index.

The formula is given either in terms of the IUPAC or Hill (1) system and the choice of formula is governed by what is usual for most current users: i.e., IUPAC for inorganic compounds, and Hill system for organic compounds. Components are ordered on a given compilation sheet according to:

(a) saturating components:
(b) non-saturating components;
(c) solvents.

In each of (a), (b) or (c), the components are arranged in order according to the IUPAC 18-column periodic table with two additional rows:

Columns 1 and 2:	H, alkali elements, ammonium, alkaline earth elements
Columns 3 to 12:	transition elements
Columns 13 to 17:	boron, carbon, nitrogen groups; chalcogenides, halogens
Column 18:	noble gases
Row 1:	Ce to Lu
Row 2:	Th to the end of the known elements, in order of atomic number.

The same order is followed in arranging the compilation sheets within a given volume.

Original Measurements: References are abbreviated in the forms given by Chemical Abstracts Service Source Index (CASSI). Names originally in other than Roman alphabets are given as transliterated by Chemical Abstracts. In the case of multiple entries (for example, translations) an asterisk indicates the publication used for compilation of the data.

Variables: Ranges of temperature, pressure, etc. are indicated here.

Prepared by: The names of all compilers are given here.

Experimental Values: Components are described as (1), (2), etc., as defined in the "Components" box. Data are reported in the units used in the original publication, with the exception that modern names for units and quantities are used; e.g., mass per cent for weight per cent; mol dm^{-3} for molar; etc. Usually, only one type of value (e.g., mass per cent) is found in the original paper, and the compiler has added the other type of value (e.g., mole per cent) from computer calculations based on 1989 atomic weights (2). Temperatures are expressed as t/°C, t/°F or T/K as in the original; if necessary, conversions to T/K are made, sometimes in the compilations and always in the critical evaluation. However, the author's units are expressed according to IUPAC recommendations (3) as far as possible.

Errors in calculations, fitting equations, etc. are noted, and where possible corrected. Material inserted by the compiler is identified by the word "compiler" or by the compiler's name in parentheses or in a footnote. In addition, compiler-calculated values of mole or mass fractions are included if the original data do not use these units. If densities are reported in the original paper, conversions from concentrations to mole fractions are included, but otherwise this is done in the evaluation, with the values and sources of the densities being quoted and referenced.

Details of smoothing equations (with limits) are included if they are present in the original publication and if the temperature or pressure ranges are wide enough to justify this procedure and if the compiler finds that the equations are consistent with the data.

The precision of the original data is preserved when derived quantities are calculated, if necessary by the inclusion of one additional significant figure. In some cases, compilers note that numerical data have been obtained from published graphs using digitizing techniques. In these cases, the precision of the data can be determined by the quality of the original graph and the limitations of the digitizing technique. In some cases graphs have been included, either to illustrate data more clearly, or if this is the only information in the original. Full grids are not usually inserted as it is not intended that users should read data from the graphs.

Method: The apparatus and procedure are mentioned briefly. Abbreviations used in Chemical Abstracts are often used here to save space, reference being made to sources of further detail if these are cited in the original paper.

Source and Purity of Materials: For each component, referred to as (1), (2), etc., the following information (in this order and in abbreviated form) is provided if available in the original paper: source and specified method of preparation; properties; degree of purity.

Estimated Error: If estimated errors were omitted by the original authors, and if relevant information is available, the compilers have attempted to estimate errors (identified by "compiler" or the compiler's name in parentheses or in a footnote) from the internal consistency of data and type of apparatus used. Methods used by the compilers for estimating and reporting errors are based on Ku and Eisenhart (4).

Comments and/or Additional Data: Many compilations include this section which provides short comments relevant to the general nature of the work or additional experimental and thermodynamic data which are judged by the compiler to be of value to the reader.

References: The format for these follows the format for the Original Measurements box, except that final page numbers are omitted. References (usually cited in the original paper) are given where relevant to interpretation of the compiled data, or where cross-reference can be made to other compilations.

Evaluations

The evaluator's task is to assess the reliability and quality of the data, to estimate errors where necessary, and to recommend "best" values. The evaluation takes the form of a summary in which all the data supplied by the compiler have been critically reviewed. There are only three boxes on a typical evaluation sheet, and these are described below.

Components: The format is the same as on the Compilation sheets.

Evaluator: The name and affiliation of the evaluator(s) and date up to which the literature was checked.

Critical Evaluation:

(a) Critical text. The evaluator checks that the compiled data are correct, assesses their reliability and quality, estimates errors where necessary, and recommends numerical values based on all the published data (including theses, patents and reports) for each given system. Thus, the evaluator reviews the merits or shortcomings of the various data. Only published data are considered. Documented rejection of some published data may occur at this stage, and the corresponding compilations may be removed.

The solubility of comparatively few systems is known with sufficient accuracy to enable a set of recommended values to be presented. Although many systems have been studied by at least two workers, the range of temperatures is often sufficiently different to make meaningful comparison impossible.

Occasionally, it is not clear why two groups of workers obtained very different but internally consistent sets of results at the same temperature, although both sets of results were obtained by reliable methods. In such cases, a definitive assessment may not be possible. In some cases, two or more sets of data have been classified as tentative even though the sets are mutually inconsistent.

(b) Fitting equations. If the use of a smoothing equation is justifiable the evaluator may provide an equation representing the solubility as a function of the variables reported on all the compilation sheets, stating the limits within which it should be used.

(c) Graphical summary. In addition to (b) above, graphical summaries are often given.

(d) Recommended values. Data are recommended if the results of at least two independent groups are available and they are in good agreement, and if the evaluator has no doubt as to the adequacy and reliability of the applied experimental and computational procedures. Data are reported as tentative if only one set of measurements is available, or if the evaluator considers some aspect of the computational or experimental method as mildly undesirable but estimates that it should cause only minor errors. Data are considered as doubtful if the evaluator considers some aspect of the computational or experimental method as undesirable but still considers the data to have some value where the order of magnitude of the solubility is needed. Data determined by an inadequate method or under ill-defined conditions are rejected. However, references to these data are included in the evaluation together with a comment by the evaluator as to the reason for their rejection.

(e) References. All pertinent references are given here, including all those publications appearing in the accompanying compilation sheets and those which, by virtue of their poor precision, have been rejected and not compiled.

(f) Units. While the original data may be reported in the units used by the investigators, the final recommended values are reported in SI units (3) when the data can be accurately converted.

QUANTITIES AND UNITS USED IN COMPILATION AND EVALUATION OF SOLUBILITY DATA

Mixtures, Solutions and Solubilities

A *mixture* (5) describes a gaseous, liquid or solid phase containing more than one substance, where the substances are all treated in the same way.

A *solution* (5) describes a liquid or solid phase containing more than one substance, when for convenience one of the substances, which is called the *solvent*, and may itself be a mixture, is treated differently than the other substances, which are called *solutes*. If the sum of the mole fractions of the solutes is small compared to unity, the solution is called a *dilute solution*.

The *solubility* of a solute 1 (solid, liquid or gas) is the analytical composition of a saturated solution, expressed in terms of the proportion of the designated solute in a designated solvent (6).

"Saturated" implies equilibrium with respect to the processes of dissolution and precipitation; the equilibrium may be stable or metastable. The solubility of a substance in metastable equilibrium is usually greater than that of the same substance in stable equilibrium. (Strictly speaking, it is the activity of the substance in metastable equilibrium that is greater.) Care must be taken to distinguish true metastability from supersaturation, where equilibrium does not exist.

Either point of view, mixture or solution, may be taken in describing solubility. The two points of view find their expression in the reference states used for definition of activities, activity coefficients and osmotic coefficients.

Note that the composition of a saturated mixture (or solution) can be described in terms of any suitable set of thermodynamic components. Thus, the solubility of a salt hydrate in water is usually given as the relative proportions of anhydrous salt in solution, rather then the relative proportions of hydrated salt and water.

Physicochemical Quantities and Units

Solubilities of solids have been the subject of research for a long time, and have been expressed in a great many ways, as described below. In each case, specification of the temperature and either partial or total pressure of the saturating gaseous component is necessary. The nomenclature and units follow, where possible, ref. (3). A few quantities follow the ISO standards (7) or the German standard (8); see a review by Cvitaš (9) for details.

A note on nomenclature. The nomenclature of the IUPAC *Green Book* (3) calls the solute component B and the solvent component A. In compilations and evaluations, the first-named component (component 1) is the solute, and the second (component 2 for a two-component system) is the solvent. The reader should bear these distinctions in nomenclature in mind when comparing equations given here with those in the *Green Book*.

1. *Mole fraction* of substance 1, x_1 or $x(1)$ (condensed phases), y_1 (gases):

$$x_1 = n_1 \Big/ \sum_{s=1}^{c} n_s \qquad [1]$$

where n_s is the amount of substance s, and c is the number of distinct ubstances present (often the number of thermodynamic components in the system). *Mole per cent* of substance 1 is 100 x_1.

2. *Ionic mole fractions* of salt i, x_{i+}, x_{i-}:

For a mixture of s binary salts i, each of which ionizes completely into ν_{i+} cations and ν_{i-} anions, with $\nu_i = \nu_{i+} + \nu_{i-}$ and a mixture of p non-electrolytes k, of which some may be considered as solvent components, a generalization of the definition in (10) gives:

$$x_{+i} = \frac{\nu_{+i}x_{+i}}{1+\sum_{j=1}^{s}(\nu_j - 1)x_j}, \qquad x_{-i} = \frac{\nu_{-i}x_{+i}}{\nu_{+i}} \qquad i = 1....s \tag{2}$$

$$x_{ok} = \frac{x_j}{1+\sum_{j=1}^{s}(\nu_j - 1)x_j}, \qquad k = (s+1)...c \tag{3}$$

The sum of these mole fractions is unity, so that, with $c = s + p$,

$$\sum_{i=1}^{s}(x_{+i} + x_{-i}) + \sum_{i=s+1}^{c} x_{oi} = 1 \tag{4}$$

General conversions to other units in multicomponent systems are complicated. For a three-component system containing non-electrolyte 1, electrolyte 2 and solvent 3,

$$x_1 = \frac{\nu_{+2}x_{o1}}{\nu_{+2} - (\nu_2 - 1)x_{+2}} \qquad x_2 = \frac{x_{+2}}{\nu_{+2} - (\nu_2 - 1)x_{+2}} \tag{5}$$

These relations are used in solubility equations for salts, and for tabulation of salt effects on solubilities of gases.

3. *Mass fraction* of substance 1, w_1 or $w(1)$:

$$w_1 = g_1 \Big/ \sum_{s=1}^{c} g_s \tag{6}$$

where g_s is the mass of substance s. *Mass per cent* of substance 1 is 100 w_1. The equivalent terms *weight fraction*, *weight per cent* and *g (1)/100 g solution* are no longer used.

4. *Solute mole fraction* of substance 1, $x_{v,1}$:

$$x_{s,1} = m_1 \Big/ \sum_{s=1}^{c'} m_s = x_1 \Big/ \sum_{s=1}^{c'} x_s \tag{7}$$

where c' is the number of solutes in the mixture. These quantities are sometimes called Jänecke mole (mass) fractions (11, 12). *Solute mass fraction* of substance 1, $w_{s,1}$, is defined analogously.

5. *Solvent mole fraction* of substance 1, $x_{v,1}$:

$$x_{v,1} = x_1 \Big/ \sum_{s=1}^{p} x_s \tag{8}$$

Here, p is the number of solvent components in the mixture. *Solvent mass fraction* of substance 1, $w_{v,1}$, is defined analogously.

6. *Molality* of solute 1 in a solvent 2, m_1:

$$m_1 = n_1 / n_2 M_2 \qquad [9]$$

SI base units: mol kg^{-1}. Here, M_2 is the molar mass of the solvent.

7. *Aquamolality, Solvomolality* of substance 1 in a mixed solvent with components 2, 3 (13), $m_1^{(3)}$:

$$m_1^{(3)} = m_1 \overline{M} / M_3 \qquad [10]$$

SI base units: mol kg^{-1}. Here, the average molar mass of the solvent is

$$\overline{M} = x_{v,2} M_2 + (1 - x_{v,2}) M_3 \qquad [11]$$

and $x_{v,2}$ is the solvent mole fraction of component 2. This term is used most frequently in discussing comparative solubilities in water (component 2) and heavy water (component 3) and in their mixtures.

8. *Amount concentration* of solute 1 in a solution of volume V, c_1:

$$c_1 = \text{[formula of solute]} = n_1 / V \qquad [12]$$

SI base units: mol m^{-3}. The symbol c_1 is preferred to [formula of solute], but both are used. The old terms *molarity, molar* and *moles per unit volume* are no longer used.

9. *Mass concentration* of solute 1 in a solution of volume V, ρ_1:

$$\rho_1 = g_1 / V = c_1 M_1 / V \qquad [13]$$

SI base units: kg m^{-3}.

10. *Mole ratio*, $r_{A,B}$ (dimensionless) (9):

$$r_{n,12} = n_1 / n_2 \qquad [14]$$

Mass ratio, symbol $\zeta_{A,B}$, may be defined analogously (9).

11. *Ionic strength*, I_m (molality basis), or I_c (concentration basis):

$$I_m = \frac{1}{2} \sum_i m_i z_i^2 , \qquad I_c = \frac{1}{2} \sum_i c_i z_i^2 \qquad [15]$$

where z_i is the charge number of ion i. While these quantities are not used generally to express solubilities, they are used to express the compositions of non-saturating components. For a single salt i with ions of charge numbers z_+ and z_-,

$$I_m = |z_+ z_-| \nu m_i , \qquad I_c = |z_+ z_-| \nu c_i \qquad [16]$$

Mole and mass fractions and mole ratios are appropriate to either the mixture or the solution point of view. The other quantities are appropriate to the solution point of view only. Conversions between pairs of these quantities can be carried out using the equations given in Table 1 at the end of this Introduction. Other useful quantities will be defined in the prefaces to individual volumes or on specific data sheets.

Salt hydrates are generally not considered to be saturating components since most solubilities are expressed in terms of the anhydrous salt. The existence of hydrates or solvates is noted carefully in the critical evaluation.

Mineralogical names are also quoted, along with their CA Registry Numbers, again usually in the text and CA Registry Numbers (where available) are given usually in the critical evaluation.

In addition to the quantities defined above, the following are useful in conversions between concentrations and other quantities.

12. *Density*, ρ:

$$\rho = g/V = \sum_{s=1}^{c} \rho_s \qquad [17]$$

SI base units: kg m^{-3}. Here g is the total mass of the system.

13. *Relative density*, $d = \rho/\rho^\circ$: the ratio of the density of a mixture at temperature t, pressure p to the density of a reference substance at temperature t', pressure p'. For liquid solutions, the reference substance is often water at 4°C, 1 bar. (In some cases 1 atm is used instead of 1 bar.) The term *specific gravity* is no longer used.

Thermodynamics of Solubility

Thermodynamic analysis of solubility phenomena provides a rational basis for the construction of functions to represent solubility data, and thus aids in evaluation, and sometimes enables thermodynamic quantities to be extracted. Both these aims are often difficult to achieve because of a lack of experimental or theoretical activity coefficients. Where thermodynamic quantities can be found, they are not evaluated critically, since this task would involve examination of a large body of data that is not directly relevant to solubility. Where possible, procedures for evaluation are based on established thermodynamic methods. Specific procedures used in a particular volume will be described in the Preface to that volume.

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Table 1. Interconversions between Quantities Used as Measures of Solubilities
c-component Systems Containing *c*-1 Solutes *i* and Single Solvent *c*

	x_i	w_i	m_i	c_i
$x_i =$	x_i	$\dfrac{1}{1+\dfrac{M_i}{M_c}\left\{\dfrac{1}{w_i}-1+\sum_{j\neq 1}^{c-1}\left(\dfrac{M_c}{M_j}-1\right)\dfrac{w_j}{w_i}\right\}}$	$\dfrac{1}{1+\dfrac{1}{m_i M_i}\left(1+\sum_{j\neq i}^{c-1} m_j M_j\right)}$	$\dfrac{1}{1+\dfrac{1}{M_c}\left(\dfrac{\rho}{c_i}-M_i\right)+\sum_{j\neq i}^{c-1}\dfrac{c_j}{c_i}\left(1-\dfrac{M_j}{M_c}\right)}$
$w_i =$	$\dfrac{1}{1+\dfrac{M_c}{M_i}\left\{\dfrac{1}{x_i}-1+\sum_{j\neq i}^{c-1}\left(\dfrac{M_j}{M_c}-1\right)\dfrac{x_j}{x_i}\right\}}$	w_i	$\dfrac{1}{1+\dfrac{1}{m_i M_i}\left(1+\sum_{j\neq i}^{c-1} m_j M_j\right)}$	$\dfrac{c_i M_i}{\rho}$
$m_i =$	$\dfrac{1}{M_c\left(\dfrac{1}{x_i}-1-\sum_{j\neq i}^{c-1}\dfrac{x_j}{x_i}\right)}$	$\dfrac{1}{M_i\left(\dfrac{1}{w_i}-1-\sum_{j\neq i}^{c-1}\dfrac{w_j}{w_i}\right)}$	m_i	$\dfrac{1}{\dfrac{1}{c_i}\left(\rho-\sum_{j\neq i}^{c-1} c_j M_j\right)-M_i}$
$c_i =$	$\dfrac{\rho}{M_i+M_c\left\{\dfrac{1}{x_i}-1+\sum_{j\neq i}^{c-1}\left(\dfrac{M_j}{M_c}-1\right)\dfrac{x_j}{x_i}\right\}}$	$\dfrac{\rho w_i}{M_i}$	$\dfrac{\rho}{\dfrac{1}{m_i}\left(1+\sum_{j\neq i}^{c-1} m_j M_j\right)+M_j}$	c_i

ρ - density of solution; M_i - molar masses of *i*. For relations for 2-component systems, set summations to 0.

Preface

Liquid alkali metals are widely used as heat transfer media because of the large temperature range of their liquid state and their excellent heat transfer properties (1,2). Their application includes large heat transfer circuits of nuclear reactors, in which hundreds of tonnes of sodium are circulating, and small amounts of alkali metals in valves of high energy engines. Since alkali metals are among the most electropositive elements, they can be used in batteries with high cell voltage and large specific energy. Even in two-phase heat transfer systems such as heat pipes, thermionic or magneto-hydrodynamic devices, alkali metals may be used as working fluids at high temperature. As alkali metals reduce many oxides, nitrides or salts to form metals, the solubility of some reducible elements may be the basis of extractive metallurgical processes.

The compatibility of the liquid alkali metals with constructional materials which are used in energy conversion devices is dependent on the solubility of metallic elements in the molten metals (1-4). A simple correlation between the solubility of solid elements in the fluid and the rate of material loss due to corrosion is valid in certain dynamic systems. The knowledge of the solubilities of metals in the alkali metals enables prediction of the corrosion behaviour of alloys. Most of the metallic elements form intermetallic compounds with alkali metals and they are not suitable as alloying elements of structural materials. The dissolution of metals, on the other hand, influences physical properties of liquid alkali metals as solvents.

The corrosion of solid metals in liquid alkali metals is frequently influenced by chemical reactions in which a third element of non-metallic character is involved. Such reactions have to be considered if an apparent solubility of a solid element is to be assessed. At appreciable chemical activity oxygen, nitrogen or carbon, ternary oxides or nitrides and binary carbides are precipitated phases which are in equilibrium with liquid phases in molten metals in many cases. The experimental solubilities are significantly influenced by the concentrations of non-metals in the liquid metal and the solid as well. The non-metallic elements can act as complexing agents increasing the concentration of the solute in liquid metals to exceed the thermodynamic solubility though the solid metal is the phase in equilibrium with the solution. The high affinity of the alkali metals to form compounds with the constituents of the atmosphere is the reason for the necessity to strictly exclude the atmosphere (to the level of traces) from the experimental conditions. Data on the solubility of metallic elements in alkali metals are, therefore, much less valuable if they are not related to concentrations of the non-metals present in the solubility system. This is particularly important in the case of transition metals.

This volume contains the collection of evaluated experimental solubility data of about seventy metallic elements in five alkali metal solvents (Li, Na, K, Rb, Cs). The systems were ordered following the IUPAC long periodic table for the solutes and following the atomic number for the solvent elements. The mutual binary systems of the alkali metals themselves are treated briefly in the first pages of this volume. They are characterized by either complete miscibility in some of the systems or large miscibility gaps in others. We present them for general information in the form of assessed phase diagrams. The detailed elaboration of this subject, including all available experimental results, will be presented in a further volume of this series dealing with solubilities of non-metallic substances in the liquid alkali metals

Solubility data of metals in liquid alkali metals are widely spread over journals, congress proceedings, rare reports of several organisations in France, Germany, the UK and the US, and unique literature which has appeared only in the Russian language. The first publications concerned with binary systems of alkali metals with other metallic elements appeared at the end of the last century; the literature is covered inclusively up to 1994.

Chemical Abstracts, Nuclear Science Abstracts and Atom Index were used as sources for reference to solubility data. It was soon discovered that the key word *solubility* was not sufficient to extract complete solubility information from the corresponding literature. Therefore, entries related to this subject were also inspected. The completeness of our investigation of the abstracts was confirmed and extended by reading of several reviews dealing with the solubility of metals in liquid alkali metals (1,2,5-8). Since none of the mentioned reviews was sufficiently complete, we decided to collect and anew assess all experimental results available. If not otherwise stated, the DATA SHEETS were prepared directly from the original papers. Any secondary sources were used only sporadically if original reports were not accessible, and is always clearly indicated on the data sheets.

Solubility data are sometimes measured under constrained pressure, since the vapour pressure of the heavier alkali metals is high in comparison to that of the solute metals. The data which are gained under such conditions do not indicate an influence of moderately high pressure on the solubility of metals in liquid alkali metals. A relation between published experimental solubility data and the values which are predicted on the basis of theoretical models can be attempted. Such predicted data may be useful to distinguish between the true solubility of a metallic element and the apparent solubility due to the formation of ternary compounds which results from the equilibration of the metal and its solution.

Schematic phase diagrams are presented for systems in which they aid in understanding the data and the conclusions. They are based on the most recent state of knowledge (8), and are presented in the CRITICAL EVALUATIONS. Some solubility diagrams are presented in form of a log solubility versus reciprocal temperature function. These figures illustrate the somewhat large scatter of data for systems in which the formation of ternary compounds interferes with the dissolution of metals.

Compositions of equilibrium solid phases were not discussed in detail in this monograph in order to avoid a repetition of the discussions in (7,8). The composition of a solute was very seldom determined in such systems in which no intermetallic compounds are formed. If such estimations were performed, it was either mentioned in a DATA SHEET or CRITICAL EVALUATION of the system. If a soluble metal formed ternary compounds with the solvent and a contaminating non-metal as potential solute, this fact was always emphasised in a CRITICAL EVALUATION. One should realize that estimations of the stoichiometry and thermal stability of such a ternary compound are experimentally difficult and frequently uncertain. Thus, the presentation of corresponding ternary phase diagrams seems to be premature in such cases.

If a solute and a solvent form a binary intermetallic compound, its composition was frequently estimated from thermal analysis experiments of selected alloy compositions. In the case of metallic systems such estimation is not precise. We omitted a presentation of solidus data in the DATA SHEETS for technical reasons, since the temperature is treated as the independent variable and the solubility as the dependent one in the whole volume. The role of the variables is reversed in thermal analysis experiments. Thus, the reader should estimate solidus lines using the selected phase diagrams in the case that it might be necessary. We decided not to place experimental values in phase diagrams since the figures would lack clarity due to overlapping of symbols.

DATA SHEETS contain all liquidus data extracted from individual papers. However, the solubility values in the CRITICAL EVALUATIONS were generally selected by the evaluators for the sides rich in alkali metals. Further recommended liquidus data may either be read from the phase diagrams or by analysing the corresponding DATA SHEETS.

Quite frequently, important solubility data are only graphically reported. They are read out visually by the compilers. The precision of the procedure is indicated in the data sheets under heading "ESTIMATED ERROR". Evaluated solubility data are tabulated at the end of the CRITICAL EVALUATIONS: if there is agreement of at least two independent studies within the experimental error, the solubility values are assigned to the "recommended" category. Values are assigned as "tentative", if only one reliable result was reported, or if the mean value of two or more reliable studies was outside the error limits. In the tabulation, three, two, or one significant figures are assigned for respective precisions that are better than ± 1 and ± 10 % and worse than ± 10 %.

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Acknowledgements

The editors gratefully acknowledge the encouraging help of IUPAC Commission V.8 and its late chairman, Prof. A.S. Kertes, under whose authorization the work was initiated. Particularly, the advice and suggestions of Profs. M.-Th. Saugier-Cohen-Adad, J.W. Lorimer and Dr. M. Salomon were fruitful. Mr. J.R. Weeks brought the two editors together for the production of this volume.

Acknowledgement is also made to the University of Warsaw, Department of Chemistry, namely Prof. Z. Galus, and to the Forschungszentrum Karlsruhe for the permission to contribute to the *Solubility Data Series* and to provide the editors with library services and technical equipment for this work. The assistance of the librarians of the Nucleonic Library of Warsaw and the contributions of Miss A. Borgstedt and Mr. G. Frees to the production of camera ready pages is gratefully acknowledged.

H.U. Borgstedt, Karlsruhe, Germany
C. Guminski, Warsaw, Poland
January 1996

COMPONENTS:

All binary combinations
(1) Lithium; Li; [7439-93-2]
(2) Sodium; Na; [7440-23-5]
(3) Potassium; K; [7440-09-7]
(4) Rubidium; Rb; [7440-17-7]
(5) Cesium; Cs; [7440-46-2]

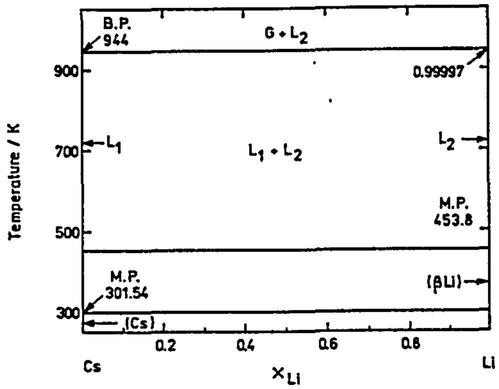
EVALUATOR:

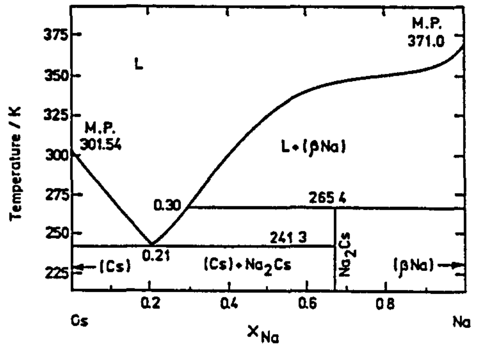
H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland

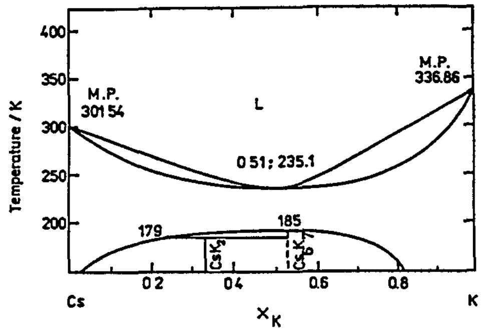
December 1992

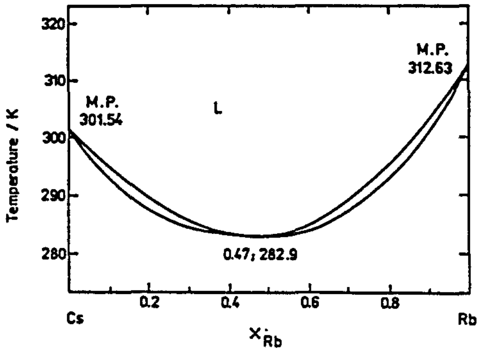
CRITICAL EVALUATION:

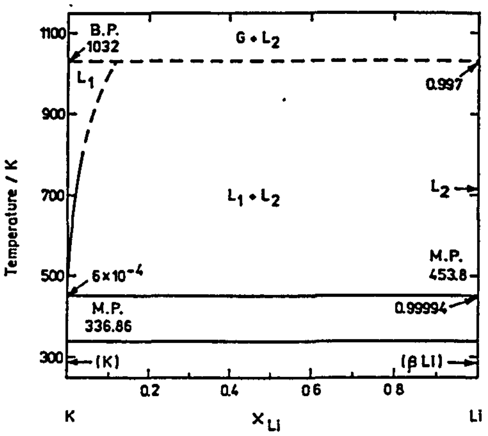
Since the formation of liquid alloys in all binary combinations of the alkali metals is characterized by either complete miscibility or extensive miscibility gaps, it was decided to present smoothed results in the form of figures. The literature is quite large and was successfully evaluated recently. The sources are: Cs-K (1,6); Cs-Li (2); Cs-Na (1); Cs-Rb (1); K-Li (3); K-Na (1); K-Rb (1); Li-Na (4); Li-Rb (5); Na-Rb (1).

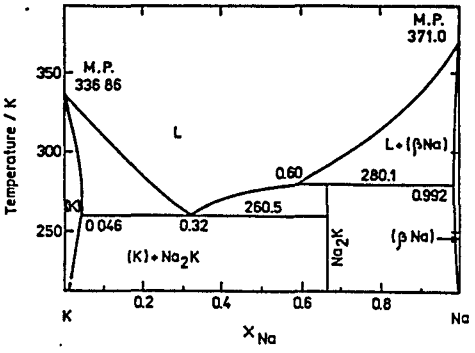






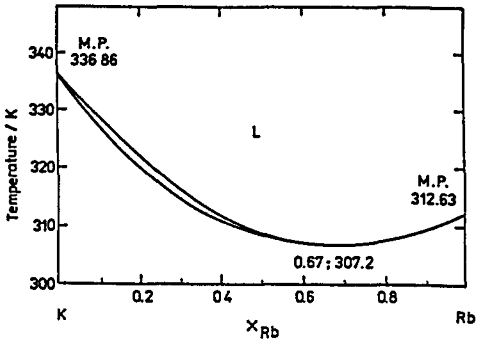


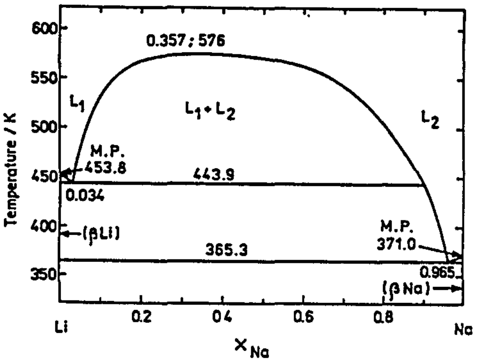


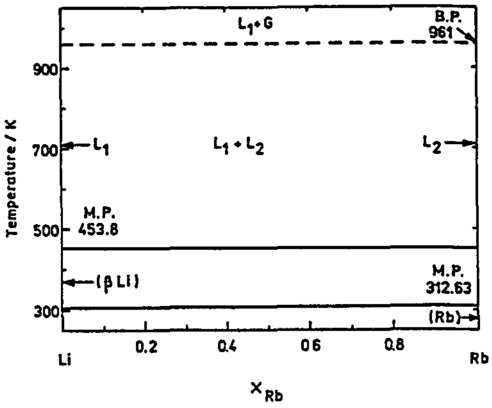


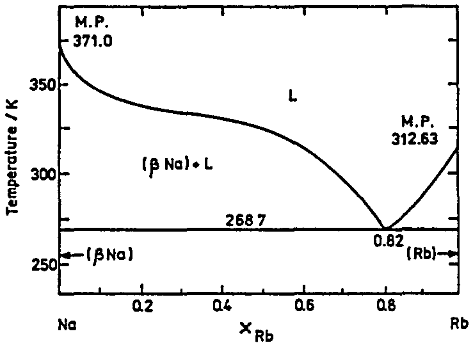
COMPONENTS:	EVALUATOR:
All binary combinations (1) Lithium; Li; [7439-93-2] (2) Sodium; Na; [7440-23-5] (3) Potassium; K; [7440-09-7] (4) Rubidium; Rb; [7440-17-7] (5) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland December 1992

CRITICAL EVALUATION:









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6. Drits, M.E.; Zusman, L.L. *Splavy Shchelochnykh i Shchelochnozemlenykh Metallov*, Metallurgia Moskva, 1986, p. 110.

COMPONENTS:	EVALUATOR:
(1) Beryllium; Be; [7440-41-7] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1988

CRITICAL EVALUATION:

The solubility of Be in liquid Li was determined by three groups of workers (1-4). Some qualitative observations were also reported by Wilkinson and Yaggee (5) who investigated the static corrosion of Be in Li at 873 K for six days; they observed a precipitation of low soluble BeNi (Ni as an impurity in the system), but no apparent dissolution of Be in Li. Van der Marel et al. (6) observed a dissolution of Be plates in liquid Li, if Li was contaminated by small amounts of Li_2O or LiOH at a temperature ~ 1123 K.

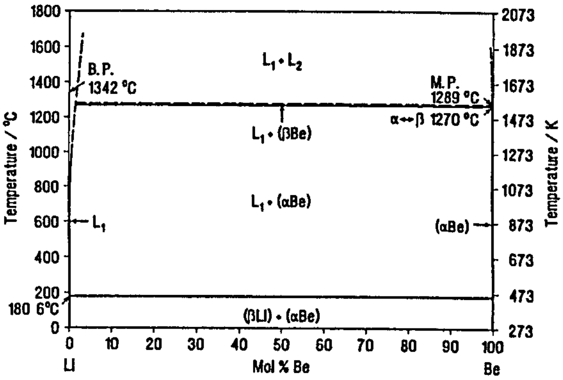
Jesseman et al. (1) determined the Be content in Li at 1005 and 1289 K as high as 0.23 and 1.1 mol% respectively. It seems that the most conclusive experiment was performed by Bychkov et al. (2,3) at 1273 K. They measured a solubility of 0.19 mol% Be and described all essential details of the determination. Klemm and Kunze (4) determined solubilities between 0.15 and 12.8 mol% Be at temperatures increasing from 573 to 1473 K. Most probably the significant weight loss of Be samples was rather due to the reaction of Be with the Fe container and not true dissolution of Be in Li. The dependence of log (*soly* of Be) vs. T^{-1} was not smooth. The possibility of such a side reaction was pointed out by Cunningham (7) and Hoffman (8) who also observed an enhanced dissolution of a Be sample in liquid Li which was encapsuled in a Fe container. The compound Be_2Fe was identified as a reaction product by means of X-ray diffraction. Hoffman (8) did not measure a dissolution of Be in liquid Li in a test for 100 h at 1089 K, if the container was made of Be. These data indicate a lower solubility of Be in liquid Li than reported in (1) and (4). Additional to the influence of the mass transfer of Be to the container material and subsequent reaction, the presence of O might also increase the apparent solubility. It is, however, difficult to evaluate the effect of such a side reaction; in (1) a content of 0.1 mol % O was mentioned.

Bechtold (9) estimated a solubility of Be in Li of $4 \cdot 10^{-5}$ and $6\ 10^{-5}$ mol% Be at 473 and 543 K on the basis of corrosion tests with electropolished Be sheets for 1360 hours. Other experimental details are not known. Migge (10,11) performed a thermodynamic analysis of the Be-Li system. He found that Be_3N_2 is the only stable compound in Li contaminated by C, N, or O. The solubility of Be_3N_2 was estimated to be $2 \cdot 10^{-4}$ and $2 \cdot 10^{-8}$ mol% at 1000 and 500 K respectively, much lower than the solubility of Be after (1-4) and (9).

We accept therefore the result of (2,3) as tentative, bearing in mind that it is almost two orders of magnitude lower than the corresponding value of (4) and one order in comparison to (1). The studies (4),(5), and (12) agree in the opinion that the saturated solution of Be is in equilibrium with almost pure Be since Be-Li intermetallics were not established in the system and solid solubility of Li in Be is not higher than 1 mol% (13). Two versions of the phase diagram were reported in (4), but neither of them can be recommended without clarification by means of further investigations of this system. It seems more likely that the liquid metals are immiscible as indicated by the phase diagram given in (14).

Tentative value of the solubility of Be in liquid Li:

T/K	*soly*/mol% Be	source
1273	0.2	(2,3)



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COMPONENTS:

(1) Beryllium; Be; [7440-41-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B.

Atom. Energiya 1959, *7*, 531-536; *Kernenergie* 1960, *3*, 763-7.

VARIABLES:

One temperature: 1273 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Be in liquid Li at 1000 °C was determined to be 0.25 mass % (0.19 mol % as calculated by the compilers). The same result is also reported in (1). Equilibration was not reached if the time of conditioning was shorter than 200 h.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The inner surface of a Be crucible was ground, electrolytically polished and etched. This crucible was gradually filled with freshly distilled Li dropping from a stainless steel condenser. The apparatus was filled with pure Ar after the distillation. The crucible filled with Li was placed inside a stainless steel container to which a cover was welded in an arc furnace. The whole apparatus was equilibrated at 1273 K for 200 h in an arc furnace. The Li solution was then cooled in less than 50 s to solidification. The content of Be was determined by a colorimetric analysis.

SOURCE AND PURITY OF MATERIALS:

Be: purified by distillation.
Li: distilled, final contents of 0.02-0.06 % Na, 0.015 % K, $(1-4)\cdot 10^{-4}$ % Fe, $\leq$ 0.002 % Mg; Si, Ni, and Cr were not detected.

ESTIMATED ERROR:

Solubility: nothing specified; precision not better than ± 10 % (by the compilers).
Temperature: nothing specified.

REFERENCES:

1. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B; *Metall. Metalloved. Chist.Met.* 1960, 2, 178-188; *Metallurgy and Metallography of Pure Metals*, Gordon and Breach, N.Y., 1962, p. 178-188.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Beryllium; Be; [7440-41-7] (2) Lithium; Li; [7439-93-2]	Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P. *US Atom.Ener.Comm. Rep. NEPA-1465*, 1950.
VARIABLES:	**PREPARED BY:**
Temperatures: 1005 - 1289 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Be in liquid Li was measured at selected temperatures.

T/°C	*soly*/mass % Be	*soly*/mol % Be [a]
732	0.26; 0.34	0.23 (mean value)
1016	1.4	1.1

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Strips of a Be sheet were placed in a pair of Armco Fe capsules. The capsules were loaded with Li in an Ar dry box, degassed and the Li melted by a pot furnace. After welding, the capsules were heated in a vacuum furnace (under a partial pressure of 2.6 10^{-6} bar) at temperatures indicated. The capsules were inserted in stainless steel plates within the furnace; the average temperature for each pair was estimated from the temperature gradient in the plate. The temperature was maintained for a period of 24 hours and the furnace was then air-cooled while still being held under low pressure. The capsules were weighed and opened. The solidified samples were leached out of the capsules with distilled water, and the Be remaining undissolved was removed with the capsule, dried, and weighed as the tare to determine the amount of Li solution (present) in the capsule. The leached material was filtered, and the residue was spectrographically analyzed for its Be content.

SOURCE AND PURITY OF MATERIALS:

Be: unspecified purity.
Li: initial impurities of 0.24 % O, < 0.02 % N, < 0.005 % Na, however, further contamination by N and O from dry box atmosphere..
H_2O: distilled.
Ar: unspecified purity.

ESTIMATED ERROR:

Solubility: precision ± 12 % (compilers).
Temperature: stability ± 20 K.

REFERENCES:

COMPONENTS:

(1) Beryllium; Be; [7440-41-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Klemm, W.; Kunze, D.

The Alkali Metals, The Chemical Society, London, 1967, p. 3-22.

VARIABLES:

Temperature: 573-1473 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Be in liquid Li was presented on figures; the numerical values are reported in (1).

t/°C	*soly*/mol % Be
300	< 0.15
500	0.31
700	0.44; 0.46
800	0.94
900	3.22
1000	7.52
1050	9.20
1100	10.38
1200	12.80

COMMENTS AND ADDITIONAL DATA:

Intermetallic phases were not observed in Be as well as in cooled Li.

The authors inserted remarks: "The values above 1050 °C were not certain due to the vaporization of Li and an attack on the Fe crucible. The solubility data supported the conclusion that restricted miscibility occurred in the liquid state. However, the presence of an eutectic type of the system could not be excluded because of the uncertainty of the results at the highest temperatures."

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A closed Armco Fe crucible, heated several hours under high vacuum, was used for the determination of the solubility. All operations were performed in an Ar atmosphere. A small piece of Be suspended in liquid Li was heated to the desired temperature. Having attained the equilibrium at times between 25 and 90 hours, Be was removed, and the weight loss was determined. The solubility was calculated on this basis. Precipitated Be was observed after the cooling of the solution and X-rays reflections of BeO were quite weak. A more detailed description of the procedure was given in (1).

SOURCE AND PURITY OF MATERIALS:

Be: no impurities detected by means of X-ray analysis, from Degussa (1).
Li: commercial product of the "highest degree of purity".
Ar: 99.9 % pure, from Linde; further "specially purified".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kunze, D. *PhD Thesis*, Univ. of Münster, Germany, 1964.

COMPONENTS:	EVALUATOR:
(1) Beryllium; Be; [7440-41-7] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1991

CRITICAL EVALUATION:

Earlier investigations of the Be-Na system were concerned with the corrosion of Be in liquid Na (1-3). Significant losses of Be were observed when the Na solvent was contaminated with O. After 1000 hours of contact at 973 K (2), however, no Be was detected in the Na solution if the content of O was $3.6 \cdot 10^{-3}$ mol %. The BeO formed is not adherent to metallic Be.

Hoffman (4) observed a fair resistance in liquid Na at 1089 K after 100 hours of equilibration due to its content of BeO. If Na is contaminated by N, the insoluble compound Be_3N_2 might be formed in the system (5).

Whitman (6) likewise reported a very low but not further specified solubility of Be in liquid Na. More recently, Aleksandrov and Dalakova (7) did not detect Be in liquid Na by spectral analysis after an equilibration of the metals for 1 h at 973-1023 K.

The impression of a negligible solubility was not confirmed in later quantitative determinations of Klemm and Kunze (8). The solubility was measured between 573 and 1123 K. The results seem to be of a too high order in comparison to other alkali metals. A Be-Fe compound might be formed on the Fe crucible surface, causing an additional loss of the Be sample. The results might be regarded as doubtful, although they fitted quite well the equation:

$$\log (soly/\text{mol }\%) = 1.374 - 1003\,(T/\text{K})^{-1} \qquad r = 0.997 \qquad \text{Eq.(1)}$$

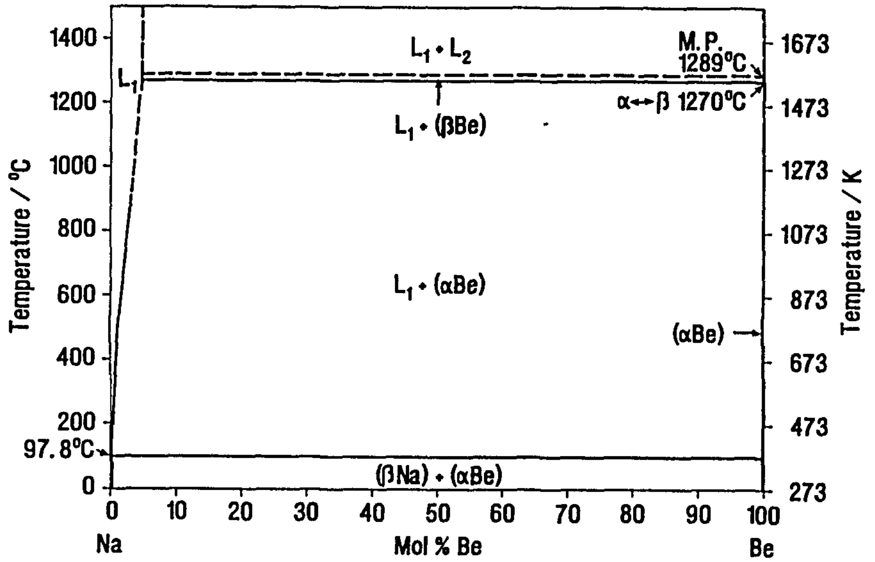
(r = correlation coefficient)

The slope of this equation is too low, if thermodynamic predictions are considered. It suggests that the apparent solubility might be related to other reactions in the solution.

The saturated solution is in equilibrium with almost pure Be (8), and the solid solubility of Na in Be is very low (6). The Be-Na system was critically evaluated by Pelton (9), who reported an assessed phase diagram at constant high pressure to keep Na in the liquid state.

Doubtful values of the solubility of Be in liquid Na

T/K	$soly$/mol % Be	source
573	0.5	(4)
673	0.8	(4)
773	1.3	(4)
873	1.9	(4)
973	2.2	Eq. (1)
1073	2.6	Eq. (1)



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4. Hoffman, E.E. *US Atom.Ener.Comm. Rep. ORNL-2924*, 1960.
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9. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1985, *6*, 33.

COMPONENTS:

(1) Beryllium; Be; [7440-41-7]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Klemm, W.; Kunze, D.

The Alkali Metals, The Chemical Society, London, 1967, p. 3-22.

VARIABLES:

Temperature: 473-1148 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Be in liquid Na at various temperatures was presented in a figure; the numerical data were reported in (1).

t/°C	*soly*/mol % Be
200	0.03
300	0.57; 0.49
400	0.78; 0.88
500	1.28; 1.26
600	1.95; 1.93
750	2.48
875	2.67

The saturated solution is in equilibrium with Be since intermetallic Be-Na phases have not been observed. According to the authors the question remains open if there exists a miscibility gap in the melt or a eutectic type of system. The liquid immiscibility is much more probable in the opinion of the evaluators.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Fe crucible was used for the determination of the solubility. It had been heated under high vacuum for several hours. All operations were performed in an Ar atmosphere. A small amount of Be was suspended in liquid Na and heated to the desired temperature. The solubility of Be in Na was probably determined by measurements of the weight loss of a Be sample after equilibration with liquid Na (1).

SOURCE AND PURITY OF MATERIALS:

Be: no impurities detected by means of X-ray analysis, from Degussa (1).
Na: analytical grade, from Merck, distilled under high vacuum.
Ar: 99.9 % pure, from Linde; further "specially purified".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kunze, D. *Ph.D. Thesis*, Univ. of Münster, Germany, 1964.

COMPONENTS:

(1) Beryllium; Be; [7440-41-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Klemm, W.; Kunze, D.

The Alkali Metals, The Chemical Society, London, 1967, p. 3-22.

VARIABLES:

One temperature: 873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Be in liquid K at 600 °C is 0.095 mol %; below 400 °C the solubility was lower than the detection limits. The value at 600 °C might be regarded as tentative, since it was determined by means of the same method and apparatus as the solubility of Be in Li, Na and Rb. The determination seems to be somewhat uncertain, as all these values of solubilities were considerably high. The saturation concentration of Be in K as reported here may show the same tendency.

COMMENTS AND ADDITIONAL DATA:

Aleksandrov and Dalakova (1) did not detect Be in liquid K by means of spectral analysis after equilibration of the metals for 1 h at 873 to 923 K.
The solid phase in equilibrium with the saturated solution seems to be almost pure Be, since no intermetallics were found by these authors, though an existence of Be_2K was previously reported in (2). The Be-K system was compiled by Pelton (3). The phase diagram is reported to be similar to that of the Be-Na system.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

For the determination of the solubility a Fe crucible was applied, which had been heated under high vacuum for several hours. All operations were performed in an Ar atmosphere. A small amount of Be was suspended in liquid K and heated to the desired temperature. The solubility of Be in K was probably determined by measurements of the weight loss of a Be sample after equilibration with liquid K.

SOURCE AND PURITY OF MATERIALS:

Be: no impurities detected by means of X-ray analysis, from Degussa (4).
K: "pure", from Merck, twice distilled under high vacuum (4).
Ar: 99.9 % pure, from Linde; further "specially purified" (4).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.
2. Jänecke, E. *Kurzgefaßtes Handbuch aller Legierungen*, Winter, Heidelberg, 1949.
3. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1985, *6*, 30.
4. Kunze, D. *Ph.D. Thesis*, Univ. of Münster, Germany, 1964.

COMPONENTS:

(1) Beryllium; Be; [7440-41-7]

(2) Rubidium; Rb; [7440-17-7]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
October 1986

CRITICAL EVALUATION:

Three different determinations of the solubility of Be in liquid Rb (1-3) have been performed. The results obtained were not in agreement, but scattered over more than one order of magnitude.

The determinations of Young and Arabian (2) are described in detail. However the solubilities observed at 811 and 1033 K were below the detection limit of $9 \cdot 10^{-3}$ mol % Be. The O content in Rb after determination was lower, thus indicating a reaction of Be with traces of O. The experiments performed by Simons et al. (1) were published in a brief study. Klemm and Kunze (3) presented a more detailed description of their work, yet it seems to be uncertain if Be interacts with a Fe crucible thus causing an increase of Be dissolution to 0.16 mol % at 873 K. The value of (1) is considered to be tentative.

The solid equilibrium phase is almost pure Be (3), the metals being probably immiscible in the liquid and solid states. The Be-Rb system was critically evaluated by Pelton (4). The phase diagram is reported to be similar to that of the Be-Na system.

Tentative value of the solubility of Be in liquid Rb

T/K	*soly*/mol % Be	source	remarks
1033	0.1	(1)	under constraining pressure

References

1. Simons, E.M. *Rep. NASA-TN-D-769*, 1961, p. 61.
2. Young, P.F.; Arabian, R.V. *US Atom.Ener.Comm. Rep. AGN-8063*, 1962; abstracted in *NASA Rep. SP-41*,Pt.I,1964, p. 167.
3. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p.3.
4. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1985, *6*, 33.

COMPONENTS:

(1) Beryllium; Be; [7440-41-7]

(2) Cesium; Cs; [7440-46-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
October 1986

CRITICAL EVALUATION:

The solubility of Be in liquid Cs was reported in (2) and (4). It seems to be difficult to obtain concordant results, as a slight decrease of the solubility of Be was observed in the temperature range of 323 to 573 K in (2). This suggests a chemical reaction related to the dissolution of Be in liquid Cs. In fact, a higher concentration of O caused an increase of the Be content in the liquid Cs phase. As it was observed in solubility tests with Be in other alkali metals, an interaction of Be with the Fe crucible may occur. Such a reaction may cause an increased apparent solubility of Be under the conditions of (4). The result of (4) obtained at 873 K is significantly higher than that of (2).

According to an evaluation test performed by Keddy (1), a severe attack of the anodized layer but no occurrence of metallic Be in Cs, was observed after testing anodized Be in liquid Cs for 21 hours at 703 K. This observation confirms the low solubility values determined by (2).

Pelton (3) critically evaluated the Be-Cs system. It seems that the metals are immiscible in the liquid state. The phase diagram should be similar to that of the Be-Na with a higher critical temperature of mixing in the liquid state. Elevated pressure has to be applied to keep Cs in the liquid phase.

Doubtful value of the solubility of Be in liquid Cs

T/K	*soly*/mol % Be	source
573	$1 \cdot 10^{-3}$	(2)

References

1. Keddy, E.S. *US Atom.Ener.Comm. Rep. LAMS-2948*, 1963.
2. Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.
3. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1985, *6*, 29.
4. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p.3.

COMPONENTS:

(1) Beryllium; Be; [7440-41-7]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Simons, E.M.

Rep. NASA-TN-D-769, 1961, p. 61-2.

VARIABLES:

One temperature: 1033 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Liquid Rb which had been in contact with Be at 1400 °F showed a pick up of $1 \cdot 10^{-2}$ mass % or $9 \cdot 10^{-2}$ mol % Be at 760 °C (as calculated by the compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiment was performed in a static sealed capsule made of Be and filled with Rb. After 500 hours of testing, the capsule was examined by means of metallographic techniques and the Be content in liquid Rb was spectrographically analyzed. Further details are likely to be inserted in (1), which was not available to the compilers.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Wagner, H.; Ludwigson, D.; Stang, J.H.; Simons, E.M. *Topic Rep. of Ai Research Manuf. Co from BMI*, 29 Oct., 1959.

COMPONENTS:

(1) Beryllium; Be; [7440-41-7]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Klemm, W.; Kunze, D.

The Alkali Metals, The Chemical Society, London, 1967, p.3-22.

VARIABLES:

One temperature: 873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Be in liquid Rb at 600 °C was determined to be 0.16 mol %. Below 400 °C the solubility was lower than the detection limits.
The equilibrium solid phase is Be, since intermetallic Be-Rb phases have not been found.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

For the determination of the solubility a Fe crucible was applied, which had been heated under high vacuum for several hours. All operations were performed in an Ar atmosphere. A small amount of Be was suspended in liquid Rb and heated to the desired temperature for 40 hours. The solubility of Be in Rb was probably determined by measurements of the weight loss of a Be sample after equilibration with liquid Rb.

SOURCE AND PURITY OF MATERIALS:

Be: no impurities detected by means of X-ray analysis, from Degussa (1).
Rb: obtained by reduction of RbCl with Ca; distilled under high vacuum.
Ar: 99.9 % pure, from Linde; further "specially purified".

ESTIMATED ERROR:

Nothing specified

REFERENCES:

1. Kunze, D. *Ph.D. Thesis*, Univ. of Münster, Germany, 1964.

COMPONENTS:

(1) Beryllium; Be; [7440-41-7]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Young, P.F.; Arabian, R.V.

US Atom.Ener.Comm. Rep. AGN-8063, 1962.

VARIABLES:

Temperature: 811 and 1033 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Be in liquid Rb at 1000 and 1400 °F was determined to be below $1\cdot10^{-4}$ mass % or $9\cdot10^{-3}$ mol % (as calculated by the compilers).

The results were at the detection limit of the method used for the determination. The solubility is understood as the Be content in the liquid phase. Therefore Be might be in the elemental form as well as in form of a compound with contaminating elements.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Be capsule, made of a hot pressed block, was cleaned with H_2SO_4, rinsed with H_2O, dried, filled with Rb and placed in a Ta furnace. The furnace was closed, evacuated and back flushed with Ar. During the repetition of the procedure, Ar was kept above atmospheric pressure. The temperature was kept for 50 hours after having reached the desired level. The furnace was inverted so that Rb with the dissolved Be could flow into a Ta sample cup and be cooled to room temperature. After solidification the sample was treated with anhydrous hexane, and further with methanol for Rb methylation. The methylate was decomposed with distilled H_2O and HCl. Having treated the sample cup with aqua regia for 1 hour in a water bath, the solution was added to the previous one. The residue was analyzed spectrographically by the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

Be: 98.36 % purity, from Beryllium Corp.; with contents of 1.63 % BeO, 0.144 % C, 0.165 % Fe, 0.088 % Al, 0.046 % Si, 0.064 % Mg, 0.04 % other impurities.
Rb: purified by passing through a micrometallic filter, gettered at 902 K, vacuum distilled, filtered into a storage tank; containing $(6-17)\cdot10^{-4}$ % O content after purification.

ESTIMATED ERROR:

Solubility: detection limit of the determination was $9\cdot10^{-3}$ mol % Be.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:

(1) Beryllium; Be; [7440-41-7]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Klemm, W.; Kunze, D.

The Alkali Metals, The Chemical Society, London, 1967, p.3-22.

VARIABLES:

One temperature: 873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Be in liquid Cs at 600 °C was determined to be 0.18 mol %. Below 400 °C the solubility was lower than the detection limits.
Solid Be is the phase in equilibrium with the saturated solution, since Be-Cs intermetallics were not detected.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

For the determination of the solubility a Fe crucible was applied, which had been heated under high vacuum for several hours. All operations were performed in an Ar atmosphere. A small piece of Be was suspended in liquid Cs and heated to the desired temperature. The solubility of Be in Cs was probably determined by measurements of the weight loss of a Be sample after equilibration with liquid Cs.

SOURCE AND PURITY OF MATERIALS:

Be: no impurities detected by means of X-ray analysis, from Degussa (1).
Cs: obtained by reduction of CsCl with Ca; distilled under high vacuum.
Ar: 99.9 % pure, from Linde; further "specially purified".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kunze, D. *Ph.D. Thesis*, Univ. of Münster, Germany, 1964.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Beryllium; Be; [7440-41-7] (2) Cesium; Cs; [7440-46-2]	Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177-80.
VARIABLES:	**PREPARED BY:**
Temperature: 323-573 K O concentration in Cs: < $8 \cdot 10^{-2}$ and 0.8 mol %	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Be in liquid Cs at various temperatures and O concentrations was reported.

t/°C	*soly*/mass % Be	*soly*/mol % Be[a]
50	$1.2 \cdot 10^{-4}$	$1.7 \cdot 10^{-3}$
150	$1.1 \cdot 10^{-4}$	$1.6 \cdot 10^{-3}$
300	$8 \cdot 10^{-5}$	$1.2 \cdot 10^{-3}$
300[b]	$7.1 \cdot 10^{-4}$	$1.0 \cdot 10^{-2}$

[a] - as calculated by the compilers
[b] - Cs contained 0.8 mol % O

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A specimen of Be metal was covered with Cs under vacuum and equilibrated for 120 hours in a glass ampoule. The O content in Cs was increased by a controlled decomposition of a $KClO_3$ - MnO_2 mixture. The ampoule glass did not undergo any visible changes. After the equilibration Cs was dissolved in H_2O and determined volumetrically. An aliquot of the solution was treated with an acid. The remaining part was acidified with HCl. Following the recipe in (1), EDTA, aluminon and an acetate buffer mixture were added. After further heating for 15 min at 358-363 K, the resulting solution was analyzed by colorimetry for its Be content.

SOURCE AND PURITY OF MATERIALS:

Be: containing ~0.03 % Si, 1 % Fe, <1 % Ti, 0.01 % Mg as main impurities.
Cs: 98-99 % purity, vacuum distilled, finally containing <0.01 % O and <1.5 % Rb as main impurities.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Godneva, M.M.; Vodyannikova, R.D. *Zh. Anal. Khim.* 1965, *20*, 831-6.

COMPONENTS:	EVALUATOR:
(1) Magnesium; Mg; [7439-95-4] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1991

CRITICAL EVALUATION:

According to the results of the preliminary experiments of Masing and Tammann (1) the liquidus curve gradually increases from the melting point of Li to that of Mg. Neither numerical nor graphical data were presented. The system was then examined independently by Henry and Cordiano (2), Grube et al. (3) and Saldau and Shamrai (4) by means of thermal analysis. The liquidus curves ofMg in these studies were in poor agreement. Similar solubility values of Mg up to 60 mol % are reported in (2) and (3). Henry and Cordiano (2) observed an inflection of the liquidus at about 60 mol % Mg, while other references (3,4) reported slight maxima at compositions corresponding to Li_2Mg_5 and $LiMg_2$, respectively, (3) and (4) eutectic compositions at 78.2 and 73 mol % Mg, respectively, were reported at approximately the same temperature of 861 K. Reference (5) presented the same results as (4) in graphical form.

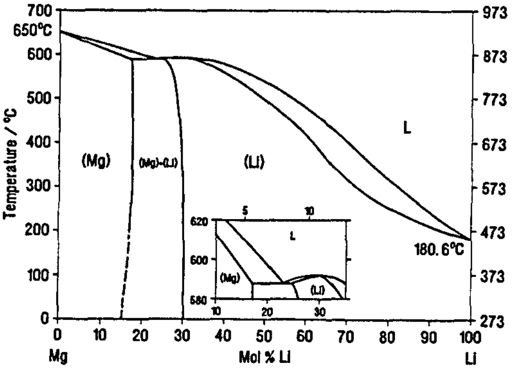
Bearing in mind the scatter of the results of preceding studies, Freeth and Raynor (6) intended to perform decisive experiments. The liquidus curve obtained in (6) was comparable to that of (3) with its maximum at 71 mol % Mg and 867 K. Solubility values up to 30 mol % Mg were observed at significantly lower temperatures. Precise resistivity measurements by Feitsma et al. (7) finally helped to get correct solubility data. The liquidus line of (7) was in fair agreement with that of (3). As the components were most contaminated in (3) and most pure in (7), the similarity of data published in the two papers is surprising. The results of (2) and (7) agreed in the range of 10-60 mol % Mg, those of (6) and (7) in the range of 50-90 mol % Mg, but the results of (4,5) and (7) agreed only in the range 0-6 mol % Mg. The high divergence of the results of (4,5) from those of (6) might be due to a formation of LiH, though (4) excluded a formation of LiH in their experiments. Most recently Schürmann and Voss (8) performed thermal analysis measurements to determine the liquidus line and the other phase relations. The results (8) were in fair agreement with those of (2),(3) and (7) in the range of 0-30 mol % Mg. Nevertheless, the liquidus curve at higher concentrations of Mg was close the values published in (4,5), with the maximum at 65 mol % and 867 K and the eutectic at 75 mol % and 860 K.

The Mg-Li system was critically evaluated by Nayeb-Hashemi et al. (9). According to (6),(10),(11) and (12) the saturated solution of Mg in liquid Li may be in equilibrium with solid Li saturated with Mg, solid Mg saturated with Li and their mixture (see the Mg-Li phase diagram based on (9)). The previously suggested formation of Mg-Li compounds (3,4) could not be confirmed. Saboungi and Hsu-Chen (13) calculated the Mg-Li phase relations on the basis of the results of (1-6,10,11) and supplementary thermodynamic data. These values differed from the selected ones given below. Saunders (14) performed a similar calculation and presented the results in a figure. They showed a qualitative agreement with the experiments.

Recommended (r) and tentative (t) values of the solubility of Mg in liquid Li

T/K	*soly*/mol % Mg	source
473	4 (r)	(3),(7),(8) [a]
573	18 (r)	(2),(3),(7),(8) [a]
673	29 (r)	(2),(3),(7),(8) [a]
773	44 (t)	(2)
865	70 (r) maximum	(3),(7)
861	77 (r) eutectic	(3),(6),(7)
873	81 (t)	(6),(7) [a]
898	90 (r)	(3),(6),(7)

[a] interpolated



References

1. Masing, G.; Tammann, G. *Z. Anorg. Chem.* 1910, *67*, 183.
2. Henry, O.H.; Cordiano, H. *Trans. AIME* 1934, *111*, 319.
3. Grube, G.; Zeppelin, H.; Bumm, H. *Z. Elektrochem.* 1934, *40*, 160.
4. Saldau, P.Ya.; Shamrai, F.I. *Z. Anorg. Chem.* 1935, *224*, 388; *Izv. Akad. Nauk. SSSR, Otdel. Matem. Estestv. Nauk, Ser. Khim.* 1936, 349.
5. Shamrai, F.I. *Izv. Akad. Nauk. SSSR, Otd. Khim. Nauk* 1947, 605.
6. Freeth, W.E.; Raynor, G.V. *J. Inst. Met.* 1953-54, *82*, 575.
7. Feitsma, P.D.; Lee, T.; van der Lugt, W. *Physica, B+C* 1978, *93*, 52.
8. Schürmann, E.; Voss, H.J. *Giessereiforschung* 1981, *33*, 35.
9. Nayeb-Hashemi, A.A.; Clark, J.B.; Pelton, A.D. *Bull. Alloy Phase Diagr.* 1984, *5*, 365; *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p.1487.
10. Hume-Rothery, W.; Raynor, G.V.; Butchers, E. *J. Inst. Met.* 1945, *71*, 589.
11. Catterall, J.A. *Nature* 1952, *169*, 336.
12. Schürmann, E.; Geißler, I.K. *Giessereiforschung* 1980, *32*, 163.
13. Saboungi, M.L.; Hsu-Chen, C. *CALPHAD* 1977, *1*, 237; *Nat. Bur. Stand. Rep. SP-496*, 1978, p. 1109.
14. Saunders, N. *CALPHAD* 1990, *14*, 61.

COMPONENTS:

(1) Magnesium; Mg; [7439-95-4]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Grube, G.; Zeppelin, H.; Bumm, H.

Z. Elektrochem. 1934, *40*, 160-4.

VARIABLES:

Temperature: 489-911 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The temperature of the liquidus line was reported.

t/°C	*soly*/mol % Li	*soly*/mol % Mg [a]	*t*/°C	*soly*/mol % Li	*soly*/mol % Mg [a]
638	5.5	94.5	591.5	30.1	69.9
625	9.5	90.5	591.5	31.0	69.0
607	16.3	83.7	589.5	34.3	65.7
602	17.3	82.7	587	36.2	63.8
601	17.7	82.3	581	38.2	61.8
599	18.6	81.4	568	42.5	57.5
591	20.3	79.7	548	47.5	52.5
587.5	21.8	78.2	512	54.8	45.2
589	22.6	77.4	468	61.0	39.0
589	23.0	77.0	440	65.0	35.0
590.5	24.0	76.0	403	70.0	30.0
591	24.5	75.4	364	75.0	25.0
592	25.1	74.9	322	79.3	20.7
592	27.5	72.5	240	89.7	10.3
592	28.8	71.2	216	94.2	5.8

Further measurements of the liquidus were performed in the vicinity of the melting point of Li_2Mg_5.

t/°C	*soly*/mol % Li	*soly*/mol % Mg [a]	*t*/°C	*soly*/mol % Li	*soly*/mol % Mg [a]
587.5 [b]	21.8	78.2	592.0	28.0	72.0
589.0	24.0	76.0	592.0	30.0	70.0
589.5	24.0	76.0	592.0	32.0	68.0
591.0	26.1	73.9			

[a] - calculated by the compilers [b] double experiment

The eutectic point was established at 78.0 mol % Mg and 585 °C by means of resistance measurements or at 587.5 °C by thermal analysis.

The melting points of Mg and Li were reported to be 650 and 179 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed in an Ar atmosphere. The Mg-Li mixtures were melted in a low-carbon Fe crucible. Corrosion attack of the melt on the crucible was not observed. Cooling curves of the liquid alloys were recorded by means of a Ni/Nichrome thermocouple, which had been calibrated against the melting points of Sn, Pb, Zn, Sb and Mg. The solid alloys were prepared in a steel container for conductivity measurements, which were performed in a glass tube.

SOURCE AND PURITY OF MATERIALS:

Mg: 99.81 % pure, from I.G.Farbenindustrie, Bitterfeld, containing 0.07 % Zn, 0.02 % Si, 0.01 % Cu, 0.04 % Mn, 0.05 % Al + Fe, according to (1).

Li: 99.0 % pure, from Metallgesellschaft A.G., Frankfurt, containing 0.62 % K, 0.14 % Na, 0.02 % Fe_2O_3, traces of Al_2O_3, 0.05 % SiO_2, 0.32 % LiN_3.

Ar: 98.2 % purity, 0.1 % O_2 and 1.7 % N_2; further purified by passing through molten Li (1).

ESTIMATED ERROR:

Nothing specified.

Solubility: precision ± 0.1 mol % (by the compilers).

Temperature: precision ± 0.5 K (by the compilers).

REFERENCES:

1. Grube, G.; Bornhak, R. *Z. Elektrochem.* 1934, *40*, 140.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Magnesium; Mg; [7439-95-4] (2) Lithium; Li; [7439-93-2]	Henry, O.H.; Cordiano, H. *Trans. AIME* 1934, *111*, 319-32.
VARIABLES:	**PREPARED BY:**
Temperature: 474-911 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus curve of the Mg-Li system was determined and the solubility of Mg in liquid Li was calculated by the compilers.

t/°C	*soly*/mass % Li	*soly*/mol % Mg	*t*/°C	*soly*/mass % Li	*soly*/mol % Mg
638	2.1	93.0	567	17.8	56.9
635	2.6	91.4	558 [a]	19.0	54.9
622	3.85	87.7	555	20.2	53.0
620 [a]	3.86	87.7	517	24.9	46.3
618	4.0	87.3	501 [a]	26.9	43.7
615	4.85	84.9	472	30.7	39.2
606 [a]	5.6	82.8	453	33.0	36.7
608	6.0	81.7	421 [a]	36.8	32.9
607	6.57	80.2	419	38.0	31.8
603 [a]	6.57	80.2	407	39.6	30.3
602	7.05	79.0	362	46.0	25.1
598	7.51	78.5	335 [a]	50.0	22.2
596 [a]	8.55	76.3	306	55.0	18.9
593	12.5	66.6	282	60.0	16.0
591	8.0-11.5	77.4-68.6	262	64.8	13.4
591 [a]	12.5	66.6	245	70.5	10.7
589	13.1	65.4	234	75.1	8.6
590	13.4	64.8	222 [a]	76.3	8.1
587 [a]	13.4	64.8	226	80.0	6.7
586	14.1	63.5	218	85.0	4.8
587 [a]	14.1	63.5	211	90.0	3.1
581	15.0	61.8	201	94.4	1.66
578	16.0	60.0			

[a] - measured by a recording potentiometer.

The melting points of Li and Mg were reported to be 186 and 651 °C, but it is not known whether they were experimentally determined or taken from literature.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in closed Fe tubes. Cooling curves of the molten alloys were recorded by means of a Cr/Alumel thermocouple which was calibrated against the melting points of Sn and Mg. The temperature was determined by a millivoltmeter or by a recording potentiometer.

SOURCE AND PURITY OF MATERIALS:

Mg: 99.91 % purity; supplied by Dow Chem. Co., containing 0.028 % Al, 0.035 % Fe, 0.020 % Si, 0.004 % Mn, 0.003 % Ni.
Li: 99.9 % purity; supplied by Maywood Chem. Works.

ESTIMATED ERROR:

Solubility: accuracy ± 0.2 % in chemical analysis of selected samples.
Temperature: precision between 1 and 2.5 K.

REFERENCES:

COMPONENTS:

(1) Magnesium; Mg; [7439-95-4]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Saldau, P.Ya.; Shamrai, F.I.

Z. Anorg. Chem. 1935, *224*, 388-98.

VARIABLES:

Temperature: 467-918 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points on the Mg-Li phase diagram were reported:

t/°C	*soly*/mol % Mg	t/°C	*soly*/mol % Mg	t/°C	*soly*/mol % Mg
194	2.6	412	27.6	589	72.4
204	4.2	455	32.4	588.2	73.2
217	5.8	477	36.3	599	75.3
227	8.0	527	42.3	611	78.4
244	9.6	539	44.2	621	80.7
253	10.2	565	49.4	622	83.2
268	12.7	580	55.2	627	84.5
286	14.7	594	62.2	634	88.5
310	16.6	598.8	65.7	637	90.9
342	19.5	599.6	66.6	639	93.3
365	23.2	596	67.7	645	95.9
389	24.6	592	69.2		

The melting point of Li was found at 180 °C.

The liquid was in equilibrium with solid Li saturated with Mg, $LiMg_2$ or solid Mg. The results were also published in (1), the same results being then graphically presented in (2). It is not obvious, if the application of an environmental shielding of the alloys with molten LiCl-KCl was of influence on the results of (1,2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The preparation of the alloys was performed in a Fe crucible in a H_2 atmosphere; formation of LiH was not observed. Mg rich alloys were prepared under the protective cover of molten LiCl-KCl (46:54). The cooling curves were recorded by means of a Kurnakow's (3) pyrometer, which had been calibrated at the melting points of Sn, Zn, Sb, and the boiling point of H_2O. The thermocouple was Pt/Pt-Rh(10 %). The Mg content in the samples was determined by the oxyquinoline method. Direct analyses of the Li content did not provide precise results.

SOURCE AND PURITY OF MATERIALS:

Mg: vacuum distilled; 99.9 % purity.
Li: 99.5 % purity.
H_2: purified by blowing through molten Na, Mg fillings at 773-823 K and molten Li (2).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Saldau, P.Ya.; Shamrai, F.I. *Izv. Akad. Nauk SSSR, Otd. Matem. Estestv. Nauk, Ser. Khim.* 1936, 349.
2. Shamrai, F.I., *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk* 1947, 605.
3. Kurnakow, N.S. *Z. Anorg. Chem.* 1907, *42*, 184; *Zh. Russ. Fiz.-Khim. Obshch.* 1904, *36*, 341.

COMPONENTS:

(1) Magnesium; Mg; [7439-95-4]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Freeth, W.E.; Raynor, G.V.

J. Inst. Met. 1953-54, *82*, 575-80.

VARIABLES:

Temperature: 461-909 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the Mg-Li phase diagram were taken from the published figure by the compilers:

t/°C	*soly*/ mol % Mg	*t*/°C	*soly*/ mol % Mg	*t*/°C	*soly*/ mol % Mg
188	2.0	573	60.2	590.5	76.5
195	4.0	578	61.3	588.5	77.3
199	5.0	584	64.4	589	77.5
201	6.0	591.5	67.2	589.5	77.7
205	7.0	593.5	69.5	594.5	79.5
217	10.0	594	70.7	600	81.0
233	15.0	594	71.5	604	82.6
264	20.0	593.5	72.1	609	83.9
330	30.0	593	73.0	616	87.0
518	46.8	592.5	74.0	624	90.0
541	51.2	591.5	75.0	636	95.0
561	56.4	591	76.0		

The melting points of Li and Mg were determined to be 180 and 647 °C, respectively.

The equilibrium solid phases were composed of Li saturated with Mg, Mg saturated with Li and their mixed crystals.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in crucibles of mild steel and washed internally with magnesia. The furnace used for thermal analysis was provided with a mechanical stirrer. Purified Ar served as an inert gas atmosphere, and the temperature differences between heater and specimen were measured by means of a standardized thermocouple. For thermal analysis, Mg-Li alloys were varied by additions of Li to Mg; freshly prepared alloys were used if necessary. Selected alloys were analyzed by Freeth and Raynor or by Johnson, Matthey & Co. Ltds. laboratory.

SOURCE AND PURITY OF MATERIALS:

Mg: 99.99 % purity; supplied by the Dominion Magnesium Comp., Toronto.
Li: supplied by New Metals & Chemical Ltd.; containing $\leq$ 0.02 % Na.
Ar: "specially purified".

ESTIMATED ERROR:

Solubility: accuracy: ± 0.5 %.
Temperature: nothing specified; read-off procedure: ± 2 K.

REFERENCES:

COMPONENTS:

(1) Magnesium; Mg; [7439-95-4]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Feitsma, P.D.; Lee, T.; van der Lugt, W.

Physica B+C 1978, *93*, 52-8.

VARIABLES:

Temperature: 489-913 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Mg-Li system was presented in a figure and experimental points were read by the compilers.

t/°C	*soly*/mol % Mg	*t*/°C	*soly*/mol % Mg
216	6	582	61.5
240	10	588	63.5
324	20.5	590	68.5
367	25	592	71
404	30	587	78 (eutectic)
442	36.5	607	83
470	38.5	625	90
512	45	640	95
550	52		

The melting points of Mg and Li were found to be 650 and 180 °C, respectively.
Additional results obtained by means of the same method were reported in (2), as follows:

t/°C	*soly*/mol % Mg	*t*/°C	*soly*/mol % Mg
240	10.0	570	58.6
330	19.3	590	68.5
380	27.0	590	80.0
480	39.7	610	87.7
530	49.6		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metallic components were melted in a stainless steel container in an Ar glove box to prepare the alloys (1). The resistivity of the samples was determined in a measuring tube made of stainless steel with Pt leads. After heating to 50 K above the liquidus temperature the alloy was stirred by means of Ar bubbling. The alloy was then slowly raised from the container into the tube. The homogeneity of the samples was checked by a repetition of the measurements after the tube had been emptied and refilled. The obtained data were accepted if the discrepancy between two measurements did not exceed 0.1 %. The temperature was gradually reduced by a few K, and even by 1 K near the liquidus. The liquidus points were determined by a sharp decrease of the resistivity versus temperature as determined by a Pt/Pt-Rh (10%) thermocouple.

SOURCE AND PURITY OF MATERIALS:

Mg: 99.95 % pure; supplied by Alfa Metals.
Li: 99.98 % pure; supplied by Koch-Light. Na = 0.0030 %; K = 0.0033 %; Cl = 0.0031 %; N = 0.0019 %; Al, Ca, Co, Cr, Cu, Fe, Ni, Si, each < 0.001 %.
Ar: N, O and H_2O content less than $1 \cdot 10^{-4}$ %.

ESTIMATED ERROR:

Solubility: nothing specified; read-out procedure ± 0.5 mol %.

Temperature: precision ± 1 K; read-out procedure ± 3 K.

REFERENCES:

1. Feitsma, P.D.; Hallers, J.J.; van der Werff, F.; van der Lugt, W. *Physica B+C* 1975, *79*, 35.
2. van Oosten, A.B.; van der Lugt, W. *Rep. Solid State Phys. Labor.*, Univ. Groningen, Netherlands, 1986.

COMPONENTS:

(1) Magnesium; Mg; [7439-95-4]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Schürmann, E.; Voss, H.J.

Giessereiforschung 1981, *33*, 35-8.

VARIABLES:

Temperature: 460-920 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the Mg-Li system were read by the compilers from a figure:

t/°C	*soly*/ mol % Mg	t/°C	*soly*/ mol % Mg	t/°C	*soly*/ mol % Mg
647	96.3	590	72.4	482	39.3
640	92.8	591	71.9	442	33.5
631	88.9	592	71.2	401	29.4
625	86.5	593	70.0	351	23.6
618	84.2	593	68.0	326	20.5
613	82.7	594	67.2	284	16.2
607	80.9	594	65.0	244	11.1
597	78.1	593	63.0	229	9.2
593	76.6	591	61.1	211	6.7
591	76.2	584	58.7	207	5.1
589	75.5	569	55.0	196	3.3
588	73.9	536	48.2	187	1.2
590	72.7	498	41.7		

The melting points of Mg and Li were found to be 649 and 180 °C, respectively. The equilibrium solid phases are composed of Li saturated with Mg, Mg saturated with Li and their mixed crystals.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

According to (1), the thermal analyses were performed in a closed steel vessel under Ar pressure. The vessel was placed in a furnace which was flooded with Ar cover gas. The temperature was measured and controlled by means of Pt/Pt-Rh (10%) thermocouples. The electronic control unit was connected to a processor, which kept the temperature constant. The lower cup of the furnace tube was opened to dip the steel vessel in water in order to quench the system. The alloys were homogenized for 15 min at 973 K before cooling.

SOURCE AND PURITY OF MATERIALS:

Mg: > 99.8 % pure with contents of < 0.10 % Si and < 0.10 % (Mn+Cu+Ni+Fe).
Li: > 99.8 % pure with contents of < 0.2 % (Na+K); vacuum remelted, solid surface removed.
Ar: nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision and stability ± 0.5 K.

REFERENCES:

1. Schürmann, E.; Voss, H.J.; *Giessereiforschung* 1981, *33*, 33-35.

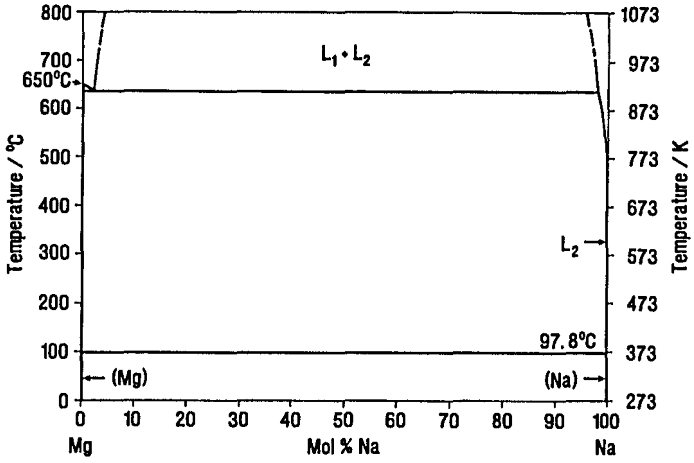
COMPONENTS:	EVALUATOR:
(1) Magnesium; Mg; [7439-95-4] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1987

CRITICAL EVALUATION:

In early experiments on the Mg-Na system, Mathewson (1) observed a limited miscibility of both metals in the liquid state. The homogeneous solution could be obtained at 911 K, if the Mg content was 1.5 mol % or less. At the same temperature the miscibility gap extended to 97.9 mol % Mg (eutectic point).
The region of dilute solutions of Mg in liquid Na was investigated by Klemm and Kunze (2). The authors reported a solubility of Mg which increased almost regularly from 0.7 to 7.2 mol % at 573 to 910 K, the value at 573 K being comparatively too low. Lantratov's (3) determination of the liquid solubility of Mg (2.5 mol % at 973 K) was close to the value of 1.8 mol % Mg found in (1), and thus threw suspicion on the results of Klemm and Kunze (2) in the dilute Mg solution range. Davis and Draycott (5) reported a solubility of 1.04 mol % Mg in liquid Na at 423 K, but did not mention the source of their data. The value seems to be to large. In a recent study Addison (7) expressed the opinion that the data of (1) and (2) are much too large, but he did not present alternatives.
Johnson and coworkers (4) presented a solubility equation for Mg in Na, which agreed neither with the experimental point of (1) nor with the values of (2).
The Mg-Na system needs further examination, especially in the range of immiscibility. Intermetallics between Mg and Na are not formed. The schematic phase diagram of the Mg-Na system is given in the figure, according to the evaluation of the Mg-Na system by Pelton (6).

Recommended (r) and tentative (t) values of the solubility of Mg in liquid Na

T/K	*soly*/mol % Mg	source	comments
910	1.5 (t)	(1)	
910	98 (r)	(1,2)	eutectic on the Mg-rich side
973	2.5 (t)	(4)	



References

1. Mathewson, C.H. *Z. Anorg. Chem.* 1906, *48*, 191.
2. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3.
3. Lantratov, M.F. *Zh. Prikl. Khim.* 1973, *46*, 1982.
4. Johnson, H.F.; McKisson, R.L.; Eichelberger, R.L.; Gehri, D.C. *Sodium Na-K Engineering Handbook*, O.J. Foust, Ed., Gordon & Breach, N.Y., 1972, vol. 1, p. 169.
5. Davis, M.; Draycott, A. *Peaceful Uses of Atom. Ener.*, U.N., N.Y., 1958, *7*, 94.
6. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1984, *5*, 454.
7. Addison, C.C. *The Chemistry of Liquid Alkali Metals*, Wiley, Chichester, 1984, p. 71.

COMPONENTS:

(1) Magnesium; Mg; [7439-95-4]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Mathewson, C.H.

Z. Anorg. Chem. 1906, *48*, 191-200.

VARIABLES:

Temperature: 911-930 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquid miscibility gap between 1.6 and 98.0 mass % Mg (1.5 and 97.9 mol % Mg respectively, as calculated by the compilers) was established at 638 °C. Mathewson reported a solubility of 1.6 mass % Mg at 657 °C, but it is likely that this value corresponded to the lower limit of immiscibility at 638 °C. A solubility of 99.1 mass or mol % Mg at 645 °C was also reported. The melting points of Na and Mg were found at 97.5 and 650 °C, respectively.
Mg-Na intermetallics were not detected in the system.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The preparation of the Mg-Na alloys was performed in high-temperature (Jena) glass (K-Ca). The surface of the glass was attacked by Na, the weight-loss of Na being negligible. The Mg attack was more distinct, however, merely traces of Ca were leached from the glass. The thermal analysis experiments were controlled by means of a Pt/Pt-Rh thermocouple. The melts were cooled by stirring with a Fe wire.

SOURCE AND PURITY OF MATERIALS:

Mg: 99.9 % purity, containing no measurable amounts of other metals.
Na: no metallic impurities detected.

ESTIMATED ERROR:

Nothing specified.
Temperature: accuracy ± 1 K (estimated by compilers).

COMPONENTS:

(1) Magnesium; Mg; [7439-95-4]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Lantratov, M.F.

Zh. Prikl. Khim. 1973, *46*, 1982-6; *J. Appl. Chem. USSR* 1973, *46*, 2107-10.

VARIABLES:

One temperature: 973 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of liquid Mg in liquid Na at 700°C is 2.5 ± 0.1 mol %. The solubility of liquid Na in liquid Mg at 700°C is 2.7 ± 0.1 mol % Na or 97.3 mol % Mg.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

According to (1) the alloys were prepared by melting weighed amounts of both metals in an apparatus of (3S-5K) high-melting borosilicate glass. The alloy was placed in an electrochemical concentration cell made of the same glass, together with pure Na. The EMF's of the cell were measured to determine the alloy composition, which changed from 0.5 to 99.0 mol % Na.

The homogeneity range of the Mg-Na melt was determined from the breaks on the dependence of E vs. log x_{Na}.

The temperature was controlled by means of an Al/Cr thermocouple. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Mg: high purity (1).
Na: high purity (1).
Ar: specially purified.

ESTIMATED ERROR:

Solubility: precision ± 4 %.
Temperature: nothing specified.

REFERENCES:

1. Lantratov, M.F.; Tsarenko, E.V. *Zh. Fiz. Khim.* 1959, *33*, 1792.

COMPONENTS: (1) Magnesium; Mg; [7439-95-4] (2) Sodium; Na; [7440-23-5]	**ORIGINAL MEASUREMENTS:** Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3-22.
VARIABLES: Temperature: 573-910 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mg in Na was presented in a figure; the numerical data were taken from (1):

t/°C	*soly*/mol % Mg
150	0.14
300	0.78
400	2.09
475	3.31
550	4.93
600	6.02; 6.17

A miscibility gap was established at 637 °C between 7.2 and 98 mol % Mg as confirmed by thermal analysis

The melting points of Na and Mg were found at 97.9 and 650°C, respectively.

Intermetallic Mg-Na compounds were not detected in the system.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

For the determination of the solubility a crucible of soft iron was applied, which had been heated under high vacuum for several hours. All operations were performed in an Ar atmosphere. A small piece of Mg was suspended in liquid Na and heated to the desired temperature. Thermal analysis of the Mg-Na alloy was performed by means of the procedure described in (1). The solubility of Mg in Na was determined by measurements of the weight loss of a Mg sample after equilibration with liquid Na for 20-60 hours. After withdrawing from Na the Mg sample was dipped in H_2O to remove adhering Na and dried.

SOURCE AND PURITY OF MATERIALS:

Mg: "highest degree of purity".
Na: analytical grade, from Merck, further distilled under high vacuum.
Ar: 99.9 % purity, from Linde, further purified in respect to O, N, and H_2O.

ESTIMATED ERROR:

Solubility: precision ± 2 % (by the compilers).
Temperature: precision ± 1 K (by the compilers).

REFERENCES:

1. Kunze, D. *M.S. Thesis*, Univ. of Münster, Germany, 1962; *Ph.D. Thesis*, Univ. of Münster, Germany, 1964.

COMPONENTS:	EVALUATOR:
(1) Magnesium; Mg; [7439-95-4] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1987

CRITICAL EVALUATION:

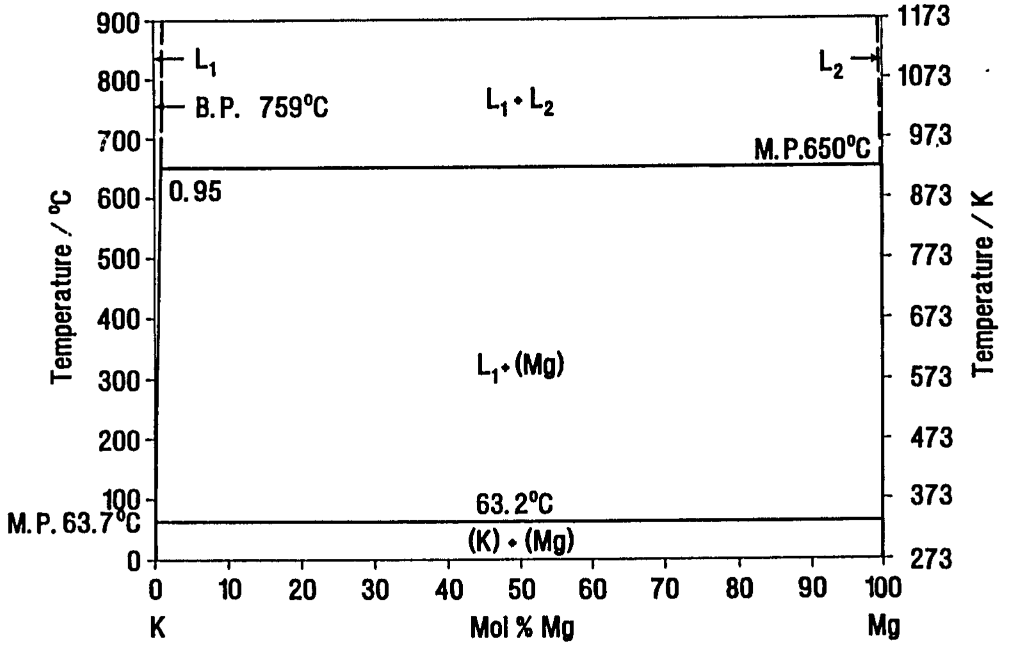
Only a few data on measurements of the solubility of Mg in liquid K are available. Klemm and Kunze (1) obtained 4 experimental points between 0.05 mol % Mg at 433 K and 0.8 mol % Mg at 873. According to their examination by means of thermal analysis a miscibility gap at the melting temperature of Mg (923 K) begins at 0.8 mol % Mg and extends to practically pure Mg. All these solubility values seem to be tentative, except for the one at 573 K.

Lantratov (2) reported a solubility of liquid Mg in liquid K equal to 1.0 mol % Mg at 973 K. The data of (1) and (2) were in fair agreement, as were earlier qualitative observations by Smith (3) of a limited miscibility of both liquid metals at 923 K. They indicate that the Mg solubility values in K are lower than in Na for a selected temperature range. Aleksandrov and Dalakova (5) did not detect Mg in liquid K by means of spectral analysis after an equilibration of the metals for 1 h at 873-923 K.

A formation of Mg-K intermetallics was not observed. The Mg-K system was critically evaluated by Pelton (4), who presented an assessed phase diagram; this is redrawn below.

Tentative values of the solubility of Mg in liquid K

T/K	$soly$/mol % Mg	source
723	0.5	(1)
873	0.8	(1)
973	1.0 (liquid Mg)	(2)



Symbols of solid elements are set in parentheses.

References

1. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3.
2. Lantratov, M.F. *Zh. Prikl. Khim.* 1973, *46*, 1982.
3. Smith, D.P. *Z. Anorg. Chem.* 1908, *56*, 109.
4. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1985, *6*, 39.
5. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Magnesium; Mg; [7439-95-4]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Klemm, W.; Kunze, D.

The Alkali Metals, The Chemical Society, London, 1967, p. 3-22.

VARIABLES:

Temperature: 443-873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mg in liquid K was reported in a figure; numerical data were taken from (1).

t/°C	*soly*/mol % Mg
150	0.02
300	0.38
450	0.49
600	0.70

A miscibility gap beginning at 650 K and at 0.8 mol % Mg was determined by thermal analysis.

The melting points of K and Mg were measured to be 63.6 and 650°C, respectively.
A formation of Mg-K intermetallics was not observed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

For the determination of the solubility a crucible of soft iron was applied, which had been heated under high vacuum for several hours. All operations were performed in an Ar atmosphere. A small piece of Mg was suspended in liquid K and heated to the desired temperature. Thermal analysis of the Mg-K alloy was performed by means of the procedure described in (1). The solubility of Mg in K was determined by measurements of the weight loss of a Mg sample after equilibration with liquid K. After withdrawing from K the Mg sample was dipped in H_2O to remove K and dried.

SOURCE AND PURITY OF MATERIALS:

Mg: "highest degree of purity".
K: high vacuum distilled, from Merck.
Ar: 99.9 % pure, from Linde, further purified in respect to O, N, and H_2O.

ESTIMATED ERROR:

Solubility: nothing specified, ± 0.05 mol % (by the compilers).
Temperature: precision ± 1 K (by the compilers).

REFERENCES:

1. Kunze, D. *M.S. Thesis*, Univ. of Münster, Germany, 1962; *Ph.D. Thesis*, Univ. of Münster, Germany, 1964.

COMPONENTS:

(1) Magnesium; Mg; [7439-95-4]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Lantratov, M.F.

Zh. Prikl. Khim. 1973, *46*, 1982-6; *Engl. Transl. J. Appl. Chem. USSR* 1973, *46*, 2107-10.

VARIABLES:

One temperature: 973 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of liquid Mg in liquid K at 700°C was determined to be 1.0 mol %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

According to (1) the alloys were prepared by melting weighed amounts of both metals in an apparatus of high-melting borosilicate glass (3S-5K). The alloy obtained was placed in an electrochemical concentration cell made of the same glass, with pure K serving as reference electrode. The EMF's of the cell were measured changing the alloy compositions from 0.2 to 80.0 mol % K. The solubility of Mg in K was determined from the break point on the dependence E vs. log x_K. The temperature was controlled by means of an Al/Cr thermocouple. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Mg: high purity (1).
K: filtered through G-3 glass filter.
Ar:"specially purified".

ESTIMATED ERROR:

Nothing specified.
Solubility: precision ± 4 % (by the compilers).

REFERENCES:

1. Lantratov, M.F.; Tsarenko, E.V. *Zh. Fiz. Khim.* 1959, *33*, 1792.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Magnesium; Mg; [7439-95-4] (2) Rubidium; Rb; [7440-17-7]	Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3-22.
VARIABLES:	**PREPARED BY:**
Temperature: 423-873 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mg in liquid Rb was reported in a figure; numerical data were taken from (1).

t/°C	*soly*/mol % Mg
150	0.06
250	0.65
300	0.84; 0.74
450	0.94; 0.88
520	0.98
600	1.08

A miscibility gap begins at 650 °C and ~ 1.2 mol % Mg as confirmed by thermal analysis.
The melting points of Rb and Mg were found to be 38.6 and 650°C, respectively.

COMMENTS AND ADDITIONAL DATA:

Intermetallic compounds could not be determined in the Mg-Rb system. The assessed phase diagram as reported by Pelton (2) is qualitatively of the same type as that of the Mg-K system; the deviation is due to the different melting points of K and Rb. The immiscibility range needs further investigation. The experimental values of the solubility show significant scatter, however, they seem to be in an acceptable order of magnitude. A fitting equation is not recommended.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

For the determination of the solubility a crucible of soft iron was used. The whole apparatus had been heated under high vacuum for several hours. All experiments were performed in an Ar atmosphere. A small piece of Mg was suspended in liquid Rb and heated to the desired temperature. Thermal analysis of the Mg-Rb alloy was performed by means of the procedure described in (1). The solubility of Mg in Rb was determined by measurements of the weight loss of a Mg sample after equilibration with liquid Rb. After withdrawing from Rb the Mg sample was dipped in H_2O to remove adhering Rb and dried.

SOURCE AND PURITY OF MATERIALS:

Mg: "highest degree of purity".
Rb: high vacuum distilled.
Ar: specially purified.

ESTIMATED ERROR:

Nothing specified.
Solubility: precision ± 0.1 mol % Mg (by the compilers).
Temperature: precision ± 5 K (by the compilers).

REFERENCES:

1. Kunze, D. *M.S. Thesis*, Univ. of Münster, Germany, 1962; *Ph.D. Thesis*, Univ. of Münster, Germany, 1964.
2. Pelton, A.D. *Bull. Alloy Phase Diagrams* 1985, *6*, 41-42.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Magnesium; Mg; [7439-95-4] (2) Cesium; Cs; [7440-46-2]	Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3-22.
VARIABLES:	**PREPARED BY:**
Temperature: 428-873 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mg in liquid Cs was measured.

t/°C	$soly$/mol % Mg
150	0.10
300	0.54
450	0.92
600	1.35
600	1.56

A miscibility gap begins at 650 °C and ~1.8 mol % Mg as confirmed by thermal analysis.

COMMENTS AND ADDITIONAL DATA:

The melting points of Cs and Mg were found to be 28.5 and 650°C, respectively.
Intermetallics could not be determined in this system. The phase diagram Mg-Cs was reportedby Pelton (3) and is of the same type as that of the Mg-K system, with the only difference in the melting points of Cs and K. The liquidus line above 650°C needs further experimental determination. The solubility results can be regarded as tentative. Small amounts of Mg in Cs were determined in compatibility tests (2) of various alloys (containing small amounts of Mg) in Cs at temperatures around 613 and 300 K and determined to be 0.04 and 0.016 mol % Mg, respectively. Since these experiments were no solubility determinations, the study (2) is not compiled.
The tabulated data were used by the compilers to calculate the solubility equation.

$$\log(soly/\text{mol \% Mg}) = 1.257 - 935(T/\text{K})^{-1} \qquad r=0.991$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

For the determination of the solubility a crucible of soft iron was used. The whole apparatus had been heated under high vacuum for several hours. All experiments were performed in an Ar atmosphere. A small piece of Mg was suspended in liquid Cs and heated to the desired temperature. Thermal analysis of the Mg-Cs alloy was performed by means of the procedure described in (1). The solubility of Mg in Cs was determined by measurements of the weight loss of a Mg sample after equilibration with liquid Cs. After withdrawing from Cs the Mg sample was dipped in H_2O to remove adherent Cs and dried.

SOURCE AND PURITY OF MATERIALS:

Mg: "highest degree of purity".
Cs: distilled in high vacuum.
Ar: specially purified.

ESTIMATED ERROR:

Nothing specified.
Solubility: precision ± 0.1 mol % Mg (by the compilers).
Temperature: precision ± 5 K (by the compilers).

REFERENCES:

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2. Holley, J.H; Neff, G.R.; Weiler, F.B.; Winslow, P.M. *Electric Propulsion Development*, E. Stuhlinger Ed., Academic Press, N.Y., 1963, p.341.
3. Pelton, A.D. *Bull. Alloy Phase Diagrams* 1985, *6*, 37.

COMPONENTS:	EVALUATOR:
(1) Calcium; Ca; [7440-70-2] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1995

CRITICAL EVALUATION:

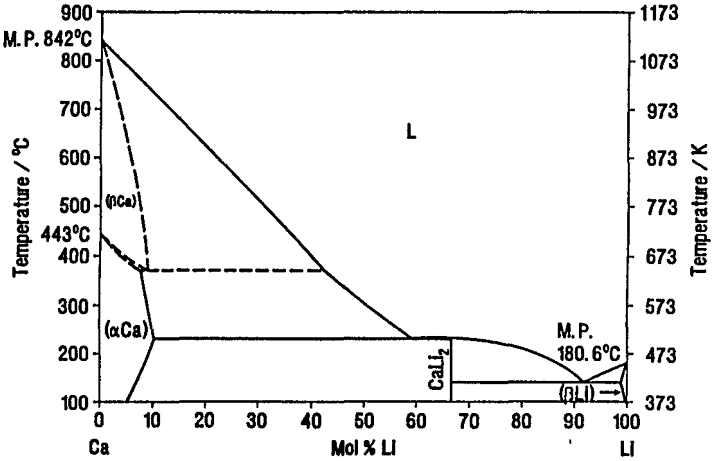
Zamotorin (1) was the first to investigate the liquidus of the Ca-Li system. However, most of his results were not confirmed in later studies. The solubility line which he had established by means of thermal analysis was at considerably higher temperatures (up to 300 K) than it should be. These deviations could not be explained, since the metals were of 99.6 % purity with a negligible N content and the experiments were performed in an Ar atmosphere. The only acceptable agreement with subsequent works is the temperature of a peritectic reaction at 498 K. Nevertheless, paper (1) is rejected and not compiled.

The Li-rich part of the phase diagram Ca-Li was studied by Wolfson (2). He used metals of a higher degree of purity than (1). Wolfson's results were subsequently confirmed in comprehensive investigations by Kanda and coworkers (3-7). Some liquidus points at higher Ca concentrations obtained by dilatometric measurements (3) may be neglected, since they deviate from results gained by thermal analysis (5-7). Some data for determinations in mixtures with high Ca content, which had been measured by means of thermal analysis, were reported graphically (5), but could not be evaluated due to the poor quality of the figures. Bale and Pelton (8) modified the Ca-Li phase diagram of Carfagno (7) by thermodynamic analysis. The liquidus line in the Ca-rich range was raised, thus causing lower solubility values at a given temperature. $CaLi_2$, which was assumed to have a peritectic decomposition at 403 K, was suggested to be congruently melting at a temperature slightly higher than 403 K; thus the new eutectic appeared at 41 mol % Ca. Another peritectic point at 643 K is most probably connected with the Ca allotropic transformation. Nevertheless, these predictions have to be confirmed by precise experiments. Accordung to preliminary experiments (9), the presence of N in the system may significantly change the phase relations.

The saturated solution of Ca in liquid Li is in equilibrium with $CaLi_2$ (3-7) or solid allotropes of Ca saturated with Li; see the Ca-Li phase diagram based on (8), redrawn in the figure below. The phases Ca_2Li (1) and CaLi (2), which had been previously reported, were not confirmed in this binary system.

Recommended (r) and tentative (t) values of the solubility of Ca in liquid Li

T/K	*soly*/mol % Ca	source	remarks
415	8 (r)	(2,3,6,7)	eutectic
473	18 (t)	(6,7)	
573	48 (t)	(6,7)	
673	59 (t)	(6,7)	
773	68 (t)	(6,7)	
873	77 (t)	(6,7) interpolation	
973	87 (t)	(6,7) interpolation	
1073	96 (t)	(6,7)	



Solid elements are indicated in parentheses

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9. Hubberstey, P.; Roberts, P.G. *Liquid Metal Systems*, H.U. Borgstedt, G. Frees, Eds., Plenum 1995, p.331.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Calcium; Ca; [7440-70-2] (2) Lithium; Li; [7439-93-2]	Wolfson, M.R. *Trans. Am. Soc. Met.* 1957, *49*, 794-804.
VARIABLES:	**PREPARED BY:**
Temperatures: 415 - 504 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

A portion of the liquidus of the Ca-Li system was determined.

t/°C	*soly*/mol % Ca
176.9	1
153.6	5
142.4	7.5
153.8	10
209.8	20
227.5	30
230.7	35

The melting points of Ca and Li were found to be at 860 and 180.5 °C, respectively.

The eutectic point for 7.7 mol % Ca was determined at 141.8 °C. The saturated solution of Ca in Li is in equilibrium with the solid solution of Ca in Li and with CaLi between the eutectic and peritectic at 230.9 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The pure metals were weighed in a dehydrated He atmosphere and subsequently placed in Armco Fe crucibles. Thermal analyses were performed in a vacuum furnace filled with He. Fe-Constantan thermocouples were used for temperature measurements. The determined liquidus was the average of three simultaneously recorded curves of temperature measurements during cooling and heating. All charges - except for Li- were kept at a temperature slightly above the melting point of Ca and were agitated for 5 minutes. The formation of solid CaLi was detected by hardness measurements.

SOURCE AND PURITY OF MATERIALS:

Ca: 99 % purity, supplied by Carbide & Carbon Chem. Co.; containing $\leq$ 0.6 % Na.
Li: 99 % purity, supplied by Maywood Chem. Works; containing $\leq$ 0.25 % Na.
He: "The Navy Grade - A" purity.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: better than + 3.8 and - 3.4 K.

REFERENCES:

COMPONENTS:

(1) Calcium; Ca; [7440-70-2]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Kanda, F.A.; King, A.J.; Keller, D.V.

US Atom.Ener.Comm. Rep. TID-5691, 1960, p. 50-3.

VARIABLES:

Temperature: 421-773 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility values of Ca in liquid Li were read from the figure by the compilers.

t/°C	*soly*/mol % Ca	t/°C	*soly*/mol % Ca	t/°C	*soly*/mol % Ca
500 [a]	75	215	21	148	8
412 [a]	62	196	18	148	6.5
370 [a]	41	184	15	155	5.5
230	29	167	13	162	4.5
220	25	160	10	172	2.3

[a] - from dilatometric measurements

$CaLi_2$ intermetallic with peritectic decomposition at 228-230 °C was formed in the system. Another peritectic point was determined at 415 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Ca was melted at 1173 K and cooled to room temperature in a Fe crucible. The surface layer of Ca with its impurities was scratched off. Li and Ca were inserted into the crucible of a high temperature dilatometer. The alloy formed during heating was stirred and homogenized. Cooling curves were made at controlled cooling rates. A sharp and reproducible break was observed at the temperature corresponding to the liquidus point. For low concentrations of Ca typical thermal analysis was performed. All operations were carried out in an Ar atmosphere. The crucibles were washed with HCl, H_2O and acetone, and finally heated to 1073 K in flowing H_2 atmosphere.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.
Ca: probable purity of 99.9+ % according to (1); supplied by the Inst. for Atom. Res. at Iowa State University.

ESTIMATED ERROR:

Nothing specified.
Solubility: ± 1 % (after read-out procedure).
Temperature: ± 5 K (after read-out procedure).

REFERENCES:

1. Kanda, F.A.; Keller, D.V. *US Atom.Ener.Comm. Rep. NYO-2731-3*, 1964.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Calcium; Ca; [7440-70-2] (2) Lithium; Li; [7439-93-2]	Carfagno, D.G. *US Atom.Ener.Comm. Rep. NYO-2731-7 (App.1)*, 1966.
VARIABLES:	**PREPARED BY:**
Temperature: 417-1091 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Points on the liquidus line of the Ca-Li system were reported.

t/°C	*soly*/mol % Ca	*t*/°C	*soly*/mol % Ca	*t*/°C	*soly*/mol % Ca
179	0.4	198	17.5	566	74.0
178	0.9	210	20.6	610	78.0
173	2.4	222	24.3	650	82.0
164	4.1	231	32.0	673	84.0
158	5.4	234	38.0	691	86.0
148	6.9	261	44.0	718	88.8
146	7.5	298	48.0	732	90.0
144	7.8	336	52.0	762	92.0
149	8.5	353	54.0	778	94.0
162	10.3	407	60.0	798	96.0
174	12.4	450	64.0	818	98.0
185	14.8	499	68.0		

The melting points of Li and Ca were found to be 180.5 and 842 °C, respectively.
The results were presented graphically in (1) and (2) and fragmentarily in (3).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

All experiments were performed in an Ar atmosphere. The components were properly weighed and the composition was subsequently determined by means of chemical analysis. The steel crucible (0.1 % C), which had previously been washed with HCl, H_2O and acetone, was used for melting and for the performance of the thermal analysis. Chromel/Alumel thermocouples were used, which had been calibrated against a Pt/Pt-Rh (10%) thermocouple as well as the melting point of Mg. The charges were heated to 50 K above the melting point of Ca and stirred vigorously. The cooling and heating curves were recorded and repeated three times. X-ray studies were performed on each solid sample.

SOURCE AND PURITY OF MATERIALS:

Ca: 99.94 % purity; supplied by Dominion Magnesium Ltd., Toronto; Mg being main impurity.
Li: 99.86 - 99.89 % purity; supplied by Foote Mineral Comp., major contaminant 0.09 - 0.10 % Na (spectrographically determined).
Ar: dried with 5A molecular sieves and passed over Ba chips at 673 K to remove O and N.

ESTIMATED ERROR:

Solubility: agreement of calculated composition and chem. anal. after investigation was within 1 %.
Temperature: precision ± 1 K.

REFERENCES:

1. Kanda, F.A.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-20849*, 1964.
2. Kanda, F.A.; Keller, D.V. *US Atom.Ener.Comm. Rep. NYO-2731-3*, 1964.
3. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom. Ener.Comm. Rep. TID-15150*, 1962, p. 27.

COMPONENTS:	EVALUATOR:
(1) Calcium; Ca; [7440-70-2] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1995

CRITICAL EVALUATION:

As Ca is important as the typical impurity in Na, the evaluators suppose that plenty of solubility data may be enclosed in internal reports of manufacturers and utilizers of metallic Na, but these papers are not available to the scientific community. The known data are fragmentary, however, it is agreed that the solubility of Ca in liquid Na is low at temperatures below 600 K and that both metals show limited miscibility in the liquid state. Moissan (1) reported low solubility of Ca in liquid Na, but he did not supply numerical data. Muthmann et al. (2) performed some experiments on the Ca-Na system at 1173-1223 K. However, the work is rejected due to an unacceptable scatter of the few reported numerical data and the lack of experimental details. Lorenz and Winzer (3,4) were the first to gain knowledge on the Ca-Na phase diagram. They determined the liquidus for the Ca-rich side of the diagram and estimated the limits of liquid immiscibility. The solubility of Ca in Na of 0.8 mol % at 973 K was reported, but the results of the solubility at higher temperatures were random and could therefore not be recommended. Rinck (5) emphasized that the Ca used in (3,4) had been contaminated with N, thus causing errors in these studies. The segregation of two liquid phases brought about a sluggish mixing, which might further explain the difficulties. Rinck (5-7) performed precise experiments on the phase relations of the system and his proposed phase diagram was sufficiently convincing. He measured the limits of the miscibility gap at temperatures between 985 and 1373 K and verified the Ca-rich part of the liquidus. The eutectic point of 84.0 mol % Ca at 973 K in (3) was corrected to be 77.0 mol % Ca at 985 K (7). Ca solubilities in Na in (7) were comparatively higher than in (3).

Our knowledge on the liquidus line between the melting point of Na and 985 K is mainly based on secondary sources (8,9). Adams and Sittig (8) presented the solubility equation:

$$\log\,(soly/\text{mass \% Ca}) = 2.5629 - 1545.6\,(T/\text{K})^{-1}, \text{ with a probable error of } \pm\, 0.14\ \% \qquad (1)$$

This equation, which is valid in the temperature range 370 to 973 K, was calculated from data of Rinck (7), Gilbert (10), and Epstein (11). The results of Rinck (7) were in rough agreement with the Eq.(1). Gilbert (10) reported solubilities of $2.3 \cdot 10^{-2}$, $2.7 \cdot 10^{-2}$, and 1.04 mol % Ca in Na at ~373, 398, and 673 K, respectively. Epstein (11) presented a set of solubility data,shown in the table, without giving experimental details.

t/°C	*soly*/mass % Ca	*soly*/mol % Ca	*t*/°C	*soly*/mass % Ca	*soly*/mol % Ca
97.81	0.025	0.0144	200	0.20	0.115
100	0.026	0.0149	300	0.74	0.42
125	0.048	0.027	400	1.8	1.04
150	0.082	0.047	500	3.7	2.2
175	0.15	0.086	700	9.4	5.6

The results of (11) are in fair agreement with Eq.(1) and with the data given by Rinck (7). Mausteller and coworkers (9) quoted an unpublished result of the solubility close to the melting point of Na, approximately amounting $6 \cdot 10^{-3}$ mol % Ca. This value, however, is only half of the value of (8). Kendall (12) analyzed Na saturated with Ca at 700 K, and reported 0.53 mol % Ca being dissolved. The data (9) and (12) are not presented in data sheets, since they differ significantly from the mean values of the solubility, and the method of determination was not reported in detail.

An inflection of the liquidus close to the temperature of allotropic transformation of Ca (~720 K) was not mentioned in (8), though such an effect might be expected. A more precise determination of the solubility is necessary to confirm this supposition. The eutectic point on the Na rich side was only 0.025 K lower than the melting point of Na (5,7).

The Ca-Na phase diagram redrawn after (13) is presented in the figure.

The presence of N or C or both of them as contaminants in the Na-Ca system leads to the formation of nitride, carbide or cyanamide of Ca which may influence the solubility equilibria (14).

Tentative values of the solubility of Ca in liquid Na

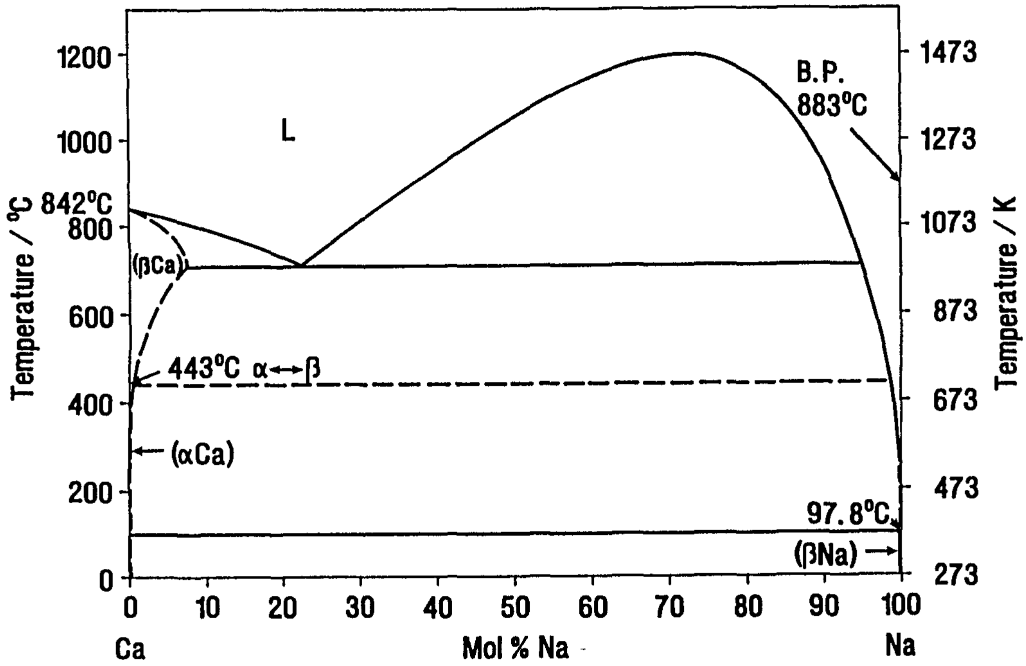
T/K	*soly*/mol % Ca	source	remarks
373	0.015	(8)	for (α Ca)
473	0.11	(8)	for (α Ca)
573	0.42	(8)	for (α Ca)
673	1.1	(8)	for (α Ca)
773	2.2	(8)	for (β Ca)
873	3.6	(8)	for (β Ca)
973	5.5	(8)	for (β Ca)
1073	7 liquid Ca	(7) interpolated	
1173	8.5 liquid Ca	(7) interpolated	at constrained pressure
1273	12 liquid Ca	(7) interpolated	at constrained pressure
1373	16.5 liquid Ca	(7)	at constrained pressure
1455	27 critical point	(7)	at constrained pressure

COMPONENTS:	EVALUATOR:
(1) Calcium; Ca; [7440-70-2] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1995

CRITICAL EVALUATION: (continued)

Tentative values of the liquidus in the Ca rich region

T/K	x_{Ca}	source
1373	39	(7)
1273	54	(7) interpolated
1173	61	(7) interpolated
1073	72	(7) interpolated
985	77 eutectic	(7)
1073	91	(7) interpolated



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2. Muthmann, W.; Weiss, L.; Metzger, J. *Liebigs Ann.* 1907, *355*, 137.
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5. Rinck, E. *C.R.Hebd. Acad. Sci.* 1930, *191*, 404.
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8. Adams, R.M.; Sittig, M. *Liquid Metals Handbook, Sodium Na-K Supplement*, C.B. Jackson, Ed., USAEC & NAVY, Washington DC, 1955, p. 3.
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13. Pelton, A.D. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 624; *Bull. Alloy Phase Diagr.* 1985, *6*, 35.
14. Pulham, R.J., Hubberstey, P. *J. Nucl. Mater.* 1983, *115*, 239.

COMPONENTS:

(1) Calcium; Ca; [7440-70-2]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Lorenz, R.; Winzer, R.

Z. Anorg. Chem. 1929, *179*, 281-6.

VARIABLES:

Temperature: 973-1083 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the liquidus line of the Ca-Na system were reported:

t/°C	*soly*/mol % Ca
790	98.5
780	97.2
760	94.4
720	86.8
700	84.0

The melting point of Ca was determined to be 809°C, this too low value indicates the presence of impurities in this metal.
The miscibility gap observed at 700°C is located between 0.8 mol % Ca (also reported in (1)) and the eutectic point at 84 mol % Ca. The experiments performed at higher temperatures result in inconclusive data. At a temperature of 810°C the immiscibility range begins at 2 % (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The thermal analysis experiments were performed in a steel container. The alloys were prepared by weighed amounts of Na and Ca. Cooling curves were recorded by means of a Pt/Pt-Rh thermocouple, which had been calibrated against the boiling points of H_2O and S as well as the melting points of Na, Pb, Sb, Zn, and NaCl. According to the authors, a formation of small amounts of carbide in the melt was observed, the solubility of which was reported to be negligible. The solubility value of Ca in Na (at a high Na content) was roughly confirmed in (1) by an investigation of the equilibrium of the reaction $2\ Na + CaCl_2 = Ca + 2\ NaCl$. The alloy-salts melt was kept in the closed Supremaxglass tube and equilibrated. It was rapidly cooled in CCl_4. The tube was broken in ether, dried and the metallic phase was treated with absolute methanol. The Na methanolate was titrated with HNO_3. The Ca residue was treated with H_2O and determined as $CaSO_4$. The salt phase was examined for Ca (as oxalate) and Cl contents and the composition of the alloy and the salt was calculated from the mass balance.

SOURCE AND PURITY OF MATERIALS:

Ca: 98,76 % purity; supplied by Kahlbaum; containing 0.14 % SiO, 0.11 % Al_2O_3, 0.11 % Fe_2O_3; almost no C and Cl.
Na: no impurities detected.
NaCl: supplied by Merck, dried 1 hour at 873 K.
$CaCl_2$: 99.30 % purity; supplied by Merck; dehydrated in china crucible and dried for ½ hour in dry HCl atmosphere at 1123 K; with contents of 0.05 % CaO, 0.12 % SiO_2, traces of Al.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 1-2 K.

REFERENCES:

1. Lorenz, R.; Winzer, R. *Z. Anorg. Chem.* 1929, *181*, 193.

COMPONENTS:

(1) Calcium; Ca; [7440-70-2]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Rinck, E.

Ann. Chim. 1932, *18*, 395-531.

VARIABLES:

Temperature: 985-1373 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

A miscibility gap between liquid Ca and Na was determined by investigation of the equilibrium of the reaction: 2 Na + $CaCl_2$ = 2 NaCl + Ca.

t/°C	*soly* in Na-rich phase/ mass % Ca	mol % Ca	*soly* Ca-rich phase/ mass% Ca	mol % Ca
712 (710)[a]	7[a]	4.0	86[a]	77.0 (eutectic)
850[b]	13[b]	7.26	83[b]	71.0
975[c]		10.85		56.2
1100[c]		16.5		39.3

[a] - as reported in (2); [b] - also reported in (1); [c] - also reported graphically in (2).

Above 1180 °C the author predicts a complete miscibility of liquid Na and Ca. The eutectic point on the Na rich side is 0.025 °C below the melting point of Na.

Thermal analysis experiments [c]:

t/°C	*soly*/mol % Ca
802	90.55
776	89.3
754, 756	86.1
738, 739	82.4
726	80.0

The melting point of Ca was measured to be 848°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Ca-Na alloy was placed in a vertical container fabricated of a decarburized steel. The apparatus was surrounded by a porcelaine tube which was closed on the lower end and plugged on the upper side. Through this plug the supply with pure Ar as well as the thermocouple and the connected stirrer were introduced. The Ni/Cr-Ni thermocouple was covered with pure Ag at its brazing, it was calibrated at the boiling points of water, naphtalene, and sulfur and the melting points of Sb and NaCl. The equipment was heated in an electrical furnace. An auxilary heater and a water cooling at the plug on top of the porcelaine tube prevented the distillation of Na. The cooling curves of several mixtures of the two metals which were primarily heated to 1173 K were recorded .

SOURCE AND PURITY OF MATERIALS:

Ca: twice vacuum distilled.
Na: commercially pure, once distilled.
$CaCl_2$: dehydrated at 1123 K in HCl atmosphere.
NaCl: remelted in a Pt crucible.
Ar: purified from O,N,H_2O.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 5 K for the alloy-salt equilibria.

REFERENCES:

1. Rinck, E. *C.R.Hebd. Acad. Sci.* 1930, *91*, 404.
2. Rinck, E. *C.R.Hebd. Acad. Sci.* 1931, *92*, 1378.

COMPONENTS:	EVALUATOR:
(1) Calcium; Ca; [7440-70-2] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1987

CRITICAL EVALUATION:

Quantitative data on the solubility of Ca in K were not reported in literature, but it is almost certain that liquid K is a less efficient solvent for Ca than is Na. These observations were described by Moissan (1), Muthmann and coworkers (2) and more recently by Klemm and Kunze (3). It seems reasonable to predict solubility values of Ca in K, since they should not be much lower than those determined for Ca in the liquid Na-K eutectic (4) in a similar temperature range; see the corresponding data sheet.
A formation of compounds between Ca and K was not observed (3), and the Ca-K phase diagram seems to be similar to that of the Mg-K system to which a line for the α Ca $\leftrightarrow$ β Ca transition has to be inserted at 716 K.

References

1. Moissan, H. *Compt. Rend.* 1898, *127*, 584; *Bull. Soc.Chim. Fr.* 1899, *21*, 871; *Ann. Chim. Phys.* 1899, *18*, 289.
2. Muthmann, W.; Weiss, L.; Metzger, J. *Liebigs Ann.* 1907, *355*, 137.
3. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chem. Soc., London, 1967, p. 3.
4. Blecherman, S.S.; Schenk, G.F.; Cleary, R.E. *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 48; *Rep. CONF-650411-4*, 1965; *Rep. CNLM-6335*, 1965.

COMPONENTS:	EVALUATOR:
(1) Calcium; Ca; [7440-70-2] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1987

CRITICAL EVALUATION:

The only uncertain information in literature indicated that the solubility of Ca in Rb is very low. A formation of Ca-Rb intermetallics was not observed (1). The solubility seems to be not of a higher degree than that of Ca in Na (see the Ca-Na system). The Ca-Rb phase diagram should be similar to that of the Sr-K system. The melting and boiling points of Rb are given as 312.63 and 961 K, respectively. The melting temperature of Ca is at 1115 K, and the α Ca $\leftrightarrow$ β Ca transformation temperature is at 716 K.

References

1. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chem. Soc., London, 1967, p. 3.

COMPONENTS:	EVALUATOR:
(1) Calcium; Ca; [7440-70-2] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1987

CRITICAL EVALUATION:

Quantitative measurements of the solubility of Ca in Cs were not reported, though the qualitative Ca content in the saturated solution was estimated to be very low. No formation of the Ca-Cs compound was observed by (1). The solubility of Ca in Cs is expected to be lower than that in Na as well as in Rb.
In compatibility tests, four alloys containing small amounts of Ca were equilibrated with Cs. After 48 hours at 673 K the Ca content increased from 0.27 to 1.0 mol %. After a 7000 hours storage at room temperature the Ca content increased from $7 \cdot 10^{-3}$ to 3 mol %. A contamination of Cs by non-metals is not known, though it might be of influence on the Ca concentration in Cs. As (2) is not a solubility determination and further details are not provided, the paper is not compiled.
The phase diagram of the Ca-Cs system should be similar to that of the Sr-K system taking into account the melting and transformation temperatures of the components: Cs at 301.53 K, Ca at 1115 K and α Ca $\leftrightarrow$ β Ca transformation at 716 K.

References

1. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chem. Soc., London, 1967, p. 3.
2. Holley, J.H.; Neff, G.R.; Weiler, F.B.; Winslow, P.M. *Electric Propulsion Development*, E. Stuhlinger, Ed., Academic Press, N.Y., 1963, p. 341.

COMPONENTS:

(1) Calcium; Ca; [7440-70-2]

(2) Potassium-Sodium eutectic;
K68,Na32; [11147-16-3]

ORIGINAL MEASUREMENTS:

Blecherman, S.S.; Schenk, G.F.; Cleary, R.E.

US Atom.Ener.Comm. Rep. CONF-650411, 1965, p. 48-53, or *CONF-650411-4*, 1965; or *Rep. CNLM-6335*, 1965.

VARIABLES:

Temperature: 531-698 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ca in liquid eutectic K-Na was read from the graph of the solubility vs. the reciprocal absolute temperature.

T/K [a]	*soly*/mol % Ca
531	$9.2\cdot10^{-3}$ [b]
632	$4.5\cdot10^{-2}$ [b]
662	$5.8\cdot10^{-2}$
698	$8.2\cdot10^{-2}$, $8.6\cdot10^{-2}$, $9.0\cdot10^{-2}$, $9.4\cdot10^{-2}$ [c], $9.7\cdot10^{-2}$, $1.0\cdot10^{-1}$ [c]; mean value: $9.3\cdot10^{-2}$

[a] - calculated by the compilers [b] - 3 agreeing results [c] - 2 agreeing results

The solubility of Ca in eutectic K68Na32 in the temperature range given above was represented by the equation:

$$\log(soly/\text{mol fraction Ca}) = 0.1487 - 2233(T/\text{K})^{-1}$$

The estimated error is ± 7 %. The equation was tested by the compilers, the results were reasonable and may be classified as tentative. The solubility of Ca in the eutectic alloy was seen to be lower than in pure Na. The decreasing influence of K on the solubility of another metal is in agreement with the general tendency of the solubility of metals in alkali metals.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Details on the experiments were not reported. The laboratory is known to have experience in the determination of solubilities in alkali metals. Probably, the inverted sampler technique with Mo receiver was applied, and the determinations were performed under Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ca: nothing specified.
K-Na eutectic: purified by contacting with Ti sponge for 24 h in a steel container; O content $\leq 2\cdot10^{-3}$ %.
Ar: O and H_2O contents $\leq 3\cdot10^{-4}$ % and $\leq 4\cdot10^{-4}$ %, respectively.

ESTIMATED ERROR:

Solubility: standard deviation ± 7 %.
Temperature: reading procedure ± 5 K.

REFERENCES:

COMPONENTS:

(1) Strontium; Sr; [7440-24-6]

(2) Lithium; Li; [7439-93-2]

EVALUATOR:

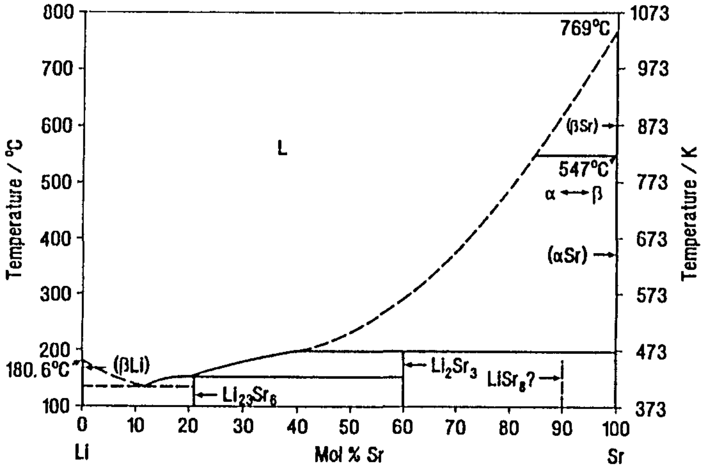
H.U. Borgstedt, Kernforschungszentrum Karlsruhe,
Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw,
Poland
July 1995

CRITICAL EVALUATION:

Wang and coworkers published several papers (1-5) on the Sr-Li system, the content of which was virtually the same. A smoothed phase diagram was presented in (2-4). Thus, only characteristic points of the liquidus might be read out; see the data sheet of (5). The proposed liquidus and relations between the phases in (1-5) were somewhat complex. Bale and Pelton (6) evaluated the system and threw suspicion on the experimental results obtained by (1-5) in the range of 40-100 mol % Sr. Wang and coworkers (1-5) noticed difficulties in approaching the equilibrium for various regions of the system, and Bale and Pelton (6) emphasized a possible contamination of Sr with H, which was likely to induce the differences in the allotropic transformation temperature of Sr. Bale and Pelton (6) proposed a new form of the Sr-Li phase diagram, taking into consideration the generally accepted transformation temperature of Sr and the thermodynamic analysis; see the figure below. Nevertheless, their theoretical study needs experimental confirmation. Hubberstey and Roberts (7) showed that the presence of N in the system may also influence the phase relations. The existence of solid Sr_6Li_{23} and Sr_3Li_2 is well documented in (3-5), while that of Sr_8Li or Sr_7Li (3-5) is doubtful.

Tentative (t) and doubtful (d) values of the solubility of Sr in liquid Li according to (1-5)

T/K	*soly*/mol % Sr
407	12 (t) eutectic
425	20 (t) peritectic
471	40 (t) peritectic
573	61 (d)
673	72 (d)
773	81 (d)
873	88 (d)
973	95 (d)



References

1. Wang, F.E. *Ph.D. thesis*, Syracuse Univ.,N.Y., 1960.
2. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-5691*, 1960, p.1.
3. Wang, F.E.; Kanda, F.A.; King, A.J. *US Atom.Ener.Comm. Rep. TID-15218*, 1961.
4. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-15150*, 1962, p.19.
5. Wang, F.E.; Kanda, F.A.; King, A.J. *J. Phys. Chem.* 1962, *66*, 2138.
6. Bale, C.W.; Pelton, A.D. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met.,Metals Park, 1986, p. 1501; *Bull. Alloy Phase Diagr.* 1989, *10*, 278.
7. Hubberstey, P.; Roberts, P.G. *Liquid Metal Systems*, H.U. Borgstedt, G. Frees, Eds., Plenum 1995, p.331.

COMPONENTS:

(1) Strontium; Sr; [7440-24-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Wang, F.E.; Kanda, F.A.; King, A.J.

J. Phys. Chem. 1962, *66*, 2138-42.

VARIABLES:

Temperature: 407-851 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The phase diagram of the Sr-Li system was presented in graphical form. The liquidus points were obtained from smoothed curves (by the compilers), as experimental points were not given.

t/°C	*soly*/mol % Sr	
134	12.5	eutectic
152	20	peritectic decomp. Sr_6Li_{23}
198	40	peritectic decomp. Sr_3Li_2
496	81	peritectic decomp. Sr_8Li
515	82	
530	83	
584	88.5	congruent melting of Sr_7Li
578	89.5	eutectic

These results were previously reported in (1), (2) (Sr-rich alloys), and (3). The data presented in (4) were slightly different, they might be considered as preliminary.
The melting points of Sr and Li were determined to be 773-774 and 180±1°C, respectively. Allotropic Sr transformations were found at 231 and 623°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

For initial investigations, samples were prepared at every 2 mol % composition interval. Smaller intervals were chosen in the range of 80-100 mol % Sr. The containers used were thin walled Fe crucibles. Appropriate quantities of the components were weighed, heated, and mixed. All operations were carried out in an Ar atmosphere. Thermal analysis of the samples was performed by means of a Chromel/Alumel thermocouple, which had been calibrated against a standard Pt/Pt-Rh(10%) thermocouple certified by the NBS.

SOURCE AND PURITY OF MATERIALS:

Sr: commercial; supplied by King Laboratories Inc.; vacuum distilled at 1023 K; with contents of 0.1 % Ca; no Ba; $\leq$ 0.01 % other impurities.
Li: 99,86 - 99.89 % purity; supplied by Foote Mineral Co.; contaning 0.09-0.10 % Na.
Ar: dried with 4A sieves, Ba chips and Ti turnings at 673-773 K.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: accuracy ± 1 K; reproducibility ± 1 K.

REFERENCES:

1. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom. Ener.Comm. Rep. TID-5691*, 1960, p. 1.
2. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom. Ener.Comm. Rep. TID-15150*, 1962, p. 19.
3. Wang, F.E.; Kanda, F.A.; King, A.J. *US Atom. Ener.Comm. Rep. TID-15218*, 1961.
4. Wang, F.E. *Ph.D. thesis*, Syracuse Univ., N.Y., 1960.

COMPONENTS:	EVALUATOR:
(1) Strontium; Sr; [7440-24-6] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1987

CRITICAL EVALUATION:

Remy and coworkers (1) were the first to report a possible miscibility of liquid Sr and Na in all proportions. The eutectic point in the Sr-Na phase diagram was established at 365.7 K and 3.2 mol % Sr; the liquidus line increased smoothly to the melting point of Sr. However, further numerical data or experimental details of the study were not provided. The paper is, therefore, not compiled.

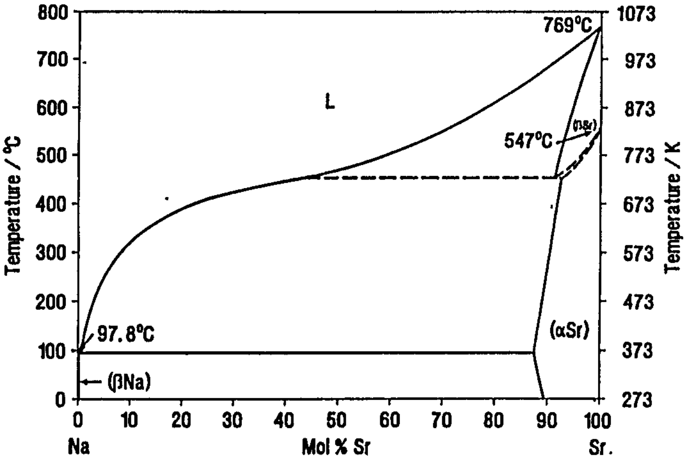
Subsequently the system was investigated intensively by Kanda et al. (2-5). Numerical values of the solubility of Sr in the entire composition range were reported in (5); the results published in other studies were presented in a graphical form. Authors, such as Remy and coworkers (1), did not observe formation of intermetallic Sr-Na, but the liquidus shape indicated occurence of 2 peritectics at 465 and 908 K. The positions of the peritectics were explained by the existence of 3 allotropic forms of Sr saturating liquid Na. Subsequently, Peterson and Colburn (6) detected that for very pure Sr only 2 allotropes exist and the transformation temperature is at 830 K. Pelton (7), therefore, suggested that the Sr used in (2-5) was contaminated by H, which is expected to have drastically changed the thermal arrests of the transformation.

Bussey and coworkers (8) clarified that the solubilities of Sr in liquid Na reported in (2-5) in the concentration range of 0-40 mol % Sr were not correct. The thermal analysis used in (2-5) caused an overestimation of the solubility values of up to 15 mol %, since the thermal arrests at the start of crystallization were not definitive. Resistivity measurements, thermal analyses at a slow cooling rate and filtrate analysis of the saturated solution reported in (8) are regarded as tentative, as they show mutual agreement.

Pelton (7) presented the Sr-Na phase diagram, modified according to the data of (8). He proposed a correction of the experimental values of (2-5) at the Na-rich part of the liquidus, lowering the temperatures by about 10-15 K, while he accepted the part between 42 and 88 mol % Sr presented in (2-5). The corrections of the liquidus at concentrations above 88 mol % Sr based on thermodynamic analysis, seem to be acceptable and might be due to Na losses in the alloy samples by volatization as well as an effect of contaminants. However, this prediction needs further experimental confirmation. The assessed Sr-Na (7) phase diagram is redrawn und presented in the figure. The formation of Sr subnitride is observed, if the system contains N as contaminant.

Tentative (t) and doubtful (d) values of the solubility of Sr in liquid Na

T/K	*soly*/mol % Sr	source
368	0.65 (t) eutectic	(8)
473	3 (t)	(8)
573	8 (t)	(8)
673	22 (t)	(8) interpolated
773	61 (d)	(5)
873	78 (d)	(5)
973	90 (d)	(5) interpolated



References

1. Remy, H.; Wolfrum, G.; Haase, H.W. *Naturwissensch.* 1957, *44*, 534.
2. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-12313*, 1961, p. 15.
3. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-15150*, 1962, p. 10.
4. Kanda, F.A.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-18619*, 1963, p. 1.
5. Roberts, W.O. *US Atom.Ener.Comm. Rep. TID-20639*, 1964; *Ph.D. thesis*, Syracuse Univer., 1964.
6. Peterson, D.T.; Colburn, R.P. *J. Phys. Chem.* 1966, *70*, 468.
7. Pelton, A.D. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Metals Park, 1986, p.1671; *Bull.Alloy Phase Diagr.* 1985, *6*, 43.
8. Bussey, P.R.; Hubberstey, P.; Pulham, R.J. *J. Chem. Soc., Dalton Trans.* 1976, *22*, 2327.
9. Hubberstey, P.; Bussey, P.R. *Liq. Met. Engin. Technol.*, BNES, London 1984, *3*, 143.

COMPONENTS:

(1) Strontium; Sr; [7440-24-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Roberts, W.O.

US Atom.Ener.Comm. Rep. TID-20639, 1964; *Ph.D. Thesis*; Syracuse Univ., N.Y., 1964.

VARIABLES:

Temperature: 368-1042 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Numerical results of the liquidus points are reported to be:

t/°C	*soly*/ mol % Sr	*t*/°C	*soly*/ mol % Sr	*t*/°C	*soly*/ mol % Sr
94.65	1.3 eutectic	330	20.0	650	85.0
95	1.6	383	25.0	680	88.0
105	2.5	398	27.6	710	91.0
114	3.1	410	31.5	719	91.5
130	4.4	425	35.6	728	93.0
150	7.4	446	44.6	745	95.0
171	10.4	503	61.0	756	97.0
192	14.0	548	70.0	766	98.6
247	16.9	604	78.5	769	99.0

The melting points of Sr and Na were determined to be 776±2 and 97.5°C, respectively. A formation of compounds was not observed.
These results were presented in graphical form in (1-3).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The applied crucibles were fabricated of low carbon steel. The crucible and the other parts of the apparatus were cleaned with soap, H_2O, HCl, and acetone. Weighed quantities of Sr and Na were placed in the crucible. The samples were heated to 1073 or even 1123 K, then soaked and vigourously stirred. Thermal analysis of the samples was performed by slow cooling and recording of the corresponding curves. Chromel/Alumel thermocouples were applied, which had been calibrated against a Pt/Pt-Rh(10%) thermocouple certified by the NBS. X-ray analysis supplemented the investigation. All operations were carried out in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Sr: supplied by King Laboratories; vacuum distilled; liquid alloys were insignificantly contaminated after longer contact with the container.
Na: supplied by Baker & Adamson Chemicals. No further purification.
Ar: purified with 5A Linde molecular sieves and heated over Ba chips.

ESTIMATED ERROR:

Solubility: precision ± 0.2 mol % (anticipated).
Temperature: accuracy ± 1 K; reproducibility ± 1 K.

REFERENCES:

1. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom. Ener.Comm. Rep. TID-12313*, 1961, p. 15.
2. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom. Ener. Comm. Rep. TID-15150*, 1962, p. 10.
3. Kanda, F.A.; Keller, D.V. *US Atom.Ener. Comm. Rep. TID-18619*, 1963, p. 1.

COMPONENTS:

(1) Strontium; Sr; [7440-24-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Bussey, P.R.; Hubberstey, P.; Pulham, R.J.

J. Chem. Soc., Dalton Trans. 1976, *22*, 2327-9.

VARIABLES:

Temperature: 369-712 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Points on the Sr-Na liquidus were reported to be:

t/°C	*soly*/mol % Sr	*t*/°C	*soly*/mol % Sr	*t*/°C	*soly*/mol % Sr
97.8	0.10[a]	199	3.08[c]	338	11.3[c]
97.4	0.19[a]	211	3.58[c]	348	12.1[c]
97.1	0.29[a]	223	4.01[c]	351	13.3[c]
95.8	0.53[a]	225	4.07[c]	373	16.4[c]
116	0.80[c]	224	4.13[b]	391	20.0[c]
136	1.03[b]	247	5.15[c]	406	23.4[c]
154	1.56[c]	267	6.42[c]	417	28.1[c]
168	2.06[c]	302	7.95[c]	426	32.4[c]
173	2.28[c]	316	9.25[c]	439	37.9[a,c]

[a] - thermal analysis; [b] - chemical analysis of filtrate; [c] - resistivity measurements.

The melting point of Na was found at 97.8°C. The authors estimated a eutectic point at 94.80°C and 0.65 mol % Sr.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solutions were prepared by weighing in the appropriate quantities of the pure components. Homogeneity of the samples was achieved at 773 K by prolonged circulation of the liquid through a steel resistivity capillary loop by means of an electromagnetic pump. The resistivity of the solutions was determined at 15 K intervals from 773 to 373 K. The temperature at which Sr precipitation commenced was clearly defined by breaks in the curves due to the different temperature dependence of the resistivity of the unsaturated and the saturated solution. Thermal analysis was performed with some samples with cooling rates of 0.006 Ks^{-1}. The solubility of Sr in liquid Na was also analyzed in further determinations after filtration of a sample in a Pyrex-glass tube. The residue on the filter was identified as α-Sr by x-ray diffraction. The filtrate was analyzed for Sr by dissolution in dilute acid followed by precipitation as $SrSO_4$. Experiments were obviously performed under inert gas atmosphere.

SOURCE AND PURITY OF MATERIALS:

Sr: 99.8 % purity; supplied by Koch-Light; surface mechanically cleaned.
Na: supplied by UKAEA, Culcheth; containing $\leq 4 \cdot 10^{-3}$ % Ca; $\leq 1 \cdot 10^{-3}$ % O.

ESTIMATED ERROR:

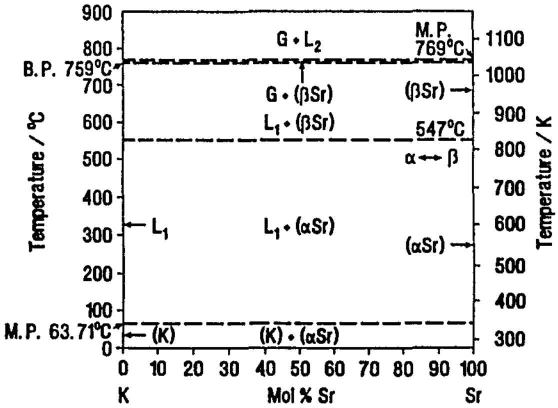
Solubility: nothing specified.
Temperature: precision not better than ± 0.05 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Strontium; Sr; [7440-24-6] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1987

CRITICAL EVALUATION:

According to qualitative observations of Klemm and Kunze (1), Sr is immiscible with K in the solid and liquid state and has a low solubility in liquid K.
Formation of intermetallic compounds was not observed in the Sr-K system. This fact is reflected in the schematic phase diagram of the Sr-K system, as an example.



References

1. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3.

COMPONENTS:	EVALUATOR:
(1) Strontium; Sr; [7440-24-6] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1987

CRITICAL EVALUATION:

According to qualitative observations of Klemm and Kunze (1), Sr and Rb are immiscible in the solid and liquid state and Sr has a low solubility in liquid Rb.
A formation of intermetallic compounds of Sr and Rb was not observed; a phase diagram of the Sr-Rb system should be analogous to the Sr-K system.

References

1. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3.

COMPONENTS:	EVALUATOR:
(1) Strontium; Sr; [7440-24-6] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1987

CRITICAL EVALUATION:

According to qualitative observations of Klemm and Kunze (1), Sr is immiscible with Cs in the solid and liquid state and has a low solubility in liquid Cs.
A formation of intermetallic compounds of Sr and Cs was not observed; a phase diagram analogous to that of the Sr-K system may be expected.

References

1. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3.

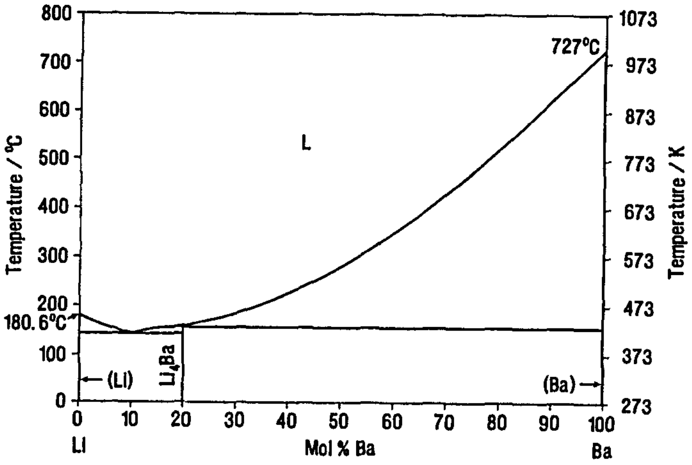
COMPONENTS:	EVALUATOR:
(1) Barium; Ba; [7440-39-3] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1995

CRITICAL EVALUATION:

The only experimental study on the Ba-Li system was published by Keller et al. (1). The liquidus proposed in the phase diagram is acceptable. However, the accuracy of the results was only ± 5 mol % Ba for Ba rich alloys, since the mass % scale used in the figure did not permit to read the data with higher precision.
The thermodynamic analysis of the Ba-Li system performed by Pelton (2) confirmed the liquidus line presented in (1).
The saturated solutions of Ba in liquid Li are in equilibrium with solid Li, $BaLi_4$ or Ba; see the phase diagram based on (2).
Hubberstey and Roberts (3) performed thermal analyses with Ba-Li alloys of various compositions. They reported two liquidus points at 432 and 419 K for 4.75 and 13.2 mol % Ba, respectively. These findings indicate that the declining part of the Li-rich liquidus should be more convex, whereas the rising part after the eutectic should be more flat than in (1). The authors (3) claimed a general confirmation of the liquidus line of the Ba-Li system of (1) and underlined the influence of N present in the system on the phase relations. Due to the lack of experimental details the publication (3) is not compiled.

Tentative (t) and doubtful (d) values of the solubility of Ba in liquid Li

T/K	*soly*/mol % Ba	source
416	10.5 (t) eutectic	(1)
429	18.4 (t) peritectic	(1)
473	31 (t)	(1)
573	54 (d)	(1) interpolation
673	67 (d)	(1) interpolation
773	77 (d)	(1) interpolation
873	85 (d)	(1) interpolation
973	97 (d)	(1) interpolation



References

1. Keller, D.V.; Kanda, F.A.; King, A.J. *J. Phys. Chem.* 1958, *62*, 732.
2. Pelton, A.D. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 415; *Bull. Alloy Phase Diagr.* 1984, *5*, 452.
3. Hubberstey, P.; Roberts, P.G. *Liquid Metal Systems*, H.U. Borgstedt, G. Frees, Eds., Plenum 1995, p.331.

COMPONENTS:

(1) Barium; Ba; [7440-39-3]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Keller, D.V.; Kanda, F.A.; King, A.J.

J. Phys. Chem. 1958, *62*, 732-3.

VARIABLES:

Temperature: 416-907 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Ba-Li system was presented graphically and the experimental points were read-off and calculated into mol % by the compilers.

t/°C	*soly*/mol % Ba	t/°C	*soly*/mol % Ba	t/°C	*soly*/mol % Ba
180	0.08	143	10.5[b]	170	27
180	0.26	147	11.5	202	31
179	0.55	151	13.2	254	44
175	1.2	154	16.4	332	60
169	2.1	155	18.1	482	75
165	3.3	156	18.4[a]	543	79
157	4.8	157	20	634	86
153	7.0	158	22		

[a] - peritectic decomposition of $BaLi_4$; [b] - eutectic given in numerical form.

The melting points of Li and Ba were found at 180.5 and 725±1°C respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Alloy samples of approximately constant volume (10 cm^3) were made up by weighing appropriate amounts of the metals (within ± 0.0001 g in an Ar atmosphere). The applied crucibles and thermocouple shields were fabricated of low-carbon steel. Normal and differential cooling curves were obtained at cooling rates of 0.7 or 2.5 K per minute. All thermal breaks were reproduced at least three times. The Chromel/Alumel thermocouples used in this study were frequently calibrated against a NBS certified Pt/Pt-Rh(10%) thermocouple. All operations were performed in an Ar atmosphere. The data obtained by thermal analysis were supplemented by an additional x-ray study.

SOURCE AND PURITY OF MATERIALS:

Ba: 99.9 % pure, supplied by King laboratories; Sr free; vacuum distilled at 850°C; containing $\leq$ 0.1 % Ca.
Li: 99,86 - 99.90 % purity, supplied by Foote Mineral Co.; containing $\leq$ 0.1 % Na.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: chemical analysis after fusion agreed within ± 0.2 % of initial composition; evaluation of the diagram up to ± 5 mol % Ba (in the Ba rich region).
Temperature: certainty ± 1 K; reading out procedure ± 3 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Barium; Ba; [7440-39-3] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1987

CRITICAL EVALUATION:

An approach to the evaluation of the Ba-Na phase diagram was primarily based on very different solubility data determined independently by Gould (1) and Miller (2). Gould reported a gradual increase of the solubility of Ba in liquid Na from 0.17 to 0.66 mol % Ba at 530 to 980 K. Miller presented a smooth liquidus of the Ba-Na system with eutectics at 3.3 and 8.5 mol % Ba at 358 and 354 K, congruently melting $BaNa_{24}$ at 3.7 mol % Ba at 362 K and peritectics for 41 and 54 mol % Ba at 476 and 533 K. Since experimental details were not specified, the work (2) is not compiled. In (1) the metals contained traces of less than 0.004 % O and the Ba applied was of 99 % purity. It is likely that the samples used for dissolution tests were not properly equilibrated. An unknown impurity may have prevented the dissolution of Ba to the saturated level, thus causing results which were 10 times too low. The results of (1) are, therefore, rejected and not compiled. Thus the solubility equations proposed in (1) and (3) are not correct. Davis and Draycott (4) reported a solubility of 0.093 mol % Ba in liquid Na at 423 K, but did not mention the source of their data. The value is strongly understated.

Studies on the Ba-Na system by Remy and coworkers (5,6) did not confirm any of the former liquidus data. In the course of the liquidus proposed by (5,6), eutectics were observed for 5.6, 12, 26 and 70.5 mol % Ba at 358, 349, 330.4 and 453 K, respectively, and maxima for the congruent melting of $BaNa_{12}$, $BaNa_6$ and BaNa at 369, 370 and 783 K, respectively. The observed melting point of Ba (983 K) indicated the presence of impurities in this metal. Remy and coworkers only obtained acceptable results by differential thermal analysis, and not by direct thermal analysis. In comparison with the subsequent studies only the region of dilute Ba solutions up to the first eutectic seems to be acceptable. Kanda and coworkers (7-12) performed a number of experiments on the Ba-Na system by means of thermal analyses, density measurements and X-ray studies. The first paper (7) might be regarded as a preliminary study and is, therefore, not compiled. The results gained at a Ba content between 45 and 60 mol % were understated more than 10 %. Subsequent studies (8-10) presented the same results as conclusive (12), but the data were only supplied in a graphic form. The authors reported that alloys with a Ba content below 20 mol % strongly tend to segregate.

As the results of (1),(2),(6), and (11) showed fundamental discrepancies, it could be assumed that the intention of subsequent work would be to give preference to one of them. However, the precise resistivity measurements of the Ba-Na liquidus up to 43.6 mol % Ba (at 528 K) performed by Addison and coworkers (13,14) confirm the data of (7-12) on one hand, but also seem to be inconsistent. (13) provides numerical values of the liquidus points, whereas (14) presents extended results given exclusively in figures. The composition of the samples in (13) and (14) is in agreement, but the temperatures show discrepancies. The authors did not provide an explanation. Though the compilers regard the values of (14) as tentative, those of (13) are also listed in the data sheet. The selection of the tentative solubility results is based on the results of (14) obtained at compositions below 28 % mol Ba and on those of (11) for above this limit.

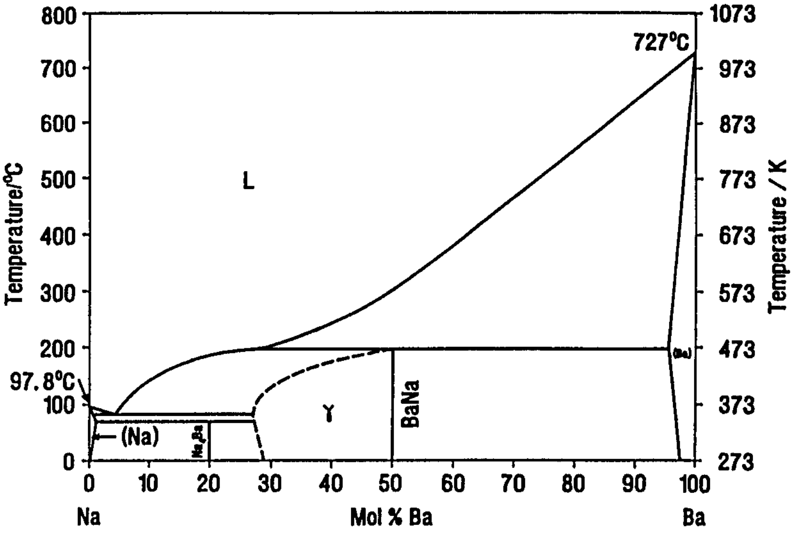
Saturated solutions of Ba in Na are in equilibrium with solid Na saturated with Ba, BaNa and Ba saturated with Na. Occurence of a γ-phase (BaNa saturated with Na), as stated in (8-12), was not detected in (14). The formation of other compounds reported in (1-3, 5-7) could not be confirmed in more recent studies. An assessed phase diagram of the Ba - Na system was presented in (15) and is redrawn in the figure.

The presence of N or C or both of them as contaminants in the Na-Ba system leads to the formation of nitride, carbide or cyanamide of Ba which may influence the solubility equilibria (16,17).

Tentative values of the solubility of Ba in liquid Na

T/K	*soly*/mol % Ba	source
356	4.5 eutectic	(14) extrapolated
373	6	(14) interpolated
470	28 peritectic	(14)
573	49	(11) interpolated
673	62	(11) interpolated
773	73	(11) interpolated
873	85	(11)
973	97	(11)

COMPONENTS:	EVALUATOR:
(1) Barium; Ba; [7440-39-3] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1987



References

1. Gould, J. *US Atom.Ener.Comm. Rep. KAPL-1398*, 1955.
2. Miller, R.R. *Naval Research Laboratory*, 1955; private communication to Adams, R.M.; Sittig, M. *Liquid Metals Handbook*, C.B. Jackson, Ed., US Atom.Ener.Comm. and NAVY, Washington, 1955, p.17.
3. ibid. p. 16.
4. Davis, M.; Draycott, A. *Peaceful Uses of Atomic Energy*,U.N., N.Y., 1958, *7*, 94.
5. Remy, H.; Wolfrum, G.; Haase, H.W. *Naturwissensch.* 1957, *44*, 534.
6. Remy, H.; Wolfrum, G.; Haase, H.W. *Schweiz. Archiv* 1960, *26*, 5.
7. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-5691*, 1960, p. 46.
8. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-12313*, 1961, p. 7.
9. Kanda, F.A.; King, A.J.; Keller; D.V. *US Atom.Ener.Comm. Rep. TID-15150*, 1962, p. 1.
10. Kanda, F.A.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-18619*, 1963, p. 14.
11. Stevens, R.M. *US Atom.Ener.Comm. Rep. TID-20637*, 1964; *Ph.D. thesis*, Syracuse Univ., 1964.
12. Kanda, F.A.; Stevens, R.M.; Keller, D.V. *J. Phys. Chem.* 1965, *69*, 3867.
13. Addison, C.C.; Creffield, G.K.; Hubberstey, P.; Pulham, R.J. *J. Chem. Soc.A* 1971, 1393.
14. Addison, C.C.; Creffield, G.K.; Hubberstey, P.; Pulham, R.J. *J. Chem. Soc.A* 1971, 2688.
15. Pelton, A.D. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 419; *Bull. Alloy Phase Diagr.* 1985, *6*, 26.
16. Hubberstey, P. *Liquid Alkali Metals*, BNES, London, 1973, 15.
17. Pulham, R.J.; Hubberstey, P. *J. Nucl. Mater.* 1983, *115*, 289.

COMPONENTS:

(1) Barium; Ba; [7440-39-3]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Remy, H.; Wolfrum, G.; Haase, H.W.

Schweiz. Archiv 1960, *26*, 5-9.

VARIABLES:

Temperature: 358-848 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Ba-Na system was determined to be:

t/°C	*soly*/mol % Ba	t/°C	*soly*/mol % Ba	t/°C	*soly*/mol % Ba
575	88.2	97	27.4	94	8.7
330	76.4	95	21.2	96	7.9
290	64.2	96.5	16.2	93	6.9
339	62.2	97	14.2	93.5	4.4
500	51.2	92	13.7	97	2.2
475	31.7	85.5	12.3		
170	29.2	90	11.1		

The eutectic points are 70.5 mol % Ba at 180°C, 26 mol % Ba at 57.2 °C, 12 mol % Ba at 76°C, and 5.6 mol % Ba at 85°C, as estimated by the authors. The compounds $BaNa_{12}$, $BaNa_6$ and BaNa have congruent melting points at 96, 97 and 510°C, the pure metals at 710 and 98°C, respectively. The proposed phase diagram was graphically presented in (1), the melting points of the $Na_{12}Ba$-Na_6Ba and Na_6Ba-NaBa eutectics were reported to be 77 and 58°C, respectively. The melting point of Ba indicates an occurrence of impurities, the results obtained at concentrations higher than 7 mol % Ba should be regarded as erroneous. The normal cooling curves show overstated results of the Ba solubility; they are therefore omitted.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Normal and differential thermoanalyses of Ba-Na alloy samples were performed. The apparatus was essentially fabricated of V2A steel, tightened with asbestos-Cu packing. The crucible furnace was made of Pythagoras glass. The Ba-Na sample in the crucible was stirred with a motor. Cooling curves were recorded by means of a Fe/Constantan thermoelement calibrated at the melting points of NaCl, Sb, Zn, Pb, Sn, and the boiling point of H_2O. The measurements were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ba: vacuum distilled from steel crucible, placed in Ni protective tube, condensed on Cu cold finger. $\leq$ 99.6 % purity with $5 \cdot 10^{-3}$ % N; Ba determined as $BaSO_4$ and N as alkalinity of NH_3 after decomposition of a sample with H_2O.
Na: supplied by Merck, high vacuum distilled in apparatus of Jena-glass.
Ar: nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.2 K (<373 K), ± 1 K (373-573 K), ± 3 K (>573 K).

REFERENCES:

1. Remy, H.; Wolfrum, G.; Haase, H.W. *Naturwissensch.* 1957, *44*, 534.

COMPONENTS:

(1) Barium; Ba; [7440-39-3]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Stevens, R.M.

US Atom.Ener.Comm. Rep. TID-20637, 1964; *Ph.D. thesis*, Syracuse Univ., N.Y., 1964.

VARIABLES:

Temperature: 355-978 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Numerical values of points on the Ba-Na liquidus were reported to be:

t/°C	*soly*/mol % Ba	*t*/°C	*soly*/mol % Ba	*t*/°C	*soly*/mol % Ba
97	0.5	131	14.3	241	40.1[a]
96	1.0	138	15.6	271	45.0[a]
94	1.6	152	17.0	315	50.0[a]
92	1.8	162	18.0	350	55.0
89	3.8	176	20.0	390	60.0[a]
85	5.1	192	22.0	424	65.0
82	5.5	199	23.7	462	70.0[a]
87	6.1[a]	200	25.0[a]	484	72.4
89	6.7	201	27.0[a]	510	76.1
94	8.3	200	28.1[a]	536	78.1
102	9.6	203	30.1[a], 30.4[a]	596	84.4
105	10.2	204	31.2[a]	636	90.0
111	11.0[a]	209	33.4[a]	674	95.0
123	12.3	211	35.3[a]	705	97.0
126	13.4	232	37.2[a]		

[a] - from density measurements.

These results were reported in graphical form in (1-4).
The melting points of Na and Ba were found at 97.5 and 729°C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

All experiments were performed in a dry Ar glove box. Weighed quantities of Ba and Na were placed in a stainless steel crucible for thermal analysis. The crucible was rinsed with CCl_4, diluted HCl, H_2O, and acetone. Chromel/Alumel thermocouples were applied which were calibrated against a Pt/Pt-Rh(10 %) thermocouple certified by the NBS and tested at the melting points of Na and Mg. Normal and differential cooling curves were recorded. The charges were heated to 1023 K, soaked, vigourously stirred and afterwards cooled. The composition of some samples was analyzed. Ba was determined as $BaSO_4$, and Na was estimated from the mass balance. Some of the determinations were performed in the high temperature recording densitometer.

Very contrasting inflections on the temperature versus density curves were observed, when crossing the liquidus. The investigation was supplemented by an x-ray study.

SOURCE AND PURITY OF MATERIALS:

Ba: supplied by King Laboratories Inc., vacuum distilled; impurities are: 0.07 % Sr, 0.03 % Ca, 0.01 % Na, 0.005 % Mg, K, 0.001 % Li; $\leq$ 0.01 % Si, Al, Ti, Fe, Cu, Mn, Sn, Ag; not detected: Ni, V, Bi, Zr, Mo, Co, Pb, Sb, As, B, Zn, W, Cd.
Na: 99.9+ % purity; supplied by Baker & Adamson Chemicals.
Ar: purified by blowing through 5A Linde molecular sieves, and through Ti and Ba chips at 673 K.

ESTIMATED ERROR:

Solubility: agreement of analysis and preparation of a sample ±1%.
Temperature: reproducibility better than ±2 K.

REFERENCES:

1. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-12313*, 1961, p. 7.
2. Kanda, F.A.; King, A.J.; Keller, D.V. *US Atom.Ener.Comm. Rep. TID-15150*, 1962, p. 1.
3. Kanda, F.A.; Keller, D.V. *US Atom.Ener. Comm. Rep. TID-18619*, 1963, p. 14.
4. Kanda, F.A., Stevens, R.M.; Keller, D.V. *J. Phys. Chem.* 1965, *69*, 3867.

COMPONENTS:

(1) Barium; Ba; [7440-39-3]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Addison, C.C.; Creffield, G.K.; Hubberstey, P.; Pulham; R.J.
J. Chem. Soc., A. 1971, 2688-2691.

VARIABLES:

Temperature: 358-528 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ba in liquid Na was presented in graphical form. A significant part of the results was reported as numerical data in paper (1), however, the discrepancies in these publications were remarkable and were not explained by the authors. The graph is regarded as tentative.

t/°C	$soly$/mol % Ba	t/°C	$soly$/mol % Ba
98	0,13	85, 84[a]	4.59
98, 97[a]	0.36	84[a]	4.6[a]
97[a]	0.75[a]	88[a]	4.89[a]
98, 96[a]	0.78	86, 88[a]	5.04
96, 94[a]	1.21	88, 96[a]	5.80
94, 92[a]	2.07	90, 114[a]	6.89
91[a]	2.2[a]	92, 122[a]	7.24
88[a]	2.5[a]	100	9.75
92, 88[a]	2.72	120	12.62
90, 85[a]	3.59	192[a]	23.7[a]
86[a]	3.7[a]	198[a]	28.43
85[a]	3.8[a]	208[a]	30.55
85[a]	4.3[a]	255[a]	43.57

[a] - taken from the graphs.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus, consisting essentially of a steel vessel, carrying a capillary loop through which the liquid alloy was circulated by means of an electromagnetic pump. The solution of Ba in Na was prepared in a cell at temperatures high enough to ensure a complete dissolution of Ba. The homogeneous solution was slowly cooled (0.2 and 0.5 K per minute at about 370 and 470 K respectively) and changes in the resistance with the temperature were determined as the solid phases separated. The temperature was measured by means of thermocouples, which had been checked against NPL thermometers. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ba: 99.99% purity (according to an analysis); supplied by Koch-Light Ltd..
Na: probably supplied by UKAEA, Culcheth (2); containing $\leq 4 \cdot 10^{-3}$ % Ca; $\leq 1 \cdot 10^{-3}$ % O.
Ar: 99.99 % purity; probably from Air Products Ltd.; further dried by passing through molecular sieves (2).

ESTIMATED ERROR:

Solubility: nothing specified; ± 1 mol %, as read-off the diagram.
Temperature: nothing specified; ± 1 K, as read-off the diagram .

REFERENCES:

1. Addison, C.C.; Creffield, G.K.; Hubberstey, P.; Pulham, R.J. *J. Chem. Soc., A.* 1971, 1393.
2. Addison, C.C.; Creffield, G.K.; Hubberstey, P.; Pulham; R.J. *J. Chem. Soc., A.* 1969, 1482.

COMPONENTS:	EVALUATOR:
(1) Barium; Ba; [7440-39-3] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1990

CRITICAL EVALUATION:

According to Klemm and Kunze (1) the Ba-K system is immiscible in the liquid and solid state. The authors report a very low solubility of Ba in liquid K, and deny formation of intermetallics. Brewer (2), however, supposed a considerable mutual liquid solubility of Ba-K in the range close to the boiling point of K. A phase diagram of the Ba-K system has not been reported so far. It is likely of the same type as of the Sr-K system.

References

1. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3.
2. Brewer, L. *US Atom.Ener.Comm. Rep. UCRL-10701*, 1963, p. 26.

COMPONENTS:	EVALUATOR:
(1) Barium; Ba; [7440-39-3] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1990

CRITICAL EVALUATION:

According to qualitative observations of Klemm and Kunze (1) Ba and Rb are immiscible in the solid and liquid state and Ba has a low solubility in liquid Rb.
Formation of intermetallic compounds of Ba and Rb was not observed; a phase diagram of the Ba-Rb system should be analogous to that of the Sr-K system.

References

1. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3.

COMPONENTS:	EVALUATOR:
(1) Barium; Ba; [7440-39-3] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1990

CRITICAL EVALUATION:

According to qualitative observations of Klemm and Kunze (1) Ba is immiscible with Cs in the solid and liquid state and has a low solubility in liquid Cs.
A formation of intermetallic compounds of Ba and Cs was not observed; a phase diagram analogous to that of the Sr-K system may be expected.

References

1. Klemm, W.; Kunze, D. *The Alkali Metals*, The Chemical Society, London, 1967, p. 3.

COMPONENTS:	EVALUATOR:
(1) Radium; Ra; [7440-14-4] (2) Lithium; Li; [7439-93-2] or Sodium; Na; [7440-23-5] or Potassium; K; [7440-09-7] or Rubidium; Rb; [7440-17-7] or Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1990

CRITICAL EVALUATION:

Experimental data on the solubility of Ra in liquid alkali metals are not available. Due to its similarity to Ca, Sr, and Ba, formation of a moderately stable compound with a peritectic decomposition and a eutectic between pure Li and a compound might be predicted for the Ra-Li system. This might also be valid for the Ra-Na system. A low solid and liquid solubility of Ra is expected in the other alkali metal systems.
Phase diagrams for the systems of Ra with the alkali metals are not available.

COMPONENTS:	EVALUATOR:
(1) Radium; Ra; [7440-14-4] (2) Lithium; Li; [7439-93-2] or Sodium; Na; [7440-23-5] or Potassium; K; [7440-09-7] or Rubidium; Rb; [7440-17-7] or Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1990

CRITICAL EVALUATION:

Experimental data on the solubility of Ra in liquid alkali metals are not available. Due to its similarity to Ca, Sr, and Ba, a formation of a moderately stable compound with a peritectic decomposition and a eutectic between pure Li and a compound might be predicted for the Ra-Li system. This might also be valid for the Ra-Na system. A low solid and liquid solubility is expected for the other alkali metal systems.
Phase diagrams for the systems of Ra with the alkali metals are not available.

References

COMPONENTS:

(1) Scandium; Sc; [7440-20-2]
or Yttrium; Y; [7440-65-5]
or Lanthanum; La; [7439-91-0]
or Cerium; Ce; [7440-45-1]
or Praseodymium; Pr; [7440-10-0]
or Neodymium; Nd; [7440-00-8]
or Prometium; Pm; [7440-12-2]
or Samarium; Sm; [7440-19-9]
or Europium; Eu; [7440-53-1]
or Gadolinium; Gd; [7440-54-2]
or Terbium; Tb; [7440-27-9]
or Dysprosium; Dy; [7429-91-6]
or Holmium; Ho; [7440-60-0]
or Erbium; Er; [7440-52-0]
or Thulium; Tm; [7440-30-4]
or Ytterbium; Yb; [7440-64-4]
or Lutetium; Lu; [7439-94-3]
or Actinum; Ac; [7440-34-8]
or Thorium; Th; [7440-29-1]
or Protactinium; Pa; [7440-13-3]
or Uranium; U; [7440-61-1]
or Neptunium; Np; [7439-99-8]
or Plutonium; Pu; [7440-07-5]

(2) Lithium; Li; [7439-93-2]
or Sodium; Na; [7440-23-5]
or Potassium; K; [7440-09-7]
or Rubidium; Rb; [7440-17-7]
or Cesium; Cs; [7440-46-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
May 1987

CRITICAL EVALUATION:

Out of the more than 100 binary combinations of the solute metals (Sc, Y, Ln, and An) in liquid alkali metals listed above, only about 20 systems were qualitatively and very few quantitatively investigated. Specific information in these systems is provided on the subsequent pages. As the physico-chemical properties of most of the solute elements are similar, the principal behaviour of the intermetallic systems can be predicted.

In the temperature range of the liquid state of the solvents the solubility of the solute seems to be below 10^{-3} mol %, and is expected to decrease gradually from Li to Cs. A colloid-like solution with finely dispersed and solute particles might easily be formed, thus causing an overestimation of the results.

It should be borne in mind that the solute metals show higher affinity to O than the solvents. Since very pure liquid alkali metals contain traces of O, even very pure solute metals are covered with oxide films. In fact, the solubility of a solute oxide or oxygen conatining compound is measured rather than that of a metallic solute, the corresponding values of the former being higher than the latter.

As an exception, the solubility behaviour of Eu, Yb, and may be Sm is more similar to that of the alkaline earth metals. Therefore, comparatively higher solubility data might be expected for these elements.

1 COMPONENTS:

(1) Scandium; Sc; [7440-20-2]

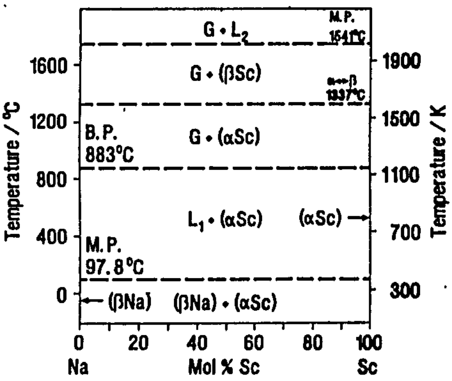
(2) Sodium; Na; [7440-23-5]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
June 1991

CRITICAL EVALUATION:

Aleksandrov and Dalakova (1) reported that no dissolution of Sc in liquid Na was detected by means of spectral analysis after equilibration of solid Sc with liquid Na at temperatures of 973-1023 K for 1 hour. The detection limit of the analytical method was not specified.
A predicted phase diagram of the Sc-Na system is shown in the figure.



References

1. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Scandium; Sc; [7440-20-2]

(2) Potassium; K; [7440-09-7]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
June 1991

CRITICAL EVALUATION:

Aleksandrov and Dalakova (1) did not observe any dissolution of Sc in liquid K by means of spectral analysis after equilibration of solid Sc with liquid K at temperatures of 873-923 K for 1 hour. The detection limit of the analytical method was not reported.
A phase diagram of the Sc-K system should be similar to that of the Sc-Na system; the boiling point of K is 1032 K.

References

1. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Yttrium; Y; [7440-65-5] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1987

CRITICAL EVALUATION:

Numerical results of the solubility determination of Y in liquid Li have not been published. However, it might be assumed that the solubility values for this system are rather low (1), as the corrosion resistance of Y in Li at 1089 K in a static system is good.

Cleary et al. (2) observed some dissolution of Y in Li in preliminary experiments performed at 1573 K. They estimated that the Y solubility was slightly higher than that of Zr at the same temperature. According to qualitative observations by (3), Y disintegrated in Li after 7 days at 1473 K, forming a dark-grey crust.

A Y-Li phase diagram can be expected to be similar to that of the Sc-Na system; the miscibility gap which occurs in the Sc-Na system might be less extended in the Y-Li system due to the indicated measurable solubility of Y in Li at higher temperatures. The melting point of Y is at 1795 K, a α Y $\leftrightarrow$ β Y transformation is located at 1715 K.

References

1. Hoffman, E.E. *US Atom.Ener.Comm. Rep. ORNL-2924*, 1960.
2. Cleary, R.E.; Blecherman, S.S.; Corliss, J.E. *US Atom.Ener.Comm. Rep. TIM-850*, 1965.
3. Adams, R.M.; Glassner, A. (coordinators) *US Atom.Ener.Comm. Rep. ANL-7245*, 1966, p. 36.

COMPONENTS:	EVALUATOR:
(1) Yttrium; Y; [7440-65-5] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1991

CRITICAL EVALUATION:

Aleksandrov and Dalakova (1) reported that no dissolution of Y in liquid Na was detected by means of spectral analysis after immersion of solid Y in liquid Na at temperatures of 973-1023 K for 1 hour. They used a spectral analytical method with unspecified detection limit.

A phase diagram of the Y-Na system should be similar to that shown for the Sc-Na system. The melting point of Y is at 1795 K, a α Y $\leftrightarrow$ β Y transformation is located at 1715 K.

References

1. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Yttrium; Y; [7440-65-5] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1991

CRITICAL EVALUATION:

Aleksandrov and Dalakova (1) did not observe any dissolution of Y in liquid K by means of spectral analysis after equilibration of solid Y with liquid K at temperatures of 873-923 K for 1 hour. The detection limit of the analytical method was not specified.

A phase diagram of the Y-K system should be similar to that shown for the Sc-Na system. Differences of the boiling and melting points and the allotropic transformation temperature of Y have to be taken into account.

References

1. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Lanthanum; La; [7439-91-0]

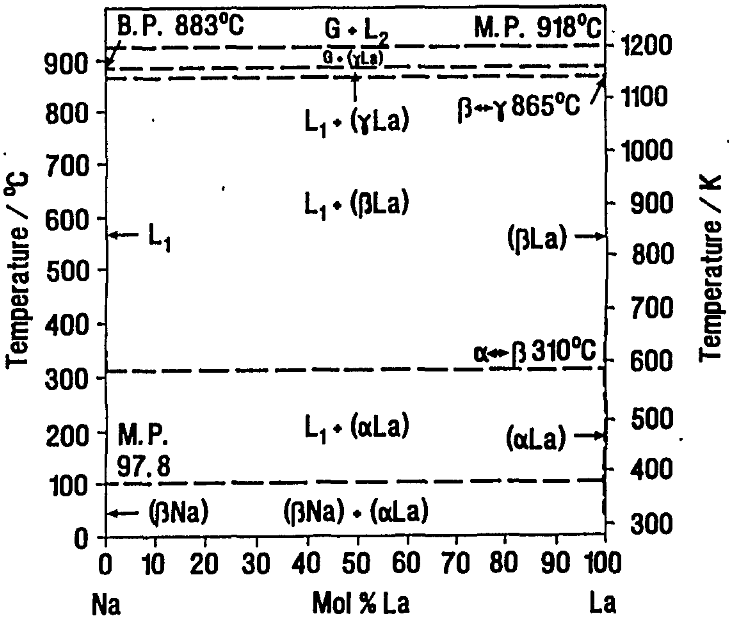
(2) Sodium; Na; [7440-23-5]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
May 1987

CRITICAL EVALUATION:

Mässenhausen's efforts (1) to establish the La-Na phase diagram by means of thermal analysis merely yielded qualitative information. The melting point of La indicated a contamination of the sample. Nevertheless, a pronounced miscibility gap of these liquid metals is obvious. La_2O_3 is formed in the system, if it is contaminated with O.
A speculative La-Na phase diagram is shown in the figure.



References

1. Mässenhausen, W. *Z. Metallk.* 1952, *43*, 53.

COMPONENTS:

(1) Lanthanum; La; [7439-91-0]

(2) Potassium; K; [7440-09-7]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
June 1987

CRITICAL EVALUATION:

Quantitative results of the La-K system were not available. Technically purified La and K were applied for thermal analysis experiments performed by Mässenhausen (1). The author claimed the occurance of a eutectic, though the metals showed no affinity and were rather immiscible.
A speculative phase diagram of the La-K system should be similar to that of the La-Na system, the boiling point of K is at 1032 K.

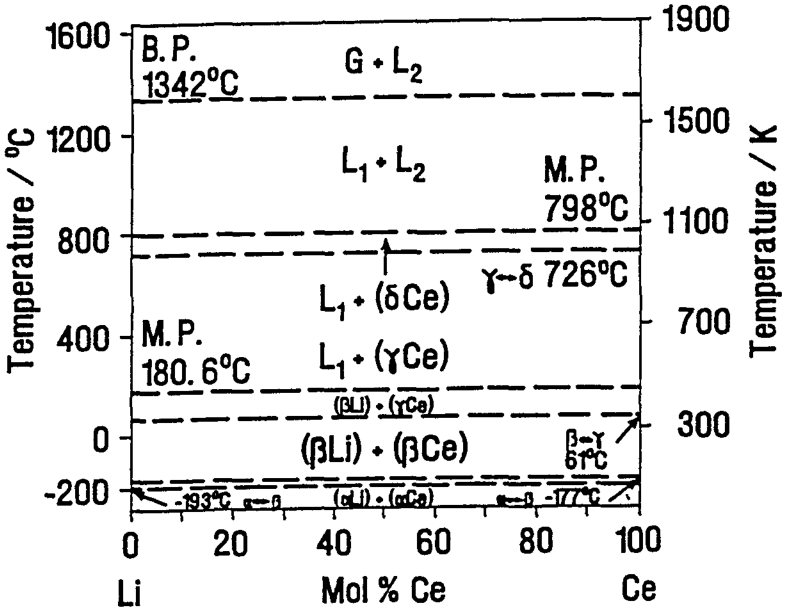
References

1. Mässenhausen, W. *Z. Metallk.* 1952, *43*, 53.

COMPONENTS:	EVALUATOR:
(1) Cerium; Ce; [7440-45-1] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1990

CRITICAL EVALUATION:

No solubility determinations of Ce in liquid Li were reported in the literature. Pavlyuk et al. (1) observed complete immiscibility of the two metals in the solid state, and a tendency to immiscibility in the liquid state is expected. Only a small solubility of Ce in liquid Li has also to be expected due to the work of Barker and Alexander (1). However, it seems that the Ce-Li system is interfered by the formation of Li_2CeN_2 and Ce_2N_2O in this system, if Li might be contaminated with traces of N and O.
A schematic phase diagram on the basis of this information is shown in the figure.



References

1. Pavlyuk, V.V.; Recharskii, V.K.; Bodak, O.L. *Dopov.Akad.Nauk Ukr.RSR, Ser.B* 1989, no. 2, 50.
2. Barker, M.G.; Alexander, I.C. *J. Chem. Soc., Dalton Trans.*, 1982, 2175.

COMPONENTS: (1) Cerium; Ce; [7440-45-1] (2) Sodium; Na; [7440-23-5]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1993

CRITICAL EVALUATION:

The solubility of Ce in liquid Na was determined independently in two laboratories. Lamprecht and Crowther (1,2) performed experiments between 403 and 723 K. They observed a slight decrease of the solubility with increasing temperature from $1.4 \cdot 10^{-6}$ to $3 \cdot 10^{-7}$ mol % Ce. In another test which was carried out at 973 K (3) the solubility limit was specified at $1 \cdot 10^{-4}$ mol % Ce, but not much below this order of magnitude. The equilibrium concentration of Ce in Na equilibrated with Ce-Co and Ce-Co-Pu alloys was found to be at the same level, though Ce forms intermetallic compounds with Co (4) and should be present in the alloy with significantly reduced activity. The gap between the results of (1,2) and (3) may be explained by the fact that materials of different purity were used. It cannot be excluded that this may influence the specific behaviour of this system. As concluded from a comparison of the Gibbs energies of formation of Ce_2O_3 and Na_2O, the reference solute in the system is probably Ce_2O_3, and not metallic Ce, which is less soluble than its oxide. This might explain the decrease of the solubility with increasing temperatures. Therefore, data could be considered as tentative.
Mignanelli et al. (5) observed a formation of $NaCeO_2$ in the temperature range of 400-800°C, if the Ce-Na system was contaminated with O. Aitken et al. (6) announced measurements of the solubility of CeO_2 in liquid Na, the results were obviously not published anywhere.
A schematic Ce-Na phase diagram should be analogous to that of the Ce-Li system, with the exception of the different melting point of the alkali metal.

References

1. Lamprecht, G.J.; *Ph.D. thesis*, Univer. of South Africa, Pretoria, 1966.
2. Lamprecht, G.J.; Crowther, P. *Trans. AIME* 1968, *242*, 2169.
3. Anonymous, *US Atom. Ener. Comm. Rep. LA-3524-MS*, 1966, p. 49.
4. Hansen, M.; Anderko, K. *Constitution of Binary Alloys*, McGraw-Hill, N.Y., 1958, p. 449.
5. Mignanelli, M.A.; Potter, P.E.; Barker, M.G. *J.Nucl.Mater.* 1981, *97*, 213.
6. Aitken, E.A.; Adamson, M.G.; Evans, S.K. *US Atom.Ener.Comm. Rep. GEAP-12489*, 1974.

COMPONENTS:

(1) Cerium; Ce; [7440-45-1]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Lamprecht, G.J.; Crowther, P.

Trans. AIME 1968, *242*, 2169-2171.

VARIABLES:

Temperature: 403-723 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ce in Na was reported in the figure; and the numerical results were reported in (1).

t/°C	*soly*/mass % Ce	*soly*/mol % Ce [a]
130[b]	$8.5 \cdot 10^{-6}$	$1.4 \cdot 10^{-6}$
149[c]	$7.8 \cdot 10^{-6}$	$1.3 \cdot 10^{-6}$
188[b]	$6.0 \cdot 10^{-6}$	$9.8 \cdot 10^{-7}$
208[c]	$4.6 \cdot 10^{-6}$	$7.5 \cdot 10^{-7}$
239[b]	$4.1 \cdot 10^{-6}$	$6.7 \cdot 10^{-7}$
189[c]	$5.0 \cdot 10^{-6}$	$9.0 \cdot 10^{-7}$
164[b]	$7.2 \cdot 10^{-6}$	$1.2 \cdot 10^{-6}$
135[b]	$8.4 \cdot 10^{-6}$	$1.4 \cdot 10^{-6}$
300[b]	$2.9 \cdot 10^{-6}$	$4.5 \cdot 10^{-7}$
380[c]	$2.1 \cdot 10^{-6}$	$3.4 \cdot 10^{-7}$
450[b]	$1.7 \cdot 10^{-6}$	$2.8 \cdot 10^{-7}$

[a] as calculated by the compilers [b] heating sequence [c] cooling sequence

A formation of an intermetallic compound was not detected in this system. The results were fitted to the solubility equation:

$$\log (soly/\text{mol \% Ce}) = -7.467 + 659 \cdot (T/\text{K})^{-1} \quad \text{std. dev.} \pm 0.017$$

The equation was tested by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A detailed description of the solubility apparatus was given in (2). Purified He was used as a cover gas in all experiments. The solubility apparatus was constructed in Pyrex glass for solubility determinations up to 250°C. To obtain a uniform amount of the required specific activity, Ce was doped with ^{141}Ce by melting together in an inert atmosphere. The remolten Ce was placed in one reaction cell, and Na in a twin one. The cells were interconnected by capillary tubing. Na could be transferred between the cells by means of vacuum or helium pressure. The metals remained in contact until there was no increase in the measured activity of Na, which had been transferred to its primary cell for these measurements. By determining the increase or decrease in specific activity of the liquid Na, the variation of solubility with temperature was followed.

SOURCE AND PURITY OF MATERIALS:

Ce: spectroscopically pure; supplied by Johnson Matthey Co.
Na: supplied by Merck; containing Cl^-, SO_4^{2-}, 0.002 % heavy metals; 0.001 % PO_4^{3-}, Fe; 0.005 % N, Ca; 0.01 % K; Na was filtrated through a 5 m filter, O content being then $1.1 \cdot 10^{-3}$ % (2).
He: purified by passing over molecular sieves, and an activated charcoal trap cooled to liquid N temperature. All traces of O, N, H_2O, CO_2 and CH_4 were removed this way (2).

ESTIMATED ERROR:

Solubility: nothing specified; precision of few % (by the compilers).
Temperature: nothing specified.

REFERENCES:

1. Lamprecht, G.J.; *Ph.D. thesis*, Univer. of South Africa, Pretoria, 1966.
2. Lamprecht, G.J.; Crowther, P.; Kemp, D.M. *J. Phys. Chem.* 1967, *71*, 4209-4212.

COMPONENTS:

(1) Cerium; Ce; [7440-45-1]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Anonymous

US Atom. Ener. Comm. Rep. LA-3524-MS, 1966, p. 49-50.

VARIABLES:

One temperature: 973 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The concentration of Ce in liquid Na after an equilibration of the metals at 700 °C (973 K) was determined. The concentration of Ce was about the same after 5 h as after 100 h of equilibration, one may conclude that the saturation level of $\le 6\cdot10^{-4}$ mass % Ce or $\le 1\cdot10^{-4}$ mol % Ce (as calculated by the compilers) was reached even after the shorter time of equilibration.
The equilibrium concentration of Ce in Na, which was obtained using Ce-Co and C-Co-Pu alloys instead of Ce for equilibration, was found at the same level.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was supplied from a thermal convection loop, one leg of which passed through an evacuable inert-gas glove box. The solubility tests were performed in Ta capsules. A Ta getter sheath was inserted between each Ta capsule and its enclosing stainless steel container. The capsule was kept in thermostatic conditions for certain periods of equilibration and then water quenched. The Na phase was removed by cutting off the part of the capsule containing Na. A film of Na along the wall of the tube was then melted and bulk Na taken off in solid form. Na was dissolved in C_2H_5OH. The analytical method for the determination of Ce was not specified.

SOURCE AND PURITY OF MATERIALS:

Ce: "pure", with contents of $\sim 5\cdot10^{-3}$ % O, $\sim 3\cdot10^{-3}$ % C, $\sim 1\cdot10^{-3}$ % H.
Na: purified by gettering with Zr, with contents of $(8\pm5)\cdot10^{-4}$ % O, $\le 1{,}5\cdot10^{-3}$ % C, $\le 1{,}0\cdot10^{-3}$ % H.

ESTIMATED ERROR:

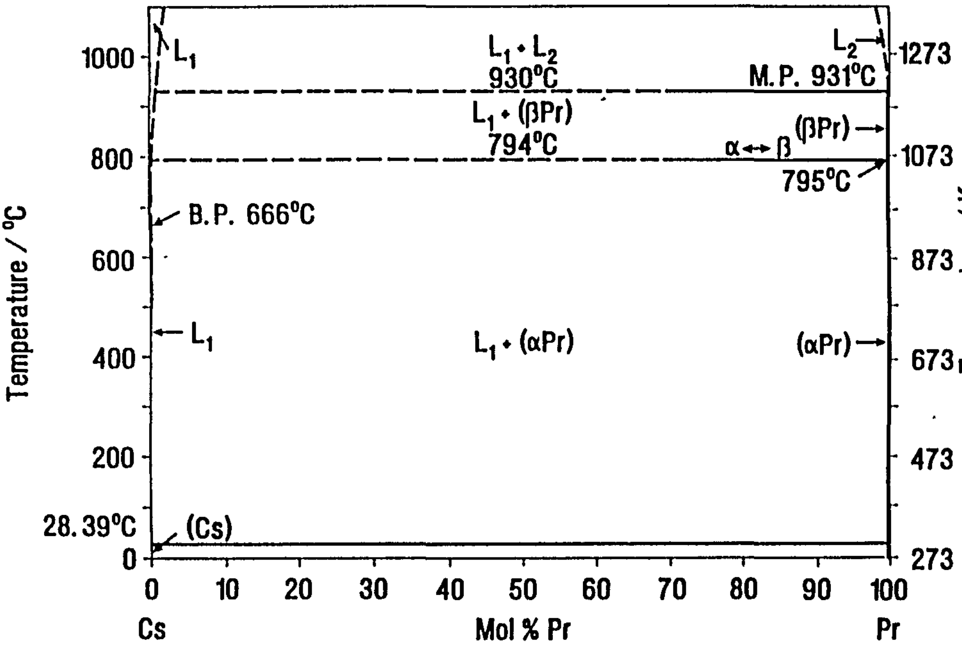
Solubility: nothing specified.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Praseodymium; Pr; [7440-10-0] (2) Caesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1991

CRITICAL EVALUATION:

Griffin and Gschneidner (1) performed differential thermal analyses of Pr-Cs alloys composed of 99.6 % Pr and of 99.97 % Cs. It was observed that about 0.4 % Cs reduces the melting point of the Pr-rich alloy as well as the α ↔ β transformation of Pr by about 1 K. The metals are nearly immiscible in the liquid state; however, exact compositions of the monotectics are unknown. A partly speculative phase diagram of the Pr-Cs system at the vapour pressure of Cs was reported in (2). The Cs-rich side was modified assuming that the Pr solubility near its melting point might not be higher than 1 mol % in analogy to other lanthanide-alkali metal systems.



References

1. Griffin, R.B.; Gschneidner, K.A. *Metall. Trans.* 1971, *2*, 2517.
2. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Mater., Materials Park, 1990, p. 1385.

COMPONENTS:	EVALUATOR:
(1) Neodymium; Nd; [7440-00-8] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1987

CRITICAL EVALUATION:

Solubility data of Nd in liquid Bi-Li melts was reported by Smith (1). The corresponding solubility of Nd (in the range of 1 mol % at 800 K) regularly increased from 0 to 25 mol % Li after an addition of Li. However, the slope of the temperature dependence of the solubility of Nd significantly changed at 38 mol % Li, indicating a change of the equlibrium solid phase. Additionally, Bi strongly interacted with Li and under these circumstances an extrapolation of the solubility data to the pure Li solvent was not reasonable.

References

1. Smith, F.J. *J. Less-Common Met.*, 1972, *27*, 195.

COMPONENTS:	EVALUATOR:
(1) Samarium; Sm; [7440-19-9] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1990

CRITICAL EVALUATION:

Smith (1) determined the solubility of Sm in liquid Pb-Li alloys at 773-973 K. The saturation concentrations of Sm in the alloys increased with increasing contents of Li. The highes value was measured to be about 12 mol % Sm at 873 K in the alloy containing 53 mol % Li. An extrapolation of these data cannot be recommended, since Sm forms stable intermetallics with Pb which forms also intermetallic compounds with Li (2). The formation of such compound may have influence on the equilibria in the ternary system. Interactions between Sm and Li are not to be expected. The solubility of Sm in Li and Li-rich alloys should be significantly lower than in the alloy with 53 mol % Li.

References

1. Smith, F.J. *J. Less-Common Met.* 1973, *32*, 297.
2. Hansen, M. Anderko, K. *Constitution of Binary Alloys*, McGraw-Hill, N.Y.; 1958.

COMPONENTS:	EVALUATOR:
(1) Samarium; Sm; [7440-19-9] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1990

CRITICAL EVALUATION:

Hoffman (1) observed a good resistance of a Sm_2O_3 sample in liquid Na at 1089 K in a static exposure of 100 h duration. Sm oxide is not reducible in liquid Na as can be concluded from tests with Ce in liquid Na. Sm_2O_3 exists in equilibrium with Na containing O, metallic Sm should be less soluble in liquid Na than its oxide.

References

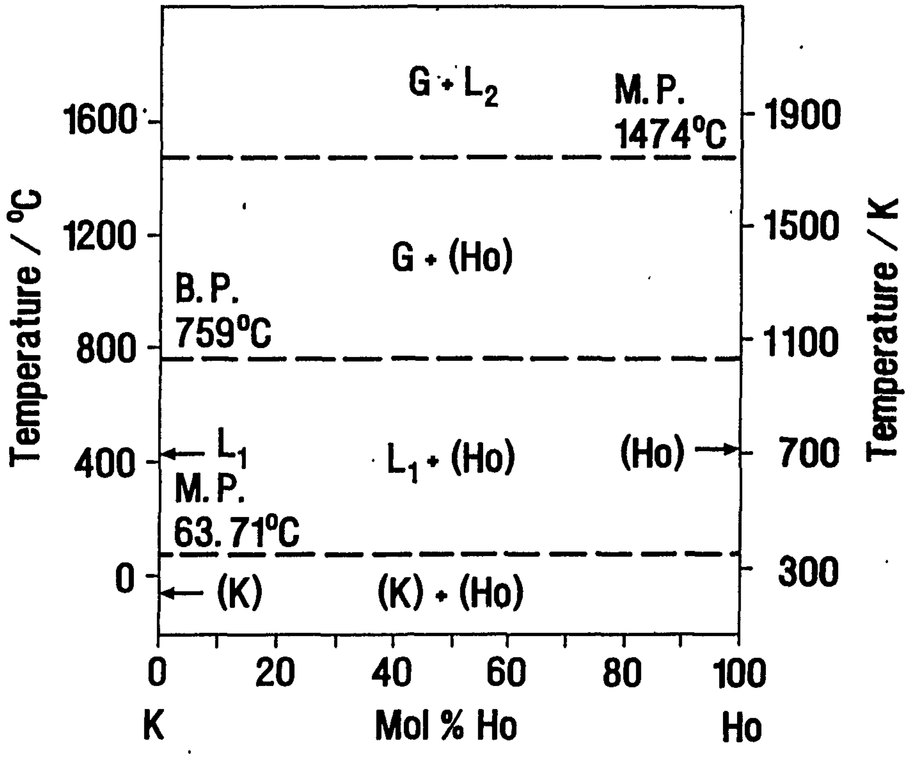
1. Hoffman, E.E. *US Atom.Ener.Comm.Rep. ORNL-2924*, 1960.

COMPONENTS:	EVALUATOR:
(1) Holmium; Ho; [7440-60-0] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1991

CRITICAL EVALUATION:

Aleksandrov and Dalakova (1) did not observe any dissolution of Ho in liquid K by means of spectral analysis after equilibration of solid Ho with liquid K at temperatures of 873-923 K for 1 hour. They did not specify the detection limit of the analytical method.

A speculative phase diagram of the Ho-K system is presented in the figure.



References

1. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

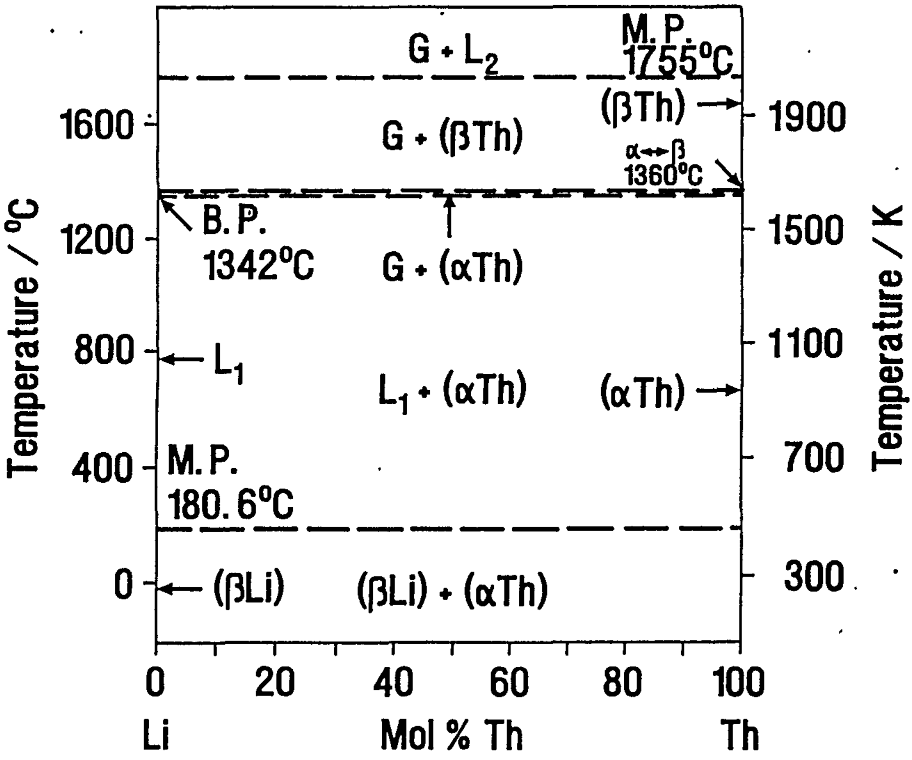
COMPONENTS:	EVALUATOR:
(1) Thorium; Th; [7440-29-1] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

The solubility of Th in liquid Li seems to be quite low. However, direct determinations of the solubility have not been performed. Wilkinson and Yaggee (1) confirmed a good corrosion resistance of Th in Li tested at 873 K for 144 hours under static conditions. A dissolution of Th was not observed. This was confirmed by Berry et al. (5) who reported similar experiments at 873 and 1273 K without presenting details.

A schematic Th-Li phase diagram can be plotted on the basis of these observations, and is presented in the figure. The presence of contaminating elements (N, O or both) in the Th-Li system , even in trace amounts, leads to the formation of surface nitrides (4,6) and oxides (4). Barker et al. (4) reported that ThO_2 does not react with pure Li. Li containing 0.3 to 4.5 at % N, however, attacks ThO_2. The reaction product is ThN, Th_2N_2O, or Li_2ThN_2 depending on the concentration of N.

Smith (2) determined the solubility of Th in liquid Bi-Li alloys at low N level to be lower than 1 mol % Th at temperatures below 900 K. The solubility of Th increased regularly with the increasing content of Li in the solution from O to 40 mol % Li. However, these data should not be extrapolated to 100 mol % Li, since the equilibrium solid phases in Bi-rich alloys with Th (3) are completely different from those in the Th-Li system. Similar measurements were performed in liquid Pb-Li alloys, for which a Th solubility of below 1 mol % Th at temperatures below 1073 was measured. The values of the solubility increased with increasing contents of Li in the liquid alloys from 0 to 44 mol % Li.



References

1. Wilkinson, W.D.; Yaggee, F.L. *US Atom.Ener.Comm. Rep. ANL-4990*, 1950.
2. Smith, F.J. *J. Less-Common Met.* 1972, *27*, 195.
3. Hansen, M.; Anderko, K. *Constitution of Binary Alloys*, McGraw-Hill, N.Y., 1958.
4. Barker, M.G.; Alexander, I.C.; Bentham, J. *J. Less-Common Met.* 1975, *42*, 241.
5. Berry, W.E.; Peoples, R.S.; Pray, H.A. *The Metal Thorium*, Am. Soc. Met., Cleveland, 1958, p. 267.
6. Barker, M.G.; Alexander, I.C. *J.Chem.Soc., Dalton Trans.* 1974, 2166.
7. Smith, F.J. *J. Less-Common Met.* 1973, *32*, 297.

COMPONENTS:	EVALUATOR:
(1) Thorium; Th; [7440-29-1] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

The observations and results concerning the solubility of Th in liquid Na which are reported in literature are contradictory.

Grube and Botzenhardt (1) determined solubility values of Th in liquid Na which were quite high; a value of 28.4 mol % Th was reported for a temperature of 439 K. The eutectic point was established at 3 mol % Th and 365 K. The occurrence of the compound $ThNa_4$ was proposed. Hayes and Gordon (5) reported that less then $1\cdot10^{-2}$ mol % Th may be dissolved in liquid Na at 573 K; experimental details were not given.

Kelman (2) was not successful in several attempts to discover a reaction of Th with Na at temperatures of 873-1073 K. Only traces of metallic Th in Na were determined after the experiments. Solid ThO_2 was accumulated at the bottom of a test autoclave in amounts proportional to the O content in Na.

Bett and Draycott (3) reported weight change data for Th samples exposed to liquid Na at elevated temperature. The oxidation product which was seen did not adhere to the Th samples.

Pearlman (4) studied the corrosion of Th exposed to stagnant Na (with O contents of 0.02-0.025 mol %) at temperatures of 884-1023 K; the test duration was 144-720 hours. The samples showed small weight gains.

The existence of a Th-Na intermetallic compound is unlikely. The Th-Na phase diagram seems to be similar to that shown for the Th-Li system, with the boiling point of Na at 1156 K. Most probably the solid phase in equilibrium with the solution is ThO_2. Metallic Th seems to be less soluble than its oxide. ThO_2 was resistant to Na at 1089 K for 100 h (6).

The data of (1) are compiled, though to solubility values and the proposed intermetallic compound cannot be recommended.

An addition of K to Na solvent did not influence the solubility of Th significantly. Foote (7) could not detect any measurable amount of Th in the K-Na eutectic at 873 K. The detection limit of the method was $3.7\cdot10^{-4}$ mol % Th, further details were not provided. Corrosion tests did not indicate any attack of the K-Na eutectic on Th in the temperature range 573 to 1173 K (2,4,8).

(5) reported solubility measurements of Th in the liquid melts of Sn-Na and Bi-Na. The addition of Na to Bi caused a decrease of the apparent solubility of Th.

References

1. Grube, G.; Botzenhardt, L. *Z. Elektrochem.* 1942, *48*, 418.
2. Kelman, L.R. *US Atom.Ener.Comm. Rep. CT-3726*, 1946.
3. Bett, F.L.; Draycott, A. *Peaceful Uses of Atomic Energy*, U.N., N.Y., 1958, *7*, 125.
4. Pearlman, H. *US Atom.Ener.Comm. Rep. NAA-SR-2225*, 1957; *US Atom.Ener.Comm. Rep. TID-7546*, 1957, p. 565.
5. Hayes, E.E.; Gordon, P.; cited by Epstein, L.; Weber, C.E. *US Atom.Ener.Comm. Rep. TID-2501*, 1951, p. 515.
6. Hoffman, E.E. *US Atom.Ener.Comm. Rep. ORNL-2924*, 1960.
7. Foote, F. cited by Epstein, L.; Weber, C.E. *US Atom.Ener.Comm. Rep. TID-2501*, 1951, p. 515.
8. Kelman, L.R.; as cited by Rough, F.A., Bauer, A.A. *US Atom.Ener.Comm. Rep. BMI-1300*, 1958, p. 129.

COMPONENTS:	EVALUATOR:
(1) Thorium; Th; [7440-29-1] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1987

CRITICAL EVALUATION:

Solubility measurements or corrosion tests of Th in unalloyed liquid K were not reported. The experiments performed with the K-Na eutectic alloy (described in the Th-Na Critical Evaluation) indicate a solubility of Th in liquid K below the detection limit in the temperature range up to 1173 K. The solubility of Th in liquid K as well as in the K-Na eutectic should be lower than in Na according to a model prediction of Niessen et al. (1). The Th-K phase diagram should be similar to that shown for the Th-Li system; the boiling point of K is at 1032 K.

References

1. Niessen, A.K.; de Boer, F.R.; Boom, R.; de Chatel, P.F.; Mattens, W.C.M.; Miedema, A.R. *CALPHAD*, 1983, *7*, 51.

COMPONENTS:

(1) Thorium; Th; [7440-29-1]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Grube, G.; Botzenhardt, L.

Z. Elektrochem. 1942, *48*, 418-425.

VARIABLES:

Temperature: 367-439 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Th in liquid Na corresponds to the points on the liquidus line.

t/°C	*soly*/mol % Th
96	0.52
95	1.09
94	2.42
117	4.07
140	6.20
149	9.02
158	12.94
165	18.78
166	28.39

The melting point of Na was determined at 97 °C.

The eutectic point was established at 3 mol % Th and 92 °C. The compound $ThNa_4$ was proposed as the solid equilibrium phase.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Th-Na alloys were prepared in a low carbon steel crucible. Thermal analyses of the alloys were performed and the corresponding cooling curves were recorded. A Ni/Ni-Cr thermoelement seemed to have been applied, which had been calibrated on the melting points of some metals. The content of the samples was confirmed by chemical analysis: Th was precipitated as oxalate and then weighed as oxide after a calcination procedure. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Th: powder of 99.77 % purity, contained 0.12 % Fe, traces of Si and O.
Na: unspecified.
Ar: specially purified (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Grube, G.; Ratsch, K. Z. *Elektrochem.* 1939, *45*, 838.

COMPONENTS:	EVALUATOR:
(1) Uranium; U; [7440-61-1] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

Qualitative information on the low solubility of U in liquid Li may be conducted from data on the corrosion resistance of U contacted with liquid Li for 144 hours at 873 K (1) and for 400 hours at 1273 K (2). Quantitative determinations of the solubility were performed by Bychkow et al. (3,4) in the temperature range from 978 to 1273 K. Except for the solubility value of $6\cdot10^{-3}$ mol % U at 1073 K, which was only 0.25 to 0.3 of the value to be expected from the other results, all the other could be fitted to the equation:

$$\log(soly/\text{mol \% U}) = 2.99 - 7190\,(T/\text{K})^{-1} \qquad r=0.987 \qquad \text{Eq.(1)}$$

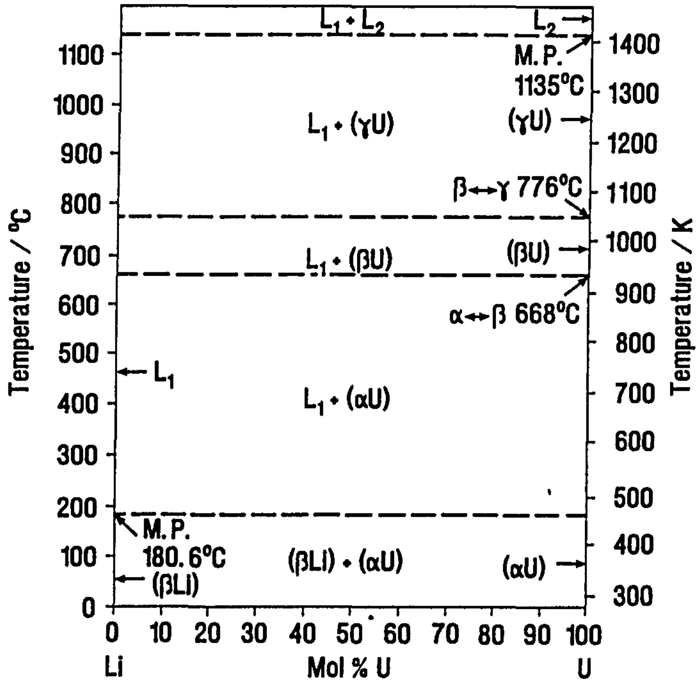
The scatter of the experimental data was not larger than ± 20 mol %. As U is known as a very effective getter for O, it seems likely that the solubility determined in (3,4) was somewhat influenced by chemical reactions. Thus, the solid phase in contact with the saturated solution might be an U oxide or nitride. Unfortunately, neither the content of O nor that of N were specified. Thus, a more precise estimation of such effects could not be provided. Besmann and Cooper (5) stated that U oxides or oxidic salts should be reduced by liquid Li to form metallic U and Li_2O.

The corrosion resistance of U in liquid Li indicates that intermetallic compounds are not formed; the saturated solution may be in equilibrium with almost pure U or an oxidized U compound.

A tentative U-Li phase diagram is shown in the figure. There is obviously not any distinct dependence of the solubility on the allotropic forms of U.

Tentative values of the solubility of U in liquid Li:

T/K	*soly* /mol % U	source	remarks
973	$4\cdot10^{-5}$	(3,4) and Eq. (1)	β U
1073	$2\cdot10^{-4}$	(3,4) and Eq. (1)	γ U
1173	$7\cdot10^{-4}$	Eq. (1)	γ U
1273	$2\cdot10^{-3}$	Eq. (1)	γ U



References

1. Wilkinson, W.D.; Yaggee, F.L. *US Atom.Ener.Comm. Rep. ANL-4991*, 1950.
2. Hoffman, E.E. *US Atom.Ener.Comm. Rep. ORNL-2924*, 1960.
3. Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B. *Atom. Energiya* 1959, *7*, 531; *Kernenergie* 1960, *3*, 763.
4. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall. Metalloved. Chist. Met.* 1960, *2*, 178.
5. Besmann, T.M.; Cooper, R.H. *US Dept. Ener. Rep. ORNL/TM-9662*, 1985.

COMPONENTS:

(1) Uranium; U; [7440-61-1]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B.

Atom. Energiya 1959, *7*, 531-536; *Kernenergie* 1960, *3*,763-767.

VARIABLES:

Temperature: 978-1273 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of U in liquid Li was determined. Values were read out the figure and recalculated to mol % by the compilers.

t/°C	*soly*/mass % U	*soly*/mol % U [a]
705	$1.5 \cdot 10^{-3}$	$4 \cdot 10^{-5}$
800	$2 \cdot 10^{-3}$	$6 \cdot 10^{-5}$
800	$6 \cdot 10^{-3}$	$1.8 \cdot 10^{-4}$
800	$8 \cdot 10^{-3}$	$2.4 \cdot 10^{-4}$
900	$2.8 \cdot 10^{-2}$	$8.4 \cdot 10^{-4}$
900	$3.5 \cdot 10^{-2}$	$1.0 \cdot 10^{-3}$
990 [b]	$5.0 \cdot 10^{-2}$	$1.5 \cdot 10^{-3}$
1000	$4.8 \cdot 10^{-2}$	$1.4\ 10^{-3}$

[a] as calculated by the compilers
[b] as read from (1) instead of the value for 1000 °C

All results were also reported in (1) except for the last one.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The inner surface of a U crucible was ground, electrolytically polished and etched. This crucible was gradually filled with freshly distilled Li dropping from a stainless steel condenser. After the process had been completed, the apparatus was filled with Ar, the crucible was placed in a stainless steel container, to which the cover was welded (in an arc furnace). Additionally, the crucible was isolated from the steel by a Mo band. The container was placed in an arc furnace and conditioned at a selected temperature for 50 hours. The Li solution was cooled to solidification in less than 50 s. The content of U in the sample was determined by colorimetric analysis. All essential operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

U: purity unspecified.
Li: contained after distillation: $(2\text{-}6) \cdot 10^{-2}$ mass % Na, 0.015 % K, $(1\text{-}4) \cdot 10^{-4}$ % Fe, 0.002 % Mg (or less); Si, Ni, and Cr not detected.
Ar: unspecified.

ESTIMATED ERROR:

Nothing specified.
Solubility: precision not better than ± 20 % (by the compilers).

REFERENCES:

1. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall. Metalloved. Chist. Met.* 1960, *2*, 178-188.

COMPONENTS:	EVALUATOR:
(1) Uranium; U; [7440-61-1] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1987

CRITICAL EVALUATION:

Though quantitative data do not result in a clear decision, the solubility of U in liquid Na has to be considered as very low. Foote (1) reported results of corrosion tests indicating that U was not appreciably attacked by liquid Na during several days at 773 K. Similar conclusions were drawn from experiments at 823 K (2) or even at 1073 K (3). In some of the corrosion tests liquid Na caused weight gains of U due to the formation of solid UO_2 on the surface of U samples (4). Other sources mention increases of weight as well as decreases (5-8) which might be due to the spallation of UO_2 layers during the exposure to liquid Na. It seems that the O content of liquid Na influences the physical properties of the UO_2 film. Dissolved U was not detected in liquid Na in (5).

Additional attempts to determine the solubility of U in liquid Na were reported by Epstein and Weber (9), they reported a solubility of $5 \cdot 10^{-4}$ mol % U in liquid Na at 573 K without giving details of the methods. Douglas (10,11) measured a decrease of the melting point of Na due to the addition of U. He estimated the solubility to be $\leq 7 \cdot 10^{-4}$ (10) or $\leq 5 \cdot 10^{-4}$ mol % U (11) at 371 K. Mogard (12) determined the U content in liquid Na equilibrated with U at 748 K at $< 1 \cdot 10^{-3}$ mol % U. These data show a decrease of the U solubility in liquid Na with increasing temperature.

It was shown in later tests that all U samples were covered with layers of UO_2. Thus, the solute in equilibrium with liquid Na is not the metal but UO_2. The resulting solution contains UO_2 or Na_3UO_4 (13) instead of metallic U. The solubility of U is still unknown, but should be lower than that of UO_2. Caputi and Adamson (13) used UO_2-PuO_2 reactor fuel as the solute, and observed a solubility of $2.2 \cdot 10^{-8}$ mol % at 848 K to $2.8 \cdot 10^{-7}$ mol % UO_2 at 1083 K. The presence of PuO_2 has certainly a negligible influence, as its solubility is also very low.

The strong discrepancies of some orders of magnitude between earlier (9-12) and more recent results (13) may be explained by the tendency of UO_2 to form a highly dispersed solid in liquid Na which caused an increased apparent solubility. Effective filter frits for such experiments should have pores of < 5 μm. It seems, therefore, that the results of (9) may be senseless, and the limits reported in (10),(11), and (12) are highly overstated. The authors of (13) found their data in acceptable agreement with those of (14) and (15). They combined these values to propose an equation for the solubility of UO_2 in liquid Na at temperatures between 850 and 1200 K (confirmed by the evaluators):

$$\log (soly/\text{mol } \% \text{ } UO_2) = -0.65 - 6000 \, (T/K)^{-1} \qquad \text{Eq.(1)}$$

The solubility in Na-K eutectic alloy is probably close to the value in Na (15); this paper is compiled in the U-(Na-K) system.

Na_3UO_4 is the equilibrium solid phase in the U-Na system contaminated with O (16).

The U-Na phase diagram should be analogous to that shown for the U-Li system, the boiling point of Na is 1156 K.

The tentative values of the solubility of UO_2 in liquid Na

T/K	$soly$/mol % UO_2	source
850	$2 \cdot 10^{-8}$	(13), Eq.(1)
973	$1.5 \cdot 10^{-7}$	(13), Eq.(1)
1073	$6 \cdot 10^{-7}$	Eq.(1)

References

1. Foote, F. *US Atom.Energ.Comm. Rep. CT-2857*, 1945.
2. Anonymous, Atom.Ener.Res.Establ., 1957; as cited by Rough, F.A.; Bauer, A.A. *US Atom.Energ.Comm. Rep. BMI-1300*, 1958.
3. Kelman, L.R. *US Atom.Energ.Comm. Rep. CT-3726*, 1946.
4. Isaacs, H.S. *J. Nucl. Mater.* 1970, *36*, 322.
5. Pearlman, H. *US Atom.Energ.Comm. Rep. NAA-SR-2225*, 1957.
6. Hoffman, E.E., Oak Ridge Nat.Labor., 1957; as reported in 5.
7. Davis, M.; Draycott, A. *Peaceful Uses of Atomic Energy*, U.N., N.Y., 1958, *7*, 94.
8. Kelman, L.R., Argonne Nat.Labor., 1955; as cited by Wilkinson, W.D., *Uranium Metal*, Interscience, N.Y., 1962, vol. II, p. 846.
9. Epstein, L.F.; Weber, C.E. *US Atom.Energ.Comm. Rep. TID-2501*, 1951, p. 515.
10. Douglas, T.B. *US Atom.Energ.Comm. Rep. AECD-3254*, 1951.
11. Douglas, T.B. *J. Res. Nat. Bur. Stand.* 1954, *52*, 223.
12. Mogard, H. *Peaceful Uses of Atomic Energy*, U.N., N.Y., 1955, *9*, 318.
13. Caputi, R.W.; Adamson, M.G. *US Dep. Ener. Rep. CONF-800401-P2*, 1980, no. 18, p. 62.
14. Bohaboy, P.E.; Regimbal, J.J.; Craig, C.N.; Gilbert, R.S. *US Atom.Energ.Comm. Rep. GEAP-13977*, 1974.
15. Davies, R.A.; Drummond, J.L. *UK Atom.Ener.Auth.Rep.TRG-2363*, 1973; *J. Brit. Nucl. Ener. Soc.* 1973, *12*, 427.
16. Lindemer, T.B.; Besman, T.M.; Johnson, C.E., *J. Nucl. Mater.* 1981, *100*, 178.

COMPONENTS:

(1) Uranium; U; [7440-61-1]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Douglas, T.B.

J. Res. Nat. Bur. Stand. 1954, *52*, 223-226.

VARIABLES:

One temperature: 371.0 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of U in liquid Na at 97.8 °C is estimated to be 0.05 mass % ($5 \cdot 10^{-3}$ mol % U as calculated by the compilers), it may, however, be much smaller. The result reported in (1) is 0.007 mass % ($7 \cdot 10^{-3}$ mol % U).
It was observed that pulverized U removes O from Na_2O which was present in Na as a contaminant. U addition to Na increased first the melting point of Na by 0.005 K, due to this getter reaction. The melting point was afterwards decreased by 0.001 to 0.003 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The equilibrium temperatures of 3 samples of Na at various stages of melting were measured. The samples represented: pure Na, Na with compact U, and Na with fine dispersed U. The samples were sealed in a He atmosphere in a small cylinder of stainless steel which was suspended on a Nichrome wire within an Ag isothermal jacket. The assembly was heated by means of a Constantan heater. A four-function differential thermocouple (Chromel/Constantan) which was calibrated against freezing Na was used to precisely measure the temperature difference between the Ag pipe and the sample. The furnace was heated to a temperature at which Na starts to melt and was kept at this temperature for at least 3 hours. The sample was then heated for separate intervals of time until the melting was complete. Thus, at any stage of the fusion the total heat which was introduced could be a measure of the molten fraction of Na. Corrections of the temperature measurements were made taking into account the response of thermocouples and the temperature gradient in the apparatus. The temperature differences between the samples were within the precision of the measurements. Assuming a real difference of 0.02 K the limiting value of the solubility was estimated applying van't Hoff's isobaric equation. A correction for the dissolved content of Na_2O in Na was also made.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.
Probably the elements were of highest purity available at that time.
U: fine dispersed material gained by means of decomposition of the hydride.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: stability ± 0.0005 K; precision ± 0.001 K; accuracy ± 0.02 K.

REFERENCES:

1. Douglas, T.B. *US Atom.Energ.Comm. Rep. AECD-3254*, 1951.

COMPONENTS:

(1) Uranium; U; [7440-61-1]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Mogard, H.

Peaceful Uses of Atomic Energy, U.N., N.Y., 1955, *9*, 318-320.

VARIABLES:

One temperature: 748 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Liquid Na which was equilibrated with a U sample at 475 °C contained 0.01 mass % or 0.001 mol % U (as calculated by the compilers).
Metallic U was covered with a film of UO_2 which was detected by X-ray examination. Traces of Na or Fe were not detected in the corroded surface of the U sample.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The two metals were placed inside mild steel tubes. The tubes and the U sample were electropolished and dried before the test. The loaded tubes were evacuated and closed by welding. They were introduced into Pyrex glass capsules with Ar atmosphere. Ca was added as an effective getter for O in a part of the tests. The surface of U samples remained nearly metallic under such conditions. The solvent Na was filtered at the selected temperature. The analytical method was not reported.

SOURCE AND PURITY OF MATERIALS:

U: "high purity", degassed by means of vacuum extraction at 873 K.
Na: analytical purity, from Merck; with a content of 0.04 % O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Uranium dioxide (Urania); UO_2; [1344-57-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Bohaboy, P.E.; Regimbal, J.J.; Craig, C.N.; Gilbert, R.S.
US Atom.Energ.Comm. Rep. GEAP-13977, 1974.

VARIABLES:

Temperature: 1158-1236 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The equilibrium concentrations of U at various sections of the capsule with $(U_{0.75}Pu_{0.25})O_2$ as solid phase in liquid Na were measured at temperatures in the range 1158-1236 K..

Capsule section	*concn*/10^{12} atoms per g Na		*concn*/mol % U [a]
	^{235}U	^{238}U	
between pump and flow meter	1200	7300	$3.2 \cdot 10^{-5}$
between pump and flow meter	650	5400	$2.3 \cdot 10^{-5}$
directly above fuel and getter	1300	9500	$4.1 \cdot 10^{-5}$
bottom of capsule	2400	12000	$5.5 \cdot 10^{-5}$

[a] - as calculated by the compilers as the sum of ^{235}U and ^{238}U

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A forced circulation capsule consisted of a loop with inner and outer annulus through which a flow of Na was maintained. The capsule was made of stainless steel, it contained a defected reactor fuel rod (mixed ^{235}U-Pu oxide), metallic ^{238}U foil getter, gas sampling lines and facilities to control the Na flow. Chromel/Alumel thermocouples were used to measure the temperature of the different positions of the capsule. The facility was operated for several days. The Na samples were obtained after cutting the capsule in sections and immersing the sections in hexane to which CH_3OH and H_2O was slowly added to dissolve Na. The walls of the apparatus were treated with HNO_3 and HCl. The resulting solutions were analyzed for their U contents by gamma spectrometry.

SOURCE AND PURITY OF MATERIALS:

UO_2: co-precipitated $(U_{0.75}Pu_{0.25})O_{1.970}$.
Na: purified by gettering with U foil, containing $<2 \cdot 10^{-5}$ % O.

ESTIMATED ERROR:

Solubility: precision ± 50 % in separate fractions.
Temperature: see the experimental range of temperatures.

REFERENCES:

COMPONENTS:

(1) Uranium; U; [7440-61-1]
or Uranium dioxide (Urania); UO_2; [1344-57-6]
(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Caputi, R.W.; Adamson, M.G.

US Dept.Ener. Rep. CONF-800401, Pt. 2, 1980, 18/62-69.

VARIABLES:

Temperature: 848-1083 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of UO_2 from mixed oxide $U_{.75}Pu_{.25}O_2$ in liquid Na was determined and presented in the figure. The values were read out and recalculated into mol % by the compilers.

t/°C	*soly*/μg U/g Na	*soly*/mol % U
575	$2.3 \cdot 10^{-3}$	$2.2 \cdot 10^{-8}$
700	$1.8 \cdot 10^{-2}$	$1.7 \cdot 10^{-7}$
800	$6.0 \cdot 10^{-3}$	$5.8 \cdot 10^{-8}$
810	$2.9 \cdot 10^{-2}$	$2.8 \cdot 10^{-7}$

Probably, UO_2 dissolves in Na as uranate Na_3UO_4.

A 10 μm frit is not effective to separate quantitatively the fine dispersed oxide as is the 5 μm filter.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility experiments were performed in a filter capsule under high purity inert atmosphere. After the sodium, the mixed oxide fuel chip, the initial composition of which was $U_{.75}Pu_{.25}O_2$, and Ni sampling foil had been loaded, the type 316 stainless steel capsule was sealed and inserted into the furnace. The location in the furnace was chosen such that the temperature of the condensing cone was in the range of 473-523 K. Once the location was set the capsule was brought up to a temperature of 823-1073 K and kept in operation for a given time. The basic principle was to maintain a flow of fresh sodium over the mixed oxide chip to establish the molecular solubility of UO_2. This flow of sodium was developed and maintained by the standard technique of refluxing. A fine metal frit was applied to retain as much of the particulate (derived from microspallation) as possible. The frit used for this purpose had a pore size of 5-10 μm. After the test was completed the Ni foil was removed for analysis of U and Pu. The samples were initially rinsed with water/alcohol to remove particulate oxide and leached with HNO_3 acid to dissolve the oxide. Finally the U was analyzed by means of mass spectrometry.

SOURCE AND PURITY OF MATERIALS:

UO_2: mixed oxide $U_{.75}Pu_{.25}O_{2.0}$ was used as UO_2 source.
Na: high purity, distilled and filtered;
the equilibrium O concentration was in the range: $2.5 \cdot 10^{-5}$ - $6.6 \cdot 10^{-4}$ % (1).

ESTIMATED ERROR:

Solubility: precision ± 50 %.
Temperature: nothing specified.

REFERENCES:

1. Adamson, M.G.; Aitken, E.A.; Jeter, D.W. *US Dept.Ener. Rep. CONF-760503-P2*, 1976, p. 866-872.

COMPONENTS:

(1) Uranium; U; [7440-61-1]
or Uranium dioxide (Urania); UO_2; [1344-57-6]
(2) Sodium-Potassium; $Na_{80}K_{20}$; [11147-16-3]

ORIGINAL MEASUREMENTS:

Davies, R.A.; Drummond, J.

J. Brit. Nucl.Ener.Soc. 1973, *12*, 427-435.

VARIABLES:

Temperature: 873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Samples of the primary circuit reactor coolant Na-K(30 %) were analyzed for their content of U; assuming that a saturation was reached, one may treat them as the solubilities. Before the occurrence of the leak in the primary circuit, 47 samples were analyzed. The mean result is 0.35 μg U/g Na-K changing from 0.01 to 3.1 μg U/g Na-K but most of the results scattered around the mean value. The values were recalculated by the compilers to a mean value of $4.1 \cdot 10^{-6}$ mol % U.
Another 47 samples were analyzed after this leak was found. The average of these measurements is 0.65 μg U/g Na-K changing from 0.03 to 3.6 μg U/g Na-K, the largest part of results is close to the average. The compilers recalculation resulted in a concentration of $7.6 \cdot 10^{-6}$ mol % U. 4 results were one order of magnitude higher, they were not taken into account.
Better reproducible results were obtained, if the immersion time of the sampling crucible was $\geq$ 6 hours.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Samples of the reactor coolant were obtained by a dip sampling technique with Ni crucibles. The immersion time was varied from 5 min to 12 hours, a typical immersion time was 6 hours. The samples were first washed with CH_3OH and then with acid (8 mol·dm^{-3} HNO_3 and 0.1 mol·dm^{-3} HCl). The resulting solutions were evaporated. The U content was measured by isotope dilution using solid source mass spectrometry after extraction into ether and separation by paper strip chromatography.

SOURCE AND PURITY OF MATERIALS:

U: from U-Mo alloy fuel.
Na: reactor grade, contaminated by the fission products, O content determined, but not reported.

ESTIMATED ERROR:

Solubility: precision within an order of magnitude (by the compilers).
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Uranium; U; [7440-61-1]

(2) Potassium; K; [7440-09-7]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
June 1989

CRITICAL EVALUATION:

According to emf measurements performed by Adamson et al. (1), the interaction product of liquid K and UO_2 has the composition KUO_3. A real solubility of metallic U in liquid K could not be determined due to the fact that U has a strong affinity for O in the system U-O-K, and O cannot be totally excluded in the experiments. The solubility of metallic U in K is, therefore, expected to be lower than that of UO_2.
The equilibrium compounds of equilibration of UO_2 or of metallic U with liquid K containing small amounts of impurities are essentially the same. A schematic U-K phase diagram should be similar to that shown for the U-Li system, with a difference in the boiling point of K at 1032 K, which is closely just the $\beta U \leftrightarrow \gamma U$ transformation temperature (1049 K).
The corrosion resistance of U in liquid K-Na alloys was studied in (2-4). The experiments indicated a negligible solubility of U in the liquid alloy. Davies and Drummond (5) determined the solubility of mixed UO_2-PuO_2 reactor fuel in the K-Na alloy at 873 K and found the apparent solubility of UO_2 to be $5 \cdot 10^{-8}$ mol %. This low value demonstrates that the solubility of metallic U in pure K may be below the detection limits of the available analytical methods.

References

1. Adamson, M.G.; Aitken, E.A.; Jeter, D.W. *US Dept.Ener. Rep. CONF-760503-P2*, 1976, p. 866.
2. Kelman, L.R. *US Atom.Ener.Comm.Rep. CT-3726*, 1946.
3. Pearlman, H.*US Atom.Ener.Comm.Rep. TID-7546*, 1957, p. 565.
4. Davis, M.; Draycott, A. *Peaceful Uses of Atomic Energy*, UN, N.Y., 1958, *7*, 94.
5. Davies, R.A.; Drummond, J.L. *J. Brit. Nucl. Ener. Soc.* 1973, *12*, 427.

COMPONENTS:

(1) Uranium; U; [7440-61-1]

(2) Rubidium; Rb; [7440-17-7]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
June 1992

CRITICAL EVALUATION:

The solubility of U in liquid Rb has not been reported in the literature. Certainly the system is similar to the systems of U with K and Cs. A schematic U-Rb phase diagram should be similar to that shown for the U-Li system, with a difference in the boiling point of Rb at 959 K. A very limited solubility of Rb in liquid U should be observed in the Rb-U system slightly contaminated with O, according to a schematic U-Rb-O phase diagram reported by Iyer et al. (1). The equilibrium solute is Rb_4UO_5 (1).

References

1. Iyer, V.S.; Venugopal, V.; Sood, D.D. *J. Radioanal. Nucl. Chem.* 1990, *143*, 157.

COMPONENTS:

(1) Uranium; U; [7440-61-1]

(2) Cesium, Cs; [7440-46-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
June 1989

CRITICAL EVALUATION:

Solubility determinations in the U-Cs system have not been reported so far. However, emf measurements were performed by Adamson et al. (1). These experiments showed that very small amounts of a UO_2 were dissolved in liquid Cs; the composition of the equilibrium phase was reported as $Cs_{0.5}UO_3$. The solubility of metallic U in Cs is certainly lower than that of UO_2.
Kohli (2) reported Cs_2UO_4 as a solid product formed in the U-Cs system contaminated with O. A schematic U-Cs phase diagram should be similar to that shown for the U-Li system, with the boiling point of Cs at 944 K.

References

1. Adamson, M.G.; Aitken, E.A.; Jeter, D.W. *US Dept.Ener. Rep. CONF-760503-P2*, 1976, p. 866.
2. Kohli, R. *Material Behavior and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum Press, N.Y., 1982, p. 345.

COMPONENTS:

(1) Plutonium; Pu; [7440-07-5]

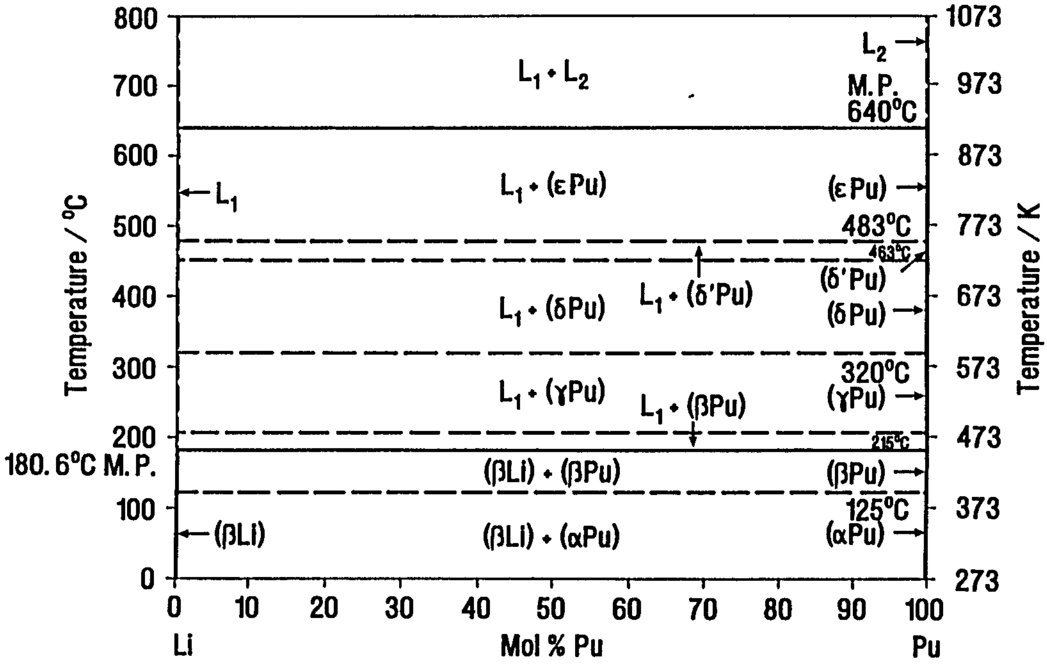
(2) Lithium; Li; [7439-93-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
November 1991

CRITICAL EVALUATION:

Experimental determinations of the solubility of Pu in liquid Li have not been published. Rough examinations of the Pu-Li system indicated a rather low solubility as well as a limited miscibility in the solid and liquid states (1,2). A schematic phase diagram of the Pu-Li system was reported in (3) and (4), and is redrawn in the figure below.



References

1. Schonfeld, F.W.; Cramer, E.M.; Miner, W.N.; Ellinger, F.H.; Coffinbery, A.S. *Progr. Nucl. Ener., Ser. V* 1959, *2*, 579.
2. Bochvar, A.A.; Konobeevskii, S.T.; Kutaitsev, V.I.; Menshikova, T.S.; Chebotarev, N.T. *Peaceful Uses of Atomic Energy*, U.N., N.Y., 1958, *6*, 184; *Atom. Energiya* 1958, *5*, 303.
3. Agapova, N.P.; Bochvar, A.A.; Zaimovskii, A.S.; Konobeevskii, S.T.; Kutaitsev, V.I. *Sovetskaya Atomnaya Nauka i Tekhnika*, Atomizadat, Moskva, 1967, p. 240.
4. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am.Soc.Mater., Materials Park, 1990, p. 2460.

COMPONENTS: (1) Plutonium; Pu; [7440-07-5] (2) Sodium; Na; [7440-23-5]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1987

CRITICAL EVALUATION:

It was established that Pu and Na are completely immiscible in the liquid and solid state in preliminary experiments performed in the USA (1) and the USSR (2). A schematic phase diagram of the Pu-Na system was reported in (3), it is analogous to that shown for the Pu-Li system with differences in the melting and boiling points of Na.
Since O is more strongly bonded by Pu than by Na, the whole surface of metallic Pu is oxidized to PuO_2 by liquid Na containing even traces of O. Thus, the solubility of PuO_2 instead of Pu in liquid Na is measured. The solubility of the metal itself seems to be significantly lower.
In the first reported determinations of the solubility of Pu in liquid Na a value of $\sim 1\cdot 10^{-5}$ mol % Pu was found at 973 K (4). This result might be due to the not adequate analytical method. A considerably lower value of $<2\cdot 10^{-6}$ mol % Pu was measured after the equilibration with a Ce-Co-Pu alloy, probably due to the lower chemical activity of Pu in the alloy in which an intermetallic phase Co-Pu might have formed.
Four different groups determined the solubility of PuO_2 in Na by equilibrating mixed reactor fuel PuO_2-UO_2 with liquid Na (5-8). Bingham and Jones (5) performed measurements at 1073 K, but they obtained results which were significantly scattered from $7\cdot 10^{-9}$ to $3.6\cdot 10^{-6}$ mol % Pu. The poor reproducibility might due to the fact that molecularly dissolved PuO_2 was not effectively separated from finely dispersed solid or colloidal PuO_2 in liquid Na. An effective separation can be obtained by means of 5 μm filters (8). The publication (5) was not available and is not compiled. The results of (5-8) were in agreement, if one bears in mind the difficulties which might occur in analyses at trace levels. The presence of U in the solute as well as in the solution does not seem to have a serious influence on the measured solubility, since Pu and U oxides are soluble in liquid Na only in trace amounts; a sufficient equilibration time should be ensured. Davies and Drummond (6) used a K-Na(80 mol %) melt as the solvent. Bohaboy et al. (7) performed experiments at ~1200 K, the results being in the range of $9\cdot 10^{-7}$ to $7\cdot 10^{-6}$ mol % Pu. Finally Caputi and Adamson (8) determined the solubility of $1.7\cdot 10^{-10}$ to $3.9\cdot 10^{-7}$ mol % Pu at 848 to 1083 K. The combined data of (6-8) fitted the least squares equation (confirmed by the evaluators):

$$\log(soly/\text{mol \% } PuO_2) = 3.36 - 10900\,(T/K)^{-1} \qquad \text{Eq.(1)}$$

Stamm et al. (9) found by neutron activation analysis an amount of < 1 mg Pu in 22000 kg of Na serving as reactor coolant at a temperature of ~800 K; the corresponding Pu concentration of $< 5\cdot 10^{-10}$ mol % Pu is in agreement with the results of (6), if one assumes saturation under the conditions of the reactor circuit. The paper (9) is not compiled, since details of the experiments were missing.

Tentative value of the solubility of PuO_2 in liquid Na:

T/K	$soly$/mol % PuO_2	source
850	$3.0\cdot 10^{-10}$	Eq.(1)
973	$1.5\cdot 10^{-8}$	Eq.(1)
1073	$1.5\cdot 10^{-7}$	(8) and Eq.(1)

References

1. Schonfeld, F.W.; Cramer, E.M.; Miner, W.N.; Ellinger, F.H.; Coffinberry, A.S. *Progr. Nucl. Ener., Ser. V.* 1959, 2, 579.
2. Bochvar, A.A.; Konobeevskii, S.T.; Kutaitsev, V.I.; Menshikova, T.S.; Chebotarev, N.T. *Peaceful Uses of Atomic Energy*, U.N.; N.Y., 1956, *6*, 184; *Atom. Energiya* 1958, *5*, 303.
3. Agapova, N.P.; Bochvar, A.A.; Zaimovskii, A.S.; Konobeevskii, S.T.; Kutaitsev, V.I. *Sovetskaya Atomnaya Nauka i Tekhnika*, Atomizadat, Moskva, 1967, p. 240.
4. Anonymous, *US Atom. Ener. Comm. Rep. LA-3524-MS*, 1966, p. 49.
5. Bingham, C.D.; Jones, L.J. *Atomics International Rep. TR-095-24-032*, 1970; as reported in (8).
6. Davies, R.A.; Drummond, J.L. *UK Atom.Ener.Auth. Rep. TRG-2363*, 1973; *J. Brit. Nucl. Ener. Soc.* 1973, *12*, 427.
7. Bohaboy, P.E.; Regimbal, J.J.; Craig, C.N.; Gilbert, R.S. *US Atom.Ener.Comm. Rep. GEAP-13977*, 1974.
8. Caputi, R.W.; Adamson, M.G. *US Dept. of Ener. Rep. CONF-800401-P2*, 1980, p. 18/62.
9. Stamm, H.H. *US Dept. of Ener. Rep. CONF-800401-P2*, 1980, p. 15/74 (discussion).

COMPONENTS:

(1) Plutonium; Pu; [7440-07-5]
or Plutonium dioxide (Plutonia); PuO_2; [12059-95-9]
(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Bohaboy, P.E.; Regimbal, J.J.; Craig, C.N.; Gilbert, R.S.
US Atom.Ener.Comm. Rep. GEAP-13977, 1974.

VARIABLES:

Temperature: 1158-1236 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Concentrations of PuO_2 at various capsule sections from $U_{0.75}Pu_{0.25}O_2$ equilibrated with liquid Na are reported.

section of capssule	*concn*/10^{12} atoms of Pu/g Na	*concn*/mol % Pu [a]
between pump and flow deflector	580	$2.2 \cdot 10^{-6}$
between pump and flow deflectðr	230	$0.9 \cdot 10^{-6}$
directly above bottom of capsule	630	$2.4 \cdot 10^{-6}$
directly above bottom of capsule	1800	$6.9 \cdot 10^{-6}$

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A forced circulation capsule was essentially a flowing Na loop with inner and outer annulus. The capsule was made of a stainless steel and contained defected reactor fuel rod (U-Pu mixed oxide), U getter, gas ampling lines and facilities to control flow of the Na. Chromel/Alumel thermocouples were used to controll the temperature of the capsule. The capsule was operated for several days. Na samples were obtained by immersing each capsule section in hexane and slowly adding CH_3OH and H_2O to dissolve Na. Walls of the apparatus were leached with HNO_3 and HCl. The resulting solutions were individually analized for Pu content by gamma spectrometry.

SOURCE AND PURITY OF MATERIALS:

PuO_2: from co-precipitated $U_{0.75}Pu_{0.25}O_{1.970}$.
Na: gettered with U foil, final content of $< 2 \cdot 10^{-5}$ % O.

ESTIMATED ERROR:

Solubility: precision ± 50 % in subsequent sections of capsules.
Temperature: nothing specified; some scores of K (by the compilers).

REFERENCES:

COMPONENTS:

(1) Plutonium; Pu; [7440-07-5]
or Plutonium dioxide (Plutonia); PuO_2; [12059-95-9]
(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Caputi, R.W.; Adamson, M.G.

US Dept.Ener.Rep. CONF-800401-P2, 1980, 18/62-69.

VARIABLES:

Temperature: 848-1083 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The PuO_2 solubilities from mixed oxide $U_{0.75}Pu_{0.25}O_2$ in liquid Na are presented in the figure. They were read out and recalculated into mol % by the compilers.

t/°C	*soly*/(μg Pu/g Na)	*soly*/mol % Pu
575	$1.8\cdot10^{-5}$	$1.7\cdot10^{-10}$
700	$6.0\cdot10^{-3}$	$5.7\cdot10^{-8}$
800	$1.5\cdot10^{-2}$	$1.5\cdot10^{-7}$
810	$4.2\cdot10^{-2}$	$3.9\ 10^{-7}$

A 10 μm frit is not effective for a quantitative separation the fine dispersed oxide as with a 5 μm filter. Pu is probably dissolved in Na as plutanate Na_3PuO_4 or solvated PuO_4^{-3} ion.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility experiments were performed in a filter capsule under high-purity inert atmosphere. The mixed oxide fuel chip, the initial composition of which was $U_{0.75}Pu_{0.25}O_2$, and Ni sampling foil had been loaded into the Na solute. The capsule of type 316 stainless steel was then sealed and inverted into the furnace. Its location in the furnace was in the zone with a temperature of 200-250°C. Once the location was set, the capsule was brought up to a temperature of (550-800°C) and kept in operation for a given time. The basic principle was to maintain a flow of fresh sodium over the mixed oxide chip to establish the molecular solubility of Pu. This flow of sodium was produced and maintained by the standard technique of refluxing. A fine metal frit was applied to retain as much of the particulate (derived from microspallation) as possible. The frit used for this purpose had a pore size of 5-10 μm. After the completion of the test the Ni foil was removed for analysis of U/Pu. The samples were initially rinsed with H_2O/alcohol to remove particulate oxide and leached with nitric acid to dissolve oxide. Finally Pu was analized by means of mass spectrometry.

SOURCE AND PURITY OF MATERIALS:

PuO_2: from $U_{0.75}Pu_{0.25}O_2$.
Na: high purity further distilled and filtered; equilibrium O concentration in the range: $2.5\cdot10^{-5}$ to $6.5.10^{-4}$ % (1).

ESTIMATED ERROR:

Solubility: precision ± 200 %.
Temperature: nothing specified.

REFERENCES:

1. Adamson, M.G.; Aitken, E.A.; Jeter, D.W. *US Dept.Ener.Rep. CONF-760503-P2*, 1976, p. 866-872.

COMPONENTS:

(1) Plutonium; Pu; [7440-07-5]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Anonymous

US Atom. Ener. Comm. Rep. LA-3524-MS, 1966, p. 49-50.

VARIABLES:

One temperature: 973 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The concentration of Pu in liquid Na after an equilibration of the metals at 700 °C (973 K) for 24 to 96 h was determined. Since the Pu concentration was practically stable with the time of equilibration, the saturation was obviously already reached after 24 h.

time/h	*soly*/mass % Pu	*soly*/mol % Pu [a]
24	$8.5 \cdot 10^{-5}$	$8.1 \cdot 10^{-6}$
48	$8.6 \cdot 10^{-5}$	$8.2 \cdot 10^{-6}$
96	$1.2 \cdot 10^{-4}$	$1.1 \cdot 10^{-5}$

[a] calculated by the compilers

The equilibrium concentration of Pu in liquid Na which was contacted to a Ce-Co-Pu alloy at 700 °C for either 5 or 100 h was $< 2 \cdot 10^{-5}$ mass % Pu or $< 2 \cdot 10^{-6}$ mol % Pu.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was supplied from a thermal convection loop, one leg of which passed through an evacuable inert-gas glove box. The solubility tests were performed in Ta capsules. A Ta getter sheath was inserted between each Ta capsule and its enclosing stainless steel container. The capsule was kept in thermostatic conditions for certain periods of equilibration and then water quenched. The Na phase was removed by cutting off the part of the capsule containing Na. A film of Na along the wall of the tube was then melted and bulk Na taken off in solid form. Na was dissolved in C_2H_5OH. The analytical method for the determination of Pu was not specified.

SOURCE AND PURITY OF MATERIALS:

Pu: "pure".

Na: purified by gettering with Zr, with contents of $(8 \pm 5) \cdot 10^{-4}$ % O, $\leq 1{,}5 \cdot 10^{-3}$ % C, $\leq 1{,}0 \cdot 10^{-3}$ % H.

ESTIMATED ERROR:

Solubility: nothing specified; precision not better than ± 10 % (by the compilers).

Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Plutonium; Pu; [7440-07-5]

(2) Potassium-Sodium eutectic; $K_{68}Na_{32}$; [11147-16-3]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
June 1987

CRITICAL EVALUATION:

Due to the strong affinity of Pu to O, PuO_2 is the solid phase in equilibrium with the liquid K-Na eutectic contaminated by O instead of metallic Pu.
Davies and Drummond (1) reported a solubility of PuO_2 in liquid K-Na at 873 K. The value which they obtained seems to be acceptable, however, there were difficulties to define the equilibrium, since the applied PuO_2 source was the mixed oxide PuO_2-UO_2, and the solvent was composed of the two alkali metals, Na and K. The saturated solution with traces of a Pu compound may contain Na_3UO_4, Na_3PuO_4, KUO_3 and a potassium plutonate the exact stoichiometry of which is unknown(2).

References

1. Davies, R.A.; Drummond, J.L. *UK Atom.Ener.Auth. Rep. TRG-2363*, 1973.
2. Adamson, M.G.; Aitken, E.A.; Jeter, D.W. *US Dept.Ener. Rep. CONF-760503-P2*, 1976, p. 866.

COMPONENTS:

(1) Plutonium; Pu; [7440-07-5]
or Plutonium dioxide (Plutonia); PuO_2; [12059-95-9]
(2) Sodium-Potassium; $Na_{70}K_{30}$; [11147-16-3]

ORIGINAL MEASUREMENTS:

Davies, R.A.; Drummond, J.

J. Brit. Nucl. Ener. Soc., 1973, *12*, 427-435.

VARIABLES:

Temperature: 873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Primary circuit reactor coolant ($Na_{70}K_{30}$ in mass %) samples were analyzed for the content of Pu. Assuming that a saturation was reached, we may treat them as the solubilities.
Before the occurrence of the primary circuit leak, 25 samples were, analyzed and the mean result was 0.4 mg Pu/g Na-K varying from 0.1 to 2.1 mg Pu/g Na-K. The recalculation results in a solubility of $4 \cdot 10^{-9}$ mol % Pu. Another 28 samples were analyzed after the primary circuit leak, and the mean result was 1.5 mg Pu/g Na-K scattering from 0.1 to 5.5 mg Pu/g Na-K. Many results were close to the mean value. The solubility is calculated to be $1.5 \cdot 10^{-8}$ mol % Pu. Three values which were one order of magnitude higher, were not taken into account.
The results were better reproducible, if the immersion time of the sampling crucible was $\geq$ 6 hours.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Samples of the reactor coolant were obtained by a dip sampling technique with Ni crucibles. Immersion time was changed from 5 min to 12 hours but typically it was 6 hours. The samples were first washed with CH_3CH and the with acid (8 mol dm^{-3} HNO_3 and 0.1 mol dm^{-3} HCl). The solutions were evaporated. The Pu content was measured by alpha-counting after separation by solvent extraction with hyamine 1622.

SOURCE AND PURITY OF MATERIALS:

Pu: from the experimental fuel
Na: reactor grade, contaminated by the fission products, O content determined yet unspecified

ESTIMATED ERROR:

Solubility: precision within the order of magnitude (by the compilers).
Temperature: nothing specified.

REFERENCES:

Davies, R.A.; Drummond, J. *J. Brit. Nucl. Ener. Soc.*, 1973, *12*, 427-435.

COMPONENTS:	EVALUATOR:
(1) Plutonium; Pu; [7440-07-5] (2) Potassium; K; [7439-09-7] or	H.U. Borgstedt, KfK, Karlsruhe, Fed. Rep. of Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1987

CRITICAL EVALUATION:

Solubility determinations of Pu in liquid K were not reported. Schonfeld et al. (1) attempted to produce Pu-K alloys and suggested an essentially complete immiscibility in the solid and liquid states. Their results were confirmed by Bochvar et al. (2), who denied a reaction of solid Pu with K. Due to the strong affinity of Pu to O, PuO_2 or a plutonate of K should be the solid phase in equilibrium with liquid K contaminated with O instead of metallic Pu.

A schematic phase diagram of the Pu-K system was reported in (3), it is analogue to that shown for the Pu-Li system with modifications due to the different melting and boiling points of K.

Davies and Drummond (1) determined a solubility of PuO_2 in liquid K-Na(80 mol %) alloy at 873 K from mixed PuO_2-UO_2 fuel. The value which they obtained (in the order of 10^{-8} mol % Pu) seems to be acceptable. However, it is difficult to define the equilibrium phases of this multicomponent system (see the data sheets for the Pu-Na system). The solubility of PuO_2 in pure liquid K should be lower than in the investigated liquid K-Na alloy, according to general tendencies observed in other systems. Adamson et al. (5) were not able to establish the stoichiometry of a potassium plutonate, the most probable solute in the apparent Pu-K(-O) system.

References

1. Schonfeld, F.W.; Cramer, E.M.; Miner, W.N.; Ellinger, F.H.; Coffinbery, A.S. *Progr. Nucl. Ener., Ser. V* 1959, *2*, 579.
2. Bochvar, A.A.; Konobeevsky, S.T.; Kutaitsev, V.I.; Menshikova, T.S.; Chebotarev, N.I. *Peaceful Uses of Atomic Energy*, U.N., N.Y., 1958, *6*, 184; *Atom. Energiya* 1958, *5*, 303.
3. Agapova, N.P.; Bochvar, A.A.; Zaimovskii, A.S.; Konobeevsky, S.T.; Kutaitsev, V.I. *Sovetskaya. Atomnaya Nauka i Tekhnika*, Atomizdat, Moskva, 1967, p. 240.
4. Davies, R.A.; Drummond, J.L. *J.Brit.Nucl.Ener.Soc.* 1973, *12*, 427.
5. Adamson, M.G.; Aitken, E.A.; Jeter, D.W. *US Dept.Ener.Rep. CONF-760503-P2*, 1976, p. 866.

COMPONENTS:	EVALUATOR:
(1) Titanium; Ti; [7440-32-6] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1990

CRITICAL EVALUATION:

Preliminary determinations of the solubility of Ti in liquid Li were performed by Jesseman et al. (1), who reported solubilities of $5.0 \cdot 10^{-3}$ and $5.4 \cdot 10^{-2}$ mol % Ti at 1005 and 1289 K. Their results deviate strongly from those published in subsequent studies and their only use is if they are regarded as conditional Ti solubilities at an O content of 0.1 mol % in liquid Li. Moreover, an interaction between the Fe container and the Ti sample via the Li medium cannot be excluded. Anderson and Stephan (15) reported an increase of the Ti solubility from 0.5 to 1.4 mol % Ti at 1005 to 1289 K; their results are significantly overestimated. Since details were not reported, the study is not compiled. Further experiments were performed by Bychkov et al. (2,3). The solubility found by these authors of $2.0 \cdot 10^{-3}$ mol % Ti at 1173 K was uncertain due to a lack of information concerning the purity of Li, which might have been contaminated by O and N. Beskorovainyi et al. (14) reported a marked dissolution of Ti in liquid Li at 1473 K, but did not provide numerical data.

Investigations by Leavenworth et al. (4,5) demonstrated a dependence of the apparent solubility of Ti in liquid Li on the N content in the system. These authors determined an almost regular increase of the solubility of Ti (scatter ± 30 %) from $7 \cdot 10^{-4}$ to $1.7 \cdot 10^{-3}$ mol % Ti in the temperature range of 952-1163 K when Li contained $1.04 \cdot 10^{-2}$ mol % N, which is in fair agreement with the value obtained by (2,3). However, a decrease of the N content to $2.7 \cdot 10^{-3}$ mol % caused a drop of nearly half of the results below the detection limit of $1.4 \cdot 10^{-4}$ mol % Ti. Kelly (6), who worked in the same laboratories as (4,5), and others calculated a linear relation based on these results.

$$\log(soly/\text{mol \% Ti}) = -2.21 - 1840(T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

Such an idealization should be treated cautiously. Since the dependence of the Ti-Li equilibrium on the concentration of N was not investigated in detail, linear extrapolation of the solubility values of Ti at various levels of N to the zero concentration of N cannot be made simply. Addison (10), for example, reported formation of Li_5TiN_3 in this system. The authors of (12) and (13) suggested a formation of TiN; TiC should also be highly stable. Such compounds might be formed, if Ti or Li contain N or C as impurities. A theoretical prediction of the solubility of Ti in liquid Li was expressed by Kuzin et al. (7) in the form:

$$\log(soly/\text{mol \% Ti}) = 4.03 - 7983(T/\text{K})^{-1} \qquad \text{Eq.(2)}$$

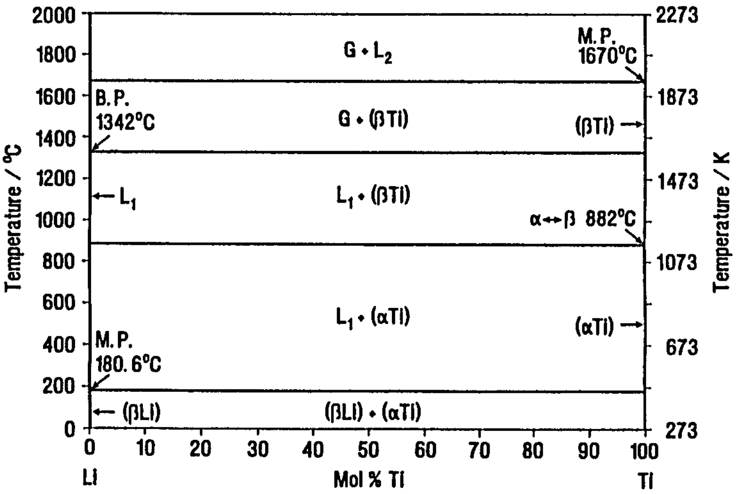
This indicated a temperature dependence of the solubility which is approximately four times steeper than in (6). Nevertheless, the predictions and experiments at $2.7 \cdot 10^{-3}$ mol % N are in acceptable agreement in the temperature range examined in (4,5). Under these circumstances it seems most reasonable [to the evaluators] to fit the experimental results at $1.04 \cdot 10^{-2}$ mol % N (4,5) to the equation:

$$\log(soly/\text{mol \% Ti}) = -1.43 - 1600(T/\text{K})^{-1} \quad \text{with } r = 0.873 \qquad \text{Eq.(3)}$$

Unfortunately the regression coefficient is very poor and the temperature dependence of the solubility is smallest. No dissolution of Ti in liquid Li was observed after an equilibration of 100 h at 1089 K (16). An equilibration of the Mo-Ti-Zr alloy (with 0.5 % Ti) with Li for 1000 hours at 1932 K did not cause an increase of the Ti content in Li which previously contained $1.4 \cdot 10^{-4}$ mol % Ti, as reported in (11). The C and N contents in Li were $8 \cdot 10^{-3}$ and $2 \cdot 10^{-3}$ mol % respectively, further details being not available.

As the solid solubility of Li in Ti is small (9), it might be assumed that the saturated solution of Ti in liquid Li is in equilibrium with pure Ti. The phase diagram of the Ti-Li system supplied by (8) is redrawn and shown in the figure below. The solubility experiments were performed below and above the transformation temperature of Ti at 1155 K. The effect of the transformation was, however, not reflected in the solubility results, most probably due to their scatter.

The tentative value of the solubility of β Ti in liquid Li at $2.7 \cdot 10^{-3}$ mol % N is $2 \cdot 10^{-4}$ mol % Ti at 1173 K.



COMPONENTS:	EVALUATOR:
(1) Titanium; Ti; [7440-32-6] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1990

CRITICAL EVALUATION: (continued)

References

1. Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P. *US Atom. Ener.Comm. Rep. NEPA-1465*, 1950.
2. Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B. *Atom. Energiya* 1959, *7*, 531; *Kernenergie* 1960, *3*, 763.
3. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall. Metalloved. Chist. Met.* 1960, 2, 178; *Metallurgy & Metallography of Pure Metals*, Gordon & Breach, New York, 1962 p. 178.
4. Leavenworth, H.W.; Cleary, R.E. *Acta Metall.* 1961, *9*, 519.
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15. Anderson, R.C.; Stephan, H.R. *US Atom.Ener.Comm. Rep. NEPA-1652*, 1950; as reported in (8)
16. Hoffman, E.E. *US Atom.Ener.Comm. Rep. ORNL-2924*, 1960.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Titanium; Ti; [7440-32-6] (2) Lithium; Li; [7439-93-2]	Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B.; *Atom. Energiya* 1959, *7*, 531-536; *Kernenergie* 1960, *3*, 763-767.
VARIABLES:	**PREPARED BY:**
One temperature: 1173 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ti in liquid Li at 900 °C was determined to be 0.014 mass%. The equivalent value calculated by the compilers is $2.0 \cdot 10^{-3}$ mol % Ti. This result had also been reported in (1,2). It seems that Li could be contaminated with N, thus causing an apparent increase of the Ti solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The inner surface of a Ti crucible was ground, electrolytically polished and etched. The crucible was gradually filled with freshly distilled Li dropping from a stainless steel condenser. After distillation the apparatus was filled with Ar. The crucible was placed in a stainless steel container, to which the cover was welded. Additionally, the crucible was isolated from the steel by a Mo band. The container was placed in an arc furnace and conditioned at 1173 K for 100 hours. The Li solution was cooled to solidification in less than 50 s. The content of Ti in the sample was determined by colorimetric analysis.	Ti: "TG-O" purity; further remelted in a special furnace. Li: containing after distillation: 0.02-0.06 % Na; 0.015 % K; (1-4) 10^{-4} % Fe; 0.002 % Mg (or less); Si, Ni, and Cr were not detected. Ar: "pure". **ESTIMATED ERROR:** Nothing specified. Solubility: precision not better than ± 10 % (compilers). **REFERENCES:** 1. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall. Metalloved. Chist. Met.* 1960, *2*, 178-188. 2. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metallurgy and Metallography of Pure Metals*, Gordon & Breach, New York, 1962, p. 178-188.

COMPONENTS:

(1) Titanium; Ti; [7440-32-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P.
US Atom.Ener.Comm. Rep. NEPA-1465, 1950.

VARIABLES:

Temperature: 1005-1289 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ti in liquid Li at selected temperatures was measured.

t/°C	*soly*/mass % Ti	*soly*/mol % Ti [a]	remarks
732	0.0285, 0.040	$5.0 \cdot 10^{-3}$ (mean value)	α Ti as solute
1016	0.34, 0.40	$5.4 \cdot 10^{-2}$ (mean value)	β Ti as solute

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Strips of a Ti sheet were placed in a pair of Armco Fe capsules. The capsules were loaded with Li in an Ar dry box, degassed and Li was melted in a pot furnace. After welding the capsules were heated in a vacuum furnace at temperatures indicated. The capsules were inserted in stainless steel plates within the furnace, the average temperature for each pair was estimated from the temperature gradient in the plates. The temperature was maintained for a period of 24 hours and the furnace was then air-cooled while still being kept under low pressure. The capsules were weighed and opened. The solidified samples were leached out of the capsules with distilled water, and the Ti remaining undissolved was removed with the capsule, dried, and weighed as the tare to determine the amount of Li solution in the capsule. The leached material was filtered, and the residue spectrographicly analysed for the Ti content.

SOURCE AND PURITY OF MATERIALS:

Ti: purity not specified.
Li: containing 0.24 % O, <0.02 % N, <0.005 % Na; probably contaminated by larger amounts of O and N in the dry box during loading operation.
The capsule: Armco Fe.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: precision ± 15 % (compilers).
Temperature: precision ± 20 K.

REFERENCES:

COMPONENTS:

(1) Titanium; Ti; [7440-32-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Leavenworth, H.W.; Cleary, R.E.

Acta Metall. 1961, *9*, 519-520.

VARIABLES:

Temperature: 952 - 1190 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ti in liquid Li at various temperatures and levels of N content in Li were presented in the figure, they values were read out by the compilers.

T/K	*N content in Li*/mol %	*soly*/mol % Ti	*T*/K	*N content in Li*/mol %	*soly*/mol % Ti
990	$2.7\cdot10^{-3}$	$7\cdot10^{-5}$	1139	$2.7\cdot10^{-3}$	$1.6\cdot10^{-4}$
999	$2.7\cdot10^{-3}$	$<1.4\cdot10^{-4}$	1152	$2.7\cdot10^{-3}$	$1.7\cdot10^{-4}$
1003	$2.7\cdot10^{-3}$	$<1.4\cdot10^{-4}$	1188	$2.7\cdot10^{-3}$	$2.2\cdot10^{-4}$ [a]
1045	$2.7\cdot10^{-3}$	$<1.4\cdot10^{-4}$	1190	$2.7\cdot10^{-3}$	$2.0\cdot10^{-4}$ [a]
1067	$2.7\cdot10^{-3}$	$<1.4\cdot10^{-4}$	952	$1.04\cdot10^{-2}$	$7\cdot10^{-4}$
1093	$2.7\cdot10^{-3}$	$<1.4\cdot10^{-4}$	1018	$1.04\cdot10^{-2}$	$1.2\cdot10^{-3}$
1101	$2.7\cdot10^{-3}$	$<1.4\cdot10^{-4}$	1086	$1.04\cdot10^{-2}$	$1.2\cdot10^{-3}$
1117	$2.7\cdot10^{-3}$	$<1.4\cdot10^{-4}$	1105	$1.04\cdot10^{-2}$	$1.5\cdot10^{-3}$
1124	$2.7\cdot10^{-3}$	$1.6\cdot10^{-4}$	1141	$1.04\cdot10^{-2}$	$1.2\cdot10^{-3}$
1127	$2.7\cdot10^{-3}$	$2.2\cdot10^{-4}$	1163	$1.04\cdot10^{-2}$	$1.7\cdot10^{-3}$ [a]
1131	$2.7\cdot10^{-3}$	$1.6\cdot10^{-4}$			

[a] - β-Ti is the equilibrium solid phase
The results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test equipment was essentially a dipping bucket apparatus, which is in detail described in (1). The specimen cup was heated in a furnace to 811 K and purified Li was added. The cup was then further heated to the desired test temperature for a period of 24 hours. Samples of the saturated solution were removed by means of a sample bucket made of Mo. The sample was allowed to cool and was dissolved in 10 % HCl in a polyethylene beaker at dry ice temperature. HCl dissolved the solute metal traces, which had been adsorbed on the walls of the bucket, the polyethylene beaker minimalized the adsorption of Ti ions after dissolution. Standard colorimetric procedures were applied to determine the solute content, flame photometry to obtain the weight of the Li sample. Agitating the liquid had no effect on the measured solubility. Data obtained at tests with increasing and decreasing temperatures were in excellent agreement. All operations were performed in an Ar atmosphere. Proper amounts of Li_3N were added to establish the N level in liquid Li.

SOURCE AND PURITY OF MATERIALS:

Ti: 99.8 % pure.
Li: 99.8 % pure; containing 0.005 % Na, 0.01 % K, 0.01 % Ca, 0.003 % Cl, 0.01 % N, 0.03 % Fe + Al, 0.07 % heavy metals; further purified by contacting with a Ti sponge at 1144 K for 2 hours, cooled to 260-315 K. $\leq 1\cdot10^{-3}$ % N; post examination indicated a higher N content due to contamination with cover gas and leakage of the apparatus.
Ar: purified.

ESTIMATED ERROR:

Solubility: detection limit $1.4\cdot10^{-4}$ mol % Ti, precision probably ± 50 % or better (compilers).
Temperature: nothing specified.

REFERENCES:

1. Leavenworth, H.W.; Cleary, R.E.; Bratton, W.D. *US Atom.Ener.Comm. Rep. PWAC-356*, 1961.

COMPONENTS:	EVALUATOR:
(1) Titanium; Ti; [7440-32-6] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1990

CRITICAL EVALUATION:

Although Bale et al. (1) presented a predicted phase diagram which is similar to that of the Ti-Li system, knowledge of the Ti-Na system is rather poor. A formation of intermetallics is not expected, and the boiling point of Na (1156 K) is very close to the α-Ti $\leftrightarrow$ β-Ti transition (1155 K) in the Ti-Na system. Corrosion experiments on Ti in liquid Na failed due to an extensive formation of TiO_2 in the test apparatus (2). Aleksandrov and Dalakova (8) reported a solution of Ti in liquid Na after 1 h in contact at 973-1023 K, however, neither quantitative results nor experimental details were given.

The only solubility determinations were performed by Eichelberger et al. (3). The eight solubility experiments, which had been performed with a high purity solute (Ti) and solvent (Na), showed largely scattered results in the range $4.8 \cdot 10^{-5}$ to $7.7 \cdot 10^{-4}$ mol % Ti at temperatures from 873 to 1173 K. As the authors did not observe any regularity in the temperature dependence of the Ti solubility in liquid Na, they did not provide data of single measurements. All results were presented in a subsequent report of the same group (4). The published values were scattered in the range of $2.9 \cdot 10^{-4}$ to $7.7 \cdot 10^{-4}$ mol % Ti and confirmed in a figure supplied in this study. Due to the scatter the influence of the Ti transformation at 1155 K on the Ti solubility in liquid Na could not be analysed.

Kuzin et al. (5) predicted a regular increase of solubility from $1.1 \cdot 10^{-7}$ to $2.0 \cdot 10^{-6}$ mol % Ti for the same temperature range (873-1173 K), which means that the obtained values would be two orders of magnitude lower than the experimental results. The experimental data of Eichelberger (3,4) seem to be influenced by side reactions. Such reactions may cause a formation of a different solid phase which is in equilibrium with the dissolved Ti. The overestimation and the temperature independence of the Ti solubility might be due to this effect. Barker and Wood (6) confirmed the formation of Na_4TiO_4 during the corrosion of Ti in liquid Na containing O. An occurrence of $NaTiO_4$ and $NaTiO_2$ was reported in (7). Solubility values cannot be suggested under these circumstances.

References

1. Bale, C.W. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 1675; *Bull.Alloy Phase Diagr.* 1989, *10*, 138.
2. Taylor, J.R.; Rodgers, S.J.; Williams, H.J. *US Atom.Ener.Comm. Rep. NP-5484*, 1954.
3. Eichelberger, R.L.; Gehri, D.C.; Sullivan, R.J. *Trans. Am. Nucl. Soc.* 1969, *12*, 614.
4. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. AI-AEC-12955*, 1970.
5. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagram Sostoyaniya*; Nauka, Moskva, 1985, p. 113.
6. Barker, M.G.; Wood, D.J. *J. Chem. Soc., Dalton Trans.* 1972, 2451.
7. Lyutyi, E.M.; Bobyk, R.I.; Gomozov, L.I.; Dedyurin, A.J. *Fiz. Khim. Mekhan. Mater.* 1987, *23*, no 1, 35.
8. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Titanium; Ti; [7440-32-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.

US Atom.Ener.Comm. Rep. AI-AEC-12955, 1970.

VARIABLES:

Temperature: 873-1173 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ti in liquid Na at various temperatures was determined:

t/°C	*soly*/mass % Ti	*soly*/mol % Ti [a]
600	$1.29\cdot10^{-3}$	$6.2\cdot10^{-4}$ [b]
600	$1.3\cdot10^{-3}$	$6.2\cdot10^{-4}$ [b]
700	$6.1\cdot10^{-4}$	$2.9\cdot10^{-4}$ [b]
750	$7\cdot10^{-4}$	$3.4\cdot10^{-4}$ [b]
800	$8\cdot10^{-4}$	$3.8\cdot10^{-4}$ [b]
900	$8.3\cdot10^{-4}$	$4.0\ 10^{-4}$ [c]
900	$7.2\cdot10^{-4}$	$3.5\cdot10^{-4}$ [c]
900	$1.6\cdot10^{-3}$	$7.7\cdot10^{-4}$ [c]

[a] - calculated by the compilers [b] - with αTi equilibrium phase [c] - with βTi equilibrium phase

The Fe collector contained less than $5\cdot10^{-4}$ % Ti. The influence of Ti getting into the solution during etching was negligible. A comprehensive version of this study was published in (1). The lowest solubility value is reported to be $4.8\cdot10^{-5}$ mol % Ti therein.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Ti crucible was cleaned with a HF, HNO_3, H_2O (1:3:4) mixture and subsequently washed with H_2O and acetone. A collector fabricated of Fe was cleaned the same way. The crucible-collector assembly was degassed for 2 hours at 350 °C. The assembly was filled with Na and welded under high vacuum. The capsules were equilibrated for 6 hours in a purified Ar atmosphere glove box at the desired temperature. Finally the whole apparatus was inverted in order to cause a flow of Na into the collector part. The entire Na sample was analysed after solidification by means of an unspecified spectrophotometric determination.

SOURCE AND PURITY OF MATERIALS:

Ti: supplied by Mater. Res. Corp., electron beam zone refined, containing 99.97 % Ti, $7.8\cdot10^{-3}$ % C, $4\cdot10^{-4}$ % H, $6.3\cdot10^{-3}$ % O, $6\cdot10^{-4}$ % N, $1.5\cdot10^{-3}$ % Al, $1.2\cdot10^{-3}$ % Cr, $8\cdot10^{-4}$ % Cu, $3.0\cdot10^{-3}$ % Fe, $1.2\cdot10^{-3}$ % Hf, $3.5\cdot10^{-4}$ % Si, Sn, $2.0\cdot10^{-3}$ % Zr, all other elements $\leq 2\cdot10^{-4}$ % each.
Na: purified, containing $8\cdot10^{-4}$ % C, $4\cdot10^{-4}$ % O, $1\cdot10^{-4}$ % Cr, $<1\cdot10^{-4}$ % Ag, Al, B, Be, Cd, Fe, Li, Mn, Mo, Pb, Ta, V, $<5\cdot10^{-4}$ % Ba, Bi, Co, Cu, Mg, Ni, Rb, Sn, Ti, $<1.0\cdot10^{-3}$ % Cs, K, Si, Zr, $<1.0\cdot10^{-2}$ % Zn.
Ar: purified.

ESTIMATED ERROR:

Nothing specified.
Solubility: standard deviation at one temperature ± 15 % (by the compilers).

REFERENCES:

1. Eichelberger, R.L.; Gehri, D.C.; Sullivan, R.J. *Trans. Am. Nucl. Soc.* 1969, *12*, 614.

COMPONENTS:	EVALUATOR:
(1) Titanium; Ti; [7440-32-6] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1990

CRITICAL EVALUATION:

Two determinations of the solubility of Ti in liquid K are of main interest. Ginell and Teitel (1) measured a solubility of $6.7\cdot10^{-3}$ mol % Ti at 1273 K in a first study and provided further solubility results of $6.8\cdot10^{-3}$ and $8.3\cdot10^{-3}$ mol % Ti at 1473 and 1573 K in a second study (2). The results obtained in (2) indicated an unexpected small temperature dependence of the solubility. The influence of increased pressure at higher temperatures on the solubility seems to be marginal, and an influence of Ti transformation at 1155 K could not be detected. According to information given in (5) the influence of Nb or Zr of the capsule material on the solubility of Ti can be regarded as negligible.

In contrast to (1,2), Stecura (3,4) could not precisely determine the Ti dissolved in K in the temperature range of 1020 to 1341 K, since the Ti amounts were below the detection limit of $3\cdot10^{-4}$ mol % Ti of the method. The results gained by (1,2) and (3,4) differ by one order of magnitude. The values determined by Stecura are to be preferred, since the centrifuge technique applied by (1,2) might not have separated effectively some finely dispersed Ti particles from the homogeneous saturated solution of Ti in liquid K and therefore caused an overestimation of the results, though substances of a higher purity had been used in (1,2). Lundberg (7) also confirmed the results of (3,4) in corrosion tests. Aleksandrov and Dalakova (8) did not observe any dissolution of Ti in liquid K after 1 h in contact at 873-923 K. However, no detection limits of the spectral analysis used were specified.

Pure Ti is expected to be in equilibrium with the saturated solution of Ti in liquid K as intermetallic Ti-K compounds have not been established. The Ti-K phase diagram reported by (6) is analogous to that shown for the Ti-Li system; the different boiling points of the alkali metals (1032 K for K) comprise the main differences.

The tentative value of the Ti solubility in liquid K is $3\cdot10^{-4}$ mol % (or less) at 1341 K and constrained pressure to keep K in the liquid state (3,4).

References

1. Ginell, W.S.; Teitel, R.J. *Trans. Am. Nucl. Soc.* 1965, *8*, 393.
2. Ginell, W.S.; Teitel, R.J. *US Atom.Ener.Comm. Rep. SM-48883*, 1965.
3. Stecura, S. *Corrosion by Liquid Metals*, J.E. Draley, J.R. Weeks, Eds., Plenum, New York, 1970, p. 601.
4. Stecura, S. *NASA Rep. TN-D-5093*, 1969.
5. de Boer, F.R.; Boom, R.; Miedema, A.R. *Physica* 1980, *101B*, 294.
6. Bale, C.W. *Binary Alloys Phase Diagrams*,T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 1453; *Bull.Alloy Phase Diagr.* 1989, *10*, 134.
7. Lundberg, L.B. *US Dept.of Ener. Rep. LA-UR-84-2375*, 1984.
8. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Titanium; Ti; [7440-32-6]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Ginell, W.S.; Teitel, R.J.

Trans. Am. Nucl. Soc. 1965, *8*, 393-394.

VARIABLES:

Temperature: 1273-1573 K.

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ti in liquid K was determined.

T/K	*O content*/mass %	*soly*/$\cdot 10^4$/mol % Ti [a]	source
1273	$(7\text{-}11)\cdot 10^{-4}$	55, 64, 82 mean 67	this work
1473	$9\cdot 10^{-4}$	50, 72, 82 mean 68	(1)
1573	$9\cdot 10^{-4}$	33, 107, 109 mean 83	(1)

[a] calculated by the compilers.

The experiments were performed with βTi at constrained pressure.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An L-shaped test capsule fabricated of a Nb-Zr(1%) alloy was placed in a centrifuge. A small dam was installed inside the capsule to regulate the amount of K which might drain into the collector part of the capsule. The capsule containing a Ti sample was filled with K, welded and heated at 100 K above the equilibration temperature for 1 hour, while the centrifuge was rotated to prevent K from flowing over the dam. The test temperature was then kept for 3-6 hours and the rotation rate was increased to force the precipitation of the solute to the bottom of the sample crucible. When the rotation rate was slowly reduced, the solution was decanted and drained into the collector. The method of further chemical analysis was not specified. All operations were performed in an Ar atmosphere chamber.

SOURCE AND PURITY OF MATERIALS:

Ti: 99.9 % purity (or better).
K: purified by hot trap with Ti-Zr(50 %) alloy chips at 1058 K; containing $(7\text{-}11)\cdot 10^{-4}$ % O.
Ar: high purity, dried and passed over Ti-Zr(50 %) alloy chips at 1173 K; containing $\leq 2\cdot 10^{-4}$ mol % H_2O.

ESTIMATED ERROR:

Nothing specified.
Solubility: precision not better than ± 20 % (compilers).

REFERENCES:

1. Ginell, W.S.; Teitel, R.J. *US Atom.Ener.Comm. Rep. SM-48883*, 1965.

COMPONENTS:

(1) Titanium; Ti; [7440-32-6]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Stecura, S.

Corrosion by Liquid Metals, J.E. Draley, J.R. Weeks, Eds., Plenum, New York, 1970, p. 601-611.

VARIABLES:

Temperature: 1020-1341 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ti in liquid K was determined at several temperatures.

T/K	*soly*/$\cdot 10^4$/mass % Ti	*soly*/mol % Ti [a]	Conditions
1341	<4, <4	$<3 \cdot 10^{-4}$	β Ti; constrained pressure
1332	<4, <4	$<3 \cdot 10^{-4}$	β Ti; constrained pressure
1245	<4, <4	$<3 \cdot 10^{-4}$	β Ti; constrained pressure
1115	<4, <4	$<3 \cdot 10^{-4}$	α Ti; constrained pressure
1020	4, <4	$<3 \cdot 10^{-4}$	α Ti; constrained pressure

[a] calculated by the compilers.

The results were also reported (1).

If K was doped with KNO_3, the Ti surface was covered with Ti_2N and TiN impeding the influence of N and O on the dissolution process. Also Ti_2O and TiO were formed at 1020 K, if the system was exclusively contaminated with O, as confirmed by x-ray diffraction data.

Additional data with K, doped with 0.5 mass % KNO_3 (0.2 mol %, as calculated by the compilers), was reported in (1).

T/K	*soly*/mass % Ti	*soly*/mol % Ti [a]	conditions
1020	$9.5 \cdot 10^{-3}$; $1.2 \cdot 10^{-2}$	$7.2 \cdot 10^{-3}$; $9.0 \cdot 10^{-3}$	α Ti
1132	$6.0 \cdot 10^{-3}$	$4.5 \cdot 10^{-3}$	α Ti; constrained pressure
1282	$2.0 \cdot 10^{-3}$; $3.0 \cdot 10^{-3}$	$1.4 \cdot 10^{-3}$; $2.0 \cdot 10^{-3}$	β Ti; constrained pressure

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The potassium and the test crucible made of Ti were sealed in a Nb capsule serving as a container for the crucible and K. The capsule assembly was heated to the desired temperature as controlled by a Pt/Pt-Rh (10 %) thermocouple and equilibrated for 24 hours. The furnace was inverted to cause the saturated K solution to drain away from the test crucible into the collecting capsule. After cooling to room temperature the apparatus was removed from the furnace and cut open. The sample was dissolved in butyl alcohol, the inside wall of the capsule was leached with HCl to remove the solute that precipitated on cooling. The Ti concentration was determined by optical spectrographic and wet chemical analysis.

SOURCE AND PURITY OF MATERIALS:

Ti: 99.7+ % pure, containing 0.0587 % O.
Nb: 99.9 % pure, containing 0.0013 % O.
K: containing <0.002 % O.

ESTIMATED ERROR:

Solubility: nothing specified, analytical detection limit $3 \cdot 10^{-4}$ mol % Ti.
Temperature: accuracy ± 8 K.

REFERENCES:

(1) Stecura, S. *NASA Rep. TN-D-5093*, 1969.

COMPONENTS:

(1) Titanium; Ti; [7440-32-6]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Young, P.F.; Arabian, R.W.

US Atom.Ener.Comm. Rep. AGN-8063, 1962.

VARIABLES:

Temperature: 1033-1363 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ti, or more precisely the Ti content in liquid Rb in equilibrium with the Mo-½Ti alloy at various temperatures, was presented in a figure; the corresponding values were read out by the compilers.

t/°F	T/K [a]	*O content*/mass %	*soly*/$\cdot 10^4$/mol % Ti [a]	conditions
1400	1033	$5.3 \cdot 10^{-3}$	4, 4, 5, 5	α Ti; constrained pressure
1700	1203	$4.9 \cdot 10^{-3}$	2, 2, 2, 4	β Ti; constrained pressure
2000	1363	$5 \cdot 10^{-4}$	5, 7	β Ti; constrained pressure

[a] calculated by the compilers.

COMMENTS AND ADDITIONAL DATA:

These results were also reported briefly in (1). Contact of Ti dissolved in Rb with the Ta sample cup did not influence the determinations, since Ti and Ta did not show affinity (2). As an interaction between Mo and Ti was negligible (2), the chemical activity of Ti in the Mo-½Ti alloy had to be lower than unity, the corrected solubility values for unalloyed Ti in liquid Rb are expected to be proportionally higher than those determined. A fitting equation could, therefore, not be established. The Ti transformation occurring at 1115 K is not clearly reflected in these solubility data. The predicted phase diagram of the Ti-Rb system reported in (3) did not indicate a formation of intermetallic compounds. The Ti-Rb phase diagram is analogue to that reported for the Ti-Li system, with the difference in the boiling points of the alkali metals (961 K for Rb).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Mo-½Ti alloy sample was degreased with acetone, pickled in a mixture of 30 cm^3 lactic acid, 10 cm^3 HNO_3, 2.5 cm^3 HF and 37.5 cm^3 H_2O, finally rinsed with H_2O, and dried. The sample capsule was placed in a Ta capsule containing Rb. The capsule was welded in an Ar atmosphere. The temperature was increased and kept at the desired level for 50 hours. Finally, the apparatus was inverted causing Rb with the dissolved Ti to drain into a Ta sample cup. The assembly was then cooled to room temperature. After solidification the O and metal concentration was analysed. The solidified Rb solution was treated with anhydrous hexane, methanol (for the Rb methylation), H_2O and HCl. The Ta sample cup was heated with aqua regia in a water bath for 1 hour and the resultant solution was added to the RbCl solution. The combined solution was taken to dryness. The dry sample was spectroscopically analysed by the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

Mo-½Ti: supplied by Cleveland Tungsten Inc., containing 99.55 % Mo, 0.43 % Ti, 0.02 % C.
Rb: supplied by MSA Research Corp., Callery, containing 0.55 % Cs, 0.056 % K, 0.0079 % Li, 0.0092 % Na, 0.0025 % Fe, $(6-17) \cdot 10^{-4}$ % O, <0.0014 % Ta, 0.0005 % Zr, 0.0014 % W, Ni, $\leq 1 \cdot 10^{-4}$ % Cr, Be, Mo, Ti, V, Co, Mn.

ESTIMATED ERROR:

Solubility: detection limit of Ti $2 \cdot 10^{-4}$ mol %, precision ± 10 %.
Temperature: precision ± 3 K.

REFERENCES:

1. Anonymous *Rep. NASA-SP-41*, 1964, p. 167.
2. de Boer, F.R.; Boom, R.; Miedema, A.R. *Physica* 1980, *101B*, 294.
3. Bale, C.W. *Binary Alloys Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 1945; *Bull. Alloy Phase Diagr.* 1989, *10*, 140.

COMPONENTS:	EVALUATOR:
(1) Titanium; Ti; [7440-32-6] (2) Cesium; Ce; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1990

CRITICAL EVALUATION:

Tepper and Greer (1,10) reported a solubility of Ti in liquid Cs at 1644 K, but it is uncertain if the true equilibration solubility of Ti in liquid Cs had been reached. Since interactions of Ti with Mo (constituent of the solute material), Nb, and Zr from the sampler are very weak or non-existent (4), the activity of Ti might be much lower than unity and, consequently, the true solubility of Ti may be higher than determined. However, stronger gettering features of Ti compared to Mo might further decrease the Ti activity in the alloy. A solubility value can therefore not be recommended. The determination was performed at elevated pressure.

Some determinations were carried out at lower temperatures. A concentration of even 0.6 mol % Ti in Cs was determined in preliminary experiments performed at 672 K after a 48-hour contact of the metals (7). According to further studies by (5,6), approximately $3\cdot10^{-2}$ mol % Ti dissolved in liquid Cs containing $(3\text{-}24)\cdot10^{-2}$ mol % O were determined after a contact of 500 hours. Winslow (6) suggested Ti dissolution in Cs in form of an oxide. As the studies (5-7) were not performed to measure the solubility and further details were not published, these publications are not compiled.

Godneva et al. (8) determined a solubility of Ti in liquid Cs below $5.5\cdot10^{-4}$ mol % Ti in the temperature range of 423 to 573 K. According to (8), Cs containing 0.8 mol % O dissolved $2.2\cdot10^{-3}$ mol % Ti at 573 K.

The low solubility of Ti in liquid Cs was qualitatively confirmed in corrosion tests. A weight change was not observed but a darkening of the Ti surface could be noticed after 100 hours at 773 K (2). An attack of Cs on Ti could not be noticed after 10000 hours of contact at 823 K (9).

The Ti-Cs phase diagram which is assessed in (3) does not indicate a formation of intermetallic compounds in the system. It is similar to that reported for the Ti-Li system, with the difference in the boiling temperature of Cs at 944 K.

References

1. Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-64-327*, 1964; *US Atom.Ener.Comm. Rep. AD-608385*, 1964; *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 323.
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3. Bale, C.W. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed. Am. Soc. Met., Metals Park, 1986, p. 905; *Bull. Alloy Phase Diagr.* 1989, *10*, 132.
4. de Boer, F.R.; Boom, R.; Miedema, A.R. *Physica* 1980, *101B*, 294.
5. Hughes Aircraft Comp. *NASA Rep. SP-41*, 1964, p. 243.
6. Winslow, P.M. *Corrosion* 1965, *21*, 341.
7. Holley, J.H.; Neff, G.R.; Weiler, F.B.; Winslow, P.M. *Electric Propulsion Development*, E.Stuhlinger, Ed., Academic Press, New York, 1963, p. 341.
8. Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.
9. Berry, W.E. *Corrosion in Nuclear Applications*, Wiley, New York, 1971, p. 303.
10. Tepper, F.; Greer, J. *US Air Force Rep. ASD-TDR-63-824*, Pt.I, 1963; *Rep. MSAR-63-61*, 1963.

COMPONENTS:

(1) Titanium; Ti; [7440-32-6]

(2) Cesium; Ce; [7440-46-2]

ORIGINAL MEASUREMENTS:

Tepper, F.; Greer, J.

US Air Force Rep. ADS-TDR-63-824, Pt.I, 1963; *Rep. MSAR-63-61*, 1963.

VARIABLES:

One temperature: 1644 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility, or precisely the content of Ti in liquid Cs after a 10 and 100 hours contact with a Mo-Ti(½%) alloy at 2500 °F is $9 \cdot 10^{-3}$ and $<6 \cdot 10^{-4}$ mass % Ti respectively, or, as calculated by the compilers, $2.5 \cdot 10^{-2}$ and $<1.6\ 10^{-3}$ mol % Ti.
Tepper and Greer (1) applied the same technique but a different sampling crucible material and got a solubility of $1.5 \cdot 10^{-2}$ mass % or $4.2 \cdot 10^{-2}$ mol % Ti, calculated by the compilers, at 1644 K and an equilibration time of 110 hours. The results were lower at shorter equilibration times.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Solubility measurements of Ti in Cs were performed by means of an inverted capsule technique. The test capsule contained two parts: the first made of Mo-Ti(½%) alloy serving as the solute, and the second made of alumina for collecting the samples. The capsule was heated for 100 hours at 1644 K under vacuum, filled with Cs and welded in an Ar atmosphere. After equilibration, the capsule was inverted and cooled. The solidified Cs in the collector part of the capsule was dissolved and the crucible was cleaned with HCl. The joined solutions were evaporated to dryness and analysed by spectrographic methods. An examination of the alumina part of the capsule revealed a complete permeation by a reaction with Cs. Therefore, the sampling part of the crucible used for the second determination (1) was made of a Nb-Zr(1%) alloy, the applied technique being essentially the same.

SOURCE AND PURITY OF MATERIALS:

Mo-Ti(½%): containing 0.5 % Ti, 0.0256 % C, 0.0033 % N, 0.0053 % O.
Alumina: "high purity"; by G.E. Lucalox, containing 0.02 % SiO_2, 0.01-0.04 % Fe_2O_3, 0.1 % MgO, 0.02 % CaO, <0.02 % Na_2O.
Cs: 99.9+ % pure, supplied by Mine Safety Appliances Res., purified by Zr turnings as hot getter in stainless steel vessel, containing 0.0028 % C, 0.0012 % O, 0.0002 % N, <0.001 % Fe.
Ar: purified by hot and cold K-Na bubbler; O and H_2O content monitored, however, unspecified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 3 K.

REFERENCES:

1. Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-64-327*, 1964; *US Atom.Ener.Comm. Rep. AD-608385*, 1964; *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 323.

COMPONENTS:

(1) Titanium; Ti; [7440-32-6]

(2) Cesium; Ce; [7440-46-2]

ORIGINAL MEASUREMENTS:

Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S.

Zh. Prikl. Khim. 1974, *47*, 2177-2180.

VARIABLES:

Temperature: 423-573 K
O concentration in Cs: 0.08 and 0.8 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ti in liquid Cs was determined at various temperatures and O concentrations.

t/°C	*soly*/mass % Ti	*soly*/mol % Ti [a]
150	$<2\cdot10^{-4}$	$<5.5\cdot10^{-4}$
200	$<2\cdot10^{-4}$	$<5.5\cdot10^{-4}$
300	$<2\cdot10^{-4}$	$<5.5\cdot10^{-4}$
300 [b]	$8\cdot10^{-4}$	$2.2\cdot10^{-3}$

[a] as calculated by the compilers.
[b] Cs containing 0.8 mol % O.

Probably, TiO_2 or a mixed oxide of Cs and Ti was formed on the surface of Ti, since the mass of Ti specimens increased during the test.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A specimen of Ti metal was covered with Cs under vacuum and equilibrated for 120 hours in a glass ampoule. The O content in Cs was increased by means of a controlled decomposition of a $KClO_3$-MnO_2 mixture. The ampoule glass did not undergo visible changes. Cs was dissolved in H_2O and volumetrically determined in the resultant hydroxide. An aliquot of the solution was treated with an acid. The remaining part was treated with ascorbic acid in an acetate buffer mixture, HCl was subsequently added until the colour of Congo red is changed from red to violet. After an addition of chromotropic acid, the resulting solution was colorimetrically analysed to determine the Ti content (1).

SOURCE AND PURITY OF MATERIALS:

Ti: containing 0.01 % Si, 0.17 % Fe, 0.54 % O, 0.05 % Mn, 0.07 % Ni, 0.009 % Mg.
Cs: 98-99 % pure, vacuum distilled, finally containing <0.01 % O, <1.5 % Rb.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Godneva, M.M.; Vodyannikova, R.D. *Zh. Anal. Khim.* 1965, *20*, 831-836.

COMPONENTS:	**EVALUATOR:**
(1) Zirconium; Zr; [7440-67-7] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1990

CRITICAL EVALUATION:

Four different sets of determinations of the solubility of Zr in liquid Li were published by Bychkov et al. (1,2), Jesseman et al. (3), Anderson and Stephen (4), and most recently Blecherman et al. (5,6). The materials applied in (5,6) were of highest purity. However, the authors reported only one preliminary result of $\leq 4.3 \cdot 10^{-4}$ mol % Zr at 1573 K. The solubility data published in (1,2) and (3) are in agreement, considering that the scatter of data at some temperatures in (1,2) is above ± 50 %. As the original of (4) is not available, the results are cited in different ways in secondary sources: Koenig (7) quoted values of $8 \cdot 10^{-4}$ and $8 \cdot 10^{-2}$ mol % Zr at 755 and 1033 K, respectively, McKisson et al. at first (8) cited 10 times lower values, while more recently (9) the same as (7). Borgstedt and Mathews (10) reported an agreement of the values of (1,2) and (4), thus indicating that the original data were 100 times lower than those published in (7). Bale (11) describes the results of (4) in a different form: >0.1 mol % Zr at 753 K, 1 mol % Zr at 1005 K, and slightly less than 1 mol % Zr at 1366 K. The data of (1,2) and (3) might be expressed by the fitting equation, differing from that given in (10):

$$\log (soly/\text{mol \% Zr}) = 0.467 - 3933\ (T/\text{K})^{-1} \qquad r = .897 \qquad \text{Eq.(1)}$$

The scatter of the solubility results is too high to reflect the Zr phase transformation at 1136 K. It should be kept in mind that the solubility values were obtained in Li contaminated significantly with O (0.1 mol % in (3)) and unspecified amounts of N and C. All these elements are strongly gettered by Zr (12), thus influencing the measured Zr content in the saturated solution in liquid Li. The influence of N seems to be most critical, since ZrO_2 in liquid Li containing N decomposes forming ZrN (13). Hoffman (14) observed a severe attack of liquid Li on a ZrO_2 sample after 100 h at 1089 K. He observed also that Zr was uniformly dissolved in Li at 1073 K for 100 h, if the equilibration was performed in a Fe container. This was not the case in experiments under the same parameters in a Zr container. Thus, it is obvious that Fe disturbs the equilibration in the Zr-Li system due to the formation of Zr-Fe intermetallics. The results of (3) have, therefore, to be considered as questionable. An increase of the Zr content in liquid Li (being formerly $2 \cdot 10^{-4}$ mol % Zr) could not be determined by (15) after an equilibration of a Mo-Ti-Zr alloy (with 0.08 % Zr) for 1000 hours at 1932 K. The C and N contents in Li were $1.7 \cdot 10^{-2}$ and $4 \cdot 10^{-3}$ % respectively, but further details were not available.

Kuzin et al. (16) predicted the solubility equation in the form:

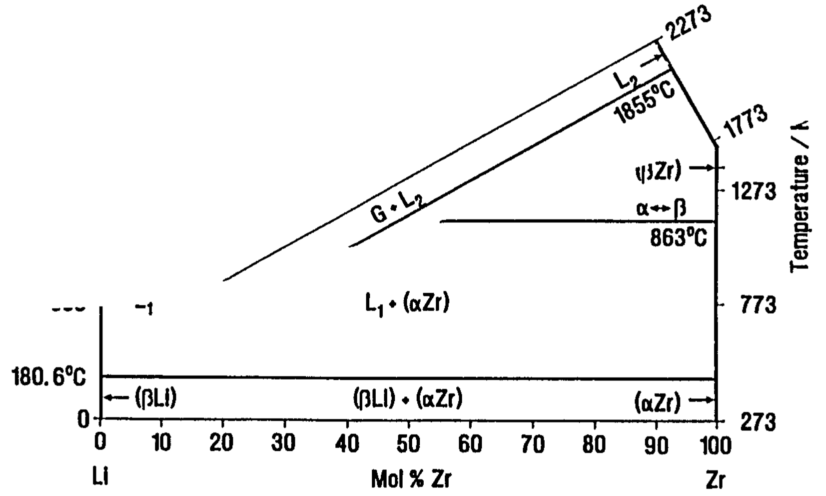
$$\log (soly/\text{mol \% Zr}) = 3.10 - 7197\ (T/\text{K})^{-1} \qquad \text{Eq.(2)}$$

The solubility results obtained by (1,2) and (3) are scattered along this line. Saunders (17) observed a good agreement of his thermodynamic modelling with the experimental values of (1,2) and (3). This would indicate an underestimation of results in (5,6) and an overestimation in (4).

The phase diagram of the Zr-Li system by (11) is redrawn in the figure. A formation of Zr-Li intermetallics was not observed. The saturated solution of Zr in liquid Li is practically in equilibrium with pure Zr, if an influence of impurities can be assumed as negligible.

Tentative values of the Zr solubility in liquid Li at ≥ 1.10-3 mol % N

T/K	*soly*/mol % Zr	source	remarks
973	$3 \cdot 10^{-4}$	(1,2),(3); Eq.(1)	for α Zr
1073	$6 \cdot 10^{-4}$	Eq.(1)	for α Zr
1173	$1 \cdot 10^{-3}$	(1,2); Eq.(1)	for β Zr
1273	$2 \cdot 10^{-3}$	(1,2),(3); Eq.(1)	for β Zr
1473	$2 \cdot 10^{-2}$	(1,2)	for β Zr



COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Lithium; Li; [7439-93-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
October 1990

CRITICAL EVALUATION: (continued)

References

1. Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B. *Atom. Energiya* 1959, *7*, 531; *Kernenergie* 1960, *3*, 763.
2. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall. Metalloved. Chist. Met.* 1960, no 2, 178; *Metallurgy and Metallography of Pure Metals*, Gordon & Breach, New York, 1962, p. 178.
3. Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P. *US Atom.Ener.Comm. Rep. NEPA-1465*, 1950; *Rep. PB-160750*, 1950.
4. Anderson, R.C.; Stephan, H.R. *US Atom.Ener.Comm. Rep. NEPA-1652*, 1950.
5. Blecherman, S.S.; Schenck, G.F.; Cleary, R.E. *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 48; *US Atom.Ener.Comm. Rep. CONF-650411-4*, 1965.
6. Cleary, R.E.; Blecherman, S.S.; Corliss, J.E. *US Atom.Ener.Comm. Rep. TIM-850*, 1965.
7. Koenig, R.F. *US Atom.Ener.Comm. Rep. KAPL-982*, 1953.
8. McKisson, R.L.; Eichelberger, R.L.; Dahleen, R.C.; Scarborough, J.M.; Argue, G.R. *NASA Rep. CR-610*, 1966; *Rep. AI-65-210*, 1965.
9. Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969, *Rep. AI-68-110*, 1968.
10. Borgstedt, H.U.; Mathews, C.K. *Applied Chemistry of the Alkali Metals*, Plenum, New York, 1986, 181.
11. Bale, C.W. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 1509; *Bull. Alloy Phase Diagr.* 1987, *8*, 48.
12. Smith, D.L.; Natesan, K. *Nucl. Technol.* 1974, *22*, 392.
13. Barker, M.G.; Alexander, I.C.; Bentham, J. *J.Less-Common Met.* 1975, *42*, 241.
14. Hoffman. E.E. US *Atom.Ener.Comm. Rep. ORNL-2924*, 1960.
15. DeMastry, J.A.; Griesenauer, N.M. *Trans. Am. Nucl. Soc.* 1965, *8*, 17.
16. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113.
17. Saunders, N. *Z. Metallkde.* 1889, *80*, 894.

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P.
US Atom.Ener.Comm. Rep. NEPA-1465, 1950.

VARIABLES:

Temperature: 972-1239 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zr in liquid Li was determined.

t/°C	*soly*/mass % Zr	*soly*/mol % Zr [a]
699	$(6.5\pm0.5)\cdot10^{-3}$	$5\cdot10^{-4}$
966	$(2.5\pm0.5)\cdot10^{-2}$	$1.9\cdot10^{-3}$

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Strips of a Zr sheet were placed in a pair of Armco Fe capsules. The capsules were loaded with Li in an Ar dry box, degassed and Li was melted in a pot furnace. After welding the capsules were heated in a vacuum furnace at 699 °C for 24 and at 966 for 100 hours. The capsules were inserted in stainless steel plates within the furnace, the average temperature for each pair was estimated from the temperature gradient in the plates. Finally, the furnace was air-cooled while still being kept under low pressure.

The capsules were weighed and opened. The solidified samples were leached out of the capsules with distilled water, and the Zr remaining undissolved was removed with the capsule, dried, and weighed as the tare to determine the amount of Li solution in the capsule. The leached material was filtered, and the residue spectrographicly analysed for the Zr content.

SOURCE AND PURITY OF MATERIALS:

Zr: purity not specified.
Li: containing 0.24 % O, <0.02 % N, <0.005 % Na; probably contaminated by larger amounts of O and N in the dry box during loading operation.
The capsule: Armco Fe.
Ar: unspecified

ESTIMATED ERROR:

Solubility: precision ± 20 % (by the compilers).
Temperature: precision ± 20 K.

REFERENCES:

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Cleary, R.E.; Blecherman, S.S.; Corliss, J.E.

US Atom.Ener.Comm. Rep. TIM-850, 1965.

VARIABLES:

One temperature: 1573 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Preliminary data obtained at 1300 °C indicate a Zr content of 33 μg Zr in 2g of liquid Li. As some volatization of Zr could not be observed, which would have caused deposition of Zr on the W receiver cup, the compilers estimate a probable solubility range of $1.3 \cdot 10^{-4}$ to $4.3 \cdot 10^{-4}$ mol % Zr on the basis of the reported amount of 80 μg Zr deposited on the receiver. The chemical activity of Zr in the equilibrated solid was below unity.
The authors reported in (1) that the solubility of Zr at 1370 °C is of the same order of magnitude as that of Nb in liquid Li. The experimental method applied for both systems seems to be identical.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Zr crucible was installed inside a W receiver capsule. The capsule was covered with a Mo wrapper foil in order to protect it from oxidation. The whole apparatus was placed in an Ar dry box. The temperature of the capsule was kept constant for 50 hours. The capsule was inverted, cooled to room temperature, cut open, and the Li solution was finally dissolved in H_2O. The W receiver was etched with HNO_3-HF solution. Both solutions were spectrographicly analyzed for the Zr content.

SOURCE AND PURITY OF MATERIALS:

Zr: containing $6.0 \cdot 10^{-2}$ % O, $7.5 \cdot 10^{-3}$ % N, and $3.5 \cdot 10^{-3}$ % C.
Li: purified by contacting with Ti sponge for 24 hours at 1144 K in a stainless steel container; then cooled to 798 K; containing $<1.5 \cdot 10^{-3}$ % N, $6 \cdot 10^{-2}$ % O, $<1 \cdot 10^{-4}$ % Nb.
Ar: pure; containing $<3 \cdot 10^{-4}$ % O, $<4 \cdot 10^{-4}$ % H_2O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Blecherman, S.S.; Schenck, G.F.; Cleary, R.E. *US Atom. Ener.Comm. Rep. CONF-650411*, 1965, p. 48; *US Atom.Ener.Comm. Rep. CONF-650411-4*, 1965; *US Atom.Ener.Comm. Rep. CNLM-6335*, 1965.

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B.;

Atom. Energiya 1959, *7*, 531-536; *Kernenergie* 1960, *3*, 763-767.

VARIABLES:

Temperature: 973-1473 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zr in liquid Li at various temperatures was presented in the figure. The values are read out and recalculated to mol % by the compilers.

t/°C	*soly*/mass % Zr	*soly*/mol % Zr
700	$3\cdot10^{-3}$	$2\cdot10^{-4}$
850	$3.5\cdot10^{-2}$	$2.7\cdot10^{-3}$
850	$4.5\cdot10^{-2}$	$3.4\cdot10^{-3}$
900	$(1-1.4)\cdot10^{-2}$ [a]	$(0.8-1.1)\cdot10^{-3}$
900	$8\cdot10^{-3}$	$6\cdot10^{-4}$
985	$8\cdot10^{-3}$	$6\cdot10^{-4}$
1000	$1.0\cdot10^{-2}$	$7.6\cdot10^{-4}$
1000	$2.5\cdot10^{-2}$	$1.9\cdot10^{-3}$
1000	$4.5\cdot10^{-2}$	$3.4\cdot10^{-3}$
1010	$8\cdot10^{-3}$	$6\cdot10^{-4}$
1010	$4.2\cdot10^{-2}$	$3.2\cdot10^{-3}$
1200	$1.5\cdot10^{-1}$	$1.1\cdot10^{-2}$
1200	$4.5\cdot10^{-1}$	$3.4\cdot10^{-2}$

[a] numerical results obtained by a determination with a Zr^{95} isotope.

The same results were also reported in (1). An interaction of the dissolved Zr with solid Nb was not observed. However, Zr reacted with Fe and Ni of the crucible material, thus forming eutectic alloys. This may further increase the apparent solubility of Zr.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The inner surface of a Zr crucible was ground, electrolytically polished and etched. The crucible was gradually filled with freshly distilled Li dripping from a stainless steel condenser. After distillation the apparatus was filled with pure Ar. The crucible was placed in a stainless steel container, to which the cover was welded. Additionally, the crucible was isolated from the steel by a Mo band. The container was placed in an arc furnace and conditioned at a selected temperature for 100 hours. The Li solution was cooled to solidification in less than 50 s. The content of Zr in the sample was determined by colorimetric analysis. The Zr content in liquid Li, which had been equilibrated with Zr powder in the Nb crucible, was determined by means of a Zr^{95} radionuclide.

SOURCE AND PURITY OF MATERIALS:

Zr: 99.9 % pure.
Li: containing after distillation: $(2-6)\cdot10^{-2}$ % Na, 0.015 % K, $(1-4)\cdot10^{-4}$ % Fe, $\le 2.5\cdot10^{-3}$ % Mg; Si, Ni, and Cr were not detected.
Ar: "pure".

ESTIMATED ERROR:

Nothing specified.
Solubility: precision not better than one order of magnitude (compilers).

REFERENCES:

1. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall. Metalloved. Chist. Met.* 1960, *2*, 178-188; *Metallurgy and Metallography of Pure Metals*, Gordon & Breach, New York, 1962, p. 178-188.

COMPONENTS: (1) Zirconium; Zr; [7440-67-7] (2) Sodium; Na; [7440-23-5]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1995

CRITICAL EVALUATION:

Bowman et al. (1,2) did not observe a significant solubility of Zr in liquid Na at temperatures up to 873 K. The method and the detection limit of analysis were not further specified. Taylor et al. (3) reported a Zr content of $4.5 \cdot 10^{-5}$ mol % in liquid Na at a temperature of 722 to 739 K, when both metals had been in contact for 720 hours, without giving more details. Aleksandrov and Dalakova (4) observed no dissolution of Zr in liquid Na after an equilibration of 1 h at 973-1023 K; a spectral analytical method of unspecified detection limits was used. A concentration of $5 \cdot 10^{-4}$ mol % Zr was determined by Fleitman et al. (5) after refluxing liquid Na in a capsule made of Nb-Zr (1%) for 720 hours at 1477 K, if the O level was below $1.4 \cdot 10^{-3}$ mol %. Further details were not reported. Humphreys (6) determined a single solubility value of $2 \cdot 10^{-6}$ mol % Zr at 995 K. Neutron-activated Zr foils were used for the determination. Further details were not reported. The author concluded that the true solubility of Zr in liquid Na is probably higher than the apparent one due to an interference of impurities in Na on the radioactivity level. This value is close to the theoretical prediction of Kuzin et a. (7).

Ewing et al. (8) and subsequently Kovacina and Miller (9) determined the Zr concentration after an equilibration of a Nb-Zr(1%) alloy with liquid Na at temperatures between 1075 and 1653 K. A special interaction between Nb and Zr could not be observed, yet the obtained solubility value may be lower than the true solubility of Zr in liquid Na, as the concentration of Zr in the alloy is small and Zr is a stronger getter of non-metallic impurities in Na than Nb. The experiments were performed under sodium vapor pressure at temperatures above 1163 K. The results of (6) and (8,9) are in fair agreement, though the data obtained by the latter are scattered in a range of up to ± 50 % of the mean value. The data determined at 1075 and 1076 K seem to be overestimated. An equilibration time of 2 hours is certainly too short.

Eichelberger and McKisson (10) performed 3 experiments, which are consistent in themselves but in substantial contrast to the results of (6) and (8,9). Their determinations indicate a regular increase of the Zr solubility from $8 \cdot 10^{-4}$ to $9 \cdot 10^{-3}$ mol % in the temperature range of 873 to 1173 K. A break in the curve log (*soly* Zr) versus T^{-1} may be related to a Zr transformation at 1136 K. The authors considered this increase of solubility to be due to a possible formation of Na_2ZrO_3 in the system. In contrast to the possible formation of Na_2ZrO_3, which had been confirmed by Barker and Wood (11) as well as by Künstler et al. (12,13), Klueh (14) indicated a possible formation of Mo_2Zr in Na containing O, if the dissolved Zr was in contact with the Mo container, as was the case at the experiments performed by Kovacina et al. (8,9). Künstler (15) determined the Zr solubility as $4 \cdot 10^{-5}$ mol % Zr at 973 K, applying ZrO_2 for equilibration. The equilibrium was reached after 10 days, although the concentrations were only slightly higher at shorter equilibration times. Hoffman (16) found ZrO_2 to be not resistant to liquid Na at 1089 K after 100 h of contact.

Recently, Künstler and Heyne (17) published a detailed study on the solubility of Zr in liquid Na in the temperature range 773 to 1273 K using Zr, ZrO_2, Na_2ZrO_3 and $BaZrO_3$ as solutes. They observed only a small increase of the solubility of α Zr with temperature, but a larger one for β Zr. The solubility of α Zr did not increase due to the addition of 0.15 mol % O, while smaller additions of NaCN, Na_2CO_3 and NaOH caused an increase. The solubilities of ZrO_2, Na_2ZrO_3 and $BaZrO_3$ in liquid Na were found to be significantly lower than of α Zr. Since α Zr sheets were covered with Na_2ZrO_3 after the equilibration, it can be concluded that O plays an essential role in the dissolution process of the metal in liquid Na. A similar effect can be assumed for C, N, H, and Ba as contaminants in Na. The equilibria were shown to be reached slowly. Thus, the results of (10) appear to be overestimated. An analysis of different parts of the equilibrated Na in (17) revealed an enrichment of Zr or its compounds close to the interfaces Na/steel/Ar and Na/steel (due to segregation during the solidification), and this may cause an overestimation of the results. Though the temperature dependence of the solubility of α Zr is only small, the values obtained by (17) seem to be reasonable at a certain level of O. They can be classified as tentative. They are in rough agreement with those of (3) and (8,9). The abrupt increase of the solubility, observed at higher temperatures by (10) and (17), is likely to reflect the $\alpha \leftrightarrow \beta$ transformation of Zr at 1136 K. Above this temperature β Zr or a Zr compound may be the solid phase in equilibrium with the solution.

Kuzin et al. (7) theoretically predicted a solubility equation in the form:

$$\log (soly/\text{mol \% } \beta \text{ Zr}) = 4.63 - 10830\ (T/\text{K})^{-1}$$

Most of the experimental data are above this line.

A schematic phase diagram of the Zr-Na system (18) indicates that intermetallics had not been formed in this system. The Zr-Na phase diagram is similar to that shown for the Zr-Li system, only differing in the boiling point of the alkali metal, 1156 K for Na.

Tentative solubility values of Zr in liquid Na containing ~ $3 \cdot 10^{-3}$ mol % O

T/K	solute	*soly*/mol % Zr	source	remarks
773	α Zr	$6\ 10^{-5}$	(17)	Zr covered with Na_2ZrO_3
873	α Zr	$7 \cdot 10^{-5}$	(17)	Zr covered with Na_2ZrO_3
973	α Zr	$7 \cdot 10^{-5}$	(17)	Zr covered with Na_2ZrO_3
1073	α Zr	$8 \cdot 10^{-5}$	(17)	Zr covered with Na_2ZrO_3
1173	β Zr	$1.2 \cdot 10^{-3}$	(17)	at Na vapour pressure
1273	β Zr	$2 \cdot 10^{-2}$	(17)	at Na vapour pressure

COMPONENTS: (1) Zirconium; Zr; [7440-67-7] (2) Sodium; Na; [7440-23-5]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1995

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COMPONENTS: (1) Zirconium; Zr; [7440-67-7] (2) Sodium; Na; [7440-23-5]	**ORIGINAL MEASUREMENTS:** Künstler, K. *Akad. Wissensch. DDR Rep. ZfK-340*, 1977, p. 44-46.
VARIABLES: One temperature: 973 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Zr content in a liquid Na solution was examined after equilibrating ZrO_2 with Na for 2 to 25 days. A practically constant concentration level of $(1\text{-}2)\cdot10^{-4}$ mass % Zr (or $(3\text{-}5)\cdot10^{-5}$ mol % Zr as calculated by the compilers) was determined at equilibration times of longer than 10 days at 700 °C. The results were scattered between $4\cdot10^{-4}$ and $1.0\cdot10^{-3}$ mass % Zr at shorter equilibration times.
The same results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

All experiments were performed in a X8CrNiTi 18 10 stainless steel capsule. Zr was introduced in form of ZrO_2 with a ^{95}Zr isotopic tracer. The capsule was filled with a ZrO_2 tablet as well as Na in an inert atmosphere inside a special glass apparatus. The capsule was welded with a plug and conditioned at the selected temperature for at least 2 days. The capsule was then quenched in liquid Na and the central part of Na was taken for analysis.

The Na sample was dissolved in a CH_3OH-H_2O (3:1) solvent. The activity of the deposit precipitated in the resulting solution was counted by means of a Ge(Li) detector after suitable preparation.

SOURCE AND PURITY OF MATERIALS:

ZrO_2: unspecified.
Na: containing $1.5\cdot10^{-3}$ % O, $1.2\cdot10^{-3}$ % C.

ESTIMATED ERROR:

Solubility: precision ± 50 %.
Temperature: nothing specified.

REFERENCES:

1. Künstler, K.; Ullmann, H. *Akad. Wissensch. DDR Rep. ZfK-337*, 1977, p. 46-53.

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Kovacina, T.A.; Miller, R.R.

Naval Res.Lab. Rep. NRL-6051, 1964.

VARIABLES:

Temperature: 1075-1653 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubility values of Zr in liquid Na at various temperatures are reported. As the Nb-Zr (1%) alloy had been equilibrated with liquid Na, the resulting concentrations were no real saturation concentrations. These concentration values were lower than the saturation values due to the lower chemical activity of Zr in the solid alloy.

t/°C	*equilibration time*/h	*soly*/mass % Zr	*soly*/mol % Zr [a]
802	2	$1.72 \cdot 10^{-4}$	$4.3 \cdot 10^{-5}$
803	8	$7.7 \cdot 10^{-5}$	$2.0 \cdot 10^{-5}$
1008	8	$5.7 \cdot 10^{-5}$	$1.5 \cdot 10^{-5}$
1181	8	$9.7 \cdot 10^{-5}$	$2.5 \cdot 10^{-5}$
1185	2	$2.4 \cdot 10^{-4}$	$6.0 \cdot 10^{-5}$
1380	8	$3.6 \cdot 10^{-4}$	$9.1 \cdot 10^{-5}$

[a] calculated by the compilers

All results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility apparatus containing a Nb-Zr(1%) alloy test crucible and a Mo recipient was filled with Na and sealed under Ar atmosphere. It was additionally wrapped with a Ti foil and placed in a pressure furnace. The system was heated to the desired temperature, which was observed and controlled by a Pt/Pt-Rh (20%) thermocouple, and equilibrated for 2 to 8 hours. The apparatus was inverted to cause Na to drain into the recipient. It was opened after cooling to room temperature. Each Na sample was reacted, diluted to volume, and an aliquot titrated to determine the sample size. The Zr solutes were separated from the resultant aqueous solution by means of a coprecipitation with $Fe(OH)_3$. The precipitate was filtered and ignited to Fe_2O_3, in which form the sample was irradiated for 1 hour in the NRL reactor and analysed by the standard comparator technique for ^{97}Zr beta activity. The solubility of Zr in liquid Na could then be calculated.

SOURCE AND PURITY OF MATERIALS:

Zr: Nb-Zr alloy; containing 1 % Zr and 99 % Nb.
Na: vacuum distilled from a Ni still into a Pyrex glass receiver, filtrated through a fine porosity Pyrex glass frit at ~383 K.
Ar: purified by passing through a molecular sieve and a heated Ti sponge.

ESTIMATED ERROR:

Solubility: detection limit $1.5 \cdot 10^{-6}$ mol % Zr.
Temperature: nothing specified.

REFERENCES:

1. Ewing, C.T.; Stone, J.P.; Spann, J.R.; Kovacina T.A.; Miller, R.R. *Naval Res.Lab. Rep. NRL-5964*, 1963.

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.

US Atom.Ener.Comm. Rep. AI-AEC-12955, 1970.

VARIABLES:

Temperature: 873-1173 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zr in liquid Na was determined at three temperatures.

t/°C	*soly*/mass % Zr	*soly*/mol % Zr [a]
600	$3.4 \cdot 10^{-3}$	$8 \cdot 10^{-4}$
750	$7.7 \cdot 10^{-3}$	$1.9 \cdot 10^{-3}$
900	$3.7 \cdot 10^{-2}$	$9 \cdot 10^{-3}$

[a] calculated by the compilers.

The results might be fitted to the equation

$$\log (soly/\text{mass \% Zr}) = 1.43 - 3460\,(T/\text{K})^{-1}$$

as confirmed by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Zr crucible was cleaned with a HF-HNO_3-H_2O (1:3:4) mixture and subsequently washed with H_2O and acetone. A collector fabricated of Fe with a Nb transition piece was cleaned the same way. The crucible-collector assembly was degassed for 2 hours at 623 K. The assembly was filled with Na and welded under high vacuum. The capsules were equilibrated for 6 hours in an Ar atmosphere glove box at the desired temperature. Finally the whole apparatus was inverted in order to cause a flow of Na into the collector part. The entire Na sample was analysed for its Zr content after solidification by means of x-ray fluorescence.

SOURCE AND PURITY OF MATERIALS:

Zr: 99.99 % pure; supplied by Mater. Res. Corp., electron beam zone refined, containing $2.0 \cdot 10^{-2}$ % O, $2.0 \cdot 10^{-3}$ % C, $3 \cdot 10^{-4}$ % H, $2 \cdot 10^{-4}$ % N, $3.0 \cdot 10^{-3}$ % Fe, $4.0 \cdot 10^{-3}$ % Hf, $3 \cdot 10^{-4}$ % Al, $2 \cdot 10^{-4}$ % Cl, all other elements $\leq 1.5 \cdot 10^{-4}$ % (each).
Na: purified, containing $8 \cdot 10^{-4}$ % C, $4 \cdot 10^{-4}$ % O, $1 \cdot 10^{-4}$ % Cr, $<1 \cdot 10^{-4}$ % Ag, Al, B, Be, Cd, Fe, Li, Mn, Mo, Pb, Ta, V, $<5 \cdot 10^{-4}$ % Ba, Bi, Co, Cu, Mg, Ni, Rb, Sn, Ti, $<1.0 \cdot 10^{-3}$ % Cs, K, Si, Zr, $<1.0 \cdot 10^{-2}$ % Zn.
Ar: purified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Künstler, K.; Heyne, H.

Liquid Metal Systems, H.U. Borgstedt, G. Frees, Eds., Plenum, N.Y., 1995, p.311-319.

VARIABLES:

Temperature: 773-1273; form of the solute.

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zr in liquid Na was determined. The equilibrium was reached after 10 days.

t/°C	*soly*/mass % Zr	*soly*/mol % Zr[a]	t/°C	*soly*/mass % Zr	*soly*/mol % Zr[a]
500[b]	$(2.5\pm0.5)\cdot10^{-4}$	$6.3\cdot10^{-5}$	800[b]	$(3.1\pm0.7)\cdot10^{-4}$	$7.8\cdot10^{-5}$
600[b]	$(2.7\pm0.3)\cdot10^{-4}$	$6.8\cdot10^{-5}$	900[c]	$4.7\cdot10^{-3}$	$1.19\cdot10^{-3}$
700[b]	$(2.8\pm0.6)\cdot10^{-4}$	$7.1\cdot10^{-5}$	1000[c]	$1.17\cdot10^{-2}$	$2.9\cdot10^{-2}$

[a] calculated by the compilers [b] α Zr [c] β Zr

The authors formulated a solubility equation which was confirmed by the compilers:

$$\log\,(soly/\text{mol \% Zr}) = -3.929 - 209\,(T/\text{K})^{-1}$$

Analyses showed that Na_2ZrO_3 and a Zr-O compound were formed on the surfaces of Zr sheets. An addition of 0.1 mass % O to Na did not cause any change of the solubility of Zr. Additions of NaCN ($7\cdot10^{-4}$ mass %), Na_2CO_3 ($6\cdot10^{-4}$ mass %) and NaOH ($1.1\cdot10^{-3}$ mass %) caused an increase of the apparent solubility, numerical results were, however not reported.

The solubility of ZrO_2, Na_2ZrO_3 and $BaZrO_3$ in Na was also studied.

t/°C	solute	*soly*/mass % Zr	*soly*/mol % Zr[a]	t/°C	solute	*soly*/mass % Zr	*soly*/mol % Zr[a]
700	ZrO_2	$(4\pm2)\cdot10^{-5}$	$1\cdot10^{-5}$	900	Na_2ZrO_3	$(4\pm2)\cdot10^{-5}$	$1\cdot10^{-5}$
800		$(5\pm3)\cdot10^{-5}$	$1.3\cdot10^{-5}$				
900		$(5\pm3)\cdot10^{-5}$	$1.3\cdot10^{-5}$	600	$BaZrO_3$	$(3\pm2)\cdot10^{-5}$	$8\cdot10^{-6}$
700	Na_2ZrO_3	$(3\pm2)\cdot10^{-5}$	$8\cdot10^{-6}$	700		$(6\pm3)\cdot10^{-5}$	$1.6\cdot10^{-5}$
800		$(4\pm2)\cdot10^{-5}$	$1\cdot10^{-5}$	800		$(9\pm3)\cdot10^{-5}$	$2\cdot10^{-5}$

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed in X8CrNiTi 18 10 stainless steel capsules. They and the Zr sheets were mechanically cleaned, treated with CH_3OH for 2 h, etched for 2-10 min in a mixture of HNO_3, HF and H_2O, rinsed with H_2O for 30 min, dried at 473 K for 24 h and stored in an Ar atmosphere. The Zr sheets and Zr compounds were radioactively labelled. They were placed in capsules of various shapes allowing filtering, decanting and centrifuging. The capsules were filled with Na, sealed with caps, welded together and maintained at given temperature up to 25 days. They were then quenched in liquid N and disassembled. Na samples were sectioned for analyses of different parts of capsules, crucibles or receivers. Na was dissolved in H_2O-CH_3OH (3:1) mixture. The Na content was determined by HCl titration, followed by adding a Zr carrier solution, concentrating to dryness and measuring the residue.

Na samples were also filtered, decanted, centrifuged in order to obtain more information on the solution process. The distribution of Zr was shown by this procedure.

This way was also applied using pellets of the Zr compounds for equilibration. All operations were performed in Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Zr: containing $<7\cdot10^{-3}$ % Al, $<1\cdot10^{-3}$ % Be, $<5\cdot10^{-3}$ % Ca, Si, Ti, $\sim3\cdot10^{-3}$ % Cr, $\sim4\cdot10^{-3}$ % Cu, $<2\cdot10^{-2}$ % Fe, $\sim1\cdot10^{-4}$ % Hf, $<1\cdot10^{-4}$ % Mg, $<5\cdot10^{-4}$ % Mn, $<7\cdot10^{-4}$ % Mo, $(2-4)\cdot10^{-2}$ % O.
ZrO_2: containing $<7\cdot10^{-3}$ % Al, $\sim1\cdot10^{-3}$ % Be, $<5\cdot10^{-3}$ % Ca, $<3\cdot10^{-3}$ % Cr, $\sim4\cdot10^{-3}$ % Cu, $\sim2\cdot10^{-2}$ % Fe, $\sim1\cdot10^{-4}$ % Hf, $<5\cdot10^{-4}$ % Li, Mg, $<1\cdot10^{-2}$ % Mo, Ni, $<9\cdot10^{-3}$ % Si, $\sim5\cdot10^{-3}$ % Ti.
Zr compounds and additives: analytical purity.
Na: pure for analysis, from Merck, with contents of $<1\cdot10^{-3}$ % Fe, $<5\cdot10^{-2}$ % Ca, $1\cdot10^{-2}$ % K, $<5\cdot10^{-4}$ % heavy metals, $1.7\cdot10^{-3}$ % O, $1\cdot10^{-4}$ % C and $3\cdot10^{-5}$ % H.
Ar: containing $<2\cdot10^{-4}$ % O, $<6\cdot10^{-4}$ % H_2O, $<1\cdot10^{-4}$ % CO_2.

ESTIMATED ERROR:

Solubility: sensitivity $1.3\cdot10^{-6}$ mol % Zr; precision better than ± 50 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Zirconium; Zr; [7440-67-7] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1987

CRITICAL EVALUATION:

According to Ginell and Teitel the Zr solubility in liquid K at temperatures of 1273, 1473, and 1573 K is $4.7 \cdot 10^{-3}$ (1), $3.3 \cdot 10^{-3}$ (2), and $3.3 \cdot 10^{-3}$ mol % (2), respectively. The results indicated a significant scatter, yet an increase of solubility with rising temperature could not be observed. Aleksandrov and Dalakova (10) did not observe any dissolution of Zr in liquid K after an equilibration of 1 h at 873-923 K; a spectral analytical method of unspecified detection limits was used.

Litman (3) estimated the Zr content in liquid K to be less than $4 \cdot 10^{-4}$ mol % at 1088 K, if the O concentration in K was in the range of 0.024 and 0.5 mol % and the metals had been equilibrated for 100 hours. The study is not compiled, as experimental details were not reported.

Eichelberger et al. (4) and Stecura (5,6) intended to perform more precise measurements of the Zr solubility in liquid K. According to Stecura (5,6) the Zr solubility did not exceed the detection limit of $4 \cdot 10^{-4}$ mol % Zr in the temperature range 1020-1332 K, reaching this limit at 1341 K. These data were in fair agreement with those estimated by (3). Similar to (1,2) Eichelberger et al. (4) obtained values scattered between $1.2 \cdot 10^{-2}$ and $3.4 \cdot 10^{-3}$ mol % Zr at temperatures from 1173 to 1473 K. A temperature dependence could not clearly be indicated. It seems to be paradox that substances of higher purity had been applied in (1),(2) and (4) compared to (3), (5) and (6).

According to Litman (3) O does not influence the Zr solubility in liquid K at the given levels. As the solubility in K was expected to be lower than in Na, the results by (5,6) at lower temperatures are to be preferred. It should be considered that all reported results were obtained under the vapor pressure of K at the equilibration temperature.

The use of a Nb container for analysis in (1,2) had no effect on the determined solubility, as a specific interaction between Zr and Nb does not exist (8). Klueh (7) reported a possible formation of Mo_2Zr in the reaction of a Mo crucible with liquid K containing measurable concentrations of O, which might have caused a divergence of the results of determinations in (4). A schematic phase diagram of the Zr-K system is reported in (9). It is similar to the Zr-Li phase diagram, a $\alpha Zr \leftrightarrow \beta Zr$ transition at 1136 K occurs at higher temperature than the boiling point of K (1032 K).

Doubtful solubility values of β Zr in liquid K

T/K	$soly$/mol % Zr	source	remarks
1341	$4 \cdot 10^{-4}$	(6)	at the vapour pressure of K at equilibrium
1473	$3 \cdot 10^{-3}$	(2),(4)	at the vapour pressure of K at equilibrium

References

1. Ginell, W.S.; Teitel, R.J. *Trans. Am. Nucl. Soc.*, 1965, *8*, 393.
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10. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Ginell, W.S.; Teitel, R.J.

Trans. Am. Nucl. Soc. 1965, *8*, 393-394.

VARIABLES:

Temperature: 1273-1573 K.

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zr in liquid K was determined.

T/°C	*O content in K*/mass %	*soly* · 10^4/mass % Zr [a]	*mean soly*/mol % Zr [a]	source
1000	$(7-11)\cdot10^{-4}$	58, 115, 156	$4.7\cdot10^{-3}$	this work
1200	$9\cdot10^{-4}$	39, 83, 110	$3.3\cdot10^{-3}$	(1)
1300	$9\cdot10^{-4}$	76, 77	$3.3\cdot10^{-3}$	(1)

[a] calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An L-shaped test capsule fabricated of a Nb-Zr(1%) alloy was placed in a centrifuge. A small dam was installed inside the capsule to regulate the amount of K which might drain into the collector part of the capsule made of vapour deposited W (2). The capsule was chemically etched and vacuum annealed at the maximum temperature of the experiments. The capsule containing a Zr sample was filled with K, welded and heated at 100 K above the equilibration temperature for 1 hour, while the centrifuge was rotated to prevent K from flowing over the dam. The test temperature was then kept for 3 hours and the rotation rate was increased to force the precipitation of the solute to the bottom of the sample crucible. When the rotation rate was slowly reduced, the solution was decanted and drained into the collector. The method of further chemical analysis was not specified. All operations were performed in an Ar-atmosphere chamber. Temperature measurements were performed applying either thermocouples or optical methods.

SOURCE AND PURITY OF MATERIALS:

Zr: 99.9 % purity.
K: purified by hot trap with Ti-Zr alloy chips at 1058 K; containing $(7-11)\cdot10^{-4}$ % O.
Ar: high purity, dried and passed over Ti-Zr alloy chips at 1173 K, containing $\leq 2\cdot10^{-4}$ mol % H_2O.

ESTIMATED ERROR:

Nothing specified.
Solubility: precision not better than ± 40 % (compilers).

REFERENCES:

1. Ginell, W.S.; Teitel, R.J.; *US Rep. SM-48883*, 1965.
2. Teitel, R.J. *Trans. Am. Nucl. Soc.* 1965, *8*, 15.

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Potassium; K, [7440-09-7]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G.

NASA Rep. CR-1371, 1969; *Rep. AI-68-110*, 1969.

VARIABLES:

Temperature: 1173-1473 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zr in liquid K at various temperatures was determined.

t/°C	*soly*/mass % Zr	*soly*/mol % Zr [a]
900	$2.82 \cdot 10^{-2}$	$1.2 \cdot 10^{-2}$
900	$6.4 \cdot 10^{-3}$	$2.8 \cdot 10^{-3}$
1200	$8.0 \cdot 10^{-3}$	$3.4 \cdot 10^{-3}$

[a] calculated by the compilers

A significant difference of the K pressure inside and outside the assembly occurring during rapid quenching might be due to a swelling of the crucible-collector assembly, thus possibly causing a negative coefficient of the temperature dependence of the solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test crucible was produced of a mechanically drilled Zr rod. It was further chemically treated in a HNO_3, H_2SO_4, HF and H_2O mixture. A Mo-Ti(0.5 %) alloy was applied as a collector material. The crucible and the collector were placed in a Mo capsule, degassed under high vacuum at 1573 K, and cooled in an Ar atmosphere. The parts were finally outgassed at 623 K to remove Ar. After filling K into the crucible, the collector with its cup was installed, and the capsule was sealed under high vacuum. The apparatus was heated to the test temperature and kept in a cup-up position for 4 hours. Finally the capsule was removed from the furnace with the cup in a position at the bottom. Thus, the liquid K in the capsule was transferred to the collector. The cooled capsule was cracked open and the collector was separated. A K sample was melted from the collector into a special glassware. It reacted with H_2O vapour in a He atmosphere acidified with HCl and HF. K remaining in the collector was very slowly dissolved in H_2O and subsequently rinsed with HCl. Zr was determined by spectrophotometry after extraction with tri-n-octylphosphine oxide from an aqueous solution. The colour was developed in the an organic phase using pyrocathehol violet in presence of pyridine.

SOURCE AND PURITY OF MATERIALS:

Zr: triple pass electron beam zone refined; supplied by Mater. Res. Corp., containing: $3.0 \cdot 10^{-3}$ % Fe, $6 \cdot 10^{-4}$ % C, $2.1 \cdot 10^{-4}$ % N, $1.25 \cdot 10^{-2}$ % O, $1 \cdot 10^{-4}$ % Ti, $4.0 \cdot 10^{-3}$ % Hf, $3 \cdot 10^{-4}$ % Al, $1.5 \cdot 10^{-4}$ % Ni, Si, $3.3 \cdot 10^{-4}$ % H, $2.0 \cdot 10^{-4}$ Cl, all other elements $\leq 1 \cdot 10^{-4}$ %.
K: unspecified source, gettered and distilled, containing $<1 \cdot 10^{-4}$ % Ag, Al, Be, Bi, Cd, Cr, Cu, Fe, Li, Mg, Mn, Mo, V, $<2 \cdot 10^{-4}$ % Ta, $<5 \cdot 10^{-4}$ % Ca, Si, Sn, Ti, $5 \cdot 10^{-4}$ % Na, O, $<1 \cdot 10^{-3}$ % B, Ba, Zr.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Stecura, S.

Corrosion by Liquid Metals, J.E.Draley, J.R.Weeks, Eds., Plenum, New York, 1970, p. 601-611.

VARIABLES:

Temperature: 1020-1341 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zr in liquid K was determined at various temperatures.

T/K	*soly* · 10^4/mass % Zr	*soly* · 10^{-4}/mol % Zr [a]
1341	10, <10	4, <4
1332	<10, <10	<4
1245	<10, <10	<4
1115	<10, <10	<4
1020	<10, <10	<4

[a] calculated by the compilers

The same results were also reported in (1).

If K was doped with KNO_3, the Zr surface was covered with a Zr nitride impeding the influence of O and N on the dissolution process. However, an increase of the solubility was not recorded.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The potassium and the test crucible made of Zr were sealed in a Nb capsule serving as a container for the crucible with K. The capsule assembly was heated to the desired temperature as controlled by a Pt/Pt-Rh (10 %) thermocouple and equilibrated for 24 hours. The furnace was inverted to cause the saturated K solution to drain away from the test crucible into the collecting capsule. After cooling to room temperature the apparatus was removed from the furnace and cut open. The sample was dissolved in butyl alcohol, the inside wall of the capsule was leached with HCl to remove the solute that precipitated on cooling. The Zr concentration was determined by optical spectrographic and wet chemical analysis.

SOURCE AND PURITY OF MATERIALS:

Zr: 99.7+ % purity, containing $7.22 \cdot 10^{-2}$ % O.
Nb: 99.9 % purity, containing $1.3 \cdot 10^{-3}$ % O.
K: containing $<2 \cdot 10^{-3}$ % O.

ESTIMATED ERROR:

Solubility: nothing specified, obtained results were on the level of the detection limit of $4 \cdot 10^{-4}$ mol % Zr.
Temperature: accuracy ± 8 K.

REFERENCES:

1. Stecura, S. *NASA Rep. TN-D-5093*, 1969.

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Rubidium; Rb; [7440-17-7]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
October 1987

CRITICAL EVALUATION:

The only experimental study of the Zr solubility in Rb was performed by Young and Arabian (1). In fact the Zr content in liquid Rb was determined, indicating that Zr can exist in the metallic as well as the compound form (after reaction with other contaminating elements). As a Nb-Zr(1%) alloy was used as the solute source for Zr, the chemical activity of Zr in the solid alloy was lower compared to pure Zr. However, insignificant interactions in the Nb-Zr system were observed (2). A more significant decrease of the solid Zr activity is certainly due to stronger gettering by Zr compared to Nb for O, N, C contaminants. Alkali metal zirconate (Rb_2ZrO_4) is very stable (4) and may be formed in the Zr-Rb system contaminated with O.
The reported solubility values increase from $7 \cdot 10^{-4}$ to $8.5 \cdot 10^{-3}$ mol % Zr in the temperature range 1033-1363 K at the vapor pressure of Rb at the equilibration temperature. As the scatter of data at 1203 K exceeds one order of magnitude, it seems unreasonable to propose a fitting equation. The solubility data also do not clearly reflect the effect of a Zr transformation at 1136 K.
The formation of intermetallics in the Zr-Rb system is not expected. A schematic partial phase diagram of the system was presented in (3), it is similar to that of the Zr-Li system. The α Zr $\leftrightarrow$ β Zr transformation occurs at higher temperature (1136 K) than the boiling point of Rb at 961 K.

Doubtful solubility values of Zr in liquid Rb from (1)

T/K	*soly*/mol % Zr	remarks
1033	$7 \cdot 10^{-4}$	for α Zr and the vapor pressure of Rb
1363	$8 \cdot 10^{-3}$	for β Zr and the vapor pressure of Rb

References

1. Young, P.F.; Arabian, R.W. *US Atom.Ener.Comm. Rep. AGN-8063*, 1962; abstracted in *NASA Rep. SP-41*, Pt.I, 1964, p. 167.
2. Miedema, A.R.; Niessen, A.K. *CALPHAD* 1983, *7*, 27.
3. Bale, C.W. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am.Soc. Met., Metals Park, 1986, p. 1953; *Bull. Alloy Phase Diagr.* 1987, *8*, 51.
4. Kohli, R. *Material Behavior and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, New York, 1982, p. 345.

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Young, P.F.; Arabian, R.W.

US Atom.Ener.Comm. Rep. AGN-8063, 1962.

VARIABLES:

Temperature: 1033-1363 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zr in liquid Rb at various temperatures was determined.

t/°F	*t*/°C [a]	*soly*/mass % Zr	*soly*/mol % Zr [a]
1400	760	$7 \cdot 10^{-4}$, $7 \cdot 10^{-4}$	$7 \cdot 10^{-4}$
1700	930	$7 \cdot 10^{-4}$, $1.1 \cdot 10^{-3}$, $1.0 \cdot 10^{-2}$	$3.7 \cdot 10^{-3}$ (mean value)
2000	1090	$7 \cdot 10^{-3}$, $1.0 \cdot 10^{-2}$	$8.5 \cdot 10^{-3}$ (mean value)

[a] calculated by the compilers

The results were also presented in (1).

The solubility data are defined as the Zr content in liquid Rb after an equilibration of a Nb-Zr(1%) alloy with Rb at a selected temperature.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A sample of the Nb-Zr alloy was degreased with acetone, pickled in a mixture of HNO_3-H_2SO_4-HF-H_2O (2:2:1:5), rinsed with H_2O and dried. The sample was placed in a Ta capsule (pickled in the same mixture), which had been filled with Rb. The capsule was then sealed in an Ar atmosphere, and heated at the selected temperature for 50 hours. On removal, the capsule was inverted, causing Rb with the dissolved Zr to flow into a sample cup. The cup was cooled to room temperature. After solidification the cup was cut open and its content analysed for O and metals. The Rb sample was treated with anhydrous hexane, CH_3OH (for the Rb methylization), distilled H_2O, and finally HCl. The resulting solution was taken to dry. The Zr content of the solid residue was analysed in the National Spectroscopic Laboratories. The sample cup was treated with aqua regia for 1 hour in a water bath and the resulting solution was added to the solution after dissolution of the sample.

SOURCE AND PURITY OF MATERIALS:

Nb-Zr alloy: supplied by Wah Chang Corp., containing 98.75 % Nb, 1.15 % Zr, $3.0 \cdot 10^{-3}$ % C, $9.4 \cdot 10^{-3}$ % N, $1.8 \cdot 10^{-2}$ % O.
Rb: purified by passing through a micrometallic filter, gettered with a Ti-Zr alloy at 866 K to remove entrained gases; vacuum distilled, filtered into the storage tank. O content after purification $1.9 \cdot 10^{-3}$, $5.5 \cdot 10^{-3}$, $1.1 \cdot 10^{-3}$ at 1033, 1203, 1363 K.

ESTIMATED ERROR:

Solubility: detection limit of $1 \cdot 10^{-4}$ mol % Zr; precision ± 10 to ± 100 %, depending on the temperature (compilers).
Temperature: precision ± 3 K.

REFERENCES:

1. Young, P.F.; Arabian, R.W. *NASA Rep. SP-41*, Pt.1, 1964, p. 167-176.

COMPONENTS: (1) Zirconium; Zr; [7440-67-7] (2) Cesium; Cs; [7440-46-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1988

CRITICAL EVALUATION:

Tepper and Greer reported a solubility of Zr in liquid Cs based on the equilibration of NbZr(1%) with the liquid metal of $1.8\cdot10^{-2}$ and $1.5\cdot10^{-3}$ mol % Zr at 1533 (1) and 1643 (2) K, respectively. For both measurements (1,2) the same technique was used, with the exception of the collector material of the saturated Cs solution: the collector in (1) was alumina, that of (2) was a Mo-Ti alloy. The alumina capsule was not used in (2) because experience in (1) showed that it had been attacked. Since interactions of Zr with Nb (the second solute component) as well as Mo and Ti (the components of the sampling capsule) are rather weak (3) any significant effects of these metals on the real Zr solubility in liquid Cs are not expected. The chemical activity of Zr in the alloy, however, is significantly lower than unity, therefore, the Zr concentration in the equilibrated solution is also lower than the saturation value. The results of the solubility measurements might additionally be influenced by the stronger gettering behavior of Zr compared to Nb concerning O and N. The high stability of the alkali metal zirconate Cs_2ZrO_3 (9) has a comparable effect. The measurements were performed under the vapor pressure of Cs at the equilibration temperature.

Godneva et al. (8) determined the Zr solubility at 473 and 573 K to be $2.0\cdot10^{-3}$ and $2.6\cdot10^{-3}$ mol % Zr, respectively. However, an increased O content in Cs decreased the Zr solubility to $8.7\cdot10^{-4}$ mol % at 573 K. A solubility value is not recommended. However, the value reported in (2) might be preferred, as the test capsule was not affected by the Cs solution.

Some corrosion tests of Zr in liquid Cs confirmed the determined low solubility values. Keddy (4) observed only a darkening of the surface after a 100 hours contact of the elements at 773 K, while Stevens (5) reported the results of corrosion tests in stagnant liquid Cs at 1144 K. According to (5) Zr had not dissolved to any appreciable degree.

The partial phase diagram of the Zr-Cs system was published in (6). It is analogous to that of the Zr-Li system. The α Zr $\leftrightarrow$ β Zr transformation occurs at a higher temperature (1136 K) than the boiling point of Cs at 944 K.

References

1. Tepper, F.; Greer, J. *US Air Force Rep. ASD-TDR-63-824*, Pt.I, 1963; *Rep. MSAR-63-61*, 1963.
2. Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-327*, 1964; *US Atom.Ener.Comm. Rep. AD-608385*, 1964; *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 323.
3. Miedema, A.R.; Niessen, A.K. *CALPHAD* 1983, *7*, 27.
4. Keddy, E.S. *US Atom.Ener.Comm. Rep. LAMS-2948*, 1963.
5. Stevens, H.L. *NASA Rep. TN-D-769*, 1961, p. 93.
6. Bale, C.W. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met.; Metals Park, 1986, p. 908; *Bull. Alloy Phase Diagr.* 1987, *8*, 44.
7. Hoffman, N.J.; Chandler, W.T. *Metall. Soc. Conf.* 1966, *30*, 509.
8. Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.
9. Kohli, R. *Material Behavior and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, New York, 1982, p. 345.

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Tepper, F.; Greer, J.

US Air Force Rep. ADS-TDR-63-824, Pt. I., 1963;
Rep. MSAR-63-61, 1963.

VARIABLES:

Temperature: 1533 and 1644 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility, or precisely the Zr content in liquid Cs after being in contact with a Nb-Zr(1%) alloy at 2300 °F is $1.25\cdot10^{-2}$ mass % or $1.8\cdot10^{-2}$ mol % Zr, as calculated by the compilers.
Tepper and Greer (1) applying the same technique but a different collector material reported a solubility of $1.0\cdot10^{-3}$ mass % or $1.5\cdot10^{-3}$ mol % Zr (as calculated by the compilers) at an equilibration time of 100 hours. The results were lower (or higher) at shorter equilibration times. The value obtained after 100 hours seems to correspond to the true equilibrium.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Solubility measurements of Zr in Cs were performed by means of an inverted capsule technique. The test capsule contained two parts: the bottom made of Nb-Zr(1%) alloy serving as the solute, and the top made of alumina for collecting the samples. The capsule was heated for 100 hours at 1644 K under vacuum, filled with Cs and welded in an Ar atmosphere. After equilibration, the capsule was inverted and cooled. The solidified Cs in the collector part of the capsule was dissolved in CH_3OH and the crucible was cleaned with HCl. The joined solutions were evaporated to dryness and analysed by spectrographical methods. An examination of the alumina part of the capsule revealed a complete permeation by a reaction with Cs. Therefore, the sampling part of the crucible applied in a second determination (1) was made of a Mo-Ti(½%) alloy, the applied technique being essentially the same.

SOURCE AND PURITY OF MATERIALS:

Nb-Zr alloy: containing 99 % Nb, 1 % Zr, $6.9\cdot10^{-3}$ % C, $1.23\cdot10^{-2}$ % N, $1.92\cdot10^{-2}$ % O.
Alumina: high purity, supplied by G.E. Lucalox, containing 0.02 % SiO_2, 0.01-0.04 % Fe_2O_3, 0.1 % MgO, 0.02 % CaO, <0.02 % Na_2O.
Cs: 99.9+ % pure, supplied by Mine Safety Applicances Res., further purified by Zr turnings as hot getter in stainless steel vessel, containing $2.8\cdot10^{-3}$ % C, $1.2\cdot10^{-3}$ % O, $<2\cdot10^{-4}$ % N, $<1\cdot10^{-3}$ % Fe.
Ar: purified by hot and cold K-Na bubbler; O and H_2O content monitored.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 3 K.

REFERENCES:

1. Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-64-327*, 1964; *US Atom.Ener.Comm. Rep. AD-608385*, 1964; *Rep. CONF-650411*, 1965, p. 323.

COMPONENTS:

(1) Zirconium; Zr; [7440-67-7]

(2) Cesium; Ce; [7440-46-2]

ORIGINAL MEASUREMENTS:

Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S.

Zh. Prikl. Khim. 1974, *47*, 2177-2180.

VARIABLES:

Temperature: 473 and 573 K
O concentrations in Cs: 0.08 and 0.8 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zr in liquid Cs at various temperatures and O concentrations was determined.

t/°C	*soly*/mass % Zr	*soly*/mol % Zr [a]
200	$1.4 \cdot 10^{-3}$	$2.0 \cdot 10^{-3}$
300	$1.8 \cdot 10^{-3}$	$2.6 \cdot 10^{-3}$
300 [b]	$6.0 \cdot 10^{-4}$	$8.7 \cdot 10^{-4}$

[a] as calculated by the compilers.
[b] Cs containing 0.8 mol % O.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Zr specimen was covered with Cs under vacuum and equilibrated for 120 hours in a glass ampoule. The O content in Cs was increased by means of a controlled decomposition of a $KClO_3$-MnO_2 mixture. The ampoule glass did not undergo visible changes. Cs was dissolved in H_2O and determined by titration with an acid. The remaining part was treated with HCl and alizarine and was then heated for 3 minutes at 373 K. The Zr content of the resulting solution was determined by colorimetrical analysis (1).

SOURCE AND PURITY OF MATERIALS:

Zr: containing 0.24 % Fe.
Cs: 98-99 % purity, vacuum distilled, finally containing <0.01 % O, <1.5 % Rb as main impurities.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Godneva, M.M.; Vodyannikova, R.D. *Zh. Anal. Khim.* 1965, *20*, 831-836.

COMPONENTS:	EVALUATOR:
(1) Hafnium; Hf; [7440-58-6] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1989

CRITICAL EVALUATION:

Eichelberger et al. (1) published experimental data on the Hf solubility in liquid Li in the temperature range 1288-1873 K under constrain pressure. Zone-refined Hf and an alloy containing 1 mass % Hf were used as the solute sources. The obtained results did not indicate a distinct dependence of the Hf solubility on the solute source or temperature, the values being scattered in the range of $2 \cdot 10^{-5}$ to $4 \cdot 10^{-5}$ mol % Hf.

Kuzin et al. (2) predicted a regular increase of the Hf solubility in liquid Li from $9\ 10^{-13}$ to $3 \cdot 10^{-5}$ mol % Hf at 625 to 1428 K. The results of (1) and (2) were only in agreement with the predictions at approximately 1450 K. The temperature dependence of the solubility is expected to be very steep.

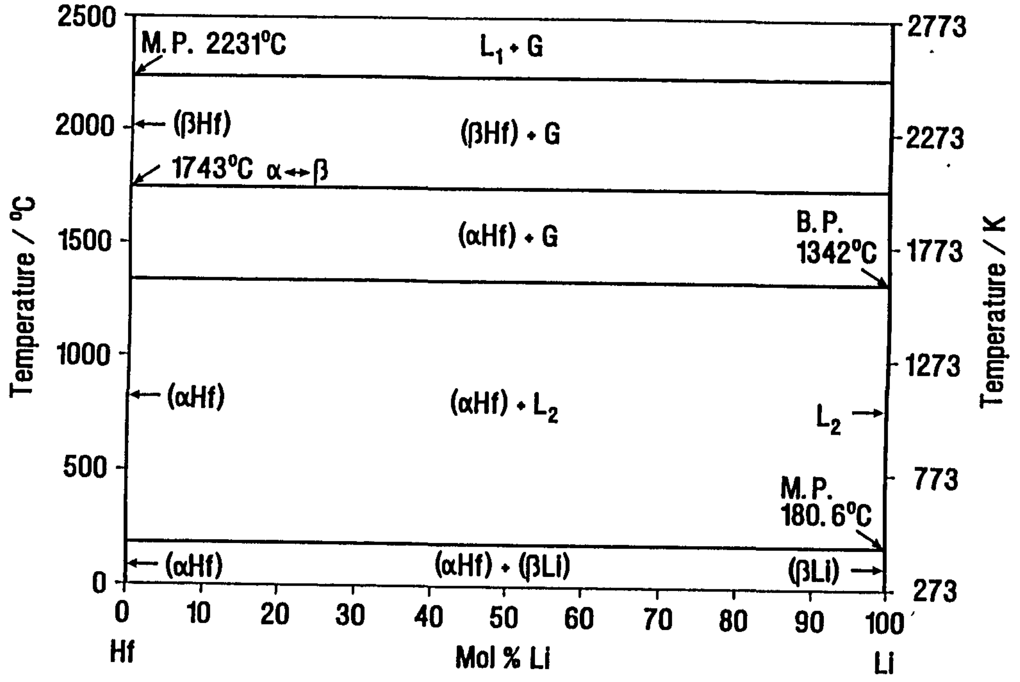
These experimental anomalies might be explained by gettering processes, in which Hf takes up N, O, or C. These processes are superimposed on the true solubility of the pure element Hf, thus causing the negligible temperature dependence.

As reported by Barker et al. (4) HfO_2 in Li is reduced to $LiHfO_2$. The reaction product might be further transformed into HfN (at lower levels) or Li_2HfN_2 in presence of N (0.11-0.53 at %).

The schematic Hf-Li phase diagram reported by Bale (3) does not indicate formation of intermetallics in the system. The diagram is redrawn in the figure.

Doubtful solubility value of Hf in liquid Li taken from (1)

T/K	$soly$/mol % Hf
1473	$2 \cdot 10^{-5}$



References

1. Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969; *Rep. AI-68-110*, 1969.
2. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroenia Diagram Sostoyania*, Nauka, Moskva, 1985, p. 113.
3. Bale, C.W. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 1289; *Bull.Alloy Phase Diagr.* 1989, *10*, 251.
4. Barker, M.G.; Alexander, I.C.; Bentham, J. *J. Less-Common Met.* 1975, *42*, 241.

COMPONENTS:

(1) Hafnium; Hf; [7440-58-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G.

NASA Rep. CR-1371, 1969; *Rep. AI-68-110*, 1969.

VARIABLES:

Temperature: 1288-1873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Hf in liquid Li at various temperatures and with different solute sources was determined.

t/°C	*soly*/mass %	*soly*/mol % Hf [a]	solute
1203	$1.1 \cdot 10^{-3}$	$4 \cdot 10^{-5}$	ASTAR-811C alloy
1410	$8 \cdot 10^{-4}$	$3 \cdot 10^{-5}$	ASTAR-811C alloy
1600	$6 \cdot 10^{-4}$	$2 \cdot 10^{-5}$	ASTAR-811C alloy
1015	$7 \cdot 10^{-4}$	$3 \cdot 10^{-5}$	Hf
1200	$6 \cdot 10^{-4}$	$2 \cdot 10^{-5}$	Hf
1410	$6 \cdot 10^{-4}$	$2 \cdot 10^{-5}$	Hf

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test crucible made of Hf was chemically treated in a HNO_3, H_2SO_4, HF, H_2O mixture. The crucible and a collector fabricated of a Mo-Ti(½%) alloy were placed in a Mo capsule, degassed in high vacuum at 1973 K, and cooled in an Ar atmosphere. The parts were finally outgassed at 623 K to remove Ar. After filling Li into the crucible the collector with its cup were installed, and the capsule was sealed under vacuum. The capsule was heated to the desired test temperature and kept in the cup-up position for 4 hours. Finally, the capsule was removed from the furnace, the cup being then at the bottom. This way the liquid Li in the capsule was transferred to the collector. After cooling the capsule was cracked open, the collector separated, and a Li sample was melted from the collector into a special glassware. The solidified Li was submerged in H_2O in an Ar atmosphere. After completion of the reaction the resulting solution was neutralized with HCl. The collector was rinsed with HF acid and the resulting solution was added to the previous one. Hf was converted to sulfate by fuming with H_2SO_4, and then, further to hydroxide with NH_3. $Fe(OH)_3$ was used as a carrier from the hydroxide. Hf was determined by x-ray spectrography with additions of Lu as an internal standard.

SOURCE AND PURITY OF MATERIALS:

Hf: electron beam zone refined, supplied by Mater. Res. Corp., containing $<8.0 \cdot 10^{-3}$ % C, $<1.0 \cdot 10^{-2}$ % O, Ta, $1.6 \cdot 10^{-3}$ % N, $1.4 \cdot 10^{-2}$ % Fe, $2.0 \cdot 10^{-3}$ % Cr, $<5.0 \cdot 10^{-3}$ % Nb, $<3.0 \cdot 10^{-3}$ % Cu, $<2.0 \cdot 10^{-3}$ % Al, Ni, Si, Ti, Ca, Sn, $<5 \cdot 10^{-4}$ % Co, Mg, Mn, Mo, V, Na, $<1.0 \cdot 10^{-3}$ % W, $1 \cdot 10^{-4}$ % U, $2 \cdot 10^{-4}$ % Cd, $2 \cdot 10^{-5}$ % B.
ASTAR-811C alloy: supplied by NASA Lewis Res. Center, containing 1 % Hf, 90 % Ta, 8 % W, 1 % Re, $2.5 \cdot 10^{-2}$ % C.
Li: supplied by General Electric Corp., hot trapped with a Zr foil getter for 126 hours at 1093 K and distilled; containing $4.4 \cdot 10^{-3}$ % C, $1.3 \cdot 10^{-3}$ % N, $3.3 \cdot 10^{-3}$ % O, $<5.0 \cdot 10^{-3}$ % B, Ba, $<2.5 \cdot 10^{-3}$ % Na, Nb, Pb, Sn, Ti, V, Zr, $<5 \cdot 10^{-4}$ % Ag, Al, Ca, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Sr.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Hafnium; Hf; [7440-58-6] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1987

CRITICAL EVALUATION:

Solubility determinations of Hf in liquid Na have not been reported in the literature. According to predictions of Kuzin et al. (1) the solubility should increase from $9 \cdot 10^{-12}$ to $1.5 \cdot 10^{-6}$ mol % Hf in the temperature range 752 to 1111 K, thus being lower than the solubilities of Ti or Zr at the corresponding temperatures.
Aleksandrov and Dalakova (3) did not detect any dissolution of Hf in liquid Na after an equilibration of 1 h at 973-1023 K; the detection level of the spectral analysis used was not specified.
Bale (2) published a predicted Hf-Na phase diagram analogous to that shown for the Hf-Li system with the difference caused by the differing boiling points of the two alkali metals.

References

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2. Bale, C.W. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 1295; *Bull. Alloy Phase Diagr.* 1987, *8*, 46.
3. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Hafnium; Hf; [7440-58-6] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1987

CRITICAL EVALUATION:

Eichelberger et al. (1) determined Hf solubility in liquid K in the temperature range of 1473-1660 K under the vapor pressure of the liquid metal at the equilibration temperature. A temperature dependence of the solubility was not observed. The obtained values were approximately at $2 \cdot 10^{-3}$ mol % Hf and seem to be overestimated, which is probably due to an extensive additional dissolution of the Hf sample by formation of Hf compounds with O and N in the liquid K metal. No dissolution of Hf in liquid K was observed at 873-923 K after an equilibration of 1 h; the detection level of the spectral analysis used was not specified (3).
Bale (2) reported a schematic phase diagram of the Hf-K system analogous to that of the Hf-Li system (see CRITICAL EVALUATION of the Hf-Li system).

Doubtful solubility value of Hf in liquid K taken from (1)

T/K	*soly*/mol % Hf	remark
1660	10^{-3}	at constrained pressure

References

1. Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969; *Rep. AI-68-110*, 1969.
2. Bale, C.W. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 1289; *Bull. Alloy Phase Diagr.* 1987, *8*, 45.
3. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Hafnium; Hf; [7440-58-6] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1987

CRITICAL EVALUATION:

Experimental results on the Hf-Rb system have not been reported in the literature. According to Bale (1) it may be concluded from its similarity to Ti and Zr that Hf does not form an intermetallic with Rb and that its solubility is not higher than the solubilities of Ti or Zr in liquid Rb at corresponding temperatures; see the Ti-Rb and Zr-Rb systems. A schematic phase diagram of the Hf-Rb system (1) is similar to that of the Hf-Li phase diagram with the difference in the boiling point of Rb at 961 K.

References

1. Bale, C.W. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 1303; *Bull. Alloy Phase Diagr.* 1987, *8*, 47.

COMPONENTS:	EVALUATOR:
(1) Hafnium; Hf; [7440-58-6] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1987

CRITICAL EVALUATION:

Stevens (1) reported results of static corrosion tests with Hf capsules containing liquid Cs at 1144 K, but did not describe solubility experiments. Corrosion attack on Hf after 720 hours contact was not observed.
According to Bale (2) it may be concluded from comparison with Ti or Zr that a formation of intermetallics between Hf and Cs is not expected and the solubility of Hf in liquid Cs is not likely to be higher than the solubilities of Ti or Zr in the same solvent at corresponding temperatures; see the Ti-Cs and Zr-Cs systems. A schematic phase diagram of the Hf-Cs is presented in (2); it is analogous to that of the Hf-Li system.

References

1. Stevens, H.L. *NASA Rep. TN-D-769*, 1961, p. 93.
2. Bale, C.W. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 887; *Bull. Alloy Phase Diagr.* 1987, *8*, 43.

COMPONENTS: (1) Vanadium; V; [7440-62-2] (2) Lithium; Li; [7439-93-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1994

CRITICAL EVALUATION:

Experimental data on V solubility in liquid Li are widely scattered. The data of Jesseman et al. (1) indicated that the observed V solubility decreased from $2\cdot10^{-3}$ to $8\cdot10^{-4}$ mol % V with temperature increasing from 1000 to 1277 K. A possible reaction of V with components of the Armco test container under the experimental conditions of (1) might explain the overestimated result obtained at 1000 K. Beskorovainyi et al. (2) reported that the V solubility at temperatures of 1273 K (or lower) was not higher than the analytical detection threshold of $4\cdot10^{-4}$ mol % V. The solubility values of (1) and (2) were in fair agreement at about 1273 K, being further confirmed by predictions of Kuzin et al. (3), whose solubility value was $8.9\cdot10^{-4}$ mol % V, as calculated by the evaluators from the equation in (3):

$$\log (soly/\text{mol \% V}) = 4.47 - 9580\,(T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

Evtikhin et al. (4) claimed the experimental confirmation of Eq.(1) between 873 and 1073 K using X-ray absorption spectrometry. The presence of N in Li did not show any influence on the solubility of V in these experiments (4). The equilibrium was reached within 1.5 h after the initial increase of the V concentration. Kirillov et al. (5) of the same laboratory measured subsequently the solubility of V in liquid Li by means of spectral analysis, and they expressed their results by a fitting equation:

$$\log(soly/\text{mol \% V}) = -(1.1\pm0.2) - (1900\pm300)(T/\text{K})^{-1} \qquad \text{Eq.(2)}$$

The temperature dependence of the solubility is very small and is in contrast to Eq.(1); the authors did not comment this fact. A solubility of $2.6\cdot10^{-3}$ mol % V at 1273 K can be estimated from Eq.(2). The earlier result in (2), which is one order of magnitude lower, appears to be more reliable. No influence of the N content up to 0.10 and 0.25 mol % on the equilibrium solubility of V was observed by (4) and (4), respectively.

The V solubility in liquid Li was estimated on the basis of corrosion tests to be about $1\cdot10^{-5}$ mol % at 823 K, if the N and C contents in Li were $(1.8\text{-}3.5)\cdot10^{-3}$ and $(0.5\text{-}12)\cdot10^{-4}$ mol %, respectively (6). As further details are not available, this paper is not compiled. Guminski and Borgstedt (7,8) estimated the solubility of V in Li to be $1.3\cdot10^{-7}$ mol % at 823 K from corrosion experiments in flowing Li. Since the purest materials were used in these tests, this value seems to be the most reliable and fits quite well to the theoretical prediction (~ 10^{-7} mol %).

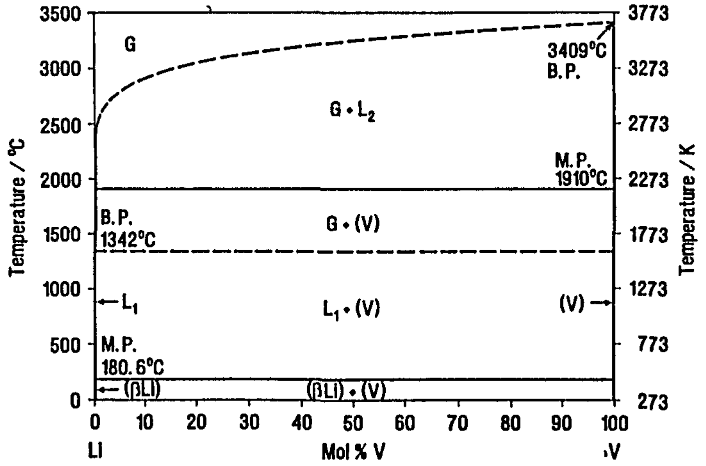
V showed good corrosion resistance to molten Li (9,10). Due to the high stability of Li_2O, the solubility measurements did not seem to be influenced by moderate amounts of O (10,11).

Li reduces VO from the V surface, an influence of N or C was certainly more problematic (6,11). V forms nitrides with N dissolved in Li (12); a solid solution of N in V, V_2N, VN and Li_7VN_4 are formed depending on the chemical activity of N (12,13,14). The solubility of these species in liquid Li seems to be higher than that of pure V, but lower than the detection level of the electrical resistivity technique (~10^{-3} mol % V) at 750 K (14). N diffuses into the bulk of V samples, while C is bound close to the surface. A carbonitride of V is formed predominantly, if both C and N are present in Li (6).

A schematic phase diagram of the V-Li system, based on (15), is redrawn below. Formation of intermetallics was not observed.

Tentative solubility value of V in liquid Li

T/K	*soly*/mol% V	source
823	$1.3\cdot10^{-7}$	(7,8)
1273	$4\cdot10^{-4}$	(1),(2)



COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Lithium; Li; [7439-93-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
July 1994

CRITICAL EVALUATION: (continued)

References

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5. Kirillov, V.B.; Krasin, V.P.; Lyublinskii, I.E.; Kuzin, A.N. *Zh. Fiz. Khim.* 1988, *62*, 3191.
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8. Guminski, C.; Borgstedt, H.U. 5th *Intern. Symp. on Solubility Phenomena*, Russ. Acad. Sci., Moskva, 1992.
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11. Smith, D.L.; Natesan, K. *Nucl. Technol.* 1974, *22*, 392.
12. Klueh, R.L. *Metall. Trans.* 1972, *3*, 2145.
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COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P.
US Atom.Ener.Comm. Rep. NEPA-1465, 1950.

VARIABLES:

Temperature: 1000-1277 K.

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of V in liquid Li was determined at two temperatures:

t/°C	*soly*/mass % V	*soly*/mol % V [a]
727	$2.0 \cdot 10^{-2}$, $1.0 \cdot 10^{-2}$	$(2.0 \pm 0.6) \cdot 10^{-3}$
1004	$6.5 \cdot 10^{-3}$, $6.0 \cdot 10^{-3}$	$(8.5 \pm 0.3) \cdot 10^{-4}$

[a] mean values calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Strips of a V sheet were placed in a pair of Armco Fe capsules. The capsules were loaded with Li in an Ar dry box, degassed and Li was melted in a pot furnace. After welding the capsules were heated in a vacuum furnace for 24 hours. The capsules were inserted in stainless steel plates within the furnace, the average temperature for each pair was estimated from the temperature gradient in the furnace. Finally, the furnace was air-cooled while still being kept under low pressure. The capsules were weighed and opened. The solidified samples were leached out of the capsules with distilled water.

The V remaining undissolved was removed with the capsule, dried, and weighed as the tare to determine the amount of Li solution in the capsule.

The leached material was filtered, and the residue spectrographically analysed for the V content.

SOURCE AND PURITY OF MATERIALS:

V: purity not specified.
Li: containing 0.24 % O, <0.02 % N, <0.005 % Na; possible contamination by larger amounts of O and N in the dry box during loading operation.

ESTIMATED ERROR:

Solubility: precision ± 30 % (by the compilers).
Temperature: stability ± 20 K.

REFERENCES:

COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Beskorovainyi, N.M.; Ioltukhovskii, A.G.; Lyublinskii, I.E.; Vasilev, V.K.
Fiz.-Khim. Mekh. Mater. 1980, no 3, 59-64.

VARIABLES:

Temperature: $\leq$1273 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of V in liquid Li at temperatures equal or lower than 1000 °C is not higher than $3 \cdot 10^{-3}$ mass % V (or $4 \cdot 10^{-4}$ mol % V, as calculated by the compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A cylindrical crucible made of V contained Li inside. It was placed in a sealed chamber having "windows" of Be, which were transparent to x-rays. Li did not contact the Be windows. The capsule was heated in a He atmosphere. A beam of x-rays was passed along the axis of the sample. The radiation passing through the sample was analysed by means of a Soller's spectrometer, its intensity was measured from both sides in relation to the K-boundary of absorption of V.

The concentration of V in the Li sample was calculated using the corresponding equation.

SOURCE AND PURITY OF MATERIALS:

V: electron-beam refined, containing ~ 10^{-2} % O, N, C.
Li: containing $5 \cdot 10^{-3}$ % K, $7.4 \cdot 10^{-2}$ % Na, $1 \cdot 10^{-3}$ % Mn, $6.5 \cdot 10^{-3}$ % Mg, $3 \cdot 10^{-3}$ % Al, O, Si, $7.9 \cdot 10^{-3}$ % Ca, Fe, $1 \cdot 10^{-3}$ % N.

ESTIMATED ERROR:

Solubility: nothing specified; sensitivity of the method $4 \cdot 10^{-4}$ mol % V.
Temperature: stability ± 1 K.

REFERENCES:

COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Kirillov, V.B.; Krasin, V.P.; Lyublinskii, I.E.; Kuzin, A.N.
Zh. Fiz. Khim. 1988, *62*, 3191-3195.

VARIABLES:

Temperature: 873-1073 K
Concentration of N: up to 0.10 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of V in liquid Li was determined, the temperature function of the solubility was fitted by the equation for the range 600 to 800 °C:

$$\log(soly/\text{mol } \% \text{ V}) = -(1900 \pm 300)(T/\text{K})^{-1} - (1.1 \pm 0.2)$$

Individual results were not reported.
The solubilities did not depend on the concentration of N up to 0.20 mass % ((0.10 mol %) in the reported determinations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Li was isothermally equilibrated for 50 hours with V in a container which was not specified. The concentration of N in Li was dotated by the addition of Li_3N. Li was analyzed for its N content by means of Kjeldahl's method and for the V content by a specially elaborated spectral analysis which was not described.

SOURCE AND PURITY OF MATERIALS:

V: not specified.
Li: not specified.

ESTIMATED ERROR:

Solubility: precision about ± 15 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Vanadium; V; [7440-62-2] (2) Lithium; Li; [7439-93-2]	Guminski, C.; Borgstedt, H.U. *Z. Metallk.* 1994, *85*, 771-774.
VARIABLES:	**PREPARED BY:**
One temperature: 823 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of V in liquid Li was estimated to be between $9.6 \cdot 10^{-8}$ and $1.66 \cdot 10^{-7}$ mol % V at 823 K. The mean value is $(1.3 \pm 0.3) \cdot 10^{-7}$ mol % V. These results were also reported in (2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed in a circuit of tubes made of stainless steel. V sheets were placed in the circulating flow of molten Li in unisothermal conditions. The circuit was equipped with an electromagnetic pump which forced the liquid Li through the tubes under turbulent flow conditions. A temperature gradient 865 - 823 K was kept in the circuit. Li was supplied from the dump tank in which it was purified by means of reaction with Ti sponge. Ar was used as inert gas cover. The V sample sheets were weighed before and after the experiments. The solubility of V in liquid Li was calculated on the basis of the equation relating the corrosion rate and the solubility (1).

SOURCE AND PURITY OF MATERIALS:

V: 99.94 % pure from Metallgesellschaft, with contents of $2.45 \cdot 10^{-2}$ % O, < $1 \cdot 10^{-2}$ % N, and $2.2 \cdot 10^{-2}$ % C.
Li: gettered with Ti sponge at 973 K for > 24 h, with contents of < $1 \cdot 10^{-3}$ % O and < $6 \cdot 10^{-3}$ % N.
Ar: purified, with contents of < $1 \cdot 10^{-4}$ % O and < $1 \cdot 10^{-4}$ % H_2O.

ESTIMATED ERROR:

Solubility: precision ± 20 %.
Temperature: precision ± 3 K.

REFERENCES:

(1) Borgstedt, H.U.; Röhrig, H.D. *J. Nucl. Mater.* 1991, *179-181*, 596.
(2) Guminski, C.; Borgstedt, H.U. 5th *Intern. Symp. on Solubility Phenomena*, Russ. Acad. Sci., Moskva, 1992.

COMPONENTS:	EVALUATOR:
(1) Vanadium; V; [7440-62-2] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1994

CRITICAL EVALUATION:

Although the several studies concerning V corrosion in liquid Na (1-7) did not yield consistent results, they provided important material for understanding and formulation of the solubility phenomenon in the V-Na system. Even small amounts of O in liquid Na diffused into solid V, thus inhibiting the dissolution of V. At higher O concentrations the VO formed on the surface did not adhere to metallic V, thus causing an elevation of the V content in liquid Na. However, the majority of V was no longer in the elemental but in the O-bound form. The authors of (5,7) therefore characterized their solubility data as "V content" rather than as "V solubility". In the first experiments (5) performed at 873 and 1073 K V of technical purity was applied. The results were $1.8 \cdot 10^{-3}$ and $9 \cdot 10^{-3}$ mol % V, respectively. Only a negligible dependence of these results on the O amounts added to Na (up to 0.19 mol %) was observed. Experimental details were described only briefly. As the results were overestimated, paper (5) is not compiled. The experimental method used for measurements by (7) at 873 K was basically the same, with the only difference that the high purity V samples were placed in Mo or stainless steel containers. The data were scattered in the range of $1 \cdot 10^{-5}$ and $3 \cdot 10^{-3}$ mol % V, the majority at approximately $5 \cdot 10^{-4}$ mol % V. As in the previous study (5) a slight increase of the V content after an increase of the O concentration from $7 \cdot 10^{-3}$ to 0.6 mol % was observed. In general, the results were not dependent on the container material. Mo did not interact with V as was likely with the components of stainless steel (Cr, Fe, Ni) (8).

Other efforts to determine V solubility in liquid Na were attempted by Eichelberger et al. (9) at temperatures from 873 to 1173 K. The few results were scattered in the range of $5 \cdot 10^{-5}$ and $1 \cdot 10^{-4}$ mol % V and did not indicate a distinct temperature dependence. Babu et al. (10) investigated the solubility at a lower temperature range (557-751 K). An almost regular increase of the V content from $1 \cdot 10^{-6}$ to $7 \cdot 10^{-6}$ mol % V in saturated liquid Na was reported for temperatures up to 646 K. At higher temperatures an abrupt decrease of the solubility of one order of magnitude was noticed, which was explained by Babu et al. (10) as a formation of intermetallics between V and the container material Ta. The presence of 3 mass % Mg in Na was expected to have a negligible effect on the V solubility. Rough agreement of the data of Babu et al. (10) with the average data of (7) can be obtained by extrapolation to higher temperatures, while the data of (9) were reported to be at significantly lower values. The O content was at minimum $5 \cdot 10^{-3}$ mass % (7), only $4 \cdot 10^{-4}$ in (9), and unspecified in (10). Thus a formulation of a general fitting equation based on these solubility data is premature. Guminski and Borgstedt (11) estimated the solubility of $1.2 \cdot 10^{-8}$ mol % at 873 K from dynamic corrosion tests under non-isothermal conditions. Since purest materials were used in these experiments, the result seems to be the most reliable. Aleksandrov and Dalakova (12) did not detect any solubility of V in liquid Na at 973-1023 K after the equilibration of the two metals for 1 h. The sensitivity of their spectral analysis was not specified. According to Kuzin et al. (13) the calculated solubilities of V in liquid Na were significantly lower than all experimental data. It is difficult to attribute the discrepancy between the theoretical and the experimental data to O influence at very low concentrations (below 10^{-4} mol %), since an influence on the V solubility at higher O levels was not observed. Barker and Morrison (14) identified $NaVO_2$ and $NaVO_4$ in the V-Na system, which had been contaminated by O. Investigations by Hooper and Trevillion (15) confirmed the formation of $NaVO_2$. A series of O solid solutions in V ($\alpha,\beta,\gamma,\delta$) were expected; however only the α-phase was found.

A schematic phase diagram of the V-Na system was reported by Smith and Lee (16) and is similar to that shown for the V-Li system. Formation of V-Na intermetallics was not observed.

Tentative solubility values of V in liquid Na

T/K	*soly*/mol % V	source
873	$1 \cdot 10^{-8}$	(11)

References

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8. DeBoer, F.R.; Boom, R.; Miedema, A.R. *Physica B* 1980, *101*, 294.
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12. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad.Nauk SSSR, Met.* 1982, no. 1, 133.
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COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Klueh, R.L.; DeVan, J.H.

J. Less-Common Met. 1973, *30*, 9-24.

VARIABLES:

One temperature: 873 K
Concentration of O: $7\cdot10^{-3}$ - 0.6 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Concentrations of V dissolved in liquid Na at 600 °C after various times of exposure, at various initial O concentrations and in different capsule materials were reported.

Exposure/h	*O concn*/mass %	Capsule	*soly*/mass % V	*soly*/mol % V [a]
100	$2.00\cdot10^{-1}$	Mo	$1.2\cdot10^{-3}$	$5.4\cdot10^{-4}$
200	$1.80\cdot10^{-1}$	Mo	$7\cdot10^{-4}$	$3.1\cdot10^{-4}$
300	$1.95\cdot10^{-1}$	Mo	$8\cdot10^{-4}$	$3.6\cdot10^{-4}$
400	$1.95\cdot10^{-1}$	Mo	$1.8\cdot10^{-3}$	$8.1\cdot10^{-4}$
500	$5\cdot10^{-3}$	Mo	$6\cdot10^{-4}$	$2.7\cdot10^{-4}$
500	$4.5\cdot10^{-2}$	Mo	$5\cdot10^{-4}$	$2.3\cdot10^{-4}$
500	$1.00\cdot10^{-1}$	Mo	$8\cdot10^{-4}$	$3.6\cdot10^{-4}$
500	$1.80\cdot10^{-1}$	Mo	$3.2\cdot10^{-3}$	$1.4\cdot10^{-3}$
500	$4.00\cdot10^{-1}$	Mo	$6.8\cdot10^{-3}$	$3.1\cdot10^{-3}$
100	$2.00\cdot10^{-1}$	stainless steel	$7\cdot10^{-4}$	$3.1\cdot10^{-4}$
200	$2.05\cdot10^{-1}$	stainless steel	$1.3\cdot10^{-3}$	$5.9\cdot10^{-4}$
300	$2.05\cdot10^{-1}$	stainless steel	$3\cdot10^{-4}$	$1.4\cdot10^{-4}$
400	$2.05\cdot10^{-1}$	stainless steel	$1.2\cdot10^{-3}$	$5.4\cdot10^{-4}$
500	$5\cdot10^{-3}$	stainless steel	$2\cdot10^{-5}$	$1\cdot10^{-5}$
500	$5.50\cdot10^{-2}$	stainless steel	$1.6\cdot10^{-3}$	$7.2\cdot10^{-4}$
500	$1.10\cdot10^{-1}$	stainless steel	$1.6\cdot10^{-3}$	$7.2\cdot10^{-4}$
500	$2.05\cdot10^{-1}$	stainless steel	$1.9\cdot10^{-3}$	$8.6\cdot10^{-4}$
500	$4.00\cdot10^{-1}$	stainless steel	$2.5\cdot10^{-3}$	$1.1\cdot10^{-3}$

[a] calculated by the compilers.

The concentrations of Mo as well as of the stainless steel components (Cr, Fe, Ni) in Na containing about 0.2 mass % O were also reported in the paper.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

V specimens as well as Na were placed in Mo or stainless steel capsules. These specimens had been vacuum annealed for 1 hour at 1173 K before testing and finally installed at the bottom of the capsule. The capsules were covered with a Ta foil liner and inserted in a stainless steel protective container. The container was kept at 873 K for 100 to 500 hours and rotated for stirring. Finally the capsules were inverted and quenched in liquid N. The O concentrations were either determined by vacuum fusion or fast neutron activation analysis. The V content in Na was determined by spectrographic analysis after dissolving the Na sample in isopropyl alcohol. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

V: special high purity, supplied by the US Bureau of Mines, containing $7.0\cdot10^{-3}$ % C, $8.0\cdot10^{-3}$ % O, $8\cdot10^{-4}$ % N.
Na: probably purified by continuous passage over a heated active metal e.g. Zr at 873 K.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.

US Atom.Ener.Comm.Rep. AI-AEC-12955, 1970.

VARIABLES:

Temperature: 873-1173 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of V in liquid Na at various temperatures is reported:

t/°C	*soly*/mass % V	*soly*/mol % V[a]
600	$2\cdot10^{-4}$	$8\cdot10^{-4}$
700	$1\cdot10^{-4}$, $2\cdot10^{-4}$	$5\cdot10^{-5}$, $1\cdot10^{-4}$
800	$1\cdot10^{-4}$	$5\cdot10^{-5}$
900	nil	nil

[a] calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The V crucible was cleaned with a HNO_3, H_2SO_4, H_2O (5:3:2) mixture and few drops of HF, and subsequently washed with H_2O and acetone. A collector fabricated of Nb-Zr (1%) was cleaned the same way. The crucible-collector assembly was degassed for 2 hours at 623 °C. The assembly was filled with Na and welded under high vacuum. The capsules were equilibrated for 6 hours in an Ar atmosphere glove box at the desired temperature. Finally the whole apparatus was inverted in order to cause a flow of Na into the collector part. The entire Na sample was analysed after solidification by means of a spectrophotometric method to determine the V content.

SOURCE AND PURITY OF MATERIALS:

V: supplied by Mater. Res. Corp., electron beam zone refined, 99.94 % purity, containing $5.7\cdot10^{-3}$ % C, $3\cdot10^{-4}$ % H, N, 2.5 10^{-2} % O, $2.0\cdot10^{-2}$ % Fe, $2.0\cdot10^{-3}$ % Ni, Si, $1.5\cdot10^{-3}$ % Pd, $6\cdot10^{-4}$ % Ti, $7\cdot10^{-4}$ % W, $<5.0\cdot10^{-3}$ % Cr, $<2.0\cdot10^{-3}$ % Ga, $8\cdot10^{-5}$ % Nb, $6\cdot10^{-5}$ % Au, all other elements $<5\cdot10^{-5}$ % each.
Na: purified, containing $8\cdot10^{-4}$ % C, $4\cdot10^{-4}$ % O, $1\cdot10^{-4}$ % Cr, $<1\cdot10^{-4}$ % Ag, Al, B, Be, Cd, Fe, Li, Mn, Mo, Pb, Ta, V, $<5\cdot10^{-4}$ % Ba, Bi, Co, Cu, Mg, Ni, Rb, Sn, Ti, $<1.0\cdot10^{-3}$ % Cs, K, Si, Zr, $<1.0\cdot10^{-2}$ % Zn.
Ar: purified.

ESTIMATED ERROR:

Solubility: detection threshold of V is $2\cdot10^{-5}$ mol %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Babu, S.R.; Periaswami, G.; Geetha, R.; Mahalingam, T.R.; Mathews, C.K.
Liquid Metal Engineering Technology, BNES, London, 1984, *1*, 271-275.

VARIABLES:

Temperature: 557 - 751 K.

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of V in liquid Na at various temperatures is reported.

T/K	*soly*/mass % V	*soly*/mol % V [a]
557	$6.5 \cdot 10^{-6}$	$9.8 \cdot 10^{-7}$
586	$1.9 \cdot 10^{-6}$	$8.6 \cdot 10^{-7}$
598	$4.9 \cdot 10^{-6}$	$2.2 \cdot 10^{-6}$
634	$9.6 \cdot 10^{-6}$	$4.3 \cdot 10^{-6}$
646	$1.53 \cdot 10^{-5}$	$6.9 \cdot 10^{-6}$
657	$3.0 \cdot 10^{-6}$	$1.4 \cdot 10^{-6}$
710	$4 \cdot 10^{-7}$	$1.8 \cdot 10^{-7}$
722	$1.3 \cdot 10^{-6}$	$5.9 \cdot 10^{-7}$
751	$8 \cdot 10^{-7}$	$3.6 \cdot 10^{-7}$

[a] calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Liquid Na was filled in a Ta crucible, and weighed amounts of (3 %) Mg metal as well as several pieces of V were added. The crucible, a centrally placed "porosint" 5 μm pore size filter and a Ta collector were installed inside the capsule. The capsule was heated in the inverted position for nearly 40 hours. Sample collection was performed by inverting the assembly and pressing the Na solution through the filter by means of 0.2 MPa Ar. The Ta crucible with the collected Na was removed, weighed and taken for vacuum distillation at 623 K. The residue left in the Ta crucible was dissolved in aqua regia, which was subsequently evaporated to dryness and further fumed with HNO_3. The pH of this solution was adjusted to 2.5. V was extracted with 2 cm^3 aliquot of 2 $mol \cdot dm^{-3}$ HCl and final dilution of the solution, the V content was analysed by means of atomic absorption spectrometric determination.

SOURCE AND PURITY OF MATERIALS:

V: 99.9 % pure, supplied by Goodfellow Metals.
Mg: 99.9 % pure.
Na: of nuclear grade purity, kept at 473 K in an inert atmosphere.
All other chemicals were analytical grade reagents.

ESTIMATED ERROR:

Solubility: precision ± 50 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Guminski, C.; Borgstedt, H.U.

Z. Metallk. 1994, *85*, 771-774.

VARIABLES:

One temperature: 873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of V in liquid Na was estimated to be $1.2 \cdot 10^{-8}$ mol % V at 873 K; this result was also reported in (2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed in a circuit of tubes made of stainless steel. V sheets were placed in the circulating flow of molten Na in unisothermal conditions. The circuit was equipped with an electromagnetic pump which forced the liquid Na through the tubes under turbulent flow conditions. A temperature gradient 973 - 823 K was kept in the circuit. Na was supplied from the purification loop in which it passed a cold trap to precipitate impurities. Ar was used as inert gas cover. The V sample sheets were weighed before and after the experiments. The solubility of V in liquid Na was calculated on the basis of the equation relating the corrosion rate and the solubility (1).

SOURCE AND PURITY OF MATERIALS:

V: 99.94 % pure from Metallgesellschaft, with contents of $2.45 \cdot 10^{-2}$ % O, < $1 \cdot 10^{-2}$ % N, and $2.2 \cdot 10^{-2}$ % C.
Na: purified by precipitation of impurities and filtering the liquid at about 393 K and gettered with Zr foil at 973 K, with contents of < $1 \cdot 10^{-4}$ % O and < $5 \cdot 10^{-4}$ % N.
Ar: purified, with contents of < $1 \cdot 10^{-4}$ % O and < $1 \cdot 10^{-4}$ % H_2O.

ESTIMATED ERROR:

Solubility: precision ± 20 %.
Temperature: precision ± 3 K.

REFERENCES:

(1) Borgstedt, H.U.; Röhrig, H.D. *J. Nucl. Mater.* 1991, *179-181*, 596.

(2) Guminski, C.; Borgstedt, H.U. 5th *Intern. Symp. on Solubility Phenomena*, Russ. Acad. Sci., Moskva, 1992.

COMPONENTS:	EVALUATOR:
(1) Vanadium; V; [7440-62-2] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1988

CRITICAL EVALUATION:

Determinations of the solubility of V in liquid K were published by Stecura (1,2). K containing less than $4 \cdot 10^{-3}$ mol % O and another purity of K to which KNO_3 was added were used in these studies. The temperature dependence of the solubility showed a decrease with increasing temperature, if the concentrations of O and N were raised from 0.14 to 1.39 and from 0.040 to 0.41 mol %, respectively. Even an inverse dependence of the solubility on temperature was observed at higher degree of contamination of K with KNO_3. N has probably little effect on the apparent solubility of V in K at low concentration levels. The influence of O seems to be more pronounced, especially at lower temperatures, if a K-V-O compound might be formed. The stability of such a compound may decrease with increasing temperature, thus causing the apparent solubility of V in K to become comparable to the solubility of the pure metal. VO and V_3N were identified as equilibrium solid phases at high KNO_3 concentrations in liquid K after the solubility tests.
The solubility data of (2) at an O level of less than $4 \cdot 10^{-3}$ mol % may be described by the relation derived by the least square method (as tested by the evaluators).

$$\log(soly/\text{mol \% V}) = (1.10 \pm 0.26) - (4530 \pm 300)(T/\text{K})^{-1}$$

Aleksandrov and Dalakova (4) did not find any dissolution of V in liquid K at 873-923 K after an equilibration of 1 h, the detection limit of their spectral analysis was not reported.
A schematic V-K phase diagram was established by (3), it is similar to that shown for the V-Li system, differing in the boiling and melting points of K. The formation of intermetallics was not observed in the V-K system.

Tentative values of the solubility of V in liquid K

T/K	*soly*/mol % V	source	remarks
1012	$4 \cdot 10^{-4}$	(2)	at K vapour pressure
1117	$1 \cdot 10^{-3}$	(2)	at K vapour pressure
1242	$3 \cdot 10^{-3}$	(2)	at K vapour pressure
1341	$5 \cdot 10^{-3}$	(2)	at K vapour pressure

References

1. Stecura, S. *NASA Rep. TN-D-5093*, 1969.
2. Stecura, S. *Corrosion by Liquid Metals*, J.E. Draley, J.R. Weeks, Eds., Plenum, N.Y., 1970, p. 601
3. Smith, J.F.; Lee, K.J. *Bull. Alloy Phase Diagr.* 1990, *11*, 249.
4. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad.Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Stecura, S.
Corrosion by Liquid Metals, J.E. Draley, J.R. Weeks, Eds., Plenum, N.Y., 1970, p. 601-611.

VARIABLES:

Temperature: 1012 - 1341 K
O content in K: 0.056-0.5 mass %
N content in K: 0.0163-0.146 mass %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The V solubilities in liquid K at various temperatures and O and N contents in Na were presented in the figure, read-off and calculated to mol % by the compilers.

T/K	O *concn*/mass %	N *concn*/mass %	*soly*/ mol % V
1012	0.056	0.0163	$8.5 \cdot 10^{-4}$, $1.1 \cdot 10^{-3}$
1012	0.25	0.073	$1.8 \cdot 10^{-3}$, $2.3 \cdot 10^{-3}$
1020	0.50	0.146	$6.2 \cdot 10^{-3}$, $6.9 \cdot 10^{-3}$
1136	0.50	0.146	$2.0 \cdot 10^{-3}$, $2.3 \cdot 10^{-3}$
1170	0.25	0.073	$1.2 \cdot 10^{-3}$, $1.5 \cdot 10^{-3}$
1215	0.056	0.0163	$3.4 \cdot 10^{-3}$, $3.8 \cdot 10^{-3}$
1285	0.50	0.146	$8.0 \cdot 10^{-4}$
1333	0.25	0.073	$8.5 \cdot 10^{-4}$, $1.2 \cdot 10^{-3}$
1341	0.056	0.0163	$3.8 \cdot 10^{-3}$, $4.9 \cdot 10^{-3}$
1012	-	-	$3.8 \cdot 10^{-4}$, $5.4 \cdot 10^{-4}$
1033	-	-	$6.1 \cdot 10^{-4}$
1036	-	-	$4.6 \cdot 10^{-4}$
1117	-	-	$7.7 \cdot 10^{-4}$, $1.1 \cdot 10^{-3}$
1242	-	-	$3.4 \cdot 10^{-3}$, $4.2 \cdot 10^{-3}$
1330	-	-	$4.2 \cdot 10^{-3}$, $5.4 \cdot 10^{-3}$
1341	-	-	$5.4 \cdot 10^{-3}$, $4.6 \cdot 10^{-3}$

O and N were introduced into in Na in form of KNO_3. The equilibrium solid phase contained VO and V_3N. The results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The K and the test crucible made of V were sealed in a Ta capsule serving as a container for the crucible and K. The capsule assembly was heated to the desired temperature as controlled by a Pt/Pt-Rh (3%) thermocouple and equilibrated for 24 hours. The furnace was inverted to cause the saturated K solution to drain away from the test crucible into the collecting capsule. After cooling to room temperature the apparatus was removed from the furnace and cut open. The sample was dissolved in C_4H_9OH, the inner wall of the capsule was leached with HCl to remove precipitated V. The V concentration was determined by optical spectrographic and wet chemical analysis.

SOURCE AND PURITY OF MATERIALS:

V: 99.7 % purity (or better), containing $6.75 \cdot 10^{-2}$ % O.
Ta: 99.9 % purity, containing $1.3 \cdot 10^{-3}$ % O.
K: containing $<2 \cdot 10^{-3}$ % O.

ESTIMATED ERROR:

Solubility: precision ± 18 %.
Temperature: accuracy ± 8 K.

REFERENCES:

1. Stecura, S. *NASA Rep. TN-D-5093*, 1969.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Vanadium; V; [7440-62-2] (2) Rubidium; Rb; [7440-17-7]	Young, P.F.; Arabian, R.W. *US Atom.Ener.Comm.Rep. AGN-8063*, 1962.
VARIABLES:	**PREPARED BY:**
Temperature: 811 and 1033 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of V in liquid Rb at 2 different temperatures is reported:

t/°F	*soly*/mass % V	*soly*/mol % V [a]
1000	$<1\cdot10^{-4}$	$<2\cdot10^{-4}$
1400 [b]	$<1\cdot10^{-4}$	$<2\cdot10^{-4}$

[a] calculated by the compilers

[b] at Rb vapor pressure

COMMENTS AND ADDITIONAL DATA:

As a possible interaction of the V test capsule with Ta, the material of the containment and collector capsule via a Rb medium is negligible (1). Thus the given detection limits should be regarded to be correct. However, the real solubility values are probably much lower than these limits. A schematic phase diagram of the V-Rb system, as reported in (2), did not indicate a formation of V-Rb intermetallics and is similar to that shown for the V-Li system, differing in the melting and boiling points of Rb.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A V test capsule, which had been cleaned in HNO_3, rinsed with H_2O and dried, was installed in a Ta containment capsule. The capsule was outgassed at the test temperature for 1000 hours and subsequently sealed in an Ar atmosphere. The temperature was elevated and kept at the desired level for 50 hours. On removal, the capsule was inverted, causing Rb with the dissolved V to flow into the Ta sample cup. The cup was cooled to room temperature. After solidification the capsule was cut open and its content analysed for O and metals. The solidified Rb solution was treated with anhydrous hexane, CH_3OH for the Rb methylization, H_2O, and finally HCl. The resulting solution was heated to dryness. The V content of the solid residue was analysed in the National Spectroscopic Laboratories. The Ta sample cup was treated with aqua regia in a water bath for 1 hour and the resulting solution was added to the RbCl solution in order to dry.

SOURCE AND PURITY OF MATERIALS:

V: 99.6 % pure, supplied by Oremet Metallurgical Corp., containing, 0.01 % C, 0.001 % H, 0.04 % O, N. Rb: the same as in the Ti-Rb system, same report, O concentration before and after the test $(6\text{-}17)\cdot10^{-4}$ and $(5.4\text{-}7.4)\cdot10^{-3}$ % O, respectively.

ESTIMATED ERROR:

Solubility: detection limit of $1\cdot10^{-4}$ mass % V.
Temperature: precision ± 3 K.

REFERENCES:

1. DeBoer, F.R.; Boom, R.; Miedema, A.R. *Physica, B* 1980, *101*, 294.
2. Smith, J.F.; Lee, K.J. *Bull. Alloy Phase Diagr.* 1990, *11*, 249.

COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Cesium; Cs; [7440-46-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
November 1988

CRITICAL EVALUATION:

It may be stated that V solubility in liquid Cs is very low, since corrosion tests of V in liquid Cs performed for 100 hours at 773 K (1) and 500 hours at 1023 K (5) did not indicate a weight change of the samples. However, Godneva et al. (4) determined V solubility in liquid Cs at temperatures from 323 to 673 K to be practically temperature independent, but dependent on the V purity. They obtained results of $\sim 1.7\cdot10^{-2}$ mol % V for a commercial product, and $\sim 5\cdot10^{-3}$ mol % for thermally refined V.
VO, which frequently covers metallic V, is also very resistive to dissolution in liquid Cs (2).
A predicted phase diagram constructed by Smith and Lee (3) is similar to that shown for the V-Li system, differing in the melting and boiling points of Cs. Formation of intermetallics in the system was not reported.

References

1. Keddy, E.S. *US Atom.Ener.Comm. Rep. LAMS-2948*, 1963.
2. Antill P.F.; Peakall, K.A.; Smart, E.F. *J. Nucl. Mater.* 1975, *56*, 47.
3. Smith, J.F.; Lee, K.J. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 908; *Bull.Alloy Phase Diagr.* 1988, *9*, 47.
4. Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.
5. Berry, W.E. *Corrosion in Nuclear Applications*, Wiley, New York, 1971, p. 303.

COMPONENTS:

(1) Vanadium; V; [7440-62-2]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S.

Zh. Prikl. Khim. 1974, *47*, 2177-2180.

VARIABLES:

Temperature: 323-673 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The V solubility in liquid Cs at several temperatures and from V samples of different V samples.

t/°C	*soly*/mass % V	*soly*/mol % V[a]
50 [b]	$6.6\cdot10^{-3}$	$1.7\cdot10^{-2}$
150 [b]	$6.6\cdot10^{-3}$	$1.7\cdot10^{-2}$
200 [b]	$6.2\cdot10^{-3}$	$1.6\cdot10^{-2}$
300 [b]	$<3\cdot10^{-4}$	$<7.8\cdot10^{-4}$
200 [c]	$2.0\cdot10^{-3}$	$5.2\cdot10^{-3}$
300 [c]	$1.3\cdot10^{-4}$	$3.4\cdot10^{-3}$
400 [c]	$2.2\cdot10^{-3}$	$5.7\cdot10^{-3}$

[a] as calculated by the compilers [b] commercially pure V [c] thermally refined V

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A V specimen was covered with Cs under vacuum and equilibrated for 120 hours in a glass ampoule. The O content in Cs was increased by means of controlled decomposition of a $KClO_3$-MnO_2 mixture. The ampoule glass did not undergo visible changes. Cs was dissolved in H_2O and determined by titration with an acid. The remaining part of the solution was treated with a mixture of urea, $KMnO_4$, and $NaNO_3$. H_3PO_4 and benzidine were added. The V content of the resulting solution was analysed colorimetrically (1).

SOURCE AND PURITY OF MATERIALS:

V: containing < 0.03 % Fe, 0.24 % Al, uncertain whether commercial or refined V was specified.
Cs: 98-99 % pure metal, vacuum distilled, finally containing <0.01 % O, <1.5 % Rb as main impurities.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Godneva, M.M.; Vodyannikova, R.D. *Zh. Anal. Khim.* 1965, *20*, 831-836.

COMPONENTS:	EVALUATOR:
(1) Niobium; Nb; [7440-03-1] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1992

CRITICAL EVALUATION:

The results of determinations of the solubility of Nb in liquid Li are scattered. Contamination of Li with O or N was found to influence the solubility in a contradictory manner. Most of the data presented in (1) indicated an almost regular increase in solubility from $< 1.5 \cdot 10^{-4}$ to $\sim 3 \cdot 10^{-3}$ mol % Nb in the temperature range of 772 to 1291 K. The results obtained after various times of equilibration indicated a complex behaviour of the system. The solubility of Nb in Li seemed not only to be influenced by the presence of O and N in Li, but also by the possible formation (2) of Nb-Fe compounds (with Fe from the crucible), which produced a higher apparent solubility. The solubility vs. temperature dependence is similar to the later published values, while the absolute values are one order of magnitude above the more reliable results of subsequent studies. The results of (3) obtained at 981 to 1183 K and different levels of N in Li indicated a significant influence of the N concentration in the range 0.0019 to 0.013 mol % N on the solubility of Nb. Further studies of the same laboratory (4,5) in which the temperature of equilibration was extended to 1813 K and the concentrations of O and N to 0.25 and 0.5 mol %, respectively, did not show an influence of O and N on the solubility of Nb. The data of (4,5) were scattered ± 60 % which was in fair agreement with the earlier results (3) at a N concentration of 0.0019 mol %. The mean value of the solubility at 1813 K was distinctly lower than the other data of the series; this was probably not caused by the elevated pressure during the test at the highest temperature. The suggestion of (6) that the Li used in (3) might have been more contaminated by components of air than by additions of Li_3N is doubtful, since the results in (4,5) did not confirm an influence of either O nor N on the Nb solubility. It is doubtful if CO_2 has an influence on the solubility of Nb in Li; its effect seems to be negligible.

Only the detection limits of the applied analytical methods were presented as Nb concentrations in the remaining studies of the solubility of Nb in Li (7,8),(9,10) and (11). Providing that precise values of the solubility were slightly lower than the limits given, a fair agreement of (9,10) with the data of (4,5) and the lowest values of (3) may be concluded. However, the limit of (7,8) was more than one order of magnitude lower than the others. According to theoretical predictions in (12-14), a regular increase of the solubility of Nb in liquid Li from $1.8 \cdot 10^{-10}$ to $5 \cdot 10^{-3}$ mol % Nb at 873 to 1813 is to be expected. Thus, merely the result of (7,8) being $< 7 \cdot 10^{-6}$ mol % Nb at 1273 K is in agreement with the prediction. The line of the average data of (3-5,10,11) intersects the predicted line at ~ 1500 K. The ratio of the slopes of these lines is more than 10, indicating a serious discrepancy between experiments and theory. An interference of non-metallic impurities in the dissolution process of Nb in Li is quite certain, but it is difficult to create the exact relationships between effects and concentrations. The possible simultaneous presence of the two elements, O and N, in Li cannot be excluded. It might be that the combination influences the solubility of Nb, while each element may be much less effective, if the other element is not present. Such mixed compounds were found in a corrosion study by (15).

Gryaznov et al. (16) observed that a significant increase of the Nb concentration in Li of $1.5 \cdot 10^{-3}$ mol % occurred at 1223 K and elevated concentrations of N in Li of 0.1 mol %. The system reached, however, the equilibrium at $1.5 \cdot 10^{-4}$ mol % Nb after 1 hour. An increase of the N content to 1.15 mol % in Li caused an initial increase of the Nb concentration to up to $1.2 \cdot 10^{-2}$ mol % Nb. The equilibrium was slowly approached in this experiment, and the equilibrium solubility value seems to be at $\sim 8 \cdot 10^{-3}$ mol % Nb; see the data sheet of the paper (11). The described experiments showed that smaller amounts of N may diffuse into the solid phase Nb. Thus, the equilibrium may only slightly be disturbed by the presence of N in the system. Larger amounts of N, however, significantly influence the kinetics of dissolution as well as the equilibrium solubility of Nb in liquid Li.

Among the products of Nb corrosion by Li several compounds were identified or postulated: Nb_2N (15,17); NbN, NbC, (17); Li_7NbN_4 (18); $LiNbO_3$ (19) and $NbN_{0.9}O_{0.1}$ (15). The equilibrium phase may also be a solid solution of the contaminating elements (O, C, N) in Nb, since Nb-Li intermetallics are not known (20). A Nb-Li schematic phase diagram from (21) was used as the basis for the figure.

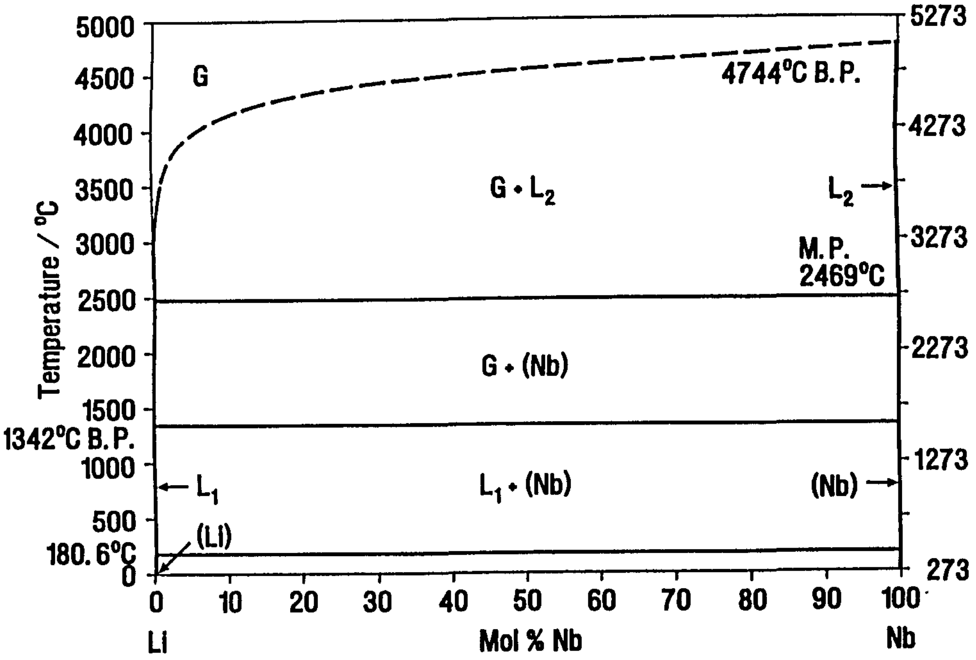
The suggested values of the solubility of Nb in liquid Li can be calculated using the fitting equation which is presented in the data sheet of (4,5). However, the limit reported in (11) is more reliable. It is ~20 % lower than that from the fitting equation of (4,5).

Tentative values of the solubility of Nb in liquid Li at ~ $1 \cdot 10^{-3}$ mol % N

T/K	*soly*/mol % Nb	source
1323	$\leq 1.5 \cdot 10^{-4}$	(11)

COMPONENTS:	EVALUATOR:
(1) Niobium; Nb; [7440-03-1] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1992

CRITICAL EVALUATION: (continued)



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11. Beskorovainyi, N.M.; Ioltukhovskii, A. G.; Lyublinskii, I.E.; Vasilev, V.K. *Fiz.-Khim. Mekh Mater.* 1980, *16*, *no 3*, 59.
12. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroenia Diagram Sostoyania, Nauka, Moskva,* 1985, p. 113.
13. Beskorovainyi, N.M.; Ioltukhovskii, A. G.; Kirillov, V.B.; Lyublinskii, I.E.; Filipkina, E.I. *Fiz.-Khim. Mekh. Mater.* 1984, *20*, *no 6*, 9.
14. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Materialy dla Atomnoi Tekhniki*, Energoatomizdat, Moskva, 1983, p. 33.
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16. Gryaznov, G.M.; Evtikhin, V.A.; Zavyalskii, L.P.; Kosukhin, A.Ya.; Lyublinskii, I.E. *Materialovedenie Zhidkometallicheskikh Sistem Termoyadernykh Reaktorov*, Energoatomizdat, Moskva, 1989, p. 109.
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COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B.

Atom. Energiya 1959, *7*, 531-536; *Kernenergie* 1960, *3*, 763-767.

VARIABLES:

One temperature: 1273 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid Li was reported to be less than 10^{-4} mass% at 1000 °C. The compilers recalculation gives a value of $7 \cdot 10^{-6}$ mol% Nb. The same information was reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The inner surface of a Nb crucible was ground, electrolytically polished and etched. This crucible was gradually filled with freshly distilled Li dropping from a stainless steel condenser. After completion of the distillation the apparatus was filled with pure Ar. The filled crucible was placed in a stainless steel container to which the cover was welded in an arc furnace.

Additionally, the crucible was isolated from the steel by a Mo band. The container was placed in an arc furnace and conditioned at 1273 K for 100 hours.

The Li solution was cooled to solidification in less than 50 s. The content of Nb in the sample was determined by colorimetric analysis.

SOURCE AND PURITY OF MATERIALS:

Nb: unspecified purity, the material was used in the form of caked briquettes.
Li: as distilled, with final contents of $(2-6) \cdot 10^{-2}$ % Na; $1.5 \cdot 10^{-2}$ % K; $(1-4) \cdot 10^{-4}$ %Fe; $\leq 2 \cdot 10^{-3}$ % Mg; Si, Ni, and Cr were not detected.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall.Metalloved.Chist.Met.* 1960, *2*, 178-188; *Metallurgy and Metallography of Pure Metals*, Gordon & Breach,N.Y., 1962,p. 178-188.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Klueh, R.L.

Metall. Trans. 1974, *5*, 875-879.

VARIABLES:

One temperature: 873 K
O concentration in Li: $5 \cdot 10^{-3}$ - 0.1 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid Li at 600 °C is lower than the detection limit of $1.0 \cdot 10^{-3}$ mass% or $7 \cdot 10^{-5}$ mol % Nb (as calculated by the compilers). The concentration of O in Li varied in the range 0.01 to 0.2 mass %, it does not show any influence on the solubility of Nb in Li. The same observation was reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The applied system consisted of a Nb specimen in contact with the solvent in a container of the same material, which in turn was encapsuled in stainless steel. The capsule was filled with liquid Li and welded in an Ar atmosphere chamber. The concentration of O was varied by adding Li_2O in order to determine the effect of O in Li.

The system was equilibrated at 873 K for 20 hours. After the test the capsule was quenched in liquid nitrogen and opened in the dry Ar box.

Li was removed by dissolving in chilled isopropyl alcohol and recovered from it as LiF. The amount of Nb in Li was determined by spectrographic analysis.

SOURCE AND PURITY OF MATERIALS:

Nb: 99.9 % pure, with a content of $8.0 \cdot 10^{-3}$ % O.
Li: purified by Zr gettering at 1073 K.

ESTIMATED ERROR:

Nothing specified

REFERENCES:

1. Klueh, R. L. *Trans. Am. Nucl. Soc.* 1972, *15*, 746-747.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Leavenworth, H.; Cleary, R.E.; Bratton, W.D.

US Atom. Ener. Comm. Rep. PWAC-356, 1961.

VARIABLES:

Temperature: 971 - 1183 K
N content in Li: $3.8\cdot10^{-3}$ - $2.6\cdot10^{-2}$%

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubilities of Nb in liquid Li containing various amounts of N were reported in the figure; they were read out and calculated by the compilers.

T/K	*N content in Li*/mass %	*soly*/mol % Nb
1042	$3.8\cdot10^{-3}$	$7.5\cdot10^{-5}$
1073	$3.8\cdot10^{-3}$	$8.2\cdot10^{-5}$
1095	$3.8\cdot10^{-3}$	$1.1\cdot10^{-4}$
1064	$7.0\cdot10^{-3}$	$9.5\cdot10^{-4}$
1068	$7.0\cdot10^{-3}$	$1.4\cdot10^{-3}$
1078	$7.0\cdot10^{-3}$	$1.5\cdot10^{-3}$, $1.2\cdot10^{-3}$
1121	$7.0\cdot10^{-3}$	$1.3\cdot10^{-3}$
1130	$7.0\cdot10^{-3}$	$1.2\cdot10^{-3}$
1183	$7.0\cdot10^{-3}$	$1.6\cdot10^{-3}$
971	$2.6\cdot10^{-2}$	$1.9\cdot10^{-3}$
1010	$2.6\cdot10^{-2}$	$2.5\cdot10^{-3}$
1064	$2.6\cdot10^{-2}$	$4.6\cdot10^{-3}$
1121	$2.6\cdot10^{-2}$	$3.2\cdot10^{-3}$, $4.8\cdot10^{-3}$
1140	$2.6\cdot10^{-2}$	$3.8\cdot10^{-3}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The specimen cup made of Nb was heated to 811 K, and purified Li with various amounts of Li_3N was added. The cup was equilibrated for 24 hours at the test temperature. A sample of the saturated solution was removed by means of the sample beaker made of Mo. The sample was allowed to cool and dissolved in 10 % HCl in a polyethylene beaker at dry ice temperature. A colorimetric method not described in detail was used to determine the Nb content and flame photometry for Li to obtain the weight of the whole sample. Measurements in purified Ar atmosphere were performed at increasing and decreasing temperatures.

SOURCE AND PURITY OF MATERIALS:

Nb: 99.8% pure.
Li: 99.8 % pure purchased following the specification: $<5\cdot10^{-3}$% Na; $<1\cdot10^{-2}$% K,Ca; $<3\cdot10^{-2}$% Cl; $<3\cdot10^{-2}$% Fe+Al; $<7\cdot10^{-2}$% heavy metals; it was further purified by gettering with Ti at 1144 K, the N content was then $<1\cdot10^{-3}$%.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Cleary, R.E.; Blecherman, S.S.; Corliss, J.E.

US Atom. Ener. Comm. Rep. TIM-850, 1965.

VARIABLES:

Temperature: 1033-1813 K;
N and O concentration in Li: 0.025-0.25 and 0.022-0.44 mol %, respectively.

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubilities of Nb in liquid Li at various temperatures and concentrations of N and O were reported.

t/°C	*addition*/mass %	*soly*/mass % Nb	*soly*/mol% Nb[a]
760	-	$4.1\cdot10^{-3}$;$1.2\cdot10^{-3}$	$3.0\cdot10^{-4}$;$9.0\cdot10^{-5}$
760	0.05 N	$9.0\cdot10^{-4}$;$1.23\cdot10^{-2}$	$6.7\cdot10^{-5}$;$9.0\cdot10^{-4}$
760	0.10 N	$3.2\cdot10^{-3}$	$2.4\cdot10^{-4}$
760	0.50 N	$1.7\cdot10^{-3}$	$1.3\cdot10^{-4}$
980	-	$3.4\cdot10^{-3}$;$3.1\cdot10^{-3}$	$2.5\ 10^{-4}$;$2.3\cdot10^{-4}$
980	0.05 N	$2.0\cdot10^{-4}$;$8.0\cdot10^{-4}$	$1.5\cdot10^{-5}$;$6.0\cdot10^{-5}$
980	0.10 N	$2.4\cdot10^{-3}$	$1.8\cdot10^{-4}$
980	0.50 N	$4.9\cdot10^{-3}$	$3.7\cdot10^{-4}$
1205	-	$3.7\cdot10^{-3}$;$2.6\cdot10^{-3}$	$2.8\cdot10^{-4}$;$1.9\cdot10^{-4}$
1205	0.05 N	$9.0\cdot10^{-4}$	$6.7\cdot10^{-5}$
1205	0.10 N	$3.2\cdot10^{-3}$	$2.4\cdot10^{-4}$
1205	0.50 N	$4.4\cdot10^{-3}$	$3.3\cdot10^{-4}$
1425	-	$1.33\cdot10^{-2}$	$9.9\cdot10^{-4}$
1425	0.05 N	$2.2\cdot10^{-3}$	$1.6\cdot10^{-4}$
1425	0.10 N	$9.7\cdot10^{-3}$	$7.2\cdot10^{-4}$
1425	0.50 N	$2.5\cdot10^{-3}$	$1.9\cdot10^{-4}$
1540[b]	-	-	$6\cdot10^{-5}$;$1.9\cdot10^{-4}$;$2.3\cdot10^{-4}$
980	0.05 O	$3.1\cdot10^{-3}$	$2.3\cdot10^{-4}$
980	0.10 O	$3.8\cdot10^{-3}$	$2.8\cdot10^{-4}$
980	0.50 O	$8\cdot10^{-4}$	$6\cdot10^{-5}$
980	1.0 O	$5.0\cdot10^{-3}$	$3.8\cdot10^{-4}$
1205	0.05 O	$6.6\cdot10^{-3}$	$4.9\cdot10^{-4}$
1205	0.50 O	$5.5.10^{-3}$	$4.1\cdot10^{-4}$
1205	1.0 O	$1.5\cdot10^{-3}$	$1.1\cdot10^{-4}$

[a] as calculated by the compilers [b] as graphically reported in (1)

Most of the results were also graphically reported in (1). The results may be fitted to the equation (as tested by the compilers):

$$\log(soly/\text{mol \% Nb}) = -2.77 - 1100\,(T/\text{K})^{-1}$$

The neglection of the values at 1540 °C seems to give the more correct correlation in the compiler's opinion.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Nb cup was mounted inside a Mo receiver capsule. The cup was filled with a weighed quantity of Li and sealed in an Ar atmosphere. For the determination of the solubility above 1478 K the capsule was enclosed in a Mo-0.5Ti container and heated in vacuo. Below this temperature the capsule was wrapped in a Ta foil, placed in an Inconel shroud and heated in air. The capsules were placed in a furnace in receiver-up position and conditioned for 100 hours at the desired temperature. The capsule was inverted at temperature and the Li saturated with Nb transferred to the receiver. The capsule was cooled to room temperature and cut open in Ar atmosphere. The solidified Li was dissolved in H_2O and the receiver cup etched in dilute HNO_3-HCl.

The Nb complex with N-benzoyl,N-phenyl-hydroxyloamine was extracted with $CHCl_3$. The extract was evaporated and converted into a small volume of aqueous solution for spectrographic measurement.

N and O were added to the system in the form of Li_2O and Li_3N, respectively.

SOURCE AND PURITY OF MATERIALS:

Nb: contained $6.5\cdot10^{-3}$ % C, $7.0\cdot10^{-3}$ % N, $7.5\cdot10^{-3}$ % O, $1\cdot10^{-3}$ % H.

Li: contained $<3\cdot10^{-2}$ % Na, $<1\cdot10^{-2}$ % K, Ca, Ni, $<5\cdot10^{-3}$ % Fe, $<2\cdot10^{-3}$ % Cl, Cr, Cd, Ln, $<1\cdot10^{-2}$ % of all others. It was further treated with Ti sponge at 1144 K for 2 h, with final content of 0.10 % O and $< 5.10^{-3}$ % N.

Ar: with contents of $< 2.5\cdot10^{-4}$ % O and $< 7.5\cdot10^{-4}$ % H_2O.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ±15 %, scatter of the results up to ±60 % (by the compilers).

REFERENCES:

1. Blecherman, S.S.; Schenck, G.F.; Cleary, R.E.; *US Atom.Ener.Comm. Rep. CONF-650411-4*, 1965; *Rep. CNLM-6335*, 1965; *Rep.CONF-650411*, 1965, p. 48.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Beskorovainyi, N.M.; Ioltukhovskii, A. G.; Lyublinskii, I.E.; Vasilev, V.K.
Fiz.-Khim. Mekh. Mater. 1980, *16*, no 3, 59-64.

VARIABLES:

Temperature: 1223-1323 K
Concentration of N in Li: $1 \cdot 10^{-3}$ - 1.15 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid Li at 1050 °C is not higher than $2 \cdot 10^{-3}$ mass % or $1.5 \cdot 10^{-4}$ mol % Nb (as recalculated by the compilers). The same result was reported in (1,2).
The authors of (3) investigated the kinetics of the dissolution of Nb in liquid Li containing N using the same technique. They observed an initial increase of the content of Nb in the solvent which was more pronounced, if the N content in Li was higher. The Nb concentration dropped down to the equilibrium value after about 1 hour. The equilibrium concentration of Nb in Li approached the value of $2 \cdot 10^{-3}$ mass % or $1.5 \cdot 10^{-4}$ mol % Nb within one hour at 950 °C and a N content of 0.1 mol %.
Another result was gained at 1025 °C and a N content of 1.15 mol %: The equilibrium concentration approached the value of ~ 0.11 mass % or $8 \cdot 10^{-3}$ mol % Nb, as read out from the figure and calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A cylindrical crucible made of Nb was placed in a sealed chamber having "windows" of Be transparent to x-rays. Liquid Li was kept inside the crucible (with small diameter) by means of surface forces in a way that it did not contact the Be. The capsule was heated in He atmosphere. A beam of x-rays was passed along the axis of the sample. The radiation passing through the Li was analyzed by means of a Soller spectrometer. Its intensity was measured from both sides in relation to the K-boundary of absorption of Nb. The quantity of Nb dissolved was calculated by means of comparisons of the absorbance before and after the equilibration. The concentration of N in Li was adjusted by means of addition of Li_3N.

SOURCE AND PURITY OF MATERIALS:

Nb: the purity was denoted as "NVCh"; the contents of O, N, C were ~0.01 % each.
Li: contained $5 \cdot 10^{-3}$ % K, $7.4 \cdot 10^{-2}$ % Na, 10^{-3} % Mn, $6.5 \cdot 10^{-3}$ % Mg, $3 \cdot 10^{-3}$ % Al, O, Si, $7.9 \cdot 10^{-3}$ % Ca, Fe, and 10^{-3} % N.
Li_3N: chemically pure.
He: nothing specified.

ESTIMATED ERROR:

Solubility: sensitivity of the method is $1.5 \cdot 10^{-4}$ mol % Nb.
Temperature: nothing specified.

REFERENCES:

1. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Materialy dla Atomnoi Tekhniki*, Energoatomizdat, Moskva, 1983, p. 33-41.
2. Beskorovainyi, N.M.; Ioltukhovskii, A. G.; Kirillov, V.B.; Lyublinskii, I.E.; Filipkina, E.I. *Fiz.-Khim. Mekh Mater.* 1984, *20, no 6*, 9-12.
3. Gryaznov, G.M.; Evtikhin, V.A.; Zavyalskii, L.P.; Kosukhin, A.Ya.; Lyublinskii, I.E. *Materialovedenie Zhidkometallicheskikh Sistem Termoyadernykh Reaktorov*, Energoatomizdat, Moskva, 1989, p. 109-111.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P.
US Atom. Ener. Comm. Rep. NEPA-1465, 1950.

VARIABLES:

Temperature: 772-1291 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubilities of Nb in liquid Li at various temperatures and times of equilibration are reported:

t/°C	*time*/h	*soly*/mass % Nb	*soly*/mol % Nb[a]
499	4	$<2.5\cdot10^{-3}$	$<1.9\cdot10^{-4}$
505	4	$<2.0\cdot10^{-3}$;$<2.5\cdot10^{-3}$	$<1.5\cdot10^{-4}$;$<1.9\cdot10^{-4}$
724	4	$<2.5\cdot10^{-3}$	$<1.9\cdot10^{-4}$
735	4	$<2.0\cdot10^{-3}$;$<2.5\cdot10^{-3}$	$<1.5\cdot10^{-4}$;$<1.9\cdot10^{-4}$
1004	4	$3.7\cdot10^{-2}$	$2.8\cdot10^{-3}$
1018	4	$3.3\cdot10^{-2}$; $3.6\cdot10^{-2}$	$2.5\cdot10^{-3}$; $2.7\cdot10^{-3}$
499	24	$<3.5\cdot10^{-3}$	$<2.6\cdot10^{-4}$
505	24	$<3.0\cdot10^{-3}$;$<3.5\cdot10^{-3}$	$<2.2\cdot10^{-4}$;$<2.6\cdot10^{-4}$
724	24	$1.2\cdot10^{-2}$	$9.0\cdot10^{-4}$
735	24	$8.0\cdot10^{-3}$; $1.2\cdot10^{-2}$	$6.0\cdot10^{-4}$; $9.0\cdot10^{-4}$
1004	24	$6.0\cdot10^{-3}$	$4.5\cdot10^{-4}$
1018	24	$5.0\cdot10^{-3}$; $6.0\cdot10^{-3}$	$3.8\cdot10^{-4}$; $4.5\cdot10^{-4}$
499	100	$<4.0\cdot10^{-3}$	$<3.0\cdot10^{-4}$
505	100	$<2.5\cdot10^{-3}$;$<2.5\cdot10^{-3}$	$<1.9\cdot10^{-4}$;$<1.9\cdot10^{-4}$
724	100	$7.0\cdot10^{-3}$	$5.2\cdot10^{-4}$
735	100	$5.0\cdot10^{-3}$; $1.1\cdot10^{-2}$	$3.8\cdot10^{-4}$; $8.2\cdot10^{-4}$
1004	100	$4.2\cdot10^{-2}$	$3.1\cdot10^{-3}$
1018	100	$4.0\cdot10^{-2}$; $1.9\cdot10^{-1}$	$3.0\cdot10^{-3}$; $1.4\cdot10^{-2}$

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Strips of a Nb sheet were placed in a capsule made of Armco Fe which had been filled with Li in Ar atmosphere. The capsule was then degassed and the Li melted in a pot furnace. The capsule which had been welded at the top was heated in a vacuum furnace with stainless steel plates for which the averaged temperatures were adjusted. The temperature was kept for equilibration, finally the core of the furnace was air cooled. The capsule was weighed and opened, the sample was leached out by distilled H_2O, Nb remained undissolved and was removed with the capsule, dried and weighed as the tare to determine the amount of Li in the capsule. The aqueous solution was filtered, and the residue was spectrographycally analyzed for its Nb content

SOURCE AND PURITY OF MATERIALS:

Nb: purity not specified.
Li: with contents of 0.24 % O, 0.02 % N, and 0.005 % Na.

ESTIMATED ERROR:

Solubility: precision typically ±30 % (by compilers).
Temperature: ±20 K.

REFERENCES:

COMPONENTS:	**EVALUATOR:**
(1) Niobium; Nb; [7440-03-1] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland December 1990

CRITICAL EVALUATION:

According to theoretical predictions by Kuzin et al.(1) the solubility of Nb in liquid Na is expected to be very small ($5.7 \cdot 10^{-15}$ and $1.8 \cdot 10^{-5}$ mol % Nb at 873 and 1473 K, respectively). Significantly higher contents of Nb in Na were observed in experimental studies. A reason for this difference might be due to the formation of NbO_2, NbO, Na_3NbO_4 or the solid solution of O in Nb (2), if O is present either in Nb or Na. These oxides undergo a transfer into the liquid Na phase which increases the apparent equilibrium concentration of Nb in Na. This dissolution mechanism was supported by Nb solubility measurements performed by Klueh (3-5) who observed an increase of the Nb solubility at 873 K of $8.4 \cdot 10^{-3}$ to $5.5 \cdot 10^{-2}$ mol % Nb, if the O content in Na increased from 0.0072 to 0.32 mol %. Higher primary contents of O in solid Nb also caused higher Nb solubility in liquid Na, but the effect of O in Na is predominant.

Three other sets of determinations (6-8,15),(9, 10) and (17,18) were performed in different laboratories. We assume, that the O level was kept constant in the experiments of (6-8, 15), since no information on the purity of Na is given. The corresponding solubility results were scattered between $1.7 \cdot 10^{-4}$ and $1.2 \cdot 10^{-2}$ mol % Nb at temperatures varying between 1073 and 1654 K. The data (6-8) fit quite well to Eq.(1) (by the evaluators), if the values at 1073-1076 K are neglected, and the equilibration time is shorter than 8 h.

$$\log(soly/\text{mol \% Nb}) = 2.74 - 8470(T/\text{K})^{-1} \qquad r = 0.953 \qquad \text{Eq.(1)}$$

The determinations of (9,10) were performed at the very low level of $8 \cdot 10^{-5}$ mol % O. The extrapolated Nb solubility values of (4, 5) were in acceptable agreement with those of (9,10) at this low O content at 873 K. At higher temperatures the results in (9, 10) were scattered for more than ± 50 % from the average. The results of Künstler (17,18) at 623 to 973 K are in acceptable agreement with the data of (5), since (17,18) used Nb_2O_5 as the solute and, therefore, the system contained a significant source of O. The data of (6-8) are regarded as the most reliable results, since they are self-consistent and close to the theoretically predicted solubility vs. temperature function of (1). The 1% Zr content in the solute does not change the chemical activity of Nb, but does decrease effectively the O activity in Nb and the O concentration in Na. This inhibits excessive dissolution of Nb-O complexes in Na. In a liquid reflux capsule made of Nb-Zr(1%) $(1.2\text{-}2.5) \cdot 10^{-3}$ mol % Nb was determined in Na by (14) after 120 hours at 1477 K, if the O level was below $1.4 \cdot 10^{-3}$ mol % O. Since further details are not provided, the report (14) is not compiled. Undetectable amounts of Nb were dissolved during 1 h equilibration of the metals at 973-1023 K (19).

Barker (11) and Mathews (20) confirmed the observations of (2) and (16) that Nb saturated with O (at lower O level) or Na_3NbO_4 (at higher O level) are the solid phases in equilibrium with the saturated solution of Nb in liquid Na at 873 K. The authors of (18) were not able to identify precisely the composition and structure of the equilibrium phase in their experiments. An influence of other contaminants on the solubility equilibria, such as N or C, seems to be negligible under the experimental conditions.

A schematic phase diagram of the Nb-Na system was presented in (12), it is quite similar to the Nb-Li phase diagram. No Nb-Na intermetallics are formed in this system (13).

Doubtful values of the solubility of Nb in liquid Na:

T/K	$soly$/mol % Nb	source	remarks
1273	$1 \cdot 10^{-4}$	Eq.(1)	at constrain pressure
1473	$8 \cdot 10^{-4}$	(8), Eq.(1)	at constrain pressure
1673	$5 \cdot 10^{-3}$	Eq.(1)	at constrain pressure
1773	$1 \cdot 10^{-2}$	(8), Eq.(1)	at constrain pressure

References

1. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113.
2. Kassner, T.F.; Smith, D.L. *US Atom.Ener.Comm. Rep. ANL-7335*, 1967.
3. Klueh, R.L. *US Atom.Ener.Comm. Rep. ORNL-4350*, 1969, p. 125.
4. Klueh, R.L. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt.I, 1969, p.171.
5. Klueh, R.L. *Corrosion* 1971, *27*, 342.
6. Ewing, C.T.; Stone, J.P.; Spann, J.R.; Steinkuller, E.W.; Kovacina, T.A.; Miller, R.R. *US Naval Res.Lab. Rep. NRL-5844*, 1962.
7. Kovacina, T.A.; Ewing, C.T.; Stone, J.P.; Miller, R.R. *US Naval Res.Lab.Rep. NRL-5904*, 1963.
8. Kovacina, T.A.; Miller, R.R. *US Naval Res.Lab. Rep. NRL-6051*, 1964.
9. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt.I, 1969, p.319.
10. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. AI-AEC-12955*, 1970.
11. Barker, M.G. *Rev.Intern.Hautes Temp.Refract.* 1979, 16, 237.
12. Smith, J.F.; Lee, K.J. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed. Am. Soc. Met., Metals Park, 1986, p.1659; *Bull.Alloy Phase Diagr.* 1988, *9*, 479.
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14. Fleitman, A.; Romano, A.; Klamut, C. *US Atom.Ener.Comm. Rep. TID-7626*,Pt.I, 1962, p. 23.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Sodium; Na; [7440-23-5]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
December 1990

CRITICAL EVALUATION: (continued)

References

15. Ewing, C.T.; Stone, J.P.; Spann,J.R.; Kovacina, T.A.; Miller, R.R. *US Naval Res.Lab. Rep. NRL-5964*, 1963.
16. Thorley, A.W.; Tyzack, C. 5th *European Congress of Corrosion*, Paris, 1973, p. 259.
17. Künstler, K. *Akad. Wissensch. DDR Rep. ZfK-340*, 1977, p. 44.
18. Künstler, K.; Ullmann, H. *Akad. Wissensch. DDR Rep. ZfK-337*, 1977, p. 46.
19. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad.Nauk SSSR, Met.* 1982, no. 1, 133.
20. Mathews, C.K. *High Temp.Sci.* 1988-89, *26*, 377.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Sodium; Na ; [7440-23-5]

ORIGINAL MEASUREMENTS:

Künstler, K.

Akad. Wissensch. DDR Rep. ZfK-340, 1977, p. 44-46.

VARIABLES:

Temperature: 623-973 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Nb content in liquid Na, which was equilibrated with Nb2O5 for more than 12 days, was determined.

t/°C	*soly*/mass % Nb [a]	*soly*/mass % Nb [b]	*soly*/mol % Nb [c]
350	$1.2\cdot10^{-3}$, $1.7\cdot10^{-3}$, $2.0\cdot10^{-3}$	$1.5\cdot10^{-3}$	$3.8\cdot10^{-4}$
500	$3.8\cdot10^{-3}$, $4.5\cdot10^{-3}$	$4.5\cdot10^{-3}$	$1.1\cdot10^{-3}$
700	$2.1\cdot10^{-2}$, $2.2\cdot10^{-2}$, $2.7\cdot10^{-2}$, $2.9\cdot10^{-2}$	$2.4\cdot10^{-2}$	$6.0\cdot10^{-3}$

[a] individual results read out from the figure
[b] mean results, presented in numerical form by the authors
[c] as calculated from the mean result by the compilers

If the equilibration lasted less than 12 days, the measured concentrations of Nb were significantly lower, by a factor of 0.1.
The same results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The determinations were carried out in a X8 CrNiTi 18 10 stainless steel capsule. Nb was introduced in form of Nb_2O_5 with ^{95}Nb as isotopic tracer. The capsule was loaded with a Nb_2O_5 pellet and a Na sample in an inert atmosphere, inside a special glass apparatus. The capsule was closed by welding with a plug and conditioned at the selected temperature for periods of time equal or longer than 12 days. The capsule was then quenched in liquid N. Only the central part of the solidified Na was taken for analysis. The Na sample was dissolved in CH_3OH-H_2O (3:1) solvent.

The radio-activity of the precipitated deposit in the resulting solution was counted with a Ge(Li) detector.

SOURCE AND PURITY OF MATERIALS:

Nb_2O_5: not specified.
Na: containing $1.5\cdot10^{-3}$ % O and $1.2\cdot10^{-3}$ % C.

ESTIMATED ERROR:

Solubility: precision ± 20 %.
Temperature: nothing specified.

REFERENCES:

1. Künstler, K.; Ullmann, H. *Akad.Wissensch. DDR Rep. ZfK-337*, 1977, p. 46.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Kovacina, T.A.; Miller, R.R.

US Naval Res.Lab. Rep. NRL- 6051, 1964.

VARIABLES:

Temperature: 1073-1654 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid Na was determined at various temperatures, the alloy Nb-Zr(1%) was equilibrated with Na. The data above 804 °C (1077 K) were obtained at constrained pressure to keep Na in the liquid state.

t/°C	*equilibration time*/h	*soly*/mass % Nb	*soly*/mol % Nb [a]
800[b], 802	2	$1.78\cdot10^{-2}$	$4.4\cdot10^{-3}$
803, 804[c]	8	$1.79\ 10^{-3}$	$4.4\cdot10^{-4}$
898[d]	8	$9.33\cdot10^{-3}$	$2.5\cdot10^{-4}$
1008	8	$7.5\cdot10^{-4}$, $7.4\cdot10^{-4}$ [c]	$1.8\cdot10^{-4}$
1151[d]	2.5	$2.38\cdot10^{-3}$	$6.0\cdot10^{-4}$
1181	8	$3.52\cdot10^{-3}$	$8.7\cdot10^{-4}$
1185, 1175[b]	2	$1.2\cdot10^{-3}$	$3.0\cdot10^{-4}$
1196[c]	8	$3.5\cdot10^{-3}$	$8.7\cdot10^{-4}$
1248[d]	3.5	$5.32\cdot10^{-3}$	$1.3\cdot10^{-3}$
1372[d]	3	$4.14\cdot10^{-3}$	$1.04\cdot10^{-3}$
1375[b]	2	$1.99\cdot10^{-1}$	$5.0\cdot10^{-2}$
1380, 1381[c]	8	$2.43\cdot10^{-2}$	$6.0\cdot10^{-4}$

[a] as calculated by the compilers
[b] as reported in (1)
[c] as reported in (2)
[d] from density experiments in (1)
The results which are not marked [b],[c], or [d] were reported in (3).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility apparatus containing a test crucible of Nb-Zr(1%) alloy and a recipient of Mo was filled with Na and sealed in an Ar atmosphere. It was wrapped with a Ti foil and placed in a pressure furnace. The system was heated to the desired temperature which was observed and controlled by a Pt/Pt-Rh(10%) thermocouple. The equilibration time was 2 - 8 h. At the end the apparatus was inverted to allow the Na to drain into the recipient. It was opened after cooling to room temperature. The Na samples were dissolved, diluted to volume, and an aliquot was titrated to determine the mass of the sample. Nb was separated from the aqueous solutions by means of co-precipitation with $Fe(OH)_3$. The $Fe(OH)_3$ was transferred to Fe_2O_3, in which form the sample was 15 min irradiated in the NRL reactor and analyzed by the standard comparator technique for ^{94}Nb beta activity.

The solubility of Nb in Na was calculated from the two analyses. Details concerning the analysis of Na of the density experiments were not reported.

SOURCE AND PURITY OF MATERIALS:

Nb-Zr(1%): 99 % Nb, 1 % Zr.
Na: vacuum distilled from a Ni still and filtered through a fine porosity Pyrex glass frit at 883 K.
Ar: purified by passing through a molecular sieve and heated Ti sponge.

ESTIMATED ERROR:

Solubility: detection limit $2\cdot10^{-6}$ mol % Nb.
Temperature: nothing specified.

REFERENCES:

1. Ewing, C.T.; Stone, J.P.; Spann, J.R.; Steinkuller, E.W.; Kovacina, T.A.; Miller, R.R. *Rep. NRL- 5844*, 1962.
2. Kovacina, T.A.; Ewing, C.T.; Stone, J.P.; Miller, R.R. *Rep. NRL- 5904*, 1963.
3. Ewing, C.T.; Stone, J.P.; Spann, J.R.; Kovacina, T.A.; Miller, R.R. *Rep. NRL- 5964*, 1963.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.

US Atom.Ener.Comm.Rep. AI-AEC-12955, 1970.

VARIABLES:

Temperature: 873-1173 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid Na at various temperatures was determined.

t/°C	*soly* /mass % Nb	*soly* /mol % Nb [a]
600 [b]	$2.8\cdot10^{-3}$	$6.9\cdot10^{-4}$
700 [b]	$7.2\cdot10^{-3}$	$1.8\cdot10^{-3}$
750	$2.85\cdot10^{-3}$	$7.1\cdot10^{-4}$
800 [b]	$8.8\cdot10^{-3}$	$2.2\cdot10^{-3}$
800 [b]	$3.1\cdot10^{-3}$	$7.7\cdot10^{-4}$
850	$6.63\cdot10^{-3}$	$1.6\cdot10^{-3}$
900 [b]	$8.8\cdot10^{-3}$	$2.2\cdot10^{-3}$
950	$2.86\cdot10^{-3}$	$7.1\cdot10^{-4}$

[a] as calculated by the compilers
[b] also reported in (1)

Combining the data of this work and those of Kovacina and Miller (2) the authors constructed the fitting equation:

$$\log(soly/\text{mass \% Nb}) = -1.8 - 654\,(T/\text{K})^{-1}$$

(confirmed by the compilers)

However, the results of this study and of (2) are largely scattered.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Nb crucible was cleaned in a HCl-H_2O (2:3) mixture followed by H_2O and acetone washing. A sample collector made of Ti was cleaned in a HNO_3-HF (3%) mixture. The crucible-collector assembly was outgassed for 2 hours at 1173 K. The assembly was filled with Na and sealed by means of welding under high vacuum. The capsule was equilibrated at the desired temperature for 6 hours. The capsule was then inverted, to cause the Na sample to flow into the collector. The entire sample of Na was analyzed after solidification by means of a spectrophotometric determination.

SOURCE AND PURITY OF MATERIALS:

Nb: 99.983 % purity, supplied by Materials Research Corp.; containing $8\cdot10^{-4}$ % C, $4\cdot10^{-5}$ % H, $4\cdot10^{-4}$ % O, $2.34\cdot10^{-3}$ % N, $1\cdot10^{-2}$ % Ta, $6.4\cdot10^{-4}$ % W ; other elements < $8\cdot10^{-5}$ %, each.
Na: 99.996 % purity, purified by hot gettering and fractional distillation, with $8\cdot10^{-4}$ % C, $6\cdot10^{-5}$ % O as major impurities; stored under high vacuum.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt.I, 1969, p. 319-324.
2. Kovacina, T.A.; Miller, R.R. *US Atom. Ener.Comm. Rep. NRL- 6051*, 1964.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Sodium ; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Klueh, R.L.

Corrosion 1971, *27*, 342-346.

VARIABLES:

One temperature: 873 K
O concentration in Na: 0.007 - 0.32 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid Na at 600 °C and various O concentrations in Na is reported:

O concn/mass %	*soly*/mass % Nb	*soly*/mol % Nb [b]
0.005	$4.0\cdot10^{-2}$	$9.9\cdot10^{-3}$
0.01	$3.5\cdot10^{-2}$	$8.7\cdot10^{-3}$
0.01 (0.013) [a]	$3.4\cdot10^{-2}$	$8.4\cdot10^{-3}$
0.015	$4.0\cdot10^{-2}$	$9.9\cdot10^{-3}$
0.02	$5.0\cdot10^{-2}$	$1.2\cdot10^{-2}$
0.03 [c]	$8.0\cdot10^{-2}$	$2.0\cdot10^{-2}$
0.05 (0.045) [a]	0.10	$2.5\cdot10^{-2}$
0.07 [c]	0.13	$3.2\cdot10^{-2}$
0.08 (0.07) [a]	$7.7\cdot10^{-2}$	$1.9\cdot10^{-2}$
0.1 [c]	0.22	$5.5\cdot10^{-2}$
0.2	0.17	$4.2\cdot10^{-2}$
0.22	0.14	$3.5\cdot10^{-2}$
0.005 [c]	$5.0\cdot10^{-2}$ [d]	$1.2\cdot10^{-2}$
0.005 [c]	$1.0\cdot10^{-1}$ [e]	$2.5\cdot10^{-2}$

[a] - as given in the figure

[b] - as calculated by the compilers

[c] - reported also in (2)

[d] - O concentration in Nb equal 9.5 x 10^{-2} mass % (0.133 mol %)

[e] - O concentration in Nb of 0.16 mass % (0.22 mol %)

The results were presented in graphical form in (1).
An extrapolation of the data by the author to the level of $1\cdot10^{-4}$ mass % O results in a solubility value of $1\cdot10^{-3}$ mass % or $2.5\cdot10^{-4}$ mol % Nb (as calculated by the compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The applied system consisted of a Nb specimen in contact with liquid Na in a Nb container (a container of the same material) which in turn, was encapsuled in stainless steel. The capsule was lined with a Ta foil, filled with liquid Na and welded in an Ar atmosphere chamber, to prevent contamination of O or Nb. The concentration of O was varied by adding N_2O (in order to determine the effect of O in Na). The system was equilibrated at 873 K for 500 hours. After testing the capsule was quenched in liquid N and opened in an Ar atmosphere chamber. Na was removed by dissolving in chilled isoprppyl alcohol and recovered from it as chloride. The amount of Nb in Na was determined by spectrosgraphic analysis.

SOURCE AND PURITY OF MATERIALS:

Nb: 99.9 % purity, containing $7\cdot10^{-3}$ % O.
Na: hot gettered with a Zr foil contact.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: accuracy of individual analyses ± 10 %.
Temperature: nothing specified.

REFERENCES:

1. Klueh, R.L. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt.I, 1969, p.171-176.
2. Klueh, R.L. *US Atom.Ener.Comm. Rep. ORNL-4350*, 1969, p. 125-126.

COMPONENTS:	EVALUATOR:
(1) Niobium; Nb; [7440-03-1] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland December 1989

CRITICAL EVALUATION:

Investigations of the solubility of Nb in liquid K are strongly related to the influence of O on the equilibria. The predicted temperature dependence of the solubility of Nb in liquid K is very large (1), if the two elements are of absolute purity. The solubility should be below the detection level of the analytical methods at temperatures below 1000 K. The concentrations of Nb which were measured in real Nb-K systems were some orders of magnitude higher than the predicted ones. This fact may be explained on the basis of the formation of K-Nb mixed oxides (2) or of the depression of the chemical activity of Nb in K due to the influence of O (3). Five independent working groups were concerned with the solubility of Nb in liquid K; the resulting information is, however, published in a large number of reports (3-14). Ginell and Teitel (4-7) determined the solubility of Nb in the temperature range 1366-1603 K at an O level of approximately $2 \cdot 10^{-3}$ mol %. These data are in fair agreement with each other, and may be regarded as tentative. Cleary et al. (8-10) published numerous results on the Nb solubility in K in the temperature range 1033 to 1478 K. The initial O content in K used in these studies of $5 \cdot 10^{-3}$ mol % was increased to 1.22 mol % by an addition of K_2O as well as K_2CO_3 or KNO_3. The average results were not strongly dependent on the temperature or the O content in K. The scatter of values was, however, as large as one order of magnitude. An extrapolation of the data (by the compilers) provided a solubility value of $2 \cdot 10^{-4}$ mol % at an O concentration of $2 \cdot 10^{-4}$ mol % at 1368 K. This result is in fair agreement with the results of (4-7). Nevertheless, the Nb solubility increased spectacularly from $3 \cdot 10^{-3}$ to 0.3 mol % with increasing O concentration (from 0.73 to 1.22 mol %) which might be due to changes in the dissolution mechanism in the system. Cleary et al.(8-10) tried to study the effect of C and N in K on the Nb solubility (introducing K_2CO_3 or KNO_3 into the solvent). They noticed only slight changes within the experimental scatter. Thus, it can be concluded that O has a predominant influence on the solubility of Nb in K.

The influence of O was confirmed in the experiments performed by Litman (11) at temperatures of 473 to 1088 K. The results are acceptably consistent. Extrapolation (made by the evaluators) of the solubility data at 673 and 873 K to an O concentration of $2 \cdot 10^{-4}$ mol % gave Nb concentrations of $2 \cdot 10^{-5}$ and $2 \cdot 10^{-4}$ mol %, respectively. It was observed that the initial concentration of O in solid Nb has little effect on the solubility of Nb in liquid K.

McKisson et al. (12-14) applied K of the highest purity with $8 \cdot 10^{-4}$ mol % O for their determinations. They found, in experiments at 1273 and 1473 K, a lower Nb solubility at the higher temperature. Even at an O concentration of $3 \cdot 10^{-3}$ mol % the Nb solubility was similarly low. A fair agreement in their results with the data of Ginell and Teitel (4-7) was achieved, when Nb-Zr(1%) or Nb-Zr(1/2%) was used as solute instead of pure Nb. It is evident that the presence of Zr, a very active getter, significantly decreased the O activity in Nb and K. The Nb-K system fitted under these conditions better to the predictions of (1).

Klueh's (3) study on the influence of O on the solubility of Nb in K at 873 K confirmed the results obtained by Litman (11). The Nb solubility data, extrapolated by (3) to the O concentration of $2 \cdot 10^{-4}$ mol %, was $2 \cdot 10^{-4}$ mol % Nb. An abrupt increase of the Nb solubility at increasing O concentrations to even 4.5 mol % could not be observed, in contradiction to the results of (8-10) at 1368 K. Thus, the degree of influence of O on the Nb solubility in K seems to be dependent on the temperature.

Stecura (15) determined the solubility of Nb in liquid K (containing $3.6 \cdot 10^{-3}$ mol % O) at temperatures 1055 to 1287 K and observed a smooth increase of the Nb equilibrium concentration from $1.9 \cdot 10^{-3}$ to $8.0 \cdot 10^{-3}$ mol %, if the value at 1162 K would be excluded. The dissolution of Nb in liquid K was further increased, if the solute contained up to 0.16 mol % O. However, even after an equilibration of 96 hours, an equilibrium could not be achieved in the system. The results of (15) are in agreement with those of (11) and the extrapolated values of (3), they are, however, slightly higher than those of (8-10).

According to Barker (2) and Litman (11), the solid phases in equilibrium are metallic Nb, a solid solution of O in Nb, Nb oxides or K_3NbO_4, if the O concentration exceeded $2 \cdot 10^{-3}$ mol %. Cleary et al. (8-10) and Stecura (15) identified the compound $KNbO_3$ which was in equilibrium with liquid K containing O, and metallic Nb.

Aleksandrov and Dalakova (16) did not observe any dissolution of Nb in liquid K after equilibrating the metals for 1 hour at 873-923 K. The detection level of the spectral analysis used to analyse K for the content of Nb was not specified.

The solubility data selected by the evaluators were obtained at $\leq 1 \cdot 10^{-3}$ mol % O. All experiments which were performed at a temperature above 1032 K were carried out at the vapor pressure of K.

Stoop et al. (17) detected neither intermetallic phases nor solid solution of K in Nb. Smith and Lee (18) presented a schematic phase diagram of the Nb-K system which is analogous to that shown for the Nb-Li system with deviations due to the boiling and melting points of K at 1032 and 336.9 K, respectively.

Tentative values of the solubility of Nb in liquid K at $\leq 1 \cdot 10^{-3}$ mol % O.

T/K	*soly*/mol % Nb	source
1273	$4 \cdot 10^{-5}$	(13) with Nb-Zr alloy as solute
1373	$2 \cdot 10^{-4}$	(8-10) extrapolated to $2 \cdot 10^{-4}$ mol % O
1473	$3 \cdot 10^{-4}$	(13) with Nb-Zr alloy as solute
1603	$1 \cdot 10^{-3}$	(6)
1873	$8 \cdot 10^{-3}$	(14) with Nb-Zr alloy

COMPONENTS:	EVALUATOR:
(1) Niobium; Nb; [7440-03-1] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland December 1989

CRITICAL EVALUATION: (continued)

References

1. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Exsperimentalnye Metody Postroenia Diagram Sostoyania*, Nauka, Moskva, 1985, p. 113.
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4. Teitel, R.J. *Trans. Am. Nucl. Soc.* 1965, *8*, 15.
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10. Cleary, R.E.; Blecherman, S.S.; Corliss, J.E. *US Atom.Ener.Comm. Rep. TIM-850*, 1965.
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13. McKisson, R.L.; Eichelberger, R.L.; Dahleen, R.C.; Scarborough, J.M.; Argue, G.R. *NASA Rep. CR-610*, 1966; *Rep. AI-65-210*, 1966.
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COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Ginell, W.S.; Teitel, R.J.

Trans. Am. Nucl. Soc. 1965, *8*, 393-394.

VARIABLES:

Temperature: 1366-1603 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid K at various temperatures are reported:

t/°C	*soly*/mass % Nb	*soly*/mol % Nb [f]
1093	$1.0\cdot10^{-3}$ [a,c]	$4.2\cdot10^{-4}$
1130	$1.0\cdot10^{-3}$ [c,e]	$4.2\cdot10^{-4}$
1225	$2.8\cdot10^{-3}$ [c,e]	$1.2\cdot10^{-3}$
1225	$9\cdot10^{-4}$ [c,e]	$3.8\cdot10^{-4}$
1245	$1.4\cdot10^{-3}$ [d,e]	$5.9\cdot10^{-4}$
1245	$1.3\cdot10^{-3}$ [d,e]	$5.5\cdot10^{-4}$
1245	$7\cdot10^{-4}$ [d,e]	$2.9\cdot10^{-4}$
1330	$3.1\cdot10^{-3}$ [b,c]	$1.3\cdot10^{-3}$

[a] reported exclusively in (1)
[b] reported exclusively in (3)
[c] equilibration 1 hour, centrifugation 3 hours
[d] equilibration 3 hours, centrifugation 6 hours
[e] reported in (3)
[f] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An L-shaped test capsule fabricated of a Nb-Zr(1%) alloy was placed in a centrifuge. A small dam was installed inside the capsule to regulate the amount of K that might drain into the collector part of the capsule which was fabricated of W. The capsule material was chemically etched inside and vacuum treated at the maximum temperature of the experiment. The capsule containing a Nb sample was filled with K, welded and heated for 1 hour at 100 K above the equilibration temperature, while the centrifuge rotated to prevent K from flowing over the dam. The test temperature was then kept for 3-6 hours and the rotation rate of the centrifuge was increased to force the solute precipitation to the bottom of the sample crucible. When the rotation rate was slowly reduced, the K solution was decanted and drained into the collector. The method of the further chemical analysis was not specified. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Nb: 99.9 % purity.
K : purified by hot trapping with Ti-Zr alloy chips at 1058 K; $(7-11)\cdot10^{-4}$ % O in [d] and $2.3\cdot10^{-3}$ % O in [c].
Ar: "high purity", dried and passed over Ti-Zr alloy chips at 1173 K, contained < $2\cdot10^{-4}$ mol % H_2O.

ESTIMATED ERROR:

Nothing specified.
Solubility: accuracy up to ± 50 % (by the compilers).

REFERENCES

1. Teitel, R.J. *Trans. Am. Nucl. Soc.* 1965, *8*, 15.
2. Ginell, W.S.; Teitel, R.J. *Douglas Aircraft Comp. Rep. SM-48883*, 1965.
3. Ginell, W.S.; Teitel, R.J. *US Atom.Ener. Comm. Rep. CONF-650411*, 1965, p. 44-47.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Niobium; Nb; [7440-03-1] (2) Potassium; K; [7440-09-7]	Cleary, R.E.; Blecherman, S.S.; Corliss, J.E. *US Atom.Ener.Comm. Rep. TIM-850*, 1965.
VARIABLES: Temperature: 1033-1478 K; O concentration in K: $4.9\cdot10^{-3}$ - 1.22 mol %	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid K as a function of temperature and O content in K is reported:

t/°C	*add.of O*/mass %	*add.of O*/mol % [a]	*soly*/mass % Nb	*soly*/mol % Nb [a]
760	-	-	$4.0\cdot10^{-3}$	$1.6\cdot10^{-3}$
760	-	-	$4.1\cdot10^{-2}$	$1.7\cdot10^{-2}$
760	-	-	$1.42\cdot10^{-2}$	$6.0\cdot10^{-3}$
760	$5.0\cdot10^{-2}$ /KO_2/	0.122	0.12	$5.1\cdot10^{-2}$
760	"	"	$2.3\cdot10^{-3}$	$9.7\cdot10^{-4}$
760	"	"	$8.9\cdot10^{-2}$	$3.7\cdot10^{-2}$
760	$5.0\cdot10^{-2}$ /K_2CO_3/	"	$8.4\cdot10^{-3}$	$3.5\cdot10^{-3}$
760	$5.0\cdot10^{-2}$ /KNO_3/	"	$2.35\cdot10^{-2}$	$9.9\cdot10^{-3}$
980	-	-	$4.1\cdot10^{-3}$	$1.7\cdot10^{-3}$
980	-	-	$9.6\cdot10^{-3}$	$4.0\cdot10^{-3}$
980	$5.0\cdot10^{-2}$ /KO_2/	0.122	$1.5\cdot10^{-3}$	$6.3\cdot10^{-4}$
980	$5.0\cdot10^{-2}$	"	$3.8\cdot10^{-2}$	$1.6\cdot10^{-2}$
980	$5.0\cdot10^{-2}$ /K_2CO_3/	"	$2.92\cdot10^{-2}$	$1.2\cdot10^{-2}$
980	$5.0\cdot10^{-2}$ /KNO_3/	"	0.133	$5.6\cdot10^{-2}$
1095	-	-	$6.3\cdot10^{-3}$	$2.6\cdot10^{-3}$
1095	-	-	$9.36\cdot10^{-2}$	$3.9\cdot10^{-2}$
1095	-	-	$6\cdot10^{-4}$	$2.5\cdot10^{-4}$
1095	-	-	$9.8\cdot10^{-3}$	$4.1\cdot10^{-3}$
1095	$5.0\cdot10^{-2}$ /KO_2/	0.122	$1.1\cdot10^{-3}$	$4.6\cdot10^{-4}$
1095	$5.0\cdot10^{-2}$	"	$5.2\cdot10^{-3}$	$2.2\cdot10^{-3}$
1095	$5.0\cdot10^{-2}$ /K_2CO_3/	"	$3.7\cdot10^{-3}$	$1.6\cdot10^{-3}$
1095	$5.0\cdot10^{-2}$ /KNO_3/	"	$2.8\cdot10^{-3}$	$1.2\cdot10^{-3}$
1095	0.100 /KO_2/	0.244	$1.5\cdot10^{-3}$	$6.3\cdot10^{-4}$
1095	"	"	$1.6\ 10^{-3}$	$6.8\cdot10^{-4}$
1095	"	"	$8.0\cdot10^{-3}$	$3.4\cdot10^{-3}$
1095	0.200 /KO_2/	0.488	$1.7\cdot10^{-3}$	$7.1\cdot10^{-4}$
1095	0.300 /KO_2/	0.732	$2.3\cdot10^{-3}$	$9.7\cdot10^{-4}$
1095	"	"	$6.7\cdot10^{-3}$	$2.8\cdot10^{-3}$
1095	0.500 /KO_2/	1.22	0.86	0.36
1095	"	"	0.73	0.31
1150	-	-	$1.6\cdot10^{-2}$	$6.8\cdot10^{-3}$
1205	-	-	$1.4\cdot10^{-3}$	$5.9\cdot10^{-4}$
1205	-	-	$3.4\cdot10^{-3}$	$1.4\cdot10^{-3}$
1205	$5.0\cdot10^{-2}$ /KO_2/	0.122	$1.6\cdot10^{-3}$	$6.8\cdot10^{-4}$
1205	$5.0\cdot10^{-2}$	"	$3.8\cdot10^{-3}$	$1.6\cdot10^{-3}$
1205	$5.0\cdot10^{-2}$ /K_2CO_3/	"	$4.4\cdot10^{-3}$	$1.9\cdot10^{-3}$
1205	$5.0\cdot10^{-2}$ /KNO_3/	"	$2.3\cdot10^{-3}$	$9.7\cdot10^{-4}$

[a] as calculated by the compilers

$KNbO_3$ and an unidentified hexagonal close packed phase were detected in the equilibrium solid product. The solubility of Nb in liquid K was determined to be below $2.0\cdot10^{-3}$ mass % or $8.4\cdot10^{-4}$ mol % Nb, as calculated by the compilers, in the temperature range of 760 - 1205°C as reported in (1).
The results obtained at 1095°C (1368 K) were also published in graphical form in (1).
Compilers' extrapolation of the lowest Nb solubility data at 1368 K in the O concentration range in K of 0.732 to $2\cdot10^{-4}$ mol % gives a concentration of Nb in K of $2\cdot10^{-4}$ mol %.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Cleary, R.E.; Blecherman, S.S.; Corliss, J.E.

US Atom.Ener.Comm. Rep. TIM-850, 1965.

VARIABLES:

Temperature: 1033-1478 K;
O concentration in K: $4.9 \cdot 10^{-3}$ - 1.22 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES: (continued)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

K was introduced into a Nb cup being mounted in a Mo receiver capsule. The capsule was sealed in an Ar atmosphere and placed in an Inconel shroud filled with Ar. The capsule was equilibrated for 100 hours at a selected temperature in the receiver-up position. Then the capsule was inverted, so the solution of Nb in liquid K could flow into the receiver part of the capsule. The solidified solution was dissolved in absolute C_2H_5OH or liquid NH_3. The receiver cup was etched with a HNO_3-HCl mixture. Both solutions were joined, a Nb N-benzoyl, N-phenyl-hydroxyloamine complex was formed and extracted into chloroform. The extract was evaporated and dissolved in a small volume of H_2O for spectrographic determination of Nb.

SOURCE AND PURITY OF MATERIALS:

Nb: containing $6.5 \cdot 10^{-3}$ % C, $7.0 \cdot 10^{-3}$ % N, $7.5 \cdot 10^{-3}$ % O, and $1.0 \cdot 10^{-3}$ % H.
K: purified by contacting with Ti sponge for 24 h at 1033 K, containing $< 2.5 \cdot 10^{-3}$ % O, $< 1 \cdot 10^{-4}$ % Nb, $< 5 \cdot 10^{-3}$ % C.
Ar: $< 2.5 \times 10^{-4}$ % O, $< 7.5 \times 10^{-4}$ % H_2O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Blecherman, S.S.; Schenck, G.F.; Cleary, R.E. *US Atom.Ener.Comm. Rep. CNLM-6335*, 1965; *Rep. CONF-650411*, 1965, p.48; *Rep. CONF-650411-4*, 1965.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Litman, A.P.

US Atom.Ener.Comm. Rep. ORNL-3751, 1965.

VARIABLES:

Temperature: 473-1088 K; O concentration in K: $1.22\cdot10^{-2}$-0.146 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Equilibrium concentrations of Nb in liquid K are reported as a function of the O content in K and temperature; they were read out from the figure and recalculated into mol % by the compilers:

t/°C	*O concn*/mass %	*O concn*/mol %	*soly*/ mol % Nb
200	$5\cdot10^{-3}$	$1.22\cdot10^{-2}$	$1.22\cdot10^{-3}$, $2.8\cdot10^{-3}$,
400	$5\cdot10^{-3}$	$1.22\cdot10^{-2}$	$8.0\cdot10^{-4}$, $4.5\cdot10^{-3}$, $5.2\cdot10^{-3}$
600	$5\cdot10^{-3}$	$1.22\cdot10^{-2}$	$6.2\cdot10^{-3}$, $7.0\cdot10^{-3}$
800	$5\cdot10^{-3}$	$1.22\cdot10^{-2}$	$1.55\cdot10^{-2}$
815	$5\cdot10^{-3}$	$1.22\cdot10^{-2}$	$1.72\cdot10^{-2}$
600	$4\cdot10^{-2}$	$9.8\cdot10^{-2}$	$2.2\cdot10^{-2}$
750	$4\cdot10^{-2}$	$9.8\cdot10^{-2}$	$2.4\cdot10^{-2}$
200	$4.5\cdot10^{-2}$	0.11	$2.1\cdot10^{-3}$, $3.7\cdot10^{-3}$
400	$4.5\cdot10^{-2}$	0.11	$1.6\cdot10^{-2}$, $1.9\cdot10^{-2}$
600	$4.5\cdot10^{-2}$	0.11	$2.0\cdot10^{-2}$
400	$6.0\cdot10^{-2}$	0.146	$3.1\cdot10^{-2}$
600	$6.0\cdot10^{-2}$	0.146	$3.9\cdot10^{-2}$

The concentrations of Nb, which had been extrapolated by the compilers to the O concentration of $2\cdot10^{-4}$ mol % at 673 and 873 K, were $2\cdot10^{-5}$ and $2\cdot10^{-4}$ mol % Nb, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test system consisted of a Nb specimen in contact with K contained in a Nb capsule. The Nb specimen, which had been degreased with acetone, vacuum annealed at 1473 to 1873 K and chemically polished in a HNO_3, HF, H_2O mixture, was placed in one part of the capsule. The capsule was encapsulated in an outer container of stainless steel and additionally wrapped with a Ta foil. The Nb specimen was restricted to the opposite end at the capsule from that part filled with K until the entire system was at test temperature. The assembly was then quickly inverted to allow liquid K to contact the specimen. The Temperature was controlled by Pt/Pt-Rh (10%) thermocouple. After completion (48-100 hours) the container was again inverted and quenched in cold oil. The capsule was cut open and the K remaining on the surface of the specimen was removed by dissolution in isopropyl alcohol. The Nb content of the alcohol solution was determined by a colorimetric procedure. O was introduced into the system in form of K_2O or KO_2. The experiments were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Nb: purity not specified, supplied by Wah Chang Corp., electron-beam remelted, containing $8.0\cdot10^{-3}$ % O, $2.5\cdot10^{-3}$ %N, $5\cdot10^{-4}$ % H, $6.0\cdot10^{-3}$ % C.
K: "low sodium grade", supplied by Mine Safety Appliances Res. Corp., containing, after filtration and gettering with Zr foil, $5.0\cdot10^{-3}$ % O, $<5\cdot10^{-4}$ % Nb, $\leq 2.0\cdot10^{-3}$ % of each transition metal.
K_2O: 98-99 % pure.
KO_2: 98-99 % pure.
Ar: 99.995 % pure.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 10 K.

REFERENCES:

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Potassium K; [7440-09-7]

ORIGINAL MEASUREMENTS:

McKisson, R.L.; Eichelberger, R.L.; Dahleen, R.C.; Scarborough, J.M.; Argue, G.R.
NASA Rep. CR-610, 1966; *Rep. AI-65-210*, 1966.

VARIABLES:

Temperature: 1273-1887 K
O concentration: $8.75\cdot10^{-4}$ - $3.5\cdot10^{-3}$ mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid K was determined.

t/°C	*O content*/mass %	*soly*/mass % Nb	*soly*/mol % Nb [a]
1000 [c]	$1.4\cdot10^{-3}$	$1.5\cdot10^{-3}$	$6.3\cdot10^{-4}$
1000	$1.4\cdot 10^{-3}$	$5.7\cdot10^{-3}$	$2.4\cdot10^{-3}$
1200	$1.4\cdot10^{-3}$	$3.3\cdot10^{-3}$	$1.4\cdot10^{-3}$
1200	$1.4\cdot10^{-3}$	$3.4\cdot10^{-3}$	$1.4\cdot10^{-3}$
1200	$1.4\cdot10^{-3}$	$6.1\cdot10^{-3}$	$2.6\cdot10^{-3}$
1000	$3.5\cdot10^{-4}$	$1.71\cdot10^{-2}$	$7.2\cdot10^{-3}$
1000	$3.5\cdot10^{-4}$	$8.8\cdot10^{-3}$	$3.7\cdot10^{-3}$
1200	$3.5\cdot10^{-4}$	$2.6\cdot10^{-3}$	$1.1\cdot10^{-3}$
1200	$3.5\cdot10^{-4}$	$7.2\cdot10^{-3}$	$3.0\cdot10^{-3}$
1200	$3.5\cdot10^{-4}$	$1.7\cdot10^{-3}$	$7.1\cdot10^{-4}$
1200	$3.5\cdot10^{-4}$	$2.9\cdot10^{-3}$	$1.2\cdot10^{-3}$
1000 [b]	$3.5\cdot10^{-4}$	$< 1\cdot10^{-4}$	$<4\cdot10^{-5}$ [b]
1200 [b]	$3.5\cdot10^{-4}$	$6\cdot10^{-4}$	$2.5\cdot10^{-4}$ [b]
1200 [b,e]	$5.0\cdot10^{-4}$	$6.0\cdot10^{-4}$	$2.5\cdot10^{-4}$ [b,e]
1382 [b,e]	$5.0\cdot10^{-4}$	$6.5\cdot10^{-4}$	$2.7\cdot10^{-4}$ [b,e]
1200 [d,e]	$5.0\cdot10^{-4}$	$1.0\cdot10^{-3}$	$4.2\cdot10^{-4}$ [d,e]
1417 [d,e]	$5.0\cdot10^{-4}$	$1.24\cdot10^{-3}$	$5.1\cdot10^{-4}$ [d,e]
1614 [d,e]	$5.0\cdot10^{-4}$	$1.91\cdot10^{-3}$	$8.1\cdot10^{-3}$ [d,e]

[a] - as calculated by the compilers
[b] - the solute source was a Nb-Zr(1%) alloy instead of pure Nb
[c] - as reported in (1)
[d] - the solute source Nb-Zr(1/2 %) alloy
[e] - as reported in (2)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test crucible was made from single crystal of Nb. The capsule body, capsule cup and collector were made of Mo. The crucible and the capsule parts were cleaned and rinsed with acetone. The apparatus was heated to a temperature of up to 2073 K, high vacuum outgassed and cooled in an Ar atmosphere. The parts were finally outgassed at 623 K to remove Ar. The purified K was filled into the crucible. The collector and the cup installed and the capsule was sealed under high vacuum by means of electron beam welding. The capsule was heated to the test temperature and kept in the cup-up position for 1/2 to 8 hours. The capsule was swung out from the furnace at the end of the test. This way liquid K from the capsule was transferred to the collector.

After the capsule had cooled, it was cracked open and the collector separated. A sample of K was melted from the collector into a special glassware. It reacted with H_2O vapour in He atmosphere, acidified with HCl and HF.

The K remaining in the collector was very slowly dissolved in H_2O and then rinsed with HCl. The Nb in the resulting solution was determined spectrophotometrically as thiocyanate complex which was extracted from the liquid solution with ethyl alcohol. High vacuum manipulators were used in all steps of experiments.

SOURCE AND PURITY OF MATERIALS:

Nb: 99.992 % purity, supplied by Material Research Corp.; containing $1.0\cdot10^{-3}$ % O, N, $8\cdot10^{-4}$ % C, < $1.0\cdot10^{-3}$ % Fe, $2.0\cdot10^{-3}$ % Mo, Ta, < $5\cdot10^{-4}$ % Si.
K: supplied by Mine Safety Appliance Res. Corp., further purified by filtering at 363 K, hot gettering with Zr at 923 K and distillation of the final product: containing $3.5\cdot10^{-4}$ % O, $4\cdot10^{-4}$ % N, $1.0\cdot10^{-3}$ % C, other elements ~ $1\cdot10^{-3}$ %, but typically few times lower.
He: unspecified.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: scatter of data within one order of magnitude.
Temperature: nothing numerically specified, precise control of temperature is reported.

REFERENCES:

1. McKisson, R.L.; Eichelberger, R.L. *NASA Rep. CR-54097*, 1965; *Rep. AI-65-93*, 1965.
2. Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969; *Rep. AI-68-110*, 1969.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Klueh, R.L.

Corrosion 1969, *25*, 416-422.

VARIABLES:

One temperature: 873 K
O concentration in K: ~ $2.4 \cdot 10^{-3}$ - 4.52 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid K at 873 K was determined as a function of the O content in K.

O concn/ mass %	*O concn*/mol % [a]	*soly*/mass % Nb	*soly*/mol % Nb [a]
$\leq 1.0 \cdot 10^{-3}$	$2.4 \cdot 10^{-3}$	$5.0 \cdot 10^{-3}$	$2.1 \cdot 10^{-3}$
$\leq 1.0 \cdot 10^{-3}$	$2.4 \cdot 10^{-3}$	$4.3 \cdot 10^{-2}$	$1.8 \cdot 10^{-2}$
$3.0 \cdot 10^{-3}$	$7.3 \cdot 10^{-3}$	$6.8 \cdot 10^{-2}$	$2.8 \cdot 10^{-2}$
$3.0 \cdot 10^{-3}$	$7.3 \cdot 10^{-3}$	$6.4 \cdot 10^{-2}$	$2.7 \cdot 10^{-2}$
$1.0 \cdot 10^{-2}$	$2.4 \cdot 10^{-2}$	$9.6 \cdot 10^{-2}$	$4.0 \cdot 10^{-2}$
$5.3 \cdot 10^{-2}$	0.13	0.106	$4.4 \cdot 10^{-2}$
0.101	0.24	0.219	$9.2 \cdot 10^{-2}$
0.203	0.49	0.338	0.14
0.39	0.95	0.36	0.15
1.9	4.5	1.34	0.57

[a] - as calculated by the compilers

The extrapolation of the data to a concentration of O of $1 \cdot 10^{-4}$ mass % ($2 \cdot 10^{-4}$ mol %) provides a saturation concentration of Nb of $4 \cdot 10^{-4}$ mass % ($2 \cdot 10^{-4}$ mol %) as was confirmed by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The applied system consisted of a Nb specimen in contact with liquid K in a Nb container (the container of the same material), which in turn, was encapsulated in stainless steel. The capsule was covered with a Ta foil, filled with liquid K and welded in an Ar atmosphere chamber (to prevent contamination of O or Nb). The concentration of O was varied by adding K_2O (in order to determine the effect of O in K). The system was equilibrated at 873 K for 500 hours. After testing the capsule was quenched in liquid N and opened in an Ar atmosphere chamber. K was removed by dissolving in chilled isopropyl alcohol and recovered from it as KF. The amount of Nb in K was determined by spectrographic analyses of the resulting KF.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.
Nb: probably the same as in (1); 99.9 % pure with $(3.2\text{-}7.0) \cdot 10^{-3}$ % O.

ESTIMATED ERROR:

Solubility: precision of analysis ± 10%.
Temperature: nothing specified.

REFERENCES:

1. Klueh, R.L. *Metall. Trans.* 1974, *5*, 875-879.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Stecura, S.

NASA Rep. TN-D-5875, 1970.

VARIABLES:

Temperature: 1055-1287 K.

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid K at various temperatures is reported.

T/K	*soly*/mass % Nb	*soly*/mol % Nb [a]
1287	$1.91\cdot10^{-2}$	$8.0\cdot10^{-3}$
1271	$1.80\cdot10^{-2}$, $1.74\cdot10^{-2}$	$7.4\cdot10^{-3}$
1162	$1.44\cdot10^{-2}$, $1.30\cdot10^{-2}$	$5.7\cdot10^{-3}$
1117	$8.2\cdot10^{-3}$, $6.6\cdot10^{-3}$	$3.1\cdot10^{-3}$
1069	$5.5\cdot10^{-3}$, $4.7\cdot10^{-3}$	$2.1\cdot10^{-3}$
1055	$4.6\cdot10^{-3}$, $4.44\cdot10^{-3}$	$1.9\cdot10^{-3}$

[a] - mean values calculated by the compilers

The results are fitted to the equation (as tested by the compilers):

$$\log(soly/\text{mol \% Nb}) = (0.84\pm0.30) - (3739\pm343)(T/\text{K})^{-1}$$

Using Nb samples doped with 0.067 - 0.157 mol % O the apparent content of Nb in liquid K increased, however, the data were time dependent even for 96 hours of test and therefore these results are not reproduced here.

K_3NbO_4 was recovered from liquid K and identified by chemical analysis and x-ray diffraction patterns.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Nb crucible and a Ni capsule were ultrasonically cleaned in CHF_3, a detergent and H_2O. The crucible was etched 2-5 min in 49 % HF and 1-2 min in HF, HNO_3, H_2O mixture (1:1:3). Both parts were rinsed with H_2O and dried under vacuum. The crucible was installed in the capsule; the upper part of the capsule served as a receiver. The crucible was filled with K in a vacuum chamber and the capsule was welded by electron beam. The capsule was placed in a furnace and heated for 24 hours. The temperature was measured with a Pt/Pt-Rh(13%) thermocouple. At the end of a test run the capsule was inverted and K collected in the receiver. After cooling to room temperature the capsule was cut open. K was dissolved in butyl alcohol and traces of Nb were leached from the capsule walls with HCl solution. Both solutions were combined and K was converted to KCl. The Nb concentration was determined by spectrophotometric analysis.

SOURCE AND PURITY OF MATERIALS:

Nb: polycrystalline, with $7\cdot10^{-4}$ % C, $6.4\cdot10^{-3}$ % O, $4.1\cdot10^{-3}$ % N, $1.4\cdot10^{-3}$ % H, $2\cdot10^{-2}$ % Ta, $6\cdot10^{-3}$ % W, other metals below $1.0\cdot10^{-3}$ % each.

K: 99.99 % pure, with $1.5\cdot10^{-3}$ % O, $2\cdot10^{-4}$ % N, $5\cdot10^{-3}$ % Rb, other metals below $3\cdot10^{-3}$ % each.

ESTIMATED ERROR:

Solubility: precision of analysis ± 8 %.

Temperature: accuracy ± 8 K, stability ± 4 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Niobium; Nb; [7440-03-1] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1991

CRITICAL EVALUATION:

Studies on the solubility of Nb in liquid Rb were performed at 3 different temperatures in the range of 1033 to 1363 K (1). The solute was the alloy of Nb with 1 mol % Zr which did not seem to have any effect on the apparent solubility of Nb. However, the activity of O in Rb as well as the solute alloy was diminished due to the stronger O gettering properties of Zr compared to Nb. The result of the solubility test indicated that the Nb content at 1033 K was below the detection limit of 1.3×10^{-3} mol %, that is equal or lower than 2.2×10^{-3} mol % at 1203 K and 2.5×10^{-3} mol % Nb at 1363 K. All these results were obtained at elevated pressure.
The low solubility of Nb in liquid Rb was qualitatively confirmed in a corrosion test (2). No corrosion of the test material after 700 hours exposure in circulating Rb at 1273 K could be observed in a Nb-Zr(1%) loop.
The evaluators concluded that an oxide mixture or double oxide of Rb and Nb should be the equilibrium solid phase, which is stable in contact to the saturated solution, if Rb contains a certain amount of O.
A schematic Nb-Rb phase diagram was presented in (3). It is similar to that shown for the Nb-Li system, differing in the melting (312.6 K) and boiling (961 K) points of Rb.

The tentative value of the solubility of Nb in liquid Rb:

T/K	*soly* /mass % Nb	source	remarks
1363	$3 \cdot 10^{-3}$	(1)	at the vapour pressure of Rb

References

1. Young, P.F.; Arabian, R.W. *US Atom.Ener.Comm. Rep. AGN-8063*, 1962; abstracted in Young, P.F.; Arabian, R.W. *NASA Rep. SP-41*, Pt.I, 1963, p. 167.
2. Parkman, M.F. Aerojet General Nucleonics, San Ramon, 1964; private communication to Gurinsky, D.H.; Weeks, J.R.; Klamut, C.J.; Rosenblum, L.; DeVan, J.H. *Peaceful Uses of Atomic Energy*, U.N.,N.Y. 1964, *9*, 550.
3. Smith, J.F.; Lee, K.J. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Mater., Materials Park, 1990, p.2755; *Bull.Alloy Phase Diagr.* 1990, *11*, 249.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Young, P.F.; Arabian, R.W.

US Atom.Ener.Comm. Rep. AGN-8063, 1962.

VARIABLES:

Temperature: 1033-1363 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubilities of Nb in liquid Rb were reported, they were regarded as the content in liquid Rb being in equilibrium with Nb-Zr(1%) alloy.

t/°F	*soly*/mass % Nb	*soly*/mol % Nb [a]
1400	$<1.4\cdot10^{-3}$, $<1.4\cdot10^{-3}$	$<1.3\cdot10^{-3}$
1700	$<1.4\cdot10^{-3}$, $2.4\cdot10^{-3}$ [b]	$<1.3\cdot10^{-3}$, $2.2\cdot10^{-3}$
2000	$2.7\cdot10^{-3}$, $2.7\cdot10^{-3}$, $3.0\cdot10^{-3}$ [c]	$2.5\cdot10^{-3}$, $2.7\cdot10^{-3}$

[a] - as calculated by the compilers
[b] - as given only in the figure
[c] - as reported also in the text

The results were also reported in (1). A corrosion of the Nb-Zr(1%) alloy was not detected, but the leaching of Zr from the alloy was observed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A specimen of a Nb-Zr alloy was degreased with aceton, pickled in a mixture of HNO_3, H_2SO_4, HF, H_2O (2:2:1:5), rinsed with H_2O, and alcohol and dried. An annealed Ta capsule (pickled in the same mixture) was filled with the alloy sample and Rb. The capsule was welded in an Ar atmosphere and flame sprayed with Al_2O_3. It was heated at the selected temperature for 50 hours and finally inverted, thus causing the Rb with dissolved Nb to flow into a Ta sample cup. The cup was cooled to room temperature. After solidification the capsule was analyzed for the contents of O and Nb. The sample was treated with anhydrous hexane, CH_3OH, H_2O and finally HCl. The combined solution was dried. The Ta sample cup was heated in bath of aqua regia for 1 h and the resulting solution was added to the RbCl solution to dry. The dry sample was spectroscopically analyzed in the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

Nb: from Nb-Zr alloy, supplied by Wah Chang Corp., containing 98.75 % Nb, 1.15 % Zr, $3.0\cdot10^{-3}$ % C, $9.4\cdot10^{-3}$ % N and $1.8\cdot10^{-2}$ % O.
Rb: supplied by MSA Research Corp., purified by passing through a micrometallic filter, gettered with Ti-Zr alloy at 866 K, distilled and finally filtered into the storage tank. O content after the purification was $(6\text{-}17)\cdot10^{-4}$ % and after test $(19\text{-}55)\cdot10^{-4}$ mass %.
Ar: nothing specified.

ESTIMATED ERROR:

Solubility: error of individual analysis ± 10%, detection limit $1.3\cdot10^{-3}$ mol % Nb.
Temperature: precision ± 3 K.

REFERENCES:

1. Young, P.F.; Arabian, R.W. *NASA Rep. SP-41*, Pt.I, 1963, p.167-176.

COMPONENTS: (1) Niobium; Nb; [7440-03-1] (2) Cesium; Cs; [7440-46-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland December 1988

CRITICAL EVALUATION:

Two different determinations of the solubility of Nb in liquid Cs have been performed at high temperatures (1,2), both using Nb-Zr(1%) alloy as the solute. In the first study (1) the result obtained at 1533 K was $1.8\cdot10^{-2}$ mol % Nb. In the second one (2) a lower value of $4.3\cdot10^{-3}$ mol % Nb was measured at 1644 K. The equilibration time was 100 h in both cases. The application of alumina as the sample crucible material, and its possible permeation by Cs was not a sufficient explanation for the overestimated result in (1). The values reported in (1,2) seemed to be based on the equilibria of pure Nb with its saturated solution in pure Cs, since Zr has a higher affinity for O than has Nb (3). The results of (2) are regarded as tentative, bearing in mind that they had been obtained under significantly elevated pressure.

The low solubility of Nb in liquid Cs was confirmed in corrosion tests by (4,5). It was also observed that the alloy Nb-Zr(1%) is more corrosion resistant than pure Nb (4). The dissolution of Nb in liquid Cs was studied at lower temperature. Godneva et al. (6) determined the solubility in the temperature range of 323 to 573 K. The presence of an additional amount of O in liquid Cs increased the amount of Cs being dissolved. It would not be reasonable to compare the results of (1,2) and (6), since there exists a considerable temperature gap between the two studies. The results of (6) seem to be overestimated. Tepper and Greer (7) observed that the solubility of Nb in Cs is relatively lower than that of Mo in the temperature range of 368 to 533 K. This observation is in partial agreement with the results of the earlier work (6).

A schematic Nb-Cs phase diagram of (8) is similar to that reported for the Nb-Li system.

Tentative value of the solubility of Nb in liquid Cs.

T/K	*soly*/mol % Nb	source	remark
1644	$4\cdot10^{-3}$	(2)	at the vapour pressure of Cs

References

1. Tepper, F.; Greer, J. *US Air Force Rep. ASD-TDR-63-824*, Pt. I, 1963.
2. Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-64-327*, 1964; *US Atom.Ener. Comm. Rep. AD-608385*, 1964; *US Atom.Ener. Comm. Rep. CONF-650411*, 1965, p. 323.
3. Barker, M.G. *Rev.Intern.Hautes Temp. Refract.* 1979, *16*, 237.
4. Chandler, W.T.; Hoffman, N.J. *US Air Force Rep. ASD-TDR-62-965*, 1963; *Metall.Soc.Conf.* 1966, *30*, 509.
5. Keddy, E.S. *US Atom.Ener. Comm. Rep. LAMS-2948*, 1963.
6. Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*,2177.
7. Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-66-280*, 1966.
8. Smith, J.F. *Bull.Alloy Phase Diagr.* 1988, *9*, 47.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Tepper, F.; Greer, J.

US Air Force Rep. ASD-TDR-63-824, Pt.I, 1963.

VARIABLES:

Temperature: 1533 and 1644 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid Cs at 2300°F was reported to be $1.25\cdot10^{-2}$ mass % or $1.8\cdot10^{-2}$ mol % Nb (as calculated by the compilers). The measured value was the Nb content in Cs which was equilibrated with Nb-Zr(1%) alloy.

The authors applied again the same technique except for the collector material, and they reported a result of $3.0\cdot10^{-3}$ mass % or $4.3\cdot10^{-3}$ mol % Nb at 2500°F (1). The equilibration time was 100 hours. The Nb content in Cs was lower at shorter times of exposition. The value which was obtained after 100 hours of exposure seems, therefore, to represent the true equilibration of the system.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The measurements of the solubility of Nb in Cs were performed by (means of applying) the inverted capsule technique. The capsule was heated under vacuum for 100 hours at test temperature filled with Cs and welded shut in an Ar atmosphere. After equilibration, the capsule was inverted and cooled. The solidified Cs in the collector part of the capsule was dissolved in CH3OH, diluted with H_2O and acidified with HCl. The joined solutions were evaporated to dryness and analyzed by means of spectrographical methods. An examination of the alumina (collector) part of the capsule revealed a complete permeation by a reaction with Cs. Therefore, the sampling (collector) part of the crucible used for the second determination was fabricated of a Mo-Ti(0.5%) alloy, the applied technique being essentially the same.

SOURCE AND PURITY OF MATERIALS:

Nb-Zr(1%) alloy: 99 % Nb, 1 % Zr, $6.9\cdot10^{-3}$ % C, $1.23\cdot10^{-3}$ % N, $1.92\cdot10^{-2}$ % O.
Cs: 99.9+ % purity, supplied by Mine Safety Appliances Research, containing $2.8\cdot10^{-3}$ % C, $1.2\cdot10^{-3}$ % O and $<2\cdot10^{-4}$ % N.
Ar: purified by a hot and cold K-Na bubbler; O and H_2O contents monitored.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 3 K.

REFERENCES:

1.Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-64-327*, 1964; *US Atom.Ener.Comm. Rep. AD-608385*, 1964; *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 323-333.

COMPONENTS:

(1) Niobium; Nb; [7440-03-1]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S.

Zh. Prikl. Khim. 1974, *74*, 2177-2180.

VARIABLES:

Temperature: 323-573 K
O concentration in Cs: 0.08 and 0.8 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Nb in liquid Cs at various temperatures and O concentration in Cs is reported:

t/°C	*soly*/mass % Nb	*soly*/mol % Nb [a]
50	$< 2 \cdot 10^{-4}$	$< 2.8 \cdot 10^{-4}$
100	$< 2 \cdot 10^{-4}$	$< 2.8 \cdot 10^{-4}$
150	$6 \cdot 10^{-4}$	$8.3 \cdot 10^{-4}$
200	$8 \cdot 10^{-4}$	$1.1 \cdot 10^{-4}$
300	$3 \cdot 10^{-4}$	$4.2 \cdot 10^{-4}$
300 [b]	$1.5 \cdot 10^{-3}$	$2.1 \cdot 10^{-3}$

[a] as calculated by the compilers
[b] Cs contained 0.8 mol % O

Probably a Nb-Cs mixed oxide was formed on the Nb surface, since an increase of mass of the Nb specimen was observed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A specimen of Nb metal was covered with Cs under vacuum and equilibrated for 120 hours in a glass ampoule. The O content in Cs was increased by means of a control decomposition of a $KClO_3$-MnO_2 mixture. The (ampoule) glass did not undergo any visible changes. The Cs sample was dissolved in H_2O and volumetrically determined (in the resultant hydroxide). An aliquot of the solution was treated with an excess of the corresponding acid. The remaining part was acidified with HCl and a H_2O-acetone (1:1) solution of "chrom-violet K" was added. The solution was heated for 5-7 min at 333-343 K, cooled and a few drops of ascorbic acid solution were added (1). The optical density of the resulting solution was measured to determine the Nb content..

SOURCE AND PURITY OF MATERIALS:

Nb: contained 0.08 % O.
Cs: 98-99 % pure metal, vacuum distilled containing < 0.01 % O and < 1.5 % Rb as main impurities.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Godneva, M.M.; Vodyannikova, R.D. *Zh. Anal. Khim.* 1965, *20*, 831-835.

COMPONENTS:	**EVALUATOR:**
(1) Tantalum; Ta; [7440-25-7] (2) Lithium; Li; [7493-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1991

CRITICAL EVALUATION:

Ta is one of the most dissolution-resistant metals in liquid Li. The measured Ta solubilities in liquid Li were orders of magnitude higher than those predicted by Kuzin et al. (1), who reported a smooth increase from $2.5 \cdot 10^{-11}$ to $2.0 \cdot 10^{-5}$ mol % Ta in the temperature range of 873 to 1428 K.

In preliminary experiments, Jesseman et al. (2) observed an increase of the Ta solubility in liquid Li from $7 \cdot 10^{-5}$ to $4 \cdot 10^{-4}$ mol % Ta with increasing temperatures from 1000 to 1277 K. They additionally confirmed that the Ta content in Li increased by one order of magnitude with the increasing equilibration period from 24 to 100 hours.

In a more recent corrosion test with a Ta specimen in liquid Li performed at 873 K, Klueh (3,4) observed that an increase of the O content in Li in the range of $4.3 \cdot 10^{-3}$ to $9.1 \cdot 10^{-2}$ mol % O did not influence the Ta content in Li, which had been lower than the analytical detection limit of $4 \cdot 10^{-5}$ mol % Ta. Stecura (5) performed similar experiments, observing an increase of the concentration of Ta in Li from $4 \cdot 10^{-4}$ to $8 \cdot 10^{-4}$ mol %, if the metals had been in contact at 1388 to 1663 K. The initial O content in Ta increased from 0.193 to 0.745 mol % O. This does not seem to have affected the Ta solubility in Li, since O was transferred from Ta into liquid Li, and a mixed Ta-Li oxide was not formed. The Li which was used for the experiments initially contained more Ta than after the equilibration.The results of (2), (3,4), and (5) thus were in fair agreement and can be fitted to the equation:

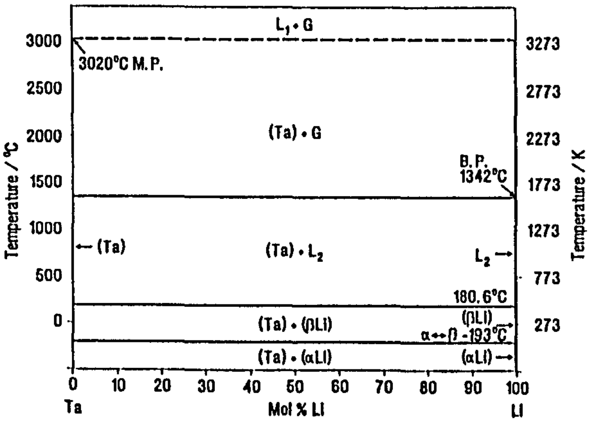
$$\log(soly/\text{mol \% Ta}) = -1.67 - 2400(T/\text{K})^{-1} \qquad r=0.992 \qquad \text{Eq.(1)}$$

One should bear in mind that for all these experiments the O content of the system was higher than 0.01 mol % O. Eichelberger et al. (6) investigated the solubility of Ta-rich alloys in liquid Li, their reported data being somewhat inconsistent. A W content of 8 mol % in both alloys did not effect the dissolution of Ta in Li; the influence of Hf as the stronger getter, however, was obvious. The results obtained in the temperature range 1280 to 1893 K were inconclusive: the scatter was more than one order of magnitude and not in agreement with the prediction line of (1); a distinct temperature dependence was not observed. The equilibration time of 4 hours was probably too short. An increase of the solubility values was due to the use of less pure Li, so these data obtained by (6) were closer to the theoretical solubility values of (1). The experimental pressure was the vapor pressure of Li in runs performed above 1617 K. Due to the above-mentioned reservations the data are not recommended.

In addition to the corrosion tests in (3-5) some other investigations should be cited which served to clarify the solubility experiments. Hahn and Jaworsky (7) found Ta to be less resistant against Li attack than Nb, Cr, Mo, and W. Busse (8) observed that O was transported from Ta to liquid Li at lower temperatures, while a high O concentration in Li may cause a dissolution of Ta at higher temperatures. Barker (9) confirmed that a mixed Ta-Li oxide was not formed in the system. However, an increase of the N concentration might cause formation of $TaN_{0.04}$ or Ta_2N. Addison (10) reported formation of Li_7TaN_4 as a corrosion product, DeVan et al. (11) observed a carbo-nitride phase precipitation at the Ta-Li boundary. The Ta-Li phase diagram is redrawn from (12) and is presented in the figure.

Tentative solubility values of Ta in liquid Li containing ~ 10^{-3} mol % N

T/K	$soly$/mol % Ta	source	remarks
873	$\leq 4 \cdot 10^{-5}$	(4)	
973	$6 \cdot 10^{-5}$	(2)	
1073	$1.2 \cdot 10^{-4}$	Eq.(1)	
1173	$2 \cdot 10^{-4}$	Eq.(1)	
1273	$3 \cdot 10^{-4}$	(2)	
1373	$4 \cdot 10^{-4}$	(5)	
1473	$5 \cdot 10^{-4}$	Eq.(1)	
1573	$6 \cdot 10^{-4}$	Eq.(1)	
1673	$8 \cdot 10^{-4}$	(5)	at the vapour pressure of Li at equilibration



COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Lithium; Li; [7493-93-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
January 1991

CRITICAL EVALUATION: (continued)

References

1. Kuzin, A.N; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroenia Diagram Sostoyania*, Nauka, Moskva, 1985, p. 113.
2. Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P. *US Atom.Ener.Comm. Rep. NEPA-1465*, 1950.
3. Klueh, R.L. *Trans. Am. Nucl. Soc.* 1972, *15*, 746.
4. Klueh, R.L. *Metall. Trans.* 1974, *5*, 875.
5. Stecura, S. *Corrosion Sci.* 1976, *16*, 233.
6. Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969; *Rep. AI-68-110*, 1969.
7. Hahn, H.; Jaworsky, M. *Metall. Soc. Conf.* 1966, *30*, 547.
8. Busse, C.A. *Corrosion Sci.* 1970, *10*, 65.
9. Barker, M.G. *Rev. Intern. Hautes Temp. Refract.* 1979, *16*, 237.
10. Addison, C.C. *The Chemistry of the Liquid Alkali Metals*, Wiley, Chichester, 1984, p. 66.
11. DeVan, J.H.; Litman, A.P.; DiStefano, J.R.; Sessions, C.E. *Alkali Metal Coolants*, IAEA, Vienna, 1967, p. 675.
12. Garg, S.P.; Venkatraman, M.; Krishnamurphy, N. *J.Alloy Phase Diagr.* 1990, *6*, 8.

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P.
US Atom.Ener.Comm. Rep. NEPA-1465, 1950.

VARIABLES:

Temperature: 1000-1277 K.

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ta solubilities in liquid Li at various temperatures and equilibration times are reported.

t/°C	*time*/h	*soly*/mass % Ta	*soly*/mol % Ta [a]
732	24	$1.8 \cdot 10^{-3}$, $2.0 \cdot 10^{-3}$	$6.9.10^{-5}$, $7.6.10^{-5}$
1004	24	$1.1 \cdot 10^{-2}$, $8.0 \cdot 10^{-3}$	$4.2.10^{-4}$, $3.0.10^{-4}$
1004	100	0.17, 0.20	$6.5 \cdot 10^{-3}$, $7.6 \cdot 10^{-3}$

[a] calculated by the compilers

An equilibration time of 24 hours seems to be insufficient.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Strips of a Ta sheet were placed in an Armco Fe capsule. The capsule was loaded with Li in an Ar dry box, degassed and Li was melted in a pot furnace. After welding the capsule was heated in a vacuum furnace with stainless steel plates, the average temperatures of which had been assigned. The temperature was maintained for a given period and the furnace was then air-cooled while still being kept under low pressure. The capsule was weighed and opened. The solidified sample was leached out of the capsule with distilled water, and the Ta remaining undissolved was removed with the capsule, dried, and weighed as the tare to determine the amount of Li solution in the capsule.

The leached material was filtered, and the residue analysed for the Ta content by means of a spectrographical method.

SOURCE AND PURITY OF MATERIALS:

Ta: unspecified.
Li: containing 0.24 % O, <0.02 % N, <0.005 % Na.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: precision ± 15 %.
Temperature: accuracy ± 20 K.

REFERENCES:

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G.

NASA Rep. CR-1371, 1969; *Rep. AI-68-110*, 1969.

VARIABLES:

Temperature: 1280-1893 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ta concentrations in liquid Li saturated with Ta rich alloys is reported.

t/°C	*soly*/mass % Ta	*soly*/mol % Ta [a]	solute
1197	$3\cdot10^{-4}$	$1.2\cdot10^{-5}$	T-111 alloy
1202	$<1\cdot10^{-4}$, $2\cdot10^{-4}$	$<4\cdot10^{-6}$, $8\cdot10^{-6}$	T-111 alloy
1203	$<1\cdot10^{-4}$, $2\cdot10^{-4}$	$<4\cdot10^{-6}$, $8\cdot10^{-6}$	T-111 alloy
	$3\cdot10^{-4}$, $3\cdot10^{-4}$	$1.2\cdot10^{-5}$, $1.2\cdot10^{-5}$	
1308	$1.1\cdot10^{-3}$	$4.2\cdot10^{-5}$	T-111 alloy
1395	$2\cdot10^{-4}$	$8\cdot10^{-6}$	T-111 alloy
1600	$2\cdot10^{-4}$	$8\cdot10^{-6}$	T-111 alloy
1363[b]	$2.0\cdot10^{-3}$	$8.0\cdot10^{-5}$	T-111 alloy
1603[b]	$1.1\cdot10^{-3}$	$4.2\cdot10^{-5}$	T-111 alloy
1007	$5\cdot10^{-4}$	$1.9\cdot10^{-5}$	ASTAR-811C alloy
1233	$3\cdot10^{-4}$	$1.2\cdot10^{-5}$	ASTAR-811C alloy
1400	$2\cdot10^{-4}$, $2\cdot10^{-4}$	$8\cdot10^{-6}$, $8\cdot10^{-6}$	ASTAR-811C alloy
1620	$<1\cdot10^{-4}$	$4\cdot10^{-6}$	ASTAR-811C alloy
1195[b]	$1.8\cdot10^{-3}$	$6.8\cdot10^{-5}$	ASTAR-811C alloy
1391[b]	$2.3\cdot10^{-3}$	$8.7\cdot10^{-5}$	ASTAR-811C alloy

[a] calculated by the compilers.

[b] less pure Li and unimproved analysis technique were applied.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Ta crucible and the Mo-Ti(½%) alloy sample collector were chemically etched and placed in a Mo capsule. All parts were degassed under high vacuum at 1973 K, cooled in an Ar atmosphere and finally outgassed at 623 K to remove Ar. After filling Li into the crucible the collector and [with] its cup were installed and the capsule was sealed under vacuum. The capsule was heated to the desired test temperature and kept in the cup-up position for 4 hours. Finally, the capsule was removed from the furnace, the cup being then at the bottom. This way the liquid Li in the capsule was transferred to the collector. After cooling the capsule was cracked open, the collector separated, and a Li sample was melted from the collector into a special glass ware. The solidified Li was submerged in H_2O in an Ar atmosphere. After completion of the reaction the resulting solution was neutralized with HCl. The collector was rinsed with a HF solution and the resulting solution was added to the previous one. Ta was extracted with hexone in presence of EDTA and $AlCl_3$ in aqueous solution. Finally Ta was reextracted with hexone to an aqueous buffer solution of phenylfluorone and the absorbance of the formed complex was measured by means of spectrophotometric analysis.

SOURCE AND PURITY OF MATERIALS:

T-111 alloy: supplied by J.T. Ryerson & Son Inc., containing 90 % Ta, 7.87 % W, 2.03 % Hf, $2.5\cdot10^{-3}$ % O, $1.0\cdot10^{-3}$ % N, $<1.0\cdot10^{-3}$ % C.

ASTAR-811C alloy: supplied by NASA Lewis Res. Center, containing 90 % Ta, 8 % W, 1 % Hf, Re, $2.5\cdot10^{-2}$ % C.

Li: supplied by General Electric Corp., hot trapped with a Zr foil getter for 126 hours at 1093 K and distilled, containing $4.4\cdot10^{-3}$ % C, $1.3\cdot10^{-3}$ % N, $3.3\cdot10^{-3}$ % O, $<5.0\cdot10^{-3}$ % B, Ba, $<2.5\cdot10^{-3}$ % Na, Nb, Pb, Sn, Ti, V, Zr, $<5\cdot10^{-4}$ % Ag, Al, Ca, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Sr, each.

Ar: nothing specified.

ESTIMATED ERROR:

Solubility: precision ± $2\cdot10^{-5}$ mol % Ta.

Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Klueh, R.L.

Metall. Trans. 1974, *5*, 875-879.

VARIABLES:

One temperature: 873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ta in liquid Li at 600 °C is below the analytical detection limit of $1.0 \cdot 10^{-3}$ mass % or $4 \cdot 10^{-5}$ mol % Ta (as calculated by the compilers).
The O concentration in Li in the range of 0.01-0.21 mass % (0.0043 to 0.091 mol % O, as calculated by the compilers) does not effect the undeterminable Ta solubility in Li. The same observation was previously reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The applied system consisted of a Ta specimen (in contact with liquid Li) in a container of the same material, which in turn was encapsulated in stainless steel. The capsule was filled with liquid Li and welded in an Ar-atmosphere chamber (to prevent a contamination of O or Ta). The concentration of O was varied by adding Li_2O (in order to determine the effect of O on Li). The system was equilibrated at 873 K for 20 hours. After the test the capsule was quenched in liquid N and opened in an Ar-atmosphere chamber. Li was removed by dissolving in chilled isopropyl alcohol and recovered from it as LiF. The Ta amount in Li was determined by spectrographic analysis.

SOURCE AND PURITY OF MATERIALS:

Ta: 99.9 % pure, containing 7.0.10-3 % O.
Li: purified by Zr gettering at 1073 K; containing $1.0 \cdot 10^{-2}$ % O.
Ar: nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

(1) Klueh, R.L. *Trans. Am. Nucl. Soc.* 1972, *15*, 746-747.

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Stecura, S.

Corrosion Sci. 1976, *16*, 233-241.

VARIABLES:

Temperature: 1388-1663 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The author reported the Ta contents in liquid Li after a 24 hours contact of the metals. As this time seems to be sufficient to reach an equilibrium in the system, it might be assumed that the Ta content in Li is equal to its solubility. However, the Ta concentration in Li before the equilibration ($2\cdot10^{-3}$ % Ta) requires some reservation in respect to the obtained results.

t/°C	*soly*/mol % Ta	*O content* in Ta/mol %
1115	$4\cdot10^{-4}$	0.193
1260	$4\cdot10^{-4}$	0.214
1260	$5\cdot10^{-4}$	0.685
1390	$8\cdot10^{-4}$	0.745

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The applied Mo capsule served as a container for the Ta crucible as well as a collector for Li at the end of a test run. The capsule and the crucible were cleaned and annealed in high vacuum for 2 hours at 1698 and 1473 K, respectively. The annealed Ta crucible was doped with O and annealed again under the same conditions. After filling Li into the crucible and installing the crucible inside the capsule, the capsule was electron-beam welded in vacuum and further heated in a vacuum furnace. The temperature inside the furnace was measured by means of a W/W-Re(26%) thermocouple properly installed in the uniform temperature zone at one inch intervals. After conditioning the system for 24 hours the capsule was inverted, cooled to room temperature, removed to an Ar filled dry box, and cut open. The bulk Li was analysed for the dissolved Ta content.

SOURCE AND PURITY OF MATERIALS:

Ta: "polycrystalline", containing $5\cdot10^{-4}$ % C, $1.2\cdot10^{-3}$ % O, $2.2\cdot10^{-3}$ % N, $5\cdot10^{-4}$ % H, $<1\cdot10^{-4}$ % Li, subsequently doped with O.

Li: "high purity", containing $2.5\cdot10^{-2}$ % O, $8\cdot10^{-4}$ % N, $5\cdot10^{-2}$ % Ta.

Ar: nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.

Temperature: precision ± 10 K.

REFERENCES:

COMPONENTS:	**EVALUATOR:**
(1) Tantalum; Ta; [7440-25-7] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1991

CRITICAL EVALUATION:

Three different groups of investigators determined the Ta solubility in liquid Na at moderately high temperatures. In an early study, Baus et al. (1) reported a solubility of $2\cdot10^{-7}$ mol % Ta at 698 K. The temperature range was extended to 598-798 K in a more recent study (2) and an increase of the solubility from $4\cdot10^{-7}$ to $4\cdot10^{-5}$ mol % Ta could be noticed. An interference of impurities with the radioactive counting was possible, but seemed not to be critical.

The subsequent work on this subject by Humphreys (3) was published in a secondary source and is therefore not compiled. The Ta solubility was measured in Zr-gettered Na contained in a Ta capsule. A neutron-activated Ta foil was placed in the capsule at the test temperature for at least 180 hours. A radioactive counting technique yielded the solubilities of $7\cdot10^{-7}$ and $1.5\cdot10^{-6}$ mol % Ta at 923 K and $2\cdot10^{-6}$ and $9\cdot10^{-6}$ mol % Ta at 1061 K. It was concluded that the systematic errors exceeded the true solubilities; thus, the data were no more precise than one order of magnitude.

Klueh (4) performed experiments at 873 K by equilibrating a Ta specimen with Na containing O in the concentration range $7\cdot10^{-3}$ to 1.7 mol %. A spectacular increase of the Ta content in liquid Na was determined. The evaluators extrapolated the apparent solubility data to the O content in Na of $1\cdot10^{-4}$ mol % O, neglecting the solubility values at $7\cdot10^{-3}$ mol % O in Na, and determined a corresponding Ta solubility in the order of 10^{-7} mol % Ta. This value was in fair agreement with the data obtained by Humphreys (3) at both temperatures. The results of Baus et al. (1) and Grand et al. (2) were one or two orders of magnitude higher. The suggested solubility values based on (3) and extrapolated results of (4) can be classified as doubtful. Undetectable amounts of Ta (by spectral analysis) were dissolved in liquid Na at 973-1023 K after 1 hour of equilibrating the metals (7).

A comparison of the apparent solubilities with theoretical predictions of Kuzin et al. (6) ($9\cdot10^{-12}$ and $1\cdot10^{-9}$ mol % Ta at 973 and 1111 K, respectively) indicates that the observed increase of the Ta concentration was induced by O present in Na. Klueh (4) identified $NaTaO_3$ as the corrosion product of the system, while Barker (5) and Mathews (8) observed formation of Na_3TaO_4 at very high O levels in Na. Thus, in practice the applied solute is solid Ta saturated with O or the mentioned tantalate rather than metallic Ta.

The Ta-Na phase diagram is published by (9); it is similar to that of the Ta-Li system.

Doubtful solubility values of Ta in liquid Na

T/K	*soly*/mol % Ta	source
873	$1\cdot10^{-7}$	(4) extrapolated
1073	$1\cdot10^{-5}$	(3)

References

1. Baus, R.A.; Bogard, A.D.; Grand, J.A.; Lockhart, L.B.; Miller, R.R.; Williams, D.D. *Peaceful Uses of Atomic Energy*, U.N., New York, 1956, *9*, 356.
2. Grand, J.A.; Baus, R.A.; Bogard, A.D.; Williams, D.D.; Lockhart, L.B.; Miller, R.R. *J.Phys. Chem.* 1959, *63*, 1192.
3. Humphreys, J.R.; personal communication to Claar, T.D. *Reactor Technol.* 1970, *13*, 124.
4. Klueh, R.L. *Metall. Trans.* 1972, *3*, 2145.
5. Barker, M.G. *Rev. Intern. Hautes Temp. Refract.* 1979, *16*, 237.
6. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroenia Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113.
7. Aleksandrov, B.N.; Dalakova, N.V. *Izv.Akad.Nauk SSSR, Met.* 1982, no. 1, 133.
8. Mathews, C.K. *High Temp. Sci.* 1988-1989, *26*, 377.
9. Garg, S.P.; Venkatraman, M.; Krishnamurphy, N. *J.Alloy Phase Diagr.* 1990, *6*, 10.

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Baus, R.A.; Bogard, A.D.; Grand, J.A.; Lockhart, L.B.; Miller, R.R.; Williams, D.D.
Peaceful Uses of Atomic Energy, U.N., N.Y., 1956, *9*, 356.

VARIABLES:

One temperature: 698 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ta solubility in liquid Na at 425 °C was reported to be $1.6\cdot10^{-8}$ g Ta per g Na ($2.0\cdot10^{-7}$ mol % Ta as calculated by the compilers). The Ta content in Na in equilibrium with a stainless steel source (containing about 0.08 mass % Ta) was merely $1\cdot10^{-11}$ g Ta per g Na at 425 °C ($1.3\cdot10^{-10}$ mol % Ta as calculated by the compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ta test crucible containing a radioactive isotope was filled with freshly distilled Na. The two metals were equilibrated at the test temperature for 72 hours. Na was permanently stirred. The Na samples were removed to determined the content of ^{182}Ta by radiochemical analysis and calculate the Ta solubility. All operations were performed in an inert atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ta: nothing specified.
Na: vacuum distilled, filtered into a Ta vessel, containing $\leq 1\cdot10^{-3}$ % O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Grand, J.A.; Baus, R.A.; Bogard, A.D.; Williams, D.D.; Lockhart, L.B.; Miller, R.R.
J.Phys. Chem. 1959, *63*, 1192.

VARIABLES:

Temperature: 598-798 K.

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ta in liquid Na is reported.

t/°C	*soly*/mass % Ta	*soly*/mol % Ta [a]
325	$3.2 \cdot 10^{-6}$	$4.1 \cdot 10^{-7}$
425	$1.9 \cdot 10^{-5}$	$2.4 \cdot 10^{-6}$
525	$2.9 \cdot 10^{-4}$	$3.7 \cdot 10^{-5}$

[a] calculated by the compilers.

The authors emphasized a possible formation of a double Ta-Na oxide in the system Na-Ta-O.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ta vessel containing a ^{182}Ta radioisotope was equilibrated with liquid Na for 24 hours. The vessel was inserted in a stainless steel container. The Na solution was withdrawn for analysis by means of a quartz pipet. Na samples of each equilibration were individually sealed in quartz. The samples were subsequently determined by radiochemical analysis to calculate the Ta solubility. All experiments were performed in Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ta: nothing specified.
Na: distilled and filtered into the equilibration apparatus, estimated O content 0.001-0.002 %.

ESTIMATED ERROR:

Nothing numerically specified.
As Na was contaminated by the components of the stainless steel, the results have to be regarded as doubtful.

REFERENCES:

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Klueh, R.L.

Metall. Trans. 1972, *3*, 2145-2150.

VARIABLES:

One temperature: 873 K
O concentration in Na: $7\cdot10^{-3}$ - 1.71 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent Ta solubility in liquid Na as a function of the O concentration in Na at 600 °C is reported.

O concn/mass %	*O concn*/mol % [a]	*soly*/mass % Ta	*soly*/mol % Ta [a]
$5\cdot10^{-3}$	$7\cdot10^{-3}$	$6.3\cdot10^{-3}$	$8.0\cdot10^{-4}$
$5\cdot10^{-3}$	$7\cdot10^{-3}$	$4.0\cdot10^{-3}$	$5.1\cdot10^{-4}$
$2.5\cdot10^{-2}$	$3.6\cdot10^{-2}$	$1.1\cdot10^{-2}$	$1.4\cdot10^{-3}$
$3\cdot10^{-2}$	$4.3\cdot10^{-2}$	$2.0\cdot10^{-3}$	$2.5\cdot10^{-4}$
$5.5\cdot10^{-2}$	$7.9\cdot10^{-2}$	$1.6\cdot10^{-3}$	$2.0\cdot10^{-3}$
$7\cdot10^{-2}$	0.10	$8\cdot10^{-3}$	$1.0\cdot10^{-3}$
0.10	0.14	$2.7\cdot10^{-2}$	$3.4\cdot10^{-3}$
0.10	0.14	$2.0\cdot10^{-2}$	$2.5\cdot10^{-3}$
0.20	0.29	$6.3\cdot10^{-3}$	$8.0\cdot10^{-3}$
0.20	0.29	$2.3\cdot10^{-2}$	$2.9\cdot10^{-3}$
0.40	0.57	0.32	$4.1\cdot10^{-2}$
0.75	1.07	1.32	0.166
1.20	1.71	1.87	0.242

[a] calculated by the compilers.

The presence of a $NaTaO_3$ precipitate on Ta could be observed after the test.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The applied system consisted of a Ta specimen in contact with liquid Na in a Ta container, which in turn was encapsulated in stainless steel. The capsule was filled and welded in Ar atmosphere to prevent a contamination of O or Ta. The O concentration in Na was varied by adding NaO_2 in order to determine the effect of O in Na. While being heated to the test temperature the specimen was in the vapour zone of the capsule. When the system reached the desired temperature, the capsule was inverted and kept in this position for 500 hours. After the test the capsule was again inverted and quenched in liquid N. Na was removed by dissolving in chilled isopropyl alcohol and recovered from it as NaF. The Ta amount in Na was determined by spectrographic analysis of NaF.

SOURCE AND PURITY OF MATERIALS:

Ta: 99.9 % purity.
Na: gettered by Zr.
Ar: nothing specified.

ESTIMATED ERROR:

Nothing specified

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Tantalum; Ta; [7440-25-7] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1991

CRITICAL EVALUATION:

The Ta solubility in liquid K for absolutely pure elements is unmeasureably low at temperatures below the boilling point of the solvent (1032 K). A comparatively marked dissolution of Ta in liquid K is to be observed in practice, if even merely traces of O are present in the system.

Early experiments were performed by Ginell and Teitel in the temperature range of 1366-1673 K. The solubility results were published in reports (1-4) and did not indicate definite temperature dependences. The data were spread in the range $8.6 \cdot 10^{-5}$ to $3.3 \cdot 10^{-3}$ mol % Ta.

The results of McKisson et al. (5) were comparatively consistent in themselves, increasing from $9 \cdot 10^{-3}$ to $6 \cdot 10^{-2}$ mol % Ta with temperatures increasing from 1073 to 1473 K. The materials had been diligently prepared to obtain a high degree of purity. However, these results were at an almost two orders of magnitude higher than those published by (1-4). The data of (5) fitted the equation:

$$\log (soly/\text{mol \% Ta}) = 0.53 - 2900\,(T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

as confirmed by the compilers. In a subsequent report of the same group of workers (6) the solubility of 3 Ta-rich alloys was investigated. The Ta concentrations in K of the Ta-W(8%)-Hf(2%) and Ta-W(8%)-Re(1%)-Hf(1%) alloys were established to be approximately $2 \cdot 10^{-4}$ mol % Ta (being practically temperature independent), whereas the Ta concentrations of the Ta-Zr(½%) alloy in saturated solutions were scattered between $8 \cdot 10^{-4}$ and 0.1 mol % Ta. It was noticed that alloys containing Hf, which is a stronger getter than Zr, are less soluble in K, thus indicating a direct influence of O on the measured solubility. Paper (6) is not compiled.

Hickam (7,8) equilibrated Ta and liquid K in a closed system at 1255 K. The solution did not contain Ta in a concentration above the detection limit of $2 \cdot 10^{-4}$ mol % Ta. A similar qualitative observation on the Ta-K system at 923 K was reported in (15). These results supported the data of (1-4).

Klueh (9,10) investigated the influence of O on the Ta solubility in liquid K in detail. His results were convincing and an extrapolation of the data to an O level of $2 \cdot 10^{-4}$ mol % O (by the evaluators) provided an estimated solubility of $1 \cdot 10^{-5}$, $2 \cdot 10^{-4}$, and $1 \cdot 10^{-3}$ mol % Ta at 873, 1073, and 1273, respectively. The extrapolated values were within the scatter band of (1-4), (7,8), and (5), (11).

Stecura (11) also investigated the Ta solubility as well as the O influence in Ta on it. The reported solubility results for undoped Ta increased almost regularly from $1.5 \cdot 10^{-3}$ to $5.0 \cdot 10^{-3}$ mol % Ta at 1055 to 1287K. The solubility equation confirmed by the evaluators is given in the form:

$$\log(soly/\text{mol \% Ta}) = (0.09 + 0.24) - (3042 \pm 276)(T/\text{K})^{-1} \qquad \text{Eq.(2)}$$

and is comparable to Eq.(1) of (5).

The above solubility data, as well as further corrosion tests (12,13), indicate a strong effect of O dissolved in liquid K as well as in solid Ta (to a significantly lower extent). The product of the interaction of O with Ta and K was identified as K_3TaO_4 (7-12). This substance is regarded to be mainly responsible for the Ta concentration in liquid K.

Either Eq.(1) or Eq.(2) may be used for the selection of the solubility at an O level of about $1 \cdot 10^{-3}$ mol %. A decrease of the O concentration in K or the O activity by a present gettering element (Hf) in Ta may cause a significant reduction of the solubility values.

The predicted Ta-K phase diagram was constructed by Garg et al. (14), it is similar to that shown for the Ta-Li system.

Tentative solubility values of Ta in liquid K at O concentration of $1 \cdot 10^{-3}$ mol %

T/K	$soly$/mol % Ta	source	remarks
1073	$2 \cdot 10^{-3}$	(11)	at K vapour pressure
1173	$3 \cdot 10^{-3}$	(11) interpolated	at K vapour pressure
1273	$5 \cdot 10^{-3}$	(11)	at K vapour pressure
1373	$7 \cdot 10^{-3}$	(11) extrapolated	at K vapour pressure

References

1. Ginell, W.S.; Teitel, R.J. *US Atom.Ener.Comm.Rep. CONF-650411*, 1965.
2. Teitel, R.J. *Trans. Am. Nucl. Soc.* 1965, *8*, 15.
3. Ginell, W.S.; Teitel, R.J. *Trans. Am. Nucl. Soc.* 1965, *8*, 393.
4. Ginell, W.S.; Teitel, R.J. *Douglas Aircraft Rep. SM-48883*, 1965.
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6. Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969; *Atomics Internat.Rep. AI-68-110*, 1969.
7. Hickam, W.G. *NASA Rep. TN-D-4213*, 1967.
8. Hickam, W.G. *J. Less-Common Met.* 1968, *14*, 315.
9. Klueh, R.L. *US Atom.Ener.Comm.Rep. ORNL-4350*, 1969, p. 120.
10. Klueh, R.L. *Corrosion* 1972, *28*, 360.
11. Stecura, S. *NASA Rep. TN-D-5875*, 1970.
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14. Garg, S.P.; Venkatraman, M.; Krishnamurphy, N. *J.Alloy Phase Diagr.* 1990, *6*, 12.
15. Aleksandrov, B.N.; Dalakova, N.V. *Izv.Akad.Nauk SSSR, Met.* 1982, no.1, 133.

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Ginell, W.S.; Teitel, R.J.

Trans. Am. Nucl. Soc. 1965, *8*, 393-394.

VARIABLES:

Temperature: 1366 - 1603 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ta in liquid K at various temperatures is reported.

T/°C	*soly*/mass % Ta	*soly*/mol % Ta ^{e}	*equilibration time*/h
$1093^{a,d}$	$4 \cdot 10^{-4}$	$8.6 \cdot 10^{-5}$	1
$1093^{a,d}$	$1.6 \cdot 10^{-3}$	$3.4 \cdot 10^{-4}$	1
1130^{c}	$1.7 \cdot 10^{-3}$	$3.7 \cdot 10^{-4}$	1
1225	$1.5 \cdot 10^{-3}$	$3.2 \cdot 10^{-4}$	1
$1245^{c,d}$	$1.51 \cdot 10^{-2}$	$3.3 \cdot 10^{-3}$	3
$1245^{c,d}$	$1.26 \cdot 10^{-2}$	$2.7 \cdot 10^{-3}$	3
1330^{c}	$1.5 \cdot 10^{-3}$	$3.2 \cdot 10^{-4}$	1
1330^{c}	$6 \cdot 10^{-4}$	$1.3 \cdot 10^{-4}$	1
1330^{c}	$1.14 \cdot 10^{-2}$	$2.5 \cdot 10^{-3}$	1

^{a} exclusively reported in (1) ^{b} exclusively reported in (3) ^{c} also reported in (3) ^{d} O concentration in K of $2.3 \cdot 10^{-3}$ mass % ^{e} calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An L-shaped test capsule fabricated of a Nb-1 Zr alloy was placed in a centrifuge. A small dam was installed inside the capsule to regulate the amount of K which might drain into the collector part of the capsule. The capsule material was chemically etched inside and vacuum treated at the maximum temperature of the experiment. The capsule containing a Ta sample was filled with K, welded and heated at 100 K above the equilibration temperature for 1 hour, while the centrifuge was rotated to prevent K from flowing over the dam. The test temperature was then kept for 1-3 hours and the rotation rate was increased to force the precipitation of the solute to the bottom of the sample crucible. When the rotation rate was slowly reduced, the solution was decanted and drained into the collector. The method of further chemical analysis was not specified. All operations were performed in an Ar-atmosphere chamber.

SOURCE AND PURITY OF MATERIALS:

Ta: 99.9 % purity.
K: purified by hot trap with Ti-Zr alloy chips at 1058 K; containing $(7\text{-}11) \cdot 10^{-4}$ % O.
Ar: "high purity", dried and passed over Ti-Zr alloy chips at 1173 K; containing $\leq 2 \cdot 10^{-4}$ % H_2O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Teitel, R.J. *Trans. Am. Nucl. Soc.* 1965, *8*, 15.
2. Ginell, W.S.; Teitel, R.J.; *Douglas Aircraft Rep. SM-48883*, 1965.
3. Ginell, W.S.; Teitel, R.J. *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 44-47.

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

McKisson, R.L.; Eichelberger, R.L:; Dahleen, R.C.; Scarborough, J.M.; Argue, G.R.
NASA Rep. CR-610, 1966; *Rep. AI-65-210*, 1966.

VARIABLES:

Temperature: 1073-1473 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ta solubilities in liquid K are reported.

t/°C	*O content in K*/mass %	*soly*/mass % Ta	*soly*/mol % Ta [a]
800	$3.5\cdot10^{-4}$	$4.13\cdot10^{-2}$	$8.9\cdot10^{-3}$
900	$3.5\cdot10^{-4}$	$5.72\cdot10^{-2}$	$1.23\cdot10^{-2}$
1000	$3.5\cdot10^{-4}$	$2.73\cdot10^{-1}$	$5.9\cdot10^{-2}$
1200	$3.5\cdot10^{-4}$	$1.84\cdot10^{-1}$	$3.98\cdot10^{-2}$
1000	$1.4\cdot10^{-3}$	$5.52\cdot10^{-2}$	$1.19\cdot10^{-2}$
1000	$1.4\cdot10^{-3}$	$3.71\cdot10^{-2}$	$8.0\cdot10^{-3}$
1000	$1.4\cdot10^{-3}$	$8.02\cdot10^{-2}$	$1.73\cdot10^{-2}$
1000	$1.4\cdot10^{-3}$	$8.17\cdot10^{-2}$	$1.76\cdot10^{-2}$
1100	$1.4\cdot10^{-3}$	$6.88\cdot10^{-2}$	$1.49\cdot10^{-2}$
1100	$1.4\cdot10^{-3}$	$2.29\cdot10^{-1}$	$4.95\cdot10^{-2}$
1200	$1.4\cdot10^{-3}$	$2.88\cdot10^{-1}$	$6.23\cdot10^{-2}$
1200	$1.4\cdot10^{-3}$	$1.52\cdot10^{-1}$	$3.29\cdot10^{-2}$

[a] calculated by the compilers.

The results might be fitted to the smoothing equation

$$\log(soly/\text{mol \% Ta}) = 0.53 - 2900(T/\text{K})^{-1}$$

as confirmed by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ta test crucible and a Mo collector were placed in a Mo capsule. Each part of the apparatus was cleaned and rinsed with acetone. The apparatus was heated to temperatures up to 2073 K, degassed under high vacuum, and cooled in an Ar atmosphere. After filling K into the crucible the collector with its cup were installed, and the capsule was sealed under high vacuum by electron-beam welding. The capsule was heated to the desired test temperature and kept in the cup-up position for 2-8 hours. Finally, the capsule was removed from the furnace, the cup being then at the bottom. This way the liquid K in the capsule was transferred to the collector. After cooling the capsule was cracked open, the collector separated, and a K sample was melted from the collector into a special glass ware. It reacted with H_2O vapour in a He atmosphere acidified with HCl and HF. The K remaining in the collector was dissolved in H_2O and rinsed with HCl. The Ta content in the resulting solution was determined by means of a spectrophotometric method as a phenylfluorene complex which was extracted from the aqueous solution by isobutyl ketone.

SOURCE AND PURITY OF MATERIALS:

Ta: triple pass electron beam zone refined, supplied by Mater. Res. Corp., containing $2.5\cdot10^{-4}$ % O, $2.5\cdot10^{-4}$ % N, $3\cdot10^{-5}$ % H, $6\cdot10^{-4}$ % C.
K: as in the Nb-K system, same report.
Ar: unspecified.
He: unspecified.

ESTIMATED ERROR:

Solubility: precision ± 50 % (compilers).
Temperature: precisely regulated stability, nothing numerically specified.

REFERENCES:

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Stecura, S.

NASA Rep. TN-D-5875, 1970.

VARIABLES:

Temperature: 1055-1287 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ta solubility in liquid K at various temperatures is reported.

T/K	*soly*/mass % Ta	*soly*/mol % Ta [a]
1287	$2.40 \cdot 10^{-2}$	$5.2 \cdot 10^{-3}$
1271	$2.22 \cdot 10^{-2}$; $2.11 \cdot 10^{-2}$	$4.7 \cdot 10^{-3}$
1182	$1.71 \cdot 10^{-2}$; $1.67 \cdot 10^{-2}$	$3.7 \cdot 10^{-3}$
1162	$1.22 \cdot 10^{-2}$; $1.12 \cdot 10^{-2}$	$2.5 \cdot 10^{-3}$
1069	$9.3 \cdot 10^{-3}$; $8.8 \cdot 10^{-3}$	$2.0 \cdot 10^{-3}$
1055	$6.9 \cdot 10^{-3}$; $5.9 \cdot 10^{-3}$	$1.4 \cdot 10^{-3}$

[a] mean values calculated by the compilers.

The results were fitted to the equation:

$$\log(soly/\text{mol \% Ta}) = (0.09 \pm 0.24) - (3048 \pm 276)(T/\text{K})^{-1}$$

the exactness of which was confirmed by the compilers.
The observed Ta content increased if Ta samples doped with 0.94-1.8 mol % O had been applied. However, the data were time dependent even at 96 hours of testing and are therefore not reproduced.
K_3TaO_4 was recovered from liquid K and identified by chemical analysis and x-ray diffraction patterns.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ta crucible and a Ni capsule were ultrasonically cleaned in CHF_3, a detergent, and H_2O. The crucible was etched in a H_2SO_4, HNO_3, HF (11:5:4) mixture for 2-5 s and the Ni was treated in HNO_3, H_3PO_4, H_2SO_4, CH_3COOH (3:1:1:5) for ½-1 min at 363 K. Both parts were rinsed in H_2O and dried under vacuum. The crucible was installed in the capsule, the upper part of which served as a receiver. The crucible was filled with K in a vacuum chamber and the capsule was electron-beam welded. The assembly was placed in a furnace and heated at the desired temperature for 24 hours, as controlled by Pt/Pt-Rh(13%) thermocouples. At the end of the test run the capsule was inverted to cause K to drain into the collector. After cooling to room temperature the capsule was cut open. K was dissolved in butyl alcohol and inside walls of the capsule were leached with HCl to remove Ta traces. The butyl alcohol and HCl solutions were combined and K was converted to KCl. The Ta content was determined by spectrophotometric analysis.

SOURCE AND PURITY OF MATERIALS:

Ta: polycrystalline, containing $5 \cdot 10^{-4}$ % C, $1.2 \cdot 10^{-3}$ % O, $1.0 \cdot 10^{-3}$ % H, $<1 \cdot 10^{-2}$ % other metals.
K: 99.99 % pure, containing $1.5 \cdot 10^{-3}$ % O, $2 \cdot 10^{-4}$ % N, $5 \cdot 10^{-3}$ % Rb, $<3 \cdot 10^{-3}$ % other metals.

ESTIMATED ERROR:

Solubility: precision of analysis ± 8 %.
Temperature: accuracy ± 8 K, stability ± 4 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Tantalum; Ta; [7440-25-7] (2) Potassium; K; [7440-09-7]	Klueh, R.L. *Corrosion* 1972, *28*, 360-367.
VARIABLES: Temperature: 873-1273 K O concentration in K: 0.024-5.0 mol %	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

In preliminary experiments the apparent Ta solubilities in liquid K were reported as a function of exposure time and O concentrations in K and Ta. The solubility values obtained at shorter times than 5 hours were omitted, as it seemed that an equilibrium was not established in the system. All recalculations to mol % were made by the compilers.

t/°C	*time*/h	*O concn in K*/mass %	*O concn*/mol %	*soly*/mass % Ta	*soly*/mol % Ta
600	500	$1.0\cdot10^{-2}$ [a]	$2.4\cdot10^{-2}$	0.23	$5.0\cdot10^{-2}$
600	500	$1.0\cdot10^{-2}$ [a]	$2.4\cdot10^{-2}$	0.30	$6.5\cdot10^{-2}$
600	500	$1.0\cdot10^{-2}$	$2.4\cdot10^{-2}$	0.22	$5.4\cdot10^{-2}$
600	500	$6.0\cdot10^{-2}$	0.15	0.63	0.14
600	500	0.1	0.14	0.42	0.10
600	500	0.14	0.34	0.57	0.14
600	500	0.34	0.83	1.2	0.46
800	100	$5.0\cdot10^{-2}$ [a]	0.12	0.25	$5.4\cdot10^{-2}$
800	100	0.195 [a]	0.48	0.76	0.16
800	100	0.20	0.49	0.26	$5.6\cdot10^{-2}$
800	100	0.20 [a]	0.49	0.31	$6.7\cdot10^{-2}$
800	100	0.20	0.49	0.69	0.15
1000	50	$1.0\cdot10^{-2}$ [a]	$2.4\cdot10^{-2}$	0.68	0.15
1000	50	$4.5\cdot10^{-2}$	0.11	0.96	2.0
1000	50	0.19 [a]	0.46	2.06	0.43
1000	50	0.20 [a]	0.49	0.78	0.19
1000	50	0.20	0.49	1.5	0.32
1000	50	0.20	0.49	1.4	0.30

[a] Ta was penetrated by K, if the O content in Ta was higher than $5\cdot10^{-2}$ mass % O.

All results were also reported in (1).

A fresch Ta specimen was always applied in further experiments.

t/°C	*time*/h	*O concn in K*/mass %	*O concn*/mol %	*soly*/mass %	*soly*/mol % Ta
600	500	$6.0\cdot10^{-2}$	0.15	0.10	$2.2\cdot10^{-2}$
600	500	0.11	0.27	0.11	$2.4\cdot10^{-2}$
600	500	0.155	0.38	0.32	$6.9\cdot10^{-2}$
600	500	0.265	0.65	0.36	$7.8\cdot10^{-2}$
600	500	0.42	1.03	0.65	0.14
800	100	$6.0\cdot10^{-2}$	0.15	0.17	$3.6\cdot10^{-2}$
800	100	0.11	0.27	0.36	$7.8\cdot10^{-2}$
800	100	0.16	0.39	0.42	$9.1\cdot10^{-2}$
800	100	0.265	0.65	0.57	0.12
800	100	0.415	1.01	0.94	0.20
1000	50	$5.0\cdot10^{-2}$	0.12	0.68	1.4
1000	50	0.105	0.26	0.86	1.8
1000	50	0.16	0.39	1.12	2.3
1000	50	0.26	0.63	1.19	2.4
1000	50	0.41	1.0	1.57	3.2

The third phase in the system is most probably K_3TaO_4.

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Klueh, R.L.

Corrosion 1972, *28*, 360-367.

VARIABLES:

Temperature: 873-1273 K
O concentration in K: 0.024-5.0 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES: (continued)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The applied system consisted of a Ta specimen in contact with liquid K in a container of the same material, which was encapsulated in stainless steel. The capsule was filled with liquid K and welded in an Ar atmosphere chamber to prevent a contamination of K or Ta. The O concentration in K was varied by adding K_2O. While being heated to the test temperature the specimen was in the vapour zone of the capsule. The capsule was subsequently inverted to expose the specimen to liquid K for 0.5 to 500 hours. After testing the capsule was again inverted, quenched in liquid N and opened in an Ar atmosphere chamber. K was removed by dissolving in chilled isopropyl alcohol and recovered from it as KF.

The Ta amount present in K was determined by spectrographical analysis of KF. The specimen and container exposed to K with high O content had a black scale that partially spalled off during exposure to alcohol. The scale was soluble in H_2O. It was not analysed in detail, but certainly contained Ta.

SOURCE AND PURITY OF MATERIALS:

Ta: 99.9 % purity.
K: purified by exposing to Zr foil at 1073 K, containing $1.0\cdot10^{-2}$ % O.
Ar: nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Klueh, R.L. *US Atom.Ener.Comm. Rep. ORNL-4350*, 1969, p. 120.

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Hickam, C.W.

J. Less-Common Met. 1968, *14*, 315-322.

VARIABLES:

One temperature: 1255 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

A soluble corrosion product could not be detected in a blank test, in which a Ta specimen, not doped with O, was exposed to liquid K. Based on the blank test and considerations on the detection limits of the analytical method for the Ta determination, the solubility value of Ta in K at 982 °C was found to be lower than $1.0\cdot10^{-3}$ mass % Ta ($2\cdot10^{-4}$ mol % Ta, as calculated by the compilers).
The corrosion product was formed in the reaction: $3\ K + Ta + 4\ O_{(Ta)} \rightarrow K_3TaO_{4(K)}$ and was identified by means of x-ray diffraction methods.
The same information was also given in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ta capsule containing a coil specimen of the same material was filled with K, capped and sealed by electron-beam welding in a special vacuum facility. The capsule was heated at the selected temperature for 96 hours in a vacuum furnace. Temperature gradients in the capsule were minimized by keeping the capsules in a closely-fitting Mo block.

After the exposure the capsule was evacuated and opened. K was removed by distillation at 589 K.

The corrosion product was recovered for analysis in an Ar-atmosphere dry box. Ta and K were determined by spectrometric as well as flame-photometric methods.

SOURCE AND PURITY OF MATERIALS:

Ta: "commercially pure", containing $2\cdot10^{-2}$ % O.
K: unspecified purity, containing $<2.0\cdot10^{-3}$ % O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

(1) Hickam, C.W. *NASA Rep. TN-D-4213*, 1967.

COMPONENTS:	EVALUATOR:
(1) Tantalum; Ta; [7440-25-7] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1991

CRITICAL EVALUATION:

According to models of the metallic cell by Miedema (1) as well as the regular solution model of Hildebrand (2) the Ta solubility in liquid Rb is expected to be unmeasurably low at temperatures below the boiling point of Rb.

Young and Arabian (3) performed solubility determinations of Ta in liquid Rb in the temperature range of 811 - 1363 K. The reported values were scattered by about two orders of magnitude for each of the 4 test temperatures. The authors regarded their results as overestimated and could not provide an explanation for the scatter. It seems that either the test apparatus had a leak through which O entered the capsule or a systematic error was made during the procedures of analysis or cleaning of the apparatus. The data at temperatures above 961 K were obtained under the vapor pressure of Rb at the equilibration temperature.

Provided that the influence of O on the apparent solubility of Ta increases from Li to Cs (see the corresponding systems), a remarkable Ta solubility in liquid Rb is to be expected on the level of lower limits reported in (3). However, the determined higher limits could merely be due to an additional contamination of the test apparatus with atmospheric gases. The solubility values suggested by (3) could therefore not be recommended without further studies.

The Ta-Rb phase diagram was constructed by Garg et al. (4), and is similar to that shown for the Ta-Li system.

References

1. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroenia Diagram Sostoyania*, Nauka, Moskva, 1985, p. 113.
2. Marcus, Y. *Introduction to Liquid State Chemistry*, Wiley, London, 1977, p. 301.
3. Young, P.F.; Arabian, R.W. *US Atom.Ener.Comm. Rep. AGN-8063*, 1962.
4. Garg, S.P.; Venkatraman, M.; Krishnamurphy, N. *J.Alloy Phase Diagr.* 1990, *6*, 145.

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Young, P.F.; Arabian, R.W.

US Atom.Ener.Comm. Rep. AGN-8063, 1962.

VARIABLES:

Temperature: 811-1363 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ta solubilities in liquid Rb were graphically reported; the values (limits) were read off the figure and calculated to mol % by the compilers.

T/°F	$soly$/mass % Ta	$soly$/mol % Ta	number of experiments
1000	$1.1\cdot10^{-2}$ - 1.3	$5.2\cdot10^{-4}$ - 0.62	6
1400	$3.5\cdot10^{-2}$ - 1.1	$1.6\cdot10^{-2}$ - 0.52	13
1700	$2.5\cdot10^{-2}$ - 2.0	$1.2\cdot10^{-2}$ - 0.95	15
2000	$4.0\cdot10^{-3}$ - 6.0	$1.9\cdot10^{-3}$ - 2.9	7

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ta specimen was degreased with acetone, pickled in a mixture of HNO_3, H_2SO_4, HF, and H_2O (2:2:1:5), rinsed with H_2O and dried. An annealed Ta capsule, which had been cleaned the same way, was loaded with the specimen as well as Rb. The capsule was then welded in an Ar atmosphere, subsequently flame sprayed with Al_2O_3 and heated at the selected temperature for 50 hours. On removal, the capsule was inverted, causing Rb to flow into the Ta sample cup. The cup was cooled to room temperature. After solidification Rb was treated with anhydrous hexane, CH_3OH, and finally HCl. The resulting solution was taken to dry. The Ta content of the solid residue was spectrographically analysed in the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

Ta: 99.9 % purity; supplied by Fansteel Metallurgical Corp. containing 0.011 % O, 0.007 % C, 0.002 % H, 0.004 % N.
Rb: same as in the Nb-Rb system, same report.
Ar: nothing specified.

ESTIMATED ERROR:

Solubility: detection limit $3\cdot10^{-3}$ mol % Ta, error of analysis ± 10 %.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS: (1) Tantalum; Ta; [7440-25-7] (2) Cesium; Cs; [7440-46-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1991

CRITICAL EVALUATION:

Two solubility determinations of Ta in liquid Cs at 673 K are reported (1,6), the results of which are somewhat confusing. A significant content of 1 mol % Ta in the liquid Cs phase was determined in a test capsule made of Ta. The Ta content in Cs was below the detection limit of 1.10-2 mol% Ta, if the Ta specimen was placed in a stainless steel capsule in a second test (6). This may have been due to a possible interaction of Ta with components of the stainless steel (8) as well as to a lower O concentration in the system resulting from the potential of stainless steel to getter O from liquid Cs.

These solubility results were in partial agreement with some corrosion tests (2-4). Neither Harvey (2) nor Keddy (3) nor Smith et al. (7) observed corrosion of Ta in Cs at 698, 773, and 817 K, whereas Winslow (4) noticed a substantial covering of Ta with a nonadherent coating at 673 K, which was probably a mixed oxide of Ta and Cs. Corrosion of Ta by Cs vapour at temperatures of 1253 and 1643 K was not noticed. According to theoretical predictions (5) the solubility value is expected to be unmeasureably low at temperatures below the melting point of Cs. Reliable solubility values of Ta in liquid Cs cannot be suggested at present.

A predicted Ta-Cs phase diagram has been reported by Garg et al. (9), and is similar to that shown for the Ta-Li system.

References

1. Anonymous, Hughes Aircraft Corp., *NASA Rep. SP-41*, 1964, 243.
2. Harvey, R. *US Atom.Ener.Comm.Rep. MND-P-3009-1*, 1959.
3. Keddy, E.S. *US Atom.Ener.Comm.Rep. LAMS-2948*, 1963.
4. Winslow, P.M. *US Atom.Ener.Comm.Rep. CONF-650411*, 1965.
5. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroenia Diagram Sostoyania*, Nauka, Moskva, 1985, 113.
6. Winslow, P.M. *Corrosion*, 1965, *21*, 341.
7. Smith, R.G.; Hargreaves, F.; Mayo, G.I.J.; Thomas, A.G. *J. Nucl. Mater.*, 1963, *10*, 191.
8. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Mater., Materials Park, 1990.
9. Garg, S.P.; Venkatraman, M.; Krishnamurphy, N. *J.Alloy Phase Diagr.* 1990, *6*, 116.

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Anonymous, Hughes Aircraft Corp.,

NASA Rep. SP-41, 1964, 243-246.

VARIABLES:

One temperature: 673 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ta solubility in liquid Cs at 400 °C was reported to be 1 mass % Ta (~0.7 mol % Ta, as calculated by the compilers). A corrosion of Ta in Cs was not observed at temperatures of 980 and 1370 °C, while the solubility of Ta considerably decreased. However, numerical results were not reported.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ta sheet was cleaned by vapour degreasing and pickling. The sheet as well as Cs were installed in the test capsule under He atmosphere. Ta was exposed to Cs under static conditions for 50-500 hours. Heating of the capsule was performed in a furnace and controlled by a pyrometer. After equilibration the Cs in the capsule was solidified in liquid N. The capsule was opened in a He atmosphere. Cs was analysed by means of a spectrographic method.

SOURCE AND PURITY OF MATERIALS:

Ta: unspecified.
Cs: unspecified, containing $3 \cdot 10^{-2}$ % O.
Capsule material unspecified.

ESTIMATED ERROR:

Nothing specified.

COMPONENTS:

(1) Tantalum; Ta; [7440-25-7]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Winslow, P.M.

Corrosion, 1965, *21*, 341-349.

VARIABLES:

One temperature: 673 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ta solubility in liquid Cs at 400 °C was reported to be 1 mass % Ta (0.7 mol% Ta, as calculated by the compilers), if a Ta capsule was applied for determination. This result is also reported in (1). If a stainless steel capsule was applied instead of Ta, the Ta content in Cs was below the detection limit of $1.5 \cdot 10^{-2}$ mass % Ta ($1.1 \cdot 10^{-2}$ mol % Ta, as calculated by the compilers). A corrosion of Ta in the Cs medium at temperatures of 980 and 1370 °C was not observed, whereas the solubility considerably decreased. However, numerical results were not reported.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ta sheet was cleaned by vapour degreasing and pickling. The sheet as well as Cs were placed in the stainless steel or Ta test capsule in a He atmosphere. Ta was exposed to Cs under static conditions for 50-500 hours. The heating of the capsule was carried out in a furnace and controlled by a pyrometer. After equilibration, the Cs in the capsule was solidified in liquid N. The capsule was opened in a He atmosphere. Cs was analysed by means of a spectrographic method.

SOURCE AND PURITY OF MATERIALS:

Ta: unspecified.
Cs: 99.9 % pure, containing $(4\text{-}30) \cdot 10^{-3}$ % O.
Stainless steel: type unspecified.

ESTIMATED ERROR:

Solubility: detection limit $1.1 \cdot 10^{-2}$ % Ta.
Temperature: ± 5 K.

REFERENCES:

1. Anonymous, Hughes Aircraft Corp., *NASA Rep. SP-41*, 1964, 243-6.

COMPONENTS:	EVALUATOR:
(1) Chromium; Cr; [7440-47-3] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1992

CRITICAL EVALUATION:

Extensive experimental work has been performed in the Cr-Li system and has yielded consistent solubility data. Wilkinson and Yaggee (1) determined the Cr content in Li by equilibrating stainless steel samples containing 16 and 18 mass % Cr at 573 K with commercial Li gettered with U turnings. The solubility data of $5\cdot10^{-4}$ and $3\cdot10^{-4}$ mol % Cr seem to be overestimated due to a probable contamination of Li. Since the purity of Li was not specified and the experiments were only fragmentarily described, the results are not compiled.

Jesseman et al. (2) performed solubility determinations in the temperature range of 755 to 1277 K and observed an increase of the average solubility from $1.2\cdot10^{-3}$ to $5.7\cdot10^{-3}$ mol % Cr. The N content in Li was specified to be less than 0.01 mol %. Although the data of (1) and (2) were in rough agreement, the results of (2) were closer to the true solubility characteristics of absolutely pure metals. Beskorovainyi and Yakovlev (3) determined the Cr solubility in the temperature range of 673 to 1492 K using technical Li of unspecified purity. The results obtained in (3) were largely scattered. The solubility data increased with temperature from $3\cdot10^{-3}$ to 0.55 mol % Cr and were more than one order of magnitude higher than those obtained by (2). The results are therefore not compiled. Hoffman (25) reported that the solubility of (Cr+Fe) in liquid Li at 1311 K was found to be 2.7 mass %. Since Cr is better soluble in liquid Li than Fe, Cr should be the dominating component in solution. This approximate solubility value of Cr is overestimated and the result is rejected.

Bychkov et al. (20,21) performed several experiments on the corrosion resistance of Yal-T stainless steel (containing 17.3 mass % Cr) in liquid Li at 1248 and 1473 K. The marked influence of N as well as the minor one of O on the Cr euqilibrium concentration in Li at 1248 K was documented for the first time. The apparent solubility result obtained at 1473 K of $2.5\cdot10^{-2}$ mol % Cr was in fair agreement with precise results of subsequent, more intensive investigations.

Subsequent determinations by Leavenworth et al. (4) carried out at two N concentrations in Li ($7.4\cdot10^{-3}$ and $3.9\cdot10^{-2}$ mol % N) confirmed an influence of N on the measured Cr solubility. Their solubility data (4,5) in the temperature range of 934-1220 K were for a long time considered to be most reliable. Kelly (6) reported a solubility equation

$$\log(soly/\text{mol \% Cr}) = -0.339 - 3040\,(T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

which is most probably based on the data of (4,5) at the lower N level. Anthrop's (7) equation based·on (5) is quite similar:

$$\log(soly/\text{mol \% Cr}) = -0.136 - 3219\,(T/\text{K})^{-1} \qquad \text{Eq.(2)}$$

Gill et al. (8) did not indicate the source of their solubility equation:

$$\log(soly/\text{mol \% Cr}) = 1.67 - 3269\,(T/\text{K})^{-1} \qquad \text{Eq.(3)}$$

The solubility values calculated on the basis of Eq.(3) are two orders of magnitude higher than those reported by (5), thus demonstrating a similar temperature dependence of the solubility.

The single result reported by Weeks (9) ($1.6\cdot10^{-4}$ mol % Cr at 873 K) was obtained rather by an extrapolation of the data of (5) than by own measurements, as experimental details are not reported. Cheburkov (10) determined a solubility of Cr in distilled Li of 0.02, 0.14, and 0.2 mol % Cr at 1073, 1273, and 1473 K, respectively. The results are in agreement with the rejected data of (3). The work is not compiled, as further information was not available.

Plekhanov et al. (11) proved a dependence of the Cr solubility in liquid Li on the N content. An increase of the apparent solubility of about two orders of magnitude was observed due to an increase of the N concentration from $5\cdot10^{-3}$ to 0.25 mol % N in Li. A C content of 0.12 mol % in Li slightly increased the solubility, whereas an O content of up to 0.215 mol % in Li did not show any measurable effect on the determined Cr solubility. The experiments were performed in the temperature range of 773-1078 K.

The system has most recently been studied by Beskorovainyi et al. (12,13) who redetermined the Cr solubility in Li by means of a direct measurement of the equilibrium concentration of Cr in the test crucible by x-ray absorption technique. The experimental results obtained were convincing, since the Li used was of a very good purity ($\leq 5\cdot10^{-4}$ mol % N). The data are precisely described by the recommended equation (tested by the compilers):

$$\log(soly/\text{mol \% Cr}) = (4.74 \pm 0.01) - (8985 \pm 25)\,(T/\text{K})^{-1} \qquad \text{Eq.(4)}$$

Moreover, these data are close to the predicted solubility expressed by the equation (15):

$$\log(soly/\text{mol \% Cr}) = 2.813 - 9094\,(T/\text{K})^{-1} \qquad \text{Eq.(5)}$$

Beskorovainyi et al. (13,15,22,23,26) also investigated the "solubility" of Cr in Li being in equilibrium with several Cr-rich alloys in the temperature range of 1073 to 1273 K. In most of the tests the increase of the Cr content in the alloys caused a slight increase of the equilibrium Cr content in Li. Beskorovainyi et al. (16) also explained the N influence on the solubility of Cr in liquid Li by means of a thermodynamic model. They calculated the interaction parameter of the Cr-Li-N system and reported its decrease by a factor of eight in the temperature range of 773 to 1073 K. This explained the overestimated data (1-3, 10,11,24) in the presence of N in Li as well as the larger overestimation of the solubility at lower temperatures. If all the results are plotted as logarithmic solubility versus reciprocal temperature (Fig. 2), the high-temperature values approach the relations reported in (12,13), as well as the predicted solubility relation Eq.(5).

COMPONENTS:	EVALUATOR:
(1) Chromium; Cr; [7440-47-3] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1992

CRITICAL EVALUATION: (continued)

The composition of the compound formed at the Cr-Li interphase, which is probably responsible for the increase of the Cr solubility, was identified as Li_9CrN_5 at a temperature of 748 K and a N activity above 0.0025 (17,27). Concluding from measurements of the electrical resistivity (27), the solubility of Li_9CrN_5 in Li is certainly higher than of Cr, but still below the detection level of the method at 748 K.

The compounds CrN and Cr_2N are formed in bulk Cr reacting with N (17). Selle (18) reported a reduction of Cr_2N by Li at temperatures below 923 K, thus increasing the N content in liquid Li.

An assessed and speculative Cr-Li phase diagram was reported by Venkatraman and Neumann (19), it is shown in Fig. 1. Evidence of the existence of Cr-Li intermetallics could not be detected. The scatter of all solubility data in the Cr-Li system is shown in Fig. 2. Eq. (4) is recommended for extrapolations in respect to the temperature.

Recommended and tentative (t) solubility values of Cr in liquid Li

T/K	*soly*/mol % Cr	source
1023	$1\cdot10^{-4}$	(12,13)
1073	$2.5\cdot10^{-4}$	(11),(12,13)
1173	$1.3\cdot10^{-3}$	(4,5),(12,13)
1273	$5.4\cdot10^{-3}$	(2),(12,13)
1473	$3\cdot10^{-2}$ (t)	(21)

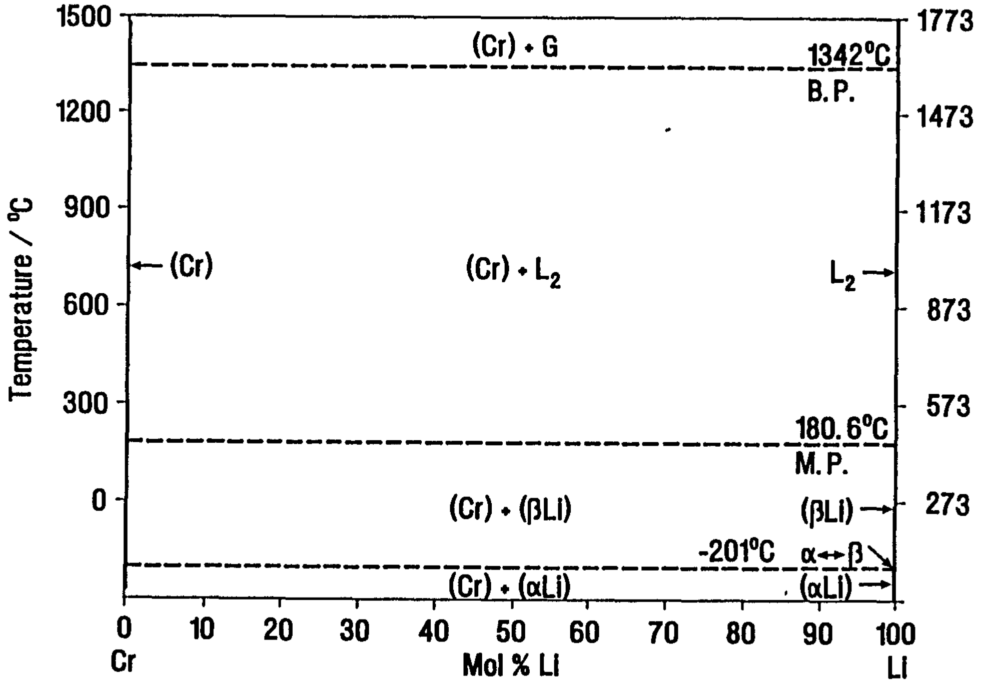

Fig. 1. The assessed Cr-Li phase diagram.

References

1. Wilkinson, W.D.; Yaggee, F.L. *US Atom.Ener.Comm. Rep. ANL-4990*, 1950.
2. Jesseman, O.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P. *US Atom. Ener.Comm. Rep. NEPA-1465*, 1950.
3. Beskorovainyi, N.M.; Yakovlev, E.I. *Metall. Metalloved. Chist. Met.*, 1960, *2*, 189.
4. Leavenworth, H.W.; Cleary, R.E.; Bratton, W.D. *US Atom.Ener.Comm. Rep. PWAC-356*, 1961.
5. Leavenworth, H.W.; Cleary, R.E. *Acta Metall.* 1961, *9*, 519.

COMPONENTS: (1) Chromium; Cr; [7440-47-3] (2) Lithium; Li; [7439-93-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1992

CRITICAL EVALUATION: (continued)

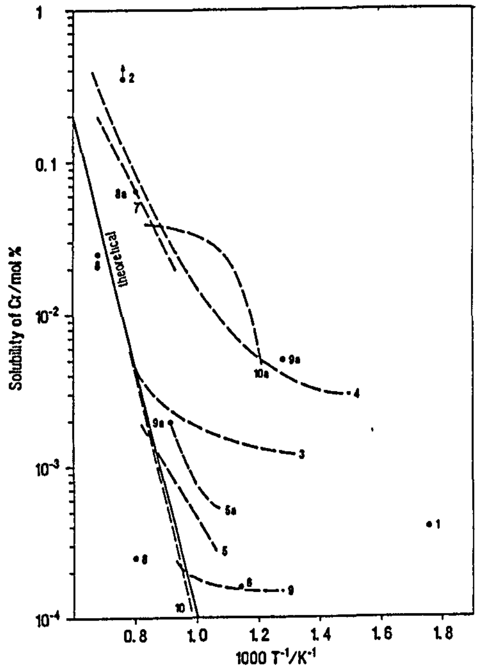

Fig. 2 The solubility of Cr in liquid Li plotted as function of the reciprocal temperature; the numbers denote: 1 - ref.(1), 2 - ref.(4), 3 - ref.(2), 4 - ref.(3), 5,5a - ref.(7), 6 - ref.(12), 7 - ref. (13), 8,8a - ref.(5,6), 9,9a - ref.(14), 10,10a - ref. (15,23). In the case of data 5a,8a,9a and 10a the N concentration in Li was higher than in the case of data 5,8,9, and 10, respectively.

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COMPONENTS:

(1) Chromium; Cr; [7440-47-3]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P.
US Atom.Ener.Comm. Rep. NEPA-1465, 1950.

VARIABLES:

Temperature: 755-1277 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubilities of Cr in liquid Li at various temperatures are reported.

$t/°C$	*test time*/h	*soly*/mass % Cr	*soly*/mol % Cr [a]
482	4	$1.6 \cdot 10^{-2}$, $8 \cdot 10^{-3}$	$1.6 \cdot 10^{-3}$
499	4	$8 \cdot 10^{-3}$	$1.1 \cdot 10^{-3}$
482	24	$1.0 \cdot 10^{-2}$, $1.6 \cdot 10^{-2}$	$1.7 \cdot 10^{-3}$
499	24	$1.5 \cdot 10^{-2}$	$2.0 \cdot 10^{-3}$
488	100	$1.0 \cdot 10^{-2}$, $8 \cdot 10^{-3}$	$1.2 \cdot 10^{-3}$ [b]
705	4	$1.2 \cdot 10^{-2}$, $1.4 \cdot 10^{-2}$	$1.7 \cdot 10^{-3}$ [b]
713	4	$1.9 \cdot 10^{-2}$	$2.5\ 10^{-3}$
705	24	$6.5 \cdot 10^{-3}$, $5.5 \cdot 10^{-3}$	$8 \cdot 10^{-4}$
713	24	$7.0 \cdot 10^{-3}$	$9.3 \cdot 10^{-4}$
705	100	$1.8 \cdot 10^{-2}$, $1.0 \cdot 10^{-2}$	$1.9 \cdot 10^{-3}$
713	100	$1.2 \cdot 10^{-2}$	$1.6 \cdot 10^{-3}$
978	4	$2.7 \cdot 10^{-2}$	$3.6 \cdot 10^{-3}$
1004	4	$3.0 \cdot 10^{-2}$	$4.0 \cdot 10^{-3}$
978	24	$3.3 \cdot 10^{-1}$, $4.5 \cdot 10^{-1}$	$5.2 \cdot 10^{-2}$
1004	24	$2.5 \cdot 10^{-1}$	$3.3 \cdot 10^{-2}$
1004	100	$4.0 \cdot 10^{-2}$, $4.8 \cdot 10^{-2}$, $4.0 \cdot 10^{-2}$	$5.7 \cdot 10^{-3}$ [b]

[a] mean values calculated by the compilers

[b] mean results suggested by the authors

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Strips of a Cr sheet were placed in a pair of Armco Fe capsules. The capsules were loaded with Li in an Ar dry box and degassed. Li was melted in a pot furnace. After welding the capsules were heated in a vacuum furnace. They were inserted in stainless steel plates within the furnace, the average temperature for each pair was estimated from the temperature gradient in the plate. The temperature was maintained for a period of 4-100 hours and the furnace was then air-cooled, while still being kept under low pressure. The capsules were weighed and opened. The solidified samples were leached out of the capsules with distilled water, and the Cr remaining undissolved was removed with the capsule, dried, and weighed as the tare to determine the amount of Li solution in the capsule. The leached material was filtered, and the residue analysed for the Cr content by means of a spectrographic method.

SOURCE AND PURITY OF MATERIALS:

Cr: unspecified.
Li: containing 0.24 % O, <0.02 % N.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: precision up to ± 30 %.
Temperature: precision ± 20 K.

REFERENCES:

COMPONENTS:

(1) Chromium; Cr; [7440-47-3]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Bychkov, Yu.F.; Rozanov, A.N.; Skorov, D.M.; Cheburkov, V.I.
Metall. Metalloved. Chist. Met. 1960, *2*, 78-92.

VARIABLES:

Temperature: 1248 and 1473 K
O and N contents in Li: 0.49 and 0.50 mol %, respectively

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The equilibrium content of Cr in liquid Li contacted with Yal-T stainless steel influenced by additions of various amounts of O or N to the solvent.

t/°C	*time*/h	*additive*/mass %	*additive*/mol % [a]	*soly*/mass %	*soly*/mol % Cr [a]
975	110	-	-	$<10^{-4}$	$<1.3\cdot10^{-5}$
975	240	-	-	$1.2\cdot10^{-3}$	$1.6\cdot10^{-4}$
975	110	0.99 % O	0.43 % O	$<10^{-4}$	$<1.3\cdot10^{-5}$
975	240	1.12 % O	0.49 % O	$2.6\cdot10^{-3}$	$3.4\cdot10^{-4}$
975	110	1.0 % N	0.50 % N	0.48	$6.4\cdot10^{-2}$
1200[b]	5	-	-	0.19	$2.5\cdot10^{-2}$

[a] calculated by the compilers
[b] reported in (1) and (2)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A specimen and a crucible of Yal-T steel were electrolytically polished. Additions of gazeous O or N to Li were performed in a separate glass apparatus. Loading of the crucible with Li and the specimen was carried out in a dry box filled with Ar. The crucible was welded and placed in a stainless steel container. The container was placed in an arc furnace and heated to the desired temperature for a given period of time. After equilibration the container was quenched in H_2O. The opened crucible was covered with paraffin. The Cr content in Li was analysed by means of a colorimetric method at the Institute of General and Inorganic Chemistry, Academy of Science USSR.

SOURCE AND PURITY OF MATERIALS:

Yal-T stainless steel: 17.3 % Cr, 10 % Ni, 0.48 % Si, 0.4 % Ti, 0.1 % C, 0.07 % Mn, 0.015 % P, 0.01 % S, rest Fe.
Li: distilled, containing $(2\text{-}6)\cdot10^{-2}$ % Na, $1\cdot10^{-2}$ % K, $4\cdot10^{-4}$ % Fe; Ni and Cr not detected.
O: from $KMnO_4$ decomposition.
N: "chemically" pure.
Ar: unspecified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B. *Atom. Energiya* 1959, *7*, 531; *Kernenergie* 1960, *3*, 763.
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COMPONENTS:

(1) Chromium; Cr; [7440-47-3]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Leavenworth, H.W.; Cleary, R.E.; Bratton, W.D.

US Atom.Ener.Comm. Rep. PWAC-356, 1961.

VARIABLES:

Temperature: 952 - 1190 K
N concentration in Li: $7.4 \cdot 10^{-3}$ and $3.9 \cdot 10^{-2}$ mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cr in liquid Li at varius temperatures and levels of N content in Li are presented in the figure and read off by the compilers;

T/K	*N concn. in Li*/mass %	*soly*/mol % Ti
948	0.015	$2.8 \cdot 10^{-4}$
1075	0.015	$6.5 \cdot 10^{-4}$
1124	0.015	$1.1 \cdot 10^{-3}$
1183	0.015	$1.3 \cdot 10^{-3}$; $1.6 \cdot 10^{-3}$
1215	0.015	$1.4\ 10^{-3}$
1220	0.015	$1.9 \cdot 10^{-3}$
934	0.079	$5.3 \cdot 10^{-4}$
987	0.079	$7 \cdot 10^{-4}$
1020	0.079	$1.5 \cdot 10^{-3}$
1031	0.079	$1.0 \cdot 10^{-3}$
1093	0.079	$2.0 \cdot 10^{-3}$

The results obtained at 0.015 mass % N in Li were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The specimen cup with a Cr layer of 10 mm inside was heated in a furnace to 811 K. Purified Li was added. The cup was then further heated to the desired temperature for a period of 24 hours. Samples of the saturated solution were removed by means of a Mo sample bucket. The sample was allowed to cool and was dissolved in 10 % HCl in a polyethylene beaker at dry ice temperature. HCl dissolved the solute metal traces, which had been adsorbed on the walls of the bucket. Standard colorimetric procedures were applied to determine the solute content, flame photometry to obtain the weight of the Li sample. Agitating the liquid during equilibration had no effect on the measured solubility. All operations were performed in an Ar atmosphere. Proper amounts of Li_3N were added to establish the N level in liquid Li.

SOURCE AND PURITY OF MATERIALS:

Cr: electroplated from H_2CrO_4 bath.
Li: 99.8 % pure; further purified by contacting with a Ti sponge at 1144 K for 2 hours, cooled to 260-315 K. N content $<5 \cdot 10^{-4}$ mol % N.
Ar: purified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

(1) Leavenworth, H.W.; Cleary, R.E. *Acta Metall.* 1961, *9*, 519-520.

COMPONENTS:

(1) Chromium; Cr; [7440-47-3]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Plekhanov, G.A.; Fedortsov-Lutikov, G.P.; Glushko, Yu.V.
Atom. Energiya 1978, *45*, 143-145; *Sov. Atom. Ener.* 1979, *45*, 818-819.

VARIABLES:

Temperature: 773 - 1078 K
O concentration in Li: $5 \cdot 10^{-3}$ or 0.22 mol %
N concentration: $4 \cdot 10^{-3}$-0.5 mol %
C concentration: $1 \cdot 10^{-3}$ or 0.12 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cr in liquid Li as a function of temperature and the contents of O, N, and C were determined.

t/°C	*additive in Li*/mol %	*soly*/mol % Cr	*mean soly*/mol % Cr
800-805	-	$2 \cdot 10^{-4}$, $2 \cdot 10^{-4}$, $3.3 \cdot 10^{-4}$	$2.4 \cdot 10^{-4}$
800-805	0.215 O; 0.025 N	$6.1 \cdot 10^{-4}$, $4.0 \cdot 10^{-4}$, $6.7 \cdot 10^{-4}$	$5.6 \cdot 10^{-4}$
800-805	0.12 C	$1.3 \cdot 10^{-4}$, $6.2 \cdot 10^{-4}$	$3.7 \cdot 10^{-4}$
800-805	0.025 N	$5.3 \cdot 10^{-4}$, $5.9 \cdot 10^{-4}$	$5.6 \cdot 10^{-4}$
800-805	0.10 N	$8.7 \cdot 10^{-4}$, $6.7 \cdot 10^{-4}$, $8.7 \cdot 10^{-4}$	$8.0 \cdot 10^{-4}$
800-805	0.25-0.27 N	$1.47 \cdot 10^{-3}$, $2.14 \cdot 10^{-3}$, $2.14 \cdot 10^{-3}$	$1.92 \cdot 10^{-3}$
650-660	-	$1.3 \cdot 10^{-4}$, $2.1 \cdot 10^{-4}$, $1.3 \cdot 10^{-4}$	$1.6 \cdot 10^{-4}$
500-505	-	-	$(1-2) \cdot 10^{-4}$
500-505	0.10 N	$1.73 \cdot 10^{-3}$, $1.60 \cdot 10^{-3}$, $1.73 \cdot 10^{-3}$	$1.70 \cdot 10^{-3}$
500-505	0.25-0.26 N	$5.1 \cdot 10^{-3}$, $5.1 \cdot 10^{-3}$, $4.8 \cdot 10^{-3}$	$5.0 \cdot 10^{-3}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An Armco Fe crucible, the inner side of which was chromium-plated , and a Mo container were placed in a stainless steel capsule. Filtered Li with additions of Li_2O, Li_3N or graphite was added to the crucible which was hermetically sealed by welding in an Ar atmosphere dry box. The system was equilibrated at the desired temperature for 24 hours. On removal, the capsule was inverted to force Li to flow into the Mo sampler. After cooling the Li sample was taken to spectral analysis.

SOURCE AND PURITY OF MATERIALS:

Cr: electroplated.
Li: containing $5 \cdot 10^{-3}$ mol % O, $(4-6) \cdot 10^{-3}$ % N, $(1-3) \cdot 10^{-3}$ mol % C.
Li_3N: pure.
Li_2O: pure.
C: spectrally pure.

ESTIMATED ERROR:

Solubility: detectivity of analysis $1 \cdot 10^{-4}$ mol % Cr; sample composition ± 1 %; precision of analysis ± 20 % (compilers).
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Chromium; Cr; [7440-47-3]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Beskorovainyi, N.M.; Vasilev, V.K.; Lyublinskii, I.E.

Metall. Metalloved. Chist. Met. 1980, *14*, 135-148.

VARIABLES:

Temperature: 773-1273 K
Concentration of N in Li: $1 \cdot 10^{-2}$ - 0.25 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cr in liquid Li at various temperatures was determined.

t/°C	*soly*/10^4 mass % Cr	*mean value*/10^4 mass % Cr	*soly*/mol % Cr
750	-	8	$1.1 \cdot 10^{-4}$
800	17, 17, 22, 17, 22	19±1	$2.5 \cdot 10^{-4}$
825	-	30±1	$4.0 \cdot 10^{-4}$
850	51, 39, 52, 44, 43	45±3	$6.0 \cdot 10^{-4}$
875	-	67±3	$8.9 \cdot 10^{-4}$
900	108, 97, 112, 91	99±5	$1.3 \cdot 10^{-3}$
925	-	142±4	$1.9 \cdot 10^{-3}$
950	200, 202	201±1	$2.7 \cdot 10^{-3}$
975	-	283±3	$3.8 \cdot 10^{-3}$
1000	-	408±26	$5.4 \cdot 10^{-3}$

The same results were reported in graphical form in (1). The fitting equation (confirmed by the compilers) is reported in the Critical Evaluation of the Cr-Li system.
Further equilibrium concentrations of Cr in Li with various Cr alloys used as solutes were graphically reported. They were read off by the compilers. The applied experimental method was the same.

Steel type	*Cr content in the alloy*/mass %	reference	*temperature range*/K	*soly range*/mass % Cr
12Kh18N10T	18.2	(2)	1098-1248	$2.3 \cdot 10^{-3}$-$1.3 \cdot 10^{-2}$
09Kh16N15M3B	15.7	(2,6)	1098-1298	$2.1 \cdot 10^{-3}$-$1.7 \cdot 10^{-2}$
0Kh16N15M3B	16.68	(1)	1098-1248	$2.2 \cdot 10^{-3}$-$1.4 \cdot 10^{-2}$
0Kh16N15M3BR	15.7	(1)	1098-1273	$1.4 \cdot 10^{-3}$-$1.5 \cdot 10^{-2}$
Kh8N8	8.1	(1)	1173-1273	$1.8 \cdot 10^{-3}$-$6.5 \cdot 10^{-3}$
Kh18N8	19.2	(1)	1148-1273	$2.0 \cdot 10^{-3}$-$8 \cdot 10^{-3}$
Kh8N18	8.4	(1)	1098-1273	$2.2 \cdot 10^{-3}$-$1.5 \cdot 10^{-2}$
Kh18N18	18.9	(1)	1198-1298	$8 \cdot 10^{-3}$-$2.3 \cdot 10^{-2}$
Kh30N30	31.7	(1)	1073-1248	$2.1 \cdot 10^{-3}$-$2.0 \cdot 10^{-2}$
1Kh13	13.0	(3)	1123-1273	$1.5 \cdot 10^{-3}$-$1.1 \cdot 10^{-2}$
1Kh12MVBFR	11.1	(3)	1123-1273	$1.3 \cdot 10^{-3}$-$9 \cdot 10^{-3}$
1Kh12NMZB	12.4	(4)	1123-1273	$1.6 \cdot 10^{-3}$-$8 \cdot 10^{-3}$
Kh12	12	(4,6)	1123-1223	$1.4 \cdot 10^{-3}$-$4.5 \cdot 10^{-3}$
10Kh18N10T[a]	18	(6)	773-1223	$2.2 \cdot 10^{-3}$-0.20
11Kh12N2M3B	12.4	(6)	1123-1273	$1.5 \cdot 10^{-3}$-$9.3 \cdot 10^{-3}$
08Kh21N5T	21	(6)	1090-1273	$1.3 \cdot 10^{-3}$-$1.3 \cdot 10^{-3}$
08Kh20N14S2	19.5	(6)	1090-1248	$2.2 \cdot 10^{-3}$-$1.4 \cdot 10^{-2}$
08Kh18N10T	18.6	(6)	1090-1298	$2.4 \cdot 10^{-3}$-$1.6 \cdot 10^{-2}$
KhN28VMAB	20.6	(6)	1090-1248	$3.0 \cdot 10^{-3}$-$1.5 \cdot 10^{-2}$
16Kh12VMFBR	11.1	(6)	1123-1273	$1.7 \cdot 10^{-3}$-$1.1 \cdot 10^{-2}$
10Kh13	13.0	(6)	1123-1273	$2.2 \cdot 10^{-3}$-$1.2 \cdot 10^{-2}$
16Kh12VMFBAR	10.9	(6)	1123-1273	$2.5 \cdot 10^{-3}$-$1.3 \cdot 10^{-2}$
Kh15MBF	15.0	(6)	1123-1223	$1.2 \cdot 10^{-3}$-$4.2 \cdot 10^{-3}$
Kh15F3	15.0	(6)	1123-1223	$1.3 \cdot 10^{-3}$-$4.3 \cdot 10^{-3}$
Kh15	15.0	(6)	1123-1223	$1.4 \cdot 10^{-3}$-$4.6 \cdot 10^{-3}$
Kh15N15	14.0	(6)	1098-1248	$2.0 \cdot 10^{-3}$-$9 \cdot 10^{-3}$
Kh15K3	15.0	(6)	1123-1223	$2.1 \cdot 10^{-3}$-$1.0 \cdot 10^{-2}$
Kh15N3	15.0	(6)	1123-1223	$2.0 \cdot 10^{-3}$-$7.3 \cdot 10^{-3}$
Kh15Yu3	15.0	(6)	1123-1223	$1.8 \cdot 10^{-3}$-$7.1 \cdot 10^{-3}$
Kh15B3	15.0	(6)	1123-1223	$1.7 \cdot 10^{-3}$-$6.3 \cdot 10^{-3}$
Kh15T3	15.0	(6)	1123-1223	$1.6 \cdot 10^{-3}$-$6.0 \cdot 10^{-3}$
Kh15M3	15.0	(6)	1123-1223	$1.5 \cdot 10^{-3}$-$5.0 \cdot 10^{-3}$

In all cases the plots log(*soly*/mass % Cr) vs. T^{-1} are linear, except [a], to which 0.25 mol % N was added.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Chromium; Cr; [7440-47-3] (2) Lithium; Li; [7439-93-2]	Beskorovainyi, N.M.; Vasilev, V.K.; Lyublinskii, I.E. *Metall. Metalloved. Chist. Met.* 1980, *14*, 135-148.
VARIABLES: Temperature: 773-1273 K Concentration of N in Li: $1 \cdot 10^{-2}$ - 0.25 mol %	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES: (continued)

The solubilities of Cr in liquid Li containing 0.5 mass % N (0.25 mol % N as calculated by the compilers) as reported in (5) and in a figure in (6); the applied experimental method was the same.

t/°C	*soly*/mass % Cr	*soly*/mol % Cr[a]	*t*/°C	*soly*/mass % Cr	*soly*/mol % Cr[a]
500	$3.6 \cdot 10^{-3}$	$4.8 \cdot 10^{-4}$	675	0.22	$2.9 \cdot 10^{-2}$
550	$3.5 \cdot 10^{-2}$	$4,7 \cdot 10^{-3}$	725	0.24	$3.2 \cdot 10^{-2}$
575	$6.5 \cdot 10^{-2}$	$8.7 \cdot 10^{-3}$	775	0.26	$3.5 \cdot 10^{-2}$
600	0.10	$1,3 \cdot 10^{-2}$	830	0.27	$3.6 \cdot 10^{-2}$
625	0.16	$2,1 \cdot 10^{-2}$	875	0.28	$3.7 \cdot 10^{-2}$
650	0.21	$2,8 \cdot 10^{-2}$	930	0.29	$3.9 \cdot 10^{-2}$

[a] calculated by the compilers

The solubilities of Cr in liquid Li containing 0.02 mass % N (0.01 mol % N as calculated by the compilers) were read out from the figure in (6) by the compilers.

t/°C	*soly*/mass % Cr	*soly*/mol % Cr[a]	*t*/°C	*soly*/mass % Cr	*soly*/mol % Cr[a]
700	$4.2 \cdot 10^{-2}$	$5.6 \cdot 10^{-3}$	850	$6 \cdot 10^{-2}$	$8 \cdot 10^{-3}$
750	$4.5 \cdot 10^{-2}$	$6.0 \cdot 10^{-3}$	900	$7 \cdot 10^{-2}$	$9 \cdot 10^{-3}$
800	$5 \cdot 10^{-2}$	$6,7 \cdot 10^{-3}$			

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A cylindrical crucible made of Cr was placed in a hermetic capsule. The crucible was loaded with Li which was kept inside by surface tension forces. A V foil was additionally placed inside. The capsule had "windows" of Be which were transparent to x-ray and did not contact with Li. The capsule was heated in a He atmosphere.

A beam of x-rays passed along the axis of the crucible. The beam passing through the sample was analysed in a Soller's spectrometer. The intensity of this beam was measured from both sides in relation to the K-boundary of absorption of Cr. The amount of Cr in Li was calculated by use of the corresponding formula. The concentration of N in Li was elevated by means of additions of Li_3N.

SOURCE AND PURITY OF MATERIALS:

Cr: "EHR" purity, remelted in Ar atmosphere.
Li: 99.6 % purity, subsequently distilled and equilibrated at 1248 K with Nb-Zr(5%) and Y; finally containing $<5 \cdot 10^{-3}$ % O and $\leq 1 \cdot 10^{-3}$ % N.
Li_3N: chemically pure.
He: unspecified.

ESTIMATED ERROR:

Solubility: precision ± 6 %; detection limit $1 \cdot 10^{-4}$ mol % Cr.
Temperature: stability ± 0.1 K.

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6. Gryaznov, G.M.; Evtikhin, V.A.; Zavyalskii, L.P.; Kosukhin, A.Ya.; Lyublinskii, I.E. *Materialovedenie Zhidkometallicheskikh Sistem Termoyadernykh Reaktorov*, Energoatomizdat, Moskva, 1989, p. 75-80, 106-108.

COMPONENTS: (1) Chromium; Cr; [7440-47-3] (2) Sodium; Na; [7440-23-5]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1995

CRITICAL EVALUATION:

Significant scatter of all experimental data of the Cr solubility in liquid Na indicates practical difficulties with this system. One probable explanation of this phenomenon might be the easy formation of Cr containing particles in liquid Na (1), if the test temperature is approached from a higher temperature level.

Taylor et al. (2) reported a saturation concentration of Cr in liquid Na of ~ $7.5 \cdot 10^{-5}$ mol % Cr at ~ 730 K and a concentration of O of ~ $8.5 \cdot 10^{-3}$ mol %. A chromium steel sample was equilibrated with Na for 720 h; further details were not given, and no data sheet is prepared.

A set of experiments on the Cr solubility in liquid Na was performed by Eichelberger et al. (3-5) who investigated the system in the temperature range 873-1273 K. The results were scattered more than one order of magnitude for a selected temperature and increased from $3.1 \cdot 10^{-5}$ to $8 \cdot 10^{-3}$ mol % Cr. The results reported in (5) - $(1.5; 1.4; 0.9; 0.7) \cdot 10^{-4}$ mol % Cr at 973, 1073, 1173, and 1273 K, respectively - seem to be preliminary since they were not confirmed in the subsequent studies (3,4) and further details were not supplied in (5).

Singer et al. (6) performed a number of experiments at 923 K with a changing equilibration time of 2 to 24 hours. The equilibrium was reached after 8 hours. The mean result of $1.8 \cdot 10^{-5}$ mol % Cr corresponds well with the previous determinations (3,4). Singer et al. (6) compared the solubility values estimated on the basis of their equation:

$$\log(\mathit{soly}/\text{mol \% Cr}) = 5.00 - 9010(T/\mathrm{K})^{-1} \qquad \text{Eq.(1)}$$

with Cr concentrations determined by five different laboratories in stainless steel loops. The amount of Cr in Na was approximately equal to or higher than that of the solubility at the maximum circuit temperature.

Aleksandrov and Dalakova (7) did not observe any dissolution of Cr in liquid Na after an equilibration of 1 h at 973-1023 K; the detection level of the spectral analysis used was not specified. Hajewska (8) reported that Cr of (18/8) stainless steel samples is unexpectedly easier dissolved in Na than the other components; however, numerical data were not published.

More recently, Pellett and Thompson (9) determined the Cr solubility in Na containing 10 times higher concentrations of O than in (3,4,6). The measurements were performed at 673 to 923 K. The values obtained were scattered from $3.9 \cdot 10^{-5}$ to $8.3 \cdot 10^{-4}$ mol % Cr, with a maximum solubility value at 773 K.

Alekseev et al. (10) studied the mass transfer in a non isothermal Na loop made of Kh18N10T stainless steel. Cr was determined by x-ray spectrometry. The experimental results were compared with modelling calculations, and a solubility equation was derived:

$$\log(\mathit{soly}/\text{mol \% Cr}) = 14.9 - 20746(T/\mathrm{K})^{-1} \qquad \text{Eq.(2)}$$

This equation is characterized by a extremely large slope and is not recommended, since its application would indicate a complete solubility of Cr in Na at ~ 1573 K which is not agreement with the findings.

The compilers suppose that the results of (3,4,6) are closer to the true Cr solubility in Na, since the applied components were of high purity. However, due to the observed scatter of data, the fitting Eq. (1) proposed by Singer et al. (6) which has been confirmed by the evaluators is merely regarded as preliminary. No satisfactory solubility equation has been reported in the opinion of one of the evaluators (11).

Kuzin et al. (12) formulated a predictive equation of the Cr solubility in liquid Na:

$$\log(\mathit{soly}/\text{mol \% Cr}) = 2.54 - 11520(T/\mathrm{K})^{-1} \qquad \text{Eq.(3)}$$

based on the cellular model of metallic solutions. Many experimental data of (3,4) are significantly lower than the prediction line.

The influence of O in Na on the solubility of Cr was experimentally confirmed by Klueh and DeVan (13), who equilibrated Na with a V specimen in a stainless steel capsule containing 18 mass % Cr at 873 K. The analysis of Na, which initially contained 0.65 mol % O indicated a variation of the Cr concentration in the range between $9 \cdot 10^{-5}$ and $4 \cdot 10^{-3}$ mol % Cr in 4 experiments. The influence of V on the Cr solubility data seems to be negligible, since the metals do not show significant interaction. The work is not compiled as further essential details were not reported. The applied method is identical with the one described in the data sheet on the V solubility in Na by the same authors.

The experimental solubilities of Cr in liquid Na are strongly influenced by the presence of O in Na, since various mixed oxides can be formed as the equilibrium solid state. The compound $NaCrO_2$ was identified as the typical reaction product. It is precipitated (14) in the system, if the solubility product of $NaCrO_2$ at a specific O level is exceeded. The measured thermodynamic stability data of this compound are scattered over a wide range (14-20). The influence of C on the Cr-Na-O system has been discussed by Mathews (20).

A predictive Cr-Na diagram was presented by Venkatraman and Neumann (21), which is similar to that shown for the Cr-Li system.

Doubtful solubility values of Cr in liquid Na at O concentration of ~10^{-4} mol %

T/K	*soly*/mol % Cr	source	remarks
873	$5 \cdot 10^{-6}$	Eq. (1)	
973	$5 \cdot 10^{-5}$	Eq. (1)	
1073	$4 \cdot 10^{-4}$	Eq. (1)	
1173	$2 \cdot 10^{-3}$	Eq. (1)	at the vapour pressure of Na
1273	$8 \cdot 10^{-3}$	Eq. (1)	at the vapour pressure of Na

COMPONENTS:	EVALUATOR:
(1) Chromium; Cr; [7440-47-3] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1995

CRITICAL EVALUATION: (continued)

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21. Venkatraman, M.; Neumann, J.P. *Bull. Alloy Phase Diagr.* 1984, *5*, 400.

COMPONENTS:

(1) Chromium; Cr; [7440-47-3]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.

US Atom.Ener.Comm. Rep. AI-AEC-12955, 1970.

VARIABLES:

Temperature: 873-1273 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cr in liquid Na at various temperatures was measured.

t/°C	*soly*/mass % Cr	*soly*/mol % Cr [a]	t/°C	*soly*/mass % Cr	*soly*/mol % Cr [a]
600	$8\cdot10^{-5}$	$3.5\cdot10^{-5}$	900	$3.4\cdot10^{-4}$ [c]	$1.5\cdot10^{-4}$
700	$1.2\cdot10^{-4}$ [c]	$5.3\cdot10^{-5}$	900	$1.4\cdot10^{-4}$ [b]	$6.2\cdot10^{-5}$
700	$8\cdot10^{-5}$ [c]	$3.5\cdot10^{-5}$	900	$1.14\cdot10^{-2}$ [c]	$5.0\cdot10^{-3}$
700	$8\cdot10^{-5}$ [b]	$3.5\cdot10^{-5}$	900	$2.7\cdot10^{-3}$	$1.2\cdot10^{-3}$
700	$1.15\cdot10^{-2}$	$5.1\cdot10^{-3}$ [d]	900	$1.82\cdot10^{-2}$ [c]	$8.0\cdot10^{-3}$
700	$7\cdot10^{-5}$	$3.1\cdot10^{-5}$	900	$1.42\cdot10^{-2}$	$6.3\cdot10^{-3}$
800	$1.0\cdot10^{-3}$ [c]	$4.4\cdot10^{-4}$	1000	$6.9\cdot10^{-4}$ [c]	$3.0\cdot10^{-4}$
800	$1.0\cdot10^{-4}$ [b]	$4.4\cdot10^{-5}$	1000	$1.3\cdot10^{-4}$ [b]	$5.7\cdot10^{-5}$
800	$1.1\cdot10^{-3}$ [c]	$4.8\cdot10^{-4}$	1000	$1.28\cdot10^{-3}$ [c]	$5.6\cdot10^{-4}$
800	$1.5\cdot10^{-3}$ [c]	$6.6\cdot10^{-4}$	1000	$7.6\cdot10^{-4}$	$3.3\cdot10^{-5}$
800	$2.8\cdot10^{-3}$ [c]	$1.2\cdot10^{-3}$	1000	$5.1\cdot10^{-3}$	$2.2\cdot10^{-3}$
800	$3.2\cdot10^{-3}$	$1.4\cdot10^{-3}$			

[a] calculated by the compilers

[b] analysed by atomic absorption spectroscopy; results of a wet chemical analysis were in the range of $(2\text{-}3)\cdot10^{-4}$ mass % Cr (1).

[c] also reported in (1)

[d] value neglected for the fitting equation

The authors proposed the fitting equation:

$$\log(soly/\text{mass \% Cr}) = 1.876 - 5632(T/\text{K})^{-1}$$

The validity of this equation is, however, questionable, since the results show considerable scatter at all temperatures.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Cr test crucible was cleaned with a HCl:H_2O (2:1) mixture, H_2O and an acetone wash. The collector was fabricated of a Nb-Zr(1%) alloy. The crucible-collector assembly was degassed for 2 hours at 623 K. The crucible was loaded with Na and the assembly was sealed by welding under high vacuum. The capsule containing the assembly was equilibrated at the desired temperature for 6 hours in an Ar atmosphere. The capsule was subsequently inverted causing Na to flow into the collector. The collector was cooled, opened and the Cr content in Na was analyzed by atomic absorption spectroscopy. In a second series of experiments Cr rods were irradiated to ^{51}Cr, cleaned, degassed and equlibrated with Na as before. The Cr content in Na was then determined by radiation counting.

SOURCE AND PURITY OF MATERIALS:

Cr: 99.996 % pure, containing $8\cdot10^{-4}$ % C, $1\cdot10^{-5}$ % H, $7\cdot10^{-4}$ % O, $1.3\cdot10^{-4}$ % N; Cr prepared by decomposition of prepurified CrI_3.

Na: 99.996 % pure, containing $8\cdot10^{-4}$ % C, $(0.6\text{-}4)\cdot10^{-4}$ % O.

Ar: unspecified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

(1) Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener. Comm. Rep. ANL-7520*, Pt.I, 1969, p. 319-324.

COMPONENTS:

(1) Chromium; Cr; [7440-47-3]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Singer, R.M.; Fleitman, A.H.; Weeks, J.R.; Isaacs, H.S.

Corrosion by Liquid Metals, J.E. Draley, J.R. Weeks, Eds., Plenum, N.Y., 1970, p. 561-576.

VARIABLES:

One temperature: 923 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The average solubility value at 923 K, as obtained by 61 measurements, is $4.1\cdot10^{-5}$ mass % Cr with a mean deviation of $2.7\cdot10^{-5}$ mass %. The results were only presented on a hardly readable graph. The value calculated by the compilers is $(1.8\pm1.2)\cdot10^{-5}$ mol % Cr.
According to the authors a segregation of Cr metallic particles at the Na/He-gas phase boundary is considered to complicate the experiments.
Cr concentrations which were measured in liquid Na taken from stainless steel circuits were of the same order as the solubility presented in this study.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Cr rod was dipped in Na, which had been filled into an etched Mo crucible. The crucible was pressurized, raised to test temperature and equilibrated for 2-24 hours. The equilibrium was reached after 8 hours. The Mo bucket was then dipped into the melt and kept there for 15 minutes. It was subsequently agitated and raised. After cooling the bucket, the Cr content was analyzed. The O level was balanced by adding Na_2O through the sampling part or by U-gettering of Na. All experiments were performed in a He atmosphere.

SOURCE AND PURITY OF MATERIALS:

Cr: unspecified.
Na: distilled, containing $<1\cdot10^{-4}$ % O, $\leq2\cdot10^{-4}$ % Cr, Fe, and Ni.
He: ultrapure grade, further purified by passing through molecular sieves and a filter, finally containing $4.5\cdot10^{-5}$ % O, $8\cdot10^{-5}$ % H_2O.

ESTIMATED ERROR:

Solubility: precision ± 35 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Chromium; ;Cr; [7440-47-3]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Pellett, C.R.; Thompson, R.

Liq. Met. Engin. Technol., BNES, London, 1985, *3*, 43-48.

VARIABLES:

Temperature: 673-923 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cr in liquid Na was determined at various temperatures.

t/°C	*soly*/mass % Cr	soly/mol % Cr [a]
400	$8.9 \cdot 10^{-5}$	$3.9 \cdot 10^{-5}$
450	$3.97 \cdot 10^{-4}$	$1.75 \cdot 10^{-4}$
500	$1.877 \cdot 10^{-3}$	$8.3 \cdot 10^{-4}$
550	$3.56 \cdot 10^{-4}$ [b]	$1.6 \cdot 10^{-4}$
600	$3.62 \cdot 10^{-4}$	$1.6 \cdot 10^{-4}$
650	$3.13 \cdot 10^{-4}$ [c]	$1.4 \cdot 10^{-4}$

[a] calculated by the compilers.

[b] average value of $(1.10, 1.90, 1.61, 1.78, 1.92, 7.22, 8.22, 5.11, 1.94, 4.85) \cdot 10^{-4}$ mass % Cr.

[c] average value of $(1.63, 5.10, 4.55, 5.48, 2.34, 1.75, 1.08) \cdot 10^{-4}$ mass % Cr.

$NaCrO_2$ was probably the equilibrium solid phase instead of metallic Cr.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The applied technique was the sealed capsule method described in (1). A Ni can was internally electroplated with Cr of 0.5 mm thickness. The metals were diffusion bonded by heating for 24 hours at 973 K in dry H. The whole crucible was irradiated to provide a specific activity of ^{51}Cr and stored for 2 weeks to allow γ emitting Ni isotopes to decay before being used. The can was filled with Na, welded in an Ar atmosphere and evacuated. It was heated at the temperature of interest for 45 hours in a furnace block in Ar atmosphere. A sample was taken by piercing the can lid with a Mo needle and withdrawing the sample through a 1.5 μm mean pore diameter Ni frit, which had been pretreated. The sample was collected in an alumina crucible and the activity of the whole sample was radiochemically counted. This was compared to the activity of a standard Cr solution of the same geometry.

SOURCE AND PURITY OF MATERIALS:

Cr: unspecified.
Na: "reactor grade" purity, distilled and electrorefined through β-alumina, containing $(1-2) \cdot 10^{-3}$ % O and < $3.4 \cdot 10^{-5}$ % Cr.
Ar: "high purity".

ESTIMATED ERROR:

Solubility: accuracy of radiochemical counting ± 10 %; precision ± 50 % (compilers).
Temperature: nothing specified.

REFERENCES:

(1) Stanaway, W.P.; Thompson, R. *Material Behaviour and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, New York, 1982, p. 421-427.

COMPONENTS:	EVALUATOR:
(1) Chromium; Cr; [7440-47-3] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1990

CRITICAL EVALUATION:

One extensive determination of the Cr solubility in liquid K was performed by Ordynskii et al. (1) in the temperature range 968-1285 K, generally under the vapour pressure of K at equilibration. The scatter of the 44 experimental points is not larger than one order of magnitude at a selected temperature, usually ± 50 %. The mean values of the solubility increased from $6\cdot10^{-5}$ to $6\ 10^{-4}$ mol % Cr for the above mentioned temperature range. Three different experimental methods were applied. Aleksandrov and Dalakova (2) did not detect any dissolution of Cr in liquid K after contacting the metals for 1 hour at 873 to 923 K; spectral analysis with unspecified detection level was used.

Schwarz (3) reported that a Cr saturation concentration of $6\cdot10^{-4}$ mol % Cr was determined in a sample of liquid K taken from a circuit at 373 K. As (3) did not provide experimental details, the work is not compiled. The O levels in K determined by (1) and (3) are significantly different ($1.7\ 10^{-3}$ and $4.9\cdot10^{-2}$, respectively); thus, an agreement of their results cannot be expected.

An increase of the O content in K certainly increases the solubility of Cr in K. Experiments of (4) have made evident, that even at low O activity $KCrO_2$ is formed in this system at temperatures between 623 to 773 K. K_4CrO_4 is formed at higher O concentrations in liquid K (5).

A speculative phase diagram was reported by Venkatraman and Neumann (6). It is similar to that which is shown in the Cr-Li system, differing in the melting point of the alkali metal.

The solubility equation proposed by (1) is valid for the solubility estimation of Cr in the temperature range 1000-1300 K, as proved by the evaluators.

$$\log(soly/\text{mol \% Cr}) = 0.033 - 4130(T/\text{K})^{-1}$$

Tentative values of the Cr solubility in liquid K at ~ $1\cdot10^{-3}$ mol % O

T/K	$soly$/mol % Cr	source	remarks
973	$6\cdot10^{-5}$	(1)	
1073	$1.5\cdot10^{-5}$	(1)	at constrained pressure
1173	$3\cdot10^{-4}$	(1)	at constrained pressure
1273	$6\cdot10^{-4}$	(1)	at constrained pressure

References

1. Ordynskii, A.M.; Popov, R.G.; Raikova, G.P.; Samsonov, N.V.; Tarbov, A.A. *Teplofiz. Vyssh. Temp.*, 1981, *19*, 1192.
2. Aleksandrov, B.N.; Dalakova, N.V.; *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.
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4. Ganesan, V.; Borgstedt, H.U. *J. Less-Common Met.*, 1985, *114*, 343.
5. Sridaran, R.; Krishnamurthy, D.; Mathews, C.K. *J. Nucl. Mater.* 1989, *167*, 265.
6. Venkatraman, M.; Neumann, J.P. *Bull. Alloy Phase Diagr.*, 1984, *5*, 398.

COMPONENTS:

(1) Chromium; Cr; [7440-47-9]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Ordynskii, A.M.; Popov, R.G.; Raikova, G.P.; Samsonov, N.V.; Tarbov, A.A.
Teplofiz. Vys. Temp. 1981, *19*, 1192-1197.

VARIABLES:

Temperature: 968-1285 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cr in liquid K was reported in a figure; the data were read off and recalculated by the compilers.

T/K	*soly*/mol % Cr
971	$3.9\cdot10^{-5}$ [a], $5.5\cdot10^{-5}$ [c], $7.1\cdot10^{-5}$ [b], $9.0\cdot10^{-5}$ [c]
976	$4.3\cdot10^{-5}$ [c]
968	$5.0\cdot10^{-5}$ [b], $6.2\cdot10^{-5}$ [a], $1.6\cdot10^{-4}$ [b]
974	$5.0\cdot10^{-5}$ [b]
1075	$9.0\cdot10^{-5}$ [a]
1072	$1.0\cdot10^{-4}$ [b]
1082	$1.1\cdot10^{-4}$ [c], $1.5\cdot10^{-4}$ [c]
1076	$1.0\cdot10^{-4}$ [c], $1.6\cdot10^{-4}$ [a], $1.9\cdot10^{-4}$ [a], $1.5\cdot10^{-4}$ [a]
1071	$1.1\cdot10^{-4}$ [c]
1069	$1.5\cdot10^{-4}$ [b], $1.6\cdot10^{-4}$ [b], $2.8\cdot10^{-4}$ [b]
1172	$1.0\cdot10^{-4}$ [b], $1.2\cdot10^{-4}$ [b], $1.3\cdot10^{-4}$ [b], $1.7\cdot10^{-4}$ [a], $2.0\cdot10^{-4}$ [b], $2.2\cdot10^{-4}$ [b], $2.8\cdot10^{-4}$ [a], $3.1\cdot10^{-4}$ [a], $3.9\cdot10^{-4}$ [b], $5.2\cdot10^{-4}$ [b], $6.1\cdot10^{-4}$ [b], $7.5\cdot10^{-4}$ [b]
1179	$1.3\cdot10^{-4}$ [b]
1183	$3.1\cdot10^{-4}$ [b]
1167	$2.7\cdot10^{-4}$ [b]
1163	$3.2\cdot10^{-4}$ [b]
1285	$4.1\cdot10^{-4}$ [b], $5.7\cdot10^{-4}$ [b]
1284	$5.2\cdot10^{-4}$ [a], $6.2\cdot10^{-4}$ [a]
1283	$7.0\cdot10^{-4}$ [b]

[a] spectral analysis, cooled with H_2O [b] spectrophotometric analysis, cooled with liquid N
[c] spectrophotometric analysis, centrifuge separation

The results were expressed by the fitting equation which was proved by the compilers:

$$\log(soly/\text{mol \% Cr}) = 0.033 - 4130\,(T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

All experiments were performed in a capsule made of 12Kh18N10T stainless steel. The capsule was filled with K. To increase the surface contact of the K with the material of the capsule, a foil made of the same material (12Kh18N10T) with a layer of electrolytic Cr of 40-50 µm thickness was embedded in it. The foil took up half the length of the capsule. The capsule was sealed, placed in a vacuum furnace, filled with inert gas, and equilibrated for at least 2½-3 hours. On removal the capsule was inverted, so the K could flow into the part not occupied by the foil, and cooled by H_2O for 50-70 sec to solidify the K. The capsule was cut open, the K sample was cut out with a bronze blade. Spectral analysis was performed after a preconcentration of the samples.
In a second analytical method the liquid K was directly poured into liquid N (1) at the equilibration temperature. The sample was analysed by means of a spectrophotometric method.

In a third method to determine the solubility at low solubilities the capsule was placed in a furnace-centrifuge at 1070 to 1125 K. The temperature was subsequently reduced and kept constant while the rotation was carried out for 2-2½ hours.

The furnace of the centrifuge was then opened and the capsule was cooled in transit with H_2O. The sample for spectrometric analysis was always taken from the upper part of the solidified K.

SOURCE AND PURITY OF MATERIALS:

Cr: electrolytic
12Kh18N10T steel: 18.2 % Cr, 9.1 % Ni, $5\cdot10^{-2}$ % C, 0.8 % Si, 1.5 % Mn, $2\cdot10^{-2}$ % S, 0.3 % Ti, $3.5\cdot10^{-2}$ % P, and Fe to balance.
K: containing $(2\text{-}5)\cdot10^{-4}$ % Cr, $7\cdot10^{-4}$ % O, $(3\text{-}5)\cdot10^{-4}$ % C, $(2\text{-}4)\cdot10^{-6}$ % H, $\leq 4\cdot10^{-5}$ % Fe, Ni.

ESTIMATED ERROR:

Solubility: detection limit 10^{-6}-10^{-5} mass % Cr.
Temperature: stability ± (3-5) K.

REFERENCES:

(1) Popov, R.G.; Raikova, G.P.; Samsonov, N.V.; Tarbov, A.A. *USSR patent no 319871*, Bull. Coll. no 33, 1971.

COMPONENTS:	EVALUATOR:
(1) Chromium; Cr; [7440-47-3] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1988

CRITICAL EVALUATION:

Data concerning the solubility of pure Cr in pure Rb are unknown. Young and Arabian (1) determined the equilibrium content of Cr in liquid Rb with the Haynes 25 alloy as the Cr source. It seems that the values of $2.1 \cdot 10^{-3}$ and $3 \cdot 10^{-3}$ mol % Cr obtained at 1033 and 1203 K, respectively, can be regarded as the apparent solubility of Cr in liquid Rb. Rb was moderately contaminated with O. Thus, the solubility of Cr was slightly higher than in pure Rb. On the other hand the Cr activity in the alloy which contained only 20 mass % Cr was considerably lower than unity, and the equilibration concentration of Cr in Rb may, therefore, be lower than its solubility in equilibrium with metallic Cr.

According to experiments performed by Suzuki et al. (2) with stainless steel specimens dipped in liquid Rb (with 22 mol % O) Cr should be less soluble in Rb than Mo, Fe, or Ni at 473 K. An opposite conclusion can be drawn from the results obtained by Pinchback et al. (5) who applied Rb with a low (unspecified) O content. The Cr content in the surface area of stainless steel was relatively lower than that of Fe and Ni, indicating a higher solubility of Cr in the temperature range 773-893 K. It cannot be excluded that the inverse solubility sequence is due to the differences in the O concentration in Rb.

Gadd and Borgstedt (3) carried out investigations on the formation of compounds in the Cr-Rb-O system, demonstrating that Cr getters O from liquid Rb to form $RbCrO_2$ at low O activity or Rb_4CrO_4 at higher O activity. A formation of a metastable phase Rb_3CrO_4 might also be possible.

A speculative Cr-Rb phase diagram reported by Venkatraman and Neumann (4) is analogous to that of the Cr-Li system.

<u>Tentative values of the Cr solubility in liquid Rb as reported by (1)</u>

T/K	*soly*/mol % Cr	remarks
1033	$2 \cdot 10^{-3}$	under constrained pressure
1203	$3 \cdot 10^{-3}$	under constrained pressure

References

1. Young, P.F.; Arabian, R.W. *US Atom.Ener.Comm. Rep. AGN-8063*, <u>1962</u>.
2. Suzuki, T.; Ohno, K.; Masuda, S.; Nakanishi, Y.; Matsui, Y. *J. Nucl. Mater.* <u>1987</u>, *148*, 230.
3. Gadd, P.G.; Borgstedt, H.U. *J. Nucl. Mater.* <u>1983</u>, *119*, 154.
4. Venkatraman, M.; Neumann, J.P. *Bull. Alloy Phase Diagr.* <u>1984</u>, 5, 402.
5. Pinchback, T.R.; Winkel, J.R.; Matlock, D.K.; Olson, D.L. *Nucl. Technol.* <u>1981</u>, *54*, 201.

COMPONENTS:

(1) Chromium; Cr; [7440-47-3]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Young, P.F.; Arabian, R.W.

US Atom.Ener.Comm. Rep. AGN-8063, 1962.

VARIABLES:

Temperature: 1033 and 1200 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cr in liquid Rb was determined, the data should be understood as apparent solubility values, since Haynes 25 alloy was used as the Cr source.

t/°F	*T*/K [a]	*soly*/mass % Cr	*soly*/mol % Cr [a]
1400	1033	$1.3\cdot10^{-3}$	$2.1\cdot10^{-3}$
1700	1200	$1.5\cdot10^{-3}$, $3.5\cdot10^{-3}$, $3.5\cdot10^{-4}$, $<1\cdot10^{-4}$	$3.0\cdot10^{-3}$ [b]

[a] calculated by the compilers

[b] mean result neglecting the last value

The formation of a mixed Cr-Rb oxide in the system is suggested.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The specimen and the capsule, both made of the Cr alloy, were degreased with acetone, pickled in a mixture of HNO_3, H_2SO_4, HF, and H_2O (2:2:1:5), rinsed with H_2O and alcohol, and dried. The sample was placed in the capsule, which had been filled with Rb. The capsule was then sealed in an Ar atmosphere, flame sprayed with Al_2O_3, and heated for 50 hours at the selected temperature. On removal, the capsule was inverted, causing Rb with the dissolved Cr to flow into a sample cup. The cup was cooled to room temperature. After solidification the cup was cut open and its content analysed. The Rb sample was treated with anhydrous hexane, CH_3OH, H_2O, and finally HCl. The resulting solution was taken to dry. The Cr content of the solid residue was analysed in the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

Haynes 25 alloy (Cr source): from Superior Tube Co., containing 20.42 % Cr, 49.65 % Co, 15.28 % W, 10.14 % Ni, 1.7 % Fe, 1.39 % Mn, 0.097 % C.
Rb: supplied by MSA Research Corp., purified by passing through a micrometallic filter, gettered with Ti-Zr alloy at 866 K, distilled and finally filtered into storage tank. O content after the purification was $(6\text{-}17)\cdot10^{-4}$ mass % and after test $(19\text{-}534)\cdot10^{-4}$ mass % (as in the Nb-Rb system, published in the same report).
Ar: nothing specified.

ESTIMATED ERROR:

Solubility: precision of analysis ± 10 %, detection limit $1\cdot10^{-4}$ mol % Cr.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS: (1) Chromium; Cr; [7440-47-3] (2) Cesium; Cs; [7440-46-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1988

CRITICAL EVALUATION:

Tepper and Greer (1) determined the Cr content in liquid Cs at 1255 K using the Haynes 25 alloy as the solute. The reported solubility limit of $<1.5\cdot10^{-3}$ mol % Cr for an equilibration time of 100 to 1000 hours seems to be correct, while the value gained after 10 hours ($4.5\cdot10^{-3}$) does not seem to be in equilibrium.
Winslow et al. (2,3) exposed 4 types of stainless steel to Cs for 48 and 312 hours at 675 K and analysed the Cr content after the test. A presence of Cr in the range of $5\cdot10^{-3}$ to 0.5 mol % Cr was observed. The initial purity of Cs was 99.9 %, the final purity after the test decreased to 96 %, indicating a marked dissolution of the test materials probably due to the presence of O in liquid Cs. The work is not compiled, as it is basically not a solubility determination and does not provide further essential details.
In contradiction to (2,3) Keddy (4) did not report a dissolution of either pure Cr or Cr containing stainless steels in Cs after testing the compatibility of Cr with liquid Cs for 100 hours at 773 K.
Godneva et al. (5) determined the Cr content in liquid Cs in the temperature range 323 to 573 K. The solubility values tend to decrease (from $4.1\cdot10^{-3}$ to $8.4\cdot10^{-4}$ mol % Cr) with increasing temperature. The enhanced dissolution of Cr seems to be due to O influence. An increase of the O concentration in Cs caused a decrease of the Cr content in Cs of one order of magnitude at 573 K, an unexpected fact. As the temperature dependence of the solubility is theoretically expected to be very steep (6), the inconsistent data of (5) need further investigation and explanation.
Due to the fact that the results of (1) were obtained at higher pressure than those of (5), and the temperature gap between (1) and (5) is very large, a comparison of these data would not be justified.
According to Antill et al. (7) the equilibrium solid phase in the Cr-Cs system contaminated with O is Cs_4CrO_4. A formation of Cs_5CrO_4 was also predicted, but could not directly be identified.
A schematic and speculative Cr-Cs phase diagram was reported by Venkatraman and Neumann (8), and it is similar to that shown for the Cr-Li system.

Doubtful values of the Cr solubility in liquid Cs

T/K	*soly*/mol % Cr	source	remarks
323	$4\cdot10^{-3}$	(5)	
423	$1\cdot10^{-3}$	(5)	
573	$8\cdot10^{-4}$	(5)	
1255	$<1\cdot10^{-3}$	(1)	at constrained pressure

References

1. Tepper, F.; Greer, J. *US Air Force Rep. ASD-TDR-63-824*, Pt.I, 1963; *Mine Safety Appliances Res. Rep. MSA-63-61*, 1963.
2. Holley, J.H.; Neff, G.R.; Weiler, F.B.; Winslow, P.M. *Electric Propulsion Development*, E. Stuhlinger, Ed., Academic Press, New York, 1963, p.341.
3. Winslow, P.M. *Corrosion* 1965, *21*, 341.
4. Keddy, E.S. *US Atom.Ener.Comm. Rep. LAMS-2948*, 1963.
5. Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.
6. Niessen, A.K.; deBoer, F.R.; Boom, R.; de Châtel, P.F.; Mattens, W.C.M.; Miedema, A.R. *CALPHAD* 1983, *7*, 51.
7. Antill, J.E.; Peakall, K.A.; Smart, E.F. *J. Nucl. Mater.* 1975, *56*, 47.
8. Venkatraman, M.; Neumann, J.P. *Bull. Alloy Phase Diagr.* 1984, *5*, 397.

COMPONENTS:

(1) Chromium; Cr; [7440-47-3]

(2) Cesium; Ce; [7440-46-2]

ORIGINAL MEASUREMENTS:

Tepper, F.; Greer, J.

US Air Force Rep. ADS-TDR-63-824, Pt.I, 1963.

VARIABLES:

One temperature: 1255 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Cr content in liquid Cs which has been equilibrated with the Haynes-25 alloy at 1800 °F was determined. The content was $1.8 \cdot 10^{-3}$ mass % Cr ($4.5 \cdot 10^{-3}$ mol % Cr, as calculated by the compilers) after 10 hours of exposure, but less than $6 \cdot 10^{-4}$ mass % (or less than $1.5 \cdot 10^{-3}$ mol % Cr, as calculated by the compilers) after 100 and 1000 hours.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Solubility measurements of Cr in Cs were performed by means of an inverted capsule technique. The testing device was composed of two parts: one was made of Cr for equilibration, and another one of alumina as receiver. The capsule was heated for 100 hours at 1644 K under vacuum, filled with Cs and welded in an Ar atmosphere. After equilibration, the capsule was inverted and cooled. The solidified Cs in the alumina part of the capsule was dissolved in CH_3OH and the crucible was cleaned with HCl. The Cr concentration was analysed by spectrographic methods.

SOURCE AND PURITY OF MATERIALS:

Haynes-25: containing 20 % Cr, 50 % Co, 15 % W, 10 % Ni, 3.7 % Fe, 2.7 % Mn, 0.051 % C, 0.0376 % N, 0.0201 % O.

Cs: 99.9+ % pure, supplied by Mine Safety Appliances Res. Co., purified by Zr turnings, finally containing $2.8 \cdot 10^{-3}$ % C, $1.2 \cdot 10^{-3}$ % O, $<2 \cdot 10^{-4}$ % N.

Ar: purified by hot and cold K-Na bubbler; O and H_2O content monitored.

ESTIMATED ERROR:

Solubility: detection limit $1.5 \cdot 10^{-3}$ mol % Cr.

Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:

(1) Chromium; Cr; [7440-47-3]

(2) Cesium; Ce; [7440-46-2]

ORIGINAL MEASUREMENTS:

Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S.

Zh. Prikl. Khim. 1974, *47*, 2177-2180.

VARIABLES:

Temperature: 323-573 K
O concentration in Cs: 0.08 and 0.8 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cr in liquid Cs at various temperatures and O concentrations was determined.

t/°C	*soly*/mass % Cr	*soly*/mol % Cr [a]
50	$1.6 \cdot 10^{-3}$	$4.1 \cdot 10^{-3}$
100	$<2 \cdot 10^{-5}$	$<5.1 \cdot 10^{-5}$
150	$4.8 \cdot 10^{-4}$	$1.2 \cdot 10^{-3}$
200	$3 \cdot 10^{-4}$	$7.6 \cdot 10^{-4}$
300	$3.3 \cdot 10^{-4}$	$8.4 \cdot 10^{-4}$
300 [b]	$3 \cdot 10^{-5}$	$7.6 \cdot 10^{-5}$

[a] as calculated by the compilers
[b] Cs containing 0.8 mol % O

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A specimen of Cr metal was covered with Cs under vacuum and equilibrated for 120 hours in a glass ampoule. The O content in Cs was increased by means of a controlled decomposition of a $KClO_3$-MnO_2 mixture. The ampoule glass did not undergo visible changes. Cs was dissolved in H_2O and volumetrically determined in the resultant hydroxide. An aliquot of the solution was treated with an acid. The remaining part was acidified with H_2SO_4 and diphenylcarbazide was added (1). The Cr content of the resulting solution was determined by means of colorimetric analysis.

SOURCE AND PURITY OF MATERIALS:

Cr: containing 1.12 % O, $8 \cdot 10^{-2}$ % Al.
Cs: 98-99 % purity, vacuum distilled, finally containing <0.01 % O, <1.5 % Rb.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

(1) Godneva, M.M.; Vodyannikova, R.D. *Zh. Anal. Khim.*, 1965, *20*, 831-836.

COMPONENTS: (1) Molybdenum; Mo; [7439-98-7] (2) Lithium; Li; [7439-93-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1989

CRITICAL EVALUATION:

Though the number of experiments (1-13) performed to determine the Mo solubility in liquid Li is rather large, the results obtained are insufficient. According to theoretical predictions solubility equation (1) and (2)

$$\log\,(soly/\text{mol \% Mo}) = 2.96 - 12932\,(T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

$$\log\,(soly/\text{mol \% Mo}) = 4.90 - 13110\,(T/\text{K})^{-1} \qquad \text{Eq.(2)}$$

were developed on the basis of the regular solution (14) and the cellular model (15). The Mo solubility is expected to be below the analytical detection limit at temperatures below 1200 K. However, measureable and comparatively large amounts of Mo were determined in liquid Li in several experiments. Information on the influence of N and O impurities in the system is contradictory in (7,8) and (13,22).

Jesseman et al. (1) determined a solubility of $1.0\cdot10^{-3}$ mol % Mo at an approximate temperature of 1280 K. The solubility in the temperature range 650-1172 K was below the detection limit of analysis which was estimated in various experiments to be between $1\cdot10^{-4}$ and $6\cdot10^{-4}$ mol % Mo. The Li used was moderately contaminated with O and N. The analytical method used by Bychkov et al. (2,3) was more sensitive; thus the detection limit was $7\cdot10^{-6}$ mol % Mo. Their results obtained at 1273 K were below the detection limit, while the Li sample equilibrated at 1473 K contained $(2.2\text{-}7.2)\cdot10^{-3}$ mol % Mo. The content of nonmetallic impurities in these experiments is unknown.

The conditions of the measurements performed by Leavenworth et al. (4,5) were more precisely defined than the previous ones. An increase of the Mo solubility from $4\cdot10^{-5}$ to $1.2\cdot10^{-4}$ mol % Mo at temperatures from 935 to 1178 K was noted. The mean results of these experiments were expressed by Kelly (6) in form of a fitting equation:

$$\log\,(soly/\text{mol \% Mo}) = -2.14 - 2160\,(T/\text{K})^{-1} \qquad \text{Eq.(3)}$$

The program was continued to evaluate the N and O influence on the equilibrium Mo concentration in liquid Li. When Li contained up to 0.21 mol % O (7) and 0.025 mol % N, the equilibrium Mo concentration in Li always was below $7\cdot10^{-6}$ mol % in the temperature range 1023-1813 K (8). An apparent solubility value of $3.9\cdot10^{-5}$ mol % Mo was determined at 1813 K and elevated pressure, the O concentration in Li was 0.21 mol %. These results are in partial agreement with (2,3), but in evident disagreement with (1) as well as (4,5). The contamination of Li and probably Mo undoubtedly increases the apparent Mo solubility, but it seems that this increase was neither due to O or N nor a combination of them.

DeMastry and Griesenauer (9) exposed Mo (containing ½ % Ti and 0.08 % Zr) samples to liquid Li at 1932 K and constrained pressure for 1000 hours. The Mo content after the test did not exceed the detection limit of $7\cdot10^{-5}$ mol % Mo, as only a slight increase of the N and C concentration in Li was observed. Since further details are not known, the paper is not compiled.

Further experiments by Eichelberger et al. (10) did not explain the discrepancies. Although the selection of very pure components had been carried out diligently, the solubility results in the temperature range 1465-1893 K were scattered between $1.4\cdot10^{-5}$ and $1.2\cdot10^{-4}$ mol % Mo and did not show any temperature dependence. The average value of all results was similar to that of (8) at 1813K.

Some solubility determinations were also carried out by Carpenter et al. (11). Numerical data were not reported, but the results were not in agreement with the theoretical predictions expressed in equation (1). Ivanov and Solovev (12) graphically reported a temperature dependence of the Mo solubility in Li which smoothly increased from $1\cdot10^{-6}$ to $2\cdot10^{-3}$ mol % Mo at 555 to 1613 K. Their work is not compiled, since the original paper was not available. The results were in partial agreement with the values obtained in (1) and had a similar coefficient of temperature dependence of the Mo solubility as in (6).

Kuzin et al. (20) determined the Mo solubility in liquid Li (containing $\leq 1\cdot10^{-3}$ % N) at 1273 K to be below $7\cdot10^{-5}$ mol % Mo. X-ray absorption spectroscopy was applied for analysis, but further details were not available.

Flament and Sannier (21) did not observe any dissolution of Mo after an exposure to liquid Li of 500 h; a Mo-rich alloy Mo-Re was equilibrated at 1473 K.

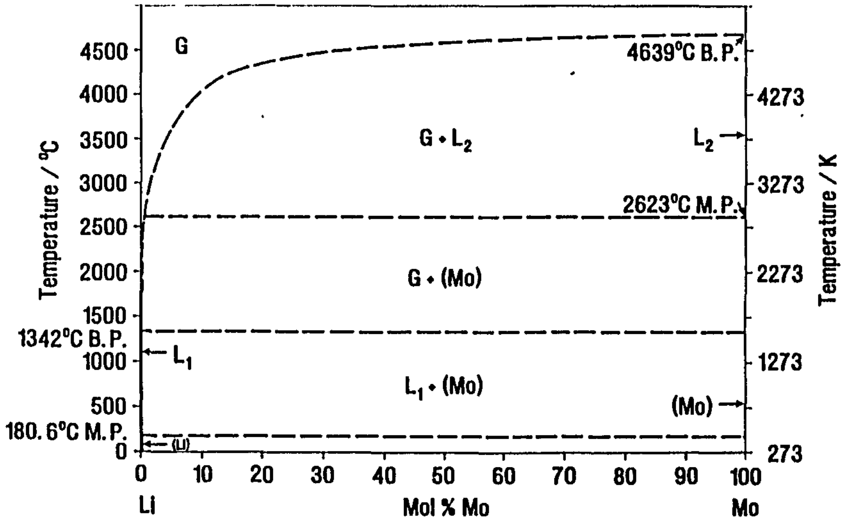
Beskorovainyi and Kirillov (13) determined the Mo solubility in Li at 1273 K containing 0.01 to 0.5 mol % N and observed an increase of the apparent solubility from $1.5\cdot10^{-3}$ to 0.1 mol % Mo. The authors explained this elevation as a consequence of a formation of Mo-N complexes in liquid Li. These complexes were investigated by electrotransport measurements. Kirillov et al. (22) extended the measurements to the temperature range 973-1473 K. They observed a substantial increase of the solubility of Mo due to an increase of the N content in Li at each of the test temperatures. Their results were in disagreement with the majority of former experimental data, especially (7,8), thermodynamic predictions (16) and corrosion tests (17). According to all these results the existence of Li-Mo-N compounds in Li is unlikely. Addison (18) predicted a formation of Li_9MoN_5. According to Lundberg and Feber (19) a formula Li_3MoN_3 is more likely. A formation of such a compound, even if primarily formed on the Mo surface, cannot be responsible for the apparent increase of the Mo solubility in the bulk. It was independently determined that Mo_2C might only be formed in the investigated system, if C was present in Li (17). Although it has not been experimentally proved, a formation of a Mo-CN compound might be possible if both contaminants were present in Li. Gryaznov et al. (23) reported that Cu introduced to the Mo-N-Li system decreases the solubility of Mo in liquid Li by 10^{-2}, most probably due to the formation of a more stable Cu-N-Li complex than the compound with Mo; see data sheet of (13).

Finally, a set of lower results of (8-10) might be selected as a tentative assessment, since they are close to the theoretical predictions of (14,15).

COMPONENTS:	EVALUATOR:
(1) Molybdenum; Mo; [7439-98-7] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1988

CRITICAL EVALUATION: (continued)

A schematic Mo-Li phase diagram, presented in (14), is redrawn in the figure below, Mo-Li intermetallics were not reported.



Tentative solubility values of Mo in liquid Li

T/K	*soly*/mol % Mo	source	remarks
1593	$\leq 7 \cdot 10^{-6}$	(8)	
1673	$1.4 \cdot 10^{-5}$	(10)	at elevated pressure
1813	$4 \cdot 10^{-5}$	(8)	at elevated pressure
1893	$8 \cdot 10^{-5}$	(9),(10)	at elevated pressure

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COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P.
US Atom.Ener.Comm. Rep. NEPA-1465, 1950.

VARIABLES:

Temperature: 650-1283 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent Mo solubilities in liquid Li at various temperatures and equilibration times are reported.

t/°F	t/°C^a	*time*/h	*soly*/mass % Mo	*soly*/mol % Mo^a
710	377	4	$<2.0\cdot10^{-3}$, $<3.5\cdot10^{-3}$	$<1.4\cdot10^{-4}$, $<2.5\cdot10^{-4}$
795	424	4	$<2.5\cdot10^{-3}$	$<1.8\cdot10^{-4}$
1060	571	4	$<3.0\cdot10^{-3}$, $<2.5\cdot10^{-3}$	$<2.2\cdot10^{-4}$, $<1.8\cdot10^{-4}$
1170	632	4	$<3.0\cdot10^{-3}$	$<2.2\cdot10^{-4}$
1535	835	4	$<4.0\cdot10^{-3}$, $<4.0\cdot10^{-3}$	$<2.8\cdot10^{-4}$, $<2.8\cdot10^{-4}$
1650	899	4	$<6.0\cdot10^{-3}$	$<4.3\cdot10^{-4}$
1820	993	4	$<1.5\cdot10^{-3}$, $<1.5\cdot10^{-3}$	$<1.1\cdot10^{-4}$, $<1.1\cdot10^{-4}$
1850	1010	4	$<1.5\cdot10^{-3}$	$<1.1\cdot10^{-4}$
710	377	24	$<4.0\cdot10^{-3}$, $<4.0\cdot10^{-3}$	$<2.8\cdot10^{-4}$, $<2.8\cdot10^{-4}$
795	424	24	$<6.0\cdot10^{-3}$	$<4.3\cdot10^{-4}$
1060	571	24	$<1.5\cdot10^{-3}$, $<1.5\cdot10^{-3}$	$<1.1\cdot10^{-4}$, $<1.1\cdot10^{-4}$
1170	632	24	$<1.5\cdot10^{-3}$	$<1.1\cdot10^{-4}$
1535	835	24	$<8.5\cdot10^{-3}$, $<8.5\cdot10^{-3}$	$<6.1\cdot10^{-4}$, $<6.1\cdot10^{-4}$
1650	899	24	$<8.5\cdot10^{-3}$	$<6.1\cdot10^{-4}$
1820	993	24	$<1.5\cdot10^{-3}$, $<1.5\cdot10^{-3}$	$<1.1\cdot10^{-4}$, $<1.1\cdot10^{-4}$
1850	1010	24	$<1.5\cdot10^{-3}$	$<1.1\cdot10^{-4}$
710	377	100	$<1.5\cdot10^{-3}$, $<1.5\cdot10^{-3}$	$<1.1\cdot10^{-4}$, $<1.1\cdot10^{-4}$
795	424	100	$<1.5\cdot10^{-3}$	$<1.1\cdot10^{-4}$
1060	571	100	$<1.5\cdot10^{-3}$, $<1.5\cdot10^{-3}$	$<1.1\cdot10^{-4}$, $<1.1\cdot10^{-4}$
1170	632	100	$<1.5\cdot10^{-3}$	$<1.1\cdot10^{-4}$
1535	835	100	$<2.5\cdot10^{-3}$, $<2.5\cdot10^{-3}$	$<1.8\cdot10^{-4}$, $<1.8\cdot10^{-4}$
1650	899	100	$<2.5\cdot10^{-3}$	$<1.8\cdot10^{-4}$
1800	982	100	$1.4\cdot10^{-2}$	$1.0\cdot10^{-3}$
1820	993	100	$1.4\cdot10^{-2}$, $1.4\cdot10^{-2}$	$1.0\cdot10^{-3}$, $1.0\cdot10^{-3}$

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Strips of a Mo sheet were placed in a Fe capsule. The capsule was loaded with Li in an Ar dry box, degassed and Li was melted in a pot furnace. After welding the capsule was heated in a vacuum furnace. The capsule was inserted in a stainless steel plate within the furnace, the average temperature of which was estimated from the temperature gradient in the plate. The temperature was maintained for a period of 4-100 hours and the furnace was then air-cooled while still being kept under low pressure. The capsule was weighed and opened. The solidified sample was leached out of the capsule with distilled water, and the Mo remaining undissolved was removed with the capsule, dried, and weighed as the tare to determine the amount of Li solution in the capsule. The leached material was filtered, and the residue spectrographically analysed for the Mo content.

SOURCE AND PURITY OF MATERIALS:

Mo: unspecified.
Li: containing 0.24 % O, <0.02 % N, a contamination with larger amounts of O and N is possible.
Capsule: Armco Fe

ESTIMATED ERROR:

Solubility: unspecified, detection limit probably $1\cdot10^{-4}$ mol % Mo.
Temperature: precision ± 20 K.

REFERENCES:

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B.;

Atom. Energiya 1959, *7*, 531-536; *Kernenergie* 1960, *3*, 763-767.

VARIABLES:

Temperature: 1273 and 1473 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mo in liquid Li at 2 temperatures was reported.

t/°C	*soly*/mass % Mo	*soly*/mol % Mo[a]
1000	$<10^{-4}$	$<7\cdot10^{-6}$
1200	0.03-0.1	$2.2\cdot10^{-3}$-$7.2\cdot10^{-3}$

[a] calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test crucible was prepared by sintering Mo powder. The inner surface of the Mo crucible was ground, electrolytically polished and etched. The crucible was gradually filled with freshly distilled Li dropping from a stainless steel condenser. After distillation the apparatus was filled with pure Ar. The crucible was placed in a stainless steel container, to which the cover was welded. Additionally, the crucible was isolated from the steel by a Mo band. The container was placed in an arc furnace and conditioned at a selected temperature for 100 hours. The Li solution was cooled to solidification in less than 50 s. The Mo content in the sample was determined by colorimetric analysis.

SOURCE AND PURITY OF MATERIALS:

Mo: unspecified.
Li: distilled, containing $(2\text{-}6)\cdot10^{-2}$ % Na, $1.5\cdot10^{-2}$ % K, $(1\text{-}4)\cdot10^{-4}$ % Fe, $\leq 2\cdot10^{-3}$ % Mg; Si, Ni, and Cr were not detected.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall. Metalloved. Chist. Met.*, 1960, *2*, 178-188; *Metallurgy and Metallography of Pure Metals*, Gordon & Breach, N.Y., 1962. p.178-188.

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Leavenworth, H.W.; Cleary, R.E.; Bratton, W.D.

US Atom.Ener.Comm. Rep. PWAC-356, 1961; abstracted in *Acta Metall.* 1961, *9*, 519-520.

VARIABLES:

Temperature: 933-1183 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mo in liquid Li were reported in a figure and read out and recalculated by the compilers.

T/K	*soly*/mol % Mo
933	$4 \cdot 10^{-5}$
1008	$6 \cdot 10^{-5}$
1045	$4 \cdot 10^{-5}$
1056	$8 \cdot 10^{-5}$
1060	$8 \cdot 10^{-5}$
1115	$1.0 \cdot 10^{-4}$, $8 \cdot 10^{-5}$
1136	$7 \cdot 10^{-5}$
1160	$1.1 \cdot 10^{-4}$
1178	$1.2 \cdot 10^{-4}$, $1.0 \cdot 10^{-4}$
1183	$6 \cdot 10^{-5}$

The results might be fitted to the equation presented in the Critical Evaluation, as determined by (1) and proved by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test equipment was essentially a dipping bucket apparatus, which is in detail described in (1). The specimen cup was heated in a furnace to 811 K and purified Li was added. The cup was then further heated to the desired test temperature for a period of 24 hours. A sample of the saturated solution was removed by means of a sample beaker made of Ti. The sample was allowed to cool and dissolved in 10 % HCl in a polyethylene beaker at dry ice temperature. A colorimetric method was applied to determine the Mo content, flame photometry to obtain the weight of the Li portion.

SOURCE AND PURITY OF MATERIALS:

Mo: 99.8 % pure.
Li: 99.8 % pure metal, further purified by gettering with a Ti sponge at 1144 K for 2 hours, cooled to 260 K; containing $<1 \cdot 10^{-3}$ % N, contaminated during test due to impurities of the cover gas and leakages of the apparatus.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Kelly, K.J. *NASA Rep. TN-D-769*, 1961, p. 27.

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Cleary, R.E.; Blecherman, S.S.; Corliss, J.E.

US Atom.Ener.Comm. Rep. TIM-850, 1965.

VARIABLES:

Temperature: 1023-1813K

N and O concentrations in Li: up to 0.21 and 0.025 mol %, respectively

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The test data indicated a Mo solubility in liquid Li at 750 to 1320 °C of less than $1.0 \cdot 10^{-4}$ mass % Mo ($7 \cdot 10^{-6}$ mol % Mo). Additions of up to 0.5 mass % O (0.21 mol % Mo) and 0.05 mass % N (0.025 mol % N) did not effect the solubility at temperatures equal to or lower than 1320 °C. A detectable quantity of $5.4 \cdot 10^{-4}$ mass % Mo was determined in Li ($3.9 \cdot 10^{-5}$ mol % Mo), which also contained 0.5 mass % O (0.21 mol % O) at a test performed at 1540 °C. All data indicated in mol % were calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Mo cup, which had been etched in HNO_3-HCl mixture, was placed inside a Nb capsule also serving as a receiver. The filled and welded assembly was covered with a Ta foil and encapsulated in a protective container of Mo-Ti(0.5%) alloy for application at temperatures above 1478 K, in an Inconel container for temperatures below 1478 K. The capsule was placed in a furnace in a receiver-up position and conditioned at the desired temperature for 100 hours. The capsule was inverted and the Li saturated with Mo transferred to the receiver. The capsule was cooled to room temperature and cut open in Ar atmosphere. The solidified Li was dissolved in H_2O, the receiver was etched in a HNO_3-HCl solution and Mo analysed as a complex with toluene-3,4-dithiol being extracted into CCl_4. The optical density of the solution was spectrophotometrically measured. N and O were added to Li as Li_3N and Li_2O, respectively.

SOURCE AND PURITY OF MATERIALS:

Mo: containing $2.3 \cdot 10^{-2}$ % C, $3.5 \cdot 10^{-3}$ % N, $1.8 \cdot 10^{-3}$ % O.

Li: purified by treating with Ti sponge at 1144 K for 2 hours, containing $\leq 5.0 \cdot 10^{-3}$ % N, ≤ 0.1 % O, $< 1.0 \cdot 10^{-2}$ % all other metals.

Ar: 99.99945 % pure with less than $1.5 \cdot 10^{-4}$ % H_2O, O_2, N_2, H_2.

ESTIMATED ERROR:

Solubility: detection limit $1 \cdot 10^{-4}$ mass % Mo ($7 \cdot 10^{-6}$ mol % Mo).

Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G.

NASA Rep. CR-1371, 1969; *Rep. AI-68-110*, 1968.

VARIABLES:

Temperature: 1465-1893 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mo in liquid Li at various temperatures was determined.

t/°C	*soly*/mass % Mo	*soly*/mol % Mo [a]
1192	$1.6 \cdot 10^{-3}$	$1.2 \cdot 10^{-4}$
1200	$6 \cdot 10^{-4}$, $4 \cdot 10^{-4}$	$4.3 \cdot 10^{-5}$, $2.9 \cdot 10^{-5}$
1390	$7 \cdot 10^{-4}$	$5.0 \cdot 10^{-5}$
1400	$2 \cdot 10^{-4}$	$1.4 \cdot 10^{-5}$
1405	$1.4 \cdot 10^{-3}$	$1.0 \cdot 10^{-4}$
1620	$1.1 \cdot 10^{-3}$	$7.9 \cdot 10^{-5}$

[a] calculated by the compilers.

The authors proposed to indicate the apparent solubility in this temperature range as $(9 \pm 5) \cdot 10^{-4}$ mass % Mo, since no distinct temperature dependence was noticed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Mo crucible and a Nb-Zr(1%) sample collector were placed in a Mo capsule. The assembly was cleaned, etched, dried, degassed under high vacuum at 1993 K and cooled in an Ar atmosphere. The final outgassing was performed at 623 K. After filling Li into the crucible the collector with its cup were installed and the capsule was sealed under vacuum. The capsule was heated to the desired test temperature and kept in the cup-up position for 4 hours. Finally, the capsule was removed from the furnace, the cup being then at the bottom. This way the liquid Li in the capsule was transferred to the collector. After cooling the capsule was cracked open, the collector separated, and a Li sample was melted from the collector into a special glass ware. The solidified Li was submerged in H_2O in an Ar atmosphere. The collector was rinsed with H_2O and hot 2 mol dm^{-3} HNO_3. The two solutions were subsequently joined. Mo was determined by means of spectrophotometric methods in form of toluene-3,4 dithiol complex in a 0.5 mol·dm^{-3} H_2SO_4 solution in presence of $FeSO_4$.

SOURCE AND PURITY OF MATERIALS:

Mo: 99.9 % pure, supplied by Materials Research Corp., containing $<3 \cdot 10^{-4}$ % O, $<1 \cdot 10^{-4}$ % H, $1.4 \cdot 10^{-2}$ % C, $9 \cdot 10^{-4}$ % N, $<1 \cdot 10^{-3}$ % Fe, Ni, Si.
Li: supplied by General Electric Corp., hot trapped with a Zr foil getter for 126 hours at 1093 K and distilled, containing $4.4 \cdot 10^{-3}$ % C, $1.3 \cdot 10^{-3}$ % N, $3.3 \cdot 10^{-3}$ % O, $<5 \cdot 10^{-3}$ % other metallic elements.
Ar: nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Beskorovainyi, N.M.; Kirillov, V.B.

Metall. Metalloved. Chist. Met. 1980, *14*, 149-156.

VARIABLES:

One temperature: 1273 K
Concentration of N in Li: 0.01-0.5 mol %
Concentration of Cu in Li: $\leq$ 0.1 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mo in liquid Li containing various amounts of N at 1273 K is reported.

N concn/mass %	*N concn*/mol % [a]	*soly*/mass % Mo	*soly*/mol % Mo[a]
0.001 [b]	0.0005	0.002	$1.4 \cdot 10^{-4}$ [b]
0.02	0.01	0.021±0.04	$1.5 \cdot 10^{-3}$
0.20	0.10	0.06±0.01	$4.3 \cdot 10^{-3}$
0.5	0.25	0.64±0.09	$4.6 \cdot 10^{-2}$
0.8	0.4	1.0	$7.2 \cdot 10^{-2}$ [c]
1.0	0.50	1.4±0.2	0.10

[a] calculated by the compilers.
[b] extrapolated value from the data of (1).
[c] reported in (2)

A formation of 2 complexes $Li_xMo_yN_z$ is suggested on the basis of electro-transport measurement, with z/y=1/2 for N concentrations of 0.01-0.1 mol % and z/y=2.3 for N concentrations of 0.1-0.5 mol %. The introduction of 1 mass % Cu (0.1 mol %) into the saturated solution of Mo in Li containing 0.5 mol % N decreases the solubility of Mo from 0.10 to $1.1 \cdot 10^{-3}$ mol % Mo after (2). A fitting equation was deduced by the authors of (2):

$$\log (soly/\text{mass \% Mo}) = -\ 1.480 - 0.476 \log (concn/\text{mass \% Cu})$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Li was equilibrated in a Mo ampoule under isothermal conditions for 50 hours. The ampoule was placed inside a stainless steel container. The N concentration in Li was regulated by additions of Li_3N. After the equilibration the container was inverted and Li flowed into a Nb receiver. The receiver was cooled and opened in an Ar atmosphere. The N and Mo content in Li was analysed. Mo was determined by spectral analysis of a H_2SO_4 solution. Additional electro-transport measurements were performed in an Ar atmosphere applying a Mo capillary and a stainless steel apparatus. Experimental details concerning the influence of Cu on the Mo-N-Li equilibria are not reported.

SOURCE AND PURITY OF MATERIALS:

Mo: indicated as "VM-1".
Li: indicated as "LE-1", 99.6 % pure.
Li_3N: chemically pure, as in the other publications of this laboratory.
Cu: nothing specified.
Ar: nothing specified.

ESTIMATED ERROR:

Solubility: precision ± 16 %.
Temperature: not specified.

REFERENCES:

1. Leavenworth, H.W.; Cleary, R.E. *Acta Metall.* 1961, *9*, 519-520.
2. Gryaznov, G.M.; Evtikhin, V.A.; Zavyalskii, L.P.; Kosukhin, A.Ya.; Lyublinskii, I.E. *Materialovedenie Zhidkometallicheskikh Sistem Termoyadernykh Reaktorov*, Energoatomizdat, Moskva, 1989, p. 102, 230.

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Kirillov, V.B.; Krasin, V.P.; Lyublinskii, I.E.; Kuzin, A.N.
Zh. Fiz. Khim. 1988, *62*, 3191-3195.

VARIABLES:

Temperature: 973-1473 K
Concentration of N in Li: 0.01-0.1 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mo in liquid Li containing various amounts of N was determined; the values were read out from the figure by the compilers.

T/K	*N concn*/mol%	*soly*/mol% Mo	*N concn*/mol%	*soly*/mol% Mo	*N concn*/mol%	*soly*/mol% Mo
973	0.01	$9.1 \cdot 10^{-5}$	0.05	$1.9 \cdot 10^{-4}$	0.1	$4.3 \cdot 10^{-4}$
1073	0.01	$4.0 \cdot 10^{-4}$	-	-	0.1	$1.13 \cdot 10^{-3}$
1173	0.01	$9.2 \cdot 10^{-4}$	0.05	$1.44 \cdot 10^{-3}$	0.1	$3.3 \cdot 10^{-3}$
1273	0.01	$1.55 \cdot 10^{-3}$	-	-	0.1	$4.8 \cdot 10^{-3}$
1373	0.01	$3.1 \cdot 10^{-3}$	0.05	$6.5 \cdot 10^{-3}$	0.1	$1.15 \cdot 10^{-2}$
1473	0.01	$5.7 \cdot 10^{-3}$	-	-	0.1	$2.3 \cdot 10^{-2}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Liquid Li was isothermally equilibrated with a Mo cup under inert atmosphere for 50 hours. The concentration of N was fixed at three levels by the addition of Li_3N. The resulting solution was analyzed for the Mo content by means of spectral analysis and for the N concentration by the Kjeldahl method after the equilibration.

SOURCE AND PURITY OF MATERIALS:

Mo: nothing specified.
Li: nothing specified, probably as in (1), 99.6 % pure, containing $2.5 \cdot 10^{-2}$ % O and $5 \cdot 10^{-3}$ % C.
Li_3N: chemically pure as in (1).

ESTIMATED ERROR:

Solubility: precision not better than ± 12 %.
Temperature: stability ± 3 K.

REFERENCES:

1. Evtikhin, V.A.; Kirillov, V.B.; Kosukhin, A.Ya.; Lyublinskii, I.E. *Fiz-Khim. Mekh. Mater.* 1986, *22*, no. 5, 45-48.

COMPONENTS:	EVALUATOR:
(1) Molybdenum; Mo; [7439-98-7] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1990

CRITICAL EVALUATION:

Aleksandrov and Dalakova (1) equilibrated Mo with liquid Na of unspecified purity for 1 h at 973-1023 K; they did not observe any dissolution of Mo by means of spectral analysis. Three experimental studies were concerned with solubility determinations of Mo in liquid Na. Eichelberger et al. (2,3) were the first to determine the solubility in the temperature range 973-1278 K, using metals of highest purity. The authors suggested a fitting equation based on 4 values increasing from $1.2 \cdot 10^{-5}$ to $3.1 \cdot 10^{-5}$ mol % Mo in the temperature range 1123-1273 K, while they omitted the two results of $2.9 \cdot 10^{-5}$ and $3.3 \cdot 10^{-5}$ mol % Mo at 973 and 1073 K. Four other results obtained at different temperatures were below the detection limit of $2.4 \cdot 10^{-5}$ mol % Mo. The proposed temperature relationship of the solubility of Mo in liquid Na may, therefore, not be correct. Some of the results (above 1157 K) were obtained at elevated pressure.

Klueh and DeVan (4) performed experiments on the solubility of Mo in Na containing about 0.28 mol % O at 873 K. The Mo content in Na after equilibration in a Mo capsule with a V specimen was $6.3 \cdot 10^{-3}$ mol % Mo (mean value), indicating a strong influence of O on the dissolution of Mo in Na.

Rajan Babu et al. (5) did not specify the O level in liquid Na. They decreased the O activity in Na by means of an addition of metallic Mg. An increase of the Mo solubility from $1.0 \cdot 10^{-6}$ to $1.0 \cdot 10^{-5}$ mol % Mo in the temperature range 555-705 K was observed by (5). Though the values were scattered in a range of ± 50 % the temperature dependence of the solubility is distinct enough and the proposed solubility equation is acceptable. An extrapolation of these data to higher temperatures would yield values of the Mo solubility of almost one order of magnitude higher than those determined by (2,3). A selection of doubtful data might be based on the results of (2,3) and (5).

All experimental solubilities were orders of magnitude higher than predicted values based on the regular solution theory (6) or the cellular model (7). The temperature dependencies of the predicted solubility versus temperature were many times steeper than those observed in the experiments by (2,3) and (5). The Mo solubility at 1156 K, for example, should be $1\ 10^{-9}$ mol % Mo (6).

The composition of the solid phase being in equilibrium with liquid Na saturated with Mo as reported in some publications is somewhat inconsistent. According to Barker (8) the compounds Na_4MoO_5, $NaMoO_2$, Na_2MoO_4 and Mo_2C (in presence of C) may be formed which is dependent on the O concentration as well as on the temperature. However, none of the ternary oxides was detected in the dynamic tests (9). Gnanasekaran et al. (10,11) determined in physico chemical investigations that solid Mo and liquid Na coexist with Na_2O at temperatures below 681 K; above this temperature the ternary compound Na_4MoO_5 coexists with the metals. A change of the slope in the solubility dependence may, therefore, be expected at a temperature ~ 680 K. Lundberg and Faber (12) predicted a formation of Na_2MoO_4 at 1400 K.

A schematic Mo-Na phase diagram is analogous to that of the Mo-Li system, presented in (5) (see Critical Evaluation of the Mo-Li system). Mo-Na intermetallics are not known.

Doubtful solubility values of Mo in liquid Na at 10^{-4} - 10^{-3} mol % O

T/K	*soly*/mol % Mo	source	remarks
573	$1.5 \cdot 10^{-6}$	(5) interpolated	
673	$6 \cdot 10^{-6}$	(5) interpolated	
1123	$1.2 \cdot 10^{-5}$	(2)	
1173	$2 \cdot 10^{-5}$	(2) interpolated	at constrained pressure
1273	$3 \cdot 10^{-5}$	(2)	at constrained pressure

References

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3. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt.I, 1969, p. 319; *Rep. AI-AEC-12680*; 1968.
4. Klueh, R.L.; DeVan, J.H. *J. Less-Common Met.* 1973, *30*, 9.
5. Rajan Babu, S.; Periaswami, G.; Geetha, R.; Mahalingam, T.R.; Mathews, C.K. *Liquid Metal Engineering and Technology*, BNES, London, 1984, *1*, 271.
6. Brewer, L.; Lamoreaux, R.H. *Atom. Ener. Rev., Spec. Issue no 7*, IAEA, Vienna, 1980, p. 195.
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12. Lundberg, L.B.; Feber, R.C. *US Dept.Ener. Rep. LA-UR-84-1952*, 1984.

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.

US Atom.Ener.Comm. Rep. AI-AEC-12955, 1970.

VARIABLES:

Temperature: 973-1278 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mo in liquid Na at various temperatures was determined:

t/°C	*soly*/mass % Mo	*soly*/mol % Mo[a]
700	$1.2\cdot10^{-4}$	$2.9\cdot10^{-5}$
800	$1.4\cdot10^{-4}$ [b], $<1.5\cdot10^{-4}$	$3.3\cdot10^{-5}$, $<3.6\cdot10^{-5}$
850	$5\cdot10^{-5}$ [b]	$1.2\cdot10^{-5*}$
900	9·10-5 [b], $<1\cdot10^{-4}$	$2.2\cdot10^{-5*}$, $<2.4\cdot10^{-5}$
950	$1.2\cdot10^{-4}$ [b]	$2.9\cdot10^{-5*}$
1000	$<1\cdot10^{-4}$, $<1\cdot10^{-4}$	$<2.4\cdot10^{-5}$, $<2.4\cdot10^{-5}$
1005	$1.3\cdot10^{-4}$ [b]	$3.1\cdot10^{-5*}$

[a] calculated by the compilers.
[b] also reported in (1) and (2).

The results marked with an asterisk were applied by the authors to form the fitting equation (proved by the compilers).

$$\log (soly/\text{mol \% Mo}) = -1.48 - 3807\ (T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Mo crucible was cleaned with a HNO_3+H_2O (1:1) mixture and subsequently washed with H_2O and acetone. A collector fabricated of a Nb-Zr(1%) alloy was cleaned the same way. The crucible-collector assembly was degassed for 2 hours at 1023 K. The assembly was filled with Na and welded under high vacuum. The capsule was equilibrated for 6 hours in an Ar atmosphere glove box at the desired temperature. Finally the whole apparatus was inverted in order to cause a flow of Na into the collector part. The entire Na sample was analysed for its Mo content after solidification by means of spectrophotometric determination.

SOURCE AND PURITY OF MATERIALS:

Mo: 99.992 % pure, supplied by Mater. Res. Corp., containing $1.0\cdot10^{-3}$ % C, $9\cdot10^{-5}$ % H, $4.3\cdot10^{-4}$ % O, $5\cdot10^{-5}$ % N.
Na: 99.996 % pure, containing $8\cdot10^{-4}$ % C, $(0.6\text{-}4)\cdot10^{-4}$ % O.
Ar: purified.

ESTIMATED ERROR:

Solubility: precision ± $5\cdot10^{-5}$ mass % at 95 % confidence level.
Temperature: nothing specified.

REFERENCES:

1. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt.I, 1969, p. 319-324.
2. McKisson, R.L.; Eichelberger, R.L.; Gehri, D.C.; Guon, J. *US Atom.Ener.Comm. Rep. AI-AEC-12680*, 1968, p. 155-163.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Molybdenum; Mo; [7439-98-7] (2) Sodium; Na; [7440-23-5]	Klueh, R.L.; DeVan, J.H. *J. Less-Common Met.* 1973, *30*, 9-24.
VARIABLES:	**PREPARED BY:**
One temperature: 873 K O concentration: 0.259-0.287 mol %	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Mo capsules had been applied for the determination of the solubility of V in liquid Na. The equilibrium concentration of Mo in Na after these experiments at a temperature of 600 °C, which could be regarded as the Mo solubility in Na containing a specified amount of O, are reported.

Exposure/h	*initial O concn in Na*/ mass %	mol %	*soly*/mass % Mo	*soly*/mol % Mo
100	0.200	0.287	$2.3 \cdot 10^{-2}$	$5.5 \cdot 10^{-3}$
200	0.180	0.259	$2.9 \cdot 10^{-2}$	$7.0 \cdot 10^{-3}$
300	0.195	0.279	$2.4\ 10^{-2}$	$5.8 \cdot 10^{-3}$
400	0.195	0.279	$2.9 \cdot 10^{-2}$	$7.0 \cdot 10^{-3}$
			mean value	$6.3 \cdot 10^{-3}$

The concentrations indicated in mol % are calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A V specimen as well as Na were placed inside a Mo capsule. The specimen was installed at the bottom of the capsule. The capsule was covered with a Ta foil liner and inserted in a stainless steel protective container. The container was kept at 873 K and rotated for 100 to 400 hours. Finally the capsule was inverted and quenched in liquid N. The O concentration was either determined by vacuum fusion or fast neutron activation analysis. The Mo content in Na was determined by a method, which was not further specified, after dissolving the Na sample in isopropyl alcohol. All operations were performed in an Ar atmosphere. The O concentration was regulated by additions of Na_20.

SOURCE AND PURITY OF MATERIALS:

Mo: nothing specified.
Na: probably purified by filtration at low temperatures and hot gettering with Zr chips at 873 K.
Ar: nothing specified.

ESTIMATED ERROR:

Solubility: precision ± 10 % (compilers).
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Babu, S.R.; Periaswami, G.; Geetha, R.; Mahalingam, T.R.; Mathews, C.K.
Liquid Metal Engineering Technology, BNES, London, 1984, *1*, 271-275.

VARIABLES:

Temperature: 555-705 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mo in liquid Na at various temperatures was measured.

T/K	*soly*/mass % Mo	*soly*/mol % Mo[a]
557	$4.2 \cdot 10^{-6}$	$1.01 \cdot 10^{-6}$
589	$9.5 \cdot 10^{-6}$	$2.3 \cdot 10^{-6}$
598	$2.18 \cdot 10^{-5}$	$5.23 \cdot 10^{-6}$
603	$1.76 \cdot 10^{-5}$	$4.22 \cdot 10^{-6}$
609	$7.4 \cdot 10^{-6}$	$1.78 \cdot 10^{-6}$
630	$2.3 \cdot 10^{-5}$	$5.52 \cdot 10^{-6}$
635	$2.43 \cdot 10^{-5}$	$5.83 \cdot 10^{-6}$
637	$1.4 \cdot 10^{-5}$	$3.36 \cdot 10^{-6}$
664	$4.2 \cdot 10^{-5}$	$1.01 \cdot 10^{-5}$
670	$4.82 \cdot 10^{-5}$	$1.16 \cdot 10^{-5}$
673	$2.1 \cdot 10^{-5}$	$5.04 \cdot 10^{-6}$
682	$2.83 \cdot 10^{-5}$	$6.79 \cdot 10^{-6}$
696	$2.26 \cdot 10^{-5}$	$5.42 \cdot 10^{-6}$
705	$4.35 \cdot 10^{-5}$	$1.04 \cdot 10^{-5}$

[a] calculated by the compilers.

The authors presented the fitting equation (confirmed by the compilers):

$$\log(soly/\text{mol \% Mo}) = -1.88 - 2200\,(T/\text{K})^{-1}$$

A slight interaction between Ta and Mo is possible (1), however, the dissolved amounts of both metals in Na are so low that their mutual influence might be regarded as negligible (compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Liquid Na was filled in a Ta crucible and weighed amounts of (3 %) Mg metal as well as several pieces of V were added. The crucible, a centrally placed "porosint" 5 µm pore size filter and a Ta collector were installed inside the capsule. The capsule was heated in the inverted position for nearly 40 hours. Sample collection was performed by inverting the assembly and pressing the Na solution through the filter by means of 0.2 MPa Ar. The Ta crucible with the collected Na was removed, weighed and taken for vacuum distillation at 623 K. The residue left in the Ta crucible was dissolved in aqua regia under weak heating. The pH of this solution was adjusted to 2.2. Mo was extracted with oxine in a methyl isobutyl ketone. The Mo content in the organic layer was analysed by means of atomic absorption spectrometric determination.

SOURCE AND PURITY OF MATERIALS:

Mo: 99.9 % pure, supplied by Goodfellow Metals.
Na:"nuclear grade purity", 99.95 % pure, kept at 473 K in an inert atmosphere.
Mg: 99.9 % pure.
Ar: unspecified.
All other chemicals were analytical grade reagents.

ESTIMATED ERROR:

Solubility: precision ± 10 %, detection limit $5 \cdot 10^{-7}$ mol % Mo.
Temperature: nothing specified.

REFERENCES:

1. Niessen, A.K.; Miedema, A.R. *Ber. Bunsenges. Phys. Chem.*, 1983, *87*, 717.

COMPONENTS:	EVALUATOR:
(1) Molybdenum; Mo; [7439-98-7] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1988

CRITICAL EVALUATION:

The solubility of Mo in liquid K was the subject of several experimental studies, however, a recommendation of solubility data is problematic, especially of data concerning pure components. Cleary et al. (1) performed solubility determinations in the temperature range 1023-1478 K applying K with an O content of $6.1 \cdot 10^{-3}$ mol %. The Mo concentration in K was below the analytical detection limit of $8 \cdot 10^{-6}$ mol % Mo. Measurable amounts of Mo in K were determined at an O concentration in K of $3.9 \cdot 10^{-2}$-1.2 mol % O. The apparent Mo solubility was determined to vary linearly with the O concentration in K, being independent on the applied additive (KO_2, KNO_3, K_2CO_3) as well as the solute source (Mo, Mo-0.5Ti). The extrapolated solubility value at 1368 K and $1.0 \cdot 10^{-3}$ mol % O in K is about $5 \cdot 10^{-5}$ mol % Mo, which is not in agreement with the value obtained at the detection limit of Mo in undoped K. An equation of the temperature dependence of the Mo solubility was reported for an O concentration of 0.12 mol %. Although the metals which McKisson et al. (2,3) applied for their solubility study were of highest purity and lowest O content in K ($1.0 \cdot 10^{-3}$ mol % O), the reported results were not as convincing as might be expected. In the first study (3), which was of preliminary character, the Mo solubility increased from $8 \cdot 10^{-5}$ to $8 \cdot 10^{-3}$ mol % Mo in the temperature range 1273-1473 K. As further experimental details are not reported, the data of (3) is compiled together with that of (2), where the authors observed an increase of the solubility from $1 \cdot 10^{-4}$ to $5 \cdot 10^{-4}$ mol % Mo in the same temperature range. They proposed a fitting equation, which seems to be preliminary, as the results obtained at 1373 K were scattered within one order of magnitude. In a subsequent report of this laboratory (4) Mo rich alloys were used for the determination of the Mo solubility. The Mo solubility of the Mo-0.5Zr alloy was one order of magnitude higher than that of the Mo-0.5Ti-0.07Zr alloy, the solubility values of pure Mo published in (2) being in between. As the presence of O in the system undoubtedly increases the apparent Mo solubility in K, it seems to be obvious that Ti is a more effective O getter in Mo than is Zr, which might be explained by the fact that Mo stronger interacts with Zr than with Ti (5).

Stecura (6,7) determined the Mo solubility in liquid K containing $4 \cdot 10^{-3}$ mol % O, reporting a smooth increase of the solubility from $2.4 \cdot 10^{-4}$ to $5 \cdot 10^{-4}$ mol % Mo in the temperature range 1040 to 1316 K. The solubility fitting equation reported in this work has a significantly less steep slope coefficient than that of (1) or (2), the results being roughly in the same order of magnitude as in (2). Litman (8) estimated the Mo content in liquid K to be less than $4 \cdot 10^{-4}$ mol % Mo, if both metals had been equilibrated at 1088 K for 100 hours and the O content in K was 0.024 to 0.5 mol %. Simons and Lagedrost (9) analysed K which had been equilibrated with Mo at 1400 K and noticed a dissolution of Mo of about $1 \cdot 10^{-3}$ mol %. The O concentration in K was $1.2 \cdot 10^{-2}$ mol %. As further details on these studies are not available, the papers are not compiled. The reported results were about 2 times higher than those presented in (2) at a similar temperature. Solubility predictions made by Brewer and Lamoreaux (10) were presented in form of an equation:

$$\log (soly/\text{mol \% Mo}) = 2.96 - 14707\,(T/\text{K})^{-1}$$

These data were merely in agreement with the data of (1), but not with all other experimental solubility data (2-4,6-8). Cleary et al. (1) identified K_2MoO_4 covering metallic Mo after equilibration with liquid K. Lindemer et al. (11) reported the formula of such a corrosion product as K_4MoO_5, while Shpilrain et al. (12) indicated it as K_2MoO_2. A thin surface layer of Mo_2C was identified, if K was doped with K_2CO_3 (1).

A schematic Mo-K phase diagram was reported in (10), which is analogous to that reported of the Mo-Li system. All solubility values measured at temperatures higher than 1032 K were obtained at constrain pressure. The suggested Mo solubility values were selected from (2-4,6,7), bearing in mind that these were apparent solubility values obtained at an O level in K of about $4 \cdot 10^{-3}$ mol % O.

Tentative values of the apparent Mo solubility in liquid K at ~ $4 \cdot 10^{-3}$ mol % O

T/K	$soly$/mol % Mo	source	remark
1073	$2.8 \cdot 10^{-4}$	(6,7)	at constrained pressure
1173	$3.5 \cdot 10^{-4}$	(6,7)	at constrained pressure
1273	$4.5 \cdot 10^{-4}$	(6,7)	at constrained pressure
1373	$6 \cdot 10^{-4}$	(2,3) and extrapol. (6,7)	at constrained pressure

References

1. Cleary, R.E.; Blecherman, S.S.; Corliss, J.E. *US Atom.Ener.Comm. Rep. TIM-850*, 1965.
2. McKisson, R.L.; Eichelberger, R.L.; Dahleen, R.C.; Scarborough, J.M.; Argue, G.R. *NASA Rep. CR-610*, 1966; *Rep. AI-65-210*, 1965.
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10. Brewer, L.; Lamoreaux, R. *Atom. Ener. Rev., Spec. Issue no 7*, IAEA, Vienna, 1980, p. 195.
11. Lindemer, T.B.; Besmann, T.M.; Johnson, C.E. *J. Nucl. Mater.* 1981, *100*, 178.
12. Shpilrain, E.E.; Fomin, V.A.; Skovorodko, S.N.; Sokol, G.F. *Issledovanie Vyazkosti Zhidkikh Metallov*, Nauka, Moskva, 1983, p. 38.

COMPONENTS:	**ORIGINAL MEASUREMENTS:**
(1) Molybdenum; Mo; [7439-98-7] (2) Potassium; K; [7440-09-7]	Cleary, R.E.; Blecherman, S.S.; Corliss, J.E. *US Atom.Ener.Comm. Rep. TIM-850*, 1965.
VARIABLES:	**PREPARED BY:**
Temperature: 433-1478 K O concentration in K: 0.039 and 1.2 mol % O	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mo in liquid K was below the analytical detection limit of $2 \cdot 10^{-5}$ mass % Mo ($8 \cdot 10^{-6}$ mol % Mo, as calculated by the compilers) in the temperature range of 750-1205 °C.

The apparent Mo solubility in liquid K containing $5.00 \cdot 10^{-2}$ % O was read off the figure.

t/°C	*soly*/mol % Mo^a	*soly*/mol % Mo^b
250	0.4	$4 \cdot 10^{-5}$
390	9	$9 \cdot 10^{-4}$
550	90	$9.0 \cdot 10^{-3}$
640	180	$1.80 \cdot 10^{-2}$

^{a} original values, however, factor 10^{-4} is necessary to fit the equation given below and other figures. ^{b} values corrected by the compilers.

The authors formulated the fitting equation:

$$\log (soly/\text{mol \% Mo}) = 1.86 - 3268\ (T/\text{K})^{-1}$$

The apparent Mo solubility in K with $5.00 \cdot 10^{-2}$ % O added as KO_2, as read off the figure.

t/°C	*soly*/mol % Mo	t/°C	*soly*/mol % Mo
200	$4 \cdot 10^{-5}$	600	$2 \cdot 10^{-2}$
400	$1 \cdot 10^{-3}$	850	$1.6 \cdot 10^{-2}$
420	$6 \cdot 10^{-4}$	1095	$1.2 \cdot 10^{-2}$, $1.6 \cdot 10^{-2}$, $1.8 \cdot 10^{-2}$, $2.0 \cdot 10^{-2}$
520	$9 \cdot 10^{-3}$	1200	$1.8 \cdot 10^{-2}$

The apparent Mo solubility in liquid K at 1095 °C depending on the O concentration in K.

O concn/mass %	solute source	*soly*/mol % Mo	*O concn*/mass %	solute source	*soly*/mol % Mo
$1.6 \cdot 10^{-2}$ KO_2	Mo-0.5%Ti	$2.6 \cdot 10^{-3}$		Mo	$3 \cdot 10^{-2}$
$4.0 \cdot 10^{-2}$ KO_2	Mo-0.5%Ti	$6 \cdot 10^{-3}$	$1.0 \cdot 10^{-1}$ KO_2	Mo	$2.3 \cdot 10^{-1}$
$4.8 \cdot 10^{-2}$ KO_2	Mo-0.5%Ti	$8 \cdot 10^{-3}$	$4.0 \cdot 10^{-1}$ KO_2	Mo	$1.0 \cdot 10^{-2}$
$1.0 \cdot 10^{-1}$ KO_2	Mo-0.5%Ti	$2.5 \cdot 10^{-2}$	$4.5 \cdot 10^{-2}$	Mo	$3.8 \cdot 10^{-2}$
$4.0 \cdot 10^{-1}$ KO_2	Mo-0.5%Ti	$1.7 \cdot 10^{-1}$	KNO_3	Mo	$2.6 \cdot 10^{-1}$
$4.8 \cdot 10^{-2}$ KO_2	Mo	$9 \cdot 10^{-3}$	$1.05 \cdot 10^{-1}$	Mo	$7 \cdot 10^{-3}$
$5.0 \cdot 10^{-2}$ KO_2	Mo	$1.2 \cdot 10^{-2}$	KNO_3	Mo	$4 \cdot 10^{-2}$
$6.0 \cdot 10^{-2}$ KO_2	Mo	$2.2 \cdot 10^{-2}$, $2.4 \cdot 10^{-2}$	$4.8 \cdot 10^{-1}$	Mo	$2.4 \cdot 10^{-1}$
			KNO_3		
			$4.8 \cdot 10^{-2}$		
			K_2CO_3		
			$1.1 \cdot 10^{-1}$		
			K_2CO_3		
			$5.0 \cdot 10^{-1}$		
			K_2CO_3		

K_2MoO_4 covering metallic Mo after equilibration with K was identified by chemical and x-rays analysis.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Mo cup, which had been etched in HNO_3-HCl, was installed inside a Nb receiver capsule. The capsule was sealed in an Ar atmosphere and placed in an Inconel shroud filled with Ar. The capsule was equilibrated in a vertical position with the solvent and the solute cup at the bottom at a selected temperature for 100 hours. It was subsequently inverted and liquid K transferred to the receiver. The capsule was cooled to room temperature and cut open in Ar atmosphere. The solidified K was dissolved in absolute alcohol, the receiver was etched in HNO_3-HCl.

Both solutions were joined and Mo was analysed as a complex with toluene-3,4-dithiol being extracted into CCl_4. The optical density of the solution was measured using a spectrophotometrical method.

O was added to K as KO_2, KNO_3, or K_2CO_3.

SOURCE AND PURITY OF MATERIALS:

Mo: containing $2.3 \cdot 10^{-2}$ % C, $3.5 \cdot 10^{-3}$ % N, $1.8 \cdot 10^{-3}$ % O.

K: containing $2.5 \cdot 10^{-3}$ % O.

Ar: 99.95 % pure with less than $1.5 \cdot 10^{-4}$ % H_2O, O_2, N_2, H_2.

ESTIMATED ERROR:

Solubility: detection limit $8 \cdot 10^{-4}$ mol % Mo; precision ± 25 % (compilers).

Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

McKisson, R.L.; Eichelberger, R.L.; Dahleen, R.C.; Scarborough, J.M.; Argue, G.R.
NASA Rep. CR-610, 1966; *Rep.AI-65-210*; 1965.

VARIABLES:

Temperature: 1273-1473 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mo in liquid K at various temperatures was determined.

t/°C	*time*/h	*soly*/mass % Mo	*soly*/mol % Mo[a]
1000	8	$4.3 \cdot 10^{-4}$	$1.7 \cdot 10^{-4}$
1000	4	$2.6 \cdot 10^{-4}$ [b]	$1.1 \cdot 10^{-4}$
1100	8	$1.3 \cdot 10^{-4}$	$5.3 \cdot 10^{-5}$
1100	4	$1.3 \cdot 10^{-3}$	$5.3 \cdot 10^{-4}$
1200	4	$1.39 \cdot 10^{-3}$	$5.7 \cdot 10^{-4}$
1200	8	$1.31 \cdot 10^{-3}$	$5.3 \cdot 10^{-4}$

[a] - as calculated by the compilers.

[b] - the value of $2.6 \cdot 10^{-3}$ mass % reported in the original was obviously a misprint, as can be seen from the primary data, source: 5 μg Mo in 1.54 g K.

The authors formulated a fitting equation which was confirmed by the compilers:

$$\log(soly/\text{mol \% Mo}) = 0.4 - 5600\,(T/\text{K})^{-1}$$

A set of probably preliminary Mo solubility results was also reported in (1).

t/°C	*time*/h	*soly*/mass % Mo	*soly*/mol % Mo[a]
1000	2	$2 \cdot 10^{-4}$	$8 \cdot 10^{-5}$
1000	8	$1.9 \cdot 10^{-3}$	$7.7 \cdot 10^{-4}$
1100	8	$1.7 \cdot 10^{-3}$	$6.9 \cdot 10^{-4}$
1200	4	$2.07 \cdot 10^{-2}$	$8.4 \cdot 10^{-3}$

[a] - as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The capsule, cap and collector were made of Ta. The Mo test crucible and the capsule parts were cleaned and rinsed with acetone, high-vacuum outgassed at 2373 K and cooled in an Ar atmosphere. A final outgassing of Ar was performed at 673 K. K was filled into the crucible. Finally the collector and the cap were installed and the capsule was sealed under high vacuum by electron-beam welding. The capsule was heated to the desired temperature and kept in the cap-up position for 2-8 hours. The capsule was swung out of the furnace through a 180° arc at the end of the test, so the liquid K was transferred from the capsule to the collector. After cooling, the capsule was cracked open and the collector was separated from the crucible. A K sample was melted from the collector into a special glassware. It reacted with H_2O vapour in He atmosphere acidified with HCl and HF. The K remaining in the collector was slowly dissolved in H_2O and rinsed with HCl. The Mo in the resulting solution was spectrographically determined in form of a toluene-3,4 dithiol complex in a 0.5 mol dm^{-3} H_2SO_4 solution in presence of $FeSO_4$.

SOURCE AND PURITY OF MATERIALS:

Mo: 99.999 % pure single crystal, supplied by Materials Research Corp., containing $1 \cdot 10^{-4}$ % O, $5 \cdot 10^{-5}$ % N, $3 \cdot 10^{-5}$ % H, $5 \cdot 10^{-4}$ % C, $5 \cdot 10^{-4}$ % Fe.

K: supplied by Mine Safety Appliance Research Corp., further purified by filtering at 363 K, hot gettering at Zr at 923 K, and distillation; containing $3.5 \cdot 10^{-4}$ % O, $4 \cdot 10^{-4}$ % N, $1.0 \cdot 10^{-3}$ % C, $<1 \cdot 10^{-3}$ % other elements.

ESTIMATED ERROR:

Solubility: very different precision (compilers).
Temperature: precision ± 2.5 K, as specified in (1).

REFERENCES:

1. McKisson, R.L.; Eichelberger, R.L. *US Atom.Ener. Comm. Rep. CONF-650411*, 1965, p. 37-42.

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G.

NASA Rep. CR-1371, 1969; *Rep. AI-68-110*, 1968.

VARIABLES:

Temperature: 1476-1683 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubilities of Mo in liquid K were measured using various Mo-rich alloys as sources.

solute	t/°C	*soly*/mass % Mo	*soly*/mol % Mo[a]
Mo-Zr(0.5%)	1203	$1.35 \cdot 10^{-2}$	$5.5 \cdot 10^{-3}$
Mo-Zr(0.5%)	1380	$7.0 \cdot 10^{-3}$	$2.8 \cdot 10^{-3}$
TZM	1400	$8.5 \cdot 10^{-4}$	$3.5 \cdot 10^{-4}$
TZM	1410	$1.5 \cdot 10^{-3}$	$6.1 \cdot 10^{-4}$

[a] calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Mo-alloy test crucible and a Nb-Zr(1%) sample collector were placed in a Mo capsule, cleaned, etched, dried, and degassed under high vacuum at 1993 K, subsequently cooled in an Ar atmosphere. The final outgassing was performed at 623 K. After filling K into the crucible, the capsule was sealed under high vacuum. The capsule was heated to the test temperature and equilibrated for 4 hours. Finally the capsule was removed from the furnace in a way that the liquid K in the capsule was transferred to the collector. The cooled capsule was cracked open and the collector was separated. A K sample was melted from the collector into a special glassware. It reacted with H_2O vapour in a He atmosphere acidified with HCl and HF. The K remaining in the collector was slowly dissolved in H_2O and subsequently rinsed with HCl. Both solutions were joined. Mo was determined by spectrophotometry in form of a toluene-3,4 dithiol complex in a 0.5 mol dm^{-3} H_2SO_4 solution in presence of $FeSO_4$.

SOURCE AND PURITY OF MATERIALS:

TZM: supplied by Climax Molybdenum Co., containing 0.5 % Ti, 0.07 % Zr, 0.03 % C, $2.0 \cdot 10^{-3}$ % O, $1.0 \cdot 10^{-3}$ % N, $5 \cdot 10^{-4}$ % H, $<1.2 \cdot 10^{-2}$ % other elements and balance Mo.
Mo-Zr(0.5%): supplied by Westinghouse Electric, Astronuclear Laboratories, containing 0.51 % Zr, $(1.2\text{-}1.5) \cdot 10^{-3}$ % C, $(1.5\text{-}3.1) \cdot 10^{-3}$ % O, $(1.5\text{-}3.4) \cdot 10^{-3}$ % N, $<2.0 \cdot 10^{-2}$ % other metals and balance Mo.
K: containing $5 \cdot 10^{-4}$ % O, $<1 \cdot 10^{-3}$ % other elements (each).
He: nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Stecura, S.

NASA Rep. TN-D-5504, 1969.

VARIABLES:

Temperature: 1040-1316 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mo in liquid K at various temperatures was determined.

T/K	*soly*/masss % Mo	*soly*/mol % Mo[a]
1040	$6 \cdot 10^{-4}$, $6 \cdot 10^{-4}$	$2.4 \cdot 10^{-4}$, $2.4 \cdot 10^{-4}$
1040	$1.1 \cdot 10^{-3}$	$4.4 \cdot 10^{-4*}$
1095	$7 \cdot 10^{-4}$, $8 \cdot 10^{-4}$	$2.8 \cdot 10^{-4}$, $3.2 \cdot 10^{-4}$
1176	$8 \cdot 10^{-4}$, $9 \cdot 10^{-4}$	$3.2 \cdot 10^{-4}$, $3.7 \cdot 10^{-4}$
1207	$9 \cdot 10^{-4}$, $1.0 \cdot 10^{-3}$	$3.7 \cdot 10^{-4}$, $4.1 \cdot 10^{-4}$
1269	$1.0 \cdot 10^{-3}$, $1.2 \cdot 10^{-3}$	$4.1 \cdot 10^{-4}$, $4.9 \cdot 10^{-4}$
1316	$1.2 \cdot 10^{-3}$, $1.4 \cdot 10^{-3}$	$4.9 \cdot 10^{-4}$, $5.3 \cdot 10^{-4}$

[a] calculated by the compilers

The authors formulated the fitting equation on the basis of all results except for the one marked with an asterisk. The equation was confirmed by the compilers.

$$\log\,(soly/\text{mol \% Mo}) = (-2.18 \pm 0.13) - (1472 \pm 156)\,(T/\text{K})^{-1}$$

The results are also graphically presented in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Mo crucible was placed in a Ni capsule, which also served as a collector at the end of a test. The crucible and the capsule were ultrasonically cleaned in CF_4 and a detergent. The crucible was etched in HNO_3+H_2O (77:23) for 2-5 min, and in HCl+H_2O (27:73) for 2 min. The capsule was etched in HNO_3+H_3PO_4+H_2SO_4+CH_3COOH (3:1:1:5) for ½-1 min at 363 K. The parts were subsequently rinsed with H_2O and dried. The crucible was filled with K in a vacuum chamber, the capsule was capped, electron-beam welded, and then placed in a furnace and heated for 24 hours. The temperature was measured by means of a Pt/Pt-Rh thermocouple. At the end of a test run the capsule was inverted and K collected. After cooling to room temperature the capsule was cut open. K was dissolved in butyl alcohol. The capsule was leached in HCl. Both solutions were joined and K was converted to KCl by evaporation. The Mo content was determined by spectrophotometric analysis.

SOURCE AND PURITY OF MATERIALS:

Mo: 99.97+ % pure, containing $2.7 \cdot 10^{-3}$ % C, $1.0 \cdot 10^{-3}$ % O, $7 \cdot 10^{-4}$ % N, $1 \cdot 10^{-4}$ % H, $<1 \cdot 10^{-3}$ % other metallic elements each.
K: 99.99 % pure, containing $<1.5 \cdot 10^{-3}$ % O, $2 \cdot 10^{-4}$ % N, $5 \cdot 10^{-3}$ % Rb, $<3 \cdot 10^{-3}$ % other metallic elements each.

ESTIMATED ERROR:

Solubility: analytical precision ± 10 %.
Temperature: accuracy ± 8 K; stability ± 4 K.

REFERENCES:

1. Stecura, S. *Corrosion by Liquid Metals*, J.E. Draley, J.R. Weeks, Eds., Plenum, New York, 1970, p. 601.

COMPONENTS:	EVALUATOR:
(1) Molybdenum; Mo; [7439-98-7] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1988

CRITICAL EVALUATION:

Practically only one paper (1) is concerned with the solubility of Mo in liquid Rb in the temperature range 1033-1363 K. For each of three test temperatures the results were scattered by more than ± 50 % and a distinct temperature dependence of the Mo solubility could not be determined. The reliability of the average solubility value of $4 \cdot 10^{-4}$ mol % Mo is difficult to estimate. Carlander (2) did not observe any corrosion of Mo by liquid Rb at 1311 K, thus supporting a low solubility value. On the other hand, Suzuki et al. (3) reported a relatively better solubility of Mo from stainless steel in liquid Rb containing 22 mol % O at 473 K compared to Cr. Mo was less soluble in liquid Rb compared to Fe and Ni.
According to theoretical predictions (4) the solubility of Mo is expected to be unmeasurably low at temperatures below 1500 K, the solubility at the boiling point of Rb (967 K), for example, was estimated to be $3 \cdot 10^{-13}$ mol % Mo. At other temperatures the predictive fitting equation might be applied:

$$\log (soly/\text{mol \% Mo}) = 2.96 - 15050\,(T/\text{K})^{-1}$$

This strong discrepancy between theory and experimental findings might be explained by an elevated apparent Mo solubility caused by the presence of O in the system. Metallic Mo is then most probably covered with solid Rb_2MoO_4 (3,5) which is in equilibrium with liquid Rb. The predictive schematic phase diagram of the Mo-Rb system is analogous to that reported for the Mo-Li system (4).

The doubtful solubility value of Mo in liquid Rb
in the temperature range 1033-1363 K, and at elevated pressure, is approximately $4 \cdot 10^{-4}$ mol % Mo.

References

1. Young, P.F.; Arabian, R.V. *US Atom.Ener.Comm. Rep. AGN-8063*, 1962; abstracted in *NASA Rep. SP-41*, 1964, p. 167.
2. Carlander, R. *US Atom.Ener.Comm. Rep. ORNL-2221*, 1957, p. 193.
3. Suzuki, T.; Ohno, K.; Masuda, S.; Nakanishi, Y.; Matsui, Y. *J. Nucl. Mater.* 1987, *148*, 230.
4. Brewer, L.; Lamoreaux, R.H. *Atom. Ener. Rev., Spec. Issue no 7*, IAEA, Vienna, 1980, p. 195.
5. Kohli, R. *Material Behaviour and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 345.

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Young, P.F.; Arabian, R.W.

US Atom.Ener.Comm. Rep. AGN-8063, 1962.

VARIABLES:

Temperature: 1033-1363 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubility of Mo in liquid Rb at various temperatures was measured; the values were read off the figure and recalculated by the compilers:

T/°F	T/K	*soly*/mass % Mo	*soly*/mol % Mo [a]
1400	1033	$2\cdot10^{-4}$, $3\cdot10^{-4}$, $6\cdot10^{-4}$, $7\cdot10^{-4}$	$4\cdot10^{-4}$ (mean value)
1700	1200	$1\cdot10^{-4}$, $2\cdot10^{-4}$, $3\cdot10^{-4}$, $4\cdot10^{-4}$	$2\cdot10^{-4}$ (mean value)
2000	1363	$3\cdot10^{-4}$, $1.0\cdot10^{-4}$	$4\cdot10^{-4}$ (mean value)

[a] - calculated by the compilers

The results were also shortly reported in (1). A fitting equation is not recommended.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Mo sample and a Ta capsule were cleaned in $CH_3COOH+HNO_3+HF+H_2O$ (30:10:2.5:37.5), rinsed with H_2O and dried in air. The sample was placed in the capsule which had previously been filled with Rb. The capsule was then sealed in an Ar atmosphere, flame sprayed with Al_2O_3 and heated at the selected temperature for 50 hours. On removal, the capsule was inverted, causing Rb with the dissolved Mo to flow into a sample cup. The cup was cooled to room temperature. After solidification it was cut open and its content analysed. The Rb sample was treated with anhydrous hexane, CH_3OH, distilled H_2O, and finally HCl. The Ta cup was treated with aqua regia and the solution was added to the previous one. The resulting solution was taken to dry. The Mo content of the solid residue was analysed in the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

Mo: 99.55 % pure, supplied by Cleveland Tungsten Inc., containing 0.43 % Ti, 0.02 % C.
Rb: purified by filtration, gettered with a Ti-Zr alloy at 866 K, vacuum distilled. O content after purification $(6-17)\cdot10^{-4}$ %.

ESTIMATED ERROR:

Solubility: detection limit $1\cdot10^{-4}$ mol % Mo.
Temperature: precision ± 3 K.

REFERENCES:

1. Young, P.F.; Arabian, R.W. *NASA Rep. SP-41*, Pt.I, 1964, p. 167-176.

COMPONENTS:	EVALUATOR:
(1) Molybdenum; Mo; [7439-98-7] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1988

CRITICAL EVALUATION:

Among the studies on interactions between Mo and Cs practically only 2 are concerned with their solubility phenomena. Tepper and Greer (1,2) determined the Mo solubility in liquid Cs at 1644 K to be $1.7 \cdot 10^{-3}$ and $1.4 \cdot 10^{-3}$ mol % Mo depending on the collector applied at the determinations. As can be seen in (1,2), the collector material (alumina (1) or Nb-Zr(1%) alloy (2)) had practically no influence on the determined solubility. The Mo content in Cs changed with the equilibration time, being most reliable at times of more than 100 hours. Godneva (3) investigated the Mo solubility in the temperature range 323-873 K. The Mo content was below $1.4 \cdot 10^{-4}$ mol % at temperatures ≤ 473 K; it increased to $2.4 \cdot 10^{-3}$ and $1.1 \cdot 10^{-3}$ mol % Mo at 573 and 873 K, respectively. A slight decrease of the solubility to $1.4 \cdot 10^{-3}$ mol % was observed at 573 K, if the O content in Cs increased to 0.8 mol % O. The latter data disagreed with corrosion observations of this system. Sedelnikov and Godneva (4) determined that the corrosivity of Cs on Mo is the stronger, the higher is the O content in Cs. Tepper and Greer (19) performed compatibility tests with a Mo-alloy in the temperature range of 368-533 K and determined that Mo is easier dissolved than Nb, but less than Fe. This observation is in partial agreement with the results of (3).

Holley et al. (5) analysed Cs which had been in contact with Mo for many hours at about 1275, 672 K and ambient temperatures. The corresponding results were $2 \cdot 10^{-2}$, 0.4 and $2 \cdot 10^{-2}$ mol % Mo. Thus, a temperature dependence could not be deduced. These experiments were corrosion tests and no solubility determinations. It is unknown whether the Cs solution was homogenous or containing a suspension of the solute metal. The applied Cs was of 99.9 % purity, the purity of Mo and the level of nonmetallic impurities in the system, however, was not specified. Winslow (6) determined up to $8 \cdot 10^{-3}$ mol % Mo in Cs containing $(3.3\text{-}25) \cdot 10^{-2}$ mol % O, which had been equilibrated for 500 hours at 673 K. As further details were not available, papers (5) and (6) are not compiled.

Chandler and Hoffman (7) observed a significant dissolution of Mo at 1644 K in boiling-refluxing Cs, but the obtained result was ony qualitative. In addition to the above-mentioned corrosion tests several others were reported. Keddy (8) did not observe any detectable changes after an 100 hours exposure of Mo to liquid Cs at 773 K. Harvey (9) did not notice corrosive effects after a 118 hours contact at 698 K. Petrick et al. (10) reported a corrosion-resistivity of Mo against Cs in a long term operation at 1273 K. Mo-rich alloys had an outstanding resistance in Cs vapour at 2144 K (13) and a good resistance at 1113 K (14). The corrosion products determined on metallic Mo were reported as MoO_3 (15) or Cs_2MoO_4 (16,20). According to Lindemer et al. (17) $Cs_6Mo_2O_9$ should be the most stable compound under experimental conditions. Inspite of all the reported facts it might be assumed that the presence of O in the system increases the apparent Mo solubility in liquid Cs and that the O activity at higher temperatures is relatively lower, which indicate an almost complete temperature independence of the solubility. According to theoretical predictions, however, the temperature dependence of the Mo solubility in absolutely pure liquid Cs should be very steep, as expressed in the equation by Brewer and Lamoreaux (18):

$$\log (soly/\text{mol \% Mo}) = 2.96 - 15327 \, (T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

The solubility calculated from Eq.(1) should practically be immeasureable, being for example $8 \cdot 10^{-14}$ and $4 \cdot 10^{-7}$ mol % Mo at 955 and 1645 K, respectively. The solubility values proposed in this asessment were selected from (1,2) and (3), bearing in mind that they were only valid at specific O levels.

A schematic phase diagram of the Mo-Cs system is analogue to that of the Mo-Li system (18).

Tentative solubility values of Mo in liquid Cs

T/K	$soly$/mol % Mo	O $concn$/mol %	source
573	$2 \cdot 10^{-3}$	$8 \cdot 10^{-2}$	(3)
873	$1 \cdot 10^{-3}$	$8 \cdot 10^{-2}$	(3)
1644	$1 \cdot 10^{-3}$	$1 \cdot 10^{-2}$	(1,2)

References

1. Tepper, F.; Greer, J. *US Air Force Rep. ASD-TDR-63-824*, Pt.I, 1963; *Rep. MSAR-63-61*, 1963.
2. Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-64-327*, 1964; abstracted in *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 323.
3. Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.
4. Sedelnikov, V.A.; Godneva, M.M. *Issledovanie Fiziko-Khimicheskikh Svoistv Soedinenii Redkikh Elementov*, Nauka, Leningrad, 1978, p. 56.
5. Holley, J.H.; Neff, G.R.; Weiler, F.B.; Winslow, P.M. *Electric Propulsion Development*, E. Stuhlinger, Ed., Academic Press, New York, 1963, p. 341.
6. Winslow, P.M. *Corrosion* 1965, *21*, 341; abstracted in *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 334.
7. Chandler, W.T.; Hoffman, N.J. *US Air Force Rep. ASD-TDR-62-965*, 1963; cited from 1.
8. Keddy, E.S. *US Atom.Ener.Comm. Rep. LAMS-2948*, 1963.
9. Harvey, R. *US Atom.Ener.Comm. Rep. MND-P-3009-1*, 1959.
10. Petrick, E.N.; Husman, O.K.; Szymanowski, H.W. *US Atom.Ener.Comm. Rep. CWR-700-10*, 1960.
11. Smith, R.G.; Hargreaves, F.; Mayo, G.T.J.; Thomas, A.G. *J. Nucl. Mater.* 1963, *10*, 191.

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Cesium; Cs; [7440-46-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
May 1988

CRITICAL EVALUATION: (continued)

References (continued)

12. Hargreaves, F.; Mayo, G.T.J.; Thomas, A.G. *J. Nucl. Mater.* 1966, *18*, 212.
13. DeMastry, J.A.; Griesenauer, N.M. *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 337; *Trans. Am. Nucl. Soc.* 1965, *8*, 17.
14. Romano, A.; Fleitman, A.; Klamut, C. *Alkali Metal Coolants*, IAEA, Vienna, 1967, p.663.
15. Hoffman, N.J.; Chandler, W.T. *Met. Soc. Conf.* 1966, *30*, 509.
16. Adamson, M.G.; Aitken, E.A. *Trans. Am. Nucl. Soc.* 1973, *17*, 195.
17. Lindemer, T.B.; Besmann, M.; Johnson, C.E. *J. Nucl. Mater.* 1981, *100*, 178.
18. Brewer, L.; Lamoreaux, R.H. *Atom. Ener. Rev., Spec. Issue no 7*, IAEA, Vienna, 1980, p. 195.
19. Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-66-280*, 1966.
20. Kohli, R. *Materials Behavior and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 345.

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Tepper, F.; Greer, J.

US Air Force Rep. ADS-TDR-63-824, Pt.I, 1963; *Rep. MSAR-63-61*, 1963.

VARIABLES:

One temperature: 1644 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The equilibrium concentration of Mo as the main component of a Mo-½Ti alloy in liquid Cs after a 10 and 100 hours equilibration at 2500 °F is $1.10{\cdot}10^{-2}$ and $1.2{\cdot}10^{-3}$ mass % Mo, respectively, or, as calculated by the compilers, $1.5{\cdot}10^{-2}$ and $1.7{\cdot}10^{-3}$ mol % Mo.
The authors (1), who applied the same technique but a different crucible material, reported a solubility of $2.5{\cdot}10^{-2}$ or $1.0{\cdot}10^{-2}$ mass % Mo after an equilibration time of 14 and 110 hours, respectively; the corresponding solubility values of $3.4{\cdot}10^{-3}$ and $1.4{\cdot}10^{-3}$ mol % Mo were calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Solubility measurements of Mo in Cs were performed by means of an inverted capsule technique, the bottom part of the capsule being made of Mo-Ti(½%), the upper part of alumina. The capsule was heated for 100 hours at 1644 K under vacuum, filled with Cs and welded in an Ar atmosphere at room temperature. After equilibration, the capsule was inverted and cooled in dry ice. The solidified Cs in the alumina part of the capsule was dissolved and the crucible was cleaned with HCl. Mo was analysed by a spectrographical method. An examination of the alumina part of the capsule revealed a complete permeation by a reaction with Cs.

Therefore, the sampling part of the crucible used for the second determination (1) was made of a Nb-Zr(1%) alloy, the applied technique being essentially the same.

SOURCE AND PURITY OF MATERIALS:

Mo source: containing 0.5 % Ti, $2.5{\cdot}10^{-2}$ % C, $3.3{\cdot}10^{-3}$ % N, $5.3{\cdot}10^{-3}$ % O.
Cs: 99.9+ % pure, supplied by Mine Safety Applicances Res., purified by Zr turnings as hot getter in stainless steel vessel, containing $2.8{\cdot}10^{-3}$ % C, $1.2\ 10^{-3}$ % O, $<2{\cdot}10^{-4}$ % N, $<1{\cdot}10^{-3}$ % Fe.
Ar: purified by hot and cold K-Na bubler; O and H_2O content monitored.
Alumina: high purity supplied by G.E. Lucalox, containing 0.02 % SiO2, 0.01-0.04 % Fe_2O_3, 0.1 % MgO, 0.02 % CaO, <0.02 % Na_2O.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 3 K.

REFERENCES:

1. Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-64-327*, 1964; abstracted in *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, 323-333.

COMPONENTS:

(1) Molybdenum; Mo; [7439-98-7]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S.

Zh. Prikl. Khim. 1974, *47*, 2177-2180.

VARIABLES:

Temperature: 323-873 K
O concentrations in Cs: 0.08 and 0.8 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Mo solubility in liquid Cs at various temperatures and O concentrations is reported.

t/°C	*soly*/mass % Mo	*soly*/mol % Mo[a]
50	$<1\cdot10^{-4}$	$<1.4\cdot10^{-4}$
100	$<1\cdot10^{-4}$	$<1.4\cdot10^{-4}$
150	$<1\cdot10^{-4}$	$<1.4\cdot10^{-4}$
200	$<1\cdot10^{-4}$	$<1.4\cdot10^{-4}$
300	$1.7\cdot10^{-3}$	$2.4\cdot10^{-3}$
600	$8\cdot10^{-4}$	$1.1\cdot10^{-3}$
300[b]	$1.0\cdot10^{-3}$	$1.4\cdot10^{-3}$

[a] as calculated by the compilers.
[b] Cs containing 0.8 mol % O.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A specimen of Mo metal was covered with Cs under vacuum and equilibrated for 120 hours in a glass ampoule. The O content in Cs was increased by means of a controlled decomposition of a $KClO_4$-MnO_2 mixture. The ampoule glass did not undergo visible changes. Cs was dissolved in H_2O and volumetrically determined in the resultant hydrogen. An aliquot of the solution was treated with an acid. The remaining part was treated with HCl and morina solution in butanol. The optical density of the Mo complex with morina in the butanol phase was colorimetrically measured (1).

SOURCE AND PURITY OF MATERIALS:

Mo: containing 0.035 % Si, 0.89 % Fe.
Cs: 98-99 % pure metal, vacuum distilled, finally containing <0.01 % O, <1.5 % Rb as main impurities.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

(1) Godneva, M.M.; Vodyannikova, R.D. *Zh. Anal. Khim.* 1965, *20*, 831-6; translated in *J. Anal. Chem. USSR* 1965, *20*, 905-909.

COMPONENTS:

(1) Tungsten; W; [7440-33-7]

(2) Lithium; Li; [7439-93-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
May 1991

CRITICAL EVALUATION:

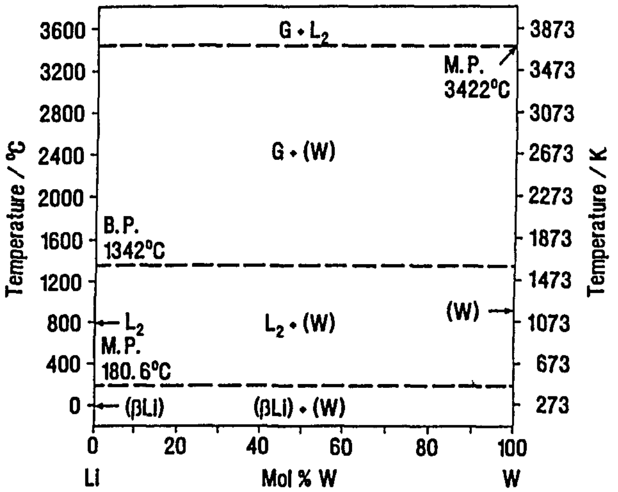
As W is the most refractory metallic element and a formation of an intermetallic compound between W and Li was not reported in literature, the solubility of W in liquid Li is expected to be extremely low as quantitatively predicted by Kuzin et al. (1) in form of the equation:

$$\log(soly/\text{mol \% W}) = 4.614 - 14200\ (T/\text{K})^{-1}$$

The experimental result of $9\cdot10^{-6}$ mol % W obtained by Eichelberger et al. (2) at 1475 K corresponds well with the prediction of (1). However, further results gained at 1675 and 1883 K indicate an independence of the solubility versus temperature, which is in contradiction with the equation. The applied materials were of very high purity, but the equilibration time might have been too short and traces of nonmetallic impurities caused the apparent solubility, which was independent on temperature.
The results of (2) were not confirmed by other determinations. Jesseman et al. determined the solubility to be as high as $4\cdot10^{-3}$ mol % W in an early study (3), whereas DeMastry (4) measured as much as $1.6\cdot10^{-2}$ mol % W at 1811 K. The overestimated results (3,4) might be explained by a reaction of W with components of the applied Fe (3) or Mo, Ti, Zr (4) containers. The presence of N probably influences the solubility of W in liquid Li, while an influence of O which was suggested by (5) seems to be improbable, since the free energy of formation of Li_2O is very low.
On the other hand, these high solubility data (3,4) were not confirmed in long operation corrosion tests performed by DeMastry and Griesenauer at 1644 K (4), Busse et al. at 1873 K (6), Hoffman at 1089 K (7), Melnikov et al. at 1773 K (8), and Wilhelm at 1273 K (9). W always showed a perfect resistance against liquid and gaseous Li. The W solubility calculated from the regular solution solubility parameter (6) is $2\cdot10^{-8}$ mol % W at 1873, which is a much lower value than in (1).
It seems that the presence of W in the system influences the solubility equilibrium. This phenomenon can also be observed for N or C (or both) present in other transition elements. Barker (10) did not mention an eventual reaction between N, C with W in Li. Addison (11) predicted a formation of Li_9WN_5. The phenomenon needs further investigation to be clarified. A schematic W-Li phase diagram is presented in the figure.

Tentative (t) and doubtful (d) solubility values of W in liquid Li containing ~ 10^{-3} mol % N

T/K	$soly$/mol % W	source	remarks
1473	$1\ 10^{-5}$ (t)	(2)	
1873	$1\cdot10^{-5}$ (d)	(2)	at constrained pressure



References

1. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113.
2. Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969; *Rep. AI-68-110*, 1968.
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4. DeMastry, J.A. *Nucl. Appl.* 1967, *3*, 127; abstracted in DeMastry, J.A.; Griesenauer, N.M. *Trans. Am. Nucl. Soc.* 1965, *8*, 17, 391.
5. Sangster, J.; Pelton, A.D. *J. Phase Equil.* 1991, *12*, 203.
6. Busse, C.A.; Geiger, F.; Pötzschke, M.; Quataert, D. *Thermionic Conversion Specialist Conference*, Houston, 1966, p. 149.
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8. Melnikov, M.V.; Larikov, L.N.; Cherepin, V.T.; Belkov, V.M.; Vasilev, M.A.; Ivashchenko, Yu. N.; Isaichev, V.I.; Maksimenko, E.A. *Fiz.-Khim.Mekh.Mater.* 1971, 7, no. 3, 90.
9. Wilhelm, C. *Mater. Design. Engin.* 1963, *58*, 97.
10. Barker, M.G.; *Rev. Int. Hautes Temp. Refract.*, 1979, *16*, 237.
11. Addison, C.C. *The Chemistry of the Liquid Alkali Metals*, Wiley, Chichester, 1984, p. 76.

COMPONENTS:

(1) Tungsten; W; [7440-33-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P.
US Atom.Ener.Comm. Rep. NEPA-1465, 1950.

VARIABLES:

One temperature: 989 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubility of W in liquid Li at 716 °C was determined to be 0.11 and 0.10 mass % W. The mean value calculated by the compilers is $4 \cdot 10^{-3}$ mol % W. W strips were completely disintegrated at higher temperatures. According to the compilers the possible formation of W-Fe compounds in the system seems to be of rather marginal effect on the evaluation of the solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Strips of W sheet were placed in an Armco Fe capsule. The capsule was loaded with Li in an Ar dry box, degassed and Li was melted in a pot furnace. After welding the capsule was heated in a vacuum furnace with stainless steel plates the average temperatures of which had been assigned. The temperature was maintained for a period of 24 hours and the furnace was then air-cooled. The capsule was weighed and opened. The solidified sample was leached out of the capsule with distilled water, and the W remaining undissolved was removed with the capsule, dried, and weighed as the tare to determine the amount of Li solution in the capsule. The leached material was filtrated, and the W content of the residue analysed by means of a spectrographic method.

SOURCE AND PURITY OF MATERIALS:

W: unspecified.
Li: containing 0.24 % O, <0.02 % N, <0.005 % Na; further contamination by larger amounts of O and N in the dry box during loading operation possible.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: precision ± 5 % (compilers).
Temperature: precision ± 20 K.

REFERENCES:

COMPONENTS:

(1) Tungsten; W; [7440-33-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G.

NASA Rep. CR-1371, 1969; *Rep. AI-68-110*, 1969.

VARIABLES:

Temperature: 1475-1883 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of W in liquid Li at various temperatures was determined.

t/°C	*soly*/mass % W	*soly*/mol % W [a]
1202	$2.5 \cdot 10^{-4}$	$9 \cdot 10^{-6}$
1400	$<1 \cdot 10^{-4}$	$<4 \cdot 10^{-6}$
1610	$2.5 \cdot 10^{-4}$	$9 \cdot 10^{-6}$

[a] calculated by the compilers.

The authors assume the solubility of W in Li to be at $2 \cdot 10^{-4}$ mol % in this temperature range.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A W test crucible and a Nb-Zr(1%) sample collector were placed in a Mo capsule, degassed in high vacuum at 1993 K, and cooled in an Ar atmosphere. The parts were finally outgassed at 623 K to remove Ar. After filling Li into the crucible, the collector with its cup were installed and the capsule was sealed under vacuum. It was heated to the desired test temperature and equilibrated for 4 hours. Finally, the capsule was removed from the furnace, the cup being then at the bottom. This way the liquid Li in the capsule was transferred to the collector. After cooling, the capsule was cracked open, the collector separated, and a Li sample was melted from the collector into a special glassware. The solidified Li was submerged in H_2O in an Ar atmosphere. The collector was rinsed with H_2O and hot 2 mol dm^{-3} HNO_3. Both solutions were joined and W was spectro-photometrically determined as a complex with toluene-3,4 dithiol in $CHCl_3$ being extracted from the aqueous solution of strong HCl with additions of $Ti_2(SO_4)_3$.

SOURCE AND PURITY OF MATERIALS:

W: vapour deposited, supplied by Atomics International, WF_6 was decomposed to W in a controlled reaction.
Nb-Zr(1%): supplied by Union Carbide, containing $7.0 \cdot 10^{-3}$ % C, $5.5 \cdot 10^{-3}$ % O, $4 \cdot 10^{-4}$ % H, $5.4 \cdot 10^{-3}$ % N.
Li: supplied by General Electric Corp., hot trapped with a Zr foil getter for 126 hours at 1093 K and distilled, containing $4.4 \cdot 10^{-3}$ % C, $1.3 \cdot 10^{-3}$ % N, $3.3 \cdot 10^{-3}$ % O, $<5.0 \cdot 10^{-3}$ % other metallic elements (each).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Tungsten; W; [7440-33-7]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

DeMastry, J.A.

Nucl. Appl. 1967, *3*, 127-134.

VARIABLES:

One temperature: 1811 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The equilibrium concentration of W in liquid Li at 2800 °F is 0.43 mass % W, or $1.6 \cdot 10^{-2}$ mol % W, as recalculated by the compilers.

An attack of Li on W was not observed at 2500 °F, whereas at 3000 °F a slight dissolution of the surface could be noticed. The capsule components, Ti and Zr, showed a stronger interaction with W than with Mo (1), which might have increased the apparent dissolution of W (compilers). W-rich alloys show a similar behaviour in liquid Li as pure W.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A W specimen in form of a small cylinder was weighed, measured and visually as well as photo-micrographically examined. A capsule made of a Mo-Ti(½%)-Zr(0.08%) alloy was loaded with 3 to 6 specimens, a corresponding amount of Li and finally welded. All operations were performed in a He glove box. The capsule was evacuated and leak-tested. It was heated in a vacuum furnace for 1000 hours. After removal from the furnace, the capsule was opened in the glove box. The specimens were removed, weighed, measured, and visually as well as photo-micrographically examined. The Li sample, taken from the capsule, was chemically analysed by a method which was not further specified.

SOURCE AND PURITY OF MATERIALS:

W: containing $5.0 \cdot 10^{-3}$ % C, $2.6 \cdot 10^{-4}$ % O, $1.1 \cdot 10^{-4}$ % N, $4 \cdot 10^{-5}$ % H, $2.0 \cdot 10^{-2}$ % Mo, $5.0 \cdot 10^{-3}$ % Si, $3.0 \cdot 10^{-3}$ % Fe, $2.0 \cdot 10^{-3}$ % Ni, $1.0 \cdot 10^{-3}$ % Cr.

Li: gettered with a Ti-Zr alloy at 739 K for 4 hours, containing $1.2 \cdot 10^{-2}$ % C, $2 \cdot 10^{-3}$ % N, $5.0 \cdot 10^{-2}$ Zr, $1.0 \cdot 10^{-2}$ % Ti, $5 \cdot 10^{-3}$ % Fe, $1 \cdot 10^{-3}$ % Ni, Cr, Cu.

He: US Atom.Ener.Comm. grade purity product, further bubbled through a liquid N trap, finally containing $3.0 \cdot 10^{-4}$ % O_2.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Niessen, A.K.; deBoer, F.R.; Boom, R.; de Châtel, P.F.; Mattens, W.C.H.; Miedema, A.R. *CALPHAD* 1983, *7*, 51-70.

COMPONENTS:	EVALUATOR:
(1) Tungsten; W; [7440-33-7] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1992

CRITICAL EVALUATION:

Solubility determinations in the W-Na system could not be found in literature. Aleksandrov and Dalakova (1) did not find any dissolution of W in liquid Na after 1 hour equilibration of the metals at 973 to 1023 K. The detectivity of the applied spectral analysis was not specified. According to earlier estimations (2,3) based on the regular solution model as well as more recent quantitative predictions (4) the solubility of W in liquid Na should be extremely low. The equation formulated in (4):

$$\log(soly/\text{mol \% W}) = 5.838 - 17340(T/\text{K})^{-1}$$

may be used to calculate the solubility, a value of $3 \cdot 10^{-12}$ mol % W was the result at 1000 K. Sangster and Pelton (5) estimated the solubility to be between the experimental values of the solubility of W in liquid Li and in liquid K. This estimation may be realistic as far as the apparent solubility is considered.

Corrosion tests of W in liquid Na (3,6-8) seem to confirm a very low solubility. However, the a presence of O in the W-Na system might increase the apparent W solubility in Na by several orders of magnitude, which can be deduced from its similarity to other transition metals.

Barker and Morris (9,10) identified the corrosion products which form at the interface of W and liquid Na. Metallic W was covered with Na_6WO_6 at static conditions at a temperature of 823, if the O content in Na was 0.45-1.5 mol % O. The ternary oxide Na_3WO_4 was observed as a surface corrosion film at dynamic tests at 533 to 744 K and an O content in Na of $7.2 \cdot 10^{-3}$ mol %. The W-Na phase diagram should be analogue to that shown for the W-Li system except the differing melting points of the alkali metals.

References

1. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.
2. McIntosh, A.B.; Broadley, J.S.; Bagley, K.Q. *UK Atom.Ener.Author. Rep. R&DB(C)-TN-31*, 1953.
3. Eldread, V.W. *Atom.Ener.Res.Establ. Rep. X/R-1806*, 1955.
4. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroenia Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113.
5. Sangster, J.; Pelton, A.D. *J. Phase Equil.* 1991, *12*, 204.
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8. Wilhelm, C. *Mater. Design Engin.* 1963, *58*, 97.
9. Barker, M.G.; Morris, C.W. *J. Less-Common Met.* 1976, *44*, 169.
10. Barker, M.G. *Rev. Inter. Hautes Temp. Refract.* 1979, *16*, 237.

COMPONENTS:	EVALUATOR:
(1) Tungsten; W; [7440-33-7] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1988

CRITICAL EVALUATION:

The two sets of experimental data concerning solubility determinations of W in liquid K were in only in rough agreement. Eichelberger et al. (1) performed three experiments in the temperature range 1476 to 1856 K at constrain pressure. The results, which were irregularly scattered between $8.4 \cdot 10^{-4}$ and $1.6 \cdot 10^{-3}$ mol % W, did not indicate any temperature dependence.

The results obtained by Stecura (2,3) are much more convincing, though the applied material was not of that high purity as in (1). The solubilities reported by Stecura almost smoothly increased, with a scatter of ± 10 %, from $4.4 \cdot 10^{-4}$ to $2.5 \cdot 10^{-3}$ mol % W in the temperature range of 1055-1328 K. An extrapolation of these data to the temperature range of (1) according to the fitting equation given in the Data Sheet yields values being roughly one order of magnitude higher than reported in (1).

The results of (2,3) are to be preferred in spite of their apparent character at an O level of $7 \cdot 10^{-4}$ mol % and in spite of the fact that the results of (1) are closer to a hypothetic W solubility in liquid K due to the application of pure metals.

DiStefano and DeVan (4) tested the corrosion resistance of the W-Re(26%) alloy in liquid K containing about $4.8 \cdot 10^{-3}$ mol % O for 5000 hours at 1523 K. The test did not reveal the presence of any measurable amount of W in K after the operation. However, the sensitivity of the applied analytical method is not specified.

Aleksandrov and Dalakova (5) did also not observe any dissolution of W in liquid K after an equilibration of 1 hour at 873-923 K. They used a spectral analytisis of unspecified detectivity.

An elevation of the apparent solubility of W in liquid K might be due to a formation of thermally stable compounds of the type $K_xW_yO_z$ (2-4) in the system, however, such a phase was not identified.

The W-K phase diagram should be similar to the schematic W-Li diagram; see the W-Li system.

Doubtful values of the solubility of W in liquid K

T/K	*soly*/mol % W	source	remarks
1073	$6 \cdot 10^{-4}$	(2,3)	at constrained pressure
1173	$1 \cdot 10^{-3}$	(2,3)	at constrained pressure
1273	$2 \cdot 10^{-3}$	(2,3)	at constrained pressure

References

1. Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969; *Rep. AI-68-110*, 1969.
2. Stecura, S. *Corrosion by Liquid Metals*, J.E. Draley, J.R. Weeks, Eds., Plenum, New York, 1970, 601.
3. Stecura, S. *NASA Rep. TN-D-5504*, 1969.
4. DiStefano, J.R.; DeVan, J.H. *Nucl. Appl. Technol.*, 1970, *8*, 29; abstracted in *US Atom. Ener.Comm. Rep. ORNL-4350*, 1969, 103.
5. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Tungsten; W; [7440-33-7] (2) Potassium; K; [7440-09-7]	Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969; *Rep. AI-68-110*, 1968.
VARIABLES:	**PREPARED BY:**
Temperature: 1476-1856 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubility of W in liquid K was determined at various temperatures.

t/°C	*soly*/mass % W	*soly*/mol % W [a]
1203	$7.6 \cdot 10^{-3}$	$1.6 \cdot 10^{-3}$
1391	$4.0 \cdot 10^{-3}$	$8.4 \cdot 10^{-4}$
1583	$6.1 \cdot 10^{-3}$	$1.3 \cdot 10^{-3}$

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A W test crucible and a Nb-Zr(1%) sample collector were placed in a Mo capsule, cleaned, etched, dried, degassed under high vacuum at 1993 K, and cooled in an Ar atmosphere. The parts were finally outgassed at 623 K. After filling K into the crucible, the capsule was sealed under vacuum. It was then heated to the desired test temperature and equilibrated for 4 hours. Finally, the capsule was removed from the furnace, so that the liquid K in the capsule was transferred to the collector. After cooling the capsule was cracked open, the collector separated, and a K sample was melted from the collector into a special glassware. The solidified K reacted with H_2O vapour in a He atmosphere, acidified with HCl and HF. K remaining in the collector was slowly dissolved in H_2O and rinsed with HCl. Both solutions were joined and W was spectrophotometrically determined as a complex with toluene-3,4 dithiol in $CHCl_3$ being extracted from the aqueous solution with additions of strong HCl and a catalytic amount of $Ti_2(SO_4)_3$.

SOURCE AND PURITY OF MATERIALS:

W: vapour deposited, prepared by Atomics International by controlled decomposition of WF_6.
Nb-Zr(1%): supplied by Union Carbide, containing $7 \cdot 10^{-3}$ % C, $5.5 \cdot 10^{-3}$ % O, $4 \cdot 10^{-4}$ % H, $5.4 \cdot 10^{-3}$ % N.
K: containing $5 \cdot 10^{-4}$ % O, $<1 \cdot 10^{-3}$ % other elements (each).
Ar: nothing specified.
He: nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Tungsten; W; [7440-33-7]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Stecura, S.

NASA Rep. TN-D-5504, 1969.

VARIABLES:

Temperature: 1055-1328 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of W in liquid K was determined at various temperatures.

T/K	*soly*/mass % W	*soly*/mol % W [a]
1328	$1.25 \cdot 10^{-5}$, $1.15 \cdot 10^{-2}$	$2.6 \cdot 10^{-3}$, $2.4 \cdot 10^{-3}$
1287	$1.35 \cdot 10^{-2}$, $9.9 \cdot 10^{-3}$	$2.8 \cdot 10^{-3}$, $2.1 \cdot 10^{-3}$
1207	$8.0 \cdot 10^{-3}$, $6.5 \cdot 10^{-3}$	$1.7 \cdot 10^{-3}$, $1.4 \cdot 10^{-3}$
1182	$6.0 \cdot 10^{-3}$	$1.3 \cdot 10^{-3}$
1124	$4.4 \cdot 10^{-3}$, $3.4 \cdot 10^{-3}$	$9.2 \cdot 10^{-4}$, $7.1 \cdot 10^{-4}$
1069	$3.0 \cdot 10^{-3}$, $2.5 \cdot 10^{-3}$	$6.3 \cdot 10^{-4}$, $5.2 \cdot 10^{-4}$
1055	$2.1 \cdot 10^{-3}$	$4.4 \cdot 10^{-4}$

[a] calculated by the compilers.

The author formulated a fitting equation, which was confirmed by the compilers:

$$\log(soly/\text{mol \% W}) = (0.34 \pm 0.21) - (3851 \pm 243)\,(T/\text{K})^{-1}$$

The results were graphically presented in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A W crucible was cleaned using a detergent in an ultrasonic field and immersed in boiling 20 mass % KOH for 5 min. After rinsing with H_2O, it was etched in a solution of HNO_3+HF+H_2O (21:4:75) for 15 min at 333 K, rinsed again with H_2O and finally dried under vacuum. A Ni capsule was etched in HNO_3+H_3PO_4+H_2SO_4+CH_3COOH (3:1:1:5) for ½-1 min at 363 K. The crucible was filled with K in a vacuum chamber, the capsule was electron-beam welded. It was placed in a furnace and heated for 24 hours. The temperature was measured by means of a Pt/Pt-Rh(13%) thermocouple. On removal, the capsule was inverted and the K collected. After cooling to room temperature, the capsule was cut open. K was dissolved in butyl alcohol. The capsule was leached in HCl. Both solutions were combined and K converted to KCl by evaporation. W was determined by spectrophotometric analysis.

SOURCE AND PURITY OF MATERIALS:

W: 99.997 % pure, containing $4 \cdot 10^{-4}$ % C, $5 \cdot 10^{-4}$ % O, $3 \cdot 10^{-4}$ % N, $1 \cdot 10^{-4}$ % H, $<1 \cdot 10^{-2}$ % metallic elements (each).
K: 99.99 % pure, containing $<1.5 \cdot 10^{-3}$ % O, $2 \cdot 10^{-4}$ % N, $5 \cdot 10^{-4}$ % Rb, $<3 \cdot 10^{-3}$ % metallic elements (each).

ESTIMATED ERROR:

Solubility: analytical precision ± 10 %.
Temperature: accuracy ± 8 K; stability ± 4 K.

REFERENCES:

1. Stecura, S. *Corrosion by Liquid Metals*, J.E. Draley, J.R. Weeks, Eds., Plenum, N. Y., 1970, p. 601.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Tungsten; W; [7440-33-7] (2) Rubidium; Rb; [7440-17-7]	Young, P.F.; Arabian, R.W. *US Atom.Ener.Comm. Rep. AGN-8063*, 1962.
VARIABLES:	**PREPARED BY:**
Temperature: 1033 and 1200 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubility of W in liquid Rb at elevated pressure and 1400 and 1700 °F, with Haynes-25 alloy serving as the W source, was below the detection limit ($1.4 \cdot 10^{-3}$ mass % W) of the method used. The corresponding value of this limit is $6.5 \cdot 10^{-4}$ mol % W, as recalculated by the compilers. Further comments do not seem to be reasonable.

COMMENTS AND ADDITIONAL DATA:

The W-Rb phase diagram is unknown, it is, however, expected to be similar to the schematic phase diagram of the W-Li system.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A W sample (Haynes-25 alloy) was cleaned in HNO_3+H_2SO_4+HF+H_2O (2:2:1:5), the Ta capsule in a mixture of CH_3COOH+HNO_3+HF+H_2O (30: 10: 2.5: 37.5). Both parts were further rinsed with H_2O, alcohol and dried in air. The sample was placed in the capsule, which had been filled with Rb. The capsule was then sealed in an Ar atmosphere, flame sprayed with Al_2O_3 and heated at the selected temperature for 50 hours. On removal, the capsule was inverted, causing Rb with the dissolved W to flow into a sample cup. The cup was cooled to room temperature. After solidification the cup was cut open, and its content was analysed. The Rb sample was treated with anhydrous hexane, CH_3OH, H_2O, and finally HCl. The Ta cup was treated with aqua regia and the solution was added to the previous one. The resulting solution was taken to dryness. The W content of the solid residue was determined in the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

W of Haynes-25 alloy: supplied by Superior Tube Co., containing 15.28 % W, 49.65 % Co, 20.42 % Cr, 10.14 % Ni, 1.7 % Fe, 1.39 % Mn, 0.097 % C.
Rb: purified by filtration, gettered with a Ti-Zr alloy at 866 K, vacuum distilled. O content $(6-17) \cdot 10^{-4}$ %.
Ar: nothing specified.

ESTIMATED ERROR:

Solubility: detection limit $1.4 \cdot 10^{-3}$ % W.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Tungsten; W; [7440-33-7] (2) Cesium; Cs [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1988

CRITICAL EVALUATION:

Practical aspects of the compatibility of W with liquid and gaseous Cs have frequently been investigated (1-10). However, only one study concerning the solubility of W in liquid Cs was published (10). Godneva et al. (10) determined quite considerable amounts of W in Cs after equilibration at 323 to 573 K. The corresponding solubility values increased from $6.5 \cdot 10^{-4}$ to $9.0 \cdot 10^{-3}$ mol % W and the plot of log solubility/W versus reciprocal temperature was almost linear [see the fitting equation in the Data Sheet of (1)]. According to this equation the expected solubility values of W in liquid Cs at higher temperatures should surely be measurable, whereas many investigators did not observe any dissolution of W at 698 K (1), 1273 K (2), 773 K (4) and 1275 K (5). A certain dissolution or corrosion of W in liquid Cs was noticed in more recent experiments by Winslow (7) at 673 K, Chandler and Hoffman (3) at 1272 K, Sedelnikov and Godneva (9) at 1073 K and DeMastry and Griesenauer (8) at 2144 K in Cs vapour under constrained conditions. However, numerical data were not published in these studies.

The mentioned discrepancies might be due to an influence of the O content in the system, but an increase of the apparent W solubility in Cs containing 0.8 mol % O (10) could not really be observed. Cs which was not gettered did not attack the W-Re(26%) alloy even at 1973 K (6). As the phenomenon needs further investigation, the results of (10) might be regarded as doubtful.

The schematic W-Cs phase diagram is expected to be similar to that of the W-Li system.

Doubtful values of the W solubility in liquid Cs

T/K	*soly*/mol % W	source
323	$6 \cdot 10^{-4}$	(10)
373	$1.3 \cdot 10^{-3}$	(10), fitting equation
473	$4 \cdot 10^{-3}$	(10), fitting equation
573	$7 \cdot 10^{-3}$	(10), fitting equation

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8. DeMastry, J.A.; Griesenauer, N.M. *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 337; *Trans. Am. Nucl.Soc.* 1965, *8*, 17.
9. Sedelnikov, V.A.; Godneva, M.M. *Issledovanie Fiziko-Khimicheskikh Svoistv Soedinenii Redkikh Elementov*, Nauka, Leningrad, 1978, p. 56.
10. Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.

COMPONENTS:

(1) Tungsten; W; [7440-33-7]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S.

Zh. Prikl. Khim. 1974, *47*, 2177-2180.

VARIABLES:

Temperature: 323-573 K
O concentration in Cs: 0.08 and 0.8 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of W in liquid Cs at various temperatures and O concentrations was measured.

t/°C	*soly*/mass % W	*soly*/mol % W [a]
50	$9 \cdot 10^{-4}$	$6.5 \cdot 10^{-4}$
100	$2.1 \cdot 10^{-3}$	$1.5 \cdot 10^{-3}$
150	$2.5 \cdot 10^{-3}$	$1.8 \cdot 10^{-3}$
200	$4.1 \cdot 10^{-3}$	$3.0 \cdot 10^{-3}$
300	$1.26 \cdot 10^{-2}$	$9.0 \cdot 10^{-3}$
300 [b]	$1.04 \cdot 10^{-2}$	$7.5 \cdot 10^{-3}$

[a] as calculated by the compilers
[b] Cs containing 0.1 mass % O; for the rest of the experiments the O content was below 0.01 mass % O

The data were fitted to the solubility equation by the compilers:

$$\log(soly/\text{mol \% W}) = -0.753 - 797(T/\text{K})^{-1} \qquad r = 0.976$$

The temperature coefficient is anomally low, probably due to the influence of O on the apparent solubility.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A W wire specimen was flooded with Cs under vacuum and equilibrated for 120 hours in a glass ampoule. The O content in Cs was increased by means of a controlled decomposition of a $KClO_4$-MnO_2 mixture. The ampoule glass did not undergo visible changes. Cs was dissolved in H_2O and its amount was determined by titration of a portion of the resulting solution with an acid. The remaining part was colorimetrically analysed to determine the W content.

SOURCE AND PURITY OF MATERIALS:

W: 99.85 % pure, containing 0.02 % S, 0.02 % Ni, 0.03 % Mo.
Cs: 98-99 % pure metal, vacuum distilled, finally containing <0.01 % O, <1.5 % Rb as main impurities.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Manganese; Mn; [7439-96-5] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1992

CRITICAL EVALUATION:

Obinata et al. (1) performed comprehensive investigations on Mn-Li alloys and determined Mn solubilities in liquid Li as well as the solubility gap of both metals in the liquid state. The results are consistent with the general image of the system. A contamination of the metals by N and O is not known. The Mn-Li samples may contain traces of Cu from the mould used for their preparation. The β Mn solubility in the temperature range 1073-1526 K may be expressed by the equation (as calculated by the compilers):

$$\log (soly/\text{mol \% } \beta\,\text{Mn}) = 0.666 - 1723\,(T/\text{K})^{-1} \quad r = 0.987 \qquad \text{Eq.(1)}$$

Lyublinskii et al. (2,3) reported solubility determinations in the temperature range 823 - 998 K. They used X-ray absorption spectroscopy analysis. However, they did not present single results, only the fitting equation:

$$\log (soly/\text{mol \% } \alpha\,\text{Mn}) = (2.582 \pm 0.198) - (3900 \pm 517)\,(T/\text{K})^{-1} \qquad \text{Eq.(2)}$$

The authors also published theoretical predictions of the solubilities of α and β Mn in liquid Li in the forms:

$$\log (soly/\text{mol \% } \alpha\,\text{Mn}) = 2.643 - 3600(T/\text{K})^{-1} \qquad \text{Eq.(3)}$$

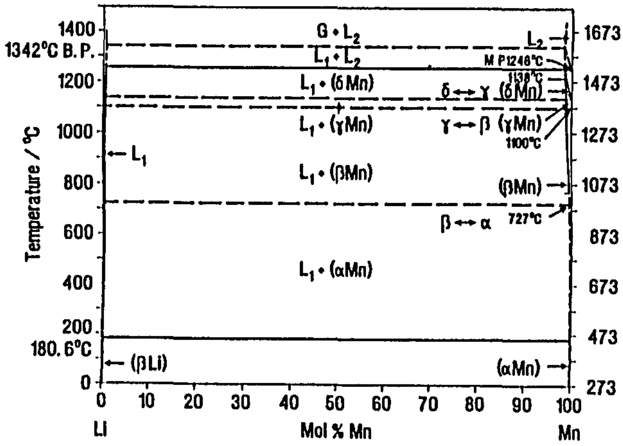
$$\log (soly/\text{mol \% } \beta\,\text{Mn}) = 2.521 - 3482\,(T/\text{K})^{-1} \qquad \text{Eq.(4)}$$

Concerning α Mn, an agreement of the theoretical prediction Eq.(3) and practical determinations Eq.(2) may be noticed, but Eq.(4) and Eq.(1) disagree in concern with the solubility of β Mn in Li. The metals used in (2,3) were of higher purity and the direct analytical method was of higher precision than in (1). The studies (2,3) are therefore to be preferred. If the Mn-Li system is contaminated with N, the formation of the compound Li_7MnN_4 (3), which has a higher solubility in Li than the metal, may disturb the Mn-Li equilibrium. This effect was, however, not studied in detail. The solubility of Mn from several types of steels is reported in (3), it was lower than from unalloyed Mn in all cases.

The Mn-Li phase diagram, based on paper (1), was reported in a corrected form in (4) and is redrawn in the figure. Mn transformation may be noticed at 1000, 1373, and 1411 K, possibly causing a temperature dependence of the solubility which may not be linear. The extrapolated solubility data of (1) and (2,3) cross at about 1000 K, which fact may reflect the α Mn ↔ β Mn transformation.

Tentative (t) and doubtful values of the Mn solubility in liquid Li

T/K	$soly$/mol % Mn	solute	source
873	$1.3 \cdot 10^{-2}$ (t)	α Mn	(2,3)
973	$4 \cdot 10^{-2}$ (t)	α Mn	(2,3)
1073	0.12 (d)	β Mn	(1)
1173	0.15 (d)	β Mn	(1)
1273	0.20 (d)	β Mn	(1)
1373	0.26 (d)	γ Mn	(1)
1473	0.31 (d)	δ Mn	(1)
1526	0.34 (d) monotectic		(1)



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4. *Binary Alloys Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Mater., Materials Park, 1986, p. 2446.

COMPONENTS:

(1) Manganese; Mn; [7439-96-5]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Obinata, I.; Takeuchi, Y.; Kurihara, K; Watanabe, M.

Metall 1965, *19*, 21-35; *Nippon Kinzoku Gakkaishi* 1964, *28*, 562-568.

VARIABLES:

Temperature: 1073-1526 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of β Mn in liquid Li at various temperatures is reported.

t/°C	*time*/hours	*soly*/mass %	*soly*/mol % [a]
800	20	0.95	0.12
900	10	1.21	0.15
1000	4	1.57	0.20
1100	2	2.10	0.26
1200	1	2.75	0.35
1253	-	2.51±0.24	0.32
1253[b]	-	99.85±0.05	98.83

[a] calculated by the compilers
[b] end of the miscibility gap at monotectic temperature.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Mn-alloys of various compositions were prepared by tungsten arc melting in a Cu mould under Ar atmosphere. The procedure was repeated several times to homogenize the sample. Thermal analyses of the samples as well as solubility determinations were performed in a W crucible placed in an airtight steel capsule. The samples were equilibrated at a selected temperature for a given time and then quickly quenched in H_2O. The Li-rich and Mn-rich phases were mechanically separated. A part of the sample phase was dipped in H_2O and the Mn remaining undissolved was analysed by means of a further unspecified method.

SOURCE AND PURITY OF MATERIALS:

Mn: 99.9 % pure (electrolytic).
Li: 99.9 pure.

ESTIMATED ERROR:

Solubility: precision ± 10 %.
Temperature: precision ± 5 K.

REFERENCES:

COMPONENTS:

(1) Manganese; Mn; [7439-96-5]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Gryaznov, G.M.; Evtikhin, V.A.; Zavyalskii, L.P.; Kosukhin, A.Ya.; Lyublinskii, I.E. *Materialovedenie Zhidkometallicheskikh Sistem Termoyadernykh Reaktorov*, Energoatomizdat, Moskva, 1989, p. 50-54, 82.

VARIABLES:

Temperature: 798-998 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of α Mn in liquid Li was determined between 550 and 700 °C. The results were presented as a fitting equation:

$$\log(soly/\text{mol }\%\ \alpha\ \text{Mn}) = (2.58\pm0.20) - (3900\pm520)(T/\text{K}^{-1})$$

The solubility values which were read out of the plot by the compilers, $7\cdot10^{-3}$ and $4.5\cdot10^{-2}$ mol % Mn at 823 and 998 K, respectively, are in agreement with the solubility equation. The equation was reported in (1).

The solubility of Mn from steels was investigated by means of the same method and reported in graphic form. The values were read out from the figure and recalculated to mol % by the compilers.

Steel type	Mn content	Temperature /K						
	mass %	923	973	1073	1098	1123	1173	1198
16Kh12VMFBAR	0.73	$4.4\cdot10^{-4}$	$6.5\cdot10^{-4}$	-	$1.4\cdot10^{-3}$	$1.9\cdot10^{-3}$	$2.2\cdot10^{-3}$	-
KhN28VMAB	0.51	-	-	$1.3\cdot10^{-4}$	-	$3.4\cdot10^{-4}$	$4.0\cdot10^{-4}$	$6.5\cdot10^{-4}$
09Kh16N15M3B+V	0.30	-	-	-	-	-	$2.8\cdot10^{-4}$	-

The steels are ordered following the contents of Cr, 10.9, 20.6, and 15.7 mass %, while the contents of Ni were 0.65, 27.5, and 14.9 mass %. The authors conclude that the activity coefficient of Mn in Cr-Ni steels should be lower than in Cr steel, a fact that is reflected in the results of the solubility measurements.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A tube made of Mn was horizontally placed in a closed capsule (2). The tube was loaded with Li which was kept inside by means of surface forces. A V foil was additionally placed in the capsule which was equipped with windows of Be transparent to X-rays and not in contact with the solute Li. The capsule was heated in a He atmosphere in a furnace. The X-ray beam passed along the tube axis. The intensity of this beam was measured in a Soller's spectrometer from both sides of the capsule in relation to the K boundary of absorption of Mn. The amount of dissolved Mn was calculated by means of the corresponding formula.

SOURCE AND PURITY OF MATERIALS:

Mn: "high purity".
Li: 99.6 % pure (2); purified by means of distillation and equilibration with Nb-Zr(5%) and Y at 1248 K; finally containing $<1\cdot10^{-3}$ % N, $<5\cdot10^{-3}$ % O, and < 0.1 % metals (mainly Na).
He: nothing specified.

ESTIMATED ERROR:

Solubility: precision ± 10%.
Temperature: stability ± 0.1 K.

REFERENCES:

1. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113-118.
2. Beskorovainyi, N.M.; Vasilev, V.K.; Lyublinskii, I.E. *Metall. Metalloved. Chist. Met.* 1980, *14*, 135-148.

COMPONENTS:	**EVALUATOR:**
(1) Manganese; Mn; [7439-96-5] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1988

CRITICAL EVALUATION:

Claar (1) reported still unpublished determinations of the Mn solubility in liquid Na. The solubility was specified to be at a measurable level. According to the authors (2), the results of the Mn solubility first published were not satisfactory. The data casually scattered between $5.4 \cdot 10^{-5}$ and $8.3 \cdot 10^{-3}$ mol % Mn in the temperature range 873-1173 K. As experimental difficulties occurred when piercing Mn particles to the collector, the authors suggested to assume a Mn solubility of less than $2 \cdot 10^{-4}$ mol % Mn in this temperature range. Aleksandrov and Dalakova (10) observed a weak solubility of Mn in liquid K, they did not report quantitative results. More recent studies on the Mn solubility by (3,4) and (5,9) reveal a better agreement, though also a scatter of these data is observed. Stanaway and Thompson (3) established some important facts on the system in preliminary experiments at 621-923 K. Significant differences of the Mn solubility after an application of either different Mn or differently lined crucibles could not be observed. A difference of one order of magnitude was observed for filtered compared to unfiltered samples. A comparison of the results obtained with pure Ni filters and those gained after coating the surface with Mn indicates, that this procedure did not influence the Mn amount in Na. In the second of their publications (4), the experimental temperature range was extended to 1023 K and the influence of O was studied by adding Na_2O_2 or MnO_2. Solubility data gained in Na which had been purified by means of gettering with a U foil showed a large degree of scattering. The solubility in the purified Na was found in the same concentration range as in "as poured" purity. The temperature dependence of the Mn solubility in Na is not just smooth, if all values of single measurements are considered (4). The results of the Mn solubility in liquid Na of reactor grade purity were represented by the fitting equation, which had been confirmed by the evaluators:

$$\log (soly/\text{mol \% Mn}) = -(2.054 \pm 0.043) - (2017 \pm 213)\,(T/\text{K})^{-1} \qquad r = 0.96 \qquad \text{Eq.(1)}$$

The results reported by Periaswami et al. (5), who applied Na of a similar purity, were expressed by the following solubility equation, as confirmed by the evaluators:

$$\log (soly/\text{mol \% Mn}) = -0.738 - 2602\,(T/\text{K})^{-1} \qquad r = 0.825 \qquad \text{Eq.(2)}$$

The data of (4) and (5) are of the same order, though the difference between both equation coefficients in the temperature range of investigation (550-820 K) is obvious. Preliminary determinations performed by Periaswami et al. (9) and also published in (5) indicate a strong dependence of the Mn solubility on the O concentration in Na. These results are not as conclusive as those in (4). The data reported in (5) are closer to the predictive solubility equation developed by Kuzin et al. (6) based on the cellular model:

$$\log (soly/\text{mol \% Mn}) = 3.906 - 5402\,(T/\text{K})^{-1} \qquad \text{Eq.(3)}$$

However, the discrepancy between theory and experiments at temperatures above 800 K and below 600 K is fundamental. An influence of the α Mn $\leftrightarrow$ β Mn transformation on the solubility cannot be observed.

The presence of O in Na undoubtedly increases the Mn solubility. The solubility data could be extrapolated from the higher to the low O concentration, using the dependencies at 723 and 923 K in (4). Such a procedure would be unreliable, since MnO is a stable oxide in this system at O concentrations up to $3.6 \cdot 10^{-3}$ mol % in Na, as determined by Barker et al. (7). Subsequently, formation of $NaMnO_2$ and $Na_4Mn_2O_5$ occurs with increasing O level in Na. The data of Periaswami et al. (5) are preferred by the evaluators, since an addition of Mg to Na is expected to markedly decrease the O concentration in Na, but does not interact with Mn (8). On the other hand, the U applied as a getter by Stanaway and Thompson (4) may interact with Mn to form stable intermetallics (8), consequently disturbing the Mn-Na solubility equilibria. The results of (5) are presented in numerical, those of (3,4) were merely presented in graphical form.

The Mn-Na phase diagram is unknown, but seems to be similar to that reported for the Mn-K system.

Tentative values of the α Mn solubility in liquid Na

T/K	$soly$/mol % Mn	source
573	$5 \cdot 10^{-6}$	(5)
673	$2 \cdot 10^{-5}$	(5)
773	$8 \cdot 10^{-5}$	(5)
873	$2 \cdot 10^{-4}$	(5) extrapolated

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4. Stanaway, W.P.; Thompson, R. *Material Behaviour and Physical Chemstry in Liquid Metal Systems*, H.U. Borgstedt, Ed.; Plenum, N.Y., 1982, p. 421.
5. Periaswami, G.; Ganesan, V.; Rajan Babu, S.; Mathews, C.K. *Material Behaviour and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 411.
6. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113.
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COMPONENTS:

(1) Manganese; Mn; [7439-96-5]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.

US Atom.Ener.Comm. Rep. AI-AEC-12955, 1970.

VARIABLES:

Temperature: 873-1173 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Mn in liquid Na was determined at 4 temperatures.

t/°C	*soly*/mass % Mn	*soly*/mol % Mn[a]
600[b]	$4 \cdot 10^{-4}$	$1.7 \cdot 10^{-4}$
700[b]	$6.6 \cdot 10^{-3}$, $1.3 \cdot 10^{-4}$	$2.6 \cdot 10^{-3}$, $5.4 \cdot 10^{-5}$
800[c]	$2.01 \cdot 10^{-2}$	$8.4 \cdot 10^{-3}$
900[c]	$5.3 \cdot 10^{-4}$, $1.9 \cdot 10^{-4}$	$2.2 \cdot 10^{-4}$, $7.9 \cdot 10^{-5}$

[a] calculated by the compilers
[b] with α Mn
[c] with β Mn

The authors who observed a transfer of Mn to the collector expressed scepticism, as far as the values should represent the pure solubility of Mn in Na. They suggested to assume a Mn solubility of less than $5 \cdot 10^{-4}$ mass % ($2.1 \cdot 10^{-4}$ mol % Mn, as calculated by the compilers) in this temperature range.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Mn test crucible was cleaned with a SiC grit blast and an acetone wash. The collector was fabricated of a Nb-Zr (1%) alloy. The crucible-collector assembly was degassed for 2 hours at 623 K. The crucible was filled with Na and the assembly was sealed by welding under high vacuum. The capsule enclosing the assembly was equilibrated for 6 hours in a purified Ar atmosphere glove box at the desired temperature. Finally the whole apparatus was inverted in order to cause a flow of Na into the collector part. The collector was opened after cooling and the Mn content in Na was analysed by means of atomic absorption spectroscopy.

SOURCE AND PURITY OF MATERIALS:

Mn: 99.96 % pure as electrolytic deposit supplied by American Potash & Chem. Corp., containing $4.0 \cdot 10^{-3}$ % C, $7.5 \cdot 10^{-4}$ % H, $3.0 \cdot 10^{-2}$ % S, $\leq 2.0 \cdot 10^{-3}$ % metallic elements (each).
Na: 99.996 % pure, containing $8 \cdot 10^{-4}$ % C, $(0.6\text{-}4) \cdot 10^{-4}$ % O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Manganese; Mn; [7439-96-5]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Stanaway, W.P.; Thompson, R.

US Dept.Ener. Rep. CONF-800401-P2, 1980, p. 18/54-61.

VARIABLES:

Temperature: 621-923 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The α Mn solubility in liquid Na at various temperatures was read off the figure and recalculated to mol % by the compilers.

T/K	*soly*/mol % Mn
621	$1.1\ 10^{-5}$
675	$7.4\cdot10^{-6}$, $8.6\cdot10^{-6}$, $1.03\cdot10^{-5}$
723	$1.7\cdot10^{-4}$ (2 results)[a]
823	$1.7\cdot10^{-5}$, $2.4\cdot10^{-5}$ [b], $3.4\cdot10^{-5}$ [b], $4.4\cdot10^{-5}$ [b], $5.3\cdot10^{-5}$, $2.2\cdot10^{-4}$ [a] (mean value of 3 results)
873	$1.3\cdot10^{-5}$, $1.8\cdot10^{-5}$
923	$1.8\cdot10^{-5}$, $4.9\cdot10^{-5}$, $7.1\cdot10^{-5}$ [b], $7.9\cdot10^{-5}$, $8.8\cdot10^{-5}$ [b], $1.3\cdot10^{-4}$, $9.6\cdot10^{-4}$ [a] (the mean value of 5 results was between $1.3\cdot10^{-4}$ and $2.6\cdot10^{-3}$)

[a] unfiltered samples
[b] filtered samples with unconditioned filters

The unmarked data are from filtered samples with unconditioned filters.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Three different types of Ni test crucibles were applied: (i) alumina covered with a Mn ingot, (ii) lined with a Mn(88 %)-Ni foil in presence of Mn, (iii) the same without Mn present. The crucibles were placed in a sealed capsule which incorporated a Mo hyperdermic piercing needle and a sintered Ni frit of 1.5μm pore size. The filters applied in some experiments were pretreated in Na at 923 K for 24 hours in presence of Mn. The equilibration time was probably 48 hours. Some samples were filtered. For the determination of the Mn solubility the sealed capsule technique was expended, so the sampling could be done by piercing the crucible lid and pipetting a sample into a separate sample crucible at equilibrium temperature. The sampling time was about 30 s, if a Ni filter was used, and was somewhat longer, if the preheated filter was applied. The Mn in the sample was probably separated from Na by means of distilling off. The analysis for Mn was performed by flameless atomic absorption spectroscopy.

SOURCE AND PURITY OF MATERIALS:

Mn: "pure".
Na: distilled twice; finally containing $(0.1\text{-}15)\cdot10^{-4}$ % O.

ESTIMATED ERROR:

Solubility: scatter of results of the filtered samples ± $2.1\cdot10^{-5}$ mol % Mn.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Manganese; Mn; [7439-96-5]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Periaswami, G.; Ganesan, V.; Rajan Babu, S.; Mathews, C.K.
Material Behaviour & Physical Chemistry in Liquid Metal Systems, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 411-420.

VARIABLES:

Temperature: 476-819 K
O concentration in Na: $1.9\cdot10^{-3}$ - $1.3\cdot10^{-2}$ mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The α Mn solubility in liquid Na containing higher amounts of O from experiments without filtering the saturated Na compared to experiments where the saturated Na had been filtered, as also reported in (1):

T/K	*O concn* in Na/mass %	*soly*/mass % Mn	*soly*/mol % Mn[a]
476	$1.32\cdot10^{-3}$	$1.0\cdot10^{-4}$	$4.2\cdot10^{-5}$
585	$2.66\cdot10^{-3}$	$2.8\cdot10^{-4}$	$1.17\cdot10^{-4}$
587	$3.90\cdot10^{-3}$	$5.4\cdot10^{-4}$	$2.2\cdot10^{-4}$
613	$8.75\cdot10^{-3}$	$2.9\cdot10^{-4}$	$1.21\cdot10^{-4}$
633	$4.30\cdot10^{-3}$	$6.0\cdot10^{-4}$	$2.5\cdot10^{-4}$
673	$3.10\cdot10^{-3}$	$4.2\cdot10^{-4}$	$1.76\cdot10^{-4}$
775	$5.30\cdot10^{-3}$	$1.00\cdot10^{-3}$	$4.2\cdot10^{-4}$
819	-	$1.05\cdot10^{-3}$	$4.4\cdot10^{-4}$

α Mn solubility data of loop experiments:

T/K	*soly*/mass % Mn	*soly*/mol % Mn [a]
581	$4.6\cdot10^{-5}$	$1.93\cdot10^{-5}$
641	$6.6\cdot10^{-5}$	$2.8\cdot10^{-5}$
686	$1.41\cdot10^{-4}$	$5.8\cdot10^{-5}$
712	$7.4\cdot10^{-5}$	$3.1\cdot10^{-5}$
744	$9.6\cdot10^{-5}$	$4.0\cdot10^{-5}$

α Mn solubility in liquid Na with low O concentration:

T/K	*soly*/mass % Mn	*soly*/mol % Mn[a]	*T*/K	*soly*/mass % Mn	*soly*/mol % Mn[a]
549	$7\cdot10^{-6}$	$2.9\cdot10^{-6}$	720	$2.22\cdot10^{-4}$	$9.3\cdot10^{-5}$
584	$1.1\cdot10^{-5}$	$4.6\cdot10^{-6}$	732	$5.4\cdot10^{-4}$	$2.3\cdot10^{-5}$
588	$1.3\cdot10^{-5}$	$5.4\cdot10^{-6}$	758	$8.9\cdot10^{-5}$	$3.7\cdot10^{-5}$
623	$4.8\cdot10^{-5}$	$2.0\cdot10^{-5}$	764	$9.4\cdot10^{-5}$	$3.9\cdot10^{-5}$
653	$3.9\cdot10^{-5}$	$1.63\cdot10^{-5}$	779	$2.25\cdot10^{-4}$	$9.4\cdot10^{-5}$
687	$1.50\cdot10^{-4}$	$6.3\cdot10^{-5}$	811	$3.50\cdot10^{-4}$	$1.46\cdot10^{-4}$
718	$2.22\cdot10^{-4}$	$9.3\cdot10^{-5}$	817	$2.22\cdot10^{-4}$	$9.3\cdot10^{-5}$

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Chips of Mn, Na, and Mg ribbons (0.7 mol %) were placed in a stainless steel crucible. The stainless steel capsule and the crucible were separated by means of a 5 μm pore size "porosint" filter. All operations were performed in an inert atmosphere glove box. The capsule was brought outside and pressurized with Ar to 1 kg cm^{-2} above atmospheric pressure. It was kept at the equilibration temperature for more than 24 hours, subsequently inverted and pressurized with Ar to 2 kg cm^{-2}. The filtered Na was collected in the Ta crucible. After cooling, the capsule was disassembled inside the glove box and the sample was removed. The sample was distilled under vacuum and the residue was dissolved in 6 mol dm^{-3} HCl-HNO_3 mixture. The Mn concentration of the solution was analysed by flameless atomic absorption spectroscopy.

Na samples, taken from a small isothermal loop by dippping a Ta crucible into Na and withdrawing it, were also analysed for O. The O content was then monitored by an electrochemical oxygen meter.

SOURCE AND PURITY OF MATERIALS:

Mn: "pure".
Na: "nuclear grade", gettered; expected O content $(1\text{-}2)\cdot10^{-4}$ %.
HCl: distilled from quartz.
HNO_3: distilled from quartz.

ESTIMATED ERROR:

Solubility: standard deviation for the entire procedure ± 12 %.
Temperature: precision ± 1 K.

REFERENCES:

1. Mathews, C.K.; Bhat, N.P.; Periaswami, G. *US Dept.Ener. Rep. CONF-800401-P2*, 1980, p. 16/22-29.

COMPONENTS:

(1) Manganese; Mn; [7439-96-5]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Stanaway, W.P.; Thompson, R.
Material Behaviour & Physical Chemistry in Liquid Metal Systems, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 421-427.

VARIABLES:

Temperature: 623-1023 K
O concentration in Na: 0.011-0.11 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The α Mn solubilities in liquid Na at various temperatures were read off the figure and recalculated to mol % Mn by the compilers.

T/K	*soly*/mol % Mn	*T*/K	*soly*/mol % Mn
623	$4.9 \cdot 10^{-6}$	823	$2.4 \cdot 10^{-5}$, $3.9 \cdot 10^{-5}$ [a]
673	$8.5 \cdot 10^{-6}$	873	$3.5 \cdot 10^{-5}$, $3.1 \cdot 10^{-5}$ [a]
723	$1.6 \cdot 10^{-5}$, $2.36 \cdot 10^{-5}$ [a]	923	$6.0 \cdot 10^{-5}$, $4.2 \cdot 10^{-4}$ [a]
773	$3.1 \cdot 10^{-5}$, $1.9 \cdot 10^{-4}$ [a]	973	$1.2 \cdot 10^{-4}$
		1023	$7.4 \cdot 10^{-5}$ [b]

[a] in Na gettered with a U foil [b] with β Mn

The α Mn solubility in O-rich Na at various temperatures as read out from the figure and recalculated to mol % Mn by the compilers.

t/°C	*O concn*/mol %	*soly*/mol % Mn [a]	*soly*/mol % Mn [b]
450	$1.0 \cdot 10^{-2}$	$2.1 \cdot 10^{-5}$	$1.7 \cdot 10^{-5}$
450	$1.1 \cdot 10^{-2}$	$4.6 \cdot 10^{-5}$	-
450	$2.3 \cdot 10^{-2}$	$1.9 \cdot 10^{-5}$	-
450	$2.5 \cdot 10^{-2}$	$3.0 \cdot 10^{-5}$	-
450	$3.2 \cdot 10^{-2}$	-	$5.4 \cdot 10^{-5}$
450	$5.0 \cdot 10^{-2}$	$3.1 \cdot 10^{-5}$, $6.9 \cdot 10^{-5}$	-
450	$5.4 \cdot 10^{-2}$	-	$4.2 \cdot 10^{-5}$
450	$6.2 \cdot 10^{-2}$	-	$8.4 \cdot 10^{-5}$
450	$9.7 \cdot 10^{-2}$	-	$7.3 \cdot 10^{-5}$
450	0.11	$6.9 \cdot 10^{-5}$	-
650	0.12	$6.7 \cdot 10^{-5}$	-
650	0.13	$8.9 \cdot 10^{-5}$	-
650	0.19	-	$5.1 \cdot 10^{-5}$
650	0.28	$1.5 \cdot 10^{-4}$	-
650	0.29	$2.9 \cdot 10^{-4}$	-
650	0.32	$1.1 \cdot 10^{-3}$	-
650	0.58	-	$2.1 \cdot 10^{-4}$
650	0.85	-	$2.1 \cdot 10^{-4}$

[a] O added as MnO_2 [b] O added as Na_2O_2

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ni can was degreased, cleaned and H_2 fired at 1023 K for 24 hours, H_2 was then pumped off under vacuum at 1023 K. An alumina crucible liner, cleaned in boiling aqua regia, was fitted inside the Ni can. A Mn ingot served as the Mn source, with MnO_2 or Na_2O_2 added as an O source as required. A strip of U foil served to reduce the O level. The crucible was then partly filled with Na dispensed from a large stainless steel pot through a Ni 1.5 μm pore size filter frit at 423 K. A Ni lid was welded onto the can. The can was heated at the desired temperature for 45 hours. All operations were performed in an Ar atmosphere. A Na sample was taken by piercing the can lid with a Mo needle and withdrawing it through the filter frit which was pretreated by immersion in Na containing Mn for 24 hours at 923 K.

The sample was collected in a weighed alumina crucible previously cleaned in boiling aqua regia and dissolved in CH_3OH. Mn was separated from the bulk Na by a wet chemical co-precipitation.

The precipitate was dissolved in concentrated HCl and the Mn content of the solution analysed by atomic absorption spectroscopy.

SOURCE AND PURITY OF MATERIALS:

Mn: unspecified.
Na: "reactor grade", containing $(1.5\text{-}2.0) \cdot 10^{-3}$ % O.
Ar: "high purity".
U: electrolytic.

ESTIMATED ERROR:

Solubility: results scatter from ± 10 % to one order of magnitude, dependent on temperature (compilers).
Temperature: nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Manganese; Mn; [7439-95-6] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1988

CRITICAL EVALUATION:

Schwarz (1) reported a Mn solubility in liquid K of $1.0 \cdot 10^{-4}$ mol % Mn at 373 K. Details of this analysis are not known. Aleksandrov and Dalakova (2) reported a weak solubility of Mn in liquid K at 873-923 K, they did not present quantitative data.

The schematic Mn-K phase diagram is shown in the figure, it can be assumed to be similar to that of the Mn-Li system.

References

1. Schwarz, N.F. *Liquid Metals Engineering and Technology*, BNES, London, 1985, *3*, 177.
2. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Manganese; Mn; [7439-95-6] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1988

CRITICAL EVALUATION:

Typical measurements of the Mn solubility in liquid Cs have not been performed. Keddy (1) did not observe a detectable Mn dissolution in Cs in a corrosion test at 773 K, but a darkening of a sample occurred. Holley et al. (2) reported a Mn content in liquid Cs of 0.02 mol %, if stainless steel containing Mn was equilibrated with Cs for 312 hours at 675 K. After a shorter contact of the metals of 48 hours at 672 K, Cs contained 0.012 mol % Mn. An increase of the Mn content in Cs from $2.5 \cdot 10^{-3}$ to 0.43 mol % Mn was observed after an 7000 hours storage at room temperature. The initial purity of Cs was 99.9 %, the final purity only 96 %, indicating a marked dissolution of the tested steel sample, which was probably due to the presence of O in Cs. Therefore a definite conclusion on the basis of these results cannot be drawn. As study (2) is not a solubility determination and further details are not described, the publication is not compiled.

The Mn-Cs phase diagram is not known, but it is assumed to be similar to that of the Mn-K phase diagram, the boiling point of Cs is at 944 K.

References

1. Keddy, E.S. *US Atom.Ener.Comm. Rep. LAMS-2948*, 1963.
2. Holley, J.H.; Neff, G.R.; Weiler, F.B.; Winslow, P.M. *Electric Propulsion Development*, E. Stuhlinger, Ed., Academic Press, New York, 1963, p. 341.

COMPONENTS:

(1) Manganese; Mn; [7439-96-5]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Young, P.F.; Arabian, R.W.

US Atom.Ener.Comm. Rep. AGN-8063, 1962.

VARIABLES:

Temperature: 1033 and 1200 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubility of β-Mn in liquid Rb is reported to be regarded as a conditional quantity, since the Mn source was the Haynes 25 alloy with a Mn content of 1.4 mass % Mn. As the Haynes 25 is a multicomponent system, it is very difficult to evaluate the interaction of the components and the Mn activity related to pure Mn.

t/°F	*T*/K	*soly*/mass % Mn	*soly*/mol % Mn[a]
1400	1033	$1\cdot10^{-4}$	$1.6\cdot10^{-4}$
1700	1200	$1\cdot10^{-4}$, $6\cdot10^{-4}$	$1.6\cdot10^{-4}$, $9\cdot10^{-4}$

[a] calculated by the compilers.

COMMENTS AND ADDITIONAL DATA:

The Mn-Rb phase diagram is unknown but is assumed to be similar to that shown for the Mn-K system.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A specimen and a sample made of a Mn containing alloy were degreased with acetone, pickled in a mixture of HNO3, H_2SO_4, HF, and H_2O (2:2:1:5), rinsed with H_2O and dried. The alloy sample was placed in a the capsule, which had been filled with Rb. The capsule was then sealed in an Ar atmosphere, flame sprayed with Al_2O_3 and heated at the selected temperature for 50 hours. Finally, the capsule was inverted, causing Rb with the dissolved Mn to flow into a sample cup. The cup was cooled to room temperature and cut open after solidification. Rb was treated with anhydrous hexane, CH_3OH, distilled H_2O, and finally HCl. The resulting solution was taken to dry. The Mn content of the solid residue was analysed in the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

Mn in Haynes 25: from Superior Tube Co., containing 1.39 % Mn, 49.65 % Co, 20.42 % Cr, 15.28 % W, 10.14 % Ni, 1.7 % Fe, 0.097 % C.
Rb: as in the Nb-Rb system, same report.

ESTIMATED ERROR:

Solubility: detection limit $1\cdot10^{-4}$ % Mn; precision of analysis ± 10 %.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Technetium; Tc; [7440-26-8] (2) Lithium; Li; [7439-93-2] or Sodium; Na; [7440-23-5] or Potassium; K; [7440-09-7] or Rubidium; Rb; [7440-17-7] or Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1988

CRITICAL EVALUATION:

Neither experimental work nor a prediction of the solubility of Tc in the liquid alkali metals, Li, Na, K, Rb, Cs, is available in literature. Since many of the physico-chemical properties of Tc are more similar to its heavier homologous element Re than to Mn, it might be assumed that the solubility values of Tc in the liquid alkali metals are slightly higher than those of Re. An analogue behaviour was observed for the pair Mo and W.

An influence of non-metallic impurities as O, H, or C on the solubility of Tc in the alkali metals is hardly predictable. It should be similar to the Re-alkali metal systems in which the influence of these elements is rather negligible.

The influence of the atomic size of the solute metal on the solubility of Tc is not known, a higher solubility in Li than in all heavier alkali metals might be assumed.

Phase diagrams of the systems of Tc with the alkali metals should be analogous to the predictive phase diagram of the Tc-Li system.

COMPONENTS:	EVALUATOR:
(1) Rhenium; Re; [7440-15-5] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

Eichelberger et al. (1) determined the Re solubility in liquid Li at 3 different temperatures in the range of 1475 and 1875 K. The solubility increased from $2.2\ 10^{-6}$ to $6.3 \cdot 10^{-6}$ mol % Re, however, the slope of logarithm of solubility versus reciprocal temperature seems to be too low.

DeMastry (2,3) observed a significant Re content of 0.015 mol % in Li when both metals had been equilibrated at 1932 K in a Mo capsule. A more detailed analysis (3) indicated that this concentration level (as well as the increased Mo concentration in Li) was mainly due to an interaction of Re and Mo forming Mo_2Re_3. A dissolution of the Mo-Re(50 %) alloy in Li under the same conditions was not observed. This is in agreement with previous observations and means that the Re solubility really has to be very low. The studies are not compiled, since experimental details were not reported.

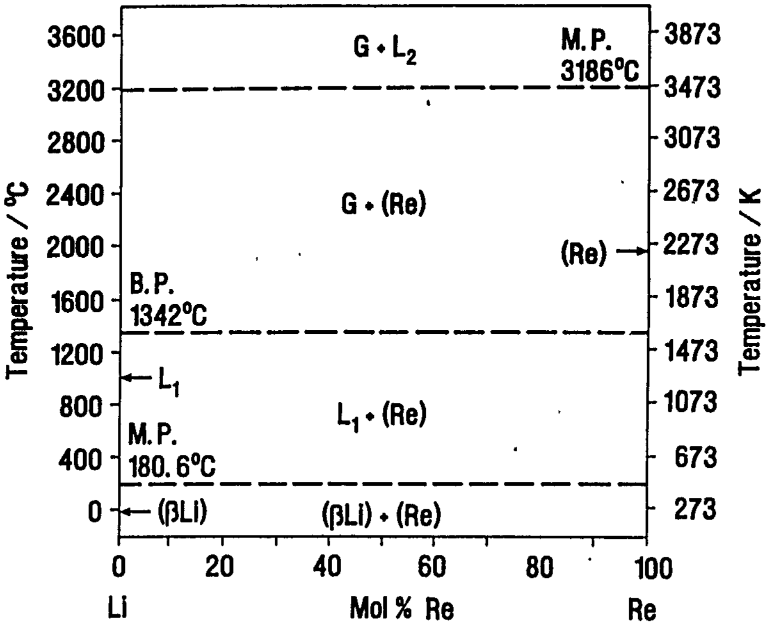
Hoffman (4) reported a good corrosion resistance of Re in liquid Li at 1089 K. Mo-Re alloys containing 13 and 41 mass % Re did not reveal corrosion due to dissolution in tests for 500 h duration at 1473 K (6).

The Re-Li phase diagram is shown in the figure. It seems that intermetallics are not formed in this system, which is in contradiction to results gained by von Grosse (5). Von Grosse observed a formation of ionic LiRe (lithium rhenide) hydrate in aqueous solution. However, the formation of an alloy of Li and Re could not be observed, due to a reaction between Li and the Pt crucible which was used as the container for this test.

An influence of nonmetallic impurities on the Re solubility in Li was not reported.

Tentative values of the Re solubility in liquid Li

T/K	*soly*/mol % Re	source	remarks
1473	$2 \cdot 10^{-6}$	(1)	
1673	$4 \cdot 10^{-6}$	(1)	at constrained pressure
1873	$6 \cdot 10^{-6}$	(1)	at constrained pressure



References

1. Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969; *Rep. AI-68-110*, 1968.
2. DeMastry, J.A. *Trans. Am. Nucl. Soc.* 1965, *8*, 391.
3. DeMastry, J.A. *Nucl. Appl.* 1967, *3*, 127.
4. Hoffman, E.E. *US Atom.Ener.Comm. Rep. ORNL-2924*, 1960.
5. von Grosse, A. *Z. Naturforsch.B* 1953, *8*, 533.
6. Flament, T.; Sannier, J. *Proc.4th Intern. Conf. on Liq.Met.Engin. and Technol.*, SFEA, Paris, 1988, p. 520/1-9.

COMPONENTS:

(1) Rhenium; Re; [7440-15-5]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G.

NASA Rep. CR-1371, 1969; *Rep. AI-68-110*, 1969.

VARIABLES:

Temperature: 1475-1875 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Re solubility in liquid Li at various temperatures is reported.

t/°C	*soly*/mass % Re	*soly*/mol % Re[a]
1202	$6 \cdot 10^{-5}$	$2.2 \cdot 10^{-6}$
1400	$1.1 \cdot 10^{-4}$	$4.1 \cdot 10^{-6}$
1602	$1.7 \cdot 10^{-4}$	$6.3 \cdot 10^{-6}$

[a] calculated by the compilers.

The compilers constructed the following fitting equation:

$$\log (soly/\text{mol \% Re}) = -3.49 - 3185 \, (T/\text{K})^{-1} \qquad r = 0.998$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Re test crucible and a Nb-Zr(1%) sample collector were placed in a Mo capsule. They were cleaned, etched, dried, and degassed under high vacuum at 1993 K and cooled in an Ar atmosphere. The parts were finally outgassed at 623 K to remove Ar. After filling Li into the crucible, the capsule assembly was sealed under vacuum. It was then heated to the desired test temperature and equilibrated for 4 hours. Finally, the capsule was removed from the furnace, the cup being at the bottom. This way the liquid Li in the capsule was transferred to the collector. After cooling, the capsule was cracked open and a Li sample was melted from the collector into a special glass ware. The solidified Li was submerged in H_2O in an Ar atmosphere. The collector was rinsed with H_2O and hot 2 mol·dm^{-3} HNO_3 and the resulting solution was added to the previous one. Re was reduced by $SnCl_2$ to form a furile dioxane complex, which was further extracted into $CHCl_3$ and spectrophotometrically determined.

SOURCE AND PURITY OF MATERIALS:

Re: supplied by Chase Brass and Copper Co., containing $4.2 \cdot 10^{-3}$ % Fe, $3.0 \cdot 10^{-3}$ % Mo, $<1 \cdot 10^{-4}$ % other metallic elements (each).
Nb-Zr(1%): supplied by Union Carbide Corp. Stellite Division, containing $7.0 \cdot 10^{-3}$ % C, $5.5 \cdot 10^{-3}$ % O, $4 \cdot 10^{-4}$ % H, $5.4 \cdot 10^{-3}$ % N.
Li: supplied by General Electric Corp., hot trapped with a Zr foil getter for 126 hours at 1093 K and distilled, containing $4.4 \cdot 10^{-3}$ % C, $1.3 \cdot 10^{-3}$ % N, $3.3 \cdot 10^{-3}$ % O, $<5 \cdot 10^{-3}$ % other metallic elements (each).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Rhenium;; Re; [7440-15-5] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1988

CRITICAL EVALUATION:

Quantitative data on the Re solubility in liquid Na are not available. Simons (1) reported that Re was corrosion resistant in liquid K-Na alloy after testing at 1144 K for 110 hours. Gukova and Ermolaev (2) did not observe an alloying of Re and Na. Aleksandrov and Dalakova (3) did not find any dissolution of Re in liquid Na after a contact of 1 h at 973-1023 K. They did not report the detection level of the spectral analysis used.
As the interaction of O with Re is comparatively weaker than that of d^2-d^6 transition metals, the influence of O on the Re solubility in Na also seems to be weaker. The Re solubility in Na is assumed to be lower than that determined for the Li solvent.
The Re-Na phase diagram should be similar to that shown for the Re-Li system.

References

1. Simons, E.M. *NASA Rep.TN-D-769*, 1961, p. 61.
2. Gukova, Yu. Ya.; Ermolaev, M.I. *Obshchie Zakonomernosti Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, p. 135.
3. Aleksandrov, B.N.; Dalakova, N.V. *Izv.Akad. Nauk SSSR, Met.* 1982, no.1, 133.

COMPONENTS:	EVALUATOR:
(1) Rhenium; Re; [7440-15-5] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1988

CRITICAL EVALUATION:

Eichelberger et al. (1) investigated the Re solubility in liquid K in the temperature range of 1473-1894 K. They determined the solubility to be below the detection limit of $2 \cdot 10^{-5}$ mol % Re and concluded that Re is the least soluble metal in liquid K as well as in other alkali metals.

Experiments performed by DiStefano and DeVan (2) confirmed this statement. A capsule made of the W-Re(26%) alloy was equilibrated with K containing $<7.2 \cdot 10^{-3}$ mol % O at 1523 K for 5000 hours. Any measurable amount of Re in K was traced after the operation. In experiments performed in a W-Re(26%) refluxing capsule the condenser contained a deposit of almost pure W and the boiler wall was enriched in Re, indicating that Re is less soluble than W at a temperature of about 1500 K. Re was not detected in liquid K by means of spectral analysis after an equilibration of 1 h at 873-923 K (4). As numerical data are not reported, the studies (2) and (4) are not compiled.

The Re-K phase diagram is not known. Gukova and Ermolaev (3) observed the formation of ReK_2 in a flame, which is in agreement with earlier theoretical predictions, but was not observed in attempts to alloy both metals (which did not show any mutual affinity).

Tentative value of the Re solubility in liquid K at elevated pressure

The Re solubility in liquid K at temperatures below 1873 K is below $2 \cdot 10^{-5}$ mol % Re.

References

1. Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G. *NASA Rep. CR-1371*, 1969; *Rep. AI-68-110*, 1968.
2. DiStefano, J.R.; DeVan, J.H. *Nucl. Appl. Technol.* 1970, *8*, 29; abstracted in *US Atom. Ener.Comm. Rep. ORNL-4350*, 1969, p. 103.
3. Gukova, Yu.Ya.; Ermolaev, M.I. *Obshchie Zakonomernosti v Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, p. 135.
4. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Rhenium; Re; [7440-15-5] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1988

CRITICAL EVALUATION:

Information concerning investigations of the Re-Rb system are not reported in literature. It may be predicted that the Re solubility in liquid Rb is not higher than that in liquid K, a predictive Re-Rb phase diagram should be similar to that of the Re-Li system.

COMPONENTS:	EVALUATOR:
(1) Rhenium; Re; [7440-15-5] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1988

CRITICAL EVALUATION:

Re metal was not attacked after a 1000 hours exposure to Cs vapour at 1810 K. The W-Re(26%) alloy was the most resistant one in a test series in which several alloys were exposed to Cs vapour at 2144 K for 100 hours. Only a slight surface dissolution was observed after 1000 hours (1). In another test performed at 1973 K non-gettered liquid Cs did not attack the alloy (2). Thus it may be concluded that Re is extremely weakly soluble in liquid Cs (3).

A predictive Re-Cs phase diagram should be similar to that of the Re-Li system.

References

1. DeMastry, J.A.; Griesenauer, N.M. *Trans. Am. Nucl. Soc.* 1965, *8*, 17; *US Atom. Ener.Comm. Rep. 650411*,
2. 1965, p. 337.
Simons, E.M.; as cited by Gurinsky, D.H.; Weeks, J.R.; Klamut, C.J.; Rosenblum, L.; DeVan, J.H. *Peaceful*
3. *Uses of Atomic Energy*, U.N.; New York, 1964, *9*, 550.
Berry, W.E. *Corrosion in Nuclear Applications*, Wiley, New York, 1971, p. 304.

COMPONENTS:

(1) Rhenium; Re; [7440-15-5]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.; Johnson, B.G.

NASA Rep. CR-1371, 1969; *Rep. AI-68-110*, 1968.

VARIABLES:

Temperature: 1473-1894 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Re in liquid K at 1200, 1415, and 1621°C was always below $1 \cdot 10^{-4}$ mass % Re or $2 \cdot 10^{-5}$ mol % Re (as calculated by the compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Re test crucible and a Nb-Zr(1%) sample collector were placed in a Mo capsule. They were cleaned, etched, dried, and degassed under high vacuum at 1993 K and cooled in an Ar atmosphere. The parts were finally outgassed at 623 K to remove Ar. After filling K into the crucible, the capsule assembly was sealed under vacuum. It was then heated to the desired test temperature and equilibrated for 4 hours. Finally, the capsule was removed from the furnace, the cup being at the bottom. This way the liquid K in the capsule was transferred to the collector. After cooling, the capsule was cracked open and a K sample was melted from the collector into a special glass ware. K reacted with H_2O vapour in a He atmosphere and was acidified with HCl and HF. K remaining in the collector was slowly dissolved in H_2O and rinsed with HCl. Both solutions were joined and Re was extracted into $CHCl_3$ as a tetraphenylarsonium complex. It was transferred into an aqueous solution as a furile dioxane complex, after a reduction by $SnCl_2$, and re-extracted into $CHCl_3$ for spectrophotometric determination.

SOURCE AND PURITY OF MATERIALS:

Re: supplied by Chase Brass and Copper Co., containing $4.2 \cdot 10^{-3}$ % Fe, $3.0 \cdot 10^{-3}$ % Mo, $1 \cdot 10^{-4}$ % other metallic elements (each).
K: containing $5 \cdot 10^{-4}$ % O, $1 \cdot 10^{-3}$ % other elements (each).
Ar: unspecified.
He: unspecified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Iron; Fe; [7439-89-6] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1991

CRITICAL EVALUATION:

Though the number of studies concerning the solubility of Fe in liquid Li is large, convincing results have only been obtained during the last years. First results on the compatibility of Fe and Li were somewhat inconclusive. The disintegration of a Fe crucible by boiling Li was reported (1), a measurable solubility of Li diffusing into solid Fe was, however, not observed by others (2,3). While one source (4) stated the possible alloying of the two metals, another one recommended Fe as the most resistive material for the preparation of Li alloys by direct alloying (5).

First determinations of the solubility of Fe in liquid Li were performed independently by Wilkinson and Yaggee (6) and Jesseman et al. (7). The apparent solubility of Fe from Armco Fe and some steels at 573 K was reported in (6). The results were scattered in the range of $8.1\cdot10^{-4}$ to $1.5\cdot10^{-2}$ mol % Fe and did not indicate any correlation with the Fe content in the equilibrated alloys. An addition of U to Li certainly decreased the content of non-metallic impurities, but as Fe and U tend to form intermetallics, the addition could have disturbed the investigated equilibria. The determinations of Jesseman et al. (7) were performed at temperatures between 697 and 1219 K. The results were dependent on the equilibration time (4-100 h), being lowest at 24 h. A decrease of the mean values of the solubility from $3\cdot10^{-2}$ to $1.5\cdot10^{-2}$ mol % Fe was observed with increasing temperature within this range, the results were roughly 10 times higher than many of those obtained in later years. It seems that the values were overstated due to impurities of the Li samples, even though the contents of N were specified to be below 0.01 mol % N.

Sand (8) determined the solubility in the temperature interval 468-1473 K. A rather weak temperature dependence of the solubility was observed at temperatures above 893 K, the results obtained at temperatures below 698 K being certainly overstated, though they were in agreement with (6). Sand detected that the apparent solubility was a few times higher, if the Fe samples were not annealed in H_2 or Li was contaminated. The equilibrium in the system was reported to be reached within a few minutes in that case.

The influence of N and O on the solubility of Fe in liquid Li was first considered by Bychkov et al. (9-11) who studied the Fe-Li equilibria in pure and O or N containing Li, using Fe or a stainless steel at temperatures of 1073 to 1473 K. As the solubility value at 1273 is only vague, the temperature dependence of the solubility (using most pure components) is only an approximation. It is, however significantly different from that of previous studies. The solubility was 10 times higher if pure Fe was used instead of stainless steel (17.3 mass % Fe) at temperatures below 1473 K. The almost negligible influence of O and N was reported to be within the error of the method.

Beskorovainyi and Yakovlev (12) reported quite opposite features of the system. They observed similarly to (7,8) a weak temperature dependence of the Fe solubility in liquid Li and a significant influence of impurities in Li causing an large increase of the Fe solubility at temperatures above 873 K. Minushkin and Steinmetz (13) determined a dissolution rate of Fe in liquid Li at 1033 K and a mean value of the Fe solubility of $4.2\cdot10^{-4}$ mol % Fe after 4 h of equilibration. Although an extension of the investigations into the temperature range 866-1144 K was announced (14) the following publications (15,16) merely described tests at 935 and 1033 K, indicating a steep increase of the Fe solubility from $3.2\cdot10^{-4}$ to $1.05\cdot10^{-3}$ mol % Fe. These data are in better agreement with (9,11) than with (7,8,12). Numerous experiments performed by Leavenworth et al. (17,18) at 929 to 1198 K were in fair agreement with (9,11,14-16), the solubility increasing from $2.6\cdot10^{-5}$ to $1.5\cdot10^{-3}$ mol % Fe with a mean scatter of data of ± 15 %. The investigated equilibrium could have been disturbed by an interaction of dissolved Fe with the Ti sample bucket by absorption of Fe or even the formation of Fe-Ti intermetallics (19).

It seems that the solubility value of Fe in Li of $1.4\cdot10^{-4}$ mol % Fe at 873 K presented by Weeks (20) was rather taken from a fitting equation than from experimental work, as the conditions of measurements were not described. The paper is, therefore, not compiled. Cheburkov (21) performed several solubility determinations in the temperature range of 1023-1473 K. His results were graphically presented in (22). The solubility of Fe in pure Li increased from $4\cdot10^{-4}$ to $7\cdot10^{-3}$ mol % Fe. The apparent solubility increased slightly from $1.5\cdot10^{-3}$ to only $2.3\cdot10^{-3}$ mol % Fe in the temperature range 430 to 1523 K, if Li contained 0.5 mol % N. The study is not compiled, since experimental details are not given and special points in the figure could not be exactly read out.

Ivanov and Solovev (23) reported an increase of the Fe solubility in Li from $6\cdot10^{-6}$ to $2\cdot10^{-3}$ mol % Fe at 623 to 1408 K (as taken from the figure of a secondary source). Their data are always in the range of the lowest corresponding solubility values reported in (8,12,15,17,20). As experimental details are unknown, the work is not compiled.

The influence of O, N, and C on the solubility of Fe in liquid Li was investigated by Plekhanov et al. (24) at temperatures of 775 and 1075 K. Apparent changes of the Fe solubility in Li containing 0.22 mol % O or 0.12 mol % C were not observed. However, an increase of the N concentration to 0.5 mol % caused an enormous increase of the Fe content in liquid Li. A set of precise measurements was performed by Beskorovainyi et al. (22,25-28), who studied the solubility of Fe in pure Li containing non-metallic additives (H,N,O). The largest increase of the solubility of Fe was due to introduction of N, O was less effective and H showed the smallest effect (26,28). The results did not confirm any of the previous data resulting from studies of the effects of non-metals on the solubility of Fe in Li (11,12,24). Since great care was taken of the purity of the materials, and the measurements were performed by means of direct technique using X-ray absorption, the results ($1.6\cdot10^{-4}$ to $6.2\cdot10^{-4}$ mol % Fe at 1248 to 1323 K) are regarded to be most reliable. Moreover, these data agree with theoretical predictions of (29,30). The same group of authors (31) formulated a thermodynamic theory of the Fe-N-Li interaction parameter, explaining the additional dissolution of Fe in liquid Li, if N is present in the system.

COMPONENTS:	EVALUATOR:
(1) Iron; Fe; [7439-89-6] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1991

CRITICAL EVALUATION: (continued)

Several compilers proposed solubility equations on the basis of various references:

Gill (32)	$\log(soly/\text{mol \% Fe}) = -0.76 - 2590(T/\text{K})^{-1}$	from (8,9,17)	(Eq.1)
Kelly (33)	$\log(soly/\text{mol \% Fe}) = -0.31 - 3100(T/\text{K})^{-1}$	from (17)	(Eq.2)
Selle (34)	$\log(soly/\text{mol \% Fe}) = -0.349 - 3034(T/\text{K})^{-1}$	unknown sources	(Eq.3)
Kubaschewski (35)	$\log(soly/\text{mol \% } \alpha \text{ Fe}) = -2.46 - 700(T/\text{K})^{-1}$	from (9,12,20)	(Eq.4)
Kubaschewski (35)	$\log(soly/\text{mol \% } \gamma \text{ Fe}) = 4.75 - 9000(T/\text{K})^{-1}$	from (9,12,20)	(Eq.5)
Beskorovainyi (22)	$\log(soly/\text{mol \% } \gamma \text{ Fe}) = (7.38 \pm 1.62) - (13890 \pm 2000)(T/\text{K})^{-1}$	from (22)	(Eq.6)

The $\alpha \leftrightarrow \gamma$ transformation of Fe at 1185 K has obviously influence on the Fe solubility in liquid Li. The slope of the temperature dependence of the solubility is less steeper for αFe than for γFe according to the theoretical predictions of Kuzin et al. (29,30):

$$\log(soly/\text{mol \% } \alpha \text{ Fe}) = 2.782 - 4810(T/\text{K})^{-1} \qquad \text{(Eq.7)}$$

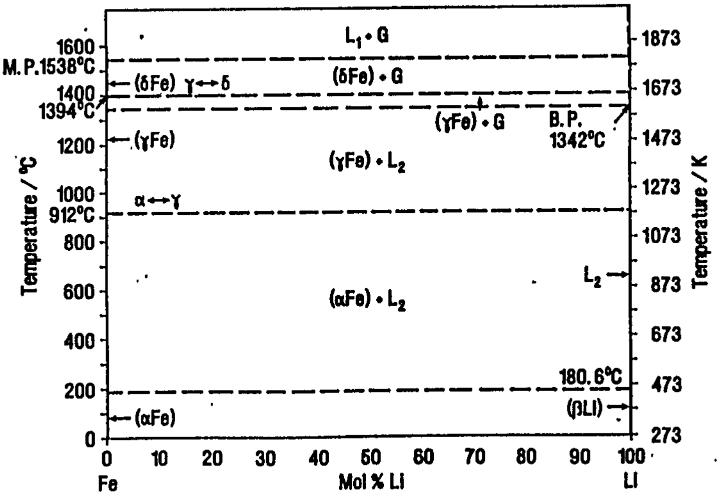
$$\log(soly/\text{mol \% } \gamma \text{ Fe}) = 5.49 - 10730(T/\text{K})^{-1} \qquad \text{(Eq.8)}$$

The correlation for αFe presented by Kubaschewski (35) is not recommended, since it is based on the overestimated solubility data at temperatures below the transformation point of Fe. Experimental values of the solubility of α Fe in liquid Li in this temperature range cannot be recommended. The data of Ivanov and Solovev (23) are closest but even still much higher than the predicted solubility of Fe in Li without contents of N.

The Fe-Li phase diagram is published in (36). The saturated solution of Fe in Li is certainly in equilibrium with almost pure Fe, since the solubility of Li in solid Fe is extremely low (2,37) and a formation of Fe-Li intermetallics can be excluded. A formation of Li_3FeN_2 in Li contaminated with N was suggested by Addison (38).

<u>The tentative values of the solubility of γ Fe in liquid Li</u>

T/K	$soly$/mol % Fe	source
1200	$6 \cdot 10^{-5}$	(22) extrapolated
1273	$3 \cdot 10^{-4}$	(22), Eq.6
1323	$7 \cdot 10^{-4}$	(22), Eq.6



COMPONENTS:	EVALUATOR:
(1) Iron; Fe; [7439-89-6] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1991

CRITICAL EVALUATION: (continued)

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COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Wilkinson, W.D.; Yaggee, F.L.

US Atom.Ener.Comm.Rep. ANL-4990, 1950.

VARIABLES:

One temperature: 573 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubility of Fe in liquid Li at 300 °C from various Fe containing alloys was reported.

Fe alloy	*Fe content*/mass %	*soly*/mass % Fe	*soly*/mol % Fe [a]
Armco	99.8	$6.5\cdot10^{-3}$, $9\cdot10^{-3}$, 0.011	$8.1\cdot10^{-4}$, $1.1\cdot10^{-3}$, $1.4\cdot10^{-3}$
Inconel	5	$8.5\cdot10^{-2}$, $9\cdot10^{-2}$	$1.1\cdot10^{-2}$, $1.1\cdot10^{-3}$
1020 steel		$1.2\cdot10^{-2}$, $1\cdot10^{-2}$	$1.5\cdot10^{-3}$, $1.2\cdot10^{-3}$
302 steel	80	$1.9\cdot10^{-2}$, $2\cdot10^{-2}$ [b]	$2.4\cdot10^{-3}$,$2.5\cdot10^{-3}$ [b]
430 steel	84	0.12	0.015
440 steel	82	0.12	0.015
347 steel	74	$3.7\cdot10^{-2}$	$4.6\cdot10^{-3}$

[a] as calculated by the compilers

[b] confirmed by the weight loss of a specimen

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Specimens of the materials under test were polished with grinding paper. They were immediately covered with mineral oil, then washed with petroleum ether and dried. A testing bomb made of steel was evacuated, and molten Li was filtered into it. A specimen and U turnings for gettering were then introduced. The apparatus was filled with Ar to pressure slightly above the atmospheric aand heated for 156 h. The bomb was opened after cooling to 473 K and mineral oil was poured over the specimen which was finally reweighed. The Li was poured into pans of oil. The method of the determination of Fe in Li was not described.

SOURCE AND PURITY OF MATERIALS:

Fe: as specified in the table.

Li: from Maywood Chem. Corp., containing 0.1 % Ca, 0.01 % Na, Fe, 0.1 % SiO_2; it was further purified by means of filtering and gettering with U chips.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Jesseman, D.S.; Roben, G.D.; Grunewald, A.L.; Fleshman, W.S.; Anderson, K.; Calkins, V.P.
US Atom.Ener.Comm.Rep. NEPA-1465, 1950.

VARIABLES:

Temperature: 697-1219 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Li at various temperatures and equilibration times was reported.

t/°F	*T*/K [a]	*equil.time*/h	*soly*/mass % Fe	*soly*/mol % Fe [a]
795	697	4	0.20, 0.17	0.025, 0.021
855	730	4	0.27, 0.09, 0.21, 0.06	0.033, 0.011, 0.026, 0.0074
1170	905	4	0.23, 0.148	0.029, 0.018
1250	950	4	0.18, 0.14, 0.25, 0.138	0.022, 0.017, 0.031, 0.017
1650	1172	4	$3.7\cdot10^{-2}$	$4.6\cdot10^{-3}$
1735	1219	4	$3.7\cdot10^{-2}$, $3.0\cdot10^{-2}$	$4.6\cdot10^{-3}$, $3.7\cdot10^{-3}$
795	697	24	0.28	0.035
855	730	24	0.32, 0.31	0.040, 0.038
1170	905	24	$6.0\cdot10^{-2}$	$7.4\cdot10^{-3}$
1250	950	24	$5.0\cdot10^{-2}$, $4.5\cdot10^{-2}$	$6.2\cdot10^{-3}$, $5.6\cdot10^{-3}$
1650	1172	24	$5.0\cdot10^{-2}$	$6.2\cdot10^{-3}$
1735	1219	24	$6.0\cdot10^{-2}$, $8.0\cdot10^{-2}$	$7.4\cdot10^{-3}$, $1.0\cdot10^{-2}$
795	697	100	0.19	0.024
855	730	100	0.48, 0.14	0.060, 0.017
1170	905	100	0.17	0.021
1250	950	100	$8.0\cdot10^{-2}$, $6.0\cdot10^{-2}$	$1.0\cdot10^{-2}$, $7.4\cdot10^{-3}$
1650	1172	100	0.12	0.015
1735	1219	100	$8.0\cdot10^{-2}$, 0.13	$1.0\cdot10^{-2}$, $1.6\cdot10^{-2}$

[a] - as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Strips of a Fe sheet were placed in a pair of Armco Fe capsules which were loaded with Li in an Ar dry box and degassed. The Li content was melted in a muffle furnace. After welding the capsule was heated in a vacuum furnace with stainless steel plates, for which the average temperature was estimated from the temperature gradient in the plate. The temperature was maintained for a period of up to 100 h, the furnace was then air cooled while still held under low pressure. The capsule was weighed and opened. The solidified samples were leached out of the crucibles with distilled H_2O, the Fe which remained undissolved was removed with the capsule, dried and weighed as the tare to determine the amount of Li solution in the capsule. The leached material was filtered, the residue was spectrographically analyzed for its Fe content.

SOURCE AND PURITY OF MATERIALS:

Fe: Armco.
Li: 99.76 % pure, containing 0.25 % O, < 0.02 % N, < $5\cdot10^{-3}$ % Na.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: precision up to ± 30 %.
Temperature: precision ± 20 K.

REFERENCES:

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Sand, J.J.

US Air Force Rep. OMCC-HEF-166, 1958.

VARIABLES:

Temperature: 468-1473 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Li at various temperatures and equilibration times was reported.

t/°C	*equil.time*/min	*soly*/mass % Fe	*soly*/mass % Fe [a]	*soly*/mol % Fe [b]
195	5	$1.25 \cdot 10^{-2}$, $1.37 \cdot 10^{-2}$		
195	10	$1.09 \cdot 10^{-2}$, $1.25 \cdot 10^{-2}$		
195	20	$6.1 \cdot 10^{-3}$, $1.43 \cdot 10^{-2}$		
195	30	$1.47 \cdot 10^{-3}$	$1.21 \cdot 10^{-2}$	$1.50 \cdot 10^{-3}$
275	65	$7.7 \cdot 10^{-3}$, $8.5 \cdot 10^{-3}$, $9.3 \cdot 10^{-3}$		
275	125	$1.00 \cdot 10^{-2}$, $8.2 \cdot 10^{-3}$		
275	360	$2.39 \cdot 10^{-2}$[c], $1.19 \cdot 10^{-2}$	$9.3 \cdot 10^{-3}$	$1.16 \cdot 10^{-3}$
350	120	$1.32 \cdot 10^{-2}$, $1.12 \cdot 10^{-2}$		
350	240	$1.86 \cdot 10^{-2}$[c], $8.0 \cdot 10^{-3}$		
350	360	$1.05 \cdot 10^{-2}$, $8.0 \cdot 10^{-3}$	$1.12 \cdot 10^{-2}$	$1.39 \cdot 10^{-3}$
425	5	$1.66 \cdot 10^{-2}$, $1.09 \cdot 10^{-2}$		
425	10	$1.10 \cdot 10^{-2}$, $1.15 \cdot 10^{-3}$, $1.33 \cdot 10^{-2}$		
425	15	$1.20 \cdot 10^{-2}$		
425	20	$1.23 \cdot 10^{-2}$, $1.12 \cdot 10^{-2}$		
425	30	$1.19 \cdot 10^{-2}$, $1.95 \cdot 10^{-2}$ [c]		
425	90	$1.34 \cdot 10^{-2}$, $7.3 \cdot 10^{-3}$		
425	180	$1.89 \cdot 10^{-2}$ [c],$2.41 \cdot 10^{-2}$[c]		
425	360	$1.29 \cdot 10^{-2}$, $1.93 \cdot 10^{-2}$[c], $1.44 \cdot 10^{-2}$	$1.17 \cdot 10^{-2}$	$1.45 \cdot 10^{-3}$
620	150	$1.09 \cdot 10^{-2}$, $2.38 \cdot 10^{-2}$[c]		
620	360	$7.1 \cdot 10^{-3}$, $1.33 \cdot 10^{-2}$		
620	840	$6.0 \cdot 10^{-3}$	$9.3 \cdot 10^{-3}$	$1.16 \cdot 10^{-3}$
700	120	$9.9 \cdot 10^{-3}$, $7.2 \cdot 10^{-3}$		
700	220	$1.36 \cdot 10^{-2}$, $1.96 \cdot 10^{-2}$[c]		
700	360	$2.46 \cdot 10^{-2}$[c], $1.33 \cdot 10^{-2}$	$1.1 \cdot 10^{-2}$	$1.37 \cdot 10^{-3}$
800	60	$1.31 \cdot 10^{-2}$, $1.12 \cdot 10^{-2}$		
800	135	$1.07 \cdot 10^{-2}$, $1.31 \cdot 10^{-2}$	$1.20 \cdot 10^{-2}$	$1.49 \cdot 10^{-3}$
1000	60	$2.51 \cdot 10^{-2}$[c], $1.50 \cdot 10^{-2}$		
1000	150	$1.73 \cdot 10^{-2}$, $1.79 \cdot 10^{-2}$	$1.67 \cdot 10^{-2}$	$2.07 \cdot 10^{-3}$
1200	20	$1.65 \cdot 10^{-2}$, $1.99 \cdot 10^{-2}$		
1200	60	$2.04 \cdot 10^{-2}$, $1.93 \cdot 10^{-2}$	$1.99 \cdot 10^{-2}$	$2.36 \cdot 10^{-3}$

[a] - mean value; [b] - mean value; [c] - neglected for calculation of mean values

The observed solubilities were larger by a factor of 2-3, if the purity of the materials was less. Such values were not considered as relevant. The equilibrium was reached within a few minutes. The author proposed a fitting equation applicable to the results in the temperature range 893-1473 K:

$$soly/10^{-4} \text{ mass \% Fe} = 0.178 \, (T/\text{K}) - 17.5$$

The evaluators confirmed that the equation is correct.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Fe capsule was degreased and annealed in a H_2 furnace at 1173 K. If necessary, it was stocked in pure oil, cleaned in acetone and placed in a dry box. The entire surface of Li was shaved with an Al knife and the Li sample was introduced into the Fe capsule which was sealed and heated in a furnace to the specified temperature.

Samples of Li were withdrawn after various equilibration times to make shure after which time equilibrium was reached. The samples were quenched in a CO_2-oil bath at 243 K and dissolved in H_2O.

Fe was colorimetrically determined with thiocyanate. The amount of Li was determined by means of direct titration with 1.0 mol·dm^{-3} HCl with bromthymol blue indicator.

SOURCE AND PURITY OF MATERIALS:

Fe: from Superior Tube Comp., containing $4 \cdot 10^{-2}$ % C, 0.25 % Mn, $1.1 \cdot 10^{-2}$ % P, and $2 \cdot 10^{-2}$ % S.

Li: 99+ % pure from Lithium Corp. of Amer., containing $5 \cdot 10^{-3}$ % Ca, $6 \cdot 10^{-2}$ % N, and $1 \cdot 10^{-3}$ % Fe.

H_2O: demineralized.

H_2: electrolytic grade, further purified by passing it through steel wool at 473 K.

ESTIMATED ERROR:

Solubility: accuracy of analytical determinations ± 0.5 %.

Temperature: stability ± 2 K.

REFERENCES:

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B.

Atom. Energiya 1959, *7*, 531-536; *Kernenergie* 1960, *3*, 763-767.

VARIABLES:

Temperature: 1173-1473 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Li was measured at several temperatures.

t/°C	*soly*/mass % Fe	*soly*/mol % Fe [a]
900	0.01	$1.2 \cdot 10^{-3}$
1000	0.02-0.1 (0.02) [b]	$2.5 \cdot 10^{-3}$ - $1.2 \cdot 10^{-2}$
1200	0.35	$4.3 \cdot 10^{-2}$

[a] as calcculated by the compilers
[b] as reported in (1)

The results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The inner surface of a Fe crucible was ground, electrolytically polished and etched. This crucible was gradually filled with freshly distilled Li dripping from a stainless steel condenser. After the process had been completed, the apparatus was filled with pure Ar. The filled crucible was placed in a stainless steel container to which the cover was welded in an arc furnace. Additionally, the crucible was isolated from the steel by a Mo band. The container was placed in an arc furnace and conditioned at 1273 K for 100 h. The Li solution was cooled to solidification in less than 50 s. The Fe content in the sample was determined by means of colorometric analysis.

SOURCE AND PURITY OF MATERIALS:

Fe: Armco, remelted and cast in vavuum.
Li: distilled, containing $(2\text{-}6) \cdot 10^{-2}$ % Na, $1.5 \cdot 10^{-2}$ % K, $(1\text{-}4) \cdot 10^{-4}$ % Fe, $\leq 2 \cdot 10^{-3}$ % Mg; Si, Ni, and Cr were not detected.
Ar: "pure".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall. Metalloved. Chist. Met.* 1960, *2*, 178-188; *Metallurgy & Metallography of Pure Metals*, Gordon & Breach, N.Y., 1962, p. 178-188.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Minushkin, B.; Steinmetz, H.

US Naval Res. Rep. NDA-2118-1, 1960; *US Atom.Ener.Comm.Rep. AD-245984*, 1960.

VARIABLES:

One temperature: 1033 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Li at 1400 °F (760 °C - by the compilers) was reported.

equil.t./h	condition	*soly*/mass % Fe	*soly*/mol % Fe [a]
0.25	static	$2.8\cdot10^{-4}$	$3.5\cdot10^{-5}$
0.50	static	$2.43\cdot10^{-3}$	$3.0\cdot10^{-4}$
1.0	static	$5.8\cdot10^{-4}$	$7.2\cdot10^{-5}$
2.0	static	$1.02\cdot10^{-3}$	$1.26\cdot10^{-4}$
4.0	static	$1.40\cdot10^{-3}$	$1.74\cdot10^{-4}$
5.5	stirred	$3.91\cdot10^{-3}$	$4.8\cdot10^{-4}$
8.0	stirred	$7.0\cdot10^{-3}$	$8.7\cdot10^{-4}$
12.0	stirred	$1.92\cdot10^{-3}$	$2.4\cdot10^{-4}$
24.0	stirred	$5.45\cdot10^{-3}$	$6.8\cdot10^{-4}$
27.5	stirred	$4.0\cdot10^{-3}$; $3.5\cdot10^{-3}$ [b]	$5.0\cdot10^{-4}$; $4.3\cdot10^{-4}$ [b]
29.5	stirred	$2.45\cdot10^{-3}$; $2.9\cdot10^{-3}$ [b]	$3.0\cdot10^{-4}$; $3.6\cdot10^{-4}$ [b]

[a] - as calculated by the compilers [b] - by means of colorimetric analysis

The mean value of all measurements after 4 h of equilibration is $(4.2\pm2.0)\cdot10^{-4}$ mol % Fe as calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A stainless steel vessel and a cylindrical stirrer were electroplated in a chloride bath containing a known quantity of ^{59}Fe. The apparatus was assembled, annealed in dry H_2 for 1 h at 1200 K and vacuum outgassed. The vessel was preheated to the test temperature at which liquid Li was transferred to the Fe liner. Samples of Li were periodically collected in stainless steel tubes and the radioactivity of ^{59}Fe was measured using a scintillation detector. Some samples of Li were dissolved in H_2O and the resulting solutions were titrated with 6 mol·dm^{-3} HCl. An excess of HCl was added, and the solution was evaporated to dryness. The residue was dissolved in H_2O, $NH_2OH\cdot HCL$ was used to get a pH = 4. Bathophenantroline was added and the formed complex was extracted with amyl alcohol. The optical density of the solution was measured in a spectro photometer and compared with standards. He was applied as a cover gas.

SOURCE AND PURITY OF MATERIALS:

Fe: electrolytical.
Li: vacuum distilled, containing $1\cdot10^{-2}$ % N, $5\cdot10^{-4}$ % Fe and $3\cdot10^{-3}$ % Ni (1).
He: unspecified.

ESTIMATED ERROR:

Solubility: precision of the analysis ± 10 %, standard deviation ± 50 %.
Temperature: precision ± 3 K.

REFERENCES:

1. Arbiter, W.; Lazerus, S. *United Nuclear Corp. Rep. NDA-39*, 1957.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Iron; Fe; [7439-89-6] (2) Lithium; Li; [7439-93-2]	Beskorovainyi, N.M.; Yakovlev, E.I. *Metall. Metalloved. Chist. Met.* 1960, *2*, 189-206; *Metallurgy & Metallography of Pure Metals*, Gordon & Breach, N.Y., 1962, p. 189-206.
VARIABLES:	**PREPARED BY:**
Temperature: 673-1273 K Purity of Li	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Li was reported. The values were read out from the figure and recalculated to mol % Fe by the compilers.

t/°C	purity of Li	*soly*/mass % Fe	*soly*/mol % Fe
400	high purity Li	$4 \cdot 10^{-3}$	$5 \cdot 10^{-4}$
600	high purity Li	$5 \cdot 10^{-3}$	$6 \cdot 10^{-4}$
800	high purity Li	$6 \cdot 10^{-3}$	$7 \cdot 10^{-4}$
1000	high purity Li	$8 \cdot 10^{-3}$	$1.0 \cdot 10^{-3}$
400	technical Li	$2 \cdot 10^{-3}$	$2 \cdot 10^{-4}$
600	technical Li	$7 \cdot 10^{-3}$	$9 \cdot 10^{-4}$
800	technical Li	0.025	$3.1 \cdot 10^{-3}$
900	technical Li	0.032	$4.5 \cdot 10^{-3}$

Both grades of purity of Li conatined $8.7 \cdot 10^{-3}$ mass % Fe.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
A Fe crucible was filled with Li and placed in a stainless steel container in an Ar atmosphere. The crucibles were equilibrated for up to 600 h, generally for 25-50 h, at the desired temperature. The whole containers were cooled with H_2O. The Fe content in Li was determined by a not further specified method.	Fe: "Armco". Li (high purity): 99.94 % pure, containing $\leq$0.025 % Na, Ca, Mg and $8.7 \cdot 10^{-3}$ % Fe. Li (technical): 98 % pure, containing 1.49 % Na, 0.31 % K, 0.18 % Mg and $8.7 \cdot 10^{-3}$ % Fe, contents of non-metallic impurities unspecified. Ar: unspecified.
	ESTIMATED ERROR:
	Solubility: precision ± $1.2 \cdot 10^{-5}$ mol % Fe. Temperature: precision ± 10 K.
	REFERENCES:

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Bychkov, Yu.F.; Rozanov, A.N.; Skorov, D.M.; Cheburkov, V.I.
Metall. Metalloved. Chist. Met. 1960, *2*, 78-92;
Metallurgy & Metallography of Pure Metals, Gordon & Breach, N.Y., 1962, p. 78-92.

VARIABLES:

Temperature: 1073-1473 K
Concentration of O and N in Li: 0.39-0.49 and 0.50-0.54 mol %, respectively

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The concentration of Fe in liquid Li containing additions of O or N which was equilibrated with Yal-T stainless steel was determined.

t/°C	*time*/h	addition/mass %	addition/mol %[a]	*soly*/mass % Fe	*soly*/mol % Fe[a]
975	110	-	-	$1.9 \cdot 10^{-3}$	$2.4 \cdot 10^{-4}$
975	240	-	-	$1 \cdot 10^{-3}$	$1.2 \cdot 10^{-4}$
975	110	0.99 % O	0.43 % O	$1.9 \cdot 10^{-3}$	$2.4 \cdot 10^{-4}$
975	240	1.12 % O	0.49 % O	$1.2 \cdot 10^{-3}$	$1.5 \cdot 10^{-4}$
975	110	1.0 % N	0.50 % N	$8 \cdot 10^{-4}$	$1.0 \cdot 10^{-4}$
800	150	-	-	$7 \cdot 10^{-3}$	$8.7 \cdot 10^{-4}$
800	150	0.9 % O	0.39 % O	$9.4 \cdot 10^{-3}$	$1.2 \cdot 10^{-3}$
800	150	1.1 % N	0.54 % N	$1.06 \cdot 10^{-2}$	$1.3 \cdot 10^{-3}$
900	220	-	-	$1.0 \cdot 10^{-3}$ [b]	$1.2 \cdot 10^{-4}$ [b]
1200	5	-	-	0.34 [b]	$4.2 \cdot 10^{-2}$ [b]

[a] - as calculated by the compilers   [b] - as reported in (1) and (2)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A specimen and a crucible made of Yal-T steel were electrolytically polished. Gazeous O or N were added to Li in a separate glass apparatus. The loading of the crucible with Li and the specimen was made in a dry Ar glove box. The crucible was closed by welding and placed in a stainless steel container. The whole set-up was placed in an arc furnace and heated to the desired temperature for a given period of time. After equilibration the container was quenched in cold H_2O. The opened crucible was covered with paraffin. The content of Fe in Li was analyzed by a colorimetric method in the Inst. of General & Inorg. Chem. Acad. Sci. of USSR.

SOURCE AND PURITY OF MATERIALS:

Yal-T steel: ~ 74 % Fe, 17.3 % Cr, 10 % Ni, 0.48 % Si, 0.4 % Ti, 0.1 % C, 0.07 % Mn, 0.015 % P, 0.01 % S.
Li: distilled, containing $(2\text{-}6) \cdot 10^{-2}$ % Na, $1 \cdot 10^{-2}$ % K, $4 \cdot 10^{-4}$ % Fe; Ni and Cr were not detected.
O: by decomposition of $KMnO_4$.
N: chemically pure.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 10 K.

REFERENCES:

1. Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B. *Atom. Energiya* 1959, *7*, 531-536; *Kernenergie* 1960, *3*, 763-767.
2. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall. Metalloved. Chist. Met.* 1960, *2*, 178-188; *Metallurgy & Metallography of Pure Metals*, Gordon & Breach, N.Y., 1962, p. 178-188.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Leavenworth, H.W.; Cleary, R.E.; Bratton, W.D.

US Atom.Ener.Comm.Rep. PWAC-356, 1961.

VARIABLES:

Temperature: 929-1198 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Li containing $9 \cdot 10^{-3}$ mass % N ($4.5 \cdot 10^{-3}$ mol % N - as calculated by the compilers) were reported in a figure, they were read out and calculated to mol % Fe by the compilers.

T/K	*soly*/mol % Fe	T/K	*soly*/mol % Fe
929	$2.6 \cdot 10^{-4}$ [a]	1157	$8.8 \cdot 10^{-4}$ [a]
1038	$5.0 \cdot 10^{-4}$ [a]	1157	$1.05 \cdot 10^{-3}$ [b]
1110	$8.9 \cdot 10^{-4}$ [b]	1157	$1.18 \cdot 10^{-3}$ [b]
1117	$6.8 \cdot 10^{-4}$ [a]	1163	$9.4 \cdot 10^{-4}$ [a]
1118	$9.0 \cdot 10^{-4}$ [b]	1174	$1.12 \cdot 10^{-3}$ [a]
1127	$8.7 \cdot 10^{-4}$ [b]	1181	$1.49 \cdot 10^{-3}$ [a]
1135	$7.7 \cdot 10^{-4}$ [b]	1186	$1.49 \cdot 10^{-3}$ [a]
1149	$9.2 \cdot 10^{-4}$ [a]	1198	$9.4 \cdot 10^{-4}$ [b]

[a] by means of radioactive tracer technique; [b] - by wet chemical analysis

All results were also reported in (1). The kinetics of the dissolution of Fe in liquid Li were investigated.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test equipment was essentially a dipping bucket apparatus which is described in (1). A Fe cup was electrolytically plated with ^{59}Fe and filled with Li under Ar atmosphere. The cup was equilibrated at the desired test temperature for 24 h. A Ti bucket was dipped into the liquid Li to take a sample for analysis. The samples was dissolved and its Fe content chemically precipitated. The activity of the precipitate was measured and the Fe content calculated.
An alternative method was the direct counting of the activity after taking out the bucket with a sample of ~ 0.5 g solution. It was observed that some ^{59}Fe was adsorbed on the outer surface of the sample cup. This may have caused an overestimation of the solubility.
The N level of Li was established by the addition of Li_3N.

SOURCE AND PURITY OF MATERIALS:

Fe: electrolytically precipitated.
Li: 99.8 % pure; further purified by contacting with Ti sponge for 2 h at 1144 K and cooled to 260-315 K; contained < $5 \cdot 10^{-4}$ mol % N.
Ar: purified.

ESTIMATED ERROR:

Nothing specified.
Solubility: precision ± 15 % (by the compilers).

REFERENCES:

1. Leavenworth, H.W.; Cleary, R.E. *Acta Metall.* 1961, *9*, 519-520.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Minushkin, B.

US Naval Res. Rep. NDA-2141-1, 1961; *US Atom.Ener. Comm.Rep. AD-259703*, 1961.

VARIABLES:

Temperature: 935 and 1033 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Li at two temperatures under various conditions was reported.

t/°C	condition	*equil.time*/h	*soly*/mass % Fe·10^{-3}	*mean value of soly*/mol % Fe [a]
662	static	0.50	1.2, 2.2	$3.2 \cdot 10^{-4}$
	static	0.83	1.9, 2.3	
	static	1.25	2.2, 3.3	
	static	1.75	3.5, 1.3	
	static	2.75	2.7, 2.0	
	static	4.50	1.5, 4.0	
	static	7.50	2.7, 4.0	
	static	24.50	1.5, 2.5	
662	dynamic	0.50	5.3, 6.1	$3.5 \cdot 10^{-4}$
	dynamic	0.75	4.3, 1.2	
	dynamic	1.08	2.3, 3.0	
	dynamic	1.50	2.0, 4.2	
	dynamic	2.50	2.6, 3.0	
	dynamic	4.50	2.0, 2.0	
	dynamic	6.50	3.6, 3.1	
	dynamic	24.50	3.0, 2.7	
760	static	2.0	7.1, 9.1	$1.05 \cdot 10^{-3}$
	static	2.29	6.7, 7.8	
	static	2.61	14.2	
	static	3.27	9.3, 12.9	
	static	4.00	6.6, 10.7	
	static	8.00	1.00, 5.9	
	static	26.00	6.8	

[a] as calculated by the compilers; the mean results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A stainless steel vessel with an inner Fe liner and a cylindrical Fe stirrer was outgassed and filled with liquid Li. He was applied as a cover gas. During the dissolution procedure, samples of the solution were removed after specified periods of time by means of a Ta bucket which could be introduced through the *Wilsons* seals in the gas locks. The bottom section of the apparatus was heated in a electric furnace the temperature of which was measured by means of a thermocouple.
The Li samples were dissolved in H2O and the resulting solutions were titrated with HCl to determine the Li content. An excess of HCl was then added to the solution which was subsequently boiled and filtered. The Fe(III) in the solution was reduced with $NH_2OH \cdot HCl$. Fe(II) formed a complex with ortho phenantroline at pH=3.5 which was determined by means of spectrophotometric method.

SOURCE AND PURITY OF MATERIALS:

Fe: 99.75 % pure Armco, containing $3 \cdot 10^{-2}$ % C, $<7 \cdot 10^{-2}$ % Mn, P, $<3 \cdot 10^{-2}$ % S, <0.12 % Si, Cu.
Li: 99.80 pure from Maywood Chem. Works, further purified by vacuum distillation, containing $5 \cdot 10^{-4}$ % Fe, $3 \cdot 10^{-3}$ % Ni, and $1 \cdot 10^{-2}$ % N.
He: unspecified.

ESTIMATED ERROR:

Solubility: precision of analysis ± 10 %, standard deviation of results better than ± 30 %.
Temperature: stability ± 3 K.

REFERENCES:

1. McKee, J.M.; Steinmetz, H. *US Atom.Ener.Comm. Rep. TID-7626*, Pt.I, 1962, p. 143.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Plekhanov, G.A.; Fedortsov-Lutikov, G.P.; Glushko, Yu.V.
Atom. Energiya 1978, *45*, 143-145.

VARIABLES:

Temperature: 773-1078 K
Contents of 0.005-0.22 mol % O, 0.004-0.5 mol % N and 0.001-0.12 mol % C in Li

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The influence of O, N, and C on the solubility of Fe in liquid Li was studied at two temperatures.

t/°C	addition to Li	*soly*/mol % Fe	*soly*/mol % Fe [a]
800-805	-	$1.0 \cdot 10^{-3}$, $8.7 \cdot 10^{-4}$, $8.7 \cdot 10^{-4}$	$9.1 \cdot 10^{-4}$
	0.215-0.220 mol % O	$1.2 \cdot 10^{-3}$, $1.0 \cdot 10^{-3}$, $8.5 \cdot 10^{-4}$	$1.0 \cdot 10^{-3}$
	0.12 mol % C	$7.5 \cdot 10^{-4}$, $8.3 \cdot 10^{-4}$	$7.9 \cdot 10^{-4}$
	0.25-0.27 mol % N	$1.86 \cdot 10^{-3}$,$1.74 \cdot 10^{-3}$, $1.74 \cdot 10^{-3}$	$1.78 \cdot 10^{-3}$
	0.50 mol % N	$6.29 \cdot 10^{-3}$,$4.48 \cdot 10^{-3}$, $5.48 \cdot 10^{-3}$	$5.4 \cdot 10^{-3}$
500-505	0.25-0.26 mol % N	$1.0 \cdot 10^{-3}$, $1.1 \cdot 10^{-3}$, $1.10 \cdot 10^{-3}$	$1.06 \cdot 10^{-3}$
	0.50 mol % N	$1.60 \cdot 10^{-3}$,$1.50 \cdot 10^{-3}$, $1.50 \cdot 10^{-3}$	$1.53 \cdot 10^{-3}$

[a] mean values

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Solid Li with additions of Li_2O, Li_3N or graphite was placed in a Fe vessel which was closed in a dry Ar glove box. The vessel was installed in a Mo container. The system was equilibrated for 24 h at the desired temperature. The apparatus was finally inverted to allow the solution to flow into the upper part of the Mo container. The cooled Li sample was analyzed by means of a spectroscopic method.

SOURCE AND PURITY OF MATERIALS:

Fe: Armco.
Li: filtered, containing $5 \cdot 10^{-3}$ mol % O, $(4\text{-}6) \cdot 10^{-3}$ mol % N, and $(1\text{-}3) \cdot 10^{-3}$ mol % C.
Li_3N: pure.
Li_2O: pure.
Graphite: spectrally pure.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: precision <± 10 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Beskorovainyi, N.M.; Vasilev, V.K.; Lyublinskii, I.E.

Metall. Metalloved. Chist. Met. 1980, *14*, 135-148.

VARIABLES:

Temperature: 773-1323 K
concentration of N in Li: $5\cdot10^{-4}$-0.25 mol %
concentration of O in Li: 0.087 mol %
concentration of H in Li: 0.34 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Li was reported.

t/°C	*soly*/mass % Fe	*soly*/mol % Fe [a]
925	$<1.0\cdot10^{-3}$	$<1.2\cdot10^{-4}$
975	$(1.3\pm0.4)\cdot10^{-3}$	$1.6\cdot10^{-4}$
1000	$(3.3\pm0.2)\cdot10^{-3}$	$4.1\cdot10^{-4}$
1050	$(5.0\pm0.9)\cdot10^{-3}$	$6.2\cdot10^{-4}$

[a] as calculated by the compilers

The results might be expressed by the fitting equation which was proved by the compilers:

$$\log(soly/\text{mol \% Fe}) = (7.38\pm1.62)-(13890\pm2000)(T/\text{K})^{-1}$$

The same results were reported in graphical form in (1-3).
The solubility of Fe in liquid Li containing non-metallic additives was also reported in (1) and (4). The experimental methods were the same as used for the pure solute. The data were read of figures and recalculated to mol % by the compilers.

t/°C	additive	*concn*/mass %	*concn*/mol %	*soly*/mass % Fe	*soly*/mol % Fe
500	N	0.49	0.244	$1.3\cdot10^{-3}$	$1.6\cdot10^{-4}$
600				$5.5\cdot10^{-3}$	$6.8\cdot10^{-4}$
650				$8.0\cdot10^{-3}$	$9.9\cdot10^{-4}$
700				$1.1\cdot10^{-2}$	$1.4\cdot10^{-3}$
750				$1.4\cdot10^{-2}$	$1.7\cdot10^{-3}$
800				$1.7\cdot10^{-2}$	$2.1\cdot10^{-3}$
850				$2.4\cdot10^{-2}$	$3.0\cdot10^{-3}$
900				$3.0\cdot10^{-2}$	$3.7\cdot10^{-3}$
950				$3.6\cdot10^{-2}$	$4.5\cdot10^{-3}$
1000				$4.4\cdot10^{-2}$	$5.5\cdot10^{-3}$
800	N	0.11	0.055	$4.3\cdot10^{-3}$	$5.3\cdot10^{-4}$
850				$9.5\cdot10^{-3}$	$1.2\cdot10^{-3}$
900				$1.3\cdot10^{-2}$	$1.6\cdot10^{-3}$
950				$1.8\cdot10^{-2}$	$2.2\cdot10^{-3}$
1000				$2.5\cdot10^{-2}$	$3.1\cdot10^{-3}$
1050				$2.9\cdot10^{-2}$	$3.6\cdot10^{-3}$
900	N	0.05	0.025	$9.2\cdot10^{-4}$	$1.14\cdot10^{-4}$
950				$1.6\cdot10^{-3}$	$2.0\cdot10^{-4}$
975				$3.2\cdot10^{-3}$	$4.0\cdot10^{-4}$
900	O	0.2	0.087	$1.2\cdot10^{-3}$	$1.5\cdot10^{-4}$
950				$2.3\cdot10^{-3}$	$2.9\cdot10^{-4}$
1000				$4.1\cdot10^{-3}$	$5.1\cdot10^{-4}$
900	H	0.05	0.34	$8.1\cdot10^{-4}$	$1.0\cdot10^{-4}$
925				$1.1\cdot10^{-3}$	$1.4\cdot10^{-4}$
950				$1.6\cdot10^{-3}$	$2.0\cdot10^{-4}$

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Beskorovainyi, N.M.; Vasilev, V.K.; Lyublinskii, I.E.

Metall. Metalloved. Chist. Met. 1980, *14*, 135-148.

VARIABLES:

Temperature: 773-1323 K
concentration of N in Li: $5 \cdot 10^{-4}$-0.25 mol %
concentration of O in Li: 0.087 mol %
concentration of H in Li: 0.34 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES: (continued)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A cylindrical crucible made of Fe was placed in a hermetical capsule. The crucible was filled with Li (and additives) which was kept inside by surface tension forces. V foil (as getter for non-metallic elements) was added to the capsule. The capsule was equipped with "Be windows" (transparent to X-rays) which were not in contact with Li. The capsule was filled with He and heated to the desired temperature. The X-ray beam passed along the crucible axis. The beam which was emitted by the sample was analyzed in a Soller's spectrometer.

The intensity of the beam was measured passing from both sides of the K-boundary absorption of Fe. The amount of Fe in the sample was calculated by the corresponding formula.

SOURCE AND PURITY OF MATERIALS:

Fe: "specially" pure metal, further remelted under Ar atmosphere and annealed 1 h at 1373 K.
Li: 99.6 % pure (LE-1), further distilled and equilibrated with Nb-Zr(5%) and Y at 1248 K; finally with contents of $<5 \cdot 10^{-3}$ % O and $\leq 1 \cdot 10^{-3}$ % N, C.
Li_3N: chemically pure.
O: from equilibration of Li with a NbO sample.
H: unspecified.
He: unspecified.

ESTIMATED ERROR:

Solubility: precision better than ± 25 %; reading-out procedure ±5 %, detection limit $1 \cdot 10^{-4}$ mol % Fe.
reading-out procedure ±3 K.
Temperature: stability ± 0.1 K; reading-out procedure ±3 K.

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COMPONENTS:	EVALUATOR:
(1) Iron; Fe; [7439-89-6] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1993

CRITICAL EVALUATION:

The system Fe-Na is undoubtedly the most intensively investigated pair of a transition and an alkali metal. Almost a thousand of solubility determinations of Fe in liquid Na were reported in the literature. Several critical evaluations of the system were published before (1-9).

First qualitative observations were reported a century ago (10), and the subsequent publications indicated that Fe is very slightly soluble in Na (11,12), as well as Na in solid Fe (13,14).

The available data on the solubility of Fe in liquid Na are scattering over three orders of magnitude for a given temperature. It is well proved that O present in Na is strongly influencing the apparent solubility of Fe in Na. The solubility of absolutely pure Fe in pure Na should increase from $1.35 \cdot 10^{-15}$ at 600 K to $1.14 \cdot 10^{-4}$ mol % Fe at 1183 K according to a theoretical prediction (15). The majority of the data is, however, much higher than the line between these two points (see figure).

The experimental values are presented in several classes depending on the level of the apparent solubility. The data reported in (7,16-26) represent the highest solubility level and show a small slope versus temperature. With the exception of the reports (17-20) in which the O concentrations were specified to be $< 6 \cdot 10^{-4}$ mol %, all other determinations were performed at O levels of the order of 10^{-2} mol %. The lowest values of the solubility were generated by (27-31). The slope versus temperature is somewhat higher in these data than in the first class. A very low O level was specified in (27), while the O content was not specified in (28,29). The solubility measurements in (27-29) were performed by means of radiochemical analysis, which was claimed to give more precise results (27). This was not confirmed by (19). The correctness of the estimation in (31) was discussed between (32,33) and (34), since the solid in equilibrium with the solution was stainless steel instead of Fe. The results for steel (30,31) were confirmed by (32), but this report claimed a 10^3 times higher solubility for Fe also compared to the data of (35). (25) reported a solubility of Fe at 1173 K from the solid phase steel 1H18N10T of 0.2 of the value with Armco Fe as the equilibrated phase. On the other hand, (36) found $3.7 \cdot 10^{-3}$ mol % Fe in Na containing 0.29 mol % O when V was equilibrated with Na in a type 304 stainless steel capsule.

The third group of solubility data was obtained by (7,21,32,37-40). These data form a bridge between those of the first and the second group. The temperature dependence of the solubility is highest and the O level was specified to be very low with the exception of (40).

We observe solubility results with temperature gradient parallel to those of the first and second group in the fourth group (28,29,41). In these experiments, O levels in Na were moderate and cautiously varied. It is uncertain wether the concentrations of O in Na were constant, since the solvent was reported to be saturated with Na_2O or NaOH (28,29). The authors of (42) received values of the solubility the averages of which slightly decreased with temperature. They could not give an explanation.

Some data were given without details of the experimental procedures. They are listed here:

Reference	(37)	(43)	(43)	(25)	(25)	(44)	(44)	(26)
T/K	773	823	823	1073	1173	873	1173	730
O concn/mol%	unknown	$1.4 \cdot 10^{-3}$	$1.4 \cdot 10^{-2}$	$4.3 \cdot 10^{-3}$	$4.3 \cdot 10^{-3}$	unknown	unknown	$8.3 \cdot 10^{-3}$
soly/mol % Fe	$<4 \cdot 10^{-5}$	$3 \cdot 10^{-4}$	$\sim 4 \cdot 10^{-4}$	$2.2 \cdot 10^{-3}$	$3.2 \cdot 10^{-3}$	$1.2 \cdot 10^{-4}$	$2 \cdot 10^{-4}$	$8.5 \cdot 10^{-3}$

Several factors may influence the solubility of Fe in Na: the O concentration in Fe as well as in Na, the equilibration time, the material of sampler, the applied analytical methods, the absence of passivating surface layers on the solid. The influence of O was not systematically studied, though a dependence of the Fe solubility on the O content of Na was detected early (28) and intensively investigated in (7,21,22,29,38,39). (24) did not observe any dependence of the solubility of Fe on the O concentration in Na. (22,39) and (43) measured solubilities which were not influenced by the O level, if it was kept below 0.1 of the saturation, while a steep gradient with the O concentration occurred at higher O contents. More recent experiments indicated an influence of O (7,21,28,29,39) even at very small O concentration (27). Theoretical considerations (45,46) imply a proportionality of the Fe solubility to the O content of the system. The strength of the Fe-O-Na interaction was described by parameters increasing with increasing temperature. This was, however, not confirmed by the thermal stability of Fe-O-Na solid compounds. (45,46) contain some incertainties and the numerical values of the interaction parameters were different. (45) included a critical concentration of O at which the solubility of Fe increased, while such value was not presented in (46). It was , however, shown (47,48) that the formation of Fe-O-Na solid compounds is possible at O concentrations above the critical value for a given temperature.

An influence of other non-metallic elements on the apparent solubility of Fe in liquid Na was not investigated in detail. N should have only negligible effect, since it has an extremely low solubility in Na. C was shown to be without any influence on the solubility of Fe in Na (4): H which was added as NaOH to Na caused an increase of the solubility proportional to the added concentration. This effect was additional to the effect of added Na_2O (28,29).

The influence of metallic elements as Ni, Mo or other components of stainless steels was discussed (6); they should not significantly influence the solubility of Fe. If Fe alloys may be used as solute, the apparent solubility of Fe should be corrected in order to unify the Fe activity. The development of Fe surfaces did not show any effect on the measured solubility (28,29).

The criterium of a sufficient time for equilibration was mentioned. (20,28,29) consider that 2 h should be sufficient to reach the equilibrium at temperatures above 500 K. (40,41) mentioned that this time should be longer than 12 h at temperatures below 973 K and this statement seems to be more reliable.

Three groups of analytical techniques were mainly applied for the determination of the solubility of Fe in liquid sodium: radiochemical, wet chemical, and measurements of corrosion grooves on the surfaces. The detection limit of the wet chemical analyses is $4 \cdot 10^{-4}$ mol % Fe, thus, all results below this limit would not be in relation

COMPONENTS:	EVALUATOR:
(1) Iron; Fe; [7439-89-6] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1993

CRITICAL EVALUATION: (continued)

to the true solubility. The radiochemical method is not limited in this way. The sensitivity of the measurement of grain boundary grooves is comparable to the radiochemical method. The method of sampling Na for analyses is of fundamental importance for the quality of the results. (49) demonstrated the consequences of the formation of Fe particles at the interface between Na and the cover gas: An enrichment of Fe by the factor of 100 was found in the interface layer compared to the bulk Na. Thus, the sampling procedure or a filtering process has to avoid the transfer of undissolved Fe into the analytical sample. The temperature has a strong influence of possible supersaturation and precipitation of Fe by cooling the samples. Many of the results in (19,20) seem to be over estimated in this way. It has to be recommended to perform such experiments only at increasing temperatures.

The selection of the most reliable data may be tried on different paths. If all values might be used, the resulting average line may be close to the results of (3,7,21,38,39). (2,5) tended to favour the high solubility data of (16-20,22-24). The selection of the low solubility data (27-29) might be recommended after the critical evaluation of the data in (6) and in (8), and these data are supported since they are in agreement with corrosion rate models (30,31,44,50,51). The intermediate values of the solubility (28,29,40) may be suggested for increasing O contents in Na after (9).

The scattered results of the determinations of the solubility of Fe in liquid Na can be explained by the assumption of two regions in which two different equilibria may appear (40). Solid Fe is in equilibrium with the saturated solution in the first region at high temperature and low O concentration, while Fe-O-Na ternary oxides are involved in the equilibria with the solution in the second region which is characterized by lower temperatures and higher O concentrations. The formation of solid Na_4FeO_3, as the most probable equilibrium phase, was reported in (4,22,48,52-55). The precise nature of the Fe-O-Na complexes is still not yet known, and the formation of Na_4FeO_3 may as well be excluded from thermodynamic reasons (56). The invariant temperature of coexistence of Na, Na_2O, Fe and Na_4FeO_3 was found in several studies to be between 626 and 760 K. Below 626 K solid Fe and liquid Na coexist with Na_2O, while they coexist with Na_4FeO_3 above a temperature of 760 K (57). The equilibrium relationships in the temperature range 626-760 K are, however, not clear and need further experimental work for explanation. Other compounds the formation of which is probable are $NaFe_2O_6$ (58), Na_2FeO_2 (54,59,60), and $Na_2Fe_2O_4$ (54,56,61-63). Thermodynamic aspects of their formation were investigated and discussed in (47,48,54,56,60-63). A phase diagram of the Fe-O-Na system was constructed by (55) at 773 to 923 K in which Na_4FeO_3 was presented as the equilibrium phase with Na saturated with Fe.

(64) contributed to the knowledge of the structure of solutions of Fe in Na with O contents. It was shown in electrotransport measurements that Fe and O atoms are mutually bonded in the medium at ~ 1073 K. The addition of Li to the solutions decreased the apparent solubility due to the decrease of the O activity.

A schematic Fe-Na phase diagram was reported in (65). Some solubility data were reported above the α Fe ↔ γ Fe allotropic transformation temperature (1185 K), but the scatter of data did not allow to draw conclusions related to the solubility of Fe allotropes in Na.

Tentative values of the solubility of α Fe in liquid Na at low O concentration ($\leq 1\cdot10^{-4}$ mol %)

T/K	*soly*/mol % Fe	source
873	$3\cdot10^{-7}$	(27)
973	$6\cdot10^{-7}$	(27)

The results presented in (28,29,40) might be used for solubilities at higher O contents, they should, however, not be higher than the solubilities from (21,24).

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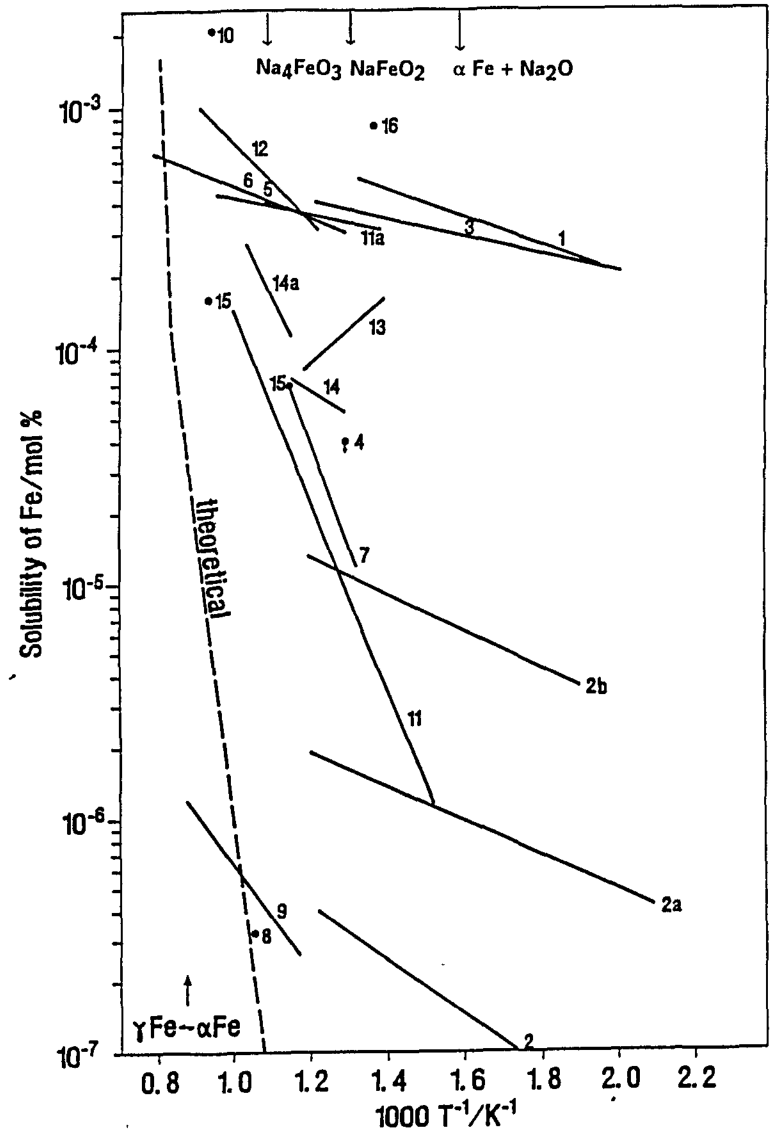
COMPONENTS:	EVALUATOR:
(1) Iron; Fe; [7439-89-6] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1993

CRITICAL EVALUATION: (continued)



The points indicated in the figure are related to the references:

1.	(23)	5.	(16)	9.	(27)	13.	(42)
2.,a,b	(28)	6.	(17-20)	10.	(25)	14.,a	(40)
3.	(24)	7.	(38,39)	11.,a	(21)	15.	(32)
4.	(37)	8.	(34)	12.	(22)	16.	(26)

In the case of 2.a and 11.a, the O concentration in Na was elevated by means of addition of Na_2O, in the case of 2.b H_2O was added; 14 and 14.a correspond to static and dynamic tests, respectively.

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COMPONENTS:	EVALUATOR:
(1) Iron; Fe; [7439-89-6] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1993

CRITICAL EVALUATION: (continued)

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COMPONENTS:	**ORIGINAL MEASUREMENTS:**
(1) Iron; Fe; [7439-89-6] (2) Sodium; Na; [7440-23-5]	Epstein, L.F. *Science* 1950, *112*, 426.
VARIABLES:	**PREPARED BY:**
Temperature: 504-756 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

33 individual measurements of the solubility of Fe in liquid Na were performed in the temperature range 231-483 °C. The data can be fitted to the linear equation by means of the least square method:

$$soly/\text{mass \% Fe} \cdot 10^4 = -1.47 + 0.030(t/°\text{C})$$

The data were alternatively presented by the authors of (2) in the forms:

$$soly/\text{mass \% Fe} \cdot 10^4 = -1.1 + 0.02767\ (t/°\text{C}),\ \text{or}$$
$$\log(soly/\text{mol \% Fe}) = -2.57 - 558.5\ (T/\text{K})^{-1}$$

Some numerical data were also given.

t/°C	$soly$/mass % Fe	$soly$/mol % Fe [a]
100	$1.5 \cdot 10^{-4}$	$6 \cdot 10^{-5}$
100	$2 \cdot 10^{-4}$ [b]	$8 \cdot 10^{-5}$
500	$1.35 \cdot 10^{-3}$	$5.5 \cdot 10^{-4}$
700	$1.83 \cdot 10^{-3}$ [b]	$7.5 \cdot 10^{-4}$

[a] as calculated by the compilers; [b] as reported in (1)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Liquid Na was heated in a Fe vessel under Ar atmosphere. Dip sampling of Na was performed at selected equilibration temperatures. The Na samples obtained by this procedure were dissolved in ethanol. The Fe content of the solution was spectrophotometrically determined using the complexometric reaction with α,α'-dipyridyl.

SOURCE AND PURITY OF MATERIALS:

Fe: nothing specified.
Na: with approximately $2 \cdot 10^{-2}$ % O.

ESTIMATED ERROR:

Solubility: precision ± $1.7 \cdot 10^{-4}$ mass % Fe.
Temperature: nothing specified.

REFERENCES:

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COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Bogard, A.D.

US Atom.Ener.Comm. Rep. NRL-4131, 1953.

VARIABLES:

Temperature: 478-835 K
Concentration of O in Na from saturated solution of Na_2O and NaOH

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe ion pure liquid Na at various temperatures.

t/°C	*soly*/mass % Fe	*soly*/mol % Fe [a]	t/°C	*soly*/mass % Fe	*soly*/mol % Fe [a]
230	$2.44\cdot10^{-7}$	$1.00\cdot10^{-7}$	404	$5.00\cdot10^{-7}$	$2.1\cdot10^{-7}$
232	$0.92\cdot10^{-7}$	$3.8\cdot10^{-8}$	483	$5.75\cdot10^{-7}$	$2.4\cdot10^{-7}$
304	$3.72\cdot10^{-7}$	$1.5\cdot10^{-7}$	521	$9.07\cdot10^{-7}$	$3.7\cdot10^{-7}$
329	$2.68\cdot10^{-7}$	$1.10\ 10^{-7}$	544	$1.06\cdot10^{-6}$	$4.3\cdot10^{-7}$

The solubility of Fe in liquid Na saturated with Na_2O at various temperatures.

t/°C	*soly*/mass % Fe	*soly*/mol % Fe [a]	t/°C	*soly*/mass % Fe	*soly*/mol % Fe [a]
205	$0.78\cdot10^{-6}$	$3.2\cdot10^{-7}$	431	$2.03\cdot10^{-6}$	$0.83\cdot10^{-6}$
277	$1.58\cdot10^{-6}$	$6.4\cdot10^{-7}$	445	$2.47\cdot10^{-6}$	$1.01\cdot10^{-6}$
331	$1.52\cdot10^{-6}$	$6.2\cdot10^{-7}$	469	$2.76\cdot10^{-6}$	$1.13\cdot10^{-6}$
349	$1.00\cdot10^{-6}$	$4.1\cdot10^{-7}$	477	$2.80\cdot10^{-6}$	$1.15\cdot10^{-6}$
380	$1.47\cdot10^{-6}$	$6.0\cdot10^{-7}$	524	$3.90\cdot10^{-6}$	$1.60\cdot10^{-6}$
381	$1.95\cdot10^{-6}$	$8.0\cdot10^{-7}$	527	$4.14\cdot10^{-6}$	$1.70\cdot10^{-6}$
391	$1.72\cdot10^{-6}$	$7.1\cdot10^{-7}$	559	$3.85\cdot10^{-6}$	$1.58\cdot10^{-6}$
402	$1.85\cdot10^{-6}$	$7.6\cdot10^{-7}$			

The solubility of Fe in liquid Na saturated with NaOH at various temperatures.

t/°C	*soly*/mass % Fe	*soly*/mol % Fe [a]	t/°C	*soly*/mass % Fe	*soly*/mol % Fe [a]
253	$8.1\cdot10^{-6}$	$3.6\cdot10^{-6}$	445	$2.03\cdot10^{-5}$	$8.3\cdot10^{-6}$
285	$9.7\cdot10^{-6}$	$4.0\cdot10^{-6}$	527	$4.58\cdot10^{-5}$	$1.88\cdot10^{-5}$
316	$1.25\cdot10^{-5}$	$5.1\cdot10^{-6}$	561	$2.47\cdot10^{-5}$	$1.01\cdot10^{-5}$
430	$2.59\cdot10^{-5}$	$1.06\cdot10^{-5}$	562	$3.32\cdot10^{-5}$	$1.32\cdot10^{-5}$

[a] as calculated by the compilers

All data were reported in (1), where the investigation of the dissolution kinetics of Fe in liquid Na was also described.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

^{59}Fe, which was obtained by means of irradiation at the ORNL reactor, was placed in a vessel of stainless steel. This was evacuated and filled with N_2. Molten Na was filtered through a Ni filter into the vessel. The temperature was adjusted to the desired value and kept for 2 hours. The Na sample was then filtered into a stainless steel receiver inside a dry box. The samples were weighed and dissolved in ethanol. The solutions were acidified with H_2SO_4 and ethanol was evaporated on a hot plate. Fe was recovered with a carrier as Fe_2O_3 and determined by means of the radioactivity.

SOURCE AND PURITY OF MATERIALS:

Fe: 99.99 % pure with contents of $< 2\cdot10^{-5}$ % Al; $1.7\cdot10^{-3}$ % Be, O; $7\cdot10^{-4}$ % Ca; $2\cdot10^{-4}$ % Cr; $5.9\cdot10^{-4}$ % Cu; $8\cdot10^{-5}$ % Ni; $6\cdot10^{-4}$ % Si; $1\cdot10^{-3}$ % C; $1.2\cdot10^{-3}$ % S; $4\cdot10^{-4}$ % N; $4\cdot10^{-4}$ % H.
Na: distilled.
N_2: purified by purging with Na-K melt.
C_2H_5OH: contained unmeasurable amounts of Fe.

ESTIMATED ERROR:

Solubility: unspecified.
Temperature: stability ± 1.5 K.

REFERENCES:

1. Baus, R.A.; Bogard, A.-D.; Grand, J.A.; Lockhart, L.B.; Miller, R.R.; Williams, D.D. *Peaceful Uses of Atomic Energy*, Unit.Nations, N.Y., 1956, *9*, 356-363.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Rodgers, S.J.; Mausteller, J.W.; Batutis, E.F.

Mine Safety Appliences Co. Rep. TR-27, 1954; *US Atom.Ener.Comm. Rep. NP-5241*, 1954.

VARIABLES:

Temperature: 486-826 K
O concentration in Na: $4\cdot10^{-3}$-$6.5\cdot10^{-2}$ mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Na at various temperatures was determined in static tests.

T/K	*soly*/mass % Fe	*soly*/mol % Fe [a]	T/K	*soly*/mass % Fe	*soly*/mol % Fe [a]
486	$5\cdot10^{-4}$; $6\cdot10^{-4}$	$2.1\cdot10^{-4}$; $2.5\cdot10^{-4}$	698	$9\cdot10^{-4}$	$3.7\cdot10^{-4}$
574	$7\cdot10^{-4}$	$2.9\cdot10^{-4}$	746	$1.3\cdot10^{-3}$	$5.3\cdot10^{-4}$
592	$7\cdot10^{-4}$	$2.9\cdot10^{-4}$	811	$9\cdot10^{-4}$ (average)	$3.7\cdot10^{-4}$
685	$9\cdot10^{-4}$	$3.7\cdot10^{-4}$	826	$1.1\cdot10^{-3}$	$4.5\cdot10^{-4}$

The solubility of Fe in liquid Na at various temperatures was determined in a set of dynamic tests.

T/K	*soly*/mass % Fe	*soly*/mol % Fe [a]	T/K	*soly*/mass % Fe	*soly*/mol % Fe [a]
486	$6\cdot10^{-4}$	$2.5\cdot10^{-4}$	685	$1.0\cdot10^{-3}$	$4.1\cdot10^{-4}$
574	$1.0\cdot10^{-3}$	$4.1\cdot10^{-4}$	791	$1.4\cdot10^{-3}$	$5.7\cdot10^{-4}$

[a] calculated by the compilers

The data were taken from the text and the figure of (1), since the original report was not available.

In the dynamic experiments at 810 K the level of the Fe concentration remained constant at about $9\cdot10^{-4}$ mass % Fe ($4\cdot10^{-4}$ mol % Fe as calculated by the compilers), if the O concentration in Na increased from $2.0\cdot10^{-3}$ to $4.5\cdot10^{-2}$ mass%. The same amount of Fe was determined in Na samples taken from a static Ni container at 810 K at O concentrations between $3\cdot10^{-3}$ to $1.0\cdot10^{-2}$ mass%, though no Fe was added.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experimental determinations were performed in (i) dynamic loops and (ii) static pots, both made of stainless steel or Ni. Na was equilibrated with a Fe specimen and then dip sampled with a Ni bucket. Fe was determined by means of the thiocyanate method after dissolving the Na samples. The bucket did not absorb Fe due to analytical tests. The results were consistent with those in which glass crucibles were used.

SOURCE AND PURITY OF MATERIALS:

Fe: nothing specified.
Na: (i) cold trapped with contents of $2.0\cdot10^{-3}$ % O; (ii) with contents of $(3.0-10)\cdot10^{-3}$ % O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Claar, T.D. *Reactor Technol.* 1970, *13*, 124-146.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Hopenfeld, J.; Robertson, W.M.

Corrosion **1969**, *25*, 365-366.

VARIABLES:

One temperature: 948 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Grain boundary grooves caused by corrosion of type 304 stainless steel in a Na loop were used to calculate the solubility of Fe in liquid Na at 675 °C.

time/hours	*groove width*/μm	*soly*/mass % Fe	*soly*/mol % Fe [a]
247	0.8	$2.1 \cdot 10^{-7}$	$8.6 \cdot 10^{-8}$
387	1.5	$7.1 \cdot 10^{-7}$	$2.9 \cdot 10^{-7}$
589	2.4	$1.9 \cdot 10^{-6}$	$7.8 \cdot 10^{-7}$
1117	1.8	$4.2 \cdot 10^{-7}$	$1.7 \cdot 10^{-7}$

[a] as calculated by the compilers

The mean values are $(8.1 \pm 6.5) \cdot 10^{-7}$ mass % Fe, and $3.3 \cdot 10^{-7}$ mol % Fe, respectively.

Borgstedt (1) stated his critical opinion on this estimation, he argued that pure Fe should be investigated instead of Cr-Ni steel; see the corresponding data sheet and discussion in (2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus was operated under He cover gas atmosphere. Na flowed through a concentric annulus the inner tube of which served as test sampler. The sample source was internally heated, its surface temperature was ~ 25 K higher than the temperature of the bulk Na. The grain boundary grooves formed by Na corrosion were observed by means of an electron microscope (at 3000x to 10000x). The groove widths were estimated using stereo pairs or single micrographs of the sample surface. The solubility was calculated using the relation of (3): $w = 5(tD_vC_s\gamma\Omega^2/kT)^{1/3}$, where w - groove width, t - time of exposure, D_v - diffusion coefficient, C_s - solubility, γ - interfacial energy, Ω - atomic volume, k - Boltzmann constant, and T - temperature.

SOURCE AND PURITY OF MATERIALS:

Fe source: 71 % Fe, 18 % Cr, 8 % Ni, 8.10-2 % C, < 2 % Mn, < 1 % Si.
Na: purified through a cold trap, with a content of $< 1.0 \cdot 10^{-3}$ % O.

ESTIMATED ERROR:

Solubility: standard deviation ± 80 %, accuracy of the groove width measurement ± 50 %.
Temperature: precision ± 2 K.

REFERENCES:

1. Borgstedt, H.U. *Corrosion* **1971**, *27*, 113-114.
2. Hopenfeld, J.; Robertson, W.M. (discussion), Borgstedt, H.U. (reply) *Corrosion* **1971**, *27*, 478.
3. Mullins, W.W. *Trans. AIME* **1960**, *218*, 354-361.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.

US Atom.Ener.Comm. Rep. AI-AEC-12834, 1969.

VARIABLES:

Temperature: 773-1273 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Na at various temperatures was measured using different collectors.

t/°C	collector	*soly*/mass % Fe	*soly*/mol % Fe[a]	*t*/°C	collector	*soly*/mass % Fe	*soly*/mol % Fe[a]
500[b]	Ti	$2.68\cdot10^{-3}$	$1.1\cdot10^{-3}$	799	Ni	$3.8\cdot10^{-3}$	$1.6\cdot10^{-3}$
595[b,c]	Ni	$1.2\cdot10^{-3}$	$4.9\cdot10^{-4}$	800[b,c]	Ti	$9.98\cdot10^{-3}$	$4.1\cdot10^{-3}$
600[b,c]	Ti	$7.0\cdot10^{-4}$	$2.9\cdot10^{-4}$	800[b,c]	Ti	$8.4\cdot10^{-4}$	$3.4\cdot10^{-4}$
650	Ti	$2.5\cdot10^{-3}$	$1.0\cdot10^{-3}$	800[b]	Ti	$1.94\cdot10^{-3}$	$8.0\cdot10^{-4}$
698[b,c]	Nb-Zr	$3.9\cdot10^{-3}$	$1.6\cdot10^{-3}$	898	Ni	$4.5\cdot10^{-3}$	$1.8\cdot10^{-3}$
700[b]	Ti	$9.9\cdot10^{-4}$	$4.1\cdot10^{-4}$	900[b,c]	Ti	$1.29\cdot10^{-3}$	$5.3\cdot10^{-4}$
709[b,c]	Ti	$1.22\cdot10^{-3}$	$5.0\cdot10^{-4}$	991[b,c]	Nb-Zr	$1.6\cdot10^{-3}$	$6.6\cdot10^{-4}$

Two analytical methods were compared in another set of experiments: radiochemical (first two columns) vs. wet chemical analysis (last two columns); Ni was the collector material.

t/°C	*soly*/mass % Fe	*soly*/mol % Fe[a]	*soly*/mass % Fe	*soly*/mol % Fe[a]
700	$1.57\cdot10^{-3}$	$6.4\cdot10^{-4}$	$1.66\cdot10^{-3}$	$6.8\cdot10^{-4}$
600	$8.5\cdot10^{-4}$	$3.5\cdot10^{-4}$	$9.9\cdot10^{-4}$	$4.1\cdot10^{-4}$
800	$1.54\cdot10^{-3}$	$6.3\cdot10^{-4}$	$3.18\cdot10^{-3}$	$1.3\cdot10^{-3}$
1000	$1.70\cdot10^{-2}$	$7.0\cdot10^{-3}$	$2.45\cdot10^{-2}$	$1.0\cdot10^{-2}$
600	$5.8\cdot10^{-5}$	$2.4\cdot10^{-5}$	$6.3\cdot10^{-4}$	$2.6\cdot10^{-4}$
700	$5.6\cdot10^{-4}$	$2.3\cdot10^{-4}$	$8.7\cdot10^{-4}$	$3.6\cdot10^{-4}$
600	$6.8\cdot10^{-5}$	$2.7\cdot10^{-5}$	$1.14\cdot10^{-3}$	$4.7\cdot10^{-4}$
800	$1.14\cdot10^{-3}$	$4.7\cdot10^{-4}$	$1.21\cdot10^{-3}$	$5.0\cdot10^{-4}$
1000	$1.85\cdot10^{-3}$	$7.6\cdot10^{-4}$	$3.18\cdot10^{-3}$	$1.3\cdot10^{-3}$
900	$1.6\cdot10^{-4}$	$6.6\cdot10^{-5}$	$4.9\cdot10^{-4}$	$2.0\cdot10^{-4}$
800	$4.1\cdot10^{-4}$	$1.7\cdot10^{-4}$	$5.3\cdot10^{-4}$	$2.2\cdot10^{-4}$
900	$3.9\cdot10^{-5}$	$1.6\cdot10^{-5}$	$2.0\cdot10^{-4}$	$8.2\cdot10^{-5}$
1000	$5.0\cdot10^{-4}$	$2.1\cdot10^{-4}$	$8.6\cdot10^{-4}$	$3.5\cdot10^{-4}$
1000	$1.4\cdot10^{-4}$	$5.7\cdot10^{-5}$	$1.15\cdot10^{-3}$	$4.7\cdot10^{-4}$
900	$1.1\cdot10^{-4}$	$4.5\cdot10^{-5}$	$1.22\cdot10^{-3}$	$5.0\cdot10^{-4}$
900	$9.0\cdot10^{-5}$	$3.7\cdot10^{-5}$	$4.7\cdot10^{-4}$	$1.9\cdot10^{-4}$
600	$1.3\cdot10^{-4}$	$5.3\cdot10^{-5}$	$7.5\cdot10^{-4}$	$3.1\cdot10^{-4}$
900	$2.4\cdot10^{-4}$	$1.0\cdot10^{-4}$	$1.35\cdot10^{-3}$	$5.5\cdot10^{-4}$
700	$1.3\cdot10^{-4}$	$5.3\cdot10^{-5}$	$8.9\cdot10^{-4}$	$3.6\cdot10^{-4}$
800	$7.0\cdot10^{-5}$	$2.9\cdot10^{-5}$	$4.9\cdot10^{-4}$	$2.0\cdot10^{-4}$
600	$9.0\cdot10^{-5}$	$3.7\cdot10^{-5}$	$7.7\cdot10^{-4}$	$3.2\cdot10^{-4}$
1000	$1.3\cdot10^{-4}$	$5.3\cdot10^{-5}$	$8.3\cdot10^{-4}$	$3.4\cdot10^{-4}$
800	$3.0\cdot10^{-4}$	$1.2\cdot10^{-4}$	$2.0\cdot10^{-3}$	$8.2\cdot10^{-4}$

[a] as calculated by the compilers [b] reported in (1) [c] reported in (2,3)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The tests were performed in an assembly consisting of a Fe test crucible and a collector made of Nb-Zr(1%) alloy, Ti, or Ni. The assembly was degassed for 2 hours at 623 K, the crucible loaded with Na and the whole equipment sealed under high vacuum. After equilibration at the desired temperature for 6 hours in Ar atmosphere the capsule was inverted in order to collect the Na into the collector which was cooled and opened. Na was dissolved and analyzed by atomic absorption spectroscopy.

In the second set of tests Fe strips were irradiated in the Shield Test Irradiation Reactor. The strips containing ^{59}Fe were equilibrated with Na within a crucible made of Nb-Zr alloy. The collector section was separated from the equilibrator after moving the Na into this component. The ^{59}Fe activity was directly counted in the Na sample.

SOURCE AND PURITY OF MATERIALS:

Fe: 99.996 % pure with contents of $8\cdot10^{-4}$ % C, $<1\cdot10^{-5}$ % H, $7.2\cdot10^{-4}$ % O, $7.0\cdot10^{-4}$ % N, $2\cdot10^{-4}$ % Ca, $8\cdot10^{-4}$ % Mg, and $\leq 1\cdot10^{-4}$ % each of other elements.
Na: 99.996 % pure with contents of $3\cdot10^{-4}$ % Fe, $1.9\cdot10^{-3}$ % C, and $1.5\cdot10^{-4}$ % O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. ANL-7520*, 1968, p.319-324.
2. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. AI-AEC-12638*, 1967, p.163-173.
3. McKisson, R.L.; Eichelberger, R.L.; Gehri, D.C.; Guon. J. *US Atom.Ener.Comm. Rep. AI-AEC-12721*, 1968 ,p. 279-308.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Singer, R.M.; Fleitman, A.H.; Weeks, J.R.; Isaacs, H.S.

Corrosion by Liq. Met., J.E. Draley, J.R. Weeks, Eds., Plenum, N.Y., 1970, p. 561-576.

VARIABLES:

Temperature: 755-1023 K

O concentration in Na: $1\cdot10^{-4}$-0.32 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Na was numerically reported in (1), except the values at 600 °C.

t/°C	*O concn*/mass %	*O concn*/mol % [a]	*soly*/mass % Fe	*soly*/mol % Fe [a]
482	$5.0\cdot10^{-3}$	$7.2\cdot10^{-3}$	$2.7\cdot10^{-5}$; $3.3\cdot10^{-5}$	$1.1\cdot10^{-5}$; $1.4\cdot10^{-5}$
486	$2.2\cdot10^{-2}$	$3.2\cdot10^{-2}$	$\leq4\cdot10^{-5}$	$\leq1.6\cdot10^{-5}$
486	$4.6\cdot10^{-2}$	$6.6\cdot10^{-2}$	$1.8\cdot10^{-4}$; $2.0\cdot10^{-4}$	$7.4\cdot10^{-5}$; $8.2\cdot10^{-5}$
486	0.1035	0.148	$1.05\cdot10^{-3}$	$4.3\cdot10^{-4}$
525	0.149	0.213	$2.9\cdot10^{-3}$	$1.2\cdot10^{-3}$
575	0.225	0.32	$5.2\cdot10^{-3}$; $8.0\cdot10^{-3}$	$2.1\cdot10^{-3}$; $3.3\cdot10^{-3}$
575	0.225	0.32	$1.62\cdot10^{-2}$; $1.46\cdot10^{-2}$	$6.6\cdot10^{-3}$; $6.0\cdot10^{-3}$
575	0.225	0.32	$6.8\cdot10^{-2}$	$2.8\cdot10^{-2}$
550	$<1\cdot10^{-4}$	$<1.4\cdot10^{-4}$	$9\cdot10^{-6}$	$3.7\cdot10^{-6}$
550	$8.5\cdot10^{-3}$	$1.22\cdot10^{-2}$	$6.6\cdot10^{-5}$	$2.7\cdot10^{-5}$
550	$3.4\cdot10^{-2}$	$4.9\cdot10^{-2}$	$7.3\cdot10^{-5}$	$3.0\cdot10^{-5}$
550	$5.7\cdot10^{-2}$	$8.1\cdot10^{-2}$	$1.54\cdot10^{-4}$	$6.3\cdot10^{-5}$
550	0.10	0.143	$1.51\cdot10^{-4}$	$6.2\cdot10^{-5}$
550	0.16	0.23	$2.8\cdot10^{-4}$	$1.1\cdot10^{-4}$
550	0.18	0.26	$3.6\cdot10^{-4}$	$1.5\cdot10^{-4}$
550	0.204	0.29	$4.2\cdot10^{-4}$	$1.7\cdot10^{-4}$
550	$1.31\cdot10^{-2}$	$1.87\cdot10^{-2}$	$1.1\cdot10^{-4}$	$4.5\cdot10^{-5}$
550	$7.2\cdot10^{-3}$	$1.03\cdot10^{-3}$	$8.7\cdot10^{-5}$	$3.6\cdot10^{-5}$
550	$5.0\cdot10^{-3}$	$7.2\cdot10^{-3}$	$6.1\cdot10^{-5}$	$2.5\cdot10^{-5}$
550	$2.5\cdot10^{-3}$	$3.6\cdot10^{-3}$	$1.07\cdot10^{-4}$	$4.4\cdot10^{-5}$
600 [b]	$1.5\cdot10^{-2}$ [c]	$6.4\cdot10^{-3}$	$1.7\cdot10^{-4}$	$7.0\cdot10^{-5}$
600 [b]	$3.5\cdot10^{-2}$ [c]	$1.5\cdot10^{-2}$	$1.5\cdot10^{-4}$	$6.2\cdot10^{-5}$
600 [b]	$6.5\cdot10^{-2}$ [c]	$2.8\cdot10^{-2}$	$1.4\cdot10^{-4}$	$5.7\cdot10^{-5}$
600 [b]	$7.2\cdot10^{-2}$ [c]	$3.1\cdot10^{-2}$	$1.5\cdot10^{-4}$	$6.2\cdot10^{-5}$
600 [b]	$7.5\cdot10^{-2}$ [c]	$3.2\cdot10^{-2}$	$2.2\cdot10^{-4}$	$9.0\cdot10^{-5}$
600 [b]	0.16 [c]	$6.9\cdot10^{-2}$	$8\cdot10^{-4}$	$3.3\cdot10^{-4}$

[a] as calculated by the compilers [b] as read from the figure [c] ratio of O concentration to O solubility in Na

Some solubility experiments were performed at 650 and 750 °C in Na containing $< 1\cdot10^{-4}$ mass % O; the results were largely less than the detection limit of $5\cdot10^{-6}$ mass % Fe ($2\cdot10^{-6}$ mol % Fe, as calculated by the compilers). The apparent solubility appeared to be independent of the O concentration, if the O concentration was not higher than 20 % of the saturation level. No detectable amounts of Fe were lost during the removal of O by means of gettering with U foil. The Fe solubilities in Na were compared with Fe concentrations in Na which was circulating in Na loops in several USA nuclear laboratories. The concentrations were of the same order as the solubilities in all cases. The equilibrium concentration of Fe in Na at 500 °C was determined in samples from the bottom as $(8\text{-}26)\cdot10^{-5}$, the bulk as $<6\cdot19^{-5}$, and the surface as $(1.0\text{-}2.6)\cdot10^{-2}$ mass % Fe (2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Fe crucible was irradiated in the Brookhaven Graphite Research Reactor and then treated in H_2 for 1 h at 873 K. Mo sampling cups were pickled in aqua regia and HCl until no Fe was detectable. Na was directly distilled into the crucible. The system was pressurized with He and equilibrated at the desired temperature for up to 24 h.

The cups were dipped into the melt, kept there for 15 min and raised. Na samples were dissolved in C_2H_5OH, and the cups were leached in 6 mol·dm^{-3} HCl for 1 h at 373 K.

Fe carrier was added and $Fe(OH)_3$ twice precipitated. ^{59}Fe was counted by an analyzer. Na_2O was added to Na in some experiments, while its O level was decreased by means of gettering with U foil (2).

SOURCE AND PURITY OF MATERIALS:

Fe: 99.999 % pure from Material Research Comp., with contents of $7\cdot10^{-4}$ % O, N and $8\cdot10^{-4}$ % C.
Na: filtered, U gettered and distilled; with contents of $<1\cdot10^{-4}$ % O.
He: ultrapure grade, additionally purified with Linde 4A molecular sieve and Zr-Ti(1:1) chips at 873 K; final contents of $4.5\cdot10^{-5}$ % O and $8\cdot10^{-5}$ % H_2O.

ESTIMATED ERROR:

Solubility: detection limit $2\cdot10^{-6}$ mol % Fe.
Temperature: nothing specified.

REFERENCES:

1. Weeks, J.R. *US Atom.Ener. Comm.Rep. BNL-50149*, 1968; Singer, R.M.; Weeks, J.R. *US Atom.Ener.Comm.Rep. ANL-7520*, Pt. I. 1968, p. 309.
2. Isaacs, H.S.; Singer, R.M.; Becker, W.W. *Corrosion by Liquid Metals*, J.E. Draley, J.R. Weeks, Eds., Plenum, N.Y., 1970, p. 577.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Fleitman, A.H.; Isaacs, H.S.

presented at the Met. Conf., Cleveland, Ohio, 1970; as reported in (1).

VARIABLES:

Temperature: 855-1136 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubilities of Fe in liquid Na were reported in a figure. They were read out and calculated in mol % by the compilers.

T/K	*soly*/mass % Fe	*soly*/mol % Fe	T/K	*soly*/mass % Fe	*soly*/mol % Fe
1136	$2.8\cdot10^{-6}$	$1.2\cdot10^{-6}$	980	$1.3\cdot10^{-6}$	$5.3\cdot10^{-7}$
1111	$2.6\cdot10^{-6}$	$1.1\cdot10^{-6}$	962	$1.2\cdot10^{-6}$	$4.9\cdot10^{-7}$
1099	$2.4\cdot10^{-6}$	$9.8\cdot10^{-7}$	952	$1.1\cdot10^{-6}$	$4.5\cdot10^{-7}$
1087	$2.2\cdot10^{-6}$	$9.0\cdot10^{-7}$	918	$1.0\cdot10^{-6}$	$4.1\cdot10^{-7}$
1075	$2.1\cdot10^{-6}$	$8.6\cdot10^{-7}$	901	$9.2\cdot10^{-7}$	$3.8\cdot10^{-7}$
1064	$1.9\cdot10^{-6}$	$7.8\cdot10^{-7}$	893	$8.3\cdot10^{-7}$	$3.4\cdot10^{-7}$
1042	$1.8\cdot10^{-6}$	$7.4\cdot10^{-7}$	877	$7.5\cdot10^{-7}$	$3.1\cdot10^{-7}$
1020	$1.6\cdot10^{-6}$	$6.6\cdot10^{-7}$	863	$7.0\cdot10^{-7}$	$2.9\cdot10^{-7}$
1000	$1.5\cdot10^{-6}$	$6.1\cdot10^{-7}$	855	$6.3\cdot10^{-7}$	$2.6\cdot10^{-7}$

The authors observed a linear relation between the apparent solubility of Fe in liquid Na and the O concentration in the solvent (which was varied in the range 0.001 to 0.1 degree of the saturation) at 650 and 750 °C. Skyrme (3) quoted similar data in a figure at 700, 550, 500 and 400 °C, a differentiation of points at respective temperature was, however, not made.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A radioactive ^{59}Fe tab was immersed in liquid Na within a Mo vessel. The tab was equilibrated for several days. Na samples were taken by means of a Mo bucket from the bulk Na, contamination of its surface was avoided. Analyses were performed by means of radiochemical measurements and wet chemical methods which resulted in less consistent data. The O concentration in Na was increased by the addition of Na_2O.

SOURCE AND PURITY OF MATERIALS:

Fe: nothing specified.
Na: with O content $< 1.0\cdot10^{-3}$ % before the addition of Na_2O.

ESTIMATED ERROR:

Solubility: nothing specified; read out procedure ± 15 %.
Temperature: nothing specified; read out procedure ± 15 K.

REFERENCES:

1. Weeks, J.R.; Isaacs, H.S. *Adv. Corrosion Sci. Technol.* 1973, *3*, 1-66; *US Atom.Ener.Comm. Rep. BNL-15731R*, 1971.
2. Singer, R.M.; Weeks, J.R. *USAEC Rep. ANL-7520*, Pt. I, 1969, p. 309-318.
3. Skyrme, G. *Central Eelectricity Generating Board Rep. RD/B/N-3924*, 1977.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Borgstedt, H.U.

Corrosion 1971, *27*, 113-114.

VARIABLES:

Temperature: 873 and 1073 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Grain boundary grooves due to liquid Na corrosion on type 304 stainless steel and Armco Fe were used as source to calculate the solubility of Fe in liquid Na at 600 °C.

equilibrated material	*groove width*/µm	*soly*/mass % Fe	*soly*/mol % Fe[a]
304 stainless steel	1.0-1.5	$8 \cdot 10^{-8}$	$3.3 \cdot 10^{-8}$
Armco Fe	20-25	$1.6 \cdot 10^{-4}$	$6.6 \cdot 10^{-5}$

[a] as calculated by the compilers

The author also reported a solubility value calculated from similar measurements of Cafasso (1) which is at $4 \cdot 10^{-4}$ mass % Fe ($1.6 \cdot 10^{-4}$ mol % Fe as calculated by the compilers) at 800 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test loop was constructed of stabilized stainless steel (2). The reaction temperatures in three parallel test sections were 500, 550, and 600 °C. Specimens in the size of sheets were solution annealed at 1050 °C and then mechanically or anodically polished. The loop was operated for 500 to 1000 hours with Ar as cover gas. The specimens were removed under Ar, adherent Na was washed by means of dipping into isopropanol, and H2O, and finally dried in CO2 stream. The grain boundary grooves on the steel specimens were measured on resin replicas of the corroded surfaces which were photographed in the range of enlargement of 3000 to 10000. The grooves on Fe specimens were measured on micrographs (enlargement 500). The solubility values were calculated on the basis of the relation given by Mullins (3); see the Data Sheet by Hopenfeld and Robertson.

SOURCE AND PURITY OF MATERIALS:

Fe: 99.8 % pure with 0.02 % C, 0.05 % Mn, 0.01 % P, 0.02 % S, 0.02 % Cu, 0.04 % O, 0.006 % N.
304 SS: 71 % Fe, 18 % Cr, 8 % Ni, 0.08 % C, < 2 % Mn, < 1 % Si.
Na: cold filtered and hot trapped with Zr foil at 973 K; final O content of < $5 \cdot 10^{-4}$ % O.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: stability ± 5 K.

REFERENCES:

1. Cafasso, F.A. *US Atom.Ener.Comm. Rep. ANL-7606*, 1969, p. 118-120.
2. Borgstedt, H.U.; Drechsler, G.; Frees, G. *Werkstoffe und Korrosion* 1967, *18*, 894-897.
3. Mullins, W.W. *Trans. AIME* 1960, *218*, 354-361.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]
(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Stanaway, W.P.; Thompson, R.
US Dept.Ener.Rep.CONF-800401-P2, 1980, p.18/54-61.

VARIABLES:

Temperature: 658-1133 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Na was determined at various concentrations of O.

t/°C	*O concn*/μg/g	*Fe concn*/μg/g	*equil.time*/h	remarks	*soly*/mol % Fe [a]
385	low	0.028		U gettered	$1.15\cdot10^{-6}$
415	125	1.35	24		$5.5\cdot10^{-5}$
445	low	0.061		U gettered	$2.5\cdot10^{-6}$
450	361	13.0	45		$5.3\cdot10^{-4}$
475	359	4.0	65		$1.6\cdot10^{-4}$
525	358	9.0	66		$3.7\cdot10^{-4}$
550	184	3.0	41		$1.2\cdot10^{-4}$
550	353	6.0	66		$2.5\cdot10^{-4}$
550	385	4.0	40		$1.6\cdot10^{-4}$
550	894	7.0	41		$2.9\cdot10^{-4}$
550	1011	8.0	21		$3.3\cdot10^{-4}$
557	low	0.643		U gettered	$2.6\cdot10^{-5}$
596	low	1.216		U gettered	$5.0\cdot10^{-5}$
600	1230	9.0	43		$3.7\cdot10^{-4}$
600	1621	8.0	22		$3.3\cdot10^{-4}$
600	1775	20.0	22		$8.2\cdot10^{-4}$
650	187	17.0	45	O_2 gas added	$7.0\cdot10^{-4}$
650	286	0.8	42		$3.3\cdot10^{-5}$
650	307	9.0	45		$3.7\cdot10^{-4}$
650	358	14.0	42		$5.7\cdot10^{-4}$
650	378	9.0	26	O_2 gas added	$3.7\cdot10^{-4}$
650	378	14.0	46	O_2 gas added	$5.7\cdot10^{-4}$
650	861	9.0	24		$3.7\cdot10^{-4}$
650	998	13.0	45		$5.3\cdot10^{-4}$
650	1140	18.0	40	O_2 gas added	$7.4\cdot10^{-4}$
650	1140	16.0	65	O_2 gas added	$6.6\cdot10^{-4}$
650	1892	37	21		$1.5\cdot10^{-3}$
650	2283	59	41	O_2 gas added	$2.4\cdot10^{-3}$
650	2320	30	23		$1.2\cdot10^{-3}$
650	3058	32	42		$1.3\cdot10^{-3}$
650	3332	33	45		$1.4\cdot10^{-3}$
660	low	2.1		U gettered	$8.6\cdot10^{-5}$
700	348	9.0	40		$3.7\cdot10^{-4}$
724	low	2.5		U gettered	$1.02\cdot10^{-4}$
750	337	11.0	65	O_2 gas added	$4.5\cdot10^{-4}$
750	1138	17.0	41	O_2 gas added	$7.0\cdot10^{-4}$
750	2286	43	40	O_2 gas added	$1.8\cdot10^{-3}$
750	3685	103	23		$4.2\cdot10^{-3}$
750	4450	76	23		$3.1\cdot10^{-3}$
775	343	9.0	41		$3.7\cdot10^{-3}$
860	5530	97.5	23		$4.0\cdot10^{-3}$
860	6046	250	23		$1.02\cdot10^{-2}$

[a] as calculated by the compilers

The same results were previously reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Fe liner was plated by welding into a Ni crucible, or Fe was electroplated inside a Ni crucible. It was irradiated for a week. A part of the liner was used for the calibration of a scintillation counter. The crucible was kept on temperature by means of a heated Cu or Ni block. The heating was regulated by thermocouples. The block was mounted inside a vacuum tight silica tube filled with inert gas. Sampling of Na was achieved by piercing the lid and the base of the crucible by means of a W spike.

Na flew into a silica or Al capsule at the bottom of the apparatus when the spike was withdrawn. The weight of Na was measured, and the sample was counted in the well of the scintillation counter. The O content of the Na samples was varied by additions of Na_2O_2 or O_2 gas, and by U gettering.

SOURCE AND PURITY OF MATERIALS:

Fe: 99.995 % pure.
Na: twice distilled, containing $(1\text{-}2)\cdot10^{-5}$ % Fe.

ESTIMATED ERROR:

Solubility: ± 20 % at 95 % confidence level.
Temperature: nothing specified.

REFERENCES:

1. Thompson, R. *UK Atom.Ener.Auth. Rep. AERE R-9172*, 1979.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Periaswami, G.; Ganesan, V.; Rajan Babu, S.; Mathews, C.K.
Material Behavior & Physical Chemistry in Liquid Metal Systems, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 411-420.

VARIABLES:

Temperature: 581-846 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Na from loop experiments:

T/K	*soly*/mass % Fe	*soly*/mol %Fe[a]
581	$1.00\cdot10^{-3}$	$4.1\cdot10^{-4}$
686	$1.04\cdot10^{-2}$	$4.3\cdot10^{-3}$
712	$2.22\cdot10^{-3}$	$9.1\cdot10^{-4}$
744	$6.1\cdot10^{-4}$	$2.5\cdot10^{-4}$

The solubility of Fe in liquid Na from the solubility experiments:

T/K	*soly*/mass % Fe	*soly*/mol %Fe[a]
846	$1.82\cdot10^{-4}$	$7.5\cdot10^{-5}$
806	$2.04\cdot10^{-4}$	$8.4\cdot10^{-5}$
779	$1.75\cdot10^{-4}$	$7.2\cdot10^{-5}$
764	$2.36\cdot10^{-4}$	$9.7\cdot10^{-5}$
740	$4.91\cdot10^{-4}$	$2.0\cdot10^{-4}$
732	$5.90\cdot10^{-4}$	$2.4\cdot10^{-4}$
729	$2.00\cdot10^{-4}$	$8.2\cdot10^{-5}$
720	$3.82\cdot10^{-4}$	$1.57\cdot10^{-4}$
718	$4.85\cdot10^{-4}$	$1.99\cdot10^{-4}$

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed in capsules and a small loop. Sheets of Fe were added to Na contained in a stainless steel crucible and equilibrated. Samples of the solutions were obtained after filtering through a porosint 5 μm pore size frit. They were analyzed for the Fe content by means of the atomic absorption spectrophotometry. The experiments were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Fe: "pure"
Na: "nuclear grade" purity; further purified by gettering with Mg.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: stability ± 1 K.

REFERENCES:

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Borgstedt, H.U.
Thermochem. and Chemical Processing, Mathews, C.K., Ed., Ind. Inst. Met., Kalpakkam, 1992, p. 141-147.

VARIABLES:

Temperature: 773-973 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some values of the solubility of Fe in liquid Na were measured under static conditions[a].

t/°C	crucible	*time*/h	*soly*/mass % Fe	mean value	*soly*/mol % Fe[b]
500	Ta	19	$9.7 \cdot 10^{-5}$; $1.59 \cdot 10^{-4}$	$(1.28 \pm 0.31) \cdot 10^{-4}$	$5.3 \cdot 10^{-5}$
600	Ni	24	$1.88 \cdot 10^{-4}$; $3.03 \cdot 10^{-4}$	$(1.77 \pm 0.34) \cdot 10^{-4}$	$7.3 \cdot 10^{-5}$
		16	$1.50 \cdot 10^{-4}$; $1.07 \cdot 10^{-4}$		
		18	$1.37 \cdot 10^{-4}$		
690	Ta	4	$6.2 \cdot 10^{-5}$; $1.02 \cdot 10^{-4}$	$(0.60 \pm 0.17) \cdot 10^{-4}$	$2.4 \cdot 10^{-5}$
			$1.8 \cdot 10^{-5}$; $5.9 \cdot 10^{-5}$		
			$5.9 \cdot 10^{-5}$		

Some values of the solubilities of Fe in liquid Na were determined from tests in a stainless steel loop.

t/°C	*time*/h	*soly*/mass % Fe	mean value	*soly*/mol % Fe[b]
600	17	$4.2 \cdot 10^{-4}$; $1.4 \cdot 10^{-4}$; $1.5 \cdot 10^{-4}$	$(2.2 \pm 0.5) \cdot 10^{-4}$	
		$1.1 \cdot 10^{-4}$; $1.9 \cdot 10^{-4}$; $3.0 \cdot 10^{-4}$	$2.7 \cdot 10^{-4}$ [c]	$1.1 \cdot 10^{-4}$ [c]
700	700	$5.1 \cdot 10^{-4}$; $4.4 \cdot 10^{-4}$; $6.5 \cdot 10^{-4}$	$(5.4 \pm 0.4) \cdot 10^{-4}$	
		$5.4 \cdot 10^{-4}$	$6.8 \cdot 10^{-4}$ [c]	$2.7 \cdot 10^{-4}$ [c]

[a] also reported in (1); [b] as calculated by the compilers [c] corrected for the chemical activity of Fe in the stainless steel.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus for static measurements was made of stainless steel and consisted of an outer chamber, an inner chamber and a sampler. The apparatus was placed in an Ar glove box and was calibrated with respect to the temperature setting. The Ni or Ta crucibles were cleaned in a mixture of CH_3COOH, H_2SO_4, and HNO_3 (5:1:3) for 15 s at 363 K. They were then rinsed in H_2O and dried at 383 K. A test crucible containing a Fe coil was placed in the inner chamber, filled with Na and equilibrated. After equilibration Na was transferred into the sampling crucibles. The samples were removed, cooled, weighed and dissolved in H_2O in an Ar atmosphere. Fe was determined by atomic absorption spectrometry. Na samples from the stainless steel loop were analyzed after equilibration in the same way. The analytical results were corrected for the Fe activity according to (2).

SOURCE AND PURITY OF MATERIALS:

Fe: nothing specified.
Na: purified by removing surface precipitates at 383 K (static conditions); cold filtered at 398 ± 5 K (loop experiments).
H_2O: ultrapure.
Ar: purified with contents of $< 1 \cdot 10^{-4}$ % O and $< 1 \cdot 10^{-4}$ % H_2O.

ESTIMATED ERROR:

Solubility: standard deviation better than ± 25 %.
Temperature: stability better than ± 5 K.

REFERENCES:

1. Awasthi, S.P.; Borgstedt, H.U.; Frees, G. *Liquid Metal Engng. and Technol.*, BNES, London, 1984, *1*, 265-269.
2. Azad, A.M.; Sreedharan, O.M.; Gnanamoorthy, J.B. *J. Nucl. Mater.* 1988, *151*, 293.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Sodium; Na; [7440-23-5]

ORIGINAL MEASUREMENTS:

Thorley, A.W.
Material Behavior & Physical Chemistry in Liquid Metal Systems, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 19-36.

VARIABLES:

Temperature: 860-1213 K
O concentration in Na: $1.4 \cdot 10^{-3}$ - $1.4 \cdot 10^{-2}$ mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Na at various temperatures, O contents in Na and static or dynamic conditions was measured. The data were presented in the figure, they were read out and calculated to mol % by the compilers.

T/K	*soly*/mass % Fe	*soly*/mol % Fe	*T*/K	*soly*/mass % Fe	*soly*/mol % Fe
860^{a}	$4.6 \cdot 10^{-4}$	$1.9 \cdot 10^{-4}$ b	1023	$9.8 \cdot 10^{-4}$	$4.0 \cdot 10^{-4}$ d (2x)
860^{a}	$5.9 \cdot 10^{-4}$	$2.4 \cdot 10^{-4}$ b	1023	$9.8 \cdot 10^{-4}$	$4.0 \cdot 10^{-4}$ b
923	$4.0 \cdot 10^{-4}$	$1.6 \cdot 10^{-4}$ d (2x)	1023	$1.07 \cdot 10^{-3}$	$4.4 \cdot 10^{-4}$ b
923	$4.0 \cdot 10^{-4}$	$1.6 \cdot 10^{-4}$ b	1023	$1.07 \cdot 10^{-3}$	$4.4 \cdot 10^{-4}$ d
923	$4.9 \cdot 10^{-4}$	$2.0 \cdot 10^{-4}$ b	1023	$1.16 \cdot 10^{-3}$	$4.8 \cdot 10^{-4}$ d
$923^{\bullet}$	$5.9 \cdot 10^{-4}$	$2.4 \cdot 10^{-4}$ b (3x)	1023	$1.70 \cdot 10^{-3}$	$7.0 \cdot 10^{-4}$ d
923	$5.9 \cdot 10^{-4}$	$2.4 \cdot 10^{-4}$ b	1023	$1.85 \cdot 10^{-3}$	$7.6 \cdot 10^{-4}$ b
923	$5.9 \cdot 10^{-4}$	$2.4 \cdot 10^{-4}$ d (2x)	1043	$8.7 \cdot 10^{-4}$	$3.6 \cdot 10^{-4}$ d
923^{a}	$5.9 \cdot 10^{-4}$	$2.4 \cdot 10^{-4}$ b	1043^{a}	$1.07 \cdot 10^{-3}$	$4.4 \cdot 10^{-4}$ c
923	$6.8 \cdot 10^{-4}$	$2.8 \cdot 10^{-4}$ b	1051^{a}	$1.79 \cdot 10^{-3}$	$7.3 \cdot 10^{-4}$ c
923^{a}	$6.8 \cdot 10^{-4}$	$2.8 \cdot 10^{-4}$ b (2x)	1083^{a}	$7.8 \cdot 10^{-4}$	$3.2 \cdot 10^{-4}$ b
923	$7.8 \cdot 10^{-4}$	$3.2 \cdot 10^{-4}$ b	1093^{a}	$7.8 \cdot 10^{-4}$	$3.2 \cdot 10^{-4}$ b
923	$7.8 \cdot 10^{-4}$	$3.2 \cdot 10^{-4}$ d (2x)	1106^{a}	$7.8 \cdot 10^{-4}$	$3.2 \cdot 10^{-4}$ b
923^{a}	$7.8 \cdot 10^{-4}$	$3.2 \cdot 10^{-4}$ b	1123	$9.8 \cdot 10^{-4}$	$4.0 \cdot 10^{-4}$ b (2x)
923^{a}	$8.7 \cdot 10^{-4}$	$3.6 \cdot 10^{-4}$ c	1123	$1.07 \cdot 10^{-3}$	$4.4 \cdot 10^{-4}$ b
923	$8.7 \cdot 10^{-4}$	$3.6 \cdot 10^{-4}$ b	1123	$1.07 \cdot 10^{-3}$	$4.4 \cdot 10^{-4}$ d
923	$8.7 \cdot 10^{-4}$	$3.6 \cdot 10^{-4}$ d (2x)	1123	$1.20 \cdot 10^{-3}$	$4.9 \cdot 10^{-4}$ d
$923^{\bullet}$	$9.8 \cdot 10^{-4}$	$4.0 \cdot 10^{-4}$ b	1123	$1.27 \cdot 10^{-3}$	$5.2 \cdot 10^{-4}$ b
923	$9.8 \cdot 10^{-4}$	$4.0 \cdot 10^{-4}$ b	1123	$1.35 \cdot 10^{-3}$	$5.5 \cdot 10^{-4}$ d
923	$9.8 \cdot 10^{-4}$	$4.0 \cdot 10^{-4}$ d (2x)	1123	$1.47 \cdot 10^{-3}$	$6.0 \cdot 10^{-4}$ b (2x)
923^{a}	$9.8 \cdot 10^{-4}$	$4.0 \cdot 10^{-4}$ c	1123^{a}	$1.56 \cdot 10^{-3}$	$6.4 \cdot 10^{-4}$ c
923^{a}	$1.07 \cdot 10^{-3}$	$4.4 \cdot 10^{-4}$ c	1123	$1.66 \cdot 10^{-3}$	$6.8 \cdot 10^{-4}$ b
923	$1.07 \cdot 10^{-3}$	$4.4 \cdot 10^{-4}$ b	1123^{a}	$1.85 \cdot 10^{-3}$	$7.6 \cdot 10^{-4}$ c
923	$1.07 \cdot 10^{-3}$	$4.4 \cdot 10^{-4}$ d	1123	$2.0 \cdot 10^{-3}$	$8.4 \cdot 10^{-4}$ b
923	$1.58 \cdot 10^{-3}$	$6.5 \cdot 10^{-4}$ d	1123	$2.0 \cdot 10^{-3}$	$8.4 \cdot 10^{-4}$ d
923	$1.75 \cdot 10^{-3}$	$7.2 \cdot 10^{-4}$ d	1123	$2.2 \cdot 10^{-3}$	$8.9 \cdot 10^{-4}$ d
973^{a}	$6.8 \cdot 10^{-4}$	$2.8 \cdot 10^{-4}$ b	1123	$2.3 \cdot 10^{-3}$	$9.5 \cdot 10^{-4}$ d
973	$9.8 \cdot 10^{-4}$	$4.0 \cdot 10^{-4}$ c	1123	$2.70 \cdot 10^{-3}$	$1.11 \cdot 10^{-3}$ d
1023	$5.9 \cdot 10^{-4}$	$2.4 \cdot 10^{-4}$ b	1182^{a}	$1.85 \cdot 10^{-3}$	$7.6 \cdot 10^{-4}$ c
1023	$6.8 \cdot 10^{-4}$	$2.8 \cdot 10^{-4}$ d (3x)	1200^{a}	$6.9 \cdot 10^{-4}$	$2.8 \cdot 10^{-4}$ b
1023	$7.8 \cdot 10^{-4}$	$3.2 \cdot 10^{-4}$ d (3x)	1213^{a}	$9.8 \cdot 10^{-4}$	$4.0 \cdot 10^{-4}$ b
1023	$8.7 \cdot 10^{-4}$	$3.6 \cdot 10^{-4}$ d			

[a] determination in the alumina crucible [•] static testing mode; the majority of tests was performed in the rotating mode [b] O concentration $1.4 \cdot 10^{-3}$ mol %; [c] O concentration $1.14 \cdot 10^{-2}$ mol %; [d] O concentration $1.4 \cdot 10^{-2}$ mol %

α Fe was identified as equilibrium solid phase.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Fe test crucible was cleaned, H_2 fired and filled with Na of known O content. Some of the capsules were rotated and tilted in a furnace during the thermal treatment, while others remained static. After the heating to the equilibration temperature some capsules were quenched in ice, while others which contained alumina top crucibles were inverted to drain Na into them before quenching. Na samples were taken using a Cu tool or by means of dissolving the contents of the alumina crucibles. The further analytical methods were not described.

The residue of the vacuum distillation of Na was examined by X-ray fluorescence analysis in the case of some samples.

SOURCE AND PURITY OF MATERIALS:

Fe: nothing specified.
Na: distilled.

ESTIMATED ERROR:

Nothing specified.
Solubility: scatter of results over half an order of magnitude at one temperature (compilers).

REFERENCES:

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Potassium-Sodium eutectic; K-Na; [11147-16-3]

ORIGINAL MEASUREMENTS:

Drugas, P.G.; Kelman, L.R.

US Atom.Ener.Comm. Rep. ANL-5359, 1953.

VARIABLES:

One temperature: 973 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in the eutectic K-Na mixture (32 mol % Na) at 700 °C was found to be $(1.5 \pm 0.3) \cdot 10^{-3}$ mass % Fe. The compilers calculated the corresponding concentration in mol % to be $9 \cdot 10^{-4}$. Blank tests were performed in parallel.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Fe crucible, filter, and Ta sampler were purified in HCl, washed in H_2O and acetone, and finally dried. The crucible was loaded with the K-Na eutectic and welded in an Ar atmosphere. It was heated for three days in a Pb bath. The apparatus was then inverted. The solution was forced through the filter into the Ta sampler. The sampler was then cooled to room temperature and opened in Ar atmosphere. The whole sample was dissolved in CH_3OH, neutralized with HCl and evaporated to dryness. The residue was dissolved in H_2O, and its Fe content was determined by a colorimetric method using o-phenantroline.

SOURCE AND PURITY OF MATERIALS:

Fe: "high purity" from Westinghouse.
K-Na: from MSA, further purified by filtering twice through ultrafine filters.
CH_3OH: reagent grade, further purification by distillation.
HCl: distilled.

ESTIMATED ERROR:

Solubility: precision ± 20 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS: (1) Iron; Fe; [7439-89-6] (2) Potassium; K; [7440-09-7]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1989

CRITICAL EVALUATION:

The possibility to form alloys of Fe and K was reported by Petrov (1). All subsequent work did not confirm this statement. No solid solutions of K in Fe were detected in (2,3). The solubility of Fe was first measured in the K-Na eutectic mixture by Kelman and Drugas (4,5). The concentration of Fe in K-Na melt at temperatures up to 1073 K did not exceed the limit of $1.2 \cdot 10^{-3}$ mol % Fe in compatibility tests (4), the same Fe concentration level was suggested for the K-Na melt before the tests. This report is not compiled since essential details were not presented. In subsequent tests (5) the solubility of Fe in the liquid alloy was determined to be $9.1 \cdot 10^{-4}$ mol % Fe at 973 K. Hopenfeld and Robertson (11) applied the measurement of grain boundary grooves after corrosion tests by which they gained a value of $1.3 \cdot 10^{-5}$ mol % Fe at 1083 K. This result is in the order of 0.1 of the results of (12). Thus, there is evidence that the solubility of Fe in K-Na alloy is lower than in K. The method used in (11) is presented in the data sheet prepared for the Fe-Na system.
In experiments of Swisher (6) the solubility of Fe in liquid K increased from $3.0 \cdot 10^{-3}$ to $9.4 \cdot 10^{-2}$ mol % Fe at a temperature increase from 941 to 1202 K. The values were roughly constant with further increases of temperature. The break in the dependence of the solubility on temperature may be rather due to effects of O than to α Fe ↔ γ Fe allotropic transformation. The author used 6 sampling cup materials (Ni, Mo, Nb, Ta, Zr, and Mo/Zr) of different effectiveness as getter for O to study the influence of O on the solubility of Fe in K. The highest apparent solubility was measured in a cup of Ni, the lowest in the cup of Zr at 1144 K. Ginell and Teitel (7,8) did also not notice any increase of the saturation concentration at temperatures of 1198 and 1273 K. Their mean value of $3.4 \cdot 10^{-2}$ mol % Fe is in fair agreement with the results of (6) at the same temperature.
The results of McKisson and Eichelberger (9) at 1073 to 1473 K were one order of magnitude lower than of (6). The temperature dependence of the mean values showed, however, a similar slope. Subsequent results (10) at 1073 to 1273 were 3 times higher than in (9) with no better reproducibility. Ordynskii et al. (12) performed a study of the solubility of Fe in liquid K at 970 to 1284 K. Stainless steel containing 70 mass % Fe served as the solute. The somewhat lower chemical activity of Fe in the steel compared with pure Fe did not correspond to the measured saturation concentrations which were on order of magnitude below of (9) and two orders below of (6). An influence of various employed techniques on the results was not reported. An increase to a double Fe concentration at saturation was observed in K with 0.2 to 0.5 mol % O. The discrepancies between these results (12) and (6,7,9,10), in which the O content in K was ~ $3 \cdot 10^{-3}$ mol %, are difficult to explain. The Fe samples used in (6,7) contained ~ 0.1 mol % O, while this level was decreased to < $3 \cdot 10^{-3}$ mol % O in (9,10). Schwarz (14) reported a solubility of Fe in liquid K at 373 K to be $6.0 \cdot 10^{-4}$ mol % Fe with austenitic stainless steel as the Fe source. This result does not fit with any extrapolation line from higher temperatures, it may have been influenced by the presence of $5 \cdot 10^{-2}$ mol % O in K. The data are not compiled since experimental details were not given.
The results of (6,9,10,12) are gained from both sides of the α ↔ γ allotropic transformation temperature of Fe. The scatter of data does not allow the evaluation of a relation of the Fe modification and its solubility. Ganesan and Borgstedt (13) identified the compound $K_6Fe_2O_6$ as the corrosion product of Fe in K with O contents, and this compound may be regarded as the solid phase in the solubility equilibrium. Sangster et al.(15) presented a schematic diagram of the Fe-K binary system which is similar to that of the Fe-Li system. Some experiments have been performed under elevated pressure in order to keep K in the liquid state. The solubility equation suggested by Kubaschewski (16) on the basis of results of (6) is not recommended. The evaluator's selection of solubility data is based on (12) and verified by the compilers.

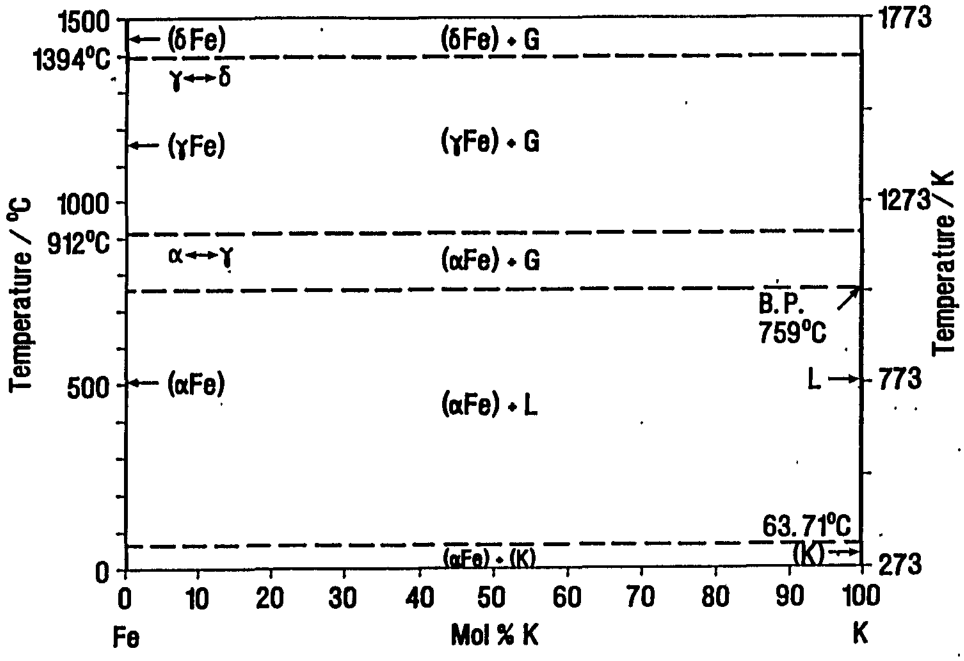
$$\log(soly/\text{mol } \% \ \alpha \text{ Fe}) = -0.571 - 3370\,(T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

The tentative values of the solubility of Fe in K

T/K	$soly$/mol % Fe	source	remarks
973	$9 \cdot 10^{-5}$ α Fe	(12), Eq.(1)	at elevated pressure
1073	$2 \cdot 10^{-4}$ α Fe	(12), Eq.(1)	at elevated pressure
1173	$4 \cdot 10^{-4}$ α Fe	(12), Eq.(1)	at elevated pressure
1273	$7 \cdot 10^{-4}$ γ Fe	(12)	at elevated pressure

COMPONENTS:	EVALUATOR:
(1) Iron; Fe; [7439-89-6] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1992

CRITICAL EVALUATION: (continued)



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7. Ginell, W.S.; Teitel, R.J. *Trans. Am. Nucl. Soc.* 1965, *8*, 393.
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11. Hopenfeld, J.; Robertson, W.M. *Corrosion* 1969, *25*, 365.
12. Ordynskii, A.M.; Popov, R.G.; Raikova, G.P.; Samsonov, N.V.; Tarbov, A.A. *Teplofiz. Vys. Temp.* 1981, *19*, 1192.
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15. Sangster, J.; Bale, C.W.; Burton, B.P. *J.Phase Equil.* 1991, *12*, 46.
16. Kubaschewski, O. *Iron-Binary Phase Diagrams*, Springer, Berlin, 1982, p. 4.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Swisher, J.H.

NASA Rep. TN-D-2734, 1965.

VARIABLES:

Temperature: 941-1328 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid K was reported in a figure; the data were read out and calculated to mol % by the compilers. A sampling cup made of Mo was used.

T/K	*soly*/mass % Fe	*soly*/mol % Fe	T/K	*soly*/mass % Fe	*soly*/mol % Fe
941[a]	$4.3\cdot10^{-3}$	$3.0\cdot10^{-3}$	1202[b]	0.13; 0.14	$9.0\cdot10^{-2}$; $9.7\cdot10^{-2}$
1031[a]	$1.5\cdot10^{-2}$	$1.05\cdot10^{-2}$	1257[b]	0.10	$7.0\cdot10^{-2}$
1144[a]	$6.2\cdot10^{-2}$; $6.7\cdot10^{-2}$	$4.3\cdot10^{-2}$; $4.7\cdot10^{-2}$	1328[b]	0.10; 0.11	$7.0\cdot10^{-2}$; $7.7\cdot10^{-2}$

[a] α -Fe [b] γ -Fe

The results can be fitted up to 1193 K by the equation which was tested by the compilers.

$$\log(soly/\text{mol \% Fe}) = 4.037 - 6166\cdot(T/\text{K})^{-1}$$

The work was also published in compact form with the equation in (1).

An effect of the materials of sampling cups on the apparent solubility was studied at 1144 K.

Material	*soly*/mass % Fe	*soly*/mol % Fe
Ni	0.354	0.247
Mo	0.0639; 0.0655	0.0447; 0.0458
Nb	0.0194; 0.0257; 0.0358	0.0136; 0.0180; 0.025
Ta	0.0117	0.00819
Zr	0.0044	0.00308
Mo with Zr	0.0077; 0.0129	0.00539; 0.00903

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility test capsule was made of Fe, sampling cups alternatively fabricated of Ni, Mo, Nb, Ta, Zr were mounted inside the capsule. This was filled with K and sealed by electron-beam welding. The assembly was equilibrated in a high vacuum furnace for some hours, the temperature of the capsule holder was measured by means of Pt/Pt-Rh(13%) thermocouples. The furnace was then inverted in order to let the sample flow into the cup. The capsule was cut open after cooling to room temperature, the K sample was dissolved in butyl alcohol. The cups were leached with HCl to remove a precipitate which was formed during the cooling procedure. Fe was colorimetrically determined in the combined solutions. The K content of the alcoholic solution was gravimetrically determined.

SOURCE AND PURITY OF MATERIALS:

Fe: 99.7952 % pure with contents of 0.015 % C, 0.051 % O, $6.8\cdot10^{-3}$ % N, 0.07 % Cu, 0.04 % Mn, 0.016 % S, $4\cdot10^{-3}$ % P, and $2\cdot10^{-3}$ % Si.

K: with a content of < $2\cdot10^{-3}$ % O.

ESTIMATED ERROR:

Solubility: standard deviation at temperatures $\leq$ 1144 K is ± 2.3 %.

Temperature: accuracy ± 3K; stability ± 1 K.

REFERENCES:

1. Swisher, J.H. *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 43.

1 COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

McKisson, R.L.; Eichelberger, R.L.

US Atom. Ener.Comm. Rep. CONF-650411, 1965, p. 37-42.

VARIABLES:

Temperature: 1073-1473 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid K was reported in the figure. The data were read out and recalculated to mol % Fe by the compilers.

t/°C	*time*/h	*soly*/mass % Fe	*soly*/mol % Fe
800	8	$1.4 \cdot 10^{-3}$; $1.7 \cdot 10^{-3}$	$9.8 \cdot 10^{-4}$; $1.2 \cdot 10^{-3}$
800	4	$2.1 \cdot 10^{-3}$	$1.5 \cdot 10^{-3}$
1000	8	$8.6 \cdot 10^{-4}$; $1.2 \cdot 10^{-3}$; $1.4 \cdot 10^{-3}$	$6.0 \cdot 10^{-4}$; $8.4 \cdot 10^{-4}$; $9.8 \cdot 10^{-4}$
1000	4	$4.2 \cdot 10^{-3}$; $7.0 \cdot 10^{-3}$	$2.9\ 10^{-3}$; $4.9 \cdot 10^{-3}$
1000	2	$5.0 \cdot 10^{-3}$	$3.5 \cdot 10^{-3}$
1200	4	$3.2 \cdot 10^{-2}$	$2.2 \cdot 10^{-2}$
1200	2	$7.9 \cdot 10^{-2}$	$5.5 \cdot 10^{-2}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The crucible which was made of a Fe single crystal was assembled with a spacer and a Ta receiver inside a capsule. The crucible was loaded with K and the capsule was closed by brazing in vacuum. Ni was used as brazing material. The capsule was heated to a selected temperature at which it was kept for the time listed in the table. Pt-Rh(1%) vs. Pt-Rh(13%) thermocouples were applied to measure the temperature at different positions of the capsule. At the end of each run the K sample was poured from the crucible to the receiver. The receiver was removed from the capsule after cooling, its content was submitted for analysis.

SOURCE AND PURITY OF MATERIALS:

Fe: triple pass zone refined single crystal.
K: filtered, hot gettered and fractionally distilled; with a final content of $1.6 \cdot 10^{-3}$ % O.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: stability ± 3 K.

REFERENCES:

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Ginell, W.S.; Teitel, R.J.

Trans. Am. Nucl. Soc. 1965, *8*, 393-394.

VARIABLES:

Temperature: 1198 and 1273 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of γ Fe in liquid K was reported.

t/°C	*soly*/mass % Fe	*soly*/mol % Fe [a]
925	$4.57 \cdot 10^{-2}$; $4.47 \cdot 10^{-2}$	$3.2 \cdot 10^{-2}$; $3.1 \cdot 10^{-2}$
1000 [b]	$5.55 \cdot 10^{-2}$; $4.44 \cdot 10^{-2}$	$3.9 \cdot 10^{-2}$; $3.1 \cdot 10^{-2}$

[a] as calculated by the compilers
[b] the data were also reported in (1)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The capsule of Nb-Zr(1%) was "L"-shaped and contained a Fe test cup, a W collector cup and a small dam positioned near the capsule bend to regulate the K flow into the collector. After chemical etching and outgassing at 1623 - 1673 K it was filled with K, closed by welding under vacuum and heated to about 100 K above the equilibrium temperature for 1 h. During this procedure the capsule was rotated in a centrifuge in order to prevent the flow of K over the dam. The test temperature was maintained for 3 h and the rate of rotation was elevated to force the precipitation of Fe at the bottom of the cup. At the end of the test the rotation was slowly reduced to allow the decantation of the solution of Fe in K over the dam and the dropping into the collector. The methods of chemical determinations were not described. The temperatures of the measurements were obtained by means of thermocouples or of optical methods. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Fe: 99.94 % pure from Westinghouse with a content of $4 \cdot 10^{-2}$ % O.
K: purified by means of gettering with chips of Ti-Zr (1:1) alloy at 1058 K; with a content of $(7-11) \cdot 10^{-4}$ % O.
Ar: "high purity" with a content of $< 2 \cdot 10^{-4}$ % H_2O.

ESTIMATED ERROR:

Solubility: precision ± 10 % (as estimated by the compilers).
Temperature: nothing specified.

REFERENCES:

1. Ginell, W.S.; Teitel, R.J. *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 44-47.; *NASA Rep. CR-82838*, 1965.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

McKisson, R.L.; Eichelberger, R.L.; Dahleen, R.C.; Scarborough, J.M.; Argue, G.R.
NASA Rep. CR-610, 1966; *Atom. Internat. Rep. AI-65-210*, 1965; *Rep. N-66-39542*, 1966.

VARIABLES:

Temperature: 1073 and 1273 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid K was reported.

t/°C	*time*/h	*O concn*/mass %	*O concn*/mol % [a]	*soly*/mass % Fe	*soly*/mol % Fe [a]
800	2.6	$4.0\cdot10^{-3}$	$9.8\cdot10^{-3}$	$2.5\cdot10^{-3}$	$1.7\cdot10^{-3}$
800	8.0	$1.4\cdot10^{-3}$	$3.4\cdot10^{-3}$	$1.47\cdot10^{-2}$	$1.0\cdot10^{-2}$
800	4.0	$1.4\cdot10^{-3}$	$3.4\cdot10^{-3}$	$7.4\cdot10^{-3}$	$5.2\cdot10^{-3}$
1000	4.0	$1.6\cdot10^{-3}$	$3.9\cdot10^{-3}$	$4.3\cdot10^{-3}$	$3.0\cdot10^{-3}$
1000	4.0	$1.4\cdot10^{-3}$	$3.4\cdot10^{-3}$	$1.97\cdot10^{-2}$	$1.4\cdot10^{-2}$
1000	8.0	$1.4\cdot10^{-3}$	$3.4\cdot10^{-3}$	$1.77\cdot10^{-2}$	$1.2\cdot10^{-2}$

[a] calculated by the compilers

The authors reported the fitting equation (tested by the compilers):

$$\log(soly/\text{mol \% Fe}) = -0.75 - 1700\,(T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Fe crucible and the components of the capsule which were made of Mo were cleaned and rinsed with acetone. They were outgassed in high vacuum at elevated temperature and finally cooled in Ar atmosphere. The crucible was loaded with K, and the capsule was closed by means of electron beam welding. The whole set-up was then heated to the test temperature and kept at this temperature for a given period. After the equilibration the capsule was inverted in order to transfer the solution in K into the collector. The capsule was opened after cooling, and the collector was separated. A sample of K was melted from the collector into a particular glassware. It reacted with H_2O vapour in He atmosphere, the aqueous solution was acidified with HCl. Fe was determined as o-phenantroline complex by means of a spectro-photometric method.

SOURCE AND PURITY OF MATERIALS:

Fe: 99.992 % pure single crystal from Materials Research Crop. with contents of $< 8\cdot10^{-4}$ % O, $1\cdot10^{-5}$ % N, $1\cdot10^{-5}$ % H, $1.0\cdot10^{-3}$ % C, $1.0\cdot10^{-4}$ % Si, $1.0\cdot10^{-4}$ % Mo.
K: as reported in Mo-K data sheet.
He: nothing specified.
Ar: nothing specified.

ESTIMATED ERROR:

Solubility: sensitivity 0.01 - 5.0 µg Fe; accuracy of chemical analysis ± 2 %.
Temperature: precision ± 2.5 K.

REFERENCES:

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Potassium; K; [7440-09-7]

ORIGINAL MEASUREMENTS:

Ordynskii, A.M.; Popov, R.G.; Raikova, G.P.; Samsonov, N.V.; Tarbov, A.A.
Teplofiz. Vys. Temp. 1981, *19*, 1192-1197.

VARIABLES:

Temperature: 970 and 1284 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid K was reported in the figure; the data were read out and recalculated to mol % by the compilers.

T/K	*soly*/mass % Fe	*soly*/mol % Fe	*T*/K	*soly*/mass % Fe	*soly*/mol % Fe
981	$9.0\cdot10^{-5}$ [c]	$6.3\cdot10^{-5}$	1068	$2.8\cdot10^{-4}$ [c]	$2.0\cdot10^{-4}$
974	$9.0\cdot10^{-5}$ [a]	$6.3\cdot10^{-5}$	1074	$2.9\cdot10^{-4}$ [a]	$2.0\cdot10^{-4}$
973	$9.5\cdot10^{-5}$ [b]	$6.7\cdot10^{-5}$	1074	$3.6\cdot10^{-4}$ [b]	$2.5\cdot10^{-4}$
973	$1.1\cdot10^{-4}$ [c]	$7.7\cdot10^{-5}$	1074	$5.1\cdot10^{-4}$ [b]	$3.6\cdot10^{-4}$
970	$1.2\cdot10^{-4}$ [c]	$8.4\cdot10^{-5}$	1166	$2.1\cdot10^{-4}$ [a]	$1.5\cdot10^{-4}$
977	$1.3\cdot10^{-4}$ [c]	$9.1\cdot10^{-5}$	1166	$2.4\cdot10^{-4}$ [b]	$1.7\cdot10^{-4}$
973	$1.5\cdot10^{-4}$ [b]	$1.05\cdot10^{-4}$	1166	$2.9\cdot10^{-4}$ [a]	$2.0\cdot10^{-4}$
973	$1.7\cdot10^{-4}$ [a]	$1.2\cdot10^{-4}$	1166	$3.1\cdot10^{-4}$ [a]	$2.2\cdot10^{-4}$
973	$2.0\cdot10^{-4}$ [b]	$1.4\cdot10^{-4}$	1166	$4.5\cdot10^{-4}$ [b]	$3.1\cdot10^{-4}$
972	$2.2\cdot10^{-4}$ [a]	$1.5\cdot10^{-4}$	1173	$5.1\cdot10^{-4}$ [a]	$3.6\cdot10^{-4}$
971	$2.5\cdot10^{-4}$ [b]	$1.7\cdot10^{-4}$	1166	$7.3\cdot10^{-4}$ [b]	$5.1\cdot10^{-4}$
971	$4.1\cdot10^{-4}$ [d]	$2.9\cdot10^{-4}$	1166	$8.3\cdot10^{-4}$ [b]	$5.8\cdot10^{-4}$
971	$5.0\cdot10^{-4}$ [d]	$3.5\cdot10^{-4}$	1166	$1.3\cdot10^{-3}$ [b]	$9.1\cdot10^{-4}$
971	$5.9\cdot10^{-4}$ [d]	$4.1\cdot10^{-4}$	1284	$5.3\cdot10^{-4}$ [b]	$3.7\cdot10^{-4}$
1079	$1.1\cdot10^{-4}$ [a]	$7.7\cdot10^{-4}$	1284	$5.7\cdot10^{-4}$ [a]	$4.0\cdot10^{-4}$
1074	$1.5\cdot10^{-4}$ [c]	$1.05\cdot10^{-4}$	1277	$6.5\cdot10^{-4}$ [b]	$4.6\cdot10^{-4}$
1076	$1.7\cdot10^{-4}$ [b]	$1.2\cdot10^{-4}$	1284	$6.8\cdot10^{-4}$ [b]	$4.8\cdot10^{-4}$
1068	$1.9\cdot10^{-4}$ [c]	$1.3\cdot10^{-4}$	1284	$7.5\cdot10^{-4}$ [a]	$5.2\cdot10^{-4}$
1074	$2.1\cdot10^{-4}$ [a]	$1.5\cdot10^{-4}$	1284	$8.5\cdot10^{-4}$ [b]	$6.0\cdot10^{-4}$
1071	$2.2\cdot10^{-4}$ [b]	$1.5\cdot10^{-4}$	1284	$1.2\cdot10^{-3}$ [b]	$8.4\cdot10^{-4}$
1075	$2.3\cdot10^{-4}$ [a]	$1.6\cdot10^{-4}$	1284	$1.4\cdot10^{-3}$ [b]	$9.8\cdot10^{-4}$
1074	$2.5\cdot10^{-4}$ [b]	$1.7\cdot10^{-4}$			

[a] spectral analysis, water cooled sample [b] spectrophotometric analysis, sample cooled by liquid N

[c] spectrophotometric analysis, centrifuge separation [d] elevated O concentration in K

The fitting equation reported by the authors is presented in the critical evaluation.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test capsule of 12Kh18N10T steel was loaded with K to be half filled, and a foil of the same steel was inserted. The capsule was then welded and placed in the Ar atmosphere of a furnace. The whole set-up was equilibrated for more than 3 h. The capsule was finally inverted to let K flow into the receiver part and cooled down with H_2O in 50-70 s. The capsule was cut open, K samples were taken out with a bronze blade. Fe was determined by spectral analysis after preconcentration.

The second method introduced the direct quenching of the K solution by pouring into liquid N (1). The determination of Fe was performed by a spectrophotometric method.

A third method used a centrifuge furnace in which the sample was first superheated and then equilibrated under rotation for 2 to 2.5 h. The capsule was cooled during rotation. The upper part of the solidified K was used for the spectrophotometric analysis.

SOURCE AND PURITY OF MATERIALS:

12Kh18N10T steel: ~ 70 % Fe, 18.2 % Cr, 9.1 % Ni, 1.5 % Mn, 0.05 % C, 0.8 % Si, 0.02 % S, 0.3 % Ti, and 0.035 % P.

K: with contents of $(3\text{-}4)\cdot10^{-5}$ % Fe, $(7\text{-}10)\cdot10^{-4}$ % O, $(3\text{-}5)\cdot10^{-4}$ % C, $(2\text{-}4)\cdot10^{-6}$ % H.

ESTIMATED ERROR:

Solubility: detection limit 10^{-6} - 10^{-5} mass % Fe.

Temperature: stability ± (3-5) K.

REFERENCES:

1. Popov, R.G.; Raikova, G.P.; Samsonov, N.V.; Tarbov, A.A. *USSR Pat. no. 319871, Bull. coll. no. 33,* 1971.

COMPONENTS: (1) Iron; Fe; [7439-89-6] (2) Rubidium; Rb; [7440-17-7]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1989

CRITICAL EVALUATION:

Young and Arabian (1) studied the dissolution of Haynes-25 alloy in liquid Rb in the temperature range 811 to 1200 K. The Fe concentration in Rb increased from $3.8 \cdot 10^{-3}$ to $8.0 \cdot 10^{-3}$ mol % Fe. The value at 1033 K, however, was $1.8 \cdot 10^{-2}$ mol % Fe, and deviated clearly from the scatter band of results. The data of this study may be influenced by interactions of Fe with other contaminants in Rb. The equilibrium concentration of Fe in Rb at higher temperatures was only slightly higher than the initial Fe content in the Rb sample. The apparent solubility of Fe in Rb has, therefore, to be considered as doubtful. The low chemical activity of Fe in the Haynes-25 alloy caused an equilibration at much lower activity than unity.

The probable sequence of the solubilities of the components of steels may be concluded from corrosion test of stainless steels in liquid Rb. Fe seems to be less soluble from type 304 stainless steel than Cr and Ni in the temperature range 773 to 892 K in Rb with contents of about 2.7 mol % O, according to Pinchback et al. (2). Similar corrosion tests of Suzuki et al. (3) indicated a better solubility of Fe than of Ni, Mo, and Cr from type 316 stainless steel in Rb with comparably high contents of O (22 mol % O). These corrosion tests are not directly comparable since the temperatures and contents of O in Rb differ.

The saturated solution of Fe in liquid Rb contaminated with O is in equilibrium with $Rb_6Fe_2O_6$ (4). The equilibrium solid phase should be Fe in the absence of sufficient quantities of O, since Rb is insoluble in Fe and does not form intermetallics with Fe (5). A Fe-Rb phase diagram was not reported. It should be similar to that of the Fe-K system, differing in the boiling and melting points of Rb at 961 and 312.6 K, respectively.

The doubtful values of the solubility of Fe in liquid Rb

T/K	*soly*/mol % Fe	source	remarks
1073	$4 \cdot 10^{-3}$ [a]	(1)	at elevated pressure
1300	$6 \cdot 10^{-3}$ [a]	(1)	at elevated pressure
1473	$8 \cdot 10^{-3}$ [b]	(1)	at elevated pressure

[a] α Fe [b] β Fe

References

1. Young, P.F.; Arabian, R.V. *US Atom.Ener.Comm. Rep. AGN-8063*, 1962.
2. Pinchback, T.R.; Winkel, J.R.; Matlock, D.K.; Olson, D.L. *Nucl. Technol.* 1981, *54*, 201.
3. Suzuki, T.; Ohno, K.; Masuda, S.; Nakanishi, Y.; Matsui, Y. *J. Nucl. Mater.* 1987, *148*, 230.
4. Gadd, P.G.; Borgstedt, H.U. *Z. Anorg. Allg. Chem.* 1985, *527*, 187.
5. Wever, F. *Arch. Eisenhüttenw.* 1928-29, *2*, 739; *Naturwissens.* 1929, *17*, 304.

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Rubidium; Rb; [7440-17-7]

ORIGINAL MEASUREMENTS:

Young, P.F.; Arabian, R.V.

US Atom.Ener.Comm. Rep. AGN-8063, 1962.

VARIABLES:

Temperature: 811-1200 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubility of Fe (as component of the alloy Haynes-25) in liquid Rb at some temperatures was reported; the values were read out from the figure and recalculated to mol % by the compilers.

t/°F	T/K	*soly*/mass % Fe	*soly*/mol % Fe
1000	811	$2.5 \cdot 10^{-3}$	$3.8 \cdot 10^{-3}$
1400	1033	$3.2 \cdot 10^{-3}$, $4.6 \cdot 10^{-3}$, $1.2 \cdot 10^{-2}$	$4.9 \cdot 10^{-3}$, $7.0 \cdot 10^{-3}$, $1.8 \cdot 10^{-2}$
1700	1200	$3.3 \cdot 10^{-3}$, $7.2 \cdot 10^{-3}$	$5.0 \cdot 10^{-3}$, $1.1 \cdot 10^{-2}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An annealed sample of alloy Haynes-25 and a Ta capsule were purified in a mixture of HNO_3, H_2SO_4, HF and H_2O (2:2:1:5), rinsed with H_2O and dried in air. The capsule was loaded with the sample and Rb, closed by welding under Ar atmosphere, flame sprayed with Al_2O_3 and heated for 50 h to the equilibration temperature. The capsule was then inverted to let the solution flow into a Ta sampling cup. The capsule was cooled and opened, the cup with the solidified solution was taken for analysis. The Rb sample was treated with anhydrous hexane, then with CH_3OH, H_2O and HCl. The cup was treated with aqua regia, and this solution was added to the first one. The whole solutions were taken to dry. The solid residue was analyzed for Fe in the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

Haynes-25: 1.7 % Fe, 49.6 % Co, 20.4 % Cr, 15.3 % W, 10.1 % Ni, 1.4 % Mn, 0.1 % C, 0.4 % Si, 0.02 % P, 0.015 % S, from Superior Tube Co..
Rb: purified by filtration, gettering with Ti-Zr alloy at 866 K and vacuum distilled; with contents of (6-17)$\cdot 10^{-4}$ % O and about $3.8 \cdot 10^{-3}$ mol % Fe.

ESTIMATED ERROR:

Solubility: detection limit $1 \cdot 10^{-4}$ mass % Fe; analytical error ± 10 %.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Iron; Fe; [7439-89-6] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1989

CRITICAL EVALUATION:

Boiling Cs does not attack Fe, as was observed by (1), and no measurable solubility of Cs in solid Fe was detected (2). Tepper and Greer (3) investigated the dissolution of Haynes-25 alloy in Cs at 1255 K. The amount of Fe determined in solution decreased with the time of equilibration from $2.1 \cdot 10^{-3}$ to $< 2.4 \cdot 10^{-4}$ mol % Fe. It is very difficult to evaluate these tests which are characterized by a low Fe content of the alloy (1.7 mass % Fe) and unstable values of the concentration of Fe in solution. The same authors (5) reported that the solubility of Fe in Cs at 368 to 533 K is higher than of Mo and lower than of Ni. Experimental details were, however, not reported.

Godneva et al. (4) determined the solubility of Fe in Cs in the temperature range 323-873 K by measurements of the concentration of Fe in the diffusion layer and the bulk of Cs. The data in the bulk of Cs were one order of magnitude higher than in the diffusion layer, and both decreased with temperature. An extrapolation of the data in the diffusion layer of (4) to high temperatures resulted in values close to the upper limit of the results of (3) at 1255 K.

A slight increase of the Fe content in Cs was reported (6) as the result of contacting stainless steel with Cs for 500 h at 673 K. Numerical results were, however, not given. Holley et al. (7) analyzed Cs after equilibration with several Fe alloys at 875, 675 K, or room temperature. The amounts of Fe were between 0.05 and 0.5 mol %; experimental details were not presented.

Corrosion tests of stainless steels which were immersed in liquid Cs for 500 h at 673 K indicated oxidation of their surfaces without significant dissolution (8). Similar conclusions were reported by Sedelnikov and Godneva (9), who studied the behavior of Armco Fe in Cs at 873 K. Experiments of Keddy (10) with stainless steel specimens did not result in changes of their masses and surface appearances after 100 h at 773 K. A good resistance of Fe was reported even after contacting it with Cs at 1273 K for 450 h (11). Different conclusions from the results which were obtained may certainly be due to different levels of O in Cs.

Lindemer et al. (12) postulated the formation of the compound Cs_2FeO_2 as the corrosion product of Fe in Cs with contents of O. This compound is likely the solid phase in equilibrium with the saturated solution. The formation of this compound may influence the level of solubility of Fe in Cs and its dependence on temperature.

A Fe-Cs phase diagram was not yet established. It should be similar to that shown for the Fe-K system; the boiling temperature of Cs is at 944 K.

The doubtful values of the solubility of Fe in liquid Cs containing < 0.08 mol % O

T/K	*soly*/mol % Fe	source
323	$7 \cdot 10^{-3}$	(4)
373	$4 \cdot 10^{-3}$	(4)
423	$2.5 \cdot 10^{-3}$	(4)
573	$1.1 \cdot 10^{-3}$	(4) extrapolated
873	$5 \cdot 10^{-4}$	(4) extrapolated
1273	$3 \cdot 10^{-4}$ [a]	(3),(4) extrapolated

[a] for γ Fe at elevated pressure to keep Cs in the liquid state

References

1. Ruff, O.; Johansen, O. *Ber. Deutsch. Chem. Ges.* 1905, *38*, 3602.
2. Wever, F. *Arch. Eisenhüttenw.* 1928-29, *2*, 739; *Naturwissens.* 1929, *17*, 304.
3. Tepper, F.; Greer, J. *US Air Force Rep. ASD-TDR-63-824*, 1963,Pt. I; *Rep. MSAR-63-61* 1963.
4. Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.
5. Tepper, F.; Greer, J. *US Air Force Rep. AFML-TR-66-280*, 1966; as cited in Berry, W.E., *Corrosion in Nuclear Applications*, Wiley, N.Y., 1971, p. 300.
6. Hughes Aircraft Comp., *NASA Rep. SP-41*, 1964, p. 243.
7. Holley, J.H.; Neff, G.R.; Weiler, F.B.; Winslow, P.M. *Electric Propulsion Development*, E. Stuhlinger, Ed., Academic, N.Y., 1963, p. 341.
8. Winslow, P.M. *US Atom. Ener. Comm. Rep. CONF-650411*, 1965, p. 334.
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11. Anonymous, *US Atom. Ener. Comm. Rep. GEST-2035*, 1964, Vol. I, p. III-101.
12. Lindemer, T.B.; Besmann, M.; Johnson, C.E. *J. Nucl. Mater.* 1981, *100*, 178.

COMPONENTS:	**ORIGINAL MEASUREMENTS:**
(1) Iron; Fe; [7439-89-6] (2) Cesium; Cs; [7440-46-2]	Tepper, F.; Greer, J. *US Air Force Rep. ASD-TDR-63-824*, Pt.I, 1963; *Rep. MSAR-63-61* 1963.
VARIABLES:	**PREPARED BY:**
One temperature: 1255 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The equilibrium concentration of Fe (as component of the alloy Haynes-25) in liquid Cs at 1800 °F was reported to be $9 \cdot 10^{-4}$, $< 6 \cdot 10^{-4}$, and $< 1 \cdot 10^{-4}$ mass % Fe after 10, 100, and 1000 h of exposure, respectively. The corresponding values, calculated by the compilers, are $2.1 \cdot 10^{-3}$, $< 1.4 \cdot 10^{-3}$, and $< 2.4 \cdot 10^{-4}$ mol % Fe. The amount of Fe which was determined after the equilibration was at a similar level as the content in the Cs samples before the test.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A test capsule was composed of the lower part made of Haynes-25 alloy and the upper made of alumina. The capsule was heated in vacuum for 100 h at 1255 K, filled with Cs to be equilibrated with the Haynes-25 and closed by means of welding under Ar atmosphere. After the equilibration of 10 to 1000 h the capsule was inverted and cooled in dry ice. The solidified Cs in the alumina part was dissolved in CH_3OH. The crucible was then cleaned with HCl, and the two solutions were combined. This combined solution was boiled to dryness and submitted to emission spectrographic analysis for the determination of the components.

SOURCE AND PURITY OF MATERIALS:

Haynes-25: 3.7 % Fe, 50 % Co, 20 % Cr, 15 % W, 10 % Ni, 2.7 % Mn, $5.1 \cdot 10^{-2}$ % C, $3.76 \cdot 10^{-2}$ % N, and $2.01 \cdot 10^{-2}$ % O.
Cs: 99.9+ % pure from Mine Safety Appliances Research, further purified with Zr turnings at elevated temperature; with contents of $2.8 \cdot 10^{-3}$ % C, $1.2 \cdot 10^{-3}$ % O, $< 2 \cdot 10^{-4}$ % N and about $1 \cdot 10^{-3}$ % Fe.
Ar: purified by means of passing through a K-Na bubbler.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:

(1) Iron; Fe; [7439-89-6]

(2) Cesium; Cs; [7440-46-2]

ORIGINAL MEASUREMENTS:

Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S.

Zh. Prikl. Khim. 1974, *47*, 2177-2180.

VARIABLES:

Temperature: 323-873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Fe in liquid Cs was reported.

t/°C	*soly*/mass % Fe	*soly*/mol % Fe [a]
50	$2.7\cdot10^{-3}$ [c]	$6.4\cdot10^{-3}$ [c]
100	$1.7\cdot10^{-3}$ [c]; $1.35\cdot10^{-2}$ [b]	$4.0\cdot10^{-3}$ [c]; $3.2\cdot10^{-2}$ [b]
150	$1.1\cdot10^{-3}$ [c]; $1.24\cdot10^{-2}$ [b]	$2.6\cdot10^{-3}$ [c]; $2.9\cdot10^{-2}$ [b]
200	$4.7\cdot10^{-3}$ [b]	$1.11\cdot10^{-2}$ [b]
300	$1.6\cdot10^{-3}$ [c]; $4.0\cdot10^{-3}$ [b]	$3.8\cdot10^{-3}$ [c]; $9.4\cdot10^{-3}$ [b]
600	$1.2\cdot10^{-3}$ [b]	$2.8\cdot10^{-3}$ [b]

[a] as calculated by the compilers
[b] determined in the bulk of Cs
[c] determined in the diffusion layer

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Fe specimen was immersed in Cs under vacuum and equilibrated for 120 h in a glass ampoule. No attack on the glass was observed after the solubility tests. Cs was then cooled and dissolved in H_2O, and its amount was determined by acidimetric titration of a portion of the solution. Another portion of the primary solution was treated with hydroxyloamine and dipyridile to complex Fe which was determined by means of colorimetric measurement (1).

SOURCE AND PURITY OF MATERIALS:

Fe: Armco.
Cs: 98-99 % pure, further purified by vacuum distillation, with final contents of < 0.01 % O and < 1.5 % Rb as main impurities.

ESTIMATED ERROR:

Nothing specified.

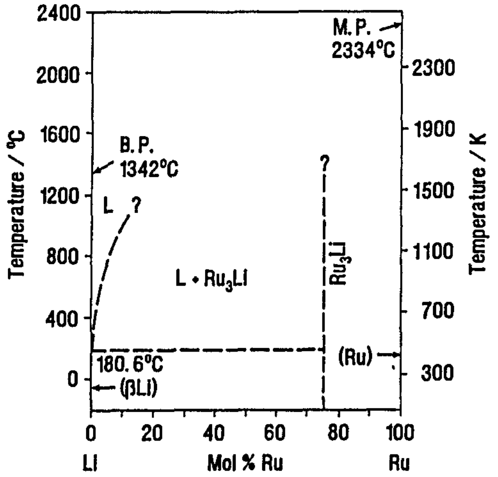
REFERENCES:

1. Godneva, M.M.; Vodyannikova, R.D. *Zh. Anal. Khim.* 1965, *20*, 831-835.

COMPONENTS:	EVALUATOR:
(1) Ruthenium; Ru; [7440-18-8] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1989

CRITICAL EVALUATION:

Liquid Li did not attack a Ru sample in a corrosion test of 6 h at temperatures up to 573 K (1). However, both the metals reacted completely at 723 K to form an intermediate phase of the approximate formula Ru_3Li (2). Quantitative data concerning the solubility of Ru in liquid Li cannot be suggested. The reaction of the two metals at 573 K needs much more time to get observable effects of alloying and to reach the true equilibrium in the system. A predicted Ru-Li phase diagram is shown in the figure.



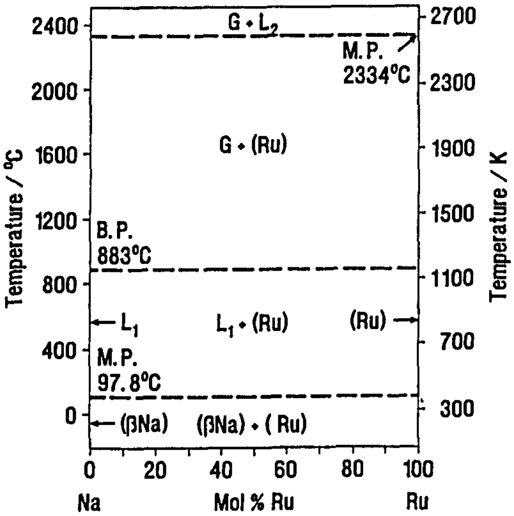
References

1. Rhys, D.W.; Price, E.G. *Met. Ind.* 1964, *105*, 243.
2. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.

COMPONENTS:	EVALUATOR:
(1) Ruthenium; Ru; [7440-18-8] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1989

CRITICAL EVALUATION:

Liquid Na did not attack a Ru sample in a corrosion test of 6o h duration at temperatures of 473 and 623 K (1). No reaction between the two metals was observed at temperatures below 973 K, and no symptoms of an alloying were detectable at higher temperatures (2). Aleksandrov and Dalakova (3) found also no dissolution of Ru in liquid Na after an equilibration for 1 h at 973-1023 K, the detection limit of their spectral analytical method was not reported. Therefore, it may be assumed that the solubility of Ru in liquid Na is negligible at temperatures up to 973 K. A speculative phase diagram of the Ru-Na system is sketched in the figure.



References

1. Rhys, D.W.; Price, E.G. *Met. Ind.*, 1964, *105*, 243.
2. Loebich, O.; Raub, C.J. *Platin. Met. Rev.*, 1981, *25*, 113.
3. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad.Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Ruthenium; Ru; [7440-18-8]

(2) Potassium; K; [7440-09-7]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
March 1989

CRITICAL EVALUATION:

An attack of liquid K on Ru at 473 and 623 K after a contact of 60 h duration could not be detected (1). A similar observation at 773 K was published by (2). Aleksandrov and Dalakova (3) did not find any dissolution of Ru in liquid K after an equilibration for 1 h at 873-923 K, the detection limit of their spectral analytical method was not reported. These qualitative statements indicate a very low Ru solubility in liquid K.
The Ru-K phase diagram should be similar to the Ru-Na phase diagram shown in the figure.

References

1. Rhys, D.W.; Price, E.G. *Met. Ind.*, 1964, *105*, 243.
2. Loebich, O.; Raub, C.J. *Platin. Met. Rev.*, 1981, *25*, 113.
3. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad.Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Ruthenium; Ru; [7440-18-8]

(2) Rubidium; Rb; [7440-17-7]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
March 1989

CRITICAL EVALUATION:

An interaction between Ru and liquid Rb at elevated temperatures was not reported in (1). However, a detailed description of this investigation was not provided. The Ru solubility is assumed to be very low, as can be concluded from its similarity with the Ru-K system.
The Ru-Rb phase diagram should be similar to the Ru-Na phase diagram shown in the figure, the boiling temperature of Rb is 961 K.

References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.

COMPONENTS:

(1) Ruthenium; Ru; [7440-18-8]

(2) Cesium; Cs; [7440-46-2]

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
March 1989

CRITICAL EVALUATION:

Experimental data on the Ru solubility in or compatibility with liquid Cs were not reported. However, the Ru solubility in liquid Cs is assumed to be negligible, as can be concluded in analogy with the solubilities in Na, K, and Rb. Similar conclusions may be drawn when comparing the predicted thermodynamic data of the dissolution process of Ru in these alkali metals (1). The Ru-Cs phase diagram should be similar to the Ru-Na phase diagram shown in the figure. The boiling temperature of Cs is 944 K.

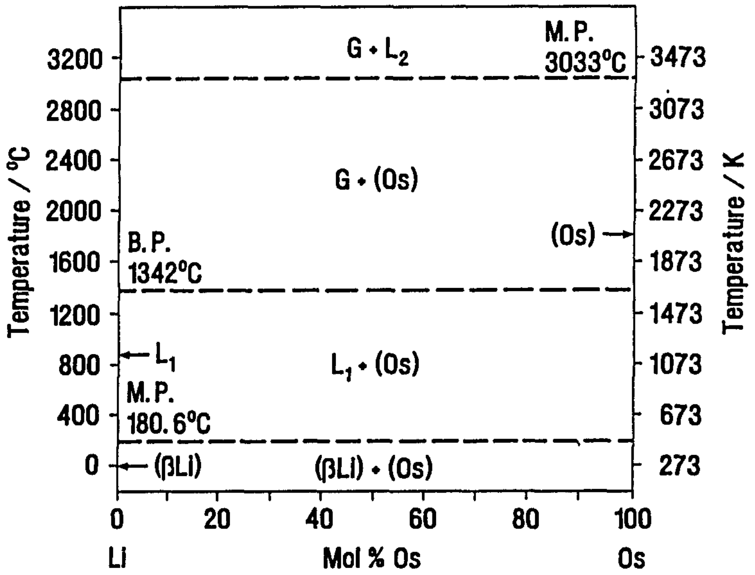
References

1. Niessen, A.K.; deBoer, F.R.; Boom, R.; deChâtel, P.F.; Mattens, W.C.M.; Miedema, A.R. *CALPHAD* 1983, *7*, 51.

COMPONENTS:	EVALUATOR:
(1) Osmium; Os; [7440-04-2] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1991

CRITICAL EVALUATION:

Loebich and Raub (1) did not detect any reaction between Os and liquid Li below 1173 K. A reaction with the container material occurred at higher temperatures. One can conclude from these observations that the solubility of Os in liquid Li should be lower than that of Mo (the container material) or that Os and Mo may react to form a Mo-Os intermetallic compound. Intermetallics or intermediate phases were not detected in the Os-Li system; a phase diagram sketched in (2) is shown in the figure.



References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.
2. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am.Soc.Mater., Materials Park, 1990, p. 2642.

COMPONENTS:	EVALUATOR:
(1) Osmium; Os; [7440-04-2] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1989

CRITICAL EVALUATION:

Os was not attacked by liquid Na at temperatures below 973 K. At higher temperatures the evaporation of Na was the only observable effect (1). It can be concluded that the solubility of Os in liquid Na should be extremely low. The Os-Na phase diagram may be expected to be similar to that of the Os-Li system.

References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.

COMPONENTS:	EVALUATOR:
(1) Osmium; Os; [7440-04-2] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1989

CRITICAL EVALUATION:

Os did not react with liquid K at temperatures up to 1073 K (1). A very low solubility of Os in liquid K is, therefore, expected. The Os-K system should be characterized by a phase diagram analogous to that of the Os-Li system. The boiling point of K is at 1032 K.

References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.

COMPONENTS:	EVALUATOR:
(1) Osmium; Os; [7440-04-2] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1989

CRITICAL EVALUATION:

No details concerning the investigation of an interaction between Os and Rb at elevated temperatures were reported in (1). It may be assumed in analogy with the system Os-K that the solubility of Os in liquid Rb is very low. The Os-Rb phase diagram should be similar to that of the Os-Li system. The boiling temperature of Rb is at 961 K.

References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.

COMPONENTS:	EVALUATOR:
(1) Osmium; Os; [7440-04-2] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1989

CRITICAL EVALUATION:

Experiments on the solubility of Os in liquid Cs or the compatibility of the two metals have not been reported so far. It may, however, be assumed in analogy with the systems Os-Li, Na, K, Rb that the solubility of Os in liquid Cs is very low. This statement is supported by the comparisons of predicted enthalpies of solution of Os in these alkali metals (1). The Os-Cs phase diagram should be similar to that of the Os-Li system. The boiling temperature of Cs is at 944 K.

References

1. Niessen, A.K.; deBoer, F.R.; Boom, R.; deChâtel, P.F.; Mattens, W.C.M.; Miedema, A.R. *CALPHAD* 1983, *7*, 51.

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Volume 34	A.N. Paruta and R. Piekos, *4-Aminobenzenesulfonamides. Part I: Non-cyclic Substituents*
Volume 35	A.N. Paruta and R. Piekos, *4-Aminobenzenesulfonamides. Part II: 5-membered Heterocyclic Substituents*
Volume 36	A.N. Paruta and R. Piekos, *4-Aminobenzenesulfonamides. Part III: 6-membered Heterocyclic Substituents and Miscellaneous Systems*
Volume 37	D.G. Shaw, *Hydrocarbons with Water and Seawater. Part I: Hydrocarbons C5 to C7*
Volume 38	D.G. Shaw, *Hydrocarbons with Water and Seawater. Part II: Hydrocarbons C8 to C36*

Volume 39	C.L. Young, *Cumulative Index: Volumes 20-38*
Volume 40	J. Hala, Halides, *Oxyhalides and Salts of Halogen Complexes of Titanium, Zirconium, Hafnium, Vanadium, Niobium and Tantalum*
Volume 41	C.-Y. Chan, I.N. Lepeshkov and K.H. Khoo, *Alkaline Earth Metal Perchlorates*
Volume 42	P.G.T. Fogg and W. Gerrard, *Hydrogen Halides in Non-aqueous Solvents*
Volume 43	R.W. Cargill, *Carbon Monoxide*
Volume 44	H. Miyamoto, E.M. Woolley and M. Salomon, *Copper and Silver Halates*
Volume 45/46	R.P.T. Tomkins and N.P. Bansal, *Gases in Molten Salts*
Volume 47	R. Cohen-Adad and J.W. Lorimer, Alkali Metal and Ammonium Halides in Water and Heavy Water (Binary Systems)
Volume 48	F. Getzen, G. Hefter and A. Maczynski, *Esters with Water. Part I: Esters 2-C to 6-C*
Volume 49	F. Getzen, G. Hefter and A. Maczynski, *Esters with Water. Part II: Esters 7-C to 32-C*
Volume 50	P.G.T. Fogg, *Carbon Dioxide in Non-aqueous Solvents at Pressures Less Than 200 kPa*
Volume 51	J.G. Osteryoung, M.M. Schneider, C. Guminski and Z. Galus, *Intermetallic Compounds in Mercury*
Volume 52	I. Lambert and H.L. Clever, *Alkaline Earth Hydroxides in Water and Aqueous Solutions*
Volume 53	C.L. Young, *Cumulative Index: Volumes 40-52*
Volume 54	W.E. Acree, Jr., *Polycyclic Aromatic Hydrocarbons in Pure and Binary Solvents*
Volume 55	S. Siekierski and S.L. Phillips, *Actinide Nitrates*
Volume 56	D. Shaw, A. Skrzecz, J.W. Lorimer and A. Maczynski, *Alcohols with Hydrocarbons*
Volume 57	W. Hayduk, *Ethene*
Volume 58	W.E. Acree, Jr., *Polycyclic Aromatic Hydrocarbons: Binary Non-aqueous Systems, Part I: Solvents A-E*
Volume 59	W.E. Acree, Jr., *Polycyclic Aromatic Hydrocarbons: Binary Non-aqueous Systems, Part II: Solvents F-Z*
Volume 60	A.L. Horvath and F.W. Getzen, *Halogenated Methanes with Water*
Volume 61	C.-Y. Chan, K.H. Khoo, E.S. Gryzlova and M.-T. Saugier-Cohen Adad, *Alkali Metal and Ammonium Perchlorates, Part I: Lithium and Sodium Perchlorates*
Volume 62	P. Scharlin, *Carbon Dioxide in Water and Aqueous Electrolyte Solutions*
Volume 63	H.U. Borgstedt and C. Guminski, *Metals in Liquid Alkali Metals, Part I: Be to Os*
Volume 64	H.U. Borgstedt and C. Guminski, *Metals in Liquid Alkali Metals, Part II: Co to Bi*