INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

ANALYTICAL CHEMISTRY DIVISION
COMMISSION ON SOLUBILITY DATA

SOLUBILITY DATA SERIES

Volume 64

METALS IN LIQUID ALKALI METALS
PART II: Co to Bi

SOLUBILITY DATA SERIES

INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY
IUPAC SECRETARIAT: Bank Court Chambers, 2-3 Pound Way
Templars Square, Cowley, Oxford 0X4 3YF, UK

SOLUBILITY DATA SERIES

Volume 64

METALS IN LIQUID ALKALI METALS

PART II: Co to Bi

Volume Editors

Hans Ulrich Borgstedt
Forschungszentrum Karlsruhe GmbH
Institut für Materialforschung III
Karlsruhe, Germany

Cezary Guminski
Department of Chemistry
University of Warsaw
Warsaw, Poland

Contributors

H.U. Borgstedt

C. Guminski

OXFORD UNIVERSITY PRESS

IUPAC Solubility Data Series Rates for 1996

Subscriptions:	UK and Europe	Rest of World
Full subscription	£280	$426
Single volume	£80	$120

Reduced rates are available to members of IUPAC. Please apply directly to the publisher for details.

Prices include postage by surface mail or, for subscribers in the USA and Canada by Airfreight or in Japan, India, Australia and New Zealand by Air Speeded Post. Airmail rates are available on request.

The IUPAC Solubility Data Series (ISSN 0191-5622) is published quarterly in March, June, September and December by Oxford University Press, Oxford, UK. Annual subscription price is US$426. The IUPAC Solubility Data Series is distributed by M.A.I.L. America, 2323 Randolph Avenue, Avenel, New Jersey 07001, USA. Periodical postage paid at Newark, New Jersey, USA and additional entry points.

US POSTMASTER: send address changes to IUPAC Solubility Data Series, c/o M.A.I.L. America, 2323 Randolph Avenue, Avenel, New Jersey 07001, USA. This issue date is September 1996.

New subscriptions may be registered at any time during the year but will be reckoned as running from January 1st of the year in which the subscription is received. Claims regarding non-receipt of issues must be received within 4 months of publication or date of order, whichever is later. Back issues are available—for information contact Journals Customer Services Department, Oxford University Press, Great Clarendon Street, Oxford OX2 6DP.

Order Information

Payment is required with all orders and may be made by the following methods:
Cheque (made payable to Oxford University Press)
National Girobank (Account 500 1056)
Credit Card (Access, Visa, American Express, Diners Club)
Direct Debit: Please send for further details
UNESCO Coupons
Bankers: Barclays Bank plc, PO Box 333, Oxford
Code 20-65-18. Account 00715654
Please send orders to: Journals Subscription Department, Oxford University Press, Walton Street, Oxford OX2 6DP. Tel: +44(0)1865 556767, Fax: +44(0)1865 267485

Any enquires regarding this publication should be addressed to:
USA and Canada: Oxford University Press, 2001 Evans Road, Cary, NC 27513, USA.
Rest of World: Journals Customer Services Department, Oxford University Press, Great Clarendon Street, Oxford OX2 6DP, UK.

Printed and bound in Great Britain by Antony Rowe Ltd, Chippenham, Wiltshire

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:

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

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

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

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

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

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

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 $w(1)$:

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

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, $x_{v,1}$:

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

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, $x_{v,1}$:

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

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.

REFERENCES

1. Hill, E.A. *J. Am. Chem. Soc.* 1900, *22*, 478.
2. IUPAC Commission on Atomic Weights and Isotopic Abundances. *Pure Appl. Chem.* 1989, *63*, 975.
3. Mills, I.; *et al.*, eds. *Quantities, Units and Symbols in Physical Chemistry* (the *Green Book*). Blackwell Scientific Publications. Oxford, UK. 1993.
4. Ku, H.H., p. 73; Eisenhart, C., p. 69; in Ku, H.H., ed. Precision Measurement and Calibration. *NBS Special Publication 300.* Vol. 1. Washington. 1969.
5. Gold, V.; et al., eds. *Compendium of Analytical Nomenclature* (the *Gold Book*). Blackwell Scientific Publications. Oxford, UK. 1987.
6. Freiser, H.; Nancollas, G.H., eds. *Compendium of Analytical Nomenclature* (the *Orange Book*). Blackwell Scientific Publications. Oxford, UK. 1987. Sect. 9.1.8.
7. ISO Standards Handbook, *Quantities and Units*, International Standards Organization, Geneva, 1993.
8. German Standard, DIN 1310, *Zusammensetzung von Mischphasen*, Beuth Verlag, Berlin, 1984.
9. Cvitaš, T. *Chem. International* 1995, *17*, No. 4, 123.
10. Robinson, R.A.; Stokes, R.H. *Electrolyte Solutions*. Butterworths. London. 1959. 2nd ed.
11. Jänecke, E. *Z. Anorg. Chem.* 1906, *51*, 132.
12. Friedman, H.L. *J Chem. Phys.* 1960, *32*, 1351.
13. Lorimer, J.W. in Cohen-Adad, R.; Lorimer, J.W. *Alkali Metal and Ammonium Chlorides in Water and Heavy Water (Binary Systems).* IUPAC Solubility Data Series, Vol. 47. Pergamon Press. Oxford, UK, 1991. p. 495.

R. Cohen-Adad
Villeurbanne, France

J.W. Lorimer
London, Ont., Canada

M. Salomon
Sea Bright, NJ, USA

M.-T. Saugier-Cohen Adad
Villeurbanne, France

December, 1995

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	$\dfrac{1}{1+\dfrac{M_i}{M_c}\left\{\dfrac{1}{w_i}-1+\sum\limits_{j\neq 1}^{c-1}\left(\dfrac{M_c}{M_j}-1\right)\dfrac{w_j}{w_i}\right\}}$	$\dfrac{1}{1+\dfrac{1}{m_i M_i}\left(1+\sum\limits_{j\neq i}^{c-1} m_j M_j\right)}$	$\dfrac{1}{1+\dfrac{1}{M_c}\left(\dfrac{\rho}{c_i}-M_i\right)+\sum\limits_{j\neq i}^{c-1}\dfrac{c_j}{c_i}\left(1-\dfrac{M_j}{M_c}\right)}$
$w_i =$	$\dfrac{1}{1+\dfrac{M_c}{M_i}\left\{\dfrac{1}{x_i}-1+\sum\limits_{j\neq i}^{c-1}\left(\dfrac{M_j}{M_c}-1\right)\dfrac{x_j}{x_i}\right\}}$	w_i	$\dfrac{1}{1+\dfrac{1}{m_i M_i}\left(1+\sum\limits_{j\neq i}^{c-1} m_j M_j\right)}$	$\dfrac{c_i M_i}{\rho}$
$m_i =$	$\dfrac{1}{M_c\left(\dfrac{1}{x_i}-1-\sum\limits_{j\neq i}^{c-1}\dfrac{x_j}{x_i}\right)}$	$\dfrac{1}{M_i\left(\dfrac{1}{w_i}-1-\sum\limits_{j\neq i}^{c-1}\dfrac{w_j}{w_i}\right)}$	m_i	$\dfrac{1}{\dfrac{1}{c_i}\left(\rho-\sum\limits_{j\neq i}^{c-1} c_j M_j\right)-M_i}$
$c_i =$	$\dfrac{\rho}{M_i+M_c\left\{\dfrac{1}{x_i}-1+\sum\limits_{j\neq i}^{c-1}\left(\dfrac{M_j}{M_c}-1\right)\dfrac{x_j}{x_i}\right\}}$	$\dfrac{\rho w_i}{M_i}$	$\dfrac{\rho}{\dfrac{1}{m_i}\left(1+\sum\limits_{j\neq i}^{c-1} m_j M_j\right)+M_j}$	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 the 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 of 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 above that in the absence of these complexing agents although 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 second part of the collection of evaluated experimental solubility data of about seventy metallic elements in five alkali metals solvents (Li, Na, K, Rb, Cs). The systems were ordered following the (IUPAC accepted) long periodic table in respect to the solutes and following the elements number of the solvent elements. The mutual binary systems of the alkali metals are briefly treated in the first pages of the first 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 of users in the form of assessed phase diagrams. The detailed discussion 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. The Chemical Abstracts, Nuclear Science Abstracts and Atom Index were used as primary sources for the reference search of solubility data. It was soon discovered that the key word *solubility* was not sufficient to extract complete solubility information from the corresponding literature. Therefore, also entries related to this subject were fruitfully 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 assess anew all experimental results available. If not otherwise stated, the DATA SHEETS were prepared directly from the original papers. Any secondary sources were used only occasionally if original reports were not accessible. This way was always clearly indicated.

The systems of mercury with alkali metals have already been treated in volume 25 of the Solubility Data Series, "Metals in Mercury", by C. Hirayama, Z. Galus, and C. Guminski. New experimental material was only discovered for the Hg-Na system, and improved our understanding of the phase relations in this system. Alloys of transition metals are often used as solutes in solubility studies. The apparent solubility is related in such cases to the chemical activity of the saturating element in the alloy which is typically below unity. Due to the technical importance of the K-Na eutectic alloy some solubility studies of elements of structural materials in the liquid alloy have been performed. These data are also included in this volume.

Solubility data are sometimes measured under constrained pressure, since the vapor 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.

Compositions of equilibrium solid phases are not discussed in detail in this monograph in order to avoid a repetition of the discussions in (7,8). The composition of a solid phase is very seldom determined in such systems in which no intermetallic compounds are formed. If those determinations are performed, it is either mentioned in a DATA SHEET or CRITICAL EVALUATION of the system. If a soluble metal forms ternary compounds with the solvent and a contaminating non-metal as potential solute, this fact is 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 is frequently estimated from thermal analysis experiments of selected alloy compositions. In the case of metallic systems such estimation is not precise. We omit 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 can estimate solidus lines using the selected phase diagrams in the case that it might be necessary. We decided not to place values on phase diagrams since the size of the figures does not lead to clarity, especially where the symbols overlap.

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 regions rich in alkali metals. Further recommended liquidus data may either be read from the phase diagrams or be extracted from the corresponding DATA SHEETS.

Quite frequently, important solubility data are only graphically reported, and they have been 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 %.

References:

1. Borgstedt, H.U. and Mathews, C.K. *Applied Chemistry of the Alkali Metals*, Plenum, N.Y., 1987, 282 p.
2. Addison, C.C. *The Chemistry of the Liquid Alkali Metals*, Wiley, Chichester, 1984, 325 p.
3. *Material Behavior and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, 450 p.
4. *Liquid Metal Systems*, H.U. Borgstedt and G. Frees, Eds., Plenum, N.Y., 1995, 420 p.
5. Claar, T.D. *Reactor Technol.* 1970, *13*, 124.
6. Anthrop, D.F. *U.S. Atom. Ener. Comm. Rep. UCRL-50315*, 1967.
7. Hansen, M.; Anderko, K. *Constitution of Binary Alloys*, McGraw-Hill, N.Y. 1958, 1250 p; Elliott, R.P. First Supplement, 1965, 950 p; Shunk, F.A. Second Supplement, 1969, 850 p.
8. Massalski, T.B. Ed., *Binary Alloy Phase Diagrams*, Amer. Soc. Mater., Materials Park, 1990, 3500 p; extended evaluations of several individual systems are published in *Bull. Alloy Phase Diagr.* 1980-1990, *1-11* and *J. Phase Equil.* 1991-1996, *12-17*.

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
May 22, 1996

COMPONENTS: (1) Cobalt; Co; [7440-48-4] (2) Lithium; Li; [7439-93-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1991

CRITICAL EVALUATION:

Kuzin et al. (1,2) reported the results of determinations of the solubility of Co in liquid Li. Specific details and individual data were not given. X-ray absorption spectrometry was applied for these measurements. It was the same method which was also used to determine the solubilities of V, Cr, Fe, and Ni in liquid Li with reliable results (see the corresponding systems). The primary measurements were performed in the temperature range 923 to 1073 K (1) and later extended to 823 K (2). The authors summarized their results in a fitting equation:

$$\log(soly/\text{mol \% Co}) = (3.149 \pm 0.627) - (5901 \pm 627)(T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

The data of the solubility of Co at 823 and 1073 K calculated on the basis of this equation were $9.5 \cdot 10^{-5}$ and $4.5\ 10^{-3}$ mol % Co, respectively. The theoretically predicted solubility of Co in liquid Li (1,2) is in rough agreement with these values.

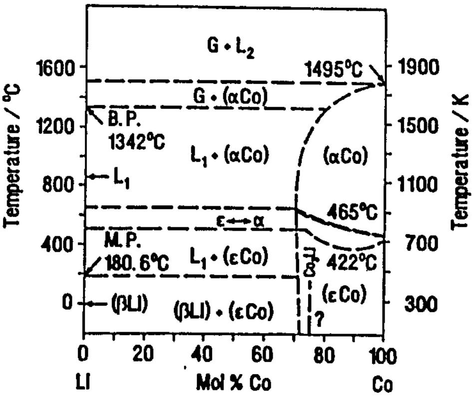
The testing of the corrosion resistance of Co in liquid Li resulted in controversial conclusions. Wilhelm (3) reported a poor corrosion behaviour of Co in liquid Li at temperatures above 473 K. Bonnemay et al. (6) heated Li in Co crucibles at 783 and 1003 K for 11 to 80 min. They observed a quite rapid penetration along grain boundaries in Co and suggested the formation of a compound Co_3Li. Magee (7) observed the formation of an unidentified phase between Co and Li during 66 h of heating at 1173 K. On the other hand, Hoffman (4) stated a very good corrosion resistance of Co (similar to Be, Cr, Re, Y) at temperatures up to 1089 K under static conditions for 100 h.

Li influences the $\epsilon \leftrightarrow \alpha$ allotropic transitions of solid Co (5) and (6), but the solid solubility of Li in Co which was suggested in (5) to be about 30 mol % Li is rather suspect. The solubility of an order of 0.1 mol % Li which was found by (6) is more probable, as stated by Sangster and Pelton (8). The composition of the solid phase in equilibrium with the liquid needs a decisive test, whether it is Co_3Li or Co saturated with Li.

A doubtful phase diagram of the Co-Li system is presented in the figure.

Tentative values of the solubility of α Co in liquid Li

T/K	$soly$/mol % Co	source
873	$2.5\ 10^{-4}$	Eq.(1)
973	$1.2 \cdot 10^{-3}$	Eq.(1)
1073	$4.5 \cdot 10^{-3}$	Eq.(1)



References

1. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Materialy dla Atomnoi Tekhniki*, Energoatomizdat, Moskva, 1983, p. 33
2. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113.
3. Willhelm, C. *Mater. Design Engin.* 1963, *58*, 97.
4. Hoffman, E.E. *U.S Atom.Ener.Comm. Rep. ORNL-2924*, 1960.
5. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 775.
6. Bonnemay, M.; Levart, E.; Bronoel, G.; Peslerbe, G.; Savy, M. *Mem. Sci. Rev. Metall.* 1965, *62*, 285.
7. Magee, C.B. *U.S Atom.Ener.Comm. Rep. DRI-2139*, 1964.
8. Sangster, J.; Pelton, A.D. *Bull. Alloy Phase Diagr.* 1990, *11*, 440.

COMPONENTS:	EVALUATOR:
(1) Cobalt; Co; [7440-48-4] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1995

CRITICAL EVALUATION:

Cockcroft (1) reported qualitative data on the low solubility of Co in liquid Na. Aleksandrov and Dalakova (2) did not observe any dissolution of Co in liquid Na at 973 to 1023 K after 1 h equilibration of the metals; the detection level of the spectral analysis was not specified. Grand et al. (3) were the first to determine the solubility in the temperature range 638-798 K. The data indicated a minimum at 698 K, which could be related to the $\alpha \leftrightarrow \epsilon$ transformation of Co at 695 K (according to the compilers). The result at 638 K is probably overestimated. It is possible that the radioactivity of impurities may increase the apparent ^{60}Co activity measurements. The solubility results obtained by Lee and Berkey (4) in the temperature range 562-817 K were significantly scattered. The mean values were about one order of magnitude higher than those reported in (3) and did not show any distinct temperature dependence. Some results of Eichelberger and McKisson (5) decreased irregularly with temperature from $3.6\ 10^{-3}$ to $8\ 10^{-5}$ mol % Co at 873 to 1173 K. These results could not be explained, since no complication of the Co-Na system by any impurity was identified. The most comprehensive experiments were performed by Thompson et al. (6,7) in the temperature range 673 to 973 K, who determined the Co solubility in liquid Na using either a Co foil in an alumina-lined Ni crucible or Co on a Ni plated crucible. The Co solubility results obtained by the second method seem to be more reliable, than for the first method; the Co activity could be decreased by an interaction with Ni. The slopes of the temperature dependence of the solubility in (6,7) determined by each method seem to be too low. Sivasubramanian et al. (8) performed a few measurements in the temperature range 673-873 K. These results at a level of 10^{-5} mol % Co did not show any distinct temperature dependence. Since a ternary oxide of Co was not found to be stable in Na at 923 K and adjacent temperatures (9,10), a higher slope of the temperature dependence of the solubility was expected to be closer to the theoretical prediction of Kuzin et al. (11):

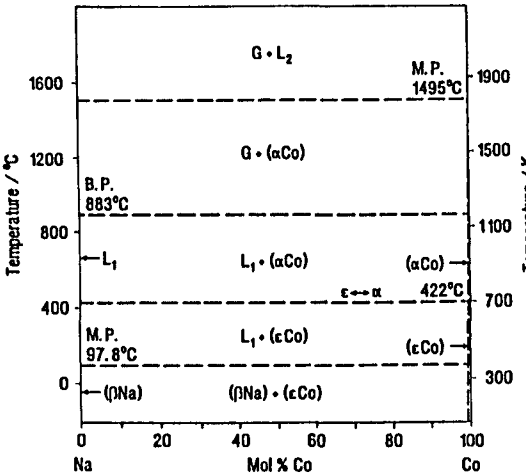
$$\log (soly/\text{mol \% Co}) = 3.98 - 6873\ (T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

The data of (6,7) and (8) show a discrepancy over two orders of magnitude; the average solubility line of all results, however, would be situated close to the theoretical equation (1). Therefore, the results of (3) obtained at 698 and 798 K are regarded as the most plausible, since they are in between. These data are also close to the predicted solubility from Eq.(1).

The saturated solution of Co in liquid Na is likely to be in equilibrium with almost pure α or ϵ Co, since formation of intermetallics in the system was not reported (12). A schematic phase diagram is presented below.

Tentative value of the Co solubility in liquid Na

T/K	$soly$/mol % Co	source	remarks
698	$8 \cdot 10^{-7}$	(3)	ϵ Co
798	$4\ 10^{-5}$	(3)	α Co



References

1. Cockcroft, J.D. *Atom.Ener.Res.Establ. Rep. D/M-6*, 1952.
2. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.
3. Grand, J.A.; Baus, R.A.; Bogard, A.D.; Williams, D.D.; Lockhard, L.B.; Miller, R.R. *J.Phys. Chem.* 1959, *63*, 1192.
4. Lee, P.K.; Berkey, E. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt.I, 1969, p. 299.
5. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm.Rep. AI-AEC-12955*, 1970.
6. Stanaway, W.P.; Thompson, R. *Material Behaviour and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 421.
7. Pellett, C.R.; Thompson, R. *Liq. Met. Engin. and Technol.*, BNES, London, 1984, *3*, p. 43.
8. Sivasubramanian, K.; Mitragotri, D.S.; Bhat, N.P. *Proceed. of NUCAR 95, Kalpakkam*, S.G. Kulkarni, S.B. Manohar, D.D. Sood, Eds., Bhabha Atomic Research Centre, Bombay, 1995, p. 262.
9. Barker, M.G.; Fairhall, G.A.; Frankham, S. *US Dep.Ener. Rep. CONF-800401*, 1980, p. 18/41.
10. Bhat, N.P.; Borgstedt, H.U. *Werkst.Korros.*, 1988, *39*, 115.
11. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113.
12. Sangster, J.; Pelton, A.D. *Bull. Alloy Phase Diagr.* 1990, *11*, 442.

COMPONENTS:

(1) Cobalt; Co; [7440-48-4]

(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*, 1192-1194.

VARIABLES:

Temperature: 638-798 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Co in liquid Na was determined.

t/°C	*soly*/mass % Co	*soly*/mol % Co [a]
365	$2.8 \cdot 10^{-6}$	$1.09 \cdot 10^{-6}$
425	$2.1 \cdot 10^{-6}$	$8.2 \cdot 10^{-7}$
525	$1.00 \cdot 10^{-4}$	$3.9 \cdot 10^{-5}$

[a] calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility determinations were performed by equilibration of Na in a closed Co vessel. Na was directly distilled and filtered into the vessel in an Ar atmosphere. The apparatus was sealed and transferred into a controlled furnace. The selected temperature was maintained for 24 hours, Na was permanently stirred. After equilibration the apparatus was opened. The Na sample was removed by means of a quartz pipette. Each Na sample was transferred into an irradiation facility together with a Co monitor sample. Subsequent radiochemical analysis of the Na and monitor samples was the basis of the calculation of the Co solubility in liquid Na.

SOURCE AND PURITY OF MATERIALS:

Co: unspecified.
Na: distilled, filtered, containing $(1\text{-}2) \cdot 10^{-3}$ % O.
Ar: unspecified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Cobalt; Co; [7440-48-4]

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

ORIGINAL MEASUREMENTS:

Lee, P.K.; Berkey, E.

US Atom.Ener.Comm. Rep. ANL-7520, Pt.I, 1969, 299-308.

VARIABLES:

Temperature: 562-817 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Co solubility in Na at some few temperatures was reported:

t/°C	*time*/h	*soly*/10^{-4} mass % Co	*soly*/10^{-5} mol % Co
289	24	0.31, 0.52, 0.19, 0.22, 0.07	1.2, 2.0, 0.74, 0.86, 0.27
347	5	1.6, 14.4, 12.2, 3.7, 2.7, 1.6, 1.1, 1.2, 0.13, 0.16, 0.11	6.2, 54, 47, 14, 10.5, 6.2, 4.3, 4.7, 0.51, 0.62, 0.43
427	4	0.29, 0.28, 0.23, 0.28, 0.12, 6.6, 3.6	1.13, 1.09, 0.89, 1.09, 0.47, 26, 14
544	4	0.49, 0.20, 0.43, 0.15, 0.14	1.9, 0.78, 1.7, 0.58, 0.55

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test apparatus consisted of a Mo capsule with a Co solute crucible and a Ta collector crucible fixed together with W springs. The parts were rinsed with H_2O, acetone and CH_3OH and outgassed under vacuum at 773 and 873 K for several hours. The apparatus was then transferred to an Ar box and heated for another hour at 423 K. The crucible was filled with Na and the apparatus was closed. The capsule was placed in a vacuum chamber and heated for the desired time. The temperature was controlled by means of two Pt/Pt-Rh(105) thermocouples. The chamber was inverted after equilibration to force the Na saturated with Co to flow into the collector. The chamber was quenched by means of spraying with liquid N. The capsule was then transferred to the Ar box and opened. Na was melted in the collector and a sample was taken by means of a stainless steel tube. The Co content of the Na samples was analysed by spark-source spectrography in Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Co: 99.992 % pure, supplied by Material Research Corp.
Ta: 99.997 % pure, supplied by Material Research Corp.
Na: "high purity", supplied by MSA Research Corp., containing $1.4 \cdot 10^{-3}$ % O (authors), $<5 \cdot 10^{-3}$ % C, $<2 \cdot 10^{-2}$ % K, $<3 \cdot 10^{-6}$ % Co, $<1.8 \cdot 10^{-3}$ % Cr, Mn, Fe, Ni (all together).
Ar: "high purity", containing $<1 \cdot 10^{-4}$ % H_2O, $<2 \cdot 10^{-4}$ % O_2.

ESTIMATED ERROR:

Solubility: analytical precision ± (5-10) %.
Temperature: stability ± 1 K.

REFERENCES:

COMPONENTS:

(1) Cobalt; Co; [7440-48-4]

(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 Co in liquid Na was determined.

t/°C	*soly*/mass % Co	*soly*/mol % Co [a]
600	$9.4\cdot10^{-3}$	$3.6\cdot10^{-3}$
700	$4\cdot10^{-4}$	$1.6\cdot10^{-4}$
800	$2\cdot10^{-4}$	$8\cdot10^{-5}$
900	$2\cdot10^{-4}$	$8\cdot10^{-5}$ [b]

[a] as calculated by the compilers [b] at slightly elevated pressure

A final conclusion was presented by the authors that the solubility of Co in liquid Na was lower than $5\cdot10^{-4}$ mass % Co (or $2\cdot10^{-4}$ mol % Co, as calculated by the compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Co test crucible was cleaned in a mixture of HNO_3, H_2SO_4, H_2O (5:3:2) and a few drops of HF, and finally washed in H_2O and acetone. A sample collector was made of Nb-Zr(1%). The crucible collector assembly was degassed at 623 K for 2 h. The crucible was then loaded with Na. The assembly was sealed by welding under high vacuum. A capsule surrounded the assembly which was equilibrated at the desired temperature for 6 h in an Ar atmosphere. The capsule was then inverted in order to transfer the Na into the collector. The assembly was opened after cooling and Na was analyzed for its Co content by means of atomic absorption spectroscopy.

SOURCE AND PURITY OF MATERIALS:

Co: 99.99 % pure with contents of $4.0\cdot10^{-3}$ % C, $1.6\cdot10^{-4}$ % H, $5.0\cdot10^{-3}$ % O, $9\cdot10^{-4}$ % N, from Material Research Corp. (electron beam zone refined).
Na: 99.996 % pure, with contents of $8\cdot10^{-4}$ % C and $(0.6\text{-}4)\cdot10^{-4}$ % O.

ESTIMATED ERROR:

Solubility: the results at 1073 and 1173 K were slightly above the analytical sensitivity limit ($4\cdot10^{-5}$ mol % Co).
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Cobalt; Co; [7440-48-4]

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

ORIGINAL MEASUREMENTS:

Pellett, C.R.; Thompson, R.

Liq. Met. Engng. Technol., BNES, London, 1984, *3*, 43-48.

VARIABLES:

Temperature: 673-973 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Co solubility in liquid Na was determined using a Co solute in form of a foil and a plated can.

	foil		can	
t/°C	*soly*/mass % Co	*soly*/mol % Co [a]	*soly*/mass % Co	*soly*/mol % Co [a]
400	$3.1 \cdot 10^{-7}$	$1.2\ 10^{-7}$	$1.08 \cdot 10^{-6}$	$4.2 \cdot 10^{-7}$
	-	-	$1.4 \cdot 10^{-5}$	$5.4 \cdot 10^{-6}$
	-	-	$4.7 \cdot 10^{-7}$	$1.8\ 10^{-7}$
	-	-	$3.8 \cdot 10^{-7}$	$1.5 \cdot 10^{-7}$
450	$4.9 \cdot 10^{-7}$	$1.9 \cdot 10^{-7}$	$7.3 \cdot 10^{-7}$	$2.8 \cdot 10^{-7}$
500	$3.8 \cdot 10^{-7}$	$1.5 \cdot 10^{-7}$	$4.6 \cdot 10^{-7}$	$1.8 \cdot 10^{-7}$
	-	-	$1.37 \cdot 10^{-6}$	$5.3 \cdot 10^{-7}$
550	$7.1 \cdot 10^{-7}$	$2.7\ 10^{-7}$	$1.15\ 10^{-6}$	$4.5\ 10^{-7}$
	-	-	$1.92 \cdot 10^{-6}$	$7.5 \cdot 10^{-7}$
600	$3.4 \cdot 10^{-7}$	$1.3 \cdot 10^{-7}$	$1.23 \cdot 10^{-5}$	$4.8 \cdot 10^{-6}$
	-	-	$3.5 \cdot 10^{-6}$	$1.3 \cdot 10^{-6}$
	-	-	$6.4 \cdot 10^{-7}$	$2.5 \cdot 10^{-7}$
	-	-	$1.89\ 10^{-6}$	$7.4 \cdot 10^{-7}$
650	$4.6\ 10^{-6}$	$1.8 \cdot 10^{-6}$	$2.3 \cdot 10^{-6}$	$9.0 \cdot 10^{-7}$
	-	-	$8.4 \cdot 10^{-6}$	$3.3 \cdot 10^{-6}$
700	$1.3\ 10^{-6}$	$5.1 \cdot 10^{-7}$	-	-

[a] calculated by the compilers.

The results obtained when using a can were expressed by the fitting equation, which had been proved by the compilers.

$$\log (soly/\text{mol \% Co}) = -4.31 - 1493\ (T/\text{K})^{-1}$$

The results gained by atomic absorption spectrometry were also reported in a previous study (1), but were neglected in the study being compiled here.

t/°C	400	450	500	550	650	700
soly/mass % Co[a]	$4\ 10^{-5}$	$5\ 10^{-6}$	$8 \cdot 10^{-5}$	$5\ 10^{-5}$	$1.3 \cdot 10^{-4}$	$4 \cdot 10^{-5}$
soly/mol % Co[b]	$1.6 \cdot 10^{-5}$	$2 \cdot 10^{-6}$	$3 \cdot 10^{-5}$	$2 \cdot 10^{-5}$	$5 \cdot 10^{-5}$	$1.6\ 10^{-5}$

[a] read out from the figure (by the compilers) [b] calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Two different equilibration methods were applied. For the first one, an activated ^{60}Co foil placed inside a Ni crucible was used, to which an alumina liner had been fitted. For the second method, a Ni crucible internally electroplated with a 0.5 mm Co plate was applied. The plate was bonded to Ni by heating to 973 K for 24 hours in dry H_2. The crucible was irradiated and then stored for 2 weeks. According to (1), the crucibles were filled with Na and a Ni lid was welded onto the crucible in an Ar atmosphere. The cans were heated at the temperature of interest for 45 hours in a Cu furnace block. A Na sample was taken by piercing the can lid with a Mo needle and withdrawing Na through a 1.5 µm mean pore Ni frit at negative pressure. It was collected in a weighed alumina crucible, which had previously been cleaned in aqua regia.

The Co in the sample was analysed by scintillation counting of ^{60}Co. The whole sample in the alumina crucible was counted and subsequently dissolved in CH_3OH. Some samples were further analysed by atomic absorption spectrometry. A Co carrier was added to the CH_3OH solution of other samples. These were re-counted as aqueous solutions after co-precipitation with $La(OH)_3$, separation and re-solution in HCl.

SOURCE AND PURITY OF MATERIALS:

Co: foil - unspecified; can - electrolytic purity.

Na: "reactor grade" purity, containing $(1-2) \cdot 10^{-3}$ % O.

Ar: "high purity".

ESTIMATED ERROR:

Solubility: analytical scatter less than ± 12 % (foil) and ± 5 % (can).

Temperature: nothing specified.

REFERENCES:

1. Stanaway, W.P.; Thompson, R. *Material Behaviour and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 421.

COMPONENTS:

(1) Cobalt; Co; [7440-48-4]

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

ORIGINAL MEASUREMENTS:

Sivasubramanian, K.; Mitragotri, D.S.; Bhat, N.P.

Proceed. of NUCAR 95, Kalpakkam, S.G. Kulkarni, S.B. Manohar, D.D. Sood, Eds., Bhabha Atomic Research Centre, Bombay, 1995, p. 262-263.

VARIABLES:

Temperature: 673-873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Co in liquid Na was determined. The results were presented in a figure. The numerical values were communicated by the authors to the compilers.

t/°C	*soly*/mass % Co	*soly*/mol % Co [a]
400	$2.36\cdot10^{-5}$; $4.84\cdot10^{-5}$	$9.21\ 10^{-6}$; $1.89\cdot10^{-5}$
450	$1.82\cdot10^{-5}$	$7.11\cdot10^{-6}$
500	$2.04\ 10^{-5}$	$7.97\cdot10^{-6}$
600	$5.47\cdot10^{-5}$; $5.52\cdot10^{-5}$	$2.14\ 10^{-5}$; $2.16\ 10^{-5}$

[a] calculated by the compilers

The authors formulated the equation of the solubility of Co, its validity was tested by the compilers:

$$\log(soly/\text{mol \% Co}) = -3.961 - 701.8(T/\text{K})^{-1}$$

The slope of this equation is definitely to low.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus for the equilibration of the metals was fabricated from AISI 304 stainless steel. The essential part of it consisted of outer and inner vessels. These were heated, and the temperature was measured by means of a Chromel/Alumel thermocouple located in a thermowell outside of the vessels. The vessel parts were degreased with NaOH, derusted with HF-HNO_3, demineralized H_2O and finally with acetone. The dried vessels were placed in an Ar atmosphere glove box. A Co plate was placed in the vessel, Na was added into the outer vessel. The inner vessel was introduced into the outer one. The complete set-up was taken out of the box and connected to the Ar gas line. The pressure in the inner vessel was kept slightly above that of the outer vessel. After 48 h of equilibration at the desired temperature, Na was forced to filtrate into the inner vessel which had been evacuated. The set-up was then cooled and dismantled. Co was determined by means of AAS after dissolving the Na sample in H_2O acidified with HNO_3.

SOURCE AND PURITY OF MATERIALS:

Co: nothing specified.
Na: "nuclear grade, purified and filtered.
Ar: "high purity"

ESTIMATED ERROR:

Solubility: precision between ± 0.5 and ± 50 %; detectivity level of the analysis 3 ng Co per cm^3.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Cobalt; Co; [7440-48-4] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

Swisher (1) reported the Co solubility in liquid K to be below the detection limit of $3 \cdot 10^{-4}$ mol % Co at either 1144 or 1328 K. The author presumes the true solubility of Co to be not much below the given limit.
Aleksandrov and Dalakova (3) did not detect any solubility of Co in liquid K at 873 to 923 K after an equilibration of 1 h; they did not specify the detection limit of the spectral analysis.
Smales (2) analysed the Co content in a K-Na melt (which is assumed to be the eutectic mixture) used as a reactor coolant. The determined Co concentration of $1\ 10^{-6}$ mol % may be regarded as a reliable result, taking into consideration that the liquid alloy was saturated with Co and the temperature of the coolant was about 873 K. The Co concentration value was lower than the one which was determined in liquid Na. This is in agreement with the assumption that an addition of K to Na may decrease the solubility. The study by Smales (2) is not compiled, as the method of radiochemical analysis was only briefly described and further details were not provided.
The predicted phase diagram of the Co-K system should be similar to that shown for the Co-Na system.

The tentative value of the Co solubility limit in liquid K at 1144 K is $3 \cdot 10^{-4}$ mol % Co.

References

1. Swisher, J.H. *NASA Rep. TN-D-2734*; 1965; *US Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 43.
2. Smales, A.A. *Peaceful Uses of Atomic Energy*, U.N., New York, 1956, *9*, 273.
3. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Cobalt; Co; [7440-48-4] (2) Potassium; K; [7440-09-7]	Swisher, J.H. *NASA Rep. TN-D-2734*; 1965.
VARIABLES:	**PREPARED BY:**
Temperature: 1144 and 1328 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Co in liquid K was found to be below $5 \cdot 10^{-4}$ mass % ($3 \cdot 10^{-4}$ mol % as calculated by the compilers) at both temperatures, 1144 and 1328 K. Some traces of dissolved Co were detectable, the analyses were, however, not sufficiently accurate to get defined values.
The same results were reported in the abstract (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A test capsule was made of Nb; a sampling cup of Mo was used. K was equilibrated in a Co cup which was enclosed inside the capsule. After loading with K the capsule was sealed by means of electron-beam welding. The whole containment was heated in a high-vacuum furnace for several hours. The temperature of the capsule holder was measured by means of Pt/Pt-Rh(13%) thermocouples. The sampling procedure was performed at the test temperature by inverting the furnace. Thus, the K sample flowed into the sampling cup.

The capsule was cut open after cooling to room temperature. K was dissolved in butyl alcohol, and the cup was leached with HCl in order to remove precipitates which may have formed during the cooling.

Co was determined by means of a colorimetric method in the combined solutions. The K content of the alcoholic solution was gravimetricly determined.

SOURCE AND PURITY OF MATERIALS:

Co: 99.99 % pure.
K: containing < $2.0 \cdot 10^{-3}$ % O.

ESTIMATED ERROR:

Solubility: determination at the detection level.
Temperature: precision ± 3 K, stability ± 3 K.

REFERENCES:

1. Swisher, J.H. *US Atom. Ener. Comm. Rep. CONF-650411*, 1965, p. 43.

COMPONENTS:

(1) Cobalt; Co; [7440-48-4]

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

ORIGINAL MEASUREMENTS:

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

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

VARIABLES:

Temperature: 1033-1200 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubility of Co (as a constituent of Haynes-25 alloy) in liquid Rb was presented in a figure; the data were read out and recalculated to mol % by the compilers. The experiments were performed at elevated pressure to keep Rb in the liquid state.

t/°F	T/K	*soly*/mass % Co	*soly*/mass % Co
1400	1033	$<1 \cdot 10^{-4}$	$<1.4 \cdot 10^{-4}$
1700	1200	$<1\ 10^{-4}$; $1.8 \cdot 10^{-4}$	$<1.4 \cdot 10^{-4}$; $2.6 \cdot 10^{-4}$

COMMENTS AND ADDITIONAL DATA:

These results may be considered as tentative data, since no other compatibility tests are not reported, and the content of Co in the alloy is high. The chemical activity of Co in the alloy does not differ much from the activity of unalloyed Co.

The Co-Rb phase diagram is not available; it should be analogous to the Co-Na phase diagram.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The annealed sample of Haynes-25 and the Ta crucible were cleaned in HNO_3, H_2SO_4, HF, and H_2O mixture, rinsed with H_2O and dried in air. The capsule was loaded with the sample and Rb, and closed by means of welding in Ar atmosphere. It was then flame sprayed with Al_2O_3 and heated for 50 h at the equilibration temperature. The capsule was inverted to let the solution flow into the Ta sampling cup. This cup was cooled to room temperature and opened after solidification of the sample. The Rb sample was treated with anhydrous hexane, then with CH_3OH, H_2O, and HCl. The cup was treated with aqua regia and the solution was added to the first one. The resulting solutions were taken to dryness. The residue was analyzed for its Co content in the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

Haynes-25: 49.6% Co, 20.4 % Cr, 15.3 % W, 10.1 % Ni, 1.7 % Fe, 1.4 % Mn, 0.4 % Si, 0.1 % C, 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 a content of $(6-17) \cdot 10^{-4}$ % O.

ESTIMATED ERROR:

Solubility: detection limit of $1 \cdot 10^{-4}$ mass % Co; analytical precision ± 10 %.

Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Cobalt; Co; [7440-48-4] (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:

Tepper and Greer (1) investigated the solubilities of the components of Haynes-25 alloy (with ~50 mass % Co) in liquid Cs at 1255 K. The Co concentrations in Cs changed with the time of equilibration which was between 10 and 1000 h. The concentration finally approached was $2.7 \cdot 10^{-3}$ mol % Co. Since the Co content of the Haynes-25 alloy is higher than 50 mol %, the determined solubility might be considered as close to the real saturation value at unit Co activity.
This result was qualitatively confirmed in compatibility studies of Winslow (2,3) at 673 K. After 500 h of contacting Co alloy with liquid or vaporized Cs, no increase of the Co concentration in Cs could be observed. Numerical values were not reported.
The Co-Cs phase diagram should be similar to the phase diagram of the Co-Na system.

Tentative value of the solubility of Co in liquid Cs at elevated pressure to keep Cs as liquid.

T/K	*soly*/mol % Co	source
1255	$3 \cdot 10^{-3}$	(1)

References

1. Tepper, F.; Greer, J. *US Air Force Rep. ASD-TDR-63-824*, Part I, 1963; Rep. *MSAR-63-61*, 1963.
2. Winslow, P.M. *US Atom.Ener.Comm.Rep. CONF-650411*, 1965, p. 334.
3. Winslow, P.M. *Corrosion*, 1965, *21*, 341.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Cobalt; Co; [7440-48-4] (2) Cesium; Cs; [7440-46-2]	Tepper, F.; Greer, J. *US Air Force Rep. ASD-TDR-63-824*, 1963, Part I; Rep. *MSAR-63-61*, 1963.
VARIABLES:	**PREPARED BY:**
One temperature: 1255 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The equilibrium composition of Co in liquid Cs after equilibrating Haynes-25 alloy with the liquid metal at 1800 °F was reported to be $1.25 \cdot 10^{-2}$, $<6 \cdot 10^{-4}$ and $1.2 \cdot 10^{-3}$ mass % Co after 10, 100 and 1000 h of exposure, respectively. The corresponding values expressed in mol % Co are $2.8 \cdot 10^{-2}$, $<1.3\ 10^{-3}$ and $2.7 \cdot 10^{-3}$, as calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A test capsule was constructed of two parts: the bottom made of Haynes-25, and the top of alumina. The capsule was heated in vacuum for 100 h at 1255 K, then filled with Cs and closed by means of welding in an Ar atmosphere. The capsule was kept at the equilibration temperature in upright position for 10 to 1000 h, after completion of the equilibration the capsule was inverted and cooled in dry ice. The solidified Cs in the alumina sampler was dissolved in CH_3OH and the crucible cleaned with HCl. The two solutions were combined and heated to dryness. The residue was analyzed by quantitative emission spectrography.

SOURCE AND PURITY OF MATERIALS:

Haynes-25: 50 % Co, 20 % Cr, 15 % W, 10 % Ni, 1.7 % Fe, 0.05 % C, 0.038 % N, 0.0201 % O.
Cs: 99.9+ % pure from Mines Safety Appliances Research, further purified by means of contacting with Zr turnings at elevated temperature; final contents of impurities were: $2.8\ 10^{-3}$ % C, $1.2\ 10^{-3}$ % O, $<2\ 10^{-4}$ % N and $1 \cdot 10^{-3}$ % Fe.
Ar: purified by passing through a K-Na bubbler.

ESTIMATED ERROR:

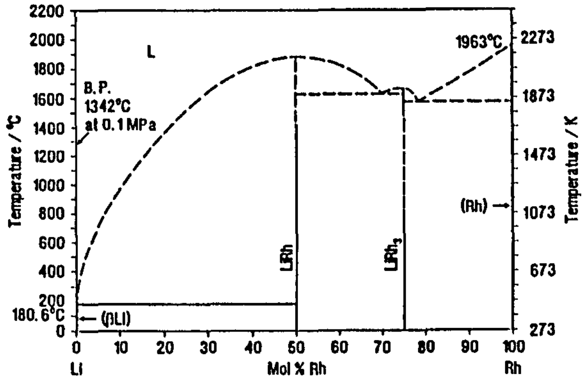
Solubility: nothing specified.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Rhodium; Rh; [7440-16-6] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1992

CRITICAL EVALUATION:

Rh and Li reacted at ~ 573 K, and the intermetallic compounds RhLi and Rh_3Li were formed as was concluded from the molar ratio of the components in the tests (1). Since RhLi showed thermal stability even above 1073 K (2), the corresponding liquidus line has to be placed significantly higher than for the Pd-Li system. The solubility of Rh in liquid Li should, therefore, be lower than of Pd. The information given by Hahn and Jaworsky (3) that Rh plated on Mo was highly resistant to liquid Li at elevated temperature seems to be incorrect in relation to the results of Loebich and Raub (1). Wheat et al. (5) performed thermal analyses with Rh-Li alloys; they did not report liquidus data. Sangster and Pelton (4) estimated an eutectic point at ~ 0.1 mol % Rh and 453.3 K on the basis of the results of (5). The schematic phase diagram, shown in the figure, was suggested by (4).



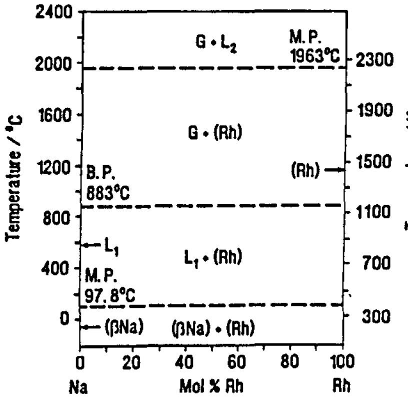
References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.
2. Sidhu, S.S.; Anderson, K.D.; Zauberis, D.D. *Acta Cryst.* 1965, *18*, 906.
3. Hahn, H.; Jaworsky, M. *Metall. Soc. Conf.* 1966, *30*, 547.
4. Sangster, J.; Pelton, A.D. *J. Phase Equil.* 1991, *12*, 682.
5. Wheat, H.G.; Cheng, C.Y.; Baynzick, R.J.; Sullivan, R.W.; Magee, C.B. *J.Less-Common Met.* 1978, *58*, P13.

COMPONENTS:	EVALUATOR:
(1) Rhodium; Rh; [7440-16-6] (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:

Rh and Na did not react below 973 K. Na evaporated from the reaction mixture at higher temperatures indicating no reduction of its vapour pressure (1). A very low solubility of Rh in liquid Na at temperatures ≤ 973 K and no formation of intermetallics has to be expected. Aleksandrov and Dalakova (2) did not observe any dissolution of Rh in liquid Na after an equilibration of 1 h at 973 to 1023 K; the detection limit of the spectral analysis used was not specified. A predictive Rh-Na phase diagram is shown in the figure.



References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.
2. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Rhodium; Rh; [7440-16-6] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

According to Loebich and Raub (1) Rh was not attacked by liquid K at temperatures up to 773 K, and the solubility of Rh in liquid K is expected to be negligible up to this temperature. Aleksandrov and Dalakova (2) did not detect any traces of Rh dissolved in liquid K after an equilibration of 1 h at 873 to 923 K; the detection limit of the the spectral analysis used was not specified.
The Rh-K phase diagram should be similar to that which is shown for the Rh-Na system. It differs in the boiling point of K at 1032 K.

References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.
2. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Rhodium; Rh; [7440-16-6] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

Loebich and Raub (1) performed experiments similar to those for the Rh-K system. However, conclusions were not provided. The solubility of Rh in liquid Rb is not expected to be higher than in liquid K.
The Rh-Rb phase diagram should be similar to that which is shown for the Rh-Na system.

References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.

COMPONENTS:	EVALUATOR:
(1) Rhodium; Rh; [7440-16-6] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

According to Berry (1), who reported on compatibility tests of Rh and Cs, Rh was not attacked after a 500 hours exposure to Cs at 1023 K. It might be assumed that the solubility of Rh in liquid Cs should be negligible at this temperature.
The Rh-Cs phase diagram should be similar to that of the Rh-Na system shown in the critical evaluation.

References

1. Berry, W.E. *Corrosion in Nuclear Applications*, Wiley, N.Y., 1971, p. 304.

COMPONENTS:

(1) Iridium; Ir; [7439-88-5]

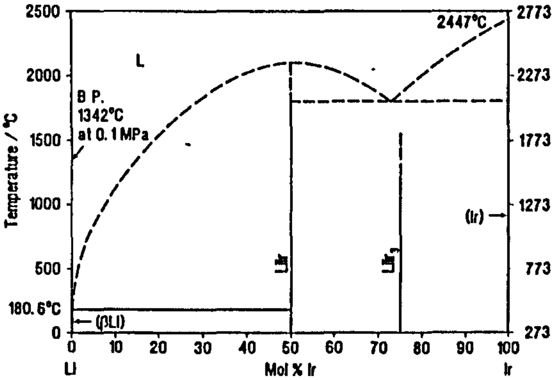
(2) Lithium; Li; [7439-93-2]

EVALUATOR:

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

CRITICAL EVALUATION:

Liquid Li did not attack Ir samples in corrosion tests of 6 h duration at 513 and 663 K (1). Loebich and Raub (2) reported, however, that a reaction between the two metals occurred at 773 and 813 K. The intermetallics IrLi and Ir_3Li were found as stable compounds. IrLi was also prepared by Varma et al. (3) by direct combination of the metals. Rapidly heated IrLi shows a melting point of about 2390 K; details of the experimental procedure were not presented. The solubility of Ir in liquid Li was not investigated; it is probably lower than of Pd at the same temperature, since Pd-Li compounds melt at lower temperatures than IrLi.
The predicted Ir-Li phase diagram after (4) is shown below. Sangster and Pelton (4) estimated that the liquid phase saturated with Ir contains less than 0.1 mol % Ir at the eutectic point on the basis of analogy to the Rh-Li system.



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. Varma, S.K.; Chang, F.C.; Magee, C.B. *J. Less-Common Met.* 1978, *60*, P47.
4. Sangster, J.; Pelton, A.D. *J.Phase Equil.* 1992, *13*, 59.

COMPONENTS:

(1) Iridium; Ir; [7439-88-5]

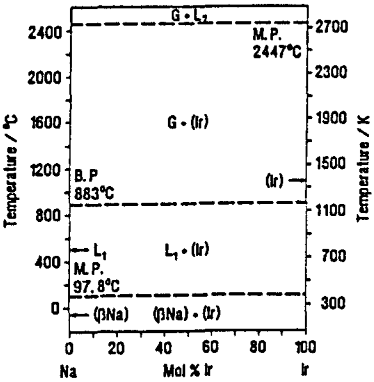
(2) Sodium; Na; [7440-23-5]

EVALUATOR:

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

CRITICAL EVALUATION:

Ir samples did not react with liquid Na at 423 and 573 K (1) as well as at 973 K (2). At still higher temperatures Na evaporated from the reaction capsule, thus indicating no reduction of its vapour pressure (2). It may be concluded that the solubility of Ir in liquid Na up to temperatures of 923 K should be negligibly small. Aleksandrov and Dalakova (3) did not detect any dissolution of Ir in liquid Na after a contact of the metals for 1 h at 973-1023 K. The detection limit of the spectral analysis used was not specified.
A schematic phase diagram of the Ir-Na system 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.
3. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Iridium; Ir; [7439-88-5] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

Ir samples were not attacked by liquid K at 384 and 533 K (1) nor at 773 K (2). It is to be expected that the solubility of Ir in liquid K should be very small up to 773 K. Even at 873-923 K no dissolution of Ir in liquid K was observed after contact of the metals for 1 h. The detection limmits of the methods used in (1-3) were not reported.
The Ir-K phase diagram should be similar to that of the Ir-Na system.

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:	EVALUATOR:
(1) Iridium; Ir; [7439-88-5] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

No conclusions were drawn from compatibility tests of Ir with liquid Rb by Loebich and Raub (1) who performed experiments similar to those of the Ir-K system. The solubility of Ir in liquid Rb is to be expected to be not larger than in liquid K.
The Ir-Rb phase diagram should be similar to that of the Ir-Na system.

References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.

COMPONENTS:	EVALUATOR:
(1) Iridium; Ir; [7439-88-5] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland May 1989

CRITICAL EVALUATION:

A negligible solubility of Ir in liquid Cs should be expected as in the other alkali metals (1). Winslow et al. (2,4) studied the compatibility of Pt-Ir(10%) alloy with liquid Cs at a temperature of 673 K. The alloy was less attacked than pure Pt; the significant dissolution of the alloy was probably due to the leaching of Pt (see Pt-Cs system).
It is likely that the Ir-Cs phase diagram is similar to that of the Ir-Na system.

References

1. Loebich, O.; Raub, C.J. *Platin. Met. Rev.* 1981, *25*, 113.
2. Holley, J.H.; Neff, G.R.; Weiler, F.B.; Winslow, P.M. *Electric Propulsion Development*, E. Stuhlinger, Ed., Academic, N.Y., 1963, p. 341.
3. Winslow, P.M. *US Atom.Ener.Comm.Rep. CONF-650411*, 1965, p. 334.
4. Winslow, P.M. *Corrosion* 1965, *21*, 341.

COMPONENTS: (1) Nickel; Ni; [7440-02-0] (2) Lithium; Li; [7439-93-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1989

CRITICAL EVALUATION:

Experimental data of the solubility of Ni in liquid Li show considerable agreement. The results scatter less than one order of magnitude at any selected temperature. Such behaviour was not frequently observed in solubility measurements of other transition metals in the liquid alkali metals.

In the first attempt Wilkinson and Yaggee (1) found that Li dissolved $1.1\cdot 10^{-2}$ mol % Ni at 573 K; a slightly higher value ($1.8\ 10^{-2}$ mol % Ni) was detected with Inconel alloy. Both values are surely one order of magnitude too high and may be rejected. At the same time Jesseman et al. (2) determined the saturating concentration of Ni at 536 to 1267 K and found an increase of the solubility with temperature, if the equilibration time was 24 or 100 hours. Equilibration was obviously not reached if the tests were not extended to more than 4 hours. Bagley and Montgomery (8) used a similar procedure to equilibrate Ni with Li in a stainless steel crucible. A correction of their results was made for the transfer of stainless steel particles into Li phase. The temperature dependence of the data of (8) between 473 and 923 K was steeper than that of (2).

The first determination using a test crucible of pure Ni were performed by Bychkov et al. (5,6,7) at 973 to 1223 K. An effect of O and N on the solubility was also investigated (7). The dissolution of Ni from a steel (8 mass % Ni) was found to be few orders of magnitude lower than the solubility of pure Ni in Li (7). Leavenworth et al. (9,10), contrary to (7), found one order of magnitude increase of saturating Ni concentration in Li for 50 % increase of Ni concentration in Li. Such an effect was not confirmed by other investigators and was probably caused by impurities in the system. The individual result reported by Weeks (3) - $7.8\ 10^{-3}$ mol % Ni at 873 K - most probably originated from an extrapolation of the data of (9) to this temperature but not from his own measurement since no experimental aspects of the estimation were described in (3). Minushkin (4) investigated the dissolution kinetics of Ni in liquid Li at 813 to 1098 K and determined the saturation concentration; these results were also reported in (17). Slightly faster dissolution kinetics were observed by (31) who used metals of higher purity.

Takeushi et al. (13) determined the solubility of Ni in liquid Li as well as a solid solubility of Li in Ni at 873 to 1473 K; the results allowed a sketch of the Ni-Li phase diagram characterized by a liquid miscibility gap. Cheburkov (14) carried out several solubility determinations in the temperature range 773 to 1390 K. His results were reported in graphical form in (15), but experimental details are not known to the evaluators. The Ni solubility in distilled Li increased from $4\ 10^{-2}$ to $4\ 10^{-1}$ mol %. It was impossible to read separate points from the figure, therefore the paper is not compiled. Katsuka and Furukawa (12) reported that Li saturated with Ni contained $2.4\cdot 10^{-2}$ mol % Ni at 873 K. Li contained 0.05 mol % N but no more details were described and the work is not compiled. Plekhanov et al. (20) again investigated the effects of O, N and C on the solubility of Ni in Li at temperatures 773 to 1078 K. An increase of the solubility was observed for every contaminant, nevertheless, it was not much higher for O and C than the experimental scatter of the individual results; the effect of N was the strongest and higher than the scatter.

Beskorovainyi et al. (15,16,19,21-27) performed precise measurements of the solubility of pure Ni and 41 types of alloys (mainly steels). The results gained with metallic Ni showed very good agreement with the theoretical predictions (21,27). The effect of H, C, O and N in Li on the Ni solubility was also determined (23,31) and was found to be weak for C, H, and O, and moderate for N. This effect was not supported by a thermodynamic theory of interactions in the Ni-Li-N system. According to this model no extra dissolution of Ni in Li due to an increase of the N content should be observed (28). Two types of logarithm of solubility vs. reciprocal temperature dependencies were observed (16,19,22,24,25,26,31): (i) linear and (ii) with inflexion between two linear segments. The inflexions reflect changes of Ni activity due to phase transformations in the solids. The most recent results of Awasthi et al. [30] in the temperature range 503 - 873 K fit very well to the data of (15,23).

Several solubility equations have been proposed:

Gill	(18)	$\log(soly/\text{mol \% Ni}) = -1.539 - 1719\ (T/\text{K})^{-1}$	from data of (5,8,10)	Eq.(1)
Kelly	(11)	$\log(soly/\text{mol \% Ni}) = 1.577 - 3230\ (T/\text{K})^{-1}$	from data of (9,10)	Eq.(2)
Beskorovainyi	(27)	$\log(soly/\text{mol \% Ni}) = 2.044 - 3177\ (T/\text{K})^{-1}$	from data of (15,23)	Eq.(3)

Eq.(3) is recommended because it is based on the preferred results. Moreover, it is most similar to the theoretical equation (21,27):

$$\log\ (soly/\text{mol \% Ni}) = 2.239 - 3613\ (T/\text{K})^{-1} \qquad \text{Eq.(4)}$$

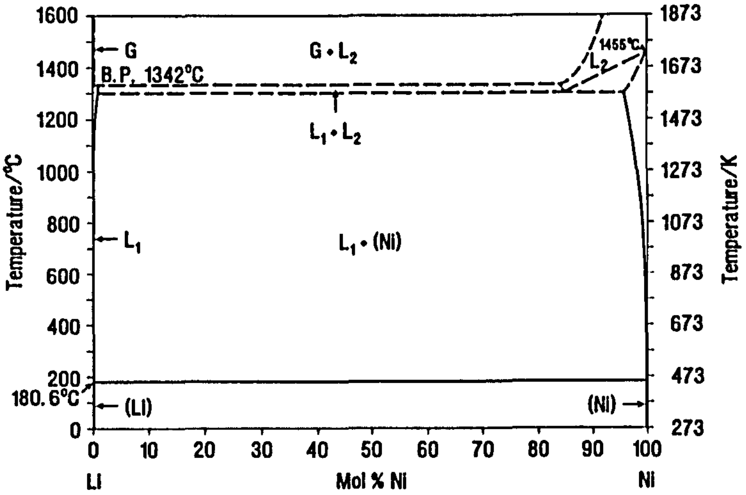
The selection of our recommended results is based on the results of (4,12,13,15,23,30) which agree typically within ± 15 %. The partial Ni-Li phase diagram was reported in (29). In the temperature range 453-1573 K, liquid Li-saturated solution is in equilibrium with solid Ni saturated with 2-4 mol % Li; see the figure.

Recommended and tentative (t) values of the solubility of Ni in liquid Li:

T/K	$soly$/mol % Ni	source	T/K	$soly$/mol % Ni	source
523	$1\ 10^{-4}$	(15),(30) interpolation	973	$6\ 10^{-2}$	(15),(23)
573	$3\cdot 10^{-4}$	(15),(30) interpolation	1073	$1.2\cdot 10^{-1}$	(13),(15) mean value
673	$2\cdot 10^{-3}$	(15),(23)	1173	$2\cdot 10^{-1}$ (t)	(13)
773	$8\cdot 10^{-3}$	(15),(30)	1273	$3\cdot 10^{-1}$	(13),(14) mean value
873	$2.5\cdot 10^{-2}$ (t)	(12),(15)	1473	$4\cdot 10^{-1}$ (t)	(13)

COMPONENTS:	EVALUATOR:
(1) Nickel; Ni; [7440-02-0] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1989

CRITICAL EVALUATION:



References:

1. Wilkinson, W.D.; Yaggee, F.L. *US Atom.Ener.Comm. Rep. ANL-4990*, 1950.
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. Weeks, J.R. *US Atom.Ener.Comm. Rep. BNL-7553*, 1963; *NASA Rep. SP-41*, 1963, p.21. .
4. Minushkin, B. *U.S. Naval Res. Rep. NDA-2141-1*, 1961; *US Atom.Ener.Comm. Rep. AD-259703*, 1961.
5. Bychkov, Yu.F.; Rozanov, A.N.; Yakovleva, V.B. *Atom.Energiya* 1959, *7*, 531; *Kernenergie* 1960, *3*, 763.
6. Bychkov, Yu.F.; Rozanov, A.N.; Rozanova, V.B. *Metall. Metalloved. Chist. Met.* 1960, *2*, 178; *Metallurgy and Metallography of Pure Metals*, Gordon & Breach, N.Y., 1962, p. 178.
7. Bychkov, Yu.F.; Rozanov, A.N.; Skorov, D.M.; Cheburkov, V.I. *Metall. Metalloved. Chist.Met.*, 1960, *2*, 78; *Metallurgy and Metallography of Pure Metals*, Gordon & Breach, N.Y., 1962, p. 78.
8. Bagley, K.Q.; Montgomery, K.R. *UK Atom.Ener.Auth. Rep. IGR-TN/C-250*, 1955.
9. Leavenworth, H.W.; Cleary, R.E.; Bratton, W.D. *US Atom.Ener.Comm.Rep. PWAC-356*, 1961.
10. Leavenworth, H.W.; Cleary, R.E. *Acta Metall.* 1961, *9*, 519.
11. Kelly, K.J. *NASA Rep. TN-D-769*, 1961, p.27.
12. Katsuta, H.; Furukawa, K. *J. Nucl.Mater.* 1977, *71*, 95.
13. Takeushi, Y.; Mochisuki, K.; Watanabe, M.; Obinata, I. *Metall* 1966, *20*, 2.
14. Cheburkov, V.I. *Ph.D.thesis*, Moskva, USSR, 1967; as cited in (15).
15. Beskorovainyi, N.M.; Vasilev, V.K.; Lyublinskii, I.E. *Metall.Metalloved. Chist. Met.* 1980, *14*, 135.
16. Beskorovainyi, N.M.; Lyublinskii, I.E. *Zashch. Met.* 1985, *21*, 609.
17. McKee, J.M.; Steinmetz, H. *US Atom.Ener.Comm. Rep. TID-7626*, Pt.I, 1961, p.143.
18. Gill, W.N.; Vanek, R.P.; Grove, C.S. *AIChE J.* 1961, *7*, 216.
19. Beskorovainyi, N.M.; Ioltukhovskii, A.G.; Lyublinskii, I.E.; Vasilev, V.K. *Fiz.-Khim.Mekh.Mater.* 1980, *16*, no 3, 59.
20. Plekhanov, G.A.; Fedortsov-Lutikov, G.P.; Glushko, Yu.V. *Atom.Energiya* 1978, *45*, 143; *Sov. Atom. Ener.*, 1979, *45*, 818.
21. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagramm Sostoyaniya*, Nauka, Moskva, 1985, p.113.
22. Beskorovainyi, N.M.; Yoltukhovskii, A.G.; Kirillov, V.B.; Lyublinskii, I.E.; Filipovich, E.I. *Fiz.-Khim. Mekh. Mater.* 1984, *20*, no 6, 9.
23. Beskorovainyi, N.M.; Ioltukhovskii, A.G. *Konstruktsionnye Materialy i Zhidkometallicheskie Teplonositeli*, Energoatomizdat, Moskva, 1983, p.71.
24. Beskorovainyi, N.M.; Ioltukhovskii, A.G.; Filipkina, E.I.; Krasin, V.P.; Radin, I.V. *Materialy dla Atomnoi Tekhniki*, Energoatomizdat, Moskva, 1983, p.23.
25. Lyublinskii, I.E.; Chernov, I.I.; Kalin, B.A.; Krasin, V.P. *Fiz. Khim.Obrabot.Mater.* 1987, no 1, 45.
26. Lyublinskii, I.E.; Kuzin, A.N.; Beskorovainyi, N.M. *Materialy dla Atomnoi Tekhniki*, Energoatomizdat, Moskva, 1983, p.41.
27 Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Materialy dla Atomnoi Tekhniki*, Energoatomizdat, Moskva, 1983, p.33.
28. Krasin, V.P.; Beskorovainyi, N.M.; Lyublinskii, I.E. *Fiz. Khim. Obrabot. Mater.* 1987, no 1, 107.
29. *Binary Alloy Phase Diagramms*, T.B. Massalski, Ed.,ASM, Metals Park, 1986, p.1494.
30. Awasthi, S.P.; Borgstedt, H.U.; Frees, G. *Liq. Met. Engin. Technol.*, BNES, London, 1984, *1*, 265.
31. Gryaznov, G.M.; Evtikhin, V.A.; Zavyalskii, L.P.; Kosukhin, A.Ya.; Lyublinskii, I.E. *Materialovedenie Zhidkometallicheskikh Sistem Termoyadernykh Reaktorov*, Energoatomizdat, Moskva, 1989, p.49, 67, 71, 76, 81, 82, 105.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

(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: 536-1267 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Li at various temperatures and equilibration times was determined.

t/°F	*T*/K [a]	*time*/h	*soly*/mass % Ni	*soly* /mol % Ni [a]
505	536	4	0.020	$2.4\cdot10^{-3}$
605	591		0.028, 0.064	$3.3\cdot10^{-3}$, $7.5\cdot10^{-3}$
745	669		0.262	$3.1\cdot10^{-2}$
920	766		0.308, 0.310	$3.6\cdot10^{-2}$, $3.7\cdot10^{-2}$
1025	825		0.25	$3.0\cdot10^{-2}$
1330	993		0.20, 0.295	$2.4\cdot10^{-2}$, $3.5\cdot10^{-2}$
1780	1243		0.70, 1.80	$8.3\cdot10^{-2}$, 0.22
1820	1267		1.67	0.20
505	536	24	0.125	0.015
605	591		0.10, 0.13	$1.2\cdot10^{-2}$, $1.5\cdot10^{-2}$
745	669		0.29	$3.4\cdot10^{-2}$
920	766		0.234, 0.22	$2.8\cdot10^{-2}$, $2.6\cdot10^{-2}$
1025	825		0.92	0.11
1330	993		1.37, 1.62	0.16, 0.195
1780	1243		1.40	0.17
605	591	100	0.090, 0.050	0.0106, 0.0059
745	669		0.40	0.047
920	766		0.41, 0.50	0.048, 0.059
1025	825		1.10	0.13
1330	993		1.40, 1.71	0.16, 0.20

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Fe /Armco/ capsule was loaded with a Ni tubing and Li in an Ar atmosphere, then degassed and Li was melted by a pot furnace. After welding, the capsule was heated in a vacuum furnace with stainless steel plates, for which average temperatures were assigned. The selected temperature was kept stable for period given and then the furnace core was air cooled. The capsule was weighed and opened. The solidified sample was leached out of the capsule with H_2O and Ni 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 analyzed spectrographically for Ni content.

SOURCE AND PURITY OF MATERIALS:

Ni: nothing specified
Li: 99.76 % pure and contained 0.24 % O, <0.02 % N and $5\cdot10^{-3}$ % Na.
H_2O: distilled.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: precision better than ± 30%.
Temperature: stability ± 20 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Nickel; Ni; [7440-02-0] (2) Lithium; Li; [7439-93-2]	Wilkinson, W.D.; Yaggee, F.L. *US Atom.Ener.Comm. Rep. ANL-4990*, 1950.
VARIABLES:	**PREPARED BY:**
Temperature: 573 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The apparent solubility of Ni from various solid metals in liquid Li at 300 °C was determined.

source	*soly*/mass % Ni	*soly*/mol % Ni [a]
Ni	0.09	0.011
Inconel	0.15	0.018

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Specimens of the tested materials were polished using grinding paper. The specimens were immediately covered with mineral oil, then washed with petroleum ether and dried. A testing autoclave which was made of steel was evacuated, and molten Li was filtered into it. The specimens and U turnings which served as getter material were introduced into the apparatus, Ar served as cover gas of slightly positive pressure. The whole apparatus was heated for 6.5 days. The autoclave was cooled to 473 K and opened, mineral oil was poured on the samples. The specimens were re-weighed. The Li was poured into pans of oil. The method to determine Ni in Li was not described.

SOURCE AND PURITY OF MATERIALS:

Ni: nothing specified.
Inconel: 80 % Ni, 15 % Cr, 5 % Fe.
Li: from Maywood Chem. Corp., with contents of 0.1 % Ca, 0.01 % Na, Fe, and 0.1 % Si; it was further purified by filtering and gettering with U turnings.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

Bagley, K.Q.; Montgomery, K.R.

UK Atom.Ener.Auth. Rep. IGR-TN/C-250, 1955.

VARIABLES:

Temperature: 473-923 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Li at various temperatures was determined.

t/°C	*Fe content*/mass %	*Ni content*/mass %	*soly*/mass % Ni [a]	*soly*/mol % Ni [b]
200-250	0.048	0.012	0.0067	0.00079
300	0.16	0.046	0.0284	0.0033
355	0.123	0.0249	0.0108	0.0013
370	0.0851	0.0267	0.0163	0.0019
398	0.153	0.0409	0.0239	0.0028
462	0.114	0.033	0.020	0.0024
487	0.77	0.0245	0.0159	0.0019
487	0.24	0.043	0.0163	0.0019
595	1.56	0.263	0.09	0.0106
650	0.77	0.24	0.158	0.019

[a] corrected by the authors [b] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus was made of stainless steel (18/8/1). A Ni gauze, degreased, cleaned and deoxidized in H_2 was placed on top of a metal sinter. The apparatus was degassed and filled with Ar. A Li ingot was introduced into the apparatus. The temperature was raised to the desired value. Homogenization was assisted by stirring for 3 hours before sampling. The removal of Ar from below the sinter caused the filtration of Li into a Ta sampling cup. The filtered sample was dissolved after cooling in H_2O and traces of Ni adhering to the cup were removed by a dilute acid. The solution was analyzed for its Li, Ni, Fe and Cr contents. Since particles of the steel were carried over into the Li filtrate, a correction for the Ni content in Li was made for the primary solubility results. It was assumed that one ninth of the Fe content is the amount of Ni which was originated from the stainless steel particles in Li.

SOURCE AND PURITY OF MATERIALS:

Ni: unspecified
Li: double distilled with final contents of $<6 \cdot 10^{-3}$ % Fe, $<4 \cdot 10^{-3}$ % Cr and $<1 \cdot 10^{-3}$ % Ni.
Steel: 73 % Fe, 18 % Cr, 8 % Ni.

ESTIMATED ERROR:

Solubility: "high" accuracy, yet numerically unspecified.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

(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: 973-1223 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Li was determined.

t/°C	*soly*/mass % Ni	*soly*/mol % Ni [a]
700	0.15	0.018
750	0.5	0.059
850	1.36	0.16
950	3.2	0.39

[a] as calculated by the compilers

The results were also reported in (1).

It was found that the equilibrium concentration at 750°C had been reached after 50 hours of conditioning.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ni test crucible was grinded, electropolished and pickled. It was gradually filled with freshly distilled Li which dropped from a stainless steel container. After the distillation was stopped, the apparatus was filled with Ar. The crucible with a cover was placed in a stainless steel container which was inlaid with a Mo liner. The container was placed in an arc furnace and conditioned for 100 hours at the desired temperature. The Li solution was cooled to solidification in about 50 s. The Ni content in the sample was determined by means of a colorimetric method of analysis.

SOURCE AND PURITY OF MATERIALS:

Ni: virtually nothing specified ("extruded from a band").
Li: distilled, with final contents of (2-6)$\cdot 10^{-2}$ % Na, 1.5$\cdot 10^{-2}$ % K, (1-4) 10^{-4} % Fe, $\leq 2\cdot 10^{-3}$ % Mg; Si, Ni, and Cr not detected.
Ar: unspecified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

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

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

(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: 1073-1473 K
concentration of N: 0.49-0.89 mol %
concentration of O: 0.44-0.85 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Influence of the contents of N and O on the solubility of Ni in liquid Li at 800°C was investigated.

addition/mass %	*addition*/mol % [a]	*soly*/mass % Ni	*soly*/mol % Ni [a]
0.985	0.49 /N/	0.60	0.071
1.77	0.89 /N/	0.60	0.071
1.0	0.44 /O/	0.43	0.051
1.93	0.85 /O/	1.25	0.149
-	-	0.43	0.051

The dissolution of Ni from Ya1-T steel in liquid Li was investigated:

t/°C	*addition*/mass %	*addition*/mol %	*time*/h	*soly*/mass % Ni	*soly*/mol % Ni [a]
900	-	-	220	< 10-4 [b]	< 10-5 [b]
975	-	-	110	< 10-4	< 10-5
975	-	-	240	0.029	3.4·10-3
975	0.99	0.43 /O/	110	0.031	3.7·10-3
975	1.12	0.49 /O/	240	0.109	1.28·10-2
975	1.0	0.50 /N/	110	0.020	2.4·10-3
1200	-	-	5	< 10-4 [b]	< 10-5 [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 Ni or Ya1-T steel, were electrolytically polished. Additions of gazeous O or N into the Li were carried out in a special glass apparatus. The crucible and the specimen was loaded with Li in a dry box filled with Ar. The crucible was closed by welding and placed in a stainless steel container. The container was transferred into an arc furnace and heated to the desired temperature for the time given. After the equilibration the container was quenched in H_2O. The opened crucible was covered with paraffin. The Ni content in Li was analyzed by a colorimetric method in the Institute of General and Inorganic Chemistry of Academy of Science of USSR.

SOURCE AND PURITY OF MATERIALS:

Ya1-T steel: 8 % Ni, 17.3 % Cr, 0.48 % Si, 0.4 % Ti, 0.1 % C, 0.07 % Mn, 0.015 % P, 0.001 % S and Fe to balance.
Ni: electrolytic and vacuum remelted in a magnetite crucible.
Li: distilled and with contents of $(2\text{-}6)\cdot10^{-2}$ % Na, $1\cdot10^{-2}$ % K, $4\cdot10^{-4}$ % Fe, Ni and Cr not detected.
O: from decomposition of $KMnO_4$.
N: chemically pure.
Ar: nothing specified.

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. Meetalloved. Chist. Met.* 1960, *2*, 178-188; *Metallurgy & Metallography of Pure Metals*, Gordon and Breach, N.Y., 1960, p. 178-188.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

(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: 925-1170 K
Concentration of N in Li: 0.0073-0.011 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Li at various temperatures and N contents in Li was reported in the figure; they were read out and calculated to mol % Ni by the compilers.

T/K	*N concn*/mass %	*N concn*/mol %	*soly*/mol % Ni
925	$1.46\cdot10^{-2}$	$7.3\ 10^{-3}$	0.0149
950	$1.46\cdot10^{-2}$	$7.3\cdot10^{-3}$	0.0162
982	$1.46\cdot10^{-2}$	$7.3\cdot10^{-3}$	0.0172
1040	$1.46\cdot10^{-2}$	$7.3\cdot10^{-3}$	0.028
1075	$1.46\cdot10^{-2}$	$7.3\cdot10^{-3}$	0.039
1098	$1.46\cdot10^{-2}$	$7.3\cdot10^{-3}$	0.037
1138	$1.46\cdot10^{-2}$	$7.3\cdot10^{-3}$	0.067
1148	$1.46\cdot10^{-2}$	$7.3\cdot10^{-3}$	0.055
1170	$1.46\ 10^{-2}$	$7.3\cdot10^{-3}$	0.089
935	$2.20\ 10^{-2}$	$1.10\cdot10^{-2}$	0.158
937	$2.20\cdot10^{-2}$	$1.10\ 10^{-2}$	0.126
993	$2.20\cdot10^{-2}$	$1.10\ 10^{-2}$	0.20
993	$2.20\cdot10^{-2}$	$1.10\cdot10^{-2}$	0.22
1045	$2.20\cdot10^{-2}$	$1.10\cdot10^{-2}$	0.31
1114	$2.20\cdot10^{-2}$	$1.10\cdot10^{-2}$	0.70
1160	$2.20\cdot10^{-2}$	$1.10\cdot10^{-2}$	0.39

The results at $7.3\cdot10^{-3}$ mol % N in Li were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ni cup was heated at 811 K in an Ar atmosphere. The cup was loaded with Li and heated to the desired temperature for 24 hours. A Li sample was taken by dipping a Mo bucket into liquid Li. The sample was allowed to cool and then dissolved in 10 % HCl in a propylene beaker at dry ice temperature. This way Ni traces were dissolved in HCl from the bucket walls. An unspecified colorimetric procedure was used to determine the Ni content and flame photometry to obtain the weight of the Li sample. An agitation during the equilibration period did not show an effect on the measured solubility. The N level in Li was established by an addition of proper amounts of Li_3N.

SOURCE AND PURITY OF MATERIALS:

Ni: 99.8 % pure.
Li: 99.8 % pure; further purified by contacting Li with Ti sponge at 1144 K for 2 hours and cooled to 260-315 K, N content < $5\cdot10^{-4}$ mol %.
Ar: purified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Leavenworth, H.W.; Cleary, R.E. *Acta Metall.* 1961, *9*, 519-520.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

(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: 813-1098 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The dissolution kinetics of Ni in liquid Li were investigated and the corresponding equilibrium concentrations were estimated:

t/°C	*time*/h	*soly*/mass % Ni	*soly* mol % Ni [a]
540	2.5	$(6.0 \pm 0.75)\cdot10^{-2}$	$(7.1 \pm 0.9)\cdot10^{-3}$
778 [b]	0.8	1.44 ± 0.12	0.172 ± 0.015
778 [c]	0.5	1.21 ± 0.073	0.145 ± 0.009
825	1.0	1.63 ± 0.05	0.196 ± 0.006

[a] as calculated by the compilers;
[b] stirred at 54 r.p.m.;
[c] stirred at 485 r.p.m.

The mean results were reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A stainless steel vessel with a Ni liner and a cylindrical Ni stirrer was filled with liquid Li. During the course of Ni dissolution, samples of Li were periodically removed by means of a Ta sampling bucket which passed through Wilson seals on gas locks. The bottom section of the apparatus was heated by an electric furnace regulated by a thermocouple. He was used as the cover gas and the apparatus was vacuum outgassed prior to each test. Li samples were dissolved in H_2O and the resulting solutions were titrated with HCl to determine the Li amount An excess of HCl was added, the solution boiled and filtered. The Ni determination was performed by means of the classical dimethylglyoxime method by weight and by the colorimetric procedure. Both results were in excellent agreement.

SOURCE AND PURITY OF MATERIALS:

Ni: 99.70 % pure, with contents of 0.04 % C; 0.07 % Mn; 0.10 % Fe; 0.008 % S; 0.03 % Si,Cu.
Li: 99.80 % pure from Maywood Chem. Works, further vacuum distilled, with contents of $5\cdot10^{-4}$ % Fe, $3\ 10^{-3}$ % Ni and $1\cdot10^{-2}$ % N.
He: nothing specified.

ESTIMATED ERROR:

Solubility: precision of analyses ± 10 %; standard deviation of the results better than ± 8 %.
Temperature: stability ± 3 K.

REFERENCES:

1. McKee, J.M.; Steinmetz, B. *US Atom.Ener.Comm. Rep. TID-7626*, Pt.1, 1962, p. 143-145.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

Takeuchi, Y.; Mochisuki, K.; Watanabe, M.; Obinata, I.

Metall 1966, *20*, 2-8.

VARIABLES:

Temperature: 873-1473 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Li as well as solid solubility of Li in Ni was determined.

t/°C	*time*/h	*soly*/mass % Ni	*soly*/mol % Ni [a]	*soly*/mass % Li	*soly*/mol % Li [a]
1200	3	3.49	0.426	0.40	3.28
1100	6	2.41	0.292	0.33	2.72
1000	10	2.38	0.287	0.32	2.64
900	24	1.95	0.235	0.28	2.32
800	48	1.28	0.153	0.26	2.16
700	72	0.73	0.087	0.25	2.07
600	100	0.39	0.046	-	-

[a] as calculated by the compilers

The thermal analysis of Ni-Li(50 mass %) alloy showed an arrest at 155 ± 5°C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Ni-Li alloys were prepared by melting Ni powder with Li in a Ni closed container under an Ar atmosphere. The melt was equilibrated for the specified time at a selected temperature. Then the container was effectively cooled down with H_2O. The Li-rich and Ni-rich phases were mechanically separated. The Ni content in Li was determined by a chemical analysis after dissolution of a sample in H_2O. Also the Ni phase was analyzed for its Li content after a sample of the alloy was quenched in a Cu crucible. The thermal analysis was performed in a W crucible, placed in a stainless steel closed container.

SOURCE AND PURITY OF MATERIALS:

Ni: 99.9 % pure with a content of 0.078 % Co, 0.008 % Cu, 0.012 % Fe, 0.023 % S and 0.003 % C.
Li: 99.9 % pure, as in (1) (see the Mn-Li system).
Ar: nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified, probably ±10 % as in (1)
Temperature: stability ± 5 K, as in (1)

REFERENCES:

1. Obinata, I.; Takeuchi, Y.; Kurihara, K.; Watanabe, M. *Metall* 1965, *19*, 21-35.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Nickel; Ni; [7440-02-0] (2) Lithium; Li; [7439-93-2]	Plekhanov, G.A; Fedortsov-Lutikov, G.P.; Glushko, Yu.V. *Atom. Energiya* 1978, *45*, 143-145.
VARIABLES: Temperature: 773-1078 K Concentration of O, N and C in liquid Li: 0.005-0.22, 0.004-0.50 and 0.001-0.12 mol %, respectively	PREPARED BY: H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The influence of O, N and C on the solubility of Ni in liquid Li at various temperatures was determined .

t/°C	*addition in Li*/mol %	*soly*/mol % Ni	*mean value*/mol % Ni [a]
800-805	-	0.136, 0.151, 0.151, 0.152, 0.170	0.152
	0.215-0.220 (O)	0.150, 0.172, 0.147	0.156
	0.12 (C)	0.175, 0.157, 0.157	0.163
	0.25-0.27 (N)	0.166, 0.178, 0.197	0.180
	0.50 (N)	0.203, 0.189	0.196
650-660	-	0.060, 0.051, 0.053	0.055
	0.26 (N)	0.075, 0.090, 0.070	0.078
500-505	-	0.0153, 0.0153, 0.0153	0.0153
	0.25-0.26 (N)	0.0214, 0.0214, 0.0262	0.0230
	0.50 (N)	0.0202, 0.0226, 0.0225	0.0218

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Solid Li with additions of Li_2O, Li_3N or graphite was placed in a Ni vessel and the vessel was closed in a dry box filled with Ar. The vessel was mounted in a Mo container. The system was equilibrated for 24 hours at the desired temperature. Then the capsule was inverted causing flow of liquid Li sample into the container. After cooling the Li sample was spectrally analyzed.

SOURCE AND PURITY OF MATERIALS:

Ni: electrolytic.
Li: filtrated with contents of $5 \cdot 10^{-3}$ 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.
C: spectrally pure.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: precision up to ± 13 %, typically ± 2 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Nickel; Ni; [7440-62-0] (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: 623-1073 K Concentration of N: $\leq 10^{-3}$ - 0.275 mol % Concentrations of O,H,C:0.087,0.35,0.07 mol %, respectively	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Li from preliminary experiments; mol % calculated by the compilers.

t/°C	*soly*/mass % Ni	*soly*/mol % Ni
550	0.174 ± 0.004	0.0205 ± 0.0005
650	0.366 ± 0.006	0.0431 ± 0.0007
800	0.960 ± 0.020	0.114 ± 0.024

The solubility of Ni in liquid Li of different purity; mol % calculated by the compilers.

	" pure LE-1" Li		purified Li	
t/°C	*soly*/mass % Ni	*soly*/ mol % Ni	*soly*/mass % Ni	*soly*/mol % Ni
350	0.0166±0.0005	$(1.96\pm0.06)\cdot10^{-3}$	0.0058±0.0003	$(6.84\pm0.36)\cdot10^{-4}$
375	-	-	0.0114±0.0003	$(1.34\pm0.04)\cdot10^{-3}$
400	0.0366±0.0007	$(4.31\pm0.08)\cdot10^{-3}$	0.0182±0.0003	$(2.15\pm0.04)\cdot10^{-3}$
425	0.0485±0.0006	$(5.72\pm0.07)\ 10^{-3}$	0.0268±0.0002	$(3.16\pm0.02)\ 10^{-3}$
450	0.0674±0.0005	$(7.95\pm0.06)\cdot10^{-3}$	0.0407±0.0007	$(4.80\pm0.08)\cdot10^{-3}$
475	0.0835±0.0003	$(9.85\pm0.04)\cdot10^{-3}$	-	-
500	0.1055±0.0003	$(1.24\pm0.004)\ 10^{-2}$	0.0721±0.0002	$(8.50\pm0.02)\cdot10^{-3}$
550	0.1635±0.0005	$(1.93\pm0.006)\cdot10^{-2}$	0.1297±0.0005	$(1.53\pm0.006)\ 10^{-2}$
600	0.2600±0.0011	$(3.07\pm0.013)\cdot10^{-2}$	0.2144±0.0003	$(2.53\pm0.004)\cdot10^{-2}$
650	0.3994±0.0003	$(4.71\pm0.004)\cdot10^{-2}$	0.3333±0.0014	$(3.93\pm0.02)\cdot10^{-2}$
700	0.5794±0.0008	$(6.83\pm0.01)\ 10^{-2}$	0.4942±0.0024	$(5.84\pm0.03)\ 10^{-2}$
750	-	-	0.7034±0.0030	$(8.31\pm0.03)\cdot10^{-2}$

A solubility equation for purified Li was fitted to the data and tested by the compilers.

$$\log(soly/\text{mol \% Ni}) = (2.101 \pm 0.063) - (3240 \pm 56)(T/\text{K})^{-1}$$

The solubility of Ni in liquid Li containing various additives was reported in (1) and (8); the data were read out and recalculated to mol % Ni by the compilers.

	soly/mol % Ni with different additives				
t/°C	0.055 mol % N	0.275 mol % N	0.087 mol % O	0.35 mol % H	0.07 mol % C
300	-	-	-	$7.0\cdot10^{-4}$	-
350	$1.8\cdot10^{-3}$	$1.9\cdot10^{-3}$	$1.0\cdot10^{-3}$	$1.5\cdot10^{-3}$	$2.2\cdot10^{-3}$
400	$4.1\cdot10^{-3}$	-	$2.5\cdot10^{-3}$	$4.1\cdot10^{-3}$	$5.3\cdot10^{-3}$
450	$7.3\cdot10^{-3}$	$1.1\cdot10^{-2}$	$4.9\ 10^{-3}$	$7.5\cdot10^{-3}$	$8.2\cdot10^{-3}$
475	$1.1\cdot10^{-2}$	$1.3\cdot10^{-2}$	$6.1\cdot10^{-3}$	-	-
500	$1.3\ 10^{-2}$	$1.7\cdot10^{-2}$	$8.6\cdot10^{-3}$	$1.3\cdot10^{-2}$	$1.2\cdot10^{-2}$
525	$1.5\cdot10^{-2}$	$2.1\cdot10^{-2}$	$1.2\cdot10^{-2}$	-	-
550	$1.8\cdot10^{-2}$	$2.7\ 10^{-2}$	$1.5\ 10^{-2}$	$1.8\cdot10^{-2}$	$1.6\cdot10^{-2}$
575	$2.4\ 10^{-2}$	$3.4\cdot10^{-2}$	-	-	-
600	$2.9\cdot10^{-2}$	$4.1\ 10^{-2}$	$2.5\ 10^{-2}$	$2.8\cdot10^{-2}$	$2.9\cdot10^{-2}$
625	$3.5\ 10^{-2}$	-	-	-	-
650	$4.7\cdot10^{-2}$	$5.7\cdot10^{-2}$	-	$4.1\ 10^{-2}$	$4.7\cdot10^{-2}$
675	$5.3\ 10^{-2}$	-	-	-	-
700	$7.0\cdot10^{-2}$	-	$5.9\cdot10^{-2}$	$6.1\cdot10^{-2}$	-

An apparent solubility of Ni from various Ni-alloys in liquid Li was also investigated; the values were read out and recalculated to mol % Ni by the compilers.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Nickel; Ni; [7440-62-0] (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: 623-1073 K Concentration of N: $\leq 10^{-3}$ - 0.275 mol % Concentrations of O,H,C:0.087,0.35,0.07 mol %, respectively	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES: (continued)

t/°C range	alloy type	Ni content mass %	*soly range*/mol % Ni	log (*soly*) vs. T^{-1} plot	reference
550- 900	Kh30N30	29.5	$1.8\ 10^{-3}$ - $3.2\cdot10^{-2}$	linear	(8)
600- 950	09Kh16N15M3B	15.2	$2.4\cdot10^{-4}$ - $1.8\cdot10^{-2}$	linear	(2,3,6)
550- 950	1Kh21N5T	5.6	$3.6\cdot10^{-4}$ - $7.7\cdot10^{-3}$	inflexional	(2)
550- 900	Kh20N30V5M3	27.6	$4.7\cdot10^{-4}$ - $2.6\cdot10^{-2}$	linear	(2)
550- 950	1Kh18N10T	10.5	$5.0\cdot10^{-4}$ - $1.4\cdot10^{-2}$	inflexional	(2,6)
550- 950	08Kh20N14S2	13.7	$5.3\cdot10^{-4}$ - $1.8\ 10^{-2}$	inflexional	(2,6)
650- 950	1Kh12NM3B	1.25	$3.6\cdot10^{-4}$ - $3.3\cdot10^{-3}$	linear	(2,6)
650- 950	1Kh12AMVBFR [a]	0.65	$6.6\cdot10^{-4}$ - $1.7\cdot10^{-3}$	linear	(2)
650- 950	1Kh12MVBFR	0.67	$3.6\ 10^{-4}$ - $9.4\ 10^{-4}$	almost linear	(2,6)
550- 850	Kh7N80M12T	80	$8.3\cdot10^{-3}$ - $9.4\ 10^{-2}$	linear	(2)
550- 900	Kh15N30 [b]	30	$1.1\cdot10^{-3}$ - $1.8\ 10^{-2}$	linear	(2)
550- 950	Kh15N15	15.2	$7.7\cdot10^{-4}$ - $1.2\ 10^{-2}$	inflexional	(2)
600- 950	0Kh16N15M3BR	14.9	$7.1\ 10^{-4}$ - $9.4\cdot10^{-3}$	linear	(3)
550- 900	Kh8N8	7.7	$8.3\cdot10^{-4}$ - $5.9\ 10^{-3}$	inflexional	(3)
550- 900	Kh18N8	7.9	$9.4\cdot10^{-4}$ - $6.5\ 10^{-3}$	inflexional	(3)
550- 900	Kh8N18	17.0	$1.2\cdot10^{-3}$ - $1.2\cdot10^{-2}$	inflexional	(3)
550- 900	Kh18N18	18.4	$1.3\cdot10^{-3}$ - $1.1\ 10^{-2}$	inflexional	(3)
550- 900	Kh20MN30	29.5	$1.5\ 10^{-3}$ - $2.6\ 10^{-2}$	inflexional	(3)
900-1000	Kh37N63	63	$7.1\cdot10^{-2}$ - $8.3\cdot10^{-2}$	linear	(3)
800- 900	Kh46N54	54	$4.5\cdot10^{-2}$ - $7.1\cdot10^{-2}$	linear	(3)
600- 800	Kh60N40	40	$9.4\cdot10^{-3}$ - $4.1\ 10^{-2}$	linear	(3)
600- 900	12Kh18N10T	9.1	$7.1\cdot10^{-4}$ - $7.7\cdot10^{-3}$	inflexional	(4)
600- 850	09Kh16N15M3B	14.7	$2.1\cdot10^{-4}$ - $8.3\cdot10^{-3}$	inflexional	(4)
600- 900	Cr-Ni steel	16.2	$1.0\cdot10^{-4}$ - $6.5\cdot10^{-3}$	linear	(5)
550- 900	0Kh18N10T	9.0	$4.7\ 10^{-4}$ - $5.9\cdot10^{-3}$	inflexional	(6)
550- 900	KhN28VMAB	27.6	$6.5\cdot10^{-4}$ - $3.3\ 10^{-2}$	linear	(6)
650-950	16Kh12VMFBR	0.65	$3.2\cdot10^{-4}$ - $9.4\ 10^{-4}$	asymptotic	(8)
600-950	11Kh12N2M2B	1.25	$1.3\cdot10^{-4}$ - $4.5\ 10^{-3}$	almost linear	(8)
550-950	08Kh18N10T	10.5	$4.9\cdot10^{-4}$ - $1.4\ 10^{-2}$	inflexional	(8)

t/°C range	alloy type	Ni content mass %	*soly range*/mol % Ni	log (*soly*) vs. T^{-1} plot	reference
550- 850	AISI 316	12.4	$3.8\cdot10^{-4}$ - $6.1\cdot10^{-3}$	inflexional	(6)
400- 850	N20	20.0	$3.4\ 10^{-4}$ - $9.9\ 10^{-3}$	almost linear	(6)
450- 900	N10	10.0	$6.5\cdot10^{-4}$ - $7.1\cdot10^{-3}$	inflexional	(6)
500- 900	N5	5.0	$2.4\cdot10^{-4}$ - $3.9\cdot10^{-3}$	inflexional	(6)
550- 950	N3	3.0	$1.8\ 10^{-4}$ - $3.3\cdot10^{-3}$	inflexional	(6)
600- 950	N2	1.5	$1.4\cdot10^{-4}$ - $1.9\cdot10^{-3}$	inflexional	(6)
450- 800	Kh13N30	30	$4.1\cdot10^{-4}$ - $1.3\cdot10^{-2}$	linear	(7)
450- 700	Kh13N25	25	$2.7\ 10^{-4}$ - $4.7\cdot10^{-3}$	linear	(7)
450- 800	Kh13N20	20	$2.1\cdot10^{-4}$ - $7.1\cdot10^{-3}$	inflexional	(7)
450- 800	Kh13N15	15	$2.0\cdot10^{-4}$ - $5.0\cdot10^{-3}$	inflexional	(7)
450- 750	Kh13N10	10	$1.9\cdot10^{-4}$ - $2.4\cdot10^{-3}$	inflexional	(7)
450- 900	Kh13N5	5	$1.8\ 10^{-4}$ - $3.2\cdot10^{-3}$	inflexional	(7)

[a] with addition of Zr, slightly decreased solubility
[b] with addition of Nb, Ti, Al, Si or Mo, decreased solubility

COMPONENTS:

(1) Nickel; Ni; [7440-62-0]

(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: 623-1073 K
Concentration of N: $\leq 10^{-3}$ - 0.275 mol %
Concentration of O,H,C: 0.087, 0.35, 0.07 mol %, respectively.

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES: (continued)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A cylindrical crucible made of Ni or Ni alloy was conditioned for 1 hour at 1373 K and placed in a hermetic capsule. The crucible was loaded with Li which was kept inside by surface tension forces and additionally V gettering foil was placed inside. The capsule with the test tube inside 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 temperarure. A X-ray beam passed along the crucible axis. The beam emitted of the sample was analyzed in a Soller's spectrometer and its intensity was measured from both sides of the K-boundary absorbtion of Ni. The amount of Ni in Li was calculated using the corresponding formula.

The concentration of N or C in Li was regulated by addition of Li_3N or graphite, respectively. O was added to Li by means of equiðlibration with Nb containing O at 1273 K for 10 h. The source of H in Li was not specified.

SOURCE AND PURITY OF MATERIALS:

Ni: "NO" purity, 99.9 % as found by the compilers, remelted in an At atmosphere.
Li(LE-1): 99.6 % pure and contained 10^{-2} % O and $3 \cdot 10^{-2}$ % N.
Li(purified): gettered in Nb-Zr (5 %) capsule which contained Y plate and finally contained $\leq 10^{-3}$ % N and $< 5\ 10^{-3}$ % O.
Li_3N: chemically pure.
C (graphite): spectrally pure.
He: nothing spcified.

ESTIMATED ERROR:

Solubility: standard deviation better than ± 5 %.
Temperature: stability ± 0.1 K.

REFERENCES:

1. Beskorovainyi, N.M.; Yoltukhovskii, A.G. *Kostruktsionnye Materialy i Zhidkometallicheskie Teplonositeli*, Energoatomizdat, Moskva, 1983, p.71.
2. Lyublinskii, I.E.; Kuzin, A.N.; Beskorovainyi, N.M. *Materialy dla Atomnoi Tekhniki*, Energoatomizdat, Moskva, 1983, p.41-52.
3. Beskorovainyi, N.M.; Yoltukhovskii, A.G.; Lyublinskii, I.E.; Vasilev, V.K.; *Fiz.-Khim. Mekhan. Mater.* 1980, *16*, no 3, 59-64.
4. Beskorovainyi, N.M.; Yoltukhovskii, A.G.; Kirilov, V.B.; Lyublinskii, I.E.; Filipkina, E.I. *Fiz.-Khim. Mekh. Mater.* 1984, *20*, no 6, 9-12.
5. Beskorovainyi, N.M.; Yoltukhovskii, A.G.; Filipkina, E.I.; Krasin, V.P.; Radin, I.V. *Materialy dla Atomnoi Tekhniki*, Energoatomizat, Moskva, 1983, p. 23-32.
6. Lyublinskii, I.E.; Beskorovainyi, N.M.; *Zashchita Met.* 1985, *21*, 609-612.
7. Lyublinskii, I.E.; Chernov, I.I.; Kalin, B.A.; Krasin, V.P. *Fiz. Khim. Obrabot. Mater.* 1987, no 1, 45-48.
8. Gryaznov, G.M.; Evtikhin, V.A.; Zavyalskii, L.P.; Kosukhin, A.Ya.; Lyublinskii, I.E. *Materialovedenie Zhidkometallicheskikh Sistem Termoyadernykh Reaktorov*, Energoatomizdat, Moskva, 1989, p.67,81,105-108.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

Awasthi, S.P.; Borgstedt, H.U.; Frees, G.

Liq. Met. Engin. Technol., BNES, London, 1984, *1*, 265-269.

VARIABLES:

Temperature: 503-873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Li was determined.

t/°C	*time*/h	*soly*/mass % Ni	*soly*/mol % Ni [a]	*mean value*/mol % Ni [a]
230	15	$7.6 \cdot 10^{-4}$, $7.6 \cdot 10^{-4}$	$9.0 \cdot 10^{-5}$, $9.0 \cdot 10^{-5}$,	$9.0\ 10^{-5}$
500	15	$6.9 \cdot 10^{-2}$, $7.3 \cdot 10^{-2}$	$8.1 \cdot 10^{-3}$, $8.6 \cdot 10^{-3}$	$(8.4 \pm 0.2) \cdot 10^{-3}$
600	10	0.10, 0.28	$1.2 \cdot 10^{-2}$, $3.3 \cdot 10^{-2}$	$(2.2 \pm 0.1) \cdot 10^{-2}$

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus was calibrated in respect to temperature control and stability. A Ni crucible and a Ta sampling crucible was cleaned in a mixture of CH_3COOH, H_2SO_4 and HNO_3 (5:1:3) for 15 s at 363 K. The crucible was placed in an apparatus with sampling facility. The Ni crucible was filled with Li and equilibrated at the desired temperature for the desired period. After the equilibration Li was transferred to the sampling crucible, cooled and weighed. The sample was dissolved in H_2O and analyzed for Ni content by atomic absorption spectrometry. The experiments were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ni: 99.4 % pure.
Li: from Metallgesellschaft, purified by remelting in Ar atmosphere.
Ar: purified to get contents of $< 1 \cdot 10^{-4}$ % afO, and $< 1 \cdot 10^{-4}$ % H_2O.

ESTIMATED ERROR:

Solubility: precision better than ± 5 %.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Nickel; Ni; [7440-02-0] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland August 1995

CRITICAL EVALUATION:

The results of experiments to determine the solubility of Ni in liquid Na are spread over 3 orders of magnitude at a given temperature. Most of them are, however, grouped over half an order of magnitude.
The result of a solubility of $9 \cdot 10^{-4}$ mol % Ni at 923 K which was reported in (1) was not supported by experimental details, and is not compiled. The temperature dependences of solubilities of Ni in Na, which were separately established by (2), (3), (4), and (5), were characterized by a similar slope which significantly differed from the more steeper theoretical prediction which was presented in (6) in form of an equation:

$$\log(\mathit{soly}/\text{mol \% Ni}) = 3.394 - 5657\,(T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

A single result of Kovacina and Miller (7), $1.1 \cdot 10^{-5}$ mol % Ni at 673 K, is in very good agreement with this theory.
No influence of O on the apparent solubility of Ni in Na was reported by (8), while several later reports (3,7,9) showed that an increasing activity of O in Na caused an increase of the apparent solubility of Ni. These observations are in disagreement with some thermodynamic considerations (10,11,12) and corrosion tests (13). The conclusions were not supported by (14) who equilibrated Na with an O content of 0.29 mol % with stainless steel containing 10 mass % Ni. The content of Ni at 873 K was close to the solubility which was determined with pure Ni in pure Na. The other components of the steel may interfere with the O activity of the liquid phase. The determinations which were performed by (15) lead to similar results. The Ni concentration in Na which was equilibrated with 1Kh18N10T stainless steel at 1173 K was $3.9 \cdot 10^{-4}$ mol % Ni, while the equilibration with 99.8 % pure Ni resulted in a value of $7.8\ 10^{-4}$ mol % Ni at the same temperature. A data sheet of this publication was not prepared, since further details of the experimental procedure were not presented. The Ni concentrations in Na circulating in loops as measured in several laboratories were tabulated in (16). Ni was generally present in amounts comparable to its solubility reported in (3).
Alekseev et al. (17) examined the mass transfer of Ni in a non isothermal Na loop made of Kh18N10T steel. The Ni content of Na was determined by x-ray spectrometry. The experimental results were related to modelling calculations, and an equation for the solubility as function of temperature was developed:

$$\log(\mathit{soly}/\text{mol \% Ni}) = -3.325 - 3615\,(T/\text{K})^{-1} \qquad \text{Eq.(2)}$$

Though the slope in this equation seems to be reliable, the negative constant is not typical. Equation (2) leads to extremely low values of the solubility. The O concentration in Na and other essential parameters of the tests are not presented. Thus it is difficult to classify this equation.
The authors of (18) showed that the presence of 1 mass % Li in Na increases the solubility of Ni by more than one order of magnitude.
A solubility equation for the Ni-Na system was proposed by (19,20) on the basis of the reliable results of (2,21,22) and (3):

$$\log(\mathit{soly}/\text{mol \% Ni}) = -2.33 - 1570\,(T/\text{K})^{-1} \qquad \text{Eq.(3)}$$

The difference between Eq.(1) and Eq.(3) is significant. The inclusion of further data of (4) and (7) would not significantly change this relationship.
The other solubility equation which was presented in (23) cannot be recommended.

$$\log(\mathit{soly}/\text{mol \% Ni}) = -2.067 - 1830\,(T/\text{K})^{-1} \qquad \text{Eq.(4)}$$

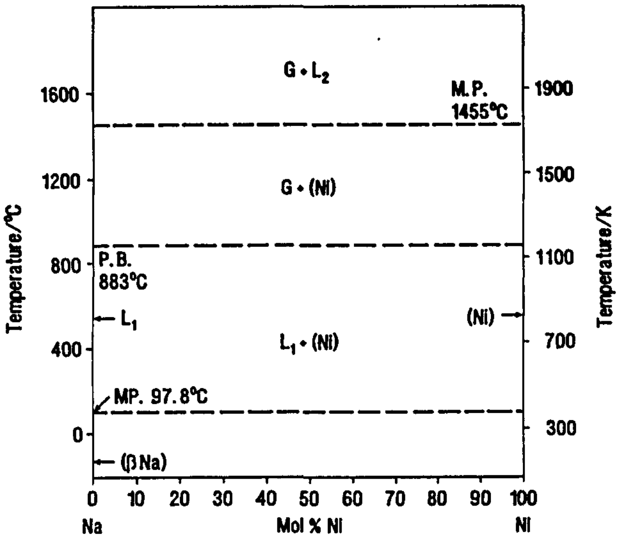
The data of (5) seem to be moderately overstated. The high values reported by (8) are probably influenced by impurities in Na. A simple explanation of the values of (9) which are one order of magnitude below the fitting Eq. (4) cannot be given. In some data of (24) the similar tendency may be due to the interference of Fe and Ni under the experimental conditions. An interaction of Fe and Ni in the binary system was observed (25). The data selected in this evaluation are slightly different from those calculated by means of Eqs. (3) or (4).
Ni-Na intermetallics do not exist and the solid solubility of Na in Ni is expected to be very low, as it is shown in the schematic phase diagram. Thus, we may assume that almost pure Ni is in equilibrium with the saturated solution in Na. A precipitation of traces of Na_2NiO_2 was observed in Na containing O (7,26). An alternative formation of $NaNiO_2$ in the Na-Ni-O system was postulated (26,27). Mathews (28) claimed that Na-Ni-O ternary compounds should not be stable in the presence of liquid Na.

Tentative values of the solubility of Ni in liquid Na at low O concentration in Na

T/K	*soly*/mol % Ni	source
673	$1\ 10^{-5}$	(7)
773	$3 \cdot 10^{-5}$	(3)
873	$1\ 10^{-4}$	(2), (3) and (18), mean value, interpolated
973	$2 \cdot 10^{-4}$	(24)

COMPONENTS: (1) Nickel; Ni; [7440-02-0] (2) Sodium; Na; [7440-23-5]	EVALUATOR: H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland August 1995

CRITICAL EVALUATION: (continued)



References

1. Thorley, A.W.; Tyzack, C. *Alkali Metal Coolants*, IAEA, Vienna, 1967, p. 97.
2. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. AI-AEC-12955*, 1970.
3. Singer, R.M.; Weeks, J.R. *US Atom.Ener.Comm.Rep.ANL-7520*, Pt.I, 1968, p. 309.
4. Lee, P.K.; Berkey, E. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt.I, 1968, p. 299.
5. Pellett, C.R.; Thompson, R. *Liq.Met. Engin. & Technol.*, BNES, London, 1984, *3*, 43.
6. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113.
7. Kovacina, T.A.; Miller, R.R. *Naval Res.Lab.Rep. NRL-6890*, 1969; *Rep. AD-688780*, 1969.
8. Rodgers, S.J.; Mausteller, J.W.; Batutis, E.F. *Mine Safety Appliances Rep. TR-27*, 1954; *US Atom.Ener. Comm. Rep. NP-5241*, 1954.
9. Kovacina, T.A.; Miller, R.R. *Nucl. Sci. Engin.* 1961, *10*, 163.
10. Beskorovainyi, N.M.; Krasin, V.P.; Kirillov, V.B. *Fiz.-Khim. Mekhan.Mater.* 1984, *20*, no. 5, 26.
11. Bhat, N.P.; Borgstedt, H.U. *Werkstoffe und Korrosion* 1988, *39*, 115.
12. Beskorovainyi, N.M.; Krasin, V.P.; Lyublinskii, I.E. *Fiz.-Khim. Obrabot. Mater.* 1987, no. 1, 107.
13. Cheng, C.E.; Ruther, W.E. *Corrosion* 1972, *28*, 20.
14. Klueh, R.L.; DeVan, J.H. *J. Less-Common Met.* 1973, *30*, 9.
15. Kozlov, F.A.; Zagorulko, Yu.I.; Bogdanovich, N.G.; Solovev, V.A.; Zotov, V.V.; Starkov, O.V.; Kozub, P.S.; Kovalev, Yu.P.; Buchelnikov, V.M.; Kraev, N.D. *USSR Rep. FEI-510*, 1974.
16. 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.
17. Alekseev, V.V.; Kozlov, F.A.; Zagorulko, Yu.I., *Liquid Metal Systems*, H.U. Borgstedt, G. Frees, Eds., Plenum, N.Y., 1995, p. 113.
18. Beskorovainyi, N.M.; Ioltukhovskii, A.G.; Filipkina, E.I.; Krasin, V.P.; Radin, I.V. *Materialy dla Atomnoi Tekhniki*, Energoatomizdat, Moskva, 1983, p. 23.
19. Claar, T.D. *React. Technol.* 1970, *13*, 124.
20. Awasthi, S.P.; Borgstedt, H.U. *J. Nucl. Mater.* 1983, *116*, 103.
21. McKisson, R.L.; Eichelberger, R.L.; Gehri, D.C.; Guon, J. *US Atom.Ener.Comm. Rep. AI-AEC-12721*, 1968, p. 279.
22. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt. I; 1968, p. 319.
23. Weeks, J.R.; Isaacs, H.S. *Adv. Corrosion Sci. Technol.* 1973, 3, 1; *US Atom.Ener.Comm. Rep. BNL-15731R*, 1971.
24. Awasthi, S.P.; Borgstedt, H.U.; Frees, G. *Liq.Met. Engin. & Technol.*, BNES, London, 1984, *1*, 265.
25. Boom, R.; deBoer, F.R.; Niessen, A.K.; Miedema, A.R. *Physica.B* 1983, *115*, 285.
26. Wu, P.C.S.; Chiotti, P.; Mason, J.T. *US Dep.Ener. Rep. CONF-760503-P2*, 1976, p. 638.
27. Lindemer. T.B.; Besmann, M.; Johnson, C.E. *J. Nucl. Mater.* 1981, *100*, 178.
28. Mathews, C.K. *High Temp.Sci.* 1988-89, *26*, 377.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

Rodgers, S.J.; Mausteller, J.W.; Batutis, E.F.

Mine Safety Appliances Rep. TR-27, 1954; *US Atom. Ener.Comm. Rep. NP-5241*, 1954.

VARIABLES:

Temperature: 477-811 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Na was determined. Since the original report was not available, the data were taken from the text and figure in (1).

t/°C	*soly*/mass % Ni	*soly*/mol % Ni [a]
204	$4 \cdot 10^{-4}$	$1.6 \cdot 10^{-4}$
538 [b]	$1.0 \cdot 10^{-3}$	$3.9\ 10^{-4}$
538 [c]	$1.0 \cdot 10^{-3}$	$3.9 \cdot 10^{-4}$

[a] as calculated by the compilers; [b] static test; [c] dynamic test.

An increase of the O concentration in Na did not change the apparent solubility of Ni in liquid Na in these tests.

The solubility data of the original work according to (2,3) are somewhat different:

t/°C	*soly*/mass % Ni	*soly*/mol % Ni [a]
200	$6.5\ 10^{-4}$	$2.6 \cdot 10^{-4}$
302	$1.0 \cdot 10^{-3}$	$3.9\ 10^{-4}$
398	$1.4 \cdot 10^{-3}$	$5.5 \cdot 10^{-4}$
496	$1.7 \cdot 10^{-3}$	$6.6 \cdot 10^{-4}$

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed in (i) dynamic loops made of Ni, and (ii) static containers made of Ni or stainless steel. Na was equilibrated in the apparatus, samples were taken by means of dip sampling using a stainless steel or glass bucket introduced through a sampling valve. Ni was determined by means of wet chemical methods using dimethylglioxime.

SOURCE AND PURITY OF MATERIALS:

Ni: nothing specified.
Na: (i) contained 0.011 % O; (ii) contained $5 \cdot 10^{-3}$ to $6.5\ 10^{-2}$ % O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Claar, T.D. *React. Technol.* 1970, *13*, 124.
2. McKisson, R.L.; Eichelberger, R.L. *NASA Rep. CR-610*, 1966.
3. Lee, P.K.; Berkey, E. *US Atom.Ener.Comm. Rep. ANL-7520*, 1968, p. 299.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

Kovacina, T.A.; Miller, R.R.

Nucl. Sci. Engin. 1961, *10*, 163-166.

VARIABLES:

Temperature: 473-873 K; O concentration in Na:
$2 \cdot 10^{-3}$ - 0.113 mass % ($3 \cdot 10^{-3}$ - 0.162 mol %)

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Na was determined at several temperatures und O contents in the liquid metal.

t/°C	*O concn in Na*/mass %	*O concn in Na*/mol % [a]	*soly*/mass % Ni	*soly*/mol % Ni [a]
200	$5 \cdot 10^{-3}$	$7 \cdot 10^{-3}$	$5 \cdot 10^{-7}$	$2 \cdot 10^{-7}$
300	$4 \cdot 10^{-3}$	$6 \cdot 10^{-3}$	$6 \cdot 10^{-7}$	$2.3 \cdot 10^{-7}$
350	$3 \cdot 10^{-3}$	$4 \cdot 10^{-3}$	$6 \cdot 10^{-7}$	$2.3 \cdot 10^{-7}$
400	$5 \cdot 10^{-3}$	$7 \cdot 10^{-3}$	$4 \cdot 10^{-7}$	$1.6 \cdot 10^{-7}$
500	$2 \cdot 10^{-3}$	$3 \cdot 10^{-3}$	$9 \cdot 10^{-7}$	$3.5 \cdot 10^{-7}$
600	$3 \cdot 10^{-3}$	$4 \cdot 10^{-3}$	$1.2 \cdot 10^{-6}$	$4.7 \cdot 10^{-7}$

The effect of the O concentration in Na on the solubility of Ni in liquid Na at 600 °C:

O concn in Na/mass %	*O concn in Na*/mol % [a]	*soly*/mass % Ni	*soly*/mol % Ni [a]
$3\ 10^{-3}$	$4 \cdot 10^{-3}$	$1.2 \cdot 10^{-6}$	$4.7 \cdot 10^{-7}$
$4.3\ 10^{-2}$	$6.0\ 10^{-3}$	$3.8 \cdot 10^{-6}$	$1.5 \cdot 10^{-6}$
$7.7 \cdot 10^{-2}$	0.108	$1.4 \cdot 10^{-5}$	$5.5 \cdot 10^{-6}$
0.113	0.162	$2.2 \cdot 10^{-5}$	$8.6 \cdot 10^{-6}$

[a] calculated by the compilers

The equilibration time of 2 h was found to be effective in separate experiments.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Radioactive ^{63}Ni was electroplated onto the inner surface of a Ni crucible, which was previously electropolished and annealed at 1273 K in H_2 atmosphere. The plated crucible was degassed in high vacuum at 873 K. Distilled and filtered Na was introduced into the equilibration apparatus in an Ar atmosphere. The temperature was raised and controlled by a furnace. Na was stored during the procedure. Samples of Na were taken out after 2-90 h of exposure and analyzed for their contents of Ni and O. Ni was separated by a standard carrier technique, the characteristic weak beta emission was counted. The O concentration in Na was increased by oxidation with a measured quantity of dry O_2.

SOURCE AND PURITY OF MATERIALS:

Ni: electroplated from ammoniacal $NiSO_4$ solution of pH=12.
Na: vacuum distilled after filtration, final content of ~$3 \cdot 10^{-3}$ % O.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

Lee, P.K.; Berkey, E.

US Atom.Ener.Comm. Rep. ANL-7520, Pt.I, 1968, p. 299-308.

VARIABLES:

Temperature: 570-722 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Na was measured.

t/°C	*time*/h	*soly*/mass %·10^{-4} Ni	*soly*/mol %·10^{-4} Ni [a]	*average soly*/mol % Ni [a]
297	25	0.92; 0.55; 0.38; 0.20	0.36; 0.21; 0.15; 0.078	1.6·10^{-5}
376	8	0.24; 0.17; 0.93; 0.73; 0.96;, 1.7; 1.8; 0.45; 0.10; 0.54	0.094; 0.066; 0.36; 0.28, 0.37; 0.66; 0.70; 0.18; 0.039; 0.21	2.7 10^{-5}
449	5	4.9; 2.8; 3.1; 1.4; 2.3; 1.0	1.9; 1.1; 1.2; 0.55; 0.90; 0.39	8.6·10^{-5}

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus consisted of a Mo capsule with a Ni equilibration crucible and a Ta collector crucible tightened together by means of springs. The parts were rinsed with H_2O, acetone, CH_3OH, and finally outgassed under vacuum at 773-873 K for several hours. They were then transferred into a dry Ar box and heated again 1 h at 423 K. The crucible was loaded with Na and the parts were tightly screwed. The capsule was placed in a vacuum chamber, the furnace was heated for the given temperature and time. The equilibration temperatures were measured using two Pt/Pt-Rh(10%) thermocouples. After the equilibration, the chamber was inverted to allow the Na to flow into the Ta collector. The assembly was cooled by means of spraying with liquid N. The vacuum chamber was opened, and the capsule transferred into the Ar box, where it was opened. Na was melted in the collector. Na samples were removed by means of a stainless steel tube and analyzed for their Ni contents by spark-source mass spectrography in the Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ni: 99.997 % pure from Materials Research Corp..
Ta: 99.997 % pure from Materials Research Corp..
Na: "high purity" from Mine Safety Res. Corp., containing 1.4·10^{-3} % O (author's analysis), <5·10^{-3} % C, <2·10^{-2} % K and (0.2-1.2)·10^{-4} % Ni.
Ar: "high purity", containing <1·10^{-4} % H_2O and < 2 10^{-4} % O_2.

ESTIMATED ERROR:

Solubility: analytical precision ±(5-10) %, standard deviation about ± 80 %.
Temperature: stability ± 1 K.

REFERENCES:

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

Singer, R.M.; Weeks, J.R.

US Atom.Ener.Comm.Rep.ANL-7520, Pt.I, 1968, p. 309-318.

VARIABLES:

Temperature: 613-873 K
O concentration in Na: $1.4\ 10^{-3}$ - $3.1\cdot 10^{-2}$ mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Na was determined and the influence of O in Na on the solubility was studied.

t/°C	*O concn in Na*/mass %	*O concn in Na*/mol % [a]	*soly*/mass % Ni	*soly*/mol % Ni [a]
340-420	$(1.0-2.0)\cdot 10^{-3}$	$(1.4-2.8)\cdot 10^{-3}$	$< 5\cdot 10^{-5}$	$< 2\cdot 10^{-5}$
446	$2.1\ 10^{-3}$	$2.9\cdot 10^{-3}$	$(6.7\pm 1.4)\cdot 10^{-5}$	$(2.6\pm 0.5)\cdot 10^{-5}$
474	$2.5\cdot 10^{-3}$	$3.5\cdot 10^{-3}$	$(7.6\pm 1.4)\cdot 10^{-5}$	$(3.0\pm 0.5)\ 10^{-5}$
485	$2.2\cdot 10^{-3}$	$3.1\cdot 10^{-3}$	$(8.9\pm 6.6)\cdot 10^{-5}$	$(3.5\pm 2.6)\cdot 10^{-5}$
500[b]	$5.2\cdot 10^{-3}$	$7.3\cdot 10^{-3}$	$(8.9\pm 1.9)\cdot 10^{-5}$	$(3.5\pm 0.7)\cdot 10^{-5}$
525	$3.3\cdot 10^{-3}$	$4.6\cdot 10^{-3}$	$(1.19\pm 0.59)\cdot 10^{-4}$	$(4.6\pm 2.3)\cdot 10^{-5}$
530	$3.1\ 10^{-3}$	$4.3\cdot 10^{-3}$	$(5.3\pm 3.3)\ 10^{-5}$	$(2.1\pm 1.3)\cdot 10^{-5}$
575	$6.9\cdot 10^{-3}$	$9.7\cdot 10^{-3}$	$(1.24\pm 0.26)\cdot 10^{-4}$	$(4.8\pm 1.0)\cdot 10^{-5}$
600	$4.6\cdot 10^{-3}$	$6.4\ 10^{-3}$	$(1.33\pm 0.48)\cdot 10^{-4}$	$(5.2\pm 1.9)\cdot 10^{-5}$

[a] as calculated by the compilers

[b] samples were taken after fast increase of temperature to 600 °C

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ni crucible was leak-checked and H_2 fired. The crucible was sealed into a solubility chamber together with a Mo sampler. Crucible and sampler were again treated with H_2 at 873 K for 1 h. The stainless steel chamber, lined with Mo, was outgassed for 1 day at a temperature exceeding the highest experimental temperature. Na was then distilled through a Nb-Zr(1%) tube into the crucible. The system was pressurized with He, heated to the desired temperature and equilibrated for up to 24 h. The twin sampling buckets were then lowered into the melt and kept there for 15 min. The chamber was opened in a dry box. The samples were dissolved in butyl chloride. The cups were leached with 6 $mol\cdot dm^{-3}$ HCl for 1 h at 373 K. Ni was spectrophotometrically determined as the dimethylglyoxime complex.

SOURCE AND PURITY OF MATERIALS:

Ni: 99.99 % pure from United Mineral and Chem. Co., with contents of $4.6\cdot 10^{-3}$ % total metallic impurities, $5.8\cdot 10^{-3}$ % sum of O, C, N.
Na: hot trapped, passed through 5 μm stainless steel filter, vacuum distilled; final contents of (1.0-2.0) 10^{-4} % Ni and $(1.0-2.0)\cdot 10^{-3}$ % O.
He: "ultrapure" with contents of $4.5\ 10^{-5}$ % O and $8\cdot 10^{-5}$ % H_2O after purification.

ESTIMATED ERROR:

Solubility: standard deviation up to ± 75 %, typically about ± 25 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

Kovacina, T.A.; Miller, R.R.

Naval Res. Lab. Rep. NRL-6890, 1969.

VARIABLES:

Temperature: 573-773 K
O concentration in Na: $2.9 \cdot 10^{-4}$ mol % O to saturation

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Na at various O concentrations in Na was determined. NiO was equilibrated with Na.

t/°C	*O concn in Na*/mass %	*O concn in Na*/mol % [a]	*soly*/mass % Ni	*soly*/mol % Ni [a]
300	saturated	saturated	$3.6 \cdot 10^{-4}$	$1.4 \cdot 10^{-4}$
400	$2.1 \cdot 10^{-4}$	$2.9 \cdot 10^{-4}$	$2.9 \cdot 10^{-5}$	$1.1 \cdot 10^{-5}$
400	$3.06 \cdot 10^{-4}$	$4.4 \cdot 10^{-4}$	$6.3 \cdot 10^{-5}$	$2.5 \cdot 10^{-5}$
400	$7.86 \cdot 10^{-4}$	$1.13\ 10^{-3}$	$1.05 \cdot 10^{-4}$	$4.1 \cdot 10^{-5}$
400	$6.21 \cdot 10^{-3}$	$8.9 \cdot 10^{-3}$	$1.12 \cdot 10^{-3}$	$4.4 \cdot 10^{-4}$
400	$1.278 \cdot 10^{-2}$	$1.79.1 \cdot 10^{-2}$	$1.16 \cdot 10^{-3}$	$4.5 \cdot 10^{-4}$
400	saturated	saturated	$1.38 \cdot 10^{-3}$	$5.4 \cdot 10^{-4}$
400	saturated	saturated	$1.36 \cdot 10^{-3}$	$5.3 \cdot 10^{-4}$
400	saturated	saturated	$6.6 \cdot 10^{-4}$	$2.6 \cdot 10^{-4}$ [b]
500	saturated	saturated	$2.13 \cdot 10^{-3}$	$8.3 \cdot 10^{-4}$

[a] as calculated by the compilers   [b] the result is underestimated as stated by the authors

The equilibrium solid phase was found to be Ni with trace amounts of Na_2NiO_2.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

All operations with metallic Na were performed in an Ar atmosphere. The equilibrium apparatus included a hermetically stainless steel sealed chamber connected with a Na still. A Mo crucible inside of the chamber was filled with directly distilled Na and equilibrated several days with various amounts of pelletized NiO. The Na sample was filtered and the Ni content was determined by thermal neutron activation analysis. The filtrate was dissolved in absolute C_2H_5OH to which H_2O was added. An aliquot of the resulting solution was titrated with acid. Fe(III) carrier was added to the entire solution. $Ni(OH)_2$ was qualitatively co-precipitated with $Fe(OH)_3$ by means of addition of NaOH. The deposit was transferred into Fe_2O_3 by ignition. The sample was irradiated in the NRL reactor, the formed ^{65}Ni was counted. The O concentration in Na was estimated on the basis of the weight of the NiO pellets which were added.

SOURCE AND PURITY OF MATERIALS:

NiO: reagent grade purity; pressed into pellets and fired at 1673 K for 15 h.
Na: vacuum distilled and filtered through a sintered glass frit.
Ar: purified.

ESTIMATED ERROR:

Solubility: sensitivity $3\ 10^{-6}$ mol % Ni.
Temperature: stability ± 2 K.

REFERENCES:

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

(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 Ni in liquid Na was measured at several temperatures.

t/°C	*soly*/mass % Ni	*soly*/mol % Ni [a]
600	$1.04\ 10^{-2}$	$4.1 \cdot 10^{-3}$
650	$2.6\ 10^{-4}$	$1.0 \cdot 10^{-4}$
700	$3.0 \cdot 10^{-4}$, $6.5 \cdot 10^{-3}$	$1.2 \cdot 10^{-4}$, $2.5 \cdot 10^{-3}$
750	$1.1 \cdot 10^{-4}$	$4.3\ 10^{-5}$
800	$4.0 \cdot 10^{-4}$, $<2\ 10^{-4}$, $1.14\ 10^{-2}$	$1.6\ 10^{-4}$, $< 7.8 \cdot 10^{-5}$, $4.4 \cdot 10^{-3}$
850	$2.5 \cdot 10^{-4}$	$9.7 \cdot 10^{-5}$
900	$4.9\ 10^{-4}$, $4.59\ 10^{-2}$	$1.9\ 10^{-4}$, $1.8 \cdot 10^{-2}$
910	$< 5\ 10^{-4}$	$< 1.9 \cdot 10^{-4}$
914	$< 5\ 10^{-4}$	$< 1.9\ 10^{-4}$
1000	$2.1 \cdot 10^{-3}$	$8.2 \cdot 10^{-4}$

[a] as calculated by the compilers

The results were previously presented in (1) and (2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ni test crucible was cleaned, and finally washed with H_2O and acetone. A collector for Na samples saturated with Ni was made of Ti, W or Nb-Zr alloy. The crucible-collector assembly was degassed at 623 K for 2 hours. The crucible was loaded with Na and the assembly sealed by welding in high vacuum. A capsule with the assembly inside was equilibrated at the desired temperature for 6 hours in an Ar atmosphere. The capsule was then inverted causing the Na sample to flow into the collector. The collector was cooled, opened and Na analyzed for its Ni content by means of atomic absorption spectroscopy.

SOURCE AND PURITY OF MATERIALS:

Ni: 99.997 % pure with contents of $1.7 \cdot 10^{-3}$ % C, $2 \cdot 10^{-5}$ % H, $1.8 \cdot 10^{-3}$ % O, $3 \cdot 10^{-4}$ % N, $1.2 \cdot 10^{-3}$ % Fe and $\leq 1.5 \cdot 10^{-4}$ other elements (each).
Na: 99.996 % pure with contents of $1.9 \cdot 10^{-3}$ % C and $1.5 \cdot 10^{-4}$ % O.
Ar: unspecified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. McKisson, R.L.; Eichelberger, R.L.; Gehri, D.C.; Guon, J. *US Atom.Ener.Comm.Rep. AI-AEC-12721*, 1968, p.279-308.
2. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener. Comm.Rep. ANL-7520*, Pt.I, 1968, p. 319-324.

COMPONENTS: (1) Nickel; Ni; [7740-02-0] (2) Sodium; Na; [7740-23-5]	**ORIGINAL MEASUREMENTS:** Klueh, R.L.; DeVan, J.H. *J. Less Common Met.* 1973, *30*, 9-24.
VARIABLES: One temperature: 873 K Concentration of O: 0.28 - 0.29 mol %	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ni concentration in liquid Na was determined after the exposure of stainless steel and V specimens to Na at 600 °C.

Exp. time/h	*O concn*/mass %	*O concn*/mol % [a]	*soly*/mass % Ni	*soly*/mol % Ni [a]
100	0.2000	0.28	$3\cdot10^{-4}$	$1.2\cdot10^{-4}$
200	0.205	0.29	$2\cdot10^{-4}$	$8\ 10^{-5}$
300	0.205	0.29	$1\ 10^{-4}$	$4\cdot10^{-5}$
400	0.205	0.29	$3\cdot10^{-4}$	$1.2\cdot10^{-4}$

[a] calculated by the compilers

Na seems to be equilibrated with Ni, however, the chemical activity of Ni in the steel is significantly lower than unity (as in pure Ni). The formation of intermetallics of Ni with V may interfere with the solubility measurements.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A stainless steel capsule containing a V specimen was filled with Na. The capsule was covered with a Ta foil liner and inserted into a stainless steel protective container. The Na sample was equilibrated in the capsule at 873 K for exposure times 100 - 500 h. After the test, the capsule was inverted and quenched in liquid N. The O concentration was determined by either vacuum fusion or fast neutron activation analysis. The Ni content in Na was determined by spectrographic analysis after dissolution of the Na sample in isopropyl alcohol. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ni: from 304L stainless steel which contained ~ 10 % Ni, 18 % Cr, < 0.03 % C, 2 % Mn, < 1 % Si and the rest Fe.
Na: purified, probably by filtration at low temperature and gettering with Zr chips at 873 K.
Ar: unspecified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Nickel; Ni; [7440-02-0] (2) Sodium; Na; [7440-23-5]	Awasthi, S.P.; Borgstedt, H.U.; Frees, G. *Liq. Met. Engin. Technol.*, BNES, London, 1984, *1*, 265-269.
VARIABLES:	**PREPARED BY:**
Temperature: 773-973 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Na was determined at some temperatures.

t/°C	*time*/h	*soly*/mass % Ni	*average soly*/mol % Ni [a]
500	19	$3.0 \cdot 10^{-5}$, $1.7 \cdot 10^{-5}$, $2.4 \cdot 10^{-5}$	$(9.0 \pm 1.6) \cdot 10^{-6}$
690	4	$1.6 \cdot 10^{-5}$, $9 \cdot 10^{-5}$, $9 \cdot 10^{-5}$, $9 \cdot 10^{-5}$	$(4.3 \pm 0.8) \cdot 10^{-6}$
700 [b]	-	$4 \cdot 10^{-4}$, $6.5 \cdot 10^{-4}$ [b]	$2.0 \cdot 10^{-4}$ [b]

[a] as calculated by the compilers,

[b] a graphically presented result from circulating Na in a test loop

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus was made of AISI 304 stainless steel and consisted of an outer chamber, an inner chamber and a sampler. The whole assembly was placed in an Ar atmosphere and was calibrated with respect to temperature setting. A Ni test crucible and a sampling Ta crucible were cleaned in mixture of CH_3COOH, H_2SO_4 and HNO_3 (5:1:3) for 15 s at 363 K, then rinsed in H_2O and dried at 383 K. The test crucible was placed in the inner chamber and filled with Na. The equilibration in the test crucible was done in the presence of Fe. After the equilibration Na was transferred into the sampling crucible. The Na sample was taken out, cooled, weighed and dissolved in H_2O in an Ar atmosphere. Ni was determined by atomic absorption spectroscopy.

SOURCE AND PURITY OF MATERIALS:

Ni: nothing specified.
Na: purified by removing its surface precipitate at 383 K.
H_2O: ultrapure.
Ar: purified, containing $<1 \cdot 10^{-4}$ % O and $< 1 \cdot 10^{-4}$ % H_2O.

ESTIMATED ERROR:

Solubility: standard deviation better than ± 20 %.
Temperature: precision better than ± 5 K (by the compilers).

REFERENCES:

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

Pellett, C.R.; Thompson, R.

Liq. Met. Engin. & Technol., BNES, London, 1984, *3*, 43-48.

VARIABLES:

Temperature: 573-948 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Na was determined at various temperatures.

t/°C	*test type*	*concen.range*/mass % Ni	*aver.soly*/mass % Ni	*soly*/mol % Ni [a]
300	column	$(1.3\text{-}4.0)\cdot 10^{-4}$	$2.02\ 10^{-4}$	$7.9\ 10^{-5}$
350	can	-	$2.92\cdot 10^{-4}$	$1.14\cdot 10^{-4}$
350	column	$(0.37\text{-}3.2)\cdot 10^{-4}$	$1.25\cdot 10^{-4}$	$4.9\cdot 10^{-5}$
365	can	-	$1.55\ 10^{-4}$	$6.0\cdot 10^{-5}$
400	can	$(0.65\text{-}11.7)\ 10^{-4}$	$4.13\ 10^{-4}$	$1.61\cdot 10^{-4}$
400	column	$(0.2\text{-}5.4)\cdot 10^{-4}$	$1.79\cdot 10^{-4}$	$7.0\ 10^{-5}$
450	can	$(1.14\text{-}3.7)\ 10^{-4}$	$2.48\ 10^{-4}$	$9.7\ 10^{-5}$
450	column	$(0.16\text{-}6.9)\ 10^{-4}$	$1.67\cdot 10^{-4}$	$6.5\cdot 10^{-5}$
470	can	-	$1.65\ 10^{-4}$	$6.4\cdot 10^{-5}$
500	can	$(0.69\text{-}1.4)\ 10^{-4}$ [b]	$4.05\cdot 10^{-4}$	$1.58\cdot 10^{-4}$
500	column	$(0.4\text{-}11.5)\cdot 10^{-4}$	$2.66\cdot 10^{-4}$	$1.04\ 10^{-4}$
550	can	$(0.5\text{-}18.7)\ 10^{-4}$	$4.02\ 10^{-4}$	$1.57\cdot 10^{-4}$
570	can	$(5.8\text{-}6.2)\ 10^{-4}$	$6.0\ 10^{-4}$	$2.3\ 10^{-4}$
575	can	$(1.3\text{-}10.1)\ 10^{-4}$	$5.7\cdot 10^{-4}$	$2.2\cdot 10^{-4}$
600	can	$(0.85\text{-}10.7)\ 10^{-4}$	$5.1\ 10^{-4}$	$2.0\ 10^{-4}$
650	can	$(0.76\text{-}14.3)\cdot 10^{-4}$	$4.29\ 10^{-4}$	$1.67\cdot 10^{-4}$
655	can	-	$2.34\cdot 10^{-4}$	$9.1\cdot 10^{-5}$
675	can	$(4.0\text{-}14.2)\ 10^{-4}$	$9.1\cdot 10^{-4}$	$3.5\ 10^{-4}$

[a] as calculated by the compilers;
[b] the upper limit seems to be 10 times higher

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test in a "can" was performed as in (1). A Ni can was degreased in acetone and cleaned in hot 5 % solution of Decon 75 prior to being H_2 fired at 1023 K for 24 h; the dissolved H_2 was subsequently pumped off under vacuum at 1023 K. The can was filled with Na through a Ni 1.5 μm pore size filter frit at 423 K. A Ni lid was welded onto the can which was heated to the temperature of interest for 24 h. A Na sample was taken by piercing the can lid with a Mo needle and with-drawing it through a Ni 1.5 μm pore size filter. The sample was collected in a weighed alumina crucible (previously cleaned in boiling aqua regia) and dissolved in CH_3OH. Ni was separated from the bulk Na by a wet chemical co-precipitation. All operations were performed in an Ar atmosphere. The precipitate was dissolved in concentrated HCl and the solution analyzed for its Ni content by atomic absorption spectroscopy. The test in "column" was performed in an apparatus which consisted of 2 parallel Ni chambers.

The first was a Na reservoir and the second was a column with a 1.5 μm pore size filter. The column was maintained at a chosen temperature by a Cu-block furnace around it.

Na was forced by Ar pressure from the reservoir into the column and equilibrated. The Na sample was collected from the column filter and analyzed in the same way as in the "can" method.

SOURCE AND PURITY OF MATERIALS:

Ni: nothing specified.
Na: vacuum distilled, further treated by electrolytical passing through a β alumina tube; final contents of $< 8\cdot 10^{-5}$ % Ni and $(1.0\text{-}2.0)\ 10^{-3}$ % O.

ESTIMATED ERROR:

Solubility: precision ± 30 % at $2\cdot 10^{-4}$ % Ni and ± 15 % at $1\ 10^{-3}$ % Ni for a confidence limit of 95 %.
Temperature: nothing specified.

REFERENCES:

1. Stanaway, W.P.; Thompson, R. *Material Behavior & Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 421.

COMPONENTS: (1) Nickel; Ni; [7440-02-0] (2) Potassium; K; [7440-09-7]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1989

CRITICAL EVALUATION:

In compatibility tests of Ni specimens in K-Na mixture at 873 K in static as well as dynamic conditions, no appreciable solubility of Ni in the melt was noticed, since the specimens did not show any weight losses (1). Aleksandrov and Dalakova (9) did not detect any dissolved Ni in liquid K after an equilibration of 1 h at 873-923 K; they did not mention the detection limit of the spectral analysis used.
Swisher (2) as well as Ginell and Teitel (3,4) performed independent determinations of the Ni solubility in liquid K in the temperature ranges 941-1328 K and 1198-1273 K, respectively. Swisher (2) observed an increase of the solubility from $3.6\ 10^{-4}$ to $3.7 \cdot 10^{-3}$ mol % Ni, whereas (3,4) detected an increase from $2.0 \cdot 10^{-4}$ to $2.7 \cdot 10^{-4}$ mol % Ni. Thus, the results in (3, 4) are one order of magnitude lower than in (2). The values of Swisher (2) are rather uncertain, since a large blank correction of $6 \cdot 10^{-4}$ mol % was necessary in his analytical procedure.
Ordynskii et al. (5) determined the Ni solubility in liquid K at 971 to 1286 K. A stainless steel with 9 mass % Ni served as the solute. Three different analytical procedures were used; the scatter of the results was similar in all cases. Since Ni is not the dominating component of the alloy and may also interact with Fe and Cr (6,7), its activity is surely decreased. The observed solubilities were still lower than in (3, 4); the mean values changed from $1.3 \cdot 10^{-5}$ to $5.5 \cdot 10^{-5}$ mol % Ni. It should be noticed that the slopes of temperature dependencies of the solubility in (2), (3, 4) and (5) are similar.
Schwarz (8) reported the Ni solubility in liquid K at 373 K of $4.1 \cdot 10^{-5}$ mol % Ni using an austenitic stainless steel as the solute; K contained $5 \cdot 10^{-2}$ mol % O. Further details of the testing procedure were not reported. The result does not fit any earlier data extrapolated from higher temperature and is probably overstated.
Nevertheless, (3) observed a significant increase of the solubility of Ni in liquid K to a value of $7.6 \cdot 10^{-3}$ mol % Ni at 1273 K, if the O content of K was $6 \cdot 10^{-3}$ mol % O. These observations suggest that O influences the Ni solubility. However, the effect seems to be smaller compared to that of the Fe-K system.
The solubility data of (3,4) are suggested in this evaluation. A Ni-K phase diagram is not available; it should be similar to that shown for the Ni-Na system.

Tentative values of the solubility of Ni in liquid K:

T/K	*soly*/mol % Ni	source
1200	2×10^{-4} [a]	(3,4)
1300	3×10^{-4} [a]	(3,4) extraploation

[a] at a pressure sufficient to keep K in the liquid state

References:

1. Kelman, L.R. *U.S. Atom. Ener. Comm. Rep. CT-3726*, 1946.
2. Swisher, J.H. *NASA Rep. TN-D-2734*, 1965; abstracted in *U.S.Atom.Ener.Comm. Rep. CONF-650411*, 1965, p. 43.
3. Ginell, W.S.; Teitel, R.J. *U.S. Atom. Ener. Comm. Rep. CONF-650411*, 1965, p. 44; *NASA Rep. CR-82838*, 1965; *Rep. 67-N-19585*, 1967.
4. Ginell, W.S.; Teitel, R.J. *Trans.Am.Nucl.Soc.* 1965, *8*, 393.
5. Ordynskii, A.M.; Popov, R.G.; Raikova, G.P.; Samsonov, N.V.; Tarbov, A.A. *Teplofiz. Vys. Temp.* 1981, *19*, 1192.
6. DeBoer, F.R.; Boom, R.; Miedema, A.R. *Physica, B* 1982, *113*, 18.
7. Boom, R.; DeBoer, F.R.; Niessen, A.K.; Miedema, A.R. *Physica, B* 1983, *115*, 285.
8. Schwarz, N.F. *Liq. Met. Engin. Technol.*, BNES, London, 1985, *3*, 177.
9. Aleksandrov, B.N.; Dalakova, N.V. *Izv.Akad.Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

(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 Ni in liquid K at two temperatures was reported:

t/°C	*soly*/mass % Ni	*soly*/mol % Ni [a]
925	$2\cdot10^{-4}$, $3\ 10^{-4}$, $4\cdot10^{-4}$	$1.3\cdot10^{-4}$, $2.0\ 10^{-4}$, $2.7\ 10^{-4}$
1000	$4\ 10^{-4}$, $1.15\ 10^{-2}$ [b]	$2.7\cdot10^{-4}$, $7.6\ 10^{-3}$ [b]

[a] as calculated by the compilers

[b] reported in (1), at O concentration of $2.3\cdot10^{-3}$ mass %

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A "L" shaped capsule was made of Nb-Zr(1%). The capsule contained inside a Ni test cup, a W collector cup and a small dam positioned near the capsule bend to regulate the K flow into the collector. The capsule was chemically etched and outgassed at 1623-1673 K. It was filled with K, welded in vacuum and heated to 100 K above the test temperature for 1 h, while a centrifuge, in which the capsule was mounted, was rotated to prevent K flow over the dam. Then the test temperature was established for 3 hours and the rotation rate elevated to force Ni precipitation at the cup bottom. At the end of the experiment the rotation was slowly reduced to allow the decantation of K over the dam into the collector. The method of the chemical analysis was not reported. The temperatures of the experiments were obtained by means of thermocouples or by an optical method. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ni: 99.97 % pure.
K :purified by contacting with Ti-Zr (1:1) alloy chips at 1058 K, with a final content of $(7\text{-}11)\cdot10^{-4}$ % O.
Ar: high purity, containing $< 2\cdot10^{-4}$ % H_2O.

ESTIMATED ERROR:

Nothing specified.
Solubility : precision ± 30 % (by the compilers).

REFERENCES:

1. Ginell, W.S.; Teitel, R.J. *U.S. Atom. Ener. Comm. Rep. CONF-650411*, 1965, p. 44-47; *NASA Rep. CR-82838*, 1965; *Rep. 67-N-19585*, 1965.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Nickel; Ni; [7440-02-0] (2) Potassium; K; [7440-09-7]	Swisher, J.H. *NASA Rep. TN-D-2734*, 1965.
VARIABLES:	**PREPARED BY:**
Temperature: 941-1328 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid K was reported in the figure. The values were read out and recalculated to mol % by the compilers.

T/K	*sampler*	*soly*/mass % Ni	*soly*/mol % Ni
941	Mo	5.5×10^{-4}	3.6×10^{-4}
1031	Mo	6.2×10^{-4}	4.1×10^{-4}
1144	Nb	1.5×10^{-3}	9.9×10^{-4}
1144	Nb	2.1×10^{-3}	1.4×10^{-3}
1257	Nb	2.4×10^{-3}	1.6×10^{-3}
1257	Nb	2.5×10^{-3}	1.7×10^{-3}
1328	Nb	5.6×10^{-3}	3.7×10^{-3}

The following fitting equation was proposed by the author and tested by the compilers:

$$\log(soly/\text{mol \% Ni}) = -0.29 - 3040(T/\text{K})^{-1}$$

The work was abstracted and the equation reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A solubility test capsule was made of Ni. A sampling cup, made alternatively of Mo or Nb, was mounted inside the capsule. The capsule was filled with K and sealed by means of electron-beam welding in a high vacuum furnace for few hours. The temperature of the sample holder was controlled with Pt/Pt-Rh(13%) thermocouples. Sampling at the test temperature was accomplished by inverting the furnace which allowed the K sample to flow into the cup. After cooling to room temperature, the capsule was cut open and the K sample dissolved in butyl alcohol. The cups were leached with HCl to remove precipitates which may have formed during the cooling. Ni was colorimetrically determined in the combined solutions. The K amount was gravimetrically determined.

SOURCE AND PURITY OF MATERIALS:

Ni: 99.9904+ % pure, containing $5.0 \cdot 10^{-3}$ % C, $1.0 \cdot 10^{-3}$ % S, < $1.0 \cdot 10^{-3}$ % Co, Si, Mn, Cu, Fe, Cr (each).
K : contained < $2.0 \cdot 10^{-3}$ % Na.

ESTIMATED ERROR:

Solubility: standard deviation ± 27.8 %, blank determined to be about $6 \cdot 10^{-4}$ mol % Ni.
Temperature: stability ± 1 K, accuracy ± 3 K.

REFERENCES:

1. Swisher, J.H. *US Atom.Ener.Comm.Rep. CONF-650411*, 1965, p. 43.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

(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: 971-1286 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid K at various temperatures was reported in figures; the values were read out and recalculated to mol % by the compilers.

T/K	*soly*/mass % Ni	*soly*/mol % Ni	T/K	*soly*/mass % Ni	*soly*/mol % Ni
973	$1.1\cdot10^{-5}$ [a]	$7.3\cdot10^{-6}$	1169	$2.3\cdot10^{-5}$ [b]	$1.5\cdot10^{-5}$
972	$1.5\cdot10^{-5}$ [c]	$1.0\cdot10^{-5}$	1171	$2.6\ 10^{-5}$ [b]	$1.7\ 10^{-5}$
972	$1.7\cdot10^{-5}$ [a]	$1.1\cdot10^{-5}$	1170	$3.0\cdot10^{-5}$ [c]	$2.0\cdot10^{-5}$
972	$2.1\ 10^{-5}$ [c]	$1.4\cdot10^{-5}$	1168	$4.4\cdot10^{-5}$ [c]	$2.9\cdot10^{-5}$
972	$2.4\ 10^{-5}$ [a]	$1.6\cdot10^{-5}$	1172	$4.4\cdot10^{-5}$ [c]	$2.9\cdot10^{-5}$
971	$2.7\cdot10^{-5}$ [c]	$1.8\ 10^{-5}$	1171	$5.0\ 10^{-5}$ [b]	$3.3\ 10^{-5}$
974	$2.7\cdot10^{-5}$ [c]	$1.8\cdot10^{-5}$	1170	$7.0\ 10^{-5}$ [c]	$4.7\ 10^{-5}$
1080	$1.6\ 10^{-5}$ [a]	$1.1\ 10^{-5}$	1170	$8.1\cdot10^{-5}$ [a]	$5.4\cdot10^{-5}$
1081	$2.0\ 10^{-5}$ [c]	$1.3\cdot10^{-5}$	1170	$9.3\cdot10^{-5}$ [c]	$6.2\cdot10^{-5}$
1074	$2.1\cdot10^{-5}$ [a]	$1.4\ 10^{-5}$	1170	$1.2\cdot10^{-4}$ [c]	$8.0\cdot10^{-5}$
1075	$3.0\cdot10^{-5}$ [a]	$2.0\ 10^{-5}$	1285	$5.2\cdot10^{-5}$ [c]	$3.5\cdot10^{-5}$
1076	$3.2\ 10^{-5}$ [c]	$2.1\cdot10^{-5}$	1285	$6.3\cdot10^{-5}$ [c]	$4.2\cdot10^{-5}$
1074	$4.0\ 10^{-5}$ [a]	$2.7\ 10^{-5}$	1284	$7.1\cdot10^{-5}$ [a]	$4.7\cdot10^{-5}$
1074	$4.9\ 10^{-5}$ [c]	$3.3\ 10^{-5}$	1285	$9.6\ 10^{-5}$ [c]	$6.6\ 10^{-5}$
1074	$5.5\cdot10^{-5}$ [c]	$3.6\cdot10^{-5}$	1286	$8.0\cdot10^{-5}$ [c]	$5.3\ 10^{-5}$
1159	$1.8\cdot10^{-5}$ [a]	$1.2\cdot10^{-5}$	1284	$1.5\ 10^{-4}$ [c]	$1.0\cdot10^{-4}$
1171	$1.9\ 10^{-5}$ [c]	$1.3\ 10^{-5}$	1284	$8.8\cdot10^{-5}$ [a]	$5.8\cdot10^{-5}$
1171	$2.1\ 10^{-5}$ [c]	$1.4\ 10^{-5}$			

[a] by spectral analysis, H_2O cooled sample
[b] by spectrophotometric analysis, H_2O cooled sample
[c] by spectrophotometric analysis, liquid N cooled sample

The authors proposed the solubility equation, which was tested by the compilers:

$$\log\,(soly/\text{mol \% Ni}) = -\,2.086 - 2760\,(T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A test capsule was made of 12Kh18N10T steel. The capsule was loaded with K and a foil of the same steel, which filled up a half of the capsule. The capsule was welded, placed in an Ar filled furnace and equilibrated for longer than 3 hours. The capsule was inverted causing K to flow into the receiver part. It was then cooled down in H_2O in 50-70 s. The capsule was cut open and K samples were withdrawn with a bronze blade. Spectral analysis was carried out after preconcentration of the sample. In the second method the liquid K saturated at test temperature was directly poured into liquid N (1). A spectrometric analysis of the sample was performed. In the third method the capsule was heated in a centrifuge furnace at a temperature higher than the test temperature. The temperature was then lowered and kept constant, while the centrifuge rotated for 2-2.5 h.

The capsule was cooled down with H_2O during the rotation. The upper part of solified K was analyzed by spectrophotometric analysis.

SOURCE AND PURITY OF MATERIALS:

12Kh18N10T steel: 9,1 % Ni, ~ 70 % Fe, 18.2 % Cr, 1.5 % Mn, 0.05 % C, 0.8 % Si, 0.02 % S, 0.3 % Ti and 0.035 % P.
K: contained $(1\text{-}3)\cdot10^{-5}$ % Ni, $(7\text{-}10)\cdot10^{-4}$ % O, $(3\text{-}5)\cdot10^{-4}$ % C, $(2\text{-}4)\cdot10^{-6}$ % H.
Ar: unspecified.

ESTIMATED ERROR:

Solubility: detection limit 10^{-6} - 10^{-5} mass % Ni.
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) Nickel; Ni; [7440-02-0]

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

EVALUATOR:

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

CRITICAL EVALUATION:

Young and Arabian (1) determined the apparent solubility of components of the alloy Haynes-25 in liquid Rb at 1033 and 1200 K. The solubility at the lower temperature was below the detection limit of $7 \cdot 10^{-5}$ mol % Ni and at the higher temperature the mean value was $7 \cdot 10^{-4}$ mol % Ni. Since Ni may interact with the components of this alloy in which it is present only in 10 mass %, one may expect rather higher values of the solubility, if the solute is pure Ni.

Some additional information on the solubility of Ni in Rb could be extracted from corrosion tests of stainless steels in liquid Rb. In the experiments of Pinchback et al. (2) Ni seems to be more soluble than Cr or Fe in the temperature range 773-893 K, if Rb contains 2.7 mol % O and the solute is 304 stainless steel. In the investigations of Suzuki et al. (3) at 473 K, in which Rb contained about 22 mol % O, Ni seemed to be less soluble than Fe but more soluble than Mo or Cr from 316 stainless steel as a solute.

The Ni-Rb phase diagram should be similar to that of the Ni-Na system.

Doubtful value of the solubility of Ni in liquid Rb.

T/K	*soly*/mol % Ni	source	remark
1200	$7 \cdot 10^{-4}$	(1)	at elevated pressure

References

1. Young, P.F.; Arabian, R.V. *U.S. 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.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

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

U.S. 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 Ni (from Haynes-25 alloy) in liquid Rb at two temperatures was reported; the values were read out from the figure and recalculated to mol % Ni by the compilers.

t/°F	T/K	*soly*/mass % Ni	*soly*/mol % Ni
1400	1033	$< 1 \cdot 10^{-4}$	$< 7 \cdot 10^{-5}$
1700	1200	$1.5 \cdot 10^{-3}$, $1.0 \cdot 10^{-3}$, $6 \cdot 10^{-4}$, $<1 \cdot 10^{-4}$	$1.0 \cdot 10^{-3}$, $7 \cdot 10^{-4}$, $4 \cdot 10^{-4}$, $<7 \cdot 10^{-5}$

The mean value of the results at 1200 K is $5 \cdot 10^{-4}$ mol % Ni as calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An annealed Haynes-25 alloy sample and a Ta capsule were cleaned in HNO_3, H_2SO_4, HF and H_2O (2:2:1:5) mixture, rinsed with H_2O and dried in air. The capsule was loaded with the sample and Rb, closed by means of welding in an Ar Atmosphere, flame sprayed with Al_2O_3 and heated for 50 hours at the selected temperature. The capsule was then inverted, causing the Rb sample to flow into a Ta sampling cup. The cup was cooled to room temperature. After the solidification the cup was cut open, and its content was analyzed. The Rb sample was treated with anhydrous hexane, CH_3OH, H_2O and HCl.

The cup was finally purified with aqua regia, and the resulting solution was joined to the preceding one. The combined solutions were taken to dry. The residue was analyzed for the Ni content in the National Spectroscopic Laboratories.

SOURCE AND PURITY OF MATERIALS:

Haynes-25: from Superior Tube Co., containing 10.1 % Ni, 49.6 % Co, 20.4 % Cr, 15.3 % W, 1.7 % Fe, 1.4 % Mn, 0.1 % C, 0.4 % Si, 0.02 % P, 0.015 % S.

Rb: purified by filtration, gettering with Ti-Zr alloy at 866 K and vacuum distillation; it contained $(6\text{-}17) \cdot 10^{-4}$ % O.

ESTIMATED ERROR:

Solubility: detection limit of $1 \cdot 10^{-4}$ mass % Ni; analytical error ± 10 %.

Temperature: precision ± 3 K.

COMPONENTS:	EVALUATOR:
(1) Nickel; Ni; [7440-02-0] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1989

CRITICAL EVALUATION:

Tepper and Greer (1) measured the saturating concentration of Ni in liquid Cs at 1255 K using Haynes-25 alloy with 10 mass % Ni as the solute. The Ni concentration was below the detection limit of $1.4 \cdot 10^{-4}$ mol % Ni, if the equilibration time was longer than 10 hours. The chemical activity of Ni in this alloy is significantly less than unity; the exact value is, however, not known. It was mentioned in another report of these authors (2) that in the temperature range of 368-533 K the solubility of Ni is higher than of Fe, Mo or Nb; the experimental details were not described.

Godneva et al. (3) performed solubility determinations in the temperature range 423-573 K and observed a decrease of Ni concentration from $3.8\ 10^{-4}$ to $2.9 \cdot 10^{-4}$ mol % Ni. The concentration increased, however, to $2.3\ 10^{-3}$ mol % Ni at 573 K, if the O concentration in Cs was 0.8 mol %. The temperature gap, which was not investigated in the works (1) and (3), is quite large, but the results of (1) are in agreement with the sequence of results of (3).

Holley (4) equilibrated steels containing Ni with liquid Cs at 675 K and room temperature and the increase of Ni concentration observed in Cs was never higher than 0.12 mol % Ni. Winslow (5, 6) seldom observed an increase of the Ni concentration in Cs after equilibration for 500 hours at 673 K. Keddy (7) reported that Ni was not leached out from its alloy with Au, Si and Mn at 773 K as were Au and Si. No details of the experiments in (4-7) were reported. No corrosion attack of liquid Cs on Ni samples was observed by (8, 11,12) at temperatures up to 873 K and at 1273 K only an intergranular penetration of Cs in Ni was detected (9, 10).

The decrease of the apparent solubility with temperature and the increase of the solubility with increase of the O concentration in the system suggest the formation of a ternary oxide in the system, which was actually observed by (3, 12).

The Ni-Cs phase diagram has not been reported in the literature so far, but should be similar to that of the Ni-Na system.

Tentative values of the Ni solubility in liquid Cs contaminated with 0.08 mol % O:

T/K	*soly*/mol % Ni	source
423	$4 \cdot 10^{-4}$	(3)
573	$3\ 10^{-4}$	(3)
1255	$>1 \cdot 10^{-4}$ ^{a}	(1)

^{a} at elevated pressure to keep Cs in the liquid state

References

1. Tepper, F.; Greer, J. *U.S. Air Force Rep. ASD-TDR-63-824*, Pt.I, 1963; *Rep. MSAR-63-61*, 1963.
2. Tepper, F.; Greer, J. *U.S. Air Force Rep. AFML-TR-66-280*, 1966; as cited by Berry, W.E. *Corrosion in Nuclear Applications*, Wiley, N.Y., 1971, p.300.
3. Godneva,M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.
4. 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.
5. Winslow, P.M. *Corrosion* 1965, *21*, 341.
6. Winslow, P.M. *U.S. Atom. Ener. Comm. Rep. CONF-650411*, 1965, p. 334.
7. Keddy, E.S. *U.S. Atom. Ener. Comm. Rep. LAMS-2948*, 1963.
8. Harvey, R. *U.S. Atom. Ener. Comm. Rep. MND-P-3009-1*, 1959.
9. Chandler, W.T. *U.S. Atom. Ener. Comm. Rep. TID-7626*, Pt.I, 1962, p. 42.
10. Smith, R.G.; Hargreaves, F.; Mayo, G.T.J.; Thomas, A.G. *J. Nucl.Mater.* 1963, *10*, 191.
11. Teitel, R.J. *NASA Rep. TN-D-769*, 1961, p. 49.
12. Sedelnikov, V.A.; Godneva, M.M. *Issledovanie Fiziko-Khimicheskikh Svoistv Soedinenii Redkikh Elementov*, Nauka, Leningrad, 1978, p. 56.

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

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

ORIGINAL MEASUREMENTS:

Tepper, F.; Greer, J.

US Air Force Rep. ASD-TDR-63-824, Pt.I, 1963; *Rep. MSAR-63-61*, 1963.

VARIABLES:

One temperature: 1255 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The equilibrium concentration of Ni in liquid Cs at 1800°F after an exposure of 10, 100 and 1000 hours was found to be $1.8\ 10^{-3}$, $< 6 \cdot 10^{-4}$ and $< 6\ 10^{-4}$ mass %, respectively, using Haynes-25 alloy as solute. The corresponding values are $4.1\ 10^{-4}$, $< 1.4\ 10^{-4}$ and $< 1.4\ 10^{-4}$ mol % Ni, respectively, as calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A test capsule was composed of two parts: the bottom made of Haynes-25 alloy and the upper made of alumina. The capsule was heated in vacuum for 100 hours at 1255 K. It was then filled with Cs and closed by means of welding in an Ar atmosphere at ambient temperature. The hermetically closed capsule was equilibrated at the desired temperature. After completion of this procedure the capsule was inverted and cooled in dry ice. The solidified Cs in the alumina part was dissolved in CH_3OH, the crucible was finally cleaned with HCl. The combined mixture of the solutions was boiled to dryness and submitted for quantitative emission spectrographic analysis.

SOURCE AND PURITY OF MATERIALS:

Haynes-25: 10 % Ni, 50 % Co, 20 % Cr, 15 % W, 3.7 % Fe, 2.7 % Mn, $5.1 \cdot 10^{-2}$ C, $2.01 \cdot 10^{-2}$ % O and $3.76 \cdot 10^{-2}$ % N.
Cs: 99.9+ % pure from Mine Safety Appliances Research and 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 $< 5\ 10^{-4}$ % Ni.
Ar: purified in a K-Na bubbler.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:

(1) Nickel; Ni; [7440-02-0]

(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: 423-573 K
O content in Cs: 0.08 and 0.8 mol %

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ni in liquid Cs was determined.

t/°C	*soly*/mass % Ni	*soly*/mol % Ni [a]
150	$1.7 \cdot 10^{-4}$	$3.8\ 10^{-4}$
200	$1.5 \cdot 10^{-4}$	$3.4 \cdot 10^{-4}$
300	$1.3 \cdot 10^{-4}$	$2.9 \cdot 10^{-4}$
300 [b]	$1.2 \cdot 10^{-3}$	$2.7 \cdot 10^{-3}$

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

A Ni-Cs mixed oxide was probably formed on the Ni surface, since the mass of the Ni specimen increased during the test.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ni specimen was covered by Cs under vacuum and equilibrated for 120 hours in a glass ampoule. No change of the glass was observed after the solubility test. Cs was cooled and dissolved in H_2O, and its amount was determined by titration of a portion of the primary solution with an acid. Another portion of the primary solution was treated with K-Na tartrate, $(NH_4)_2S_2O_8$ and dimethylglyoxime to complex Ni, which was colorimetrically determined after 10 min (1). The O concentration in Cs was increased by the controlled decomposition of $KClO_3$-MnO_2 mixture.

SOURCE AND PURITY OF MATERIALS:

Ni: "electrolytic", containing 0.76 % O and $4 \cdot 10^{-3}$ % Mg.
Cs: 98-99 % pure, further vacuum distilled, 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.

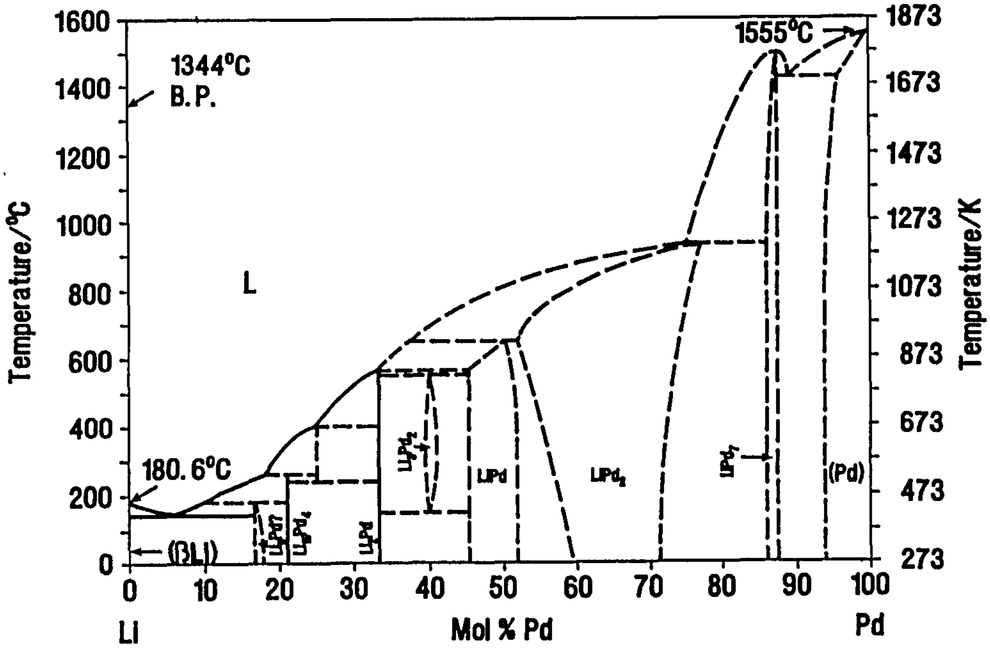
COMPONENTS:	EVALUATOR:
(1) Palladium; Pd; [7440-05-3] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1992

CRITICAL EVALUATION:

Bredzs and Schwartzbart (1) observed that an addition of 45 mol % Li to Pd caused a significant decrease of the melting point of Pd, but they did not report a value of the temperature depression.
This observation was confirmed by Loebich and Raub (2,3) who established several points on the liquidus line and sketched an incomplete phase diagram of the Pd-Li system. The diagram based on (3,5) is redrawn in the figure. Howald (4) calculated the phase diagram on the basis of thermodynamic considerations. These phase relations show some changes at the Pd-rich side in respect to the proposed data of (3). A slightly different Pd-rich part of the phase diagram was also presented by Sakamoto et al. (6). The changes did not affect the liquidus, which was not subject of the experimental study.

Tentative values of the solubility of Pd in liquid Li

T/K	*soly*/mol % Pd	source
418	6 eutectic	(2,3)
473	11	(2,3) interpolated
573	19	(2,3) interpolated
673	24	(2,3) interpolated
773	30	(2,3) interpolated
873	35	(2,3) interpolated



References

1. Bredzs, N.; Schwartzbart, H. *Welding J.* 1961, *40*, 123-s.
2. Loebich, O.; Raub, Ch.J. *J. Less-Common Met.* 1977, *55*, 67.
3. Loebich, O.; Raub, Ch.J. *Platin. Met. Rev.* 1981, *25*, 113.
4. Howald, R.A. *CALPHAD* 1990, *14*, 1.
5. Sangster, J.; Pelton, A.D. *J.Phase Equil.* 1992, *13*, 63.
6. Sakamoto, Y.; Chen, F.L.; Muto, J.; Flanagan, T.B. *Z. Phys. Chem.* 1991, *173*, 235.

COMPONENTS:

(1) Palladium; Pd; [7440-05-3]

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

ORIGINAL MEASUREMENTS:

Loebich, O.; Raub, Ch.J.

J. Less-Common Met. 1977, *55*, 67-76.

VARIABLES:

Temperature: 433-1773 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Experimental results of determinations of the liquidus line of the Pd-Li system were reported, values were read out from the figure by the compilers.

t/°C	*soly*/mol % Pd
160	7.5
245	16
370	21
455	27
540	32
567	34
665	38
~ 930	79
~ 1500	87.5

A smooth curve of the liquidus which was based on these results was reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in the analytical vessel for differential thermal analysis. Molten Li directly reacted with Pd powder enclosed in a Ta tube. The tube was then closed by flattening the ends which were twice folded and again compressed. The differential thermal analyses were performed in an Ar atmosphere. The Pd-Li alloy started to react with the Ta crucible at $\geq$ 670 °C. The results at higher temperatures were evaluated from supplementary metallographic studies.

SOURCE AND PURITY OF MATERIALS:

Pd: 99.9 % pure from Heraeus.
Li: 99 % pure from Merck with a content of 0.3 % Na.
Ar: "high purity".

ESTIMATED ERROR:

Solubility: accuracy ± 0.2 mol %; read-out procedure ± 1 mol %.
Temperature: read-out procedure ± 7 K.

REFERENCES:

1. Loebich, O.; Raub, Ch.J. *Platin. Met. Rev.* 1981, *25*, 113-120.

COMPONENTS:	EVALUATOR:
(1) Palladium; Pd; [7440-05-3] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1989

CRITICAL EVALUATION:

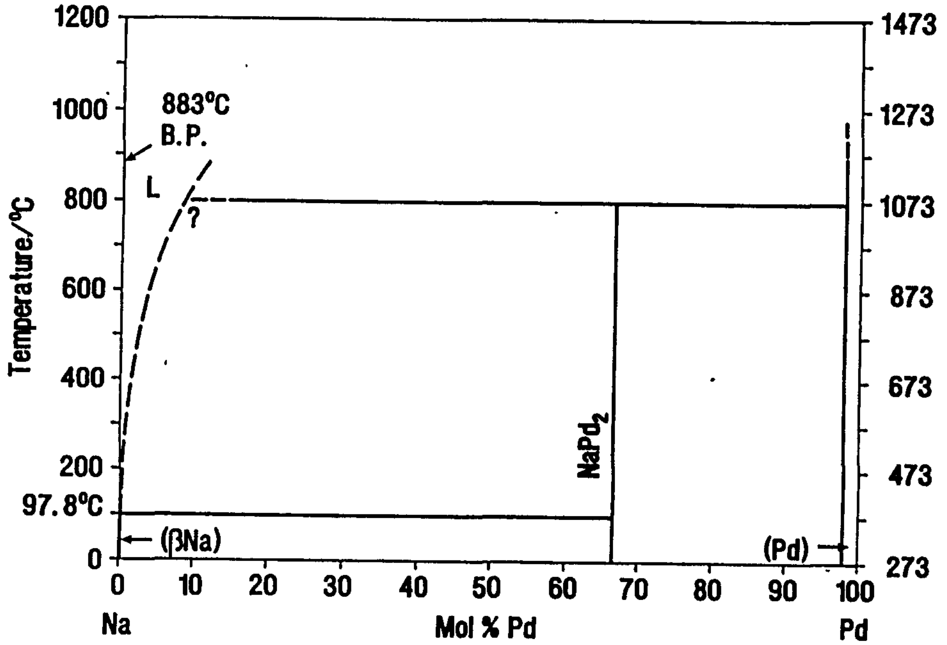
Tammann (1) found a decrease of the melting point of Na due to the addition of small amounts of Pd. A eutectic point was detected at 0.7 mol % Pd and a temperature 0.4 K below the melting point of Na. There is only scarce information on the purity of the two components and the experimental conditions. Thus, these results have to be considered as uncertain.

Aleksandrov and Dalakova (3) observed a dissolution of Pd in liquid Na at 973-1023 K after an equilibration of 1 h; they did not report the quantitative results of the spectral analysis. Loebich and Raub (2) performed some experiments on the alloying of Pd and Na. They found that the intermetallic compound in this system, Pd_2Na, decomposes peritectically at 1070±5 K.

A partial Pd-Na phase diagram was presented in (4), and is shown in the figure.

Doubtful value of the solubility of Pd in liquid Na

T/K	*soly*/mol % Pd	source
370.6	0.7 eutectic	(1)



References

1. Tammann, G. *Z. Phys. Chem.* 1889, *3*, 441.
2. Loebich, O.; Raub, Ch.J. *Platin. Met. Rev.* 1981, *25*, 113.
3. Aleksandrov, B.N.; Dalakova, N.V. *Izv.Akad.Nauk SSSR, Met.* 1982, no. 1, 133.
4. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Amer.Soc.Mater., Materials Park, 1990, p. 2724.

COMPONENTS:

(1) Palladium; Pd; [7440-05-3]

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

ORIGINAL MEASUREMENTS:

Tammann, G.

Z. Phys. Chem. 1889, *3*, 441-449.

VARIABLES:

Temperature difference: 0.4 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The depression (ΔT) of the melting point of Na due to the addition of Pd was determined.

g Pd/100g Na	*soly*/mol % Pd [a]	ΔT/K
0.56	0.121	0.07
0.97	0.209	0.11
1.54	0.332	0.26
2.22	0.477	0.31
2.91	0.625	0.40
3.34	0.717	0.40

[a] as calculated by the compilers

The melting point of Na was reported to be 370 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Portions of Pd were successively added to molten Na. The solutions were undercooled by up to 2 K. The crystal precipitation was forced by means of effective motion of the thermometer. Every determination of the temperature using the thermometer was repeated three times.

SOURCE AND PURITY OF MATERIALS:

Pd: nothing specified.
Na: "pure".

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.05 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Palladium; Pd; [7440-05-3] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1989

CRITICAL EVALUATION:

Pd does not react with liquid K and the solubility of Pd in the alkali metal seems to be very low, according to the report of Loebich and Raub (1). K was nearly totally lost by evaporation out of the reaction vessel in a test at 1073 K. This may indicate a small solid solubility of K in Pd; the formation of intermetallics is unlikely in the system. A contradictory observation was reported by Aleksandrov and Dalakova (2) who detected a significant dissolution of Pd in liquid K at 873-923 K; quantitative solubility data were not presented.
The scarce data do not allow proposal of a Pd-K phase diagram.

References

1. Loebich, O.; Raub, Ch.J. *Platin. Met. Rev.* 1981, *25*, 113.
2. Aleksandrov, B.N.; Dalakova, N.V. *Izv.Akad.Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Palladium; Pd; [7440-05-3] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1989

CRITICAL EVALUATION:

The behaviour of Pd in contact with liquid Rb is similar to that in K, as can be seen from the publication of Loebich and Raub (1). The solubility of Pd in liquid Rb as well as the mutual solid solubilities should be very low, intermetallics do not exist. A Pd-Rb phase diagram cannot be proposed due to the lack of data.

References

1. Loebich, O.; Raub, Ch.J. *Platin. Met. Rev.* 1981, *25*, 113.

COMPONENTS:	EVALUATOR:
(1) Palladium; Pd; [7440-05-3] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1989

CRITICAL EVALUATION:

The solubility of Pd in liquid Cs should be very low, and intermetallics should not exist in the system as in the systems of Pd with K and Rb.
Keddy (1) investigated the compatibility of Pd in liquid Cs at 773 K for 100 h and observed a weight loss of the Pd sample. Solubility values could not be estimated, since the mass of the solvent was not reported.
An anonymously reported observation (2) is somewhat in contrast to the results of (1). Cs vapour did not attack a Pd sample in a test of 1000 h duration at 1273 K.
There is no convincing data which allow the construction of a schematic Pd-Cs phase diagram.

References

1. Keddy, E.S. *US Atom.Ener.Comm.Rep. LAMS-2948*, 1963.
2. Anonymous, *US Atom.Ener.Comm.Rep. GEST-2035*, vol. 1, 1964; as cited by Berry, W.E. *Corrosion in Nuclear Applications*, Wiley, N.Y., 1971, p. 304.

COMPONENTS:	EVALUATOR:
(1) Platinum; Pt; [7440-06-4] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1992

CRITICAL EVALUATION:

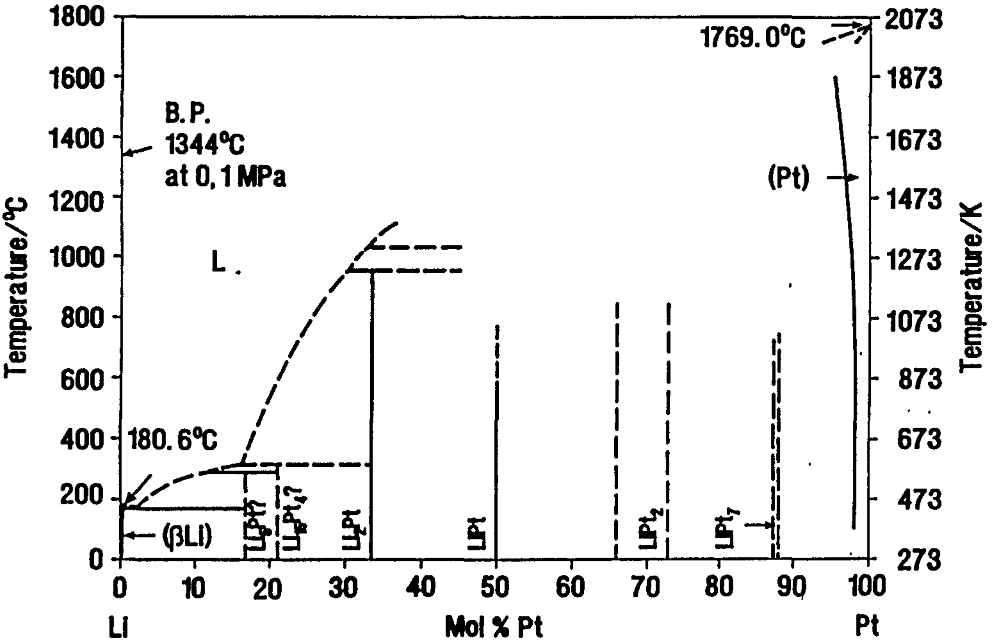
Grosse (1) and Nash et al. (2) observed that a spontaneous reaction between Pt and Li occurred at a temperature above the melting point of Li. The corrosion resistance of Pt in contact with liquid Li was found to be poor (3), this fact suggested a relatively large solubility.

Solubility measurements were performed by Loebich and Raub (4,5), who determined the liquidus of the Li rich part of the Pt - Li phase diagram. An extension of the determinations to higher Pt contents could not be made due to the interaction of the Pt-Li melt with the Ta container at temperatures above 1473 K.

The saturated solutions of Pt in liquid Li are in equilibrium with Pt-Li intermetallic compounds (2,4,5), as is shown in the partial phase diagram redrawn after (5,6).

Tentative values of the solubility of Pt in liquid Li

T/K	*soly*/mol % Pt	source
442	2 eutectic	(4) interpolated
473	3	(4) interpolated
573	13	(4) interpolated
1073	26	(4)
1347	35	(4) interpolated



References

1. Grosse, A. *Z. Naturforsch.,B* 1953, *8*, 535.
2. Nash, C.P.; Boyden, F.M.; Whittig, L.D. *J. Am. Chem. Soc.* 1960, *82*, 6203.
3. Hoffman, E.E. *US Atom.Ener.Comm. Rep. ORNL-2924*, 1960.
4. Loebich, O.; Raub, Ch.J. *J. Less-Common Met.* 1980, *70*, P47.
5. Loebich, O.; Raub, Ch.J. *Platin. Met. Rev.* 1981, *25*, 113.
6. Sangster, J.; Pelton, A.D. *J.Phase Equil.* 1991, *12*, 678.

COMPONENTS:

(1) Platinum; Pt; [7440-06-4]

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

ORIGINAL MEASUREMENTS:

Loebich, O.; Raub, Ch. J.

J. Less-Common Met. 1980, *70*, P47-P55.

VARIABLES:

Temperature: 442-1347 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Six points of the liquidus curve of the Pt-Li system were determined; the values were read out from the figure by the compilers.

t/°C	*soly*/mol % Pt
169	~2 eutectic [a]
256	5.5
295	11.5
315	16
801	26
820	27
1074	35

[a] extrapolated value

The smooth liquidus curve which was based on these measurements was reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by reaction of Pt powder and Li pieces in a closed Ta tube. After the tube was filled, it was closed by flatting the ends, which were twice folded and compressed for closure. The differential thermal analysis of the alloys was performed in the temperature range 323 - 1823 K in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Pt: nothing specified.

Li: (probably) 99 % pure from Merck with 0.3 % Na.

Ar: (probably) "high purity".

ESTIMATED ERROR:

Solubility: reading-out ± 1 mol %.

Temperature: precision ± 1 K; reading out ± 5 K.

REFERENCES:

1. Loebich, O.; Raub, Ch.J. *Platin.Met.Rev.* 1981, *25*, 113-120.

COMPONENTS:	EVALUATOR:
(1) Platinum; Pt; [7440-06-4] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1992

CRITICAL EVALUATION:

Pt reacts with Na at elevated temperature forming the intermetallic compounds NaPt and $NaPt_2$ which melt above 1173 K (1,2,3). Pt showed insignificant solubility in Na at 523 K (4) as well as at 623 K. An attack of liquid Na on Pt started at >723 K, and a remarkably increased solubility was observed at >773 K (5). Mathewson (8) took advantage of the distinctly lower solubility of Pt than Au in Na and tried to separate these metals from Au-Pt alloys. Quantitative data were not reported in any of these works.

Tammann (6) determined the depression of the melting point of Na due to the addition of 0.0078 to 0.18 mol % Pt to be 0.01 K. This result was below the precision of the method. Nevertheless, it suggested that the eutectic point should be very close to the temperature of the melting point of Na (~370 K).

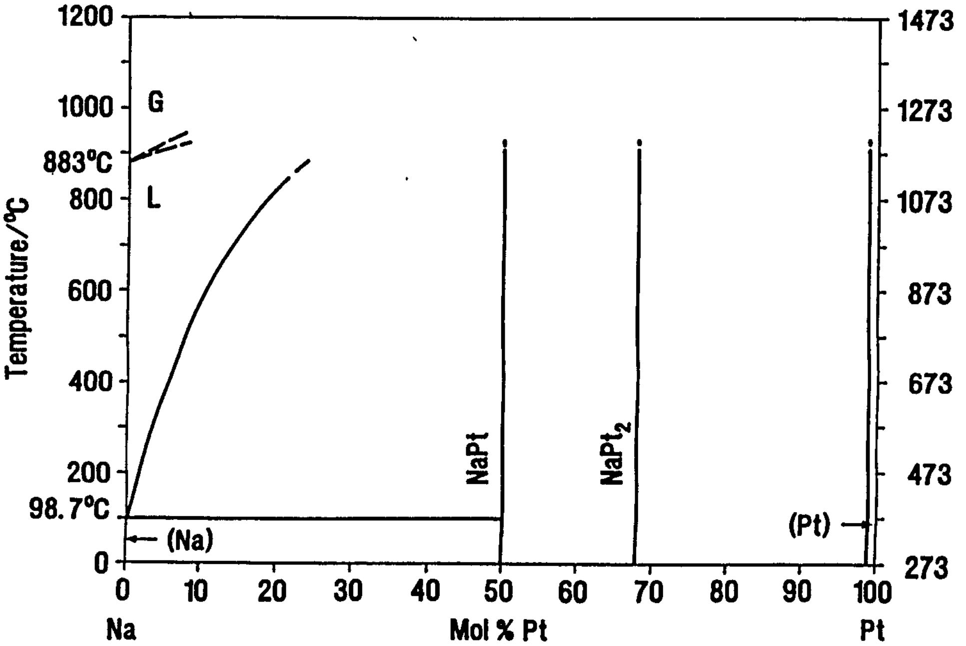
Liquidus points of 4 mixtures of Pt and Na were determined by means of differential thermal analysis by Borgstedt and Bhat (2). The results can be expressed by the equation:

$$\log(soly/\text{mol \% Pt}) = 2.35 - 845(T/\text{K})^{-1}$$

valid between 528 and 831 K. The compound which was identified as $NaPt_2$ by means of X-ray examinations (7) is the equilibrium solid phase in the temperature range of this study. Saturated solutions of Pt in liquid Na are generally in equilibrium with the Pt-Na intermetallic compounds (1,2,5). This is reflected in the Na-Pt phase diagram shown in the figure.

Tentative values of the solubility of Pt in liquid Li

T/K	*Soly*/mol % Pt	source
528	5.0	(7)
610	11.0	(7)
739	15.3	(7)
831	20.5	(7)



References

1. Loebich, O.; Raub, Ch.J. *Platin. Met. Rev.* 1981, *25*, 113.
2. Nash, C.P.; Boyden, F.M.; Whittig, L.D. *J. Am. Chem. Soc.* 1960, *82*, 6203.
3. Haber, F.; Sack, M. *Z. Elektrochem.* 1902, *8*, 245.
4. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1889, *55*, 666.
5. Hackspill, L. *7th Intern. Congress of Applied Chemistry*, London, 1909, *Sect. II*, p. 266.
6. Tammann, G. *Z. Phys. Chem.* 1889, *3*, 441.
7. Borgstedt, H.U.; Bhat, N.P. *J. Less-Common Met.* 1990, *161*, L1.
8. Mathewson, C.H. *Intern. Z. Metallogr.* 1911, *1*, 81.
9. Aleksandrov, B.N.; Dalakova, N.V. *Izv.Akad.Nauk SSSR,Met.* 1982, no.1, 133.

COMPONENTS:

(1) Platinum; Pt; [7440-06-4]

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

ORIGINAL MEASUREMENTS:

Tammann, G.

Z. Phys. Chem. 1889, *3*, 441-449.

VARIABLES:

Temperature: around the melting temperature of Na

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The melting point of Na showed a depression of 0.01 K after the addition of 0.0078 to 0.18 mol % Pt to pure Na. The melting point of Na was reported to be 370 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Succesive portions of Pt were added to molten Na. The obtained solutions were undercooled by up to 2 K, and crystal precipitation was forced by means of effective stirring with the thermometer. Every determination of the temperature was three times repeated.

SOURCE AND PURITY OF MATERIALS:

Pt: nothing specified.
Na: "pure".

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.05 K.

REFERENCES:

COMPONENTS:

(1) Platinum; Pt; [7440-06-4]

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

ORIGINAL MEASUREMENTS:

Borgstedt, H.U.; Bhat, N.P.

J. Less-Common Met. 1990, *161*, L1-L4.

VARIABLES:

Temperature: 528-831 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Pt in liquid Na was determined at four temperatures:

T/K	*soly*/mol % Pt
528	4.99
610	10.94
739	15.35
831	20.49

The solubility equation was given by the authors:

$$\log(soly/\text{mol \% Pt}) = 2.35 - 845(T/\text{K})^{-1}$$

The compound $NaPt_2$ was identified by means of X-ray examinations.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Pt and Na were exactly weighed and enclosed in small capsules of stainless steel which were closed inside an Ar atmosphere and finally welded. The total weight of the samples was between 0.1 and 0.2 g.

The capsules were first equilibrated one hour at 973 K and then used to measure the liquidus points in a DTA apparatus.

SOURCE AND PURITY OF MATERIALS:

Pt: high purity powder from Goodfellow.
Na: reactor grade, purified by means of remelting under Ar atmosphere.
Ar: 99.999 % pure, additionally purified in the gas circuit of the box, partial pressure of H_2O+O_2 $<10^{-1}$ Pa.

ESTIMATED ERROR:

Solubility: exactness of weighing 10^{-4} g
Temperature: precision ± 1 K.

REFERENCES:

COMPONENTS:

(1) Platinum; Pt; [7440-06-4]

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

EVALUATOR:

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

CRITICAL EVALUATION:

Systematic investigations of the Pt - K system have not been performed so far. Hackspill (1) observed that liquid potassium did not attack Pt below a temperature of about 673 K. The dissolution of Pt was apparently increased above 773 K. Pt seems to be more resistant to dissolution in liquid K according to more recent sources. Pt and K were heated to 1073 K, and K was nearly quantitatively evaporated (3). This indicated a small solid solubility of K in Pt and no tendency to form stable intermetallics. Aleksandrov and Dalakova (4) observed a slight dissolution of Pt in liquid K at 873-923 K after 1 h of equilibration. They did not specify the purity of the metals and the detection limit of the spectral analytical method.
The Pt-K phase diagram is still unknown and cannot be sketched without further experiments.

References

1. Hackspill, L. *7th Intern. Congress of Applied Chemistry*, London, 1909, *Sect. II*, p. 266.
2. Wilhelm, C. *Mater. Design Engin.* 1963, *58*, 97.
3. Loebich, O.; Raub, Ch.J. *Platin. Met. Rev.*, 1981, *25*, 113.
4. Aleksandrov, B.N.; Dalakova, N.V. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:

(1) Platinum; Pt; [7440-06-4]

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

EVALUATOR:

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

CRITICAL EVALUATION:

The Pt - Rb system may be considered as analogous to the Pt - K system. Only a very small solubility of Pt in Rb at temperatures below 773 K and no formation of Pt - Rb intermetallics are, therefore, expected (1).
A Pt-Rb phase diagram cannot be predicted due to the lack of reliable data.

References

1. Loebich, O.; Raub, Ch.J. *Platin. Met. Rev.* 1981, *25*, 113.

COMPONENTS:

(1) Platinum; Pt; [7440-06-4]

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

EVALUATOR:

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

CRITICAL EVALUATION:

Solubility determinations of Pt in liquid Cs were not reported. Compatibility tests of Pt in the liquid metal were, however, performed in several laboratories (1-7).
Dissolution of Pt in liquid Cs was observed in the temperature range 673-1173 K (1-3,5). Pt was selectively leached out of the Pt-Ir(10%) alloy (5-7). A weight loss of 16 % of the Pt content was found in the test in liquid Cs at 1063 K (5). Since the initial weights of sample and solvent were not reported, the solubility could not be estimated from these results. Cs vapour did not attack Pt during 1000 h exposure at 1273 K (4). Solid intermetallics are not expected in the Pt-Cs system. A phase diagram cannot yet be constructed.

References

1. Petrick, E.N.; Husmann, O.K.; Szymanowski, H.W. *US Atom.Ener.Comm.Rep. CWR-700-10*, 1960.
2. Keddy, E.S. *US Atom.Ener.Comm.Rep. LAMS-2948*, 1963.
3. Slivka, M.J. *Adv. Ener. Conver.* 1963, *3*, 157.
4. Anonymous, *US Atom.Ener.Comm.Rep. GEST-2035*, vol. 1, 1964; as cited by Berry, W.E. *Corrosion in Nuclear Applications*, Wiley, N.Y., 1971, p. 304.
5. Winslow, P.M. *US Atom.Ener.Comm.Rep. CONF-650411*, 1965, p. 334.
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, N.Y., 1963, p. 341.

COMPONENTS:	EVALUATOR:
(1) Copper; Cu; [7440-50-8] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1990

C RITICAL EVALUATION:

Pastorello (1) determined several points on the liquidus line of the Cu-Li system in the composition range 5.99-98.29 mol % Cu. Kienast et al. (2) suggested the existence of a miscibility gap in the liquid state of the system on the basis of other thermal analyses. Quantitative data were, however, not published. Hoffman (6) observed a significant dissolution of Cu in liquid Li at 1089 K, but did not report quantitative results.

Pelton critically evaluated the results of (1) by means of thermodynamic modelling. He questioned the position of the point on the liquidus at 98.26 mol % Cu and 1316 K. He also calculated a metastable miscibility gap which should be placed below the experimental liquidus line of (1). Pelton's calculation of a sequence of saturation concentrations (3) (0.01,0.04, and 0.3 mol % Cu at 453.8, 523, and 673 K, respectively) suggests that the solubility results of (1) should be lower, and their temperature dependence should be steeper.

There are further reservations of (4) with respect to the Cu-Li phase diagram boundaries which were sketched by (3), since the solid solubility of Li in Cu determined by (8) was found to be significantly lower (4). The intermetallic Cu_4Li is most probably formed at the temperature of 473-873 K (5,7). A slight exothermic effect of mixing the liquid metals was observed even at 1373 K (9). This fact indicates an interaction between the metals in the Cu-rich range.

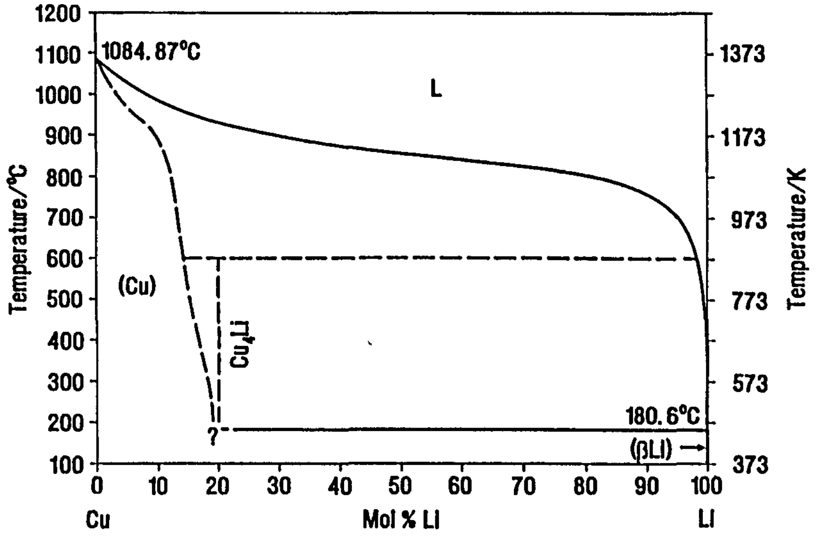
The influence of N on the Cu-Li equilibria was not studied in details; however, there may exist a strong interaction in the Cu-N-Li system according to Gryaznov et al. (10). Thus N may influence the solubility of Cu in liquid Li. (10) observed that an addition of 0.11 mol % Cu to Li saturated with 0.10 mol % Mo in the presence of 0.5 mol % N at 1273 K caused a decrease of the solubility of Mo to $1.1 \cdot 10^{-3}$ mol % Mo. This phenomenon may be explained by stronger interactions in the Cu-N-Li than in the Mo-N-Li systems.

Since no high purity materials were used in (1), and several other features of the Cu-Li phase diagram are still to be clarified, we may only suggest a doubtful value of the solubility.

A schematic phase diagram of the Cu-Li system based on (3) and (7) is shown in the figure.

Doubtful value of the solubility of Cu in liquid Li

T/K	$soly$/mol % Cu	source
1000	6	(1)



References

1. Pastorello, S. *Gazz. Chim. Ital.* 1930, *60*, 988.
2. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.
3. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, *7*, 142.
4. Mendelsohn, M.H.; Gruen,D.M.; Krauss, A.R. *J. Nucl. Mater.* 1986, *141-143*, 184.
5. Krauss, A.R.; Mendelsohn, M.H.; Gruen,D.M.; Conn, R.W.; Goebel, D.M.; Hiroaka, Y.; Leung, W.K.; Bohdanowsky, J. *US Dep.Ener.Rep. CONF-86-0807-3*, 1986.
6. Hoffman, E.E. *US Atom.Ener.Comm.Rep. ORNL-2924*, 1960.
7. Old, C.F.; Travena, P. *Met. Sci.* 1981, *15*, 281.
8. Klemm, W.; Volavsek, B. *Z.Anorg.Chem.* 1958, *296*, 184.
9. Mikhailovskaya, M.V.; Sudavtsova, V.S. *Ukr.Khim.Zh.* 1989, *55*, 1106.
10. Gryaznov, G.M.; Evtikhin, V.A.; Zavyalskii, L.P.; Kosukhin, A.Ya.; Lyublinskii, I.E. *Materialovedenie Zhidkometallicheskikh Sistem Termoyadernykh Reaktorov*, Energoatomizdat, Moskva, 1989, p. 229.

COMPONENTS:

(1) Copper; Cu; [7440-50-8]

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

ORIGINAL MEASUREMENTS:

Pastorello, S.

Gazz. Chim. Ital. 1930, *60*, 988-992.

VARIABLES:

Temperature: 1003-1316 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points of the liquidus curve of the Cu - Li system were determined.

t/°C	*soly*/mol % Cu
1039	98.29
980	90.10
963	85.62
875	50.25
800	23.15
770	11.54
730	5.99

The melting points of Cu and Li were found to be 1084 and 180 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by melting both metals in a stainless steel capsule in an Ar atmosphere. The composition of the alloys was confirmed by chemical analyses. The cooling curves were recorded using Pt/Pt-Rh thermocouples. The alloys were previously heated for some time above the liquidus temperature. The thermocouples were calibrated at the melting point of Cu. A dissolution of less than 0.1 % Fe from the capsule was found to have a negligible effect.

SOURCE AND PURITY OF MATERIALS:

Cu: electrolytic from Merck.
Li: "pure" from Kahlbaum.
Ar: purified by getter reaction with Mg and Ca at 773 K.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Copper; Cu; [7440-50-8] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

CRITICAL EVALUATION:

Mathewson (1) reported that the dissolution of Cu in liquid Na close to its melting point was not large enough to detect dissolved Cu by analytical methods. A similar observation was later stated by Gilbert (2).Aleksandrov and Dalakova (16) did not observe any dissolution of Cu in liquid Na even at 973 to 1023 K. The addition of 0.04 to 0.22 mol % Cu to Na caused only a depression of the melting point of 0.01 K, as reported by Tammann (3). The eutectic in this system should, therefore, be placed near the melting point of Na.

The solubility of Cu in liquid Na was studied in five laboratories (4-12). All authors reported a large scatter of values and an overstatement of the solubility at temperatures below 600 K (see for example the figure presented in (13)). These facts can be understood on the basis of the observation that the cooling of saturated solutions of Cu in Na for subsequent equilibration did not cause complete phase separation (10,11). Therefore, the remaining tests were performed to reach equilibrium from lower temperatures. Since no peritectic temperature is to be expected at about 600 K, and the O content of Na does not obviously influence the solubility of Cu, this phenomenon remains unexplained. Taking into account all consistent results (4-12) above 600 K the solubility equation may be formulated:

$$\log(soly/\text{mol \% Cu}) = 1.029 - 3070(T/\text{K})^{-1} \quad r=0.986 \qquad \text{Eq.(1)}$$

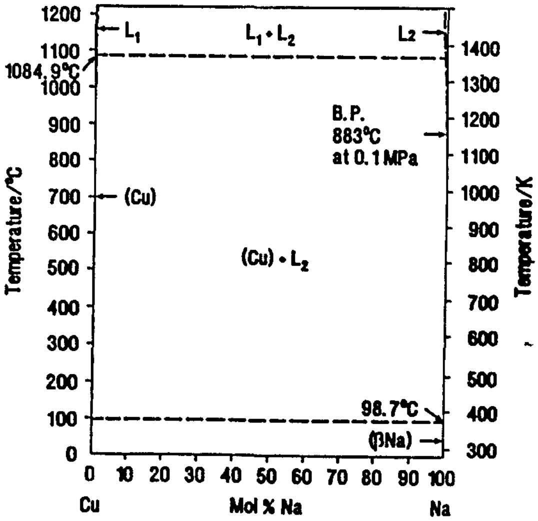
Several equations describing the temperature dependence of the solubility based on different selections of data were presented in previous papers (6,7,10,12-14). Those reported in (7,13,14) were close to the equation given here. Eichelberger and McKisson (6) postulated a break point in the solubility relation at about 773 K; in their later communications (7-9) they abandoned this interpretation of the results.

Kienast et al. (15) performed thermal analyses of the Cu-Na system and characterized it as composed of essentially immiscible components in the liquid and solid states without any intermetallic compounds. Quantitative results were, however, not reported.

A schematic phase diagram of the Cu-Na system at a pressure sufficient to keep Na in the liquid state is shown in the figure.

Recommended values of the solubility of Cu in liquid Na

T/K	$soly$/mol % Cu	source
573	$5\cdot10^{-5}$	(10), Eq.(1)
673	$3\cdot10^{-4}$	(4,7,10,12), Eq.(1)
773	$1.1\ 10^{-3}$	(5,7,10), Eq.(1)
873	$3\cdot10^{-3}$	(5,7,12), Eq.(1)
973	$7\cdot10^{-3}$	Eq.(1)



References

1. Mathewson, C.H. *Int. Z. Metallogr.* 1911, *1*, 51.
2. Gilbert, H.N. *Chem. Eng. News* 1948, *26*, 2604.
3. Tammann, G. *Z. Anorg. Chem.* 1889, *3*, 441.
4. Koenig, R.F. *US Atom.Ener.Comm. Rep. KAPL-M-RFK -12*, 1953.
5. Humphreys, J.R. *Los Alamos Sci. Lab. Rep. K-3-774*, 1958; as reported in (13).
6. Eichelberger, R.L.; McKisson, R.L. *Trans. Am. Nucl. Soc.* 1967, *10*, 495.
7. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. AI-AEC-12671*, 1968.
8. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt. I, 1968, p. 319.
9. McKisson, R.L.; Eichelberger, R.L.; Gehri, D.C.; Guon, J. *US Atom.Ener.Comm.Rep. AI-AEC-12721*, 1968, p. 279.
10. Singer, R.M.; Weeks, J.R. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt.I 1968, p.309.
11. 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.
12. Walker, R.A.; Pratt, J.N. *J. Nucl. Mater.*, 1969, *32*, 340.
13. Claar, T.D. *Reactor Technol.* 1970, *13*, 124.
14. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, *7*, 231.
15. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.
16. Aleksandrov, B.N.; Dalakova, N.V. *Izv.Akad.Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS: (1) Copper; Cu; [7440-50-8] (2) Sodium; Na; [7440-23-5]	**ORIGINAL MEASUREMENTS:** Koenig, R.F. *US Atom.Ener.Comm. Rep. KAPL-M-RFK-12*, 1953.
VARIABLES: Temperature: 393-773 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cu in liquid Na was measured in the temperature range 393 to 773 K.

t/°C	*soly*/mass % Cu	*soly*/mol % Cu [a]
120	$5.0\ 10^{-4}$	$1.8\ 10^{-4}$
200	$9.2\ 10^{-4}$	$3.3\ 10^{-4}$
300	$7.3\ 10^{-4}$	$2.6\ 10^{-4}$
400	$7.2\ 10^{-4}$	$2.6\ 10^{-4}$
500	$1.5\ 10^{-3}$	$5.4\ 10^{-4}$

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small sample of Na was equilibrated with a thick-walled Cu crucible, according to (1). The temperature control was performed using a thermostated Na bath in a stainless steel pot. Na inside the crucible was separately filled and did never contact the stainless steel surface. The Na was sampled through a port under flowing Ar after the equilibration, and analyzed for the Cu content by a wet chemical method, probably colorimetry.

SOURCE AND PURITY OF MATERIALS:

Cu: nothing specified.
Na: nothing specified.
Ar: "purified".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Claar, T.D. *Reactor Technol.* 1970, *13*, 124-146.

COMPONENTS:

(1) Copper; Cu; [7440-50-8]

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

ORIGINAL MEASUREMENTS:

Humphreys, J.R.

Los Alamos Sci. Lab. Rep. K-3-774, 1958.

VARIABLES:

Temperature: 773-973 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility values of Cu in liquid Na were taken from (1) since the original report was not available.

t/°C	*soly*/mass % Cu	*soly*/mol % Cu [a]
500	$2.5\ 10^{-3}$	$9.0\ 10^{-4}$
500	$(1.7\text{-}2.3)\ 10^{-3}$	$(6.1\text{-}8.3)\cdot 10^{-4}$
600	$(6.2\text{-}7.5)\ 10^{-3}$	$(2.2\text{-}2.7)\ 10^{-3}$
600	$(7.0\text{-}8.3)\cdot 10^{-3}$	$(2.5\text{-}3.0)\ 10^{-3}$
600	$(1.12\text{-}1.41)\ 10^{-2}$	$(4.0\text{-}5.1)\ 10^{-3}$
700	$(3.44\text{-}4.43)\cdot 10^{-2}$	$(1.24\text{-}1.60)\cdot 10^{-2}$

[a] as calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was equilibrated with neutron-activated Cu foils in a stainless steel capsule for 17 to 35 hours, according to (1). Na was analyzed for its Cu content by means of a counting technique.

SOURCE AND PURITY OF MATERIALS:

Cu: nothing specified.
Na: hot gettered with Zr foil.

ESTIMATED ERROR:

Solubility: precision better than ± 12 %.
Temperature: nothing specified.

REFERENCES:

1. 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) Copper; Cu; [7440-50-8]

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

ORIGINAL MEASUREMENTS:

Eichelberger, R.L.; McKisson, R.L.

US Atom. Ener. Comm. Rep. AI-AEC-12671, <u>1968</u>.

VARIABLES:

Temperature: 523-999 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cu in liquid Na was determined.

t/°C	*soly*/mass % Cu	*soly*/mol % Cu [b]	t/°C	*soly*/mass % Cu	*soly*/mol % Cu [b]
250	$1.3\cdot10^{-4}$	$4.7\cdot10^{-5}$	371[a]	$7.5\cdot10^{-4}$	$2.7\cdot10^{-4}$
250	$3.1\ 10^{-4}$	$1.1\cdot10^{-4}$	373[a]	$7.6\cdot10^{-4}$	$2.7\cdot10^{-4}$
265	$7.0\cdot10^{-4}$	$2.5\cdot10^{-4}$	395[a]	$1.63\cdot10^{-3}$	$5.9\cdot10^{-4}$
279	$2.56\cdot10^{-3}$	$9.3\cdot10^{-4}$	399[a]	$1.08\cdot10^{-3}$	$3.9\cdot10^{-4}$
294	$1.23\cdot10^{-3}$	$4.4\cdot10^{-4}$	500[a]	$2.28\cdot10^{-3}$	$8.2\cdot10^{-4}$
300	$8.1\cdot10^{-4}$	$2.9\cdot10^{-4}$	500[a]	$2.94\cdot10^{-3}$	$1.1\cdot10^{-3}$
302	$3.0\cdot10^{-4}$	$1.1\cdot10^{-4}$	500[a]	$2.48\cdot10^{-3}$	$9.0\cdot10^{-4}$
306	$2.7\ 10^{-3}$	$9.7\cdot10^{-4}$	502[a]	$2.65\cdot10^{-3}$	$9.6\cdot10^{-4}$
350[a]	$3.4\cdot10^{-4}$	$1.2\cdot10^{-4}$	643[a]	$2.44\cdot10^{-2}$	$8.8\cdot10^{-3}$
350[a]	$4.2\cdot10^{-4}$	$1.5\ 10^{-4}$	648[a]	$2.08\cdot10^{-2}$	$7.5\cdot10^{-3}$
350[a]	$2.7\cdot10^{-4}$	$9.8\ 10^{-5}$	650[a]	$1.49\cdot10^{-2}$	$5.4\cdot10^{-3}$
351[a]	$4.5\ 10^{-4}$	$1.6\cdot10^{-4}$	660[a]	$3.30\cdot10^{-2}$	$1.2\cdot10^{-2}$
352[a]	$4.3\ 10^{-4}$	$1.5\cdot10^{-4}$	672[a]	$3.01\cdot10^{-2}$	$1.1\cdot10^{-2}$
368[a]	$5.5\ 10^{-4}$	$2.0\cdot10^{-4}$	726[a]	$6.17\ 10^{-2}$	$2.2\cdot10^{-2}$
371[a]	$9.3\cdot10^{-4}$	$3.3\ 10^{-4}$			

[a] also reported in (1,2)   [b] calculated by the compilers

21 data from (1,2), 6 data from Humphreys' report and 12 data from the report of Singer and Weeks in the temperature range 245-547 °C were expressed by a fitting equation:

$$\log(soly/\text{mol \% Cu}) = 1.572 - 3430\ (T/\text{K})^{-1} \quad \text{(tested by the compilers)}$$

These data were expressed with two solubility equations in an earlier report (3):

$$\le 773\ \text{K} \quad \log(soly/\text{mol \% Cu}) = -0.571 - 1930\ (T/\text{K})^{-1}$$

$$\ge 773\ \text{K} \quad \log(soly/\text{mol \% Cu}) = 2.148 - 4040\ (T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The equilibration was either performed with a Cu crucible or a Cu rod in a 304 steel crucible. The crucibles were electron beam welded to a sample collector made of Nb-Zr(1%) alloy. The crucibles were loaded with Na under high vacuum, and the crucible-collector assembly sealed into a stainless steel capsule which was heated for 4 to 21 hours though it was claimed that 6 hours were sufficient to attain equilibrium. The capsule was then inverted to pour the Na with dissolved Cu into the collector.

After cooling and solidification of the Na, the collector was opened by cutting. The Na was melted out of the collector, dissolved in C_2H_5OH, and the collector was etched with 1:1 HNO_3.

Cu was determined in the combined solutions either by colorimetry using neocuproine or by atomic absorption spectrometry. The weight of Na was received from the weight difference of the assembly before opening and after removal of Na.

SOURCE AND PURITY OF MATERIALS:

Cu: "oxygen free high conductivity".
Na: purified by hot gettering and distillation with a content of $8\cdot10^{-4}$ % C, $6\cdot10^{-5}$ % O, < $5\cdot10^{-4}$ % Cu; other elements were not detected.
Nb-Zr(1%) collector: from Wah Chang Corp., contained < $1.0\cdot10^{-3}$ % Cu.

ESTIMATED ERROR:

Solubility: precision ± 15 %.
Temperature: nothing specified.

REFERENCES:

1. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener. Comm. Rep. AI-AEC-12721*, <u>1968</u>, p. 279-308.
2. Eichelberger, R.L.; McKisson, R.L. *US Atom.Ener. Comm. Rep. ANL-7520*, Pt. I, <u>1968</u>, p. 319-327.
3. Eichelberger, R.L.; McKisson, R.L. *Trans. Am. Nucl. Soc.* <u>1967</u>, *10*, 495-496.

COMPONENTS:

(1) Copper; Cu; [7440-50-8]

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

ORIGINAL MEASUREMENTS:

Singer, R.M.; Weeks, J.R.

US Atom. Ener. Comm. Rep. ANL-7520, Pt.I 1968, p.309-318.

VARIABLES:

Temperature: 404-820 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cu in liquid Na was determined in 2 runs; the results were read out from the figure and recalculated to mol % by the compilers.

Run	T/K	*soly*/mass % Cu	*soly*/mol % Cu
I	404	$>1.0 \cdot 10^{-2}$, $1.2 \cdot 10^{-4}$	$>3.6 \cdot 10^{-3}$, $4.3 \cdot 10^{-5}$
	464	$2.4 \cdot 10^{-4}$,$>1.0 \cdot 10^{-2}$	$8.7 \cdot 10^{-5}$,$>3.6 \cdot 10^{-3}$
	518	$7.5 \cdot 10^{-4}$	$2.7 \cdot 10^{-4}$
	558	$9.0 \cdot 10^{-4}$, $1.2 \cdot 10^{-3}$	$3.3 \cdot 10^{-4}$, $4.3 \cdot 10^{-4}$
	617	$4.8\ 10^{-4}$, $6.0 \cdot 10^{-4}$	$1.7 \cdot 10^{-4}$, $2.2 \cdot 10^{-4}$
	676	$1.0 \cdot 10^{-3}$	$3.6 \cdot 10^{-4}$
	757	$2.8 \cdot 10^{-3}$, $4.3 \cdot 10^{-3}$	$1.01 \cdot 10^{-3}$, $1.56 \cdot 10^{-3}$
II	518	$<2 \cdot 10^{-5}$	$<7.2 \cdot 10^{-6}$
	599	$7.9 \cdot 10^{-5}$, $2.2 \cdot 10^{-4}$	$2.3 \cdot 10^{-5}$, $8.3 \cdot 10^{-5}$
	643	$2.1 \cdot 10^{-4}$, $3.0 \cdot 10^{-4}$	$8.0 \cdot 10^{-5}$, $1.1 \cdot 10^{-4}$
	677	$5.0 \cdot 10^{-4}$, $6.5 \cdot 10^{-4}$	$1.8 \cdot 10^{-4}$, $2.3 \cdot 10^{-4}$
	698	$4.2 \cdot 10^{-4}$, $6.5 \cdot 10^{-4}$	$1.52\ 10^{-4}$, $2.3\ 10^{-4}$
	746	$9.4 \cdot 10^{-4}$, $1.4 \cdot 10^{-3}$	$3.5 \cdot 10^{-4}$, $5.1 \cdot 10^{-4}$
	820	$6.0 \cdot 10^{-3}$	$2.2 \cdot 10^{-3}$

The results of run I are obviously overestimated; the results of run II are approximated by the equation (as tested by the compilers):

$$\log(soly/\text{mol \% Cu}) = 1.88 - 3850\ (T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Cu sheet in a Cu crucible was cleaned with $(NH_4)_2S_2O_8$, washed and H_2 fired at 773 K for 1 hour. The crucible and a Mo sampling cup were sealed into the solubility apparatus and outgassed at 873 K for 1 hour. Na was filled in from a tank, the system was pressurized and the apparatus was heated to 773 K. After an equilibration period of 24 hours, 2 parallel samples were taken into the Mo cups for analyses. A lower equilibration temperature was established for the next step in run I. Since a significant scatter occurred in this run, the temperatures were raised from 518 to 820 K for equilibration in a second run.

SOURCE AND PURITY OF MATERIALS:

Cu: 99.9998 % pure (Marz grade) with contents of $2\ 10^{-4}$ % total O, C, and N.
Na: hot trapped, passed through 5 μm stainless steel filter, vacuum distilled, containing $(1.0\text{-}2.0) \cdot 10^{-3}$ % O.
He: "ultrapure", contained $4.5\ 10^{-5}$ % O and $8 \cdot 10^{-5}$ % H_2O after purification.

ESTIMATED ERROR:

Solubility: detection limit $5 \cdot 10^{-5}$ mol %; reading out procedure ± 10%.
Temperature: nothing specified; reading out procedure ± 5 K.

REFERENCES:

COMPONENTS:

(1) Copper; Cu; [7440-50-8]

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

ORIGINAL MEASUREMENTS:

Walker, R.A.; Pratt, J.N.

J. Nucl. Mater. 1969, *32*, 340-345.

VARIABLES:

Temperature: 495-913 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility values of Cu in liquid Na were determined.

T/K	*soly*/mol % Cu	T/K	*soly*/mol % Cu
912.75	$1.778 \cdot 10^{-3}$	700.25	$3.74\ 10^{-4}$
866.0	$2.135 \cdot 10^{-3}$	695.75	$9.88 \cdot 10^{-4}$
835.5	$1.227 \cdot 10^{-3}$	655.0	$2.00 \cdot 10^{-4}$
802.0	$1.143 \cdot 10^{-3}$	564.25	$1.15 \cdot 10^{-4}$
768.0	$6.85\ 10^{-4}$	494.75	$1.61 \cdot 10^{-4}$
712.75	$3.84\ 10^{-4}$		

The equilibrium solid phase was found to be virtually pure Cu. The solubility equation was derived from least square analysis of the data (except at 495, 564, and 696 K) and tested by the compilers:

$$\log\,(soly/\text{mol \% Cu}) = (0.0035 \pm 0.2408) - (2415.3 \pm 185)(T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ni crucible was held in a stainless steel thimble inside a dry Ar box and heated by means of an external resistance furnace. The temperature was measured by two chromel/alumel thermocouples. A piece of Cu was cleaned in 1:1 HNO_3 and dropped into Na just above the melting point. The system was heated at 923 K for 24 hours. During the equilibration Na was stirred with a Ni stirrer. The crucible was then cooled to the sampling temperature and held there for 12 hours. For the last hour the stirring was stopped. Na was sampled into a Ni cup. The sampling was repeated by another cooling step. The sample was dissolved in absolute C_2H_5OH after cooling. Cu was determined by a colorimetric method with diethyldithiocarbamate. The Na content was estimated from the sample weight.

SOURCE AND PURITY OF MATERIALS:

Cu: 99.999 % pure from Johnson Matthey.
Na: "high purity" from UKAEA containing $1.6 \cdot 10^{-3}$ % O.
Ar: 99.999 % pure from Air Products with contents of $< 2 \cdot 10^{-4}$ % O; further purified with 5A molecular sieves and "BTS" catalyst to remove H_2O and O traces respectively.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: average drop during sampling 3 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Copper; Cu; [7440-50-8] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

CRITICAL EVALUATION:

Solubility determinations of Cu in liquid K have not been reported so far. Aleksandrov and Dalakova (3) did not observe any dissolution of Cu in liquid K after an equilibration of the two metals for 1 h at 873-923 K; the detection limit of the spectral analysis used for the determination was not reported. The predicted solubility of Cu in liquid K at the melting point of Cu seems to be much less than 1 mol % Cu (2).
Intermetallic compounds are not formed in the system according to thermal analyses performed by Kienast et al. (1), and the two metals should be immiscible. A schematic Cu-K phase diagram should be similar to that presented for the Cu-Na system with differences in the melting and boiling points of K.

References

1. Kienast, G.; Verma, J.; Klemm, W. Z. *Anorg. Chem.* 1961, *310*, 143.
2. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, 7, 231.
3. Aleksandrov, B.N.; Dalakova, N.V. *Izv.Akad.Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Copper; Cu; [7440-50-8] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

CRITICAL EVALUATION:

Qualitative information on thermal analysis experiments presented by Kienast et al. (1) indicated that intermetallics are not formed in the Cu-Rb system and both metals are immiscible in the liquid state. The solubility of Cu in liquid Rb at the melting point of Cu seems to be significantly lower than 1 mol % Cu (2). A schematic Cu-Rb phase diagram should be similar to that presented for the Cu-Na system with differences in the melting and boiling points of Rb.

References

1. Kienast, G.; Verma, J.; Klemm, W. Z. *Anorg. Chem.* 1961, *310*, 143.
2. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, 7, 28.

COMPONENTS:	EVALUATOR:
(1) Copper; Cu; [7440-50-8] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

CRITICAL EVALUATION:

Godneva et al. (1) performed the only determination of the solubility of Cu in liquid Cs, while several laboratories studied the compatibility of Cu with the liquid metal (2-8). A general characterization of the Cu-Cs system was given on the basis of thermal analyses (9) without presenting quantitative data. The two metals do not form intermetallics, and they are practically immiscible in the liquid state.
Godneva et al. (1) observed an increase of the solubility from $4.6 \cdot 10^{-3}$ to $3.1 \cdot 10^{-2}$ mol % Cu in the temperature range 323-573 K. The results were well consistent, and the solubility equation fits them very well:

$$\log(soly/\text{mol \% Cu}) = -0.409 - 621.3(T/K)^{-1} \qquad r=0.999$$

One has to consider, however, that the slope of this curve results in a too low temperature coefficient of the solubility compared to the solubility of Cu in Na. The data of the solubility of Cu in Na were overestimated in the temperature range below 573, this error might have influenced also the results of (1) (see critical evaluation Cu-Na system).
Cu should be dissolved in liquid Cs at 675 K to the extent of $2 \cdot 10^{-4}$ mol % Cu according to Winslow's estimation on the basis of corrosion tests (7), significantly lower than the results of (1). Though this result seems to be more reliable, the work (7) is not a solubility determination and is, therefore not compiled.
Since the stability of CuO is quite low, increases of the solubility of Cu in liquid Cs due to an increase of the O concentration were never observed. A hardly understandable decrease of the solubility of Cu in Cs with a content of 0.8 mol % O was reported in (1).
All corrosion tests (2-8) indicated a slight dissolution of Cu in liquid Cs in the temperature range 673 to 1173 K. The corrosion rates increased with increasing temperature. Keddy (10) observed that Cu was not leached out of some alloys with Ag or Au at 773 K. This observation allows the conclusion that the solubility of Cu might be lower than those of Ag or Au in liquid Cs.
A predictive Cu-Cs phase diagram should be analogue to that of the Cu-Na system.

Doubtful values of the solubility of Cu in liquid Cs

T/K	$soly$/mol % Cu	source
323	$4 \cdot 10^{-3}$	(1)
373	$8 \cdot 10^{-3}$	(1)
473	$2 \cdot 10^{-2}$	(1)
573	$3 \cdot 10^{-2}$	(1)

References

1. Godneva, M.M.; Sedelnikova, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.
2. Harvey, R. *US Atom.Ener.Comm.Rep. MND-P-3009-1*, 1959.
3. Slivka, M.J. *Adv. Ener. Conver.* 1963, *3*, 157.
4. Smith, R.G.; Hargreaves, F.; Mayo, G.T.J.; Thomas, A.G. *J. Nucl. Mater.* 1963, *10*, 191.
5. Hargreaves, F.; Mayo, G.T.J.; Thomas, A.G. *J. Nucl. Mater.* 1966, *18*, 212.
6. 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.
7. Winslow, P.M. *Corrosion* 1965, *21*, 341.
8. Sedelnikov, V.A.; Godneva, M.M. *Issledovaniya Fiziko-Khimicheskikh Svoistv Soedinenii Redkikh Elementov*, Nauka, Leningrad, 1978, p. 56.
9. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.
10. Keddy, E.S. *US Atom.Ener.Comm.Rep. LAMS-2948*, 1963.

COMPONENTS:

(1) Copper; Cu; [7440-50-8]

(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 % O

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cu in liquid Cs containing 0.08 mol % O was reported.

t/°C	*soly*/ mass % Cu	*soly*/mol % Cu [a]
50	$2.2 \cdot 10^{-3}$	$4.6 \cdot 10^{-3}$
100	$4.0 \cdot 10^{-3}$	$8.4 \cdot 10^{-3}$
200	$9.6 \cdot 10^{-3}$	$2.0 \cdot 10^{-2}$
300	$1.45 \cdot 10^{-2}$	$3.1 \cdot 10^{-2}$
300 [b]	$2.7 \cdot 10^{-3}$	$5.7 \cdot 10^{-3}$

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

The results of the determinations of the Cu solubility in Cs with 0.08 mol % O are approximated by the equation (by the compilers):

$$\log(soly/\text{mol \% Cu}) = -0.409 - 621.3(T/\text{K})^{-1} \qquad r=0.999$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Cu specimen was dipped into Cs under vacuum and equilibrated for 120 hours in a glass ampoule. An attack of Cs on the glass was not observed. Cs was cooled and dissolved in H_2O and its amount was determined by titration of a portion of the primary solution with an acid. Cu(II) was extracted from another portion of the tartrate-ammonia solution into Na-diethyldithiocarbaminate in CCl_4 and the complex formed was determined by colorimetry according to (1). The O concentration in Cs was increased by a controlled decomposition of $KClO_3$-MnO_2 mixture.

SOURCE AND PURITY OF MATERIALS:

Cu: "M0" or "M1" purity containing $3 \cdot 10^{-3}$ % Mg.
Cs: 98-99 % pure, further purified by vacuum distillation; 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) Silver; Ag; [7440-22-4] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

CRITICAL EVALUATION:

Two different illustrations of the Ag-Li phase diagram were published by Pastorello (1) and by Freeth and Raynor (2). The phase relations presented in (2) are unquestionably more convincing due to the careful and exhaustive investigations as well as to the higher purity of the materials used.

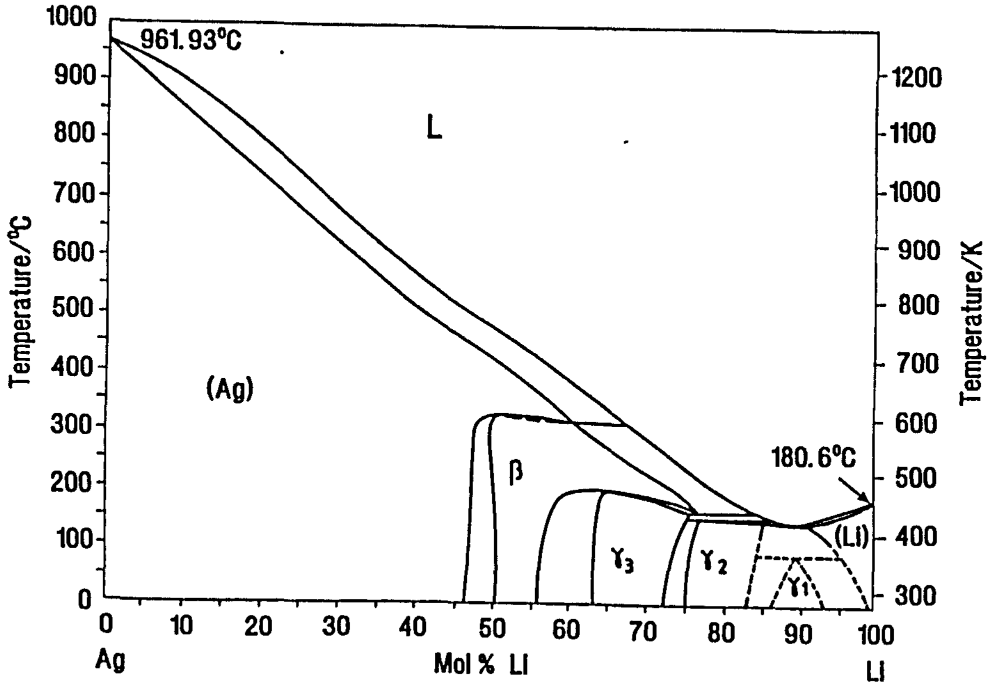
The liquidus lines of (1) and (2) show rough agreement in the composition range 80-100 mol % Ag. (1) observed the congruent melting of AgLi and Ag_3Li at 1223 and 723 K, respectively, at lower Ag concentrations. A systematic decrease of the liquidus temperature with slight peritectic inflections was reported in (2); this is indicated in the phase diagram shown in the Figure which was taken from (3). Some bends which were observed on the liquidus line of (2) were ascribed to an ordering effect at certain compositions in the liquid alloys (4).

The solubility of Ag in liquid Li was evaluated to be equal or higher than 56.5 mol % Ag at 830 K (4). This is in agreement with the results of (2). The study of Becker et al. (4) was, however, not a determination of the solubility. It is therefore not compiled.

The saturated solutions of Ag in liquid Li are in equilibrium with Ag-Li intermediate phases as is shown in the Figure.

Tentative values of the solubility of Ag in liquid Li

T/K	*soly*/mol % Ag	source
418	11 eutectic	(2)
473	19	(2) interpolated
573	30	(2) interpolated
673	40	(2)
773	51	(2) interpolated
873	62	(2)
973	71	(2)
1073	81	(2)
1173	92	(2) interpolated



References

1. Pastorello, S. *Gazz. Chim. Ital.* 1931, *61*, 47.
2. Freeth, W.E.; Raynor, G.V. *J. Inst. Met.* 1953-1954, *82*, 569.
3. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, *7*, 223.
4. Becker, W.; Schwitzgebel, G.; Ruppersberg, H. *Z. Metallk.* 1981, *72*, 186.

COMPONENTS: (1) Silver; Ag; [7440-22-4] (2) Lithium; Li; [7439-93-2]	**ORIGINAL MEASUREMENTS:** Pastorello, S. *Gazz. Chim. Ital.* 1931, *61*, 47-51.
VARIABLES: Temperature : 643-1223 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The experimental points on the Ag-Li liquidus were determined.

t/°C	*Soly*/mol % Ag	*t*/°C	*Soly*/mol % Ag
943	94.8	850	40.2
893	88.7	623	32.5
820	84.1	410	30.8
700	75.8	450	25.0
670	68.3	413	20.0
830	60.6	370	15.2
950	50.7		

The melting points of Ag and Li were reported to be 961 and 180 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by melting both the metals in a stainless steel crucible in an Ar atmosphere. The composition of the alloys was confirmed by chemical analysis. The cooling curves of the melts were recorded by means of Pt/Pt-Rh thermocouples calibrated on the melting point of Cu. Slight corrosion effects of the alloys on the material of the crucibles were observed.

SOURCE AND PURITY OF MATERIALS:

Ag: "pure" from Kahlbaum.
Li: "pure" from Kahlbaum.
Ar: purified by reaction with Ca and Mg at 773 K in a closed system.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Silver; Ag; [7440-22-4]

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

ORIGINAL MEASUREMENTS:

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

J. Inst. Met. 1953-1954, *82*, 569-574.

VARIABLES:

Temperature : 418-1227 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The experimental points from the Ag-Li liquidus were read out from the figure by the compilers.

t/°C	*Soly*/mol % Ag	*t*/°C	*Soly*/mol % Ag	*t*/°C	*Soly*/mol % Ag
182	1.0	251	25.0	433	44.2
177	2.5	256	25.0	449	46.0
174	3.0	257	25.5	457	47.0
171	3.5	266	25.7	470	48.0
168	3.7	268	26.2	485	50.0
165	4.6	276	27.0	490	50.7
163	5.0	283	28.0	497	51.2
159	6.1	306	30.7	504	52.2
156	7.2	317	32.2	525	55.1
153	8.5	340	34.0	546	57.1
151	9.0	358	36.5	564	59.2
149	10.0	382	38.5	596	62.1
145.5	10.8	396	39.8	651	66.7
150	12.0	400	40.0	705	71.7
153	13.2	401	40.5	760	76.0
164	15.0	403	41.0	805	81.0
172	16.0	408	41.2	849	85.0
190	18.0	410	41.7	892	90.0
210	20.0	413	42.0	919	93.0
235	23.0	422	43.0	954	97.0
249	24.2	425	43.2		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were melted in a furnace unit in which a mild steel crucible and an Ar atmosphere were established. Ag was first melted in order to calibrate a Chromel/Alumel thermocouple, and Li was then added. Both metals were carefully weighed. The composition of some samples was chemically analyzed.

SOURCE AND PURITY OF MATERIALS:

Ag: 99.99 % pure from Johnson, Matthey and Co., Ltd.
Li: "high purity" from New Metals and Chemicals, Ltd., and contained < 0.02 % Na.
Ar: "purified".

ESTIMATED ERROR:

Nothing specified.
Solubility: reading-out procedure ±0.1 mol %.
Temperature: reading-out procedure ± 1 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Silver; Ag; [7440-22-4] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

CRITICAL EVALUATION:

The interpretation of the solubility data for Ag in liquid Na is related to the Ag-Na phase diagram. It was stated (1) that Ag is not dissolved in liquid Na in measurable amounts even at higher temperatures. The addition of $6.4 \cdot 10^{-2}$ mol % Ag caused a depression of the melting point of Na by only 0.09 K (2). The temperature might be considered as a eutectic; the composition, however, does not correspond to the eutectic one which should be only about half of the Ag concentration. This work is not compiled. The eutectic temperature was confirmed by (3) and (13).
The liquidus line in the range of higher Ag contents was established by (3-5,13). The results in the composition range 30 to 70 mol % Ag, in which almost a plateau with a tendency of demixing was observed, were in substantial agreement. Outside this region differences of up to 50 K in the liquidus temperatures occurred. The data of (3) and (4) were consistent, and may be considered as tentative for compositions $\geq$ 30 mol % Ag. At lower Ag contents between 10 and 30 mol % the results of Quercigh and Bruni (3) and Feschotte and Spitz (13) are selected as the most reliable values.
The solubility of Ag in liquid Na between the melting point of Na and 923 K was determined in different studies (6,7,10) in which no information on experimental details was given. Kovacina (7) observed an increase of the solubility from about 0.3 to 9 mol % Ag in the temperature range 423-673 K. Brush (6) presented a solubility curve extending from 0.65 to 15 mol % Ag at 477 to 922 K. Kuzin et al. (10) expressed their results at 373 to 573 K in a solubility equation:

$$\log(soly/\text{mol \% Ag}) = (3.51 \pm 0.37) - (1980 \pm 310)(T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

The solubility data of Lamprecht and Crowther (8) and Weeks (9) were in very good agreement at temperatures below 533 K and the recommended solubility equation for the interval 371-580 K may be formulated (as found by the compilers):

$$\log(soly/\text{mol \% Ag}) = 2.682 - 1541(T/\text{K})^{-1} \quad r = 0.996 \qquad \text{Eq.(2)}$$

Claar (12) presented a different solubility equation on the basis of the data of (8) and (9). The data of Weeks (9) showed a significant scatter at > 580 K. The line drawn between the single points resulted in a slightly different temperature coefficient which was lower than that of the solubility determinations of (9). The thermal arrests in (5) and (13) at 595 K, corresponding to the peritectic decomposition of Ag_2Na, could not be confirmed or denied by the results of the solubility experiments of (9). Results of Feschotte and Spitz (13) were in agreement with data of (3,4,5) and partly (8) and with the calculated liquidus in the assessed phase diagram of (11).

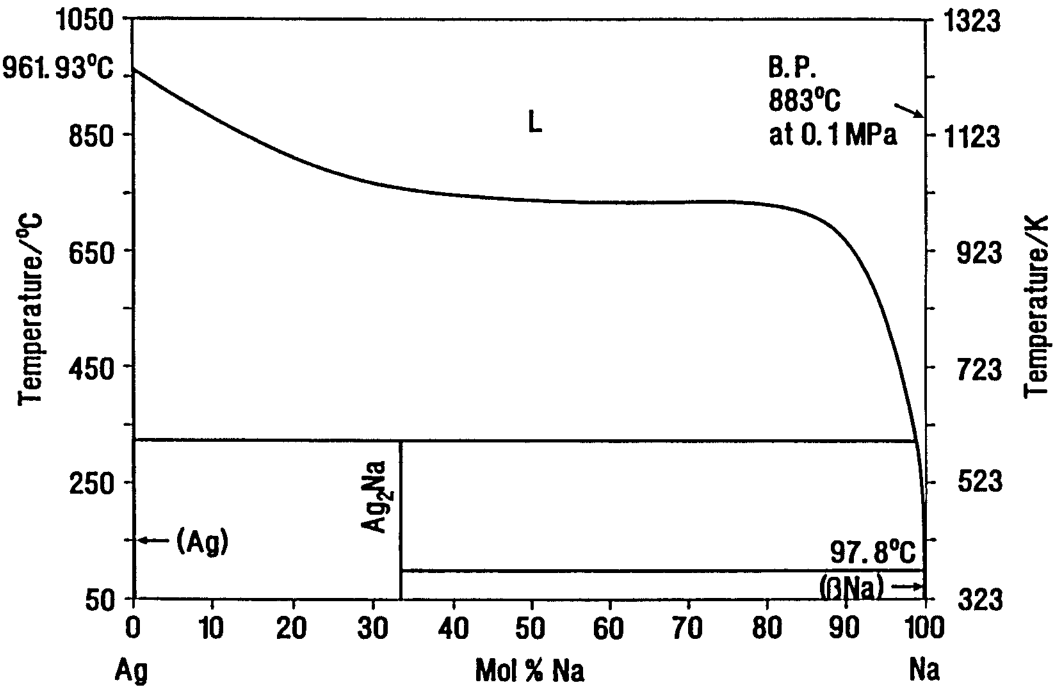
A schematic phase diagram of the Ag-Na system is redrawn after (11).

Recommended (r), tentative (t) and doubtful (d) values of the solubility of Ag in liquid Na

T/K	$soly$/mol % Ag	source
373	$3 \cdot 10^{-3}$ (t)	(8),(9) extrapolated
423	0.11 (r)	(8),(9)
473	0.27 (t)	(8),(9)
573	0.98 (t)	(8),(9) extrapolated
595	1.1 (d) peritectic	(8),(9) interpolated
773	4 (d)	(8),(13) interpolated
873	7 (d)	(3),(4),(5),(13) interpolated
973	14 (d)	(3),(4),(5),(13) interpolated
1023	55 (r)	(3),(4),(5),(13) interpolated
1073	75 (t)	(3),(4),(5) interpolated
1173	92 (t)	(3)

COMPONENTS:	EVALUATOR:
(1) Silver; Ag; [7440-22-4] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

CRITICAL EVALUATION: (continued)



References

1. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1889, *55*, 666.
2. Tammann, G. *Z. Phys. Chem.* 1889, *3*, 441.
3. Quercigh, E.; Bruni, G. *Z. Anorg. Chem.* 1910, *68*, 301.
4. Mathewson, C.H. *Intern. Z. Metallogr.* 1911, *1*, 51.
5. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.
6 Brush, E.G. *Corrosion* 1955, *11*, 299t.
7. Kovacina, T.A., Naval Research Lab., 1964; private communication to J.R. Weeks, as rep. in (9).
8. Lamprecht, G.J.; Crowther, P. *Trans. AIME* 1968, *242*, 2169; also Lamprecht, G.J. *Ph.D. thesis*, Univ. of South Africa, Pretoria, 1966.
9. Weeks, J.R. *Trans. Am. Soc. Met.* 1969, *62*, 304.
10. Kuzin, A.N.; Lyublinskii, I.E.; Beskorovainyi, N.M. *Raschety i Eksperimentalnye Metody Postroeniya Diagram Sostoyaniya*, Nauka, Moskva, 1985, p. 113.
11. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, *7*, 133.
12. Claar, T.D. *Reactor Technol.* 1970, *13*, 124.
13. Feschotte, P.; Spitz, E.L. *J.Less-Common Met.* 1988, *138*, 155.

COMPONENTS:

(1) Silver; Ag; [7440-22-4]

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

ORIGINAL MEASUREMENTS:

Quercigh, E.; Bruni, G.

Z. Anorg. Chem. 1910, *68*, 301-306.

VARIABLES:

Temperature: 938-1167 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some experimental points on the liquidus line of the system Ag-Na were reported.

t/°C	*soly*/mol % Ag	*t*/°C	*soly*/mol % Ag
894	91.8	745	40.4
873	88.6	742	35
845	86.2	730	30
825	82.3	725	25
772	71.0	722	20
764	61.0	713	15
748	50.5	665	10

The melting points of Ag and Na were found to be 961.5 and 97.5 °C, respectively. An addition of Ag to Na caused only a decrease of the melting point of Na by 0.1 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The surface of the Na samples was cut off before the alloys were prepared. Both metals were weighed and introduced into a container filled with N_2. The composition of the alloys was chemically analyzed. The alloys were heated to more than 1150 K. Cooling curves were recorded by means of a Le Chatelier's thermoelement.

SOURCE AND PURITY OF MATERIALS:

Ag: "purest" from Kahlbaum.
Na: "purest" from Kahlbaum.
N_2: "pure", additionally dried.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Silver; Ag [7440-22-4]

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

ORIGINAL MEASUREMENTS:

Mathewson, C.H.

Intern. Z. Metallogr. 1911, *1* , 51-63.

VARIABLES:

Temperature: 864-1140 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Ag-Na liquidus line were determined.

t/°C	*soly*/mol % Ag
591	8.3
686	15.6
710	23.8
735	30.9
745	41.1
747	51.4
753	61.9
781	72.2
867	88.1

The melting points of Ag and Na were found to be 960 and 97.5 °C, respectively.

Attempts to dissolve Ag in Na at temperatures close to 100 °C were not successful. The saturated solutions were observed to be in equilibrium with Ag containing less than a few tenth % Na. A decrease of the melting point of Na due to the addition of Ag was not observed.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was weighed in glass tubes in order to avoid oxidation. Weighed Ag and Na were rapidly heated in a larger Jena glass tube to the required temperature in H_2 atmosphere. A Fe wire was used to stir the melt. The cooling curves were recorded by means of a Pt/Pt-Rh thermocouple, calibrated at the melting points of Pb, Zn, Sb, and Ag. Concentrations above 50 mol % Ag were checked by means of chemical analyses. Ag was dissolved in HNO_3 and titrated with KCNS, Na was transferred into NaCl, and determined by precipitation of AgCl.

SOURCE AND PURITY OF MATERIALS:

Ag: "unquestionable" purity.
Na: gave no metallic residue after treatment with alcohol and H_2O.
H_2: "dry".

ESTIMATED ERROR:

Solubility: accuracy of sample preparation ± 0.4 %.
Temperature: precision ± 1K.

REFERENCES:

COMPONENTS:

(1) Silver; Ag; [7440-22-4]

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

ORIGINAL MEASUREMENTS:

Kienast, G.; Verma, J.; Klemm, W.

Z. Anorg. Chem. 1961, *310*, 143-169.

VARIABLES:

Temperature: 493-1203 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus temperatures of the Ag-Na system were determined.

t/°C	*soly*/mol % Ag	t/°C	*soly*/mol % Ag	t/°C	*soly*/mol % Ag
220	0.2	740	32.5	800	73.0
480	2.1	(740)	38.6	820	76.7
540	3.9	(744)	40.8	860	79.5
590	6.4	(744)	47.1	880	81.9
660	7.3	(744)	51.0	890	84.9
700	14.9	750	55.3	910	86.2
721	18.3	760	57.7	930	87.3
725	20.0	763	64.5		
730	25.6	775	69.4		
735	27.5	785	70.2		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Both metals were weighed and placed in a corund crucible inserted in a quartz tube filled with Ar according to (1). Corund as well as quartz were slightly attacked by the molten alloy. The composition of the alloys was chemically analyzed. After 60 h of equilibration at 1073 K the Al content in the molten alloy was 0.05 mass %. Cooling curves of the melt were recorded by means of a Pt/Pt-Rh thermoelement.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 2 K, according to (1).

REFERENCES:

1. Dorn, F.W.; Klemm, W. *Z. Anorg. Chem.* 1961, *309*, 189-203.

COMPONENTS:

(1) Silver; Ag; [7440-22-4]

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

ORIGINAL MEASUREMENTS:

Lamprecht, G.J.; Crowther, P.

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

VARIABLES:

Temperature: 409-533 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ag in liquid Na was determined at several temperatures.

t/°C	*soly*/mass % Ag	*soly*/mol % Ag
135.6 [a]	0.35	$7.4 \cdot 10^{-2}$
148.3 [b]	0.50	0.100
177.0 [a]	0.84	0.180
195.0 [c]	1.18	0.25
203.6 [b]	1.26	0.27
211.0 [a]	1.49	0.32
218.6 [b]	1.68	0.36
239.6 [c]	2.31	0.50
260.0 [c]	3.09	0.67

[a] heating sequence [b] cooling sequence [c] from the intermetallic compound Ag-Na

The solubility equation was presented by the authors and confirmed by the compilers:

$$\log(soly/\text{mol \% Ag}) = 2.899 - 1642(T/\text{K})^{-1}$$

The results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility apparatus was made of Pyrex glass, as described in (1,2). He was used as cover gas in all experiments. Ag was melted with radioactive ^{110}Ag, obtained by irradiation. Ag was placed in one reaction cell, Na in another one. The cells were connected by means of a capillary tube. Molten Na was transferred into the Ag cell by means of He pressure. The metals were in contact until there was no further change of the activity of Ag in liquid Na, which was transferred back into its primary cell for decay measurements. The variation of the solubility with temperature was determined by measuring the increase or decrease of the activity in the saturated Na in which the equilibrium was reached from above and below a selected temperature.

SOURCE AND PURITY OF MATERIALS:

Ag: spectroscopically pure from Johnson Matthey & Co.
Na: filtered, from Merck, containing $1.1 \cdot 10^{-3}$ mol % O.
He: purified with molecular sieves, activated charcoal trap at the temperature of liquid N_2.

ESTIMATED ERROR:

Solubility: standard deviation ± 3 %.
Temperature: nothing specified.

REFERENCES:

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

COMPONENTS:

(1) Silver; Ag; [7440-22-4]

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

ORIGINAL MEASUREMENTS:

Weeks, J.R.

Trans. Am. Soc. Met. 1969, *62*, 304.

VARIABLES:

Temperature: 373-793 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ag in liquid Na was determined at several temperatures, the data were read out from the figure by the compilers.

t/°C	*soly*/mol % Ag	t/°C	*soly*/mol % Ag
100 [a]	$4.2 \cdot 10^{-2}$	359 [b]	1.3
150 [a,b]	0.11	411 [a]	3.0
200 [a]	0.29	468 [b]	3.1
250 [b]	0.60	518 [a]	5.5
307 [a]	0.90	520 [b]	5.3

[a] after heating to equilibrium temperature [b] after coling to equilibrium temperature

The results below 307 °C were approximated by the equation,

$$\log(soly/\text{mol \% Ag}) = 2.449 - 1439(T/\text{K})^{-1}$$

and above 359 °C by the equation

$$\log(soly/\text{mol \% Ag}) = 2.891 - 1693(T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were given into a Zr crucible in a glove box. They were heated inside a stainless steel apparatus filled with He to 773 K for a weekend. Some samples were taken after the melt had been cooled, some other ones after it had been heated to the equilibrium temperature for $\geq$ 18 h. The solution was then filtered, and 10 samples were collected. The samplers were cut open after cooling. The frozen samples were dissolved in CH_3OH and H_2O. Ag, precipitated in fine crystals, was filtered, dissolved in HNO_3, and determined by means of titration with NaCl solution.

SOURCE AND PURITY OF MATERIALS:

Ag: 99.999+ % pure from American Smelting and Refining Co.
Na: from Mine Safety Appliances, further purified by hot trapping.

ESTIMATED ERROR:

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

REFERENCES:

COMPONENTS:

(1) Silver; Ag; [7440-22-4]

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

ORIGINAL MEASUREMENTS:

Feschotte, P.; Spitz, E.L.

J.Less-Common Met. 1988, *138*, 155-160.

VARIABLES:

Temperature: 453-1015 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Thermal analyses of ten samples of Ag-Na alloys with contents of up to 80 mol % Ag were performed. The results of these analyses were presented in the form of a smooth curve. They were claimed to confirm the calculated liquidus of (1) within ± 10 K. Some numerical data were inserted in the publication and some others were privately reported by the author (2).

t/°C	*soly*/mol % Ag
180 [a]	0.5
565 [a]	5.0
737	55.0
742	80.0

[a] from (2)

The peritectic temperature of the decomposition of Ag_2Na was found to be 322 °C, the eutectic temperature on the Na-rich side was found to be at 97.5±0.5 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by melting both metals in a Ni crucible at 1023-1073 K. Differential thermal analysis of the alloy sample was performed in an Ar atmosphere. The equilibrium solid phase was analyzed by means of chemical, metallographical and X-ray methods after the selective dissolution of excess Na in C_2H_5OH.

SOURCE AND PURITY OF MATERIALS:

Ag: 99.99 % pure from Métaux Précieux.
Na: 99 % pure; its surface was cut off and it was distilled twice.
Ar: 99.99 % pure.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: reproducibility ± 3 K.

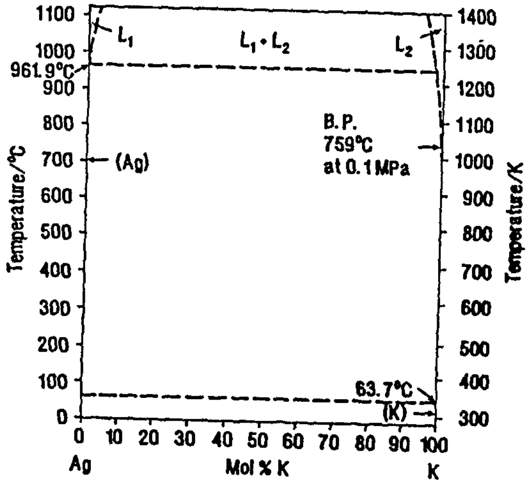
REFERENCES:

1. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, *7*, 133.
2. Feschotte, P., private communication, Univ. of Lausanne, Switzerland, 1990.

COMPONENTS:	EVALUATOR:
(1) Silver; Ag; [7440-22-4] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

CRITICAL EVALUATION:

Epstein and Weber (1) reported that the K-Na (1:1) melt which was saturated with Ag contained less than $3 \cdot 10^{-4}$ mol % Ag at room temperature; they did not describe any details of the experiments. A solubility of about 1 mol % Ag is to be expected at the melting temperature of Ag according to Pelton's prediction (2). A qualitative characterization by Kienast et al. (3) indicated that Ag and K do not form intermetallics and should have quite low mutual solubility in the liquid and solid states. Aleksandrov and Dalakova (4) reported a dissolution of Ag in liquid K at 873-923 K; they did not give quantitative information on the extent of the dissolution.
A schematic Ag-K phase diagram reflects the information of (2) and (3); the K has to be kept in the liquid state at constrained pressure.



References

1. Epstein, L.F.; Weber, C.E. *US Atom.Ener.Comm.Rep. TID-2501*, 1951, p. 515.
2. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, *7*, 223.
3. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.
4. Aleksandrov, B.N.; Dalakova, N.V. *Izv.Akad.Nauk SSSR, Met.* 1982, no. 1, 133.

COMPONENTS:	EVALUATOR:
(1) Silver; Ag; [7440-22-4] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

CRITICAL EVALUATION:

No intermetallic compounds should be formed in the Ag-Rb system, and both metals are barely miscible in the liquid and solid states according to Kienast et al. (1). Quantitative results were not reported.
A Ag-Rb phase diagram should be similar to that of the Ag-K system.

References

1. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.

COMPONENTS:	EVALUATOR:
(1) Silver; Ag; [7440-22-4] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

CRITICAL EVALUATION:

Kienast et al. (1) characterized the Ag-Cs system. They stated a strong tendency to immiscibility in the liquid and solid states and excluded the formation of intermetallic compounds between both metals. However, Gukova and Ermolaev (7) reported the detection of Ag_2Cs_3 molecules in a flame.
Godneva et al. (2) performed determinations of the solubility of Ag in liquid Cs at 323 to 573 K. They observed an increase of the Ag concentration in the saturated solutions from $6 \cdot 10^{-5}$ to $1.2 \cdot 10^{-4}$ mol % Ag. Their results at 323, 373, and 473 K showed linearity of the plot of logarithm of solubility vs. reciprocal temperature, while only one half of the other values layed on the line. Therefore, a solubility equation could not be recommended. Godneva et al. (2) observed an increase of the Ag solubility by a factor of 7, if Cs contained 0.8 mol % O. This phenomenon is not understandable, since Ag oxides are not stable and the formation of a ternary Ag-Cs-O compound seems to be impossible from chemical and thermodynamic reasons. The influence of O on the dissolution of Ag in liquid Cs was, however, confirmed in corrosion tests of Sedelnikov and Godneva (6).
Keddy (4) observed the partial dissolution of a Ag sample in corrosion tests at 773 K. Smith et al. (5) noticed moderate and severe attack of Cs on Ag at 773 and 873 K, respectively. Harvey (3) did not detect any corrosion effects in tests at 698 K.
An Ag-Cs phase diagram is not available, it should be analogous to that of the Ag-K system.

Tentative values of the solubility of Ag in liquid Cs

T/K	$soly$/mol % Ag	source
323	$6 \cdot 10^{-5}$	(2)
373	$9 \cdot 10^{-5}$	(2)
473	$1.2 \cdot 10^{-4}$	(2)

References

1. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.
2. Godneva, M.M.; Sedelnikowa, N.D.; Geizler, E.S. *Zh. Prikl. Khim.* 1974, *47*, 2177.
3. Harvey, R. *US Atom.Ener.Comm.Rep. MND-P-3009-1*, 1959.
4. Keddy, E.S. *US Atom.Ener.Comm.Rep. LAMS-2948*, 1963.
5. Smith, R.G.; Hargreaves, F.; Mayo, G.T.J.; Thomas, A.G. *J. Nucl. Mater.* 1963, *10*, 191.
6. Sedelnikov, V.A.; Godneva, M.M. *Issledovaniya Fiziko-Khimicheskikh Svoistv Soedinenii Redkikh Elementov*, Nauka, Leningrad, 1978, p. 56.
7. Gukova, Ya.Ya.; Ermolaev, M.I. *Obshchie Zakonomernosti v Stroenii Diagram Sostoiania Metallicheskikh Sistem*, Nauka, Moskva, 1973, p. 135.

COMPONENTS:

(1) Silver; Ag; [7440-22-4]

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

ORIGINAL MEASUREMENTS:

Godneva, M.M.; Sedelnikowa, N.D.; Geizler, E.S.

Zh. Prikl. Khim. 1974, *47*, 2177-2180.

VARIABLES:

Temperature: 323-573 K
O concentration in Cs: 0.08 or 0.8 mol % O

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ag in liquid Cs at several temperatures was determined.

t/°C	*soly*/mass % Ag	*soly*/mol % Ag [a]
50	$5\ 10^{-5}$	$6 \cdot 10^{-5}$
100	$7 \cdot 10^{-5}$	$9 \cdot 10^{-5}$
150	$4 \cdot 10^{-5}$	$5 \cdot 10^{-5}$
200	$1.0 \cdot 10^{-4}$	$1.2 \cdot 10^{-4}$
300	$1.0\ 10^{-4}$	$1.2 \cdot 10^{-4}$
300 [b]	$7.4 \cdot 10^{-4}$	$9 \cdot 10^{-4}$

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

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An Ag specimen was covered with liquid Cs under vacuum and equilibrated for 120 h in a glass ampoule. No change of the glass due to corrosion reactions with the liquid alloy was observed. Cs was cooled and dissolved in H_2O. Its amount was determined in this solution by titration of a portion with an acid. Ag was extracted from another portion of the solution with CCl_4 to form a complex with dithizone and colorimetrically determined (1). The O concentration in Cs was increased by means of a controlled decomposition of a $KClO_3$-MnO_2 mixture.

SOURCE AND PURITY OF MATERIALS:

Ag: 99.9 % pure, containing $7 \cdot 10^{-3}$ % Fe, $1 \cdot 10^{-2}$ % Cr, $1 \cdot 10^{-2}$ % Ni, and $<1 \cdot 10^{-3}$ % Cu.
Cs: 98-99 % pure, further purified by vacuum distillation, 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) Gold; Au; [7440-57-5] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1989

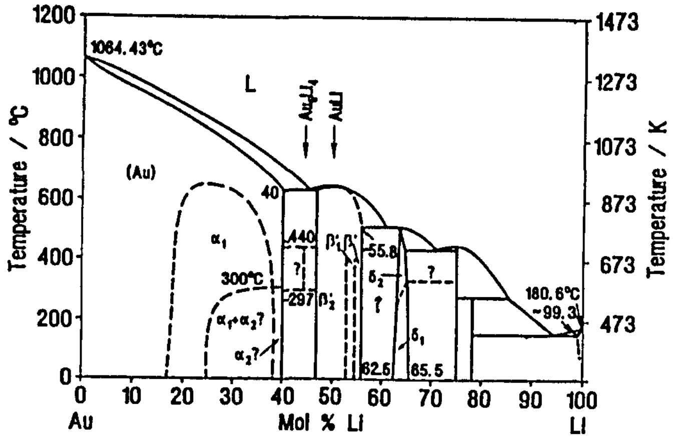
CRITICAL EVALUATION:

The liquidus line of the Au-Li system seems to be well established since almost 70 compositions were thermally analyzed by Kienast et al. (1). These results formed a logical sequence and moreover, the melting point of the compound AuLi was confirmed in conductivity measurements at 921 K by Nicoloso et al. (2) and in ^{7}Li Knight shift measurements at 918 K by van der Marel et al. (4) who did not report more details on the experiments. Nevertheless, it should be noticed that the thermal arrests for the most Li-rich alloys were higher than the melting point of pure Li. The system behaved as a typical eutectic at larger Au concentrations. This fact may be a reason to look on the temperature scale of the liquidus with caution.
The assessed Au-Li phase diagram was reported by Pelton (3) and is redrawn in the figure below. The dashed lines separating the α, β, and δ phases are not to be considered as true phase boundaries, since they were indicated only in X-ray studies as different structures. They need further examination.

Tentative and recommended (r) values of the Au solubility in liquid Li

T/K	*soly*/mol % Au		source
428	5.3	eutectic	(1)
550	14	peritectic	(1)
723	25	congruent	(1)
709	29	eutectic	(1)
784	39	peritectic	(1)
920	50 (r)	congruent	(1),(2),(4) mean value
909	55	eutectic	(1)
973	60		(1)
1073	68		(1)
1173	79		(1)

The values at higher temperatures may be estimated by interpolation in the figure.



References

1. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.
2. Nicoloso, N.; Schmutzler, R.W.; Hensel, F. *Ber. Bunsenges. Phys. Chem.* 1978, *92*, 621
3. Pelton, A. D. *Bull. Alloy Phase Diagr.* 1986 *7*, 228
4. van der Marel, C.; Geertsma, W.; Drent, E.; Kuiper, P.; van der Marel, D.; van der Lugt, W. *Z. Phys. Chem. N.F.* 1988, *156*, 569

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Kienast, G.; Verma, J.; Klemm, W.

Z. Anorg. Chem. 1961, *310*, 143-169.

VARIABLES:

Temperature : 428-1231 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus temperatures of the Au-Li system were determined.

t/°C	*soly*/mol % Au	*t*/°C	*soly*/mol % Au	*t*/°C	*soly*/mol % Au
186	0.21	449	26.4	655	57.0
184	0.34	447	27.9	661	58.0
183	0.61	457	30.2	675	59.3
181	0.72	487	32.3	706	60.8
179	0.8	495	33.2	732	61.6
175	1.5	505	34.9	744	63.8
163	3.6	509	35.5	752	65.1
155	5.1	511	37.3	776	66.0
172	6.1	517	38.5	786	67.0
180	7.5	537	40.0	813	69.1
205	9.4	566	41.3	825	70.6
223	10.2	578	42.0	844	72.1
237	11.4	591	42.7	851	73.0
257	12.5	609	44.2	855	74.2
278	14.3	628	45.9	864	75.4
299	16.6	645	48.2	871	76.5
366	18.2	645	49.5	879	77.6
393	19.4	646	50.7	895	78.7
408	20.2	655	51.7	912	79.9
422	21.4	641	53.0	917	82.0
434	22.0	638	53.8	939	83.8
448	24.1	635	54.5	958	86.0
450	25.5	651	55.8		

The points of the thermal analyses in the neighbourhood of pure Li are several K above the melting point of Li (180.6 °C)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a W crucible in an Ar atmosphere because of their high reactivity. The surface of solid Li was cut off and the sample was weighed and placed into the crucible. The corresponding amount of Au powder was added, the mixture was heated for 6 hours to 450-550 K and then the temperature was elevated to 100 K above the liquidus for some hours. Thermal analyses of the alloys were performed during the mixing. The chemical analyses were carried out after dissolution in aqua regia. Au was weighed after subsequent precipitation and Li determined by means of flame photometry.

SOURCE AND PURITY OF MATERIALS:

Au: nothing specified.
Li: from Hans-Heinrich-Hütte, contained 0.6 % Na, 0.2 % O, 0.1 % Fe_2O_3 + SiO_2 + C, 0.1 % N.
Ar: from Linde, further purified over Ti at higher temperature.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: accuracy not better than ± 10 K, (by compilers).

REFERENCES:

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Nicoloso, N.; Schmutzler, R.W.; Hensel, F.

Ber. Bunsenges. Phys. Chem. 1978, *82*, 621- 623.

VARIABLES:

One temperature : 921 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The melting point of the compound AuLi was found to be 648 °C, as was read out from the figure by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloy was prepared by melting the accurately weighed amounts of both metals under vacuum in cells made of Mo. All experimental procedures were carried out in a specially constructed metallic vacuum vessel. The conductivity measurements were made using the "four probe" method eliminating the contact resistance errors. A drop of the conductivity of the AuLi melt with decreasing temperature corresponded to the point on the liquidus line.

SOURCE AND PURITY OF MATERIALS:

Au: 99.999 % pure.
Li: 99.98 % pure.

ESTIMATED ERROR:

Solubility: accuracy ± 0.1 mol %. Temperature: nothing specified; reading-out procedure ± 3 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Gold; Au; [7440-57-5] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1990

CRITICAL EVALUATION:

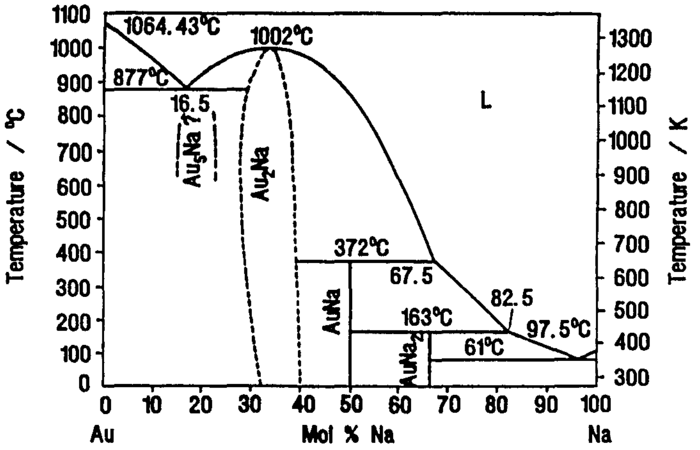
The solubility of Au in liquid Na is quite large. Additions of up to 3.5 mol % Au decrease the melting point of Na to 354 K. The early publications (1-3) show good agreement in this range of concentrations. The results of (4) differ from these data by more than the experimental error. The reliability of the data of (4) seems, therefore, to be questionable even in the range of higher temperatures. The solubility values from (3) and (4) are in a band of width ± 20 K. The single determinations of (5), (6) and (8) indicated a slightly lower solubility of Au than recorded in (4) in the temperature range 650-1050 K. The result of (3) at 975 K may be rejected, since it shows internal inconsistency and deviates from other data (4-6).

The results presented in (6) are not compiled, since experimental details were not reported. A solubility of 45 mol % Au at 1048 K was established by means of a coulometric titration using a solid electrolyte.

The saturated solutions of Au in liquid Na are in equilibrium with $AuNa_2$ (below 536 K), AuNa below 645 K) and Au_2Na up to 1275 K, as is indicated in the Au-Na phase diagram which was reported in (7). The existence of a compound Au_6Na needs to be confirmed.

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

T/K	*soly*/mol % Au	source
354	3.5 (t) eutectic	(1,3), mean value
373	4.5 (t)	(3)
436	15 (d) peritectic	(4)
473	20 (d)	(4), interpolated
573	28 (d)	(4)
645	33 (d) peritectic	(4)
673	34 (d)	(4), interpolated
773	36 (d)	(4), interpolated
873	40 (d)	(4), interpolated
973	43 (d)	(4), interpolated
1073	47 (t)	(4,6) and (8), interpolated
1173	51 (t)	(3), (4) and (8), interpolated
1275	67 (d) congruent	(4)



References

1. Heycock, C.T.; Neville, M.A. *J. Chem. Soc.* 1889, *55*, 666.
2. Tammann, G. *Z. Phys. Chem.*, *1889*, *3*, 441.
3. Mathewson, C.H. *Intern. Z. Metallogr.*, 1911, *1*, 81.
4. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.*,1961, *310*, 143.
5. Nicoloso, N.; Schmutzler, R.W.; Hensel, F. *Ber. Bunsenges. Phys. Chem.* 1978, *82*, 621.
6. Egan, J.; Algasmi, R., Brookhaven National Laboratory, 1984, private comunication to A.D. Pelton, as reported in 7.
7. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, *7*, 136.
8. Kilic, H.H.; Pietzko, S.; Schmutzler, R.W. *J. Non-Crystall. Solids* 1990, *117-118*, 521.

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Tammann, G.

Z. Phys. Chem. 1889, *3*, 441-449.

VARIABLES:

Temperature difference: 2.24 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The melting point depression ΔT of Na due to the addition of Au was determined.

soly/g Au per 100 g Na	*soly*/mol % Au [a]	ΔT/K
0.06	$7\cdot10^{-3}$	0.03
0.24	$2.8\cdot10^{-2}$	0.12
0.37	$4.3\cdot10^{-2}$	0.20
0.81	$9.4\cdot10^{-2}$	0.41
1.46	0.170	0.76
2.20	0.256	1.10
3.06	0.356	1.48
3.83	0.445	1.96
4.25	0.494	2.24

[a] calculated by the compilers

The melting point of Na was reported to be 370 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Increasing amounts of Au were added to molten Na. The solutions were undercooled for up to 2 K and the crystal precipitation was forced by effective stirring with a thermometer. Every determination of the temperature was three times repeated.

SOURCE AND PURITY OF MATERIALS:

Au: nothing specified.
Na: "pure".

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.05 K.

REFERENCES:

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Heycock, C.T.; Neville, M.A.

J. Chem. Soc. 1889, *55*, 666-676.

VARIABLES:

Temperature: 355.1-370.0 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the Au-Na system were determined twice, and the compiler assumes that average values were reported.

t/°C	*soly*/g Au/100 g Na	*soly*/mol % Au[a]
96.85	0.1138	0.1137
96.51	0.1989	0.1985
96.07	0.2957	0.2948
95.39	0.4514	0.4494
93.09	0.9655	0.9563
91.99	1.2	1.186
91.29	1.363	1.345
89.96	1.6562	1.629
88.75	1.94	1.903
88.59	1.953	1.916
87.91	2.1122	2.068
87.75	2.161	2.115
86.5	2.390	2.334
86.14	2.534	2.471
86.07	2.5343	2.472
85.03	2.781	2.706
83.99	3.022	2.933
83.89	3.0	2.913
82.99	3.211	3.111
81.92	3.453	3.338

[a] calculated by the compilers

The melting point of Na was reported to be 97.47 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test crucible and the stirrer were made of cast Fe. A carefully calibrated thermometer was used. Solid paraffin was placed in the crucible and a weighed quantity of Na was added. The crucible was heated above the melting point of Na and finely dispersed Au was added. The temperature was then raised to the boiling point of the paraffin and allowed to slowly cool with constant stirring. The temperature of the freezing point was determined twice. A piece of the solidified Na was dissolved in absolute alcohol and the Au separated out as a fine powder.

SOURCE AND PURITY OF MATERIALS:

Au: "ignited precipitated".

Na: nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.

Temperature: precision ± 0.01 K.

REFERENCES:

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Mathewson, C.H.

Intern. Z. Metallogr. 1911, *1*, 81-88.

VARIABLES:

Temperature: 355-1262 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The points of the Au-Na liquidus were determined.

t/°C	*soly*/mol % Au	*t*/°C	*soly*/mol % Au
93	0.6	989	65.9
86	2.0	985	70.3
82	3.7	967	74.1
98	4.5	917	80.4
702	34.2	905	85.6
921	52.4	944	88.8

The melting points of Au and Na were reported to be 97.5 and 1062 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was weighed in small glass tubes, preventing the oxidation of the metal. Weighed Au and Na were placed in a larger Jena glass tube and rapidly heated to the required temperature in a H_2 atmosphere. A Fe wire was used to stir the melt. Cooling curves were recorded by means of Pt/Pt-Rh thermocouple, calibrated on the melting points of Pb, Zn, Sb, and Ag. The alloys melting above 1173 K were prepared in Berlin porcelain. The composition of the alloys with Au contents higher than 50 mol % Au was corrected by chemical analyses because of volatization of Na and side reactions. Au was determined by dissolution in aqua regia, twice evaporation with HCl, precipitation with $FeSO_4$ solution igniting and weighing; Na was obtained by difference.

SOURCE AND PURITY OF MATERIALS:

Au: "pure"
Na: "pure", did not leave a metallic residue after treatment with C_2H_5OH and H_2O.

ESTIMATED ERROR:

Solubility: accuracy of sample preparation ± 0.4 %.
Temperature: precision ± 1 K.

REFERENCES:

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Kienast, G.; Verma, J.; Klemm, W.

Z. Anorg. Chem. 1961, *310*, 143-169

VARIABLES:

Temperature: 355-1278 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the Au-Na system were determined.

t/°C	*soly*/mol % Au	t/°C	*soly*/mol % Au
94	0.61	359	31.0
85	1.28	372	33.8
82	2.02	440	34.6
83	2.83	545	38.7
90	3.48	648	42.2
108	6.37	742	44.7
110	7.73	870	50.0
122	11.2	940	54.0
140	12.8	981	60.0
163	15.3	985	61.0
220	21.6	1005	67.0
305	28.7	970	73.0

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Both metals were weighed and placed in a corund crucible inserted in a quartz tube filled with Ar (1). The corund as well as the quartz were slightly attacked by the molten alloy. The composition of the alloy was chemically analyzed. The Al content of the melt was 0.05 mass % after 60 hours ofequilibration at 1073 K. Cooling curves of the molten alloys were recorded by means of a Pt/Pt-Rh thermocouple.

SOURCE AND PURITY OF MATERIALS:

Au: nothing specified.
Na: nothing specified.
Ar: from Linde, further purified by means of reaction with Ti at higher temperature.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 2 K, according to (1), accuracy not better than ± 10 K (compilers).

REFERENCES:

(1) Dorn, F.W.; Klemm, W. *Z. Anorg. Chem.* 1961, *309*, 189-203.

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Nicoloso, N.; Schmutzler, R.W.; Hensel, F.

Ber. Bunsenges. Phys. Chem. 1978, *82*, 621-623.

VARIABLES:

One temperature : 1173 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus point of the equiatomic AuNa alloy was found to be 900 °C, as was read out from the figure by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloy was prepared by melting accurately weighed amounts of both metals in vacuo in cells made of Mo or non-porous alumina. All experimental procedures were performed in a metallic vacuum chamber of special construction.

The conductivity measurements were made using the "four probe" method by which the contact resistance errors could be eliminated.

A drop of the conductivity of the melt AuNa with decreasing temperature indicated the point on the liquidus line.

SOURCE AND PURITY OF MATERIALS:

Au: 99.999 % pure.
Na: 99.98 % pure.

ESTIMATED ERROR:

Solubility: accuracy ± 0.1 mol %.
Temperature: nothing specified; reading-out procedure ± 3 K.

REFERENCES:

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Kilic, H.H.; Pietzko, S.; Schmutzler, R.W.

J. Non-Crystall. Solids 1990, *117-118*, 521-524.

VARIABLES:

Temperature: 1081 and 1138 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubilities of Au in liquid Na at 808 and 865 °C are 47.4 and 49.9 mol % Au, respectively. The values were read from the figure by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The samples were directly mixed from weighed amounts of the metals in a Mo cup under Ar atmosphere. The cup was closed under vacuum by a thin, flexible membrane. The membrane was bent, if a difference of pressure developed between the closed cup and the surroundings. The curvature of the membrane was measured by means of the deflection of a laser beam which was reflected at the central area of the membrane.

The vapour pressure of the alloy samples was determined as a function of the temperature by adjusting the supporting gas pressure to keep the membrane in its initial position.

The break of the relationship of the Na pressure (proportional to the Na activity) vs. the reciprocal temperature indicates the saturation of Au in Na of the sample.

SOURCE AND PURITY OF MATERIALS:

Au: 99.99 % pure from Degussa.
Na: 99.9+ % pure from Ventron.
Ar: "high purity".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Gold; Au; [7440-57-5] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1991

CRITICAL EVALUATION:

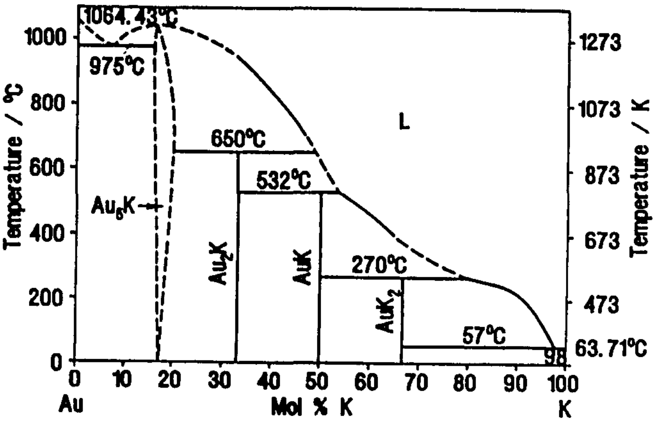
Heycock and Neville (1) observed a decrease of the melting point of K due to the addition of Au. Their result was given as an "atomic fall", i.e., the ratio of the freezing temperature decrease to the concentration of a solute. The reported "atomic fall" was 1.8 K/mol % Au. The compilers are not able to predict parameters of the eutectic point on the K-rich side of the Au-K system without any further information, though a prediction was given in (6). Brickert and Hannenbohn (2) and Kienast et al. (3) reported the co-ordinates of the eutectic point at about 3 mol % Au for 331 K and at about 2 mol % Au for 330 K, respectively. Taking into account the eutectic temperature of 331 K and the "atomic fall" from (1) one finds a composition of 3 mol % Au, confirming the result of (2) instead of (3).

The liquidus line of the Au-K system between 0.16 and 90.0 mol % Au was established by (3). One single result of (7) is in agreement with (3), while another one of (4) does not support the corresponding value obtained by (3). Moreover, some unpublished EMF experiments of (5) at 1023 K indicated significantly lower saturation concentrations of Au in K. Only doubtful data for the solubility of Au in liquid K may be suggested in this situation.

Pelton (6) presented an assessed Au-K phase diagram which is redrawn in the figure. He also expressed his doubts concerning the position of the liquidus line. The existence of Au_2K, AuK, and AuK_2 equilibrium solid phases is well documented; the fourth compound is most likely Au_6K.

Doubtful values of the solubility of Au in liquid K

T/K	*soly*/mol % Au	source
331	3 eutectic	(1,2)
373	4	(3) extrapolated
473	8	(3)
543	21 peritectic	(3)
673	34	(3) interpolated
805	45 peritectic	(3)
923	50 peritectic	(3) interpolated
1073	57	(3) and (7)
1173	61	(3) interpolated



References

1. Heycock, C.T.; Neville, F.H. *J. Chem.Soc.* 1889, *55*, 666.
2. Brickert, N.; Hannenbohn, O., private communication; quoted by Quadt, U.; Weibke, F.; Biltz, W. *Z. Anorg. Chem.* 1937, *232*, 297.
3. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.
4. Nicoloso, N.; Schmutzler, R.W.; Hensel, F. *Ber. Bunsenges. Phys. Chem.* 1978, *92*, 621.
5. Egan, J.; Algasmi, R., Brookhaven National Laboratory, private communication; quoted in 6 by A.D. Pelton.
6. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, *7*, 23.
7. Kilic, H.H.; Pietzko, S.; Schmutzler, R.W. *J. Non-Crystall. Solids* 1990, *117-118*, 521.

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Kienast, G.; Verma, J.; Klemm, W.

Z. Anorg. Chem. 1961, *310*, 143-169.

VARIABLES:

Temperature: 327-1323 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Au-K system was established.

t/°C	*soly*/mol % Au	*t*/°C	*soly*/mol % Au
59	0.18	380	33.8
58	0.86	450	38.1
(54)	1.72	510	44.1
121	4.53	640	49.2
130	5.01	720	53.3
205	7.63	800	56.8
225	9.53	860	60.0
236	10.5	920	62.6
249	14.5	940	67.0
265	18.6	(975)	79.8
270	20.5	1040	83.4
270	26.4	1050	90.0
280	30.2		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Both metals were weighed and placed in a corund crucible inserted in a quartz tube filled with Ar (1). The corund as well as the quartz were slightly attacked by the molten alloy the composition of which was determined by analysis. After 60 hours of equilibration at 1073 K, the melt contained 0.05 mass % Al. Cooling curves of the molten alloys were recorded by means of a Pt/Pt-Rh thermocouple. Some samples were analyzed after dissolution in C_2H_5OH by means of titration with an acid.

SOURCE AND PURITY OF MATERIALS:

Au: nothing specified.
K : nothing specified.
Ar: from Linde, further purified by high temperature reaction with Ti.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 2 K according to (1), accuracy not better than ± 10 K (compilers).

REFERENCES:

1. Dorn, F.W.; Klemm, W. *Z. Anorg. Chem.* 1961, *309*, 189-203.

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Nicoloso, N.; Schmutzler, R.W.; Hensel, F.

Ber. Bunsenges. Phys. Chem. 1978, *82*, 621-623.

VARIABLES:

One temperature : 803 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus point of the equiatomic AuK alloy was found to be 530 °C, as was read out from the figure by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloy was prepared by melting accurately weighed amounts of both metals in vacuo in cells made of Mo or non-porous alumina. All experimental procedures were performed in a metallic vacuum chamber of special construction. The conductivity measurements were made using the "four probe" method by which the contact resistance errors could be eliminated. A drop of the conductivity of the melt AuK with decreasing temperature indicated the point on the liquidus line.

SOURCE AND PURITY OF MATERIALS:

Au: 99.999 % pure.
K : 99.98 % pure.

ESTIMATED ERROR:

Solubility: accuracy ± 0.1 mol %.
Temperature: nothing specified; reading out procedure ± 3 K.

REFERENCES:

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Kilic, H.H.; Pietzko, S.; Schmutzler, R.W.

J. Non-Crystall. Solids 1990, *117-118*, 521-524.

VARIABLES:

One temperature : 1088 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Au in liquid K at 815 °C was found to be 59.5 mol %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The sample was prepared by direct mixing accurately weighed amounts of the metals in a Mo cup under Ar atmosphere. The cup was closed under vacuum by a thin flexible membrane. The membrane was bent off due to a pressure difference which developed between the inner of the cell and the surroundings. The curvature of the membrane was indicated by the deflection of a laser beam which was reflected at the centre of the membrane. Thus, the vapour pressure of the alloy was determined as a function of the temperature by adjusting (compensating) the supporting gas pressure to keep the membrane in the flat position.

The solubility of Au in K was indicated by the break in the curve: log of activity of K vs. reciprocal temperature.

SOURCE AND PURITY OF MATERIALS:

Au: 99.99 % pure from Degussa.
K : 99.9+ % pure from Ventron.
Ar: "high purity".

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 8 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Gold; Au; [7440-57-5] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1991

CRITICAL EVALUATION:

The liquidus line of the Au-Rb system was experimentally established by Kienast et al.(1). These determinations were not precise and temperature errors might even be ± 25 K in the Au-rich side. The results at 78.2 mol % Au and 1329 K seems to be casual. The eutectic point on the Rb-rich side was not established in (1) but calculated in (4) at 1.4 mol % Au and 307 K. Single results obtained by Nicoloso et al. (2) and by Kilic et al. (5) are in agreement with (1).

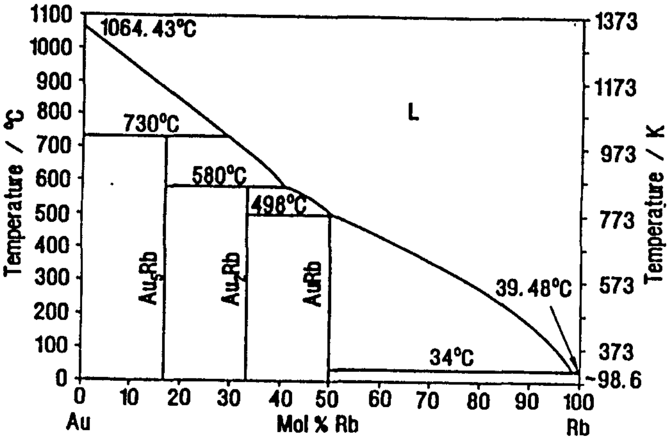
Tinelli and Holcomb (3) pointed out that the compound AuRb should have a maximum on its liquidus due to its semiconducting properties (following a general rule). This was not reflected in the data of (1).

The assessed Rb-Au phase diagram was reported by Pelton (4) and is redrawn below.

Tentative values of the solubility of Au in liquid Rb

T/K	*soly*/mol % Au	source
307	1.4 eutectic	(4) calculated
373	4	(1) extrapolated
473	13	(1) interpolated
573	22	(1) interpolated
673	34	(1) and (5) interpolated
771	49 peritectic	(1) and (2)
853	59 peritectic	(1) interpolated
973	68	(1) interpolated
1003	71 peritectic	(1) interpolated

Preliminary verification, no suggestions for the higher temperatures.



References

1. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.
2. Nicoloso, N.; Schmutzler, R.W.; Hensel, F. *Ber. Bunsenges. Phys. Chem.* 1978, *92*, 621.
3. Tinelli, G.A.; Holcomb, D.F. *J. Solid State Chem.* 1978, *25*, 157
4. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, *7*, 139.
5. Kilic, H.H.; Pietzko, S.; Schmutzler, R.W. *J. Non-Crystall. Solids* 1990, *117-118*, 521.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gold; Au; [7440-57-5] (2) Rubidium; Rb; [7440-17-7]	Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143-169.
VARIABLES:	**PREPARED BY:**
Temperature: 307-1329 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The points on the Au-Rb liquidus line were established.

t/°C	*soly*/mol % Au	*t*/°C	*soly*/mol % Au
(34)	4.0	468	45.3
135	7.6	504	49.3
170	9.5	570	55.4
215	14.2	593	61.7
285	19.9	630	62.3
336	24.0	670	66.1
390	33.1	755	71.2
425	39.8	1056	78.2
452	43.1	1026	95.0

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Both metals were weighed and placed in a corundum crucible inserted in a quartz tube filled with Ar (1). The corundum as well as the quartz were slightly attacked by the molten alloy, the composition of which was determined by analysis. After 60 hours of equilibration at 1073 K, the melt contained 0.05 mass % Al. Cooling curves of the molten alloys were recorded by means of a Pt/Pt-Rh thermocouple. Some samples were analyzed after dissolution in C_2H_5OH by means of titration with an acid.

SOURCE AND PURITY OF MATERIALS:

Au: nothing specified.
Rb: nothing specified.
Ar: from Linde, further purified by high temperature reaction with Ti.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 2 K according to (1), accuracy not better than ± 10 K (compilers).

REFERENCES:

1. Dorn, F.W.; Klemm, W. *Z. Anorg. Chem.* 1961, *309*, 189-203.

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Nicoloso, N.; Schmutzler, R.W.; Hensel, F.

Ber. Bunsenges. Phys. Chem. 1978, *82*, 621-623.

VARIABLES:

One temperature : 803 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus point of the equiatomic AuRb alloy was found to be 530 °C, as was read out from the figure by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloy was prepared by melting accurately weighed amounts of both metals in vacuo in cells made of Mo or non-porous alumina. All experimental procedures were performed in a metallic vacuum chamber of special construction. The conductivity measurements were made using the "four probe" method by which the contact resistance errors could be eliminated.

A drop of the conductivity of the melt AuK with decreasing temperature indicated the point on the liquidus line.

SOURCE AND PURITY OF MATERIALS:

Au: 99.999 % pure.
Rb: 99.98 % pure.

ESTIMATED ERROR:

Solubility: accuracy ± 0.1 mol %.
Temperature: nothing specified; reading out procedure ± 3 K.

REFERENCES:

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Kilic, H.H.; Pietzko, S.; Schmutzler, R.W.

J. Non-Crystall. Solids 1990, *117-118*, 521-524.

VARIABLES:

One temperature: 717 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Au in liquid Rb at 444 °C as read out from the figure and calculated by the compilers is 39.6 mol % Au.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The sample was prepared by direct mixing accurately weighed amounts of the metals in a Mo cup under Ar atmosphere. The cup was closed under vacuum by a thin flexible membrane. The membrane was bent off due to a pressure difference which developed between the inner of the cell and the surroundings. The curvature of the membrane was indicated by the deflection of a laser beam which was reflected at the centre of the membrane. Thus, the vapour pressure of the alloy was determined as a function of the temperature by adjusting (compensating) the supporting gas pressure to keep the membrane in the flat position.

The solubility of Au in K was indicated by the break in the curve: log of activity of K vs. reciprocal temperature.

SOURCE AND PURITY OF MATERIALS:

Au: 99.99 % pure from Degussa.
Rb: 99.9+ % pure from Ventron.
Ar: "high purity".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Gold; Au; [7440-57-5] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1991

CRITICAL EVALUATION:

Data on the Au-Cs phase diagram and particularly the liquidus line are controversial results in spite of the large amount of interest in this system in recent years. A tendency to decompose occurs due to the ionic character of the AuCs compound, and therefore it was difficult to obtain homogeneous samples of the alloys for investigations. There is no doubt that Au is comparatively easily soluble in liquid Cs since Smith et al. (1) observed a severe attack of an Au sample in liquid Cs at 573 K.

The first systematic experiments to define the Au-Cs liquidus line were performed by Kienast et al. (2) who found large solubilities of Au at low temperatures. The liquidus line was characterized by a very untypical shape from a thermodynamic point of view. Schmutzler et al. (3,4) confirmed the liquidus temperatures at 50 and 51 mol % Au. The most questionable range between 55 and 95 mol % Au still needs systematic investigation.

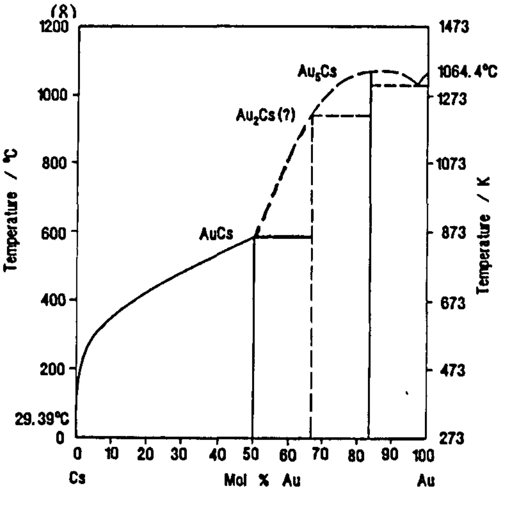
Tinelli and Holcomb (5) presented a remark that the semiconducting compound AuCs should have a maximum on the liquidus line according to a general rule. This is not observed in this case. Martin et al. (6) reported that Au-Cs alloy samples with 25 and 55 mol % Au showed crystalline X-ray reflexes at temperatures of 688 and 913 K without giving experimental details.

Eschenweck et al. (7,9) performed thermal analyses at several compositions. They observed agreement with (1) in the range 30 to 60 mol % Au and at high Au contents within 30 K. Other reliable data were obtained by Kempf (8) who supplemented his thermal analyses with vapour pressure measurements. The temperatures which were presented as results of vapour pressure experiments were not sufficiently precise, though they were in best agreement with the thermodynamic modelling of Pelton (10). The vapour pressure was more precisely measured by Kilic et al. (11), who recorded the liquidus at a 30 K higher temperature than in (8) and in agreement with (7). The data of (7), (8), and (11) are, therefore, recommended.

The saturated solution of Au in liquid Cs is in equilibrium with Au-Cs intermetallic compounds. Kempf (8) postulated the existence of a Au_2Cs phase with peritectic decomposition. The assessed tentative phase diagram was presented in (10) and is redrawn below. No solubility of Au in liquid Cs may be suggested at temperatures above 908 K, since reactions of Au-Cs alloys with the containers of Mo or corund seem to be possible.

Tentative (t) and recommended (r) values of the solubility of Au in liquid Cs

T/K	*soly*/mol % Au	source
300	0.4 (t) eutectic	(10) calculated
473	1 (t)	(8) extrapolated
573	3 (t)	(8),(11) interpolated
673	12 (t)	(11) interpolated
773	33 (t)	(7),(11) interpolated
863	50 (r) peritectic ?	(2),(3),(4),(7),(8),(9),(11)
908	55 (t)	(8)



References

1. Smith, R.G.; Hargreaves, F.; Mayo, G.T.J.; Thomas, A.G. *J. Nucl. Mater.* 1963, *10*, 191.
2. Kienast, G.; Verma, J.; Klemm, W. *Z. Anorg. Chem.* 1961, *310*, 143.
3. Schmutzler, R.W.; Hoshino, H.; Fischer, R.; Hensel, F. *Ber. Bunsenges.Phys.Chem.* 1976, *80*, 107.
4. Nicoloso, N.; Schmutzler, R.W.; Hensel, F. *Ber. Bunsenges. Phys. Chem.* 1978, *92*, 621.
5. Tinelli, G.A.; Holcomb, D.F. *J. Solid State Chem.* 1978, *25*, 157.
6. Martin, W.; Freyland, W.; Lamparter, P.; Steeb, S. *Phys. Chem. Liquids*, 1980, *10*, 61, 77.
7. Eschenweck, D. *Ph. D. Thesis*, Univ. Stuttgart, Germany, 1979.
8. Kempf, A. *Ph. D. Thesis*, Univ. Marburg, Germany, 1981.
9. Sommer, F.; Eschenweck, D.; Predel, B.; Schmutzler, R.W. *Chemical Metallurgy - A Tribute to Carl Wagner*, AIME, Warrendale, 1981, p. 19.
10. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1986, *7*, 19.
11. Kilic, H.H.; Pietzko, S.; Schmutzler, R.W. *J. Non-Crystall. Solids* 1990, *117-118*, 521.

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Kienast, G.; Verma, J.; Klemm, W.

Z. Anorg. Chem. 1961, *310*, 143-169.

VARIABLES:

Temperature : 343 - 1328 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The points on the Au-Cs liquidus line were established.

t/°C	*soly*/mol % Au	*t*/°C	*soly*/mol % Au
70	6.7	590	49.3
170	17.8	586	54.4
230	20.2	(580)	58.3
310	24.6	589	61.6
385	26.6	593	62.7
430	29.7	603	67.9
465	31.2	895	75.4
484	34.4	980	77.5
530	39.4	1053	86.0
568	44.9	1055	90.0

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Both metals were weighed and placed in a corundum crucible inserted in a quartz tube filled with Ar (1). The corundum as well as the quartz were slightly attacked by the molten alloy the composition of which was determined by analysis. After 60 hours of equilibration at 1073 K, the melt contained 0.05 mass % Al. Cooling curves of the molten alloys were recorded by means of a Pt/Pt-Rh thermocouple. Some samples were analyzed after dissolution in C_2H_5OH by means of titration with an acid.

SOURCE AND PURITY OF MATERIALS:

Au: unspecified purity, from Duisburger Kupferhütte.
Cs: nothing specified.
Ar: from Linde, further purified by high temperature reaction with Ti.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 2 K according to (1), accuracy not better than ± 10 K (compilers).

REFERENCES:

1. Dorn, F.W.; Klemm, W. *Z. Anorg. Chem.* 1961, *309*, 189-203.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gold; Au; [7440-57-5] (2) Cesium; Cs; [7440-46-2]	Nicoloso, N.; Schmutzler, R.W.; Hensel, F. *Ber. Bunsenges. Phys. Chem.* 1978, *82*, 621-623.
VARIABLES: Temperature : 853 and 863 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Two points on the liquidus of the Au-Cs system were determined, as read out from the figures by the compilers.

t/°C	*soly*/mol % Au
580	50.0
590 [a]	51

[a] as reported in (1)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloy was prepared by melting accurately weighed amounts of both metals in vacuo in cells made of Mo or non-porous alumina. All experimental procedures were performed in a metallic vacuum chamber of special construction. The conductivity measurements were made using the "four probe" method by which the contact resistance errors could be eliminated.

A drop of the conductivity of the melt Au-Cs with decreasing temperature indicated the point on the liquidus line.

SOURCE AND PURITY OF MATERIALS:

Au: 99.999 % pure.
Cs: 99.98 % pure.

ESTIMATED ERROR:

Solubility: accuracy ± 0.1 mol %.
Temperature: nothing specified; reading-out procedure ± 3 K.

REFERENCES:

1. Schmutzler, R.W.; Hoshino, H.; Fischer, R.; Hensel, F. *Ber. Bunsenges. Phys. Chem.* 1976, *80*, 107-113.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gold; Au; [7440-57-5] (2) Cesium; Cs; [7440-46-2]	Kilic, H.H.; Pietzko, S.; Schmutzler, R.W. *J. Non-Crystall. Solids* 1990, *117-118*, 521-524.
VARIABLES: Temperature: 633-863 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Au in liquid Cs was determined.

t/°C	*soly*/mol % Au	*t*/°C	*soly*/mol % Au	*t*/°C	*soly*/mol % Au
590	49.9	471	35.5	419	15.0
555	44.9	455	25.0	360	7.1

The Au-rich part of the liquidus (>80 mol % Au) was put in question.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The sample was prepared by direct mixing accurately weighed amounts of the metals in a Mo cup under Ar atmosphere. The cup was closed under vacuum by a thin, flexible membrane. The membrane was bent off due to a pressure difference which developed between the inner of the cell and the surroundings. The curvature of the membrane was indicated by the deflection of a laser beam which was reflected at the centre of the membrane. Thus, the vapour pressure of the alloy was determined as a function of the temperature by adjusting (compensating) the supporting gas pressure to keep the membrane in the flat position.

The solubility of Au in Cs was indicated by the break in the curve: log of activity of Cs vs. reciprocal temperature.

SOURCE AND PURITY OF MATERIALS:

Au: 99.99 % pure from Degussa.
Cs: 99.9+ % pure from Ventron.
Ar: "high purity".

ESTIMATED ERROR:

Nothing specified.

COMPONENTS:

(1) Gold; Au; [7440-57-5]

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

ORIGINAL MEASUREMENTS:

Eschenweck, D.

Ph. D. thesis, Univ. of Stuttgart, Germany, 1979.

VARIABLES:

Temperature: 699-1321 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus curve of the Au - Cs system were determined.

T/°C	*soly*/mol % Au
1321 [a]	95
1314 [a]	88
1300	80
1321 [a]	80
875 [b]	71.4
860	60
859	55
856	50
794	40
761	30
699	20

[a] also reported in (1)

[b] read out from figure by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by melting the two metals in an Ar atmosphere or in vacuum. The samples were heated in thin walled Mo capsules which did not react with the melts. The furnace was kept in a nearly horizontal position which allowed stirring by means of rotation of the capsules. The differential temperature changes were measured by Ni-Cr/Cr thermocouples which were calibrated on the melting points of Cu and Sb. Only cooling curves were recorded in the concentration range of less than 71.5 mol 5 Au. Cooling and heating curves were measured in the Au-rich alloys.

SOURCE AND PURITY OF MATERIALS:

Au: 99.99 % pure from Degussa.
Cs: 99.98 5 pure from Merck.
Ar: 99.99 % pure.

ESTIMATED ERROR:

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

REFERENCES:

1. Sommer, F.; Eschenweck, D.; Predel, B.; Schmutzler, R.W. *Chemical Metallurgy - a Tribute to Carl Wagner*, AIME, Warrendale, 1981, p. 19-27.

COMPONENTS: (1) Gold; Au; [7440-57-5] (2) Cesium; Cs; [7440-46-2]	**ORIGINAL MEASUREMENTS:** Kempf, A. *Ph. D. thesis*, Univ. of Marburg, Germany, 1981.
VARIABLES: Temperature: 523-1332 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus curve of the Au-Cs system were determined.

t/°C	*soly*/mol % Au	method
1062 ± 5	76.8	DTA
1068 ± 2	81.6	DTA
1069	87.4	DTA
1064 ± 1	90.7	DTA
250(-50,+30)	2.1 [a]	vapour pressure
320(-40,+30)	8.1 [a]	vapour pressure
420(±30)	25.0 [a]	vapour pressure
435(±30)	35.6 [a]	vapour pressure
450(±30)	37.4 [a]	vapour pressure
490(±30)	43.7 [a]	vapour pressure
670(0,-30)	54.7 [a]	vapour pressure
990(-40,+30)	75.1 [a]	vapour pressure

[a] the temperatures of the vapour pressure experiments were digitized from the figure, while the compositions were presented in numbers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Mo container was filled with weighed amounts of Au and Cs in Ar atmosphere of a glove box. The container was evacuated and closed with a Nb cup. It was heated above the liquidus temperature inside a furnace. The samples were homogenized for 6-7 hours by convective mixing due to the temperature gradient. The differential heating and cooling curves were recorded using Pt/Pt-Rh (10%) thermocouples, protected by corund shielding. The thermocouple was calibrated on the melting point of Au.The vapour pressure measurements were performed by Hargreaves' method in a vacuum apparatus. The cell was made of Mo, Nb, or sapphire. Condensation of Cs was visually observed via the sapphire window at a measured temperature.

SOURCE AND PURITY OF MATERIALS:

Au: 99.99 % pure from Degussa.
Cs: 99.98 % pure from Merck.
Ar: "high purity".

ESTIMATED ERROR:

Solubility: nothing specified
Temperature: precision ± 1K, accuracy < ±5 K in DTA and < ± 50 K in vapour pressure experiments.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Zinc; Zn; [7440-66-6] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1992

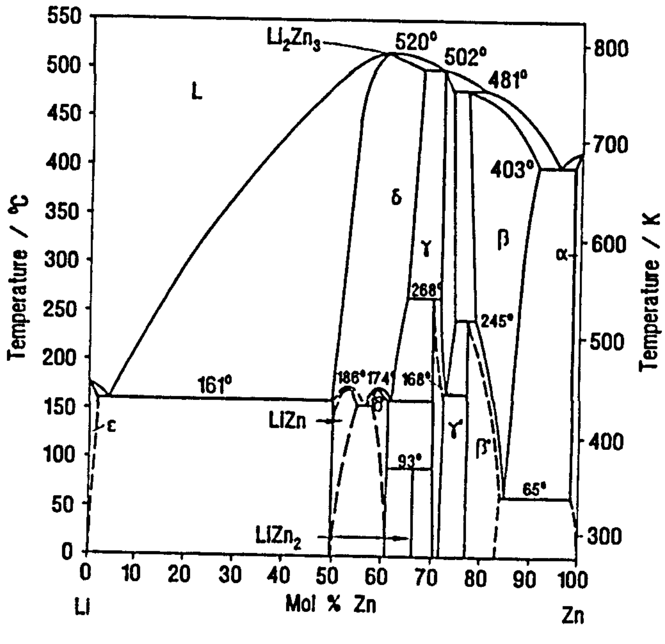
CRITICAL EVALUATION:

The Zn-rich part of the Zn-Li liquidus (with a Zn content of more than 61.6 mol %) was first determined by Fraenkel and Hahn (1). Grube and Vosskühler (2) extended the investigation to the whole range of composition. In both papers, the solubility of Zn in liquid Li was determined by thermal analysis. The results obtained by (1) and (2) for the same compositions are in agreement within few K. Weise et al. (4) found the eutectic at 96.5 mol % Zn and 676.4 K; however, they did not report experimental details.
The saturated solutions of Zn in liquid Li are in equilibrium with solid Li_2Zn_3. For higher Zn content the corresponding solid phases may be read off the figure which was redrawn from the Zn-Li phase diagram by (3).

Tentative values of the solubility of Zn in liquid Li

t/°C	*soly*/mol % Zn	source
434	4.5 (eutectic)	(2) extrapolation
473	8	(2) interpolation
573	20	(2)
673	35	(2)
773	53	(2) interpolation
793	60	(1,2)

For Zn-richer solutions see the phase diagram



References

1. Fraenkel, W.; Hahn, R.; *Metallwirtschaft* 1931, *10*, 641.
2. Grube, G.; Vosskühler, H.; *Z. Anorg. Chem.* 1933, *215*, 211.
3. Pelton, A.D. *J.Phase Equil.* 1991, *12*, 42.
4. Weise, E.; Blumenthal, A.; Hanemann, H. *Z. Metallk.* 1942, *34*, 221.

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

ORIGINAL MEASUREMENTS:

Fraenkel, W.; Hahn, R.;

Metallwirtschaft 1931, *10*, 641-642.

VARIABLES:

Temperature: 682-792 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the Zn-Li liquidus were determined.

t/°C	*soly*/mass % Li	*soly*/mol % Zn [a]
418	0.10	99.1
417	0.25	97.7
409	0.52	95.3
413	0.73	93.5
417	1.0	91.3
443	1.5	87.5
460	2.0	83.9
500	4.0	71.8
509	4.89	67.3
517	6.0	62.4
519	6.14	61.6

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A porcelain crucible and a thermoelement shielding tube were inlayed with magnesia. Zn was melted in the crucible to calibrate the thermoelement. The molten element was covered with a LiCl-Li_2CO_3 (1:1) mixture. Pieces of Li were subsequently introduced into the molten Zn by means of a Fe wire. The melt was submitted to thermal analysis after the dissolution of Li. Some samples of the alloys were chemically analyzed to confirm their compositions.

SOURCE AND PURITY OF MATERIALS:

Zn: "pure".
Li: nothing specified.

ESTIMATED ERROR:

Solubility: precision of a few %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

ORIGINAL MEASUREMENTS:

Grube, G.; Vosskühler, H.

Z. Anorg. Chem. 1933, *215*, 211-224.

VARIABLES:

Temperature: 437-793 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Zn-Li liquidus were determined.

t/°C	*soly*/mol % Zn	*t*/°C	*soly*/mol % Zn	*t*/°C	*soly*/mol % Zn
418	99	504	71	404	35
412	98	507	70	369	30
403	95	509	69	338	25
430	92	511	68	301	20
446	90	513	67	270	15
471	85	516	65	210	10
479	82	520	60	172	5
481	80	508	55	164	4
491	76	488	50	168	3
493	75	462	45	171	2
496	74	432	40	175	1

The melting points of Zn and Li were determined to be 420 and 179 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The molten alloys were prepared and studied in an Ar atmosphere. Zn-rich alloys were investigated in a graphite crucible, Li-rich alloys in an Armco Fe crucible. Cooling curves were recorded by means of a Ni/Ni-Cr thermoelement which had previously been calibrated. The thermoelement was isolated from the melt by a Fe sleeve.

SOURCE AND PURITY OF MATERIALS:

Zn: unspecified purity from Kahlbaum.
Li: 99 % pure from Metallgesellschaft A.G., containing 0.62 % K, 0.14 % Na, 0.02 % Fe_2O_3, 0.05 % SiO_2, 0.32 % Li_3N, and traces of Al_2O_3.
Ar: 98.2 % pure, containing 0.1 % O_2 and 1.7 % N_2, further purified by flowing through molten Li.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Zinc; Zn; [7440-66-6] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland December 1991

CRITICAL EVALUATION:

No dissolution of Zn in liquid Na near its melting point was observed in early work (1,2,10). Mathewson (3) performed thermal analyses in the composition range 18.9 - 98.9 mol % Zn and detected a distinct immiscibility region from almost pure Na to 93.2 mol % Zn at about 830 K. This temperature was assigned to the peritectic decomposition of the only stable compound in the Zn-Na system, $NaZn_{13}$. The solubility of Zn in liquid Na was determined by Lamprecht and Crowther (5,6) who fitted their data to the equation:

$$\log(soly/\text{mol \% Zn}) = 2.988 - 2562\ (T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

The remaining part of the studies (4,7,8,9) was devoted to the Zn-rich part of the liquidus. The agreement of the results of (3,7,8,9) is within ± 10 K and the peritectic temperature was determined as 826 ± 4 K. The eutectic point was not determined but it should be at 692 K and about 99.95 mol % Zn.

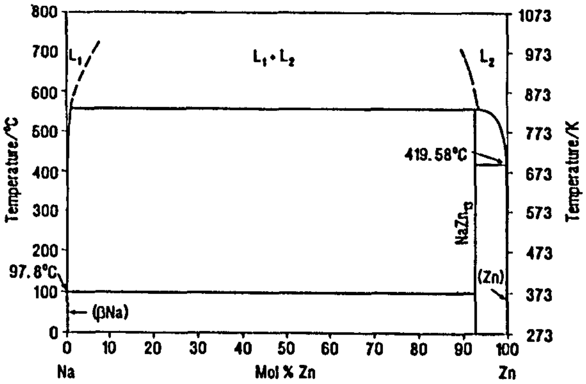
Only one point in the range of the miscibility gap was established by Lantratov et al.(4) on the Zn-rich side of the diagram. The boundaries of the liquid miscibility gap were determined by Cetin and Ross (12) by means of thermal analysis. The results agree with the data of (3,4,7,8,9) in the Zn-rich side. An extrapolation of their data to the Na-rich side reveals significant discrepancy with the data of (5,6) and the thermodynamic modelling of Pelton (11). Since the results of (5,6) appear to be based on carefully performed experiments and the criticism of (12) in respect to these results is not fully convincing, the data of (5,6) may be treated as tentative, those of (12) on the Na-rich side as doubtful. Decisive tests are needed in this range of composition. The partial Na-Zn phase diagram was reported in (11) and is redrawn below.

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

T/K	$soly$/mol % Zn	source
423	$9 \cdot 10^{-4}$	(5,6) Eq. (1)
473	$3.4\ 10^{-3}$	(5,6) Eq. (1)
573	$3.4\ 10^{-2}$	(5,6) Eq. (1)
673	0.15	(5,6) Eq. (1)
773	0.48	(5,6) Eq. (1)
826	0.8 monotectic	(5,6) Eq. (1)
973	22 (d)	(12) interpolation
1073	40 (d)	(12) interpolation

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

T/K	$soly$/mol % Na	source
692	$\leq$0.05 eutectic	(9) extrapolation
723	0.3	(9) interpolation
773	1.0	(7),(9) interpolation
823	6	(3),(7),(8) interpolation
826	7.1 peritectic	(3),(7),(8) mean value
873	7.2 (d)	(4)
973	15 (d)	(12) interpolation
1073	40 (d)	(12) interpolation



References

1. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1889, *55* , *666.*
2. Haber, F.; Sack, M. *Z. Elektrochem.* 1902, *18*, 245.
3. Mathewson, C.H. *Z. Anorg. Chem.* 1906, *48*, 191.
4. Lantratov, M.F.; Morachevskii, A.G.; Antonova, M.I. *Zh. Prikl. Khim.* 1963, *36*, 1278.
5. Lamprecht, G.J. *Ph.D. thesis*, Univ. of South Africa, Pretoria, 1966.
6. Lamprecht, G.J.; Crowther, P. *Trans. AIME* 1968, *242*, 2169.
7. Morachevskii, A.G.; Mayorova, E.A.; Vorobeva, O.I. *Elektrokhimiya* 1982, *18*, 148.
8. Häusler, W. *Z. Metallk.* 1960, *51*, 95.
9. Anderson, E.A.; Bartos, J. Argonne National Laboratory, 1962; as quoted by Johnson, I.; Dillon, I.G. *US Atom. Ener. Comm. Rep. ANL-7083*, 1965.
10. Kremann, R.; von Reininghaus, P. *Z. Metallk.* 1920, *12*, 282.
11. Pelton, A.D. *Bull.Alloy Phase Diagr.* 1987, *8*, 550.
12. Cetin, H.; Ross, R.G. *J.Phase Equil.* 1991, *12*, 6.

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

ORIGINAL MEASUREMENTS:

Mathewson, C.H.

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

VARIABLES:

Temperature: 743-826 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several temperatures on the Zn-Na liquidus line were determined.

t/°C	*soly*/mol % Zn
470-495	98.9
518-530	97.8
523-543	97.1
535-550	94.5
557	93.2
556	92.0

A miscibility gap in the Zn-Na system occurs at ~ 556 °C, additional points above this temperature were not measured. The melting point of Zn was determined as 419 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were weighed and alloyed in a high-melting Jena glass tube under H_2. The tube was slightly attacked by Na and therefore the Na content in the samples was chemically determined after the experiments. Cooling curves of the molten alloys were recorded by means of a Pt/Pt-Rh thermocouple.

SOURCE AND PURITY OF MATERIALS:

Zn: 99.9 % pure.
Na: "no other metals detected".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS: (1) Zinc; Zn; [7440-66-6] (2) Sodium; Na; [7440-23-5]	**ORIGINAL MEASUREMENTS:** Häusler, W. *Z. Metallk.* 1960, *51*, 95-100.
VARIABLES: Temperature: 803-825 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the liquidus line of the Zn-Na system were determined.

t/°C	*soly*/mass % Zn	*soly*/mol % Zn [a]
530	99.0	97.2
535	98.5	95.8
545	98.0	94.5
552	97.5	93.2

[a] calculated by the compilers

The melting points of Zn and Na were determined as 420 and 100 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Zn sample weighed and placed in a crucible mounted in a container. After adding Na into the crucible, the container was evacuated and subsequently filled with an inert gas. The closed container was heated in a furnace for 4 hours at 873-883 K and placed in a calorimeter. Cooling of the samples in the calorimeter was recorded by means of a thermometer. The liquidus line and the eutectic temperature were indicated by abrupt changes of the cooling curves.

SOURCE AND PURITY OF MATERIALS:

Zn: pure from Merck.
Na: nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

ORIGINAL MEASUREMENTS:

Anderson, E.A.; Bartos, J.

Argonne National Laboratory, 1962; as quoted by Johnson, I.; Dillon, I.G. *US Atom.Ener.Comm. Rep. ANL-7083*, 1965.

VARIABLES:

Temperature: 703-835 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Na in liquid Zn was determined at several temperatures.

t/°C	*soly*/mol % Na	*soly*/mol % Zn [a]
562	5.05	94.95
548	3.36	96.64
530	1.28	98.72
522	1.63	98.37
491	0.730	99.270
480	0.727	99.273
457	0.484	99.516
447	0.179	99.821
430	0.119	99.881

[a] calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Analyses of filtered samples were performed. Further details were not given.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

ORIGINAL MEASUREMENTS:

Lantratov, M.F.; Morachevskii, A.G.; Antonova, M.I.

Zh. Prikl. Khim. 1963, *36*, 1278-1283.

VARIABLES:

One temperature: 873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zn in liquid Na at 600 °C was determined to be 92.7 mol % Zn as estimated from the figure by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Precisely weighed amounts of the metals were melted in a glass cell in an Ar atmosphere. The emf of the cell:

Na / Na^+ (in Pyrex glass with 3.5 mass % Na_2O) / Na-Zn

was measured at the given temperature. A break on the logarithm of Na activity versus alloy composition corresponds to the solubility of Zn in Na.

SOURCE AND PURITY OF MATERIALS:

Zn: "analytically pure with no As".

Na: "practically with no metallic impurities", filtered subsequently through a capillary.

Ar: purified by means of K-Na melt.

ESTIMATED ERROR:

Solubility: precision of emf measurement ± 2 mV; precicion of estimation 0.3 mol %.

Temperature: precision ± 1 K.

REFERENCES:

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

ORIGINAL MEASUREMENTS:

Lamprecht, G.J.; Crowther, P.

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

VARIABLES:

Temperature: 454.5-816.4 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Zn in liquid Na was determined at several temperatures.

t/°C	*soly*/mass % Zn	*soly*/mol % Zn
181.3 [c]	$6.75\cdot10^{-3}$	$2.4\cdot10^{-3}$
197.3 [a]	$9.78\cdot10^{-3}$	$3.4\cdot10^{-3}$
246.3 [a]	$3.27\cdot10^{-2}$	$1.15\cdot10^{-2}$
260.0 [c]	$4.4\cdot10^{-2}$	$1.55\cdot10^{-2}$
352.0 [b]	0.254	$8.6\cdot10^{-2}$
421.5 [a]	0.566	0.20
491.2 [b]	1.24	0.44
543.2 [a]	2.00	0.72

[a] heating sequence [b] cooling sequence [c] from intermetallic compound

The solubility equation was presented by the authors and confirmed by the compilers

$$\log(soly/\text{mol \% Zn}) = 2.998 - 2562\ (T/K)^{-1}$$

The results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility apparatus was fabricated of Pyrex glass (1,2). The Zn samples contained radioactive ^{65}Zn obtained by radiation and were melted under He. Zn was placed in one reaction cell and Na in a twin one. The cells were connected by a capillary. Molten Na was transferred to the cell by means of pressure. The metals were in contact until the activity of Zn in liquid Na became constant. The solution was subsequently transferred to the primary cell for decay measurements. The variation of the solubility with temperature was determined by observing increases or decreases of the activity of saturated Na.

SOURCE AND PURITY OF MATERIALS:

Zn: spectroscopically pure from Johnson Matthey Co.
Na: filtered, from Merck containing 1.1.10-3 % O.
He: purified by means of molecular sieves, activated charcoal trapping at the temperature of liquid N.

ESTIMATED ERROR:

Solubility: standard deviation ± 4 %.
Temperature: nothing specified.

REFERENCES:

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

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

ORIGINAL MEASUREMENTS:

Morachevskii, A.G.; Mayorova, E.A.; Vorobeva, O.I.

Elektrokhimiya 1982, *18*, 148-151.

VARIABLES:

Temperature: 753-822 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Zn-Na system were determined.

T/K	*soly*/mol % Na	*soly*/mol % Zn [a]
753	0.53	99.47
755	0.90	99.1
785	1.2	98.8
791	1.9	98.1
800	2.1	97.9
810	2.4	97.6
819	3.6	96.4
817	4.2	95.8
821	5.5	94.5
825	6.3	93.7
822	7.3	92.7

[a] calculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The method applied to prepare the alloys was not described. The emf of the cell:
Na / Na^+ (in Al-Si glass with 33 mol % Na_2O) / Na-Zn
was measured at various temperatures. A break of the potential versus logarithm of the Na concentration corresponds to the solubility of Zn in Na. The Na content in the alloys was determined by flame photometry.

SOURCE AND PURITY OF MATERIALS:

Zn: analytically pure.
Na: "practically without metallic impurities".

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.1 mol %.
Temperature: read-out procedure ± 1 K.

REFERENCES:

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

ORIGINAL MEASUREMENTS:

Cetin, H.; Ross, R.G.

J. Phase Eqil. 1991, *12*, 6-9.

VARIABLES:

Temperature: 815-1090 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Zn-Na liquidus curve were determined.

t/°C	*soly*/mol % Zn
542	10
675	20
769	30
800	40
817	50
803	60
782	70
749	80
661	90

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared from precisely weighed amounts of the metalss in a glove box under flowing Ar. The metals were placed in Mo-W(30%) containers. A Ta disc was inserted between the lid and the conatiner, the lid was then tightened using 6 Ta screws. The container was placed in an outer chamber made of steel. The whole chamber was positioned in the hot zone of a furnace. The sealed system was flushed with Ar at room temperature for 30 min. and heated up to 1173 K. The furnace was left to cool. A Chromel/Alumel thermocouple which was calibrated on a Pt/Pt-Rh(13%) element served for temperature measurement.

SOURCE AND PURITY OF MATERIALS:

Zn: 99.9 % pure.
Na: 99.95 % pure.
Ar: Purified by BOX rare gas purifier.

ESTIMATED ERROR:

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

REFERENCES:

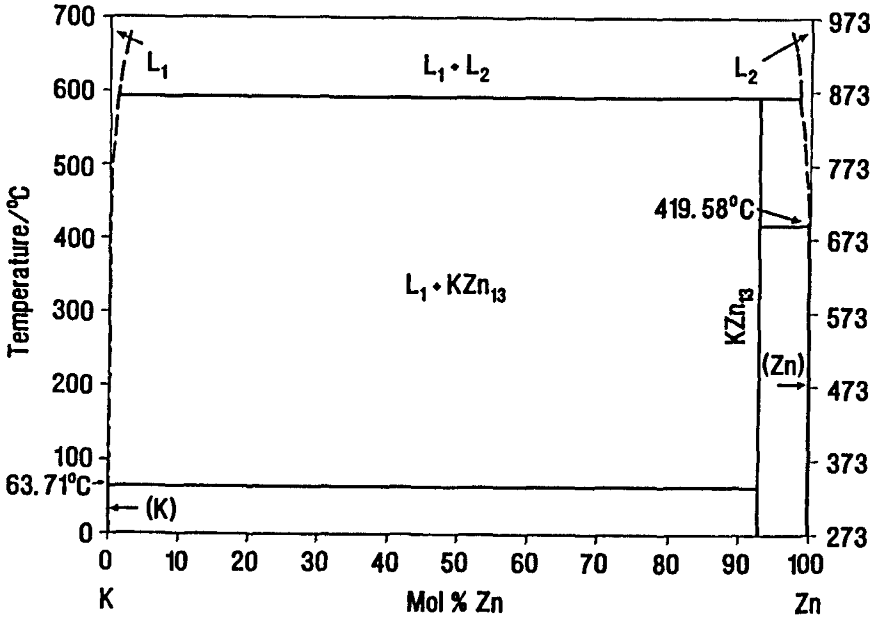
COMPONENTS:	EVALUATOR:
(1) Zinc; Zn; [7440-66-6] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland December 1989

CRITICAL EVALUATION:

Thermal analyses of Zn-K alloys in the composition range 2.0-98.0 mol % were performed by Smith (1), who observed a thermal arrest at 859±3 K for all alloys between 3.0 and 98.0 mol % Zn, indicating a miscibility gap of the metals. However, only the value of 2.0 mol % at 841 K represents a solubility value, the precision being rather poor.
Formation of the intermetallic compound KZn_{13}, stable up to about 859 K, was reported in (2). The partial phase diagram from (3) is redrawn below.

Doubtful values of the solubility of Zn in liquid K according to (1)

T/K	*soly*/mol % Zn
841	2
859	≤ 3



References

1. Smith, D.P. *Z. Anorg. Chem.* 1908, *56*, 109.
2. Zintl, E.; Haucke, W. *Z: Elektrochem.* 1938, *44*, 104.
3. Pelton, A.D. *Bull.Alloy Phase Diagr.* 1987, *8*, 548.

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

ORIGINAL MEASUREMENTS:

Smith, D.P.

Z. Anorg. Chem. 1908, *56*, 109-142.

VARIABLES:

Temperature: 841-859 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the Zn-K liquidus line were determined.

t/°C	*soly*/mol % K	*soly*/mol % Zn [a]
586	2.0	98.0
582	97.0	3.0
568	98.0	2.0

a) calculated by the compilers.

The concentration range 3-98 mol % Zn is situated in the miscibility gap.
The melting points of Zn and K were determined as 419 and 63.6 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The oxidized surface of a piece of K was cut off, the sample was subsequently washed in benzene, amyl alcohol, petroleum, and ether. The metals were placed in a tube of hard Jena glass after weighing (K in petroleum medium). The metals were melted in the tube, which had been filled with H_2. The tube containing the molten metals was placed in a thermal analysis apparatus. Cooling curves were recorded by means of a calibrated Pt/Pt-Rh thermocouple. The alloys were stirred with a glass rod during solidification.

SOURCE AND PURITY OF MATERIALS:

Zn: nothing specified.
K: containing "no traces" of Na as well as other metals.

ESTIMATED ERROR:

Nothing specified.
Temperature: precision not better than ± 3 K (compilers).

REFERENCES:

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

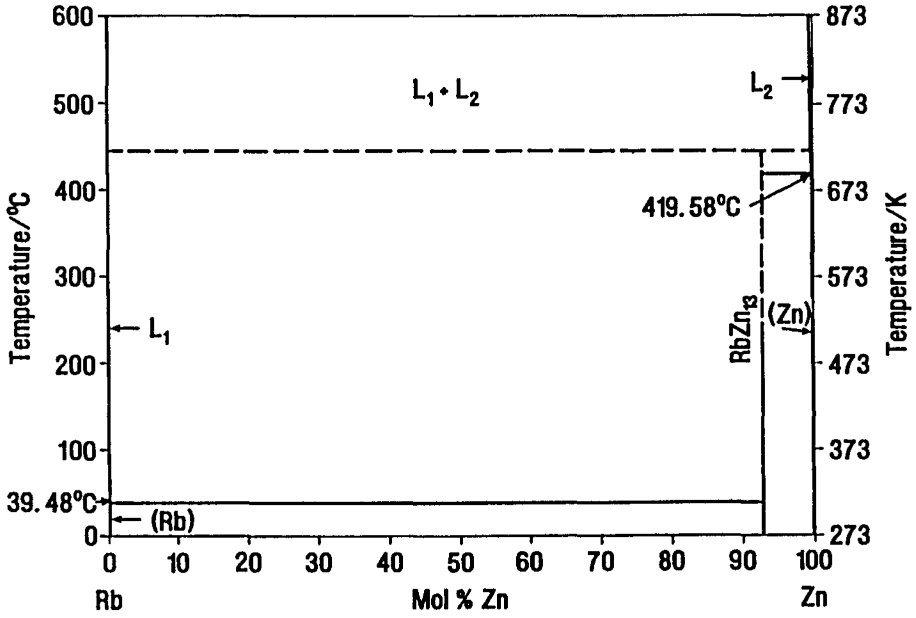
EVALUATOR:

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

CRITICAL EVALUATION:

Solubility determinations of Zn in liquid Rb were not reported in the literature. Pelton (1) estimated the solubility of Zn in liquid Rb on the basis of a comparison with the Zn-Na and Zn-K systems to be less than 0.1 mol % Zn, and the solubility of Rb in liquid Zn to be less than 0.5 mol % Rb at the temperature of the miscibility gap.

The temperature stability of intermetallic $Zn_{13}Rb$ (2) has not been reported, therefore it is impossible to specify the corresponding solid phases which are in equilibrium with the saturated solution of Zn in liquid Rb. A schematic phase diagram of the Zn-Rb system was published in (1) and is similar to that of the Zn-K system.



References

1. Pelton, A.D. *Bull. Alloy Phase Diagr.* 1987, *8*, 485.
2. Bruzzone, G. *Acta Crystall.* 1971, *B27*, 862.

COMPONENTS:

(1) Zinc; Zn; [7440-66-6]

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

EVALUATOR:

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

CRITICAL EVALUATION:

Solubility determinations of Zn in liquid Cs are not known. It seems, however, that the solubility values at moderately high temperatures are lower than those in liquid K or Rb. Bruzzone (1) reported the existence of an intermetallic phase Cs-Zn, which is not analogous to $NaZn_{13}$, KZn_{13}, and $RbZn_{13}$. The stability range of this Cs-Zn phase was not determined. The data are insufficient to predict a phase diagram of the Cs-Zn system, which should be similar to the Rb-Zn system.

References

1. Bruzzone, G. *Acta Crystall.* 1971, *B27*, 862.

COMPONENTS:	EVALUATOR:
(1) Cadmium; Cd; [7440-43-9] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland December 1989

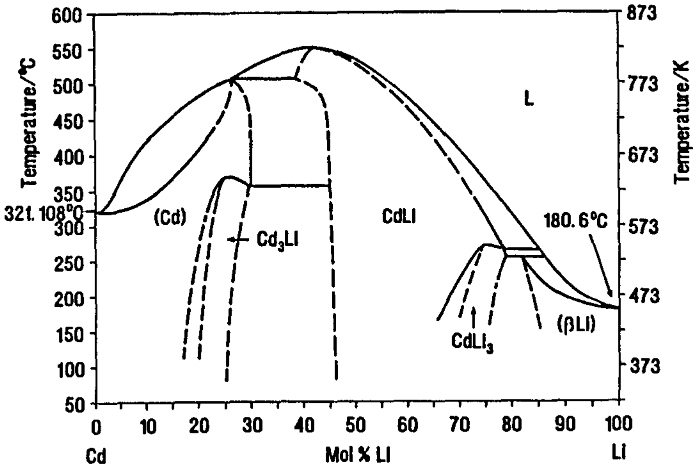
CRITICAL EVALUATION:

The melting point of an alloy formed by addition of Cd to Li was higher than that of Li. Although thermal analysis experiments by Masing and Tammann (1) and Grube et al. (2) were in fair agreement (within the experimental errors) concerning the Cd-Li liquidus, van der Marel et al.(3) and Langen et al. (9) demonstrated by means of resistivity, differential scanning calorimetry and potentiometry that the course of the liquidus in the range of 30 to 85 mol % Cd is shifted to the Cd-rich side of the diagram. Schneider and Heymer (4) also observed a distinct premelting effect below the melting point of the CdLi alloy which could be better explained by a modified phase diagram based on (3) than on the older data (1,2). Chemical analyses of the intermediate phases were not performed by (1,2,3,9). According to (3) and (9) a congruent melting point is observed at about 59 mol % Cd (instead of 50 mol % Cd), thus suggesting formation of Cd_3Li_2 rather than CdLi. The Cd_3Li_2 phase was identified neither in the earlier studies (1,2) nor in the later by (4,5,6). The existence of CdLi at lower temperatures and of Cd_3Li_2 at higher would explain the existing discrepancies, as stated by Pelton (8). Extensive reinvestigation of the Cd-Li system is necessary to present a consistent Cd-Li phase diagram in the low temperature range. A conciliatory Cd-Li phase diagram was published in (7) and (8). It is used as the basis for the figure in which the liquidus was modified according to (3) and (9).

Tentative and doubtful (d) solubility values of Cd in liquid Li

T/K	*soly*/mol % Cd	source
473	6 (d)	(2)
539	14 (d) peritectic	(2)
573	18 (d)	(2)
673	30 (d)	(2),(3) mean value of extrapolations
773	43	(3,9) interpolated
818	59	(3,9) mean value

Above 818 K the metals are miscible in every ratio. For Cd richer alloys, see the phase diagram (7).



References

1. Masing, G.; Tammann, G. *Z. Anorg. Chem.* 1910, *67*, 183.
2. Grube, G.; Vossköhler, H.; Vogt, H. *Z. Elektrochem.* 1932, *38*, 869.
3. van der Marel, C.; Vinke, G.J.B.; Hennephof, J.; van der Lugt, W. *J. Phys. Chem. Solids* 1982, *43*, 1013.
4. Schneider, A.; Heymer, G. *Z. Anorg. Chem.* 1956, *286*, 118.
5. Zintl, E.; Schneider, A. *Z. Elektrochem.* 1934, *40*, 107.
6. Baroni, A. *Z. Elektrochem.* 1934, *40*, 565.
7. Drits, M.E.; Zusman, L.L. *Splavy Shchelochnykh i Shchelochnozemlenykh Metallov*, Metallurgiya, Moskva, 1986, p.75.
8. Pelton, A.D. *Bull.Alloy Phase Diagr.* 1988, *9*, 36.
9. Langen, G.; Schwitzgebel, G.; Ruppersberg. H. *Z. Metallk.* 1983, *74*, 425.

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

Masing, G.; Tammann, G.

Z. Anorg. Chem. 1910, *67*, 183-199.

VARIABLES:

Temperature: 523-813 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Cd-Li liquidus line were determined.

t/°C	*soly*/mol % Cd
250	13.2
325	22.1
463	33.3
525	41.0
541	49.3
536	58.8
521	63.6
505	67.7
482	75.4
453	86.9
392	92.3

The melting points of Cd and Li were determined as 321 and 179 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys containing more than 60 mol % Cd were prepared in a glass tube, those with less than 60 mol % in a Fe container. The metals were weighed (Li under petroleum), placed in the containers and homogenized at approximately 823 K after heating in a H_2 atmosphere. Thermal analysis of the melts was performed by means of a thermoelement.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Solubility: precision ± 0.5 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Cadmium; Cd; [7440-43-9] (2) Lithium; Li; [7439-93-2]	Grube, G.; Vosskühler, H.; Vogt, H. *Z. Elektrochem.* 1932, *38*, 869-880.
VARIABLES:	**PREPARED BY:**
Temperature: 469-822 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Cd-Li system were determined.

t/°C	*soly*/mol % Cd	*t*/°C	*soly*/mol % Cd	*t*/°C	*soly*/mol % Cd
196	5.0	416	30.0	505	72.5
206	7.0	474	35.0	493	75.0
226	9.0	512	40.0	473	80.0
245	12.0	528	45.0	447	85.0
261	14.0	549	50.0	435	87.5
281	16.0	546	55.0	418	90.0
305	18.0	534	60.0	386	92.5
313	20.0	527	62.5	359	95.0
325	21.0	522	65.0	330	97.5
348	23.0	517	66.7		
367	25.0	512	70.0		

The melting points of Cd and Li were determined as 321 and 179 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The crucible and the shielding of the thermoelement were fabricated of Armco Fe. The crucible was filled with the metals in an Ar atmosphere and melted. Cooling curves of the melt were recorded by means of a Ni/Ni-Cr thermoelement which had previously been calibrated on the melting points of Sn, Cd, Zn, and Mg.	Cd: unspecified purity from Kahlbaum. Li: 99.0 % pure from Metallgesellschaft A.G., containing 0.62 % K, 0.14 % Na, 0.02 % Fe_2O_3, 0.05 % SiO_2, 0.32 % Li_3N and traces of Al_2O_3. Ar: 98.2 % pure with 0.1 % O_2 and 1.7 % N_2, further purified by flowing through molten Li.
	ESTIMATED ERROR:
	Nothing specified.
	REFERENCES:

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

van der Marel, C.; Vinke, G.J.B.; Hennephof, J.; van der Lugt, W.
J. Phys. Chem. Solids 1982, *43*, 1013-1014.

VARIABLES:

Temperature: 610-821 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Various points on the Cd-Li liquidus line were determined. The values were deduced from the diagram by the compilers.

t/°C	*soly*/mol % Cd	*t*/°C	*soly*/mol % Cd	*t*/°C	*soly*/mol % Cd
429 [a]	35.3	545 [a]	60.0	487 [b]	80.0
455 [a]	40.2	548 [b]	60.0	462 [a]	85.0
460 [b]	40.2	542 [a]	62.7	439 [a]	87.6
492 [a]	43.8	527 [a]	66.8	421 [b]	89.9
513 [a]	45.9	519 [a]	69.2	412 [a]	91.5
526 [a]	49.7	509 [a]	71.2	337 [a]	96.6
528 [b]	49.7	513 [b]	71.2		
527 [a]	51.2	502 [a]	72.8		
536 [a]	52.8	501 [a]	74.8		
543 [a]	56.0	491 [a]	80.0		

[a] resistivity measurement [b] differential scanning calorimetric measurement

The melting point of Cd was determined as 320 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a He-filled glove box. The metals were melted together in a crucible of (AISI 321) stainless steel and quenched on a Cu plate. The solid alloy was crushed. The resistivity as a function of temperature was measured in an apparatus of W and Al_2O_3. The recording was started at a temperature of about 15 K above the presumed liquidus, the temperature being then gradually reduced at intervals of 1 K. The slope of the resistivity as a function of temperature showed a sharp change at the liquidus point. The temperature was contolled by means of a Pt/Pt-Rh(10%) thermocouple. The sample was heated in an apparatus to approximately 873 K to perform differential scanning calorimetry and was kept at this temperature for 15 minutes. The temperature was subsequently reduced and the heat flow from the filled sample holder to its environment continuously measured. The measurements were repeated several times. Both types of experiments were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Cd: 99.99 % pure from Alfa Ventron; surface of the molten metal was skimmed.
Li: 99.9 % pure from Koch-Light Inc.; internal fraction of the molten metal was applied to avoid O and N contamination.
He: "high" purity.

ESTIMATED ERROR:

Solubility: nothing specified; read-out precision ± 0.5 mol %.
Temperature: precision ± 1 K.

REFERENCES:

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

Langen, G.; Schwitzgebel, G.; Ruppersberg. H.

Z. Metallk. 1983, *74*, 425-429.

VARIABLES:

Temperature: 774-815 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Cd-Li liquidus line were characterized by means of experiments.

t/°C	*soly*/mol %Cd	t/°C	*soly*/mol %Cd
501	72.7	542	58.0
515	70.5	540 [a]	56.5
529	67.0	529	50.1
541 [a]	62.0	515	47.2
542	60.0	501	45.0

[a] read out from the figure by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by melting the metals in an Ar atmosphere. Their composition was also varied by means of a coulometric titration. The quenched alloys were analyzed by unspecified methods. The EMF of the cell:

Li/LiF-LiCl/Cd-Li

was measured at selected temperatures. The cell was assembled in a glove box. The crucible for liquid Li was of Al_2O_3, those for the Cd-Li alloys and the cell were of Fe. Li caused a surface reduction of Al_2O_3. The electrodes were connected with Mo leads which also served for stirring of the melts. The temperature was measured using a Mo/Ni thermocouple. The solubilities were determined from breaking points of the relations of potentials vs. composition.

SOURCE AND PURITY OF MATERIALS:

Cd: 99.95 % pure from Riedel de Haen.
Li: 99.9 % pure from Koch-Light.
LiF-LiCl: "suprapure" from Merck, dry Cl_2 was bubbled through the melt for 3 hours, He gas was used for flushing.
He: "dry".
Ar: purified, contents of O, N, and $H_2O < 1 \cdot 10^{-4}\%$.
Fe: "high" purity.

ESTIMATED ERROR:

Solubility. nothing specified; precision ± 0.1 mol % (by the compilers).
Temperature: precision ± 2 K, stability ± 0.5 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Cadmium; Cd; [7440-43-9] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1990

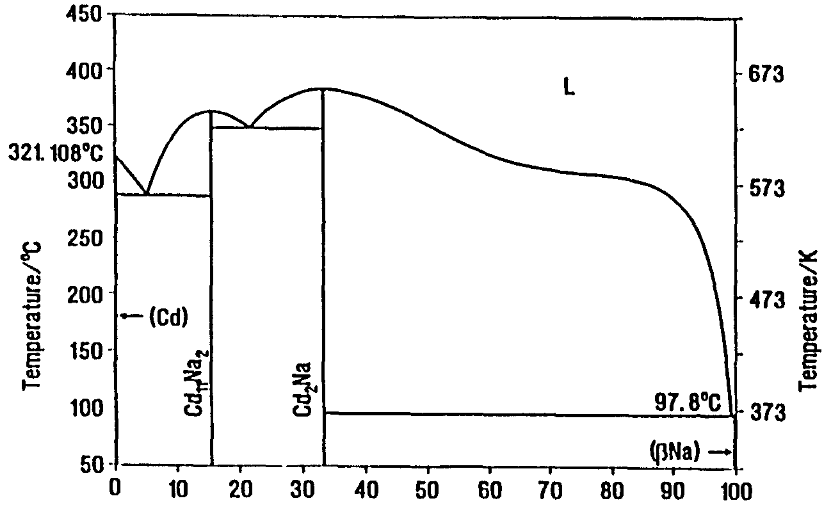
CRITICAL EVALUATION:

The liquidus of the Cd-Na system has been intensively investigated. The results, however, are only in partial agreement. The depressions of the melting point of Na due to an addition of small amounts of Cd which was observed by Heycock and Neville (1) and Tammann (2) are in fair agreement. The estimated eutectic temperature also agreed in different studies (1,2,5,11), whereas the eutectic composition varied between 0.61 and 0.79 mol % Cd. Solubility data in (4,5,6) by thermal analysis were about two times higher than those obtained by chemical analysis of the saturated solutions. The results obtained by Weeks and Davies (11) are more convincing. However, the formulation of a simple solubility equation cannot be recommended, since the dependence of the logarithm of the solubility on the reciprocal temperature is not linear.
The liquidus data of Kurnakov and Kuznetsov (5) and Mathewson (6) for alloys with a higher concentration of Cd were in fair agreement. Earlier results of (4) are regarded as preliminary to (5). The most important difference between (5) and (6) is the existence of a miscibility gap in the Na-rich side indicated in (6). Mathewson (6) determined an immiscibility range from 30 to 40 mol % Cd at 594 K. Hauffe (7) even observed such a gap at 668 K. Thermodynamic investigations of Alabyshev and Morachevskii (10) at higher temperatures also indicated a tendency of demixing in the Na-rich liquid alloys of this system. Pelton (13) calculated on the basis of thermodynamic data for this system that a metastable miscibility gap occurs 50 K below the assessed liquidus line (see phase diagram). Allaria (8) confirmed the liquidus line between 67.4 and 99.0 mol % Cd within a few K, Neethling (14) did the same for the range 85 to 95 mol % Cd. Only one result of Takeda and Tamaki (15) - 65 mol % Cd at 654 K - confirmed the earlier results. The disagreement between the results of (2) and (5) for Cd-rich alloys is certainly higher than the experimental errors of the used methods.
The formation of a Cd_2Na (precisely $Cd_{1.92}Na$) compound cannot be doubted, the composition of the second compound, which is in equilibrium with the liquid phase, being differently reported as Cd_6Na (4,5,8), Cd_5Na (6,8), or Cd_4Na. Finally, Kornenko and Zhuravlev (12) established the formula as $Cd_{11}Na_2$ based on crystallographic analysis. The Cd-Na phase diagram was assessed in (13), and is shown in the redrawn figure.

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

T/K	*soly*/mol % Cd	source
368.6	0.7 (t) eutectic	(1,3,5) mean value
473	2.4 (t)	(11)
573	12 (t)	(11)
623	50 (d)	(5,6)
657	66 (t) congruent	(5,6,15)

For Cd-rich liquid alloys see the liquidus on the figure.



COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
January 1990

CRITICAL EVALUATION:

References

1. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1889, *55*, 666.
2. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1892, *61*, 888.
3. Tammann, G. *Z. Anorg. Chem.* 1889, *3*, 441.
4. Kurnakov, N.S. *Zh.Russ.Fiz.-Khim.Obshch.* 1899, *31*, 927; *Z.Anorg.Chem.* 1900, *23*, 439.
5. Kurnakov, N.S.; Kuznetsov, A.N. *Zh.Russ.Fiz.-Khim.Obshch.* 1906, *38*, 809; *Z.Anorg.Chem.* 1907, *52*, 173.
6. Mathewson, C.H. *Z. Anorg. Chem.* 1906, *50*, 171.
7. Hauffe, K. *Z. Elektrochem.* 1940, *46*, 348.
8. Allaria, S. *Atti Reale Accad. Sci. Torino Cl.Sci.Fis.Mat.Nat.* 1942-43, *78*, 143.
9. Jänecke, E. *Z. Metallk.* 1928, *20*, 113.
10. Alabyshev, A.F.; Morachevskii, A.G. *Dokl. Akad. Nauk. SSSR* 1956, *111*, 369.
11. Weeks, J.R.; Davies, H.A. *The Alkali Metals*, The Chem. Soc., London, 1967, p. 32; *US Atom.Ener.Comm. Rep. BNL-10372*, 1964; *US Atom.Ener.Comm. Rep. CONF-660712-1*, 1966.
12. Kornenko, V.N.; Zhuravlev, N.N. *Kristallografiya* 1972, *17*, 863.
13. Pelton, A.D. *Bull.Alloy Phase Diagr.* 1988, *9*, 41.
14. Neethling, A.J. *South Afric.Atom.Ener.Board Rep. PEL-230*, 1974.
15. Takeda, S.; Tamaki, S. *J. Phys. Soc. Jap.* 1989, *58*, 1484.

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

Heycock, C.T.; Neville, F.H.

J. Chem. Soc. 1889, *55*, 666-676.

VARIABLES:

Temperatures: 368.6-370.6 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The depression of the melting point of Na after an addition of Cd was investigated.

t/°C	*soly*/atoms Cd per 100 atoms Na	*soly*/mol % Cd [a]
97.11	0.0969	0.0968
96.75	0.1964	0.196
96.42	0.288	0.287
96.04	0.3908	0.389
95.83	0.4681	0.466
95.54	0.5441	0.541
95.43 eutectic	0.6475	0.643

[a] calculated by the compilers

The melting point of Na was determined as 97.49 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test crucible was made of cast Fe. It contained holes for a Fe stirrer and a carefully scaled Hg thermometer, which could be moved along the crucible axis. Solid paraffin was placed in the crucible and a weighed quantity of Na was dropped in. The crucible was heated to 533 K and Cd was added. The alloy was then slowly cooled under constant stirring. Each temperature of the alloy freezing point was determined twice.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.01 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Cadmium; Cd; [7440-43-9] (2) Sodium; Na; [7440-23-5]	Tammann, G. *Z. Phys. Chem.* 1889, *3*, 441-449.
VARIABLES:	**PREPARED BY:**
Temperature difference: 1.81 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The depression of the melting point of Na after an addition of Cd was determined.

ΔT/K	*soly*/g Cd per 100 g Na	*soly*/mol % Cd [a]
0.04	0.11	0.022
0.14	0.21	0.043
0.21	0.36	0.074
0.43	0.64	0.131
0.58	0.91	0.186
1.11	1.57	0.320
1.49	2.16	0.440
1.74	3.06	0.622
1.81	3.90	0.792

[a] calculated by the compilers

The melting point of Na was reported to be 370 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Portions of Cd were successively added to molten Na. The resulting solutions were undercooled up to 2 K. Crystal precipitation was forced by effective stirring with a thermometer. Each temperature determination by means of the thermometer was repeated three times.

SOURCE AND PURITY OF MATERIALS:

Cd: nothing specified.
Na: "pure".

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.05 K.

REFERENCES:

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

Heycock, C.T.; Neville, F.H.

J. Chem. Soc. 1892, *61*, 888-914.

VARIABLES:

Temperature: 591.0-587.9 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The depression of the melting point of Cd after an addition of Na was investigated.

t/°C	*soly*/atoms Na per 100 atoms Cd	*soly*/mol % Cd [a]
317.82	0.577	99.43
315.76	1.017	98.99
314.72	1.27	98.75

a) calculated by the compilers

The melting point of Cd was determined as 320.41 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Weighed quantities of the metals were placed in a hard glass tube, which was subsequently exhausted and sealed. The tube was heated to red heat. The metals were mixed by shaking. These alloys were added to the bulk of a Cd block. The test crucible was fabricated of cast Fe. The molten alloy was slowly cooled under constant stirring. The freezing temperatures were determined by means of carefully calibrated thermometers.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.

Temperature: precision not better than ± 0.01 K.

REFERENCES:

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

Kurnakov, N.S.; Kuznetsov, A.N.

Zh. Russ. Fiz.-Khim. Obshch. 1906, *38*, 809-820; *Z. Anorg.Chem.* 1907, *52*, 175-185.

VARIABLES:

Temperature: 368.6-657.2 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points of the Cd-Na liquidus line were determined.

t/°C	*soly*/mol % Cd	t/°C	*soly*/mol % Cd	t/°C	*soly*/mol % Cd
95.4	0.74 eutectic	351.0	80.79	343	89.91
114[a]	1.74	356.0	81.79	341	90.23
180[a]	4.17	358.4	82.91	339	90.57
242[a]	8.12	359.0	83.05	334	91.14
321	34.08	362.5	83.99	327	91.57
343	48.60	362.8	84.64	325	91.97
350.5	51.29	363.0	85.27	319	92.16
358	55.16	363.5	85.47	316.5	92.40
366	55.75	363.5	85.75	313.5	92.69
374	58.79	362.2	86.37	308.5	93.04
379	62.02	362.8	86.68	304.5	93.21
383.0	64.33	361.5	86.81	297.5	93.89
384	69.79	360.0	87.61	291	94.50
382.0	70.25	357	87.90	301.5	95.31
379.0	72.06	356	88.01	307.5	96.50
374	73.31	351	88.61	313	97.80
365	76.10	350.5	88.97	318.5	98.83
362	77.21	348	89.32		
353.5	79.18	346.5	89.56		

[a] also reported in (1)

The melting points of Cd and Na were determined as 321 and 97.5 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a Fe crucible by dissolving weighed amounts of Cd into Na under paraffin. The Cd concentration of the alloys was analysed by CdS precipitation. Na was determined as Na_2SO_4. The freezing points of the molten alloys were measured by means of a Hg thermometer or a pyrometer calibrated on the boiling point of H_2O and the melting points of Sn, Pb, and Zn.

SOURCE AND PURITY OF MATERIALS:

Both metals from Kahlbaum; purity unspecified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 1.0 K.

REFERENCES:

(1) Kurnakov, N.S. *Zh.Russ.Fiz.-Khim.Obshch.* 1899, *31*, 927-948; *Z. Anorg.Chem.* 1900, *23*, 439-462.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Cadmium; Cd; [7440-43-9] (2) Sodium; Na; [7440-23-5]	Mathewson, C.H. *Z. Anorg. Chem.* 1906, *50*, 171-198.
VARIABLES:	**PREPARED BY:**
Temperature: 561-658 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points of the Cd-Na liquidus were determined.

t/°C	*soly*/mol % Cd	*t*/°C	*soly*/mol % Cd
288	15.0	378	73.0
318	25.0	366	76.0
329	30.0	346	79.0
329	34.5	350	80.0
331	38.0	358	83.0
342	45.0	360	83.8
354	50.0	365	85.8
370	57.0	352	87.0
382	64.0	307	92.5
385	66.7	296	95.0
383	70.0		

The melting points of Cd and Na were determined to be 322 and 97.5 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by direct melting of weighed amounts of the metals in a H_2 atmosphere. The melting was performed in a container made of high-melting Jena glass. Cooling curves of the molten alloys were recorded by means of a Pt/Pt-Rh thermocouple.

SOURCE AND PURITY OF MATERIALS:

Cd: "pure".
Na: "pure".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

Hauffe, K.

Z. Elektrochem. 1940, *46*, 348-356.

VARIABLES:

One temperature: 668 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility gap of the two liquids at 395 °C was determined to be in the range of 32 to 37 mol % Cd.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Electrochemical Cd was remelted in a H_2 atmosphere. Na was weighed in glass capillaries. Both metals were melted in a furnace and kept at a constant temperature in a H_2 atmosphere. The EMF of the cell:
Fe / Na / Na^+ (in Thüringen glass) / Na-Cd / Fe
was measured. The limits of the solubility gap were indicated by the independence of the EMF on the composition within the above mentioned range.

SOURCE AND PURITY OF MATERIALS:

Cd: chemically pure from Merck, further reelectrolysed in a $CdSO_4$ solution.
Na: analytically pure from Merck, filtrated through a glass capillary.
H: nothing specified.

ESTIMATED ERROR:

Nothing specified.
Solubility: precision ± 1 mol % (compilers).

REFERENCES:

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

Allaria, S.

Atti Reale Accad.Sci.Torino, Cl.Sci.Fis.Mat. Nat., 1942-43, *78*, 145-153.

VARIABLES:

Temperature: 567-661 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points of the liquidus of the Cd-Na system were determined.

t/°C	*soly*/mol % Na	*soly*/mol % Cd [a]
320	0.97	99.03
315	2.86	97.14
294	5.56 eutectic	94.44
345	11.10	88.90
358	14.30	85.70
356	15.06	84.94
350	16.70	83.30
348	16.85	83.15
348	17.10	82.90
340	19.4	80.6
349	20.0	80.0
370	29.2	70.8
382	30.6	69.4
388	32.6	67.4

[a] calculated by the compilers

The melting points of Cd and Na were determined as 321.8 and 97.6 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was weighed in a N_2 atmosphere. The alloys were prepared by melting the metals in an inert gas atmosphere. Thermal analysis of the molten alloys was performed as usual applying an Ag/Constantan thermocouple shielded by a silica tube. Some attack of the silica by Na was observed, however, it was said to be negligible.

SOURCE AND PURITY OF MATERIALS:

Cd: contained traces of Zn and Fe.
Na: contained 0.018 % Fe and traces of Pb and Al.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

Weeks, J.R.; Davies, H.A.

The Alkali Metals, The Chem. Soc., London, 1967, p. 32-44.

VARIABLES:

Temperature: 375-599 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Cd in liquid Na was determined; the values were read off the figure by the compilers.

t/°C	*soly*/mol % Cd	t/°C	*soly*/mol % Cd
95.5	0.61 [a] eutectic	199 [b]	2.3
102 [b]	0.68	248 [b]	4.5
123 [c]	0.82	283 [c]	9.1
151 [c]	1.3	301 [b]	12
154 [b]	1.4	326 [c]	14
177 [c]	1.9		

[a] from extrapolation [b] from cooling sequence [c] from heating sequence

The same results were reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Cd and Na were added to a Zr crucible in a vacuum inert-gas box. The apparatus lid was sealed, removed from the box and mounted in a furnace. A slight positive pressure of He was maintained above the melt. The apparatus with the samplers was kept at 673 K for 48 hours, subsequently cooled to 573 K and kept at this temperature for another 24 hours. Each sampler was preheated for 15 minutes just above the crucible and immersed in the melt. The alloy was forced through the filter inside the sampler by increasing the He pressure. The test temperature was altered after sampling and kept constant for another 24 hours. The samplers were opened, Na was dissolved and the filtrate containing Cd in acid and the Cd content were wet-chemically analysed.

SOURCE AND PURITY OF MATERIALS:

Cd: 99.999+ % pure, from American Smelting & Refining Co., containing $\leq 10^{-4}$ % other elements.
Na: 99.98 % pure from MSA Research, containing 0.01 % K, $2.5 \cdot 10^{-3}$ % Rb, $1.5 \cdot 10^{-3}$ % Cs, $1.0 \cdot 10^{-3}$ % Si, $2.0 \cdot 10^{-3}$ % C, $2 \cdot 10^{-4}$ % Fe, Al, $<1.0 \cdot 10^{-3}$ % O, $\leq 1 \cdot 10^{-4}$ % other elements; further purified.
He: "purified".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

(1) Weeks, J.R.; Davies, H.A. *US Atom.Ener.Comm. Rep. BNL-10372*, 1964; *US Atom.Ener.Comm. Rep. CONF-660712-1*, 1966.

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

Neethling, A.J.

South Afric.Atom.Ener.Board Rep. PEL-230, 1974.

VARIABLES:

Temperature: 561-632 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Three points of the liquidus of the Cd-Na system were determined.

T/K	*soly*/mol% Cd
561	95
604	90
632	85

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus was made of Pyrex glass. The amounts of metals needed for the alloy preparation were weighed, Na in a sealed capsule which was introduced into an Ar filled glove box. Cd was added to the Na in the capsule. The mixed metals were heated to about 25 K higher than the expected liquidus temperature and shaken for 30 s. The cooling procedure of the sample was continued to a temperature below the melting temperature of Na. The cooling curves were recorded by means of Chromel/Alumel thermocouples, calibrated on the melting points of Na, Sn, and Pb. The samples were chemically analysed, Na by means of flame photometry and Cd by atomic absorption spectrometry.

SOURCE AND PURITY OF MATERIALS:

Cd: 99.9 % pure from British Drug House.
Na: analytical grade from Merck, filtrated through 2G filter.
Ar: purified and dried by means of Engelhard purification system, O content less than $1 \cdot 10^{-4}$ %.

ESTIMATED ERROR:

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

REFERENCES:

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEASUREMENTS:

Takeda, S.; Tamaki, S.

J. Phys. Soc. Jap. 1989, *58*, 1484-1485.

VARIABLES:

Temperature: 578-654 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the Cd-Na liquidus were determined.

t/°C	*soly*/mol % In
376	85
381	65
322	55
305	30

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloy samples were prepared from the metals in Pyrex glass ampoules sealed in vacuum (1). The magnetic susceptibilities were measured from about 50 K below the expected liquidus temperature to about 773 K. The usual torsion balance method was applied for the measurements. The liquidus temperatures were estimated from break points on the plots of the magnetic susceptibility vs. temperature.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.
Na: probably 99.9 % pure as in (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Takeda, S.; Matsunaga, S.; Tamaki, S. *J. Phys.Soc.Jap.* 1984, *53*, 1448-1452.

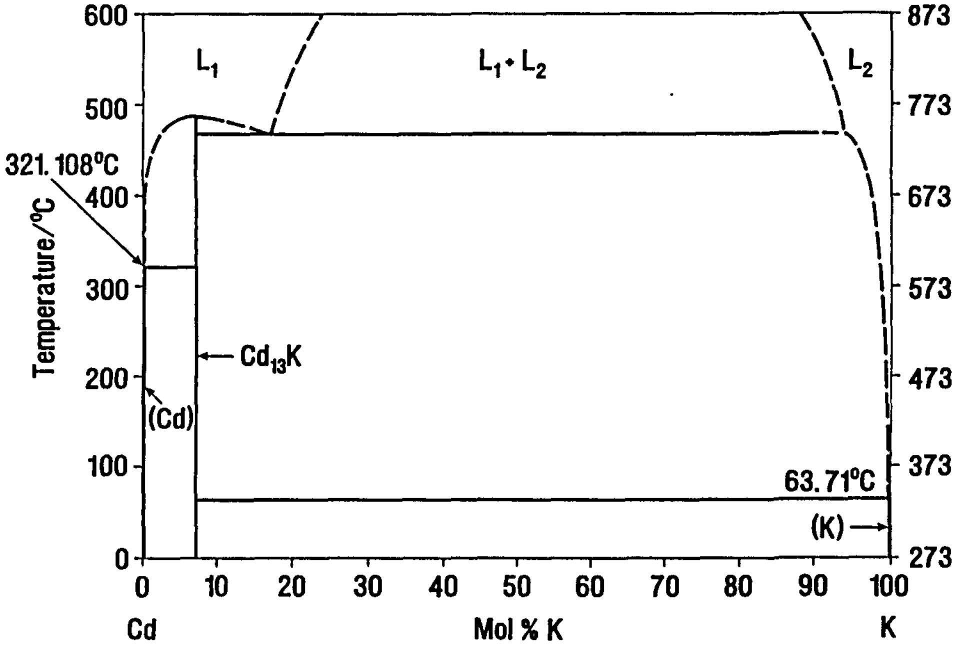
COMPONENTS:	EVALUATOR:
(1) Cadmium; Cd; [7440-43-9] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1991

CRITICAL EVALUATION:

Thermal analyses of Cd-K alloys in the composition range of 10 to 97 mol % Cd were performed by Smith (1), but only the Cd-rich region of the liquidus was well established. Smith reported occurrence of a miscibility gap in the range of 1 to 83 mol % Cd at 741 K, the estimated lower limit of which does not seem to be quantitative. The results of Lantratov and Tsarenko (2) gained at 748 and 848 K by potentiometric experiments indicate a narrower miscibility gap. The composition at the lower immiscibility limit at 748 K seems to be overestimated. Solubility values except for the Cd-rich side cannot therefore, be recommended. The saturated solutions of Cd in liquid K are in equilibrium with $Cd_{13}K$ or liquid Cd saturated with K; see the partial phase diagram of (3).

Tentative values of the solubility of Cd in liquid K

T/K	*soly*/mol % Cd		source
741	83	monotectic	(1)
760	93	congruent	(1)



References

1. Smith, D.P. *Z. Anorg. Chem.* 1908, *56*, 109.
2. Lantratov, M.F.; Tsarenko, E.V. *Zh. Prikl. Khim.* 1960, *33*, 1116.
3. Pelton, A.D. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am.Soc.Mater., Materials Park, 1990, p.990.

COMPONENTS:

(1) Cadmium; Cd; [7440-43-9]

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

ORIGINAL MEAS UREMENTS:

Smith, D.P.

Z. Anorg. Chem. 1908, *56*, 109-142.

VARIABLES:

Temperature: 725-760 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Cd-K liquidus line were determined.

t/°C	*soly*/mol % Cd
471	85.0
472	86.0
473	87.0
479	88.0
478	90.0
486	91.0
487	93.0
479	95.0
452	97.0

The melting points of Cd and K were determined as 322 and 63 °C, respectively.
A miscibility gap is indicated in the concentration range <10 to 85 mol % Cd.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The oxidized surface of K was cut off. The sample was washed in benzene, amyl alcohol, petroleum, and ether. The metals were weighed, K in a petroleum medium, and placed in a tube of hard Jena glass. The tube was filled with H_2 and the metals were subsequently melted in it. The tube was placed in an apparatus for thermal analysis, cooling curves were recorded by means of a calibrated Pt/Pt-Rh thermocouple. The alloys were stirred with a glass rod during solidification.

SOURCE AND PURITY OF MATERIALS:

Cd: nothing specified.
K: "contained no traces" of Na as well as other metals.

ESTIMATED ERROR:

Nothing specified.
Temperature: not better than ± 3 K (compilers).

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Cadmium; Cd; [7440-43-9] (2) Potassium; K; [7440-09-7]	Lantratov, M.F.; Tsarenko, E.V. *Zh. Prikl. Khim.* 1960, *33*, 1116-1128.
VARIABLES:	**PREPARED BY:**
Temperature: 748 and 848 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The limits of the miscibility gap in the Cd-K system were estimated by the compilers from a diagram of numerical values of the potential versus composition.

t/°C	*soly*/mol % Cd (lower limit)	*soly*/mol % Cd (upper limit)
475	7	72
575	10	69

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Precisely weighed amounts of the metals were filled in a glass cell which was also applied for the measurements. The preparation and the EMF experiments were performed in an Ar atmosphere. The temperature was measured by means of a Chromel/Alumel thermocouple. The EMF of the cell:

K / glass containing K^+ / Cd-K

was measured in the composition range of 5.0-97.5 mol % Cd. The miscibility limits were evaluated from the constancy of the potentials versus composition of the molten alloy.

SOURCE AND PURITY OF MATERIALS:

Cd: nothing specified.
K: purified by filtration of the molten metal.
Ar: purified by contact with K-Na melt at 723 K.

ESTIMATED ERROR:

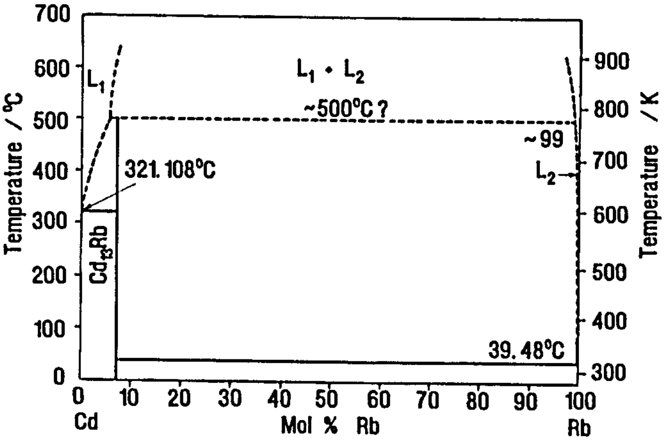
Solubility: precision ± 3 mol % (compilers).
Temperature: precision ± 1 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Cadmium; Cd; [7440-43-9] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1990

CRITICAL EVALUATION:

Solubility determinations in the liquid state have not been performed for this system. Pelton (1) predicated a liquid miscibility gap at a temperature of 773 K in the approximate range of 1 to 95 mol % Cd. The saturated solutions of Cd in liquid Rb are in equilibrium with the $Cd_{13}Rb$ intermetallic compound up to its decomposition temperature; see the partial Cd-Rb phase diagram redrawn from (1).



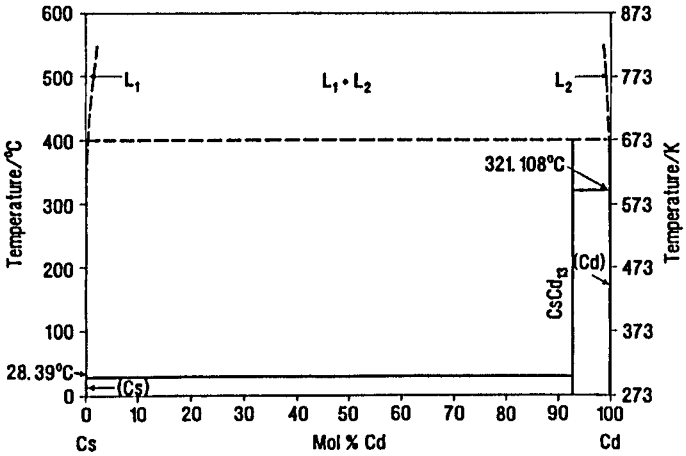
References

1. Pelton, A.D. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 681.

COMPONENTS:	EVALUATOR:
(1) Cadmium; Cd; [7440-43-9] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1990

CRITICAL EVALUATION:

Experimental work concerning solubility determinations of Cd in liquid Cs has not been reported. Pelton (1) predicted mutual solubilities of both metals of less than 1 mol % at a temperature of approximately 773 K. The saturated solutions of Cd in liquid Cs are in equilibrium with the $Cd_{13}Cs$ solid phase up to its decomposition temperature; see the partial Cd-Cs phase diagram redrawn from (1).



References

1. Pelton, A.D. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Met., Metals Park, 1986, p. 649.

COMPONENTS:

(1) Mercury; Hg; [7439-97-6]

(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
January 1990

CRITICAL EVALUATION:

All experimental data concerning the whole liquidus lines of these five systems were recently assessed and collected in a preceding volume of this series (1). Any new publications which might have changed the reported phase diagrams or solubility data are not known.

Tentative values of Hg solubility in liquid Li

T/K	*soly*/mol % Hg	T/K	*soly*/mol % Hg
434	8 eutectic	644	28 eutectic
473	12	673	34
523	14	723	38
573	17	773	40
623	21	823	44
648	25 congruent	873	50 congruent

Recommended values of Hg solubility in liquid Na

T/K	*soly*/mol % Hg	T/K	*soly*/mol % Hg
294.6	14.8 eutectic	~488	49 peritectic
307.2	15.9 peritectic	496	52 peritectic
339.2	28.2 peritectic	573	58
394	38 peritectic	~626	66.7 congruent
423	41		

Tentative values of Hg solubility in liquid K

T/K	*soly*/mol % Hg	T/K	*soly*/mol % Hg
320	6 eutectic	451	54 eutectic
373	18	473	56
423	36	543	66.7 congruent
456	50 congruent		

Tentative values of Hg solubility in liquid Rb

T/K	*soly*/mol % Hg	T/K	*soly*/mol % Hg
298	4 eutectic	443	56 peritectic
323	12	473	58
373	28	530	66.7 congruent
430	48 peritectic		

Tentative values of Hg solubility in liquid Cs

T/K	*soly*/mol % Hg	T/K	*soly*/mol % Hg
289	3 eutectic	444	56 peritectic
323	9	460	62 peritectic
373	26	481	66.7 congruent
410	40 peritectic		

References

1. Guminski, C.; Galus, Z.; Hirayama, C., eds. *Metals in Mercury, Solubility Data Series*, Vol. 25, Pergamon, Oxford, 1986, p. 1-54.

COMPONENTS:	EVALUATOR:
(1) Mercury; Hg; [7439-97-6] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1995

CRITICAL EVALUATION:

Due to the results of recent work on parts (2,3), (4-9) and the whole phase diagram of the Hg-Na system (10,11) the conclusion of Balej et al. (1) that the Hg-Na phase diagram had already been thoroughly investigated was not appropriate. Thus, parts of the phase relations have still to be considered as tentative and need further research.

Emf and thermal analysis studies of Borgstedt et al. (2,3) were performed as physico-chemical background of the extraction process of Hg by liquid Na. They generally confirmed the shape of the liquidus line between 5 and 32 mol % Hg and thermal arrests, observed at 339, 333 and 323 (±1) K, should correspond to either peritectic or polymorphic phase transitions between 26 and 38 mol % Hg. These temperatures are about the same as those reported earlier (12) without further interpretation.

Deiseroth et al. (4-9) investigated crystal structures of HgNa, α Hg_3Na_8, β Hg_3Na_8, γ Hg_3Na_8, α $HgNa_3$ and β$HgNa_3$. They supplemented their x-ray studies by DTA for the alloys containing 50 mol % Hg (5) and 23.1 to 33.3 mol % Hg (7). The melting point of HgNa was found at 485 K. It was definitely found that Hg_3Na_8, instead of Hg_2Na_5 (1,12), passes two phase transitions at 327 and 335 K. The liquidus line in this composition range was confirmed. Moreover, some thermal arrests at 330 K for 27.5 to 33.3 mol % Hg, at 292 K for 26.1 mol % Hg and 287 K for 23.1 and 24.0 mol % Hg could not be related to any defined phase transitions. The liquidus data between 25 and 30 mol % Hg would rather suggest a congruent melting of γ Hg_3Na_8. An eutectic point should appear in its vicinity, but such eutectic line was not identified except only one at the melting temperature of γ Hg_3Na_8 which would suggest a peritectic reaction. Also the thermal arrests corresponding to the α Hg_3Na_8 - β Hg_3Na_8 phase transition were only observed on the Na-richer side of the stoichiometric composition of Hg_3Na_8. If the compounds Hg_3Na_8 and $HgNa_3$ would not be daltonides and exist in certain ranges of composition, their peritectoid temperatures could be different on the Na- and Hg-richer sides.

Sun and Cao (10,11) measured the emfs of several amalgams containing 11.54 to 96.5 mol % Hg at temperatures between 240 and 600 K. They also performed thermal analysis experiments in the range 74.0 to 80.0 mol % Hg. Each part of the liquidus was expressed by a fitting equation. Thus, the temperatures of eutectics, peritectics and configurative transitions for each of the phases and equilibria were calculated. The investigators postulated a modification of the liquidus line. The parameters of the Hg-rich eutectic were confirmed. The peritectic at 430 K was unexpectedly found at 83.6 mol % Hg. Extrapolating the liquidus data on both sides of the congruently melting Hg_2Na they obtained its melting point at 635 K. This value seems to be too high, and the results of earlier measurements compiled in (12) may be more reliable. Peritectics were determined at 57.5 mol % Hg and 493.8 K, 48.5 mol % Hg and 491.6 K, 36.4 mol % Hg and 392.6 K, and 27.9 mol % Hg and 343.2 K, respectively. The parameters of the eutectic were established at 15.0 mol % Hg and 294.9 K. These values are only in qualitative agreement with (12), the parameters of the first, third and fifth point appear to be acceptable.

HgNa and Hg_3Na_8 may even melt congruently, since the experimental points of (11) in the corresponding region are scattered. Thus, only equivocal conclusions can be drawn. The revised Hg-Na phase diagram is shown in the figure. A new set of suggested liquidus lines is proposed.

Recommended values of the solubility of Na in liquid Hg

T/K	*soly*/mol % Na	source
226.0	2.7 eutectic	(1), (10), (12) mean value
273.2	4.2	(1), (11) interpolation
293.2	5.1	(1), (11) mean value
298.2	5.38	(1), (11) mean value
323.2	6.7	(1), (11) interpolation
373.2	10.2	(1), (11) interpolation
430.5	17 peritectic	(1), (10) mean value
523	21	(1), (11) mean value
630	33.3 congruent	(1), (10) mean value of temperature

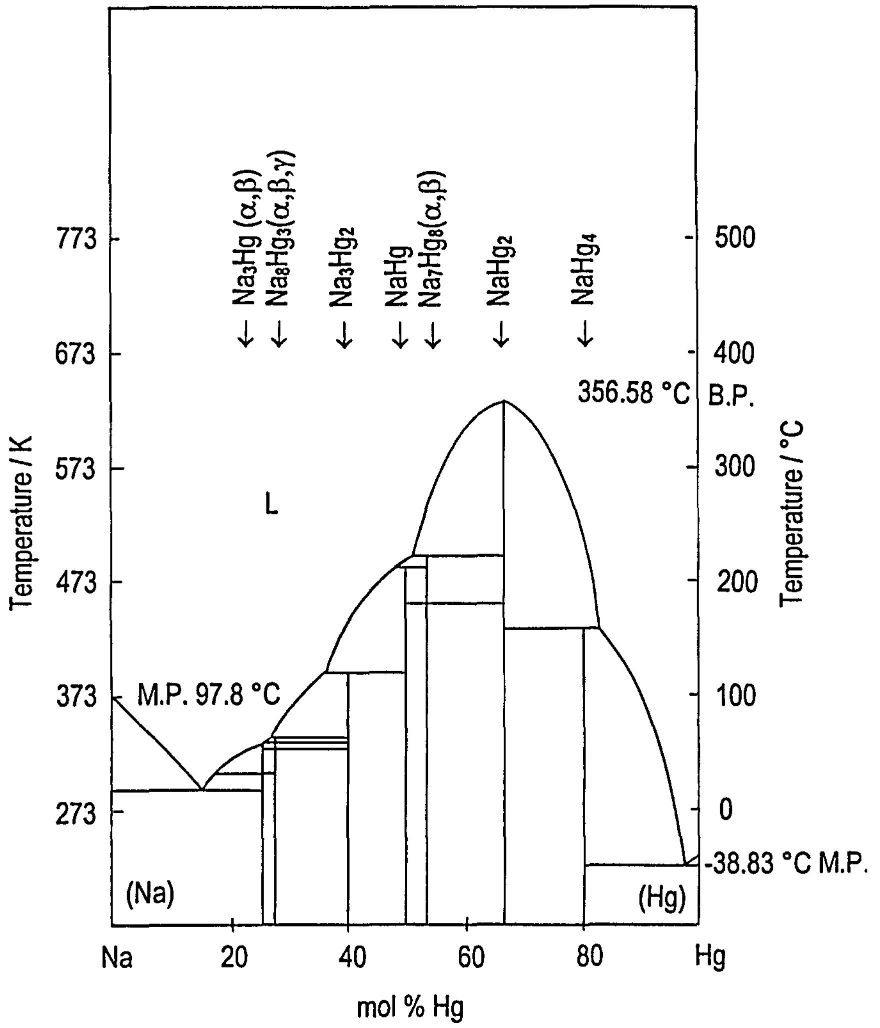
Recommended and tentative (t) values of the solubility of Hg in liquid Na

T/K	*soly*/mol % Hg	source
294.6	15.0 eutectic	(1), (10) mean value
307	17 peritectic	(1), (7,8), (10,11), (12) mean value
333	25.0 peritectic	(1), (7,8), (10,11), (12) mean value
335	26 (t) peritectic	(7)
338	27.2 (t) peritectic	(1), (7,8), (10,11), (12) mean value
393	36.5 peritectic	(1), (10,11), (12) mean value
485	48 (t) peritectic	(1), (6), (10,11), (12) mean value
495	51.5 (t) peritectic	(1), (10,11), (12) mean value
573	57 (t)	(10,11)
630	66.7 congruent	(1), (10) mean value of temperature

Quotation of references (1) and (12) includes all references presented therein.

COMPONENTS:	EVALUATOR:
(1) Mercury; Hg; [7439-97-6] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1995

CRITICAL EVALUATION: (continuation)



References

1. Balej, J.; Guminski, C.; Galus, Z. *Metals in Mercury*, *Solubility Data Series*, C. Hirayama, Ed., Pergamon, Oxford 1986, p. 13.
2. Borgstedt, H.U.; Guminski, C.; Peric, Z. *Liquid Sodium Extraction of Mercury from Wasted Batteries*, 6th Intern. Symp. on Solubility Phenomena, Buenos Aires, 1994.
3. Borgstedt, H.U.; Peric, Z. *Liquid Metal Systems*, H.U. Borgstedt, G. Frees, Eds., Plenum, N.Y. 1995, p. 363.
4. Deiseroth, H.J.; Toelstede, D. *Z. Anorg. Chem.* 1990, *587*, 103.
5. Deiseroth, H.J.; Stupperich, A.; Pankaluoto, R.; Christensen, N.E. *Z. Anorg. Chem.* 1991, *597*, 41.
6. Deiseroth, H.J.; Toelstede, D. *Z. Anorg. Chem.* 1992, *615*, 43.
7. Deiseroth, H.J.; Rochnia, M. *Z. Anorg. Chem.* 1992, *616*, 35.
8. Deiseroth, H.J.; Rochnia, M. *Angew. Chem., Intern. Ed. Engl.* 1993 *32*, 1494; *Angew. Chem.* 1993, *105*, 1556.
9. Deiseroth, H.J.; Rochnia, M. *Z. Anorg. Chem.* 1994, *620*, 1736.
10. Sun, C.; Cao, Y. *Acta Metall. Sinica, Ser. B* 1993, *6*, 256.
11. Sun, C.; Cao, Y. *J. Appl. Sci. (China)* 1994, *12*, no. 3, 287.
12. Hansen, M.; Anderko, K. *Constitution of Binary Alloys*, McGraw-Hill, New York, 1958, p.825.

COMPONENTS:

(1) Mercury; Hg; [7439-97-6]

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

ORIGINAL MEASUREMENTS:

Deiseroth, H.J.; Rochnia, M.

Z. Anorg. Chem. 1992, *616*, 35-38.

VARIABLES:

Temperature: 327 - 377 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Na-rich part of the Hg-Na liquidus was determined.

t/°C	*soly*/mol % Hg	t/°C	*soly*/mol % Hg
104	33.3	62	26.1
73	29.1	59	25.0
64	27.5	57	24.0
64	27.3	54	23.1

The melting point of Na was found at 97 °C.
It was established that no Hg_2Na_5 does occur, but the Hg_3Na_8 phase existing in three polymorphic structures. $HgNa_3$ melts at 59 °C and has two polymorphic structures.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Samples of the Hg-Na alloys of defined composition were prepared in a glass apparatus under Ar atmosphere (1). Liquid Hg was introduced through a capillary into liquid Na. The samples were homogenized with the use of a stirrer. Parts of the samples were placed in small glass tubes which were closed by sealing. The DTA and X-ray diffraction measurements of the small samples were performed simultaneously. The DTA apparatus was calibrated on the melting points of Ga and In. The rate of heating was 2 K/min.

SOURCE AND PURITY OF MATERIALS:

Hg: 99.999 % pure from Degussa; according to (2).
Na: 99+ % pure from Merck, further purified according to (2).
Ar: 99.996 % pure; further purified by passing through the blue gel, molecular sieves, P_2O_5 and Ti strips; the final H_2O content was $< 1 \cdot 10^{-4}$ %; according to (2).

ESTIMATED ERROR:

Solubility: precision better than ± 0.1 mol %.
Temperature: precision ± 0.5 K.

REFERENCES:

1. Deiseroth, H.J.; Strunck, A.; Bauhofer, W. *Z. Anorg. Chem.* 1988, *558*, 128-136.
2. Deiseroth, H.J.; Toelstede, D. *Z. Anorg. Chem.* 1992, *615*, 43-48.

COMPONENTS:

(1) Mercury; Hg; [7439-97-6]

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

ORIGINAL MEASUREMENTS:

Sun, C.; Cao, Y.

J. Appl. Sci. (China) 1994, *12*, no. 3, 287-294.

VARIABLES:

Temperature: 226-583 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Numerous points of the Hg-Na liquidus were determined.

t/°C	*soly*/mol % Na	*soly*/mol % Hg[c]	*t*/°C	*soly*/mol % Na	*soly*/mol % Hg[c]
-48 [a]	2.708	97.29	179.9	58.00	42.00
-16.0 [b]	3.500	96.50	171.3	59.00	41.00
-3.3 [b]	4.000	96.00	160.7	60.00	40.00
8.9 [b]	4.500	95.50	147.0	61.00	39.00
20.4 [b]	5.000	95.00	138.9	62.00	38.00
27.6 [b]	5.500	94.50	121.6	63.00	37.00
38.5 [b]	6.500	93.50	119.8	65.00	35.00
69.8 [b]	8.000	92.00	104.3	67.00	33.00
98.9 [b]	10.00	90.00	90.6	69.00	31.00
126.2 [b]	12.43	87.57	75.0	71.00	29.00
143.6 [b]	14.29	85.71	68.2	72.00	28.00
176.6 [b]	17.13	82.87	67.5	72.50	27.50
234.7 [b]	20.00	80.00	66.1	73.00	27.00
244.4 [b]	21.00	79.00	64.6	73.50	26.50
262.5 [b]	22.00	78.00	63.4	74.00	26.00
280.9 [b]	23.00	77.00	60.9	75.00	25.00
293.6 [b]	24.00	76.00	58.2	76.00	24.00
307.1 [b]	25.00	75.00	55.9	77.00	23.00
309.4	42.00	58.00	53.6	78.00	22.00
288.6	44.00	56.00	51.4	79.00	21.00
277.3	45.00	55.00	49.7	80.00	20.00
265.6	46.00	54.00	46.8	81.00	19.00
247.0	47.00	53.00	40.3	82.00	18.00
228.7	48.00	52.00	33.3	83.00	17.00
222.2	48.50	51.50	28.1	84.00	16.00
220.5	49.00	51.00	21.8	85.50	14.50
217.3	50.00	50.00	26.1	86.00	14.00
214.3	51.00	49.00	30.1	86.50	13.50
215.9	53.00	47.00	33.8	87.00	13.00
208.3	54.00	46.00	41.9	88.00	12.00
201.1	55.00	45.00	44.8	88.46	11.54
194.5	56.00	44.00			
187.7	57.00	43.00			

The melting point of Na was measured to be 97.7 °C.

[a] reported in (1) [b] also reported in (1) [c] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The description of the experiments is from (1). The apparatus had a glass sealing structure. Emf measurements of the cell Na/β-alumina/Na-Hg were performed at various temperatures. Na was introduced into the cell on both sides of the β-alumina tubes. Various amounts of Hg were poured into one of the compartments. The cells were evacuated to < 10^{-2} Pa.

The proper compositions of the alloys were made by constant current coulometry. The temperature was regulated and measured with the use of alcohol or oil or sand baths with a Hg thermometer and a crucible furnace using a Ni-Cr/Ni-Si thermocouple calibrated on the boiling point of H_2O and the melting points of Cd or Sn.

The saturation concentrations were estimated from the break on the relation of potential versus temperature. The liquidus line of the system was based on these points related to the analytical concentration of Hg.

SOURCE AND PURITY OF MATERIALS:

Hg: triple distilled.

Na: electrolytically purified through β-alumina solid electrolyte.

β-alumina: polycrystalline $Na_2O{\cdot}11Al_2O_3$ doped with MgO.

ESTIMATED ERROR:

Solubility: accuracy of alloy preparation ±0.2 %.

Temperature: stability between ±0.1 and ±8 K, precision ± 0.1 K.

Potential: sensitivity 1 μV.

REFERENCES:

1. Sun, C.; Cao, Y. *Acta Metall. Sinica, Ser. B* 1993, *6*, 256-262.

COMPONENTS:

(1) Mercury; Hg; [7439-97-6]

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

ORIGINAL MEASUREMENTS:

Borgstedt, H.U.; Peric, Z.

Liquid Metal Systems, H.U. Borgstedt, G. Frees, Eds., Plenum, N.Y. 1995, p. 363-368.

VARIABLES:

Temperature: 295 - 394 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Na-rich part of the Hg-Na liquidus was determined.

t/°C	*soly*/mol % Hg	t/°C	*soly*/mol % Hg
58	8 [a]	40	18 c
43	12 [a]	62	26 c
22	15 [a]	66	27 b
33	16 [b]	121	37 b

[a] thermal analysis in (1)   [b] EMF in this work   [c] EMF in (1)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experimental set-up was placed inside of a dry Ar glove box. The electrochemical cell consisted of a stainless steel container equipped with an electrical heater. A β-alumina tube hemispherically closed on one end was mounted into the container. The remaining volume of the container was filled with Na and the inner part of the tube with Hg-Na alloy. Mo electrodes were introduced into both metallic phases. The temperatures in the two metals were measured using Ni/Ni-Cr thermocouples calibrated on the melting point of Na. The concentration of Na in the alloy was varied by a constant current electrolysis. After the electrolysis steps, the potential of the cell Na/β-alumina/Na-Hg was measured as function of the temperature using the same electrodes connected to a voltmeter. The saturation points were evaluated from breaks of the potential-temperature and cooling curves recorded at selected alloy compositions.

SOURCE AND PURITY OF MATERIALS:

Hg: 99.99 % pure from Degussa.
Na: nuclear grade purity, skin of impurities scrapped regularly from the molten metal at ~370 K.
β-alumina: from ABB.
Ar: partial pressures of O and H_2O at < 0.1 Pa.

ESTIMATED ERROR:

Solubility: precision ± 0.5 mol %.
Temperature: precision ± 1 K.

REFERENCES:

1. Borgstedt, H.U.; Guminski, C.; Peric, Z. 6th *Intern. Symp. on Solubility Phenomena*, Buenos Aires, 1994, abstracts.

COMPONENTS: (1) Aluminum; Al; [7429-90-5] (2) Lithium; Li; [7439-93-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1991

CRITICAL EVALUATION:

Numerous experimental data concerning the liquidus of the Al-Li system has been generated. The scatter is significant over the whole range of compositions. A selection of the most reliable results may be supported by thermodynamic modelling of the phase diagram (11-13,18,19).
The Al-rich side of the system was more frequently studied, since such Al-Li alloys have found practical use. Czochralski and Rassow (1) determined an eutectic point at 76.8 mol % Al and 861 K. Aßmann (2) studied a similar range of compositions; he placed the eutectic at higher temperature and Li concentration. The eutectic parameters were recently reported by Hanna and Hellawell (9) and Dubost et al. (10) to be 70.2 mol % Al at 873 K and 71.3 mol % Al at 874 K, respectively. The publications (1,9,10) do not contain sufficient information on the thermal analyses and are, therefore, not compiled. Even the results of modelling estimations of the eutectic composition at 873 K are scattered: 74, 75.7, 77.6, 74.2 and 74.3 mol % Al as calculated by Saboungi and Hsu (11), McAlister (12), Sigli and Sanchez (13), Chen et al. (18) and Saunders (19), respectively. The calculation of the phase diagram from the "first principles" by Sluiter (20) is in disagreement with experiment.
The composition of the congruently melting phase was reported as AlLi; the corresponding temperature of the maximum varied from 968 (6) to 991 K (5). Grube et al. (4) claimed a miscibility gap between 40 and 55.5 mol % Al at 971 K, but this gap was never confirmed in the other investigations. The discrepancy of the liquidus data in the Li-rich region is up to 8 mol %. The results with lower Al concentrations seem to be more reliable. The result of Müller (3) at 723 K appeared to be casual, since the purity of the solvent was poor.
The work of Selman et al. (7) produced the most convincing result, however, only one value at 744 K was determined which is in perfect agreement with data of Grube et al. (4). The eutectic at the Li-rich side was determined by Saldau and Shamray (5). It seems to occur at a lower Al content as can be concluded from an extrapolation of other liquidus data.
There are three possible reasons for the bad accuracy of the liquidus data: (i) the reaction of dissolved Al with the Fe container (3,4,5,17) with formation of Al_2Fe (6); even containers made of Ta (6,7), glass (2,3) or graphite (2) may not be resistant; (ii) formation of ternary Al-Li-O oxides in the presence of O in the system (14,15); and (iii) losses of Li which may occur during the experiments (18). An Al_2O_3 sample was dissolved in liquid Li at 1089 K (16). These facts were not considered by the investigators, except (18), in the evaluation of data concerning the liquidus lines of the Al-Li system. It was proved in (18) that a crucible made of BN is sufficiently resistant to study the Al - Li system in the liquid state. The calculated phase diagrams (11-13,18,19) suggest higher values of the solubility of Al in liquid Li than the data selected from the experimental work.
The saturated solutions of Al in liquid Li are in equilibrium with the Al-Li intermediate phases as can be seen in the most advanced phase diagram (18) which is redrawn below.

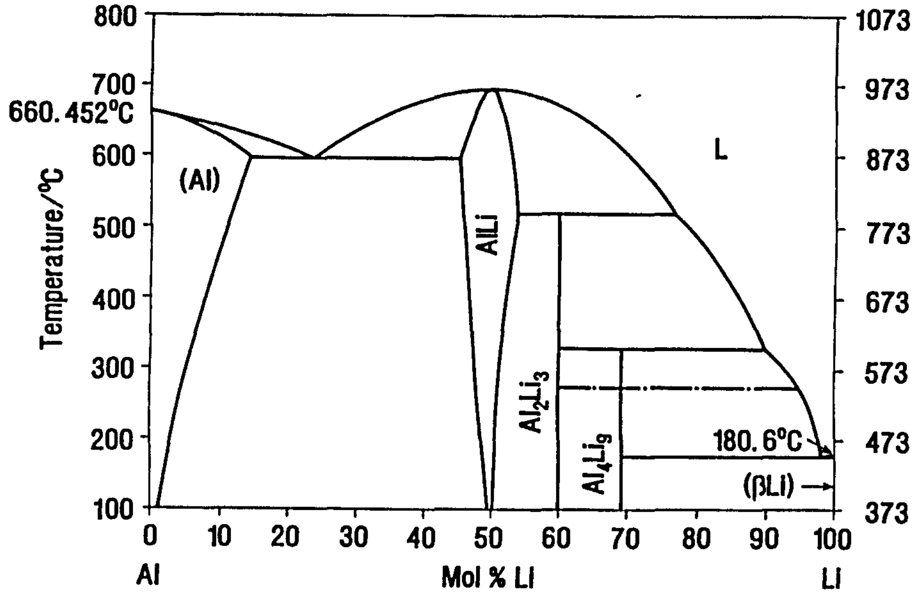
Recommended (r), tentative (t) and doubtful (d) values of the solubility of Al in liquid Li

T/K	*soly*/mol % Al	source
448	~2 (d) eutectic	(12) extrapolated
573	7 (d)	(4) extrapolated
603	10 (t) peritectic	(5)
673	12 (t)	(5)
773	20 (t)	(4,7) interpolated
793	22 (d) peritectic	(4,6) interpolated
873	30 (d)	(4) interpolated
973	50 (r) congruent	(3,4)

The two liquid metals are completely miscible above 973 K.

COMPONENTS:	EVALUATOR:
(1) Aluminum; Al; [7429-90-5] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1991

CRITICAL EVALUATION: (continued)



References

1. Czochralski, J.; Rassow, H. *Moderne Metallkunde*, Springer, Berlin, 1924, p. 36.
2. Aßmann, P. *Z. Metallk.* 1926, *18*, 51.
3. Müller, A. *Z. Metallk.* 1926, *18*, 231.
4. Grube, G.; Mohr, L.; Breuning, W. *Z. Elektrochem.* 1935, *41*, 880.
5. Shamray, F.I.; Saldau, P. Ya. *Izv. Akad. Nauk SSSR, Ser. Khim.* 1937, 631.
6. Myles, K.M.; Mrazek, F.C.; Smaga, J.A.; Settle, J.L. *US Ener.Res.Devel.Agen.Rep. ANL-76-8*, 1976, p. B50.
7. Selman, J.R.; DeNuccio, D.K.; Sy, C.J.; Steunenberg, R.K. *J. Electrochem. Soc.* 1977, *124*, 1160.
8. Pivkina, O.G.; Korsunskaya, I.A., Zinin, V.I. *Fiz. Tekhn. Vysok. Davlenii* 1984, no. 16, 61.
9. Hanna, M.D.; Hellawell, A. *Metal. Trans.,A* 1984, *15*, 595.
10. Dubost, B.; Bompard, P. Ansara, I. *J. Physique. Colloq. C3* 1987, *48*, 473.
11. Saboungi, M.L.; Hsu, C.C. *CALPHAD* 1977, *1*, 237; *US Nat. Bur. Stand. Rep. SP-469*, 1978, p. 1109.
12. McAlister, A.J. *Bull. Alloy Phase Diagr.* 1982, *3*, 177.
13. Sigli, C.; Sanchez, J.M. *Acta Metall.* 1986, *34*, 1021.
14. Konys, J.; Borgstedt, H.U. *J. Nucl. Mater.* 1985, *131*, 158.
15. Migge, H. *J. Nucl. Mater.* 1988, *155-157*, 455.
16. Hoffmann, E.E. *US Atom.Ener.Comm.Rep. ORNL-2924*, 1960.
17. Schürmann, E.; Voss, H.J. *Giessereiforsch.* 1981, *33*, 39.
18. Chen, S.W.; Jan, C.H.; Lin, J.C.; Chang, Y.A. *Metall. Trans.,A* 1989, *20*, 2247.
19. Saunders, N. *Z.Metallk.* 1989, *80*, 894.
20. Sluiter, M.; de Fontaine, D.; Guo, X.Q.; Podlonocky, R.; Freemen, A.J. *Phys.Rev.B* 1990, *42*, 10460.
21. Schürmann, E.; Geissler, I.K. *Gießereiforsch.* 1980, *32*, 163.

COMPONENTS:

(1) Aluminum; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

Aßmann, P.

Z. Metallk. 1926, *18*, 51-54.

VARIABLES:

Temperature: 871 and 968 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Al-Li system in the range of composions rich in Al was presented in a smooth curve. Two numerical values were inserted in the text.

t/°C	*soly*/mass % Li	*soly*/mol % Al [a]
598	7.8	75.3 eutectic
695	12.1	65.1

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by dissolution of Li in molten Al under LiCl melt. The crucibles and a shielding tube of the thermoelement were made of Pythagoras material or graphite inside plated with Al_2O_3+MgO+$MgCl_2$ sinter. Thermal analysis of the alloys was performed in this equipment.

SOURCE AND PURITY OF MATERIALS:

Al: 99.5 % pure with contents of 0.25 % Si, Fe.
Li: 98 % pure.

ESTIMATED ERROR:

Nothing specified.

COMPONENTS:

(1) Aluminum; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

Müller, A.

Z. Metallk. 1926, *18*, 231-232.

VARIABLES:

Temperature:723-973 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the Al-Li liquidus were determined.

t/°C	*soly*/mass % Al	*soly*/mol % Al [a]
450	30	10
700	82	53
650	88	64
593	93.8	78
635	95.9	86
650	98.3	93

[a] as calculated by the compilers

The melting points of Al and Li were found to be 657 and 179 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Weighed amounts of Al were melted under a layer of the protective mixture of LiCl+KCl eutectic in a Jena glass container. A weighed amount of Li was quickly introduced into the molten Al by means of a Fe wire. The preparation of the alloy with the high content of Li was performed by introducing Al wire into molten Li in a Fe container. A Ni/Cr-Ni thermocouple was applied to measure the temperature during the thermal analyses.

SOURCE AND PURITY OF MATERIALS:

Al: with contents of 0.26 % Fe and 0.16 % Si.
Li: 93.5 % pure, with 6 % Na and traces of K and Ca.

ESTIMATED ERROR:

Nothing specified.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Aluminum; Al; [7429-90-5] (2) Lithium; Li; [7439-93-2]	Grube, G.; Mohr, L.; Breuning, W. *Z. Elektrochem.* 1935, *41*, 880-883.
VARIABLES:	**PREPARED BY:**
Temperature: 630-973 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Al-Li liquidus were determined.

t/°C	*soly*/mol % Li	*soly*/mol % Al [a]	*t*/°C	*soly*/mol % Li	*soly*/mol % Al [a]
655	3.6	96.4	698	52.0	48.0
650	8.3	91.7	700	53.3	46.7
647	10.0	90.0	698	55.3	44.7
645	11.8	88.2	698	59.0	41.0
639	15.0	85.0	690	61.0	39.0
629	20.0	80.0	676	63.5	36.5
613	24.0	76.0	633	66.3	33.7
609	27.5	72.5	628	67.3	32.7
600	30.0	70.0	585	71.8	28.2
652	35.0	65.0	571	73.6	26.4
672	38.0	62.0	505	80.0	20.0
698	46.6	53.4	467	83.5	16.5
697	48.8	51.2	357	90.5	9.5
698	50.0	50.0			

[a] calculated by the compilers

The melting points of Al and Li were found to be 658 and 179 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a (low C) Fe crucible which was filled with both the metals in an Ar atmosphere. It was then heated to above the melting point of the sample. Cooling curves of the molten alloy were recorded by means of a Ni/Ni-Cr thermocouple which was calibrated on the melting points of Sn, Cd, Zn, and Mg. The thermocouple was sheathed against the molten alloy by a Fe tube. The Fe content of the alloy samples did never exceed the 0.5 mol % limit as was shown by chemical analyses.

SOURCE AND PURITY OF MATERIALS:

Al: 99.8 % pure.
Li: 99 % pure with contents of 0.62 % K, 0.14 % Na, 0.02 % Fe_2O_3, traces of Al_2O_3, 0.05 % SiO_2, 0.32 % Li_3N.
Ar: purified by flowing through molten Li.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Aluminum; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

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

Izv. Akad. Nauk SSSR, Ser. Khim. 1937, 631-640.

VARIABLES:

Temperature: 448-991 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Al-Li liquidus were determined.

t/°C	*soly*/mol % Li	*soly*/mol % Al [a]	*t*/°C	*soly*/mol % Li	*soly*/mol % Al [a]
657	0.93	99.07	718	50.00	50.00
654	4.46	95.54	706	52.50	47.50
651	7.90	92.10	685	56.00	44.00
650	10.45	89.55	656	60.86	39.14
640	15.73	84.27	584	64.63	35.37
618	22.80	77.20	531	69.00	31.00
612	24.60	75.40	518	71.53	28.47
602	26.30	73.70	486	79.00	21.00
622	30.20	69.80	429	83.33	16.67
641	33.00	67.00	351	88.60	11.40
662	36.50	63.50	335	89.74	10.26
679	40.00	60.00	314	91.00	9.00
690	45.00	55.00	175	96.60	3.40
709	48.00	52.00	177	98.50	1.50

[a] calculated by the compilers

The melting points of Al and Li were found to be 658 and 180 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys with up to 44 mol % Al were prepared in a Fe crucible in an Ar atmosphere. The alloys richer in Al were made in a porcelain crucible under molten KCl+LiCl eutectic mixture. Li was introduced into the molten Al by means of a W wire which also served for mixing. Thermal analyses of the alloys were performed, the temperatures were measured by means of a Pt/Pt-Rh(10%) thermocouple calibrated on the boiling point of H_2O and the melting points of Ag, Sb, Zn, Sn. The alloys were chemically analyzed for their Li contents.

SOURCE AND PURITY OF MATERIALS:

Al: 99.81 % pure with contents of 0.16 % Si and 0.03 % Fe.
Li: 99.5 % pure.
Ar: purified by contacting with Ca+$CaCO_3$ mixture at 673-773 K and finally with molten Li.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Aluminum; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

Myles, K.M.; Mrazek, F.C.; Smaga, J.A.; Settle, J.L.;

US Ener.Res.Devel.Agen.Rep. ANL-76-8, 1976, p. B50-B73.

VARIABLES:

Temperature: 621-968 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Al-Li system was determined. The experimental values were read out of the figure by the compilers.

t/°C	*soly*/mol % Al	*t*/°C	*soly*/mol % Al
348	10.0	687	53.0
459	19.6	680	54.4
539	24.2	661	61.6
596	31.4	647	64.3
625	33.2	600	68.4 (eutectic)
665	39.6	602	74.1
671	42.1	610	78.9
687	44.5	631	84.4
692	48.4	640	92.5
695	50.0	648	95.1
694	51.7		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a Ta crucible in which Al was dissolved in molten Li at a temperature 30 K above the liquidus (known from literature). The alloys were stirred, spalt cast, homogenized just below the solidus for about 3 h and quenched to room temperature. The liquidus temperatures of each of the alloys were determined using a differential thermal analyzer. The composition of the alloys was confirmed by chemical analyses.

SOURCE AND PURITY OF MATERIALS:

Al: "pure".
Li: "pure".

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.2 mol %.
Temperature: read-out procedure ± 3 K.

REFERENCES:

COMPONENTS:

(1) Aluminum; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

Selman, J.R.; DeNuccio, D.K.; Sy, C.J.; Steunenberg, R.K.
J. Electrochem. Soc. 1977, *124*, 1160-1164.

VARIABLES:

One temperature: 743 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Al in liquid Li at 470 °C was determined to be 17.2±0.3 mol % Al.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Samples of the Al-Li alloy were prepared by heating carefully weighed amounts of the metals together at 1023-1073 K, stirring the melt, and quickly absorbing it in the Feltmetal frit. The alloy samples were then annealed for 48 h at 773 K. The composition of the alloys was confirmed by chemical analyses. The chemical activity of Li in the alloy was measured electrochemically, in the cell:
Li (in Feltmetal or pool electrode) / LiF-LiCl-LiBr eutectic / Al_xLi_{1-x} (in Feltmetal).
The electrolyte (31 % LiF, 47 % LiCl and 22 % LiBr) was contained in a porous BeO crucible which separated the electrolyte from the liquid Li pool electrode. The cell temperature was measured by means of a Chromel/Alumel thermocouple sheathed with Ta. This was calibrated on the melting points of Sn, Zn, and Al. An alloy sample containing ~ 10 mol % Al was anodically oxidized under coulometric conditions, and the emf was measured. The discontinuity of the curve relating the emf vs. the composition corresponds to the phase transition at the liquidus composition. The experiments were performed in a He atmosphere.

SOURCE AND PURITY OF MATERIALS:

Al: 99.99 % pure from Materials Research Corp..
Li: unspecified purity from Foote Mineral Corp., premelted in a Ta cup at 473 K; only pure surface drops of molten Li were used in the solubility study.
He: "high purity".
LiF, LiCl, LiBr: unspecified purity, supplied by Anderson Physics Labor..
Feltmetal: porous Ni of unspecified purity from Brunswick Corp..

ESTIMATED ERROR:
Solubility: precision ± 2 %.
Temperature: stability ± 1 K.

REFERENCES:

COMPONENTS:

(1) Aluminium; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

Schürmann, E.; Voss, H.J.

Giessereiforschung 1981, *33*, 39-42.

VARIABLES:

Temperature: 466-969 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The data of the Al-Li liquidus were read out from the figure by the compilers.

t/°C	*soly*/mol % Al	*t* /°C	*soly*/mol % Al	*t*/°C	*soly*/mol % Al
193	0.5	595	30.5	633	85.5
213	1.5	624	33.5	640	89.5
243	3.0	646	41.0	655	96.0
276	4.5	686	46.0	675[a]	61.0
293	6.0	696	50.0	647[a]	66.0
321	10.0	693	54.0	627[a]	71.0
359	12.5	679	60.5	607[a]	74.0
376	13.5	660	65.0	611[a]	77.5
417	16.0	639	70.0	613[a]	79.0
457	19.5	612	74.0	627[a]	83.0
501	23.5	605	76.0	646[a]	90.5
533	26.0	609	78.0		
554	27.5	618	81.0		

[a] obtained by means of the same method as reported in (1)

The melting points of Al and Li were found at 660 and 180 °C, respectively. Four equilibrium solid phases with extended homogenity ranges were identified: ~ AlLi, ~ Al_3Li_7, ~ $AlLi_5$, and ~ $AlLi_9$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared from the metals by melting them in a closed stainless steel crucible under Ar atmosphere. The melts were conditioned in a furnace for 15 min at 973 K. Temperatures were measured and controlled by means of calibrated Pt/Pt-Rh(10%) thermoelements. Thermal analyses of the alloy samples were performed at different cooling rates: 2 and 5 K per min.

SOURCE AND PURITY OF MATERIALS:

Al: 99.99+ % pure with contents of < $1.0 \cdot 10^{-3}$ % Fe, Mg, Si, and <$8.0 \cdot 10^{-4}$ % Cu.
Li: 99.8+ % pure with contents of < 0.2 % (Na+K), vacuum remelted and surface layer removed.

ESTIMATED ERROR:

Solubility: nothing specified; read-out procedure ± 0.5 mol %.
Temperature: precision ± 0.5 K; read-out procedure ± 2 K.

REFERENCES:

1. Schürmann, E.; Geissler, I.K. *Giessereiforschung* 1980, *32*, 163-174.

COMPONENTS:

(1) Aluminium; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

Chen, S.W.; Jan, C.H.; Lin, J.C.; Chang, Y.A.

Metall. Trans. A 1989, *20*, 2247-2258.

VARIABLES:

Temperature: 873-953.5 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the Al-Li liquidus were determined.

T/K	*soly*/mol % Al	T/K	*soly*/mol % Al
953.5	59.2	893[a]	79.3
922.5[a]	66.3	912[a]	85.0
901[a]	70.0	921[a]	90.8
873	74.2	924[a]	92.0

[a] from Al-Li alloy supplied from ALCOA

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Supplied Al-Li alloy samples were surface cleaned and others were prepared from the metals. Weighed amounts of Al and Li (surface cut off) were loaded into a BN crucible, which was covered with a BN lid and placed in a quartz capsule. The complete capsule was removed from the dry box, evacuated, again filled with Ar to 34 kPa and sealed. The capsule was conditioned for 20 minutes at 973 K and quenched into ice water. The composition of the sample was analyzed using an inductively coupled plasma (ICP) method. All differential thermal analysis measurements were performed in the heating mode in order to avoid supercooling. The apparatus was calibrated on the melting points of Al and Zn.

SOURCE AND PURITY OF MATERIALS:

Al-Li alloy: from ALCOA.
Al: nothing specified.
Li: nothing specified.
Ar: with a content of $< 5 \cdot 10^{-4}$ % O.

ESTIMATED ERROR:

Solubility: better than ± 0.5 mol %.
Temperature: accuracy ± 1 K; precision ± 2 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Aluminum; Al; [7429-90-5] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1991

CRITICAL EVALUATION:

Heycock and Neville (1) did not succeed in dissolving appreciable quantities of Al in liquid Na. Mathewson (2) performed some thermal analyses and established the monotectic temperature of the Al-Na system at 930 K. He also reported that the mutual solubilities of the two metals at this temperature were below the limits of analytical detection (which were not reported).

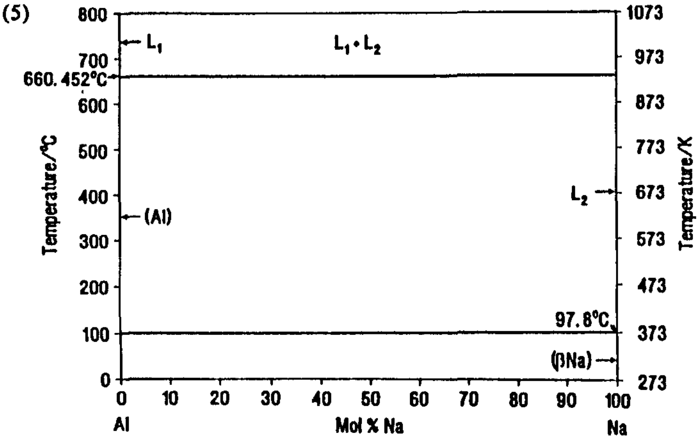
Several workers investigated the Al-rich side of the system (3-5). The results of Scheurer (3) were characterized by a significant scatter. The data of Fink et al. (4) showed a decrease of the solubility of Na in liquid Al with increasing temperature. The position of the monotectic was found at 932 K. The most precise and consistent results of this part of the system were determined by Ransley and Neufeld (5) who observed an increase of the solubility of Na in liquid Al with increasing temperature.

Only Brush (6) measured the solubility of Al in liquid Na. A smooth solubility curve was presented in (7). The reliability of the results was thought to be doubtful by the author (6), since he detected also Fe, Ni and Cr in the equilibrated Na indicating a reaction of the solvent with the stainless steel of the container. The interaction of these metals with Al is well known (8) and it has a disturbing effect on the Al-Na equilibrium. The presence of O in Na, which was not specified, may cause another interference with the Al-Na equilibrium. The formation of Al_2O_3, which is more stable than Na_2O, may protect metallic Al against dissolution in Na. A fair resistance of Al_2O_3 against liquid Na at even 1089 K, which was observed by Hoffman (9), indicates the low solubility of this compound in Na. Indeed, the apparent solubility of Al in Na was found to be almost independent of temperature. This fact supports the suggestion that a multi-component equilibrium might have occurred in the experiments of (6). The solubility equation which was reported in (10) on the basis of experiments of (6) is, therefore, not recommended. Only doubtful values of the solubility may be suggested. The temperature dependence of the solubility of Al in liquid Na should be much steeper according to the cellular model published in (11).

A partial Al-Na phase diagram was presented in (8), it is the basis of the figure below.

Doubtful values of the solubility of Al in liquid Na

T/K	*soly*/mol % Al	source
473	$1\cdot10^{-2}$	(6)
573	$1.5\ 10^{-2}$	(6)
673	$2\cdot10^{-2}$	(6)
773	$3\ 10^{-2}$	(6)
932	99.86 Al-rich monotectic	(5)



References

1. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1889, *55*, 666.
2. Mathewson, C.H. *Z. Anorg. Chem.* 1906, *48*, 191.
3. Scheuer, E. *Z. Metallk.* 1935, *27*, 83.
4. Fink, W.L.; Willey, L.A.; Stumpf, H.C. *Trans. AIME* 1948, *175*, 364.
5. Ransley, C.E.; Neufeld, H. *J. Inst. Met.* 1950-1951, *78*, 25.
6. Brush, E.G. *US Atom.Ener.Comm.Rep. KAPL-M-EGB-16*, 1954.
7. Brush, E.G. *Corrosion* 1955, *11*, 299t.
8. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Mater., Materials Park, 1990, p.176.
9. Hoffman, E.E. *US Atom.Ener.Comm.Rep. ORNL-2924*, 1960.
10. Johnson, H.E.; McKisson, R.L.; Eichelberger, R.L.; Gehri, D.C. *Sodium Na-K Engineering Handbook*, O.J. Foust, Ed., Gordon and Breach, N.Y., 1972, *1*, 169.
11. Niessen, A.K.; deBoer, F.R.; Boom, R.; de Châtel, P.F.; Mattens, W.C.M.; Miedema, A.R. *CALPHAD* 1983, *7*, 51.

COMPONENTS:

(1) Aluminum; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

Scheuer, E.

Z. Metallk. **1935**, *27*, 83-85.

VARIABLES:

Temperature: 973-1073 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Na in liquid Al was determined at three temperatures.

t/°C	*soly*/mass % Na	*soly*/mol % Al [c]
700	0.167±0.014 [a]; 0.104±0.001 [b]	99.86 [a]; 99.91 [b]
750	0.117±0.007 [a]; 0.106 [b]	99.90 [a]; 99.91 [b]
800	0.124±0.014 [a]; 0.129±0.005 [b]	99.89 [a]; 99.89 [b]

[a] from the upper fraction
[b] from the lower fraction
[c] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test crucibles made of Fe or sinter corundum were loaded with the weighed amounts of Al and Na. They were then inserted into a closed capsule which was heated in a furnace. The capsule was filled with H_2 and heated to the desired temperature, the two metals were mixed by means of a Fe tube. The system was equilibrated for 10 to 120 min. A liquid Al sample was collected by means of the tube. The apparatus was quickly cooled. The sample was dissolved in H_2O for analysis of the Na content.

SOURCE AND PURITY OF MATERIALS:

Al: 99.7 % pure, with contents of 0.10 % Si and 0.2 % Fe.
Na: "technical" purity from Degussa.
H_2: dried by contact with Na.

ESTIMATED ERROR:

Solubility: precision better than ± 10 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Aluminum; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

Fink, W.L.; Willey, L.A.; Stumpf, H.C.

Trans. AIME 1948, *175*, 364-371.

VARIABLES:

Temperature: 933-1073 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Al-Na system in the range of high Al contents was determined.

t/°C	*soly*/mass % Na	*soly*/mol % Al [a]
659.25	0.10	99.92
659.5	0.07	99.96
659.7	0.05; 0.05	99.96; 99.96
659.6	0.05	99.96
659.85	0.03_2	99.97
659.9	0.02_5	99.98
660.0	0.01	99.992
660.15	0.01	99.992
670	0.17	99.86
695	0.17	99.86
730	0.16	99.87
750	0.17	99.86
770	0.16	99.87
790	0.15	99.87
800	0.16	99.87

[a] calculated by the compilers

The melting points of Al and Na were found to be 660 and 97.5 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Weighed amounts of Al and Na were placed in an alumina crucible encapsulated in a Fe container which was closely capped, pressurized with Ar and heated to the equilibration temperature. After soaking for 2-5 h the container was quenched in a H_2O stream. Analyses were made of drillings which were taken from the bottom part. The solubility values of alloys with a Na content lower than 0.1 mass % were determined by direct and differential thermal analysis. The temperatures were measured by means of calibrated Chromel/Alumel thermocouples.

SOURCE AND PURITY OF MATERIALS:

Al: "high" purity, with contents of 0.002 % Si, Fe, Cu and $4 \cdot 10^{-4}$ % Mg.
Na: unknown purity; trimmed and rinsed with isopentane.
Ar: 99.6 % pure.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Aluminum; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

Ransley, C.E.; Neufeld, H.

J. Inst. Met. 1950-1951, *78*, 25-46.

VARIABLES:

Temperature: 938-1048 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Na in liquid Al was determined.

t/°C	*soly*/mass % Na	*soly*/mol % Al [a]
665	0.14	99.88
670	0.15, 0.15	99.87, 99.87
700	0.18	99.85
725	0.20, 0.22	99.83, 99.81
750	0.22, 0.23	99.81, 99.80
775	0.25	99.71
685	0.15_5 [b]	99.87 [b]
715	0.19_5 [b]	99.83 [b]

[a] calculated by the compilers
[b] Al of commercial purity

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An Al sample was placed in a graphite crucible. Na was pressed on top of the Al sample. The loaded crucible was slipped into a mild steel bomb which was immediately stoppered. The bomb was slowly heated to the desired temperature in an electric furnace which was kept at temperature for 30-45 min. The bomb was then taken out of the furnace and quenched by immersing its bottom in a tank of H_2O. The ingot was extracted out of the bomb with alcohol. The graphite absorbed a certain amount of Na, free Na was, however, found to be always present. The samples of alloys were analyzed for their Na content by means of an unspecified method.

SOURCE AND PURITY OF MATERIALS:

Al: "super purity" with 0.008 % Si, 0.0005 % Fe, and 0.0008 % Na; "commercial purity" with 0.22 % Si, 0.47 % Fe, and 0.003 % Na.
Na: "commercial purity".

ESTIMATED ERROR:

Nothing specified.
Solubility: precision better than ± 5 % in the determinations of Na (by the compilers).

REFERENCES:

COMPONENTS: (1) Aluminum; Al; [7429-90-5] (2) Sodium; Na; [7440-23-5]	**ORIGINAL MEASUREMENTS:** Brush, E.G. *US Atom.Ener.Comm.Rep. KAPL-M-EGB-16*, 1954.
VARIABLES: Temperature: 423-773 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Al in liquid Na was determined. The values were read out of the figure in (1) and recalculated to mol % by the compilers.

t/°C	*soly*/mol % Al
150	0.024
200	0.010
250	0.015
300	0.011
350	0.018
400	0.018
450	0.023
500	0.0064

A smooth solubility curve was reported in (2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Al strips were immersed in a stainless steel pot containing Na. The entire system was under Ar atmosphere. The molten alloy was sampled by means of an evacuated glass sampler at the selected temperature after equilibration periods of 1 to 24 h. The samples were wet chemically analyzed for contents of both the metals.

SOURCE AND PURITY OF MATERIALS:

Al: 99.95 % pure.
Na: unspecified, purified by filetering through 5 μm filter.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Claar, T.D. *Reactor Technol.* 1970, *13*, 124-146.
2. Brush, E.G. *Corrosion* 1955, *11*, 299t.

COMPONENTS:

(1) Aluminium; Al;[7429-90-5]

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

EVALUATOR:

H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland
March 1990

CRITICAL EVALUATION:

Smith (1) performed thermal analyses on several Al-K alloys. He reported that the monotectic line at 929 K extended from <10 to >95 mol % Al. He observed an interaction of the alloys with the glass of the container; the system could, therefore, be contaminated. A negligible miscibility of Al and K in the liquid state has to be concluded from some experiments on the crystallization of Al-Si(13%) alloys by Czochralski and Kaczynski (2). Aleksandrov and Dalakova (3) reported that solid Al did not dissolve in liquid K at 873-923 K during an equilibration of 1 h duration. The detection limit of the spectral analysis used was not specified. The compilers interpret these findings that liquid K may dissolve <1 mol % Al at 929 K. Intermetallic compounds are not formed in the system (4) according to the partial phase diagram which is analogous to that shown for the Al-Na system.

References

1. Smith, D.P. *Z.Anorg.Chem.* 1908, *56*, 109.
2. Czochralski, J.;Kaczynski, J. *Wiad.Inst.Metal.Metalozn.* 1937, *4*, 18.
3. Aleksandrov, B.N.;Dalakova, N.V. *Izv.Akad.Nauk SSSR, Met.* 1982, no. 1, 133.
4. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am.Soc.Mater., Materials Park, 1990, p. 165.

COMPONENTS:

(1) Aluminium; Al;[7429-90-5]

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

ORIGINAL MEASUREMENTS:

Smith, D.P.

Z.Anorg.Chem. 1908, *56*, 109-142.

VARIABLES:

One temperature: 929 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

A miscibility gap in the Al-K system was found in the concentration range <10 to >95 mol % Al at a temperature close to the melting point of Al (656 °C).
The melting temperatures of Al and K were determined to be 657 and 63.6 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

K was purified by means of cutting off the surface oxide and washing in benzene, amyl alcohol, petroleum and ether. The metals were weighed (K under petroleum) and placed in a tube of hard Jena glass. The metals were melted within this tube under H_2 atmosphere. The glass was coloured due to an interaction with the metals. The tube was placed in an apparatus for thermal analysis. Cooling curves were recorded, a calibrated Pt/Pt-Rh thermocouple was used to measure the temperatures. A glass rod served to stir the alloys during solidification.

SOURCE AND PURITY OF MATERIALS:

Al: 99.4 % pure, with contents of 0.16 % Si and traces of Fe.
K: did not contain detectable amounts of other metals or Na.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Aluminum; Al; [7429-90-5] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1990

CRITICAL EVALUATION:

Only insignificant solubility of Al in liquid Rb can be expected from tests on the recrystallization of Al-Si(13%) alloy (1). Pinchback (2), however, reported a partial dissolution of an Al sample which was equilibrated with liquid Rb containing some O at 400 K. The solubility of Al in Rb could not be estimated from the weight loss of the sample. The presence of O may increase the dissolution of Al due to the probably formation of $RbAlO_2$ (3) under such experimental conditions. Mondolfo (4) sketched the Al-rich part of the Al-Rb phase diagram which is analogous to that of the Al-K system. He estimated the solubility of Rb in liquid Al to be about 0.05 mol % at 933 K. He did not report experimental details. The phase diagram of the Al-Rb system is probably similar to that of the Al-Na system.

References

1. Czochralski, J.; Kaczynski, J. *Wiad. Inst. Metal. Metalozn.* 1937, *4*, 18.
2. Pinchback, T.R.; Winkel, J.R.; Matlock, D.K.; Olson, D.L. *Nucl. Technol.* 1981, *54*, 201.
3. Kohli, R. *Material Behavior and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 345.
4. Mondolfo, L.F. *Metallography of Aluminium Alloys*, Wiley-Chapman & Hall, N.Y., 1943, p. 36.

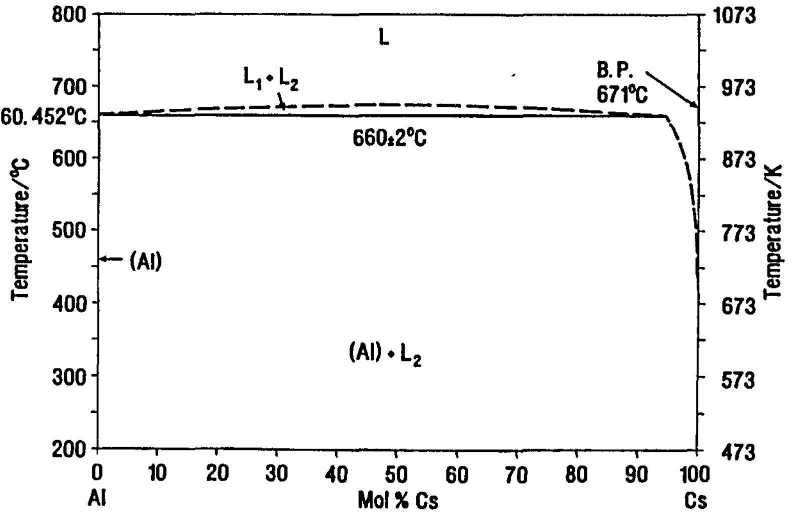
COMPONENTS:	EVALUATOR:
(1) Aluminum; Al; [7429-90-5] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1991

CRITICAL EVALUATION:

Czochralski and Kaczynski (1) performed some experiments with Al-Si(13%) alloy and Cs; the results may be interpreted that Al and Cs are immiscible in the liquid state.
Bushmanov and Yatsenko (2) investigated the central part of the Al-Cs phase diagram between 20 and 78 mol % Al using high purity starting materials. They determined the monotectic temperature at 933 K and the critical miscibility temperature at 951 K by means of thermal analysis. It is difficult to interpret the small difference between these two temperatures. The extrapolation of the liquidus to the monotectic temperature allows the estimation of the solubility of Al in liquid Cs to be a few mol % Al. This level appears to be too high in comparison with the Al-Na, Al-K and Al-Rb systems. Thermal effects might be disturbed by boiling of Cs, since the boiling point of Cs is rather low (944 K). Another complication may be caused by the possible interaction of the Ta container used with liquid Al, as it can be concluded from the Al-Ta phase diagram (9).
Mondolfo (10) estimated a solubility of Cs in liquid Al of about 0.05 mol % Cs at 933 K without giving experimental details. He sketched the Al-rich part of the Al-Cs phase diagram.
Further work on the Al-Cs system was only concerned with compatibility problems. Sedelnikov and Godneva (3) observed a slight dissolution of Al in liquid Cs at 573 K; the corrosive attack of the liquid metal was more pronounced after increasing the O content of Cs to 0.8 mol %. Al was covered with an unidentified layer in this case. The layer might be $CsAlO_2$, according to Kohli (4).
It was also reported that Al_2O_3 is slightly (5,6) or undetectably (6,7) soluble in liquid Cs. Tepper and Greer (8) detected as much as 0.59 and 2.7 mol % Al in Cs after Al_2O_3 compatibility tests with Cs at 1255 and 1644 K, respectively. It is not possible to extract solubility data from these numbers. This paper is, therefore, not compiled.
The partial and doubtful Al-Cs phase diagram published by (2) is redrawn below.

A doubtful value of the solubility of Al in liquid Cs

T/K	*soly*/mol % Al	source
933	~3 monotectic	(2) extrapolated



References

1. Czochralski, J.; Kaczynski, J. *Wiad. Inst. Metal. Metalozn.* 1937, *4*, 18.
2. Bushmanov, V.D.; Yatsenko, S.P. *Izv. Akad. Nauk SSSR, Met.* 1981, no 5, 202.
3. Sedelnikov, V.A.; Godneva, M.M. *Issledovaniya Fiziko-Khimicheskikh Svoistv Soendenienii Redkikh Elementov*, Nauka, Leningrad, 1978, p. 56.
4. Kohli, R. *Material Behavior and Physical Chemistry in Liquid Metal Systems*, H.U. Borgstedt, Ed., Plenum, N.Y., 1982, p. 345.
5. Winslow, P.M. *US Atom.Ener.Comm.Rep. CONF-650411*, 1965, p. 334.
6. 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.
7. Keddy,, E.S. *US Atom.Ener.Comm.Rep. LAMS-2948*, 1963.
8. Tepper, F.; Greer, J. *US Air Force Rep. ASD-TDR-63-824*, Part I, 1963; Rep. *MSAR-63-61*, 1963.
9. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am. Soc. Mater., Materials Park, 1990, p.218.
10. Mondolfo, L.F. *Metallography of Aluminium Alloys*, Wiley-Chapman & Hall, N.Y., 1943, p. 15.

COMPONENTS:

(1) Aluminum; Al; [7429-90-5]

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

ORIGINAL MEASUREMENTS:

Bushmanov, V.D.; Yatsenko, S.P.

Izv. Akad. Nauk SSSR, Met. **1981**, no 5, 202-204.

VARIABLES:

Temperature: 940-948 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Al-Cs liquidus were determined; the values were read out from the figure by the compilers.

t/°C	*soly*/mol % Al
667	20
671	29
673	40
674	45
675	53
674	57
673	60
672	71
670	78

The monotectic temperature was found to be 660±2 °C. Solid Al does not dissolve detectable amounts of Cs at temperatures $\leq$ 660 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Weighed amounts of Al were placed in a Ta crucible. Liquid Cs was added into the crucible from a syringe in an inert atmosphere inside a glove box. The crucible was hermetically closed. The amount of Cs was determined by means of weighing after the addition of this element. The differential thermal analyses of the alloys were performed by means of a scanning microcalorimeter.

SOURCE AND PURITY OF MATERIALS:

Al: 99.999 % pure.
Cs: 99.99 % pure.

ESTIMATED ERROR:

Solubility: precision better than ± 1 %; read out procedure ± 0.5 mol %.
Temperature: precision ± 2 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Gallium; Ga; [7440-55-3] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland March 1991

CRITICAL EVALUATION:

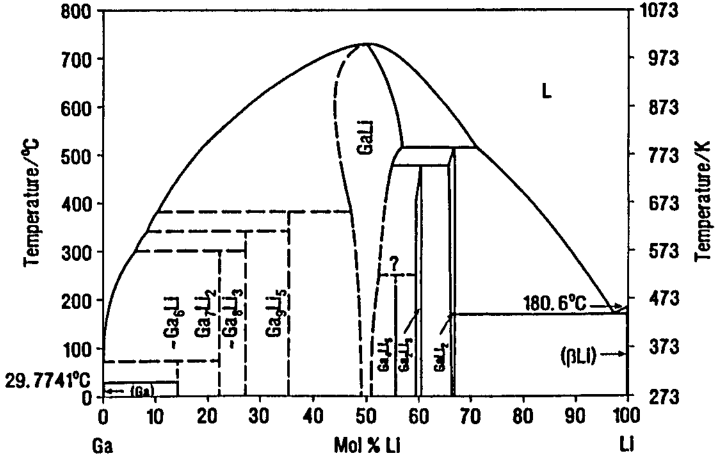
The Ga-Li phase diagram was investigated in four laboratories using thermal analysis (2-7), and in one (9) by means of the emf method after coulometric generation of the alloys. Some determinations of the solubility of Li in liquid Ga were performed by Yatsenko et al. (3-5). The liquidus results were in agreement within ± 10 K (maximum up to ± 20 K) with the exception of three values of Thümmel and Klemm (2) in the most Ga-rich region. The position of the eutectic on the Li-rich side as determined by Yatsenko et al. (3-5) was questioned by Itami et al. (7) who confirmed the value of (2).

Most of the authors (1-6) reported the maximum of the liquidus at 50 mol % Ga, while Itami et al. (7) determined it at 52.9 mol % Ga. The single result of Schneider and Hilmer (1), placing the melting point of GaLi to 1033 K is distinctly overestimated, probably due to the contamination with Fe of the container material. The work is, therefore, not compiled. The suggested values of the solubility of Ga in liquid Li were selected on the basis of mean values of the publications (2-7). Nb and Ta crucibles were used in (2) and (3) which may be the reason for contamination of the Ga-Li alloys, since Nb and Ta form intermetallics with Ga (8). No compound formation should be expected if Mo (6,9) or W (7) are used as container materials. Thus, the system should be less disturbed by contamination in (6,9) and (7).

The results concerning the number and composition of the intermetallic phases in this system are more divergent. Thümmel and Klemm (2) did not detect any intermediate phase in the Ga-rich range. The diagram given by Itami et al. (7) is ambiguous in this aspect. The most convincing picture of the phase relations was sketched by Tillard-Charbonnel and Belin (6) with four intermediate phases: Ga_6Li, Ga_7Li_2, Ga_8Li_3, and Ga_9Li_5. There is general agreement on the formation of GaLi, Ga_2Li_3, and $GaLi_2$ in the Li-rich region. An assessed phase diagram of the Ga-Li system is shown in the figure below. It is mainly based on (10); the Ga-rich side is, however, modified according to (6).

Tentative values of the solubility of Ga in liquid Li

T/K	*soly*/mol % Ga	source
443	3 (eutectic)	(2,7)
473	5	(2,7) interpolated
573	11	(2,7) interpolated
673	18	(2,7) interpolated
773	27	(2-5,7)
788	28 peritectic	(2,3,7) mean value
873	35	(2,7)
973	44	(2-5,7)
1000	50	(2-7) mean value



References

1. Schneider, A.; Hilmer, O. *Z. Anorg. Chem.* 1956, *2286*, 97.
2. Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* 1970, *376*, 44.
3. Yatsenko, S.P.; Chuntonov, K.A.; Alyamovskii, S.I.; Dieva, E.N. *Izv. Akad. Nauk SSSR, Met.* 1973, no. 1, 185.
4. Yatsenko, S.P.; Chuntonov, K.A.; Alyamovskii, S.I.; Dieva, E.N. *Obshchie Zakonomernosti v Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, p. 55.
5. Yatsenko, S.P. *J. Chim. Phys.* 1977, *74*, 836.
6. Tillard-Charbonnel, M.; Belin, C. *Compt. Rend. Ser. II*, 1988, *306*, 1161.
7. Itami, T.; Shimoji, H.; van der Lugt, W. *J. Less-Common Met.* 1989, *152*, 75.
8. Yatsenko, S.P. *Gallii*, Nauka, Moskva, 1974, p. 110.
9. Wen, C.J.; Huggins, R.A. *J.Electrochem.Soc.* 1981, *128*, 1636.
10. Sangster, J.; Pelton, A.D. *J.Phase Equil.* 1991, *12*, 33.

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Thümmel, R.; Klemm, W.

Z. Anorg. Chem. 1970, *376*, 44-63.

VARIABLES:

Temperature: 443-1000 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Ga-Li system was determined. The experimental results were read out by the compilers.

t/°C	*soly*/mol % Ga	t/°C	*soly*/mol % Ga	t/°C	*soly*/mol % Ga
172	2	555	32	714	45.5
189	4	575	33	721	47
221	6	588	34	726	49
245	8	595	35	727	50
276	10	607	36	727	51
300	11.5	620	37	726	52
333	14	630	38	722	53
366	16	639	39	712	56
389	18	651	39	685	62
421	20	660	39.5	594	71.5
449	22	670	41	451	82
471	24	682	41.5	333	88.5
489	26	689	43	170	95.5
505	28	700	43.5		
528	30	709	44		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The surface oxide was mechanically removed from Li which was then washed with CH_3OH, dried and weighed in a glass ampoule under Ar atmosphere. The alloys were prepared in a Ta crucible. These were further placed in a quartz tube container. The thermal analysis of the alloys was performed by means of Pt/Pt-Rh and Ni/Cr-Ni thermocouples. Each measurement was repeated 2-3 times.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.99 % pure from Degussa.
Li: 99.0 % pure from Merck.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 1 mol %.
Temperature: read-out procedure ± 3 K.

REFERENCES:

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Yatsenko, S.P.; Chuntonov, K.A.; Alyamovskii, S.I.; Dieva, E.N.
Izv. Akad. Nauk SSSR, Met. 1973, no. 1, 185-188.

VARIABLES:

Temperature: 423-1011 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Ga-Li system was determined. The results were also reported in (1,2).

t/°C	*soly*/mol % Ga	*t*/°C	*soly*/mol % Ga
150[a]	99	731	53
200[a]	98	738	48
250[a]	95	725	47
300[a]	93	707	45
346[a]	92	696	42
398	89	674	39
481	85	636	37
537	79	589	34
639	71	549	31
678	66	503	27
726	58	400	20
		253	10

[a] from solubility measurements

The melting temperature of Li was found to be 180 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in Nb or Ta crucibles. All operations were performed in a He atmosphere. Cooling and heating curves were recorded in a differential thermal analysis apparatus. Some results were obtained by means of saturation liquid Ga with Ga-Li alloy at selected temperature. The saturated liquid phase was analyzed for the contents of both metals.

SOURCE AND PURITY OF MATERIALS:

Ga: purity "GI-000"
Li: 99.96 % pure ^{7}Li isotope
He: purified by means of low temperature adsorption on activated charcoal

ESTIMATED ERROR:

Solubility: precision ± 1 mol % (in thermal analysis).
Temperature: precision ± 3K.

REFERENCES:

1. Yatsenko, S.P.; Chuntonov, K.A.; Alyamovskii, S.I.; Dieva, E.N. *Obshchie Zakonomernosti v Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, p. 55-59.
2. Yatsenko, S.P. *J. Chim. Phys.* 1977, *74*, 836-842.

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Wen, C.J.; Huggins, R.A.

J. Electrochem. Soc. 1981, *128*, 1636-1641.

VARIABLES:

Temperature: 663-858 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Li in liquid Ga was determined.

t/°C	*soly*/mol % Li	*soly*/mol % Ga[a]
390	10.0	90.0
415	12.6	87.4
500	18.2	81.8
585	26.1	73.9

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Coulometric titrations and measurements of the electromotive force were made using the cell:
Al, Li-Al / LiCl-KCl (eutectic) / Ga-Li.
Li was coulometrically introduced into liquid Ga which was kept in a Mo bucket. The temperature was measured by means of Chromel/Alumel thermocouples which were sheathed with Type 304 stainless steel tube. The experiments were carried out in a He atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.9999 % pure from United Minerals & Chemicals.
LiCl-KCl eutectic: probably as in (1), purchased from Lithium Corp. of America.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: stability ± 1 K.

REFERENCES:

1. Wen, C.J.; Boukamp, B.A.; Huggins, R.A.; Weppner, W. *J. Electrochem. Soc.* 1979, *126*, 2258-2262.

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Tillard-Charbonnel, M.; Belin, C.

Compt. Rend.. Ser. II 1988, *306*, 1161-1164.

VARIABLES:

Temperature: 491-991 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ga in liquid Li was determined; the values were read out from the figure by the compilers.

t/°C	*soly*/mol % Ga	t/°C	*soly*/mol % Ga	t/°C	*soly*/mol % Ga
218	98.0	495	80.5	665	64.9
272	95.3	524	78.3	672	63.9
329	92.3	540	76.6	687	61.2
378	90.0	554	75.5	683	60.3
383	89.8	561	74.8	694	59.8
407	88.9	583	72.9	697	58.3
423	87.7	602	71.4	706	56.8
432	86.6	622	69.4	711	56.0
435	86.2	631	67.2	712	55.1
458	85.3	640	66.7	715	52.6
465	84.1	655	66.1	718	50.3
484	82.3				

The melting point of Ga was found to be 29.5 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Precisely weighed amounts of both metals were placed in a Mo capsule which was hermetically enclosed and heated to high temperature in order to reach homogenization. The capsule was placed in an apparatus for the differential thermal analysis. The apparatus was calibrated on the melting points of Ga, In, Li, Sn, Pb, Te, and Al. Cooling and heating curves of the samples were recorded. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.99 % pure from Alusuisse.
Li: 99.94 % pure from Cogema.
Ar: contained less than $1 \cdot 10^{-4}$ %N_2, O_2, and H_2O, each.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.3 mol %.
Temperature: read-out procedure ± 2 K.

REFERENCES:

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Itami, T.; Shimoji, H.; van der Lugt, W.

J. Less-Common Met. 1989, *152*, 75-86.

VARIABLES:

Temperature: 443-1012 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus temperatures of the Ga-Li system were determined.

t/°C	*soly*/mol %Ga	*t*/°C	*soly*/mol % Ga	*t*/°C	*soly*/mol % Ga
207	96.0	683	64.9	601	35.4
307	93.5	717	59.9	610	35.1
316	92.0	725	57.2	584	34.4
351	90.0	731	54.9	596	33.9
396	88.2	735	53.8	571	33.2
428	86.0	739	52.9	540	31.2
434	85.0	736	51.8	529	30.1
473	83.7	733	50.9	516	28.0
486	82.3	731	50.1	488	25.1
499	81.4	731	48.5	434	20.4
514	80.1	730	47.1	401	18.5
535	79.0	717	45.9	394	16.4
555	77.5	700	44.5	364	15.2
582	75.1	693	42.8	295	10.2
612	71.9	672	42.3	234	7.3
640	69.8	673	42.3	196	5.2
653	68.5	667	40.9	170	3.0
660	66.7	659	40.0	177	1.4
662	66.7	647	38.0		

The melting point of Ga was found to be at 29,8 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Weighed amounts of Ga and Li were placed in a larger W crucible and heated to a temperature 100 K higher than the presumed melting point of the homogeneous alloys. The alloys were frequently agitated and then quenched on a Cu plate. A small part of the selected alloy was placed in a small W crucible which was inserted in to a standard stainless steel crucible of the differential thermal analysis apparatus. The samples were prepared and handled in a He-filled glove box. The apparatus was calibrated on the melting points of In, Pb, Zn, Mg, and Al. The heating and cooling cycles of each sample were several times repeated.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.999 % pure from Mitsubishi Metal, Ventrap.
Li: 99.98 % puree from Koch Light Inc..

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± few K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Gallium; Ga; [7440-55-3] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1992

CRITICAL EVALUATION:

Puschin et al. (1) observed in their early work on the Ga-Na system that the alloy Ga85Na15 is insoluble in liquid Ga at 723 K. Gilfillan and Bent (2) determined the solubility of Na in liquid Ga to be 0.003 mol % Na at 303 K in agreement with the results of Yatsenko et al. (3) who performed similar experiments at 523 to 694 K. They reported an equation (see corresponding data sheet) which fits their results and the single value of (2). The slope of this solubility equation is less steep than the results of thermal analyses of Feschotte and Rinck (4-6) and Itami et al. (7) which were obtained at higher Na contents. A plot of logarithm of solubility versus reciprocal temperature is no longer linear in this range.

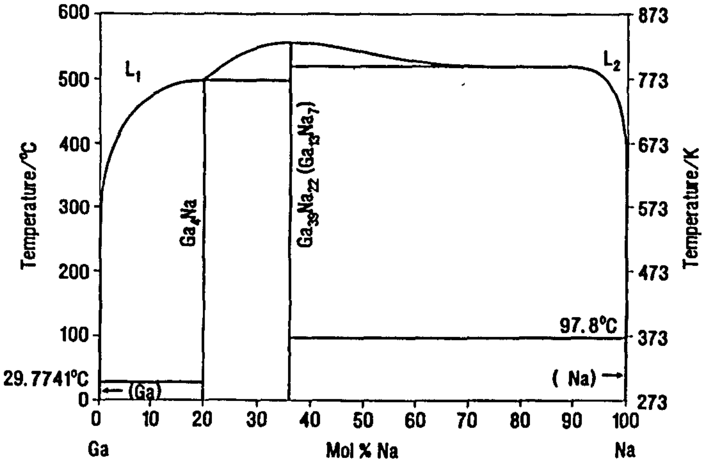
The liquidus data from (3,4,7,8) are in agreement within ± 5 K; only the value at 635.4 K from (7) deviates significantly. The data of Zhakopov et al. (9) agree with these data. They claim, however, a miscibility gap between 10 and 45 mol % Ga at a monotectic temperature of about 800 K. Such a miscibility gap was not supported by recent experimental studies (7,10). Thermodynamic modelling (11) gives no evidence for such a gap. Theoretical considerations of Pelton and Larose (12) indicate the possibility of immiscibility in the Ga-Na system even at higher temperature than 893 K. Finally, Feschotte (14) concluded that the immiscibility of this system may be observable in experiments under metastable conditions at a temperature about 20 K below the liquidus which was observed under equilibrium conditions.

The phase diagrams of Ga systems (13) indicate the possibility of side reactions of Ga and container metals: Fe in (6,7) and Ta in (3,9). The high-temperature results on Ga-Na alloys may be influenced by such contaminating reactions.

The saturated solutions are in equilibrium with two solid intermetallic compounds: Ga_4Na (7,10,11) (which was first considered as Ga_3Na), and $Ga_{39}Na_{22}$ (7,10) (which was originally formulated as $Ga_{13}Na_7$ or Ga_8Na_5). The assessed Ga-Na phase diagram was presented in (7,10,11,12,14).

Tentative and recommended (r) values of the solubility of Ga in liquid Na

T/K	*soly*/mol % Ga	source
373	0.015	(6)
473	0.06	(6) interpolated
573	0.1	(6)
673	0.5 (r)	(6,9)
773	4	(4)
831	63.5 congruent	(4,7,10) mean value



References

1. Puschin, N.A.; Stepanovic, S.; Stajic, V. *Z. Anorg. Chem.* 1932, *209*, 333.
2. Gilfillan, E.S.; Bent, H.E. *J. Am. Chem. Soc.* 1934, *56*, 1661.
3. Yatsenko, S.P.; Chuntonov, K.A., Bushmanov, V.D.; Dieva, E.N. *Structura Faz, Fazovye Prevashcheniya i Diagrammy Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1974, p.198-201
4. Feschotte, P.; Rinck, E. *Compt. Rend.* 1956, *243*, 1525.
5. Rinck, E.; Feschotte, P. *Compt. Rend.* 1960, *250*, 1489-1491.
6. Feschotte, P. *Ann. Chim.* (Paris) 1961, *6*, 1029.
7. Itami, T.; Shimoji, M.; Meijer, J.A.; Vinke, G.J.B.; Van der Marel, C.; Van der Lugt, W. *Physica* 1986, *142B*, 145.
8. Gambino, M.; Bros, J.P. *Thermochim. Acta* 1973, *6*, 129.
9. Zhakopov, Sh.R.; Chuntonov, K.A.; Ugodnikov, G.G.; Yatsenko, S.P.; Shakarov, Kh.O. *Zh. Fiz. Khim.* 1980, *54*, 1023.
10. Van der Lugt, W.; Itami, T.; Shimoji, M.; Meijer, J. *Z. Phys. Chem., N.F.* 1988, *156*, 617.
11. Zabdyr, L.A. *Monatsh. Chem.* 1988, *119*, 31.
12. Pelton, A.D.; Larose, S. *Bull. Alloy Phase Diagr.* 1990
13. Yatsenko, S.P. *Gallii*, Nauka, Moskva, 1974, p. 110.
14. Feschotte, P. *J.Less-Common Met.* 1991, *175*, L5.

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Gilfillan, E.S.; Bent, H.E.

J. Am. Chem. Soc. 1934, *56*, 1661-1663.

VARIABLES:

One temperature: 303 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Na in liquid Ga at 30 °C was determined to be about 0.001 mass % Na. The correspondent value of 0.003 mol % Na was recalculated by the compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Carefully purified Ga was weighed and placed in an evacuated apparatus. The metal was brought in contact with the electrolyte, a solution of NaI in ethylamine. Controlled amounts of Na were electrolytically reduced on a Ga cathode and introduced into the Ga phase. At the end of the electrolysis the electrode potentials were measured. The solubility of Na in Ga was probably defined by a break on the potential versus logarithm of Na concentration curve.

SOURCE AND PURITY OF MATERIALS:

Ga: "purchased from Ch. Hardy, N.Y.", further purified with H_2SO_4, heated in a quartz ampoule in H_2 atmosphere at 1073 k and in vacuo at 1273 K.
Na: nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Feschotte, P.; Rinck, E.

Compt. Rend. 1956, *243*, 1525-1528.

VARIABLES:

Temperature: 709-829 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Ga-Na system was determined. The values were read out from the figure by the compilers.

t/°C	*soly*/mol % Ga	*t*/°C	*soly*/mol % Ga	*t*/°C	*soly*/mol % Ga
456	1.7	521	29.5	550	68.0
484	2.3	523	33.1	549	68.8
498	3.1	525	36.5	548	69.4
501	4.0	526	38.6	546	70.6
505	4.5	527	41.1	537	73.7
509	5.0	530	43.3	534	74.8
512	6.0	533	47.0	526	76.4
517	8.0	540	50.7	520	77.3
515	9.0	546	53.3	504	79.4
516	10.0	551	56.0	496	80.9
517	11.5	554	57.2	494	83.7
520	12.1	555	59.7	489	86.3
520	15.5	556	60.4	477	90.2
520	19,5	556	61.6	452	94.0
520	22.7	556	62.4	436	96.0
521	26.8	555	63.9		

The melting points of Ga and Na were measured at 29.80 ± 0.02 and 98 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a Pyrex glass crucible in an Ar atmosphere. The thermal analysis of the melts was performed by means of a thermocouple cladded with an Fe tube. The heating and cooling curves were measured with Chromel/Alumel and Pt/Pt-Rh thermocouples in the temperature range 250 to 923 K.

SOURCE AND PURITY OF MATERIALS:

Ga: spectral purity
Na: "commercial", distilled after cleaning the surface by mechanical removal of layers.

ESTIMATED ERROR:

Solubility: precision ± 0.2 mol %, read out ± 0.5 mol %.
Temperature: precision ± 1 K.

REFERENCES:

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Feschotte, P.

Ann. Chim. (Paris) 1961, *6*, 1029-1070.

VARIABLES:

Temperature: 373 - 748 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ga in liquid Na was determined; the values were read out from the figure by the compilers.

t/°C	*soly*/mol % Ga	t/°C	*soly*/mol % Ga
100	0.015 [a]	380	0.26
170	0.04	400	0.48
224	0.09	415	0.56
294	0.10	430	0.84
300	0.10	442	0.92
332	0.14	450	1.18
354	0.18	475	2.0

[a] numerical result

The melting point of Ga was measured at 29.76 ± 0.01 °C. The solid equilibrium phase Ga_8Na_5 was separated and analyzed (magnetic susceptibility, conductivity, thermal analysis, calorimetry, density).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Ga with an excess of Na was placed in a Pyrex glass crucible which was operated in a furnace controlled by means of a thermocouple under Ar atmosphere. The alloys were heated to the upper temperature and kept on this level for equilibration. Then a sample of the liquid alloy was drawn using a thin Pyrex tube (1). The furnace was then cooled to another equilibration temperature. Another sample was taken at this lower temperature. The samples were dissolved in alcohol. The solutions were evaporated to dryness, the residue was dissolved in H_2O. Ga was spectrophotometrically determined using aluminon (2).

SOURCE AND PURITY OF MATERIALS:

Ga: 99.9 % pure from Pechiney.
Na: "commercial", distilled after purification by means of mechanical removal of surface layers.

ESTIMATED ERROR:

Solubility: precision ± 10 %.
Temperature: precision ± 3 K.

REFERENCES:

1. Rinck, E.; Feschotte, P. *Compt. Rend.* 1960, *250*, 1489-1491.
2. Rinck, E.; Feschotte, P. *Compt. Rend.* 1955, *240*, 1618-1620.

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Gambino, M.; Bros, J.P.

Thermochim. Acta 1973, *6*, 129-136.

VARIABLES:

Temperature: 723 and 773 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Three liquidus points were determined on the basis of calorimetric measurements.

t/°C	*soly*/mol % Ga on the Ga-rich side	 on the Na-rich side
450	~ 95 [a]	~ 3 [a]
500	79.5	

[a] read out from the figure by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A differential microcalorimetric method was applied to measure the liquidus points. The two metals were mixed in an Ar atmosphere after breaking the ampoules, the heat of mixing was measured by means of the microcalorimeter. The break point of the curve which relates the heat effect to the composition of the alloy corresponds to the liquidus point.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.9999 % pure
Na: 99.97 % pure
Ar: purified over molecular sieves and by means of heating with Ti-Zr alloy to 1100 K.

ESTIMATED ERROR:

Solubility: precision ± 0.5 mol %, compiler's evaluation from the figure ± 1 mol %.
Temperature: precision ± 1 K.

REFERENCES:

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Yatsenko, S.P.; Chuntonov, K.A., Bushmanov, V.D.; Dieva, E.N.
Struktura Faz, Fazovye Prevrashcheniya i Diagramy Sostoyaniya Metallicheskikh Sistem, Nauka, Moskva, 1974, p.198-201.

VARIABLES:

Temperature: 523-825 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Ga-Na system was determined; the values were read out from the figure by the compilers.

$t/°C$	*soly*/mol % Ga [a]	$t/°C$	*soly*/mol % Ga [b]
520	20	421	97.8
520	29	358	98.3
521	40	300	98.9
536	49	250	99.5
546	54		
549	56		
551	59		
552	60		
540	69		
508	77		

[a] from thermal analysis
[b] from solubility experiments

The solubility equation which was reported was tested by the compilers:

$$\log(soly/\text{mol \% Na}) = 2.955 - 1760\,(T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The measurements were performed in a Ta crucible in which Na was introduced in vacuo, while Ga was filled in under Ar atmosphere. The crucible was hermetically closed by means of a laser welded Ta tap. Differential thermal analysis was performed while the samples were slowly heated or cooled. The solubility of Na in liquid Ga was determined by sampling at selected temperature and analysis of Ga-rich alloys. The analytical method was not reported.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.999 % pure.
Na: 99.8 % pure.

ESTIMATED ERROR:

Solubility: read out procedure ± 1 mol %.
Temperature: precision ± 1 K; read out procedure ± 5 K.

REFERENCES:

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Zhakupov, Sh.R.; Chuntonov, K.A.; Ugodnikov, G.G.; Yatsenko, S.P.; Shakarov, Kh.O.
Zh. Fiz. Khim. 1980, *54*, 1023-1025.

VARIABLES:

Temperature: 678-827 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Ga-Na system was determined. The results were mainly presented in a figure; the values were read out by the compilers.

t/°C	*soly*/mol % Ga	*t*/°C	*soly*/mol % Ga
405	0.47 [a]	539	53
475	0.92 [a]	548	58
515	10	554	61
543	22 [b]	551	64
547	27	524	75
534	35	493	87
519	43 [b]		

[a] reported in numerical form
[b] from measurements of the electrical conductivity

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed in Ta crucibles to which Na was given in vacuo and Ga in an inert gas atmosphere. The crucible was hermetically closed by means of a Ta tap and laser welded. The magnetic susceptibility was measured between room temperature and 1073 K in steps of 3-5 K in the neighbourhood of a phase transition. The temperatures of the liquidus were indicated by sharp changes of the slope of the susceptibility versus temperature. Details concerning the measurements of the electric conductivity were not reported.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.999 % pure.
Na: 99.8 % pure.

ESTIMATED ERROR:

Solubility: read-out procedure ± 1 mol %.
Temperature: precision ± 1 K; read-out procedure not better than ± 7 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gallium; Ga; [7440-55-3] (2) Sodium; Na; [7440-23-5]	Itami, T.; Shimoji, M.; Meijer, J.A.; Vinke, G.J.B.; van der Marel, C.; van der Lugt, W. *Physica B* 1986, *142*, 145-151.
VARIABLES:	**PREPARED BY:**
Temperature: 635.4 - 832.0 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Ga-Na system was determined.

t/°C	*soly*/mol % Ga	*t*/°C	*soly*/mol % Ga	*t*/°C	*soly*/mol % Ga
362.2	2.0	532.3	44.8	528.1	75.3
493.3	5.1	528.5	46.9	519.1	77.8
514.2	10.0	536.4	50.3	499.7	79.8
520.1	13.0	545.2	55.7	498.9	82.1
517.2	15.3	553.4	60.9	487.8	86.2
517.3	22.4	554.1	61.8	486.9	87.9
520.2	27.4	558.8	63.0	477.3	89.3
500.6	31.8	555.9	63.7	460.4	92.0
521.1	34.8	553.7	64.7	432.1	95.0
518.2	40.2	552.3	67.1		
530.0	43.4	548.7	70.4		

The results are graphically reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The samples were prepared inside a dry He glove box. Weighed amounts of Ga and Na were melted at 973 K and quenched on a Cu plate. A small part of the resulting alloy was inserted into a standard stainless steel crucible of an apparatus for differential thermal analysis. The temperature was calibrated at the melting points of In, Pb, Mg, and Al. Repeated heating and cooling curves of each alloy sample were recorded.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.999 % pure from Mitsubishi Metal, Ventrap.
Na: 99.93 % pure from Merck.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. van der Lugt, W.; Itami, T.; Shimoji, M.; Meijer, J. *Z. Phys. Chem., N.F.* 1988, *156*, 617-621.

COMPONENTS:	EVALUATOR:
(1) Gallium; Ga; [7440-55-3] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1990

CRITICAL EVALUATION:

Gilfillan and Bent (1) determined the solubility of K in liquid Ga as 7 10^{-6} mol % K at 32 °C. This value is not in agreement with the results of Yatsenko and Chuntonov (2,3) obtained between 673 and 783 K. Their solubility equation results in a solubility of $3 \cdot 10^{-4}$ mol % K at 32 °C. The data of (2,3) show acceptable concordance with their thermal analyses. These data seem to be more reliable than the estimation of (1). Still higher values of the solubility of K in Ga in the range of several mol % were obtained in thermal analyses of Feschotte (4) and Tillard-Charbonnel et al. (9). This region of the system Ga - K needs further study in order to confirm the results which seem to be the most reliable (2,3).

The solubility of Ga in K was determined between 485 and 823 K by (4) by means of chemical analyses. Extrapolation of these results to higher temperatures leads to values which are more than one order of magnitude lower than the results of the thermal analyses by the same author (2,3) and (9). (9) repeated detailed investigations of the Ga-K system and explained that due to the high volality of K a temperature corresponding to the vaporization of K is measured instead of the critical temperature of miscibility in the composition range of the miscibility gap.

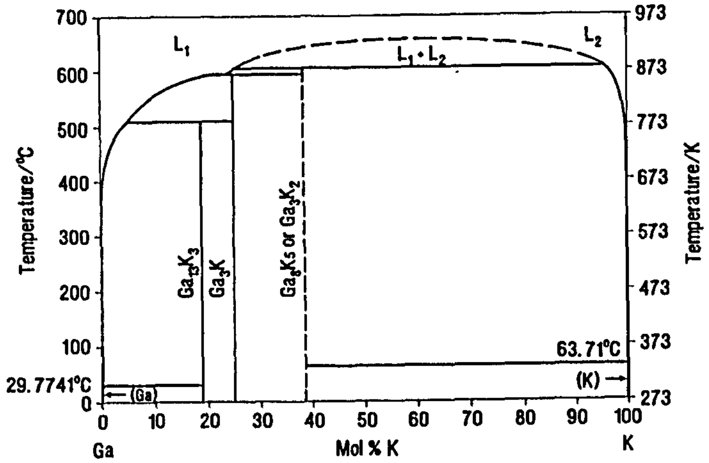
The thermal analyses of (4,5) do not agree with those of (2,3) in the region of the equiatomic composition. Although both groups of investigators detected a miscibility gap, the discrepancy of temperatures is as high as 60 K. A partly oxidation and evaporation of K under the conditions of the experiments of (4,5) may explain the discrepancies. (2,3) introduced errors into the redrawn results of (4,5). The use of Fe (4) and Ta (2) containers may cause contamination of the Ga-K alloys, since Fe and Ta form intermetallics with Ga (8).

The data reported allow only tabulation of doubtful solubility results.

Statements about the composition of the equilibrium solid phases are inconsistent. While (4,5) mentioned the existence of the phases Ga_8K_5 and Ga_4K, (2,3) identified Ga_2K and Ga_3K by means of X-ray diffraction. Van Vucht (6) showed in X-ray studies that the Ga-K solid alloy containing 20 to 40 mol % K has the crystal structure of Ga_3K. Ga_4K has to be considered as $Ga_{13}K_3$ and the existence of Ga_8K_5 cannot be excluded (7). The critical temperature of miscibility in the liquid alloys is claimed to be much higher than reported by (4,5) according to Pelton and Larose (7). The schematic phase diagram of the Ga-K system based on (9) is presented below. The use of the formula Ga_3K_2 is suggested instead of Ga_8K_5.

Doubtful values of the solubility of Ga in liquid K.

T/K	*soly*/mol % Ga	source
473	0.002	(4) extrapolated
573	0.005	(4) interpolated
673	0.010	(4) interpolated
773	0.015	(4) interpolated
874	3.5 peritectic	(4),(9) mean value
~ 908	~60 critical point	(9) extrapolated



References

1. Gilfillan, E.S.; Bent, H.E. *J. Am. Chem. Soc.* 1934, *56*, 1661.
2. Yatsenko, S.P.; Chuntonov, K.A. *Izv. Akad. Nauk SSSR, Met.* 1973, no. 6, 182.
3. Yatsenko, S.P.; Chuntonov, K.A., Bushmanov, V.D.; Dieva, E.N. *Struktura Faz, Fazovye Prevrashcheniya i Diagramy Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1974, p.198.
4. Feschotte, P. *Ann. Chim.* (Paris) 1961, *6*, 1029.
5. Rinck, E.; Feschotte, P. *Compt. Rend.* 1961, *252*, 3592.
6. van Vucht, J.H.N. *J. Less-Common Met.* 1985, *108*, 163.
7. Pelton, A.D.; Larose, S. *Bull. Alloy Phase Diagr.* 1990, *11*, 343.
8. Yatsenko, S.P. *Gallii*, Nauka, Moskva, 1974, p. 110.
9. Tillard-Charbonnel, M.; Chouoibi, N.E.; Belin, C. *Compt.Rend., Ser.II* 1990, *311*, 69.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gallium; Ga; [7440-55-3] (2) Potassium; K; [7440-09-7]	Gilfillan, E.S.; Bent, H.E. *J. Am. Chem. Soc.* 1934, *56*, 1661-1663.
VARIABLES:	**PREPARED BY:**
One temperature: 305 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of K in liquid Ga was determined as $4 \cdot 10^{-6}$ mass % K and about $7 \cdot 10^{-6}$ mol % K (as calculated by the compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Ga was carefully purified and weighed and then placed in an evacuated system. K was distilled before it was admitted into an electrolytic cell. The electrolyte was probably composed of KI in ethylamine. Measured amounts of K were electrolyzed into Ga and the concentration potentials were measured after the electrolyses were finished. The solubility of K was probably estimated from a break of the potential vs. logarithm of K concentration curve.

SOURCE AND PURITY OF MATERIALS:

Ga: "purchased from Ch. Hardy, N.Y.", purified by means of contacting with H_2SO_4, heated in a quartz ampoule in H_2 atmosphere at 1073 K and in vacuo at 1273 K.
K: nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Feschotte, P.

Ann. Chim. (Paris) 1961, *6*, 1029.

VARIABLES:

Temperature: 485-945 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Ga in liquid K was determined. The results were read out from the figure by the compilers.

t/°C	*soly*/mol % Ga [a]	*t*/°C	*soly*/mol % Ga [b]	*t*/°C	*soly*/mol %Ga [b]
212	0.004	599	3	608	75
290	0.005	624	4	601	80
343	0.009	670	15 [c]	584	86
480	0.014	672	17.5 [c]	576	91
550	0.019	667	29 [c]	541	94
		652	42 [c]	513	96
		637	54 [c]	497	97
		620	63.5 [c]	472	98
		618	69		

[a] from solubility measurements [b] from thermal analysis [c] also presented in (3)

The melting points of Ga and K were measured to be at 29.76 ± 0.01 and 63 °C, respectively. The solubility of K in liquid Ga was seen to be very small.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Ga and an excess of K were placed in a Pyrex crucible enclosed in Ar atmosphere of a furnace controlled by a thermocouple. The melt was heated to the upper test temperature and kept there for 15 min for equilibration. A sample of the alloy was taken by means of a small Pyrex tube (1). Another sample was drawn after cooling and equilibration at a lower temperature. The samples were dissolved in alcohol, the solutions were evaporated, and the residue was dissolved in H_2O. Ga was spectrophotometrically determined with aluminon (2). The thermal analysis was recorded between 250 and 973 K. The thermocouples of Chromel/Alumel or Pt/Pt-Rh were shielded with an Fe tube to protect them against the molten alloys.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.9 % pure from Pechiney.
K: commercial grade; distilled after mechanically cleaning its surface.

ESTIMATED ERROR:

Solubility: precision ± 10 % or lower.
Temperature: precision ± 5 K.

REFERENCES:

1. Rinck, E.; Feschotte, P. *Compt. Rend.* 1960, *250*, 1489-1491.
2. Rinck, E.; Feschotte, P. *Compt. Rend.* 1955, *240*, 1618-1620.
3. Rinck, E.; Feschotte, P. *Compt. Rend.* 1961, *252*, 3592-3594.

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Yatsenko, S.P.; Chuntonov, K.A.

Izv. Akad. Nauk SSSR, Met. 1973, no. 6, 182-187.

VARIABLES:

Temperature: 673-887 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Liquidus points of the system were determined. The data were taken from the figure by the compilers.

t/°C	*soly*/mol % Ga	t/°C	*soly*/mol % Ga	t/°C	*soly*/mol % Ga
604	5.0	612	49.4	595	67.8
608	10.0	610	54.0	595	69.8
609 [a]	14.2	608	59.8	590	79.8
611	19.8	603	61.6	580	85.1
613	29.6	601	63.5	565	90.0
614 [a]	39.4	596	65.1		

[a] numerical result

The same results were also reported in (1).
The experimentally measured solubility of K in liquid Ga between 400 and 510 °C was expressed by the equation:

$$\log (soly/\text{mol \% K}) = 0.57 - 1250\,(T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared and investigated in a hermetically closed Ta crucible. The metals were mixed and melted inside this crucible under a minimized free volume above the alloy. A thermocouple recorded the temperature, when the heating and cooling curves were performed. The solubility of K in Ga was determined after sampling a Ga-rich alloy fraction at a selected temperature. The samples were analyzed by means of an unknown method.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.999 % pure.
K: 99.8 % pure.

ESTIMATED ERROR:

Solubility: precision ± 0.5 %; read-out procedure ± 1 mol %.
Temperature: precision ± 2 K; read-out procedure ± 5 K.

REFERENCES:

1. Yatsenko, S.P.; Chuntonov, K.A., Bushmanov, V.D.; Dieva, E.N. *Struktura Faz, Fazovye Prevrashcheniya i Diagramy Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1974, p.198-201.

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Tillard-Charbonnel, M.; Chouaibi, N.E.; Belin, C.

Compt. Rend., Ser. II 1990, *311*, 69-72.

VARIABLES:

Temperature: 723-894 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Ga-K system was determined. The data were read out from the figure by the compilers.

t/°C	*soly*/mol %Ga	*t*/°C	*soly*/mol % Ga	*t*/°C	*soly*/mol % Ga
576	1.3	620	62.9	589	81.9
618	5.8	620	63.9	585	85.4
620	8.8	619	66.6	580	88.1
621	31.8	619	68.0	567	91.2
621	44.7	619	69.1	520	95.4
620	54.2	617	72.6	495	97.1
620	56.9	593	75.0	470	98.8
620	59.8	592	76.6	450	99.0
620	60.9	591	78.2		
620	62.0	590	80.3		

The miscibility gap was located between 3.6 and 74 mol % Ga at 601 °C. The miscibility line is flattened between 9 and 70 mol % Ga due to the evaporation of K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Ga-K alloys were prepared by melting precisely weighed samples of the two metals in a Mo crucible. The crucible was enclosed in a hermetically sealed capsule in which the alloy was homogenized by heating at high temperature. The capsule was then placed in an apparatus for the differential thermal analysis. The apparatus was calibrated on the melting points of Ga, In, Li, Sn, Pb, Te, and Al. Heating and cooling curves were recorded. All experiments were performed under Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.99 % pure from Alusuisse.
K: "for synthesis" from Merck, further purified from K_2O by filtration, centrifugation and zone remelting.
Ar: with contents of $< 1 \cdot 10^{-4}$ % N_2, O_2, H_2O.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.2 mol %.
Temperature: read-out procedure ± 2 K.

REFERENCES:

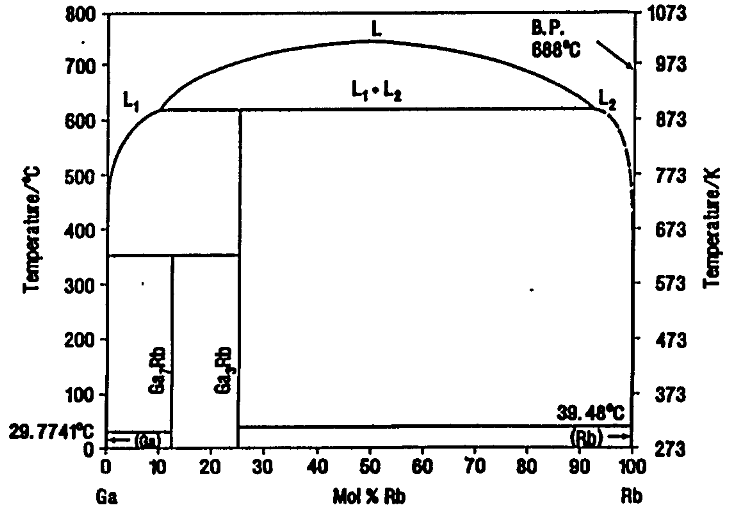
COMPONENTS:	EVALUATOR:
(1) Gallium; Ga; [7440-55-3] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1992

CRITICAL EVALUATION:

The solubility of Ga in liquid Rb at temperatures below 873 K is unknown. There are three sets of determinations of the liquidus line of the Ga-Rb system by Thümmel and Klemm (1), Yatsenko et al. (2,3) and by Tillard-Charbonnel et al. (8). Experiments between pure Ga and 40 mol % Rb were performed by (1), while (2,3 and (8) covered the whole range of compositions.
The results of the liquidus temperatures based on thermal analysis of (1), (2,3) and (8) do not agree. The data of (2,3) are about 80 K higher than the results of (1), and a similar difference occurs between (8) and (2,3). The critical temperature for the miscibility gap extending from 8 to 90 mol % Ga was found to be about 1013 K (8). The alloy samples used in (1) and (2,3) may be contaminated due to interactions of the Ta crucibles with Ga at higher temperatures, as can be concluded from the Ga-Ta phase diagram (7). Ta containers may also be corroded by Rb contaminated with O (see the Ta-Rb system). The hermetically closed Mo containers which were used by (8) may be less reactive with the components of the Ga-Rb system. Thus, competing reactions seem to be less probable in the study of (8). The data of this source are, therefore, preferred.
The number and stoichiometry of solid phases in equilibrium with the solutions need further study. The proposed phase Ga_6Rb was identified as Ga_7Rb by means of X-ray diffraction methods; the peritectic melting point is 623 K. Ga_3Rb was more precisely confirmed in X-ray diffraction experiments (4,5) than by the thermal analysis. The Ga_4Rb phase which was proposed in (1) was not confirmed in subsequent studies. Other Rb-rich phases, such as Ga_2Rb or Ga_8Rb_5, which were reported in (1) and (3) were not found in recent work (4,5). The tentative Ga-Rb phase diagram is published in (8) and is reproduced below. The very high critical temperature which was predicted in (6) is not experimentally confirmed.

Tentative solubility data of Ga in liquid Rb

T/K	*soly*/mol % Ga	source
873	4	(2,3)
894	8 monotectic	(8)
973	27	(8) interpolation
1013	50 critical point	(8)



References

1. Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* , 1970, *376*, 44.
2. Yatsenko, S.P.; Chuntonov, K.A.; Alyamovskii, S.I. *Izv. Akad. Nauk SSSR, Met.*, 1973, no.3, 233.
3. Yatsenko, S.P.; Chuntonov, K.A.; Alyamovskii, S.I.; Dieva, E.N. *Obshchie Zakonomernosti v Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, 55.
4. Van Vucht, J.H.N. *J. Less-Common Met.* 1985, *108*, 163.
5. Chuntonov, K.A.; Yatsenko, S.P. *Izv. Akad. Nauk SSSR, Neorg. Mater.* 1985, *21*, 1164.
6. Pelton, A.D.; Larose, S. *Bull. Alloy Phase Diagr.* 1990, *11*, 354.
7. Yatsenko, S.P. *Gallii*, Nauka, Moskva, 1974, p. 110.
8. Tillard-Charbonnel, M.; Chouaibi, N.E.; Belin, C. *Compt. Rend., Ser. II* 1991, *312*, 1113.

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Thümmel, R.; Klemm, W.

Z. Anorg. Chem. 1970, *376*, 44-63.

VARIABLES:

Temperature: 748-896 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ga-rich part of the liquidus in the Ga-Rb system was determined; the values were read out from the figure by the compilers.

t/°C	*soly*/mol % Ga
475	95
529	90
566	86
578	84
584	82
588	79.5
594	78
600	76.5
602	75
606	72
609	70
613	68
617	64
619	61
623	30 monotectic

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a Ta crucible with a Fe stopper which served to inhibit the evaporation of Rb. The experiments were performed in an Ar atmosphere. The thermal analytical cooling and heating curves were recorded immediately after the alloys reached the maximum temperature of 893 K. Pt/Pt-Rh and Ni/Cr-Ni thermocouples were used for the thermal analyses.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.99 % pure from Degussa
Rb: vacuum distilled

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 1 mol %.
Temperature: read-out procedure ± 3 K.

REFERENCES:

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Yatsenko, S.P.; Chuntonov, K.A.; Alyamovskii, S.I.

Izv. Akad. Nauk SSSR, Met. 1973, no.3, 233-235.

VARIABLES:

Temperature: 823-939 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Ga-Rb system was determined; the values were read out from the figure by the compilers.

t/°C	*soly*/mol % Ga
601	4
612	6
620	10
620	16
666	51
620	65
620	71
619	75
618	79
603	85
573	92
550	96

The miscibility gap at 620 °C is expected to be between 10 and 65 mol % Ga.
The same results were also presented in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a small Ta crucible which was hermetically welded. The heating and cooling curves of the thermal analysis were measured triply by means of a Chromel/Alumel thermoelement. The samples were chemically analyzed for their Rb and Ga contents. The crucible was opened in anhydrous acetone, H_2O was gradually added. Rb was determined by alkalimetric titration and Ga by gravimetric analysis.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.999 % pure
Rb: 99.9 % pure
Ta: "TVCh" designed

ESTIMATED ERROR:

Solubility: accuracy ±0.5 mol %; read-out procedure ±2 mol %.
Temperature: precision ±2 K; read-out procedure ±7 K.

REFERENCES:

1. Yatsenko, S.P.; Chuntonov, K.A.; Alyamovskii, S.I.; Dieva, E.N. *Obshchie Zakonomernosti v Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, p. 55-59.

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Tillard-Charbonnel, M.; Chouaibi, N.E.; Belin, C.

Compt. Rend., Ser. II 1991, *312*, 1113-1116.

VARIABLES:

Temperature: 811-1008 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Ga-Rb system was redetermined. The data were read out from the figure and calculated by the compilers.

t/°C	*soly*/mol % Ga	*t*/°C	*soly*/mol % Ga
538	98.8	683	78.2
564	97.7	719	67.0
591	96.6	735	49.3
600	94.9	706	31.0
618	90.9	609	5.7
675	81.0		

The liquid miscibility gap was determined to be situated between 8 and 90 mol % Ga at 621 °C.
The formula of two equilibrium solid phases were determined: Ga_7Rb and Ga_3Rb.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Ga-Rb alloys were prepared by melting precisely weighed samples of the two metals in a Mo crucible. This crucible was heated to high temperature in a hermetically closed capsule in order to homogenize the system. The capsule was placed in an apparatus for differential thermal analysis which was calibrated on the melting points of Ga, In, Li, Sn, Pb, Te, and Al. Heating and cooling curves were recorded. All operations were performed in an Ar atmosphere.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.9999 % pure from Rhône-Poulenc.
Rb: prepared by reduction of RbCl with Ca in vacuum, distilled and tested for purity by means of thermal analysis.
Ar: with contents of < 1.10-4 % N_2, O_2, and H_2O, from Jacomex.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.2 mol %.
Temperature: read-out procedure ± 1 K.

REFERENCES:

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

EVALUATOR:

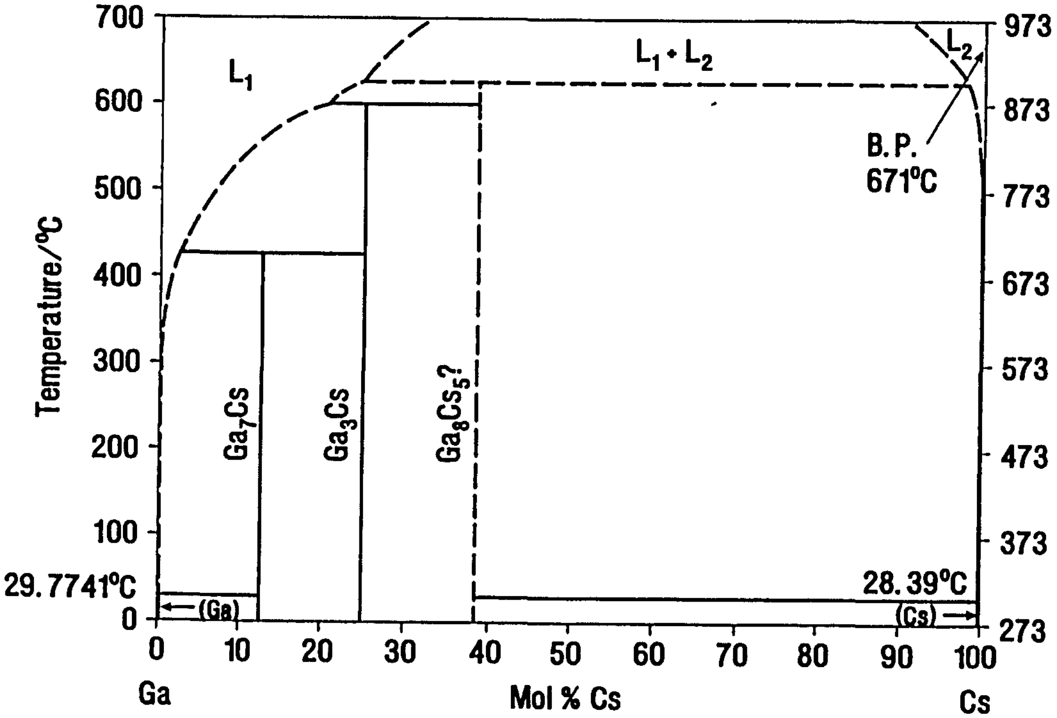
H.U. Borgstedt, Kernforschungszentrum Karlsruhe,
Germany
C. Guminski, Dept. of Chemistry, Univ. of Warsaw,
Poland
January 1991

CRITICAL EVALUATION:

The experimental results of studies of the liquidus of the Ga-Cs system are not in agreement. Thümmel and Klemm (1) defined two points on the Ga-rich side at 623 and 807 K. Yatsenko et al. (2,3) extended the investigations to the whole range of compositions. They found the liquidus on the Ga-rich side to be about 40 K higher than in (1). They proposed different compositions of the intermetallic phases which are in equilibrium with the liquid solution. Bushmanov and Yatsenko (4) further improved their earlier results, thus defining the liquidus at a 10 K higher temperature. An extended miscibility gap of two Ga-based and one Cs-based liquids was established by this group of workers (2-4). However, the existence of this gap between 2 and 73 mol % Ga with a critical temperature at only 32 K above the monotectic seems to be unlikely. This region needs further study.
Since Ta reacts with Ga at high temperatures (7), the use of Ta crucibles may lead to contamination of the Ga-Cs alloys. If Cs is additionally contaminated with O, a corrosive attack on Ta was observed (see the Ta-Cs system).
Further research seems also necessary to identify the solid phases in equilibrium with the liquids. The compound Ga_4Cs was proposed by (1) but was later formulated as Ga_6Cs (2,3), Ga_5Cs (4) and finally as Ga_7Cs (5). Ga_3Cs has a measurable range of stoichiometry up to 90 mol % Cs as (5) has evaluated from X-ray diffraction studies, in which Ga_2Cs could not be detected. The existence of Ga_8Cs_5, which was claimed in (1), needs confirmation. The tentative Ga-Cs phase diagram is published in (6) and is redrawn below..

Doubtful solubility data of Ga in liquid Cs

T/K	*soly*/mol% Ga	source
873	2 monotectic	(4)



References

1. Thümmel, R.; Klemm, W. Z. *Anorg. Chem.* 1970, *376*, 44.
2. Yatsenko, S.P.; Chuntonov, K.A. *Izv. Akad. Nauk SSSR, Met.* 1973, no.6, 182.
3. Yatsenko, S.P.; Chuntonov, K.A.; Alyamovskii, S.I.; Dieva, E.N. *Obshchie Zakonomernosti v Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, 55.
4. Bushmanov, V.D.; Yatsenko, S.P. *Izv. Akad. Nauk SSSR, Met.* 1981,no.5, 202.
5. van Vucht, J.H.N. *J. Less-Common Met.* 1985, *108*, 163.
6. Pelton, A.D.; Larose, S. *Bull. Alloy Phase Diagr.* 1990, *11*, 354.
7. Yatsenko, S.P. *Gallii*, Nauka, Moskva, 1974, p. 110.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gallium; Ga; [7440-55-3] (2) Cesium; Cs; [7440-46-2]	Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* 1970, *376*, 44-63.
VARIABLES:	**PREPARED BY:**
Two temperatures: 623 and 807 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Ga-rich part of the Ga-Cs liquidus was determined. The data were read out by the compilers.

t/°C	*soly*/mol % Ga
350	93.9
534	90

The peritectic temperature was found at about 549 °C. The equilibrium solid phases were identified as Ga_4Cs and Ga_8Cs_5.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a Ta crucible with a Fe stopper which served to inhibit the evaporation of Cs. The content of Cs had to be kept constant in these studies. The alloys were covered by a protective Ar atmosphere. Thermal analysis was performed by means of measuring cooling curves which were recorded by Pt/Pt-Rh and Ni/Ni-Cr thermocouples.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.99 % pure from Degussa.
Cs: vacuum distilled.
Ar: not specified.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.5 mol %.
Temperature: read-out procedure ± 3 K.

REFERENCES:

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Yatsenko, S.P.; Chuntonov, K.A.

Izv. Akad. Nauk SSSR, Met. 1973, no.6, 182-187.

VARIABLES:

Temperature: 833-905 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Ga-Cs system was determined over the range 5 to 91.3 mol % Ga. The data were read out from the figure by the compilers.

t/°C	*soly*/mol % Ga	t/°C	*soly*/mol % Ga
593	5.0	586	67.8
614	19.5	586	71.1
630	30.0	586	73.0
632	50.0	586	76.2
608	59.5	584	80.5
597	64.2	576	87.6
586	66.4	560	91.3

A miscibility gap was found between 8 and 66.7 mol % Ga.
The same results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Ga-Cs alloys were prepared in a small Ta crucible which was hermetically closed by means of welding. The alloy was filling almost the whole volume of the crucible. The container with the alloys was heated to about 900 K and kept at this temperature for 2-3 hours. Thermal analysis was performed by recording cooling and heating curves. The temperature was measured with a Chromel/"Kopel" thermocouple.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.999 % pure.
Cs: 99.9 % pure.
Ta: "TVCh".

ESTIMATED ERROR:

Solubility: nothing specified; read-out procedure ± 0.5 mol %.
Temperature: precision ± 2 K; read-out procedure ± 3 K.

REFERENCES:

1. Yatsenko, S.P.; Chuntonov, K.A.; Alyamovskii, S.I.; Dieva, E.N. *Obshchie Zakonomernosti v Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, 55-59.

COMPONENTS:

(1) Gallium; Ga; [7440-55-3]

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

ORIGINAL MEASUREMENTS:

Bushmanov, V.D.; Yatsenko, S.P.

Izv. Akad. Nauk SSSR, Met. 1981,no.5, 202-204.

VARIABLES:

Temperature: 869-905 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Ga-Cs liquidus line were determined; the results were read out from the figure by the compilers.

t/°C	*soly*/mol % Ga	*t*/°C	*soly*/mol % Ga
607	4.5	619	65
613	10	614	68
625	20	610	71
627	23	603	73.5
632	38.5	598	77.5
632	42	596	81
622	63		

The solubility gap was estimated to be between 3 and 25 mol % Ga at 600 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared and studied in a hermetically closed Ta or steel crucible. All operations were performed in an inert gas atmosphere. Cs was introduced into the crucible as liquid from a glass syringe. The container was nearly completely filled with the alloys. Differential thermal analysis of alloys was carried out in a differential scanning calorimeter.

SOURCE AND PURITY OF MATERIALS:

Ga: 99.999 % pure.
Cs: 99.99 % pure.

ESTIMATED ERROR:

Solubility: accuracy better than ± 1 %.
Temperature: precision ± 2 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Indium; In; [7440-74-6] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1992

CRITICAL EVALUATION:

The liquidus of the In-Li system was determined by four groups of investigators who used thermal analysis (1-4). Gasior and Schwitzgebel (5) investigated the central part of the liquidus by emf studies at 800 to 900 K. A single determination was performed by Wen and Huggins (6) who used emf measurements on an alloy prepared by coulometric titration. Other workers performed only the measurement of the melting point of InLi: 898 K (7), 903 K (8), and 908 K (9). The latter publications are not compiled, since they contained only a few experimental points.

The first study of Grube and Wolf (1) may be considered as preliminary. The liquidus points reported in (1) were up to 20 K higher than the values in (2-4) on the Li-rich side. They were up to 60 K below those in (2-4) on the In-rich side. The melting point of InLi was, however, correctly positioned. The results of Lamprecht and Crowther (2), Thümmel and Klemm (3), and Alexander et al. (4) show remarkable agreement of the liquidus temperatures (within 10 K) on the Li-rich side of the phase diagram. On the In-rich side, the data of (3) are shifted to a ~25 K higher temperature than in (2) and (4).The result of a solubility of 78.7 mol % In at 688 K (6) is underestimated by about 2 mol %. The values of (5) are in agreement with (2).

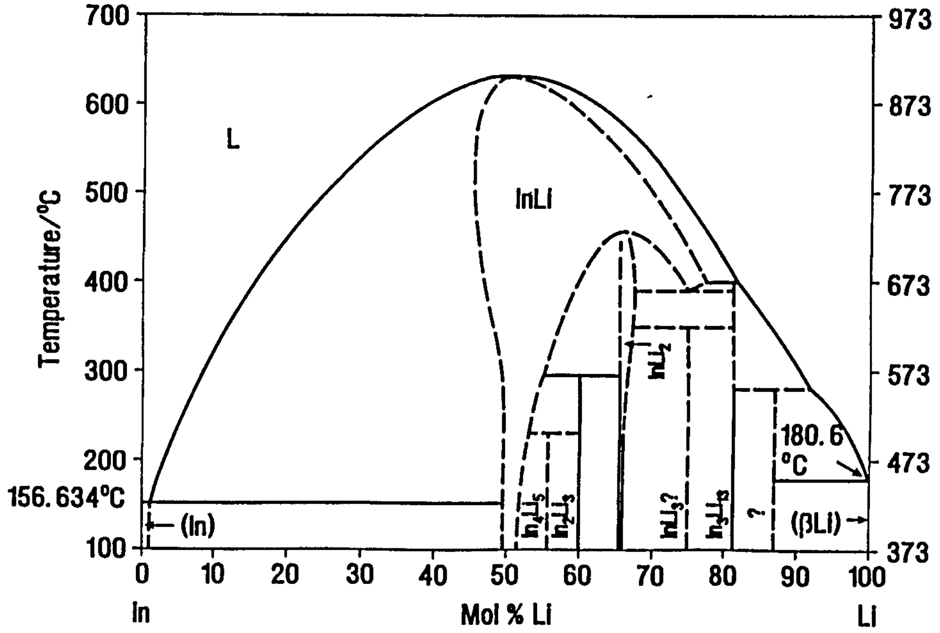
The phase diagram reported in (3) has to be looked on with reservation, since two eutectic temperatures at 425 and 429 K were recorded on both the Li-rich and the In-rich sides. No eutectic but rather peritectic temperatures were found (1,2,4) at about 432 K and 453 K, respectively. The Li-rich section needs further study.

The In-Li phase diagram which was recently published (10) contains 11 intermetallic compounds. Five of these were stable at room temperature; the compound InLi has the highest thermal stability. (3) detected only 8 intermetallics in the system. The earlier publications (1,2) were incomplete in this sense. Several equilibria in this system, presented in (4) and in (10), were not accepted in the most recent review of Sangster and Pelton (11). The diagram redrawn after (4) and (9) should be treated as doubtful in respect to the number and the compositions of the solid phases. The diagram proposed by (11) is a simplified one.

The values of the solubility of In in liquid Li were selected from the data of (2-4), some also from (1), (5), (7), (8) and (9).

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

T/K	*soly*/mol % In	source
473	(r) 4	(2,3)
533	(r) 7 peritectic	(2-4)
573	(r) 10 peritectic	(1-3)
673	(t) 18 peritectic	(2,4)
686	(t) 19 peritectic	(4)
698	(t) 20 peritectic	(2-4)
743	(r) 24 peritectic	(2-4) interpolation
773	(t) 26	(2-4) interpolation
873	(r) 36	(2-5) interpolation
903±5	(r) 50	(1-9)



COMPONENTS:	EVALUATOR:
(1) Indium; In; [7440-74-6] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1992

CRITICAL EVALUATION: (continued)

References

1. Grube, G.; Wolf, W. *Z. Elektrochem.* 1935, *41*, 675.
2. Lamprecht, G.J.; Crowther, P. *J. Inorg. Nucl. Chem.* 1969, *31*, 925.
3. Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* 1970, *376*, 44.
4. Alexander, W. A.; Calvert, L.D.; Gamble, R.H.; Schinzel, K. *Can. J. Chem.* 1976, *54*, 1052.
5. Zintl, E.; Brauer, G. *Z. Phys. Chem.*, B 1933, *20*, 245.
6. Schneider, A.; Hilmer, O. *Z. Anorg. Chem.* 1956, *286*, 97.
7. Aleksandrov, B.N.; Dalakova, N.V.; Moskalets, M.V. *Izv. Akad. Nauk SSSR, Met.* 1987, no. 3, 198.
8. Wen, C.J.; Huggins, R.A. *Mater. Res. Bull.* 1980, *15*, 1225.
9. Yatsenko, S.P. *Indii*, Nauka, Moskva, 1987, p. 93.
10. Sangster, J.; Pelton, A.D. *J.Phase Equil.* 1991, *12*, 37.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Indium; In; [7440-74-6] (2) Lithium; Li; [7439-93-2]	Wen, C.J.; Huggins, R.A. *Mater. Res. Bull.* 1980, *15*, 1225-1234.
VARIABLES:	PREPARED BY:
One temperature: 688 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Li in liquid In at 415 °C was found to be 21.3 mol %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Coulometric titrations and electromotive force measurements were performed in the cell: Al, Al-Li/ LiCl-KCl eutectic/ In-Li. Potentials of the cell were measured directly after the coulometric introduction of Li into liquid In which was contained in a small Mo bucket. The temperature was measured by means of 2 Chromel/Alumel thermocouples. The experiments were performed in a He atmosphere.	In: 99.99 % pure from Alpha, Ventron. LiCl-KCl eutectic: nothing specified.
	ESTIMATED ERROR: Nothing specified. Solubility: ± 0.2 mol % (by the compilers). Temperature: ± 0.2 K (by the compilers).
	REFERENCES:

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Grube, G.; Wolf, W.

Z. Elektrochem. 1935, *41*, 675-681.

VARIABLES:

Temperature: 488-898 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the In-Li system were determined.

t/°C	*soly*/mol % In
215	3
248	5
295	8
303	10
440	20
585	30
604	35
616	40
621	45
625	50
559	60
475	70
219	90

The melting points of In and Li were found to be 155 and 179 °C.
The reported melting temperature of InLi was equal to that determined by (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in microscale from the elements in a small Fe crucible in an Ar atmosphere. The mixtures were melted in a furnace. Thermal analysis was performed in heating and cooling turns. The temperature was measured by means of a calibrated Ni/Ni-Cr thermocouple which was isolated from the molten alloy by a Fe clad.

SOURCE AND PURITY OF MATERIALS:

In: 99.99 % pure from Sachtleben AG.
Li: 99 % pure from Metallgesellschaft AG, it contained 0.62 % K, 0.14 % Na, 0.02 % Fe_2O_3, 0.32 % Li_3N, and 0.05 % SiO_2.
Ar: purified by means of flowing through molten Li.

ESTIMATED ERROR:

Nothing specified

REFERENCES:

1. Zintl, E.; Brauer, G. *Z.Phys.Chem.B* 1933, *20*, 245.

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Lamprecht, G.J.; Crowther, P.

J. Inorg. Nucl. Chem. 1969, *31*, 925-931.

VARIABLES:

Temperature: 454-902 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Liquidus points of the In-Li system were determined; they were read-out from the figure by the compilers.

t/°C	*soly*/mol % In	*t*/°C	*soly*/mol % In	*t*/°C	*soly*/mol % In
193	3.4	553	30.5	622	52.2
262	7.1	551	30.8	613	55.5
300	10.0	567	31.8	594	59.6
334	12.0	572	32.4	562	64.2
362	15.0	581	33.4	543	68.4
393	17.7	586	33.6	497	73.5
418	20.3	588	33.8	451	77.0
453	22.4	609	37.7	406	80.6
486	24.6	619	42.5	345	86.0
519	26.6	624	46.9	266	91.8
536	28.5	629	50.0	181	96.5

The melting points of In and Li were found to be 156.2 and 179 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The oxidized surface of Li was cut off inside a pure Ar glove box, and the Li sample was sealed into a glass ampoule for weighing outside the glove box. The metals were melted together in a stainless steel tube which was suspended in an outer container filled with the pure Ar atmosphere of the glove box. The thermal analysis was performed using Chromel/Alumel thermocouples which were directly inserted into the melts.

SOURCE AND PURITY OF MATERIALS:

In: spectroscopically pure from Johnson-Matthey.
Li: 99.9 % pure from Merck.
Ar: contained below $1 \cdot 10^{-4}$ % H_2O.

ESTIMATED ERROR:

Solubility: read-out procedure ± 0.3 mol %.
Temperature: read-out procedure ± 2 K.

REFERENCES:

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Thümmel, R.; Klemm, W.

Z. Anorg. Chem. 1970, *376*, 44-63.

VARIABLES:

Temperature: 449-908 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus point of the In-Li system were determined; they were read-out from the figure by the compilers.

t/°C	*soly*/mol % In	*t*/°C	*soly*/mol % In	*t*/°C	*soly*/mol % In
400	84	626	42	449	21.5
458	79	621	41.5	438	21
508	75	621	41	422	20
538	70	617	39.5	399	19
553	68	614	38.5	386	18
569	66	609	37.5	377	16.5
579	63.5	603	36.5	362	15.5
592	62	600	35.5	344	14.5
605	60	593	34.5	333	13
617	58	585	34	318	12
622	56	573	33	308	11
628	54.5	563	32	295	9.5
632	53	553	31	275	9
633	52	543	29	258	8
634	50.5	528	28	247	6.5
635	50	516	27	234	6
635	49.5	503	26	208	5
633	47.5	491	24.5	200	4
631	46	478	24	176	2
630	44	463	23		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The oxidized surface of Li was cut off, the metal was washed in CH_3OH, dried and weighed in glass tubes filled with Ar. The alloys were prepared in a Ta crucible in Ar atmosphere. The crucible was placed in a quartz container. Thermal analyses of the alloys were performed, the experiments were 2-3 times repeated. The temperature was measured by means of Pt/Pt-Rh and of Ni/Ni-Cr thermocouples.

SOURCE AND PURITY OF MATERIALS:

In: 99.99 % pure from Duisburger Kupferhütte.
Li: 99.0 % pure from Merck.

ESTIMATED ERROR:

Solubility: read-out procedure ± 0.5 mol %.
Temperature: read-out procedure ± 2 K.

REFERENCES:

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Alexander, W. A.; Calvert, L.D.; Gamble, R.H.; Schinzel, K.
Can. J. Chem. 1976, *54*, 1052-1060.

VARIABLES:

Temperature: 432-898 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the In-Li system were determined; the values were read out from the figure by the compilers.

t/°C	*soly*/mol % In	t/°C	*soly*/mol % In	t/°C	*soly*/mol % In
220	4.2	485	24.5	622	51.4
250	5.8	520	27.3	614	54.0
265	7.3	539	28.7	607	55.9
319	11.5	557	30.9	595	59.6
394	17.4	566	31.9	570	65.5
404	18.2	572	33.5	528	71.3
409	18.6	587	35.0	481	76.4
430	20.5	593	37.9	399	83.2
440	21.6	614	41.4	262	93.6
458	22.8	624	45.2	158.5	99.6
469	23.8	625	49.2		

The melting points of In and Li were found to be 156.3 and 180.5 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

All operations with the two metals and the alloys were carried out in an Ar atmosphere of a dry box. The alloys were melted in a crucible made of type 416 stainless steel. The metals were filled into the crucible which was then transferred into a furnace and heated in order to melt the metals and alloys. Thermal analyses were performed, the cooling and heating curves were recorded by means of Pt/Pt-Rh thermocouples which were calibrated in pure metals. Samples from the top and the bottom of each ingot were chemically analyzed.

SOURCE AND PURITY OF MATERIALS:

In: 99.999 % pure from Cominco
Li: 99.98 % pure from Koch-Light
Ar: dried over "Drierite", passed through Cu-CuO catalyst, molecular sieves and Ca turnings at 928 K.

ESTIMATED ERROR:

Solubility: accuracy 1-3 % of the nominal composition.
Temperature: accuracy ± 1.5 K; read out procedure ± 2 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Indium; In; [7440-74-6] (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:

The solubility of In in liquid Na as well as the liquidus of the In-Na system have been intensively investigated. However, only qualitative agreement of the data was observed. Heycock and Neville (1) measured a decrease of the freezing temperature of Na due to additions of In. It is questionable whether the eutectic temperature was reached in this study. Heberlein (2) performed thermal analyses of In-rich alloys. This work is preliminary in character. Some of his results do not correspond to the liquidus or to the solidus of the In-Na system. Dasarathy (10) predicted liquid immiscibility in the In-Na system which was not detected by experiments.

Davies (3) and Lamprecht and Crowther (4,5) determined the solubility of In in liquid Na up to 523 and 531 K, respectively. Claar (6) suggested a solubility equation on the basis of these data. The equation cannot be accepted because of its disagreement with thermal analyses above 470 K. It is also not in accordance with the fact that the saturated solutions are in equilibrium with two solid phases, $InNa_2$ and InNa above and below 443 K. The slopes of liquidus can vary around such catatectic temperatures. This is partly reflected in the data of (3), but not of (4,5). Thus, either the solubility data of (3) and (4,5) are underestimated at higher temperatures, or the data which were gained by thermal analyses (4),(7),(14), magnetic susceptibility (15) or electrical resistivity measurements (8,9) were overestimated. The problem cannot be solved without further experiments.

Pelton and Larose (13) calculated the liquidus of the system on the basis of thermodynamic modelling in a review of this system. They found the liquidus to be situated between the scattered data of (3),(4,5) and (4),(7),(8,9),(14),(15). The results of (4),(7),(8),(14),(15) and partly of (2) between 10 and 60 mol % In are scattered up to 30 K. We give preference to the data of (4),(14) and (15), since the results of (8) give only temperature limits.

There are also disagreements on the solid phases in this system. (2) reported the formation of In_3Na, In_2Na, and InNa in the In-rich range. Two phases were observed in the study (4): InNa with a very high melting point above 823 K, and $InNa_3$ melting at 658 K. Thermal analyses performed by (5) found In_3Na_2 as the most stable solid phase, while the melting points of InNa and $InNa_3$ were corrected to significantly lower temperatures. InNa, In_8Na_5 and $InNa_2$ are formed according to Thümmel and Klemm (7). These phase relations were confirmed by Bushmanov (11) who reported the invariant temperature of In_8Na_5 at 723, InNa at 638 K and $InNa_2$ at 443 K (catatectic) and 568 K (peritectic). These data and a small smoothed phase diagram were reported in a secondary source, which is, therefore, not compiled. Aleksandrov et al. (12) measured the In-rich eutectic temperature at 423 K and the melting point of In_8Na_5 at 718 K. The information on experimental details was not sufficient to prepare a data sheet for this work. The phase diagram based on (11) and (13)is presented in the figure. The authors of (13) suggested the formation of a peritectic point on the In-rich side at 433±5 K instead of an eutectic proposed by (11).

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

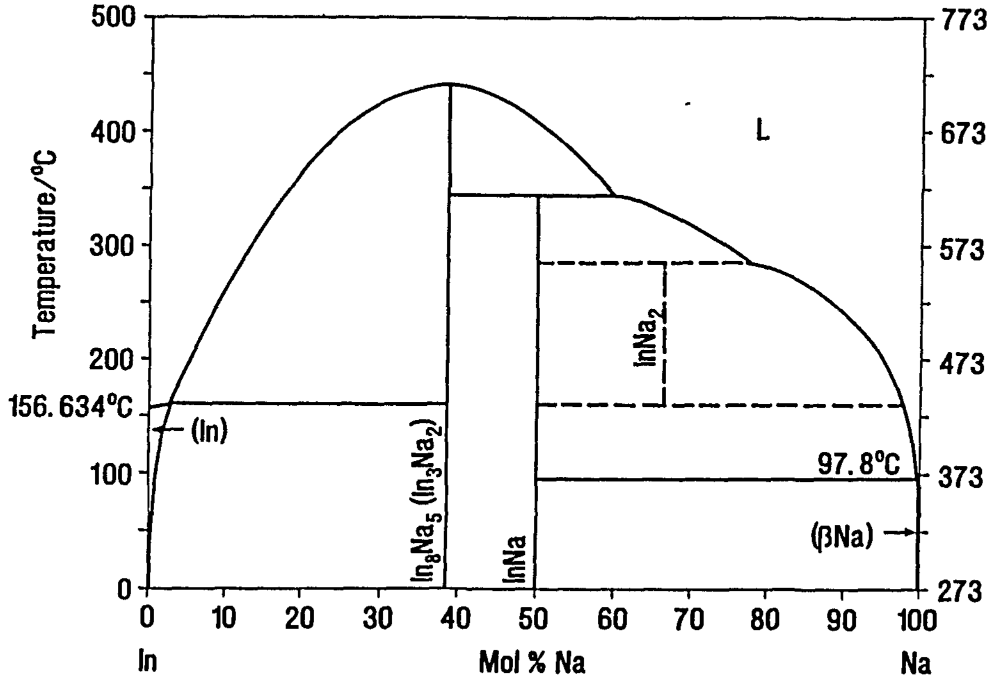
T/K	$soly$/mol % In	source
369	0.4 (t) eutectic	(1)
373	0.4 (t)	(3)
443	2.2 (t) catatectic	(4,5)
473	~5 (d)	(3,4,5,7,8) mean value
~565	~25 (d) peritectic	(4,7) mean value
618	40 (t)	(4,5),(8),(14),(15) mean value
673	48 (t)	(4,7,8) mean value
718	61.5 (t)	(4,7,11,12) mean value

References

1. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1889, *55*, 666.
2. Heberlein, M.F.W. *Trans. ASM* 1952, *44*, 545.
3. Davies, H.A. *Trans. AIME* 1967, *239*, 928.
4. Lamprecht, G.J.; Crowther, P. *J. Inorg. Nucl. Chem.* 1969, *31*, 925.
5. Lamprecht, G.J. *Ph. D. thesis*, Univ. of South Africa, Pretoria, 1966.
6. Claar, T.D. *Reactor Technol.* 1970, *13*, 124.
7. Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* 1970, *376*, 44.
8. van der Marel, C.; van Oosten, A.B.; Meijer, J.A. Vinke, G.J.B.; van der Lugt, W. *Rep. Solid State Phys. Labor.*, Groningen, Netherland, 1986.
9. Meijer, J.A.; Geertsma, W. van der Lugt, W. *J. Phys. F* 1985, *15*, 889.
10. Dasarathy, C. *Trans. AIME* 1969, *245*, 2015.
11. Bushmanov, V.D. *Ph.D thesis*, Ural. Nauchn. Tsentr Akad. Nauk SSSR, Sverdlovsk, 1980; cited by Yatsenko, S.P., *Indii*, Nauka, Moskva, 1987, p. 96.
12. Aleksandrov, B.N.; Dalakova, N.V.; Moskalets, M.V. *Izv. Akad. Nauk SSSR, Met.* 1987, no. 3, 198.
13. Larose, S.; Pelton, A.D. *J.Phase Equil.* 1991, *12*, 371.
14. Neethling, A.J. *South Afric.Atom.Ener.Board Rep. PEL-230*, 1974.
15. Takeda, S.; Tamaki, S. *J. Phys. Soc. Jap.* 1989, *58*, 1484.

COMPONENTS:	EVALUATOR:
(1) Indium; In; [7440-74-6] (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: (continued)



COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Heycock, C.T.; Neville, F.H.

J. Chem. Soc. 1889, *55*, 666-676.

VARIABLES:

Temperature: 369.3-370.7 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The changes of the melting point of Na due to additions of small amounts of In were determined.

t/°C	*soly*/atoms In per 100 atoms Na	*soly*/mol % In[a]	*t*/°C	*soly*/atoms In per 100 atoms Na	*soly*/mol % In[a]
97.51	0.0267	0.0266	96.27	0.3783	0.377
97.31	0.0774	0.0773	96.14	0.4155	0.414
96.32	0.2072	0.207	96.11	0.4267	0.425

[a] calculated by the compilers

The melting point of Na was redetermined to be 97.6 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The test crucible and the stirrer were made of cast Fe. Solid paraffin was placed in that crucible and a weighed quantity of Na was dropped in. The crucible was heated above the melting temperature of Na and In was added. A carefully calibrated thermometer was used. The temperature was then raised to the boiling point of paraffin and the metallic melt was allowed to cool slowly. During this procedure it should be constantly stirred. The temperature of the freezing point was twice determined.

SOURCE AND PURITY OF MATERIALS:

In: nothing specified.
Na: nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.01 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Indium; In; [7440-74-6] (2) Sodium; Na; [7440-23-5]	Heberlein, M. F. W. *Trans. ASM* 1952, *44*, 545-548.
VARIABLES:	**PREPARED BY:**
Temperature: 433-695 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the In - Na liquidus were determined.

t/°F	t/°C [a]	*soly*/mass % Na	*soly*/mass % In [a]
325	163	0.76	96.3
324	162	1.57	92.2[b]
329	165	2.08	90.4[b]
345	174	2.25	89.7[b]
624	329	3.90	83.2
695	368	5.07	78.9
675	357	6.38	74.6[b]
635	335	7.71	70.6[b]
791	422	8.86	67.3
791	422	15.15	52.9
787	419	17.13	49.2

a) calculated by the compilers b) evidently erroneous value

The melting point of In was found to be 155 °C, being 1.5 K lower than the contemporarily accepted value.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed as a part of a larger study and not many details were reported. The alloys were prepared and investigated under N_2 atmosphere. They were first heated to about 755 K and then slowly cooled down to room temperature. The breaking points of the cooling curves were recorded.

SOURCE AND PURITY OF MATERIALS:

Na: nothing specified.
In: nothing specified.
N: "dried".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Davies, H.A.

Trans. AIME 1967, *239*, 928-929.

VARIABLES:

Temperature: 373-541 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of In in liquid Na was determined. The values were read out from the figure by the compilers.

t/°C	*soly*/mol % In
101	0.37
112	0.55
127	0.67
140	0.81
201	3.0
227	5.6
253	9.0
268	10.4

The author proposed a solubility equation which was confirmed by the compilers:

$$\log(soly/\text{mol\% In}) = 3.89 - 1620\ (T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

In and Na were placed in a Zr crucible inside an inert gas box as described in (1). The lid of the crucible was sealed, it was then removed from the box and mounted in a furnace. The melt was kept under slightly higher pressure of He and heated to for 48 hours to 673 K together with the samplers. It was then cooled to the equilibration temperature for 24 hours. The sampler was pre-heated for 15 min in position on top of the melt, immersed into the melt and the alloy was forced through the filter into the sampler by means of increasing the He pressure. After the sampling the temperature was changed and equilibration was performed in 24 hours at the new temperature. The sampler was finally opened, Na was dissolved in C_2H_5OH, the filtrate containing In was dissolved in acid. Aliquots of the solution were analyzed for the In content by wet chemical methods.

SOURCE AND PURITY OF MATERIALS:

In: nothing specified.
Na: from MSA Research as in (1); 99.98 % pure with 0.01 % K, $2.5 \cdot 10^{-3}$ % Rb, $1.5 \cdot 10^{-3}$ % Cs, $1.0 \cdot 10^{-3}$ Si, $2 \cdot 10^{-4}$ % Fe, Al, 2 10^{-3} % C, < $1 \cdot 10^{-3}$ % O and all other impurities 10^{-4} to $5 \cdot 10^{-4}$ %.

ESTIMATED ERROR:

Solubility: read-out procedure ± 0.1 mol %.
Temperature: read-out procedure ± 2 K.

REFERENCES:

1. Weeks, J.R.; Davies, H.A. *The Alkali Metals*, The Chem. Soc., London, 1967, p. 32-44.

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Lamprecht, G.J.; Crowther, P.

J. Inorg. Nucl. Chem. 1969, *31*, 925-931.

VARIABLES:

Temperature: 389.2-712 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of In in liquid Na was determined at several temperatures.

t/°C	*soly*/mass % In	*soly*/mol % In	*t*/°C	*soly*/mass % In	*soly*/mol % In
116.0[a]	3.33	0.69	180.8[a]	11.91	2.64
120.0[c]	3.65	0.75	196.0[b]	15.00	3.41
142.0[b]	5.81	1.28	214.3[a]	19.54	4.65
159.6[c]	8.26	1.78	221.4[a]	21.45	5.19
173.0[b]	10.30	2.24	250 [c]	-	7.9 [d]

[a] heating sequence; [b] cooling sequence; [c] from equilibrium with "Na_3In" intermetallics; [d] read out from the figure by the compilers.

These results were also reported in (1). A solubility equation was established by the authors and confirmed by the compilers.

$$\log(soly/\text{mol \% In}) = 3.939 - 1597(T/\text{K})^{-1}$$

The results of thermal analyses of the liquidus of the In-Na system were read out from the figure by the compilers.

t/°C	*soly*/mol% In	*t*/°C	*soly*/mol% In	*t*/°C	*soly*/mol% In
245	11	360	42.5	436	58
268	17	375	45	439	59.5
285	22	388	47.5	439	60.5
295	26	399	49.5	436	64
305	29.5	419	51.5	432	66
318	33	423	53.5	429	67.5
335	37	428	55	416	70.5
346	39.5	432	56.5	385	77.5

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility apparatus was fabricated of Pyrex glass (1,2). The experiments were performed in a He atmosphere. In with a content of ^{114}In obtained by irradiation was placed a reaction cell, while Na was contained in a second one. The cells were connected by means of a capillary through which Na was transferred, when the pressure was increased. The metals were in contact until the activity of In in liquid Na reached a constant value. The alloy was moved to its primary cell for decay measurements. The variation of the solubility with temperature was determined through the increase of the radioactivity of the saturated solution in Na. The equilibrium was reached from below and above a selected temperature. Thermal analysis was performed in a stainless steel tube in an Ar atmosphere. The temperature was recorded by means of a Chromel/Alumel thermocouple.

SOURCE AND PURITY OF MATERIALS:

In: spectroscopically pure from Johnson-Matthey

Na: 99.9 % pure from Merck, filtered as molten, with a content of $1.1 \cdot 10^{-3}$ % O.

He: purified by passing through molecular sieves, activated charcoal and trapping at the temperature of liquid N.

Ar: purified by passing molecular sieves and BTS catalyst, with a content of $< 10^{-4}$ % O.

ESTIMATED ERROR:

Solubility: standard deviation ± 1.6 %.

Temperature: not specified; read-out procedure in the thermal analysis ± 3 K.

REFERENCES:

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

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Indium; In; [7440-74-6] (2) Sodium; Na; [7440-23-5]	Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* 1970, *376*, 44-63.
VARIABLES:	**PREPARED BY:**
Temperature: 470-721 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the In-Na system were determined; the numbers were digitized from the figure by the compilers.

t/°C	*soly*/mol % In	t/°C	*soly*/mol % In	t/°C	*soly*/mol % In
202	5.8	341	35.1	444	59.9
226	7.6	347	36.6	448	61.5
229	9.4	359	38.2	443	62.7
232	11.2	373	40.3	441	64.2
241	12.5	388	42.1	432	66.0
244	13.9	400	44.4	428	67.7
249	15.2	407	46.2	413	70.1
252	17.5	411	48.0	398	74.2
265	19.6	422	50.2	369	77.4
270	21.3	430	52.1	354	80.2
273	24.6	433	53.5	330	82.5
299	27.4	438	55.4	254	90.0
310	30.2	442	56.6	197	94.2
320	31.8	443	57.0		
331	32.8	445	58.7		

The proportionality of the temperature scale is not good.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were melted in a Ta crucible in order to prepare the alloys. A Fe stopper served to inhibit the evaporation of Na. All experimental operations were performed in an Ar atmosphere. Thermal analysis of the samples was carried out by cooling from 893 K. The temperatures were recorded by the use of Pt/Pt-Rh and Ni/Ni-Cr thermocouples.

SOURCE AND PURITY OF MATERIALS:

In: 99.99 % pure from Duisburger Kupferhütte.
Na: from Merck, oxidized surface was cut off and the metal distilled in high vacuum.

ESTIMATED ERROR:

Nothing specified.
Solubility: read out procedure ± 0.5 mol %.
Temperature: read out procedure ± 3 K.

REFERENCES:

COMPONENTS: (1) Indium; In; [7440-74-6] (2) Sodium; Na; [7440-23-5]	**ORIGINAL MEASUREMENTS:** Neethling, A.J. *South Afric.Atom.Ener.Board Rep. PEL-230*, 1974.
VARIABLES: Temperature: 430.5-710 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the In-Na system were determined.

T/K	*soly*/mol % In	T/K	*soly*/mol % In [a]
470	95	430.5	98.89
545	90	431.4	97.81
646	80	431.7	96.71
698	70	432.3	95.62
710	60	432.7	94.53
679	50	432.7	93.44
619	40	433.0	92.42
591	35	433.2	91.27
572	30		
538	20		
505	10		

[a] electrochemically prepared alloys

The melting point of In was found to be 429.4 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus was made of Pyrex glass. The amounts of metals needed for the alloy preparation were weighed, Na in a sealed capsule which was introduced into an Ar filled glove box. In was added to the Na in the capsule. The mixed metals were heated to about 25 K higher than the expected liquidus temperature and shaken for 30 s. The cooling procedure of the sample was continued to a temperature below the melting temperature of Na. The cooling curves were recorded by means of Chromel/Alumel thermocouples, calibrated on the melting points of Na, Sn, and Pb. The In-richest alloys were prepared by means of coulometric reduction of Na+ from molten NaCl. The samples were chemically analysed, Na by means of flame photometry and In by atomic absorption spectrometry.

SOURCE AND PURITY OF MATERIALS:

In: 99.9 % pure from British Drug House.
Na: analytical grade from Merck, filtrated through 2G filter.
Ar: purified and dried by means of Engelhard purification system, O content less than $1 \cdot 10^{-4}$ %.

ESTIMATED ERROR:

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

REFERENCES:

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

van der Marel, C.; Oosten, A.B.; Meijer, J.A.; Vinke, G.J.B.; van der Lugt, W.
Rep. Solid State Phys. Lab., Groningen, Netherland, 1986.

VARIABLES:

Temperature: 473-723 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The upper limit of liquidus temperatures was estimated on the basis of resistivity measurements in liquid In-Na alloys.

t/°C	*soly*/mol % In
≤450	60.0
≤400	50.0
≤350	40.0
≤300	30.0
≤275	20.0
≤225	10.0
≤200	5.0

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared inside a He-filled glove box in which the measurements were also performed (1). The melt was contained in a Type 321 stainless steel tube. A current was passed through the tube and the molten alloy. The voltage was measured across several sections of the tube to which electrodes were spot-welded. The current was commuted, and the resistances were calculated after the calibration of the apparatus. Since characteristic breaks on the curves of the resistivity vs. temperature were not observed, it may be concluded that the alloys were homogeneous over the whole range of measurements.

SOURCE AND PURITY OF MATERIALS:

In: 99.99 % pure, from Ventron.
Na: nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.
Resistivity: better than ± 2.5 %.
Temperature: nothing specified.

REFERENCES:

1. Meijer, J.A.; Geertsma, W.; van der Lugt, W. *J. Phys.,F* 1985, *15*, 899-910.

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Takeda, S.; Tamaki, S.

J. Phys. Soc. Jap. 1989, *58*, 1484-1485.

VARIABLES:

Temperature: 523-704 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the In-Na liquidus were determined.

t/°C	*soly*/mol % In
250	90
350	78
431	61
342	40
256	20

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloy samples were prepared from the metals in Pyrex glass ampoules sealed in vacuum (1). The magnetic susceptibilities were measured from about 50 K below the expected liquidus temperature to about 773 K. The usual torsion balance method was applied for the measurements. The liquidus temperatures were estimated from break points on the plots of the magnetic susceptibility vs. temperature.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.
Na: probably 99.9 % pure as in (1).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Takeda, S.; Matsunaga, S.; Tamaki, S. *J. Phys. Soc. Jap.* 1984, *53*, 1448-1452.

COMPONENTS: (1) Indium; In; [7440-74-6] (2) Potassium; K; [7440-09-7]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1992

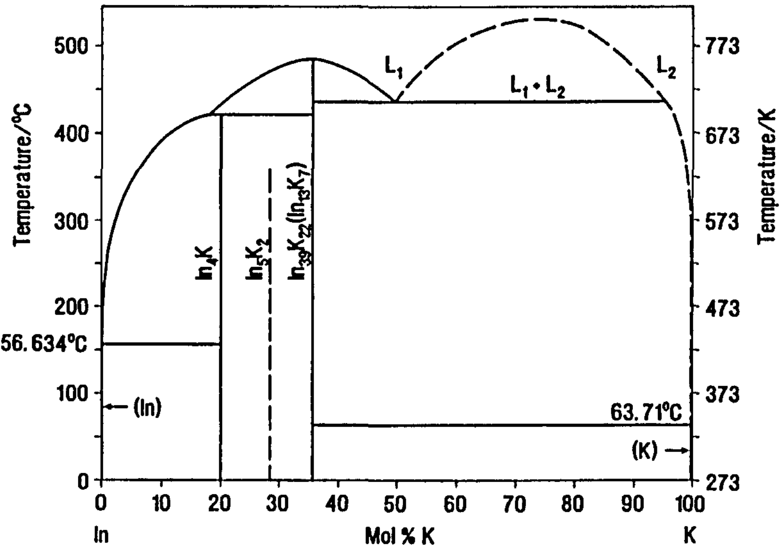
CRITICAL EVALUATION:

Dasarathy (10) predicted the liquid immiscibility of the two metals before any experimental data were known. This miscibility gap in the In-K system was later confirmed by experimental studies (2,3,5,6); its limits are, however, not precisely known.

Thümmel and Klemm (1) missed the liquid miscibility gap on the K-rich side of compositions. The range of liquid immiscibility was established by Yatsenko et al. (2) between ~8 and 41 mol % In at 709 K. Meijer et al. (3) roughly confirmed the results of (2), and established the critical temperature at ~755 K. The critical temperature was claimed to be 731 K and the range of immiscibility from 5 to 40 mol % In according to thermal analyses of Bushmanov (5). Magnetochemical measurements of Melekhov et al. (6,11) resulted in the critical temperature sat 818 K and the limits of miscibility at 707 K between 10 and 52 mol % In. Yatsenko (7) assessed the data and selected the liquidus data of (6) for the K-rich range and of (2) for the In-rich range. The original publications (5) and (6) were not available, and (11) did not present data points, these publications are therefore, not compiled.

The miscibility gap was not found in thermodynamic studies of Dergacheva et al. (8) and of Bykova and Morachevskii (9) in the temperature ranges 733-833 and 720-810 K. The activities of the metals varied smoothly with the alloy composition in the liquid phase.

Since no reliable data on the K-rich side of the liquidus were obtained, solubility values of In in liquid K are not suggested in this assessment (for example, at the monotectic temperature of 709 K, 5 to 10 mol % In may be dissolved according to the different sources). A phase diagram was constructed from (7),(12) and (13). The saturated solution of In in liquid K is in equilibrium with the compound In_8K_5 up to 709 K. This compound was reported as In_2K or In_9K_6 (13); the compositions $In_{39}K_{22}$ or $In_{13}K_7$ were proposed in (12). The homogeneity range of this compound may extend to 5 mol % at 709 K according to (14).



References

1. Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* 1970, *376*, 44.
2. Yatsenko, S.P.; Chuntonov, K.A.; Bushmanov, V.D.; Dieva, E.N. *Struktura Faz, Fazovye Prevrashcheniya i Diagrami Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1974, p. 198.
3. Meijer, J.A.; Geertsma, W.; Van der Lugt, W. *J. Phys.,F.* 1985, *15*, 899.
4. van der Marel, C.; van Oosten, A.B.; Meijer, J.A.; Vinke, G.J.B.; van der Lugt, W. *Rep. Solid State Phys. Labor.*, Groningen. Netherlands, 1986.
5. Bushmanov, V.D. *Ph.D. thesis*, Inst. Khim. Ural. Nauch. Tsentra Akad. Nauk SSSR, Sverdlovsk, 1980; as cited in 7.
6. Melekhov, L.Z., *Ph.D. thesis*, Inst. Khim. Ural. Nauch. Tsentra Akad. Nauk SSSR, Sverdlovsk, 1983; as cited in 7.
7. Yatsenko, S.P. *Indii*, Nauka, Moskva, 1987, p. 97.
8. Dergacheva, M.B.; Kozin, L.F.; Khobdabergenova, G.R. *Izv. Akad. Nauk Kaz. SSR, Ser. Khim.* 1978, no. 5, 21.
9. Bykova, M.A.; Morachevskii, A.G. *Izv. Vyssh. Ucheb. Zaved., Tsvet. Metall.* 1973, no. 1, 91.
10. Dasarathy, C. *Trans.AIME* 1969, *245*, 2015.
11. Melekhov, L.Z.; Yatsenko, S.P.; Chuntonov, K.A.; Zhakupov, Sh. R. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 3, 202.
12. Pelton, A.D.; Larose, S. *Bull.Alloy Phase Diagr.* 1990, *11*, 232.
13. Takenaka, T. Petric, A.; Saboungi, M.L. *J.Phys.Condens.Matter* 1991, *3*, 1603.
14. Okamoto, H. *J.Phase Equil.* 1992, *13*, 217.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Indium; In; [7440-74-6] (2) Potassium; K; [7440-09-7]	Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* **1970**, *376*, 44-63.
VARIABLES:	**PREPARED BY:**
Temperature: 423-747 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the In-K system were determined; they were read-out from the figure by the compilers.

t/°C	*soly*/mol % In	t/°C	*soly*/mol % In	t/°C	*soly*/mol % In
150	99 (eutectic)	447	69.5	438	54
276	94.5	453	68	428	49
347	90	458	65.5	415	44.5
407	82	468	64	412	37.5
422	78.5	474	61	409	34.5
433	75	468	60	408	19.5
436	74	454	56.5	400	9
442	71				

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a Ta crucible with a Fe stopper inhibiting the evaporation of K in Ar atmosphere. The crucible was placed in a quartz container. Thermal analyses of the alloys were performed by means of cooling from 893 K. The temperature was measured by means of Pt/Pt-Rh and of Ni/Ni-Cr thermocouples.

SOURCE AND PURITY OF MATERIALS:

In: 99.99 % pure from Duisburger Kupferhütte.
K: unspecified purity from Merck; the surface was cut off, and the metal was vacuum distilled.
Ar: unspecified.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.5 mol %.
Temperature: read-out procedure ± 3 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Indium; In; [7440-74-6] (2) Potassium; K; [7440-09-7]	Yatsenko, S.P.; Chuntonov, K.A.; Bushmanov, V.D.; Dieva, E.N. *Struktura Faz, Fazovye Prevrashcheniya i Diagramy Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1974, p. 198-201.
VARIABLES:	**PREPARED BY:**
Temperature: 698-753 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some liquidus points of the In-K system were determined; they were read out from the figure by the compilers.

t/°C	*soly*/mol % In	*t*/°C	*soly*/mol % In
425	80	473	59
439	77	467	55
448	75	454	50
463	70	442	45.5
466	65	436	41
480	61.5		

A liquid miscibility gap was observed between ~8 and ~41 mol % In at 436 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a hermetically closed Ta crucible. The free volume over the alloy sample was minimized. Thermal analyses of the alloy samples were performed in the same crucible.

SOURCE AND PURITY OF MATERIALS:

In: 99.999 % pure.
K: 99.8 % pure.

ESTIMATED ERROR:

Nothing specified.
Solubility: read out procedure ± 0.3 mol %.
Temperature: read out procedure ± 2 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Indium; In; [7440-74-6] (2) Potassium; K; [7440-09-7]	Meijer, J.A.; Geertsma, W.; van der Lugt, W. *J. Phys..F* 1985, *15*, 899-910.
VARIABLES:	**PREPARED BY:**
Temperature: 630-775 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some liquidus points of the In-K system were determined; the compositions were tabulated in (1), and temperatures were read out from the figure by the compilers.

t/°C	*soly*/mol % In	t/°C	*soly*/mol % In
357	93.99	502	70.17
378[a]	93	486	65.06
391[a]	90	502[b]	61.54
406	87.81	483	30.01
433	80.00	482	20.05
458	74.75	$\leq$440[b]	10.00

[a] from differential scanning calorimetry [b] from (1)

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The preparation of the alloys as well as the measurements were performed inside a He filled glove box. The melt was contained in a stainless steel tube and a current was passed through the tube and liquid alloy. The voltage was observed across several sections of the tube by means of steel electrodes which were spot welded to the tube wall. The current was commuted. The resistances were calculated after the calibration of the apparatus. The solubility was indicated by characteristic breaks of the curves relating the resistance vs. the composition.

SOURCE AND PURITY OF MATERIALS:

In: 99.99 % pure from Ventron.
K: 99.99 % pure from Kawecki Berylco Industries.

ESTIMATED ERROR:

Solubility: nothing specified.
Resistivity: precision better than ± 2.5 %.
Temperature: nothing specified.

REFERENCES:

1. van der Marel, C.; Vinke, G.J.B.; van der Lugt, W. *Rep. Solid State Phys. Labor.*, Groningen, Netherlands, 1986.

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Takenaka, T.; Petric, A.; Saboungi, M.-L.

J. Phys.,Condens. Matter 1991, *3*, 1603-1612.

VARIABLES:

Temperature: 670-753 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the In-K system were determined.

t/°C	*soly*/mol % In	t/°C	*soly*/mol % In	t/°C	*soly*/mol % In
397	90	475	70	479	62.5
421	85	480	65	478	62
436	80	480	64.5	470	58
459	75	480	63		

A low solubility of In in the K-rich liquid was indicated, a quantitative specification was not presented.

Three equilibrium solid phases were found: In_4K, either In_5K_2 or In_7K_3, and either In_2K or In_9K_5.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus was enclosed in a He-filled glove box. Potential differences of the two cell assembly were measured: Bi-K(8%)/K β-alumina/Pb-K(2%)/K β-alumina/In-K. K β-alumina was prepared by the exchange reaction of Na β-alumina with KCl at 1273 K for 48 h and with KNO_3 at 633 K for 48 h. The completeness of the reaction was checked by atomic absorption spectroscopy. The Pb-K(2%) alloy was contained in a Mo crucible, the reference and working alloys in the K β-alumina tubes. Wires were made of Ta. The assembly was sealed with a Kovar glass joint. The cells were loaded with the alloys inside the glove box. The experiments were started with In, K was added by means of coulometric titration. The emf's were measured after the electrolysis was interrupted. The liquidus data were received from break points on the emf vs. temperature curves.

SOURCE AND PURITY OF MATERIALS:

In: "high purity" from Federated Metals Div. of Asarco Inc..
K: 99.9 % pure with 0.015 % Rb and 0.005 % Na, supplied by Callery Chemical Corp..
He: "high purity".

ESTIMATED ERROR:

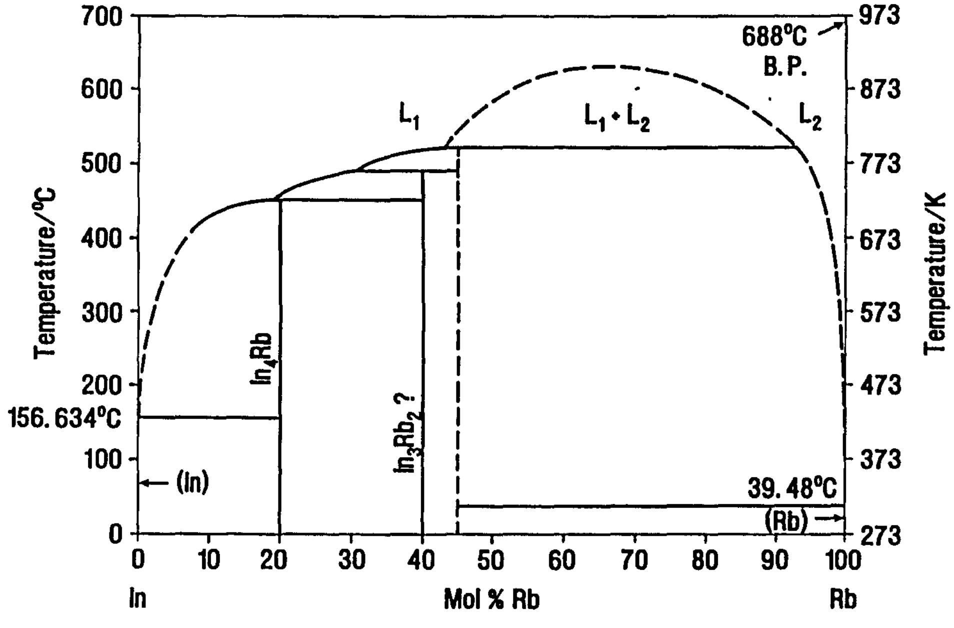
Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Indium; In; [7440-74-6] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1992

CRITICAL EVALUATION:

Dasarathy (3) predicted an immiscibility gap in In-Rb alloys. This miscibility gap was confirmed in experimental studies of the system. The liquidus data reported by Thümmel and Klemm (1) do not agree with those of Yatsenko et al. (2). The liquidus temperatures presented in (2) are up to 40 K higher than in (1). The liquidus of this system was further investigated by means of thermal analyses by Bushmanov (4) and magnetic susceptibility by Melekhov et al. (5,7) according to the compilation of (6). The data of (4) and (5) are not available; their results are published as smoothed curves in (6). Data sheets based on (4) and (5) are, therefore, not compiled. Melekhov et al. (7) gave rough information on the immiscibility range which was located between 10 and 60 mol % In at a monotectic temperature of 762 K. The critical temperature was observed at 859 K. (1) and (2) estimated the miscibility gap between ~6 and 62 mol % In and ~6 and ~55 mol % In, respectively. The corresponding critical temperature was estimated at ~795 K and ~821 K.
Since the studies did not produce consistent results on the Rb-rich side of the liquidus, solubility values of In in liquid Rb cannot be suggested. For example, the compositions of In at the monotectic temperature of 762 K were reported in the range 6 to 10 mol % In according to different sources. A schematic phase diagram of the In-Rb system after (6) is redrawn in the figure. The saturated solution of In in liquid Rb is in equilibrium with In_3Rb_2 or In_5Rb_4, if this compound exists in the system (8).



References

1. Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* 1970, *376*, 44.
2. Yatsenko, S.P.; Chuntonov, K.A.; Bushmanov, V.D.; Dieva, E.N. *Struktura Faz, Fazovye Prevrashcheniya i Diagramy Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1974, p. 198.
3. Dasarathy, C. *Trans.AIME* 1969, *245*, 2015.
4. Bushmanov, V.D. *Ph.D. thesis*, Inst. Khim. Ural. Nauch. Tsentra Akad. Nauk SSSR, Sverdlovsk, 1980; as cited in 6.
5. Melekhov, L.Z., *Ph.D. thesis*, Inst. Khim. Ural. Nauch. Tsentra Akad. Nauk SSSR, Sverdlovsk, 1983; as cited in 6.
6. Yatsenko, S.P. *Indii*, Nauka, Moskva, 1987, p. 99.
7. Melekhov, L.Z.; Yatsenko, S.P.; Chuntonov, K.A.; Zhakupov, Sh. R. *Izv. Akad. Nauk SSSR, Met.* 1982, no. 3, 202.
8. Pelton, A.D.; Larose, S. *J.Phase Equil.* 1991, *12*, 377.

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Thümmel, R.; Klemm, W.

Z. Anorg. Chem. 1970, *376*, 44-63.

VARIABLES:

Temperature: 611-789 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the In-Rb system were determined; they were read out from the figure by the compilers.

t/°C	*soly*/mol % In	*t*/°C	*soly*/mol % In
516	50	448	75
510	55	437	77.5
495	59	438	80
495	61.5	437	81
482	63.5	436	82
470	65.5	427	85
463	67	406	91.5
453	69	338	95

A liquid miscibility gap was observed between ~6 and 62 mol % In at 492 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were melted in a Ta crucible in order to prepare the alloys. A Fe stopper served to inhibit the evaporation of Rb. All experimental operations were performed in an Ar atmosphere. Thermal analysis of the samples was carried out by cooling from 893 K. The temperatures were recorded by the use of Pt/Pt-Rh and Ni/Ni-Cr thermocouples.

SOURCE AND PURITY OF MATERIALS:

In: 99.99 % pure from Duisburger Kupferhütte.
Rb: purified by distillation according to method described in (1).

ESTIMATED ERROR:

Nothing specified.
Solubility: read out procedure ± 0.5 mol %.
Temperature: read out procedure ± 3 K.

REFERENCES:

1. Biltz, W.; Weibke, F.; Eggers, H. *Z. Anorg. Chem.* 1934, *219*, 119.

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Yatsenko, S.P.; Chuntonov, K.A.; Bushmanov, V.D.; Dieva, E.N.
Struktura Faz, Fazovye Prevrashcheniya i Diagramy Sostoyania Metallicheskikh Sistem, Nauka, Moskva, 1974, p. 198-201.

VARIABLES:

Temperature: 691-795 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the In-Rb system were determined; they were read out from the figure by the compilers.

t/°C	*soly*/mol % In	*t*/°C	*soly*/mol % In
522	54	497	67
522	55	488	69
522	57	476	74
521	59	469	77.5
520	60	449	80
515	61	444	84.5
511	63	418	89
506	65		

A liquid miscibility gap was observed between ~6 and ~55 mol % In at 522 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a hermetically closed Ta crucible. The free volume over the alloy sample was minimized. Thermal analyses of the alloy samples were performed in the same crucible.

SOURCE AND PURITY OF MATERIALS:

In: 99.999 % pure.
Rb: 99.9 % pure.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.3 mol %.
Temperature: read-out procedure ± 2 K.

REFERENCES:

COMPONENTS: (1) Indium; In; [7440-74-6] (2) Cesium; Cs; [7440-46-2]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland June 1991

CRITICAL EVALUATION:

There is rough agreement in the experimental work on phase relations in the In-Cs system. However, quantitative results on the liquidus and the stoichiometry of the solid phases show an unacceptable scatter.

The liquidus line of Cs-rich alloys was determined by Dergacheva et al. (3). The solubility of In in liquid Cs seems to be overestimated if one extrapolates the data of Thümmel and Klemm (2), Bushmanov and Yatsenko (4), and Chuntonov et al. (5) from higher temperatures. The data on the solubility of Cs in liquid In on the In-rich side reported in (3) also seem to be overestimated in comparison with those of (2), (4), and (5) which agree within 25 K.

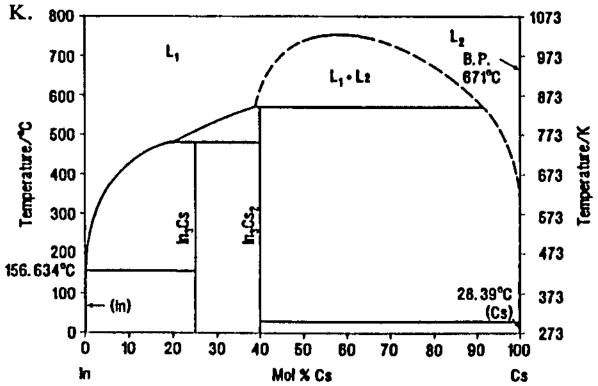
In the central part of the composition range, a liquid miscibility gap was observed experimentally in accordance with an earlier prediction of Dasarathy (1). The monotectic temperature was found at 825 K (3), 842 K (2), and 843 K (4,5,6,7). The composition limits of the miscibility gap mainly differ on the Cs-rich side: ~6 to 62 (2), 10 to 60 (3), 2 to 61 (4,6), and 9 to 60 mol % In (5,7). Consistent results of (5,7) which were obtained by means of magnetochemical and density (by γ-ray diffraction) measurements indicate a much higher critical temperature (~1000 K) than observed in the thermal analyses: 878 K (2,3) and 891 K (4,6). The higher critical temperature proposed by Chuntonov et al. (5,7) is convincing. The phase boundary curve in (5,7) is asymmetrical; this is, however, not the case in the other studies (2,3,4,6). Experimental difficulties and the scatter of results from different sources may be due to possible interactions of In-Cs alloys with container materials. Dergacheva et al. (3) used a Ti crucible several times. Ti may react with In to form Ti-In intermetallics which are moderately soluble in liquid In (8). The other investigators used Ta crucibles which are thought to be corrosion resistant against In and Cs as well. Cs contaminated with O may be even corrosive for Ta (see the Ta-Cs system). The O concentration in the samples used in these studies was not specified. Therefore, only doubtful values of the solubility of In in liquid Cs may be suggested.

The existence of the compounds In_3Cs and In_3Cs_2 is well established (9). A schematic phase diagram is redrawn in the figure after (8). The saturated solution of In in liquid Cs is in equilibrium with In_3Cs_2 up to the monotectic temperature 843 K. A critical evaluation of the In-Cs system was recently published (10).

Doubtful values of the solubility of In in liquid Cs

T/K	*soly*/mol % In	source
473	≤1	(3) interpolated
573	≤3	(3) interpolated
673	≤5	(3) interpolated
773	≤8	(3) interpolated
843	9 monotectic	(5,7)
873	12	(5,7) interpolated
973	30	(5,7) interpolated

The two metals are completely miscible at > ~ 1000 K.



References

1. Dasarathy, C. *Trans AIME* 1969, *245*, 2015.
2. Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* 1970, *376*, 44.
3. Dergacheva, M.B.; Vlasov, S.V.; Kozin, L.F.; Shalamov, A.E. *Izv. Akad. Nauk Kaz. SSR, Ser. Khim.* 1979, no. 3, 52.
4. Bushmanov, V.D; Yatsenko, S.P. *Izv. Akad. Nauk SSSR, Met.* 1981, no. 5, 202.
5. Chuntonov, K.A.; Melekhov, L.Z.; Kuznetsov, A.N.; Orlov, A.N.; Ugodnikov, G.G.; Yatsenko, S.P. *J. Less-Common Met.* 1982, *83*, 143.
6. Bushmanov, V.D. *Ph.D. thesis*, Inst. Khim. Ural. Nauch. Tsentra Akad. Nauk SSSR, Sverdlovsk, 1980; as cited in 8.
7. Melekhov, L.Z., *Ph.D. thesis*, Inst. Khim. Ural. Nauch. Tsentra Akad. Nauk SSSR, Sverdlovsk, 1983; as cited in 8.
8. Yatsenko, S.P. *Indii*, Nauka, Moskva, 1987.
9. Chuntonov, K.A.; Yatsenko, S.P.; Hryn, Yu.N. *J. Less-Common Met.* 1984, *99*, 15.
10. Pelton, A.D.; Larose, S. *Bull.Alloy Phase Diagr.* 1990, *11*, 229.

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Thümmel, R.; Klemm, W.

Z. Anorg. Chem. 1970, *376*, 44-63.

VARIABLES:

Temperature: 528-873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some liquidus points of the In-Cs system were determined; they were read out from the figure by the compilers.

t/°C	*soly*/mol % In	*t*/°C	*soly*/mol % In
255	98	519	74.5
367	94	528	72
418	90	539	70
433	88	548	68
446	86	555	66
453	84	562	64
468	82.5	566	63
487	80	569	61.5
499	78	600	60
510	76.5		

A liquid miscibility gap was estimated to be between ~6 and 62 mol % In at 569 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were melted in a Ta crucible in order to prepare the alloys. A Fe stopper served to inhibit the evaporation of Cs. All experimental operations were performed in an Ar atmosphere. Thermal analysis of the samples was carried out by cooling from 893 K. The temperatures were recorded by the use of Pt/Pt-Rh and Ni/Ni-Cr thermocouples.

SOURCE AND PURITY OF MATERIALS:

In: 99.99 % pure from Duisburger Kupferhütte.
Cs: purified by distillation according to the method described in (1).

ESTIMATED ERROR:

Nothing specified.
Solubility: read out procedure ± 0.5 mol %.
Temperature: read out procedure ± 3 K.

REFERENCES:

1. Biltz, W.; Weibke, F.; Eggers, H. *Z. Anorg. Chem.* 1934, *219*, 119.

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Dergacheva, M.B.; Vlasov, S.V.; Kozin, L.F.; Shalamov, A.E.
Izv. Akad. Nauk Kaz. SSR, Ser. Khim. 1979, no. 3, 52-54.

VARIABLES:

Temperature: 455-882 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of the In-Cs system were determined; they were read out from the figure by the compilers.

t/°C	*soly*/mol % In	*t*/°C	*soly*/mol % In
182	1	546	60
386	5	546	61.5
495	8	500	70
609	23.5	489	72.5
604	40	417	79.5
587	50	374	85
576	57	327	90
561	58		

A liquid miscibility gap was estimated to be situated between 10 and 60 mol % In at 552 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared from weighed amounts of the metals within a Ti crucible under the inert atmosphere of a dry box. The alloys were heated to 20 K above the melting temperature and tempered for 1 hour. Cooling curves were recorded by means of Chromel/Alumel thermocouples which were calibrated on the melting points of several metals and eutectic mixtures.

SOURCE AND PURITY OF MATERIALS:

In: 99.999 % pure.
Cs: 99.97 % pure.

ESTIMATED ERROR:

Solubility: nothing specified.; read-out procedure ± 0.5 mol %.
Temperature: precision ± 3 K.

REFERENCES:

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Bushmanov, V.D.; Yatsenko, S.P.

Izv. Akad. Nauk SSSR, Met. 1981, no. 5, 202-204.

VARIABLES:

Temperature: 716-896 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points of the In-Cs liquidus were reported in a figure; they were read out by the compilers.

t/°C	*soly*/mol % In	t/°C	*soly*/mol % In
600	12	580	60.5
616	16	569	62.5
621	22	568	65.5
623	32	558	67.5
621	40.5	545	72
616	44	515	77
610	48.5	478	83
605	52	464	87.5
596	55.5	443	92
590	58		

The monotectic temperature was found at 570 °C and the composition range between 2 and 61 mol % In. The results were also published in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Differential thermal analysis of the alloys was performed in a Ta crucible using a differential scanning calorimeter. The alloys were prepared in a dry box. Liquid Cs was introduced into the alloys in measured volumes by means of a glass syringe. After filling the crucible was hermetically closed by die forging.

SOURCE AND PURITY OF MATERIALS:

In: 99.999 % pure.
Cs: 99.99 % pure.

ESTIMATED ERROR:

Solubility: precision of sample preparation better than ± 1 %; read-out procedure ± 0.5 mol %.
Temperature: precision ± 2 K.

REFERENCES:

1. Bushmanov, V.D. *Ph.D. thesis*, Inst. Khim. Ural. Nauch. Tsentra Akad. Nauk SSSR, Sverdlovsk, 1980.

COMPONENTS:

(1) Indium; In; [7440-74-6]

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

ORIGINAL MEASUREMENTS:

Chuntonov, K.A.; Melekhov, L.Z.; Kuznetsov, A.N.; Orlov, A.N. Ugodnikov, G.G.; Yatsenko, S.P.
J. Less-Common Met. 1982, *83*, 143-153.

VARIABLES:

Temperature: 699-998 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Liquidus points of the In-Cs system were determined; they were read out from the figure by the compilers.

t/°C	*soly*/mol % In	t/°C	*soly*/mol % In	t/°C	*soly*/mol % In
426	90 [a]	554	67 [a]	725	50.5[a]
449	85 [b]	555	66 [b]	725	40.5[a]
482	80 [a]	568	63 [b]	710	33 [c]
513	75 [b]	690	58 [c]	722	30.5[a]
533	70 [b]	705	57.5[b]	665	20 [a]
547	68 [b]	697	56 [a]	615	13 [a]

[a] measured by means of magnetic susceptibility method; results also published in (1).
[b] by differential thermal analysis
[c] by density measurements with γ-rays

The miscibility gap was estimated between 9 and 60 mol % In at 570 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A Ta crucible was charged with Cs and encapsulated in an evacuated Pyrex ampoule. The ampoule was opened under Ar atmosphere and In was added into the crucible which was closed by Laser welding. Differential thermal analysis was performed in a commercial apparatus in the heating and cooling mode. The magnetic susceptibility of samples was measured at several temperatures. Phase transitions were estimated based on discontinuities and changes of slopes. The density of samples was measured using ^{137}Cs as γ-ray source. Break points of the density vs. temperature curves correspond to phase transitions in the liquid immiscibility range.

SOURCE AND PURITY OF MATERIALS:

In: 99.999 % pure.
Cs: 99.98 % pure.

ESTIMATED ERROR:

Solubility: precision ± 0.6 mol %; read-out procedure ± 1 mol %.
Temperature: unspecified; read-out procedure ± 3 K.

REFERENCES:

1. Melekhov, L.Z. *Ph.D. thesis*, Inst. Khim. Ural. Nauch. Tsentra Akad. Nauk SSSR, Sverdlovsk, 1983.

COMPONENTS:

(1) Thallium; Tl; [7440-28-0]

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

ORIGINAL MEASUREMENTS:

Grube, G.; Schaufler, G.

Z. Elektrochem. 1934, *40*, 593-600.

VARIABLES:

Temperature: 449-781 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES & CRITICAL EVALUATION:

The liquidus temperatures of the Tl-Li system were determined.

t/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl
275	5.0	499	55.8	445	69.2	415	78.1
246	10.1	497	56.3	446	70.3	406	78.7
214	14.9	487	57.7	448	71.3	402	78.8
249	19.9	472	60.6	444	72.3	396	79.7
299	25.0	464	61.6	443	72.6	391	80.2
354	31.0	456	62.6	437	73.5	386	81.7
406	35.2	451	63.0	441	74.1	378	83.3
448	40.0	443	63.9	444	74.4	348	86.4
496	45.1	442	64.2	446	74.8	300	89.8
508	49.9 congruent	441	65.0	447	75.2	260	92.3
508	50.1	435	66.0 eutectic	442	75.8	206	96.8
506	52.6	442	67.6	436	76.7	186	98.0
501	54.9	443	68.4	426	77.8	176	99.2

The melting points of Tl and Li were found to be 302 and 179 °C, respectively.
Aleksandrov et al. (1) reported the melting point of TlLi to be at 510 °C in agreement with these data; details of the experimental procedures were not given.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The crucible of low C Fe was filled with weighed amounts of the two metals under Ar atmosphere and heated above the melting temperature. Cooling curves were recorded by means of a Ni/Ni-Cr thermocouple in a Fe shielding which was previously calibrated on the melting points of several pure metals. The composition of the alloys was analyzed after decomposition with Hg and H_2O and titration of the resulting solution with HCl and by means of electrometric titration with $KBrO_3$ for Tl.

SOURCE AND PURITY OF MATERIALS:

Tl: 99.95 % pure from Gewerkschaft Sachtleben A.G.
Li: 99.0 % pure from Metallgesellschaft A.G., with contents of 0.62 % K; 0.14 % Na; 0.02 % Fe_2O_3; 0.05 % SiO_2; 0.32 % Li_3N; and traces of Al_2O_3.
Ar: 98.2 % pure with contents of 0.1 % O_2, and 1.7 % N_2; further purified by purging through molten Li.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Aleksandrov, B.N.; Dalakova, N.V.; Moskalets, M.V. *Izv. Akad. Nauk SSSR, Met.* 1987, no. 3, 198-202.

COMPONENTS: (1) Thallium; Tl; [7440-28-0] (2) Sodium; Na; [7440-23-5]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1991

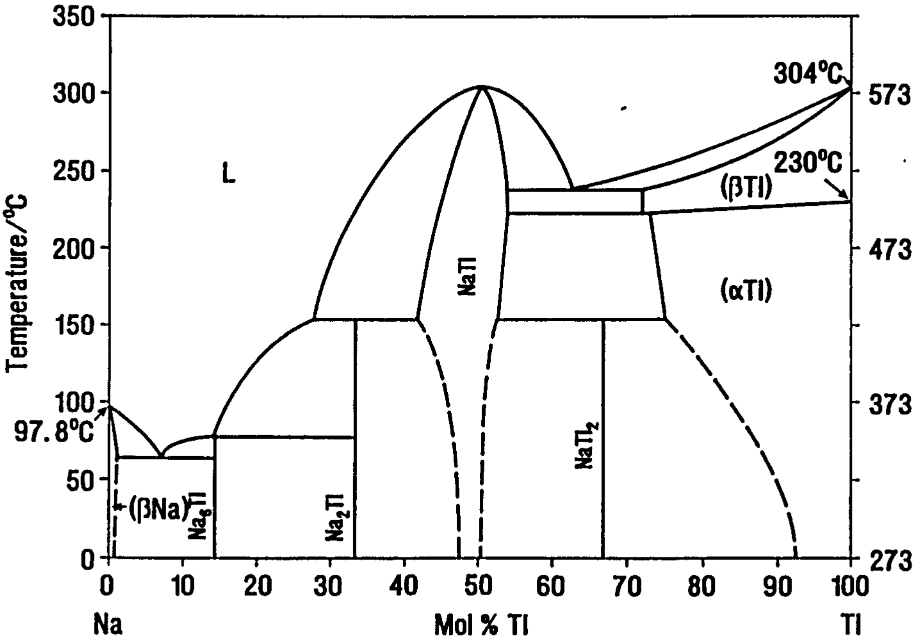
CRITICAL EVALUATION:

A depression of the melting point of Na due to an addition of Tl was independently observed by Heycock and Neville (1) and Tammann (2) using compositions of up to 4.19 (1) and 1.14 mol % Tl, respectively. The results of both studies show an agreement of ± 0.02 mol % Tl.
Kurnakov and Pushin (3) determined the liquidus line of the Tl-Na system in the whole composition range in a subsequent study, thus confirming the precise results of the previous measurements.
Exhaustive studies of all phase relations of the Tl-Na system were performed by Grube and Schmidt (4), who supported thermal analysis by means of electrical conductivity of the solid alloys. The results agreed with (3) within ± 3 K, except for the composition range 25 to 30 mol % Tl, where liquidus temperatures were found to be at up to 20 K higher. Novakovic et al. (5) optimized the Tl-Na phase diagram in respect to thermodynamic parameters, obtaining a diagram agreeing reasonably with the experimental liquidus points of (4) and fitting better to the results of (3) in the composition range 40 to 50 mol % Tl. The saturated solution of Tl in liquid Na was in equilibrium with the Tl-Na solid phase as shown in the figure which was redrawn after (5).

Recommended solubility values of Tl in liquid Na

T/K	*soly*/mol % Tl		source
337	7.1	eutectic	(3,4) mean value
350	13	peritectic	(3,4) mean value
373	16		(3,4) interpolated
426	27.5	peritectic	(3,4) mean value
473	32		(3,4) interpolated
573	46		(3,4) interpolated
578	50.0	congruent	(3,4) mean value

The metals are completely miscible at temperatures above 578 K.



References

1. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1889, *55*, 666.
2. Tammann, G. *Z. Anorg. Chem.* 1889, *3*, 441.
3. Kurnakov, N.S.; Pushin, N.A. *Zh. Russ. Fiz.-Khim. Obshch.* 1901, *33*, 565; *Z. Anorg. Chem.* 1902, *30*, 86.
4. Grube, G.; Schmidt, A. *Z. Elektrochem.* 1936, *42*, 201.
5. Novakovic, R.; Ghosh, G.; Lukas, H.L.; Ristic, M.M. *Emerging Materials by Advanced Processing*, W.A. Kaysser and J. Weber-Bock, Eds., Hirsau, 1989.

COMPONENTS:

(1) Thallium; Tl; [7440-28-0]

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

ORIGINAL MEASUREMENTS:

Heycock, C.T.; Neville, F.M.

J.Chem.Soc. 1889, *55*, 666-676

VARIABLES:

Temperature: 370.0-351.0

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The depression of the melting point of Na due to the addition of Tl was measured.

t/°C	*soly*/atoms Tl per 100 atoms Na	*soly*/mol % Tl[a]
96.81	0.164	0.164
95.81	0.408	0.406
95.04	0.635	0.631
94.34	0.753	0.747
93.05	1.033	1.022
91.65	1.41	1.39
87.3	2.36	2.31
83.64	3.14	3.04
80.75	3.78	3.64
77.87	4.38	4.19

[a] calculated by the compilers

The melting point of pure Na was reported to be 97.46 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was melted under paraffin in a crucible which was made of cast Fe. Small quantities of Tl in fine dispersion were added into the crucible. The molten alloy was allowed to cool slowly. During the cooling the alloy was stirred using a Fe stirrer. The freezing temperatures of the alloys were determined by means of carefully calibrated thermometers.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision not better than ± 0.01 K.

REFERENCES:

COMPONENTS:

(1) Thallium; Tl; [7440-28-0]

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

ORIGINAL MEASUREMENTS:

Tammann, G.

Z. Phys. Chem. 1889, *3*, 441-449.

VARIABLES:

Temperature difference 4.89 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The depression of the melting point of Na to which Tl was added was determined.

ΔT/K	*soly*/g Tl per 100 g Na	*soly*/mol % Tl [a]
0.03	0.21	0.024
0.11	0.44	0.049
0.34	0.55	0.062
0.57	1.42	0.160
0.89	2.17	0.243
1.26	2.90	0.33
1.78	4.05	0.45
2.30	5.19	0.58
3.09	6.76	0.76
3.78	8.14	0.91
4.89	10.41	1.14

[a] calculated by the compilers

The melting point of pure Na was reported to be 370 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Portions of Tl were added to molten Na. The resulting alloys were undercooled for up to 2 K and crystal precipitation was forced by stirring with the thermometer. Every determination of the temperature was three times repeated. The atmospheric conditions were not specified.

SOURCE AND PURITY OF MATERIALS:

Tl: nothing specified.
Na: "pure".

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.05 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thallium; Tl; [7440-28-0] (2) Sodium; Na; [7440-23-5]	Kurnakov, N.S.; Pushin, N.A. *Zh. Russ. Fiz.-Khim. Obshch.* 1901, *33*, 565-587.
VARIABLES:	**PREPARED BY:**
Temperature: 337.3-579 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Liquidus temperatures of the Tl-Na system were determined.

t/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl
95.5	0.35	153.0	27.55	267.5	60.10
93.4	0.80	155.5	28.12	260.6	60.96
91.3	1.34	156.7	28.77	237.7	63.38 eutectic
88.9	1.91	158.2	29.30	240.1	64.64
84.6	2.78	158.7	29.90	241.6	65.65
81.1	3.57	~206.6	33.57	243.1	66.32
78.6	4.14	242.5	37.00	245.0	67.04
75.1	4.79	260.2	39.27	247.1	68.08
72.0	5.46	274.0	41.19	249.7	69.26
68.7	6.05	287.0	43.23	251.9	70.37
64.1	7.20 eutectic	292.3	44.33	255.5	72.12
73.8	10.73	297.0	45.72	258.1	74.00
75.4	11.82	302.5	47.47	261.2	76.05
77.0	12.99	305.8	49.76	263.7	77.38
77.9	14.07	305.3	51.76	267.2	79.56
~108	18.12	302.5	53.21	269.7	81.10
120.2	19.78	300.5	53.64	275.0	84.72
128.6	21.80	295.5	55.49	279.4	87.67
133.2	22.61	291.7	56.22	283.3	90.58
135	23.10	289.5	56.62	291.7	93.98
138.5	23.76	286.9	57.20	296.0	96.23
142.6	24.60	283.1	57.86	298.2	97.78
145.2	25.30	278.3	58.59		
148.4	26.00	271.8	59.49		

The melting points of Tl and Na were found to be 301.0 and 97.0 °C, respectively.
The results were also published in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The surface layer of a Na piece was cut off under vaseline oil. The piece was cleaned with petroleum and ether. A Na sample was immediately placed under molten paraffin in a Fe crucible after weighing. A weighed amount of Tl was introduced into the crucible, and the temperature was elevated up to the melting point of the formed alloy. The melt was stirred and a cooling curve was recorded. A thermometer served for the measurement of the temperature.

SOURCE AND PURITY OF MATERIALS:

Tl: unspecified purity, from Merck.
Na: nothing specified.

ESTIMATED ERROR:

Solubility: precision in sample preparation ± 0.2 %.
Temperature: nothing specified; precision not better than ± 0.5 K (by the compilers).

REFERENCES:

1. Kurnakow, N.S.; Puschin, N.A. *Z. Anorg. Chem.* 1902, *30*, 86-108.

COMPONENTS:

(1) Thallium; Tl; [7440-28-0]

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

ORIGINAL MEASUREMENTS:

Grube, G.; Schmidt, A.

Z. Elektrochem. 1936, *42*, 201-209.

VARIABLES:

Temperature: 338-578 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus temperatures of the Tl-Na system were determined.

t/°C	*soly*/mol % Tl	t/°C	*soly*/mol % Tl	t/°C	*soly*/mol % Tl	t/°C	*soly*/mol % Tl
92.5	1.0	77.2	12.0	214	33.5	242	65.0
90.4	1.5	77.2	13.1	222	34.2	250	70.2
88.2	2.0	79.8	14.1	229	35.0	253.5	72.1
82.0	3.0	88.2	15.1	265	40.1	258.5	75.0
77.5	3.8	94.8	16.0	271	42.0	260	76.0
73.2	5.0	122.6	20.2	289	45.0	263.5	78.0
67.9	5.9	144.6	25.3	303	49.4	266	80.0
64.6	7.0	152	27.1	305	50.0	272	82.0
68.8	7.9	156	28.0	300	50.4	276	85.0
69.2	8.7	177	29.0	298	54.8	283	90.0
73.0	9.7	182.5	30.0	272	60.0	291	95.0
74.3	10.0	189	31.3	264	61.2	295	97.0
75.0	10.4	198	31.8	246.5	62.1	297.5	98.0
74.7	10.9	207	32.4	240	63.1	298.5	99.0

The melting points of Tl and Na were determined to be at 302 and 98.0 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The surfaces of Na and Tl pieces were cut off. The clean and dry metals were immediately weighed and introduced into a Fe crucible in an Ar atmosphere. The crucible was placed in a furnace and the apparatus refilled with Ar. The crucible was heated to the melting point of the alloys and the cooling curves were recorded by means of an Ag/Konstantan thermocouple calibrated on the melting point of p-dichlorobenzene, Sn, Cd, Zn, and the boiling point of H_2O. A precise thermometer was also applied for temperatures below 313 K. The Na content of the alloy samples was analyzed by means of alkalimetric titration, the Tl content by potentiometric titration with $KBrO_3$.

SOURCE AND PURITY OF MATERIALS:

Tl: 99.95 % pure from Gewerkschaft Sachtleben A.G.
Na: unspecified purity from Merck
Ar: 98.2 % pure from Lindes Eismaschinen A.G., containing 1.7 % N_2 and 0.1 % O_2; blown through pyrogallol solution, concentrated H_2SO_4, Mg turnings at 873 K and P_2O_5 granules.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Thallium; Tl; [7440-28-0]

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

EVALUATOR:

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

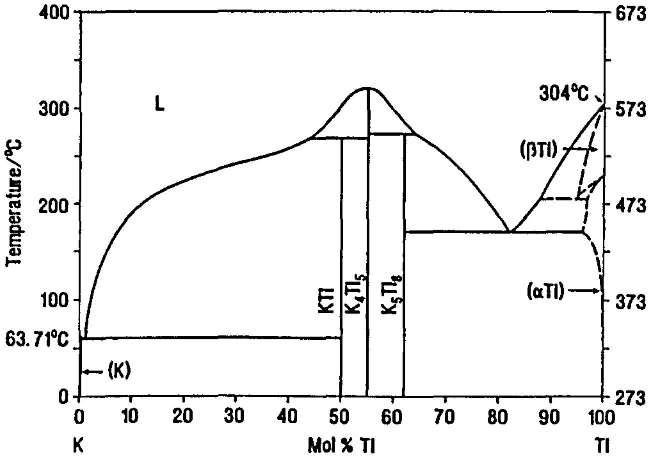
CRITICAL EVALUATION:

Large solubility of Tl in liquid K was reported in a study by Heycock and Neville (1), indicating that addition of Tl to K caused a significant depression of the melting point of K. However, numerical data were not provided in this study.
Kurnakov and Pushin (2) determined the liquidus of the Tl-K phase diagram in the whole composition range by means of thermal analysis. Their results were merely partly confirmed by subsequent analogous experiments performed by Thümmel and Klemm (3). The most serious discrepancies occurred in the composition range 40 to 85 mol % Tl.
The liquidus line of (2) seems to be in better agreement with the general rules of phase diagram relations. However, the metals used in (3) seem to be less contaminated. The suggested values therefore belong to different categories concerning the reliability of the solubility data.
The Tl-K phase diagram from (4) is redrawn in the figure.

Tentative (t) and doubtful (d) solubility values of Tl in liquid K

T/K	*soly*/mol % Tl	source
335	~ 1 (d) eutectic	(2),(3) mean value
373	3 (d)	(3) interpolated
473	~11 (d)	(2),(3) mean value
500	25 (t)	(2),(3)
573	48 (d)	(2),(3) mean value
594	56 (d)	(3)

The liquid metals are miscible at temperatures above 594 K.



References

1. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1889, *55*, 666.
2. Kurnakov, N.S.; Pushin, N.A. *Zh. Russ. Fiz.-Khim. Obshch.* 1901, *33*, 565; *Z. Anorg. Chem.* 1902, *30*, 86.
3. Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* 1970, *376*, 44.
4. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am.Soc.Mater., Materials Park, 1990, p. 2394.

COMPONENTS:

(1) Thallium; Tl; [7440-28-0]

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

ORIGINAL MEASUREMENTS:

Kurnakov, N.S.; Pushin, N.A.

Zh. Russ. Fiz.-Khim. Obshch. 1901, *33*, 565-587.

VARIABLES:

Temperature: 335.7-608 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus temperatures of the Tl-K system were determined. The data were also published in (1).

t/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl
62.5	2.33	237.7	29.67	290.0	68.18
~161	3.3	240.0	31.67	280.0	71.84
172.5	5.24	242.0	32.94	248.2	75.93
~192	8.44	254.2	37.94	222.0	78.87
~206	11.33	270.5	41.91	195.5	81.92
~211	13.02	309.0	45.83	172.5	84.30 eutectic
217.0	15.74	326.7	47.96	195.0	86.62
220.5	17.49	335.0	50.22	234.0	90.17
227.3	21.69	331.0	52.53	264.7	93.44
229.3	23.16	321.5	56.13	288.5	95.84
232.5	25.68	313.2	62.00	297.1	98.15

The melting points of Tl and K were found to be 301.0 and 62.5 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The surface layer of a K piece was cut off under vaseline oil. The piece was cleaned with petroleum and ether. A K sample was immediately placed under molten paraffin in a Fe crucible after weighing. A weighed amount of Tl was introduced into the crucible, and the temperature was elevated up to the melting point of the formed alloy. The melt was stirred and a cooling curve was recorded. A thermometer served for the measurement of the temperature.

SOURCE AND PURITY OF MATERIALS:

Tl: unspecified purity, from Merck.
K: nothing specified.

ESTIMATED ERROR:

Solubility: precision in sample preparation ± 0.2 %.
Temperature: nothing specified; precision not better than ± 0.5 K (by the compilers).

REFERENCES:

1. Kurnakow, N.S.; Puschin, N.A. *Z. Anorg. Chem.* 1902, *30*, 86-108.

COMPONENTS:

(1) Thallium; Tl; [7440-28-0]

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

ORIGINAL MEASUREMENTS:

Thümmel, R.; Klemm, W.

Z. Anorg. Chem. 1970, *376*, 44-63.

VARIABLES:

Temperature: 398-594 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus temperatures of the Tl-K system were determined. The data were read out from the figure by the compilers.

t/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl
125	5	271	45.5	271	64.5
182	10	273	47	270	66
218	17.5	284	47.5	265	67
219	20	296	50	259	68
222	22	309	52	251	70
229	25	319	54	238	72
230	28	321	55.5	228	73.5
234	30	318	56.5	214	76
235	32	307	57	199	77.5
237	33.5	291	58	184	80
241	34.5	286	59	170	82 eutectic
244	38	280	60	178	84
251	39.5	277	61	183	86.5
256	41.5	274	62	207	88.5
267	44.5	272	63	252	94

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a Ta crucible with a Fe stopper which served to inhibit the evaporation of K. The experiments were performed in an Ar atmosphere. Thermal analyses were performed by cooling from ~ 670 K. The cooling curves were measured using Pt/Pt-Rh and Ni/Cr-Ni thermocouples.

SOURCE AND PURITY OF MATERIALS:

Tl: 99 % pure from Riedel de Haen.
K: from Merck; surface of the metal was cut off, the metal was then distilled under high vacuum.
Ar: nothing specified.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.5 mol %.
Temperature: read-out procedure ± 2 K.

REFERENCES:

COMPONENTS:

(1) Thallium; Tl; [7440-28-0]

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

ORIGINAL MEASUREMENTS:

Thümmel, R.; Klemm, W.

Z. Anorg. Chem. 1970, *376*, 44-63.

VARIABLES:

Temperature: 369-650 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus temperatures of the Tl-Rb system were determined. The data were read out from the figure by the compilers.

t/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl	*t*/°C	*soly*/mol % Tl
96	2	357	49.5	350	66.5
195	5	365	51.5	346	68
233	9.5	367	52.5	326	70
281	14.5	373	53.5	281	71.5
304	20	377	54.5	275	72.5
310	25	376	55.5	273	74.5
312	27.5	374	57	272	77
316	30	373	57.5	268	79.5
318	35	370	59	232	85.5
328	40	365	61.5	198	88
339	44.5	361	63.5	209	90
348	47.5	352.5	65.5	262	95

COMMENTS AND ADDITIONAL DATA:

The liquidus line of the Tl-Rb system shows general consistency with that of the Tl-K system. It may, therefore, be concluded that the determinations of the points on the liquidus of the Tl-Rb system should be reliable. There is some doubt concerning the liquidus inflection on the Tl-rich side at 533 to 600 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a Ta crucible with a Fe stopper which served to inhibit the evaporation of Rb. The experiments were performed in an Ar atmosphere. Thermal analyses (cooling and heating curves) were carried out after heating the alloys to 893 K, the cooling curves were measured using Pt/Pt-Rh and Ni/Cr-Ni thermocouples.

SOURCE AND PURITY OF MATERIALS:

Tl: 99 % pure from Riedel de Haen.
Rb: obtained from RbCl by means of reduction with Ca, isolated and purified by distillation under high vacuum.
Ar: nothing specified.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 1 mol %.
Temperature: read-out procedure ± 3 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Thallium; Tl; [7440-28-0] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, KfK, Karlsruhe, Fed. Rep. of Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1991

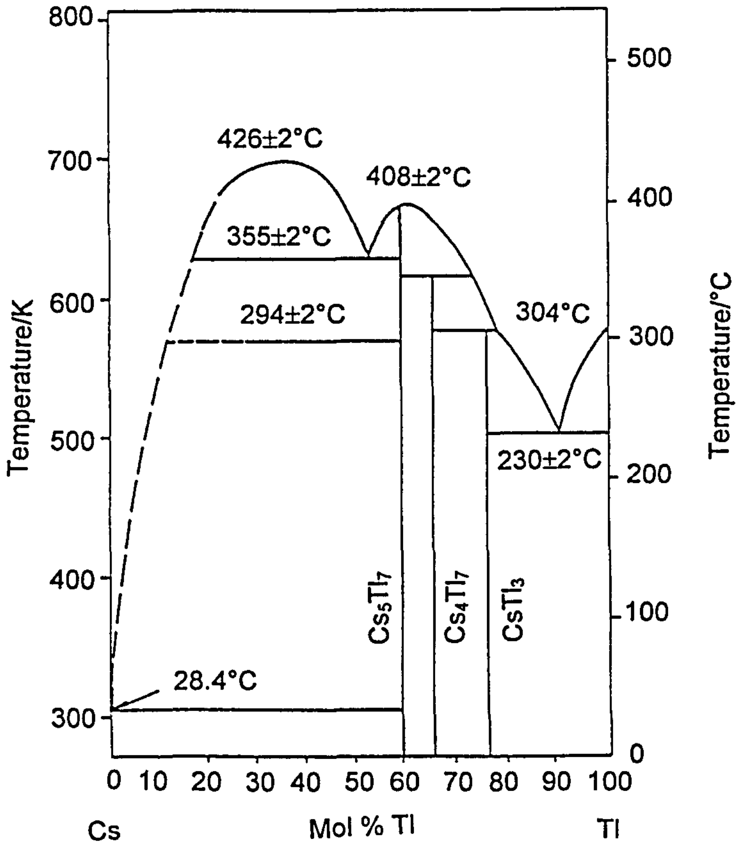
CRITICAL EVALUATION:

The phase relations and the solubility lines of the Tl-Cs system reported by Thümmel and Klemm (1) and by Bushmanov and Yatsenko (2) show significant differences. Ta crucibles were used in both studies, thus, effects of the corrosion of Ta and any contamination of the molten alloys by Ta should have been comparable in both cases. While a much higher number of single measurements were performed by (1), the other group used metals of higher purity. Therefore, the results of Bushmanov and Yatsenko (2) seem to be more convincing. The tendency to form a liquid miscibility gap in the Tl-Cs system may be predicted from Mott's rule as well as from the large difference of the atomic radii of Tl and Cs (2).
The liquid immiscibility was reported in (2) to be in the Cs-rich range, while (1) proposed an eutectic type of liquidus in this range. The inflection point at about 660 K and 50 mol % Tl which was detected by (1) seems to be unlikely, since it is not in agreement with thermodynamic factors. The region of Cs-rich alloys needs further investigation. Thus, only the solubility of Tl at higher temperatures may be suggested.
The phase diagram of the Tl-Cs system is taken from (2).

Tentative values of the Tl solubility in liquid Cs

T/K	*soly*/mol % Tl	source
628	16 monotectic	(2)
699	35 critical point	(2)

The two metals are completely miscible above 699 K (2).



References

1. Thümmel, R.; Klemm, W. *Z. Anorg. Chem.* 1970, *376*, 44.
2. Bushmanov, V.D.; Yatsenko, S.P. *Izv. Akad. Nauk SSSR, Met.* 1981, no. 5, 202.

COMPONENTS:

(1) Thallium; Tl; [7440-28-0]

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

ORIGINAL MEASUREMENTS:

Thümmel, R.; Klemm, W.

Z. Anorg. Chem. 1970, *376*, 44-63.

VARIABLES:

Temperature: 386-699 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus temperatures of the Tl-Cs system were determined; the data were read out from the figure by the compilers.

t/°C	*soly*/mol % Tl	t/°C	*soly*/mol % Tl	t/°C	*soly*/mol % Tl
113	5	424	57.5	340	74
208	10	425	58	325	76
243	15	426	58.5	317	80
268	20	424	60	301	84
305	30	417	61	280	85.5
343	40	411	62	256	88
360	45	403	64	243	88.5
373	47.5	392	66	238	90
393	51.5	385	68.5	287	95
409	55	372	70		
420	56	358	72		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a Ta crucible with a Fe stopper which served to inhibit the evaporation of Cs. The experiments were performed in an Ar atmosphere. Thermal analyses were carried out, the cooling curves were measured using Pt/Pt-Rh and Ni/Cr-Ni thermocouples.

SOURCE AND PURITY OF MATERIALS:

Tl: 99 % pure from Riedel de Haen.
Cs: obtained from CsCl by means of reduction with Ca, isolated and purified by distillation under high vacuum.
Ar: nothing specified.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 1 mol %.
Temperature: read-out procedure ± 3 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thallium; Tl; [7440-28-0] (2) Cesium; Cs; [7440-46-2]	Bushmanov, V.D.; Yatsenko, S.P. *Izv. Akad. Nauk SSSR, Met.* 1981, no. 5, 202-204.
VARIABLES:	**PREPARED BY:**
Temperature: 617-699 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus curve of the Tl-Cs system were determined. The values were read out from the figure by the compilers.

t/°C	*soly*/mol % Tl	t/°C	*soly*/mol % Tl
357	18.5	355	52 monotectic
424	29	384	54
426	40	393	62
422	42	379	65
383	50	367	68
		344	71

The monotectic temperature was found at 355 °C, and the corresponding range of compositions was between 16 and 52 mol % Tl.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were introduced into a Ta crucible in the inert atmosphere of a dry box. The volume of the crucible was filled with the mixture of metals to maximum. The container was then hermetically closed by means of die forging. Differential thermal analysis of the alloys was performed in a differential scanning calorimeter in which the closed crucible was placed.

SOURCE AND PURITY OF MATERIALS:

Tl: 99.999 % pure.
Cs: 99.99 % pure.

ESTIMATED ERROR:

Solubility: precision of sample preparation better than ± 1 %; read-out procedure ± 0.5 mol %.
Temperature: precision ± 2 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Tin; Sn; [7440-31-5] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland August 1991

CRITICAL EVALUATION:

Although the liquidus of the Sn-Li system was intensively investigated (1-6,8,9), particularly on the Sn-rich side, there is only one set of data concerning the solubility of Sn in liquid Li (7).

The earliest data reported in (1) and (2) on the liquidus of the Sn-Li system show rough agreement. They differ, however, significantly (up to 100 K) in the range 15 to 65 mol % Sn from subsequent results (3-6,8,9) in this range. The differences seem to be due to the contamination of the system in (1,2). The more recent data show agreement within ± 10 K in the whole composition range (3-6,8,9). Baradel et al. (10) detected two liquidus points at 973 K at compositions with 15.0 and 30.6 mol % Sn by means of vapour pressure measurements. Their work is not compiled, since essential details were not given.

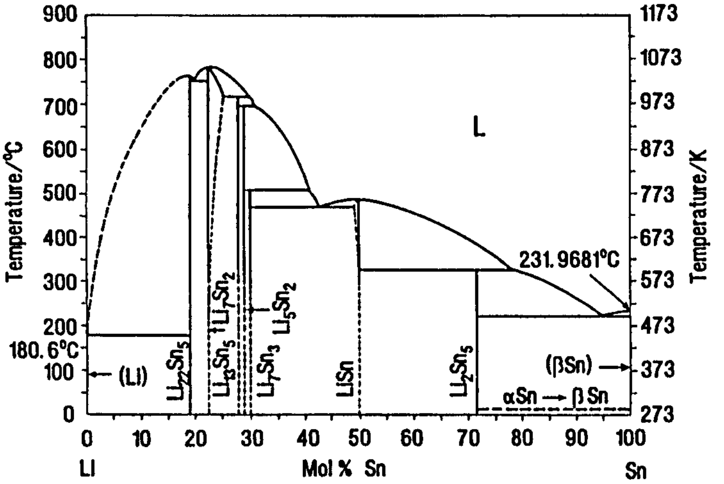
Van der Marel et al. (11) performed measurements of the electrical resistivity of liquid Sn-Li alloys in the Li-rich corner and reported several experimental difficulties. They claimed that the liquidus line of (3) is in error. The paper (11) is, however, not compiled, since it does not report numerical data. The resistivity data of Dadd et al. (7) for alloys with very low contents of Sn are self-consistent. The solubility of Sn in liquid Li smoothly increased from 0.10 to 1.29 mol % Sn in the temperature range 579 to 745 K. Their data are, however, in disagreement with the results of (2,3,10) and suggest much higher melting points of $Li_{22}Sn_5$ and Li_7Sn_2. The melting point of Li_7Sn_2 was confirmed by Bailey et al. (5). The results of (7) would be more convincing if they could be confirmed by thermal analysis or any other method and extended to higher proportions of Sn. The solubilities of Sn in liquid Li as determined by (5) are not situated between the solubilities of Pb and Ge, which might be expected. They are significantly lower than even the solubilities of Si in liquid Li.

Therefore, only the doubtful data which were reported in the earlier reports may be suggested. The liquidus line of the Li-rich alloys needs further investigation. The solid phases which are in equilibrium with the saturated liquid alloys may be seen in the Sn-Li phase diagram redrawn from (6).

Doubtful values of the solubility of Sn in liquid Li

T/K	*soly*/mol % Sn	source	*T*/K	*soly*/mol % Sn	source
673	3	(2),(3) interpolation	1038	18.5 congruent	(3)
773	6	(2),(3) interpolation	1025	20 eutectic	(3)
873	10	(3)	1056	22.2 congruent	(3)
973	15	(3),(10)			

The metals are completely miscible at temperatures >1056 K. The solubility of Li in Sn is shown in the phase diagram.



References

1. Masing, G.; Tammann, G. *Z. Anorg. Chem.* 1910, *67*, 190.
2. Baroni, A. *Atti Real. Accad. Lincei, Roma,* 1932, *16*, 153.
3. Grube, G.; Meyer, E. *Z. Elektrochem.* 1934, *40*, 771.
4. Foster, M.S.; Crouthamel, C.E.; Wood, S.E. *J. Phys. Chem.* 1966, *70*, 3042.
5. Bailey, D.M.; Skelton, W.H.; Smith, J.F. *J. Less-Common Met.* 1979, *64*, 233.
6. Wen, C.J.; Huggins, R.A. *J. Electrochem. Soc.* 1981, *128*, 1181.
7. Dadd, A.T.; Hubberstey, P.; Roberts, P.G. *J. Chem. Soc., Faraday Trans. I* 1982, *78*, 2735.
8. Barsoum, M.W.; Tuller, H.L *Metall. Trans. A* 1988, *19*, 637.
9. Kamata, M.; Ito, Y.; Inoue, M.; Oishi, J. *J. Electrochem. Soc.* 1989, *136*, 528.
10. Baradel, P.; Vermande, A.; Ansara, I.; Desre, P. *Rev. Intern. Hautes Temp. Refract.* 1971, *8*, 201.
11. van der Marel, C.; van Oosten, A.B.; Geertsma, W.; van der Lugt, W. *J. Phys.,F* 1982, *12*, 2349.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Masing, G.; Tammann, G.

Z. Anorg. Chem. 1910, *67*, 190-194.

VARIABLES:

Temperature: 487-953 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the Sn-Li liquidus line were determined.

t/°C	*soly*/mol %Sn	*t*/°C	*soly*/mol %Sn
680	20.0	463	50.0
666	24.9	455	56.1
625	29.4	423	65.2
530	34.6	360	72.3
512	35.6	314	78.3
465	40.4	268	89.3
465	45.8	214	94.6

No temperature arrests could be observed for alloys with 4.1 and 9.3 mol % Sn. Li_4Sn, Li_3Sn_2, and Li_2Sn_5 equilibrium solid phases were determined. The melting points of Sn and Li were found to be 232 and 179 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Sn-rich alloys were prepared in a glass crucible, while the Li-rich alloys were prepared in a Fe crucible in order to avoid interaction between glass and Li. The alloys were protected in a H_2 atmosphere. The metals were weighed (Li under petroleum), placed in the containers and homogenized at about 873 K. Li-rich alloys were only heated to 773 K. Thermal analyses of the melts were performed using a thermocouple.

SOURCE AND PURITY OF MATERIALS:

Sn: nothing specified.
Li: surface was cut off and the metal stored in a H_2 atmosphere.

ESTIMATED ERROR:

Solubility: precision ± 0.5 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Baroni, A.

Atti Real. Accad. Lincei Roma 1932, *16*, 153-158.

VARIABLES:

Temperature: 487-957 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Sn-Li system were determined. The values were read out from the figure by the compilers.

t/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn
359	3	472	38	300	83
487	6.5	483	40 congruent	280	85.5
627	12.5	476	44.5	254	89
684	19.5 congruent	461	49.5	231	91.5
670	22	443	57	214	94 eutectic
631	26	419	64	221	95.5
578	30	379	70.5	226	98
510	33	359	74		
458	35 eutectic	320	79.5 peritectic		

The equilibrium solid phases $SnLi_4$, Sn_2Li_3, and Sn_4Li were identified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The same method as in (1) was used, the alloys were prepared in a stainless steel crucible (type Krupp V2A) in an Ar atmosphere. The composition of the alloys was confirmed by chemical analyses. Cooling curves were recorded by means of previously calibrated Pt/Pt-Rh thermocouples which were sheathed with stainless steel clads.

SOURCE AND PURITY OF MATERIALS:

Sn: nothing specified.
Li: "pure" from Kahlbaum, as in (1).
Ar: purified by means of reaction with Ca and Mg at 773 K in a closed system.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.5 mol %.
Temperature: read-out procedure ± 1 K.

REFERENCES:

1. Pastorello, S. *Gazz.Chim.Ital.*, 1931, *61*, 47-51.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Grube, G.; Meyer, E.

Z. Elektrochem. 1934, *40*, 771-777.

VARIABLES:

Temperature: 495-1055 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Various points on the liquidus line of the Sn-Li system were determined.

t/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn
229	97.5	478	45.0	778	24.0
223	95 eutectic	475	44.0	779	23.7
251	92.5	470	43.0 eutectic	780	23.0
262	90.0	499	42.0	783	22.2 congruent
294	85.0	515	41.0	781	22.0
317	80.0	546	40.0	768	21.0
322	78.0	577	38.0	752	20.5 eutectic
338	75.0	605	37.0	760	20.3
360	72.0	638	36.0	765	20.0 congruent
370	70.0	666	33.9	761	19.3
397	66.7	672	33.4	756	18.2
414	65.0	679	32.9	745	17.5
445	60.0	688	32.1	703	15.0
474	55.0	698	31.1	653	12.5
484	51.0	706	30.3	603	10.0
485	50.0 congruent	715	29.2	527	7.5
484	49.0	720	28.2	464	5.0
483	48.0	729	27.3	362	2.5
481	47.0	745	26.8		
480	46.0	766	25.4		

The melting points of Sn and Li were determined to be at 232 and 179 °C, respectively. Congruently melting $SnLi_4$, Sn_2Li_7, and SnLi and peritectically formed Sn_2Li_5, $SnLi_2$, and Sn_2Li solid equilibrium phases were reported.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared from the metals in an Armco Fe crucible under Ar atmosphere. Corrosion attack of the crucible was not detected. The thermal analyses were recorded by means of Ni/Nichrome thermocouples which had been calibrated on the melting points of Sn, Pb, Zn, Sb, and Mg. The composition of many alloys was confirmed by chemical analyses. The alloys samples were completely decomposed by H_2O and Hg at elevated temperature. The aqueous phase was filtrated and titrated with HCl to calculate the Li content.

SOURCE AND PURITY OF MATERIALS:

Sn: "purest" from Kahlbaum.
Li: 99 % pure from Metallgesellschaft, with contents of 0.62 % K, 0.14 % Na, 0.02 % Fe_2O_3, 0.05 % SiO_2, 0.32 % Li_3N, and traces of Al_2O_3.
Ar: 98.2 % pure, containing 0.1 % O_2 and 1.7 % N_2, further purified by bubbling through molten Li.

ESTIMATED ERROR:

Solubility: precision ± 0.3 mol %.
Temperature: precision ± 2 K (by the compilers).

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Tin; Sn; [7440-31-5] (2) Lithium; Li; [7439-93-2]	Foster, M.S.; Crouthamel, C.E.; Wood, S.E. *J. Phys. Chem.* 1966, *70*, 3042-3045.
VARIABLES:	**PREPARED BY:**
Temperature: 800-950 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points of the liquidus of the Sn-Li system were determined.

T/K	*soly*/mol % Sn
800	40.5[a]
850	38[a]
900	35
950	32[a]

[a] read out from the figure by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed in the He atmosphere of a glove box. The two electrochemical cells were placed in a porous beryllia crucible:

Bi-Li/LiCl-LiF (eutectic)/Sn-Li (1)
Bi-Li/LiCl-LiF (eutectic)/Li (2)

The addition of the potentials of cells (1) and (2) at a given temperature yields the potential of the cell Li/LiCl-LiF (eutectic)/Sn-Li. The potentials were measured for more than 10 hours in order to achieve equilibrium values. The breaking points of the potential vs. composition records at constant temperature or of the potential vs. temperature curve at constant composition indicate the solubility values.

SOURCE AND PURITY OF MATERIALS:

Sn: from Baker Chem. Co.; with contents of $2 \cdot 10^{-5}$ % As, $5 \cdot 10^{-4}$ % Cu, Zn, $3 \cdot 10^{-3}$ % Fe,Pb.
Li: from Foote Mineral Co.; with contents of $3 \cdot 10^{-3}$ % Na, K, Cl, N.
LiCl-LiF: reagent grade, purified by treatment with Cl_2, flushed with He, evacuated and sealed in Pyrex glass for storage.
He: continuously purified.

ESTIMATED ERROR:

Nothing specified.
Potentials: standard deviation below ± 3 mV.
Solubility: read-out procedure ± 0.5 mol %.

REFERENCES:

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Bailey, D.M.; Skelton, W.H.; Smith, J.F.

J. Less-Common Met. 1979, *64*, 233-240.

VARIABLES:

Temperature: 759-1045 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the Sn-Li system were determined.

T/K	*soly*/mol % Sn
1045	24.0
1037	25.4 [a]
1025	26.7 [a]
1000	28.3
999	29.0
995	29.5
984	30.6 [a]
957	32.2
752	48.0
759	50.0

[a] composition confirmed by chemical analysis

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by mixing the metals in a glove box under an Ar atmosphere. The metals were sealed in a Ta container which was enclosed in a stainless steel clad. The alloys were melted and homogenized for 2 hours at 1000 to 1100 K in a rocking furnace. After that procedure the containers were removed out of the clad and differential thermal analysis was performed in a conventional apparatus. Some alloy compositions were confirmed by unspecified chemical analysis.

SOURCE AND PURITY OF MATERIALS:

Sn: analytically pure; Al, Ba, Ca, Ni, Pb, and Si present in small traces, Cu, Fe, and Mg in trace amounts.
Li: from Lithcoa, with a content of <0.005 % Na.
Ar: purified.

ESTIMATED ERROR:

Solubility: precision ± 0.1 mol %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Wen, C.J.; Huggins, R.A.

J. Electrochem. Soc. 1981, *128*, 1181-1187.

VARIABLES:

Temperature: 688-822 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the Sn-Li system were determined.

t/°C	*soly*/mol % Sn
415	63.6 [a]
466	59.3 [b]
486	52.1 [b]
549	40.0 [b]

[a] measurement at constant temperature
[b] measurement at constant composition

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Coulometric titrations and emf measurements were performed in a glove box filled with He. The cell: $Al\text{-}Li_{(s)}$/LiCl-KCl(eutectic)/Sn-Li was used for the experiments. Sn which was contained in a Mo bucket was used as starting material. The Sn-Li alloys were prepared by coulometric reduction of Li^+ on the Sn or Sn-Li electrode within the cell. Some alloys were also prepared by direct fusion of Sn and Li inside the glove box. The way of preparation was without influence on the results of potential measurements. The liquidus points were determined from breaks of the plots of potential versus logarithm of composition at constant temperature. The break point was also measured at constant composition and varying temperature. The phase transition is indicated by changes of the slope of the function of the potential versus temperature. A Chromel/Alumel thermocouple served for temperature measurement.

SOURCE AND PURITY OF MATERIALS:

Sn: 99,9 % pure from Baker.
Li: 99.9 % pure from Foote Mineral.
Al: 99.9999 % pure from Cominco.
LiCl-KCl: purchased from Lithcoa, heated in an alumina crucible to 693 K for 12 hours under He.
He: "high purity".

ESTIMATED ERROR:

Nothing specified.
Solubility: better than ± 0.3 mol % (by the compilers).
Temperature: better than ± 2 K (by the compilers).

REFERENCES:

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Dadd, A.,T.; Hubberstey, P.; Roberts, P.G.

J. Chem. Soc., Faraday Trans. I 1982, *78*, 2735-2741.

VARIABLES:

Temperature: 579-745 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Solubilities of Sn in liquid Li were determined.

T/K	*soly*/mol % Sn	T/K	*soly*/mol % Sn
579	0.10	684	0.54
608	0.15	691	0.59
615	0.20	694	0.65
636	0.26	700	0.72
648	0.30	706	0.79
660	0.37	715	0.91
663	0.40	722	0.98
673	0.46	736	1.11
679	0.49	745	1.29

The authors presented the fitting equation which was checked by the compilers:

$$\ln(soly/\text{mol fract. Sn}) = 4.742 - 6794(T/\text{K})^{-1}, \quad \text{standard deviation} = 0.0477$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Liquid Sn-Li alloys were prepared from accurately weighed amounts of the solute which was added to liquid Li in an Ar atmosphere. The resistivity of the alloys was monitored under equilibrium conditions as a function of temperature for a series of samples which contained increasing concentrations of Sn. The entering of the two-phase region was indicated by discontinuities in the resistivity versus temperature data. The apparatus used for the resistivity measurements was the same as described in (1).

SOURCE AND PURITY OF MATERIALS:

Sn: 99.999 % pure from Koch Light.
Li: 99.98 % pure from Koch Light, further purified by gettering with Y sponge at 673 K for 72 hours.
Ar: 99.99 % pure from Air Products.

ESTIMATED ERROR:

Solubility: standard deviation ± 5 %.
Temperature: nothing specified.

REFERENCES:

1. Adams, P.F.; Down, M.G.; Hubberstey, P.; Pulham, R.J. *J. Less-Common Met.* 1975, *42*, 325-334.

COMPONENTS:	**ORIGINAL MEASUREMENTS:**
(1) Tin; Sn; [7440-31-5] (2) Lithium; Li; [7439-93-2]	Barsoum, M.W.; Tuller, H.L *Metall. Trans. A* 1988, *19*, 637-644.
VARIABLES:	**PREPARED BY:**
Temperature: 603-694 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the liquidus of the Sn-Li system were determined.

t/°C	*soly*/mol % Li	*soly*/mol % Sn [b]
330 [a]	21.8	78.2
333	22.9	77.1
343 [a]	24.9	75.1
344	24.6	75.4
358 [a]	26.3	73.7
380 [a]	30.5	69.5
381	30.3	69.7
421 [a]	36.8	63.2

[a] read out from the figure by the compilers
[b] calculated by the compilers

The authors proposed an equation to fit the results, which should be valid for the range of Li concentration between 0.20 and 0.44 mol fract. Li.

$$t/°\mathrm{C} = 642\ x_{\mathrm{Li}} + 188$$

This part of the liquidus does, however, not follow a linear relationship of temperature versus concentration of Li.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The coulometric preparation of the Sn-Li alloys and the measurements of the emf were performed in a solid electrolyte galvanic cell (Al-Li/glass/Sn-Li/glass/Al-Li) which was operated inside a dry Ar glove box. Al-Li alloys were used in form of powders. The starting material Sn was contained in Mo mesh. The solid electrolyte was glass composed of $LiBO_2$ with 2 mol % Al_2O_3. The Sn-Li alloys were prepared by the coulometric reduction of Li^+ on the Sn (Sn-Li) electrode. The open circuit potentials of the cells with Sn-Li alloy of differing composition were measured during interruptions of the electrolyses. The solubility was indicated by a break point of the relation of the potential versus composition

SOURCE AND PURITY OF MATERIALS:

Sn: 99.8 % pure from Aldrich Chem. Co..
Al-Li: from Lithcoa.
Mo-mesh: from Unique Wire Weaving Co..
$LiBO_2$: 99.998 % pure from Alfa Ventron.
Ar: from Vacuum Atmosphere Co..

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.3 mol %.
Temperature: read-out procedure ± 1 K.

REFERENCES:

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Kamata, M.; Ito, Y.; Inoue, M.; Oishi, J.

J. Electrochem. Soc. 1989, *136*, 528-534.

VARIABLES:

Temperature: 742-757 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Sn in liquid Li was determined.

t/°C	*soly*/mol % Li	*soly*/mol % Sn [b]
469	43.9	56.1
477	47.7	52.3
484	50.8	49.2

[a] as calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The emf of the cell Sn-Li/LiCl-KCl/Sn-Li was measured at selected temperatures. The Sn-Li alloy was prepared in another apparatus and placed into the Pyrex glass tube with a Mo lead wire. All operations were performed in an Ar atmosphere. The breaking points of the relation of the potential versus the composition of the alloy indicated the solubilities.

SOURCE AND PURITY OF MATERIALS:

Sn: unspecified purity.
Li: unspecified purity.
LiCl-KCl: vacuum dried for more than 48 hours.
Ar: unspecified purity.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Tin; Sn; [7440-31-5] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland September 1991

CRITICAL EVALUATION:

Several solubility determinations of Sn in liquid Na have been reported (1-6). The data of Lamprecht et al. (2,3) and of Hubberstey and Pulham (6) seem to be highly precise and show agreement within ± 2 %. The upper border of the scatter band of data of Weeks (4) is close to the results of (2,3) and (6), while the average of the scattered results indicates a significantly lower solubility by a factor of 0.5. The linear solubility equation in (4) has no thermodynamic basis and is only approximate. The recommended data of the solubility of Sn in liquid Na between 370 and 580 K may be expressed by the fitting equation proposed by Lamprecht (2,3) and confirmed by the evaluators:

$$\log(soly/\text{mol \% Sn}) = 4.356 - 2312\,(T/\text{K})^{-1} \qquad \text{Eq.(1)}$$

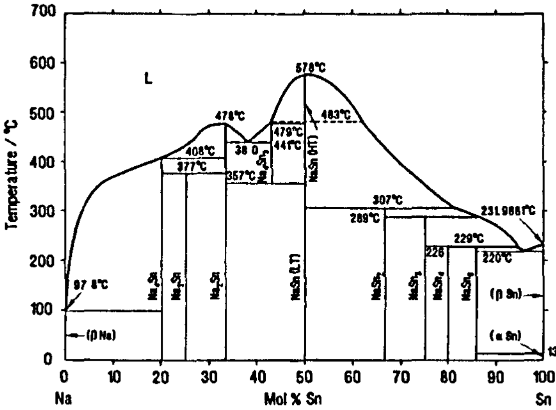
The relatively old results of Hume-Rothery (7) seem to be sufficiently precise and consistent with equation (1) at higher temperatures and concentrations of Sn up to 20 mol %. The liquidus data in this range obtained by Hubberstey and Castleman (5), Mathewson (8), and Takeda et al. (9,10) are scattered within ± 8 K, while the results of (4) significantly deviate. The extrapolated liquidus line of (6) is also situated at higher temperatures. The liquidus line at higher Sn content may be drawn between the results of (7), (8) and (9,10). The temperatures of the phase transitions between 35 and 50 mol % Sn recorded by (7) are overestimated by up to 40 K. The results of (7), (8), and (9,10) for Sn rich alloys agree within ± 5 K. The precise results of Heycock and Neville (11) for the most Sn-rich alloys are in excellent agreement with the subsequent data of (7). Hume-Rothery (14) reported the melting point of Na_2Sn at 743 K in a preliminary study and later corrected the value to 741 K (7). Brush (12) reported a smooth solubility line from the melting point of Na up to 55 mol % Sn at 728 K, the source of the solubility data was not specified. This information does obviously not give correct data, and any peritectic, eutectic and congruent melting points of the Sn-Na intermetallic are not reflected.

The Sn-Na phase diagram redrawn from (13) shows the phase relationships in the system. The compound $Na_{15}Sn_4$ instead of the earlier formulated Na_4Sn should be placed in the diagram.

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

T/K	soly/mol % Sn	source
373	0.014 (t)	(2,3) extrapolated, Eq.(1)
473	0.29 (r)	(2,3), (6) interpolated, Eq.(1)
573	2.3 (r)	(4), (6), (7) mean value, Eq.(1)
683	21.0 (t) peritectic	(5), (7), (8), (9,10) mean value
751	33 (t) congruent	(7), (8), (9,10) mean value
714	38 (t) peritectic	(7), (8) mean value
752	42 (t) peritectic	(7), (8) mean value
851	50.0 (r) congruent	(7), (8), (9,10) mean value

The liquid metals are completely miscible above 851 K.



References

1. Tammann, G. *Z. Phys. Chem.* 1889, *3*, 441.
2. Lamprecht, G.J.; Crowther, P.; Kemp, D.M., *J. Phys. Chem.* 1967, *71*, 4209.
3. Lamprecht, G.J. *Ph.D. thesis*, Univ. of South Africa, Pretoria, 1966.
4. Weeks, J.R. Brookhaven National Laboratory, 1969; cited by Claar, T.D. *Reactor Technol.* 1970, *13*, 124.
5. Hubberstey, P.; Castleman, A.W. *J. Electrochem. Soc.* 1972, *119*, 967.
6. Hubberstey, P.; Pulham, R.J. *J. Chem. Soc. Dalton Trans.* 1974, 1541.
7. Hume-Rothery, W. *J. Chem. Soc.* 1928, *131*, 947.
8. Mathewson, C.H., *Z. Anorg. Chem.* 1905, *46*, 94.
9. Takeda, S.; Matsunaga, S.; Tamaki, S. *J. Phys. Soc. Jap.* 1984, *53*, 1448.
10. Takeda, S.; Matsunaga, S.; Tamaki, S. *J. Non-Crystall. Solids* 1984, *61-62*, 29.
11. Heycock, C.T.; Neville, F.H., *J. Chem. Soc.* 1890, *57*, 376.
12. Brush, E.G. *Corrosion* 1955, *11*, 299.
13. Hansen, M.; Anderko, K. *Constitution of Binary Alloys*, McGraw-Hill, N.Y., 1958, p. 1007.
14. Hume-Rothery, W. *J. Inst. Met.* 1926, *35*, 347.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Tammann, G.

Z. Phys. Chem. 1889, *3*, 441-449.

VARIABLES:

Temperature difference: 0.07 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The depression of the melting point of Na due to the addition of Sn was determined.

ΔT/K	*soly*/g Sn per 100 g Na	*soly*/mol % Sn ^{a}
0.01	0.11	0.021
0.03	0.25	0.048
0.07	0.62	0.122

^{a} calculated by the compilers

The melting point of Na was reported to be 370 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Successive portions of Sn were added to molten Na. The resulting alloys were undercooled for up to 2 K. The precipitation of crystals was forced by effective moving a thermometer. Every determination of the temperature was three times repeated.

SOURCE AND PURITY OF MATERIALS:

Sn: nothing specified.
Na: "pure".

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.05 K.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Heycock, C.T.; Neville, F.H.

J. Chem. Soc. 1890, *57*, 376-393.

VARIABLES:

Temperature: 493-501 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the Sn-Na liquidus were determined.

t/°C	*soly*/mol Na per 100 mol Sn	*soly*/mol % Na ^{a}	*soly*/mol % Sn ^{a}
227.974	1.317	1.30	98.70
222.708	3.15	3.05	96.95
220.016	4.73	4.52	95.48

^{a} as calculated by the compilers

The melting point of Sn was determined to be 231.695 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed in a block of cast Fe. The Sn sample was covered with paraffin in order to prevent oxidation. The molten Sn was continuously stirred by means of a Fe stirrer. Na was directly added to the melt, the alloy was vigourously mixed. Carefully calibrated thermometers were used for the exact measurements of the freezing points of alloys.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.01 K.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Tin; Sn; [7440-31-5] (2) Sodium; Na; [7440-23-5]	Mathewson, C.H. *Z. Anorg. Chem.* 1905, *46*, 94-112.

VARIABLES:	PREPARED BY:
Temperature: 495-849 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Sn-Na system were determined.

t/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn
350	7.9	440	38.3	537	56.8
377	11.0	450	39.4	495	60.4
398	17.0	470	41.1	470	62.8
405	19.2	475	42.1	450	65.1
405	19.6	478	42.7	435	66.8
410	20.2	478	43.4	415	68.9
405	21.0	510	44.3	390	70.6
440	24.0	525	45.0	365	73.1
465	27.3	555	46.3	335	76.5
470	29.5	575	48.8	312	79.4
477	32.7	570	49.0	300	81.3
475	34.4	576	50.2	265	88.3
465	35.2	567	53.0	225	94.9
460	36.6	562	53.2	222	96.6
450	37.5				

The intermetallic phases $SnNa_4$, $SnNa_2$, Sn_3Na_4, SnNa, and Sn_2Na were identified. The melting points of Sn and Na were found at 232 and 97.5 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was melted under vaseline oil, cooled to freezing, and its surface was cut off. The metals were placed in a high melting Jena glass tube under H_2 atmosphere. The molten alloys were mixed by means of stirring with a Fe wire. A Pt/Pt-Rh thermoelement was used for temperature measurements in alloys containing up to 80 mol % Na. A Cu/Ni thermoelement was applied in the Na-richer alloys. The thermocouples were calibrated on the melting points of Na, Pb, Sn, Zn and Sb. Some alloys were analyzed by decomposition with H_2O. The formed NaOH was determined by means of acidimetric titration.

SOURCE AND PURITY OF MATERIALS:

Sn: nothing specified.
Na: purified by remelting and cutting off the surface layers.

ESTIMATED ERROR:

Solubility: precision ± 0.04 % in the alloy preparation and ± 0.5 mol % in the analysis.
Temperature: unspecified; precision ± 1 K (by the compilers).

REFERENCES:

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Hume-Rothery, W.

J. Chem. Soc. 1928, *131*, 947-963.

VARIABLES:

Temperature: 494-851 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

A large number of points on the liquidus line of the Sn-Na system were determined.

t/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn
231	99.62	308	80.55	450	39.10
230	98.98	312	80.03	448	38.90
225	98.12	321	78.46	443	38.07
223	96.88	331	77.97	438	35.85
221	95.91	381	72.59	449	36.19
224	95.09	433	65.32	466	34.12
232	94.19	450	64.17	478	33.3
238	93.91	489	60.95	478	32.57
248	92.21	523	58.97	476	29.87
253	91.72	543	56.74	469	27.79
254	91.37	564	53.79	449	25.16
266	89.91	575	51.48	430	23.15
267	90.03	578	50.0	419	21.90
268	89.21	574	47.57	407-408	20.0
269	88.99	559	46.10	406	18.88
278	87.18	529	44.39	398	16.95
289	86.11	480	42.34	391	14.96
289	85.75	473	41.27	367	9.94
293	84.09	458	39.89	333	4.80
297	83.15	457	39.69	290	2.10

The melting points of Sn and Na were found at 232 and 97.5 °C, respectively.
The intermetallic phases $SnNa_4$, $SnNa_3$, $SnNa_2$, Sn_3Na_4, SnNa, Sn_2Na, Sn_3Na, Sn_4Na, and Sn_6Na were detected in the system.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared from the metals in hard glass or Pyrex glass (for the Na-rich alloys) tubes under N_2 atmosphere. The tube was also equipped with a stirrer and a Chromel/Alumel thermocouple which was calibrated on the melting points of Al, Zn, Pb and Sn. Cooling and heating curves of the alloys were recorded. The composition of the alloys which were prepared from weighed amounts of the components was confirmed by chemical analyses.

SOURCE AND PURITY OF MATERIALS:

Sn: chemically pure from Capper Pass & Co.
Na: "electrolytic", no other metals were detected by ordinary methods of analysis.

ESTIMATED ERROR:

Solubility: precision ± 0.2 mol %.
Temperature: nothing specified; precision ± 0.5 K (by the compilers).

REFERENCES:

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Lamprecht, G.J.; Crowther, P.; Kemp, D.M.

J. Phys. Chem. 1967, *71*, 4209-4212.

VARIABLES:

Temperature: 393.2-531.0 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Sn in liquid Na was determined at several temperatures and was reported in graphical form. Numerical results were taken from (1).

t/°C	*soly*/mass % Sn	*soly*/mol % Sn
120.0[c]	0.153	0.0296
148.7[a]	0.399	0.0774
157.8[b]	0.496	0.0966
184.5[a]	1.052	0.205
199.6[b]	1.522	0.298
214.6[c]	2.134	0.423
229.0[a]	2.810	0.556
232.2[b]	2.774	0.550
257.8[a]	5.236	1.057

[a] heating sequence

[b] cooling sequence

[c] from the intermetallic compound ($SnNa_4$)

The solubility equation was derived by the authors and confirmed by the compilers.

$$\log (soly/\text{mol \% Sn}) = 4.356 - 2312\ (T/\text{K})^{-1}$$

The equilibrium solid phase was analyzed, the composition was found to be $Na_{3.99\pm0.04}Sn$ and the melting point was measured at 411 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility apparatus was made of Pyrex glass. He was used as cover gas in the experiments. Sn was melted with radioactive ^{113}Sn obtained by irradiation. Sn was filled in one of the reaction cells, Na in the other. The cells were interconnected by means of a capillary. Molten Na was transferred to the Sn cell by increased pressure. The two metals were in contact until no further change of the activity of Sn could be measured. The alloy was moved back to its primary cell for decay measurements. The variation of the solubility with temperature was determined by means of measuring the increase or decrease of the activity of the saturated alloy, since the equilibrium was reached from below and above the selected saturation temperature.

SOURCE AND PURITY OF MATERIALS:

Sn: spectrally pure from Johnson Matthey.
Na: from Merck with contents of 0.002 % Cl, SO_4 and heavy metals, 0.001 % Fe, PO_4, 0.005 % N, Ca, 0.01 % K and $(1.1\pm 0.2)\cdot10^{-3}$% O.
He: purified over molecular sieves, activated charcoal trapping at the temperature of liquid N_2.

ESTIMATED ERROR:

Solubility: standard deviation ± 4 %.
Temperature: stability better than ± 1 K.

REFERENCES:

1. Lamprecht, G.J. *Ph.D. thesis*, Univ. of South Africa, Pretoria, 1966.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Weeks, J.R.

Brookhaven National Laboratory, 1969, unpublished; as reported in (1).

VARIABLES:

Temperature: 443-675 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Sn in liquid Na was determined at several temperatures. The results were read-out from the figure and recalculated to mol % by the compilers.

T/K	t/°C	*soly*/mass % Sn	*soly*/mol % Sn
443	170	0.6, 0.7	0.12, 0.14
462	189	0.95, 1.1	0.19, 0.22
469	196	1.1, 1.2	0.22, 0.23
495	222	0.9	0.18
515	242	2.5, 2.6	0.49, 0.50
532	254	2.6	0.50
566	293	4.2, 4.4	0.82, 0.86
578	305	12	2.4
592	319	17	3.8
629	356	14, 28	2.7, 5.6
637	364	34	6.7
662	389	50	10.1
675	402	17	3.8

The fitting equation valid to up to 573 K was proposed by the author and tested by the compilers:

$$soly/\text{mass \% Sn} = 5.03 - 2326\ (T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Details of the method were not reported in (1), but the applied method was the same as described in (2). Sn and Na were introduced into a Zr crucible under an inert atmosphere. The apparatus was kept at 900 K for 48 hours and then cooled to the desired temperature at which it was held for 24 hours. Each sampler was preheated for 15 min and the alloy forced through the filter inside the sampler. The samples were analyzed and the procedure was repeated at another sampling temperature.

SOURCE AND PURITY OF MATERIALS:

Sn: nothing specified.
Na: 99.98 % pure from MSA Research (2).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Claar, T.D. *Reactor Technol.* 1970, *13*, 124-146.
2. Weeks, J.R.; Davies, H.A. *The Alkali Metals*, The Chem. Soc., London, 1967, p. 32-44.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Hubberstey, P.; Castleman, A.W.

J. Electrochem. Soc. 1972, *119*, 967-970.

VARIABLES:

Temperature: 613-688 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Sn in liquid Na was determined at various temperatures.

t/°C	*soly*/mol % Sn
340	5.92
355	7.46
376	10.20
389	12.72
401	15.15
413	18.12
415	19.04

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The electrochemical cell:
Na / Na^+(in Pyrex glass) / Na-Sn
was operated under He atmosphere. The apparatus was made of Pyrex glass, while stainless steel was applied for electrical connections. The apparatus was evacuated and baked at ~ 773 K for several hours. The weighed metals were introduced into a dry atmosphere box. The cell was evacuated, sealed and heated over night to ~ 25 K above the estimated liquidus temperature. The temperature was then decreased in steps of 5 K. The Na activity became constant in the two-phase region, thus indicating the liquidus temperature. Chromel/Alumel thermocouples were used to measure the temperature.

SOURCE AND PURITY OF MATERIALS:

Sn: 99.9 % pure from Allied Chem. Corp..
Na: 99.9 % pure from J.T. Baker Comp., pipetted at ~ 393 K in order to reduce concentrations of non-metals to $<1\cdot10^{-3}$ %.
He: with contents of $<1\cdot10^{-4}$ % O and $<1\cdot10^{-5}$ % H_2O.

ESTIMATED ERROR:

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

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Tin; Sn; [7440-31-5] (2) Sodium; Na; [7440-23-5]	Hubberstey, P.; Pulham, R.J. *J. Chem. Soc.,Dalton Trans.* 1974, 1541-1544.
VARIABLES:	**PREPARED BY:**
Temperature: 455-645 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Sn in liquid Na was measured at several temperatures. The data were read-off from the figures by the compilers.

t/°C	*soly*/mol % Sn	t/°C	*soly*/mol % Sn
182	0.22	308	2.6
210	0.42	320	3.0
235	0.65	330	3.6
256	0.95	345	4.3
270	1.15	356	5.3
281	1.4	372	6.9
301	2.0		

The melting point of Na was measured to be 97.83 °C. The equilibrium solid phase was identified as Na_4Sn.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The resistivity measurement apparatus was made of stainless steel (1). The alloys were prepared after weighing appropriate quantities of the metals under Ar atmosphere by means of heating to 673 K. An electromagnetic pump served to stir and homogenize the solutions. Simultaneous measurements of the resistance and the temperature were performed during the cooling of the metallic solutions. The temperature at which the saturation of Na with Sn occurred was indicated by an abrupt change of the resistance of the sample.

SOURCE AND PURITY OF MATERIALS:

Sn: 99.96 % pure from British Drug Houses.
Na: nothing specified, probably as in (1) containing $< 4\cdot10^{-3}$ % Ca and $< 1\cdot10^{-3}$ % O.

ESTIMATED ERROR:

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

REFERENCES:

1. Addison, C.C.; Creffield, G.K.; Hubberstey, P.; Pulham, R.J. *J. Chem. Soc.,A* 1969, 1482-1487.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Takeda, S.; Matsunaga, S.; Tamaki, S.

J. Phys. Soc. Jap. 1984, *53*, 1448-1452.

VARIABLES:

Temperature: 623-859 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the Sn-Na system were determined.

t/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn
375	10	503	43
388	14.6	568	45
427	20	586	50
454	25	542	55
480	30	513	60
485	34.3	384	70
476	40	350	>80

The results were also partly published in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Samples of the Sn-Na alloys were prepared in quartz or Pyrex glass ampoules which were sealed under vacuum. Measurements of the magnetic susceptibility of the samples were performed by means of the torsion balance method. The temperature of the tests was changed within 50 K below and 150 K above the estimated liquidus temperature. The liquidus temperatures were indicated by sharp changes of the susceptibility versus temperature dependencies.

SOURCE AND PURITY OF MATERIALS:

Sn: 99.999 % pure.
Na: 99.9 % pure.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: nothing specified.

REFERENCES:

1. Takeda, S.; Matsunaga, S.; Tamaki, S. *J. Non-Crystall. Solids* 1984, *61-62*, 29-34.

COMPONENTS:	EVALUATOR:
(1) Tin; Sn; [7440-31-5] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1991

CRITICAL EVALUATION:

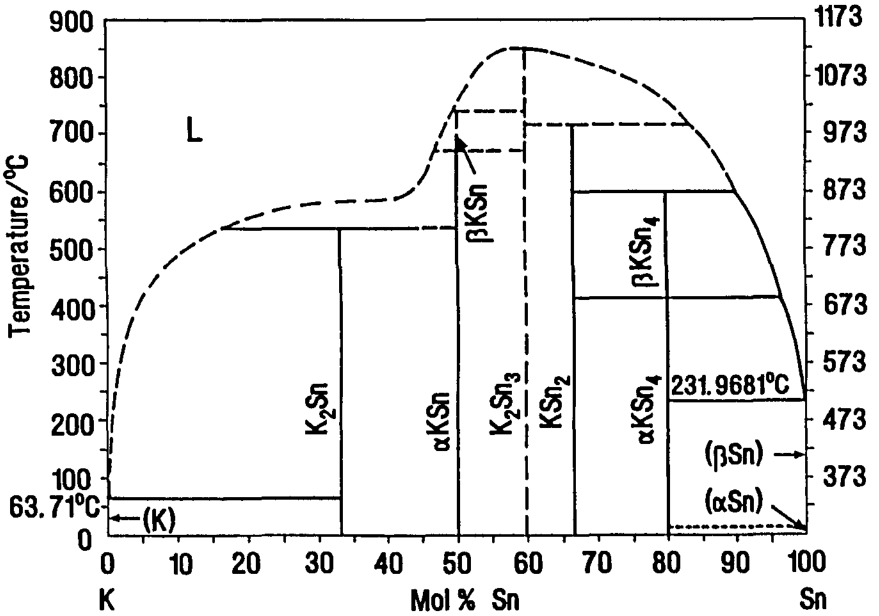
Oxidation of the alloys caused experimental difficulties in the study of the K-Sn system as was first reported by Heycock and Neville (1) who were not able to perform thermal analysis. Smith (2) investigated several alloys, the thermal arrests which were obtained at temperatures above 800 K were not certain, since the liquid alloys as well as the K vapour attacked parts of his apparatus. Hewaidi et al. (3) found a flat maximum at 1103 K for the equimolar SnK alloy by means of thermal analysis.

Drits et al. (4) performed thermal analyses of the K-Sn alloys in the whole range of compositions. Thermomagnetic measurements were carried out by Takeda and Tamaki (5). These two groups did not mention any difficulties in their studies which seem to be reliable. In spite of that their results differ significantly in many details. The observations of (3) that KSn ought to be the most stable compound in the system was confirmed in (5). The statement of (2) on the highest stability of the compound K_2Sn_3 was, however, confirmed by (4). Data of (5) suggested an eutectic point on the K-rich side which contained about 7 mol % Sn. The composition of this eutectic was claimed to be at maximum 1 mol % Sn by (2) and (4). These data (2,4) are selected as the more reliable ones due to their agreement. The system needs, however, further experiments on the K-rich side to establish the phase diagram.

The existence of some equilibrium solid phases is well proved: K_2Sn (2,4-6), KSn (2-6), KSn_2 (2,4-6), and KSn_4 (2,4-6). The formation of others as K_2Sn_3 (4) and K_4Sn (6) needs further confirmation. A schematic phase diagram after (4) is redrawn in the figure.

Doubtful data of the solubility of Sn in liquid K

T/K	*soly*/mol % Sn	source
773	11	(2); extrapolated
873	44	(4); interpolated
973	49	(4); interpolated
1013	50 peritectic	(4); interpolated
1073	52	(4)
1133	60	(4)



References

1. Heycock, C.T.; Neville,F.H. *J. Chem. Soc.* 1890, *57*, 376.
2. Smith, D.P. *Z. Anorg. Chem.* 1908, *56*, 109.
3. Hewaidy, I.F.; Busmann, E.; Klemm, W. *Z. Anorg. Chem.* 1964, *328*, 283.
4. Drits, M.E.; Fridman, A.S.; Zusman, L.L.; Kusikov, V.A. *Fazovye Ravnovesiya v Metallicheskikh Splavakh*, Nauka, Moskva, 1981, p. 176.
5. Takeda, S.; Tamaki, S. *J. Phys., F* 1988, *18*, L45.
6. Gukova, Yu.Ya.; Ermolaev, M.I. *Obshchie Zakonomernosti v Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, p. 135.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Smith, D.P.

Z. Anorg. Chem. 1908, *56*, 109-142.

VARIABLES:

Temperature: 662-1097 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Sn-K liquidus were determined.

t/°C	*soly*/mol % Sn
510	13.0
524	20.0
794 [a]	57.0
824 [a]	60.0
760 [a]	70.0
669 [a]	80.0
389	97.0

[a] these values are somewhat uncertain due to possible attack of the alloy on the container

The melting points of Sn and K were found to be at 232 and 63 °C, respectively. The solid phases SnK_2, SnK, Sn_2K, and Sn_4K were identified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Metallic K was purified by means of cutting off the surface scales and washing in benzene, amylalcohol, petroleum, and ether. Both metals were weighed (K under petroleum) and placed in a tube of hard Jena glass. The tube was filled with H_2 and the metals were melted by heating. The tube was placed in an apparatus for thermal analysis. Cooling curves were recorded, the temperatures were measured by means of a calibrated Pt/Pt-Rh thermocouple. A glass rod served for stirring the alloy during the solidification. Alloys with contents of 20 to 80 mol % Sn attacked the apparatus. The measurements were therefore not precise and irreproducible.

SOURCE AND PURITY OF MATERIALS:

Sn: nothing specified.
K: "contained no traces of Na as well as of other metals".

ESTIMATED ERROR:

Nothing specified.
Temperature: precision not better than ± 3 K (by the compilers).

REFERENCES:

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Hewaidy, I.F.; Busmann, E.; Klemm, W.

Z. Anorg. Chem. 1964, *328*, 283-293.

VARIABLES:

One temperature: 1103 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The melting temperature of the alloy with 50 mol % Sn was determined at 830 °C. Unspecified but lower temperatures were recorded on the sides of the system with higher Sn or K contents.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The preparation of the alloy was performed in a corund crucible under Ar atmosphere. The crucible was encapsulated in a steel bomb. The melting temperatures of the samples were measured by means of thermal analyses. The K content of the alloys was determined after hydrolysis with H_2O vapour and titration of the solution with HCl. Sn was gravimetrically determined as SnO_2.

SOURCE AND PURITY OF MATERIALS:

Sn: nothing specified.

K: commercial product, purified by distillation under high vacuum.

ESTIMATED ERROR:

Solubility: accuracy ± 0.2 %.

Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Drits, M.E.; Fridman, A.S.; Zusman, L.L.; Kusikov, V.A.

Fazovye Ravnovesiya v Metallicheskikh Splavakh, Nauka, Moskva, 1981, p. 176-178.

VARIABLES:

Temperature: 743-1133 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the Sn-K system were determined. The data were extracted from the figure by the compilers.

t/°C	*soly*/mol % Sn	t/°C	*soly*/mol % Sn	t/°C	*soly*/mol % Sn
555	28.5	855	57.5	820	68.5
575	39.0	860	60.0	770	78.5
660	47.0	850	63.0	680	86.5
820	52.5	840	65.0	470	95.5

The equilibrium solid phases SnK_2, SnK, Sn_3K_2, Sn_2K, and Sn_4K were identified in the system.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were introduced into a Mo crucible in an Ar atmosphere. The crucible was hermetically closed. Its tightness and corrosion resistance was tested by means of heating and cooling the alloy samples in the container several times. Thermal analyses of the alloys were performed. The alloys were analyzed for their Sn contents by an unspecified method.

SOURCE AND PURITY OF MATERIALS:

Sn: 99.9995 % pure, denoted as "OChV-000".

K: 99.96 % pure.

ESTIMATED ERROR:

Nothing specified.

Solubility: read-out procedure ± 0.5 mol %.

Temperature: read-out procedure ± 5 K.

REFERENCES:

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Takeda, S.; Tamaki, S.

J. Phys. F 1988, *18*, L45-L48.

VARIABLES:

Temperature: 434-1140 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Sn-K system were determined.

t/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn
161	10	867	50.2
439	15.2	862	59.8
566	20	798	67
679	25	700	74.2
810	45.2	610	90

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Samples of the Sn-K alloys were prepared in quartz or Pyrex glass ampoules by repeated melting in an Ar atmosphere and sealed under vacuum. Measurements of the magnetic susceptibility were performed by means of the usual torsion balance method. The temperature of the measurement was varied from 50 K below to 50 K above the expected liquidus temperature. Abrupt changes of the temperature dependence of the susceptibility indicated the liquidus temperature.

SOURCE AND PURITY OF MATERIALS:

Sn: 99.999 % pure.
K: 99.9 % pure.

ESTIMATED ERROR:

Nothing specified.
Temperature: precision ± 2 K (by the compilers).

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Tin; Sn; [7440-31-5] (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:

The first study of the Rb-Sn phase diagram was performed by Kuznetsov (1), but his thesis was not available to the evaluators. Only smoothed lines on a small figure of the corresponding phase relations were reported in an abstract. According to (1) the solubility of Sn in liquid Rb increases moderately to a value of about 3 mol % Sn at about 830 K. A miscibility gap extends from ~3 to 40 mol % Sn at this temperature, and the critical miscibility temperature is ~ 40 K higher. In the Sn-rich part of the system the liquidus line strongly increases to more than 1300 K. The maximum of a congruently melting compound was extrapolated to be either at a composition of SnRb or of Sn_3Rb_2. A further increase of the Sn content causes a decrease of the liquidus temperature down to the melting temperature of Sn.

Drits et al. (2) studied the Rb-Sn phase diagram by means of thermal analysis in the whole range of compositions. They observed the same equilibrium solid phases, RbSn, Rb_2Sn_3, $RbSn_2$, and $RbSn_4$, as in (1). The liquidus arrests were, however, found at temperatures up to 200 K below those in (1). The miscibility gap was reported in (2) at a higher temperature of 883 K in the range between 14 and 41 mol % Sn.

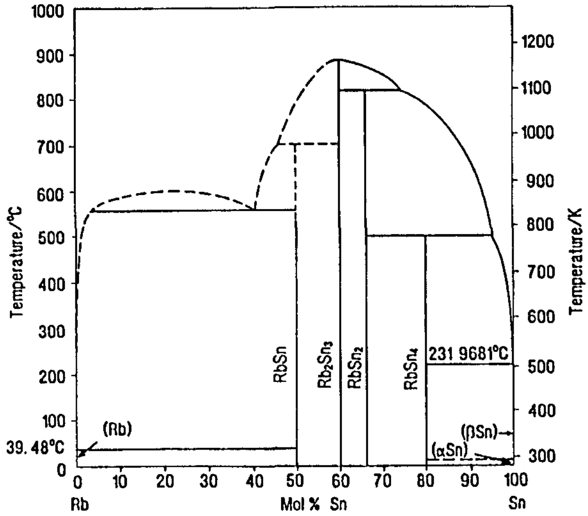
The evaluators assume the Rb-rich part of the diagram presented by (1) to be more reliable, while the Sn-rich part reported by (2) seems to be more convincing.

Gukova and Ermolaev (3) identified five equilibrium solid phases: Rb_4Sn, Rb_2Sn, RbSn, $RbSn_2$, and $RbSn_4$. A schematic phase diagram was compiled on the basis of the data of (1) and (2) and is presented in the figure.

Doubtful values of the solubility of Sn in liquid Rb

T/K	*soly*/mol % Sn	source
~830	~3 monotectic	(1)
973	45	(2)
1073	50	(2) interpolated
1163	60	(2) extrapolated

The two liquid metals are completely miscible above 1163 K.



References

1. Kuznetsov, A.N. *Ph.D. thesis*, Inst. Khim. Akad. Nauk SSSR, Sverdlovsk, 1978; abstracted in Chuntonov, K.A.; Yatsenko, S.P. *Zh. Fiz. Khim.* 1978, *52*, 2145.
2. Drits, M.E.; Fridman, A.S.; Zusman, L.L.; Kusikov, V.A. *Fazovye Ravnovesiya v Metallicheskikh Splavakh*, Nauka, Moskva, 1981, p. 176.
3. Gukova, Yu.Ya.; Ermolaev, M.I. *Obshchie Zakonomernosti v Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, p. 135.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Drits, M.E.; Fridman, A.S.; Zusman, L.L.; Kusikov, V.A.
Fazovye Ravnovesiya v Metallicheskikh Splavakh, Nauka, Moskva, 1981, p. 176-178.

VARIABLES:

Temperature: 808-1153 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the Sn-Rb system were determined. The data were extracted from the figure by the compilers.

t/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn
710	45.5	810	74.5
835	52.0	770	81.0
875	56.0	715	87.0
880	61.5	640	90.5
870	63.5	535	94.0
855	68.5		

The monotectic at 610 °C was found between about 15 and 42 mol % Sn.

The equilibrium solid phases in the system are: SnRb, Sn_3Rb_2, Sn_2Rb, and Sn_4Rb.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were introduced into a Mo crucible in an Ar atmosphere. The crucible was hermetically closed. Its tightness and corrosion resistance was tested by means of heating and cooling the alloy samples in the container several times. Thermal analyses of the alloys were performed. The alloys were analyzed for their Sn contents by an unspecified method.

SOURCE AND PURITY OF MATERIALS:

Sn: 99.9995 % pure, denoted as "OChV-000".
Rb: 99.96 % pure.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.5 mol %.
Temperature: read-out procedure ± 5 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Tin; Sn; [7440-31-5] (2) Caesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland October 1991

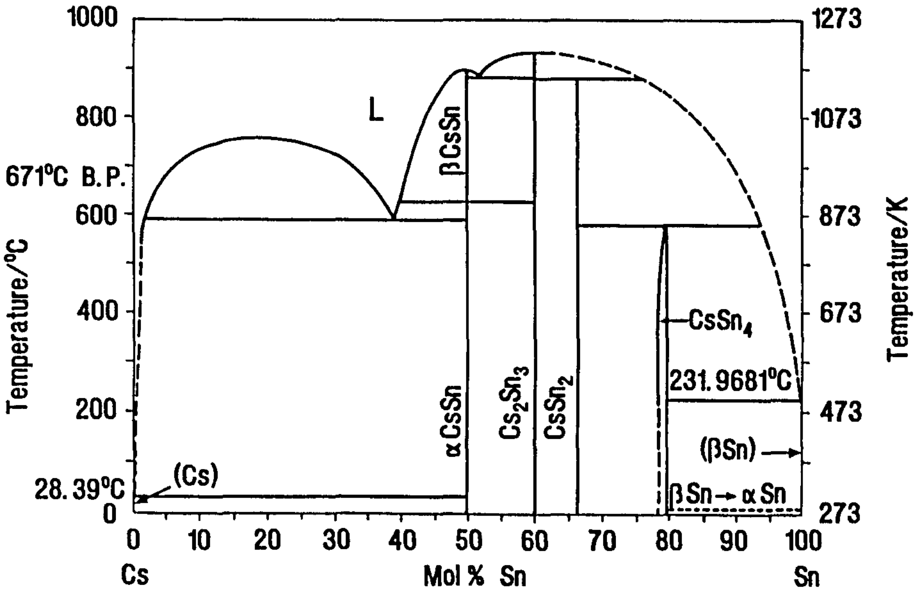
CRITICAL EVALUATION:

The results obtained by two groups of investigators, (1,2) and (3,4), agree roughly in the Sn-rich range of the system. The compound Cs_2Sn_3 with congruent melting point at 1203 K (1,2) or 1208 K (3,4) was found in both studies. On the Cs-rich side Drits et al. (1,2) detected a peritectic decomposition of CsSn at 903 K, whereas Yatsenko et al. (3,4) reported congruent melting of CsSn at 1157 K and its phase transformation at 903 K. The miscibility gap should be between 10 and 40 mol % Sn according to (1,2), though no experimental points were obtained in this part of the liquidus. The gap was located between 2 and 39 mol % Sn by Yatsenko et al (3,4).
It seems that the more reliable results in the Cs-rich part of the system were obtained by (3,4) in analogy to the Sn-Rb system. The analogy indicates that the results of (1,2) seem to be more reliable in the Sn-rich side. The schematic phase diagram (see figure) was prepared on the basis of these presumptions.
A number of equilibrium solid phases are well proved: $CsSn_2$ (1-5), Cs_2Sn_3 (1-4), CsSn (1-6), Cs_2Sn ((1-5). The existence of ~$CsSn_6$ (3,4), $CsSn_4$ (1,2,5), $CsSn_3$ (5) and Cs_4Sn (5) needs to be confirmed. Strobel and Hatterer (6) reported CsSn to be the only stable phase.

Doubtful values of the solubility of Sn in liquid Cs

T/K	*soly*/mol % Sn	source
863	~2 monotectic	(3,4)
1033	20 critical point	(3,4)
1073	44	(3,4) interpolation
1173	56	(1,2) interpolation
1205	60	(1-4) mean value of temperatures

Both liquid metals are completely miscible above 1205 K.



References
1. Drits, M.E.; Fridman, A.S.; Zusman, L.L.; Kusikov, V.A. *Fazovye Ravnovesiya v Metallicheskikh Splavakh*, Nauka, Moskva, 1981, p. 176.
2. Drits, M.E.; Fridman, A.S.; Zusman, L.L.; Kusikov, V.A. *Izv. Akad. Nauk SSSR, Met.* 1979, no. 6, 204.
3. Yatsenko, S.P.; Melekhov, L.Z.; Chuntonov, K.A.; Kuznetsov, A.N.; Miroshnikova, L.D. *Dep. VINITI no. 5974-82*, 1982.
4. Melekhov, L.Z.; Yatsenko, S.P.; Chuntonov, K.A.; Grin, Yu.N. *Izv. Akad. Nauk SSSR, Met.* 1987, no. 2, 201.
5. Gukova, Yu.Ya.; Ermolaev, M.I. *Obshchie Zakonomernosti v Stroenii Diagram Sostoyaniya Metallicheskikh Sistem*, Nauka, Moskva, 1973, p. 135.
6. Strobel, P.; Hatterer, A. *J. Chim. Phys.* 1979, *76*, 375.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Drits, M.E.; Fridman, A.S.; Zusman, L.L.; Kusikov, V.A.
Fazovye Ravnovesiya v Metallicheskikh Splavakh, Nauka, Moskva, 1981, p. 176-178.

VARIABLES:

Temperature: 653-1303 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the Sn-Cs system were determined. The data were extracted from the figure by the compilers.

t/°C	*soly*/mol % Sn	*t*/°C	*soly*/mol % Sn
380	6	930	60
630	42	924	62.5
730	46	914	70.5
762	47.5	894	72.5
790	49	875	76
849	51	724	88.5
912	56.5	470	97

The monotectic at 605 °C was found between about 10 and 39 mol % Sn.

The equilibrium solid phases in the system are: $SnCs_2$, SnCs, Sn_3Cs_2, Sn_2Cs, and Sn_4Cs. The results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were introduced into a Mo crucible in an Ar atmosphere. The crucible was hermetically closed. Its tightness and corrosion resistance was tested by means of heating and cooling the alloy samples in the container several times. Thermal analyses of the alloys were performed. The alloys were analyzed for their Sn contents by an unspecified method.

SOURCE AND PURITY OF MATERIALS:

Sn: 99.9995 % pure, denoted as "OChV-000".
Cs: 99.96 % pure.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.3 mol %.
Temperature: read-out procedure ± 3 K.

REFERENCES:

1. Drits, M.E.; Fridman, A.S.; Zusman, L.L.; Kusikov, V.A. *Izv. Akad. Nauk SSSR, Met.* 1979, no. 6, 204-206.

COMPONENTS:

(1) Tin; Sn; [7440-31-5]

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

ORIGINAL MEASUREMENTS:

Melekhov, L.Z.; Yatsenko, S.P.; Chuntonov, K.A.; Grin, Yu.N.
Izv. Akad. Nauk SSSR, Met. 1987, no. 2, 201-203.

VARIABLES:

Temperature: 853-1208 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the Sn-Cs system were determined.

t/°C	*soly*/mol % Sn	t/°C	*soly*/mol % Sn	t/°C	*soly*/mol % Sn
721 [a]	10	903 [c]	53	866 [c]	69
760 [a]	20	925 [c]	57	853 [c]	70.5
718 [a]	30	935 [c]	60	847 [c]	74.5
660 [b]	36.7	914 [c]	62.5	821 [c]	84.5
600 [b]	38.2	903 [c]	64	779 [c]	89.5
821 [c]	44.6	890 [c]	66	692 [c]	97
883 [c]	50	868 [c]	67	580 [c]	99.5

[a] from measurements of the magnetic susceptibility
[b] from solubility measurements
[c] from differential thermal analysis

All data except the solubility measurements (b) were also reported in (1). Several solid equilibrium phases were found: ~Sn_6Cs, Sn_2Cs, Sn_3Cs_2, and SnCs.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Weighed amounts of the two metals were placed in a Ta crucible which was laser beam welded under vacuum. The differential thermal analysis of the alloy samples was performed in a suitable commercial apparatus. The magnetic susceptibility was measured by means of the Faraday method in high vacuum at various temperatures. The liquidus temperature was estimated as corresponding to a break point of the thermomagnetic curve. Solubility measurements were performed in 12Kh18N10T thin walled stainless steel tubes. They were filled with the alloys and hermetically closed before the equilibration at a selected temperature. The tube was then divided at the level at which the upper of the two immiscible phases was found. This phase was evaluated by means of chemical analysis.

SOURCE AND PURITY OF MATERIALS:

Sn: 99.999 % pure.
Cs: 99.98 % pure.

ESTIMATED ERROR:

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

REFERENCES:

1. Yatsenko, S.P.; Melekhov, L.Z.; Chuntonov, K.A.; Kuznetsov, A.N.; Miroshnikova, L.D. *Dep. VINITI no. 5974-82*, 1982.

COMPONENTS:	EVALUATOR:
Lead; Pb; [7439-92-1] Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland August 1992

CRITICAL EVALUATION:

The liquidus line of the Pb-Li system was studied by means of various techniques. The use of thermal analysis was reported in (1,2,3,4,9,13), (5) measured the vapour pressure of Li by means of Knudsen-effusion mass spectrometry. The other methods were potentiometry (6), calorimetry (7,10,13), resistivity (8,13,15), and density measurements (11). Grube and Klaiber (2) were the only ones who studied the whole range of compositions. Meijer (13) reported a complete confirmation of the results of (2), but he did not present numerical values or a figure. The data of (4) are not compiled because of the lack of experimental information.

The composition of the eutectic at 83 mol % Pb and 508 K which seemed to be well established by (1,2,3,7,10) was recently redetermined by Hubberstey et al. (15) to be 84.3 mol % Pb; the melting point of the intermetallic phase LiPb was found to be 757±1 K (2,4,10,12). The liquidus line of (2) between 30 and 40 mol % Pb was modified in the measurements of (5,6,11). The melting point of Li_7Pb_2 was determined to be 999 K (2,12); a similar value is obtained, if one extrapolates the data of (11). The fragmentary liquidus data of (9) who give the melting point of Li_7Pb_2 as much as 26 K higher seem to be overestimated in spite of the fact that the authors claim the advantageous use of a sealed capsule in order to prevent the distillation of Li. The liquidus temperatures reported by (1) were up to 100 K higher than the accepted values of (2),(11) and (15).

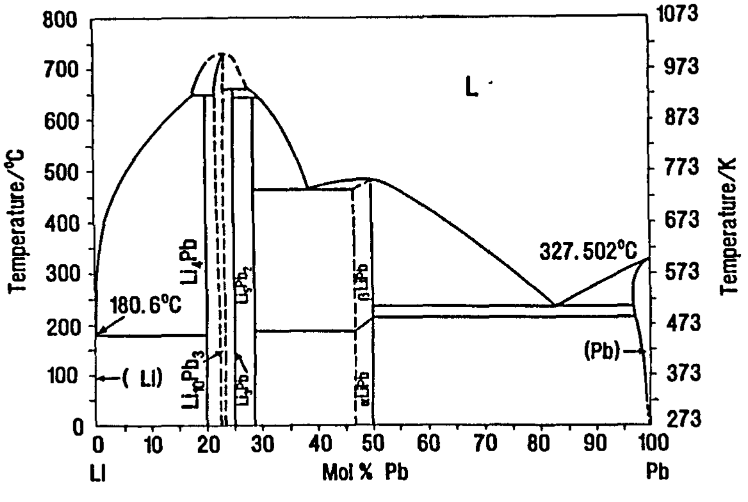
The solubility determinations of Dadd et al. (8) are in better agreement with the results of Saar and Ruppersberg (11) than with those of Grube and Klaiber (2) in the range of higher Pb concentrations. The solubility equation proposed by (8) was proved to be applicable in the temperature range 500 to 700 K:

$$\log (soly/\text{mol \% Pb}) = 4.483 - 2919(T/\text{K})^{-1} \qquad \text{Eq.1}$$

Saturated solutions of Pb in liquid Li are in equilibrium with Pb-Li intermetallic compounds. This is indicated in the phase diagram based on (2), taking into account the information in (14) and in this evaluation.

Tentative values of the solubility of Pb in liquid Li

T/K	$soly$/mol % Pb	source
500	0.044	(8); Eq.(1)
600	0.41	(8); Eq.(1)
700	2.1	(8); Eq.(1)
800	7	(2),(11); interpolation
900	15	(11)
920	17 peritectic	(2)
~ 999	22.2 congruent	(2),(12)



References

1. Czochralski, J.; Rassow, E. *Z. Metallk.* 1927, *19*, 111.
2. Grube, G.; Klaiber, H. *Z. Elektrochem.* 1934, *40*, 745.
3. Pogodin, S.A.; Shpichinetskii, E.S. *Izv. Sekt. Fiz.-Khim. Anal.* 1947, *15*, 88.
4. Zalkin, A.; Ramsey, W.J. *J. Phys. Chem.* 1957, *61*, 1413.
5. Neubert, A. *J. Chem. Thermodyn.* 1979, *11*, 971.
6. Becker, W.; Schwitzgebel, G.; Ruppersberg, H. *Z. Metallk.* 1981, *72*, 186.
7 Reiter, F.; Rota, R.; Camposilavan, J. *Fusion Technology* , Pergamon, Oxford, 1982, p. 671.
8. Dadd, A.T.; Hubberstey, P.; Roberts, P.G. *J. Chem. Soc., Faraday Trans. I* 1982, *78*, 2735.
9. Watson, W.R.; Pulham, R.J. *Liquid Metals Engineering and Technology*, Brit. Nucl. Ener. Soc., London, 1985, *3*, 99.
10. Jauch, U.; Karcher, V.; Schulz, B. *Kernforschungszentrum Karlsruhe Rep. KfK-4144*, 1986.
11. Saar, J.; Ruppersberg, H. *J. Phys..F* 1987, *17*, 305.
12. Sommer, F. *Z. Phys. Chem.*, *N.F.* 1988, *156*, 593.
13. Meijer, J.A. *Ph.D. thesis*, Univ. of Groningen, Netherlands, 1988, p. 41.
14. Elliott, R.P. *Constitution of Binary Alloys*, McGraw-Hill, New York, 1965, p. 585.
15. Hubberstey, P.; Sample, T.; Barker, M.G. *J.Nucl.Mater.* 1992, *191-194*, 283.

COMPONENTS:

Lead; Pb; [7439-92-1]

Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Czochralski, J.; Rassow, E.

Z. Metallk. 1927, *19*, 111-112.

VARIABLES:

Temperature: 503-743 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Pb-Li system were determined.

t/°C	*soly*/mass % Li	*soly*/mol % Pb[a]	t/°C	*soly*/mass % Li	*soly*/mol % Pb[a]
329	0.02	99.4	240	0.6	84.7
311	0.04	98.8	230[b]	0.65	83.7
309	0.09	97.4	330	0.8	80.6
310	0.18	94.9	380	1.05	75.9
295	0.24	93.4	400	1.2	73.4
289	0.27	92.5	440	1.4	70.2
272	0.36	91.2	465	1.75	63.3
255	0.5	87.3	470	2.15	60.4
250	0.55	86.0			

[a] calculated by the compilers
[b] eutectic

The melting point of Pb was determined to be at 329 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared and investigated in a fire-clay crucible. The alloys were covered with a LiCl-LiF melt in order to protect them against the atmosphere. The composition of each of the alloys was determined by means of chemical analysis. Cooling curves of the molten alloys were measured.

SOURCE AND PURITY OF MATERIALS:

Pb: "purest available".
Li: 98 % pure.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

Lead; Pb; [7439-92-1]

Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Grube, G.; Klaiber, H.

Z. Elektrochem. 1934, *40*, 745-753.

VARIABLES:

Temperature: 508-999 k

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Various points on the liquidus line of the Pb-Li system were determined.

t/°C	*soly*/mol %Pb	*t*/°C	*soly*/mol %Pb	*t*/°C	*soly*/mol %Pb
326	99.0	480	50.6	683	26.1
324	98.0	482	50.0	693	25.1
320	97.0	482	49.0	700	24.7
314	96.5	481.5	48.2	714	24.0
310	96.0	481	47.2	724	22.9
306	95.0	480	46.0	726	22.2 congruent
275	90.0	476	45.0	724	21.9
244	85.0	470	40.1	719	20.8
235	83.0 eutectic	537	35.0	710	19.9
261	80.0	612	32.5	693	18.9
307	75.0	641	30.0	674	18.2
340	68.8	644	29.6	642	17.0
390	65.0	649	28.7	637	16.5
427	59.7	658	27.9	627	16.0
462	55.0	664	27.3	545	10.0
470	52.5	668	26.8	488	5.0

The melting points of Pb and Li were found to be 327 and 179 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a crucible which was made of Armco Fe, this material served also to shield the thermocouple. The alloys did not attack the crucible and shielding. The mixture in the crucible was melted under Ar atmosphere. Cooling and heating curves were determined by means of a Ni/Ni-Cr thermocouple which was previously calibrated on the melting points of Sn, Cd, Zn, and Mg. The composition of the majority of the alloys was confirmed by chemical analysis.

SOURCE AND PURITY OF MATERIALS:

Pb: "purest" from Kahlbaum.
Li: 99.0 % pure from Metallgesellschaft; with contents of 0.62 % K, 0.14 % Na, 0.02 % Fe_2O_3, 0.05 % SiO_2, 0.32 % Li_3N, and traces of Al_2O_3.
Ar: 98.2 % pure with 0.1 % O_2 and 1.7 % N_2; further purified by means of bubbling through molten Li.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

Lead; Pb; [7439-92-1]

Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Pogodin, S.A.; Shpichinetskii, E.S.

Izv. Sekt. Fiz.-Khim. Anal. 1947, *15*, 88-95.

VARIABLES:

Temperature: 579-508.7 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the liquidus line of the Pb-Li system were determined.

t/°C	*soly*/mol % Li	*soly*/mol % Pb[a]
306	4.29	95.71
280	9.00	91.00
261	11.18	88.82
235.5	16.35	83.65 eutectic
250	17.99	82.01

[a] calculated by the compilers

The melting point of Pb was found to be 327 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Pb-rich alloys were prepared in a Fe crucible under a cover of molten paraffin. Li was introduced into molten Pb under vigourous stirring. The compositions of the alloys were confirmed by chemical analyses. The input alloy was remelted with various amounts of Pb in vacuo. Finally, cooling curves were recorded by means of a pyrometer.

SOURCE AND PURITY OF MATERIALS:

Pb: chemically pure from Laborreaktivsbyt.
Li: from Giretmet with contents of 0.14 % Fe; 0.09 % (Na+K), Cl; 0.07 % Si.

ESTIMATED ERROR:

Solubility: better than ± 3 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
Lead; Pb; [7439-92-1] Lithium; Li; [7439-93-2]	Neubert, A. *J. Chem. Thermodyn.* 1979, *11*, 971-977.
VARIABLES:	**PREPARED BY:**
Temperature: 750-850 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Three points on the liquidus line of the Pb-Li system were determined.

T/K	*soly*/mol % Pb
750	39.2
800	37.4
850	35.2

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloy samples were prepared from weighed amounts of the metals which were mixed and melted together at a temperature of 20 K above the liquidus temperature. The vapour pressure of Li over the liquid and solid alloys was measured using Knudsen-effusion mass spectrometry at selected temperatures. The Knudsen cells were made of Mo, the liners were of Armco Fe or stainless steel. These materials were not corroded by the molten alloys. The temperature was measured by means of a Ni/Ni-Cr thermocouple which was calibrated on the melting point of Ag. The liquidus points were indicated by changes of the slope of the relation of Li activity versus the composition of the alloy.

SOURCE AND PURITY OF MATERIALS:

Pb: "purissimum A" grade from Fluka AG.
Li: 99.99 % pure from ROC/RIC, Sun Valley, Calif., USA.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

Lead; Pb; [7439-92-1]

Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Becker, W.; Schwitzgebel, G.; Ruppersberg, H.

Z. Metallk. 1981, *72*, 186-190.

VARIABLES:

Two temperatures: 800 and 900 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of two Pb-Li alloys were determined.

T/K	*soly*/mol %Pb
800	38.0
900	31.6

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by melting the metals inside an Ar glove box. They were analyzed by means of (unspecified) "normal techniques". The potentials of the cell Li/LiCl-LiF/Pb-Li were measured at the two temperatures. The cell was assembled in the glove box. The crucible which contained the cell was made of Al_2O_3, BeO or Fe, and the electrode container was of Fe. The cell was encapsuled in a quartz tube together with a Mo/Ni thermocouple which served to measure the cell temperature.

The solubility was indicated by the change of the slope of the relations of potential versus composition.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.999 % pure from Roth.
Li: 99.9 % pure from Ventron.
LiCl-LiF: "suprapure" from Merck; dry Cl_2 was bubbled through the melt for 3 hours, the melt was flushed with dry He and then solidified.
He: "dry".
Ar: containing less than $1 \cdot 10^{-4}$ % O, H_2O and N (each).

ESTIMATED ERROR:

Solubility: nothing specified; ± 0.1 mol % (by the compilers).
Temperature: stability ± 0.5 K.

REFERENCES:

COMPONENTS:

Lead; Pb; [7439-92-1]

Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Reiter, F.; Rota, R.; Camposilavan, J.

Fusion Technology, Pergamon, Oxford, 1982, p. 671-674.

VARIABLES:

One temperature: 507.9 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The melting point of the eutectic mixture with 83 mol % Pb was found at 507.9 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloy was prepared from weighed amounts of the components in a vacuum chamber. Test cells of type 304 stainless steel were baked in vacuum at 1073 K for several hours. They were filled with the liquid alloy and closed by electron beam welding. Enthalpy measurements of each cell were performed twice in a differential heat flow calorimeter. A Ni/Ni-Cr thermocouple which was precisely calibrated on the melting point of Sn was used to record the temperature.

The melting temperature was indicated by a sharp change of the enthalpy or the specific heat.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.
Error of the calorimetric measurement: below ± 1 %.

REFERENCES:

COMPONENTS:

Lead; Pb; [7439-92-1]

Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Dadd, A.T.; Hubberstey, P.; Roberts, P.G.

J. Chem. Soc., Faraday Trans. I 1982, *78*, 2735-2741.

VARIABLES:

Temperature: 513-670 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Solubilities of Pb in liquid Li were determined.

T/K	*soly*/mol % Pb	T/K	*soly*/mol % Pb
513	0.08	624	0.64
549	0.16	635	0.73
575	0.27	633	0.77
590	0.33	640	0.83
598	0.37	648	0.96
606	0.43	651	1.04
613	0.53	666	1.26
617	0.58	670	1.40

The authors presented the equation fitting the measured points, the equation was tested by the compilers.

$$\ln(soly/\text{mol fract. Pb}) = 5.717 - 6722(T/\text{K})^{-1} \qquad \text{standard deviation} = 0.0647$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The liquid alloy was prepared from appropriately weighed amounts of the solute element which was added to liquid Li. The resistivity of the alloy was monitored under equilibrium conditions as a function of temperature for a series of samples of increasing concentration. Discontinuities of the resistivity versus temperature data indicate the transition from the homogeneous liquid to the two-phase region. The apparatus used for the resistivity measurements was the same as in (1).

SOURCE AND PURITY OF MATERIALS:

Pb: 99.999 % pure from Koch Light.
Li: 99.98 % pure from Koch Light; Further purified by means of gettering with Ti sponge at 673 K for 72 hours.
Ar: 99.99 % pure from Air Products.

ESTIMATED ERROR:

Solubility: standard deviation ± 7%.
Temperature: nothing specified.

REFERENCES:

1. Adams, P.F.; Down, M.G.; Hubberstey, P.; Pulham, R.J. *J. Less-Common Met.* 1975, *42*, 325-334.

COMPONENTS:	ORIGINAL MEASUREMENTS:
Lead; Pb; [7439-92-1] Lithium; Li; [7439-93-2]	Watson, W.R.; Pulham, R.J. *Liquid Metals Enigneering and Technology*, Brit.Nucl.Ener.Soc., London, 1985, *3*, 99-102.
VARIABLES:	**PREPARED BY:**
Temperature: 951-1025 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Pb-Li system were determined; the data were read out from the figure by the compilers.

T/K	*soly*/mol % Pb	T/K	*soly*/mol % Pb
988	20.1	1021	22.9
996	20.4	1020	23.1
1008	20.8	1015	23.8
1013	21.1	1006	24.4
1021	21.5	999	24.8
1023	21.8	996	24.9
1025	22.1	998	25.0
1024	22.2	951	27.8
1023	22.4		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were enclosed into sealed capsules of type 316 stainless steel which were equipped with a thermocouple pocket. A slender neck of the capsules allowed pure metals to be added after cutting and once more closing by means of welding. The mixtures were held at constant temperature of 1100 K for 10^5 s. Thermal analysis was performed during the cooling. The Chromel/Alumel thermocouples were calibrated on the melting points of Sn, Pb, Zn, Sb, and Ag.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.999 % pure from Goodfellow Metals.
Li: 99.8 % pure from Koch Light.

ESTIMATED ERROR:

Solubility: nothing specified; read-out procedure ± 0.1 mol %.
Temperature: precision ± 2 K.

REFERENCES:

COMPONENTS: Lead; Pb; [7439-92-1] Lithium; Li; [7439-93-2]	**ORIGINAL MEASUREMENTS:** Jauch, U.; Karcher, V.; Schulz, B. *Kernforschungszentrum Karlsruhe Rep. KfK-4144*, 1986.
VARIABLES: Two temperatures: 508 and 757 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus points of two Pb-Li alloys were determined.

T/K	*soly*/mol % Pb
508[a]	83
757[b]	50
758[c]	50

[a] the temperature was roughly confirmed in measurements of the thermal diffusivity, thermal conductivity and electric resistivity

[b] on heating

[c] on cooling

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloy with 83 mol % Pb was prepared in a stainless steel crucible at 623 K. The equiatomic alloy was melted at 825 K in a crucible of Al_2O_3. The experiments were performed in He atmosphere. Thermocouples which were calibrated on the melting and boiling points of H_2O were applied to record the thermal analyses. The samples were analyzed for the Li content by means of atomic absorption spectroscopy.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.99 % pure from Ventron.
Li: 99.4 % pure from Metallgesellschaft, Frankfurt, with contents of 0.5 % Na, 0.01 % K, 0.03 % Ca, $<1 \cdot 10^{-2}$ % Al, and $<3 \cdot 10^{-2}$ % Si.
He: 99.999 % pure, purified by passing through Ti chips at 895 K with contents of $<1 \cdot 10^{-4}$ % O_2, N_2, and H_2O.

ESTIMATED ERROR:

Solubility: accuracy ± 0.2 mol %.
Temperature: precision ± 0.5 K.

REFERENCES:

COMPONENTS:

Lead; Pb; [7439-92-1]

Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Saar, J. Ruppersberg, H.

J. Phys.,F 1987, *17*, 305-314.

VARIABLES:

Temperature: 535-985 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Pb-Li system were determined.

T/K	*soly*/mol %Pb	T/K	*soly*/mol %Pb
840	10	745	40.1
900	14.85	750	51.15
985	19.85	695	60
975	24.85	535	80
905	30.55		

The melting points of Pb and Li were found at 327 and 181 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus made of stainless steel was placed inside a dry Ar glove box. The alloys were prepared by means of melting the two metals. The density of the alloys was determined as a function of the temperature from the breaking points of curves relating the density of an alloy of constant composition on temperature. The composition of the alloys was checked by a chemical analysis after the density measurements.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.999 % pure.
Li: 99.9 % pure.
Ar: "high purity".

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: stability ± 0.05 K; reproducibility ± 1 K.

REFERENCES:

COMPONENTS:

Lead; Pb; [7439-92-1]

Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Sommer, F.

Z. Phys. Chem., N.F. 1988, *156*, 593-597.

VARIABLES:

Two temperatures: 756 and 999 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The melting points of two alloys were determined.

T/K	*soly*/mol % Pb
756	50.0
999	22.2

A premelting effect on the alloy with 50 mol % Pb was observed which seems to be rather caused by an increasing concentration of lattice defects in the solid phase than by impurities.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The specific heat of the liquid alloys was determined by means of the drop calorimetric heat content measurement. The alloys were heated up to the temperature of measurement in a furnace. The calorimeter was operated at room temperature. The alloys were enclosed by tight Mo ampoules. The calorimeter was calibrated on an ampoule containing Sn as a well defined reference material. The melting temperature corresponded to the jump of the heat content.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

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

REFERENCES:

COMPONENTS:

Lead; Pb; [7439-92-1]

Lithium; Li; [7439-93-2]

ORIGINAL MEASUREMENTS:

Hubberstey, P.; Sample, T.; Barker, M.G.

J.Nucl.Mater. 1992, *191-194*, 283-287.

VARIABLES:

Temperature: 509-600 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Many points on the Pb-rich part of the Pb-Li liquidus were determined.

t/°C	*soly*/mol % Li	*soly*/mol % Pb[a]	*t*/°C	*soly*/mol % Li	*soly*/mol % Pb[a]
324	1.11	98.89	274	10.1	89.9
319	1.35	98.65	270	10.16	89.84
320	1.60	98.40	269	11.06	88.94
320	2.03	97.97	266	11.16	88.84
318	2.24	97.76	260	11.96	88.04
314	2.56	97.44	258	12.34	87.66
315	2.86	97.14	255	12.55	87.45
313	3.01	96.99	253	12.83	87.17
312	3.38	96.62	248	13.15	86.85
303	4.66	95.34	246	13.89	86.11
302	4.97	95.03	246	14.05	95.95
301	5.05	94.95	244	14.23	85.77
295	5.59	94.41	246	14.36	85.64
299	6.0	94.0	238	15.52	84.48
293	6.13	93.87	236	15.60	84,40
295	6.21	93.79	237	15.74	84.26
293	6.70	93.30	241	16.9	83.1
293	7.02	92.98	238	17.04	82.96
288	7.45	92.55	243	17.11	82.89
285	7.96	92.04	239	17.26	82.74
280	8.21	91.79	238	17.28	82.72
286	8.34	91.66	241	17.31	82.69
275	9.26	90.74	245	18.50	81.50
276	9.32	90.68	258	20.5	79.50
278	9.68	90.32	275	22.13	77.13
275	9.68	90.32			

[a] as calculated by the compilers

The melting point of Pb was found at 327 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The resistivity method was applied for the determination of the solubility. The apparatus consisted of a facility to introduce the metals into a vessel made of AISI type 316 stainless steel under inert cover gas, an electromagnetic pump to mix the metals, an air furnace with a circulation fan to maintain constant temperature and a device to measure the resistance. The temperature was measured by means of Chromel/Alumel thermocouples calibrated on the melting points of Pb and the eutectic temperature. The vessel was calibrated for the determination of the resistivity. Small aliquots of Li were added, after each addition the resistance of the vessel was determined as a function of temperature. Data were generally accumulated at 15 K intervals, and at 1 K intervals close to the phase transition (± 5K).

SOURCE AND PURITY OF MATERIALS:

Pb: 99.999% pure from Fluka; surface was mechanically cleaned (1).

Li: 99.8 % pure from Koch Light; surface was mechanically cleaned in an Ar atmosphere (1).

Ar: unspecified.

ESTIMATED ERROR:

Solubility: precision ± 0.2 mol %.

Temperature: stability and precision ± 1 K.

REFERENCES:

1. Hubberstey, P.; Barker, M.G.; Sample, T. *Fusion Engin.Design* 1991, *14*, 227-235.

COMPONENTS:	EVALUATOR:
(1) Lead; Pb; [7439-92-1] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland April 1992

CRITICAL EVALUATION:

The solubility of Pb in liquid Na was determined by Lamprecht et al. (9,10), Hubberstey and Pulham (11) and Hubberstey and Castleman (12). Some additional results on the solubility were obtained in other studies of the Pb-Na liquidus (4,5,14,16). The results of Kurnakov and Kuznetsov (4) deviated from the other measurements by up to 30 K. The data of Tammann (1) and Heycock and Neville (2) near the Na-rich eutectic point were not confirmed in subsequent studies. The most comprehensive study was performed by Hubberstey and Pulham (11), but it did not result in a completely linear plot of the logarithm of the solubility vs. reciprocal temperature. Their results and those from (4,5,12,14) which were determined between the eutectic point (0.09 mol % Pb and 370.5 K) and 600 K may be approximated by the fitting equation found by the compilers:

$$\log(soly/\text{mol \% Pb}) = 4.309 - 1983(T/\text{K})^{-1} \qquad r=0.987 \qquad \text{Eq.(1)}$$

Lamprecht et al. (9,10) proposed another solubility equation on the basis of their own results (see the data sheet), Eq.(1) is, however, preferable, since it was based on a larger number of experimental data taken from different sources. Differences between the results in (9,10) and (11) do not seem to be due to different purity of the materials used, the methods applied in both studies appear also convincing.

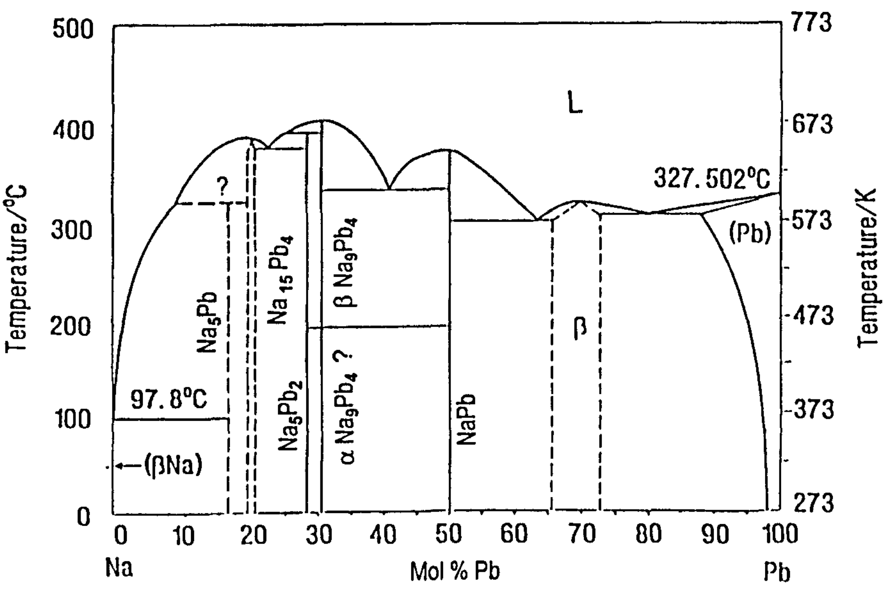
The liquidus line of the alloys richer in Pb was investigated by (4-8,13-17). The results of (4) and (15) showed the largest deviation from the mean line, while the other data scattered within a ± 10 K band. The paper (17) was not compiled since it did not present sufficient experimental details. (18) reported the confirmation of the results of (15) without the presention of quantitative data.

The Pb-Na phase relations are shown in the diagram after (19); the phase Na_5Pb is added according to (10).

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

T/K	$soly$/mol % Pb		source
370.5	0.09	eutectic	Eq.(1), (11)
473	1.3		Eq.(1)
573	7.0		Eq.(1), (5),(11),(12); mean value
659	20 (r)	congruent	(5),(14),(16)
648	24	eutectic	(5),(7),(16); mean value
664	27	peritectic	(16)
677	31 (r)	congruent	(5),(14),(16),(17); mean value

The two liquid metals are completely miscible above 677 K.



COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

EVALUATOR:

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

References

1. Tammann, G. *Z. Phys. Chem.* 1889, *3*, 441.
2. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1889, *55*, 666.
3. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1892, *61*, 888.
4. Kurnakov, N.S.; Kuznetsov, A.N. *Z.Anorg.Chem.* 1900, *23*, 439; *Zh.Russ.Fiz.-Khim.Obshch.* 1899, *31*, 927.
5. Mathewson, C.H. *Z. Anorg. Chem.* 1906, *50*, 171.
6. Goebel, J. *Z. Metallk.* 1922, *14*, 425.
7. Calingaert, G.; Boesch, W.J. *J. Am. Chem. Soc.* 1923, *45*, 1901.
8. Kleiber, H. *Z. Elektrochem.* 1936, *42*, 258.
9. Lamprecht, G.J. *Ph.D. thesis*, Univ. of South Africa, Pretoria, 1966.
10. Lamprecht, G.J.; Dicks, L.; Crowther, P. *J. Phys. Chem.* 1968, *72*, 1439.
11. Hubberstey, P.; Pulham, R.J. *J. Chem. Soc., Dalton Trans.* 1972, 819.
12. Hubberstey, P.; Castleman, A.W. *J. Electrochem. Soc.* 1972, *119*, 963.
13. Saboungi, M.L.; Reijers, H.T.J.; Blander, M.; Johnson, G.K. *J. Chem. Phys.* 1988, *89*, 5869.
14. Ruppersberg, H.; Jost, J. *Thermochim. Acta* 1989, *151*, 187.
15. Kurnakov, N.S.; Pogodin, S.A.; Shpichinetskii, E.S.; Zorin, V.S. *Izv.Sekt.Fiz.Khim. Anal.* 1940, *13*, 233.
16. Krohn, I.T.; Werner, R.C.; Shapiro, H. *J. Am. Chem. Soc.* 1955, *77*, 2110.
17. Aleksandrov, B.N.; Dalakova, N.V. Moskalets, M.V. *Izv.Akad.Nauk SSSR, Met.* 1987, no. 3, 198.
18. Morachevskii, A.G.; Shoikhet, D.N. *Nauch.-Tekhn. Infor.Bull*
19. *.LPI* 1961, no. 10, 67.
    Binary Alloy Phase Diagrams, T.B. ;Massalski, Ed., Am.Soc.Mater., Materials Park, 1990, p. 2722.

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Tammann, G.

Z. Phys. Chem. 1889, *3*, 441-449.

VARIABLES:

Temperature difference: 0.21 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The depression of the melting point of Na due to additions of Pb was determined:

ΔT/K	*soly*/g Pb per 100 g Na	*soly*/mol % Pb [a]
0.05	0.26	0.029
0.16	0.54	0.060
0.21	1.25	0.138

[a] calculated by the compilers

The melting point of Na was reported to be 370 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Successive portions of Pb were added to molten Na. The resulting alloys were undercooled for up to 2 K. The precipitation of crystals was forced by effective moving a thermometer. Every determination of the temperature was three times repeated.

SOURCE AND PURITY OF MATERIALS:

Pb: nothing specified.
Na: "pure".

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.05 K.

REFERENCES:

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Heycock, C.T.; Neville, F.H.

J. Chem. Soc. 1892, *61*, 888-914.

VARIABLES:

Temperature: 370.5-599.5 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the Pb-Na liquidus were determined.

t/°C	*soly*/mol Na per 100 mol Pb	*soly*/mol % Pb [a]
326.35	0.92	99.09
325.16	1.974	98.06
323.88	3.125	96.97
322.46	4.475	95.72
97.39	-	0.0426 [b]

[a] as calculated by the compilers [b] as reported in (1)

The melting points of Pb were determined to be 327.5 and 97.55 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The experiments were performed in a block of cast Fe. The Pb sample was covered with paraffin to prevent oxidation. The Pb-rich alloys were prepared from weighed amounts of metals in a hard glass tube which was heated and shaken. The alloys prepared in this way were added to molten Pb. A Fe stirrer was used to homogenize the mixture. The Na-rich alloy (1) was prepared in a Fe crucible. Carefully calibrated thermometers were used for the exact measurements of the freezing points of alloys.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: precision ± 0.01 K.

REFERENCES:

1. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1889, *55*, 666-676.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lead; Pb; [7439-92-1] (2) Sodium; Na; [7440-23-5]	Kurnakov, N.S.; Kuznetsov, A.N. *Zh. Russ. Fiz.-Khim. Obshch.* 1899, *31*, 927-948.
VARIABLES:	**PREPARED BY:**
Temperature: 368-693 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Pb-Na system were determined.

t/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb
95	0.80	304.5	60.00	314.0	75.00
252	3.80	300	61.36	308.7	77.50
268	5.75	297.5	62.50	307.5	80.00
339	10.56	285	62.81	309.0	82.50
389	17.07	305.5	65.00	310.5	85.00
396	22.69	327	67.27	312.0	87.50
420	29.01	311.5	67.50	314.0	90.00
391	38.15	314.5	70.00	315.5	92.50
382	46.47	316.0	72.50	318.5	95.00

The melting point of Pb was found at 326.0 °C. The results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in a glass tube under H_2 atmosphere. The alloys were chemically analyzed for their Pb content. The freezing points were determined by means of a pyrometer, which was calibrated on the boiling point of H_2O and the melting points of Sn, Pb, Cd, Sb, Zn, and Ag, or a mercury thermometer.

SOURCE AND PURITY OF MATERIALS:

Pb: nothing specified.
Na: from Kahlbaum.

ESTIMATED ERROR:

Solubility: precision not better than ± 0.1 mol % (by the compilers).
Temperature: precision ± 1.0 K.

REFERENCES:

1. Kurnakov, N.S.; Kuznetsov, A.N. *Z. Anorg. Chem.* 1900, *23*, 439-462;

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Mathewson, C.H.

Z. Anorg. Chem. 1906, *50*, 171-198.

VARIABLES:

Temperature: 566-678 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Pb-Na system were determined.

t/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb
293	6.6	403	29.0	343	58.6
355	13.8	404	31.5	317	61.2
385	19.4	405	32.9 congruent	301	62.7
386	20.6	404	33.8	308	66.1
384	22.8	386	36.8	310	67.0
379	23.7	351	40.3	318	69.9
373	24.2 eutectic	329	41.5 eutectic	319	72.6 congruent
378	24.6	343	44.3	318	75.0
392	26.1	362	47.4	317	76.6
394	26.8	367	50.6	307	78.9
399	28.0	359	54.5	312	85.6

The melting points of Na and Pb were found at 97.5 and 326.9 °C, respectively.

$PbNa_4$, $PbNa_2$, PbNa, and Pb_5Na_2 were identified as equilibrium solid phases.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was melted under Vaseline oil, cooled to freezing, and its surface was cut off. The metals were placed in a high melting Jena glass tube under H_2 atmosphere. The molten alloys were mixed by means of stirring with a Fe wire. Thermal analytical arrests were taken from cooling curves. A Pt/Pt-Rh thermocouple was used for temperature measurements. It was calibrated on the melting points of Na, Pb, Sn, Zn, and Sb as in (1). Some alloys were analyzed by decomposition with H_2O. The formed NaOH was acidimetricly titrated.

SOURCE AND PURITY OF MATERIALS:

Pb: "pure".
Na: "pure"; purified by remelting and cutting off the surface (1).

ESTIMATED ERROR:

Nothing specified.
Solubility: precision ± 0.5 mol % in analogy to (1).
Temperature: precision ± 1 K (by the compilers).

REFERENCES:

1. Mathewson, C.H. *Z. Anorg. Chem.* 1905, *46*, 94-112.

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Goebel, J.

Z. Metallk. 1922, *14*, 425-432.

VARIABLES:

Temperature: 581-639 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the liquidus line of the Pb-Na system were determined.

t/°C	*soly*/mass % Na	*soly*/mol % Pb[a]
324	0.80	93.2
323	0.98	91.8
323	1.08	91.1
308	2.71	79.9 eutectic
320	3.81	73.7
320	4.22	71.6
301	6.25	62.5 eutectic
312	6.41	61.8
366	9.80	51.5

[a] as calculated by the compilers

The melting point of Pb was found at 327 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A weighed amount of Pb was melted in a Fe crucible. Na pieces were introduced into the melt. Each alloy was chemically analyzed and placed in a closed graphite crucible in a H_2 atmosphere. Thermal analytical cooling curves were recorded by means of a Cu/Konstantan thermocouple which was previously calibrated on the boiling point of H_2O and the melting points of Sn, Pb, and Zn.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.
Solubility: precision not better than ± 0.1 mol % (by the compilers).
Temperature: precision not better than ± 2 K (by the compilers).

REFERENCES:

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Calingaert, G.; Boesch, W.J.

J. Am. Chem. Soc. 1923, *45*, 1901-1904.

VARIABLES:

Temperature: 597-673 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The Na-rich side of the liquidus line of the Pb-Na system was partially determined.

t/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb
365	48.7	388	35.1	398	28.6
358	45.9	396	33.1	396	27.7
324	40.6	397	33.0	395	27.1
366	38.5	395	31.0	391	26.8
377	35.8	396	30.4	372	24.3
382	35.5	400	29.4		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were contained in a Ni crucible and stirred with a Fe agitator. They were heated in a well-lagged electrical furnace, while a slow flow of H_2 passed through the apparatus. Cooling curves were measured by means of a calibrated Chromel/Alumel thermocouple which was protected in a Pyrex glass tube in order to avoid reactions with the alloys. The compositions of the alloys were determined by means of chemical analyses. The alloys were decomposed with H_2O and HNO_3, and Na was gravimetrically determined after converting into sulfate and separation from Pb.

SOURCE AND PURITY OF MATERIALS:

Pb: "pure" as indicated through the determination of the melting point and chemical analysis.
Na: melted and cut under paraffin oil.

ESTIMATED ERROR:

Solubility: accuracy ± 0.5 mol %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS: (1) Lead; Pb; [7439-92-1] (2) Sodium; Na; [7440-23-5]	**ORIGINAL MEASUREMENTS:** Kleiber, H. *Z. Elektrochem.* 1936, *42*, 258-264.
VARIABLES: Two temperatures: 571 and 578 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Two eutectic points in the Pb-rich side of the phase diagram were determined.

t/°C	*soly*/mol % Pb
305	80
298	64.3

It was also reported that Siegert (1) confirmed the liquidus line of Mathewson (2) with improvements reported by Calingaert and Boesch (3) by means of thermal analysis in the same laboratory. Numerical date were, however, not quoted.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared from the elements in an Ar atmosphere. They were homogenized for 5 days at 543 K and equilibrated for 8 days in evacuated glass tubes at selected temperatures. The electric resistance of the alloys was measured by means of a compensation method.

SOURCE AND PURITY OF MATERIALS:

Pb: analytically pure from Kahlbaum.
Na: analytically pure from Merck.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Siegert, H. *Ph.D. thesis*, Technische Hochschule, Stuttgart, Germany, 1935.
2. Mathewson, C.H. *Z. Anorg. Chem.* 1906, *50*, 171-198.
3. Calingaert, G.; Boesch, W.J. *J. Am. Chem. Soc.* 1923, *45*, 1901-1904.

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Kurnakov, N.S.; Pogodin, S.A.; Shpichinetskii, E.S.; Zorin, V.S.
Izv. Sekt. Fiz.-Khim. Anal. 1940, *13*, 233-250.

VARIABLES:

Temperature: 576-643 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

A part of the liquidus of the Pb-Na system was determined.

t/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb
370	50.0	320	67.0	315[a]	75.3
369	51.4	321	70.0	309[a]	78.3
367	52.9	320	73.0	303[a]	81.7
358	56.1	316	75.0	309[a]	85.4
333	57.0	313	77.5	312[a]	87.9
307	60.0	314.5	80.0	317[a]	91.9
309	62.0	319.5	90.0	320[a]	95.4
316	65.0	321	95.0		

[a] published in (1), obtained by means of thermal analysis by Ageev and Shoikhet

The melting point of Pb was found to be 327 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Pieces of Na were introduced inside a special Fe tube into molten Pb which was contained in a Fe crucible under paraffin. The ligature which was obtained in this way comprised 12 mass % Na; its composition was confirmed by chemical analysis. Corresponding amounts of Pb were added to the ligature in a container made of Pyrex glass. The container was evacuated and heated. Cooling curves were recorded by means of a Ni-Cr/Constantan thermocouple calibrated on the melting points of Zn, Pb and Sn.

SOURCE AND PURITY OF MATERIALS:

Pb: chemically pure, made in USSR, with the same melting point and hardness as one from Kahlbaum.
Na: chemically pure, made in USSR.

ESTIMATED ERROR:

Solubility: accuracy ± 0.05 %.
Temperature: nothing specified.

REFERENCES:

1. Ageev, N.V.; Talyzin, N.Ya. *Izv. Sekt. Fiz.-Khim. Anal.* 1940, *13*, 251-255.

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Krohn, I.T.; Werner, R.C.; Shapiro, H.

J. Am. Chem. Soc. 1955, *77*, 2110-2113.

VARIABLES:

Temperature: 609-677 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the Na rich part of the liquidus line of the Pb-Na system were determined.

t/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb
386.2	19.81	402.1	28.88	400.0	33.23
386.0	21.25	402.0	29.24	400.0	33.89
377.4	23.16	402.8	29.49	361.0	40.61
382.0	24.50	402.6	30.00	336.0	41.81
394.5	26.00	402.0	30.08	357.0	44.58
393.2	26.44	403.6	30.59 congruent	367.5	47.66
399.8	27.42	403.5	30.61	371.0	48.99
397.5	27.57	403.4	31.10	371.5	49.64
399.5	28.51	402.0	31.33	370.0	51.09
402.5	28.65	402	32.11		

The equilibrium compound Pb_4Na_9 was identified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were melted in a Fe crucible under N_2 or Ar atmosphere. Cooling curves of the resulting alloys were recorded by means of a Fe/Constantan thermocouple. During this procedure the molten alloys were agitated with a Fe ring which was immersed into the melt and connected to a vibrator. The solid alloys were broken up at room temperature under N_2 atmosphere and chemically analyzed for the Na content.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.98 % pure from National Lead Co.
Na: 99.7$^+$ % pure.

ESTIMATED ERROR:

Solubility: accuracy of the analysis between ± 0.06 and ± 0.16 mol % Pb.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Lamprecht, G.J.; Dicks, L.; Crowther, P.

J. Phys. Chem. 1968, *72*, 1439-1441.

VARIABLES:

Temperature: 392.1-516.7 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Pb in liquid Na was determined at several temperatures.

t/°C	*soly*/mass % Pb	*soly*/mol % Pb
118.9	0.58	0.065
143.2	1.36	0.153
165.2	2.77	0.32
197.0	7.25	0.86
205.8	8.19	0.98
227.1	13.66	1.73
243.5	18.40	2.44

The same results were reported in (1).

The solubility equation was derived by the authors and confirmed by the compilers.

$$\log(soly/\text{mol \% Pb}) = 5.515 - 2639\ (T/\text{K})^{-1}$$

Two equilibrium phases precipitated out of the solutions. Their compositions were determined to be $\mathrm{PbNa}_{5\ 05\pm0.09}$ and $\mathrm{PbNa}_{4\ 02\pm0.06}$ melting at 391 and 374 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus for the measurements of the solubility was made of Pyrex glass, He was used as inert cover gas. After equilibrium had been reached the solution in Na was drawn off and analyzed for Pb and Na. Pb was determined as the $PbCrO_4$, and Na by means of direct acid-base titration. The equilibrium solid phases were analyzed in the same way.

SOURCE AND PURITY OF MATERIALS:

Pb: spectrally pure from Johnson Matthey.
Na: from Merck, filtered at 383 °C with contents of 0.002 % Cl, SO_4, heavy metals, 0.001 % Fe, PO_4, 0.005 % N, Ca, 0.01 % K, and $(1.1\pm0.2)\cdot10^{-3}$ % O.
He: purified with molecular sieves and activated charcoal trapping at the temperature of liquid N; any contents of O_2, H_2O, CO_2, N_2, and CH_4 were not detectable.

ESTIMATED ERROR:

Solubility: standard deviation ± 3 %.
Temperature: stability better than ± 1K.

REFERENCES:

1. Lamprecht, G.J. *Ph.D. thesis*, Univ. of South Africa, Pretoria, 1966.

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Hubberstey, P.; Pulham, R.J.

J. Chem. Soc., Dalton Trans. 1972, 819-821.

VARIABLES:

Temperature: 370.5-573 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Pb in liquid Na was determined at various temperatures. The data were read from the figure by the compilers.

t/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb
98.4	0.109	151.0	0.34	232	2.56
104.0	0.12	159.3	0.43	245	3.09
105.6	0.13	168.8	0.55	262	4.02
110.3	0.14	181.4	0.69	268	4.77
112.2	0.15	189.3	0.82	286.5	5.91
125.7	0.22	202.5	1.16	288	6.49
136.7	0.26	223	1.89	295	7.09
				300	7.83

The depression of the freezing points due to additions of Pb to liquid Na was measured.

t/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb
97.75	0.007	97.60	0.045
97.73	0.013	97.55	0.056
97.74	0.016	97.52	0.068
97.74	0.024	97.35	0.078
97.64	0.031	97.43	0.091

The melting point of Na was determined at 97.83 °C.
The eutectic point was established at 0.1 mol % Pb and 97.32 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The determinations were performed by means of thermal analysis and the measurement of the electrical resistivity. A cell for the resistivity measurement was baked out under vacuum at 573 K. The cell was filled with Na under Ar atmosphere. The resistance was measured, while the temperature was decreased in steps. The cell was then charged with Pb and the procedure was repeated, after the equilibrium was reached at the given temperature. Details of the thermal analysis were not reported.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.999 % pure from Koch-Light.
Na: "purified".

ESTIMATED ERROR:

Solubility: nothing specified; read-out procedure between ± 0.002 mol % and ± 0.02 mol %.
Temperature: stability ± 0.5 K; read-out procedure between ± 0.02 K and ± 0.2 K.

REFERENCES:

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Hubberstey, P.; Castleman, A.W.

J. Electrochem. Soc. 1972, *119*, 963-966.

VARIABLES:

Temperature: 587-625 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Pb in liquid Na was determined.

T/K	t/°C[a]	soly/mol % Pb
587	314	7.79
598	325	10.01
609	336	12.65
625	352	14.61

[a] calculated by the compilers

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The electrochemical cell Na/Na$^+$(in Pyrex glass)/Na-Pb was operated under He atmosphere. The apparatus was made of Pyrex glass, while stainless steel was applied for electrical connections. The apparatus was evacuated and baked at ~773 K for several hours. The weighed metals were introduced into a dry atmosphere box. The cell was evacuated, sealed and heated over night to ~25 K above the estimated liquidus temperature. The temperature was then decreased in steps of 5 K.

The Na activity became constant in the two-phase region, thus indicating the liquidus temperature.

Chromel/Alumel thermocouples were used to measure the temperature.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.9 % pure from Allied Chem. Corporation.

Na: 99.9 % pure from J.T. Baker Comp., pipetted at ≈393 K in order to reduce concentrations of non-metals to <1 10^{-3} %.

He: with contents of <1·10^{-4} % O and <1·10^{-5} % H_2O.

ESTIMATED ERROR:

Solubility: nothing specified.

Temperature: stability ± 0.5 K.

REFERENCES:

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Saboungi, M.L.; Reijers, H.T.J.; Blander, M.; Johnson, G.K.

J. Chem. Phys. 1988, *89*, 5869-5875.

VARIABLES:

One temperature: 642 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The melting temperature of the equimolar alloy PbNa was found at 642 K or 369 °C (as calculated by the compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were weighed and loaded into a heavy-walled Ta crucible inside a He filled glove box. The filling tube which was welded to one end of the capsule was then crimped and sealed by arc melting in the glove box. Drop calorimetry on the metals was performed in a system consisting of a Mo-core furnace and a Cu block calorimeter.

The enthalpy relative to 298.15 K of the empty Ta capsule was determined over the same range of experimental parameters.

The liquidus temperature corresponds to the enthalpy (H_T-H_{298}) jump on the corresponding plot.

SOURCE AND PURITY OF MATERIALS:

Pb: filtered as molten.

Na: "high purity".

He: with contents of < 1·10^{-4} % H_2O and O_2 (each).

ESTIMATED ERROR:

Solubility: accuracy ± 0.05 mol %.

Temperature: precision ± 2 K.

REFERENCES:

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Ruppersberg, H.; Jost, J.

Thermochim. Acta 1989, *151*, 187-195.

VARIABLES:

Temperature: 550-677 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Pb-Na system were determined.

T/K	*t*/°C[a]	*soly*/mol % Pb
550	277	5
600	327	10
659	386	20
677	404	31
665	392	35
606	333	41
645	372	50
573	300	62
593	320	72
580	307	80

[a] as calculated by the compilers

The melting points of Pb and Na were found at 327 and 96.3 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were melted and studied in a vessel made of low C steel. The vessel was equipped with a sealed-in thermocouple in its centre. The whole system was operated in a dry Ar glove box. The densities of molten alloy samples were measured using the maximum bubble pressure apparatus. The liquidus temperature was determined on the basis of the break point in the density vs. temperature diagram.

SOURCE AND PURITY OF MATERIALS:

Pb: nothing specified.
Na: nothing specified.
Ar: "purified".

ESTIMATED ERROR:

Solubility: reproducibility ± 1.5 %, standard deviation ± 0.8 %.

Temperature: reproducibility ± 1 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Lead; Pb; [7439-92-1] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1991

CRITICAL EVALUATION:

Smith (1) was confronted with experimental difficulties in his thermo-analytical study of the K-Pb system. He postulated the existence of a liquid miscibility gap between 26 and 66 mol % Pb at about 841 K. The range of immiscibility was confirmed in later work of other laboratories. The liquidus data of (1) were only confirmed for compositions with more than 72 mol % Pb. Shoikhet et al.(2) showed by means of thermal analyses that the miscibility gap does not exist, however, a compound KPb occurs, melting congruently at ~848 K. Other, peritectically-formed equilibrium phases were reported as previously (1), but a new phase, K_2Pb_3, was discovered. The existence of the congruently melting KPb was also deduced from potentiometric measurements of Lantratov (3); however, he did not report numerical results. Morachevskii and Alabyshev (4) measured the melting point of KPb at 851 K; they did not give any details of the used method.

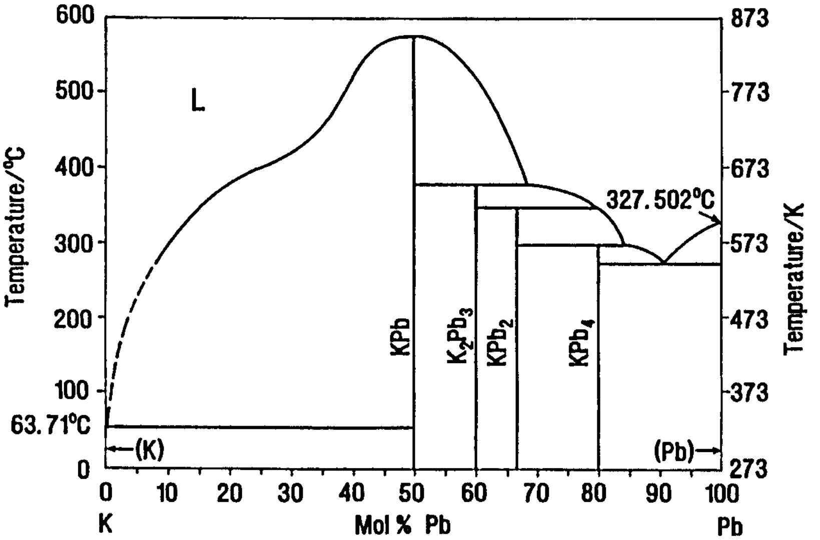
Meijer et al. (5,6) studied again the whole range of composition of K-Pb alloys by means of differential scanning calorimetry. They found the liquidus line about 5 - 10 K higher than that of (2). Johnson and Saboungi (7) determined the melting point of KPb at 862 K in calorimetric experiments and claimed this temperature to be in excellent agreement with unpublished data which were gained in their own potentiometric measurements. The recorded temperature seems to be overestimated by about 10 K. Saar and Ruppersberg (8) determined the liquidus line of the K-Pb system from density measurements. Their data are in good agreement with the results of (2) and (5,6), particularly on the Pb-rich side. Aleksandrov (9) reported the melting point of KPb at 853 K, the experimental method was not described.

The solubility of small amounts of Pb in liquid K at temperatures below 570 K has never been investigated. A schematic K-Pb phase diagram is shown in the figure.

Tentative values of the solubility of Pb in liquid K.

T/K	*soly*/mol % Pb	source
573	10	(5,6)
673	25	(2) and (5,6); interpolated
773	39	(2), (5,6) and (8); interpolated
849	50	(2), (4), (5,6), (8) and (9); mean value

The two liquid metals are completely miscible at a temperature above 849 K.



References

1. Smith, D.P. *Z. Anorg. Chem.* 1908, *56*, 109.
2. Shoikhet, D.N.; Morachevskii, A.G.; Alabyshev, A.F. *Zh. Neorg. Khim.* 1959, *4*, 1616.
3. Lantratov, M.F. *Zh. Fiz. Khim.* 1960, *34*, 782.
4. Morachevskii, A.G.; Alabyshev, A.F. *Fizicheskaya Khimiya Rasplavlennykh Solei*, Naukovaya Dumka, Kiev, 1965, p. 231.
5. Meijer, J.A.; Geertsma, W.; Van der Lugt, W. *J. Phys., F* 1985, *15*, 899.
6. Meijer, J.A. *Ph.D. thesis*, Groningen Univ., Netherlands, 1988.
7. Johnson, G.K.; Saboungi, M.L. *J. Chem. Phys.* 1987, *86*, 6376.
8. Saar, J.; Ruppersberg, H. *Z. Phys. Chem.N.F.* 1988, *156*, 587.
9. Aleksandrov, B.N.; Dalakova, N.V.; Moskalets, M.V. *Izv. Akad. Nauk SSSR, Met.* 1987, no. 3, 198.

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Smith, D.P.

Z. Anorg. Chem. 1908, *56*, 109-142.

VARIABLES:

Temperature: 553-842 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the K-Pb system were determined.

t/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb
568	30.0	354	75.0	294	85.0
569	37.0	340	76.0	296	86.0
565	40.0	330	78.0	287	88.0
567	45.0	316	80.0	285	89.0
569	50.0	322	81.0	280	90.7
556	53.0	312	82.0	284	91.5
366	72.0	313	83.0	316	95.0
360	74.0	307	83.4		

The melting points of Pb and K were found to be at 327 and 62 °C, respectively.
Three solid phases were identified: KPb_4, KPb_2, and K_2Pb.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

K was purified by cutting off the oxidized surface. It was washed in benzene, amyl alcohol, petroleum, and ether. The two metals were weighed (K under petroleum) and placed in a tube of hard Jena glass for Pb-rich alloys or of Fe for K-rich alloys. The tube was filled with H_2, and the metals were melted in this atmosphere by heating. The tubes were placed in an apparatus for thermal analysis. Cooling curves were recorded by means of a calibrated Pt/Pt-Rh thermocouple.

SOURCE AND PURITY OF MATERIALS:

Pb: nothing specified.
K: did not contain any traces of Na or any other metals.

ESTIMATED ERROR:

Nothing specified.
Temperature: not better than ± 3 K (by the compilers).

REFERENCES:

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Shoikhet, D.N.; Morachevskii, A.G.; Alabyshev, A.F.

Zh. Neorg. Khim. 1959, *4*, 1616-1619.

VARIABLES:

Temperature: 555-853 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Pb-K system were determined.

t/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb
310	96.8	535	58.0	540	40.2
293	94.4	550	55.5	513	39.5
282	89.8	545	55.0	485	37.4
287	88.2	562	54.8	485	37.0
313	82.3	562	53.3	450	33.5
337	78.6	570	51.5	410	27.5
362	74.7	578	51.5	405	26.1
378 [a]	71.5	580	49.8	385	24.0
440	65.8	570	49.7	360	20.0
486	63.0	562	46.0	340	17.0
518	59.8	550	43.7	356	16.6
528	59.5	560	43.5	315	12.5

[a] double result

The solid phases which were identified in the system were Pb_4K, Pb_2K, Pb_3K_2, and PbK.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in an Armco Fe crucible under Ar atmosphere. The filled crucible was placed in a quartz tube which was sealed and heated in a furnace. Thermal analyses were performed in which the cooling curves were recorded by means of a Chromel/Alumel thermocouple. This was calibrated on the melting points of Pb, Sn, Zn, and Sb. The alloys were vigourously mixed. The composition of the samples was determined by chemical analyses.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.9 % pure.
K: distilled, with a content of 0.09 % Na.
Ar: contained 0.35 % O_2.

ESTIMATED ERROR:

Nothing specified.
Solubility: precision ± 0.5 mol % (by the compilers).
Temperature: precision ± 3 K (by the compilers).

REFERENCES:

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Morachevskii, A.G.; Shoikhet, D.H.

Nauch.-Tekhn. Infor. Bull. LPI 1961, no. 10, 67-71.

VARIABLES:

Temperature: 545-851 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the Pb-K system were determined.

t/°C	*soly*/mol % Pb	t/°C	*soly*/mol % Pb
578	50.0	308	81.8
455	66.7	310	81.9
372	70.7	282	85.5
362	74.6	272	90.2
352	75		

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Molten K was introduced through a capillary into a glass tube which was subsequently sealed. This tube was placed in a low C Fe crucible in an Ar atmosphere and broken over Pb. The metals were melted and mixed in this crucible which was enclosed in a quartz container. Thermal analyses were performed, the temperatures were measured by means of a Chromel/Alumel thermocouple.

SOURCE AND PURITY OF MATERIALS:

Pb: specified as "SO", fabricated in USSR.
K: with a content of 0.6 % Na, purified by decantation of the molten metal.
Ar: "purified".

ESTIMATED ERROR:

Nothing specified

REFERENCES:

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Meijer, J.A.; Geertsma, W.; Van der Lugt, W.

J. Phys..F 1985, *15*, 899-910.

VARIABLES:

Temperature: 556-848 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Pb-K system were determined. They were taken from the figure and digitized by the compilers.

t/°C	*soly*/mol % Pb	*t*/°C	*soly*/mol % Pb
283	8.4	549	55.3
300	10.0	524	60.3
393	23.1	471	64.7
420	29.9	408	67.6
479	37.5	347	80.2
573	47.2	315	84.2
575	50.0		

The results were also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The surfaces of the melts of both metals were skimmed to remove the adhering oxides. The preparation of the alloys and the measurements were performed in a dry He glove box. The liquid alloys were contained in AISI 316 stainless steel crucibles. The liquidus temperatures were determined from differential scanning calorimetry experiments.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.999 % pure from Ventron.
K: 99.99 % pure from Kawecki Berylco Industries.
He: contained $< 1 \cdot 10^{-4}$ % O_2 and H_2O.

ESTIMATED ERROR:

Solubility: nothing specified; read-out procedure ± 0.3 mol %.
Temperature: precision ± 2 K.

REFERENCES:

1. Meijer, J.A. *Ph.D. thesis*, Groningen Univ., Netherlands, 1988.

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Johnson, G.K.; Saboungi, M.L.

J. Chem. Phys. 1987, *86*, 6376-6380.

VARIABLES:

One temperature: 862 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The melting point of the equimolar PbK alloy was determined to be 862 K or 589 °C (as calculated by the compilers). This result is claimed to be in excellent agreement with a result from unpublished emf measurements which were performed in the same laboratory.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were loaded into a thick-walled Ta capsule. Its filling tube was then crushed, cut and sealed by means of arc welding in a dry He glove box. The drop calorimetric system consisted of a resistance heated Mo core furnace and a Cu block calorimeter. The temperature of the furnace was measured by a Pt/Pt-Rh(10%) thermocouple. The calorimeter was calibrated, and enthalpies at 298 K and selected temperatures were measured. The liquidus point was identified by means of a jump of (H_T-H_{298}) at the solid-liquid transition.

SOURCE AND PURITY OF MATERIALS:

Pb: "high purity", filtered after melting.
K: "high purity".
He: contained less than $1\cdot10^{-4}$ % O_2 and H_2O.

ESTIMATED ERROR:

Solubility: deviation from stoichiometry 0.6 %.
Temperature: precision ± 2 K.

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Saar, J.; Ruppersberg, H.

Z. Phys. Chem.,N.F. 1988, *156*, 587-591.

VARIABLES:

Temperature: 550-843 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the Pb-K system were determined.

T/K	*t*/°C[a]	*soly*/mol % Pb	*T*/K	*t*/°C[a]	*soly*/mol % Pb
580	303	10.95	800	527	61.15
650	377	20.4	690	417	70
705	432	31.1	600	327	81.15
795	522	40.35	550	277	90.25
843	570	50.15			

[a] calculated by the compilers

The melting points of Pb and K were found at 600 and 336 K, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus was made of stainless steel (1). The samples were prepared in a dry Ar glove box in which the metals were melted. The densities of the samples were measured as a function of the temperature.

The liquidus points were indicated by a break of the curves of the density versus temperature at a constant composition.

The compositions of the alloys were checked by means of chemical analyses after the measurements.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.999 % pure (1).
K: "high purity".
Ar: "high purity".

ESTIMATED ERROR:

Solubility: nothing specified.
Temperature: stability ± 0.05 K; reproducibility ± 1 K, as in (1).

REFERENCES:

1. Saar, J.; Ruppersberg, H. *J. Phys.,F* 1987, *17*, 305-314.

COMPONENTS: (1) Lead; Pb; [7439-92-1] (2) Rubidium; Rb; [7440-17-7]	**EVALUATOR:** H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1991

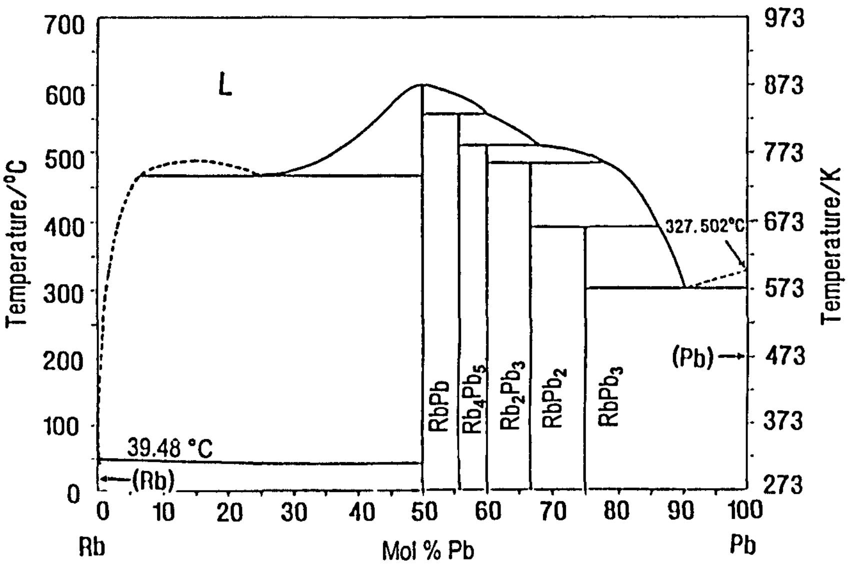
CRITICAL EVALUATION:

Kuznetsov et al. (1) performed thermal analyses of the Rb-Pb system over the whole composition range. The liquidus line which they deduced from their measurements on the Pb-rich side reflects the formation of an eutectic, four peritectic inflections of compounds and one congruently melting RbPb. The thermal arrests on the Rb-rich side were somewhat scattered. The authors proposed therefore the formation of a miscibility gap at 738 K extending from about 5 to 25 mol % Pb, and a critical point at 761 K and 15 mol % Pb. This proposed miscibility gap needs further investigation since neither K-Pb nor Cs-Pb exhibit miscibility gaps; only small changes of the temperature with the composition occur in the corresponding ranges.
Saboungi et al. (2) found the melting point of RbPb to be about 9 K lower than (1) by means of calorimetric measurements. This difference is larger than the sum of the experimental errors of both studies.
A schematic Rb-Pb phase diagram is presented in the figure.

Tentative values of the solubility of Pb in liquid Rb

T/K	*soly*/mol % Pb	source
773	37	(1); interpolation
873	50 congruent	(1)

Above 874 K the two liquid metals are completely miscible.



References

1. Kuznetsov, A.N.; Chuntonov, K.A.; Yatsenko, S.P. *Izv. Akad. Nauk SSSR, Met.* 1977, no. 5, 223.
2. Saboungi, M.L.; Reijers, H.T.J.; Blander, M.; Johnson, G.K. *J. Chem Phys.* 1988, *89*, 5869.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lead; Pb; [7439-92-1] (2) Rubidium; Rb; [7440-17-7]	Kuznetsov, A.N.; Chuntonov, K.A.; Yatsenko, S.P. *Izv. Akad. Nauk SSSR, Met.* 1977, no. 5, 223-225.
VARIABLES:	**PREPARED BY:**
Temperature: 573-874 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Pb-Rb system were determined. The data were read-out from the figure by the compilers.

t/°C	*soly*/mol % Pb	t/°C	*soly*/mol % Pb	t/°C	*soly*/mol % Pb
488	14.5	590	44.0	542	61.5
466	24.5	601	49.0	527	64.0
479	29.0	593	51.5	510	67.0
489	34.5	583	54.0	484	80.0
527	40.5	565	57.0	402	84.5
581	43.5	549	59.5	391	85.0
				300	89.5

Five equilibrium solid phases were determined: Pb_3Rb, Pb_2Rb, Pb_3Rb_2, Pb_6Rb_4, PbRb; the composition of the first and the last phases are well proved. A miscibility gap was predicted to be between 5 and 25 mol % Pb at 465 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The Rb-rich alloys were prepared and studied in a hermetically closed Ta crucible, and the Pb-rich alloys in sealed Pyrex glass ampoules. The filling with Rb was performed under petroleum which was subsequently washed out with ether. The residue of the ether was removed through evaporation under vacuum at room temperature. The heating and cooling curves for thermal analysis were triple recorded for each of the samples, a Chromel/Alumel thermocouple was used for temperature measurements. The Rb content of the samples was analytically determined. The crucible was opened under anhydrous acetone to which H2O was gradually added. The Rb content of the resulting solution was titrated with acid (1).

SOURCE AND PURITY OF MATERIALS:

Pb: 99.999 % pure.
Rb: 99.9 % pure.

ESTIMATED ERROR:

Solubility: accuracy of composition ± 1.5 %; read-out procedure ± 0.5 mol %.
Temperature: precision better than ± 3 K.

REFERENCES:

1. Yatsenko, S.P.; Chuntonov. K.A., Alyamovskii, S.I. *Izv. Akad. Nauk SSSR, Met.* 1973, no. 3, 233-235.

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Saboungi, M.L.; Reijers, H.T.J.; Blander, M.; Johnson, G.K.
J. Chem. Phys. 1988, *89*, 5869-5875.

VARIABLES:

One temperature: 865 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The melting point of the equimolar RbPb alloy was found to be at 865 K or 592 °C (as calculated by the compilers).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were loaded into a thick-walled Ta capsule. Its filling tube was then crushed, cut and sealed by means of arc welding in a dry He glove box. The drop calorimetric system consisted of a resistance heated Mo core furnace and a Cu block calorimeter. The temperature of the furnace was measured by a Pt/Pt-Rh thermocouple. The calorimeter was calibrated, and enthalpies at 298 K and selected temperatures were measured. The liquidus point was identified by means of a jump of (H_T-H_{298}) at the solid-liquid transition.

SOURCE AND PURITY OF MATERIALS:

Pb: "high purity", filtered after melting.
Rb: "high purity".
He: contained less than $1 \cdot 10^{-4}$ % O_2 and H_2O (each).

ESTIMATED ERROR:

Solubility: deviation from stoichiometry ≤ 0.1 %.
Temperature: precision ± 1 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Lead; Pb; [7439-92-1] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland July 1992

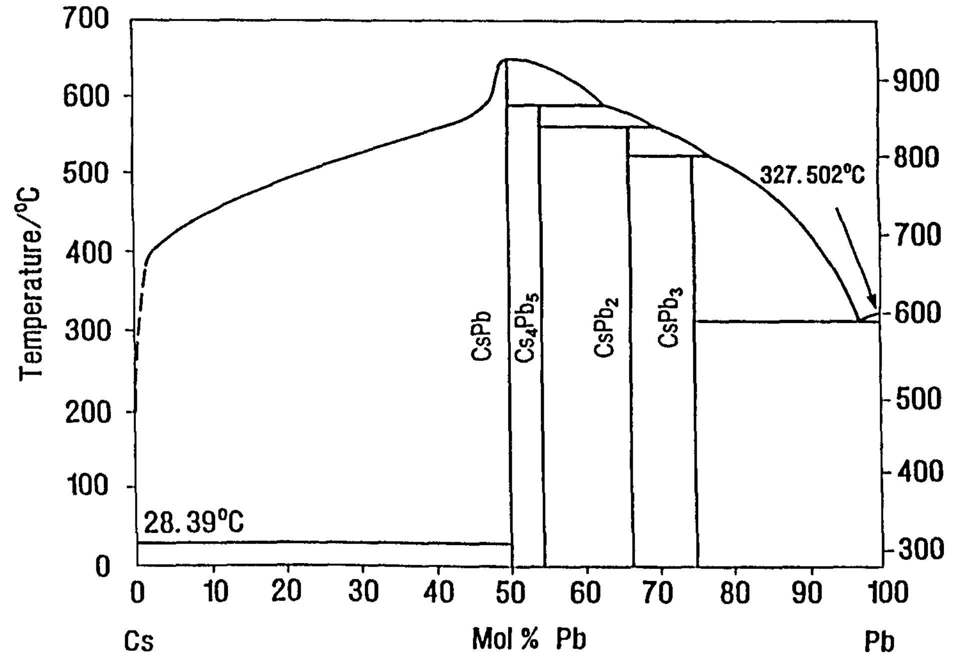
CRITICAL EVALUATION:

The liquidus of the Cs-Pb system was investigated in three laboratories. Meijer et al. (1,2) and Zusman (3) used thermal analysis and differential scanning calorimetry, respectively, in the whole range of alloy compositions. Saboungi et al. (4,5) performed drop calorimetry experiments for the equimolar Cs-Pb alloy and identified its phase transitions at a temperature roughly corresponding to the melting of CsPb and the peritectic decomposition of Cs_4Pb_5 as detected earlier by (3). The sample used by (4,5) probably contained the two phases. The melting point of CsPb, reported by (1,2), was about 30 K lower than in (3) and (4). The shape of the liquidus line as determined by (1,2) in the ranges $<$ 25 and $>$ 75 mol % Pb seems to be more convincing than the line given in (3). The eutectics are placed too far from the pure components in this latter publication, and the solubility of CsPb in liquid Cs is too high compared to the K-Pb and Rb-Pb systems. The purity of the elements was not reported in (3), while the description of (1,2) is hardly sufficient. Therefore, only doubtful solubilities of Pb in liquid Cs can be presented.
The most rational data of all cited studies are combined to propose a schematic phase diagram.

Doubtful values of the solubility of Pb in liquid Cs

T/K	*soly*/mol % Pb	source
673	2	(1,2); extrapolated
773	25	(1,2), (3); interpolated
873	47	(3); interpolated
923	50 congruent	(3), (4,5); mean value of temperatures

The two liquid metals are completely miscible at $\geq$ 923 K.



References

1. Meijer, J.A.; Vinke, G.J.B..; Van der Lugt, W. *J. Phys..F* 1986, *16*, 845.
2. Meijer, J.A. *Ph.D. thesis*, Groningen Univ., Netherlands, 1988.
3. Zusman, L.L. *Fiziko-Mekhanicheskie i Teplofizicheskie Svoisvta Metallov*, Nauka, Moskva, 1976, p. 103.
4. Saboungi, M.L.; Reijers, H.T.J.; Blander, M.; Johnson, G.K. *J. Chem.Phys.* 1988, *89*, 5869.
5. Saboungi, M.L.; Price, D.L. *J. Non-Crystalline Solids*, 1992, *150*, 260.

COMPONENTS:

(1) Lead; Pb; [7439-92-1]

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

ORIGINAL MEASUREMENTS:

Zusman, L.L.

Fiziko-Mekhanicheskie i Teplofizicheskie Svoistva Metallov, Nauka, Moskva, 1976, p. 103-105.

VARIABLES:

Temperature: 586-926 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Pb-Cs system was determined; the points were read out from the figure by the compilers.

t/°C	*soly*/mol % Pb	t/°C	*soly*/mol % Pb
376	15	606	60.5
442	20	577	68.5
480	25	556	71.5
518	30	529	76
535	35	476	81
550	40	410	85
574	45	313	90
653	50.5	326	94.5
635	56.5		

Four equilibrium solid phases were established: Pb_3Cs, Pb_2Cs, Pb_5Cs_4, and PbCs.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A glass ampoule containing metallic Cs was broken under transformer oil. The exact volume of Cs from which the mass was calculated was dosed by means of a special apparatus. The Cs sample was transferred into a glove box filled with Ar. The sample was cleaned in ether and placed in a Nb crucible containing the weighed Pb sample. The crucible was hermetically closed in a special apparatus. Differential thermal analysis was performed in a commercial apparatus which was calibrated on a Cu sample. These parts of the experiments were done under He atmosphere. Heating-cooling cycles were several times repeated.

SOURCE AND PURITY OF MATERIALS:

Pb: nothing specified.
Cs: "defined purity", yet unspecified.
He and Ar: purified by means of gettering with Ti turnings.

ESTIMATED ERROR:

Solubility: precision ± 0.5 %.
Temperature: precision ± 1 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lead; Pb; [7439-92-1] (2) Caesium; Cs; [7440-46-2]	Meijer, J.A.; Vinke, G.J.B..; Van der Lugt, W. *J. Phys.,F* 1986, *16*, 845-851.
VARIABLES:	**PREPARED BY:**
Temperature: 618-893 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Pb-K system were determined. They were taken from the figure and digitized by the compilers.

t/°C	*soly*/mol % Pb	t/°C	*soly*/mol % Pb	t/°C	*soly*/mol % Pb
420	5	562	44.5	540	67.5
435	10	593	47.5	535	70
455	15	620	50	525	75
469	18.5	609	53	500	79.5
473	25	593	55.5	460	85
488	29.5	571	60	414	90.5
500	34	542	65	345	95.5
524	39.5				

The results were also presented in (1).

Three equilibrium solid phases were suggested: PbCs, Pb_2Cs, and Pb_3Cs, they were, however, not identified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Cs was supplied in glass ampoules. The surfaces of the melts of both metals were skimmed to remove the adhering oxides. The preparation of the alloys and the measurements were performed in a dry He glove box. The two solid metals were placed in AISI 316 stainless steel crucibles provided by the commercial producer of the differential scanning calorimeter, in which heating and cooling curves were recorded.

SOURCE AND PURITY OF MATERIALS:

Pb: 99.999 % pure from Ventron.
Cs: 99.99+ % pure from Kawecki Berylco Industries.
He: contained < $1 \cdot 10^{-4}$ % O_2 and H_2O (each).

ESTIMATED ERROR:

Solubility: nothing specified; read-out procedure ± 0.3 mol %.
Temperature: precision ± 3 K.

REFERENCES:

1. Meijer, J.A. *Ph.D. thesis*, Groningen Univ., Netherlands, 1988.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Lead; Pb; [7439-92-1] (2) Caesium; Cs; [7440-46-2]	Saboungi, M.L.; Reijers, H.T.J.; Blander, M.; Johnson, G.K. *J. Chem. Phys.* 1988, *89*, 5869-5875.
VARIABLES:	**PREPARED BY:**
Temperature: 869 and 920 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Two temperatures of phase transitions for the equimolar alloy PbCs were observed at 869 and 920 K (647 and 596 °C as calculated by the compilers). The upper recorded value is probably the melting point of PbCs.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were loaded into a thick-walled Ta capsule. Its filling tube was then crushed, cut and sealed by means of arc welding in a dry He glove box. The drop calorimetric system consisted of a resistance heated Mo core furnace and a Cu block calorimeter. The temperature of the furnace was measured by a Pt/Pt-Rh thermocouple. The calorimeter was calibrated, and enthalpies at 298 K and selected temperatures were measured. The liquidus point was identified by means of a jump of (H_T-H_{298}) at the solid-liquid transition.

SOURCE AND PURITY OF MATERIALS:

Pb: "high purity", filtered after melting.
Cs: "high purity".
He: contained less than $1 \cdot 10^{-4}$ % O_2 and H_2O (each).

ESTIMATED ERROR:

Solubility: deviation from stoichiometry $\leq$0.1 %.
Temperature: precision ± 1 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Antimony; Sb; [7440-36-0] (2) Lithium; Li; [7439-93-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1991

CRITICAL EVALUATION:

Neither the solubility of Sb in liquid Li nor the liquidus of the Sb-Li system are known. Lebeau (1) identified Li_3Sb as a product of the reaction of Li and Sb in liquid NH_3 inside a sealed tube. The melting point of Li_3Sb was estimated to be higher than 1223 K. Brauer and Zintl (2) confirmed the formation of Li_3Sb in the direct reaction of the metals. They were able to roughly estimate the melting point of Li_3Sb as being between 1423 and 1573 K, and they observed a side reaction of the alloy with the Fe container. Gérardin and Aubry (3) prepared another compound Li_2Sb by means of the reaction of Sb with Li_3N. Li_2Sb was stable up to 1273 K and decomposed to the elements at 1473 K.

It might be concluded that the compound Li_3Sb should be the most stable one in the Sb-Li system with the melting point above 1273 K in analogy to the Sb-Na and Bi-Li systems. It may be expected, therefore, that the solubility of Sb in liquid lithium is similar as or lower than the solubility of Sb in liquid Na; see the Sb-Na system for comparison.

References

1. Lebeau, P. *Compt. Rend.* 1902, *134*, 284.
2. Brauer, G.; Zintl, E. *Z. Phys. Chem., B* 1937, *37*, 323.
3. Gérardin, R.; Aubry, J. *Compt. Rend., C* 1974, *278*, 1097.

COMPONENTS:	EVALUATOR:
(1) Antimony; Sb; [7440-36-0] (2) Sodium; Na; [7440-23-5]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland November 1991

CRITICAL EVALUATION:

Tammann (1) performed a first attempt to study the solvent properties of liquid Na in relation to Sb. He found that the melting point of Na was elevated by 0.02 K due to the addition of 0.075 to 0.47 mol % Sb. He measured, almost certainly, the peritectic temperature of a heterogeneous alloy instead of a part of the Na-Sb liquidus.

Mathewson (2) showed in thermal analyses of the Na-Sb system in the whole composition range that the solubility of Sb in Na in the neighborhood of the melting point of Na should be much smaller than that reported in (1). Ugai (3) confirmed the melting point of Na_3Sb and NaSb determined by (2).

Lamprecht (4) determined the solubility of Sb in liquid Na between 457 and 823 K. His results fit very well the line relating log (*soly*) vs. reciprocal temperature. Similar measurements of Weeks (5) at 611 to 847 K were scattered, and the slope of the average differed significantly from that of (4).

Evans and Watson (6) measured about 80 values of the solubility of Sb in liquid Na between 460 and 816 K. Their results were slightly scattered and showed better agreement with those of (5) than those of (4). A solubility equation based on the data of (5) and (6) may be recommended:

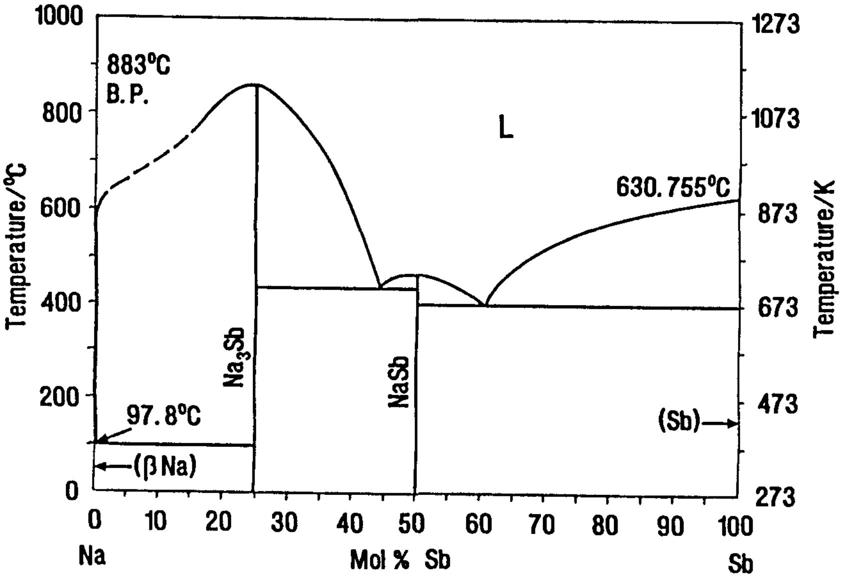
$$\log (soly/\text{mol \% Sb}) = (3.94 \pm 0.01) - (3940 \pm 30)(T/\text{K})^{-1}$$

An extrapolation of the solubility data reported in (4), (5), and (6) to higher temperatures suggests that Mathewson's liquidus values (2) for the Na-rich alloys are erroneous. The schematic Sb-Na phase diagram is shown in the figure.

Recommended (r), tentative (t) and doubtful (d) values of the solubility of Sb in liquid Na

T/K	*soly*/mol % Sb	source
473	$4 \cdot 10^{-5}$ (t)	(6)
573	$1 \cdot 10^{-3}$ (r)	(4), (5), (6); interpolated
673	$1 \cdot 10^{-2}$ (t)	(6)
773	$7 \cdot 10^{-2}$ (t)	(6)
873	0.3 (d)	(6); extrapolated
973	10 (d)	(2); interpolated
1073	19 (t)	(2); interpolated
1132	25 (r) congruent	(2), (3)

The two liquid metals are completely miscible at temperatures above 1132 K.



References

1. Tammann, G. *Z. Phys. Chem.* 1889, *3*, 441.
2. Mathewson, C.H. *Z. Anorg. Chem.* 1906, *50*, 171.
3. Ugai, Ya. A. *Voprosy Metallurgii i Fiziki Poluprovodnikov*, Izdat. Akad. Nauk SSSR, Moskva 1961, p. 107.
4. Lamprecht, G.J. *Ph.D. thesis*, Univ. of South Africa, Pretoria, 1966.
5. Weeks, J.R. Brookhaven National Laboratory, 1969, unpublished; as cited by Claar, T.D. *Reactor Technol.* 1970, *13*, 124.
6. Evans, H.E.; Watson, W.R. *J. Nucl. Mater.* 1971, *40*, 195.

COMPONENTS:

(1) Antimony; Sb; [7440-36-0]

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

ORIGINAL MEASUREMENTS:

Mathewson, C.H.

Z. Anorg. Chem. 1906, *50*, 171-198.

VARIABLES:

Temperature: 673-1129 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Na-Sb system were determined.

t/°C	*soly*/mol % Sb	*t*/°C	*soly*/mol % Sb	*t*/°C	*soly*/mol % Sb
443	5.4	447	46.5	400	60.6
762	15.9	462	48.7	450	65.4
856	25.4	465	51.3	555	80.6
772	33.9	458	53.7	593	90.4
578	41.6	440	56.8	620	95.0
435	44.5				

The melting points of Sb and Na were found to be at 97.5 and 630.6 °C, respectively.

Two equilibrium solid phases were identified: Na_3Sb and NaSb.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was melted under Vaseline oil, cooled to freezing, and its surface was cut off. Weighed amounts of the metals were placed in a high melting Jena glass tube under H_2 atmosphere. The molten alloys were mixed by means of stirring with a Fe wire. Thermal analytical arrests were taken from cooling curves. A Pt/Pt-Rh thermocouple was used for temperature measurements. It was calibrated on the melting points of Na, Pb, Sn, Zn, and Sb as in (1). Some alloys were analyzed by decomposition with H_2O. The formed NaOH was acidimetricly titrated.

SOURCE AND PURITY OF MATERIALS:

Sb: nothing specified.
Na: "pure"; purified by remelting and cutting off the surface (1).

ESTIMATED ERROR:

Nothing specified.
Solubility: precision ± 0.5 mol % in analogy to (1).
Temperature: precision ± 1 K (by the compilers).

REFERENCES:

1. Mathewson, C.H. *Z. Anorg. Chem.* 1905, *46*, 94-112.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Antimony; Sb; [7440-36-0] (2) Sodium; Na; [7440-23-5]	Ugai, Ya. A. *Voprosy Metallurgii i Fiziki Poluprovodnikov*, Izdat. Akad. Nauk SSSR, Moskva 1961, p. 107-109.
VARIABLES:	**PREPARED BY:**
Two temperatures: 738 and 1132 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The melting points of the two compounds Na_3Sb and NaSb were determined to be 859 and 465 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Proper amounts of both metals were placed into a steel crucible which was inserted in a refractory capsule hermetically closed. An inert dry gas served to protect the alloys. The capsule was heated in a furnace in which thermal analyses could be performed using a typical apparatus with unspecified thermocouple. The compositions of the alloys were checked by means of chemical analyses.

SOURCE AND PURITY OF MATERIALS:

Sb: 99.9979 % pure, denoted as "Su-000".
Na: 99.8 % pure.

ESTIMATED ERROR:

Solubility: accuracy of chemical analysis ± 0.01 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Antimony; Sb; [7440-36-0]

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

ORIGINAL MEASUREMENTS:

Lamprecht, G.J.

Ph.D. thesis, Univ. of South Africa, Pretoria, 1966.

VARIABLES:

Temperature: 456.6-823.2 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Sb in liquid Na was determined at several temperatures.

t/°C	*soly*/mass % Sb	*soly*/mol % Sb
183.4[c]	$1.7 \cdot 10^{-3}$	$3.3\ 10^{-4}$
193.0[a]	$2.2 \cdot 10^{-3}$	$4.2 \cdot 10^{-4}$
248.7[c]	$5.9 \cdot 10^{-3}$	$1.12 \cdot 10^{-3}$
279.6[a]	$9.2 \cdot 10^{-3}$	$1.7 \cdot 10^{-3}$
362.8[a]	$2.65 \cdot 10^{-2}$	$5.0 \cdot 10^{-3}$
367.8[b]	$2.47 \cdot 10^{-2}$	$4.7 \cdot 10^{-3}$
437.2[a]	$4.9 \cdot 10^{-2}$	$9.2 \cdot 10^{-3}$
467.5[b]	$6.35 \cdot 10^{-2}$	$1.2 \cdot 10^{-2}$
550.0[a]	$1.082 \cdot 10^{-1}$	$2.0 \cdot 10^{-2}$

[a] heating sequence [b] cooling sequence [c] from intermetallic compounds

The solubility equation was derived by the author and confirmed by the compilers:

$$\log(soly/\text{mol \% Sb}) = 0.548 - 1831\ (T/\text{K})^{-1}$$

The equilibrium solid phase was found to have changing composition, from $SbNa_{\sim 10}$ at 423 K to $SbNa_{\sim 5}$ at 573 K.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility apparatus was made of Pyrex glass. The experiments were performed in a He atmosphere. Sb was melted with ^{124}Sb obtained by irradiation. Sb was placed in one reaction cell and Na in a twin one. The cells were interconnected by means of a capillary. Molten Na was transferred into the first cell due to increased pressure. The metals were contacted until no further change of the activity of Sb in liquid Na occurred. The solution was transferred to the second cell for decay measurement. The variation of the solubility with the temperature was determined on the basis of the increase or decrease of the activity of the saturated Na. The equilibrium was reached from below and above a selected temperature.

SOURCE AND PURITY OF MATERIALS:

Sb: spectrally pure from Johnson Matthey.
Na: from Merck with contents of 0.002 % Cl, SO_4 and heavy metals; 0.001 % Fe, PO_4; 0.005 % N, Ca; 0.01 % K, and (1.1±0.2) 10^{-3} % O.
He: purified with molecular sieves, activated charcoal trapping at the temperature of liquid N.

ESTIMATED ERROR:

Solubility: standard deviation ± 4 %.
Temperature: stability better than ± 1 K.

REFERENCES:

COMPONENTS:

(1) Antimony; Sb; [7440-36-0]

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

ORIGINAL MEASUREMENTS:

Weeks, J.R.

Brookhaven National Laboratory, 1969, unpublished, as reported in (1).

VARIABLES:

Temperature: 611-847 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Sb in liquid Na was determined. The results were read out from the figure in (1) and recalculated to mol % by the compilers.

T/K	t/°C	*soly*/mass % Sb	*soly*/mol % Sb
611	338	$1.1\ 10^{-2}$	$2.0 \cdot 10^{-3}$
625	353	$5.0 \cdot 10^{-3}$	$9.5 \cdot 10^{-4}$
651	378	$6.0 \cdot 10^{-3}$	$1.1 \cdot 10^{-3}$
664	391	$2.2 \cdot 10^{-2}$	$4.2 \cdot 10^{-3}$
678	405	$4.2 \cdot 10^{-2}$	$7.9\ 10^{-3}$
724	451	$7.7 \cdot 10^{-2}$	$1.4 \cdot 10^{-2}$
771	498	$5.4\ 10^{-2}$	$1.0 \cdot 10^{-2}$
776	504	$3.1 \cdot 10^{-2}$	$5.9 \cdot 10^{-3}$
847	574	$3.4\ 10^{-1}$	$6.4 \cdot 10^{-2}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

No details of the determinations were reported in (1). The method was the same as was used for the determination of the solubility of Cd in liquid Na (2). The metals were introduced into a Zr crucible under inert atmosphere. The apparatus was held at 900 K for 48 hours and then cooled to the desired temperature at which it was kept for 24 hours. Each sampler was preheated for 15 minutes and the alloy was filtered into the sampler. The samples were analyzed and another test temperature was settled for a new equilibration over 24 hours.

SOURCE AND PURITY OF MATERIALS:

Sb: nothing specified.
Na: 99.98 % pure from MSA Research as in(2).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Claar, T.D. *Reactor Technol.* 1970, *13*, 124-146.
2. Weeks, J.R.; Davies, H.A. *The Alkali Metals*, The Chem. Soc., London, 1967, p. 32-37.

COMPONENTS:

(1) Antimony; Sb; [7440-36-0]

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

ORIGINAL MEASUREMENTS:

Evans, H.E.; Watson, W.R.

J. Nucl. Mater. 1971, *40*, 195-204.

VARIABLES:

Temperature: 460-816 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Sb in liquid Na was determined. The data were read out from the figure and recalculated to mol % Sb by the compilers.

t/°C	*soly*/mol % Sb	*t*/°C	*soly*/mol % Sb	*t*/°C	*soly*/mol % Sb
187	$1.9\cdot10^{-5}$	329	$2.3\cdot10^{-3}$	402	$1.3\cdot10^{-2}$
190	$2.9\cdot10^{-5}$	338	$2.4\cdot10^{-3}$	404	$1.2\cdot10^{-2}$
194	$3.5\cdot10^{-5}$	338	$3.7\ 10^{-3}$	405	$1.4\cdot10^{-2}$
200	$5.3\cdot10^{-5}$	340	$3.8\cdot10^{-3}$	406	$1.9\cdot10^{-2}$
201	$3.2\cdot10^{-5}$	347	$2.8\cdot10^{-3}$	408	$1.4\cdot10^{-2}$
201	$4.2\ 10^{-5}$	348	$4.5\cdot10^{-3}$	414	$1.5\cdot10^{-2}$
203	$4.9\cdot10^{-5}$	351	$5.6\cdot10^{-3}$	420	$2.0\cdot10^{-2}$
204	$6.0\cdot10^{-5}$	353	$5.3\cdot10^{-3}$	425	$1.9\cdot10^{-2}$
205	$4.2\cdot10^{-5}$	364	$6.2\cdot10^{-3}$	441	$2.3\cdot10^{-2}$
207	$7.0\cdot10^{-5}$	366	$6.2\cdot10^{-3}$	449	$3.1\cdot10^{-2}$
207	$6.8\cdot10^{-5}$	368	$6.8\cdot10^{-3}$	456	$3.8\cdot10^{-2}$
207	$6.4\cdot10^{-5}$	372	$5.9\cdot10^{-3}$	462	$4.2\cdot10^{-2}$
208	$5.0\ 10^{-5}$	374	$7.5\cdot10^{-3}$	473	$5.8\cdot10^{-2}$
209	$5.9\cdot10^{-5}$	378	$6.4\cdot10^{-3}$	481	$5.1\cdot10^{-2}$
212	$5.9\cdot10^{-5}$	378	$8.1\cdot10^{-3}$	484	$6.8\cdot10^{-2}$
212	$7.4\cdot10^{-5}$	379	$7.4\cdot10^{-3}$	496	$5.9\cdot10^{-2}$
215	$7.7\cdot10^{-5}$	382	$8.1\ 10^{-3}$	496	$6.2\cdot10^{-2}$
219	$1.1\cdot10^{-4}$	383	$8.0\ 10^{-3}$	509	$8.1\cdot10^{-2}$
219	$1.0\cdot10^{-4}$	386	$9.3\cdot10^{-3}$	512	$9.3\cdot10^{-2}$
219	$9.4\cdot10^{-5}$	388	$8.1\cdot10^{-3}$	516	$1.05\cdot10^{-1}$
219	$9.0\cdot10^{-5}$	391	$1.0\cdot10^{-2}$	518	$8.9\cdot10^{-2}$
219	$8.1\cdot10^{-5}$	394	$8.9\ 10^{-3}$	534	$1.18\ 10^{-1}$
224	$8.1\cdot10^{-5}$	394	$1.0\cdot10^{-2}$	536	$1.36\cdot10^{-1}$
230	$1.5\cdot10^{-4}$	394	$1.2\cdot10^{-2}$	536	$1.53\cdot10^{-1}$
236	$1.7\cdot10^{-4}$	398	$1.2\cdot10^{-2}$	543	$1.53\cdot10^{-1}$
242	$1.9\ 10^{-4}$	399	$1.1\ 10^{-2}$		

The data were fitted to the equation which was tested by the compilers:

$$\log\,(soly/\text{mol \% Sb}) = (3.937\pm0.007) - (3943\pm23)(T/\text{K})^{-1}$$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Irradiated Sb was placed in a stainless steel capsule in which strips of the same material were also suspended. Na was added in an evacuated filling rig, and a cap was arc welded on the capsule under Ar atmosphere. Further additions of Sb were made in a glove box in which the capsules were opened and closed again. The capsule was equilibrated at a selected temperature. The uniformity of the ^{124}Sb distribution in the capsule was measured using a γ-detector. At saturation Sb is deposited, and an equilibrium of the distribution of Sb between the solution and the stainless steel absorber is reached. The percentage of the deposited Sb was evaluated from the distribution equilibrium. The weight of dissolved Sb could then be calculated with the knowledge of the amount of added Sb.

SOURCE AND PURITY OF MATERIALS:

Sb: unspecified purity from Hopkin and Williams Ltd.

Na: reactor grade purity from UKAEA, Culcheth Laboratories.

ESTIMATED ERROR:

Solubility: standard deviation ± 2.5 %.

Temperature: accuracy between ± 0.5 and ± 2.5 K; higher scatter at higher temperatures.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Antimony; Sb; [7440-36-0] (2) Potassium; K; [7440-09-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland December 1991

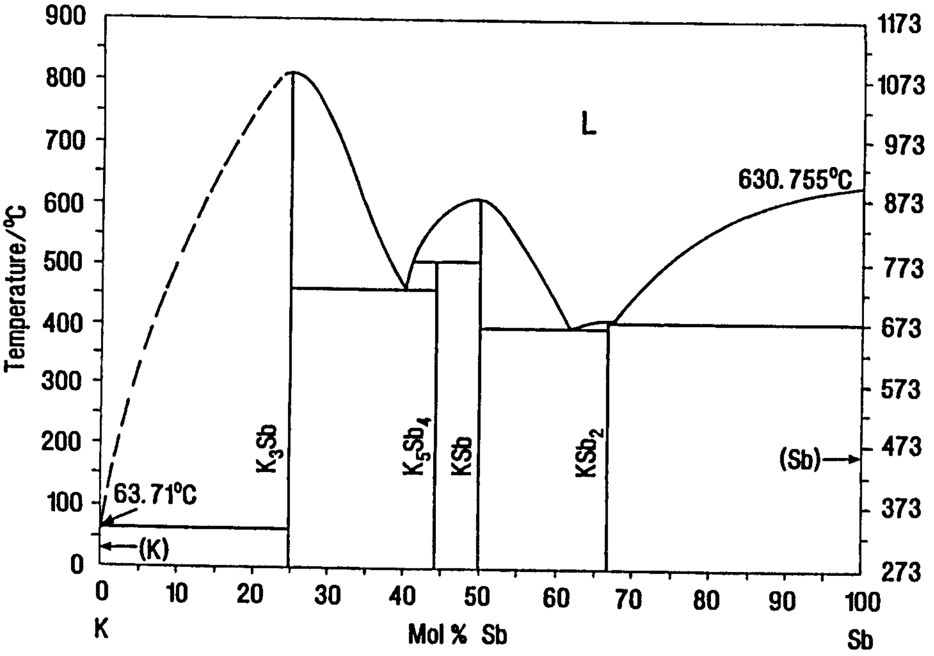
CRITICAL EVALUATION:

There are two experimental studies (1,2) of the liquidus of the K-Sb system on the basis of thermal analysis. Since the thermal arrests of the liquidus observed by Parravano (1) between 30 and 38 mol % Sb were up to 50 K lower than those recorded by Dorn and Klemm (2), it is likely that the melting point of K_3Sb and the liquidus of the K-richest part are placed at too low temperatures. Thus, too high solubilities of Sb in liquid K might be suggested. The liquidus line of (2) may be more realistic for the Sb-rich side.
The schematic K-Sb phase diagram is shown in the figure after (4). The equilibrium solid phases, K_3Sb, K_5Sb_4, KSb, and KSb_2, reported by (2) were confirmed by (3).

Doubtful values of the solubility of Sb in liquid K

T/K	*soly*/mol % Sb	source
523	3	(1)
673	7	(1)
773	10	(1); interpolated
873	14	(1)
973	18	(1); interpolated
1085	25 congruent	(1)

The miscibility of the two metals is unlimited in the liquid state above 1085 K.



References

1. Parravano, N. *Gazz. Chim. Ital.* 1915, *45*, Pt. I, 485.
2. Dorn, F.W.; Klemm, W. *Z. Anorg. Chem.* 1961, *309*, 189.
3. Kansky, E. *Thin Solid Films* 1976, *34*, 139.
4. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am.Soc.Mater., Materials Park, 1990, p. 2386.

COMPONENTS:

(1) Antimony; Sb; [7440-36-0]

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

ORIGINAL MEASUREMENTS:

Parravano, N.

Gazz. Chim. Ital. 1915, *45*, Pt. I, 485-489.

VARIABLES:

Temperature: 423-1085 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the K-Sb system was determined.

t/°C	*soly*/mass % Sb	*soly*/mol % Sb [a]	*t*/°C	*soly*/mass % Sb	*soly*/mol % Sb [a]
250	10	3.4	575	70	42.8
420	20	7.4	600	73	46.5
545	30	12.1	605	73.5	49.7
575	33	13.7	585	78	53.2
812	50.61	24.8 congruent	555	80	56.2
740	53	26.6	520	82	59.4
700	55	28.2	490	84	62.8
685	56	29.0	450	85	64.5
650	58	30.7	430	86	66.4
550	60	32.5	445	88	70.2
485	62.5	34.9 eutectic	505	90	74.3
505	65	37.4	545	92	78.7
545	68	40.6	585	94	83.1

[a] calculated by the compilers

The melting points of Sb and K were found to be at 631 and 63.6 °C, respectively. The solid equilibrium phases, KSb and K_3Sb, were identified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The surface of K was cut off under benzene with amyl alcohol and washed with ether. The metal was then immediately weighed. Containers were made for all alloy compositions of Fe to avoid corrosion, since the reaction of the mixed components is significantly exothermic. A porcelain tube sheath for the thermocouple was used for K-rich alloys, and a quartz tube for the same purpose in Sb-rich alloys. The thermal analyses of the alloy samples were performed under H_2 atmosphere.

SOURCE AND PURITY OF MATERIALS:

Sb: "pure" from Kahlbaum.
K: "pure" from Kahlbaum.
H_2: "dried".

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Antimony; Sb; [7440-36-0]

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

ORIGINAL MEASUREMENTS:

Dorn, F.W.; Klemm, W.

Z. Anorg. Chem. 1961, *309*, 189-203.

VARIABLES:

Temperature: 665-1010 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Parts of the liquidus line of the K-Sb system were determined.

t/°C	*soly*/mol % Sb	t/°C	*soly*/mol % Sb	t/°C	*soly*/mol % Sb
737	30.6	596.5	51.9	411	65.9
648	33.9	576	53.9	411	66.5
513	37.2	526	56.8	410	67.5 eutectic
487	39.6	446	59.7	431	68.9
508	41.5	440	60.2	450	70.0
550	43.6	397.5	61.3 eutectic	467	71.5
569	44.7	398.5	62.5	491.5	73.7
596	46.7	401	62.7	519	76.3
606.0	48.1	399.0	63.7	540.5	79.1
610.0	50.0 congruent	411	65.0		

The melting points of Sb and K were found to be at 630.5 and 63.4 °C, respectively.
Four equilibrium solid phases were identified: K_3Sb, K_5Sb_4, KSb, and KSb_2.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in an apparatus made of quartz. The apparatus was filled with Ar, K was introduced inserted in glass ampoules. The amounts of molten K, which were transferred into the apparatus, were evaluated from the weight differences before and after the transfer procedure. Sb was carefully introduced into the K samples. The apparatus was heated under Ar atmosphere. Thermal analysis was performed by means of a Pt/Pt-Rh thermocouple. The composition of the alloys was determined by chemical analyses.

SOURCE AND PURITY OF MATERIALS:

Sb: obtained by reduction of Sb_2O_3 with KCN (both from Merck), and purified by distillation at 923-973 K in high vacuum.
K: "commercial product", purified by distillation in high vacuum.

ESTIMATED ERROR:

Solubility: precision ± 0.2 mol %.
Temperature: precision between ± 0.5 and ± 1.5 K.

REFERENCES:

COMPONENTS:

(1) Antimony; Sb; [7440-36-0]

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

ORIGINAL MEASUREMENTS:

Dorn, F.W.; Klemm, W.

Z. Anorg. Chem. 1961, *309*, 189-203.

VARIABLES:

Temperature: 678-1006 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

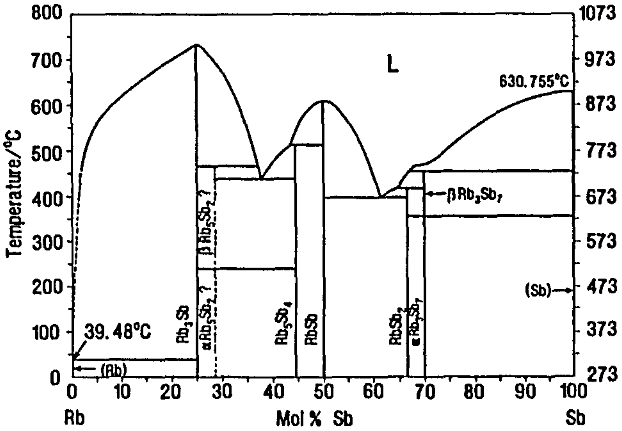
EXPERIMENTAL VALUES:

The liquidus line of the Sb-Rb system was determined. The solubility data are reliable.

t/°C	*soly*/mol % Sb	*t*/°C	*soly*/mol % Sb	*t*/°C	*soly*/mol % Sb	*t*/°C	*soly*/mol % Sb
482	2.04	481	36.5	603	51.6	446	
521	3.04	452	37.6	597	52.3	450.5	66.5
543	4.07	454	39.0	587	53.2	456	67.2
564	5.04	473	40.0	571	54.3	465	67.9
604	7.51	491	40.9	553	55.1	467	68.6
623	10.2	507	42.0	543	55.9	468	69.3
636	12.5	516	43.0	526	56.8	470	70.1
659	15.3	540	44.0	491	57.7	475	70.7
696	20.7	559	44.8	463	58.8	484	71.5
733	25.0	569	45.7	440	59.8	507	73.1
680	29.9	585	46.5	410	60.8	524	74.9
649	31.2	596	47.3	405	61.8	538	76.6
627	32.2	603	48.1	412	62.7	558	78.2
592	33.4	607.0	48.8	417	63.5	573	80.6
543	34.8	609.0	49.5	430	64.3	593	82.9
485	36.1	610.0	50.2	433	65.0	608	85.5
442	36.9	607	50.9	441	65.8		92.5

COMMENTS AND ADDITIONAL DATA:

The melting points of Sb and Rb were found to be at 630.5 and 38 °C, respectively. Some equilibrium solid phases were identified: Rb_3Sb, Rb_5Sb_2(?), Rb_5Sb_4, RbSb, $RbSb_2$, and Rb_3Sb_7 as it is shown in the figure of the Sb-Rb phase diagram (2). The composition of the solid phases was confirmed by (1).



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in an apparatus made of quartz. The apparatus was filled with Ar, Rb was introduced inserted in glass ampoules. The amounts of molten Rb, which were transferred into the apparatus, were evaluated from the weight differences before and after the transfer procedure.

Sb was carefully introduced into the Rb samples and the apparatus was heated under Ar atmosphere.

Thermal analysis was performed by means of a Pt/Pt-Rh thermocouple. The composition of the alloys was determined by chemical analyses.

SOURCE AND PURITY OF MATERIALS:

Sb: obtained by reduction of Sb_2O_3 with KCN (both from Merck), and purified by distillation at 923-973 K in high vacuum.

Rb: obtained by reduction of RbCl with Ca at 973 K in vacuum, finally vacuum distilled.

ESTIMATED ERROR:

Solubility: precision ± 0.2 mol %.

Temperature: precision between ± 0.5 and ± 1.5 K.

REFERENCES:

1. Kansky, E. *Thin Solid Films* 1976, *34*, 139-144.
2. *Binary Alloy Phase Diagrams*, T.B. Massalski, Ed., Am.Soc.Mater., Materials Park, 1990, p. 3189.

COMPONENTS:	EVALUATOR:
(1) Antimony; Sb; [7440-36-0] (2) Cesium; Cs [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland December 1991

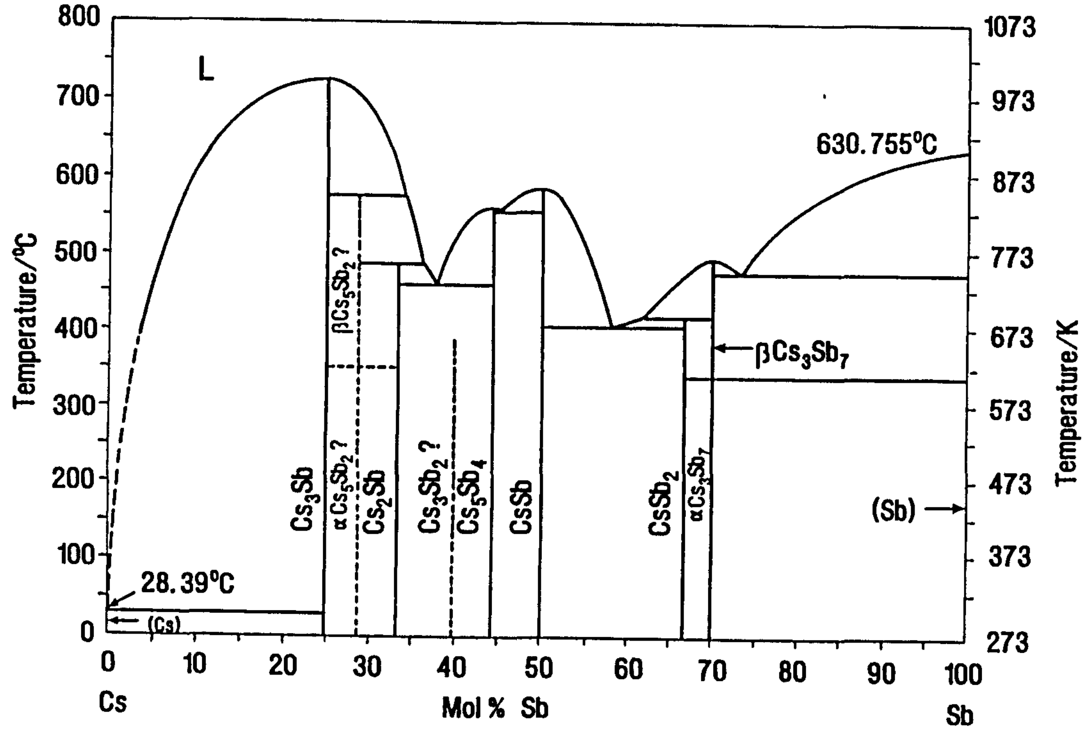
CRITICAL EVALUATION:

Dorn and Klemm (1) applied the thermal analysis to determine the liquidus of the Cs-Sb system. Their results seem to be reliable. Kansky (2) reported the existence of more equilibrium solid phases in the system. These findings are inserted in the Sb-Cs phase diagram shown in the figure.

Tentative values of the solubility of Sb in liquid Cs

T/K	*soly*/mol % Sb	source
673	2	(1)
773	5	(1)
873	12	(1); interpolated
973	19	(1); interpolated
998	25 congruent	(1)

The two liquid metals are completely miscible above 998 K.



References

1. Dorn, F.W.; Klemm, W. *Z. Anorg. Chem.* 1961, *309*, 189.
2. Kansky, E. *Thin Solid Films* 1976, *34*, 139.

COMPONENTS:

(1) Antimony; Sb; [7440-36-0]

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

ORIGINAL MEASUREMENTS:

Dorn, F.W.; Klemm, W.

Z. Anorg. Chem. 1961, *309*, 189-203.

VARIABLES:

Temperature: 677-998 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Cs-Sb system was determined.

t/°C	*soly*/mol % Sb	*t*/°C	*soly*/mol % Sb	*t*/°C	*soly*/mol % Sb	*t*/°C	*soly*/mol % Sb
411	2.1	686	30.5	561	52.7	493	70.0
446	3.1	667	31.6	546	53.7	485	71.0
475	4.1	638	32.6	523	54.9	480	72.0
500	5.2	596	33.6	482	56.6	473	73.0
544	7.9	560	34.6	445	57.7	473	74.0 eutectic
581	10.6	536	35.7	404	58.7	487	75.0
616	13.4	499	36.7	404	59.7	494	76.0
652	16.1	477	37.7	410	60.7	511	77.2
692	18.9	467	38.7	415	61.8	525	78.3
725	25.0 congruent	502	39.7	(419)	63.1	547.5	79.6
696	29.8	529	40.7	434	58.0	559.5	81.0
682	30.6	547	42.0	404	59.0	(566)	82.2
674	31.2	559.0	43.3	405	60.6	587	85.0
655	32.1	560.0	44.2	409	61.0	597	86.7
625	33.0	554.5	45.2	411	62.0	607	88.0
578	34.1	563	46.2	407	63.0	606	88.2
552	35.1	573	47.2	427	64.0	607	89.1
520	36.2	582.5	48.4	448	65.0	610	90.4
493	37.0	585.5	49.7 congruent	467	66.15	613	91.8
478	37.7	582	50.7	472	66.67	616	93.5
467	38.7	572	51.7	483	68.0	617.5	94.8
				489	69.0	619	96.1

The melting points of Sb and Cs were found to be at 630.5 and 38 °C, respectively.

Some equilibrium solid phases were identified: Cs_3Sb, Cs_2Sb_5(?), Cs_2Sb, Cs_5Sb_4, CsSb, $CsSb_2$, and Cs_3Sb_7.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in an apparatus made of quartz. The apparatus was filled with Ar, Cs was introduced inserted in glass ampoules. The amounts of molten Cs, which were transferred into the apparatus, were evaluated from the weight differences before and after the transfer procedure. Sb was carefully introduced into the Cs samples. The apparatus was heated under Ar atmosphere. Thermal analysis was performed by means of a Pt/Pt-Rh thermocouple. The composition of the alloys was determined by chemical analyses.

SOURCE AND PURITY OF MATERIALS:

Sb: obtained by reduction of Sb_2O_3 with KCN (both from Merck), and purified by distillation at 923-973 K in high vacuum.
Cs: obtained by reduction of CsCl with Ca at 973 K in vacuum.

ESTIMATED ERROR:

Solubility: precision ± 0.2 mol %.
Temperature: precision between ± 0.5 and ± 1.5 K.

REFERENCES:

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

EVALUATOR:

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

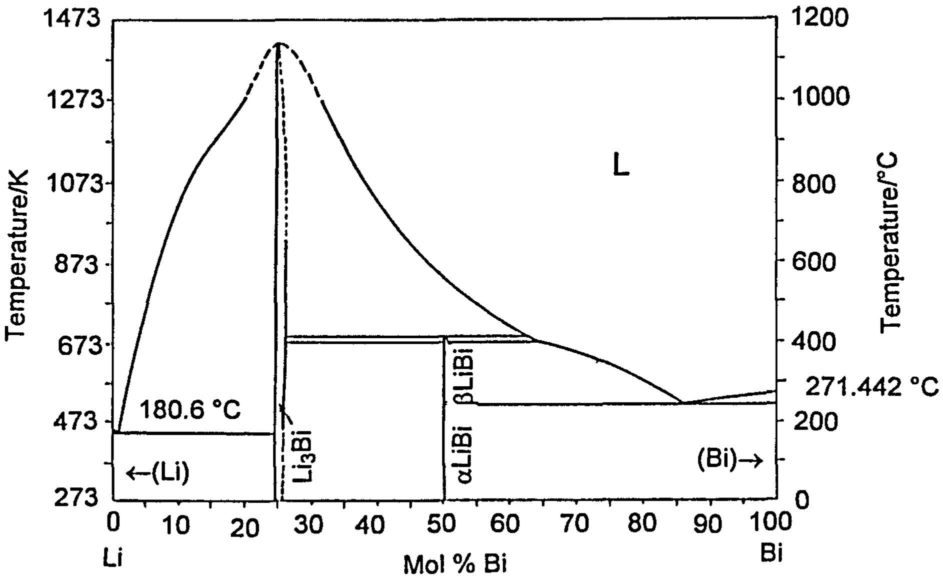
CRITICAL EVALUATION:

Grube et al. (1) performed thermal analyses of Li-Bi alloys over the whole composition range. They observed a quite sharp maximum on the liquidus line which was related to the congruently melting compound Li_3Bi (m.p. 1418 K). The eutectic composition on the Li-rich side of the system, which was originally proposed to contain 2.5 mol % Bi, should be shifted to less than 10^{-2} mol % Bi in analogy to the well-established Na-Bi system. Foster et al. (2) confirmed the liquidus in the range 41.5 to 55 mol % Bi and Weppner and Huggins (5) in the range 54.5-69.5 mol % Bi by means of emf measurements. Steinleitner et al. (3) reported excess volume measurements on liquid Li-Bi alloys between 21.1 and 26.8 mol % Bi at 1263 to 1323 K. These alloys should contain the solid phase Li_3Bi according to (1). Therefore, the suggested solubility data are classified as doubtful. The Li-Bi phase diagram is redrawn from (4) in the figure.

Doubtful values of the solubility of Bi in liquid Li.

T/K	*soly*/mol % Bi	source
773	4	(1); extrapolated
873	6	(1); interpolated
973	9	(1); interpolated
1073	11	(1); interpolated
1173	14	(1); interpolated
1273	18	(1); interpolated
1373	22	(1); interpolated
1418	25 congruent	(1)

The two metals are completely miscible in the liquid state above 1418 K.



References

1. Grube G.; Voßkühler H.; Schlecht, H. *Z. Elektrochem.* 1934, *40*, 270.
2. Foster, M.S.; Wood, S.E.; Crouthamel, C.E. *Inorg. Chem.* 1964, *3*, 1428.
3. Steinleitner, G.; Freyland, W.; Hensel, F. *Ber. Bunsenges. Phys. Chem.* 1975, *79*, 1186.
4. Sangster, J.; Pelton, A.D. *J.Phase Equil.* 1991, *12*, 447.
5. Weppner, W.; Huggins, R.A. *J.Electrochem.Soc.* 1978, *125*, 7.

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Grube G.; Voßkühler, H.; Schlecht, H.

Z. Elektrochem. 1934, *40*, 270-274.

VARIABLES:

Temperature: 513-1418 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Li-Bi system was determined.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi
535	5.0	970	31.0	460	60.0
729	10.0	990	31.5	417	63.0
922	15.0	884	35.0	401	65.0
957	17.0	840	37.0	392	67.5
1013	20.0	810	38.0	371	70.0
1076	23.0	772	40.0	366	71.5
1140	24.5	707	43.0	341	75.0
1145	25.0 congruent	684	45.0	302	80.0
1122	26.0	651	47.5	252	85.0
1105	27.0	645	48.0	240	86.0 eutectic
1087	28.0	615	50.0	251	90.0
1037	29.0	535	55.0	262	95.0
1007	30.0	497	57.5		

The melting points of Bi and Li were found to be 271 and 180 °C, respectively.

The solid equilibrium phases Li_3Bi and LiBi were identified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared under purified Ar atmosphere in a crucible made of Fe with low C content. The shielding of the thermocouple was made of the same material. The alloys were melted and cooled to solidification. Cooling curves were recorded using a Ni/Ni-Cr thermocouple which was calibrated on the melting points of Sn, Cd, Zn, and Mg (1). Some of the alloys were chemically analyzed after the thermal analyses by means of unspecified analytical methods.

SOURCE AND PURITY OF MATERIALS:

Bi: unspecified purity, from Kahlbaum.
Li: 99 % pure with contents of 0.62 % K, 0.14 % Na, 0.02 % Fe_2O_3, 0.05 % SiO_2, 0.32 % Li_3N, and traces of Al_2O_3 (1).
Ar: 98.2 % pure with 0.1 % O_2 and 1.7 % N_2, further purified by means of flowing through molten Li.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Grube G.; Voßkühler, H.; Vogt, H. *Z. Elektrochem.* 1932, *38*, 869-880.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Bismuth; Bi; [7440-69-9] (2) Lithium; Li; [7439-93-2]	Foster, M.S.; Wood, S.E.; Crouthamel, C.E. *Inorg. Chem.* 1964, *3*, 1428-1431.
VARIABLES:	**PREPARED BY:**
Temperature: 800-1000 K	H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the Li-Bi liquidus were determined. The compositions of the saturated solutions were read out from the figure by the compilers.

T/K	*soly*/mol % Bi
800	55
850	51.5
900	48
950	45
1000	41.5

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The electrochemical cells were operated in a Ta furnace attached to a dry He box. A porous BeO crucible contained the alloy and separated it from direct contact with the eutectic salt melt LiCl-LiF. Li was contained in a sintered 430 stainless steel fibre sponge. Ta wires were used for electrical connections with the electrodes. The alloys were prepared from appropriate quantities of the metals. The potentials of cells: Li/LiCl-LiF/Bi-Li were measured as a function of the composition and temperature. The solubility values were determined from the fitting equation relating the potential on the temperature and composition at the point at which the potential of the electrode is equal to the potential of the reference electrode, liquid Bi saturated with solid Li_3Bi.

SOURCE AND PURITY OF MATERIALS:

Bi: from United Mineral and Chem. Corp., N.Y., with contents of $4\ 10^{-4}$ % Ag; $1{\cdot}10^{-4}$ % Cu,Fe; $2{\cdot}10^{-4}$ % Pb; further purified by melting and filtering under He atmosphere.
Li: from Foote Mineral Comp., with contents of $3{\cdot}10^{-3}$ % Na, Cl; $2.8{\cdot}10^{-3}$ % K and $3.1\ 10^{-3}$ % N.
LiCl and LiF: reagent grade purity, purified by means of passing through Cl_2 and He, stored in He atmosphere.
He: purified on a Pd catalyst, molecular sieves, activated charcoal immersed in liquid N; contained $(1\text{-}5){\cdot}10^{-5}$ % H_2O and $< 1.0{\cdot}10^{-3}$ % O_2, N_2.

ESTIMATED ERROR:
Nothing specified.
Potential: standard deviation ± 3 mV

REFERENCES:

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Weppner, W.; Huggins, R.A.

J. Electrochem. Soc. 1978, *125*, 7-14.

VARIABLES:

Temperature: 648-836 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points of the Li-Bi liquidus were determined.

t/°C	*soly*/mol % Bi
375	69.5
409	62.5
454	60.5
563	54.5

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Electrochemical titration and measurements of potential differences were performed in a galvanic cell: AlLi-Al/LiCl-KCl eutectic/Bi-Li. Electric leads were made of Mo. Bi, which was contained in a Mo bucket, was used as starting material. Alumina crucibles were used as containers for the molten salt. Temperatures were measured by means of Chromel/Alumel thermocouples protected by stainless steel tubes. The coulometric titrations were performed under constant current through the cell. The titrations were made in both current directions. All operations were performed in a He atmosphere. The liquidus points were determined from break points of the relation of logarithm of potential vs. composition of the samples.

SOURCE AND PURITY OF MATERIALS:

Bi: 99.999+ % pure.
Li: 99.9+ % pure.
LiCl-KCl: spectrographic purity from Anderson Physics Lab., finally dried.

ESTIMATED ERROR:

Nothing specified. Potential stability ± 1 mV.
Solubility: precision ± 0.3 mol % (by the compilers).
Temperature: precision ± 2 K (by the compilers).

REFERENCES:

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

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

CRITICAL EVALUATION:

Though the Na-Bi system was frequently investigated, a selection of precise values of the solubilities and liquidus points is difficult due to the significant scatter of the experimental data over the whole composition range. Tammann (1) observed an increase of the melting temperature by 0.02 K due to the addition of 0.01 to 0.22 mol % Bi to Na; he almost certainly measured the peritectic temperature instead of a part of the liquidus (the work is not compiled). Heycock and Neville (2) recorded the depression of the melting temperature of Bi by additions of Na. Their results did not fit the subsequent observations of (5) (still lower temperatures) or (3),(4),(13),(16) (higher temperatures), and the accepted value of melting point of Bi at 544.592 K.
Kurnakov and Kuznetsov (3) studied the shape of the Na-Bi phase diagram by means of thermal analysis. They found the melting point of Na_3Bi to be about 120 K below the value which was reported in (6), (9), (12), (13); the eutectic temperature on the Bi-rich side was also not confirmed. A similar study of Mathewson (4) in which thermal analysis was applied agreed roughly with (3). The melting point of Na_3Bi was reported (4) to be 1049 K, a still too low value. This result was confirmed by Vournasos (5) who also determined the melting point of the alloy with 50 mol % Bi to be at 718 K and of that with 67.7 mol % Bi to be at 487 K. These two values reflect the peritectic and eutectic temperatures; they do not correspond to liquidus points, and (5) is therefore not compiled.
Fischer et al. (6) corrected the melting point of Na_3Bi to the value of 1115 K [see data sheet in (13)]. Gehri et al. (9) placed it at 1118 K, Frederikson et al. (12) at 1124 K [see data sheet in (13)], and Johnson et al. (13) at 1121 K. The liquidus lines in the Bi-rich region which were reported in (13) and (16) are in agreement within ± 5 K; thus, their mean values may be accepted as tentative.
The solubility of Bi in liquid Na was determined by Foster (7), Gehri et al. (8-10), Walker and Pratt (14), and Cornell (17). Electron spin resonance measurements were performed by the author of (17) which were used to calculate the solubility of Bi in liquid Na from 373 to 473 K. An increase from $4{\cdot}10^{-4}$ to $2.3{\cdot}10^{-3}$ mol % Bi was found. These values are orders of magnitude above the accepted values. The original paper (17) was not available, so the work was not compiled. The solubility data of (7), (8-10), (13), and (14) are in a scatter band of ± 10 %, and a plot of logarithm of solubility vs. reciprocal temperature is not linear. The second-order equation given by (14) and tested by the compilers seems to fit best the four sets of data between 373 and 1050 K.

$$\log(soly/\text{mol \% Bi}) = 7.7169 - 8131.6(T/\text{K})^{-1} + 1.3774{\cdot}10^{6}(T/\text{K})^{-2} \qquad \text{Eq.(1)}$$

Claar (15) and Gehri et al. (9,10) expressed the solubility curve by two equations:

$$\log(soly/\text{mol \% Bi}) = 1.188 - 2100(T/\text{K})^{-1} \text{ for 398 to 563 K} \qquad \text{Eq.(2)}$$
$$\log(soly/\text{mol \% Bi}) = 4.900 - 4178(T/\text{K})^{-1} \text{ for 563 to 923 K} \qquad \text{Eq.(3)}$$

Since no solid phase transformation of $BiNa_3$ was observed in the Bi-Na phase diagram at about 563 K, the suggestion to use Eqs. (2) and (3) does not have a sufficient rationale. Gehri et al. (9,10) argued that the necessity to use two equations may be due to the influence of Bi-Na-O interactions. Such interactions do not occur at higher temperatures, at which the equilibrium solute is $BiNa_3$. Further experiments are necessary in order to either accept or reject this concept. The schematic Na-Bi phase diagram based on (13) is redrawn in the figure; it would appear that the melting point of Na_3Bi recorded by (3) is in fact its solid transformation temperature observed by (9).

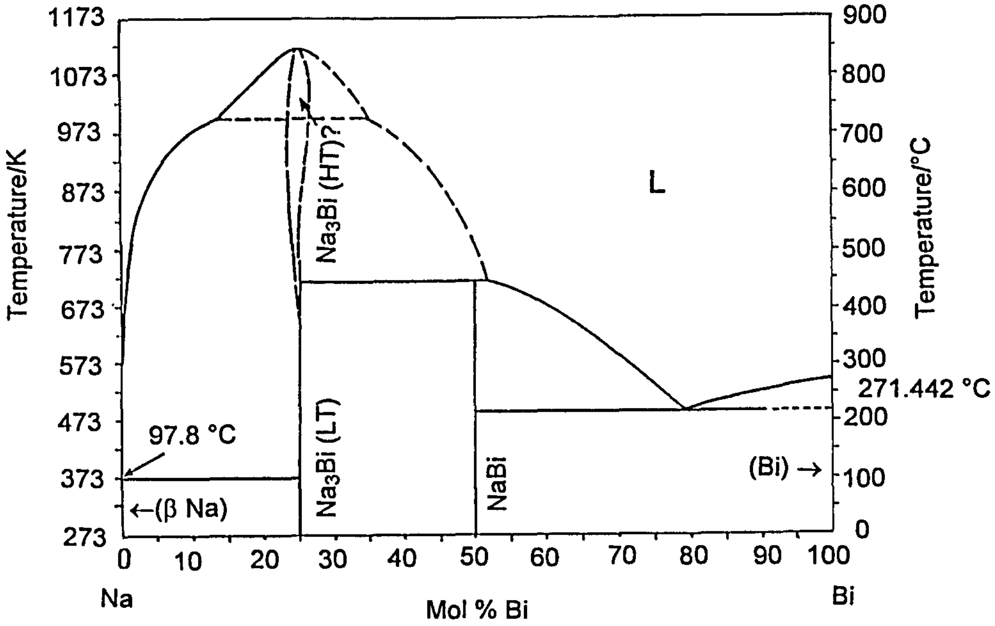
Recommended (r) and tentative (t) solubility values of Bi in liquid Na

T/K	*soly*/mol % Bi		source
373	$6.5{\cdot}10^{-5}$	(t)	Eq.(1)
473	$4.8{\cdot}10^{-4}$	(t)	Eq.(1); mean value of (9),(7)
573	$5.3\ 10^{-3}$	(t)	Eq.(1); (14)
673	$4.7{\cdot}10^{-2}$	(t)	Eq.(1)
773	$3.1{\cdot}10^{-1}$	(t)	Eq.(1); mean value of (9),(14)
873	1.6	(t)	Eq.(1)
973	9	(t)	(13),(14)
1073	20	(t)	(6),(13)
1121	25	(r)	(9),(12),(13); mean value

The two metals are completely miscible in the liquid state above 1121 K.

COMPONENTS: (1) Bismuth; Bi; [7440-69-9] (2) Sodium; Na; [7440-23-5]	EVALUATOR: H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland January 1992

CRITICAL EVALUATION: (continued)



References

1. Tamman, G. *Z. Phys. Chem.* 1889, *3*, 441.
2. Heycock, C.T.; Neville, F.H. *J. Chem. Soc.* 1892, *61*, 888.
3. Kurnakov, N.S.; Kuznetsov, A.N. *Zh. Russ. Fiz.-Khim. Obshch.* 1899, *31*, 927; *Z. Anorg. Chem.* 1900, *23*, 439.
4. Mathewson, C.H. *Z. Anorg. Chem.* 1906, *50*, 171.
5. Vournasos, A.G. *Compt. Rend.* 1911, *152*, 714; *Ber. Deutsch. Chem. Ges.* 1911, *44*, 3267.
6. Fischer, A.K.; Johnson, S.A.; Wood, S.E. *J. Phys. Chem.* 1967, *71*, 1465.
7. Foster, M.S. *Argonne National Laboratory*, 1968; reported in (9) and (13).
8. McKisson, R.L.; Eichelberger, R.L.; Gehri, D.C.; Guon, J. *U.S. Atom. Ener. Comm. Rep. AI-AEC-12744* 1968, p. 131.
9. Gehri, D.C.; Sullivan, R.J. *U.S. Atom. Ener. Comm. Rep. AI-AEC-12770*, 1969.
10. Eichelberger, R.L.; Gehri, D.C.; Sullivan, R.J. *Trans. Am. Nucl. Soc.* 1969, *12*, 614.
11. Meyer, R.J.; Johnson, C.E.; Ross, L.E. *U.S. Atom. Ener. Comm. Rep. ANL-7520* 1969, Pt. I, p. 291.
12. Fredrickson, D.R.; Chasanov, M.G.; Barnes, R.D.; Johnson, S.A. *High Temp. Sci.* 1970, *2*, 259.
13. Johnson, C.E.; Fisher, A.K *J. Less-Common Met.* 1970, *20*, 339.
14. Walker, R.A.; Pratt, J.N. *J. Nucl. Mater.* 1970, *34*, 165.
15. Claar, T.D. *Reactor Technol.* 1970, *13*, 124.
16. Yih, T.S.; Thompson, J.C. *J. Phys., F* 1982, *12*, 1625.
17. Cornell, E.K. *Ph.D. thesis*, Univ. of Illinois, 1969; as cited by (14).

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Heycock, C.T.; Neville, F.H.

J. Chem. Soc. 1892, *61*, 888-914.

VARIABLES:

Temperature: 531.2-538.0 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The depression of the melting point of Bi due to the addition of Na was determined.

t/°C	*soly*/mol Na per 100 mol Bi	*soly*/mol % Bi [a]
264.86	0.825	99.18
261.43	2.41	97.65
258.06	4.00	96.16

[a] as calculated by the compilers

The melting point of Bi was found to be at 266.34 °C.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The weighed amounts of the two metals were filled into a hard glass tube which was then evacuated and sealed. The tube with content was heated to red heat and well shaken. The alloy made in this way was added to the solvent in a block.

The thermometers used for measurements of the melting points were calibrated on the boiling points of Hg and S.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Solubility: nothing specified.

Temperature: precision ± 0.05 K.

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Kurnakov, N.S.; Kuznetsov, A.N.

Zh. Russ. Fiz.-Khim. Obshch. 1899, *31*, 927-948.

VARIABLES:

Temperature: 477-1003 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the liquidus line of the Na-Bi system were determined.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi [a]
352.0	0.5	236	75.0
~730	25.0	204	80.0
367	65.0	232	85.0
300	70.0	246	90.0

The melting points of Bi and Na were found to be at 268 and 96.0 °C, respectively.

The same results were reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared in glass tubes under H_2 atmosphere. Their Bi content was analytically determined. The freezing points were measured by means of a pyrometer or a Hg thermometer. The pyrometer was calibrated on the boiling point of H_2O and the melting points of Sn, Cd, Sb, Zn, and Ag.

SOURCE AND PURITY OF MATERIALS:

Bi: nothing specified.

Na: from Kahlbaum.

ESTIMATED ERROR:

Solubility: nothing specified; not better than ±0.1 mol % Bi (by the compilers).

Temperature: precision ± 1.5 K.

REFERENCES:

1. Kurnakow, N.S.; Kusnetsow, A.N. *Z. Anorg. Chem.* 1900, *23*, 439-462.

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Mathewson, C.H.

Z. Anorg. Chem. 1906, *50*, 171-198.

VARIABLES:

Temperature: 491-1048 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Na-Bi system were determined.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi
700	10.7	553	48.3	430	56.3
755	20.7	507	50.5	370	66.4
775	25.5	495	51.3	218	78.2
750	30.5	446	53.1	248	90.1
675	40.8				

The melting points of Bi and Na were found to be at 273 and 97.5 °C, respectively.

Two equilibrium solid phases were identified: Na_3Bi and NaBi.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Weighed amounts of Bi were gradually introduced into molten Na in a high melting Jena glass tube under H_2 atmosphere. The molten alloys were mixed by means of stirring with a Fe wire. Thermal analytical arrests were taken from cooling curves. A Pt/Pt-Rh thermocouple was used for temperature measurements. It was calibrated on the melting points of Na, Pb, Sn, Zn, and Sb as in (1). The alloys were analyzed by decomposition with H_2O. The formed NaOH was acidimetricly titrated.

SOURCE AND PURITY OF MATERIALS:

Bi: nothing specified.
Na: "pure"; purified by remelting and cutting off the surface (1).

ESTIMATED ERROR:

Nothing specified.
Solubility: precision ± 0.5 mol %, in analogy to (1).
Temperature: precision ± 1 K (by the compilers).

REFERENCES:

1. Mathewson, C.H. *Z. Anorg. Chem.* 1905, *46*, 94-112.

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Foster, M.S.

Argonne National Laboratory, 1968; reported in (1) and (2).

VARIABLES:

Temperature: 398-523 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Bi in liquid Na was determined at various temperatures.

t/°C[a]	*soly*/mol % Bi[a]	t/°C[a]	*soly*/mol % Bi[a]
125	$7.1 \cdot 10^{-5}$; $7.8 \cdot 10^{-5}$	200	$7.84 \cdot 10^{-4}$
150	$2.67 \cdot 10^{-4}$	225	$1.35 \cdot 10^{-3}$
175	$2.29 \cdot 10^{-4}$; $3.31\ 10^{-4}$	250	$2.28 \cdot 10^{-3}$

[a] as numerically reported in (1) and graphically reported in (2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The metals were equilibrated in an appropriate container within a dry Ar glove box. The solutions were stirred and maintained at the selected temperatures for 4 to 24 hours. A sample of the solution was then taken through a fine porosity glass frit. Chemical analyses were performed using atomic absorption spectrometry.

SOURCE AND PURITY OF MATERIALS:

Bi: nothing specified; probably 99.999 % pure as in other studies of this laboratory.
Na: "reagent grade" purity with a content of ~$5.0 \cdot 10^{-3}$ % O.

ESTIMATED ERROR:

Solubility: precision ± 5 %.
Temperature: nothing specified.

REFERENCES:

1. Gehri, D.C.; Sullivan, R.J. *U.S. Atom. Ener. Comm. Rep. AI-AEC-12770*, 1969.
2. Claar, T.D. *Reactor Technol.* 1970, *13*, 124-146.

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Gehri, D.C.; Sullivan, R.J.

U.S. Atom. Ener. Comm. Rep. AI-AEC-12770, 1969.

VARIABLES:

Temperature: 473-1118 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Bi in liquid Na was determined.

t/°C	*soly*/mass % Bi	*soly*/mol % Bi
200[a]	$3.6 \cdot 10^{-3}$	$4.0 \cdot 10^{-4}$
300[a]	$1.5 \cdot 10^{-2}$	$1.7 \cdot 10^{-3}$
400[a]	$2.5 \cdot 10^{-1}$	$2.8 \cdot 10^{-2}$
201.5	$4.2 \cdot 10^{-3}$	$4.6 \cdot 10^{-4}$
203	$3.8 \cdot 10^{-3}$	$4.2 \cdot 10^{-4}$
302	$1.85 \cdot 10^{-3}$	$2.0\ 10^{-3}$
401	0.525	$5.8 \cdot 10^{-2}$
402	0.25	$2.8 \cdot 10^{-2}$
500	2.36	0.26
500.5	2.34	0.26
648.5	13.04	1.62

[a] as reported in (1)

The cooling curve of molten Na_3Bi showed inflections at 845 °C (melting) and at 725 °C (solid phase transition in Na_3Bi).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

$BiNa_3$ solute was used to saturate liquid Na with Bi. It was prepared in a stainless steel foil cup within a stainless steel capsule to which proper amounts of the metals were given under Ar atmosphere. The composition of the solute was determined by analysis. A 150 μm stainless steel screen served to separate the solute from a sample collector. The equilibration was continued for ~20 hours after which the capsule was inverted. The solution saturated with Bi was collected in the sample collector and analyzed for Bi by means of atomic absorption spectroscopy after stepwise dissolution in C_2H_5OH, H_2O, and diluted HNO_3.

SOURCE AND PURITY OF MATERIALS:

Bi: 99.99+ % pure with contents of $<1 \cdot 10^{-3}$ % Fe, Sb, As, Zn; $1 \cdot 10^{-3}$ % Ag+Pb+Cu+Te.
Na: very high purity, hot gettered, distilled and stored in high vacuum, with contents of $9 \cdot 10^{-4}$ % C, $< 3 \cdot 10^{-4}$ % O; $< 1 \cdot 10^{-3}$ % metallic elements.

ESTIMATED ERROR:

Solubility: precision typically ± 1 %.
Temperature: nothing specified.

REFERENCES:

1. McKisson, R.L.; Eichelberger, R.L.; Gehri, D.C.; Guon, J. *US Atom.Ener.Comm. Rep. AI-AEC-12744* 1968, p. 131-138.

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Johnson, C.E.; Fisher, A.K.

J. Less-Common Met. 1970, *20*, 339-344.

VARIABLES:

Temperature: 499.5-1121.3 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus line of the Na-Bi system were determined.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi
650.0	5.0	848.1	25.0	236.2	80.0
714.9	10.0	703.7	35.0	261.3	90.2
751.4	15.0	553.8	43.0		
769.8	17.5	444.4	51.8	840 [c]	25.0
792.6	20.0	441.0	53.0	850.4 [a]	25.0
797.0	20.0	372.1	65.0	820 [b]	22.5
803.0	21.0	249.2	75.0	842 [b]	25.0
820.0	22.5	226.3	78.1	797 [b]	20.0

[a] as reported in (1) [b] from vapour pressure measurements performed in the same laboratory (3)
[c] from thermal analysis reported in (3)

The data which are listed above were graphically reported in (2).
The melting points of Bi and Na were found to be at 271.3 and 97.8 °C, respectively.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared under He atmosphere from weighed amounts of the metals inside of a 347 stainless steel sample capsule which was evacuated and placed in an apparatus for differential thermal analysis. Pt/Pt-Rh(10%) thermocouples which were calibrated on the melting points of Zn and Al (certified by the NBS) were used to record the heating and cooling curves. The experiments with Bi-rich alloys were performed in an open container. An apparatus for vapour pressure measurements (3) was made of 316 stainless steel (which was insignificantly attacked by the alloy). The boiling point method was applied. The liquidus data were obtained from break points on the plots relating the total vapour pressure with the reciprocal temperature.

SOURCE AND PURITY OF MATERIALS:

Bi: 99.999 % pure from United Mineral and Chem. Corp., further melted and filtered.
Na: reagent grade pure from B & A, surface layer was removed.
He: contents of $5 \cdot 10^{-5}$ % H_2O and $< 1 \cdot 10^{-3}$ % $O_2 + N_2$.

ESTIMATED ERROR:

Solubility: precision ± 0.05 mol %.
Temperature: accuracy ± 0.4 K.

REFERENCES:

1. Fredrickson, D.R.; Chasanov, M.G.; Barnes, R.D.; Johnson, S.A. *High Temp. Sci.* 1970, *2*, 259-264.
2. Meyer, R.J.; Johnson, C.E.; Ross, L.E. *US Atom.Ener.Comm. Rep. ANL-7520*, Pt.I, 1969, p. 291-294.
3. Fisher, A.K.; Johnson, S.A.; Wood, S.E. *J. Phys. Chem.* 1967, *71*, 1465-1472.

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Walker, R.A.; Pratt, J.N.

J. Nucl. Mater. 1970, *34*, 165-173.

VARIABLES:

Temperature: 525.7-836.2 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The solubility of Bi in liquid Na was determined at various temperatures.

t/°C	*soly*/mol % Bi	t/°C	*soly*/mol % Bi
563.0	0.911	402.0	$3.31 \cdot 10^{-2}$
514.0	1.240	370.0	$3.98 \cdot 10^{-2}$
493.0	0.368	332.0	$1.49\ 10^{-2}$
475.0	0.137	292.5	$5.5 \cdot 10^{-3}$
434.0	$8.98\ 10^{-2}$	252.5	$9 \cdot 10^{-4}$

The data were approximated by the fitting equation (tested by the compilers):

$$\log(soly/\text{mol \% Bi}) = (5.0045 \pm 0.4770) - (4189.0 \pm 317.1)(T/\text{K})^{-1}$$

The solid equilibrium phase Na_3Bi was identified.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Na was equilibrated with an excess of Bi in a Ni crucible which was heated inside of a dry Ar box and stirred with a Ni rod. The system was equilibrated 12 hours. The solution was not mixed during the last hour of the equilibration in order to allow a sedimentation of the excess of Bi. Samples were taken at the equilibration temperatures by means of small sampler cups. The samples were removed from the dry box and dissolved in C_2H_5OH. The solution was analyzed for Bi by means of a gravimetric technique involving the formation of $BiPO_4$.

SOURCE AND PURITY OF MATERIALS:

Bi: 99,9993+ % pure, from Matthey Johnson & Co.
Na: "high" purity, from UKAEA, Harwell, distilled, with a content of $1.6 \cdot 10^{-3}$ % O.
Ar: purified.

ESTIMATED ERROR:

Solubility: standard deviation ± 9 %.
Temperature: nothing specified.

REFERENCES:

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Yih, T.S.; Thompson, J.C.

J. Phys., F 1982, *12*, 1625-1636.

VARIABLES:

Temperature: 502-731 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Several points on the liquidus of the Na-Bi system were determined.

t/°C	*soly*/mol % Bi	t/°C	*soly*/mol % Bi
458	52.00	310	73.00
447	54.00	258	78.76
428	58.00	244	80.95
394	63.00	229	87.57
356	68.00	267	91.73

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The potential differences between the alloy and the reference Na were measured by means of the electrochemical cell: Bi-Na / Na^+ in ß-alumina / Na. The insulators were made of ceramics (MACOR), the container was of stainless steel. The measurements were performed at constant composition under variation of the temperature. Changes of the composition were carried out by electrolysis through the cell at constant current. Cooling and heating curves were recorded. The solubility data were evaluated from the break points of the curves of potential vs. temperature.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Solubility: accuracy ± 0.1 mol %.

Temperature: precision: ± 1 K.

REFERENCES:

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

EVALUATOR:

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

CRITICAL EVALUATION:

The K-Bi phase diagram was experimentally investigated in three laboratories (1,2,3). The liquidus data of Petric et al. (3) are in some ranges of composition up to 30 K higher than in the earlier studies (1) and (2). The results of (3) are preferable due to the highest purity of the materials used in this study, while the results of (1) and (2) may be influenced by volatization of K under their experimental conditions.
The solubility of Bi in liquid K was investigated in (3); (1) reported two points in the range of higher Bi compositions. The data of (3) are considered to be reliable, while the two points reported in (1) may be erroneous. The results of the solubility measurements of (3) were approximated by fitting equation by the compilers:

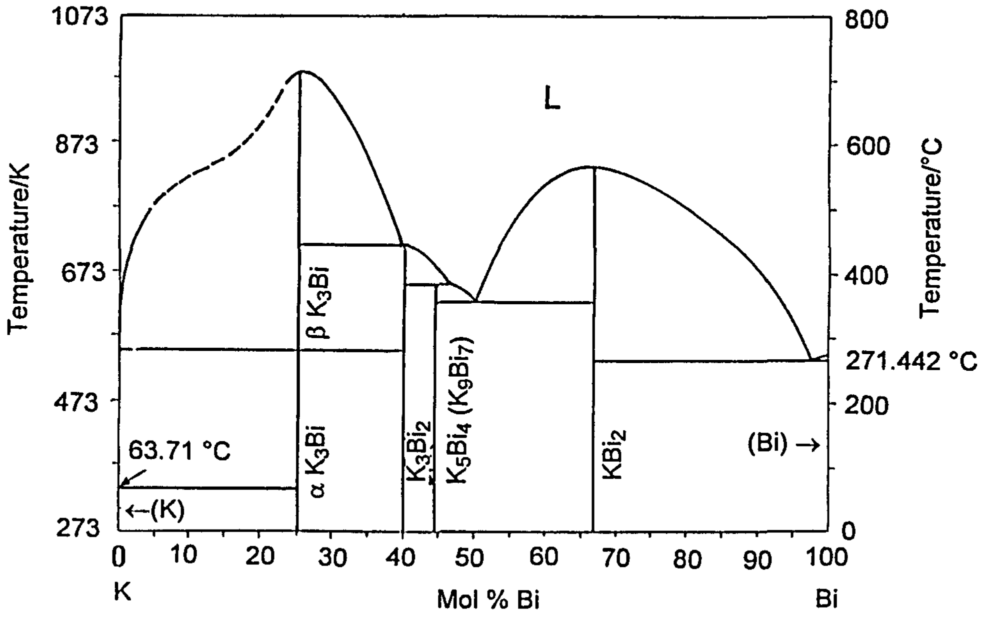
$$\log(soly/\text{mol \% Bi}) = 4.867 - 3290(T/\text{K})^{-1} \qquad r = 0.9988 \qquad \text{Eq.(1)}$$

The equation is valid in the temperature range 608 to 758 K and appears to be reasonably applicable at lower temperatures. The solubility equation which was reported in (4) does not fit precisely the data reported in (3). The schematic K-Bi phase diagram is redrawn from (4).

Tentative (t) and doubtful (d) values of the solubility of Bi in liquid K.

T/K	*soly*/mol % Bi	source
573	0.1 (t)	Eq.(1)
673	0.9 (t)	(3); interpolated
773	4 (d)	Eq.(1)
873	18 (d)	(1)
988	25 (d) congruent	(3); extrapolated

The two liquid metals are completely miscible at temperatures above 988 ± 25 K.



References

1. Smith, D.P. *Z. Anorg. Chem.* 1908, *56*, 109.
2. Gnutzmann, G.; Klemm, W. *Z. Anorg. Chem.* 1961, *309*, 181.
3. Petric, A.; Pelton, A.D.; Saboungi, M.-L. *J. Phys., F* 1988, *18*, 1473.
4. Petric, A.; Pelton, A.D. *J. Phase Equil.* 1991, *12*, 29.

COMPONENTS: (1) Bismuth; Bi; [7440-69-9] (2) Potassium; K; [7440-09-7]	**ORIGINAL MEASUREMENTS:** Smith, D.P. *Z. Anorg. Chem.* 1908, *56*, 109-142.
VARIABLES: Temperature: 541-944 K	**PREPARED BY:** H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the K-Bi system was partially determined.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi
268	99.0	552	65.0	412	42.0
279	97.0	543	60.0	420	40.0
379	90.0	513	55.0	422	35.0
475	80.0	441	53.0	617	30.0
516	75.0	341	50.0 eutectic	671	25.0 congruent
542	70.0	366	47.0	628	20.0
538	68.0	384	45.0	606	18.0

The melting points of K and Bi were determined to be at 63 and 269 °C, respectively.
Two equilibrium solid phases were identified: KBi_2 and K_3Bi.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The oxidized surface of metallic K was cut off, the metal was then washed in benzene, amyl alcohol, petroleum and ether. Both metals were weighed (K under petroleum) and placed in an container made of Fe (for K-rich alloys) or hard Jena glass (for Bi-rich alloys). The containers were filled with H_2. Only small portions of Bi were added into the melt because of the strongly exothermic effect of mixing. The thermal analyses were performed by means of the determination of cooling curves with the aid of a calibrated Pt/Pt-Rh thermocouple.

SOURCE AND PURITY OF MATERIALS:

Bi: nothing specified.
K: did not contain traces of Na or of other metals.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Gnutzmann, G.; Klemm, W.

Z. Anorg. Chem. 1961, *309*, 181-188.

VARIABLES:

Temperature: 623-905 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the K-Bi system was determined; the data were read out from the figure by the compilers.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi
632	29.0	421	42.9	360	48.8
597	31.7	413	43.9	350	49.8
467	38.3	402	44.9	413	51.6
441	40.6	375	46.6	565	66.6 congruent
432	41.8	370	47.8	504	79.2

The melting points of K and Bi were determined to be at 63.4 and 271 °C, respectively.
Four equilibrium solid phases were identified: KBi_2, K_5Bi_4, K_3Bi_2, and K_3Bi.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by melting of the components in a corund crucible. This was placed in a quartz apparatus in an Ar atmosphere. Thermal analyses of the samples were performed, the temperatures were measured by means of a Pt/Pt-Rh thermocouple which was calibrated on the melting points of Sn, Cd, Zn, NaCl, and Ag. The composition of the alloys was analyzed after the dissolution of the alloys in HNO_3 at 195 K in a capsule which was hermetically closed. Bi was gravimetrically determined as $BiPO_4$, and K as sulphate.

SOURCE AND PURITY OF MATERIALS:

Bi: analytical grade, from Merck.
K: "commercial", purified by means of high vacuum distillation.
Ar: "purified".

ESTIMATED ERROR:

Solubility: nothing specified; read-out procedure ± 0.3 mol %.
Temperature: reproducibility ± 2 K; read-out procedure ± 3 K.

REFERENCES:

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Petric, A.; Pelton, A.D.; Saboungi, M.-L.

J. Phys., F 1988, *18*, 1473-1489.

VARIABLES:

Temperature: 608-952 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the K-Bi system was determined.

t/°C	*soly*/mol % Bi	t/°C	*soly*/mol % Bi	t/°C	*soly*/mol % Bi
383	92	532	58.8	485	3.4
433	88	438	42.1	463	2.5
445	86.5	457	39.1	443	1.8
473	83.7	472	38.7	412	1.1
488	82	479	38.6	391	0.8
543	60	495	38	374	0.6
540	59.6	600	33.9	356	0.4
536	59.2	679	28.9	335	0.3

Four equilibrium solid phases were identified: K_3Bi, K_3Bi_2, K_5Bi_4, and KBi_2.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus was enclosed in a He-filled glove box. Potential differences of the two cell assembly were measured: Bi-K(12%)/K β-alumina/Pb-K(2%)/K β-alumina/Bi-K. K β-alumina was prepared by the exchange reaction of Na β-alumina with KCl at 1273 K for 48 h and with KNO_3 at 633 K for 48 h. The completeness of the reaction was checked by atomic absorption spectroscopy. The Pb-K(2%) alloy was contained in a Mo crucible, the reference and working alloys in the K β-alumina tubes. Wires were made of Ta. The assembly was sealed with Corning 1720 glass. The cells were loaded with the metals inside the glove box. The K concentrations were varied by means of coulometric titration. The emf's were measured after the electrolysis was interrupted. The liquidus data were received from break points of the emf vs. temperature curves.

SOURCE AND PURITY OF MATERIALS:

Bi: 99.95 % pure.
K: 99.9 % pure with 0.015 % Rb and 0.005 % Na, supplied by Callery Chemical Corpor.

ESTIMATED ERROR:

Nothing specified.
Solubility: precision ± 7 % up to 3.4 mol % Bi (by the compilers).
Temperature: precision ± 3 K (by the compilers).

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Bismuth; Bi; [7440-69-9] (2) Rubidium; Rb; [7440-17-7]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1992

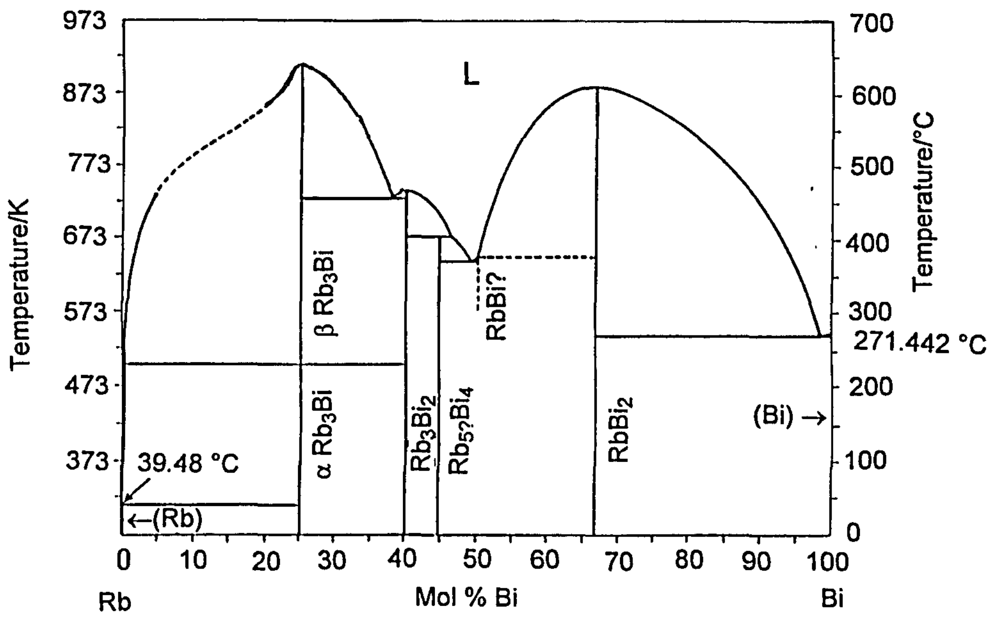
CRITICAL EVALUATION:

The Rb-Bi phase diagram was experimentally investigated in several laboratories (1,2,3,4). The results of Mingazin (1) may be treated as preliminary; the melting point of $RbBi_2$ was determined at 923 K. The studies of (1) were extended by Zhuravlev et al. (2) who roughly determined the complete liquidus line of the Rb-Bi system. Gnutzmann and Klemm (3) reported a detailed study of the liquidus, and observed different liquidus temperatures up to 80 K compared to the data of (2); the melting point of $RbBi_2$ was found by (3) to be about 50 K lower than that found by (1). The concentration range between 40 and 50 mol % Bi was intensively studied by (3), and the peritectic formation of the compounds Rb_3Bi_2 and Rb_6Bi_4 was indicated. Petric et al. (4) confirmed the liquidus line of (3) except the Bi-rich range in which (4) recorded up to 40 K higher liquidus temperatures than (3). The phase diagram of (4) may be accepted as the best one, and is redrawn below.
The solubility of Bi in liquid Rb was determined by (4). The results at higher concentrations of Bi (18 to 25 mol % Bi) which were reported by (2), (3), and (4) are scattered within ± 30 K. The data of (3) and (4) are preferred.

Tentative (t) and doubtful (d) values of the solubility of Bi in liquid Rb

T/K	*soly*/mol % Bi	source
623	0.8 (t)	(4)
673	1.6 (t)	(4) interpolated
773	10 (d)	(3) and (4) interpolated
873	23 (t)	(4)
915	25 (t) congruent	(2), (3)

The two metals are completely miscible above 915 K.



References

1. Mingazin, T.A. *Izv. Akad. Nauk Turkmen. SSR* 1958, *no.1*, 91
2. Zhuravlev, N.N.; Mingazin, T.A.; Zhdanov, G.S. *Zh. Eksper. Teoret. Fiz.* 1958, *34*, 820.
3. Gnutzmann, G.; Klemm, W. *Z. Anorg. Chem.* 1961, *309*, 181.
4. Petric, A.; Pelton, A.D.; Saboungi, M.-L. *J. Electrochem. Soc.* 1988, *135*, 2754.

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Zhuravlev, N.N.; Mingazin, T.A.; Zhdanov, G.S.

Zh. Eksper. Teoret. Fiz. 1958, *34*, 820-826.

VARIABLES:

Temperature: 628-935 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

Some points on the liquidus line of the Rb-Bi system were determined; the data were read out from the figure by the compilers.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi
585	18.0	454	39.5	662	63.6
600	20.0	392	41.0	660	66.7
640	25.0 congruent	380	43.2	635	70.0
620	29.0	355	45.4	585	70.7
577	34.2	583	53.2	450	80.5

Two equilibrium solid phases were identified, $RbBi_2$ and Rb_3Bi, and the formation of Rb_2Bi and Rb_3Bi_2 was suggested.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared from the metals in quartz ampoules. A Fe crucible was placed in this ampoule for the preparation of Rb-rich alloys. Cooling and heating curves were recorded after melting an alloy sample in a small furnace. The composition of the alloys was confirmed by chemical analyses. The experiments were performed in an inert atmosphere.

SOURCE AND PURITY OF MATERIALS:

Bi: "chemically pure".
Rb: (i) "chemically pure" and (ii) containing 3 % K; the results were not influenced by the different grades of purity.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.5 mol %.
Temperature: read-out procedure ± 5 K.

REFERENCES:

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Gnutzmann, G.; Klemm, W.

Z. Anorg. Chem. 1961, *309*, 181-188.

VARIABLES:

Temperature: 573-915 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Rb-Bi system was determined; the data were read-out from the figure by the compilers.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi
532	18.0	452	41.0	488	53.9
642	25.0 congruent	448	42.2	597	63.7
614	29.5	435	42.9	605	66.7 congruent
576	31.9	431	43.3	469	84.4
555	33.2	400	45.7	378	91.7
506	35.8	381	48.2	352	93.9
464	39.0	356	49.6	300	96.3
450	40.0				

The melting points of Rb and Bi were determined to be at 38.8 and 271 °C, respectively.
Four equilibrium solid phases were identified: $RbBi_2$, Rb_5Bi_4, Rb_3Bi_2, and Rb_3Bi.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by melting of the components in a corund crucible. This was placed in a quartz apparatus in an Ar atmosphere. Thermal analyses of the samples were performed, the temperatures were measured by means of a Pt/Pt-Rh thermocouple which was calibrated on the melting points of Sn, Cd, Zn, NaCl, and Ag. The composition of the alloys was analyzed after the dissolution of the alloys in HNO_3 at 195 K in a capsule which was hermetically closed. Bi was gravimetrically determined as $BiPO_4$, and Rb as sulphate.

SOURCE AND PURITY OF MATERIALS:

Bi: analytical grade, from Merck.
Rb: obtained by reduction of RbCl with Ca at about 973 K in vacuum; the metallic product was vacuum distilled.
Ar: "purified".

ESTIMATED ERROR:

Solubility: nothing specified; read-out procedure ± 0.3 mol %.
Temperature: reproducibility ± 2 K; read-out procedure ± 3 K.

REFERENCES:

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Petric, A.; Pelton, A.D.; Saboungi, M.-L.

J. Electrochem. Soc. 1988, *135*, 2754-2760.

VARIABLES:

Temperature: 598-873 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus of the Rb-Bi system was determined.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi
325	97	375	48.8	600	29.6
450	90	385	48	600	23
534	82	390	47.5	446	3.9
600	71.8	397	46.8	427	2.7
600	62.6	403	46.2	409	1.9
536	56	409	46	389	1.2
494	54	419	45.5	374	0.9
415	51	460	42	356	0.8
376	50	470	40		
370	49 eutectic	455	39		

Rb_3Bi, Rb_3Bi_2, Rb_6Bi_4, and $RbBi_2$ were identified as solid equilibrium phases, the formation of RbBi was suggested.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The apparatus was enclosed in a He-filled glove box. Potential differences of the two cell assembly were measured: Bi-Rb(3%)/Rb β'-alumina/Bi-Rb(2%)/Rb β'-alumina/Bi-Rb. Rb β'-alumina was prepared by the exchange reaction of Na β'-alumina with RbCl at 1273 K for 48 h. The reaction resulted in an exchange of Rb of 94-96 %. The Bi-Rb(2%) alloy was contained in a Mo crucible, the reference and working alloys in the Rb β'-alumina tubes. Wires were made of Ta. The assembly was sealed with fiber glass which was modified by addition of 3 % BaO. The cells were loaded with the alloys inside the glove box. The Rb concentrations were varied by means of coulometric titration. The emf's were measured after the electrolysis was interrupted. The liquidus data were received from break points of the emf vs. temperature curves.

SOURCE AND PURITY OF MATERIALS:

Bi: 99.95 % pure.
Rb: 99.85 % pure with 0.1 % Cs, other impurities in the 10^{-4} % level, supplied by Callery Chemical Corp.

ESTIMATED ERROR:

Solubility: precision ± 0.3 mol % for Bi-rich alloys, a few % for diluted alloys.
Temperature: precision ± 4 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Bismuth; Bi; [7440-69-9] (2) Cesium; Cs; [7440-46-2]	H.U. Borgstedt, Kernforschungszentrum Karlsruhe, Germany C. Guminski, Dept. of Chemistry, Univ. of Warsaw, Poland February 1992

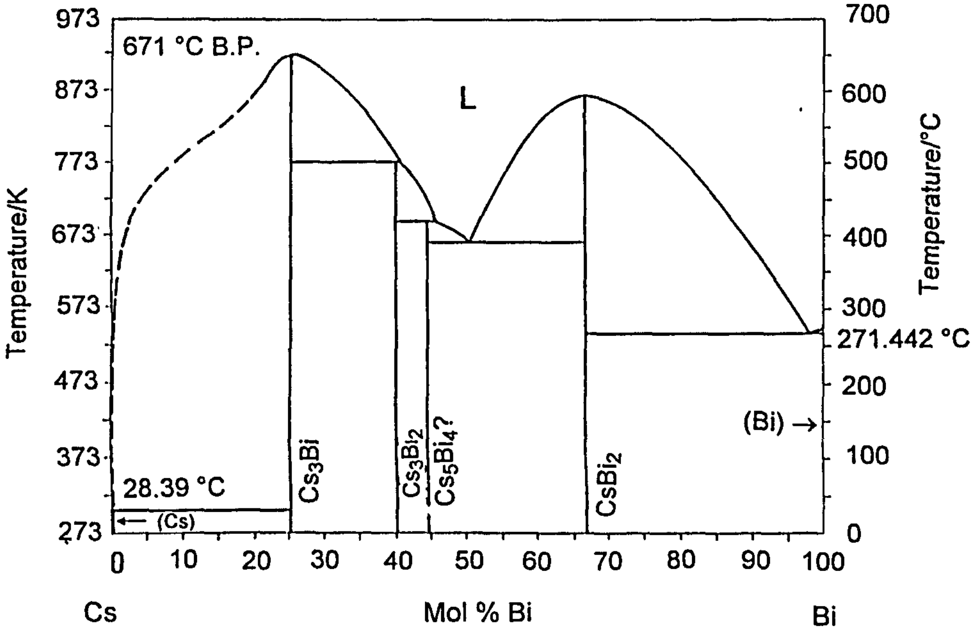
CRITICAL EVALUATION:

The liquidus of the Cs-Bi phase diagram is only known in the range of Bi concentrations above 21.5 mol % Bi (1,2). Therefore, the solubility of Bi in liquid Cs can only be suggested for temperatures which are higher than 868 K. Sangster and Pelton (3) estimated a solubility of 1 mol % Bi at 673 K and less than $1 \cdot 10^{-4}$ mol % Bi at the melting point of Cs. The liquidus data which were obtained by Gnutzmann and Klemm (2) reflected the formation of four intermetallic compounds in the system. These data are, thus, more convincing than those which were reported by Zhuravlev, who identified only two congruently melting phases, $CsBi_2$ and Cs_3Bi. The discrepancy between the liquidus data of the two sources reaches up to 60 K.
The schematic Cs-Bi phase diagram is based on (2) and (3), the probable liquidus of the Cs-rich alloys is outlined by a dashed line in analogy to the K-Bi and Rb-Bi systems.

Tentative values of the solubility of Bi in liquid Cs

T/K	*soly*/mol % Bi	source
873	22	(2)
908	25 congruent	(2)

The two liquid metals are completely miscible at temperatures above 908 K.



References

1. Zhuravlev, N.N. *Zh. Eksper. Teoret. Fiz.* 1958, *34*, 827.
2. Gnutzmann, G.; Klemm, W. *Z. Anorg. Chem.* 1961, *309*, 181.
3. Sangster, J.; Pelton, A.D. *J.Phase Equil.* 1991, *12*, 443.

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Zhuravlev, N.N.

Zh. Eksper. Teoret. Fiz. 1958, *34*, 827-829.

VARIABLES:

Temperature: 523-903 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Cs-Bi system was determined; the data were read out from the figure by the compilers.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi
657	21.8	605	59.7	440	78.3
590	30.6	620	63.2	425	83.7
490	38.9	630	66.8	358	89.4
390	43.7	603	70.5	330	92.3
553	54.1	480	73.1	250	96.9

Two equilibrium solid phases were identified, $CsBi_2$ and Cs_3Bi, the formation of Cs_2Bi was suggested.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared from the metals in quartz ampoules. A Fe crucible was placed in this ampoule for the preparation of Cs-rich alloys. Cooling and heating curves were recorded after melting an alloy sample in a small furnace. The composition of the alloys was confirmed by chemical analyses. The experiments were performed in an inert atmosphere.

SOURCE AND PURITY OF MATERIALS:

Bi: 99.99 % pure.
Cs: 99.99 % pure with Ca as the main impurity.

ESTIMATED ERROR:

Nothing specified.
Solubility: read-out procedure ± 0.5 mol %.
Temperature: read-out procedure ± 5 K.

REFERENCES:

COMPONENTS:

(1) Bismuth; Bi; [7440-69-9]

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

ORIGINAL MEASUREMENTS:

Gnutzmann, G.; Klemm, W.

Z. Anorg. Chem. 1961, *309*, 181-188.

VARIABLES:

Temperature: 583-908 K

PREPARED BY:

H.U. Borgstedt and C. Guminski

EXPERIMENTAL VALUES:

The liquidus line of the Cs-Bi system was determined; the data were read out from the figure by the compilers.

t/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi	*t*/°C	*soly*/mol % Bi
595	21.5	499	40.6	589	67.7
635	25.0 congruent	489	41.5	566	72.4
627	26.5	464	43.3	532	76.3
620	28.7	419	44.6	486	80.5
590	31.7	409	46.3	456	83.5
553	34.9	387	48.5	409	86.6
545	36.0	381	49.6	393	89.1
500	38.4	491	54.4	310	94.5
		590	64.6		

The melting points of Cs and Bi were determined to be at 28.9 and 271 °C, respectively.
Four equilibrium solid phases were identified: $CsBi_2$, Cs_5Bi_4, Cs_3Bi_2, and Cs_3Bi.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The alloys were prepared by melting of the components in a corund crucible. This was placed in a quartz apparatus in an Ar atmosphere. Thermal analyses of the samples were performed, the temperatures were measured by means of a Pt/Pt-Rh thermocouple which was calibrated on the melting points of Sn, Cd, Zn, NaCl, and Ag. The composition of the alloys was analyzed after the dissolution of the alloys in HNO_3 at 195 K in a capsule which was hermetically closed. Bi was gravimetrically determined as $BiPO_4$, and Cs as sulphate.

SOURCE AND PURITY OF MATERIALS:

Bi: analytical grade, from Merck.
Cs: obtained by reduction of CsCl with Ca at about 973 K in vacuum; the metallic product was vacuum distilled.
Ar: "purified".

ESTIMATED ERROR:

Solubility: nothing specified; read-out procedure ± 0.3 mol %.
Temperature: reproducibility ± 2 K; read-out procedure ± 3 K.

REFERENCES:

SOLUBILITY DATA SERIES

Volume 1	H.L. Clever, *Helium and Neon*
Volume 2	H.L. Clever, *Krypton, Xenon and Radon*
Volume 3	M. Salomon, *Silver Azide, Cyanide, Cyanamides, Cyanate, Selenocyanate and Thiocyanate*
Volume 4	H.L. Clever, *Argon*
Volume 5/6	C.L. Young, *Hydrogen and Deuterium*
Volume 7	R. Battino, *Oxygen and Ozone*
Volume 8	C.L. Young, *Oxides of Nitrogen*
Volume 9	W. Hayduk, *Ethane*
Volume 10	R. Battino, *Nitrogen and Air*
Volume 11	B. Scrosati and C.A. Vincent, *Alkali Metal, Alkaline Earth Metal and Ammonium Halides, Amide Solvents*
Volume 12	C.L. Young, *Sulfur Dioxide, Chlorine, Fluorine and Chlorine Oxides*
Vollume 13	13 S. Siekierski, T. Mioduski and M. Salomon, *Scandium, Yttrium, Lanthanum and Lanthanide Nitrates*
Volume 14	H. Miyamoto, M. Salomon and H.L. Clever, *Alkaline Earth Metal Halates*
Volume 15	A.F.M. Barton, *Alcohols with Water*
Volume 16/17	E. Tomlinson and A. Regosch, Antibiotics: 1,β-Lactam Antibiotics
Volume 18	O. Popovych, *Tetraphenylborates*
Volume 19	C.L. Young, *Cumulative Index: Volumes 1-18*
Volume 20	A.L. Horvath and F.W. Getzen, *Halogenated Benzenes, Toluenes and Phenols with Water*
Volume 21	C.L. Young and P.G.T. Fogg, *Ammonia, Amines, Phosphine, Arsine, Stibine, Silane, Germane and Stannane in Organic Solvents*
Volume 22	T. Mioduski and M. Salomon, *Scandium, Yttrium, Lanthanum and Lanthanide Halides in Non-aqueous Solvents*
Volume 23	T.P. Dirkse, *Copper, Silver, Gold and Zinc, Cadmium, Mercury Oxides and Hydroxides*
Volume 24	W. Hayduk, *Propane, Butane and 2-Methylpropane*
Volume 25	C. Hirayama, Z. Galus and C. Guminski, *Metals in Mercury*
Volume 26	M.R. Masson, H.D. Lutz and B. Engelen, *Sulfites, Selenites and Tellurites*
Volume 27/28	H.L. Clever and C.L. Young, *Methane*
Volume 29	H.L. Clever, *Mercury in Liquids, Compressed Gases, Molten Salts and Other Elements*
Volume 30	H. Miyamoto and M. Salomon, *Alkali Metal Halates, Ammonium Iodate and Iodic Acid*
Volume 31	J. Eysseltova and T.P. Dirkse, *Alkali Metal Orthophosphates*
Volume 32	P.G.T. Fogg and C.L. Young, *Hydrogen Sulfide, Deuterium Sulfide and Hydrogen Selenide*
Volume 33	P. Franzosini, *Molten Alkali Metal Alkanoates*
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*
Volume 65	J.J. Fritz and E. Königsberger, *Copper(I) Halides and Pseudohalides*