COMPONENTS:	EVALUATOR:
(1) Thorium; Th; [7440-29-1] (2) Mercury; Hg; [7439-97-6]	C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION:

Messing and Dean (1) found that the solubility of thorium in mercury increased from 1.82×10^{-3} to 2.55×10^{-2} at % in the temperature range of 313 to 629 K. Jangg and Palman (2) determined thorium solubilities ranging from 1.3×10^{-3} to 3.5×10^{-2} at % at 293 to 673 K. The solubilities reported by (1) and (2) are similar, and in the opinion of the evaluators these are the most accurate data; both groups of workers employed equilibration, filtration, and chemical analyses of the amalgams for their solubility determinations. Room temperature determinations reported by other workers, 7×10^{-3} (3) and 1.36×10^{-2} at % (4) at 298 K, are rejected because they are much higher than those determined by (1) and (2). Much higher solubilities were obtained by Domagala and coworkers (5) who reported 0.53 to 4.8 at % in the temperature range of 337 to 571 K. Kozin's (6) predicted value of 7.3×10^{-5} at % at 298 K is much too low.

The saturated thorium amalgams are in equilibrium with the compounds $ThHg_3$, $ThHg_2$ and ThHg which are stable up to 773, 860 and 920 K, respectively (5,7).

The solubility of thorium in saturated uranium amalgam has been reported to be approximately one-half that in mercury (1).

Tentative values of the solubility of Th in Hg:

T/K	Soly/at %	Reference
293	1.3×10^{-3}	[2]
298	1.5×10^{-3} [a]	[1,2]
323	2.3×10^{-3} [a]	[1,2]
373	4.6×10^{-3} [a]	[1,2]
473	1.2×10^{-2} [a]	[1,2]
573	2.1×10^{-2} [a]	[1,2]
673	3.2×10^{-2} [a]	[1,2]

[a] Interpolated value from data of (1) and (2).

References

1. Messing, A.F.; Dean, O.C. *U.S. At. Ener. Comm. Rep., ORNL-2871*, 1960.
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6. Kozin, L.F. *Fiziko-Khimicheskie Osnovy Amalgamnoi Metallurgii*, Nauka, Alma-Ata, 1964.
7. Jangg, G.; Steppan, F. *Z. Metallk.* 1965, *56*, 172.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thorium; Th; [7440-29-1] (2) Mercury; Hg; [7439-97-6]	Messing, A.F.; Dean, O.C. *U.S. At. Ener. Comm. Rep., ORNL-2871,* 1960.
VARIABLES: Temperature: 40-356°C	**PREPARED BY:** C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of thorium in mercury.

t/°C	Soly/mass %	Soly/at %
40	0.00211	0.00182
60	0.00313	0.00270
120	0.00675	0.00583
160	0.00921	0.00790
200	0.0120	0.0104
220	0.0151	0.0130
280	0.0203	0.0175
300	0.0235	0.0203
356	0.0295	0.0255

The authors observed that the solubility of thorium in saturated uranium amalgam is approximately one-half that in pure mercury.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Mercury and thorium, after drying and outgassing in the stainless steel dissolver, were kept for several days at the desired temperature. After equilibration, a sample of liquid amalgam was forced through the filter. The sample was collected, dissolved in nitric acid, and analyzed for thorium and mercury.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: standard deviation in fitted equation is 0.02046.

Temp: nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thorium; Th; [7440-29-1] (2) Mercury; Hg; [7439-97-6]	Jangg, G.; Palman, H. *Z. Metallk.* 1963, *54*, 364-9.
VARIABLES: Temperature: 20-400°C	**PREPARED BY:** C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of thorium is presented graphically as a function of temperature. The data points on the curve were read off by the compilers:

t/°C	Soly/10^3 at %
20	1.3
50	2.8
100	4.5
150	8.1
200	10
250	17
300	20
350	28
400	35

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The heterogeneous amalgam was introduced into a specially constructed apparatus made of refractory chromium steel. Such steel apparatus could be used because the solubility of iron in mercury is very low and the chromium (III) oxide film inhibits the wetting of the steel by mercury. After twelve hours of equilibration at the temperature of the experiment, the amalgam was filtered through the sintered iron-frit under the pressure of purified nitrogen. Usually, 3- to 4-fold filtration was necessary. The metal content was then analytically determined in the filtered saturated amalgam. For experiments carried out below 320°C, amalgam was equilibrated in a glass vessel. The analytical procedure is not described in the paper.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: precision ± 5%.

Temp: precision ± 2 K.

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Uranium; U; [7440-61-1] (2) Mercury; Hg; [7439-97-6]	C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION:

There have been numerous reports on the solubility determination of uranium in mercury, many of which in more recent years have been from laboratories associated with the processing of nuclear fuels. However, a number of the determinations, especially near room temperature, are either too low or too high. Tammann and Hinnüber (1) reported a solubility of 1.1×10^{-4} at % at 291 K, while several workers only reported the upper limit of 1×10^{-3} at % at room temperature (2,17,22,23,24); these results are all too low and are rejected. Chang and coworkers (12,21) reported solubilities as high as 6.2×10^{-2} at % at room temperature, and these high values are rejected. At higher temperatures, Magel and Dallas (9) obtained a solubility of 0.1 at % at 348 and 536 K; these results also are rejected because of the lack of experimental details. Kozin's (6) predicted solubility of 3.5×10^{-4} at % at 298 K is too low because his equation neglected the U-Hg interactions.

Ahmann and coworkers (3) reported solubilities of uranium at five temperatures between 298 and 623 K, of which the values at 373, 573 and 623 K are acceptable. Jangg and Palman (4) and Messing and Dean (5) employed similar methods for the equilibration and chemical analysis of the amalgams to determine the uranium solubilities over a wide temperature range. The solubilities determined by (4) at 293 to 540 K increased from 4.2×10^{-3} to 0.33 at %, while those determined by (5) at 313 to 629 K increased from 5.6×10^{-3} to 1.02 at % at increasing temperatures. The results of (4) and (5) are in good agreement and are considered by the evaluators to be the most accurate.

Kobayashi and coworkers (8,20) reported an acceptable solubility of 3.7×10^{-3} at % at room temperature, but an earlier determination (19) of 1.8×10^{-2} at % was too high and is rejected. Schweitzer (28) determined the solubilities at 296 to 526 K, with end values of 4.2×10^{-3} and 3.2×10^{-2} at % in this temperature range. Although the solubilities at both ends of the temperature range are acceptable, those at intermediate temperatures are up to 30% too low, and no experimental details are known to the evaluators. Ettmayer and Jangg (27) reported a solubility of 0.6 at % at 573 K. Forsberg (15), from vapor pressure measurements, reported an upper limit of 1.1 at % for the solubility of uranium at 630 K. Wymer (16) estimated a solubility of 0.94 at % at 630 K, while Morrison and Blanco (17,25), without giving details, reported 0.85 at % at the same temperature. Dean and coworkers (23,24) estimated that the saturated amalgam contains 0.95 at % at 630 K and at least 19 at % at 873 K and 23 atm.

Frost (7) presented a complete phase diagram, but the most recent work of Lee and coworkers (11) has shown that the phase diagram presented by Frost is incorrect. Also, the solubilities taken from the liquidus of Frost's phase diagram are of an order of magnitude too high at 373 and 628 K. The error in the work of (7) may be attributed to an incomplete dehydrogenation of the uranium which was used, and to the possible reaction of the amalgam with nitrogen and the quartz container. Moreover, the investigation of Forsberg (15) and Lee et al. (11) showed a strong influence of pressure on the decomposition temperature of the U-Hg solid phases. Based on a thermodynamic analysis of this system, Lee (26) predicted another version of the U-Hg phase diagram with congruent melting of UHg_2 at 913 K and eutectic point at 748 K for 65 at % U. However, thermal analysis experiments of Lee et al. (11) did not confirm the prediction. The determined points on the liquidus reach a value of 10.0 at % U at 1118 K.

Although there have been several empirical equations fitted to the solubilities as a function of the temperature (5,11,14,28), there appears to be relatively poor agreement among these equations. This system needs further work in the composition range of 33 to 100 at % U. The saturated uranium amalgams are in equilibrium with U-Hg solid intermetallic compounds (3,7,10,11,13,26), as shown in the phase diagram, Fig. 1, reported by (11).

Kinetics of U dissolution in Hg and the saturated amalgam was investigated in (17,18,23,24).

Addition of Mg or Bi increases, and addition of Na or Th decreases, the uranium solubility in mercury (5,23,24).

(Continued next page)

COMPONENTS:	EVALUATOR:
(1) Uranium; U; [7440-61-1] (2) Mercury; Hg; [7439-97-6]	C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION: (Continued)

Tentative values of the solubility of U in Hg:

T/K	Soly/at %	Reference
293	4.0×10^{-3}	[4,8,20,28]
323	9×10^{-3}	[4,5]
373	2.5×10^{-2}	[3,5]
473	0.17	[4]
573	0.5	[3,5,27]
673	1.5[a]	[5,11,15]
773	2.5[a]	[11]
873	4[a]	[11]
973	6[a]	[11]
1073	8[a]	[11]

[a] Solubility obtained by interpolation of data in cited references.

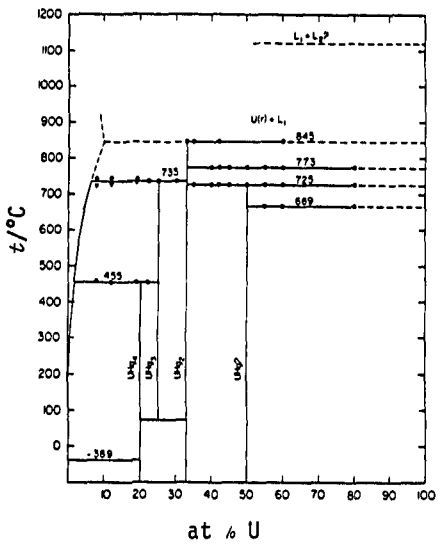

Fig. 1. U-Hg phase diagram under constrained vapor (11).

COMPONENTS:	EVALUATOR:
(1) Uranium; U; [7440-61-1] (2) Mercury; Hg; [7439-97-6]	C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION:

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4. Jangg, G.; Palman, H. *Z. Metallk.* 1963, *54*, 364.
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12. Yu, T.L.; Lee, Y.S.; Chuang, Y.D.; Chang, C.T. *J. Nucl. Sci. Technol.* 1979, *16*, 508.
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17. Morrison, B.H.; Blanco, R.E. *U.S. At. Ener. Comm. Rep., CF-56-1-151*, 1956.
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21. Lee, H.C.; Wang, L.C.; Hung, H.H.; Chang, C.T. *J. Chem. Soc., Chem. Commun.* 1975, 124.
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25. Blanco, R.E. *Nucl. Sci. Eng.* 1956, *1*, 409.
26. Lee, T.S. *U.S. At. Ener. Comm. Rep., IS-T-824*, 1979.
27. Ettmayer, P.; Jangg, G. *Monatsh. Chem.* 1973, *104*, 1120.
28. Schweitzer, D.G. *Brookhaven National Laboratory* 1958; unpublished results communicated to the evaluators by J. R. Weeks.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Uranium; U; [7440-61-1] (2) Mercury; Hg; [7439-97-6]	Ahmann, D.H.; Baldwin, R.R.; Wilson, A.S. *U.S. At. Ener. Comm. Rep. CT-2960*, 1945.
VARIABLES: Temperature: 25-350°C	**PREPARED BY:** C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of uranium in mercury:

t/°C	Soly/mass %	Soly/at %[a]
25	0.001-0.01	0.0008-0.008
100	0.03	0.025
200	0.05	0.042
300	0.50	0.42
350	1.06	0.89

[a] by compilers

The solid phases in equilibrium with the homogeneous amalgam are UHg_4, UHg_3 and UHg_2.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

1 to 2% uranium amalgams were placed on a fine-porosity sintered-glass filter in a special apparatus. The amalgams were covered with Na_2CO_3 to protect them to some extent from air. The apparatus was then heated to desired temperature and centrifuged immediately for 20 to 30 sec. Control runs with the asbestos packed centrifuge cup indicated that the temperature dropped only about 10° at 300°C during the centrifugation. The filtrate after a given run was then analyzed for uranium and mercury.

SOURCE AND PURITY OF MATERIALS:

Uranium purity was better than 99.9%. Mercury was washed with nitric acid, then triple distilled in glass.

ESTIMATED ERROR:

Soly: not specified; error probably quite high (compilers).

Temp: nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Uranium; U; [7440-61-1] (2) Mercury; Hg; [7439-97-6]	Dean, O.C.; Sturch, E.S.; Morrison, B.V.; Blanco, R.E. *U.S. At. Ener. Comm. Rep., ORNL-2242*, 1957.
VARIABLES: Temperature: 298-873 K	**PREPARED BY:** C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of uranium in mercury:

T/K	Soly/mass %	Soly/at %[a]	Pressure/atm
298	$<1 \times 10^{-3}$	--	--
629	1.12	0.95	--
873		≥ 19	23

These results were also presented in (1); kinetics of dissolution and the solubilities of U in Hg and in Bi, Mg and Na amalgams were investigated in this work.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A sample of U was heated in boiling Hg for 30 min. under an argon atmosphere. The amalgam was filtered at 629 and at 298 K, and the filtrates were analyzed after dissolution in nitric acid. The method of estimation at 873 K is not specified.

SOURCE AND PURITY OF MATERIALS:

Nothing specified but probably the same as in (2): i.e., U of highest purity available. Hg purified by filtering, washing with HNO_3 and double distillation under vacuum

ESTIMATED ERROR:

Soly: nothing specified; precision better than $\pm$ 5% (compilers).

Temp: nothing specified.

REFERENCES:

1. Dean, O.C. *Progr. Nucl. Ener., Ser. 3* 1958, *2*, 412-9.

2. Forsberg, H.C. *U.S. At. Ener. Comm. Rep., ORNL-2885*, 1960.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Uranium; U; [7440-61-1] (2) Mercury; Hg; [7439-97-6]	Messing, A.F.; Dean, O.C. *U.S. At. Ener. Comm. Rep., ORNL-2871*, 1960.
VARIABLES: Temperature: 40-356°C	**PREPARED BY:** C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of uranium in mercury:

t/°C	Soly/mass %	Soly/at %
40	0.0067	0.0056
50	0.0093	0.0078
70	0.0155	0.0131
100	0.0340	0.0286
145	0.0826±0.0007	0.0696
150	0.0930	0.0783
205	0.234±0.004	0.197
250	0.436±0.017	0.368
300	0.727±0.002	0.613
356	1.21±0.03	1.02

The authors also reported that the solubility of uranium in 0.1 mass % magnesium amalgam was higher than in pure mercury, and that the solubility in saturated thorium amalgam was lower than in mercury.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Mercury and uranium, after drying and outgassing in the stainless steel dissolver, were kept for several days at the desired temperature. After equilibration, a sample of liquid amalgam was forced through the filter and filtrate was collected, dissolved in nitric acid, and submitted for analysis for uranium and mercury.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: standard deviation of fitted equation was 0.05136.

Temp: nothing specified.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Uranium; U; [7440-61-1] (2) Mercury; Hg; [7439-97-6]	Jangg, G.; Palman, H. *Z. Metallk.* 1963, *54*, 364-9.
VARIABLES: Temperature: 20-267 K	**PREPARED BY:** C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of uranium in mercury was presented graphically as a function of temperature. Numerical values of the data points were read from the curve by the compilers.

t/°C	Soly/at %
20	4.2×10^{-3}
50	9.8×10^{-3}
100	3.2×10^{-2}
150	7.4×10^{-2}
162	9.6×10^{-2}
200	0.17
243	0.25
250	0.28
267	0.33

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The heterogeneous amalgam was introduced into a specially constructed apparatus made of glass. After twelve hours of equilibration at the temperature of the experiment, the amalgam was filtered through the sintered-glass frit under the pressure of purified nitrogen. The metal content was then analytically determined in the filtered saturated amalgam by an unspecified method.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: accuracy ± 5%.

Temp: precision ± 2 K.

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Uranium; U; [7440-61-1] (2) Mercury; Hg; [7439-97-6]	Ettmayer, P.; Jangg, G. *Monatsh. Chem.* 1973, *104*, 1120-30.
VARIABLES:	**PREPARED BY:**
One temperature: 573 K	C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of U in Hg at 573 K was reported to be 0.6 mass %. The atomic % solubility calculated by the compilers is 0.5 at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Uranium amalgam was obtained by dissolution of U turnings in Hg. The materials were placed in a bomb and heated to 723-773 K. The amalgam was filtered and analyzed by an unspecified method.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: nothing specified; about ± 5% (compilers).

Temp: precision ± 2 K (compilers).

REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Uranium; U; [7440-61-1] (2) Mercury; Hg; [7439-97-6]	Lee, T.S.; Chiotti, P.; Mason, J.T. *J. Less-Common Metals* 1979, *66*, 33-40.
VARIABLES: Temperature: 455-845°C Pressure	**PREPARED BY:** C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The points on the U-Hg liquidus line were determined under constrained pressure:

t/°C	Soly/at %	Pressure/atm
455	2.0	--
735	<6.5	--
845	9.5-10.0	90

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Preequilibrated alloy or the separate metals were sealed in tantalum capsules in a He atmosphere; a thermocouple well was sealed to the bottom of the capsule. Differential thermal analysis was made in a He atmosphere by inserting the filled and an empty capsule in a nickel block. Chromel-Alumel thermocouples were used for the DTA; the samples were heated in a split tube furnace.

SOURCE AND PURITY OF MATERIALS:

Reactor grade U and high purity, triply distilled Hg were used. Chemical analysis of U showed 1-5 x 10^{-2} and 0.5-10 x 10^{-2} mass % of oxygen and carbon, respectively. The alloys contained less than 6 x 10^{-2} mass % of Ta.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: nothing specified; $\pm$ 5 K (by compilers).

REFERENCES:

COMPONENTS:	EVALUATOR:
(1) Plutonium; Pu; [7440-07-5] (2) Mercury; Hg; [7439-97-6]	C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION:

White (1) reported that the solubility of plutonium in mercury at room temperature is 1.36×10^{-2} at %. Bowersox and Leary (2,3) made more extensive measurements of the plutonium solubility; these authors reported that the solubility increases from 1.31×10^{-2} to 0.561 at % in the temperature range of 294 to 598 K. The result of (1) is in good agreement with those reported by Bowersox and Leary.

The saturated plutonium amalgam is in equilibrium with the Pu-Hg intermetallic compound, Pu_5Hg_{21} or $PuHg_3$ (2,4); however, the temperature range of stability for these compounds have not been established. The partial phase diagram has been reported by (5) and (6).

The recommended (r) and tentative values of the solubility of Pu in Hg:

T/K	Soly/at %	Reference
293	1.3×10^{-2} (r)	[1,2]
298	1.5×10^{-2}	[2]
323	2.6×10^{-2}	[2]
373	6.4×10^{-2}	[2][a]
473	2.2×10^{-1}	[2]
573	4.8×10^{-1}	[2]

[a] Interpolated value from data of (2).

References

1. White, A.G. *At. Ener. Res. Establ. Rep., C/R 1468*, 1955.
2. Bowersox, D.F.; Leary, J.A. *J. Inorg. Nucl. Chem.* 1959, *9*, 108.
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6. Blank, H.; Brossmann, G.; Kemmerick, M. *F.R.G. At. Ener. Comm. Rep., KFK-105*, 1962, p. 137.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Plutonium; Pu; [7440-07-5] (2) Mercury; Hg; [7439-97-6]	Bowersox, D.F.; Leary, J.A. *J. Inorg. Nucl. Chem.* 1959, *9*, 108-112.
VARIABLES: Temperature: 20-325°C	**PREPARED BY:** C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

Solubility of plutonium in mercury:

t/°C	Soly/10^2 at %
20[a]	1.61
21	1.31
24	1.61
50	2.55
100	6.25
150[a]	12.6
190	18.2
200	19.0
225	27.5
260	38.0
280	42.1
300	49.6
325[a]	56.1

[a] also reported in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Mercury was outgassed in the reaction vessel at 250°C, then cooled to room temperature. The vessel was filled with helium and freshly machined plutonium turnings were added. The evacuation and filling of the vessel with helium were repeated several times. The mixture of the metals was held at 250-300°C for one day and was shaken periodically by hand. The temperature of the vessel was adjusted at desired level. The liquid phase was sampled periodically and filtered through a sintered-glass filter. Plutonium was leached from the filtrate by contacting with concentrated HCl for one day. The solution was analyzed by radio-assay for Pu content.

SOURCE AND PURITY OF MATERIALS:

99.8% pure plutonium and triply-distilled mercury were used.

ESTIMATED ERROR:

Soly: accuracy ± 1%.

Temp: precision ± 2%.

REFERENCES:

1. Bowersox, D.F.; Leary, J.A. *U.S. At. Ener. Comm. Rep., LAMS-2518*, 1961.