- (1) Boron; B; [7440-42-8]
- (2) Mercury; Hg; [7439-97-6]

#### **EVALUATOR:**

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

#### CRITICAL EVALUATION:

There are no experimental data on the solubility of boron in mercury, but the solubility is expected to be very low. From his semiempirical equations Kozin first estimated (1) a 298 K solubility of 3.1 x  $10^{-12}$  at %, and he subsequently estimated (2) a solubility of 4.75 x  $10^{-9}$  at % at the same temperature. Neither of the estimated solubilities can be recommended by the evaluators.

Based on the experimental observations of Wald and Stormont (3), Moffatt (4) constructed a schematic phase diagram of the B-Hg system. No stable compounds or solid solutions of boron and mercury are formed in this system.

#### References

- Kozin, L.F. Tr. Inst. Khim. Nauk Akad. Nauk Kaz. SSR 1962, 9, 101.
   Kozin, L.F. Fiziko-Khimicheskie Osnovy Amalgamoi Metallurgii, Nauka, Alma-Ata, 1964.
- Wald, F.; Stormont, R.W. J. Less-Common Metals 1965, 9, 423.
   Moffatt, W.G. The Handbook of Binary Phase Diagrams, Vol. I, Genium Publishing Corp., Schenectady, NY 1978.

84 Aluminum

COMPONENTS:

(1) Aluminum; A1; [7429-90-5]

(2) Mercury; Hg; [7439-97-6]

**EVALUATOR:** 

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

#### CRITICAL EVALUATION:

The solubility of aluminum in mercury near room temperature is low and some early reports (1-3) indicated only that the solubility limit is below  $10^{-2}$  at %. The first precise determination of the solubility was reported by Fogh (4) who found  $1.4 \times 10^{-2}$ and 2.79 at % aluminum in the saturated amalgam at room temperature and at the boiling point, respectively; more recent measurements confirm these estimates. Shalaevskaya and coworkers (5-7) reported that the solubility increased from  $8.9 \times 10^{-3}$  to  $1.63 \times 10^{-2}$ at % in the temperature range of 293 to 323 K. These values are of the proper magnitude but their dependence on temperature is too low. The potentiometric measurements of Ziegel and coworkers (8) resulted in a solubility of 1.3 x  $10^{-2}$  at % at 303 K; this value lies between the results of (4) and (5-7). If aluminum interacts with the amalgamated silver (5-7) and platinum (8) of the working electrodes in the potentiometric measurements, then the results of (5-8) may be slightly understated. Kozin's (9) predicted solubility of 0.22 at % at 298 K is much too high. Smits and De Gruyter (10,11) conducted thermoanalytical measurements at higher temperatures and reported the phase diagram for this system; the numerical data for the liquidus were reported by De Gruyter (12). Klemm and Weiss (13) determined the solubility between 695 and 868 K by equilibration of the metals and chemical analysis of the saturated liquids; these authors found that the solubility increased from 7.5 to 82.7 at % in this temperature range. The latter solubilities were in good agreement with those reported by De Gruyter. In a lower temperature range of 333 to 573 K, Schmidt (14) reported that the solubilities increased from  $4.5 \times 10^{-2}$  to 1.25 at %, respectively. The latter results are in good agreement with those determined by Liebhafsky (15) at 349 to 585 K. Jangg and Palman (16), without presenting their data, stated that the solubility of aluminum from their measurements agreed to within +5% with those of (12), (13) and (15).

The saturated aluminum amalgams are in equilibrium with solid aluminum, and no Al-Hg phases are known to exist (12). The phase diagram for this system is shown in Fig. 1 (17).

Tentative and recommended (r) values of aluminum solubility in mercury:

<u>T/K</u>	Soly/at %	Reference
293	0.014	[4]
298	0.016 <sup>a</sup>	[4,14]
373	0.10 <sup>b</sup>	[14,15]
473	0.51	[14]
573	1.3 <sup>b</sup>	[14,15]
673	5.6	[15]
773	17	[13]
873	84 (r)	[12,13]

<sup>&</sup>lt;sup>a</sup>Interpolated value from data of cited references.

bMean value from cited references.

- (1) Aluminum; A1; [7429-90-5]
- (2) Mercury; Hg; [7439-97-6]

#### EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

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CRITICAL EVALUATION: (continued)

mass %

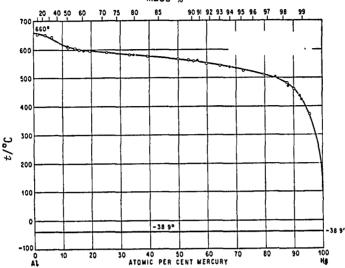


Fig. 1. The Al-Hg system (17).

# References

- 1. Mylius, F.; Rose, F. Z. Instrumentenk. 1893, 13, 81.
- 2. Kremann, R.; Müler, R. Z. Metallk. 1920, 12, 311.
- Strachan, J.F.; Harris, N.L. J. Inst. Metals 1956-57, 85, 17. Fogh, I. Kgl. Dansk. Vidensk. Selsk. Mat. Fys. Medd. 1921, III, No. 15.
- Shalaevskaya, V.N.; Igolinskii, V.A.; Kataev, G.A. Dep. VINITI, 588-75, 1975; Abstracted in Zh. Fiz. Khim. 1975, 49, 1587; Uspekhi Polarogr. s Nakopl., Tomsk, 1973, p. 115.
- Shalaevskaya, V.N.; Igolinskii, V.A. Zh. Prikl. Khim. 1975, 48, 1152.
   Igolinskii, V.A.; Shalaevskaya, V.N.; Guryanova, O.N.; Igolinskaya, I.M.; Kotova, N.A. Sovr. Probl. Polarografii s Nakopleniem, Tomsk, 1975, p. 150.
- Ziegel, G.; Peled, E.; Gileadi, E. Electrochim. Acta 1978, 23, 363.
- Kozin, L.F. Fiziko-Khimicheskie Osnovy Amalgamnoi Metallurgii, 1964.
- 10. Smits, A.; De Gruyter, C.J. Proc. Acad. Sci. Amsterdam 1921, 23, 966; Versl. Akad. Wetensch. 1921, 29, 747.

- Smits, A. Z. Elektrochem. 1924, 30, 424.
   De Gruyter, C.J. Rec. Trav. Chim. 1925, 44, 937.
   Klemm, W.; Weiss, P. Z. Anorg. Chem. 1940, 245, 285.
- Schmidt, W. Metall. 1949, 3, 10.

- Liebhafsky, H.A. J. Am. Chem. Soc. 1949, 71, 1468.
   Jangg, G.; Palman, H. Z. Metallk. 1963, 54, 364.
   Hansen, M.; Anderko, K. Constitution of Binary Alloys, McGraw-Hill, N.Y., 1958, p. 99.

COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Aluminum; A1; [7429-90-5] (2) Mercury; Hg; [7439-97-6]	Fogh, I.  Kgl. Dansk. Vidensk. Selsk. Mat. Fys.  Medd. 1921, III, No. 15.	
VARIABLES:	PREPARED BY:	
Temperature	C. Guminski; Z. Galus	

# EXPERIMENTAL VALUES:

Solubility of aluminum in boiling mercury and at room temperature were determined to be 0.385  $\pm$  0.002 and 0.0019  $\pm$  0.0001 mass %, respectively. The respective atomic % solubilities calculated by the compilers are 2.79 and 0.014 at %.

The author reported  ${\rm Al_2Hg_3}$  as a phase in equilibrium with the saturated amalgams. However, this was not confirmed in later works.

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

A piece of aluminum was heated in hydrogen atmosphere in Jena-glass tube. Then this piece was placed under the surface of mercury and the system was boiled for 2-3 hours. The amalgams were filtered with the use of glass-wool. The samples were weighed, then treated with HCl. Aluminum was determined as  $\mathrm{Al}_2\mathrm{O}_3$ .

# SOURCE AND PURITY OF MATERIALS:

Nothing specified.

#### ESTIMATED ERROR:

Soly: precision better than  $\pm$  5%.

Temp: nothing specified.

Aluminum 87

COMPONENTS: ORIGINAL MEASUREMENTS:

(1) Aluminum; A1; [7429-90-5](2) Mercury; Hg; [7439-97-6]

Rec. Trav. Chim. 1925, 44, 937-48.

VARIABLES:

PREPARED BY:

Temperature: 369-652°C

C. Guminski; Z. Galus

De Gruyter, C.J.

#### EXPERIMENTAL VALUES:

Crystallization temperatures were reported as a function of aluminum concentration:

t/°C	at % A1	<u>t/°C</u>	at % Al	t/°C	at % A1
652	98.6	595	80.46	550	40.17
650	95.87	590	74.56	542	35.46
643	93.5	582	66.7	524	27.38
613	88.16	576	60.55	510	20.36
610	87.99	566	50.0	479	12.42
604	85.36	561	46.54	460	10.0
600	84.17	558	44.73	369	4.55

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Aluminum and mercury were mixed in glass tubes, then the tubes were sealed and heated and cooling curves were recorded.

# SOURCE AND PURITY OF MATERIALS:

Aluminum supplied by Kahlbaum; purity not specified.

Mercury purity not specified.

#### ESTIMATED ERROR:

Soly: nothing specified.

Temp: nothing specified; precision no better than few degrees (compilers).

- (1) Aluminum; A1; [7429-90-5]
- (2) Mercury; Hg; [7439-97-6]

#### ORIGINAL MEASUREMENTS:

Klemm, W.; Weiss, P.

Z. Anorg. Chem. 1940, 245, 285-7.

VARIABLES:

PREPARED BY:

Temperature: 422-595°C

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Solubility of aluminum in mercury:

t/°C	Soly/at %	Soly/mass %
422	7.5	1.01
435	7.9	1.14
470	12.2	1.83
502	16.5	2.20
537	32.2	5.99
560	43.4	9.33
581	65.4	20.61
595	82.7	39.15

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The metals were sealed in evacuated quartz tubes then heated for 24 hours at the desired temperatures. After equilibration, each tube was turned up and the amalgam was filtered through a narrow constriction in the tube. The filtrate was treated with dilute HCl, and the mercury was dried and weighed. Aluminum in the solution was determined as  $Al_2O_3$  after precipitation with ammonium hydroxide.

# SOURCE AND PURITY OF MATERIALS:

Nothing specified.

#### ESTIMATED ERROR:

Soly: precision  $\pm$  1%. Temp: precision  $\pm$  2 K.

Aluminum 89

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Aluminum; A1; [7429-90-5] (2) Mercury; Hg; [7439-97-6]	Liebhafsky, H.A.  J. Am. Chem. Soc. 1949, 71, 1468-70.
VARIABLES:	PREPARED BY:
Temperature: 76-400°C	C. Guminski; Z. Galus

# EXPERIMENTAL VALUES:

Solubility of aluminum in mercury:

<u>t/°C</u>	Soly/mass %	Soly/at % <sup>a</sup>
76	$9.0 \times 10^{-3}$	0.067
101	$1.5 \times 10^{-2}$	0.11
103	$1.7 \times 10^{-2}$	0.13
125	$2.4 \times 10^{-2}$	0.18
160	$3.5 \times 10^{-2}$	0.26
260	0.11	0.81
307 <sup>b</sup>		1.8
312	0.18	1.32
400 <sup>b</sup>		5.6

<sup>&</sup>lt;sup>a</sup>by compilers.

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The saturated amalgam was obtained by rotating an Al rod, which was used as the stirrer, in the amalgam which was always flushed with hydrogen to prevent oxidation of the amalgam. Samples of the amalgam were extracted with a glass sampling tube at the equilibration temperatures. The amalgam was then treated with 2 mol dm<sup>-3</sup> HCl and the evolved H<sub>2</sub> was measured with a gas burette to determine the Al content. The Hg was determined volumetrically. Norton and Harrington used the procedure of Klemm and Weiss (2).

# SOURCE AND PURITY OF MATERIALS:

Aluminum purity was 99+%.

Mercury purity not specified.

#### ESTIMATED ERROR:

Soly: precision no better than several

percent (compilers).

Temp: nothing specified.

- Norton, F.H.; Harrington, R.H. Unpublished work.
- 2. Klemm, W.; Weiss, P. Z. Anorg. Chem. 1940, 245, 285.

<sup>&</sup>lt;sup>b</sup>Unpublished data of Norton and Harrington (1).

- (1) Aluminum; A1; [7429-90-5]
- (2) Mercury; Hg; [7439-97-6]

# ORIGINAL MEASUREMENTS:

Schmidt, W.

Metall. 1949, 3, 10-13.

#### VARIABLES:

Temperature: 60-300°C

# PREPARED BY:

C. Guminski; Z. Galus

# EXPERIMENTAL VALUES:

Solubility of aluminum in mercury:

t/°C	Soly/mass %	Soly/at % <sup>a</sup>
60	$6 \times 10^{-3}$	0.045
100	$1.2 \times 10^{-2}$	0.089
150	$3.4 \times 10^{-2}$	0.25
200	$6.9 \times 10^{-2}$	0.51
300	0.17	1.25

aby compilers.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

No experimental details were given, but the results compare favorably with other published measurements. The determinations were performed in the laboratory of Firma W. Schmidt, Leichtmetallhütte, in München, W. Germany.

#### SOURCE AND PURITY OF MATERIALS:

Nothing specified.

# ESTIMATED ERROR:

Nothing specified.

Aluminum 91

#### COMPONENTS:

- (1) Aluminum; A1; [7429-90-5]
- (2) Mercury; Hg; [7439-97-6]

#### ORIGINAL MEASUREMENTS:

Shalaevskaya, V.N.; Igolinskii, V.A. Zh. Prikl. Khim. 1975, 48, 1152-4.

#### VARIABLES:

Temperature: 20-50°C

#### PREPARED BY:

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Solubility of aluminum in mercury:

<u>t/°C</u>	Soly/10 <sup>3</sup> mass %	Soly/10 <sup>3</sup> at %
20	1.18	8.85
30	1.28	9.74
40	1.40	10.77
50	3.10 <sup>a</sup>	16.33

 $<sup>^{\</sup>rm a}$ This value should be 2.22 x  $10^{-3}$ ; the compilers attribute this error to a misprint in the paper.

The above data were reported in (1) and (2).

# AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Small aluminum cylinder pressed exactly into a silver tube was polished in 0.5% solution of  ${\rm Hg}_2({\rm NO}_3)_2$  for subsequent amalgamation of the surface with a drop of mercury. The thickness of the mercury film on the aluminum was measured. The tube was then placed in an electrolyte (0.5 mol dm $^{-3}$  KAlO2, 1 mol dm $^{-3}$  KOH, 1.5 mol dm $^{-3}$  KCI) and was polarized anodically. The stationary oxidation current was recorded and the solubility was calculated from the slope of the curve relating the current to the thickness of the mercury film. The measurements were performed in an argon atmosphere.

# SOURCE AND PURITY OF MATERIALS:

Aluminum was of high purity.

Mercury purity not specified.

#### ESTIMATED ERROR:

Soly: nothing specified. Temp: precision + 0.1 K.

- Igolinskii, V.A.; Shalaevskaya, V.N.; Guyanova, O.N.; Igolinskaya, I.M.; Kotova, N.A. Sovr. Probl. Polarografii s Nakopleniem, Tomsk, 1975, p. 150.
- s Nakopleniem, Tomsk, 1975, p. 150.

  2. Shalaevskaya, V.N.; Igolinskii, V.A.; Kataev, G.A. Dep. VINITI, 588-75, 1975; abstracted in Zh. Fiz. Khim. 1975, 49, 1587.

- (1) Aluminum; A1; [7429-90-5]
- (2) Mercury; Hg; [7439-97-6]

#### ORIGINAL MEASUREMENTS:

Ziegel, S.; Peled, E.; Gileadi, E. Electrochim. Acta 1978, 23, 363-8.

#### VARIABLES:

PREPARED BY:

One temperature: 303 K

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The solubility of aluminum in mercury at 303 K was reported to be  $(17-18) \times 10^{-4}$  mass %. The atom % solubility calculated by the compilers is 1.3 x  $10^{-2}$  at %.

This result may be understated (see below under Method).

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Aluminum amalgam was prepared into mercury drop electrodes suspended on the tip of a platinum wire. The electroreduction was carried out in a solution of 1.3 mol dm<sup>-3</sup> AlBr<sub>3</sub> + 0.52 mol dm<sup>-3</sup> KBr in toluene at constant current. Then the open circuit potentials were measured at times longer than 300 s. The inflection on the curve relating reversible potential vs. logarithm of the charge passed corresponds to the saturation point of aluminum in mercury. All experiments were performed in a glovebox under an atmosphere of purified nitrogen or argon. It is possible part of Al reacted with Pt surface, so that concentration of Al in the Hg drop was decreased.

# SOURCE AND PURITY OF MATERIALS:

Toluene was dried by refluxing on Na, followed by 2 steps of vacuum distillation and drying by molecular sieves. AlBr<sub>3</sub> was purified by double vacuum sublimation. KBr was dried by heating overnight at 523 K in vac. Final purification of solution achieved by placing Al wire in Hg pool in cell and stirring several hours. Hg (Frutarom AR) was cleaned first by washing with conc. H<sub>2</sub>SO<sub>4</sub>, then rinse with 10% HNO<sub>3</sub> and triple distilled H<sub>2</sub>O, and vac. distilled.

#### ESTIMATED ERROR:

Soly: precision about  $\pm$  10% (compilers).

Temp: nothing specified.

- (1) Gallium; Ga; [7440-55-3]
- (2) Mercury; Hg; [7439-97-6]

#### **EVALUATOR:**

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

#### CRITICAL EVALUATION:

Gallium melts at 303 K, but the solubility of the liquid metal in mercury is only a few atom percent between 273 and 373 K. Gilfillan and Bent (1), from freezing point depression measurements, found that the solubility at 233.5 K is 0.37 at %.

Spicer and Bartholomay (2) equilibrated the saturated amalgams at 308 and 373 K, and they determined the solubility of gallium by chemical analysis of the liquid phase. At 308 K the solubility in the mercury-rich and the gallium-rich liquid was 3.6 and 97.6 at %, respectively; at 373 K the corresponding solubilities were 3.9 and 96.8 at %. Although the solubilities at 308 K are satisfactory the values at 373 K are erroneous. This study suggested that a critical miscibility temperature is non-existent at normal pressures.

Predel (3) determined the phase diagram of the Ga-Hg system by thermal analysis and found the critical miscibility point at 477 K at 50 at % Ga and the monotectic point at 300.88 K at 98.49 at % Ga. Nizhnik and Zvagolskaya (4), from potentiometric and analytical measurements, determined a solubility of 3.81 at % at 303 K; this value is in good agreement with Predel's solubility curve. Yatsenko and Druzhinina (5) determined the solubility of gallium at 283 to 368 K by equilibration and chemical analysis of the liquid phases. At 308 K the latter authors were in agreement with ref. (2), but the solubilities at higher temperatures were lower than those reported by Predel (3). It should be noted here that the critical temperature first reported by Predel was confirmed by Shürmann and Parks (6) and by D'Abramo et al. (7) who determined the temperatures at 476.48 and 475.58 K, respectively. Schürmann and Parks employed high-precision electrical resistivity measurements, while D'Abramo et al. utilized neutron radiography. A comparison of the data of ref. (3) with those of (6) and (7) shows that the liquidus curve of Predel should be slightly modified toward lower temperatures to give a better fit to the solubility data of Yatsenko and Druzhinina. More recently, Gaune-Escard and Bros (8) employed calorimetric measurements to redetermine the liquidus line of the Ga-Hg system; these authors also incorporated some unpublished data of Amarell (9) and confirmed the earlier liquidus reported by Predel (3).

Grosse (10,11) determined the solubility of liquid Ga at 293 K and of solid Ga at 273 and 254 K; because the melting point of Ga is 303 K, the liquid Ga system was metastable. In spite of the apparently high precision of Grosse's measurements, Lindauer (12) expressed skepticism because the difference between the solubilities of solid and liquid Ga, i.e.,  $3.1 \pm 0.05$  and  $3.28 \pm 0.05$  at %, respectively, is not significantly higher than the precision of the method used for the measurements.

Kozin (13) predicted a solubility of 98.6 at % at 298 K, but this solubility is clearly too high for the Hg-rich region. However, the calculated solubility is nearly the same as that of the supercooled amalgam in the Ga-rich region; in this region no liquid phase is stable below 300.9 K. The solubility of 3.7 x  $10^{-2}$  at % reported by Stepanova and Zakharov (14) at 298 K is too low and is rejected.

At temperatures below 301.0 K the saturated amalgams are in equilibrium with solid gallium which is saturated with a small amount of mercury. Between 301.0 and 475.6 K two immiscible phases are in equilibrium, as shown in Fig. 1 (3).

COM	(PO	NEN	TS:

- (1) Gallium; Ga; [7440-55-3]
- (2) Mercury; Hg; [7439-97-6]

# EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

CRITICAL EVALUATION: (Continued)

Recommended (r) and tentative solubility of gallium in mercury; see Fig. 1 for complete solubility.

Hg-rich region

		<del></del>
<u>T/K</u>	Soly/at %	Reference
254	1.1	11
273	1.9	10
293	3.1 Ga (solid) 3.3 Ga (liquid)	5 10
298	3.4 <sup>a</sup>	5
301	3.8	4
323	4.8 <sup>a</sup>	3,8,9
373	8.2	9
473	42	3
476	50.0 (r)	3,6,7

<sup>&</sup>lt;sup>a</sup>Interpolated value from cited references.

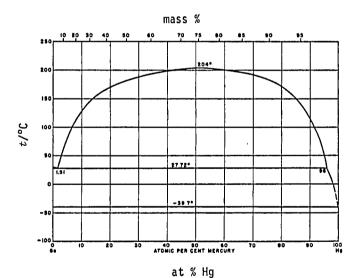


Fig. 1. The Ga-Hg system (3).

- (1) Gallium; Ga; [7440-55-3]
- (2) Mercury; Hg; [7439-97-6]

#### **EVALUATOR:**

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

CRITICAL EVALUATION: (continued)

#### References

- Gilfillan, E.S.; Bent, H.E. J. Am. Chem. Soc. 1934, 56, 1661.
   Spicer, W.M.; Bartholomay, H.W. J. Am. Chem. Soc. 1951, 73, 868.

- Spicer, W.R.; Baitholomay, R.W. J. Am. Chem. Soc. 1931, 70, 603.
   Predel, B. Z. Phys. Chem., N.F. 1960, 24, 206.
   Nizhnik, A.T.; Zvagolskaya, E.V. Zh. Neorg. Khim. 1961, 6, 1006.
   Yatsenko, S.P.; Druzhinina, E.P. Zh. Neorg. Khim. 1961, 6, 1902.

- Schürmann, H.K.; Parks, R.D. Phys. Rev. Letters 1971, 26, 367, 835.
   D'Abramo, G.; Ricci, F.P.; Menzinger, F. Phys. Rev. Letters 1972, 28, 22.
   Gaune-Escard, M.; Bros, J.P. Thermochim. Acta 1979, 31, 323.
- 9. Amarell, G. Ph.D. Thesis, Karlsruhe, 1958; as cited in ref. (8).
  10. Grosse, A.V. U.S. At. Ener. Comm. Rep., NYO-2082-4, 1966.
  11. Grosse, A.V. U.S. At. Ener. Comm. Rep., NYO-2082-12, 1967.
  12. Lindauer, G.C. U.S. At. Ener. Comm. Rep., BNL-50048, 1967.

- 13. Kozin, L.F. Fiziko-Khimicheskie Osnovy Amalgamnoi Metallurgii, Nauka, Alma-Ata,
- 14. Stepanova, O.S.; Zakharov, M.S. Izv. Tomsk. Politekhn. Inst. 1966, 151, 21.

#### EXPERIMENTAL VALUES:

The solubility of gallium in mercury at  $30^{\circ}\text{C}$  was determined to be 1.3-1.5 mass % by a potentiometric method; by equilibration and chemical analysis the solubility was determined to be 1.36 mass %. The atomic % solubility calculated by the compilers is 3.81 at %.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The amalgams were prepared by mixing accurately weighed specimens of the metals in hot water which was slightly acidified with HCl. The amalgams were kept in a closed vessel under a solution of acidified GaCl3. The potentiometric solubility measurements were presumably made on concentration cells under a protective atmosphere of nitrogen. The solubility was determined from the breakpoint in the plot of EMF against Ga concentration. The solubility from chemical analysis was determined by equilibrating amalgams with 0.1-10 mass % Ga in weighed, glass tubes. Samples were taken from the equilibrated amalgam, then treated with HCl to dissolve the Ga. At low concentrations of Ga this metal was determined colorimetrically, while at high concentrations it was determined gravimetrically.

# SOURCE AND PURITY OF MATERIALS:

Gallium was 99.99% pure.

Mercury was polarographic grade.

#### ESTIMATED ERROR:

Soly: no better than few percent (by compilers).

Temp: nothing specified.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gallium; Ga; [7440-55-3] (2) Mercury; Hg; [7439-97-6]	Spicer, W.M.; Bartholomay, H.W.  J. Am. Chem. Soc. 1951, 73, 868-9.
VARIABLES: Temperature: 35-100°C	PREPARED BY: C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The solubility of gallium in mercury-rich and in gallium-rich regions was determined at  $35 \text{ and } 100^{\circ}\text{C}$ .

Mercury-rich		Gallium-rich		
t/°C	Soly/mass %	Soly/at % <sup>a</sup>	Soly/mass %	Soly/at % <sup>a</sup>
35	$1.3 \pm 0.1$	3.6	$93.3 \pm 0.4$	97.6
100	1.4	3.9	$91.4 \pm 0.2$	96.8

<sup>&</sup>lt;sup>a</sup>by compilers

The result at  $100^{\circ}\text{C}$  for the Hg-rich region is too low, probably because part of the gallium was oxidized (compilers).

# AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Weighed portions of both metals were placed in a glass tube and covered with a solution of GaCl<sub>3</sub> in dilute HCl, then the samples were equilibrated with frequent shaking at constant temperature. After equilibration, several small samples were taken from each layer and weighed, then the gallium was extracted with HCl and the mercury was washed, dried and reweighed.

# SOURCE AND PURITY OF MATERIALS:

Gallium from Aluminum Company of America was 99.95% pure.

Mercury was purified by washing with nitric acid and water, then dried and distilled.

#### ESTIMATED ERROR:

Soly: precision better than  $\pm$  7%.

Temp: nothing specified.

# ORIGINAL MEASUREMENTS: COMPONENTS: (1) Gallium; Ga; [7440-55-3] Predel, B. (2) Mercury; Hg; [7439-97-6] Z. Phys. Chem. N.F. 1960, 24, 206-16. VARIABLES: PREPARED BY:

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Temperature: 337-477 K

The data were reported graphically as a phase diagram for the Ga-Hg system. The liquidus data points were read from the curve by the compilers.

<u>T/K</u>	at % Hg	<u>T/K</u>	at % Hg
337	4.1	469	38.4
370	6.6	473	43.8
385	8.0	477	50.6
391	8.7	475	55.4
407	11.0	471	61.0
404	11.2	468	66.2
419	13.3	463	72.0
428	15.0	456	75.8
447	20.3	443	81.0
449	21.2	429	84.5
458	25.7	397	89.3
457	26.4	356	93.0
464	31.1	337	94.2

The eutectic point was determined at 1.51 at % Hg and 27.72°C.

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

The freezing points were determined from cooling curves on amalgam samples which were protected from oxidation by an atmosphere of nitrogen. The temperatures were determined with a NiCr-Ni thermocouple. Heating curves also were obtained to ascertain the cooling curve data. Although not reported, the amalgams were presumably prepared by mixing desired amounts of the metals.

# SOURCE AND PURITY OF MATERIALS:

Gallium purity was 99.999%.

Mercury was purified by vacuum distillation.

#### ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision  $\frac{+}{1}$  0.01 K in measurements, but  $\frac{+}{1}$  K  $\frac{1}{1}$ n read-out values by

compilers.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gallium; Ga; [7440-55-3] (2) Mercury, Hg; [7439-97-6]	Yatsenko, S.P.; Druzhinina, E.P. Zh. Neorg. Khim. 1961, 6, 1902-4.
VARIABLES: Temperature: 10-95°C	PREPARED BY: C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The solubility of gallium in the Ga- and Hg-rich regions were determined:

	Soly in Hg-	rich region	Soly in Ga-ri	ch region
<u>t/°C</u>	Mass %	at % <sup>a</sup>	Mass %	at %a
10	0.86	2.44	(96.25) <sup>b</sup>	(98.68)
22	1.13	3.19	(95.0) <sup>b</sup>	(98.17)
30.5	1.20	3.38	94.6	98.06
35	1.30	3.65	93.9	97.73
50	1.49	4.17	93.1	97.43
65	1.72	4.82	92.0	97.07
80	1.90	5.29	91.0	96.67
95	2.22	6.14	. 89.7	96.16

<sup>&</sup>lt;sup>a</sup>by compilers.

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Gallium amalgams were obtained by electrolysis of Ga from  $Ga_2(SO_4)_3$  onto a mercury cathode. The mixture was agitated and equilibrated in a thermostat under a  $Ga_2(SO_4)_3$  solution. A steel ball on top of the Hg layer indicated the phase boundary. After equilibration, samples of amalgams were taken from both layers and the analysis made by: 1) dissolving the weighed sample in HCl and determination of Ga by titration with EDTA; 2) anodic oxidation of the amalgam where the end point of the dissolution was controlled potentiometrically.

# SOURCE AND PURITY OF MATERIALS:

Mercury was specified as "pure".

Gallium purity not specified.

# ESTIMATED ERROR:

Soly: precision  $\pm$  1.5%. Temp: nothing specified.

<sup>&</sup>lt;sup>b</sup>The values in parentheses are for the metastable region of liquid Ga layer.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Gallium; Ga; [7440-55-3] (2) Mercury; Hg; [7439-97-6]	Grosse, A.V.  U.S. At. Ener. Comm. Rep., NYO-2082-4,  1966.
VARIABLES:	PREPARED BY:
Temperature: (-18.8)-20°C	C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The solubility of liquid gallium in mercury by equilibration at  $20^{\circ}\text{C}$  was determined to be 1.16 mass %. The solubility determined by first heating the amalgam, followed by equilibration at  $20^{\circ}\text{C}$ , was found to be 1.12 mass %. The average value was 1.14 + 0.02 mass %, or 3.28 + 0.05 at % for this metastable equilibrium.

The solubility of solid gallium in mercury at -18.8 and  $0.0^{\circ}$ C were determined to be 1.15 and 1.90 at %, respectively.

The determination at 0.0 and  $-18.8^{\circ}$ C were probably made by the same method; the result at  $0.0^{\circ}$ C was published in (1).

# AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

In the first determination the unsaturated Ga amalgam was contacted with supercooled liquid Ga for about 30 h, and the area of the liquid blister on the amalgam remained constant for 2 weeks. The amount of Ga dissolved was found from the mass balance with the help of the blister-area vs. volume relationship established in separate experiments. In the second determination a blister of Ga was immersed in unsaturated Ga amalgam and was warmed to about 40°C with stirring in order to quickly dissolve the Ga. The mixture was cooled and allowed to stand for many hours at 20°C. The undissolved Ga blister was weighed to determine the solubility. The heterogeneous Ga amalgam was cooled to -18.8°C. It was filtered and Ga content was determined in the filtrate by addition of HCl at room temperature. Concentration of Ga was calculated from amount of H, evolved by the reaction.

# SOURCE AND PURITY OF MATERIALS:

Gallium from Aluminum Company of America was 99.99% pure.

Mercury from Bethlehem Apparatus Co. was triply vacuum distilled material; impurity content was less than 2 x  $10^{-5}$ %.

#### ESTIMATED ERROR:

Soly: precision  $\pm$  2%. Temp: precision  $\pm$  0.5 K.

# REFERENCES:

 Grosse, A.V.
 U.S. At. Ener. Comm. Rep., NYO-2082-12, 1967.

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# 

#### **EXPERIMENTAL VALUES:**

The critical temperature for miscibility at 50 at % Ga was determined to be  $203.32 \pm 0.50^{\circ}$ C in (1). The critical temperature reported by (2) was  $475.48 \pm 0.01$  K (202.33°C by compilers).

# AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

- (1) Weighed amounts of gallium and mercury were mixed in a Pyrex tube provided with ten tungsten electrodes at 15 mm intervals. The tube was placed in a thermostat and stirred at 250°C, then the temperature was slowly lowered. The resistance between different layers of the amalgam was measured as a function of temperature and R/Rc was plotted against temperature, where R is the measured resistance and Rc is the critical resistance at the critical temperature,  $\partial(R/Rc)/\partial T=0$ .
- (2) The amalgams were prepared by mixing the metals in near to equimolar ratios in a stainless steel cell. Neutron transmission measurements were made at decreasing temperatures starting at a temperature about 10°C above the critical temperature. There was a sharp change in transmission at the critical temperature for complete miscibility.

# SOURCE AND PURITY OF MATERIALS:

- (1) Gallium from Eagle-Picher Co. was 99.99% pure. Mercury from Beckman Instruments was 99.99999% pure.
- (2) Gallium from Fluka AG was 99.99% pure. Mercury from Rudipoint was 99.9% pure.

# ESTIMATED ERROR:

Temp: precision  $\pm$  0.50 K in (1);  $\pm$  0.01 K in (2).

COMPONENTS:

(1) Gallium; Ga; [7440-55-3] Gaune-Escard, M.; Bros, J.P.

(2) Mercury; Hg; [7439-97-6] Thermochim. Acta 1979, 31, 323-39.

VARIABLES:
Temperature: 313-466 K C. Guminski; Z. Galus

# EXPERIMENTAL VALUES:

Liquidus points were determined from microcalorimetric measurements:

	This	work	Unpublished work (1)		
<u>T/K</u>	x	x(Na)		Na)	
313	0.042	0.976	0.041	0.980	
353	0.063	0.954	0.066	0.950	
373	0.935 <sup>a</sup>	0.938	0.082	0.935	
423	0.150	0.870	0.150	0.860	
466	0.360	0.720	0.300	0.672	

<sup>&</sup>lt;sup>a</sup>obvious misprint in publication.

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

The liquidus points were determined microcalorimetrically: The metals mixed by breaking ampule, presumably containing Ga, in the Hg at equilibrated temperature and enthalpy of mixing determined from heat effect. Enthalpy determined as function of composition,  $\mathbf{x}_{\text{Ga}}$ , at each temperature, and breakpoint in plot of  $\Delta H_M$  vs.  $\mathbf{x}_{\text{Ga}}$  is liquidus at that temperature. Calorimeter calibrated by Joule effect. Measurements made under pressurized argon; both metals were protected from oxidation with layer of oil.

#### SOURCE AND PURITY OF MATERIALS:

Ga and Hg purity not specified.

Ar was grade "U" from Air Liquide Co.

#### ESTIMATED ERROR:

 $\Delta H_{M}$ : precision 2-6%.

Temp: not specified.

#### REFERENCES:

Amarell, G.
 *Dissert. Dokt. Naturwiss.*, Karlsruhe,
 1958.

- (1) Indium; In; [7440-74-6]
- (2) Mercury; Hg; [7439-97-6]

#### EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

#### CRITICAL EVALUATION:

The phase equilibria of the In-Hg system have been studied extensively, but most of the data have been reported graphically as phase diagrams only. In some of the reports the phase diagram appeared in relatively small figures and it was not possible to precisely read the numerical values of the liquidus from these phase diagrams.

Parks and Moran (1) reported the first study of the solubility of In in Hg, but these authors reported the indium solubilities of only 2.15 to 2.27 at % at 273 to 323 K; these very low values are rejected. Ito and coworkers (2) reported the In-Hg phase diagram and showed that indium has an appreciable solubility at room temperatures. Although the shape of their phase diagram was similar to those reported by subsequent authors, the liquidus temperatures of Ito et al. were too low by a few degrees, probably because of impurities in the indium which was used. Spicer and Banick (3), from thermoanalytical measurements, reported more accurate liquidus temperatures in the region of 68.05 to 100 at % In; the liquidus temperature increased monotonically from 283.5 to 429.2 K, respectively, in this range, and the authors fitted an equation for the solubility as a function of the temperature. Kozin and coworkers determined the phase diagram from thermoanalytical (4,5) and from potentiometric (6) measurements. Several other determinations of the phase diagram were reported during the years 1962-1964 (7-13), but Chiaranzelli and Brown (8) were the only authors to report numerical liquidus data. Robert and Thibault (14) also reported a phase diagram for the In-Hg system, but the liquidus between 7 and 25 at % In by these authors is not in agreement with those of the other accepted measurements; five different In-Hg compounds were proposed in this range by these authors. More recently, Franck (15,16) determined the liquidus in the In-rich region from vapor pressure measurements, while Hilpert (17) applied thermal analysis to confirm the liquidus temperature of  $352~\mathrm{K}$  at  $80~\mathrm{at}$  % In.

Kozin's (18) calculated solubility of 67.95 at % at 298 K is in good agreement with the accepted experimental solubility of 70 at %. Lieb1 (19) also has reported an indium solubility of 68 at % at room temperature, determined by coulometry, but no other details were reported for this measurement.

From potentiometric measurements at 293 K, Sundén (20) reported a solubility of 68 at %. The solubility measurement of Strachan and Harris (21) at room temperature is inconsequential.

Table 1 summarizes the congruently melting and the eutectic points which were derived from the phase diagrams reported by the various authors. The variation of the composition is approximately ±1 at %, while that for the temperature is ±1 K. In spite of the high precision in each data set reported in the literature, these variations arise because of the difficulty in exactly reading the data from the graphical presentations of the phase diagrams.

The saturated indium amalgams are in equilibrium with In-Hg intermetallic solid phases. The compounds,  $In_{11}Hg$ , InHg,  $InHg_4$  and  $InHg_5$  or  $InHg_6$ , have been identified with some certainty, but others, such as  $InHg_{11}$ ,  $InHg_9$ ,  $InHg_3$ ,  $In_5Hg_7$  and  $In_7Hg$ , are of doubtful existence. Fig. 1 shows the phase diagram reported by (5).

(1) Indium; In; [7440-74-6]

(2) Mercury; Hg; [7439-97-6]

EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

CRITICAL EVALUATION: (continued)

Tentative and recommended (r) values of In solubility in Hg:

	Hg-rich Re		In-rich Re	
<u>T/K</u>	Soly/at %	Ref.	Soly/at %	Ref.
235.6	34.4 (r) <sup>a</sup>			
242.6			62.3 (r) <sup>a</sup>	
254.7	7.5	[4]	62.3 (r) <sup>a</sup> 64 (r) <sup>b</sup>	[7,13]
258.7	14.3 (r) <sup>a</sup>			
273.2			66.5 (r) <sup>b</sup>	[7,8]
293.2			68.0 (r)	[6,20]
298.2			70.0	[4]
323.2			75.3	[6]
373			85 (r) <sup>b</sup>	[4,5,10,15]

<sup>&</sup>lt;sup>a</sup>Average of all reported data.

TABLE 1

Summary of Melting Points of Congruently Melting Compounds and Eutectic Points

Melting Points. T/K

Eutectic Points

mercing rounces, 1/K							
InHg <sub>6</sub>	InHg <sub>5</sub>	InHg	<i>T</i> /K	at % In	T/K	at % In	Ref.
	256	250	235.5	34.3	240.5	63.6	[2]
258.8		254.6	236.5	32.8	243.1	63.0	[4]
	258.6±0.3	254.0±0.3	235.8±0.3	34.0	242.4±0.3	61.7	[7]
260.8±0.4		256.6±0.1	240.1±0.±	34.3±0.5	244.6±0.1	63.6±0.5	[8]
	260.0	254.7	236.4	33.3	243.2	62.7	[9]
259.0±0.2		253.9±0.2	236.0±0.2	34.7±0.2	242.1±0.2	61.5±0.3	[10]
260±1		255±1	237±1	35	242.5±1	60	[11]
258.2		254.0	236.4	34.1	242.6	61.2	[12]
257.5		254.5	236	34.7	243	63.0	[13]
259.2±0.5		255.0±0.5	236.4±0.5	34.9	243.5±0.5	62.5	[14]
258.2±0.5		254.6±0.5	235.7±0.5	35.0	241.7±0.5	63.0	[5]

(Continued next page)

bInterpolated from data in cited references.

- (1) Indium; In; [7440-74-6]
- (2) Mercury; Hg; [7439-97-6]

#### **EVALUATOR:**

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION: (continued)

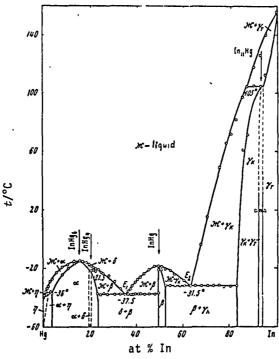


Fig. 1. The In-Hg system (5)

# References

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- 15.
- 16.
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   Sunden, N. Z. Elektrochem. 1953, 57, 100.
   Strachan, J.F.; Harris, N.L. J. Inst. Metals 1956-57, 85, 17.

- (1) Indium; In; [7440-74-6]
- (2) Mercury; Hg; [7439-97-6]

#### ORIGINAL MEASUREMENTS:

Kozin, L.F.; Tananaeva, N.N.

Zh. Neorg. Khim. 1961, 6, 909-12.

#### VARIABLES:

#### PREPARED BY:

Temperature: (-38)-150°C

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Liquidus temperatures of the In-Hg system were abstracted from the phase diagram by the compilers:

	t/°C	at % In	t/°C	at % In	t/°C	at % In	<u>t/°C</u>	at % In
	-38.0	0.1	-14.4	14.00	-20.7	45.00	66.0	77.50
	-37.1	0.25	-14.5	15.00	-19.2	47.50	79.0	80.00
	-36.0	0.30	-14.7	16.00	-18.9	48.00	80.0	80.25
	-35.1	0.50	-14.9	17.00	-18.65	49.00	90.0	82.50
ļ	-33.2	1.00	-15.7	19.00	-18.6	50.00	101.0	85.00
	-28.0	2.50	-16.0	20.00	-18.65	51.00	103.0	85.50
	-26.6	3.00	-18.2	22.50	-19.2	52.00	106.0	86.00
	-24.5	4.00	-20.4	25.00	-20.5	55.00	108.0	87.50
į	-22.5	5.00	-24.0	27.50	-26.00	60.00	114.0	88.00
	-18.4	7.50	-29.2	30.00	-26.4	61.50	123.0	90.00
	-16.9	9.00	-32.6	32.00	-26.0	63.00	134.0	94.60
	-16.0	10.00	-31.4	35.00	+25.0	70.00	150.0	97.50
	-14.83	12.00	-27.6	37.50	+37.0	72.50		
ļ	-14.5	13.00	-24.9	40.00	53.0	75.00		

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Separate alloys were prepared for each composition from pure metals; the containment tube was probably glass. The samples were covered with a thin layer of glycerol. The samples were cooled and heating curves were determined. Calibrated thermometers were used for the heating curves, but a Pt, Pt-Rh thermocouple was used to record the differential heating curves.

#### SOURCE AND PURITY OF MATERIALS:

Mercury was purified by treatment with  $HNO_3-Hg_2(NO_3)_2$ , then distilled twice in vacuum.

Indium was 99.999% pure.

# ESTIMATED ERROR:

Soly: nothing specified. Temp: precision + 0.1 K.

- (1) Indium; In; [7440-74-6]
- (2) Mercury; Hg; [7439-97-6]

# ORIGINAL MEASUREMENTS:

Spicer, W.M.; Banick, C.J.

J. Am. Chem. Soc. 1953, 75, 2268-2269.

#### **VARIABLES:**

Temperature: 10-151°C

#### PREPARED BY:

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The liquidus temperatures of indium-rich amalgams were reported:

t/°C	mass % In	at % In
151.3	97.46	98.53
135.1	90.12	94.09
121.7	84.07	90.22
108.2	79.30	87.00
94.2	74.70	83.77
78.1	69.84	80.19
59.2	64.89	76.36
37.6	60.01	72.40
10.3	54.92	68.05

aby compilers.

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Indium-rich alloys were made by adding the desired amount of Hg to the previously analyzed alloy in a test tube. The amalgams were protected from oxidation with mineral oil. The amalgam was analyzed gravimetrically by dissolving the In in conc. HCl then weighing the Hg residue. Cooling curves were determined by inserting the glass-clad copper-constant thermocouple into the amalgam and reading the temperature with a precision potentiometer. Down to 60°C the samples were cooled in a tube furnace, then at lower temperatures the sample tube was placed in water jacket and the latter was cooled with various solutions to obtain cooling curves.

# SOURCE AND PURITY OF MATERIALS:

Indium was 99.97% pure.

Mercury was purified by spraying through a column of dilute HNO3, washed, dried, then distilled under vacuum.

#### ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision  $\pm$  0.02 K.

108	Indium			
COMPONENTS:	ORIGINAL MEASUREMENTS:			
(1) Indium; In; [7440-74-6] (2) Mercury; Hg; [7439-97-6]	Ito, H.; Ogawa, E.; Yanagase, T. Nippon Kinzoku Gakkaishi 1951, 15B, 382-4.			
VARIABLES:	PREPARED BY:			
Temperature	C. Guminski; Z. Galus			
were determined to be -17 and -23°C, re	% In, and that of InHg, at 50.0 at % In, espectively.			

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

The amalgams were prepared by mixing weighed amounts of the metals in a sealed glass tube. The freezing points were determined by thermal analysis. The temperatures were measured with a copperconstantan thermocouple which was carefully calibrated by comparison with a calibrated Pt-PtRh thermocouple and with a mercury thermometer.

# SOURCE AND PURITY OF MATERIALS:

Indium was electrolytic material from zinc-fusion residue which was obtained from Hikoshima Refinery. Mercury was purified by vacuum distillation.

# ESTIMATED ERROR:

Soly: nothing specified.

Temp: nothing specified; precision no better than  $\pm$  0.5 K (compilers).

# COMPONENTS: (1) Indium; In; [7440-74-6] Sunden, N. (2) Mercury; Hg; [7439-97-6] Z. Elektrochem. 1953, 57, 100-2. VARIABLES: One temperature: 20°C PREPARED BY: C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Solubility of indium in mercury at  $20.0^{\circ}\text{C}$  was reported to be 55 mass %. The atom % solubility calculated by the compilers is 68 at %.

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The amalgams were prepared by dissolution of indium in mercury under a nitrogen atmosphere. Potentials of the cell,

Hg,  $Hg_2Cl_2$ ,  $NaCl | In(ClO_4)_3 | In(Hg)$ 

were measured. The plot of EMF against the logarithm of amalgam concentration showed a breakpoint at saturation.

#### SOURCE AND PURITY OF MATERIALS:

Indium was 99.97% pure from Indium Corp. of America.

Mercury was distilled.

Other chemicals were analytically pure from Merck, or they were recrystallized before use.

# ESTIMATED ERROR:

Soly: nothing specified; precision

probably no better than + 1%

(compilers).

Temp: nothing specified.

# COMPONENTS: (1) Indium; In; [7440-74-6] (2) Mercury; Hg; [7439-97-6] VARIABLES: Tr. Inst. Khim. Nauk Akad. Nauk Kaz. SSR 1962, 9, 71-80. PREPARED BY: C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Solubility of indium in mercury:

<u>t/°C</u>	Soly/at %
20	68.0
50	73.3
80	80.0

Similar measurements at -1.5 to  $14^{\circ}\text{C}$  gave unreliable results, probably because of slow equilibration at lower temperatures.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Amalgams were prepared by dissolution of various amounts of indium in mercury. Potentials of the cell,

 $In(Hg) | 0.1 \text{ mol dm}^{-3} In(ClO_4)_3$ , 0.9 mol dm $^{-3}$  NaClO<sub>4</sub> | NaCl,  $Hg_2Cl_2$ , Hg

were determined. The solutions were protected from oxidation with a stream of pure nitrogen. The plot of EMF against the logarithm of the amalgams concentration showed a breakpoint at saturation.

# SOURCE AND PURITY OF MATERIALS:

The salts were twice recrystallized.

Mercury was purified chemically, then twice distilled.

Indium was 99.999% pure.

#### ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision  $\pm$  0.2 K.

Indium 111

#### COMPONENTS:

- (1) Indium; In; [7440-74-6]
- (2) Mercury; Hg; [7439-97-6]

# ORIGINAL MEASUREMENTS:

Eggert, G.L.

Trans. ASM 1962, 55, 891-97.

#### VARIABLES:

#### PREPARED BY:

Temperature: (-36)-141°C

C. Guminski; Z. Galus

#### **EXPERIMENTAL VALUES:**

The author determined the complete phase diagram and reported numerical values only for the eutectics at -37.4 and  $-30.8^{\circ}$ C (at 34.0 and 61.7 at %, respectively), and for the congruent melting points at -14.6 and  $-19.2^{\circ}$ C (at 16.7 and 50.0 at % In, respectively). The following data points were read from the phase diagram by the compilers:

Soly/at %	t/°C	Soly/at %	t/°C	Soly/at %	t/°C	Soly/at %	<u>t/°C</u>
1.0	-35.6	22.8	-19.0	44.7	-21.0	70.0	26.9
3.0	-29.5	24.0	-20.7	47.6	-22.7	72.8	38.8
5.0	-25.7	24.8	-21.3	52.4	-20.0	79.8	70.0
8.0	-22.0	27.4	-25.8	54.0	-21.8	82.7	90.0
10.0	-18.0	29.0	-28.6	57.3	-23.6	83.2	93.5
12.0	-16.7	31.3	-32.6	58.7	-25.0	84.3	105.2
15.0	-14.8	35.0	-34.4	59.2	-27.9	86.8	119.0
17.4	-14.7	37.0	-29.7	60.0	-28.9	88.8	132.4
18.4	-14.7	39.0	-27.1	63.5	-17.1	93.0	141.2
20.0	-16.2	42.6	-22.7	66.8	+5.0		

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Weighed quantities of the metals were mixed at room temperature in glass tubes. The latter were inserted inside a larger glass tube jacket and the space between tubes was packed with Cu wool. The assembly was immersed in a mixture of dry-ice and trichloroethylene to obtain cooling curves; temperatures were determined with a calibrated copper-constantan thermocouple inserted into the liquid amalgam and the data were recorded on a strip-chart recorder. Precise thermopotentials at occurrences on the cooling curves were measured with a precision potentiometer. Low temperature microscopy was observed on a microscope stage upon repeated melting and freezing.

# SOURCE AND PURITY OF MATERIALS:

Indium was 99.98% pure from Indium Corp. of America.

Mercury purity was 99.9995%.

#### ESTIMATED ERROR:

Soly: nothing specified. Temp: precision  $\pm$  0.3 K.

- (1) Indium; In; [7440-74-6]
- (2) Mercury; Hg; [7439-97-6]

#### ORIGINAL MEASUREMENTS:

Chiaranzelli, R.V.; Brown, O.L.I.

J. Chem. Eng. Data 1962, 7, 477-78.

#### VARIABLES:

PREPARED BY:

Temperature: (-37)-11°C

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The liquidus temperatures of the saturated indium amalgams were determined:

ı									
ļ	mass % Hg	at % In <sup>a</sup>	<u>t/°C</u>	mass % Hg	at % Ina	<u>t/°C</u>	mass % Hg	at % In <sup>a</sup>	<u>t/°C</u>
	99.95	0.087	-36.7	96.00	6.79	-17.4	82.35	27.24	-22.0
Ì	99.90	0.17	-35.3	94.63	9.02	-16.0	79.02	31.69	-28.3
1	99.80	0.35	-34.0	93.21	11.30	-12.3	76.89	34.43	-32.0
	99.56	0.77	-31.6	91.85	13.42	-12.0	75.32	36.41	-30.0
	99.21	1.37	-29.4	91.25	14.35	-12.4	69.22	43.72	-19.1
	98.90	1.91	-26.0	90.70	15.19	-12.8	66.15	47.21	-17.0
	98.73	2.25	-24.0	90.40	15.75	-14.1	63.66	43.74	-16.6
	98.70	2.30	-25.3	90.07	16.15	-13.0	58.91	54.93	-19.2
	98.60	2.46	-24.5	88.98	16.33	-13.9	54.03	59.79	-25.0
	98.37	2.81	-23.5	87.54	19.92	-14.6	51.53	62.17	-27.6
	98.03	3.39	-24.7	87.06	20.54	-16.0	49.99	63.61	-28.6
İ	97.49	4.30	-19.7	85.82	22.40	-17.0	48.02	65.41	-3.5
	96.97	5.18	-21.0	84.50	24.27	-18.0	46.03	67.20	+10.8
i	i								

<sup>&</sup>lt;sup>a</sup>by compilers.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Weighed portions of the metals were mixed in Pyrex test tubes, and generally heated and cooled while stirring for several cycles. Some of the alloys were covered with mineral oil, but no oxidation was noticeable on unprotected samples. Heating and cooling curves were observed with a calibrated, glass-sheathed copperconstantan thermocouple.

# SOURCE AND PURITY OF MATERIALS:

Mercury was 99.9999% pure.

Indium was 99.9995% pure.

# ESTIMATED ERROR:

Soly: precision better than  $\pm$  0.7%.

Temp: precision  $\pm$  0.02 K.

# 

#### EXPERIMENTAL VALUES:

The data were presented as a phase diagram. The following numerical liquidus data were reported:

t/°C	at % In
-13.2	16.6
-36.8	33.3
-18.5	50.0
-30.0	62.7

The results at the higher temperatures show excellent agreement with (1).

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Appropriate amounts of both metals were melted in a closed glass container, and cooling curves were recorded with calibrated alcohol and mercury thermometers. Samples of amalgams were analyzed by unspecified method.

# SOURCE AND PURITY OF MATERIALS:

Indium was specified as being of high purity.

Mercury was treated with H<sub>2</sub>SO<sub>4</sub> then triply-distilled under vacuum.

#### ESTIMATED ERROR:

Nothing specified.

- 1. Spicer, W.M.; Banick, C.J.
  - J. Am. Chem. Soc. 1953, 75, 2268.

- (1) Indium; In; [7440-74-6]
- (2) Mercury; Hg; [7439-97-6]

#### ORIGINAL MEASUREMENTS:

Coles, B.R.; Merriam, M.F.; Fisk, Z. J. Less-Common Met. 1963, 5, 41-48.

#### **VARIABLES:**

#### PREPARED BY:

Temperature: (-37)-143°C

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The phase diagram for the In-Hg system was presented, and numerical values were reported only for the congruently melting, peritectic and eutectic points. These points were as follows:

at % In 14.2±0.2<sup>a</sup>

34.7±0.2<sup>b</sup> 50.0±0.2<sup>a</sup>
-37.2±0.2 -19.3±0.2

61.5±0.4<sup>b</sup> 86.6<sup>c</sup>

t/°C

-14.2±0.2 -37.2±0.2

31.0±0.2

108±1

Other liquidus points were read from the phase diagram by the compilers:

at % In	t/°C	at % In	t/°C	at % In	t/°C	at % In	t/°C
0.9	-34.9	18.2	-16.2	58.7	-25.3	89.3	118.5
1.8	-32.3	20.0	-18.5	60.8	-28.9	91.4	125.9
2.8	-28.5	25.1	-23.6	62.3	-27.9	92.9	132.3
5.9	-21.0	29.7	-29.5	64.6	-11.0	93.6	134.3
7.4	-18.7	33.0	-35.1	67.6	+13.8	94.1	136.4
9.6	-16.0	36.2	-35.1	80.5	81.5	95.9	142.8
11.1	-15.5	40.5	-27.7	84.2	98.0		
12.4	-15.2	44.1	-22.6	86.9	109.7		
16.5	-15.1	54.2	-20.2	88.3	114.3		

<sup>&</sup>lt;sup>a</sup>Congruent melting point.

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Desired quantities of each metal to yield 50--100 g of a given amalgam were melted and stirred in an alumina crucible exposed to air. Temperature of the melt was determined with a calibrated, glass-sheathed, copper-constantan thermocouple which was inserted into the alloy during the determination of the heating and cooling curves. X-ray diffraction data, using CuK $\alpha$  radiation, were obtained to identify crystal phases.

#### SOURCE AND PURITY OF MATERIALS:

Indium from Indium Corp. of America was better than 99.999% pure.

Mercury, "Vacumetal" from Metal Salts Corp., was better than 99.999% pure.

# ESTIMATED ERROR:

Soly: precision  $\pm$  1%. Temp: precision  $\pm$  0.2 K.

bEutectic point.

CPeritectic point.

(1) Indium; In; [7440-74-6]

(2) Mercury; Hg; [7439-97-6]

ORIGINAL MEASUREMENTS:

Cusack, N.; Kendall, P.; Fielder, M. Phil. Mag., Ser. 8, 1964, 10, 871-82.

VARIABLES:

PREPARED BY:

Temperature: (-37)-(-16)°C

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The data were presented only as the liquidus curve for the In-Hg system. The data points were read off the curve by the compilers:

Soly/at %	t/°C_	Soly/at %	t/°C	Soly/at %	t/°C
2.3	-31	34.7	-37	57.0	-20
6.0	-21	37.3	-30	61.0	-25
14.0	-15.5	40.5	-26	63.0	-30
22.0	-20	47.0	-20.5	64.0	-20
31.5	-30	51.3	-18.5		

# AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Amalgams were prepared, presumably, by weighing desired quantities of each metal with subsequent mixing and alloying in vacuo. The freezing points were obtained from cooling curves.

# SOURCE AND PURITY OF MATERIALS:

Indium from BDH and from L. Light and Co. was 99.999% pure.

Mercury was purified by multiple distillation and had only  $10^{-4}\ \mathrm{mass}\ \%$  of impurities.

# ESTIMATED ERROR:

Soly: nothing specified. Temp: nothing specified.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Indium; In; [7440-74-6]	Morawietz, W.
(1) Indium; In; [7440-74-6] (2) Mercury; Hg; [7439-97-6]	Chem. Ing. Tech. <u>1964</u> 36, 638-45.
(2) notoury, ng, [/437-7/-0]	000000 1000 10000 1704 00, 030-43.
VARIABLES:	PREPARED BY:
Temperature	C. Guminski; Z. Galus
EXPERIMENTAL VALUES:	
The results were presented as a phase diagra	m. The indium solubility at room
temperature was reported to be 120 parts In/ corresponding atomic % solubility calculated	100 parts Hg by mass. The by the compilers is 67.7 at %.
corresponding acoust a correspond carearated	· ····································
	1
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
The alloys were obtained by electro-	Indium was stated as being of high
reduction, and thermal analysis curves	purity.
were recorded. Detailed description of the method was not specified.	Mercury purity not specified.
	ESTIMATED ERROR:
	Nothing specified.
	DEFEDENCYS.
	REFERENCES:
Ī	1

Indium 117

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Indium; In; [7440-74-6] (2) Mercury; Hg; [7439-97-6]	<ol> <li>Kozin, L.F.; Sudakov, V.A.         <i>Izv. Akad. Nauk Kaz. SSR, Ser. Khim.</i> <u>1970</u>, No. 1, 50-5.</li> <li>Same authors.         <i>Izv. Akad. Nauk SSSR, Metally</i> <u>1970</u>, No. 5, 197-201.</li> </ol>
VARIABLES:	PREPARED BY:
Temperature: (-37)-140°C	C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The data were presented graphically as a partial phase diagram in (1). The complete phase diagram was presented in (2), and numerical data were presented for the congruently melting, eutectic, and peritectic points. The experimental liquidus points were read off the curve in (1) by the compilers. The phase diagram from (2) is presented in the critical evaluation, Fig. 1.

<u>t/°C</u>	at % In	Ref.
-15.0	14.8	[2]
-37.5	35.0	[2]
-18.6	50.0	[2]
-31.5	63.0	[2]
97.0	84.8	[1]
105	85.0	[2]
105.2	87.2	[1]
118.7	89.5	[1]
127	93.0	[1]
139.6	95.6	[1]

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Details of the procedure were not described in (1), but it was probably identical to that in (2). The amalgams were prepared by precisely weighing the metals in an atmosphere of dry carbon dioxide, then the samples were sealed in glass tubes. The melting points were obtained from cooling curves; the temperatures were determined with Pt, Pt-Rh calibrated thermocouples.

# SOURCE AND PURITY OF MATERIALS:

Indium was 99.999% pure.

Mercury was specified as "R-O".

#### ESTIMATED ERROR:

Soly: precision  $\pm$  0.01% in (2). Temp: precision  $\pm$  0.5 K in (2).

118 Indium

# ORIGINAL MEASUREMENTS: COMPONENTS: (1) Indium; In; [7440-74-6] Franck, G. (2) Mercury; Hg; [7439-97-6] Tech.-Wiss. Abh. Osram Gesel. 1973, 11, 101-105. Z. Naturforsch., A 1971, 26, 150-3. VARIABLES: PREPARED BY: Temperature: 80-130°C C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The data were reported graphically as a liquidus curve. The following points on the liquidus were read off by the compilers:

<u>t/°C</u>	at % Hg	at % In
130	7.5	92.5
120	9.7	90.3
110	12.5	87.5
100	15.2	84.8
90	18.0	82.0
80	19.6	80.4

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Method of preparation of alloys was not described. The alloy, in the form of cubes approximately 1 mm3, was vacuumsealed in a Supracil silica cuvette. The vapor pressure of the alloys was determined as a function of temperature by measuring the Hg 2537  $\hbox{\normalfont\AA}$  resonance line absorption, and comparing that for the alloy vapor against that of pure Hg to eliminate the effect of Doppler line broadening in the absorption. The freezing point of the alloy was determined as the breakpoint in the relationship of the optical absorption as a function of temperature.

# SOURCE AND PURITY OF MATERIALS:

Mercury was specified as being of high purity.

Indium purity was not specified.

#### ESTIMATED ERROR:

Temp: nothing specified.

Composition: precision  $\pm$  0.3%

(compilers).

- (1) Thallium; T1; [7440-28-0]
- (2) Mercury; Hg; [7439-97-6]

#### **EVALUATOR:**

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

# CRITICAL EVALUATION:

Tammann (1) reported the first solubility study in the T1-Hg system; he observed that the addition of 0.469 at % T1 into mercury depressed the freezing point of Hg by 0.81 K. The fact that thallium has a high solubility in mercury near room temperature was indicated by an early potentiometric study by Spencer (2) who reported a solubility of 41.5 at % at 291 K. Sucheni (3) also reported an early potentiometric study at 273 and 310 K, and he observed that amalgams which contained more than 43 at % T1 are heterogeneous at 310 K; his solubility of 28 at % at 273 K is too high as compared to later works.

Kurnakov and Pushin (4) were the first to report a phase diagram for this system. However, their thermoanalytical determination of the liquidus in the range of 8 to 40 at % T1 did not agree with later works by other more accurate measurements. Pushin (5) subsequently redetermined and corrected the liquidus in the range of 19.1 to 39.5 at % T1. The measurements of Pavlovich (6) were in agreement with (4) in the range of 0-8 and 40-100 at % T1, but the former author showed that the maximum in the liquidus occurred at 29 at % T1 and 288 K, as compared to 33.33 at % T1 found by (4). Roos (7) also determined the phase diagram for this system from a detailed study which took into account the effect of impurities; he found the first eutectic at 214.2 K at 8.56 at % and the second at 273.78 K at 40.0 at % Tl. Roos found that the coordinates for the maximum in the liquidus were 28.6 at % T1 at 287.6 K. Richards and Daniels (8) and Richards and Smyth (9) applied thermal analysis and potentiometry to confirm the results of Roos; however, Richards et al. found slightly higher solubilities at lower temperatures and slightly lower solubilities at higher temperatures as compared to Roos. Kozin (10) employed potentiometric measurements at 298 to 353 K and found that the solubility of thallium increased from 42.6 to 53.2 at % in this temperature range; these results were in agreement with the earlier measurements. Claire and Rey (11) verified parts of the T1-Hg phase diagram in the thallium-rich region. Moser (12), without presenting experimental detail, reported the eutectics at 213.2 and 272.4 K at 8.5 and 40.0 at % T1, respectively; Siede (13) also found the first eutectic at 8.5 at % T1, but at 214.8 K. Resistivity measurements performed by Schulz and Spiegler (14) confirmed the melting temperature of T12Hg5 at 287.7 K.

Without presenting details of his density measurements of Tl amalgams, Kanda (15) reported a solubility of 42 at % Tl at 296 K. Strachan and Harris (16) reported only that the solubility is higher than 13.1 at % at room temperature. Kozin's (17) predicted solubility of 34.6 at % at 298 K is too low. Zebreva and coworkers (18) determined a solubility of 44.0 at % at 298 K by thermometric titration; this value is slightly too high.

Richter and Pistorius investigated the effect of pressure on the congruently melting point  $(Tl_2Hg_5)$  (19) and on the eutectic points (20), and these authors observed that the above temperatures increased nearly linearly with increasing pressure up to approximately 30 kbar. Based on these measurements, liquidus lines for the Tl-Hg system were presented for the pressure range of 0 to 50 kbar.

The phase diagram for the T1-Hg system is shown in Fig. 1 (21).

COMPONENTS:

(1) Thallium; T1; [7440-28-0]

(2) Mercury; Hg; [7439-97-6]

C. Guminski; Z. Galus
Department of Chemistry
University of Warsaw
Warsaw, Poland
July, 1985

CRITICAL EVALUATION: (continued)

Recommended Solubility of Thallium in Mercury

<u>T/K</u>	Soly/at %	Reference
214	8.5 <sup>a</sup>	6,7,12,13,20
245	12	7
274	19	5,6,8
274	40 <sup>a</sup>	7,20
288	28.6	5,6,7,14,19
293	42 <sup>b</sup>	2,8,15
298	42.7 <sup>b</sup>	10,18,15
323	47 <sup>C</sup>	4,8,10
373	56 <sup>c</sup>	4,6
473	76 <sup>c</sup>	4,7,11
573	99 <sup>c</sup>	9,11

aeutectic point.

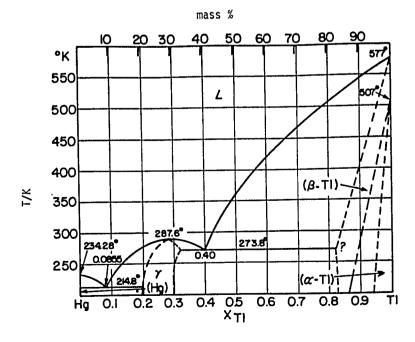


Fig. 1. Hg-T1 system (21)

(Continued next page)

baverage value of data from cited references.

 $<sup>^{\</sup>mathrm{c}}$  interpolated from data of cited references.

- (1) Thallium; T1; [7440-28-0]
- (2) Mercury; Hg; [7439-97-6]

#### **EVALUATOR:**

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

CRITICAL EVALUATION: (continued)

#### References

- Tammann, G. Z. Phys. Chem. <u>1889</u>, 3, 443. Spencer, J. F. Z. Elektrochem. <u>1905</u>, 11, 681.
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- Moser, H. Phys. Z. 1936, 37, 885. 12.
- 13. Siede, B. Metall 1963, 17, 1031.
- 14. Schulz, L.G.; Spiegler, P. Trans. Metall. Soc. AIME 1959, 215, 87.
- 15.
- Kanda, F.A. U.S. At. Energ. Comm. Rep., NYO-2731-4, 1967.
  Strachan, J.F.; Harris, N.L. J. Inst. Metals 1956-57, 85, 17. 16.
- Kozin, L.F. Fiziko-Khimicheskie Osnovy Amalgamnoi Metallurgii, Nauka, Alma-Ata, 1964.
- Zebreva, A.I.; Filippova, L.M.; Omarova, N.D.; Gayfullin, A.Sh. Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol. 1976, 19, 1043. 18.
- Richter, P.W.; Pistorius, C.W.F.T. J. Less-Common Metals 1972, 29, 217.
- 20. Richter, P.W.; Pistorius, C.W.F.T. Acta Met. 1973, 21, 391.
- Hultgren, R.; Desai, P.D.; Hawkins, D.T.; Gleiser, M.; Kelley, K.K. Selected Values of the Thermodynamic Properties of Binary Alloys, Am. Soc. Met., Metals Park, OH, 1973, p. 990.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thallium; T1; [7440-28-0] (2) Mercury; Hg; [7439-97-6]	Tammann, G.  Z. Phys. Chem. <u>1889</u> , 3, 443-9.
VARIABLES: Temperature: 234 K	PREPARED BY: C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Depression of the melting point of mercury,  $\Delta T$ , was determined after addition of small amounts of thallium:

ΔT/K	mass % Tl	at % Tl <sup>a</sup>
0.01	0.034	0.034
0.18	0.079	0.078
0.30	0.143	0.141
0.35	0.226	0.222
0.62	0.395	0.388
0.81	0.480	0.469

aby compilers.

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The melting temperature were determined, but absolute values not given. Details of experiment not specified, therefore, compilers assume that  $\Delta T/K$  in the above table is based on the melting point of Hg of 234.28 K (1).

# SOURCE AND PURITY OF MATERIALS:

Nothing specified.

#### ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision + 0.05 K.

#### REFERENCES:

 Hultgren, R.; Desai, P.D.; Hawkins, D.T.; Gleiser, M.; Kelley, K.K. Selected Values of the Thermodynamic Properties of Binary Alloys, Am. Soc. Met., Metals Park, OH, 1973, p. 990.

# 

#### EXPERIMENTAL VALUES:

Crystallization temperatures of saturated thallium amalgams were reported for two series of measurements.

# Series I

t/°C	at % T1	t/°C	at % T1	t/°C	at % T1
-40	1.0	15	28.8	68	50.7
-48	4.9	15	29.1	116	58.9
-60	8.0	14	31.7	155	68.4
-16	14.6	13.5	32.8	221	82.8
+ 1	18.1	12	33.8	261	90.8
4	19.9	7	38.0	276	95.0
8	21.0	6	39.2	285	97.0
11	23.5	29	44.2	297	99.0
14	25.0				

#### Series II

t/°C	at % T1	t/°C	at % T1
13.5	25.8	13.9	32.4
14.6	27.6	13.5	33.0
14.8	28.7	12.9	33.7
14.8	29.7	12.0	34.7
14.6	30.7	11.0	35.6
14.3	31.6	9.8	36.5

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The amalgams were obtained by mixing the two metals, with heating if needed, and the cooling curves were recorded with the use of thermoelement. For 0-20% T1, the heating curves also were recorded. The alloys were protected against oxidation with vaseline.

# SOURCE AND PURITY OF MATERIALS:

Pure thallium from Kahlbaum.

Mercury purity not specified.

# ESTIMATED ERROR:

Soly: precision  $\pm$  1%. Temp: precision  $\pm$  1 K.

# COMPONENTS: (1) Thallium; T1; [7440-28-0] Roos, G.D. (2) Mercury; Hg; [7439-97-6] Z. Anorg. Chem. 1916, 94, 358-70. VARIABLES: Temperature: (-59)-261°C PREPARED BY: C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Crystallization temperatures of thallium amalgams determined in four series:

# Kahlbaum T1 under CO2 atmosphere

t/°C	at % Tl	t/°C	at % Tl	t/°C	at % Tl	t/°C	at % T1
-43.4	2.43	-28.4	12.06	14.40	18.90	5.00	37.90
-46.5	4.2	-10.0	14.9	14.22	29.90	0.62	40.0
-47.0	5.4	11.5	23.0	14.14	30.20	75.5	50.68
-53.0	7.0	13.20	25.1	13.66	31.20	138.0	62.65
-59.0	8.56	13.40	25.9	12.75	32.50	183.5	72.24
-51.0	9.1	14.05	27.10	11.95	33.33	222.0	81.54
-45.8	9.8	14.37	28.10	9.90	35.00	261.5	90.31
-38.4	10.5						

#### II. Kahlbaum Tl under petroleum

t/°C	at % Tl	_t/°C	at % T
14.0	41.25	12.85	32.3
2.4	40.5	13.75	31.2
4.40	38.2	14.30	29.8
8.50	36.0	14.45	28.4
10.90	34.1	14.30	27.6

# III. Thou Tl in CO2 atmosphere

t/°C	at % T1	t/°C	at % T1
9.5	35.2	14.0	29.9
11.8	33.33	14.18	29.1
12.6	32.1	14.25	28.3
13.2	31.45	14.10	27.7
13.58	30.8		

# IV. Electrolytic Tl in CO2 atmosphere

t/°C	at % Tl	t/°C	at % T1
13.40	31.70	14.40	27.36
14.41	29.50	13.95	26.25
14.53	28.56	12.55	24.18

Author found that Thöl Tl contained small amounts of Pb, resulting in decreased M.P. for  ${\rm Tl}_2{\rm Hg}_5$ . Therefore, results with Kahlbaum and electrolytic Tl were recommended.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Weighed quantities of the metals were mixed and cooling curves were determined with either a mercury thermometer or thermocouples. The amalgams were protected against oxidation with either petroleum or pure, dry CO<sub>2</sub>.

# SOURCE AND PURITY OF MATERIALS:

Pure thallium from A. Thol and from Kahlbaum, and electrolytically prepared by the authors.

# ESTIMATED ERROR:

Soly: nothing specified. Temp: precision  $\pm$  0.01 K.

- (1) Thallium; T1; [7440-28-0]
- (2) Mercury; Hg; [7439-97-6]

# ORIGINAL MEASUREMENTS:

Richards, T.W.; Daniels, F.

J. Am. Chem. Soc. 1919, 41, 1732-68.

#### VARIABLES:

# Temperature: (-6.5)-40°C

#### PREPARED BY:

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Freezing points of amalgams determined thermometrically:

27.24

20.31

17.97

16.65

t/°C	Series I mass % Tl	at % Tl
+1.6	42.8	42.3
5.3	38.8	38.3
12.0	34.0	33.5
13.9	31.7	31.3
14.9	29.1	28.7
14.3	26.4	26.0
12.3	24.2	23.8
3.0	19.5	19.2

	Series II	
<u>t/°C</u>	mass % Tl	at % Tl
9.2	36.5	36.0
11.7	34.4	33.9
14.1	31.5	31.1
14.8	29.0	28.6
13.2	25.4	25.0
11.5	23.8	23.4
4.0	20.0	19.7

#### Series III mass % Tl t/°C at % T1 +0.9 40.90 40.47 5.9 38.83 38.37 9.5 37.19 36.71 12.8 32.63 32.31

27.60

20.63

18.27

16.92

from EN	IF measi	irements
Freezing	points	determined

<u>t/°C</u>	mass % T1	at % T1
20.00	43.3	42.8
30.00	44.5	44.0
40.00	45.8	45.3

#### AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

14.3

5.7 -0.9

-6.5

Amalgams were prepared by mixing weighed amounts of Hg and Tl in a closed tube which contained acid of known concentration. The acid neutralized any  $Tl_2O$  on the metal, and the net Tl was determined by back-titration of the acid with standard alkali. The clean amalgam was removed from the tube under a  ${\rm H}_{2}$  atmosphere and used for the various measurements. The freezing points in Series I and II were made on small amounts of concentrated amalgams contained in a small glass bulb with a thermometer placed in the bulb; the freezing point was determined by plunging the bulb in cold water. Series III was determined on larger amounts of amalgam with a Beckmann freezing point apparatus. In the EMF method, the potential of the cell,

was determined at a fixed temperature at increasing Tl concentration. At the saturation point the EMF attained constant reading.

## SOURCE AND PURITY OF MATERIALS:

Thallium from various sources was transformed into  $Tl_2SO_4$ , the latter recrystallized more than 3 times after contact with very pure, electrolytic Tl, then the pure Tl was prepared by electrolysis of the sulfate solution which also contained  $(NH_4)_2C_2O_4$ . Mercury was purified with  $H_2SO_4$ , then with  $Hg_2(NO_3)_2$ - $HNO_3$  mixture, then vacuum distilled and finally distilled under hydrogen.

## ESTIMATED ERROR:

Composition: precision better than  $\pm$  0.3%.

Temp: precision of thermal analysis better than + 0.1 K; EMF: + 0.01 K.

#### COMPONENTS:

(1) Thallium; T1; [7440-28-0]

(2) Mercury; Hg; [7439-97-6]

#### ORIGINAL MEASUREMENTS:

Richards, T.W.; Smyth, C.P.

J. Am. Chem. Soc. 1922, 44, 524-45.

#### VARIABLES:

#### PREPARED BY:

Temperature: 231-300°C

C. Guminski; Z. Galus

#### **EXPERIMENTAL VALUES:**

Freezing points of thallium amalgams were presented graphically; the mass % data were read from the graph by the compilers and recalculated to at %:

	H	g
t/°C	mass %	at %
299.5	1.0	1.0
295.5	2.3	2.3
292	3.2	3.1
289.5	3.6	3.5
287.5	4.2	4.1
283.0	5.4	5.3
277.5	6.9	6.8
272.5	7.7	7.6
257.5	11.3	11.1
244.5	14.5	14.3
232.0	17.5	17.2

	Н	g
t/°C	mass %	at %
276	7.8	7.7
272	8.8	8.7
264.5	10.5	10.3
257.5	12.0	11.8
252.3	13.5	13.3
246.5	15.0	14.8
238.5	16.5	16.2
231.5	18.2	17.9

# AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

The amalgams were prepared by mixing weighed quantities of the metals in an earthenware dish; the mixture was covered with a layer of paraffin, and the amalgam formed by gently heating the dish. Cooling curves were determined in a large glass tube with a thermometer inserted into the amalgam. The amalgams were analyzed by decomposing with standardized acid and back titration of the acid with standard alkali.

# SOURCE AND PURITY OF MATERIALS:

Crude Tl was purified by treatment with dil. H<sub>2</sub>SO<sub>4</sub>, filtered, and TlCl precipitated from the filtrate with dil. HCl. The TlCl was converted to the sulfate and recrystallized at least twice. Tl was electrolytically prepared as a sponge from the aqueous sulfate solution, then fused and filtered through a capillary as bright metal.

Hg was purified with  $Hg_2(NO_3)_2$ , then distilled.

#### ESTIMATED ERROR:

Temp: precision  $\pm$  0.1 K.

Precision of chemical analysis:  $\pm$  0.2%.

# 

#### EXPERIMENTAL VALUES:

Crystallization temperatures of thallium amalgams were reported:

t/°C	at % Hg	±/°C	at % Hg
2.6	60.5	14.5	71.4
4.3	61.4	14.4	72.2
6.9	62.8	14.3	72.8
8.8	64.0	14.0	73.6
10.2	65.0	13.4	74.6
11.7	66.2	12.5	75.5
12.8	67.5	11.5	76.4
13.8	68.8	9.1	77.9
14.2	69.8	7.8	78.6
14.4	70.3	5.2	79.7
14.5	71.0	2.6	80.9

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Thermal analysis was utilized to determine the crystallization temperatures, but experimental details were not given. The method was probably similar to, or an improved version of, that in (1).

# SOURCE AND PURITY OF MATERIALS:

Nothing specified.

# ESTIMATED ERROR:

Soly: nothing specified.

Temp: nothing specified; probably + 0.1 K (compilers).

#### REFERENCES:

Kurnakov, N.S.; Pushin, N.A.
 Anorg. Chem. 1902, 30, 86.

COMPONENTS:

(1) Thallium; T1; [7440-28-0] Schulz, L.G.; Spiegler, P.

(2) Mercury; Hg; [7439-97-6] Trans. Metall. Soc. AIME 1959, 215, 87-90.

VARIABLES:

One temperature PREPARED BY:

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

The melting point of the congruently melting compound,  ${\rm Tl}_2{\rm Hg}_5$ , was confirmed to be 14.5°C. The solubility of Tl at this temperature, therefore, is 28.6 at % (compilers).

# AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

The alloys of composition, 28.6  $\pm$  0.2 at % T1, were prepared either by directly mixing weighed amounts of the metals in the measurement cell or by premixing the metals then loading the amalgam into the cell under vacuum. The cell consisted of two Teflon-cup reservoirs connected at the end of a capillary tube in which were placed the thin electrodes. Of several metals used for the electrodes, nickel gave the most uniform results. The specific resistivity of the amalgam was obtained by comparing the resistance of the amalgam against that of pure Hg in the same cell. The melting point of Tl<sub>2</sub>Hg<sub>5</sub> was obtained by measuring the resistance of the liquid amalgam as the temperature was decreased from 24°C to lower temperatures. There was a linear decrease in resistance with decreasing temperature down to 16°C, then at temperatures below 14.5°C the resistance remained constant.

# SOURCE AND PURITY OF MATERIALS:

Mercury of "triply distilled quality" from Bethlehem Apparatus Co., Inc.

Thallium purity not specified.

#### ESTIMATED ERROR:

Precision of chemical analysis: ± 1%.

Temp: precision + 0.2 K.

- (1) Thallium; T1; [7440-28-0]
- (2) Mercury; Hg; [7439-97-6]

# ORIGINAL MEASUREMENTS:

Kozin, L.F.

Tr. Inst. Khim. Nauk. Akad. Nauk Kaz. SSR 1962, 9, 71-80.

#### VARIABLES:

# Temperature: 25-80°C

## PREPARED BY:

C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Solubility of thallium in mercury:

<u>t/°C</u>	Soly/at %
25	42.6
40	46.7
60	49.8
80	53.2

Measurements at 5 and  $15^{\circ}\text{C}$  also were made, but results were practically identical to that at  $25^{\circ}\text{C}$ .

# AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

The amalgams were obtained by dissolution of thallium in mercury. The potentials of the cell,

 $T1(Hg)_x | T1C10_4$  (0.1 mol dm<sup>-3</sup>) + NaC10<sub>4</sub> (0.9 mol dm<sup>-3</sup>) | NaC1, Hg<sub>2</sub>C1<sub>2</sub>, Hg

were determined. Amalgams were protected from oxidation by passing pure nitrogen over the cell. The saturation point corresponded to any inflection in the curve relating cell EMF to log of Tl concentration.

#### SOURCE AND PURITY OF MATERIALS:

Salts were recrystallized twice. Mercury was purified chemically and double distilled. Thallium was 99.999% pure.

#### ESTIMATED ERROR:

Soly: nothing specified.
Temp: precision + 0.2 K.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thallium; T1; [7440-28-0] (2) Mercury; Hg; [7439-97-6]	Richter, P.W.; Pistorius, C.W.F.T.  J. Less-Common Metals 1972, 29, 217-19.
VARIABLES: Pressure	PREPARED BY: C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Melting point of  ${\rm Tl_2Hg_5}$  (28.6 at % T1) was presented graphically as a function of pressure. Experimental points were fitted by equation,

$$t/^{\circ}C = 13.7 + 3.44 P$$

where P is in kbar. Standard deviation was 1.3°C. The data points were read from the curve by the compilers:

P/kbar	T/K	
	a	
0	286.9±0.5 <sup>a</sup>	
2.8	294.9	
3.9	298.5	
5.4	303.5	
7.0	309.4	
8.6	314.4	
10.4	319.7	
11.4	324.3	
12.6	329.6	
13.2	331.1	
22.6	363.7	
27.4	381.1	
28.6	385.5	
30.0	389.9	
30.9	393.7	
32.8	399.0	

 $<sup>^{\</sup>mathbf{a}}$ numerical value is given for atmospheric pressure only.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The metals were weighed and thoroughly mixed at room temperature.  ${\rm Tl}_2{\rm Hg}_5$  obtained was stored at 273 K under nitrogen. Pressure was generated in a piston-cylinder apparatus. The melting points were observed by means of differential thermal analysis; Chromel-Alumel thermocouples were used. The samples were contained in stainless steel or aluminum capsules with no evidence of contamination.

# SOURCE AND PURITY OF MATERIALS:

99.999% pure T1 from Koch-Light.

Triply-distilled mercury from Johnson-Matthey & Co.

#### ESTIMATED ERROR:

Temp: precision + 1 K.

Pressure: precision + 0.5 kbar.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thallium; T1; [7440-28-0] (2) Mercury; Hg; [7439-97-6]	Richter, P.W.; Pistorius, C.W.F.T.  Acta Met. 1973, 21, 391-94.
VARIABLES:	PREPARED BY:
Pressure	C. Guminski; Z. Galus

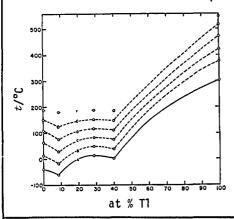
#### EXPERIMENTAL VALUES:

The pressure dependence of the eutectic temperatures was determined and fitted to the equations, where P is in kbar and t in  ${}^{\circ}\mathrm{C}$ :

(I)  $t/^{\circ}C = 60.0 + 4.09 P + 0.0132 P^{2}$  for eutectic at 8.5 at % T1

(II)  $t/^{\circ}C = 0.9 + 3.65 P$  for eutectic at 40 at % T1.

The authors found eutectic temperatures at 1 bar to be -60  $\pm$  1°C and 0.9  $\pm$  0.5°C, respectively, at 8.5 and 40 at % T1. There was only a very slight pressure dependence in eq. (I). The published pressure dependence of the melting points of Hg (1), Hg<sub>5</sub>T1<sub>2</sub> (2), and T1 (3) were used with the eutectic data to construct liquidus curves at various pressures, as shown in the figure. In the construction of liquidus lines it was assumed that the eutectic composition was independent of pressure.



Liquidus lines in the system Hg-Tl at various pressures.

A: atmospheric pressure;

B: 10 kbar;

C: 20 kbar;

D: 30 kbar;

E: 40 kbar;

F: Extrapolated to 50 kbar.

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Thallium and mercury in the eutectic compositions were mixed at room temperature, then stored under  $N_2$ . Samples for measurements were contained in stainless steel capsules, with no evidence of contamination. In order to prevent leakage, the pressure plate was first cooled to well below the eutectic points before pressure was applied by a piston to seal a capsule in situ. Heating and cooling rates in the differential thermal analyses were in the range of  $0.4-1.1^{\circ}\text{C/sec}$ , and temperatures were measured with a Chromel-Alumel thermocouple.

#### SOURCE AND PURITY OF MATERIALS:

Tl: 99.999% pure from Koch-Light.

Hg: triple distilled from JohnsonMatthey Co.

#### ESTIMATED ERROR:

Temp: precision + 1 K.

Pressure: precision + 0.5 kbar.

- Klement, W.; Jayaraman, A.; Kennedy, G.C. Phys. Rev. <u>1963</u>, 131, 1.
- Richter, P.W.; Pistorius, C.W.F.T.
   J. Less-Common Metals 1972, 29, 217.
- Adler, P.N.; Margolin, H. Trans. Met. Soc. AIME 1964, 230, 1048.

132 Ina	illium
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thallium; T1; [7440-28-0] (2) Mercury; Hg; [7439-97-6]	Zebreva, A.I.; Filippova, L.M.; Omarova, N.D.; Gayfullin, A.Sh.  Izv. Vyssh. Ucheb. Zaved., Khim. Khim. Tekhnol. 1976, 19, 1043-6.
VARIABLES:	PREPARED BY:
One temperature: 25°C	C. Guminski; Z. Galus
EXPERIMENTAL VALUES:	
Solubility of thallium in mercury at 25°C was	as reported to be 44.0 at %.
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: The heterogeneous thallium amalgam was	SOURCE AND PURITY OF MATERIALS: "Pure" metals were used.
prepared by mixing weighed amounts of the metals. Heat effects (Q) were recorded when subsequent portions of mercury were added. The inflection point on a plot of Q vs. amalgam concentration corresponds to the solubility of thallium in mercury.	Tale metals were used.
	ESTIMATED ERROR:
	Soly: accuracy no better than a few percent (compilers).
	Temp: not specified.
	REFERENCES:

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Thallium; T1; [7440-28-0] (2) Mercury; Hg; [7439-97-6]	Claire, Y.; Rey, J.  J. Less-Common Metals 1980, 70, 33-8.
VARIABLES: Temperature: 279-556 K	PREPARED BY: C. Guminski; Z. Galus

#### EXPERIMENTAL VALUES:

Liquidus points in the T1-Hg system were determined:

<u>T/K</u>	Soly/at %	_T/K	Soly/at %
279	22.5	491	79.59
282	23.2	'504	83.16
282	36.1	517	86.85
279	37.2	534	90.83
273.5	40.5	551	94.62
457	72.52	556	96.82

Partial molar enthalpy and integral enthalpy of mixing are presented for various temperatures and concentrations.

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The amalgams were prepared by mixing weighed portions of the metals which were contained in evacuated glass ampules. The liquidus were obtained mostly by differential thermal analysis by slowly heating the ampules followed by slow cooling. The liquidus also was determined by microcalorimetry by plotting the enthalpy of mixing against the composition at a fixed temperature; the breakpoint in the curve corresponded to the liquidus temperature, or other phase changes.

# SOURCE AND PURITY OF MATERIALS:

Both metals were of 99.999% purity.

# ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision probably better than

+ 1 K (compilers).