

<p>COMPONENTS:</p> <p>(1) Nickel; Ni; [7440-02-0]</p> <p>(2) Mercury; Hg; [7439-97-6]</p>	<p>EVALUATOR:</p> <p>C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland</p> <p>July, 1985</p>
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CRITICAL EVALUATION:

The solubility of nickel in mercury is very low in the region of room temperature. Although there have been many determinations, the following reported solubilities, expressed in atomic %, have varied over five orders of magnitude: 2×10^{-3} at 290 K (1), 0.5 at 291 K (2), 4.8×10^{-4} at 293 K (3), less than 7×10^{-5} at 293 K (4), less than 7×10^{-6} at 303 K (5), 7×10^{-3} at room temperature (6), 1.7×10^{-5} at room temperature (7), 4.8×10^{-4} at 298 K (8), 4.8×10^{-5} at 293 K (9), 1.6×10^{-4} at 298 K (10,11), 1.0×10^{-4} at 290 K (12,13), 1.4×10^{-5} at room temperature (14), 7×10^{-6} probably at 303 K (15), and 6.7×10^{-5} at 293 K (16). The above determinations were made by various methods: EMF (1), magnetic susceptibility (2), chemical analysis (3-6, 8,9), coulometry (7), voltammetry (10,11), chronoamperometry (12,13,16), chronopotentiometry (14), and pulse polarography (15). The wide variation in the reported solubilities shows that the system is very susceptible to oversaturation. All results, except that of (5) and (15), are too high and are rejected. Kozin's prediction of 1.0×10^{-5} at 298 K is also too high.

There was better agreement of the solubility data at higher temperatures. Epstein (8) reported a nickel solubility of 4.8×10^{-3} at % at 573 K, but no details of the analytical procedure were given. Toner (18) reported that the solubility of nickel increased from 4.3×10^{-5} to 1.09×10^{-2} at % in the temperature range of 401 to 605 K; a break on the solubility vs. temperature curve was observed at 520 K. Jangg and Palman (9) reported that the solubility increased from 4.8×10^{-5} to 2.9×10^{-2} at % in the range of 293-826 K. These authors observed a break in the solubility curve at 498 K. Weeks (19-21) determined the solubility at 773-1023 K and agreed with the high temperature values of Jangg and Palman; at 1023 K the solubility was found to be 0.24 at %. A single determination of the Ni solubility by Parkman and Whaley (22,23), 3.8×10^{-2} at % at 866 K, agrees well with the results of (9, 19-21). The results in (9, 19-23) were presented graphically; the only numerical values presented were 6.1×10^{-2} at % at 923 K (20) and 3.4×10^{-2} at % at 873 K (19).

Barański and Galus (24) explained part of the discrepancies in the nickel solubilities reported by various authors at temperatures below 500 K. The disparities are attributed to the formation of NiHg_2 , NiHg_3 and NiHg_4 , and to the differences in solubilities of these compounds and nickel. The authors state that true equilibrium is attained only for NiHg_4 ; this compound is formed in the last step in the reaction between electrolytically introduced nickel and mercury. The solubilities were determined from potentiometric measurements, and unit activity coefficient of Ni was assumed. Because the activity coefficient in the homogeneous amalgam is most probably less than unity, the nickel solubility would be higher than it would be with the above assumption. The solubilities of the compounds increase in the order $\text{NiHg}_4 < \text{NiHg}_3 < \text{NiHg}_2 < \text{Ni}$, but it should be indicated that the equilibrium with the last two species is unstable. Below 500 K there is good agreement of the NiHg_4 solubility values of (24) and (18).

The existence of NiHg_4 up to 493 K and NiHg_3 up to 483 K have been confirmed, and NiHg_2 is stable to approximately 458 K (4, 24-26). Above 493 K the saturated amalgams are in equilibrium with pure nickel.

(Continued next page)

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

Recommended (r) and tentative values of nickel solubility in mercury:

<u>T/K</u>	<u>Soly/at %</u>	<u>Reference</u>
293	1×10^{-7}	[24]
298	2×10^{-7}	[24]
323	2×10^{-6}	[24]
373	4×10^{-5}	[24]
473	2×10^{-3a}	[18,9]
573	7×10^{-3a}	[18,9]
673	1.5×10^{-2}	[9]
773	2.5×10^{-2}	[9]
873	3.5×10^{-2} (r) ^a	[19,22]
973	5×10^{-2b}	[21], [18,9,19,22]

^aMean value from cited references.

^bExtrapolated value from data of cited references.

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COMPONENTS: (1) Nickel; Ni; [7440-02-0] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: deWet, J.F.; Haul, R.A.W. <i>Z. Anorg. Chem.</i> <u>1954</u> , 277, 96-112.
VARIABLES: One temperature: 303 K	PREPARED BY: C. Guminski; Z. Galus
EXPERIMENTAL VALUES: Solubility of nickel in mercury at 303 K was found to be less than 2×10^{-6} mass %. The corresponding atomic % solubility limit calculated by the compilers is 7×10^{-6} at %. Solid NiHg_4 was reported to be in equilibrium with the saturated amalgam.	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: Heterogenous nickel amalgam was obtained by electrolysis of 1 mol dm^{-3} NiSO_4 solution at the mercury-pool cathode. The amalgam was washed with water, dried with acetone and sealed off under vacuum in a glass tube. A portion of the amalgam was introduced into a centrifuge vessel and after sufficiently long centrifuging the homogenous part of the amalgam was taken for analysis. Mercury was carefully distilled off and the residue was dissolved in 2.8 mol dm^{-3} HCl. The resulting solution was analyzed spectrochemically for nickel content.	SOURCE AND PURITY OF MATERIALS: $\text{NiSO}_4 \cdot 6\text{-}7\text{H}_2\text{O}$ was Analar grade from Hopkins and Williams. Purified mercury was distilled under vacuum and was found to be spectrochemically free from traces of nickel. Water was triply distilled. ESTIMATED ERROR: Soly: analytical detection limit was 2×10^{-6} mass % Ni in the amalgam. Temp: nothing specified. REFERENCES:

COMPONENTS: (1) Nickel; Ni; [7440-02-0] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: Toner, D.F. <i>U.S. At. Ener. Comm. Rep., ORNL-2839, 1959, pp. 187-191.</i>																																										
VARIABLES: Temperature: 128-332°C	PREPARED BY: C. Guminski; Z. Galus																																										
EXPERIMENTAL VALUES: The mass % solubility of nickel in mercury was reported graphically as a function of temperature; the solubility values were read from the plotted data and the corresponding atomic % conversion was calculated by the compilers. <table border="1" data-bbox="408 568 1067 946"> <thead> <tr> <th>$t/^\circ\text{C}$</th> <th>$\text{Soly/mass \%} \times 10^4$</th> <th>$\text{Soly/at \%} \times 10^3$</th> </tr> </thead> <tbody> <tr><td>128</td><td>0.125</td><td>0.043</td></tr> <tr><td>162</td><td>0.90</td><td>0.31</td></tr> <tr><td>173</td><td>2.2</td><td>0.75</td></tr> <tr><td>181</td><td>1.3</td><td>0.44</td></tr> <tr><td>205</td><td>4.2</td><td>1.43</td></tr> <tr><td>227</td><td>9.0</td><td>3.1</td></tr> <tr><td>243</td><td>12</td><td>4.1</td></tr> <tr><td>245</td><td>14</td><td>4.8</td></tr> <tr><td>258</td><td>19</td><td>6.5</td></tr> <tr><td>278</td><td>36</td><td>12.2</td></tr> <tr><td>308</td><td>25</td><td>8.5</td></tr> <tr><td>327</td><td>26</td><td>8.9</td></tr> <tr><td>332</td><td>32</td><td>10.9</td></tr> </tbody> </table>		$t/^\circ\text{C}$	$\text{Soly/mass \%} \times 10^4$	$\text{Soly/at \%} \times 10^3$	128	0.125	0.043	162	0.90	0.31	173	2.2	0.75	181	1.3	0.44	205	4.2	1.43	227	9.0	3.1	243	12	4.1	245	14	4.8	258	19	6.5	278	36	12.2	308	25	8.5	327	26	8.9	332	32	10.9
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METHOD/APPARATUS/PROCEDURE: A Ni specimen was inserted into the isothermal hot zone of a Hg thermal convection loop made of quartz. The sample was mechanically polished with a grit paper or electropolished. The system was operated under a hydrogen atmosphere. A series of thermocouples indicated the temperature profile in the loop. A Hg sample was extracted through a fritted disk and then chemically analyzed. The measurements were performed at various times of Hg circulation in the loop. Constant values of the Ni solubility in Hg were obtained after over 10 hours of equilibration.	SOURCE AND PURITY OF MATERIALS: Nothing specified. ESTIMATED ERROR: Soly: nothing specified; precision probably better than $\pm 20\%$. Temp: nothing specified. REFERENCES:																																										

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VARIABLES: Temperature: 20-553°C	PREPARED BY: C. Guminski; Z. Galus																																																																
EXPERIMENTAL VALUES: The mass % solubility of nickel in mercury was presented graphically as a function of temperature. The data points were read from the curve and converted to atomic % by the compilers. <table border="1" data-bbox="161 576 1136 886"> <thead> <tr> <th rowspan="2">$t/^\circ\text{C}$</th> <th colspan="2">Soly</th> <th rowspan="2">$t/^\circ\text{C}$</th> <th colspan="2">Soly</th> </tr> <tr> <th>mass % x 10^4</th> <th>at % x 10^4</th> <th>mass % x 10^3</th> <th>at % x 10^3</th> </tr> </thead> <tbody> <tr><td>20</td><td>0.14</td><td>0.48</td><td>236</td><td>1.1</td><td>3.8</td></tr> <tr><td>50</td><td>0.36</td><td>1.2</td><td>243</td><td>1.2</td><td>4.1</td></tr> <tr><td>100</td><td>1.1</td><td>3.7</td><td>252</td><td>1.3</td><td>4.5</td></tr> <tr><td>150</td><td>2.6</td><td>8.8</td><td>302</td><td>2.1</td><td>7.2</td></tr> <tr><td>200</td><td>5.0</td><td>17</td><td>353</td><td>3.3</td><td>11</td></tr> <tr><td>225</td><td>6.2</td><td>21</td><td>402</td><td>4.3</td><td>15</td></tr> <tr><td>230</td><td>8.5</td><td>29</td><td>454</td><td>5.7</td><td>20</td></tr> <tr><td>232</td><td>9.4</td><td>32</td><td>503</td><td>7.1</td><td>24</td></tr> <tr><td>234</td><td>10</td><td>34</td><td>553</td><td>8.5</td><td>29</td></tr> </tbody> </table> <p data-bbox="131 907 1009 934">It was reported that NiHg₄ is in equilibrium with the liquid below 225°C.</p> <p data-bbox="131 955 707 982">The results below 225°C appear to be overstated.</p>		$t/^\circ\text{C}$	Soly		$t/^\circ\text{C}$	Soly		mass % x 10^4	at % x 10^4	mass % x 10^3	at % x 10^3	20	0.14	0.48	236	1.1	3.8	50	0.36	1.2	243	1.2	4.1	100	1.1	3.7	252	1.3	4.5	150	2.6	8.8	302	2.1	7.2	200	5.0	17	353	3.3	11	225	6.2	21	402	4.3	15	230	8.5	29	454	5.7	20	232	9.4	32	503	7.1	24	234	10	34	553	8.5	29
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METHOD/APPARATUS/PROCEDURE: The heterogeneous amalgam was introduced into 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 a pressure of purified nitrogen. Usually 3- to 4-fold filtration was necessary. The nickel content was then analytically determined in the filtered amalgam. For experiments carried out below 320°C, amalgam was equilibrated in a glass vessel. The analytical procedures are not described in the paper.	SOURCE AND PURITY OF MATERIALS: Nothing specified. <table border="1" data-bbox="664 1597 1205 1732"> <tbody> <tr> <td colspan="2">ESTIMATED ERROR:</td> </tr> <tr> <td>Soly:</td> <td>precision \pm 5%.</td> </tr> <tr> <td>Temp:</td> <td>precision \pm 2 K.</td> </tr> </tbody> </table> REFERENCES:	ESTIMATED ERROR:		Soly:	precision \pm 5%.	Temp:	precision \pm 2 K.																																																										
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VARIABLES: One temperature: 866 K	PREPARED BY: C. Guminski; Z. Galus
EXPERIMENTAL VALUES: The mass % solubility of nickel in mercury was presented graphically as a function of temperature; the compilers read off a value of 1.1×10^{-2} mass % at 866 K from the curve. The corresponding atomic % solubility calculated by the compilers is 3.8×10^{-2} at %.	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: Specimen of Ni was placed in contact with Hg in a glass capsule. The capsule was sealed under vacuum after at least 16 h outgassing of Hg. The capsule was heated to the desired temperature and held for 16 hr. A sample of the solution was then collected and cooled. Hg was separated from the sample by molecular distillation, and the residue was taken into acid solution, dried and analyzed by emission spectroscopy.	SOURCE AND PURITY OF MATERIALS: Nothing specified. ESTIMATED ERROR: Soly: nothing specified. Temp: precision ± 3 K. REFERENCES:

COMPONENTS: (1) Nickel; Ni; [7440-02-0] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: 1. Weeks, J.R. <i>Corrosion</i> 1967, 23, 98-106. 2. Weeks, J.R.; Fink, S. <i>U.S. At. Ener. Comm. Rep., BNL-900,</i> 1964, p. 136.																										
VARIABLES: Temperature: 500-755°C	PREPARED BY: C. Guminski; Z. Galus																										
EXPERIMENTAL VALUES: The solubility of nickel in mercury as a function of temperature was presented graphically. The data points were read off the plot and recalculated to at % by compilers. <table data-bbox="480 559 864 922" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th style="text-align: center;"><u>t/°C</u></th> <th style="text-align: center;"><u>Soly/at % x 10²</u></th> </tr> </thead> <tbody> <tr><td style="text-align: center;">665</td><td style="text-align: center;">43</td></tr> <tr><td style="text-align: center;">750</td><td style="text-align: center;">24</td></tr> <tr><td style="text-align: center;">500</td><td style="text-align: center;">13</td></tr> <tr><td style="text-align: center;">725</td><td style="text-align: center;">12</td></tr> <tr><td style="text-align: center;">700</td><td style="text-align: center;">9.7</td></tr> <tr><td style="text-align: center;">755</td><td style="text-align: center;">7.3</td></tr> <tr><td style="text-align: center;">655</td><td style="text-align: center;">6.5</td></tr> <tr><td style="text-align: center;">700</td><td style="text-align: center;">5.5</td></tr> <tr><td style="text-align: center;">605</td><td style="text-align: center;">4.5</td></tr> <tr><td style="text-align: center;">550</td><td style="text-align: center;">3.8</td></tr> <tr><td style="text-align: center;">500</td><td style="text-align: center;">2.9</td></tr> <tr><td style="text-align: center;">625</td><td style="text-align: center;">2.4</td></tr> </tbody> </table>		<u>t/°C</u>	<u>Soly/at % x 10²</u>	665	43	750	24	500	13	725	12	700	9.7	755	7.3	655	6.5	700	5.5	605	4.5	550	3.8	500	2.9	625	2.4
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METHOD/APPARATUS/PROCEDURE: Hg and Ni were equilibrated in a quartz capsule consisting of two chambers separated by a sintered quartz filter. The capsule was sealed with the metals in the larger chamber, then the capsule was placed inside of a stainless steel bomb which contained some Hg to equalize the pressure inside of the quartz capsule. The bomb was sealed and placed inside of an electric oven which was mounted on a centrifuge. The sample was equilibrated for 72 hr under stationary condition at the desired temperature; subsequently, the equilibrated amalgam was centrifuged at temperature to filter and separate the liquid phase. After cooling the capsule a known quantity of filtrate was analyzed by distilling off the mercury, the residue dissolved in HF-HNO ₃ or aqua regia and the Ni determined spectrographically.	SOURCE AND PURITY OF MATERIALS: Triple-distilled, reagent grade mercury was used. ESTIMATED ERROR: Soly: nothing specified. Temp: precision ± 2 K. REFERENCES:																										

COMPONENTS: (1) Nickel; Ni; [7440-02-0] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: Barański, A.; Galus, Z. <i>J. Electroanal. Chem.</i> <u>1973</u> , <i>46</i> , 289-305.																																																					
VARIABLES: Temperature: 20-500°C	PREPARED BY: C. Guminski; Z. Galus																																																					
EXPERIMENTAL VALUES: Solubilities of Ni and the compounds NiHg ₄ , NiHg ₃ and NiHg ₂ in mercury at various temperatures. <table border="1" data-bbox="189 543 1254 825" style="margin: 10px auto;"> <thead> <tr> <th rowspan="2">Solute</th> <th colspan="8">Soly/at %</th> </tr> <tr> <th>20°C</th> <th>50°C</th> <th>100°C</th> <th>150°C</th> <th>200°C</th> <th>300°C</th> <th>400°C</th> <th>500°C</th> </tr> </thead> <tbody> <tr> <td>NiHg₄</td> <td>1.5x10⁻⁷</td> <td>1.8x10⁻⁶</td> <td>4.1x10⁻⁵</td> <td>4.7x10⁻⁴</td> <td>3.0x10⁻³</td> <td>-</td> <td>-</td> <td>-</td> </tr> <tr> <td>NiHg₃</td> <td>3.4x10⁻⁶</td> <td>1.9x10⁻⁵</td> <td>1.6x10⁻⁴</td> <td>9.3x10⁻⁴</td> <td>3.6x10⁻³</td> <td>-</td> <td>-</td> <td>-</td> </tr> <tr> <td>NiHg₂</td> <td>2.3x10⁻⁵</td> <td>8.5x10⁻⁵</td> <td><u>4.7x10⁻⁴</u></td> <td><u>1.8x10⁻³</u></td> <td>-</td> <td>-</td> <td>-</td> <td>-</td> </tr> <tr> <td>Ni</td> <td>1.1x10⁻³</td> <td>1.6x10⁻³</td> <td>2.4x10⁻³</td> <td>3.3x10⁻³</td> <td>4.2x10⁻³</td> <td><u>6.1x10⁻³</u></td> <td><u>8.1x10⁻³</u></td> <td>9.8x10⁻³</td> </tr> </tbody> </table> <p data-bbox="189 860 1195 956">It was assumed that the nickel activity in the homogeneous amalgam was unity; this assumption may not have been valid. The underlined results were obtained by long extrapolation, so that the solubility of pure nickel at higher temperatures was understated.</p>		Solute	Soly/at %								20°C	50°C	100°C	150°C	200°C	300°C	400°C	500°C	NiHg ₄	1.5x10 ⁻⁷	1.8x10 ⁻⁶	4.1x10 ⁻⁵	4.7x10 ⁻⁴	3.0x10 ⁻³	-	-	-	NiHg ₃	3.4x10 ⁻⁶	1.9x10 ⁻⁵	1.6x10 ⁻⁴	9.3x10 ⁻⁴	3.6x10 ⁻³	-	-	-	NiHg ₂	2.3x10 ⁻⁵	8.5x10 ⁻⁵	<u>4.7x10⁻⁴</u>	<u>1.8x10⁻³</u>	-	-	-	-	Ni	1.1x10 ⁻³	1.6x10 ⁻³	2.4x10 ⁻³	3.3x10 ⁻³	4.2x10 ⁻³	<u>6.1x10⁻³</u>	<u>8.1x10⁻³</u>	9.8x10 ⁻³
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AUXILIARY INFORMATION																																																						
METHOD/APPARATUS/PROCEDURE: The intermetallic compounds of Ni and Hg were prepared by electrolysis on various cathodes and the amalgam was used as an electrode for the cell: $\text{Ni(Hg)}_x 6 \text{ mol dm}^{-3} \text{ CaCl}_2, X \text{ mol dm}^{-3} \text{ NiCl}_2 $ $6 \text{ mol dm}^{-3} \text{ CaCl}_2 \text{Hg}_2\text{Cl}_2, \text{Hg}$ at different temperatures. The solubilities were calculated from the measured EMF.	SOURCE AND PURITY OF MATERIALS: All chemicals of reagent grade from Ciech were additionally purified by crystallization. Mercury was chemically purified by shaking with acidic solution of Hg ₂ (NO ₃) ₂ , washed and then distilled under reduced pressure. ESTIMATED ERROR: Soly: large because equilibrium was not attained in case of Ni, NiHg ₂ and NiHg ₃ ; potential reproducibility was 2-3 mV. Temp: nothing specified. REFERENCES:																																																					

COMPONENTS: (1) Nickel; Ni; [7440-02-0] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: Zutić, V.; Batel, R.; Chevalet, J. <i>J. Electroanal. Chem.</i> <u>1979</u> , <i>106</i> , 115-25.
VARIABLES: One temperature: 30°C	PREPARED BY: C. Guminski; Z. Galus
EXPERIMENTAL VALUES: Solubility of Ni in Hg, probably at 30°C, was reported to be $5 \times 10^{-6} \text{ mol dm}^{-3}$. The corresponding atomic % solubility calculated by the compilers is 7×10^{-6} at %. NiHg ₃ is assumed to crystallize in the amalgam so that the solubility is referred to this compound. Kinetics of the crystallization was also investigated.	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: The reduction of Ni(II) ($5 \times 10^{-4} - 1 \times 10^{-2} \text{ mol dm}^{-3}$) in $10 \text{ mol dm}^{-3} \text{ LiCl}$ and subsequent oxidation of Ni amalgam were performed at the dropping mercury electrode. The generation potential was scanned along the whole Ni(II) reduction wave and oxidation of Ni from the amalgam formed was carried out at plateau of the polarographic anodic wave (-0.15 V vs. SCE). The Ni solubility was estimated from a relation of critical concentration of Ni on generation time.	SOURCE AND PURITY OF MATERIALS: NiCl ₂ was "Prolabo" from Rhône-Poulenc (Co content below $5 \times 10^{-3}\%$). LiCl was analytical grade and was heated several hours at 500°C. Mercury was double distilled. Water was triple distilled. ESTIMATED ERROR: Soly: nothing specified. Temp: precision $\pm 2 \text{ K}$. REFERENCES:

<p>COMPONENTS:</p> <p>(1) Palladium; Pd; [7440-05-3] (2) Mercury; Hg; [7439-97-6]</p>	<p>EVALUATOR:</p> <p>C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland</p> <p>July, 1985</p>																		
<p>CRITICAL EVALUATION:</p> <p>There have been only three reports of the experimental determination of palladium in mercury. Jangg and Gröhl (1) determined the solubility over a temperature range of 298 to 573 K, and reported a solubility of 5.1×10^{-3} at % at 298 K; a smooth curve was plotted through the data in this work. The room temperature value of Butler and Makrides (2), 5.5×10^{-3} at %, is in good agreement with that of Jangg and Gröhl. The room temperature solubility of 1.2×10^{-2} at % reported by Strachan and Harris (3) is twofold higher than those of (1) and (2). The data of Jangg and Gröhl appear to be the most accurate, and these authors reported their experimental procedure in some detail.</p> <p>Kozin (4,5) predicted the solubility of Pd in Hg at 298 K, but his values are more than a hundredfold too low.</p> <p>Messing and Dean (6) reported that the solubility of palladium in saturated uranium amalgam is nearly a hundredfold lower than in pure mercury.</p> <p>Palladium forms the intermediate compounds, PdHg₄ (stable up to 363 K), Pd₂Hg₅ (stable up to 511 K) and PdHg (1,7); Pd₂Hg₃ also has been reported (8), but this compound was shown not to exist in this system (1,7).</p> <p>Tentative solubilities of Pd in Hg:</p> <table border="1" data-bbox="439 889 994 1113"> <thead> <tr> <th><u>T/K</u></th> <th><u>Soly/at % x 10³</u></th> <th><u>Reference</u></th> </tr> </thead> <tbody> <tr> <td>298</td> <td>5.1</td> <td>[1,2]</td> </tr> <tr> <td>323</td> <td>5.4</td> <td>[1]</td> </tr> <tr> <td>373</td> <td>11</td> <td>[1]</td> </tr> <tr> <td>473</td> <td>71</td> <td>[1]</td> </tr> <tr> <td>573</td> <td>370</td> <td>[1]</td> </tr> </tbody> </table> <p><u>References</u></p> <ol style="list-style-type: none"> Jangg, G.; Gröhl, W. <i>Z. Metallk.</i> <u>1965</u>, <i>56</i>, 232. Butler, J.N.; Makrides, A.C. <i>Trans. Faraday Soc.</i> <u>1964</u>, <i>60</i>, 938. Strachan, J.F.; Harris, N.L. <i>J. Inst. Metals</i> <u>1956-57</u>, <i>85</i>, 17. Kozin, L.F. <i>Tr. Inst. Khim. Nauk Akad. Nauk Kaz. SSR</i> <u>1962</u>, <i>9</i>, 101. Kozin, L.F. <i>Fiziko Khimicheskie Osnovy Amalgamnoi Metallurgii</i>, Nauka, Alma-Ata, 1964. Messing, A.F.; Dean, O.C. <i>U.S. At. Energy Comm. Rep.</i>, ORNL-2871, <u>1960</u>. Galus, Z. <i>Crit. Rev. Anal. Chem.</i> <u>1975</u>, <i>5</i>, 359. Bittner, H.; Novotny, H. <i>Monatsh. Chem.</i> <u>1953</u>, <i>84</i>, 211. 		<u>T/K</u>	<u>Soly/at % x 10³</u>	<u>Reference</u>	298	5.1	[1,2]	323	5.4	[1]	373	11	[1]	473	71	[1]	573	370	[1]
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COMPONENTS: (1) Palladium; Pd; [7440-05-3] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: Strachan, J.F.; Harris, N.L. <i>J. Inst. Metals</i> <u>1956-57</u> , 85, 17-24.
VARIABLES: Room temperature	PREPARED BY: C. Guminski; Z. Galus
EXPERIMENTAL VALUES: The solubility of palladium in mercury at room temperature was reported to be 0.012 at %.	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: Mercury and palladium were equilibrated in evacuated glass tubes and maintained either at room temperature or at 773 K for times lasting for many hours. The solubility was determined from the change in weight of the specimens after equilibration, and by chemical analysis of the amalgam after filtration through a sintered glass filter. The analytical method was not specified.	SOURCE AND PURITY OF MATERIALS: 99.997% pure mercury was submitted to cleaning, filtration, drying and distillation before use. Palladium was 99 to 99.99% pure. ESTIMATED ERROR: Soly: precision \pm 20%. Temp: not specified. REFERENCES:

COMPONENTS: (1) Palladium; Pd; [7440-05-3] (2) Mercury; Hg [7439-97-6]	ORIGINAL MEASUREMENTS: Butler, J.N.; Makrides, A.C. <i>Trans. Faraday Soc.</i> <u>1964</u> , <i>60</i> , 938-946.
VARIABLES: Room temperature measurement	PREPARED BY: C. Guminski; Z. Galus
EXPERIMENTAL VALUES: The solubility of palladium in mercury at room temperature was reported to be 5.5×10^{-3} at %.	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: Mercury was saturated with palladium in a sealed glass tube for few days at 523 K. The amalgam was then cooled down to room temperature and filtered through a sintered glass filter. The filtrate was analyzed spectroscopically.	SOURCE AND PURITY OF MATERIALS: Palladium purity not specified. Triply-distilled mercury was used. Content of other metals in the amalgam was below 10^{-4} %.
	ESTIMATED ERROR: Nothing specified.
	REFERENCES:

COMPONENTS: (1) Palladium; Pd; [7440-05-3] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: Jangg, G.; Gröhl, W. <i>Z. Metallk.</i> 1965, 56, 232-34.																														
VARIABLES: Temperature: 25-300°C	PREPARED BY: C. Guminski; Z. Galus																														
EXPERIMENTAL VALUES: The solubility of palladium in mercury: <table data-bbox="389 513 940 872" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th style="text-align: center;"><u>t/°C</u></th> <th style="text-align: center;"><u>Soly/mass %</u></th> <th style="text-align: center;"><u>Soly/at %^a</u></th> </tr> </thead> <tbody> <tr><td style="text-align: center;">25</td><td style="text-align: center;">0.0027</td><td style="text-align: center;">0.0051</td></tr> <tr><td style="text-align: center;">50</td><td style="text-align: center;">0.0029</td><td style="text-align: center;">0.0054</td></tr> <tr><td style="text-align: center;">90</td><td style="text-align: center;">0.0047</td><td style="text-align: center;">0.0089</td></tr> <tr><td style="text-align: center;">100</td><td style="text-align: center;">0.0060</td><td style="text-align: center;">0.011</td></tr> <tr><td style="text-align: center;">150</td><td style="text-align: center;">0.015</td><td style="text-align: center;">0.031</td></tr> <tr><td style="text-align: center;">200</td><td style="text-align: center;">0.038</td><td style="text-align: center;">0.071</td></tr> <tr><td style="text-align: center;">238</td><td style="text-align: center;">0.089</td><td style="text-align: center;">0.17</td></tr> <tr><td style="text-align: center;">250</td><td style="text-align: center;">0.099</td><td style="text-align: center;">0.19</td></tr> <tr><td style="text-align: center;">300</td><td style="text-align: center;">0.20</td><td style="text-align: center;">0.37</td></tr> </tbody> </table> <p style="text-align: center;">^aby compilers.</p> <p>Additional data were presented graphically. There were two breaks in the reciprocal temperature-solubility plot, at 90 and 240°C; these corresponded to the decomposition temperatures of PdHg₄ and Pd₂Hg₅, respectively.</p>		<u>t/°C</u>	<u>Soly/mass %</u>	<u>Soly/at %^a</u>	25	0.0027	0.0051	50	0.0029	0.0054	90	0.0047	0.0089	100	0.0060	0.011	150	0.015	0.031	200	0.038	0.071	238	0.089	0.17	250	0.099	0.19	300	0.20	0.37
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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 a pressure of purified nitrogen. The palladium content in the filtered, saturated amalgam was determined by an unspecified analytical method.	SOURCE AND PURITY OF MATERIALS: Not specified in detail. <table data-bbox="690 1585 1232 1719" style="margin-top: 10px;"> <tbody> <tr> <td>ESTIMATED ERROR:</td> </tr> <tr> <td>Soly: nothing specified; precision better than $\pm 10\%$ (compilers).</td> </tr> <tr> <td>Temp: nothing specified.</td> </tr> <tr> <td>REFERENCES:</td> </tr> </tbody> </table>	ESTIMATED ERROR:	Soly: nothing specified; precision better than $\pm 10\%$ (compilers).	Temp: nothing specified.	REFERENCES:																										
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<p>COMPONENTS:</p> <p>(1) Platinum; Pt; [7440-06-4]</p> <p>(2) Mercury; Hg; [7439-97-6]</p>	<p>EVALUATOR:</p> <p>C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985</p>
<p>CRITICAL EVALUATION:</p> <p>There is a large variation on the reported solubilities of platinum in mercury. The solubility is very low, and the presence of platinum oxides on the surface of the metal further inhibits its dissolution in mercury. Plaksin and Suvorovskaya (1,2) determined the solubility in the range of 289-473 K by filtration and analyses of the saturated solution, and they found that the solubility increased from 2.05×10^{-2} to 1.77 at % in this temperature range. At room temperature, Strachan and Harris (3) reported a solubility of 0.002 at %, while Butler and Makrides (4) obtained a solubility of 0.028 at %; the latter solubility is in agreement with that by ref. (2). Yoshida (5) reported that the solubility is less than 0.01 at %.</p> <p>Kozin, with the use of his semiempirical equations at 298 K, predicted solubilities of 2.6×10^{-9} (6) and 3.1×10^{-7} at % (7); these results are too low because interaction of the metals was neglected.</p> <p>Jangg and Dörtbudak (8) determined the solubilities at 374-593 K, and they found that the solubility increased from 3.4×10^{-5} to 9.0×10^{-4} at % in this temperature range. These solubilities are significantly lower than the experimental determination of the previous authors. Based on the work of Barlow and Planting (9), the evaluators are of the opinion that the solubility of platinum at 573 K should be $\approx 2 \times 10^{-2}$ at %.</p> <p>There is a variation of approximately 10^3 in the reported solubilities, and though the experimental procedures were similar there was a lack of detailed description by the different authors. This has resulted in some difficulty in assigning the more accurate measurements. Although the data in refs. (1,2,4) are similar, the results appear to be too high. Recent, careful electroanalytical measurements in the evaluators' laboratory (10) resulted in a solubility of 5×10^{-4} at % at 298 K. In this work, it was found that the platinum had to be equilibrated with mercury at 600 K for at least a week, followed by an equilibration at 298 K for at least two weeks. Shorter equilibration times resulted in erroneous solubilities; the discrepancies in previously reported data probably are the result of incomplete equilibration. In view of the evaluators' 298 K determination, it appears that the data of Jangg and Dörtbudak (8) should be rejected.</p> <p>During investigations of the corrosion of pure metals in refluxing mercury at 756 K it was found that the solubility of platinum is similar to, or lower than, those of aluminum and manganese, but higher than those of nickel, titanium and zirconium (11); this observation adds further proof that the results of (8) are too low.</p> <p>The saturated platinum amalgams are in equilibrium with the intermediate solid phases (2,5,8,9,12), PtHg₄, PtHg₂ and PtHg; the latter two compounds are stable to 523 K and the first compound is stable to 873 K.</p> <p>The tentative value for the solubility of platinum in mercury at 298 K is 5×10^{-4} at % (10).</p> <p>References</p> <ol style="list-style-type: none"> 1. Plaksin, I.N.; Suvorovskaya, N.A. <i>Zh. Fiz. Khim.</i> 1941, 15, 978. 2. Plaksin, I.N.; Suvorovskaya, N.A. <i>Izv. Sekt. Platiny</i> 1945, 18, 67. 3. Strachan, J.F.; Harris, N.L. <i>J. Inst. Metals</i> 1956-57, 85, 17. 4. Butler, J.N.; Makrides, A.C. <i>Trans. Faraday Soc.</i> 1964, 60, 938. 5. Yoshida, Z. <i>Bull. Chem. Soc. Jap.</i> 1981, 54, 556. 6. Kozin, L.F. <i>Tr. Inst. Khim. Nauk Akad. Nauk Kaz. SSR</i> 1962, 9, 101. 7. Kozin, L.F. <i>Fiziko-Khimicheskie Osnovy Amalgamoi Metallurgii</i>, Nauka, Alma-Ata, 1964. 8. Jangg, G.; Dörtbudak, T. <i>Z. Metallk.</i> 1973, 64, 715. 9. Barlow, M.; Planting, P.J. <i>Z. Metallk.</i> 1969, 60, 292. 10. Guminski, C.; Roslonek, H.; Galus, Z. <i>J. Electroanal. Chem.</i> 1983, 158, 357. 11. Fleitman, A.H.; Weeks, J.R. <i>Nucl. Eng. Des.</i> 1971, 16, 266. 12. Jangg, G.; Steppan, F. <i>Z. Metallk.</i> 1965, 56, 172. 	

COMPONENTS: (1) Platinum; Pt; [7440-06-4] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: Plaksin, I.N.; Suvorovskaya, N.A. <i>Izv. Sekt. Platiny</i> <u>1945</u> , 18, 67-76.																																
VARIABLES: Temperature: 16-200°C	PREPARED BY: C. Guminski; Z. Galus																																
EXPERIMENTAL VALUES: Solubility of platinum in mercury: <table border="1" data-bbox="210 497 1007 797" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th><u>t/°C</u></th> <th><u>Soly/at %</u></th> <th><u>t/°C</u></th> <th><u>Soly/at %</u></th> </tr> </thead> <tbody> <tr> <td>16.5</td> <td>0.0205</td> <td>86.0</td> <td>0.904</td> </tr> <tr> <td>17.5</td> <td>0.0513</td> <td>101.0</td> <td>0.980</td> </tr> <tr> <td>20.0</td> <td>0.0021</td> <td>131.5</td> <td>1.040</td> </tr> <tr> <td>24.0</td> <td>0.102</td> <td>144.0</td> <td>1.080</td> </tr> <tr> <td>39.5</td> <td>0.151</td> <td>167.0</td> <td>1.12</td> </tr> <tr> <td>54.0</td> <td>0.202</td> <td>171.0</td> <td>1.20</td> </tr> <tr> <td>71.0</td> <td>0.910</td> <td>200.0</td> <td>1.77</td> </tr> </tbody> </table> <p>Pt-Hg phase diagram was presented; Pt₃Hg, Pt₂Hg and PtHg are in equilibrium with the saturated amalgams.</p> <p>Substantially the same results of the solubility were reported in the previous work by the same authors (1).</p>		<u>t/°C</u>	<u>Soly/at %</u>	<u>t/°C</u>	<u>Soly/at %</u>	16.5	0.0205	86.0	0.904	17.5	0.0513	101.0	0.980	20.0	0.0021	131.5	1.040	24.0	0.102	144.0	1.080	39.5	0.151	167.0	1.12	54.0	0.202	171.0	1.20	71.0	0.910	200.0	1.77
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AUXILIARY INFORMATION																																	
METHOD/APPARATUS/PROCEDURE: Purified platinum was dissolved in mercury during cathodic polarization of Pt while in contact with Hg and H ₂ SO ₄ solution. Prepared amalgams were kept in a thermostat for 2 hours and filtered through a capillary of 0.24-0.4 mm diameter. The filtration and storing of amalgam were done with the use of a special air-free glass apparatus. The mercury was evaporated from the filtrate and platinum was analyzed by colorimetric, cupellation, or gravimetric method, depending upon the metal content.	SOURCE AND PURITY OF MATERIALS: Mercury purified with HNO ₃ and distilled under vacuum. Platinum, 99.84% pure, was dissolved in aqua regia, then transformed into (NH ₄) ₂ PtCl ₄ and reduced to the metallic state with HCOOH. ESTIMATED ERROR: Soly: precision no better than several percent (compilers). Temp: precision no better than ± 0.5 K (compilers). REFERENCES: 1. Plaksin, I.N.; Suvorovskaya, N.A. <i>Zh. Fiz. Khim.</i> <u>1941</u> , 15, 978.																																

COMPONENTS: (1) Platinum; Pt; [7440-06-4] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: Strachan, J.F.; Harris, N.L. <i>J. Inst. Metals</i> <u>1956-57</u> , 85, 17-24.
VARIABLES: Room temperature	PREPARED BY: C. Guminski; Z. Galus
EXPERIMENTAL VALUES: Solubility of platinum in mercury at room temperature was reported to be 0.002 at %.	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: A preweighed piece of platinum was equilibrated with Hg in an evacuated glass tube over long periods of time. After equilibration, the amalgam was filtered through a sintered glass filter of 90-150 μm pore size. The solubility was determined from: (1) the weight loss of the platinum; and (2) analysis of the filtrate by an unspecified method.	SOURCE AND PURITY OF MATERIALS: Mercury: 99.997% pure was filtered, dried and distilled before use. Platinum: minimum 99.99% pure. ESTIMATED ERROR: Soly: precision \pm 50%. Temp: nothing specified. REFERENCES:

COMPONENTS: (1) Platinum; Pt; [7440-06-4] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: Butler, J.N.; Makrides, A.C. <i>Trans. Faraday Soc.</i> <u>1964</u> <i>60</i> , 938-46.			
VARIABLES: Room temperature	PREPARED BY: C. Guminski; Z. Galus			
EXPERIMENTAL VALUES: The solubility of platinum in mercury at room temperature was reported to be 0.028 at %.				
AUXILIARY INFORMATION				
METHOD/APPARATUS/PROCEDURE: Mercury was saturated with platinum in a sealed glass tube for few days at 523 K, then the amalgam was cooled down to room temperature and filtered through a sintered glass filter. The filtrate was analyzed spectroscopically.	<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td data-bbox="669 1259 1225 1587"> SOURCE AND PURITY OF MATERIALS: Purity of platinum not specified. Triply distilled mercury was used. Content of other metals in the amalgam was below $10^{-4}\%$. </td> </tr> <tr> <td data-bbox="669 1587 1225 1721"> ESTIMATED ERROR: Nothing specified. </td> </tr> <tr> <td data-bbox="669 1721 1225 1929"> REFERENCES: (This section is currently empty) </td> </tr> </table>	SOURCE AND PURITY OF MATERIALS: Purity of platinum not specified. Triply distilled mercury was used. Content of other metals in the amalgam was below $10^{-4}\%$.	ESTIMATED ERROR: Nothing specified.	REFERENCES: (This section is currently empty)
SOURCE AND PURITY OF MATERIALS: Purity of platinum not specified. Triply distilled mercury was used. Content of other metals in the amalgam was below $10^{-4}\%$.				
ESTIMATED ERROR: Nothing specified.				
REFERENCES: (This section is currently empty)				

COMPONENTS: (1) Platinum; Pt; [7440-06-4] (2) Mercury; Hg; [7439-97-6]	ORIGINAL MEASUREMENTS: Guminski, C.; Roslonek, H.; Galus, Z. <i>J. Electroanal. Chem.</i> <u>1983</u> , <i>158</i> , 357-68.
VARIABLES: One temperature: 298 K	PREPARED BY: C. Guminski; Z. Galus
EXPERIMENTAL VALUES: Solubility of platinum in mercury at 298 K was reported to be $(5 \pm 1) \times 10^{-4}$ at %. When the dissolution time was shorter than a week, or the conditioning shorter than 2 weeks, the solubilities determined were lower and higher, respectively.	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE: A clean and degreased Pt foil was placed in a closed glass vessel with mercury and heated at 600 K for 8 hours per day during a period of 2 months. After a subsequent month of conditioning at 298 K the heterogeneous amalgam was filtered through a sintered-glass crucible under vacuum. Employing a hanging-drop electrode filled with the saturated amalgam, cyclic chronopotentiometric curves were recorded in 0.01 mol dm ⁻³ ZnCl ₂ + 1 mol dm ⁻³ NaCl solution. The current density was varied between 10.8 and 1080 $\mu\text{A cm}^{-2}$ and reduction time from 5 to 100 s. The differences of oxidation times observed were due to bonding of Zn by Pt to form PtZn ₂ , a stable compound which is rapidly formed and is insoluble in mercury. The calculations of the solubility were based on these differences.	SOURCE AND PURITY OF MATERIALS: Mercury (from Ciech) was chemically purified with acidic Hg ₂ (NO ₃) ₂ and then twice distilled in vacuum. Platinum (from Polish Mint) was 99.9999% pure. ZnCl ₂ and NaCl (from Ciech) were analytically pure; their solutions were additionally refined by a cathodic electrolysis at -0.9 V vs. SCE.
ESTIMATED ERROR: Soly: precision \pm 20%. Temp: \pm 1 K.	
REFERENCES:	