

<p>COMPONENTS:</p> <p>(1) Mercury(II) oxide; HgO; [21908-53-2]</p> <p>(2) Water, H₂O; [7732-18-5]</p>	<p>EVALUATOR:</p> <p>T. P. Dirkse Department of Chemistry Calvin College Grand Rapids, Michigan 49506, U.S.A. March 1983</p>
<p>CRITICAL EVALUATION:</p> <p>Mercury is found in two oxidation states in its compounds: + and 2+. However, mercury does not show this dual behavior in its stable oxides. In the early literature Hg₂O was considered to be one of the oxides of mercury and several studies were carried out to determine quantitatively some of its physical properties.</p> <p>Bugarszky (1) made e.m.f. measurements of cells containing Hg₂O/Hg electrodes. Later, Brodsky (2) measured the temperature dependence of the activity coefficients of Hg₂(NO₃)₂ in water, and the E° value for the Hg²⁺/Hg electrode and from these results and those of Bugarszky calculated the solubility product of Hg₂O in water to be 1.8 x 10⁻²⁴ mol³ dm⁻⁹ at 298 K.</p> <p>Allmand (3) attempted to repeat some of this work but found that the Hg₂O electrodes he prepared gave the same values as did HgO electrodes. Identical values were obtained whether or not light was excluded. Therefore, Allmand concluded that all calculations based on the e.m.f. of Hg₂O electrodes have dubious value because Hg₂O is unstable in aqueous solution and these electrodes then are always contaminated with an unknown quantity of HgO. Later (4) it was shown by X-ray analysis that Hg₂O is an intimate mixture of HgO and metallic mercury. Sanemasa (5) made a kinetic study of this disproportionation of Hg₂O in water.</p> <p>No further data on Hg₂O are included because of this instability.</p> <p>HgO crystals are red or yellow, depending on the method of preparation. When Hg(NO₃)₂ is heated, red crystals of HgO are obtained, while when OH⁻ ions are added to aqueous solutions of Hg²⁺ ions in the cold, yellow crystals of HgO precipitate. The difference between these two forms has been the subject of some disagreement. Cohen (6), as a result of some very careful work, obtained a value of 0.685 mV for the e.m.f. of the following cell:</p> $\text{Hg} \mid \text{HgO}(\text{yellow}) \mid \text{alkali} \mid \text{HgO}(\text{red}) \mid \text{Hg.} \quad (1)$ <p>This value remained constant for 3 days but only after a week had elapsed. From this Cohen concluded that the red and yellow forms were isomers and, at 298.2 K, the red form was the less stable and the less soluble. He stated that the red and yellow forms dissolved at different rates and he gave this as the reason it took about a week for the cell to reach equilibrium.</p> <p>Ostwald (7) maintained that the difference between the red and yellow forms was merely one of particle size. This results in a difference in solubility, and Ostwald insisted that this was the cause of the e.m.f. of the cell represented in equation (1). As proof of this contention Ostwald ground up red crystals of HgO and obtained yellow ones. He also studied the extent to which the following reaction proceeded:</p> $\text{HgO}(\text{s}) + \text{H}_2\text{O} + 2 \text{KBr} \cdot \text{aq} = 2 \text{KOH} \cdot \text{aq} + \text{HgBr}_2 \cdot \text{aq.} \quad (2)$ <p>He found that the amount of KOH produced depended on the particle size of the HgO.</p> <p>Hulett (8) pursued this matter further. He found that by digestion and decantation he could separate the most finely divided particles (which had a yellow color) from a sample of red HgO. Although he made only qualitative measurements he also found that by grinding red HgO he could increase its solubility in water at 298 K. He concluded from this that the red and yellow forms were not isomers but merely differed in particle size.</p> <p>Schoch (9) made a microscopic examination of the two forms of HgO and concluded that there were two crystal forms: prismatic (red) and square tablets (yellow). He also found that the color did change with particle size and with heating, but</p>	

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<p>CRITICAL EVALUATION:</p> <p>maintained that color is no criterion of crystal form because the finely ground red form changed color on heating but the finely ground yellow form did not. He concluded that the red form is probably the less soluble one at room temperature (this is not in agreement with solubility results determined later by others).</p> <p>Allmand (3) measured the e.m.f. of the following cell:</p> $\text{Hg} \mid \text{HgO(s)} \mid \text{aq. alkali} \mid \text{calomel electrode.} \quad (3)$ <p>He used both red and yellow HgO. No solubility values are given in the paper but Allmand made the following observations: (a) in general, the red HgO gives the lower e.m.f. value and, consequently, is less soluble and less stable; (b) the e.m.f.'s generally decreased with time, and while this ordinarily indicates a phase change, this cannot be the explanation here because this decrease occurred with both forms of HgO; (c) the electrodes with the highest e.m.f. values had the smallest particle size when viewed microscopically; (d) the e.m.f. of a cell such as that of equation (1) was less than the experimental uncertainty of the measurement. Consequently, Allmand concluded also that the red and yellow forms were not isomers but differed only in particle size.</p> <p>Ostwald (7) determined the amount of KOH produced by reaction (2). He used both forms of HgO and found that the differences were within the experimental uncertainty. He also found that by grinding the oxides more finely a larger amount of KOH was formed. Garrett and Hirschler (10) did observe differences in the solubility of the two forms but hesitated to conclude that this difference was significant enough to warrant the assumption that the two forms are isomers. The solubility of the two forms in HNO₃ (11) show no significant differences. Schick (12) observed that mixtures of the red HgO in water sometimes deposited a yellow form of the oxide on the walls of the container. He also found that mixtures of HgO with a small amount of water became redder in color when warmed and yellower when cooled.</p> <p>Consequently, it appears that the differences that have been observed can be accounted for adequately by assuming that the yellow and red forms differ in particle size and the more finely divided the particles are (yellower) the larger the solubility value. This conclusion is confirmed by careful X-ray powder diffraction measurements, which are the same for both forms of HgO (13).</p> <p>Solubility in water.</p> <p>Only five papers report a directly determined value for the solubility of HgO in water. Schick (12), Fuseya (14), Garrett and Hirschler (10), and Herz and Hiebenthal (15) made the determination at 298.2 K while Salem (16) made the determination at 308.2 K. Schick and Garrett and Hirschler used both red and yellow HgO while the others used only the red form. In view of the relationship between solubility and particle size the best results are those in which the system was allowed to equilibrate the longest time. Schick does not state how many days were allowed for equilibration but indications are that it was a week or more. He found very little difference between the solubilities of the red and yellow forms. Garrett and Hirschler found a larger difference but were reluctant to state the significance of this difference. The values they obtained with red HgO were less reproducible than those obtained with yellow HgO, probably because of non-uniformity of particle size. In view of all this the solubility of HgO in water is within 3% of $2.37 \times 10^{-4} \text{ mol dm}^{-3}$ at 298.2 K and within 3% of $3.47 \times 10^{-4} \text{ mol dm}^{-3}$ at 308.2 K.</p> <p>Five papers report a value for the solubility product of Hg(OH)₂. Labendzinski (17) gives a value of 1.5×10^{-26} which was determined in his laboratory by Fulda, a colleague. However, there is no indication as to the experimental basis for this value, although it appears to be based on an e.m.f. measurement. No temperature is given. Grossmann (18) used the dissociation constants of HgBr₄²⁻ and Hg(SCN)₄²⁻ which</p>	

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<p>CRITICAL EVALUATION:</p> <p>he and others measured and the assumed reaction (4) to derive a value of $4 \times 10^{-26} \text{ mol}^3 \text{ dm}^{-9}$</p> $\text{Hg(OH)}_2 + 4 \text{X}^- = \text{HgX}_4^{2-} + 2 \text{OH}^- \quad (4)$ <p>for the value of K_{s0} of Hg(OH)_2 at 298 K.</p> <p>Allmand (3) measured the e.m.f.'s of cells containing the Hg HgO alkali electrode and a calomel electrode. He does not give the values for the cell e.m.f.'s but only his derived half-cell values. In deriving these he made the following assumptions: (a) E for the normal calomel electrode is 0.283 V; (b) $K_w = 0.56 \times 10^{-14}$; (c) E° for the Hg²⁺/Hg⁰ electrode is 0.835 V; (d) the junction potential between the NaOH and KCl solutions is 0.020 V while that between the KOH and KCl solutions is 0.015 V. All these values are at 291 K. He also made assumptions about the degree of ionization of the alkali solutions. These values differed by as much as 10% from the activity coefficients of these same solutions as determined later (19). With these assumptions he derived a value of $4 \times 10^{-26} \text{ mol}^3 \text{ dm}^{-9}$ for K_{s0} for Hg(OH)_2 at 291 K.</p> <p>Maijs (20) derived a value of $\text{p}K_{s0} = 15-26$ for Hg(OH)_2. This was based on calculations involving a thermochemical cycle, semiempirical relationships, and published data of the pH of complete precipitation of Hg(OH)_2 from aqueous solutions. No temperature is specified.</p> <p>Solubility measurements at a constant ionic strength of 3 mol dm^{-3} (using perchlorate ion to maintain ionic strength) also gave a value of $10^{-26} \text{ mol}^3 \text{ dm}^{-9}$ for the solubility product constant at 298 K (21). However, in a 3 mol dm^{-3} solution of NaClO_4 the solubility of Hg(OH)_2 was $1.78 \times 10^{-4} \text{ mol dm}^{-3}$. This is slightly less than the solubility in water and was attributed to a "salting out" effect.</p> <p>Because of the divergent values reported, the solubility product of Hg(OH)_2 can only be said to be approximately $10^{-26} \text{ mol}^3 \text{ dm}^{-9}$ at room temperature.</p> <p>Solubility as a function of pH.</p> <p>Much of the interest in the solubility of HgO in aqueous solutions has as its purpose the determination of the extent to which the Hg(OH)_2 formed in solution acts as an acid and as a base. As an acid it may ionize as follows:</p> $\text{Hg(OH)}_2 = \text{H}^+ + \text{HHgO}_2^- \quad (5)$ $\text{HHgO}_2^- = \text{H}^+ + \text{HgO}_2^{2-} \quad (6)$ <p>The equilibrium quotients are:</p> $K_5 = m_{\text{H}^+} \ominus m_{\text{HHgO}_2^-} / m_{\text{Hg(OH)}_2} \quad (7)$ <p>and</p> $K_6 = m_{\text{H}^+} \ominus m_{\text{HgO}_2^{2-}} / m_{\text{HHgO}_2^-} \quad (8)$ <p>In alkaline solutions the following reactions take place.</p> $\text{HgO(s)} + \text{OH}^- = \text{HHgO}_2^- \quad (9)$ $\text{HgO(s)} + 2 \text{OH}^- = \text{HgO}_2^{2-} + \text{H}_2\text{O} \quad (10)$	

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<p>CRITICAL EVALUATION:</p> <p>The equilibrium constant expressions for the above reactions are:</p> $K_9^{\circ} = a_{\text{HHgO}_2^-} / a_{\text{OH}^-} \quad (11)$ <p>and</p> $K_{10}^{\circ} = a_{\text{HgO}_2^{2-}} / (a_{\text{OH}^-})^2 \quad (12)$ <p>and the total solubility, m_T, may then be expressed as</p> $m_T = m_{\text{Hg(OH)}_2} + m_{\text{HHgO}_2^-} + m_{\text{HgO}_2^{2-}} \quad (13)$ <p>Making substitutions from the equilibrium constant expressions and assuming the validity of the Debye-Huckel relationship for activity coefficients in these dilute solutions, equation (13), following a procedure described earlier (22), reduces to</p> $m_T = m_{\text{Hg(OH)}_2} + K_9^{\circ} \cdot m_{\text{OH}^-} + K_{10}^{\circ} \cdot (m_{\text{OH}^-})^2 / \gamma_{\text{NaOH}} \cdot a_{\text{H}_2\text{O}} \quad (14)$ <p>Fuseya (14) and Garrett and Hirschler (10) found a linear relationship between the solubility of HgO and molality of base up to a base molality of 1 mol kg⁻¹. This indicates that the last term in equation (14) contributes less than the experimental uncertainty and the concentration of HgO₂²⁻ in these dilute solutions is negligible. Equation (14) then, in effect, becomes</p> $m_T = m_{\text{Hg(OH)}_2} + K_9^{\circ} \cdot m_{\text{OH}^-} \quad (15)$ <p>A plot of m_T vs alkali concentration gives an intercept of 2.4×10^{-4} mol Hg(OH)₂ dm⁻³. This is the solubility of Hg(OH)₂ at 298.2 K and the value of the equilibrium quotient, K_{16}, for reaction (16).</p> $\text{HgO(s)} + \text{H}_2\text{O} = \text{Hg(OH)}_2(\text{sln}) \quad (16)$ $K_{16} = m_{\text{Hg(OH)}_2} \quad (17)$ <p>The slope of the line is 3.1×10^{-5} and this is the value of K_9° at 298.2 K. From the values of K_9°, K_{16}, and the ion product constant of water, K_5 can be calculated and its value at 298.2 K is 1.4×10^{-15} mol dm⁻³.</p> <p>The work of Garrett and Hirschler (10) shows that in alkalies the solubility of HgO is independent of the cation but does depend on the nature of the anion as well as on the total ionic strength. However, in the absence of alkalies, the solubility does appear to depend on the nature of the cation as well (15). In chloride solutions the increase in solubility due to the presence of the cation was Li⁺ > Na⁺ > K⁺. But the solubility is not a linear function of the concentration of the salts used. No work has been reported dealing more specifically with the effect of the total ionic strength in alkaline solutions.</p> <p>In neutral and acid solutions Hg(OH)₂ acts as a base and may ionize as follows:</p> $\text{Hg(OH)}_2 = \text{HgOH}^+ + \text{OH}^- \quad (18)$ $\text{Hg(OH)}_2 = \text{Hg}^{2+} + 2 \text{OH}^- \quad (19)$	

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<p>CRITICAL EVALUATION:</p> <p>or</p> $\text{HgO(s)} + \text{H}^+ = \text{HgOH}^+ \quad (20)$ $\text{HgO(s)} + 2 \text{H}^+ = \text{Hg}^{2+} + \text{H}_2\text{O} \quad (21)$ <p>The corresponding equilibrium quotients are:</p> $K_{18} = m_{\text{HgOH}^+} \cdot m_{\text{OH}^-} / m_{\text{Hg(OH)}_2} \quad (22)$ $K_{19} = m_{\text{Hg}^{2+}} \cdot (m_{\text{OH}^-})^2 / m_{\text{Hg(OH)}_2} \quad (23)$ $K_{20} = m_{\text{HgOH}^+} / m_{\text{H}^+} \quad (24)$ $K_{21} = m_{\text{Hg}^{2+}} / (m_{\text{H}^+})^2 \quad (25)$ <p>Evaluation of the equilibrium constants for reactions (18) -(21) is complicated by the fact that the solubility of HgO in acid solutions is affected by the anions present because of the formation of complex ions involving these anions. This is particularly true of the halide ions. Another complication is that the solubility is large enough so that a significant, but unknown, amount of H⁺ ions is used up in the dissolving process. Garrett and Howell (11) used a method of successive approximations to arrive at the following values: K₂₀ = 0.17 and K₂₁ = 53, both at 298.2 K. These values, together with the ion product constant of water and the solubility of undissociated Hg(OH)₂ in water give K₁₈ = 7.1 × 10⁻¹² mol dm⁻³ at 298.2 K and K₁₉ = 2.2 × 10⁻²³ mol² dm⁻⁶ at 298.2 K. The latter value is slightly less than that calculated from the solubility of HgO in alkali solutions. However, it is the better value because of the uncertainties in the K_s^o value for Hg(OH)₂.</p> <p>A summary of the variation of the solubility of HgO with pH is given on Figure 1.</p> <p>Oka (23) reports a value of 10⁻²² mol² dm⁻⁶ at 298 K for K₁₉. This was obviously calculated from data obtained by titrating a solution of Hg(NO₃)₂ in HNO₃ with NaOH. However, no indications are given as to what the calculations were or how they were made. Nor is there any indication of the experimental uncertainty. Consequently, the value of K₁₉ given above is to be preferred to that of Oka.</p> <p>Bilinski, et al. (24) determined values for K₁₈ and K₁₉ in an indirect manner. They measured the solubility of HgCO₃·2HgO at a constant ionic strength of 0.5 mol dm⁻³ over a wide range of pH. At a pH above about 9.6 yellow HgO was the only solid phase appearing at 298 K. The data are presented only in graphical form. However, the numerical data were kindly made available to us in a personal communication from Dr. H. Bilinski. The solubility of HgO at pH = 10.4 is 4.64 × 10⁻⁴ mol dm⁻³. It is independent of the carbonate ion concentration up to a carbonate ion concentration of about 0.01 mol dm⁻³. This solubility is slightly larger than that shown in Figure 1 at the corresponding pH. This is due to the presence of mercury-carbonate in addition to mercury-hydroxo solute species.</p> <p>An equation for the total solubility of mercury in the form of the simplest polynomial in H⁺ ion concentration that would fit the data was developed⁽²⁴⁾. From this treatment a value of K₁₈ = 6.8 × 10⁻¹² mol dm⁻³ was obtained. This is within the experimental uncertainty of the value given above.</p> <p>A value of K₁₉ = 5 × 10⁻²² mol² dm⁻⁶ was also deduced from this treatment. This is about an order of magnitude larger than the value given above. However, because of the indirectness of the method used, the lower value is the preferred one.</p>	

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 (2) Water; H₂O; [7732-18-5]

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CRITICAL EVALUATION:

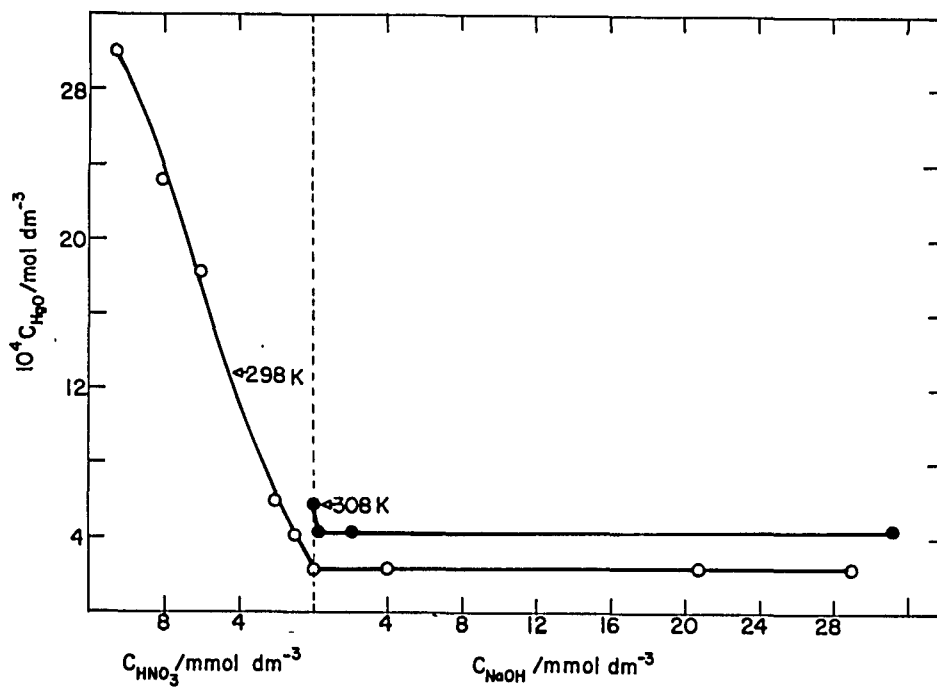


Figure 1. Solubility of HgO as a function of acidity-alkalinity.

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CRITICAL EVALUATION:

A summary of the equilibrium constants for various reactions involving HgO and Hg(OH)₂ is given in Table I.

Table I

Tentative equilibrium constant values at 298 K for HgO/Hg(OH)₂ system, based on solubility measurements

$\text{Hg(OH)}_2(\text{sln}) = \text{H}^+ + \text{HHgO}_2^-$	$K_5 = 1.4 \times 10^{-15} \text{ mol dm}^{-3}$
$\text{HgO}(\text{s}) + \text{OH}^- = \text{HHgO}_2^-$	$K_9 = 3.1 \times 10^{-5}$
$\text{HgO}(\text{s}) + \text{H}_2\text{O} = \text{Hg(OH)}_2(\text{sln})$	$K_{16} = 2.4 \times 10^{-4} \text{ mol dm}^{-3}$
$\text{Hg(OH)}_2(\text{sln}) = \text{HgOH}^+ + \text{OH}^-$	$K_{18} = 7.1 \times 10^{-12} \text{ mol dm}^{-3}$
$\text{Hg(OH)}_2(\text{sln}) = \text{Hg}^{2+} + 2 \text{OH}^-$	$K_{19} = 2.2 \times 10^{-23} \text{ mol}^2 \text{ dm}^{-6}$
$\text{HgO}(\text{s}) + \text{H}^+ = \text{HgOH}^+$	$K_{20} = 0.17$
$\text{HgO}(\text{s}) + 2 \text{H}^+ = \text{Hg}^{2+} + \text{H}_2\text{O}$	$K_{21} = 53 \text{ mol}^{-1} \text{ dm}^3$

K_s° for Hg(OH)₂ is approximately $10^{-26} \text{ mol}^3 \text{ dm}^{-9}$.

The above values are considered as tentative because there is a lack of corroborating work reported.

Some solubility determinations have also been made in aqueous HCl (25) and in aqueous HF (26, 27). The solubility values here tend to become erratic because of complexes formed with the acid anion. Thus, in HCl solutions a black solid phase, 2HgO·HgCl₂, was formed in molalities of HCl as low as 0.007 mol/kg. Above molalities of 0.1 mol/kg it appeared to be the only solid phase present and its solubility in water at 298 K was measured to be $11.4 \times 10^{-4} \text{ mol/kg H}_2\text{O}$.

Likewise, the solubility of HgO in aqueous HF is accompanied by the formation of HgO·HF and HgF₂·2H₂O. Jaeger (26) does not explicitly state the nature of the equilibrium solid phase but the implication is that it is HgO. Up to HF concentrations of 4 mol dm^{-3} the solubility of HgO in aqueous HF at 298 K (26, 27) can be expressed as

$$C_{\text{HgO}} = 0.103 C_{\text{HF}} \quad (26)$$

At HF concentrations greater than 4 mol dm^{-3} the data available are erratic, perhaps due to the appearance of more than one solid phase.

A few data are available on the solubility of HgO in aqueous solutions of sodium sulfide (28) at 298 K. However, these data cannot be evaluated because of insufficient experimental detail in the article and the lack of other work dealing with the same system.

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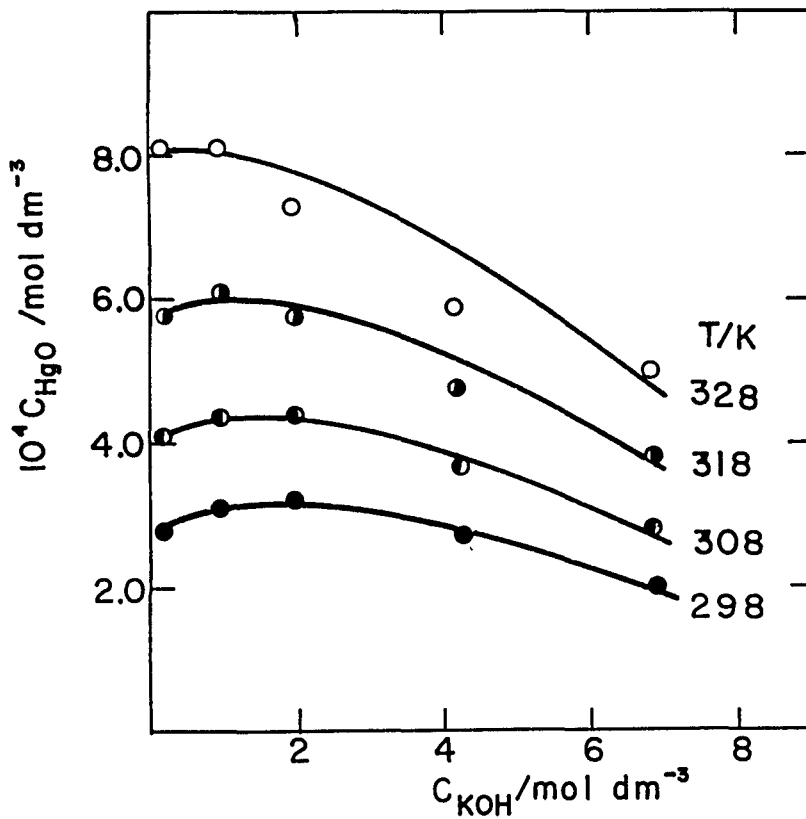


Figure 2. Solubility of HgO in aqueous KOH, ref. (35).

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CRITICAL EVALUATION:

Solubility at higher temperatures

The solubility of HgO in water at 308 K is 3.5×10^{-4} mol dm⁻³. The solubility in NaOH solutions is also larger at this temperature than at 298 K. Furthermore, the point of minimum solubility is in the very dilute alkaline region, Figure 1, rather than in water as it is at 298 K. This indicates that raising the temperature increases the basic nature of Hg(OH)₂ relative to its acid character.

Scholder and Staufenbiel (29) attempted to prepare mercurates in a manner similar to that by which they prepared cadmates. They were unsuccessful in their attempts to isolate solid mercurates. But in the course of this work they did measure the solubility of HgO in 18.8 mol NaOH dm⁻³ at 415 K. They obtained a value of 2.1 g HgO dm⁻³, i.e., 9.7×10^{-3} mol dm⁻³. However, this is the only value reported and only a scant bit of information is given as to the experimental details and no information about the analytical method used to obtain this value.

The solubility of HgO in aqueous KOH solutions has been measured at elevated temperatures using an e.m.f. method (35). The solubility of HgO increases with increasing temperature and has a maximum at a KOH concentration of about 1 mol dm⁻³, Figure 2. No other work has been reported for these same conditions, and in no instance has a maximum in solubility been noted in either KOH or NaOH solutions. Therefore, although the work has been carried out carefully, the results can only be considered tentative.

Unfortunately, no pH values are given for the acetic acid-acetate mixtures used for the solubility study at 308 K (30). The data show no apparent relationship between solubility of HgO and calculated pH values. The solubility values are perhaps affected by the formation of various acetate complexes with the Hg²⁺ ions but the data are too scattered to allow for any quantitative interpretation of this possibility.

Table II

Recommended solubility values for HgO in water.

<u>temp</u>	<u>Solubility</u>
298.2 K	2.37×10^{-4} mol dm ⁻³
308.2 K	3.47×10^{-4} mol dm ⁻³

Solubility of HgO in sea water

Interest in this system is related to the fact that HgO is an ingredient of some marine antifouling paints. Two papers deal with this subject (31, 32). Only one (32) gives experimental data. These data were obtained in Baltic Sea water. Solubility data were also obtained in water and these agree with the values recommended in Table II above. However, in the article there is a disagreement in the values reported for the solubility of the oxides and nowhere is this disagreement resolved.

The other article (31) also deals with the solubility of HgO in sea water at a pH = 8.1. Only one value is reported and that value is obtained, not from experiment, but from a calculation using the solubility product of Hg(OH)₂ and the dissociation constant of HgCl₂. No temperature is specified. The one value given is $80,000 \times 10^{-6}$ g ml⁻¹. This is equivalent to 0.37 mol dm⁻³ which is considerably larger than the value reported for Baltic Sea water (32).

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CRITICAL EVALUATION:

Solubility of HgO under hydrothermal conditions.

In recent years the solubility of HgO has been of interest to those studying various geochemical processes. As yet relatively few data have been reported in this area. Shlyapnikov and Shtern (33,34) measured the solubility of HgO in NaCl solutions exposed to a CO₂ atmosphere at varying pressures. At a pressure of 50 atm CO₂ the solubility of HgO in water at 293 K is reported as 0.0010 mol dm⁻³. This is larger than the solubility in water when exposed to ordinary atmospheres. It is also about twice as large as the value in carbonate solutions (24). However, no information is given as to the experimental uncertainty. Because of this and the fact that no other similar data have been reported, the solubility values given by Shlyapnikov and Shtern are tentative and must await further confirmation.

The solubility of HgO in solutions under a carbon dioxide atmosphere at an elevated pressure increases markedly as the concentration of NaCl in the water increases. This is due to the formation of mercury-chloro and basic mercury-carbonato solute species.

<p>COMPONENTS:</p> <p>(1) Mercury(II) oxide; HgO; [21908-53-2]</p> <p>(2) Water; H₂O; [7732-18-5]</p>	<p>EVALUATOR:</p> <p>T. P. Dirkse Department of Chemistry Calvin College Grand Rapids, Michigan 49506, U.S.A. March 1983</p>
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