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## Partitioning of mercury in aqueous biphasic systems and on ABEC<sup>TM</sup> resins

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### Abstract

Poly(ethylene glycol)-based aqueous biphasic systems (PEG-ABS) can be utilized to separate and recover metal ions in environmental and hydrometallurgical applications. A concurrent study was conducted comparing the partitioning of mercury between aqueous layers in an ABS [Me-PEG-5000/(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>] and partitioning of mercury from aqueous solutions to aqueous biphasic extraction chromatographic (ABEC-5000) resins. In ammonium sulfate solutions, mercury partitions to the salt-rich phase in ABS, but by using halide ion extractants, mercury will partition to the PEG-rich phase after formation of a chloro, bromo or iodo complex. The efficacy of the extractant increases in the order Cl<sup>-</sup> < Br<sup>-</sup> < I<sup>-</sup>. This behavior is also observed using the ABEC resins where halo complexes of mercury will adsorb to the resin from (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solutions with retention following the same order. The onset of mercury extraction or adsorption is different for the three extractants, occurring at the lowest extractant concentration for I<sup>-</sup>, followed by Br<sup>-</sup>, and then Cl<sup>-</sup>. Fluoride does not extract mercury. Extraction or adsorption of mercury is improved at the lowest halide concentrations in the presence of sulfuric acid. The addition of sulfuric acid to (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solution results in ABEC retention of mercury even in the absence of halide extractant. © 1998 Elsevier Science B.V. All rights reserved.

*Keywords:* Partitioning; Aqueous two-phase systems; Aqueous biphasic extraction chromatography; Mercury

### 1. Introduction

Aqueous biphasic systems (ABS), formed when a water soluble polymer [e.g., poly(ethylene glycol), PEG] is mixed with certain water-structuring salts [e.g., (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, K<sub>2</sub>CO<sub>3</sub>, K<sub>3</sub>PO<sub>4</sub>] [1–4], and the related aqueous biphasic extraction chromatographic resins (ABEC) [5,6], comprised of poly(ethylene glycol) covalently bound to an inert support, may represent relatively benign systems for extractive processes for the separation of metal ions and organic molecules in industrial separations as well as

environmental remediation applications [7–9]. Separation processes are ubiquitous in industry and systems employed in liquid–liquid separations processes frequently involve the use of toxic and volatile organic compounds.

Given the current emphasis on “Green Chemistry” and the desire to prevent pollution by redesign of industrial processes, ABS and ABEC resins represent an economical avenue for “clean” separations [10]. Unfortunately, the partitioning behavior of solutes in ABS and with ABEC resins is poorly understood at present, especially for the potentially most useful polymer salt systems whose physical (and presumably extractive) properties are most nearly similar to current aqueous/organic systems.

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Such data is essential for the efficient evaluation and application of these systems.

Applications for improved separations of mercury arise both in removal of mercury from the environment and in reduction of mercury contamination in hydrometallurgical processes. Mercury(II) must often be separated from sulfuric acid solutions of Cu(II) in the processing of copper ores. The importance of mercury as an environmental contaminant stems from its ubiquitous nature, due largely to the multitude of sources, its volatility, its mobility, and its persistence in the environment [11]. The major form of mercury in the atmosphere is Hg(0), but the major form of deposited mercury is Hg(II) [12]. In this report, the partitioning of Hg(II) in Me-PEG-5000/(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> ABS and onto ABEC-5000 resins is detailed as a function of sodium halide extractant and sulfuric acid concentration.

## 2. Experimental

The chemicals, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, K<sub>2</sub>CO<sub>3</sub>, NaOH, NaI, NaBr, NaCl, NaF, and poly(ethylene glycol) 5000 monomethyl ether, were obtained from Aldrich (Milwaukee, WI, USA) and were of reagent grade. ABEC-5000 was obtained from EIChrom Industries (Darien, IL, USA). The <sup>203</sup>Hg tracer (as HgCl<sub>2</sub>) was obtained from Isotope Products Laboratories (Burbank, CA, USA). The HgCl<sub>2</sub> was converted to Hg(NO<sub>3</sub>)<sub>2</sub> by evaporation to near dryness twice in concentrated HNO<sub>3</sub> and then diluted to an activity of ca. 0.03 μCi/μl.

The experiments were carried out using the methods from reference [13]. Liquid–liquid distribution ratios (*D*) were determined by mixing 1 ml of Me-PEG-5000–water (40%, w/w) solution with 1 ml of a salt stock solution of known concentration. The systems thus prepared were vortex mixed for 2 min and then centrifuged (2000 *g*) for 2 min. Each system was then spiked with 2 μl of the previously prepared <sup>203</sup>Hg(NO<sub>3</sub>)<sub>2</sub> tracer. The system was then vortex mixed and centrifuged (2000 *g*) twice for 2 min each. Equal aliquots of each phase were then analyzed for Hg by radiometric counting using a Packard Cobra II Auto-Gamma Spectrometer (Packard Instrument, Downers Grove, IL, USA). The distribution ratio represents the total concentration of

mercury in the upper PEG-rich phase divided by the concentration of the total mercury concentration in the lower salt-rich phase. Since equal aliquots of each phase were analyzed and the activity of the mercury tracer is directly proportional to its concentration, *D* can be defined as:

$$D = \frac{\text{Activity in counts per minute PEG-rich phase}}{\text{Activity in counts per minute salt-rich phase}}$$

Batch mode dry-weight distribution values were determined as follows. A salt stock solution was spiked by adding 2 μl of mercury tracer per ml of salt stock solution and a 100 μl sample was removed and counted to determine the initial activity of the solution. One ml of the spiked solution was then contacted with ca. 15–20 mg of ABEC-5000. After centrifugation, the solution was stirred for 30 min, centrifuged (2000 *g*), and then stirred for another 30 min. The solution was then filtered through a 0.45 μm filter and a 100 μl sample removed for the radiometric determination of the final solution activity. The radiometrically determined distribution ratio (*D<sub>w</sub>*) is calculated from the following equation:

$$D_w = \frac{A_i - A_f}{A_i} \cdot \frac{\text{contact volume}}{\text{resin mass} \cdot \text{dwcf}}$$

where *A<sub>i</sub>* is the activity in counts per minute in the initial sample and *A<sub>f</sub>* is the activity in counts per minute after contact with the adsorbent. The contact volume is the total volume of the adsorbate, and dwcf is the dry weight conversion factor of the resin.

$$\text{dwcf} = \frac{\text{mass dehydrated resin}}{\text{mass wet resin}}$$

The dwcf is determined from the weight of the resin dried to constant weight at 100°C in a suitable drying oven.

## 3. Results and discussion

In the formation of PEG/salt ABS, the ability of a salt to form an ABS can be related to the salt anion's Gibbs free energy of hydration [14]. Although both cation and anion contribute to the salting-out phenomenon, the contribution of the anion dominates. The more negative the Δ*G*<sub>hyd</sub> of the anion, the less

salt is required to salt-out the PEG. Using the same consideration, metal ions with relatively small magnitudes of  $\Delta G_{\text{hyd}}$  are chaotropic, tend to destabilize the hydrogen bonding structure of water, and partition to the PEG-rich phase in an ABS [14]. The relatively soft anion  $\text{TcO}_4^-$ , with  $\Delta G_{\text{hyd}} \approx -175$  kJ/mol [15] partitions quantitatively to the PEG-rich phase in ABS [7,8]. Hard metal cations with relatively large magnitudes of  $\Delta G_{\text{hyd}}$  report to the structured salt-rich phase [5].

In order to extract hard metal cations from the ordered salt-rich phase to the PEG-rich phase, it is necessary to change the hydration environment of the metal ion. This can be accomplished using extractants, which may either be organic ligands (e.g., crown ethers [5,16]) which give a metal ion a more hydrophobic exterior, or they may be inorganic anions (e.g.,  $\text{I}^-$ ,  $\text{SCN}^-$ ) which form softer extractable anionic complexes [5,17]. In both cases, it is necessary to take into consideration the strength of the ligand–metal interaction and the hydration properties of the extractant–metal complex.

In our research comparing liquid–liquid extraction using ABS and adsorption using ABEC, the partitioning of the solutes to the PEG-rich phase follows the same trends as the adsorption to the ABEC resin [5]. Chaotropic ions in high ionic strength solutions of salting-out agents have an affinity for the ABEC resin. Thus, extractions modeled using ABS systems, may in practice be applied using batch or column adsorption to ABEC.

Mercury(II) as the free or hydrated ion, with  $\Delta G_{\text{hyd}} = -1495$  kJ/mol [15], would be expected to remain in the salt-rich phase of an ABS and not to be retained by the ABEC resin. Following a procedure developed for extraction of Bi(III), Cd(II) and Pb(II) in ABS [5,17], it should be possible to extract Hg(II) using halide ions to form an extractable metal–halide complex.

In the absence of an extractant, Hg(II) behaves as most other metal cations. The distribution ratio of 0.25 (Table 1) in Me-PEG-5000/3.5 M  $(\text{NH}_4)_2\text{SO}_4$  ABS (40%, w/w), and the ABEC-5000  $D_w$  of 2.44 from 3.5 M  $(\text{NH}_4)_2\text{SO}_4$ , both indicate little affinity of the Hg(II) ion for the PEG-rich phase or the ABEC resin.

Fig. 1 illustrates the partitioning behavior of Hg(II) in an ABS formed by mixing equal aliquots

of Me-PEG-5000 (40%, w/w) and 3.5 M  $(\text{NH}_4)_2\text{SO}_4$ , where the salt stock solution contains increasing quantities of sodium halide salts. As we have previously observed for extraction of Bi(III) [17], the halide salts (except for NaF) enhance the partitioning of Hg(II) from the salt-rich to PEG-rich phases. It is interesting to note the different effects of the series of halide extractants. The iodide ion has the smallest magnitude of  $\Delta G_{\text{hyd}}$  and consequently, the highest formation constant [18] with Hg. The trend in increasingly negative Gibbs free energy of hydration and decreasing formation constants for Hg(II) complexation (Table 1) follows the decreasing size of the halide ions in the series  $\text{I}^- > \text{Br}^- > \text{Cl}^- > \text{F}^-$ . (It should be noted that the hard fluoride ion, alone in the halide series, is actually capable of salting-out PEG to form an ABS).

It would be expected that given equivalent charges, the largest and most chaotropic anion ( $\text{I}^-$ ) would produce the most chaotropic metal–halide complex. (It would have the smallest charge:size ratio). In addition, significant complexation with Hg(II) should occur for this anion at lower halide concentration [as evidenced by the formation constants of the  $\text{HgI}_4^{2-}$  complex [18] versus the other metal–halide anions (Table 1)]. Thus, enhancement of Hg(II) partitioning to the PEG-rich phase occurs at a much lower NaI concentration than observed for NaBr or for NaCl.

The distribution ratios increase toward a maximum of 1000. The NaI extractant reaches this level at the lowest NaX concentration, however, by 0.5 M NaBr, the  $D$  values are the same whether iodide or bromide is used. The distribution ratios in the NaCl systems never quite reach the 1000 plateau, but are still increasing at 0.5 M NaCl.

NaF has no effect except at higher concentrations where it actually depresses the distribution ratios for Hg. This may be explained by the lack of complex formation and the tendency of  $\text{F}^-$  to salt-out PEG. As the concentration of fluoride is increased, the binodal is shifted toward lower concentrations, resulting in a greater difference in composition between the two phases. This enhances partitioning of a solute in the direction of preference. Since Hg(II) prefers the salt-rich phase in the absence of a suitable extractant, the distribution ratios decrease as the influence of  $\text{F}^-$  is felt on the ABS.

Table 1  
Parameters for the halide ions and mercury halide complexes

Halide	Complex	$\Delta G_{\text{hyd}}$ (kJ/mol) [15]	$D$ (Hg)		log $K$ ( $\mu = 0.5$ ) [18]
			0.3 M NaX/ 3.5 M (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0.4 M NaX/ 3.5 M (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	
No halide	Hg(II)	−1495	0.25	2.44	
F <sup>−</sup>	HgF <sup>+</sup>	−345	0.203	14.7	1.03
Cl <sup>−</sup>	HgCl <sup>+</sup>	−270	4.04	2160	6.74
	HgCl <sub>2</sub>				13.22
	HgCl <sub>3</sub> <sup>−</sup>				14.2
	HgCl <sub>4</sub> <sup>2−</sup>				15.2
	HgCl <sup>+</sup> /H <sup>+</sup> <sup>a</sup>				3.05
Br <sup>−</sup>	HgBr <sup>+</sup>	−250	424	16 200	9.07
	HgBr <sub>2</sub>				17.27
	HgBr <sub>3</sub> <sup>−</sup>				19.7
	HgBr <sub>4</sub> <sup>2−</sup>				21.2
	HgBr <sup>+</sup> /H <sup>+</sup> <sup>a</sup>				3.37
I <sup>−</sup>	HgI <sup>+</sup>	−220	822	195 000	12.87
	HgI <sub>2</sub>				23.82
	HgI <sub>3</sub> <sup>−</sup>				27.6
	HgI <sub>4</sub> <sup>2−</sup>				29.8
	HgI <sup>+</sup> /H <sup>+</sup> <sup>a</sup>				4.0
HSO <sub>4</sub> <sup>−</sup>		−1145 (SO <sub>4</sub> <sup>2−</sup> )			
	HgHSO <sub>4</sub> <sup>+</sup>				1.34
	Hg(HSO <sub>4</sub> ) <sub>2</sub>				2.4

<sup>a</sup> [MX]/[MOHX][H].

The uptake of Hg(II) with ABEC-5000 from the same ammonium sulfate solutions used to generate the biphasic systems above, is depicted in Fig. 2. The values of  $D_w$  are higher than observed for the  $D$  values in the liquid–liquid ABS, but the trends observed are the same: (1) the  $D_w$  values in the NaI and NaBr systems tend toward a common maximum (171 000) at the highest halide concentrations; (2) these same values in the NaCl system are still trending upward although not quite reaching the 100 000 level; and (3) NaF has no effect on the  $D_w$  values for Hg(II). In addition, the onset of increasing  $D_w$  with increasing [NaX] again occurs earliest for NaI, followed by NaBr, and finally by NaCl. The higher  $D_w$  values observed for ABEC compared to

distribution ratios for ABS, may be explained by a higher effective PEG concentration on the resin bead. (That is, the local concentration the solution actually sees on the resin is effectively higher than in the liquid–liquid ABS). Because of the higher  $D_w$  values, the halide extractants increase Hg partitioning at lower NaX concentrations, and even by 0.1 M NaX, there is significant separation between the  $D_w$  values measured with each halide salt. It is interesting to note that although higher, there is a maximum  $D_w$ , just as there is a maximum  $D$  in the ABS.

Although there will be some differences in salt concentration between the salt-rich phase in ABS versus the salt stock solution contacted with ABEC resins, the trends in uptake measured in ABS can

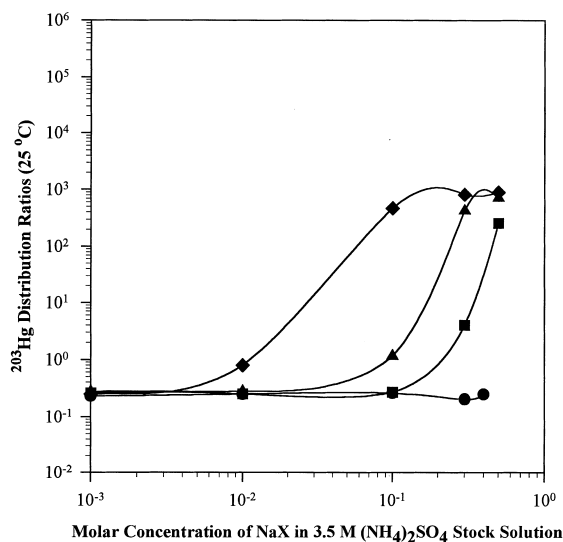


Fig. 1. Distribution ratios for  $^{203}\text{Hg}$  in ABS formed by mixing equal aliquots of 40% (w/w) Me-PEG-5000 and 3.5 M  $(\text{NH}_4)_2\text{SO}_4$  (pH 5.5) containing increasing amounts of sodium halide: (◆) NaI; (▲) NaBr; (■) NaCl; (●) NaF. The distribution ratio from this ABS without added halide salt is 0.25.

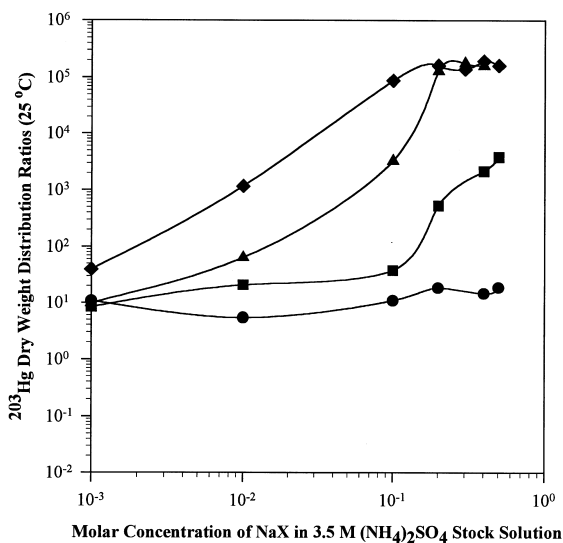


Fig. 2. Dry weight distribution ratios with ABEC-5000 for  $^{203}\text{Hg}$  from 3.5 M  $(\text{NH}_4)_2\text{SO}_4$  (pH 5.5) containing increasing amounts of sodium halide: (◆) NaI; (▲) NaBr; (■) NaCl; (●) NaF. The dry weight distribution ratio from this salt stock solution without added halide salt is 2.44.

still be used to predict ABEC performance. This will be true regardless of whether ABEC operates with an adsorption or partition mechanism. We are currently developing an assay that will allow a more direct comparison of ABS and ABEC.

Many applications of mercury separation in hydro-metallurgy present themselves if it is possible to selectively partition Hg(II) in sulfuric acid or sulfate solutions. Purification of copper in ore processing (following sulfuric acid leaching) by removal of Hg(II) is but one example. In order to further investigate the affects of sulfuric acid on the partitioning of Hg(II) (since acid is known to destroy biphasic behavior [19]), we studied the adsorption of Hg(II) to ABEC-5000 from  $\text{H}_2\text{SO}_4$ , from various concentrations of  $\text{H}_2\text{SO}_4$  in 3.5 M total  $\text{SO}_4^{2-}$ , and from solutions containing NaX/2 M  $\text{H}_2\text{SO}_4$ /3.5 M total  $\text{SO}_4^{2-}$ .

The results depicted in Figs. 3 and 4, provide some interesting insight into these systems. From 3.5 M  $(\text{NH}_4)_2\text{SO}_4$ , ABS behavior is expected and is in fact observed. The Hg(II) ions do not adsorb to the ABEC resin. In the presence of small concentrations of sulfuric acid, however, the  $D_w$  values are ca. 200 (Fig. 3), despite the fact that this solution will not

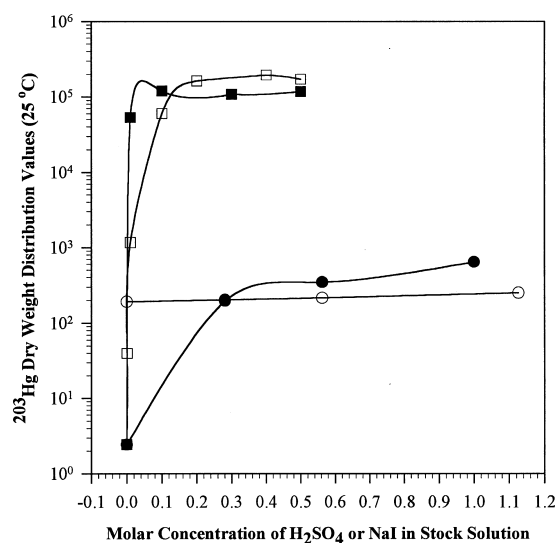


Fig. 3. Dry weight distribution ratios with ABEC-5000 for  $^{203}\text{Hg}$  from several solutions with varying concentrations of NaI,  $\text{H}_2\text{SO}_4$  and  $(\text{NH}_4)_2\text{SO}_4$ : (□) [NaI] in 3.5 M  $(\text{NH}_4)_2\text{SO}_4$ ; (■) [NaI] in 2 M  $\text{H}_2\text{SO}_4$ /3.5 M total  $\text{SO}_4^{2-}$ ; (●) [ $\text{H}_2\text{SO}_4$ ] in 3.5 M total  $\text{SO}_4^{2-}$ ; (○) [ $\text{H}_2\text{SO}_4$ ].

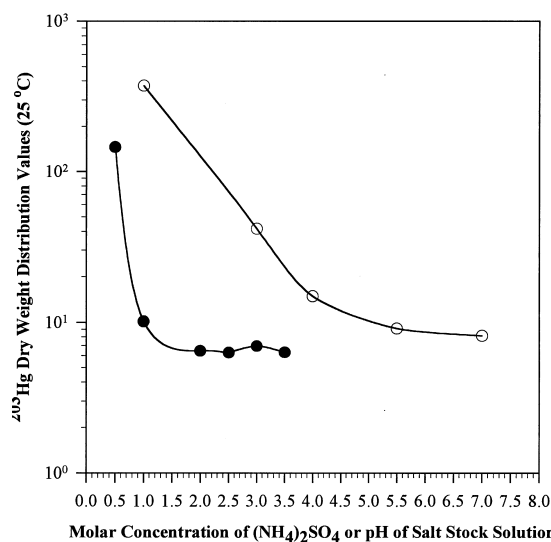


Fig. 4. Dry weight distribution ratios with ABEC-5000 for <sup>203</sup>Hg from 2.5 M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> as a function of pH and from (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solutions of increasing concentration: (○) pH in 2.5 M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>; (●) [(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>].

salt-out Me-PEG-5000 and will thus not support an ABS. It is also apparent that there is little affect of increasing sulfuric acid concentration, the apparent maximum  $D_w$  reached at the lowest acid concentration studied.

Since ammonium sulfate is itself acidic, we tested the uptake of Hg(II) from increasing concentrations of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and found that the  $D_w$  values dropped quickly from 146 at 0.5 M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> to 6.3 at 3.5 M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> (Fig. 4). This pH affect can be duplicated by varying the pH in a given concentration of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>. At pH 1, the Hg  $D_w$  from 2.5 M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> is 371, but drops steadily to 8.2 by a pH of 7.

The acid catalyzed formation constants for the Hg(II) halide complexes are given in Table 1. From the results in Fig. 3 it is apparent that the presence of acid enhances the uptake of the mercury halide complexes, presumably by enhancing the formation constants. It is noteworthy that the maximum  $D_w$  using NaI is reached at the lowest studied concentration of NaI when the salt solution is sufficiently acidic, but the 171 000  $D_w$  is still not exceeded.

Fig. 5 presents the results for uptake of Hg(II) from solutions that are 2 M in (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 3.5 M in

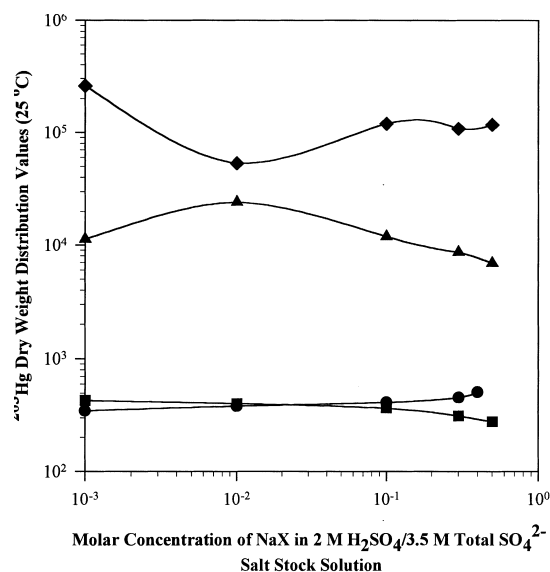


Fig. 5. Dry weight distribution ratios with ABEC-5000 for <sup>203</sup>Hg from solutions of 2 M H<sub>2</sub>SO<sub>4</sub>/3.5 M total SO<sub>4</sub><sup>2-</sup>, and containing increasing amounts of sodium halide: (◆) NaI; (▲) NaBr; (■) NaCl; (●) NaF. The dry weight distribution ratio from this salt stock solution without added halide salt is 399.

total sulfate [using (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>], and have increasing amounts of NaX. Increasing the sodium halide concentration, now has very little affect, with the maximum  $D_w$  achieved at the lowest halide concentration studied (0.001 M). The NaCl and NaF values are identical to the  $D_w$  values from similar solutions without NaX present (Fig. 3). The NaI values have immediately reached their 171 000 maximum value, but the NaBr  $D_w$  values actually have a lower maximum (10 000) than observed in the absence of the acid.

It is clear that speciation is very important in understanding the partitioning behavior of metal ion solutes. Further work is needed to define each species present in the solutions studied here, especially for those systems for which ABEC retention occurred in the absence of biphasic forming conditions. The uptake observed from acid alone and from water were clearly unexpected from previous studies, yet these results may hold the promise for future applications of ABEC. Removal of Hg from the environment would likely occur from groundwater with low ionic strength. Separation of Hg(II) from Cu(II) or other metals in ore refining, would be

most beneficial if it was possible to carry out the separation from sulfuric acid. Thus, the unexpected results provide the impetus for further research in this area.

#### 4. Conclusions

Mercury(II) can be partitioned to a PEG-rich phase or to ABEC resin from high ionic strength sulfate solutions using halide ions to produce extractable Hg(II)–halide complexes. The efficacy of the extractants follows the Gibbs free energy of hydration and HgX formation constants of the halide ions:  $F^- < Cl^- < Br^- < I^-$ . Distribution ratios as high as 907 are possible in Me-PEG-5000%/3.5 M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> ABS (40%, w/w) using NaI, and  $D_w$  values as high as 171 000 are observed using ABEC to adsorb Hg(II) from the same solutions. Acid enhances the adsorption to ABEC whether in the presence or absence of extractant or even biphasic forming salt. Further work with these systems will be directed toward relating the speciation of Hg(II) complexes to partitioning behavior and to application of ABS and ABEC resins to Hg(II) removal from the environment or in hydrometallurgical applications.

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