

Ultralow Concentration Mercury Treatment Using Chemical Reduction and Air Stripping (U)

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Abstract

Field, laboratory and engineering data confirmed the efficacy of chemical reduction and air stripping as an ultralow concentration mercury treatment concept for water containing Hg(II). The simple process consists of dosing the water with low levels of stannous chloride (Sn(II)) to convert the mercury to Hg⁰. This mercury species can easily be removed from the water by air stripping or sparging. We used samples of groundwater containing mercury concentrations of approximately 138 ng/L (0.00069 μMol/L) for the study. In undosed samples, sparging removed 0% of the initial mercury. Removal in the treated samples varied by reagent dose. Low doses (<0.001 μMol/L stannous chloride) showed little removal. Stannous chloride doses above 0.05 μMol/L showed relatively complete removal (>94%) and final mercury concentrations < 10 ng/L (<0.00005 μMol/L). At stannous chloride doses between 0.001 μMol/L and 0.05 μMol/L, mercury removal was a function of the dose. In general, effective mercury removal was achieved with doses ranging from approximately 5x to 25x the theoretical minimum stoichiometry (based on the redox reaction between Hg(II) and Sn(II)). A kinetic study indicated that addition of the Sn(II) reagent resulted in rapid reduction of Hg(II) to Hg(0). In treated samples, the purging process, rather than the mercury conversion kinetics, controlled the mercury removal rate from solution. The data indicate that the reduction of mercury is highly favored and that stannous chloride reagent efficiently targets the Hg(II) contaminant in the presence of competing reactions.

Introduction

Over the past ten years the U. S. Environmental Protection Agency (EPA) has developed a policy aimed at curtailing mercury emissions. This policy has been articulated in the PBT (Persistent, Bioaccumulative and Toxic) Pollutants Program (1, 2), the White House Clean Water Action Plan (3), the Mercury Report to Congress (4), and the U.S.-Canada Bilateral Toxics Agreement (5). In each case, mercury was identified as a particularly significant contaminant and specific actions to reduce and/or eliminate mercury were required. Significantly, the surface water protection standards promulgated to support these various initiatives, typically in the range of 1 to 50 ng/L total mercury, are significantly below drinking water standards (typically 2000 ng/L). Technical evaluation and support of low ng/L target concentrations required development and approval of improved analytical methods (EPA Method 1631 (6)). A policy of regulating mercury to ultralow concentrations will impact industry, municipalities and other government agencies. New water treatment approaches are needed to support rational implementation of such a mercury reduction policy. To be viable, these approaches must treat large volumes of water containing trace levels of mercury in the presence of other ions at a unit cost that is below conventional metals removal methods. Integration of such treatment approaches into a balanced and technically-based mercury policy will assist in reducing mercury releases and impacts to levels that are “as low as reasonably achievable.”

We are examining a simple “alternative” treatment method to remove mercury from water and wastewater. The basis for this project is the chemistry embodied in various analytical methods for mercury (6, 7). In these methods, inorganic Hg(II) is

reduced to Hg^0 using stannous (Sn(II)) chloride. Hg^0 is volatile and can be removed from the water by simple air-water contact. In the lab, a small sparge apparatus is used to strip the mercury. For full-scale treatment, air stripping, spraying, or sparging are examples of inexpensive air-water contactors for the mercury removal step. Figure 1 is a schematic diagram depicting the simple laboratory and full-scale concept.

In the reference analytical methods, the reaction of tin and inorganic mercury appears rapid and thermodynamically favored. Significantly, tin-based analytical methods rely on using excess reagent to assure that the reaction is complete. Such high reagent concentrations may be incompatible with practical implementation of this process for large volume water treatment. Research by Southworth (8) suggested that tin levels that are only 4 to 5 times stoichiometry may convert the available inorganic mercury to Hg^0 . Such a stoichiometry, under their particular set of water chemistry conditions, suggested that the mercury-tin reaction is relatively specific. These ratios also indicated that treatment may be possible using tin concentrations that are well within safe-protective levels for both ecological and human health. Further, Southworth (8) documented that the strippability of the resulting Hg^0 is predictable and that required air-water ratios are favorable (e.g., ratios less than 20 provided removal). Successful development of a chemical-reduction-based treatment system requires additional data related to the key scientific questions (stoichiometry, robustness, etc.) and engineering evaluation of the achievability of reliable long-term operation. These data, and similar low concentration performance data for traditional treatment methods are needed to support a technically based mercury policy (9,10).

Relevant Mercury Chemistry

The chemistry of mercury in the aqueous systems is complicated by multiple redox states, a tendency to form complex ions, and potential biological transformation. Mercury can exist naturally as the elemental form (Hg^0), a univalent form [Hg(I)], and a divalent form [Hg(II)]. Figure 2 shows the relationship between the Sn(II)/Sn(IV) redox couple and the speciation of mercury in aqueous solution. In highly oxidized natural waters (above a pE of approximately 8), thermodynamics predicts that mercury will occur as Hg(II), typically as a chloride or hydroxide complex. The equal activity line of the Sn(II)/Sn(IV) couple is located within the field of dominance of elemental mercury indicating that Sn(II) will reduce Hg(II) to elemental mercury. This occurs by the following reaction:



Thus, when sufficient stannous chloride is added to water containing dissolved Hg(II), virtually all Hg(II) is expected to be reduced to elemental mercury. Importantly, mercury occurs primarily as Hg(II), and its complexes, in many natural waters and wastewaters, even water where Hg^0 is thermodynamically predicted based on stability (e.g., pE < 8). In these systems, the reaction with tin facilitates the transformation of the Hg(II) to the expected zero valence oxidation state. In all cases, the stannous ion must be added in sufficient quantity that a 1:1 Hg:Sn stoichiometry is available in the presence of competing redox reactions.

The remediation technology being tested exploits the redox chemistry of mercury and the relatively high vapor pressure of elemental mercury. Consistent with equation 1, a solution containing stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) is used as the primary reagent. The

stannous chloride will also react with other dissolved constituents such as oxygen, nitrate, and organic matter. Oxygen and nitrate are typically present in groundwater at concentrations that are much greater than concentrations of mercury. Likewise, in groundwater containing organic contaminants, mercury concentrations may be small compared to these compounds. The relatively high concentrations of competing constituents relative to mercury may result in a substantially higher dose requirement of stannous chloride than for simple stoichiometric reduction of mercury. We performed our experiments under a variety of conditions to develop a preliminary evaluation of the reaction stoichiometry under a range of matrix conditions.

Experimental Section

Overview

Treatment performance was assessed using two distinct approaches. The primary approach was a dose-response study to determine the effectiveness of the treatment under different water quality and reagent dosing conditions. The second was a kinetic study to determine if the mercury reduction step would be rate limiting in a practical application. The analytical methods used to support both studies are summarized below followed by a description of key aspects of each of the performance assessment studies.

Analytical

Frontier Geosciences (Seattle WA) analyzed all of the samples in this study using cold vapor atomic fluorescence spectrometry (CVAFS) – EPA method 1631 (6). As specified in this method, we determined the detection limit and standard curve, and analyzed a certified reference material, along with each set of samples. For every 20 field samples, we analyzed at least three method blanks, analyzed a representative field

sample in triplicate, and performed a standard addition. Throughout the study, we estimated the method detection limit (MDL) for total mercury was approximately 0.05 to 0.1 ng/L, the relative percent difference (RPD) was <10% and the recovery of mercury from standard material and standard additions was > 94%.

Dose Response Study

These experiments evaluated the residual total mercury concentration in treated samples as a function of stannous chloride doses. The matrix water used for this study was collected from the feed and effluent of an existing groundwater treatment system. The primary selection criteria for the water were: 1) concentrations of mercury should be well characterized and stable, 2) concentrations of mercury should be elevated above background but less than drinking water standards, 3) water from the selected source should be accessible for sampling and study, 4) mercury in the water should be primarily in the form of Hg(II), and 5) if possible, the water should discharge to a surface water outfall and represent a possible future treatment target. Based on these criteria, we selected the influent and effluent of the A-Area stripper at the Department of Energy Savannah River Site (SRS) for the work.

The water originates from groundwater wells installed to remediate a plume of chlorinated solvents. The feed and effluent of the air stripper have been well characterized and provide a very stable source of water that contains approximately 120 to 150 ng/L total mercury (0.00060 to 0.00075 $\mu\text{Mol/L}$). Previous speciation studies on these waters indicated that >95% of the mercury is Hg(II). Interestingly, since an air stripper is already in place, the measured concentrations indicate that mercury is not being removed by this unit operation without the benefit of reagent addition. The use of

both feed and effluent water provides an interesting comparison of stannous chloride effectiveness in the presence of varying competing reactions. The feed contains significant levels of chlorinated organic solvents (880 $\mu\text{g/L}$ trichloroethylene and 25 $\mu\text{g/L}$ tetrachloroethylene) while the effluent contains <1 $\mu\text{g/L}$ of these constituents. Dissolved oxygen is present in both the feed (approximately 3 mg/L) and the effluent (approximately 9 mg/L). To supplement these two water types, we performed runs on A-Area Stripper effluent water that was pre-sparged with nitrogen to remove both dissolved oxygen as well as any residual volatile organic compounds. This pre-sparged water provided data supporting estimation of the optimum theoretical performance of the treatment method. Thus, each of the stannous chloride doses was tested using the three matrix waters – stripper feed, stripper effluent, and nitrogen-sparged stripper effluent. The effluent from the A-Area Stripper discharges to the A-01 Outfall and represents a significant fraction of the outfall flow volume. Thus, the stripper represents a potential treatment target if ultralow concentration outfall standards are promulgated.

Figure 3 is a simplified diagram of the equipment used for the study. All dose-response work was performed a nitrogen-filled mercury-free polycarbonate glovebox set up in the field or the same glovebox set up in a nearby support laboratory. The sparge containers consisted of modified 500 mL glass bottles with teflon lined polyethylene lids. Each lid was equipped with two polyvinylidene fluoride (PVDF) bulkhead unions. A coarse PYREX diffuser was attached to the air inlet union. The tubing used in the system was either PVDF or glass. The high purity laboratory grade nitrogen and zero air were treated using MERSORB (sulfur impregnated activated carbon manufactured by NUCON International, Columbus OH) to provide a low mercury environment within the glovebox

and to provide sparge gas. Handling and sampling were performed using powder free latex gloves and clean procedures.

Prior to the actual dose experiments, a blank run was performed on every cleaned sparge vessel. This blank run was followed by experimental run(s) that tested stannous chloride dosages from 0 to approximately 3400 $\mu\text{Mol/L}$ (or 766 mg/L – mass based reagent doses expressed in terms of stannous chloride dihydrate). Specifically, we tested reagent doses of 0 $\mu\text{Mol/L}$ (0 mg/L), 0.00000269 $\mu\text{Mol/L}$ (0.000000616 mg/L), 0.0000273 $\mu\text{Mol/L}$ (0.00000616 mg/L), 0.00137 $\mu\text{Mol/L}$ (0.000308 mg/L), 0.0133 $\mu\text{Mol/L}$ (0.003 mg/L), 0.665 $\mu\text{Mol/L}$ (0.15 mg/L), 6.83 $\mu\text{Mol/L}$ (1.54 mg/L), 340 $\mu\text{Mol/L}$ (76.6 mg/L), and 3400 $\mu\text{Mol/L}$ (766 mg/L). The protocol allowed study of doses at basic science and practical engineering levels and extended up to the approximate dosages actually used in the 1631 analytical method.

Following dosing we sparged each sample using air and an excess air-water ratio of approximately 30 to 1. The resulting samples were sealed and sent for total mercury analysis in the laboratory.

Kinetic Study

A kinetic study was performed to confirm that the reaction was sufficiently rapid and complete to be practical. This study was performed in the laboratories of Frontier Geosciences in Seattle WA to allow rapid subsampling following dosing and to avoid any possible artifacts associated with dosing and shipping. The study was designed to collect the gas phase reaction product ($\text{Hg}(0)$) as it was generated and sparged from the system. A range of concentrations, doses and temperatures were examined (Table 1). The kinetic study was performed using both deionized water and groundwater (a 5 L composite of

A01 Stripper effluent collected in a teflon bottle was used as the representative groundwater).

Purging experiments were conducted in acid cleaned 500 mL borosilicate gas washing bottles. Each bottle had a total volume of 575 mL and was charged with 500 mL of aqueous sample for each experiment. After dosing with stannous chloride, each sample was purged with purified nitrogen at a flow rate of 220 mL/min. Hg(0) evaded from the sample was collected on freshly blanked gold coated quartz sand traps (the same traps used in performing EPA analytical method 1631). Based on the gas flow rate and water volume, the purge rate with regard to the aqueous sample was 0.44 volumes/min. The samples were each purged for a total of 60 minutes. All of the stripped Hg(0) was collected on sequential gold/sand traps. In general, the traps were collected in 5-minute increments (or composited to 5 minute increments for reporting). Each 5-minute increment represents an air-water ratio of 2.2. In addition to the experimental runs, two blank runs were performed. For the blank runs, all of the Hg(0) collected during a 60 minute purging of mercury free reagent water was collected. These runs indicated a procedural blank of 0.0006 ng/minute – this value was subtracted from the raw results prior to final tabulation and further calculation. To provide an overall mass balance and assure experimental quality, samples of the bulk solution before and after the purging period were collected for each run. The mass balances (recovery of evaded mercury plus residual mercury in the reaction vessel) for all of the runs ranged from 85% to 109%. To simplify the analysis and calculations presented below, mercury removals are expressed in terms of actual mercury recovered in each run.

Results and Discussion

The dose response results are shown in Figure 4. Except as noted below, there was no apparent difference in the treatment effectiveness for the various waters tested. In the undosed samples, the treatment removed 0% (\pm approximately 15%). Removal in the treated samples varied by dose. Low doses ($<0.001 \mu\text{Mol/L}$ stannous chloride) showed little removal. Stannous chloride reagent doses above $0.05 \mu\text{Mol/L}$ showed relatively complete removal ($>94\%$) – the residual total mercury in all of these samples was reduced to levels below 10 ng/L ($0.00005 \mu\text{Mol/L}$).

Reagent doses between $0.001 \mu\text{Mol/L}$ and $0.01 \mu\text{Mol/L}$ responded in a regular manner generally consistent with the stoichiometry of the redox reaction between Hg(II) and Sn(II) . The curved lines on Figure 4 are calculations indicating expected performance based on 1x, 5x and 25x the theoretical stoichiometry. With only one exception, the data in this critical reagent dose range fell between 1x to 5x the theoretical optimum performance. This result is consistent with the data reported by Southworth (8). Competing reactions (e.g., with the organic contaminants in the stripper feed water and/or dissolved oxygen in the stripper feed water and the stripper effluent) do not appear to be adversely scavenging the reagent, despite the presence of competing molecules in solution at levels that are several orders of magnitude higher than the target Hg(II) . Notably, one sample was only partially treated at a dose of approximately 25x the theoretical stoichiometry. This single data point (for air stripper effluent) may indicate a detectable influence of high dissolved oxygen on treatment efficiency. A replicate of this sample, however, showed effective mercury removal to $<10 \text{ ng/L}$. Based on the results, a target dose range of 5x to 25x stoichiometry would be a reasonable design basis for treatment of waters similar to those tested.

The kinetic study indicated that addition of Sn(II) results in rapid reduction of Hg(II) to Hg(0). For all of the Sn(II) addition experiments, the mercury evasion rate appears to be controlled by the purging process rather than the reaction rate. In the three experiments conducted at 22.8° C., the mean removal rate ranged from about 27 to 29% of the Hg present per volume of purge gas (i.e., for each 500 mL of purge gas representing a 1:1 air:water ratio). For the single tin-dosed experiment at 0° C., the mean removal rate fell to about 19% of the Hg present per volume of purge gas. In both deionized water and in groundwater, virtually no mercury was evaded without the addition of Sn(II). These conclusions are clearly documented in Figure 5. This figure plots the laboratory data along with lines showing theoretical performance. The theoretical lines have not been fitted to the data and are based solely on purge efficiency calculated from literature values of solubility and vapor pressure of elemental mercury (Table 2).

For these relatively simple conditions, the literature values of vapor pressure and solubility can be combined into a “dimensionless” Henry’s Law constant (H’):

$$H' = \frac{\text{(vapor pressure in units of mg Hg per L of air)}}{\text{(solubility in units of mg Hg per L of water)}} \quad (2)$$

Based on mass balance and integration over time, the purge efficiency can then be approximated as a function of H’ and the ratio of air volume purged to water volume in vessel (η'):

$$\text{Hg}_{(\eta')} / \text{Hg}_{(0)} = e^{-(H' \eta')} \quad (3)$$

Normalized removal rates calculated using this equation correspond well with the those reported from the raw data (29% of the Hg present per volume of purge gas at 22.8° C. and 16% of the Hg present per volume of purge gas at 0° C). The theoretical lines in Figure 5, based solely on expected sparge rate, provide a reasonable match the measured data. This suggests that sparging controlled the mercury removal rate in the experiments and that the reaction kinetics ($\text{Hg(II)} \Rightarrow \text{Hg}^0$) are relatively fast and are not the rate limiting step in the process.

Chemical reduction followed by air stripping is a promising, low-cost option for treating mercury in some outfalls. This technique does not produce any liquid or solid secondary wastes and has low capital, maintenance, and operations costs (13). Offgas treatment is not normally required for the expected air concentrations and mass release. If necessary, however, offgas treatment could be incorporated using MERSORB or an alternative low temperature gas phase Hg^0 treatment system. Pilot data indicate that chemical reduction and air stripping can reduce total mercury concentration in water to levels ranging from 1 to 10 ng/L for appropriate wastewater. Chemical reduction followed by air stripping is an example of the type of cost-effective option made possible by customizing chemistry and engineering to site-specific conditions. The work also highlights the general importance of understanding, and designing for, the chemical speciation of target metals. The cost of stannous chloride reduction and air stripping would be significantly lower than traditional metals treatment technologies such as ion

exchange. As a result, this approach may be a useful tool to help mitigate the release of low levels of mercury to surface water.

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Table 1. Various conditions tested in Stage 2 Kinetic Study

Run No.	Sample Matrix	Total Mercury $\mu\text{Mol/L}$ (ng/L)	Temperature degrees C.	Stannous chloride dose $\mu\text{Mol/L}$ (mg/L) note a	pH
1 (note b)	DI water	0.0005 (100)	22.8	3370 (760)	0.9
2	DI water	0.0005 (100)	0	0 (0)	5
3 (note c)	groundwater	0.00069 (138)	0	0.084 (0.019)	ambient
4	groundwater	0.00069 (138)	22.8	0	ambient
5	groundwater	0.00069 (138)	22.8	0.034 (0.0076)	ambient
6	groundwater	0.00069 (138)	22.8	0.0084 (0.0019)	ambient

Notes:

a = mass based doses are in terms of stannous chloride dihydrate.

b = these are the same conditions as EPA Method 1631. DI water = deionized water.

c = ambient pH approximately 5

Table 2. Literature values of solubility and vapor pressure used to calculate Henry's Law and theoretical sparge performance

Temperature ($^{\circ}\text{C}$)	vapor pressure (mg Hg / L air)	solubility (mg Hg / L water)	H' ("dimensionless" Henry's Law Constant)
0	0.0027	0.015	0.18
22.8	0.018	0.053	0.34

Chemistry data interpolated from Sanamesa (11). See also Clever (12).

Figure Captions:

Figure 1. Schematic diagrams of the simple process of mercury removal by means of chemical reduction followed by stripping or sparging in (a) the laboratory and (b) an example full-scale system.

Figure 2. Relation between the Sn(II)/Sn(IV) redox couple and mercury speciation. $[Cl^-] = 2 \text{ mg/L}$. The coarse dashed line shows the boundary between Sn(II) and Sn(IV) fields of dominance. The fine dashed line represents the lower stability limit of water.

Figure 3. Overall schematic of dose-response study system

Figure 4. Dose-response study results. For this figure, the average C_0 is 138 ng/L (0.00069 $\mu\text{Mol/L}$) and “effective” treatment line is drawn at approximately 10 ng/L (5E-5 $\mu\text{Mol/L}$).

Figure 5. Kinetic Study Results

Figure 1

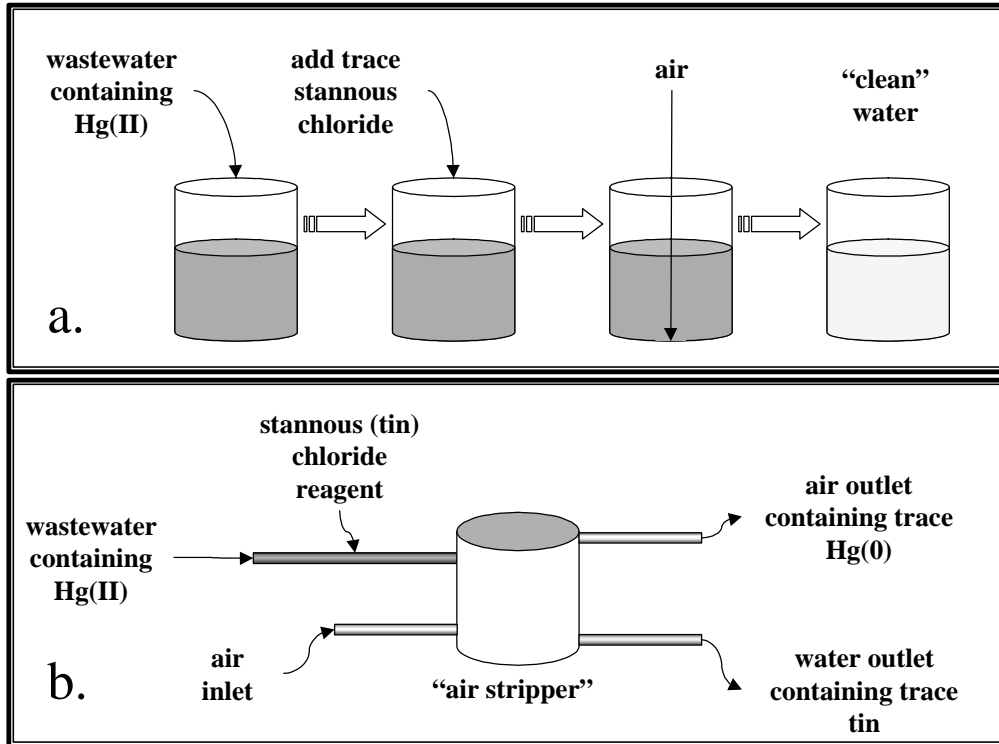


Figure 2

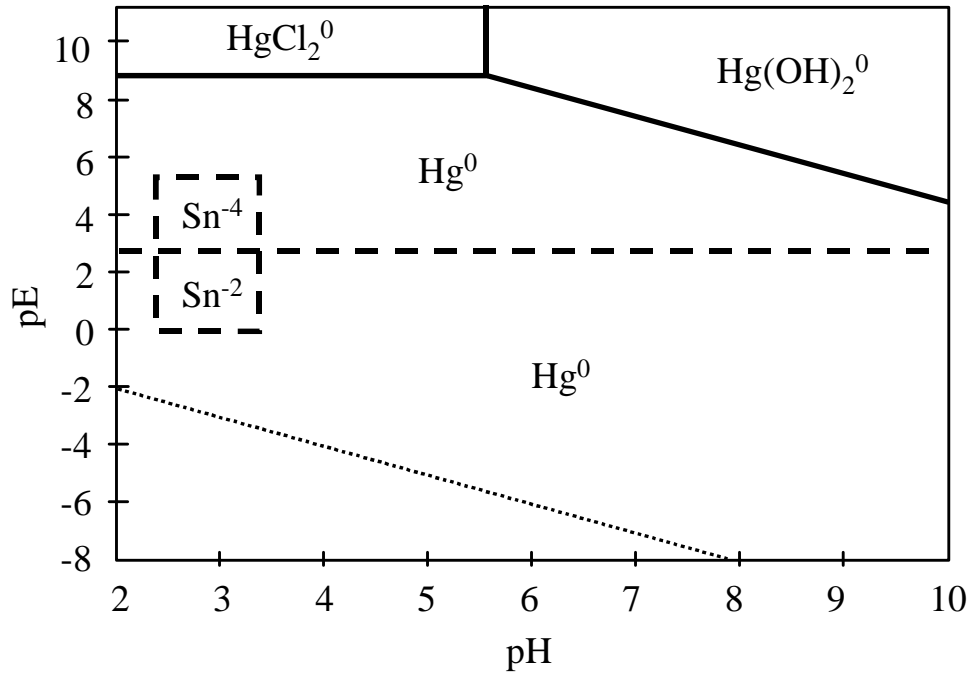


Figure 3

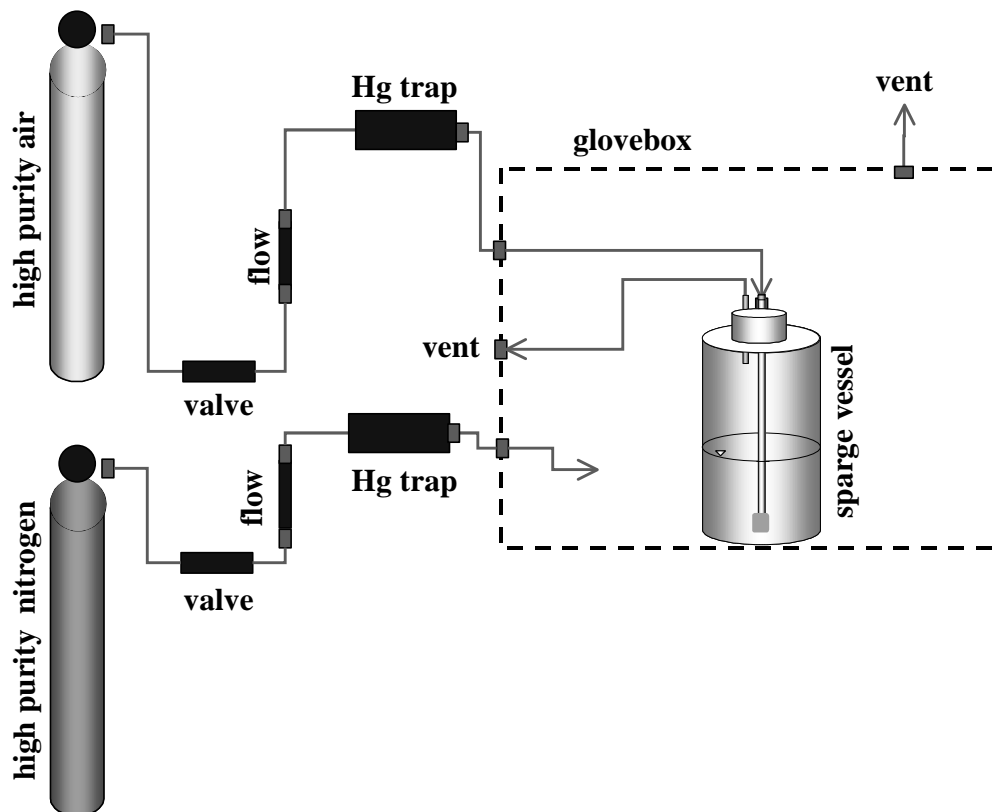


Figure 4

