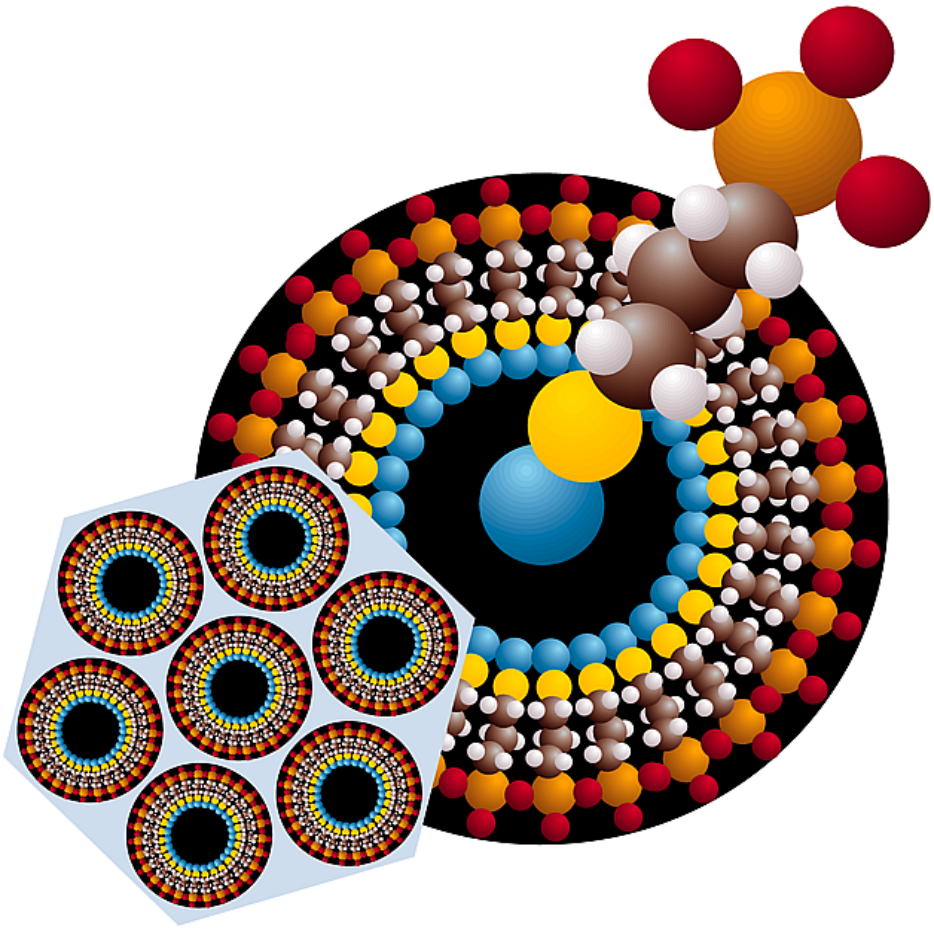
Pacific Northwest National Laboratory
Operated by Battelle for the U.S. Department of Energy

SAMMS® Technical Summary



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SAMMS Technology Summary (revised Feb. 2009)
Pacific Northwest National Laboratory

SAMMS® Technical Summary

The highly functionalized and extensive surface area of SAMMS® produce a material with fast kinetics, high material loading, and excellent selectivity.

SAMMS is an award-winning technology with broad applications in the remediation, water treatment, catalyst, sensor and controlled-release markets. A summary of the development status of SAMMS for the water treatment and metals recovery markets is provided in this document. The Pacific Northwest National Laboratory and Steward Environmental Solutions are teaming in the development and manufacture of this exciting material.

Technology Overview

SAMMS—Self-Assembled Monolayers on Mesoporous Supports—are created by attaching a monolayer of molecules to mesoporous ceramic supports (Figure 1). The larger pore size offered by the mesoporous materials (20-200 Å) enables attachment of the monolayer as well as access to the binding sites within the pores. The high surface area of the materials (~1000 m^2/g) also allows an extremely high density of binding sites. Together, these properties produce a material with fast kinetics, high material loading, and excellent selectivity. Both the monolayer and the mesoporous support can be tailored for a specific application. For example, the functional group at the free end of the monolayer can be designed to selectively bind targeted molecules while the pore size, monolayer length, and density can be adjusted to give the material specific diffusive and kinetic properties.

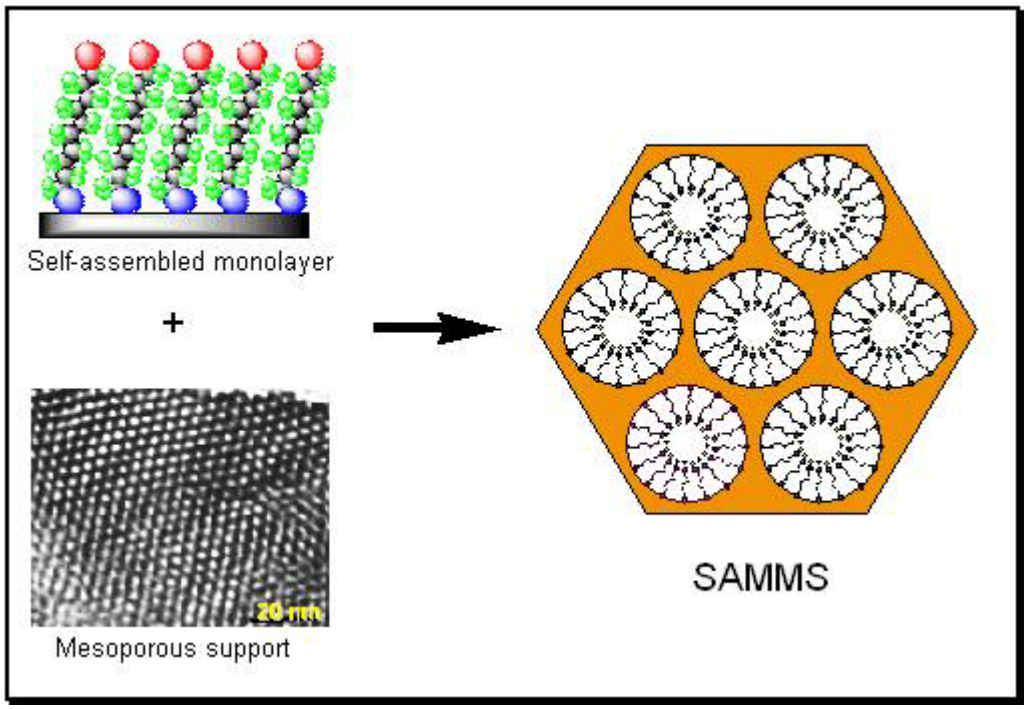

Figure 1. SAMMS is a Hybrid of Two Frontiers of Materials Science: Self-Assembly Techniques and Mesoporous Materials.

Functional groups tested to date have allowed the uptake of precious and hard and soft transition metal ions, actinides, and oxometallate anions from liquids.

Functionalized Forms

Multiple versions of SAMMS can be constructed by attaching different functional groups at the monolayer interface. Functional groups tested to date have allowed the uptake of hard and soft transition metals, precious metals, actinides, and anions from liquids. A summary of a few of the different functional forms and their selectivity is presented in Table 1.

Table 1. SAMMS® Functional Form and Affinities

SAMMS Form	High Affinity	Poor or No Affinity
Thiol- SAMMS	Hg, Ag, Au, Cu, Cd,Pb	Group 1A (H, Li, Na, K, Rb, Cs, Fr), Group 2A (Be, Mg, Ca, Sr, Ba, Ra), Zn, Al
Chelate- SAMMS (1)	Cu, Ni, Co, Zn, etc.	Group 1A and 2A cations (except H+)
Anion- SAMMS (2)	Chromate, Arsenate, etc.	SO_4^{2-}, Cl^-, NO_3^-, etc.
HOPO- SAMMS (3)	Am, Np, Pu, Th, U	Group 1A, 2A, transition metal cations

(1) Functionalized with metal-chelating ligand
(2) Chelated metal complex
(3) Hydroxypyridinone ligands for actinide removal

Thiol-SAMMS® has shown the unique ability to bind cationic, organic, metallic, and complexed forms of mercury.

Thiol-SAMMS is the most advanced in terms of development and understanding. Selectivity, isotherm, kinetic, stability, and regeneration data on thiol-SAMMS are available for multiple metals. Chelate-SAMMS, anion-SAMMS and HOPO-SAMMS forms are also fairly well developed, and representative recent results are included here. For reasons of clarity and simplicity, this document will primarily address the technical results of thiol-SAMMS.

Thiol-SAMMS®

Thiol-SAMMS was specifically developed for the removal of mercury from liquid media (both aqueous and non-aqueous). Thiol-SAMMS has shown the unique ability to bind cationic, organic, metallic, and complexed forms of mercury. Because of the high surface area, high binding site population, and tailored functionality, results of tests with mercury demonstrate high loadings (up to 635 mg Hg/g SAMMS), high affinity (distribution coefficient, or K_d, ~1 x 10^8) and rapid sorption kinetics (minutes); all made possible through the use of these molecularly-engineered materials. The efficiency of thiol-SAMMS in removing Hg from a non-aqueous system (e.g. vacuum pump oil) has also been demonstrated with mercury with excellent results. The binding affinity of thiol-SAMMS for selected metal species is summarized in Table 2, showing for each species the influent and effluent concentration, loading level, and resultant K_d. The results show that thiol-SAMMS can selectively adsorb, in addition to mercury, other soft Lewis acid cations (e.g. Ag, Cd, Cu, and Pb). In all cases, thiol-SAMMS show minimal interference from alkali and alkali earth metals, such as Na^+, Mg^{2+}, and Ca^{2+}.

Thiol-SAMMS® show minimal interference from alkali and alkali earth metals, such as Na^+, Mg^{2+}, and Ca^{2+}

Table 2. Binding Affinity of Thiol-SAMMS® for Selected Metal Species

Metal	C_i(μg/L)	C_f(μg/L)	Loading (mg/g)	K_d (mL/g)
Ag(I)	90	1	0.0089	8900
Ca(II)	2070	2070	0	0
Cd(II)	4670	32	0.4638	14467
Co(II)	2810	2670	0.0140	5
Cu(II)	2240	<5	>0.2235	>44700
Eu(III)	9010	1220	0.7790	639
Hg(II)	487	0	1.0146	1×10^8
Mg(II)	1580	1580	0	0
Pb(II)	3040	300	0.2740	913
Zn(II)	2790	2410	0.0380	16

The adsorption isotherms show that the thiol-SAMMS® have very high affinity for toxic metal ions, removing them to very low concentration (e.g. <10 ppt for Hg).

Adsorption Isotherms for Hg, Ag, Cd, Cu, and Pb

Adsorption isotherm data have been collected for Hg, Ag, Cd, Cu, and Pb using thiol-SAMMS. The most extensive data have been obtained for Hg, covering a wide range of solution concentrations (many orders of magnitude), multiple Hg species, and competition studies to evaluate the impact of complexants. Data for Ag, Cd, Cu, and Pb span a smaller range of equilibrium concentrations, and include pH and complexant effects. These isotherm data are available upon request. A summary of the maximum metal loading is provided in Table 3.

Table 3. Maximum Metal Loading for Thiol-SAMMS®

Metal	Loading (mg/g)	Loading (mmol/g)
Ag	440	4.08
Cd	97	0.86
Cu	40	0.63
Hg	635	3.17
Pb	122	0.59

Kinetics

Kinetic experiments exploring the adsorption of mercury in 10- and 500-ppm mercury solutions demonstrate the rapid binding kinetics of thiol-SAMMS. As illustrated in Figure 2, SAMMS was able to rapidly reduce the mercury concentration from 10-ppm to 3.1 ppb within 5 minutes. For the 500 ppb case (not shown), the Hg concentration was reduced to 0.5 ppb within 5 minutes. Also shown in this figure is the binding kinetics of a commercial ion exchange resin. Thiol-SAMMS is roughly 500 times faster at sorbing Hg than is the polymer-based system. This rate difference is directly attributable to the rigid, open pore structure of SAMMS leaving all of the binding sites available at all times to bind metal ions.

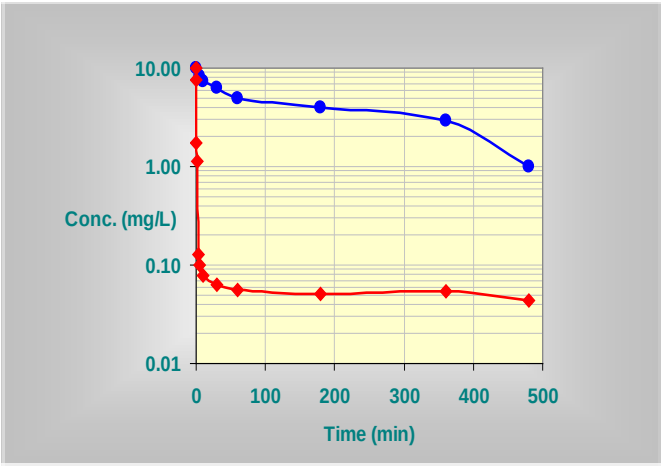

Figure 2. SAMMS Mercury-Binding Kinetics in a 10,000-ppb Mercury Solution (the red line is the SAMMS kinetics profile; the blue line is for the comparable polymer-based system (GT-73).

Effect of pH and Chelating Ligands

Mercury complexes of ammonia, EDTA, cyanide, and fulvate had no measurable negative impact on mercury binding.

Previous adsorption tests with thiol-SAMMS showed that Hg adsorption slightly increased with increasing pH of the contacting solution. The enhanced adsorption suggests that increasing the ionization of the thiol groups by increasing the pH would increase the metal adsorption.
To explore this phenomenon further, a set of experiments were conducted to determine the extent of pH dependent adsorption of Ag, Cd, Cu, and Pb using thiol-SAMMS. For these tests, the pH of the equilibrating solution (0.1 M $NaNO_3$) was varied from ~3.1 to ~8.5. As a result of metal precipitation, adsorption experiments for Cu and Pb were not feasible above pH values of ~5.2 and ~6.7 respectively. The results show that Ag was completely adsorbed by thiol-SAMMS with very high affinity (K_d) at all pH values. In contrast, the adsorption densities of Cd, Cu(II), and Pb were low at the lowest pH (~3.1) and, as predicted, increased with increasing pH. Enhanced adsorption of these metals at higher pH was also reflected in increases of one to two orders of magnitude in the K_d values.

Testing with model organic complexing ligands (ammonia, ethylenediaminetetraacetic acid (EDTA), cyanide, and fulvate) was conducted to evaluate adsorption characteristics of thiol-SAMMS with solutions containing 1:1 mercury-ligand complexes. Mercury complexes of these ligands had no negative impact on the ability of thiol-SAMMS to bind mercury. Organic ligands similar to these model ligands are expected to have little effect on SAMMS adsorption. Similar results were observed with inorganic mercury complexes (e.g. chloride, iodide).

For the other metal ions, experiments were conducted with EDTA because of its strong binding of metals and widespread industrial use. The molar concentration ratio of ligand to metal in these experiments ranged from 0.65 to 2.3. Data from the EDTA experiments show that at fixed pH, the extent of adsorption of these metals is influenced by both the degree of complexation as well as the relative stability of respective EDTA complexes. Because little complexation of Ag was expected, there were no differences in Ag adsorption with or without EDTA in equilibrating solutions. EDTA complexation did not seem to diminish the adsorption of Cd by thiol-SAMMS.

Cu adsorption, however, was significantly reduced in the presence of EDTA. The formation of stable Pb-EDTA complexes almost completely suppressed the adsorption of Pb.

Mercury-loaded SAMMS® have remained stable with exposure to temperatures of 150°C in air for up to 50 hours and up to 70°C in water for 24 hours.

Performance multiple metals

The effectiveness of thiol-SAMMS in removing multiple metals from a batch solution was recently demonstrated. A solution containing Ag, Cd, Cu, and Pb, each at 0.05 mmol/L (~5, ~6, ~3, and ~10 ppm, respectively), was prepared in a matrix of 0.1 M $NaNO_3$. The solution was equilibrated with thiol-SAMMS at various solution-to-solid ratios ranging from 250 to 5000. Metal adsorption was calculated using measured equilibrium concentrations. The selectivity of metals removed is as follows: Ag ≅ Cu > Cd > Pb.

- Copper was removed from 3 ppm to <8 ppb ($K_d = 1.5 \times 10^4$)
- Silver from 5 ppm to <2 ppb ($K_d = 2.5 \times 10^4$)
- Cadmium from 6 ppm to 0.2 ppm ($K_d = 8.1 \times 10^3$)
- Lead from 10 ppm to < 1 ppm ($K_d = 4.7 \times 10^3$)

These results show that SAMMS can effectively remove multiple metals simultaneously from a complex matrix containing multiple metals.

Precious Metals Recovery

Preliminary data has shown that thiol-SAMMS is also effective at recovering precious metals, like Au, Ag, Pd and Pt from aqueous media. Thiol-SAMMS has a much higher metal binding affinity and capacity than does the more commonly used activated carbon. Thiol-SAMMS made using supercritical fluid (SCF) methodology has been shown to have higher silane population density, lower defect density and excellent stability towards alkaline conditions, so it may be possible to apply these sorbents towards precious metal recovery in the mining industry.

Stability

SAMMS has also been subjected to chemical and physical stability testing. Mercury-loaded SAMMS have remained stable with exposure to temperatures of 150°C in air for up to 50 hours (no Hg released), and up to 70°C in water for 24 hours (Hg released equaled ~0.4% of the Hg sorbed). SAMMS loaded with Hg have also been subjected to the Environmental Protection Agency's Toxic Characteristic Leaching Procedure Tests (TCLP). Releases for these RCRA metals were two orders of magnitude below EPA's standards. Thus, the loaded thiol-SAMMS materials may be disposed of as a conventional waste. We expect similar behavior for other metal laden thiol-SAMMS (e.g. Ag, Cu). Stability tests for other flavors of SAMMS are planned for the near future. Finally, SAMMS is resistant to biodegradation, as the pore size of the mesoporous supports is far too small to allow access to the bacteria that could metabolize and release bound metals to the environment.

Regeneration

The conditions under which SAMMS may be regenerated depends on the nature of the metal-ligand bond formed between the SAMMS and the target analyte. We have demonstrated that Hg

laden thiol-SAMMS can be regenerated using a simple HCl rinse. A similar acid strip has been shown to be effective for the carboxylic acid forms of SAMMS used to bind the lanthanides (e.g. Gly-UR SAMMS), and these materials have been shown to be able to go through over a dozen regen cycles with no loss of sorbent activity. Regeneration of the cesium sorbent (i.e. ferrocyanide SAMMS) can be accomplished by oxidizing the ferrocyanide center to the ferricyanide oxidation state and washing the Cs of the sorbent, then reducing the Fe(III) center back to Fe(II). Regeneration of other SAMMS forms should also be possible under conditions appropriate for that particular metal-ligand bonding arrangement.

Chelate-SAMMS®

Research and development is continuing on the preparation and testing of metal-chelating SAMMS. Chelating functional groups (such as EDA, ethylenediamine triacetic acid, 1,10-phenanthroline, HOPO, etc.) are attached to the surface of the mesoporous materials using similar chemistry as is used for preparation of the thiol-SAMMS. The chelating group is tailored for the targeted metal; for example, EDA for Cu and HOPO for the actinides. Results have shown that selective removal of metal ions can be accomplished by various chelate-SAMMS forms, and research is continuing to optimize the synthesis and metal loadings.

Anion-SAMMS®

Nearly complete removal of arsenate and chromate is achieved in the presence of interfering anions for solutions containing up to 100 ppm toxic metal anions under a variety of conditions.

We have synthesized and demonstrated the use of cationic metal complexes immobilized on mesoporous silica as a novel anion-binding materials for toxic anions such as chromate, arsenate, and pertechnetate. This approach allows the construction of binding sites that satisfy the stereoelectronic requirements of tetrahedral anions, as well as allowing a direct metal-anion interaction. These SAMMS can remove oxometallate anions like chromate and arsenate to low levels, even in the presence of large excesses of sulfate ion. Nearly complete removal of arsenate and chromate has been achieved in the presence of interfering anions for solutions containing up to 100 ppm toxic metal anions under a variety of conditions. These anion-SAMMS materials remain effective at even higher concentration solutions (in excess of 1000-ppm anions). Anion loading of more than 130 mg/g (1.12 mmol/g) of SAMMS and distribution coefficients of more than 100,000 have been observed for chromate anion. These properties are comparable to the performance of thiol-SAMMS. Our anion removal tests were performed in water containing 1-, 10-, and 100-ppm arsenate and chromate with a water-to-sorbent (SAMMS) ratio of 100. In each of these tests, virtually all of the chromate was removed in a single treatment. The presence of competing sulfate anions (at 150 ppm) had little effect on chromate binding. At the same solution-to-sorbent ratio (100 mL/g), chromate concentrations higher than 1000 ppm began to saturate the binding sites. For a much higher solution-to-silica ratio (500 mL/g), 100% removal of the chromate is observed for chromate concentrations up to 100 ppm.

Similar results were also obtained for arsenate removal. The maximum loading capacity is 140 mg/g or 1.00 mmol/g. Under the same conditions, the residual concentrations of arsenate are all slightly higher than chromate at low anion concentrations. This suggests the Cu-EDA binding chemistry has somewhat higher affinity for chromate than arsenate under these conditions. The test results can be summarized as follows:

- The anion-SAMMS materials are efficient oxometallate anion binding materials. Virtually complete oxometallate anion removal can be achieved over a wide concentration range.
- Oxoanions (chromate and arsenate) are preferentially bound over sulfates.
- Low concentrations of chromate and arsenate can be removed from high concentrations of sulfates
- The binding chemistry is more sensitive for chromate than arsenate at low concentrations.

Engineered Forms

SAMMS are to be available as a powder (particle size can be tailored to fit the application, from roughly 10 microns, to well over 100 microns) from Steward Environmental Solutions. What's more, this basic powder form can be modified into a variety of engineered forms (e.g. extrudates, monoliths, etc.) to be more readily integrated into existing ion exchange systems/infrastructure. SAMMS have been impregnated into proprietary filter membranes and tested in a flowing system for mercury removal. The results demonstrated that membrane-supported SAMMS performed similarly to the unsupported powder. We are also working with Perry Equipment Corporation (PECO) to integrate SAMMS (and related nanoporous sorbent materials) into filter cartridge canisters that will be useful in a wide variety of industrial applications.

Field Demonstrations

There have been a number of field demonstrations of SAMMS to address Department of Energy as well as commercial waste treatment problems, either currently underway, or that have recently been completed. The first example of using thiol-SAMMS to treat actual chemical waste was performed at PNNL on 10L of laboratory waste that contained 146 ppm of Hg. This was treated with a single batch contact of 86g of thiol-SAMMS, and the final Hg concentration was approximately 0.04 ppm. Similarly, a second waste stream comprised of 200L of EVS scrubber waste, containing 4.64 ppm Hg, was treated with thiol-SAMMS to achieve a final Hg concentration of 0.05 ppm. Both of these treatments resulted in significant savings in terms of disposal costs (~10-fold).

A third example was performed at Oak Ridge National Laboratory. This case is noteworthy because the medium was non-aqueous, contaminated vacuum pump oil, which is not only very hydrophobic, but also very viscous. This wastestream contained Hg concentrations as high as 50 ppm, and single treatment with thiol-SAMMS was easily able to reduce the Hg concentration to well below the target threshold of 0.2 ppm. Thiol-SAMMS® is the only technology that is proven to be effective at removing Hg from such hydrophobic, viscous media.

Figure 3. Photo of the apparatus used to test thiol-SAMMS to remove Hg from contaminated vacuum pump oil (treatment was performed in the black 55-gal barrel with the impeller, then the vacuum pump oil was removed, filtered and pumped into the silver receiving barrel).

A field demonstration/deployment has also been carried out at Idaho National Laboratory. In this case, the wastestream was composed of a complex aqueous slurry of heavy metals, radionuclides (e.g. Cs-137, Co-60, Eu-152), suspended solids, halogenated solvents (TCE, TCA, PCE, etc.), and other components. The Hg concentration was measured to be 1.57%, and the speciation was unknown (thought to be mixed?). There was a viable strategy in place for solvent removal, and the plan was to solidify the waste to immobilize the radionuclides (using Waterworks SP-400). Unfortunately, this approach still left the Hg in a leachable state, and the final waste would not pass TCLP. Preliminary testing showed that thiol-SAMMS effectively immobilized the Hg in place and that the resultant wasteform could pass TCLP by a significant margin. The entire contents of the waste tank were subsequently treated successfully using thiol-SAMMS.

There have been a number of examples in the commercial sector as well. One customer had a well that contained approximately 25 ppb Hg, and had been trying unsuccessfully to treat it with carbon type adsorbents. Their Hg reduction target was ≤ 12 ppt output. This well-water contained over 20 inorganic species. Steward Environmental Solutions conducted bench testing (batch) and demonstrated that thiol-SAMMS was able to reduce the Hg concentration down to ~14 ppt. Steward Environmental Solutions conducted additional field testing using their 10 gpm continuous flow SAMMS filtration system. These tests ran for 1,300 hours and processed over 450,000 gallons of well water. Thiol-SAMMS reduced the Hg concentration from input ~25 ppb (input) down to below detection limits (EPA 245.7) at output (~ 5 ppt), well below 12 ppt mercury reduction goal. The Hg laden SAMMS were tested and passed TCLP.

Another commercial customer had well water that was very dark brown in color (humic acid) and contained high concentrations of total dissolved solids and organics. Activated carbon had failed repeated tests due to fouling. The well water contained more than twenty known species, including sulfide, iron and chlorobenzene, in addition to 2 ppb Hg. Preliminary batch testing with thiol-SAMMS demonstrated that it was possible to reduce the Hg concentration sufficiently to meet an initial target of 90% reduction. A field test was conducted using the Steward Environmental Solutions 10 gpm continuous flow SAMMS filtration system, along with ozone

Figure 4. Steward Environmental Solutions 10 gpm continuous flow SAMMS® filtration system.

pretreatment to reduce organics. Thiol-SAMMS successfully reduced the Hg concentration from ~2,000 ppt (input) down to ~ 10 ppt (output), well below the 90% mercury reduction goal. Over 40,000 gallons of well water were processed in this continuous flow test. Once again, the Hg laden SAMMS from the field test were subjected to TCLP and passed.

Another commercial customer had a need to reduce the Hg concentration down to 1.3 ppt or lower. Their existing water treatment system was not able to achieve this ultra low concentration. Thiol-SAMMS was tested as an alternative. Steward Environmental Solutions performed a simple batch test, and was successful in reducing the Hg concentration from 4.5 ppt down to 0.7 ppt (well below the targeted 1.3 ppt) in just 6 minutes.

Other demonstrations are currently underway.

Competitive Assessment: Mercury Removal

The following technologies represent the primary competition in the area of dilute mercury recovery, removal, and/or separation.

- Mercu-RE (gases), sorbent-based (water), and sulfur-based (radioactively contaminated mercury) (ADA Technologies, Engelwood, CO)
- Sulfur-impregnated carbon (Kanazawa University, Kanazawa, Japan)
- Activated carbon absorption (Norit Americas Inc., Prior, OK)
- Duolite™ GT-73 ion exchange organic resin (Rohm and Haas Company, Philadelphia, PA)
- Colloid precipitate flotation (Mansoura University, Damietta, Egypt)
- Microemulsion liquid membranes (Rutgers University, Newark, NJ)
- Amalgamation (a generic process).

Table 5 compares these technologies' performance to SAMMS. At the mercury concentrations of interest, SAMMS loading is considerably higher than that of the next best available technology. This higher loading capacity, along with the faster kinetics, superior selectivity, and reduced

disposal costs (since less sorbent is needed to remove a given amount of heavy metal contaminant) translates into real life-cycle cost savings.

Table 5. Competitive Advantages of SAMMS® for Mercury Removal

	Carbon	GT-73	Colloid/Precip.	Amalgam	Liquid membranes	SAMMS	Advantages
Organic liquids	No	No	No	No	No	Yes	Only technology suitable for organics
Aqueous liquids	Yes	Yes	Yes	Yes	Yes	Yes	Achieves lower concentrations to meet regulatory requirements
Binding kinetics	Medium	Medium	Slow	Slow	Slow	Fast	Reduced reaction time, faster throughput
Metal loading, (g/kg at 0.2 ppb Hg)	0.025	6	N/A	No	N/A	80	Significantly higher loading capacity, needs no material regeneration
Selectivity	Low	High	Medium	N/A	Medium	Very High	Most selective, needs less material
Chemicals needed	No	No	Yes	N/A	Yes	N	No need for additional tanks, process equipment, etc., no secondary waste generated, thus reduced treatment cost during application
Stable final waste form	No	No	No	N/A	No	Yes	Only technology that requires no additional treatment before disposal
Final waste volume	Large	Medium	Medium	N/A	Medium	Very Low	Greatly reduced material and disposal costs
Overall cost	High	Medium	High	High	High	Low	Significantly cheaper than next best available technologies
Decontamination factor	Low	Medium	Medium	N/A	Medium	High	Only technology that meets stringent r regulatory requirements

SAMMS®-Lixiviant Systems

A novel proprietary lixiviant system that is compatible with SAMMS has also been developed to allow the cost effective treatment of mercury in soils and sludges. The lixiviant has also shown to be effective for completely removing nickel and uranium and between 40-60% removal of cadmium, silver, and molybdenum in well characterized soils.

Availability

Steward Environmental Solutions, in collaboration with Battelle, have established commercial production capacity for the manufacture of SAMMS, and are currently producing materials. Quantities of SAMMS are available for testing by potential end-users and resellers. Our scientists and engineers will work with you to determine the best form of SAMMS and the optimal delivery vehicle.

Current R&D Activities

We are extending our database on SAMMS performance. Testing is proceeding on:

- New chelate-SAMMS for transition metals in aqueous systems.
- Stability under extreme process conditions and as a final waste form.
- Regeneration methods and the regeneration effectiveness.
- Integration into novel engineered forms.
- Precious metal recovery.
- Actinide removal from a range of radioactive environments.
- Fundamental science issues related to the synthesis and application of SAMMS.

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