STEP Mercury Removal Technology Demonstration Project

Final Report

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Disclaimer

This Report is a product of the Strategic Envirotechnology Partnership (STEP). All opinions, suggestions, recommendations, and conclusions expressed in this Report are those of the authors, with the exception of the Vendor reports contained in Appendices G through I.

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This Report is intended to assist the users and potential users of mercury removal technologies to make informed decisions about the process and environmental performance of three specific mercury removal technologies. It is not intended to communicate the full market scope or competitive position of the three participating Vendors and their technologies, but rather to illustrate the effectiveness of the technologies in treating actual mercury-containing wastewater streams. This Report is not a comprehensive evaluation of all available pretreatment technologies that could be applied to mercury-containing wastewater streams.

It must be emphasized that the pilot tests described in this Report were only intended to evaluate technical performance and were performed on samples of three particular wastestreams. Results of similar testing on other wastestream samples may differ. Furthermore, because of the limited nature of pilot-scale performance testing, any descriptive information and cost estimates provided by the Vendors relative to full-scale pretreatment systems should be considered as preliminary only.

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Executive Summary

The Strategic Envirotechnology Partnership (STEP) Mercury Removal Technology Demonstration Project verified the commercial viability of a number of treatment technologies that can continuously achieve mercury removal rates to 1 µg/l (1 part per billion) and below from hospital clinical and research laboratory wastestreams. The study also developed a standardized sampling and mercury source identification protocol to help hospitals systematically sample and identify point-of-use mercury contamination from clinical and research laboratories. The STEP project brought diverse expertise together (university, technical assistance and regulatory) bringing impartiality and credibility to the study. The study serves to inform future mercury technology development and accelerate technology acceptance and deployment.

The Demonstration Project builds on bench scale feasibility testing (using clinical laboratory wastewater) completed by the Massachusetts Water Resources Authority's (MWRA) Technology Identification Subgroup in 1997, part of the MWRA's Phase II Mercury Workgroup efforts to identify methods and techniques that would help hospitals achieve compliance with MWRA's enforcement action threshold value of 1 µg/l for mercury. The MWRA Mercury Workgroup was a public/private voluntary stakeholder effort aimed at reducing mercury discharges to the MWRA system. Substantial reduction in anthropogenic mercury release to the environment is necessary to protect public health and improve environmental quality. The virtual elimination of mercury from products remains a priority for the STEP Partners (the Executive Environmental Office of Environmental Affairs and the University of Massachusetts) and the MWRA.

TECHNOLOGY DEMONSTRATION

Mercury analyses for the Demonstration Project were performed in the trace metal analytical facilities of the University of Massachusetts - Boston, based on a modification of EPA Method 245.1. The technique is capable of achieving an analytical detection limit of 0.002 µg/l. All sampling and analyses were conducted using trace-metal-clean techniques to prevent contamination and achieve analytical resolution at the ng/l (nanograms/liter) concentration range. Limits of detection obtained throughout the course of this project were generally ≤ 0.01 µg/l.

Aero-Terra-Aqua Technologies Corporation (ATA), DuBois Chemicals/Prosys Corporation, and ICET, Inc. agreed to pilot-test their promising technologies for the removal of mercury from wastewater. ATA and ICET, Inc. were identified as candidate technologies based on their promising results in the bench scale feasibility testing with clinical laboratory wastewater conducted by the MWRA in 1997. DuBois Chemicals provided STEP with bench testing data using research laboratory wastewater from a Boston area medical facility that showed their ability to remove mercury to the 1 µg/l level. They partnered with Prosys Corporation, a membrane filtration vendor, to provide a complete pretreatment system.

The three technologies were tested at a total of three host facilities. Two were hospitals: Newton-Wellesley Hospital represented a high mercury concentration/low flow clinical

laboratory site; and Brigham & Women's Hospital, a low mercury concentration/high flow clinical laboratory site. The ICET technology was also tested at the USDA Human Nutrition Research Center on Aging at Tufts University, a high mercury concentration/low flow research laboratory site.

Prior to the initiation of pilot testing, the temporal variability (over a 24-hour period) in the concentration of mercury in the effluents of the three sites was assessed. Preliminary bench tests were also conducted by the vendors to determine final pilot system design (including an assessment of preconditioning requirements) and initial operating conditions for the pilot testing. The vendor bench testing was performed using wastewater samples obtained from the three host facilities. All the vendors were successful in removing mercury to less than 1 µg/l in their bench testing runs.

High-resolution sampling of the wastestreams to be treated at each site indicated that the bulk of the mercury discharged at the three sites coincided with periods of active use of the laboratories discharging to the wastestream and thus could not be attributed to desorption of mercury from the host site's waste collection system. Large, short-term temporal excursions in mercury concentration were also observed. Especially important in these findings is that the contemporary discharge of mercury from all three sites probably involves active use of reagents containing high concentrations of mercury that are released to the waste collection system of the facilities.

All three technologies participating in the STEP Demonstration Project were tested as end-of-pipe technologies. ATA's technology is based on several chemically enhanced sorbents marketed as AQUA-FIX™ that are designed to remove dissolved and ionic forms of metals, and was tested at Newton-Wellesley Hospital. The DuBois/Prosys technology treats a wastewater stream with chemicals to create a suspension of contaminant-containing solids that are then removed using a membrane filter, and was tested at Brigham & Women's Hospital. ICET's system was tested at Brigham & Women's Hospital and the USDA Human Nutrition Research Center on Aging at Tufts University. Their technology utilizes surface modified activated carbon sorbents capable of removing heavy metals from wastewater. All three systems were able to reduce mercury in the discharges treated to concentrations below, and in some cases well below, 1 µg/l. The DuBois/Prosys and ICET systems produced consistent results while the ATA system achieved only intermittent success. Mercury concentrations in excess of 1 µg/l in wastewaters treated using each system were occasionally observed. These exceedances generally coincided with incidents of extreme excursion of the mercury concentration in the influent to the pretreatment system. Once optimal operating conditions had been achieved, overall treatment performance to produce concentrations below 1 µg/l was relatively stable.

ATA's pilot unit consisted of six cartridge housings connected in series to contain the prefilters (for particulate removal) and AQUA-FIX™ media cartridges used in the tests. Some of the runs performed included a preconditioning step prior to the cartridge train that involved oxidizing the wastewater with bleach. Preconditioning of the wastewater was required to achieve mercury levels of 1 µg/l or less in the effluent.

In the first round of the DuBois/Prosys testing, effluent mercury levels below 2 µg/l were typically achieved, even during spikes in the influent mercury concentration. Levels below 1 µg/l were seen, but were not consistently attained. A second round of testing was conducted after several modifications were made to the system in attempts at achieving consistent removal of mercury below 1 µg/l. At the end of the test period (after replacement of a ruptured membrane and the addition of a coagulant to destabilize and aggregate colloidal forms of mercury), the system consistently removed mercury to <1 µg/l in the effluent, even during a period of very high influent mercury levels (up to 109 µg/l).

The **ICET** pilot unit consisted of preoxidizing the influent wastewater using bleach followed by passage through two prefilters. Downstream of the prefilters, the wastewater passed through a bed of granular activated carbon and then three beds of ICET's media prior to being discharged. The pilot testing demonstrated that the ICET system was capable of consistently removing mercury to below 1 µg/l, even during several periods of high mercury levels in the influent, at both facilities at which it was tested (Brigham & Women's Hospital and the USDA Human Nutrition Research Center on Aging at Tufts University).

A significant lesson learned from this project and a critical factor for consideration by all mercury technology developers, technology advocates and end users is an understanding that successful end-of-pipe performance depends heavily on preconditioning the wastewater before treatment. The study found that a significant portion of the mercury in the wastestreams was in a colloidal and organically complexed form, which is not readily available for capture by a sorbent-based system (e.g., the ATA and ICET technologies). An improved understanding of the chemical form and reactivity of the mercury in these wastestreams will greatly enhance future technology development successes. Thus, while removal of mercury from wastestreams to levels below 1 µg/l is technically feasible, success can only be achieved through a comprehensive assessment of wastestream characteristics and preconditioning prior to the mercury removal step. However, considering the variable nature of the wastewater treated in this project, reaching acceptable levels of mercury in the treated effluent may not be as difficult as previously thought.

PRESCREENING FOR APPLICATION OF POINT-OF-USE TECHNOLOGIES

Point-of-use prescreening was used to help identify activities at each site where high levels of mercury were entering the waste collection system of the host site and thus locations where source reduction and/or application of point-of-use technologies might be implemented. A protocol for conducting the prescreening analyses was developed. Briefly, the protocol requires the host site to first identify the source of any large volume flows entering the wastewater conveyance system which, upon testing, hopefully can be eliminated as a significant source of mercury flux in the system. If not, end-of-pipe removal technology would most likely have to be applied. If the results indicate smaller volume flows as the source of mercury, additional screening at individual laboratory sinks is justified to identify major sources. The results of the point-of-use testing largely confirmed the belief that the most probable sources of mercury are those containing high concentrations of mercury discharged in low volumes to the waste collection system.

Point-of-use testing was conducted at Newton-Wellesley and Tufts. It was not conducted at Brigham and Women's due to an inability to identify the large volume flow source(s) at that facility. While no actual pilot testing of technologies in point-of-use applications was conducted due to limitations in resources, the conduct of the prescreening analysis led to several conclusions. First, the results of screening analyses can be used to achieve source reduction by avoiding the use of reagents containing high concentrations of mercury. Where such action cannot be accomplished for reasons such as the lack of availability of suitable substitute reagents and/or excessive cost, screening analyses can identify wastestreams that can be treated using appropriate point-of-use capture technologies.

Second, none of the host sites had accurate quantitative assessments of the origin of their wastewater flows. Reliable wastewater flow and source data is critical to the success of point-of-use prescreening analyses. Flux determinations (requiring the monitoring of flow) rather than just concentrations in wastewater would be of greater use in identifying the magnitude and temporal variation in the discharge of mercury from facilities.

Third, at both sites investigated using the screening techniques, the major sources of mercury were confined to just two point-of-use locations. The vast majority of locations sampled at both Newton-Wellesley and Tufts contained low concentrations of mercury.

Fourth, the identification of these high point-of-use concentrations is consistent with the conclusion that most of the mercury being discharged at the host sites during this investigation was due to the active release of mercury from contemporary laboratory activities, and not from desorption from the wastewater collection systems at these sites.

Finally, the screening protocol used, when coupled with accurate high quality analysis capable of resolving differences at < 1 μg/l concentrations, can be used to achieve high resolution sampling. Even slightly elevated concentrations of mercury (>0.05 μg/l) in discharges can be readily detected and used to guide appropriate action.

I. Background

Mercury is a metal that poses significant environmental and health risks. It is present in the environment in three major forms, elemental, ionic, and organic. The latter form, which is typified by methyl mercury, is the most dangerous since it bioaccumulates, i.e., it builds up in the muscle tissue of living organisms. Reproductive failures and other health problems may result from this accumulation in both wildlife and humans. Short-term high exposures can cause poisoning, bronchitis, and pneumonitis. Repeated exposure to relatively low levels can lead to effects such as muscle tremors, nerve damage, or personality changes. Because of this potential for creating severe environmental and human health problems, there is an extensive effort underway, both in Massachusetts and nationally, to reduce the amount of mercury that is discharged to the environment.

The Massachusetts Water Resources Authority (MWRA) is a public agency charged with supplying water and sewerage services to the Boston metropolitan area. The MWRA's Sewer Use Discharge Regulations prohibit the discharge of mercury to the sanitary sewer system. The agency uses an enforcement action threshold value of 1.0 μg/l (one part per billion) for enforcing the mercury limit.

The MWRA actively encourages efforts that eliminate or minimize the amount of mercury discharged to the sewer system. In 1994, the MWRA supported the formation of the Mercury Workgroup as a joint public/private stakeholder effort to reduce the impacts resulting from clinical and research wastewater discharged to the sewer. The Workgroup, through its associated Subgroups, was asked to identify methods and techniques that would help hospitals achieve compliance with MWRA's end-of-pipe mercury standards. As a result, a source reduction program was developed that helped some hospitals eliminate mercury from wastewater by a factor of ten. Efforts to identify new products and additional source reduction strategies continue and are encouraged. Although source reduction achieved a significant decrease in mercury, a gap exists between what can now be achieved using source reduction methods and the MWRA's end-of-pipe standards. To fill this gap, the Technology Identification Subgroup, as part of Phase II Mercury Workgroup efforts, solicited, selected, tested and evaluated mercury treatment and control technologies at the bench scale level. The results of this effort are described in the Technology Identification Subgroup Report [(1)].

The STrategic Envirotechnology Partnership (STEP) was launched in 1994. STEP is a cooperative effort between the University of Massachusetts System (UMass) and the Executive Office of Environmental Affairs (EOEA). STEP's mission is to promote the growth of new environmental and energy efficient technologies in Massachusetts. STEP services include: regulatory and business planning assistance, technology demonstration and verification, and marketing assistance.

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1 MWRA/MASCO Mercury Work Group Technology Identification Subgroup Report, MWRA, December 1997. (MASCO is the Medical Academic &Scientific Community Organization, Inc.)

II. Introduction/Objectives

The STEP Mercury Removal Technology Demonstration Project builds on the bench scale feasibility testing (using clinical laboratory wastewater) completed by MWRA's Technology Identification Subgroup. STEP organized, implemented and supported the demonstration and evaluation of three mercury removal technologies at three host facilities. This effort was also designed to help the participating vendors gain technology acceptance and stimulate commercialization efforts through the development and review of third party generated cost and performance data.

The primary objective of the Demonstration Project was to perform field testing to demonstrate the consistent performance of promising mercury pretreatment technologies in the removal of mercury to 1 µg/l (MWRA's enforcement action limit) or lower in clinical and research laboratory wastestreams. As such, it was ***not*** the goal of the project to bring any of the participating facilities into compliance with MWRA requirements.

The project was originally envisioned to only assess technology performance in an end-of-pipe application, i.e., treatment prior to discharge to the sewer. During the planning stages of the project, however, it was decided to expand the project to allow the technology vendors the choice of end-of-pipe and/or point-of-use application of their systems. Point-of-use testing would involve testing systems on specific discharges close to the point where the discharge was generated, e.g., the discharge from a group of clinical analyzers. The goal of this testing would be to demonstrate consistent performance in the removal of mercury to 1 µg/l or lower for the specific discharges to be tested. It was recognized that the removal of mercury to $\leq$ 1 µg/l in this type of testing would not necessarily mean that the end-of-pipe mercury concentration would be the same because the potential for mercury release from a facility's plumbing would not be considered. Notwithstanding this drawback, point-of-use testing was considered an important component of the project because it promotes source reduction by requiring facilities to more closely examine the specific sources of mercury in their discharges and, where such discharges are difficult to avoid, allows the possibility of removing mercury from high concentration wastes using more cost-effective mercury removal technologies then currently employed.

This report summarizes the results of STEP's Mercury Removal Technology Demonstration Project. It includes descriptions of the types of wastewater tested and the species of mercury in the wastewater, and outlines the implementation of the project - the selection of technology vendors and host facilities, the development of the scope of work for end-of-pipe field tests, the results of those tests, and the results of point-of-use prescreening that was conducted.

STEP is a program that is designed to support technology vendors in the marketplace. One form of support the program provides is the third party assessment of the performance of environmental technologies. This report describes such a performance assessment for three mercury removal technologies. It is believed that the results of this project, coupled with MWRA's Mercury Management Guidebook and Pretreatment Guidance Manual, will provide facilities discharging mercury with useful information with which to make better technology decisions and implement more cost-effective mercury reduction and control methods.

However, it should be noted that STEP did not perform an exhaustive search of mercury removal technologies in conducting this demonstration project. Consequently, the results of this project do not represent an assessment of the performance of all technologies that could potentially achieve mercury removal to $\leq$ 1 µg/l. It also must be understood that the pilot tests were performed on samples of particular wastestreams; it cannot be assumed that similar results will be achieved on different wastestreams. The participating vendors should be contacted directly for more information about their technologies and about treatability and pilot testing requirements. All vendor cost estimates included in this report should be considered as preliminary only.

Recently, the MWRA instituted an interim enforcement policy that included issuing enforcement orders to facilities in the MWRA service area. The enforcement program was separate from the MWRA's participation in this pilot project, and participating host facilities were not exempted from meeting any existing or newly issued compliance and enforcement requirements. Because of the ongoing enforcement actions, the host facilities' participation was acknowledged within the structure of the enforcement orders. The pilot test units were tested on side streams, prior to the MWRA compliance point. The wastewater discharges from the pilot test units were exempted from all compliance and enforcement requirements.

III. Demonstration Project Approach

The STEP project team used the following process in implementing the Demonstration Project –

- Selected vendors of mercury removal technologies to be pilot tested.
- Selected host sites for the testing.
- Conducted site visits for the vendors participating in the testing.
- Developed the scope of work for field testing of end-of-pipe mercury removal systems.
- Vendors performed preliminary bench testing using wastewater from all sites.
- STEP provided technical advice and provided analysis of mercury concentrations using trace-metal-clean techniques.
- Vendors conducted pilot testing of end-of-pipe systems, and submitted test reports.
- STEP conducted point-of-use prescreening analyses.

Each of these steps is described in detail in the subsequent sections of this report.

IV. Selection Of Mercury Removal Technology Vendors

Established Selection Criteria

STEP determined that only those vendors meeting the following criteria would be considered for participation in the project:

- The technology had demonstrated, through previous bench testing and/or pilot testing, the potential to achieve mercury removal to 1 µg/l or less in clinical and/or research laboratory wastewater.
- The technology is commercially available.
- The vendor was willing to accept the vendor responsibilities developed by STEP in order to facilitate the testing (e.g., design, construct, install, and operate the pilot unit (all costs borne by vendor), accept that test results will be made public).
- The installation and operation of pilot units would not disrupt the operations of the host facilities.
- The technology would be applicable in treating wastestreams with high mercury concentration and low flow, and low mercury concentration and high flow, in order to assess system performance under several conditions. For the purposes of this program, high mercury concentration was defined as $\geq$10 µg/l, and high flow was >10,000 gpd.

Identified Candidate Technologies

A total of six companies, representing five technologies, were identified as potential participants in the project. Three of them (Aero-Terra-Aqua Technologies Corporation (ATA), ICET, Inc., and SolmeteX, Inc.) were invited to participate by STEP on the basis of their promising results in the bench scale feasibility testing with clinical laboratory wastewater conducted by the Technology Identification Subgroup. ICET's system utilizes a surface modified activated carbon to remove mercury, and the ATA and SolmeteX systems use other specialized adsorbents. Table 1 summarizes the results for these companies in the feasibility testing.

Table 1
Summary of Wastewater Mercury Removals
Bench-Scale Feasibility Testing Project (1)

Participating Vendor	Number of Test Runs	Influent Mercury (µg/L)	Final Effluent Mercury (µg/L)	Test Removals (%)
ATA Technologies Corp.	1	33.0 – 41.3	0.112	99.7
ICET, Inc.	4	12.8 – 17.1	0.1 - 4.8	71.7 - 99.3
SolmeteX, Inc.	9	12.8 – 24.9	0.114 - 1.1	94.4 - 99.2

(1) These results are based upon mercury concentration data produced by the MWRA Central Laboratory for samples from bench-scale feasibility test runs conducted by the participating vendors on a clinical laboratory wastewater.

The other potential candidates were companies that were not included in the bench scale feasibility testing, but approached STEP about participating in the demonstration project. One of these companies, DuBois Chemicals, provided STEP with data from bench testing they had conducted on research laboratory wastewater at a Boston area medical facility that established their potential to remove mercury to the 1 µg/l level through the use of chemical precipitants. They indicated, however, that they would need to partner with a membrane filtration vendor in order to provide a complete pretreatment system, and subsequently entered into a joint agreement with Prosys Corporation.

The other company that requested to participate in the demonstration project was the B.G. Wickberg Company, Inc. This company markets systems using Mersorb™, a sulfur-impregnated activated carbon, and has experience using this product to treat laboratory wastes and scrubber system wastewater streams from medical waste incinerators.

The six companies were sent information that provided an overview of the project and outlined the responsibilities of the vendor, STEP, and host sites for their use in determining whether they wanted to participate in the project. A copy of this information is included as Appendix A.

Final Vendor Selection

After reviewing the information on the project, as well as numerous discussions with STEP to address questions and concerns, the following five vendors, representing four technologies, agreed to participate in the project: ATA; DuBois/Prosys; ICET; and SolmeteX. Formal selection letters from Environmental Affairs Secretary Trudy Coxe were sent to each of these vendors (see Appendix B). Brief descriptions of the participating technologies are provided below.

Aero-Terra-Aqua (ATA) Technologies Corporation

ATA's technology is based on several chemically enhanced sorbents marketed as AQUA-FIX ™. These adsorbents are manufactured in granular and block form, and are designed to remove dissolved and ionic forms of metals. The removal process consists of three separate sorption steps:

1) adsorption of the metal ion onto the adsorbent's high surface area,
2) ionic interaction by absorption to polar sites, and
3) chemical bonding of the ions through chelation and ion exchange.

The combination of these processes allows for rapid and substantial metal removal. The adsorbents have been effective in the presence of surfactants and strong chelating agents. The AQUA-FIX ™ materials can be regenerated by rinsing with a dilute mineral acid. The use of filtering devices (such as particulate filters and activated carbon columns) to remove solids and organic compounds before the adsorbent bed(s) has significantly extended the bed life between backwashings and regenerations.

From ATA's experience in applications of their adsorbent technology, the following was found to optimize performance:

- Increasing retention times (contact) improves metal removal kinetics,
- Reduction of competing ions and enhancement of metal uptake can be achieved by a two-stage serially-operated AQUA-FIX ^{TM} system,
- Reduction of organic mercury concentrations with carbon pretreatment can enhance mercury reduction,
- Frequent column backwashing may be necessary for high solids-bearing wastewater streams, if prefiltration is not done.
- The optimum pH range for the adsorbent is 5.0 - 8.0 S.U.

DuBois/Prosys

The technology used by these two companies consists of first treating a wastewater stream with chemicals to create a suspension of solids that are then removed using a membrane filter. These companies have successfully used this approach to remove a variety of heavy metals (other than mercury) from hydroxide streams in a number of industrial applications. DuBois Chemicals found in bench-scale tests on research laboratory wastewater that the addition of two of their products (COAGULITE 222, a chemical coagulant, and EMR II, a chemical precipitant) to the wastewater at a pH of about 9.3, followed by filtration, was effective in removing mercury from the wastestream. The solids created by the chemical addition are removed using a Prosys crossflow microfiltration membrane system. This process involves the solid/liquid separation by a membrane at a simultaneously superimposed crossflow. The particles separated from the liquid by the membrane are continuously removed by the crossflow action at the membrane surface. The system is equipped with an integral backpulse component that maintains a clean membrane resulting in a longer period of operation between chemical cleanings.

The vendors cite the following advantages to their system:

- absolute separation of particles larger than 0.2 microns.
- dissolved components pass through the membrane.
- proven in large industrial plants.
- complete solid/liquid separation in one operation.
- system capacity is easy to adjust.
- the use of EMR II and COAGULITE 222 reduces sludge volume requiring disposal.

ICET, Inc.

ICET has been developing sorbent technologies for a number of years. The company's expertise is in the surface modification of activated carbon and sand to achieve sorbents capable of removing a wide range of heavy metals from wastewater.

Features of ICET activated carbon-based sorbents include:

- high mercury adsorption capacity.
- flexibility with respect to mercury levels and flow rates,.
- operation at alkaline or neutral pH.
- ambient temperature operation.
- the ability to recover and recycle mercury.

ICET has determined that the design of a successful pretreatment system for hospital wastewater streams must consider the potential for wastewater matrix interferences due to the variety of organics, trace metals and viable microorganisms present in the wastestreams.

The system utilized by the company in their bench scale feasibility testing consisted of a prefilter (to remove organic and inorganic particulate), a bed of activated carbon (for removing organics and additional particulate), an ICET media bed selected for heavy metal removal, and finally an ICET media bed selected for final mercury adsorption.

SolmeteX, Inc.

SolmeteX has developed an adsorbent called Keyle:X™ that is highly selective for ionic forms of mercury. This material applies the technology of selective chromatography, which is borrowed from the biotech industry. SolmeteX claims that the typical saturation loading of Keyle:X™ media is 38 to 45 percent mercury by weight, and that because of the higher selectivity of the media, the physical size of a SolmeteX system is smaller than those using other adsorbents. In addition to providing traditional end-of-pipe mercury pretreatment systems, the company has incorporated mercury removal into small point-of-use systems that offer the opportunity to prevent mercury contamination from reaching large volumes of wastewater.

The company uses cartridges filled with Keyle:X™ in their systems. Saturated Keyle:X™ can be distinguished by its change in color from yellow to black as the saturation front moves down the column of material. SolmeteX manufactures its cartridges in clear PVC so that the color change can be easily observed. Since the medium offers much higher selectivity and concentration (loading) factors than other adsorbents, such as ion exchange resins, mercury recovery from the spent medium is possible by having the user send cartridges of fully saturated material to a reclaimer where they can be burned for recovery of the mercury.

A SolmeteX mercury removal system includes a pretreatment step in which the wastewater is oxidized with hypochlorite solution at a pH of 6.5 to 6.7 to assure that the mercury in the wastewater is converted to soluble ions that can be removed by the Keyle:X™ media[(2)]. Chlorine demand is highly variable depending upon the waste stream. The Keyle:X™ media is not adversely affected by oxidizing agents. Additional pretreatment upstream of the Keyle:X™

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2 The oxidation step was developed with STEP assistance during the STEP-sponsored demonstration of the SolmeteX technology at the B.U. Medical Center where the technology was used to treat the wastewater discharged from the scrubber on the medical waste incinerator: Phase I report - Mercury Removal from Medical Waste Incinerator Scrubber Water, SolmeteX, Inc., December 16, 1996, and Update to Phase I Report - Mercury Removal From Medical Waste Incinerator Scrubber Water, SolmeteX, Inc., June 19, 1997.

columns is required to remove particulate and/or oil and grease in the wastewater. Since Keyle:X^{TM} is specifically designed to maximize mercury removal, the removal of other heavy metals in the wastestream, if necessary, would likely require the use of separate columns filled with a different media.

V. Selection Of Pilot Testing Host Facilities

Selection of the Types of Wastewater to be Tested

Because the demonstration project was conducted as an extension of the bench scale feasibility testing conducted by the Technology Identification Subgroup, the STEP project team decided, at a minimum, to perform the pilot testing on the same type of wastewater utilized in the earlier work, i.e., hospital clinical laboratory wastewater.

The Technology Identification Subgroup had selected clinical laboratory wastewater based on several factors. First, resource constraints limited the bench testing to only one type of wastewater. In order to select the wastewater to be used, the Subgroup reviewed the results of a sampling program conducted by the MWRA/MASCO Mercury Work Group Wastewater Characterization (WWC) Subgroup for five types of facilities - incinerators, power plants, hospital clinical laboratories, hospital laundries, and hospital research laboratories. It was determined that the largest mercury concentrations were in the discharges from clinical and research laboratories, and that the compositions of the two types of wastewater were similar. Specifically, the clinical laboratory used in the WWC sampling program showed parameter concentrations that were equal to the overall average of the parameter concentrations for the research laboratories tested, although the results for the research laboratory discharges showed a higher degree of variability. This similarity, coupled with the fact that the volume of clinical laboratory wastewater discharged to the MWRA system is much greater than the discharge from research laboratories, led to the selection of clinical laboratory wastewater for the bench testing project.

The STEP project team also selected research laboratory wastewater for use in the pilot project since it was felt that it would be important to evaluate the performance of mercury removal technologies using a wastestream with a greater variability in mercury concentration. Consequently, wastewater from both clinical and research laboratories were used in the pilot testing.

Description of Clinical and Research Laboratory Wastewaters

To understand the characteristics of these wastestreams and the difficulties that might be encountered when attempting their pretreatment, it is important to understand the operations or sources of waste that contribute pollutants to the individual wastestreams. In this way, the chemical constituents of each wastestream can be predicted and potential interferences in a mercury removal process can be anticipated.

The following are brief overviews of typical processes that produce wastewater in clinical and research laboratories. Each overview includes an interpretation of the analytical data generated from the wastewater sampling and analyses that were performed at representative facilities by the Wastewater Characterization (WWC) Subgroup.

Clinical Laboratories

Most clinical laboratories perform a wide range of services but not every clinical laboratory is the same. Some clinical laboratories are independent of hospitals. Generally, the larger the hospital, the greater the extent of services offered by the clinical laboratory. The types of processes performed in a clinical laboratory can include: anatomic pathology (including routine histology and cytology), chemistry, drug monitoring and toxicology, hematology, immunology and serology, microbiology, transfusion medicine, and urinalysis. Additional activities include cytogenetics, flow cytometry, histocompatibility testing, molecular pathology, mycology, and nuclear medicine.

Wastewater from a "typical" clinical laboratory could contain ionic, organically complexed, colloidal and particulate mercury and organomercuric compounds, other heavy metals, organic chemicals, blood products and body fluids, formaldehyde, buffers, dilute mineral acids/bases, phosphates, oxidizers, oil & grease, and particulate materials. Data from the WWC Subgroup's Wastewater Characterization Study suggests that clinical laboratory wastewater would have higher biochemical demand (BOD) and chemical oxygen demand (COD) than domestic sewage. Because there is usually some standardization of the work performed in a clinical laboratory, the wastewater from a specific lab may be somewhat consistent in quality and characteristics over long periods.

Research Laboratories

Perhaps the most diverse and unpredictable wastestreams are those discharged from research laboratories. Many medical institutions are conducting "cutting edge" studies in infectious disease control, blood chemistry, pathology, animal research and inorganic chemistry. Wastes may be produced in significant quantities for short periods or not at all for extended periods. Research laboratory facilities in hospitals can range from one to two laboratory sinks that produce "tens of gallons" each day to hundreds of sinks and related fixtures generating waste volumes in excess of fifty thousand gallons per day.

Wastes can originate from either automated instrumentation or from manual processes and may contain the following pollutants: oxidizers (disinfecting media such as bleach, iodine, peroxides, etc.), radionuclides, proteins (tissue and immunodiagnostics), oil & grease (from vacuum pumps and other rotating equipment), heavy metals (analytical reagents), organic solvents, blood products and other body fluids (urea is a well-known chelator of heavy metals), formaldehyde, phosphates and detergents (from glass cleaning and instrument sterilizing processes), and photographic imaging chemicals (desilvered spent fixer and developer solutions). Data from the Work Group's Wastewater Characterization Study suggests that (BOD) and (COD) are lower than for clinical laboratories but above average compared with domestic sewage.

Mercury Species in Wastewater

One result of the bench feasibility testing of mercury removal technologies was that it is important to understand the various species (forms) of mercury and how they might interact with other contaminants in the wastestream, especially because most mercury pretreatment technologies can effectively remove only certain mercury species. The various chemical species of mercury that can exist in wastewater are elemental, ionic, inorganically and organically complexed, and organometallic forms. In addition, these various species of mercury may bind to particulate matter in the wastewater to form colloidal and filterable particles containing mercury.

Elemental mercury is typically found in thermometers, manometers, sphygmometers, fluorescent lamps and switching devices. This form of mercury is a silver-colored liquid at room temperature with a specific gravity of 13 (*i.e.*, it is 13 times heavier than water), and it is only slightly soluble in water. Elemental mercury slowly vaporizes at room temperature and can cause dangerous vapor concentrations in enclosed rooms. The vapor form of elemental mercury is readily absorbed through the lungs and is very toxic. Elemental mercury may be combined with other metals to form amalgams (alloys).

Ionic forms of mercury include the free ion and complexes produced when mercury atoms form covalent bonds with halogens and other inorganic ligands (complex ions). The free mercury ion can exist in two forms, i.e., the mercuric form, a single ion with an overall +2 charge (Hg^{++}) or, under mildly reducing conditions, as the mercurous form that is diatomic with an overall +2 charge (Hg_2^{++}). The mercuric form readily forms salts (e.g., mercuric chloride - $HgCl_2$) that are soluble in water. Mercuric chloride and Calomel (mercurous chloride – Hg_2Cl_2) are often used in medical applications.

Organometallic mercury (e.g., methyl mercury) consists of mercury atoms covalently bonded to organic groups. Often called organomercuric compounds, these forms of mercury are quite soluble in water and wastewater and are extremely toxic to aquatic life. These compounds are readily absorbed by fish from their aqueous environment and tend to become highly concentrated (bioaccumulated) in the fish tissues. If fish having substantial bioaccumulated organic mercury are consumed, there can be major human health concerns. Inorganic mercury in the environment can be converted by microbiological activity into methyl mercury compounds that can be absorbed by fish. Mercury may also form stable organic complexes with a wide variety of organic ligands found in both natural and wastewaters, a condition which often results in most of the mercury present in such waters existing as organometallic or organically complexed mercury.

These various species of mercury can bind to particulate matter that exists in wastewater. Particulate-bound mercury can enter the food chain through ingestion (filter feeding organisms) or through re-conversion to and uptake of dissolved forms. Mercury-laden particulate matter can range in size from tens of microns to sub-micron (colloidal). Typical EPA methodology (Methods 200.7, 200.9, and 245.1) separate dissolved from particulate mercury by filtration through a 0.45 micron (µm) membrane filter.

As a physical species of mercury (instead of the previous chemical species), particulate mercury can often be a significant fraction of total mercury in a wastewater stream. Moreover, accumulations of elemental mercury or mercury-laden solids in plumbing systems (at elbows, traps, and other points) can cause chronic mercury contamination of the wastewater stream.

Pilot Testing Site Selection Process

The following process was used to identify and select the host facilities for the project:

- *Established selection criteria* – the STEP project team developed the following criteria that all potential sites would need to meet –
    - the facility would be within the MWRA service district.
    - to assess system performance under several conditions, candidate sites would have wastestreams showing either high mercury concentration and low flow, or low mercury concentration and high flow (for the purposes of this program, high mercury concentration was defined as $\geq$10 µg/l, and high flow was >10,000 gpd).
    - the wastestream(s) of interest at a candidate site were to be easily accessible, with sufficient space in close proximity to the wastestream(s) to locate pilot testing equipment.
    - facility management was willing to accept the host site responsibilities developed by STEP in order to facilitate the testing (e.g., provide all utilities necessary for testing, assume the cost for waste disposal, accept that test results would be made public).

- *Identified candidate sites* - using MWRA sampling data for late-1996 to mid-1997, the team identified potential wastestreams at seven facilities – Beth-Israel Deaconess Hospital; Brigham & Women's Hospital; the Lahey Clinic in Burlington; Massachusetts General Hospital; Newton-Wellesley Hospital; SmithKline-Beecham Clinical Laboratory; and the USDA Human Nutrition Research Center on Aging at Tufts University.

- *Solicited site interest* – each of these seven facilities was invited to participate in the pilot-testing project. They were sent information describing the project along with a survey form to return to STEP that would allow for an assessment of the facility's suitability as a test site, as well as their willingness to participate in the project. A copy of the information packet and survey form sent to the facilities is included as Appendix C.

- *Meetings with interested sites* – a total of four facilities agreed to further discussions with the project team concerning their participation in the project - Brigham & Women's Hospital, Massachusetts General Hospital, Newton-Wellesley Hospital, and the USDA Human Nutrition Research Center on Aging at Tufts University. Meetings were held with each of the facilities to discuss the project in detail and review the responsibilities of STEP, the technology vendors, and the host sites. The meetings were held to determine the facilities' level of interest in participating in the project, address their questions and concerns, and assess the viability of the potential testing location(s) STEP had identified at the site.

- *Final site selection* – on the basis of these discussions, three facilities were selected to serve as test sites. Formal letters of notification from Environmental Affairs Secretary Trudy Coxe were sent to each facility (see Appendix D). The three facilities are described below:
  - **Newton-Wellesley Hospital** (high mercury concentration/low flow site) - the hospital collects all mercury-contaminated wastewater from it's clinical laboratories and blood bank in six 300 gallon tanks. The wastewater is removed from the site every 10 - 12 days for off-site treatment. Average monthly flow was reported to be about 4,500 gallons with mercury concentrations typically in the 20 - 30 µg/l range. Three of the 300 gallon collection tanks were designated as feed tanks for the pilot testing. The hospital installed plumbing connecting three of these tanks to the pilot units to be tested.
  - **Brigham & Women's Hospital** (low mercury concentration/high flow site) - this site has a clinical laboratory wastestream with a flow of about 10,000 gpd and mercury concentrations ranging from <1 to 11 µg/l reported during 1997. The pH of the wastestream is adjusted prior to discharge to the MWRA. The wastewater used in the pilot units was taken as a slipstream from the outlet of the pH adjustment system.
  - **USDA Human Nutrition Research Center on Aging at Tufts University** (high mercury concentration/low flow site) - this site has a research laboratory wastestream with a flow of about 1,400 gpd and mercury concentrations ranging between 2 and 66 µg/l reported during 1997. All the wastewater from this facility flows through a settling tank prior to pH adjustment and discharge to MWRA. The wastewater used in the pilot units was taken as a slipstream from the inlet to the settling tank. The effluent from the pilot units was discharged into the settling tank.

VI. Technology Vendor Visits To Host Facilities

At the completion of vendor and host site selection, and prior to the start of pilot testing, STEP conducted visits of the three host facilities for the technology vendors so they could meet site personnel, become familiar with the sites (e.g., wastewater characteristics, access to discharge to be tested, and area available to install their equipment), and identify any concerns they had. ATA visited the sites in November 1997, and ICET, Prosys/DuBois, and SolmeteX made their tours in December 1997.

Based on these visits, SolmeteX notified STEP that they would prefer to test their system as a point-of-use application, rather than as an end-of-pipe application, since they felt they already had sufficient data to characterize the performance of their technology as an end-of-pipe application. STEP considered this request, and met with the sites to assess their interest and viability to allow this type of testing. On the basis of this review, STEP offered all vendors the option of pilot testing their system as a point-of-use application. The final vendor decisions concerning the type of testing they would perform are summarized below –

- End-of-pipe only – ATA and Prosys/DuBois.
- Both end-of-pipe and point-of-use – ICET.
- Point-of-use only – SolmeteX.

VII. Scope Of Work For End-Of-Pipe Pilot Testing

The STEP project team developed a detailed written protocol outlining how the pilot testing was to be conducted. Appendix E is a copy of this document, "Scope of Work – Field Testing of End-of-Pipe Mercury Removal Technologies". This document outlines the responsibilities of the vendors with regard to the design, construction, installation, and operation of their pilot unit. STEP specified that runs would consist of 8 to 10 hours of continuous operation on weekdays during normal business hours, and that sample collection would only occur during these periods. The duration of testing at each of the three host facilities was also delineated. Prior to initiating testing, the vendors were required to submit experimental plans that included detailed descriptions of their pilot system, including drawings, and the experimental protocol they would be following. Upon completing their testing, the vendors were asked to submit final reports to STEP that included the following: the bench testing results; the methods used in pilot testing; the pilot test results (including the mercury sampling results); full scale system considerations; and conclusions (these reports are attached as Appendices G, H and I).

The responsibilities of the host facilities are also described in the scope of work, and are primarily associated with assisting the vendors with the installation of their systems, covering the costs of utilities used in the testing at their facility, and managing the handling and disposal of waste materials produced during the testing, including disposal costs.

Lastly, STEP's role in the project is defined. In addition to the project management, coordination of vendor testing, and oversight provided by Dr. John Raschko of the Office of Technical Assistance, STEP was also responsible for all sampling and mercury analyses for the project under the supervision of Dr. Gordon Wallace of UMass Boston. Dr. Wallace has developed a technique for performing mercury analyses, based on modifying EPA Method 245.1, capable of routinely achieving an analytical detection limit of 0.002 µg/l using a CETAC Model 6000 Mercury Analyzer. The sampling and analytical support is summarized below, and was conducted according to the Quality Control plan developed by Dr. Wallace (See Scope of Work, Attachments 3 and 4):

- *Characterization of effluents prior to initiation of pilot testing* - the effluent waste stream being used at each host facility was sampled on an hourly basis over a twenty four hour period. Each hourly sample was collected as a composite of samples collected at 15 minute intervals to capture short-term changes in concentration. The sampling was conducted using automatic samplers loaned to STEP by MWRA. This sampling allowed STEP to provide the vendors information on the temporal heterogeneity of the effluent before conducting the field tests.

- *Sample analysis in support of preliminary bench tests* - analysis of samples generated in the conduct of preliminary bench-scale tests to optimize pre-conditioning of the effluent for mercury removal before actual conduct of the field tests was supported.

- *Characterization of influent and effluent streams during operation of pilot units* – autosamplers provided by MWRA were used to collect influent and effluent samples that were analyzed by UMass Boston. In general, hourly samples were collected during the first several days after startup of a pilot unit to assist the vendors in fine-tuning the operation of their systems. Once this was achieved, daily composite samples were collected for the duration of the test period.

VIII. Activities Conducted Prior to Pilot Testing

Bench Testing By Technology Vendors

Prior to performing any field testing, the vendors conducted bench testing at their facilities in order to determine final pilot system design (including an assessment of preconditioning requirements) and initial operating conditions for the pilot testing. This testing was done using wastewater samples obtained from all three sites by experienced MWRA sampling associates in late January 1998. Five-gallon sample containers were packed in ice-filled coolers for overnight shipment to each participating vendor. Each vendor had the opportunity to specify the number of five-gallon samples they received. UMass Boston analyzed the mercury levels in the wastewater samples sent to the vendors as well as samples submitted by the vendors to assess their bench scale performance. The results of these tests for DuBois/Prosys and ICET are presented in their reports found in Appendices H and I, respectively. ATA's bench test results were not included in their pilot testing report, and the results of analyses performed by UMass Boston are presented in Table 2 below. They chose not to perform bench testing using wastewater from Newton-Wellesley Hospital since they had participated in the 1997 bench scale feasibility testing conducted by the MWRA Technology Identification Subgroup which was carried out using wastewater provided by this facility.

Table 2
Summary of ATA Technologies Bench Testing Conducted Prior To Pilot Testing

Host Facility	Mean Influent Mercury (µg/L)	Mean Effluent Mercury (µg/L)
Brigham & Women's Hospital	24.2	0.53
USDA Human Nutrition Research Center on Aging at Tufts University	41.7	0.15

Mercury Analysis Of Discharges To Be Treated Conducted Prior To Pilot Testing

High-resolution sampling of the wastestreams to be treated was conducted before pilot tests at each site to determine the mercury concentrations (expressed as µg/l) over a 24-hour period. The results of this sampling at all three sites indicated a pattern of discharge consistent with contemporary release of mercury to the wastewater stream and not a general "bleeding" of mercury into the system by desorption from surfaces in contact with the wastestream. Fluxes of mercury (expressed as mg/hour) were estimated at Brigham and Women's Hospital where the wastewater flowrate was monitored continuously. Mercury fluxes were not calculated for Newton-Wellesley Hospital and the USDA Human Nutrition Research Center on Aging at Tufts University because accurate flow readings were not available at the time of sampling.

<u>*Brigham and Women's Hospital*</u>

The variability in mercury concentration and flux in two surveys (3/26-27/98 and 4/23-24/99) at Brigham and Women's Hospital are presented in Figures VIII-1 through VIII-4. Samples were taken from two locations at this site. The first location (Tank) was at the outlet of the pH adjustment system that served as the source of the wastestream used in the pilot testing. The second sampling station (Pipe) was at the location where the MWRA obtains its monitoring samples for this wastestream. It is located about 50 m downstream from the pH adjustment system and at the point where the wastestream is discharged into the MWRA system.

Sampling was initiated at ~2PM on 3/26/98 and at ~ 12PM on 4/23/99. The initial sample taken on 3/26/98 contained an extraordinarily high concentration of mercury (~600 µg/l, Figure VIII-1) and may reflect the accumulation of mercury-enriched particles in the inlet tube of the automatic sampler before sampling was initiated. No such anomaly was observed in the pipe sample from this survey or in the April survey (Figure VIII-2). Both samplings at each location reveal an apparent bimodal pattern of discharge with maxima occurring in concentration in the late morning (11-12PM) and late afternoon (3-5PM). Differences in the magnitude and duration of mercury concentrations and fluxes obtained at the two sampling locations may reflect temporal variations on a time scale shorter than the 15 minute interval sampling used to generate the hourly composites coupled with differences in the time of sampling at each location. In-pipe diffusion may also have contributed to these differences. Nevertheless, the maxima observed are consistent with the discharge of waste generated from analytical activities initiated at the beginning of the workday and after lunch. Maximum concentrations reached over 60 µg/l (excepting the anomalous observation of 600 µg/l) in the March 1998 samples and approached 90 µg/l in the April 1999 samples. Outside of normal working hours, concentrations decreased to lower values that generally fell between 1 and 6 µg/l with the exception of the early morning hours (2 – 6AM) in the April 1999 sampling when concentrations fell below 1 µg/l. The flux of mercury from the waste collection system (Figures VIII-3 and VIII-4) generally reflected the patterns in concentration. The majority of the release of mercury occurred between 9AM and 6PM.

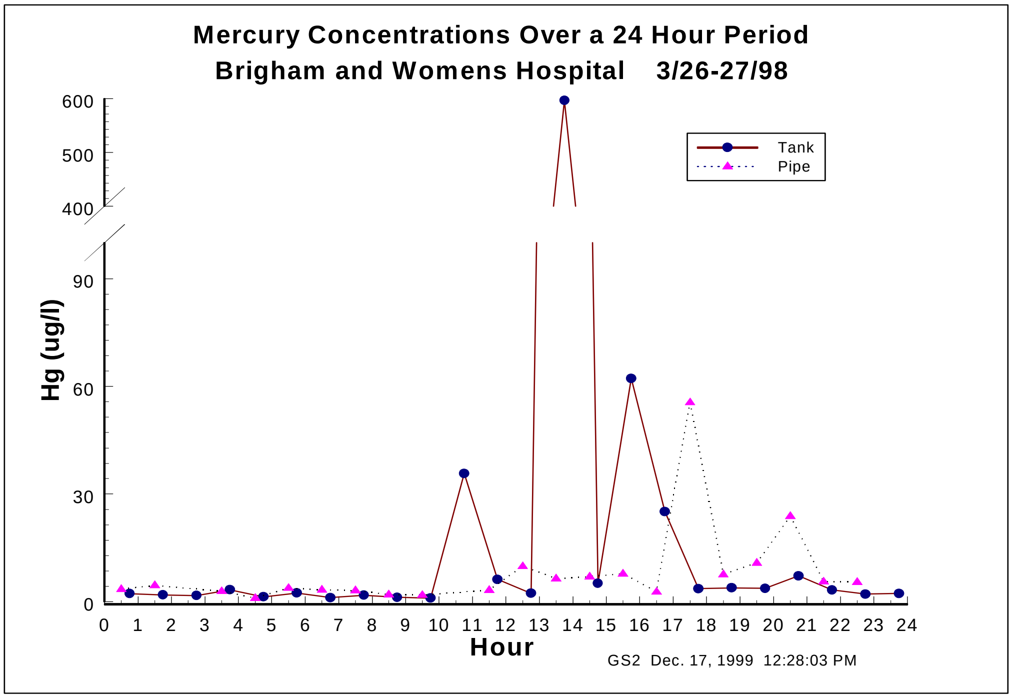

Figure VIII-1: Concentration of mercury over 24 hours. Sampling was initiated on March 26 at 1:45 PM (Hour 13.75 in figure) and completed at 12:45 AM on March 27.

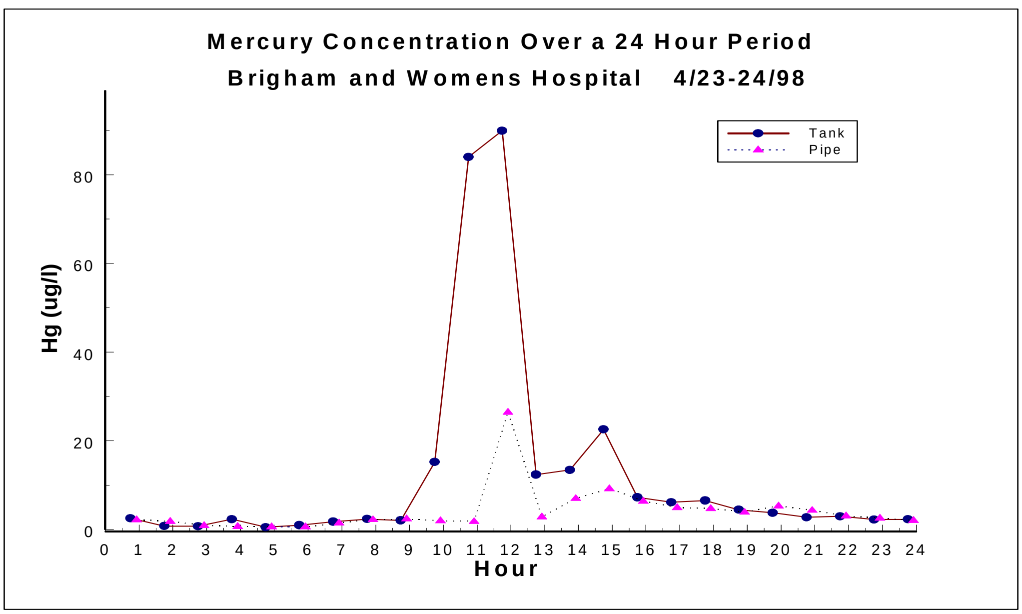

Figure VIII-2: Concentration of mercury over 24 hours. Sampling was initiated on April 23 at 12:00 PM (Hour 12 in figure) and completed at 12:00 PM on April 24.

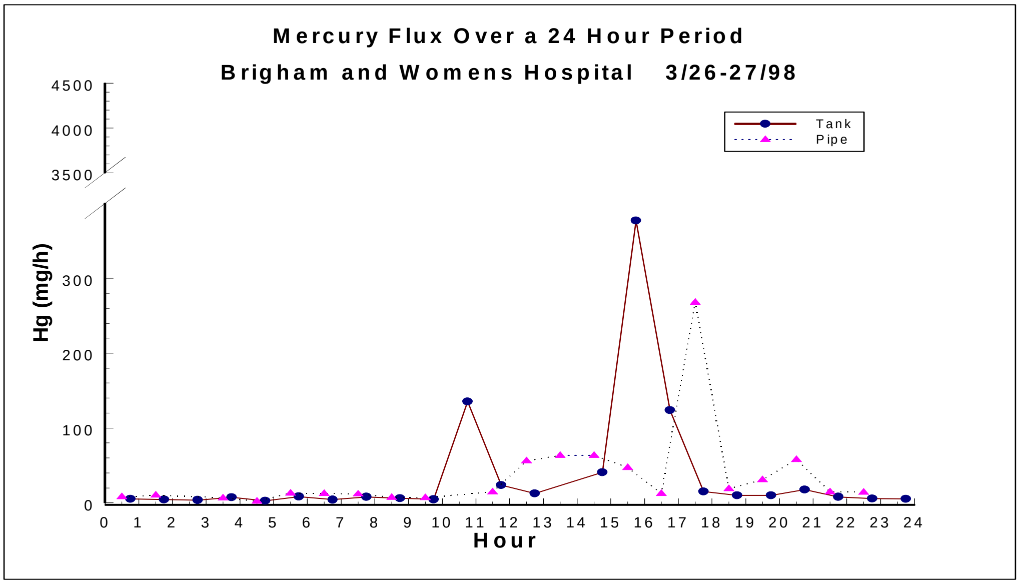

Figure VIII-3: Flux of mercury over 24 hours using flow data provided by Brigham and Women's Hospital and data in Figure VIII-1. Note that the flux from the anomalous concentration in Figure VIII-1 is not included in this plot (see text).

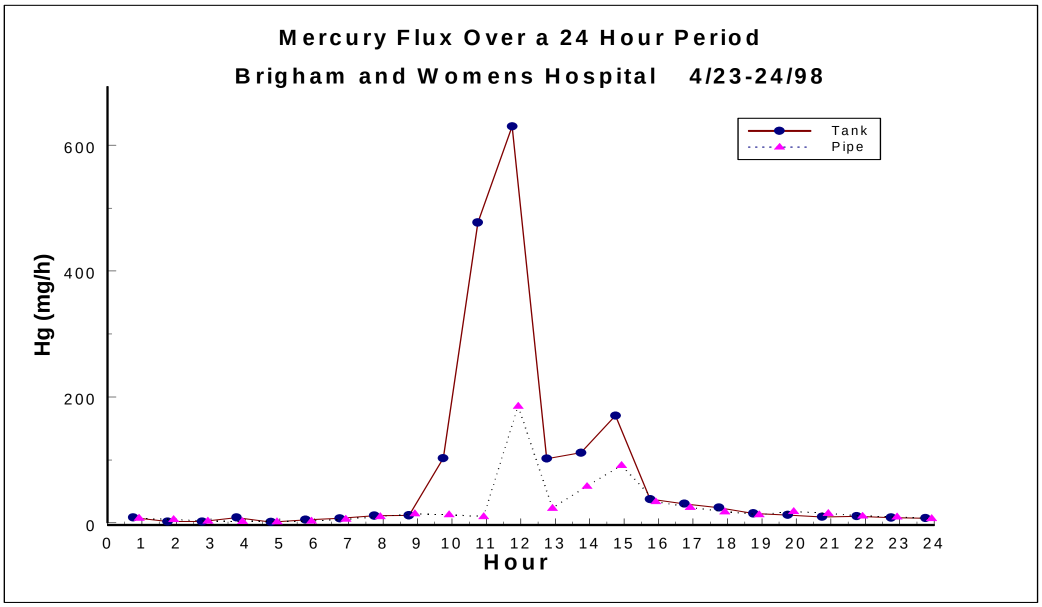

Figure VIII-4: Flux of mercury over 24 hours using flow data provided by Brigham and Women's Hospital and data in Figure VIII-2.

<u>*Newton-Wellesley Hospital*</u>

There was a single 24-hour sampling conducted at Newton-Wellesley Hospital from the drain line delivering wastewater from the clinical laboratories to the holding tanks used by the hospital to store their liquid wastes for removal offsite (see above). Concentrations observed between the initiation of sampling on 9/30/98 at 8:30AM to 8:30AM the following morning are presented in Figure VIII–5. Total flow was estimated at irregular intervals by observation in changes in volume of the tank being used to collect the wastewater during sampling. Concentrations after 11:30PM reflect a period when there was little or no flow. Of the total 225 gallons collected over the 24 hr period, only about 50 gallons were collected over the 14 hour period between 6:30PM and 8:30AM the following morning. Although the data are limited, the results are consistent with those observed at Brigham and Women's where the bulk of the mercury was discharged during regular working hours (in this case between 8:30AM and 6:30PM). The initial sample had the highest concentration observed (~ 19 µg/l), and was blue in color and smelled like gram stain. The samples collected between 7:30PM and 12:30PM were also high in mercury and highly colored (yellow and purple) suggesting, perhaps, that the cleanup from laboratory staining activities may have been a source.

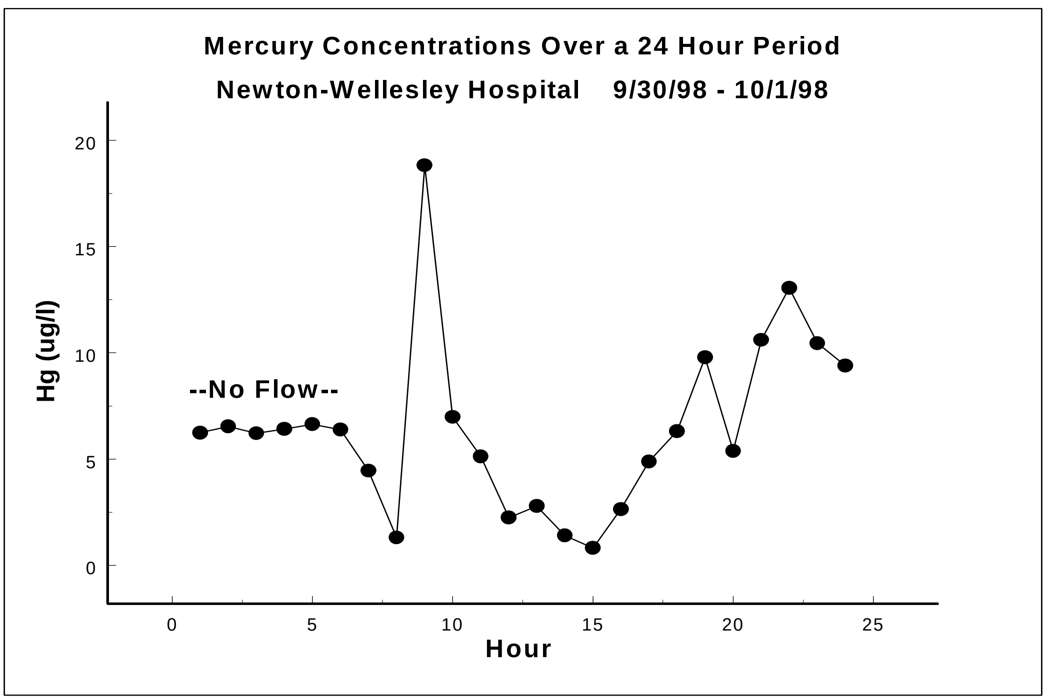

Figure VIII-5: Concentration of mercury over 24 hours. Sampling was initiated on September 30 at 8:30AM (Hour 9 in figure) and completed at 8:30 on October 1, 1998.

USDA Human Nutrition Research Center on Aging at Tufts University

Twenty-four hour sampling was conducted on three different occasions at this site: March 26-27, 1998; June 4-5, 1998 and March 24-25, 1999. The initial samples were taken at 2:30PM, 2:00PM and 8:00AM, respectively. In addition, a series of shorter hourly sampling activities were conducted. This hourly sampling was conducted between 7AM and 5PM on two successive days in October 1998 (6^{th} and 7^{th}) and between 7AM and 7PM in November 1998 (19^{th} and 20^{th}). Overnight wastewater flowrates were too low to allow 24-hour sampling during these two periods.

Concentrations in the March 1998 samples (Figure VIII-6) were generally between 1 and 2 µg/l except between 12 and 4PM where concentrations exceeded 2 µg/l in three out of the four samples collected in that time period. Concentrations were generally much higher in the June 1998 samples (Figure VIII-7) and reached highs of 45 µg/l in the 5-6AM sample and 25 µg/l in the 11AM-12PM sample. All of the highest concentrations in the June samples were observed between 5AM and 5PM. Hourly samples collected in October 1998 were quite low and did not exceed 0.4 µg/l (Figure VIII-8). Samples collected between 7AM and 2PM on the following day were also quite low but then increased sharply after 2PM to reach a maximum concentration of 3.2 µg/l in the 3-4PM sample. In the second two-day series of hourly samples collected in November 1998 between 7AM and 7PM (Figure VIII-9), two of the samples taken on the morning of the 19^{th} contained very high concentrations of mercury (10.9 and 48.8 µg/l). Concentrations then fell below 1 µg/l after 2PM. Concentrations were much lower in the samples collected on the 20^{th} with a maximum of 1.9 µg/l occurring in the 10-11AM sample and then decreasing to concentrations well below 1 µg/l thereafter. The last 24-hour sampling that occurred in March 1999 illustrated the dramatic temporal variability in concentrations that can occur in such wastestreams (Figure VIII-10). The initial sample collected between 7 and 8AM was among the highest observed in all the samples collected as part of the initial sampling at the three sites (234 µg/l). The concentration in the next sample dropped by an order of magnitude and then by another two orders of magnitude in the following sample. Concentrations in the subsequent samples generally remained below 0.1 µg/l. The variability of the concentrations over time are consistent with greater variability in mercury use in the laboratory typical of a research facility in contrast to the pattern of usage in a clinical laboratory where most of the analytical work is routine. In addition, the use of reagents with extremely high mercury concentrations (see Section X), coupled with lower overall wastewater flow at this site, tends to amplify mercury concentrations in the waste stream.

While we cannot confirm that the two samples first collected in the March 1999 series were not due to contamination despite our best efforts to use trace-metal clean techniques, it seems unlikely. Studies performed prior to 24-hour sampling and pilot testing confirmed that autosampler blanks were acceptable (<0.05 µg/l, determined by analysis of samples collected from a deionized water reservoir). Furthermore, procedural blanks throughout the project were always close to our detection limit (typically <0.01 µg/l) and precision, assessed by analysis of replicate samples and replicate analysis of the same sample, was generally better than +/- 5%. We thus suspect that excursions such as those seen in March 1999 are real and reflect the discharge of high mercury-containing laboratory wastes, an observation consistent with the results of our "prescreening" studies conducted at Newton-Wellesley and Tufts that are discussed in Section X.

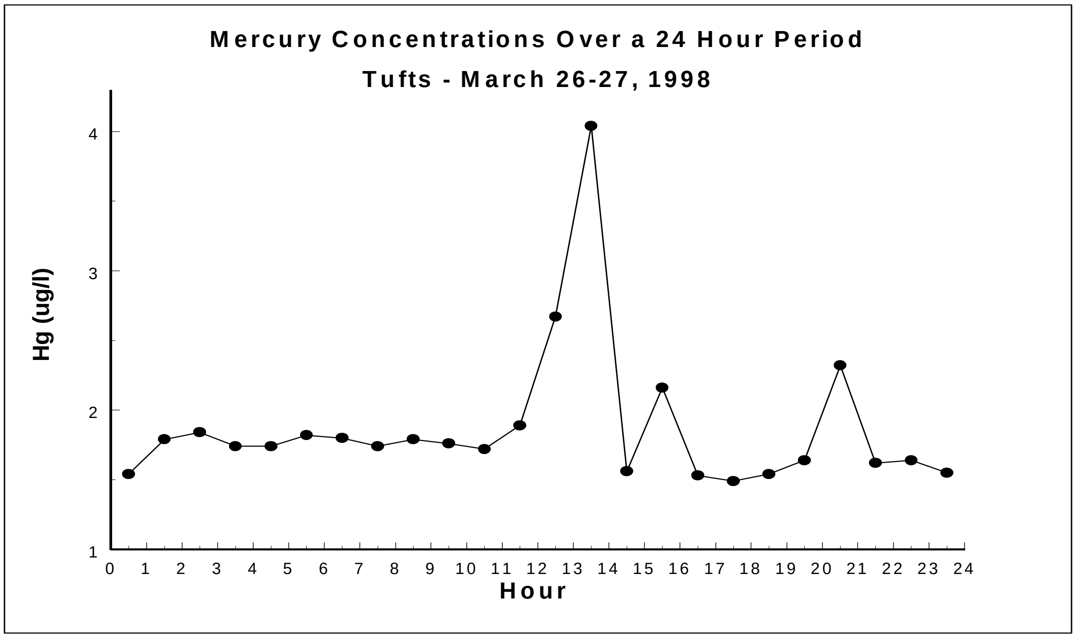

Figure VIII-6: Concentration of mercury over 24 hours. Sampling was initiated on March 26 at 2:30 PM (Hour 14.5 in figure) and completed at 2:30 PM on March 27,

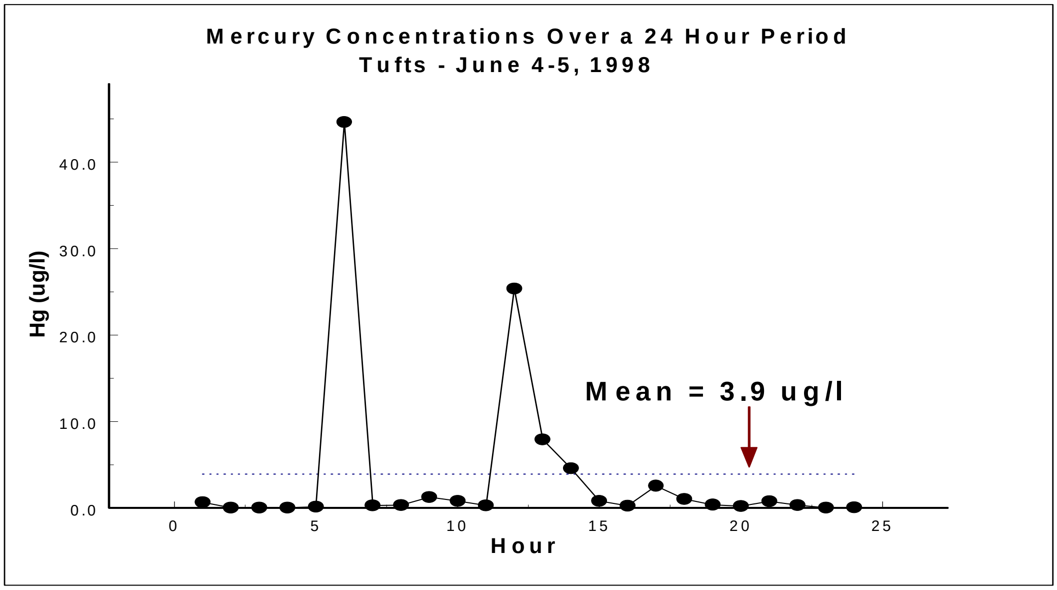

Figure VIII-7: Concentration of mercury over 24 hours. Sampling was initiated on June 4 at 2:00 PM (Hour 15 in figure) and completed at 2:00 PM on June 5, 1998.

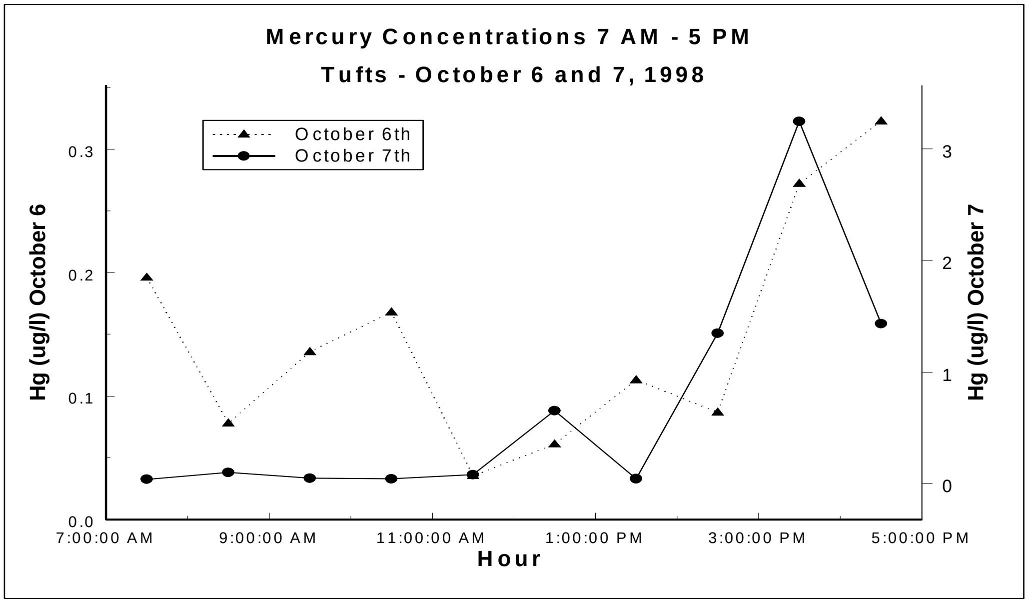

Figure VIII-8: Hourly samples taken between 7 AM and 5 PM on October 6 (left axis) and October 7 (right axis). Note differences in scale.

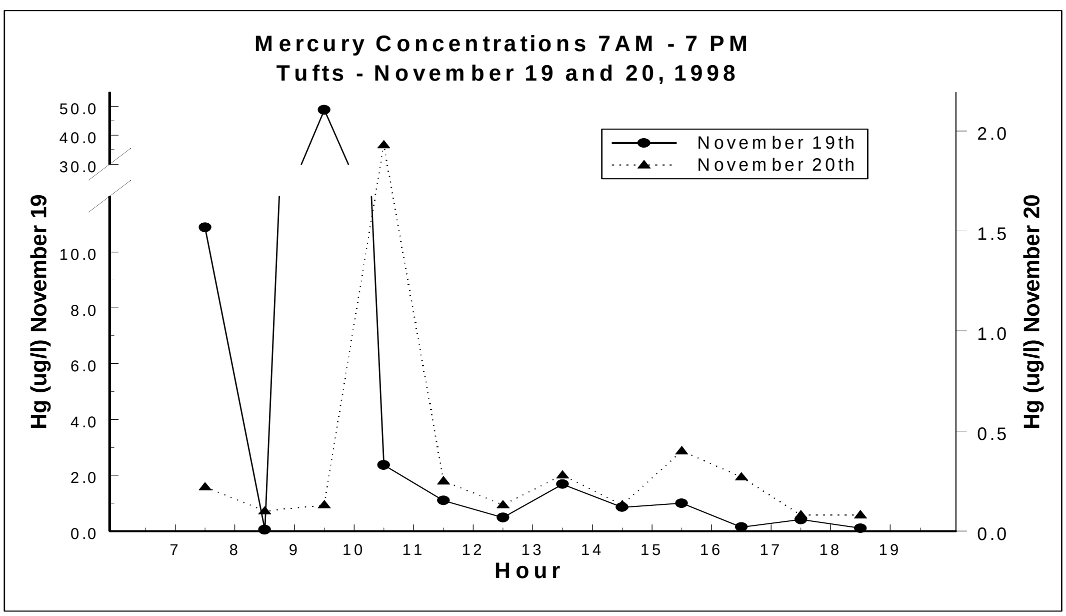

Figure VIII-9: Hourly samples taken between 7 AM (hour 7.5) and 7 PM (hour 18.5) on November 19 (left axis) and November 20, 1998 (right axis). Note differences in scale

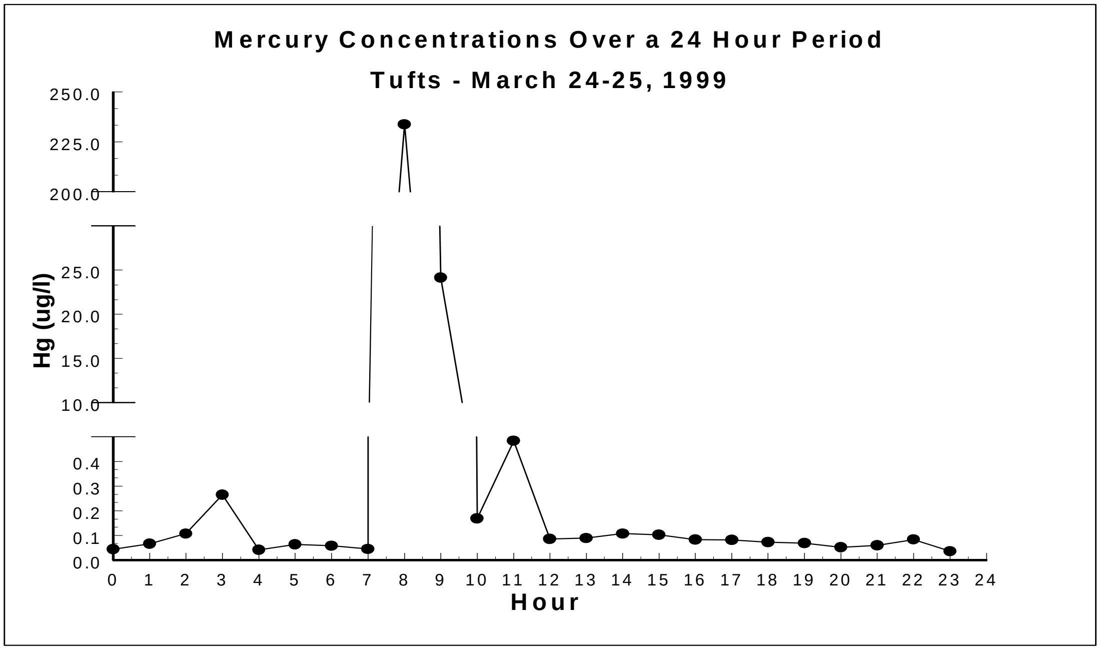

Figure VIII-10: Concentration of mercury over 24 hours. Sampling was initiated on March 24 at 8:00 AM (hour 8 in figure) and completed at 8:00 AM on March 25, 1999. Note scale changes.

Summary

In summary, the initial sampling at all three sites produced two conclusions:

- The bulk of the mercury discharged at the three sites coincided with periods of active use of the laboratories involved and thus could not be attributed to desorption of mercury from the host site's waste collection system.

- Temporal excursions in mercury concentration could be substantial and short-lived. Thus, in the absence of an appropriately sized equalization tank to smooth out mercury concentrations, end-of-pipe technologies used to remove mercury from the wastestream would have to have extraordinarily high removal efficiencies.

Especially important in these findings is that the contemporary discharge of mercury from all three sites probably involves active use of reagents containing high concentrations of mercury that are released to the waste collection system of the sites. We will further support this conclusion in our discussion of the prescreening results later in this report (Section X).

IX. Pilot Testing of End-of-Pipe Mercury Removal Technologies

The pilot testing of the three end-of-pipe technologies took place between late May 1998 and June 1999. As described in Section VII, the original plan for the demonstration project was to test the three systems at all the sites. Several factors, however, contributed to STEP having to scale back the testing schedule. These included the need for more extensive system shakedown and debugging for several of the vendors, the eventual decision by these vendors to limit their testing to only one site, and the decision by Newton-Wellesley to withdraw from the project after only one vendor had conducted testing at that site. The actual testing conducted is summarized in this section, and is described in detail in the vendor reports in Appendices G, H and I.

ATA Technologies

ATA performed several rounds of testing at Newton-Wellesley Hospital, ranging in duration from one day to about a week, from late May 1998 through December 1998, the results of which are summarized in Figure IX-1a. This figure demonstrates that removal was generally more successful when a preoxidation step was included in the process. The company's final report (see Appendix G) describes the December 1998 testing only. Their pilot unit, not including equipment associated with preconditioning the wastewater, consisted of six cartridge housings connected in series which were used to contain the prefilters (for particulate removal) and media cartridges used in the tests. Over the course of their several rounds of testing on a clinical laboratory wastewater, the company examined a variety of operating conditions (e.g., with and without preconditioning, using several types and combinations of AQUA-FIX ™ media, modifying prefiltration) in their efforts to achieve consistent mercury removal to below 1 µg/l. The results of the testing performed in December 1998, which was the most successful, are summarized in Figure IX-1b. This series of runs was comprised of three phases, all of which included preoxidizing the wastewater with bleach in a 55 gallon pretreatment tank as the first treatment step. The differences between the three phases were primarily in the amount of media used. In the first phase, which was 1-1/2 days in duration, the preoxidized wastewater passed through two prefilters (1 micron followed by 0.5 micron), a cartridge of granular AQUA-FIX ™ 701 media, and three cartridges of granular AQUA-FIX ™ 401 media (solid circles in Figure IX-1b). The second phase was 2-1/2 days long and was the same as the first phase except that only two cartridges of granular AQUA-FIX ™ 401 media were used (crosses in Figure IX-1b). The final phase of testing, which was the least successful, was conducted for 1-1/2 days and was the same as the second phase with the exception that only one granular AQUA-FIX ™ 401 media cartridge was used and a second 1 micron filter was installed directly upstream of it (squares in Figure IX-1b).

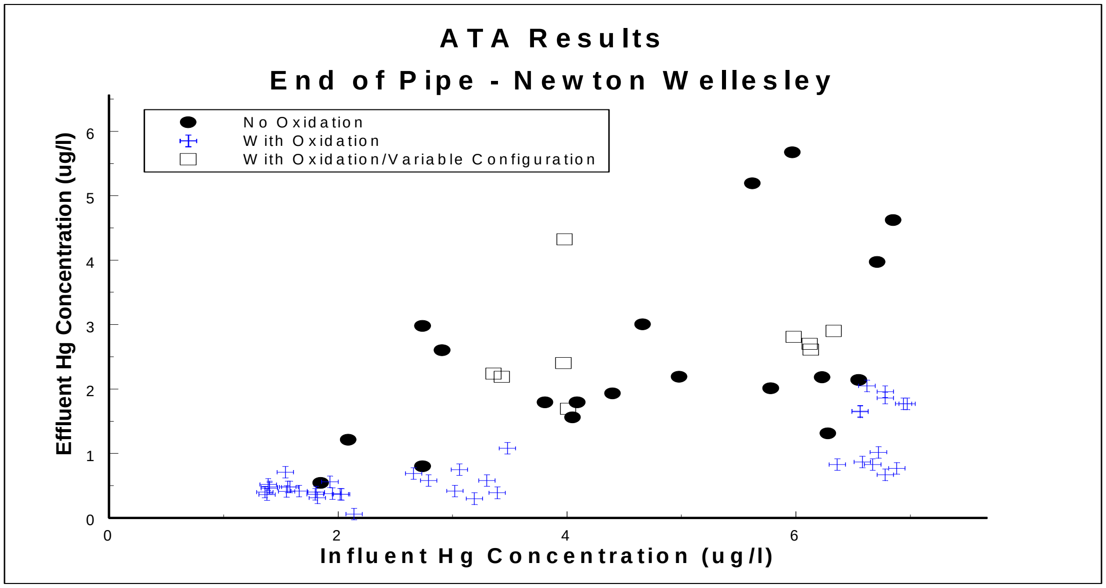

Figure IX-1a: Mercury concentration of effluent plotted as function of influent mercury concentration for all pilot testing of the ATA system at Newton-Wellesley Hospital. Variable configuration data (open squares) refers to data obtained at the end of there December testing when the configuration of sorbents and filters in their treatment system were being changed. See their report (Appendix G) and Figure IX-1b for details.

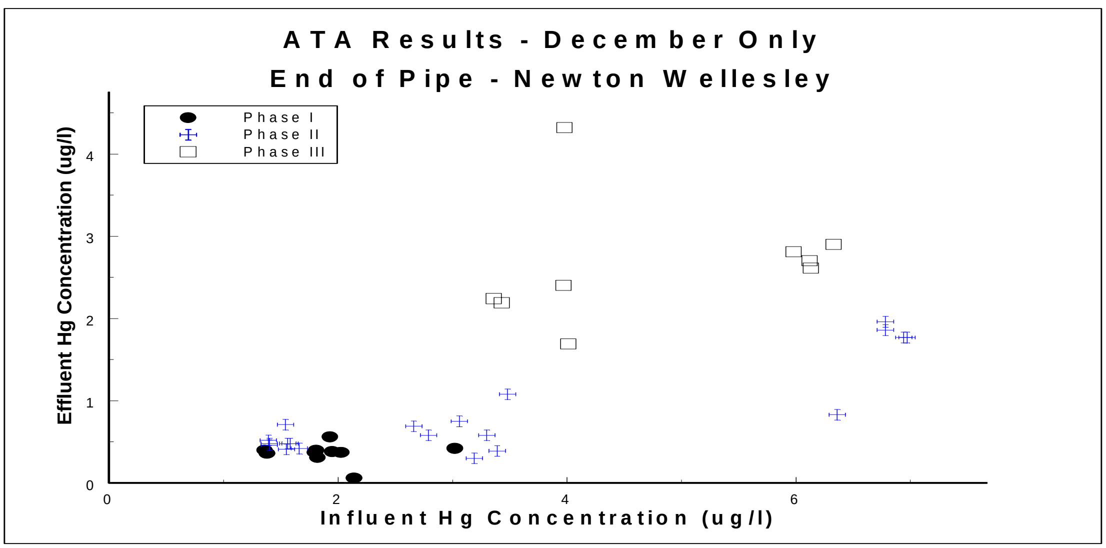

Figure IX-1b: Mercury concentration of effluent plotted as function of influent mercury concentration for Phases I-III December, 1998 pilot testing of the ATA system at Newton-Wellesley Hospital. Phase III data were obtained at the end of the testing when they were varying the configuration of sorbents and filters in their treatment system. See text and their report for details (Appendix G).

<u>*DuBois/Prosys*</u>

These companies used their pilot unit in two rounds of testing on the clinical laboratory wastewater at Brigham & Women's Hospital over the period June 1998 through December 1998 (see Appendix H). The first phase was conducted from mid June 1998 to late July 1998. The treatment approach used during that period involved first adding DuBois EMR-II, a chemical precipitant, to the wastewater in a mixed reaction tank (an EMR-II concentration of 200 ppm was maintained). The pH of the wastewater was also adjusted in this tank. From the reaction tank, the water flowed into a concentrate tank from which it was pumped through a 0.2 micron tangential-flow membrane. The water passing through the membrane was the cleaned effluent from the system, and was discharged to the sewer. The untreated wastewater exiting the membrane was returned to the concentrate tank. Except for a period when the pH controller was malfunctioning, the results of this phase of testing were generally favorable. Effluent mercury levels below 2 µg/l were typically achieved, even during spikes in the influent mercury concentration, but levels below 1 µg/l, while seen, could not be consistently attained (solid circles in Figure IX-2).

After making several modifications to the system, a second round of testing was conducted from late October 1998 through mid December 1998 to determine whether consistent removal to below 1 µg/l could be achieved. The changes made were the use of DuBois COAGULITE 222, a chemical coagulant, in concert with the EMR II; the addition of a small surge tank upstream of the reaction tank to allow for more uniform flow to the reaction tank; and the installation of a data-logging device to accurately record pH values in both the reaction tank and final effluent. The results for the first several weeks of testing under these new conditions revealed that the membrane had ruptured. Once it was replaced, however, the system consistently removed mercury to < 1 µg/l in the effluent, even during a period of very high influent mercury levels (up to 109 µg/l) at the end of the test period (crosses in Figure IX-2).

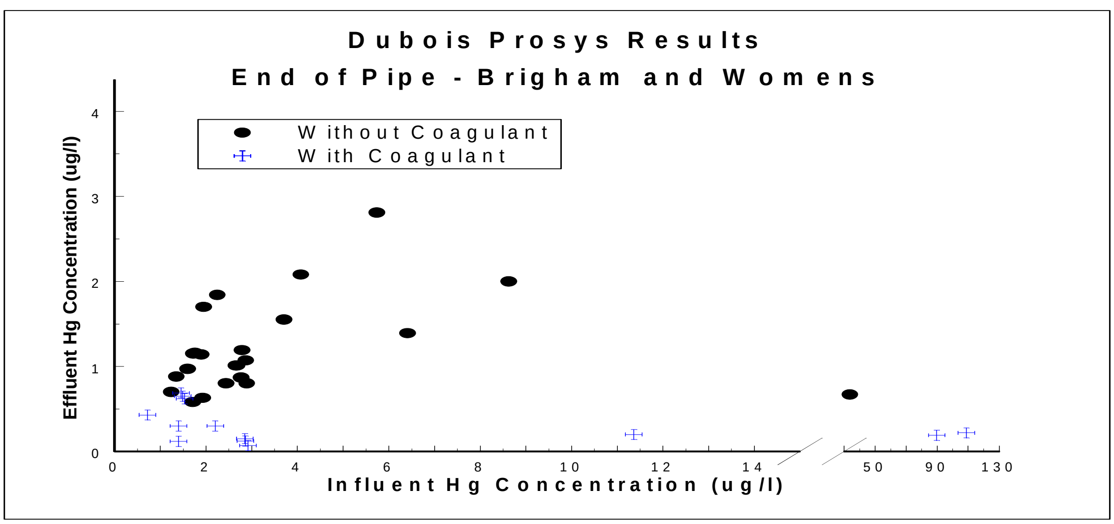

Figure IX-2: Mercury concentration of effluent plotted as function of influent mercury concentration for the pilot testing of the DuBois/Prosys system at Brigham and Women's Hospital. Note split scale on X axis. Use of the coagulant probably destabilized colloidal forms of mercury to filterable particles allowing their removal. See their report for details (Appendix H).

ICET, Inc.

ICET used their pilot unit to treat clinical laboratory wastewater for about six weeks at Brigham & Women's Hospital during the period January 1999 to March 1999, and to treat the research laboratory wastewater for about five weeks at the USDA Human Nutrition Research Center on Aging at Tufts University from May 1999 to June 1999 (see Appendix I). The unit operated nearly continuously at both sites according to the operating schedule specified in the scope of work for the project (8:00 a.m. to 4:00 p.m. on weekdays). The treatment scheme used was the same at both sites. It consisted of preoxidation of the influent wastewater using bleach followed by passage through 25 micron and 5 micron prefilters. Downstream of the prefilters, the wastewater passed through a bed of granular activated carbon and then three beds of ICET's media prior to being discharged.

The results for the testing at Brigham & Women's Hospital are summarized in Figure IX-3 and show that, with the exception of the last several days of the testing, the ICET system was capable of removing mercury to below 1 µg/l on a consistent basis. Near the end of the testing at this site, there were several spikes of mercury in the influent, one in excess of 300 µg/l, that resulted in effluent mercury levels > 1 µg/l. It should be noted, however, that even during this period, the removal efficiency of the system was greater than 72%, and was 96% at the time of the 300 µg/l spike.

Figure IX-4 summarizes the results for the testing at Tufts, and shows that the system consistently removed mercury to well below 1 µg/l, even during several periods of high mercury levels (up to 25 µg/l) in the influent. Prior to the testing at this site, the design of the float valve in the preoxidation tank was changed. The use of the new valve improved the operation of the pilot unit by allowing it to operate more continuously.

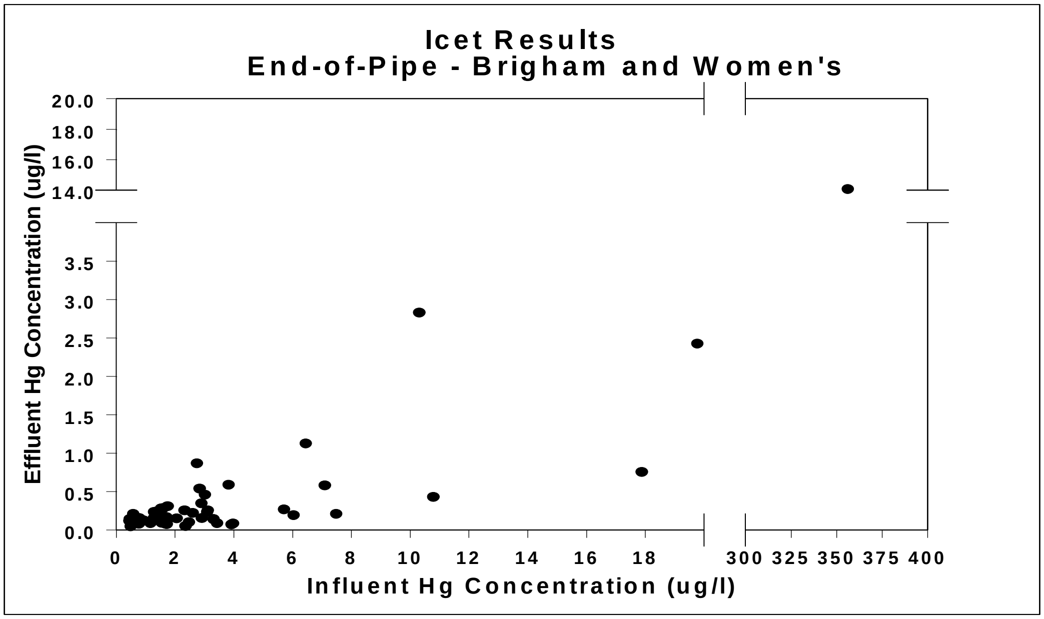

Figure IX-3: Mercury concentration of effluent plotted as function of influent mercury concentration for the pilot testing of the ICET system at Brigham and Women's Hospital. Note split scale on X and Y axes. See their report for details (Appendix I).

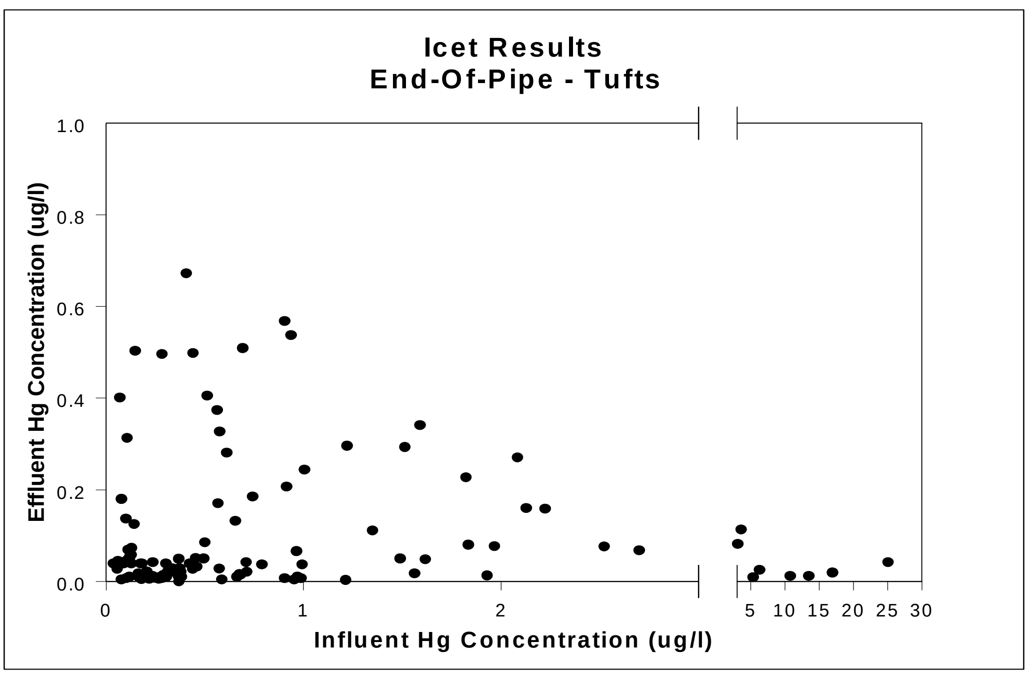

Figure IX-4: Mercury concentration of effluent plotted as function of influent mercury concentration for the pilot testing of the ICET system at Tufts. Note split scale on X axis and scale on Y axis. See their report for details (Appendix I).

Conclusions From The End-Of-Pipe Testing

Based on the solid foundation provided by the bench-scale feasibility testing conducted by MWRA, the STEP Demonstration Project developed a significant amount of additional information both on the characteristics of mercury in clinical and research laboratory wastewaters and the efficacy of several technologies to remove it. Specifically, the STEP Demonstration Project found that:

- All the vendors produced results indicating that mercury concentrations in clinical and research laboratory waste streams at or below the MWRA enforcement limit of 1 µg/l can be achieved. The DuBois/Prosys and ICET systems produced more consistent results while the ATA system had only intermittent success. None has achieved absolute success in removing mercury to below 1 µg/l at all times.

- A major barrier to achievement of consistent removal of mercury to concentrations below 1 µg/l is adequate preconditioning of the wastestream before application of the technologies employed by the vendors. Observations to date suggest that a significant fraction of the mercury in the wastestreams is in a colloidal and organically complexed form and not readily available to the media being employed by the vendors in the field tests. A greater understanding of the chemical form and kinetic lability of the mercury in these wastestreams would greatly enhance success in their treatment. The use of preconditioning (oxidation by ATA and ICET and coagulation by DuBois/Prosys) produced results that were greatly improved and more consistent.

- Concentrations of mercury in the influent to the pilot units, as well as in hourly samples taken over a 24-hour period from wastestreams at the three sites, indicated that discharges of wastewater with high mercury concentrations are still being released to the MWRA sewer system. Concentrations were frequently in the 2 to 20 µg/l range, but in a number of cases were in the high 10s and sometimes well in excess of 100 µg/l. This indicates that opportunities still exist to reduce mercury levels through the implementation of source controls and improved mercury management programs.

- A distinct temporal variability is observed in mercury concentration indicating that most of the mercury is released during normal working hours at the three facilities that were tested.

- While removal of mercury from wastestreams to levels below 1 µg/l is technically feasible, success will only be achieved through a comprehensive assessment of wastestream characteristics and implementation of appropriate technologies in both the preconditioning as well as mercury removal phases. Those vendors incorporating both aspects in their businesses will be those most likely to succeed and should have a distinctly competitive advantage.

X. Point-of-Use Prescreening Analyses

As noted earlier, point-of-use prescreening was used to help identify activities at each site where high levels of mercury were entering the waste collection system of the host site. Identifying such activities is significant because it offers the opportunity to eliminate the discharge of mercury through the use of non-mercury containing replacement materials. In the event such source reduction is not possible and treatment is still necessary, prescreening analyses can be beneficial by showing it may be possible to lower treatment costs by treating a small volume at the point-of-use rather than the entire wastewater stream using an end-of-pipe system.

The protocol used in the prescreening analyses is given in Appendix F. Briefly, the protocol requires the host site to first identify the source of any large volume flows entering the wastewater conveyance system. These flows are then tested and hopefully can be eliminated as a significant source of mercury flux in the system. If not, end-of-pipe removal technology would most likely have to be applied. Once this phase is completed, and the results indicate smaller volume flows as the source of mercury, additional screening at individual laboratory sinks is justified to identify major sources. The guiding philosophy here is that the most probable sources of mercury are those containing high concentrations of mercury discharged in low volumes to the waste collection system. The results of the point-of-use testing largely confirmed that belief.

Point-of-use testing was conducted at Newton-Wellesley and Tufts. It was not conducted at Brigham and Women's due to an inability to identify the large volume flow source(s) at that facility (see below).

<u>Newton-Wellesley Hospital</u>

Attempts to identify flows at Newton-Wellesley were unsuccessful in accounting for the total flow observed reaching the collection tanks. Because of the relatively small clinical laboratory facilities and small volume of wastewater generated per day, a prescreening exercise was conducted. Of the 29 samples collected over a six-day period, only 4 samples contained concentrations of mercury in excess of 1 µg/l and these ranged between 6.2 to 25.3 µg/l. Two samples contained concentrations between 0.1 and 1 µg/l and the concentration in the remainder of the samples were below 0.1 µg/l. The elevated concentrations were observed in samples from only two locations, the processing sink in the microbiology lab and one of the sinks in the chemistry lab, though the specific sources of the mercury were not identified. While consistent with the belief that most mercury discharges in such facilities are limited to a few low-flow discharges, a mass balance of mercury could not be achieved. This was probably due to an inability of the facility to accurately determine the sources of all flows entering the wastewater system and establish a mass balance for the wastewater being collected from the laboratories. The facility declined to participate in further exercises of this type due to the increased effort that would be required to identify these flows.

USDA Human Nutrition Research Center on Aging at Tufts University

A more extensive survey was conducted at the Tufts facility and involved the collection of all wastes discharged to the wastewater collection system over a five week period. The total volume discharged by the research laboratories at this facility was about 1,000 gallons/day and consistent with the expected usage of water at this facility. No large flows were identified at this site. Laboratory facilities were examined on four different floors with the goal of sampling each laboratory over a five day period. Results were consistent with those found at Newton-Wellesley in that of the 104 samples taken over the five weeks, only eight samples exceeded 1 µg/l. Of those, five were from one laboratory, two from a second, and one from a third. The two samples in the second laboratory were exceptional in that they both contained mg/l concentrations of mercury (3.0 and 7.7 mg/l, which correspond to 3,000 and 7,700 µg/l, respectively). One of the five samples in the first laboratory approached mg/l concentrations at 0.93 mg/l. Concentrations in the remaining five samples that exceeded 1 µg/l were much lower and ranged between 1.8 and 16.7 µg/l. An additional 23 samples contained mercury between 0.1 and 1.0 µg/l. Seventeen of these 23 samples were obtained from five of the 10 laboratories having concentrations in this range. Thus in this survey, the vast majority of mercury discharged to the wastewater collection system was from just 2 laboratories.

Further investigation of the potential sources of mercury focused on the use of an animal testing kit (Quantikine, R&D Systems, 614 McKinley Place NE Minneapolis, MN 55413) in one of the Tufts laboratories where the highest concentrations were observed. Analysis of the concentrations of mercury in the reagents in the kit revealed that 5 of the 9 reagents in the kit contained mercury $\geq$ 0.1 mg/l (100 µg/l). One contained 0.1 mg/l, three contained 50 mg/l and the fifth 500 mg/l. The use and subsequent discharge of less than 10 milliliters of the reagent with the highest concentration (Anti-human 1L-1β conjugate) on any day would alone produce mercury concentrations in excess of 1 µg/l in the approximate 1,000 gpd discharged at the Tufts facility. The reagent kit label did not indicate the presence of mercury in any of the reagents. Nor did requests for information from the manufacturer by Fred Yule, Physical Plant Manager, regarding the mercury content of the reagent kit meet with any success. The facility learned that no non-mercury containing alternatives to this test kit were available, and currently collects the wastes from the operations using this test kit for off-site disposal.

Brigham and Women’s Hospital

As indicated earlier, point-of-use testing at Brigham and Women’s was not conducted because initial attempts to identify all the sources of the large clinical laboratory wastestream (> 10,000 gpd) at this facility failed. Point-of-use testing at the Brigham and Women’s site might be useful but in the absence of knowledge of the source(s) of wastewater being discharged, may not be successful.

Summary

While no actual pilot testing of technologies in point-of-use applications was conducted due to limitations in resources, the conduct of the prescreening analysis led to several conclusions:

- The results of screening analyses can be used to achieve source reduction by avoiding the use of reagents containing high concentrations of mercury. Where such action cannot be accomplished for reasons such as the lack of availability of suitable substitute reagents and/or excessive cost, screening analyses can identify wastestreams that can be treated using the appropriate point-of-use capture technologies.

- None of the host sites had accurate quantitative assessments of the origin of their wastewater flows. Reliable wastewater flow and source data is critical to the success of point-of-use prescreening analyses.

- At both sites investigated using the screening techniques, the major sources of mercury were confined to two point-of-use locations. Concentrations in wastewater discharged from these sites were much higher than those in samples from other point-of-use locations. The vast majority of locations sampled at both Newton-Wellesley and Tufts contained low concentrations of mercury.

- The identification of these high point-of-use concentrations is consistent with the conclusion that most of the mercury being discharged at the host sites during this investigation was due to the active release of mercury from contemporary laboratory activities and not from desorption from the wastewater collection systems at these sites.

- The screening protocol used, when coupled with accurate high quality analysis capable of resolving differences at < 1 μg/l concentrations, can be used to achieve high resolution sampling. Even slightly elevated concentrations of mercury (>0.05 μg/l) in discharges can be readily detected and used to guide appropriate action.

XI. Summary Conclusions and Recommendations

- All of the end-of-pipe technologies tested were able to reduce mercury in the discharges treated to concentrations below, and in some cases well below, 1 µg/l. However, preconditioning of the wastestream was required in all cases to consistently obtain the low levels of mercury required in the treated wastewater. Successful use of end-of-pipe technologies will therefore require initial testing and fine-tuning of the process used to remove mercury, especially with respect to the preconditioning of the waste stream before treatment. However, considering the variable nature of the wastewater treated in this project, reaching acceptable levels of mercury in the treated effluent may not be as difficult as previously thought.

- Resolution of the pattern of mercury discharge using 24 hourly samples (composited from samples collected at 15 minute intervals) can be used to determine whether mercury release to the system reflects current active discharge of mercury or desorption of sorbed mercury from the surfaces of the wastewater conveyance system. The results of this sampling indicated that the mercury in the wastewater tested at the three host facilities in this project was the result of active discharge from laboratory activities.

- Screening of wastewater discharged at appropriate point-of-use locations can be used to assess whether implementation of source reduction or use of point-of-use or end-of-pipe technologies to reduce mercury concentrations is most appropriate. Prior quantification of the sources of water to an individual facility's wastewater collection system is critical to the success of such screening activities. Flux determinations (requiring the use of flow meters) rather than just concentrations in wastewater would be of greater use in identifying the magnitude and temporal variation in the discharge of mercury from facilities.

- The use of trace-metal clean techniques and performance-based measurement of mercury can produce consistently reliable results with detection limits less than 0.01 µg/l. Resolution afforded by such analyses allows sensitive identification of even minor discharges of mercury at a facility.

Appendix E[1]

Scope of Work for End-of-Pipe Pilot Testing

1. Please note that only Appendices E and F are contained in this electronic version of the report; Appendices A-D and G-I are available in hard copy only.

STEP/MWRA Mercury Workgroup

Scope Of Work
Field Testing of End of Pipe Mercury Removal Technologies

1. Background

The MWRA continues to encourage efforts that eliminate or minimize the amount of mercury discharged to the sewer system. To reduce the impacts resulting from clinical and research wastewater discharged to the sewer, the MWRA supported the formation of public/private stakeholder workgroups. The workgroups were asked to identify methods and techniques that would help hospitals achieve compliance with MWRA's end of pipe mercury standards. As a result, a source reduction program was developed that helped some hospitals eliminate mercury from wastewater by a factor of ten. Efforts to identify new products and additional source reductions strategies continues and is encouraged. Although source reduction achieved a significant decrease in mercury, a gap exists between what can now be achieved using source reduction methods and the MWRA's end of pipe standards. To fill this gap another MWRA workgroup (the Technology Identification Subgroup) solicited, selected, tested and evaluated mercury treatment and control technologies at the bench scale level. The results of this effort are described in MWRA's Technology Identification Subgroup Report.

Recently, the MWRA instituted interim mercury standards and stepped up enforcement efforts. These enforcement efforts are to be considered separate from the MWRA's participation in this pilot project and participating hospitals are not exempted from meeting any existing or newly issued compliance and enforcement requirements unless the MWRA provides written approval for actions/remedies undertaken by the host hospitals as part of this project. These agreements must be reached separately between the appropriate host hospital and the MWRA parties.

In 1994, with funding provided by the Massachusetts legislators, the STrategic Envirotechnology Partnership (STEP) was launched. STEP is a cooperative effort between the University of Massachusetts System (UMass), the Department of Economic Development (DED) and the Executive Office of Environmental Affairs (EOEA). STEP's mission is to promote the growth of new environmental and energy efficient technologies in Massachusetts. STEP services include: regulatory and business planning assistance, technology demonstration and verification and marketing assistance.

The STEP Mercury Removal Technology Demonstration Project will build on the existing bench scale work completed by the MWRA's workgroup. STEP will organize, implement and support a demonstration and evaluation of end of pipe technologies at three host sites. This effort is also designed to help vendors gain technology acceptance and stimulate commercialization efforts through the development and review of third party generated cost and performance data. STEP will produce a technical report that summarizes the results of the demonstration. It is believed that STEP's Mercury Removal Technology Demonstration Project Report, coupled with the MWRA's Source

Reduction Guidance Document and Pretreatment Guidance Manual, will provide hospitals with the necessary information to make better technology decisions and implement more cost-effective mercury reduction and control methods. The scope of work that follows will outline the specific data collection objectives; it is expected that all vendors will operate their systems according to this scope of work or the vendor's participation will end and the existing data collected will not be evaluated or reported by STEP as part of this project.

2. Objectives

Field testing of technologies is to demonstrate consistent performance of potential end of pipe pretreatment technologies in the removal of mercury to 1 μg/l or lower in clinical and/or research waste streams. Participating Vendors are expected to install and operate pilot-plant systems at three selected sites for a period of up to 8 weeks per site (pilot units will be tested in accordance with the operating schedule described in Section 4 below).

3. Host Sites

Site selection from those institutions indicating a desire to participate in the pilot testing phase of the project was made on the basis of a number of criteria including:

- Reported effluent concentrations of mercury
- Flow
- Access to waste stream
- Willingness of the host site to participate and facilitate the testing

Three different sites have been chosen. They are:

- **Newton-Wellesley Hospital - High concentration, low flow.** The hospital currently collects all Hg-contaminated wastewater from it's clinical laboratories and blood bank and has it removed from the site every 10 - 12 days for treatment elsewhere. Average monthly flow is reported to be about 4,500 gallons with Hg concentrations typically in the 20 - 30 μg/l range. Vendors will not be asked to spend a full eight weeks at this site. Processing of collected waste (volume of about 350 to 700 gallons) using at least three separate batch treatments over a three week period should suffice for evaluation purposes.

- **Brigham & Women's Hospital - Low concentration, high flow.** This site has a clinical laboratory waste stream with a flow of about 10,000 gpd and Hg concentrations ranging between <1 to 11 μg/l reported over the last year. Vendors will operate their treatment systems over an eight week period at this site.

- **USDA Human Nutrition Research Center on Aging at Tufts University - High concentration, low flow.** This site has a research laboratory waste stream with a flow of about 1,400 gpd and Hg concentrations ranging between 2 and 66 μg/l reported over the last year. Vendors will operate their treatment systems over a five week period at this site. This waste stream may have a high lipid content (oil and grease).

The responsibilities of the host sites are described in Attachment 1.

4. Technology Vendor Responsibilities

All Vendors participating in the pilot testing program shall comply with the following requirements:

- design, construct, install, and start-up end of pipe pilot unit(s); remove equipment at completion of testing at each of the three host sites (all costs borne by Vendor). All pilot units will be designed to treat wastewater at a minimum flowrate of 1.0 gpm, and will include calibrated waste water flowmeters with resettable totalizers.

- the design, installation, and operation of all pilot units will be in compliance with all applicable codes and regulations (e.g., the Massachusetts Uniform State Plumbing Code (248 CMR 2.0), and National Electrical Codes).

- furnish all equipment and supplies (e.g., media, chemicals, pumps, equalization and/or preconditioning tanks, plumbing, electrical panel) needed for the project, except for the following:
    - sample bottles (these will be provided by Dr. Gordon Wallace of the University of Massachusetts at Boston (UMass Boston)).
    - plumbing supplies for the connection between the source of wastewater used in the testing and the Vendors pilot unit(s) (these will be provided by the host sites, as well as the installation of the connection).

  The quantity of media and chemicals supplied will be sufficient to meet the operating schedule described below without interruption. A reasonable quantity of spare parts will be supplied with the pilot units(s), e.g., piping, fittings, valves, filters, etc.

- the pilot unit(s) will have all flow directions physically marked on the piping, and all valves will be labeled as to use and position (e.g., open and closed).

- operate and maintain all equipment for the duration of the project, including any troubleshooting or process modifications/optimization (sample acquisition will be performed by UMass Boston personnel). The Vendor will make every effort to use their own personnel for system operation and maintenance. However, if the Vendor can justify why they cannot supply their own personnel for the project, the Vendor will train others (e.g., STEP personnel) to operate and maintain the system (troubleshooting and process modifications/optimization remain the responsibility of the Vendor). The operating day will consist of 8 to 10 hours of continuous operation on weekdays during normal business hours; sample collection will only occur during these periods. Vendors may operate their units for more hours per day or on weekends and holidays at their discretion, but only if they will have personnel overseeing the operation of the pilot unit(s). The operation of pilot units at the various host sites will be in accordance with the following schedule (the overall schedule for the project is shown in Attachment 2):

- Brigham and Womens Hospital - continuous operation five days per week for a minimum of eight weeks.
- USDA Human Nutrition Research Center on Aging at Tufts University - continuous operation five days per week for a minimum of five weeks.
- Newton-Wellesley Hospital - pilot unit operation at this site will be on a batch-wise basis. Wastewater will be collected in the hospital's storage tanks (accumulating about 350 to 700 gallons of water), after which it is treated in the pilot units. The vendors will operate their pilot units so that it will take a minimum of two operating days to process an entire batch of wastewater. This process will be conducted for three consecutive weeks, resulting in a minimum of six operating days for each pilot unit tested. During the third week, the wastewater treated will include the discharge from the dishwasher in the clinical laboratories.

- maintain operating logs during the testing. The data to be collected should include the following information, as applicable, as well as any other information the Vendor feels is important to characterize the operation and maintenance of their system(s):

  – hourly and average daily wastewater flowrate through each pilot unit (gpm).

  – daily and total volume of wastewater treated in each pilot unit (gallons).

  – pH of wastewater before and after treatment.

  – chemical usage (preconditioning, regeneration, etc.)

  – type of chemicals used, daily usage, rate of addition (for continuous feed), time of addition and amount added (for batch feeding).

  – waste disposal - type of waste material generated, date and time of generation, amount generated, date of disposal.

- prepare an experimental plan for the project that addresses the elements described below. **Five** (5) copies of the completed plan will be submitted to STEP at the address given in Section 8 by **April 24, 1998** (the Vendor will not initiate any pilot testing until their experimental plan has been approved by STEP):

  – *project background* - description of the technology to be used, goals of the pilot testing.

  – *process description* - description of <u>all</u> equipment and instrumentation used in the pilot unit(s), including any equipment used for regenerating the system (if applicable). This section should include discussion of the entire mercury removal process relative to the need for (or advantages of) raw wastewater equalization, pH control, and biological sterilization and for final treated effluent neutralization (the discussion shall consider the allowed effluent pH range to be 5.5 to 10.5 Standard Units). The design of the pilot unit(s) must provide easily accessible sampling points to facilitate sampling of the influent to the system as well as the effluent from the system. The Vendors should consider sampling access to intermediate stages in multi-stage treatment designs if they feel sampling at those points would be useful. This section should also include equipment sizes and capacities, and should specify plumbing and utility requirements (electrical voltages and phases, compressed air, etc.) to facilitate installation assistance by the host sites. Lastly, this section should include a list of all waste materials that will be generated by the pilot testing that will require disposal, including the approximate quantity and appropriate disposal method for each waste material identified.

- *piping and instrumentation diagram (P&ID) of the unit(s) to be tested* - provide a detailed schematic of the pilot unit(s) showing the entire system from the point of connection with the host site to the discharge of treated wastewater. This diagram should show all sampling ports to be used during the testing.

- *experimental protocol* - describe how pilot unit(s) will be operated for the duration of testing at each host site, including the operation of any equipment used for regenerating the system (if applicable). This discussion should include a description of all operating parameters to be used in the testing (flowrates, temperature, residence times in preconditioning systems, etc.). Include copies of the operating log sheets to be used during the pilot testing. Describe potential process modifications that may be required to reach the goal of <1 μg/l of mercury in the effluent, and the possible schedule impacts (including how they can be minimized) if such changes are found to be needed.

- *sampling and analysis protocols* - sample acquisition and analysis will be performed by UMass Boston in accordance with the Quality Control and Sampling Schedule requirements described in Attachments 3 and 4.

- *project schedule* - for the period the Vendor is testing their pilot unit(s).

- *personnel involved in the project* - provide a list of all Vendor personnel participating in the testing, along with phone numbers for key contacts.

- work with Host Sites to make arrangements for the disposal of waste materials produced during the testing (e.g., waste chemicals, spent media). (Disposal costs borne by Host Sites)

- participate in project review meetings coordinated by STEP.

- prepare final report (based on the requirements of the Section 7 (Reports) below).

5. "Pre-Conditioning" Assessment

The testing is to include "systems" considerations for handling of interfering wastewater constituents and for optimization of the Mercury removal process. There will almost certainly be a need to "pre-condition" the waste stream before application of the individual technologies. It is expected that the Vendors will conduct initial bench testing of the individual waste streams before installation of the pilot system to minimize on site start-up delays.

6. Analytical Assistance

Depending on the efficiency of the technology being applied, mercury concentrations in the effluent of the pilot systems may be well below the 1 μg/l level. Commercial labs using routine EPA protocols often have difficulty in providing rigorous quantitative analyzes of samples at or below 1 μg/l. For that reason, collection and analysis of samples for Hg will be the responsibility of UMass Boston under the supervision of Dr. Gordon Wallace. Samples will be analyzed where possible using standard EPA protocols or slight modifications of that protocol (see Attachments 3 and 4). Expected detection limits will be in the single ng/l range, well below that usually achieved in routine application of EPA methodology. Occasionally it may be necessary or desirable for the Vendor to collect samples for analysis. Because of the low concentrations expected and the possibility of contamination each Vendor will be asked to identify designated personnel who will be trained by Dr. Wallace or an associate to collect the samples in a non-contaminating manner. All sample containers will be supplied by UMass Boston. Analyses, barring unforeseen circumstances, will be completed and the data reduced within 24 - 48 hours of collection. Details in the analytical protocol are provided below.

UMass Boston will conduct intensive analysis of the Hg concentration of the waste water effluents at each site to be treated in the pilot units before the field testing begins and make this data available to all Vendors participating in the project as well as the Host sites. This will allow some indication of the short-term variability in Hg concentrations at each site for Vendor use in designing pre-conditioning requirements. Furthermore, UMass Boston will provide analytical assistance in support of the bench-scale preliminary assessment of pre-conditioning needs to each Vendor. Intensive analytical support will also be provided during the initial start-up period to facilitate "fine-tuning" of the systems and for trouble-shooting purposes. The anticipated schedule of such activities is described in Attachment 4.

7. Reports

UMass Boston shall provide timely reports of the analysis of all samples and blanks to the Vendors and members of the STEP/MWRA workgroup. The reports shall identify any problems encountered, especially those that compromise or otherwise affect the quality of the data. The results of the analysis of blanks, replicate and split samples will be reported and clearly identified. All detailed records and documentation of sample collection, processing and analysis will be maintained until all parties agree that the project has been completed.

The Vendor shall issue a final report four weeks after the completion of testing and receipt of all data for each site. An electronic version of the final report is to be submitted as a word processing file (software requirements will be specified at a later date) to the STEP contact identified in Section 8 and should contain the following sections:

- Introduction/Background
- Bench Testing Results
- Methods used in Pilot Testing (including Treatment Protocol and Preconditioning Considerations)
- Pilot Test Results
- Full Scale System Considerations (Design, Operation, Siting, Discussion of economic feasibility of full scale systems*, etc.) - this section should include, based on the pilot test results, a description of equipment sizing and useful life, materials of construction, annual chemical requirements (preconditioning, regeneration, etc.), annual utility consumption, types and quantities (monthly basis) of wastes generated, waste disposal methods, and operating and maintenance requirements. For systems employing any sorbent media, additional information required includes: media life; contaminant loading capacity (e.g., Hg); replacement criteria; and loading capacity and useful life after repeated regeneration (if applicable).
- Discussion/Conclusions
- Appendices (including all analytical data and operating logs)
- The final reports will be reviewed by the STEP/MWRA workgroup within four weeks of receipt and comments forwarded to the Vendor. A revised final report will be submitted to STEP within two weeks of receipt of the reviewers comments.

** The project sponsors realize that a balance needs to be struck between vendor's potential desire to reserve proprietary sales and pricing information, and the public sector's need to make informed and responsible policy decisions based on the best possible information that can be made available. Since the economics of compliance with regulatory requirements are an important policy related issue, it needs to be addressed in the most open way possible by each vendor. At a minimum, based on overall pilot testing results and other considerations, an approximation of capital and operating costs for systems necessary to achieve a mercury discharge concentration of <1 µg/l for each test site should be developed (operating costs can be expressed in terms of $/gallon treated).*

8. STEP Contacts

The following individuals have been designated as STEP contacts:

1. For submittal of all project documents (e.g., experimental plans and final reports), and technical inquiries (excluding sampling and analysis issues):

Dr. John Raschko
Massachusetts Office of Technical Assistance
100 Cambridge Street, Room 2109
Boston, MA 02202
Phone: (617) 626-1060
Fax: (617) 626-1095
john.raschko@state.ma.us

2. For inquiries on sampling and analysis issues:

Dr. Gordon Wallace
University of Massachusetts at Boston
100 Morrissey Blvd.
Boston, MA 02125
Phone: (617) 287-7447
Fax: (617) 287-7474
wallace@umbsky.cc.umb.edu

Attachment 1

Host Site Responsibilities

All Host Sites participating in the pilot testing program shall comply with the following requirements:

- review experimental plans prepared by Vendors; recommend changes as necessary.

- assist Vendors with the installation of equipment, and equipment removal at completion of testing, including providing the connection between the source of wastewater used in the testing and the Vendor's pilot unit(s) (plumbing, electrical, etc.), as well as access to utilities (e.g., power, compressed air). The design, installation, and operation of this interconnection will be in compliance with all applicable codes and regulations (e.g., the Massachusetts Uniform State Plumbing Code, 248 CMR 2.0).

- manage the handling and disposal of waste materials produced during the testing (e.g., waste chemicals and spent media). (The vendors will provide assistance in these activities as described on page 4 under Process Description))

- cover the following costs associated with the testing:
  - utilities (power, water, etc.).
  - material and labor costs for installing the connection between the source of wastewater and the Vendors pilot unit(s).
  - handling and disposal of waste materials produced during the testing.

- participate in project review meetings coordinated by STEP.

- review final reports prepared by Vendors.

- report all activities related to its Sewer Use Discharge Permit and/or enforcement order to the MWRA during the course of the project.

Attachment 2

Schedule for End of Pipe Testing of Mercury Removal Technologies

Attachment 3

Quality Control

Procedural Blanks

Three procedural blanks (high purity water carried through the same sample handling procedure and processes as the samples), or a number equivalent to 10% of the number of samples analyzed, whichever is greater, will be run in each analytical run performed.

Field Blanks

To assess possible contamination in the sample collection and handling at the field sites, duplicate samples of high purity water will be transferred into sample bottles at each of the field sites during each day's sampling when discreet samples are collected. When composite samples are being collected over longer periods of time, two open containers, placed adjacent to the composite sample container, will be exposed for the same duration over which the composite sample is collected. The analysis of field blanks will be identical to that of the actual samples.

Sample Collection, Handling and Analysis

- Contamination from dust and residual vapor phase mercury, improper handling (plastic gloves should be used at all times), improperly cleaned labware and cross-contamination from samples with very high mercury concentrations are known sources of error. All labware will be Teflon or glass cleaned using standard procedures routinely used in the trace metal analytical facility at UMass Boston. This includes hot concentrated nitric acid cleaning of glassware or a series of ultrasonic-assisted cleaning steps for Teflon using 4M HNO_3, 4M HCl, and high purity de-ionized water. Glass and Teflon cleaned by these procedures are stored containing high purity 0.01M HNO_3 until used.

- Sample volumes of about 350 ml will be collected in the above glass or Teflon sample bottles. Glass bottles will generally be used for high concentration samples and Teflon bottles for samples anticipated having very low Hg concentrations (< 1 μg/l). Sample containers used for the collection of influent and effluent samples shall be kept separate whenever possible to minimize cross contamination. Preparation of sample bottles between use may be less rigorous than the initial cleaning described above depending on the nature and concentration of samples previously contained in the sample bottles. In some cases simple cleaning by rinsing with concentrated nitric acid may be sufficient. Ongoing evaluation of sample bottle cleaning will be verified by use of the sample bottles to prepare procedural blanks.

Attachment 3 (cont'd)

Quality Control

- Sample containers should be stored in plastic bags after cleaning and after filling with sample to minimize the possibility of contamination.

- Each sample bottle will be labeled for identification of contents, test run, treatment step, date, and qualifying comments.

- Samples will be preserved on site by addition of high purity concentrated nitric acid to reduce sample pH to less than 2. Verification that a pH < 2 is achieved shall be accomplished by measurement of a suitable sub-sample of the original sample at the site to assure proper acidification. The amount of acid used shall be recorded.

- At least three, or a number equal to no less than 10 % of the samples collected, will be collected in duplicate for analysis in each sampling event. Split samples, at a frequency and number to be mutually agreed upon by both UMass Boston and the Massachusetts Water Resources Authority (MWRA), shall be provided to the MWRA for analysis. Split samples shall be prepared in the field by collection of a sample in a two-liter Teflon or polycarbonate container, mixed, and then subdivided into sample bottles provided by the MWRA as well as those routinely used by UMass Boston. Splits of similarly prepared field blanks will also be provided to the MWRA.

- EPA method 245.1, or suitably documented modifications thereof, will be used for total mercury analysis at UMass Boston, using equipment and personnel capable of achieving an analytical detection limit of 0.002 µg/L. All samples with concentrations expected to be below 1 µg/l processed at UMass Boston will be prepared in a class 100 clean bench. EPA method 245.1 will be used for total mercury analysis at the MWRA, which is currently capable of achieving an analytical detection limit of 0.05 µg/l.

- Data reports for each analytical run shall include copies of all executed "chain-of-custody" sheets for samples not collected by UMass Boston. Each data report shall clearly identify sample contents, test run, treatment step, date, and qualifying comments. The results of all blank analyses and duplicate sample analyses will be provided.

Attachment 4

Sampling Schedule

The following are estimates of the sampling activity that UMass Boston expects to be able to supply in support of the project.

- **Characterization of effluent prior to initiation of field tests**. UMass Boston will sample the effluent waste stream at each Host site on an hourly basis over a twelve hour period. This sampling will allow us to provide the Vendors information on the temporal heterogeneity of the effluent before the actual conduct of the field tests. If time permits we will conduct this sampling over a consecutive two day period.

- **Sample Analysis in support of preliminary bench tests.** Analysis of samples generated in the conduct of preliminary bench-scale tests to optimize "pre-conditioning" of the effluent for Hg removal before actual conduct of the field tests will be supported. The Vendor must discuss sampling requirements with UMass Boston prior to the conduct of such tests to reach agreement on the number and timing of samples generated in support of this phase of the project.

- **Characterization of effluent for the first week of field tests**. UMass Boston will sample both influent and effluent streams over the following intervals following initiation of the field test by each vendor.

    - Hourly over a ten hour period for two consecutive days after initiation of the field testing. Sampling will begin at a time mutually agreed to by UMass Boston and the Vendor. If conditions warrant, extra sampling will be conducted as needed to "fine-tune" the treatment process.

    - Over the next two day period (days three and four) sampling of both the influent and effluent will be conducted every two hours over a ten hour period each day.

    - Sampling intensity over the next three days of operation (days five through seven) will be conducted at the beginning, middle and end of a ten hour period each day.

- **Characterization of effluent for weeks 2 through 8 of the field tests.** Pending results of the first weeks sampling efforts, sampling may be converted to collection of daily composite samples of the effluent for the duration of the field test. Sampling intensity may be increased to address Vendor needs in the case of anomalous results. Composite samplers are to be provided by the MWRA. If for some reason the samplers are not available, a grab sample of the effluent will be taken at the middle of each day the composite sampler is not available.

Attachment 4 (cont'd)

Sampling Schedule

- **Special characterization studies.** In some cases it may be advantageous to obtain additional information on the physical form of the mercury in the waste stream. Filtration through selective size membranes including the use of ultrafiltration techniques can be applied if necessary. Such studies will be conducted by mutual agreement between the Vendor and UMass Boston.

- **Conflict resolution.** In some instances the need for analytical services may temporarily exceed the capacity of the UMass Boston laboratory. When such conflicts arise priority will be given to support of the regular sampling program monitoring performance of ongoing field testing.

Appendix F

Protocol Used in Point-of-Use Prescreening Analyses

Sampling Strategy for Use in Screening Individual Laboratory Mercury Discharges

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Prescreening Protocol for Laboratory-Based Mercury Assessment

In order to maximize the potential success of individual point-of-use (POU) systems at laboratory sites where there is a significant discharge of Hg, we are recommending that host sites first go through a systematic screening process. This process consists of two stages. The first is to locate large volume discharges and then determine whether they contain significant concentrations of mercury. Doing so allows us to potentially eliminate these high flows from consideration and focus the application of POU systems to lower volume flows with higher concentrations of mercury. The second stage is to screen the laboratory wastes generated from each laboratory to identify the principal sources of mercury release and subsequently target these for the POU field testing. The processes for doing so are described as follows.

<u>Identification of large flows:</u>

Assuming the host site can identify large volume flows, we will sample these flows by taking single grab samples once a day over a five day period. If the mercury concentration in these flows is $\leq$ 0.2 ug/l, application of POU treatment technology will be focused on the remaining lower volume flows after further screening as described below.

<u>Screening of low volume flows:</u>

Each laboratory's waste will be screened by collecting all waste before disposal down the drain by subsampling all waste generated according to the following procedure.

All waste generated by each laboratory should be collected in one or more containers before disposal down the laboratory sink drains. A 20 l (or smaller depending on anticipated volume of disposal) plastic container such as a polyethylene carboy with a spigot at the bottom located at each sink would be a good choice of container. When the carboy is full, or at the end of each working day, whichever occurs first, the container is thoroughly mixed and a subsample of 300 ml is withdrawn and transferred to a sample bottle provided by the University of Massachusetts at Boston (UMB). The total volume collected, pH, and the date and time of collection is recorded each time in a sampling log and the remainder discarded down the sink. Checking the pH is important as a pH higher than 6 may lead to potentially significant Hg losses to the walls of the container.

This process is repeated as needed throughout the working day. Carboys and transfer glassware should be rinsed with 10% HCl and deionized water between uses to avoid carry over from one waste collection to the next. The carboy and any glassware used to make the transfer to the UMB should be previously rinsed in 10% HCl before first use. At the end of each day the collection containers are emptied, rinsed with 10 % HCl, deionized water, and stored closed overnight until the next use. This process is repeated each day for five consecutive working days. In the event that someone should bypass the collection process accidentally, the approximate volume, type and location of the disposal should be noted in the sampling log. A detailed stepwise procedure is attached.

We can generally collect and process no more than 20-40 samples a day. For purposes of illustration, assume laboratories have two sinks, each of which are anticipated to receive 40 l/d of laboratory waste. We would anticipate obtaining about 4 samples from each lab per day. Thus a maximum of 10 laboratories could be sampled over each 5 day period. This illustration suggests that each sink generates 40 l or about 10 gallons of waste per sink per day. If there were 100 sinks the total volume of wastewater volume discharged per day would be about 1000 gallons per day. The volume of water usually used to rinse wastes and clean laboratory apparatus probably exceeds this volume substantially but will not be monitored as part of this exercise. However it is our working hypothesis that the major mass of Hg eventually discharged to the sink will be contained in the concentrated wastes captured in the waste collection containers. Indeed we suspect that the actual volume of concentrated wastes collected and subsampled may be much less than 40 l/sink/day.

Caution Note:

Because waste waters from different laboratory operations are being combined in concentrated form, some consideration of the types of waste to be combined must be undertaken to avoid undesirable and potentially hazardous reactions (e.g., the combination of strong acids and bases or strong oxidizers with strong reductants). If the types of wastes likely to be combined in any one of the laboratory collection vessels might lead to such a situation, addition of 2-3 liters of deionized water to the container before concentrated wastes are added may be desirable. If so, the volume of deionized water added to the container should be entered into the log to permit correction of the data for any dilution made. If at all possible such dilution should be avoided but, as usual, laboratory safety takes precedence.

Laboratory Waste Collection Procedure for Hg Screening Purposes:

2. Establish the location and identity of all laboratory locations where laboratory waste is to be collected.
3. Provide sampling log for each location with columns facilitating the recording of:
   A. Total volume of waste collected and pH
   B. Volume and designation of subsample collected for Hg analysis
   C. Date and Time of collection of subsample
   D. Initial of laboratory personnel who recorded the data
   E. Any other pertinent comments useful in identifying any unusual events that might be relevant to influencing the Hg content of the sample.
4. Obtain a suitably sized polyethylene or other plastic container for collection of the waste at each site.
5. Clean the containers used for wastewater collection and subsequent transfer to the Hg sampling bottles using the following procedure.
   A. Wash with Micro or some other appropriate laboratory cleaner
   B. Rinse thoroughly with deionized water
   C. Rinse with 10% HCl
   D. Rinse thoroughly with deionized water
   E. Store covered until use
6. Label each waste collection container with a unique identifying label.
7. Collect all waste in designated container.
8. When container is full or at the end of the working day, whichever occurs first, thoroughly mix and check the pH. Mix again and withdraw 300 ml immediately after mixing for transfer to Hg sample bottle provided by UMB. Label the Hg subsample bottle.
9. Enter all data in 2. above in sample log.
10. Discard remainder of collected wastewater down the sink with normal rinsing of material down the drain.
11. Rinse the collection container with deionized water, 10 % HCl, and then deionized water again before resuming collection of laboratory waste.
12. Repeat collection procedure as needed.

Draft Sample Log Format

Waste Collection Sample Name	Date	Time	Volume (l)	Hg Subsample Bottle Name	Collector	Comments
Sink # 1 Microbiology Lab	6/5/98	10:30 AM	20.2 l	S1ML-1	Igor	pH . 5 Waste from protein analysis