Mercury Work Group
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Guidelines for the Preparation of a Request for Proposals for an In-Situ Pilot-Scale Feasibility Study of Mercury Removal Technology

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1.0 Introduction

This Request for Proposals (RFP) describes the requirements of an *in situ* pilot-scale feasibility study (FS) of an industrial wastewater treatment (IWWT) system specifically designed for mercury removal. The requirements for completing this FS are described more completely in the following sections, but the following summary is provided to introduce potentially interested vendors to this project.

The Massachusetts Water Resources Authority (MWRA) presently has an enforcement limit of 0.001 milligram (mg) per liter (L) or 1.0 parts per billion (ppb) for mercury discharged by permitted users to the MWRA collection system. This standard has proven to be difficult to achieve for many MWRA customers even when using aggressive pollution prevention methods coupled with traditional end-of-pipe IWWT systems. Therefore, pilot testing of mercury-removal IWWT systems is being requested by a consortium to investigate mercury removal technologies that have the potential to meet the stringent MWRA requirements.

The successful bidder (Vendor) will be required to provide all necessary IWWT equipment components, appurtenances, interconnections, resources and labor as may be required to install the equipment on-site, establish its performance as "steady state" over a period of two to three weeks, then operate the equipment for a period of not less than two (2) consecutive processing weeks (14 straight days) to demonstrate its ability to lower effluent mercury concentrations to minimum levels. The vendor will also be required to conduct sampling and analysis, as described below, necessary to obtain information on serial process operation removal efficiency under load. Following completion of the pilot testing, the vendor will be required to remove the equipment and restore the area to its original condition. The vendor will then be required to prepare a report summarizing field conditions and results obtained, interpret the analytical results of the testing, identifying any operational and maintenance difficulties encountered, and provide a detailed estimate of scale-up costs for the full-system design inclusive of design parameters, sustained system performance and costs (capital, installation and operating). A proposed report outline is presented in Section 4.0.

2.0 Wastewater Characterization / Design Basis

The mercury content of the wastewater produced by the facility undergoing pilot-testing will vary depending upon whether the facility includes clinical laboratories, research laboratories, incinerators and/or laundry facilities. Extensive studies have been conducted to help quantify the mercury concentrations present in wastewater streams discharged from each of these specific hospital facilities. A summary of the mercury data is presented in Table 1. A more complete summary of mercury and other pollutant parameters known to be present is presented in Table 2.

Table 1: Total Mercury Concentration in Typical Wastewater by Area

Area	Minimum(ug/L)	Maximum(ug/L)	Average(ug/L)
Clinical Laboratory	15.4	35.5	21.7
Research Laboratory	3	20.3	8.4
Incinerator	47.9	97.4	68.5
Laundry	4.3	43.5	20.6
Other	0.4	3.2	1.4

Table 2 - Typical Wastewater Composition by Facility Type

		Facility Type															
		Clinical Laboratory				***Research Laboratory***				***Incinerator Scrubber***				***Hospital Laundry***			
Analyte	**Units**	**No. of Samples**	**Min**	**Max**	**Avg**	**No. of Samples**	**Min**	**Max**	**Avg**	**No. of Samples**	**Min**	**Max**	**Avg**	**No. of Samples**	**Min**	**Max**	**Avg**
Mercury																	
Total Mercury	ug/L	5	15	36	22	6	3	20	8	6	48	97	69	6	4	44	21
Dissolved Mercury	ug/L	5	14	35	21	6	1	18	7	6	5	95	59	5	1	10	6
Heavy Metals																	
Antimony	ug/L	N.A.	-	-	-	N.A.				3	25	37	29	3	25	25	25
Dissolved Antimony	ug/L	N.A.	-	-	-	N.A.				3	25	26	25	3	25	25	25
Arsenic	ug/L	N.A.	-	-	-	3	25	26	26	N.A.	-	-	-	3	25	25	25
Dissolved Arsenic	ug/L	N.A.	-	-	-	3	25	27	26	N.A.	-	-	-	3	25	25	25
Beryllium	ug/L	N.A.	-	-	-	N.A.				N.A.	-	-	-	3	1	1	1
Dissolved Beryllium	ug/L	N.A.	-	-	-	N.A.				N.A.	-	-	-	3	1	1	1
Boron	ug/L	4	3,270	20,900	9,567	3	1,010	1,330	1,143	3	300	300	300	3	300	300	300
Dissolved Boron	ug/L	4	2,360	21,900	9,406	3	999	1,580	1,193	3	300	607	444	3	300	300	300
Cadmium	ug/L	N.A.	-	-	-	3	2	3	2	N.A.	-	-	-	3	2	2	2
Dissolved Cadmium	ug/L	N.A.	-	-	-	3	2	3	3	N.A.	-	-	-	3	2	2	2
Chromium	ug/L	3	6	25	16	3	2	3	2	3	2	5	4	3	2	4	2
Dissolved Chromium	ug/L	3	2	18	12	3	2	3	3	3	2	2	2	3	2	2	2
Hexavalent Chromium	ug/L	N.A.	-	-	-	N.A.				N.A.	-	-	-	3	11	11	11
Copper	ug/L	3	428	729	546	3	160	166	162	3	12	16	15	3	72	158	105
Dissolved Copper	ug/L	3	386	725	526	3	115	167	147	3	5	13	10	3	56	76	67
Lead	ug/L	3	12	19	16	3	12	23	16	3	15	20	17	3	16	145	61
Dissolved Lead	ug/L	3	12	19	17	3	10	18	13	3	3	13	8	3	5	21	10
Molybdenum	ug/L	N.A.	-	-	-	3	7	13	9	N.A.	-	-	-	3	7	7	7
Dissolved Molybdenum	ug/L	N.A.	-	-	-	3	7	16	10	N.A.	-	-	-	3	7	7	11
Nickel	ug/L	3	9	26	15	3	3	9	7	3	6	12	9	3	3	14	7
Dissolved Nickel	ug/L	3	6	21	13	3	3	7	4	3	3	8	5	3	3	5	4
Selenium	ug/L	N.A.	-	-	-	N.A.				N.A.	-	-	-	3	30	30	30
Dissolved Selenium	ug/L	N.A.	-	-	-	N.A.				N.A.	-	-	-	3	30	30	30
Silver	ug/L	3	22	87	57	3	72	329	194	N.A.	-	-	-	3	1	1	1
Dissolved Silver	ug/L	3	16	84	51	3	34	267	105	N.A.	-	-	-	3	1	1	1
Thallium	ug/L	N.A.	-	-	-	N.A.				3	35	37	36	3	35	35	35
Dissolved Thallium	ug/L	N.A.	-	-	-	N.A.				4	2	7	3	3	2	2	2
Zinc	ug/L	3	52	105	75	3	30	48	37	3	16	22	20	3	32	172	80
Dissolved Zinc	ug/L	3	54	114	76	3	34	42	38	3	16	24	19	3	25	50	37
Analyte	**Units**	**No. of Samples**	**Min**	**Max**	**Avg**	**No. of Samples**	**Min**	**Max**	**Avg**	**No. of Samples**	**Min**	**Max**	**Avg**	**No. of Samples**	**Min**	**Max**	**Avg**
Organics																	
Acetone	ug/L	N.A.	-	-	-	3	39.2	81	58.5	3	5	10	6.7	3	5.73	65	26.8
M.P.-Xylene	ug/L	8	5	14000	3400	4	10.6	136	53.5	N.A.	-	-	-	3	5	6	5.3
Chloroform	ug/L	8	5	131	54.4	4	10.4	460	215.8	3	5	8.6	6.5	3	2	17.9	11.9

O-Xylene	ug/L	8	5	3910	906	3	3.74	69.1	25.9	N.A.	-	-	-	2	5	5	5
Ethylbenzene	ug/L	8	5	3200	736	3	2	28.3	11	N.A.	-	-	-	N.A.	-	-	-
Bromodichloromethane	ug/L	N.A.	-	-	-	N.A.				N.A.	-	-	-	3	1.17	10	4.12
Bromofluorobenzene(s)	ug/L	N.A.	-	-	-	N.A.				N.A.	-	-	-	2	90	96	93
Styrene	ug/L	N.A.	-	-	-	N.A.				N.A.	-	-	-	3	5	10	6.7
Tetrachloroethene	ug/L	N.A.	-	-	-	N.A.				N.A.	-	-	-	3	2	5	4
Toluene	ug/L	N.A.	-	-	-	N.A.				N.A.	-	-	-	3	1	5	3.7
Indicator Parameters																	
Alkalinity	mg/L	3	162	340	247	3	33	51	42	3	40	89	61	3	48	108	81
Ammonia	mg/L	3	4	12	7	3	2	8	5	N.A.	-	-	-	3	2	2	2
BOD	mg/L	3	3,780	4,300	4,080	2	104	476	290	3	2	20	13	3	27	55	44
COD	mg/L	3	7,550	23,500	13,143	3	188	737	381	3	43	62	52	3	99	220	142
Fats, Oil, and Grease	mg/L	3	16	48	27	N.A.				N.A.	-	-	-	3	7	13	9
Flow	GPD	6	120	120	120	6	34,000	34,000	34,000	6	5,000	5,000	5,000	6	10,000	10,000	10,000
Petroleum Hydrocarbon	mg/L	3	0	2	1	3	0	0	0	3	0	2	1	3	0	2	1
pH	s.u.	6	7	9	8	6	4	9	8	6	5	9	7	6	7	10	8
TDS	mg/L	3	1,810	5,560	3,393	3	190	278	243	3	1,640	2,190	1,950	3	156	364	279
Temperature	Deg C	6	15	25	22	6	18	25	23	6	22	40	32	6	23	29	25
TSS	mg/L	3	10	28	16	3	6	10	8	3	1	1	1	3	10	34	21
Others																	
Cyanide	mg/L	3	0.01	0.34	0.12	N.A.				N.A.	-	-	-	3	0.01	0.036	0.022
Formaldehyde	mg/L	4	7.63	93.2	43.1	3	1.5	3.1	2.3	3	0.1	0.27	0.16	3	0.1	0.32	0.17
MBAS	mg/L	3	0.038	3.3	1.12	3	0.1	0.84	0.43	3	0.0758	0.125	0.097	3	0.63	0.69	0.65
Orthophosphorus	mg/L	3	46.6	84.8	66.1	3	0.25	0.89	0.48	N.A.	-	-	-	3	1.7	2.3	2.1
Phosphorus	ug/L	3	41700	85900	69600	3	595	1200	813	N.A.	-	-	-	3	5130	13200	10077
Sulfate	mg/L	3	15.8	28	22.6	3	13.8	18.7	16.7	3	22	24.2	22.9	3	7.79	8.38	8.04
Sulfide	mg/L	3	1	1.5	1.2	3	1	1.82	1.53	3	1	2.62	1.54	3	1	1.16	1.05
Total Chlorine Residual	mg/L	1	0.31	0.31	0.31	N.A.				3	0.02	0.05	0.03	N.A.	-	-	-

Notes:

- ug/L micrograms per liter, parts per billion
- N.A. Not Analyzed
- mg/l milligrams per liter, parts per million
- s.u. Standard Units
- GDP Gallons per Day

Table 2 - Typical Wastewater Composition by Facility Type - In HTML format

Table 2 - Typical Wastewater Composition by Facility Type - download as an Excel file.

The pilot system must not increase the concentration of any other regulated pollutants to levels greater than that allowed by the MWRA standards as are codified in Section 360 CMR 10.024, a

copy of which is included as Attachment A. *[Note: This section must be completed for the specific wastewater stream(s) to be included in the pilot test, and the entire RFP must be tailored specifically for your institution as noted in the cover letter accompanying this RFP. For technical assistance in how to modify this generic RFP into a site-specific document, you may wish to contact Bob Gingras, Vice President Wastewater Compliance Programs, EBI Consulting, 617-715-1816, bgingras@ebiconsultants.com.]*

3.0 Scope of Work

The scope of work has been separated into activities that are the responsibility of the institution (client) and those that are the responsibility of the vendor.

For this pilot study, the client agrees to:

- provide supervised access to the facility, as needed, to design and install the pilot test equipment.

- provide access to electricity, potable water, process water, compressed air, and other common industrial utilities present at the facility during the time of this pilot test. ***Note***: The vendor will be responsible for plumbing into the existing wastewater collection and discharge system(s) to obtain the side stream to be tested and for connecting into whichever of the other facility utility services as may be required to operate the pilot test equipment.

- provide disposal of all *aqueous* process wastewater residuals from testing. For this project, aqueous residuals are limited to the wastewater being subjected to the pilot testing. ***Note***: As specified in the vendor's responsibilities below, the vendor is responsible for disposal of all hazardous wastes, concentrated backflushing wastewaters (that may contain pollutants at concentrations in excess of MWRA discharge limits), excess treatment chemicals and any other chemical wastes generated by the pilot test system.

- provide access to historical wastewater monitoring records.

- conduct sampling and/or perform analysis of split/duplicate samples, provided by the vendor, at a Massachusetts certified contract analytical laboratory at the client's expense.

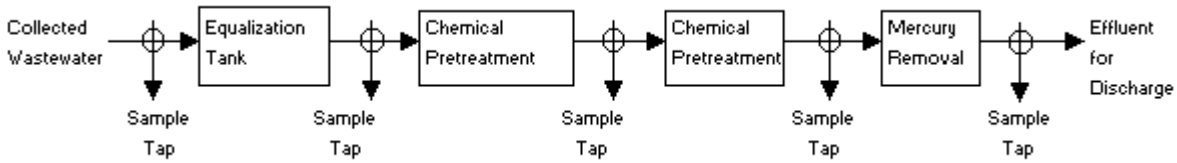
In the proposal responding to this RFP, bidders must:

- provide a detailed description of pilot testing equipment for technical feasibility review, including the system type (resin-based, adsorption, electrolytic, etc.) and materials of construction (PVC, stainless steel, etc.). The pilot testing equipment must include, at a minimum, an equalization vessel providing 30 minutes retention time, a prefilter system, of specified rating, and sampling ports in the effluent line from each of the unit operations included in the system. See Figure 1 for an illustration of where sampling ports would be required for a conceptual system.

- specify the utilities required for the pilot-scale pretreatment system.

- provide a detailed breakdown of the fixed price cost that will be incurred by the client in bidder's completion of the ***in situ*** pilot trial as described herein.

Figure 1. Illustration of Process Sampling Requirements



Note: Five sampling points would be required for this treatment system. The number of process samples required to be taken will vary based upon the design of the treatment system. The vendor must estimate the number of samples to be analyzed to demonstrate attainment of steady-state based upon anticipated wastewater characteristics and ease of treatment system startup.

- estimate the personnel and operating space requirements of the pilot-scale system.
- estimate the residuals to be generated by the pilot-scale system, and specify how they will be managed.
- specify the lead time required for delivery of the pilot test equipment.
- include references from three recent clients that have used the same technology proposed for use in the pilot study.

Subsequent to award, the vendor must:

- submit a draft pilot test workplan to the client for review and comment prior to equipment installation. This workplan must specify how the vendor proposes to remove the side stream to be tested from the facility piping. Following receipt of client's comments to the draft pilot test workplan, a final workplan must be submitted.
- design, deliver, furnish and install all equipment, labor and appurtenances required to establish, to steady state operation over a period of 2 to 3 weeks, an *in situ* system designed for mercury removal. The system must be provided with sample ports designed to allow sampling following each of the unit operations (as indicated in Figure 1) to allow determination of serial mercury removal rates throughout the system.
- plumb into the existing wastewater discharge line to obtain the side stream needed for the pilot testing.
- conduct sampling as per 40 CFR 136 requirements and submit flow proportional samples taken over a complete process day to an EPA-approved and MA DEP certified laboratory for analysis. Grab samples should be obtained to analyze for pollutants that cannot be accurately analyzed from composite samples. EPA method 245.1 capable of achieving a method detection limit (MDL) of 0.2 ppb (g/L) must be used for mercury analysis (see Attachment B for method specifications). The vendor is expected to have and follow a standard quality assurance/quality control (QA/QC) procedure during this sampling. The QA/QC procedure may be either an EPA protocol or a vendor-developed procedure, if it meets EPA minimum requirements. One set of flow proportional day-long composite samples must be collected from each sampling port present in the system on each day for the last ten (10) days of the pilot test period following attainment of steady-state. These samples shall be subsequently analyzed by the vendor for mercury content (at a

minimum). The client reserves the right to split a portion of each sample for his own analysis.

Subsequent to completion of field activities, the vendor must present his findings in a summary report which shall address each of the following issues in sufficient detail as may allow interpretation of the information by the client.

- specify the utilities required for the full-scale treatment system.
- prepare equipment lists and process schematics of the proposed full-scale treatment system.
- summarize the data obtained during the pilot testing and estimate the capital, installation and O&M costs for a full-scale system. See Section 4.0 below.
- estimate the spatial (envelope) requirements of the full-scale system.
- estimate the residuals to be generated by the full-scale system.
- conclude that the pilot test, as conducted, should be considered a success or provide recommended steps to improve the performance of the system tested. If the results indicate that the IWWT system tested is not likely to achieve the desired mercury concentrations, provide suggestions on how to enhance the existing equipment or suggest equipment to be used in place of the equipment tested.

4.0 Deliverables

As noted above in Section 3.0, the primary project deliverable will be the pilot test summary report. In this report, the vendor must provide: a succinct presentation and interpretation of the pilot test data; an estimate of the design and implementation (capital and installation) costs ($\pm$10 percent); and the expected performance of a full-size system. This report must include an appendix or attachment containing all raw mercury concentration data obtained during the pilot testing.

The report shall be of a format chosen by the vendor. The following format is suggested, as it may facilitate inclusion of all information required by this RFP:

1. Introduction/Background
2. Test Materials, Procedures, and Experimental Protocol
3. Pretreatment Considerations (including interferences)
4. Test Results (including total and dissolved removal rates for mercury and the 12 Priority Pollutant Metals)
5. Full Scale System Considerations (cost estimate for recommended full-scale treatment system to within $\pm$10 percent, O&M Requirements).
6. Discussion/Conclusions
7. Appendices (including detailed including information on reactor design information such as hydraulic retention time and loading rates, all analytical test results generated during the study, sampling QA/QC procedures followed, quantification and characterization of all residuals generated, and a summary of any operating and maintenance difficulties encountered during the pilot testing)

5.0 Cost and Schedule

Each bidder is required to include a firm (not-to-exceed) cost for this project and an anticipated schedule of execution based upon the information provided in this RFP. The not-to-exceed cost estimate must include allowances for the equipment, labor and appurtenances required to install and operate the system for a two week period after attaining steady state (2 to 3 weeks). The information contained in this RFP is expected to provide all of the information necessary to allow each bidder to prepare an accurate cost estimate and implementation schedule. However, the

person specified in the cover letter accompanying this RFP may be contacted if clarifying information is required.
This RFP should specify the length of time required to initiate installation of the pilot test equipment after authorization to proceed has been received, along with an estimate of how long it will take the wastewater pretreatment system to attain steady state. Bidders should provide an estimate of the time that will be required to debug and reach steady-state operations.
Pilot test equipment must be removed within two weeks following completion of the ten (10) day effluent testing period. The Report, described in Section 4.0, must be submitted to the client no later than six weeks following completion of the pilot test.

- Example Vendor Cover Letter
- Client Identification Sheet
- **Attachment A**
  MWRA Discharge Standards [360 CMR 10.024]
- **Attachment B**
  Analytical Method for Mercury in Wastewater
  [Method ARCI 245.1]

***** Example Vendor Cover Letter******

January 1, 1997

Mr. John Smith
Mercury-B-Gone, Inc.
Compliance Avenue
Boston, MA 01234

Re: ***Request for Proposal***

Dear Mr. Smith:

The Healing Hospital invites you to submit a proposal for the completion of an *in situ* mercury removal pilot study to be conducted on our regulated wastewater stream. As is detailed in the attached Request for Proposal (RFP), our institution discharges an effluent which may contain trace concentrations of mercury to the Massachusetts Water Resources Authority (MWRA). The MWRA has imposed an enforcement limit of 1.0 g/L on mercury discharges to their collection system, and we are investigating sources of industrial wastewater pretreatment (IWWT) systems that may be capable of attaining this limit. Your firm has been identified to us as one offering pretreatment systems potentially capable of achieving the MWRA's mercury discharge limit.

The vendor whose proposal is selected in response to this RFP will be responsible for designing, installing, operating and, eventually, removing an ***in situ*** pilot scale IWWT system within our facility to demonstrate its capacity for meeting or exceeding all MWRA effluent discharge requirements. As specified in the RFP, sampling points must be provided at and between each of the process units supplied within the pilot scale system to allow the determination of serial mercury removal efficiency through the system. A series of "compliance sampling" events will also need to be conducted once the system attains steady state. The Vendor's final product will be a report detailing field conditions and results obtained, then interpreting this information to full scale conditions inclusive of design parameters, sustained system performance and costs (capital, installation and operating).

I will serve as the facility contact and can be reached at (617) 123-4567 to answer any questions that you may have prior to the submission of a detailed quotation. Proposal are due within three weeks of the date of this cover letter. Note that the institution reserves the right, in its sole discretion, to reject any or all proposals received.

Thank you, in advance, for your participation on this important project.

Sincerely,

John Q. Public
Vice President, Facilities

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Form 1 - Client Identification Sheet

1.0 Name of Institution _____

2.0 Institution Point of Contact

Title _____

Address _____

Phone Number _____

Telefax Number _____

3.0 Date RFP Issued

Proposal Required By _____

Pilot Test Startup Desired By _____

Pilot Test Completion Desired By _____

Report Due Date _____

4.0 Facility Industrial Wastewater Discharge Rate

Average _____ gpd _____ gpm

Maximum _____ gpd _____ gpm

5.0 Effluent chemistry resembles the Clinical Incinerator Laundry Research stream chemistry as provided in Table 2 of this RFP (circle whichever is appropriate).

6.0 Facility Specific Issues _____

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Attachment A: 360 CMR 10 - MWRA Sewer Use Regulations
10.024: Specific Discharge Limitations/Local Limits

(1) Any discharge of the following materials is prohibited:

(a) Mercury;

(b) Polychlorinated Biphenyls (PCBs);

(c) Pesticides, including, but not limited to, Dieldrin, Chlordane, 1,1,1-Trichloro-2,2-bis(p-chlorophenyl)-ethane (4-4 DDT), Demeton, Endosulfan I, Endosulfan II, Endrin,Guthion, Heptachlor, Malathion, Methoxychlor, Mirex, Parathion, Acrolein, Aldrin,Tetrachlorodiphenylethane (TDE), 1,1-Dichloro-2,2-bis(p-chlorophenyl)ethene (DDE),Hexachlorocyclo- hexane, Lindane, Benzene-cis-hexachloride and benzene-trans-hexachloride (BHC), Hexachlorocyclopentadiene, and Toxaphene;

(d) In the Metropolitan Sewerage Service Area, Phenanthrene.

(2) No Person shall discharge, or cause or allow to be discharged, directly or indirectly into the Authority Sewerage System waters or Waste containing concentrations of the following materials in excess of the values shown in Table 1 below.

Table 1: Daily Maximum Limits

	Daily Maximum Limit mg/1 Metropolitan Sewerage Service Area
Antimony (total)	10.0
Arsenic (total)	0.5
Boron (total)	20.0
Cadmium (total)	0.1
Chlorinated Naphthalenes	0.8
Chromium (hexavalent)	0.5
Chromium (total)	1.0
Copper (total)	1.5
Cyanide (total)	0.5
Fluoranthene	1.5
Hexachlorobutadiene	3.0
Lead (total)	0.2
Nickel (total	) 1.0
Pentachlorophenol	0.05
Phenol	5.0
Phenolic Compounds (as defined herein)	0.5
Selenium (total)	5.0
Silver (total)	2.0
Toxic Organics	1.0

Each Toxic Organic not elsewhere limited in 360 CMR 10.000	
Total Toxic Organics	5.0
Trichloroethylene	0.07
Zinc (total)	1.0

	Daily Limit mg/1 Clinton Sewerage Service Area
Arsenic (total)	1.0
Cadmium (total)	0.5
Chromium (hexavalent)	1.0
Chromium (total)	5.0
Copper (total)	1.5
Cyanide (total)	1.0
Lead (total)	0.4
Nickel (total)	4.0
Selenium (total)	1.0
Silver (total)	2.0
Toxic Organics Each Toxic Organic not elsewhere limited in 360 CMR 10.000	1.0
Total Toxic Organics	5.0
Zinc (total)	5.0

(3) The Daily Maximum Limit shall be determined on the basis of a Composite Sample, except a Grab Sample or Samples may be used in the following circumstances:

(a) for a batch discharge, when the Grab Sample would be fairly representative of the batch as a whole;

(b) for pollutants that are not amenable to composite sampling of an industrial wastestream by use of an automatic wastewater sampler, such as hexavalent chromium and volatile organics; or,

(c) when the Authority determines it is not feasible or appropriate to take a Composite Sample, such as when the discharge is of too short duration for automatic composite sampling or the sampling location is inaccessible for an automatic wastewater sampler.

A Composite Sample shall be taken at preselected intervals throughout the time in a day during which the wastewater is discharged, except a shorter time period may be used if that shorter time period encompasses all or most of the wastewater discharge that day or if a shorter period is authorized by the Authority. A batch discharge may have a Composite Sample taken throughout the discharge of the batch. A flow proportioned Composite Sample shall be taken, except a time interval Composite Sample may be taken when the Authority determines it is not feasible or appropriate to take a flow proportioned Composite Sample. If a Grab Sample may appropriately be used, and more than one grab sample of a discharge is taken during the day, the Daily Maximum Limit shall be measured by either the mathematical average of the analytical results of the grab samples or the analytical result of a composite of the grab samples.

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ARCI 245.1
MERCURY IN LIQUID
Revised 12/1996 245.1-2

1.0 SCOPE AND APPLICATION:

1.1 Method 245.1 is a cold vapor atomic absorption procedure approved for determining the concentration of mercury (organic and inorganic) in mobility- procedure extracts, aqueous wastes, and ground waters. All samples must be subject to the appropriate dissolution step prior to analysis.

2.0 SUMMARY OF METHOD:

2.1 Prior to analysis, the liquid sample must be prepared according to the procedure discussed in this method.

2.2 Method 245.1, a cold vapor atomic absorption technique, is based on the absorption of radiation at 245 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical detection limit for the instrument used is 0.0002mg/L. TCLP analysis detection limits for this method are 0.002mg/L.

3.0 INTERFERENCES:

3.1 Potassium permanganate is added to eliminate possible interferences from sulfide.

3.2 Copper has also been reported to interfere; however, copper concentrations as high 10mg/L had no effect on recovery, of mercury from spiked samples.

4.0 APPARATUS AND MATERIALS

4.1 Mercury Analyzer, Perkin Elmer, FIMS (Flow Injection Mercury System): A single beam atomic-absorption spectrometer with integral flow injection sample-delivery system.

4.2 BOD bottles: 300ml BOD bottles rinsed with concentrated sulfuric acid and reagent grade water.

4.3 Water Bath: Water Bath capable of reaching 95 degrees Celsius.

5.0 REAGENTS:

5.1 ASTM Type II water (ASTD D1193): Water should be monitored for impurities.

5.2 Sulfuric Acid: Concentrated, Reagent Grade.

5.3 Nitric Acid: Concentrated, Reagent Grade of low mercury content.

5.4 Hydrochloric Acid: Concentrated, Reagent Grade.

5.6 3% Hydrochloric Acid: Add 30m/L concentrated Hydrochloric Acid to 500m/L of reagent grade water, dilute to 1 L.

5.7 1.1% Stannous Chloride in 3% Hydrochloric Acid: Dissolve 11 grams of Stannous Chloride in 500ml of reagent grade water, Add 30ml of Hydrochloric acid to the solution and dilute to 1L with reagent grade water.

5.8 Sodium Chloride-Hydroxylamine Hydrochloride Solution: Dissolve 60grams of NaCI and 60grams of Hydroxylamine hydrochloride in 400ml of reagent grade water, dilute to 500ml.

5.9 Potassium Permanganate: Dissolve 50grams of Potassium Permanganate in 900ml of reagent grade water, dilute to 1L.

5.10 Potassium Persulfate: Dissolve 50grams of potassium persulfate in 900ml of reagent grade water, dilute to I L.

5.11 Stock Mercury Standard: 1000ppm standard purchased.

5.12 Mercury Working Standard: Make successive dilutions of the stock mercury standard to obtain a working standard containing 0.1 g/ml with a total volume of 1Liter. The working standard is to be prepared daily.

6.0 SAMPLE PRESERVATION, AND HANDLING

6.1 Aqueous samples must be acidified to a pH<2 with HNO3. The suggested maximum holding time for these samples is 38 days in glass containers and 13 days in plastic containers.

6.2 Nonaqueous samples shall be refrigerated, when possible, and analyzed as soon as possible.

7.0 PROCEDURE

7.1 STANDARD PREPARATION: Prepare standard curve using the working mercury standard by transferring 0.0, 0.4, 1.0, 2.5, and 5.0 ml of standard into four BOD bottles. Bring bottles up to volume (100mL) with reagent grade water. Add to each bottle 8mL of 2:1 sulfuric-nitric acid, and 15 mL of potassium permanganate to each and let stand for 15 minutes. If purple color remains constant for 15 minutes, add 8mL of potassium persulfate to each bottle and heat for 2 hours in a water bath maintained at 95 degrees C. After 2 hours, remove the sample bottles from the water bath and allow them to cool. When they have cooled, add 6mL of hydroxylamine hydrochloride to each bottle and swirl until solution is decolorized. Prepare along with each run, a continuing calibration standard consisting of 2.0 mL of working standard from a different stock source as the curve. Every 10 samples analyzed is also to be accompanied by matrix spike, and spike

duplicate with the familiar matrix. These samples are to be prepared using the same standard source as the ccv standards.

7.2 SAMPLE PREPARATION: Transfer 100mL of sample or an aliquot diluted to 100mL to a 300mL BOD bottle. Add 8 mL of 2:1 sulfuric-nitric acid and swirl sample to mix. Add 15mL of potassium permanganate to each sample and allow to sit for 15 minutes. Sewage samples may require additional potassium permanganate. Ensure that the same amounts of potassium permanganate are added to standards and blanks, as all samples should receive the same amount of potassium permanganate until the purple color persists. Add 8mL of potassium persulfate and swirl sample bottles to mix. Samples should then be heated in a water bath for 2 minutes at 95 degrees Celsius. After 2 hours remove the samples from the water bath and allow

to cool. When cool, add 6mL of hydroxylamine hydrochloride and swirl until the solution has decolorized.

7.3 ANALYSIS: All samples and standards are to be read using the FIMS system. After allowing the FIMS to warm up (energy level > 40) analyze the standard calibration curve obtaining a graph of the curve. Read the CCV and depending on the percent recovery of the standard (80%-120%) accept the curve or rerun the curve if not within accepted parameters. Analyze samples, recording results in ug/L and converting to mg/L if needed. Detection limits for non-diluted samples is 0.0002mg/L. Detection Limits for TCLP metals is 0.002mg/L.

8.0 QUALITY CONTROL

8.1 Calibration Curves: Calibration curves are to be composed of a minimum of four standards as well as a method blank. All calibration curves are to be printed out and saved in the mercury data binder. Calibration is checked for every run through the use of the check standard analysis with the frequency 15%. If percent recovery for ICV is out of 20% range, rerun the calibration standards and remake the check standards from an independent preparation stock standard.

8.2 Dilution: Samples that are higher than the highest sample used to calibrate the instrument must be diluted and rerun with results falling into the range of the curve standards.

8.3 Blanks: One method blank is to be run with every batch of mercury samples analyzed to determine if contamination or any memory effects are occurring.

8.4 Matrix Spikes: Matrix spikes and spike dupes are to be run every I0 samples. Recovery range should not exceed 20%.

11.0 REFERENCES

11.1 Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82- 055, December 1982, Method 245.1.

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