Offsite


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November 6, 1996

Matt Johansen, DOE/AIP/POC
Dept. of Energy
Los Alamos Area Office
MS: A316
Los Alamos, NM 87545

Re: Submittal of results of mercury (Hg) sampling of waters and sediments collected in tributaries of the Rio Grande in White Rock Canyon, Los Alamos, New Mexico.

Dear Mr. Johansen:

The New Mexico Environment Department (NMED) Surface Water Quality Bureau (SWQB) conducted low-level Hg sampling of surface waters and sediments in tributaries of the Rio Grande during March and July of 1995. The results of this sampling was requested by representatives of ESH - 18 at the October 25, 1995, monthly technical meeting held in Santa Fe. Enclosed is the data requested along with the draft sampling protocols for the NMED low-level Hg project.

Please contact Ralph Ford-Schmid at 827-1536 if you have any questions regarding this matter.

Sincerely,

Stephen Yanicak

Steve Yanicak,
Point of Contact/LANL,
NMED, DOE OB

Enclosures

SY:rfs

cc: w/o enclosure
Ed Kelley, Director, NMED, WWMD

w/enclosure
Neil Weber, Chief, NMED, DOE OB
Steve Rae, LANL, ESH-18, MS K490

LANL-ES DATA - M.N.

4083

| Water Samples | | | | | | | |
|---|---|---|---|---|---|---|---|
| Sample Location | Latitude | Longitude | Date | Mercury ng/l | | | |
| | | | | A/ | B/ | C/ | mean |
| Bayo Canyon | 35 50 34 | 106 09 39 | 3/24/95 | 12.0 | 14.2 | 15.2 | 13.8 |
| Los Alamos Canyon | 35 50 34 | 106 09 39 | 3/24/95 | 0.0 | 0.0 | 0.0 | 0.0 |
| Mortandad Can. abv R. Grande | 35 49 44 | 106 10 19 | 7/18/95 | 4.3 | 3.2 | 2.3 | 3.3 |
| Spring 3A | 35 49 07 | 106 10 42 | 7/18/95 | 0.0 | 0.0 | 0.4* | 0.1* |
| Pajarito Can. abv R. Grande | 35 48 11 | 106 11 40 | 7/18/95 | 0.0 | 0.0 | 0.0 | 0.0 |
| Ancho Can. abv R. Grande | 35 46 17 | 106 13 11 | 7/18/95 | 0.8 | 0.9 | 1.6 | 1.1 |
| Chaquehui Can. | 35 45 49 | 106 14 31 | 7/19/95 | 1.4 | 1.1 | 0.6* | 1.0 |
| Frijoles Can. abv R. Grande | 35 45 16 | 106 21 12 | 7/19/95 | 0.0 | 0.0 | 0.0 | 0.0 |
| Los Alamos Canyon Above Rio Grande | 36 53 10 | 106 08 36 | 7/20/95 | 0.0 | 0.0 | 0.0 | 0.0 |
| Los Alamos Canyon Below SR 502 | 36 53 10 | 106 08 36 | 7/20/95 | 0.0 | 0.0 | 0.0 | 0.0 |
| Shaded station pairs are QA/QC replicates | | | | | | | |
| | | | | | | | |
| Sediment Hg ng/g | | | | | | | |
| Los Alamos Canyon Above Rio Grande sediments (0.341) | 36 53 10 | 106 08 36 | 7/20/95 | 5 | | | |
| Mortandad sediments (0.113 g) | 35 49 44 | 106 10 19 | 7/18/95 | 21 | | | |
| Ancho sediments (0.235 g) | 35 46 17 | 106 13 11 | 7/18/95 | 7 | | | |
| Chaquehui sediments (.260 g) | 35 45 49 | 106 14 31 | 7/19/95 | 6 | | | |

* Below 0.7 ng/L Method Detection Limit

# PROVISIONAL FIELD PROTOCOLS FOR MERCURY SAMPLING OF NATURAL WATERS AND SEDIMENTS IN NEW MEXICO

DRAFT

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# DRAFT SAMPLING PROTOCOLS FOR NMED LOW LEVEL MERCURY PROJECT

## 3.0 Requirements and limitations of method:

### 3.1 Requirements of Method

As a general rule, mercury (Hg) concentrations in natural waters are miniscule, often lower than one part per trillion (ppt). At this level of concentration there is considerable risk of sample contamination at all stages of field and laboratory sample handling. The protocols described here are designed to eliminate, to the greatest extent feasible, the contamination of samples during collection and transport while keeping cost to a minimum.

In an effort to simplify the ultra-clean protocols developed by Bloom (Bloom, N.S., 1994), EPA (EPA, 1994 draft), and others, which are prohibitively expensive, and require specialized laboratory support, this protocol relies almost exclusively on relatively inexpensive, disposable supplies. Because the mercury content, if any, of "off the shelf" materials cannot always be determined in advance, rigorous QA/QC controls must be followed to identify any source of contamination.

## 3.2 Limitations of Method

These protocols are designed for the sampling of a single parameter (Hg) only, and will require significant modification and verification before they can be applied to multiple parameter "ultra-clean" sampling regimes.

These protocols are designed for the sampling of natural waters and sediments only; the high levels of mercury found in some contaminated areas obviate the need for such rigorous sampling regimes.

## 4.0 Materials:

clear or white plastic storage boxes (2)

clear or white plastic trash bags (new materials only)

portable table

white "Tyvek" sleeve protectors

color free "zip-lock" bags (assorted sizes)

disposable, color free, 5ml plastic cups

color free "Whirl-pacs"

plastic wrap

adhesive tape

PETG plastic sterile media bottles

Teflon PFA 250ml bottle

Teflon TFE tape

powder-free clean room gloves

Eppendorf model 4810 adjustable volume pipetters

10ul-100ul and 500ul-2500ul

disposable tips for above

"Sharpie" laboratory marker

Fisher "Trace Metal Grade" concentrated HCl

EPS Chemicals "Merconwipes" Hg suppressant towlettes

stainless steel surgical scissors

individually wrapped sterile plastic sampling scoops

## 5.0 Field kit cleaning and preparation:

### 5.1 Basic decontamination

Before any sampling run, the two field boxes are emptied, washed, and decontaminated by wiping with "Merconwipes" towlettes by gloved technicians. One of the field boxes is designated "clean", the other, "dirty".

### 5.2 Decontamination and stocking: basic supplies

The "dirty" box is restocked with bags, extra factory-sealed sample bottles, gloves, and other non-clean items. All commercial containers ie, plastic wrap and ziplock boxes etc, are "Merconwiped" prior to placement in the field box.

## 5.3 Decontamination and stocking: clean supplies

The contents of the "clean" box are removed from their old trash bag and placed on a clean, white paper surface by gloved technicians. The old trash bag is removed from the "clean" box and all contents are removed from their old "zip-lock" bags and placed in new bags. Sample bottles and pipetter tips are double bagged. All old bags are discarded. Preservative acid is replenished at this time if necessary, and the cap re-wrapped with Teflon tape. As each item is re-bagged, it is placed in a new trash bag. When all "clean" items have been placed in the new trash bag, that bag is placed in the field "clean" box, which is then sealed.

Should a technician's gloves be contaminated by contact with an object that is not "clean", they must be changed or decontaminated with Merconwipe towlettes. Care must be taken to avoid contamination of materials that will come in direct contact with the sample by "Merconwipe" solution.

## 6.0 Water sampling field protocol

-On selection of an appropriate sampling location, the field boxes and table are taken as near to the water as is feasible. Care is taken to select a location providing safe access to large or fast moving rivers.

-The table is set up, and covered with new plastic wrap, taking

care not to touch the work surface.

-The technician who will do the actual collecting and preserving of the sample, designated "clean hands" (CH), then dons boots, if necessary, and a clean pair of Tyvek sleeves.

-The assisting technician, "dirty hands" (DH), then opens the dirty box, opens the glove bag and dons a pair of gloves. The Merconwipe container is placed on the table and a towlette removed and made available for use. The marking pen is removed from its bag and placed on the table.

-DH then gloves CH, making sure that the glove cuff overlaps the bottom of the Tyvek sleeve. The glove bag is then closed.

DH then opens the clean box, Merconwipes hands, and opens the trash bag containing sampling equipment and supplies.

-DH removes the double-bagged sample bottles and opens the outer bag.

-CH opens the inner bag, removes the sample containers, placing them on the table, and closes the inner bag.

-DH closes the outer bag, replaces it in the trash bag, closes the trash bag, and replaces the lid on the clean box.

-CH labels the sample containers with the station designation and the date/time group with the lab marker.

-CH then removes the factory seal from a sample bottle, moves into the water, faces into the current, and after submerging the bottle, removes the cap, allowing the bottle to fill completely, and replaces the cap while still submerged. This procedure is repeated until all replicates have been completed.

-DH then opens the clean box, Merconwipes hands, opens the trash

bag and removes those items needed for sample preservation and bottle sealing, placing the individual bags on one end of the table.

-CH is then re-gloved by DH.

-The bottle sealing tape is dumped from its bag by DH and laid flat on the table by CH.

-DH opens the bag of 5ml plastic cups and CH removes a nested set of two, securing them in the center hole of the tape roll for stability.

-DH opens the bag containing the field acid bottle.

-CH removes and unseals the acid bottle, decants an aliquot of acid into the plastic cup, and replaces the acid bottle cap. The cap will not be re-taped for the remainder of the sampling run.

-CH removes the pipetter from its bag, which is opened by DH.

-DH opens the outer bag of the pipetter tips.

-CH opens the inner bag, and with DH manipulating the tips from the outside, attaches a tip to the pipetter and closes the inner bag.

-After checking the pipetter volume setting, CH acidifies the samples to 1% $^{v}/_{v}$ HCl, opening and closing one bottle at a time.

-CH discards the pipette tip, excess acid and cup, and replaces the pipetter in its bag, which is held open and re-sealed by DH.

-CH tightens the sample bottle caps.

-CH then applies a minimum of two wraps of Teflon TFE tape to the caps of the sample bottles. The tape is cut by DH using "Merconwiped" surgical scissors.

-DH opens the bag containing the Whirlpacs. All replicates from

a given station are then double bagged by CH. (At this point the samples may be handled by DH.)

-All equipment and supplies not already secured in their bags are bagged and put back in the trash bag in the clean box, which is then closed for transport to the next station.

-The bags containing the sample bottles are placed in "Merconwiped" styrofoam shipping containers for transport to the analytical facility.

## 7.0 Sediment sampling

While mercury concentrations in water are typically in the very low parts per trillion range, concentrations in sediment exist in the parts per billion range. As the degree of sample contamination likely at this level of concentration is very low, elaborate sampling procedures as described above are unnecessary. Nevertheless, to minimize the chance of gross contamination of sediment samples, the sampling activities are conducted using a simplified "clean" technique.

### 7.1 Sediment sampling field protocol

-After all water samples have been secured in the shipping container, DH re-opens the bag of Whirlpacs.

-CH, still gloved (or re-gloved, if necessary) then withdraws two Whirlpacs and marks them, using the lab marker, with the appropriate station designation, date/time group, and any other pertinent information.

-DH then opens the bag containing the individually wrapped sampling scoops.

-CH withdraws a scoop, removes it from its packaging, opens a Whirlpac, and proceeds to the waterbody.

-CH uses the scoop to transfer portions of the sediment into the Whirlpac, taking care to select the finest particle size material available.

-When a sufficient quantity of material has been collected, the Whirlpac is sealed, placed in the second Whirlpac, and secured in the shipping container. The scoop is discarded.

## 8.0 Quality Control Requirements:

### 8.1 Replicate samples

As these protocols are experimental, rigorous quality control is critical. Existing laboratory QA/QC protocols for this method (245.7) call for multiple aliquot samples for each station. At a minimum, ten percent of the samples submitted will be blind replicate stations. These replicate aliquots are collected in alternating sequence concurrently with the actual sample aliquots, and are submitted as separate, reasonable stations with different collection times. The analyzing laboratory is notified of the replicate pairs only after results have been reported out.

## 8.2 Spiked replicates

In addition to the blind replicate stations, spiked replicates are prepared for approximately ten percent of all stations submitted. Stock solution for the spiked replicates is prepared at the analytical facility and aliquots of the same batch are used for both field and laboratory spike recovery analyses.

## 8.3 Reagent spikes

One set each of reagent blanks (3) and laboratory fortified reagent matrices (3) are submitted with each sample set.

Reagent blanks are prepared to determine mercury background in each new lot of preservative acid, pipetter tips, acid transfer cups and new field acid containers.

## 8.4 Laboratory air exposure blanks

Periodic 72 hr acidified air exposure blanks (3 open blanks and one sealed blank) will be prepared to monitor for airborne Hg in the NMED laboratory facility.

## 8.5 Sediment QA/QC

Quality control in sediment sampling and analysis is problematic at best. Because of sediment sorting due to streamflow energy

dynamics, and the propensity of mercury to concentrate in organically enriched and small particle size sediments, replicate samples drawn from the same general area may yield widely variable analytical results. Mechanical homogenization of sample aliquots is, conceivably, a way to minimize the inherent variability in sediment samples; in practice, however, uniform homogenization of sediments in a reliably clean manner is difficult. Development of a method to perform sample homogenization is a priority of this project, and will be accomplished as time allows.

### 8.5.1 Replicate sediment samples

Approximately ten percent of sediment samples submitted for analysis will be blind replicates. Replicates will be taken from the same immediate area, and will be as closely matched for organic content and particle size as is possible under field conditions. Replicates will be assigned realistic station designations and different date/time groups. The analytical facility will be informed of the replicates only after results have been reported.

### 8.5.2 Split samples

Where laboratory support of sufficient analytical resolution exists, replicate samples will be submitted to two independent laboratories. Samples will be collected concurrently from the

same depositional areas and handled identically until submission to the respective laboratories for analysis. Results of these analyses will be reviewed and transmitted to both analytical facilities.

## 9.0 Acknowledgements

The New Mexico Environment Department wishes to thank the EPA Environmental Monitoring Systems Laboratory in Cincinatti, OH, and its contractor, DynCorp/TAI for their considerable help and technical assistance in the devlopment and implementation of these protocols. Without their guidance and patience the NMED Fish Contaminant Study could not have gotten off the ground.

## 10.0 Literature Cited

Bloom, Nicolas S., 1994, Mercury as a case study of ultra-clean sample handling and storage in aquatic tracemetal research, in press: Environmental Laboratory.

EPA, 1994 (Draft), Method 1669 Sampling ambient water for determination of trace metals at EPA water quality criteria levels, USEPA Office of Water, Office of Science and Technology, Engineering and Analysis Division, 401 M St. SW, Washington, DC 20460.

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|---|---|---|
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| Co./Dept. NMED-SWQB | Co DynCorp / TAI | |
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SOP #: WA08-002
Revision #: 1
Date: 11/30/95

# Operation and Preventive Maintenance for the PSA HG Analyzer

## 1. Scope and Application

1.1 This SOP describes procedures for the routine operation and maintenance of the PSA Mercury (Hg) analyzing system, which includes a Merlin Hg Fluorescence system, Galahad Hg Concentration system, Cold Vapor Generator and Autosampler. This instrument analyzes ultra-low (0 to 100 pp trillion of Hg in water and soil samples. This system is fully supported by a PC compatible computer that includes instrument driven software.

## 2. Principle

2.1 A digested sample reacts with acidic stannous chloride which converts the mercury (II) present to mercury (0) vapor. The mercury vapor is removed from the solution via a stream of argon and detected by atomic fluorescence.

2.2 The recommended digestion procedure utilizes free bromine, and is capable of breaking down all commonly occurring organic mercury species to mercury (II).

2.3 An alternative procedure for the analysis of Hg would involve the use of potassium permanganate in lieu of potassium bromide\bromate. Prior to using this procedure, the draft method should be reviewed in anticipation of possible complications.

## 3. References

3.1 PS Analytical Merlin Plus Manual, Section 9.0 Cold Vapor Method Technique. Draft Method "Determination of Mercury by Automated Cold Vapor Atomic Fluorescence Spectrometry", Section 11.0.

3.2 Additional instructions are provided by the Questron Corp. entitled "Guide to Getting Started For The First Time", this can be found in the manual for the Merlin Plus System.

3.3 Associated SOPs - Hg Sample Digestion #WA09-001.

4. System Operation

4.1 Step 1: Turn on the equipment and allow at least 30 minutes for the system to stabilize. The instrument should be configured as shown in figure 1, of the Draft Method.

The laser printer can not be used as an output device for this system. As such the T-switch should always be on B, for the autosampler. In addition the auto data switch should be selected for B (printer output).

Step 2: When the computer is booted, at the prompt type HG, to run the Touchstone system for Hg analysis.

Step 3: Set up the sample analysis time parameters either manually or through the instrument software settings. These settings should include Delay time, Rise and Analysis time and Memory time. All parameter settings are outline in the Draft Method.

Step 4: Using the right arrow, move the highlighted cursor to Options, press Enter, with the bar at "Autosampler ON" press Enter to turn it off if you are not using the autosampler.

Step 5: Using the left arrow, move the highlighted cursor to Calibrate, press Enter, select New Curve and complete the questions according to the conditions outlined in the Draft Method.

Step 6: Inspect and replace tubing as needed or recommended. For a continuous flow, tubing lengths for the pump, autosampler and distance between the vapor generator & detector should be as short as possible.

Step 7: Run the samples according to the sequence outlined in the Draft Method. The concentration of the five calibration standards must be between 0 and 75 ppt (i.e. 0, 2.5, 5, 10, 20 & 75). After running the last calibration standard, the instrument will paused to show the calibration curve. Review it, and if within limits, press Escape, move the highlighted bar to Analyze, press Enter, select Batch, press Enter, follow instructions.

Step 8: If one or more calibration points are outside limits, press Escape, then move the bar to Calibrate, press Enter, select Amend Curve, press Enter, select RunEdit, press Enter, select the first point to be corrected by moving the pointing arrow with the UP or DOWN arrows, press Enter, select Add and

reanalyze. At the end of the analysis, if the result is better than the first one, press "I" for Ignore with the bar highlighting the undesired result. Press Enter, and, if needed, repeat the procedure with the next point(s),until the desire limits are obtained. Select Quit, press Enter, then proceed with the sample analyses as indicated in Step 7.

Step 9: If a sample concentration is higher than the most concentrated calibration standard, this sample must be diluted. Take an aliquote of the sample and complete to volume, in a new conical vial, with a digested blank solution (i.e. a solution made with ASTM Type II water + the digestion reagents). Mix by inverting once and reanalyze.

Step 10: After the last sample is analyzed, press Escape to stop the next cycle in the software (this will not stop the analyzer). Then check the results to see if all the QA/QC standards are within limits. To do this, move the highlighted bar to Results, press Enter, press "D" for Display. If the results are within limits, press "F3" to print the results, turn the detector off, reduce the Argon flow by 50% at the manometer output valve, and cycle the instrument about 5 times using water in all the lines.

Step 11: To get a hard copy of the calibration curve, after the results have been printed, move the highlighted bar to Calibrate, press Enter, move the bar to Curve Plot, press Enter, press "F3" to print the calibration curve.

Step 12: Release the tension from the tubing in the peristaltic pump, turned the instrument off and proceed to calculate results.

SOP #:WA08-001
Revision #: 1
Date: 11/30/95

# Hg Sample Digestion

## 1. Scope and Application

1.1 This SOP describes procedures for the digestion of aqueous and sediment samples for the determination of total mercury using the PSA Mercury (Hg) analyzing system, which includes a Merlin Hg Fluorescence system, Cold Vapor Generator, Galahad Hg Concentration system and Autosampler (last two are optional equipment). In addition, this system is fully supported by a PC compatible computer that includes instrument driven software.

## 2. Principle

2.1 A digested sample reacts with acidic stannous chloride which converts the mercury (II) present to mercury (0) vapor. The mercury vapor is removed from the solution via a stream of argon and detected by atomic fluorescence.

2.2 The recommended digestion procedure utilizes free bromine, and is capable of breaking down all commonly occurring organic mercury species to mercury (II).

2.3 An alternative procedure for the analysis of Hg would involve the use of potassium permanganate in lieu of potassium bromide\bromate. Prior to using this procedure, the draft method should be reviewed in anticipation of possible complications.

## 2. References

2.1 PS Analytical Merlin Plus Manual, Section 9.0 Cold Vapor Method Technique.

2.2 USEPA Draft Method 245.7 "Determination of Mercury by Automated Cold Vapor Atomic Fluorescence Spectrometry", Section 11.0.

2.3 Associated SOP - Operation and Preventive Maintenance for the PSA HG Analyzer #WA08-002.

## 3. Sample Digestion (Aqueous Samples)

3.1 Step 1: Label cap and tube of 50 mL conical polypropylene vials.

Step 2: Introduce the labeled tubes in mercury-free environmental chamber, where the digestion reagents are stored. Follow the protocol for sample introduction of your particular environmental chamber.

Step 3: Flush the reagent pumps no less than five times each before pumping the reagents into the conical vials.

Step 4: Remove the caps of the vials. Pump first the 1 mL of potassium bromate/bromide solution and then pump the 5 mL of 50% HCl solution. Cap all the vials, making sure not to exchange caps with vials.

Step 5: Remove the vials from the environmental chamber according to the chamber's protocol.

Step 6: Complete to volume with ASTM Type II water the vials labeled to contain the calibration curve standards, blanks and QA/QC standards.

Step 7: Complete to volume the rest of the vials using their corresponding sample.

Step 8: If the samples contain particulates, they must be filtered before being digested. Label lids and body of 115 mL 0.45 um Nylon filters. Use one filter/sampling site. Pre-wash the filters with 2 x 125 mL of 1% HCl solution. Discard the filtrates. Filter @ 50 mL of the sample and completely discard the filtrate. Filter enough sample to complete the analysis. Complete to volume the corresponding conical vial.

Step 9: Invert once all the vials, and allow the digestion to proceed for at least 30 minutes.

Step 10: After 30 minutes, add 30 uL of 12% hydroxylamine hydrochloride solution to stop the digestion. Invert once all the vials. The samples are ready to be analyzed.

4. <u>Sample Digestion (Sediment Samples)</u>

4.1 Step 1: Label cap and tube of 50 mL conical polypropylene vials.

Step 2: Introduce the labeled tubes in mercury-free environmental chamber, where the digestion reagents are stored. Follow the protocol for sample introduction of your particular environmental chamber.

Step 3: Flush the reagent pumps no less than five times each before pumping the reagents into the conical vials.

Step 4: Remove the caps of the vials. Pump first the 1 mL of potassium bromate/bromide solution and then pump the 5 mL of 50% HCl solution. Cap all the vials, making sure not to exchange caps with vials.

Step 5: Remove the vials from the environmental chamber

according to the chamber's protocol.

Step 6: Complete to volume with ASTM Type II water the vials labeled to contain the calibration curve standards, blanks and QA/QC standards.

Step 7: Tare labeled 50 mL conical vials.

Step 8: With a TEFLON coated spatula wiped with Merconwipe, rinsed with ASTM Type water and hand-shake dry, transfer into the vial a small amount of the sediment (0.2 to 0.5 g if it is moist, considerably less if it is dry). Weigh the vial again and calculate the weigh of the sample. If the sample is moist, tare a 115 mL 0.45 um filter and transfer @ 1 gm of sample, weigh the filter. Let it dry overnight under vacuum, no heat. Weigh the filter again. Calculate the percentage of dry sediment in the sample.

Step 9: Pour the digestion mixture contained in one of the digestion vials into the vial with the sample. Swirl the vial to ensure contact of the sample with the solution.

Step 10: Complete to volume with ASTM Type II water. Mix by inverting once.

Step 11: After 30 minutes, add 30 uL of 12% hydroxylamine hydrochloride solution to stop the digestion. Invert once all the vials. The samples are ready to be analyzed.

Step 12: Be careful not to aspirate any sediments when analyzing the samples. It can be accomplished by letting the samples stand undisturbed until is time to be analyzed.

SOP #:WA08-004
Revision #: 1
Date: 11/30/95

# Hg Data Reduction

## 1. Scope and Application

1.1 This SOP describes procedures for the data reduction after analysis of aqueous and sediment samples for the determination of total mercury using the PSA Mercury (Hg) analyzing system, which includes a Merlin Hg Fluorescence system, Cold Vapor Generator, Galahad Hg Concentration system and Autosampler (last two are optional equipment). In addition, this system is fully supported by a PC compatible computer that includes instrument driven software.

## 2. Principle

2.1 A digested sample reacts with acidic stannous chloride which converts the mercury (II) present to mercury (0) vapor. The mercury vapor is removed from the solution via a stream of argon and detected by atomic fluorescence.

2.2 The recommended digestion procedure utilizes free bromine, and is capable of breaking down all commonly occurring organic mercury species to mercury (II).

2.3 An alternative procedure for the analysis of Hg would involve the use of potassium permanganate in lieu of potassium bromide\bromate. Prior to using this procedure, the draft method should be reviewed in anticipation of possible complications.

## 2. References

2.1 PS Analytical Merlin Plus Manual, Section 9.0 Cold Vapor Method Technique.

2.2 USEPA Draft Method 245.7 "Determination of Mercury by Automated Cold Vapor Atomic Fluorescence Spectrometry", Section 11.0.

2.3 Associated SOP - Operation and Preventive Maintenance for the PSA HG Analyzer #WA08-002.

2.4 Associated SOP - Hg Sample Digestion #WA08-001.

## 3. Data Reduction (Aqueous Samples)

3.1 Step 1: After finishing with the sample analysis, print out the report.

Step 2: All the samples that were prepared according to the sample digestion SOP must be corrected by multiplying the reported result (in ppt) by the correction factor (50/44).

Step 3: All the samples that were diluted because of having a high Hg content, should be corrected by multiplying by their appropriate dilution factor.

4. Data Reduction (Sediment Samples)

4.1 Step 1: After finishing with the sample analysis, print out the report.

Step 2: All the samples that were diluted because of having a high Hg content, should be corrected by multiplying by their appropriate dilution factor. This is to determine the correct amount of sample analyzed.

Step 3: Calculate the Hg concentration using the following procedure: (1) calculate the amount of sample in each sample analyzed. (2) Knowing the ppt of Hg in the sample, calculate the amount of Hg in the 50 mL of sample; do this by dividing the ppt of Hg result by 20. (3) Then divide this result by the sample weight to obtain the results in (ng Hg/g Sediment) or ppb. Put into an equation:

$$\text{ppb Hg} = \underbrace{(\text{ng Hg}/50\text{ mL})}_{(A)}/\underbrace{(\text{sample dry weight})}_{(B)}$$

(A) = (reported ppt Hg result)/20

(B) = wet sample weight x (100 - % moisture)