Analysis of Methylmercury in Suspended Matter by Distillation, Gas Chromatography Separation, and Speciated Isotope Dilution Mass Spectrometry

Ogorek, J.

Scope and Application

The following standard operating procedure (SOP) describes the (1) preparation of particulate matter (filtrand) on quartz fiber filters (QFF) by distillation, and (2) subsequent analysis for methylmercury (MeHg). Analysis is facilitated by coupling the Brooks-Rand "MERX" Automated Methylmercury Analytical System with the Elan Inductively Coupled Plasma-Mass Spectrometer (ICPMS). Quantification of MeHg is by isotope dilution. This method is used by the Wisconsin Mercury Research Laboratory (WMRL) to determine MeHg concentrations in particulate matter (filtrand retained on 47mm QFF's) from water samples. Quality assurance and control protocols are employed throughout sample distillation and analysis, including: laboratory practices to prevent sample contamination, analysis of certified reference materials (CRM) or MeHg method spikes, and method blanks.

Distillation is required to disassociate MeHg from particulate matter and eliminate matrix interferences. Particulate matter captured of QFF are placed in Teflon distillation vials, to which 50 ml of reagent water, isotopically enriched MeHg spike, and 2 ml of acidified KCl/$CuSO_4$ solution are added. Distillation vials are heated in an aluminum block (121º C), and purged with argon gas. The resulting vapor is carried to and condensed in chilled Teflon receiving vials. Distillation is stopped when approximately 25% of the water remains in the distillation vessels.

The MERX consists of three interconnected modules (autosampler, purge and trap module, GC/Pyrolytic module), and is coupled to the ICPMS for detection. In 42 ml glass vials, distillate is buffered to a pH of 4.5 – 5.0 and treated with Sodium Tetraethylborate (NaTEB), resulting in ethylation of oxidized mercury species. These volatile ethylated species (as well as elemental mercury) are stripped from the liquid phase with argon gas, retained on Tenex traps, desorbed back into the sample stream, and separated with a gas chromatography column. Each ethylated mercury species is released from the column *en masse* into the sample stream, thermally oxidized to elemental mercury, and introduced to the ICPMS. Elemental mercury in the sample stream is ionized by a radio-frequency generated plasma field, and detected using Speciated Isotope Dilution Mass Spectrometry (SIDMS).

This document is intended as an SOP designed to guide the user through MeHg analysis specific to the WMRL. Additional details helpful to the analyst can also be found following the SOP (Appendices 1-3), and is intended as a quick reference bench guide. However, the analyst is required to be familiar with the detailed SOP as well as the original user's manuals provided by Brooks-Rand and Elan which will be referred to when appropriate.

Safety Concerns

Multiple safety concerns are present in the conduct of this method. Persons involved must have read, understood, and signed the Chemical Hygiene Plan for

the WMRL prior to potential exposure to any chemicals. Although MeHg is an extremely toxic organic metal, concentrations encountered in samples and working standards on this instrument are generally low. However, caution should still be exercised to limit chronic exposure during daily operations. Concentrated stock solutions containing elevated MeHg levels are occasionally encountered, and should only be handled by experienced lab personnel. Reagents used in this method include strong acid and an organometallic ethylating compound. The analyst must have a thorough understanding of these chemicals, including their required safety protocols, prior to their use. More detailed information is included for each reagent later in this SOP, and additional information can be found in the attached material data safety sheet. During analysis the automated sample introduction system may begin moving without warning and presents a mechanical hazard. Finally, equipment and sample in excess of 100º C will be encountered and presents a burn hazard.

Distillation Procedure

A typical distillation contains up to 34 particulate matter samples, four blanks, and two CRM's (or alternatively two MeHg method spikes). In small sample sets four method blanks, and two CRM's should still be included in each run.

1. Throughout the distillation process, it is important for the analyst to develop and maintain a structured and organized system. Each distillation and receiving vessel has a unique identification code etched onto the wall. The corresponding vials must be appropriately linked to each other, as well as back to the original sample. Good record keeping must occur not only throughout the distillation process, but must be similarly well documented in the Excel data sheet (See Appendix 1 for more details). A template of the Excel data sheet can be found in the "ID TRACER TEMPLATE" folder (HG4→ hg4data→Isotope methyl data).

2. Before beginning sample setup, turn on the aluminum block heater so that it can reach temperature while samples are being prepared.

3. Arrange an adequate number of clean Teflon distillation and receiving vials into four wire racks.

4. Carefully place a thawed QFF sample filter into each distillation vial, being careful not to lose any filtrand. For method blanks, place a clean QFF into the distillation vial. Weigh approximately 50 mg of CRM into two distillation vials; replace the CRM with two MeHg method spikes of 100 ul working solution if appropriate CRM is not available. Pipette 50 ml of reagent water and 2 ml of acidified $CuSO_4$ solution into each of the distillation vials. Be sure that you record the sample ID, distillation vial ID, distillation vial tare weight, and CRM mass/method spike volume into the appropriate places in the Excel data sheet.

5. Add 50 µl of the isotopically enriched ($Me^{199}Hg$) working solution. If working with isotopically enriched samples, be sure that the spiked isotope fraction has not been amended in the sample.

6. Fit each distillation vial with a combined distillation cap/transfer tube assembly corresponding to the block position to be occupied by that vial (each cap has been engraved with a number between 1 and 40).

7. To each of the receiving vials, add 10 ml of reagent water. Be sure that you record the receiving vial ID and receiving vial tare weight.

8. Fit each receiving vial with a distillation cap corresponding to the rack position to be occupied by that vial (each cap has been engraved with a number between 1 and 40).

9. Place the distillation vials in the bore holes of the preheated (approximately 120º C) aluminum block. Attach an argon gas line to each of the distillation caps and ensure that gas flows through the sample.

10. In an ordered manner, thread the transfer tubes through the Teflon ports in the side of the cooler.

11. In the cooler, attach each transfer tube to the corresponding receiving vial. Ensure that gas flows in the reagent water of the receiving vial.

12. Throughout the distillation, check the temperature of the heating block often. Adjust to maintain a temperature of 121º C (± 5).

13. Check the distillation vials regularly. Samples are finished distilling when approximately 25% of the original reagent water is left.

14. Once a sample is finished, disconnect the transfer tube from the receiving vial and the gas line from the distillation cap. Cover the gas inlet port of the distillation cap quickly with a gloved finger to keep the sample in the vial from discharging.

15. Thoroughly rinse the combined distillation cap/transfer tube assembly with copious amounts of reagent water. Place the caps in the laminar flow hood to dry.

16. Once the entire distillation is finished, remove the receiving vials from the cooler and remove the distillation caps. Record the mass of the receiving vial in the Excel data sheet and attach a standard cap.

17. Distillates should be analyzed within 48 hours of distillation.

Brooks-Rand Operation

Start Up

1. Check that all modules of the instrument have power and the Argon gas supply is turned on. Empty the waste receptacle located on the floor.

2. If necessary, open the Mercury Guru4 software with the shortcut on the desktop.

3. Open the analytical file BR549.brd (found on HG4) and save the file as data (from the "File" dropdown menu).

4. From the "Instrument" dropdown menu, select "Connect", prompting a popup window displaying three communication ports. Select the appropriate ports (CVAFS = ICPMS, Purge and Trap = COM5, and Autosampler = COM4) and click "Accept". The communication status at the top of the screen will turn green indicating connection with each module.

Preparation of Vials for Analysis

The MERX instrument is designed to operate on a specific mixture of reagents that are prepared in sealed 42 ml amber glass vials. The autosampler holds three removable 24 vial sample racks, each consisting of 3 rows of 8 vials. Vial number one is the upper right position, with vial position descending from right to left-then top to bottom. Once prepared, the vials are sealed to the atmosphere and remain viable for analysis up to 48 hours. A typical analytical run is shown in Appendix 2.

1. Place the clean vials in the sample rack and add approximately 40 ml of sample distillate or reagent water (for instrument blanks and MeHg standard additions) to each vial.

2. Record the identity of the sample, analytical vial number, date, and standard information on a bench sheet (Appendix 3).

3. Adjust the pH of the mixture to 4.5 – 5.0 by adding 200 μL of the sodium acetate buffer reagent to every vial.

4. Add 50 μL of 1% NaTEB to every vial.

    a. NaTEB is an unstable reagent and must always remain at freezing temperatures to slow degradation. Begin thawing several minutes before use but always make sure that some frozen NaTEB remains

in the vial. Promptly cap and return the vial of NaTEB to the freezer after use.

b. NaTEB is toxic and spontaneously combustible in air. Only open vials and dispense NaTEB under a fume hood. Add NaTEB directly to the sample mixture (not to the glass surface inside the vial) to reduce volatilization.

5. Fill the vials with reagent water using a squirt bottle until a reverse meniscus forms (convex water surface). Seal the vial carefully (without headspace or spilling) with a new clean cap and septa assembly. Vigorously shake the vial, check for any air bubbles in the vial, and refill if necessary.

6. Place the full rack on the autosampler tray, making sure that the rack is properly positioned and oriented.

Elan ICPMS Operation

Start up (see figures below for reference)

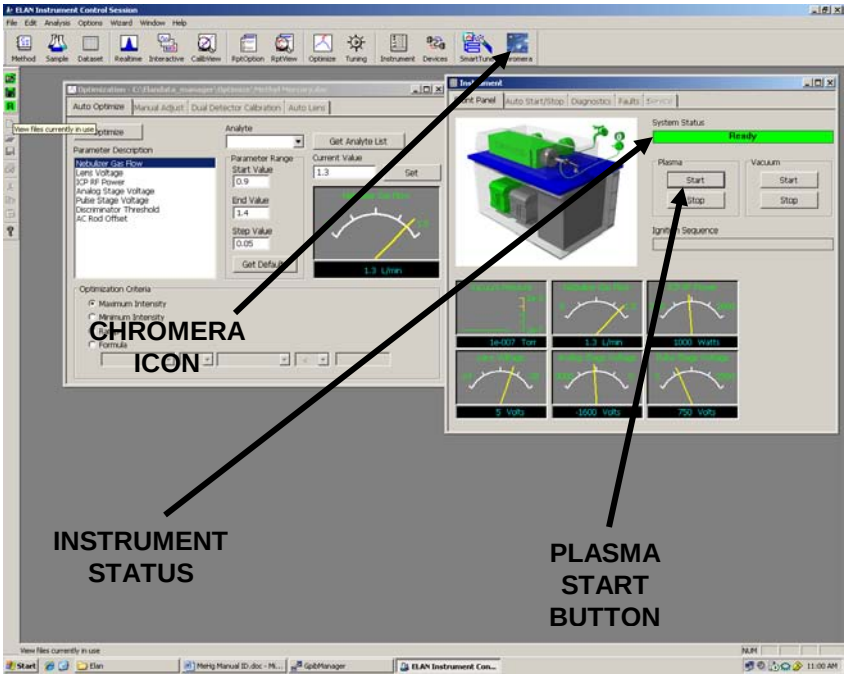
1. Start the Elan software from the desktop shortcut.

2. Turn on the coolant pump with the switch near the door and check that the external mass flow controller displays 30.0 ml/minute.

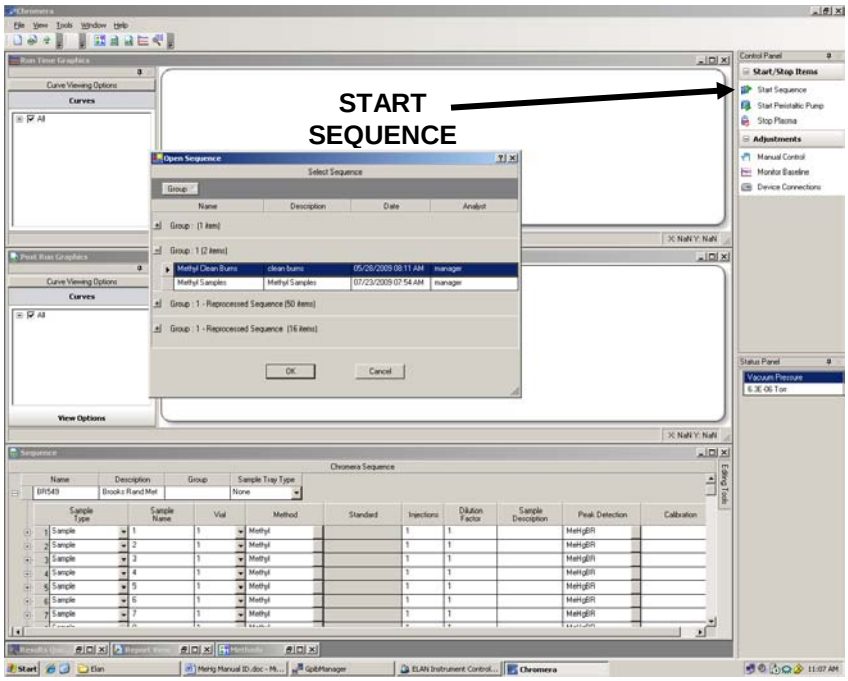
3. Click the "**R"** button in the upper left corner of the workspace and verify that the Methyl Mercury**.**dac file has been loaded.

4. Click the "instrument" icon to display the ICPMS status.

5. When the status of the instrument displays "Ready" click the "Start" button to initiate the plasma field.



6. Launch the Chromera software by clicking the icon at the far right of the toolbar.

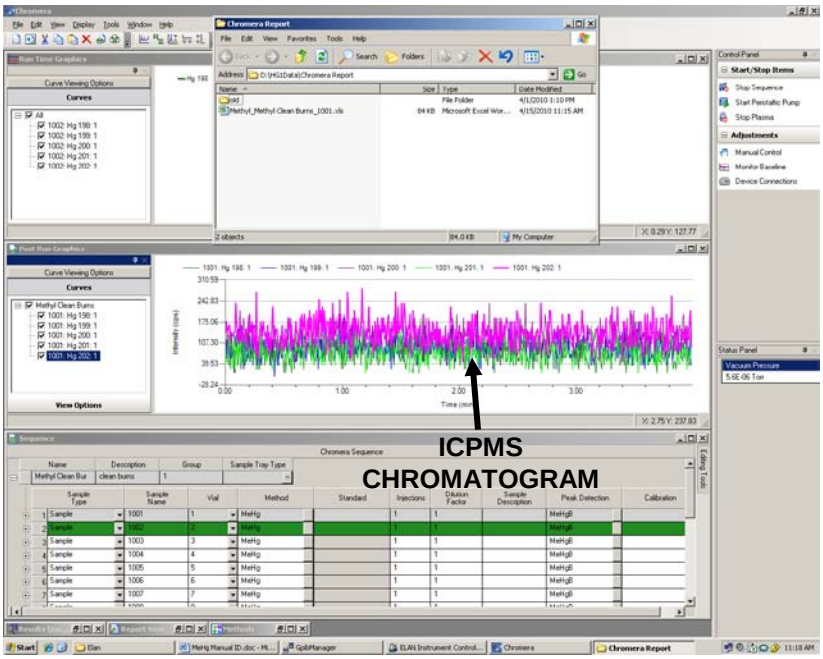


7. Click on the sequence window to make it the active application and choose “Open Sequence” from the File menu. The group “BR549” should have 75 preprogrammed analytical sequences.

8. In the Control Panel, select “Start Sequence”. The first line of the sequence should turn green. It is necessary to start the analytical sequence in the Chromera software (to “prepare” the ICPMS detector to detect the sample) prior to initiating the Brooks Rand for sample analysis.

9. Activate the Brooks Rand software. Under the Automation tab, select the number of vials to be analyzed and the starting position. Click the start button to activate the Brooks Rand modules and subsequent detection by the ICPMS.

10. After approximately 15 minutes, chromatograms will start appearing in the Chromera software. The initial three chromatograms will be Tenex trap desorption blanks, and the following are the chromatograms from the analytical vials.



11. The report files for the integration of the chromatograms will be sent to the “Chromera Report” folder. Record the file number of the report file for each analytical event in the bench sheet.

12. Copy the peak areas out of the Chromera report files and paste them into the Excel data sheet. Orientate the data horizontally by selecting Paste Special-Transpose and paste into the appropriate conversion section located in the “peak area-curve” workbook. The data will be converted to non-comma delimited. Copy and Paste these non-comma data into the appropriate field of the workbook.

Reagents and Standards

Reagents

All reagents and/or dry chemicals used to make reagents must be of the highest purity available from the vendor and shown to be low in mercury. Upon receipt at the laboratory, containers will be marked with the date of receipt and stored in the appropriate areas. When reagents are mixed for use in this method, the person who mixes them will initial and date the reagent container. Reagents and manufacture instructions follow below.

Reagent Water: Ultra pure reagent grade water containing less than 0.1 ng/L Hg with a resistance greater than 18 MΩ-cm. The water is delivered through a 0.2 µm filter, as obtained from a Millipore Academic water-purification system or equivalent.

Argon: Ultra high purity grade 5.0 Argon is used as the carrier gas in the analytical system. The Argon is first passed through a gold bead trap to remove any Hg.

Sodium Acetate Buffer: To make a stock solution of sodium acetate buffer, measure approximately 50 ml reagent grade water, 47.2 ml glacial acetic acid, and 108.8 g sodium acetate into a 500 ml Teflon bottle. Bring up to 400 ml volume, shake until all solids dissolve, and expose to ultraviolet light for 1 week (acetate buffer may be contaminated with MeHg). Transfer to a 125 ml Teflon bottle for use as a working solution.

Sodium Tetraethylborate (NaTEB): Sodium tetraethyborate is a toxic organometallic compound that is spontaneously combustible in the presence of oxygen and other oxidizing chemicals (such as strong acids), and volatilizes toxic gases (triethyl boron). Sodium tetraethylborate has a distinctive “sweet” smell, and should be considered an indication of analyst exposure. Although the long-term health affects of NaTEB exposure is unknown, it should be assumed that repeated exposure may have adverse health affects. All use of NaTEB should take place inside a high-volume fume hood, and special consideration for equipment exposed to NaTEB in the fume hood (i.e. gloves, wipes, pipette tips, containers, etc...) must be made.

Pure solid NaTEB is purchased in 1 gram sealed glass vials (stored under N_2 gas) and kept in the freezer until use. To dilute NaTEB to a 1% working solution, dissolve 2 g of KOH in 100 mL of reagent water in a 125 ml Teflon vial and chill to sub-freezing temperatures. Check the condition of the solution often. As soon as the KOH solution begins freezing, remove the vial of $NaBEt_4$ from the freezer and score the neck of the bottle with a glass cutter or the back of a ceramic knife. Wrap the vial in a lab wipe and break the neck of the vial. It is best to work quickly at this point as to keep the pure

NaTEB cold and to limit its exposure to oxygen to reduce the risk of combustion. Immediately dump the pure NaTEB into the 2% KOH solution and gently swirl to dissolve. Rinse the glass vial with the solution if any significant amount of NaTEB remains in the vial. When the NaTEB solution is almost entirely melted, homogenize, and pour equally into 20 clean chilled 5 mL Teflon vials. Cap the vials, store in a sealed bag, and record the date prepared. This solution should be kept frozen and made fresh every 2 weeks. Never use $NaBEt_4$ solid or solutions that are yellow in color. Following use, NaTEB should be stored in an appropriately labeled and sealed bag in the freezer until the solution can be disposed of properly.

To dispose of old or unused portions of the 1% NaTEB solutions, thaw the vials and pour into a beaker under a fume hood. Fill the beaker with an equivalent volume of 6M HCl (50% concentrated solution), place on a hotplate, boil down to half-volume, and then discard the remaining solution as an acid waste. Never dispose of concentrated NaTEB in this fashion, as that it will combust, but rather dilute to a 1% concentration with water and then process as previously described.

1M KOH rinse solution: In a 500 ml Teflon bottle, add 28 g of KOH to 250 ml of reagent water and bring up to 500 ml.

Aqua Regia rinse solution: In a 1000 ml Teflon bottle, add 25 and 75 ml of concentrated HNO_3 and HCl (respectively) to approximately 100 ml of reagent water and bring up to 1000 ml.

Acidified $KCL/CuSO_4$ solution: In a 500 ml Teflon bottle, add 100 ml of reagent water and 100 ml of H_2SO_4. Add 100 ml of 20% KCl solution (20 grams of KCl into a 100 ml volume of reagent water). Add 200 ml of 25% $CuSO_4$ solution (125 g of $CuSO_4$ into a 500 ml volume of reagent water). Shake well until well mixed.

Standards

Upon receipt at the laboratory or on the day of preparation, standards should be labeled with the date received or made and the initials of the person preparing them. Highly concentrated stock solutions should be stored away from the main working areas to prevent contamination of the clean lab. Working standards and (if necessary) subsequent sub-stock dilutions should be made in a class A volumetric flask in a matrix of reagent grade water at a 2% and 0.2% concentration of glacial acetic acid and hydrochloric acid, respectively. This solution should be transferred to a Teflon bottle designated specifically for mercury standards, stored in an amber bag at 4^{o} C, and remade every 6 months. All standards must be assigned a unique letter-number-letter identification code and be entered into the laboratory database system. Isotopcially enriched MeHg standard concentrations are determined by ICPMS analysis. For working solutions of native MeHg, allow the solution to equilibrate for at least 24 hours

and then determine the concentration by analysis via cold vapor atomic fluorescence spectrometry as follows:

1. Mass of mercury in the MeHg standard: To four 15 ml Teflon vials, add 8.0 ml of reagent grade water, 1.000 ml of the MeHg working standard, and 1.0 ml of BrCl.

2. Blank contribution of mercury: To four 15 ml Teflon vials, add 9.0 ml of reagent grade water and 1.0 ml of BrCl.

3. Store the vials in a rack, seal in a bag, and heat in an oven to 50^{o} C for eight hours.

4. Analyze the contents of the eight Teflon vials by EPA method 1631.

5. Analyze four 1.000 ml additions of the MeHg working standard to determine the $SnCl_2$ reducible fraction of Hg^{II}

6. Subtract the average blank mercury mass and the $SnCl_2$ reducible fraction of Hg^{II} from the total mercury mass determined MeHg working standard to determine the actual MeHg mass in the vials and subsequent concentration.

Quality Assurance and Control Objectives

During the analytical run, the analyst must evaluate the calibration data, instrument blank values, CRM/method spike recoveries, and check standard recoveries to ensure acceptance criteria are being met. The “summary” workbook in the Excel data sheet is where this information is displayed.

Certified Reference Material/MeHg method spike

A CRM should be analyzed at least twice per distillation, with a recovery within 75-125% of its certified value. If an appropriate CRM is not available, a MeHg method spike (100 µl working standard addition) may be substituted. The CRM should be of the same matrix as the samples and be reasonably similar to the expected concentrations. In the event of a failed CRM/method spike recovery repeat the analysis (if possible) to rule out a spurious result and seek the guidance of the quality assurance officer.

Currently, there are no available reference materials certified for MeHg that are either at an appropriate concentration or composed of a suitable matrix.

Instrumental Carryover

Instrumental carryover is assessed by the instrument blanks, which is the analysis of reagent water (with buffer and NaTEB), and should be analyzed throughout the run. Excessive instrument carryover indicates that the sample train has been contaminated with MeHg and requires cleaning (see below).

Method Blank

A method blank should be analyzed at least once every ten samples. Method blanks are part of the distillation set up and consist of a distillation vial with reagent water, a clean QFF filter, 2 ml of 25% $CuSO_4$, and an isotopic MeHg spike. Elevated method blanks indicate contamination in the distillation vials, QFF, or reagents and are used to calculate the DDL (daily detection limit) for the extraction batch. The Absolute Detection Limit (three times the standard deviation of the method blank masses) must be less than or equal to 0.0024.

Instrument Calibration

Instrument calibration requires the measurement of mass bias (measured isotopic fractionation of native MeHg compared to published values) and the analysis of five isotopically spiked native MeHg standards (reverse ID). The native MeHg mass in the reverse ID calibration and check standards should be near that expected in the samples.

Reverse ID calibration: Add 25 – 200 µl of native MeHg standard to 5 analytical vials. Add 25 – 200 µl of isotopically enriched MeHg standard to these same vials. Continue with the setup of analytical vials as previously described. Enter the volume of native and isotopically enriched standards added to the appropriate fields of the “peak area-curve” workbook.

Reverse ID check standard: A reverse ID check standard is prepared similar to the reverse ID calibration vials. Vary the volumes of native MeHg standard throughout the check standards so that the mass brackets the native MeHg mass of the samples-with emphasis near the most common levels within the samples. At the minimum, a reverse ID check standard should be analyzed to verify instrument calibration in every eighth position, and have a measured mass within 80 – 120% of its true value. Enter the volumes of native and isotopically enriched standards added to the appropriate fields of the “peak area-curve” workbook. The failure of subsequent check standards is an indication of instrumental drift which may require recalibration of the instrument.

Mass Bias: The typical calibration curve (created with native MeHg) is used to determine the mass bias correction. Create a five point calibration curve with volumes of native MeHg standard spanning 10 – 600 µl. Mass bias is determined by dividing the measured ratio of the isotopes of interest

(amended isotope/non-amended isotope) by published values (IUPAC) and must be within 5% of the expected value. The standard deviation for the ratio among the measured isotopes must be less than 10%.

Isotopic Fractionation of Enriched Standard: The enriched isotopes used to create the reverse ID calibration/check standards and to amend environmental samples is contaminated with small amounts of other isotopes. Due to the sensitive nature of isotopic analysis, the instrument must be calibrated to account for these contaminants. The isotopic fractionation of the enriched isotopes can be found in the "isotope info" workbook. See the quality control officer for further direction as to the appropriate source for which to reference.

Additional Instructions

Instrument Maintenance

The MERX system requires some short- and long-term maintenance. Empty the waste receptacle daily to prevent the overflow of spent sample medium. The purge vessel and sample lines should be cleaned monthly or sooner if necessary. The three analytical Tenex traps will last for approximately 2000 desorption's each before they need replacing, and the detector lamp life is approximately 4-6 months. See pages 29-30 of the MERX user's guide for detailed instructions for lamp and trap replacement.

Sample Line/Purge Vessel Cleaning

The purge vessel and sample line will require monthly cleaning under regular use or sooner as evidenced by elevated instrument blanks. Clean this equipment in the following order: 1 M KOH (8 hrs), reagent water rinse, 10% aqua regia (8 hrs), and reagent water rinse. Dry the purge vessel with Argon gas and double bag prior to storage.

Equipment Cleaning

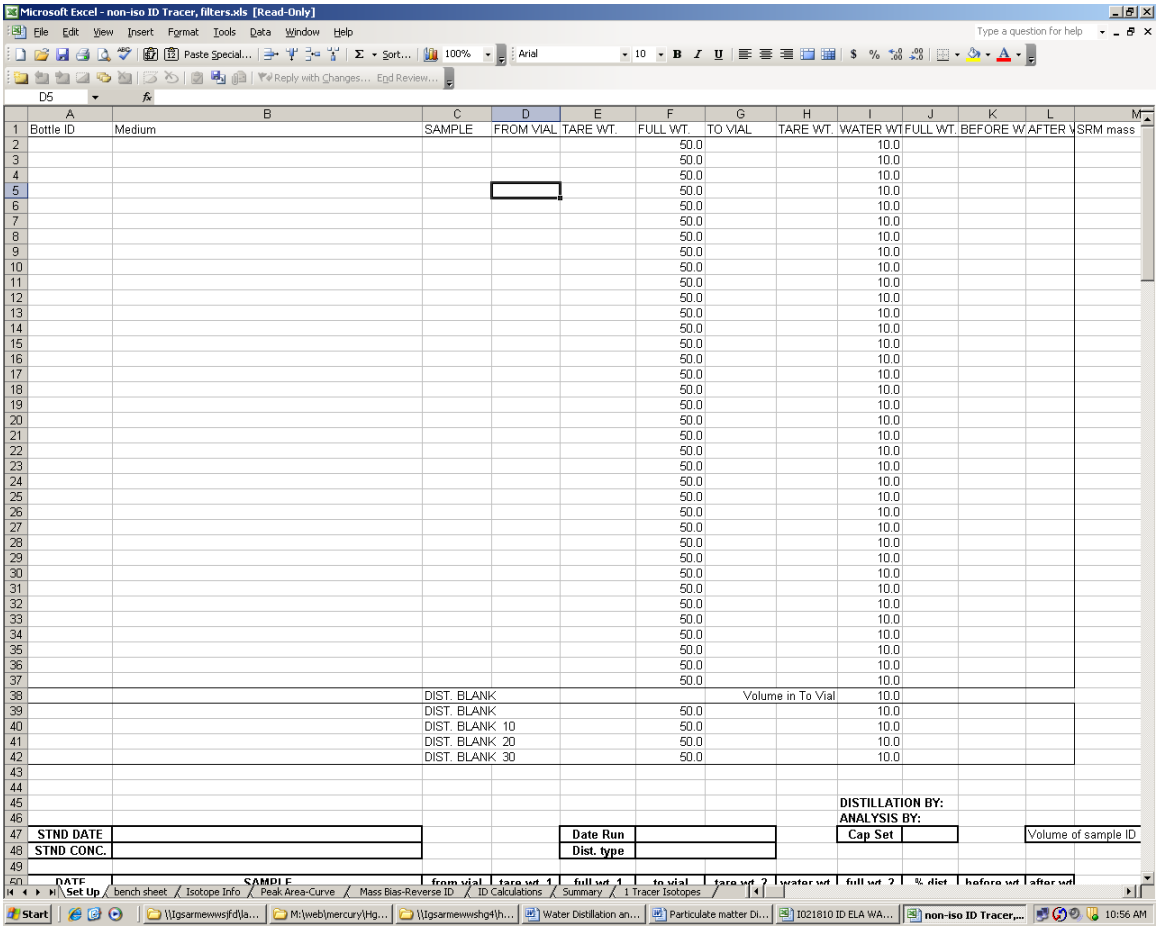
Trace level mercury analyses of samples at parts per billion concentrations are susceptible to contamination. Equipment that comes into contact with samples or reagents should be free of residual mercury and can consist of (but not be limited to) Teflon, glass, and polypropylene containers. Brand new and previously used Teflon equipment should be washed in acid before use. The equipment is first rinsed with tap water, and then cleaned by immersing in 4 N HCl heated to 65°C for at least 12 hours (48 hours for new Teflon equipment). Immediately following removal from the bath, equipment is completely immersed in reagent-grade water and then additionally triple-rinsed in reagent-grade water. After rinsing, each container is air dried under a mercury-free class 100 laminar flow hood. Dry equipment is stored double bagged in zip-type bags.

Analytical Vial Cleaning

Clean the amber glass vials with the following method. Wash the vials with lab detergent and rinse with reagent water. Once dry, wrap the vials in aluminum foil and heat at 550 º C for 2 hrs. Inspect the vials prior to use for chips or cracks.

Shutdown**:** Turn off the plasma and exit Elan. Do not save changes to the method file when prompted. Turn the coolant pump off. Create a new folder in the "old" folder in the Chromera Report folder. The naming convention is Mxxxxxx, where xxxxxx represents the six digit date MMDDYY. Cut all of the reports from the Chromera Report folder (except the "old" folder) and paste them into the new folder within the old folder.

Appendix 1. Additional instructions for distillation data entry.



Enter the following information into the appropriate fields: STND DATE, STND CONC., Date Run, DISTILLATION BY, and ANALYSIS BY.

Enter the 3 digit numeric codes (etched onto the wall of the vial) for the corresponding distillation and receiving vials into the columns titled “FROM VIAL” and “TO VIAL” (respectively). Be sure to enter the blanks into the appropriate area.

Enter the corresponding sample ID’s into the column titled “BOTTLE ID”. Certified reference materials should be placed equally throughout the sample group.

Enter the full weight of the distillation vials after the sample has been added to the column “FULL WT”.

Enter the tare weights for the distillation and receiving vials, and the sample information and volume filtered using the database query tool found in *query_TAre_FieldSampleInfo.xls*. Follow the directions in each tab to query for the needed information and enter it into the spreadsheet.

Following the distillation, enter the final mass of the receiving vial into the column titled “FULL WT.”

Following the analysis, enter the peak area of the analyzed samples into the column titled “PEAK AREA”.

APPENDIX 2. Example of a typical analytical run.

Rack 1

8 Check Std.	7 Instrument Blank	6 Sample 6	5 Sample 5	4 Sample 4	3 Sample 3	2 Sample 2	1 Sample 1
16 Check Std.	15 Instrument Blank	14 Sample 12	13 Sample 11	12 Sample 10	11 Sample 9	10 Sample 8	9 Sample 7
24 Check Std.	23 Sample 19	22 Sample 18	21 Sample 17	20 Sample 16	19 Sample 15	18 Sample 14	17 Sample 13

Rack 2

32 Check Std.	31 Instrument Blank	30 Sample 25	29 Sample 24	28 Sample 23	27 Sample 22	26 Sample 21	25 Sample 20
40 Check Std.	39 Instrument Blank	38 Sample 31	37 Sample 30	36 Sample 29	35 Sample 28	34 Sample 27	33 Sample 26
48 Check Std.	47 Sample 38	46 Sample 37	45 Sample 36	44 Sample 35	43 Sample 34	42 Sample 33	41 Sample 32

Rack 3

56	55	54	53	52 Check Std.	51 Instrument Blank	50 Sample 40	49 Sample 39
64	63	62	61	60	59	58	57
72	70	70	69	68	67	66	65

Appendix 3. Analytical bench sheet for the ICPMS.

Date: _____ ID Std: _____ Native Std: _____ Initials: _____

ID	Sample ID	Vial ID	Chromera File #
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			
31			
32			

ID	Sample ID	Vial ID	Chromera File #
33			
34			
35			
36			
37			
38			
39			
40			
41			
42			
43			
44			
45			
46			
47			
48			
49			
50			
51			
52			
53			
54			
55			
56			
57			
58			
59			
60			
61			
62			
63			
64			