

**USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA**

STANDARD OPERATING PROCEDURE 515

**DETERMINATION OF MERCURY IN WATER BY COLD VAPOR ATOMIC
ABSORPTION SPECTROMETRY**

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ABSORPTION SPECTROMETRY**

This SOP was prepared by Lockheed Martin Environmental Services (LMES) for the United States Environmental Protection Agency (USEPA) under the Environmental Services Assistance Team (ESAT) contract (EPA contract No. 68D60005).

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1 SCOPE AND APPLICATION

- 1.1 This SOP describes the instrumental analytical procedures for mercury by cold vapor atomic absorption (CVAA). This method is applicable for drinking, ground, surface, sea and brackish water, domestic and industrial wastes. This method applies to the determination of total mercury (organic + inorganic) by Method 245.1. Sample and standard preparation procedures are described in SOP 415.
- 1.2 The reporting limit (RL) is 0.20 $\mu\text{g/L}$ for total mercury.

2 METHOD SUMMARY

An aliquot of a water sample is transferred to a BOD bottle or equivalent closed-system container. The sample is digested with a dilute potassium permanganate-potassium persulfate solution for two hours at 95°C. The digestion oxidizes all forms of mercury to Hg(II). The Hg(II) in the digested water sample is reduced with stannous chloride to elemental mercury which is sparged from the sample and detected by atomic absorption. The measurement step is performed using an automated mercury analyzer.

3 DEFINITIONS

- 3.1 Analytical Sample - Any sample in which mercury is being determined, excluding standards, method blanks, or QC reference samples.
- 3.2 Calibration Blank (CB) - A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.
- 3.3 Calibration Standard (CAL) - A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 3.4 Field Reagent Blank (FRB) - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to the sampling site conditions, storage, preservation, and all analytical procedures. The purposes of the FRB is to determine if contamination is occurring in the field environment. Note: Field reagent blanks cannot be used for LD or LFM analyses.
- 3.5 Field Duplicates (FD) - Two separate samples collected at the same time and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection and storage as well as with laboratory procedures.

- 3.6 Instrument Performance Check Solution (IPC) - A standard containing the analytes of interest which is used to verify the accuracy of the analysis and monitor instrument drift. It is analyzed periodically throughout an analysis sequence.
- 3.7 Laboratory Duplicate (LD) - An aliquot of sample prepared and analyzed separately with identical procedures. Analysis of the sample and LD indicates precision associated with the laboratory procedures, but not with sample collection, preservation or storage procedures.
- 3.8 Laboratory Fortified Blank (LFB) - An aliquot of reagent water or other blank matrix to which known quantities of the method analyte is added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 3.9 Laboratory Fortified Sample Matrix (LFM) - An aliquot of an analytical sample to which known quantities of the method analyte is added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analyte in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.
- 3.10 Laboratory Reagent Blank (LRB) - An aliquot of reagent water or other blank matrix that is treated exactly as a sample. The LRB is used to detect sample contamination resulting from the procedures used to prepare and analyze the samples in the laboratory environment.
- 3.11 Linear Dynamic Range (LDR) - The concentration range over which the instrument response to an analyte is linear.
- 3.12 Method Detection Limit (MDL) - The minimum concentration of an analyte in an that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.
- 3.13 Performance Evaluation Sample (PE) - A solution of method analyte distributed by the Quality Assurance Research Division (QARD), Environmental Monitoring Systems Laboratory (EMSL-Cincinnati), U.S. Environmental Protection Agency, Cincinnati, Ohio, to multiple laboratories for analysis. A volume of the solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used by QARD to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst. Analyte true values are unknown to the analyst.

- 3.14 Reporting Limit (RL) - The concentration at which confidence in the reported value requires no qualifying remarks. The RL should be approximately 3-5 times the MDL. A standard is run at the RL to verify acceptable data quality.
- 3.15 Quality Control Sample (QCS) - An independent solution of the method analyte of known concentration. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check either laboratory or instrument performance with externally prepared test materials.
- 3.16 Standard Addition (SA) - The addition of a known amount of analyte to the sample in order to determine the relative response of the detector to an analyte within the sample matrix. The relative response is then used to assess either an operative matrix effect or the sample analyte concentration.
- 3.17 Stock Standard Solution (SSS) - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

4 HEALTH AND SAFETY

- 4.1 The toxicity and carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Specifically, concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large amounts of water for at least 15 minutes. Contact your Supervisor or Health and Safety Coordinator to determine if additional treatment is required.
- 4.2 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Exhaust or carrier gases should be vented to a fume hood. The analyst should use chemical resistant gloves when handling concentrated mercury standards.
- 4.3 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.
- 4.4 Areas of high, lethal voltages exist within the instrument. Never touch parts of the instrument which are not intended for access by the instrument operator.

5 SAMPLE HANDLING AND PRESERVATION

- 5.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis and minimize waste disposal.
- 5.2 Samples are received at the Region 9 Laboratory by EPA staff in Room 503. Sample IDs and dates of collection are verified against the chain-of-custody form.
- 5.3 Samples must be received and stored at $4 \pm 2^{\circ}\text{C}$. Any deviations from the $4 \pm 2^{\circ}\text{C}$ temperature requirements must be noted in the case narrative.
- 5.4 Samples must be received preserved with HNO_3 to $\text{pH} < 2$. Any deviations from the preservation must be noted in the case narrative.
- 5.5 Holding Time - The maximum sample holding time for mercury is 28 days from the time of collection.

6 INTERFERENCES

- 6.1 Interferences have been reported for waters containing sulfide, chloride, copper and tellurium. Organic compounds which have broad band UV absorbance (around 253.7 nm) are confirmed interferences. The concentration levels for interferants are difficult to define. This suggests that quality control procedures (Sect. 8) must be strictly followed.
- 6.2 Volatile materials (e.g. chlorine) which absorb at 253.7 nm will cause a positive interference. In order to remove any interfering volatile materials, addition of stannous chloride solution should be followed by swirling of the digestion vessel before pouring the contents into autosampler vials.
- 6.3 Low level mercury sample preparation, digestion, and analysis may be subject to environmental contamination if performed in areas with ambient backgrounds where mercury was previously employed as an analytical reagent in analyses such as total Kjeldahl nitrogen (TKN) or chemical oxygen demand (COD).

7 APPARATUS AND MATERIALS

- 7.1 Apparatus
 - 7.1.1 Leeman PS200 Mercury Analyzer - includes the optical cell, mercury lamp, peristaltic pump, drying tube, sample and reagent tubing and Leeman PS200 software.
 - 7.1.2 Okidata Microline 320 printer, or equivalent.
 - 7.1.3 Stirrer plate and stirring bars.
 - 7.1.4 50-mL plastic standard cups (Leeman P/N 116-2102).

- 7.1.5 Disposable borosilicate autosampler vials, 16x100 mm (VWR P/N 60825-618)
- 7.1.6 Class "S" weights.
- 7.1.7 Plastic or Teflon spray bottle.
- 7.1.8 Analytical balance capable of weighing to the nearest 0.01 g.
- 7.1.9 Drying tubes.

7.2 Glassware

- 7.2.1 Volumetric Class "A" Flasks - 1000-mL, 500-mL, 250-mL and 100-mL.
- 7.2.2 Volumetric Class "A" Pipettes - 50-mL, 25-mL, 10-mL and 5-mL.
- 7.2.3 Glass or quartz wool.

7.3 Materials and Reagents

Reagents may contain elemental impurities which might affect analytical data. Only high-purity reagents that conform to the American Chemical Society (ACS) specifications should be used. If the purity of a reagent is in question, analyze for contamination.

- 7.3.1 Reagent water - All references to reagent water in this method refer to ASTM Type II grade water.
- 7.3.2 Hydrochloric Acid (HCl), concentrated, trace metals grade or better.
- 7.3.3 Stannous Chloride, SnCl_2 , ACS Reagent Grade or better, suitable for mercury determination..
- 7.3.4 10% SnCl_2 in 10% HCl. Dissolve 100 g of stannous chloride (7.3.3) in 100 mL HCl (7.3.2) and dilute to 1L with reagent water. Place a stirring bar into the solution. Record the preparation of this reagent in the Inorganic Standards and Reagents Preparation Log Book (Attachment E).
- 7.3.5 Argon gas, ICP grade.
- 7.3.7 Magnesium Perchlorate, powder or granular.
- 7.3.8 10% HCl. Add 200 mL HCl (7.3.2) to approximately 1.5 liters reagent water and dilute to 2L with reagent water. Record the preparation of this reagent in the Inorganic Standards and Reagents Preparation Log Book (Attachment E).

8 QUALITY CONTROL PROCEDURES

EPA Region 9 Laboratory operates a formal quality control (QC) program. As it relates to this SOP, the QC program consists of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks, QCS samples and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

8.1 Initial Demonstration of Performance - Prior to use in routine analysis, the method must be characterized in terms of calibration linearity and MDL.

8.1.1 LDR - Absorbance vs. mercury concentration is generally a linear function over a limited concentration range. Prior to using this method for routine analysis, the calibration range must be characterized and linearity demonstrated. The characterization must be verified annually or whenever a significant change in instrument response is observed or expected (e.g, a new optical cell is installed). The initial characterization is performed by analyzing a calibration blank and a series of calibration standards with a concentration range spanning the expected sample concentration range. The resultant absorbance vs. concentration data are plotted and examined for linearity using professional judgement. A calibration line is calculated by linear regression, using at least 4 data points. To be considered linear, the correlation coefficient must exceed 0.995. Additionally, the recovery for the standards used in the calibration line must be within 90 - 110%. The recovery is calculated by entering the standard response back into the calibration line as follows:

$$\% R = \frac{(Y - b)}{m} \times \frac{1}{T} \times 100$$

Where

- %R = percent recovery of the standard
- Y = peak-area or peak-height of standard
- b = y-intercept of calibration line
- m = slope of calibration line
- T = true concentration of calibration standard

If the linearity criteria are not met, the calibration range must be adjusted until the criteria are met.

8.1.2 MDL - The MDL must be established using reagent water fortified at a concentration of two to three times the estimated instrument detection limit. An MDL in reagent water represent a best case situation and does not reflect possible matrix effects of real world samples.

To determine the MDL, process seven replicate aliquots of the fortified reagent water through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = t \times s$$

Where

- MDL= method detection limit
- t = student t value (3.14 for n=7, 99% confidence interval)

s = standard deviation

If the relative standard deviation (RSD) from the analyses of the seven aliquots is <10%, the mercury concentration in the reagent water may have been too high, which will bias the MDL value. In such a case, the MDL study should be repeated with a lower mercury concentration. The MDL should be determined annually or whenever analytical conditions are changed significantly.

8.2 Routine Analytical Quality Control

8.2.1 IPC - The accuracy and stability of the calibration is verified by the periodic analysis of an IPC standard. It must be analyzed immediately after calibration, after every 10 samples, and at the end of an analytical run. The recovery of analytes in the IPC is calculated as follows;

$$\% R = \frac{M}{T} \times 100$$

Where

%R = percent recovery of the standard

M = measured concentration of the analyte, $\mu\text{g/L}$

T = true concentration of the analyte in the IPC

Recovery of the IPC analyzed immediately after calibration must be within 95-105% or the analysis must be terminated. Subsequent analyses of the IPC solution must be within 90 - 110% or the analyses must be terminated. The cause of the poor recovery must be determined and the problem corrected. The instrument must be re-calibrated and all samples not bracketed by acceptable IPC results must be reanalyzed.

8.2.2 CB - The stability of the calibration curve baseline must be monitored by analyzing a CB immediately after every IPC standard. If the absolute value of the CB result is less than or equal to the RL, the result is acceptable. If the absolute value of the CB result exceeds the RL, the analysis must be terminated. The cause of the high CB result must be determined and the problem corrected. The instrument must be re-calibrated and all samples not bracketed by acceptable CB results must be reanalyzed.

8.2.3 RL - The accuracy of the calibration at the reporting limit shall be verified by the periodic analysis of an RL standard. The RL must be analyzed at the beginning of each analytical run, immediately after the QCS. The recovery of mercury in the RL is calculated as follows;

$$\% R = \frac{M}{T} \times 100$$

Where

- %R = percent recovery of the standard
- M = measured concentration of the analyte, $\mu\text{g/L}$
- T = true concentration of the mercury in the RL

If the RL recovery exceeds the limits of 60 - 140%, the analysis shall be terminated. The cause of the poor recovery must be determined and the problem corrected. The instrument must be re-calibrated and all affected samples must be reanalyzed. If, after recalibration, the RL recovery still exceeds the 60-140% limits, the calibration standards must be reprepared and the instrument recalibrated.

- 8.2.4 LRB - The laboratory must analyze at least one LRB daily or with each batch of 20 or fewer samples of the same matrix, whichever is more frequent. LRB data are used to assess contamination from the laboratory environment. LRB values that exceed the reporting limit indicate potential laboratory contamination. If the potential contamination significantly impacts the analytical results, the LRB must be re-prepared along with the affected samples, and re-analyzed. Unless specified otherwise by project DQOs, the following statements describe when samples must be re-prepared and re-analyzed;
- 1) If the sample mercury concentration is greater than or equal to 10 times the MDL and the LRB mercury concentration is greater than 10% of the sample mercury concentration, the LRB and sample must be re-prepared and re-analyzed
 - 2) If the sample mercury concentration is less than 10 times the MDL and the LRB mercury concentration is greater than 2.2 times the MDL, the LRB and sample must be re-prepared and re-analyzed.
- 8.2.5 LFB - One LFB must be prepared and analyzed with each analytical run. The recovery of mercury in the LFB is calculated as follows:

$$\% R = \frac{LFB}{s} \times 100$$

Where

- %R = percent recovery
- LFB = measured concentration of mercury in the LFB
- s = expected mercury concentration in the LFB

The recovery of mercury in the LFB must be within the 85-115% limits. If the recovery of mercury exceeds the limits, it is judged to be out-of-control, and the source of the problem must be identified and resolved before continuing analyses.

- 8.2.6 LFM - The LFM is designed to provide information about the effect of sample matrix on the measurement system. One LFM must be prepared for every 10 routine samples of the same matrix in a sample batch (e.g., 1 LFM for a batch containing 1-10 routine samples,

2 LFM's for a batch containing 1-20 routine samples, etc.). Samples identified as field blanks cannot be used for duplicate sample analysis. The recovery of mercury in the LFM is calculated as follows:

$$\%R = \frac{C_{lfm} - C}{s} \times 100$$

Where

- $\%R$ = percent recovery
- C_{lfm} = measured concentration in the LFM corrected for sample preparation and any dilutions
- C = measured concentration of mercury in the routine sample corrected for sample preparation and any dilutions
- s = expected mercury concentration in the LFM, corrected for sample preparation and any dilutions

If the value of s exceeds 3.3 times the value of C , the acceptance window for $\%R$ is 70-130%. If the recovery of mercury falls outside the acceptance window other QC data must be examined to determine if a matrix problem exists. If the laboratory performance is in control (i.e., the IPC, QCS, and LFB results are acceptable), the poor LFM recovery is most likely matrix related. Lab duplicate results should also be examined to gain additional insight as to whether the matrix components or matrix heterogeneity are the cause of the unacceptable recovery. In either case, the problem should be discussed in the case narrative and the data user informed that the result for mercury in the unfortified sample is suspect due either to heterogeneous nature of the sample or a matrix effect. Flag any out-of-control results with an "N".

- 8.2.7 LD - Sample homogeneity can affect the quality and interpretation of the data. LD results can be used to assess sample homogeneity. One LD must be prepared for every 10 routine samples of the same matrix in a sample batch (e.g., 1 LD for a batch containing 1-10 routine samples, 2 LDs for a batch containing 11-20 routine samples, etc.). Samples identified as field blanks cannot be used for duplicate sample analysis. Homogenize the routine sample selected as the LD, obtain a representative aliquot, and proceed with sample preparation and analysis, treating the LD sample as a routine sample. Calculate the relative percent difference using the following equation;

$$\%RPD = \frac{|C_{ld} - C|}{(C_{ld} + C) / 2} \times 100$$

Where

- $\%RPD$ = relative percent difference
- C_{ld} = measured mercury in the LD
- C = measured mercury in the routine sample

The relative percent difference (RPD) must be $\leq 20\%$ for samples with mercury levels greater than or equal to $0.2 \mu\text{g/L}$. The absolute difference between duplicate results must be less than the reporting limit for samples containing less than $0.2 \mu\text{g/L}$. If the control limits are exceeded, flag all associated mercury results with an asterisk (*). Document actions in the case narrative.

- 8.2.8 QCS Analysis - The mercury concentrations in a QCS sample must be measured daily or once per batch of 20 samples, whichever is more frequent. The concentration of analyte in the QCS should be in the upper half of the calibrated range. The recoveries must be within 90-110% or the samples in the batch must be reanalyzed.

$$\% R = \frac{C_m}{C_t} \times 100$$

Where

- $\%R$ = relative percent recovery
 C_m = measured mercury concentration in the QCS
 C_t = true mercury concentration in the QCS

9 ANALYTICAL PROCEDURES

- 9.1 Sample Preparation - The sample and standard preparation procedure for mercury analysis is found in SOP 415.
- 9.2 Calibration and Standardization - The analyst is advised to follow the recommended operating conditions provided by the manufacturer. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions satisfy the analytical requirements, and to maintain quality control data verifying instrument performance. Typical operating parameters for the Leeman PS200 Mercury Analyzer are as follows:

Parameter	Setting
Argon Flow Rate	0.30 LPM
Pump Rate	5.0 mL/min

- 9.3 Analytical Run Sequence -

- 9.3.1 Set-up and configure the instrument and data system. The Leeman PS200 mercury analyzer will be operational from a cold start (i.e. Leeman PS200 turned off) after an approximate 2.5 hour warm-up period. Inspect the pump tubing for pinching. Replace the pump tubing if excessive wear is apparent. Clamp the pump tubings in the holder and snap to seal. Prepare a fresh magnesium perchlorate drying tube by inserting some glass

or quartz wool into a clean, dry tube, and adding magnesium perchlorate to fill the tube up to the neck. Tap the tube to allow magnesium perchlorate to settle (do not compact the magnesium perchlorate), plug the drying tube with some more glass or quartz wool and attach the drying-tube fittings so that they are finger-tight. Pour out the reservoir containing reagent water into a waste container and add 10% hydrochloric acid (7.3.8) to a level approximately 1 inch from the top. Lightly oil the autosampler traverse arm and wipe clean.

- 9.3.2 After the 2.5 hour warm-up sequence has been completed, check the aperture reading by getting into the **Utility** menu and then into the **Diagnostics** menu. The aperture should be -100 to +100 (an ideal reading is zero); if not, take off the front panel of the PS200 and, using the hex wrench provided, gently adjust the bottom nut (if the value is negative) or the top nut (if the value is positive) so that the aperture reading is within the range and then replace the front panel. Use the **User Name** menu to enter analyst initials and then get back to the **Main Menu**. Select the **Protocol** (Mercury) and enter in a file name (for example, 20729981 which corresponds to instrument #2, the date, and the first run of the day). Transfer the calibration standards (in order, 0, 0.2, 0.5, 1.0, 5.0 and 15.0 ug/L mercury) into the first 6 spots in the 50mL standards tube rack. Place the bottle of 10% SnCl₂ in 10% HCl onto a stirring plate, stir, and put the tubing for the reductant into the container.

Operating parameters for Mercury protocol are:

Parameter	Setting
Argon Flow Rate	0.30 LPM
Pump Rate	5.0 mL/min

- 9.3.3 Transfer the QA/QC standards and samples into clean autosampler vials and place into the 44-sample capacity rack.
- 9.3.4 Enter the autosampler loading list into data system using **Rack Entry** and **Setup**, including all required QC and routine samples. Print the autosampler entries by pressing the F3 key.
- 9.3.5 Calibrate the instrument using the six standards. After calibration, get into **Calibration** on the **Main Menu** and then get into the **Line Calibration** menu. The current curve will be highlighted on the graph. Check the correlation coefficient (r); the correlation coefficient of the curve must be ≥ 0.995 or the instrument must be recalibrated. If the correlation coefficient meets the 0.995 requirement, press the A key to Accept the curve and press the F3 key to print the curve.

9.3.6 Start autosampler analysis sequence and analyze samples. Review results for QC compliance and off-scale results. Identify samples which must be re-analyzed in a different analytical run. Samples having analytes at concentrations higher than the linear dynamic range must be diluted into range and re-analyzed.

An example of a loading list for an analytical run sequence is listed in the table below.

Seq.	Desc.	Seq.	Desc.	Seq.	Desc.	Seq.	Desc.
1	IPC	13	IPC	25	IPC	37	
2	CB	14	CB	26	CB	38	
3	QCS	15	S5	27	S13	39	
4	RL	16	S6	28	S14	40	
5	LRB	17	S7	29	S15	41	
6	LFB	18	S8	30	S16	42	
7	S1	19	S09	31	S17	43	
8	S1-LD	20	S10	32	S18	44	
9	S1-LFM	21	S11	33	S19	45	
10	S2	22	S11-LD	34	S20		
11	S3	23	S11-LFM	35	IPC		
12	S4	24	S12	36	CB		

9.4 Data Reduction and Reporting - After set-up and calibration the instrument reports results for the analyzed solution in the units of $\mu\text{g/L}$. The concentrations reported by the instrument must be corrected for any dilutions performed as part of the sample preparation or analysis process. All results should be reported using no more than three significant figures; however, no values of less significance than the MDL may be reported (i.e., values near the MDL will be reported with only two significant figures).

9.5 Aqueous Samples - Data for aqueous samples should be reported in units of $\mu\text{g/L}$ using the following calculation;

$$C = \frac{M}{D}$$

where:

C = final reported concentration, in $\mu\text{g/L}$

M = measured concentration reported by instrument, in $\mu\text{g/L}$

D = dilution factor, to account for dilution performed after sample preparation

- 9.6 Rounding - For rounding results, adhere to the following rules:
- If the number following those to be retained is less than 5, round down;
 - If the number following those to be retained is greater than 5, round up; or
 - If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

10 DOCUMENTATION

- 10.1 Leeman PS200 Mercury Run Log - Entries are made for the case number, SDG number, date of analysis, site name, file name, analyst initials, lab sample IDs, client sample IDs, and comments, if any. The run log is maintained in Room 302.
- 10.2 Standards Documentation - Copies of all standard solution preparation entries (CAL, IPC, RL, LFB, LFM, QCS, etc.) must be submitted with each data package. (Attachment E)
- 10.3 Case Narrative and Deliverable

The data package consists of the case narrative, reports and data, as listed in the table below.

Data Package Contents
Case Narrative: Discusses any problems encountered, both technical and administrative, the corrective action taken, and the resolution. All universal analytical flags should also be discussed in the case narrative.
Tabulated sample results on an analytical spreadsheet, with units, sample collection dates, client sample IDs, laboratory IDs and station locations. (Attachment C)
Method blank data included on the analytical spreadsheet. (Attachment C)
Duplicate results on a QC summary report with calculated relative percent difference (RPD). (Attachment D)
QCS, LFM and LFB results on a QC summary report with calculated percent recoveries (%R). (Attachment D)
Raw sample, standard and QC data, including run logs and any work sheets, if applicable. (Attachment B)

11 REFERENCES

- 11.1 Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry, EPA Method 245.1, Environmental Monitoring Systems Laboratory, Revision 3.0, May 1994.
- 11.2 Leeman PS200 Operation and Maintenance Manual, Leeman Labs, Hudson, New Hampshire 03051, 1993.
- 11.3 Region 9 SOP 125, *Sample Disposal*.
- 11.4 Region 9 SOP 610, *Inorganic Data Package Assembly*.
- 11.5 Region 9 SOP 110, *Sample Receipt, Log-in and Storage*.

Attachment A
Deviations From Reference Method

- A.1 The sample and standard preparation procedures are covered in SOP 415.
- A.2 This SOP uses the Leeman PS200 Mercury Analyzer (an automated instrument) which requires only a small sample aliquot (approximately 5 mL) to perform the mercury analysis. Method 245.1 as written requires purging of the headspace in the digestion vessel, the addition of stannous chloride solution to the entire digested sample and then analysis of the sample. It is a destructive technique in that the entire sample is used. Samples which are over-range require dilution and redigestion in a subsequent digestion batch. This SOP allows for dilution of over-range samples since only 5 mL of sample was used during the initial analysis