

- TITLE:** TEST METHOD FOR SPECIATED METHOD 30B WITH THERMAL DESORPTION ANALYSIS
- SCOPE:** Describes the sampling, calibration, and sample analysis procedure for performing speciated mercury measurements using Method 30B
- PURPOSE:** To describe the test method for speciated mercury measurements using sorbent traps.

## 1.0 SUMMARY OF TEST METHOD

Known volumes of flue gas are extracted from a stack or duct through paired, in-stack sorbent media traps at an appropriate flow rate. Collection of mercury on the sorbent media in the stack mitigates potential loss of mercury during transport through a probe/sample line. For each test run, paired train sampling is required to determine measurement precision and verify acceptability of the measured emissions data. For each run the paired traps consist of one speciated trap and one total trap spiked with a known amount of Hg<sup>0</sup>. With this configuration a field recovery test is possible.

The sorbent traps are recovered from the sampling system, prepared for analysis as needed, and analyzed using the thermal desorption technique.

## 2.0 DEFINITIONS

- 2.1 *Analytical System* is the combined equipment and apparatus used to perform sample analyses. This includes any associated sample preparation apparatus e.g., digestion equipment, spiking systems, reduction devices, etc., as well as analytical instrumentation such as UV AA and UV AF cold vapor analyzers.
- 2.2 *Calibration Standards* are the Hg containing solutions prepared from NIST traceable standards and are used to directly calibrate analytical systems.
- 2.3 *Independent Calibration Standard* is a NIST traceable standard from an independent source or supplier that is used to confirm the integrity of the calibration standards used.
- 2.4 *NIST* means the National Institute of Standards and Technology, located in Gaithersburg, Maryland
- 2.5 *Run* means a series of gas samples taken successively from the stack or duct. A test normally consists of a specific number of runs.
- 2.6 *Sorbent Trap* means a cartridge or sleeve containing a sorbent media (typically activated

carbon treated with iodine or some other halogen) with multiple sections separated by an inert material such as glass wool. These sorbent traps are optimized for the quantitative capture of elemental and oxidized forms of Hg and can be analyzed by multiple techniques.

- 2.7 *Test* refers to the series of runs required by the applicable regulation.
- 2.8 *Thermal Analysis* means an analytical technique where the contents of the sorbent traps are analyzed using a thermal technique (desorption or combustion) to release the captured Hg in a detectable form for quantification.

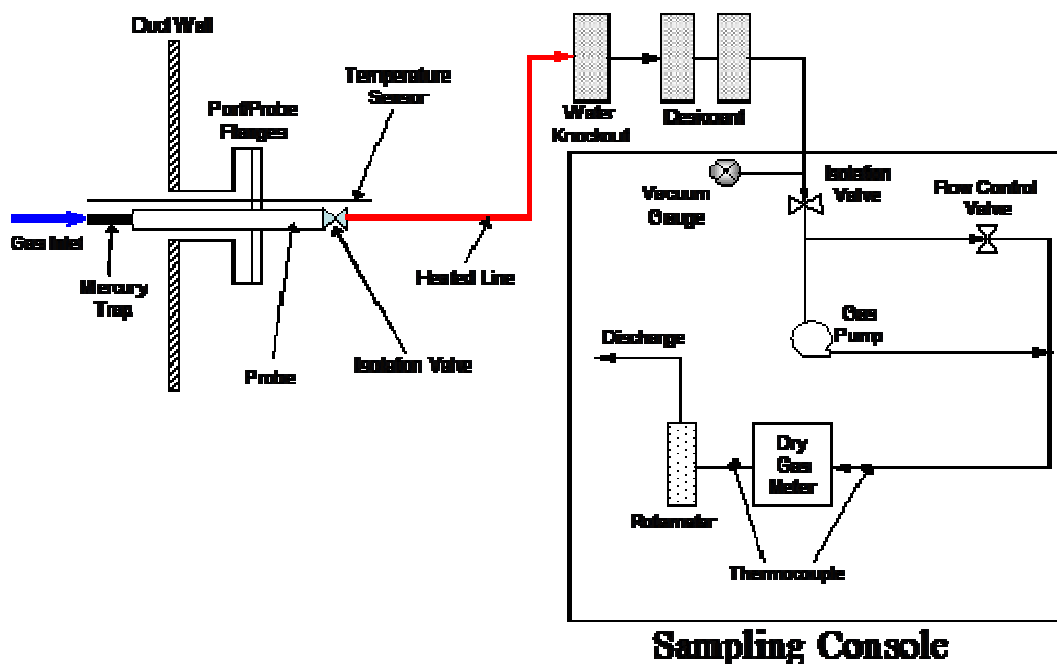
### **3.0 SAFETY**

- 3.1 *Site Hazards*. Prior to applying these procedures/specifications in the field, the potential hazards at the test site should be considered; advance coordination with the site is critical to understand the conditions and applicable safety policies. At a minimum, portions of the sampling system will be hot, requiring appropriate gloves, long sleeves, and caution in handling this equipment.
- 3.2 *Laboratory Safety*. Policies should be in place to minimize risk of chemical exposure and to properly handle waste disposal in the laboratory. Personnel shall wear appropriate laboratory attire according to a Chemical Hygiene Plan established by the laboratory.
- 3.3 *Reagent Toxicity/Carcinogenicity*. The toxicity and carcinogenicity of any reagents used must be considered. Depending upon the sampling and analytical technologies selected, this measurement may involve hazardous materials, operations, and equipment and this method does not address all of the safety problems associated with implementing this approach. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicable regulatory limitations prior to performance. Any chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized. Chemists should refer to the Material Safety Data Sheet (MSDS) for each chemical used.
- 3.4 *Waste Disposal*. Any wastes generated by this procedure must be disposed of according to a hazardous materials management plan that details and tracks various waste streams and disposal procedures.

### **4.0 EQUIPMENT AND SUPPLIES**

The following list is presented as an example of key equipment and supplies likely required to measure vapor-phase Hg using a sorbent trap sampling system. It is recognized that additional equipment and supplies may be needed. Collection of paired samples is required, using one speciated trap and one total trap spiked with a known amount of Hg<sup>0</sup>

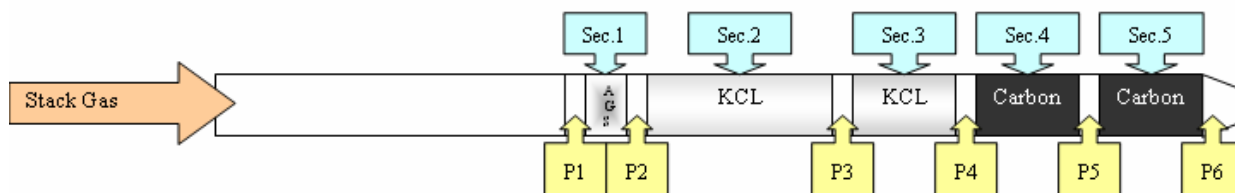
- 4.1 *Sorbent Trap Sampling System*. A typical sorbent trap sampling system is shown in Figure 30B-1. The sorbent trap sampling system shall include the following components:



**Figure 30B-1. Schematic of Method 30B Sampling Train**

- 4.1.1 *Total Sorbent Traps.* The sorbent media used to collect Hg must be configured in a trap with at least two distinct segments or sections, connected in series that are amenable to separate analyses. Section 1 is designated for primary capture of gaseous Hg. Section 2 is designated as a backup section for determination of vapor phase Hg breakthrough. Each sorbent trap must be inscribed or otherwise permanently marked with a unique identification number, for tracking purposes. The sorbent media may be any collection material (e.g., carbon, chemically-treated filter, etc.) capable of quantitatively capturing and recovering for subsequent analysis, all gaseous forms of Hg in the emissions from the intended application. Selection of the sorbent media shall be based on the material's ability to achieve the performance criteria contained in this method as well as the sorbent's vapor phase Hg capture efficiency for the emissions matrix and the expected sampling duration at the test site. The sorbent media must be obtained from a source that can demonstrate their quality assurance and quality control.
- 4.1.2 *Speciated Sorbent Traps.* The sorbent media used to collect Hg must be configured in a trap with five distinct segments or sections, connected in series that are amenable to separate analyses (see Figure 30B-2). Section 1 is designated for scrubbing acid gases prior to the Hg capture sections. Section 2 is designated for primary capture of oxidized mercury. Section 3 is designated as a backup section for determination of oxidized Hg breakthrough. Section 4 is designated for primary capture of elemental mercury. Section 5 is designated as a backup section for determination of elemental Hg Breakthrough. Each sorbent trap must be inscribed or otherwise permanently marked with a unique identification number, for tracking purposes. The sorbent media for sections 2 and 3 is potassium chloride (KCL) which is capable of quantitatively capturing and recovering for

subsequent analysis, all oxidized forms of Hg in the emissions from the intended application. The sorbent media for sections 4 and 5 is iodated carbon which is capable of quantitatively capturing and recovering for subsequent analysis, elemental Hg in the emissions from the intended application. Selection of the sorbent media shall be based on the material's ability to achieve the performance criteria contained in this method as well as the sorbent's vapor phase Hg capture efficiency for the emissions matrix and the expected sampling duration at the test site. The sorbent media must be obtained from a source that can demonstrate their quality assurance and quality control.



Speciation Trap Sections listed in direction from stack gas entry:

1. Acid Gas Scrubber (AGS)
2. Oxidized Mercury Analytical Bed
3. Oxidized Mercury Breakthrough Bed
4. Elemental Mercury Analytical Bed
5. Elemental Mercury Breakthrough Bed

**Figure 30B-2. Schematic of Method 30B Speciated Trap**

- 4.1.3 *Sampling Probe Assembly.* Each probe assembly shall have a leak-free attachment to the sorbent trap(s). Each sorbent trap must be mounted at the entrance of or within the probe such that the gas sampled enters the trap directly. Each probe/sorbent trap assembly must be heated to a temperature sufficient to prevent liquid condensation in the sorbent trap(s). Auxiliary heating is required only where the stack temperature is too low to prevent condensation. The use of an air cooled probe may be required if the stack temperature is high enough to melt the KCL sections of the trap. The speciated traps **MUST BE MAINTAINED AT A TEMPERATURE BETWEEN 100 °C AND 130 °C** to both prevent liquid condensation and not decrease the capture affinity for oxidized mercury of the KCL sections. Use a calibrated thermocouple to monitor the stack temperature. A single probe capable of operating the paired sorbent traps may be used. Alternatively, individual probe/sorbent trap assemblies may be used, provided that the individual sorbent traps are co-located to ensure representative Hg monitoring.
- 4.1.4 *Moisture Removal Device.* A moisture removal device or system, shall be used to remove water vapor from the gas stream prior to entering dry gas flow metering devices.
- 4.1.5 *Vacuum Pump.* Use a leak-tight, vacuum pump capable of operating within the system's flow range.
- 4.1.6 *Gas Flow Meter.* A gas flow meter (such as a dry gas meter, thermal mass flow meter, or other suitable measurement device) shall be used to determine total sample volume on a

dry basis, in units of standard cubic meters. The meter must be sufficiently accurate to measure the total sample volume within 2 percent and must be calibrated at the selected flow rates across the range of sample flow rates at which the sampling train will be operated. The gas flow meter shall be equipped with any necessary auxiliary measurement devices (e.g. temperature sensors, pressure measurement devices) needed to correct the sample volume to standard conditions.

- 4.1.7 *Sample Flow Rate Meter and Controller.* Use a flow rate indicator and controller for maintaining necessary sampling flow rates.
- 4.1.8 *Temperature Sensor.* Same as Section 6.1.1.7 of Method 5.
- 4.1.9 *Barometer.* Same as Section 6.1.2 of Method 5.
- 4.1.10 *Data Logger (optional).* Device for recording associated and necessary ancillary information (e.g., temperatures, pressures, flow, time, etc).
- 4.1.11 *Gaseous Hg<sup>0</sup> Sorbent Trap Spiking System.* A known mass of gaseous Hg<sup>0</sup> must be either present on or spiked onto the first section of sorbent traps in order to perform the Hg<sup>0</sup> analytical bias test. Any approach capable of quantitatively delivering known masses of Hg<sup>0</sup> onto sorbent traps is acceptable. Several spiking technologies or devices are available to meet this objective. Their practicality is a function of Hg mass spike levels. For low levels, NIST-certified or NIST-traceable gas generators or tanks may be suitable. An alternative system, capable of delivering almost any mass required, makes use of NIST-certified or NIST-traceable Hg salt solutions (e.g., HgCl<sub>2</sub>, Hg(NO<sub>3</sub>)<sub>2</sub>). With this system, an aliquot of known volume and concentration is added to a reaction vessel containing a reducing agent (e.g., stannous chloride); the Hg salt solution is reduced to Hg<sup>0</sup> and purged onto the sorbent trap using an impinger sparging system.
- 4.1.12 *Sample Analysis Equipment.* Any analytical system capable of quantitatively recovering and quantifying oxidized and elemental Hg from the sorbent media selected by thermal desorption is acceptable provided that the analysis can meet the performance criteria described in this method.
- 4.1.13 *Moisture Measurement System.* If correction of the measured Hg emissions for moisture is required, either Method 4 or other moisture measurement methods approved by the Administrator will be needed to measure stack gas moisture content.

## **5.0 REAGENTS AND STANDARDS**

- 5.1 *Reagents and Standards.* Only NIST-certified or NIST-traceable calibration standards, standard reference materials, and reagents shall be used for the tests and procedures required by this method.

**Note:** The calibration curve for the measurement of the KCL sections must be generated using HgCl<sub>2</sub>.

- 5.2 *Sorbent Trap Media.* The sorbent trap media shall be prepared such that the material used for testing is of known and acceptable quality. Sorbent supplier quality assurance/quality control measures to ensure appropriate and consistent performance such as sorptive capacity, uniformity of preparation treatments, and background levels shall be considered.

## 6.0 SAMPLE COLLECTION

Prior to sampling determine or measure the flue gas measurement environment characteristics (gas temperature, static pressure, gas velocity, stack moisture, etc.) in order to determine ancillary requirements such as probe heating and cooling requirements (if any), initial sampling rate, moisture management, etc.

- 6.1 *Pre-test leak check.* Perform a leak check of the sampling system with the sorbent traps in place. For each of the paired sampling trains, draw a vacuum in the train, and adjust the vacuum to ~15" Hg; and, using the gas flow meter, determine leak rate. The leak rate for an individual train must not exceed 4 percent of the target sampling rate. Once the leak check passes this criterion, carefully release the vacuum in the sample train then seal the sorbent trap inlet until the probe is ready for insertion into the stack or duct.
- 6.2 Ensure that the sorbent trap temperature is at approximately 110 °C. It is critical that the temperature is above 100 °C to prevent liquid condensation and below 130 °C to prevent decreased affinity for oxidized mercury of the KCL sections.
- 6.3 Remove the plug from the end of each sorbent trap and store each plug in a clean sorbent trap storage container. Remove the stack or duct port cap and insert the probe(s). Secure the probe(s) and ensure that no leakage occurs between the duct and environment.
- 6.4 Record initial data including the sorbent trap ID, date and the run start time.
- 6.5 Record the initial gas flow meter reading, stack temperature, meter temperatures, and any other appropriate information, before beginning sampling. Begin sampling and target a sampling flow rate between 0.3 and 0.5 L/min. **SAMPLING RATES GREATER THAN 0.5 L/MIN MAY CAUSE BREAKTHROUGH ISSUES.** Then, at regular intervals ( $\leq 5$  minutes) during the sampling period, record the date and time, the sample flow rate, the gas meter reading, the stack temperature, the flow meter temperatures, temperatures of heated equipment such as the vacuum lines and the probes (if heated), and the sampling system vacuum readings. Adjust the sampling flow rate as necessary to maintain the initial sample flow rate.
- 6.6 Ensure that the sorbent trap temperature is at approximately 110 °C. It is critical that the temperature is above 100 °C to prevent liquid condensation and below 130 °C to prevent decreased affinity for oxidized mercury of the KCL sections.
- 6.7 Obtain and record any essential operating data for the facility during the test period, e.g., the barometric pressure must be obtained for correcting sample volume to standard conditions. At the end of the data collection period, record the final gas flow meter reading and the final values of all other essential parameters.

- 6.8 *Post-Test Leak Check.* When sampling is completed, turn off the sample pump, remove the probe(s) with sorbent traps from the port and carefully seal the end of each sorbent trap. Perform another leak check of each sampling train with the sorbent trap in place, at the maximum vacuum reached during the sampling period. Record the leakage rates and vacuums. The leakage rate for each train must not exceed 4 percent of the average sampling rate for the data collection period. Following each leak check, carefully release the vacuum in the sample train.
- 6.9 *Sample Recovery.* Recover each sampled sorbent trap by removing it from the probe and sealing both ends. Wipe any deposited material from the outside of the sorbent trap. Place the sorbent trap into an appropriate sample storage container and store/preserve in appropriate manner (see Section 6.10).
- 6.10 *Sample Handling, Preservation, Storage, and Transport.* While the performance criteria of this approach provide for verification of appropriate sample handling, it is still important that the user consider, determine, and plan for suitable sample preservation, storage, transport, and holding times for these measurements. Therefore, procedures in ASTM WK223 “Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis.” shall be followed for all samples. To avoid Hg contamination of the samples, special attention should be paid to cleanliness during transport, field handling, sampling, recovery, and laboratory analysis, as well as during preparation of the sorbent cartridges. Collection and analysis of blank samples (e.g., reagent, sorbent, field, etc.,) is useful in verifying the absence of contaminant Hg.
- 6.11 *Sample Custody.* Proper procedures and documentation for sample chain of custody are critical to ensuring data integrity. The chain of custody procedures in ASTM D4840-99 “Standard Guide for Sampling Chain-of-Custody Procedures” shall be followed for all samples (including field samples and blanks).

## **7.0 ANALYTICAL PERFORMANCE TESTS**

- 7.1 *Hg<sup>0</sup> and HgCl<sub>2</sub> Analytical Bias Test.* As indicated in Method 30B, before analyzing any field samples, the laboratory must demonstrate the ability to recover and accurately quantify Hg<sup>0</sup> and HgCl<sub>2</sub> from the chosen sorbent media(s) by performing the following analytical bias test for sorbent traps spiked with Hg<sup>0</sup> and HgCl<sub>2</sub>. The analytical bias test is performed at a minimum of two distinct sorbent trap Hg loadings that will (1) represent the lower and upper bound of sample Hg loadings for application of the analytical technique to the field samples and (2) be used for data validation.

For speciated traps the Hg<sup>0</sup> Analytical Bias Test should be performed using the carbon as the sorbent media and the HgCl<sub>2</sub> Analytical Bias Test should be performed using KCL as the sorbent media.

- 7.2 *Hg<sup>0</sup> and HgCl<sub>2</sub> Analytical Bias Test Procedures.* Determine the lower and upper bound mass loadings. The minimum sample mass established in Section 8.2.2.2 of Method 30B can be used for the lower bound Hg mass loading although lower Hg loading levels are



acceptable. The upper bound Hg loading level should be an estimate of the greatest mass loading that may result as a function of stack concentration and volume sampled. As previously noted, this test defines the bounds that actual field samples must be within in order to be valid.

7.2.1 *Hg<sup>0</sup> Analytical Bias Test.* See section 8.2.3.1.1 of Method 30B – This test should be performed on the media (carbon) used to capture elemental mercury.

7.2.2 *HgCl<sub>2</sub> Analytical Bias Test.* See section 8.2.3.1.2 of Method 30B – This test should be performed on the media (KCL) used to capture oxidized mercury.

## 8.0 ANALYTICAL SYSTEM CALIBRATION

The calibration must be performed in the same matrix as the samples to be analyzed. In this case, there are two media types: 1) iodated carbon – for the elemental mercury capture sections and 2) KCL – for the oxidized mercury capture sections. The analyst must be sure to use the same volume/mass of media as in the sample when performing the calibration. Briefly, mercury free media (carbon or KCL) is placed in the sample boat and a small volume (no more than 100  $\mu$ l) of a known concentration NIST traceable Hg salt standard solution is applied directly onto the surface of the media (**IT IS CRITICAL THAT THE SOLUTION IS ON THE SURFACE OF THE MEDIA**). The media/Hg “calibration standard” is then thermally desorbed and analyzed. The calibration curve should be prepared through a mass range that is relevant to the actual flue gas concentration levels of interest.

**Note:** The typical concentration range of interest for tubes sampled for 2 hours at 0.4 lpm (48 liters total) ranges from 0.4 to 16  $\mu$ g/m<sup>3</sup> which corresponds to mass loadings of 20 to 800 ng. If one assumes that the speciation is 50% then the sections would have mass loadings of 10 to 400 ng.

For each calibration curve, the value of the square of the linear correlation coefficient, i.e.,  $r^2$ , must be  $\geq 0.99$ , and the analyzer response must be within  $\pm 10$  percent of the reference value at each upscale calibration point. Calibrations must be performed on the day of the analysis, before analyzing any of the samples. Following calibration, an independently prepared standard (not from same calibration stock solution) shall be analyzed. The measured value of the independently prepared standard must be within  $\pm 10$  percent of the expected value.

8.1 *Oxidized Mercury Calibration.* Calibrate analyzer as per method 30B using KCL as the calibration substrate and a NIST traceable HgCl<sub>2</sub> standard.

8.1.1 Prior to calibration rid all sample boats of mercury by placing in the analyzer furnace for 1 minute, a blast furnace set at 700 °C for 5 minutes or with a blow torch.

8.1.2 Add the appropriate mass/volume of KCL to the sample boat and apply an appropriate volume of NIST traceable HgCl<sub>2</sub> standard to the surface of the iodated carbon. **COVER WITH SODIUM CARBONATE.**

8.1.3 Insert sample boat into the analyzer and analyze.



- 8.1.4 Repeat steps 7.1.2 through 7.1.3 until all standards have been analyzed.
- 8.1.5 Check the calibration curve by preparing an Independent Calibration Verification (ICV) QC sample. This is done by spiking a known amount of mercury onto the KCL using a different Hg standard than the one used to generate the calibration curve. This QC check sample should be at the mid-point of the calibration curve.
- 8.2 *Elemental Mercury Calibration.* Calibrate analyzer as per method 30B using iodated carbon as the calibration substrate and a NIST traceable HgCl<sub>2</sub> or Hg(NO<sub>3</sub>)<sub>2</sub> standard.
- 8.2.1 Prior to calibration rid all sample boats of mercury by placing in the analyzer furnace for 1 minute, a blast furnace set at 700 °C for 5 minutes or with a blow torch.
- 8.2.2 Add the appropriate mass/volume of iodated carbon to the sample boat and apply an appropriate volume of NIST traceable Hg standard to the surface of the iodated carbon. Cover the carbon with a thin layer of sodium carbonate and remove any excess sodium carbonate above the edges of the sample boat.
- 8.2.3 Insert sample boat into the analyzer and analyze.
- 8.2.4 Repeat steps 7.2.2 through 7.2.3 until all standards have been analyzed.
- 8.2.5 Check the calibration curve by preparing an Independent Calibration Verification (ICV) QC sample. This is done by spiking a known amount of mercury onto the iodated carbon using a different Hg standard than the one used to generate the calibration curve. This QC check sample should be at the mid-point of the calibration curve.

## 9.0 SAMPLE ANALYSIS

After preparing the oxidized and elemental calibration curves you are ready to analyze samples. It is important to note that sections 1, 2 and 3 should be quantitated using the oxidized g calibration curve and sections 4 and 5 should be quantitated using the elemental Hg calibration curve. Sections 1 and 2 should be regarded as the primary capture of oxidized mercury, therefore the mass of mercury measured in sections 1 and 2 should be added together.

- 9.1 Prior to analysis rid all sample boats of mercury by placing in the analyzer furnace for 1 minute, a blast furnace set at 700 °C for 5 minutes or with a blow torch.
- 9.2 Prepare the sample by carefully cutting the glass sorbent tube with a glass cutter just above the plug (P1) of the first section (See Figure 30B-2). Carefully remove the plug and wrap it in aluminum foil. Transfer the sorbent media to a sample boat or boats depending upon the sample boat size. The glass wool plug wrapped in aluminum foil may be analyzed along with the sorbent media or separately. COVER WITH SODIUM CARBONATE.
- 9.3 Insert sample boat into the analyzer and analyze.
- 9.4 Carefully remove the plug (P2) in front of section two (KCL) and wrap it in aluminum foil

(See Figure 30B-2). Transfer the sorbent media to a sample boat or boats depending upon the sample boat size. The glass wool plug wrapped in aluminum foil may be analyzed along with the sorbent media or separately. COVER WITH SODIUM CARBONATE.

- 9.5 Insert sample boat into the analyzer and analyze.
- 9.6 Carefully remove the plug (P3) in front of section three (KCL) and wrap it in aluminum foil (See Figure 30B-2). Transfer the sorbent media to a sample boat or boats depending upon the sample boat size. The glass wool plug wrapped in aluminum foil may be analyzed along with the sorbent media or separately. COVER WITH SODIUM CARBONATE.
- 9.7 Insert sample boat into the analyzer and analyze.
- 9.8 Carefully remove the plug (P4) in front of section four (iodated carbon) and wrap it in aluminum foil (See Figure 30B-2). Transfer the sorbent media to a sample boat or boats depending upon the sample boat size. The glass wool plug wrapped in aluminum foil may be analyzed along with the sorbent media or separately. Cover the carbon with a thin layer of sodium carbonate and remove any excess sodium carbonate above the edges of the sample boat.
- 9.9 Insert sample boat into the analyzer and analyze.
- 9.10 Carefully remove the plug (P5) in front of section five (iodated carbon) and wrap it in aluminum foil (See Figure 30B-2). Transfer the sorbent media to a sample boat or boats depending upon the sample boat size. The glass wool plug wrapped in aluminum foil may be analyzed along with the sorbent media or separately. Cover the carbon with a thin layer of sodium carbonate and remove any excess sodium carbonate above the edges of the sample boat.
- 9.11 Insert sample boat into the analyzer and analyze.
- 9.12 Repeat steps 9.1 through 9.11 for each speciated sorbent trap
- 9.13 After each pair of traps check the stability of the calibration curve by preparing an Independent Calibration Verification (ICV) QC sample for each media type (both KCL and iodated carbon). This is done by spiking a known amount of mercury onto the media using the same Hg standard used to generate the calibration curve(s). This QC check sample should be at the mid-point of the calibration curve.

## **10.0 QUALITY ASSURANCE AND CONTROL**

Table 9-1 in Method 30B summarizes the QA/QC performance criteria that are used to validate the Hg emissions data from Method 30B sorbent trap measurement systems. It is important to note that the Field Recovery Test defined in Method 30B can be performed using an Hg<sup>0</sup> spike as described in section 10.1 below.

- 10.1 *Field Recovery Test.* The field recovery test requires a total of three sets of paired samples. Each pair shall contain one speciated trap and one total trap spiked with a known amount of Hg<sup>0</sup>. The mass of Hg<sup>0</sup> spiked on the total trap is determined as shown in section 8.2.6.1 in Method 30B. As in Method 30B the QA/QC criteria is an average recovery between 85% and 115%. The field recovery test % recovery calculation is shown in section 11.2 of this method.
- 10.2 *Paired Train Agreement.* The QA/QC criteria is  $\leq 10\%$  Relative Deviation (RD) mass for Hg concentrations  $> 1 \mu\text{g/dscm}$ ;  $\leq 20\%$  RD or  $\leq 0.2 \mu\text{g/dscm}$  absolute difference for Hg concentrations  $\leq 1 \mu\text{g/dscm}$ . The paired train agreement calculation is shown in section 11.3 of this method.
- 10.3 *Oxidized (KCL) Breakthrough.* The QA/QC criteria is that section 3 has  $\leq 10\%$  of the section 1 and 2 Hg masses for Hg concentrations  $> 1 \mu\text{g/dscm}$ ; section 3 has  $\leq 20\%$  of section 1 and 2 Hg masses for Hg concentrations  $< 1 \mu\text{g/dscm}$ . The oxidized (KCL) breakthrough calculation is shown in section 11.5 of this method.
- 10.4 *Probe Temperature.* The average and peak probe temperature during sampling must be reported and a probe temperature less than or equal to  $120^\circ\text{C}$  shall be targeted.

## 11.0 CALCULATIONS AND DATA ANALYSIS

The calculations and data analysis procedures are shown in section 12.0 of Method 30B. The calculations and data analysis procedures specific to speciated Method 30B are shown below:

11.1 *Nomenclature.* The terms used in the equations are defined as follows:

- B = Breakthrough (%)
- $m_1$  = Mass of Hg measured on sorbent trap section 1 ( $\mu\text{g}$ ).
- $m_2$  = Mass of Hg measured on sorbent trap section 2 ( $\mu\text{g}$ ).
- $m_3$  = Mass of Hg measured on sorbent trap section 3 ( $\mu\text{g}$ ).
- $m_{\text{spiked}}$  = Mass of Hg spiked in field Recovery Test ( $\mu\text{g}$ ).
- $m_{\text{total trap}}$  = Total mass of Hg measured on the spiked total trap in Field Recovery Test ( $\mu\text{g}$ ).
- $m_{\text{speciated trap}}$  = Total mass of Hg measured on the speciated trap in Field Recovery Test ( $\mu\text{g}$ ).
- $V_{\text{total trap}}$  = Volume of gas sampled, spiked total trap in Field Recovery Test (dscm).
- $V_{\text{speciated trap}}$  = Volume of gas sampled, speciated trap in Field Recovery Test (dscm).

R = Percentage of spiked mass recovered (%)

$C_{rec}$  = Concentration of spiked compound measured ( $\mu\text{g}/\text{m}^3$ )

$C_{total\ trap}$  = Concentration of Hg for the sample collection period, for total sorbent trap ( $\mu\text{g}/\text{dscm}$ ).

$C_{speciated\ trap}$  = Concentration of Hg for the sample collection period, for speciated sorbent trap ( $\mu\text{g}/\text{dscm}$ ).

RD = Relative deviation between the Hg concentrations from traps “total” and “speciated”

### 11.2 Calculation of Measured Spike Hg Concentration (Field Recovery Test).

Calculate the measured spike concentration using Equation 30B-1

$$C_{rec} = \frac{m_{total\ trap}}{V_{total\ trap}} - \frac{m_{speciated\ trap}}{V_{speciated\ trap}} \quad \text{Eq. 30B-1}$$

Then calculate the spiked Hg recovery, R, using Equation 30B-2

$$R = \frac{C_{rec} \times V_{total\ trap}}{m_{spiked}} \times 100 \quad \text{Eq. 30B-2}$$

### 11.3 Calculation of Hg Concentration. Calculate the concentration of the total trap using Equation 30B-3. Calculate the concentration of the speciated trap using Equation 30B-4

$$C_{total\ trap} = \frac{|m_{total\ trap} - m_{spiked}|}{V_{total\ trap}} \quad \text{Eq. 30B-3}$$

$$C_{speciated\ trap} = \frac{|m_{speciated\ trap}|}{V_{speciated\ trap}} \quad \text{Eq. 30B-4}$$

### 11.4 Calculation of Paired Trap Agreement. Calculate the relative deviation (RD) between the Hg concentration measured with the paired sorbent traps using Equation 30B-5.

$$RD = \frac{|C_{total\ trap} - C_{speciated\ trap}|}{C_{total\ trap} + C_{speciated\ trap}} \times 100 \quad \text{Eq. 30B-5}$$

11.5 *Calculation of Oxidized (KCL) Breakthrough.* Use Equation 30B-6 to calculate the percent breakthrough to the third section (second KCL section) of the sorbent trap.

$$B = \frac{|m_3|}{m_1 + m_2} \times 100$$

Eq. 30B-6