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GUIDANCE

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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

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SESDGUID-105-R1, *Analysis of Mercury in Air,* Replaces SESDGUID-105-R0 **General:** Corrected any typographical, grammatical and/or editorial errors. **Title Page:** Changed Enforcement and Investigation Branch Chief from Archie Lee to Danny France. Changed Field Quality Manager from Laura Ackerman to Bobby Lewis. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history. **Section 1.2:** Added the following statement: "Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use."	**March 6, 2013**
SESDGUID-105-R0, *Analysis of Mercury in Air,* Original Issue.	**August 7, 2009**

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to use and observe when operating the Tekran Mercury Vapour Analyzer model 2537A for the analysis of mercury in air, as well as the general and specific procedures, methods and considerations to use when using the Tekran Model 2505 Mercury Vapour Calibration Unit to calibrate and/or audit the 2537A.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when operating, calibrating or auditing the Tekran Mercury Vapour Analyzer model 2537A (herein after referred to as the **2537A**) or the Tekran Model 2505 Mercury Vapour Calibration Unit (herein after referred to as the **2505)**. On the occasion that SESD field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used for any aspect of the use of the 2537A or the 2505, the variant procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the SESD LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

Tekran Model 2505 Mercury Vapour Calibration Unit User Manual

Tekran Vapour Analyzer 2537A User Manual

1.5 General Precautions

1.5.1 General Safety

Refer to the SESD Safety, Health and Environmental Management Program Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Instrument-Specific Safety

The 2537A itself presents few Health & Safety concerns. However, the instrument is typically used in areas where mercury contamination is suspected. The individual responsible for sampling the air should use personal protective equipment (PPE) stipulated by the site safety officer. The manufacturer includes a tube filled with activated charcoal that can be placed on the vents to absorb the mercury vented from the instrument. The mercury lamp emits UV radiation; never look directly into the lamp and avoid exposing skin and eyes to the UV rays. The 2505 does contain a small amount of elemental mercury. Although the mercury is contained, care should be taken to maintain the instrument in an upright position. In the event that mercury spills or leaks from the 2505, a mercury spill kit should be used to clean the spill. A digital micro-liter syringe is used to perform injections on the front panel of the 2537A. Care should be taken to avoid needle-stick injuries.

1.5.3 Procedural Precautions

Although the 2537A can handle air samples with high mercury concentrations, the instrument is very sensitive and is capable of analyzing very low concentrations. The user must avoid exposing the instrument case and parts to high levels of mercury. Mercury contamination residing on the instrument's components could skew both analyses and calibrations. Under no circumstances should visible mercury be injected into the 2537A.

2 General Operational Guidance

2.1 Instrument Operation

The 2537A is designed to "continuously" analyze air for total gaseous mercury. The 2537A uses Cold Vapour Atomic Fluorescence Spectrophotometry (CVAFS) for the detection of mercury. The instrument has been shown to be linear from 0.1 ng/M^3 to ~1500 ng/M^3.

2.2 Sample Collection Procedure

The Tekran Vapour Analyzer 2537A continuously samples ambient air for gaseous mercury. It performs the analysis periodically as determined by the method entered by the operator into the on-board computer. The frequency of analysis (and thus the duration of the sample collection) can be varied from about every 4 minutes to 15 minutes.

The analysis begins when air is drawn into the instrument through the Sample Air Inlet. The instrument's on board computer switches a set of valves that introduces sample into one of two gold matrix cartridges at a time. As the sample passes through the cartridge, the mercury in the air amalgamates onto the encased gold mesh screen. While one cartridge samples the air, the onboard computer analyzes the other cartridge. At the start of the analysis cycle, the instrument sweeps the cartridge with argon gas, and then heats the cartridge. Mercury that was adsorbed onto the gold matrix is released during heating in argon.

The mercury is then swept into the detector by the flow of the argon carrier gas. U.V. light at 253.7 nm excites any mercury atoms present, which fluoresce and re-radiate at the same wavelength. A UV detector views the cell at right angles to the incident light. Direct light from the source is not seen, however, the fluorescence produced by the mercury in the cell is observed by the UV detector. The intensity of the fluorescence is directly proportional to the amount of mercury in the cell. Once the mass of mercury present is known, the on-board computer calculates the concentration of mercury as the mass of mercury divided by the volume of air sampled. The on-board computer sends the analysis results to a display screen and to a data-collecting computer. Although the instrument switches between chambers instantaneously, the analysis of the mercury within the chamber and calculations involved require an unavoidable lag time before the results are available. When the instrument is used for onsite sampling/analysis, the analyst must take care to link the readout to the appropriate sample.

2.3 Interferences

Due to the unique design of the 2537A, there are no known chemical interferences. The only materials that enter the instrument are gases and vapors, and the only material that is retained by the gold screen is mercury. All other materials pass through the cartridge.

The mercury is desorbed and analyzed with no other materials present to interfere with the fluorescence detection.

2.4 Required Equipment

- The Tekran 2537A

- The Tekran Model 2505 Mercury Vapor Calibration Unit. Although this item is transportable, movement can disrupt the internal mercury bead. If any of the internal mercury adheres to the syringe during calibration, the actual amount of mercury present in the sample will be drastically underreported, leading to an inflated response factor. During injection audits, or when calibration of the 2537A's internal permeation source is required, it may be necessary to transport the 2505 to the field. Whenever the unit is moved, at least 10 minutes should pass to allow the internal mercury bead and the temperature to stabilize before use. If the "Wait" indicator is visible on the 2505 display, then more time is needed for the unit to stabilize. The temperature of the Calibration Unit should always be set at least 2 degrees C below ambient temperature to prevent mercury from condensing within the syringe.

- Teflon® pre-filter. A 5µ 47mm Teflon® prefilter should be used on the inlet end of the sample line to prevent contamination of the sample line from the inlet to the instrument. The instrument has two additional 47mm Teflon® filters on its ambient air inlet and the zero air inlet.

- ¼" O.D. Teflon® tubing. The tubing is used to collect the sample and should be long enough to reach the most distant sample location, but not longer than 100 feet.

- A computer to record the data. (Must be capable of running the TEKRAN data capture program).

- A scrubber tube filled with activated carbon, to attach to the vents.

- Additional front panel septums and necessary tools.

- A mercury spill kit should accompany the 2505 if it is transported to the field.

- 10, 25 or 50µL syringes. The syringes should be gas tight, with Teflon® tipped barrels. Tekran recommends using syringes with side venting needles because they are less likely to plug on extracted septum material. The use of a digital syringe is highly recommended. The use of a digital syringe allows for faster and more consistent injections. Read the User's Manual to learn how to condition the syringes for use with the primary standard.

2.5 Reagents and Standards

- High Purity Argon to act as the carrier gas.
- Compressed zero air or high purity Nitrogen to act as the Zero Air.
- Elemental Mercury (<5ml) for use in the Tekran 2505 Cold Vapor Calibrator as a primary standard.

2.6 Calibration / Verification

2.6.1 Internal Calibration

A mercury permeation tube is housed in a temperature controlled chamber within the Tekran 2537A for routine internal calibration of the instrument. This tube is maintained at 50° C. The permeation tube constantly emits mercury at a low rate that does not vary as long as the temperature remains stable and as long as there is carrier flow over the permeation tube. This mercury can be directed to the cartridges and sets the response factor of the instrument automatically. The analyst can initiate an internal calibration in three different ways. First, the method for the on-board computer can be programmed to start the calibration at a specific time. Next, the analyst may use the keyboard to start the calibration from two different operating modes. Finally, the instrument will start a calibration when an electric circuit switch on the back panel is closed from a remote device.

The permeation rate of the mercury permeation tube is only constant once the permeation tube has reached a constant temperature and has reached equilibrium. Upon instrument startup, the calibration source should be allowed to stabilize for 12-hours with power applied and carrier gas connected before a calibration is attempted. Typically, the method stored in the on-board computer will be set to perform one calibration per 24-hour period following this warm up period. Different study requirements may require different calibration intervals.

The User's Manual provides much more detail than this document. The analyst should read and understand the manual for a complete description of the calibration process. The automated calibration is initiated using the front panel control. The steps are outlined as follows:

- From the **MAIN MENU** screen, use the arrow keys to highlight **CALIBRATE** and press **<Enter>** to start the calibration.
- From the **CALIBRATE MENU** screen, highlight **TYPE** and press **<Enter>**
- Select the **SOURCE** option
- From the **CALIBRATE MENU** screen, highlight **CALIB** and press **<Enter>**

- From the **CALIBRATE CYCLE** screen, highlight **FULL** and press **<Enter>**
- From the **CALIBRATE:CALIB** screen, highlight **BOTH** and press **<Enter>**

2.6.2 Verification / Calibration

It is important to routinely verify the permeation rate of the 2537A's internal permeation tube. A manual injection procedure must be performed to verify and/or calibrate the internal permeation rate. The manual injection procedure utilizes the well documented vapor pressure of mercury to produce a "primary standard". When elemental mercury is enclosed in a sealed chamber at a fixed temperature, it reaches equilibrium and a gaseous concentration of mercury of fixed concentration is present. The amount of mercury contained per unit volume at a given temperature is a well known function that is available in tables. To simplify this procedure, the 2505 mercury vapor calibration unit is used. It is typically operated at 15° C (as stated earlier, it should be maintained at least two degrees below ambient to prevent condensation of mercury on the syringes). The **verification** process (often used during an audit) involves injecting a known quantity of gaseous mercury at the front panel of the 2537A, or at sample inlet adapter, while the instrument is sampling zero air, in order to verify the accuracy of the internal permeation source and/or the integrity of the sampling system. The **calibration** process adjusts the instrument's RESPONSE FACTOR in relation to the internal permeation source.

The User's Manual provides much more detail than this document. The analyst should read and understand the manual for a complete description of the manual verification/calibration process. If necessary, the manual calibration is initiated using the front panel control. The calibration steps are outlined as follows:

- From the **MAIN MENU** screen, use the arrow keys to highlight **CALIBRATE** and press **<Enter>** to start the calibration.
- From the **CALIBRATE MENU** screen, highlight **TYPE** and press **<Enter>**
- Select the **INJECT** option and press <**Enter**>

- From the **CALIBRATE MENU** screen, highlight **CALIB** and press **<Enter>**
- From the **CALIBRATE CYCLE** screen, highlight **FULL** and press **<Enter>**
- From the **CALIBRATE: CALIB** screen, highlight **BOTH** and press **<Enter>**

The calibration begins with the Clean Cartridge A cycle, and proceeds to the Clean Cartridge B cycle, followed by the Zero Cartridge A, Zero Cartridge B, Span Cartridge A and finally Span Cartridge B cycle. During the Span cycles, the instrument will prompt the analyst when it is time to inject the primary standard. The amount of standard required is calculated and preset during method development. After a successful calibration, the instrument will calculate the results for both cartridges, including their Blank Correction (Blcorr) and Response Factor (Respfctr). The operator should ensure that the response factor does not change more than ±10% between calibrations or corrective maintenance may be necessary. Now the instrument is ready to measure samples.

2.7 Analysis and Operation

Once the instrument is calibrated and/or verified, the operation is straightforward. From the main menu screen, highlight **RUN** and press **<Enter>**. The instrument will start sampling and analyzing the air following the method stored in memory. The results are displayed on screen, but can also be sent to an attached computer.

2.8 Calculations

The instrument's on-board processor calculates the concentrations automatically. The user need not perform any calculations during routine sampling and analysis.

2.9 Flow Rate Checks

The sample flow rate of the 2537A is controlled by an electronic mass flow controller which in turn is controlled by the on-board computer. Since the mercury permeation tube used to calibrate the instrument emits a constant mass of mercury per unit time, changes in sample flow rate won't always be apparent during calibrations. However, changes in flow rate would result in direct errors in the quanitation of the ambient air samples. So, it is important to periodically check the flow rate to verify that the electronic flow controller is working properly. Sample flow rate is best checked against a primary flow calibrator such as a soap-film bubble meter or a positive displacement calibrator such as a Dry Cal®**.** The calibration is conducted by attaching the calibrator to the inlet and measuring the flow rate. If the flow rate doesn't match the flow rate stated on the instrument display, the mass flow controller should be recalibrated as per the Tekran manual.

2.10 Records Management and Documentation

The data sent to the attached computer should be stored for the duration of the project on the computer, but backup copies of the data should be placed onto removable discs and stored for record keeping as often as and as soon as practicable. All field activities should be documented in a bound logbook according to the procedures found in SESD Operating Procedure for Field Records and Documentation, (SESDPROC-204).