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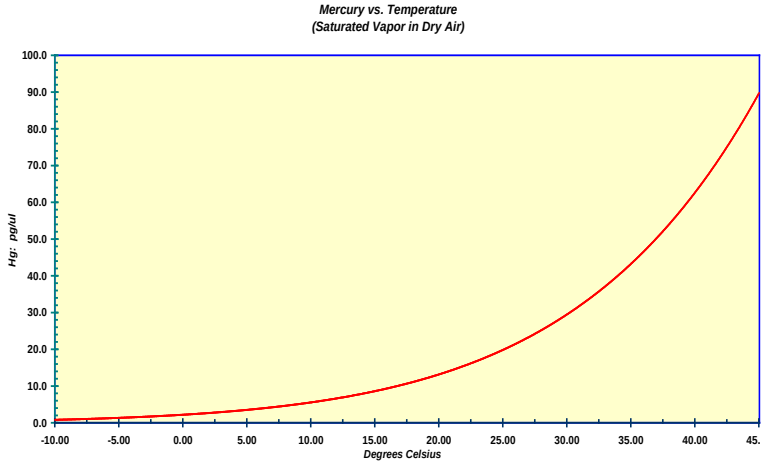
Low Level Mercury Analysis - Gas Phase Calibration

Background Information

Rev. 090415

One of the greatest difficulties in the monitoring of low levels of mercury in the atmosphere or in process gases is calibration. Low-level (ng/m^3) mercury vapor gaseous standards are not stable and cannot be supplied in cylinders. Neither the US National Institute of Standards and Technology (NIST), nor any other organization provides any means, methods, or reference materials to assist with this type of measurement.



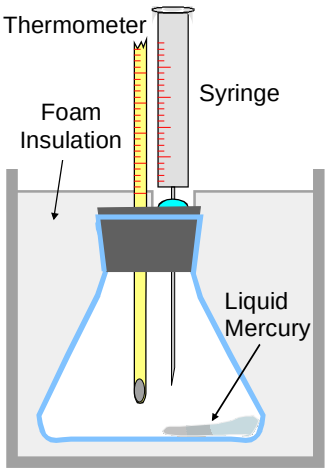
Standard Calibration Method

The usual method of calibration used by researchers is saturated mercury vapor injection[1]. This requires keeping a container filled with dry air and a small amount of liquid mercury at a constant, known temperature. The vapor pressure of mercury as a function of temperature is well documented. (See Graph at Left) The function is exponential, with increases of approximately 8% to 10% per degree centigrade. Accurate measurement of vapor temperature is thus critical to ensure overall accuracy.

Gas tight syringes with Teflon[7] tipped plungers are used to inject known amounts of mercury into the analytical system for calibration. The saturated mercury vapor must be kept below ambient temperature in order to prevent condensation of liquid mercury within the syringe. This can impact precision and may cause serious contamination of the analytical system.

Insulated Flask

The simplest implementation of a calibration source involves filling a vial with some mercury and insulating the entire assembly with foam. This is a very straightforward solution that virtually anyone can build. However, there are several problems with this basic approach.



- The source is not chilled below ambient. This can allow the condensation of microscopic droplets of liquid mercury within the needle and barrel of the syringe, causing severe contamination of your analytical equipment when injections are made.
- The source is not temperature controlled and the internal temperature is subject to constant change. Lengthy settling times are required for the air/mercury vapor mixture to reach equilibrium.
- The entry of heat through the body of standard glass thermometers can severely impact the measured vapor temperature reading.

References

1. *"The Accuracy of the Vapour-Injection Calibration Method for the Determination of Mercury by Amalgamation/Cold Vapour Atomic Absorption Spectrometry", R. Dumarey et.al., Analytica Chimica Acta, 170 (1985) 337-340.*

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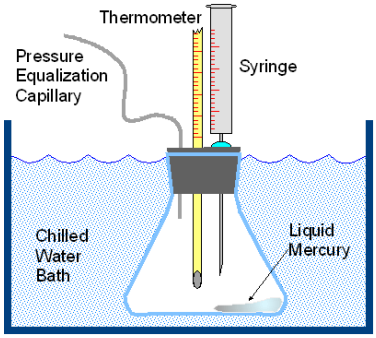
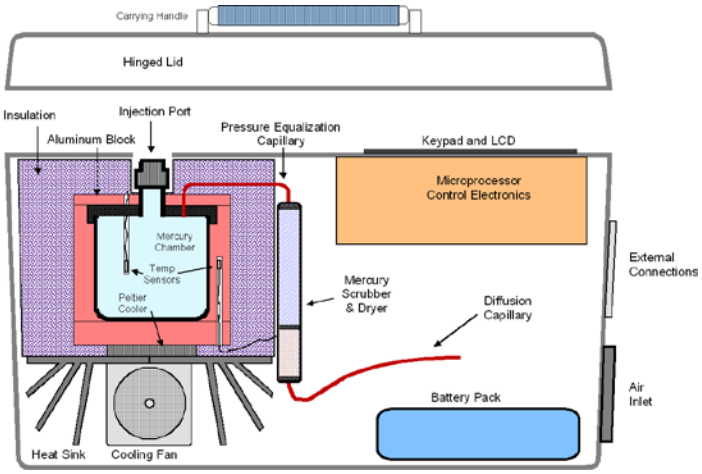
Chilled Water Bath

A more sophisticated implementation of the method utilizes a chilled water bath. This also suffers from several problems.



- Chilled water baths are bulky, take significant amounts of power and are not easily transported.
- Lengthy settling times (on the order of days) are required to let the source stabilize after transport. In addition to the time taken to re-establish temperature of the apparatus, a lengthy period is required for the air/mercury vapor mixture to reach equilibrium.
- The entry of heat through the body of standard glass thermometers can severely impact the measured vapor temperature reading. Many implementations only measure the temperature of the water bath, causing inaccuracy due to heat entry through the top of the flask.



Precision Portable Source

The requirement to periodically verify the performance of analyzers operating in the field added a new sense of urgency to solving the problems that exist with conventional sources. Most organizations have QA/QC protocols that require periodic, on-site audits of all air monitoring instrumentation. It was found soon after initial deployment of the *Tekran*® *Model 2537* ambient air analyzer that a major cause of disagreements between units operating in the field was differences in the customers' vapor sources used to characterize the internal calibration source of the analyzer.

In response to this demand, *Tekran* introduced the *Model 2505*, a temperature controlled saturated mercury vapor source. A thermoelectric cooler allows precise control of the reservoir temperature. The unit measures the reservoir temperature, accepts an injection volume, and calculates the amount of mercury in the injection. The device is portable, operating from either 12 VDC or line power.

The *Model 2505's* dual temperature sensors are calibrated against a NIST traceable source. Each Hamilton Digital Syringe comes with a NIST traceable certificate. Thus, all input variables and injection amounts are traceable in the Tekran *Model 2505* system. This unit is an ideal *primary* calibration standard for performing field and laboratory audits on the *Model 2537* analyzer or for calibrating any gas phase analytical system. The *Model 2505* solves the problems inherent with chilled temperature baths.

- The source weighs less than 5 kg. (10 lb.) A convenient carrying handle allows field transport.
- The unit may be kept powered up at all times, allowing the chamber to always maintain a constant temperature. This ensures that the vapor constantly remains at equilibrium, eliminating lengthy stabilization periods. The *Model 2505* can operate from an automotive cigarette lighter using the adaptor cable supplied.
- The unit has two sensors. One is used to control the isothermal block containing the vapor chamber. The second is housed within the chamber itself in order to measure the precise vapor temperature. The sensor leads are pre-cooled to minimize heat entry.

Note: The 2505 is shipped without Hg to prevent contamination during shipping. The unit must be loaded with Hg by the customer prior to use!