Performing a Bias Test (Method 30-B) and Spike Recovery Test (Appendix-K) for the Ohio Lumex RA-915+/RP-M324 Sorbent Trap Mercury Analyzer

Method 30-B requires that prior to sample analysis a *Bias Test* be performed on the mercury analyzer. The Appendix-K Method requires that a *Spike Recovery Test* be performed on the analyzer prior to analyzing samples using that method. Since the two tests are very similar, it is advantageous to perform them at the same time. This document will outline the steps necessary to perform the Bias Test and then list the extra steps to provide a valid Spike Recovery Test.

The Method 30-B Bias Test is required in order to show that the analytical instrument is able to measure both elemental and oxidized mercury without bias. Three sorbent traps spiked with elemental mercury at each of two levels (a low level and a high level between which actual samples are expected to fall) are analyzed to demonstrate instrument accuracy in quantifying elemental mercury. Then, two sets of three standards made with aqueous mercury solutions are analyzed at the same mercury levels as the sorbent traps. As the water evaporates during analysis, for a short instance, the mercury exists as a mercury salt (oxidized mercury) until it is then broken down thermally and measured.

Here are steps to perform a Bias Test on the Ohio Lumex Sorbent Trap Mercury Analyzer.

- Put the instrument in its most sensitive mode (profile # 1) or in the configuration that you will use for analyzing 30-B samples.
- Calibrate the instrument as you would in typical use to make sure it's measuring with accuracy and precision (run 5 or 6 points typically from 5 ng to 2,000 ng) also run a blank and a 2nd source standard. The calibration should meet the requirements of Method 30-B and the Appendix-K method. Analyze Continuing Calibration Verification Standards (CCVSs) routinely as you continue.
- Analyze three low-level traps (20 ng is a good level). The average recovery for the three analyses needs to be from 90% to 110% of the spiked amount (18 ng to 22 ng for traps spiked at 20 ng)
- Analyze three high-level traps (choose a level that is just below the highest point in your calibration to provide the maximum range). The average recovery for these three analyses also needs to be from 90% to 110% of the spiked amount.
- Next, make three standards using aqueous solutions (preserved in acid) at the same mercury mass level as the low-level spiked sorbent traps (i.e. 20ng in this example) and analyze them. The average recovery of the three must be 90% to 110% of the standard amount.

- Make three standards using aqueous solutions (preserved in acid) at the same mercury mass level as the high-level spiked sorbent traps and analyze them making sure the average recovery of the three is 90% to 110% of the standard amount.
- If all the sets of three had average recoveries between 90% and 110% of the actual amounts, the Bias Test meets the method guidelines and the analyzer can be used to analyze samples that have section-one sample amounts between the high and low points that you chose. (Section-one sample amounts below the amount you chose can be analyzed as well if they are from a low-level source).

Additional Steps to Complete the Appendix-K Spike Recovery Test

Once the steps above have been performed, very little is needed to complete the Spike Recovery Test.

- Analyze three sorbent traps spiked with elemental mercury at a medium-level that is between the low-level and high-level used above.
- If the average recovery of these three sorbent traps is between 85% and 115% of the actual amount, the results from these analyses and those of the spiked traps above represent a valid Appendix-K Spike Recovery Test. (Since the recovery requirement for the Appendix-K method is 85% to 115%, the passing results for the 30-B sorbent traps at 90% to 110% are more than adequate for this method)