

Elemental Analysis Manual

for Food and Related Products

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3.6.2 Cold Vapor Atomic Absorption Spectrometer

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This section provides information to assist the analyst on assuring analytical instrumentation is performing properly.

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GLOSSARY

3.6.2.1. Interferences

Mercury contamination from reagents, containers, miscellaneous laboratory supplies, and mercury vapor in laboratory air may cause erroneously high results unless suitable contamination control procedures are used. Contamination was minimized during validation of this method by using disposable, plastic laboratory containers and pipette tips that did not need acid-cleaning, using ultra pure acids, acid-cleaning 2-L Teflon[®] containers in which reagents were prepared, acid-cleaning ware and preparing reagents as close in time as possible to the time of use, and purging stannous chloride reducing solution with argon to remove mercury contamination. Other procedures that were found necessary to minimize contamination from laboratory air included putting caps and caps in place as much as possible, using laboratory hoods only when needed to exhaust acid fumes, and placing ultra pure acids in a secondary sealed container to minimize transport of mercury vapor through reagent bottle walls. These procedures were adequate to keep mercury contamination in solutions below approximately 0.002 µg/L during the course of analysis and make possible accurate quantification of ≥ 0.01 mg/kg levels in seafood. Additional quality control procedures, more rigorous than those in this method, must be used if lower concentrations need to be quantified accurately.

Nonspecific absorption due to molecular species in the absorption cell may produce erroneously high results. Molecular gases are minimized by using ultra high purity argon to carry mercury vapor through the atomic absorption cell and ensuring that all connections in the carrier gas path are tight. Water vapor is removed from the carrier stream by passing it through drying device. Other dissolved gases are removed by shaking the hot, decomposition acid mixture and allowing it to degas.

Spectral interferences due to direct line overlap of other elements are rare in atomic absorption and are further minimized by the vapor generation step in which mercury, but not other elements, is reduced to atomic vapor that absorbs radiation at 253.7 nm.

3.6.2.2. Instrument Setup

Set up instrument, turn on power, and warm-up instrument as directed in operator manuals provided by manufacturer. Three or more hours may be necessary to warm up electronics and detector and ensure absence of drift during analysis of solutions containing mercury concentrations < 1 µg/L. Turn on and warm up Hg lamp ≥ 30 minutes before analyzing solutions.

Inspect peristaltic pump tubing and replace it with new tubing if flat or worn spots are observed. Start gas and liquid flows and ensure that liquid flow through uptake tubing, gas-liquid separator, and drain tubing is as described in operator manuals. Condition new tubing for 30-60 minutes before analyzing sample solutions by pumping acid concentrations equal to those that will be pumped through tubes during analyses. Old analytical solutions from previously digested and analyzed samples may be combined and used to condition new sample uptake tubing. If necessary, re-adjust clamp tension on pump tubing after tubing is conditioned.

When instrument warm-up is achieved, zero the instrument, then immediately analyze a standard blank once and a standard solution with high concentration 2 or more times. Visually inspect instrument response profiles and calculate instrument sensitivity and percent relative standard deviation of the high concentration standard solution. Measure pump speed (revolutions/minute) and solution uptake rate (mL/minute) using a graduated cylinder and stopwatch. Adjust operating conditions if necessary.

3.6.2.3. Pre-standardization Checks

- (1) Instrument sensitivity check—Adequate instrument sensitivity is demonstrated by analyzing a standard solution and calculating instrument sensitivity, \hat{A} . Choose a standard in the middle standard calibration range. This instrument sensitivity must be within 20% of the instrument manufacturer's specification. If proper instrument sensitivity cannot be demonstrated, determine and correct problem before standardization.
- (2) Instrument stability check—Instrument stability must be demonstrated by analyzing a standard solution a minimum of 5 times. Choose a standard that results in the middle of the linear range. The resulting RSD of absorbance signals must be $\leq 2\%$. If $RSD > 2\%$, determine and correct problem before standardization.

3.6.2.4. Standardization Verification

To ensure accuracy of standardization, instrument standardization is verified initially, during and after an analytical run and by the analysis of a reference material.

- (1) Initial standardization verification—Analyze the low and high concentration standard solutions and standard blank as check solutions immediately following instrument standardization. Check solution recovery for low and the high standard solutions must be 95-105%. Results for the standard blank must be less than the ASDL. If either of these conditions is not met, diagnose and correct the problem(s) and re-standardize instrument.
- (2) Continuing standardization verification—To verify lack of instrumental drift, analyze a check solution at a frequency of 10% of analytical solutions and at end of analytical run. Control limits for check solutions are $100 \pm 10\%$ of expected concentrations. If control limits are not met analysis must be discontinued, cause of deviation determined and instrument re-standardized. All analytical solutions following the last acceptable check solution must be re-analyzed. This procedure ensures all groups of 10 or less analytical solution analyses are bracketed by valid standardization verification checks.

3.6.2.5. Analysis Checks

- (1) Standard curve—The value for correlation coefficient (r) must be ≥ 0.998 . The highest standard must be within the LDR. A value less than this control limit is an indication of a problem with preparation or standardization due to one or more standard solutions or the standard blank. If display of the standard curve (absorbance vs. concentration) indicates which standard solution is bad, provide re-standardization data for that standard solution. Otherwise, re-standardize with all standard solutions. If re-standardization does not fix the problem, then prepare new standard solutions and re-standardize instrument.
- (2) Peak Profile—Examine the peak profile (shape) of each analytical solution. The profiles of the standard solutions should be very close to the manufacturer's example of an ideal peak.
- (3) Carry-over—Verify absence of carry-over of mercury(II) ion from previous solutions by analyzing standard blank.