

Determination of Mercury in Hg Standard Solutions at the Lower Range Limit

LECO Corporation; Saint Joseph, Michigan USA

Instrument: AMA254



Sample Preparation

The aqueous solutions are filled directly into the sample boat by means of a suitable pipette.

Sample Weight

200 to 500 mg, balance precision of 0.1 mg or better

Accessories

614-822-114 Large Nickel Boats

Calibration Samples

LECO 502-813 Fly Ash, LECO 502-499 Dry Sludge, (BCR 143r), LECO 502-649 Dry Sludge (NIST 2781), or other suitable reference material

Analysis Time

~10 minutes

Method Profile

Drying Time:	140 seconds
Decomposition Time:	200 seconds
Cuvette Clear Time:	45 seconds
Dosing Delay Time	0 seconds
Cell Selection:	Auto Select
Metric for Calculations:	Peak Area

Procedure

- Determine the blank as follows.
 - Enter "Blank" from the drop-down menu under the "Name" column.
 - Click "Analyze"; the door will open and the nickel loop will be presented.
 - Carefully place a 614-822-114 Large Nickel Boat into the nickel loop using clean tweezers.
 - Click "OK" in the "Load Sample" window; the door will close and the analysis sequence will start automatically.
 - Repeat steps 1a through 1d two more times. The system and boats will be purged of any interfering elements.
- Calibrate the instrument as defined in the instructional manual.
 - Analyze various sample weights of a relevant reference material in accordance to the absolute amount of mercury required to calibrate an appropriate dynamic range. The calibration samples are weighed into the 614-822-114 Large Nickel Boat.

- Enter each calibration sample with the appropriate ID code from the drop-down menu, and sample weight from an external balance measurement.
- Click "Analyze"; the door will open and the nickel loop will be presented.
- If there is a boat in the nickel loop, remove it and keep for later use.
- Carefully place the calibration sample boat into the nickel loop using clean tweezers.
- Click "OK" in the "Load Sample" window; the door will close and the analysis sequence will start automatically.
- Repeat steps 2a through 2f as per the calibration procedures.

Note: The first analyzed sample after a long delay should be discarded. This sample should be considered a conditioner for the system, and not used for the actual calibration.

- Complete a calibration by following the calibration procedure as outlined in the manual.
 - Verify the calibration by analyzing one of the calibration samples again. It should be within the expected tolerances. If not, repeat steps 2a through 2i.
- Analyze the samples as follows.
 - Weigh ~200 mg of the high concentration sample into a 614-822-114 Large Nickel Boat.
NOTE: Use ~500 mg for low concentration samples.
 - Enter a sample identification in the Name column and the sample weight in the Mass column.
 - Click "Analyze"; the door will open and the nickel loop will be presented.
 - If there is a boat in the nickel loop, remove it and keep for later use.
 - Carefully place the sample boat into the nickel loop using clean tweezers.
 - Click "OK" in the "Load Sample" window; the door will close and the analysis sequence will start automatically.

Typical Results

Blank Results (ng)

0.015	0.014
0.016	0.016
0.020	0.011

Mean Value: 0.015

SD: 0.002

RSD: 18.31%

Nominal Value 200 ng/l = 0.04 ng/200 µl

Sample Weight (mg)	ng	ppm
200	0.048	0.00024
200	0.047	0.00024
200	0.052	0.00026
200	0.045	0.00022
200	0.040	0.00020

Mean Value: 0.00023 ppm

SD: 0.00002 ppm

RSD: 9.92%

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