

Determination of Mercury in Sewage Containing Hg-Loaded Particles

LECO Corporation; Saint Joseph, Michigan USA

Instrument: AMA254

Sample Preparation

After shaking, the sample can be placed directly into a boat sitting on a balance with a suitable pipette.

Sample Weight

200 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 (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

1. Determine the blank as follows.
 - a. Enter "Blank" from the drop-down menu under the "Name" column.
 - b. Click "Analyze"; the door will open and the nickel loop will be presented.
 - c. Carefully place a 614-822-114 Large Nickel Boat into the nickel loop using clean tweezers.
 - d. Click "OK" in the "Load Sample" window; the door will close and the analysis sequence will start automatically.
 - e. Repeat steps 1a through 1d two more times. The system and boats will be purged of any interfering elements.
2. Calibrate the instrument as defined in the instructional manual.
 - a. 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.
 - 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

Sewage Sample Before Particle Sieve Expected Content >0.05 ppm

Sample Weight (mg)	ng	ppm
200.0	12.347	0.0617
200.0	12.907	0.0645
200.0	11.941	0.0597

Mean Value: 0.0620 ppm

SD: 0.0024 ppm

RSD: 3.89%

Sewage Sample Before Particle Sieve Expected Content >0.05 ppm

Sample Weight (mg)	ng	ppm
200.0	8.156	0.0408
200.0	8.688	0.0435
200.0	8.116	0.0406

Mean Value: 0.0416 ppm

SD: 0.0016 ppm

RSD: 3.89%

Remarks

This application is used in process control to observe a limit of 0.05 ppm.

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