

Total Mercury in Coal and Combustion Residues

LECO Corporation; Saint Joseph, Michigan USA

Instrument: AMA254



Accessories

614-822-102 Small Nickel Boats

Note: Boats should be pre-baked at 400°C or analyzed (without a sample) before loading a sample.

Sample Weight

60 to 80 mg (0.060 to 0.080 g)

Calibration Standard

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

Analysis Time

~ 8 minutes

Method Profile

Drying Time:	60 seconds
Decomposition Time:	200 seconds
Cuvette Clear Time:	45 seconds
Dosing Delay Time:	0 seconds
Cell Selection:	Auto Select
Metric for Calculations:	Peak Area

NOTE: Method for Quicksilver Windows® Software Version 2.0.

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 614-822-102 Small 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 1c 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. Determine various weights in accordance to the absolute amount of mercury required to calibrate an appropriate dynamic range. The calibration samples are weighed into the 614-822-102 Small Nickel Boat.
 - b. Enter each calibration sample with their appropriate ID code from the drop-down menu, and sample weight from an external balance measurement.

- c. Click "Analyze"; the door will open and the nickel loop will be presented.
- d. If there is a boat in the nickel loop, remove it and keep for later use.
- e. Carefully place the calibration sample boat into the nickel loop using clean tweezers.
- f. Click "OK" in the "Load Sample" window; the door will close and the analysis sequence will start automatically.
- g. 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.

- h. Complete a calibration by following the calibration procedure as outlined in the manual.
- i. 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 again.

3. Analyze the samples as follows.
 - a. Weigh ~80 mg of the sample into a 614-822-102 Small Nickel Boat.
 - b. Enter a sample identification in the Name column and the sample weight in the Mass column.
 - c. Click "Analyze"; the door will open and the nickel loop will be presented.
 - d. If there is a boat in the nickel loop, remove it and keep for later use.
 - e. Carefully place the sample boat into the nickel loop using clean tweezers.
 - f. Click "OK" in the "Load Sample" window; the door will close and the analysis sequence will start automatically.

Typical Results

Sample	Weight(g)	Hg (ng)	Hg (ppm)
NIST	0.0810	6.5	0.080
1630a	0.0807	7.0	0.087
Coal	0.0813	6.2	0.077
	0.0800	6.6	0.083
	0.0818	6.1	0.074

Avg (ppm) **0.080**
Std **0.005**
RSD **6.3%**

Sample	Weight(g)	Hg (ng)	Hg (ppm)
NIST	0.0610	8.9	0.145
1633b	0.0610	8.7	0.143
Fly Ash	0.0608	9.2	0.152
	0.0608	9.6	0.157
	0.0601	8.9	0.147

Avg (ppm) **0.149**
Std **0.006**
RSD **3.8%**

Sample	Weight (g)	Hg (ng)	Hg (ppm)
Coal #1	0.0850	10.0	0.118
	0.0815	8.7	0.107
	0.0858	9.0	0.105
	0.0854	10.0	0.117
	0.0859	9.9	0.115

Avg (ppm) **0.112**
Std **0.006**
RSD **5.4%**

Sample	Weight (g)	Hg (ng)	Hg (ppm)
Coal #2	0.0855	24.9	0.291
	0.0863	24.1	0.280
	0.0872	23.8	0.273
	0.0847	26.7	0.315
	0.0862	23.5	0.273

Avg (ppm) **0.286**
Std **0.018**
RSD **6.3%**