Total Mercury in Soils

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

25 to 80 mg (0.025 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.
- Calibrate the instrument as defined in the instructional manual.
 - a. Weigh various weights in accordance to the absolute amount of mercury required to calibrate an appropriate dynamic range. The calibration samples are weighted 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 ~50 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(mg) | Hg (ng) | Hg (ppm) |
|--------------|------------|-----------|----------|
| NIST 2709 | 47.3 | 67.9 | 1.435 |
| Soil (as is) | 50.5 | 72.5 | 1.435 |
| | 48.1 | 72.1 | 1.479 |
| | 48.3 | 69.1 | 1.432 |
| | 49.5 | 71.1 | 1.436 |
| | | Avg (ppm) | 1.44 |
| | | Std | 0.02 |
| | | RSD | 1.39% |
| | | | |
| Sample | Weight(mg) | Hg (ng) | Hg (ppm) |
| Soil #1 | 76.8 | 17.1 | 0.223 |
| | 77.5 | 16.6 | 0.214 |
| | 75.9 | 16.5 | 0.217 |
| | 78.4 | 17.6 | 0.224 |
| | 74.3 | 16.5 | 0.222 |
| | | Avg (ppm) | 0.220 |
| | | Std | 0.004 |
| | | RSD | 1.96% |
| | | | |
| Sample | Weight(mg) | Hg (ng) | Hg (ppm) |
| Soil #2 | 23.2 | 166.5 | 7.18 |
| | 25.8 | 174.0 | 6.74 |
| | 23.7 | 171.7 | 7.25 |
| | 22.4 | 159.9 | 7.14 |
| | 29.2 | 194.6 | 6.70 |
| | | Avg (ppm) | 7.00 |
| | | Std | 0.261 |
| | | RSD | 3.7% |



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