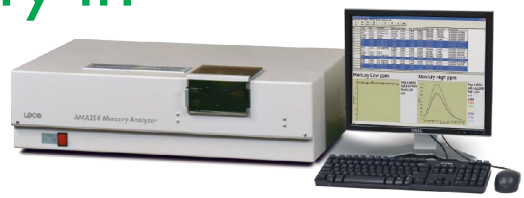
Determination of Mercury in Sewage Sludge

LECO Corporation; Saint Joseph, Michigan USA



Instrument: AMA254

Sample Preparation

The dried powder samples can be weighed directly into the boat. Drying should be done at temperatures below 70°C.

Sample Weight

Approximately 50 mg; balance precision 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 ~ 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

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.
   b. Enter each calibration sample with the 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.
3. Analyze the samples as follows.
   a. Weigh ~50 mg of the high concentration sample into a 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

Sewage Sludge, Fine • Nominal Value: 6 ppm

Sample Weight (mg)	*ng*	*ppm*
44.5	263.66	5.925
46.7	281.97	6.038
43.9	258.92	5.898
Mean Value:	*5.954 ppm*	
SD:	*0.074 ppm*	
RSD:	*1.25%*	

Sewage Sludge, Coarse • Nominal Value: 6.4 ppm

Sample Weight (mg)	*ng*	*ppm*
50.8	329.52	6.487
47.3	299.03	6.322
51.4	328.55	6.392
Mean Value:	*6.400 ppm*	
SD:	*0.083 ppm*	
RSD:	*1.29%*	

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Organic Application Note