Determination of Mercury in Cement Raw Meal, Flue Ash, and Clay Slate/Coke Mixture

LECO Corporation; Saint Joseph, Michigan USA

Instrument: AMA254

Sample Preparation
The sample can be weighed directly into the sample boat.

Sample Weight
Approximately 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
~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 ~100 mg of the high concentration sample into a 614-822-114 Large Nickel Boat.

      NOTE: Use ~200 mg for low concentration samples.
   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.

Organic Application Note

Delivering the Right Results

Typical Results

Flue Ash • Expected Content <0.5 ppm

Sample Weight (mg)	*ng*	*ppm*
140.2	39.40	0.281
143.5	39.89	0.278
135.9	38.73	0.285
Mean Value:	*0.281 ppm*	
SD:	*0.004 ppm*	
RSD:	*1.25%*	

Cement Raw Meal • Preheated in Tube Furnace to 700°C • Expected Content <0.05 ppm

Sample Weight (mg)	*ng*	*ppm*
201.6	0.169	0.00084
203.7	0.161	0.00079
197.4	0.164	0.00083
Mean Value:	*0.00082 ppm*	
SD:	*0.00003 ppm*	
RSD:	*3.23%*	

Clay Slate/Coke Blend • Expected Content 0.1 ppm

Sample Weight (mg)	*ng*	*ppm*
80.5	7.583	0.0942
140.6	12.57	0.0894
136.5	13.34	0.0977
Mean Value:	*0.0938 ppm*	
SD:	*0.0042 ppm*	
RSD:	*4.44%*	

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