Organic Application Note

Total Mercury in Industrial Wastes

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

50 to 250 mg (0.050 to 0.250 g)

Calibration Standard

NIST SRM 1633b (coal fly ash), NIST SRM 2709 (soil), and NIST SRM 2711 (soil); or equivalent Certified Reference Materials.

Furnace Temperature

550° C for catalyst; 750° C for decomp

Analysis Time

~ 6 minutes

Method Profile

Drying Time: 60 seconds
Decomposition Time: 250 seconds
Cuvette Cleaning Cycle: 45 seconds
Peak Used: Low for wastewater; high for others
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 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 614-822-102 Small 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.

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3. Analyze the samples as follows:
   a. Weigh ~60 mg of the high concentration sample into a 614-822-102 Small 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.

Typical Results

Sample	*Weight(g)*	*Hg (ppm)*
Oil/Sludge	0.0498	1.61
	0.0619	1.69
	0.0558	1.69
	Avg. (ppm)	***1.66***
Sand	0.0604	4.95
	0.0566	5.08
	0.0538	4.98
	0.0534	5.08
	Avg. (ppm)	***5.02***

Sample	*Weight(g)*	*Hg (ppm)*
Wastewater	0.1563	0.004
	0.2235	0.003
	0.2042	0.003
	Avg. (ppm)	***0.0033***
Shale	0.1781	0.025
	0.1711	0.022
	0.1671	0.022
	Avg. (ppm)	***0.023***

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