

Determination of Mercury in Plant Tissue (Vegetable Food)

LECO Corporation; Saint Joseph, Michigan USA



Instrument: AMA254

Sample Preparation

The dried and powdered samples can be weighed directly into the boat placed on the balance. Freeze drying can also be used as a method of drying.

Sample Weight

Up to 150 mg; balance precision of 0.1 mg or better (higher sample weights are possible)

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.

Typical Results

Lettuce, Nominal Value 0.035 ppm

Sample Weight (mg)	ng	ppm
108.9	4.106	0.0377
113.7	3.684	0.0324
102.4	3.686	0.0360
111.9	4.096	0.0366
97.5	3.227	0.0331
96.8	3.543	0.0366
121.8	4.287	0.0352
125.7	4.563	0.0363
112.9	3.726	0.0330
127.5	4.144	0.0325
117.4	3.780	0.0322

Mean Value: 0.0347 ppm

SD: 0.0021 ppm

RSD: 5.95%

Wheat Flour, Nominal Value 0.015 ppm

Sample Weight (mg)	ng	ppm
123.7	1.905	0.0154
144.6	2.212	0.0153
120.2	1.827	0.0152
137.8	2.150	0.0156
133.7	2.086	0.0156
149.1	2.251	0.0151

Mean Value: 0.0154 ppm

SD: 0.0002 ppm

RSD: 1.34%

Apple Powder, Nominal Value 0.011 ppm

Sample Weight (mg)	ng	ppm
96.5	1.409	0.0146
89.8	1.158	0.0129
102.4	1.393	0.0136
98.7	1.352	0.0137
86.5	1.202	0.0139
94.6	1.305	0.0138
93.8	1.398	0.0149
99.9	1.459	0.0146
102.1	1.480	0.0145
96.3	1.329	0.0138

Mean Value: 0.0140 ppm

SD: 0.0006 ppm

RSD: 4.33%

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