

Mercury (Hg) in Spinach Leaves (NIST 1570)

Method: US EPA 7473

Category: Food

Technique: CAA

Summary

This technical note will describe the analysis of trace elements in Spinach Leaves (NIST CRM 1570) using the Teledyne Leeman Labs Hydra IIc mercury analyzer. This method utilized the moisture control system as described in AN1701 ([viewable here](#)) to enhance the performance of the Hydra IIc in standard, direct combustion mode for total mercury in this matrix.

Direct combustion mercury analysis, as described in US EPA 7473, is a simple method which eliminates lengthy sample preparations and the hazardous wastes generated from wet chemistry techniques. With no sample pretreatment, total mercury results are obtained faster and more economically using the Teledyne Leeman Labs Hydra IIc mercury analyzer for direct combustion atomic absorption (CAA).

Weighed samples were introduced into the analyzer using an automated sequence. The unattended analysis of 10 samples was completed in approximately 1 hour.

The Hydra IIc also complies with ASTM 6277 and ASTM 7623 for direct analysis of mercury content by thermal decomposition.

Instrumentation

Hydra IIc CAA Combustion mercury analyzer, Envoy software version 2.2, quartz boats (calibration), nickel boats (samples), analytical balance, hot plate, watch glasses, disposable spatulas, pipettes/tips, and labware/reagents for aqueous calibration standard preparation.

Method Parameters

Parameter	°C	Seconds	Other
Oxygen Flow			350 mL/min
Drying	300	30	
Decomposition	800	150	
Catalyst	600	50	
Amalgamator	700	30	
Integration		80	

Calibration

Aqueous intermediate standards were prepared in 1% HNO₃ acid for mercury stability. Various weights of intermediate standards were added to quartz boats for total mass in ng of Hg, as listed below. Both Low and High Concentration ranges used a quadratic fit.

Low Concentration	Blank, 0.1, 0.5, 1, 2, 5, 10, 20, 50 ng
High Concentration	50, 100, 200, 400, 600, 1000 ng

Sample Weight

Average sample weight was 0.2000g.

Procedure

1. Homogenize sample in the container
2. Tare boats and add sample to boats
3. Load boats onto the autosampler sample boat shuttle
4. Run Hydra IIc sequence using listed parameters
5. Determine %moisture in CRM for data analysis

Results

Apple Leaves CRM 1515 (0.0430 µg/g)	0.0460 Recovery 106 %
Loamy Sand 3 CRM-021 (4.70 µg/g)	4.2142 Recovery 90 %
Spinach Leaves CRM 1570	0.0328
Spinach Leaves CRM 1570	0.0323
Spinach Leaves CRM 1570	0.0321
Spinach Leaves CRM 1570	0.0312
Spinach Leaves CRM 1570	0.0312
Spinach Leaves CRM 1570	0.0314
Spinach Leaves CRM 1570	0.0308
Avg	0.0317 ± 0.0005 @ 95 %
STDEV	0.0007
MDL	0.0018 @ 95 %
Min	0.0308
Max	0.0328
Loamy Sand 3 CRM-021 (4.70 µg/g)	4.1156 RPD 2.4 %

Conclusion

The calibration curve check recoveries of 106% and 90% demonstrate the system is in control and stable. The certified value for NIST CRM 1570 is 0.030 ±0.005 µg/g. This analysis of CRM 1570 had a recovery of 0.0317 ±0.0005 µg/g which is 106% of the certified value. The %RPD between the check standard before and after the run was 2.4%.

With the addition of the external moisture control system, the Hydra IIc in standard, direct combustion mode is ideal for the determination of mercury in CRM 1570 - Trace Elements in Spinach Leaves.