TELEDYNE LEEMAN LABS
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Technical Note: Hg/FD-2202

Mercury (Hg) in Kelp Powder (NIST 3232)

Method: US EPA 7473 **Category: Food** **Technique: CAA**

Summary

This technical note will describe the analysis of total mercury in Kelp Powder (NIST CRM 3232), using the Teledyne Leeman Labs' Hydra II_C mercury analyzer.

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 at less expense using the Teledyne Leeman Labs' Hydra II_C 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.

Instrumentation

Hydra II_C CAA 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		60	

Calibration

Aqueous intermediate standards were prepared in 1% HNO_3 acid for mercury stability. Various weights of intermediate calibration standards were added to quartz boats for total mass in ng of Hg, as listed below. Both Low and High Concentration ranges utilized 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

Procedure

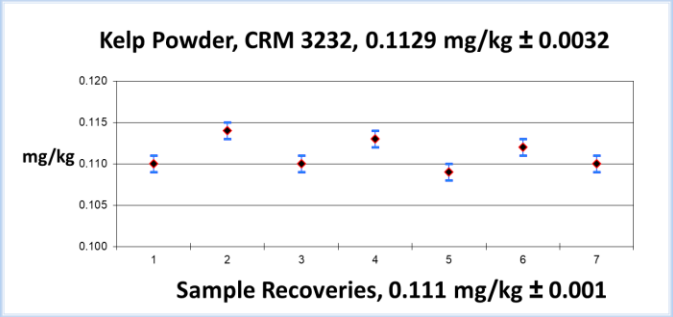
1. Homogenized sample in the container
2. Tare boats and add sample to boats
3. Load boats onto the sample boat shuttle
4. Run Hydra II_C using parameters listed with an automated sequence
5. Determine percent moisture in CRM for data analysis

Sample Weight

Average sample weight was 0.0997 g.

Results

	mg/kg
Apple Leaves CRM 1515 (0.0430 mg/kg)	0.045 105 % Recovery
Loamy Sand 3 CRM-021 (4.70 mg/kg)	4.472 95 % Recovery
Kelp Powder NIST 3232 (1)	0.110
Kelp Powder NIST 3232 (2)	0.114
Kelp Powder NIST 3232 (3)	0.110
Kelp Powder NIST 3232 (4)	0.113
Kelp Powder NIST 3232 (5)	0.109
Kelp Powder NIST 3232 (6)	0.112
Kelp Powder NIST 3232 (7)	0.110
Avg	0.111 ± 0.001 @ 95 %
STDEV	0.002
MDL	0.004 @ 95 %
Min	0.109
Max	0.114
Loamy Sand 3 CRM-021 (4.70 mg/kg)	4.620 RPD 3.4 %



Conclusion

The calibration curve check standard recoveries of 105% and 95% demonstrate the system is in control and stable. The certified value for NIST CRM 3232 is 0.1129 ±0.0032 mg/kg. This analysis of CRM 3232 had a recovery of 0.111 ±0.0013 mg/kg, which is 98% of the certified value. The relative percent difference (RPD) between the check standard analyzed before and after the run was 3.4%. The Hydra II_C in standard mode is an ideal system for the determination of total mercury in NIST CRM 3232 Kelp Powder.