TELEDYNE LEEMAN LABS
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Technical Note: Hg/ENV-1707

Pine Needles (SRM 1575)

Category: Environmental **Technique: CAA**

Summary

This technical note will describe the analysis of Pine Needles (SRM 1575), from NIST, on the Teledyne Leeman Labs Hydra IIC Mercury Analyzer. This method utilized the moisture control system as described in Teledyne Leeman Labs Application Note – AN1701 (Viewable Here), to enhance the performance of the Hydra IIC in standard direct combustion mode for total mercury determination in this matrix.

Sample pretreatment and the generation of wastes associated with wet chemistry are eliminated when using the Hydra IIC mercury analyzer in standard combustion mode for the determination of total mercury in this SRM.

Weighed samples were introduced into the analyzer using an automated sequence and the unattended analysis of each individual sample was completed in ~6.0 minutes.

Direct analysis of mercury content by Thermal Decomposition is described in methods USEPA 7473 and ASTM 6722 and 7623.

Instrumentation

Hydra IIC CVAA Combustion Analyzer, Envoy software version 2.2, quartz boats (calibration), nickel boats (samples), analytical balance, disposable spatulas, pipette and tips, labware/reagents for calibration standard preparation.

Method Parameters

	°C	Seconds	Other
Drying	300	45	
Catalyst	600	30	
Decomposition*	800	120	
Oxygen Flow			350 ml/min
Integration		100	
Amalgamator	700	40	

* A controlled increase to Decomposition Temperature was used. Add or edit the Temperature Ramp startup.ini file line to: “TempRamp 60”.

Calibration

Aqueous standards were prepared in 1% HNO_3 acid for mercury stability.

Low Concentration	Blank, 0.1, 0.5, 1, 2, 5, 10, 20 ng
High Concentration	50, 100, 200, 400, 600, 800, 1000 ng

Sample Weight

Sample weight average was ~0.050 g.

Procedure

1. Homogenize the sample in the container
2. Tare boat(s) and add sample(s) into boat(s)
3. Load boats onto the sample boat shuttle
4. Run Hydra IIC in standard mode using an automated sequence

Results

	ng/g	
Coal Fly Ash SRM 1005 ng/g	1057.61	105.2 % Recovery
SRM 1575	129.44	
SRM 1575	132.83	
SRM 1575	128.49	
SRM 1575	132.81	
SRM 1575	137.44	
SRM 1575	130.22	
SRM 1575	131.10	
Avg	131.76	± 0.2.23 @ 95 %
STDEV	2.98	
MDL	5.80	@ 95 %
Min	128.49	
Max	137.44	
Coal Fly Ash SRM 1005 ng/g	959.96	95.5 % Recovery

Conclusion

The QC recoveries of 105.2 to 95.5% demonstrate that the system is in control and stable. The certified value for SRM 1575 is 150 ±50 ng/g.

With the addition of the moisture control system, the Hydra IIC in standard mode is an ideal system for the determination of concentration of mercury in Pine Needles (SRM 1575).