

Rice Flour (SRM 1568b)

Category: Food
Technique: CAA

Summary

This technical note will describe the analysis of Rice Flour (SRM 1568b), 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 analyzer 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% HNO₃ 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 ~0.17 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	982.25	97.7 % Recovery
Rice Flour	6.30	
Rice Flour	6.09	
Rice Flour	6.10	
Rice Flour	6.02	
Rice Flour	6.07	
Rice Flour	5.92	
Rice Flour	6.11	
Avg	6.09 ± 0.08 @ 95 %	
STDEV	0.11	
MDL	0.22 @ 95 %	
Min	5.92	
Max	6.30	
Coal Fly Ash SRM 1005 ng/g	965.04	96.0 % Recovery

Conclusion

The QC recoveries of 96.0 to 97.7% demonstrate that the system is in control and stable. The certified value for SRM 1568b is 5.91 ± .36 ng/g.

With the addition of the moisture control system, the Hydra IIc in standard mode is an ideal system for determination of mercury concentration in Rice Flour (SRM 1568b).