TELEDYNE LEEMAN LABS
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Technical Note: Hg/PC-1802

Residual Fuel Oil (NIST SRM 1619b)

Category: Petrochemical **Technique: CAA**

Summary

This technical note describes the analysis of Residual Fuel Oil (NIST Standard Reference Material® 1619b), 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 are 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	150	30	
Catalyst	750	30	
Decomposition	800	180	
Oxygen Flow			350 ml/min
Integration		80	
Amalgamator	700	30	

Calibration

Aqueous intermediate standards were prepared in 1% HNO_3 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 utilized a quadratic fit with the blank being an empty quartz boat. A single blank and 50 ng Hg standard was applied to both calibration fits.

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

Sample Weight

The sample weight average was ~0.09 g. **Note: It is recommended that the sample weight for fuel oil and other petroleum products be kept as close as possible to 0.08 g.**

Procedure

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

Results

	ng/g	
Oil Standard QC 3.5 ng/g	3.58	102.3 % Recovery
SRM 1633c 1005 ng/g	1034.28	102.9 % Recovery
Hg in Residual Fuel Oil (1619b) 1	4.34	
Hg in Residual Fuel Oil (1619b) 2	3.91	
Hg in Residual Fuel Oil (1619b) 3	3.88	
Hg in Residual Fuel Oil (1619b) 4	3.59	
Hg in Residual Fuel Oil (1619b) 5	3.81	
Hg in Residual Fuel Oil (1619b) 6	3.90	
Hg in Residual Fuel Oil (1619b) 7	4.12	
Avg	3.94 ± 0.18 @ 95 %	
STDEV	0.24	
MDL	0.58 @ 95 %	
Min	3.59	
Max	4.34	
Spike	3.33	79 % Recovery
Oil Standard QC 3.5 ng/g	3.77	107.7 % Recovery

Conclusion

The QC recoveries of 102.3 to 107.7% demonstrate that the system is in control and stable for analysis of trace Hg in residual fuel oil. The trace Hg certified value for SRM 1619b is 3.46 ± 0.74 ng/g. The calculated MDL for this analytical system, under the method conditions presented in this technical note, is ≤ 0.58 ng/g at a 95% level of confidence. The spike consisted of 0.073 g of SRM 1619b and 0.081 g of 3.5 ng/g oil standard for a combined weight of 0.154 g, and was used to calculate the returned concentration of 3.33 ng/g. The percentage of recovery was calculated using the formula below:

$(3.33 - 1.87_{\text{SRM contribution}}) / 1.84_{\text{Spike Contribution}} = 79\%_{\text{Recovery}}$

Note: This calculation assumes a 100% weight adjusted SRM contribution of 3.94 ng/g.

With the addition of the moisture control system, the Hydra IIC in standard mode is an ideal system for determination of mercury in Residual Fuel Oil (SRM 1619b).