TELEDYNE LEEMAN LABS
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Technical Note: Hg/PC-2203

Mercury (Hg) in Coal (QAR-CRM-1)

Method ID: ASTM 6722 **Category: Petrochemical** **Technique: CAA**

Summary

This technical note will describe the analysis of total mercury in Coal (QAR-CRM-1), using the Teledyne Leeman Labs' Hydra II_C mercury analyzer. This method utilized the moisture control system as described in AN1701 (viewable here) to enhance the performance of the Hydra II_C in standard, direct combustion mode for total mercury in this matrix.

Direct combustion mercury analysis, as described in ASTM 6722, is a simple method which eliminates lengthy sample preparations and the hazardous waste 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 samples was completed at a rate of ~6 min/sample.

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 (mL/min)			350
Drying	300	30	
Temp Ramp*		60	
Decomposition	800	120	
Catalyst	600	20	
Amalgamator	700	30	
Integration		80	
Low Peak Abs Limit*			380000
NafionFurnaceTemp*			600
Elute Warm Temp Override*			175

*Adjusted or added via the startup.ini text file located in the Envoy software folder.

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, 5, 10, 20, 50 ng
High Concentration	Blank, 50, 100, 200, 400, 600, 800, 1000 ng

Procedure

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

Sample Weight

Average sample weight range was >0.1 g, but less than 0.15 g.

Results

	ng/g	
RM 8414 (5 ng/g ± 3.0)	6.6	132 % Recovery
SRM 1633c 1005 ng/g	916	91 % Recovery
QAR-CRM-1 (1)	128	
QAR-CRM-1 (2)	123	
QAR-CRM-1 (3)	125	
QAR-CRM-1 (4)	126	
QAR-CRM-1 (5)	125	
QAR-CRM-1 (6)	125	
QAR-CRM-1 (7)	124	
Avg	125 ± 1 @ 95 % Confidence	
STDEV	1.6	
MDL	4 @ 95 % Confidence	
Min	123	
Max	128	
RM 8414 (5 ng/g ± 3.0)	5.6	112 % Recovery
SRM 1633c 1005 ng/g	930	93 % Recovery

Conclusion

The calibration check standard of 132% and 112% recovery for RM 8414 at the trace level, and 91% and 93% for NIST 1633c in the normal range, demonstrate the system is in control and stable. The certified value for QAR-CRM-1 is 121 ±3 ng/g @ 99% confidence. This analysis of QAR-CRM-1 had a recovery of 125 ±1 ng/g, which is 103.3% of the certified value. The Hydra II_C in standard mode is an ideal system for the determination of total mercury in Coal (QAR-CRM-1).