

Mercury (Hg) in Coal (QAR-CRM-9)

Method ID: ASTM 6722
Category: Petrochemical
Technique: CAA

Summary

This technical note will describe the analysis of total mercury in coal (QAR-CRM-9), using the Teledyne Leeman Labs Hydra IIc mercury analyzer. This method utilized the moisture control system as described in AN1701 ([viewable here](#)) to enhance the performance of the Hydra IIc 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 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 IIc mercury analyzer for direct combustion atomic absorption (CAA).

Weighed samples were introduced into the analyzer using an automated sequence. The unattended analysis of samples were completed at a rate of ~ 6 min / sample.

Instrumentation

Hydra IIc 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

	°C	Seconds	Other
Oxygen Flow (mL/min)			350
Drying	300	30	
TempRamp*		60	
Decomposition	800	120	
Catalyst	600	20	
Amalgamator	700	30	
Integration		80	
LowPeakAbsLimit*			380000
NafionFurnaceTemp*			600
EluteWarmTempOverride*			175

*Adjusted in, or added to, the startup.ini text file located in the Envoy folder.

Calibration

Aqueous intermediate standards were prepared in 1% HNO₃ 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 sample boat shuttle
4. Run Hydra IIc using parameters listed with an automated sequence
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	
LECO 502-686 (107 ng/g)	104	97.2 % Recovery
SRM 1633c (1005 ng/g)	945	94.0 % Recovery
QAR-CRM-9 (1)	148	
QAR-CRM-9 (2)	143	
QAR-CRM-9 (3)	151	
QAR-CRM-9 (4)	133	
QAR-CRM-9 (5)	148	
QAR-CRM-9 (6)	145	
QAR-CRM-9 (7)	150	
Avg	145 ± 4.4 @ 95 % Confidence	
STDEV	6	
MDL	15 @ 95 % Confidence	
Min	133	
Max	151	
LECO 502-686 (107 ng/g)	105	98.1 % Recovery
SRM 1633c (1005 ng/g)	952	94.7 % Recovery

Conclusion

The calibration curve check standard of 97.2% and 98.1% recovery for LECO 502-686, and 94.0% and 94.7% for NIST 1633c demonstrate the system is in control and stable in both the high and low sensitivity calibration ranges. The certified value for QAR-CRM-9 is 132 ± 6 ng/g at 99% confidence with an acceptable range of (106 - 158) ng/g. This analysis of QAR-CRM-9 had a recovery of 145 ± 4.4 ng/g, which is 109.8% of the certified value. The Hydra IIc in standard mode is an ideal system for the determination of total mercury in coal, QAR-CRM-9.