TELEDYNE LEEMAN LABS
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Technical Note: Hg/ENV-1901

Coal Fly Ash (NIST 1633c)

Category: Environmental **Technique: CVAA**

Summary

This technical note describes the analysis of Coal Fly Ash (NIST 1633c) by EPA 7471B on the QuickTrace® M-7600 mercury analyzer. The analysis utilized a reduced internal diameter (ID) tubing for the stannous chloride ($SnCl_2$), sample and waste lines. This configuration reduces reagent and waste and is validated in Application Note: AN1905 – Green Chemistry: Decreased Reagent Consumption and Waste Using Reduced ID Tubing on the QuickTrace® M-7600 CVAA Mercury Analyzer (Viewable Here).

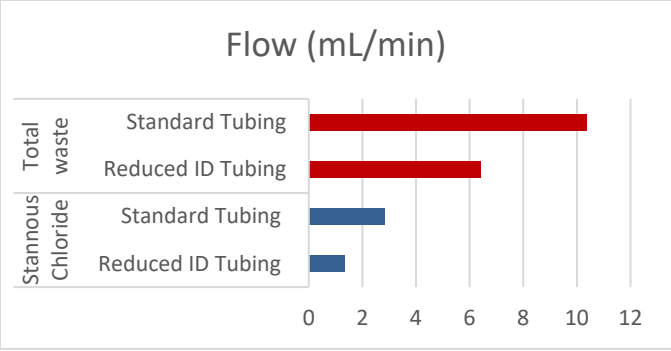
Instrumentation

Analyzer and ASX-560 autosampler for automated analysis. $SnCl_2$ pump tubing was orange/yellow 0.51 mm (TLL PN 15-4309-102). Sample and waste tubing were white/white 1.02 mm (TLL PN 15-4308-102).

Method Parameters

Parameter	Value
Sample Uptake (sec)	35
Rinse Time (sec)	80
Gas Flow (mL/min)	100
Pump Speed (%)	50
Replicate Read Time (sec)	1.5
Replicates	4

Sample flows were optimized by adjusting clamp pressure according to the *QuickTrace® M-7600 User Manual*. The reduced ID tubing (0.51 mm) used 53% less $SnCl_2$ than the standard tubing (0.76 mm). Total waste was reduced by ~40%.



Calibration

Six standards (0, 0.2, 1, 2, 5 and 10 µg/L) were digested and analyzed with the samples according to EPA 7471B. The calibration curve must be linear with a correlation of 0.998 or better. The calibration was verified with a digested 5 µg/L second-source standard with a ± 10% acceptance limit.

Sample Preparation

The sample weight average was ~0.2 g. Samples were digested on a graphite digestion block. 2.5 mL of DI water and 2.5 mL of aqua regia were added to each sample. Samples were heated at 95 °C for 2 min and then cooled for 5 min. 7.5 mL of 5% potassium permanganate ($KMnO_4$) and 25 mL of DI water was then added. Samples were agitated, loosely capped and heated for 30 min at 95 °C. After cooling, 3 mL of 12% hydroxylamine hydrochloride (NH_2OH HCl) was added to reduce the $KMnO_4$. Finally samples were brought to a final volume of 50 mL with DI water and mixed prior to analysis.

Procedure

Samples and standards were loaded onto the ASX 560. Inorganic mercury was reduced to elemental mercury by excess online addition of 10% $SnCl_2$ in 7% HCl.

Results

ICV (second source) - µg/L	4.76	95.2 % Recovery
	mg/Kg	
Hg in Coal Fly Ash (1633c) 1	1.020	
Hg in Coal Fly Ash (1633c) 2	1.011	
Hg in Coal Fly Ash (1633c) 3	1.018	
Hg in Coal Fly Ash (1633c) 4	1.005	
Hg in Coal Fly Ash (1633c) 5	1.014	
Hg in Coal Fly Ash (1633c) 6	0.996	
Hg in Coal Fly Ash (1633c) 7	1.008	
Avg	1.010 ± 0.006 @ 95 %	
STDEV	0.008	
MDL	0.020 @ 95 %	
Min	0.996	
Max	1.020	
CCV (second source) - µg/L	5.27	105.4 % Recovery

Conclusion

The QC recoveries of 95.2 to 105.4% demonstrate that the system is in control and stable for analysis of trace Hg in coal fly ash. The trace Hg certified value for 1633c is 1.005 ± 0.022 µg/g. The calculated MDL for the system under the method conditions is ≤0.02 µg/g with a confidence level of 95%.

Using the reduced ID pump tubing saved reagent consumption, decreased waste and was an ideal configuration for the determination of mercury in Coal Fly Ash (NIST 1633c).