Coal/Coal Fly Ash

Determination of Mercury in Coal Samples Using Direct Mercury Analysis

Although tougher laws and highly advanced mercury containment techniques have reduced the mercury emissions from coal fired plants, they continue to be one of the largest anthropogenic sources of mercury. The United Nations Environment Program's 2013 report mentioned that around 475 tons of mercury was released from coal burning activities in 2010. With a better understanding of the health hazards caused by long term mercury exposure, the focus on mercury analysis in the coal and mining industry has never been stronger.

Summary

With the combustion of coal in utility plants, mercury is released into the atmosphere. This atmospheric mercury combines with rain water and seeps down into our water bodies, where it enters our food chain. Exposure to high levels of mercury can cause harmful health effects including damage to the nervous system, chromosomal damage, allergic skin reactions and negative reproductive effects, making mercury analysis of coal very important.

Coal samples conventionally needed to be digested in acid prior to mercury analysis on instruments such as CVAA and ICP. Also, with coal being an extremely stable matrix, digestion was often challenging and required properly maintained digestion equipment, additional labor hours, expensive acids and waste disposal, which reduced the productivity of a lab.

Instrumentation

Direct mercury analysis, as described in EPA Method 7473, is a cost-effective, proven alternative to these labor-intensive, wet chemistry techniques. Direct Mercury Analysis involves an integrated sequence of combustion, catalytic conversion, amalgamation and detection using AA. Direct analysis affords the laboratory many benefits including:

- Reduced Sample Turnaround (6 Minutes)
- No Sample Preparation
- Reduced Hazardous Waste Generation
- Reduction of Analytical Errors
- General Cost Savings (70% vs. CVAA)

Milestone's DMA-80 provides a highly flexible platform with an extremely wide dynamic detection range to analyze coal samples with varying mercury levels. Additional features such as internal temperature monitoring, auto-blanking and pre-heated cuvettes ensure a complete and safe decomposition of the highly organic and oily samples without any contamination or memory effects.

The DMA-80 features a circular, stainless steel, interchangeable 40 position autosampler for virtually limitless throughput and can accommodate both nickel (500 mg) and quartz boats (1500 uL) depending on the requirements of the application. It operates from a single-phase 110/220V, 50/60 Hz power supply and requires regular grade oxygen as a carrier gas.

Calibration

The DMA-80 can be calibrated using aqueous standards or Standard Reference Materials (SRM). The DMA-80 used for this experiment had a tri-cell spectrophotometer and covered a dynamic range of 0.0015-1200ng Hg. Each cell was calibrated using different volumes of 1ppm and 0.1 ppm

stock solutions, prepared from an NIST traceable 1000ppm stock solution (VHG Labs).

Experiment and Results

In the first experiment, replicates of SRM 1633b (coal fly ash) were analyzed to validate the reproducibility and the accuracy of the system. The results are provided below:

Table 1. Summary of QA/QC Analysis (Coal Fly Ash)

Sample	Expected Conc. (ppm)	Concentration (ppm)
SRM 1633b	0.14+/-0.02	0.142
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Next, 5 different coal samples were each analyzed 4 times to study the concentration range of mercury in coal. The results obtained are shown below:

Table 2. Analysis of Unknown Coal Samples

Sample	Concentration (ppm)
A	0.15+/-0.02
B	0.148+/-0.02
C	0.039+/-0.02
D	0.1+/-0.02
E	0.1+/-0.02

The heating profile used for the complete decomposition of the sample matrix and the subsequent release of mercury was as follows:

Table 3. Profile

Step	Time (hh:mm:ss)	Temperature (°C)
1	00:00:30	200
2	00:01:00	250
3	00:02:00	750
4	00:01:30	750

Max Start Temperature: 200°C
Catalyst Temperature: 600°C
Purge Time: 60 seconds
Amalgamation Time: 12 seconds at 900°C
Oxygen Flow: 120 mL/min

Conclusion

A laboratory analyzing mercury in coal samples is required to maintain high throughput while keeping its costs under control. The DMA-80 is an excellent tool as it yields results in ~6 min/sample and proves to be proficient, matrix-independent and cost effective while completely eliminating the challenges of sample preparation posed by conventional mercury analysis techniques.

Learn more or request an onsite demonstration:
info@milestonesci.com or 1-866-995-5100