innovación tecnológica para laboratorio Rafer

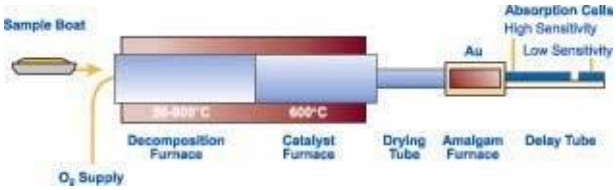
Determinación de Mercurio en Carbones y cenizas volantes
Analizador Hydra-II C

Introduction

The level of mercury found in coal can vary dramatically. Using clean fuels may be critical in satisfying emission requirements for the Mercury Rule of the Clean Air Act (CAA). Also, the monetary value of the fly ash residue resulting from the combustion of coal may be inversely proportional to its mercury content. The Hydra-C Direct Mercury Analyzer provides fast, simple and convenient analyses of these materials without sample pretreatment or production of hazardous chemical waste. A typical sample analysis takes about 5 minutes. The Hydra-C employs U.S. EPA Method 7473 which has been approved for both laboratory and field analysis.

Instrumentation

The Hydra-C (shown to the right) is fully automated for unattended operation. It comes complete with a 70 position autosampler that has on-the-fly loading capability for virtually unlimited sampler capacity. Hydra-C operates from a single 110/220V, 50/60 Hz power supply and oxygen supplied at 15-20 psig. All instrument operating parameters (e.g. furnace temperatures, gas flows, autosampler control) and process stages are computer controlled for ease-of-use. The figure below Hydra-C Mercury Analyzer shows the system design with gas flow from left to right.



Principle of Operation

A weighed sample is introduced into the decomposition furnace, oxygen begins to flow over the sample and the furnace temperature is increased in two stages; first to dry the sample, then to decompose it.

A small amount of sample (typically 0.05 to 1 gram) is weighed and deposited into a sample boat. The weighed sample is then introduced into the Hydra C where oxygen begins to flow over the sample. The decomposition furnace temperature is then increased in two stages; first to dry the sample, then to decompose it. The evolved gases are carried through a heated catalyst to produce free mercury while removing halogens, nitrogen oxides, and sulfur oxides. The remaining combustion products including elemental mercury (Hg^0) are swept through a gold amalgamation trap where elemental Hg is trapped and concentrated. After the amalgamation step, the trap is heated to release the mercury into a carrier gas which transports it into an atomic absorption spectrometer.

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Experimental

Table I shows the instrument parameters employed for coal and fly ash. For this analysis nickel boats were used for all samples.

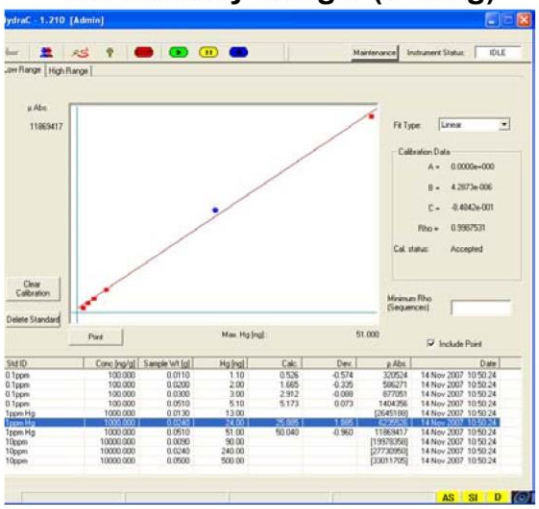
Table I: System Parameters

Parameter	Setting
Dry	300°C for 45 sec.
Decomposition	800°C for 150 sec.
Catalyst	600°C
Catalyst Wait Period	60 sec.
Gold Trap	700°C for 30 sec.
Measurement	90 sec.
Oxygen Flow	300 ml/min

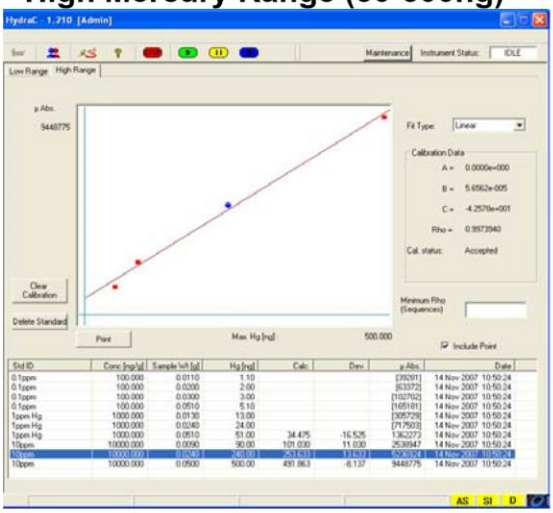
Calibration

Calibration was completed using aqueous standards prepared in 10% HNO_3. Working standards were blank, 0.1, and 1.0 ppm at several different injection weights. The calibration curve plot microabsorbance vs total mercury injected.

Low Mercury Range (0-50ng)



High Mercury Range (50-500ng)



Results

Two certified reference materials were analyzed for mercury and the results appear below in Table II.

Sample Matrix	CRM Name	Certified Concentration (PPM)	Measured Concentration (PPM)
Coal	HC-35150	0.176	0.177
Fly Ash	1633b	0.143	0.132

Conclusion

Results obtained for coal and fly ash showed excellent correlation with certified values and were obtained without any sample treatment in about 5 minutes per sample.