Hydra-C Application Note 1057

The Determination of Mercury in Waste Oil Using the *Hydra-C* Mercury Analyzer

Introduction

It is important to determine the concentration of mercury in waste oil prior to disposal. Any waste oil containing more than 0.2 mg/L mercury is characterized as hazardous waste and cannot be burned for energy recovery (Code of Federal Regulations 40 CFR 261.24). The ***Hydra-C Direct Mercury Analyzer*** provides fast, simple and convenient analysis of this material without sample pretreatment or production of hazardous chemical waste. Typical analysis takes about 5 minutes per sample. ***Hydra-C*** employs EPA Method 7473 which is approved for both laboratory and field analysis for mercury in solids and solutions using Thermal Decomposition, Amalgamation and Atomic Absorption Spectroscopy.

Instrumentation

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

Hydra-C Mercury Analyzer

Principle of Operation

Hydra-C operates on the principle of thermal decomposition to liberate elemental mercury from solid or liquid samples. Figure 1 shows a schematic diagram of Hydra-C's principle of operation. First, a weighed sample is deposited into a sample boat and introduced into the decomposition furnace. After the furnace is closed, an oxidant (typically oxygen or compressed air) begins to flow over the sample and the furnace temperature is ramped in two stages; first to dry the sample, then to decompose it. The analytical process typically involves combusting (thermal decomposition) the sample at high temperatures with oxygen; although, for some applications gentle heating of the sample in air is adequate to release the mercury.

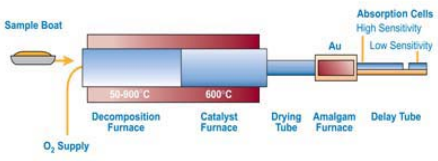

Figure 1: Hydra-C Principle of Operation

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During the combustion step 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) are swept first through a dryer and then through a gold amalgamation trap where all elemental mercury is captured. Following the decomposition step, the amalgamation trap is heated and the free mercury is carried into an atomic absorption spectrometer. The mercury level is reported using a wide dynamic range detection system that operates from 0.005 ng (its detection limit) to its upper limit of 1000 ng. For applications requiring significantly higher detection capability an optional high range detection system is available which can be used to analyze samples containing up to 20,000 ng of Hg.

Experimental

Table I shows the instrument parameters employed for the analysis of waste oil. For these analyses nickel boats were used for all samples and standards and air was used as the oxidant gas.

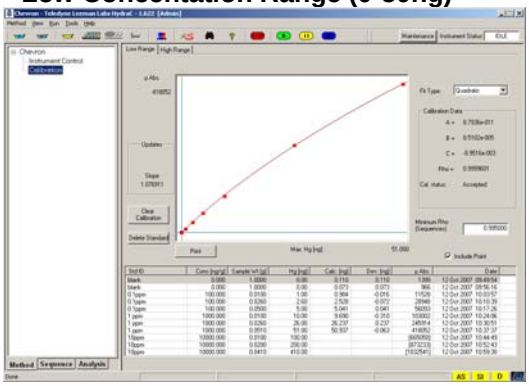
Table I: System Parameters

Parameter	Setting
Dry	100°C for 30 sec.
Decomposition	800°C for 200 sec.
Catalyst	600°C
Catalyst Wait Period	60 sec.
Gold Trap	600°C for 30 sec.
Measurement	100 sec.
Air Flow	350 ml/min

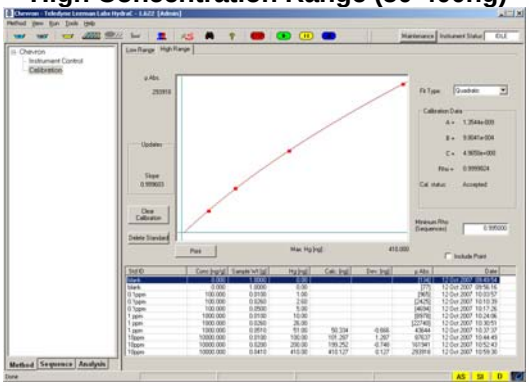
Calibration

The calibration curves were created by injecting differing masses of Conostan 100 ppm mercury in base oil.

Low Concentation Range (0-50ng)



High Concentration Range (50-400ng)



Results

Several reference materials were analyzed for mercury and the results appear below in Table 2.

Table 2: Reference Materials

Sample	TLL Average (ppb)	TLL Std Dev (ppb)	Certified (ppb)	% Recovery
Oil1	524.9	8.59	520	100.9
Oil2	733.4	10.8	720	101.9
Oil3	521.9	11.2	520	100.4
Oil4	2.030	0.61	NA	NA
Oil5	917.2	10.7	920	99.7
Oil6	317.7	5.6	320	99.3

Conclusion

Results obtained for oils showed excellent correlation with certified values and were obtained without any sample treatment in about 5 minutes per sample. The Hydra-C provides a fast and simple way to qualify oils for energy recovery.