U6000AT+ Application Note

Determination of Wear-Metals in Lubricating Oil Using Ultrasonic Nebulization / Membrane Desolvation with ICP-AES Detection

Lubricating oils are essential for the proper function of a wide variety of machines, particularly engines used in automobiles, trucks, aircraft, and construction and agricultural equipment. Monitoring the levels of wear metals in lubricating oils from various engine components facilitates proper maintenance of the equipment and can help prevent costly repairs.

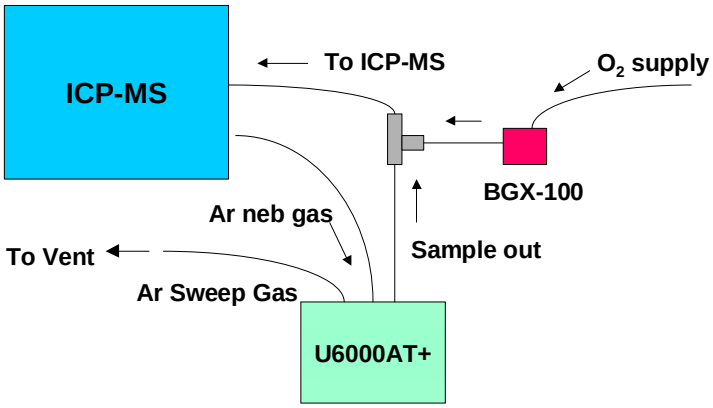
Inductively coupled plasma atomic emission spectrometry (ICP-AES) is a widely used technique for parts per billion (ppb) and sub parts per billion detection in a wide variety of sample matrices. However, introduction of organic solvents to an ICP-AES instrument can present a number of problems: plasma instability or failure, elevated background emission, and carbon buildup on the ICP torch and injector.

The CETAC U6000AT+ Ultrasonic Nebulizer / Membrane Desolvator removes most of the diluting organic solvent during analyte transport to the ICP-AES instrument, allowing stable plasma operation. In addition, the U6000AT+ can help improve analyte sensitivity after dilution.

The CETAC BGX-100 Blend Gas Accessory is used to add a low-flow of oxygen (approx. 10 mL/min) between the U6000AT+ and the ICP-AES; this prevents any buildup of carbon on the ICP torch. A block diagram shows the experimental setup.

U6000AT+ Ultrasonic Nebulizer

Block Diagram



Sample Preparation

The sample analyzed is NIST SRM 1085a, Wear-Metals in Lubricating Oil. A sample of 1.5 grams of the sample was accurately weighed and diluted with a spectrophotometric grade of toluene. The dilution was measured to approximately 50 grams total (sample plus diluent) and accurately recorded.

Operating Conditions

ICP-AES: Thermo Iris Intrepid II XSP
Plasma power: 1250 W
Nebulizer gas pressure: 25 psi
Integration time: 10s low wavelength range, 5s high wavelength range

CETAC U6000AT+:
Heater temp: 110^{0}C
Cooler temp: -10^{0}C
Membrane oven temp: 160^{0}C
Sweep gas flow: 2.00 L/min
Uptake rate: 1.0 mL/min
Inlet pump tubing: Viton™
Drain pump tubing: Viton™
Oxygen rate (BGX-100): 20 mL/min

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Calibration

The ICP-AES instrument was calibrated using a 2-point calibration curve; no internal standard was used. Standards were prepared using the Conostan S-21 organometallic standard diluted with toluene.

Results

Analytical results for 12 metals are tabulated below. Measured values are the average of 6 replicate runs of SRM 1085a; uncertainties are based upon a 95% confidence interval (2σ). Excellent agreement was obtained with certified values. Note that the value in parenthesis for Al is not certified but given for reference information.

Element	Wavelength (nm)	Measured (ug/g)	Certified (ug/g)
Al	167.081	292.4 +/- 6.4	(289)*
Ag	328.068	301.8 +/- 6.8	305.7 +/- 5.8
Cr	283.563	295.6 +/- 6.9	296.3 +/- 3.3
Cu	324.754	297.9 +/- 6.1	295.1 +/- 6.8
Fe	259.940	297.9 +/- 6.1	296.8 +/- 2.7
Mg	279.553	292.3 +/- 11.9	296.0 +/- 3.1
Mo	204.598	304.2 +/- 4.8	302.9 +/- 4.1
Ni	231.604	300.3 +/- 8.8	302.9 +/- 6.8
Pb	220.353	292.6 +/- 10.9	297.4 +/- 9.6
Sn	189.989	299.5 +/- 17.5	296.0 +/- 13.4
Ti	334.941	301.6 +/- 13.4	305.1 +/- 10.0
V	309.311	293.1 +/- 9.6	292.4 +/- 9.9