SHIMADZU

Application News

No.J95

Inductively Coupled Plasma Atomic Emission Spectrometry

Analysis of Additive Elements in Lubricating Oil Using ICPE-9000

Additives consisting of various types of organic metal substances are added to lubricating oils to enhance performance. They include detergents (to prevent and inhibit the deposition of degradation products formed during high-temperature operation of engines, etc.), antioxidants (which react with free radicals and peroxides to suppress the formation of varnish and sludge that originate during oxidation of oil), corrosion inhibitors (to neutralize the corrosive oxidation products formed due to degradation of the lubricant), solid lubricants (used as powder or thin film to reduce friction and wear), etc. Analysis of these additive elements is important for quality management of lubricating oils, and ICP emission spectrometry is one of the effective methods available for this analysis.
Here, using the ICPE-9000 multitype inductively coupled plasma emission spectrometer, we conducted analysis of additive elements in commercially available engine oil, ATF oil and gear oil. The ICPE-9000 can simultaneously measure multiple elements, and quickly analyze many additive elements.

Samples

· Engine oil (3 types)
· ATF oil (1 type)
· Gear oil (2 types)

Sample Preparation

Each sample was weighed out to 1 g, diluted with 100 mL of xylene (100-to-1 dilution), and then measured. In the case of sample 6 (gear oil), a 10 mL aliquot of the 100-to-1 dilution was further diluted with 50 mL of xylene (500 to 1 dilution) to serve as the measurement sample. When diluting the samples, Y (yttrium) was added to the sample solutions at the rate of 1 mg/L to be used as an internal standard element. Furthermore, the 100-to-1 dilution of sample 1 (engine oil) was again diluted by 5 to 1 to prepare a very concentration solution (500-to-1 dilution). The standard samples were prepared by appropriately diluting the SPEX oil base multi-element mixture of 21elements (900 ppm, 500 ppm) and the CONOSTAN oil base single element standard solutions (5000 ppm), respectively, with xylene. As with the measurement samples, Y (yttrium) was added at 1 mg/L.

Analysis

Quantitative analysis of the lubricant samples was conducted by the calibration curve-internal standard method using the ICPE-9000.
Regarding sample 1 (engine oil), the dilution test was conducted using the 100-to-1 and 500-to-1 diluted samples. The following expression was used for the dilution test.

Dilution test value (%) = I /S × 100
(I: 100-to-1 dilution sample quantitation value,
S: 500-to-1 dilution sample quantitation value × 5)

Analytical Conditions

Instrument	: ICPE-9000
Radio Frequency Power	: 1.4 kW
Plasma Gas Flowrate	: 16 L/min
Auxiliary Gas Flowrate	: 1.4 L/min
Carrier Gas Flowrate	: 0.70 L/min
Sample Introduction	: Nebulizer 10
Spray Chamber	: Co-axial chamber
Plasma Torch	: Torch
Observation	: Radial

Analytical Results

Table 1 shows the quantitative analysis results. The dilution test value using sample 1 (engine oil) was excellent, achieving nearly 100 %.
In addition, highly stable test results over 3 hours were obtained using sample 1 (engine oil), as shown in Fig. 1. Excellent repeatability was obtained for all elements in the measurements, repeated over a period of 3 hours. An RSD (relative standard deviation) of 1 % or less was obtained for each element. The plasma torch utilized in Shimadzu's ICP emission spectrometer provides measurement with extremely low precipitation of carbon, eliminating the necessity to introduce oxygen as a means of suppressing carbon precipitation even when measuring organic solvent samples. The stable results obtained in the analyses presented here also were achieved over a long period of time without introducing oxygen.
Fig. 2 shows spectral profiles of Ca and Mo.
Fig. 3 shows the calibration curves for Ca, Mo, and Zn.

Table 1 Quantitative Analysis Results for Lubricating Oils (µg/g)

	Engine Oil			ATF Oil	Gear Oil	
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
B	113 (101)	83	9.1	68	18	2040
Ca	1070 (96)	2920	1720	165	67	73
Mg	737 (99)	19	5.2	1.0	1.7	5.9
Mo	84 (103)	96	60	–	–	–
P	604 (100)	696	682	278	984	3320
Zn	699 (104)	841	770	19	8.6	2640

The values in parentheses for sample 1: Dilution test value (%) = I / S × 100
(I: quantitation value prior to sample dilution, S: Quantitation value × 5 of 5-to-1 diluted sample)

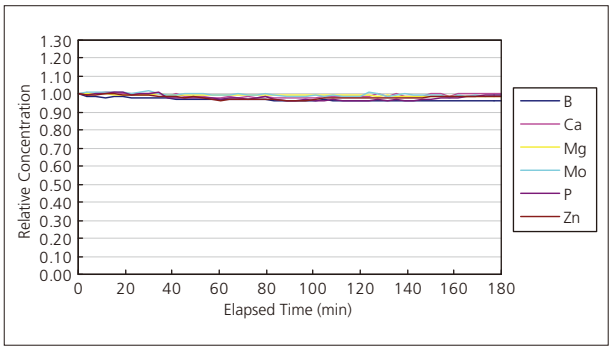

Fig. 1 Variation with Time Over 3 Hours (Sample1: Engine Oil)

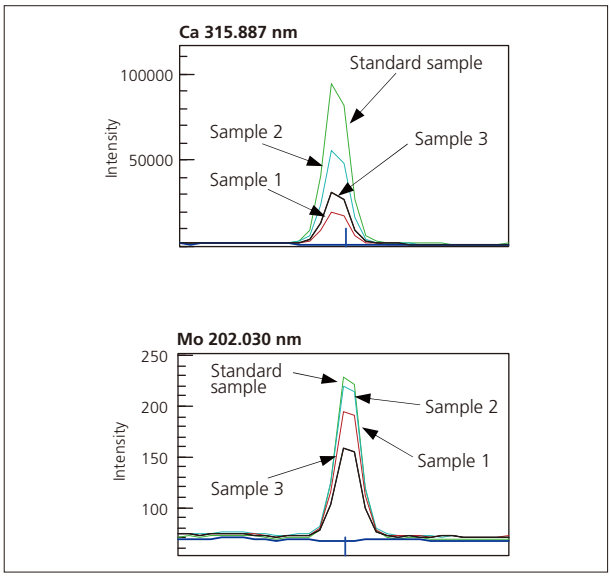

Fig. 2 Spectral Profiles of Ca and Mo

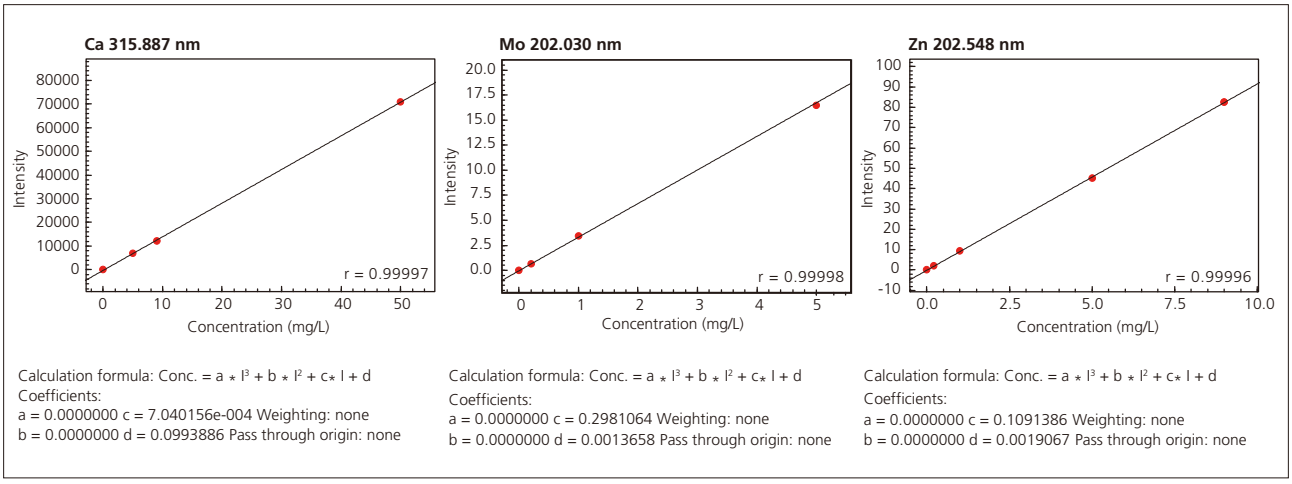

Fig. 3 Calibration Curves for Ca, Mo and Zn

First Edition: March, 2012

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