Application Note

Oil Analysis

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1 Introduction

This Application Note describes a classical method for the analysis of lubricating oils. The spectrometric analysis of lubricating oil is common practice where large amount of lubricating oils are involved or where engine wear is critical. For example, it is used to control oil from planes, boats, buses, trains, and large automotives. The same oil is sampled over a period of time and analyzed, and by monitoring of the concentrations of the wear metals enables identification engine component suffering wear.

2 Principle

2.1 Technique used

The elemental analysis of solutions was undertaken by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). The sample is nebulized then transferred to an argon plasma. It is decomposed, atomized and ionized whereby the atoms and ions are excited. We measure the intensity of the light emitted when the atoms or ions return to lower levels of energy. Each element emits light at characteristic wavelengths and these lines can be used for quantitative analysis after a calibration.

2.2 Wavelength choice

The choice of the wavelength in a given matrix can be made using the "profile" function, or by using Win-IMAGE, which is rapid semi-quantitative analysis mode using multiple wavelengths. The principle is the same in either case: record the scans of analytes at low concentration, and of the matrix. By superimposing the spectra, we see possible interferences.

3 Sample Preparation

Different oil samples can have a large variation in their viscosity, ranging from 50 to 1000 cs. So the nebulization efficiency of the oil can be very different from one oil sample to the next. To decrease the viscosity and to obtain the same viscosity value for all samples, they were diluted by 10 in kerosene, white spirit, xylene or kerdane. For viscous oils of 1000 cs, a 100 dilution is required.

4 Instrument specification

The work was done on a JY 2000.The specifications of this instrument are listed below in Tables 1 and 2.

Table 1: Specification of spectrometer

Parameters	Specifications
Mounting	Czerny-Turner
Focal length	0.64 m
Grating number of grooves	2 400 gr/mm
Variable resolution	No
Nitrogen purge	Yes

Table 2: Specification of RF Generator

Parameters	Specifications
Observation	Radial
Frequency	40.68 MHz
Control of gas flowrate	By computer
Control of pump flow	By computer
Cooling	Air

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5 Operating conditions

The operating conditions are listed in Table 3 below.

Table 3: Operating conditions

Parameter	Condition
RF Generator power	1200 W
Plasma gas flowrate	14 L/min
Auxiliary gas flowrate	0.8 L/min
Sheath gas flowrate	0.3 L/min
Nebulizer flowrate	2.51 bars
Sample uptake	0.8 mL/min
Type of nebulizer	JY 0.7
Type of spray chamber	Scott
Argon humidifier	No
Injector tube diameter	3.0 mm

6 Wavelength selection and analytical conditions

Table 4: Wavelength selection and analytical conditions

Element	Measured points/ calculated points	Analysis mode	Integration time (sec)
Ag	7/5	Gaussian	0.2
Al	7/5	Gaussian	0.1
B	7/5	Gaussian	0.1
Ca	7/5	Gaussian	0.1
Cr	7/5	Gaussian	0.1
Cu	7/5	Gaussian	0.1
Fe	7/5	Gaussian	0.1
Mo	7/5	Gaussian	0.1
Pb	7/5	Gaussian	0.1
Si	7/5	Gaussian	0.1
Sn	7/5	Gaussian	0.2
Zn	7/5	Gaussian	0.1

Table 5: High calibration standard

Element	High calibration point (ppm)
Ag	10
Al	50
B	100
Ca	5000
Cu	150
Cr	100
Fe	200
Mo	100
Pb	150
Si	50
Sn	10
Zn	100

7 Limits of detection

The limits of detection are calculated using the following formula:

$$LOD = k \times BEC \times RSD_0$$

With:
LOD = limit of detection,
k = 3 for the normal 3-sigma values,
BEC = Background equivalent concentration,
RSD_0= relative standard deviation of the blank.

To calculate the LOD, a calibration curve is constructed using two points, 0 ppm and 5 ppm, or some concentration where the calibration is linear; this gives the BEC. The RSD_0 is evaluated by running the blank ten times.

Typical detection limits in a solution of 10 % oil in kerosene are shown in Table 6 below.

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Table 6: Limits of detection

Element	Wevelength (nm)	LOD (ppb)
Ag	328.068	1.4
Al	167.020	2.3
Al	308.215	6.5
Al	396.152	9.9
B	249.773	4.5
Ba	455.403	0.2
Ca	317.933	1.5
Cd	228.802	1.3
Cu	324.754	0.7
Mg	279.553	0.5
Mn	257.610	0.5
Mo	202.030	2.0
Na	588.995	4.9
Ni	231.604	0.9
P	178.229	14.4
Pb	220.353	20.2
Si	251.611	3.4
Sn	189.989	14.7
Ti	334.941	1.1
V	292.402	0.8
V	310.230	0.5
Zn	213.856	1.0

8 Conclusion

Because the aim of the analysis lubricating oils is to monitor the oil of an engine and get a trend analysis over time, the best accuracy is not required. The trend analysis allows the analyst to see that the oil is changing and when the engine needs stripping down before a catastrophic failure. The use of the internal standard is not required because the results are only compared to previous results. The procedure is thus very simple: new two point calibration each time the sample is analyzed and dilution of the samples by 1:5, 1:10 or 1:100, depending on the viscosity of the oil samples and whether lower detection limits are required.

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