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Analysis of oils
ARL OPTIM'X WDXRF Simultaneous-Sequential Spectrometer

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ARL OPTIM'X, lubricants, oils, X-ray fluorescence

ARL OPTIM'X XRF spectrometer

Introduction
X-ray fluorescence allows the measurement of key elements already present in or added to oils, in the parts per million to percentage level concentration range.

Instrumentation
The Thermo Scientific ARL OPTIM'X is a wavelength dispersive XRF instrument designed for ease of use with minimal running and maintenance costs. Its optimized geometry coupled with its low power Rh anode X-ray tube provides the high sensitivity required for this demanding application. The spectral resolution achieved by the ARL OPTIM'X is about 10 times better than any EDXRF instrument.

It also provides superior precision, as well as short and long term stability. Additionally the instrument does not require external nor internal water cooling.

The SmartGonio™ of the ARL OPTIM'X can measure all elements from F (Z=9) to U (Z=92) - except Ru, Rh, Pd, Ag and Cd as these elements suffer interference from the Rh lines of the X-ray tube. Two fixed channels can be fitted alongside the SmartGonio™ to reduce the total analysis time or to get exceptional results on two selected elements. Alternatively the ARL OPTIM'X can be equipped with 8 fixed channels in a simultaneous configuration.

The ARL OPTIM'X includes an option to switch environment from vacuum to helium which allows the analysis of liquids.

Ease of operation is obtained through OXSAS, our modern, powerful and user-friendly software supporting instrument operation and data handling..

Sample preparation
Using WDXRF, oil samples can be analyzed directly as received without dilution. Sample preparation involves simply pouring the oil samples (containing up to 50 ppm or more of the elements of interest) directly into special liquid cells fitted with 4 µ polypropylene film as shown in the above picture.

Analytical procedure
Sensitivities and limits of detection are calculated from the resulting calibration curves, using the base oil as a blank. In practice, the counting time (analysis time) chosen for each element depends on the precision and throughput required. In Table 1, limits of detection have been calculated for 120 seconds counting time.

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Results

The elements measured, crystals and detectors combinations used, Standard Error of Estimate (SEE) and Limits Of Detection (LOD) achieved are shown in Table 1.

Tables 2 and 3 below show the results of a reproducibility test obtained by performing seven consecutive analyses on a sample. For each repeat, a new liquid cell was prepared. Fixed channels for sulphur (S) and lead (Pb) have been used with a counting time of 120 seconds. The results show the excellent precision obtained for S and Pb with the ARL OPTIM'X using only 50W power.

Element	SmartGonio configuration	SEE [ppm]	SmartGonio LOD (ppm)	Fixed Channel LOD [ppm]
Mg	AX06/FPC	1.7	8	8
Al	PET/FPC	1.8	4.2	3.1
Si	PET/FPC	2.1	4	3.2
P	PET/FPC	0.7	2	1.5
S	PET/FPC	0.7	1.7	1.2
K	LIF200/FPC	0.9	1.4	n.m.
Ca	LIF200/FPC	n.a.	1.5	1.7
V	LIF200/FPC	1.1	1	n.m.
Cr	LIF200/FPC	n.a.	1	n.m.
Mn	LIF200/FPC	n.a.	1	n.m.
Fe	LIF200/FPC	n.a.	1.1	0.8
Ni	LIF200/SC	0.7	0.6	n.m.
Cu	LIF200/SC	n.a	0.8	n.m.
Zn	LIF200/SC	n.a.	0.6	n.m.
Pb	LIF200/SC	1.2	1.7	1

Table 1. Analytical results using 50W power.

FPC: Flow proportional counter
SC: Scintillation counter
SEE: Standard error estimate = a measure of accuracy

LOD: Limit of detection = 3 (BEC/Qt)
n.a.: not available as only two samples were available for this element

n.m.: not measured as this fixed channel was not fitted on the test instrument.

CELL No	S [ppm]
S1	25.4
S2	25.7
S3	26.4
S4	26.0
S5	25.0
S6	25.9
S7	26.7
Average	25.9
Std.Dev.	0.6

Table 2. Reproducibility test on S analysis - 7 new liquid cells filled with the same sample.

CELL No	Pb [ppm]
Pb1	5.1
Pb2	4.9
Pb3	4.0
Pb4	4.0
Pb5	4.1
Pb6	4.8
Pb7	5.6
Average	4.7
Std.Dev.	0.6

Table 2. Reproducibility test on S analysis - 7 new liquid cells filled with the same sample.

Concentration values in ppm
Average: Average [ppm]
Std.Dev.: Standard deviation (1 sigma) [ppm]

Conclusion

Limits of detection at ppm level are achieved by the ARL OPTIM'X WDXRF instrument for most elements found in oil samples. High reproducibility can be obtained even for elements present at low concentration levels.