SRS 3400
THE ANALYSIS OF TRACE METALS IN OIL

Background

For many years X-ray fluorescence spectrometry (XRF) has been used as a routine laboratory tool in the refinery and petrochemical industries.

A diverse range of applications has emerged for this established technique. These include simple, single element calibrations such as sulphur in diesel and lead in gasoline or more complex methods for the analysis of sulphur, nickel and vanadium in fuel oils and additives in lubricating oils.

In recent times more glamorous uses of XRF have been developed. Formula 1 and Touring Car racing teams now regularly monitor the levels of wear metals generated by their vehicles engines. A sharp rise in a particular metal concentration gives an early warning of possible component failure. This saves further damage to the engine and the high costs of replacement.

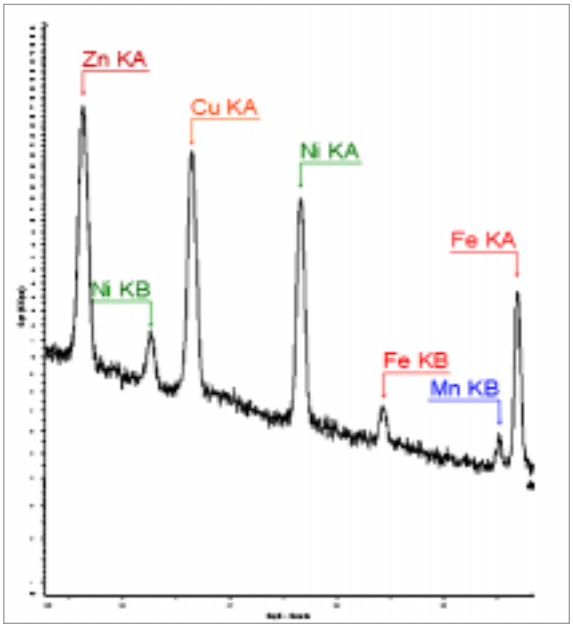

Figure 1. Typical Spectrum of Metals in Oil

Instrumental

The BRUKER AXS SRS 3400 (shown above) is a high performance Sequential Wavelength Dispersive XRF spectrometer. It is fited with a 4kW end window Rhodium X-ray tube, up to ten primary beam filters, up to four collimators and up to eight analysing crystals.

Flow and scintillation counters can be used either independently or in tandem. All of this is coupled with BRUKER's superb goniometer technology.

A wide range of sample changers is available, from 1 to 200 positions, and these can be directly linked into automated sample handling systems.

Software

Perhaps the most outstanding feature of the SRS 3400 analytical system is the inclusion of the SPECTRA^{plus} software package. This is a fully interactive program that uses the Windows NT platform for maximium speed and stability.

It comes complete with a standardless analysis routine, a rapid data retrieval system and full networking capability.

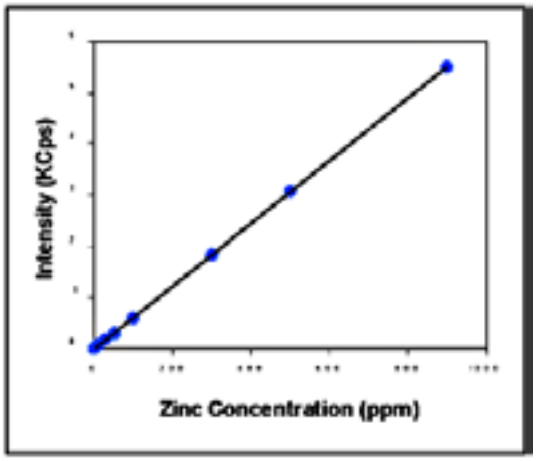

Figure 2. Zinc Calibration Curve

Analyte	Counting Time	Detection Limit
Na	120 sec.	10.0 ppm
P	20 sec.	0.5 ppm
Ca	20 sec.	0.4 ppm
V	40 sec.	0.5 ppm
Cr	30 sec.	0.5 ppm
Fe	10 sec.	0.5 ppm
Cu	10 sec.	0.5 ppm
Zn	10 sec.	0.5 ppm
Mo	20 sec.	0.5 ppm
Ba	75 sec.	2,0 ppm
Pb	30 sec.	0.5 ppm

Table 1. Examples of Detection Limits for this Application

Calibration and Results

This application note details the analysis of wear metals in oil samples.

The instrument was calibrated using eight certified reference standards. These standards contained twenty one trace metals with a concentration range of zero to nine hundred parts per million (ppm).

Minimal sample preparation is required. The standards were simply poured into Bruker liquid sample cups fitted with 1.5mm polyester film. The analysis took place in a helium path. The zinc calibration line is shown in figure 2.

The precision data quoted was collected from ten analyses. Long term stability data is available upon request.

Analyte	Calibration Accuracy	Precision R.S.D.
Na	18.2 ppm	9.4 %
Mg	7.6 ppm	6.9 %
Al	4.0 ppm	3.2 %
Si	4.6 ppm	3.4 %
P	6.0 ppm	1.8 %
Ca	2.9 ppm	2.8 %
Ti	2.2 ppm	<1.0 %
V	1.7 ppm	1.0 %
Cr	1.9 ppm	1.1 %
Mn	1.9 ppm	0.8 %
Fe	1.6 ppm	0.9 %
Ni	1.8 ppm	1.0 %
Cu	1.4 ppm	0.8 %
Zn	1.3 ppm	0.8 %
Mo	3.3 ppm	0.8 %
Ag	3.8 ppm	1.7 %
Sn	3.3 ppm	1.8 %
Ba	2.2 ppm	<1.0 %
Pb	1.1 ppm	1.0 %

Table 2. Further details of calibration and instrument data

Conclusion

This application note has summarised the outstanding performance of the BRUKER SRS 3400 in the field of wear metal analysis.

Please contact Bruker AXS for other XRF applications in the petrochemical industry.

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