APPLICATION NOTE

Analysis of wear metals in oil under ambient air using the ARL QUANT'X EDXRF

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Background

Monitoring the presence of wear metals in lubricants, oils and other machine fluids is a preventive measure to identify potential problems with an engine. Early detection of wear metals in lubricants and greases avoids expensive damage and downtime to high value engines, gears, generators, turbines and other equipment.

Instrument

The ARL QUANT'X EDXRF spectrometer used for this application is equipped with a 50 kV, 50 W silver target X-ray tube and a silicon drift detector (SDD) of the latest generation. The ARL QUANT'X employs primary filtered radiation to excite the sample. A set of nine filters specifically designed to optimize the peak-to-background for elements from F to Am ensures that the ARL QUANT'X is easily adaptable per application or element range.

Sample preparation

The oil sample is measured as such by transferring 3 grams of product into a sample cup of 32 mm outer diameter, sealed with a 4 micron polypropylene film.

Excitation conditions

Table 1 below shows the excitation condition used to perform the analysis. Three different filter conditions have been used to cover 14 elements. A live time of 100 s is used for every condition. Figure 1 shows a typical spectrum obtained using condition Mid Zc of a sample containing several elements of interest at 50 ppm. Condition Mid Zc is optimized to excite copper, zinc and lead but also excites many of the other wear metals typically monitored.

Calibration

Linear calibration curves relating net intensities to concentrations are set up using standards prepared using a Conostan S21 50 ppm standard, diluted using 75 cSt blank oil (also from Conostan). Three concentration levels; 10 ppm, 20 ppm, 50 ppm and a blank were prepared to set up the curves and to determine the detection limits. Figures 2a and 2b show the calibration curves obtained for titanium (Ti) and nickel (Ni). Root mean square errors (RMSE) of 0.6 ppm (Ti) and 0.2 ppm (Ni) are obtained. Table 2 also shows the RMSE values obtained for the other elements of interest.

Limit of detection

To determine the limit of detection (LoD), 10 XRF cups were filed with 3 grams of the blank oil. The LoD is calculated to be three times the standard deviation of the concentrations derived for each element. Table 2 shows the results.

Conclusion

The unique capabilities of the ARL QUANT'X EDXRF spectrometer provide fast, accurate and repeatable monitoring of wear metals in lubricants, oils and machine fluids. Analysis of such samples can be done under ambient air which is convenient, shortens analysis time and offers savings on costly helium gas consumption.

Table 1: Excitation condition used for wear metals in oil

Condition	Filter	Voltage (kV)	Current (mA)	Atmosphere	Live Time (s)	Analytes
Mid Za	Thin Ag	18	Auto	Air	100	Ti, V, Cr, Mn, Fe, Ni, Ba
Mid Zc	Thick Ag	30	Auto	Air	100	Cu, Zn, Pb
High Zb	Thick Cu	50	Auto	Air	100	Mo, Ag, Cd, Sn

Figure 1: Typical spectrum for wear elements at 50 ppm in oil using condition Mid Zc (30 kV, thick Ag filter) in an air atmosphere

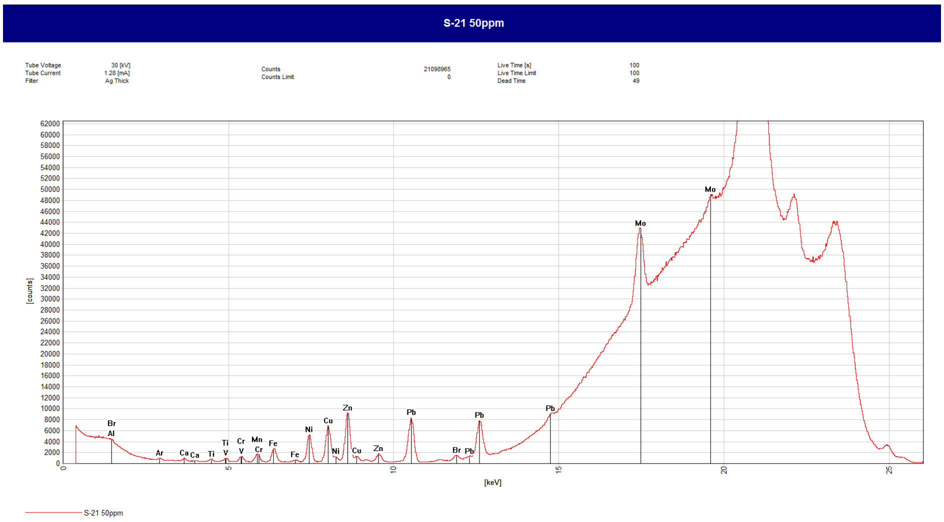


Table 2: Concentration range, RMSE and LoD values for wear metals in oil

Element	Line	Conc. range [ppm]	RMSE [ppm]	LoD, 100s live time [ppm]
Ti	Kα	0 - 50	0.59	2.3
V	Kα	0 - 50	0.03	0.7
Cr	Kα	0 - 50	0.48	0.5
Mn	Kα	0 - 50	0.51	0.3
Fe	Kα	0 - 50	0.03	0.2
Ni	Kα	0 - 50	0.21	0.1
Cu	Kα	0 - 50	0.05	0.2
Zn	Kα	0 - 50	0.06	0.1
Mo	Kα	0 - 50	0.22	0.3
Ag	Kα	0 - 50	0.35	0.5
Cd	Kα	0 - 50	0.79	0.7
Sn	Kα	0 - 50	0.85	1.3
Ba	Lα	0 - 50	0.95	2.0
Pb	Lα	0 - 50	0.12	0.2

Figure 2a: Calculated versus given concentrations in the case of titanium (Ti)

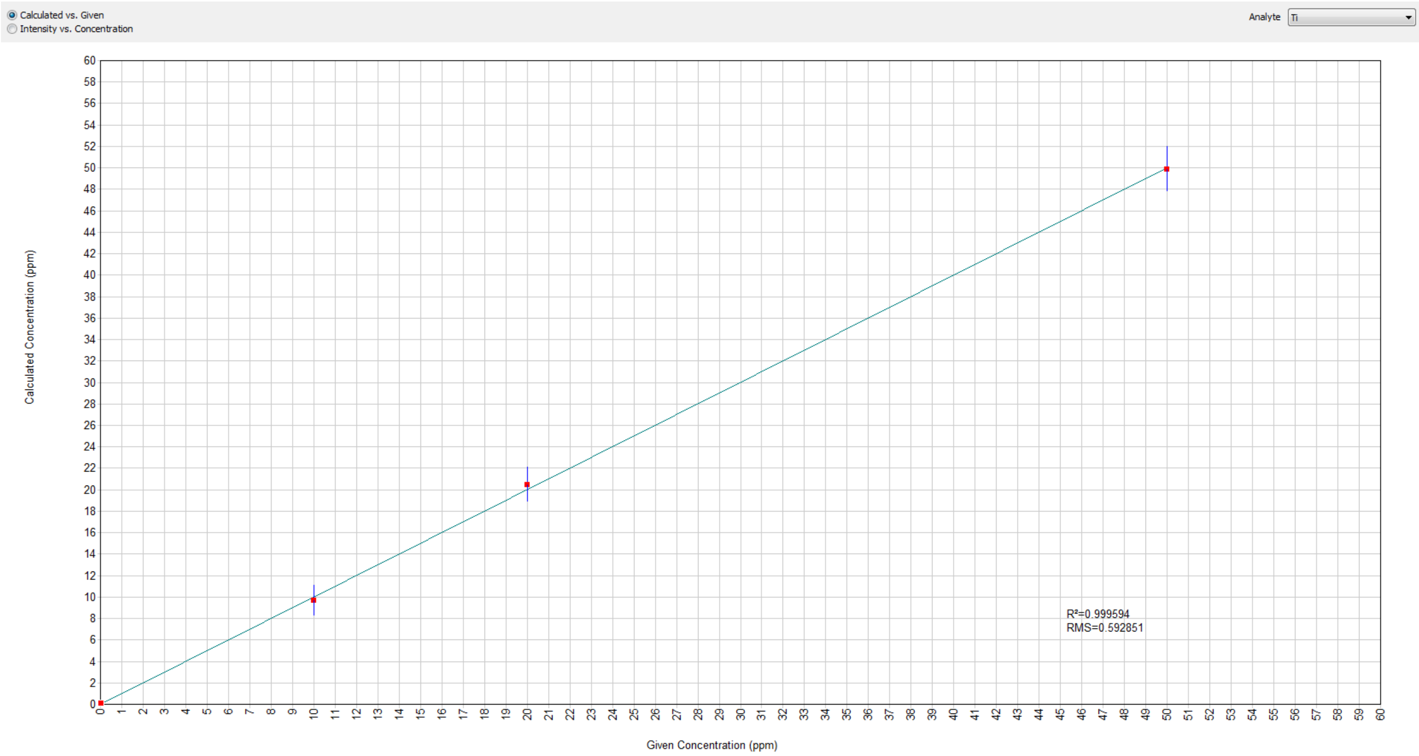
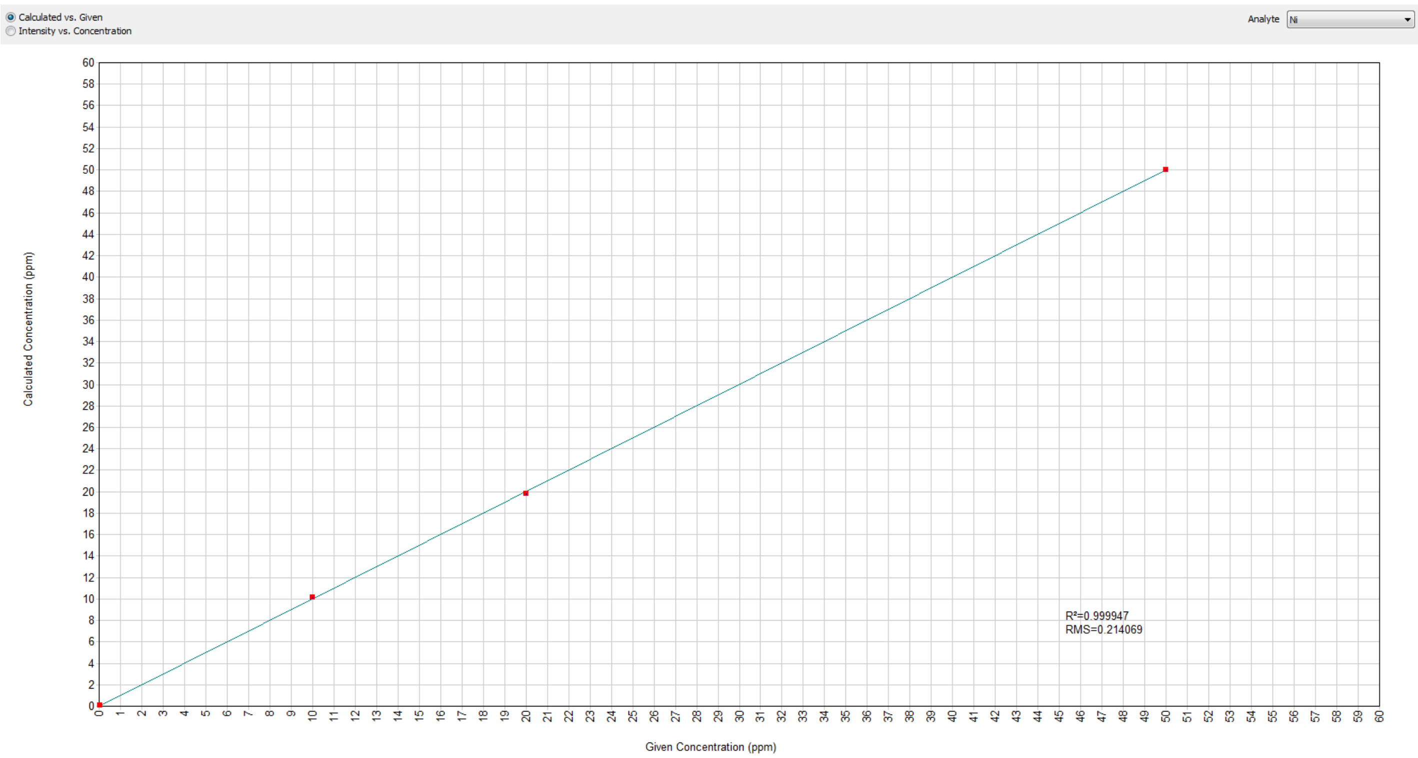


Figure 2b: Calculated versus given concentrations in the case of nickel (Ni)



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