

Analysis of sulfur and chlorine in waste oils under air conditions

*ARL QUANT'X with Si(Li) Technology
Energy-Dispersive X-Ray Fluorescence Spectrometer*

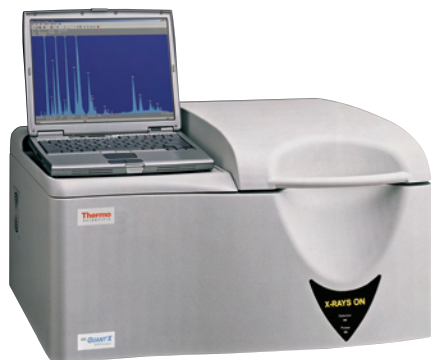
Introduction



This study describes the use of the Thermo Scientific ARL QUANT'X EDXRF spectrometer for the analysis of light elements in waste oils at low concentrations under air conditions. Sulfur and chlorine are common contaminants in used oils that can damage equipment or pollute the atmosphere if burned as fuels.

Energy Dispersive X-Ray Fluorescence (EDXRF) is a fast, economical analysis technique for elements in a wide variety of samples, either liquids or solids. Advantages of EDXRF over other analytical techniques include speed of analysis, little if any sample preparation and nondestructive analysis of samples.

Instrumentation



The Thermo Scientific ARL QUANT'X spectrometer is designed for the most challenging analytical requirements in laboratory and manufacturing environments. Its power and flexibility maximize productivity across a wide range of elemental analysis applications. Designed for easy transport and robustness, the ARL QUANT'X can also be relocated to facilitate on-site analysis as necessary.

Equipped with a unique multi-stage Si(Li) Peltier cooled detector, the instrument offers the best energy resolution and the highest peak-to-background ratio for detecting ppm levels of light elements without peripheral dependence on helium or liquid nitrogen, resulting in significant cost savings and ease of use. Analysis under air is further enabled by the high powered X-ray tube and efficient close coupling distance to the sample. Results are notable for low detection limits coupled with high precision.

Calibration and results

The standards used for calibration were n-butyl sulfite (sulfur) and chlorooctane (chlorine). Calibration was performed in the range of 5 to 1000 ppm. To reach an acceptable compromise between acquisition time and limit of detection, two excitation conditions were employed with 7 minutes counting time per condition.

Standard Units	S ppm	Cl ppm
std 0	0	0
std5-1	8.7	8.2
std5-2	6	5.6
std10-1	11.2	10.5
std10-2	11.5	10.8
std100	107	101
std250	223	210
std500	442	417
std750	674	635
std1000	995	938

Table 1: Certified concentrations of the calibration standards used

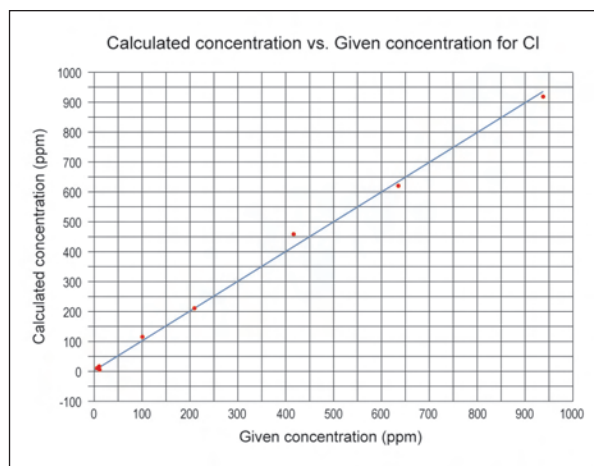
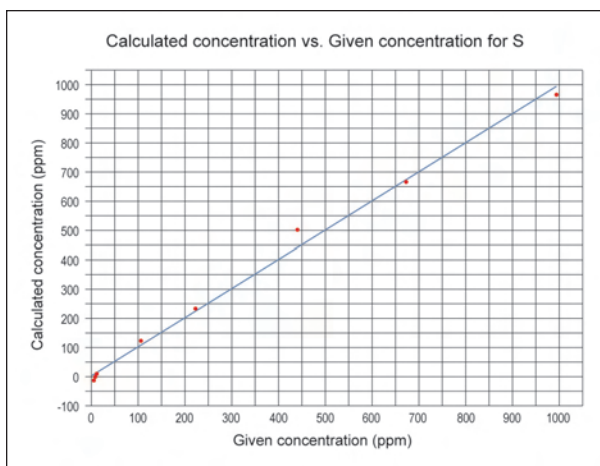


Figure 1: S calibration from 0 to 1000 ppm

Figure 2: Cl calibration from 0 to 1000 ppm

	S ppm	Cl ppm		S ppm	Cl ppm		S ppm	Cl ppm
sample1 01	156	154	sample2 01	231	219	sample3 01	425	421
sample1 02	154	158	sample2 02	221	220	sample3 02	429	432
sample1 03	167	135	sample2 03	214	219	sample3 03	433	404
sample1 04	148	154	sample2 04	221	233	sample3 04	410	420
sample1 05	142	170	sample2 05	222	210	sample3 05	423	431
sample1 06	152	144	sample2 06	228	233	sample3 06	434	432
sample1 07	151	138	sample2 07	209	222	sample3 07	441	425
sample1 08	152	154	sample2 08	216	215	sample3 08	439	401
sample1 09	132	145	sample2 09	232	224	sample3 09	433	409
sample1 10	156	124	sample2 10	253	222	sample3 10	434	404
average	151	148	average	225	222	average	430	418
sigma	9	13	sigma	12	7	sigma	9	12

Table 2: Precision for S and Cl under air conditions at 150, 225 and 430 ppm

Analytical precision

Repeatability on the 3 unknown samples in the range of 150 ppm to 500 ppm is excellent and exceeds requirements for most sulfur and chlorine analysis in waste oils.

As S and Cl are volatile elements and no helium flux is used, the atmosphere inside the chamber is stable and the concentrations of the elements do not change significantly, even after ten consecutive measurements.

Conclusion

These results demonstrate that the ARL QUANT[®]X with its highly sensitive Peltier-cooled detector successfully quantifies low concentrations of Cl and S in used oils under air conditions, providing exceptional ease of use and minimal analysis and maintenance costs. Acquisition time is quick, less than 10 minutes per element. If required with certain oils, the ARL QUANT[®]X can also quantify additional contaminant elements with a few minutes more of analysis. With the added benefit of field transportability, the ARL QUANT[®]X is ideal for fast, accurate and repeatable results for all manner of used oil analyses.

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AN41907_E 12/09C