Analysis of Sulfur and Chlorine in Oil under Air Conditions, using EDXRF

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This Application note describes the use of the Thermo Scientific ARL QUANT'X EDXRF spectrometer for the analysis of light elements like sulfur and chlorine in oil, at low concentration levels under air conditions. Equipped with a unique multi stage Si(Li) Peltier cooled detector, the ARL QUANT'X offers the best energy resolution and the highest peak to background ratio necessary for detecting ppm levels of S and Cl without the need for peripheral dependence, like helium or liquid nitrogen. Analysis of S and Cl under air conditions is made possible due to the high power on the X-ray tube, the close coupling distances: X-ray tube-sample-detector and the large solid stage Si(Li) crystal, thus eliminating the need for Helium or vacuum conditions, resulting in significant cost savings and ease of maintenance. The level of quantifications reached with the ARL QUANT'X under these conditions easily meets the requirements for analyzing light elements in oil.

Instrumentation

Energy Dispersive Xray Fluorescence (EDXRF) is a fast and economical technique for analysis of all inorganic elements in a variety of states, shapes and sizes. The most common advantages of EDXRF over other analytical techniques include speed of analysis and relatively little sample preparation, if any at all. X-rays are also non-destructive, as long as the sample isn't alive.

The ARL QUANT'X Energy Dispersive Xray Fluorescence Spectrometer from Thermo Fisher Scientific is designed for the most challenging analytical requirements in laboratory and manufacturing environments. Its power and flexibility maximizes productivity across a wide range of elemental analysis applications. Designed for easy transport and robustness, the ARL QUANT'X can be placed close to the place where analysis is required. For example, it can be used for screening oil from electrical transformers.

Calibration and results

This note (full details of which can be found on www.thermo.com/xray) demonstrates the high precision and low detection limits capacity of the ARL QUANT'X ED-XRF when performing analysis of S and Cl in oil under air conditions. The standards used to calibrate are N-butyls sulfite for sulfur and chloro-octane

for 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 required with a time of 7 minutes per condition. This is possible thanks to the close coupling distances: tube-sample-detector that a direct excitation system like the ARL QUANT'X can provide.

The repeatability of 3 unknown samples in the range of 150ppm to 500ppm was excellent and exceeds requirements for most sulfur and chlorine analysis in the oil industry.

As S and Cl are volatile elements and no helium flux was used, the atmosphere inside the chamber was stable and the concentrations of the elements did not change significantly, even on the repeatability of ten measurements.

Conclusion

The results show that the ARL QUANT'X can perform Cl and S analysis in oil under air conditions at low concentration levels, minimizing the cost of analysis and maintenance of the system. The time of acquisition is lower than 10 minutes per element.

In addition to the measurement of these 2 specific elements, the ARL QUANT'X can also perform the quantification of many more elements, such as Cr, Fe, Zn, Mo, W, Pb, Sn, etc., in only a few additional minutes. This is due to the high sensitivity of the Peltier-cooled detector and effective use of direct filtered excitation. With the added benefit of field transportability, the ARL QUANT'X is ideal for fast, accurate and repeatable results in the large field of oil and petrochemical analysis.