Application Note EDXRF1312

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EDXRF1312 - Analysis of Lube Oils

Scope

The measurement of phosphorus (P), sulfur (S), calcium (Ca), and zinc (Zn) in lube oil is demonstrated as per ASTM D6481.

Background

ASTM D6481 is a standard test method for measuring lube oil using EDXRF. The elements and concentration ranges covered by D6481 are specified in Table 1 of the method.

ASTM D6481-99 (2010)
Table 1 Elements and Range of Concentrations Determined

Element	PLOQ in mass%	Max concentration in mass%
Phosphorus	0.02 to 0.3 mass %	0.10
Sulfur	0.05 to 1.0 mass %	0.125
Calcium	0.02 to 1.0 mass %	1.94
Zinc	0.01 to 0.3 mass %	0.05

Quality control and quality assurance during the lube oil manufacturing process is essential, as various formulations give different lubricity properties. A fast, simple method of analyzing lube oils is important throughout the QC/QA process. Rigaku meets this industry need with a high-performance, low-cost benchtop EDXRF system. Rugged and reliable, the NEX QC+ is an ideal tool, with simple and intuitive software for the non-technical operator.

Calibration

Empirical calibrations were built using a suite of 23 commercially available calibration standards. The suite of calibration standards must be representative of the lube oil formulation to be analyzed. Use of empirical calibration ensures the particular lube oil formulation is exactly characterized and modeled, which yields optimum accuracy. Elements in the lube oil should vary evenly over each concentration range of interest, and the elements in the oil should vary independently of each other. Alpha corrections are then employed to automatically compensate for variations in X-ray absorption/enhancement effects within the sample due to the independent variations in element concentration. A summary of the empirical calibrations is shown here.

Element	Concentration range	Standard error of estimate
P	0.002 – 0.250 %	0.006
S	0.050 – 2.500 %	0.032
Ca	0.001 – 0.500 %	0.0005
Zn	0.001 – 0.250 %	0.0012

Repeatability

Representative samples from the calibration suite were chosen to demonstrate typical instrument repeatability (precision). Ten repeat analyses were performed with the sample in static position.

Sample: 7 **Units: %**				
Element	**Standard value**	**Average value**	**Std. dev**	**% Relative dev**
P	0.100	0.1007	0.0007	0.7 %
S	1.251	1.272	0.007	0.6 %
Ca	0.350	0.3578	0.0015	0.4 %
Zn	0.001	0.0011	0.0001	10 %

Sample: 9 **Units: %**				
Element	**Standard value**	**Average value**	**Std. dev**	**% Relative dev**
P	0	---	---	---
S	0.750	0.7479	0.0034	0.5 %
Ca	0.300	0.3055	0.0017	0.6 %
Zn	0	---	---	---

Sample: 21 Units: %

Element	Standard value	Average value	Std. dev	% Relative dev
P	0.250	0.2492	0.0041	1.6 %
S	2.500	2.514	0.014	0.6 %
Ca	0.010	0.0120	0.0002	2.0 %
Zn	0.050	0.0487	0.0002	0.4 %

Sample: 6 **Units: %**				
Element	**Standard value**	**Average value**	**Std. dev**	**% Relative dev**
P	0.010	0.0110	0.0003	3.0 %
S	0.250	0.246	0.001	0.4 %
Ca	0.001	0.0019	0.0001	10 %
Zn	0.225	0.2198	0.0006	0.3 %

Conclusion

The NEX QC+ offers analysts and technicians a simple yet powerful and versatile system for quantifying elemental composition using the empirical approach. The results of this study indicate that given stable samples, proper sample handling and proper calibration technique, the Rigaku NEX QC+ EDXRF can achieve excellent results in monitoring the elemental concentration of lubricating oils.

Related products

NEX QC Series

Combines quality, affordability, and performance for a wide range of applications