Application Note EDXRF1970

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EDXRF1970 - Metals In Crude & Resid As Per ASTM D8252

Scope

The analysis of nickel, vanadium and iron in crude and residual oil as per ASTM D8252 is demonstrated. The measurement also includes the analysis of sulfur complying with ASTM D4294.

Background

The quality and grade of crude oil in part depends on the metal content as well as the sulfur content. Nickel and vanadium are the critical metals, as well as iron in some crudes and in residual oils and the metal content is especially important in the quality of bunker fuels. ASTM D8252 addresses the need to measure low levels of nickel and vanadium in crude to meet the NYMEX/CME specifications for light sweet crude oil futures contract concerning maximum allowable levels the Ni and V.

To meet the needs of the industry, Rigaku offers the NEX CG, a simple, versatile, an EDXRF analyzer ideally suited for the analysis of sulfur and metals in crude, residual oil and marine fuels. The NEX CG uses full 90o Cartesian Geometry Polarization monochromatic excitation giving superior light element performance as well as the metals.

Model: NEX CG

Units

1 ppm = 1 mg/kg

Calibration

Empirical calibrations were built using a suite of 12 commercially available calibration standards. A summary of the empirical calibrations is shown here.

Element	Concentration range
Ni	3 – 50 ppm
V	3 – 50 ppm
Fe	3 – 50 ppm
S	0.100 – 5.000 mass%

Repeatability (Precision)

To demonstrate repeatability, samples were measured in static position for ten repeat analyses with typical results shown below.

Sample: Std 10				
Element	**Standard value**	**Average value**	**Std dev**	**% Relative dev**
Ni ppm	51	49.8	0.5	1.0
V ppm	5.0	5.43	0.29	5.8

Fe ppm	10.0	10.6	0.6	5.7
S mass%	4.501	4.470	0.010	0.2

Sample: Std 12				
Element	**Standard value**	**Average value**	**Std dev**	**% Relative dev**
Ni ppm	30	30.5	0.6	2.0
V ppm	50	50.1	0.5	1.0
Fe ppm	5.0	5.03	0.45	9.0
S mass%	0.250	0.252	0.001	0.4

Sample: Std 3				
Element	**Standard value**	**Average value**	**Std dev**	**% Relative dev**
Ni ppm	5	5.05	0.23	4.6
V ppm	30	30.3	0.4	1.3
Fe ppm	40	41.4	0.7	1.8
S mass%	1.000	0.986	0.002	0.2

ASTM D8252-19

In ASTM little r is called repeatability and represents the precision of the measurement. Little r is 2.77 times the 1σ standard deviation precision of repeat measurements. Comparison of NEX CG instrument r to the method r of D8252 shows NEX CG complies with the ASTM method for the measurement of Ni and V in crude and residual oil.

Units: mg/kg (ppm)					
Element	**Standard value**	**Average value**	**Standard deviation (1σ)**	**Instrument r**	**D8252-19 r**
Ni	5.0	5.05	0.23	0.64	1.1
V	5.0	5.43	0.29	0.8	1.0

Conclusion

The performance shown here demonstrates the ability of the NEX CG for the measurement of metals in crude and residual oil as per ASTM D8252 and sulfur in compliance with D4294. NEX CG is an excellent all-in-one analyzer complying with the following ASTM and US EPA test methods.

Method	**Application**
D8252	Vanadium and Nickel in Crude & Residual Oil

D4294	Sulfur in Petroleum and Petroleum Products
D7220	Sulfur in Automotive, Heating and Jet Fuels
EPA ULSD	Ultra-low Sulfur Diesel
EPA Tier 3 Gasoline	Ultra-low Sulfur Gasoline
D4929 Part C	Organic Chloride Content in Crude Oil
D5059	Lead in Gasoline
D6481	P, S, Ca, Zn in Lubricating Oils
D7751	Mg, P, S, Cl, Ca, Zn, Mo Additive Elements in Lubricating Oils

Related products

NEX CG II Series

High-performance *indirect excitation* EDXRF for complex ap
plications with trace elements and variable base matrices