APPLICATION NOTE

Analysis of total chlorine in oil by XRF at 1500W

ARL PERFORM'X Sequential X-Ray Fluorescence Spectrometer

Introduction

In order to demonstrate the analysis of chlorine (Cl) in oil, a calibration curve has been constructed according to ASTM D4929 method except that we have used a rhodium anode tube.

Instrumentation

The powerful and stable Thermo Scientific™ ARL™ PERFORM'X WD-XRF instrument has been used. It includes an option for change-over of the analysis environment from vacuum to helium atmosphere which allows liquids analysis.

The analytical conditions are shown below.

Chlorine analysis in oils

The ASTM D4929 method has been chosen for low Cl in oil. Five standard samples were prepared in accordance with the norm to construct a calibration curve. This method proposes the use of net peak intensities, hence a background position was chosen. The counting time on the Cl peak and the background position are identical.

From a concentrate product which contained 1000 ppm of Chlorine a mother solution was prepared by diluting this concentrate with Iso-Octane. This mother solution was then used to prepare several samples with various Chlorine concentrations.

Instrument	ARL PERFORM'X
Crystal	Ge 111
Collimator	0.40
Detector	FPC
kV – mA	30 – 50
Power	1500 watts
Time / Line [1]	180 sec
Total time	360 sec

◄ The instrument settings

The net intensity for chlorine (peak minus background) for each standard is related to the Cl content in ppm, which results in the calibration curve shown in Figure 1. Table 1 gives the numerical results including the absolute difference between nominal and calculated concentrations, as well as the Standard Error of Estimate.

Sample #	Cl intensity	Nom. conc. ppm	Calc. conc. ppm	Absol. diff. ppm
0	108	0	0.3	0.3
1	167	7.9	8	0.1
2	239	17.7	17.5	0.2
3	409	40.4	39.7	0.7
4	572	60.4	61	0.6
5	655	71.9	71.8	0.1

SEE = 0.51 ppm
Correlation error R^2 = 0.999753

Table 1: Calibration results for five standard samples with low Cl content
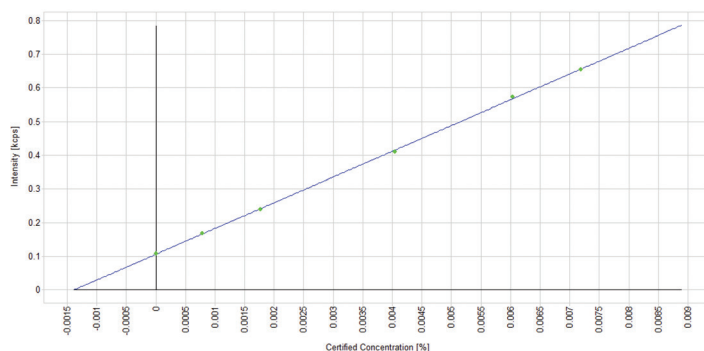
Cl in oil - low levels

Figure 1: Cl calibration curve

Repeatability test on oil samples containing Cl

A repeatability test for the analysis of Cl has been done using one oil sample with 1.4 ppm level using the same analytical conditions shown overleaf.

Six liquid cells were prepared and analyzed. The results obtained are the following:

Sample	Cl concentration in ppm
Cell 1	1.2
Cell 2	1.5
Cell 3	1.2
Cell 4	1.5
Cell 5	1.2
Cell 6	1.8
Average	1.4
SD	0.6

Table 2: Repeatability test

Conclusion

A good calibration curve can be obtained with the ARL PERFORM'X XRF for Cl determination in oil when applying the appropriate ASTM standard method although we used a rhodium anode tube.

Thanks to the reproducibility of loading and to the ARL PERFORM'X innovative helium shutter protecting the goniometer chamber from the helium environment, excellent repeatability of analysis can be demonstrated for Cl analysis at low level.

The results obtained show that good accuracy and precision can be achieved with the ARL PERFORM'X Sequential XRF instrument. This instrument is well suited for the analysis of Cl in petrochemical products.

Thermo Scientific ARL PERFORM'X Sequential XRF

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