Application Note: 41653

Chlorine Analysis in Fuel Oils

Thermo Scientific ARL PERFORM'X Series Advanced X-Ray Fluorescence Spectrometers

Key Words

- ARL PERFORM'X 4200 W
- Chlorine analysis in fuel oils
- XRF
- X-ray fluorescence

Introduction

Monitoring chlorine in fuel oil is a very important evaluation for many petrochemical producers and users of this product for a variety of different reasons ranging from quality control, preventative maintenance, environmental regulations or failure analysis. The introduction of chlorine can come from lubrication additive packages, contamination from environmental sources or as a product of wear.

Chlorine analysis in fuel oil can be performed in many ways including bomb calorimeter, potentiometer titration and X-ray fluorescence (XRF). The simplest and fastest method of analysis is by XRF.

This technique offers excellent repeatability and resolution for elements ranging from Beryllium (Be) to Uranium (U). In addition, it allows petroleum based products to be measured directly without dilution, which significantly reduces sample preparation time and increases speed and throughput of analyses.

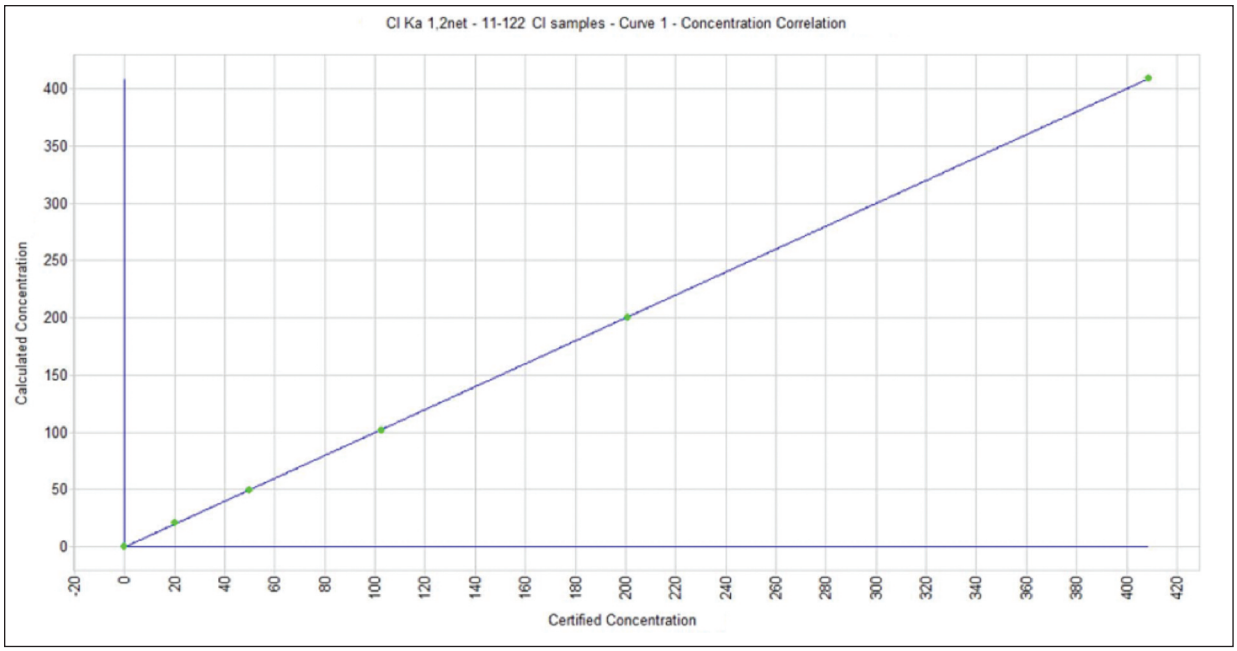
Instrument

Thermo Scientific ARL PERFORM'X series spectrometer used in this analysis was a 4200 watt system. This system is configured with 6 primary beam filters, 4 collimators, up to nine crystals, two detectors, helium purge and our 5GN+ Rh X-ray tube for best performance from ultra-light to heaviest elements thanks to its 50 micron Be window. This new X-ray tube fitted with a low current filament ensures an unequalled analytical stability month after month.

The ARL PERFORM'X offers the ultimate in performance and sample analysis safety. Its unique LoadSafe design includes a series of features that prevent any trouble during sample pumping and loading. Liquid cassette recognition prevents any liquid sample to be exposed to vacuum by mistake. Over exposure safety automatically ejects a liquid sample if X-ray exposure time is too long.

The Secutainer system protects the primary chamber by vacuum collecting any loose powders in a specially designed container, easily removed and cleaned by any operator. For spectral chamber protection, the ARL PERFORM'X uses a helium shutter designed for absolute protection of your goniometer during liquid analysis under helium operation. In the "LoadSafe Ultra" configuration, a special X-ray tube shield provides total protection against sample breakage or liquid cell rupture.

The ARL PERFORM'X spectrometer also features small spot and elemental mapping analysis down to 0.5 mm areas. These features enhance the capabilities of this XRF system by providing additional screening, contamination identification, inclusion analysis and segregation/non-homogeneity mapping on solid samples.



Graph 1: Plot of calculated concentration vs. given concentration in units of ppm (part per million) using 60 seconds counting time and background correction.

Calibration

A linear regression calibration was created by measuring several certified reference materials and plotting the measured intensities against the given concentrations for each sample. The results are illustrated in graph 1. The plot shows a very high degree of linearity with extremely low standard estimate of error (SEE) and limit of detection (LoD) for Cl of 0.54 ppm, which has been summarized in Table 1.

Element	R^2 Correlation	Typical SEE (PPM)	LoD (PPM)
Cl	0.999964	1.03	0.54

Table 1: LoD = Limit of Detection; SEE = Standard Estimate of Error.
LoD is stated for 100s count time.

A reproducibility test has been done using 3 different sample cells. The result shown in Table 2 is excellent with a standard deviation of 0.21 ppm at a level of 18 ppm Cl.

Elements	Cl Result (PPM)
Run nr 1	18.4
Run nr 2	18.1
Run nr 3	18.5
Average	18.3
SD	0.21

Table 2: Reproducibility using 3 different sample cells.
SD = Standard Deviation.
Results were obtained using 60 seconds counting time.

Conclusion

It is seen that analysis of chlorine in fuel base samples can be performed with ease on the ARL PERFORM'X sequential XRF spectrometer. The precision and accuracy are excellent. All calibration ranges can be extended with the simple addition of more certified reference standards.

Furthermore, operation is made easy through the state-of-the-art Thermo Scientific OXSAS software which operates with the latest Microsoft Windows© 7 packages.

To see our complete X-ray product portfolio, visit **www.thermoscientific.com/xray**

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