APPLICATION NOTE

Total chloride in catalysts with Thermo Scientific ARL PERFORM'X Advanced WDXRF Spectrometer

Introduction

The UOP method 979-02 describes the method for determination of the total chloride content in fresh, regenerated and spent alumina supported catalysts by wavelength dispersive X-ray fluorescence (XRF) spectrometry.

Instrument

The Thermo Scientific™ ARL™ PERFORM'X series spectrometer has been configured with only the essentials regarding the analysis of catalysts. The instrument is equipped with two crystals (AX06 and Ge111), one collimator and one detector (FPC).

This affordable configuration enables the analysis of all elements ranging from F to Fe. Two power levels are available (1500 W and 2500 W) which both operate without an external water chiller. The ARL PERFORM'X analyzer also allows for field upgrades (crystals or detector) to meet any specification and extend the analysis range from beryllium to trans-uranics, as do all other ARL PERFORM'X systems.

As can be seen from the below tables the required performance for catalysts will be easily reached within a minute of analysis or less. At 1500 W a counting time of 60 seconds provides an excellent precision.

Thermo Scientific ARL PERFORM'X series spectrometer used in this analysis was a 1500 watt system. This system is configured with a rhodium anode X-ray tube for high performance from ultra-light to heaviest elements. This new X-ray tube fitted with a low current filament ensures an unequalled analytical stability month after month.

The Secutainer system protects the primary chamber by vacuum collecting any loose powders in a specially designed container, easily removed and cleaned by the operator.

Instrument control and data handling

The powerful and user-friendly Thermo Scientific OXSAS software supports spectrometer operation and data handling. The advanced design of the OXSAS platform allows evolution to meet each customer's needs with up-to-date solutions throughout the lifetime of the XRF instrument.

Calibration

The UOP 979 method calls for the determination of the net peak intensity of chlorine to be plotted against the concentration values. Prior to establish the calibration curve a scan on Cl Kα peak was realized and is shown in Figure 1. It helps choose the correct background positions.

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Figure 1: Scan on chlorine Kα peak and choice of the background positions (30kV – 50mA)

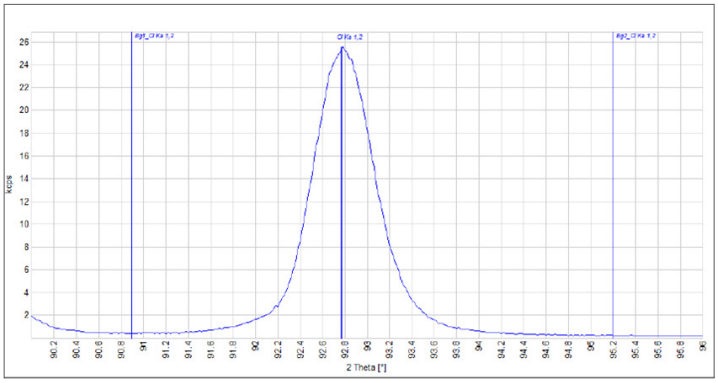
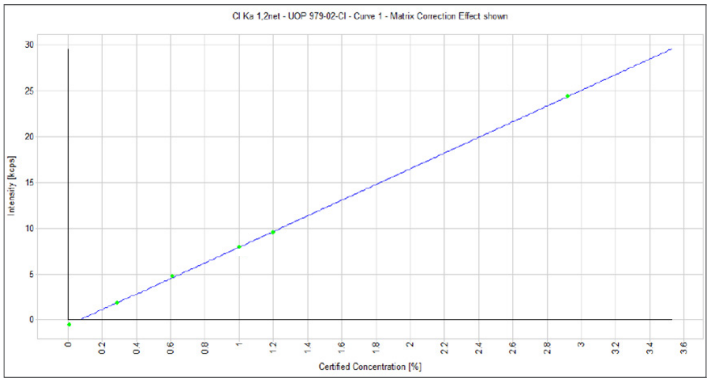


Figure 2: Regression for chlorine using 30 kV – 50 mA X-ray tube conditions



A set of reference samples with known chlorine content were used in the creation of the chlorine regression plot. The graph is a linear regression of the known concentrations plotted against the measured intensities. The samples were ground to a fine powder and measured as loose powder under helium atmosphere. The standard error of estimation (SEE) is 0.013% and is the average accuracy of analysis in the range from 0.2 % to 2.9 %.

Precision results

Precision tests on a pressed catalyst sample were carried out by running the sample for five repeat analyses at 60 seconds counting time. The results are summarized in Table 1. At 1% chlorine the excellent standard deviation (SD) of 0.002% is achieved using 60 seconds counting time on peak and background.

If faster analysis is required the counting time could be reduced drastically by factor four to 15 seconds which would result in doubling the typical precision to 0.004% at a Cl content of 1%. Despite the shorter counting time this is still an excellent precision.

Table 1: Chlorine content results

Run	Chlorine content by WDXRF (%)
1	1.0044
2	1.0093
3	1.0066
4	1.0087
5	1.0086
Average	1.0075
Std deviation	0.002

Conclusion

The results show that chlorine content in catalysts can easily be determined with the ARL PERFORM'X sequential XRF spectrometer. Both precision and accuracy are very good in this matrix type for routine analysis.

Furthermore, operation is made easy through the most advanced state-of-the-art Thermo Scientific OXSAS WDXRF software which operates with the latest Microsoft Windows® 10 package.

Find out more at **www.thermofisher.com/performx**

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