S4 EXPLORER
THE ANALYSIS OF FLUORINE IN LIMESTONE

Introduction

For many years X-ray fluorescence spectrometry (XRF) has been used as a process control tool in the manufacture of cement products.

XRF analysis is used for a whole host of sample types generated by the cement making process. These include the raw materials, cement raw meals, clinker cements and finished cements. Increasingly XRF is also being used to monitor alternative fuel and waste materials.

This report outlines the analysis of Fluorine in one of the key cement raw materials – limestone. It aims to demonstrate the superb light element performance of the Bruker AXS S4 EXPLORER.

Instrumental

The Bruker AXS S4 EXPLORER (Figure 1.) is a revolutionary step forward in the development of sequential wavelength dispersive XRF spectrometers. It comprises all of the usual components - a 1kW end window Rhodium X-ray tube, up to ten primary beam filters, up to four collimators and up to eight analysing crystals.

Scintillation and gas-proportional counters are both available. All of this is coupled with Bruker AXS´ superb goniometer technology.

With both vacuum and helium systems available the analysis of soild and liquid samples is possible.

Figure 1. S4 EXPLORER

However the S4 EXPLORER is unique amongst XRF spectrometers with features such as:

- Space required is only 131 cm x 84 cm x 88 cm, which means less than 0.75m^2 floorspace needed!
- No requirement for flow counter gas!
- No requirement for cooling water!
- No requirement for compressed air!

A wide range of sample changers are available, from 1 to 108 positions, and these can be directly linked into automated sample handling systems.

Software

Perhaps the most outstanding feature of the S4 EXPLORER analytical system is the inclusion of the SPECTRA*plus* software package. This is a fully interactive program that uses the Windows NT platform for maximium speed and stability.

It comes complete with a standardless analysis routine, a variable alpha matrix correction program, a rapid data retrieval system and full networking capability.

Calibration and Results

A set of five standards was produced in order to calibrate the instrument. A base limestone was 'spiked' with Calcium Fluoride to give a concentration range of zero to 0.4 % Fluorine.

The standards were prepared as pressed pellets. The powders were ground with a binding aid and pressed into 40 mm discs.

An analytical method was developed to measure the standards. Special care was taken to select background positions not influenced by the L-Series lines of Iron and Manganese.

The pellets were placed into standard S4 EXPLORER sample cups and analysed to produce a calibration curve. This curve is shown in Figure 2. The calibration was produced from a simple linear regression of fluorine concentration against net intensity.

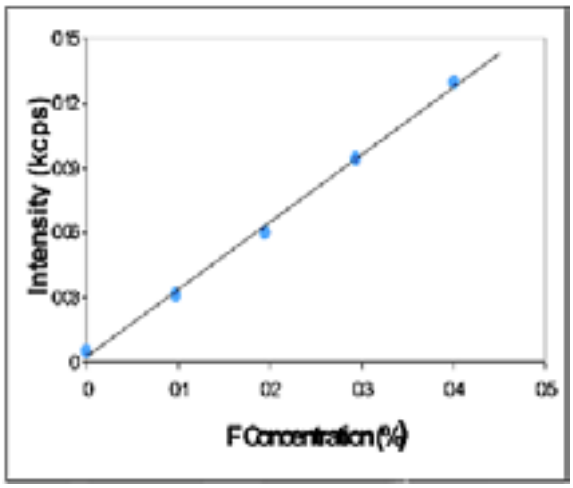

Figure 2. Fluorine Calibration Curve

The standard error of the calibration was 0.0071 % or 71 parts per million (ppm) fluorine.

The three sigma Lower Limit of Detection (LLD) for Fluorine in a limestone matrix was determined to be 108 ppm. This was based on one hundred seconds analysis time.

Conclusion

This application note has summarised the outstanding performance of the S4 EXPLORER for the analysis of Fluorine in limestone.

It has also demonstrated the general quality of light element analysis using the S4 EXPLORER.

Further application notes are available which describe other types of cement analysis using Bruker AXS analytical X-ray equipment.

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