

S4 EXPLORER

DETERMINATION OF FLUORINE ON FILTER PAPERS

Introduction

Recovery of environmental and waste materials these days is an item of utmost concern. Elemental analysis and techniques to analyse these elements in the most accurate way and, preferably with a low limit of detection, do become more and more important. XRF is one of these techniques in which a broad range of elements and a broad range of concentrations can be analysed, although light elements in low concentrations, such as Fluorine, may need special preparations in order to get an accurate and precise result.

Preparation

For the XRF analysis of liquid samples, disposable plastic liquid cups are used. A very thin Mylar or polypropylene foil is applied as a covering in the bottom of the liquid cup.

These liquid cup foils absorb the longer wavelengths limiting the elemental range of the XRF analysis to all elements heavier than Fluorine. For this reason, direct trace element analysis of Fluorine (and lighter elements) in liquids is not possible and other preparation methods have to be applied, such as the preparation on filter papers.

- Preparation of F standard solutions: blank, 310, 625, 1250, 2500, 5000 ppm (the 5000 ppm NaF-solution was used as a stock-solution in order to prepare the dilutions).
- 100 μ l of the standard solutions was put on a paper filter of 50 mm diameter with a hydrophobe ring of 25 mm (Figure 2).



Figure 1: The plug 'n analyse S4 EXPLORER

- Drying of the filter paper was accomplished by laying it about 3 cm above an electric fire at a temperature of 150 degree C (is the faster method, can also be air-dried)
- The filter sample was put in a sample holder with a diameter of 34 mm and covered by a non-back scattering cup. The collimator mask applied was, in this case, 23 mm.

| Standard Name | Certified Concentr. (%) | XRF Concentr. (%) | Absolute Deviation (%) | Relative Deviation (%) |
|---------------|-------------------------|-------------------|------------------------|------------------------|
| Blank | 0,0000 | -0,0020 | -0,0020 | |
| F-310 | 0,0310 | 0,0322 | 0,0012 | 3,86 |
| F-625 | 0,0625 | 0,0651 | 0,0026 | 4,16 |
| F-1250 | 0,1250 | 0,1232 | -0,0018 | -1,41 |
| F-2500 | 0,2500 | 0,2500 | -0,0000 | |
| F-5000 | 0,5000 | 0,5000 | 0,0000 | 0,0052 |

Table 1: Fluorine calibration data



Figure 2: Filter paper with hydrofobe ring

Calibration

The calibration details are given in Table 1 and the curve for Fluorine is shown in Figure 3. The measuring time was 60 seconds.

Conclusion

The three sigma Lower Limit of Detection (LLD) for Fluorine on paper filters was determined to be 44 ppm at 100 seconds measuring time.

This application note summarizes the outstanding performance of the S4 EXPLORER for the WD-XRF analysis of a very light trace element at minimal

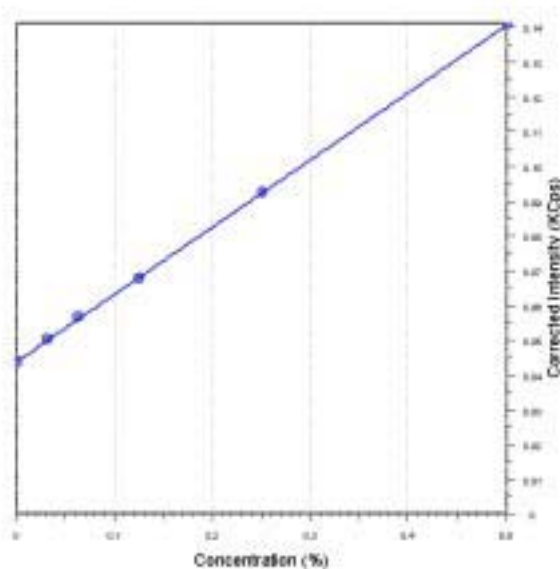


Figure 3: Fluorine calibration curve

operation costs. The innovative 1.000 W X-ray tube technology, which doesn't require external cooling water, and the Pro4 sealed proportional detector, which doesn't require detector gas or the periodical replacement of the detector window and detector wire, are but two advantages of the S4 EXPLORER "plug 'n analyse" high precision X-ray spectrometer with the full analytical performance of traditional high power 3 to 4 kW X-ray instruments.

Author: Pol De Pape, Bruker AXS GmbH Karlsruhe

BRUKER AXS GMBH

OESTLICHE RHEINBRUECKENSTR. 50
D-76187 KARLSRUHE
GERMANY

TEL. (+49) (721) 595-2888
FAX (+49) (721) 595-4587
<http://www.bruker-axs.de>
Email: info@bruker-axs.de

BRUKER AXS, INC.

5465 EAST CHERYL PARKWAY
MADISON, WI 53711-5373
USA

TEL. (+1) (800) 234-XRAY
TEL. (+1) (608) 276-3000
FAX (+1) (608) 276-3006
<http://www.bruker-axs.com>
Email: info@bruker-axs.com