



TXRF

Qualitative and Quantitative Analysis of Filter Samples by TXRF Spectrometry

Application Note # XRF 428

Introduction

The analysis of filter material is of crucial importance for environmental and health risk investigations. Besides grain size distribution and organic contaminations, the concentration of inorganic components, especially heavy metals is of great interest.

For this purpose methods like ICP-OES or AAS are often used, but require a time consuming digestion of the sample. In contrast, EDXRF systems allow a non-destructive analysis, but show restrictions regarding the detection limits and the accuracy of the external calibration.

TXRF combines the almost non-destructive sample preparation of EDXRF with the sensitivity of ICP-OES and AAS, even when analysing just minimal amounts of sample material deposited on a filter.

In this study typical filter samples from different European sources were analysed qualitatively and quantitatively, keeping the sample preparation as fast and simple as possible.

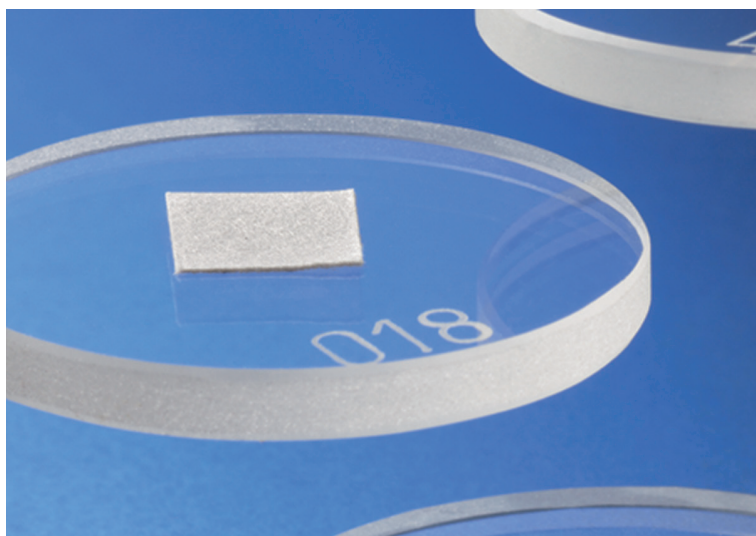


Figure 1

Filter sample prepared on a quartz carrier for qualitative analysis

Instrumentation

All measurements were performed using the bench top TXRF spectrometer S2 PICOFOX. This instrument is equipped with an aircooled low power X-ray tube (Mo target), a multilayer monochromator with 80% reflectivity and the liquid nitrogen-free XFlash® Silicon Drift Detector (SDD) with an energy resolution of < 159 eV (Mn K α).

Sample preparation and analysis

A picture showing the sample preparation of filter material for qualitative analysis is given in Figure 1.

A small part of the filter (approximately 3 x 3 mm) was separated by a scalpel. The cut-out was transferred to a quartz glass carrier and fixed with a thin film of desiccator grease, which was tested previously to be free of contaminants. By this kind of preparation, sample particles would remain on the carrier, and thus the accuracy of further quantitative analyses would be compromised, when preparing the cut-out with the particle - loaded side oriented towards the carrier surface. For this reason, comparative measurements were performed with the cut-out oriented down- and upwards relative to the carrier surface.

The measurement time was in general set to 1000 seconds.

Results

The spectra of the filter measurements are displayed in Figures 3a and 3b, and the data are summarized in Table 1. The use of a TXRF spectrometer in combination with the described sample preparation allows the simultaneous detection of elements in the range from sulfur to lead. Comparing the measurement results of the different orientations of the filters, it is evident that the peak intensities are significantly higher when the particle – loaded side of the analyzed sample was orientated towards the carrier.

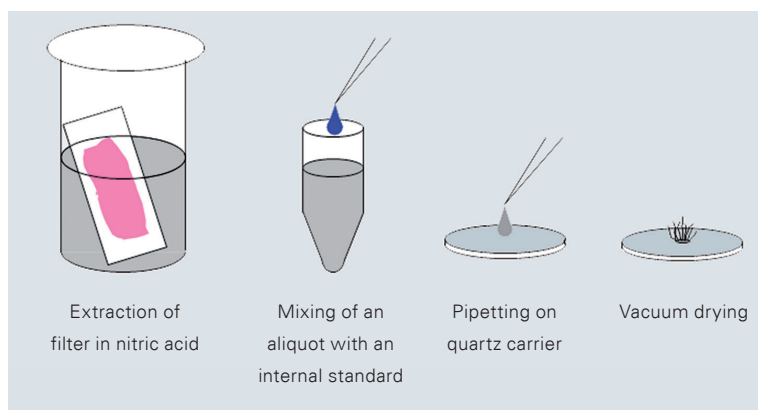
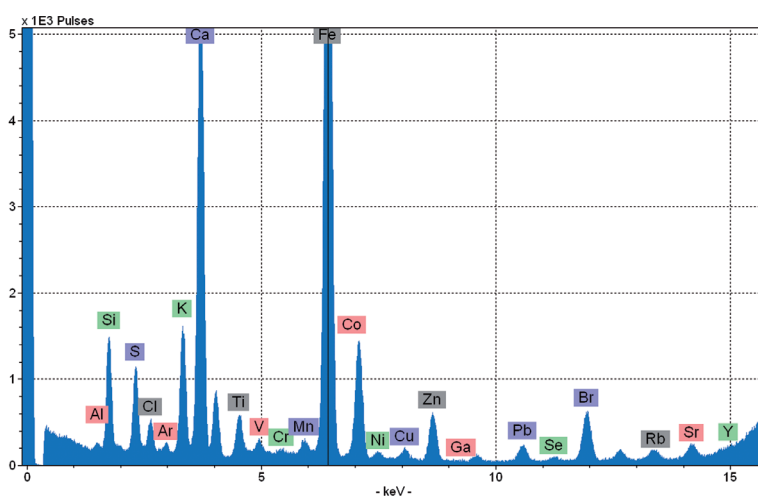
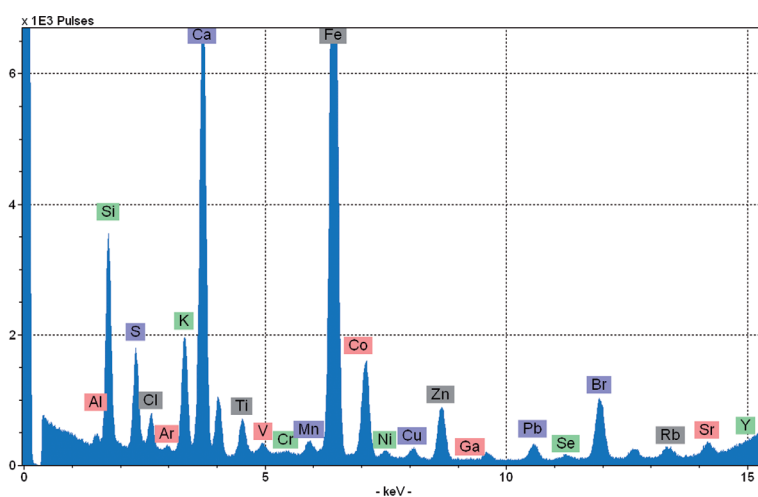


Figure 2
Filter preparation for quantitative TXRF analysis



a)



b)

Figure 3
Spectra of the analyzed filter sample 1f, (a) particle-loaded side up, (b) particle-loaded side down

Sample preparation for quantitative results

Common methods for the quantitative preparation of filter materials require the complete digestion of the filter, with or without preceding ashing or the extraction of the filtered material in ultrapure acids.

In this study sample preparation was done by a straightforward extraction. A schematic illustration of the sample preparation is given in Figure 2.

The filter material was transferred to a thoroughly cleaned glass beaker, filled with 5 ml of ultrapure nitric acid and treated in an ultrasonic bath for 5 minutes.

An aliquot of 1 ml of the resulting extraction was transferred to a vial and 10 μ l of internal standard solution (Ga, 1 mg/l, Merck) were added. After homogenisation, 10 μ l were pipetted onto a sample carrier and subsequently dried in a desiccator.

From each extraction three samples were prepared and analysed using measurement times of 1000 seconds. Additionally, a blank filter was prepared in the same manner for monitoring any contamination during the preparation.

Results

The results, which are listed in Table 2, are shown in mg/l of the extraction.

The software of the S2 PICOFOX also allows the output of the results in absolute values (μ g) or in mass per filter area (μ g/cm²).

A large number of elements from Mg to Pb could be detected and quantified. Comparison with the blank filter sample enables the characterization of significant amounts of several pollutants.

Element	Line	Energy (keV)	Filter side up	Filter side down
S	K12	2.309	11582	16972
Ci	K12	2.622	4527	6417
K	K12	3.314	19468	23980
Ca	K12	3.692	83039	101472
Ti	K12	4.512	7620	8809
V	K12	4.953	2689	2681
Cr	K12	5.415	1176	1056
Mn	K12	5.900	3248	3918
Fe	K12	6.405	169233	1869
Ni	K12	7.48	1931	2539
Cu	K12	8.046	2501	3038
Zn	K12	8.637	9933	14936
Se	K12	11.224	680	1176
Br	K12	11.924	11324	18950
Rb	K12	13.396	1403	1852
Sr	K12	14.165	1590	1472
Pb	L1	10.551	4995	6430

Table 1

Results of the qualitative analysis of filter 1, intensities in net count areas

Conclusion

It is shown that TXRF analysis is a suitable technique for qualitative and quantitative analysis of filter material. Unlike other methods such as EDXRF or ICP-OES, there is no need for external calibration and unknown samples can be analysed with little effort.

Element	Filter 2 (mg/l)	Filter f1 (mg/l)	Blank filter (mg/l)
Mg	not detected	22.5	not detected
P	not detected	1.20	not detected
S	31.1	1.80	not detected
Cl	6.82	0.471	0.680
K	24.1	5.79	not detected
Ca	80.6	3.82	0.185
Ti	not detected	0.108	not detected
V	not detected	0.034	not detected
Cr	0.876	<LOD	not detected
Mn	0.241	0.022	not detected
Fe	29.4	0.957	0.048
Ni	0.439	0.005	not detected
Cu	1.18	2.21	not detected
Zn	4.93	0.242	0.021
As	not detected	0.003	not detected
Br	0.296	0.015	0.016
Rb	<LOD	0.003	not detected
Sr	0.160	0.012	not detected
Ba	1.86	not detected	not detected
Pb	0.697	0.017	not detected

Table 2

Results of the TXRF analysis of two filter samples and one blank filter

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