S2 PICOFOX
TOTAL REFLECTION X-RAY FLUORESCENCE SPECTROSCOPY - WORKING PRINCIPLES

Main principle

The main principle of X-Ray Fluorescence Spectroscopy (XRF) is based on the fact, that atoms, when irradiated with X-Rays, radiate secondary X-Rays – the Fluorescence radiation.

On this basis XRF-analysis is possible because:

- The wavelength and energy of the Fluorescence radiation is specific for each element.
- The concentration of each element can be calculated using the intensity of the Fluorescence radiation.

TXRF analysis using the S2 PICOFOX

The working principle of Total reflection X-Ray Fluorescence spectroscopy as realized in the S2 PICOFOX spectrometer is shown in Figure 1. The X-Ray beam, generated by the Molybdenum-tube, is reflected on a Ni/C-multilayer resulting in a monochromatic X-Ray beam. This small beam passes the sample holder carrying the sample at a very small angle (0.3 – 0.6 °) causing total reflection of the beam. The characteristic Fluorescence radiation emitted by the sample is detected by an energy-dispersive detector (XFlash® detector) and the intensity is measured by means of an amplifier coupled to a multichannel analyzer.

The main difference with respect to common XRF-spectrometers is the use of monochromatic radiation and the total reflection optic. Illuminating the sample with a totally reflected beam reduces the absorption as well as the scattering of the beam in the sample and its matrix. Resulting benefits are a largely reduced background noise, and consequently much higher sensitivities and the significantly reduction of matrix effects.

One major advantage of TXRF, compared to atomic spectroscopy methods like AAS or ICP-OES, is the avoidance of memory effects. The technical parameters of the S2 PICOFOX spectrometer are summarized in Table 1.

Sample types and preparation

A summary of samples types, which can be analyzed by means of TXRF is given in Table 1, showing the great variety of applications. For TXRF analysis all samples must be prepared on a sample tray, which reflects X-Ray radiation. For this purpose the usage of trays with a diameter of 30 mm, made of acrylic or quartz glass is common.

Liquids can be preparated directly on the sample tray. An amount of several µl is transferred to the glass disc using a pipette and subsequently evaporated in a desiccator or drying oven (Figure 3.).

For solid samples different kinds of preparation are possible. Powdered samples (suspended matter, soils, minerals, metals, pigments, biogenous solids etc.) can be analyzed directly after preparation of the material on the sample tray. Typically, a few µg of sample material are transferred, using a Q-tip or a lint-free tissue.

In a similar way the direct preparation of single microsamples (particles, slivers etc.) is possible.

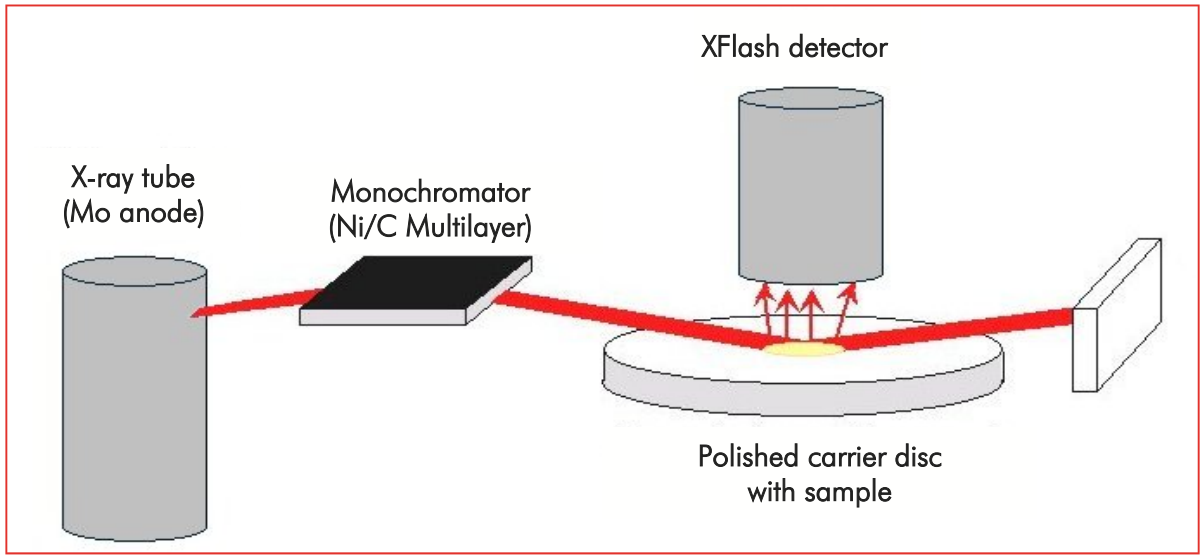

Figure 1: Schematic working principle of the S2 PICOFOX spectrometer

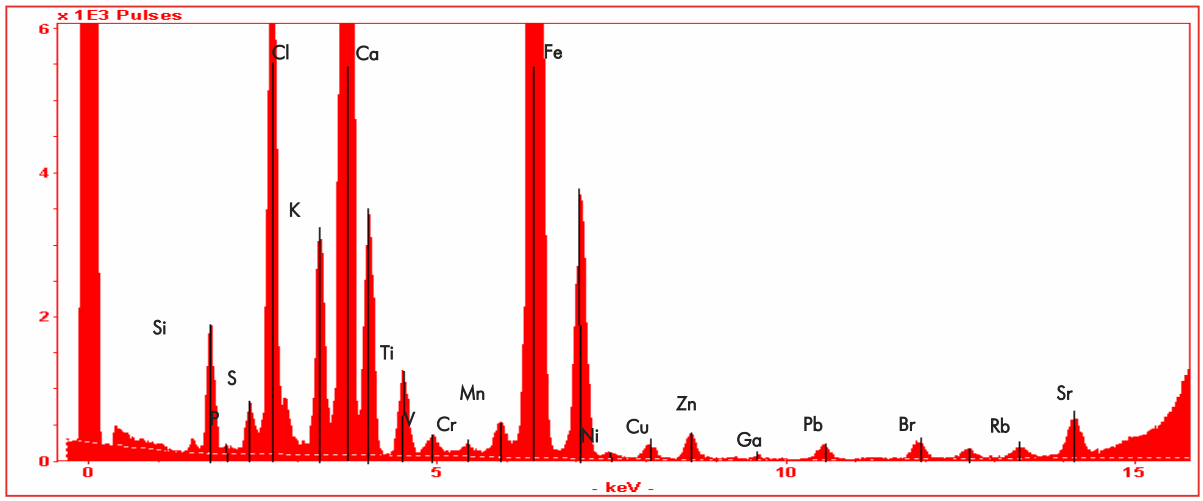

Figure 2. Typical energy dispersive spectra of the S2 PICOFOX

Table 1. Technical parameters of the S2 PICOFOX spectrometer

Tube	Metal-ceramic (50 W) Air-cooled Mo-Anode
Optics	Ni/C-Monochromator (17,5 keV)
Detector	XFlash® Area: 10 mm^2 FWHM: < 160 eV @ MnKα
Size	450 x 590 x 300 mm
Weight	37 kg

Alternatively, powdered solids can be prepared as a suspension with volatile solvents like acetone or methanol. The suspension is then pipetted onto the sample tray. In Figure 3 the sample preparation is described for solids, which were digested by microwave digestion.

Analysis and quantification

In general all elements starting from Sodium up to Uranium (excl. Niobium, Molybdenum and Technetium) can be analyzed by the S2 PICOFOX (Figure 4.). TXRF analysis is based on internal standardization. Therefore, an element, which is not present in the sample, must be added for quantification (Figure 3).

Table 2: Sample materials analyzable by TXRF (KLOCKENKÄMPER, 1997)

Liquids	Solids (Anorganic)	Solids (Biogenous)
Water potable, river, rain, sea and waste water	Soil: mud, sediments, sewage sludge	Plant material: algae, hay, leaves, braid, moss, needles, roots, wood
Body fluids blood, serum, urine	Suspended particles: aerosols, dusts, flue ash	Nutrition: fish, (sea-) fruits, meat, mushrooms, nuts, vegetables
	Minerals: ores, rocks, silicates, silicon	
Pure chemicals: acids, bases, solvents, water	Pigments: creams, inks, oil paint, powder	Tissue: hair, kidney, liver, lung, nails
	Metals: aluminium, iron, steel	
Oils and crude oil: combustibles, crude oil, fat and grease	Thin layers: contaminations, films, foils, layers, precipitates	

The complete process of analysis and quantification is described by the following steps:

- Measurement of the complete spectrum.
  All detectable elements are measured simultaneously.
- Evaluation of the measured spectra
  All identified elements have to be marked for further quantification, which can be done manually or automatically by the software.
- Spectra deconvolution
  On the basis of the chosen elements, the software performs the deconvolution of the spectra. The net intensities of the element peaks are calculated with regard to corrections of line overlaps, background factors, escape peak correction etc.
- Calculation of concentrations
  The element concentration is calculated by the simple formula:

$$c_x = \frac{N_x / S_x}{N_{is} / S_{is}} \cdot c_{is}$$

where N is the net intensity, S the relative sensitivity and c the concentration - each either of the analyte x or the internal standard is, as indicated. The typical detection limits of the S2 PICOFOX in aqueous solutions is presented in Figure 5.

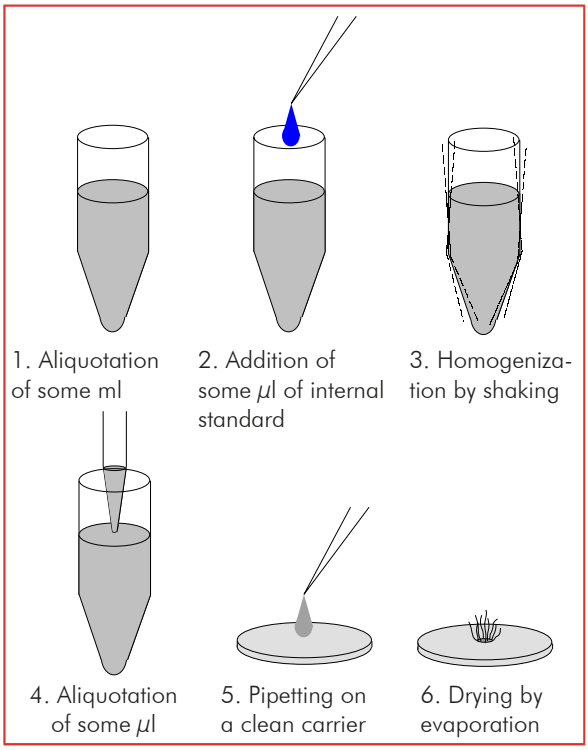

Figure 3: Preparation steps for the TXRF analysis of liquids

Summary

The S2 PICOFOX TXRF spectrometer is a versatile instrument for trace element analysis of different kinds of samples. It is completely independend of any cooling media and therefore applicable for on-site analysis. Further benefits of the S2 PICOFOX are the simple calibration routine, the absence of matrix or memory effects and the ability for fast multi-element analysis.

Literature

KLOCKENKÄMPER:
Total-Reflection X-Ray Fluorescence Analysis, John Wiley & Sons, 1997.

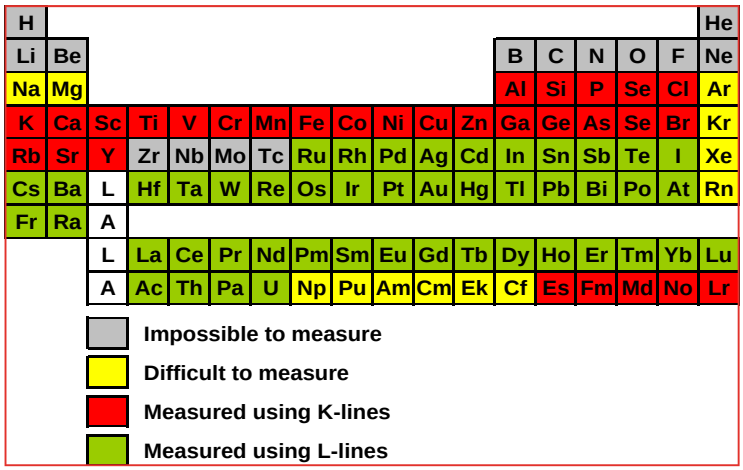

Figure 4: Overview of the measurability of elements using the S2 PICOFOX spectrometer

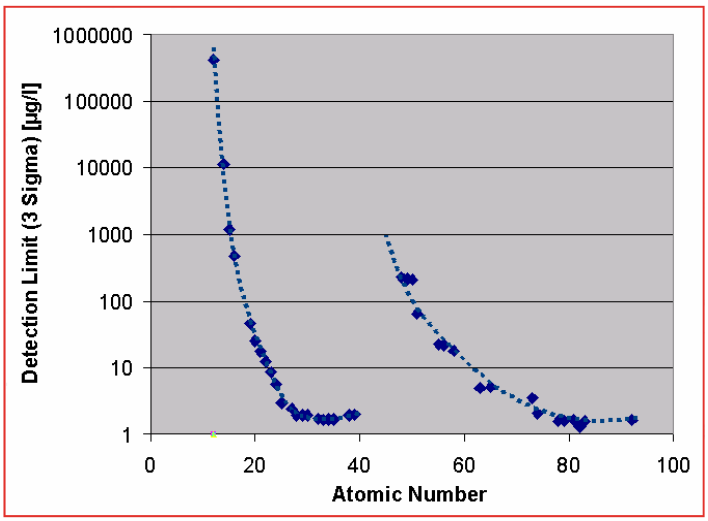

Figure 5: Low Limits of Detection for the S2 PICOFOX

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