

QUANTITATIVE X-RAY FLUORESCENCE ANALYSIS AT THE ESRF ID18F MICROPROBE

*B. Vekemans*¹, *L. Vincze*¹, *A. Somogyi*¹, *M. Drakopoulos*², *L. Kempenaers*¹,
*A. Snigirev*², and *F. Adams*¹

¹ *MiTAC, University of Antwerp, Universiteitsplein 1, B-2610 Wilrijk, Belgium*

² *ID22 ESRF, BP 220-F-38043, Grenoble Cedex, France*

Scanning micro X-ray fluorescence (μ -XRF) spectrometry at 3rd generation synchrotron sources offers the unique combination of multi-element compositional analysis with (sub-) micron-sized spatial resolution and trace-element sensitivity in an essentially non-destructive manner. The technique is especially appreciated for its potential to yield quantitative analytical results for practically the whole atomic number range ($Z > 13$ in air) with the exception of the light elements, reaching detection limits down to ppb levels for the most efficiently excited/detected elements.

The new ID18F microprobe at the European Synchrotron Radiation Facility (ESRF, Grenoble, France) is constructed for quantitative micro X-ray fluorescence analysis by the Micro-Fluorescence Imaging and Diffraction (ID22) group of the ESRF in collaboration with the Micro and Trace Analysis Center (MiTAC) of the University of Antwerp (U.I.A., Antwerp, Belgium). During the design and construction phase of this instrument special emphasis was placed on the stability of the set-up and to the precise monitoring of the microscopic X-ray beam impinging on the sample in terms of intensity and to a lesser extent degree of linear polarization. The short and long-term stability of the ID18F microprobe, which is one of the most important aspects of quantitative micro-analytical experiments, was characterized by repeated XRF measurements on standard reference materials. Based on these measurements, different normalization techniques are evaluated and compared.

Another important requirement of obtaining accurate analytical XRF data next to instrumental stability is the ability to (i) extract and (ii) convert experimental X-ray line intensities into elemental concentrations. Therefore for the quantification, a two-step approach is adopted. In the first step, the collected XRF spectra are deconvoluted based on a non-linear least-square fitting algorithm (AXIL) providing input (i.e., the extracted characteristic line intensities) for a detailed Monte Carlo (MC) simulation code specifically developed for XRF spectroscopy. The iterative use of the latter gives a 'no-compromise' solution for the quantification problem, taking into account specific experimental (excitation/detection) conditions, as well as sample characteristics (absorption and enhancement effects, sample topology, heterogeneity etc.).

The above described algorithm will be presented together with the evaluation of the quantification method using XRF spectra collected at ID18F from various (standard) reference materials.
