Sample preparation

Up to 100 mg of sample are mixed up with 8.9 g of Dilithiumtetraborate (LTB), purchased from FLUXANA, high purity grade and molten in a Pt/Au crucible at around 1300 °C. The melt is poured into molds, although Pt/Au, to form glass disks of 40 mm diameter. Disks are handled with care to prevent any surface contamination.

Instrument and measuring

The XRF instrument is a wavelength dispersive device *S8-Tiger*, derived from BRUKER-AXS. The radiation source is a tube with Rh-anode operated with maximum 60 kV, 170 mA and 4 kW. The exiting radiation can by filtered optionally with different Al and Cu filters to cut of Compton and Raleigh scattering of the characteristic (Rh) part. The system is equipped with collimators, ranging optional from 0.17 to 0.46 degree. A set of analyzer crystals is available, LiF 200 (2 d = 0.4026 nm) and LiF 220 (2 d = 0.2848 nm) being the most common for the energy range from 5 to 50 keV. The signal is detected by a scintillation counter in the lower energy range (up to 10 keV) and a flow-counter in the upper range. The tube-current is automatically adapted to the intensity to avoid detector saturation and dead-time problems.

The instrumental energy resolution depends on the collimator and the crystal – with LiF 200 and the 0.23° collimator the resolution is about 60 eV at 7.04 keV (Fe Kα).

Amount of a given element is calculated by iteration following the Lachance-Traill formalism based on peak-high intensity; background correction is done by 2-point linear fitting.

The information depth (related to the fluorescence volume) depends on the energy of the X-ray line observed and the matrix composition. It ranges from 10 and more centimeters for high energetic lines in light sample matrices to the sub-micron range for the lines of light elements in heavy matrices.

Data evaluation is done with the software *Spectra-plus*, purchased from BRUKER-AXS. Sample concentration is calculated by iterations.

The glass specimen are picked up by an autosampling system of the XRF instrument and measured in vacuum. The sample is rotated with a frequency of 0.5 min-1 during measurement.

Calibration Procedure

As the chemical state of a given element is almost of no concern for the method, standard samples for calibration are prepared from the most appropriate compound available. On the other hand, matrix composition and preparation (glass-melt, boric-acid-pellet or other) will matter. It is desired that calibration standards and samples for analysis are identic in these parameters.

For a multi-element problem the parameter space is reduced. A balance point composition close to the assumed sample composition is defined. For a set of calibration standards, the concentration of one element is varied around this center, the ratio of all other elements kept constant.

The calibration factors are calculated by linear regression, plotting the α-corrected intensities (Lachance-Traill) versus chemical concentration in the calibration standards. Normally, intercept and α are not fixed but optimized and calculated from the regression routine.