SR-TXRF at Beamline L: Performance and XANES Applications


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At HASYLAB, beamline L a vacuum chamber for SR-TXRF analysis is now available which can easily be installed using the adjustment components for microanalysis present at the beamline [1]. The detector is in the final version a VORTEX Silicon Drift Detector (SDD) with 50 mm² active area from Radiant Detector Technologies. With the Ni/C multilayer monochromator set to 17 keV on a sample containing 100 pg of Ni extrapolated detection limits of 8 fg have been obtained using the 50 mm² SDD for 1000 s live time.

Various applications have been tested, specially of specimen with very small sample mass as ng amounts are enough to achieve an intensive fluorescence signal. The applications for aerosol samples are described in another report of this volume. An important application is the determination of the chemical state of a particular element present in the sample by XANES with tuned SR. Using the Si 111 crystal monochromator also available at beamline L, XANES measurements to determine the chemical state have been performed, but with lower sensitivity than typical TXRF measurements, as the flux transmitted by the crystal monochromator is about a factor of 100 lower than that transmitted by the multilayer monochromator. XANES measurements of As in xylem sap from cucumber plants fed with As III and As V have been performed and detection limits of 170 ppt in xylem sap have been achieved (see Fig. 2). This sample represents an organic matrix prepared without digestion or any other sample preparation technique, except drying the liquid on a hot plate. The result of the XANES measurements is shown in Fig. 3 showing 2 identical spectra from the 2 xylem saps. The oxidations
states of the two saps seem to be identical, but are different from that of a dimethyl arsinic (DMA) acid standard (see Fig. 3 left)

![Fig. 2 Spectrum obtained during XANES scan from xylem sap. Detection limits in the range of 170 ppt for As have been found.](image1)

![Fig.3.XANES spectra of As in xylem sap feeded with AsIII, AsV and a standard sample from DMA (left), XANES spectra of Cr from urban dust SRM 1648](image2)

Finally, speciation on Cr in urban dust on the SRM 1648 has been performed. It turned out to be CrIII as can be seen in Fig. 3 (right).

In conclusion, with the multilayer monochromator detection limits in the low fg range can be achieved. Using the Si111 monochromator XANES measurements can be performed, detection limits of As in organic matrix were found to be 170 ppt. Also the chemical state of Cr in the SRM 1648 urban dust has been determined to be CrIII. So, speciation on samples with small sample mass available could be performed using the SR-TXRF setup at beamline L.

**References**


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