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Development of X-ray fluorescence methods by use of a Mo X-ray tube and their application in the case of biological samples

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Abstract

In the present work two different experimental arrangements, aiming in the X-ray fluorescence analysis of solid and liquid biological samples respectively, were developed and studied both theoretically and experimentally in combination with a Mo anode X-ray tube: The secondary target XRF (STXRF) and the Total Reflection XRF set-up (TXRF).

The secondary target XRF irradiation chamber was designed and constructed in our laboratory. Careful choice of the materials combined with the tight geometry used, minimize the presence of interference peaks in the spectrum and offer high intensity of the exciting beam at the sample position. Optimum performance was ensured by means of an extended theoretical and experimental study of various parameters affecting the analytical capabilities of secondary target XRF systems, such as: The choice of secondary target material and the operational conditions of the X-ray tube. The secondary target XRF set-up was applied in the determination of the concentrations of trace elements in a pool blood serum sample. A simple preparation technique, like the freeze-drying process was employed, resulting in detection limits of 15 ng/ml for most metal trace elements and for a 6000 sec measuring time. The accuracy of the whole experimental procedure, examined by internal standardization and by analyzing the reference standard material A-13, was found to be about 4% over the whole atomic number range. For the determination of the mass absorption coefficients, necessary in the quantification step, a modified version of the emission transmission method was employed.

Total Reflection X-Ray Fluorescence (TXRF) is a well-established method, mainly applied in the analysis of liquid samples, offering very low detection limits in most of the cases. Direct application of the TXRF method is not so efficient in blood serum analysis, since the high content of the organic matrix increases significantly the background due to Compton scattering. Chemical treatment of the blood serum samples and related preconcentration techniques have been suggested in literature, but they are time consuming and increase the possibility of adding contaminants in the sample. In this work, the applicability of direct TXRF analysis in blood serum samples was examined. The insertion of a Mo filter after the cut-off reflector has been found to improve significantly the peak to background ratio, especially for the elements of interest such as Cu, Zn, Se and Br. The influence of self-absorption phenomena in the quantification procedure was also investigated with respect to the internal standard used and the sample mass analyzed. Precision and accuracy in the analysis was found to be about 4% over the whole atomic number range. Detection limits were in the order of 25ng/ml for most metal trace elements and a measuring time of 3600 sec.