

WATER ANALYSIS USING A PROTOTYPE MONOCHROMATIC MICROFOCUS GRAZING INCIDENCE X-RAY FLUORESCENCE DEVICE COMPARING PICOLITER AND NANOLITER DEPOSITION AS SAMPLE PREPARATION APPROACH

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Determination of trace elemental amounts in water is the major focus of many analytical approaches. Analysis of contaminants in water is crucial to ensure a safe and healthy working and living environment. There are many examples for the need of toxic element determination in water such as the analysis of As in well water, monitoring of tracer elements like Cr, Pb, Cd, Mn, Hg and Sb in industrial waste water and the trace elemental analysis in biological fluids like urine.

Inductive Coupled Plasma (ICP) Optic Emission Spectrometry (OES) and ICP Mass Spectrometry (MS) are frequently used for water analysis as they provide limits of detection typically in the single digit $\mu\text{g/L}$ to single digit sub- ng/L range depending on the method and the element. Drawbacks of these methods are the need to dilute the sample which is unfavorable where sample amounts are limited. They are hampered by high matrix concentrations which limits their use for the analysis of biological fluids and sea water. Additionally the need for high purity Ar is a significant impediment to field deployment.

Therefore in this study we evaluate the performance of grazing incidence monochromatic micro X-ray fluorescence (GIMMXRF) for analysis of contaminants in aqueous samples. The GIMMXRF is a surface analytical technique for ultra-trace analysis of particles, residues, and impurities on smooth surfaces such as dried water droplets on a polymeric film. The instrument employs a doubly-curved crystal (DCC) optic which captures a wide angle of X-ray photons from an X-ray source and forms a converging monochromatic beam. This lowers the background significantly and improves the S/N for elemental determinations providing high sensitivity and low detection limits.

The small size of the prototype (1 x 0.4 x 0.3 m) enabled by using a low power X-ray tube and a pin diode detector makes it a convenient instrument to be used in the field.

The capability to analyze submicroliter amounts of water samples is a clear advantage compared to the methods mentioned above; still high matrix concentrations can also hamper the GIMMXRF analysis. Since the control of the drying process is limited, specimens may suffer from inhomogeneous distribution of standard and analyte and easily exceed the critical thickness required for a precise analysis with GIMMXRF (ca. 4 μm). Smaller thickness and higher homogeneity of the specimens can be achieved by preparing the sample in arrays of very small droplets. Therefore in this study we compare the precision and sensitivity of analysis of aqueous samples prepared in arrays of nanoliter versus picoliter volumes.