

SAMPLE PREPARATION STRATEGIES FOR TRACE AND SPECIATION ANALYSIS BY TXRF SPECTROMETRY: PAST, PRESENT AND FUTURE

E.Marguí¹, I.Queral², M.Hidalgo¹, C.Streli³

¹ Department of Chemistry, University of Girona, Campus Montilivi, 17071 Girona, Spain.

² Laboratory of X-ray Analytical Applications (LARX). Institute of Earth Sciences “Jaume Almera”, CSIC. Solé Sabarís s/n. 08028 Barcelona, Spain

³ Atominstitut TU Wien, Stadionallee 2, 1020 Wien, Austria

Due to the inherent surface sensitivity of TXRF, since its development, this technique has been primarily used for controlling contamination of silicon wafers in the semiconductor industry and for trace element analysis of liquid samples deposited on a reflective carrier. However, the use of TXRF in the field of chemical analysis has been reduced in the last years mostly due to the increasing use of ICP-MS instrumentation. Although ICP-MS present sufficient sensitivity and multielement capability for the determination of elements at trace and ultratrace levels, the complexity of most environmental matrices significantly hamper the analysis. An additional shortcoming is that several mL of liquid solution are needed to perform analyses and thus, the application to mass-limited samples is restricted. For this reason, in some applications, above all concerning to the analysis of complex liquid samples, mass-limited samples and suspensions, TXRF spectrometry is still unique.

In the last decades, most of the published TXRF analyses were performed using large-scaled instruments with high-power X-ray tubes, demanding water-cooling systems and liquid-nitrogen cooled detectors. However, in recent years, the development and commercialization of bench top TXRF instrumentation, which offer extreme simplicity of operation in a low-cost compact design, have promoted its application in industry as well as in research activities for trace element analysis. Despite that the limits of detection using such instrumentation are higher than those achieved by large-scaled equipments, they are suitable for some environmental applications. In any case, an improvement of the limits of detection can be assessed by using sample preparation or preconcentration strategies prior the TXRF analysis. In this respect is important to remark the recent development of faster, simpler, inexpensive and more environmentally-friendly analytical procedures for metal preconcentration within the framework of the so-called “green analytical chemistry” such as liquid-phase microextraction (LPME). Taking into account the micro-analytical capability of TXRF spectrometry, the combination with LPME procedures to isolate metals or metallic species from the liquid solutions can significantly improve the analytical performance of trace and speciation metal analysis by TXRF.

In the present contribution, the feasibility of several analytical approaches combined with TXRF for trace and speciation analysis of different type of samples in the industrial and environmental field is presented. This analytical strategies included the direct analysis of the samples by depositing some μL of sample on a reflective carrier and also the development of fast and simple chemical strategies to decrease matrix effects and improve the limits of detection for metal determination, such as cation exchange procedures to reduce the high matrix element concentration or LLLME procedures to isolate and preconcentrate the target analytes. These analytical methodologies have been applied in combination with different configurations of TXRF spectrometers (Mo/W X-ray tubes), including laboratory instrumentation (X-ray tubes up to 3kW) and benchtop equipments (50W power), to achieve reliable analytical results for environmental and industrial analysis at many different scales.

Our results give insight into the possibilities and drawbacks of TXRF analysis and to a certain extent the potential applications in the environmental and industrial field. It is expected that future improvements in instrumentation and sample preparation procedures could increase, even more, the analytical sensitivity and thus, TXRF spectrometry could offer new possibilities in these fields in the upcoming years.