Analytical requirements for quantitative X-ray

fluorescence nano-imaging of metal traces in solid samples

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A breakthrough generation of hard X-rays nano-imaging beamlines, focusing 10¹²⁻¹³ photons/s in a few tens of nm diameter spots in the 10 to 30 keV range, is under construction or commissioning at upgraded third generation synchrotrons. Hard X-ray nano-XRF imaging opens the fields impeded by the quantification of metal trace elements (few to hundreds of ppm), for non-destructive, *in-situ* XRF imaging of nano-structures (50-1000 nm) embedded in inorganic and/or few micrometers sample volumes, on/in a sample holder/container/device. Most applications are no longer limited by the Minimum Detection Limits (MDL) nor by a lack of computational tools necessary for the XRF analyses as was the case for micro-XRF in former times [1, 2]. The key issue that is nowadays emerging is the sample preparation.

Adopting the perspective of users, we present an inexhaustive overview of the specific key issues emerging from the first experiments carried out at the ID16B hard X-ray nanoprobe of the ESRF [3]. The evaluation of the limiting factors of specific nano-XRF analyses, the sample preparation and the related analytical sensitivities that can be expected are fully covered and illustrated by a unique nano-XRF experimental study of a 3.35 Ga old microstructure of putative microbial origin from Barberton (South Africa). The emphasis is laid on practical guidelines to anticipate the listed issues and on their rationale, so the range of this survey concerns all the fields investigating solid samples. A customized use of Focused Ion Beams (FIB) backed by state of the art Monte Carlo XRF modeling to initiate preparations of new samples and certified standards provides a way of getting below 5 ppm MDLs for acquisition-times of 10 seconds with an analytical precision better than 10 % [3].

[1] K. Janssens, W. De Nolf, G. Van Der Snickt, L. Vincze, B. Vekemans, R., Terzano, F.E. Brenker, TrAC. 29 (2010) 464-478.

[2] S. Vogt and A. Lanzirotti, Synchrotron Radiat. News, 26 (2013) 32-38.

[3] L. Lemelle, A. Simionovici, T. Schoonjans, R. Tucoulou, E. Enrico, M. Salomé, A. Hofmann, B. Cavalazzi. TrAC 91 (2017) 104-111.