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Synchrotron Radiation X-Ray Fluorescence nanoanalyses of the metallome of a ∼3.3 Ga-old microbial biofilm from the Barberton greenstone belt, South Africa.

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Combining in situ nanometer-scale techniques on the fossilized Josefsdal Chert Microbial Biofilm (JCMB) reveals a distinct vertical structural and compositional organisation: the lower part is calcified as aragonite, while the upper non-calcified kerogenous layer is characterised by up to 1% sulphur [1]. The in situ analysis of all the metals as a group represents a useful microbial fingerprint [2] and we will continue to explore it.
Synchrotron Radiation X-Ray Fluorescence maps of high spatial resolution (< 0.3 μm) were recorded on a unique FIB section (15 x 10 x 3 μm3) of the JCMB. A 300 nm resolution was reached at 2500 eV on the ID21 scanning X-ray microscope (SXM) and a 120x165 (horizontal x vertical) nm at 17450 eV on ID22NI at the European Synchrotron Radiation Facility (ESRF). All maps reveal chemical heterogeneities not previously discernible by scanning the same FIB cut using micron resolution. The feasibility of high-resolution analyses with high flux on rock samples was first shown for samples that had in this respect an ideal hotspot geometry in a uniform silica matrix [3, 4]. Our FIB sample preparation ensures negligible thickness variations so quantitation of all the metals in the JCMB is presently only limited by the important intrinsic heterogeneity of the sample. Methods to deal with micrometer bulk heterogeneity have just been developed by performing redundant volumetric scans in fluorescence tomography to counterbalance the complex sample geometry [5]. An alternative methodology more adapted to the thin slice geometry is tested here.
A relationship between the ratio of the Compton to Rayleigh (C-R) scatterings and the average atomic number (Zave), only established with unpolarized X-rays [6], was measured using fully polarized synchrotron beams [7]. C-R peaks measured on thick Astimex standards (1 mm) and those calculated from Monte-Carlo simulations of thick and thin (100 μm and 1 μm) samples having the same compositions were analyzed using a version of the PyMCA software [8] specially optimised for this purpose. The empirical relations of the type Zave = a•(C/R)b obtained for the three analyses sets provide a comprehensive set of calibrations suitable for any sample of any thickness. On the basis of these calibrations, we inferred positions and concentrations of undetected low-Z phases in the JCMB and further corrected the concentrations of the detected metals in the organic phases.

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