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Light elements (C, N,O) quantification by TEM-EDS

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EDX spectroscopy in transmission electron microscopes (TEM) has made tremendous progresses recently regarding the collection efficiency of light elements (C, N, O), thanks to the new generation of windowless silicon drift detectors. Quantifying these elements was so far perilous because of low signal to noise ratio and poor reproducibility. Another major limitation was the significant absorption of low energy X-rays within the samples, which must be corrected and requires a good knowledge of the thickness and density. Here, we determined the calibration factors (k-factors), we developed new methods for estimating the mass thickness (thickness*density) and quantified the uncertainties related to the absorption correction procedure. This development allows us to map and quantify -pixel by pixel- the composition of light elements-bearing materials, such as organics and/or water bearing phases.

We acquired EDX spectrum-image of standards across regions of variable thicknesses. k-factors are obtained by extrapolating the elemental ratio (C/Si for instance for a silicon carbide) to an equivalent thickness of zero, i.e. where no absorption occurs, using the extrapolation method of [1]. For oxygen, we used various silicates and obtained a mean value of 0.98. For nitrogen, we used Si_3N_4 and obtained a value of 1.24. For carbon, we used silicon carbide and calcite. The situation is more complex, the calibration curve appearing as non linear (Fig.). This might be due to an over-simplification of the extrapolation equations [1]. In the absence of alternative theories, we extracted k-factors values obtained for different thickness ranges and then extrapolated these values. The average C/Si k-factor 1.42 (±0.06). We also estimated the quantification uncertainties related to the absorption correction by running an 'inverse' quantification computed through HyperSpy [2]. We created various thickness maps and observed how the C/Si ratio varies. For an organic material (density ~ 1.5, thickness ~ 100 nm), a change of 15% of the mass thickness induces less than 5% deviation of the C/Si. High precision N/C and O/C ratios of organics can thus be obtained. Applications on Orgueil will be presented.

[1] Horita, Z. et al. (1987). *Ultramicroscopy*, *21*(3), 271-276.
[2] De la Pena et al. https://doi.org/10.5281/zenodo.592838