
Reducing the gap between field and laboratory x-ray fluorescence analysis

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Soil samples preparation methods for X-ray fluorescence analysis diverge between different studies. An official method is described in US-EPA 3200, but many authors do not follow this recommendation and propose adaptation according to the specificity of the analyzed matrix. Furthermore, XRF analysis is supposed to be rapid and precise for in situ mapping or lab bench measurements. The succession of preparation procedures can delay the acquisition of results: if the duration of samples preparation is equivalent or superior to a standard total acid-extraction procedure, the use of the XRF method can be questionable.

Portable XRF devices (pXRF) are typically designed for in situ mapping. However, researchers frequently collected and analyzed soil samples back home in their laboratory, for different reasons: (1) need to investigate deeper soil horizons, (2) reduction of time-consuming

field analysis (soil sampling can actually be faster than XRF analysis), (3) comfort and safety reasons (i.e. weather conditions), (4) requirement of extra analyses, etc.... As the soil samples are supposed to represent the field conditions, the correlation between field results and laboratory results is decisive.

The first experiments in this study aimed at optimizing the analyzing time and ensure measurement quality: (1) calibrations using different organic and mineral reference materials, (2) effect of time measurement on calibration. Then, we focused on the effects of sample preparation of XRF measurements, by comparing different procedures: (a) Directly from field, (b) after drying, (c) after sieving 2 mm, (d) after grinding at 6m/s, until 180 microns (80mesh), (e) after burning at 550oC (elimination of organic matter). We confirmed that a 2 min time measurement is sufficient for accurate and precise pXRF results. As expected, a strong effect of the organic matter appears during the analysis. We could distinctly model two calibration curves using two groups of certified references material: 8 mineral samples (soils and sediments) and 19 organic samples. Within the second group, we included aquatic plants, lichen, peat, leaves and coal samples. The slope of the calibration curve is three to four times attenuated in these organic samples, so specific calibration according to soil matrix is mandatory. Concerning the preparation procedure, most of the elements (40 of 50 elements) presented a low variability (<8%) within analytical replicates, even after shaking the powder within the cup. Among the main elements, only Fe, Ca and Zr had >8% variability. Burning of the sample at 550°C does not change the chemical composition (which must be corrected due to the loss of organic matter). Surprisingly, even the 2mm sieving does not significantly modify the XRF measurements. Finally, we observe that fine grindind (<180 µm)

provides results far from the real field values, probably because the procedure generates metal-rich fine particles that can accumulate at the bottom of the capsule where the XRF measurement is made. We conclude that the preparation of the samples should be minimal to avoid laboratory bias. This makes the XRF scans faster, which is the main purpose of this type of analysis and allows to get closer to the real conditions of the field.
