

## **Deciphering Ni sequestration in soil ferromanganese nodules by combining X-ray fluorescence, absorption, and diffraction at micrometer scales of resolution**

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### **ABSTRACT**

X-ray microprobes are among the most important new analytical techniques to emerge from third generation synchrotron facilities. Here we show how X-ray fluorescence, diffraction, and absorption can be used in parallel to determine the structural form of trace elements in heterogeneous matrices at the micrometer-scale of resolution. Scanning X-ray microfluorescence ( $\mu$ SXRF) and microdiffraction ( $\mu$ SXRD) first are used to identify the host solid phase by mapping the distributions of elements and solid species, respectively. Micro-extended X-ray absorption fine structure ( $\mu$ EXAFS) spectroscopy is then used to determine the mechanism of trace element binding by the host phase at the molecular scale. To illustrate the complementary application of these three techniques, we studied how nickel is sequestered in soil ferromanganese nodules, an overwhelmingly complex natural matrix consisting of submicrometer to nanometer sized particles with varying structures and chemical compositions. We show that nickel substitutes for  $Mn^{3+}$  in the manganese layer of the  $MnO_2$ - $Al(OH)_3$  mixed-layer oxide lithiophorite. The affinity of Ni for lithiophorite was characteristic of micronodules sampled from soils across the U.S.A. and Europe. Since many natural and synthetic materials are heterogeneous at nanometer to micrometer scales, the synergistic use of  $\mu$ SXRF,  $\mu$ SXRD, and  $\mu$ EXAFS is expected to have broad applications to earth and materials science.