Goldschmidt 2025 Abstract
https://doi.org/10.7185/gold2025.28987

Can we distinguish colloidal-bound from mineral-included metal species using AF4-ICP-MS measurements?

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Asymmetric flow field-flow fractionation linked to inductively coupled plasma mass spectrometer (AF4-ICP-MS) is a powerful technique for addressing the size distribution and elemental composition of natural nanoparticles systems (NPS). Trace metals (TM) distribution among the different natural nanoparticles (NNPs) size-populations can be quantitatively measured, giving insights into their fate, transport, and potential bioavailability. Despite AF4 being a powerful technique in terms of nano-scale size resolution, the state of matter of TM (i.e. metal-chelated, adsorbed or included into / forming mineral phase) is impossible to accede by using common detectors associated online. We propose herein a methodology based on ligand-exchange, to distinguish “bound” from “(nano)particulate” metal species within NPS. The example of NPS (< 1.2 µm) concentrated (>1 kDa) from Paudèze River (CH), will be presented employing 30 min and 48h incubations with ethylenediaminetetraacetate (EDTA) and deferoxamine mesylate (DFOM).

To selectively focus on chelates formed with humic-like substances (HS), a 300 Da cut-off membrane was first used in AF4. Both EDTA and DFOM employed at 1mM were efficient, according to their complexation constants, to dissociate Cu-, Fe-, Pb-chelates formed with HS, without perturbating the UV fractogram associated with macromolecular humic substances. However, the results highlight that the difference in carrier fluid used for the analysis (10mM NH4NO3 vs. (NH4)2CO3) led to differences in the rejection of EDTA- or DFOM- metal complexes formed, impairing the quantification of Pb/Cu/Fe or Fe-only in the presence of EDTA or DFOM respectively, when using NH4NO3. A membrane of larger poor size (1 kDa cut-off) made of regenerated cellulose and a carrier fluid of 0.1 mM (NH4)2CO3 was used to distinguish TM fractions associated with the NNPs. Minor changes were observed at short-term incubation for Fe-or Pb-NNPs, and only a slight decrease in their proportions for 48h incubation was measured suggesting the presence of inorganic iron with minor dissolution under our conditions.

Future AF4-ICP-MS measurements combined with other ligand types on different NPS will specify how such a strategy enables distinguishing “bound” vs. “mineral-included” TM at the nanoscale. Such new developments are important to estimate TM bio-accessibility within NPS.