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The potential of on-line continuous leach ICP-MS analysis for linking trace elements to mineralogy

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A set of five soil samples was subjected to an on-line continuous leach inductively coupled plasma mass spectrometry experiment, with progressively reactive solvents (0.01M CaCl2, 0.1 M HNO_3, 1M HNO_3, 4M HNO_3) Each sample was packed in a quartz tube (Ø= 1 cm, length 2 cm) and diluted 1:1 with acid washed quartz to prevent clogging. The gas that was produced during the extraction was removed by leading the effluent into a small container, from where the sample was directly pumped into the ICP-MS. 115In was used as an internal standard. Continuous leach experiments have the advantage of real time (every 2 seconds) full elemental analysis. Mineral breakdown reactions can be monitored via the major elements. The trace elements associated with the minerals are monitored simultaneously, thus eliminating the uncertainties of host mineral-trace element combinations in traditional off-line sequential extractions. The continuous leach experimental data are correlated to XRD-results for mineralogy and total elemental concentrations.
The soil samples used were collected from different sites in the Koiliaris River watershed, Crete, Greece 1). The selection of the sites was based on variability in bedrock (limestone, metamorphic and alluvial sediments) and current land use (grape farming, olive trees). Soils were sampled at two depths: at the surface and just above the bedrock. No large differences in the major elements between the two depths were measured. To provide background to the on-line sequential data, also total concentrations of the major elements were analysed by XRF and the mineralogy was analysed by XRD. The fraction <2mm was sieved and digested with HF, HClO4 and HNO_3 for additional trace element analysis.

1) See related abstract Roskam et al., 2014: REE profiles in continuous leach ICP-MS (CL-ICP-MS) experiments in soil, linked to REE profiles in surface water in the Koiliaris River Critical Zone Observatory (CZO), Crete, Greece.