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A new methodological approach for the assessment of heavy metal pollution in soils: the use of Laser Ablation ICP-MS in thin sections coupled with traditional micromorphological and geochemical techniques

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We recently proposed an innovative methodological approach for the study of trace element distribution in soils: geochemical spot analyses were performed with laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) on thin sections prepared from undisturbed soil samples, combined with traditional micromorphological observations using both optical microscopy and scanning electron microscopy equipped with chemical microprobe analysis (SEM-EDS). This method allowed us a deeper understanding of pedogenetic processes, especially in terms of microscale spatial variability and particle/element retention and mobility, through chemical analysis of different subportions of soil horizons (illuvial pedofeatures, pedogenic matrix and skeletal rock fragments). Now we apply this new technique to detect microscale distribution of heavy metals in the above discrete soil features of different organic-mineral (A) and argillic (Bt) horizons from two soil profiles in SE Sardinia, Italy. Among all the trace elements detected, here we focus on Ti, V, Cr, Mn, Fe, Co, Cu, Ni, Zn, Cd, Mo, Ag, Sn, Sb, Pb, As, Th and U. Ablation was performed with a 213 nm laser beam on 80 μm thick sections obtained from the same impregnated and consolidated blocks used for classical (30 μm) thin sections, representing their counterparts. This setup permitted in situ analysis of chemical concentrations with a high degree of accuracy and precision, and detection limits from 1-10 mg/kg up to 0.1 and 0.01 mg/kg, i.e. considerably lower than those reached by other microanalytical techniques (EDS, WDS, etc.). Trace element composition of the clay fraction from all soil horizons of these profiles was also determined using solution nebulization ICP-MS analysis and compared with that of corresponding bulk samples (fine earth). These results were integrated with those obtained from above laser ablation ICP-MS investigations.

An overall trend of increase is observed for many heavy metals (and other trace elements, rare earths included) from rock fragments to soil matrix (and to clay coatings in Bt horizons), especially in those horizons with higher degree of soil maturity. In contrast, poorly developed soil horizons exhibit element concentrations of matrix portions more similar to those of parent rock fragments. This behaviour clearly suggests a pedogenetic control on element fractionation, mainly by neogenesis of clay minerals (and possibly of Fe-oxides/hydroxides) from the weathering of primary components, subsequent metal adsorption onto reactive sites of secondary minerals or organic matter, and further concentration by illuviation processes of the fine fraction (in argillic horizons).

SEM-EDS microprobe analysis very rarely permitted detection of heavy metal and rare earth element concentrations in soil matrix and clay coatings. On the other hand, small grains of REE- and heavy metal-rich accessory minerals (ranging in size from a few to some tens of microns) were identified by SEM-EDS within all soil subportions of some horizons or close to the studied soil profiles: thorium- and uranium-bearing REE-phosphate (monazite) and yttrium-phosphate (xenotime), Zr-silicate (zircon), Fe- and (Fe-)Ti-oxyhydroxides (sphene and ilmenite), hydrated iron arsenate (scorodite), iron chromium oxide (chromite), various Cu, Zn and Pb oxides, sulphides or sulfates, among which lead sulphide (galena), iron lead hydroxysulfate (plumbojarosite) and arsenopyrite (iron arsenic sulphide) occur. They all likely represent the main sources of heavy metals in the studied soils. Such an occurrence, together with particularly higher metal concentrations measured by laser ablation analysis in some samples, are clear evidence of soil pollution, mostly caused by dispersal of waste deposits produced by exploitation activities in nowadays abandoned mines from surrounding areas.