Evaluation of labile metal pools in soils from a highly industrialised wetland area of southwestern Spain by single and sequential extraction methods

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The Huelva estuary, on the Atlantic coast of southern Spain, is a wetland strongly polluted by acid mine drainage and industrial effluents from a range of chemical factories and oil refineries. Average total metal concentrations in 15 topsoil samples (<2 mm size particle), determined by XRF spectrometry, are (values in mg kg^{-1} dry wt.): 737 for Cu, 400 for Zn, 179 for Pb, 98 for Cr and 90 for As. Other metals analysed (Ni, V, Co and Cd) are within the soil geochemical background concentrations. Single and sequential extraction procedures were conducted in order to assess the mobile and potentially mobile fractions in soils. Metal concentrations in all the extract solutions were measured by ICP-AES.

The most labile metal fraction assessed by single extraction with both deionised water and a mild neutral salt solution (0.01 M $CaCl_2$) is generally negligible (< 1%). Locally, in acid soils (pH~ 4.2) affected by chemical spills, the water-soluble fraction of Cu and Zn accounts respectively for 11.8% and 6.2% of the total content. In such soils, the $CaCl_2$-extractable concentrations are up to about 63% of Cu and 27% of Zn, indicating a significant exchangeable metal pool. The percentages extracted with 0.05 M EDTA at pH 7 are much higher (up to 61% of Cu; 58% of Pb; 46% of Zn; and 14% of As) than those obtained using the other two single extraction methods. Chemical partitioning of these mobilisable fractions, achieved using the modified BCR three-step sequential extraction scheme [1], suggests that Zn is preferentially associated with acid-soluble phases, Pb and As are bound to Fe oxy-hydroxides (reducible fraction), and Cu appears to be similarly partitioned into reducible and oxidisable phases.

[1] Rauret *et al.* (1999) *J. Environ. Monit.* **1**, 57-61.

Timing of injection and of thermal maturation in a mid-crustal Variscan bimodal intrusion

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Recent advances in IDTIMS U/Pb geochronology allow uncertainties of better than 0.1% in the $^{206}Pb/^{238}U$ date of individual zircon analyses. However, increased analytical precision requires greater caution assessing geological uncertainties such as lead loss, incorporation of xenocrystic or antecrystic zircons, and protracted zircon crystallization This is especially important when constraining magma emplacement times, which thus may or may not be recorded by zircon dates within a rock. Combining zircon dates with careful field observation can help assess the accuracy of interpreting zircon age data in terms of magma injection, for example by documenting the relative ages of successive magma pulses. High-precision IDTIMS U/Pb zircon dates corroborate relative ages of incremental magma pulses recorded in the St-Jean-du-Doigt (SJDD) bimodal layered intrusion (Brittany, France). Early injections emplaced at low rates in a cool environment with little interaction among successive magma pulses. Later injections occur at higher rate in a progressively hotter environment with protracted mafic felsic magma interaction. Zircon dates confirm that the early activity predates the thermally mature episode by about 1 Ma, starting at ca. 348 Ma, whereas the last pulse is recorded at ca. 346 Ma. Dates on single zircon grains displaying core-rim relationships span about 4 Ma (350-346 Ma), which we interpret to represent two distinct crystallization events. Hf isotopic data show homogeneous values for cores and rims, precluding inheritance. This could relate to zircon saturation fluctuations at the emplacement depth, or to antecrystic zircons recording pre-emplacement magmatic growth.