Sequential extraction procedure

Five fractions were extracted from 1 g of wet solid sample using different reagents:

- Soluble and exchangeable metals (extracted with 8 mL MgCl₂ (0.5 M), pH 7.1 h at room temperature)

- Carbonate bound (leached by 8 mL NaOAc 1 M buffered with HOAc, pH 5.5 h at room temperature)

- Reducible or bound to Fe-Mn oxides (extracted with 20 mL NH₂OH-HCl (0.04 M) in 25% (v/v) HOAc, 6 h at 96°C)

- Oxidisable or bound to organic matter (released by 3 mL HNO₃, 5 mL H₂O₂ (30%) pH 2.2 h at 85°C, then 3 mL H₂O₂ (30%) pH 2.3 h at 85°C, and 5 mL NH₄OAc (3.2 M) in 20% (v/v) HNO₃, 0.5 h at room temperature). Solution was diluted to 20 mL.

- Residual metal fraction (dissolved by acid attack with HNO₃, HF, and HCl).