



Sequential leaching is a technique to group elements with their host mineral phase or with a group of minerals. It consists of four steps. (1) Exchangeable cations and calcium carbonates: 1 g of powdered sample was stirred with 1 M acetic acid/Na acetate buffer (pH 5); (2) Easily reducible fraction: The residue of leach 1 was stirred with 0.1 M hydroxylamine hydrochloride solution (pH 2); (3) Moderately reducible fraction: 0.2 M oxalic acid/ammonia oxalate buffer (pH 3.5) was added to the solid residue of step 2; (4) Residual fraction: The residue from step 3 was digested in Teflon bombs in a microwave, using a mixture of 48% HF and 65% HNO₃ (proportion 2/1) and dried; then 25% HCl was added and dried again. All steps were carried out twice. Hydrogen peroxide was used to dissolve organic matter.

Sequential leaching experiments were carried out in the laboratory at the USGS, Pacific Coastal and Marine Science Center. Al, As, Ba, Ca, Cd, Co, Cr, Cs, Cu, Fe, K, Li, Mg, Mn, Ni, Pb, Ti, Th, U, V, Zn, and Y were measured in the four phases using an ICP-MS PerkinElmer NexION 300Q (USGS, Menlo Park, operators N. Konstantinova). For each element, the sum of the amounts released by leaching steps 1-4 was compared with the bulk analysis data to check recovery, which was between 80% and 120%.