Trace metal stream contamination in a post peak water context: Learnings from the Cordillera Blanca, Peru

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Supplemental Materials

Details of extraction protocols

This section provides descriptions and details concerning the sequential digestion to matrix (SDM) protocol:

Step 1 (Exchangeable). 10 mL of Milli-Q water was added to 50 g of sediments in a 50 mL HDPE vial. Tubes were then shaken at 280 rpm in an Infors HT Ecotron at ambient air temperature. After 16 hrs, the tube was centrifuged at 3000 rpm¹ for 20 min and rinsed 3 times with 2 mL of Milli-Q water. Supernatants were removed and put into 15 mL tubes ready for analysis, and residues were dried at 40 °C.

Step 2 (Carbonatic phase). 10 mL of 0.11 mol.l⁴ acetic acid² was added to the residue, and the tube was shaken for 16 hrs at 280 rpm, centrifuged at 3000 rpm, rinsed 3 times with acetic acid 0.11M. Supernatants were removed and put into 15 mL tubes ready for analysis, and residues were dried at 40 °C.

Step 3 (Reducible). 10 mL of hydroxylammonium chloride 0.1M was added to the residue³. Tubes were then shaken at 280 rpm at air temperature. After 16 hrs, the tube was centrifuged at 3000 rpm for 20 min and rinsed 3 times with 2 mL of hydroxylammonium chloride 0.1M. Supernatants were removed and put into 15 mL tubes ready for analysis, and residues were dried at 40 °C.

Step 4 (Oxidizable). 1 mL of 8.8 M hydrogen peroxide⁴ was added to the residue and then warmed up on a hotplate at 85 °C until the solution was almost dry. This was repeated four times. After cooling, 10 mL of ammonium acetate, previously acidified to pH 2 with nitric acid, was added, and the tubes were then shaken at 280 rpm at air temperature. After 16 hrs, the tube was centrifuged at 3000 rpm for 20 min and rinsed 3 times with 2mL of ammonium acetate 1M. Supernatants were removed and put into 15mL tubes ready for analysis, and residues were dried at 40 °C.

Step 5 (sulfur). 1 mL of 8N nitric acid⁵ was added to the residue and evaporated at 85 °C until almost dry. 1 mL more of nitric acid was then added, shaken for 2 hrs at 280 rpm, centrifuged, rinsed 3 times with 8N nitric acid and diluted with Milli-Q water to 2% nitric acid (v/v), ready to be analysed.

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