Complete dissolution of geological samples using NH_4HF_2 in a microwave oven

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The obtention of trace element data of rock samples still relies, mostly, on methods that require the full dissolution of a representative powdered test portion. They consist either on an aggressive and time consuming multi-acid digestion or fusion with appropriate fluxes. In recent years, some authors introduced ammonium bifluoride as a substitute to HF in a procedure accomplished in about 10 h. Here we describe a fast and efficient dissolution method that also employs NH_4HF_2, but all digestion and evaporation steps occur in a microwave oven. The whole procedure takes 3 to 4 h and is entirely performed in a closed vessel system, therefore minimizing sample contamination.

A portion (100 mg) of each test sample was directly weighed in PTFE liners of the microwave oven (Multiwave PRO, Anton Paar, Austria). Aliquots of 1-2 mL of NH_4HF_2 (saturated solution) and 1 mL of HNO_3, both purified by sub-boiling, were added along with ultrapure water. The vessels were closed and inserted in the microwave oven rotor. The decomposition step consists of a ramp to achieve 1300 W in 5 min and a hold for 25 min, resulting in a minimum temperature of 190 °C and pressure of 5 MPa. It was followed by evaporation in 2 quick steps (10 min) with 1 mL HNO_3. Finally, samples were taken up with 2 mL HNO_3 and 5 mL ultrapure water. Samples were gravimetrically brought to 30 g and additionally diluted before analysis.

Trace elements were measured on a Q-ICP-MS with CC technology (XSeriesII, Thermo, Germany), after tuning the instrument to maximize intensities and minimize the production of bivalent ions and oxides. Calibration was performed with a set of multielement solutions prepared from mono-element solutions of 100 μg/g (AccuStandard, EUA).

Analysis of several replicates of eighteen reference materials mostly of igneous rocks and with a broad range of silica content resulted in recoveries mostly better than 90%. Besides, results submitted in two rounds of the Geo*PT* proficiency testing programme for whole rock techniques had satisfactory scores. To our knowledge, this is a first report that successfully employs a microwave oven method to dissolve resistant minerals bearing rock samples, independent of their Si or Zr content.