Rapid bulk rock decomposition by ammonium fluoride (NH₄F) in open-vessels by an elevated digestion temperature

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Complete dissolution is essential to achieve accurate analytical results for geological samples. Hydrofluoric acid is the most effective mineral acid for breaking up strong Si-O bonds to form SiF₆⁻² ions in acidic solution. In this study, decomposition technique using the neutral solid compound NH₄F in open vessel (Savillex Teflon vial) has been investigated for multi-element analysis of different types of rock reference materials. The higher boiling point (260 °C) of NH₄F allows for an elevated digestion temperature in open vessels, which enables the decomposition of refractory phases. It took 1-1.5 h for Zr to be completely recovered in GSP-2 at 250 °C, which is ~12 times faster than using conventional closed-vessel acid digestion at 190 °C (high-pressure PTFE digestion bomb). Unlike in NH₄F-assisted high pressure acid digestion, our results clearly indicate that adding HNO₃ severely inhibited the digestion capabilities of NH₄F for refractory minerals such as zircon in open vessel. The most outstanding advantage of the new method is that the digestion can be performed in a conventional Savillex Teflon vial instead of using a high pressure PTFE digestion bomb. Moreover, the NH₄F-open-vessel acid digestion is not hampered by the formation of insoluble fluorides, which represent another important advantage of this new sample decomposition method. Similar to HF and HNO₃, ultra-pure NH₄F can be produced using a conventional PFA sub-boiling purification system, and it does not induce new interference species in ICP-MS analysis. It is also worth emphasized that this reagent is removed by taking the sample to dryness, which is important to keep the total dissolved solid of the final solution presented to the instrument low. The developed NH₄F-open-vessel acid digestion has been successfully applied to the digestion of a series of international geological reference materials. This simple, effective, and comparatively safe dissolution method shows a great potential for the digestion of geological samples.

Fluid inclusion study of Maoping W deposit, southern Jiangxi province

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The Maoping tungsten deposit is a large W-Sn-poly metallic deposit discovered recently in southern Jiangxi province. It comprises two types of ore-bodies, i.e. quartz-vein and greisenized-granite type. The present paper studies the fluid inclusions in quartz and topaz occurring in wolframite-quartz-veins, which mainly includes microthermometry and Laser Raman analysis. Besides, the homogenization temperatures of fluid inclusions in wolframite were also determined under an infra-red microscope. Result shows that fluid inclusions in both quartz and topaz are mainly of liquid-rich two-phase aqueous solution (H₂O-NaCl), in addition of small amount CO₂-bearing three-phase aqueous solution in quartz. The homogenization temperatures from quartz show wider variation range and can be divided into two main periods, indicating two stages of ore precipitation. The CO₂-bearing three-phase inclusions have similar homogenization temperatures to the higher temperature period of above inclusions, but lower salinities and different homogenizing ways. Fluid inclusions in wolframite have highest homogenization temperatures and salinities, whereas those in topaz also show high homogenization temperatures and salinities, and narrow variation range. The characteristics of homogenization temperatures and salinities from fluid inclusions in quartz reveal the multiple-stage of ore forming process and the complexity of fluid evolution. It is concluded from this fluid inclusion that the early-stage boiling and late-stage mixing is probably the major mechanism of formation of wolframite-quartz-vein type ore in the Maoping deposit.