minerals

MDPI

Supplementary document

Analytical Methods

1. Electron microprobe mineral analyses

Thirty-six refined polished thin sections were prepared for mineralogical and petrographic studies using optical polarizing microscope at the Geology Department, Assuit University (Egypt). A subset of these sections was selected and coated with carbon for the analysis of major minerals (K-feldspar, plagioclase, amphibole, and biotite) and ore minerals (zircon, thorite, columbite, and nioboaeschynite) using a CAMECA SX5 electron microprobe equipped with four WDS and one EDS spectrometers at the Department of Lithospheric Research, University of Vienna (Austria). The analyses were conducted with an acceleration voltage of 15 kV, a beam current of 20nA, and a beam diameter ranging from 1 to 5 μm. Major elements had a peak count-time of 20 s and a background time of 10 s, while trace elements had a peak count-time of 40–60 s and a background time of 20–30 s. The raw data was corrected using the PAP correction method, and calibration was performed using natural and synthetic standards.

2. Whole-rock geochemical analysis

A total of 20 representative samples were selected for whole-rock major, trace, and REEs analyses. Before analysis, the samples were cleaned, ground in an electric agate mill, homogenized, dried at 110 °C, and fired at 850 °C. Whole-rock major were analyzed with a sequential Phillips PW 2400 X-ray spectrometer at the Department of Lithospheric Research, University of Vienna, using fused pellets. Replicate analyses of geo-standard GSR-3 provided an overall procedural error better than 2% for major elements.

Trace and REEs were analyzed using Laser Ablation ICP-MS at Central Analytical Facilities Lab, Stellenbosch University (South Africa). The fusion disks were prepared with an automatic Claisse M4 Gas Fusion instrument and ultrapure Claisse Flux, using a ratio of 1:10 sample: flux, coarsely crushed, and a chip of sample mounted along with up to 12 other samples in a 2.4 cm round resin disk. The mount was mapped, and then polished for analysis. A Resolution 193nm Excimer laser from Applied Spectra connected to an Agilent 7700 Q ICP-MS is used in the analysis of trace elements in bulk rock samples as well as on single mineral grains. Before analysis, the ICP-MS is optimized for sensitivity and low oxide ratios (< 0.2%) by tuning both the ICP and laser parameters while ablating a line on NIST612. Ablation is performed in He gas at a flow rate of 0.35L/min, then mixed with argon (0.9L/min) and Nitrogen (0.003L/min) just before introduction into the ICP plasma. For traces in fusions, 2 spots of 104μm are ablated on each sample using a frequency of 8Hz and fluence of ~3.5 J/cm2. NIST 610 glass was used for quantification and analyzed every 15 samples, along with BCR-2G and BHVO-2G. A fusion control standard from certified basaltic reference material (BCR-2 and BHVO-1) is also analyzed in the beginning of a sequence to verify the effective ablation of the fused material. Data processing was done using the LA-ICP-MS data reduction software package LADR from Norris Scientific (Norris et al., 2018).