

Dating a Variscan pressure - temperature loop with staurolite

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Additional specifications on sample preparation and analytical techniques

Two single staurolite crystals of about 12 by 7 by 4 mm were selected for the analyses. Both crystals showed the simple zonation described in the text. Rim (r) and core (c) were separated from each other using a miniature diamond saw device usually used for the preparation of inclusion thin slabs. The other crystal was treated as a bulk sample (t) and both U and Pb were measured. The separates were first crushed by hand in an agate mortar to grain sizes between 40 and 150 μm . Quartz, muscovite, biotite, fibrolite, garnet, and other minor solid inclusions were separated from staurolite by means of heavy liquids and subsequent hand picking. The garnet separate was obtained from crushed bulk staurolite-bearing metasediment. After ultrasonic cleaning in acetone and triply distilled H_2O , the concentrates, each weighing between 200 and 300 mg (in the case of garnet, 100 mg), were then stepwise leached with acids listed in Table 1 following a slightly modified procedure described in Frei and Kamber (1995). The final step [4] consisted of total dissolution of the residual. For the total bulk staurolite sample [t] each leach solution was aliquoted and $\sim 1/3$ of the total leach

volume was spiked with a mixed ^{208}Pb - ^{235}U tracer to determine the concentrations. U and Pb were separated on 0.5 ml quartz-glass columns charged with DOWEX AG 1x8 ® anion exchange resin using conventional HBr-HCl-HNO₃ recipes. Total Pb procedure blanks amounted to ~150 pg, whereas a U blank of <20 pg was added. Pb was statically measured on a VG Sector multicup mass-spectrometer using zone-refined 20 µm Re filaments and a silica gel - H₃PO₄ loading technique. U was analyzed on a single-cup AVCO mass-spectrometer from triple Ta-Re-Ta filaments. Fractionation of Pb amounted to $0.85 \pm 0.13 \text{ ‰} / \text{AMU}$, calculated from 85 analyses of the NBS 981 Pb standard during the past two years in our laboratory. Calculation of the final ^{206}Pb , ^{207}Pb , $^{208}\text{Pb}/^{204}\text{Pb}$ ratios and the error correlations followed the procedure of Ludwig (1980). Isochrons were calculated with ISOPLOT v 2.03 (Ludwig, 1990).

REFERENCES CITED

- Ludwig, K. R., 1980, A computer program to convert raw U-Th-Pb isotope ratios to blank-corrected isotope ratios and concentrations with associated error-correlations: US Geological Survey Open-File Report OF-82-820.
- Ludwig, K. R., 1990, ISOPLOT for MS-DOS, a plotting and regression program for radiogenic isotope data, for IBM-PC compatible computers, version 2.03: US Geological Survey Open-File Report 88-0557.