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# Contributions to precision and accuracy of monazite microprobe ages

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## ABSTRACT

We examine the factors controlling accuracy and precision of monazite microprobe ages, using a JEOL 733 Superprobe equipped with 4 PET crystals, and both 1-atm gas flow Ar X-ray detectors and sealed Xe X-ray detectors. Multiple PET crystals allow for simultaneous determination of Pb concentration on up to 3 detectors, and the effects of different detector gases on spectral form can be addressed. Numerous factors in the X-ray production, detection, and counting sequence affect spectral form, including: choice of accelerating voltage, changes in *d*-spacing of the diffraction crystal, use of X-ray collimation slits, and type of detector gas. The energy difference between Ar$K\alpha$ X-rays and Xe$L\alpha$ X-rays results in, for 1-atm Ar detectors, escape peaks of second-order LREE *L* line X-rays that cannot be filtered using differential mode PHA. The second-order LREE energies are passed to the counter and produce, for a 140 mm Rowland circle, several problematic interferences in the Pb region of a monazite wavelength-dispersive (WD) spectrum. WD monazite spectra produced with Xe detectors are free from second-order LREE interferences in the Pb region; escape peaks of the second-order LREE are filterable with differential mode PHA if Xe detectors are employed.

Silicon, Ca, Y, Ce, P, Th, U, and Pb (2 spectrometers) are measured as part of the monazite microprobe dating protocol; $\pm 2\sigma$ variations in elements fixed for ZAF corrections do not affect the age outside of analytical uncertainty. Th$M\alpha$, U$M\beta$, and Pb$M\alpha$ are the analyzed lines of the age components. Corrections for interference of Th$M\zeta_{1,2}$ and Y$L\gamma_{2,3}$ on Pb$M\alpha$ are significant, but can be done precisely, and reduce the precision of the$M\alpha$ analysis by a trivially small amount. Th$M\gamma$, $M3$-$N4$, and $M5$-$P3$ interferences on U$M\beta$ can be corrected, as well, but Th$M5$ and $M4$ absorption edges in high-Th samples make estimation of U$M\beta$ background problematic. Background fits for U$M\beta$ peaks show that linear vs. exponential fits for U$M\beta$ do not, in general, produce statistically significant differences in microprobe ages. However, linear vs. exponential background fits for Pb$M\alpha$ peaks do produce significantly different ages, most likely because of (1) low Pb concentrations relative to U; (2) Th$M\zeta_1$ interference on backgrounds between Th$M\zeta_1$ and Pb$M\beta$; and (3) S$K\alpha$ and $K\beta$ interference in S-bearing monazite.

For 6-min analyses (3 min peak, 3 min background) at 25 keV and 200 nA, $1\sigma$ Pb precisions are approximately 3–4% at 1700 ppm and 9.5% at 750 ppm; at 15 keV, precision decreases by roughly 25% of the 25 keV value. These precisions are constant for fixed current, analysis time, and concentration, but the statistical precision of distinct populations of monazite grains (domains) is a function of the total number of analyses within the domain. Instrumental errors (current measurement, dead time, pulse shift, *d*-spacing change) add 1–10% to random errors, but errors caused by pulse shift and *d*-spacing changes can be accounted for and corrected. Decreasing accelerating voltage from 25 to 15 keV decreases ZAF correction factors by as much as 50% relative, but replicate age analyses of Trebilcock monazite at 15 and 25 keV are statistically indistinguishable. Grain orientation, miscalculated background intensity, uncorrected interferences, and surface effects also introduce systematic errors. Accurate background interpolation and interference correction reduces systematic error to approximately 5–20% in addition to random (counting) error.

Microprobe ages (~420 Ma) and $^{208}Pb/^{232}Th$ SIMS ages (~430 Ma) of monazite from Vermont are in agreement to within ~10 m.y. The discrepancy between U-Th-total Pb microprobe ages and $^{208}Pb/^{232}Th$ ages is removed when the high background measurement for Pb$M\alpha$ is shifted to the short-wavelength side of Pb$M\beta$, removing a possible Th$M\zeta_1$ interference.