

U-Pb Geochronology of Columbite-Tantalite by LA-ICP-MS: Method Evaluation and Reference Material Development

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Columbite-tantalite, characterized by high U and Pb concentrations, is a critical material for U–Pb geochronology, particularly in rare-metal granites, pegmatites, alkaline, and alkaline-carbonatitic systems. To achieve accurate U–Pb dating of columbite-tantalite using LA-ICP-MS, matrix-matched reference materials are required to correct elemental fractionation. However, most available reference materials contain appreciable amounts of common Pb, and those suitable for primary calibration are extremely lacked. In this study, we present an evaluation of columbite-tantalite U–Pb dating results obtained with both matrix-matched and non-matrix-matched approaches. Additionally, two new potential reference materials, OXF, and Coltan2, were evaluated for LA-ICP-MS U–Pb dating of columbite–tantalite. Under matrix-matched conditions, accurate U–Pb ages were achieved using large spot size (32 μm , 44 μm), low repetition rate (2, 4, 6 Hz), and low energy density (4, 6 J/cm²). Moreover, accurate U–Pb ages were obtained by calibrating against zircon Tanz with water vapor introduction, combined with large spot size (32 μm , 44 μm) and low repetition rate (2, 4 Hz). The U and Pb content of OXF are 428 $\pm$ 156 ppm (2s) and 20.0 $\pm$ 10.0 ppm (2s), and 1379 $\pm$ 172 ppm (2s) and 163 $\pm$ 103 ppm (2s) for Coltan2, respectively. The weighted mean ²⁰⁶Pb/²³⁸U ages from CA-ID-TIMS analyses are 262.85 $\pm$ 0.64 Ma (MSWD = 4.4, 2s) and 512.41 $\pm$ 0.45 Ma (2s, n = 7), respectively. The obtained weighted mean ²⁰⁶Pb/²³⁸U ages are 262.83 $\pm$ 0.29 Ma (OXF, 2s, n = 358) and 510.8 $\pm$ 3.6 Ma (Coltan2, 2s, n = 75) from LA-ICP-MS determination, which are consistent with the CA-ID-TIMS results within the analytical uncertainties. The results demonstrated homogeneous distribution of U–Pb ages in OXF and Coltan2, indicating their suitability as primary reference materials for LA-ICP-MS analysis.