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Complete zircon and chromite digestion by sintering of granite, rhyolite, andesite and harzburgite rock reference materials for geochronological purposes

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Zircon ($ZrSiO_4$) is a common accessory mineral in nature that occurs in a wide variety of sedimentary, igneous, and metamorphic rocks. Zircon has the ability to retain substantial chemical and isotopic information that are used in range of geochemical and geo- chronological investigations. Sample digestion of such rock types is a limiting factor due to the chemical inertness of zircon ($ZrSiO_4$) tourmaline, chromite, barite, monazite, sphene, xenotime etc. as the accuracy of results relies mainly on recovery of analytes from these minerals. Dissolution by wet acid digestions are often incomplete and high blank and total dissolved solids (TDS) contents with alkali fusions lead to an underestimation of analyte concentrations. Hence an effective analytical procedure, that successfully dissolves refractory minerals such as zircon is needed to be employed for reliable analytical results.

Na_2O_2 digestion [1] was applied in characterisation of granite (G-3), rhyolite (MRH), andesite (MGL-AND) and harzburgite (MUH-1) powdered reference material with solution based ICP-MS analysis. In this study we undertake a systematic evaluation of decomposition time and sample:Na_2O_2 ratio and test portion size after minimising effect of all other constraints that makes homogeneity ambiguous. In recovering zircon and chromite 100 mg test portion was mixed with different amounts of Na_2O_2 i.e. 100-600 mg. Impact of decomposition time was observed by systematically increasing heating time from 30-45 minutes to 90-120 minutes at 480˚C. Different test portion sizes 100-500 mg of samples were digested to control variance of inhomogeneity.

An improved recovery of zirconium in zircon in granite (G-3), rhyolite MRH), andesite (MGL-AND) and chromite in harzburgite (MUH-1) was obtained by increasing heating time (2h) at 480˚C and by keeping (1:6) ratio of sample:Na_2O_2. Through this work it has been established that due to presence of zircon and chromite, decomposition time and sample:Na_2O_2 ratio has to be increased for an accurate content determination and complete release of analytes for geochronological studies. Larger test portion size reduces the heterogeneity issues in granites in particular [2]. No significant blanks issues were observed and interferences were controlled using QQQ MS mode of ICP-MS.

References

[1] Meisel, T., N. Schöner, et al. (2002). "Determination of Rare Earth Elements, Y, Th, Zr, Hf, Nb and Ta in Geological Reference Materials G-2, G-3, SCo-1 and WGB-1 by Sodium Peroxide Sintering and Inductively Coupled Plasma-Mass Spectrometry." Geostandards Newsletter 26(1): 53-61.

[2] Bokhari SNH., Meisel T (2013) "The Determination of Homogeneity of Geological Reference Material" Mineralogical Magazine, 77(5): 731.