

Quantifying more than half the periodic table of elements in uranium ore concentrates: Results of the IAEA's interlaboratory comparison 2011-2012, and two new quality control materials

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The analysis of trace elements (impurities) in uranium ores and uranium ore concentrates (UOC), as well as uranium-bearing materials in general, is performed in a variety of fields, e.g., in geochemical research, for quality control in the production stream converting uranium ores to nuclear fuel, as material signatures in nuclear forensics, or in international nuclear safeguards to support the verification of States' declarations.

An interlaboratory comparison on the quantification of impurities in UOC was organized by the International Atomic Energy Agency (IAEA) during 2011-2012. The goal was to probe the analytical capabilities of the participating laboratories (i.e., the current state-of-the-practice), to determine any differences between laboratories and their potential causes, to identify chemical elements that are problematic and/or difficult to analyse with established analytical techniques, and to assess the measurement uncertainties. Six participating laboratories were tasked to quantify 69 impurity elements – more than half the periodic table – in two different UOC materials. The impurity concentrations, normalized to uranium content, covered more than six orders of magnitude. Inductively-coupled plasma mass spectrometry (ICPMS) was the main technique used by the participating laboratories.

A summary of the comparative results will be presented, highlighting the current state-of-the-practice in ICPMS analytical approaches as employed by the participants. Lessons learned, including the identification of polyatomic interferences and the challenge of estimating measurement uncertainties, will be discussed. This interlaboratory comparison yielded consensus values for the two UOC materials. They are suitable for future use as quality control materials addressing gaps in the availability of reference materials for impurity elements in uranium-bearing materials.

C, N₂, Ar, He in fluid inclusions in a garnet lherzolite from Oasis Jetty, East Antarctica

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To reveal the sources and evolution of fluids during mantle metasomatism under East Antarctic we have carried out optical, thermobarogeochemical and Raman-spectroscopy investigations and studied C, N, Ar and He isotopic and elemental ratios in Px separate from Oasis Jetty garnet lherzolite by stepwise crushing method. The sample contains abundant fluid inclusions of different generations and compositions. The applied methods have revealed two major fluid activity stages. The early stage fluid inclusions are characterized by the most complex composition and high pressures (>13 kbar), some of them reach 10 μm in size. The late stage fluid inclusions (P ≥ 5-7 kbar) are all very small (2-5 μm and less); the predominance of CO₂ over all other gases is dramatically increasing in them, as well as the role of H₂O.

Our isotope-geochemical data confirm the existence of these two fluid sources. The high pressure and larger size fluid inclusions of the early stage mostly opened at the very first crushing steps are characterised by low C/N₂, C/Ar, N₂/Ar (31-34) ratios, typical for the MORB chilled glasses δ¹³C values in CO₂ (-4.7‰), heavy nitrogen (up to +2.05±0.28‰), and slightly elevated ⁴⁰Ar/³⁶Ar ratios (up to 533±10). The late stage fluid inclusions being progressively opened with increasing number of strokes are better characterized by the gases extracted at the very last crushing steps. They have 2-3 orders of magnitude higher C/N₂ and C/Ar ratios, close to the atmospheric N₂/Ar (86) and ⁴⁰Ar/³⁶Ar ratios (down to 370±7), lighter δ¹³C values in CO₂ (down to -6.8 ‰), and nitrogen (-2.28±0.26 ‰). The elemental and isotope ratios (N₂/⁴⁰Ar, ⁴⁰Ar/³⁶Ar, ⁴He/⁴⁰Ar*, δ¹⁵N) correlate to each other, reflecting mixing between early and late stage fluids. The latter is the result of a mixture between MORB-type mantle and atmospheric components. The early stage fluids could be the result of more complex mantle-crustal components mixture with high proportion of atmosphere derived argon. Typical for intra-plate mantle xenoliths element fractionation due to fluid-melt partitioning is observed in low ⁴He/⁴⁰Ar* ratios (0.1-0.7, compared to the mantle production ratio of 1.5-2). Some isotope fractionation (for N₂ and C) also could not be excluded.