(O-43)

Pseudo-bulk elemental analysis of pegmatite lithium ores by LA-ICP-MS

<u>Andrew M. Zipkin</u> and Gideon Bartov

Eurofins EAG Laboratories, Liverpool, New York, USA

The lithium-ion battery supply chain is built on lithium-rich brine and "hard rock" lithium ore used for lithium carbonate and hydroxide production. The most important minerals for hard rock lithium are spodumene and petalite, which may co-occur in lithium-bearing pegmatite. The new work presented here uses LA-ICP-MS for major and minor element analysis of lithium ores and seeks to overcome the disadvantages of techniques that require digestion or fusion sample preparation. Typically, elemental analysis of lithium ore is accomplished by one or more of the following: sodium peroxide fusion paired with ICP-OES and/or ICP-MS, 4-acid digestion paired with ICP-OES and/or ICP-MS, and borate fusion paired with XRF. These techniques yield a bulk measurement of the ore. The major weakness of such approaches is sample preparation. Fusion and acid digestion are time-consuming, may lead to the loss of volatile elements, introduce opportunities for contamination, generate hazardous waste, and are destructive to the sample. LA-ICP-MS on pelletized ore powder mitigates the weaknesses of existing approaches but introduces a new challenge: it is a microanalytical technique that may not yield results representative of the bulk ore.

LA-ICP-MS is reliant upon matrix-matched calibrants that are homogeneous at the microanalytical scale. Nanopowders of rocks, sediments, and hard tissue have become increasingly available as microanalytical reference materials over the last decade. Here, we applied LA-ICP-MS to five lithium ore and ore concentrate reference materials that are commercially available from myStandards GmbH as nanopowder pellets. Using an ESL imageGEO 193 laser system coupled to a Thermo Scientific iCAP TQ ICP-MS, we explored combinations of calibrants before determining that a single point calibration using OREAS 999 ore concentrate (2.67 % Li) yielded the best results and most efficient instrument operation when measuring OREAS 750 – 753 for method validation. In addition to Li, analytes included Na, Mg, Al, Si, P, K, Ca, Ti, Mn, and Fe. Each secondary reference material was ablated 25 times over five days for a pseudo-bulk analysis. For OREAS 750-752 all analytes exhibited overall mean recovery within 10% of reference values, while OREAS 753 showed recovery within 25% of reference values for Mg, Ca, and Ti and within 10% for the other elements. Notably, data reduction in Iolite v4 relied upon the Si reference values for internal standardization.

Lastly, we analyzed a non-nanopowder lithium ore reference material, NCS DC 86303, as a pressed pellet made in-house. The certificate for NCS DC 86303 states only that particle size is <200 mesh (<75 µm); microscopy found aberrantly large discrete particles with maximum dimensions up to 400 µm. This non-microanalytical reference material was analyzed as a proxy for an unknown milled ore sample. Mean Si concentration for LA-ICP-MS internal standardization was determined by SEM-EDS at three locations on the pressed pellet since for an unknown sample a reference value would be unavailable. Based on ten replicate ablations, again calibrated with OREAS 999, mean recovery was within 25% of reference values for all analytes except Na and K, which yielded 133% and 137% recovery, respectively. With further development, matrix-matched, empirically calibrated LA-ICP-MS will be a promising alternative to existing approaches to lithium ore elemental analysis.