Methodology for solute characterization of fluid inclusions by petrographic and SEM/EDS complementary analysis

F. Tweedale[1], J.J. Hanley[1], D.J. Kontak[2] and N. Rogers[3]

1. Department of Geology, Saint Mary's University, Halifax, Nova Scotia
2. Department of Earth Sciences, Laurentian University, Sudbury, Ontario
3. Geological Survey of Canada, 601 Booth Street, Ottawa, Ontario

Abstract: A cost-effective, simple, and time-efficient method to determine the bulk composition of fluid inclusions is evaporate mound analysis. This method is semi-quantitative and determines inclusion composition by integrating SEM imaging with energy-dispersive analysis of precipitates, or mounds, produced by thermal decrepitation of fluid-inclusions. The method is applicable to magmatic-hydrothermal systems where fluid inclusions contain solute ions (e.g., Na, K, Ca, Cl, F). In order to assess the application of this method for evaluating hydrothermal evolution and metal fertility with regards to intrusion-related mineralisation, a test study is being conducted on the large (7800 km^2) and variably mineralized (e.g., Sn, W, Cu, U, Mo, Ta) South Mountain Batholith (SMB) of Nova Scotia.

Decrepitate mounds were analyzed using a LEO 1450VP (SEM) imaging system linked to an Oxford X-Max 80 mm^2 SDD detector energy-dispersive detector. Based on decrepitating over a range of temperatures, from 325°C to 500°C, it appears that T = 500°C is optimal to produce large, well-shaped, and readily identifiable mounds. To optimize analysis time and, hence, increase research efficiency while maintaining result accuracy, data were collected with 5, 10, and 30 second acquisition times. The number of analyses required to produce representative results was also tested by comparing the results for 4, 8, 16, 32, and 64 mound analyses for individual samples. Results indicate that optimal procedures require multiple (N = 12) point-mode analyses on individual decrepitate mounds to substantiate mound heterogeneity, and that in order to accurately reflect in-situ fractionation a single, 10 second raster-mode analysis is the best approach.

These optimal analytical protocols are being applied to a regional study of the SMB to determine their suitability as mineral fertility indicator and/or vector to ore mineralisation. This test case is the first of its kind conducted on a batholithic scale, with the resulting methodological protocols being readily exportable for the mineral fertility assessment of other regions. Data interpretation protocols integrate a granitic petrographic alteration index, fluid inclusion types, density and evaporate mound chemistry. Samples are chosen such that the entire batholith may be assessed, with all mapped lithologies represented. Fluid compositions determined thus far include brines with 5-20 % fluorine, which is quantitatively indeterminable using other methods, and has been linked as primary control on the transport and deposition of ore in porphyry- and greisen-style mineralisation.

Originally presented Geology Matters - Exploration and Mining Expo Nova Scotia, Halifax, NS, November 12-13, 2014.

Corresponding author: Fergus Tweedale (fergus.tweedale@smu.ca)

Tweedale, F., Hanley, J.J., Kontak, D.J., and Rogers, N., 2015. Methodology for solute characterization of fluid inclusions by petrographic and SEM/EDS complementary analysis; *in* TGI 4 – Intrusion Related Mineralisation Project: New Vectors to Buried Porphyry-Style Mineralisation, (ed.) N. Rogers; Geological Survey of Canada, Open File 7843, p. 569-570.

Methodology for solute characterization of fluid inclusions by petrographic and SEM/EDS complementary analysis

Fergus Tweedale[*1], Jacob Hanley[1], Dan Kontak[2] and Neil Rogers[3]

[1]Saint Mary's University, Halifax (fergus.tweedale@smu.ca); [2]Laurentian University, Sudbury; [3]Geological Survey of Canada, Ottawa

INTRODUCTION

Hydrothermal-magmatic mineral deposits are an important subset of all mineral deposits, and given the nature of these deposits, it is expected that a fingerprint of the mineralizing fluids may be manifested both petrographically and by the chemistry of aqueous fluid inclusions hosted in the rocks on a scale equal to or larger than the mineralized centres. The combination of petrography and SEM/EDS evaporate mound analysis is an effective technique for characterizing fluid inclusion types, and for quantitatively assessing fluid-inclusion solute compositions. The methodology described herein is applied to a granitoid sample suite (N = 66) that is representative of a large (~7500 Km^2), variably mineralized batholith. The integration of results from qualitative petrography and SEM/EDS chemical analysis provides a rigorous assessment of fluid - rock interaction that could help guide future localized research in the South Mountain Batholith. However, the robust nature of the technique makes possible its application to any geological setting in which fluid inclusions occur. Beyond the practical benefits of cost efficiency and of a relatively simple methodology, the technique described in this paper also features particular analytical advantages over other, more costly and time-consuming techniques that are commonly applied to fluid-inclusion research.

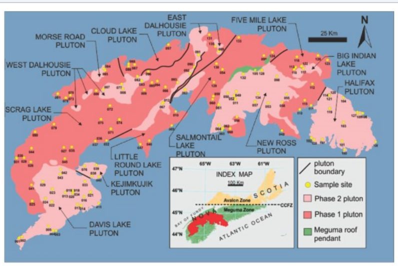

Fig. 1. Geological map of study area. The Devonian South Mountain Batholith of Nova Scotia is a mineralized granitoid complex composed of two contiguous phases of plutons. An objective of this research to evaluate the chemical composition of FIs across the entire SMB. (Map boundaries: NSDNR)

METHODOLOGY

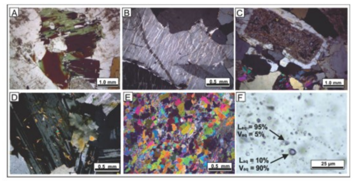

Fig. 2. Photomicrographs of polished thin sections illustrating degree of fluid:rock interaction in the South Mountain Batholith. A. Well-developed chloritization of primary biotite grain. B. K-feldspar megacryst exhibiting flame perthite. C. Coarse-grained feldspar grain exhibiting sericitization of core. D. Sausssuritization of Ca-plagioclase. E. Intergrowth of white mica. F. Fluid-inclusion assemblages hosted in annealed quartz

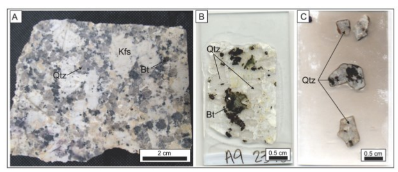

Fig. 3. Preparation of samples for SEM/EDS analysis. A. A slab sample for one hundred 10 x 10 Km2 grid squares covering the study area is collected. B. Fluid inclusion wafers are prepared for petrographic evaluation and identifying of targeted fluid inclusions. C. Chips containing abundant fluid inclusions, hosted by quartz, are cut from the wafer and immediately carbon coated (10 – 20 μm veneer).

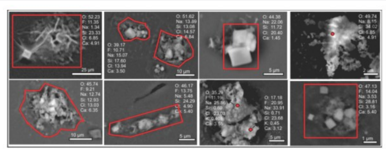

Fig. 4. Evaporate mounds. During sample heating, fluid inclusion decrepitate, or burst, and solute species precipitate within the vicinity of evacuated fluid inclusion pits. Evaporate mounds are amenable to EDS analysis, from which the chemical composition of the fluid inclusions is determined. Red boxes bound areas of raster mode analyses and red circles indicate location of point mode analyses.

RESULTS

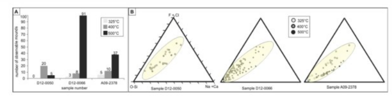

Fig. 5. Optimizing decrepitation temperature. A. Histogram showing number of mounds generated vs oven temperature. B. Composition of evaporate mounds.

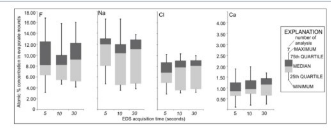

Fig. 6. Optimizing run time. Plots of mound analysis vs EDS acquisition time.

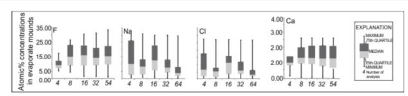

Fig. 7. Optimizing sampling. Plots of mound composition vs number of analysis.

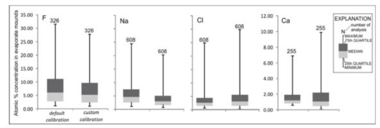

Fig. 8. Calibration of instrument to improve analytical precision.

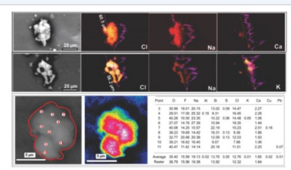

Fig.9. Substantiating mound heterogeneity (fractionation during decrepitation).

DISCUSSION

The results produced from this study suggest two points worthy of discussion: 1) the presence of Ca-bearing fluid, 2) enrichment of fluorine in F-Na-Ca FI populations Fig. 10).

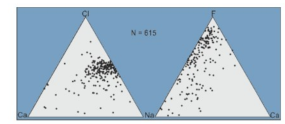

Fig. 10. Ternary plots of major solute compositions of SMB fluid inclusions.

The unexpected enrichment of F is likely the first recognition of this phenomenon in granitic bodies on such a scale anywhere. Enrichment of F suggests the generation of an F-rich fluid is part of the natural evolution of the system and consistent with the presence of topaz in the most evolved magmatic rocks and as part of mineralized greisens. In addition, that F enrichment occurs in the same fluid with Ca indicates a possible cause and effect relationship which we suggest relates to the reactive capacity of the original exsolved Na-F fluid.

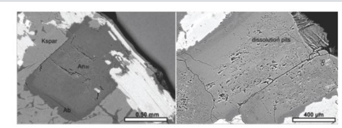

Fig. 11. BSE image images of a SMB monzogranite. A) Plagioclase (An30) grain mantled by albite and surrounded by perthitic K-feldspar. B) Close-up of the plagioclase core showing pitted area surrounded by the fresh plagioclase

The Ca-bearing fluid may reflect the petrographic observation of extensive albitization of plagioclase, which occurs in all samples. This alteration process could explain the distinctive pitted texture in the altered feldspar and the liberation of calcium to the fluid. The process is attributed to the reaction of an orthomagmatic fluid with already crystallized granite.

Natural Resources Canada   Ressources naturelles Canada   Canada