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Mines

DATABASE OF FLUORITE MINERALIZATION AND OF THE HOST ROCKS IN THE ST. LAWRENCE AREA, BURIN PENINSULA (NTS MAP AREAS 01L/13, 14 AND 01M/03)

Z. Magyarosi, N. Pochereva and G. Layne

Open File NFLD/3381

St. John's, Newfoundland
November, 2019

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SUMMARY

This Open File release contains data on 211 samples from the St. Lawrence area, Burin Peninsula (NTS map areas 01L/13, 14 and 01M/03). The database includes whole-rock geochemical analyses, rock-slab photos, BSE images, X-ray maps, modal mineralogy of four samples, Pb isotope data from whole rocks and galena, and trace- and rare-earth element (TE-REE) data on fluorite. The purpose of this study was to investigate the newly discovered AGS fluorite deposit and associated rocks. Additional publications will be released, as they become available, summarizing the results of this and related studies.

NOTES ON THE DATABASE

This open-file report consists of a database of 211 samples collected in the St. Lawrence area (NTS map areas 01L/13 (Lamaline), 01L/14 (St. Lawrence) and 01M/03 (Marystown); Figure 1). Sampling focused on the AGS fluorite deposit, several granite-hosted fluorite veins, the St. Lawrence Granite and Inlet Group sedimentary rocks hosting fluorite mineralization. The data collected include whole-rock geochemical analyses (Appendix B); photos of cut rock-slabs (Appendix E); BSE images, X-ray maps, and modal mineralogy of four samples (Appendix F); Pb isotope data from whole rock (Appendix G), and galena hosted in fluorite (Appendix H); and TE-REE data on the variable phases of fluorite mineralization (Appendix I). Analyses of standards and duplicates in the geochemical data are included in appendices C and D, respectively. The data are in comma-separated value (csv) format and are available from the Natural Resources website. Table 1 comprises a list of abbreviations used in this open-file data report.

Whole-rock geochemical analyses were completed at the geochemical laboratory of the Geological Survey of Newfoundland and Labrador (GSNL) in St. John's. The data include the location of the samples in UTM coordinates in NAD27 (Zone 21), NTS map areas, the name or symbol of the sampled formation and/or group, a brief description (lithology and grain size) and whole-rock geochemical analyses (Appendix B). Table 2 consists of a list of analytical methods used for each element. Most of the major elements were analyzed with ICP-OES following borate fusion. FeO was measured by the titration method and LOI by the gravimetric method. Most of the trace elements were analyzed using ICP-MS following borate fusion, and the rest using ICP-OES following four-acid digestion. Silver was analyzed using ICP-OES following nitric acid digestion. Fluoride was analyzed using ion-specific electrode (ISE). The analytical procedures are given in Finch *et al.* (2018). Major elements are reported in wt. % and

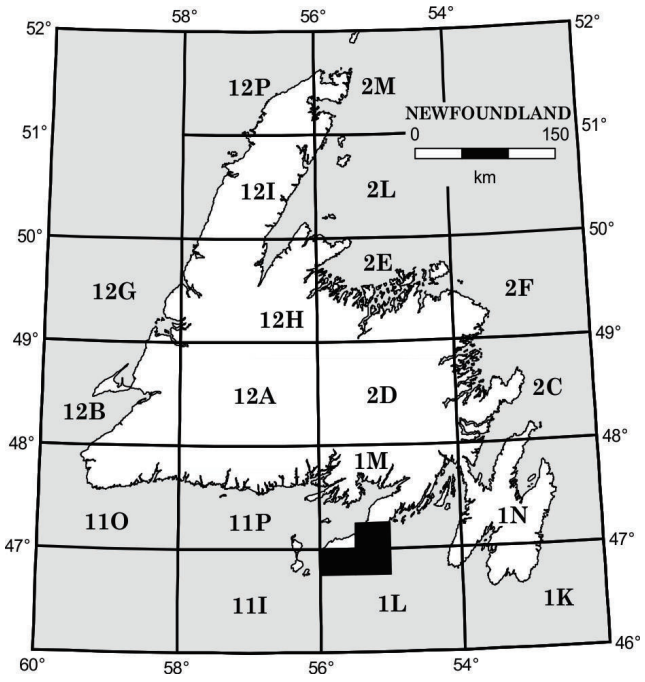

Figure 1. *Location map of the study areas.*

Table 1. List of abbreviations

Abbreviation	Explanation
-99	Sample not analyzed for that element
($^{208}Pb/^{204}Pb$)c	Calculated ratio of Pb isotopes
($^{208}Pb/^{204}Pb$)m	Measured ratio of Pb isotopes
A	Angstrom
BSE	Back-scattered electron
EDX	Energy dispersive x-ray
Fe_2O_3T	Total measured iron
Grav.	Gravimetric
ICP-MS FUS	Inductively Coupled Plasma Mass Spectrometry following lithium metaborate/tetraborate fusion
ICP-OES-4 Acid	Inductively Coupled Plasma Optical Emission Spectrometry following HF-HCl-HNO_3-$HClO_4$ acid digestion
ICP-OES-FUS	Inductively Coupled Plasma Optical Emission Spectrometry following lithium metaborate/tetraborate fusion
ICP-OES-HNO_3	Inductively Coupled Plasma Optical Emission Spectrometry following nitric acid digestion
ISE	Ion-selective electrode
LOI	Loss-on-ignition
MLA	Mineral liberation analysis
n	Number of cycles of peak switching analysis used to calculate the results of the spot analysis
N	Normality of a solution expressed as number of equivalents per litre
N/A	Not available
negative detection limit	Below detection limit
P (%)	Poisson: "probability of a number of relatively rare events occurring in a fixed time if these events occur with a known average rate, and are independent of the time since the last event" (https://www.dur.ac.uk/physics/students/labs/skills/data/poisson/)
ppm	Parts per million
REE	Rare-earth elements
SD	Standard deviation
SE	Standard error of the mean
SEM	Scanning electron microscope
SIMS	Secondary ion mass spectrometry
SLG	St. Lawrence Granite
TE-REE	Trace and rare-earth elements
TIMS	Thermal ionization mass spectrometry
wt. %	Weight percent

Table 2. Analytical methods for the elements

Element	Analytical Method
SiO_2, Al_2O_3, Fe_2O_3T, MgO, CaO, Na_2O, K_2O, TiO_2, MnO, P_2O_5, Cr, Ba, Be, Sc, Zr	ICP-OES-FUS
Fe_2O_3	Calculation
FeO	Titration
LOI	Gravimetric
Ga, Ge, Sr, Y, Nb, Mo, Sn, Cs, La, Ce, Pr, Nd, Sm, Eu, Tb, Gd, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, W, Tl, Bi, Th, U	ICP-MS-FUS
As, Cd, Co, Cu, Li, Mo, Ni, Pb, Rb, S, V, Zn	ICP-OES-4 Acid
Ag	ICP-OES following nitric acid digestion
F	Alkaline fusion with ion-selective electrode

trace elements are reported in ppm. Negative detection limit values represent analyses below the detection limit and -99 represents samples that were not analyzed for that element. Fe_2O_3T is the total measured iron. Quality assurance and quality control (QA/QC) procedures and information on the standards are included in Finch *et al.* (2018). Information on standards not included in Finch *et al.* (2018) are summarized in Appendix A.

Photos of cut rock-slabs of samples from the fluorite veins are included in Appendix E. The appendix includes a table containing the location and a brief description of the samples.

BSE images, X-ray maps and modal mineralogy of four samples were completed using the SEM at Memorial University of Newfoundland Micro Analysis Facility (MUN MAF-IIC) (Appendix F). SEMs are used for imaging small specimens at high magnifications (up to 100 000 times) by collecting emitted electrons. The SEM at MUN is an FEI Quanta 400, capable of working at variable pressures, equipped with EDX and capable of x-ray aided image analysis (MLA), which allows the SEM to quantify automatically mineral abundances in a selected area (https://www.mun.ca/creait/).

Pb isotopes of the whole rocks were completed using TIMS at the MUN Terra Facility (Appendix G). The method of analysis is described in Pochereva (2019): "Approximately 0.2 g of powder is dissolved in Savilex© Teflon beakers using a mixture of HF–HNO_3 acids. After five days of digestion, the solution is evaporated to dryness and then taken up in 6 N HCl acid for two days. The solution is then evaporated again and taken up in HBr. Pb elution is achieved using the

standard anionic HBr–HCl chromatography. All reagents are purified by sub-boiling to insure low contamination levels. All isotopic ratios are obtained using a multi-collector Finnigan Mat 262 mass spectrometer in static mode. All reported Pb isotopic ratios are corrected for mass fractionation by an in-run factor (usually 0.1–0.2% per atomic mass unit) that is obtained by measuring the deviation from repeated analyses of the NBS 981 standard. In-run precisions on all isotopic ratios are given at 95% confidence level."

TE-REE analyses in fluorite and Pb isotope analyses in galena were completed at MUN using SIMS (appendices H and I). All analyzed galena are hosted in fluorite veins. TE-REE analysis in fluorite were designed to include most textures and generations of fluorite mineralization. Appendix I contains photos of the fluorite samples with the exact location of each TE-REE analytical point. Sample preparation and the analytical method are described in Pochereva (2019): "A 17mm x 17 mm area on each sample was selected for analysis. This area was cut into a 17mm x 17mm wafer using a diamond-embedded blade on a TECHCUT 5 programmable saw from ALLIED High Tech Products Inc. The wafers were then mounted in epoxy inside the one-inch aluminum rings, where they cured for 24 hours. The samples were subsequently polished down to 1 μm using a Tegramin-30 polisher by Struers. Polisher, sample holder as well as samples were ultrasonically cleaned between each increasingly fine polishing stage to prevent contamination by previous polishing grit. Samples were characterized using petrographic microscopy, then photographed and catalogued for reference before SIMS and SEM analysis. The Cameca IMS 4f SIMS instrument in the Memorial University MAF-IIC facility was used for Pb isotope analysis of galena, and for TE-REE analyses of fluorite and one point of calcite (17ZM212A01–1). The SIMS instrument generates a focused beam of ions within a vacuum chamber. The ion beam interacts with sample surface sputtering (removing) ions as well as neutral atoms from the sample material. The sputtered ions are accelerated, focused and then sent through the mass spectrometer to be analyzed. This micro-analytical approach allows for the precise detection of isotopic ratios and elemental concentrations. One of the strengths to using SIMS is sample preservation because the resulting material removed from sputtering is only about 3 cubic μm (https://serc.carleton.edu/msu_nanotech/methods/SIMS.html), with a lateral resolution for Pb isotope or trace-element microanalysis of better than 20 μm. Preceding SIMS analysis, a thin layer of gold (approx. 300 Å) was applied to the samples using a sputter coater. Once coated in gold, the galena samples were analyzed for Pb isotopes by using an O- primary ion beam, following the detailed procedures documented in Gill *et al.* (2019). The fluorite samples were analyzed for rare earth elements later. Each sample was analyzed using 1-6 spots, depending on size and quantity of desired targets, in order to account for zonal variation. Photos were taken of samples using a petrographic microscope/camera as well as with a SEM, to document/label each SIMS analysis location. Raw data from SIMS analysis of Pb isotopes was corrected for instrumental fractionation using galena reference material JMBH, which yields an external precision of +/- 0.10-0.15% (Gill *et al.*, 2019)."

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Appendix A
Additional Standard Information

List of abbreviations

Abbreviation	Explanation
Rec Val	"Recommended" or, if asterisked, "information" value for that element, in that certified reference material (CRM), normally derived as the arithmetic mean of multiple analyses (after the removal of outliers), at several laboratories, by the supplier of the CRM.
A. Mean	Arithmetic mean (after the removal of outliers) of a variable number of analyses (*see* "Count", below) at the GSNL laboratory.
Std. Dev.	Standard deviation of the same analyses used to calculate the mean, above.
UCL	Upper control limit. Calculated as the mean plus two standard deviations, as calculated above.
LCL	Lower control limit. Calculated as the mean minus two standard deviations. Analyses of the CRM that fall between the UCL and the LCL, and the batch of samples into which it was inserted, are generally considered acceptable.
Count	The number of analyses used to calculate the arithmetic mean, standard deviation, UCL and LCL.

CH-2

Element Units	Ag ppm
Rec Val	24.2
A. Mean	10.1
Std. Dev.	2.2
UCL	14.5
LCL	5.8
Count	239

SU-1

Element Units	Ag ppm
Rec Val	4.3
A. Mean	2.7
Std. Dev.	0.4
UCL	3.5
LCL	1.9
Count	235

SY-3

Element Units	F ppm
Rec Val	6960
A. Mean	6460
Std. Dev.	565
UCL	7589
LCL	5331

MA-N

Element Units	F ppm
Rec Val	17000
A. Mean	13051
Std. Dev.	1126
UCL	15303
LCL	10800

USGS XR3

Element Units	F ppm
Rec Val	86200
A. Mean	78136
Std. Dev.	4722
UCL	87581
LCL	68691

APPENDICES B–I

Appendices B–I are available as digital comma-separated files (.csv) or zip files (for images) through this link.

Appendix B: Sample locations, description and whole rock major- and trace-element geochemistry
Appendix C: Standard analyses
Appendix D: Duplicate analyses
Appendix E: Rock slabs
- csv file - sample location and description
- zip file - 2017-2018 images
- zip file - AGS images

Appendix F: BSE images and x-ray maps of four samples
- csv file - sample modal mineralogy
- zip file - images

Appendix G: Whole rock lead isotope analyses
Appendix H: Lead isotope analyses of galena
Appendix I: Trace and rare-earth elements (TE-REE) analyses of fluorite
- csv file - data
- zip file - images