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Geological Survey of Canada
Scientific Presentation 152

Comparative pXRF and lab ICP-ES/MS methods for mineral resource assessment, Northwest Territories

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Canada

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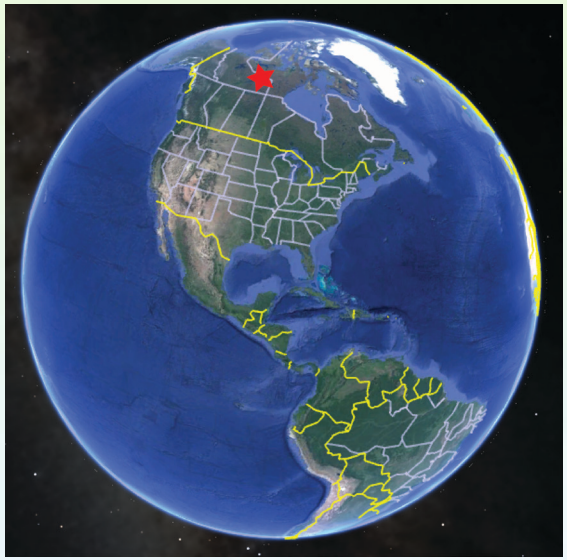
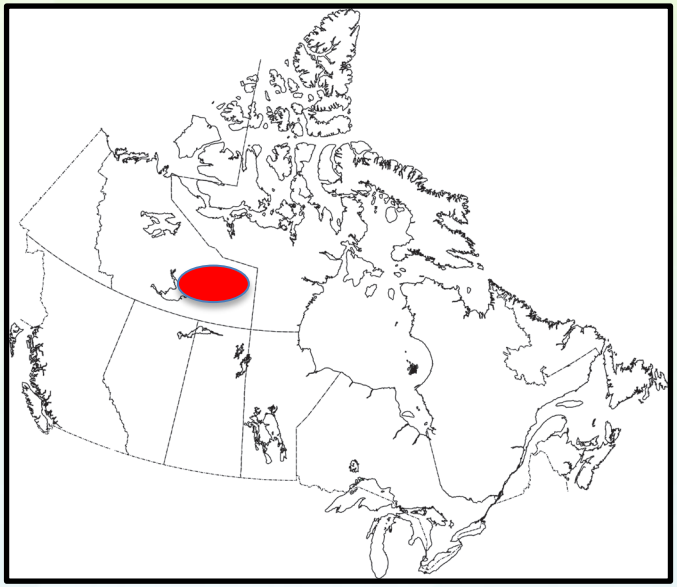
R.D. Knight and B.A. Kjarsgaard
Geological Survey of Canada
October 2022





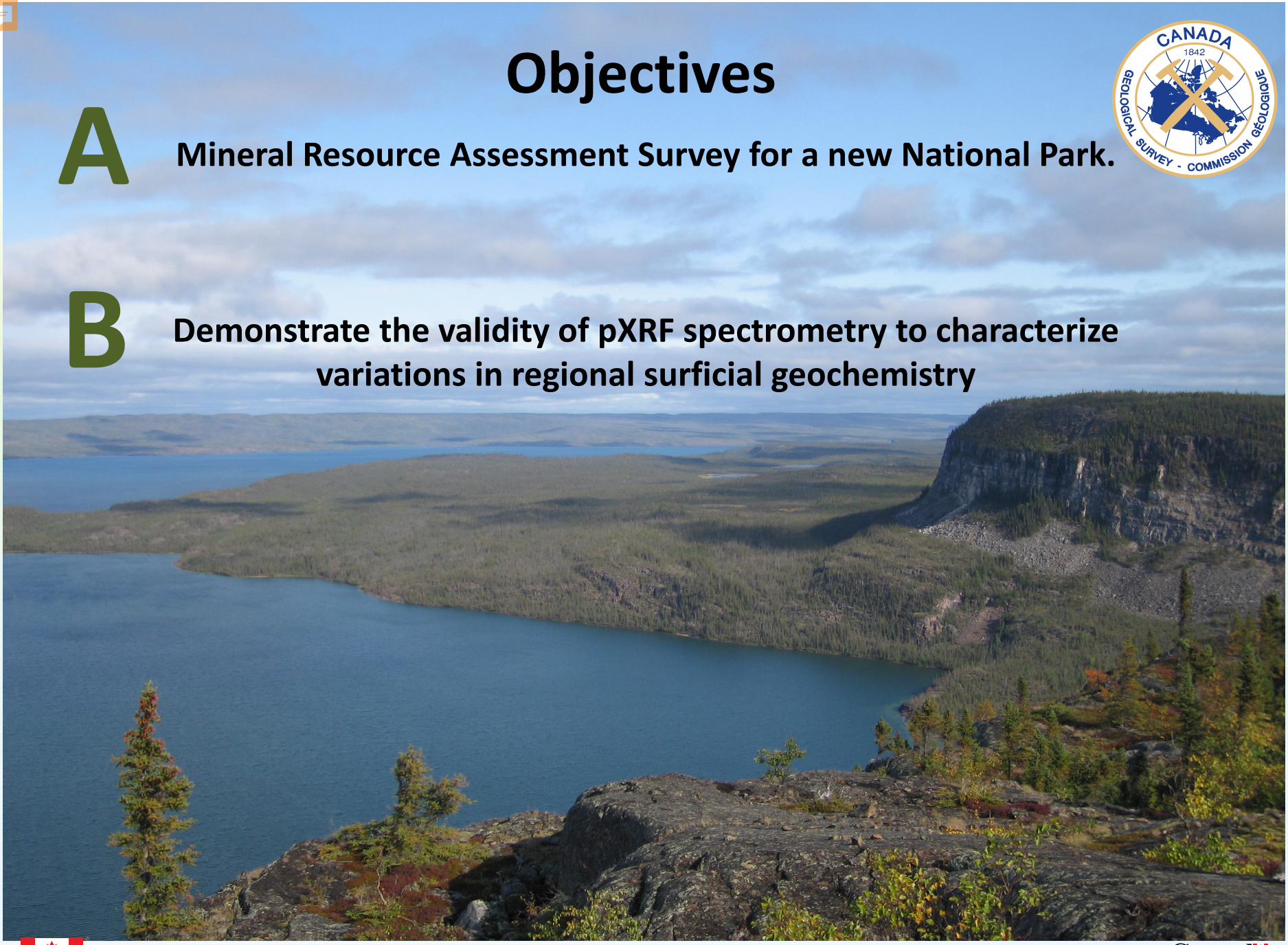
29th IAGS2022
Viña del Mar, Chile

CANADA 1842 GEOLOGICAL SURVEY - COMMISSION GÉOLOGIQUE

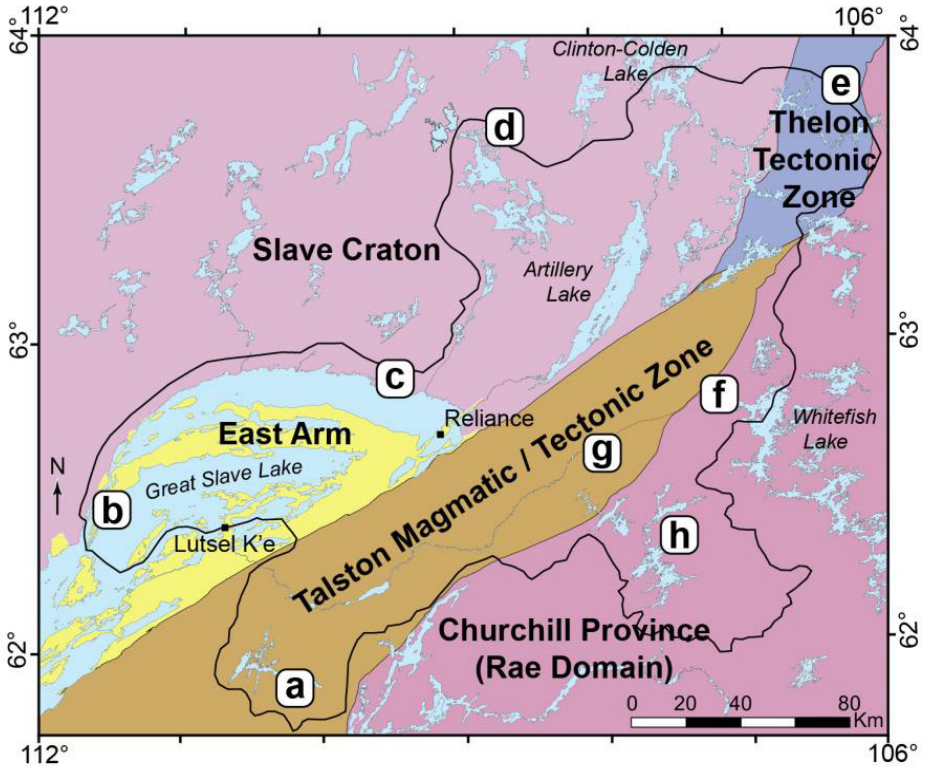
Objectives

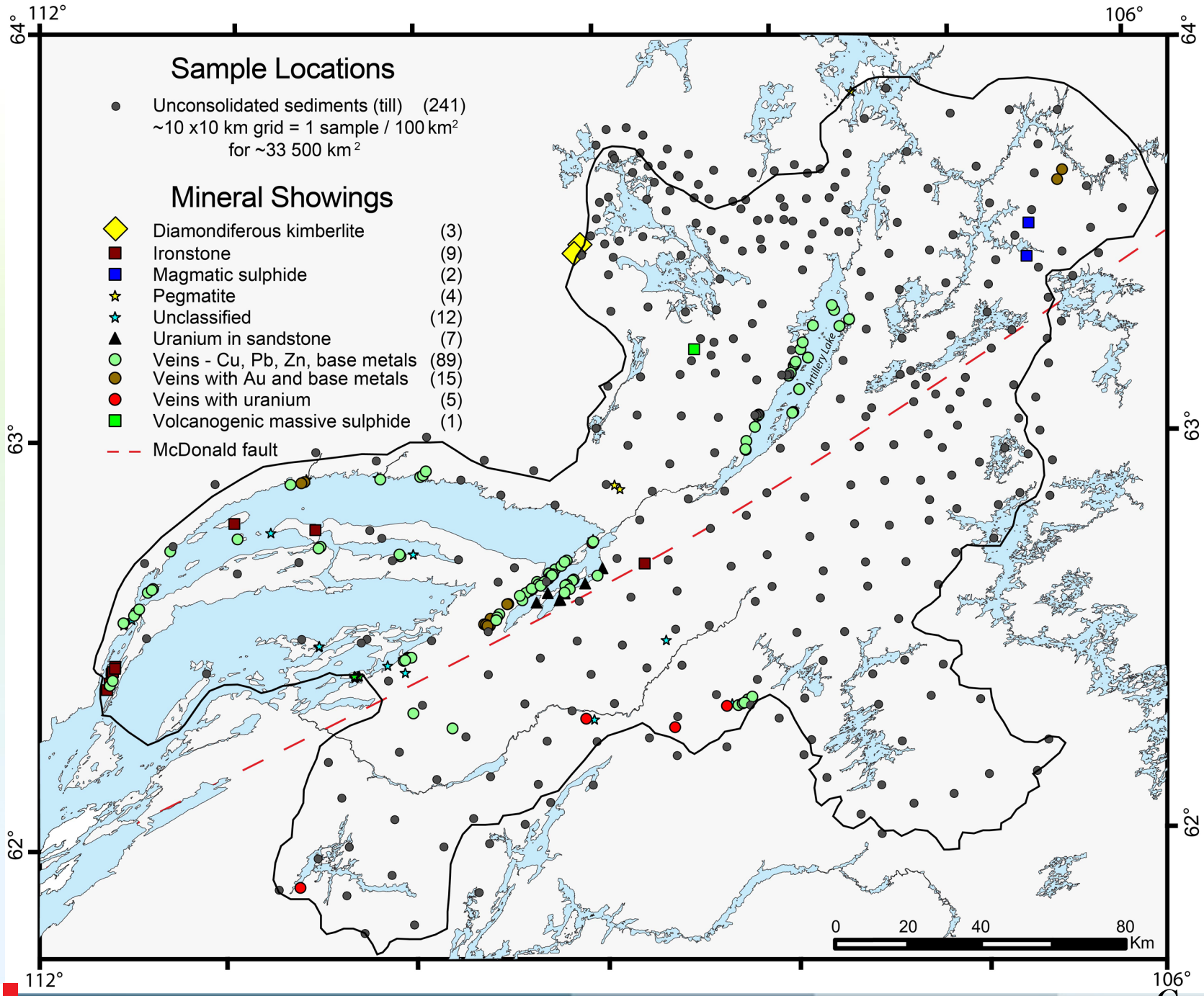
A Mineral Resource Assessment Survey for a new National Park.

B Demonstrate the validity of pXRF spectrometry to characterize variations in regional surficial geochemistry



Research Area:
northern boreal forest to tundra transition




Sample Locations
Unconsolidated sediments (till) (241)
~10 x10 km grid = 1 sample / 100 km²
for ~33 500 km²
Mineral Showings
Diamondiferous kimberlite (3)
Ironstone (9)
Magmatic sulphide (2)
Pegmatite (4)
Unclassified (12)
Uranium in sandstone (7)
Veins - Cu, Pb, Zn, base metals (89)
Veins with Au and base metals (15)
Veins with uranium (5)
Volcanogenic massive sulphide (1)
McDonald fault
Artillery Lake
112°
106°
64°
63°
62°
0 20 40 80
Km

Methods

A Analyze samples using comprehensive, multiple laboratory ICP-ES/MS methods for <2 mm and **<0.063 mm** size fractions:

1) **aqua – regia,**
2) **multi – acid digestion,**
3) **lithium metaborate/tetraborate fusion**

B Analyze samples using pXRF spectrometry in both Mining and **Soil modes**:

1) original un-processed sample,
2) **partially processed using shaking/vibrating method**,
3) processed <2 mm size fraction,
4) processed <0.063 mm size fraction

Analytical Protocols - laboratory chemistry

1) 1kg sample sent to Bureau Veritas (ACME) - Vancouver Canada
2) Sample split to 500 g (500 g original shipped to GSC)
3) Dried at 60^{0}C, disaggregated, sieved to < 2mm; <0.063 mm

Aqua regia

Dissolution in nitric and hydrochloric acid (HNO_3-HCl @ 95°C), followed by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) for 64 elements

Multi - acid

Dissolution in nitric, perchloric, and hydrofluoric acid (HNO_3-$HClO_4$-HF), taken to dryness and then the residue dissolved in hydrochloric acid (HCl), followed by Inductively Coupled Plasma Emission Spectroscopy ICP-ES (major elements) and ICP-MS (trace elements) for 54 elements

Fusion

Lithium metaborate/tetraborate fusion disc followed by dilute nitric acid (HNO_3) digestion of the fused disc, and analysis by Inductively Coupled Plasma Emission Spectroscopy ICP-ES (major elements) and ICP-MS (trace elements) for 51 elements

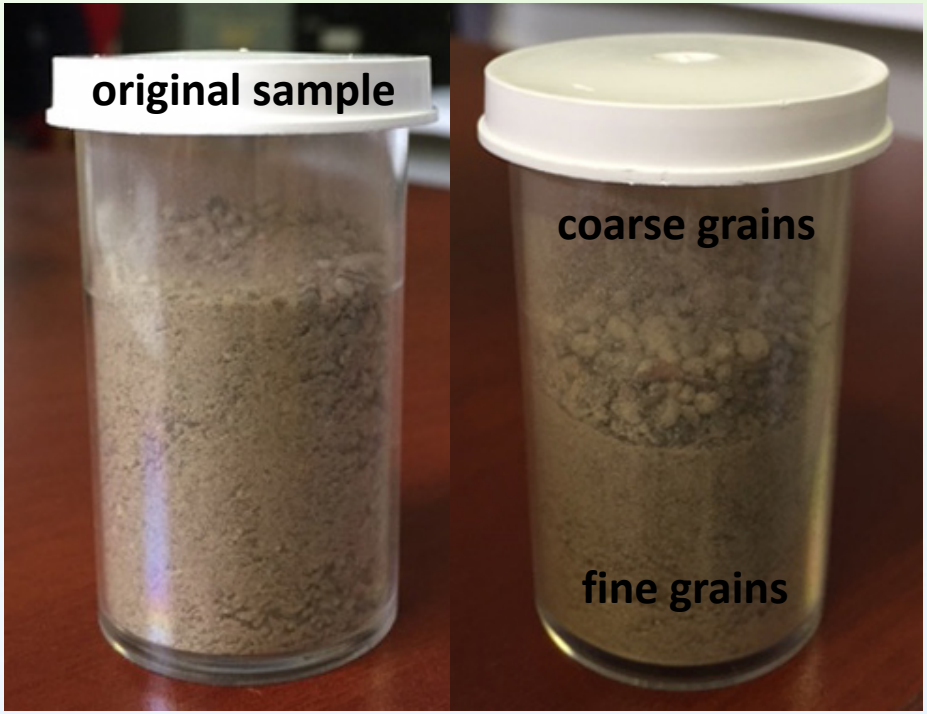
Analytical Protocols - pXRF

1) Original unprocessed sample
2) Dried, sieved <2 mm
3) **Dried and sample shaken/vibrated in vial** (new technique)
4) Dried, sieved <0.063 mm

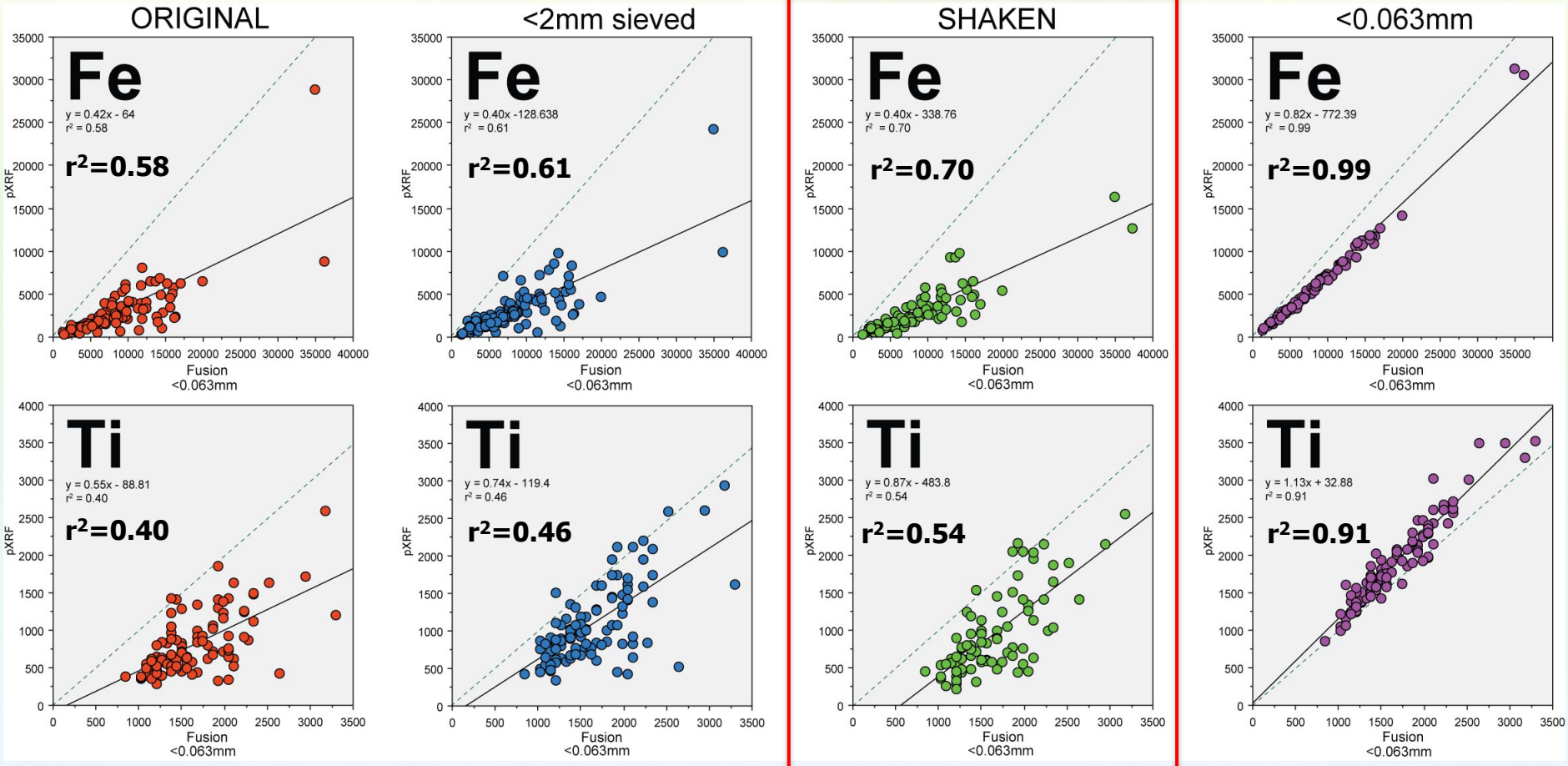
Shaking Method "Granular Convection"

1) Dry sample
2) 100 ml vial
3) Invert sample and vibrate
4) Invert, cover, 6 micron film
5) Analyze in test stand
6) **Soil mode 60 sec/filter**
7) *Mining mode 45 sec/filter*

Pebbles recovered from 2mm sieve

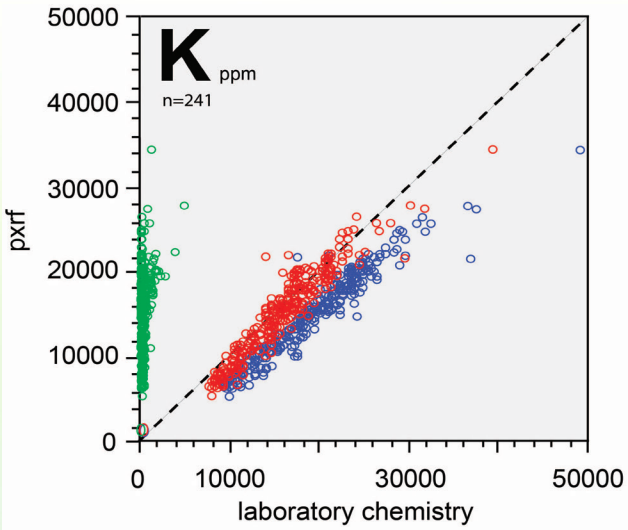


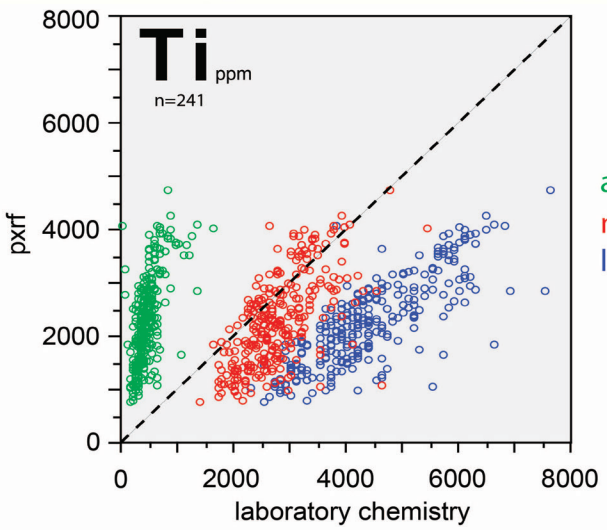
Comparison of Fusion chemistry with pXRF(Soil mode): un-processed, <2 mm, “shaken”, <0.063 mm

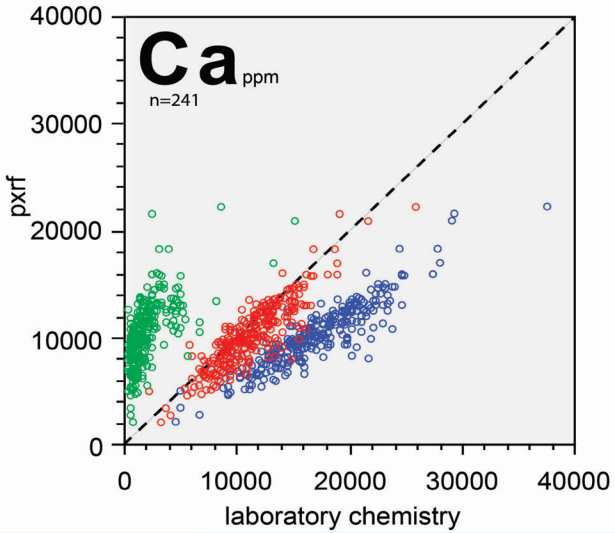


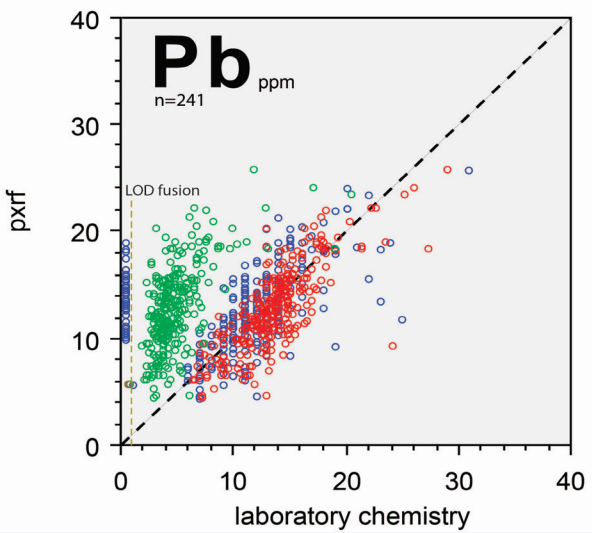
Increasing r^2

Comparison: laboratory and pXRF (<0.063 mm)









aqua regia (partial digestion)
multi acid (near-total digestion)
lithium borate fusion (total digestion)

pXRF is a 'total' analysis and should be compared to near total or total lab analyses

Correlation between pXRF and aqua regia (partial digestion) is typically poor

Data Presentation

A

Scattergram comparison:
multi-acid or fusion (<0.063 mm)
versus pXRF ('shaken')

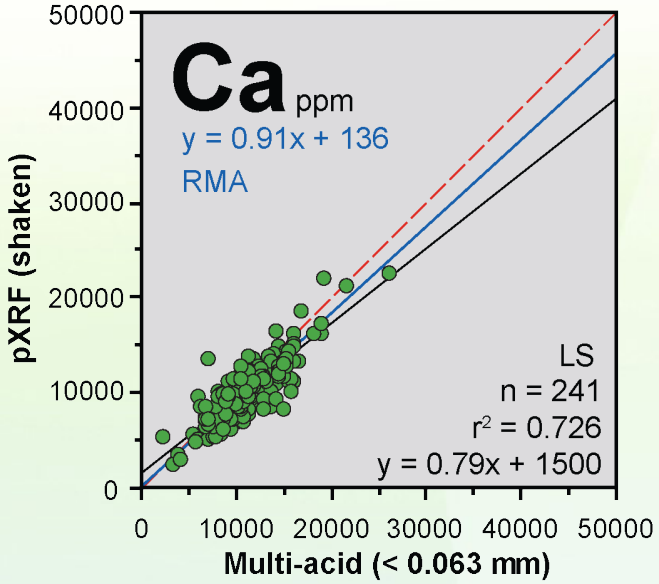
B

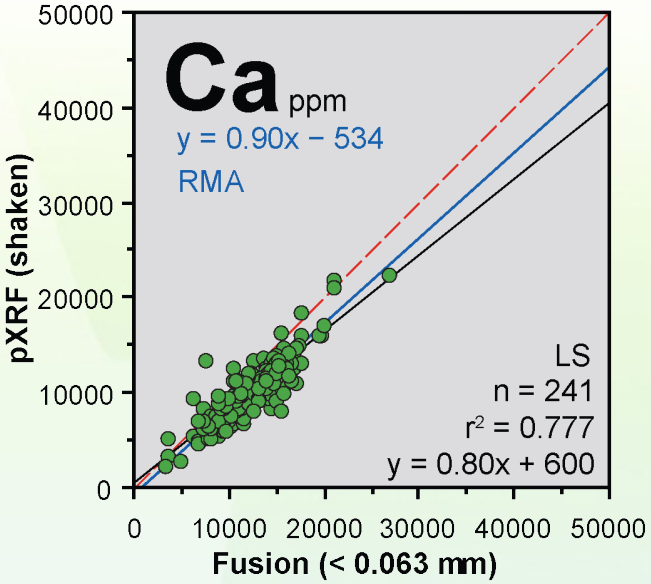
Natural Neighbor Interpolated Maps

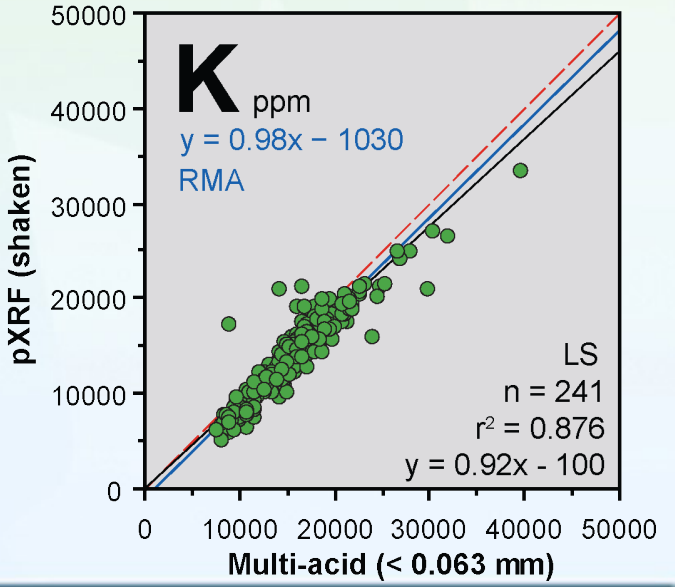
MapInfo – Vertical Mapper
Cell Size – 100 m
Aggregation distance 500 m
Fixed colour profile, 0, 50th, 95th, 98th, and 100th percentiles

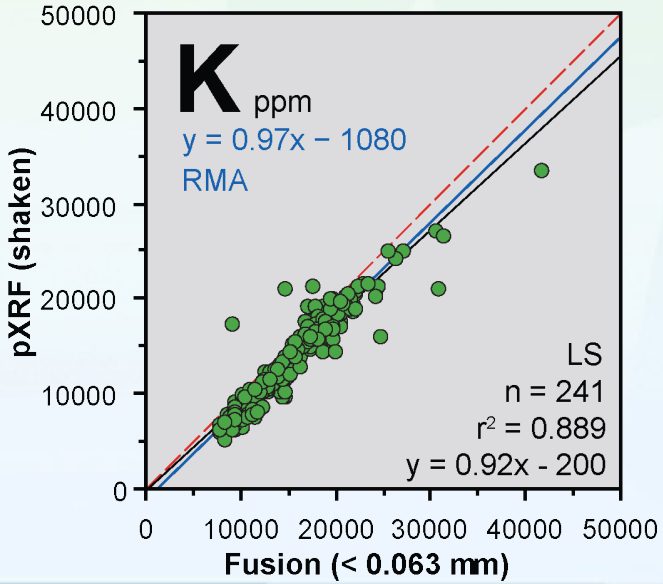
Note: maps have a relative concentration scale, i.e., the range of concentration level differs for each map, depending on the analytical method and detection limit.

Comparison multi-acid and fusion (<0.063 mm) with pXRF ('shaken')



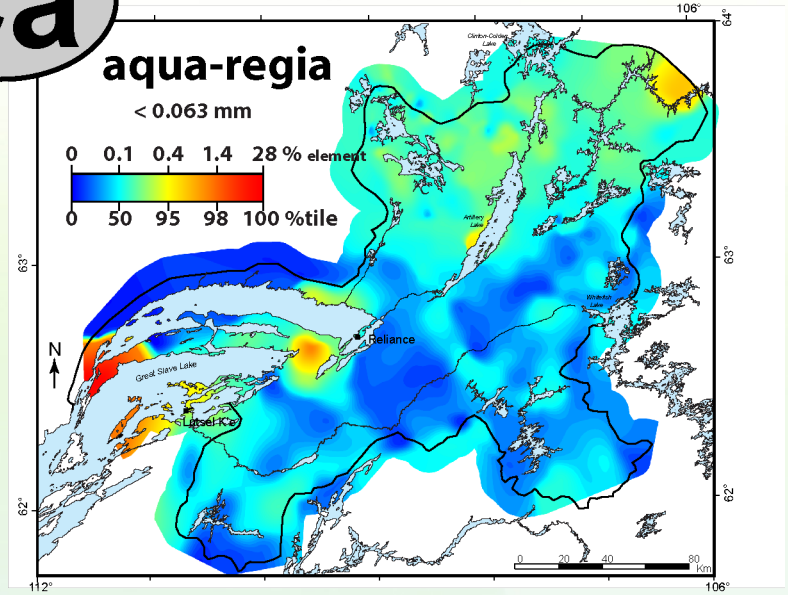


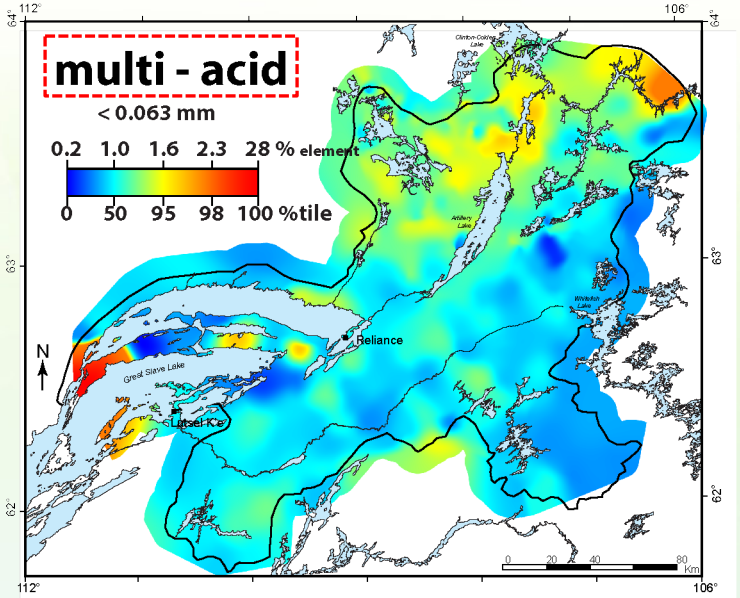


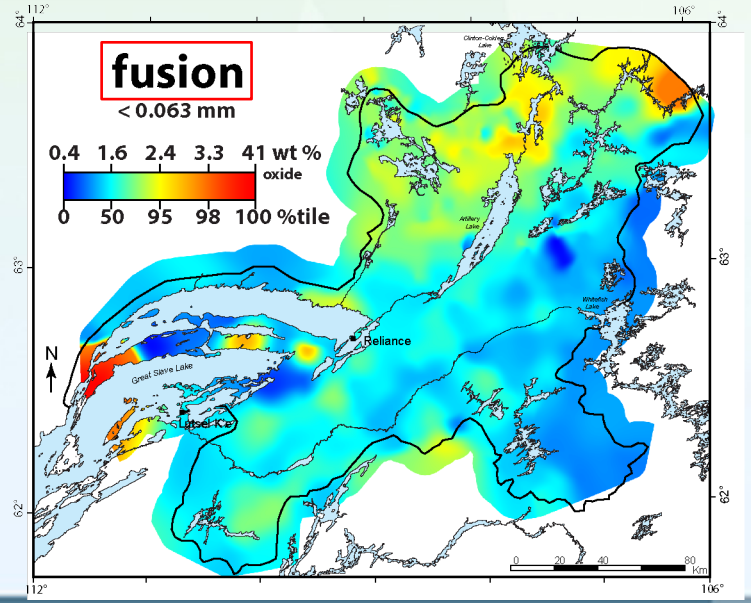


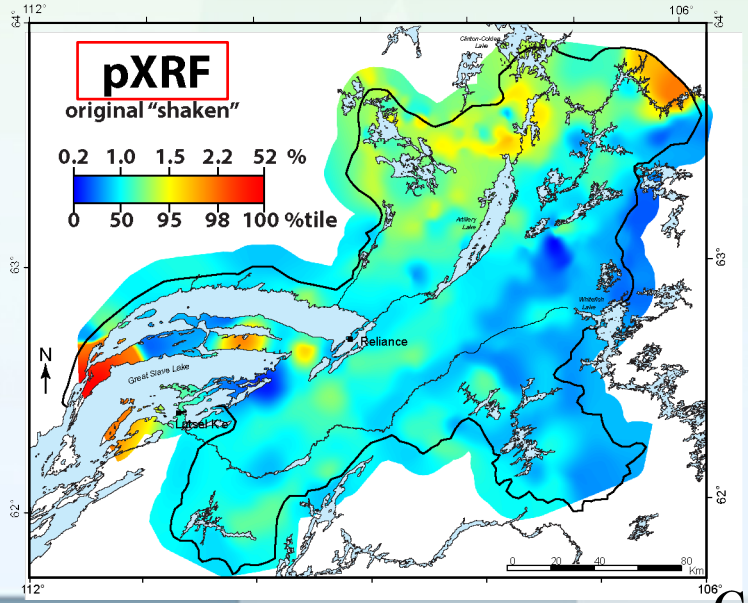
Comparison of Ca by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

Ca



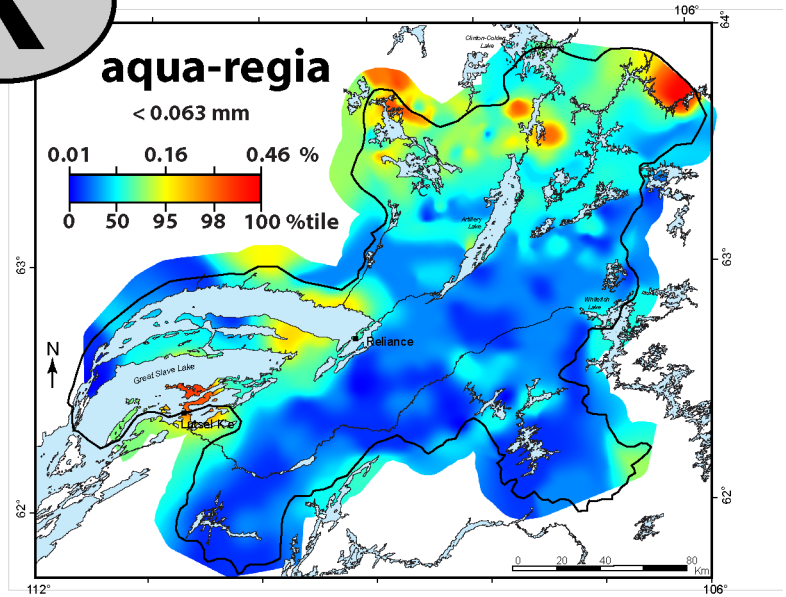


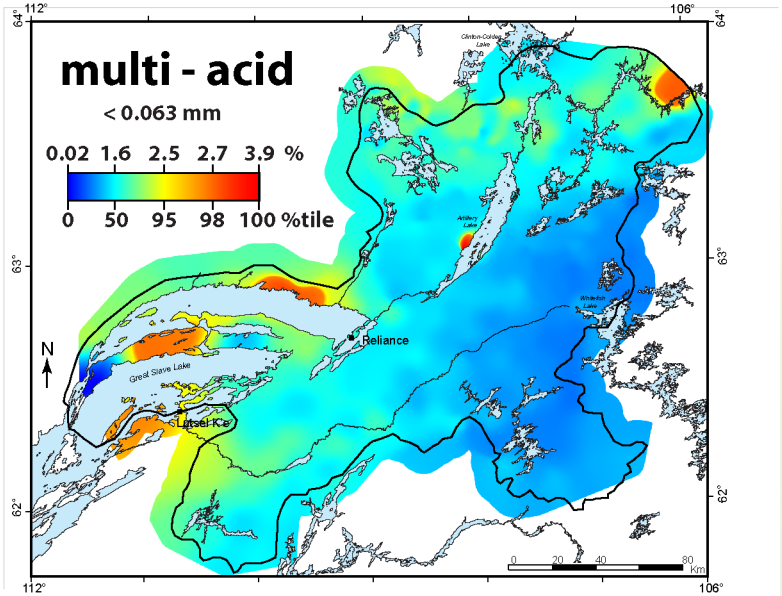


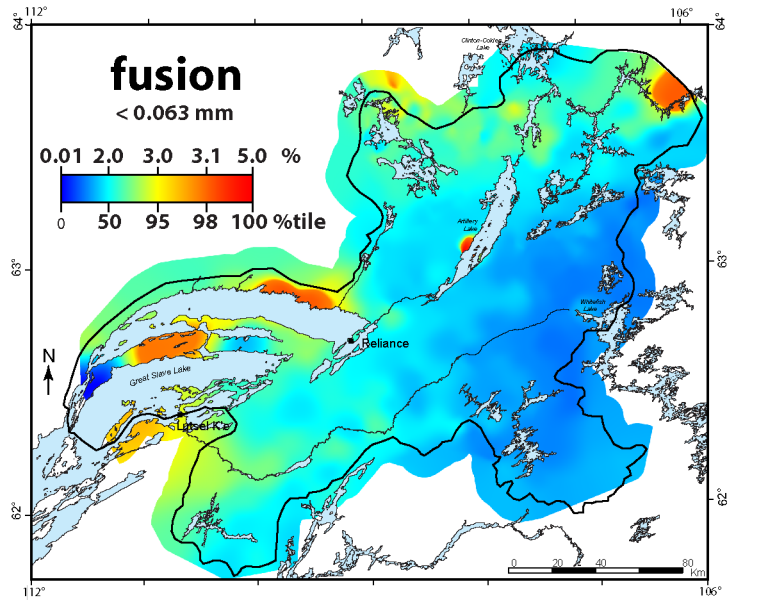


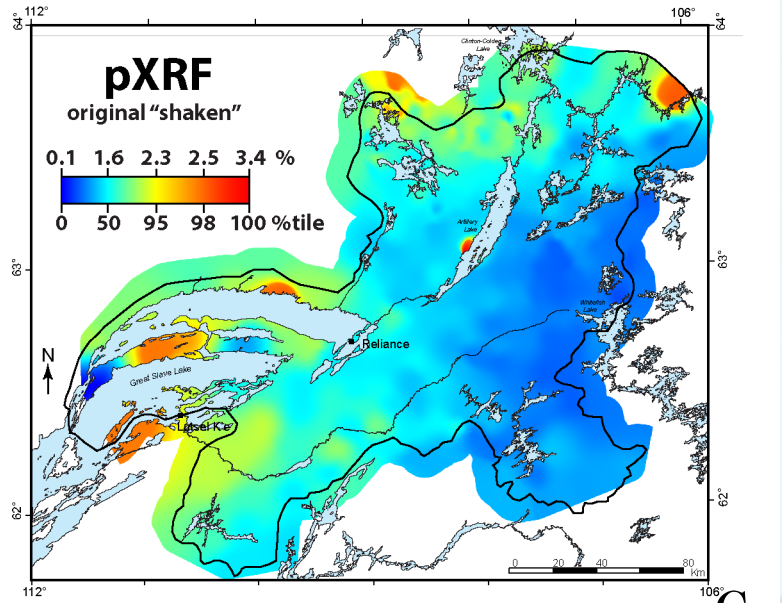
Comparison of K by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

K



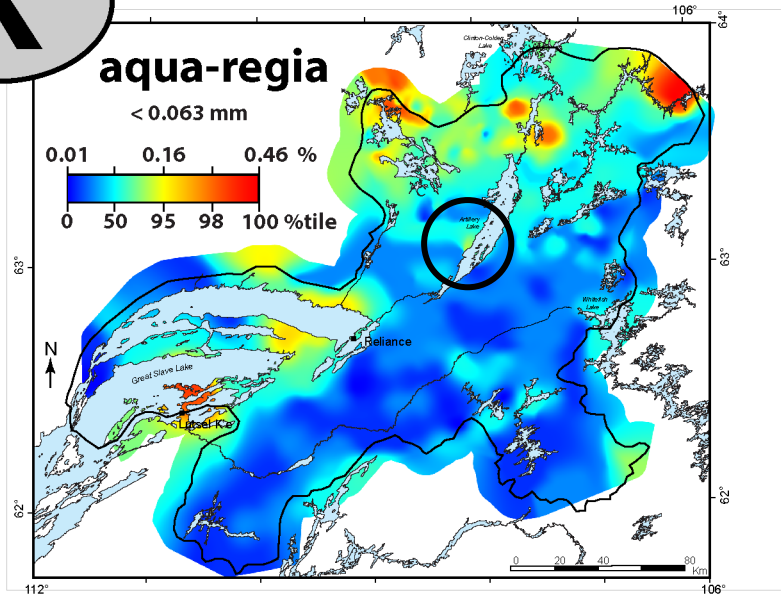


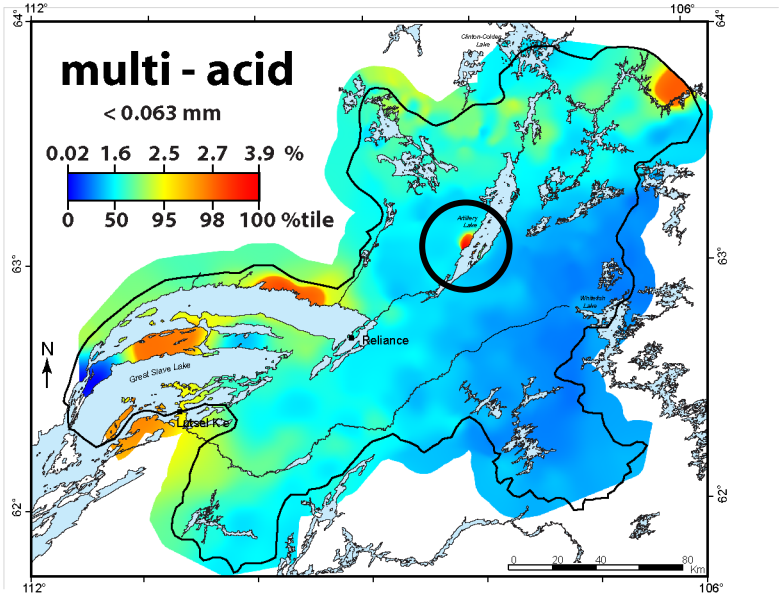


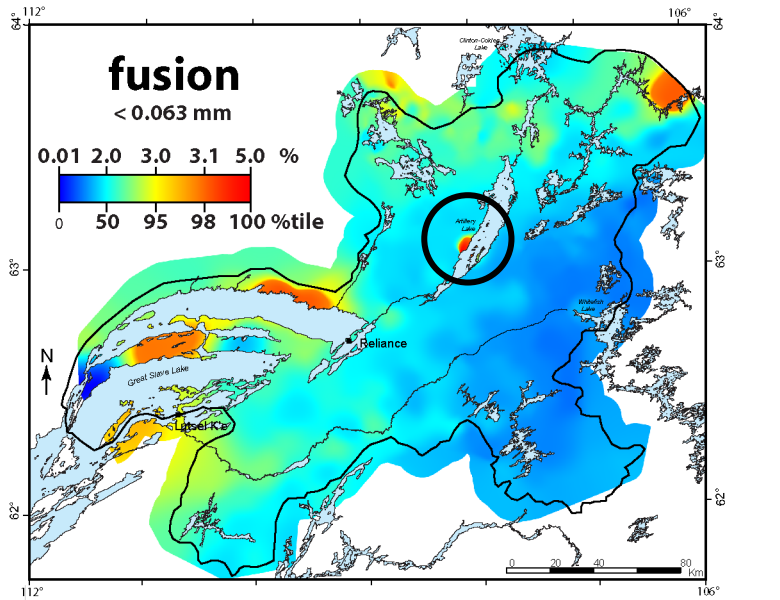


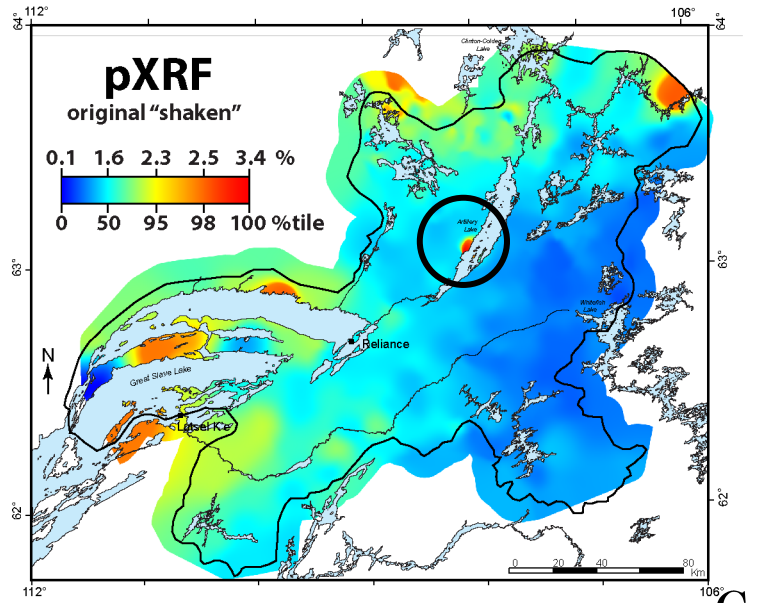
Comparison of K by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

K

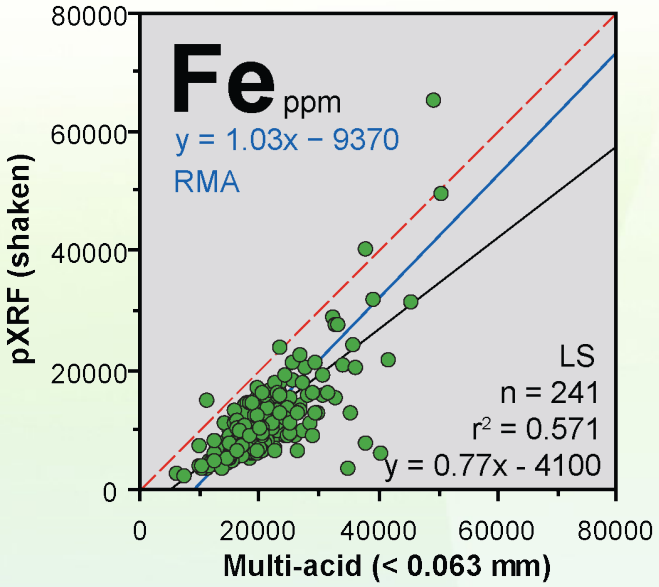


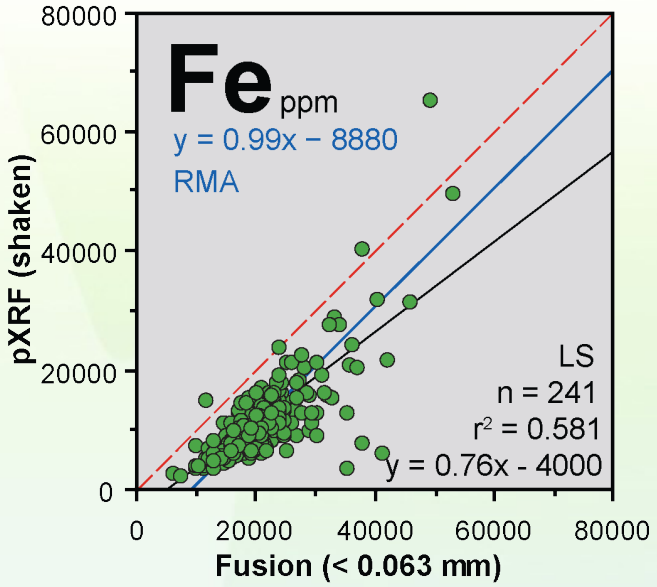


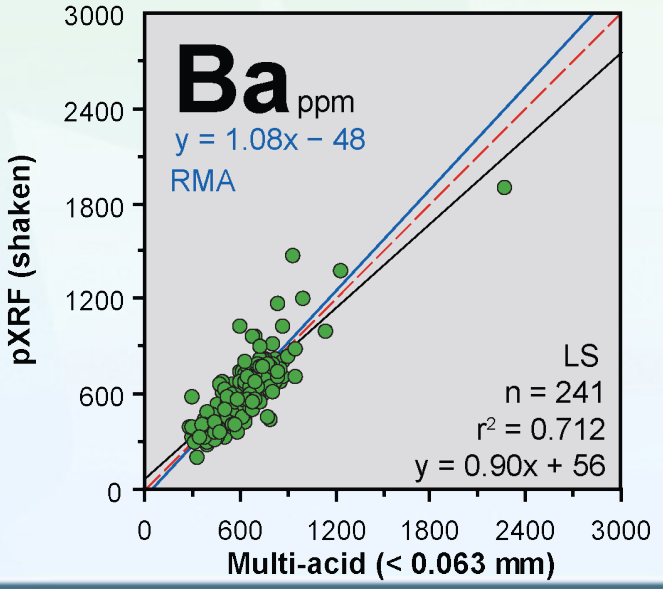


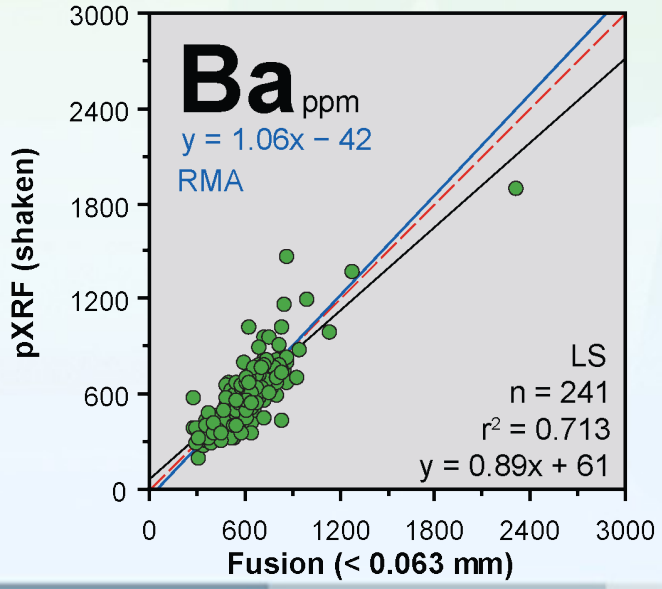


Comparison multi-acid and fusion (<0.063 mm) with pXRF ('shaken')



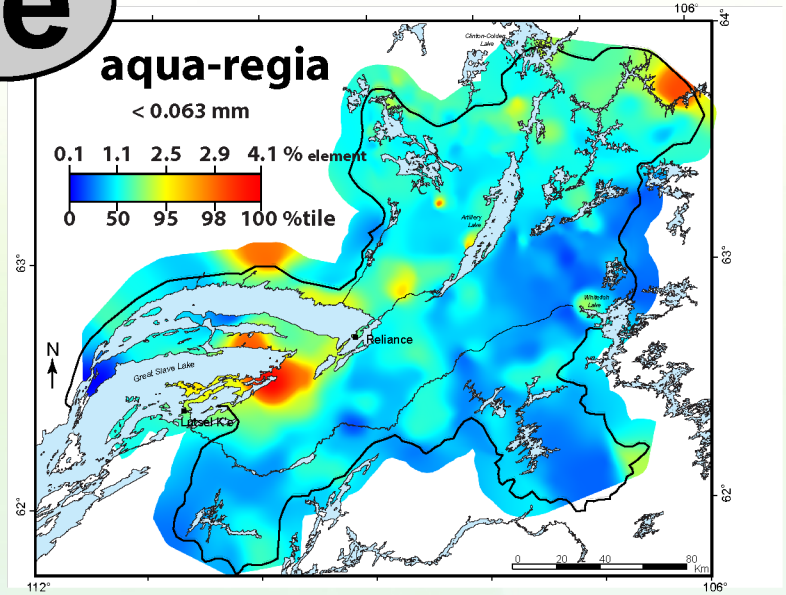


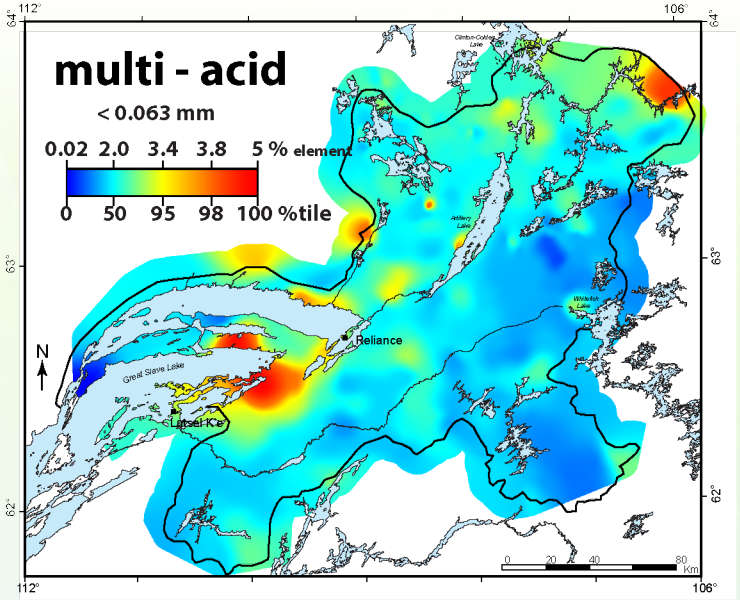


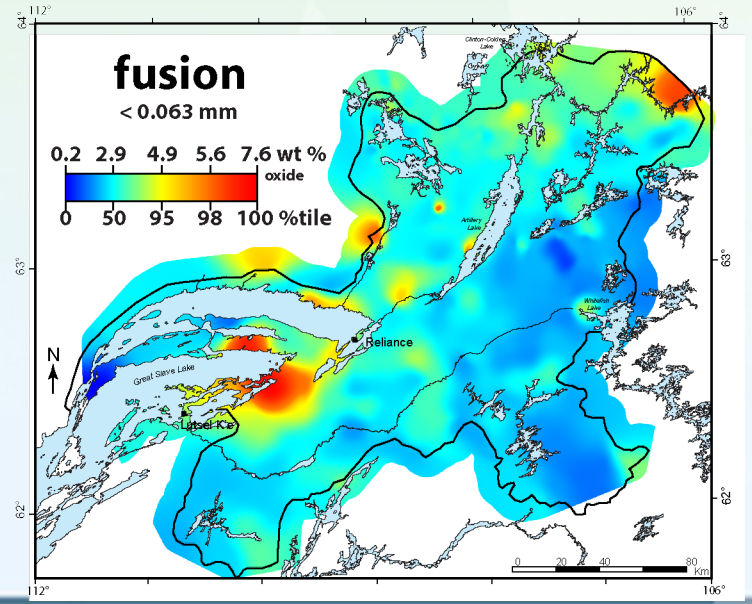


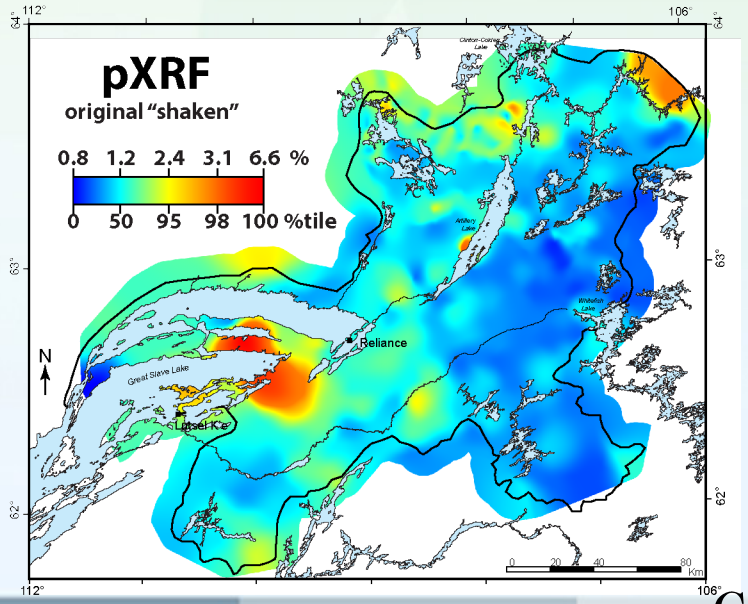
Comparison of Fe by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

Fe



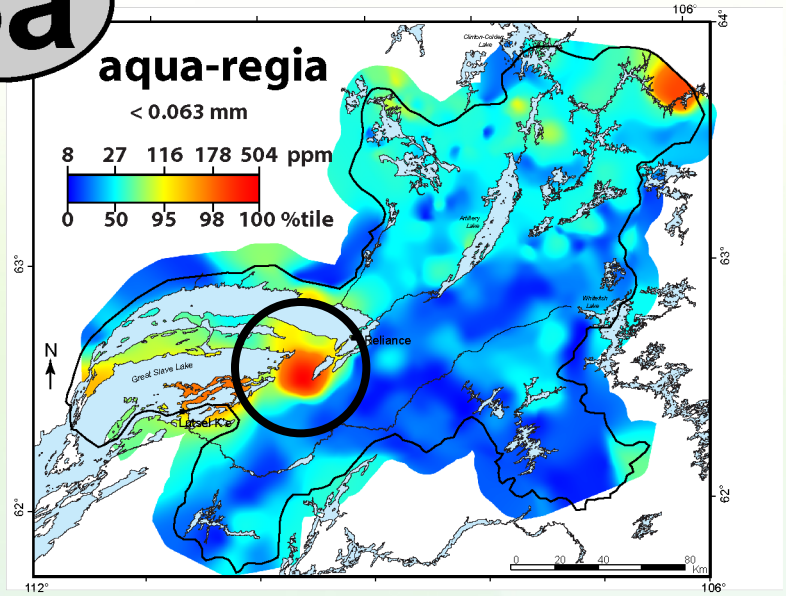


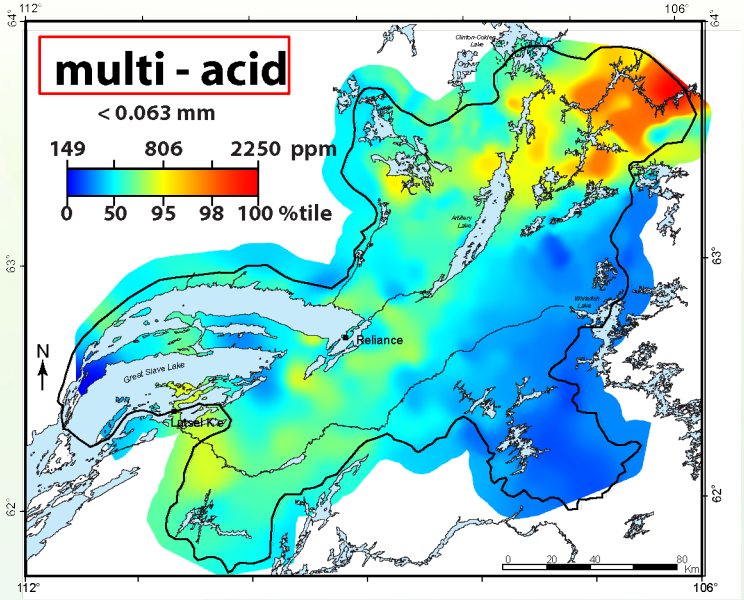


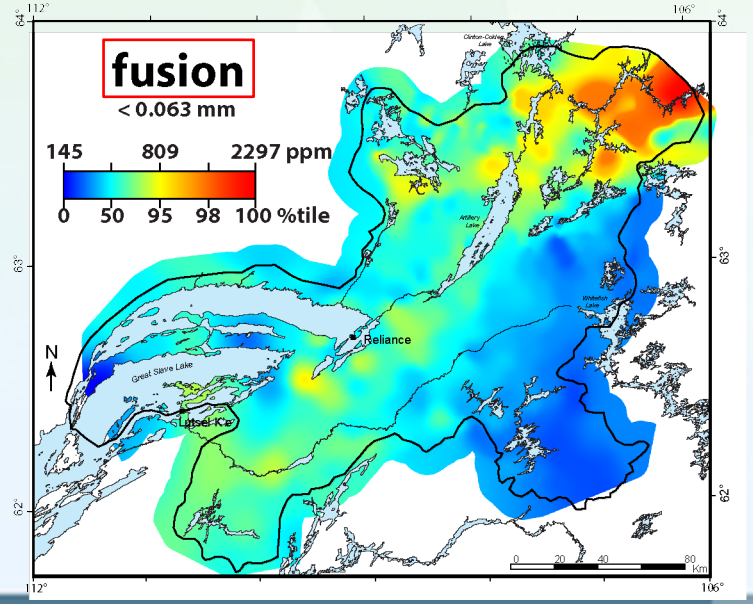


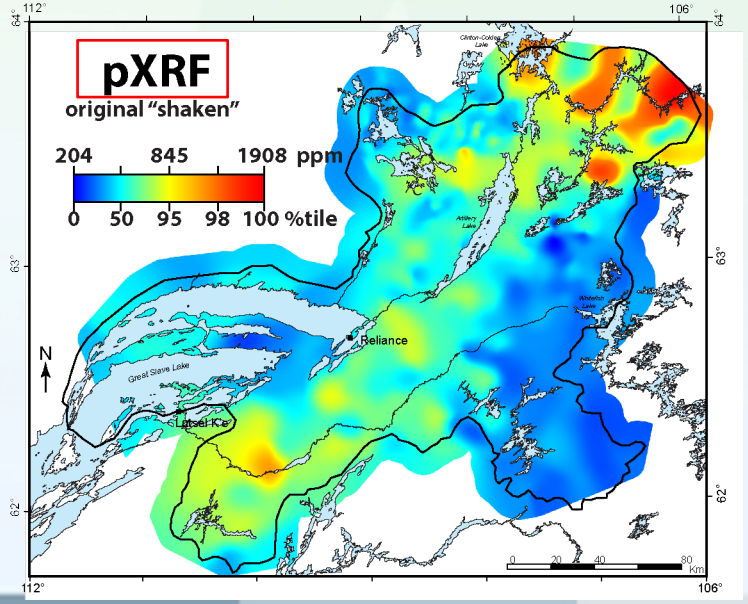
Comparison of Ba by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

Ba

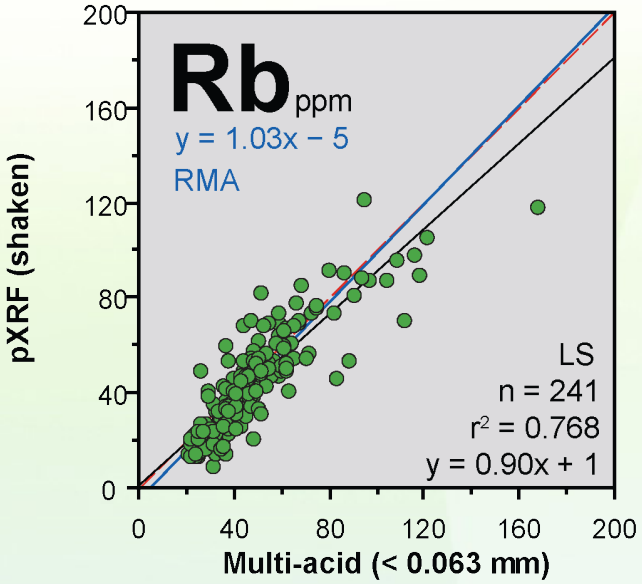


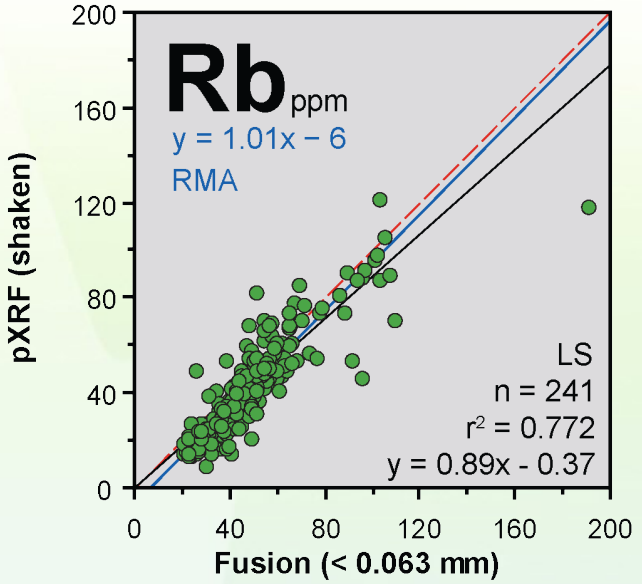


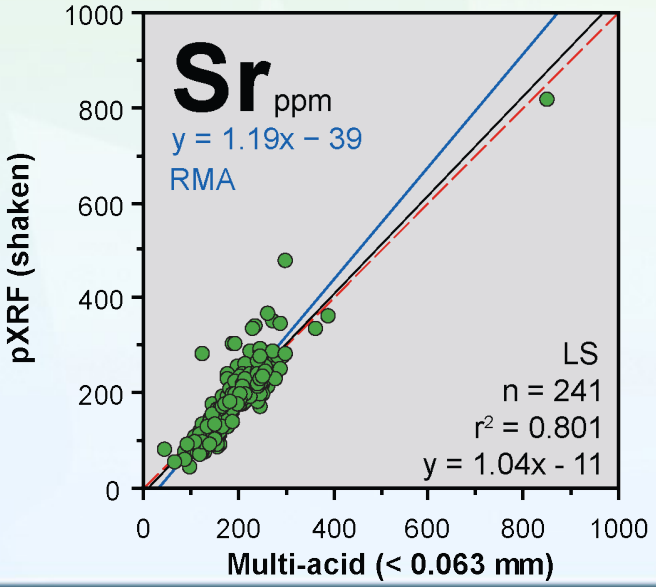


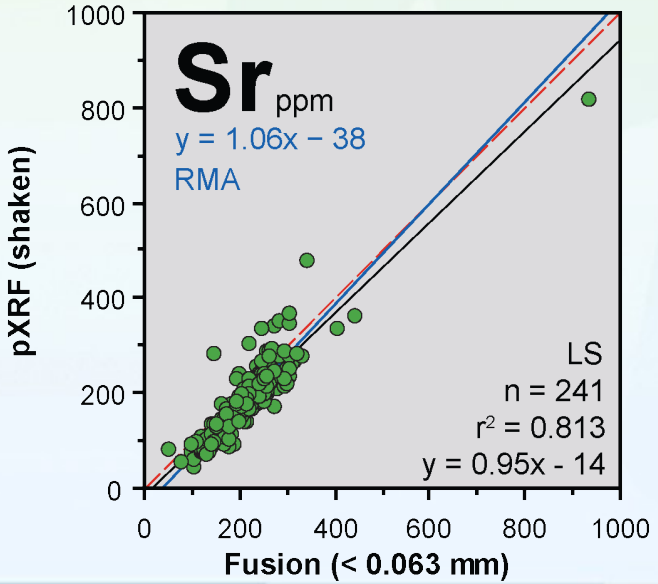


Comparison multi-acid and fusion (<0.063 mm) with pXRF ('shaken')



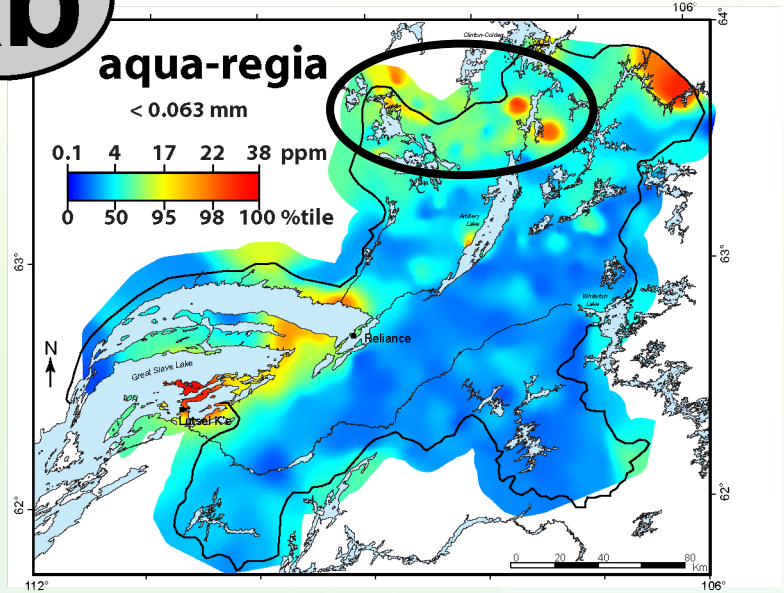


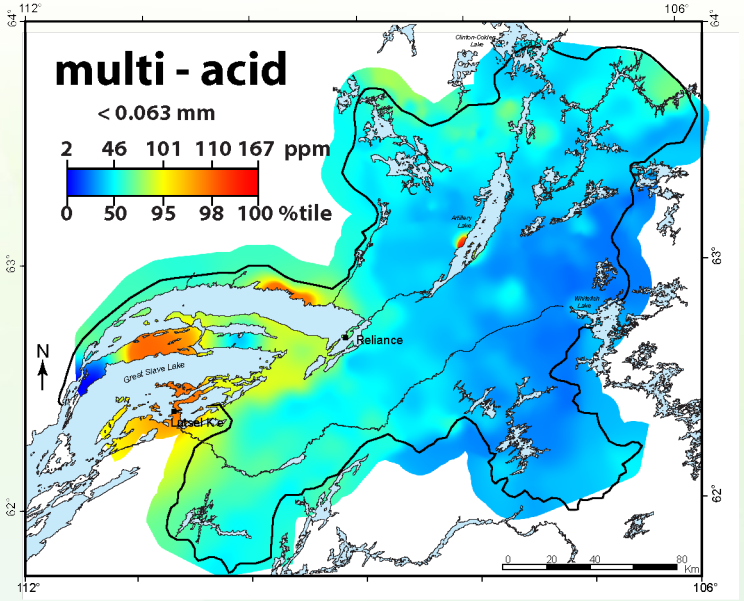


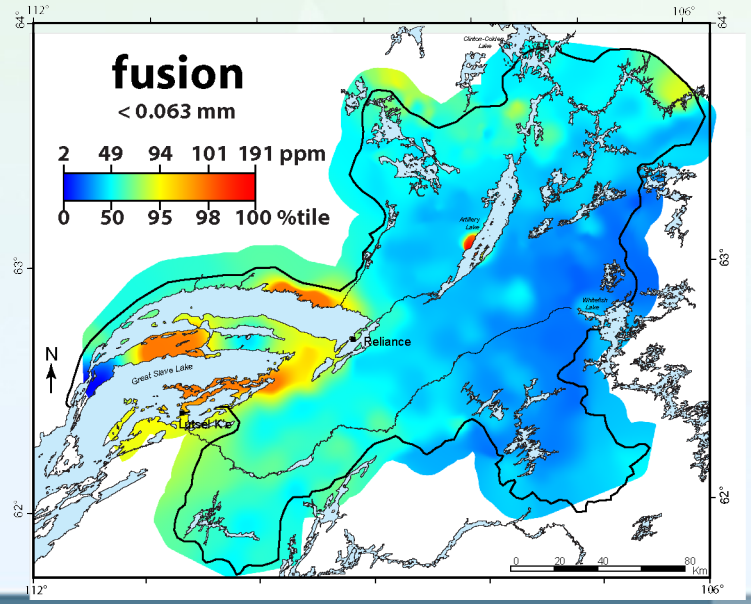


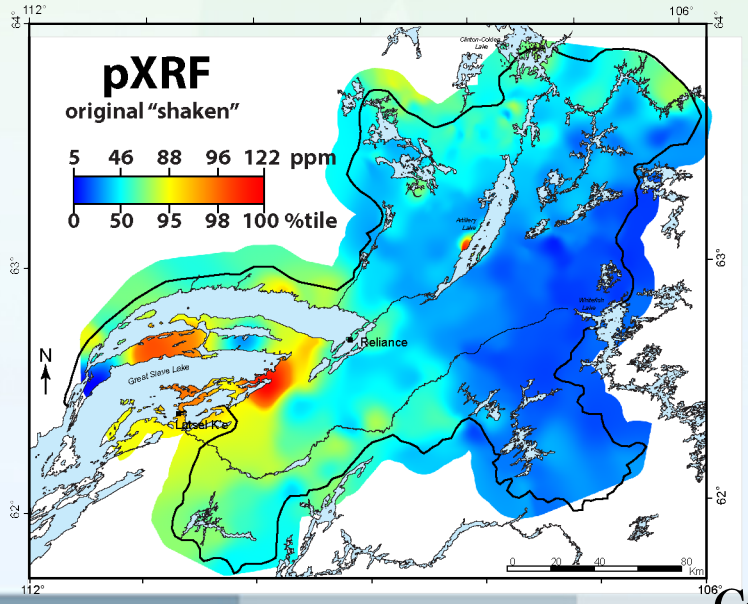
Comparison of Rb by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

Rb



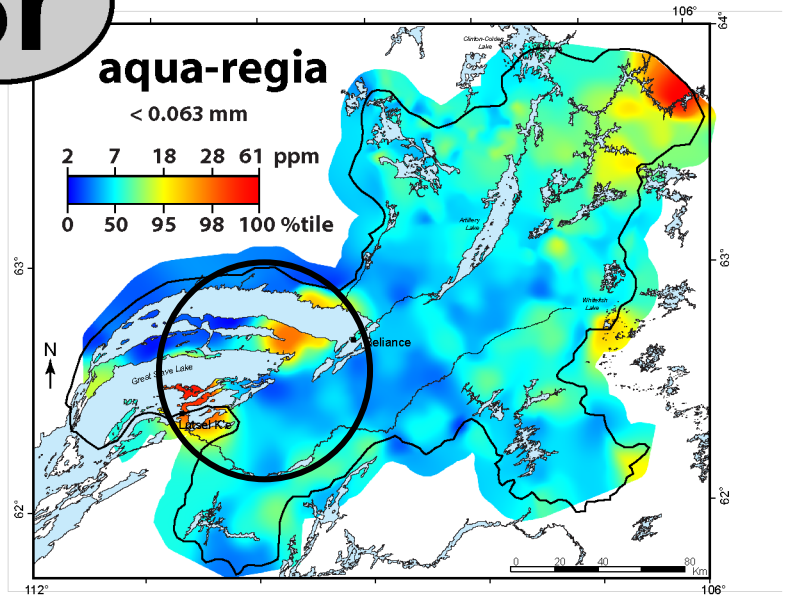


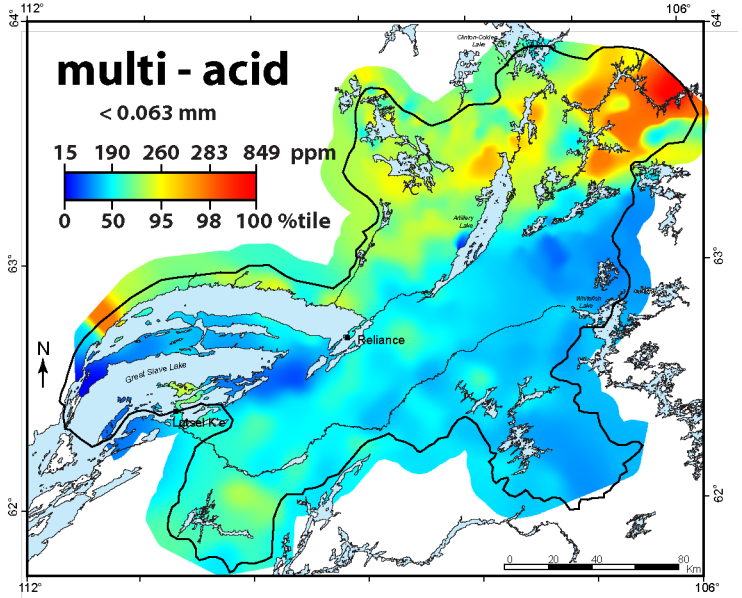


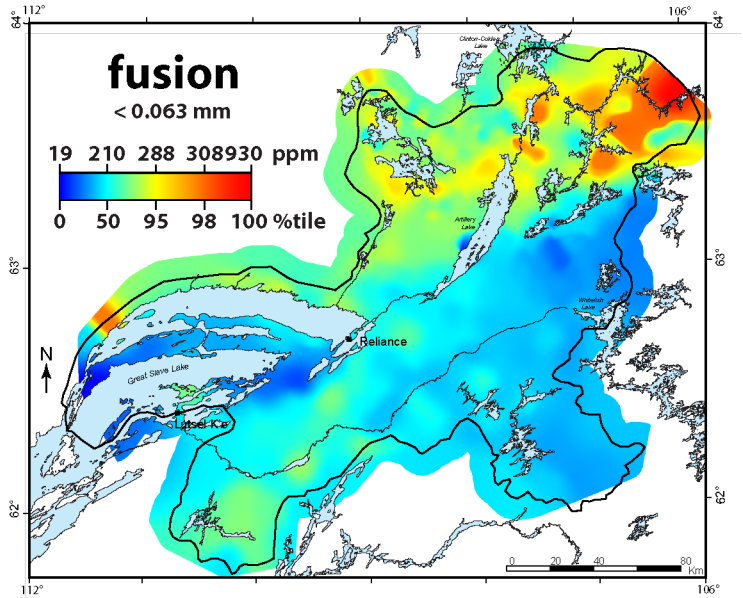


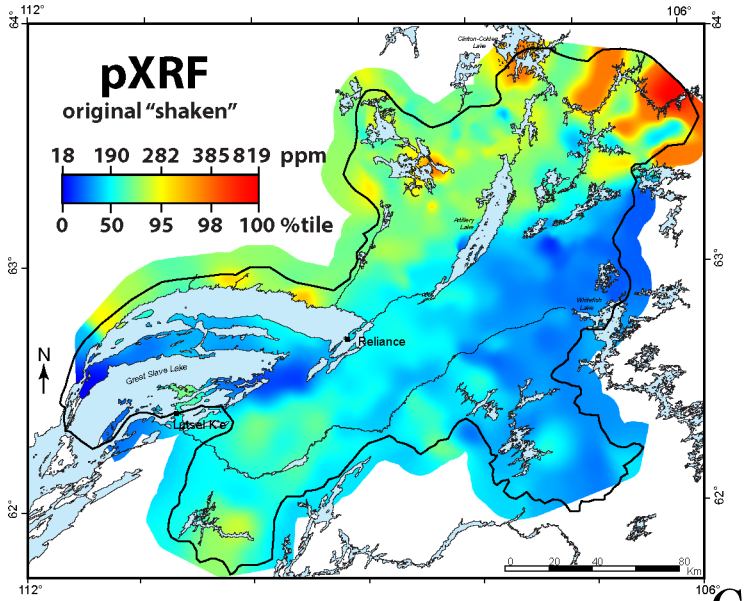
Comparison of Sr by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

Sr

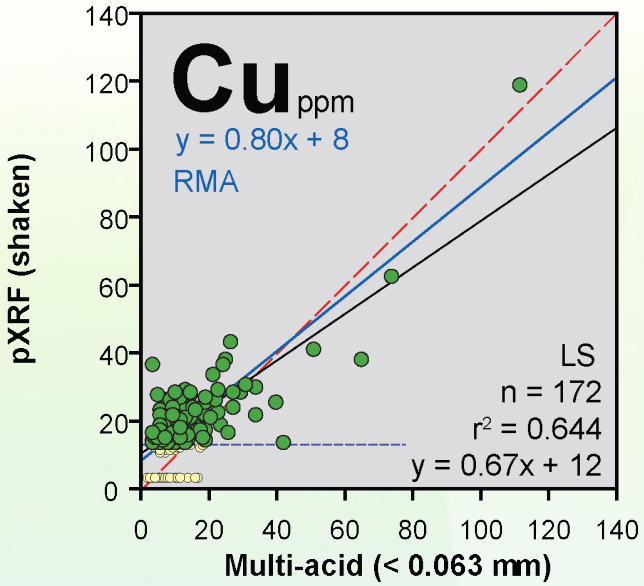


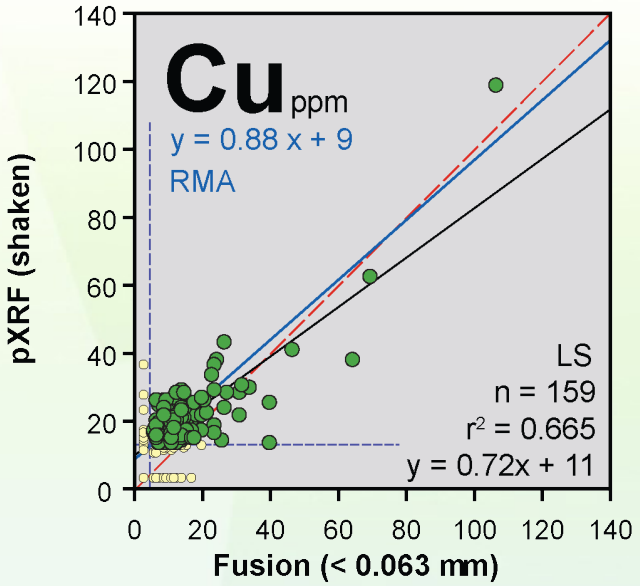


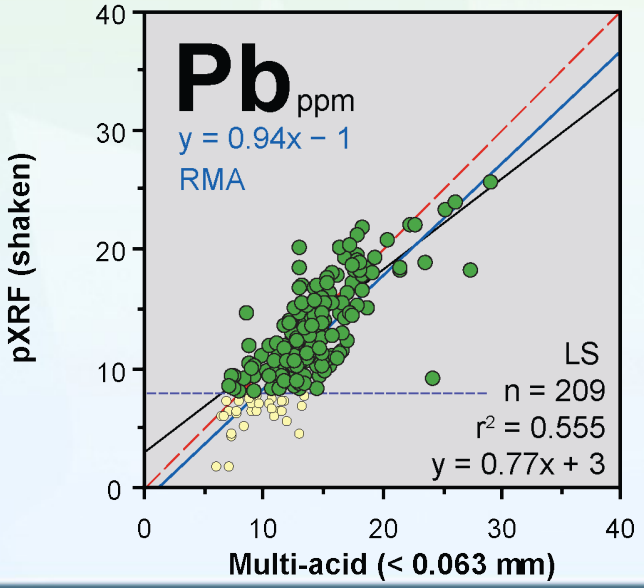


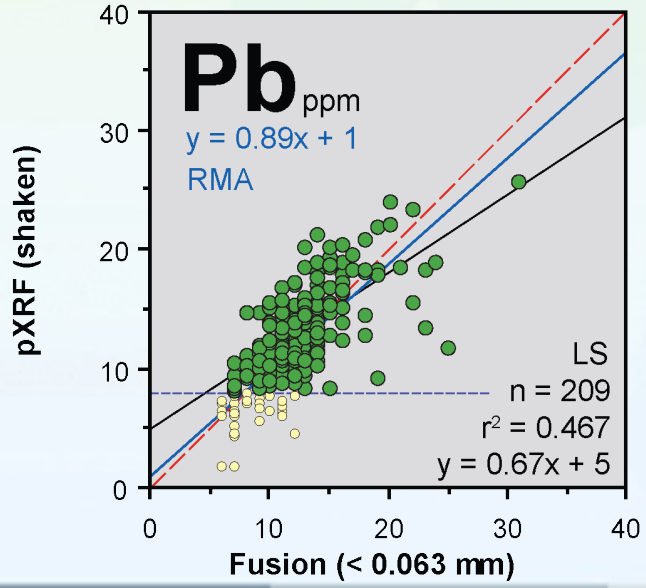


Comparison multi-acid and fusion (<0.063 mm) with pXRF ('shaken')



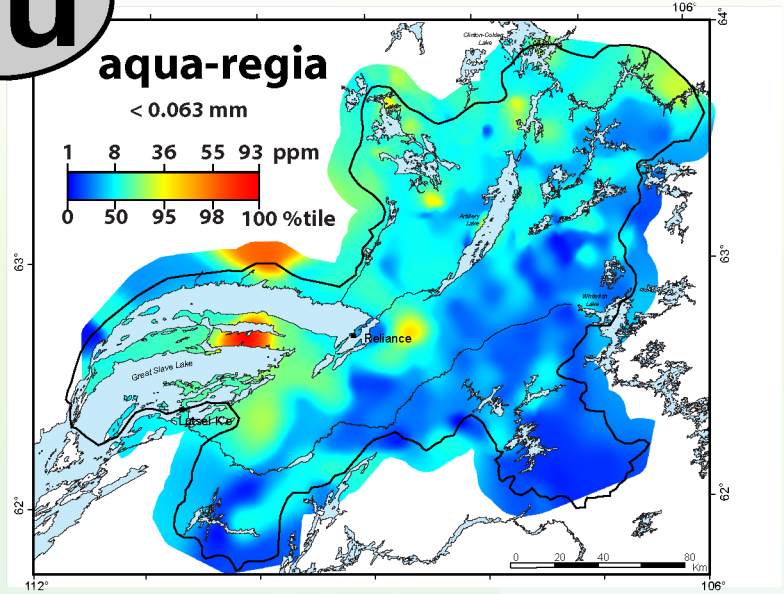


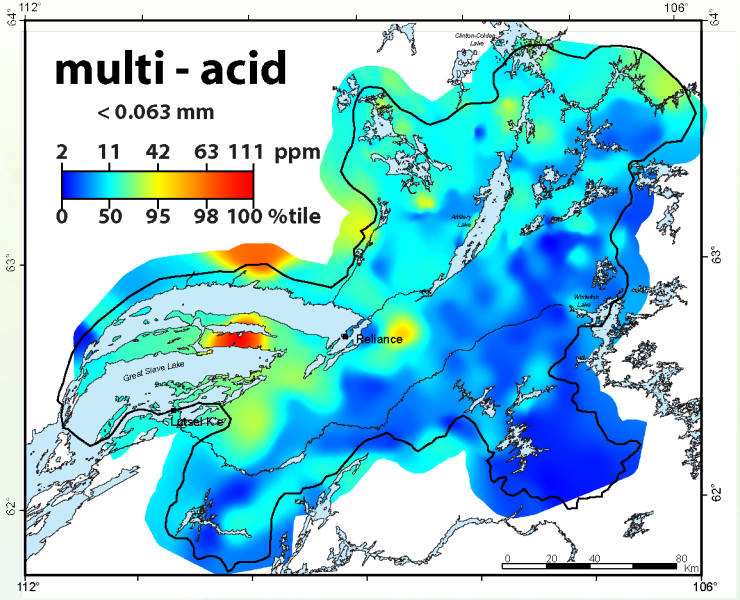


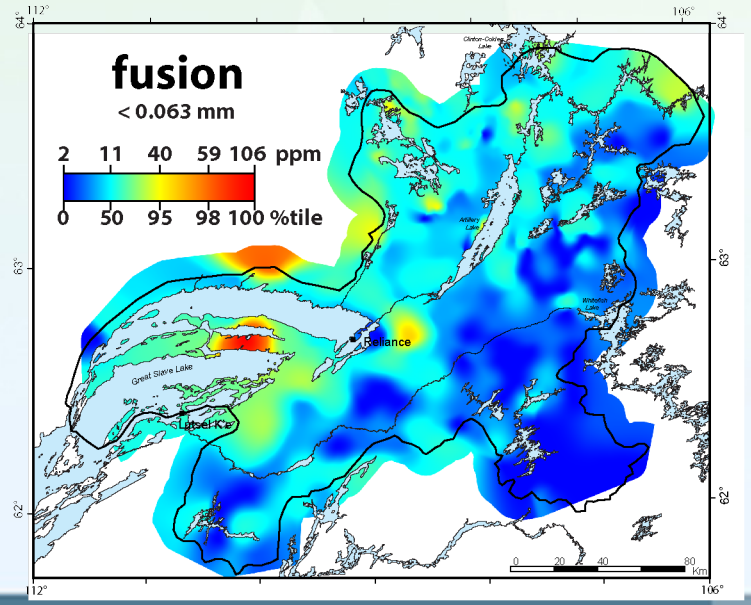


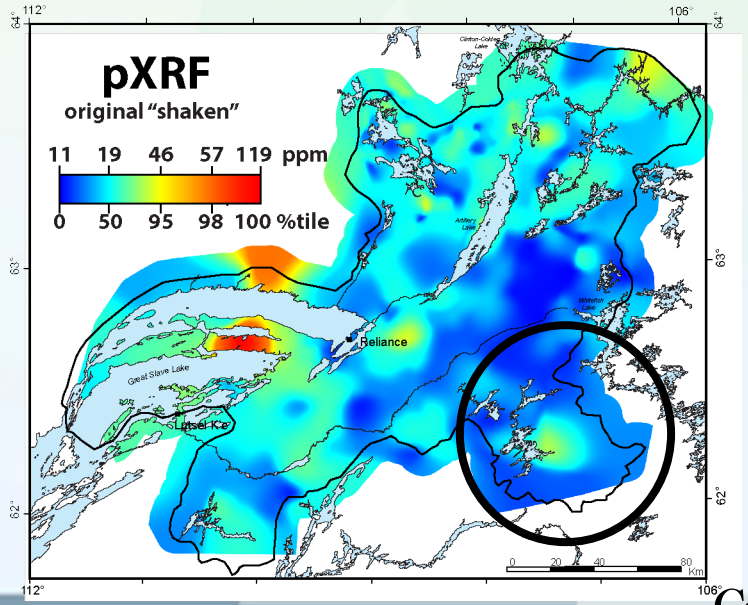
Comparison of Cu by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

Cu



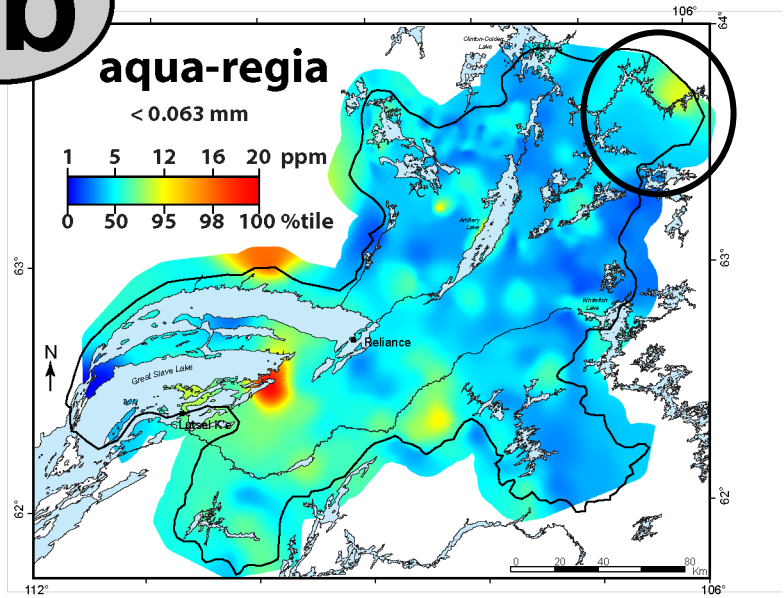


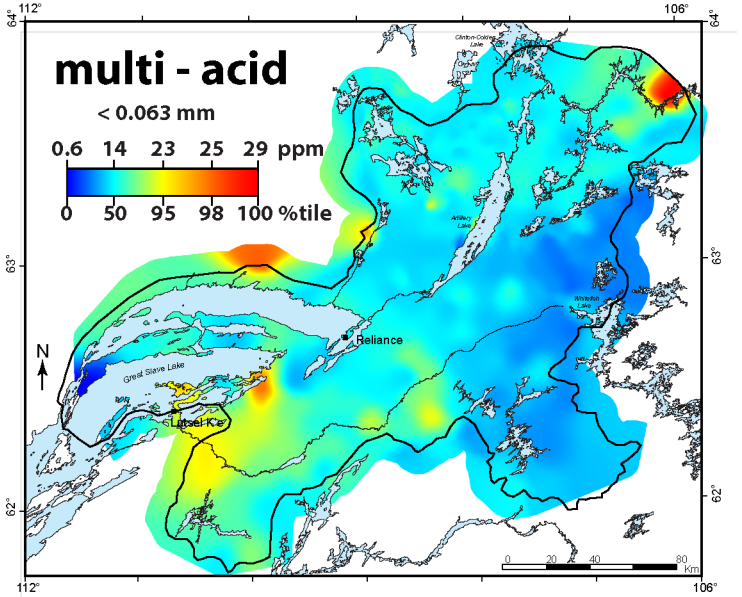


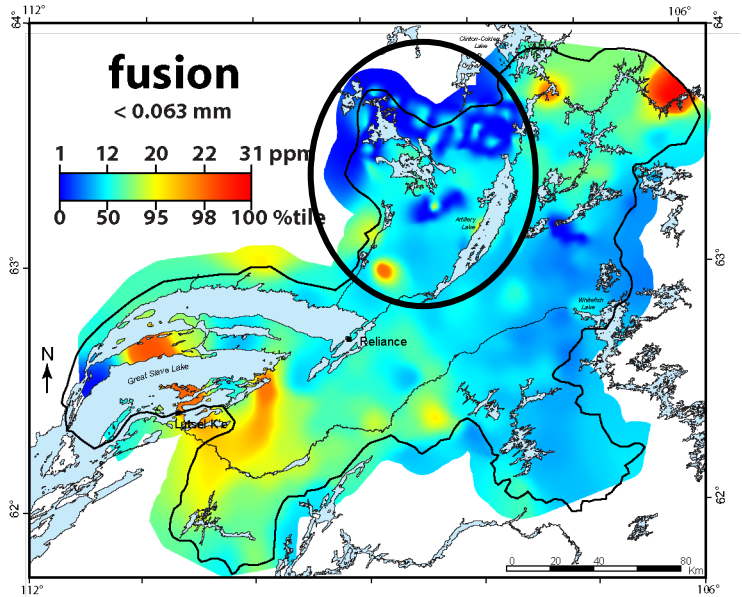


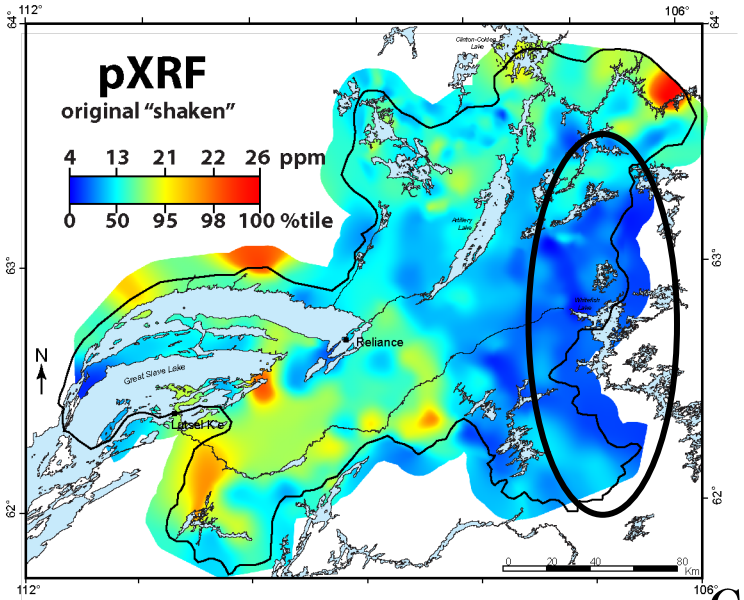
Comparison of Pb by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

Pb

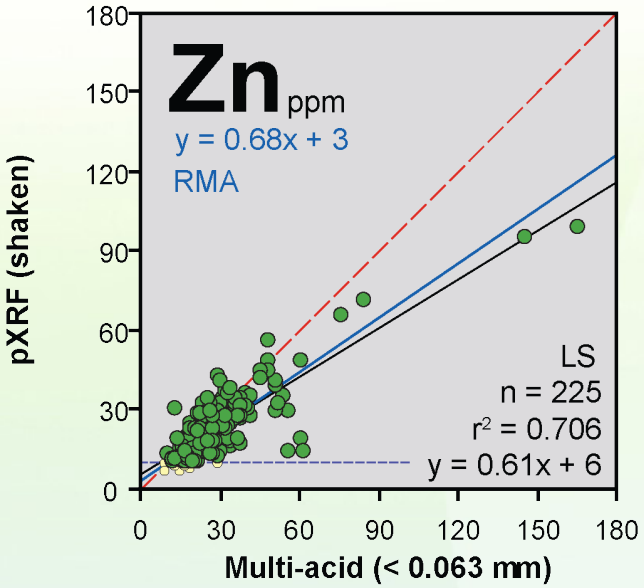


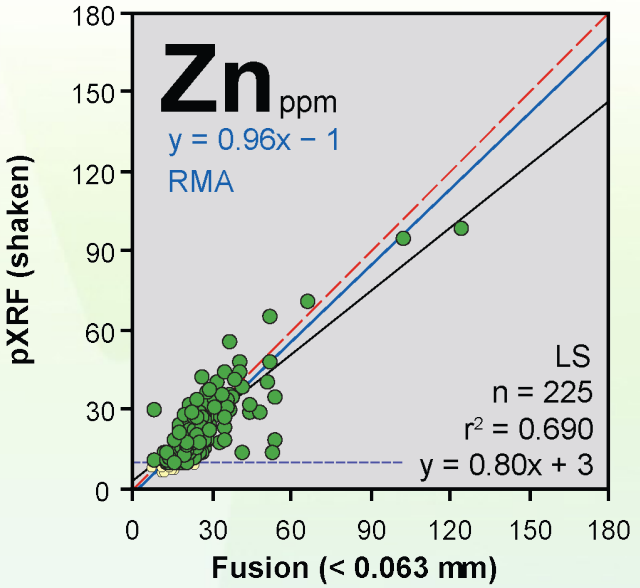


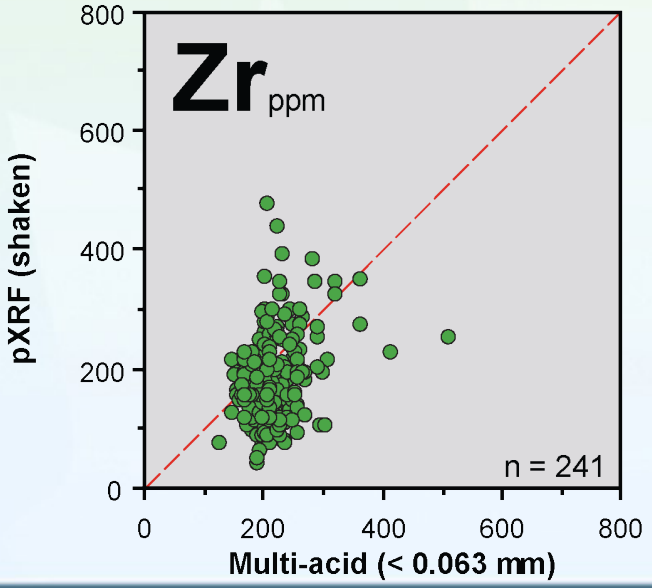


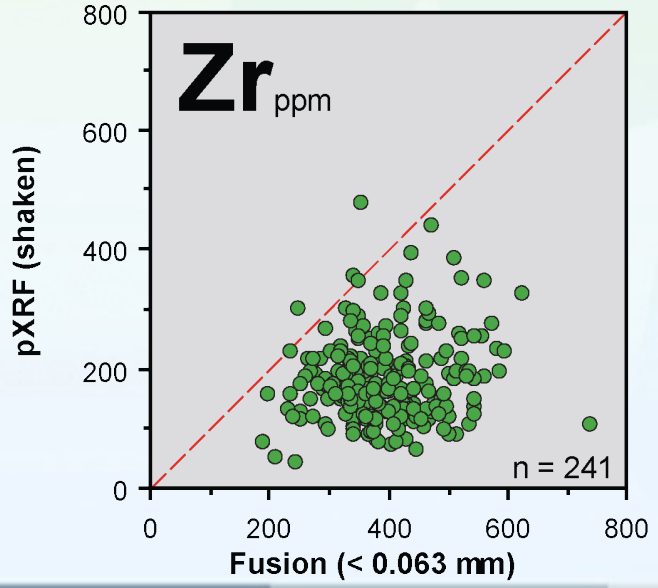


Comparison multi-acid and fusion (<0.063 mm) with pXRF ('shaken')



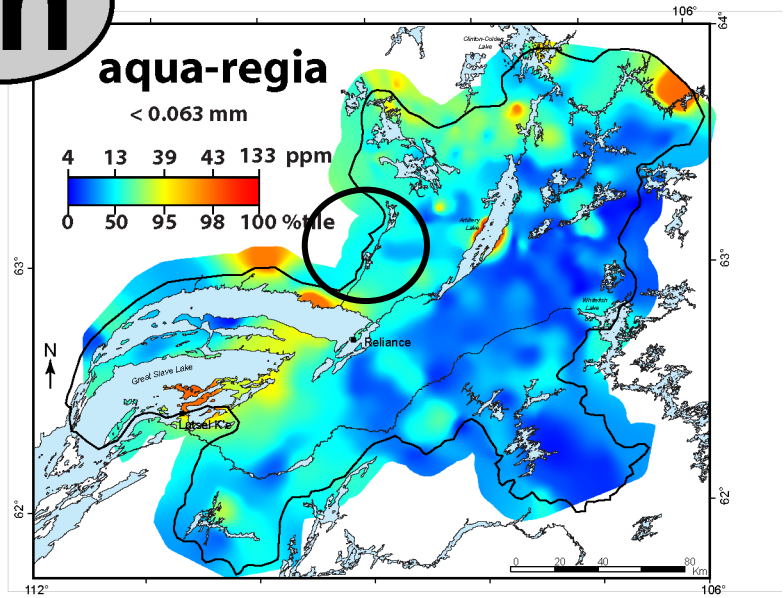


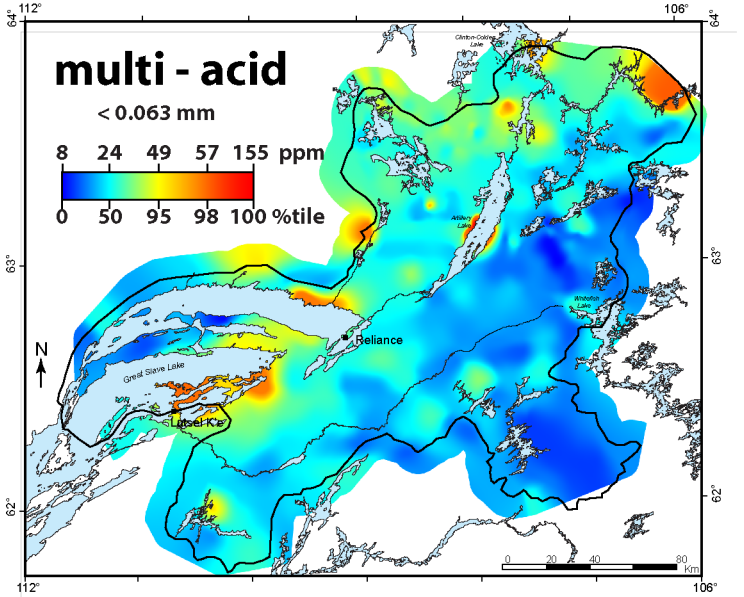


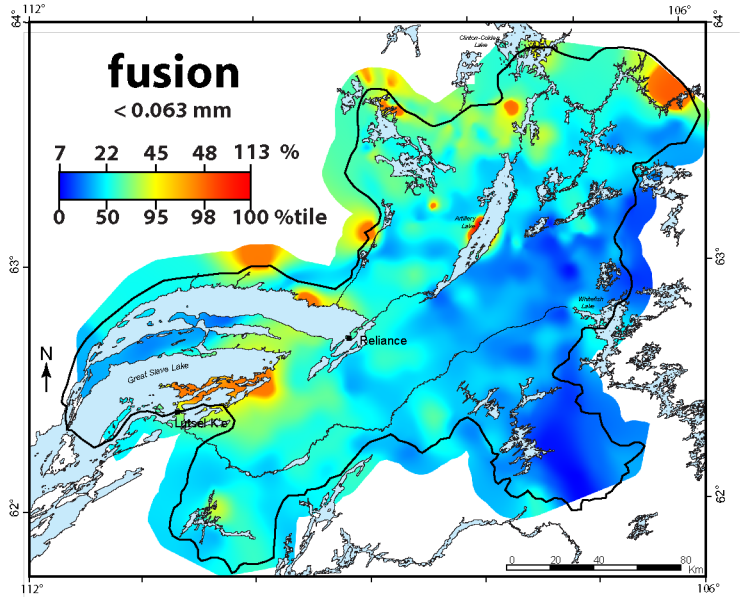


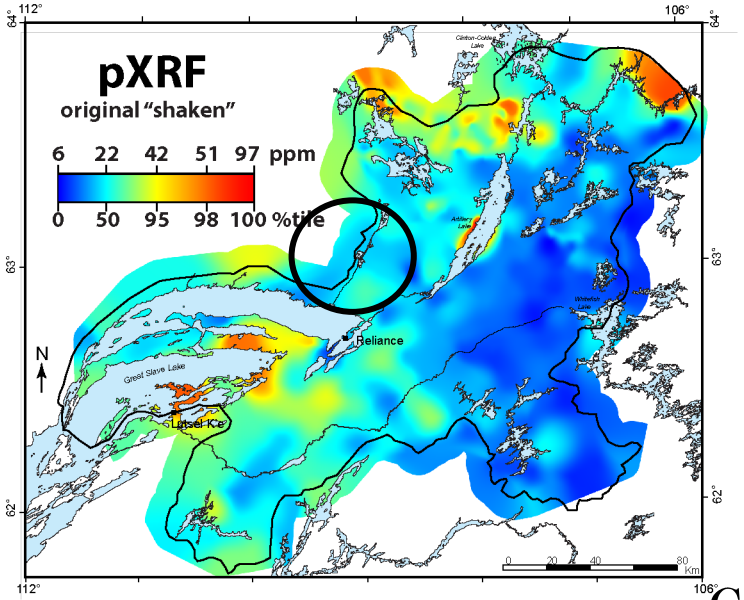
Comparison of Zn by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

Zn



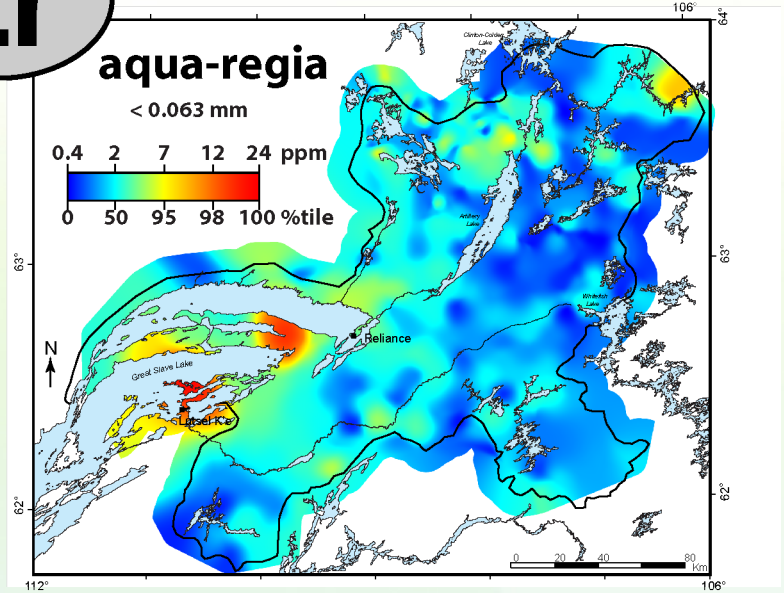


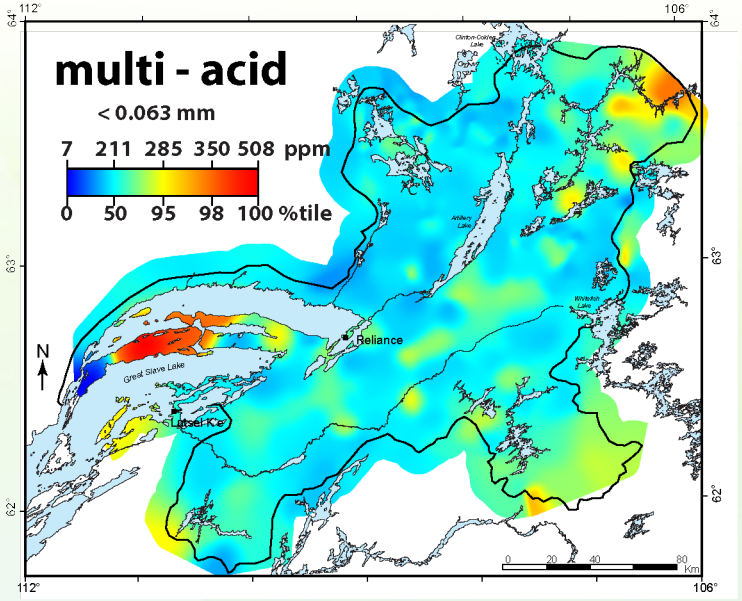


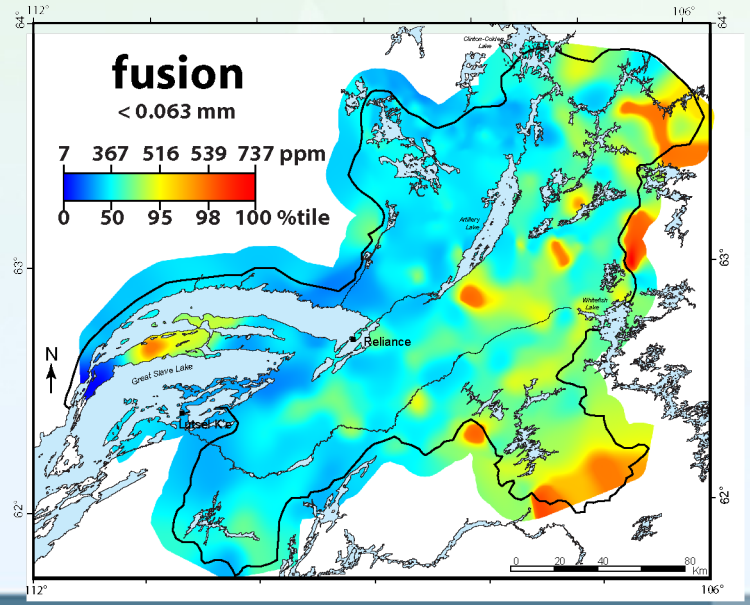


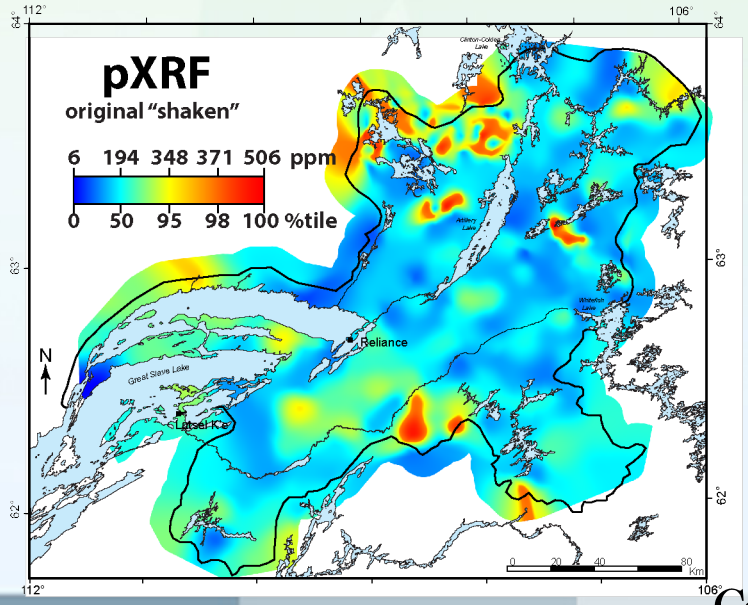
Comparison of Zr by pXRF ('shaken') with aqua regia, multi-acid and fusion methods

Zr









Summary

- Interpolated maps produced from pXRF data are comparable to geochemical maps produced using traditional laboratory multi-acid and fusion analytical methods.

- It is advisable to dry, and sieve samples to <2 mm or smaller prior to analysis by pXRF. Utilizing the inverting and 'shaking' technique produces a more clay-silt rich fraction at the beam interface compared to an unprocessed or <2 mm size fraction.

- *Portable XRF spectrometry allows for better use of in-field time as a tool to prioritize resources to re-sample specific sub-regions at higher density.*

Summary – Part 2
Affiliated GSC Research

- ~ 9000 analyses of Certified and Standard Reference materials
- ~ 4000 analyses of sediment (unconsolidated) and rock samples

	Near Definitive	Quantitative	Qualitative	Inconclusive
	$r^2 \geq 0.85$, RSD $\leq$ 10%, $y = mx + b$	$r^2 \geq 0.7$, RSD $<$ 20%, $y = mx + b$	$r^2 < 0.7$, RSD $>$ 20%, $y \neq x$	$r^2 \leq 0.6$, RSD $>$ 20%, $y \neq x$
Soil Mode	Ca, Fe, Rb, Sr, Ti, Zn, Zr	As, Ba, Cu, K, Mn, Pb, V	Cr, Mo, S, Se, Th, U	Ag, Au, Cd, Co, Cs, Hg, Ni, Pd, Sb,(Sc) Sn, Te, W
Mining Cu/Zn Mode	K, Ba, Ca, Fe, Rb, Sr, Ti, Y, Zn, Zr	As, La, Mn, Pb, S, U, W	Cu, Mo, Th	Ag, Al, Au, Bi, Cd, Ce, Cl, Co, Cr, Mg, Nb, Nd, Ni, P, Pr, Sb, Se, Si, Sn, V

Summary – Part 3
Analytical considerations

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An analytical protocol for determining the elemental chemistry of Quaternary sediments using a portable X-ray fluorescence spectrometer

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Quaternary sediments
Sample preparation
Dwell time
Instrument drift
Variable grain sizes

ABSTRACT

The modern geologist has a plethora of portable digital tools at their disposal including, GPS, in-field computers providing access to imagery, maps, and on site real time geochemistry provided by portable X-ray fluorescence spectrometry (pXRF). This paper presents an analytical protocol for the examination of Quaternary glaciogenic sediments derived from different bedrock terrains using a portable spectrometer. The protocol outlines best practices for the collection of geochemical data using pXRF from glacioiogenic sediments with generally continental crustal abundance elemental concentrations at a fraction of the cost of traditional laboratory methods. The analytical protocol considers 1) sample preparation, 2) sample analysis, and 3) data cleaning, examination and presentation. The protocol takes into consideration pXRF fundamentals related to, instrument configuration, and an assessment of measurement parameters, including peak overlaps, dwell time, instrument drift, in-situ versus processed samples, variable grain size and moisture content effects, and comparison to traditional laboratory methods. Protocol development occurred over a number of years and analysis of over 4000 geological samples and ~9000 analysis of Certified and Standard Reference Materials.