Geological Survey of Canada
Scientific Presentation 149

QUANTIFYING THE INVISIBLE: PXRF ANALYSES OF THREE BOREHOLES, BRITISH COLUMBIA AND ONTARIO

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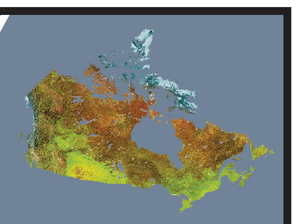

29th IAGS2022 Viña del Mar, Chile

Objectives

Portable X-ray fluorescence spectrometry (pXRF) has revolutionized the gathering of geochemical data for mineral exploration, environmental surveys and opened new avenues of research and applications in chemostratigraphy.

Demonstrate pXRF Spectrometry to quantify the invisible on a budget

1) Enhance the understanding of geochemical controls on groundwater chemistry
2) Guide, refine and enhance cost effectiveness of field sampling
3) Define changes in sediment lithology and provenance
4) Define unrecognizable formational boundaries

Methods

Quaternary Sediments

1) Dry and disaggregate sample
2) Sieve to <63 µm (silt + clay)
3) Place sample in 23 mm diameter plastic vials, to a height of 30 mm, to obtain sufficient density and "infinite thickness"
4) Seal vials with 4 µm thick Chemplex Prolene
5) Analyzed sample using Thermo Scientific, Niton XL3t GOLDD spectrometer in Soil mode with a 60 second dwell time per filter
6) Insert a Teflon and silica blank, plus multiple CRM's, at the beginning and end of every session and after every10 samples

Bedrock Core

1) Core was washed with distilled water to remove contaminants
2) Analyzed samples using Thermo Scientific, Niton XL3t GOLDD spectrometer in Mining mode with a 45 second dwell time per filter
3) pXRF was mounted on a modified drill press with an external roller system to move core boxes under the spectrometer window
4) Insert a Teflon and silica blank, plus multiple CRM's, at the beginning and end of every session and after every 20 samples

Conclusions

pXRF analyses can be used as a low-cost analytical tool when traditional geochemical analysis (ICP/MS-ES) are beyond project scope and budget.

Our data supports the application of pXRF spectrometry to provide near-instantaneous elemental composition of major and trace elements of rock and unconsolidated sediments for chemostratigraphic studies. Research at the Geological Survey of Canada (published in Applied Geochemistry 2021) identifies ~17 elements with near definitive or quantitative results.

	Near Definitive	Quantitative	Qualitative	Inconclusive
	$r^2 \geq 0.85$, RSD ≤ 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

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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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ABSTRACT
The modern geologist has a plethora of portable digital tools at their disposal including, GPS in-field computers providing accessto 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 glaciogenic 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.

1. Introduction

Portable X-ray fluorescence spectrometry (pXRF) has revolutionized the gathering of geochemical data for mineral exploration, environmental surveys and opened new avenues of research and applications in chemostratigraphy. Ongoing advances in pXRF technology have resulted in the ability to collect geochemical data that would otherwise be beyond the financial scope of many projects where traditional laboratory geochemical analysis (i.e., comprehensive aqua regia, plus multi acid, plus fusion digestions followed by inductively-coupled plasma-mass spectrometry (ICP-MS/ES)), can cost ~100$/sample. Besides being cost effective, the spectrometer is an excellent tool for real-time analyses and screening purposes for a suite of elements ranging from magnesium (Mg) to uranium (U). The development of pXRF as a mainstream geochemical analytical tool is summarized in Glanzman and Closs (2007), updated by Lemiere (2018), and compared with results from traditional laboratory methods by Rouillon and Taylor (2016).

Although there have been over 250 peer-reviewed journal papers on the use of pXRF technology in the geosciences only a few provide details on analytical protocols to obtain precise and accurate data (Laiho and Peltamäki, 2005; Fisher et al., 2014; Gazley et al., 2014a, 2014b; Sarala 2016; Adams et al., 2020; Ravansari et al., 2020). Several papers do review specific factors affecting pXRF performance with emphasis on sample heterogeneity, analysis time, instrument stability, protective films, sample thickness, analyte interferences, detector resolution, and instrument drift (Hall et al., 2012; Weindorf et al., 2014; and Ravansari et al., 2020).

Since 2009, the Geological Survey of Canada (GSC) has employed pXRF spectrometry to characterize the chemical composition of glacial Quaternary sediments, which blanket most of Canada and the northern United States. With completion of each research project the protocol was modified to incorporate gained knowledge and has recently been applied to a subsurface aquifer/aquitard characterization study in southern Ontario, Canada (Russell and Kjarsgaard, 2020).

The objective of this paper is to outline an analytical protocol, and supporting background information on protocol development, for portable XRF analysis of glaciogenic sediments. To constrain the protocol and ensure analytical confidence extensive characterization of Certified Reference Materials (CRM's), Standard Reference Materials (SRM's) was completed, and combined with ~4000 Quaternary sediment samples. During the course of this work insights were developed regarding choosing a "fit-for-purpose" instrument, problematic peak

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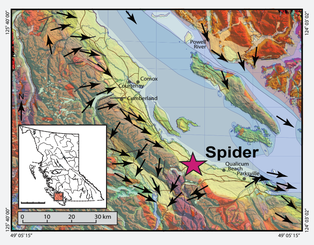
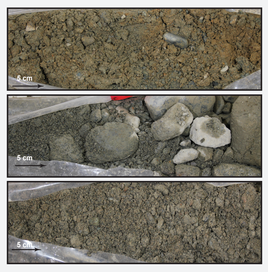
Quaternary Sediments - Southwestern British Columbia

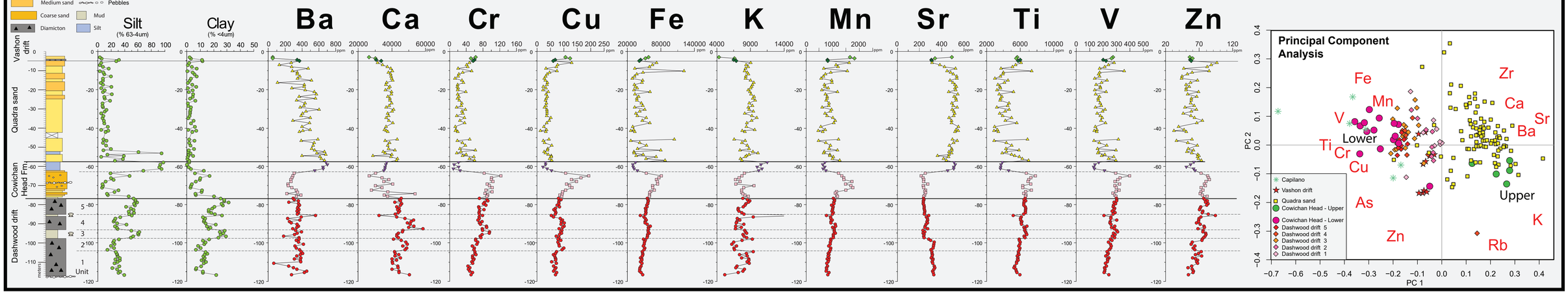


The Cowichan Head Fm. consists of a lower and upper member. however the geochemical signature shows that the provenance of the lower member is similar to the underlying Dashwood drift whereas the provenance of the upper member is similar to the overlying Quadra sand.



Nanaimo - Spider Soil Mode

Location : Quacilum, British Columbia
Project : Groundwater Assessment
Study Area : Nanaimo Lowlands
Size Fraction : <0.063 mm
Original Material : Freeze-dried, disaggregated, seived
Vial Window Material : 4 micron SpectroCertified Mylar polyester
pXRF : Niton XL3t GOLDD, 50-kV Cygnet X-ray tube
Dwell Time : 60 seconds per High, Main, and Low filter
GSC Open File 7651

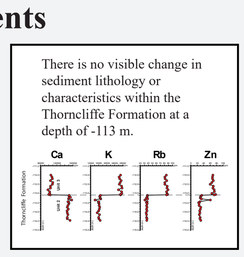


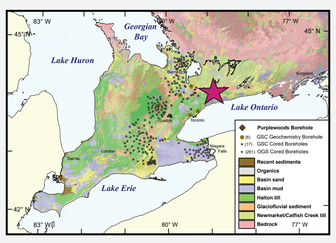
Quaternary Sediments Southern Ontario

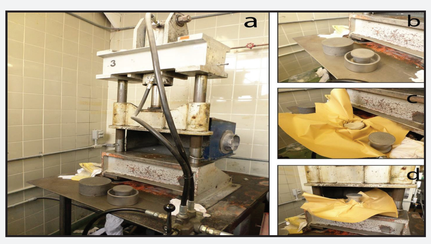
Sediment types
- Soil profile
- Clay
- Silt
- Sand
- Diamicton
- Gravel
- Bedrock
- Missing core

LOD Limits of Detection

There is no visible change in sediment lithology or characteristics within the Thorncliffe Formation at a depth of ~113 m.



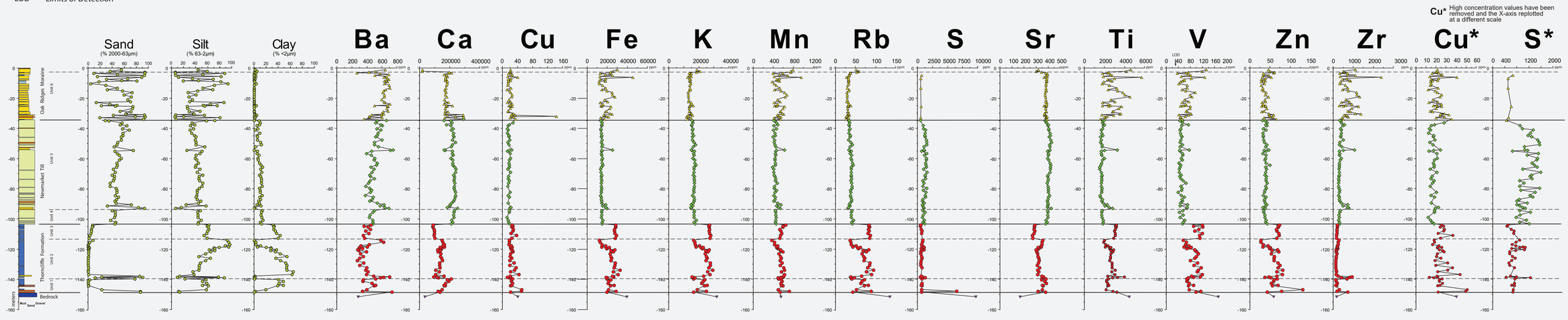




Purple Woods Soil Mode

Location: Purple Woods Conservation Area, Ontario
Project: Groundwater Assessment
Study Area: Oak Ridge Moraine
Size Fraction: <0.063 mm
Original Material: Disaggregated, seived
Vial Window Material: 4 micron SpectroCertified Mylar polypropylene
pXRF: Niton XL3t GOLDD, 50-kV Cygnet X-ray tube
Dwell Time: 60 seconds per High, Main, and Low filter
GSC Open File 7899

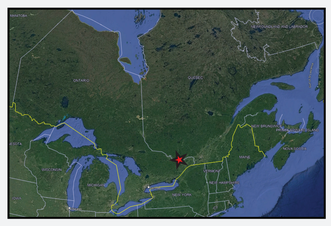
Cu* High concentration values have been removed and the X-axis replotted at a different scale

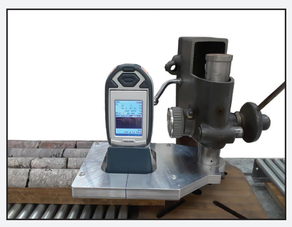


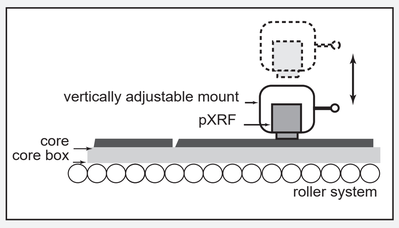
Precambrian and Ordovician Bedrock Southeastern Ontario

Paleozoic bedrock
- Sandstone - bioturbated
- Sandstone - shaley
- Sandstone
- Quartz Conglomerate
- Dolomite
- Dolomite - sandy
- Dolomitic sandstone

Precambrian bedrock
- Pegmatite
- Granite
- Syenite
- Gneiss - felsic
- Gneiss - mafic

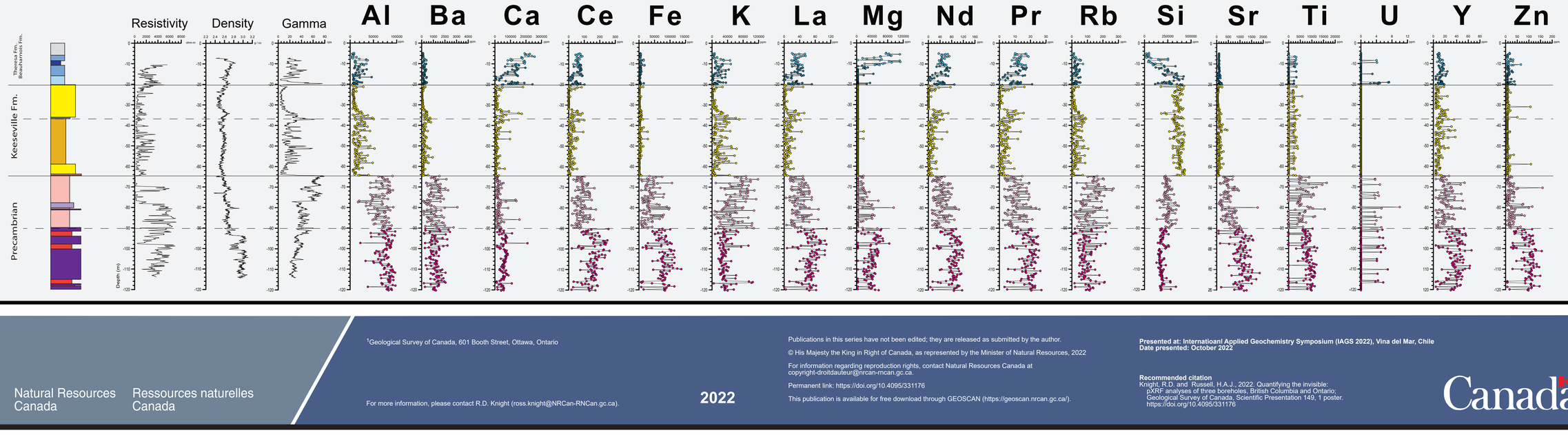






Bells Corners BC81-2 Mining Mode

Location : Ottawa, Ontario
Project : Groundwater Assessment
Study Area : Bells Corners
Original Material : Rock
Vial Window Material : 4 micron SpectroCertified Mylar polyester
pXRF : Niton XL3t GOLDD, 50-kV Cygnet X-ray tube
Dwell Time : 45 seconds per High, Main, Low and Light filter
GSC Open File 8913



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