Natural Resources Canada | Ressources naturelles Canada

GEOLOGICAL SURVEY OF CANADA
OPEN FILE 7712

Science Laboratory Network: Science Contributions to Geological Survey of Canada Programs-2013-2014

A.N. Rencz

2014

Canada

GEOLOGICAL SURVEY OF CANADA
OPEN FILE 7712

Science Laboratory Network: Science Contributions to Geological Survey of Canada Programs-2013-2014

A.N. Rencz

2014

doi:10.4095/295587

This publication is available for free download through GEOSCAN (***http://geoscan.nrcan.gc.ca/***).

Recommended citation
Rencz, A.N., 2014. Science Laboratory Network: Science Contributions to GSC Programs-2013-2014; Geological Survey of Canada, Open File 7712, 61 p. doi:10.4095/295587

Publications in this series have not been edited; they are released as submitted by the author.

Science Laboratory Network (SLN): Contributing to GSC Programs- 2013-2014

Executive Summary

The Science Laboratory Network (SLN) is the network of laboratories across the Geological Survey of Canada (GSC) that provides innovative and specialized laboratory results in support of GSC programs. The Network was established in 2006 and now comprises five lab groups involving over 70 staff members across all GSC offices. Each group has a unique strategic direction and a capacity to carry out that mandate.

The majority of the work is done through lab agreements which are collaborative projects between a lab proponent and a project proponent in the GSC. In 2013-14 the laboratories undertook 131 agreements in support of all programs; over 85% of work was done in the GEM, PSG, TGI, GNES or EGP programs. In 2013-14 there were also 32 VNR agreements (Vote Net Revenue) which are agreements with external partners in support of mutually advantageous projects.

The laboratories support the program agenda by providing innovative research and supplying high quality results where the capacity is not available in commercial laboratories. Each laboratory has a specific expertise, with highly qualified personnel, as well as specialised instruments. The laboratories have produced a range of accomplishments and scientific achievements which are highlighted in this report.

Andy Rencz

SLN Coordinator

List of Abbreviations

CCD	Central Canada Division
CNGO	Canada-Nunavut Geoscience Office
CQG	Centre Geoscientifique de Québec
EGP	Environmental Geoscience Program
ESS	Earth Science Sector
FY	Fiscal Year
GEM	Geomapping for Energy and Minerals
GGP	Groundwater Geoscience Program
GNES	Geoscience for New Energy Supply
GSC	Geological Survey of Canada
GSC-A	Geological Survey of Canada - Atlantic
GSC-C	Geological Survey of Canada - Calgary
GSC-P	Geological Survey of Canada - Pacific
GSC-Q	Geological Survey of Canada - Quebec
IGGG	Isotope Geochemistry & Geochronology Lab Group
IGRL	Inorganic Geochemistry Research Lab Group
INRS	Institut national de la recherche scientifique
LSA	Laboratory Study Agreement
LSI	Light Stable Isotope
MOU	Memoranda of Understanding
MPP	Mineralogy and Physical Properties Lab Group
NCD	Northern Canada Division
NRCan	Natural Resources Canada
OGPet	Organic Geochemistry and Petrology Lab Group
PAA	Program Activity Architecture

Paleo Paleontology Lab Group

PSG Public Safety Geoscience

QA/QC Quality assessment / Quality control

SHRIMP Sensitive High Resolution Ion Microprobe

SLN Science Laboratory Network

TGI Targeted Geoscience Initiative

UNCLOS United Nations Convention on the Law of the Sea

VNR Vote Net Revenue

………..

TABLE OF CONTENTS

1. INTRODUCTION

Laboratories have always been an integral part of geological surveys around the world by providing basic data required in projects and by driving new ideas through innovative technologies to test hypotheses. In the Geologic Survey of Canada (GSC) there are a number of scientific laboratories in disciplines such as geochemistry, geochronology, paleontology, petrology, mineralogy, geophysics, and sedimentology. Prior to 2006, the laboratories at the GSC developed independently and each lab "serviced" the specific needs of the Division in which they were located. In 2006, the Science Laboratory Network (SLN) was created by bringing together most laboratories across the GSC to create one cohesive unit. Together these laboratories would most effectively provide the expertise required across the GSC.

The purpose of the report is to provide the long term strategy, objectives, structure and contributions of the Science Laboratory Network as well as review the activities and accomplishments of SLN in 2013-2014. A compendium report provides a description of the capacity and expertise across SLN (Rencz et al. 2014).

1.1 Science Laboratory Network: History and Mandate

The Science Laboratory Network (SLN) was created in 2006 to provide a horizontal, pan-divisional structure for laboratories that would support all programs across the Geological Survey of Canada (GSC). SLN operates by organizing and directing the activities of laboratory scientists and support staff, in a manner analogous to Programs. Prior to SLN there were a number of laboratories that provided services to GSC scientists. They were managed by the particular Division in which they resided and primarily worked on projects within that Division. It was the responsibility of the Division to provide adequate funding.

In 2006 the majority of GSC labs were integrated into the SLN. The intent was to foster networking between the labs and thereby increase effectiveness and efficiency. Another goal of the SLN was to encourage innovation, while recognizing that certain activities cannot be obtained from external sources. Although the people and capital remained the responsibility of the Divisions, the scope of the work would be determined by SLN.

Study agreements, which are "negotiated" arrangements between a lab and project proponent became the working model for the labs. An important component of this arrangement was that the agreements should focus on innovative research. Service type arrangements would be contracted out unless there was a good reason not to. Funding for the labs was included as part of the agreements. Therefore Divisions were no longer responsible, as all operating funds would come from the programs. The goal of this arrangement is that the labs would become more self-sustaining and provide direct contributions to the program structure.

1.2 Organization of SLN

SLN is comprised of the research and technical staff that work within the laboratories across the GSC. The outline of the organizational structure is presented in Figure 1.2-1

Figure 1.2-1: The general overview of the organizational structure of the Science Laboratory Network

Overall, there is a *Coordinator* of the laboratories who is responsible for leading, managing and reporting on all activities in SLN. This position reports to the Director of the Central Canada Division (Bernard Vigneault) who in turn reports to the Director General of the Central and Northern Canada Branch (Donna Kirkwood). As established in 2006 there are five laboratory groups, each of which represents a broad thematic range of activities as identified in Table 1. These are managed by *Lab Leaders* who provide the scientific direction and supervision for the work in each Group, as well as support interactions between the various Groups. Each of the five groups is divided into lab facilities (Table 1) which undertake a range of analyses aimed at the overall strategy of the lab group. The final organizational level is the *Laboratory*, which is the working level that focuses on one or several types of analyses and typically has a specific instrument that provides those results. The laboratory functions within each of the five lab groups are described in a separate document to be published in the fall of 2014.

Table 1 - SLN Lab Group and Facilities with contact person

Lab Group/Lab Facility		Contact
Inorganic Geochemistry and Research Laboratory		Simon Jackson
1.1	Environment and Surficial (Ottawa)	Paul Gammon
1.2	Analytical Chemistry (Ottawa)	Simon Jackson
1.3	Marine Geochemistry (Dartmouth)	Michael Parsons
1.4	Labo-CGQ INRS (Quebec)	Serge Paradis
Paleontology		Sandy McCracken
2.1	Paleontology (Calgary)	Sandy McCracken
2.2	Conodont (Vancouver)	Mike Orchard
2.3	Macrofossil (Vancouver)	Jim Haggart
2.4	Palynology (Quebec)	Esther Asselin
2.5	Palynology (Dartmouth)	Rob Fensome
Mineralogy and Physical Properties		Jeanne Percival
3.1	Mineralogy (Ottawa)	Jeanne Percival
3.2	Marine Core and Sedimentology (Dartmouth)	Kimberly Jenner
3.3	Sedimentology (Ottawa)	Shauna Madore
3.4	Paleomagnetism & Petrophysics (Sidney)	Randy Enkin
Isotopic Geochemistry & Geochronology		Bill Davis
4.1	Radiogenic Isotope Tracer (Ottawa)	Bill Davis
4.2	Light Stable Isotope (Ottawa)	Bruce Taylor
4.3	Delta (Quebec)	Martine Savard
Organic Geochemistry & Petrology		Andy Mort
5.1	Organic Geochemistry (Calgary)	Andy Mort
5.2	Organic Petrology (Calgary)	Hamed Sanei

5.3	Organic Petrology (Dartmouth)	Vacant

There are 71 people within GSC that contribute to SLN. The list of these people in the various lab groups is provided in Appendix 1.

1.3 STRATEGIC DIRECTIONS:

Overall the strategy of SLN follows from the mission statement for GSC:

"The Geological Survey of Canada provides public geoscience knowledge to inform decision-making for internationally competitive mineral and energy sectors, for effective environmental stewardship and wise land use, and for the safety and security of Canadians."

In support of this mission the laboratories provide fundamental and innovative information to support program delivery at the GSC. In particular, the laboratories within SLN focus on the first two components of the mission statement, which focuses on the knowledge and development to enhance the mineral and energy sectors and for environmental stewardship. *This is accomplished through a network of expertise and state of the art laboratory equipment in each of the six divisions across GSC.*

Thus the strategic direction for SLN is:

1: Provide high-value laboratory research support for all PAA Programs– particularly innovative and specialized laboratory analysis and data interpretation.

The primary role of the SLN is to support research in GSC programs by providing innovative and specialized laboratory analyses in collaboration with researchers. It is not the role of the labs to provide routine analyses that is readily provided by external laboratories; however the labs, where possible, should provide an oversight role notably in QA/QC to ensure a corporate scientific integrity to the data.

2: Create collaborations with the Canadian geoscience laboratory network- including strategic collaboration with academia to leverage our resources.

The work in the laboratories would be enhanced through collaboration with external organizations that will take advantage of the expertise and resources in each organization. In this way resources can be focused on a specific problem to enhance scientific excellence.

3: Provide competitive advantage to Canadian Government and Industry by timely access to innovative analytical results as well as transfer of innovative technologies to industry.

In support GSC@ 175 the strategy fosters the development of open data geoscience through sound stewardship of laboratory data.

The basis for an effective laboratory network in a research environment is the development of innovative technologies and methodologies. These will be communicated to OGD's and industry to facilitate their uptake.

As a network, the SLN is well positioned to provide effective analytical leadership to all the programs. Decisions on research direction, capital acquisition and human resource needs are made based on collective input. This way the network facilitates an efficient allocation of resources as there is no duplication between the labs. Collaborations with other laboratories across Canada, particularly in academia, and with a limited number of international partners, are in place to enhance the scientific excellence of the laboratories.

Each of the five lab groups has a ***strategic direction*** and a set of objectives that identifies the areas of scientific focus that are required to support the Programs direction. These activities are unique for each group and in total the network of laboratories is designed to support all the programs. These are revisited on an annual basis. However, they have not changed substantially over the past few years.

1.3.1 INORGANIC GEOCHEMISTRY RESEARCH LABORATORY GROUP: IGRL

Characterize the inorganic geochemistry of geologic materials by developing innovative methods for indicators in difficult–to-analyze material. In support of defining geochemical processes in ore forming bodies as well as identifying the movement and impact of elements in environment's under development

Objectives:

- Develop and implement laser-ablation-ICP-MS based trace element analysis of minerals and inclusions. Particularly to ultra-low concentration levels.
- Develop and implement high precision ***in situ*** and whole-rock isotopic determination of previously unattainable isotopic ratios for many elements via MC-ICP-MS.
- Improve accuracy and utility of field-based elemental and speciation analysis.
- Conduct biogeochemical pilot studies to quantify biogeochemical processes and controls in environmental systems.
- Sequential extraction analyses for exploration; non-routine analyses of samples with difficult matrices where commercial analytical protocols are inappropriate for low-level detection limits (e.g. high sulphide ores, mine wastes, snow, marine sediment pore waters, C-rich shales).

1.3.2 PALEONTOLOGY LABORATORY GROUP: PALEO

Identification of fossils to provide the geological age and depositional setting for ancient sedimentary environments. In support of basin analyses to develop exploration models for hydrocarbons and minerals.

Objectives:

- Refining the biostratigraphic and geological age framework of sedimentary basins; and enhancing the understanding of the hydrocarbon potential of basins, time/space distribution of host rocks for energy and mineral resources and aquifers.
- Interpreting the depositional environment, paleoclimate, provincialism, and paleogeography of sedimentary strata.
- Discovering the thermal histories of rocks as related to hydrocarbon generation, maturation, and mineralization.

1.3.3 Mineralogy and Physical Properties: MPP

The laboratories address a range of issues to resolve geophysical, geotechnical, sedimentological, stratigraphic and mineralogical problems. Some routine analyses are contracted out, except where the additional transport of samples to external labs would jeopardize sample integrity; analyses requiring continuous interaction with project scientists are carried out in-house. MPP lab staff collaborates on a regular basis with scientists to jointly develop new techniques and innovation in, but not restricted to, the following areas.

Objectives:

- Non–destructive mineralogical analyses through quantitative analysis and elemental composition at micro and nano scales; development of field-based methods.
- Integrated measurement of magnetic, electrical and other physical rock properties data. These link geological mapping and geophysical surveys for advanced mineral deposit models, mostly for GEM and TGI Programs.
- Continuous physical property, geotechnical and geochemical characterization of unconsolidated cores to provide a stratigraphic framework within which geohazard events can be placed. This data is critical to the identification and characterization of offshore failure events under the Public Safety Geoscience Program.
- Physical characterization of unconsolidated materials through textural analysis and grain characterization to constrain both surficial and down-core geological processes.
- Sample preparation for external laboratory analyses incorporating rigorous QA/QC protocols.

1.3.4 Isotope Geochemistry and Geochronology Laboratory Group: IGGG

Provides expertise and method development on radiogenic and stable isotope-based analysis for tracing crustal, environmental and marine processes, and for dating geologic events. In support of regional geology studies, ore deposit modelling and environmental stewardship.

Objectives:

- High-precision geochronology to calibrate, test and refine geological and metallogenic models for resource assessment.
- In situ, micron-scale isotopic analytical techniques as chronometers and tracers of complex geological processes.
- Thermochronological research into quantitative modelling of low-temperature thermal histories for basin and tectonic analyses.
- R&D for producing new methods for stable isotope analyses used in understanding environmental and hydrological processes, fingerprinting sources of contamination, and assessing and dating past perturbations of light element cycles.
- Micro-analytical and in situ stable isotope analysis models of fluid/rock interaction, mineralization processes, and genesis of sedimentary and crystalline rocks.

1.3.5 Organic Geochemistry and Petrology: OGPet

Geochemical and petrographic characterization of conventional and unconventional petroleum systems. Environmental geochemical characterization of areas of potential resource extraction & development. In support of unconventional reservoir characterization and hydrocarbon resource assessment.

Objectives:

- Method development for improved characterization of petroleum systems. Expanding the established focus on transformation of kerogen to crude oil via bitumen.
- Research and develop new analytical methods for unconventional energy systems with a focus on self-sourced hydrocarbon reservoirs.
- Characterisation of how organic compounds concentrate and speciate anthropogenic-derived metals and non-metals in the environment.

2 SLN- Program Interaction: contributing to the PAA

All activities within SLN are conducted through agreements, either internal Laboratory Study Agreements (LSA) or with external partners through vote net revenue (VNR) agreements. The activity must support a program objective as well as support the strategic direction of the lab. Funding for the lab is derived exclusively from these agreements and therefore Divisions are no longer responsible for operating funds. It was the hope of this arrangement that the labs would become more self-sustaining and provide direct contributions to the program structure.

Overall, in 2013-14 there were 131 lab study agreements (LSA) and 32 vote net revenue agreements (VNR) with combined revenue of 857 thousand dollars (Table 2).

Table 2 - Revenues for SLN in FY 2013-14

SLN Revenue	Number of Agreements	FY 2013-2014
LSA	131	$ 661,352.03
VNR	32	$ 195,723.70
Total	163	$ 857,075.73

2.1 Laboratory Study Agreements

Study agreements, which are "negotiated" arrangements between a lab and project proponent are the working model for the labs. LSA's are laboratory based research activities developed by a project proponent who is working on an approved GSC science program and a lab proponent who provides access to the laboratory resources. An important component of this arrangement was that the agreements should focus on innovative research. Service type arrangements would be contracted out unless there was a good reason not to.

There is an on-line tool for developing and managing study agreements. The tool requires a description of the project including a detailed accounting of the analyses that is required. The system produces a searchable data base of all approved projects as well as the financial details for the project. The tool is available to all staff of GSC through the following link (***http://sln.nrcan.gc.ca/docs/index-eng.html***).

In the period since 2006 there have been over 900 agreements with 2012-2013 representing the greatest number of agreements- 166.

Table 3 - Laboratory Study Agreement form 2006-07 to 2013-14

SLN / Years	2006-07	2007-08	2008-09	2009-10	2010-11	2011-12	2012-13	2013-14
Amount in K	756	757	1,150	1,185	1,003	951	1,104	661
Number of Agreement	125	102	135	133	127	145	166	131

The majority of funding in 2013-2014 came from two programs (TGI-40% and EGP-30%). The first is a C-Based program (TGI4) that responds to GSC's role in the development of natural resources, while the EGP responds to the environmental stewardship aspect of resource development. Thus these two programs account for 70% of the revenues within SLN. Although the contribution to other programs is less; SLN contributes to all of the scientific programs across GSC (Figure 2.1-1). In addition, the laboratories contribute to INRS through an MOU and there are several agreements with the Canada-Nunavut Geoscience Office, Geo-mapping for Energy and Minerals, Geoscience for New Energy Supply, and Public Safety Geohazard.

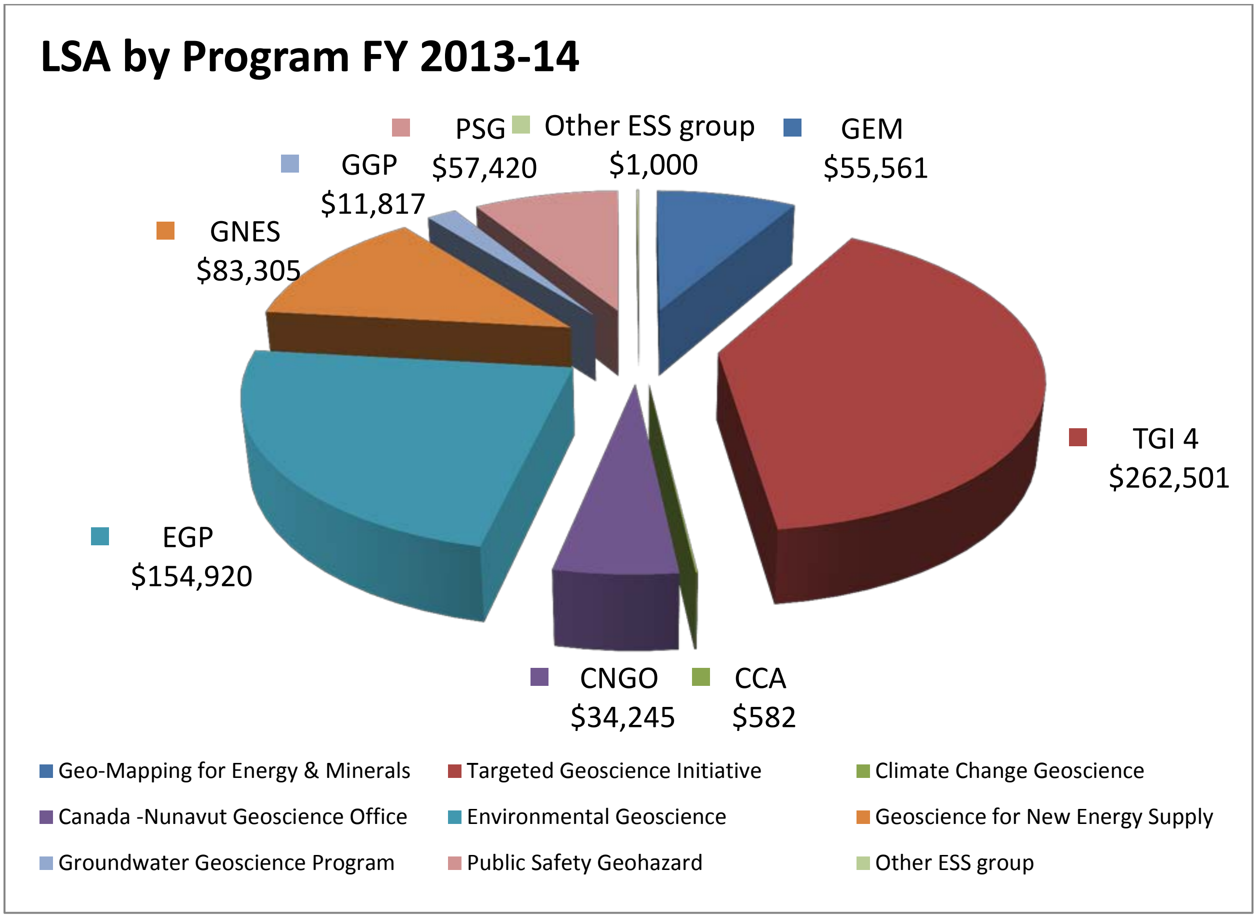

Figure 2.1-1 Contribution from GSC programs to Laboratory study agreement in FY 2013-14

The five lab groups have different capacities and therefore respond to different programs. The Inorganic Geochemistry Research Lab Group (IGRL) provides geochemical research to the TGI and EGP program. The Paleontology Lab (Paleo) works almost exclusively under the GEM program and within that program mainly on the hydrocarbon component. The Mineralogy and Physical Properties Group (MPP) contributes to most programs with a concentration in the GEM (both energy and minerals components) and the Public Safety Program. The Isotope Geochemistry and Geochronology Laboratory Group (IGGG) is predominantly focused on the GEM program. However the LSI facility within the group is aligned with TGI and the DeltaLab Facility is mainly focused on the Environmental Program including groundwater. The OGPet group works mainly on GEM activities, typically in hydrocarbons, however the group also contributes to EGP GNES programs.

2.2 Vote Net Revenue

Vote net revenue agreements are opportunities to work with external partners when the project contributes to the goals of SLN and the services are not readily available in the private sector.

Table 4 - Vote Net Revenue in SLN from 2010-11 to 2013-14

VNR/ Year	FY 2010-11	FY 2011-12	FY 2012-13	FY 2013-14
# of VNR	42	31	19	32
Amount	$ 88,234.00	$ 161,932.65	$ 79,892.96	$ 195,723.70

The amount of funding is significantly less than the amount from LSA's. In 2013-14 the amount of funding was 195K. As noted in each lab received some funding from VNR. The OGPet group accounted for 45% of the total revenue with most of this coming from characterization of organic natter using pyrolysis equipment in the Calgary Lab.

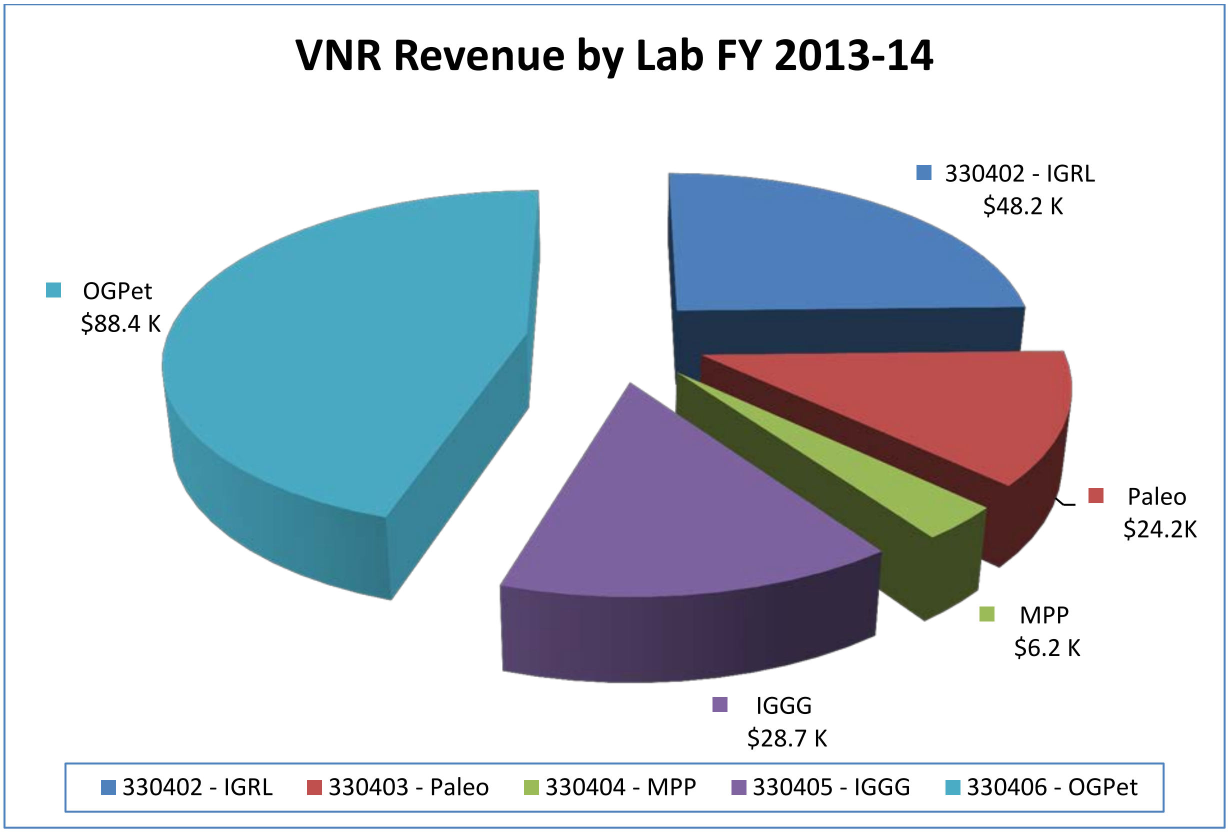

Figure 2.2-1: Vote Net Venue for SLN in 2013-2014.

3 SLN CONTRIBUTIONS

The focus of the laboratories, as noted in the Strategy for SLN- 1.3, is to provide fundamental and innovative information to advance the programs in GSC. This section will review the accomplishments which identify the overall productivity of the group as well as highlight scientific achievements which describe several of the notable scientific contributions from the various laboratories in 2013-2014.

3.1 INORGANIC GEOCHEMISTRY RESEARCH LABORATORY GROUP: CONTRIBUTIONS

In 2013-2014, the IGRL contributed to 56 laboratory study agreements with GSC scientists and 16 agreements with external scientists (mostly academic institutions with students working on GSC-funded projects). In conducting these analytical projects, the group provided over 18,000 elemental and isotopic analyses (not inclusive of analyses performed at INRS) (see Table below for breakdown). The analyses included data for most of the elements in the periodic table and isotopic analyses for 6 different heavy stable isotopic systems. Much of this work is non-routine and involved development of procedures prior to generating the data required by Program scientists to answer their specific questions. Examples of these developments are described under IGRL Scientific Achievements.

3.1.1 IGRL ACCOMPLISHMENTS:

The IGRL produced a number of major accomplishments in 2013-2014. These include:

Lab development:

- Rejuvenation of GSC-A Marine Geochemistry Laboratory with hiring of Lori Campbell in January 2013 to operate the CHNS and Hg analyzers.
- Completed development of procedures on Mg isotope and Te isotope analysis:

Fornadel, A.P., Spry, P.G., Jackson, S.E., Mathur, R.D., Chapman, J.B., and Girard, I., 2014, Methods for the determination of stable Te isotopes of minerals in the system Au–Ag–Te by MC-ICP-MS: J. Analytical Atomic Spectrometry, v. 29, p. 623-637.

- Development of procedures for Zn isotope analysis of tree-ring samples;
- Development of procedures for Mo isotope analysis;
- Development of improved procedures for in situ trace-level PGE analysis and mapping using LA-ICP-MS with collision cell technology;
- Development of procedures for in situ Pb isotope mapping.

Lab Throughput:

- Completed geochemical analysis for a number of elements and isotopes:

Litho-Geochemistry Laboratories					
LA-ICP-MS facility	Clients GSC	Spot/line analyses[1]	Clients external[2]	Spot/line analyses[1]	Total analyses
Spot analysis	11	3,652	14	3,640	7292
Element mapping	5	3,940	1	288	4228
Method development	3	207	1	326	533
Totals		7,799		4,254	12,053
MC-ICP-MS facility	Clients GSC	Days	Clients external	Days	Total analyses[3]
Mg isotopes	1	6			
Fe isotopes	3	42	1	12	
Ni isotopes	1	10			
Cu isotopes[4]	3	8			
Zn isotopes	1	33			
Te isotopes			1	15	
Method development (Fe, Zn, Mo)		12			
Troubleshooting and maintenance		29			
Totals		140		27	1,670
Environmental Geochemistry Laboratories					
	Clients GSC	Analyses	Clients external	Analyses	Total analyses
Anions	7	670			670
ICP-MS	11	1,514			1514

ICP-ES	10	1,082			1082
Dissolved organic carbon	4	125			125
Alkalinity	4	125			125
Pb isotopes	3	145			145
Totals		3,801			3,801
Marine Geochemistry Laboratories					
	Clients GSC	Analyses	Clients external	Analyses	Total analyses
Hg analysis		500			500
C analysis		200			200
Totals		700			700

Table 5 - Analyses performed in IGRL, April 1st 2013 to March 15th 2014

Notes:

1: Each line of an element map counted as one analysis

2: External clients include mostly students working on GSC-funded projects, and visiting scientists.

3: Total MC-ICP-MS analyses calculated assuming approximately 10 samples analysed per day.

4: Includes solution and in situ analyses.

3.1.2 IGRL: Scientific Achievements

3.1.2.1 Low – Level solid phase mercury analyses

Michael Parsons & Bill LeBlanc, Marine Geochemistry Research Facility, Dartmouth

Focus Areas:

Analyses of background-level Hg concentrations in low-mass solid samples

Contribution to program:

Problem**:** To establish "background" Hg concentrations to understand fluxes from geogenic and anthropogenic sources.

Solution**:** Measurements of low levels of Hg in samples weighing less than 100 mg have recently been made on a wide range of samples from projects in the Environmental Geoscience and

GEM programs. These analyses suggest that increased Hg fluxes may have contributed to widespread extinctions during the Permo–Triassic Biotic Crises (Grasby et al. 2013). Mercury data from our lab have also been used to understand the impacts of industrial emissions in the Athabasca oil sands region (Neville et al. 2013) and are currently being used to assess the effects of climate change in the North.

Achievement:

Development of new calibration procedures to accurately and reproducibly measure Hg concentrations down to 5 ppb in samples of sediment, soil, and rock weighing as little as 50 mg (dry weight). This capability is important for various ESS programs where only small sample sizes are available (e.g. Arctic lake sediments sampled at millimetre-scale resolution to assess recent changes in natural and anthropogenic Hg fluxes). Most commercial laboratories cannot provide accurate Hg results for samples that are less than 500-1000 mg dry weight. The Leco AMA-254 can produce quantitative Hg analyses in less than 8 minutes/sample via thermal decomposition, and requires no sample pre-treatment or use of hazardous chemicals.

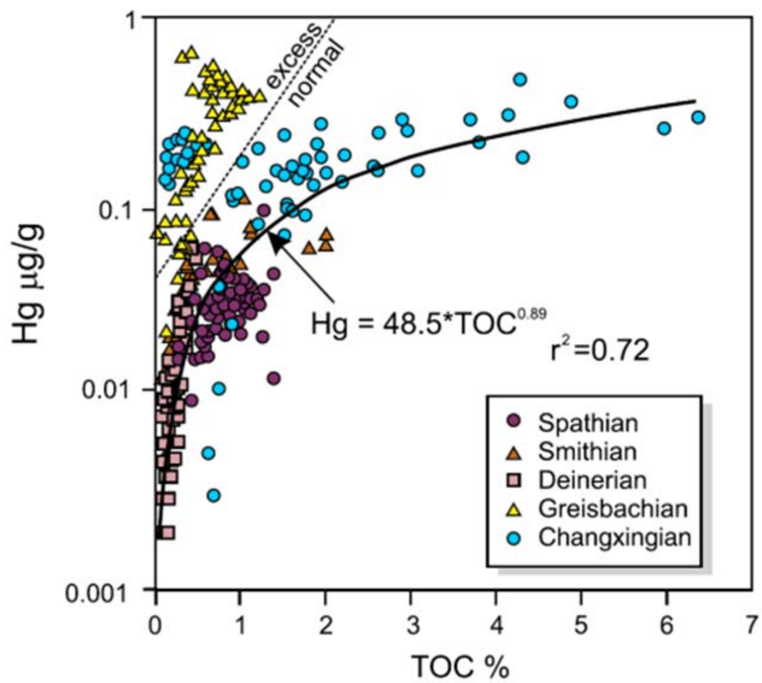

Figure 3.1.2.1-1: Relationship between TOC and Hg

Publication:

Grasby, S.E., Sanei, H., Beauchamp, B., Chen, Z. (2013) Mercury deposition through the Permo–Triassic Biotic Crisis. Chemical Geology, v. 351, pp. 209-216.

Neville, L.A., Patterson, R.T., Gammon, P., Macumber, A.L. (2013) Relationship between ecological indicators (Arcellacea), total mercury concentrations and grain size in lakes within the

Athabasca oil sands region, Alberta. Environmental Earth Sciences, DOI 10.1007/s12665-013-2979-6.

3.1.2.2 Title: Environmental monitoring of Athabasca Oil Sands using tree-ring chemistry

Laureanne Dinis, Martine Savard and Paul Gammon, Environmental Geochemistry Research Facility, Ottawa

Focus Area:
Development of procedures for ultra-low level, digestion and isotopic analysis.

Contribution to Program

Problem: The Athabasca oil sands industry is one of the most economically important hydrocarbon resources in Canada. However, there are no baseline data to determine either the source of hazardous metals emitted by the industrial operations, the distribution of hazardous materials emitted by the industry, or the environmental impact of hazardous metals emitted by the industrial operations. The Environmental Geoscience Program – CORES Project aim is to provide new geochemical methods for tracing and quantifying industrial emissions within the Athabasca Oil Sands Region. This study looked into a new method for fingerprinting Zn emissions from the industrial operations using Zn and Pb isotopic and concentration data in spruce tree rings. The very low Pb and Zn concentrations in tree rings made this particularly challenging research.

Achievement:
1) Tracking down and eliminating all potential sources of contamination to provide accurate Zn and Pb isotopic analyses; 2) Generating a new analytical protocol that enabled the analysis of large sample sizes to overcome the low concentrations – this included compositing coeval wood from multiple trees; ashing of large wood samples; transfer of ash to beakers for digestion using HF-HNO_3; 3) Developing ion-exchange chromatography to isolate and concentrate Zn; 4) Developing mass spectrometry techniques to measure the Zn stable isotopic ratios using standard bracketing.
Overall the achievement was the development of procedures for sampling and ultra-low level, digestion and isotopic analysis (Pb and Zn) of tree rings from the Athabasca Oil Sands operation.

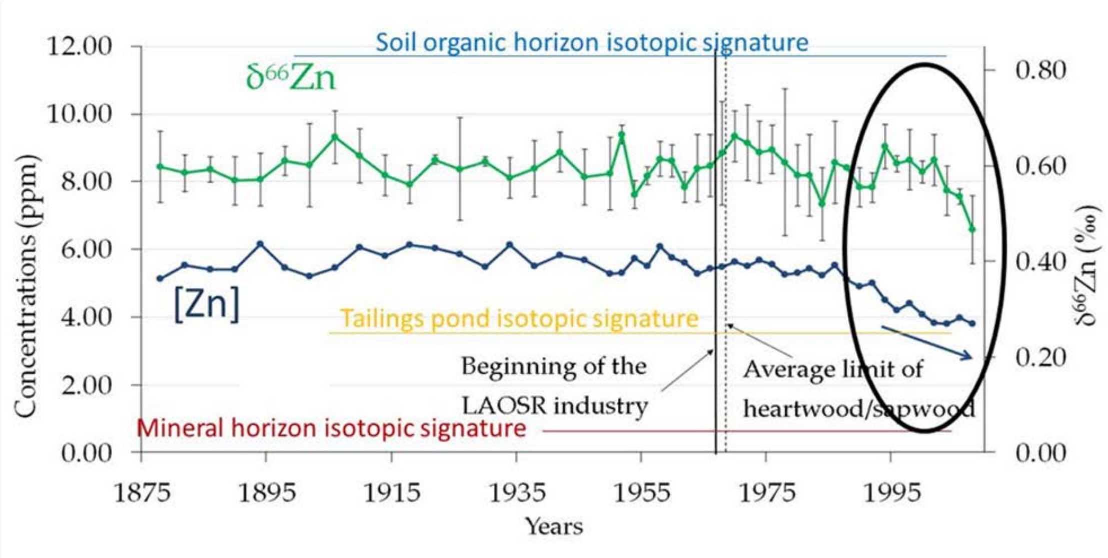

Figure 3.1.2.2-1 Zn isotopic analyses for this site, which is ~25 km downwind of the main mining operations. This is the first tree-ring Zn isotopic study. The last 20 years of tree ring data show a pronounced decrease in Zn isotopic signatures that indicate there is likely an influence from tailings pond material. This agrees with other evidence that indicates "fugitive dust" blowing off tailing ponds operations are probably the most important source of metal contamination in the area. It also corresponds with the ramp-up in oil sands operations. The data provide a baseline for pre-industrial times. Note the low Zn concentrations: Zn is a micronutrient for trees and 50 ppm is considered a Zn-deficient tree. These trees contain the lowest Zn concentrations ever reported, let alone ever analysed for isotopic signatures. Such low background sites are ideal for monitoring the impact of industrial emissions, but in this case came with significant analytical challenges. The decrease in Zn concentrations that is coincident with the isotopic decrease may be due to tree stress.

Publications:

Dinis L., Savard M.M., Gammon P., Bégin C., and Vaive J. (submitted). Influence of climate and industrial emissions on spruce tree-ring Pb isotopes analyzed at ppb concentrations in the Athabasca oil sands region. Submitted to Science of the Total Environment.

Dinis L., Savard M.M., Gammon P., Bégin C., Girard, I., and Vaive J. (In Prep.). Tree-ring sampling and treatment methods to evaluate Zn isotopes in the Athabasca oil sands region: Inferring climate and anthropogenic influences

3.1.2.3 Title: LA-ICP-MS element mapping

Simon Jackson and Zhaoping Yang, Lithogeochemical Research Lab

Focus Area:
Laser Ablation ICP-MS – in situ mineral-scale trace element (ppb) and isotopic analysis and mapping

Contribution to Program

Problem**:** What is the source of Au in orogenic deposits?

Approach: Test case application of LA-ICP-MS element and isotopic mapping to the Musselwhite orogenic Au deposit. An example of the great potential of the technique is an evaluation of nodular diagenetic pyrite as a source of the gold at the Musselwhite deposit, NW Ontario (B. Dubé, TGI-4).

The Musselwhite gold deposit is located within the North Caribou Greenstone Belt, which is dominated by mafic volcanics, banded-iron formation, felsic volcanics, siliciclastic sediments and local argillaceous units bearing diagenetic pyrite nodules ranging in size up to > 1 cm in diameter. All units in the immediate mine area have been metamorphosed to amphibolite facies.

Achievement:

LA-ICP-MS element mapping of nodular and recrystallized framboidal pyrite from the Musselwhite area (Figure 3.1.2.3-1) reveals significant gold and other metal enrichments in the diagenetic pyrite nodule, while surrounding recrystallized pyrite and pyrrhotite are barren of most elements. Large et al. (2011) have recently proposed that metamorphic recrystallization of diagenetic pyrite with > 250 ppb of invisible gold has the potential to release sufficient gold to produce economic gold deposits. Our results offer diagenetic pyrite as a potential source of the Musselwhite gold; moreover the Pb isotopic composition the nodule is significantly different than its host sediment but agrees very closely with the Pb isotopic composition of the ore (2.407 ± 0.039). The data illustrate the potential of LA-ICP-MS element mapping in exploration for orogenic gold deposits in metamorphosed volcano-sedimentary terrains.

The element mapping technique has received rapid uptake by GSC Program and has been applied in a large number of GEM and TGI-4 funded projects, including GEM Edges and Great Bear and TGI-4 Lode Gold, VMS and Magmatic Ni-Cu-PGE-Cr.

Over the past three years, the IGRL has developed the world's first LA-ICP-MS data acquisition and processing protocols for generating high resolution (< 20 μm) 2-D trace element concentration maps of multi-mineral sample areas. Sub-ppm detection limits allow mapping of elements at concentrations 3-4 orders of magnitude lower than attainable using EPMA imaging. This technology allows the quantitative study of the micro-scale distribution of scarce but key elements including: ore (e.g., Au, PGE), pathfinder (e.g., As, Te) and process-sensitive (e.g., REE) elements formed during crystallisation, precipitation and leaching processes involved in, for example, mineralizing systems. More often than not, it reveals complex histories that are not otherwise evident and provides answers to questions on process timing, number of generations of a mineral, metal sources, etc.

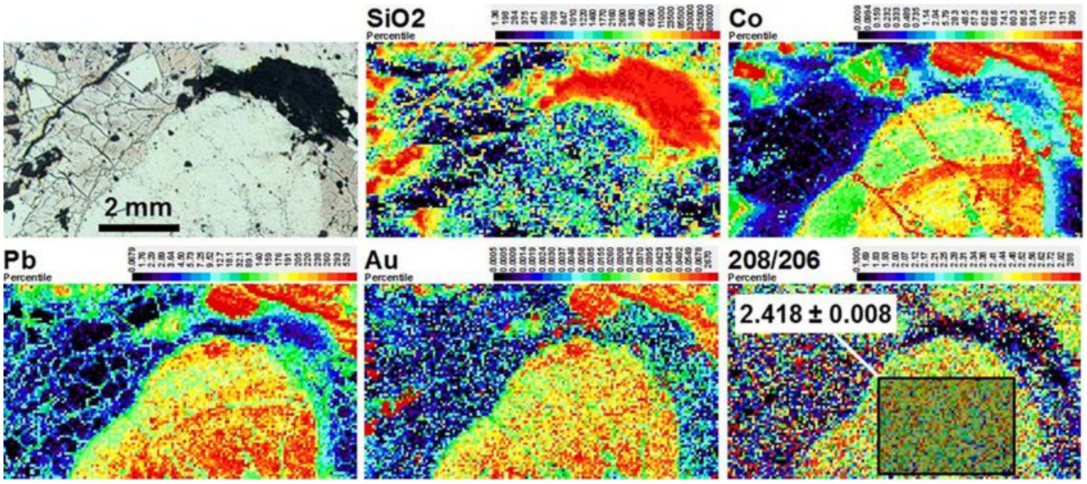

Figure 3.1.2.3-1 Element and Pb isotope maps for a pyrite nodule (centre) from the vicinity of the Musselwhite mine. Note the high concentrations of Au and Pb (and other trace elements) in the nodule compared to recrystallised pyrite (euhedral, white) and pyrrhotite (brown). Element scales are in ppm.

Publications:

Jackson, S., Dubé, B., Chapman,J., Montreuil, J.-F., Yang, Z., Applications of quantitative, in situ, plasma spectrochemistry in mineral exploration geochemistry: Plenary lecture, Winter Conference on Plasma Spectrochemistry, Amelia Island, Florida, January 2014.

Jackson, S.E., Dube, B., Chapman, J., and Gao-J-F, 2013, Applications of LA-ICP-MS element mapping in mineral deposit research and exploration: Invited Lecture, Mineral Deposit Research for a High-Tech World, 12th SGA Biennial Meeting, Proceedings, v. 1, p. 201-204, Uppsala, Sweden, August 2013.

Jackson, S., Gao J.-F., and Dubé, B., New developments in mineral-scale trace element analysis and mapping in ore deposit research and exploration: Examples from gold deposits of Québec and Ontario: Invited Lecture, Québec Mines, Québec City, November, 2013.

3.2 Paleontology Lab Group: Contributions

In 2013-2014, the PaleoLab contributed to 13 laboratory study agreements with GSC scientists mainly in the energy component of the GEM program. In addition the Lab Group undertook 16 agreements with external scientists (mostly academic institutions or provincial governments. funded projects). In conducting these analytical projects, the group processed over 670 samples and over 87 reports as per below. The material is maintained in collections contributing to a growing inventory of collections (over 100,000 collections representing over 300,000 species.) As well the reports contribute to the catalogue of over 8900 Paleontological reports. The analyses are highly specialized and typically support basin analysis that could not have been completed without the lab information. . Several of the highlights are noted under Scientific Achievements.

3.2.1 *Paleontology Lab ("PaleoLab") Accomplishments:*

Lab Throughput

- Project samples (670) – Conodonts (81), Palynology (347 + 78 for GSC Atlantic), Foraminifers (144), Macrofossils (20)
- Non-project (74) – Geological Surveys (BC, NL, AB, NT, USGS), University of Washington, University of Calgary, University of Ottawa, University of North Carolina, University of Leeds, Tyrrell Museum, Aeon Consulting, Husky Energy, New Gold Incorporated
- Publications – 25 External, 3 Government, 26 Paleontology Reports, 33 Abstracts
- Program/Projects supported: Western Arctic, Yukon and Liard Basin, Eastern Arctic, Hudson Bay Basin-Fox Basin, Mackenzie Delta and Corridor (all GEM), Environmental, Arctic Islands Hydrocarbon, Shale Reservoir Characterization (PERD), GNES Shale Resource Assessment
- ~170 GSC Paleontology Reports for GEM 2008-2013
- Journal papers, many with multidisciplinary authorship on genus revisions, new taxa, taxonomy, biostratigraphy involving multiple phyla zonations, paleoenvironments, isotopes, bentonites, paleoclimates, stratigraphy, sedimentology, basin evolution, heavy metals, marine reptiles, lobsters
- Reports on conodonts from NL, ON, BC, NT and NU
- Reports on palynology from ON, AB, AK
- Reports on trace fossils and Burgess Shale-like fossils (NT)
- Reports on molluscs (BC, NU)
    - In addition the group continues to add to two paleontological data bases
    - Over 8900 Paleontological Reports since 1940s

- Collections represent an incalculable amount of operational and salary dollars
- Representing about 100,000 collections of millions of specimens and 300,000 species
- Searchable: country, province, NTS, age, stratigraphy, fossil type, paleoenvironment, maturity, author, title, report number, etc.
- Recent uses: GEM projects, BC Ministry of Agriculture & Lands, Yukon Geological Survey

3.2.2 *Scientific Achievements*

3.2.2.1: Revision of stratigraphy of potential source and reservoir strata, Eagle Plain, Yukon Territory

Jim Haggart, Macro-fossil Facility, Vancouver

Focus Area:

Refining of the stratigraphic and age framework of sedimentary basins, enhancing the understanding of the hydrocarbon potential of basins, time/space distribution of host rocks for energy and mineral resources and aquifers.

Achievement:

Haggart assembled a team of paleontologists and sedimentologists to assess the existing stratigraphic framework of potential oil- and gas-bearing Cretaceous strata of Eagle Plain basin, Yukon Territory. Marine ammonite and bivalve fossils, as well as non-marine bivalves, established that unconformities in the succession are of much lesser extent than previously assumed, and likely represent local events. Fossil dinoflagellates and foraminifera also helped to constrain the age of units, allowing more precise regional correlation with strata of northeast British Columbia, the Mackenzie Delta, the Alaska North Slope, and Arctic Canada. Dinoflagellate and pollen fossils further helped to establish the relative percentage of marine and non-marine facies present in the succession, aiding in the interpretation of changing sedimentary environments. A revised model of sedimentation in the region provides important new data on paleogeographic evolution of the foreland basin in Cretaceous time.

Contribution to Program:

A revised understanding of the stratigraphic succession and depositional setting of the Eagle Plain Cretaceous basin provides a more comprehensive insight into the development of the basin and its potential for hosting a petroleum system.

Publication:

Haggart, J.W., Bell, K.M., Schröder-Adams, C.J., Campbell, J.A., Mahoney, J.B., and Jackson, K. 2013. New biostratigraphic data from Cretaceous strata of the Eagle Plain region, northern Yukon: reassessment of age, regional stratigraphic relationships, and depositional controls. *Bulletin of Canadian Petroleum Geology*, v. *61 (2): 101-132.*

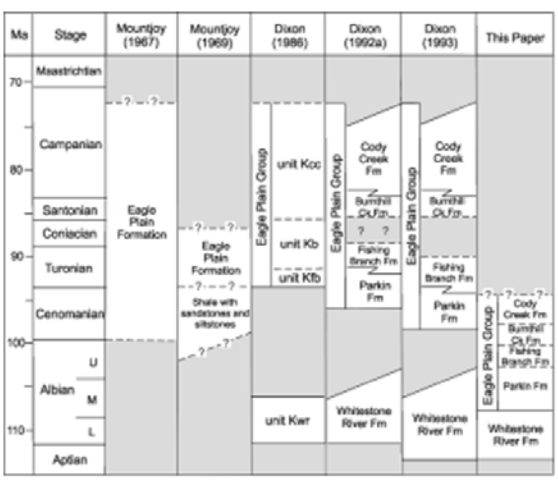

Figure 3.2.2.1-1 Changes in stratigraphic nomenclature and ages for Eagle Plain region, Yukon. Evolution in the interpretation since 1967 from one generalized formation ca. 72-99 Ma in age to present interpretation of an older group composed of several formations

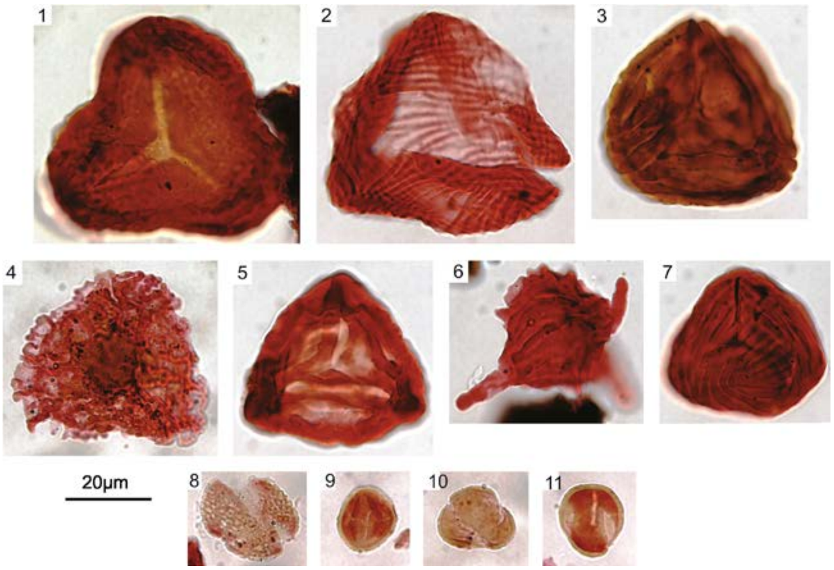

Figure 3.2.2.1-2 Palynomorphs from Eagle Plain Group, Yukon.

3.2.2.2: Quantitative palynostratigraphy of Mesozoic strata provides insight into ancient climates of the Canadian Arctic

Jennifer Galloway, Paleontology Facility, Calgary

Focus Area:

Refining the stratigraphic and age framework of sedimentary basins, enhancing the understanding of the hydrocarbon potential of basins through evaluation of depositional environments, and time/space distribution of host rocks for energy and mineral resources

Achievement:

The potential for detailed regional correlation and age determination is limited by the predominance of long-ranging fossil types. With GEM-1, we advanced the understanding of Meosozoic chronostratigraphy in Sverdrup Basin. This was by linking palynoassemblages to paleoclimate events that have time significance by integration with other microfossil groups and by using a variety of geological techniques. We used quantitative palynological analyses of outcrop collections of Mesozoic strata to produce a unique palynological signature for select stratigraphic units. This approach has refined the age control and depositional environment reconstruction in the Arctic and has provided a tool for statistically-based basin correlation. We built upon these successes by using material from an oil and gas well drilled near the depocentre of Sverdrup Basin on Ellef Ringnes Island to further our studies. Wells drilled for hydrocarbon exploration in the Arctic provide a unique opportunity to investigate continuous stratigraphic records that are not possible using outcrop studies alone. Using several statistical approaches, we defined palynoassemblages within a Late Jurassic-Early Cretaceous succession preserved in this Ellef Ringes well. Our results demonstrated that a large environmental shift occurred in the late Valangian/early Hauterivian when climate conditions changed from seasonally arid to cooler and humid. The resulting palynological assemblage change due to this significant climate event represents an important biogeochron can be used as a time marker for strata correlation in the Canadian Arctic and other high latitude regions.

Contribution to Program:

By applying novel statistics analyses to our palynostratigraphic data contained in samples collected from outcrop and the Ellef Ringes oil and gas well, we identified biogeochronological markers that refine our understanding of the age of important stratigraphic units in the Sverdrup Basin and provide a basis for basin correlation. The improved understanding of the geological evolution of the Sverdrup Basin is important because Mesozoic strata represent important hydrocarbon reservoirs in the Canadian Arctic and elsewhere.

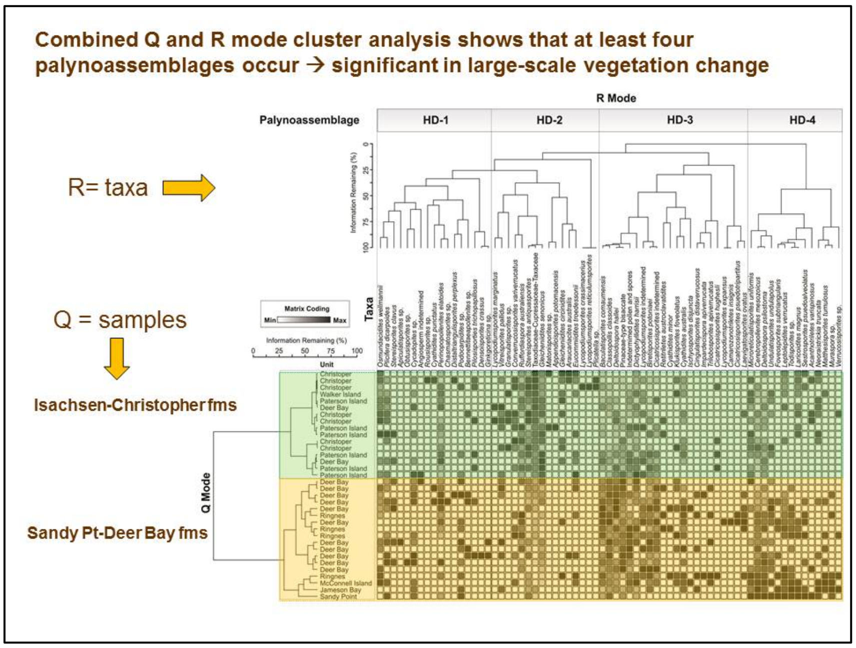

Figure 3.2.2.2-1 Cluster analysis of species versus stratigraphic occurrences from the Ellef Ringnes oil and gas well.

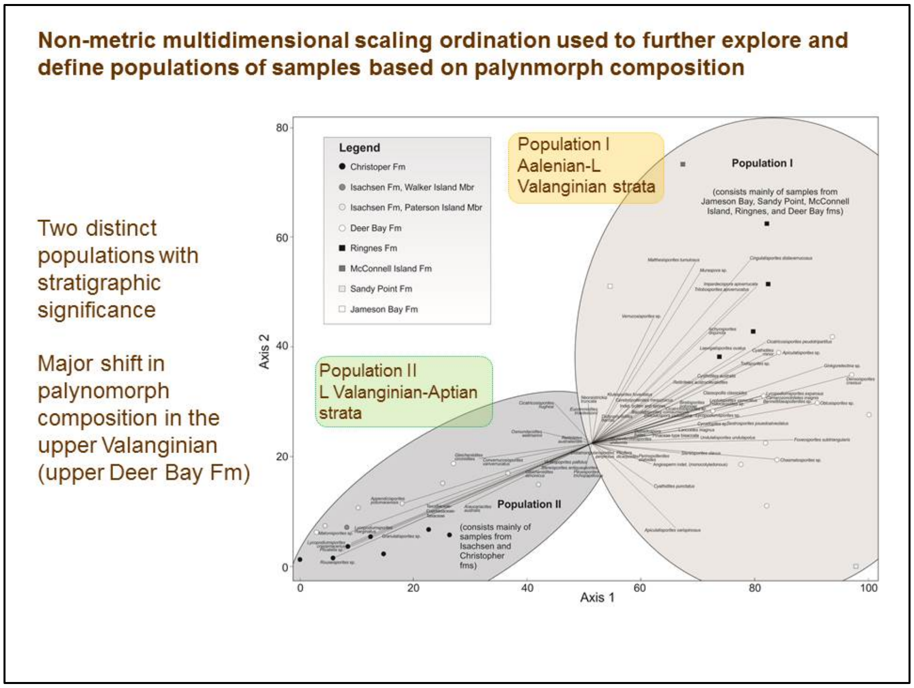

Figure 3.2.2.2-2 Statistical analyses of data from the Ellef Ringnes well show the major palynological assemblage change. This shift is a biogeochronological marker.

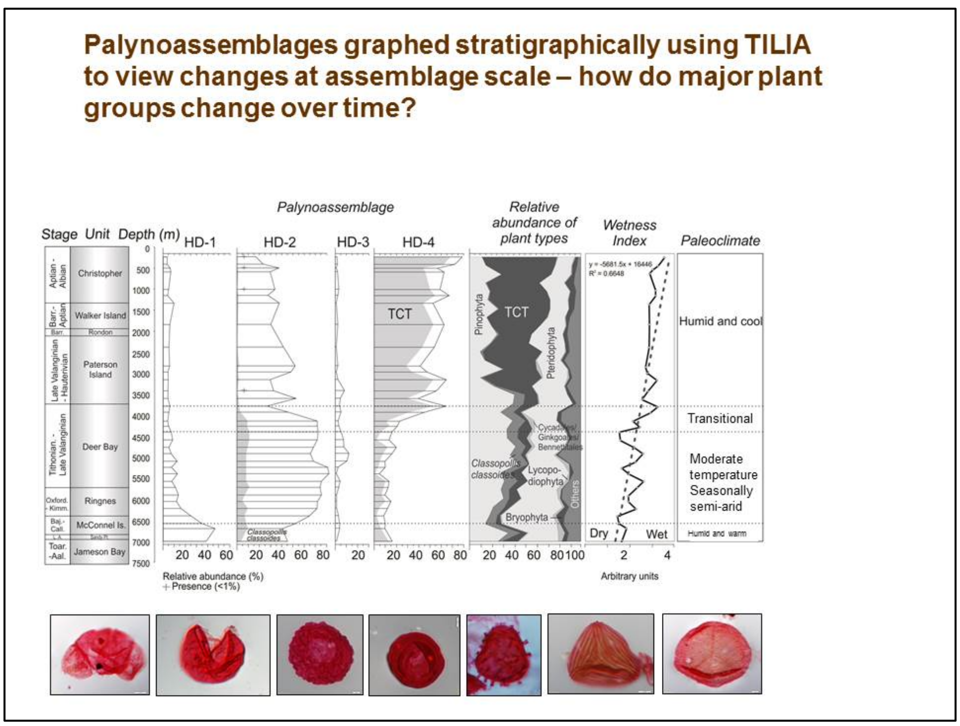

Figure 3.2.2.2-3 Stratigraphic palynoassemblage changes of pollen groups that occur in Late Jurassic-Early Cretaceous strata from the Ellef Ringnes well show that climate transitioned from seasonally semi-arid to cool and humid. This shift resulted in the expansion of upland conifers (TCT) at the expense of taxa that are able to thrive in dry conditions.

Publication:

Galloway, J.M., Sweet, A., Sanei, H., Dewing, K., Hadlari, T., Embry, A.F., Swindles, G.T. 2013. Middle Jurassic to Lower Cretaceous paleoclimate of Sverdrup Basin, Canadian Arctic Archipelago inferred from the palynostratigraphy. *Marine and Petroleum Geology, v. 44: 240-255.*

3.2.2.3: Improved sedimentary basin models through biostratigraphy
Sandy McCracken, Paleontology Facility, Calgary

Focus Area:

Samples collected by field geologists are studied by PaleoLab scientists to aid in the interpretation of sedimentary basins. During bedrock geological mapping, geologists assign rock outcrops to formations based on their physical characteristics (e.g., colour, rock type, weathering profile). In a mapping area there may be more than one formation with similar characteristics and outcrops can be misidentified; resulting in errors as the map is compiled. PaleoLab provides important data to avoid these misinterpretations.

Achievement:

The Northwest Territories Geoscience Office has undertaken new mapping in a region of the Mackenzie Mountains with relatively complex stratigraphy and structure. GEM-Energy supported the conodont study of rock samples from this field work. This confirmed some field identifications of formations but corrected others; including situations where misinterpretations of many millions of years had been made. Other samples came from outcrops that could not be identified at all in the field but the new fossil data suggest to what formation the rocks should be assigned. This results in more accurate maps that will contribute to understanding the regional context and evolution of the Mackenzie Corridor oil and gas exploration region.

Figure 3.2.2.3-1 Devonian conodonts (CAI=5) from the Marmot Formation, NWT.

Contribution to Program:

Correct stratigraphic interpretations are a requisite in sedimentary basin studies. Paleontology improves the accuracy of interpretations and lessens the risk in hydrocarbon (and mineral) exploration.

Publication:

McCracken, A.D. 2013. Report on 37 middle Ordovician through Late Devonian conodont samples from the Cloudy, Hailstone, Grizzly Bear, Arnica, Marmot, Duo Lake, Nahanni, Mount Kindle, Canol, and unknown formations, District of Mackenzie, NWT submitted under R.B. MacNaughton's Mackenzie Delta and Corridor: Mapping for Energy (MADACOR) Project in 2012. NTS 106B/01, 106B/05, 106B/06, 106B/07, 106B/10. Con. No. 1780. *Geolgical Survey of Canada Paleontological Report 5-ADM-2013.*

3.2.2.4: Improved age control on Mackenzie Delta stratigraphy
James White, Paleontology Facility, Calgary

Focus Area:

Refining the stratigraphic and age framework of sedimentary basins, and enhancing the understanding of the hydrocarbon potential of basins, time/space distribution of host rocks for energy, mineral resources and aquifers.

Achievement:

Reworking of fossil pollen is a classic biostratigraphic problem that can give rise to erroneous age determinations. The problem is especially acute if material is recycled from strata not much older than the beds being studied or consists of long-ranging taxa. How then do you know if you have a pollen assemblage that is indigenous and worthy of interpretation, or one that should be ignored? Reworking of pollen is a particular problem in delta environments, which undergo numerous, closely timed intervals of deposition and erosion. Careful study of ancient pollen samples from the Mallik 5L-38 well in the Beaufort-Mackenzie Basin provided new insights into how to recognize reworked pollen. It yielded new age constraints on formations and delineated several periods of non-deposition or erosion. Starting at the top of the well and working downward, the 0 to 270 m interval is younger than 3 million years (Ma) old. From 340 to 700 m is ~20 Ma and from 700 to 900 m is ~28 Ma. Significant erosion near 930 m may have resulted from Antarctic glaciation-induced sea level fall. Coals between 933.65 and 1081.90 m are about 34 Ma.

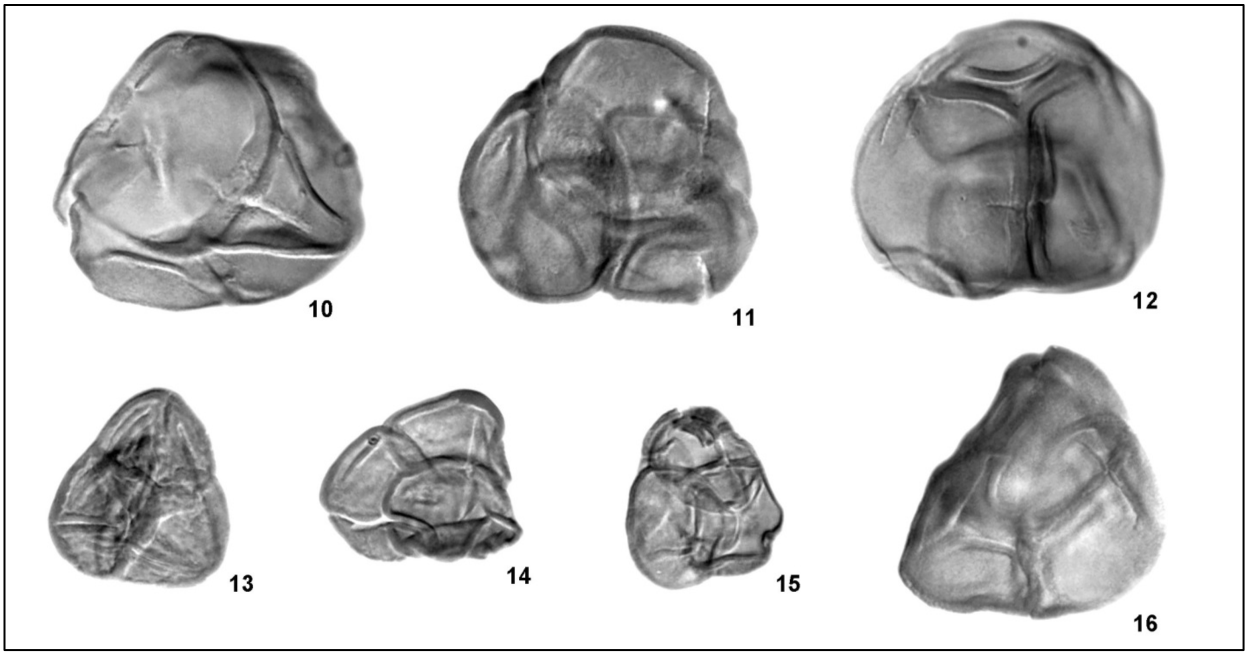

Figure 3.2.2.4-1. Pollen from the Mallik 5L-38 gas hydrate research well, Beaufort-Mackenzie Basin.

Contribution to Program:

New insights into recognizing recycled pollen will improve the reliability of future biostratigraphic work in the Beaufort-Mackenzie Basin and elsewhere. Such environments commonly are prospective for oil and gas exploration; therefore, this work should lead to increased exploration success, a key outcome of the GEM Program. Likewise, improved time constraints on the succession beneath the Mackenzie Delta will lead to improved basin models, with a positive effect on exploration.

Publication:

White, J.M., *in press*. The palynostratigraphy, age and environment of strata penetrated by the Mallik 5L-38 gas hydrate research well determined by differentiating the recycled and contemporaneous palynomorphs. *Geological Survey of Canada, Bulletin 604.*

3.3 Mineralogy and Physical Properties Group: Contributions

Overall, MPP contributed to 58 laboratory study agreements between GSC scientists and other labs and 5 agreements with external consultants or organizations. Of the 58 agreements 14 are multi-disciplinary projects with other lab groups. Because of the diverse capacity of the group it contributes to all programs: GEM (8), TGI-4 (25), Environmental Geosciences (6), Public Safety (5), Nunavut Geoscience (6), Groundwater (5), Climate Change (1), Collections (1), and CCMEO (1). The breakdown with respect to facility and lab shows that all labs are well engaged in all programs across the GSC and ESS. Mineralogy (Ottawa) includes the lapidary facilities (21 projects) at Tunney's Pasture and Booth St., microbeam lab which includes SEM (20 projects plus 7 dedicated SHRIMP projects) and microprobe (10 projects), and XRD (13 projects). The Ottawa Sedimentology facility was engaged in 17 projects and the Sedimentology and Core-Processing facility in GSC Atlantic carried out 3 major projects. The Paleomagnetics and Petrophysics facility in GSC-Pacific managed 4 major projects and has close ties with the GSC-Atlantic facility. All of these facilities and laboratories hired FWSEP, RAP (1) or Co-Op (1) students to assist in ensuring project outputs were completed in timely fashion.

The focus of the laboratories, as noted in the Strategy for SLN- 1.3, is to provide fundamental and innovative information to advance the programs in GSC. This section will review the overall accomplishments of the group as well as highlight scientific achievements of the various laboratories in providing leadership.

3.3.1 MPP Labs Accomplishments:

Paleomagnetism and Petrophysics Facility, Sydney

Lab Development:

- Acquisition of Accupyc1200 gas displacement pycnometer
- Acquisition of GDD SCIP time domain electrical chargeability meter
- Acquisition of Terraplus KT1- susceptibility and conductivity meter
- Transport and refurbishment of University of Toronto Princeton Magnetics alternating gradient magnetometer
- Development of Zarcfit analysis method for electrical impedance spectra.
- Study of petrophysical properties of rocks from the aureole of known deposits (especially Lalor (Manitoba) and Malartic (Quebec), complemented by lithological, mineralogical and geochemical analyses performed by other members of TGI4 and the CMIC Footprints Consortium.
- •Malartic: surprizing observation that mineralization in sediments leads to density decrease along with porosity decrease, resistivity decrease, magnetic susceptibility decrease, and chargeability increase. Hypothesis that mineralization is accompanied by calcite-filling of porosity along with reduction of magnetite to pyrite.
- •Establishing a network of petrophysics laboratories and initiating interlaboratory comparison.

Lab Throughput:

- 1100 samples Archimedean Density and Porosity
- 300 samples Gas Displacement Pycnometer volumes
- 1100 samples Electrical Impedance Spectra
- 800 samples Magnetic Susceptibility and Remanence
- 300 core sections Whole Core Multi-Sensor Scans
- 200 core sections Split Core Images
- 100 core sections Split Core Multi-Sensor Scans

Marine Sedimentology and Core Processing Facility, Dartmouth

Lab Developments:

- Obtained core correlations required for regional stratigraphic assessment studies
- Purchased a new dynamic image analysis instrument (30 µm to 32000 µm) for grain size and shape characterization
- Enhancements to the sedimentology lab's custom Particle Sizing System (PSS) to increase productivity for data management and upload into the Expedition Database.
- Development of several VNR agreements that involved Memorial University, Dalhousie University and Fisheries and Oceans Canada
- Improved understanding of pXRF analyses on marine sediment cores important for technique development

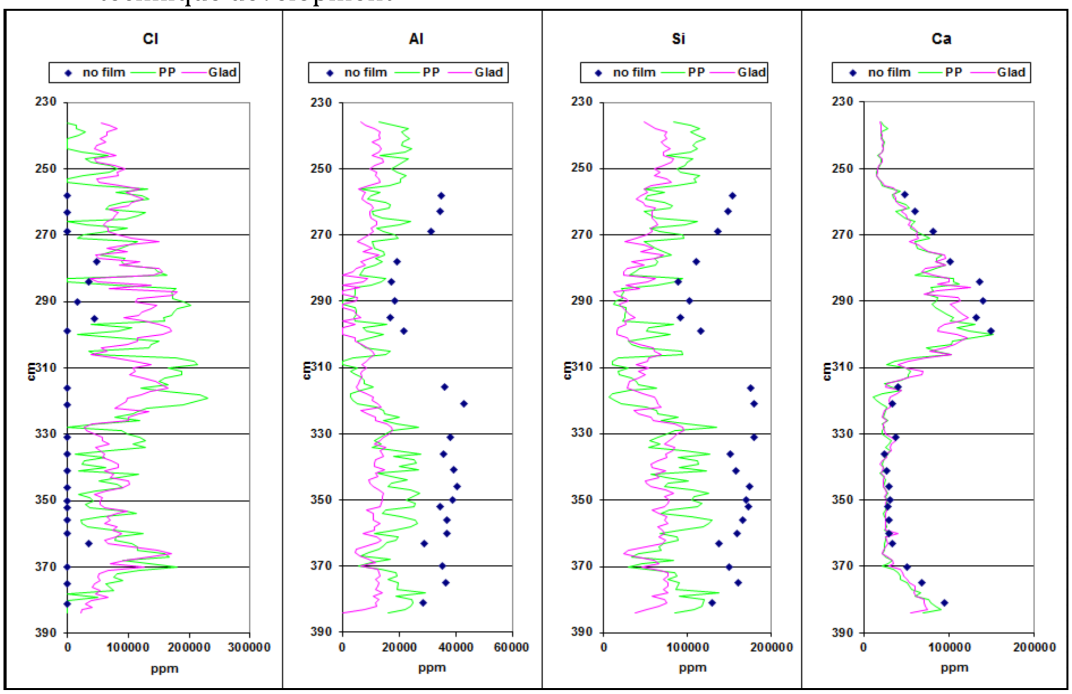

Figure 3.3.1-1 Testing of different thin films for covering marine sediment cores during pXRF analyses. The film is required to prevent contamination of the instrument. The instrument was tested with no film, a polypropylene thin film (PP) and generic clingwrap (Glad). Observed absorption of low energy X-rays by both thin films which caused underrepresentation of lighter elements (Al, Si) with exception of Cl which was overrepresented. Heavier elements (≥Ca) were less affected. This indicates that the water that pools between the plastic film and the wet sediment sample, effectively creating a water film, has significant absorption of low energy x-rays, and is important to technique development.

Lab Throughput:
Sedimentology Lab
764 samples for grain size samples using various methodologies
24 <2um clay slide preparations
48 heavy mineral separations

Marine Core lab
150 m of split core with full suite of analyses
200 m of core logged with pXRF

Sedimentology Laboratory Facility, Ottawa

Lab Development:

- Acquisition of Beckman Coulter LS13 320
- Acquisition of LECO RC 612
    - The adoption of the LECO RC612 instrument from IGRL (spring 2013) has resulted in months of method development and testing conducted by Claudia Moore and Alain Grenier.

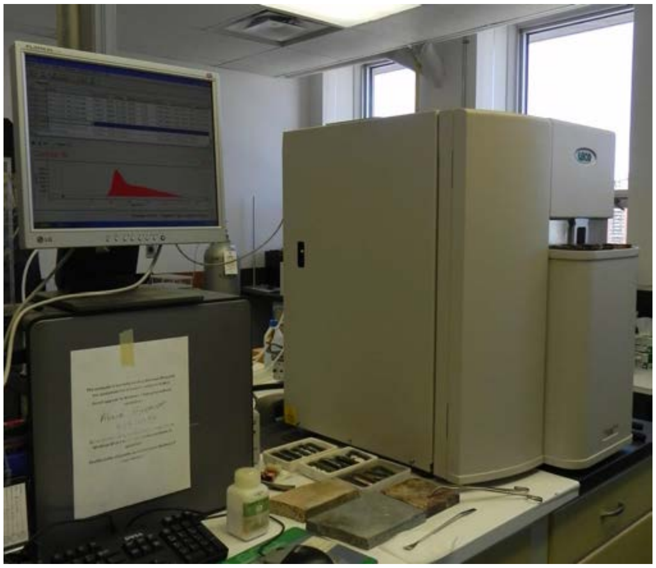

Figure 3.3.1-2 The RC-612 on loan from IGRL-Ottawa.

Lab Throughput:

- 1544 samples received
- 1044 samples Total / Organic Carbon
- 464 samples Complete Grainsize
- 672 samples Archives
- 226 clay separations
- 239 Munsell Colour
- 244 samples Calcite/Dolomite
- 483 samples sieving
- 484 samples Geochem Handling
- 309 samples Freeze Drying

Mineralogy, Microbeam and Lapidary Facilities:

Lab Development:

- XRD: Developed a novel technique to prepare cation-saturated clay samples for XRD analysis.
- IR: Initiated development of a spectral library of clay minerals (Kodama suite). Borrowed instrument (TerraSpec Pro from ASD Inc.) and standardized spectral collection and calibration (with help from two student assistants: Philip Belley and Trevor Flynn).

- Probe: Closed probe (Dec. 2013) due to technical problems. Initiated completing probe analysis at University of Ottawa facility.
- SEM: Upgraded Oxford software (INCA Feature) to search, image, analyse, and report particle data from polished thin sections.

Lab Throughput:

- XRD: Completed analyses of about 300 samples including quantitative interpretation for whole rock and clay-size separates, based on 9 study requests
- Probe: Completed 12 LSA projects using GSC microprobe and the University of Ottawa facility. Overall, about 3500 analyses were completed.
- SEM: In response to 23 lab study agreements, over 1200 hours were logged on the SEM. In addition 60 SHRIMP mounts for geochronology were examined and imaged. The studies supported GEM, TGI, Environment, Groundwater and CNGO projects. In addition, autoradiographs for GEM and Environmental projects were completed.
- CL: Introduced CL-imaging microscope for preparatory work in other labs (e.g., microprobe, SEM, ICPMS-laser ablation).
- Lapidary: Provided lapidary support for on-going active projects, and for academic, private sector and archived Collections in the National Reference Collections Facility. Between two lab facilities (601 Booth and Tunney's pasture), over 2600 actions were completed. These included cutting for slabs, preparing stubs for thin section, grinding and polishing for archive and photography, trimming and cleaning samples prior to pulverization for geochemistry.

3.3.2 MPP Scientific Achievements

3.3.2.1: Optimization of Rock Physical Properties Measurements for Mineral Exploration
Randy Enkin, Paleomagnetism and Petrophysics Laboratory, Sydney, B.C.

Focus Area:

Rock physical properties are the link between geological and geophysical analysis of mineral deposits.

Achievement:

This work was based on physical properties, petrological and geochemical study of samples from 6 porphyry deposits in British Columbia (Enkin and Mitchinson, 2013).

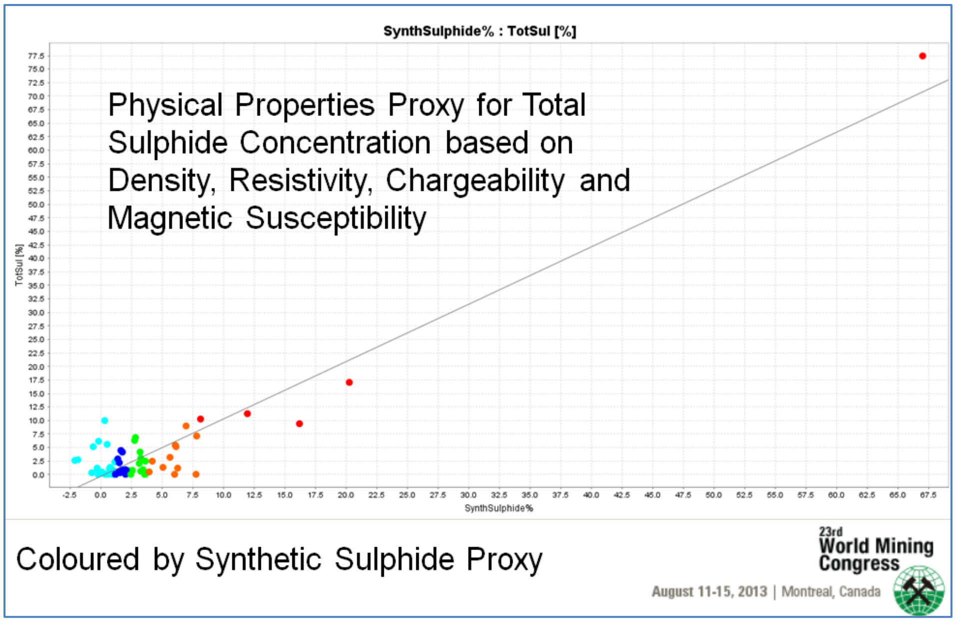

Figure 3.3.2.1-1 Physical properties of samples from porphry deposits..

Contribution to Program:

Program: TGI4 Project: Methodologies

- Development and optimization of new techniques and analysis methods for measuring density and porosity, electrical resistivity and chargeability, magnetic susceptibility and remanence.
- The approach is now being extended to other deposit types in collaboration the CMIC Footprints Project.

The Paleomagnetism and Petrophysics Laboratory has developed new methods to produce proxy estimates of mineralization using only measurements that can be determined from surface geophysical surveys. Specifically, principal component analysis is used to fit a hyperplane to low-mineralization physical properties, and the distance along the perpendicular (i.e. along the minimum eigen-vector) is shown to be a relatively predictive proxy for mineralization.

Publications:

Enkin, R.J., The Rock Physical Property Database of British Columbia, and the Distinct Signature of the Chilcotin Basalts, Canadian Journal of Earth Sciences, submitted, 2013.

Hamilton, T.S., Enkin, R.J. Reidel, M., Rogers, G.C. and Pohlman, J., Paleoseismicity of the Cascadia Accretionary Wedge off of Western Canada: Slipstream; an Early Holocene Slump and Turbidite Record from the Frontal Ridge, submitted, 2013

Enkin, R.J., Dallimore, A., Baker, J., Southon, J. R., Ivanochko, An 11,000 year earthquake chronology of the northern part of the Cascadia subduction zone from anoxic Effingham Inlet, British Columbia, based on a high-resolution radiocarbon Bayesian age model of core MD02-2494, Canadian Journal of Earth Sciences, 50, 746-760, doi:10.1139/cjes-2012-0150, 2013.

Hayward, N., Enkin, R.J., Corriveau, L., and Montreuil, J.-F., "The application of semi-quantitative methods for the targeting of IOCG mineralisation in the Great Bear Magmatic Zone, Northwest Territories, Canada, from potential field and physical property data", Journal of Applied Geophysics, 94, 42-58, doi:10.1016/j.jappgeo.2013.03.017, 2013

Mitchinson, D., and Enkin, R.J., and Hart, C.J.R., "Linking Porphyry Deposit Geology to Geophysics via Physical Properties: Adding Value to Geoscience BC Geophysical Data", Geoscience BC Report 2013-14, 116 p., 2014

3.3.2.2: Physical and chemical characterization of Marine Cores on Hudson Expedition

Kimberley Jenner, Owen Brown and Jenna Higgins, Marine Sedimentology and Core Processing Facility, Dartmouth

Focus Area:

Physical and compositional characterization of fragile, temperature sensitive marine cores provide continuous down core measurements for ties to seismic data, prediction of stratigraphy, sedimentology and geotechnical properties, evaluation of pore water, and calibration of field instruments.

Scientific Achievement:

Played a significant role in the CCGS Hudson Expedition 2013-029 to Baffin Bay and led the collection and initial handling of sediment cores in the field to ensure the integrity of the samples for subsequent analyses at GSC-A. Provided the field-based measurements of key physical properties required prior to shipping samples back to the lab.

Marine labs and expertise are being used to create scientific and regulatory impact on the Canada's east coast oil exploration and development. As an example pXRF, X-ray, grainsize, core photography and color analyses, oxygen isotopes, and advanced geotechnical testing were all used to develop a detailed regional age stratigraphy that constrained the age of very large submarine landslides (20 km run-outs, 65 km long, and scarps ~ 70 m high). We now quote a 1 in 500 year recurrence for such slides. This has implications for the design and approval of the field. Consequently, Statoil are developing a team who will be addressing these new risk aspects.

Publications:

Campbell, D.C., 2014. CCGS Hudson Expedition 2013-029, Geological hazard assessment of Baffin Bay and biodiversity assessment of Hatton Basin, August 14-September 16, 2013; Geological Survey of Canada, Open File 7594, 124 p. doi:10.4095/293694

Cameron, G. D. M., Piper, D. J. W. and MacKillop, K. 2014. Sediment failures in northern Flemish Pass. Geological Survey of Canada, Open File 7566, 141 pages, doi:10.4095/293680

3.3.2.3: Reducing Contamination in Geochemical Processing

Shauna Madore (Manager), Alain Grenier, Claudia Moore, Miriam Wygergangs

Focus Area:
Textural analysis and grain characterization of unconsolidated materials (soils, sediments) to constrain surficial and marine geology processes. Develop rapid, high resolution, digital measurements.

Scientific Achievements:

The Sedimentology Laboratory provides sample preparation, analysis of several physical and a few chemical properties from unconsolidated geological material.

Over the past 3 years the Sedimentology lab has undertaken three phases of study with regards to the analysis of levels of contamination present when using different cleaning methods on electrowelded sieves. The specific purpose of the study described here is to determine how effective commonly used sieve cleaning methods are at preventing/limiting cross contamination between till samples. This study has been effective at showing how possible Pb and Zn contamination is likely to occur. Future work will focus on contamination of Au and PGEs.

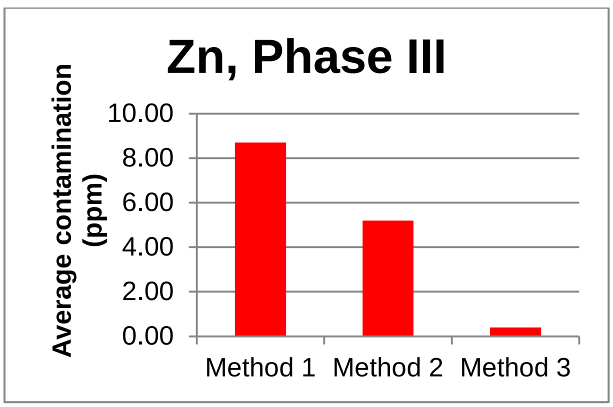

Figure 3.3.2.3-1 Observed contamination in Phase III geochemical results (aqua regia digest on 30 gram sample).

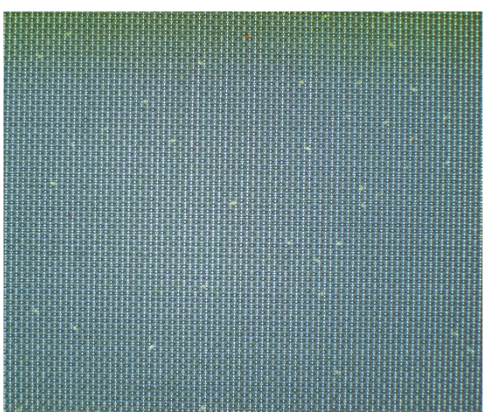

Figure 3.3.2.3-2 Trapped grains in the <63 micron sieve mesh following Method 1 cleaning (Phase III).

Contribution to Program:

Total carbon measurements, sieving and grain size analyses are widely used across all Programs (GEM, TGI4, Climate Change and Environmental Geosciences).

Geochemical analysis is completed on most samples processed in the Sedimentology laboratory. In gaining a better understanding on how possible contamination is occurring and what cleaning methods are most effective would greatly improve analytical results. Suggested cleaning protocols would be made available to other government and commercial labs that conduct similar sample preparation.

Publications:

Grenier, A., Connell-Madore, S., McClenaghan, M.B., Wygergangs, M. and Moore, C.S. In prep.: Study of cleaning methods on electro-welded sieves to reduce/eliminate carry-over contamination between till samples. Current Research

Connell-Madore, S., Grenier, A., Wygergangs, M. and Percival, J.B. In prep.: Investigation into the measurement of soluble salts/conductivity in clay-rich soils, Technical note.

3.3.2.4 Cathodoluminescence Petrography for Mineral Characterization

Jeanne Percival, Katherine Venance. Mineralogy, Microbeam and Lapidary Laboratories

Focus area:
Improve quantitative mineralogical analysis and their elemental composition at micro and nano scales using X- ray diffraction, (XRD), Infrared (IR), Scanning Electron Microscopy (SEM), Electron Microprobe (EMP) and Synchrotron techniques. Provide efficient and high quality sample preparation for geochemical and petrographic studies.

Achievement:

An older Relion Cathodoluminescence Microscope has been resurrected with an improved processing software package. The GSC cathodoluminescence (CL) lab comprises a cold cathode electron gun attached to the petrographic microscope. Cathodoluminescence petrography is a qualitative technique that can provide essential information on provenance, growth fabrics, diagenetic textures and mineral zonation. It highlights variation in trace element composition of many minerals of interest (apatite, feldspar, calcite, quartz) and can be used – as in this study- to target areas of interest prior to EPMA and/or LA-ICP MS analysis.
Colour and intensity are functions of both the sample (phosphor) and the operating conditions (accelerating voltage and beam current). This is a qualitative technique at the GSC lab, since there is not a CL spectrometer attached.

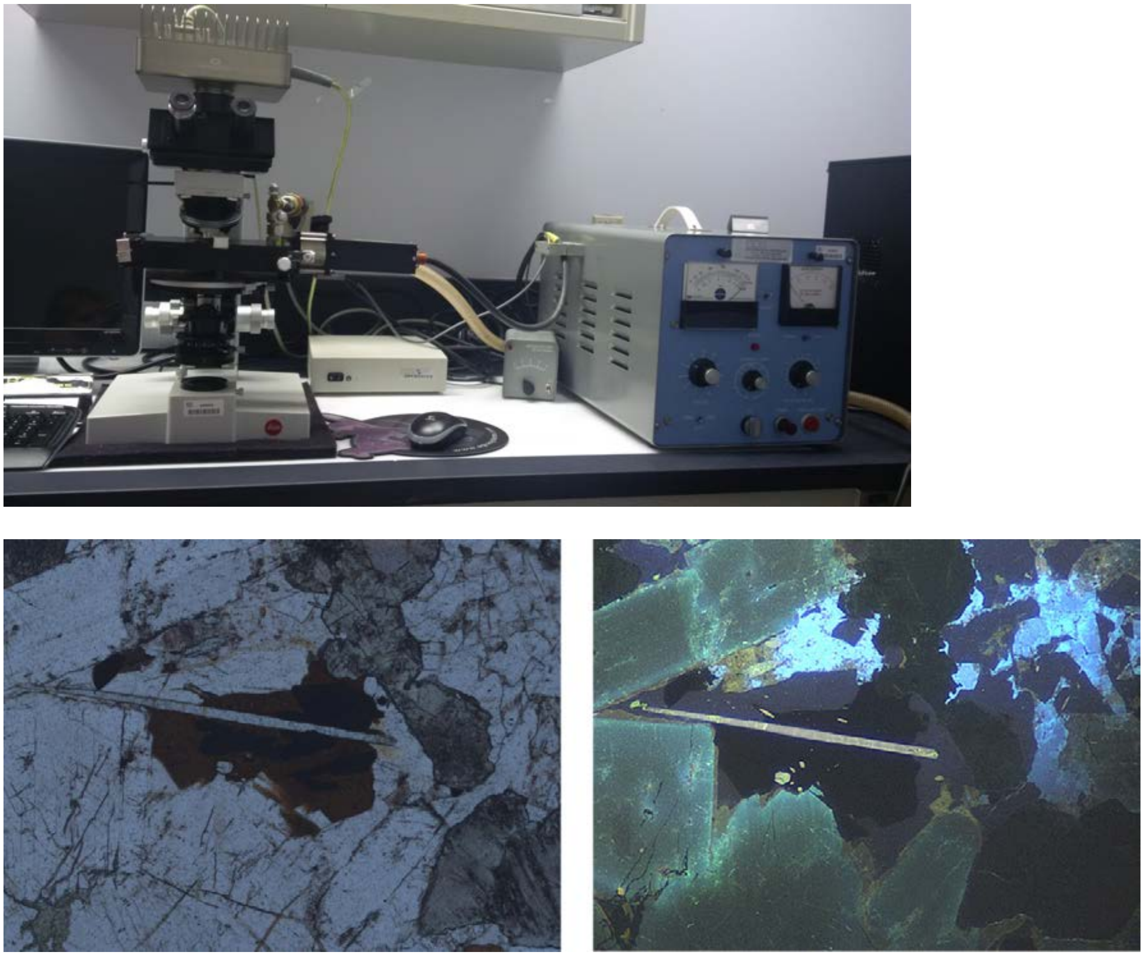

Figure 3.3.2.4-1: Cathode luminescence equipment and images at GSC.

Contribution to Program:

Currently this is being used in a Magmatic – Hydrothermal NiCuPGE Study for TGI-4.

Publications:

Watts, K., Hanley, J.J., Ames, D.E., Kontak, D., Petrus, J. and Veksler, I. (2014) A melt inclusion study of the Sudbury Igneous Complex (Ontario, Canada), part I: evidence for two-liquid immiscibility and constraints on trace element distribution. In prep for submission to Journal of Petrology, 63 pages.

Watts, K., Hanley, J.J., Ames, D.E., Petrus, J., and Kontak, D., (2014) A melt inclusion study of the Sudbury Igneous Complex (Ontario, Canada), part II: metallic trace elements in apatite and melt, and applications to exploration for magmatic sulfide deposits. In prep for submission to Journal of Petrology, 39 pages.

3.3.2.5: Scientific Achievement: Particle Size Analyses using Inca Feature Software
Pat Hunt, Microbeam Facility, Ottawa

Focus Area:

The technique is extremely useful with polished thin sections or grain mounts (A) to identify and analyse (EDS) many particles. Once the greyscale is set (B), INCA37 detects every grain. Each grain is then assessed for its morphology and chemistry (C, D). When the run is complete, (typically 1-3 hours depending upon number of particles), specific chemical elements are selected. Particle data can then be plotted and analysed (G) or exported to Excel® (H).

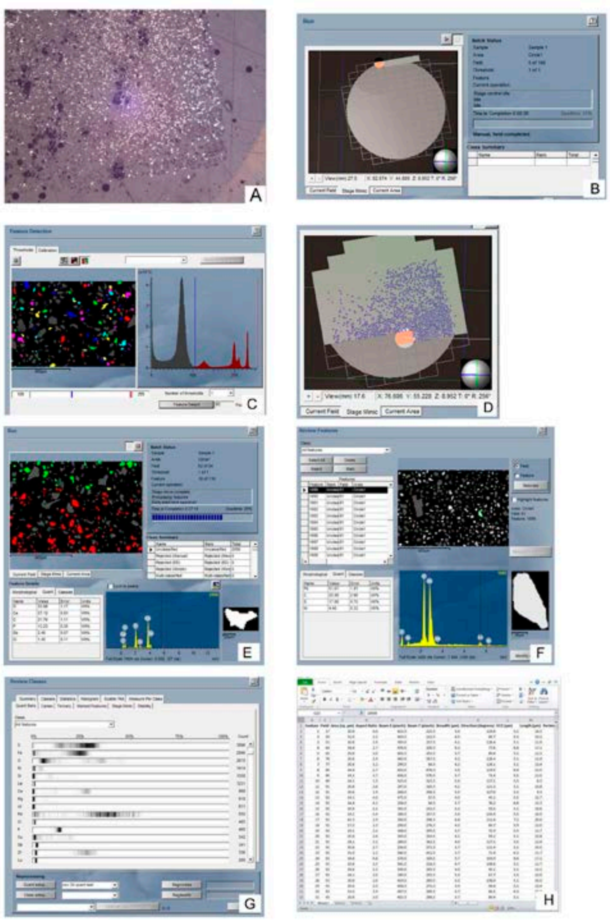

Figure 3.3.2.5-1 Technique for polished thin sections or grain mounts.

Contributions to Program:

This software is currently available for use in any project, for any program. This has become a standard protocol for searching **minerals** for geochronological applications. New applications include searching for PGEs for TGI-4 projects.

3.4 CONTRIBUTIONS: ISOTOPE GEOCHEMISTRY AND GEOCHRONOLOGY

A total of thirty-five laboratory study agreements were initiated and completed by the IGRL as well as four agreements with external partners. Overall the lab group contributes to all programs however each facility has their speciality: the geochronology and radiogenic facility mainly contributed to the TGI-IV and GEM program, while the Delta Facility contributed mainly to the environmental program and the light stable isotope facility contributes only to TGI-IV. The group is highly involved in the development and application of new techniques in isotope geochemistry which are identified in accomplishments and several are highlighted under Scientific Achievements.

3.4.1 *IGGG ACCOMPLISHMENTS*

Geochronology and Radiogenic Isotopes Facility

Lab Developments:

- Developed new analytical protocols for in situ xenotime analysis by ion microprobe. Application to dating the time of alteration associated with mineralization in the Meliadine gold deposit, TGI4 Gold systems.
- Major contributor to TGI4 program in U-Pb ages refined structural of Musselwhite mine, geochronological test of super domain Ni-Cu
- Core contribution to GEM Frontiers with over 50 new U-Pb ages for the Rae Craton, refining tectonic history of the area and establishing extent of crustal age domains – preliminary results released in Open File report.
- Six projects completed under the auspices of the SHRIMP Business plan providing access to University researchers and provincial/territorial government surveys (Waterloo, Simon Fraser, University of Ottawa, Saskatchewan Geological Survey, Northwest Territories Geoscience Office, Canada Nunavut Geoscience office).
- Developed UV laser capabilities for in situ Ar-Ar analysis including installation and testing of a 213 nm wavelength UV laser. Permits detailed characterization of thermal histories with current applications in the TGI-IV intrusion-related project.
- Outreach activities - Two new outreach products created and staff volunteered at events during National Science and Technology week and the Cool Science event at the Museum of Science and Technology.

Delta Facility

- Significant contributor to ESS's Environmental Geoscience Program, primarily through work carried out under framework of the CORES (2009-2014) Project
- Departmental Achievement Awards given to two Delta-lab scientists (Ahad & Savard) for their contribution to environmental research in the oil sands region
- Delta-lab PhD student Josué Jautzy won the Feb 2014 *Étudiants-chercheurs étoiles des trois Fonds de recherche du Québec* award for best *Nature and Technology* research paper (published May 2013 in *ES&T*):

LSI Facility

- Designed and implemented technological advancements to improve the accuracy of in-situ S-isotope analyses by fluorination system (MILES) – a unique analytical capability in Canada.

- Developed collaboration with researchers at McGill University to combine the unique sampling capabilities at the GSC with instrumentation at McGill for accurate in-situ micro-analyses of all four stable S isotopes (32, 33, 34, and 36). Applications to TGI-IV program.
- Completed an S-isotope study of IOCG deposits (NWT); Open File report, journal paper, and several abstracts prepared for submission with co-authors.
- Completed initial phase of in-situ S-isotope study of the Prairie Creek deposit (TGI-IV SEDEX project), demonstrating thermal zoning in deposit related deposit origin; geothermometry of ore formation; and distinction of two separate mineralizing events.
- Demonstrated that Oxygen isotope mapping of the hydrothermal system at the Izok Lake deposit, Nunavut (TGI-IV Slave VMS) can be used to effectively vector to the ore deposit; potential as an exploration tool demonstrated to company.

3.4.2 IGGG Scientific Achievements

3.4.2.1: In Situ Boron Isotopic analysis of tourmaline using the SHRIMP ion microprobe

Bill Davis – Isotope Geochemistry and Geochronology

Focus Area:

In situ, micron-scale isotopic analytical techniques as chronometers and tracers of complex geological processes - *SHRIMP ion microprobe*

Achievement:

Over the past year, the IGGG has implemented in situ B isotopic analyses of tourmaline by SHRIMP ion probe. Boron isotopic composition is one of the most diagnostic signatures of fluids derived from marine waters or from marine metasedimentary rocks (Figure 3.4.2.1-1). The B isotopic composition of tourmaline, a common boron-bearing mineral in alteration assemblages of mineral deposits (Figure 3.4.2.1-2), can be used to characterize the source of boron in mineralizing hydrothermal brines. Interaction with marine fluids or fluids derived from marine sedimentary rocks is thought to be a key factor for developing mineralizing hydrothermal brines in unconformity uranium systems, as well as in some intrusion-related Iron Oxide-Copper-Gold systems. Boron isotopic analysis provides a means to test and further develop these models.

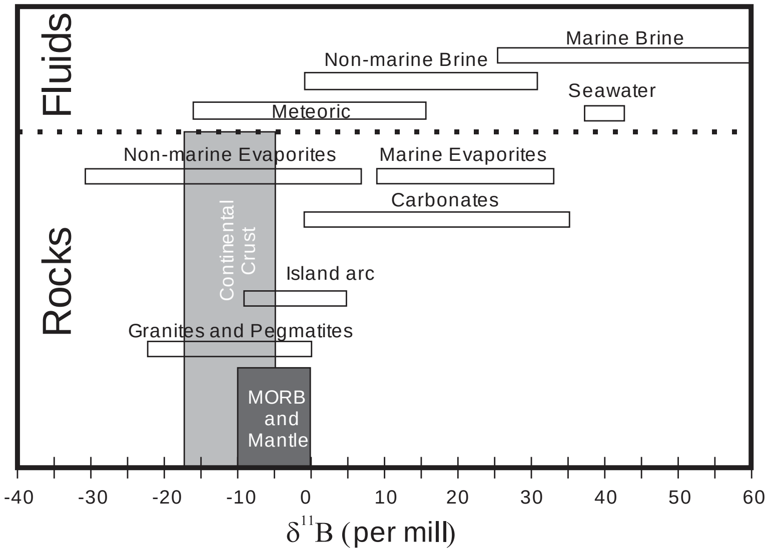

Figure 3.4.2.1-1 Boron isotopic composition of comment fluid and rock materials showing the distinctly positive boron isotopic values of marine rocks and fluids relative to typical values for continental crust

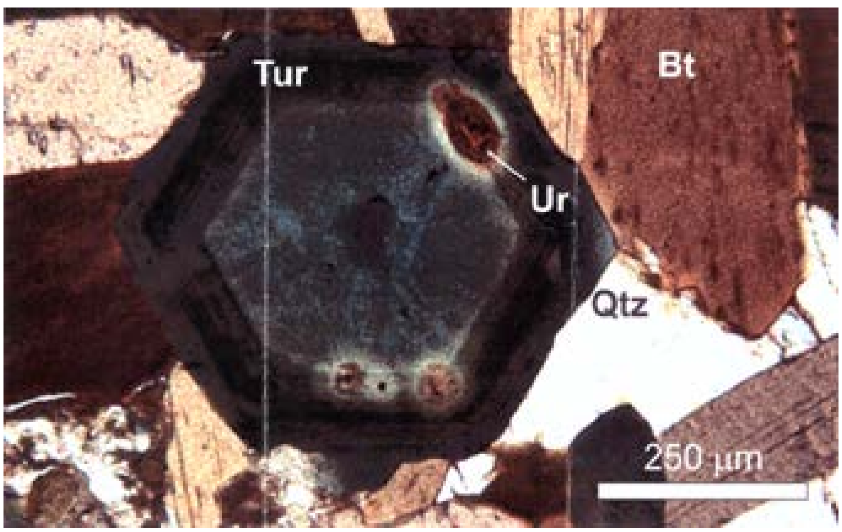

Figure3.4.2.1-2 Example of tourmaline (Tur) hosting uranium mineralization (Ur) at the Nori deposit, Great Bear magmatic zone, N.W.T.

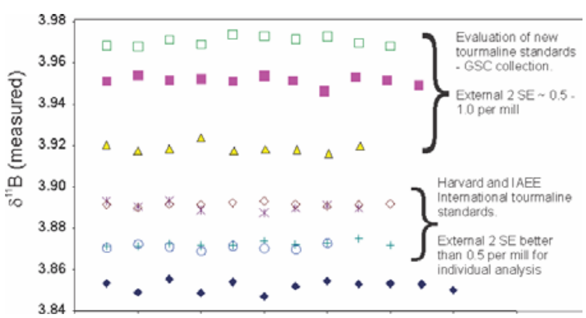

Figure 3.4.2.1-3 Analytical results for tourmaline standards and evaluation of new GSC internal standards

New analytical protocols have been developed and suitable mineral standards characterized to permit accurate analyses ***otopic compositions of common fluid and rock materials*** at the sub 20 □mscale with a precision of ~0.5 per mill or better

Contribution to Program

Development of the in situ boron isotopic technique is currently being applied in the TGI-4 program, including the uranium and intrusion-related projects. Three pilot studies investigating the source of alteration fluids in unconformity uranium deposits in the Athabasca basin, IOCG deposits in the Great Bear magmatic zone and the Windjam porphyry system in the Cordillera are ongoing.

3.4.2.2 Source apportionment of polycyclic aromatic hydrocarbons (PAHs) in the Athabasca oil sands region

Jason M. E. Ahad and Martine M. Savard, IGGG, Delta-Lab, CGC-Québec
Josué Jautzy, doctoral student, INRS (under supervision of Jason Ahad)

Focus Area:

Method developments for stable isotope analyses used in tracing environmental and hydrological processes

Achievement:

The continued development of Canada's Athabasca oil sands has raised concerns about its potential impact on the surrounding environment. Of particular interest are polycyclic aromatic hydrocarbons (PAHs), a group of organic contaminants that are toxic and known or confirmed carcinogens. Naturally present in bitumen, PAHs are also released into the environment through the incomplete combustion of organic matter, whether from modern biomass or fossil fuels, and via diagenetic processes. Evaluating the impact that mining activities has on the environment thus requires discrimination between anthropogenic and natural inputs.

Over the past several years, research carried out in the Delta-lab has focused on the application of compound-specific isotope analysis (CSIA) of PAHs as a tool for source apportionment in the Athabasca oil sands region. These types of analyses in organic-rich sediments with generally low levels of PAHs required significant modification and refinement of clean-up and extraction techniques, as well as new approaches to assessing accuracy and precision. In conjunction with depositional fluxes and molecular diagnostic ratios, $\delta^{13}C$ signatures of PAHs in sediment cores from two lakes east of the main area of mining operations (Figure 3.4.2.2-1) demonstrated an increasingly larger input of petroleum-derived (i.e., petrogenic) PAHs over the past 30 years (Jautzy et al., 2013a). The information obtained from compound-specific $\delta^{13}C$ analyses provided evidence to suggest that bitumen in dust particles associated with wind erosion from open pit mines was the principal petrogenic source. Not only was this the first study to use CSIA as a tool to examine sources of PAHs in the Athabasca oil sands region, it was also the first successful application of CSIA to evaluate sources of organic contaminants in dated boreal lake sediments. Work continues on method development required for accurate and precise measurements of δ^2H isotopes in PAHs, as well as for separation and cleanup techniques required for compound-specific radiocarbon (^{14}C) analysis (Jautzy et al., 2013b). The combined use of these approaches – never before carried out – will mark a significant analytical achievement in applied isotope geochemistry.

Contribution to Program

Research into source apportionment of PAHs in the Athabasca oil sands region has been carried out under the framework of the Environmental Geosciences Program's CORES Project (Coal and Oil sands Environmental Sustainability; 2009-2014). The CORES project was designed to contribute to a long-term NRCan objective of effectively identifying geochemical disturbances and risks to ecosystems related to mining natural resources, and mitigating the potential risks to ecosystem health. A precise understanding of sources of organic contaminants will assist provincial and federal environmental agencies in developing more efficient environmental guidelines in relation to the development of the oil sands.

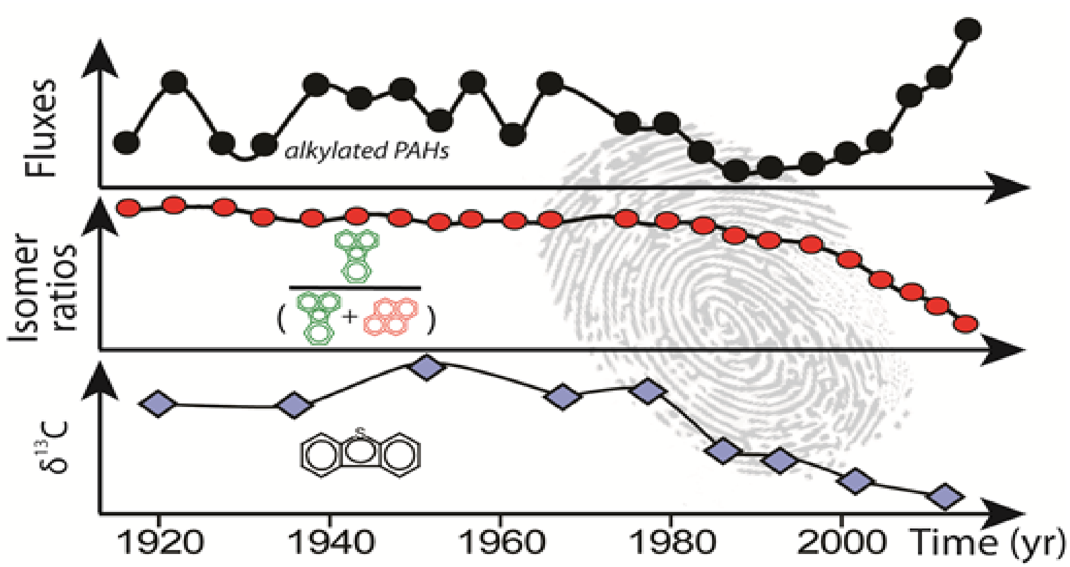

Figure 3.4.2.2-1: Alkylated PAH fluxes, diagnostic ratios (isomer ratios), and compound-specific $\delta^{13}C$ signatures (e.g., dibenzothiophene) in a lake situated 55 km east of the main area of oil sands mining operations. The trends demonstrate an increasingly larger input of petroleum-derived (i.e., petrogenic) PAHs over the past 30 years (Jautzy et al., 2013a).

Publications:

Jautzy, J., Ahad, J.M.E., Gobeil, C., Savard, M.M., 2013a. Century-long source apportionment of PAHs in Athabasca oil sands region lakes using diagnostic ratios and compound-specific carbon isotope signatures. Environmental Science & Technology 47, 6155−6163.

Jautzy, J., Ahad, J.M.E., Hall, R.I., Wiklund, J.A., Gobeil, C., Savard, M.M., 2013b. Compound-specific radiocarbon analysis to evaluate the contribution of Peace River floodings to the PAH background in the Peace-Athabasca Delta, American Geophysical Union Fall Meeting, San Francisco, CA, USA.

3.4.2.3 Oxygen isotope Geochemistry to map paleo-hydrothermal system at Izok Lake.

Bruce Taylor, LSI, Ottawa

Focus Area:
Micro-analytical and in situ stable isotope analysis to model fluid/rock interaction, mineralization processes, and genesis of sedimentary and crystalline rocks.

Contribution to Program

Oxygen isotope at Izok Lake mapping is on-going, with the goal to refine the zoning pattern shown in Figure 3.4.2.3-1 and, where drilling access permits, document the feeder zone used by mineralizing fluids. Detailed (sample site scale) comparisons of results for oxygen isotope analysis, calculated Hashimoto index, and ground- and air borne alteration mapping by spectral (reflectance) analysis are underway. The end result will be a refined exploration protocol that incorporates these techniques.

Achievement:

Whole-rock oxygen isotope techniques were employed to map the paleo-hydrothermal system responsible for the Izok Lake Zn-Cu-Pb-Ag VMS deposit in the Slave Province. Previous studies by the LSI lab have established these techniques as a robust strategy in mineralized terrains, even to the highest grades of metamorphism. Mapping the paleo-hydrology of mineralized systems, guided by predictable patterns of oxygen isotope alteration, can assist both regional and local exploration. The Izok Lake study (below) is on-going.

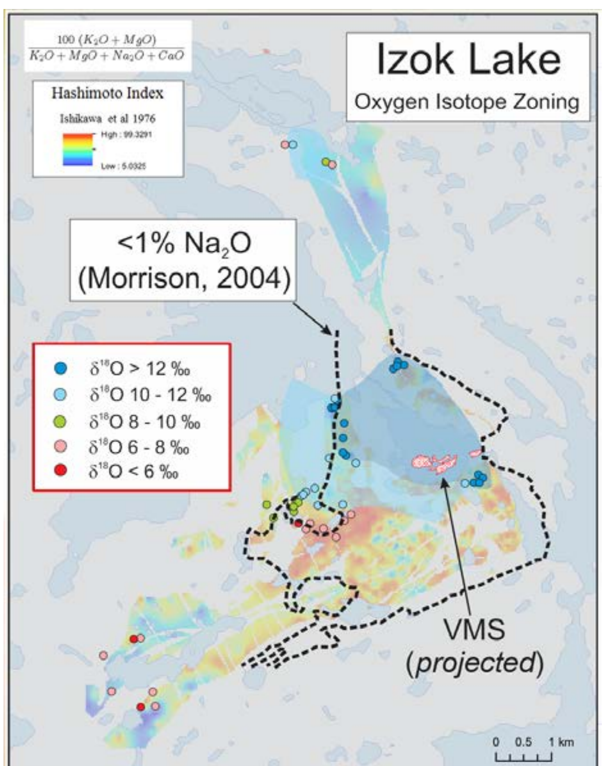

Figure 3.4.2.3-1 Map of Oxygen isotope zoning at Izok Lake compares zoning to the Hashimoto index for alteration (based on whole-rock chemical analyses) and the isopleth of 1% Na_2O (a general indication of hydrothermal alteration). The zoning pattern reflects an up-right hydrothermal system, viewed from the top (hanging wall); blue zones record ^{18}O enrichment under waning temperatures and fluid flow. Projected VMS ore bodies are encompassed within the "bulls eye" of the mapped zoning. An ^{18}O-depleted, high-temperature up-flow zone is expected in the footwall beneath the deposit.

3.4.2.4 In situ UV laser $^{40}Ar/^{39}Ar$ analysis

Dawn Kellett and Nancy Joyce, Ar-Ar Lab Facility, Ottawa

Focus Areas:

Thermochronology and high-spatial resolution geochronology

Contribution to Programs

Over the past 12 months, researchers in the Noble Gas laboratory at the GSC have tested and developed its capacity for *in situ* UV laser $^{40}Ar/^{39}Ar$ analysis. This novel technique has two key purposes. The first is to provide data for thermochronological modelling of individual crystals to determine cooling rates of tectonic terranes. The second is to determine the spatial distribution of Ar within single crystals and discrete compositional and/or structural domains within rock thick sections, for example to constrain multiple low temperature crystallization/recrystallization events. The laboratory innovation included testing and then replacing the lab's existing UV laser with a laser of shorter wavelength to increase spatial resolution, improve sampling control and improve optics.

The *in situ* UV laser technique has been applied to the measurement of $^{40}Ar/^{39}Ar$ age transects across single muscovite crystals in the CNGO Hall Peninsula regional mapping project. PHD student Diane Skipton (UOttawa) is now modeling her resulting Ar data to determine the regional cooling history for the Trans-Hudson orogen in Hall Peninsula. This modeling will allow her to test a hypothesis that initial slow cooling of the orogen was perturbed by late tectonic collapse. The *in situ* UV laser technique is also being applied in the TGI4 VMS project, in which the regional metamorphic history of the Bathurst mining camp is compared to a distinct unit, the Tomogonops Formation, to test a geophysical forward model suggesting that it was accreted subsequent to the main metamorphism and deformation of the mining camp. The region has been poly-metamorphosed and poly-deformed at low metamorphic grade, thus *in situ* $^{40}Ar/^{39}Ar$ age dating of discrete cleavage planes will provide a hitherto unachievable control on the time progression of deformation in the region.

Publications:

Skipton, D.R., Kellett, D.A., Joyce, N., Schneider, D.A., and St.-Onge, M.R., 2014. Using UV laser $^{40}Ar/^{39}Ar$ thermochronology of muscovite to elucidate the cooling history of the orogenic middle crust of the Trans-Hudson orogen, Baffin Island, Nunavut. GACMAC, Fredericton, Canada

Landry, M., Kellett, D.A., and Rogers, N., 2014. Timing of metamorphism of the Tomogonops Formation, Bathurst Mining Camp, New Brunswick and implications for regional structural development. Prospectors and Developers Assoc. Canada annual conference, Toronto, Canada

3.5 Organic Geochemistry and Petrology Group: Contributions

A total of twelve laboratory study agreements were initiated and completed by OGPet as well as 14 agreements with external partners. The majority of by the OGPet agreements deal with energy issues and consequently most of the agreements are with GEM or the GNES program. The group is highly involved in the development and application of new techniques in isotope geochemistry which are identified in accomplishments and several are highlighted under Scientific Achievements.

3.5.1 OGPet Accomplishments

Lab Throughput:

- 4882 RockEval/TOC analyses
- 917 GC/GCMS analyses - Biomarker Analysis
- 584 pellets for Vitrinite Reflectance analysis

3.5.2 OGPet Scientific Achievements

3.5.2.1 Thermal Desorption/Pyrolysis-Gas chromatography-Mass spectrometry (TD/Py-GC-MS/FID) analysis for unconventional hydrocarbon reservoir characterization

Dennis Jiang, Marina Milovic, Rachel Robinson and Andy Mort, Organic Geochemistry Facility

Focus Area:

Molecular geochemical characterization and analysis of unconventional shale and tight reservoir samples

Contribution to Program

Problem: The S1 peak from Rock-Eval analysis has been a key input parameter for hydrocarbon resource assessment especially for unconventional shale reservoirs, and this has been based on the assumption that S1 peak represents the free petroleum hydrocarbons currently present in the rock samples. As S1 peak is a bulk measurement of the amount of hydrocarbons, both natural and synthetic hosted in the rock samples, it is necessary to investigate whether the S1 peak from Rock-Eval analysis is composed of only indigenous petroleum hydrocarbons, or includes hydrocarbons introduced from drilling activities and to to what extent.

Solution: Selected shale core and cuttings samples were subjected to TD-GC-MS/FID analysis to characterize the molecular composition of their S1 peaks. The thermal desorption is carried out in the EGA pyrolyzer furnace at 300oC for 3 minutes, which is the same condition as the Rock-Eval thermal desorption treatment for S1 peak. The released S1-hydrocarbons are transferred directly to the coupled GC-MS/FID system to obtain its fingerprint. Analysis of Devonian Duvernay, Triassic Montney and Jurassic Nordegg shale core samples from Western Canada Sedimentary basins revealed various degree of contamination by the synthetic oil (e.g. nC14 alkenes, Figure 3.5.2.1-1) used in the drilling mud. Drilling mud invasion has also been identified in Ordovician Utica and Lorraine cores from Quebec and cuttings samples from Liard basin of British Columbia.

Achievement:
Development of methodologies for direct on-line GC-MD/FID analysis of hydrocarbons liberated from rock samples by thermal desorption at <300oC and/or by pyrolysis at specified temperatures utilizing the newly purchased EGA/PY-3030D Multi-shot Pyrolyzer and GC-MSD/FID systems. The analytical capability allows (1) effective and efficient molecular characterization of free petroleum hydrocarbons hosted in shale samples from unconventional resources drilling to assess their fluid property; (2) quick and reliable evaluation of contamination to core and cuttings samples and its effect on their Rock-Eval results which have been routinely used for resource assessment; (3) improved assessment of hydrocarbon potential of source rock systems via on-line compositional analysis of the various petroleum components obtained from both open and closed pyrolysis. The facility can be operated automatically to analyze over 40 samples in a cycle, and requires no use of hazardous solvents and no sample pre-treatment other than grinding.

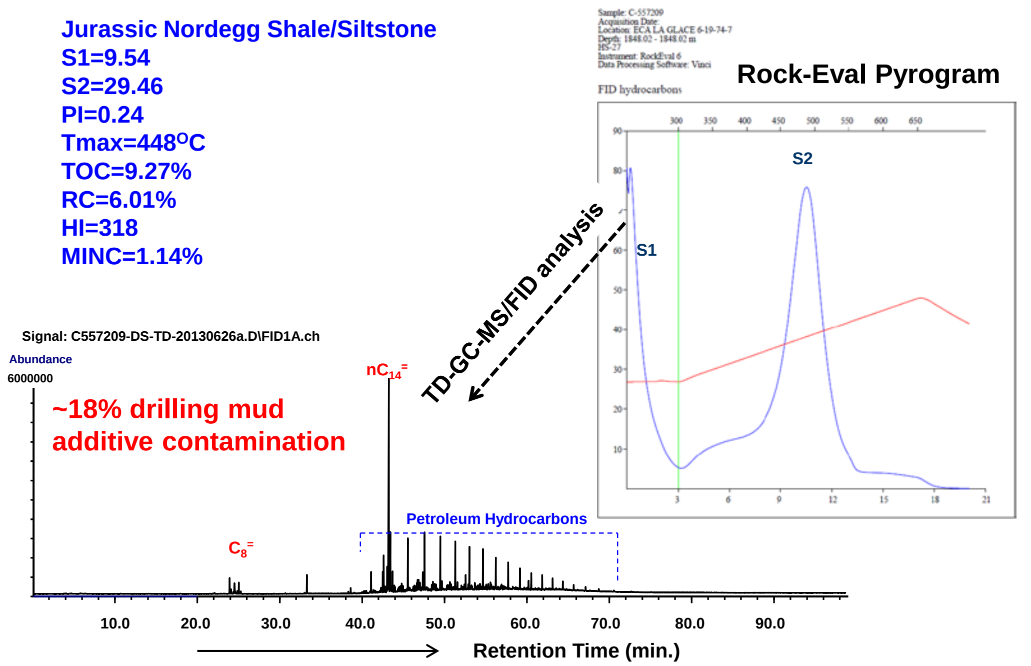

Figure 3.5.2.1-1 Rock-Eval pyrogram (upper right) and TD-GC-MS/FID trace (bottom left) of S1 peak showing the composition of free hydrocarbons in a Nordegg shale core sample. $nC_{14}^{=}$ are the nC14 alkenes used as synthetic oil in drilling mud.

Publications:

McMechan, M E; Obermajer, M; Stewart, K R; Jiang, C; Kung, L. (2013) Rock-Eval/TOC data for selected samples from boreholes in the Toad River area (NTS 94-N), Liard Basin, northeast British Columbia. Geological Survey of Canada, Open File 7480.

Haeri Ardakani, O., Sanei, H., Lavoie, D., Chen, Z., Jiang, C., and Mechti, N., (2014). Thermal maturity and organic petrology of the Upper Ordovician Utica and Lorraine shales, southern Quebec, Canada. Geoconvention 2014, 12-14 May, Calgary, Canada.

Haeri Ardakani, O., Sanei, H., Lavoie, D., Chen, Z., Jiang, C., and Mechti, N., (2014). Organic Matter Characterization of the Upper Ordovician Utica and Lorraine shales, Southern Quebec, Canada. AAPG International Conference & Exhibition 2014, 14-17 September, Istanbul, Turkey.

3.5.2.2 Nanoscale reflectance and fluorescence spectra measurements for thermal maturity and reservoir properties measurements

Hamed Sanei, Omid Haeri-Ardakani, Julito Reyes
Organic Petrology Research Facility, Calgary

Focus Area: Analyses of thermal maturity indicators on submicron organic particles and innovative measurements of rock's physical properties

Contribution to Program

Problem: Unconventional petroleum rocks contain small quantity of organic matter, often less than 1 wt%. Previous measurements of thermal maturity on organic particles used 15 micron measuring aperture. In the organic lean sample, it is almost impossible to find a dispersed organic particle large enough to provide a properly polished 15 micron diameter surface. Also conventional thermal maturity indicators such as bitumen and vitrinite reflectance measurments may not be applicable in certain samples.

Solution: We have developed a method to measure optical properties on the nanoscal organic particles. This provides abundance of measurements in a single sample and improves the statistical significance of the measurements in a single sample. Large number of readings in a single sample provides detailed information on multiple generations of hydrocarbon, reworked input, diagentic degradation of organic matter, etc. Furthermore, we have developed a quick and economical method to measure particle size distribution and porosity measurements in an unconventional reservoir samples. This method will provide alternative mean of measuring reservoir properties simultaneously with detailed organic petrology.

Achievement:

Acquisition of a new organic petrology microscope and upgrade of another microscope system has enabled us to provide robust measurements of thermal maturity indicators in the organic lean tight to shale rocks. We are implementing new technique to measure reliable thermal maturity on dispersed organic matter in the rocks with TOC of less than 0.5%. This involves measurements on nanoscal particles, which provide abundance of measurements in a single sample. This improves the statistical significance of the measurements in a single sample. Also with increased frequency of readings in a single sample, multiple data populations emerge, which can provide useful information such as multiple generations of hydrocarbon, reworked input, diagentic degradation of organic matter, etc. We have also developed alternative techniques to measure thermal maturity using innovative fluoerecence spectrometry.

Furthermore, we have developed a quick and economical method to measure particle size distribution and porosity measurements in an unconventional reservoir samples. This method will provide alternative mean of measuring reservoir properties simultaneously with detailed organic petrology.

Publications:

Hamed Sanei. 2014. Geochemical and petrological control on the reservoir quality of the unconventional petroleum systems in western Canada. European Geosciences Union General Assembly 2014 Vienna, Austria, 27 April–02 May 2014

Stephen Grasby, Benoit Beauchamp, and Hamed Sanei (2014). Recurrent Early Triassic marine anoxia, impacts of volcanics? European Geosciences Union General Assembly 2014 Vienna, Austria, 27 April–02 May 2014

Hamed Sanei 2013. Unconventional Petroleum systems; and role of organic matter in defining the reservoir characteristics. GEOSCIENCE SEMINAR, June 10, 2013, Geoscience, auditoriet 1671-137, Aarhus University, Aarhus, Denmark.

3.5.2.3 Organic Petrology and vitrinite thermal maturation profiles for eight Yukon petroleum exploration wells in Eagle Plain and Liard basins

J. Reyes, S. Saad, and L.S. Lane, Petrology Lab Facility, Calgary

Focus Area:

Organic Petrology and vitrinite reflectance of dispersed organic matter in Eagle Plains and Liard Basins.

Contribution to Program

This study is part of the GEM program. The findings contributed to the overall understanding of the burial and thermo tectonic history of the Eagle Plains and Liard Basins and its oil and gas potential.

Problem: There are large gaps in existing data pertaining to complex nature of the basins burial and thermo tectonic history of the Eagle Plains and Liard Basins.

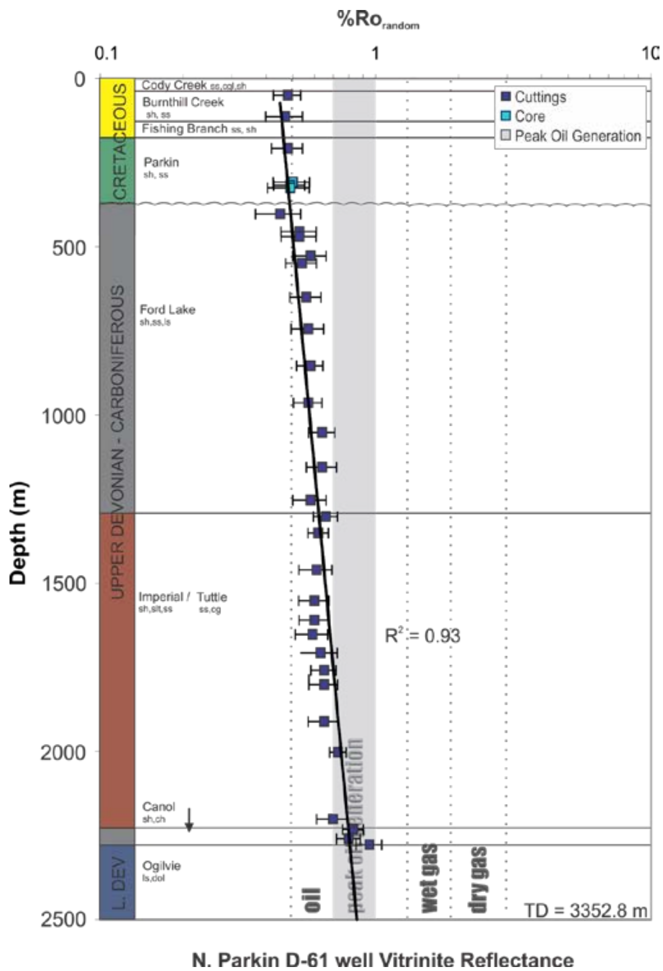

Figure 3.5.3.2-1 Random vitrinite reflectance values plotted against depth from the North Parkin D-61 well. Formation tops are modified from Dixon, 1992; Pugh, 1983 and Morrow, 1999, based on Lane et al., 2012.

Approach: Identify existing knowledge gap and provide new data and interpretation. Perform detailed and comprehensive petrographic analysis of the organic macerals using reflecting microscope equipped with Photometer and UV light for both vitrinite reflectance (VR) measurement and fluorescence microscopy. Provide thermal maturity profile.

Achievement:

The vitrinite reflectance analysis show that most of the Upper Devonian to Lower Cretaceous strata (Whitestone River, Imperial, Tuttle, Blackie, Hart River and Canol formations) reached the early stage of oil generation (>0.5 %Ro) or are at maturity levels equivalent to the oil generating window (0.70-1.30 %Ro) with the exception of the Blackstone D-77 well located in the southernmost part of the basin, which reaches the wet to dry gas window (>1.30 %Ro) in Carboniferous strata. The high thermal maturity of the Blackstone D-77 well as compared to the other wells can be attributed to deeper burial depths but is possibly due to the hydrothermal anomalies observed in the Ogilvie Formation, which shows evidence of dolomitization and thermal cracking. Thick sections of the Hope N-53, Ellen C-24 and Shaeffer Creek O-22 wells attained the peak oil generating window (0.70 to 1.00 %Ro) including a thick section of the Whitestone River Formation in N. Hope N-53 well.

%Ro values for the Canol Formation range between ~0.8% (N. Parkin D-61) to ~1.8% (Blackstone D- 77) in wells that encountered this unit. In nearly all wells, mid-Cretaceous strata are within the oil window (Ro>0.5%). Most of the samples from the Cretaceous units for both Ellen C-24 and Shaeffer Creek O-22 wells are immature (<0.50 %Ro) or in the biogenic gas window. No thermal discontinuity at the sub-Mesozoic unconformity, demonstrating that the thermal peak post-dates mid-Cretaceous time throughout the basin. At the sub-Mesozoic unconformity, vitrinite reflectance varies from ~0.4% in several wells, up to ~1.0% (N. Hope N-53); but there appears to be little correlation between thermal maturity and present burial depth. The kerogen type by source is dominated by Type II and Type II/III of mostly marine depositional environment.

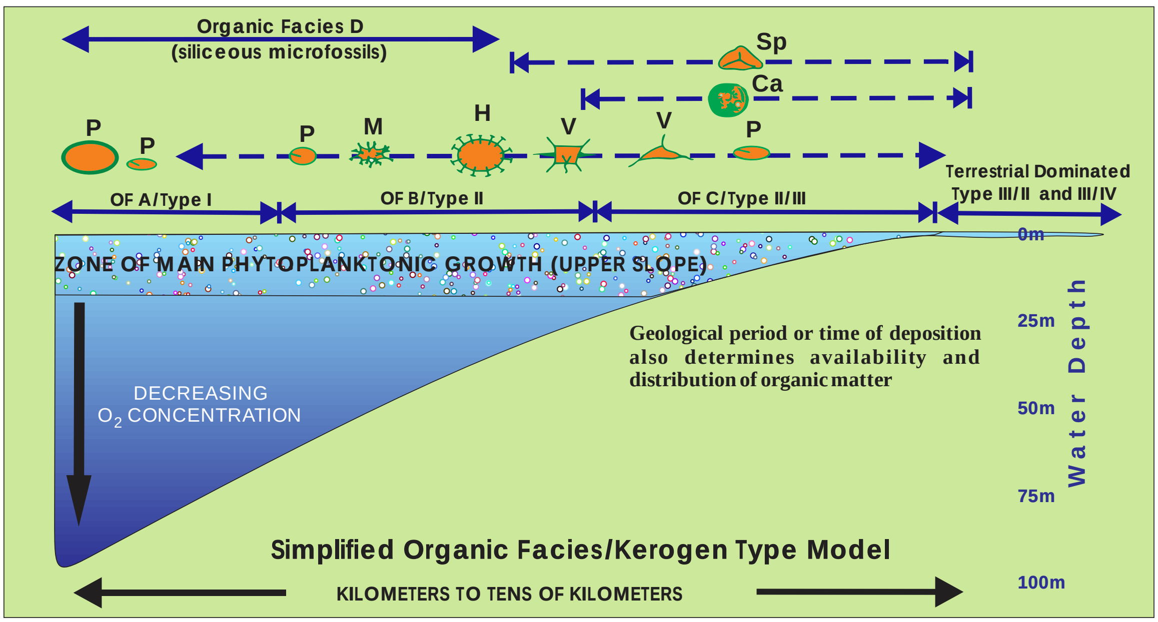

Figure 3.5.2.3-2 Simplified organic facies (OF)/kerogen type by source model for Eagle Plain Basin, Yukon, Canada base on observed dispersed organic matter (DOM). Organic facies A, B and C (Type I, II, II/III) is characterized by unicellular prasinophyte alginite (e.g. planktonic green microalgae, "P"), spiny acanthomorphic acritarchs (planktonic, green microalgae; "H, M, V", coccoidal alginite (planktonic green and/or blue green microalgae, "Ca") with minor to trace amount of input from terrestrial plant-derived macerals (i.e. spores "Sp"). Organic facies D is characterized by the deepest part of the basin where siliceous microfossils (mainly Radiolarian-derived) are most abundant. The presence of Radiolaria in the shallow end of the basin may be associated with areas of upwelling. Organic facies B and C(Type (II and II/III) are located in intermediate depths and shallow part of the shelf/platform region of the basin while OF A (Type I) represent the alginite rich deeper part of the basin. Period of marine regression and transgression also influenced the organic facies distribution in the basin.

Publications:

J. Reyes, S. Saad, and L.S. Lane. 2013. Organic Petrology and vitrinite thermal maturation profiles for eight Yukon petroleum exploration wells in Eagle Plain and Liard basins. Geological Survey of Canada Open File 7056.

J. Reyes, S. Saad, and L.S. Lane. 2013. Abstract. Vitrinite Thermal Maturation Profiles of Five Eagle Plains Petroleum Exploration Wells, Yukon, Canada. The 65th Annual Meeting of ICCP and 30th Annual Meeting of TSOP Aug. 25 to Sept. 4, 2013. Sosnowiec, Poland. p.114.

J. Reyes, S. Saad, and L.S. Lane. 2012. Regional Thermal Maturity Trend in Eagle Plain, Yukon. CSPG GeoConvention. Calgary, Alberta, Canada. May 14 - 18, 2012

3.5.2.4 A Standardized Approach to Optical Thermal Maturity Assessment with Application to the Beaufort-Mackenzie Basin, Arctic Canada

D.R. Issler, J. Reyes, Z. Chen, K. Hu, E. Negulic, and L.D. Stasiuk, Shell Canada Limited

Focus Area:

Standardization of Vitrinite Reflectance in Shale Petroleum systems.

Contribution to Program:

This study is part of the GEM program on integrating existing data on basins with new petrologic data to provide new insight about the history of a basin. The findings contributed to the improvement of the overall understanding of the burial and thermo tectonic history of the Beaufort-Mackenzie Basin, Arctic Canada and its oil and gas potential.

Problem**:** A careful examination of new and legacy %Ro data has shown that organic matter recycling is the dominant factor affecting the quality of vitrinite reflectance measurements for the Beaufort-Mackenzie region.

Approach: The data were quality-checked by comparison with complementary thermal maturity indicators such as Rock-Eval Tmax, liptinite reflectance, and the degree of apatite fission track annealing, and were quality-ranked on a scale of "A" to "C", depending on the amount of information available for each set of analyses (e.g. petrographic descriptions, raw measurements, etc.). Where possible, raw legacy measurements were reinterpreted based on the analysis or reanalysis of samples in the same wells or in the same stratigraphic successions of nearby wells. The "cleaned-up" %Ro data have been used to map regional maturity trends across the basin. Preliminary analysis shows that thermal maturity for a given depth decreases northward from onshore exhumed areas along the southern basin margin to offshore, rapidly-deposited Cenozoic successions beneath the Beaufort Sea. These data are valuable for ongoing petroleum assessment studies and for constraining basin thermal modelling.

Achievement:

New and legacy percent vitrinite reflectance (%Ro) data were compiled by the Geological Survey of Canada (GSC) for approximately 80 wells in the Beaufort-Mackenzie Basin. Legacy %Ro data from different sources have been reinterpreted and integrated with new measurements to provide a standardized set of quality-assessed %Ro data. Complementary paleotemperature-sensitive indicators (e.g. Rock-Eval Tmax, liptinite reflectance, degree of apatite fission track annealing) aided in the quality assessment of the %Ro data. These %Ro data are consistent with regional structural, depositional, and erosion trends and they provide a useful paleotemperature framework for petroleum systems studies and basin thermal modeling. The maturity evaluation method presented here could prove useful in the study of other sedimentary basins with diverse data sources. We have attempted to standardize the data obtained from different sources based on what we have learned about organic recycling and other factors that affect the quality of the measurements.

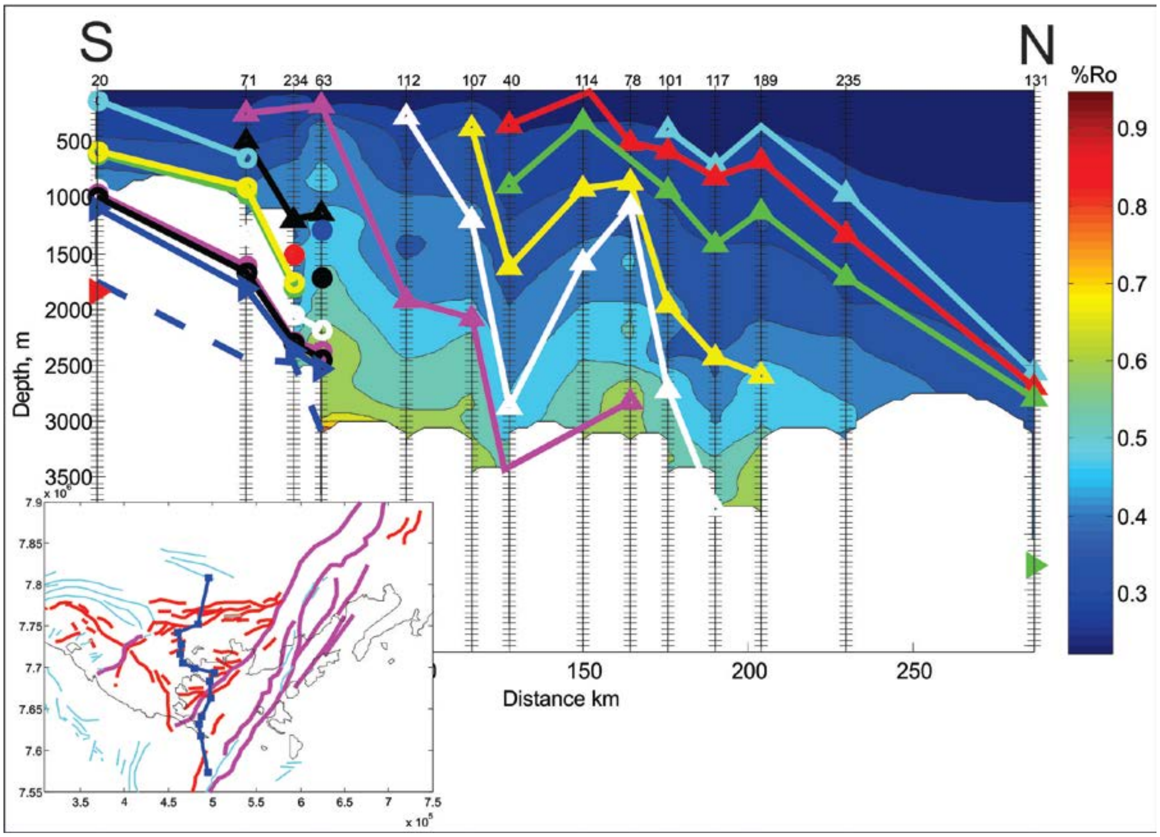

Figure 3.5.2.4-1 North-south cross-section through Beaufort-Mackenzie Basin (blue line on inset map) showing %Ro trends with respect to various stratigraphic successions (coloured lines on cross-section).

Publications:

D.R. Issler, J. Reyes, Z. Chen1, K. Hu, E. Negulic, A.M. Grist, L. Stasiuk, (2013). A Standardized Approach to Optical Thermal Maturity Assessment with Application to the Beaufort-Mackenzie Basin, Arctic Canada. GEOLOGICAL SURVEY OF CANADA, OPEN FILE 6978.

D.R. Issler, J. Reyes, Z. Chen1, K. Hu, E. Negulic, A.M. Grist, L. Stasiuk; F. Goodarzi; (2012). Thermal history analysis of the Beaufort-Mackenzie Basin, Arctic Canada in, Proceedings of the 32nd Annual GCSSEPM Foundation Bob F. Perkins Research Conference; Rosen, N C (ed.); Weimer, P (ed.); Coutes dos Anjos, S M (ed.); Henrickson, S (ed.); Marques, E (ed.); Mayall, M (ed.); Fillon, R (ed.); D'Agostino, T (ed.); Saller, A (ed.); Campion, K (ed.); Huang, T (ed.); Sarg, R (ed.); Schroeder, F (ed.); 2012; p. 609-641; 1 DVD (ESS Cont.# 20110324)

D.R. Issler, J. Reyes, Z. Chen1, K. Hu, E. Negulic, A.M. Grist, L. Stasiuk (2012). A Standardized Approach to Optical Thermal Maturity Assessment with Application to the Beaufort-Mackenzie Basin, Arctic Canada. CSPG GeoConvention 2012, Calgary, Alberta, Canada. May 14 - 18, 2012

3.5.2.5 Interlaboratory Study to Establish Precision Statements for ASTM [ASTM D7708], Test Method for Microscopical Determination of the Reflectance of Vitrinite Dispersed in Sedimentary Rocks

Hamed Sanei and Julito Reyes. Petrology Lab Facility, Calgary
Paul C. Hackley. - U.S. Geological Survey, Reston, Virginia, USA

Focus Area:

Standardization of Vitrinite Reflectance in Shale Petroleum systems.

Contribution to Program:

Participating is this working group and interlaboratory studies are important in maintaining and improving the quality of the data and information's the GSC SLN Organic Petrology Lab-Calgary provides to all the stake holders that uses our services (Programs like GEM, GNES, PERD; Academia, Provincial and Territorial partners, and Industries).

Problem: Lack of standardized method identification, measurement and reporting.

Approach: Provide standardized identification, measurement and reporting of all available information's to end user from academia, government decision makers and private company. Information's that is comparable to all laboratory purveyor of organic petrology analysis.

Achievement:

Since 2008, the GSC SLN Organic Petrology Lab has been participating in working group to identify primary vitrinite in shale with participants from more than twenty governments, private and academic laboratories from around the world. The group is comprised of members from The Society Organic Petrology (TSOP) and The International Committee for Coal Science and Organic Petrology (ICCP). This working group led to the interlaboratory study with a goal of to establishing precision statements for ASTM D7708, Test Method for Microscopical Determination of the Reflectance of Vitrinite Dispersed in Sedimentary Rocks.

To date, four ballots were prepared for ASTM D7708 passed the ASTM D05.28 petrography subcommittee ballot with no negative votes or comments. These ballots will next be voted on at the main D05 Coal and Coke Committee meeting. Should they pass the main committee vote they will be included in the 2014 version of the standard.

ACKNOWLEDGEMENTS

The report is a compilation of input from across SLN and would not have been possible without the collaboration of all members of the Network. Special thanks are given to the five lab leaders (Simon

Jackson, Sandy McCracken, Jeanne Percival, Bill Davis and Andy Mort) who helped to compile the information and review parts of the report. Mike Parsons provided a critical review. Michelle English helped in the organization and editing of the report. A special thanks is given to Rainy Nguyen for her support to all aspects of the report.

APPENDIX 1: SCIENCE LABORATORY NETWORK ORGANIZATION CHART