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**Regional-scale lake-sediment sampling and analytical  
protocols with examples from the Geological Survey of  
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**J.E. Bourdeau and R.D. Dyer**

**2023**

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# **Regional-scale lake-sediment sampling and analytical protocols with examples from the Geological Survey of Canada**

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**2023**

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# Regional-scale lake sediment sampling and analytical protocols with examples from the Geological Survey of Canada

## Abstract

Regional-scale lake sediment surveys have been successfully used since the 1970s as a means for reconnaissance geochemical exploration. Lake sediment sampling is typically performed in areas with a lack of streams and an overabundance of small-sized ( $\leq 5$  km across) lakes. Lake sediments are known to have major, minor and trace element concentrations that reflect the local geology. Overall, surveys are planned and conducted following four distinct stages: 1) background research, 2) orientation survey, 3) regional survey, and 4) detailed survey. At the Geological Survey of Canada, samples are usually collected from a helicopter with floats. Sample density ranges from 1 sample per 6 to 13 km<sup>2</sup>. Samples are collected from the centre of the lake using a gravity torpedo sampler which corresponds to a hollow-pipe, butterfly bottom-valved sampler attached by a rope to the helicopter. Collected sediment samples are then placed in labelled bags and left to air dry. Detailed field notes and additional samples (field duplicates), for the purpose of a quality assurance and quality control program, are also taken. Samples are then milled and sent to analytical laboratories for element determination. Commonly used analytical methods include: X-ray fluorescence (XRF), atomic absorption spectroscopy (AAS), inductively coupled plasma-atomic emission spectrometry (ICP-AES) and -mass spectrometry (ICP-MS), instrumental neutron activation analysis (INAA), and/or determination of volatile compounds and organic carbon using Loss on Ignition (LOI). Analytical data is first evaluated for quality (contamination, accuracy and precision). Numerous options for the analysis of lake sediment data exist, ranging from simple basic element concentration maps and statistical graphical displays with summary statistics, to employing multivariate and machine learning methodologies. By adopting the set of guidelines and examples presented in this report, scientific researchers, exploration geologists, geochemists and citizen scientists will be able to directly compare lake sediment datasets from anywhere in Canada.

## Résumé

Les levés de sédiments lacustres à l'échelle régionale ont été utilisés avec succès depuis les années 1970 comme moyen d'exploration géochimique de reconnaissance. L'échantillonnage des sédiments lacustres est généralement effectué dans des zones dépourvues de cours d'eau et présentant une surabondance de lacs de petite taille (5 km de diamètre). Les sédiments lacustres sont connus pour avoir des concentrations d'éléments majeurs, mineurs et traces qui reflètent la géologie locale. Dans l'ensemble, les levés sont planifiés et réalisés en quatre étapes distinctes : 1) recherche de fond, 2) levé d'orientation, 3) levé régional, et 4) levé détaillé. À la Commission géologique du Canada, les échantillons sont habituellement prélevés à partir d'un hélicoptère muni de flotteurs. La densité d'échantillonnage varie de 1 échantillon par 6 à 13 km<sup>2</sup>. Les échantillons sont prélevés au centre du lac à l'aide d'un échantillonneur à torpille gravitaire qui correspond à un tube creux avec une vanne de fond papillon attaché par une corde à l'hélicoptère. Les échantillons de sédiments recueillis sont ensuite placés dans des sacs étiquetés et laissés sécher à l'air libre. Des notes de terrain détaillées et des échantillons supplémentaires (doubles de terrain), aux fins d'un programme d'assurance et de contrôle de la qualité, sont également prélevés. Les échantillons sont ensuite broyés et envoyés aux laboratoires d'analyse pour la détermination des éléments. Les méthodes analytiques couramment utilisées comprennent : la fluorescence aux rayons X (XRF), la spectroscopie d'absorption atomique (AAS), la spectrométrie d'émission atomique à plasma à couplage inductif (ICP-AES) et la spectrométrie de masse (ICP-MS), l'analyse instrumentale par activation neutronique (INAA), et/ou la détermination des composés volatils et du carbone organique à l'aide de la perte au feu (LOI). Les données analytiques sont d'abord évaluées pour leur qualité (contamination, exactitude et précision). Il existe de nombreuses options pour l'analyse des données sur les sédiments lacustres, allant des simples cartes de concentration d'éléments avec des affichages graphiques statistiques ainsi que des statistiques récapitulatives, à l'utilisation de méthodologies multivariées et à d'algorithmes d'apprentissage automatique. En adoptant l'ensemble de lignes directrices et d'exemples présentés dans ce rapport, les chercheurs scientifiques, les géologues d'exploration, les géochimistes et les scientifiques citoyens pourront comparer directement les ensembles de données sur les sédiments lacustres de n'importe quel endroit au Canada.

# 1 Introduction

Mineral exploration is a high-risk enterprise, with odds of success often quoted to be as low as 1:1,000 (Govett, 1983). Therefore, efforts to reduce risks and enhance chances of success are highly desirable. One such effort is to conduct a well-planned geochemical exploration survey. Geochemical exploration surveys are not only limited to mineral exploration, but can also be conducted for environmental and geological reasons to encourage economic development. Sampling is a process that occurs at the beginning of the data lifecycle, during data generation. Its design is shaped by downstream requirements that were iteratively engineered during data usage, regarding the characteristics of data that include notions of accuracy, abundance, spatial variability, etc. As such, existing downstream requirements for geochemical survey, and therefore, all sampling processes were shaped by the traditional usage scenario of mapping.

Stream sediment and till sampling have been used effectively for decades to rapidly survey regions in an attempt to outline potential areas for further work (Plant et al., 1989; Cameron, 1994; McClenaghan et al., 2020). However, some regions have an insufficient abundance of active streams to support stream sediment sampling. An example of such a region is the Canadian Shield. Overall, the Canadian Shield has poor drainage, a low topography and high water table, caused by from multiple warm (interglacial) and cold (glacial) cycles during the Quaternary Period. Especially, glacial activity by Laurentide Ice Sheet during the Last Glacial Period (c. 115,000 to 11,700 years ago) created an overabundance of small-sized lakes. Terrains similar to the Canadian Shield occur in the northern hemisphere. For these regions, the cost-effectiveness of sampling lake sediments can be significantly better than alternatives, such as glacial till (Davenport et al., 1997). Furthermore, several studies have demonstrated that lake sediment samples can be an effective proxy for glacial till (Bajc and Hall, 2000; Rencz et al., 2002). As a result, lake sediment sampling as a method of geochemical exploration was developed in the late 1960s with the first regional-scale reconnaissance surveys conducted in the early 1970s (Hornbrook, 1987; Friske, 1991; Cameron, 1994; McCurdy et al., 2014).

Lake sediment surveys are generally conducted for geochemical reconnaissance purposes (Friske, 1991; Cameron, 1994). Similar to stream sediment and till surveys, lake sediment surveys are designed to outline promising areas for future work (Cameron, 1994; McClenaghan et al., 2020). It is well known that lake sediments have major, minor and trace element concentrations that reflect the local geology (Coker et al., 1979; Cameron, 1994). From this relationship, lake sediment surveys can be used to target undiscovered mineral deposits (Kerr and Davenport, 1990; Davenport et al., 1997; Grunsky et al., 2014; Zhang et al., 2021a). Even though regional-scale surveys have low sample densities (1 sample per 6 to 13 km<sup>2</sup>), mineral deposits often occur in clusters together with numerous minor occurrences (Govett, 1983; Friske, 1991; Friske and Hornbrook, 1991). Thus, regional-scale lake sediment surveys are often able to identify areas of higher mineral resource potential.

There exists a wealth of publicly-available regional lake sediment data at the Geological Survey of Canada (GSC) and with several provincial geological surveys (e.g., Ontario and Québec). Much of the data collected at the GSC was as part of Federal-Provincial Development Programs (1973-1974), the Uranium Reconnaissance Program (1975-1978) and the Survey's National Geochemical Reconnaissance (NGR) Program (1983-1993) (Hornbrook and Davenport, 1974; Hornbrook and Garrett, 1976; Friske, 1991; Friske and Hornbrook, 1991; McCurdy et al., 2014). Regional geochemical surveys carried out by the GSC, in partnership with provinces and territories cover over 20% of Canada's landmass (McCurdy et al., 2014). Since 2010, there has been an effort to re-analyze existing lake sediment samples in surveyed areas with recognized mineral resource potential (McCurdy et al., 2013). Significant advances in analytical instrumentation, that allow for more elements to be determined at higher precision and with lower detection limits compared to older analyses are providing new insights (Cohen et al., 2010). As of 2023, new regional lake sediment surveys will be conducted in Canada's north under the Geo-Mapping for Energy and Minerals- (GEM) GeoNorth Program (2020-2027). All gathered data (including metadata) are published in open files (GEOSCAN) and archived in a data swamp (a data repository that is unstructured, ungoverned, contains inconsistent metadata, no automated processes and no regular scrubbing; the Canadian Database of Geochemical Surveys; CDoGS). Some of the reports are also available through provincial and territorial portals.

Since the development of lake sediment sampling in the 1970s and the seminal works of [Coker et al. \(1979\)](#); [Hornbrook and Friske \(1987\)](#); [Friske \(1991\)](#); [Friske and Hornbrook \(1991\)](#) and [Cameron \(1994\)](#), there has not been a report describing the planning and methodology required for conducting this type of a survey. This report aims to provide general guidelines to plan and conduct a traditional lake sediment survey. Specifically, we provide foundational knowledge of lake processes, planning and execution of regional-scale lake sediment sampling, preparation and analysis of the samples, and finally evaluation and usage of data. However, we recognize that every survey is unique and may require modifications in planning and execution. We also recognize that technologies, survey design and the field of geochemistry are continuously evolving, and thus, new ideas/concepts, methodologies and instruments may complement and in some cases supplant the ones described here. Along this train of thought, we recognize that the adoption of new, data-driven methods such as artificial intelligence and machine learning, may substantially motivate the rapid evolution of traditional surveying methods and the associated data lifecycle from generation, management to usage. This report is intended for anyone interested in regional geochemical exploration, for which lake sediment surveys are potentially useful and efficacious, by using procedures at the GSC as examples and guidelines. If steps presented in this report are followed, it would be possible to achieve quality results that are fit-for-purpose for traditional downstream uses. Furthermore, this report is primarily intended for compatibility with existing data, by specifying processes that are necessary to generate data that would be analytically and, to the extent possible from a sampling perspective, structurally comparable with other datasets collected from across Canada.

## 2 Survey planning and design

The planning and design of a regional-scale lake sediment geochemical survey will largely depend on the objectives of the survey. Objectives may include, but are not limited to, the: 1) characterization of geochemical background (bedrock), 2) detection of geochemical anomalies (mineral exploration) and/or, 3) characterization of a geochemical baseline (environmental surveys) ([Govett, 1983](#); [Garrett, 1983](#); [Cameron, 1994](#); [Demetrides et al., 2018](#)). At the GSC, the goals of lake sediment surveys are to: 1) conduct and publish results associated with regional geochemical drainage surveys; 2) guide and stimulate mineral exploration; 3) provide geological and environmental baselines; and 4) develop methodologies ([Friske, 1991](#)).

Numerous steps are required, or should be considered, when planning and designing a survey. Regardless of the objective(s), ultimately a survey designer has to balance costs versus the probability of hitting a target ([Garrett, 1983](#)). Therefore, the choice of a (or multiple) target, as well as samples deemed to appropriately represent a population, are critical for conducting an effective survey ([Garrett, 1983](#)). Furthermore, in order to be cost- and time-effective the planning and design of a survey can be divided into 4 stages, namely: 1) background research, 2) orientation survey, 3) regional survey, and 4) detailed survey. In addition, the preparation of a health and safety program should be considered as early as possible when planning and designing a survey.

### 2.1 Stage 1: Background research

The purpose of this stage is to collect as much information and data as possible with the goal of selecting an area for further investigation ([Govett, 1983](#); [Demetrides et al., 2018](#)). If an area of interest has already been adequately sampled, several steps in stages 1 and 2 can be omitted. The information/data to collect can be grouped together by theme and can include, but is not limited to ([Govett, 1983](#); [Garrett, 1983](#); [Friske, 1991](#); [Cameron, 1994](#); [Gomasasca, 2009](#); [Demetrides et al., 2018](#)):

- **Geomatics data:** Satellite images, light detection and ranging (LiDAR), digital elevation models (DEMs), areal photos, topographic maps, geophysical survey data.
- **Previous research:** Reports, geological maps, published articles, industry assessments, NI 43-101 reports (standards of disclosure for mineral projects within Canada).
- **Accessibility:** Political/cultural situation, climate, sanitary conditions, availability of qualified personnel.

- **Policy and Governance:** Governmental policies, laws, historical context, community support, population density, possible stakeholders.
- **Financial:** Governmental/industry support, incentives, pre-existing infrastructure.

## 2.2 Stage 2: Orientation survey

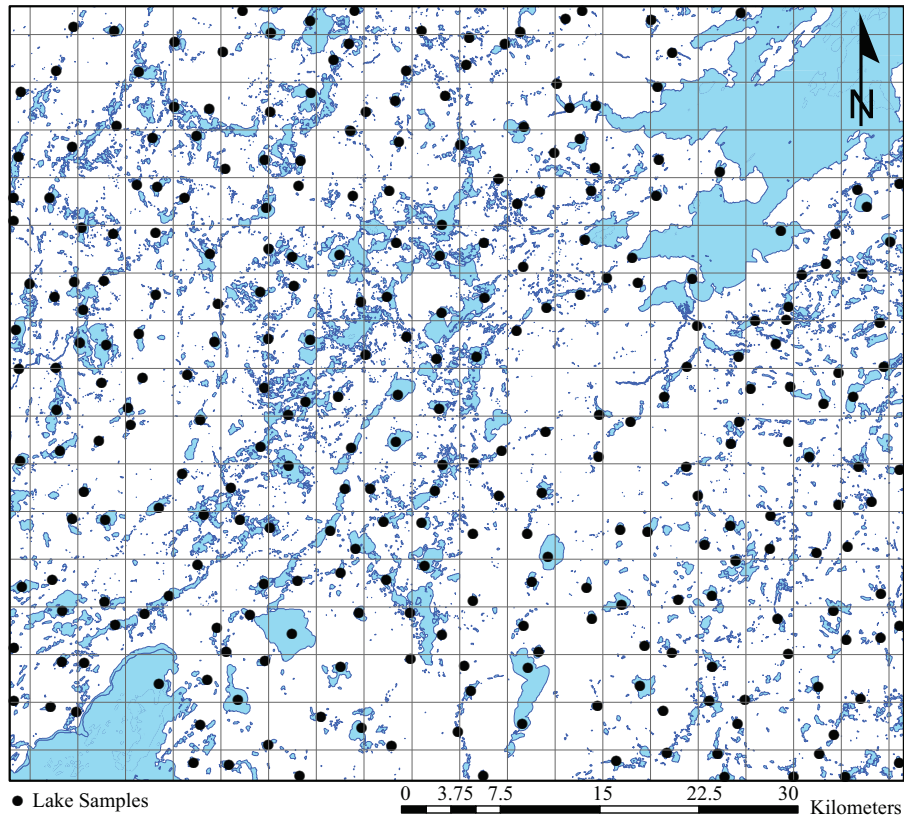
The goal of the orientation survey is to assess the area and, to a lesser extent, find suitable targets for future work (Govett, 1983; Demetrides et al., 2018). It is conducted when first arriving in the area. The purpose of this stage is not to find a potential resource, if the goal of the survey is to conduct mineral exploration. Rather, the orientation survey will help determine parameters to be used during the regional survey (stage 3). Examples of parameters include: what sample medium will be collected (including the size fraction), how to collect samples and at what density, how to prepare and analyze samples, what is the current climate, how has past climate modified the landscape, what are the effects of topography on drainage, what are the dispersion patterns, what is the probability of success/failure of the survey, among others (Govett, 1983; Garrett, 1983; Friske, 1991; Demetrides et al., 2018). Specifically relating to lake sediment sampling, parameters include: do lake bottoms have sufficient material (gyttja), are the landscapes amenable to the dispersion of metals/minerals from bedrock(s) to lake(s), is the area covered by glacial drift and if so is this drift exotic or local, among others (Karrow and Geddes, 1987; Friske, 1991; Cameron, 1994). Towards the end of the orientation survey, the steps to monitor and control sampling (for quality assurance and quality control [QA/QC] program see subsection 4.2) will also be established (Friske, 1991). Sampling at this stage is typically done at a low density of approximately 1 sample per 200 km<sup>2</sup> (Govett, 1983).

## 2.3 Stage 3: Regional survey

Once an area has been assessed (following the orientation survey), selected (depending on the objective of the survey) and the geochemical procedures to be applied have been determined, the regional survey can begin. The goal of the regional survey is to sample the entire area ( $\geq 5,000$  km<sup>2</sup>; Govett, 1983) at an appropriate density to obtain a reliable geochemical representation of it. From this data, the regional geochemical background can be defined and from this anomalous geochemical patterns can be detected (Demetrides et al., 2018). At the GSC, regional-scale lake sediment geochemical surveys are typically planned at the scale of a National Topographic System (NTS) 1:250,000 sheet, and less often at the scale of a NTS 1:50,000 sheet (Friske and Hornbrook, 1991). Lake sediment surveying at the GSC is typically conducted at a density of approximately 1 sample per 13 km<sup>2</sup> (or 1 per 5 miles<sup>2</sup>), but can reach a density of 1 sample per 6 km<sup>2</sup> (Friske, 1991; Friske and Hornbrook, 1991).

Samples in lake sediment surveys cannot be collected in a systematic manner (i.e., evenly spaced grid) due to the availability and/or spatial distribution of lakes. To resolve the issue, sampling is conducted in a stratified random manner (Garrett, 1983). Essentially, an area (e.g., NTS 1:250,000 sheet) is divided into equal-sized cells of a determined size (e.g., 13 km<sup>2</sup>; Fig. 1). A sample site (lake) is then selected within each cell. This manner of conducting the survey makes sampling random as long as the distribution of the lakes is essentially random with no strong underlying structural control. Lake sediment sample sites are deemed representative of a drainage catchment area when metals/minerals, either in solution or as fine particles, can settle within a lake (Friske, 1991; Friske and Hornbrook, 1991). Therefore, lakes with an active inflow and outflow are preferentially selected. Moderately sized lakes, measuring between 1-5 km in length and of depths  $\geq 3$  m depth are preferred since they usually contain more organic-rich sediments compared to smaller-sized lakes (Friske, 1991; Friske and Hornbrook, 1991; Cameron, 1994). Round lakes are also preferable to lakes with many arms as this simplifies sampling. Sampling is typically conducted in centre-lake basins, where: 1) sediment is usually thicker; 2) metal concentrations are higher and more homogeneous; and 3) fine sediment deposition has been focused compared to near-shore (Coker and Nichol, 1974; Cameron, 1980, 1994). If a larger lake is to be sampled, or if a lake completely occupies a cell, then an inlet bay that has a profundal basin or deep bays can be

used instead (Friske, 1991; Friske and Hornbrook, 1991; Cameron, 1994). Larger lakes are avoided since they have a greater inflow/outflow of waters and sediments, diluting some metal concentrations (Cameron, 1994). In addition, larger lakes with greater inflow/outflow are often biased with larger amounts of inorganic sediments, although exceptions occur. Lake sediments with higher contents of inorganic sediment (lower loss on ignition) often report elevated amounts of lithophile elements (e.g., Cr, Li and V) as compared to samples rich in organic matter (higher loss on ignition).



**Figure 1.** Sampling site selection for a regional-scale lake sediment survey. These sample sites are part of a regional-scale survey conducted by the Geological Survey of Canada in 1986 and published by Hornbrook and Friske (1987). The sample sites (lakes in blue colour) are located in north-central Saskatchewan, NTS 074-B and G. Each cell (black line) covers an area of 13 km<sup>2</sup>.

A number of parameters need to be considered when conducting a regional survey. These include, but are not limited to: how samples will be documented (including numbering, GPS coordinates, field observations, photos, etc.); what will be the costs of collecting and preparing samples; how to avoid contamination (e.g., from roads, agricultural activity, etc.); and what will be the total number of samples collected (Garrett, 1983; Sharpe et al., 2019). Additionally, a robust scheme for quantifying data contamination, accuracy, precision and variability is necessary (Geboy and Engle, 2011; McCurdy and Garrett, 2016) (discussed in subsection 4.2). This scheme will undoubtedly include extra samples such as: 1) blank samples (quartzite, volcanic glasses or coarse clean sand), 2) field duplicates, 3) analytical duplicates, and 4) survey control and/or internationally certified reference materials (Geboy and Engle, 2011; Piercey, 2014).

The consistency of individual surveys over time is another important consideration, especially for government-funded surveys. At the GSC, samples are always collected at the centre of lakes, dried, milled, sieved and analyzed for a suite of core elements (Ag, Cu, Co, Fe, Mn, Ni, Pb, U, Zn and others) (Friske, 1991). Furthermore, control reference materials are carried over from previous surveys, which allows for the compatibility of the data between different surveys throughout the years.



## 2.4 Stage 4: Detailed survey

Although this report focuses on regional-scale surveys, detailed surveys ( $\leq 5,000 \text{ km}^2$ ) frequently follow (Govett, 1983; Demetrides et al., 2018). The goal of the detailed survey is to detect anomalous geochemical signatures which may indicate a potential mineral/metal resource, or source of contamination (environmental survey). Of course, the detailed survey is designed based on the results obtained from the regional scale survey, hence it is sometimes referred to as a 'follow-up' survey (Demetrides et al., 2018). It will share many planning and design criteria as the regional survey, but on a larger-scale map. Sampling is typically conducted at every viable lake in the study area, which may result in a density ranging from 1 sample per  $5 \text{ km}^2$  to  $25 \text{ m}^2$  (Govett, 1983; Demetrides et al., 2018), and may involve other types of samples (e.g., till, soil, vegetation, water, streams, etc.).

## 2.5 Health and safety program

Regional-scale surveys are predominantly conducted in remote areas. Thus, the health and safety of all personnel is paramount. To ensure a safe outcome, it is strongly recommended that a health and safety program be developed, or adhered to if adopting an already existing program. Additionally, survey leaders should ensure that all personnel have the appropriate health and safety training (e.g., first aid, predator awareness). Such a program should consider:

- **Health and wellness:** How can we promote a healthy work environment?
- **Workplace inspection:** How is equipment going to be checked?
- **Risk assessment:** What are the expected hazards and how to control or eliminate them?
- **Emergency response:** What is the emergency plan for handling a situation?
- **Accident investigation:** How to document, report and find the root cause of an accident to avoid a potential repeat?

Appropriate personal protection/safety equipment is also important. Personnel should be dressed appropriately with regards to the work environment (field or laboratory). In the field, important items include: a first aid kit, safety glasses, work gloves, emergency drop pack (if being dropped off by a helicopter), signal flag, flares and flare gun, GPS, satellite phone (or other method of communication), life vests and brightly coloured clothing (for visibility).

# 3 Lake sediments, sampling and field equipment

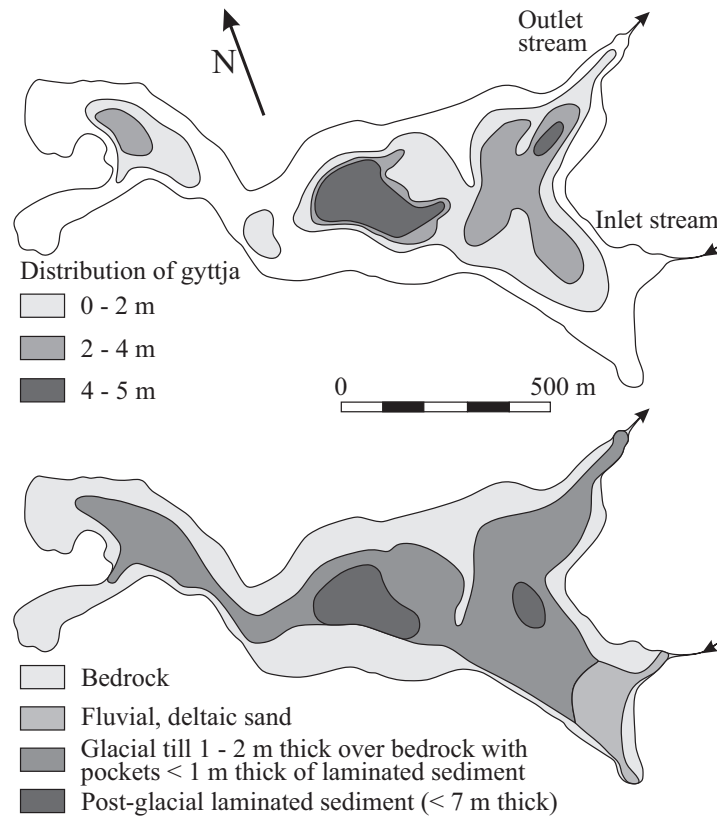
Conducting sampling is the most critical, costly and difficult to repeat portion of any geochemical survey (Demetrides et al., 2018). Consequently, recording field notes and sampling should be conducted diligently. When sampling, the implied expectation is that a sample will be mineralogically and chemically representative to that of the sampled body/area. Biases must be avoided, selected samples must be representative of the feature from which they were sampled or derived from. It is therefore important to understand the origin and formation processes of lake sediments, as well as how to adequately sample them.

## 3.1 Lakes and lake sediments

Lakes are formed by a variety of processes. These can include fluvial mechanisms (e.g., fluvial damming, oxbow, levee lakes), tectonic, volcanic, eolian, chemical, biogenic and/or glacial forces (Wetzel, 2001a). The majority of lakes sampled in regional-scale lake sediment surveys in Canada are of glacial origin (Friske and Hornbrook, 1991; Cameron, 1994). Essentially, ice sheets moving over an area not only level the area, but also locally scour it, resulting in a large number of lakes once the ice retreats. Predominantly scoured areas include

faults, brecciated zones, altered/weathered rocks and/or less-competent lithologies in comparison with country rocks (Larson and Schaeztl, 2001; Wetzel, 2001a). Additionally, glacial-related lakes may form by moraine damming, in depressions that were originally occupied by residual ice and by the local melting of permafrost (Cameron, 1994).

Lakes are inevitably filled by sediments, which in turn, are primarily derived from local substrates including glacial deposits, bedrock lithologies and organic debris (Fig. 2). Usually, the most dominant surficial or bedrock lithology of the area will provide the largest contribution of elements to lake sediments (Hornbrook and Garrett, 1976; Jonasson, 1976; Friske, 1991). Additional factors and processes responsible for the occurrence and distribution of elements into lakes include: degree of chemical and physical weathering, post-depositional processes, sedimentation, glaciation, and contributions from atmospheric, anthropogenic and organic sources (Friske and Hornbrook, 1991; Babeesh et al., 2017; Sahoo et al., 2019). Lake sediments themselves are a mixture of mechanically (e.g., heavy mineral fragments, including Au, Rare Earth Elements [REEs]) and chemically (hydromorphic) dispersed elements (solution precipitation). The proportion and amount of elements either mechanically or chemically dispersed will vary from lake to lake, based on lake inflow/outflow, pH levels, composition of source material, scavenging by hydrous Fe and Mn oxyhydroxides, etc. (Friske, 1991). As a general guide, under oxidizing conditions and at pH levels between 5 to 8, the order of element mobility is: Mo > F, Zn, Ag, U, As, Hg > Mn, Pb, Cu, Ni, Co > Fe (Rose et al., 1979). Both Fe and Mn oxyhydroxides in solution and in organic matter are known to scavenge trace elements, often resulting in anomalously high concentrations. This is however not true for all trace elements and for all localities. Therefore, additional verification of the data, helped by lake water analyses, can help interpret these anomalies.



**Figure 2.** Distribution of pre-glacial (bedrock, fluvial sand), glacial (till) and modern (post-glacial laminated sediments) lake sediments in Turkey Lake, Ontario. Schematic representation modified from Shilts and Farrell (1982).

Of particular interest to mineral exploration, lake sediments can also record the presence of a single or

multiple nearby mineral occurrences and/or ore deposits. Although the size of an ore deposit is small compared to the entire drainage basin, itself defined as an area of land where water collects and drains into a common outlet (a lake), the ore deposit can significantly influence the composition of sediments via variations in trace elements and rapid oxidation of the ore (Friske, 1991; Friske and Hornbrook, 1991). Trace elements such as Cu, Pb, Zn, Ag, Mo, etc. usually occur in greater concentrations (several orders of magnitude compared to country rocks) in several types of ore deposits (e.g., volcanic massive sulphide ore deposits). Lake sediments near these ore deposits will undoubtedly contain a higher concentration of these elements, thus indicating the presence of an ore deposit nearby. Furthermore, ore mineralization is often associated with sulphide minerals, which upon exposure, undergo rapid oxidation, thus releasing these elements for sequestration in lake sediments. However, exceptions exist. For example, ore deposits in the Night Hawk Lake area in the district of Cochrane, Ontario, are covered by impervious glacial clay (Clay Belt) which inhibits oxidation and solution transport (Leahy, 1971). In this particular instance, lake sediment sampling was ineffective as elements associated with ore deposits remain trapped in place by the impervious clay cover.

Most lake sediment surveys are conducted in areas that are topographically relatively flat, preferably in areas with a thin, discontinuous glacial till cover, as opposed to an area characterized by thick/extensive sedimentary sequences (e.g., clay) covering the underlying glacial (i.e., till) and/or bedrock unit(s). Tills are usually composed of silt to boulder-sized fragments of bedrock material that has been mixed and transported some distance by ice, and therefore have a chemical signature that is similar to the underlying bedrock (McClenaghan et al., 2020). As tills are re-worked by precipitations and fluvial transport, and ultimately deposited in lake bottoms, lake sediments will therefore have a chemical signature that mimics that of the tills (Friske, 1991; Cameron, 1994; Bajc and Hall, 2000; Rencz et al., 2002). Thus, it is important to consider the effects of down-ice dispersal of material when interpreting any lake sediment data. This means that a glacial dispersal trend emanating from an ore deposit, for example, could be captured over several lakes, or drainage basins, increasing the chance of discovery. However, some backtracking in previous up-ice directions might be necessary to find the original source (see subsection 6.2 for an example).

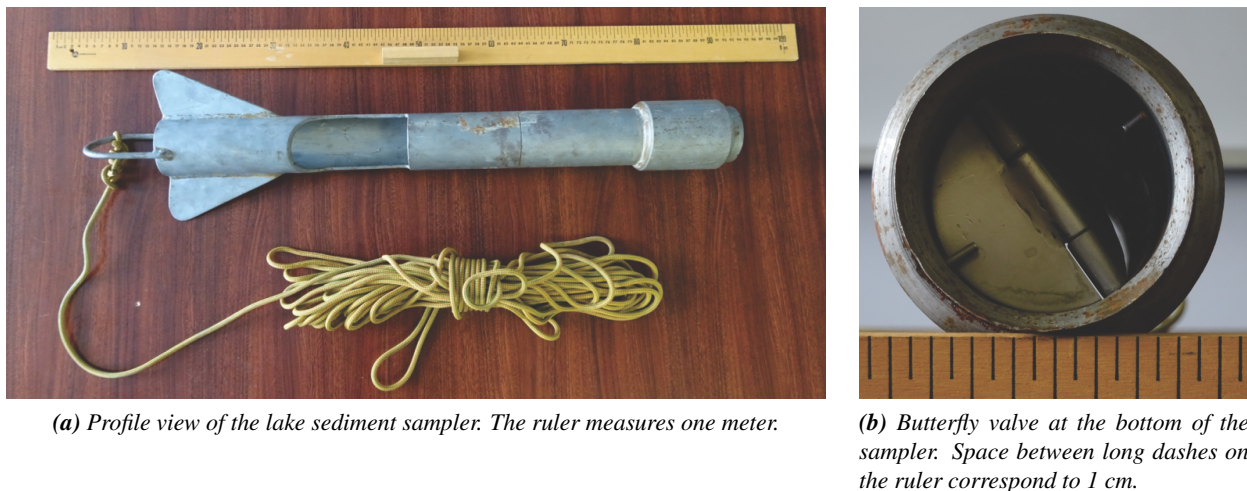
Occasionally, regional bedrock lithologies are covered by exotic glacial material. For example, large areas of the Precambrian Shield in Québec, Ontario and Manitoba are covered by a till enriched with Paleozoic carbonate detritus transported by glaciers from the Hudson Bay Lowlands (Karrow and Geddes, 1987). High amounts of carbonate material can create potential issues such as unexpected till compositions and high lake alkalinity, which can both affect the occurrence and distribution of elements in lakes. Special precautions, such as consulting available maps, tracking regional glacial movements and investigating glacial till composition(s) where lakes are highly alkaline can help identify affected areas, thus helping with future data interpretation.

Lake sediment material can be classified into 3 groups: 1) inorganic sediments, 2) organic sediments and, 3) organic gels (Timperley et al., 1973; Jonasson, 1976; Friske, 1991) (Fig. 2). Inorganic sediments are mixtures of sand, silt and clay with little organic matter. Organic sediments are mixtures of organic gels, inorganic sediments and immature organic debris (leaves, seeds, small twigs). Both inorganic and organic sediments are chiefly found at the inflow/outflow of lakes as well as near lake shores. Lastly, organic gels (gyttja) correspond to mature sediments (clay-sized material with oxyhydroxides of Mn and Fe) that are organic-rich (diatoms, pollen, algae, spore cases and fibrous organic material) and greenish-brown to grey in colour (Hornbrook, 1987). Organic gels are found in less active parts of lakes, typically at depth in their centre. The gels may be underlain by till, pre-glacial sediments, such as fluvial-deltaic sands from past inlets/outlets, or in direct contact with bedrock (Fig. 2). Overall, organic gels are the preferred lake sediment samples because they: 1) have a homogeneous distribution of elements/minerals compared to near-shore sediments; 2) have higher concentrations of elements precipitated from solution compared to near-shore sediments; 3) have consistent volatile element-contents reflecting organic activity which contributed to element precipitation from solution; 4) are situated in a chemically more stable area as compared to near-shore sediments which are subject to seasonal shifts; and 5) are easier to sample from a helicopter (Hornbrook, 1987; Cameron, 1994).



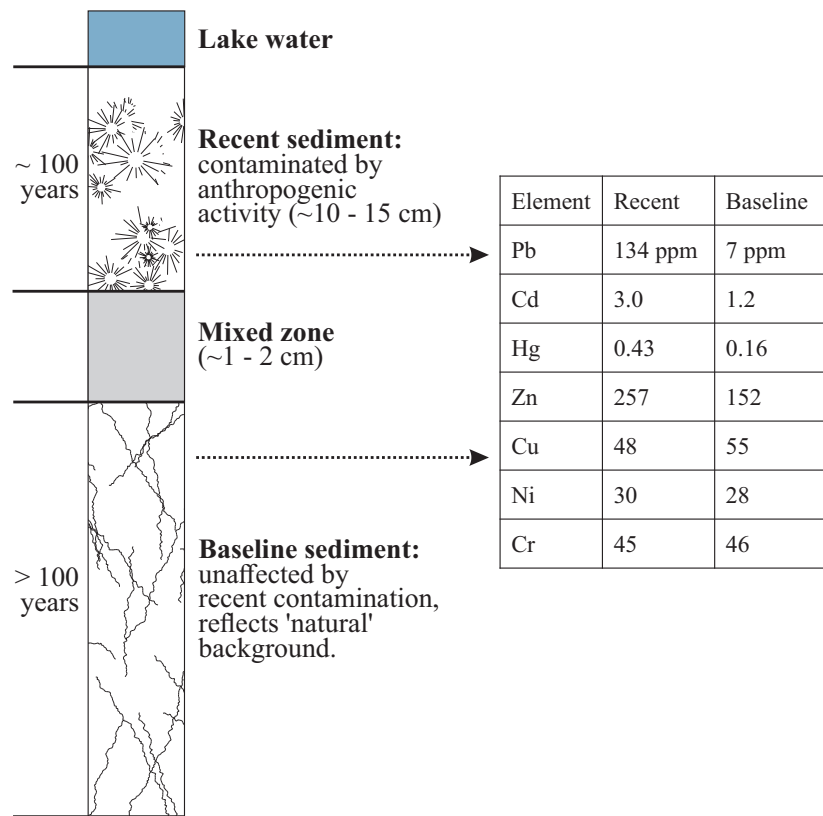
### 3.2 Sampling, equipment and field data collection

Lake sediment samples are usually collected from a helicopter with floats, but samples can also be collected from boats or any other stable sampling platform. It is important that the helicopter/boat remains stationary for the duration of the sample collection. The sampling crew consists of a helicopter/boat pilot, a navigator/data recorder and a lake sediment sampler technician. Samples can be collected using a variety of sediment sampling devices which can be categorized as either gravity, trigger-weight or piston-type (Columbia University, 2022). Most sampling devices are designed for detailed, site-specific research studies and are therefore ill-suited for rapid, regional-scale surveys. Consequently, the GSC developed a gravity torpedo sampler, also known as the Hornbrook sampler, in the 1970s, the design of which is still currently used (Garrett et al., 2008). The device itself is a hollow-pipe, butterfly bottom-valved sampler that is attached by a rope to an external winch on the fuselage of the helicopter (Fig. 3) (Friske, 1991; Friske and Hornbrook, 1991; Davenport et al., 1997). Variations of this sampler have been developed by other Canadian provinces and exploration companies (Dyer and Hamilton, 2007). The gravity sampler is dropped vertically into the lake waters where it sinks to the bottom of the lake and, in most cases, penetrates the soft lake bottom sediment. The device itself weighs 7.1 kg, collects a 30-35 cm sediment section (e.g., Fig. 4), corresponding to  $\sim 1.2$  kg of lake sediment (Cameron, 1994). The sampling device is then pulled up to the surface with the butterfly valves extending as the sampler is pulled up, thus preventing the escape of lake sediments (Fig. 3). The material is retrieved by inverting the sampling device, then inserting a plastic scoop into the bottom port followed by thumping on the device with a wood mallet/plank. The upper 10 - 15 cm of the lake sediment column is usually discarded as it could contain nodules, crusts and/or record anthropogenic activities (Johnson et al., 1986; Cameron, 1994; Davenport et al., 1997). The sample is then placed in a high wet-strength paper bag, which allows the sample to dry without opening the bag. The sampling device and any related sampling equipment is washed between samples to avoid cross-contamination. Using this sampling method, between 13-15 samples be collected in an hour at a sampling density of 1 sample per 13 km<sup>2</sup> (Friske and Hornbrook, 1991; Cameron, 1994). Lake waters may be routinely collected at all lake sediment sampling sites before obtaining the sediment sample.



**Figure 3.** Lake sediment sampler used at the Geological Survey of Canada. The device weighs 7.1 kg and can collect a 30-35 cm lake sediment section, corresponding to a weight of  $\sim 1.2$  kg of lake sediment. Photographs by S.J.A. Day. NRCan photos 2023-015 and 2023-016, respectively.

It is inevitable that some issues will be encountered while sampling. For example, if the intended lake site does not provide safe landing, an adjacent area can be selected rather than leaving the area unsampled. Another common issue is that of contamination, which should be minimized. If the sampler has been in contact with fuel or oil, hands should be washed thoroughly before sampling. Additionally, the sampler should refrain from



**Figure 4.** Simplified lake sediment section. The section includes trace-element data from Turkey Lake in north-western Ontario. Figure modified from [Davenport et al. \(1997\)](#). Trace element data from [Johnson et al. \(1986\)](#).

wearing jewellery and/or watches during sample collection ([Sharpe et al., 2019](#)). Contamination from residential or agricultural areas is best avoided by collecting upstream from dams, reservoirs and/or pumping stations ([Friske and Hornbrook, 1991](#); [Sharpe et al., 2019](#)). At bridges or railway crossings, sampling should be conducted at least 50 m upstream to avoid contamination from roads, exhaust, culverts, road dust, etc. Contamination from mining activity (wind-blown dust, tailing materials) is best avoided by sampling as far away as possible. Another issue that can be encountered is called the ‘particle sparsity’ or ‘nugget effect’, which relates to the presence of coarse grains in a sample ([Govett, 1983](#); [Dominy, 2014](#)). This effect is particularly prevalent for Au and Platinum Group Elements (PGEs), as these elements tend to occur as coarse grains or nuggets, and similarly for heavy minerals containing Cr, Sn, W, REEs, etc. The inherent unique properties of Au, PGEs, and heavy minerals due to their high specific gravity (density) can lead to heterogeneous distribution in any surficial media ([Friske, 1991](#); [Dyer and Barnett, 2007](#)). The presence of coarse grains in a small sample can therefore skew the results, leading to anomalies that should be investigated. To mitigate against the nugget effect, a large sample should be collected. [Govett \(1983\)](#) calculated that for lake sediments, a sample should weigh no less than 8 grams (after drying and sieving at the -80 mesh [177  $\mu\text{m}$ ]) to minimize the nugget effect. It should be noted that the nugget effect is unavoidable, but organic-rich lake sediments seem less susceptible to the issue as compared to more inorganic-rich lake sediments. Additionally, in the case of Au or PGEs exploration, other elements that do not form coarse grains (e.g., As, Mo, Sb, Hg, W, Cu, Ni and U) can be used instead ([Hornbrook, 1987](#); [Friske, 1991](#); [Dyer and Barnett, 2007](#)). However, the presence/absence of other elements (listed above) will greatly depend on the mineralogical assemblage of the mineral deposit.

Recording detailed field notes is essential during sample collection. Data collected typically includes: position (GPS coordinates), sample description, sample depth, size of lake, nearby bedrock lithology, vegetation

present, photographs, etc. At the GSC, field cards were specifically designed and are described in [Garrett \(1974\)](#) and [Friske and Hornbrook \(1991\)](#) to support lake sediment surveys (Fig. 5). Each card contains a number of columns and rows. The first 12 columns contain the alpha-numeric sample number, e.g., 074A\_\_891176. The first alphanumeric group, 074A corresponds to the 1:250,000 NTS sheet number. When a survey is conducted at the scale of an NTS 1:250,000 sheet, 2 columns are left blank following the alpha-numeric group. However, when a survey is conducted at the scale of an NTS 1:50,000 scale sheet, the alpha-numeric group will include the number of the 1:50,000 sheet in the two columns that were left blank (e.g., 074A04). The remainder of the sample number includes the year (columns 7-8), field crew number (column 9) and the number of samples collected by an individual in the current year (columns 10-12). The use of a crew number allows for up to 4 samplers. Typically, odd numbers (1, 3, 5, 7) indicate lake sediment samples whereas even numbers indicate lake water samples. Next, the UTM zone is recorded in columns 13-14. In Canada UTM zones range from 07-22. The easting (6-digit) and northing (7-digit) are measured in meters and recorded in columns 15-20 and 21-27, respectively. Nearby rock lithology is also recorded using a 4 character mnemonic (e.g., Granite = GRNT) in columns 28-31. However, recording the rock lithology is subjective since oftentimes there are no outcrops that can be observed from the lake. The surface area of the lake is recorded in columns 32-35, the water depth, in meters, in columns 36-38. If the sample is used as a field duplicate, it is indicated in columns 39-40. Other parameters that can be recorded include local topographic relief, composition of the sample, sediment colour, suspended matter and other user definable columns if need be.

**Figure 5.** Paper-based lake sediment survey field card previously used at the Geological Survey of Canada. These cards are now discontinued in favor of directly digitally recorded data.

Nowadays, field notes are recorded digitally. Digitally recording information provides numerous advantages including simultaneously recording coordinates and taking photographs. Screen shots of the digital field notes recording system used at the GSC are presented in Figure 6.

After each work day samples should be checked to make sure that their numbering is correct, that they match with the field cards/notes and that the field cards/notes have been completed correctly. Sample bags should also be checked to make sure that they are not perforated. Sample numbers and field data should subsequently be recorded into a computer management system for ease of tracking and to avoid potential issues, such as the loss/destruction of field cards/notebook. A procedure for regular back-ups (hard drives or cloud storage) should be established. Field data as recorded in the computer management system should also be periodically updated as tasks in the survey are completed. A master list of sample identification numbers should also be created to track the progress of sample preparation and analysis, and to ensure QA/QC samples are correctly inserted.

2015 NGR Lake Survey - 075B

NGR Lake Survey  
NORTHWEST TERRITORIES  
GEOLOGICAL SURVEY

Gyttja  GSC  OGS  Surface (~0.5m)  Bottom  m

Routine  FD1  BD  
 NULL  FD2  CR

Site Characteristics **Sediment Sample** Water Sample Site Photo Detail Photo

Get Location Data  
60.006380  
-106.043692  
15/07/2015  
03:26:56

Navigators: HF, MW

Field Comment: >>Field Comment<<

NTS Sheet: 075B, Sample #: 2015, Target: 1002, Unique ID: 075B\_2015\_1002

New Sample

(a) First tab which records sampling site characteristics, such as sample number, coordinates, lake area, topography, possible contamination, etc.

2015 NGR Lake Survey - 075B

NGR Lake Survey  
NORTHWEST TERRITORIES  
GEOLOGICAL SURVEY

Gyttja  GSC  OGS  Surface (~0.5m)  Bottom  m

Routine  FD1  BD  
 NULL  FD2  CR

Site Characteristics Sediment Sample **Water Sample** Site Photo Detail Photo

Top Sample ID: 075B-2015-1002TS

Sediment Colour: Dark brown

Sediment Odour: [Input Field]

Top Sediment

Sediment Composition

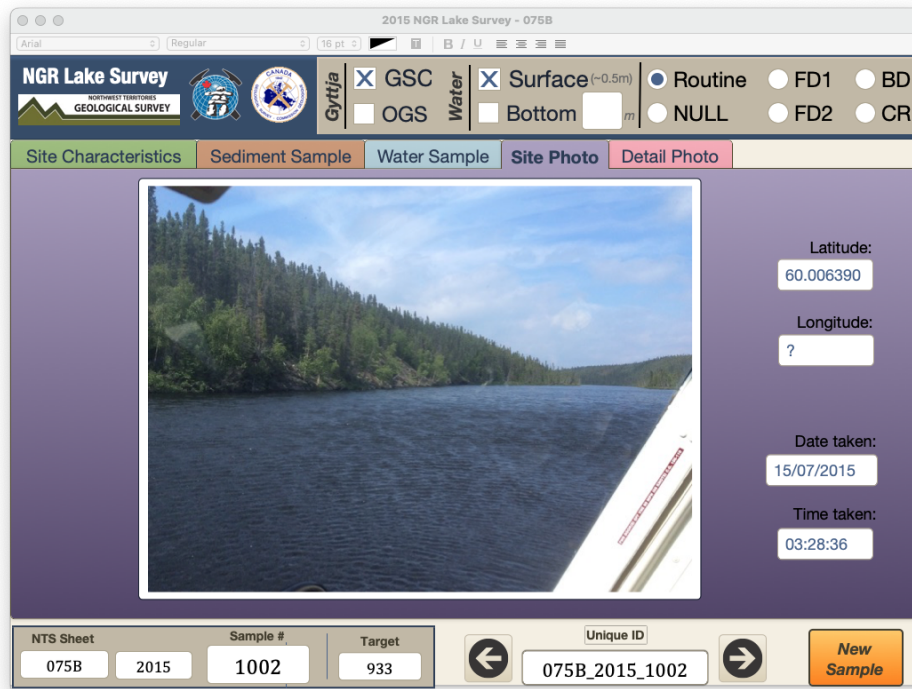
Sand	Silt & Clay	Organics	Gyttja
0	0	0	100

NTS Sheet: 075B, Sample #: 2015, Target: 1002, Unique ID: 075B\_2015\_1002

New Sample

(b) Second tab which records characteristics of the lake sediment sample, such as colour, smell and composition.





(c) Fourth tab which records a site photo. The third tab (not shown) is used to record lake water parameters and samples. A fifth tab (not shown) records a photo of the lake sediment sample.

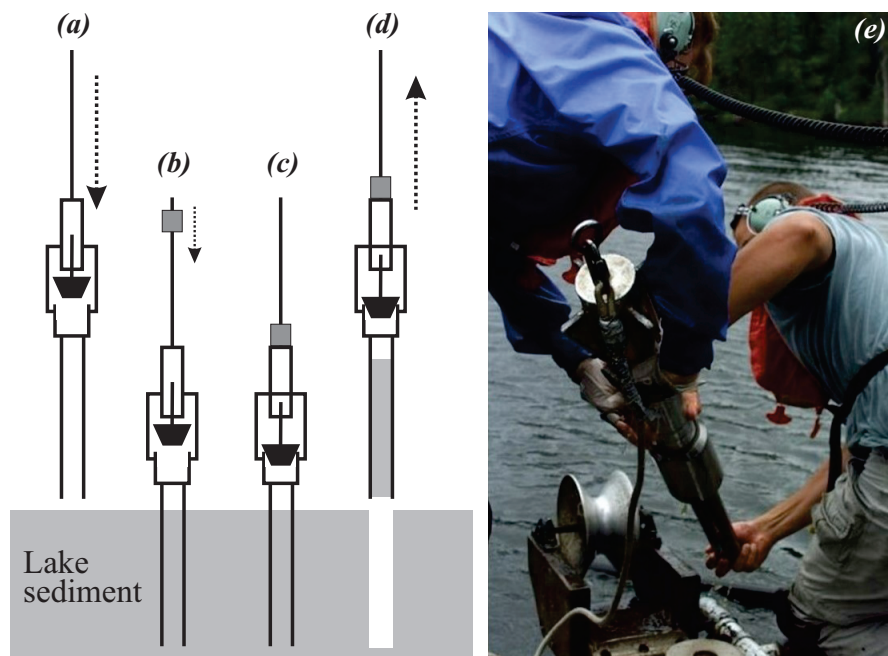
**Figure 6.** Digital lake sediment field record currently used at the Geological Survey of Canada.

### 3.3 Lake sediment cores

Although not a typical component of regional-scale lake sediment sampling surveys, lake sediment cores can also be considered and collected. Lake sediment samples and lake sediment cores differ in that samples contain mixed sediments (lost stratigraphic/sedimentary succession), whereas cores have preserved stratigraphic/sedimentary succession. The following is intended as a simplistic overview of the use, collection, analysis and interpretation of lake sediment cores. Additional information is provided by [Last and Smol \(2001a,b\)](#); [Smol et al. \(2001a,b\)](#); [Wetzel \(2001b\)](#); [Van Metre et al. \(2004\)](#); [Douglas \(2013\)](#), and references therein.

The coring of lake sediments begun in the 1970s and was strongly enabled by the application of radioactive isotope dating ([Chow et al., 1973](#); [Goldberg et al., 1976, 1977, 1978, 1979](#)). Lake sediment cores can be used to monitor concentrations of metals and metalloids (e.g., Pb, Hg, Zn, Cr, Cd, Cu, As, etc.), organic matter and/or man-made organic contaminants (e.g., fossil fuel, industrial activity, sewage, dioxins, polychlorinated biphenyls [PCBs], etc.) that accumulated in a lake through time ([Valette-Silver, 1993](#); [Wetzel, 2001b](#); [Stern et al., 2009](#)). Lake sediment cores can also be used to reconstruct human activity in a particular area. Many types of coring devices have been developed, depending on the depth of the water, the length of the desired sample and the nature of the lake bottom material. At its most basic, a core sampling device consists of a steel, plastic or glass tube (Fig. 7). Several lake sediment core sampling devices have been designed by the academic, governmental and private sectors ([Cavanagh et al., 1994](#); [Van Metre et al., 2004](#); [Dyer and Hamilton, 2007](#); [Mingram et al., 2007](#); [Douglas, 2013](#); [U.S. Environmental Protection Agency, 2020](#)).

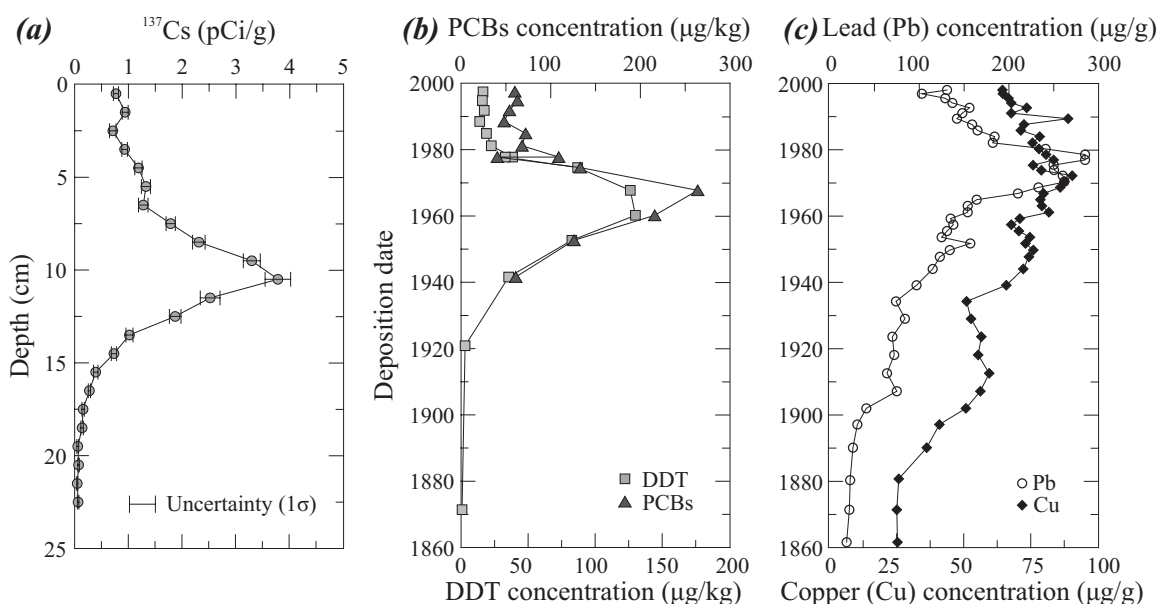
Lake sediment cores are preferably extracted from the centre of a lake in order to avoid flocculent layers (individual, minute, suspended particles held together into clot-like masses) and sediment disruptions ([Van Metre et al., 2004](#); [Stern et al., 2009](#)). Collected cores, optimally frozen first, can then be sliced into intervals (e.g., 0.5 - 1 cm) with the material from each slice placed in a labelled plastic bag and sealed. Material is then dried (preferably freeze-dried) and stored at low temperatures (<5°C), in the dark, prior to isotopic dating and/or



**Figure 7.** Example of a lake sediment gravity corer. Schematic diagrams modified from Douglas (2013). (a) The gravity corer with an open core breach is lowered to the bottom of the lake and penetrates the lake sediments. (b-c) Once at a sufficient depth within the lake sediments, a weighted messenger is dropped along the coring line to activate a plunger that seals the top of the core. (d) Sealing the top of the core creates suction that holds the lake sediments within the sampling tube. The gravity corer is then pulled back to the surface for the extraction of the core. (e) Retrieval and extraction of a lake sediment core. Courtesy of the Ontario Geological Survey.

contaminant analysis. Material from each interval can also be used to conduct grain-size, mineralogical and biological (zoology, biomarkers) analyses (Douglas, 2013). Isotopes commonly used for dating lake sediment cores include:  $^{228}\text{Th}$  ( $\lambda = 1.91$  yr),  $^{210}\text{Pb}$  ( $\lambda = 22.3$  yr),  $^{137}\text{Cs}$  ( $\lambda = 30$  yr),  $^{21}\text{Si}$  ( $\lambda = 276$  yr),  $^{226}\text{Ra}$  ( $\lambda = 1620$  yr),  $^{14}\text{C}$  ( $\lambda = 5730$  yr),  $^{239}\text{Pu}$  ( $\lambda = 2.4 \times 10^4$  yr),  $^{240}\text{Pu}$  ( $\lambda = 6.6 \times 10^6$  yr) (Valette-Silver, 1993). Because of their short half lives ( $\lambda$ ),  $^{228}\text{Th}$ ,  $^{210}\text{Pb}$  and  $^{137}\text{Cs}$  are the most popular isotopes used to date recent lake sediments. Lakes chosen for chronostratigraphic study should not be overly acidic as there is a possibility that some isotopes ( $^{210}\text{Pb}$  and  $^{137}\text{Cs}$ ) will be re-distributed through the sediments and possibly liberated into the lake waters (Valette-Silver, 1993).

Previous studies using lake sediment cores have highlighted a global increase in contamination caused by anthropogenic activity, as denoted by obvious increases in trace metal (Pb, Zn, Cr, Cd, Cu, Ag, V and Mo) concentrations. However, both natural and geological processes may also influence the distribution of trace metals (especially Hg), such that anthropogenic activities can be difficult to ascertain (Rasmussen et al., 1998). Globally, trace metal contamination began in the early 1800s and rapidly increased in concentrations in the 1990s (Katz and Kaplan, 1981; Valette-Silver, 1993; Van Metre et al., 2004) (Fig. 8). Furthermore, a sharp increase in trace metals (especially Pb) is denoted in the 1940s, globally, and reached a climax around the 1960s-1970s. In comparison, the 1970s sediments are seven times more enriched in Pb than sediments from the 1900s, apparently caused by atmospheric deposition of Pb mostly related to the widespread use of leaded gasoline (Chow et al., 1973). Man-made compounds (e.g., dioxins and PCBs) appeared globally in the lake sediments in the early 1940s-1950s, and reached a climax in the 1960s-1970s (Valette-Silver, 1993; Van Metre et al., 2004) (Fig. 8). Since the 1960s-1970s, both trace metal concentrations and man-made contaminants have been steadily decreasing globally.



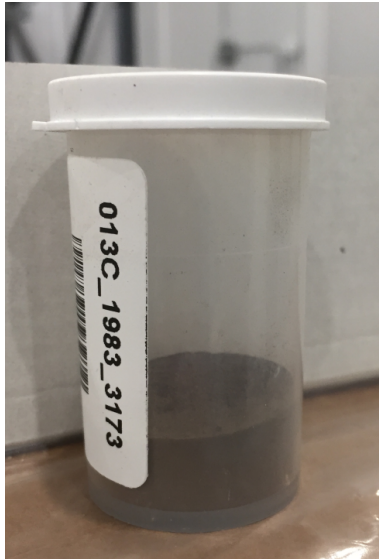
**Figure 8.** Example of chemical constituent profiles in a lake sediment core from Lake Washington (WA), United States. Data from [Van Metre et al. \(2004\)](#). (a) The  $^{137}\text{Cs}$  activity peak at 10 - 11 cm depth was assigned a date of 1964, resulting in a mass accumulation rate of  $0.055 \text{ g}/\text{cm}^2\text{-year}$  for the core. The mass accumulation rate was then used to assign dates to the core. (b) Concentrations of man-made contaminants. Both PCBs, DDT progressively increase from the 1860s-1940s. Concentrations climax around the 1960s-1970s and steadily decrease afterwards. (c) Concentrations in trace metals. Both Pb and Cu progressively increase from the 1860s-1960s. Concentrations climax around the 1970s-1980s and steadily decrease afterwards.

## 4 Preparing samples for geochemical analyses

### 4.1 Sample preparation

Samples are first left to air dry. Drying can be done outside in the sun (no rain) or in a tent with fans at a temperature of  $\leq 40^\circ\text{C}$  for 10 days, or until dry ([Friske and Hornbrook, 1991](#)). A final drying at a laboratory (temperature of  $\leq 40^\circ\text{C}$ ), may be required and is a prudent measure. It is preferable to dry the samples prior to shipping for further processing as this would save on costs as well as reduce the risk of sample bag ruptures. Once samples are dried, they are broken into small fragments. Organic gels tend to become one hard lump when fully dried. Samples can be broken up by pounding on the bag with a wooden striker or mallet. Comminution is typically done using a ceramic mill with either ceramic balls or ring and puck. The milled sample is then sieved through a -80 mesh ( $177\mu\text{m}$ ). The -80 mesh is preferable since sediments are fine enough (clay and fine-grained material) to reduce the amount of 'dilutant' quartz, thus allowing for reliable subsequent element determination ([Fletcher, 1981](#); [Garrett, 2019](#)). Each sample is then placed in a labelled polyethylene container for future use and/or longer-term archiving (Fig. 9). Between each sample, the sieve is cleaned using a paint brush and by blowing with a jet of compressed air. If used, the mill is cleaned with ground quartz ([Friske and Hornbrook, 1991](#)). Furthermore, sieving and milling should be conducted in well ventilated areas as to minimize dust cross-contamination between samples.

Samples ready to be sent to a laboratory for chemical analysis should be clearly labelled and shipped in a secure container. Samples associated with the QA/QC program also need to be prepared and inserted with routine samples prior to being sent off to the laboratory. The container should also be clearly labelled with the name and address of the laboratory and of the sender. A paper of the sample list should be included with the shipment and a digital copy of the sample list can also be e-mailed to the recipient. As an extra precaution,



(a) Individual lake sediment sample archived in a labelled polyethylene container. The container measures about 7 cm in height. Sample photographed is part of the NGR program sample collection.



(b) Collection of archived NGR program lake sediment and stream samples. The samples are packed on skids as they arrived (early 2022) at the new Geological Survey of Canada archive facility in Ottawa, Canada.

**Figure 9.** Archived lake and stream sediment samples at the Geological Survey of Canada. Photographs by J.E. Bourdeau. NRCan photos 2023-017 and 2023-018, respectively.

pictures of samples (with clear labelling) and containers can be taken before shipment.

## 4.2 Quality assurance and quality control (QA/QC) program

A critical part of any survey is the inclusion of a quality management system or program. This report only summarizes aspects of a quality management program that are specifically traditional to geochemical surveys and are captured in in-discipline literature. Therefore, this report explicitly does not consider greater aspects of a quality management framework that include modern data management notions of data accessibility and interpretability (Henderson et al., 2017), catering for transdisciplinary (artificial intelligence, machine learning and geodata science) uses, and such, which are much broader in scope and are undeveloped presently in the geochemical data lifecycle. A properly constructed traditional QA/QC program should identify sources of analytical errors and establish the limitations of the analytical results (Knott et al., 1993; Ontario Securities Commission, 2011; Geboy and Engle, 2011; Piercey, 2014; Vann et al., 2014; Grunsky and de Caritat, 2017). A QA/QC program can monitor for accuracy, precision and fitness-for-purpose of the data, as well as potential contamination.

A robust traditional QA/QC program is even more essential when work is conducted by a third party, such as by a contractor, which is typically the case at the GSC. In such cases, tight control over the survey is necessary to ensure that all pre-established protocols and methods are adhered to. Tight adherence to survey protocols and methods ensures that the data is consistent, reliable and can be used for the construction of a systematic geochemical database, which is the case at the GSC.

Within a QA/QC program a number of different samples are used. At the GSC, blank samples are used to monitor and quantify laboratory contamination. Reference materials which correspond to bulk homogeneous materials prepared at the GSC, are used to monitor analytical drift both within and between survey datasets. Reference materials are samples that have been measured using several analytical methods (e.g., X-ray fluorescence, inductively coupled plasma-mass spectrometry, etc.) and/or in many different laboratories. The results from the



repeat analyses are then given a certified value which is accompanied with an uncertainty. Reference materials can either be ‘in house’ or ‘controlled’, that is, developed by a laboratory, agency or scientist for internal use, or ‘certified’ which is internationally recognized and accompanied by a certificate. Special care should be taken when choosing reference materials. In order for reference materials to be effective, the samples should have a comparable composition, concentration and matrix as the lake sediment survey samples (Thompson, 1983; Geboy and Engle, 2011). Overall, certified reference materials are inserted less frequently to assess accuracy to international standards. Analytical duplicates permit the estimation of precision across the ranges of the data acquired in the survey. The fitness-for-purpose of the data is determined by using field duplicate samples, which allows a comparison between within-site versus between-site variabilities, calculated using the Analysis of Variance (ANOVA). As a general rule, blank and control reference samples should represent between 2-5% of samples, analytical and field duplicates between 10-20% of samples (Reimann et al., 2008; Geboy and Engle, 2011).

To obtain quality results, the GSC has developed a sample grouping design which includes control reference materials, analytical and field duplicate samples. The sample grouping design is explained in detail in McCurdy and Garrett (2016) and presented in Figure 10. Essentially, samples are divided into blocks of 20 samples. Three locations in the block (chosen at random) will correspond to a field duplicate, an analytical duplicate and a control reference material (Friske, 1991; Friske and Hornbrook, 1991; McCurdy and Garrett, 2016). The analytical duplicate, split from a routine sample (chosen at random) is placed as the first sample in the block. Field duplicates are sampled in the field (site chosen at random) whereas both the analytical duplicate and the control reference material are inserted during sample preparation, using their pre-allocated positions (numbers). The analytical duplicate may be prepared from one of the field duplicates, in which case a more insightful ANOVA can be undertaken (Garrett, 2013a).

Sample	Sample type	Sample	Sample type
074A 141001	Analytical duplicate, split from 074A 141005	074A 141011	Routine
074A 141002	Routine	074A 141012	Routine
074A 141003	Routine	074A 141013	Field duplicate 1, routine sample
074A 141004	Routine	074A 141014	Field duplicate 2
074A 141005	Routine and split for analytical duplicate	074A 141015	Routine
074A 141006	Routine	074A 141016	Routine
074A 141007	Routine	074A 141017	Routine
074A 141008	Control Reference Material	074A 141018	Routine
074A 141009	Routine	074A 141019	Routine
074A 141010	Routine	074A 141020	Routine

**Figure 10.** Schematic representation of a sample block used at the Geological Survey of Canada. A sample block is composed of a total of 20 samples, which includes routine, control reference material (or rarely a certified reference material), analytical and field duplicate samples. Whenever possible, the analytical duplicate sample is created from one of the field duplicates which provides a more robust analysis of variance (ANOVA).

Now-a-days numerous statistical software and/or programming languages (e.g., Statistica, GNU PSPP, Statistical lab, Python, Perl, among many others) are available to assist with calculations presented below. At the GSC, a package called ‘rgr’, which uses the R programming language (<https://www.r-project.org/>), allows for semi-automated calculation of QA/QC results (Garrett, 2013b, 2018; McCurdy and Garrett, 2016).

### 4.2.1 Contamination

Blank samples are used to detect and quantify contamination. Contamination can occur at any stage of the sampling, sample preparation and analysis process. Examples of contamination include: poor cleaning of equipment between samples, use of sub-quality equipment, unclean acids during digestion, instrumentation memory effects during analysis, etc. (Geboy and Engle, 2011; Piercey, 2014). Thus, blank samples can be introduced at any stage to monitor and quantify contamination from a suspect source. There are three main types of blank samples. Total procedural blanks (or total analytical blanks) are samples that contain no material (empty bag or vial) or a very clean substitute for the material (i.e., 18.2 M $\Omega$ -cm water), but are processed and analyzed in the same manner as a routine sample. Total procedural blanks are typically used to monitor laboratory contamination. Field blanks are samples of materials containing below detection limit levels of elements to be determined (e.g., quartzite, volcanic glasses, clean sand, etc.) that have been exposed to the same field and transport conditions as routine samples. Field blank samples are used to monitor contamination from field sampling. Finally, trip blanks also contain field blank material but are not exposed to the field environment, they are created during sample transport in order to monitor contamination from transport. At present, neither field or trip blank samples are used at the GSC.

At the GSC, total procedural blanks are used to monitor laboratory contamination. Contamination can be quickly identified using a modified version of Shewart control charts (Piercey, 2014) (Fig. 11). On the X-axis, the blank analyses are presented in the sequence that they were analyzed. On the Y-axis are the results obtained for the element of interest. The mean ( $\bar{x}_i$ ; Equation 1) and standard deviation ( $s_i$ ; Equation 2) can be calculated for each element ( $x_i$ ) among a number of samples ( $n$ ) in the figures. Note that if censored values are halved, as this is common practice with censored values (Sanford et al., 1993; Grunsky et al., 2014), most values will fall below the detection limit in the Shewart control charts (e.g., Fig. 11). Contamination can be confirmed if a single or multiple values are above the lower detection limit and standard deviation. It should be noted that measurement of elemental concentrations at such low concentrations (near or at instrumental detection limits) are plagued by large random variations, as well as a number of other issues (Bernal, 2013). Therefore, some leniency may be required when choosing a cut-off value for a determined element. In most cases, if there are: 1)  $\geq 4$  values in a row which are above the lower detection limit and standard deviation ( $1s$ ), and/or 2) substantial outliers ( $\leq 3s$ ), contamination can be considered significant (Piercey, 2014).

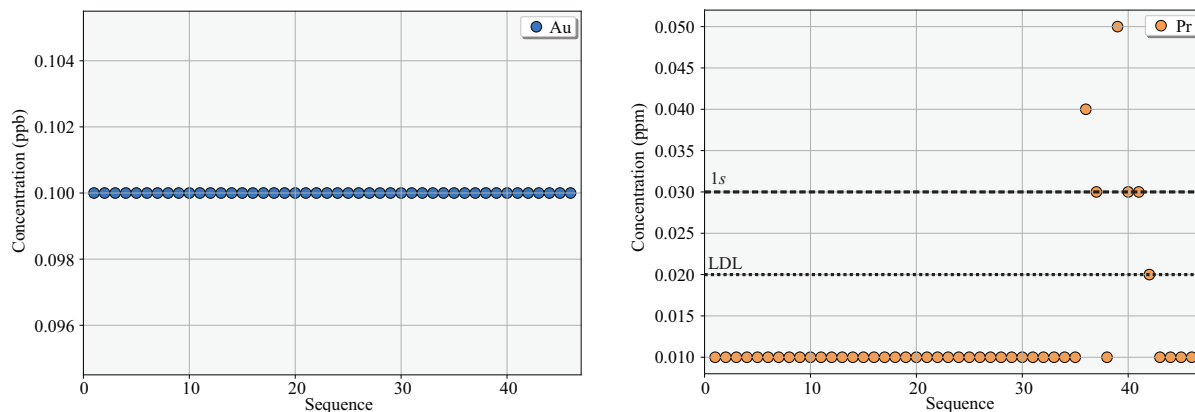
$$\bar{x}_i = \frac{\sum_{i=1}^n x_i}{n} \quad (1)$$

$$s_i = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x}_i)^2}{n - 1}} \quad (2)$$

### 4.2.2 Accuracy

Accuracy describes how close a measured value is to a known or accepted value. In the context of a geochemical survey, the known or accepted value is determined using specific samples known as reference materials. At the GSC, four control (or in house) lake sediment reference materials/samples (LKSD-1 to 4) are used. The re-use of these four control reference samples over numerous surveys allows for direct comparison of analytical datasets. Further information, elemental values and standard deviations are available by contacting (CANMET), or by consulting Lynch (1990, 1999) and Hechler (2013). Unfortunately, some of the control reference materials are exhausted and no longer available. New control reference materials are planned in the near future. The composition of the reference samples is as follows:

- **LKSD-1:** Mixture of lake sediment from Joe Lake (NTS 31F) and Brady Lake (NTS 31M) in Ontario.
- **LKSD-2:** Mixture of lake sediment from Calabogie Lake (NTS 31F) in Ontario and the east arm of the Great Bear Lake in the Northwest Territories (NTS 86K and 86L).



(a) Shewart control plot for Au. Note that all values fall below the lower detection limit (0.2 ppb) and thus contamination is not considered to be an issue.

(b) Shewart control plot for Pr. Note that a few samples are outliers and could indicate potential contamination issues.

**Figure 11.** Representative Shewart control plots to quantify contamination using total procedural blank samples obtained from an analytical facility. Number of samples for each element is 46. LDL = lower detection limit.

- **LKSD-3:** Mixture of lake sediment from Calabogie Lake (NTS 31F) in Ontario and a composite mixture of lake sediments from NTS 64L, 64M, 31M, 31N, 32C, 32D, 41P and 42A.
- **LKSD-4:** Mixture of lake sediments from Big Gull Lake (NTS 31C) in Ontario, Key and Seahorse lakes (NTS 74H) in Saskatchewan.

To quantify accuracy, analyses from reference materials can be compared to the published or accompanying certificate. To do so, the mean (Equation 1), standard deviation (Equation 2) and relative standard deviation (RSD; Equation 3) can be calculated for each element. The RSD provides an indication of the precision of the data and can be expressed as a percent (%). Although a precise cut-off will depend on the nature and goal of the study/survey, in most cases, a RSD <20% is an indication of accurate data, whereas a high (>20%) RSD could indicate accuracy issues.

$$RSD = \frac{s_i}{\bar{x}_i} \times 100\% \quad (3)$$

As an additional measure, the relative error (RE) can also be calculated. The relative error provides a semi-quantitative indication of how close the measured mean ( $\bar{x}_i$ ) is to the accepted one ( $\bar{x}_a$ ). The relative error can be reported in percent (%), as presented in Equation 4. Occasionally, some of the accepted values are decades old, which results in large relative error values. In such cases, the relative error can only be used in a semi-quantitative manner.

$$RE = \frac{|\bar{x}_i - \bar{x}_a|}{\bar{x}_a} \times 100\% \quad (4)$$

### 4.2.3 Precision

Precision is defined as a measure of the reproducibility of a measurement (Piercey, 2014). In the context of a geochemical survey, analytical duplicate samples are used to provide an estimate of precision. An analytical duplicate is created from splitting the material from a routine sample into two or more portions and analyzing these fractions at a laboratory using the same method and equipment, preferably within a short time interval (McCurdy and Garrett, 2016). At the GSC, a sample, preferably one of the field duplicates, is split into 2 (a pair; Fig. 10), with elemental compositions analyzed in one sample ( $x_i$ ) and the other sample ( $x'_i$ ). Preferably, a large

number of pairs are analyzed to minimize the impact of rare errors that may occur. However, analyzing a large number of pairs does not reduce the frequency of random errors. To determine precision, we first calculate the mean ( $\bar{x}_p$ ) and standard deviation ( $s_p$ ) of each elemental composition of all  $n$  samples as:

$$\bar{x}_p = \frac{1}{n} \sum_{i=1}^n \frac{(x_i + x'_i)}{2} \quad (5)$$

$$s_p = \sqrt{\frac{\sum_{i=1}^n (x_i - x'_i)^2}{2n}} \quad (6)$$

With the mean and standard deviation calculated, we can now calculate the RSD which provides an estimate for precision ( $RSD_p$ ). In essence, a low RSD percentage corresponds to a low variability and a higher percentage indicates a more variable dataset. Although a precise cut-off will depend on the nature and goal of the survey, in most cases, a RSD <20% is an indication of good precision, whereas elements with a high (>20%) RSD could indicate precision issues. The RSD is calculated via:

$$RSD_p = \frac{s_p}{\bar{x}_p} \times 100\% \quad (7)$$

In circumstances where one or both values for an elemental composition from a pair are below the lower detection limit or above the upper detection limit, the pair is preferably removed before calculations. This is because there is an established relationship between concentration and precision, where elements at concentrations near their detection limits will yield poor precision results (Thompson and Howarth, 1978). Another consideration is the number of pairs available for analysis. A larger number of pairs ( $\geq 30$  at the GSC) will be less prone to the effects of random errors and thus more likely to yield reliable precision estimates (McCurdy and Garrett, 2016). In cases where there is a significant deviation between pairs of analyses, affected samples may require re-analysis.

#### 4.2.4 Testing if the data is fit-for-purpose using ANOVA

In the context of a geochemical survey that is intended for single element mapping, it is important to understand the significance of regional geochemical variability relative to that at a local scale. One-way ANOVA can be used to quantify the significance of the regional geochemical variability (Garrett, 1983; McKillup and Dyar, 2012; Garrett, 2013b; McCurdy and Garrett, 2016). During a regional-scale survey, field duplicate samples (two or more per duplicated site) are collected within a few meters of each other to characterize the local variability at a sample site ('within' site). The variability at the survey scale is captured by samples that are taken between sites ('between' sites). Any statistical analysis program can be used to perform the ANOVA, which typically present a summary of results that includes the F-statistic ( $F$ ) and a p-value. The F-statistic, or variance ratio, is calculated as the ratio of 'between' site mean squares ( $(ms_i^b)^2$ ) and the 'within' sites mean square ( $(ms_i^w)^2$ ) for each element  $i$ :

$$F = \frac{(ms_i^b)^2}{(ms_i^w)^2} \quad (8)$$

Typically, the >95<sup>th</sup> percentile confidence level is chosen (which implies a p=0.05) with degrees of freedom  $n - 1$  and  $n$ , where  $n$  is the number of duplicate pairs. To calculate the percentage of variance 'between' sites (bpct), we can use the F-statistic:

$$bpct = 100 \times \left( \frac{F - 1}{F + 1} \right). \quad (9)$$

Finally, the percentage of variance 'within' sites (wpct) can be given as:

$$wpct = 100 - bpct \quad (10)$$

A log-transformation ( $\log_{10}$ ) of the data is often carried out prior to ANOVA to ensure the homogeneity of variance (homoscedasticity), a requirement of ANOVA (Bartlett, 1947). For each element, if one or both values from a field duplicate pair fall below the lower detection limit or above the upper detection limit, the pair is typically removed before calculations. Results derived from elements with few pairs remaining ( $\leq 30$  pairs at the GSC) are typically less reliable and could warrant further investigation (McCurdy and Garrett, 2016). Essentially, elements with a low F-statistic combined with a  $p \geq 0.05$  are deemed suspect and should be investigated further. Furthermore, such elements will typically have a greater percentage variation within sites as compared to between sites, indicating that this element could be problematic when used for mapping purposes. It should be noted that for purposes other than traditional single element regional mapping, ANOVA results may, or may not, be significant in the application of geochemical data.

### 4.3 Re-analysis of archived samples

Archive samples are sometimes selected for re-analysis based on the mineral potential of an area, combined with recent and significant advances in analytical instrumentation, which allows more elements to be determined, and overall, at a higher precision with lower detection limits compared to older analyses (e.g., Table 1). For example, surveys conducted at the GSC in the 1970s only determined 12 variables using a combination of 4 methods. Nowadays, with one instrument, up to 65 variables can be determined. Re-analysis of archived samples is also cost-effective, at approximately 5% of the cost of a new field survey (McCurdy et al., 2014).

**Table 1.** Summary of methods used and variables determined at the Geological Survey of Canada from the 1970s to 2020s. Over the decades, significant advances in instrumentation allowed for more elements to be determined. Elements are arranged alphabetically.

Year	Method	Variables determined
1976	AAS	Ag, Co, Cu, Fe, Ni, Mn, Mo, Pb, Zn
	COL	As
	GRAV	LOI
	NAA	U
1985	AAS	Ag, As, Cd, Co, Cu, Fe, Hg, Ni, Mn, Mo, Pb, Sb, V, Zn
	GRAV	LOI
	FNAA	Au
	ISE	F
	NAA	U
1990	AAS	Ag, Cd, Co, Cu, Fe, Hg, Ni, Mn, Pb, V, Zn
	GRAV	LOI
	ISE	F
	INAA	Na, Sc, Cr, Fe, Co, Ni, As, Br, Rb, Mo, Sb, Cs, Ba, La, Ce, Sm, Eu, Tb, Yb, Lu, Hf, Ta, W, Th, U, Au
2020	ICP-MS	Ag, Al, As, Au, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Hg, Ho, In, K, La, Li, Lu, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Pd, Pr, Pt, Rb, Re, S, Sb, Sc, Se, Sm, Sn, Sr, Ta, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Yb, Zn, Zr
	INAA	Ag, As, Au, B, Ba, Cd, Ce, Co, Cr, Cs, Eu, Fe, Hf, Ir, La, Lu, Mo, Na, Ni, Rb, Sb, Sc, Se, Sm, Sn, Ta, Tb, Te, Th, Ti, U, W, Yb, Zn, Zr

AAS – Atomic absorption spectroscopy  
COL – Colorimetry  
FNAA – Fire assay with neutron activation analysis  
GRAV – Gravimetry at approx. 500°C  
ICP-MS – Inductively coupled plasma–mass spectrometry  
INAA – Instrumental neutron activation analysis  
ISE – Ion selective electrode  
NAA – Neutron activation analysis by delayed neutron counting

Even though the re-analysis procedure of samples is quite similar to newly collected samples, there are a few differing factors. Notably, most of the steps required for survey planning and design (regional survey, stage 3) have already been completed. However, some components of survey planning and design can be altered if need be (e.g., sample labelling, site description based on new satellite imagery). Furthermore, based on the re-analysis data, a detailed survey (stage 4) may follow.

Archived samples should be checked prior to re-analysis to ascertain that there is sufficient material (Fig. 9). Samples should also be checked thoroughly to make sure that the containers are still closed tightly and that no material has contaminated the samples. If necessary, samples may be crushed, milled and sieved again.

A new QA/QC program is necessary for survey sample re-analysis. In particular, new blank, control reference materials (or CRMs) and analytical duplicate samples will need to be chosen/prepared, whereas field duplicate samples will be inherited from the original survey (if they exist) and re-analyzed among the remainder of the samples.

Occasionally, some survey samples become irreplaceable. For example, an older surveyed area is now a protected area or national park, meaning that no further sampling can take place. Another example is where sampling occurred before major infrastructure/economic development (e.g., a mine, highway). For these occasions, it is worthwhile to carefully consider the method of analysis - whether it is destructive or not. A destructive analytical method is when a sample (or a fraction of it) can no longer be used for subsequent analysis after element determination. A non-destructive analytical method should be chosen instead to ensure there will be enough material for future analyses. Instrumental neutron activation analysis (INAA) and 'portable' X-ray fluorescence analysis (detailed in the following section) are both non-destructive analytical methods.

## 5 Conducting geochemical analyses

There is no single analytical method to analyze lake sediments. The goal of a regional-scale survey is to obtain a reliable geochemical representation of the area. Therefore, there is a need to choose an analytical method that is cost-effective, has low detection limits, is accurate and precise, destroys no to very little material (i.e., acid digestion), and is able to determine a large number of elements/oxides. Since no single method can determine all of the elements in the periodic table, a combination of methods are usually chosen, based on the objective of the survey. Common analytical methods include X-ray fluorescence (XRF), atomic absorption spectroscopy (AAS), inductively coupled plasma-mass spectrometry (ICP-MS) or inductively coupled plasma-atomic emission spectrometry (ICP-AES), instrumental neutron activation analysis (INAA) and determination of volatile compounds using the Loss on Ignition (LOI), which are detailed below.

### 5.1 X-ray fluorescence (XRF)

The XRF analytical method is non-destructive, rapid, cost-effective and has been used reliably for more than six decades. This method can analyze up to 80 elements with concentrations ranging from 100 wt% to a few ppm (Rollinson, 1993; Fitton, 1997; Wirth, 2020). However there are drawbacks: 1) analysis requires large samples (>1 g), 2) materials analyzed need to be homogeneous, 3) elements lighter than Na cannot be detected, and 4) determined elements must be in high abundance ( $\geq 1$  ppm) (Wirth, 2020). Since 1994, the typically bulky XRF laboratory instrument became hand-held and called the 'portable XRF' or pXRF instrument (Cuffari, 2016). Recent improvements of the pXRF instrument include improved safety, faster analysis times and lower detection limits. Although the pXRF instrument does not yield as accurate and precise results compared to the larger, laboratory-sized XRF instrument, it can prove advantageous in the field setting to provide cost-effective and rapid data (Knight et al., 2021).



## 5.2 Atomic absorption spectroscopy (AAS)

The AAS method, developed in the 1950s, is technically simple, very cost-effective and has been used reliably for over five decades. This method was widely used in the past, but has since been progressively disused in favour of more modern methods (e.g., ICP-AES, ICP-MS). This method can determine over 40 elements, including all major elements with the exception of P which has a higher detection limit (1 ppm) (Rollinson, 1993; Rowland, 1997). An advantage of using the AAS method is that it can determine light elements such as Be and Li, which cannot be determined by XRF. However, AAS cannot compete with more rapid, multi-element determination methods such as XRF and ICP-MS/AES. Rather, the method determines one element at a time, although this limitation has recently been overcome by fitting instruments with multi-turret light sources. The sample also needs to be prepared into a solution (acid digestion), which unfortunately destroys the portion of sample. Nonetheless, AAS is frequently used as a complementary method to XRF and ICP-MS/AES.

## 5.3 Inductively coupled plasma-atomic emission spectrometry (ICP-AES)

Even though the ICP-AES method is newer (1970s), it is now well established in the field of geochemistry. This method can detect over 70 elements, including major, minor and trace elements, at very low detection limits ( $\sim 0.05$  ppm) (Walsh, 1997). An advantage of using the ICP-AES method is that it can simultaneously determine a large number of elements within a single sample. The analysis of a prepared sample is also relatively quick ( $\sim 1$  minute) (Rollinson, 1993). However, both Rb and Cs cannot be determined (poor analytical performance due to spectral ranges outside of instrument performance) and it might be difficult to detect some trace elements with very low concentrations ( $\leq 1$  ppm) (Walsh, 1997). Furthermore, a small portion of the sample must first be digested in order to allow for elemental detection, thus rendering this method destructive. Nonetheless, this method is one of the most widely used for routine element determination.

## 5.4 Inductively coupled plasma-mass spectrometry (ICP-MS)

The ICP-MS analytical method is rapid, accurate, precise and can simultaneously detect most elements in the periodic table, including some isotopes (Jarvis, 1997). This method is particularly useful in detecting elements that are present at very low concentrations. For example, the ICP-MS method can achieve detection limits between 0.01 - 0.001 ppm for REEs, which are an essential part of many recent geochemical studies. A small portion of the sample is first digested, rendering this method destructive. Nowadays, the ICP-MS is a preferred method for the detection of trace elements.

There are a number of digestion protocols available. Among them, the aqua regia ( $3\text{HCl} + \text{HNO}_3$ ) digestion is particularly effective at digesting base metals hosted in sulphide and some clay minerals. Specifically, the aqua regia digestion is a partial to near-total digestion (in the context of lake sediments) that is particularly apt at highlighting the signal from sulphide and oxide minerals without dissolving the more refractory minerals (e.g., REE minerals, cassiterite, wolframite, barite, chromite, ilmenite, rutile, sphene, monazite, zircon and garnet) (Gaudio et al., 2007; Crock and Lamothe, 2011). Some laboratories use a modified aqua regia digestion (1:1:1  $\text{HNO}_3:\text{HCl}:\text{H}_2\text{O}$ ) which performs just as well. The use of the aqua regia digestion is highly recommended since it has been used successfully since the 1970s at the GSC and elsewhere as well.

## 5.5 Instrumental neutron activation analysis (INAA)

The INAA method is relatively cost-effective and non-destructive, although it requires a large amount of material (over 0.2 g). Over 60 elements can be determined by this method, including REEs, PGEs and a number of high-field strength elements (Rollinson, 1993; Parry, 1997; Eby, 2017). In particular, Au has the lowest detection limit (1 ppb) compared to all other elements detected using INAA, making this method ideal for gold exploration (McConnell and Davenport, 1989; Parry, 1997). Drawbacks include a need for the sample to be irradiated, requiring certain precautions, and that some elements of geological interest (Nb, Y, Rb, Sr, Y and Zr)

are better determined by other analytical methods such as XRF and/or ICP-MS/AES. Overall, INAA has very few limitations and can determine elements in a wide variety of sample types (Eby, 2017).

## 5.6 Determination of volatile compounds using Loss on Ignition (LOI)

The method is used to determine the relative proportion of organic (e.g., water, carbon, organic matter and other volatile compounds) versus inorganic (elements) material present in the sample. For lake sediment samples, the LOI may reach up to 70%. However, a drawback is that this method does not differentiate between these compounds, but does give a rough measure of the organic content in the sediment (Heiri et al., 2001). Overall, it is preferable to heat lake sediment samples in steps (e.g., step 1: 105°C for nitrogen, 2: 371°C for oxygen, 3: 500-550°C for organic carbon, and 4: 1000°C for inorganic carbon) to differentiate between various volatile compounds (Santisteban et al., 2004; Geoscience Laboratories (Geo Labs), 2020).

## 5.7 Data considerations

In the last two decades, analytical instrumentation has greatly improved, resulting in greater accuracy, precision and lower detection limits, all at a lower cost (Cohen et al., 2010; Piercey, 2014). Technological advancements, the incorporation of computers and the creation of semi- to fully-automated analytical equipment have also significantly contributed in rapid but ‘still’ data acquisition (although with very low volume and velocity and therefore, not big data). For example, in the 1970s, a total of 12 elements were determined, whereas now 65 elements are routinely determined for lake sediments samples at the GSC (Friske, 1991; McCurdy et al., 2017) (Table 1). The design and implementation of a data governance and formalized management framework to ensure the effective and efficient use of data (see Henderson et al., 2017; U.S. Geological Survey, 2020; Seequent, 2021 for information) will be a key determinant in the adoption of large-scale survey data, and therefore to realize the full value of data. A robust data governance framework should also enable safeguarding of data for future applications or re-purposing (Henderson et al., 2017), because there is an explicit desire internationally but particularly also in Canada (of Canada, 2022) to integrate transdisciplinary methods in geosciences and adjacent fields to further extract previously undetected anomalies, create prospectivity models, refine mineral system models, construct regional-scale mineral assessments, etc. (e.g., Bergen et al., 2019; Lawley et al., 2021; Zhang et al., 2021a). As the integration for transdisciplinary methods progresses, it would be inevitable that sampling processes and associated data-specific practices would change (e.g., quality management programs), because specifications of data would be re-engineered to suit changing downstream purposes.

# 6 Data evaluation

The prime use of lake sediment geochemical survey data is to either aid and stimulate mineral exploration, support geological mapping of bedrock and surface materials, and/or conduct environmental surveys (Hornbrook and Friske, 1987; Friske and Hornbrook, 1991). Historical or legacy data can also provide a baseline for future work and to help track environmental changes. Historically, the release of lake sediment geochemical data (especially gold) has set off staking rushes (Friske and Hornbrook, 1991; Davenport et al., 1997). This section details the various steps required to use the geochemical data results, as well as some prominent techniques that can help with data interpretation.

## 6.1 Evaluation of the quality of the data

The first step when receiving geochemical results from an analytical laboratory is to ascertain that the number of samples sent for analysis matches with the number of analyzed samples. Sample identification codes should also be checked against a master list to make sure there are no errors, omissions or spelling mistakes. Additionally, the geochemical data should be visually inspected to make sure that each element is listed (as listed



in the laboratory brochure) and that values for each element are within reasonable ranges (within the bounds of the lower and upper detection limits). Assuming that the geochemical data from the analytical laboratory passes these checks, the data should then be copied and stored in a reliable and dedicated database and/or data storage devices (e.g., cloud storage, external hard drives, private company servers). At the GSC, geochemical data results are recorded in the: 1) Sedimentology Laboratory Database; and 2) the GSC's [Canadian Database of Geochemical Surveys](#) (a data swamp).

QA/QC reports are generated from the control samples that were inserted among routine samples. The basic steps on how to generate QA/QC results, along with formulas, are provided in subsection 4.2. Alternative QA/QC methods are also presented in [Reimann et al. \(2008\)](#); [McKillup and Dyar \(2012\)](#), and [Piercey \(2014\)](#). The analysis of QA/QC data can be carried out manually, although preferably using a statistical or programming language such as: Statistica, SAS University edition, GNU PSPP, Statistical lab, Perl Data Language, Python, among many others. At the GSC, a package called 'rgr', using the R programming language (<https://www.r-project.org/>), allows for semi-automated calculation of QA/QC results and preparation of relevant graphics ([Garrett, 2013b, 2018](#)).

If the data presents serious quality issues (labelling issues, contamination, inaccurate or imprecise data), the analytical laboratory should be contacted and the issue discussed, and if applicable, affected samples should be re-analyzed. If sample preparation is thought to be the issue, it should be discussed with staff at the preparation laboratory and, if applicable, affected samples should be re-prepared.

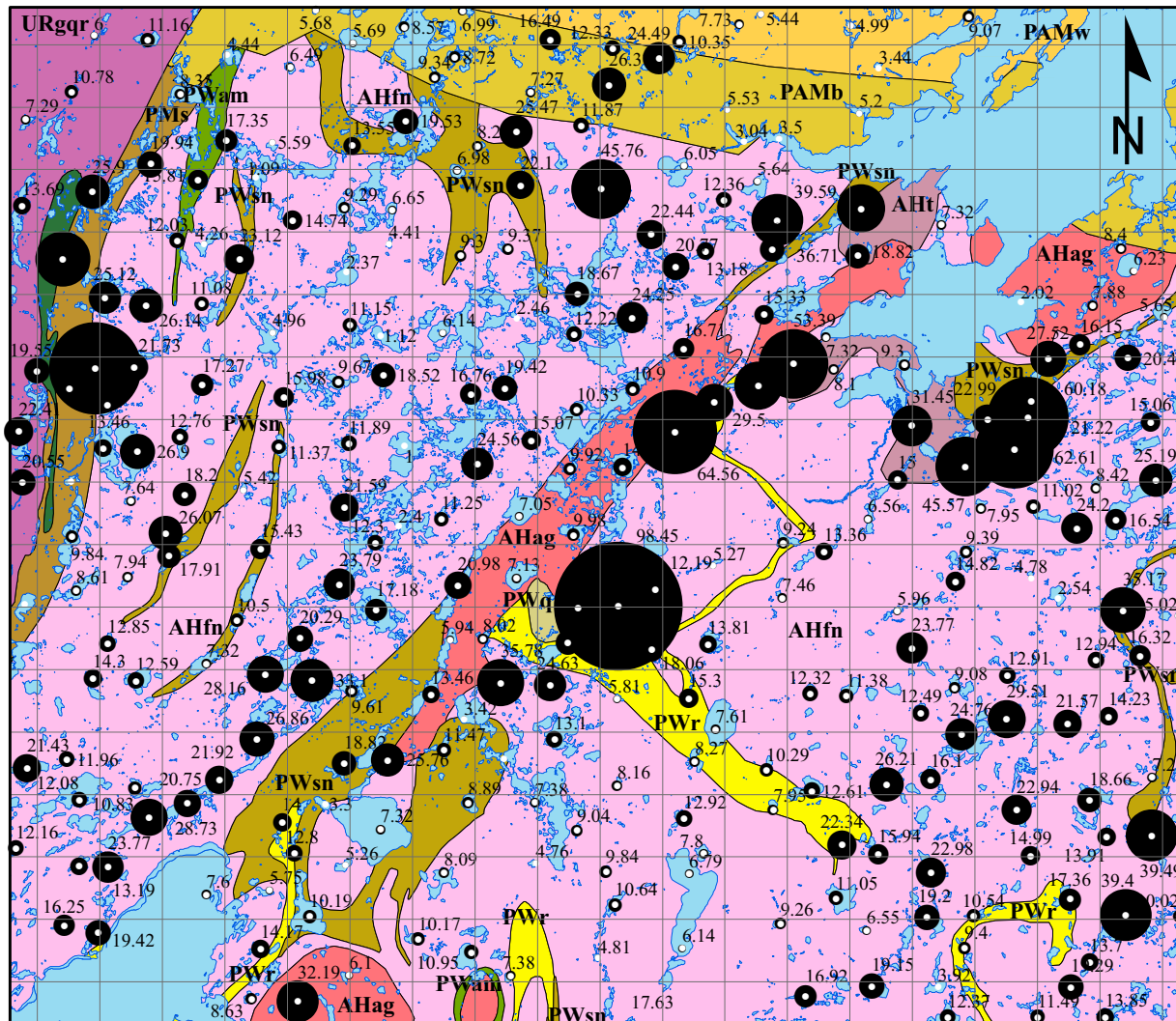
## 6.2 Data analysis

Basic element concentration maps can be useful for all 'resource' elements, such as gold, uranium, cobalt, nickel, copper, lithium, rare earth elements, zinc, arsenic, etc. ([Cameron, 1994](#); [Davenport et al., 1997](#); [McCurdy et al., 2017](#)) (Fig. 12). These maps are quick and easy to produce, providing near-instant guidance for mineral exploration or environmental studies (e.g., source of contamination). Other elements, such as Ni, Cu and K (among others) can also support bedrock geological mapping as element concentrations in lake sediment samples correspond well to the bedrock type from which they were derived ([Cameron, 1994](#); [Reimann and Garrett, 2005](#); [Shahrestani et al., 2018](#)).

Several factors need to be considered when analyzing lake sediment data, including (among others) the: 1) variations of element concentrations due to size of catchment basin and/or due to the bulk composition (organic vs inorganic) of the sample; 2) variable geology (bedrock and surficial) and their effect(s) on geochemical background values; 3) landscape/topography (a significant topography can result in stronger geochemical signals compared to flat landscapes); 4) effects of mass wasting events; 5) effects of scavenging of metals onto secondary Fe and Mn oxides or organic matter; and 6) influence of pH and Eh which controls metal solubility ([Hawkes, 1976](#); [Bonham-Carter and Goodfellow, 1986](#); [Cameron, 1994](#); [Barnett and Dyer, 2005](#); [Carranza, 2009a](#); [Arne et al., 2018](#); [Shahrestani et al., 2018](#)).

Analysis of lake sediment geochemical data typically begins with rudimentary methods of data 'mining' or 'discovery'. This involves a combination of statistical graphical displays, summary statistics (e.g., median, median absolute deviation, standard deviation, percentiles) and spatial data plotting (using Geographic Information System [GIS] programs). This preliminary examination of the geochemical data typically teases out obvious outliers (anomalies) that may have exploration interest, especially if coupled with other lines of evidence (favourable bedrock geology, geological structures, known mineral occurrences, etc.). These rudimentary techniques are essentially an effort to determine background and anomaly thresholds and are a component of spatial data analysis (SDA) and exploratory data analysis (EDA) (e.g., [Tukey, 1977](#); [Carranza, 2009b](#); [Chiprés et al., 2009](#)).

Estimating the background concentration of elements is a major challenge ([Reimann and Garrett, 2005](#); [Reimann et al., 2005, 2018](#)). Background values may need to be removed from measured elemental concentrations of lake sediment samples to enhance anomalies (mineral deposit or source of contamination). Numerous techniques have been proposed to filter background concentrations. Among them are those that use: fractal



**Legend**

- Lake samples
- Cu

**Saskatchewan geology 1M**

**Paleoproterozoic**

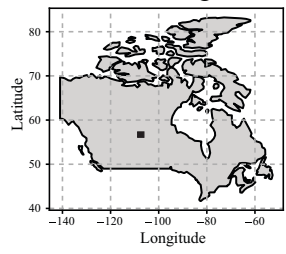
- PAMb Conglomeratic quartz arenite ± pebbly quartz arenite
- PAMw Clay intraclast-rich quartz arenite
- PMS Undifferentiated sedimentary and minor volcanic rocks
- PWam Amphibolite, mafic volcanic rocks, arkose, psammonpelite
- PWq Quartzite, pelite, calc-silicate rocks, minor pebble conglomerate
- PWr Arkose, calcereous arkose, psammite, psammonpelite
- PWsn Pelite to psammonpelite

**Neoproterozoic to Paleoproterozoic**

- URggr Retrogressed granitic to quartz dioritic gneiss

**Neoproterozoic**

- AHag Granite augen granite, leucogranite, derived gneiss
- AHfn Tonalite-granodiorite gneiss and migmatite
- AHt Granodiorite to leuconorite



**Figure 12.** Example of an element concentration map, using Cu concentrations north-central Saskatchewan (NTS 074-B and G). Study area is the same as presented in Fig. 1. The concentration of Cu at each sample site is visualized by the size of the black circle. Copper values (in ppm) accompany the black circles. The element concentration map includes the bedrock geology which can help with data analysis and subsequent interpretations. Bedrock geological map provided by the Saskatchewan Geological Survey.

analysis (Lima, 2018), Z-scores (Davenport et al., 1997), multiple regression analysis (Bonham-Carter and Goodfellow, 1986; Carranza and Hale, 1997; Shahrestani et al., 2018), catchment geology weighted methods (Bonham-Carter et al., 1986), among others.

Multivariate methodologies, detailed by Grunsky and de Caritat (2019), have been used successfully to identify geochemical anomalies as well as to aid bedrock mapping. Data is first conditioned. Elemental values that are incomplete, inaccurate and imprecise are removed from the dataset. Censored data (data below lower detection limits) is either replaced with estimates using 1/2 of the lower detection limit (Sanford et al., 1993; Grunsky et al., 2014) or by using the  $k$ -nearest neighbour algorithm (Hron et al., 2010). The data is then transformed using the centred log ratio (CLR transformation) to remove effects of closure (Aitchison, 1982). The transformed data is then analyzed using multivariate methods such as principal component analysis (PCA), independent component analysis (ICA), multi-dimensional scaling (MDS) or random forest (RF). Principal components chiefly reflect the stoichiometry of rock-forming minerals. Hence, maps of the dominant principal components demonstrate regional lithologies and can aid bedrock mapping (Grunsky and de Caritat, 2017; Arne et al., 2018). To identify geochemical anomalies, linear regressions of the element of interest against the dominant principal components will yield elemental residual values which can then be mapped (using simple kriging) to reveal areas of interest. Figure 13 presents the results of a study in eastern Labrador using the multivariate PCA methodology.

The latest methodology developed to enhance geochemical anomalies uses machine learning algorithms. Zhang et al. (2021b,a) developed a new machine learning methodology that predicts trace element concentrations using major and minor element data. The predicted elemental concentrations, can be compared to actual measured concentrations and the residuals (actual minus predicted or predicted minus actual) can more clearly identify anomalies. Regression residuals from actual minus predicted correspond to geochemical anomalies, which can be mapped using simple kriging. Interestingly, data conditioning, replacement of censored values and data transformation (e.g., CLR) is not necessary using this methodology. Figure 14 presents the results of a study in eastern Labrador using the machine learning methodology. Results obtained using the machine learning methodology are both more accurate and selective compared to the well-established multivariate methodology, as detailed at length in Zhang et al. (2021a) (also compare Figs. 13 and 14).

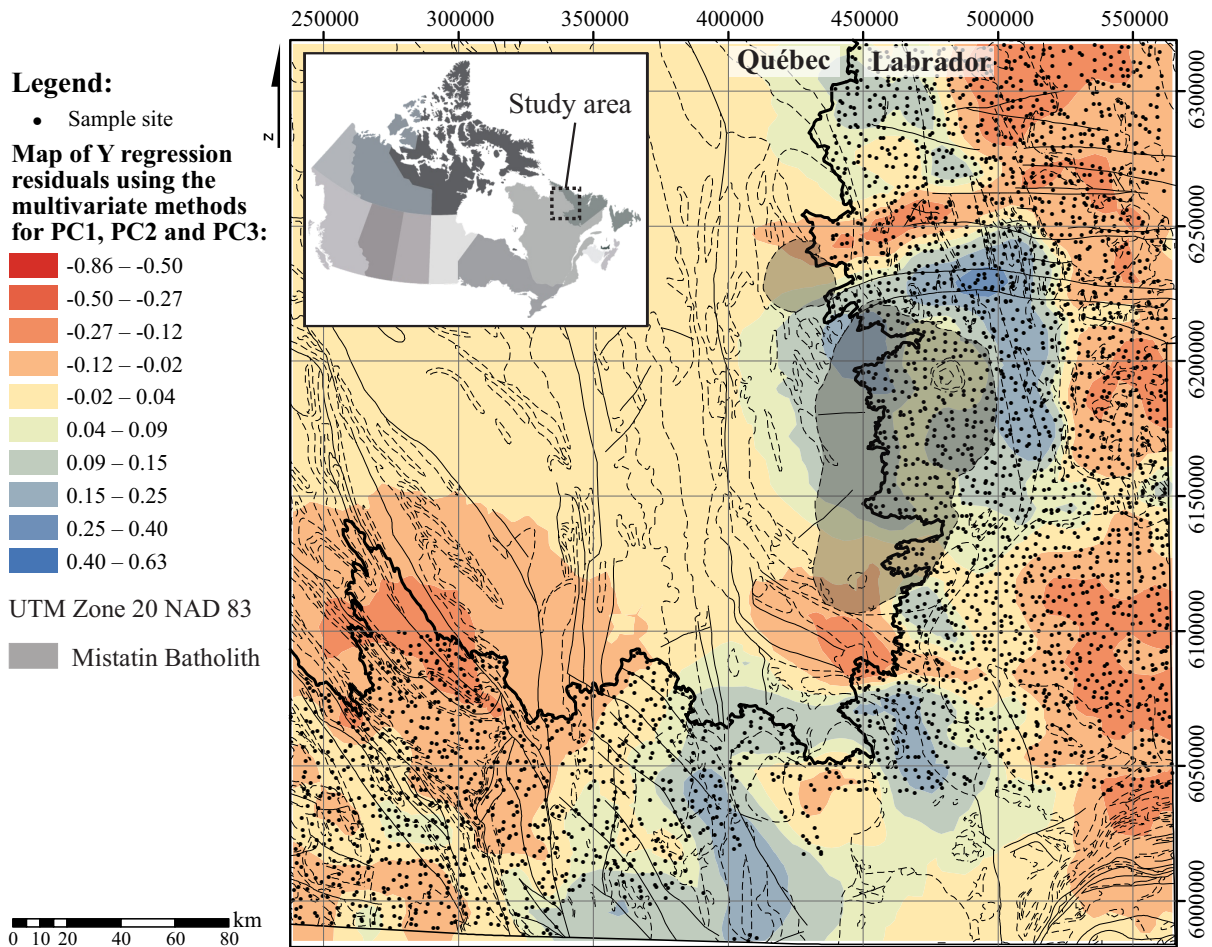
### 6.3 Publishing and data archiving

Publishing analytical data and data interpretations is strongly encouraged for many reasons, as it: 1) provides a permanent copy of the data; 2) ensures high-scientific standards (reproducibility); 3) increases accessibility to all sectors; 4) increases the dissemination of new idea(s); and 5) promotes data re-use (McKee, 2010; Henderson et al., 2017). Archiving data (cloud services, DVDs, external hard drives, private servers) is another possibility to ensure the preservation of the data, however, its access will be restricted to the data users. At the GSC, the data collected and generated by each survey (typically 1:250,000 scale NTS sheet) is released as an open file (GEOSCAN). Each open file generally contains: 1) background information, 2) sample collection and analytical procedures, 3) a map showing sample locations, 4) QA/QC results, and 5) all accompanying data and metadata (nowadays in appendices). Older open file reports (pre-2000s) also include symbol-trend maps accompanied by bedrock geological maps. These maps provide a rapid means of evaluating the surveyed area for any geochemical anomalies for the purpose of mineral exploration (Friske and Hornbrook, 1991).

Publication of the data and data interpretations should always include a copy of the original data (and certificates, if applicable) as reported by the analytical laboratory (however, checked for labelling issues/errors). If some samples were re-analyzed because of quality issues, both the original and re-analyzed data should be included, accompanied by a clear explanation and identification of the errors. The method used to interpret the data should also be described to ensure reproducibility, and dissemination of new idea(s) if applicable.

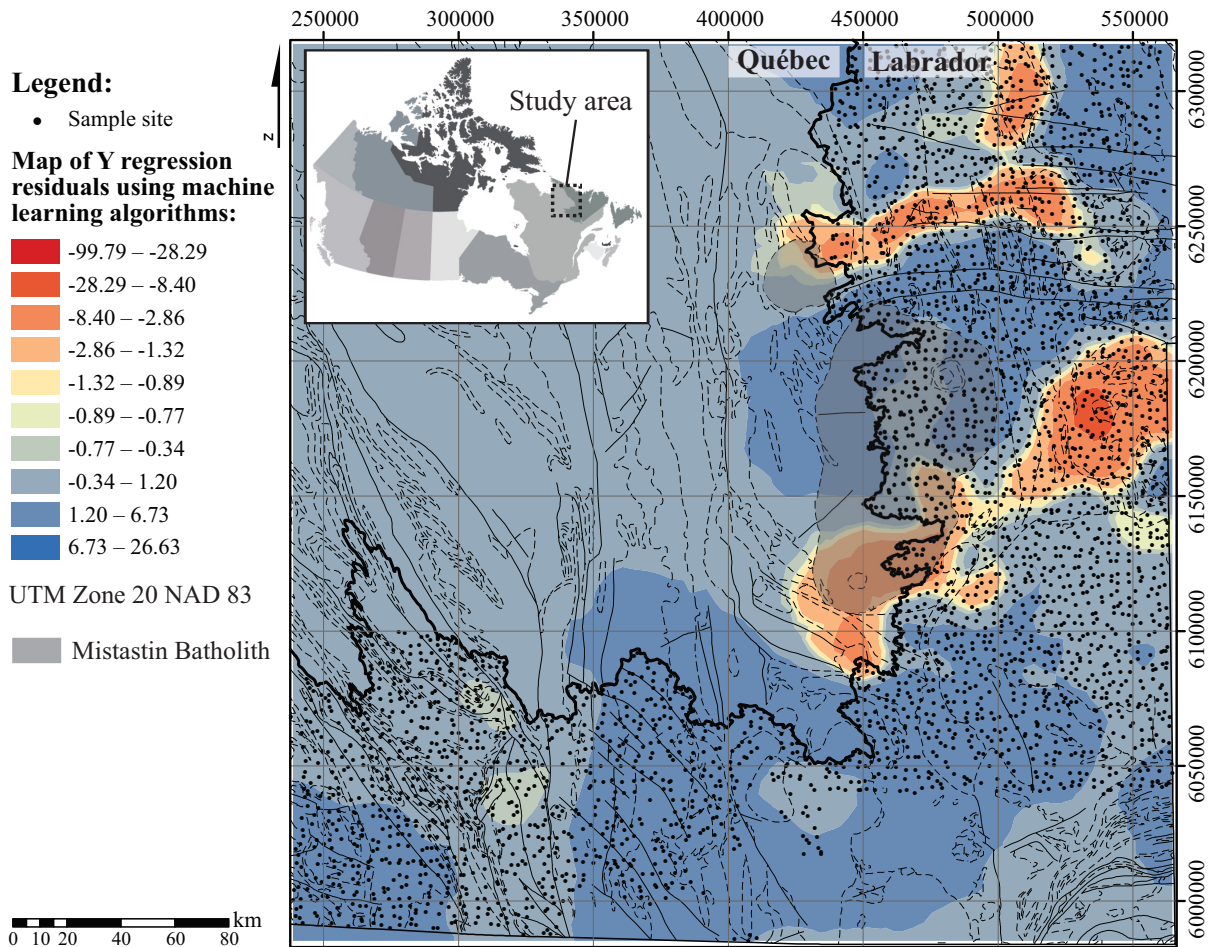
### 6.4 Metadata

It is important that any publication includes essential metadata. Metadata, defined as ‘data about data’, is critical to give context, information and aspects to the data. It is often used to summarize basic information



**Figure 13.** Regional prospectivity map of Y using the multivariate PCA geochemical anomaly methodology detailed in [Grunsky and de Caritat \(2019\)](#). Lake sediment samples (black circles) are located in eastern Labrador (NTS 13-L, 13-M, 14-D, 23-I and 23-J). The Mistatin Batholith (shaded grey), characterized by intermediate potassic intrusive rocks ([van der Leeden, 1995](#)), is locally highly enriched in REEs, Zr, Y,  $\pm$  Be, Nb, U and Th ([Hammouche et al., 2012](#)). A well-known ENE-trending glacial dispersal train ([Batterson, 1989](#) and references therein) emanating from the northern intrusion of the batholith is visible.





**Figure 14.** Regional prospectivity map of Y using the machine learning-based geochemical anomaly methodology developed by Zhang *et al.* (2021b,a). Lake sediment samples (black circles) are located in eastern Labrador (NTS 13-L, 13-M, 14-D, 23-I and 23-J). The Mistastin Batholith (shaded gray) is chiefly composed of intermediate potassic intrusive rocks (van der Leeden, 1995) and is locally highly enriched in REEs, Zr, Y,  $\pm$  Be, Nb, U and Th (Hammouche *et al.*, 2012). Two valid ENE-trending glacial dispersal trains emanating from the southern and northern portion of the batholith are visible (Zhang *et al.*, 2021a).

about the data which can make tracking and working with the data easier. Given the complex nature of a regional-scale lake sediment survey, metadata can be recorded during numerous steps of the survey. At the GSC, metadata is mainly collected during sample collection, sample preparation and geochemical analysis. Table 2 presents a minimum list of metadata criteria. Other criteria are also possible based on the needs/objectives of the geochemical survey.

**Table 2.** *Essential metadata to be recorded during sample collection, sample preparation and geochemical analysis in the context of a regional-scale lake sediment survey.*

<b>Sample collection</b>	<b>Sample preparation</b>	<b>Geochemical analysis</b>
Project name	Laboratory name	Laboratory name
Funding source	Laboratory package code	Laboratory package code
Province/Territory	Preparation staff/contractor name	Laboratory detection limits
NTS sheet number	Preparation methodology	Laboratory brochure
Datum for coordinates	Sieving: mesh size	Date of sample submission
Sampler name	Milling: type of mill	Date of data receipt
Sampling design	Number of samples	Preparation methodology
Sampling method	Certificate numbers	Assayer/contractor name
Sample density	Date of sample preparation	Laboratory QA/QC procedures
Sample collection dates		Certificate numbers
Number of samples		Number of elements determined
Sampling equipment		Upper and lower detection limits
Sample medium		

## 7 Conclusions

Regional-scale lake sediment surveys have been successfully employed to characterize geochemical background, detect geochemical anomalies and provide environmental baselines since the 1970s. The continuous use of this type of survey at the GSC, at provincial/territorial agencies, industry and academia have further developed, tested and refined this methodology. This protocol manual presents the cumulative knowledge and up-to-date developments on the subject of regional-scale lake sediment surveys. Major concepts, including survey planning and design, how to collect, prepare and analyze lake sediment samples, as well as how to evaluate the resulting data were all discussed in this report. By adopting this set of guidelines, scientific researchers, exploration geologists, geochemists and citizen scientists will be able to directly compare lake sediment datasets from anywhere in Canada with the assurance of adequate QA/QC results for all data collected.

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## References

- Aitchison, J., 1982. The statistical analysis of compositional data; *Journal of the Royal Statistical Society: Series B*, v. 44, no. 2, p. 139–160. doi:10.1111/j.2517-6161.1982.tb01195.x.
- Arne, D.C., Mackie, R., Pennimpede, C. and Grunsky, E., 2018. Intergrated assessment of regional stream-sediment geochemistry for metallic deposits in Northwestern British Columbia (parts of NTS 093, 094, 103, 104); *in* *Geosciences BC Summary of Activities 2017; 2018-1, Minerals and Mining*, Geosciences BC, p. 23–30.
- Babeesh, C., Lone, A. and Achyuthan, H., 2017. Geochemistry of Manasbal lake sediments, Kashmir: Weathering, provenance and tectonic setting; *Journal of the Geological Society of India*, v. 89, p. 563–572. doi:10.1007/s12594-017-0645-4.
- Bajc, A.F. and Hall, G.E.M., 2000. Geochemical response to surficial media, North and East Ranges, Sudbury Basin; Ontario Geological Survey, Open File 6033, 286 p.
- Barnett, P.J. and Dyer, R.D., 2005. PGE surficial geochemistry case studies project: Lake Nipigon region geoscience initiative; Ontario Geological Survey, Open File 6167, 144 p.
- Bartlett, M.S., 1947. The use of transformations; *Biometrics*, v. 3, no. 1, p. 39–52. doi:10.2307/3001536.
- Batterson, M.J., 1989. Glacial dispersal from the Strange Lake alkalic complex, northern Labrador; *in* *Drift prospecting*, (eds.) R.N.W. DiLabio and W.B. Coker; Geological Survey of Canada 89-20, p. 31–40. doi:10.4095/127362.
- Bergen, K.A., Johnson, P.A., de Hoop, M.V. and Beroza, G.C., 2019. Machine learning for data-driven discoverin in solid Earth geoscience; *Science*, v. 363, no. 6433, p. eaau0323. doi:10.1126/science.aau0323.
- Bernal, E., 2013. Limit of detection and limit of quantification determination in gas chromatography; *in* *Advances in gas chromatography*, (ed.) X. Guo; IntechOpen. doi:10.5772/57341.
- Bonham-Carter, G.F. and Goodfellow, W.D., 1986. Background corrections to stream geochemical data using digitized drainage and geological maps: application to Selwyn Basin, Yukon and Northwest Territories; *Journal of Geochemical Exploration*, v. 25, no. 1-2, p. 139–155. doi:10.1016/0375-6742(86)90011-7.
- Bonham-Carter, G.F., Rogers, P.J. and Ellwood, D.J., 1986. Catchment basin analysis applied to surficial geochemical data, Cobequid Highlands, Nova Scotia; *Journal of Geochemical Exploration*, v. 29, no. 1-3, p. 259–278. doi:10.1016/0375-6742(87)90081-1.
- Cameron, E.M., 1980. Geochemical exploration for uranium in northern lakes; *Journal of Geochemical Exploration*, v. 13, no. 2-3, p. 221–250. doi:10.1016/0375-6742(80)90008-4.
- Cameron, E.M., 1994. Lake sediment sampling in mineral exploration; *in* *Drainage Geochemistry in Mineral Exploration*, v. 6, (eds.) M. Hale and J.A. Plant; Elsevier Science, p. 227–267. doi:10.1016/B978-0-444-81854-6.50013-4.
- Carranza, E.J.M., 2009a. Catchment analysis of stream sediment anomalies; *in* *Geochemical anomaly and mineral prospectivity mapping in GIS*, (ed.) E.J.M. Carranza; Elsevier Science, p. 115–144.
- Carranza, E.J.M., 2009b. Exploratory analysis of geochemical anomalies; *in* *Geochemical anomaly and mineral prospectivity mapping in GIS*, (ed.) E.J.M. Carranza; Elsevier Science, p. 51–84.

- Carranza, E.J.M. and Hale, M., 1997. A catchment basin approach to the analysis of reconnaissance geochemical-geological data from Albay Province, Philippines; *Journal of Geochemical Exploration*, v. 60, no. 2, p. 157–171. doi:10.1016/S0375-6742(97)00032-0.
- Cavanagh, N., Nordin, R.N., Swain, L.G. and Pommen, L.W., 1994. Lake and stream bottom sediment sampling manual; BC Ministry of Environment, Lands and Parks, Open file, 28 p.
- Chiprés, J.A., Castro-Larragoitia, J. and Monroy, M.G., 2009. Exploratory and spatial data analysis (EDA-SDA) for determining regional background levels and anomalies of potentially toxic elements in soils from Catorce-Matehuala, Mexico; *Applied Geochemistry*, v. 24, no. 8, p. 1579–1589. doi:10.1016/j.apgeochem.2009.04.022.
- Chow, T.J., Bruland, K.W., Bertine, K.K., Soutar, A., Kodie, M. and Goldberg, E.D., 1973. Lead pollution: records in southern California coastal sediments; *Science*, v. 181, no. 4099, p. 551–552. doi:10.1126/science.181.4099.551.
- Cohen, D.R., Kelley, D.L., Anand, R. and Coker, W.B., 2010. Major advances in exploration geochemistry, 1998–2007; *Geochemistry: Exploration, Environment and Analysis*, v. 10, p. 3–16. doi:10.1144/1467-7873/09-215.
- Coker, W.B., Hornbrook, E.W.H. and Cameron, E.M., 1979. Lake sediment geochemistry applied to mineral exploration; *in* *Geophysics and Geochemistry in the search for metallic ores*, (ed.) P.J. Hood; Geological Survey of Canada, Economic Geology Report 31, p. 435–476. doi:10.4095/106037.
- Coker, W.B. and Nichol, I., 1974. The relation of lake sediment geochemistry to mineralization in the northwestern Ontario region of the Canadian Shield; *Economic Geology*, v. 70, no. 1, p. 202–218. doi:10.2113/gsecongeo.70.1.202.
- Columbia University, 2022. Lamont-Doherty core repository: types of samples; Columbia University. <https://corerepository.ldeo.columbia.edu/content/types-samples>. [accessed February 14, 2022].
- Crock, J.G. and Lamothe, P.J., 2011. Inorganic chemical analysis of environmental materials - a lecture series; U.S. Geological Survey, Open File 2011-1193, 177 p. doi:10.3133/ofr20111193.
- Cuffari, B., 2016. Evolution of handheld XRF analysis in mining; AZO Mining. <https://www.azominer.com/Article.aspx?ArticleID=1344>. [accessed November 20, 2021].
- Davenport, P.H., Friske, P.W.B. and Beaumier, M., 1997. The application of lake sediment geochemistry to mineral exploration: recent advances and examples from Canada; *in* *Proceedings of Exploration 97: Fourth Decennial International Conference on Mineral Exploration*, (ed.) A.G. Gubins; 33, p. 261–270.
- Demetrides, A., Smith, D.B. and Wang, X., 2018. General concepts of geochemical mapping at global, regional, and local scales for mineral exploration and environmental purposes; *Geochimica Brasiliensis*, v. 32, no. 2, p. 136–179. doi:10.21715/GB2358-2812.2018322136.
- Dominy, S.C., 2014. Predicting the unpredictable - evaluating high-nugget effect gold deposits; *in* *Monograph 30 - Mineral Resource and Ore Reserve Estimation - The AusIMM guide to good practice*; AusIMM, second edition, p. 659–678.
- Douglas, M.S.V., 2013. Paleolimnology; *in* *Encyclopedia of Quaternary Science*, (eds.) S.A. Elias and C.J. Mock; Elsevier, second edition, p. 259–270. doi:10.1016/B978-0-444-53643-3.00232-6.
- Dyer, R. and Hamilton, S., 2007. The Ontario Geological Survey lake sediment geochemical program: progress towards a geochemical map of Ontario, Canada; *in* *EXPLORE Newsletter*; 135, Association of Applied Geochemists, p. 2–11.



- Dyer, R.D. and Barnett, P.J., 2007. Multimedia exploration strategies for PGEs: insights from the Surficial Geochemistry Case Studies Project, Lake Nipigon Region Geoscience Initiative, northwestern Ontario; Canadian Journal of Earth Sciences, v. 44, no. 8, p. 136–179. doi:10.1139/e07-031.
- Eby, N., 2017. Instrumental neutrol activation analysis INAA; Science education resource centre at Carleton College. [https://serc.carleton.edu/research\\_education/geochemsheets/techniques/INAA.html](https://serc.carleton.edu/research_education/geochemsheets/techniques/INAA.html). [accessed November 29, 2021].
- Fitton, G., 1997. X-ray fluorecence spectrometry; *in* Modern analytical geochemistry, (ed.) R. Gill; Routledge, p. 87–115.
- Fletcher, W.K., 1981. Analytical methods in geochemical prospecting: Sample preparation; *in* Handbook of Exploration Geochemistry, v. 1, (ed.) G.J.S. Govett; Elsevier Scientific Publishing, p. 255. doi:10.1016/B978-0-444-41930-9.50008-8.
- Friske, P.W.B., 1991. The application of lake sediment geochemistry in mineral exploration; *in* Exploration Geochemistry Workshop, (eds.) J.M. Franklin, J.M. Duke, W.W. Shilts, W.B. Coker, P.W.B. Friske, Y.T. Maurice, S.B. Ballantyne, C.E. Dunn, G.E.M. Hall and R.G. Garrett; Geological Survey of Canada, Open File 2390, p. 157–180. doi:10.4095/132392.
- Friske, P.W.B. and Hornbrook, E.H.W., 1991. Canada’s national geochemical reconnaissance programme; *in* Transactions of the Institution of Mining and Metallurgy, Section B. Applied Earth Science, v. 100; p. B47–B56.
- Garrett, R.G., 1974. Field data aquisition methods for applied geochemical surveys at the Geological Survey of Canada; Geological Survey of Canada, Open File 74-52, 42 p. doi:10.4095/102559.
- Garrett, R.G., 1983. Sampling methodology; *in* Statistics and data analysis in geochemical prospecting, v. 2, (ed.) R.J. Howarth; Elsevier Science, p. 83–100. doi:10.1016/B978-0-444-42038-1.50010-1.
- Garrett, R.G., 2013a. Assessment of local spatial and analytical variability in regional geochemical surveys with a simple sampling scheme; Geochemistry: Exploration, Environment, Analysis, v. 13, p. 349–354. doi:10.1144/geochem2011-085.
- Garrett, R.G., 2013b. The ‘rgr’ package for the R open source statistical computing and graphics environment - a tool to support geochemical data interpretation; Geochemistry: Exploration, Environment, Analysis, v. 13, p. 355–378. doi:10.1144/geochem2011-106.
- Garrett, R.G., 2018. Rgr package: Applied geochemistry eda; R-Project. <https://CRAN.R-project.org/package=rgr>. [accessed June 20, 2022].
- Garrett, R.G., 2019. Why minus 80 mesh; *in* EXPLORE Newsletter; 185, Association of Applied Geochemists, p. 1–10.
- Garrett, R.G., Reimann, C., Smith, D.B. and Xie, X., 2008. From geochemical prospecting to international geochemical mapping: a historical overview; Geochemistry: Exoloration, Environment, Analysis, v. 8, p. 205–217. doi:10.1144/1467-7873/08-174.
- Gaudino, S., Galas, C., Belli, M., Barbizzi, S., de Zorzi, P., Jaćimović, R., Jeran, Z., Pati, A. and Sansone, U., 2007. The role of differing soil sample digestion methods on trace elements analysis: a comparison of ICP-MS and INAA measurement results; Accreditation and quality assurance, v. 12, p. 84–93. doi:10.1007/s00769-006-0238-1.
- Geboy, N.J. and Engle, M.A., 2011. Quality assurance and quality control for geochemical data: a primer for the research scientist; U.S. Geological Survey, Open File 2011-1187, 33 p.

- Geoscience Laboratories (Geo Labs), 2020. 2020 schedule of fees and services; Geological Survey of Ontario, Ontario Ministry of Energy, Northern Development and Mines, Open File 12, 36 p.
- Goldberg, E.D., Gamble, E., Griffin, J.J. and Koide, M., 1977. Pollution history of Narragansett Bay as recorded in its sediments; *Estuarine and Coastal Marine Science*, v. 5, no. 4, p. 549–556. doi:10.1016/0302-3524(77)90101-3.
- Goldberg, E.D., Griffin, J.J., Hodge, V., Koide, M. and Windom, H., 1979. Pollution history of the Savannah River Estuary; *Environmental Science and Technology*, v. 13, no. 5, p. 588–594. doi:10.1021/es60153a012.
- Goldberg, E.D., Hodge, V., Koide, M. and Griffin, J.J., 1976. Metal pollution in Tokyo as recorded in sediments of the Palace Moat; *Geochemical Journal*, v. 10, no. 4, p. 165–174. doi:https://doi.org/10.2343/geochemj.10.165.
- Goldberg, E.D., Hodge, V., Koide, M., Griffin, J.J., Gamble, E., Bricker, O.P., Matisoff, G., Holdren, G.R. and Braun, R., 1978. A pollution history of Chesapeake Bay; *Geochimica et Cosmochimica Acta*, v. 42, no. 9, p. 1413–1425. doi:https://doi.org/10.1016/0016-7037(78)90047-9.
- Gomasca, M.A., 2009. *Basics of Geomatics*; Springer, 698 p.
- Govett, G.J.S., 1983. Geochemistry in the exploration sequence; *in Handbook of Exploration Geochemistry*, v. 3, (ed.) G.J.S. Govett; Elsevier, p. 7–15. doi:10.1016/B978-0-444-42021-3.50006-9.
- Grunsky, E.C. and de Caritat, P., 2017. Advances in the use of geochemical data for mineral exploration; *in Proceedings of Exploration 17: Sixth Decennial International Conference on Mineral Exploration*, (eds.) V. Tschirhart and M.D. Thomas; Decennial Minerals Exploration Conferences, p. 441–456.
- Grunsky, E.C. and de Caritat, P., 2019. State-of-the-art analysis of geochemical data for mineral exploration; *Geochemistry: Exploration, Environment, Analysis*, v. 20, p. 217–232. doi:10.1144/geochem2019-031.
- Grunsky, E.C., Mueller, U.A. and Corrigan, D., 2014. A study of the lake sediment geochemistry of the Melville Peninsula using multivariate methods: applications for predictive geological mapping; *Journal of Geochemical Exploration*, v. 141, p. 15–41. doi:10.1016/j.gexplo.2013.07.013.
- Hammouche, H., Legoux, C., Goutier, J. and Dion, C., 2012. Géologie de la région du lac Zeni; Ministère des Ressources Naturelles, Québec, Open File RG 2012-02, 35 p.
- Hawkes, H.E., 1976. The downstream dilution of stream sediment anomalies; *Journal of Geochemical Exploration*, v. 6, p. 345–358. doi:10.1016/0375-6742(76)90023-6.
- Hechler, J.H., 2013. QA/QC: summary of 2012-2013 quality-control data at the Geoscience Laboratory; *in Summary of field work and other activities 2013*; Ontario Geological Survey, Open File Report 6290, p. 1–7.
- Heiri, O., Lotter, A.F. and Lemcke, G., 2001. Loss on ignition as a method for estimating organic and carbonate content in sediments: reproducibility and comparability of results; *Journal of Paleolimnology*, v. 25, p. 101–110. doi:10.1023/A:1008119611481.
- Henderson, D., Earley, S. and Sebastian-Coleman, L., 2017. DAMA-DMBOK: Data management body of knowledge; DAMA International, Technics Publications, 628 p. second edition.
- Hornbrook, E.H.W., 1987. Lake sediment geochemistry: Canadian application in the eighties; *in Exploration '87 Proceedings*; Geochemical methods: their application to ore exploration; p. 405–416.
- Hornbrook, E.H.W. and Davenport, P.H., 1974. Exploration geochemistry - Newfoundland; *in Report of activities part B, November 1973 to March 1974*, (ed.) R.G. Blackadar; 74-1B, Geological Survey of Canada, p. 60. doi:10.4095/104676.

- Hornbrook, E.H.W. and Friske, P.W.B., 1987. Regional lake sediment and water geochemical data, Saskatchewan 1986, GSC OF-1359, NGR 95-1986, NTS 74A(NW), 74B(N1/2), 74G(s1/2), 74H(SW); Geological Survey of Canada, Open File 1359, 130 p. doi:10.4095/130264.
- Hornbrook, E.H.W. and Garrett, R.G., 1976. Regional geochemical lake sediment survey east-central Saskatchewan; Geological Survey of Canada, Open File 75-41, 20 p. doi:10.4095/102612.
- Hron, K., Templ, M. and Filzmoser, P., 2010. State-of-the-art analysis of geochemical data for mineral exploration; Computational Statistics and Data Analysis, v. 54, p. 3095–3107. doi:10.1016/j.csda.2009.11.023.
- Jarvis, K.E., 1997. Inductively coupled plasma-mass spectrometry (ICP-MS); in Modern analytical geochemistry, (ed.) R. Gill; Routledge, p. 171–187.
- Johnson, M.G., Culp, J.R. and George, S.E., 1986. Temporal and spatial trends in metal loadings to sediments of the Turkey Lakes, Ontario; Canadian Journal of Fisheries and Aquatic Sciences, v. 43, p. 754–762. doi:10.1139/f86-093.
- Jonasson, I.R., 1976. Detailed hydrogeochemistry of two small lakes in the Grenville geological province; Geological Survey of Canada, Open File 76-13, 37 p. doi:10.4095/102621.
- Karrow, P.F. and Geddes, R.S., 1987. Drift carbonate on the Canadian Shield; Canadian Journal of Earth Sciences, v. 24, p. 365–369. doi:10.1139/e87-036.
- Katz, A. and Kaplan, I.R., 1981. Heavy metal behavior in coastal sediments of southern California: a critical review and synthesis; Marine Chemistry, v. 10, no. 4, p. 216–299. doi:10.1016/0304-4203(81)90010-4.
- Kerr, A. and Davenport, P.H., 1990. Application of geochemical mapping techniques to a complex Precambrian shield area in Labrador; Journal of Geochemical Exploration, v. 39, p. 225–247. doi:10.1016/0375-6742(90)90075-L.
- Knight, R.D., Kjarsgaard, B.A. and Russell, H.A., 2021. An analytical protocol for determining the elemental chemistry of quaternary sediments using a portable x-ray fluorescence spectrometer; Applied Geochemistry, v. 131, p. 105026. doi:10.1016/j.apgeochem.2021.105026.
- Knott, J.M., Glysson, G.D., Malo, B.A. and Schroder, L.J., 1993. Quality assurance plan for the collection and processing of sediment data by the U.S. Geological Survey, water resources division; U.S. Geological Survey, Open File 92-499, 22 p. doi:10.3133/ofr92499.
- Larson, G. and Schaetzl, R., 2001. Origin and evolution of the Great Lakes; Journal of Great Lakes Research, v. 27, no. 3, p. 518–546. doi:10.1016/S0380-1330(01)70665-X.
- Last, W.M. and Smol, J.P., 2001a. Tracking environmental change using lake sediments, volume 1: basin analysis, coring, and chronological techniques; Springer, 548 p.
- Last, W.M. and Smol, J.P., 2001b. Tracking environmental change using lake sediments, volume 2: physical and geochemical methods; Springer, 504 p.
- Lawley, C.J.M., Tschirhart, V., Smith, J.W., Pehrsson, S.J., Schetselaar, E.M., Schaeffer, A.J., Houlé, M.G. and Eglington, B.M., 2021. Prospectivity modeling of Canadian magmatic Ni ( $\pm$  Cu  $\pm$  Co  $\pm$  PGE) sulphide mineral systems; Ore Geology Reviews, v. 132, p. 103985. doi:10.1016/j.oregeorev.2021.103985.
- Leahy, E.J., 1971. Geology of the Night Hawk Lake area, district of Cochrane; Ontario Geological Survey, Open File 96, 89 p.

- Lima, A., 2018. Evaluation of geochemical background at regional and local scales by fractal filtering technique: case studies in selected Italian areas; *in* Environmental Geochemistry, (eds.) B. De Vivo, H.E. Belkin and A. Lima; Elsevier, second edition, p. 115–132. doi:10.1016/B978-0-444-63763-5.00016-1.
- Lynch, J., 1990. Provisional elemental values for eight new geochemical lake sediment and stream sediment reference materials LKSD-1, LKSD-2, LKSD-3, LKSD-4, STSD-1, STSD-2, STSD-3 and STSD-4; *Geostandards Newsletter*, v. 14, no. 1, p. 153–157.
- Lynch, J., 1999. Additional provisional elemental values for LKSD-1, LKSD-2, LKSD-3, LKSD-4, STSD-1, STSD-2, STSD-3 and STSD-4; *Geostandards Newsletter*, v. 23, no. 2, p. 251–260.
- McClenaghan, M.B., Spirito, W.A., Plouffe, A., McMartin, I., Campbell, J.E., Paulen, R.C., Garrett, R.G., Hall, G.E.M., Pelchat, P. and Gautier, M.S., 2020. Geological Survey of Canada till-sampling and analytical protocols: from field to archive, 2020 update; Geological Survey of Canada, Open File 8591, 82 p. doi:10.4095/326162.
- McConnell, J. and Davenport, P., 1989. Gold and associated trace elements in Newfoundland lake sediment: their application to gold exploration; *Journal of Geochemical Exploration*, v. 32, no. 1-3, p. 33–50. doi:10.1016/0375-6742(89)90042-3.
- McCurdy, M.W., Böhm, C.O., Anderson, S.D., Gauthier, M.S. and Amor, S.D., 2017. Geochemical data for lake sediments in the Superior Province of Manitoba (NTS 53-E, 53-L and 53-M; Geological Survey of Canada, Open File 8179, 27 p. doi:10.4095/299680.
- McCurdy, M.W., Campbell, J.E., Böhm, C.O., Trommenlen, M.S. and Syme, E.C., 2013. Regional lake sediment geochemical data, Kasmere Lake - Nueltin Lake area, Manitoba, NTS 64-K, 64N, 64-O; Geological Survey of Canada, Open File 7309, 17 p. doi:10.4095/292267.
- McCurdy, M.W. and Garrett, R.G., 2016. Geochemical data quality control for soil, till and lake and stream sediment samples; Geological Survey of Canada, Open File 7944, 40 p. doi:10.4095/297562.
- McCurdy, M.W., Spirito, W.A., Grunsky, E.C., Day, S.J.A., McNeil, R.J. and Coker, W.B., 2014. The evolution of the geological survey of Canada's regional reconnaissance geochemical drainage sediment and water surveys; *in* EXPLORE Newsletter; 163, Association of Applied Geochemists, p. 1–10.
- McKee, L., 2010. 18 reasons for open publication of geoscience data; IEEE Earthzine, Open Geospatial Consortium. <https://earthzine.org/18-reasons-for-open-publication-of-geoscience-data/>. [accessed November 29, 2021].
- McKillup, S. and Dyar, M.D., 2012. *Geostatistics explained*; Cambridge University Press, 396 p. doi:10.1017/CBO9780511807558.
- Mingram, J., Negendank, J.F.W., Brauer, A., Berger, D., Hendrich, A., Köhler, M. and Usinger, H., 2007. Long cores from small lakes - recovering up to 100 m-long lake sediment sequences with a high-precision rod-operated piston corer (Usinger-corer); *Journal of Paleolimnology*, v. 37, p. 517–528. doi:10.1007/s10933-006-9035-4.
- of Canada, G., 2022. The Canadian critical minerals strategy; Government of Canada, Open file, 58 p.
- Ontario Securities Commission, 2011. OSC Bulletin, v. 34, Chap. Rules and Policies: NI 43-101 standards of disclosure for mineral projects, form 43-101F1 Technical Report and related consequential amendments; The Ontario Securities Commission, p. 7043–7086.

- Parry, S.J., 1997. Neutron activation analysis; *in* Modern analytical geochemistry, (ed.) R. Gill; Routledge, p. 116–134.
- Piercey, S.J., 2014. Modern analytical facilities 2. A review of quality assurance and quality control (QA/QC) procedures for lithochemical data; *Geoscience Canada*, v. 41, p. 75–88. [doi:10.12789/geocanj.2014.41.035](https://doi.org/10.12789/geocanj.2014.41.035).
- Plant, J.A., Hale, M. and Ridgeway, J., 1989. Regional geochemistry based on stream sediment sampling; *in* Proceedings of Exploration 87, v. 3, (ed.) G.D. Garland; Ontario Geological Survey, p. 384–404.
- Rasmussen, P.E., Villard, D.J., Gardner, H.D., Fortescue, J.A.C., Schiff, S.L. and Shilts, W.W., 1998. Mercury in lake sediments of the Precambrian Shield near Huntsville, Ontario, Canada; *Environmental Geology*, v. 33, p. 170–182. [doi:10.1007/s002540050236](https://doi.org/10.1007/s002540050236).
- Reimann, C., Fabian, K., Birke, M., Filzmoser, P., Demetriades, A., Négrel, P., Oorts, K., Matschullat, J. and deCaritat, P., 2018. GEMAS: Establishing geochemical background and threshold for 53 chemical elements in European agricultural soil; *Applied Geochemistry*, v. 88, p. 302–318. [doi:10.1016/j.apgeochem.2017.01.021](https://doi.org/10.1016/j.apgeochem.2017.01.021).
- Reimann, C., Filzmoser, P., Garrett, R. and Dutter, R., 2008. *Statistical data analysis explained*; John Wiley & Sons, Ltd., 343 p.
- Reimann, C., Filzmoser, P. and Garrett, R.G., 2005. Background and threshold: critical comparison of methods of determination; *Science of the total environment*, v. 346, no. 1-3, p. 1–16. [doi:10.1016/j.scitotenv.2004.11.023](https://doi.org/10.1016/j.scitotenv.2004.11.023).
- Reimann, C. and Garrett, R.G., 2005. Geochemical background - concept and reality; *Science of the total environment*, v. 350, no. 1-3, p. 12–27. [doi:10.1016/j.apgeochem.2017.01.021](https://doi.org/10.1016/j.apgeochem.2017.01.021).
- Rencz, A.N., Klassen, R.A. and Moore, A., 2002. Comparison of geochemical data derived from till and lake sediment samples, Labrador, Canada; *Geochemistry: Exploration, Environment, Analysis*, v. 2, p. 27–35. [doi:10.1144/1467-787302-003](https://doi.org/10.1144/1467-787302-003).
- Rollinson, H., 1993. *Using geochemical data: evaluation, presentation, interpretation*; Pearson, Prentice Hall, 352 p.
- Rose, A.W., Hawkes, H.E. and Webb, J.S., 1979. *Geochemistry in mineral exploration*; Academic Press Inc., London, 657 p. second edition.
- Rowland, A.P., 1997. Atomic absorption spectroscopy and other solution methods; *in* Modern analytical geochemistry, (ed.) R. Gill; Routledge, p. 67–86.
- Sahoo, P.K., Guimarães, J.T.F., Souza-Filho, P.W.M., Powell, M.A., da Silva, M.S., Moraes, A.M., Alves, R., Leite, A.S., Júnior, W.N., Rodrigues, T.M., Costa, V.E. and Dall’Agnol, R., 2019. Statistical analysis of lake sediment geochemical data for understanding surface geological factors and processes: an example from Amazonian upland lakes, Brazil; *CATENA*, v. 175, p. 47–62. [doi:10.1016/j.catena.2018.12.003](https://doi.org/10.1016/j.catena.2018.12.003).
- Sanford, R.F., Pierson, C.T. and Crovelli, R.A., 1993. An objective replacement method for censored geochemical data; *Mathematical Geology*, v. 25, no. 1, p. 59–80. [doi:10.1007/BF00890676](https://doi.org/10.1007/BF00890676).
- Santisteban, J.I., Mediavilla, R., Lopez-Pamo, E., Dabrio, C.J., Zapata, M., Garcia, M., Castano, S. and Martínez-Alfaro, P.E., 2004. Loss on ignition: a qualitative or quantitative method for organic matter and carbonate mineral content in sediments?; *Journal of Paleolimnology*, v. 32, no. 3, p. 287–299. [doi:10.1023/B:JOPL.0000042999.30131.5b](https://doi.org/10.1023/B:JOPL.0000042999.30131.5b).
- Seequent, 2021. *Geoscience data management report - 2021 and beyond*; Bentley systems company, 23 p.



- Shahrestani, S., Mokhari, A.R. and Alipour-Asll, M., 2018. Assessment of estimated bedrock and stream sediment geochemical backgrounds in catchment basin analysis; *Natural Resources Research*, v. 28, p. 1071–1087. doi:10.1007/s11053-018-9437-9.
- Sharpe, D.R., Klassen, R.A., Russell, H.A.J., Brennand, T.A., Dexter, K., Logan, C.E. and Stepner, D.A.J., 2019. Regional geochemical survey of earth materials in southern Ontario: a geochemical baseline for environmental and human health protection; Geological Survey of Canada, Open File 8452, 43 p. doi:10.4095/308373.
- Shilts, W.W. and Farrell, L.E., 1982. Subbottom profiling of Canadian Shield lakes - implications for interpreting effects of acid rain; Geological Survey of Canada, Open File 82-1B, 209-221 p. doi:10.4095/119342.
- Smol, J.P., Birks, H.J. and Last, W.M., 2001a. Tracking environmental change using lake sediments, volume 3: terrestrial, algal, and siliceous indicators; Springer, 371 p.
- Smol, J.P., Birks, H.J. and Last, W.M., 2001b. Tracking environmental change using lake sediments, volume 4: Zoological indicators; Springer, 217 p.
- Stern, G.A., Sanei, H., Roach, P., Delaronde, J. and Outridge, P.M., 2009. Historical interrelated variations of mercury and aquatic organic matter in lake sediment cores from a subarctic lake in Yukon, Canada: further evidence toward the algal-mercury scavenging hypothesis; *Environmental Science & Technology*, v. 43, p. 7684–7690. doi:10.1021/es902186s.
- Thompson, M., 1983. Control procedures in geochemical analyses; *in* *Statistics and Data Analysis in Geochemical Prospecting*, v. 2, (ed.) R.J. Howarth; Elsevier, p. 39–58. doi:10.1016/B978-0-444-42038-1.50008-3.
- Thompson, M. and Howarth, R.J., 1978. A new approach to the estimation of analytical precision; *Journal of Geochemical Exploration*, v. 9, no. 1, p. 23–30. doi:10.1016/0375-6742(78)90035-3.
- Timperley, M.H., Jonasson, I.R. and Allan, R.J., 1973. Sub-aquatic organic gels: a medium for geochemical prospecting in the southern Canadian Shield; *in* *Report of Activities, Part A; Paper 73-1A*, Geological Survey of Canada, p. 58–62.
- Tukey, J.W., 1977. *Exploratory data analysis*; Addison-Wesley, Reading Mass, 688 p.
- U.S. Environmental Protection Agency, 2020. Operating procedure: sediment sampling; U.S. Environmental Protection Agency, Laboratory services and applied science division, Open File LSASDPROC-200-R4, 17 p.
- U.S. Geological Survey, 2020. Data management website; U.S. Geological Survey. doi:10.5066/F7MW2G15.
- Valette-Silver, N.J., 1993. The use of sediment cores to reconstruct historical trends in contamination of estuarine and coastal sediments; *Estuaries*, v. 16, no. 3B, p. 577–588. doi:10.2307/1352796.
- van der Leeden, J., 1995. Géologie de la région du lac Mistinibi, Territoire du Nouveau-Québec; Ministère des Ressources Naturelles, Québec, Open File MB 95-45, 107 p.
- Van Metre, P.C., Wilson, J.T., Fuller, C.C., Callender, E. and Mahler, B.J., 2004. Collection, analysis, and age-dating of sediment cores from 56 U.S. lakes and reservoirs sampled by the U.S. Geological Survey, 1992-2001; U.S. Geological Survey, National Water-Quality Assessment Program, Open File 2004-5184, 187 p. doi:10.3133/sir20045184.
- Vann, J., Stephenson, P.R., Jackson, S. and Pilger, G., 2014. Common sense and good communication - effective team-based estimation and classification of mineral resources and ore reserves; *in* *Monograph 30 - Mineral Resource and Ore Reserve Estimation - The AusIMM guide to good practice*; AusIMM, second edition, p. 19–31.

- Walsh, J.N., 1997. Inductively coupled plasma-atomic emission spectrometry (ICP-AES); *in* Modern analytical geochemistry, (ed.) R. Gill; Routledge, p. 41–66.
- Wetzel, R., 2001a. Limnology - Lake and river ecosystems; Academic Press, Elsevier, 1003 p. third edition.
- Wetzel, R., 2001b. Past productivity: Paleolimnology; *in* Limnology - Lake and river ecosystems; Academic Press, Elsevier, third edition, p. 785–804.
- Wirth, K., 2020. X-ray fluorescence (XRF); Science education resource centre at Carleton College. [https://serc.carleton.edu/research\\_education/geochemsheets/techniques/XRF.html](https://serc.carleton.edu/research_education/geochemsheets/techniques/XRF.html). [accessed November 29, 2021].
- Zhang, S.E., Bourdeau, J.E., Nwaila, G.T. and Corrigan, D., 2021a. Towards a fully data-driven prospectivity mapping methodology: case study of the Southeastern Churchill Province, Quebec and Labrador; *Artificial Intelligence in Geosciences*, v. 2, p. 128–147. doi:10.1016/j.aiig.2022.02.002.
- Zhang, S.E., Nwaila, G.T., Bourdeau, J.E. and Ashwal, L.D., 2021b. Machine learning-based prediction of trace element concentration using data from the Karoo large igneous province and its application in prospectivity mapping; *Artificial Intelligence in Geosciences*, v. 2, p. 60–75. doi:10.1016/j.aiig.2021.11.002.