

Geoscience BC

Regional Geochemical Surveys

Standards of Execution for RGS/NGR Surveys

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1.0 Introduction

The following are the general standards of execution for completion of RGS/NGR geochemical surveys. RGS-type surveys funded by Geoscience BC are expected to adhere to the principle of these standards. Modification of the standards is allowable if outlined and justified in the funding proposal.

2.0 Sample Collection and Data Recording

2.1 Sample Location: Stream Sediments and Stream Waters

Proposed sample locations should be established before going to the field. However, a degree of flexibility is expected when selecting the actual sample site in the field relative to proposed sites.

The following factors should be considered in choosing a sample site:

- Active flowing first or second order streams that have a drainage basin area between 2 and 15 square kilometres.
- Within the active stream channel (subject to annual flooding).
- Approximately 50 metres upstream from sources of possible contamination, from a confluence and from a high tide mark
- Upstream from lakes, ponds and marshes.

Streams containing abundant fine-grained sediment (silts and clays) that have clean flowing water are the most preferred. Streams with contamination from mining activity, logging, road culverts, etc. are to be avoided either by choosing an alternate stream or sampling upstream from the identified source of contamination.

The proponent is expected to make every reasonable effort to collect a sample of sediment and water at every proposed site. If the stream is dry, the collection of only a sediment sample is acceptable. However, under no circumstance is it acceptable to collect only a water sample. The proponent is also required to collect a field site duplicate sediment and water sample at regular intervals (see section 2.5).

Sample Medium: Stream Sediment

A minimum of 50 grams of -80 mesh (< 0.177 mm) stream sediment is required for subsequent analyses and archiving. Typically a sample bag full of fine-grained sediment (2 kg) is needed to produce 50 grams of -80 mesh fraction. Only active (in the process of being hydraulically moved), fine-grained stream sediment found within the active stream channel is acceptable. Samples with a dominant component of gravel, sand, glacial rock flour, bank material, organics (weeds, roots or wood fragments) are not acceptable. Should only coarse sand and gravel be present and an alternate stream not available, then at least two full bags of sediment will be needed.

Sample Medium: Stream Water

Stream water sampling involves collecting unfiltered water into 250 millilitre PVC bottles at every sample site. The following factors should be considered when collecting acceptable water samples:

- Clear water devoid of suspended solids (organic material and sediment).
- Water bottles must be two-thirds full. Do not fill to maximum capacity.

2.2 Lake Sediments and Water

Lake sediments are typically taken from the centre of a lake that has a surface area of preferably less than 5 square kilometres and a depth of greater than 3 metres. Modification of these standards may be

necessary depending on the size and depth of lakes in the area. In some cases sub-basins within larger lakes can be sampled.

A bottom-valved, hollow-pipe sampler is used to collect one kilogram or so of wet lake sediment. The sampler is vented at the top, allowing the top few centimetres of sediment to escape so that possible contamination in the upper levels of sediment can be avoided. Typically, one kilogram of the organic gel, the preferred collection material, is about 95 per cent water, and once dried, about 50 grams of material remain for analysis.

Lake waters are collected from below the surface of the lake from the same location the sediment sample is collected. Samples are contained in PVC bottles.

2.3 Marking Site Locations

Stream sediment sample sites should be clearly marked in the field with materials that will remain visible for several years. The sample ID number must be written on the tag and the tag firmly attached to a permanent feature such as a tree. The tag must be clearly visible from the stream to allow easy follow-up in later surveys.

2.4 Sample Location Maps and Field Records

The proponent must ensure that all proposed sample locations are well laid out in advance of the survey and that actual sample locations are properly located in the field. The proponent should establish an effective numbering system for each area. Records of the sample media, sample site and local terrain should be recorded for each station.

2.5 Quality Control

One of the most important characteristics of RGS/NGR surveys is the structure of the sampling routine. Each block of 20 consecutive field numbers consists of 17 routine field samples, a field duplicate sample, a blind (analytical) duplicate sample and a control reference sample. The field duplicate sample is a separate sample collected at one of the 17 routine sites, at the discretion of the sampling team. One number, always the first in a block of 20 (i.e. 001, 021, 041, etc.) is reserved for a blind duplicate. The sample preparation laboratory splits a sample in the block, preferably one of the field duplicate samples, and places one of the splits into the blind duplicate position. A randomly pre-selected number within a block of 20 is reserved for a control reference sample. Control reference samples are lake or stream sediments with well-established analytical values. A control sample may be available from the BC Geological Survey to the successful proponent(s).

Field duplicates, blind duplicates and control reference samples are incorporated in every block of 20 samples, and are used to monitor and control sampling and analytical variance. As a result of stringent quality control and consistency of analytical methods over time, it is possible to generate a regional compilation for an element with minimal boundary effects between different surveys using the same analytical method.

3.0 Sample Preparation and Analysis

3.1 Sample Preparation

Sediment samples should be air dried between 30°C and 50°C in a sample dry facility set up in the field. Material finer than 1 millimetre can be recovered in the field by sieving each sample through a -18 mesh ASTM screen. Samples may be dried at the laboratory, but extra care must be taken to avoid broken bags or contaminated samples. At the laboratory, the -80 mesh (<177 µm) fraction is to be obtained by dry

sieving and is to be used for all subsequent analytical determinations. Any remaining -80 mesh sediment and a representative sample of +80 to -18 mesh fraction is to be archived for future analyses.

3.2 Analytical Quality Control

Control reference material and analytical duplicate samples will be inserted into each analytical block of twenty sediment samples to monitor analytical variation (see section 2.5)

3.3 Sediment Sample Analysis

The analytical methods to determine elements in stream and lake sediments are aqua regia digestion-inductively coupled plasma mass spectroscopy (ICPMS), aqua regia digestion-inductively coupled plasma emission spectroscopy (ICPES) and epi-thermal instrumental neutron activation analysis (INAA). A complete list of elements and stated detection limits are given in Table 1 and 2. While INAA is a non-destructive method and estimates the 'total' element concentration, it cannot measure lead and copper. These elements are determined on a separate sample by aqua regia-ICPMS. Aqua regia digestion, while very effective for dissolving gold, carbonates and sulphides in a sample, cannot completely break down alumino-silicate, oxide and other refractory minerals such as barite. Therefore, the element concentrations determined by aqua regia-ICPMS are considered to be 'partial'. As a result, elements by ICPMS may be determined at lower concentrations relative to INAA.

3.3.1 Inductively Coupled Mass Spectroscopy

A split of the -80 mesh material is to be analyzed for 37 elements including aluminum, antimony, arsenic, barium, bismuth, cadmium, calcium, chromium, cobalt, copper, gallium, gold, iron, lanthanum, lead, magnesium, manganese, mercury, molybdenum, nickel, phosphorus, potassium, scandium, selenium, silver, sodium, strontium, sulphur, tellurium, thallium, thorium, titanium, tungsten, uranium, vanadium and zinc. Typically a 1 gram sample is leached with 6 millilitres of hydrochloric acid, nitric acid and distilled, deionized water (2:2:2 v/v) at 95°C for one hour and then diluted to 20 millilitres. The solution is analysed by ICPMS and ICPES.

Table 1: Detection Limits: ICPMS (Sediments)

Element		Detection Limit	Units	Method
Aluminum	Al	0.01	%	ICPMS
Antimony	Sb	0.02	ppm	ICPMS
Arsenic	As	0.1	ppm	ICPMS
Barium	Ba	0.5	ppm	ICPMS
Bismuth	Bi	0.02	ppm	ICPMS
Cadmium	Cd	0.01	ppm	ICPMS
Calcium	Ca	0.01	%	ICPMS
Chromium	Cr	0.5	ppm	ICPMS
Cobalt	Co	0.1	ppm	ICPMS
Copper	Cu	0.01	ppm	ICPMS
Gallium	Ga	0.2	ppm	ICPMS
Gold	Au	0.2	ppb	ICPMS
Iron	Fe	0.01	%	ICPMS
Lanthanum	La	0.5	ppm	ICPMS

Lead	Pb	0.01	ppm	ICPMS
Magnesium	Mg	0.01	%	ICPMS
Manganese	Mn	1	ppm	ICPMS
Mercury	Hg	5	ppb	ICPMS
Molybdenum	Mo	0.01	ppm	ICPMS
Nickel	Ni	0.1	ppm	ICPMS
Phosphorus	P	0.001	%	ICPMS
Potassium	K	0.01	%	ICPMS
Scandium	Sc	0.1	ppm	ICPMS
Selenium	Se	0.1	ppm	ICPMS
Silver	Ag	2	ppb	ICPMS
Sodium	Na	0.001	%	ICPMS
Strontium	Sr	0.5	ppm	ICPMS
Sulphur	S	0.02	%	ICPMS
Tellurium	Te	0.02	ppm	ICPMS
Thallium	Tl	0.02	ppm	ICPMS
Thorium	Th	0.1	ppm	ICPMS
Titanium	Ti	0.001	%	ICPMS
Tungsten	W	0.2	ppm	ICPMS
Uranium	U	0.1	ppm	ICPMS
Vanadium	V	2	ppm	ICPMS
Zinc	Zn	0.1	ppm	ICPMS

3.3.2 Instrumental Neutron Activation Analysis

Weighed and encapsulated samples are to be packaged for irradiation along with internal standards and international reference materials. Samples and standards are irradiated together with neutron flux monitors in a reactor. After a suitable decay period, samples are to be measured on a high-resolution germanium detector. Typical counting times are 500 seconds. Elements determined by INAA include: antimony, arsenic, barium, bromine, cadmium, cerium, cesium, chromium, cobalt, europium, gold, hafnium, iridium, iron, lanthanum, lutetium, molybdenum, nickel, rubidium, samarium, scandium, selenium, silver, sodium, tantalum, tellurium, terbium, thorium, tin, tungsten, uranium, ytterbium, zinc and zirconium.

3.3.3 Other Sediment Analysis

Loss-on-ignition is determined using a 500 mg sample. The sample, weighed into a 30 ml beaker, is placed in a cold muffle furnace and heated at 500°C for four hours, cooled to room temperature and then re-weighed (GRAV).

Fluorine is determined using a method described by Ficklin (1970). A 250 mg sample is sintered with 1 gram of a flux consisting of two parts by weight sodium carbonate and one part by weight potassium nitrate. The residue is then leached with water. The sodium carbonate is neutralized with 10 ml 10% (w/v) citric acid and the resulting solution is diluted to 100 ml with water. The pH of the solution should range from 5.5 to 6.5. The fluoride content of the test solution is measured using a fluoride ion electrode (ION). Standard solutions contain sodium carbonate and citric acid in the same quantities as the sample solution.

Table 2: Detection Limits: INAA, F and LOI in Sediments, and Un-Filtered Waters

Element		Detection Limit	Units	Method
Antimony	Sb	0.1	ppm	INAA
Arsenic	As	0.5	ppm	INAA
Barium	Ba	50	ppm	INAA
Bromine	Br	0.5	ppm	INAA
Cerium	Ce	5	ppm	INAA
Cesium	Cs	0.5	ppm	INAA
Chromium	Cr	20	ppm	INAA
Cobalt	Co	5	ppm	INAA
Europium	Eu	1	ppm	INAA
Gold	Au	2	ppb	INAA
Hafnium	Hf	1	ppm	INAA
Iron	Fe	0.2	%	INAA
Lanthanum	La	2	ppm	INAA
Lutetium	Lu	0.2	ppm	INAA
Rubidium	Rb	5	ppm	INAA
Samarium	Sm	0.1	ppm	INAA
Scandium	Sc	0.2	ppm	INAA
Sodium	Na	0.02	%	INAA
Tantalum	Ta	0.5	ppm	INAA
Terbium	Tb	0.5	ppm	INAA
Thorium	Th	0.2	ppm	INAA
Tungsten	W	1	ppm	INAA
Uranium	U	0.2	ppm	INAA
Ytterbium	Yb	2	ppm	INAA
Sample Weight	Wt	0.01	gm	GRAV
Fluorine	F	10	ppm	ION
Loss on Ignition	LOI	0.1	%	GRAV
pH	pH			GCE
Uranium	UW	0.05	ppb	LIF
Fluoride	FW	20	ppb	ION
Sulphate	SO4	1	ppm	TURB

3.4 Water Analysis

Unfiltered stream water samples are analyzed for pH, conductivity and fluoride.

Hydrogen ion activity (pH) was measured with a combination glass-calomel electrode and a pH meter (GCE).

Fluoride was determined by ion selective electrode (ION). A 20 millilitre aliquot of the sample was mixed with 20 millilitres of TISAB II (total ionic strength adjustment buffer) buffer solution. Fluoride was determined with an Orion fluoride electrode in conjunction with a Corning ion meter.

4.0 Additional Information

Additional information about BC's RGS program can be found here:

<http://www.em.gov.bc.ca/Mining/Geosurv/Geochinv/geochem.htm>

Additional information about the federal NGR program can be found here:

http://gsc.nrcan.gc.ca/geochem/ngr/index_e.php