DOC-2016-298

Methylmercury Monitoring Plan

For Surface Water Quality

Muskrat Falls Reservoir, Churchill River and Lake Melville

Initial Plan Prepared: October 17, 2016
Revised: December 19, 2016
Revised: September 29, 2017
Revised: June 23, 2020
Revised: September 30, 2020

Introduction:

Environmental monitoring generally is comprised of three media – water, sediment and biota. Water and sediment monitoring is regulated by the Department of Municipal Affairs and Environment while biota monitoring is regulated by the Federal Department of Fisheries and Oceans.

Biota tissue testing for methylmercury is essential as the tissue is the ultimate receptor for any environmental change and the best indicator to monitor potential impacts on consumer health. As per the Aquatic Environmental Effects Monitoring Plan (AEEMP), subsequent reports and other data, methylmercury data is available for biota (fish and seal). In addition, in July 2016, the Department of Fisheries and Oceans advised the proponent of required modifications to the AEEMP to include sampling in the eastern portion of Lake Melville, as well as modifications to reporting protocols. The AEEMP and related addendums can be found at: https://muskratfalls.nalcorenergy.com/environment/generation/

As the AEEMP monitoring focuses primarily on biota, it was agreed that additional methylmercury monitoring of water and sediment from upstream of the proposed Muskrat Falls Reservoir to the downstream outlet of Lake Melville would provide valuable information. Under Section 31 of the *Water Resources Act*, the Minister may order a licensee or other person to undertake a required level of water quality monitoring. Nalcor has agreed to engage an independent consultant to implement this monitoring plan for the Lower Churchill Project, focusing solely on water and sediment, to augment the information collected under the AEEMP.

While neither the Churchill River nor Lake Melville are sources of drinking water for any community, methylmercury information in water and sediment could be used as an early indicator for methylmercury in biota tissue, consumption of which might lead to health issues. The early indicator information from this plan, together with biota tissue testing data from the AEEMP, will provide the relevant provincial and federal agencies important information so any necessary action to protect human health can be taken at the earliest opportunity.

1. Objectives:

The objective of this plan is to assess change in the level of methylmercury in water and sediment due to the creation of the Muskrat Falls Reservoir. The approach that will be used to assess these changes is to directly measure the net change of methylmercury in water and sediment at various locations from upstream of the proposed Muskrat Falls Reservoir to the downstream outlet of Lake Melville. Any changes in methylmercury concentrations in the

upstream environment will provide an early indicator for potential changes in the downstream environment.

The plan will initially monitor temporal and spatial aspects of ambient water and sediment quality through the collection of grab samples with special emphasis on testing for total mercury and methylmercury. Section 3 outlines the procedural details for water and sediment samples to be collected in order to capture baseline, inundation and post-inundation methylmercury data.

The sampling locations, frequency and duration may be adjusted on an adaptive basis as results become available. The overall plan will be revisited for appropriate changes once the monitoring results related to the first phase of flooding have been analyzed.

2. Oversight

This monitoring plan will be overseen by the Department of Municipal Affairs and Environment, until the establishment of the Independent Expert Advisory Committee (IEAC).

3. Methodology:

The environmental monitoring proposed under this plan will focus on water and sediment grab sampling at sites which are selected in a scientific manner to capture the physiographic diversity of the project area.

Sampling sites identified in this plan are based on the physiographic (features and attributes of land surface) and hydrologic (rainfall, runoff, inflow, outflow, etc.) features of the area. The plan has also taken into consideration spatial and temporal factors. In order to capture the vertical profile of methylmercury, six sites (one site in the Muskrat Falls reservoir and five sites in Lake Melville) have been identified for multiple depth sampling.

The Department of Municipal Affairs and Environment requires that all monitoring results adhere to the *Accredited Laboratory Policy*. The objective of the policy is to ensure that environmental information produced and provided to the Province is comparable, of known quality and adequate for its intended purpose, thereby providing a reliable and harmonized basis for characterization and management of the Newfoundland and Labrador environment.

In accordance with the policy, the Department requires the use of laboratories which have a recognized form of laboratory accreditation to perform the required analyses. Accreditation obtained from an accreditation body that is a signatory to the International Laboratory Accreditation Cooperation (ILAC) Agreement and based on ISO 17025 is considered a recognized form of accreditation. The Canadian Association for Laboratory Accreditation

(CALA) is signatory to ILAC. There are several laboratories in Canada that are accredited by the Canadian Association for Laboratory Accreditation (CALA) for methylmercury in water, soil and/ or tissue. The selected laboratory provides direction for sample collection, storage and handling to ensure the integrity of the sample. Deviations from the accredited method, quality protocols, QA/QC or sample integrity concerns are reported by the laboratory as part of adherence to the international standard on which the accreditation is based.

The *Accredited Laboratory Policy* document is available on the departmental website at:
http://www.mae.gov.nl.ca/env_protection/lab_policy.pdf

Sample collection methodology and protocols for water and sediment sampling are provided in Appendix A. The Scope of Accreditation and Certification for the selected laboratory (Flett Research Ltd.) analyzing methylmercury in water and sediment is contained in Appendix B.

4. Monitoring Guidance Framework:

The following guiding principles will be taken into consideration during the implementation of this plan.

- As shown in Figure 1, the Department of Municipal Affairs and Environment currently operates five real time water quantity and quality monitoring stations within the Lower Churchill project area as well as two real-time weather stations. Grab water samples are also collected at the five real-time water monitoring stations during the ice free period from June to October. All these monitoring activities, which are separate from this monitoring plan, will continue, and the collected data will be available to all stakeholders.

- Approximate sample locations, sampling frequency and duration are indicated in Tables 1 & 2 and Figure 2. Using a GPS or similar device, the coordinates of each sampling location shall be recorded and submitted to the Department of Municipal Affairs and Environment. The location of each sampling point shall remain consistent over the life of the monitoring programs, unless otherwise approved by the Department of Municipal Affairs and Environment.

- Water samples are required to be collected in representative areas.

- Baseline water samples are to be collected at all sampling sites - Sites #N.1 to #N.13 (as indicated in Figure 2) and analyzed for total mercury and methylmercury, preferably one week apart if possible, but no less than 48 hours apart. Selected samples will be analyzed

for dissolved methylmercury as well as for methylmercury on suspended particulate matter. Other chemical parameters to be analyzed are identified in Appendix C.

  - Note: Baseline data has already been collected (October 14 & 16 and November 5) for the majority of sites, with the exception of sites #N.6, #N.8, #N.9, #N.11 and #N.12. Sites which already meet the baseline minimum requirement as stated in Table 2 need not be resampled.

- During inundation and post-inundation, water samples are to be collected at all sampling sites - Sites #N.1 to #N.13 (as indicated in Figure 2) and analyzed for total mercury and methylmercury. Samples will be split and analyzed for dissolved and suspended methylmercury concentrations separately. Other chemical parameters to be analyzed are identified in Appendix C.

- The feasibility of sediment sampling at the sites indicated on the map will be determined by flow regimes, depth and substrate condition in consultation between the proponent and the Department of Municipal Affairs and Environment. Other chemical parameters to be analyzed from sediment samples are identified in Appendix C.

- Select water samples (as indicated in Tables 1 and 2 and in red in Figure 2) will be required to be taken at multiple depths. In freshwater, the preferable locations will be near the surface, mid water column, and near the bottom, depending on the depth of the water at the location. Multiple depths sampling in Lake Melville should be targeted, with samples collected at 1m and at a depth dependent on halocline/thermocline at the time of sampling.

- If any samples cannot be collected for any reason, this must be communicated by the proponent to the Department of Municipal Affairs and Environment, with the appropriate justification.

- A statistical power analysis provides a guide for the design and planning of scientific studies and is used to indicate the sample size needed to detect environmental change. The sampling encompasses 13 different sites with a minimum of 10 samples collected at each site. Based on the Oct 14, 2016 and Oct 16 sampling data made available from Nalcor, methylmercury levels are approximately 0.026 ± 0.0241 ng/L for Lake Melville. The methylmercury levels are approximately 0.018 ± 0.0083 ng/L for Lower Churchill River. For Lake Melville methylmercury, with 10 samples in total taken from each lake location, there is a power of 0.93 to detect a difference of 0.0483 ng/L or more between each lake location. A sample size of 10 results in a power of 0.99 to detect differences in concentration greater than 0.0483 ng/L at any one lake location. For Churchill River methylmercury, with 10 samples taken in total from each river location, there is a power of 0.92 to detect a difference of 0.0167 ng/L or more between each river location. A sample size of 10 results in a power of 0.99 to detect differences in concentration greater than 0.0167 ng/L at any one river location. The detected difference is sufficiently lower

than the CCME guideline for aquatic life of 4 ng/L for methylmercury. Based on this analysis, and performing the power analysis using geographic location, and various water bodies (lakes, river) the monitoring plan has sufficient statistical power to detect changes in water quality from baseline conditions for each station in this plan based on existing methylmercury concentrations. More detail on the power analysis is provided in Appendix D.

- Chemical analysis of the water samples shall be carried out by Flett Laboratories, which is a commercial laboratory with a recognized form of accreditation for methylmercury (see Appendix B). The proponent shall ensure the detection limits to be used by the accredited laboratory are acceptable to the Department of Municipal Affairs and Environment. The method detection level is specified as 0.01 ng/l. Samples may be split and analyzed separately at a different accredited lab periodically for quality assurance purposes.

- As they become available, sample analysis results are to be provided to the Department of Municipal Affairs and Environment, using the attached template (see Appendix E). Laboratory Certificates of Analysis will be required to be submitted as supporting documentation. Results will be made publicly available (subject to development of appropriate communications plans).

- The proponent shall bear all expenses incurred in carrying out the environmental monitoring, required analyses and reporting.

- The overall monitoring plan will be revisited for appropriate changes once the monitoring results related to the first phase of flooding have been analyzed.

- Monitoring will be re-evaluated as results are made available to determine if the sampling program needs to be modified or extended.

- The Department of Municipal Affairs and Environment may order the proponent to alter the monitoring program or require additional testing at any time under several circumstances including when there is potential for an adverse environmental effect.

- The proponent may, at any time, request that the monitoring program or requirements in terms of sampling frequency and locations be altered by requesting the change in writing to the Department of Municipal Affairs and Environment with sufficient justification.

5. Considerations:

The following factors should be taken into consideration in overseeing the implementation of this monitoring plan:

- Sample collection will require samplers to cover a length of over 200 km in rough terrain.
- Weather conditions and site accessibility may play a significant role in whether planned samples can be collected or not.
- Occupational health and safety related measures must be a priority when implementing the sampling program.
- Analytical lab requirements for sample storage, holding time and shipping may require adjustments to the sampling frequency.
- Sediment sampling will take place only at selected sites depending on flow regime, depth and substrate conditions.

Addendum (September 29, 2017)

As recommended by the scientific sub-committee of the Independent Experts Advisory Committee (IEAC), and accepted by the Minister of Municipal Affairs and Environment on September 29, 2017, the surface water quality sampling and testing is revised as follows:

- The frequency of weekly water samples may be reduced to semi-monthly water samples when the water temperature is below 6°C and there are no significant changes in the reservoir water level.
- Lower (more sensitive) method detection limits (MDL) for total mercury, sulphate, total phosphorous, and total suspended sediments in water and total mercury in sediment shall be used in the laboratory analysis.

Addendum (June 23, 2020)

Following the accreditation of AGAT Laboratories (Calgary), mercury and methylmercury analysis no longer needed to be sub-contracted to Flett Research Ltd and instead samples could be analyzed by AGAT. Detection limits remain the same, namely 0.010 ng/L. A memo on mercury analysis, CALA scope of Accreditation and Certificate of Accreditation are attached as Appendix F.

As a result of COVID-19, travel restrictions were put in place for sampling teams and airline connections for the shipping of samples (without exceeding sample hold time) became challenging. Additionally, Universal Helicopters closed and therefore helicopters with floats became unavailable. As a result, sampling locations and collection methods had to be modified. A memo on *Logistic Considerations for Ongoing Methylmercury Water Sampling* is attached as Appendix G.

Addendum (September 30, 2020)

At the request of the Department of Environment, Climate Change and Municipalities, further information about the *AGAT Laboratories Methylmercury Reference Method and QA/QC Procedures* was attached as Appendix H. This information is provided as a letter to Mr. James H McCarthy, Senior Associate Biologist, Wood PLC. dated September 28, 2020.

Table 1 – Sampling Locations & Coordinates (as per Figure 2)

Area	Station Number & Location		Northing	Easting	Zone
Upstream of Reservoir	N.1 – Below Grizzle Rapids		5869577	606592	20
Within Reservoir	N.2* – Reservoir below Pinus River		5877481	619387	20
	N.3* – Reservoir between Pinus River & Upper Brook		5886480	628976	20
	N.4 – Above Muskrat Falls	Top	5902568	647135	20
		Mid			
		Bottom			
River below Reservoir	N.5 – Below Muskrat Falls		5901860	651010	20
	N.6 – Causeway		5904834	666977	20
	N.7 – English Point		5913613	687135	20
Lake Melville to Outlet	N.8 – Goose Bay (East of Rabbit Island)	1m	5920014	693950	20
		Targeted Depth (Above Halocline)**			
	N.9 – Inflow to Lake Melville	1m	5937268	303217	21
		Targeted Depth (Above Halocline)**			
	N.10 – Middle of Lake Melville	1m	5954514	302600	21
		Targeted Depth (Above Halocline)**			
	N.11 – Middle of Lake Melville	1m	5953492	335067	21
		Targeted Depth (Above Halocline)**			
	N.12 – Northeastern Lake Melville (near Neveisik Island)	1m	5971440	365225	21
		Targeted Depth (Above Halocline)**			
	N.13 – Near Rigolet (Lake outflow)		6002098	405601	21

*As the inundation of the reservoir is anticipated to progress slowly, it is acceptable to commence the sampling (sites #N.2 & #N.3) after the sampling location has been inundated using a staggered approach.

** Sampling depth will be determined on a sample-by-sample basis according to in-situ location of halocline.

Table 2: Sampling Frequency & Duration

Station Number & Location			Frequency & Duration	Total Samples
N.1–Below Grizzle Rapids		Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.2*–Reservoir below Pinus River		Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.3*–Reservoir between Pinus River & Upper Brook		Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.4–Above Muskrat Falls	Top	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
	Mid	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
	Bottom	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.5–Below Muskrat Falls		Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.6–Causeway		Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.7–English Point		Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.8–Goose Bay (East of Rabbit Island)	1m	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
	Targeted Depth	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.9 – Inflow to Lake Melville	1m	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
	Targeted Depth	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.10–Middle of Lake Melville	1m	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
	Targeted Depth	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.11–Middle of Lake Melville	1m	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
	Targeted Depth	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.12–Northeastern Lake Melville (near Neveisik Island)	1m	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
	Targeted Depth	Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10
N.13 – Near Rigolet (Lake outflow)		Baseline	Minimum twice before headpond formation (48+ hours apart)	2
		Inundation/Post-Inundation	Weekly (10 weeks)	10

Notes:

- *As the inundation of the reservoir is anticipated to progress slowly, it is acceptable to commence the sampling after the sampling location has been inundated using a staggered approach.
- During and after inundation, water samples collected should be analyzed as split samples whereby the dissolved vs. suspended concentrations are measured separately.
- Sediment samples are to be collected where feasible.
- The overall plan will be revisited for appropriate changes once the initial sampling results related to the first phase of flooding have been reviewed.

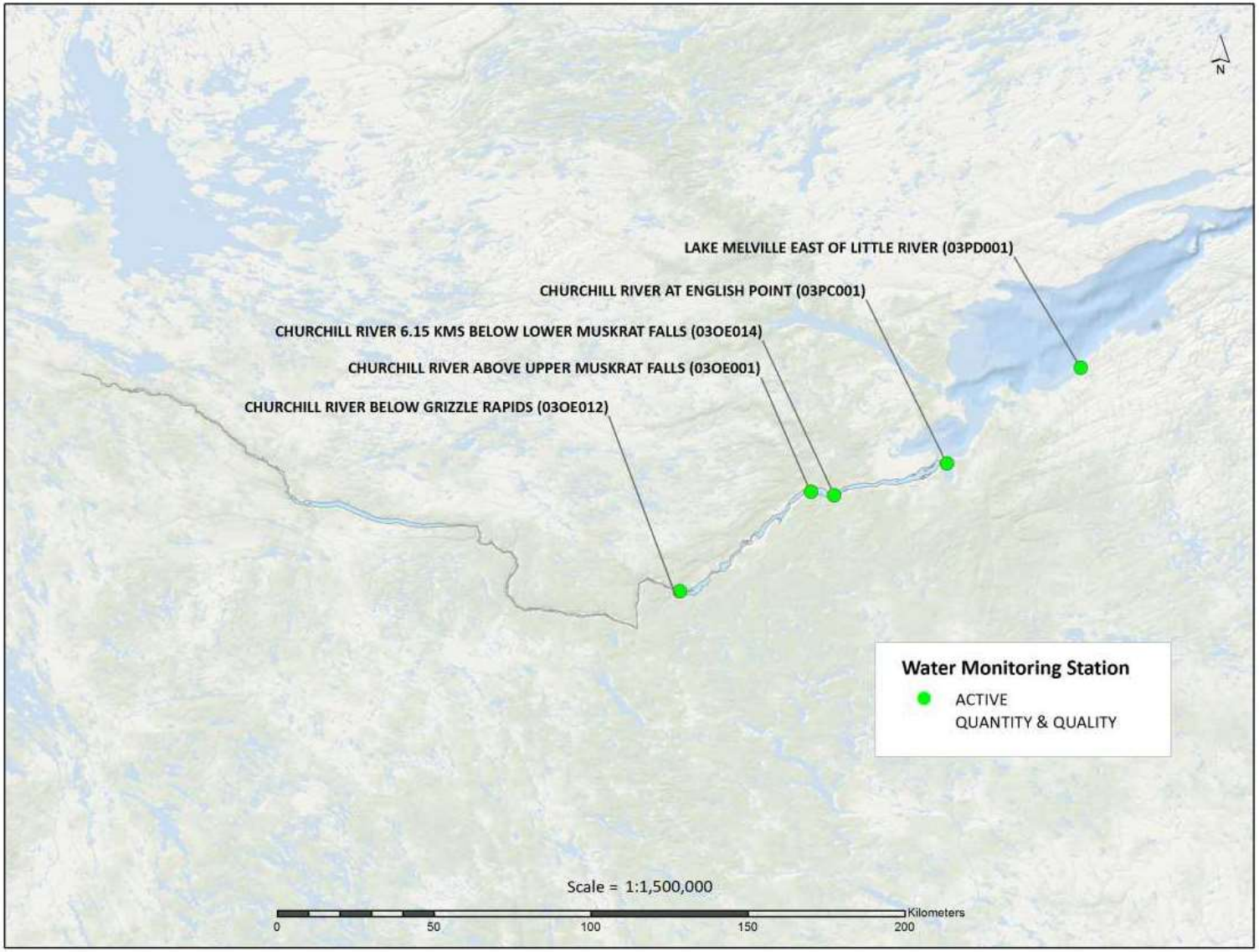

Figure 1: Current real-time water quality monitoring stations in the Lower Churchill Project Area

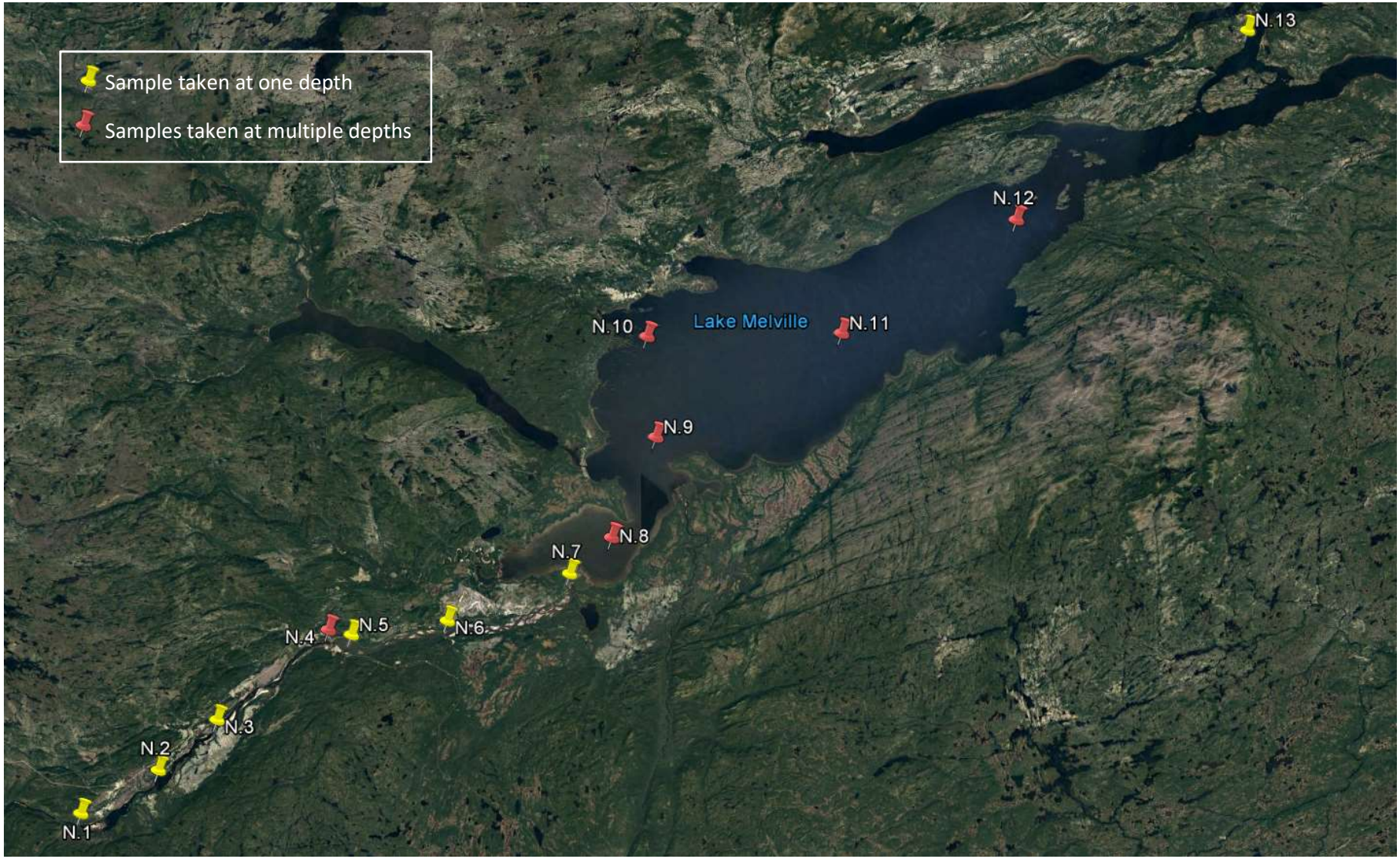

Figure 2: Methylmercury Monitoring stations in Churchill River and Lake Melville

Appendix A: Sampling Protocols and Methods

Work Instruction

Methylmercury in Water and Sediment Sampling

Document number:	WI-73-01
Document owner:	Matthew Gosse
Document author:	Matthew Gosse
Revision:	01
Revision date:	11 October 2016
This document supports	Baseline sampling for the Lower Churchill Project (Environmental Effects Monitoring).
About this document:	Collection techniques and shipping methods for methylmercury sampling
Who this document applies to:	This document applies to field staff completing methylmercury sampling
Responsibility for this document:	The functional responsibility for the development, review and maintenance of this document rests with the Project Manager/Field Supervisor

CONTENTS

1 DEFINITIONS

The following terms are used within this document.

Term	Definition
Methylmercury	Bioavailable, organic form of mercury. Often associated with bioaccumulation
Niskin Bottle	Sampling apparatus for collecting water samples from various depths in the water column
Sediment Grab	Sampling apparatus (i.e. Ponar or Eckman dredge) for collecting sediment samples from benthic habitats

2 INSTRUCTIONS

This work instruction is specific to the program being completed by Nalcor Energy in relation to the Muskrat Falls portion of the Lower Churchill Project. This program will consist of baseline sampling (prior to inundation) and post-inundation sampling. This program has been designed to address concerns regarding methylmercury accumulation and to satisfy monitoring objectives outlined by the Province of Newfoundland and Labrador, Department of Environment and Climate

Change (DECC).

2.1 Sample Location

Sample locations have been outlined by DECC and revised as per the Work Plan above.

2.2 Sampling Timeline

Baseline sampling will be completed prior to inundation, with a total of two sampling events, not less than 48 hours apart. Post inundation sampling will be completed as per the schedule outlined by the Work Plan.

2.3 Baseline sampling

Two baseline samples will be completed at each of the sampling locations, no less than 48 hours apart. These samples will be collected within two weeks of reservoir inundation.

2.4 Sampling Equipment

Various equipment will be required to complete the sampling program. This includes:

1. Water sample bottles (provided by Flett Research)
2. Sediment sample jars (to be picked up from Agat Depot in Goose Bay)
3. Sediment Grab (ponar or Eckman dredge)
4. Niskin Bottle
5. Cooler for sample storage
6. Sharpie for labelling (ideally done previous night)
7. Nitrile gloves
8. GPS
9. PPE (safety glasses, PFD)

2.5 Travel to Sampling Sites

Given the distance between sampling locations, the majority of the sampling will be completed with the use of a helicopter. While in the helicopter, all work will be completed under the supervision and direction of the pilot. This will also act as the sampling platform for baseline sampling, and weekly post inundation sampling.

Daily (i.e., every three days) post inundation sampling will be completed with the use of a boat (see sample breakdown in Work Plan).

2.6 Collection of Water Samples

Standard water sampling protocols apply to sampling for methylmercury in water. Many of the identified sample sites are surface water. In order to maintain sample integrity, clean nitrile gloves must be worn for each sample (i.e. changed between sample locations). Below are steps to sample collection:

1. Remove sample bottle from cooler and labelled.
2. Clean nitrile gloves are put on.
3. Bottle is lower to water surface, uncapped, and rinsed twice with sample water.
4. Bottle is then lowered to approximately 10cm below water surface and filled to the indicated fill line. Bottle must be held near the bottom to avoid contamination near the mouth.
5. Bottle is capped, returned to Ziploc bag, and placed in the cooler for storage and transport.

Two sample locations require collection from various depths within the water column. These samples will be collected using a Niskin bottle. Again, clean nitrile gloves (i.e. changed between each sample) must be worn to maintain sample integrity and avoid contamination. The steps for sampling at depths are below:

1. Set up Niskin bottle, and check functionality.
2. Rinse Niskin bottle with sample water.
3. Lower Niskin to desired depth.
4. Release messenger.
5. Retrieve Niskin.
6. Remove sample bottle from Ziploc bag and Label it.
7. Don clean nitrile gloves.
8. Uncap sample bottle, and rinse twice with sample water.
9. Fill sample bottle

10. Bottle is capped, returned to Ziploc bag, and placed in the cooler for storage and transport.

Once collection is completed, all bottles must be capped and secured, and placed in the cooler for safe transport.

Detailed collection methods are presented in the attached document from Flett Research.

2.7 Collection of Sediment Samples

Similar to the water sampling outlined above, clean nitrile gloves must be worn in order to collect sediment samples. Sediment samples are collected using a sediment grab (i.e. Ponar or Eckman dredge). Below are the steps to collecting sediment:

1. Set up dredge and ensure functionality. This step will vary depending upon the sampling apparatus being used. In the case of a Ponar, ensure messenger is attached to line.
2. Lower dredge to sediment.
3. Trigger dredge (release messenger for ponar, or release tension on line for Eckman).
4. Bring dredge back to surface.
5. Collect sediment sample from middle of dredge and place in a lab provided bottle.
6. Rinse dredge and repeat as necessary.

Detailed collection methods are presented in the attached document from Flett Research.

2.8 Sample Labelling

All samples should be labeled with the sample site, sample depth (where applicable) and sample date.

2.9 Sample Storage

All sediment samples must be stored cool (i.e. in the refrigerator). Water samples must be frozen, and shipped in a frozen state.

2.10 Chain of Custody and Air Canada Cargo Shipping

An Agat Chain of Custody must be included in each shipment.

Air Canada Cargo (ACC) waybills can also be completed prior to arrival at the airport.

3 REFERENCES

4 REVISION HISTORY

Rev. No.	Rev. Date	Revision By	Description of Revisions
01	October 11, 2016	M. Gosse	Initial document creation

5 APPENDICES

Flett Research Sampling Procedure - Mercury Water Sample Collection and Handling

Flett Research Sampling Procedure – Mercury: Sediment/Soil Sample Collection and Handling

FLETT RESEARCH LTD

440 DeSalaberry Ave., Winnipeg, Manitoba, Canada R2L 0Y7 Fax/Tel (204)667-2505
E-mail: flett@flettresearch.ca Webpage: http://www.flettresearch.ca

MERCURY – Sediment/Soil Sample Collection and Handling

The method by which you collected your samples (dredge, grab, core etc) helps us to determine how your samples should be handled once they arrive at the lab. We also need to know if you require the results to be reported on a wet or dry weight basis; this is normally specified by the regulatory body you are reporting to. If samples have been taken by dredge* or they are coarse and/or inhomogeneous in nature, they should be freeze dried and ground prior to analysis. Drying, grinding or even sieving may be a requirement of your regulator.

Precautions:

Of particular concern to the laboratory with soil samples is the level of contamination. We are an ultra-trace laboratory and a site that is high in total mercury could put our analyser out of service for weeks. A sample from a site that is rich in heavy metals could do similar damage.

Keep in mind that only small amounts of sample are analysed so it is important that samples are as homogeneous as possible. Mercury tends to be associated with small particles and thus it makes sense to do size separations on gravely soils, so as to determine the relative distribution of fines to coarse material. The fine material can be measured for Hg content, and the overall Hg content of the soil can be calculated according to the relative fraction of the fine materials. Similarly, samples with the presence of organic matter such as roots, grass, sticks etc make obtaining a representative subsample very difficult. The client should discuss their requirements before analyses are attempted e.g. are larger twigs, rocks, shells etc to be excluded from analysis? Typically the finer materials are analysed after being ground or ball milled

*When samples are collected by dredge, they lose unknown amounts of water and as a result the %Loss on Drying value is not representative of the original sample. Because the %LOD value is not accurate, we dry the sample by freeze drying prior to analysis. The samples are analyzed dry and reported in ng Hg/dry gram. Analysis of dry material allows meaningful comparisons between dredge samples. If freeze dried sediment is coarse and inhomogeneous, grinding by ball mill may be required.

Sample requirements:
As long as we have at least 1 gram (preferably at least 5 grams) of reasonably homogenous wet sediment, we can perform the mercury and methyl mercury analyses as needed.

Sampling Containers:

Unsaturated sediment/soil samples may be placed into 250mL, tightly sealing, pre-cleaned, HD polyethylene wide mouth jars or new Zip Loc bags (double). Do not fill jars more than 2/3 full.

In general, plastic bags are not considered a preferred method of storage because they are more permeable than other heavier vessels (mercury levels changing from the original levels in the sample, through loss or gain of mercury through its container). Also, when frozen, bagged samples are fragile and may tear when knocked together during transit, or when being moved in a freezer.

Pre-cleaned, wide mouth glass jars may also be used but are susceptible to breaking particularly when full of frozen sediment. Glass sediment jars should be filled no more than 50% full of wet sediment.

Sampling:

Wear clean vinyl disposable gloves when handling the sample and tools. A stainless steel spoon, washed with detergent and tap or clean lake water, can be used for dispensing dredge sediment or grab soil into 250 ml precleaned HD polyethylene jars. It is recommended that gloved hands be washed in clean water between samples. If this is not possible, then new clean gloves should be put on between samples.

Sampling and analysis of core samples often require special care. It is best to contact our lab before core sampling.

Polyethylene sediment jars should be filled no more than 2/3 full and glass sediment jars no more than 50% full of wet sediment.

Make certain the caps are tightly closed and that no sediment covers the sealing surfaces of the jar before closing. Label the cap and the jar body. We recommend the use of permanent inks for labeling as paper labels may have trouble staying adhered to the frozen jars.

Preservation:

Wet sediments can be preserved either through refrigeration or freezing, see holding time below.

Samples will normally be frozen at the laboratory so it is important to choose (and fill) sample containers carefully, such that the possibility of damage to the container and possible loss of sample due to breakage from expanding ice be minimized when the sample is frozen. Polyethylene sediment jars should be filled no more than 2/3 full and glass sediment jars no more than 50% full of wet sediment.

Holding time:

Recommended holding time for frozen (~ -20°C) sediment is 180 days or 28 days when refrigerated (~1-6°C). All samples should be kept in the dark. Dried sediments are generally considered to have an extended storage lifetime (>>180 days).

Transportation:

Please try to ship the samples on either a Monday, Tuesday or a Wednesday, as this will ensure that they do not arrive at our laboratory on a weekend when no-one will be here to receive them. In general pack samples in the same manner in which you received them when returning them to Flett.

Make sure the jars are upright and well padded against breakage during transport. Ship to the lab in a cooler on blue ice if overnight delivery is certain. If delivery could be longer, send on an appropriate weight of dry ice in a well insulated cooler. The coolers should be well taped to prevent opening and to exclude dust during transport.

NOTES:

Please fill in the accompanying data sheet and return it with the samples.

Please notify us (email, phone or fax) when the samples are being shipped so that we can watch for them on our end and ensure that they arrive on time. If we experience any problems in receiving the samples we can trace them immediately and notify you of any potential problems.

FLETT RESEARCH LTD

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MERCURY WATER SAMPLE COLLECTION AND HANDLING

Natural water samples are the most sensitive to contamination because the concentration of mercury is often very low. Most clean natural surface waters contain less than 15 ng/L total Hg with typical measures between 0.5 and 10 ng/L. Shield lakes usually contain less than 5 ng/L. Lakes Huron and Superior, and a number of northern Canadian lakes, often contain less than 1 ng/L total Hg. Good deionized (DI) water from our lab is usually between 0.03 and 0.07 ng/L total Hg.

It takes very little mercury contamination to significantly raise the apparent concentration of mercury in the sample therefore it is very important that the bottles, sampling apparatus (if used), and handling practices introduce little additional mercury to the sample. Method blanks are regularly analysed in our lab and they show that typically less than 0.05 ng total Hg/L is introduced to the sample from the sample bottles or lab handling.

Risk of contaminating water samples during sampling is significant. The procedures in this document should help to minimize contamination. The use of proper sampling techniques will help to ensure the validity of test results and that the sample is a true representation of the source from which it was taken.

General Considerations for Low Level Mercury Sampling in Water:

A fibreglass boat is preferred for mercury sampling, simply because it metal free. A clean and dry boat will likely avoid more contamination problems than a dirty leaking boat (bailing, draining or pumping dirty water into the lake is not an option when sampling at the same site). If the exterior and interior of the boat are clean, contamination is less likely when a sample line accidentally comes in contact with the boat. Oil and grease, together with dust/soil, may be difficult to remove from sampling equipment and almost certainly contain significant amounts of mercury. If the boat is powered, the engine should not be leaking hydrocarbons into the water that is being sampled. Avoid sampling in the path that any boat has recently travelled.

If at anchor during sampling, use a clean anchor line. Anchor from the bow and sample near the bow, away from the engine but as far from the anchor line as possible. If possible, use an Echo-sounder to measure the water depth. This will allow you to avoid contacting the bottom if using a Kemmerer sampler; contacting the sediments will contaminate the water sample and almost certainly increase the apparent Hg concentration. If the bottom is hit by the Kemmerer, it will be necessary to rinse the sampler and change sampling locations.

If drifting with the wind during sampling, use a clean paddle at the stern to keep the stern (engine) into the wind; perform sampling from the leeward side of the boat because this will ensure that only uncontaminated water is being obtained.

If under power (moving forward) during sampling, go dead slow, into the current if present, and sample from the bow.

Sample at shallow depths before deeper depths at the same site. The "deep" water sample should be collected after all of the surface samples have been obtained, but before you deploy an anchor or use any other equipment that requires a line to be in deep water where you wish to sample. This is to avoid contamination of the mercury sample.

Avoid sampling in significant rain events because rainwater often contains much higher total Hg than surface waters.

Sample Bottles:

Samples should be collected only into rigorously cleaned Teflon bottles. Under no circumstances should ordinary plastic (i.e. polyethylene, polypropylene or PVC) containers be used, as they are very permeable to Hg gas from the air. Ashed or rigorously cleaned glass bottles with fluoropolymer lined caps are also acceptable for sample collection. PETG also works well for methyl Hg samples but is not suitable for total Hg samples.

It is critical that the bottles have tightly sealing caps to avoid diffusion of atmospheric Hg through the threads (Gill and Fitzgerald, 1985).

Use 125 mL bottles for Total Hg samples and 250 mL bottles for Methyl Hg, if possible.

Double-Bagged Bottles: These Teflon or glass sample bottles are clean on both the interior and exterior and enclosed in 2 plastic ziploc bags. Double-bagged bottles can be used for any type of sampling but may be less convenient to use than un-bagged or boxed bottles if a clean exterior is unnecessary. **Dip samples should only be carried out with double-bagged bottles** because they can be dipped into the sample medium and will not contaminate the water being sampled. During non-freezing weather, the bottle will usually be shipped containing clean 0.4 % HCl. During freezing weather, when the contents may be accidentally frozen, the bottles may be shipped empty.

Un-bagged and/or Boxed Glass Bottles: Pre-cleaned glass samples bottles from I-Chem have proven satisfactory when sample is poured or pumped into the bottles. They are **not suitable for dip sampling** because the exterior has not been cleaned. Glass bottles are usually shipped empty and may be packaged in lots of 12, in the manufacturer's original cardboard boxes, or may be packaged individually in bubble wrap. Sending the glass sample bottles un-bagged is advantageous because field crews do not have to unbag/rebag the bottles, which saves considerable time, particularly in cold weather.

Single-bagged PETG Bottles: Pre-cleaned PETG bottles have also been shown to work well for methyl Hg samples but are **not** suitable for total Hg samples.

Fill Volumes: Use the table below as guidance when filling sample bottles.

Bottle Type	Glass			PETG	Teflon		
Sample Type	Total Mercury		Methyl Mercury	Methyl Mercury **ONLY**	Total Mercury		Methyl Mercury
Season	Summer	Winter	All year	All year	Summer	Winter	All year
Fill to	~95%	~50-60% (if risk of freezing); otherwise ~90%	~50-60%	225 mL graduated line	~95%	~85%	~80-85%

Clean Hands/Dirty Hands Protocols: Trace level mercury samples are collected using rigorous ultra-clean protocols (Gill and Fitzgerald, 1985) which require two or more people with frequently changed, unpowdered clean-room gloves to work together. On site one person is designated as Clean Hands and a second person as Dirty Hands. Specific tasks are assigned to each sampler which helps to prevent contamination of samples. Clean Hands takes care of all sampling operations that involve items which come into contact with the sample and dirty hands takes care

of all sampling operations that involve contact with potential sources of contamination. More detailed instructions are provided in the sampling sections below.

Dip Sampling of Surface Water – Use Only Double-Bagged Bottles, or single-bagged PETG

Bottles for dip sampling are clean on both the interior and exterior and are enclosed in two plastic ziploc bags (one bag for PETG).

Bottles must not be labelled prior to use because they will be dipped into the sample medium. Bottles can be labelled with water proof marker or adhesive labels after sampling or labels can be added to the Ziploc bag. Teflon bottles from Flett are inscribed with a tracking ID which can be associated with the sample on the sample submission form.

Use 125 mL bottles for Total Hg samples and 250 mL bottles for Methyl Hg, if possible.

Samples are collected using rigorous ultra-clean protocols (Gill and Fitzgerald, 1985) which are summarized as follows:

1.) At least two persons, wearing fresh unpowdered clean-room gloves at all times, are required on a sampling crew.

2.) One person ("dirty hands") pulls a bagged bottle from the box/cooler, labels the outer bag with a waterproof maker and then opens the outer dirty bag, avoiding touching inside that bag.

3.) The other person ("clean hands") reaches in, opens the inner bag, and pulls out the sample bottle.

4.) The bottle is opened and the acidified water (if present) is discarded downstream of the sampling site.

5.) "Clean hands" rinses the bottle at least twice with sample water, discarding the rinse water downstream, and then fills the bottle, holding it near the bottom during filling with the mouth about 10 cm below the water surface facing into the current flow. When sampling from a boat (or aircraft), the boat should be slowly moving and the sample taken from the side of the boat closest to the direction of movement. Filling volume should be in accordance with the table above.

6.) The cap is replaced, firmly tightened and the bottle re-bagged in the opposite order from which it was removed.

7.) Clean-room gloves are changed between sample locations and whenever something not known to be clean is touched. If obvious contamination of the "clean hand" gloves has not occurred, they may be retained in a clean plastic bag for use by the "dirty hands" person at the next sampling location.

Pump Sampling Using Un-bagged Glass or single-bagged PETG or double-bagged Teflon Bottles

When using a peristaltic pump sample bottles are not dipped into the sample medium, and therefore only the bottle interior needs to be clean. Un-bagged or boxed glass bottles are normally sent. Bottles are clean on the interior but may have dust on the exterior surface. The exterior surface should be wiped with a clean paper towel before opening or handling by sampling crew who are wearing cleanroom gloves.

Glass bottles can be labelled with water proof marker or adhesive labels prior to use because they will not be dipped into the sample medium.

If Teflon bottles have been sent, they will normally be enclosed in 2 plastic Ziploc bags. They are clean on both the interior and exterior and can be used for dip sampling, or receiving water from a pump sampler. During non-freezing weather, the bottle will usually be shipped containing clean 0.4 % HCl. During freezing weather, when the contents may be accidentally frozen, the bottles may be shipped empty. It is preferable that marker or adhesive labels are not used on Teflon bottles; labels can be added to the Ziploc bag. Each Teflon bottle is inscribed with a tracking ID which can be associated with the sample on the sample submission form.

Use 125 mL bottles for Total Hg samples and 250 mL bottles for Methyl Hg, if possible.

Samples are collected using rigorous ultra-clean protocols (Gill and Fitzgerald, 1985) which are summarized as follows:

1.) It is recommended that at least 10 pump/tubing volumes of sample water be passed before beginning to rinse the bottles. It is best to have 2 sampling personnel, one who handles the pump and another who holds the bottle. Try to wash the exterior of the pump delivery tube by pointing the tube vertically like a fountain and allowing the exiting water stream to fall back onto the tube for several seconds.

2.) The bottle person, wearing cleanroom gloves, uncaps the bottle and if present, the acidified water inside the bottle is discarded downstream of the sampling site. The same person then places the bottle mouth in front of the sample water stream, being careful NOT to insert the tube into the bottle. This is to avoid contaminating the bottle with Hg which may be on the tube exterior walls. After filling to about 30% capacity, the bottle is swirled and the rinse water is discarded. A second rinse is similarly performed. Finally, the bottle(s) is/are filled with sample and tightly capped, with filling carried out according to the table above.

3.) Clean-room gloves are changed between sample locations and whenever something not known to be clean is touched. If obvious contamination of the "clean hand" gloves has not occurred, they may be retained in a clean plastic bag for use by the "dirty hands" person at the next sampling location.

*** The above are extracted from protocols of Nicolas Bloom, John Rudd and Bob Flett - Sept. 1993

Sampling with a Kemmerer sampler, Un-bagged Glass or single-bagged PETG or double-bagged Teflon Bottles

When a discrete sampler such as a Kemmerer is used, sample bottles are not dipped into the sample medium, and therefore only the bottle interior needs to be clean. It is common, when a client is using a Kemmerer or other similar sampler, to send them un-bagged glass sample bottles. There may be dust on the exterior surface. The exterior surface should be wiped with a clean paper towel before opening or handling by sampling crew who are wearing cleanroom gloves.

Glass bottles can be labelled with water proof marker or adhesive labels prior to use because they will not be dipped into the sample medium.

If Teflon bottles have been sent, they will normally be enclosed in 2 plastic Ziploc bags. They are clean on both the interior and exterior and can be used for dip sampling, or receiving water from a pump sampler. During non-freezing weather, the bottle will usually be shipped containing clean 0.4 % HCl. During freezing weather, when the contents may be accidentally frozen, the bottles may be shipped empty. It is preferable that marker or adhesive labels are not used on Teflon bottles; labels can be added to the Ziploc bag. Each Teflon bottle is inscribed with a tracking ID which can be associated with the sample on the sample submission form.

The main concern is to keep the Kemmerer bottle, messenger and sample line clean. When not actually in the water, the sampling equipment should be placed back into the clean bags in which it is stored and transported. None of the sampling equipment should be placed directly on a boat surface. The persons handling the sampler must wear clean disposable vinyl (or similar) unpowdered gloves and touch nothing else with their gloves. If they do touch surfaces not known to be clean, they should change their gloves for new ones before proceeding.

Use 125 mL bottles for Total Hg samples and 250 mL bottles for Methyl Hg, if possible.

Samples are collected using rigorous ultra-clean protocols (Gill and Fitzgerald, 1985) which are summarized as follows:

1.) At least two persons, wearing fresh unpowdered clean-room gloves at all times, are required on a sampling crew. **When using a discrete sampler such as a Kemmerer**, it will be easier to have 2 "clean hands" people: one to hold the sampler and another to handle and fill the sample bottle.
2.) One person ("dirty hands") pulls a bagged bottle from the box, labels the outer bag with a waterproof maker and then opens the outer dirty bag, avoiding touching inside that bag.
3.) The other person ("clean hands") reaches in, opens the inner bag, and pulls out the sample bottle.
4.) The bottle is opened and the acidified water (if present) is discarded downstream of the sampling site.
5.) "Clean hands" rinses the bottle at least twice with sample water from the Kemmerer, discarding the rinse water downstream, and then fills the bottle(s) with filling carried out according to the table above.
6.) The cap is replaced, firmly tightened and the bottle re-bagged in the opposite order from which it was removed.
7.) Clean-room gloves are changed between sample locations and whenever something not known to be clean is touched. If obvious contamination of the "clean hand" gloves has not occurred, they may be retained in a clean plastic bag for use by the "dirty hands" person at the next sampling location.

*** The above are extracted from protocols of Nicolas Bloom, John Rudd and Bob Flett - Sept. 1993

Preservation:

Please remember to fill samples bottles in accordance with the chart found earlier in this document to avoid loss of samples due to breakage, especially when freezing methyl Hg or shipping in cold weather.

Methyl mercury samples are preserved by refrigeration in the dark at about 4°C if the samples can be returned to the laboratory on ice packs within 48 hr of sampling. If methyl Hg samples cannot be returned within 48 hours (to meet the EPA recommended holding time), they must be frozen. In the frozen state the samples will be stable for many weeks. **If you are freezing the methyl Hg samples in glass bottles, it is best to lay them on their sides in the freezer, to help minimize the risk of bottle breakage.**

Total mercury samples need not be preserved as long as they are received by the lab within 28 days of sampling. **Do not freeze** total mercury samples because there is evidence that Hg^{2+} converts to Hg^0 during freezing and there is a loss of this volatile form of Hg upon thawing the sample.

Transportation:

Please try to ship the samples on either a Monday, Tuesday or a Wednesday, as this will ensure that they do not arrive at our laboratory on a weekend when no-one will be here to receive them. In general pack samples in the same manner in which you received them when returning them to Flett.

Methyl Mercury: When ready to ship, remove the methyl Hg samples from the refrigerator or freezer and place them in an **upright position** into the cooler supplied by the lab. **Any glass bottles should be placed in their bubble pouches** and should be well-packed such that they cannot be thrown around the cooler during transport. This is particularly important when shipping frozen samples and will help minimize the risk of bottle breakage (and possible loss of sample) during transport.

Teflon and PETG bottles are not as fragile as glass when frozen and do not require individual bubble pouches. Double-bag the bottle shipment in 2 new plastic garbage bags and place them in the cooler supplied by the lab. Add freezer packs (or dry ice if necessary, but not regular water ice) to keep samples frozen. Use additional soft padding to keep the freezer packs from contacting and possibly breaking the bottles. There should be no empty space in the cooler when it is ready to ship.

Total Mercury: The total Hg samples are similarly protected and placed in the cooler provided by the lab. This should be separate cooler from the methyl samples. During warm weather the cooler can be submitted without freezer packs but when shipping in freezing weather *room temperature* freezer packs should be included with the samples.

The coolers should be well taped to prevent opening and to exclude dust during transport.

NOTES:

For Methyl Mercury in glass bottles that are able to be frozen before transport – freeze samples on their sides reduce the risk of bottle breakage (when actually shipping, bottles should be in the upright position).

Please fill in the accompanying data sheet and return it with the samples.

Please return all extra bottles as **there will be a charge for all bottles not returned. The Teflon bottles cost more than $90 each.**

Please notify us (email, phone or fax) when the samples are being shipped so that we can watch for them on our end and ensure that they arrive on time. If we experience any problems in receiving the samples we can trace them immediately and notify you of any potential problems.

Appendix B: Flett Research Inc Scope of Accreditation and Certificate

CALA ACCREDITATION PROGRAM
FINAL SCOPE OF TESTING

3306

LABORATORY NAME: Flett Research Ltd.

MATRIX

Solids (Inorganic)

APPENDIX NO. / NAME

002 Mercury - Soil

METHOD	METHOD REFERENCE	LAB METHOD I.D.
CVAFS - DIGESTION	modified from ANAL. CHIM. ACTA 281: 135-152 and EPA 1631E	T00130

Parameters:

Mercury*

011 Mercury - Soil

METHOD	METHOD REFERENCE	LAB METHOD I.D.
MERCURY ANALYZER	modified from EPA 7473	T00210

Parameters:

Mercury*

015 Methyl Mercury - Soil

METHOD	METHOD REFERENCE	LAB METHOD I.D.
CVAFS - DISTILLATION (AUTOMATED)	modified from ANAL. CHIM. ACTA 281:135-152 and EPA 1630	M10240

Parameters:

Methyl Mercury

Solids (Radiochemistry)

APPENDIX NO. / NAME

017 Lead-210 (Po-210) - Soil

METHOD	METHOD REFERENCE	LAB METHOD I.D.
DIGESTION/DISTILLATION-ALPHA SPECTROSCOPY	modified from EAKINS and MORRISON. JOURNAL OF APPLIED RADIATION and ISOTOPES 29, 531-536	N20110

Parameters:

Lead-210 (Polonium-210)

018 Radionuclides - Soil

METHOD	METHOD REFERENCE	LAB METHOD I.D.
GAMMA SPECTROMETRY (HPGe)	modified from EML HASL-300 METHOD GA-01-R	N30120

Parameters:

Actinium-228
Americium-241
Bismuth-214
Cesium-134
Cesium-137
Cobalt-60
Lead-212
Lead-214
Potassium-40
Thorium-234

019 Radium-226 - Soil

METHOD	METHOD REFERENCE	LAB METHOD I.D.
DIGESTION-RN-222 EMANATION-ALPHA SPECTROMETRY	modified from MATHIEU BISCAYE, LUPTON and HAMMOND. HEALTH PHYSICS 55, 989-992	N40110

Parameters:

Radium-226 (Rn-222)

Tissue (Inorganic)

APPENDIX NO. / NAME

003

METHOD	METHOD REFERENCE	LAB METHOD I.D.
	modified from ANAL. CHEM. 48: 926-928 and EPA 1631E	T00110

Parameters:

Mercury

* CALA Proficiency Testing (PT) Program analyte

Current scope as of 3/3/2016

1

CALA ACCREDITATION PROGRAM
FINAL SCOPE OF TESTING

3306

LABORATORY NAME: Flett Research Ltd.

MATRIX

010 Mercury - Biological Tissue

METHOD	METHOD REFERENCE	LAB METHOD I.D.
MERCURY ANALYZER	modified from EPA 7473	T00210

Parameters:

Mercury

012 Methyl Mercury - Biological Tissue

METHOD	METHOD REFERENCE	LAB METHOD I.D.
CVAFS - DIGESTION	modified from EPA 1630 and CAN. J. FISH. AQUAT. SCI. 49:1010-1017	M 10220

Parameters:

Methyl Mercury

Water (Inorganic)

APPENDIX NO. / NAME

001 Mercury - Water

METHOD	METHOD REFERENCE	LAB METHOD I.D.
CVAFS - OXIDATION	modified from EPA 1631E	T00120

Parameters:

Mercury*

016 Methyl Mercury - Water

METHOD	METHOD REFERENCE	LAB METHOD I.D.
CVAFS - DISTILLATION	modified from EPA 1630	M10210

Parameters:

Methyl Mercury

† "OSDWA" indicates the appendix is used for the analysis of Ontario drinking water samples, which is subject to the rules and related regulations under the Ontario "Safe Drinking Water Act" (2002).

PT REQUIREMENTS: All tests appearing in the scope of testing must be supported by PT testing where available. Therefore, analytes with a status of Withdrawn, Suspended, or not yet proficient, will NOT appear on the Final Scope of Testing. Once proficiency has been achieved, the affected analyte(s) will appear on the Scope of Testing. Please refer to P02-03 *CALA Program Description - Proficiency Testing (PT) Requirements for Accreditation.*

The list of tests and measurement capabilities for which a laboratory is accredited can change at any time due to circumstances such as scope extensions, voluntary withdrawal of tests by the laboratory and suspension. Scopes are published by the CALA via the Internet at http://www.cala.ca/cala_directories.html

* CALA Proficiency Testing (PT) Program analyte

Canadian Association for Laboratory Accreditation Inc.

CALA

Certificate of Accreditation

Flett Research Ltd.
440 DeSalaberry Ave.
Winnipeg, Manitoba

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

CALA

Accreditation No.: A3306
Issued On: March 2, 2016
Accreditation Date: June 30, 2005
Expiry Date: August 31, 2018

President & CEO

CALA

APPENDIX C: Monitoring Parameters

Matrix	Parameter
Sediment	Methylmercury (ng/g)
	Total Mercury (mg/kg)
	Sulphide (mg/kg)
	Available Sulphur (mg/kg)
Water *Laboratory Analysis*	Dissolved MeHg (ng/L)
	Total MeHg (ng/L)
	Total Mercury (ng/L)
	Sulphate (mg/L)
	Sulphide (mg/L)
	Ammonia as N (mg/L)
	Total Kjeldahl Nitrogen as N
	Total Phosphorous as P (mg/L)
	Total Organic Carbon (mg/L)
	Dissolved Organic Carbon (mg/L)
	Total Suspended Solids (mg/L)
Water *In-Situ*	Conductivity (umho/cm)
	Dissolved Oxygen (mg/L)
	pH
	Salinity (ppt)
	Temperature (^{o}C)
	Total Dissolved Solids
	Turbidity (NTU)

APPENDIX D: POWER ANALYSIS FOR TOTAL AND METHYLMERCURY

SAMPLING SITE LOCATION

A total of 11 sites sampled by Nalcor on October 14 and 16, 2016 were used in this analysis. Ten samples for Lake Melville were used for the computation of the lake portion of the power analysis while 20 samples for Lower Churchill River were used for the computation of the river portion of the power analysis.

DATA ANALYSIS

In all cases, unfiltered data for methylmercury was used to perform the power analysis. The actual value for method detection limit (~) was used for statistical measurements and power analysis.

The count, mean and standard deviation of methylmercury for Lake Melville and the Lower Churchill River is shown below:

Statistics	**Lake Melville**	**Lower Churchill River**
Count	10	20
Mean (ng/L)	0.0260	0.0180
St. Dev. (ng/L)	0.0241	0.0083
Mean + 1 S.D.	0.0501	0.0263
Mean – 1 S.D.	0.0019	0.0097
Highest Difference in Mean (ng/L)	0.0483	0.0167

Methylmercury has a CCME aquatic life guideline of **4 ng/L**.

POWER ANALYSIS – Lake Melville

The following table shows the power analysis results for **Lake Melville** using 10 samples:

Power Test	**Methylmercury (ng/L)**
Using 10 samples per site	
Power (at one site) - One Sample t	0.99
Power (between each site) - ANOVA	0.93

POWER ANALYSIS – Lower Churchill River

The following table shows the power analysis results for the **Lower Churchill River** using 20 samples or using 10 samples:

	Methylmercury (ng/L)
Using 20 samples per site	
Power (at one site) - One Sample t	1
Power (between each site) - ANOVA	0.99
Using 10 samples per site	
Power (at one site) - One Sample t	0.99
Power (between each site) - ANOVA	0.92

CONCLUSION:

A statistical power analysis provides a guide for the design and planning of scientific studies and is used to indicate the sample size needed to detect environmental change. The sampling encompasses 13 different sites in lakes and rivers with a minimum of 10 samples collected at each site.

Based on the Oct 14, 2016 and Oct 16 sampling data made available from NALCOR, methylmercury levels are approximately 0.026 ± 0.0241 ng/L for Lake Melville. The levels are approximately 0.018 ± 0.0083 ng/L for Lower Churchill River.

For Lake Melville methylmercury, with 10 samples taken from each lake location, there is a power of 0.93 to detect a difference of 0.0483 ng/L or more between each lake location. A sample size of 10 results in a power of 0.99 to detect differences in concentration greater than 0.0483 ng/L at any one lake location.

For Churchill River methylmercury, with 10 samples taken from each river location, there is a power of 0.92 to detect a difference of 0.0167 ng/L or more between each river location. A sample size of 10 results in a power of 0.99 to detect differences in concentration greater than 0.0167 ng/L at any one river location.

The detected difference is sufficiently lower than the CCME guideline for aquatic life of 4 ng/L for methylmercury. Based on this analysis, and performing the power analysis using geographic location, and various water bodies (lake, river) the monitoring plan has sufficient statistical power to detect changes in water quality from baseline conditions for each station in this plan based on existing methylmercury concentrations.

APPENDIX E: Sample Submission Templates

Water:

				Laboratory Analysis											*In Situ*						
Sample Site	Sample Date	Sample Depth	Sample Type	Dissolved MeHg (ng/L)	Total MeHg (ng/L)	Total Mercury (ng/L)	Sulphate (mg/L)	Sulphide (mg/L)	Ammonia as N (mg/L)	Total Kjeldahl Nitrogen as N	Total Phosphorous as P (mg/L)	Total Organic Carbon (mg/L)	Dissolved Organic Carbon (mg/L)	Total Suspended Solids (mg/L)	Conductivity (umho/cm)	Dissolved Oxygen (mg/L)	pH	Salinity (ppt)	Temperature (°C)	Total Dissolved Solids (mg/L)	Turbidity (NTU)
Detection Limits				0.01	0.01	1.9	2	0.05	0.05	0.4	0.03	0.5	0.5	5	NA	NA	NA	NA	NA	NA	0.1
1	14-Oct-16	Surface	Baseline																		
2	14-Oct-16	Surface	Baseline																		
3	14-Oct-16	Surface	Baseline																		
4	14-Oct-16	Surface	Baseline																		
4	14-Oct-16	Mid	Baseline																		
4	14-Oct-16	Bottom	Baseline																		
5	14-Oct-16	Surface	Baseline																		
6	14-Oct-16	Surface	Baseline																		
7	14-Oct-16	Surface	Baseline																		
8	14-Oct-16	Surface	Baseline																		
9	14-Oct-16	Surface	Baseline																		
10	14-Oct-16	Surface	Baseline																		
10	14-Oct-16	Mid	Baseline																		
10	14-Oct-16	Bottom	Baseline																		
11	14-Oct-16	Surface	Baseline																		
1	16-Oct-16	Surface	Baseline																		
2	16-Oct-16	Surface	Baseline																		
3	16-Oct-16	Surface	Baseline																		
4	16-Oct-16	Surface	Baseline																		
4	16-Oct-16	Mid	Baseline																		
4	16-Oct-16	Bottom	Baseline																		
5	16-Oct-16	Surface	Baseline																		
6	16-Oct-16	Surface	Baseline																		
7	16-Oct-16	Surface	Baseline																		
8	16-Oct-16	Surface	Baseline																		
9	16-Oct-16	Surface	Baseline																		
10	16-Oct-16	Surface	Baseline																		
10	16-Oct-16	Mid	Baseline																		
10	16-Oct-16	Bottom	Baseline																		
11	16-Oct-16	Surface	Baseline																		

Sediment:

Sample Site	Sample Date	Sample Type	Laboratory Analysis			
			MethylMercury (ng/g)	Total Mercury (mg/kg)	Sulphide (mg/kg)	Available Sulfur (mg/kg)
		RDL	1.3	0.05	0.05	0.05
1	14-Oct-16	Baseline				
2	14-Oct-16	Baseline				
3	14-Oct-16	Baseline				
4	14-Oct-16	Baseline				
4	14-Oct-16	Baseline				
4	14-Oct-16	Baseline				
5	14-Oct-16	Baseline				
6	14-Oct-16	Baseline				
7	14-Oct-16	Baseline				
8	14-Oct-16	Baseline				
9	14-Oct-16	Baseline				
10	14-Oct-16	Baseline				
10	14-Oct-16	Baseline				
10	14-Oct-16	Baseline				
11	14-Oct-16	Baseline				
1	16-Oct-16	Baseline				
2	16-Oct-16	Baseline				
3	16-Oct-16	Baseline				
4	16-Oct-16	Baseline				
4	16-Oct-16	Baseline				
4	16-Oct-16	Baseline				
5	16-Oct-16	Baseline				
6	16-Oct-16	Baseline				
7	16-Oct-16	Baseline				
8	16-Oct-16	Baseline				
9	16-Oct-16	Baseline				
10	16-Oct-16	Baseline				
10	16-Oct-16	Baseline				
10	16-Oct-16	Baseline				
11	16-Oct-16	Baseline				

June 1, 2020

Jackie Wells
Environmental Specialist
Environment & Sustainability Department
Nalcor Energy
St. John's, NL

Dear Jackie,

RE: Mercury analysis by AGAT Labs

Wood was approached by AGAT labs in February 2019 regarding their recent certification for Hg analysis to the specifications and detection limits required by our monitoring programs. Up to March 21, 2019, AGAT were sub-contracting out the lower detection limit and methylmercury (MeHg) analysis to Flett Research Labs in Winnipeg. As you know, there have been some challenges in getting samples to Winnipeg within recommended hold times and we have paid a premium on flights and additional costs for filtration prior to shipping.

AGAT provided the raw lab data to Wood Environment & Infrastructure Solutions (Wood) for analysis of comparability. The following is a summary of that analysis.

Data Comparison

The data provided was analyzed using R statistical software and "lme4" package. Each sample collected in the field was split into two separate samples in the lab and analyzed at both the AGAT and Flett labs. The data was prepared by removing all samples below detection limits as well as its paired sample so that only paired samples from the same location with recorded concentrations were used. Data was checked for normality and if non-normal, Wilcoxon signed rank tests were used. All analysis was completed at p value < 0.05 level of significance.

Dissolved MeHg in Water

Mean MeHg in water samples (n=18 pairs) from Flett and AGAT were 0.02984 and 0.03889 ng/L, respectively. The data were not normally distributed therefore Wilcoxson signed rank tests were completed to determine if the mean difference between the paired samples were significantly different from zero. The test resulted in a p value of 0.076 (>0.05) and therefore no significant difference between lab results.

Total Hg in Water

Mean Total Hg in water samples (n=59) from Flett and AGAT were 1.411 and 1.567 ng/L, respectively. The data were normally distributed therefore paired t-tests were completed to determine if the mean difference between the paired samples were significantly different from zero. The test resulted in a p value of <0.001 (<0.05) and therefore a significant difference between lab results was detected. Based on the mean values, the AGAT lab appears to be estimating slightly higher than Flett.

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Registered in Canada
No. 773289-9; GST: 899879050 RT0008; DUNS: 25-362-6642

Continued...

The values from both certified labs appear reasonable for all analysis; however, AGAT's analysis appears to have a slightly higher concentrations of Total Hg in water than Flett.

Ongoing Data Validation

It was confirmed that AGAT will provide the Flett lab with at least three sub-samples per set (i.e., three water samples for every week of collection) for Quality Assurance (QA) purposes. This will be used as a direct check on sample results.

Closure

After review of the data provided, we feel the AGAT lab analysis is reasonable and comparable to previous Flett results; however, AGAT does appear to have higher Total Mercury results on paired samples. Having AGAT complete these analyses locally will allow greater control over sample integrity and reporting; however, we would highly recommend that the Flett sub-samples continue to be part of the protocol, whenever hold times allow. The Flett replicate samples have been requested for sites N1 (control), N4 surface (headpond), and N5 (just downstream of headpond) so that the most likely sites for comparisons are captured. AGAT has agreed to this.

We will continue to compare AGAT and Flett paired sub-samples for similarity as the sample size increase.

Should you have any questions or require clarification, please feel free to contact the undersigned at your convenience.

Yours sincerely,

Wood Environment & Infrastructure Solutions,
a Division of Wood Canada Limited

Prepared by:

James McCarthy, MSc, CFP
Associate Biologist, Ecosystem Lead

Reviewed by:

Michael Teasdale, M.Sc.
Senior Biologist

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Canadian Association for Laboratory Accreditation Inc.

CALA

Certificate of Accreditation

AGAT Laboratories
AGAT Laboratories (Calgary)
11 Morris Drive
Unit 122
Dartmouth, Nova Scotia

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

Accreditation No.: A3588
Issued On: January 22, 2019
Accreditation Date: December 7, 2007
Expiry Date: July 22, 2021

Andrew M. Adams
President & CEO

CALA

CALA
Canadian Association for Laboratory Accreditation Inc.

CALA Directory of Laboratories

Membership Number: 3588
Laboratory Name: AGAT Laboratories (Dartmouth)
Parent Institution: AGAT Laboratories Ltd.
Address: 11 Morris Drive Unit 122 Dartmouth NS B3B 1M2
Contact: Ms. Pam Reyno
Phone: (902) 468-8718
Fax: (902) 468-8924
Email: reyno@agatlabs.com; vhill@agatlabs.com

Standard: Conforms with requirements of ISO/IEC 17025
Clients Served: All Interested Parties
Revised On: May 8, 2020
Valid To: July 22, 2021

Scope of Accreditation

Air (Inorganic)
Ammonia - Air (212)
INOR-121-6003, INOR-121-6022; SM 4500 NH3 F
COLORIMETRIC
Ammonia

Air (Inorganic)
Total Suspended Particulates - Air [Particulate] (128)
INOR-121-6041; EPA 5
GRAVIMETRIC
Particulate

Air (Organic)
Total Petroleum Hydrocarbons (TPH) - Air (087)
ORG-120-5109; modified from ATLANTIC RBCA GUIDELINES FOR LABORATORIES TIER 1/TIER 2 PETROLEUM HYDROCARBONS METHODS and NIOSH 1500
GC/FID
Aliphatic >C10-C12
Aliphatic >C12-C16
Aliphatic >C16-C21
Aliphatic C6-C8
Aliphatic >C8-C10
Aromatic >C10-C12 fraction
Aromatic >C12-C16 fraction
Aromatic >C8-C10 fraction
Benzene
>C10-C21

† "OSDWA" indicates the appendix is used for the analysis of Ontario drinking water samples, which is subject to the rules and related regulations under the Ontario "Safe Drinking Water Act" (2002).

The list of tests and measurement capabilities for which a laboratory is accredited can change at any time due to circumstances such as scope extensions, voluntary withdrawal of tests by the laboratory and suspension. Scopes are published by the CALA via the Internet at http://www.cala.ca/cala_directories.html

Water (Inorganic)
Mercury - Water (032)
MET-121-6100, MET-121-6107; modified from EPA 245.1 and SM 3112 B
COLD VAPOUR AA - DIGESTION
Mercury

Water (Inorganic)
Mercury - Water (200)
MET-121-6114, MET-121-6115; EPA 1631
COLD VAPOUR ATOMIC FLUORESCENCE SPECTROPHOTOMETRY
Mercury

Water (Inorganic)
Methyl Mercury - Water (203)
MET-121-6116, MET-121-6117; EPA 1630
COLD VAPOUR ATOMIC FLUORESCENCE SPECTROPHOTOMETRY
Methyl mercury

Water (Inorganic)
Oil and Grease - Water (066)
ORG-120-5105; modified from EPA 1664A
GRAVIMETRIC
Mineral Oil and Grease
Total Oil and Grease

Water (Inorganic)
pH - Water (035)
INOR-121-6001; modified from SM 4500-H+ B
pH METER
pH

Water (Inorganic)
Phenols - Water (211)
INOR-121-6011; SM 5530
AUTO COLOR
Total Phenolics

Water (Inorganic)
Phosphate - Water (034)
INOR-121-6012; modified from EPA 365.1 and SM 4500-P F
AUTO COLOR
Phosphate

Water (Inorganic)
Reactive Silica - Water (057)
INOR-121-6028; EPA 370.1 and SM 4500-SIO2 E
AUTO COLOR
Reactive Silica

Water (Inorganic)
Redox Potential (ORP) - Water (122)
INOR-121-6042; modified from SM 2580 B
ELECTRODE
Redox Potential

† "OSDWA" indicates the appendix is used for the analysis of Ontario drinking water samples, which is subject to the rules and related regulations under the Ontario "Safe Drinking Water Act" (2002).

The list of tests and measurement capabilities for which a laboratory is accredited can change at any time due to circumstances such as scope extensions, voluntary withdrawal of tests by the laboratory and suspension. Scopes are published by the CALA via the Internet at http://www.cala.ca/cala_directories.html

wood.

Memo

To: Jackie Wells

From: James McCarthy

cc:

Date: May 27, 2020

Re. Logistic Considerations for Ongoing Methylmercury Water Sampling, Muskrat Falls

Jackie

As per our discussion regarding the unfortunate closing of Universal Helicopters, the team has been formulating a revised logistic plan associated with the possible challenge of sampling without a helicopter on fixed floats. At the moment, a revised sampling plan includes use of a helicopter that is not on fixed floats as well as our boats. Provided below is a summary of our plans for each existing sample location.

The key challenges in providing samples for every location, with the use of a helicopter on skids, will be meeting the sample hold times, achieving sub surface samples (i.e. mid, bottom and halocline), and ensuring no contamination from disturbed bottom sediments.

If you have any questions or require additional details, please feel free to contact me at your convenience.

Yours sincerely,

Wood Environment & Infrastructure Solutions,

a Division of Wood Americas Limited

Prepared by:

Jim McCarthy, MSc, CFP
Senior Associate Biologist

Reviewed by:

Matthew Gosse, B.Sc
Biologist

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Continued...

Table 1: Logistic solutions for each sample location, Muskrat Falls Water Sampling.

Site	Sample Collection
N1	Current location requires helicopter on floats to safely sample (no nearshore landing locations). However, the control location only needs to be upriver of the MF Reservoir therefore, the site can be moved to a location that can be reached by helicopter on skids and sampled from shore. Shore sample Work Instruction (WI) will be implemented (see attached).
N2	Current location requires helicopter on floats to safely sample (no nearshore landing locations). This site can be accessed by boat via the Pinus River launch. The challenge of returning to Goose Bay and shipping to the lab to meet the 24 hour hold time for dissolved MeHg will be met by limiting the number of samples collected via the Pinus River launch (N2, N3 – possibly N4: see below). This requires a team of two; however, weekly sampling will begin soon which will require two team members to collect the full sample suite.
N3	See N2 above.
N4 Surface, Mid, Bottom	Current location requires helicopter on floats to safely sample (has sub-surface sample requirements). This site can be accessed by boat via the Muskrat Falls Facility launch or the Pinus River launch. The onsite launch reduces travel time to ease shipping logistics; however, with COVID-19 it is suggested that the Pinus launch be used, if weather is acceptable. It is estimated that all Reservoir sites could be completed within one day from the Pinus River launch given proper weather conditions. This requires a team of two; however, weekly sampling will begin soon which will require two team members to collect the full sample suite.
N5	Current location requires helicopter on floats to safely sample; however, a revised site can be located nearshore. Therefore, site can be accessed by helicopter on skids. Shore sample WI will be implemented (see attached).
N6	Similar to N5 above. Alternatively, this site can be accessed by vehicle at the Black Rock Bridge.
N7	Similar to N5 above. Alternatively, this site can be accessed by vehicle from Mud Lake Road.
N8 Surface	Current location requires helicopter on floats to safely sample, particularly for the sub-surface halocline sample; however, a revised site can be located nearshore to collect the surface sample using a helicopter on skids. Shore sample WI will be implemented (see attached).
N8 Halocline	Based on weather conditions, an alternative will be to collect the surface and halocline samples from boat when conditions allow.
N9 Surface	See N8 Surface above.
N9 Halocline	See N8 Halocline above.
N10 Surface	See N8 Surface above.
N10 Halocline	See N8 Halocline above, but only if weather conditions are exceptional.
N11 Surface	See N8 Surface above.
N11 Halocline	Given the distance needed to travel by boat, this sample may not be collected consistently. It will be sampled when it can be coupled with other boat-based sampling in the area.
N12 Surface	See N8 Surface above.
N12 Halocline	Given the distance needed to travel by boat, this sample may not be collected consistently. It will be sampled when it can be coupled with other boat-based sampling in the area.
N13	Current location can be sampled from shore using a helicopter on skids. Shore sample WI will be implemented (see attached).
Dup	Can be easily collected randomly at any of the successfully accessed sample sites.

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wood.

Collecting Water Samples from Shore

Document number:	**WI-AQENV-XX**
Applicability:	Ecosystems Group, St. John's, NL
Document owner:	James McCarthy
Document reviewer:	James McCarthy
Document author:	Matthew Gosse
Revision:	01
Revision date:	27-May-2020
This document supports	NA
Responsibility for this document:	The functional responsibility for the development, review and maintenance of this document rests with the Ecosystem Group Lead.

Contents

1 Purpose and Scope

To collect surface water samples from shore while avoiding potential contamination of sediments. Sediment contamination is a particular concern when dealing with certain analytes (i.e. total suspended solids or total methylmercury), or analytes with low detection limits (i.e. total mercury or total phosphorus). This work instruction is prepared specifically for the sampling setup used for the Muskrat Falls Methylmercury Water Sampling program which utilizes a pump and hose for water collection, however, it can be adapted for other sampling apparatuses as needed.

2 Roles and Responsibilities

Function	Role/Responsibility
Field staff	• Collect water samples • Pack, transport and ship samples to the lab
Data analyst	• Enter data and ensure QA/QC • Complete any analysis that is required

3 Procedure

3.1 Methodology

Prior to beginning the field portion of the water collection, all sample bottles should be pre-labelled and grouped by site.

For the Muskrat Falls Methlymercury Water Sampling Program, the majority of the shoreline sites would be accessed by helicopter on skids. Upon landing near the collection site, once the pilot has given the all clear to exit the aircraft, gather the sampling gear and walk it to the shoreline. Look for an appropriate sampling area, away from eddies, heavy wave action or anything else that would create visibly increased turbidity or sample integrity (i.e. any objects in the water). Ideally, areas with steady drop offs and larger substrate would be preferable, as there is less likelihood of disturbing any fine substrates. Figure 1 shows a general sampling setup using a pump that will not disturb substrates.

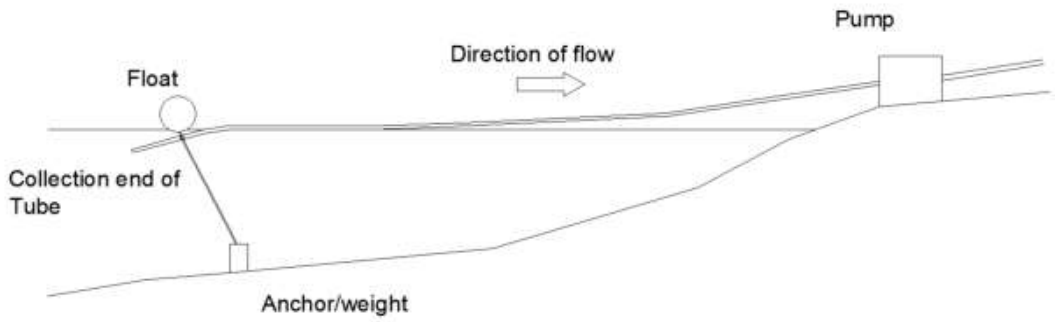

Figure 1: General schematic of shoreline sampling set up using pump

Once an appropriate location is found, the hose, with anchor and float attached, can be thrown away from the shoreline to collect water below the surface, without disturbing the bottom. When you are satisfied with the position of the house, turn on the pump, and allow it to flush out before collection. Fill each bottle directly from the pump. Using a clear plastic bottle (if one is included) inspect the sample for any particulates, beyond what would be expected in the sample. Take a picture of the bottles and the site fpr a record of sample condition.

In situ water quality (i.e. collected with a YSI), can be set up at this time. Move away for the the pump set up along the shoreline, and place the probe in the water. Follow directions for the unit being used. Ensure you allow time for the recording to stabilize.

If when arriving on site, no suitable sites can be identified, make note of the conditions present, and take photographs. If a near by site looks better, consider moving collection to that area. Consider the intention of the program prior to moving sites, and discuss any modifications with Project Manager prior to submitting for analysis.

3.2 Considerations for Field Collection

- Avoid wading in the water near the collection site as much as possible.
- Where possible, have the hose intake pointing upriver. If in a lake or bay, avoid areas of heavy wave action.
- If contamination is expected, stop collection and move the hose to a better suited location.

3.3 Shipping of Water Samples

Prior to beginning the water sampling program, be sure you have finalized logistics around shipping, and ensure you are familiar with the various hold times. Take care to ensure that samples are packed well with ice and any packing materials that may be required (i.e. bubble wrap or cardboard).

3.4 Materials

- Pump
- Hose
- Anchor
- Field Book
- Water Quality Meter (i.e. YSI or Hydrolab)
- Field Book and Pencil

- Float
- Laboratory supplied sample bottles
- Camera
- GPS

3.5 Health and Safety Considerations

- Installation will include work in/near open water and along stream/pond banks. Ensure PFD is worn, and proper footwear to keep you dry, and support ankles on uneven surfaces.
- Ensure nitrile gloves are worn at all times during sampling. This ensures sample integrity, and adds a layer of protection when dealing with samples requiring preservatives.

4 Revision History

Rev no.	Rev date	Rev by	Summary of changes
Rev. 01	May 27, 2020	Jim McCarthy	Draft review for Nalcor submission

Appendix H – AGAT QA/QC

AGAT Laboratories

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11 Morris Drive, Unit 122
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September 28, 2020

James H McCarthy, MSc CFP
Senior Associate Biologist, Ecosystem NL Group Lead
Wood PLC
St. John's, NL

RE: AGAT Laboratories' Methylmercury Reference Method and QA/QC Procedures.

Jim

As requested, we are sending the requested information regarding our Methylmercury analysis method and QA/QC procedures.

The reference method used for the methyl mercury analysis is: ***USEPA Method 1630, Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap, and CVAFS***. This method has been assessed and accredited by CALA; this includes a site visit and review of the method as performed in the lab as well as successful performance in a proficiency testing program. Our standard QA/QC procedures also include the following:

1. **Method Blanks:** Method blanks are blanks brought through the entire sample preparation/digestion/distillation process. We currently prepare a filter method blank with each batch of samples received from Wood for the Muskrat Falls project to ensure our filtration and preservation procedure provides little to no contamination.

    a. Currently our St. John's laboratory is doing the initial preparation for the methyl mercury analysis to ensure the 48 hour hold-time is met. A clean room specifically for these samples was designed and a filter method blank with each batch of samples is prepared.

2. **Calibration/Reagent Blanks:** These blanks ensure the lab grade water and reagents used in the analysis are not contributing to the measured result. Elevated calibration/reagent blanks would trigger a maintenance/recalibration and/or preparation of fresh reagent.

3. **Duplicate Analysis:** One sample per analytical batch is prepared and analyzed in duplicate.

4. **Matrix Spike:** One sample in every 10 samples is prepared with a spiking solution. For each batch, one sample spike/sample spike duplicate must be analyzed.

5. **On-Going Precision and Recovery (OPR) QC Standard:** A second source standard is prepared to verify the calibration and to determine the efficiency of the digestion/distillation process.

6. **Laboratory Control Sample:** A water sample of known concentration is carried through the analytical process. The results of analysis of these samples must meet established acceptance criteria. Analytical results that are outside of these criteria trigger an investigation to identify corrective actions as well as reanalysis of the analytical batch.

7. **Batch Set-up:** With the current instrument set-up, each sample aliquot is analyzed twice. This provides additional confidence in the analytical data reported and allows the lab to identify any anomalous results prior to data reporting.

8. **Comparison to Total and Dissolved Hg:** Results for Total MeHg and Dissolved MeHg for each location are compared to ensure Total MeHg is greater than the dissolved aliquot. If this is not the case, the data is reviewed, and sample analysis is repeated on a fresh aliquot.

9. **Review of Data with Historical Results:** AGAT has been providing this analysis either through a subcontract lab or internally for several years. Analytical results for current samples are compared with historically reported values. Any data points that are not in line with historic data will be reviewed and/or reanalyzed for confirmation.

These items are quite standard to all laboratory procedures. Based on CCME protocols and reference methods there is acceptance criteria for each QA/QC component. Any values outside the acceptance criteria will trigger data review and /or re analysis.

In addition, analysis is carried out by a Senior Laboratory Analyst with 15 years' experience working in the lab at various levels. This analyst is a member of the NunatuKavut community and has an in-depth knowledge of the sampling program used for the Muskrat Falls project, as well as a strong knowledge of the geographical region of which provides the analyst of an understanding of the sites, and significance of each result. Lastly, the senior laboratory analyst has been monitoring the trends (seasonal) and staying well informed of events that may potentially cause anomalies (flooding) in the data.

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I trust this of assistance. Please contact me if you have any additional questions.

Best Regards

James MacDonald
Technical Services Manager – Atlantic Canada