

Determination of Sodium and Chloride Pore Water Concentrations of Peat/Muskeg – Prescriptive (Proposed)

Parameter	Sodium Pore Water Concentration Chloride Pore Water Concentration		
Analytical Method	Sodium and Chloride pore water concentration determination for peat or muskeg waters by direct pore water analysis, fixed ratio leach (2:1 as-received), or saturated paste extraction.		
Introduction	<p>This method measures sodium and chloride pore water concentrations (as mg/L) in peat ecosystems or muskegs and has been proposed for use by the Oil and Gas Commission (OGC) (refer to Reference 2, Alternative Salt Guidelines for British Columbia Boreal Peatland Releases: Scientific Derivation Document). This draft version is not approved for use for numerical delineation and site investigation purposes at contaminated sites regulated under the BC Contaminated Sites Regulation. However, it may be considered for use in detailed risk assessment at contaminated sites regulated under the BC Contaminated Sites Regulation; consult the director for advice.</p> <p>Because the moisture content of peat is normally very high (typically > 80-90%), sodium and chloride ions in peat generally exist almost exclusively in dissolved form within the moisture of the material (i.e. pore water or soil solution).</p>		
Method Summary	<p>Sodium and Chloride ions are either measured directly from pore water samples (first preference where practical) or are extracted from peat using either a fixed ratio leach (2:1 as-received) or using the saturated paste extraction procedure. For the fixed ratio leach or saturated paste procedures, pore water concentrations are then calculated based on the moisture content of the material.</p> <p>This method is prescriptive. It must be followed exactly as described. Where minor deviations are permitted, this is indicated in the text.</p>		
MDL(s) and EMS Analyte Code(s)	<u>Analyte</u>	<u>Approx. MDL</u>	<u>EMS Analyte Code</u>
	Sodium (pore water)	5 mg/L	-
	Chloride (pore water)	10 mg/L	-
EMS Method Code	<p>To be determined.</p> <p>Refer to EMS Parameter Dictionary on the ministry website for all current EMS codes.</p>		
Matrix	Peat / Muskeg		
Interferences and Precautions	Refer to supporting instrumental test methods for sodium and chloride. The interferences encountered will differ depending on the analytical technique used.		
Sample Handling and Preservation	Use a clean sealed polyethylene or glass container for sample collection. Plastic bags are suitable. No preservation is applicable.		
Stability	<p>Holding Time: 14 days (moisture measurements must be representative of field conditions).</p> <p>Storage: Store samples refrigerated at ≤ 6°C to protect against evaporation of moisture.</p>		
Option 1: Direct Pore Water Analysis	<p>Where practical, direct pore water analysis is the preferred procedure for this method. Direct pore water analysis should be conducted if representative pore water can be directly isolated and collected at the sampling location, or if pore water can be physically separated and isolated from a representative wet, as-received peat sample at the laboratory, either by centrifugation or (if available) by use of an API Filter Press or hydraulic soil press / sediment squeezer (as described in ASTM D4542-15).</p> <p>Centrifuge and/or filter a portion of the isolated pore water prior to direct analysis for sodium and/or chloride, following an approved BC ENV analytical method.</p>		

**Option 2:
Fixed Ratio
Extraction,
2:1 As-Received**

If direct pore water isolation and analysis is not practical, fixed-ratio extraction is the preferred extraction procedure for this method, due to its simplicity and reduced potential for error or contamination by additional handling steps.

1. Peat samples are processed as-received, without prior drying, grinding, sieving, or other pre-treatments. Samples must be representative of field conditions; if samples have dried (evaporated) since sampling, test results (as mg/L pore water concentrations) will be biased high. Homogenize sample well prior to sub-sampling.
2. Take a representative sub-sample (nominal 15 ± 2 grams, 13 grams minimum wet weight). Conditions described below assume a nominal 15 grams sample size. Larger samples may be used if method conditions described are scaled-up.
3. Accurately weigh a 50 mL (or larger) plastic or glass centrifuge tube to the nearest 0.01 g. Select a centrifuge tube type that can withstand a temperature of 105°C.
4. Transfer 15 ± 2 grams wet weight of the as-received peat/muskeg sample to the pre-weighed centrifuge tube. Accurately weigh the centrifuge tube plus wet peat sample to the nearest 0.01 g. This is *Weight Centrifuge Tube + Wet Sample* below.
5. For typical peat samples, accurately add 30 mL deionized water. For peat samples with lower than usual moisture contents, additional water may be added if necessary to generate enough free water for analysis. Accurately measure and record the total amount of deionized water added (measure to at least the nearest 0.5 mL). The amount of *Deionized Water Added* may be measured by volume or by weight difference.
6. Vigorously mix sample and deionized water for at least 30 minutes using a wrist-action shaker, shaker table, or tumbler device.
7. Centrifuge the sample tube at approximately 3,000 rpm for ~10-20 minutes to separate and isolate as much as possible of the aqueous extract from the solids fraction.
8. Transfer the majority of the separated aqueous phase from the extraction tube to a new container by decanting or by syringe or pipette. Avoid transferring any significant portion of the solids, so that an accurate dry weight of solids extracted can be determined from the extraction tube.
9. Filter a portion of the overlying water prior to sodium and/or chloride analysis following an approved BC ENV analytical method.
10. Direct Moisture Determination. Directly determine the moisture content of the sub-sample that was extracted by drying the sample tube containing the extracted wet solids at 105°C (dry overnight or ~16 hours, and verify that the procedure and oven used achieves dryness to constant weight; this method requires a highly accurate moisture measurement to deliver accurate pore water concentrations).
11. Data Calculations and Reporting – At minimum, calculate and report the following:
 - i) Sub-sample Moisture (mL) = Sub-sample Moisture (g) =
 $\{\text{Wt Centrifuge Tube + Wet Sample (g)}\} - \{\text{Wt Centrifuge Tube + Dry Sample (g)}\}$
 - ii) % Moisture of As-Received Sample =
 $100\% \times \text{Sub-sample Moisture (g)} / \{\{\text{Wt Cent. Tube + Wet Sample (g)}\} - \{\text{Wt Cent. Tube (g)}\}\}$
 - iii) Leachable Na &/or Cl concentration as mg/kg (dry weight of sample):
 $[\text{Na or Cl}] \text{ (mg/kg)} = [\text{Leachate Conc. (mg/L)}] \times [\text{Total Leachate Vol (L)}] / \text{Sample Dry Wt (kg)}$

Where:
Sample Dry Wt (kg) = $[\{\text{Wt Centrifuge Tube + Dry Sample (g)}\} - \{\text{Wt Centrifuge Tube (g)}\}] \div 1000$
Total Leachate Vol (mL) = $\{\text{Deionized Water Added (mL)}\} + \{\text{Sub-sample Moisture (mL)}\}$
Total Leachate Vol (L) = $\text{Total Leachate Vol (mL)} \div 1000$
 - iv) Leachable Na &/or Cl concentration expressed as mg/L of pore water:
 $[\text{Na or Cl}] \text{ (mg/L in pore water)} =$
 $[\text{Leachate Concentration (mg/L)}] \times [\text{Total Leachate Vol (mL)}] / [\text{Sub-sample Moisture (mL)}]$
 - v) Report test results as being produced from the Fixed Ratio Leach Method (2:1 as-received).

**Example of Fixed Ratio Leachate Method
(2:1 As-Received)**



Un-centrifuged Centrifuged

15g wet peat
+ 30 mL water

**Option 3:
Saturated Paste
Extraction**

Although either direct pore water analysis or fixed-ratio extraction are preferred, the saturated paste extraction procedure may also be used to measure pore water sodium and chloride concentrations, and has the advantage of also being applicable for use with regulatory sodium and chloride CSR determinations which require the saturated paste method. Furthermore, by this technique, pore water concentrations can be calculated from historical test results if as-received moisture measurements are available.

1. Follow the BC Lab Manual procedure for Saturated Paste Extractions, with the exception that for this method, peat samples must be processed as-received, without prior drying, grinding, sieving, or other pre-treatments. Samples must be representative of field conditions; if samples have dried (evaporated) since sampling, test results (as mg/L pore water concentrations) will be biased high. Homogenize sample well prior to sub-sampling for moisture and sodium/chloride leach procedure.
2. Data Calculations and Reporting – At minimum, calculate and report the following:
 - i) % Moisture of as-received sample.
 - ii) Leachable Na &/or Cl concentration as mg/kg (Dry Wt of sample).

$$[\text{Na or Cl}] \text{ (mg/kg)} = [\text{Na or Cl}] \text{ (mg/L of Sat Paste leachate)} \times [\text{Sat\%} / 100]$$
 - iii) Leachable Na &/or Cl concentration expressed as mg/L of pore water:

$$[\text{Na or Cl}] \text{ (mg/L pore water)} =$$

$$[\text{Sat Paste Leachate (mg/L)}] \times [\text{Sat\%} / 100] \times [(100 - \% \text{Moisture}) / \% \text{Moisture}]$$

Where:
 Sat% = Saturation % = [Wt water (g) at saturation / Dry Wt of peat] * 100%
 %Moisture is moisture content of as-received sample.
 - iv) Report test results as being produced from the Saturated Paste Extraction Method (as-received).

Example Sat Paste Calculations for Peat:

Saturation % = 1200

Peat Sample Moisture (as received) = 85%

[Chloride] in Sat Paste Extract = 100 mg/L

[Chloride] in peat (as mg/dry kg) = 100 mg/L x 1,200/100 = 1,200 mg/kg

Pore Water [Chloride] = 100 mg/L * (1,200/100) x [(100-85)/85] = 212 mg/L

Quality Control

Summary of QC Requirements		
QC Component	Minimum Frequency	Minimum Data Quality Objectives
Method Blank (MB)	One per batch (max 20 samples)	Less than reported DL
Lab Control Sample (LCS), Post Extraction	One per batch (max 20 samples)	80 – 120%
Lab Duplicates (DUP)	One per batch (max 20 samples)	30% RPD [or within 2x reported DL for low level results]
Matrix Spike (MS), Post Extraction	One per batch (max 20 samples)	70 – 130%
If DQOs are not met, repeat testing or report qualified test results.		

References

1. Saturated Paste Extraction for Soils, BC Lab Manual.
2. Final Report, Alternative Salt Guidelines for British Columbia Boreal Peatland Releases: Scientific Derivation Document, prepared for Steering Committee – BC OGRIS Salinity in Wetlands Research Project, Dr. Doug Bright, Hemmera Envirochem Inc., file 1692-002, January 2018.

Revision History

Dec 17, 2020 First version for publication in BC Lab Manual.