Log of Revisions

Date	Rev	Description	Author
05-Nov-02	1.0	-New Document following common format and numbering system for two-site laboratory system	DEP
15-Dec-04	1.1	Renamed method Biochemical Oxygen Demand Section 1.2 and 1.3 added for application and scope of method in CBOD and BOD determination Section 3.2- Added Section 4.1- Added temperature range Section 4.3- Added samples types requiring chlorine determination Section 5- Added relevant SOPs Section 6- Added use of reagent preparation logs Section 6- Added shelf life requirements for reagents Section 6.1- Added relevant SOP Section 6.8.2- Added Section 6.9- Removed Section 6.10 & 6.11- Renumbered as 6.9 & 6.10 Section 6.10.1- Added storage temperature conditions Section 7.1- Added time for aeration Section 7.2- Clarified wording for use of bottle stopper Section 7.2.1.7- Added note Section 7.2.1.11- Added data recording requirement Section 7.3- Clarified pH adjustment requirements Section 7.11- Added matrix spike preparation Section 7.11 to 7.13- Renumbered as 7.12 to 7.14 Section 7.15- Added document procedures Section 8.3- Added spike calculation Section 8.3- Renumbered as 8.4 Section 8.5- Added LIMS data entry requirements Section 9- Updated method validation section Section 10- Added reference to relevant in-house SOPs	DEP
10-Sep-06	1.2	Update Reference to 21st Ed. of Standard Methods for the Examination of Water and Wastewater Update Method Validation Section (9.0 to 9.6)	GC/DEP
30-Oct-2006	1.3	Section 6.9- Added storage conditions for seeding material. Section 5 Note- Added use of dishwasher and clarified rinsing to remove copper. Other minor grammar/wording changes.	DEP
21-Oct-2008	1.4	Section 6 – Added storage conditions to reagents where required.	GC/DEP

Document Review

This document was last reviewed and authorized by:

Laboratory Branch Manager _____ Date _____

Total Suspended Solids

1.0 Scope and Application

1.1 The method is applicable to surface, ground, and waste waters in the range of 1 - 20,000 mg/L. The method detection limit is 3 mg/L.

2.0 Principle and Theory

2.1 Principles

2.1.1 Total suspended solids (TSS) is the term applied to the material retained by a glass fibre filter and dried to a constant weight in an oven at a defined temperature. Methods that utilize different pore size filter paper or alternative drying temperatures will not provide comparable analytical results.

2.2 Interferences

2.2.1 Large non-homogenous particles should be excluded from the test aliquot if it is determined that their inclusion is not desired in the final results. Floating oil and grease, if present, should be included in the sample and dispersed by a blender before withdrawing a sub-sample for filtration.

2.2.2 Filter clogging, from excessive solids captured in the filter, may prolong filtration time and produce high results. To overcome this, use a smaller sample volume for filtration, or use a larger size filter.

2.2.3 The type of filtration apparatus, filter material, pre-washing, post-washing and drying temperature are specified to minimize affects due to these variables.

2.2.4 Samples with high dissolved solids may cause positive interferences. These effects can be minimized with adequate washing to remove dissolved solids which may get trapped in the filter pores.

3.0 Safety

3.1 Wear gloves and safety glasses. Check individual MSDS prior to handling any chemicals.

3.2 **Refer to Caduceon Safety Manual for general safety procedures.**

4.0 Sample Requirements

4.1 Sample Collection

4.1.1 For water samples containing little or no visible suspended solids, 200mL to 500mL sample volume may be required for analysis. Samples containing many suspended solids require less volume for analysis. Samples should be collected in a plastic or glass bottle and transported to the laboratory as soon as possible for analysis. If the test is not performed immediately, the sample must be stored at 4 ± 3°C.

4.1.2 There must be no preservation added to samples collected for TSS analysis.

4.2 Sample Holding Time

4.2.1 Analysis should be performed within 7 days to prevent any change in the condition of the sample submitted.

5.0 Equipment

5.1 Whatman 934AH glass microfibre filter (or equivalent), 47 mm diameter

5.2 Filtration apparatus suitable for filters in section 5.1

5.3 1L vacuum flask

5.4 Aluminium dishes to hold filters

5.5 Forceps to handle filters

5.6 Oven set at 105 ± 5°C (temperature accuracy verified as per Caduceon SOP-10)

5.7 Desiccator

5.8 Graduated cylinders, various sizes (dispensing volumes verified as per Caduceon SOP-08)

5.9 Wide mouth graduated pipettes, various sizes (dispensing volumes verified as per Caduceon SOP-08).

5.10 Reusable 120 mL Plastic (HDPE or PTFE) bottles for QC preparation. (NOTE: QC bottles should be thoroughly rinsed to ensure removal of all suspended solids prior to re-use)

5.11 Analytical balance, 0.0001g capacity (balance calibration verified as per Caduceon SOP-09)

5.12 Clean all glassware/pipettes and filtration apparatus with hot water followed by a final rinse with reagent grade water. If necessary, wash labware with soap and water then rinse thoroughly with reagent water.

6.0 Reagents

6.1 Reagent grade water (DW) (to meet specifications as per Caduceon SOP-04)

6.2 In-house Quality Control Standard Solution, 100 mg TSS/L (QCTSS-01): Weigh approximately 0.0100g (±0.0020g) of the in-house Q.C. standard Kieselgur into a weigh dish. Quantitatively transfer the solid standard into a 120mL plastic bottle containing about 50mL DW. Mix well and dilute to approximately 100 mL. Keep stored in jar. Prepare as needed. Note that the entire contents of the jar are analysed in one fraction. (NOTE: The percent recovery of the measured mass must fall within the QC limits)

7.0 Test Procedure

7.1 Determination of Filter Weight

7.1.1 Place the filter in a uniquely numbered aluminium weigh dish. Dry it in an oven for at least 1 hour at 105 ± 5 °C. Remove the aluminium dish and place in the desiccator for at least 1 hour to allow the filter to cool to room temperature.

7.1.2 Zero the analytical balance according to the manufacturer's operating manual. Place the filter on the balance pan and record the weight of the filter on the analyst work sheet.

7.2 Sample Analytical Procedure

7.2.1 Allow samples to reach room temperature (minimum 1 hour) before proceeding with analysis.

7.2.2 After determining the filter weight in section 7.1, place the filter on the filtration apparatus. Quickly measure an appropriate aliquot of well-mixed homogenized sample with a graduated cylinder or wide mouth graduated pipette. The sample volume analyzed should be sufficiently small to prevent the filter from clogging as outlined in section 2.2.2. If more than 10 minutes is required to complete filtration increase the filter size or decrease the sample volume. The corrective action should not produce less than 0.0025g of dry residue on the filter.

7.2.3 Filter sample under vacuum. Rinse graduated cylinder or pipette with reagent grade water into the filter unit to ensure complete transfer of the sample. Rinse the sides of the filtration unit during the filtering process to prevent suspended solids from adhering. Continue vacuum suction to remove all traces of free water. Carefully remove the filter from the filtration unit, and replace it in the uniquely numbered aluminium dish. Record the dish # on the analyst work sheet.

7.2.4 Dry the filter in an oven for at least 1 hour at 105 ± 5 °C. Be sure to use the top rack in order to reduce the chance of contamination. Remove the aluminium dish and place in the desiccator for at least 1 hour to allow the filter to cool to room temperature.

7.2.5 Zero the analytical balance according to the manufacturer's operating manual. Place the filter on the balance pan and record the weight of the filter and suspended solids on the analyst work sheet.

7.3 Analytical Run Structure

7.3.1 Each daily run must be structured to include a standard and reagent water blank at the beginning and end of each run. A duplicate and QC are analyzed after every 10 samples. A blank is analyzed after every 20 samples.

7.3.2 At the end of the analytical run, each QC sample (i.e. QCTSS-01, blank and duplicate) must be compared to its acceptable limits found in the QC log. If a QC result falls outside its acceptable range, corrective action must be performed as follows.

7.3.2.1 If more than one QC standard (QCTSS-01) falls outside its limits, re-analyse another aliquot of the solution and all affected samples, analyzed just prior to the failed QC.

7.3.2.1.1 If the result of the new QC solution is acceptable, the new analytical results can be reported. If acceptable results are still not achieved for the QC, equipment may need to be serviced. Consult with the laboratory supervisor for direction.

7.3.2.1.2 No further analysis may be conducted until the problem has been successfully corrected.

7.3.2.1.3 Sample results may only be reported if qualified with a comment indicating failed QC.

7.3.2.2 If only one QCTSS-01 has a low recovery within the analytical batch, results may be reported with a qualifying statement.

7.3.2.3 If a duplicate falls outside its limits, re-analyze another aliquot of the solution. If this result is still unacceptable, select a different sample for duplicate analysis and analyze it accordingly.

7.3.2.2.1 If the results of the new solution analysis are acceptable continue with the analytical run.

7.3.2.2.2 If acceptable results are still not achieved, and the QC standard, QCTSS-01, is acceptable, the sample results may be reported with

a qualifying statement.

7.3.2.2.3 Report all initial sample results (each result for the duplicate pair) for failed duplicate samples, and qualify the data to indicate that the results are suspect due to possible matrix interferences.

7.3.2.3 Record the non-conformance and corrective actions performed on the method non-conformity log (as per SOP-43).

7.3.2.4 All samples analyzed just prior to non-conforming QC standards (section 7.3.2.1), must be re-analyzed. If there is insufficient sample for re-analysis or the sample has passed its holding time, report the results with a qualifying statement to indicate failed QC during the analytical run.

7.4 Documentation Procedures

7.4.1 Document all required information on the TSS Analysis worksheet when analyzing samples and QC solutions. Transfer data to the equivalent excel spreadsheet to calculate sample concentrations and percent recovery of QCTSS-01. (K:WP51/Lab/Calibration curves/TSS,VSS_[most recent month]_[current year]D.xls)

7.4.2 Document all required information in TSS Standard Preparation log when preparing QC Standards.

7.4.3 Document all maintenance, comments and any changes in the equipment log book.

7.4.4 QC data must be entered into the QC log data file by the analyst.

7.4.5 The QC log data file is found on the Ottawa server: WP51\LAB\QC LOGS\Inorganic Chemistry\TSS.xls.

7.4.6 Open this file and enter the data in the appropriate fields; Date, Expected Concentration for QCTSS-01, Found Concentration for QCTSS-01, Duplicates and Blanks.

7.4.7 The excel program will calculate the % recovery of the QCTSS-01; and the % difference of the duplicate samples and the average concentration of the blanks.

7.4.8 The excel program files will be maintained by the QA officer.

7.4.9 Control charts will be monitored for trend analysis as per SOP-07 Control Charting.

8.0 Calculations & Reporting

8.1

$$T = \frac{(A - B) \times 1000000}{V}$$

Where T = concentration of total suspended solids (mg TSS/L)
A = weight of the filter and suspended solids, section 7.2.4 (g)
B = weight of the filter, section 7.1.3 (g)
V = volume of sample analyzed (mL)

Note: The data collected is entered into an excel spreadsheet and the concentration of TSS for samples is calculated by the software. This spreadsheet is saved to the Caduceon server, and also printed out and stamped as valid, to be kept with the analyst worksheet.

8.2 Blank Correction

8.2.1 All reagent blanks that are less than the detection limit are automatically accepted. No correction is needed for the sample concentrations.

8.2.2 When the reagent blank has a calculated concentration greater than the method detection limit, use the result of the blank to correct for possible contamination or method non-conformance. The correction equation follows.

8.2.2.1 $A = B - C$

Where A = corrected TSS concentration
B = uncorrected TSS concentration
C = reagent blank TSS concentration

Be sure that the values for A, B, and C are expressed in the proper units for each type of sample analyzed. The Excel spreadsheet will automatically perform this calculation for all samples. If C is a negative value, the spreadsheet corrects the blank to 0, and corrects all samples accordingly (A=B+C).

8.3 Refer to Caduceon SOP-39 LIMS Training for details of the recording of results in the Laboratory Information Management System (LIMS).

8.3.1 The final calculated sample result is entered in the Laboratory Run module of the LIMS. This result will be displayed on the final Certificate of Analysis generated from the LIMS.

9.0 Method Validation

9.1 Method Validation Data

	Data Points	Reporting MDL (Calculated)
MDL (mg/L)	9	3(2.1)
Precision (%) *	1532	4.9
Accuracy (%) *	1532	98.7

* Based on on-going QCTSS-01 results (01-Sep-06)

9.2 Quality Control Standards

Sample ID	Number of Data Points	Expected (% recovery)	Mean (% recovery)	Average Bias (% recovery)	Standard Deviation	UCL (%)	LCL (%)
QCTSS-01	1532	100	98.7	1.3	2.8	107	90

9.3 Duplicates

Analytical Range (mg/L)	Number of Data Sets	Acceptable Limits-RPD %
≤ 30	573	202
> 30	631	32

9.4 Method Uncertainty

9.4.1 The expanded uncertainty is determined as per SOP-23 . The data accumulated in the QC log is used to calculate the expanded uncertainty detailed in the following table.

62/67

Typical Concentration (mg/L)	Combined Uncertainty (mg/L)	Expanded Uncertainty * (Percent)
10	2.7	42
250	22.0	17
500	42.7	17

* Uncertainty at 95% confidence interval (coverage factor k=2)

9.5 Method Performance

9.5.1 The Method Performance is monitored by the results of PT sample analysis. Each round's reported results and consensus values are entered in the Method QC log. The % Recovery is calculated on a minimum of 8 sample results. The maximum allowable deviation of a PT result is based on 3sd.

Mean % Recovery	101.1
Standard Deviation (sd)	4.9
3sd (99% confidence level)	14.7
Number of Data Points (n)	28

9.6 Based on the method validation data supplied above, this method has been deemed as fit for its intended use (as stated in Section 1 of this document).

10.0 References

10.1 Standard Methods for the Examination of Water and Wastewater, 21st Ed., 2540D, 2005

10.2 Methods for Chemical Analysis of Water and Wastes, USEPA, p 160.2-1; 1983

10.3 Protocol for the Sampling and Analysis of Industrial/Municipal Wastewater- MISA, p 103-104; 1994

10.4 Caduceon SOP-04, Preparation of De-ionized Water

10.5 Caduceon SOP-07, Control Charting

10.6 Caduceon SOP-08, Verifying Delivery Volumes

10.7 Caduceon SOP-09, Balance Calibration and Verification

10.8 Caduceon SOP-10, Thermometer Calibration and Verification

10.9 Caduceon SOP-23, Determination of Uncertainty in Measurement

10.10 Caduceon SOP-39, LIMS Training

10.11 Caduceon SOP-43, Non-conformity Logs

10.12 Caduceon Safety Manual

Log of Revisions

Date	Rev	Description	Author
05-Nov-02	1.0	-New Document following common format and numbering system for two-site laboratory system	DEP
18-Dec-04	1.1	Section 4.1.1- Added temperature range Section 4.1.2- Added statement about no preservation Section 5- Added references to SOPs Section 5.12- Updated cleaning instructions Section 6.1- Added reference to SOP Section 7.1 & 7.2- Added data recording and other minor clarifications Section 7.3- Added criteria for data acceptance and corrective actions for non-conformities Section 7.4- Added Document procedures Section 8.3- Added LIMS data entry requirements Section 9- Method validation revised to include limits, and uncertainty Section 10- Reference list updated and expanded to include relevant SOPs	DEP
31-Oct-06	1.2	Section 9.0 to 9.5 Updated Method validation data with results available on 01-Sep-06.	GC
21-Jul-2008	1.3	Section 3.2 - Reference to Safety Manual Sections 5.9, 10, 12- Additions to equipment list Section 6.2 – Addition of QC Standards ID, mass range and QC acceptance limts based on actual mass measured. Section 7.2.1 – Added statement about letting samples get to room temperature Section 7.3.2.2 – Added need for qualifying statement on results when 1 QC within analytical batch has low recoveries. Section 7.4.1 – Location where raw data is transferred to for final calculations added. Section 8.1 – Added statements on saving, printing & validating spreadsheet used for TSS calculations. Section 8.2.2.1 – Added statement on the handling of –ve reagent blank values in TSS calculations. Section 10.1 – Updated SM reference to 21st Ed.	GC/TH

Document Review

This document was last reviewed and authorized by:

Laboratory Branch Manager _____ Date _____

APPENDIX-G

INAC INSPECTOR'S INSPECTION REPORT IN 2009

Indian and Northern Affairs Canada | Affaires Indiennes et du Nord Canada

WATER USE INSPECTION REPORT

Date: 18/8/08	**Licensee Rep.** (Name/Title): David Arreak – Muni Works Foreman
Licensee: Hamlet of Clyde River	**Licence No: 3BM-CLY0308**

WATER SUPPLY

Source(s): Water Source Lake	**Quantity used: Unknown** – No Records for
Owner:/Operator: Hamlet of Clyde River	No Chlorine for treatment – Bleach added to trucks

Indicate: **A** - Acceptable **U** - Unacceptable **NA** - Not Applicable **NI** - Not Inspected

Intake Facilities: A	**Storage Structure:** A	**Treatment Systems:** U	**Chemical Storage:** NI
Flow Meas. Device: U	**Conveyance Lines:** A	**Pumping Stations:** A	**Screen : NI**

Comments: Chlorination system has not been operable for a number of months. Chlorine not shipped because hazardous material. Records were not available for review.

WASTE DISPOSAL

Sewage: Sewage Treatment System (Prim./Sec/Ter.)**: Lagoon system**

Natural Water Body: No	**Continuous Discharge** (land or water)**: Land/wetland**	
Seasonal Discharge: NA	**Wetlands Treatment:** Y	**Trench:** None

Solid Waste: Some segregation. **Owner/Operator:** Hamlet of Clyde River

Landfill: A- some segregation	**Burn & Landfill:** A	**Other:** Waste oil segregated

Indicate: **A** - Acceptable **U** - Unacceptable **NA** - Not Applicable **NI** - Not Inspected

Discharge Quality: U	**Decant Structure:** NI	**Erosion:** A
Discharge Meas. Device: NA	**Dyke Inspection:** NA	**Seepages:** NA
Dams, Dykes: NI	**Freeboard: NA**	**Spills:** U
Construction: NI	**O&M Plan:** U	**A&R Plan:** U
Periods of Discharge: Cont.	**Effluent Discharge Rate:** Unknown	

Comments: Lagoon requires work to ensure another failure does not occur. Overflow drain has been installed which is allowing raw sewage to discharge directly to environment. Hazardous wastes area has many spills and overturned drums-requires immediate remedial work. Verbal Direction to address both provided on site.

FUEL STORAGE:

Waste Oil Storage: NI **Owner/Operator:** Nunavut Power Corp.

Indicate: **A** - Acceptable **U** - Unacceptable **NA** - Not Applicable **NI** - Not Inspected

Berms & Liners: NI	**Water within Berms:** NI	**Evidence of Leaks:** NI
Drainage Pipes: NI	**Pump Station & Catchments Berm:** NI	
Pipeline Condition: NI	**Condition of Tanks: NI**	

SURVEILLANCE NETWORK PROGRAM (SNP)

Samples Collected: 0	**Owner /Operator:** No samples from Municipality have been submitted	
Samples Collected: 2	**INAC:** Potable- Lake, Effluent discharge,	
Signs Posted	**SNP:** None	**Warning: Some signage missing**
Records & Reporting: No records of water usage, waste discharge.		
Geotechnical Inspection: N/A		

Non-Compliance of Act or Licence: The Hamlet of Clyde River's Water License expires in September of this year. The Municipality is advised to contact the Nunavut Water Board as soon as possible to process an application and avoid the possibility of operating without a license. The Community has not been collecting samples nor filing the required paperwork. Immediate work is required at the Sewage Lagoon and to address spills and leaking drums at Hazardous wastes area.

Hamlet staff also directed to ensure proper Chlorination and treatment of Potable water is reinstituted without delay.

A.Keim
Inspector's Name

Sent by E-mail
Inspector's Signature