APPENDIX-L

QA/QC PLAN OF WASTEWATER QUALITY MONITORING

QA/QC of Wastewater Quality Monitoring of the Hamlet of Cape Dorset, Baffin Region, Nunavut.

Hamlet of Cape Dorset is using 3 cells lagoons for treating about 46,033.80 cubic meters community wastewater annually. These are not Engineered facilities. Therefore as built drawings and O&M manual are not available. The new Sewage Lagoon at P Lake was built in 2007 and licensed in 2008 but not commissioned yet. The as built drawings and O&M manual are up to date for this facility. The Monitoring points and compliance points are well established. The hamlet is planning monitoring the wastewater quality once this new facility is commissioned. They will be using the services of the following accredited lab in Ottawa.

Caduceon Environmental Laboratories
2378 Holly Lane,
Ottawa, ON, Canada K1V 7P1
Tel: (613) 526-0123
Fax: (613) 526-1244
Website: www.caduceonlabs.com

It is noted that all the communities of Baffin Region are using the facilities of this lab.

Normally Hamlet will collect sample bottles with the instruction of sampling, preservation and shipping guidelines from the lab about a month before the date of decanting the Lagoon. In this case the hamlet staffs will collect samples alone or together with Regional Engineer or Municipal Technical Officer or INAC Inspector depending on their availability. The lab QA/QC Plan and Procedure is attached.

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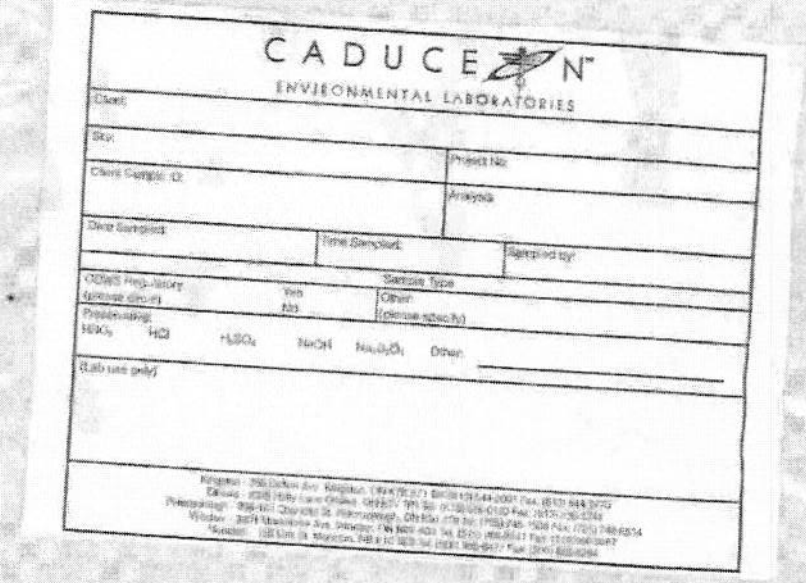

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Client:

Site:

Project No:

Client Sample ID:

Analysis

Date Sampled:

Time Sampled:

Sampled by:

ODWS Regulatory (please circle)

Yes

No

Sample Type

Other: (please specify)

Preservative:

HNO_3 HCl H_2SO_4 NaOH $Na_2S_2O_3$ Other:

(Lab use only)

Kingston -
Ottawa -
Peterborough -
Windsor -
Sudbury -

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Testing Requirements

- ☐ O.Reg 153/04 (1___ 2___ 3___ 4___ 5___ 6___)
- ☐ MISA Guidelines
- ☐ Provincial Water Quality Objectives
- ☐ Sewer Use By-Law: _____
- ☐ Other: _____
- ☐ ODWS (Non Regulatory)
- ☐ O.Reg 558 Leachate Analysis
- Disposal Site: _____
- ☐ Landfill Monitoring

Turnaround Time Requested

Rush 24 Hr	☐	100% Surcharge	
Rush 48 Hr	☐	50% Surcharge	
Rush 72 Hr	☐	25% Surcharge	
5-7 Day	☐	Standard	
Specific Date:		_____	

Organization:	Address and Invoicing Address (if different)	
Contact:		
Tel:		
Fax:	Quote No.:	Project Name:
Email:	P.O. No.:	Additional Info:

ANALYSES REQUESTED (Print Test in Boxes)

REPORT NUMBER:

* Sample Matrix Legend: WW=Waste Water SW=Surface Water GW=Groundwater LS=Liquid Sludge SS=Solid Sludge S=Soil Sed=Sediment PC=Paint Chips F=Filter

Lab No:	Sample Identification	Sample Matrix *	Date Collected (yy-mm-dd)	Time Collected	Indicate Test For Each Sample By Using A Check Mark In The Box Provided	Field pH	Field Temp.	# Bottles/ Sample	Field Filtered(Y/N)

Are any samples listed above intended for Human Consumption? ☐ Yes ☐ No (If yes, submit all drinking water samples on a drinking water Chain of Custody)

Sample Submission Information	Shipping Information		Reporting and Invoicing	LABORATORY USE ONLY
Sampled By (print):	Courier (Client account) ☐	Invoice for Shipping ☐	Report by Fax ☐	Received By (print): Signature:
Submitted By (print):	Courier (Caduceon account) ☐		Report by Email ☐	Date(yy-mm-dd) Received: Time Received:
Signature:	Drop Off ☐	# of Pieces	Invoice by Email ☐	Laboratory Prepared Bottles: ☐ Yes ☐ No
Date(yy-mm-dd): Time:	Caduceon (Pick-up) ☐		Invoice by Mail ☐	Sample Temperature °C: Labeled by:

Laboratory Locations/Shipping Addresses

Kingston Lab - 285 Dalton Ave., Kingston, ON K7K 6Z1, Tel: (613) 544-2001 Fax: (613) 544-2770 Email: contactkingston@caduceonlabs.com
Ottawa Lab - 2378 Holly Lane, Ottawa, ON K1V 7P1, Tel: (613) 526-0123 Fax: (613) 526-1244 Email: contactottawa@caduceonlabs.com
Peterborough Lab - #206-160 Charlotte St., Peterborough, ON K9J 2T8, Tel: (705) 748-1506 Fax: (705) 748-6514 Email: contactpeterborough@caduceonlabs.com
Windsor Lab - #5-3201 Marentette Ave., Windsor, ON N8X 4G3, Tel: (519) 966-9541 Fax: (519) 966-9567 Email: contactwindsor@caduceonlabs.com
Moncton Lab - 150 Lutz St., Moncton, NB E1C 5E9, Tel: (506) 855-6472 Fax: (506) 855-8294 Email: contactmoncton@caduceonlabs.com

Comments:

QUALITY ASSURANCE/QUALITY CONTROL SUMMARY

LABORATORY MISSION

The policy of CADUCEON is to provide the highest standards of analytical service to its clients.

Quality Objectives

- ✓ To ensure a Quality System that is documented, communicated, understood, implemented and incorporates adequate review, audit and internal quality control.
- ✓ To ensure areas of Continual Improvement are identified by all staff and management, and that management will develop and implement effective strategies to achieve this Continual Improvement.
- ✓ To ensure personnel are adequately supervised and are proficient to carry out assigned activities.
- ✓ To ensure test methods and related work instructions are validated and incorporate adequate quality control.
- ✓ To ensure all equipment, supplies and services are functioning properly and/or meet required specifications.
- ✓ To ensure facilities are adequate to carry out the testing activity.
- ✓ To ensure that test results are supported by a traceable system of measurement and accorded uncertainties appropriate to requirements.
- ✓ To ensure sample management that incorporates adequate procedures for the security, receipt, identification, checking, routing, storage and disposal of all samples.
- ✓ To ensure data management that incorporates adequate procedures for the security, recording, calculation, validation, authorization, transmittal, storage and disposal of all test data and related records.
- ✓ To ensure workload management that incorporates acceptable holding time, turnaround time and verification of resource availability prior to the acceptance of additional testing.
- ✓ To ensure that achieving these quality objectives will enable the management system to meet all client requirements.

QUALITY PROGRAM

This program, which applies to all sample submissions to the laboratory, has been designed to comply with Canadian federal and provincial regulatory agencies, the U.S. Environmental Protection Agency, and the New York State Department of Health data quality objectives.

The elements of Quality Control/Quality Assurance outlined in this report are consistently employed at CADUCEON and are described in detail in our Quality Assurance Manual. This document includes our QA policy and Standard Operating Procedures (SOPs) for analysis methods and staff training protocols. The quality assurance objectives are translated into specific requirements for individual analyses and are written into all SOPs. Prior to beginning work using a given method, employees are instructed to read both the QA objectives and the relevant SOPs. Accompanied with hands-on training under the supervision of a senior analyst, the successful transfer of information is completed. Annual audits on technologists and analysts are performed to confirm compliance with SOP specifications.

IN-HOUSE ELEMENTS OF QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

CADUCEON endeavours to provide clients with analytical data of the highest quality. We ensure quality by maintaining several layers of data approval whereby, at any point during the processing of samples, CADUCEON personnel have the authority to reject a set of data based upon results falling outside of their respective QA/QC limits.

A summary of the various steps (as a minimum) followed during routine analyses is presented below:

Element	Frequency	Control Limits
Sample Containers	Precleaned to EPA Specifications	non detect
Reagents	Prepared from ACS grade chemicals or better	
Travel & Field Blanks	Provided upon request	<MDL
Replicate Analysis	1 in 20 or 1 per batch	± 20%
Matrix Spikes	1 in 20 or 1 per batch	± 3 S.D.
Instrument Calibrations:		
Inorganic	Multipoint Daily	r^2>0.999
	Verified every 10 samples	± 10% of initial
Organic	Multipoint	RSD ± 20%
	Verified Daily	± 3 S.D.
Surrogate Standards	All organic samples	± 3 S.D.
Internal Standards	All volatile and semi-volatile organic samples	
Method Blanks	Minimum 1 in 20 or 1 per batch	<MDL
Standard Reference Material	1 per batch or monthly	SRM Limits
Control Charts	Maintain Daily	± 3 S.D.
Method Audits	Yearly	Adhere to method specific limits (SOPs)

In addition, all of the above QA/QC data are catalogued for ease of retrieval should the data need to be reviewed. All samples are retained for one month following the transmission of the final report to the client.

ACCREDITATION and PROFICIENCY

CADUCEON has been a member of the Canadian Association for Laboratory Accreditation (CALA) since 1988. Following an independent laboratory audit, the laboratory received full accreditation from CALA for specific parameters. Subsequently, CADUCEON was awarded an ISO Guide 17025 accreditation by the Standards Council of Canada. In addition, a list of our scopes of accreditation with CALA can be found at the following locations: http://www.cala.ca/index.html

INTER-LABORATORY STUDIES

As part of the accreditation/proficiency programs of CALA, the performance of CADUCEON is monitored through the analysis of unknown proficiency samples submitted by an external agency. In addition, CADUCEON regularly takes part in inter-laboratory studies organized by organisations such as the Canada Centre for Inland Waters (CCIW), the National Water Research Institute: Long Range Transport of Airborne Pollutants (LRTAP) and CALA.

REFERENCE METHODS

The analytical methods employed at CADUCEON are based on the industry recognized and approved reference publications listed below:

- APHA-AWWA-WPCF – "Standard Methods for the Examination of Water and Wastewater,"
- Ontario Ministry of the Environment, "Protocol of Accepted Drinking Water Testing Methods", March 17, 2008.
- Ontario Ministry of the Environment, Approved Analytical Methods, Laboratory Services Branch
- Ontario Ministry of the Environment, "Guidance on Sampling and Analytical Methods for use at Contaminated Sites in Ontario," April 01, 2009
- Ontario Ministry of the Environment, "Protocol for the Sampling and Analysis of Industrial/Municipal Wastewater," January 1999
- ASTM – American Society for Testing and Materials
- AOAC "Official Methods of Analysis"
- CCME – "Guidance Manual on Sampling, Analysis and Data Management for Contaminated Sites," December 1993
- Environment Canada
- US EPA 500, 600 and SW846 Series Methodologies
- Other recognized regulatory and industry sources

Sample Bottle Requirements

1.0 Scope

1.1 This standard operating procedure (SOP) provides instructions on the provision of sampling materials and the steps required for the documentation of bottle requests received from the client. A detailed summary of parameters, sample containers, volumes, preservatives and holding times can be found in Appendices A to C.

2.0 Purpose

2.1 This SOP will ensure that the client is provided with the appropriate bottles and preservatives for field sampling.

2.2 The necessary records will be kept as per instruction in this SOP.

3.0 Procedure

3.1 All containers supplied to clients will be pre-cleaned, be of the required material (i.e. glass/plastic) and volume, contain the appropriate preservative (note: the preservative used should be clearly indicated in the appropriate section on the label) and be labelled.

3.2 When a client requests sample containers and supplies, the information is recorded in the Bottle Request Log. The person receiving the bottle request is responsible for documenting the following information:

- Date of order
- Order received by
- Company Name and Address
- Contact Name
- Shipping Address if different from above
- Date Required
- Detailed Parameter List or Quotation Number if available
- Any special instructions/requests (i.e. additional supplies, travel/field blanks, duplicates, spikes, bottle seals etc.)

Once the bottle order has been completed the person completing the bottle order shall sign and date the bottle request. The request can then be filed in the Bottle Request Log.

3.3 All bottles will be provided to the client with the appropriate packaging to minimize receiving damaged bottles as a result of shipping and handling in the field and during transit to the lab.

3.4 The client shall be responsible for labelling the sample containers and completing the chain of custody record prior to submitting samples to the lab. (refer to SOP-01 and SOP-02).

3.5 A detailed summary of parameters, sample containers, volumes, preservatives and holding times can be found in Appendices A to C.

4.0 Sample Handling Practices Specifically for Drinking Water Samples

4.1 Drinking-water samples should not be filtered in the field or at the laboratory prior to analysis. As it is not expected that the consumer filters their water prior to drinking it, unfiltered samples will provide a more representative sample of what the consumer is drinking. Unfiltered samples for the measurement of organic compounds and microbiological parameters are very important because many organic compounds adsorb to the particulate present in a water sample and membrane filtering will remove bacteria from the sample. Filtering is not permitted in order to compensate for poor sampling technique or the use of inappropriate methods of analysis.

Appendix A: Individual Parameters for Water Analysis

Parameter	Sample Containers		Minimum Volume (mL)	Preservative	Storage Conditions	Holding Times		
	Size (mL)	Type				Caduceon	EPA/SM(Reg.)	MOE
GENERAL CHEMISTRY, PHYSICAL PROPERTIES								
Alkalinity	500	P	50	None	1	7d	14d/14d	7d
Ammonia (NH3)	125	P or G	50	pH <2 H_2SO_4/None	1	28d/3d	28d/28d	10d
BOD5/CBOD5	500	P	300	None	1	4d	48h/48h	4d
Bromide	500	P	50	None	1	28d	-	-
Chloride	500	P	50	None	1	28 d	28d/28d	30d
COD	125, 250	P or G	50	pH<2 H_2SO_4	1	28 d	28d/28d	30d
Colour	500	P	100	None	1	48h/7d	48h/48h	7d
Conductivity	500	P	100	None	1	4d	28d/28d	4d
Cyanide (free)	125	P	50	pH >12 NaOH	1, in dark	7d	-/14 d	7d(MISA)
Cyanide (total)	125	P	50	pH >12 NaOH	1	6 m	14d/14d	6 m
Fluoride	500	P	50	None	1	28d	28d/28d	30d
Hardness	250	P	100	pH<2 HNO_3	2	28d	6m/6m	28d
Hydrogen Sulphide (H2S)	125, 250	P or G	100	2N zinc acetate + pH>9 NaOH	1	7d	7d/7d	7d(MISA)
Mercury	250	P,G,AG	100	$K_2Cr_2O_7$ + HNO_3	2	7d	28d/-	14d, 7d(MISA)
Metals- except Mercury	250	P	100	pH<2 HNO_3	2	60d	6m/6m	60d
Nitrate (N)	500	P	50	None	1	7d	48h/48h	7d
Nitrite (N)	500	P	50	None	1	7d	48h/48h	7d
Nitrate-Nitrite (N)	500	P	50	None	1	7d	48h/48h	7d
Nitrogen (Total Kjeldahl)	125, 250	P or G	100	pH<2 H_2SO_4	1	28d	28d/-	-
Organic Carbon, Dissolved (DOC)	125	G or P	50	Field filter + pH <2 H_2SO_4 /None	1	28d/7d	-	-
Organic Carbon, Total (TOC)	125	G or P	50	pH<2 H_2SO_4	1	28d	28d/28d	-
Oil & Grease, Total, A/V/Mineral	1000	G	1000	HCl/None	1	28d/7d	28d/28d	7d(MISA)
pH	500	P	100	None	1	4d	Imm./Imm.	4d/asap(MISA)
Phenolics (4-aap) *	60,125, 250	AG	50	pH<2 H_2SO_4	1	28d	28d/28d	30d(MISA)
Phosphate, dissolved (P)	125	P	50	Filter, analyze ASAP/pH<2 H_2SO_4	1	48h/28d	48h/-	-
Phosphorus (total)	125, 250	P or G	100	pH<2 H_2SO_4	1	28d	28d/-	30d(MISA)
Solids (TS,TSS,TDS,VS,VSS)	500	P	500	None	1	7d	7d/2-7d	7d(MISA)-
Silica	125, 250	P	100	pH<2 HNO_3	2	28d	28d/-	-
Sulfate	500	P	50	None	1	28d	28d/28d	30d(MISA)
Turbidity	500	P	100	analyze ASAP	1	48h/7d	48h/48h	48h(MISA)

MICROBIOLOGICAL								
Coliforms, Total, Fecal, Escherichia	300, 250	SP	100 (per test)	None, $Na_2S_2O_3$(chlorinated)	1	48h	-/30h	48h/24h(MISA)
Background	300, 250	SP	100	None, $Na_2S_2O_3$(chlorinated)	1	48h	-/30h	48h
Heterotrophic Plate Count	300, 250	SP	50	None, $Na_2S_2O_3$(chlorinated)	1	48h	-/24h	48h
Fecal Streptococcus	300, 250	SP	100	None, $Na_2S_2O_3$(chlorinated)	1	48h	-/24h	48h
Pseudomonas	300, 250	SP	100	None, $Na_2S_2O_3$(chlorinated)	1	48h	-/24h	48h
Iron Related Bacteria	300, 250	SP	100	None	1	48h	-	-
Chlorophyll-a	1000	AG	1000	None, Wrap in Aluminum Foil	1, in dark	30d	- /30d	-
ORGANICS								
Diquat/Paraquat	1000	P	250	None, $Na_2S_2O_3$(chlorinated)	1	14dpre/20dpost	7dpre/21dpost	20d
Glyphosate	1000	P	50	None, $Na_2S_2O_3$(chlorinated)	1	14d	14d	20d
Glycols	40	GV	40	None	1	7d		
OC Pesticides	1000	AG	1000	None, $Na_2S_2O_3$(chlorinated)	1	10dpre/40dpost	14dpre/30dpost	42d
PAH's	1000	AG	1000 (x2)	None	1	14dpre/40dpost	14dpre/30dpost	35d
PCB's	1000	AG	1000	None, $Na_2S_2O_3$(chlorinated)	1	10dpre/40dpost	14dpre/30dpost	42d
PHC (F1)	40	AGV	40 (x2)	None, $Na_2S_2O_3$(chlorinated), HCl	1	14d	-	7d
PHC (F2-F4)	1000	AG	1000	None	1	14d	-	14dpre/7dpost
Phenols by GC/MS	1000	AG	1000	None	1	7dpre/ 30dpost	14dpre/30dpost	20d/30d(MISA)
SVOC (Acid, Base/Neutral Ext.)	1000	AG	1000 (x2)	None	1	14dpre/40dpost	14dpre/30dpost	30d
VOC's	40	AGV	40 (x2)	None, $Na_2S_2O_3$(chlorinated), HCl	1	7 to 14d	14d/14d	*14d, 7to14(MISA)
SUBCONTRACTED PARAMETERS								
Dioxins/Furans	1000	AG	1000	None	1	30d	30d	
Formaldehyde	1000	AG	1000	None	1	7d		
NDMA	1000	AG	1000 (x2)	None	1	10d		10 d
NTA	1000	AG	100	None	1	30d		30 d
Radionuclides (Gross Alpha, Beta and Tritium)	1000	P	1000	None / HNO_3	1	10d / 6m		
Radionuclide (ODWS Table 3)	1000	P	1000 (x3)	None / HNO_3	1	10d / 6m		

Sample Container Codes:

P = Plastic, either HDPE or PETE

G = Glass, GV = Glass Vial

AG = Amber Glass, AGV = Amber Glass Vial,

SP = Sterile Plastic

* Teflon-lined phenate free cap

Storage Conditions Codes:

1 = 4 ± 3°C

2 = Room Temperature (if preserved)

d = days

m = months

Imm = Immediate

Appendix B: Soil Sample Analysis/General

Parameter	Sample Containers		Minimum Volume (mL)	Preservative	Storage Conditions	Holding Times		
	Size (mL)	Type				Caduceon	EPA	MOE
PHC (F2-F4)	180	AGJ	180	None	1	7 d	-	14 d
BTEX/PHC (F1)	100	AGJ	50	None	1	7 d	-	7 d
VOC's	100	AGJ	50	None	1	7 d	14 d	7 to 14 d
Metals (including mercury)	180	AGJ	180	None	2	28 d	28 d	Indefinite
Inorganic General	180	AGJ	180	None	2	see individual	see individual	see individual
Oil & Grease	180	AGJ	180	None	1	28 d	-	-
Nutrients (TOC,TP,TKN)	180	AGJ	180	None	2	28 d	-	-
Anions	180	AGJ	180	None	2	28 d	-	-
Semivolatiles	180	AGJ	180	None	1	see individual	see individual	see individual
Pesticides	180	AGJ	180	None	1	see individual	see individual	see individual

Sample Container Codes:
AGJ = Amber Glass Jar

Storage Conditions Codes:
1 = 4 ± 3°C
2 = Room Temperature

Indefinite = indefinite when dried
individual = individual parameter test method
d = days m = months

Appendix C: Bottles required for Regulatory Ontario Drinking Water Submissions

Parameter	Bottle	Sampling	Storage
THM's	Two - 40 mL VOC amber glass vials, $Na_2S_2O_3$ added	Fill slowly and completely - no air bubbles present	4 ± 3°C
Nitrate and Nitrite	125 mL HDPE, 250 mL HDPE or 500mL PETE, no preservative (4°C)	Grab	4 ± 3°C

Schedule 23: Inorganic Parameters

Parameter	Bottle	Sampling	Storage
Metals Mercury	250 mL HDPE, HNO_3 added $K_2Cr_2O_7 + HNO_3$	No rinsing. Be careful of acid preservative	4 ± 3°C

Schedule 24: Organic Parameters

Parameter	Bottle	Sampling	Storage
VOC's	Two - 40 mL VOC amber glass vials, $Na_2S_2O_3$ added	Fill slowly and completely - no air bubbles present	4 ± 3°C
Pesticides	**2**- 1 L Amber Glass, no preservative – **Pest MS,** **1** - 1 L HDPE, $Na_2S_2O_3$ added - **Diquat, Paraquat & Glyphosate** 2 - 1 L Amber Glass, no preservative – OC Pesticides	Grab	4 ± 3°C

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	GC/DEP
03-Sep-03	1.1	Updated Document to reflect the changes due to the changes in the ODWS (i.e O. Reg 170/03)	GC
08-Apr-05	1.2	Section 4.0 Added to address specific policies pertaining to the collection and handling of Drinking Water Samples under the Safe Drinking Water Act Appendic C – Number of bottles required for Schedule 24 Pesticides Sampling updated	GC/DEP
22-Nov-07	2.0	Sections 1, 2 & 3 rewritten to encompass company wide policies Appendix C removed as too lab specific Appendices A & B updated Appendix D renamed Appendix C	SB/GC/DEP

Document Review
This document was last reviewed and authorized by:

Laboratory Branch Manager _____ Date _____

Biochemical Oxygen Demand

1.0 Scope and Application

1.1 The Biochemical Oxygen Demand (BOD_5) is an indicator of the dissolved oxygen required for the decomposition of organic materials by aerobic bacteria. The BOD_5 is used to assess the oxygen demand of organic wastes, to determine the biodegradable loadings to treatment plants, and to evaluate the efficiency of waste treatments. The test requirement for a five day incubation period imposes a lengthy time delay in establishing the demand of a particular waste. This method is applicable for BOD determination in waters such as ground water, surface water, and waste waters.

1.2 The analytical range for this method is approximately 1 mg/L to 30000 mg/L with a maximum sample volume of 300mL.

1.3 By adding nitrogen inhibitor to analysis bottle, carbonaceous BOD ($CBOD_5$) may be determined. The scope and application is the same as for BOD determination.

2.0 Principle and Theory

2.1 Principles

2.1.1 The BOD_5 is a measure of dissolved oxygen depletion during a 5 day incubation period at a specified temperature (20°C). The units are milligrams per litre as oxygen. It is determined by diluting a suitable sample aliquot with dilution water. The dilution should be such that about 50 % of the dissolved oxygen is depleted after 5 days incubation. The dissolved oxygen is determined using a dissolved oxygen probe, as soon as possible after set-up; and again after the incubation period. The BOD_5 is expressed as the amount of dissolved oxygen in mg utilized by 1 litre of sample during a 5 day incubation period at 20°C. The principle of the oxygen electrode is based on the relationship between the concentration of dissolved oxygen in a sample and the current generated by its reduction under controlled conditions. The electrode probe contains an electrolytic cell separated from test sample by a teflon membrane which is permeable to dissolved oxygen. When the probe is immersed in a sample, a portion of the oxygen in the sample diffuses through the membrane into the electrolytic cell and is reduced at the cathode. The resultant change in current is directly proportional to the oxygen concentration present in the sample, and is read out digitally as mg/L of O_2. A suitable velocity of water across the membrane is maintained by a motorized stirrer and a built-in thermistor compensates for temperature variations.

2.2 Interferences

2.2.1 Most heavy metals, sulphates , and cyanide interfere with biological processes. Any gases, which diffuse through the membrane and enter into a redox reaction, will interfere. Chlorine is the most commonly known gas of this type and its interference can be nullified by raising the pH to 12 and converting the gas to hypochlorous ion.

2.2.2 Prolonged exposure to oily samples will produce a film on the membrane which might retard the diffusion of oxygen. Such film should be removed by placing the electrode membrane in hot water for a short period of time.

2.2.3 Prolonged exposure to samples containing hydrogen sulphide will result in corrosion of the lead anode. Periodic cleaning of the electrodes with 50% HCl solution controls this poisoning problem.

3.0 Safety

3.1 Extreme caution should be exercised when preparing acidic and caustic solutions. A full face shield, an apron and gloves should be worn when working with concentrated solutions. Goggles, gloves and a lab

coat are suitable for working with diluted solutions. Refer to each chemical MSDS for detailed hazards.

3.2 Refer to Caduceon Safety Manual for general laboratory safety practices and procedures.

4.0 Sample Requirements

4.1 Samples must be collected in plastic or glass bottles, and must be refrigerated as soon as possible after sampling at 4 ± 3°C; and stored in the dark to minimize bacterial decomposition and photosynthetic activity. Store not longer than 96 hours. Preservatives are not employed because they may retard bacterial action during incubation. The minimum sample volume submitted for analysis is 500 milliliters.

4.2 Samples containing caustic alkalinity or acidity are neutralized to pH 5.0 to 8.0 with H_2SO_4 or NaOH using a pH meter.

4.3 Chlorine residuals, if present in a sample, are overcome by dechlorinating the sample by adding Na_2SO_3 solution and seeding the dilution water. The dechlorinating procedure applies for effluent samples and treated drinking water samples. Other samples must be treated if from an industrial process using chlorine in the manufacturing stream.

4.4 Samples supersaturated with dissolved oxygen must have the dissolved oxygen content reduced to saturation, 9.17mg/L at 20°C, by aerating with compressed air or by vigorous shaking of the sample in its container.

5.0 Equipment

5.1 BOD bottles - 300mL glass

5.2 20L carboy

5.3 Incubator, thermostatically controlled at 20°C ± 1°C (temperature reading accuracy verified as per Caduceon SOP-10)

5.4 Winkler titration apparatus

5.5 Dissolved oxygen electrode

5.6 Volumetric flasks, Class A

5.7 Volumetric pipets, Class A

5.8 Disposable graduated pipets, 10mL size

5.9 25.0mL graduated buret, 0.1mL graduation (dispensing volume accuracy verified as per Caduceon SOP-08)

5.10 Filters, Whatman GF/C or equivalent

5.11 Graduated cylinders, various sizes (dispensing volume accuracy verified as per Caduceon SOP-08)

Note: Clean glassware (except BOD bottles) with phosphate-free detergent; rinse thoroughly with reagent grade water, and drain. Dry flasks and pipets before re-use
BOD bottles must be cleaned as follows:

- Place all bottles in the designated dishwasher and wash on normal cycle using phosphate-free detergent;
- Let drain, then rinse well with Reagent-grade water to remove possible copper contamination;
- Allow the bottles to dry before use.

6.0 Reagents

Note: Record all reagent preparation in the appropriate reagent preparation log.

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

6.2 Reagents for sample pretreatment:

6.2.1 1N Sodium hydroxide solution: In a 100mL volumetric flask containing approximately 50mL reagent grade water, dissolve 4.0g NaOH. Dilute to mark. Prepare fresh every six months or as needed. Store at room temperature.

6.2.2 1N Sulfuric acid solution: In a 100mL volumetric flask containing about 50mL reagent grade water, add 2.8mL concentrated H_2SO_4. Dilute to mark. Prepare fresh yearly or as needed. Store at room temperature.

6.2.3 50% Acetic acid solution (v/v): In a 200mL volumetric flask containing approximately 50mL reagent grade water, add 100mL concentrated acetic acid. Dilute to mark. Prepare fresh weekly or as needed. Store at room temperature.

6.2.4 5% Potassium iodide solution (w/v): In a 200mL volumetric flask containing approximately 100mL reagent grade water, dissolve 10g KI. Dilute to mark. Prepare fresh weekly or as needed. Store at room temperature.

6.2.5 Sodium sulphite solution: In a 100mL volumetric flask containing approximately 50mL reagent grade water, dissolve 0.158g Na_2SO_3. Dilute to mark. Make fresh daily. Prepare fresh daily. Store at room temperature.

6.2.6 Nitrogen Inhibitor Hach 2579-24 (2-chloro-6 (trichloro methyl) pyridine). Refer to manufacturer certificate for holding time/expiry date. Store at room temperature.

6.3 Dilution water reagents:

6.3.1 Phosphate buffer solution: In a 500mL volumetric flask, dissolve 4.25g KH_2PO_4, 10.9g K_2HPO_4, 16.7g $Na_2PO_4 \cdot 7H_2O$ and 0.85g NH_4CL in about 500mL reagent grade water. Dilute to mark. Discard reagent if there is any sign of biological growth in the stock bottle. Prepare fresh monthly or as needed. Store at room temperature.

6.3.2 Magnesium sulphate solution: In a 500mL volumetric flask, dissolve 11.25g $MgSO_4 \cdot 7H_2O$ in reagent grade water and dilute to mark. Prepare fresh monthly or as needed. Store at room temperature.

6.3.3 Calcium chloride solution: In a 500mL volumetric flask, dissolve 13.75g $CaCl_2$ in reagent grade water and dilute to mark. Prepare fresh monthly or as needed. Store at room temperature.

6.3.4 Ferric chloride solution: In a 500mL volumetric flask, dissolve 0.075g $FeCl_3$ in reagent grade water and dilute to mark. Prepare fresh monthly or as needed. Store at room temperature.

6.4 Reagents for Winkler Dissolved Oxygen (DO) Determination:

6.4.1 Manganese sulphate solution: Dissolve 364g $MnSO_4 \cdot H_2O$ in reagent grade water. Filter the solution through a Whatman #4 filter and dilute to 1L in a 1.0L volumetric flask. Prepare fresh yearly or as needed. Store at room temperature.

6.4.2 Alkali-Iodide-Azide Reagent: In a 1.0L volumetric flask, dissolve 500g NaOH and 150g KI in 800mL of reagent grade water. To this solution, add 10g NaN_3 (sodium azide) dissolved in 90mL of reagent grade water. Dilute to mark. Prepare fresh yearly or as needed. Store at room temperature.

6.4.3 1:1 Sulphuric acid: To a 200mL volumetric flask containing approximately 75mL reagent grade water, add slowly 100mL concentrated sulfuric acid. Mix thoroughly and allow to cool to room temperature. Dilute to mark with reagent grade water. Prepare fresh yearly or as needed. Store at room temperature.

6.4.4 Starch Solution: Dissolve 3.00g soluble starch arrowroot powder and 0.625g salicylic acid ($C_6H_4(OH)COOH$) with approximately 50mL reagent grade water. Slowly add, with stirring, to

about 400mL of boiling reagent grade water in a 500mL volumetric flask. Dilute to mark; allow to boil for a few minutes, and let settle overnight. Retain supernatant. Prepare fresh weekly or as needed. Store at room temperature.

6.5 Potassium Bi-Iodate Stock Solution 0.10 N: In a 1.0L volumetric flask, dissolve 3.24g of $KH(IO_3)_2$ in reagent grade water and dilute to mark. Prepare fresh every six months or as needed. Keep refrigerated at 4 ± 3°C.

6.6 Potassium Bi-Iodate Working Standard Solution 0.0250 N: Using a 25.0mL volumetric pipet, transfer 25.0mL of potassium bi-iodate stock solution to a 100mL volumetric flask. Dilute to mark with reagent grade water. Prepare fresh daily. Store at room temperature.

6.7 1:10 Sulphuric acid: To a 200mL volumetric flask containing approximately 150mL reagent grade water, add slowly 20mL concentrated sulfuric acid. Mix thoroughly and allow to cool to room temperature. Dilute to mark with reagent grade water. Prepare fresh yearly or as needed. Store at room temperature.

6.8 Sodium Thiosulphate ~0.025 N: In a 1.0 L volumetric flask, dissolve 6.205g $Na_2S_2O_3 \cdot 5H_2O$ in freshly boiled reagent grade water and mix thoroughly. Preserve with 2.0g NaOH. Allow solution to cool to room temperature and dilute to mark with freshly boiled reagent grade water. Prepare fresh monthly or as needed. Store at room temperature.

6.8.1 Standardization of Sodium Thiosulphate solution: Dissolve approximately 2.0g of iodate-free KI in an erlynmeyer flask containing 100 to 150mL distilled water. Using volumetric pipettes, add 2.0mL of 1:10 sulphuric acid solution, followed by 20.0mL potassium bi-iodate working standard solution (section 6.6). Dilute to approximately 200mL with reagent grade water and titrate with sodium thiosulphate solution (section 6.8). When a pale straw colour is reached, add approximately 2mL of starch solution and complete the titration to the colourless end point. The normality of the thiosulphate solution is calculated as:

$$A = \frac{B \times C}{D}$$

A = normality of sodium thiosulphate solution
B = normality of potassium bi-iodate working standard
C = volume (mL) of potassium bi-iodate working standard used
D = volume (mL) of sodium thiosulphate solution added

6.8.2 Record the normality of the sodium thiosulphate solution in the analyst work book. Standardization is performed when the reagent is prepared.

6.9 Seeding Material - must be obtained fresh weekly from RMOC or other sewage facility. Store in a plastic container, labelled with the date obtained and the expiry date. Keep refrigerated at 4 ± 3°C.

6.10 Standard Preparation

6.10.1 Stock Standard, 200mg/L (Glucose-glutamic acid solution): Dry glucose and glutamic acid at 105°C for one hour. In a 1.0L volumetric flask, add 150mg glucose and 150mg glutamic acid. Dilute to mark with reagent grade water. Prepare fresh monthly. Store at 4 ± 3°C.

6.10.2 QC-01 Working Standard, 50mg/L: Take 125mL of stock standard (200mg/L) and dilute to 500mL. Transfer 20mL of this working standard solution into a BOD bottle. Add 2mL of seed and fill with dilution water. Prepare fresh daily.

7.0 Test Procedure

7.1 Preparation of dilution water: Place desired volume of reagent grade water in a suitable bottle and add 1mL each of phosphate buffer, $MgSO_4$, $CaCl_2$ and $FeCl_3$ solutions per liter of water. Saturate with DO by aerating with organic-free filtered air for 15-20 minutes.

7.2 Calibration and Operation of the Oxygen Meter:

7.2.1 The oxygen meter is calibrated against a sample of known DO concentration as determined by the Winkler method. Calibration is routinely performed against dilution water.

7.2.1.1 Fill 4 BOD bottles with dilution water.

7.2.1.2 Determine the Winkler DO concentration in the first two bottles by adding 2mL $MnSO_4 \cdot H_2O$ and 2mL of alkali-azide reagent to each bottle.

7.2.1.3 Close the bottle carefully with the stopper to exclude air bubbles and mix by inverting the bottles several times.

7.2.1.4 When precipitation has sufficiently settled, add 2.0mL H_2SO_4.

7.2.1.5 Close the bottle carefully with the stopper again and mix by inverting several times.

7.2.1.6 A 200mL aliquot of sample is titrated with 0.025 N $Na_2S_2O_3$. Add 2.0mL starch solution towards the end of the titration when a pale straw colour has been reached. Titrate to a clear end point.

7.2.1.7 The volume of sodium thiosulphate standard solution dispensed equals the DO of the aerated water. Correct this value for the true normality of the sodium thiosulphate standard solution (obtained in section 6.8.1) using the following calculation:

$$A = \frac{B \times 0.025}{C}$$

A = corrected DO value
B = volume of $Na_2S_2O_3$ dispensed
C = actual normality of $Na_2S_2O_3$ (section 6.8.1)

Note: The DO meter probe is adjusted based on the average of two DO results.

7.2.1.8 Insert the oxygen probe into one of the two remaining BOD bottles and switch the stirrer on.

7.2.1.9 Switch instrument to zero position and zero the meter with the zero knob.

7.2.1.10 Switch instrument to calibrating position and calibrate meter according to the DO value obtained from titration.

7.2.1.11 Verify the DO measurement with the oxygen probe and meter on the remaining bottle of dilution water and record the result in the analyst workbook.

7.3 pH adjustment for samples with pH outside range 5.0 to 8.0: Using a pH meter (see Caduceon method, A-pH-01), adjust the pH of the sample by adding 1N NaOH (pH < 5.0) or 1N H_2SO_4 (pH > 8.0) dropwise until the sample pH is within the required range. Ensure that the change in sample volume is less than 1 percent and record the volume and type of solution added.

Note: Check the initial pH of each sample prior to set-up with pH paper, and use the pH meter only in cases where pH adjustment is required.

7.4 Removal of residual chlorine: Place 50mL of sample, 5mL of 5% potassium iodide solution, 5mL of 50% acetic acid solution, and about 3mL starch solution into a 250mL erlenmeyer flask. Mix by swirling. If residual chlorine is present in the sample, the colour of the solution will turn to dark blue. Add 0.01N Na_2SO_3 solution dropwise, while swirling, until the the solution turns clear. Record the amount of 0.01N Na_2SO_3 required per 50mL of sample directly onto the sample bottle. The required amount of 0.01N

Na_2SO_3 necessary for neutralization will be added later, to portions of the original sample prior to dilution.
Note: Excess Na_2SO_3 exerts an oxygen demand and reacts slowly with certain organic chloramine compounds that may be present in chlorinated samples.

7.5 Filtration: Samples submitted for filtered BOD_5, are filtered through a Whatman GF/C filter, prior to dilution.

7.6 Sample seeding: For samples needing pH adjustment, add seeding material. For samples containing residual chlorine, add the required amount of 0.01N Na_2SO_3 solution and seeding material (approximately 2mL). All industrial wastes and final effluent samples should be seeded as mentioned above.

7.7 Sample dilutions: Using a wide-tip volumetric pipet or a graduated cylinder, add the desired sample volume to individual BOD bottles of known capacity. Fill bottles with enough dilution water, seeded if necessary, so that insertion of the stopper will displace all air, leaving no bubbles.

7.7.1 For dilutions greater than 1 : 100, make a primary dilution in a graduated cylinder before making final dilution in the bottle.

7.8 Dilution Water Blanks: Use a dilution water blank as a rough check on quality of unseeded dilutions, water and cleanliness of incubation bottles. The DO uptake should not be more than 0.2mg/L.

7.9 Seed Control: Determine BOD of seeding material as for any other sample. The DO uptake of seeded dilution water should be between 0.6 and 1.0mg/L.

7.10 Quality Control Checks: Take 125mL of glucose-glutamic acid (200mg/L) and dilute to 500mL. Transfer 20 mL of glucose-glutamic acid reagent into BOD bottle. Add 2mL of seed and fill with dilution water.

7.11 Matrix Spike Preparation: Transfer 10mL of 50 mg/L working standard (section 6.101.2) to the BOD bottle containing the appropriate amount of diluted sample. The target value is 1.76 mg/L of O_2 depletion.

7.12 Determine the initial DO on all BOD bottles, using the oxygen probe and meter. Stopper tightly; water seal and incubate for 5 days at 20 ± 2°C. Rinse DO electrode between determinations to prevent cross-contamination of samples.

7.13 Determine final DO for each bottle, as in section **7.12**, after 5 days of incubation.

Note: Each set-up must include dilution water blanks, standards, and seeded dilution water. These must be set up in duplicate. Samples are prepared at two to five different dilutions. Duplicate samples are analyzed at a level of at least once after every ten samples and a matrix spike after every thirty samples. If the relative deviation in duplicate and spike results is greater than the control limits (refer to the QC log), the duplicate sample and spike recovery results are reported to the client with a comment to qualify the data as suspect. Sample holding time prevents samples from being re-analyzed if duplicate results are not within the required range.

7.14 Carbonaceous B.O.D.: Requires the addition of ~0.16 g (2 shots from the dispenser) of nitrogen inhibitor which is added to each dilution bottle of the samples requiring C.B.O.D.. The amount of the inhibitor put in each bottle (including standards and blanks) is done with the chemical applicator supplied from Hach.

7.15 Documentation Procedures

7.15.1 Document all required information in the BOD Analysis workbook when analyzing samples and QC solutions.

7.15.2 Document all required information in BOD Standard/Reagent Preparation logs when

preparing Reagents and QC Standards.

7.15.3 Document all maintenance, comments and any changes in the instrument log book.

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

7.15.5 The QC log data file is found on the Ottawa server: ...QC LOGS\Organic Chemistry\BOD.xls.

7.15.6 Open this file and enter the data in the appropriate fields; Date, Expected Concentration for working QC-01, Found Concentration for working QC-01, Duplicate and Spike results and Blanks.

7.15.7 The excel program will calculate the % recoveries of the QC-01 and spikes and the % difference of the duplicate samples and the average concentration of the blanks.

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

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

8.0 Calculations & Reporting

8.1 When sample is not seeded:

$$A = \frac{(B - C) \times 300}{D}$$

A = concentration of BOD_5 (mg/L)
B = initial DO reading (mg/L)
C = final DO reading (mg/L)
D = volume of sample analyzed (mL)

8.2 When sample is seeded:

$$A = \frac{[(B - C) - E] \times 300}{D}$$

A = concentration of BOD_5 (mg/L)
B = initial DO reading (mg/L)
C = final DO reading (mg/L)
D = volume of sample analyzed (mL)
E = DO reading of seed (mg/L)

8.3 When a sample is spiked:

8.3.1 Depletion of working standard = DO (initial) – DO (final). Target result is 1.76 mg/L

8.3.2 Depletion of sample = DO (initial) – DO (final)

8.3.3 Depletion of spiked sample = DO (initial) – DO (final)

8.3.4 Actual Spike amount recovered, as depletion = Depletion (8.3.3) – Depeltion (8.3.2)

8.3.5 % Spike Recovery = $\frac{\text{Result (8.3.4)} \times 100}{\text{Result (8.3.1)}}$

8.4 Criteria for Result Acceptance:

8.4.1 The following criteria must be met for a result to be accepted as valid.

8.4.1.1 The final DO reading must be ≥ 1mg/L.

8.4.1.2 The difference between the final DO reading and the initial DO reading must be ≥ 1mg/L.

8.4.2.3 There must be no evidence of sample toxicity at higher sample concentrations, or the existance of an obvious anomaly. All acceptable dilution results are averaged to obtain the reportable result.

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

8.5.1 The calculated sample result is entered in the Laboratory Run module of the LIMS. The LIMS will display the correct reportable result.

9.0 Method Validation & Method Performance

9.1 Method Validation Data

	Data Points	Calculation (Reporting MDL)
MDL (mg/L)	10	1.1 (1)
Precision (%)*	10	3.8
Accuracy (%) *	10	101

* Based on 10mg/L standard results

9.2 Quality Control Standards

Sample ID	Number of Data Points	Expected (mg/L)	Mean (mg/L)	Average Bias (mg/L)	Standard Deviation	UCL (mg/L)	LCL (mg/L)
QC-01	56	50	50.8	0.8	2.0	57	45
BODSP-01 Spike (O_2 Depletion)	10	1.76	1.76	0.00	0.27	2.56	0.97

9.3 Duplicates

Analytical Range (mg/L)	Number of Data Sets	Acceptable Limits-RPD %
≤ 10	55	100
> 10	70	26

9.4 Method Uncertainty

9.5.1 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.

Typical Concentration (mg/L)	Combined Uncertainty (mg/L)	Expanded Uncertainty * (Percent)
5	0.7	27
50	5.2	20
200	20.3	20

* 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	104
Standard Deviation (sd)	8.5
3sd (99% confidence level)	25.5
Number of Data Points (n)	14

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, 5210-B, 21st Ed., 2005

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

10.3 Caduceon SOP-07, Control Charting

10.4 Caduceon SOP-08, Verifying Delivery Volumes

10.5 Caduceon SOP-10, Thermometer Calibration and Verification

10.6 Caduceon SOP-23, Determination of Uncertainty in Measurement

10.7 Caduceon SOP-39, LIMS Training

10.8 Caduceon SOP-43, Non-conformity Logs

10.9 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
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.

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 limits 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 _____