

**Fraser River
Action Plan**



**Recommended
Guidelines for
Wastewater
Characterization
in the Fraser
River Basin**

**Volume II
Draft Methods
Manual**



CANADA'S GREEN PLAN
LE PLAN VERT DU CANADA

Canada

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**RECOMMENDED GUIDELINES
FOR WASTEWATER CHARACTERIZATION
IN THE FRASER RIVER BASIN**

**VOLUME II
DRAFT METHODS MANUAL**

DOE FRAP 1993-11

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PREFACE

This consultants' report contains the results of a project conducted under contract to Environment Canada. The purpose of publishing a draft manual at this time is to provide an opportunity for its review by individuals, agencies, and companies participating in the Fraser River Action Plan.

Comments regarding this report are welcomed and should be addressed to:

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EXECUTIVE SUMMARY

The Fraser River Action Plan, an initiative under Canada's Green Plan, has a key goal to implement a cooperative pollution abatement program to reverse the trend of environmental degradation in the Fraser River ecosystem. Before undertaking a pollution abatement program, it is necessary to identify the sources of specific contaminants. Therefore, the Fraser Pollution Abatement Office (FPAO) is implementing a Wastewater Characterization Program that addresses major industrial and municipal effluents and point source discharges from lighter industries and agriculture.

The objectives of the Wastewater Characterization Program are threefold:

- to determine sources and loadings of contaminants which have already been identified as concerns in the Fraser Basin;
- to identify the presence and, if they exist, the loadings of persistent toxic substances defined under the Canadian Environmental Protection Act; and
- to identify toxic or otherwise environmentally disruptive effluents.

It is envisaged that wastewater characterization will be conducted jointly by FPAO, other government agencies, crown corporations, first nations, industries, and/or consultants. In order to ensure comparability of data generated at different sites and by different agencies, a consistent set of field sampling protocols and analytical procedures must be established. This Guideline' Document has been developed to provide consistent protocols for field sampling and analytical methods to be used for the Wastewater Characterization Program. The document outlines:

- guidelines on field sampling procedures for chemical characterization, including sample collection, preservation, processing, and transportation;
- methods of measuring effluent flow rate;
- field quality assurance measures;
- preferred and alternate methods for chemical analyses;
- data reporting specifications; and
- collection, analysis, and reporting requirements for effluent toxicity tests.

By following the procedures in this document, different individuals and agencies can generate reliable and comparable data on contaminant concentrations and loadings and effluent toxicity.

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INTRODUCTION

1.1 The Wastewater Characterization Program

The Fraser River Action Plan, an initiative under Canada's Green Plan, has a key goal to implement a cooperative pollution abatement program to reverse the trend of environmental degradation in the Fraser River ecosystem. The specific targets of the pollution abatement program are to:

- reduce by 30% the total discharge of environmentally disruptive effluents entering waters of the Fraser River Basin by the year 1997; and
- reduce the release of persistent toxic substances entering the waters of the Basin, to the extent attainable by best practicable technology.

Persistent toxic substances are defined by the Priority Substances List (PSL) and Toxic Substances List (TSL) of the Canadian Environmental Protection Act (CEPA).

The Fraser Pollution Abatement Office (FPAO) has the responsibility to determine strategies for meeting these pollution abatement targets. The FPAO's first task is to identify the sources of specific contaminants through a program to characterize wastewater discharges in the Fraser Basin. The Wastewater Characterization Program addresses major industrial and municipal effluents and point source discharges from lighter industries and agriculture.

The objectives of the Wastewater Characterization Program are threefold:

- to determine sources and loadings of contaminants which have already been identified as concerns in the Fraser Basin;
- to identify the presence and, if they exist, the loadings of persistent toxic substances as defined under CEPA; and
- to identify toxic or otherwise environmentally disruptive effluents.

To meet these objectives, FPAO contracted consultants to recommend monitoring parameters and toxicity tests. Volume I of this series, the *Development Document* (Norecol 1993), provides the rationale for the selection of physical/chemical monitoring parameters. Another document (Environmental Management Associates and Hydroqual Laboratories 1993) describes the selection of toxicity tests.

1.2 Objectives of the Methods Manual

It is envisaged that wastewater characterization will be conducted jointly by FPAO, other government agencies, crown corporations, first nations, industries, and/or consultants. In order to ensure comparability of data generated at different sites and by different agencies, a consistent set of field sampling protocols and analytical procedures must be established. Therefore, this Methods Manual was developed to provide consistent protocols for field sampling and analytical methods to be used in the quantitative assessment of wastewater discharges within the Fraser River Basin. The document outlines:

- guidelines on field sampling procedures for chemical characterization, including sample collection, preservation, processing, and transportation;
- methods of measuring effluent flow rate;
- field quality assurance measures;
- preferred and alternate methods for chemical analyses;
- data reporting specifications; and
- collection, analysis, and reporting requirements for effluent toxicity tests.

In addition, the glossary in Appendix 1 provides definitions of the types of effluent discharges and sampling methods referred to in this document.

By following the procedures in the following chapters, different individuals and agencies can generate reliable and comparable data on contaminant concentrations and loadings and effluent toxicity.

PARAMETER SELECTION

Volume I of this series, the Development *Document*, presents the list of parameters recommended for wastewater characterization in the Fraser Basin and provides the rationale for their selection. This section summarizes the parameters and indicates the industries to which each applies.

These lists are intended as the starting point for any wastewater characterization field program. They identify the parameters to be measured at specific types of industrial sites. In practice, site specific knowledge may also be used to determine the parameters to be measured at a particular discharge. Chapter 3 describes the sample collection and handling procedures that apply to each parameter.

Monitoring parameters for the Fraser Basin Wastewater Characterization Program are divided into two groups:

- core parameters to be measured at all sites, and
- industry-specific parameters to be measured in effluents from the particular industries known or suspected to discharge them.

The core parameters include those physical and chemical parameters which provide information on general effluent characteristics and/or aid in the interpretation of toxicity test results. Table 2-1 lists the core parameters and the key reasons for measuring them.

Table 2-2 lists the industry-specific parameters, indicating the industries to which they apply. Several of the industries listed in this table encompass several subsectors. Not all of the parameters suggested for the major industry group apply to all subsectors. Tables 2-3 through 2-6 indicate parameters for specific subsectors within the sewage treatment, food production/food processing, concrete and industrial minerals, and forest products sectors.

TABLE 2-1

**CORE PARAMETERS FOR FRASER BASIN WASTEWATER CHARACTERIZATION
(TO BE MEASURED FOR ALL OUTFALLS)**

Temperature (field measurement)
Dissolved oxygen (field measurement)
pH (field and laboratory measurement)
Alkalinity
Conductivity (Specific conductance)
Total suspended solids (TSS) .
Dissolved organic carbon (DOC)
Chemical oxygen demand (COD)

TABLE 2-2

INDUSTRY SPECIFIC MONITORING PARAMETERS FOR WASTEWATER CHARACTERIZATION IN THE FRASER BASIN

PARAMETER	MUNICIPAL WWTP	FOOD PROD. PROCESSING	CONCRETE/ INDUST. MIN	FOREST PRODUCTS	PULP AND PAPER	CHEMICAL PRODUCTS	PLASTICS INDUSTRY	PETROLEUM INDUSTRY	METAL FINISHING	MINING & REFINING
Ammonia	X	X							X	X
Nitrite	X	X		X	X		X		X	X
Cyanide					X				X	
Metals*	X	X (1)	X	X (3)	X	X	X	X	X (5)	X (5)
Chromium**	X			X (3)	X	X	X	X	X (5)	X (5)
Arsenic	X			X (3)	X	X	X	X	X (5)	X (5)
Mercury	X				X	X	X	X		X
Chlorophenols*	X			X (3)	X		X			
Chloroguaiacols*					X					
Chlorocatechols*					X					
Chloroform					X					
Nonylphenol	X									
Dioxins/furans*	X				X (7)					
Polycyclic aromatic hydrocarbons*	X			X (3)	X	X		X	X	
Trichlorobenzenes	X				X (7)	X	X	X		
Tetrachlorobenzenes	X				X (7)	X	X	X		
Pentachlorobenzene	X				X (7)	X	X	X		
Hexachlorobenzene	X				X (7)	X	X	X	X	
1,4-Dichlorobenzene	X				X (7)	X	X	X		
1,2-Dichlorobenzene	X				X (7)	X	X	X		
1,2-Dichloroethane	X				X (7)	X	X	X		X
1,1,1-Trichloroethane	X				X (7)	X	X	X		
1,1,2,2-Tetrachloroethane	X				X (7)	X	X	X		X
Dichloromethane	X					X	X			
Trichloroethylene	X				X (7)	X	X	X		
Tetrachloroethylene	X				X (7)	X	X	X		
Benzene	X				X	X	X	X	X	
Toluene	X					X	X	X	X	X
Styrene						X	X			
Xylene	X				X	X	X	X	X	
Resin acids	X	X (2)		X	X	X				
Adsorbable organic halogens (AOX)					X					
Didecyl dimethyl ammonium chloride (DDAC)				X (3)						
3-Iodo-2-propynyl butyl carbamate (IPBC)				X (3)						
Biochemical oxygen demand (BOD5)	X	X	X	X (4)	X					
Chlorine, residual (field measurement)	X	X (6)								

WWTP - Wastewater Treatment Plant

* See Table 7-1 for complete listings

In general "Metals" refers to total metals; dissolved metals should be measured where indicated or to identify unknown toxicant

** Cr(VI) can also be measured to identify unknown toxicant

Notes:

- (1) Where metals used (canning)
- (2) Where woodwaste is used
- (3) Where specific wood preservatives/ anti-sapstain chemicals used (DDAC and IPBC measured on experimental basis only)
- (4) Where oil and grease are expected in effluent
- (5) Measure both total and dissolved metals
- (6) If wastewater is chlorinated
- (7) If pulp is bleached by chlorine or chlorine dioxide

TABLE 2-3				
INDUSTRY SPECIFIC MONITORING PARAMETERS FOR DOMESTIC SEWAGE EFFLUENTS IN THE FRASER BASIN				
TYPE	BOD5	AMMONIA	NITRITE	CHLORINATED VOLATILES*
Municipal Wastewater (WWTP)	Measure all parameters listed in Table 2-2			
Laundromats with Dry Cleaning Facilities	X	X	X	X
Private Domestic Sewage Discharges	X	X	X	
Industry Sanitary Effluent	X	X	X	

* Trichloroethylene and tetrachloroethylene

TABLE 2-4						
INDUSTRY SPECIFIC MONITORING PARAMETERS FOR FOOD PRODUCTION/FOOD PROCESSING INDUSTRIES IN THE FRASER BASIN						
TYPE	RESIN ACIDS	BOD5	AMMONIA	NITRITE	TOTAL METALS**	CHLORINE, RESIDUAL
Livestock Production	X*	X	X	X		
Meat/Poultry Production	X*	X	X	X		
Egg Production	X*	X	X	X		
Fish Farms/Hatcheries		X	X	X		
Fish Processing		X	X	X	X	X***
Canned Fruits and Vegetables		X	X	X	X	
Dairy/Fluid Milk	X*	X	X	X		
Feed Industry	X*	X	X	X		
Brewery		X	X	X	X	
Farms/Nurseries	X*	X	X	X		

* Where effluent includes runoff from woodwaste used as bedding, mulch, etc.

** Measure metals where canning process used; measure dissolved metals only if necessary to investigate source of effluent toxicity

*** Where effluent is chlorinated

TABLE 2-5**INDUSTRY SPECIFIC MONITORING PARAMETERS FOR CONCRETE AND INDUSTRIAL MINERALS INDUSTRIES IN THE FRASER BASIN**

TYPE	BOD5	TOTAL METALS*
Cement and Concrete	X	X
Sand and Gravel		X
Non-metallic minerals		X

* Measure dissolved metals to investigate source of effluent toxicity

TABLE 2-6

INDUSTRY SPECIFIC MONITORING PARAMETERS FOR FOREST PRODUCTS INDUSTRIES IN THE FRASER BASIN

ANTISAPSTAIN OR WOOD PRESERVATIVE USED	CHLORO-PHENOLS	PAH	METALS*	ARSENIC*	CR(VI)	DDAC (2)	IPBC (2)	BOD5	RESIN ACIDS	AMMONIA	NITRITE
DDAC-Based Products						X		X	X	X	X
NP-1 (DDAC+IPBC)						X	X	X	X	X	X
Pentachlorophenol	X							X	X	X	X
Creosote		X						X	X	X	X
Chromated Copper Arsenate			X	X	X (1)			X (3)	X	X	X
Ammoniacal Copper Arsenate			X	X				X (3)	X	X	X

* Measure both total and dissolved metals (if using AA, focus on copper and chromium); also total and dissolved arsenic

Notes:

- (1) Optional Cr(VI) analysis to investigate source of effluent toxicity
- (2) Recommended on experimental basis until method performance confirmed
- (3) Measure where woodwaste leachate is present

PREPARATION FOR SAMPLING

3.1 Introduction

Chapters 3 and 4 present specific sampling methodologies for use in the Wastewater Characterization Program. This chapter discusses the preparation necessary prior to undertaking a field sampling trip. Chapter 4 presents detailed field methods for collecting the samples and preparing sample composites. The procedures described in Chapters 3 and 4 apply to the collection of wastewater samples for chemical analysis. Wastewater sampling protocols for toxicity testing are presented in Chapter 9.

It is envisaged that most wastewaters will be sampled twice over a six-year period. Samples will normally be collected as flow proportionate composites over a 24-h day or other period equivalent to an operating day. For effluents whose quality is expected to vary substantially from day to day, collection of three samples over a one week period is recommended. Because of the limited duration of sampling planned for each site, the following discussion assumes that all samples will be collected manually.

Portable automatic sampling equipment is available for collecting flow proportionate composites, and it may be used, provided that material composition and temperature stability requirements are met. Due to variability in features and programmability of different models, details for using automatic samplers are not given here. However, the requirements for types of material that come in contact with the sample, storage temperature, and other handling precautions discussed in the following section also apply to sampling with automatic devices.

Actual field sampling programs must be designed with consideration of individual site circumstances. Sampling protocols discussed in Chapters 3 and 4 provide alternate methods for different site conditions. Definitions of the effluent discharge types and sampling methods referenced here are given in the glossary (Appendix 1).

3.2 Preparation Steps

Any field sampling program requires thorough preparation prior to leaving for the site. This preparation includes obtaining as much information as possible about the site and effluent characteristics, determining the numbers and types of samples to collect, contacting a qualified analytical laboratory to arrange for the required analyses, preparing sampling bottles and equipment, and assembling the proper labels, field logs

and laboratory submission forms. The following sections give detailed preparation instructions.

3.3 Obtain Site Information

The first step in preparing for the sampling program is to obtain as much information as possible about the site. Obtain and review discharge permits and any other available information regarding discharge access points and potential sampling locations. If possible, tentatively identify access points, and determine the number of locations to be sampled.

Site personnel should be contacted directly to confirm current operating status and obtain site-specific information. Table 3-1 lists the types of information that should be obtained before beginning a sampling program.

3.4 Tentatively Select Sampling Locations

Depending on the site and industry sector, some facilities will have more than one final discharge point. After obtaining the site specific information, determine the number of effluents to sample. If there is a limitation on the number of samples to be collected at any one facility, samples should be collected in the following order of priority for most industrial plants:

- Process Effluent
- Combined Effluent
- Storm Runoff
- Cooling Water
- Sanitary Effluent

Some exceptions apply. For example, at wood preservation plants, lumber mills, and concrete plants, storm runoff is usually of greatest concern. Also, if slimicides are used in the cooling water system, cooling water may be of greater concern.

3.5 Determine Sampling Frequency and Approach

The sampling period over which samples are collected, the sampling frequency within that period, and the decision of whether to collect single grab or composite samples will depend upon the type of effluent and expected variability in the effluent quality and flow. Recommended sampling frequencies are listed below. Figure 3-1 illustrates the main factors involved in selecting the sampling approach for a particular wastewater discharge,

Table 3-1
Sample Questionnaire for Plant Personnel

The following information should be obtained from plant personnel before the site visit:

- 1 How many final discharge points exist for each of the following effluent types?
 - Process Effluent
 - Combined Effluent
 - Cooling Water
 - Sanitary Effluent
 - Storm water

- 2 What is the type of access at each final discharge point?
 e.g. open channel, flowing pipe, valve, manhole)

- 3 What is the operating/discharge schedule which affects each point?
 (e.g. 8 hour shift, 24 hours per day, batch/periodic discharge, washdown events)

- 4 What is the estimated flow rate for each point?
 What is the diurnal flow variability?

- 5 Are flow data available?
 Are instantaneous data recorded on a data logger?

- 6 Is wash water available (for equipment cleaning)?
 Is there access to a power supply (for pumping)?
 Is lab space available for sample preparation?

- 7 Are any variations from normal operating practice anticipated
 (e.g. planned shutdown, process upset)

- 8 What are the site specific safety requirements?
 Is any safety training required prior to working at the site?
 Are there any hazardous chemicals used or anticipated present in the effluent?
 What shelter is available?

- 9 Other comments

Table 3-2

Sample Bottles, Preservation, Handling and Storage Requirements for Fraser Basin Wastewater Characterization Program Parameters

PARAMETER	Analytical Test Group	Volume		Preservative/ Temperature	Container	Maximum	Special Precautions
		Preferred	Minimum			Storage (Days)	
Chloroguaiacols (See Table 7-1) Chlorocatechols (See Table 7-1) Chlorophenols (See Table 7-1) Nonylphenol	Chloroguaiacols Chlorocatechols Chlorophenols Nonylphenol	1 L 1 L 1 L 1 L <i>Total</i>	800 mL 800 mL 800 mL 800 mL <i>1 X 1L</i>	4 °C	Amber Glass, Solvent Cleaned, Teflon Cap Liner	30 *1	
Dioxins/Furans (See Table 7-1)	Chlorinated Dibenzo-p--dioxins and Dibenzofurans	4 X 1 Litre	2 X 1 Litre	4 °C	Amber Glass, Solvent Cleaned	30 *1	Contact surfaces must be glass, teflon or stainless steel
Polycyclic aromatic hydrocarbons (See Table 7-1)	PAHs	1 L	800 mL	4 °C	Amber Glass, Solvent Cleaned, Teflon Cap Liner	30 *1	
Trichlorobenzenes Tetrachlorobenzenes Pentachlorobenzene Hexachlorobenzene	Chlorobenzenes	1 Litre 1 Litre 1 Litre 1 Litre <i>Total</i>	1 Litre 1 Litre 1 Litre 1 Litre <i>1 X 1L</i>	4 °C	Amber Glass, Solvent Cleaned, Teflon Cap Liner	30 *1	
1,4-Dichlorobenzene 1,2-Dichlorobenzene Tetrachloroethylene 1,1,1-Trichloroethane 1,2-Dichloroethane 1,1,2,2-Tetrachloroethane Trichloroethylene Dichloromethane Chloroform	Volatiles, Halogenated	3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL <i>Total</i>	3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL <i>3 x 40 mL</i>	4 °C	Vials *	7	Special sampling precautions necessary to ensure no headspace or air bubbles present in sample. If residual chlorine is suspected, precharge container with Na2SO3 crystals (approximately 10 crystals per vial) Volatiles must be sampled as 3 grabs.

Table 3-2

Sample Bottles, Preservation, Handling and Storage Requirements for Fraser Basin Wastewater Characterization Program Parameters

PARAMETER	Analytical Test Group	Volume		Preservative/ Temperature	Container	Maximum Storage (Days)	Special Precautions
		Preferred	Minimum				
Toluene Benzene Styrene Xylenes	Volatiles, Non-Halogenated	3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL <i>Total</i>	3 x 40 mL 3 x 40 mL 3 x 40 mL 3 x 40 mL <i>3 x 40 mL</i>	4 °C	Vials *	7	Special sampling precautions necessary to ensure no headspace or air bubbles present in sample. If residual chlorine is suspected, precharge container with Na ₂ SO ₃ crystals Volatiles must be sampled as 3 grabs.
Resin Acids (See Table 7-1)	Resin Acids	1 Litre	1 Litre		Amber Glass, Solvent Cleaned, Teflon Cap Liner	3	Contact surfaces must be glass, teflon or stainless steel
Adsorbable Organic Halides (AOX)	Adsorbable Organic Halides	500 mL	500 mL	pH to < 2 with HNO ₃	Amber Glass, Acid Rinsed, Heat-treated	30	No headspace in bottle
DDAC IPBC	Anti-sapstain Compounds	1 L 1 L <i>Total</i>	1 L 1 L <i>1 X 1 Litre</i>	5 mL - Rexonic NLS-7 (200 mg/L) & 10 mL Formaldehyde(37%)/L	Glass	14	
Biochemical Oxygen Demand (BOD ₅)	Biochemical Oxygen Demand	1 litre	500 mL	4 °C	HDLPE	4	No headspace in bottle

Notes :

* - No headspace or air bubbles in a 40 ml amber glass vial sealed with a teflon lined septum cap.

*1 - Samples for organic analysis should be extracted within 7 days of sample collection and analyzed within 30 days.

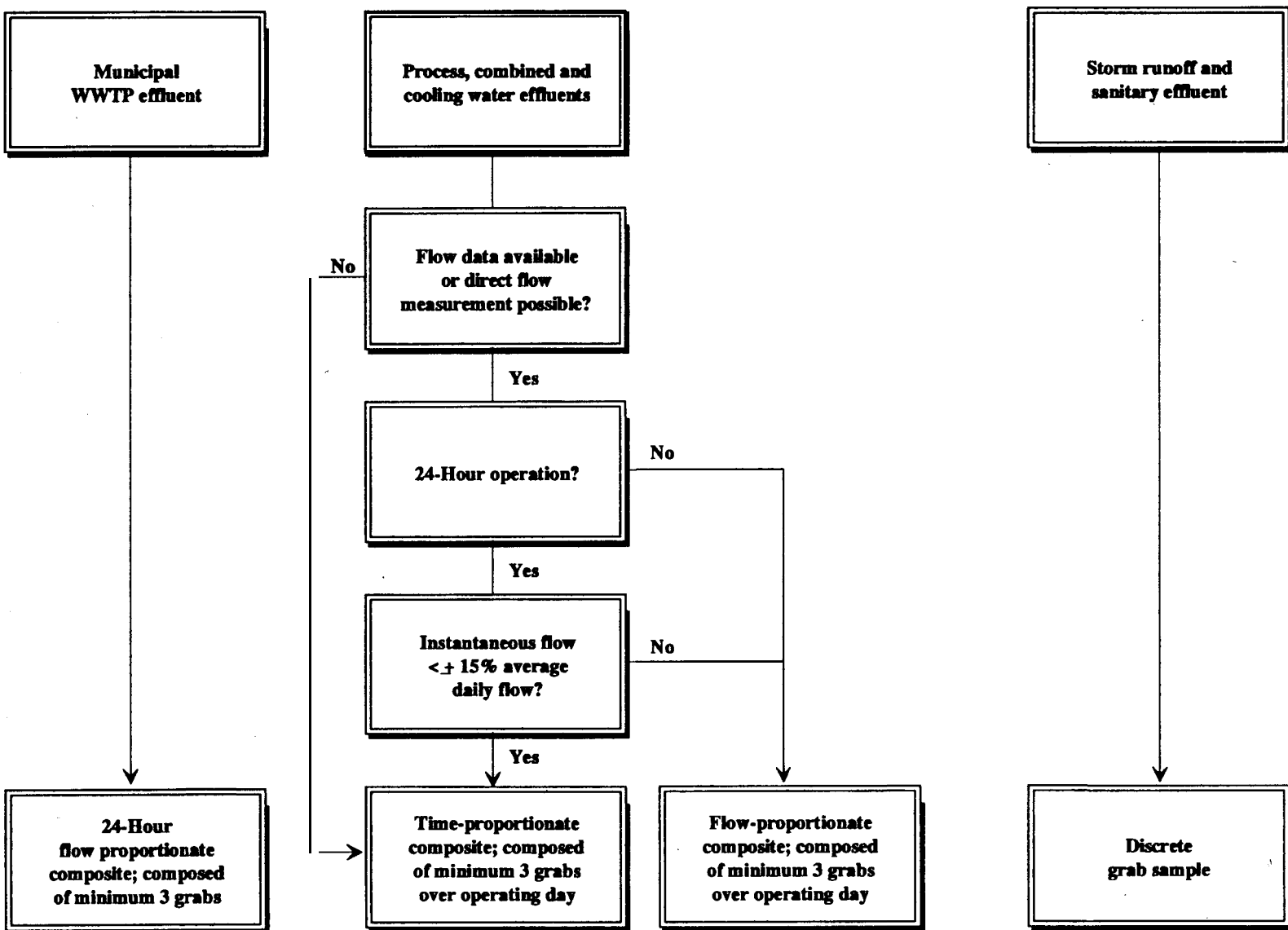
Samples with more than one test with similar preservative requirements, may share a sample container as long as there is sufficient sample volume to cover minimum volumes for all tests required. These are shown in bold where appropriate.

The typical volume requirements listed may vary depending on the laboratory's ability to meet the MDLs.

HDLPE = High Density Linear Polyethylene

FIGURE 3-1

RECOMMENDED SAMPLING APPROACH



3.5.1 Municipal Wastewater Treatment Plant Effluent

The known diurnal variation in municipal wastewater treatment plant discharges requires flow proportionate sampling over a 24-h period. A minimum of three grab samples shall be combined in volumes proportional to flow to produce a composite approaching a representative sample of the 24-h discharge. If prior knowledge of the weekly discharge characteristics suggests variation in flow according to day of the week, three days of sampling (one flow proportionate composite per day) is recommended.

3.5.2 Process, Combined, and Cooling Water Effluents

A minimum of three manual grabs shall be combined to form a single composite sample representative of the operating day or shift. The sample should be obtained as a flow proportionate composite by collecting grab samples at equal time intervals over the sampling period and combining the grab samples in volumes proportional to flow. There are two circumstances under which flow proportionate composite samples would not be taken:

- flow data is not available and direct flow measurement is not possible; and
- the effluent discharges 24 h per day with instantaneous flow variations less than $\pm 15\%$ of the daily average flow.

In these situations, a time proportionate sample may be obtained by collecting and combining equal volume sub-samples at equal time intervals over the sampling period. In either case (flow or time proportional sampling), the time interval between grab samples should be spaced as follows:

- minimum 6 to 8 hours apart over a 24 hour operating day, or
- at equally spaced intervals over the operating shift (if different from 24 hours), but at least 2 hours apart.

Knowledge of site specific circumstances should be used to optimize the sampling frequency for a particular discharge. For example, the incidence of washdown events should trigger time-specific sampling to capture the effect of such flows, if the timing of these events is predictable. Where substantial day to day variation in effluent characteristics is suspected (**eg.**, batch operations), three days of sampling (one composite per day) should be conducted to obtain a representative picture of effluent variability.

In addition, where the budget allows, three sample days at each site is recommended to assess day-to-day variability.

3.5.3 Storm Runoff and Sanitary Effluent

Storm runoff (due to its intermittent nature) and industrial plant sanitary effluent (due to low volume and fairly consistent quality) can be sampled as single grab samples. It will be necessary to sample storm/site runoff when there is sufficient flow, which is usually during or immediately after a rain event. Flexibility is required for sites where stormwater is the major concern (wood preservative plants, lumber mills, concrete plants). These sites should only be sampled when there is sufficient rain to create flowing runoff.

3.6 **Arrange Analyses, Assemble Sample Bottles and Preservatives**

Based on the type of industry and number of discharges to be sampled, determine the types and numbers of analyses required (refer to Chapter 2 for industry specific parameters.) To arrange for the analyses, contact an analytical laboratory capable of performing the required analyses in accordance with the methods outlined in Section 6 (Table 6-1). The laboratory should be contacted as early as possible to ensure that they can provide the required turn around time for the analyses.

Using the information on industry type and required number of samples, determine the number of sample bottles that are required for the parameter list appropriate to the industry sector being tested. Don't forget bottles for quality control samples (see Chapter 6). Assemble at least half a dozen extra bottles for each parameter group to be used in case breakage occurs or some unusual site conditions make it desirable to collect extra samples.

Table 3-2 lists the types of sample bottles required and preservatives necessary for the parameters of concern. It also indicates parameters that can be analyzed from the same sample bottle.

In most cases, the laboratory performing the analysis will provide precleaned sample bottles and preservatives. Early contact with the laboratory will ensure that they can prepare and deliver the bottles on time. The sample volume requirements often depend upon the laboratory performing the analysis. Therefore, the requirements listed in Table 3-2 should be confirmed when ordering the bottles.

Laboratory sample bottles should be proven clean before beginning the field program. Randomly select a representative container from each batch of containers that have been shipped or cleaned together. Rinse with preservative and reagent water, and analyze the rinsate for the parameter(s) of concern. For organic parameters proof one bottle per batch by rinsing with dichloromethane and analyzing the rinsate. Proofing should be done for each analytical test group under consideration. It maybe possible to arrange for the analytical laboratory to supply proofed containers accompanied by the cleaning protocols and rinsate data.

Table 3-2

Sample Bottles, Preservation, Handling and Storage Requirements for Fraser Basin Wastewater Characterization Program Parameters

PARAMETER	Analytical Test Group	Volume		Preservative/ Temperature	Container	Maximum	Special Precautions
		Preferred	Minimum			Storage (Days)	
Core Parameters							
Temperature	Test performed in field immediately upon sampling.						
Dissolved Oxygen	Test performed in field immediately upon sampling.						
Chlorine, Total Residual	Test performed in field immediately upon sampling.						
pH	Hydrogen Ion	100 mL	50 mL	4 °C	HDLPE	3	
Specific Conductance	Conductivity	500 mL	50 mL	4 °C	HDLPE	4	
Total Suspended Solids (TSS)	Total Suspended Solids	500 mL	200 mL	4 °C	HDLPE	7	
Alkalinity	Alkalinity	100 mL	100 mL	4 °C	HDLPE	3	
		Total	1 X 500 mL				
Dissolved Organic Carbon (DOC)	Organic Carbon	500 mL	100 mL	i) 4 °C	HDLPE	3	Storage times for acidified samples are 10 days for DOC, 28 days for COD
Chemical Oxygen Demand (COD)	Chemical Oxygen Demand	500 mL	100 mL	ii) 50% H2SO4 to pH <2	HDLPE	3	
		Total	1 X 500 mL				
Industry Specific Parameters							
Ammonia (NH3)	Ammonia	500 mL	150 mL	50 % H2SO4 to pH <2	HDLPE	10	
Nitrite (NO2)	Nitrite	250	50	4 °C	HDLPE	5	
		Total	1 X 500 mL				
Cyanide, SAD and WAD	Cyanide	1 Litre	500 mL	10 N NaOH to pH >12	HDLPE	7	Preservative must be placed in container prior to adding sample
Total Metals (see Table 7-1)	Metals	250 mL	100 mL	HNO3 to pH <2	HDLPE,	30	Additional sample containers are required for dissolved metals; dissolved metals samples require field filtration.
Arsenic	Hydrides	50 mL	50 mL	HNO3 to pH <2	Acid Rinsed		
		Total	1 X 500 mL				
Mercury	Mercury	500 mL	500 mL	3 mL 10% K2Cr2O7 & 3 mL conc. H2SO4 / 500mL	Glass, Teflon Cap Liner	30	

Also assemble enough coolers to contain all of the laboratory containers. An easy way to plan this is to arrange for the laboratory to package the sample bottles individually in the coolers for shipping. This ensures that all samples will fit into the available coolers and that each bottle will be secure. Coolers should be well insulated and provide enough extra room for chemical or natural ice packs. Arrange to have extra coolers and ice in which to store large containers (flasks or carboys) of composite samples during the collection period.

3.7 **Select Sampling Equipment**

Based on the site information obtained and the number of sampling points, prepare a checklist of all required equipment and supplies. Table 3-3 presents a suggested checklist. The list includes a variety of equipment which maybe needed to sample the different types of effluents and access points that may be encountered in the field. The list also includes equipment for mixing composite samples, cleaning equipment, and taking field measurements (eg., pH and dissolved oxygen meters, thermometers).

Acceptable equipment for manual sampling includes stainless steel pails, telescopic/fixed poles and/or peristaltic pumps. Variations may be needed for some site specific conditions. However, all sampling materials that come into contact with the sample should be made of stainless steel, glass or teflon. Occasional exceptions can be made for the sake of practicality, such as the use of short sections of surgical silicone tubing in peristaltic pumps, but these materials should be kept to a minimum.

The following paragraphs briefly describe the common types of sampling equipment and the circumstances for which they are most appropriate:

- Stainless steel pails are useful when a significant flow is present, large volumes of sample are required, or where the drop to the effluent stream is too far for poles or pumps. The pails are very rugged and are quite easy to clean. Stainless steel pails in various sizes are available from laboratory supply companies and specialty metal fabricators. Pails are usually lowered into the effluent stream on a rope. They can be used with any length of rope, but a 15m rope should be sufficient for most applications. A clasp should be tied on one end of the rope to allow easy attachment and removal of the rope from the pail handle.
- **Telescopic/fixed poles** are useful when it is desirable to take the samples directly into the laboratory containers (eg., when collecting single grab samples) or when the access to the effluent stream is restricted and will not allow a large sampling device such as a pail to pass. The bottles are clamped directly to the end of the pole. There are specialty poles that allow the sample container's cap to be removed in the effluent, which is useful for collecting

Table 3-3

General Sampling Checklist

Sampling Equipment

- stainless steel pail(s)
- 15m polypropylene or nylon rope with clasp
- fixed or telescopic pole with bottle clamps of various sizes
- battery operated peristaltic pump, plus:
 - sections of surgical grade silicone rubber tubing for pump; should use new section for each sampling site and each new sampling period
 - 3/8" or 1/2" polypropylene hose (for intake to peristaltic pump)
(sufficient length to accommodate sampling access)
 - use new sections for each sampling site and each new sampling period
- portable analytical equipment such as pH meters, dissolved oxygen meters (with membrane replacement kit), mercury thermometers in metal cases, chlorine Hach kit

Sample Bottles/Glassware

- sufficient number of bottles to take primary and QA/QC samples - bring extra bottles in case of breakage or a change in the scope of the program occurs (ie. extra samples are required)
- if time-proportionate sampling:
 - premark bottles in equal volumetric proportions
- if flow-proportionate sampling:
 - 100ml, 500ml, and 1000ml glass graduated cylinders -if compositing is to be carried out in the field
 - 5L or 10L glass bottles or carboys with teflon-lined lids for storing samples to be composited (actual carboy volume depends on minimum sample volume requirements for total group of parameters)
- glass funnels
- glass or stainless steel stir rods
- bottles should be packed separately, away from equipment and reagents

Preservation Supplies

- a complete set of preservation chemicals as required for the collected samples (include MSDS sheets)
- small insulated cooler or secure box to store preservatives
- plastic/glass disposable pipets or an autopipet tip for each type of liquid preservative
- pH papers - for measuring sample pH after preserving
- field filtration kit and plastic forceps (if sampling dissolved metals)

Cleaning Supplies

- powdered soap as recommended by lab suppliers for cleaning glassware (include MSDS sheet)
- distilled/deionized water for equipment cleaning
- bottle brushes (various sizes)
- pesticide-grade acetone or methanol (include MSDS sheet)
- container for waste solvent
- paper towelling (for wiping outside surfaces only)

Table 3-3

General Sampling Checklist

Storage Supplies

- sufficient insulated coolers to contain all sample bottles (including flasks\carboys used to prepare composite samples)
- chemical or natural ice packs
- resealable bags for handling natural ice
- thermometer for monitoring cooler temperatures
- bubble pack for cushioning glass sample bottles

Documentation

- field notebook or log sheets
- sample labels
- fine point indelible marker
- laboratory submission sheets

Safety Equipment

- non-talc latex disposable gloves (lab or medical grade)
 - safety footwear
 - safety glasses
 - hard hat
 - safety vest
 - hearing protection
- * Note that many industrial facilities will not let personnel on site without the proper safety equipment

The majority of this equipment is available from laboratory supply companies.

samples from mid-depth. The sampling poles are normally made from aluminum to reduce weight and increase the manoeuvrability of long extended poles. Pole lengths from 2m to 5m are fairly standard. Required lengths greater than 5m usually require custom fabrication. Most of these poles are available from laboratory supply companies, especially those specializing in water sampling equipment. A good source of telescoping poles is a pool supply company. The poles supplied for cleaning pools can be modified to accept a large gear clamp for holding lab containers.

- **Peristaltic pumps** are good for shallow effluents that will not allow pails or sample containers to be submerged. As a result of the pump design, no portion of the pump is in contact with the sample; the sample remains within the tubing (see Table 3-3 for acceptable tubing materials). Most pumps are small enough that they can easily be picked up and moved by one person. The drawbacks to a pump are that it requires a power source (although battery powered models are available) and its lift is limited to approximately 7 m. Peristaltic pumps are available from most laboratory supply companies,

3.8 **Prepare Sampling Equipment**

Clean and inspect all grab sampling equipment that is to be taken to the site. Table 3-4 provides specific procedures for cleaning field equipment. Wrap cleaned equipment and contact openings of pump hoses in solvent-washed aluminum foil for transportation to the field. Be sure to take extra aluminum foil to wrap/cover equipment in the field between sampling times.

3.9 **Prepare Documentation**

The following documentation must be available (and used) in the field:

- *Field Log Sheet*

A field notebook or log sheet should be prepared and taken to the field to record conditions and observations at the time of sampling. A list of the information that should be recorded is given in Table 3-5.

- *Sample Bottle Labels*

All samples must be properly labelled. Appropriate blank labels can be obtained from the laboratory performing the analysis. Sample labels can be partially prepared beforehand using an indelible (waterproof) marker. This helps to create legible labels. The following label information is required:

Table 3-4

Cleaning Procedure for Field Equipment

The following procedures are to be used in the laboratory prior to departure and in the field between sampling locations (different effluent streams or different industrial sites)

- | | |
|---------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Step 1 | <ul style="list-style-type: none">• Using bottle brushes scrub all sampling equipment with hot soapy water. Pay particular attention to areas that will contact the sample or effluent stream. Soap should be laboratory-grade detergent, phosphate free if possible. |
| Step 2 | <ul style="list-style-type: none">• If piped water is available, rinse the equipment 2 or 3 times with normal tap water. Ensure that no soap remains on the equipment. Soapy water should be dumped down a sanitary drain where available or on the ground well away from the sample point. |
| Step 3 | <ul style="list-style-type: none">• Rinse equipment with ultrapure (distilled/deionized) water 2 or 3 times. Water should be dumped down a sanitary drain where available or on the ground well away from the sample point. |
| Step 4 | <ul style="list-style-type: none">• Rinse all metal and glass equipment with pesticide-grade acetone. Collect the waste solvent in a container for removal from the sampling site. |
| Step 5 | <ul style="list-style-type: none">• It is important to let all equipment air dry in a contaminant-free environment before reusing, to eliminate traces of solvent. While drying, keep equipment sheltered from dust, precipitation, etc. Equipment should be covered with solvent washed aluminum foil between uses. |

Table 3-5

Example Field Log Sheet

Complete 1 form for each sampling location

Plant Name:

Sample Start (date, time):

Sampling Location:

Sample End (date, time):

1. Describe sampling point: (attach site sketch)

2. Type of Effluent : (e.g. process, combined, cooling water, runoff, sanitary)

3. Personnel:

Samplers:

Plant Personnel:

4. Sampling Technique:

Single Grab Parameters:

Interval #'s:

Manual Composite:

No of grabs: _____ **Time based:** ☐ **Flow based:** ☐

Other:

5. Equipment used: Stainless bucket:

Make and model #:

Telescopic pole:

Peristaltic pump:

Other:

6. Flow Measurement:

Interval #

Time

Flow (units)

Instrument used:

Table 3-5
Example Field Log Sheet

7. Field Measurements:

Parameter	Time	Measurement (units)
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Instruments used:

8. Site Observations:

Effluent description (colour, odour):

Weather:

9. Plant/Process Considerations:

10. Samples Submitted: (attach copy of Laboratory Submission Sheet)

11. Other Comments?

- Plant name and sampling location (or code);
- Date and time of sampling (start/end for composite samples);
- Analytical parameters;
- Preservatives used (if any);
- Sampling personnel; and
- Effluent flow rate (if applicable).

Note that some of this information will need to be added in the field. Be sure to include indelible markers in the field kit.

- *Laboratory Submission Forms*

A laboratory submission form must be prepared and submitted along with the samples to the laboratory performing the analysis. Ensure that forms are available in the field.

3.10 Make Final Phone Call to Plant Personnel

Prior to departing for the field, phone the onsite contact to confirm your sampling plans and arrival time and to ensure that the intended sample stream is accessible. You should also confirm that the plant is discharging and determine whether there are any process upsets or other unusual circumstances that would cause the samples collected to be unrepresentative of typical wastewater quality.

FIELD SAMPLING PROTOCOLS

This chapter presents detailed field methods for collecting the samples for chemical analysis in the Wastewater Characterization Program. Protocols for preparation of composite samples are presented in Section 4.4. Section 4.9 presents measures for dealing with some problems commonly encountered in the field.

4.1 Selection of Sampling Point

Final selection of the sampling site must be made in the field where it is possible to observe the effluent flow and confirm the locations of all contributing streams. If possible, a preliminary site visit to determine sampling locations and assess equipment needs is recommended.

Samples should be collected from a discharge pipe, conduit or ditch that is flowing at the time of sampling, since this will be representative of the quality of effluent discharged to the receiving water body. Choose the actual sampling point based on the following selection criteria:

- a) If the industry currently collects monitoring data, the sampling location should be the same point that is used for routine or regulatory monitoring.
- b) If the facility does **not** currently monitor effluent quality, choose a point that is:
 - downstream of all contributing streams;
 - downstream of the chlorination/dechlorination contact **tanks**, if chlorination is being used (**eg.**, sewage or fish processing plant effluent);
 - sufficient distance from the final contributor to allow for good mixing;
 - not influenced by the receiving water body;
 - of sufficient velocity to suspend solids; and
 - safe to access day or night.

The exact location should be documented with a written description of the location and a site sketch showing the sampling point.

4.2 Final Preparation of Sample Bottles

The appropriate label should be added to each laboratory bottle prior to the introduction of the sample. Labels will stick better to warm and dry bottles,

especially glass ones. Therefore, if labels have not already been attached to the bottles, put them on prior to collecting single grab samples or before beginning to fill the bottles with composite samples. Make sure you have the correct label or the correctly labelled bottle for the sample you are collecting. Add relevant information, such as sampling time, to the label.

4.3 **Collection of Samples**

The appropriate method for collecting grab samples (steel bucket, pole, or peristaltic pump) depends upon the outfall structure. Table 4-1 summarizes protocols for collecting samples from different types of outfalls. These methods are appropriate for collecting either single samples or grab samples that will be combined to make composite samples,

Special precautions are necessary for sampling volatile organics. Samples for volatile organics must be collected as single grabs (not composite). The samples should be handled as follows:

- Wherever possible, collect effluent directly into the appropriate laboratory container (40mL vial).
- Slightly overfill sample vials.
- Allow any air bubbles to escape.
- Replace the cap tightly allowing excess sample to squeeze out, so that no headspace is present.
- Invert and tap bottle to observe air bubbles. If any are present, discard and repeat sample.

Specific precautions for handling other parameters are given in Table 3-2.

Samples should be kept above freezing and under 10°C (preferably at 4°C) in a dark environment until the sampling is completed and they have been transferred to the analytical laboratory. They should never be stored in direct sunlight, even for short periods of time. To provide the proper storage conditions, place the samples on ice in coolers as soon as they are collected. Either chemical or natural ice packs can be used. Natural ice packs are preferred as they are easier to replenish. To keep ice contained and the insides of the coolers dry, natural ice should be stored in resealable bags. This also allows for some flexibility in the placement of the ice packs in the coolers.

Table 4-1
Instructions for Manual Grab Sample Collection

Method 1	Method 2	Method 3
<i>Open channel, easy access</i> <i>Suitable for all parameters</i> <i>Requires sufficient depth in channel</i>	<i>Difficult to reach streams, limited access</i> <i>Suitable for all parameters that are not prepreserved</i> <i>Preferred for volatile organics</i> <i>Suitable for low flows</i>	<i>Restricted access, up to 7 meters below surface</i> <i>Not recommended for volatiles</i> <i>Suitable for very low flows</i>
<i>Stainless Steel Pail</i>	<i>Fixed or Telescopic Pole</i>	<i>Peristaltic Pump</i>
<ul style="list-style-type: none"> - Secure rope to pail handle - Drop pail into effluent stream and fill - Retrieve pail - Swirl sample in pail then discard back into effluent stream - Retrieve and discard second sample - Drop pail back into effluent stream, sink below surface to mid-depth - Retrieve sample - Mix gently using a glass or stainless steel rod to suspend solids and transfer sample as indicated in (A),(B) or (C) below - If taking a volatile sample by this method, submerge bottle directly in bucket, and follow instructions for minimizing headspace - Resample stream for remaining parameters as needed 	<ul style="list-style-type: none"> - If using telescopic pole, fix pole to desired length - Clamp sample bottle to pole: glass collection bottles can be reused if making composite, use laboratory bottles for volatiles or single grabs - Submerge bottle below surface to middle of effluent stream - Retrieve bottle - Discard collected sample - Retrieve and discard second sample - Resubmerge bottle below surface of effluent stream - Follow (A), (B), or (C) below - Repeat process, minimizing time between sub-samples 	<ul style="list-style-type: none"> - Place a new section of silicone tubing in the pump - Attach length of polypropylene hose to pump with a weight and strainer on end - Place weighted end in effluent stream, with sample intake at mid-depth - Add additional length of polypropylene hose to outlet to direct sample into lab containers or large glass collection vessel - Run the pump and discharge back into effluent for about one minute to rinse hose - Direct pump into bottles or vessel, as in (A), (B), or (C) below.

- | | |
|---------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| (A) For Single Grab: | <ul style="list-style-type: none"> - Pour sample directly into laboratory bottles as appropriate. - or into a large precleaned glass pouring cup if retrieval equipment is too large to easily pour into bottles. Preserve sample bottles as necessary. |
| (B) For Flow Proportioned Composite: | <ul style="list-style-type: none"> - Pour sufficient sample volume into a large glass container - Store in dark at 4 °C - Follow compositing procedures in Table 4-2 |
| (C) For Time Proportioned Composite: | <ul style="list-style-type: none"> - Partially fill laboratory bottles as specified in Table 4-3 |

Precautions:

- Always wear disposable gloves when handling effluent samples.
- Avoid touching the lips of sample bottles or the insides of caps.

The temperature can be monitored by placing a thermometer in the cooler. The thermometer should be protected from breakage. Non-mercury (alcohol) thermometers are recommended for use in coolers.

4.4 Preparation of Composite Samples from Manual Grabs

To obtain a composite sample representative of one operating day or shift, the individual grab samples shall be combined to form one composite sample for laboratory submission (for all parameters except volatile). The criteria for deciding whether to obtain a time-proportionate or flow-proportionate composite are described in Section 3.5. This section presents instructions for the preparation of either type of composite sample from a series of grab samples.

In most cases, composite samples will be prepared by combining grab samples in quantities proportional to the flows at the times of sample collection. Flow proportionality can be based on instantaneous flow readings at the time of sampling or on average flow over an interval that includes the sampling event. Table 4-2 indicates the steps taken to prepare a flow proportionate composite sample.

In some cases, composite samples will be prepared by combining grab samples of equal volumes. Table 4-3 indicates the steps taken to prepare a time-proportionate composite sample.

Tables 4-2 and 4-3 also give methods for handling quality control samples, where required. Chapter 5 provides guidelines for determining which quality control samples should be taken.

4.5 Sample Preservation

After the composites have been prepared (or as soon as single grabs have been collected), samples for certain parameters must be preserved. Sample preservation requirements for specific parameters were presented in Table 3-2.

Chemical preservatives can be corrosive or reactive and must be handled with extreme caution.

Where necessary, preservatives should be added as follows:

- For most parameters, preserve samples only when the laboratory bottles are full (not while portions of the composite are still to be collected).
- Preserve samples for cyanide analyses as each portion of the composite is collected.

Table 4-2 Instructions for Preparation of Flow-Proportionate Composite Samples		
<i>Primary S a m p l e</i>	<i>QC Samples (as required)*</i>	
	<i>Duplicate</i>	<i>Trip Blank</i>
<p>Collect all aliquots from one grab interval into a large glass flask or carboy; cap with teflon-lined lid. Repeat using separate carboy for each grab interval. Obtain the flow rate corresponding to each grab sample. Determine weighted sub-sample volumes per grab sample (see example below) - weighted volume must be worked out for each lab bottle size Align lab bottles for easy liquid transfer and remove caps. Mix sample thoroughly as indicated in Table 4-3. Measure out the calculated volume requirements using a graduated cylinder. Pour into appropriate lab bottles. Repeat for each grab sample, mixing carboy contents before each set of measurements. Add preservatives as specified for parameters (See Table 3-2). Cap lab bottles, shake to mix, and store in coolers.</p> <p>Note: If flow proportioning is done by the analytical laboratory, flow rate must be included on the label. The individual grab samples to be used for the composite must be clearly identified.</p>	<p>- Prepare exactly as primary sample.</p> <p>- Align bottles</p> <p>- Pour portion of sample into duplicate bottle for same parameter. - Add sample preservatives as for primary sample. - Cap and store</p>	<p>- Open'Blanks</p> <p>- Add sample preservatives as for primary sample. - Cap and store</p> <p>- Repeat for each sub-sample</p>
<p>- Cap all bottles securely and store in coolers at 4°C. Care should be taken not to leave samples exposed to heat. - Complete field log sheet and document all QC samples collected. - Complete sample submission form and pack in plastic bag with sample bottles.</p>		

* **Specific QC** requirements for the **parameters of concern** are given in **Chapter 6.0**

Example of calculation for **determining** flow-weighted volumes:

Sample **size** needed for lab **submission** = 1000 ML

#of sample portions per composite = 3

Interval 1 flow rate= 100 **litres/minute**

Interval 2 flow rate= 250 **litres/minute**

Interval 3 flow rate= 12.5 **litres/minute**

Total flow measured during sampling = 475 Mm/minute

Sub-sample 1 volume. $100/475 \bullet 1000$ mL. 211 **mL**

Sub-sample 2 volume= $250/475 \bullet 1000$ mL = 526 **mL**

Sub-sample 3 volume= $125/475 \bullet 1000$ mL = 263 **mL**

General Information:

- Always wear **disposable** gloves when handling effluent **samples**.
- **Collect** Manual Grab samples as indicated in Table 4-1.
- Flow-proportioning should be done in the laboratory wherever possible.

Table 4-3

Instructions for Preparation of Time-Based Composite Samples

<i>Primary Sample</i>	<i>QC Samples (as required)*</i>	
	<i>Duplicate</i>	<i>Trip Blank</i>
<p>Mark lab bottles into equal volumetric portions based on the number of samples be collected for each composite (i.e. 3-part composite will require that the bottles be marked off in thirds) Bottles can be marked with a thick indelible marker or with small strips of masking tape</p> <p>Align lab bottles for easy liquid transfer and remove caps</p> <p>Swirl sample container or stir with acetone-rinsed glass rod; disrupt rotation of body of liquid to prevent solids from being centrifuged toward walls of flask; pour portion of well mixed sample into laboratory bottle and fill up to marker</p> <p>Cap bottles and store in coolers until next sample portion is collected</p> <p>Repeat above steps for each sub-sample of composite</p> <p>Add sample preservatives as specified for parameter (See Table 3-2)</p>	<p>- Prepare exactly as primary sample</p> <p>- Align bottles</p> <p>- Pour portion of well mixed sample into duplicate bottle for same parameter</p> <p>- Cap and store</p> <p>- Repeat for each sub-sample</p> <p>- Add sample preservatives as for primary sample</p>	<p>...</p> <p>- Open Blanks</p> <p>.....</p> <p>- Cap and store</p> <p>- Repeat for each sub-sample</p> <p>- Add sample preservatives as for primary sample</p>
<p>- Cap all bottles securely and store in coolers at 4°C. Care should be taken not to leave samples exposed to heat</p> <p>- Complete field log sheet and document all QC samples collected</p> <p>- Complete sample submission form and pack in plastic bag with sample bottles</p>		

• Specific **QA/QC** requirements for the parameters of concern are given in Chapter 6.0

General Information:

- Bottles should be **pre-marked** before shipment to the field
- Collect Manual Grab samples as indicated in Table 3-7
- Always wear disposable gloves when handling effluent samples

- Preserve samples for dissolved metals analyses only after filtering the samples.
- Use glass/plastic disposable pipettes or automatic dispensing pipettes.
- Use a different disposable pipette or pipette tip for each type of preservative solution.
- Do not allow the dispensers to make contact with the sample. If contact inadvertently occurs, dispose of pipette and use a new one, if preserving a series of samples from several different effluent streams.
- Add preservatives carefully, a few drops at a time.
- Then cap the sample and mix by inverting the bottle.
- Measure pH with pH paper, and continue adding preservative until the desired pH is reached.
- If a precipitate forms during addition of the preservative, note it on the field log sheet.
- Never composite preserved samples with unpreserved samples or samples containing different types of preservatives.

Samples for dissolved metals should be filtered within a few hours of collection. Filtration should be done in a clean, dust-free area, if possible. When the sample site is in the same city as the analytical- laboratory, it may be possible to deliver the unpreserved sample to the laboratory for filtration. Be sure to confirm this option with laboratory personnel prior to leaving for the site.

Small, manually-induced vacuum filtration kits are available for field use. Some laboratories may supply them.

Instructions for filtering metals samples are as follows:

- Rinse the filtration equipment with distilled/deionized water prior to use and between samples.
- Use a clean (unused) 0.45- μm filter for each sample. If the effluent contains a large amount of suspended material, it may be necessary to change the filter several times for each sample.
- Handle the filter with plastic forceps. Never let your hands touch the filter or any part of the filtration equipment that will contact the sample.

- Pass a small portion of the sample through the filter, swirl in the catch flask, and discard. Then filter the remaining sample.
- Transfer the filtrate to a sample bottle and preserve as indicated in Table 3-2.
- For quality control always collect a rinsate sample (filtration blank) prior to filtering the first sample. When filtering several samples with the same equipment, collect another rinsate after all the samples have been filtered.

Pack samples in a cooler for storage/transportation as soon as they have been preserved.

4.6 Equipment Cleaning in the Field ,

It is extremely important to clean all sampling equipment properly between individual samples to minimize cross-contamination. Table 3-4 presents acceptable procedures for cleaning sampling equipment between uses. ‘

Equipment consisting of relatively inert materials such as stainless steel, glass, and teflon is reasonably easy to clean. Other types of plastics such as polypropylene are easily scratched and adsorb certain organics. This makes them difficult to clean, and these materials should be replaced between sampling sites or periods.

Whenever possible, field cleaning should be minimized by providing dedicated sampling equipment for each site for the duration of the sampling trip. ‘

4.7 Field Measurements

Temperature, dissolved oxygen, pH, and, where applicable, residual chlorine should ‘ be measured in the field. Temperature and dissolved oxygen measurements should be taken each time a sample is collected. Measuring these parameters in composite samples is not appropriate. The pH of both individual and composites samples should be measured.

Measure temperature, dissolved oxygen, and pH from a separate aliquot that will not be added to any composite sample or submitted to the laboratory for analysis. Discard the aliquot after completing the measurements.

Measure temperature in a sample that has been collected in or transferred to a glass or plastic container. Use a mercury-filled Celsius thermometer or an alcohol-filled thermometer that has been calibrated with a mercury thermometer. The thermometer should have a scale marked in 0.1°C increments.

Collect grab samples for dissolved oxygen very carefully to avoid aeration. Measure immediately with a field meter. Because physical shock during transportation can disturb the calibration, the meter should be calibrated prior to making the first measurement at each site. Follow the manufacturer's instructions for operating the meter. Allow the probe to stabilize to the sample temperature before taking the reading. Be aware that reactive gasses, such as chlorine and hydrogen sulphide, can interfere with the analysis or desensitize the probe.

Take pH measurements with a field meter, following the manufacturer's instructions. Agitate the probe constantly while making the measurement. Allow at least 30 seconds for the measurement to stabilize before recording the pH.

Measure chlorine with a field kit (Hach or similar), following the manufacturer's instructions for total residual chlorine measurements.

4.8 **Preparation of Samples for Laboratory Submission**

Samples should be packed securely in coolers with ice packs for transportation to the laboratory. They should be shipped as soon as possible after collection, preferably the same day.

Maximum holding times (the maximum times from sample collection to laboratory extraction or analysis) are given in Table 3-2. Inform the laboratory of any delay in transportation to minimize the chances of exceeding holding times. Do not rely on receiving personnel to note the collection date,

Label all coolers with the following information:

- name and address of analytical laboratory;
- name and phone number of laboratory contact;
- name and phone number of shipper; and
- piece number (ie., 1 of 4).

Each shipment should be accompanied by a laboratory transmittal form. Make duplicates and keep a copy. The form should list each sample bottle individually and indicate the parameters to be measured. The laboratory submission form can also be used to document the date, time, and ownership at the time of sample transfer, which may assist in locating any misplaced samples or coolers. Table 4-4 provides an example of a laboratory submission form.

Fraser Basin Wastewater Characterization Program

Sample Laboratory Submission Form

Sampled By: _____

Custody Relinquished by: _____ **Date/Time:** _____ **Submitted by:** _____
Received by: _____ **Date/Time:** _____ **Company:** _____
Sample Storage: _____ **Address:** _____
Composite Prepared By: _____ **Date/Time:** _____ **Phone No:** _____
Fax No. _____

Notes/Unusual Observations: _____

4.9 Common Sampling Problems

The following is a short discussion of common problems encountered in the field with some suggestions for avoiding or overcoming them.

4.9.1 Weather

The weather can adversely affect a sample monitoring program. Temperature is probably the main factor to consider. Temperatures above 4°C are not conducive to the storage of samples for analysis. In warm weather sample bottles should be kept in an insulated cooler with ice or chemical ice packs. If the temperature is below freezing, arrange to store samples inside a building at the plant, if possible (but in a cooler at 4°C).

4.9.2 Low Discharge

Small and medium sized industries often have non-processing times where they discharge little or no effluent. If it is necessary to obtain samples during these periods a number of difficulties may be encountered. Most manual sampling equipment is designed to be submerged in the effluent stream in order to obtain a sample. During extremely low flows this may not be possible. It is best to avoid these situations by discussing conditions with plant personnel prior to traveling to the site. If low discharge periods cannot be avoided, they should be well documented.

4.9.3 Safety Considerations

In all situations, it is recommended to send two field personnel to collect samples, or to have on-site personnel accompany the field staff, since unanticipated circumstances may arise that could pose a safety risk. Such conditions could include icy/slippery conditions or difficult to access effluents, among others.

In some industrial settings, it may be necessary to sample through a manhole or sewer access. In such cases it will be necessary to train staff in gas testing procedures and confined space entry safety precautions. Manholes should never be opened without gas testing, as explosive or hazardous fumes could be present. Worker's Compensation Board requirements must always be followed.

FLOW MEASUREMENT

5.1 Introduction

Since one of the objectives of the Wastewater Characterization Program is to determine contaminant loadings, it is important to know the rate of effluent flow over the sampling period. In addition, if samples are to be composite on a flow proportional basis (Section 3.5), then flow measurements are necessary to determine the appropriate proportions. Therefore, this chapter addresses methods of measuring effluent flow.

Given the range of industry and effluent types within the Fraser Basin, a wide range of effluent conveyance and outfall structures likely will be encountered. It is not possible for this document to provide precise instructions for measuring flow in all possible effluents. Rather, the document describes the different types of flow monitoring devices and approaches that are available and the situations in which each is appropriate.

5.2 Permanent Flow Monitoring Stations

Many industries in the Fraser Basin are required to monitor and report effluent discharge rates as part of their Waste Management Permit requirements. These sites are likely to have permanent flow monitoring installations, possibly with continuous recording devices.

Before starting the field monitoring program, determine whether onsite flow data or measurement equipment is available. Data collected with permanently-installed equipment and a continuous recorder are likely to be more accurate ~~than~~ any that could be collected during a single visit. Continuous flow data should be used, if available. If equipment is installed but data are not recorded continuously, it is still desirable to make flow measurements with onsite equipment (assuming that it is functioning properly).

However, in some cases use of onsite data or flow measurement systems may not be appropriate. For example, a weir or flume may be installed in the process effluent stream but be located upstream of the confluence of a stormwater conveyance. Therefore, the effluent flow data would not measure total discharge from the site.

5.3 Temporary Flow Measurement Methods

In situations where there is no permanent flow monitoring system, it may be possible to install a temporary flow monitoring device and/or obtain point measurements. Depending upon the effluent structure and the equipment on hand, discharge can be determined either by direct measurement or by estimation based on velocity. The following sections discuss some direct-discharge and velocity-area methods of estimating effluent flows.

Two basic types of effluent conveyance structures, open pipe and closed conduit, are likely to be encountered during the Wastewater Characterization Program. For the purposes of flow measurements, an open channel represents any conduit other than a full pipe. In most industrial settings, flow will be through an open ditch, culvert, or partially full pipe. Closed conduit (full pipe) discharges are likely to have flow monitoring equipment already installed. However, some types of flow monitoring equipment are suitable for monitoring full pipes, if required.

5.3.1 Direct-Discharge Methods

Direct discharge measurements include those in which the rate of effluent discharge is related to one or two easily-measured variables. The following paragraphs briefly discuss some devices and approaches for making direct-discharge measurements on effluents:

- **Palmer-Bowlus flume:** This device is a small, moveable control structure that is appropriate for open channel discharge measurements. It is commonly used to measure wastewater flows in sewers. The flume acts as a hydraulic control in which critical flow is developed (as the effluent backs up above the flume). The rate of discharge is then related to upstream depth. Metcalf and Eddy (1979) describe the calculation relating depth to discharge.
- **Weirs:** Like flumes, weirs are used to control flow in open channels. It may be possible to install a temporary weir in a ditch or other open effluent conveyance structure. The weir controls flow by forming a barrier over which the effluent is forced to flow. The flow rate is determined by measuring the head (the difference in elevation between the crest of the weir and the surface of the liquid in the channel). The effluent flow is calculated from a rating curve in which the flow rate is plotted against the observed head. It is possible to purchase V-notch weirs for which the ratings curve has been predetermined and the discharges corresponding to certain elevations have been marked on the weir itself.
- **Tracers:** Chemicals (such as **NaCl**) or radioactive tracers can be used in either open channel or closed conduit situations (assuming that upstream

access to the closed pipe is available). A known concentration of the chemical or radioactive substance is added continuously, at a constant rate, to the effluent stream. At a distance far enough downstream to ensure complete mixing, samples are collected, and the concentration of the tracer is measured in the laboratory. The flow can then be determined from a mass-balance equation. The usefulness of this approach is limited by the requirement for adding the tracer at a constant rate, which requires controlled dosing equipment, but tracers can be used without constant dosing for velocity discharge measurements (Section 5.3.2). However, due to perceived concerns about the use of radioactive substances, radioactive tracers are not recommended for use in the Fraser Basin Wastewater Characterization Program

- **Volumetric measurement** (the bucket and stopwatch method): Relatively small discharges at the end of a pipe or culvert can easily be measured with a stopwatch and graduated container (or container of known volume). A minimum of three measurements should be taken and averaged to determine the flow when using this method.
- **Computation:** Effluent discharge rate in an open channel can be computed based on the depth of flow, channel slope, and coefficient of roughness (Metcalf and Eddy 1979). This approach is applicable where it is possible to access the effluent channel to measure depth and slope. It is also necessary to select a value for the coefficient of roughness.' Thus, the resulting discharge rate is at best an approximation, whose accuracy depends upon the steadiness of flow at the time of observation and the precision with which the coefficient of roughness is assumed for the existing conditions.

5.3.2 Velocity-Area Measurements

Velocity-area measurement methods provide an indirect measure of effluent discharge rate. The measurement devices indicate the velocity of effluent travel, and the actual discharge is determined by multiplying this velocity by the cross sectional area of the effluent conveyance structure. The following paragraphs describe some equipment and techniques for determining flow velocity and converting the data to discharge rates:

- **Current meters:** Current meters (the same devices used for stream flow measurements) can be used to determine the velocities of effluents flowing in large sewers, open channels, or ditches. The use of current meters may be limited by accessibility of the channel, the presence of large amounts suspended matter (which can clog the meter), and very low flows (meters are available to measure velocities as low as 2.5 cm/s). Flow readings can be made in turbid effluents by using a meter equipped with a digital display or

a headset that provides an auditory signal. To convert velocity to discharge, it is necessary to measure the channel cross-sectional area at the measurement point.

- **Electromagnetic pressure transducers:** Portable electromagnetic flow monitors are available for field use. A sensor that contains both an electromagnetic velocity transducer and pressure transducer can be installed on the bottom (invert) of a pipe or channel. The sensor measures both velocity and fluid level. This information is used in combination with the pipe dimensions to determine flow volume. Therefore, the pipe must be measured before the sensor is installed. Since the velocity transducer is electromagnetic, the fluid must be conductive (not usually a limiting factor in effluents).
- **Ultrasonic flow monitors:** A portable, submersible ultrasonic velocity sensor accompanied by a portable data logger can be used to measure liquid velocity and depth in an open channel or partially filled pipe. The transducer is generally mounted at the bottom of the pipe or channel. The depth and velocity measurements are used with pipe dimensions to calculate flow volumes. This type of equipment will work in liquids that contain suspended solids or in slightly aerated liquids without suspended solids.
- **Ultrasonic flow monitors, non-contact:** Non-contact ultrasonic flow monitors may be useful in some situations, but the nature of the power supply and the installation requirements limit their application for most short-term monitoring. Non-contact ultrasonic sensors can be used in dirty (high suspended solids), viscous or corrosive effluents because the sensor is installed above the effluent (where it measures depth by the time it takes for a signal to deflect off the effluent and return to the sensor). These types of sensors can be used to measure flow where open channel control devices (flumes, weirs) are available. They also can be used to measure flow in closed systems where direct access to the conduit is not possible. Access to the outside of the conduit is necessary, as transducers are attached for the measurements. These units provide a digital measurement of fluid velocity, which can be used with conduit dimensions to calculate flow volume.
- **Tracers:** Chemical or radioactive tracers can be used to measure effluent flow velocity in open channels or in closed pipes, where the upstream and downstream ends of the pipe are accessible. Chemical or radioactive tracers are usually injected upstream of two control points. The time it takes for the tracer to pass the two points is measured, and the velocity is computed by dividing the distance between the two points by the travel time. If **NaCl** is used as a tracer, its passage can be measured with a conductivity meter, while radiotracers can be measured with radioactive counters. The usefulness of **NaCl** as a tracer may be limited in highly conductive effluents.

- **Dye tracers:** Dyes can be used as tracers in open channels where the passage of the dye is visible. Dye tracers have proved particularly useful in small-pipe sewers (Metcalf and Eddy 1979). Dyes work best where the effluent flow is practically steady and uniform. The dye is added at the upper end of the test section and the time of its arrival at the lower end is determined. The time of appearance and disappearance of the dye can be noted quite precisely by using a bright dye (such as eosin) and a bright plate suspended horizontally in the effluent channel at the lower end of the test section. The mean time between appearance and disappearance of the dye on the plate represents the average time of flow. Other suitable dyes include fluorescein, congo red, potassium permanganate, rhodamine B, and Pontacyl Brilliant Pink B. Prior to using dye tracers, it is important to inform the company and local government agencies.

5.4 Decisions Regarding Flow Monitoring Equipment

The appropriate flow monitoring equipment or technique will vary from site to site. Therefore, preparation for the site visit should include making decisions about the flow monitoring equipment to take into the field. The questions posed to onsite personnel (Section 3.2) should be detailed enough to identify the situations in which flow monitoring will be necessary and the type(s) of equipment most appropriate for the site conditions.

FIELD QUALITY ASSURANCE QUALITY CONTROL

6.1 Introduction

Before any sampling program is designed, it is essential to develop a Quality Assurance/Quality Control (QA/QC) program that will ensure all appropriate laboratory and field handling procedures are being followed. The QA/QC program can assist in minimizing, detecting, and correcting field sampling and laboratory errors that can affect data reliability.

A QA/QC program is successful when the data collected are consistent, and the level of the uncertainty is acceptable for the end use of the information. The QA/QC program establishes data quality objectives, which define the acceptable level of uncertainty for the data. It also defines corrective actions to be taken if the data quality fails to meet the objectives.

A field QA/QC program consists of the following components:

- use of clean, uncontaminated equipment and glassware;
- following proper and consistent sample collection methodologies (described in Chapter 4);
- collecting field quality control samples to measure data accuracy (by identifying contamination or confirming its absence);
- calibrating and maintaining of field meters;
- keeping adequate field notes (see Table 3-5); and
- training and evaluating field technicians.

This chapter describes the field quality control (QC) program for the Wastewater Characterization Program. It recommends types and numbers of QC samples. Where appropriate, it defines data quality objectives and identifies corrective actions.

6.2 Quality Control Samples

Typical QC samples include rinsates (equipment blanks), duplicates, and field (transportation) blanks. The quality control samples described in the following sections should be incorporated into the field program.

6.2.1 Equipment Proofing

Prior to beginning a field testing program, all sample bottles and reagents to be used should be proved free of contamination that may affect validity of results. Section 3.2 includes a description of bottle proofing. The laboratory providing the sampling containers and reagent may be able to provide the required information along with the sampling equipment. Reagents obtained from sources other than the analytical laboratory should also be proofed.

6.2.2 Field QC Samples

Field QC samples are collected to measure sampling variability and to determine whether any contamination was introduced during sample collection. They consist of field duplicates, traveling blanks, traveling spiked blanks, and equipment rinsates.

The number of samples and frequency of collection depend in part upon the project budget and the potential concerns associated with a particular effluent or particular parameters. Therefore, the final decision on which QC samples to include in a given sample set must be made by the FPAO in consultation with field staff. In making the decision, the following guidelines should be considered:

- Where the effluent is considered non-homogeneous, analysis of field duplicates is recommended.
- Where contamination at the site or during sample collection might be a problem, the analysis of traveling blanks is recommended.
- Where losses of the analytes of interest (due to degradation or handling and storage) must be considered, the analysis of traveling spiked blanks is recommended.

Table 6-1 indicates the types of field QC samples that generally apply to the analytical groups included in the Wastewater Characterization Program. Table 6-2 defines the different types of QC samples.

To determine the frequency and number of samples, the following guidelines should be used:

- A complete set of field QC samples (as indicated in Table 6-1) should be taken for 5% (one out of every twenty) of the samples collected. If fewer than 20 samples are collected during a full year of the program, a complete set of QC samples should be collected once per year.

Table 6-1
Appropriate Field QC Samples for Fraser Basin
Wastewater Characterization Parameters

PARAMETER	Field Duplicate	Traveling Blank	Spiked Blank
Core Parameters			
Temperature			
Dissolved Oxygen			
pH	✓		
Alkalinity	✓	✓	
Conductivity	✓		
Total Suspended Solids	✓	✓	
Dissolved Organic Carbon (DOC)	✓	✓	
Chemical Oxygen Demand (COD)	✓	✓	
Industry Specific Parameters			
Ammonia	✓	✓	
Nitrite	✓	✓	
Cyanide	✓	✓	
Metals	✓	✓	
Arsenic	✓	✓	
Mercury	✓	✓	
Chloroguaiacols	✓	✓	✓
Chlorocatechols	✓	✓	✓
Chlorophenols	✓	✓	✓
Nonylphenol	✓	✓	✓
Dioxins/Furans	✓	✓	
Polycyclic aromatic hydrocarbons	✓	✓	✓

Table 6-1
Appropriate Field QC Samples for Fraser Basin
Wastewater Characterization Parameters

PARAMETER	Field Duplicate	Traveling Blank	Spiked Blank
Trichlorobenzenes	✓	✓	✓
Tetrachlorobenzenes	✓	✓	✓
Pentachlorobenzene	✓	✓	✓
Hexachlorobenzene	✓	✓	✓
1,4-Dichlorobenzene	✓	✓	✓
1,2-Dichlorobenzene	✓	✓	✓
Tetrachloroethylene	✓	✓	✓
1,1,1-Trichloroethane	✓	✓	✓
1,2-Dichloroethane	✓	✓	✓
1,1,2,2-Tetrachloroethane	✓	✓	✓
Trichloroethylene	✓	✓	✓
Dichloromethane	✓	✓	✓
Chloroform	✓	✓	✓
Toluene	✓	✓	✓
Benzene	✓	✓	✓
Styrene	✓	✓	✓
Xylene	✓	✓	✓
Resin Acids	✓	✓	
AOX (Absorbable Organic Halides)	✓	✓	
DDAC	✓	✓	
DOC	✓	✓	
Biochemical Oxygen Demand (BOD5)	✓	✓	

Notes:

Where more than one parameter is analyzed from the same bottle (as shown by sample groupings)

QA/QC samples can also be handled as one sample.

Adapted from Ontario Ministry of the Environment MISA Monitoring program

TABLE 6-2

DEFINITIONS OF FIELD QUALITY CONTROL SAMPLE TYPES

Field Duplicate

Field duplicate samples are two separate samples from the same source sampled at essentially the same time and under the same conditions or in a manner that minimizes the differences between them.

Field duplicates are stored, prepared and analyzed using the same methodology. Equal volumes of sample should be collected into two identical containers and preserved and stored in an identical manner.

Duplicates can be submitted to the laboratory without the knowledge that they are QC samples. These “blind” duplicates can eliminate any potential biases during laboratory analysis.

Analytical results of field duplicates illustrate the degree of sampling error, and to some degree, homogeneity of the sample source.

Traveling Blank

A traveling blank is a prepared sample of distilled or deionized, analyte-free water that is placed in a container identical to that of the sample. The analysis of a traveling blank may provide an indication of problems caused by sample handling, containers, sample contamination due to contaminants or particulate in the atmosphere and any other contaminants introduced during shipping or storage.

A traveling blank is stored, prepared and analyzed using the same methods used for the effluent sample. The blank is prepared by the laboratory and remains sealed as it is transported to the sampling location. The blank sample is opened during sample collection, preserved if appropriate, and then returned to the laboratory for analysis.

Traveling Spiked Blanks

A traveling spiked blank is a sample of deionized/distilled water to which a known quantity of the analyte of interest has been added.

A traveling spiked blank may provide an indication of the degree of degradation or loss of target parameters from the time of sampling to the time of analysis.

The spiked blank is transported to the sampling point and then returned unopened to the laboratory for analysis. The traveling spike should accompany the sampling containers to the site within 24 hours from the time it was prepared.

- A set of duplicate samples (for specific parameters of concern or interest) should be collected once during every field trip.
- Duplicates should also be taken at any time field personnel suspect the effluent or sample composite is significantly non-homogenous.

Duplicate measurements should always be included for parameters measured with field meters. The recommended number of duplicates and data quality objectives (where established) are as follows:

- Dissolved oxygen measurements should also be taken at least once per site. The repeatability of the measurements should be 0.1 **mg/L**.
- Duplicate pH measurements should be made at least once at each site.

6.2.3 Field Rinsates

Rinsates (equipment blanks) can be used to proof collection equipment that is used at more than one site. At least one rinsate for the parameter(s) of greatest concern should be included in any sample set where transfer of contaminants among sites is a possibility. Equipment is proofed in a manner similar to that described for sample containers. Rinsates should also be used when field-filtering for dissolved metals (see Section- 4.5).

6.2.4 Data Quality Objectives

The quality control data should be evaluated using the following data quality objectives, and followed up with the appropriate corrective action, if indicated:

- For field blanks and rinsates, the concentrations of all parameters should be less than the method detection limit or less than 10% of the levels in the samples. When the data fail to meet this objective, the field collection and/or cleaning procedures should be scrutinized, and inadequacies should be corrected. If the samples have been preserved, the preservative should be tested for contamination, if it has not already been proofed (see Section 6.2.1).
- There are-no firm objectives for differences among field duplicates, which will depend in part upon the variability of the effluent. However, a good rule of thumb is to investigate the causes of differences between duplicates greater than 50%. (This percentage can be relaxed for parameters having concentrations near the analytical detection limit.) The investigation should focus on the method used to prepare composite samples and potential sources of non-representative composite samples. For grab samples, sources of

contamination or non-representative samples should be considered, but differences could reflect actual variability in the effluent.

6.3 Calibration and Maintenance of Field Equipment

6.3.1 Field Thermometers

Field thermometers should always be packed carefully for transportation. Ideally, thermometers should be placed in individual metal cases. To the extent possible, they should be kept away from samples, sample containers, and other equipment that will contact the samples. If a mercury thermometer breaks, the mercury vapour can contaminate samples or bottles. To remove the risk of mercury contamination, an alternative is to use alcohol-filled thermometers that have been calibrated against a mercury thermometer.

6.3.2 Dissolved Oxygen Meters

As noted in Section 4.7, the dissolved oxygen meter should be calibrated prior to taking the first measurement at each site. The air calibration method is acceptable and is the easiest method to perform in the field. However, the overall error will be lower if the instrument can be calibrated at a temperature similar to that of the effluent. A water calibration at the effluent's temperature should be used, if possible, when the temperature of the effluent is substantially different from that of the air.

Dissolved oxygen meters should receive preventive maintenance according to the manufacturer's instructions. Maintenance of the probe is most important. It should always be stored in a humid environment to prevent the membrane from drying out. Prior to departing for the field, the membrane should be checked for dryness or oxygen bubbles, and replaced, if necessary. The machine should also be checked, and the membrane should be changed if it does not seem to be functioning properly.

If changing the membrane does not correct the apparent malfunction, consult the manufacturer's trouble shooting guide. If the problem cannot be corrected, have the meter repaired promptly by a qualified technician.

6.3.3 pH Meters

Field pH meters should be calibrated according the manufacturer's specifications. Measurements of pH are reliable only if the instrument has been calibrated with standard buffers that bracket the pH range of the effluent. The calibration should be done at the beginning of each set of pH measurements.

If the pH meter does not appear to be functioning properly, consult the manufacturer's trouble-shooting guide. Check for common problems such as a dirty electrode,

inadequate stirring, or inadequate solution in the internal reference portion of the electrode. If the meter still does not function properly after a simple adjustment, have it repaired promptly by a qualified service technician.

6.3.4 Field Chlorine Kit

The field calorimetry measurement for chlorine must be calibrated against a colour and turbidity blank. The wastewater itself should be used for this blank. No test reagent is added to the wastewater blank; thus, the only colour in the blank is that of the wastewater itself.

6.4 Technician Training and Evaluation

All field technicians should be thoroughly trained in sample collection methods, with emphasis on precautions for preventing sample contamination. The supervisor or project manager should review all field QC results. If field blanks reveal contamination, the supervisor should review the field protocols with the technicians and observe their sampling techniques. Similarly, if the correspondence between field duplicates is poor, the supervisor should observe the technicians' methods of preparing composite samples and provide additional training, if warranted.

ANALYTICAL METHODS

7.1 Rationale

Consistent analytical methods are necessary to ensure consistent and comparable results from different wastewater characterization studies. This chapter presents preferred and alternate methods for analyzing the parameters indicated in Chapter 2. Requiring that all laboratories analyzing samples for the Fraser Basin Wastewater Characterization Program use these methods will help to ensure that data obtained at different sites by different agencies and/or contractors are comparable.

7.2 Method Selection

The sample preparation and analytical methods were chosen based on common practice and standard methodologies employed by government and commercial laboratories throughout North America. Where alternate methods are cited, the most commonly used method, which normally has the most extensive available method performance data, was chosen as the recommended method. Alternate methods, which can be used to produce accurate analysis, are available for some parameters, but usually they are less commonly used and have less supporting performance data. These methods have been given secondary (alternate) status.

The availability of acceptable method performance data was the primary criterion used to select preferred and alternate measurement procedures. A second criterion for method selection (which was especially important for organics analyses) was the degree of data confidence obtained by using one method or another. For example, gas chromatography/mass spectrometry (GC/MS) provides a more certain detection of organic compounds than gas chromatography using other less specific detectors. Therefore, GC/MS was selected as the preferred analytical method.

A third selection criterion was detection limit. A method is acceptable only if it can provide the detection limit required for the intended data use. For example, high resolution mass spectrometry rather than low resolution mass spectrometry was selected for dioxin/furan analysis because extremely low levels of these very toxic substances can impact the aquatic environment.

7.3 Preferred and Alternate Methods

Table 7-1 lists the preferred and alternate analytical methods for the Fraser Basin Wastewater Characterization Program. It summarizes the recommended sample preparation and instrument measurement principles and also indicates the method detection limits.

The detection limits listed in Table 7-1 are those that are easily achievable using modern analytical instrumentation. For very complex effluents containing high concentrations of potentially interfering substances, it may not be possible to achieve the detection limits indicated.

In interpreting the data, one must be aware that data at the detection limit are only accurate to $\pm 100\%$. The limit of quantitation is defined as three times the method detection limit. At the limit of quantitation the accuracy of the data is expected to be $\pm 30\%$.

TABLE 7-1

ANALYSIS PRINCIPLES AND METHOD DETECTION LIMITS FOR WASTEWATER CHARACTERIZATION IN THE FRASER BASIN

Analytical Test Group	Parameter	Sample Preparation Method Principles	Instrumental Measurement Method Principles	Reference Method	Analytical Detection Limit (mg/L)
Hydrogen Ion	pH	Field and laboratory measurement	Recommended: pH electrode and pH meter	EC Vers.1.1/88 SM18(4500B)	N/A
Alkalinity	Alkalinity	Preparation for measurement system as appropriate.	Potentiometric titration	EC Vers.1.2/87 SM18(2320B)	5
Conductivity	Specific Conductance	Preparation for measurement system as appropriate.	Recommended: Conductivity meter and cell. Note: Measurement made at 25° C using thermostat or jacketed cell.	EC Vers.2.0/90 SM18(2510B)	5 uS/cm
Total Suspended Solids	Total Suspended Solids (TSS)	Preparation for measurement system as appropriate by: Filtration using a glass fibre filter with particle retention equal to 1 micrometer.	Recommended: Drying of filter and particulates at 103±3°C and gravimetry.	EC Vers.1.2/91 SM18(2540D)	10
Dissolved Organic Carbon	Dissolved Organic Carbon (DOC)	Preparation for measurement system as appropriate by: Filtration using a glass fibre filter with particle retention equal to 1 micrometer. Or analysis of supernatant of a settled sample where volatile/purgeable organics may represent a major portion of the DOC.	Recommended: Quantitative conversion of carbon to carbon dioxide by : (i) ultraviolet persulfate digestion; or (ii) combustion with a catalyst (temperature is catalyst dependent); or (iii) combustion at >1100°C Followed by infrared or colourimetric detection DOC may be determined directly using a sample free of inorg. carbon or as the difference between total carbon and inorganic carbon.	EC Vers.2.0/93 SM18 (5310)	5

TABLE 7-1

ANALYSIS PRINCIPLES AND METHOD DETECTION LIMITS FOR WASTEWATER CHARACTERIZATION IN THE FRASER BASIN

Analytical Test Group	Parameter	Sample Preparation Method Principles	Instrumental Measurement Method Principles	Reference Method	Analytical Detection Limit (mg/L)
Chemical Oxygen Demand	Chemical Oxygen Demand (COD)	Preparation for measurement system as appropriate by: Recommended: Reflux in dichromate Alternate: Oven digestion at 150°C in the presence of oxidizing reagents.	Recommended: Back titration of colourimetric measurement of trivalent chromium (CrIII)	BC MOE SM18(5220)	10
Ammonia	Ammonia plus Ammonium	Preparation for measurement system as appropriate	Recommended: Colourimetry or Specific Ion Electrode or Titration or Ion Chromatography	EC Vers.1.2/84 SM(4500H)	0.005 as nitrogen
Nitrite	Nitrite	Preparation for measurement system as appropriate.	Recommended: Colourimetry or Ion Chromatography	EC Vers.1.2/84	0.005 as nitrogen
Cyanide	Strong Acid Dissociable Cyanide (SAD)	Acid Distillation	Recommended: Colourimetry or Ion Chromatography or Specific Ion Electrode or Titration. Alternate: Polarography via the method of standard addition in the presence of suitable electrolyte.	EC Vers.2.0/84 SM18(4500C)	0.01

TABLE 7-1

ANALYSIS PRINCIPLES AND METHOD DETECTION LIMITS FOR WASTEWATER CHARACTERIZATION IN THE FRASER BASIN

Analytical Test Group	Parameter	Sample Preparation Method Principles	Instrumental Measurement Method Principles	Reference Method	Analytical Detection Limit (mg/L)
Cyanide	Weak Acid Dissociable Cyanide (WAD)	Preparation for measurement system as appropriate.	Recommended: Colourimetry Alternate: Ion chromatography with Electrochemical detector.	EC Vers.1.0/87 SM18(4500 I)	0.01
Total Metals	Aluminum Beryllium Boron Cadmium Chromium Cobalt Copper Iron Lead Molybdenum Nickel Silver Strontium Thallium Tin Vanadium Zinc	Recommended: Nitric acid or aqua regia digestion Alternate: Other acid digestion as appropriate	Recommended: Flame Atomic Absorption Spectrometry and/or Emission Spectrometry - Inductively Coupled Plasma (ICP) Alternative: Polarography via the method of standard addition in the presence of suitable electrolyte or Graphite Furnace AA	EC Vers.2.6/91 SM18(3500B) SM18(3500C)	0.03 0.01 0.05 0.002 0.02 0.02 0.01 0.08 0.03 0.02 0.02 0.03 0.02 0.03 0.06 0.03 0.01

TABLE 7-1

ANALYSIS PRINCIPLES AND METHOD DETECTION LIMITS FOR WASTEWATER CHARACTERIZATION IN THE FRASER BASIN

Analytical Test Group	Parameter	Sample Preparation Method Principles	Instrumental Measurement Method Principles	Reference Method	Analytical Detection Limit (mg/L)
Dissolved Metals	Aluminum	Filter through 0.45 µm membrane filter	Recommended: Flame Atomic Absorption Spectrometry and/or Emission Spectrometry - Inductively Coupled Plasma (ICP) Alternative: Polarography via the method of standard addition in the presence of suitable electrolyte or Graphite Furnace AA	EC Vers.2.6/91 SM18(3500B) SM18(3500C)	0.03
	Beryllium				0.01
	Boron				0.05
	Cadmium				0.002
	Chromium				0.02
	Cobalt				0.02
	Copper				0.01
	Iron				0.08
	Lead				0.03
	Molybdenum				0.02
	Nickel				0.02
	Silver				0.03
	Strontium				0.02
	Thallium				0.03
	Tin				0.06
	Vanadium				0.03
	Zinc				0.01
Mercury	Mercury	Oxidative acid digestion	Recommended: Cold Vapour Atomic Absorption Alternate: Hydride- Cold Vapour AA	EC Vers.2.0/87 SM18(3112B)	0.0001
Chromium (Hexavalent)	Chromium (VI)	Recommended: (i)None Alternate: (ii)Solvent Extraction	Recommended: (i) Colourimetry (ii) Atomic Absorption Alternative: Polarography via the method of standard addition in the presence of suitable electrolyte.	Ontario MOE (E3056A) SM18(3500D)	0.01

TABLE 7-1

ANALYSIS PRINCIPLES AND METHOD DETECTION LIMITS FOR WASTEWATER CHARACTERIZATION IN THE FRASER BASIN

Analytical Test Group	Parameter	Sample Preparation Method Principles	Instrumental Measurement Method Principles	Reference Method	Analytical Detection Limit (mg/L)
Hydrides	Arsenic	Acid Digestion	Recommended: Hydride Generation in conjunction with Atomic Absorption, or with Inductively Coupled Plasma or with Inductively Coupled Plasma/MS Alternate: Graphite Furnace AA or Polarography via the method of standard addition in the presence of suitable electrolyte.	EC Vers.2.1/90 SM18(3114C)	0.005
Chlorophenols	Chlorophenol, penta- Chlorophenol, (2,3,4,6+2,3,5,6) Chlorophenol, 2,3,4,5-tetra- Chlorophenol, 2,3,4-tri- Chlorophenol, 2,3,5-tri- Chlorophenol, 2,3,6-tri- Chlorophenol, 2,4,5-tri- Chlorophenol, 2,4,6-tri- Chlorophenol, 2,4-di- Chlorophenol, 2,6-di-	Recommended: In situ acetylation, extraction in the presence of ascorbic acid to prevent oxidation of catechols. Alternate: Acidify, liquid/liquid extraction, Methylation	Recommended: Gas Chromatography/Mass Spectrometry, Capillary Column Alternate: Gas Chromatography/Mass Spectrometry or Electron Capture, Capillary Column	US EPA 1653 EC Vers.3.0/91 SM18(6420B)	0.005 0.005 0.005 0.005 0.005 0.005 0.005 0.005 0.005 0.005
Chloroguaiacols	Chloroguaiacol, tetra- Chloroguaiacol, 3,4,5-tri- Chloroguaiacol, 3,4,6-tri- Chloroguaiacol, 4,5,6-tri- Chloroguaiacol, 4,5-di- Chloroguaiacol, 4,6-di- Chloroguaiacol, 5- Chloroguaiacol, 6-				0.005 0.005 0.005 0.005 0.005 0.005 0.005 0.005

TABLE 7-1

ANALYSIS PRINCIPLES AND METHOD DETECTION LIMITS FOR WASTEWATER CHARACTERIZATION IN THE FRASER BASIN

Analytical Test Group	Parameter	Sample Preparation Method Principles	Instrumental Measurement Method Principles	Reference Method	Analytical Detection Limit (mg/L)
Chlorocatechols	Chlorocatechol, tetra- Chlorocatechol, 3,4,5-tri- Chlorocatechol, 3,4-di- Chlorocatechol, 3,5-di- Chlorocatechol, 4,5-di- Chlorocatechol, 4-	As for chlorophenols and chloroguaiacols	As for chlorophenols and chloroguaiacols	As for chlorophenols and chloroguaiacols	0.005 0.005 0.005 0.005 0.005 0.005
Nonylphenol	Nonylphenol				0.005
Chlorinated Dibenzo-p-dioxins	2,3,7,8-T4CDD 1,2,3,7,8-P5CDD 1,2,3,4,7,8-H6CDD 1,2,3,6,7,8-H6CDD 1,2,3,7,8,9-H6CDD 1,2,3,4,6,7,8-H7CDD OCDD	Filter sample, extract solids by Soxhlet using toluene; extract filtrate normally, combine both extracts.	Recommended: Gas Chromatography/High Resolution Mass Spectrometry (GC/HRMS) Capillary Column Alternate: Low resolution MS may be acceptable with effective cleanup or GC/MS/MS	Environment Canada - EPS Report 1/RM/19 1992	10 pg/L 20 pg/L 20 pg/L 20 pg/L 20 pg/L 30 pg/L 40 pg/L
Chlorinated Dibenzofurans	2,3,7,8-T4CDF 1,2,3,7,8-P5CDF 2,3,4,7,8-P5CDF 1,2,3,4,7,8-H6CDF 1,2,3,6,7,8-H6CDF 2,3,4,6,7,8-H6CDF 1,2,3,7,8,9-H6CDF 1,2,3,4,6,7,8-H7CDF 1,2,3,4,7,8,9-H7CDF OCDF				10 pg/L 20 pg/L 20 pg/L 20 pg/L 20 pg/L 20 pg/L 20 pg/L 30 pg/L 30 pg/L 40 pg/L

TABLE 7-1

ANALYSIS PRINCIPLES AND METHOD DETECTION LIMITS FOR WASTEWATER CHARACTERIZATION IN THE FRASER BASIN

Analytical Test Group	Parameter	Sample Preparation Method Principles	Instrumental Measurement Method Principles	Reference Method	Analytical Detection Limit (mg/L)
Polycyclic Aromatic Hydrocarbons (PAHs)	Acenaphthene	Liquid/liquid extraction	Recommended: Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column.	SM18(6410B) 40CFR Part 136, 1984 -(625)	0.002
	Acenaphthylene				0.002
	Anthracene				0.002
	Benz(a)anthracene				0.002
	Benzo(a)pyrene				0.002
	Benzo(b+k)fluoranthene				0.002
	Benzo(ghi)perylene				0.002
	Chrysene				0.002
	Dibenz(ah)anthracene				0.002
	Fluoranthene				0.002
	Fluorene				0.002
	Indeno(123-cd)pyrene				0.002
	Naphthalene				0.002
	Phenanthrene				0.002
	Pyrene				0.002
Chlorobenzenes	1,2,4-trichlorobenzene	Liquid/liquid extraction. Neutral pH. Cleanup if necessary.	Recommended: (i) Gas Chromatography/Electron Capture Detector (GC/ECD) Capillary Column. (ii) Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column.		0.0001
	1,2,3-trichlorobenzene				0.0001
	1,2,4,5-tetrachlorobenzene				0.0001
	1,2,3,4-tetrachlorobenzene				0.0001
	Pentachlorobenzene				0.0001
	Hexachlorobenzene				0.0001
					0.0001

TABLE 7-1

ANALYSIS PRINCIPLES AND METHOD DETECTION LIMITS FOR WASTEWATER CHARACTERIZATION IN THE FRASER BASIN

Analytical Test Group	Parameter	Sample Preparation Method Principles	Instrumental Measurement Method Principles	Reference Method	Analytical Detection Limit (mg/L)
Volatiles, Halogenated	1,4-dichlorobenzene 1,2-dichlorobenzene Chloroform 1,2-Dichloroethane 1,1,1-Trichloroethane 1,1,2,2-Tetrachloroethane Dichloromethane Trichloroethylene Tetrachloroethylene	Purge and trap.	Recommended: Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column Alternate: Gas Chromatography/Hall Electrolytic Conductivity Detector	40CFR Part 136, 1984 EPA-624 EPA-601	0.005 0.005 0.005 0.005 0.005 0.005 0.005
Volatiles, Non-halogenated	Benzene Toluene Ethylbenzene Styrene o-xylene m-xylene + p-xylene	Purge and trap. Alternate Procedure: Headspace	Recommended: Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column Alternate: Gas Chrom./Photo Ionization Detector Gas Chrom./Mass Spectrometry	40CFR Part 136, 1984 EPA-624 EPA-602 EC Vers.1.0/93	0.001 0.001 0.001 0.001 0.001 0.001
Resin Acids	Abietic Acid Chlorodehydroabietic Acid Dehydroabietic Acid Isopimaric Acid Levopimaric Acid Neoabietic Acid Sandaracopimaric Acid Dichlorodehydroabietic Acid	pH adjusted to 9, Liquid/liquid extraction with methyl t-butyl ether, Methylation	Recommended: Gas Chromatography/Mass Spectrometry, Capillary Column	EC Vers.2.5/91	0.005 0.005 0.005 0.005 0.005 0.005 0.005 0.005

TABLE 7-1

ANALYSIS PRINCIPLES AND METHOD DETECTION LIMITS FOR WASTEWATER CHARACTERIZATION IN THE FRASER BASIN

Analytical Test Group	Parameter	Sample Preparation Method Principles	Instrumental Measurement Method Principles	Reference Method	Analytical Detection Limit (mg/L)
Adsorbable Organic Halogen (AOX)	Adsorbable Organic Halogen (AOX) Note: Analysis should be carried out in an environment free of chlorinated solvents.	Carbon adsorption (column or shaker) at pH 2 followed by nitrate wash, Dohrmann charcoal 100-200 mesh granular activated carbon or equivalent.	Pyrolysis in an oxygen rich atmosphere followed by microcoulometric analysis.	EC Vers.1.0/92 SM18(5320B)	0.05 based on 2,4,6-trichloro phenol
Anti-sapstain Compounds	Didecyl dimethyl ammonium chloride (DDAC)	Liquid-liquid extraction	Recommended: Gas Chromatography with Nitrogen/ Phosphorus Detector (GC/NPD) Capillary Column	EC Vers.1.0/91	0.01
	1-Iodo-2-Propynyl Butyl Carbamate (IPBC)	Liquid-liquid extaction	Recommended: Gas Chromatography with Nitrogen/ Phosphorus Detector (GC/NPD) Capillary Column	EC Vers.2.0/93	0.01
Biochemical Oxygen Demand (BOD5)	Biochemical Oxygen Demand - 5 Day (BOD5)	Preparation for measurement system as appropriate e.g. destruction of chlorine, neutralization of pH, and stabilizaition of sample at 20°C. Preparation of seed and dilution water as appropriate. Dilution of sample to provide adequate oxygen depletion over 5 day period.	Recommended: Dissolve oxygen determination by Winkler Method or by oxygen electrode verified by Winkler Method for one sample per analytical run on says of analysis.	BC MOE SM18(5210B)	10

REFERENCES:

1. Methods Manual/Environment Canada, Conservation and Protection Laboratories, Pacific and Yukon Region
2. Laboratory Manual for the Chemical Analysis of Waters, Wastewaters, Sediments and Biological Materials. N.R Mc Quaker, BC Ministry of Environment, 1976.
3. Standard Methods for the Examination of Water and Wastewater. 18th Edition 1992.
4. Federal Register, Part VIII, US Environmental Protectio Agency, 40 CFR Part 136
5. Reference Method for the Determination of Polychlorinated Dibenzo-para-dioxins(PCDDs) and Polychlorinated dibenzofurans (PCDFs) in Pulp and Paper Mill Effluents. Environment Canada Report EPS 1/RM/19, 1992

REPORTING REQUIREMENTS

8.1 Introduction

Once the analytical data have been received and verified, they must be reported in a format that allows them to be entered into Environment Canada's Envirodat database. The Envirodat database assigns a unique code number to each parameter and each analytical method. Table 8-1 indicates the **Envirodat** code number for each of the recommended wastewater characterization parameters and for each of the recommended preferred and alternate analytical methods. All chemical characterization data should be reported using these codes. The data report must also contain the station and sample information required for input to **Envirodat**.

Prepare a hard (typed) copy of the data report. Divide the report into five sections:

- Station Information
- Sample Information
- Sample Summary
- Analysis Results
- Quality Control Data

In addition to the hard copy report, all tables of sample analysis results and Envirodat codes must be provided on a 3 1/2 inch floppy diskette that can be used on a **DOS**-operated IBM-PC(c) or compatible. If possible, use Lotus **1-2-3®** or **Excel®** tables.

8.2 Station Information

In this first section include the following information:

- Site name;
- Discharge identification;
- Latitude;
- Longitude;
- UTM Zone;
- Easting; and
- Northing.

TABLE 8-1

ENVIRODAT CODES FOR FRASER BASIN WASTEWATER CHARACTERIZATION PARAMETERS

VMV CODE	VARIABLE CODE	VARIABLE NAME	UNITS	METHOD CODE	METHOD NAME
CORE PARAMETERS					
2061	840	Temperature	Deg. C	38	Mercury Thermometer
8102	659	Dissolved Oxygen	mg/L	325	Oxygen Meter/Probe
10301	687	pH	pH units	389	Electrode
10108	364	Alkalinity	(CaCO ₃) mg/L	370	Potentiometric Titr.
2041	817	Conductivity	usie/cm	32	Meter
10401	749	Total Suspended Solids	mg/L	396	Gravimetric
6101	478	Dissolved Organic Carbon (DOC)	mg/L	95	Infrared Method
320	1048	Chemical Oxygen Demand (COD)	mg/L	343	Titration
INDUSTRY SPECIFIC PARAMETERS					
7566	604	Ammonia plus Ammonium	mg/L	2347	Colourimetry
7564	604	Ammonia plus Ammonium	mg/L	267	Ion Chromatography
7206	613	Nitrite	mg/L	239	Colourimetry
444	612	Nitrite	mg/L	617	Ion Chromatography
NA	428	Strong Acid Dissociable Cyanide	NA	NA	Colourimetry
NA	427	Weak Acid Dissociable Cyanide	NA	NA	Colourimetry
13006	373	Aluminum, total	mg/L	479	AA
13009	373	Aluminum, total	mg/L	1502	ICP
13004	373	Aluminum, total	mg/L	481	Graphite Furnace AA
13102	370	Aluminum, dissolved	mg/L	479	AA
13109	370	Aluminum, dissolved	mg/L	1502	ICP
13105	370	Aluminum, dissolved	mg/L	481	Graphite Furnace AA
4009	393	Beryllium, total	mg/L	1502	ICP
4103	391	Beryllium, dissolved	mg/L	1502	ICP
5001	401	Boron, total	mg/L	83	AA
5107	399	Boron, dissolved	mg/L	1502	ICP
48000	412	Cadmium, total	mg/L	479	AA
48009	412	Cadmium, total	mg/L	1502	ICP
48003	412	Cadmium, total	mg/L	481	Graphite Furnace AA
48101	409	Cadmium, dissolved	mg/L	1161	AA
48109	409	Cadmium, dissolved	mg/L	1502	ICP
48103	409	Cadmium, dissolved	mg/L	481	Graphite Furnace AA
24002	463	Chromium, total	mg/L	1113	AA
24009	463	Chromium, total	mg/L	1502	ICP
24004	463	Chromium, total	mg/L	481	Graphite Furnace AA
24104	460	Chromium, dissolved	mg/L	1113	AA
24111	460	Chromium, dissolved	mg/L	1516	ICP
24056	460	Chromium, dissolved	mg/L	481	Graphite Furnace AA
27001	456	Cobalt, total	mg/L	479	AA
27009	456	Cobalt, total	mg/L	1502	ICP
27003	456	Cobalt, total	mg/L	481	Graphite Furnace AA
27101	453	Cobalt, dissolved	mg/L	479	AA
27109	453	Cobalt, dissolved	mg/L	1502	ICP
27107	453	Cobalt, dissolved	mg/L	1232	Graphite Furnace AA
29006	470	Copper, total	mg/L	479	AA
29009	470	Copper, total	mg/L	1502	ICP
29003	470	Copper, total	mg/L	481	Graphite Furnace AA

TABLE 8-1

ENVIRODAT CODES FOR FRASER BASIN WASTEWATER CHARACTERIZATION PARAMETERS

VMV CODE	VARIABLE CODE	VARIABLE NAME	UNITS	METHOD CODE	METHOD NAME
29106	467	Copper, dissolved	mg/L	479	AA
29109	467	Copper, dissolved	mg/L	1502	ICP
29107	467	Copper, dissolved	mg/L	481	Graphite Furnace AA
26004	519	Iron, total	mg/L	479	AA
26009	519	Iron, total	mg/L	1502	ICP
26104	516	Iron, dissolved	mg/L	1196	AA
26109	516	Iron, dissolved	mg/L	1502	ICP
26107	516	Iron, dissolved	mg/L	481	Graphite Furnace AA
82001	683	Lead, total	mg/L	479	AA
82009	683	Lead, total	mg/L	1502	ICP
82004	683	Lead, total	mg/L	481	Graphite Furnace AA
82101	678	Lead, dissolved	mg/L	479	AA
82109	678	Lead, dissolved	mg/L	1502	ICP
82104	678	Lead, dissolved	mg/L	481	Graphite Furnace AA
42000	595	Molybdenum, total	mg/L	1113	AA
42009	595	Molybdenum, total	mg/L	1502	ICP
42006	595	Molybdenum, total	mg/L	481	Graphite Furnace AA
42101	593	Molybdenum, dissolved	mg/L	1113	AA
42109	593	Molybdenum, dissolved	mg/L	1502	ICP
28001	610	Nickel, total	mg/L	479	AA
28009	610	Nickel, total	mg/L	1502	ICP
28101	607	Nickel, dissolved	mg/L	479	AA
28109	607	Nickel, dissolved	mg/L	1502	ICP
28107	607	Nickel, dissolved	mg/L	481	Graphite Furnace AA
47001	350	Silver, total	mg/L	1481	AA
326	350	Silver, total	mg/L	2577	ICP
47005	350	Silver, total	mg/L	481	Graphite Furnace AA
47101	348	Silver, dissolved	mg/L	1481	AA
353	348	Silver, dissolved	mg/L	2577	ICP
38001	823	Strontium, total	mg/L	479	AA
38009	823	Strontium, total	mg/L	1502	ICP
38101	819	Strontium, dissolved	mg/L	479	AA
38109	819	Strontium, dissolved	mg/L	1502	ICP
81001	861	Thallium, total	mg/L	479	AA
349	861	Thallium, total	mg/L	2577	ICP
81004	861	Thallium, total	mg/L	481	Graphite Furnace AA
81101	859	Thallium, dissolved	mg/L	479	AA
376	859	Thallium, dissolved	mg/L	2577	ICP
50005	812	Tin, total	mg/L	1170	AA
346	812	Tin, total	mg/L	2577	ICP
50006	812	Tin, total	mg/L	1532	Graphite Furnace AA
50101	810	Tin, dissolved	mg/L	1161	AA
373	810	Tin, dissolved	mg/L	2577	ICP
23001	878	Vanadium, total	mg/L	1089	AA
351	878	Vanadium, total	mg/L	2577	ICP
23101	876	Vanadium, dissolved	mg/L	1098	AA
23109	876	Vanadium, dissolved	mg/L	1502	ICP

TABLE 8-1

ENVIRODAT CODES FOR FRASER BASIN WASTEWATER CHARACTERIZATION PARAMETERS

VMV CODE	VARIABLE CODE	VARIABLE NAME	UNITS	METHOD CODE	METHOD NAME
30004	890	Zinc, total	mg/L	479	AA
30009	890	Zinc, total	mg/L	1502	ICP
30003	890	Zinc, total	mg/L	481	Graphite Furnace AA
30104	887	Zinc, dissolved	mg/L	479	AA
30109	887	Zinc, dissolved	mg/L	1502	ICP
30107	887	Zinc, dissolved	mg/L	481	Graphite Furnace AA
80011	549	Mercury, total	ug/L	1594	Flameless AA
24101	458	Chromium (VI)	mg/L	1127	Colourimetric
33008	379	Arsenic, total	mg/L	1367	Hydride ICP
33108	376	Arsenic, dissolved	mg/L	1367	Hydride ICP
17804	307	Pentachlorophenol	ug/L	643	Acetylation GC/MS
95141	307	Pentachlorophenol	ug/L	2316	Methylation GC/MS
17723	228	2,3,4,6-Tetrachlorophenol	ng/L	643	Acetylation GC/MS
17721	321	2,3,5,6-Tetrachlorophenol	ng/L	643	Acetylation GC/MS
17720	198	2,3,4,5-Tetrachlorophenol	ug/L	643	Acetylation GC/MS
17710	123	2,3,4-Trichlorophenol	ug/L	643	Acetylation GC/MS
17711	320	2,3,5-Trichlorophenol	ug/L	643	Acetylation GC/MS
17712	319	2,3,6-Trichlorophenol	ug/L	643	Acetylation GC/MS
17714	333	2,4,5-Trichlorophenol	ng/L	643	Acetylation GC/MS
17713	308	2,4,6-Trichlorophenol	ug/L	643	Acetylation GC/MS
17702	96	2,4-Dichlorophenol	ug/L	643	Acetylation GC/MS
17704	305	2,6-Dichlorophenol	ug/L	643	Acetylation GC/MS
NA	957	Tetrachloroguaiacol	NA	NA	Acetylation GC/MS
NA	955	3,4,5-Trichloroguaiacol	NA	NA	Acetylation GC/MS
NA	NA	3,4,6-Trichloroguaiacol	NA	NA	Acetylation GC/MS
NA	NA	4,5,6-Trichloroguaiacol	NA	NA	Acetylation GC/MS
NA	NA	4,5-Dichloroguaiacol	NA	NA	Acetylation GC/MS
NA	NA	4,6-Dichloroguaiacol	NA	NA	Acetylation GC/MS
NA	NA	5-Chloroguaiacol	NA	NA	Acetylation GC/MS
NA	NA	6-Chloroguaiacol	NA	NA	Acetylation GC/MS
NA	958	Tetrachlorocatechol	NA	NA	Acetylation GC/MS
NA	956	3,4,5-Trichlorocatechol	NA	NA	Acetylation GC/MS
NA	NA	3,4-dichlorocatechol	NA	NA	Acetylation GC/MS
NA	NA	3,5-dichlorocatechol	NA	NA	Acetylation GC/MS
NA	NA	4,5-dichlorocatechol	NA	NA	Acetylation GC/MS
NA	NA	4-Chlorocatechol	NA	NA	Acetylation GC/MS
NA	619	Nonylphenol	NA	NA	Acetylation GC/MS
NA	129	2,3,7,8-T4CDD	NA	NA	GC/High Res. MS
NA	NA	1,2,3,7,8-P5CDD	NA	NA	GC/High Res. MS
NA	NA	1,2,3,4,7,8-H6CDD	NA	NA	GC/High Res. MS
NA	NA	1,2,3,6,7,8-H6CDD	NA	NA	GC/High Res. MS
NA	NA	1,2,3,7,8,9-H6CDD	NA	NA	GC/High Res. MS
NA	NA	1,2,3,4,6,7,8-H7CDD	NA	NA	GC/High Res. MS
NA	922	O8CDD	NA	NA	GC/High Res. MS
NA	923	2,3,7,8-TCDF	NA	NA	GC/High Res. MS
NA	NA	1,2,3,7,8-P5CDF	NA	NA	GC/High Res. MS
NA	NA	2,3,4,7,8-P5CDF	NA	NA	GC/High Res. MS

TABLE 8-1

ENVIRODAT CODES FOR FRASER BASIN WASTEWATER CHARACTERIZATION PARAMETERS

VMV CODE	VARIABLE CODE	VARIABLE NAME	UNITS	METHOD CODE	METHOD NAME
NA	NA	1,2,3,4,7,8-H6CDF	NA	NA	GC/High Res. MS
NA	NA	2,3,4,6,7,8-H6CDF	NA	NA	GC/High Res. MS
NA	NA	1,2,3,7,8,9-H6CDF	NA	NA	GC/High Res. MS
NA	NA	1,2,3,7,8,9-H6CDF	NA	NA	GC/High Res. MS
NA	NA	1,2,3,4,6,7,8-H7CDF	NA	NA	GC/High Res. MS
NA	NA	1,2,3,4,7,8,9-H7CDF	NA	NA	GC/High Res. MS
NA	928	O8CDF	NA	NA	GC/High Res. MS
96216	292	Acenaphthene	ng/L	1981	GC/MS
96215	148	Acenaphthylene	ng/L	1981	GC/MS
NA	93	Anthracene	NA	NA	GC/MS
NA	219	Benz(a)anthracene	NA	NA	GC/MS
96223	204	Benzo(a)pyrene	ng/L	1981	GC/MS
96222	147	Benzo(k)fluoranthene	ng/L	1981	GC/MS
96221	145	Benzo(b)fluoranthene	ng/L	1981	GC/MS
NA	132	Benzo(ghi)perylene	NA	NA	GC/MS
NA	155	Chrysene	NA	NA	GC/MS
NA	212	Dibenz(ah)anthracene	NA	NA	GC/MS
96220	146	Fluoranthene	ng/L	1981	GC/MS
96217	303	Fluorene	ng/L	1981	GC/MS
96224	140	Indeno(123-cd)pyrene	ng/L	1981	GC/MS
NA	312	Naphthalene	NA	NA	GC/MS
96218	299	Phenanthrene	ng/L	1981	GC/MS
96219	107	Pyrene	ng/L	1981	GC/MS
17831	95	1,2,4-trichlorobenzene	ug/L	677	GC/ECD
17832	304	1,2,3-trichlorobenzene	ug/L	677	GC/ECD
17841	247	1,2,4,5-tetrachlorobenzene	ug/L	677	GC/ECD
17842	245	1,2,3,4-tetrachlorobenzene	ug/L	677	GC/ECD
17850	237	Pentachlorobenzene	ug/L	677	GC/ECD
17812	91	Hexachlorobenzene	ug/L	677	GC/ECD
95213	64	1,4-dichlorobenzene	ug/L	2321	P&T GC/MS
95211	329	1,2-dichlorobenzene	ug/L	2321	P&T GC/MS
95215	66	1,2-Dichloroethane	ug/L	2321	P&T GC/MS
95227	257	1,1,1-Trichloroethane	ug/L	2321	P&T GC/MS
95224	290	1,1,2,2-Tetrachloroethane	ug/L	2321	P&T GC/MS
95222	272	Dichloromethane	ug/L	2321	P&T GC/MS
95231	289	Trichloroethylene	ug/L	2321	P&T GC/MS
95225	106	Tetrachloroethylene	ug/L	2321	P&T GC/MS
95208	250	Chloroform	ug/L	2321	P&T GC/MS
95200	256	Benzene	ug/L	2321	P&T GC/MS
95226	73	Toluene	ug/L	2321	P&T GC/MS
95221	52	Ethylbenzene	ug/L	2321	P&T GC/MS
95223	53	Styrene	ug/L	2321	P&T GC/MS
95076	328	o-Xylenes	ug/L	2321	P&T GC/MS
95077	113	m-xylene + p-xylene	ug/L	2321	P&T GC/MS

TABLE 8-1

ENVIRODAT CODES FOR FRASER BASIN WASTEWATER CHARACTERIZATION PARAMETERS

VMV CODE	VARIABLE CODE	VARIABLE NAME	UNITS	METHOD CODE	METHOD NAME
NA	950	Abietic Acid	NA	NA	GC/MS
NA	952	Chlorodehydroabietic Acid	NA	NA	GC/MS
NA	949	Dehydroabietic Acid	NA	NA	GC/MS
NA	947	Isopimaric Acid	NA	NA	GC/MS
NA	948	Levopimaric Acid	NA	NA	GC/MS
NA	951	Neoabietic Acid	NA	NA	GC/MS
NA	946	Sandaracopimaric Acid	NA	NA	GC/MS
NA	953	Dichlorodehydroabietic Acid	NA	NA	GC/MS
65	944	Adsorbable Organic Halogen (AOX)	ug/L	2375	Carbon Adsorption
NA	NA	Didecyl Dimethyl Ammonium Chloride (DDAC)	NA	NA	GC/NPD
NA	NA	1-Iodo-2-Propynyl Butyl Carbamate (IPBC)	NA	NA	GC/NPD
17102	423	Chlorine, Total Residual	mg/L	624	Field Kit - DPD Colourimetric
8202	652	Biological Oxygen Demand(BOD-5 d)	mg/L	331	Oxygen Probe

NA = NOT AVAILABLE

8.3 Sample Information

For each sample include the following information:

- Laboratory sample number;
- Agency or company who collected the sample; ,
- Sampling time and date, as follows:
 - Sampling start time: day/month/year, hour/minute
 - Sampling end time: day/month/year, hour/minute
- Time zone (time zone applicable to the date and time above);
- Sample type (eg., grab, composite, field duplicate, travel blank, field spike, filter or field rinsate lab duplicate, lab blank, or lab spike);
- Collection (how the sample was collected, eg. bucket or peristaltic pump); and
- Comments.

8.4 Summary of Analytical Method

In this section, include a short summary of the analytical procedures followed, mentioning the sample preparation techniques, instruments used, detection limit reporting for chromatographic analyses, and any changes from the published analytical methods. Specify references.

8.5 Analysis Results

Analysis results for each sample are tabulated in the fourth section of the report. The report of analytical results must include all of the following information:

- Laboratory sample number;
- VMV code (from Table 8-1);
- Value (analysis result in units defined by the VMV code);
- Units of measurement;
- Sample detection limit;
- Measurement date (the date the analytical value was determined; for field measurements, this is the same as the sampling date);
- Name of laboratory that performed the analysis;
- Pretreatment (eg. field filtered or preserved or lab filtered); and
- Observation (comment or qualifier, eg. reported data value is questionable, or suspected laboratory contamination).

8.6 **Quality Control Data**

Values obtained for replicate samples may be reported as a column in the analysis results table (fourth section of the data report). All other quality control data should be presented in this last section of the report.

TOXICITY TESTS

9.1 Introduction

The intent of this chapter is to provide instructions for the collection and toxicity testing of effluent samples for the Wastewater Characterization Program. There are three sections dealing with the following topics:

- sample collection
- sample transport and storage
- testing and reporting

Each step involved in the collection and toxicity testing of a wastewater sample is covered in detail. Table 9-1 illustrates a checklist for use in preparing for toxicity sampling. It also indicates information that should be reported by the testing facility.

The instructions encompass quality assurance practices required within the accreditation program of the Canadian Association for Environmental Analytical Laboratories (CAEAL) Inc. along with those specified in the test protocols published and supported by Environment Canada (see references). These quality assurance practices are paramount for the collection of reliable and meaningful data.

9.2 Preparation and Sample Collection

This first component involves the following tasks:

- coordination of sampling and testing
- collection of the sample
- transport of the sample to the test facility
- storage during transport and prior to testing.

Before collecting the samples, first contact the test facility and give them notice of your intent to collect a sample. One to two weeks is usually sufficient advance notice. Coordinating sampling with the test facility will ensure that they will be ready to process the sample in an efficient manner. This is particularly important for the more complicated chronic tests. If notice is given verbally, follow it up immediately with a written communication of your intent to collect a sample.

TABLE 9-1

CHECKLIST FOR SAMPLING AND TOXICITY TESTING OF AQUEOUS SAMPLES

ITEM	COMPLETED
	DATE/INITIAL
PREPARATION	
notify the testing facility of your intent to sample	
obtain containers and forms	
SAMPLING	
rinse containers three times with sample	
- fill and seal containers, minimize headspace	
- label containers and initial seals	
- fill out sample information form	
transport containers to testing facility	
INFORMATION REPORTED BY THE TESTING FACILITY	
sample information (type of substance, location, sampler, date, how sampled, contact person)	
- location of test facility (address and contacts)	
complete reference for the test method	
test conditions (dates started/finished, investigators, temperatures, light levels)	
test results (end points and method of calculation)	
reference toxicant data (endpoint and method of calculation)	
- monitoring data (biological and chemical)	
observations during test and other comments	

The samples must be collected in new plastic containers (polyethylene or polypropylene). Containers must not be recycled or reused. Glass and teflon-lined containers can also be used but these are often too impractical and expensive for larger volumes. The test facility may provide sample containers. Recommended containers include 5-gallon (20-L) polyethylene pails with lids. These pails are inexpensive, easy to fill, stackable, they can be labelled directly, and the lids form watertight seals. Rectangular, polyethylene gasoline cans (Jerry-cans) also make convenient containers. However, the warning labels imprinted on the sides may cause problems for shippers.

The type of container and its composition (polyethylene, polypropylene or teflon) should be recorded. If a plastic liner is used, its composition should be recorded as well.

To avoid problems inherent in preparing homogeneous, large-volume composites, the samples for toxicity tests should be collected as single grabs. The samples should be taken at the mid-point of the sampling period for chemical characterization (see Section 3.5).

The sample volumes for each test are specified in Table 9-2. A minimum of 44 L is required for the trout, Ceriodaphnia, bioluminescence and algal growth tests (a greater volume may be required for a salmonid larval growth test). Ideally, 60 L be collected. The extra volume can be used for chemical testing or archived until the tests are completed.

9.3 Sampling Protocol

The containers must be rinsed three times with the sample before filling. Each rinse should be with a volume not less than 5% of the total container volume. The rinse volumes can also be used to rinse the bottoms of the lids. Fill the containers to minimize the head space and then seal them firmly with lids. Wrap packing tape around the lid at the top of the pail and over any spouts in the lid. Initial the tape and label the pail and lid with a permanent black marker. The taping and initialing of the tape are simple things to do but they provide valuable information on sample integrity between the point and time of collection and receipt at the test facility. They provide a simple means to determine if the sample was opened or tampered with during transit.

Both the pail and lid must be labelled with the following information.

- type of substance (effluent, surface water, etc.)
- location of sample (company, municipality, etc.)
- name of sampler
- date sampled

TABLE 9-2		
SAMPLE VOLUMES REQUIRED TO CONDUCT SELECTED BIOLOGICAL TESTS		
TEST	VOLUME (L)	
	FULL DILUTION	SCREENING
Rainbow trout lethality	40	20
Bacterial luminescence inhibition	0.1	0.01
<i>Ceriodaphnia</i> survival and reproduction	3	0.2
Algal growth stimulation/inhibition	0.5	0.5

Note: Decision to conduct full dilution or screening tests made in consultation with FPAO

- how sampled (grab or composite)
- contact person (**name**, address, and phone number).

If the sample is collected in more than one container then each container must be assigned one number out of the total number of containers (e.g., 1 of 3, 2 of 3, and 3 of 3).

9.4 Sample Transport and Storage

The containers must be shipped to the test facility accompanied by a sample information form. Samples should be kept cool during shipment. Table 9-3 presents a sample information form. The chain-of-custody information is to ensure that someone is always accountable for the sample and that custody is recorded in an orderly and clear fashion. If the sample changes carriers during transit, then the carrier must sign the chain-of-custody section of the form accompanying the samples. The person receiving the sample at the test facility must also sign for the sample to complete the record. The temperature of the sample on receipt should be logged. Observations of sample conditions (e.g., ice present, container intact) should also be recorded. A copy of the completed form should then be returned to the sampler.

The sample must arrive at the test facility within 48 h of collection and chronic toxicity tests must be initiated within 72 h of collection. The trout and *Ceriodaphnia* protocols permit up to 5 days to elapse between the time of sampling and test initiation. “However, for purposes of simplicity and uniformity, all tests should be initiated within 72 h of sample collection. The sample must be stored at $5^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

9.5 Testing

Each test facility must have written standard operating procedures (SOPS) for conducting each test (see also Section 9.6.1). The SOPS must cover the method of data analysis and determination of endpoints. The standard operating procedures and records of performance in any inter-laboratory test programs must be available for inspection by the FPAO. The test facility must follow the appropriate Environment Canada test procedures. These procedures are published (see references) and available from the following Environment Canada offices:

EP Publications
Conservation and Protection
Ottawa, ON
K1A 0H3
(613) 953-5921

Communications Directorate
Conservation and Protection
224 West Esplanade
North Vancouver, BC
V7M 3H7
(604) 666-5900

9.6 Quality Assurance

The assurance of quality data is important for the implementation of the FPAO testing program. Guidelines are required to cover all aspects of sample collection, transport, storage and testing along with the analysis and reporting of the results. Adherence to guidelines makes it easier to interpret the test data and provides a basis for dealing with outliers.

9.6.1 Standard Operating Procedures

Different laboratories will be conducting the tests for the FPAO; to ensure comparable and reliable results, each facility must have defined standard operating procedures (SOPS) for conducting tests, managing data, and performing other laboratory functions such as maintaining cultures and equipment. Basic toxicity test procedures are well defined in published protocols. However, there is some room for interpretation in the implementation of each protocol. These kinds of details must be documented as part of a laboratory's standard operating procedures.

9.6.2 Quality Assurance Program

As well as standard operating procedures, testing facilities should have a Quality Assurance Program that outlines their quality assurance and quality control practices. Quality assurance encompasses all aspects of laboratory operations which may directly or indirectly involve a specific test. This can include everything from the collection of the sample, transport, storage, and testing to data recording, analysis, interpretation and reporting. Direct factors are the procedures and conditions under which the test is conducted. Indirect factors are those that may not directly effect a test but which could have some influence on the test results. Indirect factors include the care and maintenance of the test organisms, environmental conditions in the test facility, dilution water quality and staff training.

Quality control is an integral component of quality assurance. Quality control procedures are used to measure and assess data quality. Reference toxicants and the use of warning charts, along with participation in inter-laboratory test programs are quality control procedures. Reference toxicants used in Environment Canada protocols are listed in Table 9-4.

TABLE 9-4

REFERENCE TOXICANTS RECOMMENDED FOR BIOLOGICAL TESTING

TEST	REFERENCE TOXICANTS			
	ZnSO₄	PHENOL	KCl	NaCl
Rainbow trout lethality	+	+	+	
Bacterial luminescence inhibition	+	+		
<i>Ceriodaphnia</i> survival and reproduction			+	+
Algal growth stimulation/inhibition	+		+	+

- Control charts are an integral component of a quality assurance program. They provide a means to determine if the test organisms are responding in a reproducible fashion based on historical data. The control chart incorporates many direct and indirect factors that can influence a test. It also provides some assurance about the facility's ability to conduct the test.

Test facilities should run reference toxicants on a regular basis; if they have not run a reference toxicant in the last month, then one should be requested prior to submission of any samples. The reference toxicant result should then be compared against their control chart for that test. If the test is under control, then the facility has demonstrated its ability to run the test and provide reliable data.

9.7 **Reporting**

The reporting requirements for each test are summarized in Table 9-5. This table was compiled based on Environment Canada test protocols. In summary, the following general information is required with each test report:

- sample information
- location of the test facility
- test method reference
- test conditions
- test result
- reference toxicant data
- monitoring data (biological and chemical)
- observations during test and other comments.

This information is required to ensure data quality. Any additional information collected during the test must be kept on file by the testing facility for at least two years.

A control or warning chart for a reference toxicant must also be submitted with each test result. The reference-toxicant test result that corresponds with the sample must be clearly marked on this chart. Warning limits that are statistically defined from historical test data must also be marked on the control chart.

TABLE 9-5

REPORTING REQUIREMENTS FOR THE TOXICITY TESTING PROGRAM

REPORTING REQUIREMENT	<i>TROUT</i>	BACTERIAL LUMINESCENCE	CERIODAPHNIA	ALGAL GROWTH
SAMPLE INFORMATION				
type of sample	+	+	+	+
sample location	+	+	+	+
date & time of sampling	+	+	+	+
sampler	+	+	+	+
colour & odour	+	+	+	+
TEST CONDITIONS AND REPORTING				
method reference	+	+	+	+
test facility (name, location & contact person)	+	+	+	+
date test initiated	+	+	+	+
date test terminated	+	+	+	+
endpoints (report method by which NOEC and LOEC are calculated)	LC50/LC25	IC50/IC20	SURVIVAL	IC50/IC25/NOEC/LOEC
			LC50/LC25/NOEC/LOEC	
			REPRODUCTION	
			IC50/IC25/NOEC/LOEC	
method of analysis	+	+	+	+
MONITORING DATA				
pH, conductance, dissolved oxygen	+		+	+
ammonium	+		+	
alkalinity, hardness, free chlorine			+	
mortality over time	+		+	
weight	+			
length	+			
young production over time			+	
immobility			+	
observations on behaviour	+		+	

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Environmental Management Associates and Hydroqual Laboratories Ltd. · 1993. Evaluation of the PEEP Index and Recommended Toxicity Tests for the Fraser River Basin. Prepared for the Fraser Pollution Abatement Office, North Vancouver, British Columbia.

Environment Canada, 1990a. Biological Test Method: Acute Lethality Test Using Rainbow Trout. Report EPS 1/RM/9

Environment Canada, 1990b. Biological Test Method: Acute Lethality test Using *Daphnia* spp. Report EPS 1/RM/11.

Environment Canada, 1990c. Biological Test Method: Guidance Document on Control of Toxicity Test Precision using Reference Toxicants. EPS Report 1/RM/12.

Environment Canada, 1990d. Biological Test Method: Reference Method for Determining the Acute Lethality of Effluents to Rainbow Trout. Reference Method EPS 1/RM/13.

Environment Canada, 1990e. Biological Test Method: Reference Method for Determining Acute Lethality of Effluents to *Daphnia magna*. Reference Method EPS 1/RM/14.

Environment Canada, 1992a. Biological Test Method: Test of Reproduction and Survival Using the Cladoceran *Ceriodaphnia dubia*. Report EPS 1/RM/21.

Environment Canada, 1992b. Biological Test Method: Test of Larval Growth and Survival Using Fathead Minnows. Report EPS 1/RM/22.

Environment Canada, 1992c. Biological Test Method: Growth Inhibition Test Using the Freshwater Alga *Selenastrum capricornutum*. Report EPS 1/RM/25

Environment Canada, 1992d. Biological Test Method: Toxicity Test Using Luminescent Bacteria (*Photobacterium phosphoreum*). Report EPS 1/RM/24

REFERENCES

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Metcalf and Eddy Inc. 1979. Wastewater Engineering: Treatment, Disposal, Reuse. Tata McGraw-Hill Publishing Company Ltd., New Delhi.

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Ontario Ministry of the Environment (OMOE). 1988a. Ontario Regulation 695/88. Effluent Monitoring - General, 1988.

Ontario Ministry of Environment (OMOE). 1991. Protocols for the Sampling and Analysis of Industrial/Municipal Wastewater, June, 1991.

Appendix 1

Glossary

GLOSSARY

Process Effluent. The MISA Monitoring Regulation [Ontario Regulation 695/88] defines a process effluent as “any effluent that comes into contact by design with any industrial process”. This definition is very broad and mainly concerns any water that would come into contact with raw materials, manufactured products, or waste products. Contact cooling water would be classified as a process effluent.

Cooling Water. MISA defines a cooling water as “water that is used in an industrial process for the purpose of removing heat and that is not intended to come in contact with process materials”. Cooling waters are usually continuous streams with little variability in their quality.

Sanitary Effluent includes effluents from washrooms, washing areas, and food preparation areas such as cafeterias. For office, tourist, retail establishments, and some industries which do not use or consume water, sanitary effluents are usually the only discharges present. The flows of sanitary effluents typically are variable. In the evenings when most of these establishments are closed, the flows usually drop considerably or cease.

Stormwater can be defined as “run-off from a storm event or thaw that is discharged from a developed area of the plant directly or indirectly to a surface watercourse”. Some regulations have specified that a storm event occurs when more than 5 mm of rainfall over a 24-hour period is measured within a 2 kilometre distance (OMOE 1988a). Stormwaters are typically collected by roof drains, parking lot drains and ditches. Stormwaters often have variable flow, which is dependent on rainfall intensity and duration.

Combined Effluent. The definition of combined effluent adopted by MISA was flow “resulting from the intentional combination of process effluent with cooling water”. For the purpose of these guidelines a combined effluent can be the combination of any of the above discharges. Run off collected in ditch systems or culverts from agricultural operations can be considered a combined effluent. This discharge can result from a combination of excess irrigation waters and storm events.

Municipal Wastewater Treatment Plant (WWTP) Effluent. Municipal wastewater treatment plants typically receive a combination of domestic sewage, some effluent from local industries, and, in some cases, urban stormwater. Thus, their effluents may show some of the characteristics of sanitary effluents, industrial effluents, and urban runoff. Flows through municipal wastewater treatment plants typically vary on a daily cycle. ,

Table 3.18. Precision in the INA Analysis of dry vegetation

	Au ng/g	Ag µg/g	As µg/g	Ba µg/g	Br µg/g	Ca %	Co µg/g	Cr µg/g	Cu µg/g	Fe %	Hf µg/g	Hg µg/g	Ir ng/L	K %	Mo µg/g	Na µg/g	Ni µg/g	Pb µg/g
CLV-1	10.3	<0.3	6.7	<10	3.9	0.95	0.7	4.2	<0.05	0.152	0.37	<0.31	INT	0.30	INT	228	17	4
	11.8	<0.3	7.6	<10	4.7	0.95	0.8	5.6	<0.05	0.188	0.38	<0.34	INT	0.33	INT	277	33	4
	13.5	<0.3	7.2	<10	4.6	0.97	0.9	4.6	0.07	0.173	0.39	<0.33	INT	0.37	INT	284	20	4
V-4	1.3	<0.3	0.43	20	2.4	0.81	0.5	3.3	<0.05	0.090	0.22	0.12	<0.1	0.20	0.12	461	<2	2
	1.0	<0.3	0.44	28	2.8	0.95	0.8	4.0	0.08	0.105	0.28	<0.05	<0.1	0.23	0.11	547	4	2
	1.0	<0.3	0.28	17	2.0	0.59	0.4	2.8	<0.05	0.076	0.20	<0.05	<0.1	0.15	0.21	417	3	<1
	0.7	<0.3	0.30	19	1.9	0.55	0.4	2.4	<0.05	0.073	0.22	<0.05	<0.1	0.14	0.11	410	4	2
BLIND DUPLICATES																		
01 ES.TW	1.7	<0.3	0.22	53	1.9	0.81	0.2	0.7	<0.05	0.024	<0.05	0.07	<0.1	0.64	<0.05	64	<2	4
	1.9	<0.3	0.28	49	1.5	0.73	0.2	0.8	<0.05	0.022	<0.05	<0.05	<0.1	0.60	0.07	58	3	4
02 ES.TW	1.1	<0.3	0.14	76	1.0	0.83	0.2	0.6	0.18	0.013	<0.05	0.08	<0.1	0.50	<0.05	36.8	<2	4
	1.0	<0.3	0.15	82	1.1	0.84	0.2	0.8	0.19	0.014	<0.05	0.08	<0.1	0.55	0.05	37.8	2	4
15 ES.BK	11.7	<0.3	1.8	160	9.8	0.98	1.7	3.1	0.12	0.165	0.14	0.12	<0.1	0.32	<0.05	287	8	4
	13.8	<0.3	1.8	160	9.8	1.0	1.7	3.1	<0.05	0.163	0.15	0.10	<0.1	0.30	<0.05	285	8	5
08 ES.TW	7.7	<0.3	5.9	64	0.88	0.81	0.4	0.6	0.15	0.068	<0.05	<0.05	<0.1	0.72	<0.05	32.3	<2	7
	8.3	<0.3	7.3	63	0.88	0.81	0.4	0.6	0.16	0.083	<0.05	<0.05	<0.1	0.70	0.09	33.1	<2	7
02 DF.TW	2.1	<0.3	0.05	48	0.82	0.44	0.2	0.3	<0.05	0.008	<0.05	<0.05	<0.1	0.52	<0.05	22.8	<2	4
	2.3	<0.3	0.05	49	0.84	0.45	0.2	0.4	<0.05	0.008	<0.05	<0.05	<0.1	0.53	<0.05	23.4	2	4

08 W1.TW	1.4	<0.3	0.16	8	0.88	0.51	0.2	<0.3	<0.05	0.005	<0.05	<0.05	<0.1	0.06	<0.05	11.9	<2	8
	1.4	<0.3	0.20	8	0.90	0.44	0.1	<0.3	<0.05	0.005	<0.05	<0.05	<0.1	0.57	<0.05	10.9	<2	8
13b W1.TW	1.8	<0.3	0.38	9	0.25	0.38	0.3	<0.3	<0.05	0.005	<0.05	<0.05	<0.1	0.49	<0.05	9.9	<2	3
	1.8	<0.3	0.42	11	0.25	0.37	0.3	<0.3	<0.05	0.005	<0.05	<0.05	<0.1	0.52	<0.05	9.3	<2	4