



Standard Operating Procedure

Scientific and Analytical Services

Page 1 of 10

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.

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Prepared by: CFG

Document No: SAS
SOP0012

Sample handling and Storage Requirements for Water and Wastewater Samples

1. Purpose

This procedure has been written to provide guidance to persons performing sample handling and storage of water and wastewaters.

2. Scope

This procedure applies all persons performing sample handling and storage of waters and wastewaters.

3. Responsibility

- Water and Waste Services (A directorate of Mackay Regional Council) Scientific and Analytical Services (SAS) are responsible for performing this SOP.
- Persons responsible must be trained in this SOP.

4. Safety

Prepare Job Hazard Analysis (JHA) for all tasks identified to be hazardous. All staff are trained in the Procedure IS-ISPR-210. Obtain a copy of this procedure from the Mackay Regional Council Intranet. Using this procedure fill out the form “Job Hazard Analysis” on the Doc. Code: IS-ISFM-138.

- Wear gloves and follow good hygiene practices when working with sewage samples.
- Avoid working over water where possible.
- Wide Brim Hat, Sunglasses, Sunscreen, Long Pants, and Safety Footwear.

5. References

AS/NZS 5667.1:1998 Water quality – Sampling; Part 1: Guidance on the design of sampling programs, sampling techniques and the preservation and handling of samples.

AS/NZS 2031. Selection of containers and preservation of water samples for chemical and microbiological analysis.

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Standard Operating Procedure

Scientific and Analytical Services

Page 2 of 10

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.

Issue Date: 23/06/2015
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*AS/NZS 2031.2 Part 2: Microbiological
Scientific and Analytical Services Laboratory Methods Manual.
Scientific and Analytical Services Laboratory Calibration and Equipment Manual.
Scientific and Analytical Services Laboratory LIMS Manual.
Scientific and Analytical Services Safety Policies and Procedures Manual.*

6. Definitions

Analyte: The constituent to be analysed

Maximum recommended

holding time: The time from when the sample is taken to when the analytical process is commenced.

Sample: A portion, ideally representative, removed from a specified body of water, wither discretely or continuously, for the purpose of examination of various defined characteristics.

Sampling: The process of removing a sample of a body of water for the purpose of examination of various defined characteristics.

7. Procedure

7.1 Requirements for sampling

The sampling SOPs are used to ensure the correct sample collection vessels are prepared and taken on site with the sampler. Also to ensure all chemicals/preservatives required for sample preservation are taken on site and added to the sample vessel before or after collection.

Containers for general sample collection may be rinsed several times prior to filling with sample to ensure a representative sample is obtained.

For microbiological analyses, sterile sample containers are to be handled using aseptic techniques to ensure no undue contamination. These containers are not to be pre-rinsed.

7.2 Sample Preservation - General Requirements

Waters, waste waters, bottom sediments and sludges are susceptible to change to differing extents as a result of physical, chemical or biological reactions which may take place between the time of sampling and analysis. The nature and rate of these changes are often such that, if the necessary precautions are not taken during sampling, transport and storage, the concentrations determined will be different

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Standard Operating Procedure

Scientific and Analytical Services

Page 3 of 10

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.

Issue Date: 23/06/2015
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from those existing at the time of sampling. These changes are often sufficiently rapid to modify the sample considerably in the space of several hours. Certain constituents should be measured in situ or in the field to obtain accurate results. It should be stressed that, particularly if there is any doubt, the laboratory supervisor should be consulted before deciding on the precise method of handling and preservation.

The extent of these changes is dependent on the chemical and biological nature of the sample, its temperature, its exposure to light, the nature of the container in which it is placed, the time between sampling and analysis and the conditions to which it is submitted, e.g. agitation during transport.

Some more specific causes of variations are as follows:

- (a) Presence of bacteria, algae and other organisms can consume certain constituents present in the samples. They can also modify the nature of the constituents to produce new constituents. This biological activity affects, for example, the concentrations of dissolved oxygen, carbon dioxide, nitrogen compounds, phosphorus and sometimes silicon.
- (b) Certain compounds can be oxidized by the dissolved oxygen contained in the samples or by atmospheric oxygen, (e.g. organic compounds, Fe(II) and sulfides).
- (c) Certain substances can precipitate out, (e.g. calcium carbonate, metals and metallic compounds such as $Al(OH)_3$) or be lost to the vapour phase (e.g. oxygen, cyanides and mercury).
- (d) The pH, conductivity, carbon dioxide content and similar can be modified by the absorption of carbon dioxide from the air.
- (e) Dissolved metals or metals in a colloidal state, as well as certain organic compounds can be irreversibly adsorbed or absorbed by the surface of containers or solid materials in the samples.
- (f) Polymerized products can depolymerize and conversely, simple compounds can polymerize.

Numerous investigations which have been carried out in order to recommend methods which will enable samples to be stored without modification of their composition, but it is impossible to give absolute rules which will cover all cases and all situations. In every case the method of storage must be compatible with the analytical techniques which will be used. One object of Table 1 and Table 2 is to describe the most commonly used preservation techniques. However, there should be no significant difference between the results of a determination carried out immediately and the result obtained after preservation. The analyst should verify, taking into account particularly the method of analysis which is intended to be used, whether the suggestions in Tables 1 and 2 are suitable for the sample concerned. For preservation of microbiological samples, reference should be made to AS 2031.2.

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7.3 Sampling and Storage of Samples for Chemical Analysis

REFRIGERATION OR FREEZING

General

The sample should be kept at a temperature lower than that during filling. Refrigeration or freezing of samples is only truly effective if it is applied immediately after the collection of the samples. This necessitates the use of cool boxes or refrigerators at the sampling site.

Refrigeration

Refrigeration entails the placement of samples in a refrigerator, using crushed ice in a portable cooler or other device to cool the sample to a temperature of between 1°C and 4°C. In most cases, this is sufficient to preserve the sample during the transport to the laboratory and for a relatively short period of time before the analysis. Refrigeration cannot be considered as a means of long-term storage, particularly in the case of waste water samples.

NOTE: For preservation of microbiological samples reference should be made to AS 2031.2.

Freezing

Freezing to a temperature of –20°C allows, in general, an increase in the period of storage. For some analytes, such as nutrients, freezing is the preferred method of preservation.

Do not refreeze samples. Sufficient individual portions should be collected in cases where analyses may be conducted at different times or locations. When thawed, samples should be thoroughly mixed and allowed to reach ambient temperature before any measurements are made.

NOTES:

- 1 Sample containers, whose contents are frozen as part of their preservation, should not be completely filled.
- 2 Glass containers are not suitable for freezing. The use of plastic containers is strongly recommended.
- 3 Quick freezing with dry ice is the most satisfactory procedure.
- 4.



SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.

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SOP0012

USE OF PRESERVATIVES

Certain physical and chemical constituents can be stabilized by the addition of chemical compounds. The chemical compounds can be added either—

- (a) to the container before the sample is taken (the container should not be rinsed with the sample water if this is the case); or
- (b) directly to the sample after it has been taken.

Various chemical compounds, at concentrations equally varied, have been proposed.

Those most commonly used are—

- (i) acids;
 - (ii) basic solutions;
 - (iii) biocides; and
 - (iv) particular reagents, necessary for the specific preservation of certain constituents,
- e.g. the determination of oxygen, total cyanides or sulfides all require a previous fixation of the sample in the field.

Preservatives should be added in the form of concentrated solutions so that addition of only small volumes is necessary. This enables the corresponding dilution to be disregarded in most cases, otherwise dilution of the sample should be taken into account during the analysis and the calculation of the results.

For some determinations, particularly trace element analysis, it is essential to carry out a blank test to take into account possible introduction by the preservatives of an additional amount of the analytes of interest, e.g. acids can introduce significant amounts of arsenic, lead and mercury. It is also essential that the preservatives do not interfere with the analysis.

REAGENTS

General The following reagents are used for preservation of samples and should only be prepared according to individual sampling requirements in Table 1 or Table 2. Unless otherwise specified, all reagents used should be of at least analytical reagent grade and water should be of at least ISO 3696, Type II purity. Acids referred to in this Standard are the commercially available 'concentrated' acids.

Safety considerations It should be remembered that certain preservatives (e.g. acids, mercury (II) chloride, chloroform) need to be used with caution because of the danger involved in their handling. Sampling personnel should be warned of these dangers and the necessary safety procedures.



Standard Operating Procedure

Scientific and Analytical Services

Page 6 of 10

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.

Issue Date: 23/06/2015
Iss.: 4, Rev.: 1

Prepared by: CFG

Document No: SAS
SOP0012

11.4.3 Solids

- 11.4.3.1 Ascorbic acid
- 11.4.3.2 Potassium dichromate (K₂Cr₂O₇)
- 11.4.3.3 Sodium hydroxide (NaOH)
- 11.4.3.4 Sodium iodide (NaI)
- 11.4.3.5 Sodium sulfite (Na₂SO₃)
- 11.4.3.6 Sodium thiosulfate (Na₂S₂O₃)

11.4.4 Solutions

- 11.4.4.1 Zinc acetate solution (10% m/v) Dissolve 10 g of zinc acetate and dilute to 100 mL with water.
- 11.4.4.2 Orthophosphoric acid (r 1.75 g/mL)
- 11.4.4.3 Hydrochloric acid (r 1.16 g/mL)
- 11.4.4.4 Nitric acid (r 1.42 g/mL)
- 11.4.4.5 Sulfuric acid (r 1.84 g/mL)
- 11.4.4.6 Sodium hydroxide solution (40% m/v) Dissolve 40 g of sodium hydroxide and dilute to 100 mL with water.
- 11.4.4.7 Formaldehyde solution (40% v/v).
- 11.4.4.8 Nitric acid solution (50% v/v)
- 11.4.4.9 Sodium hypochlorite solution (10% m/v)
- 11.4.4.10 EDTA solution (2.5% m/v) Dissolve 2.5 g of di-sodium EDTA and dilute to 100 mL with water.
- 11.4.4.11 Copper-DMP reagent Dissolve 0.15 g of 2,9-dimethyl-1, 10-phenanthroline hydrochloride (DMP) in water, add 25 mL of copper sulphate solution (2 g/L) and 125 mL of pH 4.8 buffer solution. Dilute to 250 mL with water.

11.5 EXTRACTION For some organic materials, an initial on-site extraction may be advantageous. Alternative procedures such as on-site adsorption techniques or on-site headspace collection may also be employed where appropriate. Samples should be refrigerated until they are analysed. **There should be minimal headspace.**

11.6 SAMPLING DETAILS FOR INDIVIDUAL DETERMINATIONS

Sample volumes listed in Table 1 and Table 2 represent typical volumes required for an analyst to perform a single determination on the sample. Where more than one method is available for a particular analyte, the sample volumes pertain to the method which requires the maximum sample volume. In some cases, it may therefore be possible to take a smaller volume of sample. Where a preservation procedure requires the addition of acid to a sample, the pH of the raw water should be determined on-site on a separate but representative sample of that water. Additional requirements to those set out in Table 1 and Table 2 may also be necessary depending on the water body sampled, concentration levels present or the analytical methods to be used.

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SOP0012

11.7 FILTRATION OR CENTRIFUGING OF SAMPLES

Suspended matter, sediment, algae and other microorganisms may be removed, either at the time of taking the sample or immediately afterwards, by filtration through filter paper or membrane filter or by centrifuging. Filtration is not applicable if the filter is likely to retain one or more of the analytes. It is essential that the filter is not a cause of contamination and if necessary washed before use in a manner consistent with the final method of analysis.

NOTE: Analysis may require the separation of 'soluble' and 'insoluble' forms by filtration. The pore size of the filter will affect the analyte distribution. 0.45 µm filters are the most commonly accepted.

TABLE 1 TECHNIQUES GENERALLY SUITABLE FOR THE PRESERVATION OF WATER SAMPLES—PHYSICO-CHEMICAL AND CHEMICAL ANALYSIS

ANALYTE	VOLUME REQUIRED (mL)	COLLECTI ON VESSEL	PRESERVATION	TIME SPAN FOR ANALYSIS	MAXIMUM RECOMMENDED HOLDING TIME
Acidity and alkalinity	500	P or G	Refrigerate	24 h	Analyse ASAP, samples should preferably be analysed in the field
Aluminum	500	P or G	Acidify with nitric acid to pH 1 to 2	1 month	
Ammonia	500	P or G	Refrigerate	6 h recommended (with a maximum of 18-24 hours)	Unfiltered sample
			Filter on site (0.45µ) and refrigerate	24 h	
			Filter on site (0.45µ) and freeze	1 month	
Arsenic	100	P or G	Acidify with nitric or hydrochloric acid to pH 1 to 2.	1 month	Hydrochloric acid should be used if the hydride technique is used for analysis
BOD	1000	P or G	Refrigerate and store in dark	Within 24 h	Refrigerate <4°C -24 Hrs

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Issue Date: 23/06/2015
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Document No: SAS
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ANALYTE	VOLUME REQUIRED (mL)	COLLECTI ON VESSEL	PRESERVATION	TIME SPAN FOR ANALYSIS	MAXIMUM RECOMMENDED HOLDING TIME
Bromide	100	P or G	Refrigerate and store in dark	1 month	
Cadmium	100	P or G	Acidify with nitric acid to pH 1 to 2	1 month	
Calcium	100	P	None required	1 week	Acidification permits the determination of calcium and other metals from the same sample
			Acidify with nitric acid to pH 1 to 2 and refrigerate	1 month	
Carbon, total organic (TOC)	100	G, amber with PTFE cap liner	Acidify with phosphoric acid to pH 1 to 2, refrigerate and store in the dark	1 week	Test should be carried out as soon as possible
		P	Freeze	1 month	
Chemical oxygen demand (COD)	100	P or G	Acidify with H ₂ SO ₄ to pH 1 to 2, refrigerate <4°C and store in the dark	1 week	Glass containers are preferable for samples with COD <5mg/L
		P	Freeze	1 month	
Chloride	100	P or G	None required	1 month	
Chlorine -Total & Free	500	P or G	Keep out of direct sunlight	Analyse ASAP	Analysis should be carried out in the field with in 5 minutes of sample collection
Chlorophyll	1000	P or G	Refrigerate	24 h	Transport in the dark
Chromium (total)	100	P or G	Acidify with nitric acid to pH 1 to 2	1 month	
Chromium (VI)	100	P or G	Refrigerate	24 h	

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Issue Date: 23/06/2015
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Document No: SAS
SOP0012

ANALYTE	VOLUME REQUIRED (mL)	COLLECTI ON VESSEL	PRESERVATION	TIME SPAN FOR ANALYSIS	MAXIMUM RECOMMENDED HOLDING TIME
Cobalt	100	P or G	Acidify with nitric acid to pH 1 to 2	1 month	
Colour	500	P or G	Refrigerate and store in the dark	2 d	Storage of 7 d at RT for drinking water
Conductivity	100	P or G	None required	24 h	Test should be carried out in field for samples of <20µS
			Refrigerate	1 month	
Copper	100	P or G	Acidify with nitric acid to pH 1 to 2	1 month	
Detergents					See surfactants
DO	300	P or G	None required	Analyse ASAP	Test should be carried out in field, excessive turbulence should be avoided to minimize oxygen entrainment
Fluoride	100	P	None required	1 month	PTFE containers are not suitable
Hardness					See Calcium
Hydro-carbons	100				See oil and grease
Iron	500	P or G	Acidify with nitric acid to pH 1 to 2	1 month	
Lead	100	P or G	Acidify with nitric acid to pH 1 to 2	1 month	

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.

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Document No: SAS
SOP0012

ANALYTE	VOLUME REQUIRED (mL)	COLLECTI ON VESSEL	PRESERVATION	TIME SPAN FOR ANALYSIS	MAXIMUM RECOMMENDED HOLDING TIME
Magnesium	100	P	None required	1 week	Samples with pH > 8 or high carbonate content to be analysed solely for calcium, magnesium or hardness should
			Acidify with nitric acid to pH 1 to 2, refrigerate	1 month	Acidification permits the determination of magnesium and other metals from the same sample.
Manganese	100	P or G	Acidify with nitric acid to pH 1 to 2.	1 month	
Mercury	100	P or G	Acidify with nitric acid to pH 1 to 2 . Take 50 mL add 50 mL HCL, store at room temperature	1 month	Hydride:
Mercury	100	P or G	Acidify with nitric acid to pH 1 to 2 and add potassium dichromate to give 0.05% (m/v) final concentration.	1 month	Hydride:
Molybdenum	100	P or G	Acidify with nitric acid to pH 1 to 2	1 month	
Nickel	100	P or G	Acidify with nitric acid to pH 1 to 2	1 month	
Nitrate	250	P or G	Refrigerate	24 h	Unfiltered sample
			Filter on site (0.45µ) and freeze	1 month	



Standard Operating Procedure

Scientific and Analytical Services

Page 11 of 10

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.	Issue Date: 23/06/2015 Iss.: 4, Rev.: 1	Prepared by: CFG	Document No: SAS SOP0012
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ANALYTE	VOLUME REQUIRED (mL)	COLLECTI ON VESSEL	PRESERVATION	TIME SPAN FOR ANALYSIS	MAXIMUM RECOMMENDED HOLDING TIME
Nitrite	200	P or G	Immediate analysis		Analyse as soon as possible after collection
			Freeze	2 days	
Nitrogen, total (TN)	500	P or G	Refrigerate	24 h	
			Freeze	1 month	
Nitrogen, total Kjeldahl (TKN)	500	P or G	Acidify with H ₂ SO ₄ to pH 1 to 2 & refrigerate <4°C	24 h	
			Refrigerate	24 h	
			Freeze	1 month	
Oil & Grease	100-120	Glass, solvent washed	Refrigerate	24 h	Do not pre-rinse container with sample
			Acidify with H ₂ SO ₄ to pH 1 to 2 & refrigerate	1 month	
Pesticides	1000 - 3000	G, solvent washed with PTFE Cap liner	Refrigerate	7 d	Do not pre-rinse container with sample
pH	100	P or G	Refrigerate	6 h	Analyse ASAP or Test should be carried out in field
Phosphate, dissolved	50	P or G	Filter on site (0.45µ) and refrigerate	24 h	
			Filter on site (0.45µ) and freeze	1 month	
Phosphorus, total	500	P or G	Refrigerate	24 h	
			Freeze	1 month	

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.

Issue Date: 23/06/2015
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SOP0012

ANALYTE	VOLUME REQUIRED (mL)	COLLECTI ON VESSEL	PRESERVATION	TIME SPAN FOR ANALYSIS	MAXIMUM RECOMMENDED HOLDING TIME
			Acidify with H ₂ SO ₄ to pH 1 to 2 & refrigerate <4°C	1 month	
Potassium	100	P	None required	1 month	Acidification permits the determination of potassium and other metals from the same sample.
			Acidify with nitric acid to pH 1 to 2, refrigerate	1 month	
Radio-nuclotides	1000	G	Refrigerate	24 h	
Selenium	500	P or G	Acidify with nitric or hydrochloric acid to pH 1 to 2	1 month	
Sodium	100	P	None required	1 month	Acidification permits the determination of potassium and other metals from the same sample.
			Acidify with nitric acid to pH 1 to 2, refrigerate	1 month	
Solids, dissolved	500	P or G	Refrigerate	24 h	
Solids, suspended	500 or 1000	P or G	Refrigerate	24 h	
Solids, total	500	P or G	Refrigerate	24 h	
Solids, volatile	500	P or G	Refrigerate	24 h	
Sulfate	200	P or G	Refrigerate	1 week	

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.

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ANALYTE	VOLUME REQUIRED (mL)	COLLECTI ON VESSEL	PRESERVATION	TIME SPAN FOR ANALYSIS	MAXIMUM RECOMMENDED HOLDING TIME
Sulfide, total	500	P or G	None required		Test should be carried out in field
Surfactants (anionic)	500	G, rinsed with methanol	Add 40% (v/v) formaldehyde solution to give 1% (v/v) final concentration and refrigerate	1 month	Glassware must not be detergent washed
SVOCs	100	Amber G, and preservative	Refrigerate	7 d	Do not pre-rinse container with sample
Tri-halomethanes (THMs)	40 x 2	Amber G, solvent washed with PTFE Cap liner and preservative	Refrigerate	7 d	Do not pre-rinse container with sample
Turbidity	100	P or G	None required	24 h	Test should be carried out in field
Zinc	100	P or G	Acidify with nitric acid to pH 1 to 2, refrigerate	1 month	

Notes: P = plastic containers, eg polyethylene, PTFE, polypropylene, PET and similar; G = borosilicate glass container. Refrigerate = cool to between 1°C and 4°C.

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.

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SOP0012

TABLE 2 TECHNIQUES GENERALLY SUITABLE FOR THE PRESERVATION OF WATER SAMPLES—RADIOCHEMICAL ANALYSIS

Determinand	Type of container (See Note 1)	Typical volume, mL (See Note 2)	Filling technique Preservation procedures (See Notes 3 and 4)	Maximum recommended holding time	Comment
Alpha and beta activity (gross)	P or G	1 000 (ALS with red & green stripe)	Fill container completely to exclude air. Acidify with nitric acid to pH 1 to 2	1 month	
Uranium	P	Sample volume of between 1 and 5 L	Acidify with nitric acid to pH less than 1	2 weeks	

WARNING: IT IS ESSENTIAL THAT RADIOACTIVE DUST IS NOT INHALED OR LEFT ON THE BODY OR CLOTHING.

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.	Issue Date: 23/06/2015 Iss.: 4, Rev.: 1	Prepared by: CFG	Document No: SAS SOP0012
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7.4 Sampling and Storage of Samples for Microbiological Analysis

ANALYSIS	VOLUME REQUIRED (mL)	COLLECTION VESSEL	PRESERVATION	TIME SPAN FOR ANALYSIS	MAXIMUM RECOMMENDED HOLDING TIME
<i>Coliforms, total</i>	220	Sterile P	If residual chlorine is present, add Na ₂ S ₂ O ₃	6 h	Refrigerate <4 °C -24 Hrs
<i>Coliforms, faecal</i>	220	Sterile P	If residual chlorine is present, add Na ₂ S ₂ O ₃	6 h	Refrigerate <4 °C -24 Hrs
<i>Coliforms, E.coli</i>	220	Sterile P	If residual chlorine is present, add Na ₂ S ₂ O ₃	6 h	Refrigerate <4 °C -24 Hrs
<i>Heterotrophic Plate Count</i>	220	Sterile P	If residual chlorine is present, add Na ₂ S ₂ O ₃	6 h	Refrigerate <4 °C -24 Hrs
<i>Pseudomonas</i>	500	Sterile P	Addition of Na ₂ S ₂ O ₃ is not required	6 to 12 h	Refrigerate <4 °C -24 Hrs

Notes: P = plastic containers, eg polyethylene, PTFE, polypropylene, PET and similar; G = borosilicate glass container. Refrigerate = cool to between 1°C and 4°C. Na₂S₂O₃ Solution 0.1 mL of 3% solution for drinking water samples – in 220 mL will give a final concentration of 9 mg/L in the sample and neutralize up to 2.5 mg/L residual chlorine.

Start microbial examination of a water sample promptly after collection to avoid unpredictable changes. If samples cannot be processed within 1 h after collection, use an iced cooler for storage during transport to the laboratory.

Hold temperature for all stream pollution, drinking and waste water samples is below 10 °C. Refrigerate samples on delivery to the laboratory and commence analysis within 6 h. A maximum recommended holding time is 24 h.

The laboratory is to store the samples refrigerated until ready to analyse, there is no requirement to room to room temperature prior to analysis.

8. Documentation

Form SAS – 0003016 Checklist of Equipment and Chemicals for Sampling Waste Water

Form SAS – 0003017 Checklist of Equipment and Chemicals for Sampling of Drinking Water



Standard Operating Procedure

Scientific and Analytical Services

SAS SOP0012 – Sample handling and Storage Requirements for Water and Wastewater Samples.

Issue Date: 23/06/2015
Iss.: 4, Rev.: 1

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Document No: SAS
SOP0012

- Form SAS – 0003018 Checklist of Equipment and Chemicals for Sampling Environmental Waters – Ambient/Event
- Form SAS – 0003019 Checklist of Equipment and Chemicals for Sampling of Seaforth Camping Area – Ground Water and Septics
- Form SAS – 0003038 Checklist for Landfill Bore Sampling.

9. Reference Personnel

Changes to this document should be referred to:

- Principal Scientist, SAS

10. Document History

Date	Revision Number	Revised by	Authorised by
24/08/2010	Issue 3; Rev 0	Christine Galea	Stuart Boyd
27/07/2013	Issue 3; Rev 2	Christine Galea	Stuart Boyd
23/01/2015	Issue 4 Rev 0	Christine Galea	Christine Galea

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