SAMPLING AND ANALYSIS OF WATERS, WASTEWATERS, SOILS AND WASTES

CONTENTS

1 INTRODUCTION

Environmental samples are analysed for a range of purposes including meeting statutory requirements of the *Environment Protection Act 1970* and the *Pollution of Waters by Oils and Noxious Substances Act 1986*.

It is important to obtain samples that faithfully represent a waste or element of the environment from which they are taken. Care must be taken in the field to ensure samples are not contaminated during collection, and analyte concentrations do not change between the time of collection and analysis.

Steps needed in any environmental monitoring program should include, but are not limited to:

1. determining the objectives of the monitoring program
2. selecting and accurately analysing chemical, physical or biological indicators which are relevant to the objectives of the monitoring program
3. selecting the appropriate sampling equipment
4. mapping out the location and site to determine the number and type of samples needed
5. obtaining a representative sample or samples
6. accurately recording site observations and measurements
7. appropriate labelling, preserving, storing and transporting of sample for analysis
8. reporting results accurately and completely
9. providing informed interpretation.

Since it is not possible to address all issues that can arise in the field, advice may be needed from specialists including statisticians, chemists, microbiologists or hydrogeologists on the behaviour of a pollutant in different elements of the environment.

1.1 Using this guide

This Guide provides general direction on appropriate sampling, preservation, storage, analytical and quality assurance procedures. It should be used for environmental monitoring programs, assessments, risk management, investigations and audits. The target audience for this publication includes, but is not limited to:

- laboratories
- environmental consultants
- licence holders
- custodians of waste/sites containing waste.

While specific roles of parties involved with the sampling/analysis of wastes are not within the scope of this guide, such parties are expected to have a level of expertise enabling them to adequately carry out relevant tasks required within the context of this document.

This document covers waters (including groundwaters and wastewaters), wastes and soils, but not biota. It must be used for analyses for the purposes of the *Environment Protection Act 1970* and the *Pollution of Waters by Oils and Noxious Substances Act 1986*, unless other procedures are approved by EPA Victoria.

This guide is also a companion publication to *A Guide to the Sampling and Analysis of Air Emissions* (EPA publication 440).

People undertaking sampling must operate within a system accredited by the National Association of Testing Authorities (NATA) or they must meet the following requirements:

This guidance forms part of the Industrial Waste Resource Guidelines (IWRG), which offer guidance for wastes and resources regulated under the *Environment Protection (Industrial Waste Resource) Regulations 2009* (the Regulations). Publication IWRG701 – June 2009.

- They must have had hands-on training with an appropriate body experienced in sampling. They must have demonstrated knowledge and ability to safely take, preserve, store and transport samples within the requirements of this document. This includes refresher training, with records kept on the nature and frequency of the training provided.
- The laboratory conducting the analysis must provide appropriately prepared sample containers and preservatives, for the analytes of interest.
- Satisfactory sampling records must be prepared and maintained by samplers, so that laboratory results can be linked back to the date, time and location of the sample collection.

1.2 Planning a sampling program

No single method applies to all monitoring and assessment needs. The design of a successful sampling strategy depends on determining the objectives and the hypothesis to be tested. Wherever possible, an objective should be expressed as a statistically testable hypothesis.

Any sampling program needs to be based on a good understanding of the spatial and temporal distribution of the indicator and its physico-chemical behaviour in the environmental element being investigated. Statistical methods should be employed to ensure that the selected sampling locations and timing represent both indicator behaviour and the discharge or study area, so that spatial and temporal attributes are correctly represented.

For elements of the environment where a pollutant's distribution is not homogeneous, a good understanding of the factors that affect this distribution will assist in developing a statistical basis for obtaining representative samples. For example, the spatial distribution of a pollutant could be affected by spot spills onto soils. In the case of water bodies, understanding the vertical stratification in large water bodies and the effects of mixing in flowing streams, may be important in characterising them.

Temporal attributes of an environment indicating variations in time should be accurately characterised by the selected sampling strategy. Examples of temporal variations include changes in industrial processes over a periodic cycle that affect effluent quality and storm events where short-term peak stormwater pollutant concentrations enter natural waterways.

Composite sampling (collected samples are mixed to give an 'average' concentration) is also a useful screening tool that can represent study areas or flows that are heterogeneous in space or time. This may be unsuitable for detecting 'hot spots' because polluted single samples may become diluted, resulting in the 'hot spot' being undetected.

Some pollutants, e.g. oil which floats on still water, do not mix with the surrounding matrix. If the objective is to quantify its concentration, it may be difficult taking a representative volume of the water body. In such cases, the impact may be governed by the area covered, which needs to be estimated in the field. However, if the objective is to characterise the nature of the oil, then skimming the oil off the water surface will be sufficient.

When sampling wastes stored in a drum or other storage container, it should not be assumed that the contents of the drum are homogeneous; the sampling strategy should account for the nature and quantities of any distinct liquid or solid layers in the container.

If a program objective requires pollutant loads to be calculated, then accurate flow, volume or mass measurement will be required at the sampling point.

The analytical method to be used will be determined by detection limits and the precision required.. For example, ambient heavy metal concentrations in seawater will be in the part per billion range or lower, while determining heavy metals in polluted sludge will be many orders of magnitude higher. In some instances inexpensive screening tests may be acceptable, while in other programs a high level of accuracy will be required.

2 SAMPLE COLLECTION

Various physical, chemical and biological processes can affect a sample from the time of collection to that of analysis. The use of appropriate sampling equipment, containers and preservation methods to maintain sample integrity will prevent/minimise these effects. Samples must also be analysed within stipulated holding time limits.

Care is required to avoid contamination of the sample during sampling, handling and transport to the laboratory.

2.1 Health and safety precautions

Relevant risk assessments and occupational health and safety protocols need to be followed when handling wastes in the field or laboratory. Details of these are not provided in this guide. It is assumed that the wastes handler will be competent in this area and that these details have been provided by the relevant employer or from resources such as standards for a given procedure. Any personal protective equipment (PPE) required must be used by people having adequate experience and knowledge in their use.

Precautions taken and protective equipment and clothing used should be reflected by the associated level of risk. When in doubt, assume the worst case outcome will occur.

2.2 Sampling devices

Sampling devices should be made from materials that have minimum interaction with, and do not contaminate or disturb the sample.

They need to be appropriately cleaned between samples. In some cases, it may be necessary to collect the final rinsate for analysis to demonstrate that the sampling device has been sufficiently cleaned to avoid potential errors in results due to cross contamination.

2.3 Sample containers

Containers, which are usually glass, polyethylene, polypropylene or a fluoropolymer (e.g. PTFE), are selected according to their lack of interaction with analytical parameters. For example, glass is suitable for samples containing trace organics, as leaching and adsorption are minimal, but is unsuitable for sampling most trace inorganics because active sites on its surface can bind inorganic ions.

Containers must be clean and may need to be retained and submitted to the laboratory for analysis as a blank. Where reagents are added during the preservation step, a sample of the added reagents must also be submitted to the laboratory for analysis as a reagent blank.

2.4 Sampling waters

Where very low ambient concentrations are expected, nothing should be in contact with the insides of containers, lids and collection vessels, to avoid/minimise contamination.

When sampling for volatile species, to avoid losses, the sample vial/bottle should be filled gently to reduce agitation that might drive off volatile compounds. Such samples should be immediately cooled (on ice) in transit to the analysing laboratory.

Phase separated materials such as hydrocarbons and other organic contaminants should be identified as measurable separate layers, or observable sheens.

2.4.1 Sampling surface waters

For well mixed waters, a sample taken 100 mm below the surface, away from the edge, may be adequate. Deep and stratified waters may require special devices (such as a Van Dorn sampler) and careful handling techniques for unstable chemical species. A hand or power-driven pump with an extended inlet tube may also be useful to draw water from selected depths.

When sampling shallow waters, contamination from disturbed sediment should be avoided by using an extended inlet of thin tube on the sample bottle and drawing water in by suction. To collect a sample of the surface layer, the container should be held horizontally in the water, half submerged. To collect a sample of water beneath a surface layer, a syringe or other device with an extended inlet tube that is capable of piercing the surface layer, may be appropriate, depending on the thickness of the surface layer.

2.4.2 Sampling groundwaters

Groundwater sampling should be undertaken in accordance with *Groundwater sampling guidelines* (EPA publication 669, 2000).

Regular testing of groundwater quality is usually done from monitoring bores. These bores should be constructed according to the guidelines of the Agriculture and Resource Management Council of Australia and New Zealand (ARMCANZ 2003).

2.4.3 Sampling a waste discharge

The most representative waste discharge sample is from a point where the effluent is thoroughly mixed and close to the discharging premises' outlet. For a licensed discharge, a sampling point will normally be described in the licence where samples must always be taken.

2.4.4 Use of automatic samplers

The probe for automatic samplers should be placed sufficiently far from both the surface and bottom of the water body to avoid samples being affected by the air/water or sediment/water interface.

2.5 Sampling soils

Sampling and analysis plans should be devised in accordance with the requirements of the *NEPM Guideline on Laboratory Analysis of Potentially Contaminated Soils* (NEPC latest version) and/or a comparable publication.

Before sampling, vegetation and other non-soil material (including rocks and concrete) should be removed. This removed material may be subsequently characterised if necessary.

When sampling soils for volatile contaminants, precautions must be taken to prevent evaporative losses as detailed in AS 4482.2 (1999).

Collection of samples should be accomplished with minimal disturbance, using a coring device. Core soil samples should either be immersed in methanol in the field or placed in vials that will also act as a purge vessel in the laboratory, providing more accurate results than placing samples in jars (USEPA 1991).

If the soils to be sampled are suspected of being acid sulfate or potential acid sulfate soils, EPA Victoria's *Acid sulfate soil and rock* (EPA publication 655.1, 2009) and/or Australian standards AS4969.0 (2008) to AS4969.14 (2009) should be consulted for details on their sampling and handling.

For details of the sampling and determination of asbestos in soil Australian standard AS4964 (2004) may be consulted.

When sampling from a test pit, samples should be taken from the lowest point first to prevent cross contamination from other sampling points.

2.6 Sampling sediments

The best locations for sampling sediments are where fine materials accumulate. These are generally confined to areas where there is little or no flow.

For organic and inorganic analyses, sampling devices should be constructed from metal and plastic respectively.

Where there is a lack of fine sediment, more than one scoop or grab sample may be necessary to obtain a sufficient amount of material.

2.7 Sampling wastes

Sampling wastes can be difficult if the wastes are heterogeneous, contain many different types of waste, or the contamination is not evenly distributed. In these circumstances, it can be useful to keep different types of waste separate (for example by separating the phases of a multi-phase waste), or to separate different portions that contain high levels of contaminants. General guidance on sampling can be obtained from *Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sample Correctness and Statistical Process Control* (Pitard, 1993) or *Sampling for Analytical Purposes* (Gy 1999)

Liquid wastes should be handled according to methods for sampling waters, while waste soils should be treated according to the guidelines for soils above.

For solid wastes with particle sizes greater than soils, or non-uniform particle sizes, Australian Standard 1141.3:1996, (Standards Australia, 1996) may be relevant in some cases. Wastes containing biosolids should be handled and treated according to the procedures listed for liquid and solid wastes (Table 3).

2.8 Preserving samples

Since samples must be chemically/physically preserved as soon as possible after sampling to avoid/minimise biological, chemical or physical changes that can occur between time of collection and analysis..

2.8.1 Freezing

Water and soil samples should be frozen in amounts needed for tests that are to be carried out at a given time to avoid repeated thawing and re-freezing if the total analysis is spread over a period of time.

For liquid samples, provide sufficient air gaps in containers to allow for expansion during freezing.

Thawed samples must be mixed and allowed to reach an ambient temperature before analysis.

2.8.2 Cooling

Samples that require cooling should be stored under ice in transit and then refrigerated after arriving at the laboratory.

2.8.3 Acidification

Acidification of water samples (pH < 2) preserves most trace metals and reduces precipitation, microbial activity and sorption losses to container walls. The acid used (analytical grade, low metal content) must be included in the blank(s) to be analysed in the laboratory. For groundwaters and dissolved metals in water samples, acidification should only be carried out on filtered samples.

2.8.4 Reagent addition

Reagents (high grade) may be added to samples to chemically preserve the analytical parameter. Again blanks of these should be provided to the laboratory, so that contamination levels can be checked. Such reagents should not interfere with an analysis, e.g. cannot use nitric acid (HNO_3) when testing for nitrates (NO_3^-).

2.8.5 Solvent extraction

When a solvent is used to extract analyte from a matrix, e.g. organic pollutants such as hydrocarbons, polycyclic aromatic hydrocarbons (PAHs) and some pesticides, solvent samples should also be submitted as a blank for analysis.

2.8.6 Field filtration

Filtration of water samples in the field may be required in the following circumstances:

- where organic and inorganic contaminants adsorb onto suspended matter in water
- where dissolved contaminant levels or contaminants associated with suspended matter need to be determined.

Filtering should occur immediately after sample collection.

Filters/filtering devices must be clean and should be provided to the laboratory to determine their blank levels. On-site (between samples) final rinses from filtration equipment should also be submitted to the laboratory as 'rinsate blanks'.

2.8.7 Preserving soil samples

Moisture in soil samples can accelerate microbial action changing the concentration of some contaminants present. In these circumstances, it is recommended to store the soil refrigerated (≤ 6°C).

2.9 Labelling and logging

Samples should be securely labelled with a unique sample number at the sampling site.

Sample logs and/or submission sheets must show all relevant information, including location, time and details of any sample pre-treatment.

A submission sheet and chain of custody (COC) form must accompany all samples submitted to the laboratory to ensure sample traceability.

2.10 Transporting samples

Samples should be securely transported to the analysing laboratory as soon as possible after collection. Refer to the appendices for guidance on holding times.

If there is concern that contamination has occurred, the sample and container should be discarded and a fresh sample collected.

SAMPLE HANDLING AND PREPARATION CHECKLIST

- ❑ Determine precautions to be taken in the field.
- ❑ Observe all safety precautions during sampling, in particular taking care to avoid contact with contaminated samples.
- ❑ Ensure sample container selection, preservation procedures and holding times stipulated here are followed.
- ❑ Where reagents are added to the sample or the sample is filtered, ensure that blanks are collected for analysis.
- ❑ Ensure samples are not contaminated in the field, or in transit and are secured during transport to avoid damage.
- ❑ Complete the identification and description of sample on the submission sheet, including any treatment of the sample undertaken in the field.
- ❑ Transport sample(s) to laboratory as soon as possible
- ❑ Preserve and/or analyse samples as soon as possible

3 ANALYTICAL METHODS AND QUALITY ASSURANCE PROCEDURES

3.1 Approved laboratories

Only NATA-accredited laboratories should perform analyses of all tests conducted. Especially for statutory purposes (*Environment Protection Act 1970* or the *Pollution or Waters by Oils and Noxious Substances Act 1986*) unless permission is given by EPA Victoria to use a non-accredited laboratory.

3.2 Approved analytical methods

Only analytical methods recommended here, or those which are validated and shown to be proficiently equivalent for each environmental matrix may be used.

For statutory testing, methods not based on any of the methods in the approved references can only be used with prior approval of EPA Victoria. Validation of the proposed procedure must be demonstrated before approval can be granted.

For all methods used, the laboratory needs to demonstrate that it can accurately analyse for the relevant analytes, in the types of environmental samples, and in the concentration range normally encountered. This can be done by either:

- proficiency tests

or

- checking against standard reference materials (SRM), certified reference materials (CRM) or spike recovery.

It is also necessary to determine the precision (reproducibility and repeatability), selectivity, limits of detection, linearity and concentration ranges of a method.

Procedures that should be followed for method validation and verification are available in *Guidelines for the Validation and Verification of Chemical Test Methods* (NATA Technical Note No. 17; NATA 2009).

3.3 Limits of detection and reporting

The limit of detection is defined as the lowest concentration of an analytical parameter in a sample that can be detected, but not necessarily quantified. The limit of reporting (also known as the 'limit of quantitation') is defined as the lowest concentration of an analytical parameter that can be determined with acceptable precision and accuracy. In practice, the limit of reporting is frequently taken to be ten times the limit of detection (NATA, 2009). However, some laboratories may use limits of reporting that are five times the limit of detection (APHA 2005).

Details for establishing limits of detection and reporting can be found in NATA's Technical Note No. 17 (2009).

3.4 Waters, wastewaters and groundwaters

For waters, wastewaters and groundwaters, methods selected from the standard references listed below* should be used.

1. American Public Health Association (APHA) 2005, *Standard Methods for the Examination of Water and Wastewater*.
2. US Environmental Protection Agency SW846 on-line, *Methods for Chemical Analysis of Water and Wastes*, www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm#table
3. American Society for Testing and Materials (ASTM), *Water and Environmental Technology*.
4. US Environment Protection Agency 1978, *Microbiological Methods for Monitoring the Environment, Water and Wastes*.
5. Department of the Environment 1994, *The Bacteriological Examination of Drinking Water Supplies*, Report on Public Health and Medical Subjects, No. 71, Method for the Examination of Waters and Associated Material.
6. Relevant Australian standards.
7. Relevant ISO standards

3.4.1 Trace analysis

Publications such as USEPA Method 1669 (1996b) should be used for details of sampling and analysis of waters at trace levels ($\leq \mu g/L$). For guidance on the installation and use of clean rooms and clean workstations relevant to this, Australian Standards 1386.5-6 (Standards Australia 1989) may also be consulted.

Trace level analysis methods for seawaters can be obtained from either *Methods of Seawater Analysis* (Grasshoff 1983), *A Manual of Chemical and Biological Methods for Seawater Analysis* (Parsons 1989) or *A Practical Handbook of Seawater Analysis* (Strickland 1974).

3.4.2 In situ measurements

Common *in situ* measurements include:

- pH
- temperature
- turbidity
- dissolved oxygen
- conductivity
- some ions, e.g. fluoride (F^-) and sulfide (S^{-2}) (using ion selective electrodes).

Manufacturers' instructions are the best guide for the use of any particular field instrument which must

* The latest editions of these references at the time of publishing this *Guide* are referenced. Where they are superseded, the most recent edition should be used.

always be correctly calibrated. For continuous monitoring, any calibration regime must be based on a sound knowledge of the nature of the effluent stream. Further guidance of this may be found in *Process Instruments and Controls Handbook* (Considine 1985) or a more recent equivalent publication.

3.4.3 Radioactivity measurements

Suitable methods for the measurement of gross radioactivity can be found in international standards ISO 9696 (2007) and ISO 9697 (2008). For measuring radioactivity in soils, use the methods included in *Eastern Environmental Radiation Facility Radiation Procedures Manual* (Lieberman 1984).

3.5 Soils and sediments

For the analysis of soils, NEPM Schedule B(3) *Guideline on Laboratory Analysis of Potentially Contaminated Soils* (NEPC, most recent) or US EPA SW846 on-line *Test Methods for Evaluating Solid Wastes: Chemical/ Physical Methods* (www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm#table) should be followed.

As previously mentioned, for the analysis of acid sulfate soils or potential acid sulfate soils, EPA's *Acid sulfate soil and rock* (EPA publication 655.1 2009) and/or AS 4969.0 (2008) to AS 4969.14 (2009) should be consulted.

Relevant codes of practice, published as part of the EPA Best Practice Environmental Management Series, contain details of tests to be used to determine soil permeability. For example, the requirements for testing soil percolation rates for septic tank installations are given in *Code of practice – Onsite wastewater management* (EPA publication 891 2008). In the absence of a relevant code of practice, refer to American Society for Testing and Materials (ASTM) D5126–90 (2004) and Australian Standard 1289.6.7.3 (1999).

As for waters, a range of *in situ* measurements may be appropriate for characterising soils, for example, field soil gas measurements e.g. a photo-ioinisation analyser.

3.6 Wastes

Procedures to determine total concentrations of a range of contaminants in wastes are listed in USEPA SW-846 On-Line.

Other waste characteristics which may have an environmental impact also need to be measured and are described in the following sections.

3.6.1 Leachability and leachates

Leachable organics (volatile and semi-volatile), metals and anions (except cyanide) may be determined using the Australian Standard Leaching Procedure (ASLP) as per Australian Standards 4439.2 and 4439.3 (Standards Australia 1997a & b).

Alternatively, the Toxicity Characteristic Leaching Procedure (TCLP) (USEPA method 1311, (1992), USEPA, SW-846 on-line is available for such use if permitted.

The difference in the two is that the former has a wider range of leaching reagents allowed. All methods are designed to simulate leaching conditions in the environment to determine available pollutants. The leaching reagent should be chosen according to the environmental conditions the wastes are, or will be, exposed to.

Leachable cyanide may be determined by Method 1312, the Synthetic Precipitation Leaching Procedure (USEPA 1994) or by leaching with distilled or de-ionised water, using the methods in AS4439.3 (1997b).

Collected leachates should be analysed using methods listed for waters and wastewaters.

3.6.2 Flammability and ignitability

Flammability of liquid wastes may be assessed according to ASTM Method D3278–96 (2004a)e1 (small scale closed cup apparatus).

'Ignitability' is when a waste burns when ignited. This characteristic can be measured using USEPA Method 1030 (1996a).

3.6.3 Corrosivity

'Corrosivity' is defined as the ability of a substance to attack human skin or plant and equipment. Often this is due to extreme acidity or alkalinity so waste pH is normally tested. To measure corrosivity of a waste towards steel, USEPA Method 1110A, 'Corrosivity toward Steel' (USEPA 2004) may be used.

3.6.4 Free liquid determination

Free liquid may be determined using USEPA Method 9095B (2004): '*Paint Filter Liquids Test*'

3.7 Volatile contaminants in soils and wastes

As samples for volatile analysis cannot be taken from thoroughly homogenised bulk samples, these may not necessarily be representative of the whole material. A sufficient number of samples need to be taken to confidently obtain an accurate measure of average concentrations.

Volatile components should be determined using the 'purge and trap', procedure. Methods involving measurement of headspace concentrations may be less rigorous and should only be used as a screening tool. Refer to methods outlined in *USEPA SW-846 on-line* and/or AS 4482.2 (1999) for both of these procedures.

3.8 Qualitative analysis

References, such as *Spot Tests In Organic Analysis* (Feigl and Anger 1966), *Spot Tests in Inorganic Analysis* (Feigl and Anger 1972) and *Vogel's Qualitative Inorganic Analysis* (Vogel 1996) are a useful qualitative analysis resource.

For solid materials having a limited solubility, x-ray diffraction (XRD) analysis may provide useful information on the identity of compounds present in the sample. However, XRD has some limitations with only crystalline substances giving an XRD response.

3.9 Toxicity screening testing

Microtox® (Hinwood 1990), or another proficiently equivalent screening technique is recommended as a screening toxicity test.

3.10 Quality assurance

A laboratory quality assurance system is a requirement of NATA accreditation. Laboratories should seek to constantly assess their competence by participating, whenever possible, in inter-laboratory proficiency programs. Additional details on quality assurance and quality control are presented in Appendix E.

Analysts receiving samples need to ensure that they were collected in appropriate containers and they have been preserved in a manner recommended in this guide. A statement should be included in the report detailing any deviations from these requirements.

4 REPORTING AND REVIEW OF RESULTS

4.1 Analytical reports

The analytical report must have sufficient information for the end user to make a critical evaluation of its contents. This report format must also comply with NATA requirements.

Information typically reported for each parameter determined, provided by the person taking the sample or the laboratory, should include:

- sample identification (e.g., description, location, sample number and unique laboratory number)
- date and time of sampling
- field observations and *in situ* measurements
- field pre-treatment sample preservation procedures, if any
- reference to analytical method used.
- date of analysis
- accurate description of the parameter
- results
- notations of any deviation from recommended sampling or analytical procedures.

The limit of detection for each analyte should be quoted with quantitative test results. Concentrations below the limit of reporting should be quoted as a 'less than' (<) figure. The mean uncertainty (MU) of results should also be reported.

Results are typically reported in the following concentration units:

- mg/L or µg/L in liquids
- mg/kg or µg/kg in solids, and
- organisms/100 mL for bacterial organisms in liquids.
- For radioactivity measurements–
  - Bq/L for liquids
  - Bq/g for solids.

A statement of the spike recovery achieved, providing information on the quality of the test result, should also be reported.

4.2 Reviewing data

When reviewing data, as a general rule, duplicates should agree within 10 to 20 % of each other and spike recovery values should be between 80 and 120 %.

If monitoring is being undertaken as a discharge licence condition, then whenever the licence emission limits are exceeded, the breach should be reported immediately to EPA Victoria, in accordance with the licence conditions. The reasons leading to the breach and action taken to ensure future licence compliance should be included in this report.

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APPENDICES

APPENDIX A: WATERS, GROUNDWATERS AND WASTEWATERS – CONTAINERS, PRESERVATION AND HOLDING TIMES

Sample containers and their preparation

Selection and preparation of containers, sample pre-treatment, preservation of samples in transit and subsequent holding times and storage conditions must comply with this Appendix, which is based on information sourced from AS/NZS 5667.1:1998*, USEPA SW846 on-line (www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm#table), USEPA title 40 of the Code of Federal Regulations (CFR) Chapter 1(Environment Protection), subchapter D-water programs, Part 136.3-identification of test procedures (USEPA 2007), APHA (2005), ISO 5667-3:2003(E) and Rayment & Higginson (1992). Typical volumes listed here are for a single determination, and should only be used as a guide. To determine very low concentrations that may be present in uncontaminated samples, larger volumes may be required. Typical volumes are dependent on the analytical method used, and the analyst should be consulted on their requirements prior to sampling. Unless otherwise stated, the requirements listed are those for quantitative determinations. Containers and all sampling equipment should be clean and free from relevant contamination. Temperature of samples when taken should be recorded, as well as transport conditions and preservation and storage conditions.

Note 1#: These recommendations are only a guide. Selecting sample and digestion volumes, preservation procedures and holding times and conditions should be based on the nature of the sample, the intended end use of the data and the data quality objectives. Alternative storage conditions may be acceptable as long as analyte stability within a matrix that does not compromise data quality objectives can be demonstrated.

Note 2: In a given sample, the analyte requiring the most preservation treatment as well as the shortest holding time should dictate the preservation treatment of sample overall

Note 3:

- Preservation procedure refers to treatment of sample after collection, either in transit or upon arrival to the laboratory.
- Holding time is the recommended maximum period from sample collection until sample analysis.

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* *EPA is grateful to Standards Australia for the permission to reproduce information presented in AS/NZS 5667.1:1998 on which this Table is partially based.*

\# *US EPA SW-846 on-line, http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm#table*

Table 1: Waters, groundwaters and wastewaters: container types, transport, preservation and sample holding times

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
Acidity and alkalinity	Polyethylene, PTFE or borosilicate glass	500	Fill bottle to exclude air. Transport under ice		Recommend 24 hours, but. 14 days acceptable	Refrigerate (≤ 6°C)	Samples should preferably be analysed in the field, particularly if they contain high levels of dissolved gases.
Ammonia	Polyethylene, PTFE or glass	500	Transport under ice	Filter sample on site (0.45 µm cellulose acetate membrane filter). Acidify with sulfuric acid to pH < 2, or freeze upon receipt by laboratory	Analyse within 24 hours Up to 28 days acceptable	Refrigerate (≤ 6°C) Refrigerate (≤ 6°C) if acidifying, otherwise freeze (- 20 °C)	Pressure filtering is preferred.
Anions: • bicarbonate (HCO_3^-) • carbonate (CO_3^{-2}) • chloride (Cl^-) • sulfate (SO_4^{-2})	Polyethylene, PTFE or borosilicate glass	100	Fill bottle to exclude air. Transport under ice		28 days. For HCO_3^- & CO_3^{-2}, recommend 24 hours, but 14 days acceptable.	Refrigerate (≤ 6°C)	
Bacteria: • Coliforms (total) • *E. coli* • *Enterococci*	Sterile polyethylene or glass, and containing pre-sterilised sodium thiosulfate ($Na_2S_2O_3$)	500	Allow > 2.5 cm headspace (for mixing). Do not rinse container before taking sample Transport under ice	0.0008% $Na_2S_2O_3$	6 hours	Cool (≤ 10°C)	Samples should preferably be analysed as soon as possible. In exceptional circumstances, such as sampling in a remote location, a holding time of up to 24 hours is acceptable.
Biochemical oxygen demand (BOD) Carbonaceous biochemical oxygen demand (CBOD)	Plastic or glass (preferably amber glass)	500	Fill bottle to exclude air. Transport under ice away from light		48 hours	Preferable to analyse as soon as possible. Otherwise, refrigerate (≤ 6°C) in the dark.	Do not pre-rinse container with sample. Glass containers should be used for samples with low BOD (<5 mg/L). Nitrification inhibition is not to be implemented when performing the BOD test unless CBOD is required.

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
Boron	PTFE or quartz	100	Fill bottle to exclude air. Transport under ice.	Acidify with nitric acid to pH < 2.	28 days preferred Up to six months allowed.		
Bromate	Polyethylene, PTFE or glass	100	Transport under ice.	50 mg/L ethylene diamine (EDA)	7 days 28 days if preserved with EDA	Refrigerate (< 6°C)	
Bromide	Polyethylene, PTFE or glass	500	Transport under ice		28 days	Refrigerate (< 6°C)	
Bromine (residual)	Polyethylene or glass	500	Transport under ice away from light		24 hours	Refrigerate (< 6°C) in the dark	Should analyse as soon as possible. Samples should be kept out of direct sunlight.
Carbon dioxide	Polyethylene, PTFE or glass	500	Fill container completely to exclude air. Transport under ice.		No holding time	Refrigerate (< 6°C)	Determine as soon as possible.
Carbon, total organic (TOC)	Polyethylene, PTFE or amber glass container with PTFE cap liner.	100	Transport under ice away from light.	Acidify (sulfuric, hydrochloric, or phosphoric acid) to < pH 2,	7 days recommended. 28 days allowed.	Refrigerate (< 6°C) in dark	Analyse as soon as possible. Keep away from light.
Cations: • calcium (Ca) • magnesium (Mg) • potassium (K) • sodium (Na)	Polyethylene or PTFE	500	Fill container completely to exclude air. Transport under ice	Acidify with nitric acid to pH < 2.	< 6 months; 7 days without acidifying sample.	Refrigerate (< 6°C)	Acidification allows determination of other metals in the sample.
Chemical oxygen demand (COD)	Glass, PTFE or polyethylene	100	Fill container completely to exclude air. Transport under ice away from light.	Preferable to analyse as soon as possible. Otherwise, acidify with sulfuric acid to pH < 2 . Freeze (only if polyethylene containers are used).	28 days 28 days	Refrigerate (< 6°C) in dark	Glass containers are preferable for samples with low COD (<5 mg/L). Keep away from light.
Chlorate	Polyethylene, PTFE or glass	500	Transport under ice		7 days	Refrigerate (< 6°C)	

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
Chlorine (residual)	Polyethylene, PTFE or glass	500	Keep sample out of direct sunlight.		Begin analysis within five minutes of sample collection. Maximum holding time is 15 min	Keep sample out of direct sunlight	Analysis must be carried out in the field.
Chlorine dioxide	Polyethylene, PTFE or glass	500	Keep sample away from light.		Begin analysis within five minutes of sample collection.	Keep sample away from light.	Analysis must be carried out in the field.
Chlorite	Polyethylene, PTFE or glass	500	Transport under ice, away from light		Five minutes. Should analyse immediately	Refrigerate (≤ 6°C) in dark	Analysis should be carried out in the field
Chloramine	Polyethylene or glass	500	Keep sample away from light.		Begin analysis within five minutes of sample collection.		Analysis should be carried out in the field.
Chlorophyll	Polyethylene, PTFE or amber glass	1000	Transport under ice away from light	Filter (0.45↔m glass fibre) and rapid freeze (e.g. snap freeze using liquid nitrogen in situ or upon receipt to laboratory) filter paper in the dark.	28 days	Freeze (-80 ºC)	Filters must not be touched with fingers and all sample-handling apparatus must be kept free of acids, as this causes degradation of chlorophylls to phaeophytins. Filter and process samples promptly at the time of collection or upon receipt at laboratory, ensuring minimum exposure to light. Only use polyethylene containers when snap freezing sample filters.
					24 hours without filtering	Refrigerate (≤ 6°C) in dark	
Colour	Polyethylene, PTFE or glass	500	Transport under ice, in the dark		48 hours	Refrigerate (≤ 6°C) in dark	
Cyanide	Polyethylene, PTFE or glass	500	Transport under ice away from light.	If no interfering compounds are present, then add sodium hydroxide solution to pH ≥ 12.	24 hours if sulfide present, otherwise 14 days	Refrigerate (≤ 6°C) in dark	Refer to Table II in *USEPA CFR40 Part 136.3* (2007) for details of treating samples to mitigate potential interfering entities present, e.g. sulfides or oxidising agents. Adjusting sample pH to > 12 should be carried out after completing this step.

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
Electrical Conductivity	Polyethylene or glass	500	Fill container completely to exclude air. Transport under ice		24 hours 28 days if refrigerated	Refrigerate (≤ 6°C)	Preferably on site (*in situ*) using a calibrated meter.
Fluoride	Polyethylene	500		None required.	28 days		
Gases (dissolved)	Glass with PTFE lined lids or septum caps	1000	Fill container to completely exclude air. Transport under ice For purge and trap analysis collect samples in duplicate or triplicate in 40 mL vials with PTFE faced septum	Acidify to pH < 2 with H_2SO_4, HCl or solid $NaHSO_4$	As per information for volatile organic hydrocarbons	Refrigerate (≤ 6°C) Store in area free of solvent fumes.	See information for volatile organic hydrocarbons
Hardness	Polyethylene, PTFE or glass	500	Fill bottle to exclude air. Transport under ice.	Acidify with nitric or to pH < 2	< 6 months; 7 days without acidification of sample	Refrigerate (≤ 6°C)	
Iodide	Polyethylene, PTFE or glass	500	Transport under ice		28 days	Refrigerate (≤ 6°C)	
Iodine	Glass, polyethylene, PTFE or	500	Transport under ice in the dark.		Immediate analysis preferred (15 min) but up to 24 hours allowed	Refrigerate (≤ 6°C) in dark	Immediate analysis recommended
Lignins and tannins	Glass with PTFE lined lid	250	Transport under ice		7 days	Refrigerate (≤ 6°C)	

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
Metals: • aluminium(Al) • antimony (Sb) • arsenic (As). • barium (Ba) • beryllium (Be) • cadmium (Cd) • chromium (Cr_{total}) • cobalt (Co) • copper (Cu) • ferrous iron(Fe^{2+}) • gold (Au) • iron (Fe_{total}) • lead (Pb) • lithium (Li) • manganese (Mn) • molybdenum (Mo) • nickel (Ni) • selenium (Se) • silver (Ag) • tin (Sn) • uranium (U) • vanadium(V) • zinc (Zn)	Polyethylene, PTFE or glass. PTFE preferred. For Ag, wrap sample bottle in foil, or use amber glass	500	Transport under ice For Fe^{2+}, fill container completely to exclude air.	Acidify with nitric acid to pH < 2. For dissolved metals, filter immediately, and then acidify. For As and Se, acidify with nitric or hydrochloric acid to pH < 2. For Fe^{2+}, acidify with hydrochloric acid to pH < 2.	< 6 months		Note: Silver photographic waste not suitable for acidification as this can cause precipitation of some silver complexes. Acid washed polyethylene; polycarbonate or fluoropolymer containers should be used for determinations at very low concentrations, such as encountered in uncontaminated streams and seawater. For Sb & As, hydrochloric acid should be used for acidification if the hydride generation technique is used for analysis.
Chromium (Cr^{6+}) hexavalent	Polyethylene or glass	500	Transport under ice		24 hours	Refrigerate (≤ 6 °C)	Acid washed polyethylene, polycarbonate or fluoropolymer containers should be used for determinations at very low concentrations, such as encountered in uncontaminated streams and seawater. Sample containers should be thoroughly rinsed after acid washing to ensure there is no residual nitric acid present.

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
Mercury (Hg_{total})	PTFE with PTFE or PTFE-lined caps. Can also use acid washed borosilicate glass if no other metals are being analysed. Polyethylene (PE) not recommended.	500	Transport under ice	5 mL/L 12 M HCl or bromine monochloride (BrCl) as detailed in USEPA method 1631, rev E. (2002) For dissolved Hg, a sample is filtered (0.45 μm) prior to preservation, and accompanied by a blank that has been filtered under the same conditions.	28 days		For contaminated waters more oxidant may be required. The analyst should be consulted for further instruction. Acid washed fluoropolymer or borosilicate containers with a fluoropolymer lined lid should be used for determinations at very low concentrations, such as encountered in uncontaminated streams and seawater.
Natural attenuation of hydrocarbons (including those chlorinated) in groundwater: - alkalinity - arsenic (As) - electrical conductivity - hydrogen gas ($H_{2(g)}$) - iron (ferrous, Fe^{2+}) - manganese (Mn^{2+}) - methane (CH_4) - nitrate (NO_3^-) - oxidation/reduction potential (ORP) - pH - sulphate (SO_4^{-2}) - temperature	Glass with PTFE lid	1000	Transport under ice. When sampling, minimise aeration with no air space remaining	See information for relevant analyte			See USEPA (1998) for further details
Nitrate (NO_3^-)	Polyethylene, PTFE or glass	500	Transport under ice	Acidify with HCl to pH <2	48 hours without acidification 7 days with acidification	Refrigerate (< 6°C)	

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
			Filter on site (0.45 µm cellulose acetate membrane filter) and freeze sample immediately upon collection.		28 days if frozen	Freeze (-20 ºC)	
Nitrite (NO_2^-)	Polyethylene, PTFE or glass	200	Transport under ice		48 hours	Refrigerate (≤ 6°C)	
Nitrogen (Kjeldahl, total)	Polyethylene, PTFE or glass	500	Transport under ice	Acidify with H_2SO_4 to pH< 2 and refrigerate or freeze	28 days	Refrigerate (≤ 6°C) after acidification, or only freeze (-20 ºC) sample immediately upon receipt	The sample may be acidified with sulfuric acid to pH <2 if required for other analyses.
Odour	Polyethylene, PTFE or glass	500	Transport under ice	Refrigerate (< 4°C)	6 hours	Refrigerate (≤ 6°C)-	Analyse asap. Storage not recommended.
Organic carbon (total)							See Carbon, total organic (TOC)
Oxygen, dissolved (DO)	Glass BOD bottle with top	300	Exclude air from bottle and seal.		Analyse immediately on site (*in situ)*		Excessive turbulence should be avoided to minimise oxygen entrainment. The meter must be calibrated on the day of use and checked after measurements. Winkler titration may be delayed after acidification to fix oxygen
				Fix oxygen with the azide-Winkler method to the acidification step	8 hours	Store in dark	See also APHA (2005) method 4500-O C.

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
pH	Polyethylene, PTFE or borosilicate glass	100	Fill bottle to exclude air. Transport under ice		Determine *in situ* if possible, or upon arrival to laboratory.	Analyse immediately	The meter must be calibrated on the day of use and preferably checked after measurements. Lab analysis useful for confirmation and also to determine any change during transit.
Phosphate (ortho or dissolved) (PO_4^{3-})	Polyethylene or glass	50 to 300	Filter on site (0.45 μm cellulose acetate membrane filter). Transport under ice		48 hours 28 days	 Freeze (< -20 ºC) after filtration to extend holding time	Should analyse as soon as possible
Phosphorus (total)	Polyethylene, PTFE or glass	300	Transport under ice	 Freeze Acidify with H_2SO_4 to pH < 2	24 hours without acidification or freezing 28 days 28 days	 Freeze (-20 ºC) immediately upon receipt Refrigerate (≤ 6°C)	 The use of the acid preservation method should not be used for the persulphate oxidation method of analysis.
Radioactivity (specific forms)						Refrigerate (≤ 6°C)	See also Table 4 in ISO 5667-3:2003(E), or USEPA 40CFR136.3 (2007) Ch. I (7-1-08 Edition)
Radioactivity, α and β activity (gross)	Polyethylene, PTFE or glass	1000	Fill container to exclude air. Transport under ice.	Acidify with nitric acid to pH < 2	28 days	Refrigerate (≤ 6°C) in dark.	Do not acidify if sample is evaporated before analysis
Silica (reactive) (SiO_2)	Polyethylene, PTFE or quartz	200	Transport under ice. Filter in the field (0.45 μm cellulose acetate membrane filter)		28 days	Refrigerate (≤ 6°C). Do not freeze.	Turbid river samples should be filtered in the field (0.45 μm cellulose acetate membrane filter).

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
Solids: • dissolved • Suspended • total	Polyethylene, PTFE or glass	500	Transport under ice. Fill container to exclude air.		7 days	Refrigerate (≤ 6°C)	Dissolved solids also known as 'filterable residues' and 'total dissolved solids' (TDS). Suspended solids (SS) also known as 'non-filterable residues' (NFRs) and SS.
Sulfide (dissolved) (S^{-2})	Polyethylene	50 by pipette	Transport under ice away from light	Add 10 mL copper-2,9 dimethyl-1,10-phenanthroline (DMP) reagent.	12 hours	Refrigerate (≤ 6 ºC) away from light	See also AS 3550.1 1988
Sulfide (total)	Polyethylene, PTFE or glass	500	Completely fill bottle without aeration. Transport under ice.	Fix samples immediately on site by adding 2 mL of 10% (m/v) zinc acetate solution per 500 mL of sample and NaOH to pH>9	Determine on site if not preserving 7 days	Refrigerate (≤ 6 ºC)	For chlorinated samples, add 80 mg ascorbic acid per 100 mL sample to prior to analysis, as per ISO 5667-3:2003(E)
Sulfite (SO_3^{-2})	Polyethylene, PTFE or glass	500	Fill container completely to exclude air.	Fix in the field by addition of 10 mL of 2.5% EDTA solution per 1 L.	Analyse immediately if not preserved. 2 days		
Surfactants: • anionic • cationic	Glass rinsed with methanol	500	Fill container to exclude air. Transport under ice.	Acidify with sulfuric acid to pH < 2 (check acidity first) Add 40% formaldehyde solution to give 1% (v/v) final concentration	48 hours 96 hours 48 hours	Refrigerate (≤ 6 ºC)	Glassware should not have been previously washed with detergent. Can be combined with non-ionic surfactant as per ISO 5667-3:2003(E)

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
• non-ionic				Add 40% formaldehyde solution to give 1% (v/v) final concentration	28 days		
Temperature	Polyethylene, PTFE or glass		Not applicable.	Not required	Determine *in situ*	Not applicable	Analyse immediately
Total organic carbon (TOC)							See entry for Carbon, total organic
Total dissolved solids (TDS)							See entry for Solids (dissolved)
Total solids							See entry for Solids (total)
Total suspended solids (TSS)							See entry for Solids (suspended)
Toxicity (by Microtox®)	Borosilicate glass with PTFE screw top cap	200	Fill container to exclude air. Transport under ice.		Preferable to analyse within first 2 hours of collection, but can hold up to 36 hours according to USEPA 40CFR136.3 (2007)	Refrigerate (≤ 6 ºC)	Record temperature of sample upon collection, and upon receipt to laboratory to ensure temperature fluctuation does not influence outcome of test.
Turbidity	Polyethylene, PTFE or glass	100	Transport under ice, in dark		Up to 48 hours	Refrigerate (≤ 6°C) in dark.	Recommend immediate on-site analysis if possible

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
Organics							
Volatiles organic compounds (VOCs) including hydrocarbons and • **Monocyclic aromatic hydrocarbons (MAHs)** • **Halogenated hydrocarbons**	Glass with PTFE lined lids or septum caps	500 or 40 ml vials	Fill container to completely exclude air. Transport under ice For purge and trap analysis collect samples in duplicate, or triplicate, in 40 ml vials with PTFE faced septum	Acidify to pH < 2 with H_2SO_4, HCl or solid $NaHSO_4$	Analyse as soon as possible within 7 days 14 days	Refrigerate (≤ 6°C) Store in an area free of solvent fumes.	Do not pre-rinse container with sample. If carbonaceous material, MTBE or other fuel oxygenate ethers present and a high temperature sample preparative method is to be used, do not acid preserve sample. If vinyl chloride styrene, or 2-chloroethyl vinyl ether are of interest, collect second set of samples without acid preservatives and analyse as soon as possible. If residual chlorine is present, add 80 mg sodium thiosulphate ($Na_2S_2O_3$) per 1000mL of sample before adding acid.
Trihalomethanes		100	Fill container to exclude air.		14 days		If residual chlorine is present, for each 40 mL of sample add 3mg of sodium thiosulphate.
Semi volatile organic compounds (SVOCs) including hydrocarbons such as TPH, TRH & TRPH* • Polychlorinated biphenyls (PCBs) • With residual chlorinePolycyclic aromatic hydrocarbons (PAHs)	Amber glass container with PTFE lined lid.	500	Transport under ice away from light	If residual chlorine is present, add 80 mg sodium thiosulphate ($Na_2S_2O_3$) per 1000mL sample	7 days 40 days after extraction.	Refrigerate (≤ 6°C)	Refer to NEPM Schedule B(3) for elaboration of hydrocarbon nomenclature Can add $Na_2S_2O_3$ to container prior to field use.

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
Herbicides		1000					
• acidic	Glass with PTFE cap liner		Do not completely fill container.	Acidify with hydrochloric acid to pH < 2	14 days		.
• non-acidic	Amber glass with PTFE cap liner				7 days		
• glyphosate	Polypropylene				14 days	Refrigerate (≤ 6°C) in the dark.	
Hydrazine	Glass	500	Transport in dark	Acidify with 100 mL concentrated hydrochloric acid for every litre of sample (i.e. to 1 mol/litre)	24 hours	Store in the dark.	
Pesticides							Extract the sample in the container as part of the sample extraction procedure
• carbamates	Amber glass	1000			28 days		.
• nitrogen-containing, organochlorine and organophosphate pesticides	Glass with PTFE cap liner	1000 to 3000	Do not completely fill container.		7 days		

Analytical parameter	Container*	Typical volume (mL)	Sampling and transport	Preservation	Maximum holding time	Storage	Comments
Phenolic compounds	Solvent washed glass (amber) with PTFE cap liner	1000	Do not completely fill sample container. Transport under ice.		24 hours	Refrigerate (≤ 6°C) in dark.	Do not pre-rinse container with sample. Oxidising agents such as chlorine may be neutralised by the addition of excess sodium arsenite or iron (II) sulfate prior to acidification. If residual chlorine is present, add 80 mg sodium thiosulfate ($Na_2S_2O_3$) per 1000mL of sample. Sulfur dioxide or hydrogen sulfide may be removed by briefly aerating the acidified sample.
				Acidify to pH< 2 with orthophosphoric acid, hydrochloric acid or sulfuric acid.	21 days	Refrigerate (≤ 6°C) in dark.	

**TPH= total petroleum hydrocarbons; TRH=total recoverable hydrocarbons; TRPH=total recoverable petroleum hydrocarbons*

APPENDIX B: SOILS AND SEDIMENTS – CONTAINERS, PRESERVATION AND HOLDING TIMES

Information here has been sourced from various references including NEPM Schedule B(3) *Guideline on Laboratory Analysis of Potentially Contaminated Soils*, USEPA SW846 on-line, www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm#table, and some Australian standards. Unless otherwise specified, samples should be transported under ice and refrigerated (≤ 6°C) when stored. The analyst should always be consulted for advice on actual sample sizes required (250-500 g per sample is a typical amount).

Table 2: Soils & sediments: container types, preservation and maximum sample holding times

Analytical parameter	Container	Sampling & Transport	Preservation	Maximum holding time	Storage	Comments
Acid generating capacity/ Acid sulfate soils	Sealable plastic bag	Exclude as much air as possible from the bag and seal. Transport under ice.	Drying of the sample at 80– 85°C (fan forced air extracting oven). After drying, store sealed in cool dry place.	Submit to laboratory and commence drying step within 24 hours. 6 weeks after drying	Freeze sample until ready to dry	If monosulfides are suspected to be present, the sample must be freeze-dried. Refer to AS4969 series (2008/2009)
Anions: • bromide (Br^-) • chloride (Cl^-) • fluoride (F^-) • sulfate (SO_4^{-2})	PTFE, plastic	Transport sealed, under ice		28 days	Refrigerate (≤ 6 ºC)	Field moist or air dry
Asbestos	Glass or LDPE			indefinite		Refer to AS 4482.1-2005 & AS4964-2004 Use appropriate personal protective equipment (ppe), especially breathing apparatus and skin protection Polypropylene containers are unsuitable.
Bromide (water soluble)	Polyethylene or glass	Transport under ice		28 days	Refrigerate (≤ 6 ºC)	Air dry
Carbon, organic	Glass with PTFE lined cap	Transport under ice away from light	Keep in airtight container	28 days	Refrigerate (≤ 6 ºC)	Air dry
Cation exchange capacity Exchangeable cations	Acid washed polyethylene	Transport under ice		28 days	Refrigerate (≤ 6 ºC)	Air dry
Chloride (water soluble)	Polyethylene or glass	Transport under ice if field moist		28 days	Refrigerate (≤ 6 ºC) if field moist	Field moist or air dry

Analytical parameter	Container	Sampling & Transport	Preservation	Maximum holding time	Storage	Comments
Cyanide	Glass, polyethylene or PTFE	Transport under ice		14 days	Refrigerate (≤ 6 ºC) away from light	Field moist
Electrical conductivity	Polyethylene or glass	Transport under ice		7 days	Refrigerate (≤ 6 ºC)	Air dry
Fluoride	Polyethylene	Transport under ice		28 days	Refrigerate (< 6 ºC)	Field moist or air dry. See also ISO 5667.3:2003
Metals (leachable/soluble & total) Mercury (Hg) & hexavalent chromium (Cr^{6+})	PTFE, plastic, glass Acid washed polyethylene	Transport under ice		6 months 28 days For Cr(VI), holding time is 28 days till extraction, and after extraction a further 7 days for analysis	Refrigerate (≤ 6 ºC)	Field moist or air dry Field moist.
Moisture content	PTFE, plastic, glass	Transport under ice		14 days	Refrigerated (≤ 6 ºC) in airtight container to avoid moisture loss	Field moist Carry out on the same day as sample extraction for other analytical parameters
pH	Polyethylene, PTFE or glass	Transport under ice	None.	24 hours recommended. 7 days allowed	Refrigerated (≤ 6 ºC)	Should analyse immediately (in the field) Air dry, or field moist. Need to specify which is used. In latter case, should also report moisture content
Sulfate	Polyethylene, PTFE or glass	Transport under ice		28 days	Refrigerated (≤ 6 ºC)	Field moist or air dry
Sulfide	Polyethylene, PTFE or glass	Transport under ice in airtight container	Add sufficient 1 molar zinc acetate to fully cover soil surface and make headspace	7 days	Refrigerated (≤ 6 ºC) headspace free	Field moist
Sulfur – total	Polyethylene, PTFE or glass			7 days	Refrigerated (≤ 6 ºC)	Field moist or air dry
Organics						

Analytical parameter	Container	Sampling & Transport	Preservation	Maximum holding time	Storage	Comments
Volatile organic compounds (VOCs) Vinyl chloride, styrene or 2-chloroethyl vinyl ether	Glass with PTFE lined lid/septum	Transport under ice, in dark	Refer to individual methods	14 days 7 days	Refrigerate (≤ 6 ºC), in dark	Pre-rinsing of containers with solvent not encouraged due to unnecessary waste and potential of introducing contamination into sample. Should not collect from surface, unless there has been a recent spill.
Semi-volatile organi compounds (SVOCs) including hydrocarbons such as TPH, TRH & TRPH* • PAHs • Pesticides & herbicides • Phenolics • Polychlorinated biphenyls (PCBs) • Other	Glass with PTFE lined lid	Transport under ice, in dark		14 days, except for PCBs (28 days)	Refrigerate (≤ 6 ºC), in dark	Pre-rinsing of containers with solvent not encouraged due to unnecessary waste as well as potential of introducing contamination into sample.

***TPH= total petroleum hydrocarbons; TRH=total recoverable hydrocarbons; TRPH=total recoverable petroleum hydrocarbons**

APPENDIX C: CONCENTRATED LIQUID WASTES, SLUDGES AND SOLID WASTES, OTHER THAN SOILS AND SEDIMENTS – CONTAINERS, PRESERVATION AND HOLDING TIMES

For samples collected to determine specific components, the following sampling and preservation procedures should be followed. When samples are collected for general identification, refer to Table 3a, at the end of this Appendix for sampling and preservation protocols.

Information here has been sourced from resources used to compile Tables 1 and 2. Additional details on sampling of soils and sediments can be obtained from USEPA Publication: *Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods,* USEPA Publication No SW-846 on-line, www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm#table, or a comparable publication.

Aqueous trade waste sampling should be handled in accordance with requirements set out in Table 1 (Waters, wastewaters & groundwaters).

Table 3: Concentrated liquid wastes, sludges and solid wastes, other than soils and sediments: container types, preservation and maximum sample holding times

Analytical parameter	Container	Sampling & transport	Preservation	Maximum holding time	Storage	Comments
Asbestos	Glass, PTFE LDPE, or zip lock bags	Transport sealed.	Depends on other analytes present	Depends on other analytes present	Depends on other analytes present	Refer to AS 4482.1-2005, AS4964-2004, & WA Dept of Health, 2008. Use appropriate personal protective equipment (ppe), especially breathing apparatus and skin protection
Carbon, total organic (TOC)	Glass with PTFE lined lid	Transport sealed under ice away from light	For liquid samples, acidify to pH < 2 using sulfuric acid or hydrochloric acid.	28 days	Refrigerate (≤ 6 ºC) in dark	
Chloride (Cl^-)	Polyethylene, PTFE or glass		None.	28 days		
Cyanide (CN^-)	Polyethylene or PTFE	Transport under ice in the dark	For liquid samples: • adjust to pH>12 with 50% sodium hydroxide • if oxidising agents (e.g. chlorine) are present add excess ascorbic acid (0.6 g per litre) until starch iodide paper fails to turn blue on contact with the sample)	14 days	Refrigerate (≤ 6 ºC) in the dark	

Analytical parameter	Container	Sampling & transport	Preservation	Maximum holding time	Storage	Comments
Metals	Polyethylene, PTFE or glass	Transport under ice	None required for solids For liquid samples, • total determinations: acidify to pH<2 with nitric acid. • dissolved analytes: filter (0.45 μm), then acidify, suspended solids: filter and retain residue for analysis	6 months	Refrigerate (≤ 6 ºC)	
Mercury (Hg)			For liquid samples, • Total: acidify to pH<2 with nitric acid • Soluble: filter, then acidify to pH < 2	28 days	Refrigerate (≤ 6 ºC)	
Hexavalent chromium (Cr^{6+})			.	Solid samples: 30 days until extraction 7 days after extraction. Liquid samples: 24 hours	Refrigerate (≤ 6 ºC)	
Nitrate (NO_3^-)	Polyethylene, PTFE or glass	Transport under ice	• None • Acidify to pH < 7 with HCl • Acidify to pH < 2 with H_2SO_4	• 24 hours • 7 days • 28 days	Refrigerate (≤ 6 ºC)	
pH	Polyethylene, PTFE or glass	Transport under ice	•	24 hours recommended. 7 days allowed	Refrigerate (≤ 6 ºC)	For liquid samples, should analyse immediately if possible.
Sulfate (SO_4^{-2})	Polyethylene, PTFE or glass	Transport under ice		28 days	Refrigerate (≤ 6 ºC)	

Analytical parameter	Container	Sampling & transport	Preservation	Maximum holding time	Storage	Comments
Sulfide (S^{-2})	Polyethylene, PTFE or glass	Transport under ice. For liquid samples, fill bottle completely and stopper with minimum aeration	For solid samples, fill the surface of the solid with 1 M zinc acetate until moistened and store headspace free. For liquid samples, add 4 drops 1 M zinc acetate per 100 mL, then adjust pH to >9 using 6 M sodium hydroxide. If sample is chlorinated, add 80 mg ascorbic acid per 100 mL sample prior to analysis.	7 days	Refrigerate (≤ 6 ºC)	
Organics						
Volatile organic compounds (VOCs)	Polyethylene, PTFE or glass	Transport under ice in sealed container		Solids: 7 days Liquids: 7 days (14 days if acidified)	Refrigerate (≤ 6 ºC)	
Semivolatile organic compounds (SVOCs) including hydrocarbons such as TPH, TRH & TRPH* • Dioxins and furans, • Hydrocarbons (halogenated) • Polychlorinated biphenyls (PCBs)	Glass with PTFE-lined cap	Transport under ice		Solid samples: 14 days until extraction and 40 days after extraction	Refrigerate (≤ 6 ºC)	
• Phthalate esters • Polycyclic aromatic hydrocarbons (PAHs)	Amber glass with PTFE-lined cap	Transport under ice away from light		Liquid samples: 7 days until extraction and 40 days after extraction	Refrigerate (≤ 6 ºC) in the dark	For liquid samples, if residual chlorine is present, add 80 mg sodium thiosulfate ($Na_2S_2O_3$) per 1000mL of sample. Can add $Na_2S_2O_3$ in container prior to sampling in the field
			Freeze	28 days if freezing	Freeze (< - 20 ºC)	

Analytical parameter	Container	Sampling & transport	Preservation	Maximum holding time	Storage	Comments
Pesticides and herbicides (organochlorine & organophosphate)	Glass with PTFE-lined cap	Transport under ice	For liquid samples, if residual chlorine is present, add sodium thiosulfate ($Na_2S_2O_3$, 80 mg per 1000mL sample.)	Solids: 14 days until extraction; 40 days after extraction Liquids: 7 days until extraction; 40 days after extraction	Refrigerate (≤ 6 ºC)	Do not rinse sample bottles with sample. Can add $Na_2S_2O_3$ in container prior to sampling in the field
Phenols	Amber glass with PTFE-lined cap	Transport under ice in the dark.	Acidify to pH < 2 with H_2SO_4 If residual chlorine is present, add sodium thiosulfate ($Na_2S_2O_3$, 80 mg per 1000mL of sample).	Solids: 14 days until extraction; 40 days after extraction. Liquids: 7 days until extraction; 40 days after extraction Chlorophenols: 2 days to extraction	Refrigerate (≤ 6 ºC) in the dark	Do not rinse sample bottles with sample.

**TPH= total petroleum hydrocarbons; TRH=total recoverable hydrocarbons; TRPH=total recoverable petroleum hydrocarbons*

Table 3a: Applicable conditions for samples being collected for general identification

Sample	Container	Preservation/storage	Maximum holding time
Concentrated wastes & solids	Glass with PTFE or PTFE lined lid/cap fluoropolymer cap	Refrigerate (≤6 ºC)	14 days
Aqueous samples		Adjust to pH <2 with hydrochloric acid. Refrigerate (≤6 ºC).	7 days
Sludges		Refrigerate (≤6 ºC)	7 days

APPENDIX D: RECOMMENDED METHODS FOR THE ANALYSIS OF TOTAL CONTAMINANT LEVELS IN SOLID WASTE

Methods described in Table 4 are recommended for the analysis of solid wastes and have been mostly sourced from the NEPM Schedule B(3) guidelines. Parameters not listed here should be analysed using recognised standard methods (latest version).

Any extraction/analytical procedure that can be shown to be proficiently equivalent or better than that stated here may be used for a given analyte/matrix as long as it is NATA-accredited.

Table 4: Recommended methods for analysing contaminants in solid waste

Chemical contaminants	Recommended extraction &/or analytical method(s)	Comments
Metals, including: • antimony (Sb) • arsenic (As) • barium (Ba) • beryllium (Be) • boron (B) • cadmium (Cd) • copper (Cu) • lead (Pb) • molybdenum (Mo) • nickel (Ni) • selenium (Se) • silver (Ag) • zinc (Zn)	• NEPM Methods 201, 202 or 203 • USEPA Methods 3051A, 3050B, 6010C, 6020A, 200.7 & 200.8 • APHA methods 3110 to 3125	
Mercury (Hg)	• NEPM Method 203 • USEPA Methods 3051A, 7471B & 6020A	Mercury in solid and semi-waste by cold vapour method
Hexavalent chromium (Cr^{6+})	• USEPA Method 3060A	
Fluoride (F^-)	• NEPM Method 404 • ASTM method 3269-96 (2001) (sodium fusion followed by ion chromatography) • APHA method 4500-F^-	

Chemical contaminants	Recommended extraction &/or analytical method(s)	Comments
Cyanide total & cyanide amenable	• NEPM Method 403 • USEPA Methods 9012B (colorimetric), 9010C (distillation) & 9014 (spectrophotometric & titrimetric methods) • APHA method 4500-CN^-	
Tributyltin (TBT)	USEPA Method 8323* APHA method 6710	Suitable extraction procedure followed by GC-ICP-MS should be acceptable
Organochlorine pesticides, including: • Aldrin • Dieldrin • DDT • DDD • DDE • Chlordane • Heptachlor	• NEPM Method 504 • USEPA Methods 8081B (GC method), 8270D & 3500C#	End over end extraction in solvent is a viable alternative if it can be shown to perform similarly to soxhlet /sonication method
Chlorinated herbicides:	• NEPM Method 508 • USEPA Methods 8151A & 3580A	
Benzo(a)pyrene PAHs (total)	NEPM Method 502 USEPA Methods 8100, 3500C# & 8270D	End over end extraction in solvent is a viable alternative if can be shown to perform similarly to soxhlet/sonication method

Chemical contaminants	Recommended extraction &/or analytical method(s)	Comments
Volatile organic compounds including: • benzene • carbon tetrachloride • chlorobenzene • chloroform • dichloromethane (methylene chloride) • ethylbenzene • methyl ethyl ketone (2-butanone) • styrene • 1,1,1,2-tetrachloroethane • 1,1,2,2-tetrachloroethane • Tetrachloroethene • Toluene • 1,1,1-Trichloroethane • 1,1,2-Trichloroethane • Trichloroethene • Vinyl chloride • Xylenes (total)	• NEPM Method 501 • USEPA Methods 8021B , 3500C#, 5021 & 8260C	
Selected chlorinated hydrocarbons including: • 1,2-dichlorobenzene • 1,4-dichlorobenzene • 1,2-dichloroethane • 1,1-dichloroethene • 1,2-dichloroethene • Hexachlorobutadiene • Trichlorobenzene (total)	• NEPM Method 501 or 503 • USEPA Methods 8021B, 3500C# , 8260C & 8270D	
Phenols, including: • 2 Chlorophenol • Cresol (total) • 2,4-Dichlorophenol • Phenols (total, non-halogenated) • 2,4,5-Trichlorophenol • 2,4,6-Trichlorophenol	• NEPM Method 507 • USEPA Methods 8041A, 3500C# & 8270D	End over end extraction in solvent is a viable alternative if can be shown to perform similarly to soxhlet/sonication method

Chemical contaminants	Recommended extraction &/or analytical method(s)	Comments
• 2,4-Dinitrotoluene • Nitrobenzene	USEPA Methods 8091, 3500C# , 8260C & 8270D	End over end extraction in solvent is a viable alternative if can be shown to perform similarly to soxhlet/sonication method
Polychlorinated biphenyls (PCBs)	• NEPM Method 504 • USEPA Methods 8082A & 8270D • EPA Victoria Method 6013	End over end extraction in solvent is a viable alternative if can be shown to perform/outperform soxlet/sonication method
Phthalate esters: di (2 ethylhexyl) phthalate	• NEPM Method 509 • USEPA Methods 8061A& 8270D	End over end extraction in solvent is a viable alternative if can be shown to perform/outperform soxlet/sonication method
Organic hydrocarbons **including TPH, TRH & TRPH*#**: • C6-C10 • C10-C40	• NEPM Method 506 • USEPA Methods 8015C , 8260C & 8440 • USEPA Extraction Methods 3560 , 3660B & 3545A (SFE for semivolatiles) • USEPA Methods 3540C (soxhlet extraction) & 3550C (ultrasonic extraction) (solvent extraction for semi-volatiles)	GC-MS is main option for C_6-C_{10} while GC-FID is the main option for C_{10}-C_{40} fraction. End over end extraction in solvent is a viable alternative if can be shown to perform similarly to soxhlet/sonication method Need to state if results are for cleaned up samples. Appropriate clean up of sample will be necessary to remove interfering compounds such as fatty acids.

* Determination of organotins by micro-liquid chromatography-electrospray ion trap mass spectrometry
# General procedure for organic extraction and sample preparation
SFE= supercritical fluid extraction
*#TPH= total petroleum hydrocarbons; TRH=total recoverable hydrocarbons; TRPH=total recoverable petroleum hydrocarbons

APPENDIX E: QUALITY ASSURANCE SYSTEMS

Quality assurance procedures are mandatory for NATA accreditation. 'Quality assurance' (QA) and 'quality control' (QC), integral to laboratory analysis activities, are defined below.

Quality assurance

Quality assurance (QA) is all of the actions, procedures, checks and decisions undertaken to ensure the accuracy and reliability of analytical results.

The following publications may be referred to when developing a QA program:

1. NEPM, *Guideline on Laboratory Analysis of Potentially Contaminated Soils* (NEPC most recent).
2. American Public Health Association, *Standard Methods for the Examination of Water and Wastewater*, Method 1020 (APHA, 2005).
3. US Environment Protection Agency, *Test Methods for Evaluating Solid Waste*, SW-846 on-line, Chapter 2 (USEPA), www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm#table.
4. National Association of Testing Authorities (NATA) Technical Note No. 23 (2008).

Quality control

Quality control (QC) is the part of QA which monitors and measures the effectiveness of its procedures including measurement of reagent quality, cleanliness of apparatus, accuracy and precision of methods and instrumentation and reliability implemented daily in the laboratory.

QC procedures

Analysts should implement the following QC steps with each analytical batch, or with each twenty samples, whichever is the smaller.

Analysis blank – determination of the contribution to the analytical signal by reagents, glassware etc. The contribution measured should be subtracted from the gross analytical signal for each analysis, before calculation of each sample's measured concentration.

Replicate analysis – repeated analysis of at least one sample from the batch.

Laboratory control samples – comprise a control matrix (for example deionised or tap water) or a replicate portion of a sample under analysis, which include added components representative of the analytical parameter class at concentrations easily quantified and within the concentration range expected for real samples.

Surrogate spikes – known additions to each sample, blank and matrix spike or reference sample of compounds that are similar to the analytical parameters of interest in terms of:

- extraction
- recovery through clean-up procedures,
- response to chromatographic or other determination

but which:

- are not expected to be found in real samples
- will not interfere with quantification of any analytical parameter of interest
- may be separately and independently quantified.

Surrogate spikes are added to the analysis portion before extraction. The purpose of surrogates is to provide a means of checking that no gross errors leading to significant analytical parameter losses have occurred at any stage of the procedure.

In the case of organic analyses, the surrogate spike compounds may be deuterated, alkylated or halogenated analogues, or structural isomers of compounds under analysis.

Internal standards – are added to samples after all extraction, clean-up and concentration steps have been completed. This addition is a constant amount of one or more compounds with similar qualities to surrogate compounds.

The purpose of internal standards is to check the consistency of the analytical step (for example, injection volumes, instrument sensitivity and retention times for chromatographic systems) and provide a reference against which results may be adjusted in case of variation.

Use of internal standards is required for chromatographic analysis of organics.

Reference materials – are homogeneous materials that have been rigorously characterised by several different procedures. They may be prepared in-house or obtained from commercial suppliers. Reference materials should be certified and traceable to a recognised authority. A reference material of comparable matrix should be analysed with each batch of samples.

QC records

Records of results of QC procedures should be maintained to establish method reliability, confidence intervals for analysis results, and trends in precision and accuracy.

NATA accreditation includes regular reviews of analytical methods and QA systems.