



Assessment of Water Quality: Sampling & Sample Preparation

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INTRODUCTION

- Developing sampling plan
- Sample collection and field testing procedures
- Sample Identification, transport and storage

Developing sampling plan

Key considerations

- 1. Monitoring objective

To clearly identify the objective of sampling – monitoring, routine or research purpose

- 2. Variability consideration

Time and **space**, determine the number of **sites**, number of **replicates** and the **frequency** of sample collection. High variability in the environment or the industrial discharge combined with poor sampling design or too few samples can result in data that is too variable to reveal an impact disturbance or trend.

Variation in water quality can occur at different frequencies including :

- **diurnal variation**—changes in dissolved oxygen and pH throughout the day due to respiration and photosynthesis, changes in water temperature
- **depth variation**—stratification effects can occur in lagoons and lakes as well as in rivers, creeks, marine water and estuaries
- **seasonal and event variation**—variations in flow and salinity due to rainfall and temperature
- **tidal variations**—variation in flow direction and volume due to tide (in rivers and estuaries as well as marine environment)
- **spatial variation**—occurs in the receiving environment due to a range of factors from natural biological variability, wave action and turbulence through to flow and concentration modifications around structures such as jetties and weirs.

Knowledge of the variations likely to affect sampling results are important in selecting the frequency and pattern of sampling as well as sampling points.

- 3. Precision required

The amount and frequency of sampling is sufficient to provide confidence in the interpretation of results. May be falsely detecting environmental harm when it has not occurred and not detecting environmental harm when it has occurred.

- 4. Logistical and OHS&W issues

- 5. Cost

Elements of a sampling plan

1. Duration of sampling

Example: For regulatory monitoring, sampling will generally be ongoing to show continued compliance with criteria, or to monitor the ongoing influence of discharges on the receiving environment.

2. Sampling locations

The **design of a sampling plan** should ensure samples are collected at sites and times that provide a **representative** sample, thus providing an accurate description of the overall quality of particular water bodies.

Sampling sites should be located in areas that are **safe to access**, accessible under **all conditions** of flow and discharge, be **well mixed** to ensure a homogenous sample is collected and be **easily identifiable** for later sampling. **Permanent sampling locations** should be established in any sampling environment to ensure that representative samples can be **compared over time**.

Example:

- In the case of examining the **effect of a point source discharge**, one should include the following sample locations:
 - end-of-pipe samples to characterise the discharge
 - in the receiving water upstream of the point of entry
 - in the waterbody at the point of entry
 - multiple samples at progressive distances downstream from the point of entry.
- In deeper or poorly mixed waters, a ‘surface sample’ may not accurately describe the characteristics of the entire water column. These can include integrated vertical column samples at given depths.

Elements of a sampling plan

3. Sampling frequency

There are no strict rules regarding how frequently sampling should occur, but the sampling frequency will be dictated by the **variability of the discharge** and the **objectives** of the plan.

During the planning stages, consider the aims of the study and choose a sampling frequency that has the best chance of providing the information required to meet the objectives of the plan.

If samples are **not taken frequently** enough, the characteristics of the pollutants might **not be adequately** described resulting in a **poor understanding** of the system and potentially **inaccurate reporting** of compliance or non-compliance. Alternatively, **overly frequent** sampling may be a **waste of time and resources**.



4. Analytes

Key considerations when choosing analytes include the form of analyte (eg total metals, dissolved metals, chemical speciation) and the confounding factors (eg faecal coliforms are often used to indicate contamination from human faeces, however in lagoons faecal coliforms may be the result of the presence of birds).

Planning of sampling event

Careful planning and preparation of a sampling event is important and will save time and reduce the number of difficulties

1. Logistics

The basic steps for planning a sampling event are as follows:

1. **Review** the monitoring plan, including monitoring locations, number of samples required, sampling methods, and Occupational Health, Safety and Welfare (OHS&W) issues.
2. **Inform the client** or property owner of your intended schedule
3. **Co-ordinate with the analytical laboratory (communication)**. Obtain appropriate sample containers. Discuss any problems you foresee, for example, with procedures, containers or limitations of reporting.
4. **Schedule the monitoring event**, including planning how and when you will transport the samples back to the laboratory. The aim is to have all samples preserved and delivered to the laboratory as quickly as possible and within recommended holding times. This is especially relevant for samples with holding times of 24 hours or less.
5. **Organise and review** site maps and locations to determine logistics of sampling including sampling order. Sampling order should be designed to avoid cross-contamination.
6. **Check** that you have all the equipment required for the sampling event. **Test** that the equipment is operational and calibrated.
7. Fill out as much paperwork as practical before sampling such as **preparation of labels**.

2. Equipments

Major items of equipment:

- Paper work and record keeping: The sampling plan, or concise sampling schedule and map should provide information such as location of monitoring points, the number and type of samples that need to be collected and container types. Records of observations and actions can be critical for future reference.
- Navigational aids: GPS
- Field testing meters
- Sampling containers
- Other sampling equipment: sampling rods, bucket and rope depth equipment or filtration equipment
- Esky or refrigerator:
- Camera

3. Occupational health, safety and welfare

A safety plan should be developed to address risks and may include such things as:

- hazard identification, risk assessment and hazard control measures. Typical hazards in sampling include:
 - vehicle breakdown or accident, bogging in wet conditions
 - exposure to hazardous substances eg decontamination chemicals, analytes, toxic products formed from sample preparation or stabilisation (eg acidification) and toxic gases such as hydrogen sulphide
 - temperature hazards, typically sunburn and heatstroke
 - working in, over or adjacent to water
 - poisonous animals (spiders, snakes) and plants
- actions to be undertaken to remove, reduce or control risk
- emergency procedures and information such as location of nearest medical facility.

The right **safety equipment** will make the task safer.

This equipment can be preventative or provide assistance in the case of an incident.

an example list of the type of **personal protective equipment (PPE)** that may be required for sampling in the field.

Safety equipment

First-aid kit

Sunscreen/sunglasses

Drinking water

Mobile phone/communication equipment

PPE – wide brimmed hat
wet weather gear
waders/rubber boots
disposable overalls

Antiseptic hand wash

Lifejackets/EPIRB



Sample collection and field testing procedures

I. Field observations

Observations of **field conditions** or any **abnormalities** that could assist in the interpretation of monitoring data are to be recorded. This can provide useful information about the water being sampled, which can help **diagnose the source and potential impact** of pollutants found by chemical analysis. Examples of such field conditions are as follows:

- recent rain can wash potential pollutants from surrounding land into waterways
- winds may drive some constituents toward one side of the waterbody or create mixing which might help dissipate them more quickly
- shading from cloud and vegetation can influence the level of dissolved oxygen.

2. Field measurements

Analytes that **quickly degrade** which can be significantly changed during transport and storage after they are sampled **must be tested in the field**.

Recommend for the following analytes :

- dissolved oxygen (DO)
- temperature
- pH
- conductivity
- redox (reduction/oxidation potential)
- turbidity
- chlorine.

Reliably measured using **multi-parameter meters**

Need to **calibrate** before use, recorded on log book including dates, temperatures and calibration readings.

This will provide a record of the performance of each meter and provide evidence that quality procedures are being employed.



Examples of the various types of field meters available. From left to right: a dissolved oxygen meter, a multi-parameter meter and a redox meter.

3. Collection of samples for analysis

3.1 Sampling equipment

The sampling equipment is inert, that is, it does not cause contamination or interference with the sample.

For example:

- Organics have a tendency to adsorb to plastic (including polyethylene, polypropylene and polycarbonate). Therefore, stainless steel equipment such as buckets and sampling rods should be used. Glass sample containers are preferred.
- When sampling for metal analytes the use of metal equipment, and some glasses such as soda glass should be avoided. Rubber can also cause contamination when sampling for trace concentrations. Plastic equipment should be used when possible when analysing for metals.
- When sampling for analytes that are the major constituents of glass (eg sodium, potassium, boron and silicon), glass equipment and containers should be avoided.

Refer note of APPENDIX 2

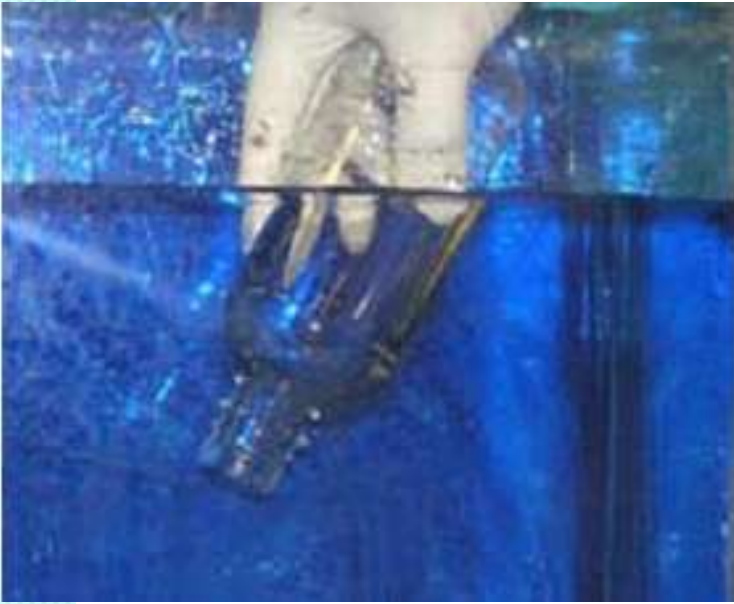
3.2 Surface samples—grab sampling

Sub-surface water sampling - when the waterbody is **shallow and well mixed**, taken from approximately 20 to 30 cm depth, ensure no floating films or organic material are collected unless they are of specific interest. Do not stirring up and collect of bottom sediments ; compromise sample integrity.

When practical, collect the sample **directly into the sample container**. Where this is not practical (such as when a sample cannot be collected without loss of preservative) an **intermediate container with sampling rod** may be used.

When sampling by hand, inserting the container into the water vertically with the neck facing down. Once at the required depth, the container can then be inverted, allowing the sample to flow in. The mouth of the container should be faced into the current while keeping the hands, sampler and any other equipment (eg boat) downstream to minimise the chance of contamination. Refer to

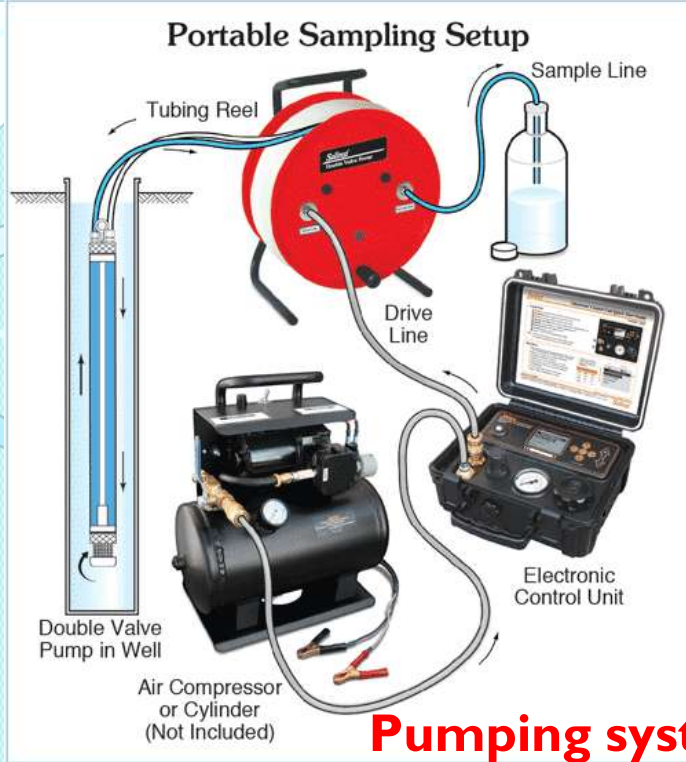
Appendix 2 – filling technique



3.3 Sampling at depths

When samples are required from particular depths, such as for depth profiling, specific equipment is required. The equipment falls into 3 general categories as follows:

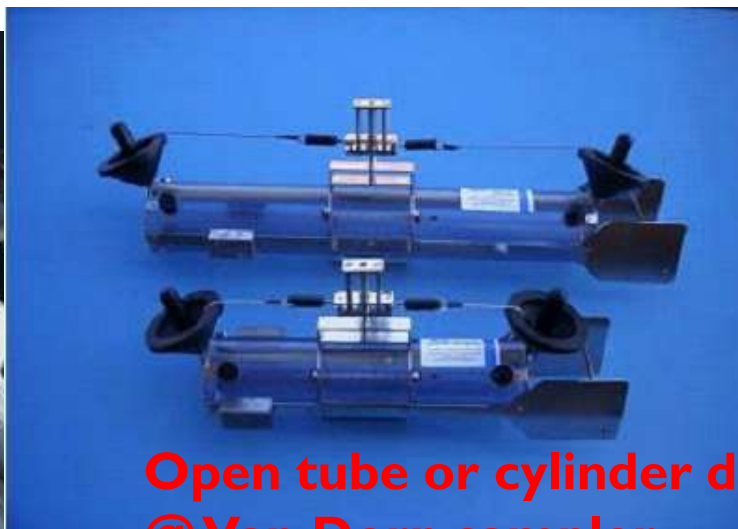
- Pumping system
- Sealed immersion devices
- Open tube or cylinder devices



Pumping system



Sealed immersion device



**Open tube or cylinder devices
@ Van Dorn sampler**

3.4 Field filtration

Field filtration can be undertaken for a number of purposes:

- to maximise a sample's integrity during transport from the sampling site to the
- to separate the total and the soluble portions of analytes (eg dissolved metals)—the soluble portion of an analyte is generally more bio-available, and therefore can have greater impact upon the ecosystem
- to extract filtered material for biological analytes such as chlorophyll and algae
- to separate biomass when undertaking wastewater analysis.

There are various methods and equipment available for field filtration from **simple gravity** or **syringe pressure systems** to more complex **pump operated pressure or vacuum systems**. The best method to use will depend upon the analysis to be performed or analyte to be determined.



Sample Identification, transport and storage

- **Labelling and identification**

Sample containers should be marked in such a way that they can be clearly identified and distinguished from other samples .

Labelling on samples should contain as much information as practical. Labels may contain:

- date of sampling
- time of sampling
- location and name of sampling site (include GPS coordinates if available)
- job or project number
- name of sampler
- container pre-treatment and preservations added
- other observations that may affect the method or results of the analysis.



Example of Sampling Water Labeling

PATHCENTRE
WATER EXAMINATION CONTAINER
(Sterile, with Sodium Thiosulphate)

Sender No. _____

Site Code: _____

Point of Collection: _____

Source: _____

Collection Date: _____

Time: _____

Transport Temperature Bacteriology: 4C
Protozoology: Ambient

Authority/Shire/Location _____

PLEASE FILL IN ALL DETAILS 900005

Prior to taking a sample the following information should be provided on all sample bottles:

Sender reference number

Site code

Point of Collection

(Aquatic Facility Name and pool (ie toddler's pool))

Source (ie Pool outlet)

Date and time of collection

Transport temperature (4C or ambient)

Authority or Company Name



- **Chain of Custody**

Chain of Custody procedures and documentation **demonstrate sample control.**

This gives **confidence** that the sample integrity has not been compromised and imperative if the samples are to be used in legal proceedings or if there is any suspicion that the samples might be tampered with at any stage of the process.

The Chain of Custody documentation is a record used **to trace possession and handling** of a sample from the time of collection through analysis, reporting and disposal.

Example: Chain of Custody Form

Customer	SA EPA	Laboratory	ABC Labs
Project Name	Clean lake	Contact Name	Sam Linard
Reference Number	05112/36	Address	524 Magill Road, Tranmere
Contact	P Bond	Phone	8234 5678
Phone	8123 4567		

Sample I.D.	Sample	Matrix (Water, soil)	Sampling		Container			Analysis required						Notes ←Max LOR	
			Date	Time	Type	Size (mL)	Preserv.	0.05 Ammonia	5 BOD 5	0.02 Phos					
W1-1	W1	Water	1/11/05	2.00pm	G	1000	4 °C	x	x						Ammonia—total as N
W1-2	W1	Water	1/11/05	2.00pm	G	250	4 °C			x					Phosphorus—total as P
W1-3	W1	Filter	1/11/05	2:30pm	Filter paper										2 Litres filtrate

Sample relinquished by:				Sample received by:					Agreed condition (temp, intact, etc)
Name/organisation	Initials	Date	Time	Name/organisation	Signature	Date	Time	Sample Condition	
Sampler				Courier					
Courier				Laboratory					

• Transport and storage

Ensure that samples are **not significantly altered** in condition and are in a state **fit for analysis** at the laboratory. **Contamination** of samples can easily occur during transport due to container cross-contamination, packaging material or chilling products.

During sample **storage**, degradation can occur due to **lack of appropriate preservation**, inappropriate **storage conditions**, **excessive storage times** and sample **cross-contamination**.

The key aspects of effective transport and storage are to:

- ensure samples are appropriately packed to **avoid breakage** and cross-contamination
- reduce sample degradation through **appropriate preservation**
- ensure time between sampling and analysing does **not exceed holding time**
- sample containers should be **sealed**, carefully packed with an appropriate packing material, chilled or frozen (as required) and transported in an appropriate cooler (esky) or fridge.

• Preservation techniques

To minimise changes to the sample following sampling.

Refer Appendix 2 - Required preservation techniques for specific analytes.

Some common preservatives are described below.

➤ **Refrigeration:** Keeping samples between **1°C and 4°C** will preserve the majority of **physical, chemical and biological** characteristics in the **short term (< 24 hours)**. Ice can be used to rapidly cool samples to 4°C before transport.

➤ **Freezing:** In general, freezing at **<-20°C** will prolong the storage period. Filtering samples in the field before freezing may be required. This is usually done for soluble nutrients, particularly when same-day delivery to the laboratory is not possible.

➤ **Chemical addition:**

The appropriate **chemical preservative and dose rate** can **vary** between analytes and according to container size.

Need to **consult** the analytical laboratory which chemical preservative is appropriate for the analytical technique employed by that laboratory.

Preservatives include **acidic and basic solutions**, and **biocides**.

It is important these are used in the form of concentrated solutions so that the volume of preservatives required is minimised.

All preservation procedures employed should be recorded such as on the field sheet or chain of custody form.

• **Holding times**

While **preservation** techniques can **reduce degradation** rates they **may not completely halt** changes.

All analytes therefore have a holding time, which is the **maximum time** that can elapse **between sampling and analysis**, and where the sample is unlikely to be significantly modified under the recommended preservation conditions.

Samples must be **delivered** to the laboratory **within the required holding times**.



Terima kasih