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Disclaimer

The guidance and recommendations provided in this manual are intended for competent users operating within the norms and laws of their respective countries. SST does not accept liability resulting from the use of the sample preparation manual.



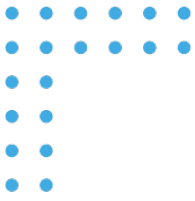
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PREFACE

As research on plastic-related pollutants grows in Africa, it will become increasingly important to use harmonized methods to prepare and analyse samples for further analyses, so that results can be compared across the continent, and even globally. As such, in 2022 Sustainable Seas Trust hosted a workshop with researchers from African countries, to demonstrate methods for preparing biological samples (in that case, fish and bivalves) for the analysis of plastic-related pollutants. At the workshop, delegates expressed an interest in methods for preparing sediment samples for plastic-related pollutants, and that is how this chapter (Chapter 4) came into being. Chapter 4 has been published as an individual stand-alone chapter, but it forms part of the 2022 edition of the Sample Preparation Manual for the Analysis of Plastic-Related Pollutants. Plastic-related pollutants can accumulate in sediment, posing a risk to benthic organisms, and therefore it is important to determine baselines for Africa as well as monitoring pollutant levels.

As with the third chapter, Chapter 4 includes many photographs taken in real-life field and laboratory conditions, to cater for researchers who may not have English as a first language or who may be new to this kind of work. Dr Brent Newman has been instrumental in developing this chapter and writes from years of experience of collecting sediment (amongst other) samples.

Dr Danica Marlin

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CHAPTER 4

SEDIMENT SAMPLE COLLECTION AND PREPARATION FOR THE ANALYSIS OF PLASTIC-RELATED POLLUTANTS

4.1. OVERVIEW

A complex mixture of chemicals is usually associated with (micro)plastics in the marine environment. These include chemicals used in the production of plastics, which are intrinsic constituents (chemical ingredients belonging to plastics). Plastics are produced by polymerizing (chemically combining) monomers, such as ethylene and propylene, to form polymers, such as polyethylene and polypropylene. Different plastic types are produced using a variety of chemicals, including solvents and certain chemicals that act as catalysts (a substance that aids in speeding up a chemical reaction). More chemicals are then added to give plastic its specific characteristics, such as flexibility, strength, colour, and ultraviolet (UV) resistance. These additives include UV-stabilisers, fillers, pigments, phthalates, 4-nonylphenol, and flame retardants^{1,2,3,4,5}. Several of these chemicals are known to be toxic to plants, animals, and humans, but there is uncertainty on the degree to which plastics and microplastics are a source of chemicals to fauna, flora, and humans. Bisphenol-A, for example, is a known endocrine disruptor⁶, while styrene and polyvinyl chloride (PVC) may be carcinogenic and/or mutagenic^{6,7}. Other contaminants in the surrounding water may also adsorb onto (attach to) (micro) plastics. These include a range of metals^{8,9,10,11,12,13}, and organic chemicals, such as polychlorinated biphenyls, polycyclic aromatic hydrocarbons, organochlorine pesticides, brominated and fluorinated flame retardants, and perfluoroalkyl acids^{14,15,16,17,18}. In this section, these chemicals are referred to as plastic-related pollutants.

Plastics that are introduced by man to the marine environment may sink after a fairly short time (hours to days), but many remain suspended in the water column or float at the surface for an extended period (months to years). Various physical processes (exposure to UV light, mechanical action) degrade large items of plastic, fragmenting them into even smaller particles until eventually they are classified as microplastics, i.e. plastic particles less than 5 mm in size^{5,6}. Microplastics are also intentionally produced, such as for cosmetic products or as nurdles⁶. Please see [Chapter 1](#) for more details on microplastics. In time, microplastics may become negatively buoyant and sink to the seafloor or riverbed. Macro- and microplastics can thus transfer intrinsic plastic-related and adsorbed pollutants to the sediment and environment^{5,19}. If the plastic-related pollutants are released from the microplastics or the microplastics are ingested, the pollutants may pose a toxic risk to benthic (organisms living in or on the sediment) and epibenthic (organisms living on or attached to hard surfaces on the seafloor or riverbed) fauna and flora (**Figure 1**).

There is currently no clear understanding on the contribution of microplastics to the chemical contamination of sediment. However, it seems logical that intrinsic and adsorbed plastic-related pollutants on microplastics will contribute to the contamination of sediment, based on the volumes of microplastic particles reported in some estuarine and marine environments^{5,6}. It is important to note that contaminants that adsorb onto microplastics, and the chemicals used in the production of plastics, have other anthropogenic sources besides the plastics themselves. This makes it difficult if not impossible, in most cases, to conclude that microplastics are the only source of the chemicals in sediment.

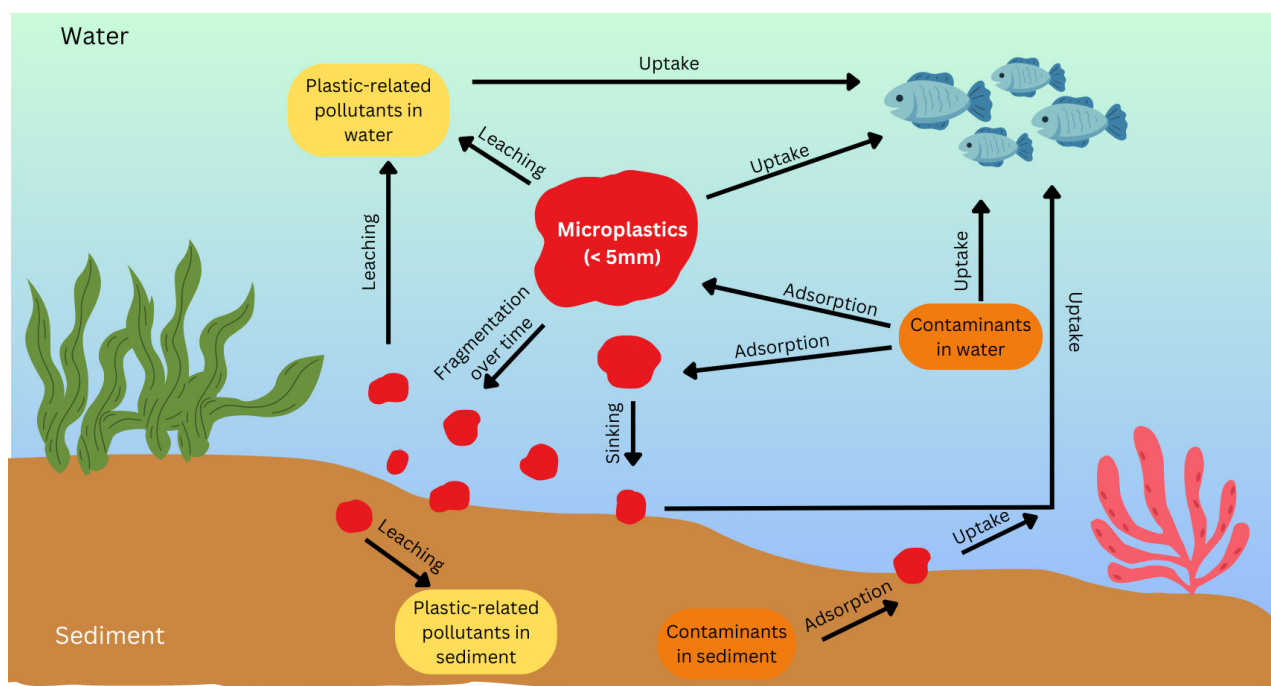


Figure 1: (Micro)plastic fate and behaviour in water and sediment (modified from <https://wasserdreinull.de/en/knowledge/microplastics/>). (Micro)plastic particles can leach plastic-related pollutants into the water and sediment, where their uptake can pose potential harm to fauna and flora. The pollutants can also be re-adsorbed to plastic particles along with other contaminants, such as metals, pesticides, and flame retardants, present in the water and sediment.

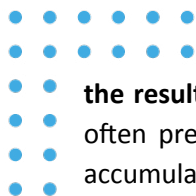
4.2. OBJECTIVES

The objectives of this chapter are to outline 1) the equipment and materials that are required and 2) the methods that should be followed to sample sediment for the analysis of plastic-related pollutants. There are many excellent guidance documents that provide information on sediment sampling, processing, and physical and chemical analysis*. Most environmental agencies in the USA publish standard operating procedures for this purpose on their websites, which are an excellent source of information. The purpose of this chapter is thus not to outline in detail sediment sampling, processing, and analytical procedures that should be followed in the field and laboratory, but rather to provide generic guidance on the sampling and processing of sediment.

4.3. SEDIMENT TYPES

Many chemicals accumulate preferentially in muddy sediment with a high particulate organic matter content (e.g. the grains of mud, or particles of organic matter such as plant or animal residues) rather than in sandy sediment. The reason is that many chemicals adsorb onto suspended particulate matter in the water column. The particles and associated chemicals eventually settle on the seabed or riverbed in areas where the current strength is so weak the particles are no longer held in suspension in the water column. The surface of mud grains and particulate organic matter is also electrically charged, which aids in the adsorption of certain chemicals. In contrast, the surface of sand grains is generally not electrically charged. Mud and particulate organic matter also present a far larger surface area per unit volume for chemical adsorption than sandy sediment. **The selection of a sediment sampling site can thus greatly influence**

*<https://www.epa.gov/foia/sediment-sampling-operating-procedure>; also see references ²⁰ and ²¹.



the results. Projects that focus on identifying and quantifying the presence of contaminants in sediment often preferentially target fine-grained, organically-rich sediment since contaminants are most likely to accumulate in this sediment type. Microplastics are fine particles, and they can reasonably be expected to also settle in areas where the current strength is weak and thus show an abundance related to sediment grain size. If so, this could be used to inform where to sample preferentially if the study aim is to sample in areas of highest microplastic abundance and associated chemicals in sediment. Studies have found that microplastics do accumulate in depositional zones, i.e. where sediment accumulates (e.g.^{22,23,24,25}), but other studies report no clear relationship between sediment grain size and microplastic abundance^{26,27,28,29}. **Studies on microplastics and their associated contaminants must thus make allowance for sampling different types of sediment** rather than favouring one type unless it has been shown in previous studies that there is a strong relationship between microplastic abundance and the type of the sediment in the study area.

4.4. SAMPLE COLLECTION AND PREPARATION METHODS

4.4.1. OVERVIEW

A generic guide for the sampling and processing of sediment for chemical analysis is provided below. The focus is largely on the sampling of subtidal sediment as this is more complicated and requires a wider range of equipment than the sampling of sediment in the intertidal zone (**Figure 2**). However, brief reference is made to the sampling of sediment in the intertidal zone.

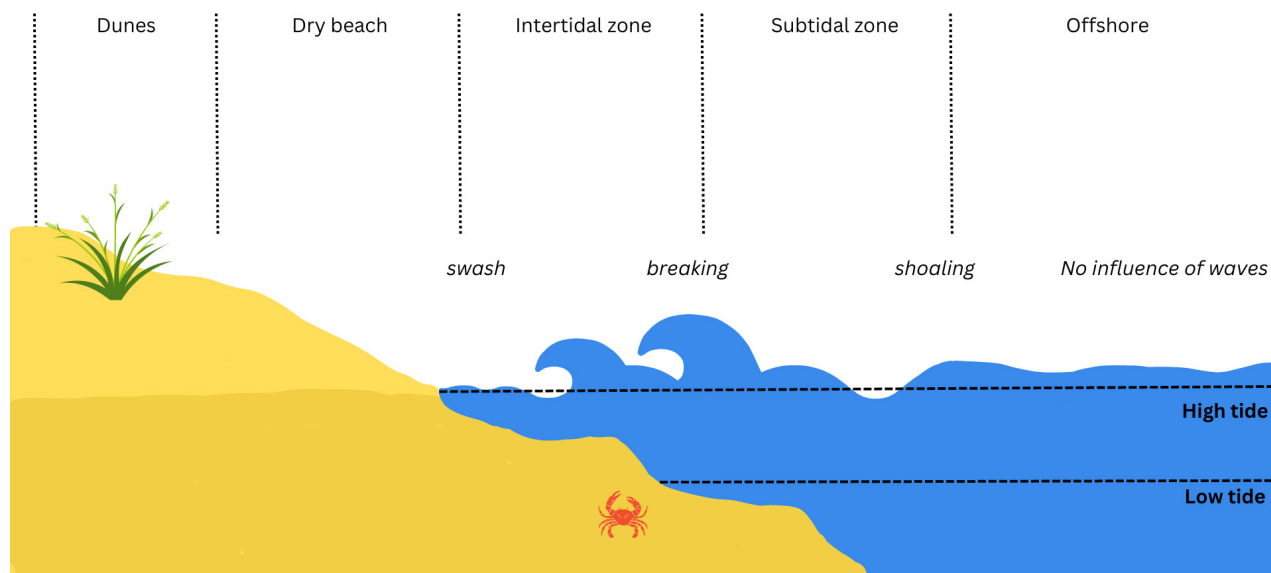


Figure 2: Subtidal and intertidal zones of the marine environment (modified from³⁰).

Figure 3 provides an overview of the main steps needed to collect and process sediment samples, with detailed methods under sections 4.4.2. *Pre-fieldwork and laboratory analysis planning and preparation*, 4.4.3. *Sediment collection*, and 4.4.4. *Sample processing*.

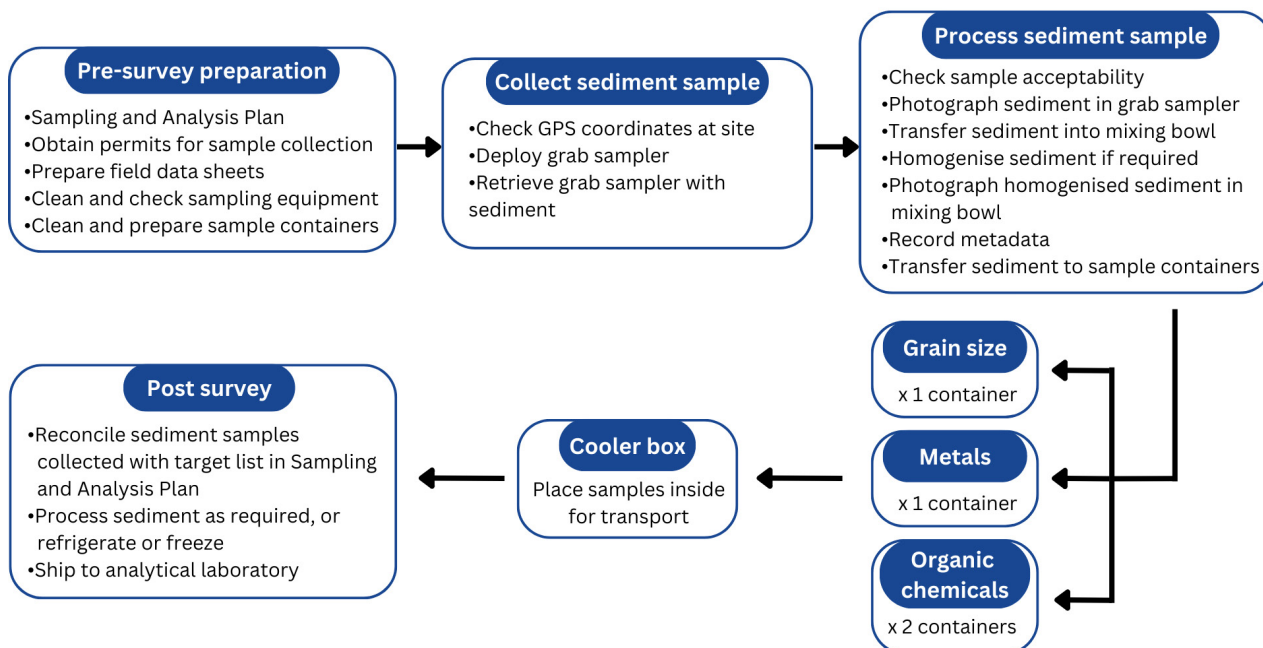
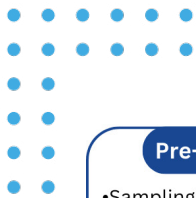


Figure 3: Summary of the methods for sediment sample collection and processing. Metadata includes data such as the date, site, sediment type, etc. of a particular sample. Organic chemicals here refer to plastic-related pollutants.



Note: the number of containers is given as an example and more can be used if needed.

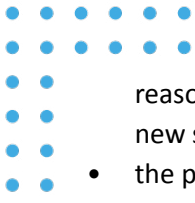
4.4.2. PRE-FIELDWORK AND LABORATORY ANALYSIS PLANNING AND PREPARATION

A considerable amount of planning and preparation is necessary before fieldwork and laboratory analyses can proceed. The tasks that should be performed and the equipment and consumables that should be prepared before fieldwork and laboratory analyses are study specific. The generic tasks, equipment, and consumables outlined below are thus not exhaustive and investigators might identify additional tasks and requirements specific to their planned study. The salient point is that proper planning and preparation before fieldwork and laboratory analyses will avoid challenges, complications, and frustration in the field, and will also avoid the manpower, logistical, and financial implications of having to repeat a survey because of poor preparation and planning.

a) Prepare a Sampling and Analysis Plan

A **Sampling and Analysis Plan** should be prepared by the lead investigator (often called the Project Leader) before fieldwork and laboratory analyses. A Sampling and Analysis Plan is a document that provides, amongst other information, the following:

- the background to and objectives for a project,
- the names, roles, and responsibilities of project participants,
- the survey vessel that will be used,
- the positions, Global Positioning System (GPS) points, where sediment will be collected (and if for some



reason sediment cannot be collected at a site, the procedures that must be followed for identifying a new site),

- the procedures that must be followed in the field for sediment collection and processing, including the appropriate material type of sample containers,
- acceptable sample holding periods before the initiation of laboratory analyses,
- the volume of sediment required by the analytical laboratory, and
- the procedures and methods that will be followed in the laboratory for the detection and quantification of chemical concentrations in the sediment.

There are many strategies for deciding on where to sample sediment in an aquatic ecosystem, including random, grid, systematic, stratified, or judgemental. It is beyond the scope of this guidance to describe these survey strategies apart from stating that the strategy identified must be informed by the objectives of the project. For example, if an objective is to map the spatial extent of sediment contamination in an area by a certain chemical, then the sediment should be sampled at positions arranged in a grid. Guidance on different types of survey strategies is provided in USEPA ³¹. In addition, the procedures that should be followed in the field and laboratory should ideally be referenced to Standard Operating Procedures (SOPs). These are procedures that outline in greater detail how different tasks should be performed, such as the procedure for operating a sediment sampling device or for cleaning sampling containers.

It will be apparent from the above that **the preparation of a Sampling and Analysis Plan requires early consultation** with project participants, including the survey vessel service provider and personnel from the analytical laboratory that will analyse the sediment. It is important to note that several laboratories might be required to analyse sediment depending on their areas of expertise and the scope of the analyses required.

Project participants must be familiar with the Sampling and Analysis Plan and referenced Standard Operating Procedures before starting project related fieldwork and laboratory analyses. An in person or virtual meeting to discuss the Sampling and Analysis plan is recommended as this might identify inconsistencies or challenges relating to fieldwork and laboratory analyses that were not identified during the preparation of the plan, which can be remedied before the project starts. This will limit the opportunity for errors and omissions in the field and laboratory due to misunderstandings on the scope of work and in this way save time and money by avoiding the need to repeat the sampling and analysis of sediment if challenges are encountered after the fact. If the field personnel are inexperienced in the sampling of sediment and/or will use new equipment for sediment sampling, then it is strongly recommended that the field personnel undertake **a practice (or 'dry') run** of the procedures that will be followed for sediment sampling and processing. If funds are a challenge the practice run can be done in shallow water along a shoreline without the aid of a survey vessel if the sediment sampling equipment is light. However, a practice run can also be done on land even if this will not entirely replicate the sediment sampling process. This will ensure that problems and challenges identified can be rectified before dedicated project fieldwork proceeds.

b) Obtain permits for sample collection

Permits or other permissions might be required for the sampling of sediment in some areas, such as in a port or a marine protected area (MPA). If the sediment will be analysed at a laboratory outside of the country of sampling, it is important to note that many countries require that the shipper include a permit for the import of environmental samples for biosecurity reasons. Permits and other permissions should be secured early in the project planning process. A Sampling and Analysis Plan provides a convenient way of communicating the study objectives, study area, and study activities to permitting authorities.



c) *Quality Assurance and Quality Control*

- It is good practice to implement a field **Quality Assurance and Quality Control (QA/QC) plan**. The Sampling and Analysis Plan should include specifications for field QA/QC, which is usually focused on determining the following:
 - small spatial scale variability of sediment in the study area, and
 - accuracy of sample processing and laboratory analysis using field duplicates.

There are three different types of QA/QC samples: 1) **Field replicates** are separate sediment samples collected at a site. They are used to provide an understanding on the small spatial scale variability in the physical and chemical properties of the sediment i.e. to determine how representative your sample is of the area. 2) **Field duplicates** are prepared from the same sediment grab sample and are used to provide an understanding on the consistency of the sample processing procedure and laboratory measurement variability (these samples may be analysed by the same laboratory, or separate laboratories). The physical and chemical measurements made on these samples should differ by only a small amount if the sediment was efficiently homogenised and split. Care must be taken to ensure the samples are split in a way that ensures sample homogeneity to the extent possible – one cannot blame a laboratory for variable results if the sample splitting procedure was poor. Field duplicates, or any other QA/QC samples, should be provided with an identifying code that is different to the primary “sample” for the site, so the analytical laboratory has no idea the sediment was sampled at the same site. 3) It is important to note that analytical laboratories usually routinely prepare **spilt samples** and **analytical blanks** to measure the accuracy and variability of the analytical technique amongst samples (investigators should understand the QA/QC procedures and requirements of the analytical laboratory that will be used). You may add your own requirements, but this will come at an additional cost – e.g. if you ask the laboratory to analyse extra samples for QA/QC purposes, this will be the same cost as running a sample.

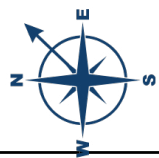
d) *Prepare a field logbook*

Field data sheets bound together to form a **field logbook** are essential for recording conditions in the field at the time of sampling, such as the weather and sea state. Field data sheets must also make allowance for:

- recording the sampling site code/identifier,
- GPS coordinates (or waypoint) of the position where sediment [waypoint (field)] was sampled if it was different to that targeted,
- the date and time of sediment sampling,
- the type of equipment used to sample the sediment,
- the type and number of storage containers into which sediment subsamples were transferred,
- the nature (e.g. sandy, muddy), colour, and aroma of the sediment sampled, and
- references to photographs of the sediment taken in the field.

An example of a field data sheet is provided in **Figure 4**.

SEDIMENT SAMPLING FIELD DATA SHEET

Project:	Durban Bay sediment contamination study			Date:	5 October 2022
Sampling Personnel:	Brent Newman (Lead), John Smith, Jane Doe			Sign (Lead):	
Site/Sample Identifier:	DBN A2/9/22 (QA/QC site)			Start Time:	08h33
Sky Cover:	Clear	Partly Cloudy	Overcast	Rain	Hard
Precipitation:	None	Drizzle	Rain	Rain	Moderate
Waypoint (Targeted):	29°53'22.37"S, 31°00'26.88"E				Wind speed
Waypoint (Field):	29°53'22.21"S, 31°00'26.73"E				Light
Collection Device:	Ponar - 5 grab samples collected (one discarded - stone caught in grab jaw)				
Photographs:	#'s 186, 187, 188, 189 (Nikon D5300)				
Sample Type:	Grain Size/TOC 200mL HDPE	Metals 50 mL HDPE	Organics 250 mL amber glass		
Number of Containers:	2	2	2		
Sediment Composition:	Shelly	Gravel	VC Sand	C Sand	Fine Sand
Sediment Colour:	Black	D/Brown	Brown	L/Brown	Grey
Sediment Aroma:	None	Sulphides	Sewage	Petroleum	Mixed
Comments:	<p>QA/QC site - extra grab samples collected Extra sample storage containers filled Sediment was highly anoxic - very strong H₂S smell and black in colour Sediment texture is mud</p>				

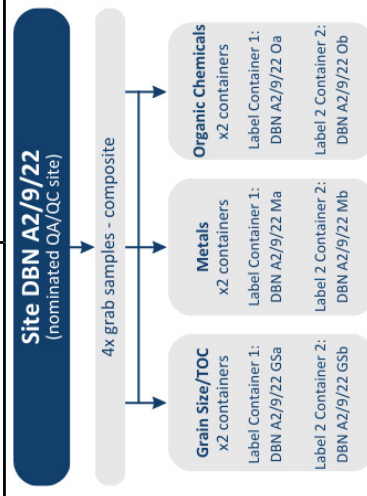
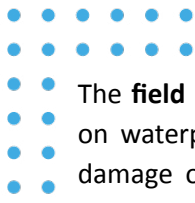


Figure 4: An example of a field data sheet, with a flow chart for different analysis types. The text and lines provide an example of the information that might be recorded in the field.



The **field data sheet** can be modified to suit the project. The field data sheets should ideally be printed on waterproof paper (e.g. Rite in the Rain® paper) as frequent handling with wet hands leads to the damage of paper field data sheets and logbooks. However, waterproof paper is expensive and might not be available to the field team. In this case, each field sheet should be placed in a plastic filing sleeve and should only be handled by field personnel with dry hands. A handy hint is to take plenty of paper towelling used in the kitchen at home so that field personnel can dry their hands before handling field notes and logbooks, and to use pencil rather than pen to write with as pen ink will run when wet.

Some investigators use a Munsell chart to record the colour of sediment against a pre-defined colour scale to minimise differences in colour perception amongst field personnel. If this approach is followed a Munsell chart must be included as part of your equipment and should ideally be laminated to avoid water and handling damage. Munsell charts are not freely available, but similar charts can be downloaded free of charge from the internet. However, a Munsell Chart developed for geological use (often not freely available) is preferable. Alternatively, investigators may define their own colours on a broad scale that are specific to the area they work in and then be consistent with defining the colours. An example of this is provided in **Figure 5**.

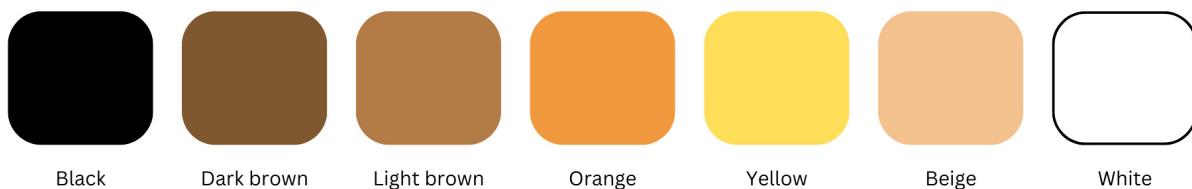


Figure 5: Colour chart example.

Field data sheets must include a section for noting observations not covered by the above. This could, for example, include notes on whether items not representative of the sediment were removed during its processing, such as plastic, plant leaves, stones, 'large' benthic macrofauna (e.g. crabs), and so on. A voice recorder can be used in the field to record observations in preference to, but ideally as a supplement to, field notes.

Observations made on field data sheets and voice recordings often prove to be invaluable during data analysis, which may occur a considerable time after fieldwork when memories on observations made and conditions in the field are vague, if not entirely lost. Field data sheets or voice recordings may, for example, allow an investigator to determine if observations in the field correlate with, or might explain the results of the physical or chemical analysis of sediment (e.g. a stormwater outfall was observed near a site). Field data sheets should be scanned and stored electronically, and voice recordings should be transcribed as soon as possible after fieldwork. A post-survey meeting with field personnel is also recommended to, amongst other issues, confirm records made in field notes and identify ways to improve field procedures in future if necessary.

e) Vessel requirements

The qualified vessel skipper should always have at hand several laminated copies of an aerial photograph of the study area that shows the sampling site positions, and a list of the sampling sites and their GPS coordinates. An example of a field map is provided in **Figure 6**. The skipper should mark off the sites as they are sampled. The GPS coordinates for each site should be programmed onto a portable (**Figure 7**) or vessel mounted GPS before entering the field and checked to verify they were inputted correctly. Site coordinates can usually be loaded easily onto a GPS electronically, minimising the chance for errors.



Figure 6: An example of a field map showing the positions where sediment is to be sampled. The vessel skipper should ideally have a separate sheet showing the order in which the sites should be sampled. In this case, the sediment at Sites A1, A2, and A3 was known from previous surveys to be the most contaminated and was sampled last to limit the possibility of contaminant transfer amongst samples.



Figure 7: An example of a hand-held GPS. These small, lightweight versions of GPSs are useful as they can be used on any type of vessel and can also be carried to log site positions when sampling in the intertidal zone along the shoreline. The drawback is they have a small screen that is difficult to read when steering a vessel. Vessels thus usually have a larger, clearly visible, and permanently mounted GPS on the steering console or in the bridge that allows the helmsman to keep both hands on the steering.

It is difficult to sample sediment at the precise GPS coordinates to those identified before fieldwork unless the utmost care is taken. An experienced vessel skipper will nevertheless be able to position the survey vessel very near the targeted GPS coordinates for a site. However, when sampling from a small survey vessel that may be anchored only from the bow, the vessel will inevitably move under the influence of wind and surface waves. The vessel anchor should thus be deployed upwind of a sampling site so the vessel drifts back onto the site as the anchor rope tightens. GPSs also have varying degrees of accuracy. Allowance should thus be made during planning for a small difference in the targeted and sampled site, and the actual site coordinates [waypoint (field)] should be recorded. Small differences are not usually of particular concern if the sediment on the bottom is homogenous. There might be situations where it is important to ensure the sampled position corresponds as closely as possible to a position identified before fieldwork. If so, a Differential Global Positioning System will be required, and the vessel may need to anchor from the bow and stern and then ‘walk’ itself onto the site by sequentially tightening the anchor ropes. This again highlights the need for proper planning and preparation before fieldwork, in this case to understand the survey vessel capabilities and to ensure a sophisticated GPS is available.



f) Check and clean sampling equipment

- Sediment sampling equipment (e.g. grabs, winches) must be checked to determine if it is in good working order well before fieldwork. This will avoid delays if equipment requires repairs or replacement parts. It is strongly recommended that such checks also be made on equipment after fieldwork is completed, to allow time to make repairs before the next round of scheduled fieldwork. Sampling equipment and cooler boxes should also be cleaned prior to fieldwork. Cleaning may include washing with a detergent solution (e.g. Alconox, Luminox) in warm water and scrubbing with a hard brush or scrubbing pad, rinsing with tap water, spraying with a solvent (e.g. acetone, isopropanol), and then again rinsing with tap water. Large, heavy items of sampling equipment, such as grabs, can also be cleaned using a (high-pressure) hose, taking care not to damage moving parts. Table 1 provides a summary of what you will need.

Table 1: Equipment list for cleaning sampling equipment.

Equipment	Cleaning materials
Grabs	Warm water and hard brush Detergent solution (Alconox, Luminox) Hose (high-pressure if available)
Winches: Manual Mechanical	Hose (wash winch drum and cable/Dymeema rope) Hose (avoiding electrical components)
Cooler boxes	Warm water and hard brush Detergent solution (Alconox, Luminox) Solvent spray (acetone, isopropanol)

g) Source and clean sample storage containers and sample processing utensils

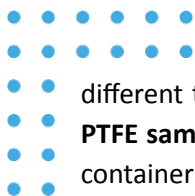
i) Overview and requirements

It is important to ensure appropriate sediment sample storage containers are sourced **before fieldwork**. Sample storage containers should be of a wide mouth type to make it easier to transfer sediment to them in the field when a vessel is moving, and to aid sediment removal in the laboratory. Examples of possible sediment sample storage containers are provided in **Figure 9**. Organic chemicals tend to adsorb onto plastic surfaces, while certain types of plastic and glass can leach chemicals into a sample. **Sample containers should thus be selected so they neither contaminate a sample nor promote the loss of chemicals through adsorption (Table 2)**. Sample storage containers for sediment that is destined for **organic chemical analysis (e.g. plastic-related pollutants, such as BPA)** should ideally be composed of **borosilicate glass with a polytetrafluoroethylene (PTFE; Teflon®) lid liner**. Borosilicate glass is chemically inert and chemically stable.



Figure 8: Example of the RIC code of HDPE.

NB: Although PTFE is a plastic, it is chemically inert and chemically stable, but it **should not be used** if the targeted chemicals are polyfluorinated organic compounds as these are constituents of PTFE. If storage containers with PTFE lid liners are not available, then **pre-cleaned aluminium** foil can be used to line the lid (dull side down). The glass should ideally be of an **amber** type to limit light penetration, which can degrade photosensitive chemicals. If amber glass containers are not available, then the containers should be wrapped in **aluminium foil** or some other opaque material to limit light penetration. Sediment that is destined for **metal and other analyses**, such as grain size and total organic carbon, can be stored in **high-density polyethylene (HDPE)** sample storage containers. If sourcing



different types of sample storage containers for **multiple chemical analyses** is challenging, then **HDPE or PTFE sample storage containers** can be used. As far as possible, however, use HDPE containers. Often the container will have the International Resin Identification Coding System (abbreviated as RIC) on the bottom; for HDPE this is number 2, which is usually shown in a three-arrow recycling symbol (**Figure 8**). There is not an RIC for PTFE. Sample storage containers with PTFE lid liners can be purchased from commercial vendors.

Table 2: Summary of equipment and material type for different analyses.

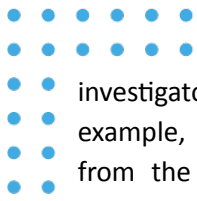
Item	Material Type	
	Metal Analysis	Organic pollutant analysis
Laboratory coat	100% cotton, acid resistant	100% cotton
Gloves	Acid resistant (elbow length)	Latex
Storage containers	HDPE or Teflon®	Borosilicate glass with Teflon® lined lid (line the lid with aluminium foil if it is not Teflon®), <i>alternatively</i> HDPE or Teflon® containers
Plastic bags	PP plastic (e.g. Ziploc)	PP plastic (e.g. Ziploc)
Utensils	Teflon®, borosilicate glass bowl HDPE scoops	Teflon®, high quality stainless steel or borosilicate glass bowl, stainless steel or HDPE scoops



Figure 9: Sediment sample storage containers should be composed of an appropriate material for the required analyses and have a wide mouth. Sediment that is destined for organic chemical analysis should be stored in glass containers. Sediment that is destined for grain size or metal analysis should be stored in high-density polyethylene containers (e.g. pill containers such as the last two

shown above). The clear glass container has a Teflon lined lid, but the lid for the amber glass container is not Teflon lined. A sheet of aluminium foil can be used to separate sediment from any potentially contaminating materials in the lid.

If sample storage containers are sourced from an analytical laboratory, these will usually be supplied pre-cleaned, but this should be verified with the laboratory. Certified clean sample storage containers can be purchased from commercial vendors, but these are not readily available in Africa and are expensive as they must be imported (cost of about US\$10-25 per container depending on size, excluding import costs; price estimate based on quotes obtained in 2022). If pre-cleaned sample storage containers are not obtained from an analytical laboratory or a commercial vendor, then



investigators must ensure they are **cleaned before fieldwork**. New sample storage containers may, for example, contain agents on their surface to release them from production moulds and contaminants from the factory where they were produced, and therefore need to be cleaned well before use.

ii) How to clean sample containers

Sample storage containers should be thoroughly **washed** in a laboratory quality non-phosphate based detergent (e.g. Alconox, Luminox) in warm water. Investigators do not need to use gloves for this purpose, but it is always recommended. The detergent should be removed completely by rinsing the containers in (running) tap water. **Important: Glass sample storage containers** should also be rinsed with a **solvent** (e.g. acetone, isopropanol) and then **soaked** in deionised water, which should ideally be replaced **three times** before the containers are dried. **High density polyethylene (HDPE) sample storage containers** can be soaked in a **10% nitric acid (HNO₃)** or **5% hydrochloric acid (HCl) solution** overnight after a detergent wash, and then soaked in deionised water, which again should ideally be replaced **three times** before the containers are completely dried. Sample storage containers and lids can be dried at a low heat in an oven, or in air covered loosely with an aluminium foil tent (**Figure 10**), in the latter case. Ensure no moisture or liquid is left behind before the containers are packed. The sample storage containers with lids on should then be packed in a plastic bag and stored in a clean cooler box or similar, ready for fieldwork (**Table 3**).

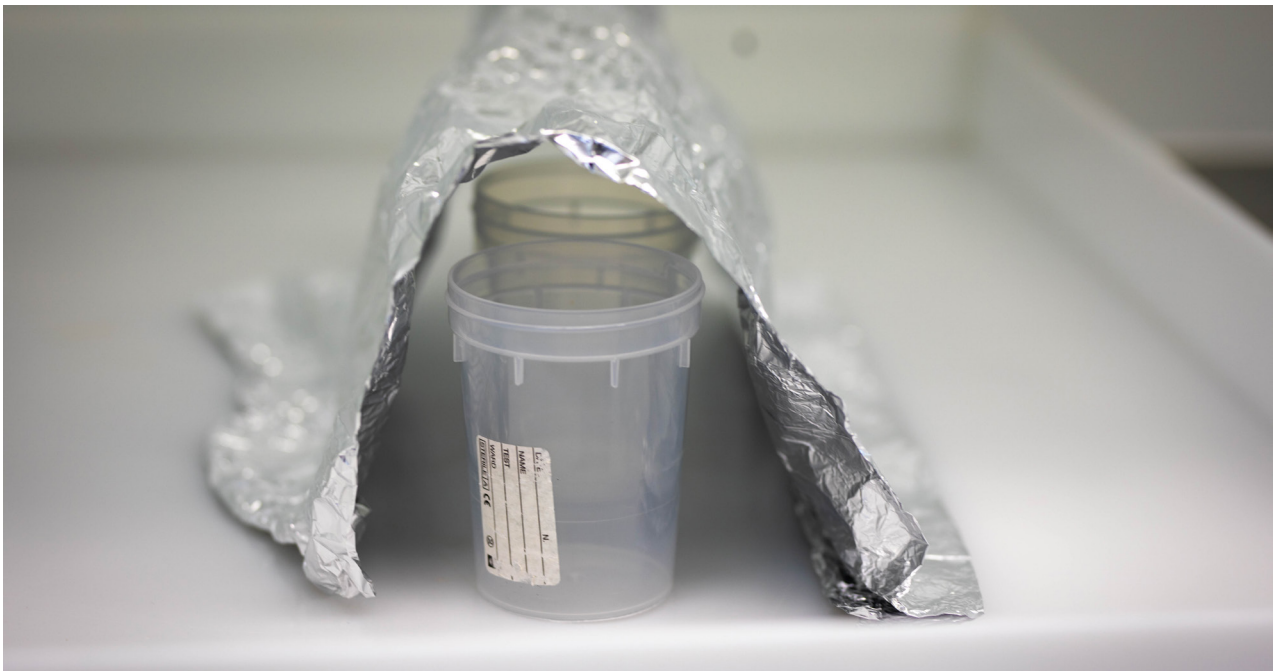
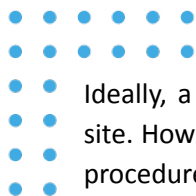


Figure 10: Sample containers drying under an aluminium tent.

iii) How to clean sample utensils

All **utensils** that will be used to process sediment in the field, such as scoops, spoons, and bowls, should be made of **inert materials** such as Teflon®, high quality stainless steel, HDPE, or borosilicate glass. These utensils should be cleaned following the same procedure for sample storage containers. They should also be **sealed in plastic bags or aluminium foil and stored in a clean cooler box or similar**, ready for fieldwork.



Ideally, a separate set of utensils should be used in the field to process the sediment sampled at each site. However, this is often not practically (e.g. limited space on survey vessel) or financially possible and a procedure must thus be formulated for the **cleaning of sediment processing utensils in the field to limit the cross contamination** of sediment amongst sampling sites. This will usually involve scrubbing the utensil with a scrubbing pad, rinsing with site water, followed by deionised water, spraying with a solvent such as acetone, and then rinsing in site water again or spraying with deionised water, whichever is convenient (**Table 3**).

Table 3: Equipment list for sample container and utensil cleaning and preparation.

YOU WILL NEED	
<input type="radio"/> Containers (check material type for analysis as per Table 2) and prepare extra containers	<input type="radio"/> Detergent solution (Alconox, Luminox)
<input type="radio"/> Sediment utensils (scoops, spoons, and bowls - check material type for analysis as per Table 2)	<input type="radio"/> Warm water and scrubbing utensil
<input type="radio"/> Cooler boxes and ice bricks/bottles	<input type="radio"/> Solvent spray (acetone, isopropanol)
<input type="radio"/> Plastic bags - thick bags for durability are preferred	<input type="radio"/> Clean aluminium foil
<input type="radio"/> Deionised or Milli-Q water	<input type="radio"/> Drying oven (optional)
<input type="radio"/> Large area to air dry cleaned containers and utensils (under cleaned aluminium foil tent)	<input type="radio"/> Acid - 10% nitric acid (HNO ₃) or 5% hydrochloric acid (HCl) solution for HDPE or Teflon®

iv) Label sample containers

Sample storage containers should ideally be **labelled before fieldwork** using a waterproof marker pen – attempting to label sample containers in the field on a small vessel when it is rocking due to wind and waves hitting the vessel is challenging and usually leads to illegible labelling. Remember that some other person will ultimately need to read the sample label, so the labels should be as legible as possible. An example of a sample storage container label is provided in **Figure 11**. Sample storage container labels must obviously be waterproof. Printing companies that print labels for milk and other containers for liquids that are refrigerated are a useful source of labels, but many commercial vendors of sample storage containers also print labels. The information on a sample storage container label should, at a minimum, include the project name, the site/sample code, and date of collection. **The sample storage container labels should cross reference to the site/sample code on field data sheets** and the Chain of Custody form used to hand the samples over to the analytical laboratory (discussed further below). If sample storage containers of the same type are to be stored together in a box and removed as required in the field, then it is recommended that the labelling includes all necessary information apart from the site/sample code, which can be added in the field. However, with proper planning the sequence of sample collection should be known before entering the field and sample containers with site/sample codes can be packed accordingly. If sample storage containers are sourced from a laboratory, these will usually be provided with a label affixed to the side. **Containers should be labelled on their sides and, if possible, also on their lids** (even if the lid label only denotes the sample/site code). Labelling on the lid assists in post fieldwork sample reconciliation and **ensures that lids are returned to the correct container** in the laboratory during sample processing and analysis. Remember to label QA/QC containers with unique identifier codes.



Note: Extra sample storage containers should always be included for fieldwork, to replace containers that might be broken or otherwise damaged and to allow for unplanned opportunistic sampling.

CSIR

Client: Project: Date:

Lab no.:

Sample ID:

Figure 11: An example of a label for a sediment sample storage container. The Sample ID box is for the unique sample code of each sample. The box named Lab no. is for the laboratory to record the unique sample reference code generated when the sample is logged into a Laboratory Information Management System.

4.4.3. SEDIMENT COLLECTION

The equipment that is used to sample sediment for physical and chemical analysis is identical to that used to sample sediment for microplastic particle analysis. Many types of equipment can be used to sample sediment depending on the objectives of the study, whether the sediment to be sampled is in the intertidal or subtidal zone, the survey vessel that will be used for sediment sampling, and the nature of the sediment (e.g. sandy, muddy). **A comprehensive field equipment check list is provided in Table 4**, based on the equipment preparation in section 4.4.2. *Pre-fieldwork and laboratory analysis planning and preparation.*

Table 4: Comprehensive sampling equipment list required for fieldwork.

ITEM		
1.	Basic health and safety equipment, including appropriate field clothing, wet weather gear, footwear, and hardhat/hat. Other health and safety equipment, including life vests, must be provided by the survey vessel operator.	<input type="radio"/>
2.	Comprehensive first aid kit.	<input type="radio"/>
3.	Lunch and drinking water – ensure all field personnel have sufficient drinking water to avoid dehydration.	<input type="radio"/>
4.	Communication devices (radio and mobile phone) pre-loaded with channels/contact numbers in the event of an emergency.	<input type="radio"/>
5.	Global Positioning System (GPS; include spare batteries) with sediment sampling site positions pre-loaded.	<input type="radio"/>
6.	Permits providing permission for sample collection that can be presented to authorities (e.g. environmental authorities) in the field if requested.	<input type="radio"/>
7.	Waterproof camera (include spare batteries).	<input type="radio"/>
8.	Clipboard with a waterproof cover.	<input type="radio"/>
9.	Sampling schedule.	<input type="radio"/>
10.	Laminated maps/aerial photographs of the study area/s, showing the positions of sediment sampling sites.	<input type="radio"/>
11.	Field data sheets (ideally on waterproof paper) in a logbook, or alternatively on a clipboard with a waterproof cover.	<input type="radio"/>
12.	Grab and deployment rope/cable (ensure deployment rope/cable is long enough to reach the bottom).	<input type="radio"/>
13.	Protective gloves if the grab is to be deployed by hand.	<input type="radio"/>
14.	Large glass bowls or other mixing containers (including spares).	<input type="radio"/>
15.	Stainless steel scoop or spoon (including spares).	<input type="radio"/>
16.	HDPE scoop or spoon (including spares).	<input type="radio"/>
17.	Sample storage containers as required (pre-labeled and including containers for QC/QA and spares).	<input type="radio"/>
18.	Spare sample labels.	<input type="radio"/>
19.	Permanent marker pen (several) – enamel paint pens are useful.	<input type="radio"/>
20.	Other stationary (e.g. pencils, eraser).	<input type="radio"/>
21.	Disposable latex gloves (sufficient for a pair per sampling site per field personnel).	<input type="radio"/>
22.	Cooler boxes with ice/dry ice/ice bricks.	<input type="radio"/>
23.	Solvent (pesticide grade acetone).	<input type="radio"/>
24.	Deionised or Milli-Q water for rinsing (several liters) and funnel for transferring it to squirt bottles.	<input type="radio"/>
25.	Squirt bottles for Deionised or Milli-Q water and solvents.	<input type="radio"/>
26.	Waste solvent collection container.	<input type="radio"/>
27.	Spare equipment and tools (e.g. replacement shackles for grab, crimping tool, spanners and wrenches, zip ties).	<input type="radio"/>
28.	Paper toweling.	<input type="radio"/>
29.	Siphoning hose made of non-contaminating material to remove overlying water from grab.	<input type="radio"/>
30.	Bin bags to collect used gloves, paper toweling etc.	<input type="radio"/>
31.	Chain of Custody form if the samples will be transported directly from the field to the analytical laboratory.	<input type="radio"/>

a) Sediment sampling habitats and equipment types

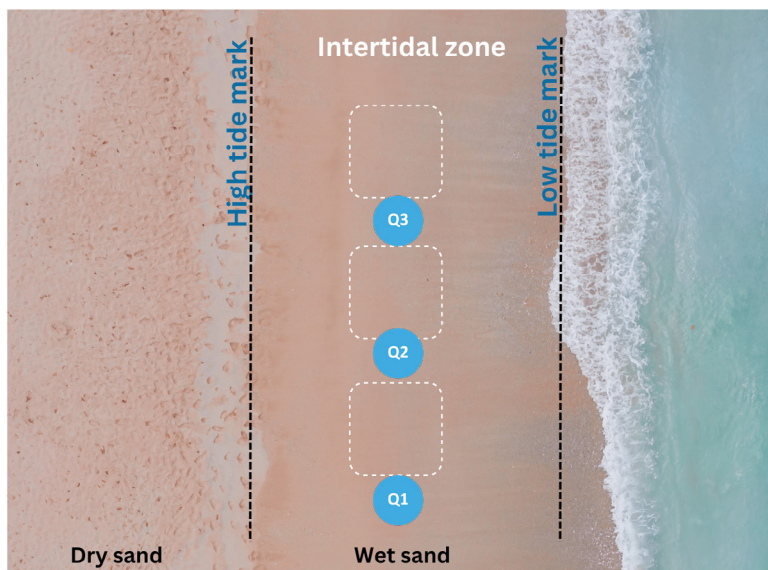


Figure 12: Illustration of sediment quadrat sampling in the intertidal zone.

Intertidal sediment is the simplest to sample because it is easily accessible during low tide. Common intertidal sediment sampling techniques include the removal of the upper layer (often to about 5 cm depth) of sediment encompassed within quadrats positioned alongshore or perpendicular to the water's edge (**Figure 12**). The quadrat size will be specific to the project. The quadrat should be of a size that provides sufficient sediment for the required analyses. The sediment is usually removed from quadrats using a stainless steel or HDPE scoop, trowel, shovel, or similar apparatus. A stainless steel or polyvinylchloride push corer can also be used if there is a need to collect sediment to a depth

somewhat below the sediment surface (e.g. Culligan et al. ³²), noting that these types of corers often lead to the compression of sediment so the length of the sediment core retrieved may not correspond to the depth to which the core was forced into the sediment. Guidance for sampling sediment in the intertidal zone can be found in ^{33,34,35}. See section 4.4.4. *Sediment processing* for methods on processing sediment samples after sample collection.

The sampling of **subtidal sediment** usually involves the use of a grab sampler or, less commonly, a coring device. Mudroch and MacKnight ³⁶ and Mudroch and Azcue ³⁷ provide useful information on the types, uses, advantages, and limitations of sediment sampling devices. A **grab sampler** is a device with hinged jaws that are locked in the open position on the survey vessel (see examples of different grabs in **Figures 13, 14 and 15**). The grab sampler is then lowered to the bottom on a cable or rope. Tension on the cable or rope ensures the grab sampler jaws remain open as it descends to the bottom. When the grab sampler jaws contact the bottom the tension on the cable or rope is released, which in turn releases the locking mechanism. When tension is again applied to the cable or rope during retrieval the jaws close, retaining a sample of sediment (there are some grab samplers that rotate into sediment, but these are not often used). The jaws of grabs often overlap to minimise sample washout. Grab samplers are generally relatively easy to operate and, depending on their design, can usually sample a range of sediment types that are of interest for physical, chemical, and biological analysis. However, grabs are often heavy and some pose risks while they are being primed for deployment and **should only be operated by field personnel that have been properly trained**. In most cases, only the top, i.e. surface, few centimetres of the sediment in the grab is removed for physical and chemical analysis.

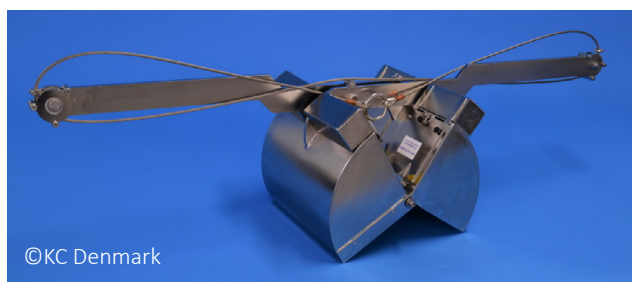
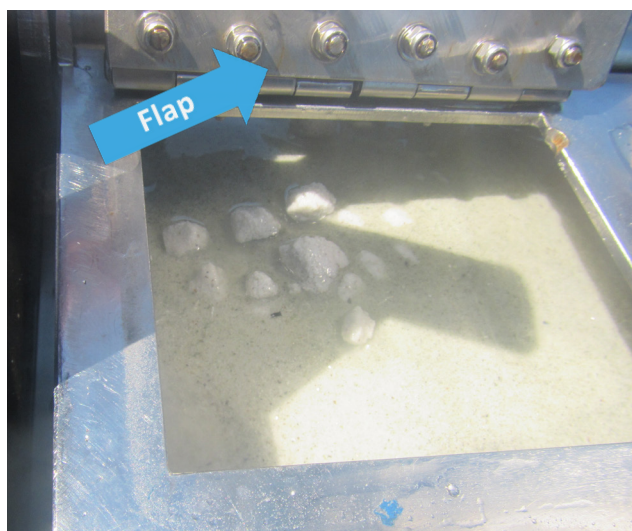
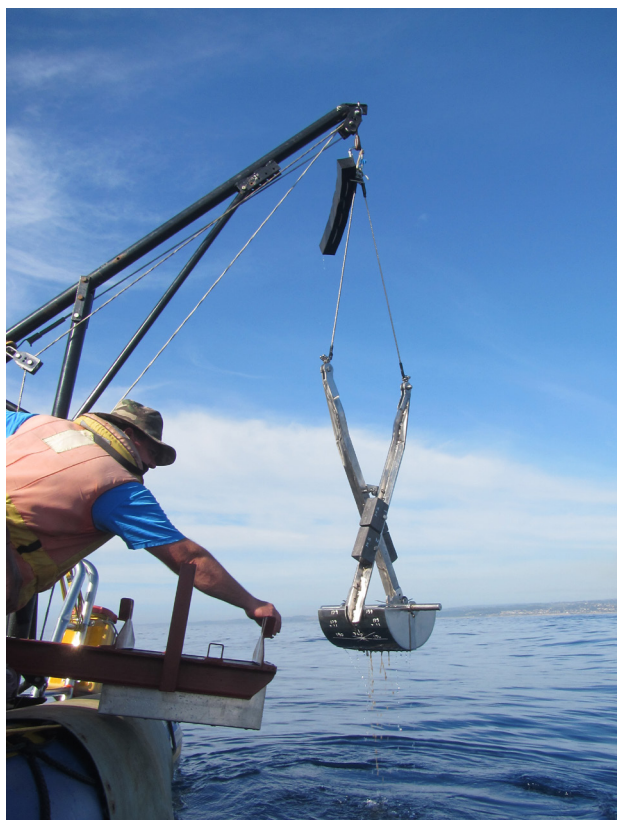


Figure 13: van Veen grab samplers. Note the top flap in the top right photo that allows access to surface sediment in the grab. The sediment in this photo was collected near a marine outfall discharging phosphogypsum (byproduct of the phosphate fertilizer production process) to the sea. The white colour of the sediment reflects contamination by the phosphogypsum and is not a sediment colour that will ordinarily be found in coastal waters.

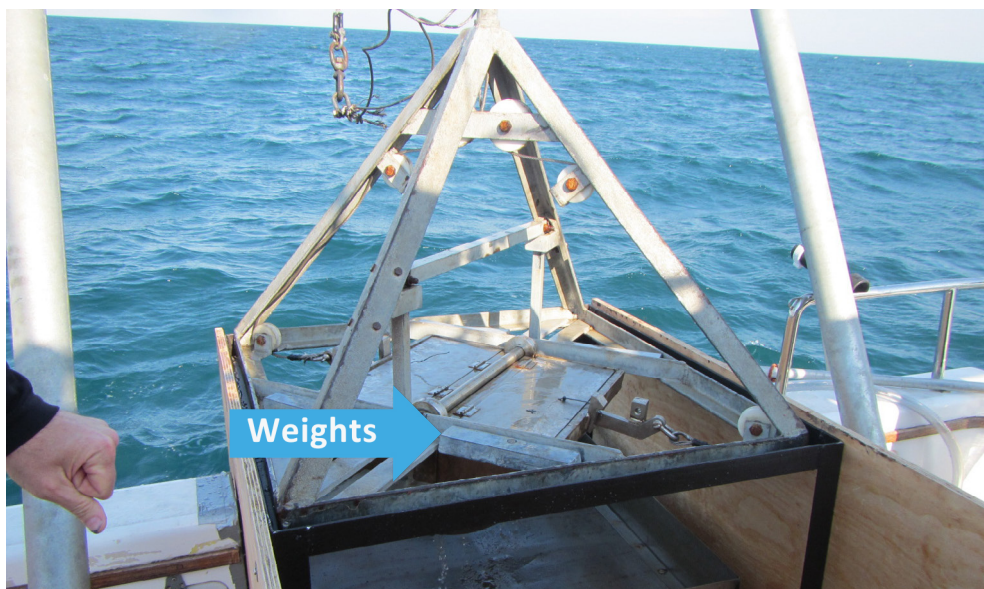


Figure 14: A Day grab sampler with a sediment sample retrieved from the seabed. Note the weights on the grab frame that can be removed or added to if required. The grab rests on a black metal grab stand. The top flaps in this grab extend across the grab width, and were closed when the photo was taken.

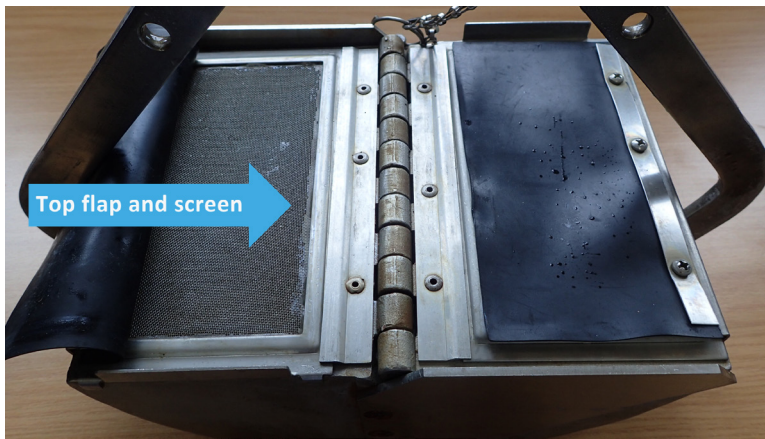
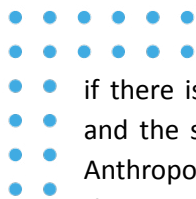


Figure 15: The petite Ponar grab is small enough to be operated by hand. Note the top flap and screen that can be removed to allow access to surface sediment in the grab to obtain your sample.

Grab samplers sample surface and near surface sediment, generally to about 5-15 cm depth depending on their size, mode of operation, and the nature of the sediment. Grab samplers thus sample sediment deposited in the Anthropocene (the current geological age). Sediment sampled using grab samplers is usually used to investigate spatial trends in the variable that is being investigated. For example, an investigator might wish to map the spatial trend in surface sediment contamination by a particular chemical across a port. A challenge with the use of grab samplers is that it is difficult to standardise the volume of sediment sampled because the penetration (or bite) of the jaws into sediment varies depending on the nature of the sediment. **Useful information:** for any grab sampler, a larger volume of sediment is usually sampled if the sediment is muddy and a smaller volume if it is sandy. This complicates direct comparisons of benthic macrofaunal communities amongst sites that are characterised by different types of sediment, but this is not often a problem for physical and chemical analyses which tend to focus on the surface few centimetres of sediment in a grab sampler. However, this may become a problem in studies that require large volumes of sediment for analysis.

There are many types of grab samplers that can be used depending on the nature of the sediment (e.g. whether it is sandy or muddy) and the depth of the water column. Amongst the most often used grab samplers are the van Veen grab, Day grab, and Ponar grab (**Figures 13, 14 and 15**). These grab samplers are available in different sizes and weights. Some grab samplers, such as the petite Ponar grab (about 12 kg when empty) and mini van Veen grab (as little as 0.5 kg when empty), are small and light enough to be operated by hand or using a lightweight davit onboard a small vessel (the davit can be operated using a small electric winch or a hand-operated winch). The disadvantage of these grabs is that they sample a relatively small volume of sediment. The sampling of sediment in deep water requires the use of a larger, heavier grab sampler (such as the Day grab sampler) since the grab samplers' weight is needed for it to pass through the water column to the bottom against currents. The empty weights of large grab samplers vary widely but most are in range of ~30-60 kg. Their weight can be increased or decreased by adding or removing weights from the grab sampler frame or arms (**Figure 14**). Due to their weight, large grab samplers can only be operated from a relatively large vessel using a crane or A-frame and an electric or hydraulic winch.

Coring devices can sample sediment to a depth of about 50 cm through to several meters depending on their design (see examples in **Figure 16**). Coring devices are usually used to investigate historical changes in microplastic abundance and chemical concentrations in sediment, provided the area is depositional over time and the sediment has not been significantly disturbed (e.g.³⁸). Coring devices are also used



if there is a need to maintain an oxygen free environment. Depending on the penetration of the corer and the site in question, the sediment sampled can include that deposited throughout and before the Anthropocene. Sediment studies using coring devices are most effective at investigating historical changes if the deposition date of sediment through the retrieved sediment core is determined using radionuclides (e.g. ^{210}Pb , ^{137}Cs). Coring devices have the advantage that they disturb sediment less than grab samplers and can be used to collect undisturbed sediment, including at the sediment-water interface, but have the disadvantage in that they sample only a relatively small volume of sediment at any given depth.

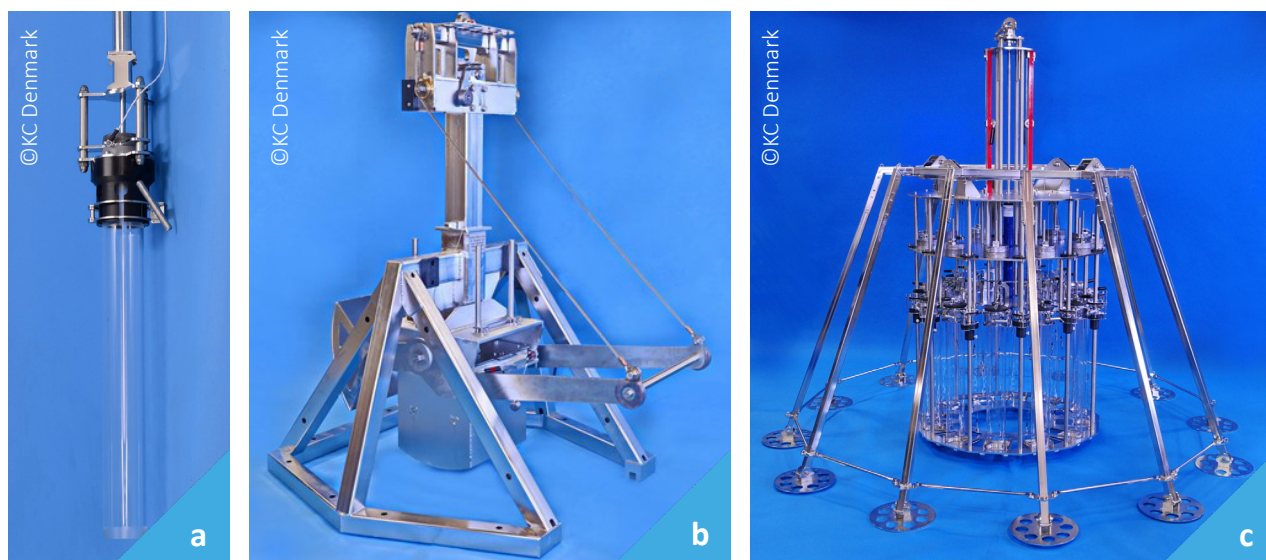
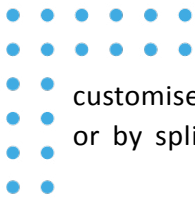


Figure 16: Examples of some sediment coring devices: hand operated corer (a), box corer (b), multi-corer (c).

There are many types of coring devices that can be used to collect sediment. In shallow waters, hand operated corers are useful as they can be operated from a small vessel (**Figure 16a**). A disadvantage of hand-operated corers is that they are usually only able to sample a small volume of sediment because the barrel is narrow, and the corer does not penetrate deeply into the sediment (usually a meter or less depending on the composition of the sediment). A solution to these problems is to use a box corer or a multi-corer, which retrieve larger volumes of sediment (**Figure 16b and c**). However, these sampling devices are too heavy to be operated by hand and can only be operated from a relatively large vessel using a crane or A-frame and an electric or hydraulic winch. Gravity, multi-, and box corers usually penetrate the sediment to a depth of up to a meter (and often less) and are usually only efficient in sampling soft, fine-grained (i.e. easily penetrable) sediment. They are far less effective for sampling sandy sediment, which becomes firmer when compressed and because of friction with the core barrel. Large coring devices can penetrate various types of sediment and are used to collect long cores of several meters. These include gravity corers, vibracorers, and piston corers. These corers are heavy and can only be operated from a vessel with a crane or A-frame and an electric or hydraulic winch. These types of coring devices are more commonly used for geotechnical surveys, or to examine trends in geochemical properties of sediment extending to periods well before the Anthropocene.

Depending on the coring device and survey vessel, sediment cores can be processed **after collection on the vessel**, or they can be held and processed **in the laboratory**. There must thus be sufficient space on the survey vessel for their cold storage if they are not immediately processed. Sediment core processing usually involves extruding the sediment from a core barrel or box corer using a



customised extrusion device and then slicing the sediment as it is extruded at defined intervals, or by splitting the core liner along its length and then extracting sediment at defined intervals.

Since **grab samplers** are more often used for the sampling of sediment, the following section focusses on generic sampling methods related to grab samplers. The grab sampler used will depend on many factors, as stated above, but most importantly the purpose of the study, the water column depth, and size of the survey vessel. Investigators must also consider that some grab samplers have surface flaps to allow access to surface sediment in the grab, but others have a permanently sealed top. **Investigators should ideally always use a grab sampler with surface flaps or screens.**

b) Preparing your grab sampler before sample collection

Prior to collecting sediment, the outside of a grab sampler should be washed with a solvent and rinsed in site water once in the field. A hard plastic brush is useful for this purpose on a small survey vessel. Larger survey vessels usually have a hose onboard that can be used to clean the grab using site water. If organic chemicals (e.g. BPA) are part of the targeted chemicals, then the inside of the grab sampler should ideally be sprayed with a **solvent, such as acetone** (it is in fact good practice to do this regardless of the pollutants that will be targeted). The solvent can be applied to the inside of the grab sampler using a spray bottle, but this is difficult if the grab is large and heavy and must thus be done from the underside. Care should be taken to retain solvent dripping from the grab sampler in this process. The grab sampler should then be rinsed in site water. If the site is shallow and the grab sampler is small, the jaws should be locked into the open position and the grab sampler should then be dipped repeatedly into the water for a site water rinse. The same process can be used for a large grab sampler, but adequate rinsing in site water will only occur if the water is deep enough, as the grab sampler is lowered to the bottom.

c) Sediment sampling using a grab sampler:

- i) There may be considerable **small-scale spatial variability** in the physical and chemical properties of, and microplastic abundance in, sediment at a sampling site. A single grab sample is unlikely to provide an estimate of the average physical and chemical properties of sediment at the site, and obviously provides no understanding of the variability. A sampling site should thus ideally be represented by **several** (most often 3, but 5 are often recommended) replicate grab samples collected in a radius of a few meters, making sure that the grab samples do not overlap. Overlapping can be avoided by moving the vessel very slightly, or by deploying the crane or A-frame different distances or directions from the side of the survey vessel. The **replicate grab samples should ideally be physically and chemically analysed separately to quantify the variability at the site**, but this adds to survey costs and many investigators thus composite (combine) the sediment from replicate grab samples for analysis. This is a cost-effective and acceptable way of estimating the **average** physical and chemical properties of sediment at a site, but it does not provide an understanding on the maximum concentration for a chemical (or other variable) in sediment at a site. Ideally, replicate grab samples collected at a subset of sites should be analysed separately to estimate sampling uncertainty due to variability in the sediment. However, this cannot be extrapolated to all sites.

Depending on the type of equipment used to sample sediment and the objectives of the study, it might be necessary to deploy the grab sampler several times to provide sufficient sediment for the required analyses. In this case, the sediment sampled by separate grab sampler deployments is composited for analysis. Thus, the top few centimetres of sediment from each grab sample are



combined. The collection of replicate grab samples might not always be feasible. For example, collecting replicate grab samples in very deep water is time consuming and the site is thus usually represented by a single sample. The collection of certain types of core samples is also time consuming and expensive and a site is thus usually represented by a single core. The grab sampler or coring device must thus be of an adequate size to provide the required volume of sediment for analyses.

ii) **Once a grab sampler is retrieved**, it is usually placed onto and secured to a sturdy grab stand on the survey vessel (**Figure 14**). The grab stand ensures the grab does not move and remains level. Large grab samplers invariably have top flaps (sometimes called trap doors) that can be opened to allow access to the surface of the sediment in the grab (**Figure 13**). The top flaps usually half-open to limit the resistance of the grab sampler as it is lowered to the bottom and in this way reduces the pressure wave when the grab sampler contacts the bottom. A pressure wave can disperse fine-grained material on the sediment surface before the grab sampler reaches the bottom. The top flaps close under their own weight when the grab sampler contacts the bottom and remain closed when it is retrieved, preventing the washout of sediment from the grab sampler. In some types of grab samplers (e.g. Ponar grab) there is a removable mesh screen beneath the top flaps to prevent the escape of mobile invertebrates and other fauna that might be captured in the sampler (**Figure 15**). In the context of sediment sampling for physical and chemical analysis, as stated above, the top flaps allow access to the surface of relatively undisturbed sediment in the grab sampler.

iii) If a grab sampler is operating efficiently, there will be clear water overlying sediment in the grab sampler when it is secured to the grab stand (**Figure 17**). The water must be removed. In larger types of grab samplers, the water is usually siphoned off using clean flexible tubing, taking care to not remove the surface sediment in the process. The grab sampler can be gently inclined to one side to aid water removal. Some types of (usually small) grab samplers do not have top flaps but rather holes in their top corners, for example the grab in **Figure 18**. The holes allow water to pass through the grab sampler as it is lowered to the bottom and for water to be expelled when the grab sampler closes. The holes also provide a convenient mechanism for bleeding water overlying sediment in the grab sampler. In this case the grab sampler contents should be allowed to settle for a short period once it is retrieved. The grab sampler should then be gently inclined to allow water overlying sediment to drain through the hole (**Figure 18**). A grab sampler with sediment and overlying water should never be 'dumped' into a container, with the sediment for physical and chemical analysis then removed from the container, since fine-grained material will invariably be suspended in the water, making it impossible to retrieve a representative subsample of the sediment. The challenge with **grab samplers that do not have top flaps** is that it is difficult to access the surface of the sediment, which as stated above, is usually the focus of physical and chemical analyses on sediment. In this case, the grab sampler can be carefully opened into a container that has the same shape as the grab sampler jaws. Once the sediment is deposited into the container, subsamples can be removed from its surface. It should, however, be noted that this procedure is not always effective because sediment may stick to the sides of a grab sampler, making it impossible to deposit an undisturbed sample.

iv) Once the overlying water is removed, a decision must be made on **whether the grab sample is acceptable**. The criteria that are used to determine grab sample acceptability include whether sufficient sediment was sampled (a minimum volume/level of fill must be specified in the Sampling and Analysis Plan), if the sediment surface in the grab sampler is level (if there is a mound on the bottom, the grab sampler may sample only part of the mound and sediment in the grab will not be level), and the absence of surface sediment disturbance indicating washout (this is often evident as a depression in the sediment in the middle of the grab sampler).



Figure 17: A retrieved grab sampler showing water overlying sediment in the grab. The presence of clear water overlying sediment in the grab sampler is one of the criteria used to accept a sediment sample for processing. The water must be siphoned off using a tube before sediment is removed from the grab.

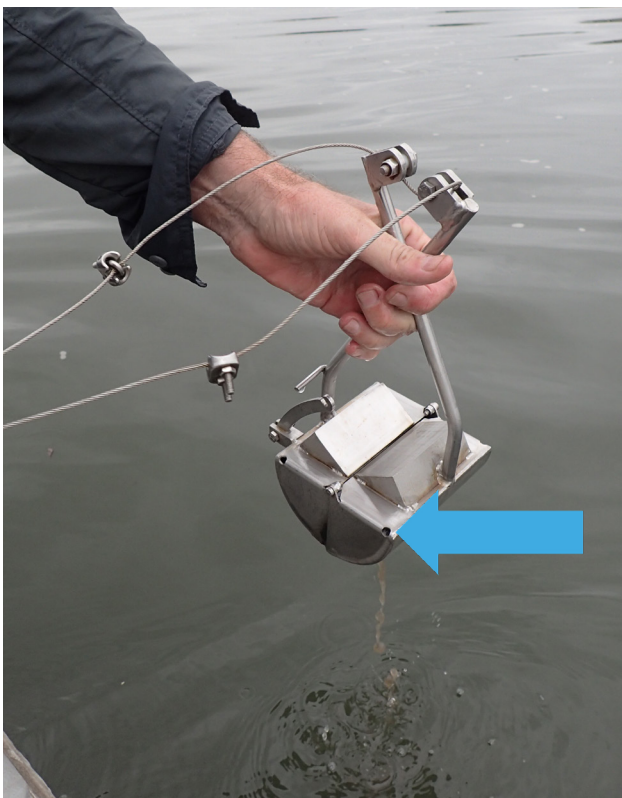


Figure 18: An example of a grab with no top flaps. Water overlying sediment in the grab is drained slowly by angling the grab toward one of the holes in the top corners.

- v) If the grab sample is accepted, **observations should be made on:**
- the sediment in the grab sampler (e.g. is there evidence for fauna or marine algal material on the sediment surface),
 - the depth of the sediment should be measured by inserting a graduated steel or plastic rod into the deepest part of the grab sampler (do this to one side of the grab sampler so any possible contaminants introduced by the ruler are not included in removed sediment), and
 - the sediment should then ideally be photographed in the grab sampler. Photography is obviously not possible for some small grab samplers that do not have top flaps and their entire contents need to be emptied into a suitable holding or mixing container (after removing overlying water).

A useful hint for photography is to first photograph the lid of a sample container or a plastic slate with the project and/or site name before photographing the sediment.

All photographs taken thereafter until the next photograph of a sample container lid or plastic slate with the project and site name will be for that site. Alternatively, the lid of the sample container or plastic slate can be placed in a position so that it and the sediment are included in a photograph.



Figure 19: Record observations on the field data sheet.

vi) After enough sediment has been removed from the grab sampler and processed (see below), the remaining sediment can be discarded. The grab sampler should be thoroughly **cleaned** using a hard brush and rinsing in site water, and then spraying the inside with a **solvent** before the collection of sediment at a new site. The grab sampler should be cleaned with a hard brush between separate grab samples at a site, but it does not need to be solvent rinsed. **Remember to collect field replicate samples for QA/QC.**

4.4.4. SAMPLE PROCESSING

If sediment must be sampled for **several types of analyses** (e.g. grain size, chemical, microplastics), a **useful hint** is to include a flow chart in field data sheets that outlines the number of grab samples that must be collected for each site and how the sediment from each grab should be processed for different analyses (including for QA/QC purposes). An example of a flow chart included in a field data sheet is provided in **Figure 4**. As discussed above, the volume of sediment sampled by a particular type of grab sampler might not be sufficient for the required analyses, especially if it is one of the small hand-operated types. In this case it will be necessary to deploy the grab sampler two or more times at the site and to then composite the sediment. The procedure that should be followed in this regard should also be documented in a flow chart.

a) What do to with your collected sediment sample in the field (e.g. on the vessel):

i) Sediment for physical and chemical analysis should be removed from the surface few centimetres of the sediment in the grab sampler using a **scoop or spoon composed of a material that will not**

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introduce the **targeted chemicals to the sediment (Table 2; Figure 20)**. This will typically be an **HDPE scoop or spoon for inorganic chemicals and a stainless steel or Teflon® scoop or spoon for organic chemicals**. The sediment should ideally be removed from the central part of the grab sampler to avoid contaminants that might be present on the sides. It should be **noted** again that some small grab samplers do not have flaps for surface access to sediment in the grab sampler and in this case the contents of the grab sampler must be emptied into a bowl or similar container for processing.



Figure 20: Sediment for analysis is usually removed from the top few centimetres of sediment in a grab. In this case, a scoop was used to remove the required volume of sediment from each quadrant of the grab (specific to the grab in this photo), and the volumes were then combined to provide sufficient sediment for analysis.

ii) The sediment should be **transferred** from the grab sampler **into a glass bowl or other mixing container** and then **homogenised** using a scoop or spoon (remember to use the right material type), noting there are circumstances where homogenisation is not recommended since this alters the physical and chemical properties of sediment (e.g. it may induce the volatilisation of certain chemicals). Homogenisation involves the mixing of sediment in the bowl or container until it is of a consistent colour and texture (**Figure 21**). A mechanical stirrer can be used to assist in homogenising sediment, but these are most often used in the laboratory and are impractical on some (small) types of survey vessel because they need a power supply. However, battery operated drills with paddles composed of non-contaminating material can be used to homogenise sediment onboard even small survey vessels. The homogenised sediment should ideally be photographed in the mixing bowl or container (**e.g. Figure 21**). Homogenisation by hand becomes increasingly difficult with larger volumes and coarser grain size sediment. In the case of sandy sediment, it is often necessary to first ‘cut’ the sediment in a mixing bowl or container by sliding a scoop or spoon through it before it can be homogenised.

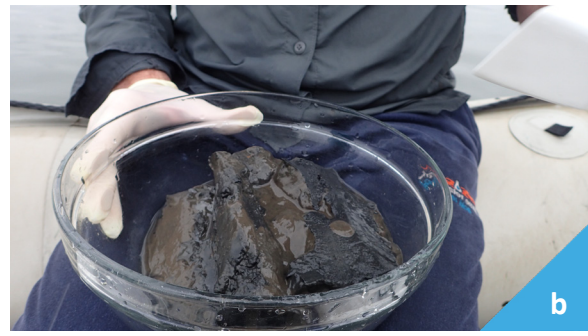


Figure 21: After transferring sediment to a mixing bowl (a) the sediment must be vigorously homogenised until it has a consistent colour and texture (b and c). The sediment can then be transferred to sample storage containers (d). Note in this example the grab does not have top flaps and the entire contents were thus emptied into the glass bowl.

iii) The homogenised sediment can be **subsampled in the field**, which involves the **transfer of aliquots of the sediment to sample containers (previously labelled) for different analyses (Figure 21(d) and Figure 22)**. Alternatively, the sediment may be transferred from a grab to a large sample storage container without homogenising it, pending its further processing and subsampling in the laboratory. If a subsampling procedure is followed in the field, **the sediment must be continuously homogenised** in the mixing bowl or container between aliquot transfer to sample storage containers. If not, some of the heavier material can sink so that layers form in the sediment. In this case, removing sediment from the surface in the mixing bowl or container will not provide a representative subsample of the sediment. Sediment processing in the field is most effectively accomplished by two field personnel, one to open and pass sample containers to the other who homogenises and then transfers subsamples of sediment to storage containers. **Remember to collect field duplicate samples for QA/QC. Clean the mixing bowl by rinsing in site water and spraying it with acetone after each use (Figure 22).**

iv) The first person then seals and transfers the containers to a cooler box. Small volumes of sediment should ideally be transferred **concurrently** amongst sample storage containers until they contain sufficient sediment for analysis rather than filling containers one at a time, to ensure the sediment distributed amongst the containers is as similar as possible.

v) If the sediment is to be **further processed in the laboratory** or if it will be stored in a **fridge** until analysis, the sample storage container should be filled with sediment to the brim to reduce oxygen exposure. If the sediment is to be frozen until further processing or analysis, the sample storage container should be filled to about 85% of its volume (i.e. leave 15% headspace) to allow for the expansion of water in the sample during freezing. Failure to provide a headspace will result in the cracking or splitting of sample containers as water in the sample expands as it freezes.



Figure 22: a) Transferred sediment samples in the appropriate sample containers for specific analyses. b) Spraying the mixing bowl with acetone before transferring the next sediment sample into it.

vi) In some circumstances it is critical to maintain the sampled sediment under **anoxic conditions**. If this is the case, the sample storage container should be purged using an inert gas (e.g. nitrogen) before filling and then again before capping the container tightly. However, this is difficult to achieve in the field if the survey vessel is small because wind will simply dissipate the gas. Many investigators do not thus follow this process but rather transfer the sediment without homogenising into an airtight storage container or take a small core from a grab that is sealed airtight until the sample can be processed in the laboratory in a glove box supplied with an inert gas.

vii) Sediment samples should be kept cool in the field by placing them in a cooler box with ice bricks, bags of ice, or dry ice, to minimise chemical changes in the sediment (**Figure 23**). **Ice bricks or dry ice** are preferred because they do not produce melted ice water, which may enter sample containers if they are not properly sealed. Lightweight sample containers may also float in the melted ice water and get damaged as they bump against other sample storage containers. If ice is used it should thus ideally be contained in a leak proof bag to avoid the cooler box filling with melted ice water. It is important to note that some analyses might require that the sediment not be frozen. The containers in which this sediment is stored should be kept clear of dry ice, which may be cold enough to cause freezing in parts of the container. If dry ice is used it must be handled with gloves and should ideally be wrapped in newspaper to prolong its life, although pelletised dry ice is often used as this it can be placed in the spaces between sample containers.



Figure 23: Samples should be stored in a cooler box in the field. In this case, the samples for each site are stored in separate bags. Note that the ice bricks have been removed from the cooler box for display purposes.

b) Notes on particle size of sediment samples

Many investigators analyse sediment as collected, that is, regardless of its particle size. However, some regulatory authorities require the reporting of chemical concentrations according to a specific fraction of the sediment (e.g. <2 mm, <62 μm). In this case, the sediment must be passed through a sieve, either in the field or more commonly in the laboratory. A common procedure for sieving wet sediment without using water is press sieving. This involves transferring aliquots of sediment onto a sieve of the appropriate mesh size and then pressing the sediment through the sieve using chemically inert paddles. This is a relatively straightforward process if the sediment is comprised of mud sized material and the mesh size is large (e.g. 2 mm), but becomes increasingly difficult and time consuming if the sediment is sandy and/or contains a 'large' amount of shell material or other debris as this tends to block the sieve. Small amounts of sandy sediment must thus be press sieved at a time.

c) Procedures on return to the laboratory

i) Sample reconciliation

The first step on returning from the field is to **reconcile the number and types of sample storage containers** filled with sediment in the field and the number and type stipulated in the Sampling and Analysis Plan (**Figure 24**). Although this should already have been checked in the field, it is nevertheless prudent to run through these checks again. If there is an omission this should immediately be reported to the project leader, who must decide on how to proceed (e.g. accept sample omission or return and collect another sample at the relevant site). The project leader must also be immediately informed if it was not possible to collect sediment at a site (e.g. because of the presence of a rocky substrate), even after following the procedure stipulated in the Sampling and Analysis Plan for alternate site identification. Once reconciled, place the samples in the fridge or freezer.

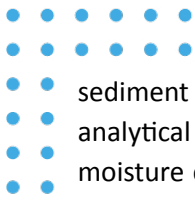


Figure 24: Samples should be reconciled in the laboratory with the Sampling and Analysis Plan and field sample log and data sheets and then transferred to a fridge or freezer. Note the site codes on the lids of the containers. The site code and additional information were also included on sample labels affixed to the sides of the containers.

ii) Sample processing in the laboratory

If the sediment was subsampled and transferred to sample containers for different types of analyses in the field, the samples can be shipped wet to the recipient analytical laboratory that will perform the chemical analyses. If the sediment was transferred to a single large storage container in the field, the investigator may need to process the sediment further for different types of analyses on return to the laboratory. **The process followed is essentially the same as subsampling in the field.** The sediment can then be shipped wet to the recipient analytical laboratory. Alternatively, an investigator might task the recipient analytical laboratory with the responsibility for processing the sediment sample for different types of analyses, but this will usually come at an additional cost. Investigators should note that sediment samples stored unfrozen must be analysed within the certain timeframe (called a **holding time or period**) after sampling depending on the analysis of concern. Samples should ideally be analysed as soon as possible after collection. Generally, extractable organic chemicals must be extracted within 7 days of collection and analysed within 30 days of extraction. Samples for metal analysis can be stored frozen for up to 6 months apart from mercury and chromium, which should be analysed within 6 weeks. Exceeding the holding period may result in changes in the sediment sample to a degree the results of analyses on the sediment after this period are **unreliable** in terms of documenting the presence and concentration of contaminants in the sediment at the time of sampling. Investigators should **ask the recipient analytical laboratory** for guidance on appropriate holding periods for sediment samples. Investigators must familiarise themselves with the holding periods and must take this time in addition to the time it takes to ship samples to the analytical laboratory into account.

The recipient analytical laboratory can be tasked to receive and analyse the sediment wet (as received). In this case the laboratory will dry a subsample of the sediment to determine its moisture content to allow the expression of chemical concentrations on a **dry weight basis**. Alternatively, the recipient analytical laboratory will **freeze dry**, and if necessary, also **ball mill** (grind/blend) the sediment before analysis. The investigator should inquire on the process followed by the recipient analytical laboratory, or task the laboratory accordingly. Ball milling is advantageous as it ensures that 'large' chunks of material (e.g. shell remains) in a sample do not dominate a subsample removed for chemical analysis. This might 'dilute' chemical concentrations in the sediment as shell material may, for example, contain low chemical concentrations but adds mass to the sample. Sediment samples that are destined for physical analysis should not be ball milled. Some investigators will **freeze dry and ball mill sediment** in their own laboratory before shipping it to a recipient analytical laboratory for analysis. Dried sediment is lighter and occupies a smaller volume than wet



sediment and does not need to be maintained at $< 4^{\circ}\text{C}$. The benefit is saving on shipping costs if the recipient analytical laboratory is some distance away. If this approach is followed the investigator must determine the moisture content of the sediment. **Freeze drying and ball milling is, however, not recommended for the analysis of certain organic chemicals.** Investigators should ask for guidance from the recipient analytical laboratory in this context. Sediment samples can also be air or oven dried, but this may lead to the sediment forming a hard 'brick' if it is muddy. The 'brick' creates problems for subsequent analyses and must either be ball milled or pulverised using a pestle and mortar. Oven drying is **not suitable** for certain types of analyses since some chemicals may be liberated from or altered in the sediment during drying at a high heat.

A useful tip: Fieldwork is often time consuming, logistically demanding, and costly, especially if done in remote areas or far offshore. It is thus often difficult and expensive, and in some cases essentially impossible to return to a sampling site for a follow-up survey. It is thus recommended that investigators sample **sufficient sediment for an archive**. The archived sediment might never be required, but if there is an accident with samples during shipment to a laboratory or the results of analyses reveal an interesting issue that warrants further investigation through additional analyses, the archived samples can, under appropriate circumstances, be used for this purpose. Archived sediment should be processed, prepared, and stored in the same way as the sediment destined for other analyses. As stated above, there are **holding periods** for different chemical analyses, even if the sediment is stored frozen. Sediment cannot thus be held forever, but if stored frozen or freeze dried the holding period for many chemicals in sediment is weeks or months, providing sufficient time to decide if further analyses on the archived sample is required.

iii) Complete Chain of Custody forms

The recipient analytical laboratory will usually provide a Chain of Custody form. This is a form used to record the sample/site name (or code) and the analyses required. The form will also include the date on which the samples were released by the sampling organisation to a shipping company or directly to the laboratory. An example of a Chain of Custody form is provided in **Figure 25**. The recipient analytical laboratory will return (a copy of) the Chain of Custody form, which will now include the time and date when the samples were received, who received the samples, and a reconciliation of the samples received amongst other information. **Investigators must specify on the Chain of Custody form what QA and QC procedures they require the recipient analytical laboratory to run on the samples in addition to those that are routinely performed by analytical laboratories** (such as the analysis of method blanks, sample duplicates, and laboratory control spikes).

The recipient analytical laboratory will usually acknowledge sample receipt via a Laboratory Information Management System via email. The acknowledgment will typically include:

- the number of samples received,
- the site codes of samples received,
- the sample matrix (in this case sediment), the condition of the samples received (e.g. temperature, all sample storage containers intact), and
- the required analyses per sample as taken from the Chain of Custody form amongst other information.

This information should be checked to ensure the laboratory has faithfully recorded the required analyses, or if analyses were inadvertently omitted when completing the Chain of Custody form.

Once the analyses are completed the analytical laboratory will provide the results, including those for QA/QC analyses. It is imperative that investigators **immediately check the results**. A recipient analytical laboratory cannot hold samples indefinitely and if any problems are identified and repeat analyses are required these should be initiated as soon as possible to remain within designated holding periods to the extent possible.

iv) Shipping samples

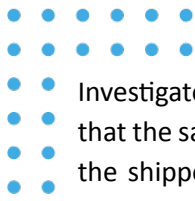
If sediment samples are to be shipped to a recipient analytical laboratory rather than hand delivered, care must be taken to maintain the integrity of the samples. **Freeze drying and ball milling** samples is recommended to ensure their integrity, if the analysis type allows it. **Keeping the samples cool** is the main priority if they are not freeze dried and ball milled before shipping. It is thus important to ship the samples in a sturdy cooler box that has a high degree of insulation. The samples can be kept cool using ice bricks, bags of ice, or dry ice. The advantage of using dry ice and ice bricks is that no water will leak from the cooler box during shipping, which may be a problem with bagged ice if the cooler box does not seal properly. If water leaks from a cooler box, most shipping companies will not ship the samples, or the shipment will be delayed until it can be established what is leaking from the cooler box. Furthermore, when ice melts, the contents in the cooler box are free to shift with the ice water if they were not tightly packed, leading to breakage. The ice water can also enter the sample containers if they were not properly sealed. The use of **dry ice** must be verified with the shipping company. Many shipping companies will not ship cooler boxes with dry ice, but specialised shipping companies will. If dry ice is used it must be handled with gloves and should be wrapped in newspaper to prolong its life if in block form.

If the samples will not reach the recipient analytical laboratory within about 24 hrs, investigators should ideally use a shipping company that will replenish dry ice in the cooler box at regular intervals to ensure the samples reach the laboratory cool.

Sediment sample containers must be packed in an upright position in the cooler box. Glass containers should be wrapped in bubble wrap to avoid breakage (**Figure 26**). A layer of cardboard and bubble wrap should be placed on the bottom of the cooler box interior to act as a shock absorber, and if relevant also between layers of samples in the cooler box. Always ensure that bubble wrap or some other packaging material is squeezed into open spaces amongst sample storage containers, especially if these are glass. The most common cause of damage to sample storage containers in transit to a recipient laboratory is when they move and bump against one another in a cooler box. Cooler boxes should never be overpacked, so they become too heavy to lift. In these cases, the shipping company personnel may manhandle the cooler box, or the cooler box handles may break, resulting in damage to the cooler box and/or the sediment sample storage containers.



Figure 26: Sample storage containers should be wrapped in bubble wrap to prevent damage while they are being shipped to the recipient analytical laboratory. The sediment samples in this example were freeze dried and ball milled before shipping. Note the site codes on the lids of the containers. The site code and additional information was also included on sample labels affixed to the sides of the containers.



Investigators must provide the recipient analytical laboratory with a warning via email and/or telephone that the samples are on their way. Most shipping companies have an online system that conveniently allows the shipper to track a shipments progress toward delivery to the recipient analytical laboratory using a waybill. It is recommended that the recipient analytical laboratory be provided with the waybill number so they too can track the shipment.

In most countries, customs authorities will not allow environmental samples without a permit for their import to enter the country for biosecurity reasons. It is imperative that investigators identify the permit requirements for the relevant country and obtain the permits well in advance of fieldwork. The permits must be provided to the shipping company.

v) Sample analyses and statistics

The procedures followed in the laboratory for the chemical analysis of sediment will depend on the targeted chemicals. These procedures should be described in the Sampling and Analysis Plan, as provided by the analytical laboratory appointed to analyse the samples. Chemicals that are additives of plastic, or that might be absorbed onto plastics are usually analysed by some form of Gas Chromatography, but occasionally also by High Pressure Liquid Chromatography. **It is important to define the QA/QC procedures** in consultation with the analytical laboratory before the collection of sediment in the field.

The recipient analytical laboratory will provide the results of chemical analyses on sediment as a **concentration per dry weight of sediment**. It is important to stipulate the **concentration units per dry weight** in scientific reports and publications, regardless of the fact this is the usual convention. The results of QA/QC procedures should also be reported. Since sediment for chemical analysis usually comprises a single sample per site, comparisons amongst sites are usually made using tables or visually in graphs rather than statistically (which requires three or more replicate samples). Other procedures that might have relevance to data analysis include the use of cumulative frequency or box plots to show the distribution of chemical concentrations measured and correlation or regression analysis to describe relationships between chemical concentrations or between chemical concentrations and other properties of the sediment (e.g. grain size, total organic carbon). Chemical concentrations are also often compared to sediment quality guidelines if these have been defined for the chemical/s of concern. Sediment quality guidelines are used to estimate the possible toxicological implications of chemical concentrations in sediment to sediment-dwelling organisms.

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