

Guidelines for the Sampling and Analysis of Cuttings Piles

OSPAR Agreement 2017-03[[1]](#footnote-1)

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# Abbreviations

|  |  |
| --- | --- |
| **AAS** | Atomic Absorption Spectroscopy |
| **AES** | Atomic Emission Spectroscopy |
| **Ag** | Silver |
| **Al**  | Aluminium |
| **APE** | Alkylphenyl Ethoxylates |
| **As** | Arsenic |
| **ASE** | Accelerated Solvent Extraction |
| **ASTM** | American Society for Testing and Materials |
| **AUV** | Autonomous Underwater Vehicle |
| **Ba** | Barium |
| **BAC** | Background Assessment Concentrations |
| **BAT** | Best Available Techniques |
| **BC** | Background Concentrations |
| **BEP** | Best Environmental Practice |
| **BGS** | British Geological Survey |
| **Cd** | Cadmium |
| **CEMP** | Coordinated Environmental Management Programme |
| **CEN** | European Committee for Standardisation |
| **CPT** | Cone penetration testing |
| **Cr** | Chromium |
| **CRM** | Certified Reference Material |
| **Cu** | Copper |
| **DBT** | Dibenzothiophene |
| **DGPS** | Differential Global Positioning System |
| **Dw** | Dry weight |
| **EAC** | Environmental Assessment Criteria |
| **ECD** | Electron capture detector |
| **ED** | Endocrine Disruptor |
| **ERL** | Effects Range Low |
| **Fe** | Iron |
| **GBS** | Gravity base structure |
| **GC-ECD** | Gas chromatography-electron capture detection |
| **GC-FID** | Gas Chromatography Flame Ionisation |
| **GIS** | Geographical Information Systems |
| **HF** | Hydrofluoric acid |
| **Hg** | Mercury |
| **ICES** | International Council for the Exploration of the Sea |
| **ICP** | Inductively Coupled Plasma |
| **IHRM** | In house reference materials |
| **INAA** | Neutron activation analysis |
| **ISO**  | International Organisation for Standardisation |
| **JAMP** | Joint Assessment and Monitoring Programme |
| **LiDAR** | Light Detection and Ranging |
| **Li** | Lithium |
| **LOD** | Limit of detection |
| **LSA** | Low specific activity |
| **M, D, TBT** | Mono, di, tri-butyl tin |
| **MARUM** | Centre for Marine Environmental Sciences of the University of Bremen, Germany |
| **Mn** | Manganese |
| **MS** | Mass Spectrometer |
| **NEA** | Norwegian Environment Agency |
| **Ni** | Nickel |
| **NOIA** | Norwegian Oil Industry Association |
| **NORM** | Naturally Occurring Radioactive Material |
| **NPD** | Collective term for naphthalene, phenanthrene, dibenzothiophene, and their C1 to C4 substituted compounds |
| **NRC** | National Research Council |
| **OBM** | Oil Based Mud |
| **OIC** | Offshore Industry Committee |
| **OPF** | Organic phase drilling fluids |
| **OSPAR** | Oslo and Paris Convention for the Protection of the Marine Environment of the north east Atlantic |
| **PAH** | Polycyclic aromatic hydrocarbons |
| **PAO** | Poly Alpha Olefin |
| **Pb** | Lead |
| **PCB** | Polychlorinated biphenyls |
| **Phi** | Base two logarithmic phi scale used for particle size distribution |
| **PLONOR**  | Pose little or no risk |
| **PSD** | Particle size distribution |
| **QA** | Quality assurance |
| **QUASIMEME** | Quality Assurance of Information for Marine Environmental Monitoring in Europe |
| **Ra** | Radium  |
| **ROV** | Remotely Operated Vehicle |
| **SBM** | Synthetic Based Mud |
| **Sn** | Tin |
| **SPLP** | Synthetic Precipitation Leaching Procedure |
| **Sr** | Strontium |
| **TBT** | Tri-butyltin |
| **THC** | Total Hydrocarbons |
| **TN** | Total Nitrogen |
| **TOC** | Total organic carbon |
| **TOM** | Total organic matter |
| **TPH CWG** | Total Petroleum Hydrocarbon Criteria Working Group |
| **UKCS** | United Kingdom Continental Shelf |
| **UKOOA** | United Kingdom Offshore Operators Association |
| **UNCLOS** | United Nations Convention on the Law of the Sea |
| **US EPA** | US Environment Protection Agency |
| **V** | Vanadium |
| **WBM** | Water Based Mud |
| **WHO** | World Health Organisation |
| **XRF** | X-ray fluorescence analysis |
| **Zn** | Zinc |

# Introduction

## Scope

These guidelines for the sampling and analysis of chemical determinands drill cuttings piles have been developed to promote a more consistent approach across OSPAR Contracting Parties and offshore operators.

OSPAR Decision 2006/5 (OSPAR, 2006) requires operators to assess cuttings piles against oil loss and persistence thresholds, based on existing data (referred to as Stage 1). If either threshold is exceeded the operator is required to characterise the cuttings pile and review the impacts (referred to as Stage 2). Characterisation in Stage 2 includes determining the location, area, topography, volume, physical characteristics, chemical content and biological characterisation of the cuttings pile.

The aim of the guidelines is to provide the operator and regulators with a framework for the characterisation and assessment of cuttings piles under Stage 2 of Decision 2006/5, ensuring that the samples collected are representative and relevant to the decommissioning process. Samples collected should also be comparable with historical data sets from the site and/or at other locations.

These guidelines therefore cover the following:

* Sampling strategy (number of samples, location and depth);
* Field sampling and handling methods; and
* Laboratory analytical techniques for:
	+ Physical parameters (e.g. particle size analysis, moisture content etc.); and
	+ Chemical determinands (e.g. hydrocarbons, metals etc.).

Biological characterisation of the cuttings pile is outwith the scope of these guidelines and detailed sampling guidelines, such as ISO 16665:2014 are available elsewhere. In addition the guidance does not cover far-field sampling to determine any effects of the drill cuttings on the area surrounding the discrete cuttings piles.

Discharges of Naturally Occurring Radioactive Material (NORM) from the oil and gas industry can also impact the cuttings piles, and previous drill cuttings studies have included the analysis of radionuclides, for example at Murchison (FUGRO/ERT, 2011). However, the analysis of NORM is considered to be outwith the scope of these guidelines.

## Methodology

The guidelines have been developed based on:

* Existing United Kingdom (UK) data and experience of sampling of drill cuttings piles, notably at Murchison (Fugro ERT, 2011), Brent (Shell UK, 2016) and NW Hutton (Gardline, 2013);
* A summary of UK work undertaken to date on cuttings characterisation (Genesis, 2016);
* A review of existing Norwegian guidelines for the characterisation of drill cuttings (Norwegian Oil Industry Association (NOIA), 2003). These guidelines are currently being updated and if there are any significant changes these guidelines should be also reviewed;
* More general environmental monitoring guidelines for the Norwegian continental shelf (Norwegian Environment Agency, 2015);
* A review of historical Norwegian cuttings piles data (Det Norske Veritas, 2004);
* OSPAR Guidelines for Monitoring the Environmental Impact of Offshore Oil and Gas Activities, (OSPAR 2004);
* JAMP Guidelines for Monitoring Contaminants in Sediments, (OSPAR, 2015);
* International standards such as ISO 5667-19: 2004 which is applicable to the sampling of marine sediments (not necessarily in relation to oil and gas impacts);
* Guidance Document No.19: Guidance on surface water chemical monitoring under the Water Framework Directive (2009); and
* Sampling and analysis guidelines used by other industries (e.g. UK onshore site investigation guidelines, UK Environment Agency 2000).

# collection of background information

## Data Requirements

A sampling strategy for cuttings piles cannot be developed unless information on the location and extent of the cuttings pile is known. Where there is no data or insufficient data on the cuttings pile location and size, this data will need to be collected prior to the development of the sampling strategy. The following data should be collected to determine the nature and extent of the required investigations:

* Location of cuttings pile;
* Area of seabed covered by cuttings pile and topography of the cuttings pile and/or an any existing estimates of the cuttings pile size;
* History of drilling at the location (number and timing of wells, discharge regime including discharge rate and depth of discharge, disturbance of cuttings pile after drilling);
* Types of discharge (OBM, synthetic based mud (SBM) or water based mud (WBM));
* Information on any known accidental discharges around the platform;
* Existing data on physical, chemical and biological characteristics of cuttings pile;
* Existing data on physical, chemical and biological characteristics of surrounding seabed; and
* Existing data on the hydrodynamic environment (water depth, currents and tides).

There are no OSPAR guidelines for mapping cuttings piles and the development of standard protocols for the collection of this data is outwith the scope of these guidelines. Mapping of cuttings piles can be achieved using standard geophysical mapping techniques commonly employed during routine site and pipeline route surveys and a list of available methods is provided below. A recent review of the assessment and decommissioning of cuttings piles in the North Sea (Oil and Gas UK in preparation) also provides details of currently available technology for the mapping of cuttings piles.

Mapping technologies include acoustic methods such as:

* Single beam echo sounder;
* Multi beam echo sounders;
* Side scan sonar;
* Sub-bottom profiler;
* Parametric sub-bottom profiler; and
* Synthetic aperture sonar.

Mapping can provide information on the area of coverage of the cuttings piles, including a topographic representation of the cuttings under and around an installation. Some of the acoustic techniques may require visual validation and this can be provided by video and still imaging of localised areas of the pile. If required, cameras mounted on a drop frame or on a remotely operated vehicle (ROV) can be used to validate the information collected using an acoustic system. Such techniques have been available and used routinely by the oil and gas industry in the North Sea for many years, however, the difficulties posed by the presence of the infrastructure (creating acoustic blind spots) and in identifying the underlying natural seabed can lead to widely varying calculations of the volume of the pile. Subsequent sampling of the cuttings pile may therefore be required to allow the depth of cuttings overlying the natural seabed to be measured and, in turn, to calculate an accurate pile volume.

There are also other emerging technologies which could be considered for mapping, including:

* Subsea Light Detection and Ranging (LiDAR), LiDAR technology uses light sensors to measure the distance between the sensor and the target object;
* Static subsea acoustic scanning systems;
* PanGeo subsea acoustic corer;
* Use of Autonomous Underwater vehicles (AUV) equipped with cameras and side scan sonar or 3-D micro seismic survey;
* Coda Echoscope real time 3-D sonar;
* Phase differencing bathymetric sonar (interferometric); and
* Optical mapping technologies.

## Screening Process

Previous guidelines on sampling and assessment of cuttings piles, such as the Norwegian guidelines (NOIA, 2003), recommended a screening process to determine which piles required further investigation. Where it is known that only WBM cuttings have been discharged it can usually be concluded that no further investigation is required. However, where existing records are insufficient to confirm whether or not OBM or SBM has been used, sampling of the cuttings pile must be undertaken.

Any proposals not to sample the cuttings pile or the seabed prior to decommissioning operations would require a robust justification, and would be considered on a case by case basis by the relevant regulatory authority.

## Development of Sampling Programme

The development of the sampling programme for cuttings piles should be aligned with the objectives of the study. The sampling programme should ensure that data collected are representative of the cuttings pile, to allow the cuttings pile to be characterised, with a view to establishing the best option for decommissioning.

The sampling programme needs to consider differences in operational practices encountered across the countries included in the OSPAR region, whilst ensuring consistency in the selection of parameters and the approach to sampling and analysis to enable the results of the investigations to be compared both over time and across a wide area.

The sampling programme should include:

* Detailed sampling strategy - number and position of sampling stations, number and depth of samples and the frequency of sampling (Section 3.0);
* Field sampling protocol and subsequent handling and storage techniques (Section 4.0); and
* Selection of chemical determinands for analysis and laboratory analytical techniques (Section 5.0).

A sampling programme should be prepared and should always be discussed with the regulatory authority prior to undertaking the field work.

# sampling strategy

The objectives of the survey should be clearly defined from the outset and the number of sample stations, station locations and number of samples at each station should be established prior to the initiation of the survey. Any sampling strategy should also incorporate a level of flexibility to allow modification to be made in the field depending on problems encountered and results obtained, whilst ensuring that the survey objectives are met. The selected location of sampling stations should allow for full characterisation of the cuttings pile.

## Number and Positioning of Sample Stations within the Cuttings Pile

The location of sample stations should generally be based on a radiating transect design with one axis along the main current direction and one perpendicular to this axis (NOIA, 2003 and OSPAR, 2004). The location of sample stations also needs to consider the results of mapping and the location of the deepest accumulation of cuttings. It is generally recommended that the number of stations should increase with increasing cuttings pile size, but conditions around specific platforms should be considered on a case by case basis. Large cuttings piles where discharges have occurred sporadically over an extended period of time are likely to have a highly heterogeneous chemical composition, both vertically and horizontally (Genesis, 2016) and therefore would require a more extensive sampling regime than more homogeneous piles.

The location of the cuttings pile relative to the platform/structure is influenced by the type of structure (e.g. steel jacket, gravity base structure (GBS), subsea structure); by whether drilling was undertaken from the platform or from a drilling vessel; by the position and height above seabed of the point of discharge; and by local hydrodynamics. For a subsea structure and a steel jacket, the cuttings pile is likely to be located within and to one side of the structure, most likely stretching out in the direction of the prevailing water current (NOIA, 2003).

## Sample Depth

In order to gain an accurate description of the physical and chemical characteristics of a cuttings pile it is useful to obtain one or more deep cores through the pile. Ideally the core(s) should penetrate to the underlying natural seabed sediments, but it is recognised that this is not always possible. It is preferable for the core(s) to be undisturbed in order to allow analyses of sediments from discrete horizons.

Existing and emerging technologies for sampling are discussed in detail in Section 4.0. The location of cuttings piles under an installation and the unconsolidated nature of the piles can make the use of coring techniques difficult. Long core samples can be obtained using existing coring techniques and a core depth of 8.7 m through a cuttings pile has been achieved on the UKCS (Marathon Oil, 2015).

More commonly, penetration depths of less than 1 m have been reached and the cores frequently do not reach the natural seabed surface sediments.

For small drill cuttings piles located within the footprint of the platform, sampling may prove particularly difficult and may be limited to shallow sampling using an ROV or from the platform deck.

## Frequency and Timing of Sampling

One of the key aims of sampling cuttings piles is to inform the assessment of decommissioning options for a specific field and associated infrastructure. The sampling should therefore be undertaken prior to submission of detailed decommissioning plans.

Any post decommissioning surveys of the drill cuttings should be agreed with the regulatory authority during decommissioning planning, and any necessary surveys undertaken at agreed intervals to determine any change to the characteristics of the cuttings pile.

# sample collection and handling

## Introduction

The choice of cuttings piles sampling method will be influenced by the cuttings piles characteristics, the water depth, field conditions in relation to the platform and cuttings pile geometry, and the type of analyses required. The type of analyses will also determines the size of the samples and the methods to be used for preserving and storing the samples. Therefore, sampling techniques should be selected on a case by case basis taking into account all of the above factors.

## Cuttings Pile Sampling Techniques

Techniques that are routinely used to sample cuttings piles in the North Sea include:

* Grabs;
* Vibrocores;
* Gravity cores;
* Box cores;
* Craib, multiple and mega corers; and
* Cone penetration testing (CPT).

Existing technology is capable of collecting a complete core of a cuttings pile but it may not be possible to sample the full depth of the cuttings pile due to the difficulty associated with deploying the sampling equipment directly below or immediately adjacent to an installation. Ideally, in order to undertake a full chemical analysis of a cuttings pile, samples from the entire depth of the pile would be required. Advantages and disadvantages of available technologies are provided in Sections 4.2.1 to 4.2.5 and of emerging technologies in Section 4.2.6.

New sampling devices may be developed and the sections below should therefore be considered as a current overview only. Up to date information on sampling devices should be sought during survey planning. However, the advantages of using new technology should be weighed against retaining consistency of equipment to monitor the cuttings pile over time. It may therefore be necessary to use two sample methods in parallel when introducing a new method.

Wherever possible the sampling methods used should be capable of sampling the cuttings pile without disturbing its structure. It is generally preferable to obtain undisturbed samples but it is recognised that the benefits of using some sampling devices may outweigh the drawback of not being able to obtain an undisturbed sample. It must also be recognised that there may be difficulties in drawing direct comparisons between samples collected with different equipment.

### Grab Samples

The grab sampler is effectively an articulated bucket which closes when it comes into contact with the seabed, collecting a sample of the surface deposits. Commonly used types of grab are the van Veen grab, the Day grab, the Smith-McIntyre grab and the Hamon grab (see ISO 16665: 2014, Annex A).

**Advantages**

* Relatively small, simple and easy to operate, although less so in the case of larger hydraulic unitsthat have the advantage of greater recovery (NOIA, 2003).

**Disadvantages**

* Sample recovery can be unreliable;
* Shallow depth of penetration;
* Disturbed sample; and
* Potential for washout of finer fraction of recovered sample rendering particle size analysis unreliable.

**Used for**

* Shallow sediment depth penetration (0.10 to 0.20 m), for chemical and physical samples where potential disturbance does not matter.

### Vibrocorer

The vibrocorer is a method of driving a steel core barrel of between 75 mm and 100 mm in diameter and approximately 3 – 6 m in length into the seabed. The equipment is deployed on a single steel lifting cable with an associated electric or hydraulic umbilical cable (NOIA, 2003).

**Advantages**

* Maximum penetration of about 6 m; and
* The method is relatively simple, inexpensive and lightweight (around 0.75 - 1.25 tonnes in air typically). The lifting capacity must allow for barrel retraction forces and the weight of the cored sediment.

**Disadvantages**

* Accurate definition of cuttings pile may be impaired by plugging, compaction or core loss, causing sections of the cuttings pile profile to be missed or misinterpreted;
* The coring method can induce disturbance with the effect that subsequent laboratory tests for parameters such as shear strength and consolidation of the cuttings pile may produce unrepresentative low values; and
* For some vessels the equipment can prove cumbersome to handle particularly for the longer barrelled models. For example, some “6 metre” vibrocorers have a total height of around 7.5 m and a maximum base width of 5 m.

**Used for**

* Collection of chemical and physical samples from greater sediment depths.

### Gravity Corers

The standard gravity corer normally comprises a core barrel, liner and cutting shoe, very similar to those used with vibrocorers. They are deployed on a single steel lifting cable and penetration is achieved by allowing the unit to free fall the last 5 to 10 metres to the seabed (NOIA, 2003).

**Advantages**

* Quick, inexpensive and simple and does not cause as much disturbance in soft clays as a vibrocorer; and
* Maximum penetration 5 to 6 m in soft sediments (note that larger gravity corers exist which can take cores of 35 to 40 m length but these are unlikely to be required for cuttings pile sampling and would require larger vessels for operation).

**Disadvantages**

* Poor penetration in stiff clays or granular sediments;
* Poor recovery in some sediments;
* A “free fall” (freewheel equipped) winch is required; and
* If a “trip-release” mechanism is used, this can be cumbersome to handle on deck and potentially dangerous because of the possibility of inadvertent triggering.

**Used for**

* Collection of relatively undisturbed chemical and physical samples from greater sediment depths.

### Box Corer

Box corers are widely used for sampling a relatively undisturbed large block of sediment. The system operates by means of a mechanical trigger once the corer has embedded itself in the seabed. After sample collection the box usually has a closed top and the bottom is sealed by closely fitting shovels mounted on pivot arms. Removable weights allow the cores to be used in a range of different sediments. The corer is recovered to the ship’s deck using either an “A” frame winch and wire or a Hiab type crane and wire. The total wire load is typically 1 to 2.5 tonnes.

**Advantages**

* Relatively undisturbed sample for biological and chemical sampling;

**Disadvantages**

* Shallow depth of penetration (typically 600 mm);
* Light box corers have limited use in sandy sediments (ISO 16665: 2014);
* Large box corers require a large vessel and sampling can be time consuming.

**Used for**

* Chemical and geochemical sampling;

### Craib, Multiple and Mega Corers

The Craib corer comprises a metal frame and single cylindrical core that is pushed gently into the sediment by means of a hydraulic mechanism. Multiple tube corers or mega corers (typically with 4 to 12 tubes) have been developed using the same principle (ISO 16665: 2014).

The core tubes are attached to the head which has a series of spring loaded arms which swing under the tubes to seal the tube bottom and at the same time plug the top of the tubes to prevent the sample being disturbed during recovery. A series of lead weights are attached to the corer head which can be adjusted to accommodate different sediment types and to regulate the depth of sample taken. The corer is lowered on a wire to the sea bed until the frame makes contact and the deployment wire becomes “slack”.  At this point the core tubes are driven into the sediment by gravity, at a controlled rate of descent governed by the hydraulic damper and the weight of lead on the corer head.

Samples are generally 600 mm long and two diameters are normally available (640 mm or 1,100 mm).

**Advantages**

* Undisturbed samples;
* Multiple samples from same location;
* Function well in soft muddy to sandy sediments;

**Disadvantages**

* Not suitable for gravelly or stony areas;
* Single Craib corer provides only small sample size.

**Used for**

* Chemical and geochemical sampling;
* Multiple corers are often used for biological sampling in deep water.

### Cone Penetration Testing

Cone Penetration Testing (CPT) involves the measurement of the resistance to the controlled penetration into the ground of a steel rod with a conical tip. The CPT is an excellent device for the logging of layering. Parameters such as sediment type, relative density, shear strength and stress history can then be derived from the direct measurements and calculated ratios using empirical correlations (NOIA, 2003).

**Advantages**

* Usually guarantees greater penetration than grab sampling, gravity corers and vibrocorers;
* Provides a complete stratigraphic profile (continuous measurement);
* Gives data in real time allowing almost immediate interpretation of ground conditions;
* Can reduce the amount of time-consuming laboratory testing;
* Provides the only reliable method of determining the relative densities of cohesionless sediments;
* Testing is very rapid and, depending on test spacing, the seabed unit may be left outboard between tests; and
* Additional sensors may be added to the cone to give indications on degree of contamination (e.g. electrical resistivity and fluorescence probes).

**Disadvantages**

* Although light weight systems are available, typically 5 tonnes to 10 tonnes units are required to ensure sufficient reaction force to achieve the desired penetration in dense sands/stiff clays;
* The technical complexity and precision engineering involved in many of the components means that it is a more costly piece of equipment and also requires several highly trained personnel to operate (However, moves towards lighter and simpler systems are beginning to mitigate the weight and cost disadvantages); and
* A competent geotechnical engineer is normally required to process and interpret the test data.

**Used for**

* Samples for collection of geotechnical data.

### Other Technologies

A number of technologies have been identified as potentially useful for the sampling of cuttings piles. These technologies are either developed but not widely available, or are emerging technologies. Although these technologies are not commonly employed for sampling cuttings piles they may be considered in future sampling programmes.

These technologies include:

* Selcore hydrostatic hammer sampling; similar to a gravity corer, this employs a hydrostatically driven hammer system to aid penetration;
* Rotary/rock core sampling; employs a rotary drilling mechanism to penetrate the pile;
* Drilling and wireline sampling; uses drilling and testing through the drill string employing a number of different sampling tools;
* British Geological Society (BGS) wireline system; retrieves core samples from within a drill string;
* “WISON XP” sampling; a downhole CPT and push sampling system that allows both CPT samples and soil samples to be taken;
* “Geobor S” coring and CPT sampling; similar to the “Wison XP” system but with higher push capacity and larger core diameter;
* “HYACE” core sampling; a coring system that uses a water-driven rotary motor to drive the core into the sediment;
* Sonic sampling; uses the resonant frequency of the formation to fluidise the sediment and allow penetration of the drill string;
* BGS 15 m multi-barrelled rock drill; a self-contained drilling system utilizing mining and ROV technology that can penetrate the seabed up to 15 m;
* MARUM (Centre for Marine Environmental Sciences of the University of Bremen, Germany) “MeBo”; a sub-sea drill rig that can sample to 80 m below the seabed. Can collect cores by push coring or rotary drilling. Weighs 10 tonnes in air so deployment within platform structure may be difficult; and
* Mini-cones; not CPTs in the strictest sense as they are smaller than the currently defined size range.

The majority of the existing and emerging techniques are, as would be expected, large and very heavy. Deploying such equipment within the confines of a platform jacket could prove difficult and require a safe and stable deployment system that could be achieved by cross-hauling. This is a tried and tested technique that has been used for many years. However, it does require careful planning if it is to be executed safely. The unconsolidated nature of the cuttings pile material also leads to problems in obtaining undisturbed cores as most core systems rely on some degree of compaction of the sediment to aid recovery.

## Field measurements

### Cuttings pile appearance

Description of the cuttings pile should be carried out in accordance with a recognised standard, for example:

* ISO 14688-1: 2002 Geotechnical investigation and testing – Identification and classification of soil, Part 1: Identification and description; and
* BS5930: 2015 Code of Practice for Ground Investigations.

The following should be described, irrespective of survey method, as soon as a sample is secured on the deck of the sampling installation or vessel (in line with OSPAR, 2004 and OSPAR, 2015):

* Colour (e.g. Munsell soil colour chart);
* Homogeneity (presence or absence of stratification);
* The presence or absence of animals (as an indication of bioturbation);
* Textural description (e.g. a initial grain size and organic content assessment);
* Surface structure (e.g. ripples);
* Smell (e.g. H2S or oil); and
* Visual contamination (e.g. oil sheen).

### Field measurements

The following measurements should be made on sample recovery (ISO 16665: 2014):

* pH, measured electronically;
* Redox, measured electronically (measure of sediment oxygenation, can be carried out along a depth profile in the sediment to assess redox potential discontinuity depth); and
* Dissolved oxygen, measured with a dissolved oxygen probe (see ISO 5814: 2012 Water Quality, determination of dissolved oxygen – electrochemical probe method).

Measurements can be carried out either:

* *In situ* within the sediment by lowering appropriately designed electrodes into the seabed; or
* On grab samples by inserting the standard electrode assembly into the sediment through the inspection lid before processing the sample; or
* On core samples by insertion of the electrodes into the top of the cores. This must be done within five minutes of exposure of the sample to atmospheric oxygen. Depending on what is being measured and the method employed it may be possible to store core samples for a few hours prior to sampling but they must be kept protected from sunlight and at a temperature as close as possible to in situ temperature.

Currently leachate analyses are usually undertaken in the laboratory some time after sampling, and they have therefore been included in Section 5. However, consideration should be given to emerging techniques for field analysis of leachates (see Section 5.4).

## Handling of Samples

The required single sample size and the handling and preservation of the sample will vary according to the type of analyses to be undertaken. The type of analyses and the nature of the seabed also influence the methods available for collecting the samples (see Section 4.2).

Sampling methods and pre-treatment, preservation and storage requirements are summarised in Table 4.1 for physical and chemical samples. There is no single protocol available covering all these aspects but the information provided is collated from a wide variety of sources, principally:

* OSPAR JAMP Guidelines for Monitoring Contaminants in Sediments, 2015;
* OGP Offshore environmental monitoring for the oil and gas industry, 2012; and
* Norwegian Environment Agency (NEA), Environmental monitoring of petroleum activities on the Norwegian continental shelf, 2015.

Cores should be transported in an upright position, and procedures should be carried out as soon as possible after the sample is taken to avoid changes in temperature and oxygen levels which can impact geochemical and chemical processes in the cuttings. If the sample is to be left in the core liner before extraction, the core liner should be capped to avoid contamination of the sample. Samples on deck should always be covered to protect them from sunlight and contamination e.g. soot particles from vessel’s exhaust (ISO 5667-19: 2004).

Samples collected offshore can often be stored for a significant length of time. The selected storage method should ensure that there is no cross contamination from the container and that potential changes to the samples are minimised. The analysing laboratory should provide standard sampling and storage procedures as part of the survey scope to ensure correct sample handling and storage. The same type of packaging should be used for all samples to be analysed for the same determinand. Staff undertaking sampling should be fully trained and experienced, where necessary with adequate supervision to ensure procedures are followed.

All procedures should follow good laboratory practice to avoid affecting the analytical results, and the handling of samples to be sent for analysis should be restricted to extraction of individual samples for sectioning.

All equipment used should be cleaned between sampling locations and should be of a suitable material to prevent cross contamination of the samples, e.g. inert plastic or stainless steel. Cleaning can be undertaken using seawater or freshwater and surfactants (ISO 5667-19: 2004).

Table 4‑1 Handling and Treatment of Samples

| **Type of sample** | **Sampling method**  | **Sample size**  | **Sub sampling, Pre-treatment and Preservation** | **Container type** | **Storage** |
| --- | --- | --- | --- | --- | --- |
| Physical parameters | Grab or corer. Use of CPT for geotechnical parameters. | Generally 0.1 m2 to a depth of 0.1 m (OGP, 2012) per sample.300 g (Norwegian Environment Agency, 2015) for grain size analysis. Larger samples are required from poorly sorted sediment. | No pre-treatment. | Can be stored in cores or liners or transferred to plastic jars, boxes or bags. Ensure fully sealed to preserve moisture. Metal containers are not recommended as they can contaminate the sample. | Freeze (< -20oc) (NEA, 2015) |
| Chemical determinands: metals and Total Organic Carbon (TOC) | Grab or corer.Where samples are to be sectioned across several depths then the corer should avoid disturbing sediment layers. | Generally 0.1 m2 to a depth of 0.1 m (OGP, 2012) per sample.Typically 50 g for metals and 100 g for TOC (Norwegian Environment Agency, 2015). Allow 500 g. | Sectioning of core by depth may be required. Further details in Appendix A based on NOIA, 2003. This should be done immediately after sampling and before any pre-treatment.Remove large debris and detritus by sieving using 2 mm sieve.Stainless steel or non-metal implements for handling (including sieving). Where a metal grab has been used, sub sampling of sediment within 1 cm of walls of sampling device should be avoided. | Glass or plastic jars. Except mercury: must be stored in glass jars as mercury can move through the walls of plastic containers (OSPAR, 2015). | Freeze (< -20oc) or freeze dried (OSPAR, 2015) |
| Chemical determinands: organic compounds | Grab or corer. Where samples are to be sectioned across several depths then the corer should avoid disturbing sediment layers. | Generally 0.1 m2 to a depth of 0.1 m (OGP, 2012) per sample.Typically 300 g (Norwegian Environment Agency, 2015) for polycyclic aromatic hydrocarbons (PAHs) and 300 g for total hydrocarbons (THCs).  | No pre-treatment. Direct exposure to sunlight or strong light should be avoided.Stainless steel or glass implements for handling. | Solvent rinsed pre-incinerated amber glass jars (OGP, 2012). | Freeze (< -20oc) or freeze dried (OSPAR, 2015) |

## Sample Identification and Records

Each sampling station must be allocated a unique reference number and repeat samples from that station must be easily distinguished from previous samples. The chosen numbering scheme should be compatible with data management and Geographical Information Systems (GIS).

The location of the sample stations needs to be accurately recorded using a differential global positioning system (DGPS) to ensure repeatability for future surveys. ISO 16665: 2014 recommends a minimum accuracy of +/- 50 m in open waters. Although ISO 16665: 2014 applies to biological samples, a minimum similar accuracy is recommended for physical and chemical sample stations.

In line with ISO 5667-19: 2004 the following information should be recorded for each sample:

* Person responsible for sampling and sub-sampling;
* Project or contract identification code;
* Geographical coordinates;
* Date and time for each sample;
* Sediment core length or grab sediment depth;
* Visual description of the sediment (see Section 4.3.1);
* Sampler used;
* Sectioning intervals;
* Water depth; and
* Meteorological data (daily).

Any conditions that could potentially affect the analytical results should also be noted in the sample log.

ISO 5667: 2004 provides an example of a form for recording details during sediment sampling.

Each sample should be clearly labelled with:

* Identification code of the sampling station; and
* Date, time, coordinates and section interval (depth).

Container information should not be pre-labelled with information as this practice reportedly increases the possibility of sample misidentification (ISO 5667: 2004).

Appropriate chain of custody documentation should be provided, filled in prior to sample despatch and copies transmitted as required (normally one copy retained by sampler, one copy placed in container for despatch and one copy sent directly to laboratory).

## Quality Assurance

For each field sampling programme, a person responsible for quality assurance (QA) should be appointed. Any deviations from set procedures should be reported and recorded.

Laboratories may require duplicate samples as part of QA processes for the laboratory analyses. Duplicate samples for chemical analyses are provided by “splitting” an individual sample into two identical samples for analysis. Duplicate samples provide a means for the laboratory to undertake QA of the sample analysis process and to determine laboratory analytical variability and/or sediment heterogeneity. Only a percentage of the duplicate samples are usually analysed for QA processes. QA within the laboratory is discussed in Section 5.4.

# selection of Determinands and techniques for laboratory analysis

It is recommended that analysis of the determinands detailed below is considered for the cuttings piles characterisation. Depending on site history and location some analyses may not be required but this should be reviewed on a case by case basis. The aim of undertaking a broad spectrum of analyses is to allow consistency across all cuttings piles’ investigations and to facilitate easier comparison of data across the OSPAR area and from year to year. It should be noted that, where appropriate, ISO standards have been referenced, but Contracting Parties may also have their own national standards or quality assurance schemes that should also be considered.

## Geotechnical and Material Properties

### Particle Size Distribution

Grain size analysis or particle size distribution (PSD) provides a description of the sediment composition in terms of the number of particles of pre-selected particle diameters.

The PSD can be determined by a combination of sieving and weighing, and sedimentation or laser diffraction, characterising the size distribution in 0.5 or 1 phi intervals from 2,000 µm to 63 µm. The silt/clay fraction (< 63 µm) is analysed by sedimentation (pipette or sedigraph) or laser granulometry. The weight of each sediment fraction should be determined to the nearest 0.01 g as the basis for calculating the cumulative weight percent distribution for each station. Median particle diameter, standard deviation, as well as skewness and kurtosis of the grain size distribution may be calculated from the same analyses (OSPAR, 2004).

Full details of the methodology are given in ISO 11277:2009 Soil quality: Determination of particle size distribution in mineral soil material - Method by sieving and sedimentation (last reviewed in 2015) and in BS 1377: 1990 Methods of test for soils for civil engineering purposes - classification tests, (N.B. partially replaced in 2010).

### Moisture Content

Moisture content is the mass of water in the sample expressed as a percentage of the mass of solids. Full details of the methodology are given in American Society for Testing and Materials (ASTM) D2216-10 Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass and BS EN ISO 17892-1: 2014 Geotechnical investigation and testing.

### Atterberg Limits Test

The Atterberg Limits are a basic measure of the critical water content of clayey materials, such as its shrinkage limit, plastic limit, and liquid limit. As dry, clayey soil takes on increasing amounts of water, it undergoes distinct changes in behaviour and consistency. Depending on the water content of the soil, it may appear in four states: solid, semi-solid, plastic and liquid. In each state, the consistency and behaviour of a soil is different and consequently so are its engineering properties.

Full details of the methodology for determining Atterberg limits are given in ASTM D4318 - 10e1: Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils and BS1377-2: 1990 Methods of test for soils for civil engineering purposes - classification tests.

### Total Organic Carbon, Total Nitrogen and Organic Matter

Total Organic Carbon (TOC), total nitrogen (TN) and total organic matter (TOM) should be determined in all samples.

Samples should be analysed for TOC content using an elemental analyser. Methods must meet limits of detection of 0.03% and limits of quantification of 0.09%. The standard method is given in ISO 10694: 1995 Soil quality – determination of organic and total carbon after dry combustion (elementary analysis).

TN is measured after high temperature (1,800 oC) combustion of crushed, freeze dried material in a CHN-elemental analyser. The standard method is given in ISO 11261: 1995 Soil quality - Determination of total nitrogen -- Modified Kjeldahl method.

Organic matter is analysed as weight loss of the dry sample after incineration at a controlled temperature of about 500 oC (loss on ignition). Pre-treatment with hydrochloric acid to remove inorganic carbon (i.e. carbonate) is recommended (NOIA, 2003) or using a sulphurous acid digest (Marine Management Organisation, 2015, see: <https://www.gov.uk/government/publications/marine-licensing-physical-and-chemical-determinands-for-sediment-sampling/physical-determinands>).

The measurement and subsequent removal of organic matter should not be undertaken prior to particle size analysis as organic matter is considered an integral component of the sediment (NMBAQC, 2016). Pre-treatment to remove inorganic carbon is also not recommended prior to particle size analysis, as shell debris can be component of sediment.

## Chemical Determinands

### Benchmark Concentrations

OSPAR has defined Environmental Assessment Criteria (EAC) as part of their Coordinated Environmental Management Programme (CEMP). EACs represent the contaminant concentration in the environment below which no chronic effects are expected to occur in marine species, including the most sensitive species. Therefore, concentrations below the EACs are considered to present no significant risk to the environment. EACs for a range of contaminants were proposed in 2004, and updated EACs for polychlorinated biphenyls (PCBs) were proposed in 2008. Further EACs continue to be developed for use in data assessments (OSPAR, 2000, 2009a and 2009b). Where EACs are not available Effects Range Low (ERL) values developed by the US Environmental Protection Agency (US EPA) can be used instead. ERLs are defined as the lower tenth percentile of the data set of concentrations in sediment which were associated with biological effects. Adverse effects on organisms are rarely observed when concentrations fall below the ERL value. EACs and ERLs are not directly equivalent but ERLs can be used for sediment assessment where recommended EACs are not available (OSPAR 2009c).

Under CEMP, OSPAR has also defined Background Concentrations (BC) and Background Assessment Concentrations (BAC). BCs represent concentrations of substances at remote or pristine sites free of anthropogenic inputs, and describe the environmental conditions that would be expected in the absence of industrial developments. BCs for man-made substances are zero. BCs are based on historical and contemporary data and are applied across the OSPAR maritime area. There will be substantial variability in the natural background concentrations of contaminants across the maritime area because of differing geological or oceanographic characteristics. Local conditions of the study area should therefore be taken into account when assessing the significance of any exceedance of a BC.

Where there have been difficulties in assembling a dataset from remote or pristine areas to derive BCs, low concentrations (LC) are used (OSPAR, 2009c). These are concentrations from areas considered to be remote but which could not be guaranteed to be outside the influence of long range atmospheric transport of contaminants.

BACs are statistical tools defined in relation to the BCs which enable statistical testing of whether observed concentrations can be considered to be near background concentrations. BACs are calculated according to the method set out in Section 4 of the CEMP Assessment Manual (OSPAR, 2008). The outcome of this method is that, on the basis of what is known about variability in observations, there is a 90% probability that the observed mean concentration will be below the BAC when the true mean concentration is at the BC. Where this is the case, the concentrations can be regarded as “near background” (for naturally occurring substances) or “close to zero” (for man-made substances).

The selection of parameters for analysis of samples from cuttings piles has been made on the basis of known contaminants present in drilling muds, taking into consideration the availability of recognised background levels. This allows the analysis results to be put into a meaningful context for assessment.

### Endocrine Disrupters

The World Health Organisation (WHO) International Programme on Chemical Safety (WHO/PCS/EDC/02.2, WHO, 2002) defines an Endocrine Disruptor (ED) as “an exogenous substance or mixture that alters the function(s) of the endocrine system and consequently causes adverse health effects in an intact organism, or its progeny, or (sub) populations”.

EDs are often pervasive and widely dispersed in the environment. Some are persistent, can be transported long distances across national boundaries, and have been found in virtually all regions of the world. Others are rapidly degraded in the environment or human body or may be present for only short periods of time but sometimes at critical periods of development. (WHO, 2013).

ED chemicals include polychlorinated biphenyls (PCBs), alkylphenyl ethoxylates (APEs) and the mono, di, tri-butyltins (M, D, TBT) discussed in Sections 5.2.6, 5.2.7 and 5.2.8 respectively.

### Metals

**Determinand Selection**

Drilling cuttings can contain substantial amounts of barium sulphate (barites) as a weighting agent. Barites also contains measurable concentrations of heavy metals as impurities including cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb), mercury (Hg) and zinc (Zn) (National Research Council (NRC), 1983). Heavy metals are also present in other mud components such as clays, either as impurities or additives (both arsenic (As) and nickel (Ni) are additives used in drilling muds).

There are inputs of metals into the water column and marine sediments from other anthropogenic sources, including riverine inputs, sea-based inputs (e.g. offshore oil and gas industry activities other than drilling, mariculture and the disposal of dredged materials), (OSPAR, 2000) and atmospheric inputs. OSPAR has identified Cd, Pb and Hg as pollutants for which reducing concentrations in the marine environment is a priority.

The most abundant metal in the majority of drilling muds is barium (Ba). It has a low solubility in seawater and a low bioavailability and toxicity to marine organisms and is not expected to cause harm to the marine environment, and barites is classified as a PLONOR (pose little or no risk) chemical (i.e. not expected to cause harm to the marine environment and is on the OSPAR PLONOR list) (ERMS, 2006). However, it should be included in the determinands analysed because of the large volumes of barites used in many types of drilling muds. In addition, it can give a good indication of the likely extent of contamination as previous studies have shown excellent correlation between Ba concentrations, other heavy metal concentrations and hydrocarbon concentrations.

In any study of cuttings piles it is important to be able to distinguish between background concentrations in the sediment (which may originate from other anthropogenic sources) and contamination resulting from the drill cuttings.

The available BCs and BACs for heavy metals are shown in Table 5-1.

Table 5‑1 OSPAR heavy metal benchmark concentrations

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Heavy metal** | **BC1 mg/kg dry weight (dw)** | **BAC2 mg/kg dw** | **EAC mg/kg dw** | **ERL mg/kg dw** |
| Arsenic | 15 | 25 | - | - |
| Cadmium | 0.2 | 0.31 | 0.06 | 1.2 |
| Chromium | 60 | 81 | - | 81 |
| Copper | 20 | 27 | - | 34 |
| Lead | 25 | 38 | 2.2 | 47 |
| Mercury | 0.05 | 0.07 | 0.220 | 0.15 |
| Nickel | 30 | 36 | - | - |
| Zinc  | 90 | 122 | - | 150 |

Sources: 1 OSPAR, 2009a and 2 OSPAR, 2009b

Notes: BC and BAC are both normalised to 5% Aluminium, and EAC is normalised to 1% TOC.

BACs for Spain and Portugal differ from the values shown in the table above and should be checked in OSPAR, 2009b.

All cuttings piles analyses must include Ba (no benchmark concentrations have been developed) and the metals listed in Table 5-1.

Other metals (e.g. vanadium (V), silver (Ag), iron (Fe), manganese (Mn), tin (Sn) and strontium (Sr)) have all been included in some previous surveys and can be included at the discretion of the operator.

Lithium (Li) and aluminium (Al) must also be included to allow for normalisation of analyses (see following section).

**Analytical methods**

Available analytical methods for determination of metals in sediments are set out in detail in OSPAR, 2015, Technical Annex 6. If there are high concentrations of barium present in drill cuttings then different extraction techniques need to be considered (Hartley, 1996).

Trace metals may occur in both fine and sand fractions of sediments. However, most natural and anthropogenic substances (metals and organic contaminants) show a much higher affinity to fine particulate matter than the coarse fraction.

Total methods, such as procedures involving total dissolution of sediment samples with hydrofluoric acid (HF) prior to analysis, or non-destructive methods without digestion such as neutron activation analysis (INAA) and X-ray fluorescence analysis, can be used to determine total trace metal content in the whole sediment sample. In contrast, methods using a partial digestion with only strong acids, e.g. nitric acid or *aqua regia*, mainly measure trace metals in the fine fraction, and only extract small amounts of trace metals from the coarse fraction.

As noted previously, metals show a much higher affinity to fine particulate matter compared to the coarse fraction and this is further influenced by the presence of organic matter and clay minerals which contribute to the affinity. It is, therefore, essential to normalise for the effects of grain size or organic carbon in order to provide a basis for reliable assessments of temporal trends and for meaningful comparisons of the occurrence of substances in sediments of variable bulk properties, with background assessment criteria and environmental assessment criteria derived for a defined sediment composition.

OSPAR 2015, Annex 5 provides a detailed discussion of possible approaches to normalisation. The preferred approach is for analyses to be undertaken on the <63 µm wet-sieved fraction, followed by normalisation of analytical results using cofactors such as grain size and organic carbon content. Aluminium and lithium which are associated with the clay fraction can also be used as cofactors and should therefore be included in the metal analyses.

Analysis of metals in solution resulting from acid digestion should be undertaken using multi-element techniques such as Inductively Coupled Plasma (ICP) attached to either an emission spectrometer (ICP-AES) or a mass spectrometer (ICP-MS), which allows rapid analysis of a wide range of metals. The standard method is described in US EPA method 6010C ICP-AES, (US EPA, 2000).

The selected analytical method should take into account differing limits of detection (LOD) to ensure that the LOD for the selected method is low enough for the required assessment. LODs for different methods are given in OSPAR 2015, Annex 6 and are reproduced in Table 5-2.

Table 5‑2 Typical LODs for the determination of trace metals with different techniques

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Method** | **Al** | **Li** | **As** | **Cd** | **Cr** | **Cu** | **Hg** | **Ni** | **Pb** | **Zn** |
| AAS / flame | 5 | 0.2 |  | 0.5 | 5 | 2 |  | 5 | 5 | 10 |
| AAS / graphite furnace, hydride technique, cold vapour | <1 | <1- | 0.2 | 0.02 | <1 | <1 | 0.05 | <1 | <1 | - |
| ICP – AES  | 10 | 10 | 10 | 0.5 | 1 | 1 | - | 2 | 5 | 1 |
| ICP – MS | 40 | 0.1 | 1 | 0.01 | 0.2 | 0.1 | 0.05 | 0.2 | 0.2 | 2 |
| X-ray fluorescence analysis (XRF) | 1,000 | - | - | - | 10 | 10 | - | 10 | 10 | 20 |
| Neutron activation analysis (INAA) | - | - | 0.3 | 1 | 0.8 | - | 0.1 | - | - | 2 |
| Fluorescence spectrometry | - | - | - | - | - | - | 0.1 | - | - | - |

Source: OSPAR, 2015

Notes: Units in mg/kg dw based on typical sample intakes (0.5 – 1 g)

### Total Hydrocarbons and Assessment of Hydrocarbon Material

**Determinand Selection**

THC is the basic parameter used to estimate the total amount and distribution of oil present in a cuttings pile.

The United Kingdom Offshore Operators Association (UKOOA) drill cuttings initiative (UKOOA, 2002) and OSPAR Recommendation 2006/5 (OSPAR, 2006) defined the following thresholds:

* The potential environmental impact is considered significant if the rate of loss of hydrocarbons to the water column from a cuttings pile is greater than 100 Te/year; and
* The potential environmental impact is considered insignificant if the rate of loss of hydrocarbons to the water column is less than 10 Te/year and if the area of seabed impacted by THC at 50 mg/kg over time is less than 500 km2/year.

The THC 50mg/kg threshold is used as the likely concentration above which biological impact from the cuttings can be discerned (UKOOA, 2002). No BCs or BACs have been defined for THC.

**Analytical Methods**

Total hydrocarbon content is measured by gas chromatography-flame ionisation detection (GC-FID). The standard method is described in Analysis of Petroleum Hydrocarbons in Environmental Media, Total Petroleum Hydrocarbon Working Group Series, Volume 1, March 1998 and US EPA Method 8015B, Revision 2, December 1996 Non halogenated Volatile Organics Using GC-FID. GC-FID typically detects carbon ranges between C6 and C36, with a LOD of 10 mg/kg in soil (US EPA, 1996a) and the chromatogram allows product identification to be carried out.

The overall distribution of hydrocarbon components in the gas chromatograms can be used to determine the origin of the hydrocarbon material present in the samples. This should allow different types of drilling fluids to be identified (i.e. diesel, synthetic and low toxicity mineral oil based muds).

### Polycyclic Aromatic Hydrocarbons

**Determinand Selection**

PAHs arise from incomplete combustion processes and from both natural and anthropogenic sources, although the latter generally predominate. PAHs are also found in oil and oil products, and these include a wide range of alkylated PAHs formed as a result of diagenetic processes, whereas PAHs from combustion sources comprise mainly parent (non-alkylated) PAHs.

The US EPA have identified 16 priority PAHs of which nine were selected by OSPAR (OSPAR, 2000) as the focus for their studies. These are the 4 to 6 ring compounds and are of particular importance due to their toxic nature even at very low concentrations. CEMP benchmark concentrations have been developed for the nine OSPAR priority PAHs plus naphthalene and dibenzothiophenes (DBT). The CEMP benchmark concentrations are shown in Table 5-3.

Table 5‑3 OSPAR PAH benchmark concentrations

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **PAHs** | **BC1 µg/kg dw** | **BAC1 µg/kg dw** | **EAC1 µg/kg dw** | **ERL1 µg/kg dw** |
| Naphthalene | 5 | 8 | 43 | 160 |
| Phenanthrene | 17 | 32 | 1250 | 240 |
| Anthracene | 3 | 5 | 78 | 85 |
| Fluoranthene | 20 | 39 | 250 | 600 |
| Pyrene | 13 | 24 | 350 | 665 |
| Benzo (a) anthracene | 9 | 16 | 1.5 | 261 |
| Chrysene | 11 | 20 | - | 384 |
| Benzo (a) pyrene | 15 | 30 | 625 | 430 |
| Benzo (ghi) perylene | 45 | 80 | 2.1 | 85 |
| Indeno (1,2,3-cd) pyrene | 50 | 103 | 1.5 | 2404 |
| DBT | 0.6 | - | - | 190 |

Sources: 1 OSPAR, 2009a, 2009b and 2009c

Notes: BC, BAC and EAC normalised to 2.5% TOC,

BACs for Spain and Portugal differ from values shown in the table above and should be checked in OSPAR, 2009b.

**Analytical Techniques**

Available analytical methods for determination of PAHs in sediments are set out in detail in OSPAR, 2015, Technical Annex 3.

PAHs should be determined by gas chromatography / mass spectroscopy (GC-MS), and results should be reported for individual components, sum NPD (the collective term for naphthalene, phenanthrene, dibenzothiophene and their C1 to C4 substituted compounds) and sum PAH. A standard analytical method is provided in EPA Method 8270C Semivolatile organic compounds by GC-MS (US EPA, 1996b).

### Polychlorinated Biphenyls

**Determinand Selection**

PCBs are synthetic mixtures of chlorinated hydrocarbons used extensively in Europe from 1929 until the mid-1980s. PCBs suitability for industrial applications is related to their chemical inertness, resistance to heat, non-flammability, low vapour pressure and high dielectric constant (WHO, 2013). PCBs were used on North Sea installations prior to the 1980s.

OSPAR CEMP has concentrated on seven PCB congeners (referred to as the International Council for the Exploration of the Sea (ICES)/Dutch/EC7) which cover the range of toxicological properties of the group. PCBs are known EDs.

CEMP benchmark concentrations are shown in Table 5-4.

Table 5‑4 OSPAR PCB benchmark concentrations

|  |  |  |  |
| --- | --- | --- | --- |
| **PCBs** | **BC/LC1 µg/kg dw** | **BAC1 µg/kg dw** | **EAC1 µg/kg dw** |
| CB28 | 0.0/0.05 | 0.22 | 1.7 |
| CB52 | 0.0/0.05 | 0.12 | 2.7 |
| CB101 | 0.0/0.05 | 0.14 | 3.0 |
| CB118 | 0.0/0.05 | 0.17 | 0.6 |
| CB138 | 0.0/0.05 | 0.15 | 7.9 |
| CB153 | 0.0/0.05 | 0.19 | 40 |
| CB180 | 0.0/0.05 | 0.10 | 12 |

Sources: 1 OSPAR, 2009a, 2009b and 2009c

Notes: BC and BAC normalised to 2.5% TOC

**Analytical Techniques**

Available analytical methods for determination of PCBs in sediments are set out in detail in OSPAR, 2015, Technical Annex 2. The analysis of PCBs in sediments generally involves extraction with organic solvents, clean-up (removal of sulphur and column fractionation), and gas chromatographic separation with an electron-capture detector (ECD) or mass spectrometric detection (OSPAR, 2015).

The recommended analysis method is the ICES/Dutch 7 GC-ECD, described in ISO 10382: 2002 Soil quality - Determination of organochlorine pesticides and polychlorinated biphenyls- Gas-chromatographic method with electron capture detection.

### Alkylphenols and Alkyl phenol ethoxylates

**Determinand Selection**

Nonylphenol, octylphenol and their APE derivates are suspected EDs which induce sex change in male fish (WHO, 2002). The main releases to the environment occur through discharge of waste water from terrestrial industrial activities. Alkylphenols are natural constituents of petroleum and are found in produced water discharges from offshore oil and gas installations.

There are no CEMP assessment criteria for alkyl phenols or APEs but limited data is available in an annex to the document Status and Trend of Marine Chemical Pollution (OSPAR, 2009d, 2009e, 2006).

**Analytical Techniques**

APEs, octylphenol, and nonylphenol are analysed by GC-MS. APEs are not included in the OSPAR 2015 guidelines. Previous studies (FUGRO/ ERT, 2011) analysed APEs using GC-MS following Accelerated Solvent Extraction (ASE) extraction using an acetone/hexane solvent mixture.

The US EPA (MedinaVera *et al*., 2001) reported a lack of certified standards and analytical methods for APEs. ASE is used for the extraction of solids (MedinaVera *et al*., 2001).

The following standard covers test methods for liquid and solid samples:

ASTM D4252 – 89 (2009) Standard Test Methods for Chemical Analysis of Alcohol Ethoxylates and Alkylphenol Ethoxylates.

The following standards exist for waters:

ASTM D7065 - 11 (2011) Standard Test Method for Determination of Nonylphenol, Bisphenol A, p-tert -Octylphenol, Nonylphenol Monoethoxylate and Nonylphenol Diethoxylate in Environmental Waters by Gas Chromatography Mass Spectrometry;

ISO 18857-2:2009 Water quality -- Determination of selected alkylphenols -- Part 2: Gas chromatographic-mass spectrometric determination of alkylphenols, their ethoxylates and bisphenol A in non-filtered samples following solid-phase extraction and derivatisation.

### Mono, di and tri-butyltins

**Determinand Selection**

Organotins is the group name for compounds of mono-, di-, tri-, (M/D/TBTs) and tetrabutyltin and triphenyltin. They are entirely manmade and as a result most organisms have developed little or no resistance/tolerance to them, (OSPAR, 2005). Organotins are the most widely used metallo-organic compounds and total world-wide use of organotin compounds increased dramatically from the 1960s to the mid-1980s. Organotin compounds are potent toxins and due to their strong toxic effects have been used in a variety of applications: agricultural pesticides, slimicides in cooling waters, timber preservatives, molluscicides and marine antifouling paints. Tributyltin (TBT) was used from the 1960s onwards as a biocide in antifouling coatings on underwater structures,

M/D/TBTs are very persistent in the environment and are known EDs. Toxicological effects due to TBT have been observed at very low concentrations and therefore assessment criteria (EAC) for TBT are very low. No formal BAC or EAC values have been set through CEMP. OSPAR, 2004 (Annex 7.2) suggests a provisional EAC range of 0.01 µg/kg dw to 0.15 µg/kg dw, based on the environmental quality standard for TBT in sediment proposed for the Water Framework Directive. There is no suggested updated value in OSPAR 2009b.

**Analytical Techniques**

Available analytical methods for determination of M/D/TBTS in sediments are set out in detail in OSPAR, 2015, Technical Annex 4.

Analysis is undertaken by GC-MS. The recommended analysis method is ISO 23161:2009 Soil quality - Determination of selected organotin compounds – Gas chromatographic method.

## Leachate analysis

The leaching rate of hydrocarbons is a function of the specific chemical’s physical/chemical characteristics, the type of cuttings pile or sediments and the hydrology in the area. Leaching of hydrocarbons and other chemical contaminants to the surrounding environment will depend on the exchange between pore water and surrounding waters, the transport and resettlement of particles, the biological activity in the bottom sediments, and oxygen availability. There is limited understanding of most of the processes that influence leaching processes in cuttings piles, and the majority of work performed on hydrocarbon leaching has been done in laboratories, not in the field (Breuer *et al.,* 1999).

Estimates of hydrocarbon leaching to the environment can be done using laboratory tests (batch-tests, column or flow-over tests), or by field monitoring (passive samplers, water and sediment analyses). Both methods have limitations, as laboratory tests often overestimate the leaching due to exaggerated exposure, while field monitoring is often limited due to poor detection limits and reduced sensitivity for relevant contaminants.

Laboratory leaching tests involve contacting one or more samples of the study material with a liquid to determine which constituents will be leached by (or dissolved into) the liquid and potentially released to the environment in a liquid phase.

Batch extraction tests typically involve mixing a sample of sediment with a specific amount of leaching solution without renewal of the leaching solution. The mixing is followed by filtration and analysis of the filtered liquid phase (the laboratory leachate). The standard test used is ISO/TS 21268-1: 2007 Soil quality – leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials – Part 1: Batch test using a liquid to solid ratio of 2l/kg dry matter.

In dynamic tests, a specific amount of leaching solution and test material are mixed and the leaching solution is periodically or continuously renewed. The mixing is performed over a relatively long time period (days to months) compared to extraction tests. ASTM method D4874 Standard method for leaching solid material in a column apparatus can be used.

There are emerging technologies (O&G UK, in preparation) for leachate analysis, most of which depend on the use of infinite sink devices or passive samplers including:

* Boxcore leach test; intact boxcores returned to test facility and infinite sink device exposed in the water column. Flux is calculated from the amount of chemical in the device, the exposure time and the surface area of the core;
* Microcosm leaching test; cuttings pile material is added to the microcosm and water column, cyclohexane as an infinite sink layer is added on top of the water to absorb contaminants. The flux is calculated as for a boxcore leach test;
* Core leaching test (*ex situ*); there are two versions, the first again uses an infinite sink device (Solid Phase Extraction disc) and the second recirculates water through a contaminant trap with the flux calculated as for a boxcore leach test;
* *In situ* benthic chamber; a benthic flux chamber containing an infinite sink device is deployed onto the cuttings pile. On recovery the infinite sink device is analysed for contaminants and the results are used along with the area of the chamber to calculate the flux;
* *In situ* emission monitoring with passive sampling devices and sediment traps; a network of passive sampling devices and/ or sediment traps is deployed vertically and/or horizontally in the vicinity of the area of interest over a selected period of time. Analysis of the samples can allow contaminant flux estimations to be made;
* Sediment/water activity ratio (*in/ex situ*); measure freely dissolved contaminants in the water column (using passive sampling devices) and bed sediment pore water to determine possible gradients and hence the direction and strength of the flux; and
* Non-exhaustive batch extractions (*ex situ*); uses infinite sink polymer to determine contaminant fractions over time to obtain the total ‘releasable’ or ‘extractable’ amount and the de-sorbtion rate. The total contaminant concentration in the original material is also needed for the calculation.

Potential technologies that have been identified to measure leaching rates from cuttings piles include *in situ* real time sensors making use of optical fibres to measure fluorescence in the water column and pore water and pelagic/demersal organism monitoring (*in situ*) whereby the contaminant burden or a biomarker response in organisms can be compared with concentrations measured in overlying water using passive samplers. This may employ the use of caged organisms at defined locations.

The adapted diffusion cell technology is a method where the contaminant mass transfer rate between two slices of silicone is measured. The medium is generally an aqueous solution. However, it may be possible to adapt this test to evaluate mass transfer from or across cuttings pile materials.

*In situ* measurements have been taken at the cuttings-water interface using a benthic lander (self-contained platforms designed to work remotely on the seabed) fitted with measurement probes (Shimmield *et. al.,* 2000) but these were focused on the determination of electrochemical conditions (pH, hydrogen sulphide concentrations, and redox potential).

In conclusion, there are presently no established methodologies that could be applied to accurately measure the rate of oil loss into the water column from cuttings piles. Analytical difficulties are compounded by the relatively complex flux mechanisms that will occur at the cuttings pile-water interface. Some available leaching tests are simple to conduct but their applicability and the interpretation of the results in terms of *in situ* leach rates is challenging. The data can nevertheless be useful for estimating worst-case impacts of cuttings pile disturbance. The emerging and potential technologies, when applied to cuttings piles, may provide information that can be related more or less directly to *in situ* hydrocarbon leaching rates and provide a more robust result to assess piles against the OSPAR leach rate criterion.

## Laboratory Accreditation and Quality Assurance

Development of and adherence to national, European or international standardised methods (where they exist) is a statutory requirement in the Water Framework and the Marine Strategy Framework Directives. The adoption of standardised methods ensures consistency between organisations and will allow for the production of data which is of an equivalent scientific quality and comparability.

Any laboratory used for physical, chemical or biological analysis must be able to confirm its accreditation through ISO/IEC 17025: 2005 General requirements for the competence of testing and calibration laboratories, or participation in other recognised schemes such as the Quality Assurance of Information for Marine Environmental Monitoring in Europe (QUASIMEME). QUASIMEME undertakes routine laboratory performance studies which provide the basis of external quality assurance for institutes that make regular chemical measurements in the marine environment.

Any laboratory involved in sediment analysis for a specific sampling programme must be able to provide details of its QA policy and information on its procedures, including:

* The use of control reference materials, either certified reference materials (CRMs) or in-house reference materials (IHRMs);
* The frequency with which blanks are incorporated into the analytical runs;
* The procedures to be followed if reagent limits are exceeded; and
* Methods for quantifying LODs etc.

All methods should be subject to regular checks and calibration, and laboratories used for analysis should participate in regular inter-comparison or proficiency exercises.

More detailed laboratory QA methods are discussed in Annexes 2, 3, 4 and 6 of OSPAR, 2015 in relation to PCBs, PAHs, M/D/TBTs and metals respectively.

# SUMmary and conclusions

These guidelines should be used for the sampling of drill cuttings piles and surrounding sediment in conjunction with existing protocols:

* OSPAR 2015 JAMP Guidelines for Monitoring Contaminants in Sediments; and
* Relevant ISO guidelines relating to sample collection and handlings.

The objective of the guidelines is to ensure consistency in the selection of determinands and the approach to sampling and analysis, to enable the results of the investigations to be compared over time, between OSPAR Contracting Parties and between different operators.

Guidelines have been provided in relation to sample collection (location, depth, sample numbers) as wells as the handling and storage of samples once they have been retrieved from the seabed.

A standard sampling suite (Table 6-1) has been proposed in line with available reference data.

Table 6‑1 Standard Laboratory Sampling Suite

| **Parameter** | **Key Reference/Method** |
| --- | --- |
| PSD | ISO 11277:2009 Soil quality: Determination of particle size distribution in mineral soil material or equivalent. |
| Moisture content | ASTM D2216-10 Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass or equivalent.ISO 111465: 1993 Soil quality – Determination of dry matter and water content on a mass basis – gravimetric method. |
| Atterberg limits | ASTM D4318 – 10e1: Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils or equivalent. |
| TOC, TN and TOM | Not covered in OSPAR, 2015.ISO 10694: 1995 Soil quality – determination of organic and total carbon after dry combustion (elementary analysis).ISO 11261: 1995 Soil quality – Determination of total nitrogen – Modified Kjeldahl method. |
| Metals: As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn, Li, Al | OSPAR 2015, Technical Annex 6.ICP-AES and ICP-MS.EN ISO 17294-2:2004 Water quality. Application of inductively coupled plasma mass spectrometry (ICP-MS) – Part 2: Determination of 62 elements. EPA method 6010C ICP-AES |
| THC | Not covered in OSPAR, 2015.Analysis of Petroleum Hydrocarbons in Environmental Media, Total Petroleum Hydrocarbon Working Group Series, Volume 1, March 1998.EPA Method 8015: Non halogenated organics using GC-FID – includes total petroleum hydrocarbons. |
| PAHs | OSPAR, 2015, Technical Annex 3.US EPA 16 by GC-MS, OSPAR 2015, Technical Annex 3ISO 18287:2006 Soil quality – Determination of polycyclic aromatic hydrocarbons (PAH) – Gas chromatographic method with mass spectrometric detection (GC-MS).EPA Method 8270C Semivolatile organic compounds by GC-MS. |
| PCBs | OSPAR, 2015, Technical Annex 2.ICES/Dutch 7GC-MS, OSPAR 2015, Technical Annex 2.ISO 10382:2002 Soil quality – Determination of organochlorine pesticides and polychlorinated biphenyls – Gas-chromatographic method with electron capture detection (includes determination of 7 PCBs). |
| APEs | Not included in OSPAR 2015.GC-MS |
| M/D/TBTs | OSPAR 2015, Technical Annex 4ISO 23161:2009 Soil quality – Determination of selected organotin compounds – Gas chromatographic method.  |
| Leachate analysis | ISO/TS 21268-1: 2007 Soil quality – leaching procedures for subsequent chemical and ecotoxicological testing of soil and soil materials – Part 1: Batch test using a liquid to solid ratio of 2l/kg dry matter*In situ* analysis may be possible in the future. |

QA procedures should be in place at all stages of the sampling and analysis to ensure reproducibility of results.

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# appendix A guidelines for sectioning cores

Samples taken from drill cuttings piles using corers can be sectioned to allow analyses of physical and chemical determinands at different depths within the pile.

Core samples should preferably be sectioned immediately after retrieval on deck and the various sub samples preserved as appropriate for specific analyses.

Sectioning is generally performed by piston-pushing the core upwards from the bottom and slicing the top parts off one by one. For longer cores, the corer itself is cut horizontally into parts with the material inside and secured for further analysis. The sectioning program should take into account any distinct layers or strata in the core, and should ensure that each stratum deemed important is sampled individually. Inclusion of several distinct strata in one sample should be avoided. If a core is homogenous in appearance it can be sectioned into standardised parts according to Figure A1.



Source: NOIA, 2003

Figure A‑1 Example of deep core sectioning



Source: NOIA, 2003

Note: Reference to Annex 3.2 in box refers to NOIA Annex 3.2. Equivalent information can be found in Section 3.2 of these guidelines

Figure A‑2 Example of shallow core sectioning

1. English only [↑](#footnote-ref-1)