European Maritime Safety Agency

INTERDISCIPLINARY PRACTICAL GUIDELINES ON OIL SPILL SAMPLING IN EUROPE

EMSA

INTERDISCIPLINARY PRACTICAL GUIDELINES ON OIL SPILL SAMPLING IN EUROPE

Developed by experts from EU/EFTA countries under the framework of EMSA's Consultative Technical Group for Marine Pollution Preparedness and Response (CTG MPPR)

EMSA

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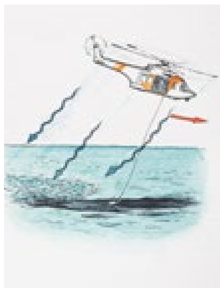

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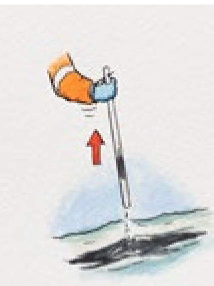

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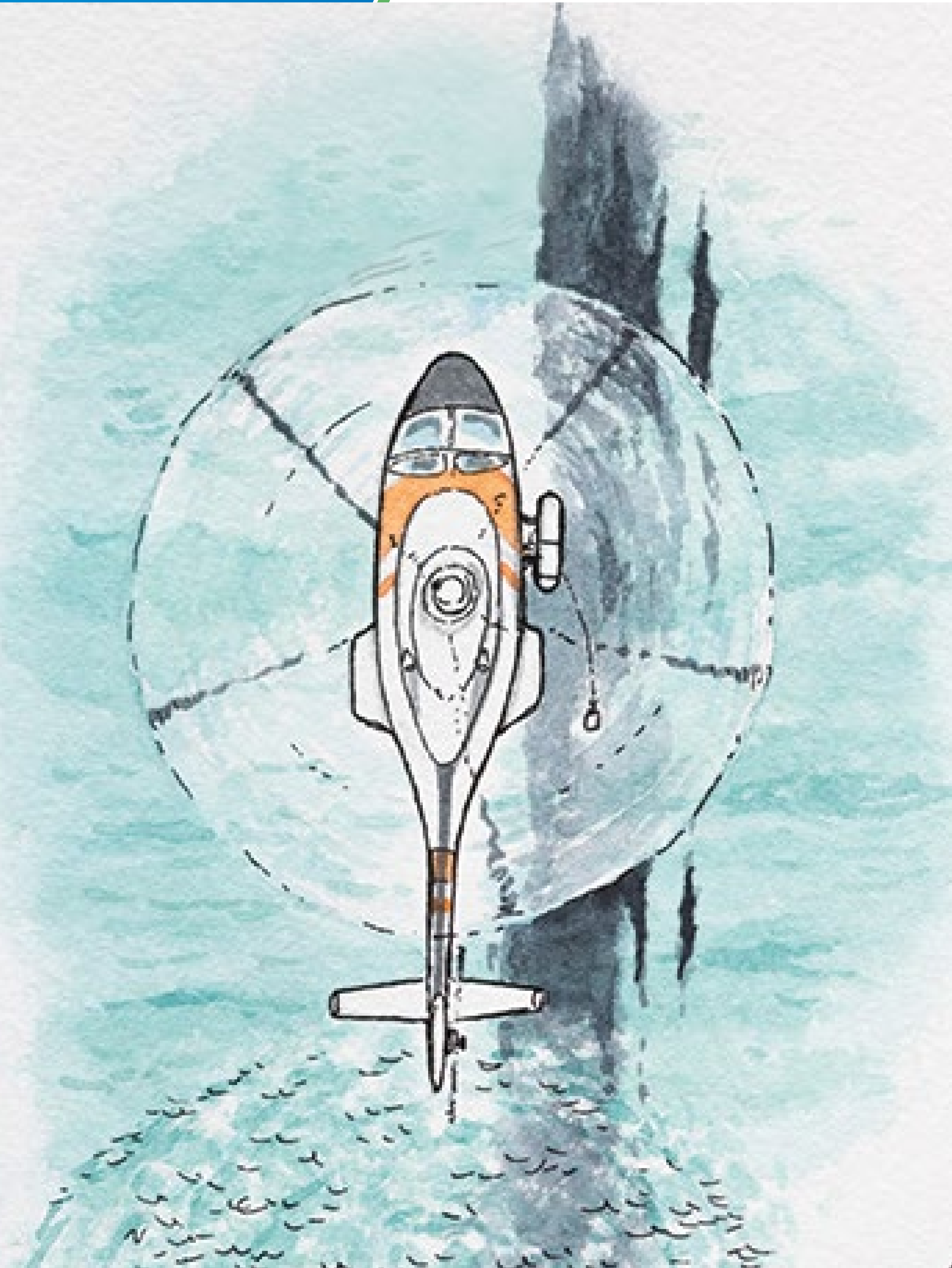


FOREWORD FROM THE EXECUTIVE DIRECTOR

I am pleased to introduce these interdisciplinary practical guidelines on oil spill sampling in Europe. These guidelines were developed by a group of experts from the EU/EFTA Member States and the European Maritime Safety Agency (EMSA) within the framework of the Consultative Technical Group for Marine Pollution Preparedness and Response (CTG MPPR) and constitute a valuable information source regarding forensic oil spill sampling born out of real-incident experiences.

When dealing with oil spills, sampling supports the preservation of evidence and, therefore needs to be done according to high quality standards. Building on existing national practices and on the work of the Bonn Agreement Oil Spill Identification Network of Experts (OSINet), these guidelines are an interdisciplinary, hands-on manual for forensic oil spill sampling, intended to provide current best sampling practice, always to be used taking into consideration the national legislation in place.

The document describes sampling techniques, equipment, documentation, and procedures, provides good practice guidance for forensic oil spill sampling, and includes relevant case studies from selected past oil spills. Six stand-alone scenarios for different sampling situations (i.e., inshore, in harbours, on beaches, from maritime units, on board ships, from helicopter), complete the guidelines by collecting all necessary information for a specific sampling situation in one template, which may be used directly in the field. Technical drawings developed specifically for these guidelines aim to facilitate the visualisation of the various sampling methods and procedures to be followed.

The relevant practical and operational experience of the countries involved in this work, combined with EMSA's support, resulted in these Interdisciplinary practical guidelines on oil spill sampling in Europe, which, together with the six situational sampling scenarios, we hope may further support the authorities involved in oil spill sampling across Europe, as well as those wishing to develop their sampling capacity.

This document demonstrates once again the immense value that comes from such cooperation in maintaining and sharing relevant expertise and I thank all the experts involved in its development.

Maja Markovčić Kostelac
Executive Director

For most of history, man has had to fight nature to survive; in this century he is beginning to realize that, in order to survive, he must protect it.

Jacques-Yves Cousteau

ACKNOWLEDGEMENTS

The European Maritime Safety Agency (EMSA) on behalf of the Consultative Technical Group for Marine Pollution Preparedness and Response (CTG MPPR) would like to thank the members of the Working Group for their efforts and contributions in developing this document:

- Gracia Alburquerque, Spanish Maritime Safety and Rescue Agency (SASEMAR)
- Josep Bayona, Institute of Environmental Assessment and Water Research (IDAEA)-CSIC Lab, Spain
- Hilde Dolva, Norwegian Coastal Administration (NCA)
- Carmen Domínguez, IDAEA-CSIC Lab, Spain
- Hernán Javier del Frade de Blas, D.G.M.M., Ministerio de Transportes, Movilidad y Agenda Urbana, Spain
- Cornelis Kooistra, Rijkswaterstaat CIV RWS Lab, the Netherlands
- Uta R. Kraus, Federal Maritime & Hydrographic Agency (BSH), Germany
- Marijke Neyts, KBIN-OD-Nature Lab, Belgium

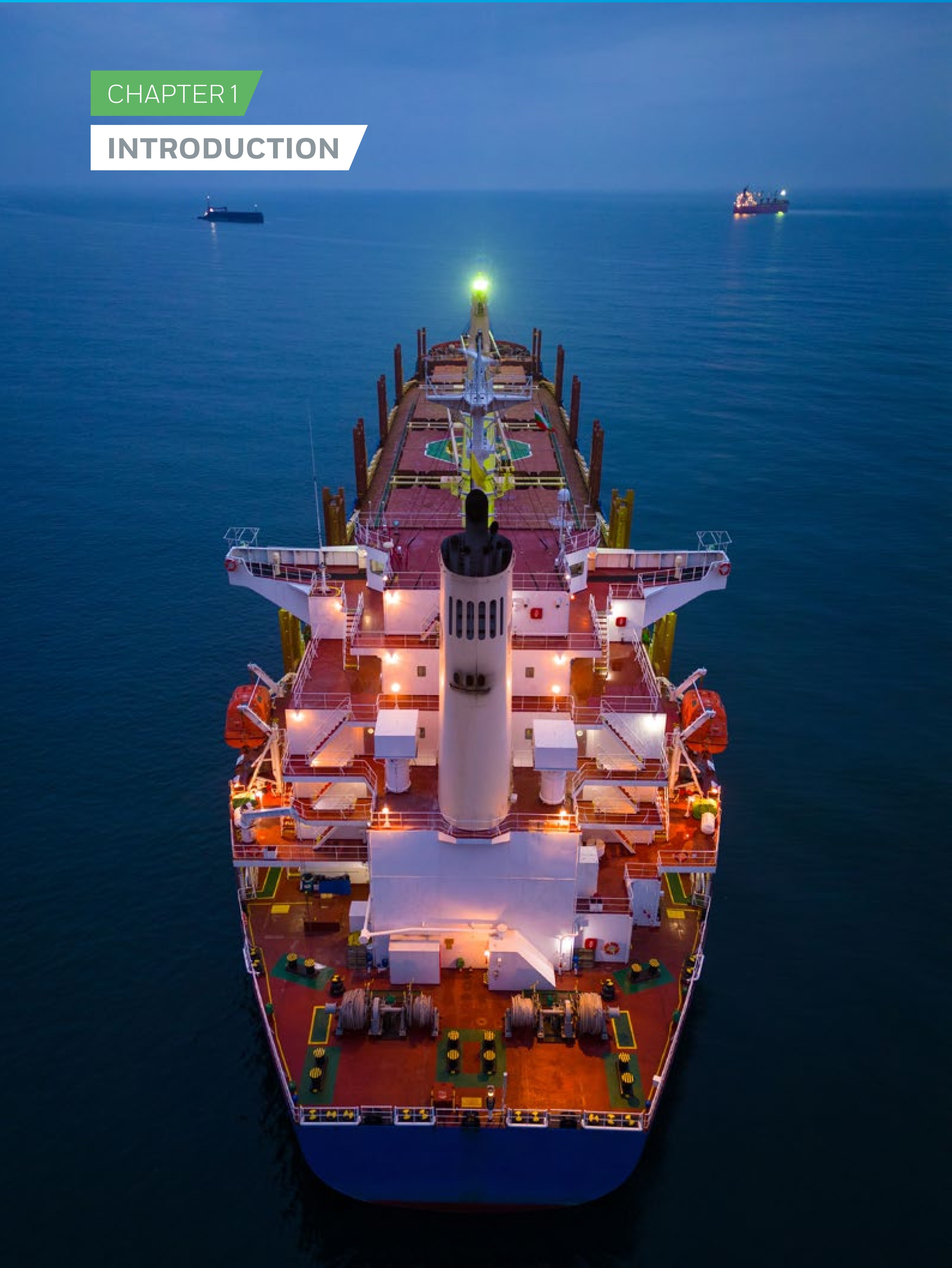
DISCLAIMER

These guidelines were developed by a Working Group comprised of experts from EU and EFTA countries within the framework of the EMSA Consultative Technical Group for Marine Pollution Preparedness and Response (CTG MPPR). The document is intended to provide practical guidance and information based on existing practices, documentation and real incident experience in Europe and is a non-binding document. Nothing in this guidance should be construed as generating mandatory or legal requirements on any of the involved parties

Under no circumstance do these guidelines replace individual, legal or technical advice rendered considering the individual circumstances of each case and situation, nor do they replace national requirements, regulations, recommendations and practices applicable in each country. Under no circumstances shall EMSA or any of the other contributors be liable for any loss, damage, liability or expense incurred or suffered that is claimed to have resulted from the interpretation and the use of the information presented in this guidance document.

CHAPTER 1

INTRODUCTION



In recent years, maritime oil pollution in Europe is declining due to international regulations and regular maritime surveillance. However, the probability of an accidental oil-spill incident in European seas remains high due to both an increase in seaborne traffic [1] and the increase and expansion of offshore oil and gas activities resulting in a rising number of oil and gas installations [2]. Globally, the number of large spills (> 700 tonnes) has significantly declined over the last few decades with currently 1.8 of these spills occurring on average per year between 2010-2019 [3].

However, the location of large spills is not predictable. Prominent examples from the last two decades are the accidents of the Tricolor (2002) and the Flinterstar (2015) in the North Sea and of the Prestige (2002) off the Spanish North Atlantic Coast. In case of large spills, international cooperation not only is important in direct response activities but is extremely helpful for characterizing waterborne oils and the identification of possible sources. Since large-scale spills often reach across national borders, harmonised oil sampling protocols and concerted analysis with comparable results can create legally defensible cases to approach the issue of compensation claims, noting also that costs for sampling and sample analysis may also form part of the claimed costs [4].

Furthermore, such procedures are helpful to consistently catalogue and record not only accidental spills but also, illegal discharges. With this approach, (smaller) mystery spills where initially no detectable source is obvious, can be tracked and recorded in a way that permits authorities to trace the potential source of the spill over long distances. Furthermore, international cooperation, like the oil spill identification network of experts within the Bonn-Agreement (OSINet), can not only facilitate the creation of such harmonised protocols but can help with the implementation of their day-to-day use. This allows an even deeper level of coordination. For example, OSINet utilises an online analytical tool and database of forensic oil fingerprints that allows the real-time comparisons of oils from around the world through web-based computerised oil spill analysis (COSIWeb).

1.1 PURPOSE

Today, enhanced technology and improved ship designs make seaborne ship traffic safer. On the other hand, the increase in both traffic and offshore activities provides new challenges in risk management and response preparedness. In this aspect, EMSA aims to help facilitate coordinated preparedness and response activities at EU level and supports diverse maritime users across Europe with the provision of oil and chemical spill response services that can be activated by these users to complement existing pollution response capacities. EMSA's CleanSeaNet European satellite-based oil spill monitoring and vessel detection service can provide additional support with the detection of possible oil spills on the sea surface and the identification of potential polluters. As the primary responsibility to react to an incident always remains with the affected Member State, additional ways for coordinated action become more important.

Oil spills in the environment are liable to prosecution. In large incidents like ship disasters at sea, the consequences for the environment can be massive, resulting in high costs for cleaning, environmental remediation and compensation. Consequently, substantial claims against the perpetrator can arise. However, incontestable evidence that links the discharge to a polluter is needed for prosecution.

In such cases, oil spill forensic analysis can provide measures to prove the origin of a pollution event by connecting the oil spill with its source (e.g. a certain ship). While spill identification by chemical analysis is by no means the only tool available to law enforcement, it is an important, in fact sometimes the most important, instrument providing evidence in criminal proceedings. As such, it is crucial to carry it out in the most robust, standardised and reliable way possible. However, without oil samples of good quality, analyses cannot be expected to yield defensible results.

KEY MESSAGE

FORENSIC OIL SPILL ANALYSIS REQUIRES SAMPLING ACCORDING TO HIGH QUALITY STANDARDS.

It is therefore vital to recognise oil sampling as preservation of evidence to prove environmental crimes and it has to be handled accordingly with care and with the proper procedures. Sample takers should be regularly trained in the required practical skills and need to keep an up-to-date knowledge of oil spill sampling methodology.

Accordingly, the purpose of this document is to provide best practice guidance on the principles of oil spills forensics in an interdisciplinary fashion and on the basis of real incident experience. It should provide Member States with a resource to enhance their knowledge regarding oil spill sampling. Furthermore, the document will serve as the main reference document for any EMSA workshops for exchange of experience on oil spill sampling held under the CTG MPPR work programme.

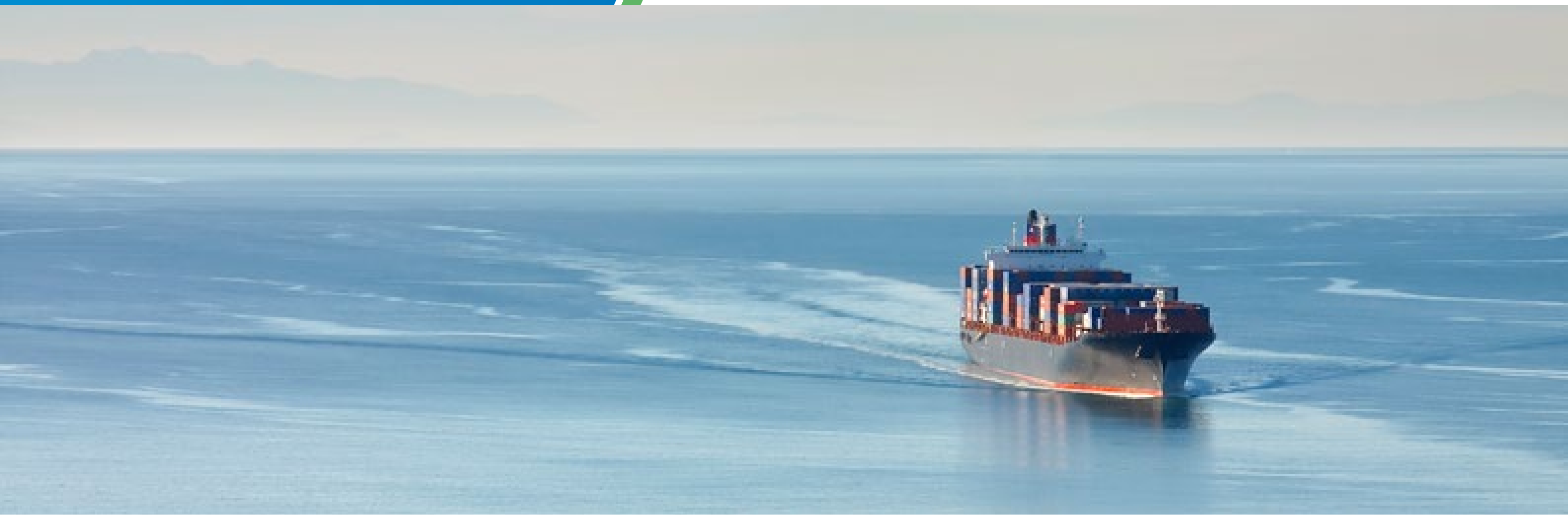
For direct use in a certain sampling situation, this document provides **six oil spill sampling scenarios** as unique stand-alone chapters containing in a concise manner all the crucial information on a specific sampling situation (e.g. inshore, harbours, rocky shores etc.).

TIP

PROVIDE REGULAR PRACTICAL TRAINING FOR SAMPLE TAKERS

1.2 SCOPE

These guidelines describe the practical steps of oil sampling for oil spill forensic analysis when dealing with accidental or deliberate spills of petroleum oils and petroleum oil products in aquatic systems (limnic and marine waters). Based on real incident experience and existing oil sampling practices, it gives practical guidance regarding sampling strategies and provides common, harmonised methods for appointed sampling personnel (e.g. responders; police; army; investigators) dealing with oil spills in the field. Such information may prove useful for countries that have sampling procedures in place and may want to update these, as well as for countries that may wish to (further) develop their sampling capacity.



This document is based on the experience of the countries that have contributed to it and in particular on the work of OSINet. The guidelines focus on forensic oil sampling; techniques relating to oil spill response (including aspects of dispersant application[1] and effectiveness) and oil recovery are outside of the scope of this document. The same applies to analytical fingerprinting of oil spills, which is only referenced in this document where it has direct implications for sampling procedures and sampling strategies. Furthermore, claim and compensation management are not within the scope of these guidelines. In regard of these topics, other guidance documents are available (e.g., EN 15522, [2, 5-13]. Sampling for long-term environmental monitoring, as well as sampling related to sulphur content of fuels [14] are also, outside the scope of this document, along with sampling of materials other than petroleum oil (products) (see chapter 12.4).

These practical guidelines are intended to provide best practice guidance and are non-binding. Nothing in this guidance document should be construed as generating mandatory or legal requirements on any of the involved parties.

1.3 BACKGROUND AND CONNECTION WITH OTHER SAMPLING GUIDANCE

The beginnings of the application of oil spill forensics reach back to the 1970’s and 1980’s when scientists started to use analytical methods from petrochemical exploration works on oil spills. Their efforts led to ever more precise fingerprinting methods. However, techniques used for both oil sampling and analysis were not internationally harmonised and results from different laboratories were rarely comparable.

Based on the NORDTEST method (NORDTEST 1991), experts of OSINet developed a common methodology for oil identification work which was first published in 2006 as a two-part technical report by the European Committee of Standardisation (CEN). CEN/Tr 15522-Part 1 [15] described sampling techniques and the handling of oil samples prior to their arrival at the forensic laboratory, while CEN/Tr 15522-Part 2 [16] covered laboratory procedures of oil spill identification methodology, analytical techniques, data processing, data treatment, and interpretation and reporting of results. In 2023, CEN/Tr 15522 was converted into the two-part EN standard EN 15522 [17, 18].

[1] A short introduction to a field test procedure for dispersant effectiveness is included in Appendix 4. For further information on this topic, see reading list at the end of the document.

In 2016, the EMSA CTG MPPR established a working group of experts from EU/EFTA countries to develop a course programme for oil sampling based on the exchange of experience 'from Member States for Member States' and to disseminate knowledge of environmental oil spill forensics by the compilation of practical guidelines on oil spill sampling. A 'pilot' course on oil spill sampling was developed and held in 2018, whilst the work drafting the guidelines begun in 2019.

These guidelines are explicitly formulated as an addition to EN 15522-1, not as a repetition. For this reason, oil sample analysis and oil identification have been kept outside the scope of this guidance document, as they are covered in detail in EN15522-2.

This document has been prepared on the basis of current knowledge and experience from specialized institutions within the Member States. It is largely based on the expertise of oil spill analysts as well as national trainers of oil spill sampling personnel and from experts in OSINet. For further research into the topic, additional information can also be found in guidance documents available from different international and national institutions [2, 6, 13, 19-22].

1.4 INTRODUCTION TO OIL SAMPLING FOR OIL SPILL FORENSIC ANALYSIS

In the context of oil spill forensics, collecting oil samples represents the preservation of evidence. This alone indicates the necessity to use best practice and sophisticated techniques.

KEY MESSAGE

COLLECTING FORENSIC OIL SAMPLES REPRESENTS THE PRESERVATION OF EVIDENCE!

It is important to always initiate sampling before clean-up measures are started. Not only can sample analysis give important support information for the planning of response and cleanup work [23], but for oil identification it is crucial to secure unbiased and uncontaminated oil samples. All spills encountered and all potential sources of spills should be sampled. It is important to take samples from both spill and source even on occasions where it seems quite clear from where the spill originates[2]. Background locations nearby, meaning sites where oil contamination is not apparent (see chapter 8.1) and that share environmental characteristics such as temperature and salinity with the contaminated site, should also be sampled to determine background conditions of the contaminated site (background samples / field blanks).

It is important to keep in mind that each oil spill incident is unique, and response actions are adapted to each situation in accordance with national regulations.

KEY MESSAGE

ALWAYS TAKE SAMPLES BEFORE OIL SPILL CLEANING ACTIVITIES START AND ACCOMPANY CLEAN-UP WITH A FITTING SAMPLING PROGRAM!

[2] Differences in national approaches exist – some countries do not use oil samples in cases of illegal discharges but base prosecution on CleanSeaNet alerts, aerial surveillance and other evidence in which case this issue is irrelevant; in other countries prosecution is not possible without samples from spill and sources in which case this sampling strategy is a basic requirement. In the procedural description, this document assumes that sample taking is applied in the national legal context.

CHAPTER 2

SAMPLE COORDINATORS

Sample coordination and sample taking are tasks and roles that should be defined in the National Contingency Plan to assure correct and sufficient sample collection.

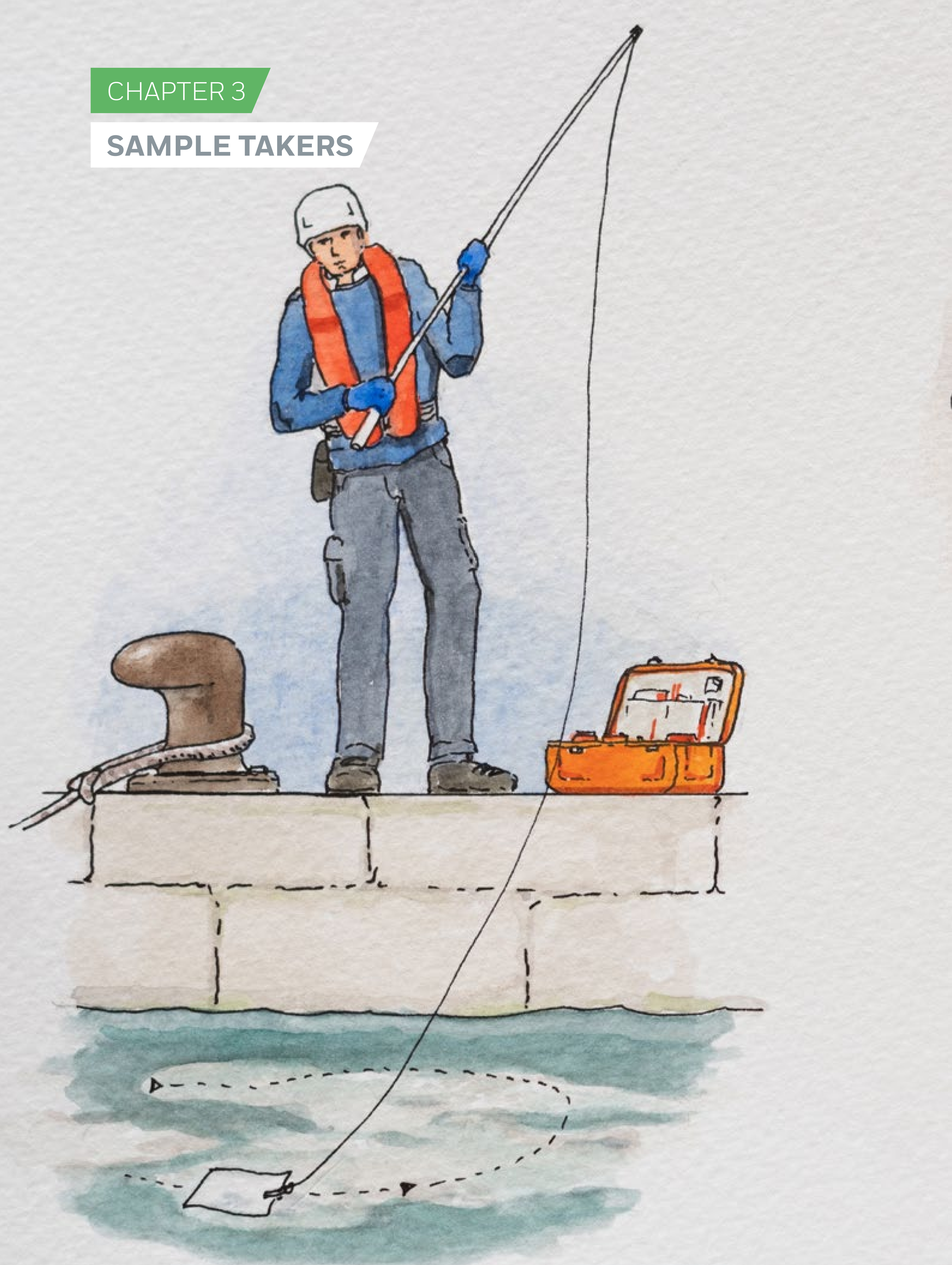
The sample coordinator is responsible for establishing a sampling plan, considering the pollution, the (known or possible) source(s) and the available mobilization of units and sample takers.

Tasks of the sample coordinator can include:

- Designing a sampling plan (if suitable including a map with sampling points) for the specific incident, specifying the location and amounts of samples to be taken:
  - o from the different pollution spots considering the duration of the spill;
  - o from the known source or possible sources;
  - o background samples/field blanks;
- Coordinating the sample takers;
- Providing the sampling kits and when needed, replacing the sampling equipment;
- Coordinating the delivery of the samples to the appropriate laboratories for the required analysis/storage;
- Providing information about appropriate storage condition of samples during storage/before delivery;
- Managing the relationship with the laboratory and the coordination with other organisations involved;
- Providing advice for sample takers about relevant safety measures (see chapter 5).

CHAPTER 3

SAMPLE TAKERS



Oil samples should always be taken exclusively by well-trained and experienced persons. In many countries, court-approved samples can only be taken by specialized, certified sample takers. However, in other countries, basically everybody can carry out the oil sampling while the emphasis applied to the evidence stemming from such a sample is determined by the courts. Since sampling is the crucial key activity for the forensic analysis of oil spills, it should be ensured that sample takers are regularly trained and up to date with regulation changes and informed about new sampling equipment and its proper use. Furthermore, sample takers must be carefully trained in proper sample documentation.

Mistakes made during sampling are not correctable at a later stage, thus the correct sampling is the basic prerequisite for all legal proceedings.

Tasks of the sample taker can include:

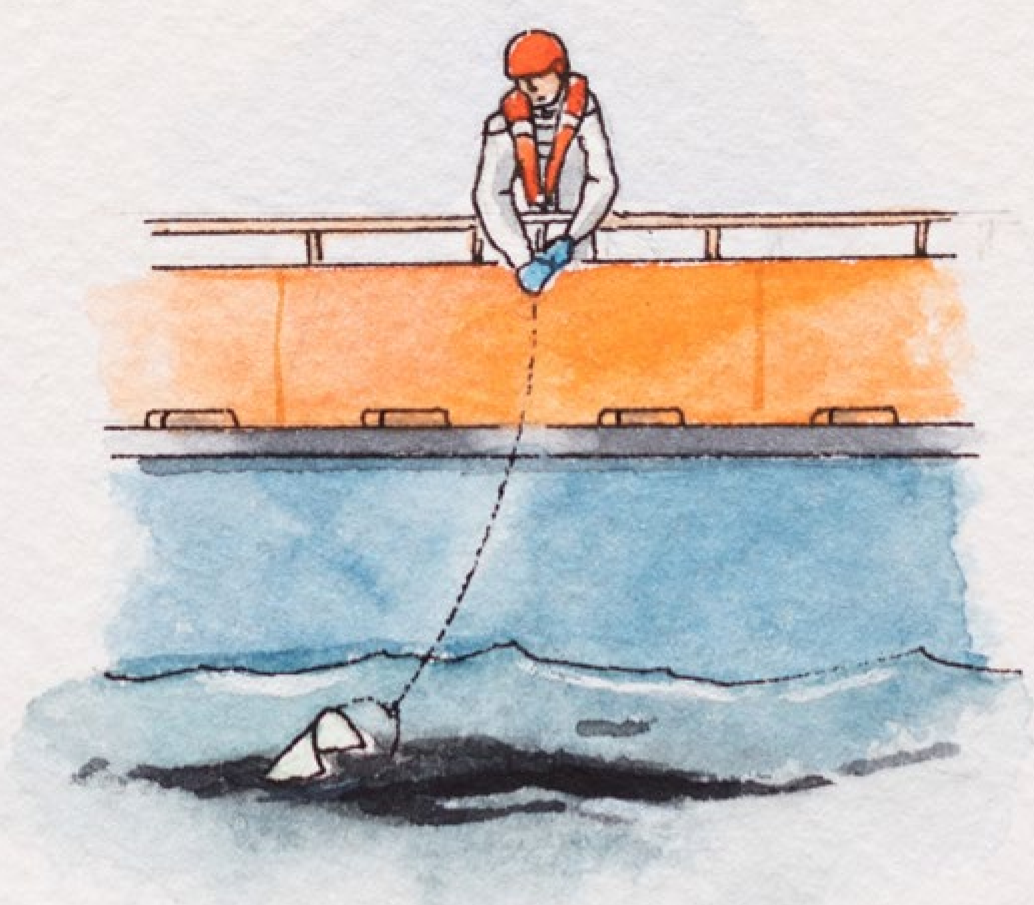
1. Taking the samples in accordance with the sampling plan;
2. Documenting and properly labelling and sealing the sample(s);
3. Communication with the laboratory;
4. Preparing the sample for transport and/or assuring correct storage during storage time before transport.

KEY MESSAGE

OIL SAMPLES SHOULD BE TAKEN EXCLUSIVELY BY WELL-TRAINED AND EXPERIENCED PERSONS.

CHAPTER 4

GOOD SAMPLING PRACTICE



The most important aspects of good sampling practice are:

- Working according to standardised methods;
- Utilising the proper sampling equipment (e.g. exclusively using suitable sampling containers);
- Detailed, meaningful documentation (including standardised sample description forms as well as maps and/or pictures, video) which can be used even years after the incident happened;
- Using a checklist in the field to ensure that no procedural step is forgotten or missed (Table 1);
- Making sure to avoid cross contamination (e.g. by always using single-use sampling material in the field, such as disposable nitrile gloves, with each sample requiring the use of new gloves);
- Managing the relationship with the laboratory and the coordination with other organisations involved.

Table 1: Example of a sampling checklist for field work

AREA	CHECK	COMMENTS (OPTIONAL)
All relevant spots sampled?[3]	-	-
Background samples/field blanks taken?	-	-
Peculiarities? Something noteworthy? Note it down!	-	-
All samples properly labelled?	-	-
Sample Submission Form/Request for Analysis Form completed?	-	-
Chain of custody form completed?	-	-
Do the sample IDs match on all documents (e.g. analysis request form, chain of custody form, sample label on the sample)?	-	-
Are samples packed shatter-proof and cooled (4 ± 3°C) in a way (e.g. by using cooling pads) to ensure that they will remain cooled during the entire transportation time?	-	-
Is all necessary documentation packed with the samples for transport (if samples are brought directly from the field to the lab)?	-	-
Anything missing from the sampling kit? Note it down for later restocking.	-	-

[3] See also chapter 6

CHAPTER 5

HEALTH HAZARDS AND SAFETY ISSUES RELATED TO OIL SAMPLING

KEY MESSAGE

RISKS RELATED TO SAMPLING MUST ALWAYS BE PROPERLY AND CONTINUOUSLY ASSESSED.

Sample takers should always keep in mind that essentially all petroleum oils and oil products have the potential to cause harm to humans, animals and the environment, either on their own or through interaction with other factors. Oils, whether in the crude form or as refined products, have hazardous properties that may include:

- Flammability
- Explosive and toxic vapours
- Toxicity
- Displacement of oxygen, especially in closed spaces
- Changes in the integrity of surfaces as the oil can cause a slip hazard

For that reason, it is imperative to use adequate personal protective equipment (PPE) and in case of flammability to use gas detectors, when preparing to sample. In the case of small marine spills, it might be sufficient to wear nitrile gloves. In other situations, especially when the sampling is part of a large-scale oil response operation, when it takes place in narrow, small or enclosed spaces or when clean verified samples of oil from the potential sources are needed, it may be necessary to:

- Use flammable gas detectors, and other gas detectors and monitoring devices. The most common personal detector device sold today in the oil industry is a 4-gas unit that measures flammability (LEL), oxygen (O_2), carbon monoxide (CO) and hydrogen sulphide (H_2S). 5-gas detectors are increasingly being used, incorporating a PID photoionization sensor into 4-gas detectors, also allowing the detection of volatile organic compounds (VOCs) present in oils (such as benzene, toluene, xylene…)[4] and some inorganic gases, which might be produced under some environmental circumstances.

- Wear appropriate PPE, as a minimum: head protection, protective suits, respiratory protection, nitrile gloves and protective goggles and safety boots/shoes. The use of full protective gear might influence the practicalities of the oil sampling, especially in hot climates.

- Sampling on board ships is a highly technical and dangerous activity that should be carried out by law enforcement personnel with experience of working on ships, such as maritime police, customs officers, marine surveyors, or by salvors. They should always be accompanied by the vessel's crew.

REMEMBER TO ALWAYS CONSIDER:

- Risk for fire and/or explosion (do not perform sampling if there is a risk);
- Oxygen deprivation or asphyxia;
- Toxic fumes (use suitable respiratory protection);
- Slippery conditions caused by oil in contaminated areas.

Furthermore, reference should always be made to relevant current regulations concerning the site of the sampling, Safety Material Data Sheets and other applicable specific health and safety instructions.

For more information regarding health and safety considerations during oil spill response operations, see the "EU/EFTA States practical guidelines on health and safety of oil spill responders" developed under the CTG MPPR.

KEY MESSAGE

DURING SAMPLING, HEALTH AND SAFETY OF THE SAMPLE TAKER MUST ALWAYS HAVE PRIORITY AND SAMPLING MUST BE CARRIED OUT IN A PRUDENT HEALTH AND SAFETY MANNER.

[4] Please note that PID sensors do not detect all VOCs (e.g. ethane is not detected)

CHAPTER 6

BASIC PRACTICAL PRINCIPLES OF FORENSIC OIL SAMPLING

This chapter brings together the important basic principles of forensic oil spill sampling. Single aspects will be addressed in more detail throughout the document where necessary.

- ✓ Wear appropriate PPE when taking samples.
- ✓ Always wear nitrile gloves while sampling. Use a pair of new, clean gloves for each sample.
- ✓ Take samples in an appropriate timescale, meaning as quickly as possible after the spill occurs and always before oil recovery or cleaning procedures begin.
- ✓ Use appropriate sampling equipment and appropriate sample containers (e.g. glass bottles with an inert lining in the lids).
- ✓ Treat oil sampling as preservation of evidence – which it is.
- ✓ Choose representative sampling spots within the oil spill. Areas in the centre of the spill and/or areas where the spill is the thickest are best places to take meaningful samples. Don't sample the fringes of an oil spill.
- ✓ Take a sufficient number of samples and appropriate amounts / volume, see chapter 7.4). Large oil spills should be sampled at different spots. This is even more important if the spill shows different appearances in different areas (change of colour, change of texture/thickness). Even from smaller spills it is advisable to take at least two samples[5].
- ✓ When taking samples from possible sources, take them from different compartments (e.g. tanks on ships, decks, cofferdams) and sample from all compartments[6].
- ✓ Take samples from all possible sources, even if the connection between the spill and one source seems unambiguous in the field[7].

[5] Please note that this requirement refers to taking two samples from one (small) spill at two different locations of the spill. It does not describe the number of sample replicates to be taken per sampling point. These requirements differ between Member States (or sometimes even between laboratories in the same country) and sampling has to be carried out accordingly. However, please be aware that if retained samples are taken from a spill to be stored in their original condition for later comparison that, without stabilisation, the stored sample will degrade further while in storage (the degree of the so-called secondary weathering depends on the storage conditions, the oil amount and the oil type).

[6] Please note that if a large number of samples are taken, it does not necessarily result to every sample being analysed and subsequent high laboratory costs. It might be possible to determine a result with only a fraction of the acquired samples. However, often in oil spill sampling, there is no chance to go back, no second possibility to acquire more samples at a later time. If sufficient samples are taken in the beginning and if they are stabilised at the laboratory, they can be used weeks or months later if they are needed. In relation to the disposal of un-used samples, an arrangement regarding waste-management has to be put in place with the clear indication of who is responsible for the waste disposal of unused sample material (sample takers, the laboratory or a third party) at the appropriate time.

[7] Reporting the water and ambient temperature is important to account for weathering effects of the oil from both the spill and the different sources. Depending on the situation, in special cases, samples from a source might be more weathered than the spill (e.g. if a spill occurs in cold/icy conditions).

- ✓ Never accept samples from third parties (e.g. from crew on-board ship), always sample yourself!
- ✓ If there is a continuous oil spill over a longer time span, samples should be taken in regular intervals throughout the spill duration;
- ✓ If there is suspicion that the oil spill is mixed with other substances (e.g. another type of oil or some cleaning agent / dispersant), a pure sample of the second substance should also be taken if possible.
- ✓ Collect background samples/field blanks, meaning samples from the vicinity of the oil spill but unaffected by it. Thus, background contamination can be identified (e.g. pre-existing oil in a harbour);
- ✓ Avoid the contamination of the samples by algae, sand, debris, wood etc. These should not be included in the samples;
- ✓ Record the date and time and any relevant accompanying environmental parameters (e.g. temperature of water and air) of the sampling situation .
- ✓ Avoid contamination of sampling bottles by not touching the inside of the bottles.
- ✓ Touch ethylene tetrafluoroethylene (ETFE) nets or steel/PTFE (polytetrafluoroethylene) spatulas only with gloved hands and handle them as little as possible.
- ✓ To prevent overflowing of the oil, do not fill sampling containers to their maximum capacity, fill sampling bottles to not more than 75-90%.
- ✓ After sampling, all sample containers have to be cleaned on the outside, if contaminated by oil residues.
- ✓ Immediately after sampling (and cleaning the outside of the sample containers, if necessary), label sample containers to avoid confusion of samples and seal them with security labels to begin the chain of custody procedure.
- ✓ Fill in the sampling documentation completely and legibly, including a precise description of the sampling location (e.g. GPS coordinates; unambiguous description; identification of sampling points in a tank plan or piping diagram of a ship).
- ✓ If possible and appropriate, take pictures/videos of the sampling site and/or draw a sketch.
- ✓ Directly after sampling, samples should be cooled (4 ± 3)°C and kept in the dark; samples must not be frozen during transport or storage.
- ✓ Samples should be transferred to the analysing laboratory immediately after sampling.
- ✓ Samples, as well as all sampling equipment, must at all times be stored and used in a way that prevents all possibilities of tampering, manipulation and/or contamination.

- Sample containers (e.g. glass bottles with inert lining in the lids) that have been used or that contain sampling material must not be stored or transported in direct proximity with unused, clean sampling equipment.
- Sample containers (e.g. glass bottles with inert lining in the lids) must not be reused. The use of single-use sampling material in the field is generally regarded best practice (gloves, bottles etc.).
- Reusable sampling equipment (e.g. steel or PTFE spatulas) has to be cleaned carefully in the laboratory before reuse.
- Take samples always from the least contaminated to the most highly contaminated location to avoid cross contamination.

CHAPTER 7

SAMPLING EQUIPMENT AND MAINTENANCE

7.1 SAMPLING KIT AND TOOLS

The contents of a generic sampling kit are described in Table 2[8]. The list is not exhaustive and is meant as a universal guide. It may be necessary to adjust this list for special sampling requirements (e.g. special PPE for certain sampling environments, enhanced cooling equipment etc.).

While there is not one single suitable sampling kit for all purposes, and equipment might differ from application to application, there are general aspects to keep in mind and mistakes to be avoided in oil sampling. For example, it is strongly recommended to not use typical sampling equipment designed for the sampling of surface water, like typical bottle holders, since such devices are often made of plastic material that cannot be cleaned properly either in the field or at the lab. Since it is imperative to avoid cross-contamination by the transfer of oil residue from one oil sample (or one sampling spot) to another, the use of such equipment must be discouraged (see chapter 7.2).

- Generally, only inert materials like ETFE, stainless steel or glass should come into contact with the oil sample to avoid sample contamination by the sampling equipment.
- If equipment comes into contact with oil, it has to be discarded afterwards (single use articles) or not used again before proper cleaning in the laboratory (e.g. steel spatulas) because multiple uses will inevitably lead to cross contamination.

KEY MESSAGE

FOR SAMPLING OF OIL, AVOID TYPICAL EQUIPMENT DESIGNED FOR THE SAMPLING OF SURFACE WATER SINCE THESE GENERALLY INCLUDE PLASTIC PARTS WHICH CANNOT BE CLEANED SATISFACTORILY.

7.2 CLEANING OF SAMPLING EQUIPMENT DURING FIELD CAMPAIGNS

Avoiding cross contamination by already used and therefore oiled equipment is absolutely essential for the success of forensic analysis of oil spills. If remaining oil from a previous sample contaminates following samples, the results of the oil's fingerprint analysis will lead to inconclusive or false matches.

Therefore, whenever possible, the use of single-use-material in the field is regarded best practice. Gloves, bottles, spatulas etc. should only be used once and then discarded or, alternatively in case of re-usable material, cleaned thoroughly at the laboratory using organic solvents. Such cleaning is not possible in the field, especially due to the toxicity of the solvents used, which means they cannot be handled safely outside the lab.

[8] Based on the experience of OSINet

Typical equipment for surface water sampling, which is typically made of plastic, cannot be cleaned with organic solvents. Furthermore, most sampling devices are of such design that they will inevitably come into contact with the oil during the sampling process, resulting in an equally oiled bottle as well as an oiled sampling device which cannot be properly cleaned in the field before further sampling actions.

KEY MESSAGE

USE SINGLE-USE-MATERIALS FOR OIL SAMPLING.

7.3 CLEANING AND STORAGE IN-BETWEEN FIELD CAMPAIGNS

Reusable sampling equipment which has come into contact with oil during the sampling has to be cleaned very carefully in the laboratory before further use. Petroleum oils are often not completely removable by use of typical "household detergents" like dishwashing liquid. For such cleaning procedures, organic solvents have to be applied. Such solvents destroy plastic surfaces. For this reason, only inert equipment (made of glass, steel or PTFE) should be reused. Sampling containers, such as sampling bottles are not to be reused to credibly eliminate the possibility of cross contamination.

Where it is deemed necessary, clean, ready-to-use equipment can be wrapped and sealed with a paper seal. This prevents the mix-up with uncleaned equipment.

In some cases, oil sampling equipment might be held in storage for long periods of time before being used. If there is a doubt that equipment might be held in storage for too long periods in which the equipment might lose some crucial characteristics (for example the lining of the lids of the glass bottles might get loose or poriferous; security seals might not be adhesive enough after a while etc.), such paper seals could be complemented with an expiration date.

7.4 REQUIRED SAMPLE QUANTITIES

For the forensic analysis of oil spills, very small amounts of oil are sufficient (less than 1 mL). However, sample takers should always aim for sampling a larger amount of oil, since the effects of secondary weathering processes in the sample will be reduced when larger amounts of oil are sampled. Therefore, better analytical results will be obtained. On the other hand, no amount of oil should be deemed to be too small. In case of sampling with the ETFE net, even if there is no visible oil on the net, there may still be a sufficient amount of oil on the net for analysis.

TIP

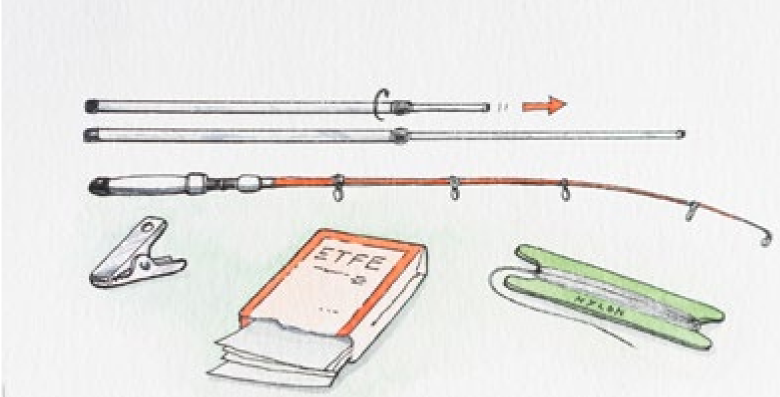
NO AMOUNT OF OIL SHOULD BE DEEMED TO BE TOO SMALL FOR ANALYSIS. IN CASE OF SAMPLING WITH THE ETFE NET, EVEN IF THERE IS NO VISIBLE OIL ON THE NET, THERE MAY STILL BE A SUFFICIENT AMOUNT OF OIL ON THE NET FOR ANALYSIS.

In contrast to this, when analysing physical parameters (such as viscosity, pour point, flash point, density etc.), larger amounts of oil (hundreds of millilitres up to litres) will be needed. Even with sophisticated oil sampling techniques, it is often not possible to collect enough spilled oil for such analyses, for example in the case of spills of light fuels, where sampling is carried out with the ETFE net, it might not be possible to extract a large enough amount of oil. However, this may be possible for large spills or under certain environmental conditions.

Often, oil forensic laboratories do not run tests regarding physical oil parameters, which means a second laboratory would be needed. In such cases, it should be clarified when designing the sample plan how much sample material is needed by which laboratory using which sampling containers, to avoid ending up with insufficient amounts or cross-delivery of samples. This can result in analyses being run with oils from different sampling dates or places, or even that oil samples are combined to raise the required amount of oil, which is not compatible with meaningful and court-proof procedures.

TIP

ALTHOUGH SMALL AMOUNTS OF OIL ARE SUFFICIENT FOR OIL SPILL FORENSIC ANALYSIS, TAKE AS MUCH OIL SAMPLE MATERIAL AS IS POSSIBLE BY FILLING UP SAMPLING BOTTLES. HOWEVER, NEVER FILL BOTTLES TO MORE THAN 75-90% (NEVER TO THE BRIM).



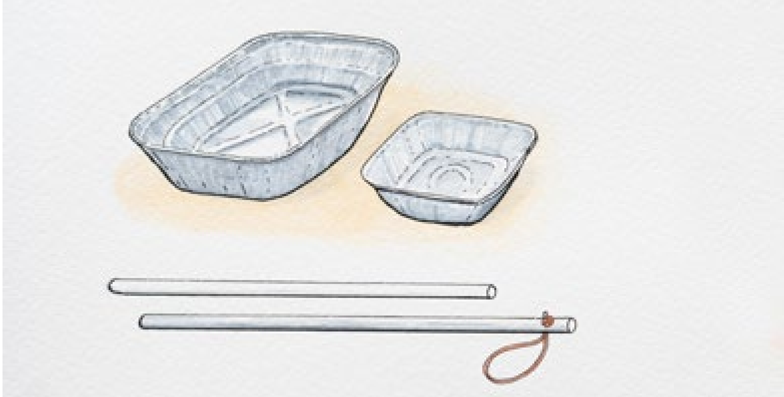


Table 2: Recommnded Equipment for a generic Sampling Kit

GENERAL
Scissors
Cutter
Ruler/tape measure
Flashlight
Several meters of thin rope (e.g. for lowering bucket into waters)
GPS device for geo-referencing the sampling site
Thermometer (for taking surface water and ambient temperature)
Oil test paper (Figure 1)[9]

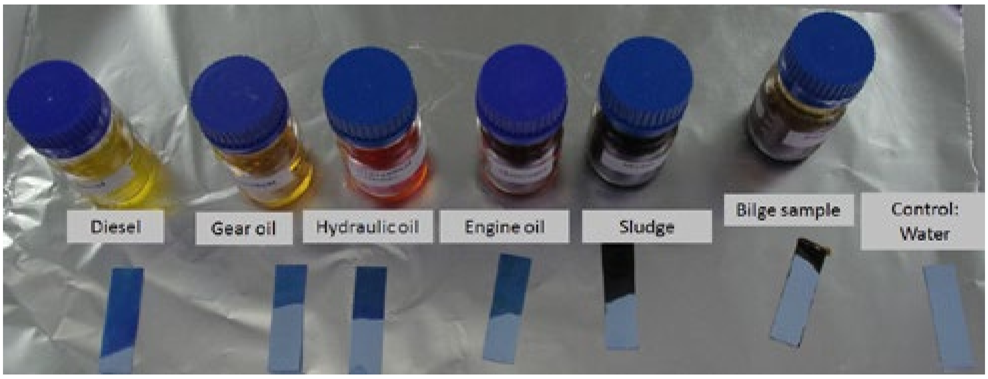


Figure 1: Oil test paper used on different oil types and on water (far right side). The presence of hydrocarbons is indicated by the reaction of the paper (credits: U. Kraus, BSH).

[9] Oil test paper can be used to distinguish between the presence of water and oil (for example in a tank or when sheens visible on a water surface are suspected to be from algae. If the negative test with oil test paper is enough to determine not to take a sample is subject to varying national regulations and laws.

SAMPLING
Glass bottles with inert lining in the lids (e.g. PTFE, ETFE etc)[10]
ETFE net (typical mesh opening width: 150 or 250 μm)
(Telescope) sampling rod (aluminium or stainless steel) and/or fishing rod
Nitrile gloves (disposable)
Aluminium foil containers
Aluminium foil (preferably of thickness of 0.03 mm or more)
Polyethylene (PE) cornet or conical PTFE bag[11]
Adapter for cornet
Nylon cord
Clips (stainless steel)
Stainless steel/PTFE spatula
Stake of clean plastic buckets[12] (e.g. 5 L)
Sampling pole (stainless steel or PTFE)
Sampling device for helicopter-based sampling

LABELLING
Labels for sampling bottles (preferably waterproof)
Tamper-evidence labels/Security seals or tape /evidence bags

[10] Typical bottle sizes for oil sampling range from 100 mL to over 400 mL, depending on limiting factors in logistics or storage capacity. Wide neck bottles are generally advantageous; brown glass bottles might help with protection from sunlight, but their content is not visible clearly and from samples in such bottles, no pictures can be taken. Therefore, clear glass bottles are preferable (as long as sun protection can be provided in another way, e.g. by wrapping the sample glasses into aluminium foil). Furthermore, when choosing a sampling bottle, care is necessary to choose one with a well-produced lid, otherwise there is a real risk to have silicon rubber (back side of the inert liner in the lid) contaminating the sample with siloxanes.

[11] Alternatively, a custom-made option can be used (see chapter 8.2.1).

[12] Since these buckets are exclusively meant for single-use only and contact time with oil is very limited if the sampling is done correctly, plastic is the most feasible option in this case. Steel or PTFE buckets would have to be cleaned with solvents between sampling of different sites or at different times, which is rarely possible in the field (see chapter 7.3).

DOCUMENTATION
Pen
Pencil
Permanent marker (e.g. Edding)
Sampling Manual
Sample Submission Form/Request for Analysis Form
Chain of Custody Form
List of sampling equipment/check list
Camera

PACKAGE AND SHIPPING
Cooler
Cardboard packaging
Adhesive tape

The means of transport for the sampling equipment depends strongly on the on-site situation. In some cases, water-tight hard cases are the best option, while in others backpacks are much more appropriate as mean of transport for the basic sampling equipment (Figure 2).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NCA (Norway)


NCA (Norway)


SASEMAR (Spain)

SASEMAR (Spain)

Figure 2: Examples of various means of transportation of the sampling equipment

CHAPTER 8

SAMPLING PROCEDURES

8.1 BACKGROUND SAMPLES AND BLANKS

Background samples are needed in an environmental crime investigation to determine the background level of substances of interest at the location of the spill. For example, in case of oil spills on water, a sample from a water body close to the oil spill but unaffected by it would be sampled ("clean water sample"). By analysing this background sample, the oil already present at the location at the time of a spill and therefore not caused by it, should be distinguishable.

It is imperative that the background sample is taken in the same way as the spill sample to avoid differences in the contaminant uptake by different sampling methods. If spill samples are taken by use of an ETFE net, the background sample must be taken with an ETFE net as well. For example, taking a sample of actual water as a background sample would under-represent contaminants in the sample in relation to a sample taken with an ETFE net.

In case of large or fragmented spills, it is advisable to take more than one background sample. Since they are part of the legal sampling, background samples are to be treated in the same way as the samples of the oil spill in regard of sealing, labelling, documentation and storage.

Principally, it is advisable to take the background sample before the spill samples to make sure that there is no cross-contamination from the higher polluted area. However, in many real scenarios, time is of the essence in securing meaningful spill samples (for example, if the spill is highly susceptible to on-site weathering effects or to loss by drift) and, in those cases, the background sample should be taken last[13].

KEY MESSAGE

TAKE, SEAL, LABEL, DOCUMENT AND STORE THE BACKGROUND SAMPLES IN THE SAME WAY AS THE OIL SAMPLES.

Blank samples are (artificial) samples designed to demonstrate that collected samples have not been contaminated by transport and handling in the field, by sampling equipment or sampling containers or by laboratory equipment, detergents or solvents. As every sampling situation is unique, the appropriate use of blanks differs from case to case. While laboratory blanks are outside the obligation of sample takers, sampling equipment especially should be routinely checked to not have an impact on the collected samples. If for example there is doubt that certain parts of sampling equipment could have an effect on the subsequent analysis, unused samples of this equipment should be handed in as blanks together with the samples for analysis. The same applies if oil samples can only be retrieved from oil intermingled with other material like absorbent material etc. In such a case, a sample of the material without oil has to be handed in to the laboratory as a blank sample[14].

[13] While proving no background contamination took place can be important to a case, various practical limitations can prevent the sampling of background samples, such as weather conditions or flight time restrictions when sampling from the air via helicopter. However, such restrictions especially apply to remote locations while pre-spill contamination is particularly an issue in areas markedly impacted by human activity (harbours, ship lanes, shorelines).

[14] Please note that such "mixed" samples always present problems to analysis – or they can make analysis impossible. It must always be the aim of the sample taker to retrieve untainted oil samples.

8.2 SAMPLING OIL ON WATER

KEY MESSAGE

IT IS ADVISABLE TO CHOOSE SAMPLING POINTS THAT ARE LOCATED AT THE THICKEST CONCENTRATION OF THE OIL SPILL SINCE WHEN SAMPLING OIL FROM WATER, IT IS IMPORTANT TO COLLECT AS MUCH OF THE OIL AS POSSIBLE WITH AS LITTLE ACCOMPANYING WATER AS MANAGEABLE.

By doing so, one ensures to reduce the impact of weathering as far as possible since oil at the fringes of a spill is generally impacted more severely by weathering than oil in the centre of a spill.

1. Depending on the type of spilled oil, different sampling methods are applicable. Which sampling technique is chosen depends on the kind of oil to be collected. Light fuels in thin layers (sheens) are preferably sampled in a different manner than tar balls.

2. Additionally, spatial conditions might determine the use of the sampling equipment. In narrow spaces, large sampling devices might not be manoeuvrable or appropriate.

3. The situational scenario of the spill and where the sampling is taking place will also define the way of sampling and the equipment to be used, as shown below.

8.2.1 SAMPLING SOLIDIFIED OIL (THICK LAYERS OF OIL, MOUSE, TAR BALLS)

Thick layers of oil (for example typical heavy fuel oil spills) and other solid appearances of petroleum oil are preferably sampled with the following equipment:

- Aluminium foil containers (clean)
- Polyethylene (PE) cornet or conical PTFE bag
- Clean plastic bucket (with or without holes)
- Sampling pole (stainless steel or PTFE)

When the spill to be sampled is in easy reach of the sample taker, **aluminium foil containers** (Figure 3) are good tools to catch the oil layer and to separate it from the water. Start by punching small holes into the bottom of the aluminium foil container with the use of scissors or a cutter from the sampling kit. Then dip the container into the oil spill (using gloved hands!). Oil will be collected in the container while the water drains away. If necessary, repeat the motion. When enough oil has accumulated in the container, it can be poured into a glass bottle or be transferred using a stainless-steel spatula.

Aluminium foil containers are especially handy for sampling solid lumps of oily material or tar balls. They are caught in the container and after drainage of the water, the aluminium container is simply closed by the accompanying lid or just folded around the collected, solid sample without further getting into contact with it. If necessary, supplementary casing can be applied that prevents remaining excess water or oil from the sample from leaking.

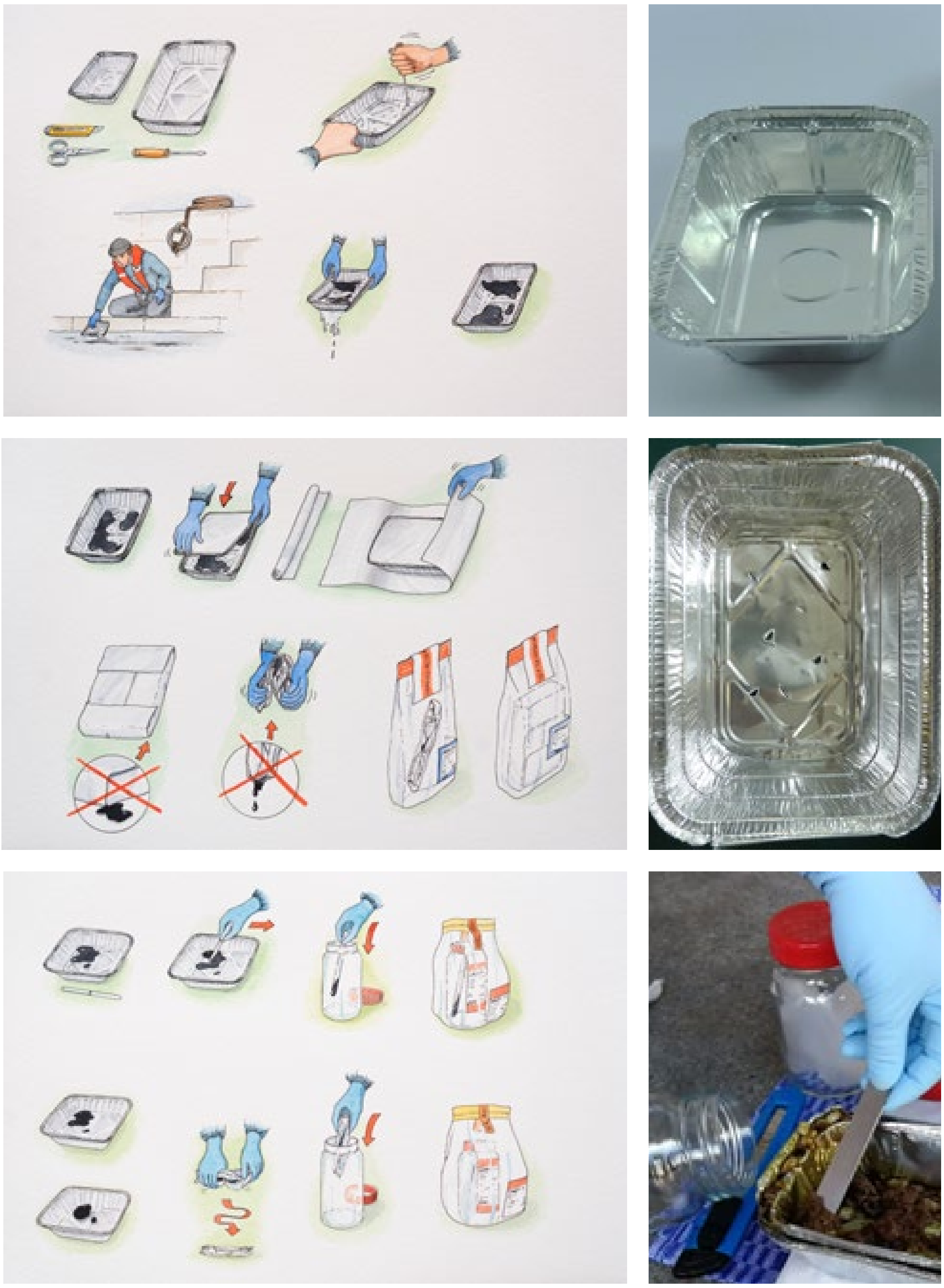
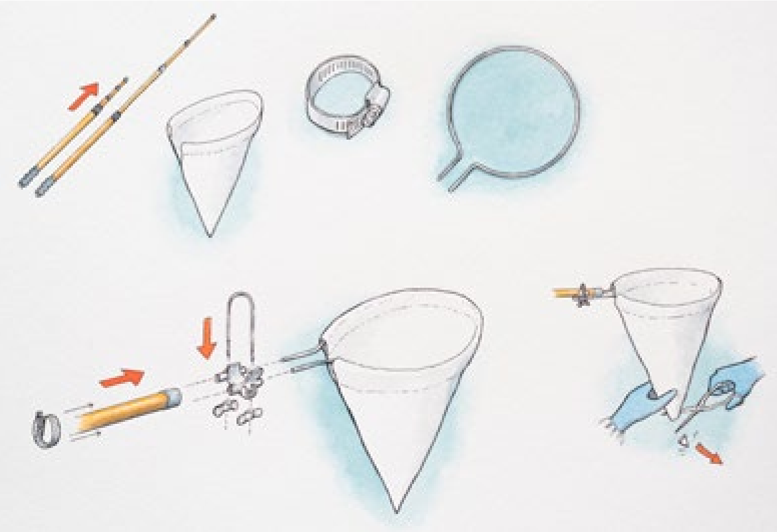
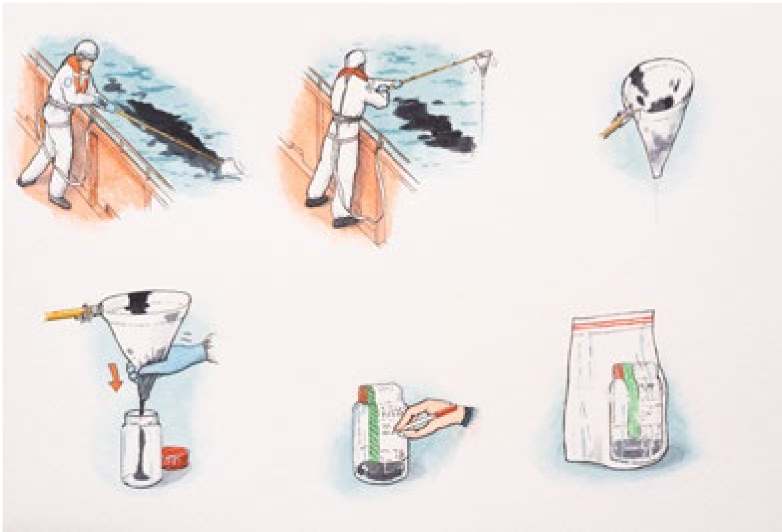


Figure 3: Aluminium foil containers used for oil sampling. Credits: U. Kraus, BSH



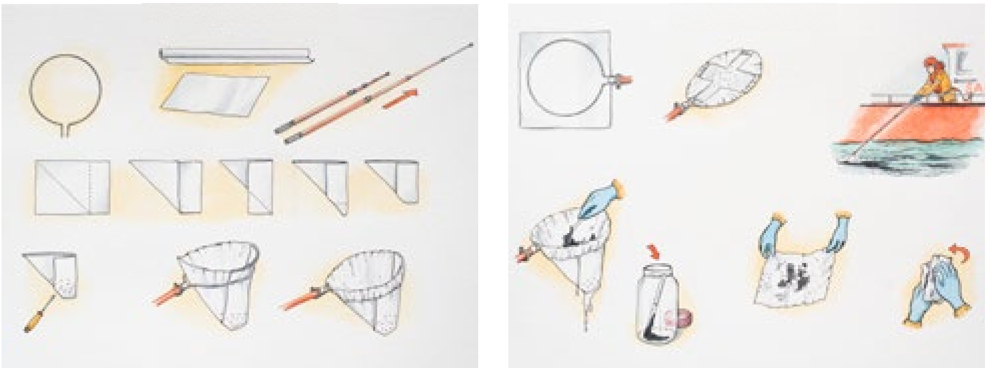


In case an oil spill is not directly reachable, **extendable rods** with a holder (e.g. a metal ring) fitted with a **polyethylene cornet** or **conical PTFE bag** can be used. The cornet or bag is attached to the ring and the bottom tip of the bag is cut off, creating a funnel with a small outlet. By sweeping the device through the oil spill, oil and water are collected in the cornet/bag. While the water will drain from it, the oil congregates as a layer on top of the water and therefore, can be retained in the bag/cornet and can be transferred to a clean glass bottle.

Training on oil spill sampling under the CTG MPPR work programme in Jovellanos Centre, September 2018.

As an alternative, **aluminium foil** can be attached to the metal ring (Figure 4) to create a more or less funnel-shaped form (depending on the oil layer to be sampled) which is perforated before use. While sweeping the aluminium funnel through the oil spill, water drains through the perforation while oil gets trapped in the funnel. After sampling, the oil is either transferred to a glass bottle using a stainless-steel spatula or the whole aluminium foil is detached from the holder, folded carefully and further prepared for transport. If the collected sample is small or if it is appropriate to only contain parts of the sample material, it can be placed in a wide-mouth glass bottle. If the sample material is too large for a sampling glass and if it is dry material, it can be wrapped in more aluminium foil and sealed with security tape. If the sample, however, contains water, it must be kept separately from all other samples to avoid cross contamination through leaking water.

Figure 4: Custom-made sampling device to be used instead of a PE cornet (please note that during a sampling exercise, an apple was used as “tar ball” in the right-hand picture). Credits: U. Kraus, BSH



In case of very large spills with heavy oils, it can be problematic to collect unbiased samples without cross-contamination while being in a completely oiled environment (e.g. on board of a pollution response ship or on a heavily oiled beach). This is especially complicated if the sample takers wear full protective gear, which can make sampling difficult. However, since large-scale spills are especially likely to lead to judicial proceedings, care has to be taken to ensure samples are taken in a way that is defensible in court.

By lowering a **clean plastic bucket** (without holes, see Figure 5; also Table 2 (incl. footnote nº 12 in chapter 7) directly into the oil spill, for example from the deck of a response ship, oil can be retrieved from a certain area of the spill close to the vessel in a controlled way [15]. Then, sampling oil from the bucket into glass bottles is much more practicable, easier and accurate. It is imperative to use a new bucket for each sampling spot, even if the same spill is sampled sometime later again.

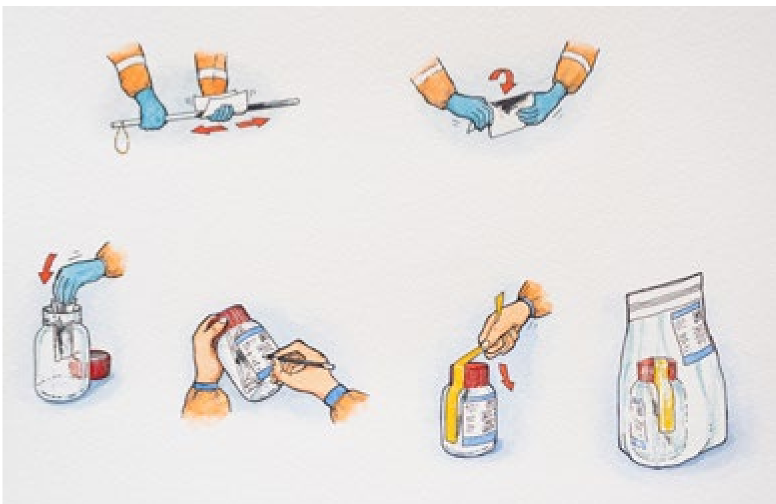
[15] This technique is also useful to look at a newly encountered oil spill up close, which might help with the determination of the next procedural steps.

If the rope with which the bucket was lowered to the spill was oiled, the oiled part should be removed (cut) so to have an oil-free rope ready for the next sampling. Otherwise, cross contamination is likely to occur.

In the same way as described above for other sampling devices (e.g. aluminium foil container), it is also possible to perforate a sampling bucket before use (i.e. create small holes in the bottom of the bucket).

Figure 5: Stake of clean buckets (5 L) for single use.
Credits: U. Kraus, BSH

Another possibility to sample thick oil layers can be a clean **pole of stainless steel** or PTFE which is swept through the spill and the oil is subsequently wiped off the pole with an ETFE net which is then placed into a sampling bottle. For each new sample (except replicates from the same sampling point), a fresh, unused pole has to be used.



If no other sampling equipment is available, it is possible to use a sampling bottle directly. This is not problematic when tar balls or lumps of solidified oil are sampled. However, it is more complicated to use this method directly to skim floating oil from the water surface. This is because the sample taker will not be able to prevent the oil contaminating the outside of the sampling bottle. This leads to problems with proper cleaning and avoiding cross contamination of other samples during sample taking, handling and transportation.

KEY MESSAGE

THE DIRECT USE OF A SAMPLING BOTTLE FOR FLOATING OIL SHOULD BE THE LAST RESORT IN THE ABSENCE OF OTHER OPTIONS AND THE INCREASED RISKS OF CONTAMINATION HAVE TO BE PROPERLY ADDRESSED.

8.2.2 SAMPLING LIQUID OIL[16] (INCLUDING SHEENS)

Light oils like diesels often present themselves as thin layers or sheens on the water surface. For such thin layers of oil floating on water, **ETFE nets** are the preferred method for sampling. ETFE is inert and does not react with the chemicals used in oil analysis and is therefore more suitable than other wiping material. The composition and structure of the ETFE net material ensures a high uptake rate of oil (typical mesh opening widths in use are 150 or 250µm). Thus, if an oil sheen is visible on the water, it is likely that by using the ETFE net, enough oil can be sampled from the water for analytical procedures.

On the other hand, ETFE nets are susceptible to contaminations since they very easily attract all kinds of oily substances. Therefore, there is high risk of the collection of unwanted contaminants besides the targeted oil. This is especially crucial when the overall amount of sampled oil is small and subsequently, contaminations can have profound effects for the analysis. For this reason, handling of ETFE nets must always be carried out wearing gloves and should be kept to the unavoidable minimum.

Pre-checked[17] ETFE nets are transported in clean glass bottles. Alternatively, they are supplied in clean PE-bags. For sampling, take the ETFE net out of the bottle/bag and swipe it repeatedly through the oil layer on the water. If necessary, the net can be attached to the line of a fishing rod or other sampling device (Figure 6). Attach the line by a single-use stainless-steel clip and discard the clip and any oiled parts of the line after sampling[18].

[16] The term "liquid oil" describes all types of oil that are fluid enough to attach to the ETFE net, unrelated to the specific oil type.

[17] New ETFE nets have to be checked for contamination before use (e.g. by substances used during production). If need be, they have to be cleaned in the laboratory before deployment in the field.

[18] If working in windy conditions, it might be necessary to weigh the net down with a float (like the ones used for fishing) that is attached to the lower end of the net by another line. Such floats and their lines have to be discarded and disposed of after.

Often, the net will be colored by the oil attached to it. In other cases, no discoloration of the net will be visible. However, this does not mean that not sufficient oil has been collected. After swiping the net repeatedly through the spill, retract it, detach it from the clip (always use gloves!) and place the net in the bottle/bag without delay. Leaving the oiled net in the open for long can quickly lead to evaporation losses of the sampled oil.

Oil sampling by ETFE nets with the help of an extendable rod or fishing rod is well suited for sampling in areas with limited space (e.g. sampling in the engine room of a ship or sampling between embankment and the side of a ship in a harbor (see Figure 7). Furthermore, ETFE nets are used with sampling devices from high altitudes (see chapter 8.4).

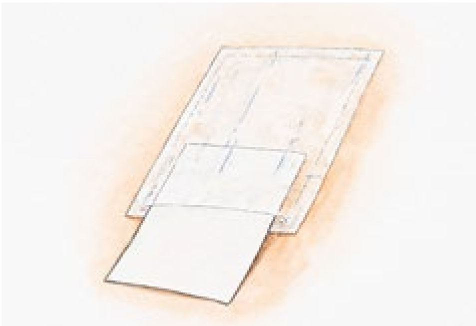
KEY MESSAGE

HANDLING OF ETFE NETS MUST ALWAYS BE CARRIED OUT WEARING GLOVES AND SHOULD BE KEPT TO THE UNAVOIDABLE MINIMUM.

Figure 6: Nylon line with stainless steel clip (single use article). Credits: U. Kraus, BSH

Figure 7: Examples for the use of the ETFE net. Credits: U. Kraus, BSH

As described in the previous chapter, if no other sampling equipment is available, it is possible to use a sampling bottle directly to skim the oil from the sea surface. While this is not recommended for thick oil layers, it is even less advisable for sheens since it is almost impossible to collect a reasonable amount of oil by simply skimming the surface of a water body into the glass bottle. Moreover, the amount of water in the sampling bottle remains high, which leads to biological degradation and secondary weathering of the small amount of oil in the sample. At the same time, laboratory work on such samples is much more complex and time-consuming. Overall, this is not a sampling approach which could be expected to lead to good results. For that reason, some laboratories do not admit samples with a high content of water for analysis.



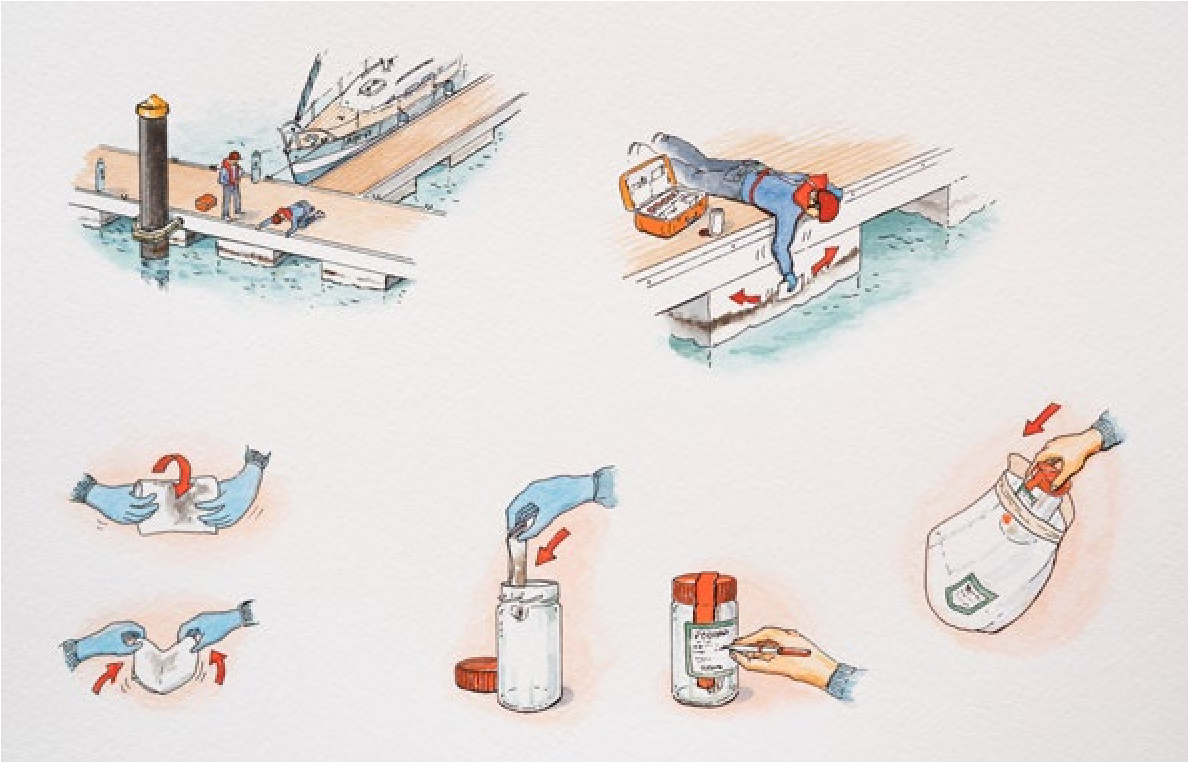
8.3 SAMPLING OIL FROM SOLID SURFACES (HARBOUR STRUCTURES, ROCKS, BEACHES, MACHINERY, ETC.)

When sampling oil from solid surfaces like harbour structures, rocks, debris on beaches, machinery etc., the **ETFE net** is useful for swipe samples (Figure 8). Alternatively, a **stainless-steel spatula** can be used to scrape oil from the surface (Figure 9) with the spatula and the spatula being transferred to a glass sampling bottle afterwards.

Figure 8: Taking a swipe sample with the ETFE net. Credits: U. Kraus, BSH

In all cases, it is important to ensure that only the oil layer is swiped or scraped without including underlying material in the sample. If there is a possibility of picking up any additional or underlying material, it is especially important to collect background samples from the surface in question.

Figure 9: Stainless steel spatula and its use for scrape sampling. Credits: U. Kraus, BSH



8.4 SAMPLING OIL FROM HEIGHTS (E.G. HELICOPTERS, HIGH EMBANKMENTS OR FROM THE SIDES OF LARGER SHIPS)

For sampling oil spills on water surfaces from high altitudes, e.g. from helicopters, high embankments or from the sides of larger ships, special equipment is used.

While different types of sampling devices are in use, they generally consist of a **floating body** which acts as counterweight to get an ETFE net to the water surface and to keep it afloat. The sampling device is lowered into the oil spill from above, where it floats and brings **an ETFE net** into contact with the spill.

Sampling apparatus have been developed using a floating body of empty PTFE bottles with an inert cylinder attached to it, which will float horizontally on the water surface due to the buoyancy of the empty bottles. An ETFE net is situated in the tube (Figure 10). When the apparatus drifts on the sea surface, oil will attach to the ETFE net. After sampling, the cylinder with the ETFE net is removed from the floatation device and packed for analysis.

Floating bodies can also be made from aluminium (Figure 11, Figure 12). The procedure remains principally the same and the ETFE net deployed by help of the floating device is transferred into a clean sampling bottle after the device is lifted back on board (always use gloves!). Alternatively, the sampling device with the net still attached might be placed in special containers provided for transport.

Since cleaning of such sampling devices cannot be achieved in the field, several of these devices should be kept available on-site and a new sampling device needs to be used per sample. The wet or contaminated part of the rope used to launch and retrieve these devices also needs to be changed between sampling. In case of sampling from a helicopter, where a change of the winch cable is not possible, it is crucial to equip each sample device with a long enough line to attach it to the winch cable to make sure the cable itself does not get into contact with the oil on the water.

Another sampling device used from heights, especially in remote sea areas, are **sampling buoys.** They can be released from fixed-wing aircrafts unable to be positioned over a certain spot for any length of time. Sampling buoys can be deployed from pressurized high-flying aircraft through gravity tubes or from un-pressurized high-flying aircraft. The air-deployable sampling device is specially designed to survive the impact when thrown into the slick. It consists of a buoy to which sampling pad (ETFE/PTFE net) is attached, a parachute, to act as a drift anchor after deployment and a GNSS tracker or a VHF transmitter. The latter allow the retrieval of the sample-buoy" through a vessel.

Once the sample-buoy is collected from the water, the sampling pad is removed from the buoy and placed into a glass bottle using gloves and avoiding touch as much as possible.

For successful sampling, the crew must follow the air-deployment instructions given by the distributor, including the appropriate speed, altitude and range of acceptable wind speed.

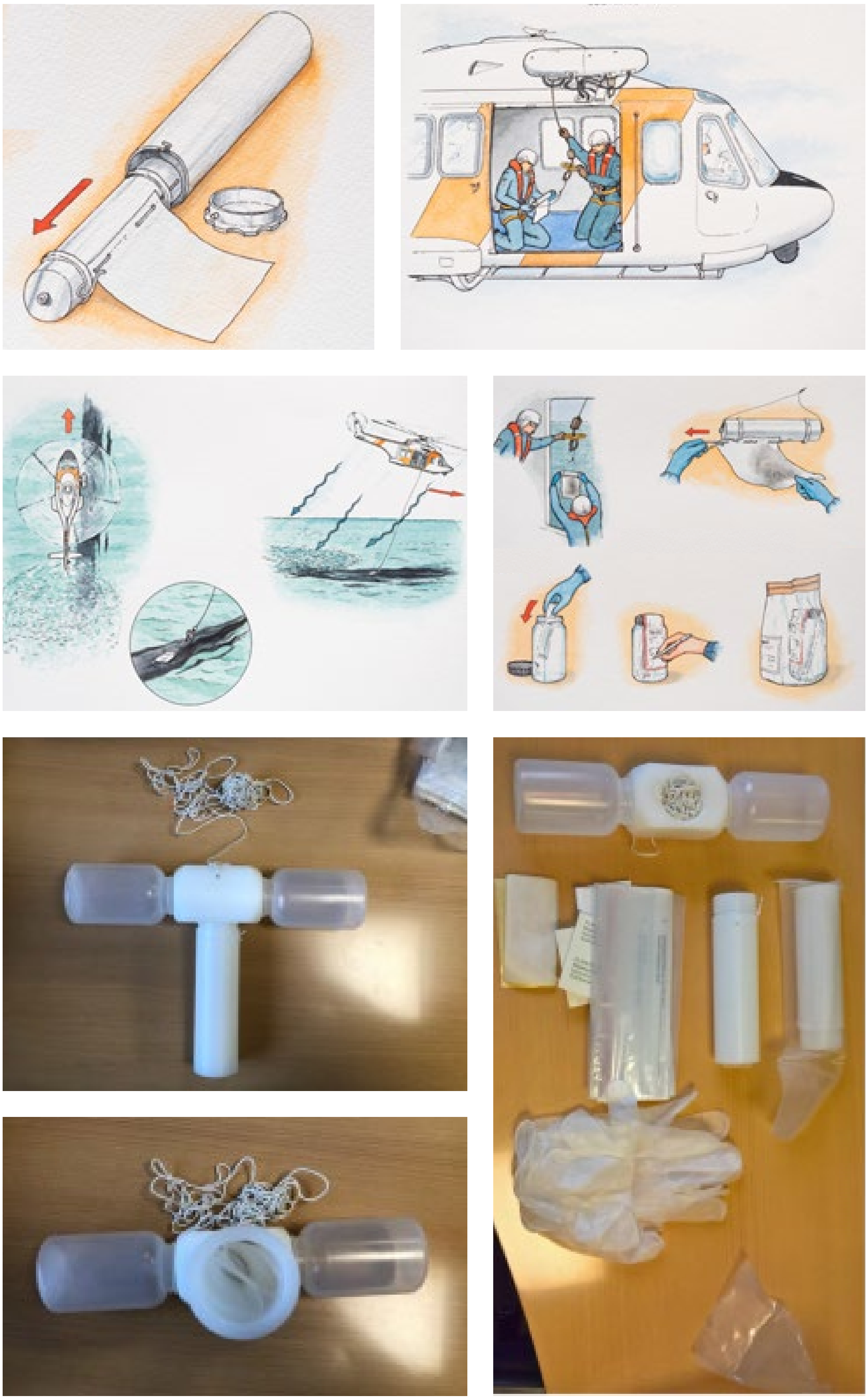


Figure 10: Sampling device set for oil sampling from heights, using a floating device, nylon cylinder and ETFE net. The long white cylinder that is attached to the flotation device has ETFE net attached on the inside with a Nylon strip. Top left image shows the set ready for sampling. Bottom left picture shows the ETFE net inside the nylon cylinder. The Nylon cylinder with the ETFE net is removed from the floatation device after sampling, and packed for analysis. Credits: Danish Defence.

Figure 11: Example of a sampling device for oil sampling from heights with a floating body of aluminum. Upper left side: pre-assembled sampling device with mounted ETFE net. Left side middle and below: Pre-mounted sampling device in/beside the transportation cylinder. Credits: Pictures left side: BSH; picture right side: German Federal Police Department for Maritime Security

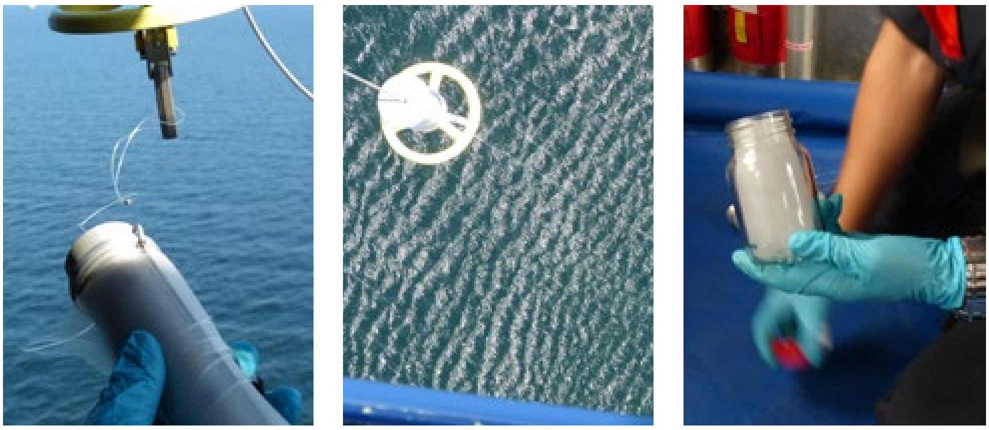


Figure 12: Sampling onboard a helicopter. Credits: German Federal Police Department for Maritime Security

8.5 SAMPLING OIL FROM OFF-SHORE INSTALLATIONS (PLATFORMS, WIND FARMS ETC.)

Sampling from off-shore installations such as platforms (off-shore drilling rigs as well as converter platforms from wind farms) or wind turbines often resembles sampling strategies used in other situations.

If for example, oil from a hydraulic crane has been spilled, the sampling situation might resemble a situation on the deck of a ship. If the oil spill occurred in the engine room, sampling on off-shore installations resembles very closely the one in a ship's engine. While sampling on an oil rig probably can take place on the structure itself, oil which is dripping from the blades of a wind turbine into the sea is normally sampled from a ship or boat. A difference and difficulty to account for when sampling off-shore installations is the additional obstacle of reaching the structure (for example, entry into the area of a wind farm or an oil rig needs special permissions and on a practical side, berthing manoeuvres can be challenging).

8.6 SAMPLING OF OIL ON BOARD SHIPS

Sampling on board ships can mean taking samples from diverse vessels such as sport yachts to an oil tanker (Figure 13). This indicates that sample takers can encounter very different sampling environments, working environments and conditions. For this reason, sampling on board ships is exclusively done by specially trained people. It is outside of the possibilities of these guidelines to account in detail for all individual circumstances.

KEY MESSAGE

SAMPLING ON BOARD OF A SHIP MUST ALWAYS BE CARRIED OUT BY SPECIALLY TRAINED PERSONNEL!

When sampling on board of a ship, special attention must be paid to health and safety measures (see chapter 5). Samples are collected at machinery spaces with excessive heat, in physically cramped positions (manhole sampling), oxygen deficient environments and/or surrounded by toxic fumes and liquids, so the use of appropriate and mandatory PPE is obligatory.

Typical areas for oil sampling on ships are cargo and fuel tanks[19] , bilge(s) and in case of large ships, sludge tanks and oily water separators [24]. Additionally, in case of large ships, purifiers, incinerators, and cargo areas can also be of interest.

[19] The manual sampling of liquid petroleum oils from ship tanks is described in detail in ISO 3170:2004 "Petroleum liquids - Manual sampling" [19], therefore, sampling from ship tanks is not addressed in detail in this guideline. However, in Appendix 3, principle steps of such sampling are described.

The manual sampling of liquid petroleum oils from ship tanks is described elsewhere (24) so will not be described again here. Depending on the situation, the most usable tool for sampling outside of these areas on board of a ship is the **ETFE net**, especially when a swipe sample can be taken. As described already (see chapter 8.3), when taking swipe samples, it is important not to press too hard on the sampled surfaces to avoid rubbing off underlying material. If there is a possibility of picking up underlying material, it is especially important to collect background samples from the surface in question.

KEY MESSAGE

WHENEVER IT SEEMS DIFFICULT TO AVOID COLLECTING UNDERLYING MATERIAL WHEN COLLECTING SWIPE SAMPLES, ENSURE TO TAKE BACKGROUND SAMPLES FROM THE SWIPING AREA.

ETFE nets are also often useful for small bilges or tight areas in the engine room (if need be, by use of a line to drop the net into small spaces, see Figure 7). On the other hand, for larger pooled spills, a single use, **clean bucket** could initially be used to take a sample. In a second step, the sample is then taken from the filled bucket by help of an ETFE net, which then can easily and securely be placed in a clean glass bottle (see page 39). As described in previous chapters, if no other sampling equipment is available, it is possible to use a sampling bottle directly. However, with the exception of sampling from dripping lines or pipes directly, it is not advisable. For example, if a sampling bottle is dipped into a bilge, it gets oiled from the outside and on the brim, which makes it nearly impossible to avoid cross contamination.

More information about the legal framework and procedural steps on board of a ship besides the sample taking are given in Appendix 3.

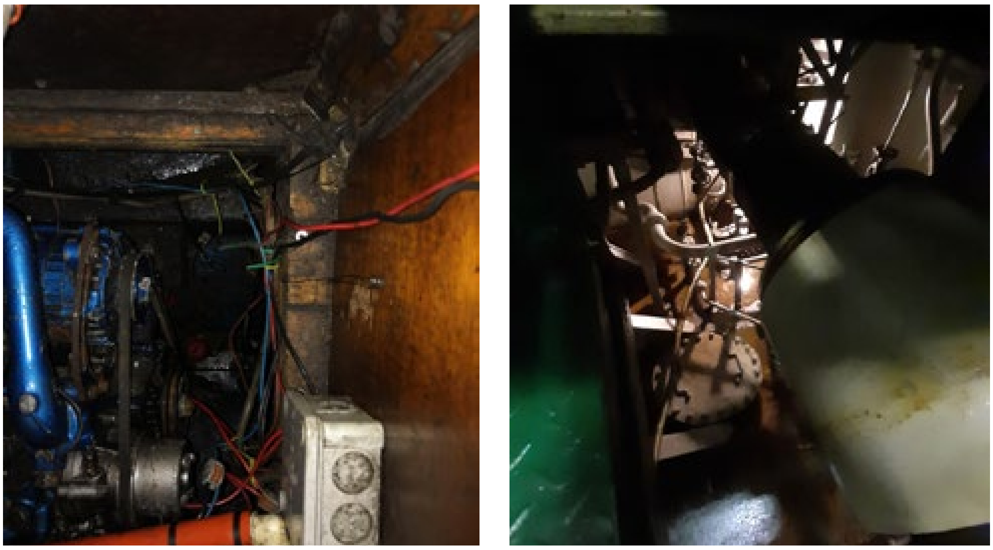


Figure 13: On the left: Bilge area of a small coaster. The tight space is most easily sampled with an ETFE net on a (fishing) line. Credits: WSA Lübeck, Germany. On the right: Bilge of a commercial vessel. The large space (sometimes several stories high) requires often additional equipment (e.g. a rod or a fishing line) to get the ETFE net to the bottom. Credits: D.G.M.M., Ministerio de Transportes, Movilidad y Agenda Urbana, Spain

8.7 SAMPLING OF OIL FROM WRECKS

Sampling oil from wrecks has been an emerging issue over the last two decades [25-27]. Sampling of an already leaking wreck or a wreck expected to leak (e.g. tank) allows the identification of the stored oil. Furthermore, it is important in the preparation of the salvage of a vessel with oil still on board.

Sampling from wrecks is a highly specialised technical procedure which starts with the assessment if oil is trapped inside a sunken vessel. If this is the case, a procedure of hot tapping (or pressure tapping) is performed, in which the oil-holding tank is punctured and hot steam is pressed into the tank to liquefy the oil inside before pumping it to the surface. Today, this is usually done by use of remotely operated vehicles (ROVs) since the work is, especially with increasing water depth, dangerous or impossible to perform for divers [26]. Since this sampling process is highly specialised and performed exclusively by specialised companies, it is not addressed in a dedicated sampling scenario in this document.

However, if there is oil already leaking from a sunken vessel, divers or a ROV's arm or -manipulator can be used to collect a sample underwater. Alternatively, if oil has already reached the water surface, sampling techniques used for sampling described in the document from maritime units can be applied.

8.8 SAMPLING OIL FROM OILED ANIMALS

When taking samples from oiled animals (e.g., marine/aquatic birds, marine mammals, sea turtles etc), it is important to distinguish between sampling from dead and sampling from live animals and to coordinate closely with the authorities coordinating the wildlife response or with wildlife experts. Live animals being collected on the shoreline may either be euthanised or treated for rehabilitation, while dead animals should be collected for the purpose of scientific impact assessment, which may include necropsies to determine species, sex etc. In these cases, it may be easiest to obtain oil samples directly from the wildlife response teams (in impact assessment facilities or from live animals in rehabilitation centres). It should be considered in the sampling strategy that oiled animals may come ashore in locations other than where the oil does or that oiled animals may continue to come ashore after the oil spill is contained/cleared up.

SAMPLING FROM DEAD ANIMALS

Sampling experts can treat collected dead animals as a "solid object" for an oil swipe with an ETFE net. Since the ETFE net attracts all kinds of substances, it is important to only sample the oil, by swabbing it off the contaminated feathers, body, wings or fur of the animal. Alternatively, tissue-free samples, like clipped oiled feathers from dead birds or oiled fur from marine mammals, are suitable as samples. For oil identification purposes, it should always be avoided to send whole animals, body tissue etc. to a lab, as these may become rotten during transport. In situations where sending whole body tissues cannot be avoided, this should be coordinated with the impact assessment (necropsy) unit of the oiled wildlife response. The sampling strategy should also define what to do with dead animals once they have been sampled (disposal or keep for further scientific research).

SAMPLING FROM LIVE ANIMALS

Sampling from live animals should be kept to a minimum and carried out in consultation with authorities and the wildlife experts involved. As a general principle, sampling should not lead to creating wounds, or any other compromise of the animal's health or its chance to be successfully rehabilitated. Clipping feathers or fur could affect the waterproofing in the rehabilitation process and needs to be carefully considered by the leading rehabilitation experts.

HUMAN HEALTH AND SAFETY

Sampling of oil from dead or live animals needs full consideration of human health and safety issues, including potential injuries from handling live animals and potential for spread of zoonotic diseases such as Avian Influenza. Expert advice and assistance as well as specialist equipment may be needed, under supervision of national or local authorities.

Further information can be provided by the EUROWA network (www.eurowa.eu).

KEY MESSAGE

WHEN TAKING SAMPLES FROM OILED ANIMALS IT IS IMPORTANT TO DISTINGUISH BETWEEN SAMPLING FROM DEAD AND SAMPLING FROM LIVE ANIMALS (THE LATTER UNDER STRICT ANIMAL WELFARE AND HEALTH & SAFETY CONSIDERATIONS).

ALWAYS COORDINATE SAMPLING OF WILDLIFE (WHETHER LIVE OR DEAD) WITH THE AUTHORITIES IN CHARGE OF THE WILDLIFE RESPONSE AND CONDUCT SAMPLING WITH THE ASSSISTANCE OF WILDLIFE EXPERTS.

credits: Rijkswaterstaat

CHAPTER 9

SAMPLE PROCESSING

SASEMAR material – photo taken by EMSA

9.1 LABELLING

All samples have to be labelled with suitable labels directly after sampling (best to use water- and oil-resistant labels, however, these are sometimes difficult to write upon). Labels have to contain all essential information to make sure that a sample is unambiguously identified and retraceable (Figure 14).

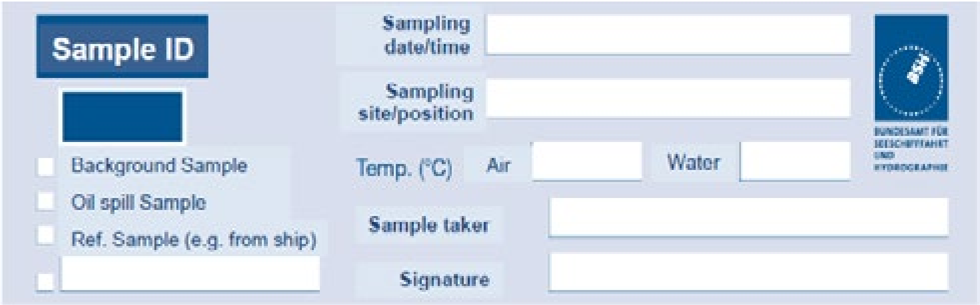

Sample ID
Sampling date/time
Sampling site/position
Background Sample
Oil spill Sample
Ref. Sample (e.g. from ship)
Temp. (°C) Air
Water
Sample taker
Signature
BUNDESAMT FÜR SEESCHIFFFAHRT UND HYDROGRAPHIE

Figure 14: Example of a sample lable (courtesy of BSH, Germany)

KEY MESSAGE

PROPER LABELLING ENSURES THAT A SAMPLE IS UNAMBIGUOUSLY IDENTIFIED.

9.2 SEALING

After sampling, legal samples must be sealed in a temper-proof way, either by the use of manipulation-proof security tapes, evidence bags or by sealing the transport containers with adequate plastic security seals (see examples in figure 15).

Figure 15: Different possibilities to seal samples (credits: upper left: SASEMAR Spain, lower left and right: BSH, Germany)

KEY MESSAGE

SAMPLES MUST BE SEALED IN A TAMPER-PROOF WAY!

9.3 TRANSPORT

All samples have to be kept in the dark and cooled (4 ± 3)°C during transport to the laboratory. It is imperative for successful laboratory analysis, that samples are brought to the laboratory directly after sampling and that there are no delays or prolonged storage times. Different climatic situations require different measures to ensure that samples are continuously cooled during transport. Furthermore, the travel time during transport also affects the cooling requirements. The easiest way to prove continuous cooling is the insertion of a temperature logger in the transport box or cooler.

Samples must always be transported with the proper accompanying paperwork. As minimum requirements, the analysis request form and the chain of custody form should be kept with the samples. Under no circumstances should samples be shipped without the proper documentation. In the worst case, samples arriving at the laboratory without documentation might get lost or might be destroyed without analysis due to the impossibility to trace their origin.

9.3.1 TRANSPORT BY COURIER

Depending on how the samples are packed, the courier has to sign off on the chain of custody form (see chapter 10.2.1).

9.3.2 TRANSPORT VIA MAIL

When oil samples are sent by mail, it has to be ensured that glass bottles are packed in a way that they do not leak or shatter. If samples contain free water or oil (for example samples from a suspected ship), absorbing material like oil absorbing pads should be introduced into the packaging to prevent leakage should the glass bottle be compromised during transfer.

It is advisable, especially in summer, to not ship samples on Thursdays or Fridays since there is a very realistic chance that the samples will get stuck in the mail over the weekend which prolongs transportation time and enhances the risk that samples get too warm. In this case, it is preferable to keep the samples refrigerated over the weekend and ship them out on Monday.

When shipping oil samples, it is important to adhere to all applicable national and/or international regulations and to check what type of regulations may specifically apply in each case.

KEY MESSAGE

OIL SAMPLES SHOULD BE TRANSPORTED TO THE LABORATORY IMMEDIATELY. THEY MUST BE KEPT IN THE DARK AND COOLED (4 ± 3) °C DURING TRANSPORT. SAMPLES MUST ALWAYS BE ACCOMPANIED BY THE ANALYSIS REQUEST FORM AND THE CHAIN OF CUSTODY DOCUMENTATION.

CHAPTER 10

SAMPLING DOCUMENTATION

10.1 SAMPLING FIELD NOTES

Sampling field notes are the backbone of the sampling documentation. They have to be precise and written in a way that they are meaningful, as it may be necessary to reconstruct the sampling situation years after the sampling has taken place. Court cases take a long time, so documentation should provide suitable information for all stakeholders years after its compilation.

Pictures or short videos of the sampling situation as well as of the sampling actions can greatly enhance the value of field notes.

KEY MESSAGE

SAMPLING FIELD NOTES ARE THE BACKBONE OF THE SAMPLING DOCUMENTATION.

10.2 REQUIREMENTS FOR LEGAL PURPOSES

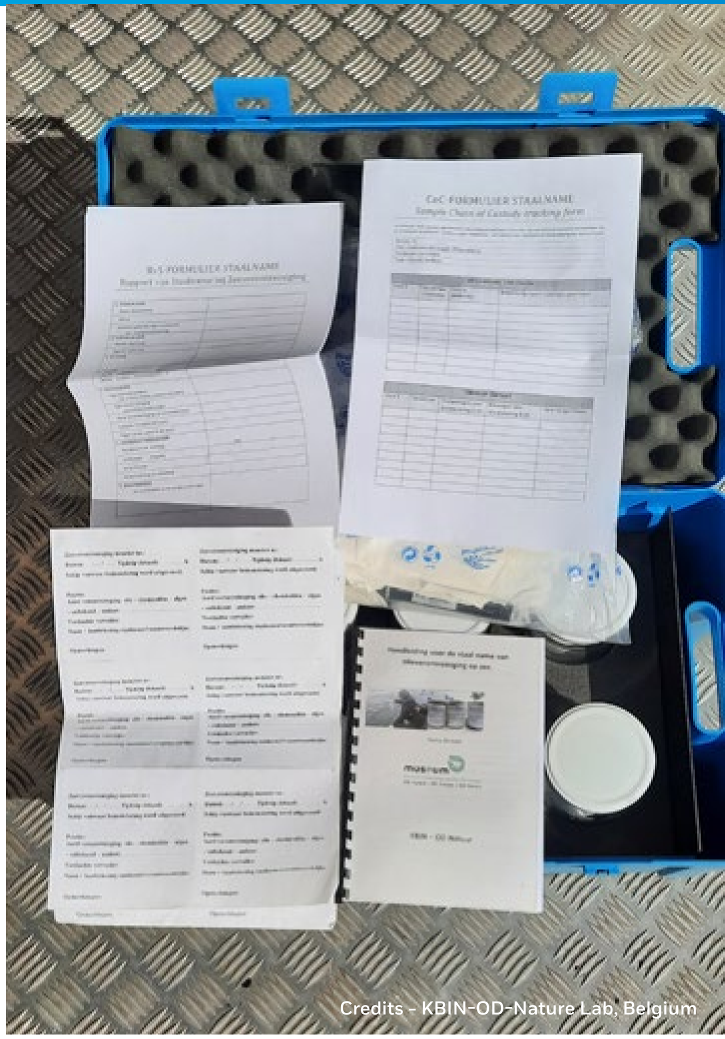
For legal sampling[20] the use of a standardised form of documentation is advisable (Figure 16). It helps to ensure all important points are addressed and it makes sampling documentation comparable. As a minimum, the documentation should cover the following points:

- Identification number of the sample(s);
- Information about the sample taker (e.g. name, rank/position, organisation / institution);
- Signature of the sample taker (and if required, signature of witnesses[21]);
- Contact information of the recipient of the results/the client (including a phone number for questions and further information).
- Information of date, time and location of the sampling (e.g. GPS coordinates);
- Identification of sampling points in a tank plan or piping diagram of a ship);
- Description of the samples (e.g. type of sample);
- Transport/Shipping information;
- Naming of the requested laboratory analysis.

To ensure that all sample takers use the same terminology, it can be helpful to provide a short glossary of the most important terms (e.g. swipe sample, background sample etc.), for example on the reverse of the Sample Submission Form (Figure 16 and Appendix 2).

[20] For definition and more information, see Glossary

[21] As revealed in a questionnaire among the EU member states in 2016, demands regarding legal sampling differ widely between single countries. “Workshop on oil fingerprinting and the work of the OSINet” held on 2 March 2016 at EMSA under the CTG MPPR work.



10.2.1 CHAIN OF CUSTODY

By default, all oil samples taken in oil spill forensic work are potential legal samples (meaning they are used in court cases), which makes the written record of custody mandatory. According to [19], **samples are in custody**, if they are:

1. in actual physical possession or;
2. in sight after having been in the physical possession of a person or,
3. if they are tamper-proof and locked up while being in physical possession.

The chain of custody form (see Figure 16, below) is therefore used to trace the possession of the samples over the whole investigation process from the moment the sample was taken to the moment the sample is destroyed at the laboratory after investigations are completed and/or the case is closed.

Everybody, who has direct contact with the samples, must document this and has to sign off on the samples as soon as the custody is transferred to another person. Please note, that in case of transportation by mail, the postman usually does not have to sign the chain of custody form, since samples prepared for shipping should be securely wrapped (see chapter 9.3) and therefore should not be accessible. Therefore, the condition of the received package should be noted by the receiver.

Working document in English (for information only) – be advised that only the German version is approved

BSH
BUNDESAMT FÜR SEESCHIFFFAHRT UND HYDROGRAPHIE

Sample Submission Form/Request for Analysis Form

Sample taker (name/institution)	
Client	
Recipient of results	
Address	
Phone	
Billing addressee	
External code (Client)	

Page No. ☐ of ☐

Delivery date/date of arrival ☐

Time ☐

Priority ☐ high ☐ normal ☐ extraction/conservation only

	Name of Sample Taker	Sample ID	Lab-ID	Matrix	Sampling date	Sampling time	Sample Temp. Control (at arrival at lab)	Sample description	Analysis requested Level 1 (GC-FID)	Analysis requested Level 2 (GC-MS)
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

Filled out by lab

Labor Sülldorf · Ölforensik · Sachgebiet M34 · Wüstland 2 · 22589 Hamburg · oelforensik@bsh.de · 040-3190-3352

Version 1.1

External code (Client) ☐

Internal case No. ☐

BSH
BUNDESAMT FÜR SEESCHIFFFAHRT UND HYDROGRAPHIE

Chain of Custody Form

- this document has to be kept with the Sample Submission Form -

Please note: The first entry is to be made by the sample taker!

Page No. ☐ of ☐

	Ceded by	Name (printed) / Signature	Date	Time	Received by	Name (printed) / Signature	Reason for transfer
1	Ceded by				Received by		
2	Ceded by				Received by		
3	Ceded by				Received by		
4	Ceded by				Received by		
5	Ceded by				Received by		
6	Ceded by				Received by		
7	Ceded by				Received by		
8	Ceded by				Received by		
9	Ceded by				Received by		
10	Ceded by				Received by		

Samples destroyed after termination of the proceedings ☐

Date ☐ Name (printed) / Signature ☐

Labor Sülldorf · Ölforensik · Sachgebiet M34 · Wüstland 2 · 22589 Hamburg · oelforensik@bsh.de · 040-3190-3352

Version 1.1

Figure 16: Examples of a sample submission form/request for analysis form (above) and a chain of custody form (below); courtesy of BSH, Germany)

CHAPTER 11

INTERNATIONAL CO-OPERATION

International co-operation in oil spill forensics has a long tradition, especially since trans-boundary spills regularly highlight the difficulties of oil spill identification in an international context and demonstrate the importance and added value of harmonised approaches in international co-operation.

OSINet[22] , the oil spill identification network of experts within the Bonn Agreement, was set up in 2005 following difficulties in identifying sources from the 2002 'Tricolor' oil spill accident. OSINet facilitates cooperation, mutual assistance and regular intercalibration studies in oil spill analysis for its members. For 17 years, OSINet comprised of experts not only from Europe, but from up to 47 laboratories around the world. OSINet's expertise is further utilised by participating in other co-operations like the drafting of this document, which aims to share and exchange good practice and expertise in oil spill sampling in Europe.

International cooperation is at the heart of marine emergency response efforts as well as regulations to prevent such incidents, which support the enforcement chain. Numerous sub-regional, regional, EU-wide and international mechanisms are in place and relevant guidance documents have been developed by, among others, Interpol [19] and IMO (for details, see Appendix 3).

[22] OSINet - https://www.bonnagreement.org/activities/osinet

CHAPTER 12

FUTURE DEVELOPMENTS, POSSIBILITIES AND CHALLENGES

12.1 THE USE OF REMOTELY PILOTED AND AUTONOMOUS VEHICLES FOR OIL SPILL DETECTION AND MONITORING

For the detection and monitoring of oil spills and their environmental fate, remotely piloted or autonomous vehicles are increasingly coming into focus. Remotely operated underwater vehicles (ROUV, or more generally ROV) have already been used earlier for the collection of spilled oil, to determine the best response and/or to determine the chemical profile of the oil (see Figure 17). Over the last decade, research projects have investigated the use of the technology for more elaborate tasks, such as the projects "Underwater Robotics Ready for Oil Spill – URready4OS", the "Expanded Underwater Robotics Ready for Oil Spill – URready4OS" [28] and the EU Horizon 2020 project "GRACE - Integrated oil spill response actions and environmental effect" [29]. Several guidance documents have addressed special issues (e.g. remote sensing under ice [30]). Practical issues can arise from the contamination of an ROUV during the oil sampling process, which has to be addressed and solved before its "reuse" to prevent cross-contamination between samples.

Today, the availability of mature, affordable smart technology helps to enhance the development of autonomous vehicles operating underwater (autonomous underwater vehicles - AUVs), on the surface (unmanned surface vehicles - USVs) or in the air (unmanned aerial vehicles - UAVs) to help detect, track and investigate oil spill events.

Figure 17: Oil sampling by help of a ROV (credits: SASEMAR, Spain)

12.2 SAMPLING SUBMERGED OIL AND SUNKEN OIL

To date, the characteristics of non-floating oil are far less well understood than those of floating oil. Research regarding the possibilities of detecting and tracking non-floating oil has gained momentum over the last decade, for example in connection with the Deep Water Horizon incident [31, 32]. The same applies for modelling techniques and research into recovery of non-floating oil [33-35]. Moreover, exploration of the state of the deep sea in general is increasingly addressed as an important issue regarding ecosystem conservation and sustainable management. For example, the Deep-Ocean Stewardship Initiative (DOSI[23]), an international network of experts that study human activity effects on the deep ocean, address the impact of oil residue on the sea bottom environment. However, it is in many aspects a field of work still in development.

It is outside of the scope of this document to give a detailed review of the state of the art in the field of non-floating oil. Therefore, some general information is given below and references for further information are included.

In general, non-floating oil is further distinguished as sunken oil (spilled oil that has sunken to the bottom of the water body) or submerged oil (spilled oil not on the water surface but in the water column, temporarily or over longer periods of time, for more detailed definitions see [33]).

For sunken oil, several existing sampling techniques are used based on sediment analysis of the sea-bed (e.g. grab sampler or box corer, see Figure 18, Figure 19), however, their use has proven to be limited and problematic especially in greater water depths and under dynamic environmental conditions ([36]; [33]). For submerged oil, even the detection of the oil is challenging, especially at greater water depths, and requires special equipment (e.g. submersible fluorometers or submersible mass spectrometers, see [37]) and specially trained personnel.

Figure 18: A box corer used for taking samples from the sea floor (left) and a sediment core extracted from the sea floor (left). Credits: U. Kraus, BSH

[23] https://www.dosi-project.org

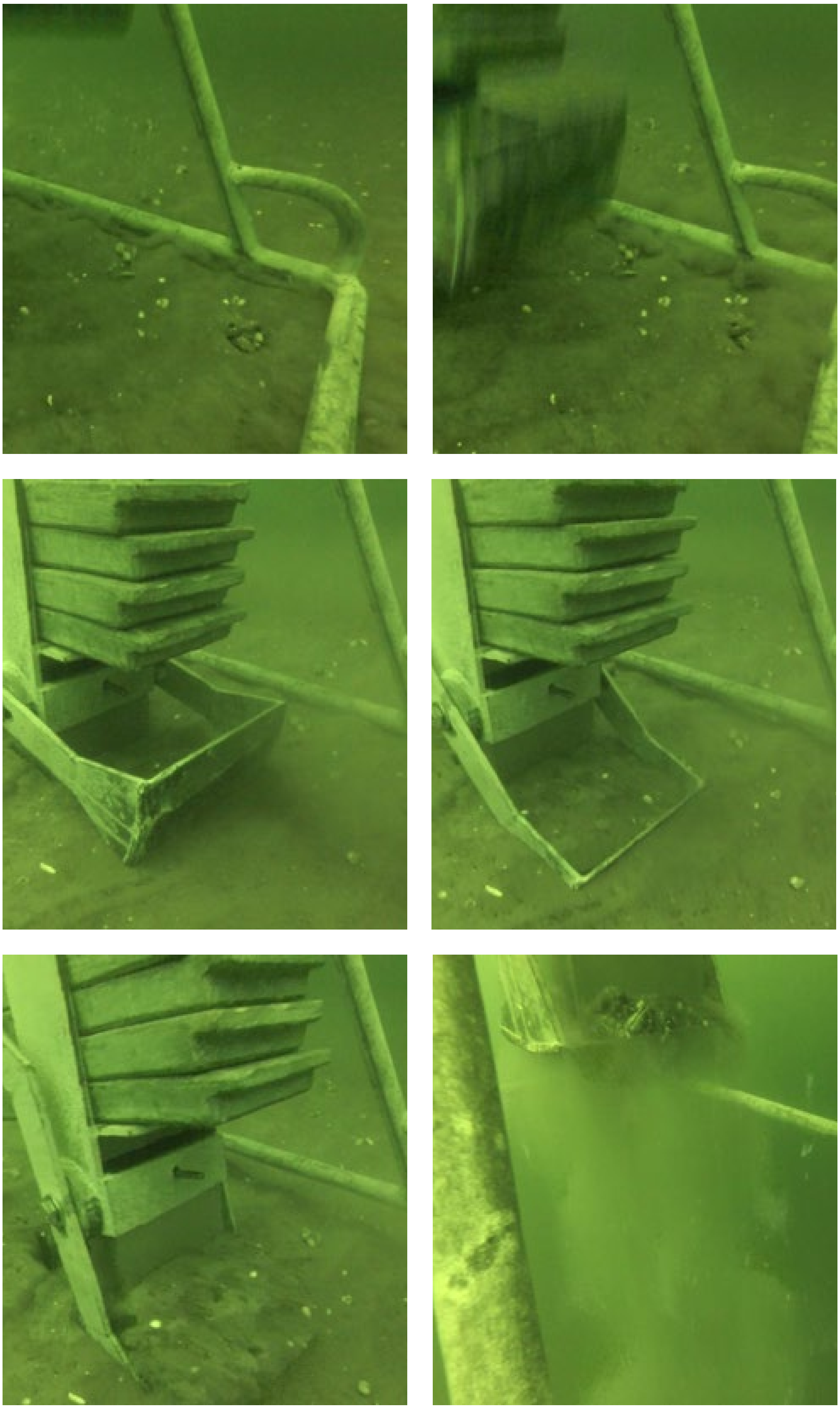


Figure 19: A box corer collecting a sediment core at the sea bottom (sequence from upper left to lower right). Credits: N. Fitz, BSH

12.3 NEW FUEL TYPES AND PERSISTENT FLOATERS

In a global effort to cut sulphur oxide (SOx) emissions from ships, the global sulphur cap entered into force on 1 January 2020. With this legislation, the maximum sulphur content in marine ship fuels is reduced to 0.5% (from former 3.5% (sulphur content of conventional heavy fuel oil (HFO); MARPOL Annex VI, regulation 14)[24] .

To comply with this requirement, several strategies are applicable, one of which is using compliant fuel with a sulphur content of not more than 0.5% (low sulphur fuel oil, LSFO). In anticipation of the upcoming regulations, an increasing number of LSFOs became available on the market. Furthermore, special emission control areas (ECAs) (currently) in Europe and North America demand the use of fuel with not more than 0.1% sulphur (ultra low sulphur fuel oils, ULSFO).

Additionally to LSFO and ULSFO, plant-based fuels ("biofuels") are increasingly used in the marine environment. For example, biodiesels (fatty acid methyl esters (FAMEs)) and Hydrotreated Vegetable Oil (HVO) are possible substitutes for conventional diesel fuels [38]. Furthermore, liquefied natural gas (LNG) is increasingly used in ships.

To supply compliant fuel oil (=fuel with reduced sulphur content), refineries can blend fuel oils with a higher and lower sulphur content, namely distillate and residue fuels, or remove sulphur from high sulphur residual oils. Additives may be used to enhance other properties, such as lubricity, since the low sulphur content makes these fuels more corrosive. During travel, ships nowadays use a variety of fuels, depending on the area they are in. However, fuel change-over procedures from a high sulphur fuel oil to a sulphur reduced fuel are complicated procedures that have to be rightly timed and executed to prevent damage to the engine, or in the worst case, complete engine failure [39]. Therefore, exact timetables and calculators are used to plan such changeovers carefully.

Beside these "new fuels", persistent floating substances like paraffin waxes, falling into the regulations under MARPOL Annex II, are increasingly recognised as a matter of concern within the marine environment due to increasing numbers of reported spills. As a consequence, on 1 January 2021, an amendment to MARPOL Annex II came into force for North West European waters, the Baltic Sea, Western European waters and the Norwegian Sea. This requires chemical tankers that unload a cargo of "persistent floaters" (as per definition in MARPOL Annex II, Regulation 1, § 23) to carry out a tank prewash and discharge the prewash material to a reception facility at the current port.

In regards of sampling, for most of these "new fuels" and MARPOL Annex II substances, the **ETFE net** should be a suitable sampling device that, to the current knowledge of the authors, will attract a sufficient amount of material for oil spill forensic analysis. In case of solidified materials like paraffin waxes, all sampling strategies described in this document referring to solidified oil can be used.

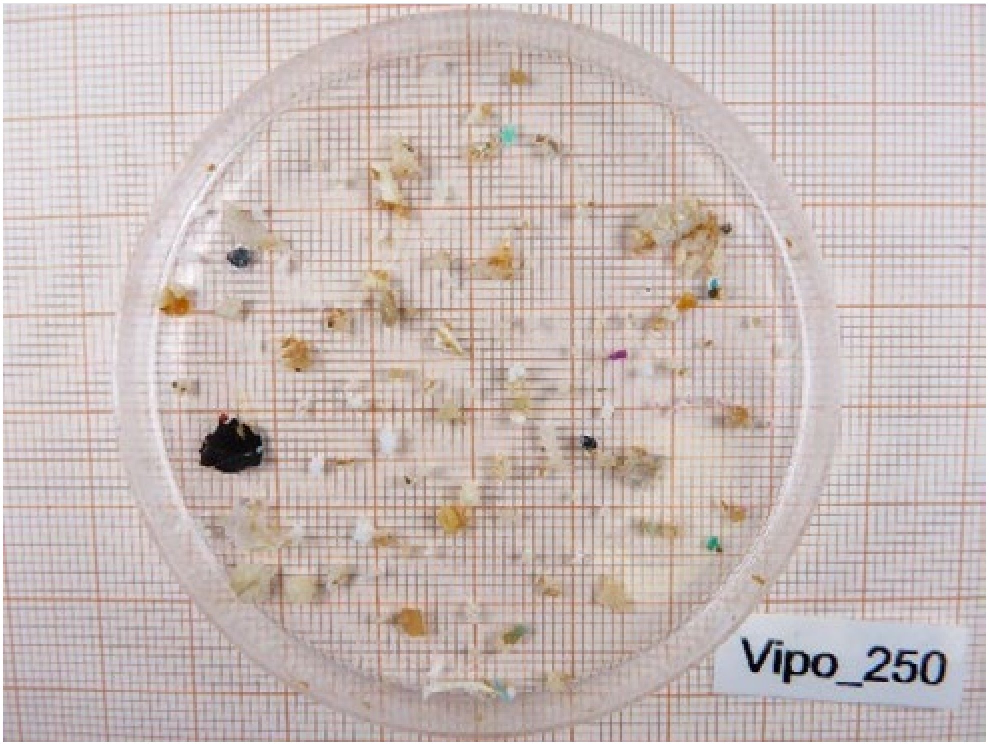
[24] https://www.imo.org/en/OurWork/Environment/Pages/Sulphur-oxides-(SOx)-%E2%80%93-Regulation-14.aspx

12.4 SAMPLING OF MATERIAL OTHER THAN OIL

As described above, new fuel types display unique properties and traditional oil sampling or response measures cannot always be used. This is even truer for other environmental pollution that is found in the marine environment.

Sampling of material other than oil is not within the scope of these guidelines.

However, many goods, e.g. plastic pellets/nurdles and paraffin waxes are transported by ship in bulk and pose an environmental risk at sea in case of spillage. Some of the sampling equipment described in the guidelines, such as glass bottles, are suitable for sampling other material than oil. Sampling of such materials allows for spill identification linked to a source material, as well as chemical characterization of the spill. For plastic pellets and other goods, analyses might be needed to check the potentially harmful substances in the material. This information is required to assess environmental impact and health and safety issues for response operations [40].

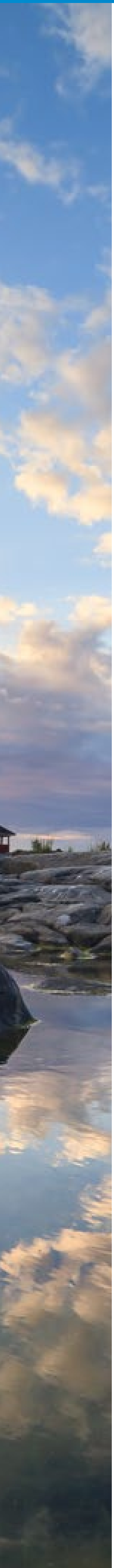


Material found on the ocean surface by help of a manta trawler. Credits: N. Fitz & L. Piephoe, BSH

CHAPTER 13

SUMMARY OF KEY POINTS

Summary of key points



- Forensic analysis of oil spills requires sampling according to high quality standards.
- Regular practical training is required for sample takers.
- Collecting forensic oil samples represents the preservation of evidence.
- Always take samples before oil spill cleaning activities start and accompany clean-up with a fitting sample plan.
- Oil samples should be taken exclusively by well-trained and experienced persons.
- During sampling, health and safety of the sample taker must always have priority and sampling must be carried out in a prudent health and safety manner.
- For sampling of oil, avoid typical equipment designed for the sampling of surface water since these generally include plastic parts which cannot be cleaned satisfactorily.
- Use single-use-materials for oil sampling.
- No amount of oil should be deemed to be too small. In case of sampling with the ETFE net, even if there is no visible oil on the net, the attached amount of oil can be enough for the forensic analysis of oil spills.
- Although small amounts of oil are sufficient for forensic analysis of oil spills, take as much oil sample material as is possible by filling up sampling bottles. However, never fill bottles to more than 75-90% (never to the brim).
- Take, seal, label, document and store the background samples in the same way as the oil samples.
- When sampling oil floating on the water, it is important to collect as much of the oil as possible with as little accompanying water as manageable. It is advisable to choose sampling points that are located at the thickest concentration of the oil spill.
- The direct use of a sampling bottle for floating oil should be the last resort in the absence of other options and the increased risks of contamination have to be properly addressed.
- Handling of ETFE nets must always be carried out wearing gloves and should be kept to the unavoidable minimum.
- Sampling on board of a ship must always be carried out by specially trained personnel.
- Whenever it seems difficult to avoid collecting underlying material in the process of collecting swipe samples, background samples should be taken from the swiping area.
- Proper labelling ensures that a sample is unambiguously identified.
- Samples must be sealed in a tamper-proof way.
- Oil samples should be transported to the laboratory immediately.
- They must be kept in the dark and cooled (4 ± 3) °C during transport.
- Samples must always be accompanied by the analysis request form and the chain of custody documentation.
- Sampling field notes are the backbone of the sampling documentation.

CHAPTER 14

CASE STUDIES

Note: Please bear in mind that the sampling of background samples is not explicitly discussed in the cases.

14.1 ARNHEM, THE NETHERLANDS

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING IN HARBOURS; SAMPLING ON BOARD SHIP

Incident description

On 26 December 2013, an oil spill took place in Arnhem, an inland harbour at the river Rhine in the Netherlands. A map of this harbour is presented in Figure 20. An estimated amount of at least 10 cubic metres of light fuel oil was spilled. This resulted in an unusual thick layer, up to several centimetres, caused by a strong wind keeping the spill concentrated in the corner of the harbour.

On the first day of the incident, three samples were taken of the thick layer that floated on the water surface because the identity of the spill was unknown. Because of the strong odour, it was suggested that the spill could be turpentine, from a chemical company in the area.

Sample taking and case work

Analysis identified the spill as diesel with a small amount of biodiesel.
On 7 and 11 January samples from all possible sources were taken. These comprised of fourteen different fuel tanks from eleven different ships. All ships that had been in the harbour during the incident and that had a fuel tank capacity large enough to cause a spill of ten cubic metres were included in the investigation. Among the ships were river cruise ships in winter storage, a tugboat and a crane ship.

Comparison of the analytical data of all the samples showed that only one fuel tank sample had no significant difference with the spill samples. The compositional differences of the fuel from three ships however were small, probably because the ships had been bunkered from the same source. When ships are bunkered with distillate fuels, there is always some remaining fuel in the tanks, this can result in unique mixtures. As long as compositional differences are larger than the variance of the analytical method, these differences can still be used as evidence of significant compositional differences between samples.

Figure 20: Map of the inland harbour of Arnhem, Netherlands, at river Rhine.

Lesson learned

For this case, it was important to collect samples from all possible sources.

14.2 AMSTERDAM, THE NETHERLANDS

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING IN HARBOURS; SAMPLING ON BOARD SHIP

Incident description

On 3 January 2011, an oil spill took place in the port of Amsterdam, an inland harbour in the Netherlands, which is connected to the North Sea by the 21 km long North Sea Canal. A picture of the map of the port of Amsterdam, with the spill indicated, is presented in Figure 21. A coaster coming from the Mercury harbour hit the side of an oil tanker that was on its way on the North Sea Canal. The full load of the tanker, about 250 tons of heavy fuel oil, was spilled. After the incident the leaking tanker was moved to a nearby dead-end branch of the canal and an oil boom was placed at the entrance of the branch to limit the further spreading of the oil. A strong wind, especially during the night of 6 January caused oil to enter into the canal, which polluted several other parts of the port of Amsterdam.

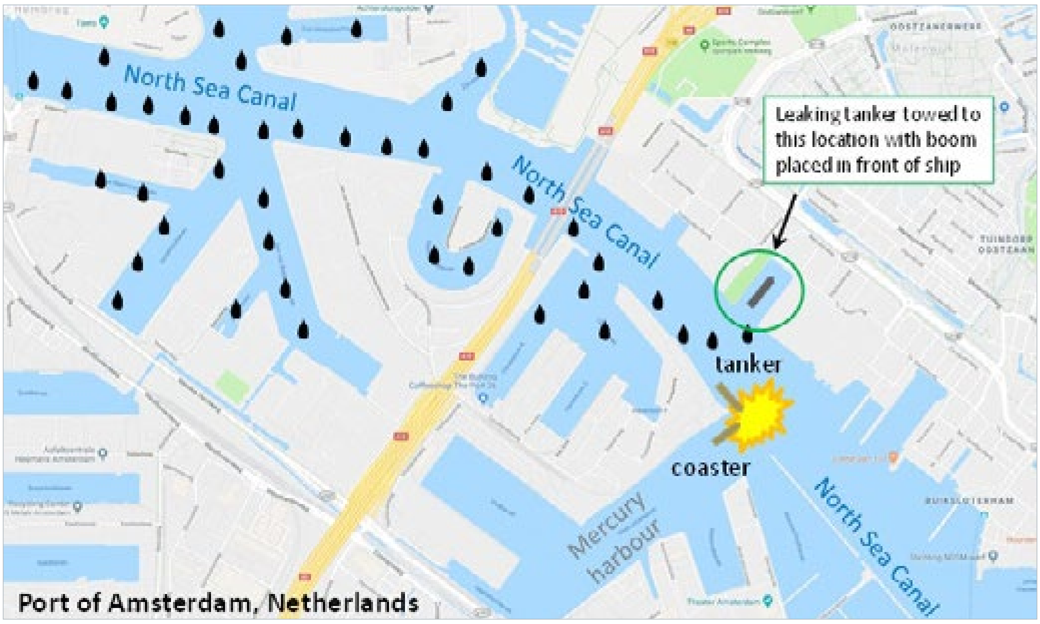


Figure 21: Map of the inland harbour of Amsterdam, Netherlands, at North Sea Canal. The initially polluted area is indicated with oil droplets.

Sample taking and case work

Between 13 January and 16 March, fifteen samples were collected. Samples were taken on board of the tanker (tanks and deck), from the surface water at six different locations, the shore, the hull of a ship, another ship, a landing stage and fishing nets. A picture of the map of the port of Amsterdam, with all sample locations indicated, is given in Figure 22. Samples were collected with ETFE nets. Fingerprinting of the oil provided evidence that all samples, except from one surface water sample, were matching each other.

14.2 AMSTERDAM, THE NETHERLANDS (CONT.)

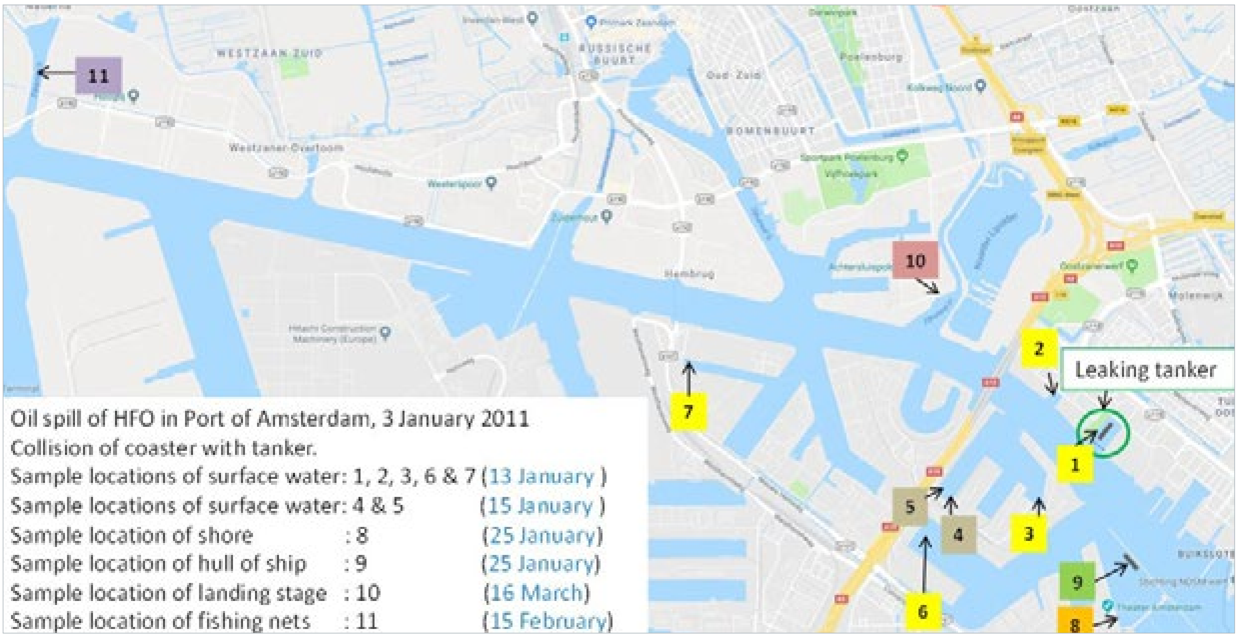


Figure 22: Locations that have been sampled for oil spills after the collision of a coaster with a tanker in the port of Amsterdam on 3 January 2011

Lesson learned

For this case, it was important to collect samples during the time frame the spill continued.

14.3 TELEMARK, NORWAY (MS FULL CITY)

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING INSHORE; SAMPLING BEACHES AND ROCKY SHORES; SAMPLING ON BOARD SHIP

Incident description

On 31 July 2009, the Panamanian-registered MS “Full City” ran aground at Såstein, southwest of Langesund in Norway (Figure 23). At the time, the ship was carrying 1,154 m3 of heavy fuel oil (IFO 180). Some fuel tanks were damaged during the grounding, and it was later estimated that 293 m3 of heavy oil leaked out. Initially the oil spread northeast, with slicks being observed in the bays at Langesund. Later, the oil drifted even further northeast to Vestfold, and then southwest to the southern part of Norway. See the map below for indication of polluted area. The oil followed the current southwards. The oil-exposed area is full of islands and different shoreline types. The oil polluted 200 locations and in total polluted 75 km of shoreline (Figure 24).

14.3 TELEMARK, NORWAY (MS FULL CITY) (CONT.)

Figure 23: The Full City grounding. The incident happened in late July 2009. Credit: Norwegian Coastal Administration

Sample taking and case work

The incident led to an extensive clean-up operation that lasted close to 1½ years. The incident was given prominent coverage by the media, and garnered a lot of public interest due to the contamination of popular beaches in the area.

The incident happened at high tide and led to oil contamination of a broad coastal belt. Extensive sampling was needed to verify that the oil was from the vessel Full City. This was important to secure evidence and further needed in the claim management process. Fingerprint analysis was used to secure evidence.

Sampling was performed at different places, especially at the most southern oil-contaminated sites. Samples were taken from the different tanks of the vessel. A positive match for the samples taken along the coast and the oil from the vessel was found. In 2010 the clean-up work started again in the springtime. When oil was found in new areas and sampling was needed.

The samples were analyzed according to CEN/TR 15522, GC-FID and GC-MS analyses were performed. In total, about 130 samples were taken. Both the police and the Norwegian Coastal Administration took oil samples. However, it was not necessary to analyze all the samples. A clear match was obtained for most of the samples. The analysis of the samples taken very close to the vessel and the ones furthest away from the spill indicated clearly the same source.

This case study shows that sampling is needed for several purposes, e.g, for compiling an oil budget and investigating which shoreline-cleaning agents are efficient as the oil weathered heavily on shore.

14.3 TELEMARK, NORWAY (MS FULL CITY) (CONT.)

Figure 24: The red line indicates coastline that was exposed to oil due to the Full City incident.

The following sampling was undertaken (Figure 25):

- On the shoreline,
- Background samples from the closest non-contaminated areas to the incident
- On open sea
- Of the different tanks from the vessel, the vessel had several mixtures of heavy fuel oils
- Of the weathered oil inshore to check the efficiency of different cleaning agents. Analyses were performed e.g. to check viscosity.
- Of collected oil and oil debris to estimate the oil recovery budget (i.e. data (volume) on where the oil has been recovered – see Figure 26), which is based on pure oil. Analyses were performed to check water content.

In such incidents, all sampling sites should preferably be included in an operation map. For the Full City incident, an Excel sheet was used to keep track of sampling sites. Sampling of the oil over a time-period, throughout the whole operation, also gave information of how the oil weathered. In addition, sampling also gave useful information concerning lighter components in the oil that is of special concern when handling oil due to their higher environmental availability and toxicity.

14.3 TELEMARK, NORWAY (MS FULL CITY) (CONT.)

Figure 25: Pictures of sampling and sampling equipment used for securing of evidence for oil identification. Credits: Norwegian Coastal Administration

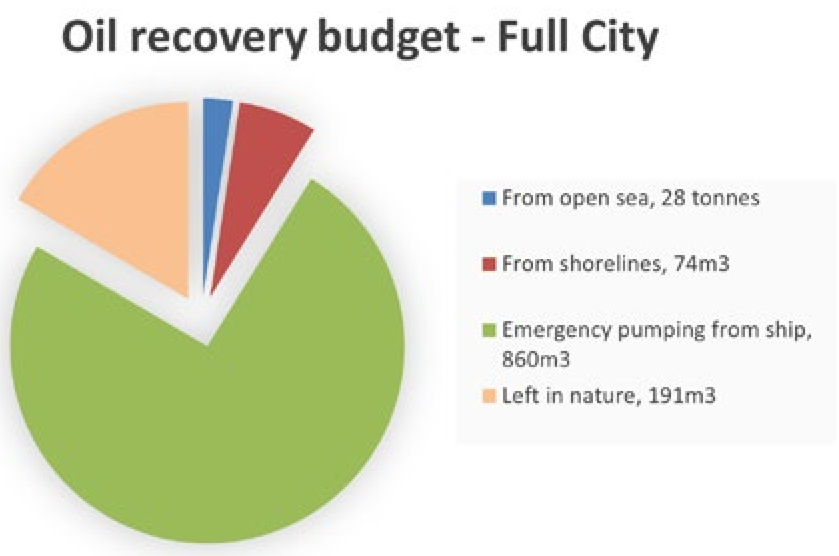


Figure 26: Oil recovery budget of the incident

14.3 TELEMARK, NORWAY (MS FULL CITY) (CONT.)

Lessons learned – Full City incident:

1. Sampling verified the source to the spill, which is very important concerning securing evidence and claim management. Sampling of spill sites far from the incident is also needed to verify the source.
2. Sampling gave input to operative handling of the spill, especially as the oil weathered and shoreline cleaning became more challenging.
3. Sampling sites should preferably be included in an operation map.

14.4 SPANISH EEZ NEAR THE CANARY ISLANDS

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING FROM MARITIME UNITS; SAMPLING ON BOARD SHIP

Incident description

During the night of 14th May 2012, the surveillance plane SASEMAR 103, at about 40 miles north of the Canary Islands, observed a container ship. The ship was on its way to Algeciras. A slick of 34.8 km^2 was observed, connecting to the stern of the vessel.

The following morning, a patrol boat was sent to collect samples from the oil slick. Using polyethylene cornets and ETFE nets, different samples were taken of the oil layer floating on the water.

Later, on the 29th of May, when the suspicious vessel docked at Algeciras harbor, it was inspected by the port authorities. Although the vessel denied the discharge when interrogated, oil samples were collected from three different tanks onboard with polyethylene cornets (sludge tank, bilge tank and main shaft well) for comparison to the slick samples.

Sample taking and case work

Analysis identified the spill as a mixture of fuel oil and lubricating oil consistent with a discharge of bilge oil. The comparison of the analytical data showed a positive match between the spill samples with that of the vessel's sludge tank despite some slight differences attributed to weathering of the released oil.

Lesson learned

The crucial point in this case was that adequate sampling of all relevant tanks was performed.

14.5 SPANISH EEZ OFF VALENCIA

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING BEACHED AND ROCKY SHORES; SAMPLING FROM MARITIME UNITS; SAMPLING ON BOARD SHIP

Incident description

On 22 May 2013, the captain of a container carrier reported an operational incident to the maritime authorities resulting in the accidental spill of fuel oil 40 miles offshore of Valencia. Spill samples were immediately collected in the vicinity of the vessel and also, on the day of the accident, from the suspected tank. However, some days later (31 May to 15 June 2013), new oil slicks appeared on the sea.

Almost one month later (6 to 14 July 2013), oil residues had spread widely on Valencia and Castellón beaches and coastal zones. The main interest of the analysis was to identify the source of all these samples, particularly, if they were related to the aforementioned spill.

Sample taking and case work

Sampling was carried out with polyethylene cornets and ETFE nets, depending on the situation. A map of the affected coast, with all sampling locations indicated, is given in Figure 27. In accord with the information provided by the captain of the container, the spill samples were identified as fuel oil. Moreover, samples collected on beaches and coastal zones allowed the responders to conclude a match to the vessel's fuel oil despite the spilled oil having been subjected to weathering.

Figure 27: Sampling sites at the coast off Valencia. Credits: CSIC, Spain

14.5 SPANISH EEZ OFF VALENCIA (CONT.)

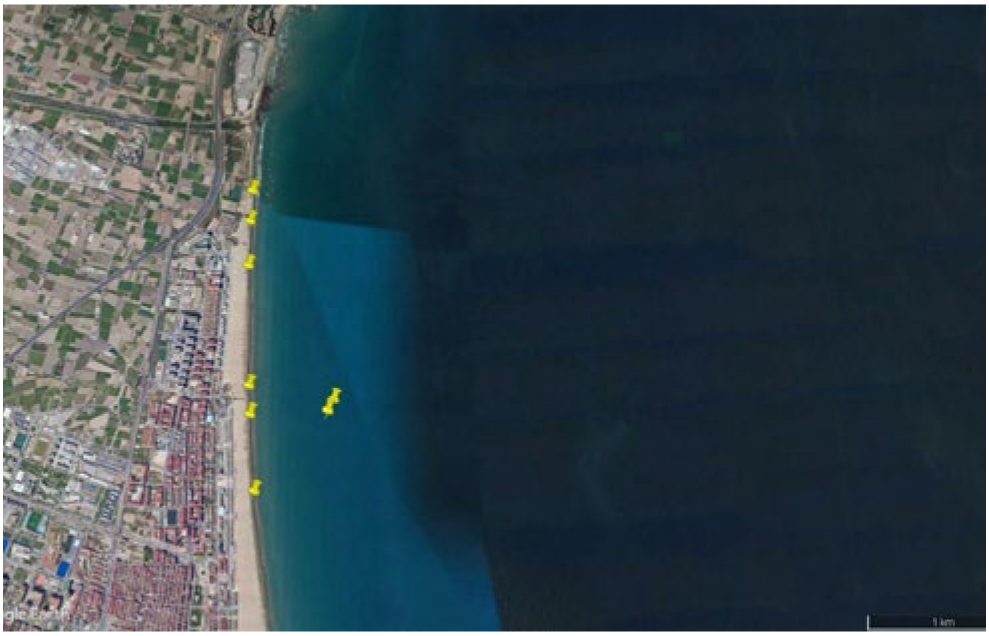


Figure 27 cont: Sampling sites at the coast off Valencia. Credits: CSIC, Spain

Lesson learned

The true extent of an oil spill might not at once be apparent and even oil appearing later might be linked to an earlier spill event.

14.6 SPANISH EEZ NEAR THE CANARY ISLANDS (I)

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING FROM MARITIME UNITS

Incident description

In 2015 an alert was reported through EMSA CleanSeaNet to the Spanish authorities. A ship was suspected of a possible illegal oil discharge whilst sailing through the Spanish EEZ near the Canary Islands. A plane was sent to the area (Figure 28). Through visual observation and the use of sensors, the presence of petroleum oil was confirmed and a connection between the discharge and the ship was established. Under Spanish Law and precedents, these facts are the only evidence needed to initiate a procedure in a case of illegal discharge of petroleum oil.

Sample taking and case work

A SASEMAR ship was sent to the area to take samples of the discharge. Samples were analysed at the laboratory confirming that the spill consisted of petroleum oil. A procedure was initiated and the defendant claimed that the discharge was not from their ship, but from a sunken ship in the area that was leaking fuel. Samples were taken from the discharge of the sunken ship and compared to the samples taken in the ships' wake, showing that they were from a completely different type of petroleum oil, establishing the culprit.

14.6 SPANISH EEZ NEAR THE CANARY ISLANDS (I) (CONT.)

Figure 28: Aerial photos from the spill (above) and photo from sample taking (below). Credits: SASEMAR, Spain

Lesson learned

Analysis of oil samples helps to establish the link between spills and disputed sources.

14.7 SPANISH EEZ NEAR THE CANARY ISLANDS (II)

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING BEACHED AND ROCKY SHORES; SAMPLING FROM MARITIME UNITS

Incident description

On 11 April 2015, the fishing vessel Oleg Naydenov, sailing under Russian flag, was ready to leave the Port of Las Palmas (Gran Canaria Island) when a fire was declared on board. Due to the risk posed by the ship's fire for the population and the environment it was towed offshore. The ship ultimately sank in the morning of 15 April 2015, 15 miles south of the island, at a depth of 2,700 meters. At the time, it carried 1,400 tons of fuel, 30 tons of diesel and 70 tons of lubricating oil. The subsequent aerial surveillance of the area quickly identified a number of oil slicks drifting to the south-west.

14.7 SPANISH EEZ NEAR THE CANARY ISLANDS (II) (CONT.)

Sample taking and case work

The incident resulted in 30 sets of samples, which were collected with polyethylene cornets or ETFE nets between 15 April and 15 May 2015. They were collected from the open sea and from the shoreline in areas close to the incident to obtain background samples. A map of the area, with all sampling locations indicated, is given in Figure 29.

One of the main aims of the monitoring was to obtain a comprehensive picture of the fate of the spilled oil in the marine environment and, indirectly, to identify any possible illegal discharges in the area after the spill. However, the problem in this case was the mixing of the different petroleum oil products carried by the sunken vessel. Analysis identified the samples as heavy fuel oil containing different amounts of lubricating oil. Despite the different grades of mixing, analysis confirmed a match between the fuel oil in all samples.

Varying features are common in spills of waste oils (e.g., bilge residues, sludge, slops) where different mixtures can be found in different tanks and samples. These differences have to be considered in the assessment of analytical analysis and the applied analytical methods must make such assessments possible.

Figure 29: Sampling sites around Gran Canaria Island. Credits: CSIC, Spain

Lesson learned

Monitoring the discharge of a sunken ship closely allows to distinguish these residues from other illegal/accidental discharges occurring in the area after the spill.

14.8 SW COAST OF THE IBERIAN PENINSULA

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING FROM MARITIME UNITS SAMPLING FROM HELICOPTERS

Incident description

In November 2019, an alert was reported through EMSA CleanSeaNet (CSN) to the Spanish authorities (Figure 30). Some possible spills were detected in the SW coast of the Iberian Peninsula and were investigated. The spills supposedly originated from a ship that was in the area sailing with a westerly course. It was a chemical tanker that, when contacted, stated that they were cleaning a vegetable oil – an operation that MARPOL allows – as a cargo residue from the last voyage. The satellite image showed that the appearance of the slicks was more prone to be of mineral than vegetable origin. The pollution lasted more than 12 hours at sea and two consecutive CSN alert reports were sent to the Spanish Authorities regarding this case. A helicopter was mobilized for surveillance and monitoring, and took samples from the polluted area (Figure 31, Figure 32, Figure 33).

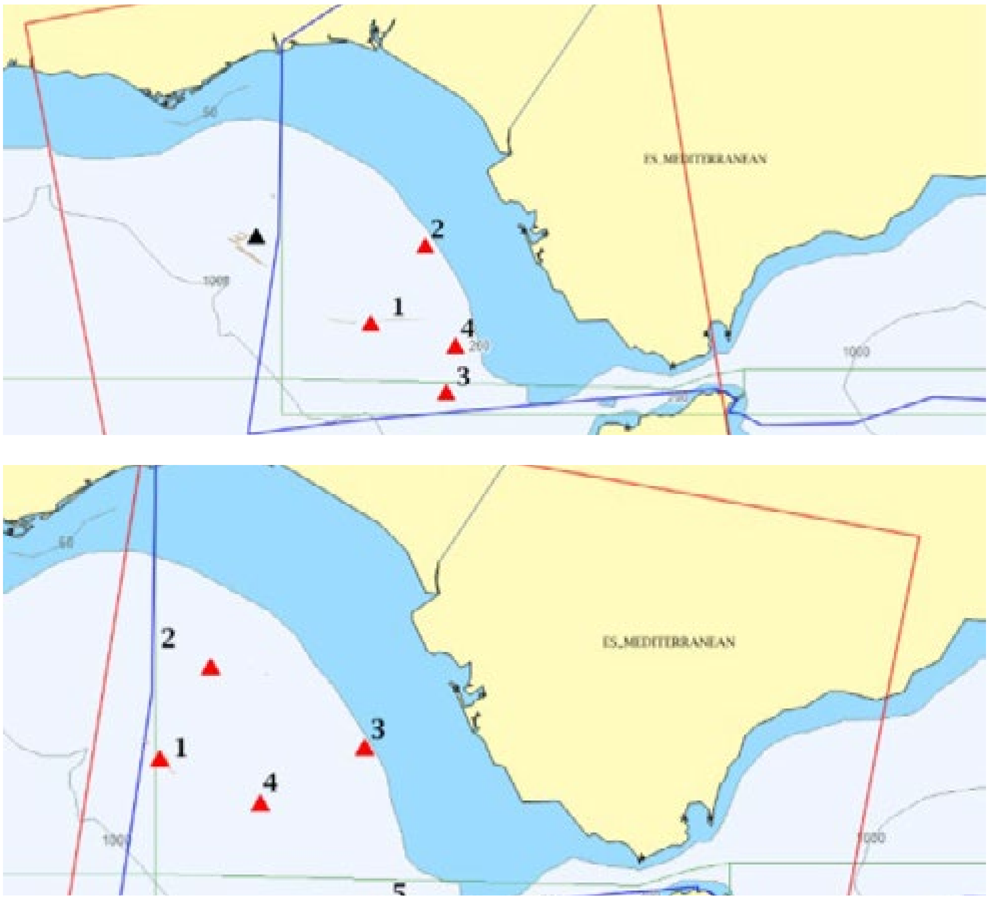


Figure 30: CleanSeaNet Alert Report from 2019-11-06 18:26:03 UTC (above) and from 2019-11-07 06:27:27 (below)

14.8 SW COAST OF THE IBERIAN PENINSULA (CONT.)

Sample taking and case work

Three samples were obtained from different parts of the slicks. Analyses showed that one of the samples was of a vegetable oil, another was a mix of a vegetable oil and a petroleum oil and the last was clearly petroleum oil.

Lesson learned

This shows that in some cases, the illegal discharge can be hidden with a legal one and, in this case, sampling at different places to know the type of oil in the water was the key point to know if there was a breach of MARPOL regulations.

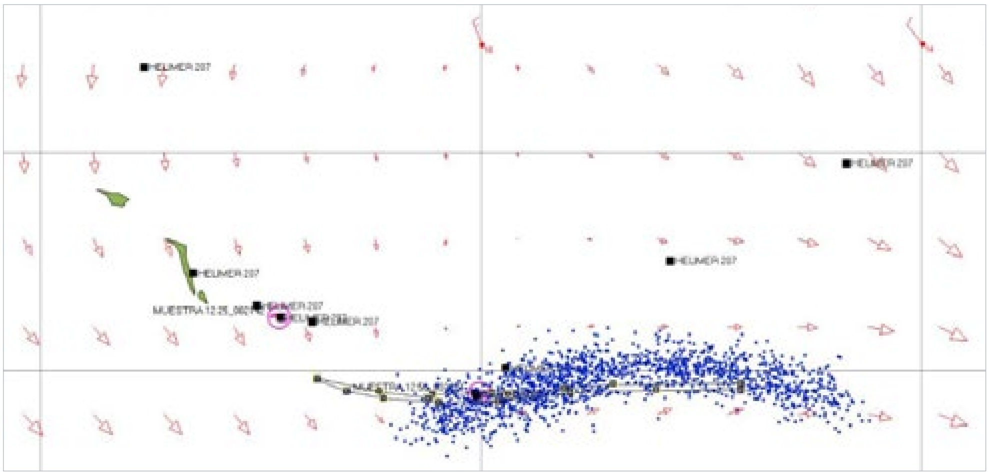


Figure 31: Helicopter sampling points (pink circles)

Figure 32: Photos from sampling point 1 (above); sampling point 2 (below). Credits: SASEMAR, Spain

14.8 SW COAST OF THE IBERIAN PENINSULA (CONT.)

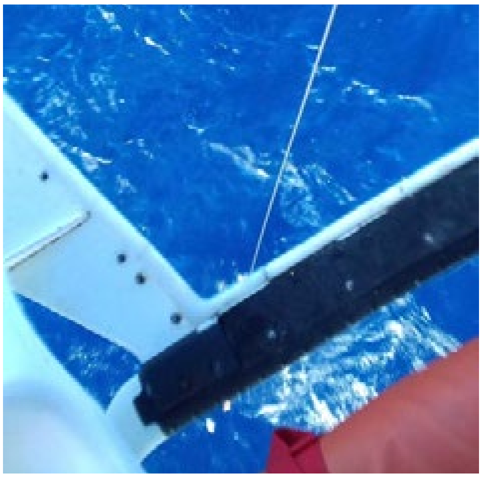


Figure 33: Photo from sampling point 3 (left) and the analytical results for each sample (right): Credits: SASEMAR, Spain

14.9 DANISH EEZ

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING FROM MARITIME UNITS, SAMPLING INSHORE, SAMPLING BEACHES AND ROCKY SHORES, SAMPLING HARBOURS

Incident description

On the 4th of January 2020, the Danish Maritime Assistance Service was notified of a spill that had occurred during a loading operation from a tank on shore to a berthed ship.

During the operation, the wind picked up fast and the ship's mooring lines broke. The captain started the ship's engine and cut the remaining mooring lines in order to not drift into shallow waters.

The loading arm broke and the loading hose, still full of Vacuum gas oil (VGO), ruptured and the full volume of 30 m^3 VGO was spilled into the water.

Immediately after the spill, the oil was visible 25-30 meters from the shoreline.

Some oil reached the shoreline on the first day, and 20 tons of sand and oil mixture were removed from the nearby beaches.

On the 5th, the wind shifted and carried the oil spill away from the shoreline, along with some yellow/greenish lumps found along the polluted coastline (Figure 34). A Danish oil spill recovery vessel (OSRV) encountered a large oil concentration (Figure 35) that had moved along the coast. Most of the visible oil was too viscous to recover; however, they encountered a patch with yellow lumps (Figure 36) and started to recover what was possible.

14.9 DANISH EEZ (CONT.)

Figure 34: Small yellow lumps on a nearby beach. Credits: Danish Defence

Several patches of oil were observed during the 5th and 6th but due to the low viscosity of VGO, only a small amount of the spilled oil was recovered. On the 7th, the VGO was no longer visible on the surface of the sea.

Case work and sampling strategies

There was no sampling manager to coordinate the sampling response but the Danish contingency plan contains guidelines for sampling in order to secure sufficient numbers of samples.

On land, samples were taken from the tank, loading arm and hose.

At sea, samples were taken from various patches of oil by the OSRV, as well as from recovered oil and from equipment used to recover spilled oil (Figure 34).

Two different background samples were taken to establish a baseline of oil content of the water.

Sampling was carried out with glass containers and PTFE nets.

A total of 16 samples were taken by the Danish Navy and 2 by the municipal authorities.

14.9 DANISH EEZ (CONT.)

Figure 35: Danish OSRV transiting through windrows of VGO. Credits: Danish Defence

Figure 36: Yellow lumps in the sweep of Danish OSRV. Credits: Danish Defence

Lesson learned:

Depending on weather conditions, oil spills can drift wide distances in a short time and pollute new (coastal) areas.

14.10 BELGIAN EEZ (M/V FLINTERSTAR)

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING FROM MARITIME UNITS, SAMPLING INSHORE, SAMPLING BEACHES AND ROCKY SHORES.

Incident description

On the 6th October 2015, m/v Flinterstar sunk in the Belgian territorial waters after a collision with the LNG tanker Al Oraiq (Figure 37, Figure 38, Figure 39, Figure 41). Oil was leaking from the m/v Flinterstar (Fuel oil on board m/v Flinterstar: 428t HFO + 135t MDO). Response operations started immediately in order to combat the oil spill and remove the oil remaining in the ship's tanks. The oil removal operation was completed on 2nd November 2015.

BELGIAN EEZ (M/V FLINTERSTAR) (CONT.)

The following figures show the spill site, the sunking ship and provide information about the response activities (drift modeling - see figure 40) and the sampling campaign carried out (see figure 42, figure 43, Table 3 and Table 4).

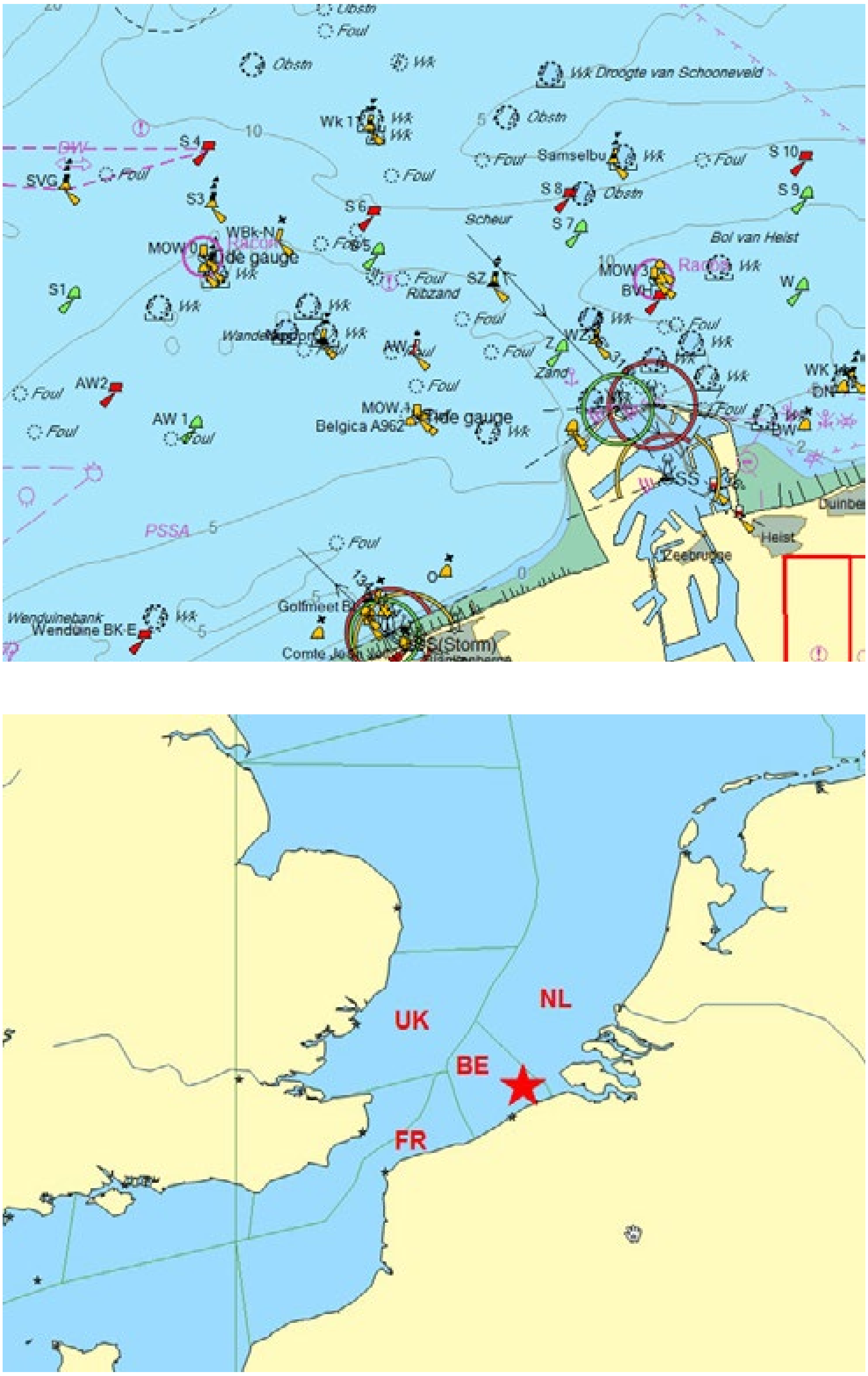


Figure 37: Location of the accident

BELGIAN EEZ (M/V FLINTERSTAR) (CONT.)

Figure 38: Situation a few hours after the collision. Credits: E. Donnay DG Environment Belgium

Figure 39: Situation after the ship completely sank. Credits: E. Donnay DG Environment Belgium

BELGIAN EEZ (M/V FLINTERSTAR) (CONT.)

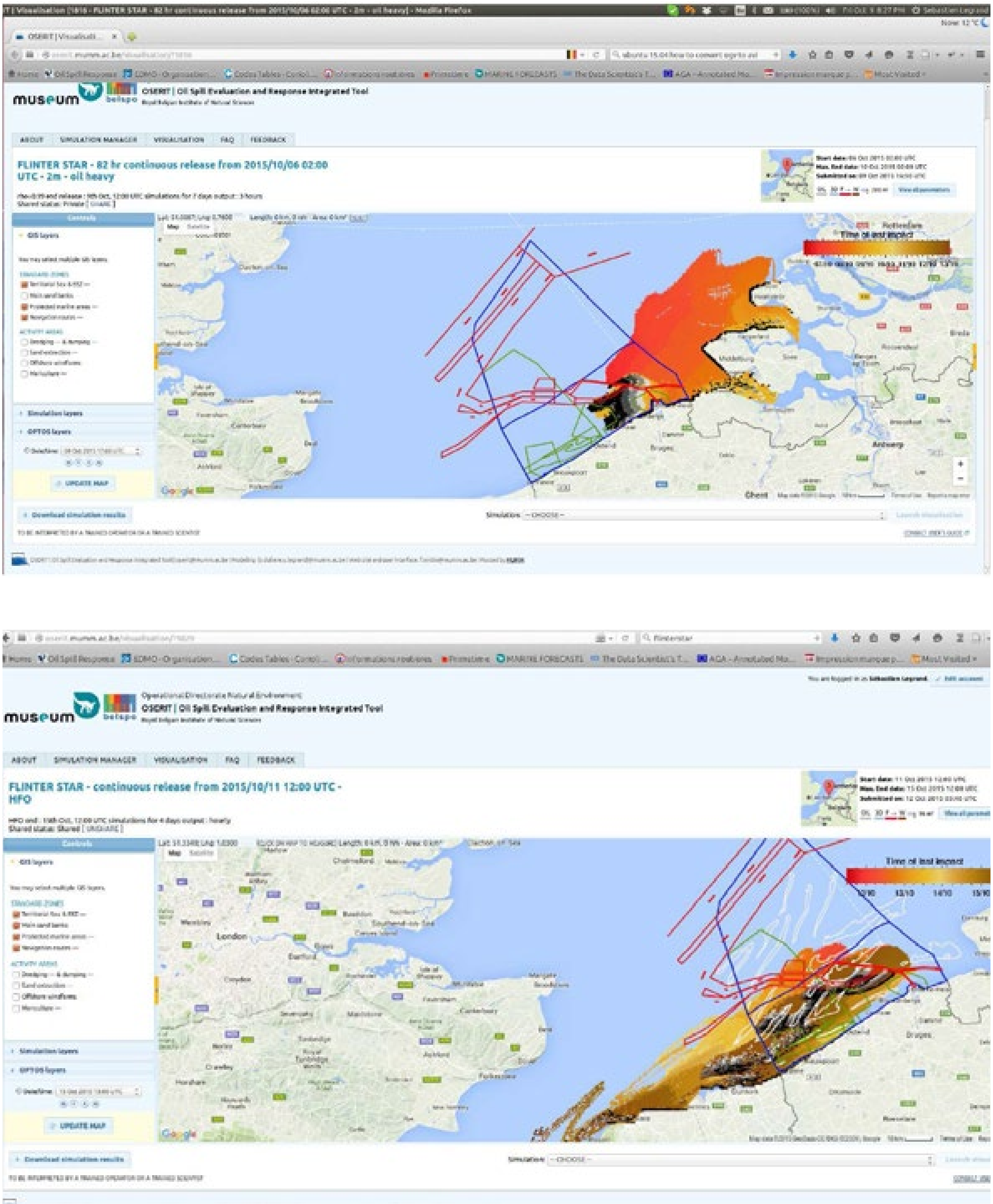


Figure 40: Oil drift simulations (worst case scenario)

BELGIAN EEZ (M/V FLINTERSTAR) (CONT.)

Figure 41: Oil pollution from M/v Flinterstar and a sampling team in action. Credits: E. Donnay DG Environment Belgium

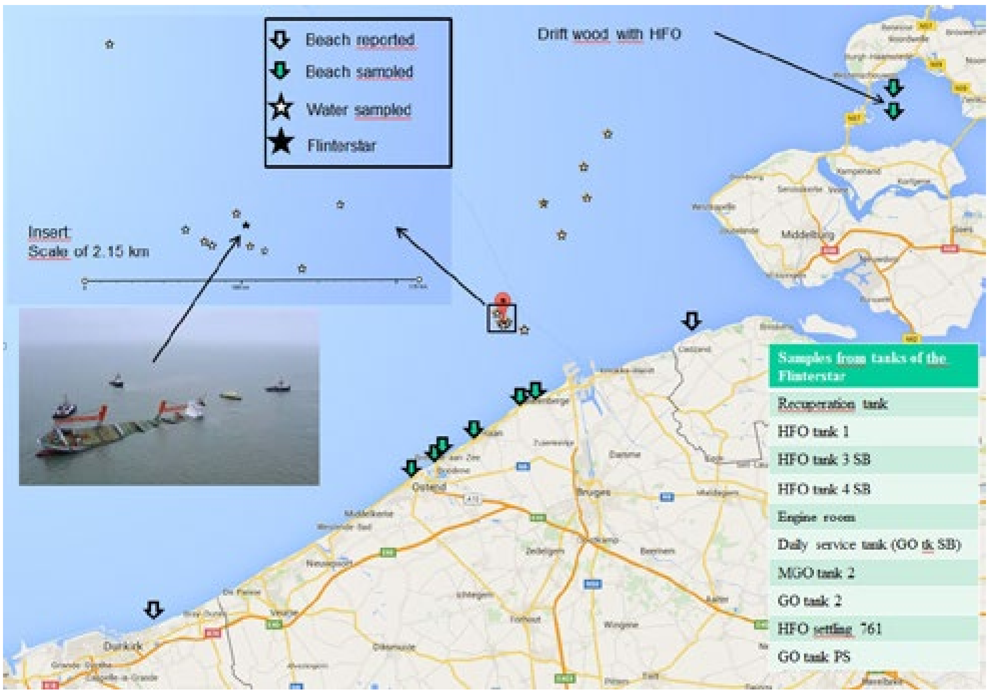


Figure 42: Sampling locations in Belgium, the Netherlands and France

BELGIAN EEZ (M/V FLINTERSTAR) (CONT.)

Figure 43: Samples from the sampling campaign. Credits: Rijkswaterstaat, The Netherlands

Table 3: Overview of the oil samples taken during the Flinterstar incident

31 from pollution at sea (as close to the wreck as possible, more or less daily) 6 from oil on the shore 1 from an oiled seabird 10 reference samples from the tanks of the Flinterstar (taken by salvors) Total: 48 samples of which 21 were analysed
Remarks: *All samples normally taken in tripicate (if not divided in 3 in the lab)* *All samples systematically registered by police* *5 samples were analysed by Cedre (FR). NL used the results in COSI-Web for comparison*

BELGIAN EEZ (M/V FLINTERSTAR) (CONT.)

Table 4: Conclusions from the oil identification analysis

Samples sorted on sampling date			Conclusion
Location	Flinterstar	collision on 06/10/2015	
NI-1-3329.1	Water sample Oosterschelde (drift wood)	09/10/2015 HFO	Match
NI-1-3329.2	Water sample Oosterschelde (drift wood)	09/10/2015 HFO	Match
BE-1-6042.121	Beach sample Ostende Oosteroever	15/10/2015 HFO	Match
BE-1-6042.141	Beach sample OstendeRaversijde	16/10/2015 HFO	Match
BE-1-6042.291	Beach sample Ostende Oosteroever	18/10/2015 HFO	Match
BE-1-6042.241	Beach sample Blankenberge	21/10/2015 HFO	Match
BE-1-6042.251	Beach sample Blankenberge thv zeedijk 130	21/10/2015 HFO	Match
BE-1-6042.471	Feather auk 3298 De Haan	29/10/2015 HFO	Non-match
France	Beach of commune de Leffrinckoucke	14/10/2015	Match
Samples NL	2		
Samples BE	6		
Samples FR	1		

Lessons learned:

1. A clear sampling strategy and using standardized procedures like EN 15522 (2023) are key elements for establishing the causal link between the observed pollution and the source of the pollution (Flinterstar).
2. COSIWeb proved to be useful for sharing information about samples and results of analyses between the affected countries.

14.11 THE CARIBBEAN

CORRESPONDING OIL SAMPLING SCENARIOS: SAMPLING INSHORE, SAMPLING BEACHES AND ROCKY SHORES

Incident description:

On 23 April 2017 at 2.45 pm, Tank 70 at Petrotrin oil refinery in Pointe-a-Pierre, Trinidad, sprung a leak at its base. At the time, the tank with the capacity of 24000 m^3 contained 2000 m^3 of HFO of which 48 m^3 were spilled.

The incident was not reported to surrounding nations.

Between 13 May 2017 and 15 June 2017, oil pollution was detected in the national EEZs of Bonaire, Aruba, Curacao and Venezuela (Figure 44).

14.11 THE CARIBBEAN (CONT.)

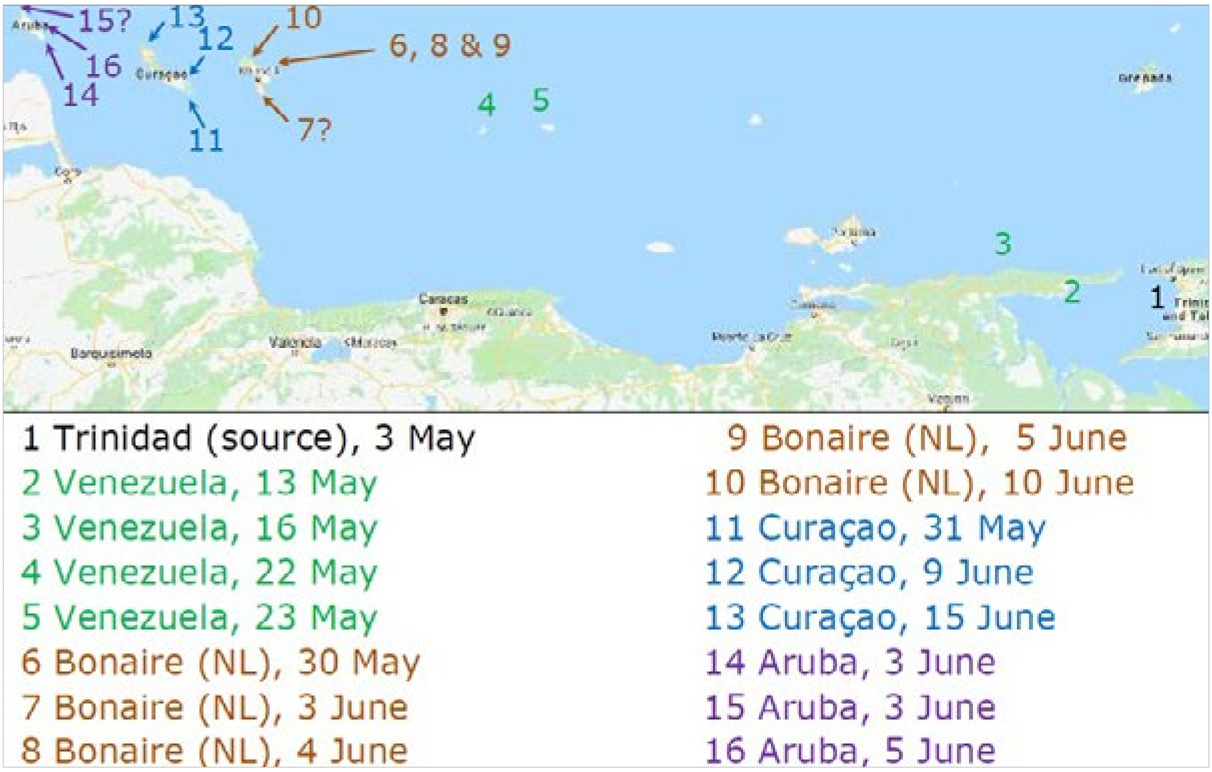


Figure 44: Sampling sites of oil spill samples (2-16) and point of origin (1)

Sample taking and case work:

56 samples were collected in total. Coordination of sample exchange was done by RAC/REMPEITC-Caribe, Willemstad, Curacao. Samples were sent to the Netherlands (18 samples, examples see figure 45), Venezuela (11 samples) and Trinidad (27) for analysis.

Results of the investigation showed that the pollution consists of petroleum oil. The pollution was further identified as HFO.

All the analysed samples from Bonaire, Aruba, Curacao and Venezuela showed a match with the analysed sample from Tank 70 of Petrotrin at Pointe-a-Pierre, Trinidad.

Lessons learned:

The challenge in this case was identifying the source using a reasonable amount of analysed samples.

In every oil spill case, it is important to make sure to collect enough samples from all relevant spots, covering all distinct geographical locations over the entire time span during which the spill occurs. It is crucial to keep in mind that the time span for proper sample collection might be short.

All collected samples should be stored in such a way that analysis is possible - immediately or in the future. Agreeing with involved parties (prosecutor) beforehand which samples should be analysed preferentially to solve the case will quicken the process of providing relevant analytical data while help limiting laboratory costs. In case the first batch of analysed samples does not yield clear results, analysis of additional samples can be considered.

14.11 THE CARIBBEAN (CONT.)

Figure 45: Examples of samples from Bonaire received by the Netherlands. Credits: Rijkswaterstaat, The Netherlands

14.12 NORTHERN FINISTÈRE, FRANCE

On 16th November 2019, the MRCC Corsen received reports of potential pollution in northern Finistère. The same day, oiled birds were found on the shoreline between the beaches of Saint-Samson and Saint-Jean-du-Doigt.

As soon as this information was confirmed, satellite, aerial, shipborne and onshore surveys were launched to detect the pollution. Despite the resources deployed, no pollution was detected.

However around forty birds, some bearing traces of oil, were found on the beaches of northern Finistère and Côtes d'Armor and were taken to the LPO Ile Grande rehabilitation centre. At the request of the Maritime Prefecture for the Atlantic, Cedre analysed samples taken from several oiled birds. The oil analysed showed major similarities with the heavy fuel oil of the Tanio, an oil tanker that sank on 7 March 1980 off the northern coast of Finistère (western Brittany).

On 21st and 22nd November 2019, the French Navy sent the Pégase (a tripartite minehunter) to determine the precise location of the wreck of the Tanio, which lies off the coast of Batz Island at a depth of 80-90 metres. No pollution was detected by the Pégase around the wreck.

Surveys of the northern Finistère shoreline were reinforced and maritime surveillance continued.

14.12 NORTHERN FINISTÈRE, FRANCE (CONT.)

Further investigations are scheduled to be conducted to assess the state of the wreck and to detect any possible leaks of oil remaining in the bow section of the Tanio.

In early December 2019, the French Navy deployed a ROV (Remote Operated Vehicle). After several investigation dives, a few minor oil leaks were detected. These leaks were coming from two drilled holes fitted with valves, due to operations conducted in 1980 to pump out part of the Tanio's cargo. The valves had been torn out of the two holes.

From 5th to 8th September 2020, the leaks were plugged.

In late 2020, oiled birds were found on the shores of Finistère. At the request of the Maritime Prefecture for the Atlantic, Cedre conducted tests and, as in November 2019, the oil analysed showed major similarities with the heavy fuel oil of the Tanio. On 6th and 7th January 2021, an operation was conducted to assess the state of the wreck. This new investigation showed that 3 of the 10 plates fitted in September 2020 had been torn off by fishing gear and that one of the openings is leaking oil.

The feasibility of subsequent intervention is currently being studied and the maritime authorities are on high vigilance for the monitoring of this wreck.

[From: https://wwz.cedre.fr/en/Resources/Spills/Spills/Tanio/40-years-on; reprinted with the kind permission of CEDRE, France. For more information about the Tanio incident, see: https://wwz.cedre.fr/en/Resources/Spills/Spills/Tanio.]

Lesson learned:

Even old wrecks need continued monitoring and regular surveillance since their physical condition can deteriorate or they can be damaged by outer forces (e.g. fishing gear, storms).

CHAPTER 15

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APPENDIX 1

GLOSSARY OF TERMS

Background sample	Sample of water from the vicinity of an oil spill but unaffected by it ("clean water sample").
Blank	Used in quality control to determine background contamination of either spaces (e.g. sampling site = field blank), materials (e.g. laboratory equipment, detergents, solvents) or whole procedures (e.g. analytical procedures). Non-natural samples that aim to monitor if contamination of the sample material occurred during sampling, handling and analysis. Such contamination can occur in the field (e.g. sampling site = field blank), through materials used during sampling or analysis (e.g. sampling and laboratory equipment, detergents, solvents) or through whole procedures (e.g. analytical procedures).
CEN	European Committee for Standardisation (French: Comité Européen de Normalisation).
Chain of custody documentation	Chronological evidence defining the history of an item, such as a sample, and identifying an individual responsible for custody of the item at each point in time. (according to ISO 16165)
CleanSeaNet	European satellite-based oil spill monitoring and vessel detection service, set up and operated by EMSA.
Clearwater sample	Sometimes used as description of a background sample in aquatic environments; use not recommended.
Coastal state	Coastal States are universally understood to be States with a sea-coastline. A coastal State's jurisdiction relates to its own maritime zones, and encompasses the resources and activities therein as well as external impacts on them[25].
COSIWeb	Computerised oil spill identification, web-based.
Custody	Physical possession or control. A sample is under custody if it is in an individual's possession or under the immediate control of an individual to prevent alternation of characteristics. (according to ISO 16165).
ECA	Emission Control Area.

[25] https://www.oxfordhandbooks.com/view/10.1093/law/9780198715481.001.0001/oxfordhb-9780198715481-e-

EMSA	European Maritime Safety Agency.
EN	European norm.
ETFE	Ethylene tetrafluoroethylene.
FAMEs	Fatty acid methyl esters.
Field blank	See Blank.
Flag state	Country of registry of a sea going vessel.[26]
GNSS	Global Navigation Satellite System.
GPS	Global Positioning System.
HELCOM	Baltic Marine Environment Protection Commission (Helsinki Commission).
HSE	Health, Safety & Environment.
HVO	Hydrotreated Vegetable Oil.
IBTS	Integrated Bilge Water Treatment System.
IMO	International Maritime Organization.
Interpol	International Criminal Police Organization.

[26] https://stats.oecd.org/glossary/detail.asp?ID=4236; https://stats.oecd.org/glossary/detail.asp?ID=1001

IOPP Certificate	International Oil Pollution Prevention Certificate.
ITOPF	International Tanker Owners Pollution Federation
Legal sampling	Sampling which will result in the subsequent analysis being used as evidence in a court case. Standardised procedures have to be followed, documentation must be complete and the chain of custody is needed to proof the integrity of the samples. By default, sampling in an oil spill event is always preservation of evidence and therefore legal sampling.
MARPOL	International Convention for the Prevention of Pollution from Ships, developed by the IMO.
Matrix	All compounds of a sample besides oil or the environmental compartment the oil sample was taken from (e.g. water).
MEPC	Marine Environment Protection Committee (IMO).
ODMS	Oil Discharge Monitoring System.
Oil	Petroleum in any form including crude oil, fuel oil, sludge, oil refused and refined products (other than those petrochemicals which are subject to the provisions of Annex II of the MARPOL Convention) and, without limiting the generality of the foregoing, includes the substances listed in appendix I to this Annex 1 (Definition of oil from MARPOL convention Annex 1).
ORB	Oil record book.
OSINet	Oil spill identification network of experts within the Bonn-Agreement.
OWS	Oily Water Separator.
Paris MoU (also: PMoU)	Paris Memorandum of Understanding.
PE	Polyethylene.

Petroleum oil	Material consisting of, or derived from, a mixture of liquid or semi-solid organic compounds, principally hydrocarbons (according to ISO 16165).
PMoU (also: Paris MoU)	Paris Memorandum of Understanding.
Port state control	Competence of States to exercise jurisdiction over foreign vessels within their ports, namely to unheraldedly inspect foreign-flagged ships in national ports to verify compliance with international regulations.
PPE	Personal Protective Equipment
PSCO	Port State Control Officer, carries out port State control
PTFE	Polytetrafluoroethylene
ROUV	Remotely operated underwater vehicle
ROV	Remotely Operated Vehicle
RPAS	Remotely Piloted Aircraft System
Scrape sample	Sample taken by collecting sample material from its place of origin with help of a stiff sampling device (e.g. spatula)
Sheen	Very thin oil slicks with a silvery or rainbow-colored appearance and with a thickness of less than 0,001 mm (according to ISO 16165)
Source sample	Sample of the oil discharged by the pollution emitter (source); preferably collected directly at the source in close timely proximity to the discharge event to prevent contamination and degradation of the sample
SOx	Sulphur Oxides

Storage time	Amount of time from the moment the sample is taken until the sample is stabilized (by extraction) and/or analysed
Swab sample	See Swipe Sample
Swipe sample	Sample taken by swiping a flexible sampling device (e.g. ETFE net) over a hard surface (e.g. rock, engine etc.)
UAV	Unmanned Aerial Vehicle; known also as unmanned aircraft (UA) or unmanned aircraft system (UAS)
ULSFO	Ultra Low Sulphur Fuel Oil
UNCLOS	United Nations Convention on the Law of the Sea (also called the Law of the Sea Convention or the Law of the Sea Treaty)
VOCs	Volatile organic compounds
VGO	Vacuum gas oil
Wipe sample	See Swipe Sample

APPENDIX 2

SAMPLING DOCUMENTATION

EXAMPLES FROM DIFFERENT COUNTRIES

APPENDIX 2.1

Credits: SINTEF, Norway

Date	
Place	
Remarks , comments (e.g. sampling number):	
Name of the sample taker:	

SINTEF

Accompanying letter for a sample taken at sea / on shore for securing of evidence for oil spill identification :

Sender:	Location, date:
Contact person and phone no	
Recipient: SINTEF Materialer og kjemi **Delivery address:**	
Reference person in SINTEF:	

Sampling specifications:

Date:	Time:	Location	Name of sampling person:	Place for sticker (ID- no) from the safety bag)

Description of sample/sampling/type of pollution (e.g. thin film/thick emulsion/oil lump/ thickness, colour, high or low viscosity, where in the slick was the sample taken, sketch – next page). Sea / air temperature. If necessary, provide appendixes. Purpose of analysis. Remember pagination

Turn page

If relevant: Sketch the pollution. Mark where in the oil slick/pollution the sampling was done. Give approximate area of the oil slick on the sketch.

Checklist for sampling and shipping:

1. What does the pollution look like? Evaluate the situation and move on to the correct procedure in the collection of procedures:

   - *Sampling of thin oil film*
   - *Sampling of highly viscous oil, emulsions and oil lumps*
   - *Sampling on shore*
   - *Sampling of oil from contaminated sea birds and marine mammals*

2. Choose the right equipment from the suitcase. The white cardboard box will always be in use while sampling. Use of aluminum beaker and knife are described in the procedures.

3. Take the sample by following the correct procedure.

4. Pack the sample following the procedure for packing and shipment. Remember to fill in and pack the accompanying letter for each sample.

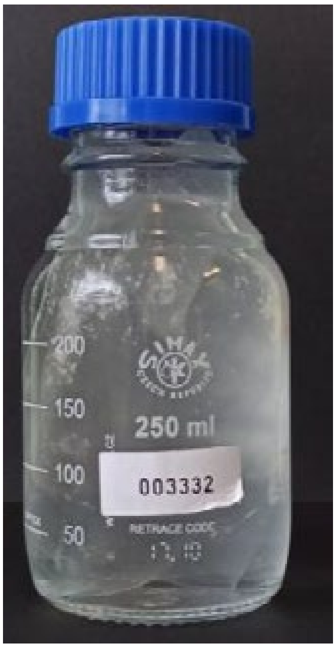
5. Fill inn the observation form .

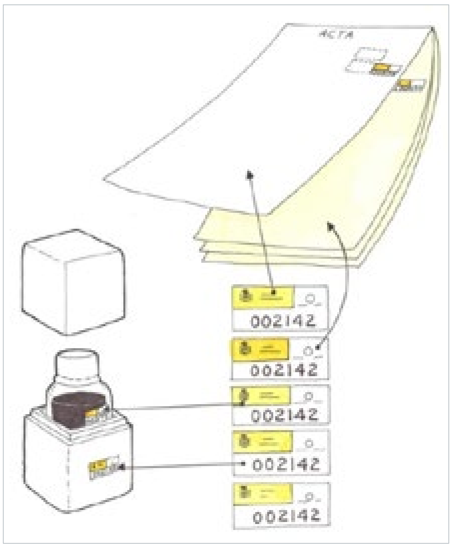
6. Notify SINTEF that samples are being shipped on telephone: ____ or email: ____

7. Make sure the samples are shipped using the most rapid method. If only 1-2 of the white boxes have been used, send the sample directly in the white box. If all three boxes are used and/or the content of the suitcase has been contaminated with oil, send the whole suitcase to SINTEF. The suitcase and all three boxes are pre marked with the correct address. A new clean suitcase or white box will immediately be sent in return from SINTEF to you.

APPENDIX 2.2

Credits: Spanish Maritime Safety and Rescue Agency (SASEMAR), Spain





Each evidence bag (already labelled with a unique ID number) comes with further six labels with the identical ID number. For each sample, ID labels are placed on the bottle, on the thermal insulated box, and in the chain of custody documents.

MINISTERIO DE FOMENTO

Salvamento Marítimo

SAMPLING INFORMATION AND CHAIN OF CUSTODY

Paste the label Nº ID bottle 1

With date............................ at .... hours, (name and surname of the sample taker) ...., with national identity card nº...and contact telephone·.............................. and as representative of............(name the organisation)..., has taken........... bottles from, (iidentification numbers are indicated at the top of this report) The details of the present sample collection are indicated below:

MOVILISED UNIT(8): ________ **Suspect source**(10): ________
CASE Nº(9): ________ **Pictures taken** (11): ☐Yes, ☐No

Place where the sample is taken
Location: ________
Geographical position (________ : ________
☐ **Sea**:
In port: ☐yes, ☐No Which? ________
☐surface ☐between waters ☐sea bottom
☐ **Land**:
☐beach ☐rocky shore ☐others ________
☐**Ship**: ________
☐Tank ________
☐Bilge ☐Others ________
☐ **Installation/Plant** (17): ________
☐Tank ________
☐pipeline ________ ☐Others ________

WEATHER CONDITIONS
Wind : ________
Temperature: Sea: ________ Ambient ________
Visibility : ________ **Sea state**): ________

SPILL DESCRIPTION
Presence of sheens: ☐yes, ☐No
Dimension :
Nature (hydrocarbon, jellyfish, etc)
Consistency: ☐Solid, ☐Liquid, ☐Semisolid ☐Other (specify):
Shape: ☐continuous surface, ☐strips, ☐cakes (>1 m^2), ☐tar balls; ☐Others (specify):

Appearance: ☐Sheen; ☐Rainbow; ☐Metallic; ☐discontinuous true oil colour; ☐continuous true oil colour; ☐Other (specify):

SAMPLE DESCRIPCTION
ODOUR: ________
Colour: ________
Presence of impurities : ☐yes, ☐No which?
____
Sample materials: ☐polietilene cone, ☐EFTE sheet, ☐Other (specify):
____

Further remarks: ________
____
____
____
____

And for the record, the present record is extended which, once read by all the appearing parties and finding it in accordance with its content, is signed, in the place and date indicated above, by the following witnesses and by the person filling in this document:

	Witnesses		PERSON WHO FILL THE DOC
NAME			
ID NUMBER			
SIGNATURE			

CHAIN OF CUSTODY. The bottle with ID no. indicated on the label located at the top of the sheet is delivered to

PERSON	COMPANY	DATE	SIGNATURE

Credits: Spanish Maritime Safety and Rescue Agency (SASEMAR), Spain

MINISTERIO DE FOMENTO

Salvamento Marítimo

SAMPLING INFORMATION AND CHAIN OF CUSTODY

Paste the label Nº ID bottle 2

With date............................ at .... hours, (name and surname of the sample taker) ...., with national identity card nº..and contact telephone·............................. and as representative of............(name the organisation)..., has taken............ bottles from, (iidentification numbers are indicated at the top of this report) The details of the present sample collection are indicated below:

MOVILISED UNIT(8): ______ **Suspect source**(10): ______
CASE Nº(9): ______ **Pictures taken** (11): ☐Yes, ☐No

Place where the sample is taken
Location: ______
Geographical position (______ : ______
☐ **Sea**:
In port: ☐yes, ☐No Which? ______
☐surface ☐between waters ☐sea bottom
☐ **Land**:
☐beach ☐rocky shore ☐others ______
☐**Ship:** ______
☐Tank ______
☐Bilge ☐Others ______
☐ **Installation/Plant** (17): ______
☐Tank ______
☐pipeline ______ ☐Others ______

WEATHER CONDITIONS
Wind : ______
Temperature: Sea: ______ Ambient ______
Visibility : ______ **Sea state**): ______

SPILL DESCRIPTION
Presence of sheens: ☐yes, ☐No
Dimension :
Nature (hydrocarbon, jellyfish, etc)
Consistency: ☐Solid, ☐Liquid, ☐Semisolid ☐Other (specify):
Shape: ☐continuous surface, ☐strips, ☐cakes (>1 m²), ☐tar balls; ☐Others (specify):
Appearance: ☐Sheen; ☐Rainbow; ☐Metallic; ☐discontinuous true oil colour; ☐continuoous true oil colour; ☐Other (specify):

SAMPLE DESCRIPCTION
ODOUR: ______
Colour: ______
Presence of impurities : ☐yes, ☐No which? ______
Sample materials: ☐polietilene cone, ☐EFTE sheet, ☐Other (specify): ______

Further remarks: ______

And for the record, the present record is extended which, once read by all the appearing parties and finding it in accordance with its content, is signed, in the place and date indicated above, by the following witnesses and by the person filling in this document:

	Witnesses		PERSON WHO FILL THE DOC
NAME	______	______	
ID NUMBER	______	______	
SIGNATURE	______	______	______

CHAIN OF CUSTODY. The bottle with ID no. indicated on the label located at the top of the sheet is delivered to

PERSON	COMPANY	DATE	SIGNATURE

Credits: Spanish Maritime Safety and Rescue Agency (SASEMAR), Spain

MINISTERIO DE FOMENTO — Salvamento Marítimo

SAMPLING INFORMATION AND CHAIN OF CUSTODY

Paste the label Nº ID bottle 3

With date.......................... at .... hours, (name and surname of the sample taker) ...., with national identity card nº..and contact telephone·.............................. and as representative of...........(name the organisation)..., has taken........... bottles from, (iidentification numbers are indicated at the top of this report) The details of the present sample collection are indicated below:

MOVILISED UNIT(8): ______ **Suspect source**(10): ______
CASE Nº(9): ______ **Pictures taken** (11): ☐Yes, ☐No

Place where the sample is taken
Location: ______
Geographical position (______ : ______
☐ **Sea**:
In port: ☐yes, ☐No Which? ______
☐surface ☐between waters ☐sea bottom
☐ **Land**:
☐beach ☐rocky shore ☐others ______
☐ **Ship:** ______
☐Tank ______
☐Bilge ☐Others ______
☐ **Installation/Plant** (17): ______
☐Tank ______
☐pipeline ______ ☐Others ______

WEATHER CONDITIONS
Wind : ______
Temperature: Sea: ______ Ambient ______
Visibility : ______ **Sea state**): ______

SPILL DESCRIPTION
Presence of sheens: ☐yes, ☐No
Dimension :
Nature (hydrocarbon, jellyfish, etc)
Consistency: ☐Solid, ☐Liquid, ☐Semisolid ☐Other (specify):
Shape: ☐continuous surface, ☐strips, ☐cakes (>1 m²), ☐tar balls; ☐Others (specify):

Appearance: ☐Sheen; ☐Rainbow; ☐Metallic; ☐discontinuous true oil colour; ☐continuoous true oil colour; ☐Other (specify):

SAMPLE DESCRIPCTION
ODOUR: ______
Colour: ______
Presence of impurities : ☐yes, ☐No which? ______
Sample materials: ☐polietilene cone, ☐EFTE sheet, ☐Other (specify): ______

Further remarks: ______

And for the record, the present record is extended which, once read by all the appearing parties and finding it in accordance with its content, is signed, in the place and date indicated above, by the following witnesses and by the person filling in this document:

	Witnesses		PERSON WHO FILL THE DOC
NAME	______	______	
ID NUMBER	______	______	
SIGNATURE	______	______	______

CHAIN OF CUSTODY. The bottle with ID no. indicated on the label located at the top of the sheet is delivered to

PERSON	COMPANY	DATE	SIGNATURE

Credits: Spanish Maritime Safety and Rescue Agency (SASEMAR), Spain

MINISTERIO DE FOMENTO | Salvamento Marítimo

SAMPLING INFORMATION AND CHAIN OF CUSTODY

STICK THE LABEL ID	BOTTLE 1
BOTTLE 3	BOTTLE 2

With date............................ at .... hours, (name and surname of the sample taker) ...., with national identity card nº..and contact telephone·.............................. and as representative of............(name the organisation)..., has taken........... bottles from, (iidentification numbers are indicated at the top of this report) The details of the present sample collection are indicated below:

MOVILISED UNIT(8): ______ **Suspect source**(10): ______
CASE Nº(9): ______ **Pictures taken** (11): ☐Yes, ☐No

Place where the sample is taken
Location: ______
Geographical position (______ : ______
☐ **Sea**:
In port: ☐yes, ☐No Which? ______
☐surface ☐between waters ☐sea bottom
☐ **Land**:
☐beach ☐rocky shore ☐others ______
☐ **Ship:** ______
☐Tank ______
☐Bilge ☐Others ______
☐ **Installation/Plant** (17): ______
☐Tank ______
☐pipeline ______ ☐Others ______

WEATHER CONDITIONS
Wind : ______
Temperature: Sea: ______ Ambient ______
Visibility : ______ **Sea state**): ______

SPILL DESCRIPTION
Presence of sheens: ☐yes, ☐No
Dimension :
Nature (hydrocarbon, jellyfish, etc)
Consistency: ☐Solid, ☐Liquid, ☐Semisolid ☐Other (specify):
Shape: ☐continuous surface, ☐strips, ☐cakes (>1 m²), ☐tar balls; ☐Others (specify):

Appearance: ☐Sheen; ☐Rainbow; ☐Metallic; ☐discontinuous true oil colour; ☐continuous true oil colour; ☐Other (specify):

SAMPLE DESCRIPCTION
ODOUR: ______
Colour: ______
Presence of impurities : ☐yes, ☐No which? ______
Sample materials: ☐polietilene cone, ☐EFTE sheet, ☐Other (specify): ______

Further remarks: ______

And for the record, the present record is extended which, once read by all the appearing parties and finding it in accordance with its content, is signed, in the place and date indicated above, by the following witnesses and by the person filling in this document:

	Witnesses		PERSON WHO FILL THE DOC
NAME	______	______	
ID NUMBER	______	______	
SIGNATURE	______	______	______

SHIPPING DESTINATION:

Nº ID	DESTINATION

Credits: Spanish Maritime Safety and Rescue Agency (SASEMAR), Spain

APPENDIX 2.3

Credits: KBIN-OD-Nature Lab, Belgium

Marine pollution sample No.:

Date: .. / .. / ....   Local time: ......

Ship from which sampling was carried out:

....

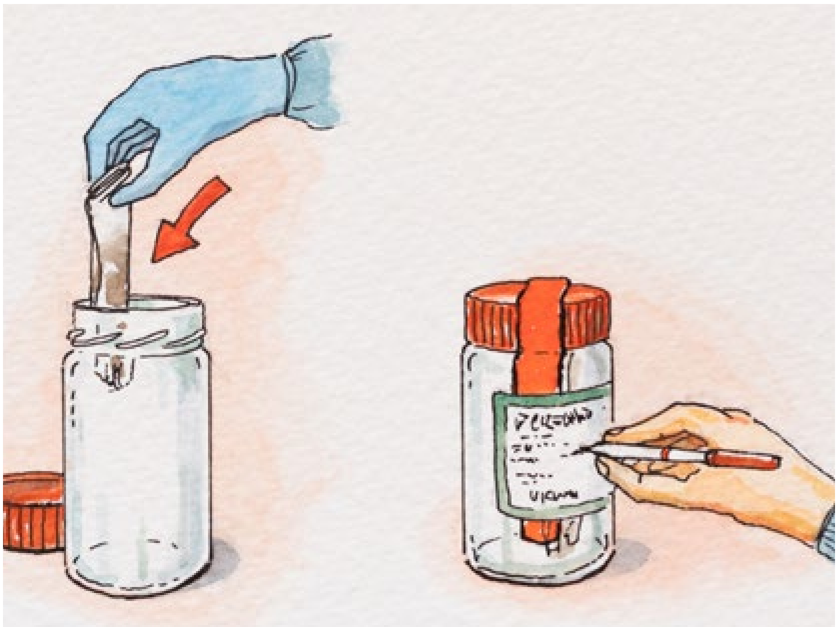
Position:

Type of pollution: oil - chemicals - algae -

unknown - other: ....

Suspected polluter:
(name + signature sample-taker/responsible party) :

Notes:



kustwacht
garde côtière
coast guard

RvS- SAMPLING FORM

Report of Sampling at Sea Pollution

1. Sample taker Name sampletaker	
Service	
Platform used for sampling (e.g. name of patrol vessel)	
2. DATE AND HOUR Date (dd.mm.jjjj)	
Time (lokal time)	
3. N° SAMPLE	
4. POSITION Position (coordinats)	
Location (sandbank, beach, shiptank, ...)	
5. DESCRIPION Type OF incident (vb. MARPOL incident; shipping incident)	
Type of contamination (oil/chemical/algae/other)	
Color contamination on water surface	
Ships in the vicinity (name)	
Birds in the water in the stain?	
6. WEATHER Wind force and direction	.... (Bft) ....
Wave height - seaway	....
Tide (HT/LT)	
Flow direction and velocity	....
7. Notes (e.g. difficulties in taking samples)	

Credits: KBIN-OD-Nature Lab, Belgium

COC-FORMULIER STAALNAME

SAMPLE CHAIN OF CUSTODY TRACKING FORM

This form must be signed off every time samples are surrendered/transferred, in order to obtain a complete logistic chain of custody of the samples. In this way, samples remain traceable, from the moment the sample is taken until it is submitted to a court of law.

Case n°:	
Sampling Officer/Department:	
Suspect (polluter):	
Type of incident:	

DESCRIPTION OF SAMPLES			
Sample #	**Date/hour sampling**	**Location Sampling**	**Description (stamp-#, markings; remarks)**

CHAIN OF CUSTODY				
Sample #	**Date/hour sampling**	**Transferred by (signature & ID)**	**Received by (signature & ID)**	**Remarks / Location**

APPENDIX 2.4

Credits: Rijkswaterstaat CIV RWS Lab, the Netherlands

Rijkswaterstaat
Ministry of Infrastructure and Water Management

Rijkswaterstaat RWS-Laboratory — Sample information form for forensic analysis requests

Case number: F	- Case number can be requested from Ronald van der Vliet, tel. 088-7973795 *(8 AM – 5 PM)* - Request of sample transport, tel. 088-7973793

Client — **Additional report to**

-	name:		-	name:	
-	name of agency:		-	name of agency:	
-	office address:		-	office address:	
-	ZIP code & city:		-	ZIP code & city:	
-	telephone number:		-	telephone number:	

Sample information *	F		F		F	
number of bottles						
preservation according standard procedures	yes	no	yes	no	yes	no
discrete (grab) or composite sample	discrete	composite	discrete	composite	discrete	composite
date and time of sampling						
sample description / location						
requested analysis / parameters						
name and agency sample taker						
For reporting to PowerBrowser:						
facility - and sample location code						
date and time of inspection						
code of inspector						
LIMS number						
remarks and observations (can be continued on page 3)						

* Sample information will be copied to the client report, as much as possible.

RWS company information — *Page 1 of 3*

Rijkswaterstaat
Ministry of Infrastructure and Water Management

Rijkswaterstaat RWS-Laboratory

Sample information form for forensic analysis requests

Case number: F (see front page)

Sample transfer from Client to RWS-Laboratory

Sealed by:		Seal code(s):		Date:	
Seal broken by:		Seal code(s)::		Date:	

	TRANSMISSION			RECEPTION		
Date	Agency and department.	Name	Signature	Agency and department.	Name	Signature

Sample transfer within RWS-Laboratory

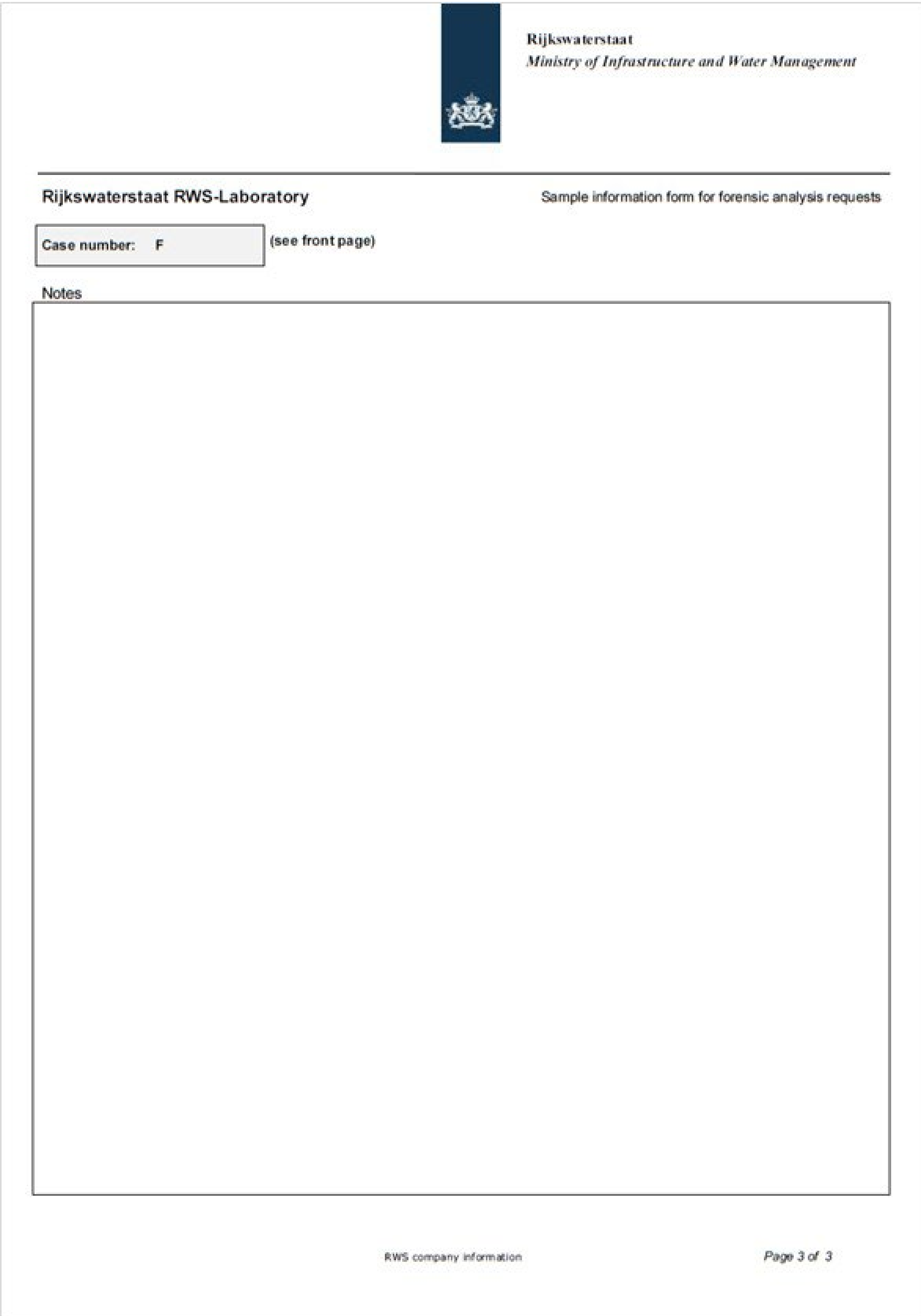
		TRANSMISSION			RECEPTION		
Date	Sample	Lab section	Name	Signature	Lab section	Name	Signature

Outsourcing

Sample numbers	
Contractor	
Remarks	

RWS company information Page 2 of 3

Credits: Rijkswaterstaat CIV RWS Lab, the Netherlands



Rijkswaterstaat
Ministry of Infrastructure and Water Management

Rijkswaterstaat RWS-Laboratory

Sample information form for forensic analysis requests

Case number: F	(see front page)

Notes

RWS company information

Page 3 of 3

Credits: Rijkswaterstaat CIV RWS Lab, the Netherlands

APPENDIX 3

ADDITIONAL INFORMATION REGARDING SAMPLING ON BOARD SHIPS AND FURTHER LEGAL ASPECTS/ENFORCEMENT

Sometimes the relation between a slick on the sea surface and the ship that caused it is not easy to establish. In these cases, states resort to sample taking in order to demonstrate that relation between the slick and the oil on board. Samples should be taken from all vessels identified as being a possible source of the pollution, to find the culprit or to eliminate suspects. Sampling can demonstrate the relation between ship and slick, but care has to be taken because an invalid sample may "free" a liable ship. International Law regarding ship-source pollution makes references to evidence gathering, including legal sampling, in many conventions, mainly at the United Nations Convention on the Law of the Sea (UNCLOS) and the International Convention for the Prevention of Pollution from Ships, 1973 as modified by the Protocol of 1978 (MARPOL).

EVIDENCE SAMPLING PROCEDURE KEYPOINTS

Note: only to be done by specially trained personnel!

- Take your own samples, do not accept samples from the ship crew to avoid manipulation.
- The color of the oil should not be the reason to eliminate tanks to be sampled. To take samples from all the potential internal sources on-board gives more chances of finding the origin of the spill.
- Samples should be taken from the fuel tank, day tank, cargo tank, waste oil or slops tank, and bilge. The sample taker should also compare any soundings taken, with the ship's log and the records in the Oil Record Book, as these may indicate where a spill came from.
- Drawings such as the "piping system", "tank plan", "capacity plan" and "air, filling and sounding pipes" may also give valuable information. They show the positions and capacities of the different tanks and normally also indicate what types of oil are carried therein. Also they can indicate piping and pathways that could be used for illicit discharges.
- Sometimes, oil or dirt under a flange that's not dripping is an indication that the pipe has been dismounted. Also, excessive paint on flanges or flange's bolts with scratched paint are indications of tampering, especially when the pipe goes to an overboard discharge valve.
- Once it has been decided where samples should be taken, methods to be used are:
    - draining directly from tanks
    - taking samples from bilge with a bucket
    - taking samples through manholes or sounding openings
    - taking samples by ETFE net; and,
    - taking samples from tanks through sound piping (this could use a glass test-tube weighted for sinking or a heavy brass tube.
- Samples of heavy oil residues from sludge tanks could be difficult due to the viscosity of the oil. An ETFE net can help.
- Samples should be taken at the upper, middle and bottom levels of the oil in the tank.
- Attention should be paid to the fact that the oil may not be homogeneously distributed.

TIP

OIL SAMPLES FROM TANKS SHOULD BE TAKEN FROM THE UPPER, MIDDLE AND BOTTOM LEVELS BECAUSE OF POSSIBLE INHOMOGENEITIES WITHIN A TANK

- Owing to the risk of electrostatic charges, sampling in tanks containing flammable gases must only be carried out using a sampling device hanging on a string of natural, not synthetic, material.

LAW

A State shall be entitled to act on board of a foreign vessel. Conventional law (UNCLOS & MARPOL) allows the inspection of foreign vessel while in port. Conventional law establishes jurisdictional matters, focusing on permanent jurisdiction of the vessel's flag State. Due to the international nature of navigation, evidence obtained in one state may be linked to oil spills effects that become manifest in other states.

UNCLOS includes rules asserting jurisdiction over vessels of other States than the flag State, based on the maritime space where the vessel is. MARPOL and UNCLOS rules on jurisdiction are not identical, although MARPOL Article 9 establishes that jurisdiction matters shall be interpreted according to UNCLOS. UNCLOS addresses aspects of investigation and evidence gathering in procedures of ship-source pollution and their validation.

UNCLOS, the main relevant international instrument on the use of oceans, makes some references to evidence in relation with pollution from ships. References can be found in the following articles:

- 223 (Measures to facilitate proceedings);
- 217 (Enforcement by flag States);
- 218 (Enforcement by port States);
- 220 (Enforcement by coastal States); and
- 226 (Investigation of foreign vessels).

MARPOL, as "technical" instrument, also addresses issues surrounding the need for evidence of violation of its regulations. References can be found in these articles:

- 4 (Violation)
- 6 (Detection of Violations and Enforcement of the Convention)
- Annex I (Regulations 15.7 & 34.7)

EU Law is also a legal source in relation with oil pollution:

- Directive 2005/35/EC of the European parliament and of the council, of 7 September 2005, on ship-source pollution and on the introduction of penalties for infringements. Amended through Directive 2009/123/EC. Follows the UNCLOS and MARPOL statements on evidence collection. Please note this piece of legislation is being updated at the time of writing.

- Directive (EU) 2019/883 of the European Parliament and of the Council of 17 April 2019 on port reception facilities for the delivery of waste from ships, amending Directive 2010/65/EU and repealing Directive 2000/59/EC, indicates the possibility of inspections on board vessels to check compliance with MARPOL.
- Directive 2009/16/EC of the European parliament and of the council, of 23 April 2009, on port State control. Establishes the procedures on port State control inspections.
- Directives 2002/59/EC (amended through 2009/17/EC) and 2010/65/EU, may also be relevant on investigation of suspected ships.

Recap:

- MARPOL and UNCLOS indicate the possibility, or the mandate, to investigate pollution incidents.
- Inspection by the port State, according to MARPOL, doesn't need the suspicion of a violation of pollution prevention regulation.
- The maritime space where the incident occurs determines the powers of the coastal State. These powers decrease with increasing distance from shore.
- If legal sampling is demanded under the applicable jurisdiction it must be performed as collection of evidence.

Legal and technical documents and references to oil spill sampling

There is no internationally agreed procedure on sampling. Sampling procedures are only generally referred to in MARPOL and IMO Resolutions, other sources must be relied upon.

Soft law establishes advice and good practices: recommendations, guidelines and handbooks are useful indicating what are the safeguards to be considered while sampling.

IMO Resolution Res. A.1138(31), Procedures for port state control, is relevant to this issue. Other IMO Soft law sources can be found in Circulars and Manuals.

There are also other international – and municipal - sources to consider on sampling, especially those elaborated within regional organizations.

IMO:

IMO Manual on Oil Pollution, Section VI: IMO Guidelines for Sampling and Identification of Oil Spills. This document gives relevant information on oil sampling (assesses aspects on sampling: sampling equipment, safety, procedures...). To a lesser degree, these circulars are relevant to oily bilge water, sludge and Oil Water Separators (OWS):

- MEPC.1/Circ.642 (2008 Revised guidelines for systems for handling oily wastes in machinery spaces of ships incorporating guidance notes for an integrated Bilge Water Treatment System (IBTS)). Amended by MEPC.1/Circ.676 and MEPC.1/Circ.760
- MEPC.1/Circ.677 (Guide to diagnosing contaminants in oily bilge water to maintain, operate and troubleshoot bilge water treatment systems)
- MSC MEPC.4/Circ.3 (Blanking of bilge discharge piping systems in port)

International Organizations sampling manuals and references:

- EMSA: Addressing Illegal Discharges in the Marine Environment (https://emsa.europa.eu/publications/inventories/item/1879-addressing-illegal-discharges-in-the-marine-environment.html)
- INTERPOL: Illegal Oil Discharges from Vessels Investigative Manual (http://archive.iwlearn.net/sprep.org/legal/documents/OilDischargesManual.pdf)

Industry:

- ITOPF: Effects of Oil Pollution on the Marine Environment. Technical Information Paper, 13 (http://www.itopf.org/knowledge-resources/documents-guides/document/tip-13-effects-of-oil-pollution-on-the-marine-environment)

Regional Agreements:

- PMoU: Paris MoU 2011 Instruction 44/2011/20: Procedure for Investigation under MARPOL (PMoU confidential document)
- Bonn Agreement: North Sea Manual on Maritime Oil Pollution Offences (https://www.ospar.org/work-areas/cross-cutting-issues/north-sea-network).

  Also, the work of Bonn-OSINet is of paramount importance, as it includes the experience of the intitutions of the Bonn Agreement specialized on oil spill identification (https://www.bonnagreement.org/site/assets/files/1087/chapter32_oil_spill_identification.pdf).

- HELCOM: HELCOM Manual on Co-operation in Response to Marine Pollution within the framework of the Convention on the Protection of the Marine Environment of the Baltic Sea Area (http://www.helcom.fi/Lists/Publications/HELCOM%20Manual%20on%20Co-operation%20in%20Response%20to%20Marine%20Pollution%20-%20Volume%201.pdf)

KEY SAMPLING AREAS

a) Bilge

Bilge water originates from leakages from many sources including the engines cooling systems, stuffing box, pipelines, condensation, cleaning etc. However, it could be mixed with oil from machinery, pipes or tank leakages.

Oily bilge water could be stored in a specific tank; although this tank is not mandatory. If it exists, it shall be identified and included in the IOPP Certificate.

Oily bilge water is allowed to be discharged to the sea, but only through an approved oily water separator (OWS), with a maximum oil concentration of 15 ppm, and without substances not permitted to be disposed at sea (it doesn't mean that bilge emergency overboard pumping line shall be blanked [MSC MEPC.4/Circ.3]).

Excessive bilge water could indicate a failure of the ship's machinery or systems. In order to avoid detection - or to spend too much money delivering excessive oily waste to reception facilities - oily bilge wate may be pumped to the sea unlawfully, avoiding the low rate of discharge controlled by the OWS.

Integrated Bilge Water Treatment System (IBTS):MEPC.1/Circ.642. A Statement of Fact about IBTS could be issued by the flag State Administration.

b) Sludge tanks

Oil residue (sludge) means the residual waste oil products such as those resulting from the purification of fuel or lubricating oil used in main or auxiliary machinery, or the separated waste oil from bilge water separators, oil filtering equipment or oil collected in drip trays, and waste hydraulic and lubricating oils (MEPC.1/Circ.642).

Sludge should be disposed to reception facilities through a standard discharge connection. A Sludge discharge pipeline should not be connected to other systems. In addition, sludge could be burned in an incinerator on board, however this is heavily controlled by MARPOL Annex VI, Regulation 16.

Sludge shall be stored in specific tanks. Tanks are mandatory and shall be identified and included in the IOPP Certificate.

Other tanks that should be considered for sampling include the day tank, cargo tanks and slop tanks.

c) Oily Water Separator (OWS)

An OWS is mandatory for ships over 400 GTs (Annex I/ Reg. 14).

Guidelines on specifications of OWS are included in Resolutions A.393(X), MEPC.60(33), and MEPC.107(49). Either applies depending of the keel laying date of the vessel.

The discharge must not bypass the 15 ppm bilge separator. The discharge piping system of the 15 ppm bilge water separator should be completely separate from the bilge pumping and ballast water system (except the recycling line).

d) Cargo areas

The Oil Record Book should be checked to know which tanks have been loaded, and which operations with respect to oil have been made:

Cargo pump room is a common place for oil drips which could be discharged into the sea.

Cargo tanks: although stripping systems have been optimized, in old vessels an incompatible product with the next cargo may result in a discharge overboard and therefore a possible source of slicks.

Ballast tanks: older tankers that still place ballast in their cargo tanks, and those that may use this process to aid stability, could exceed the rate of discharge in an oily water separator or by-pass the Oil/Water Interface Detector. Oil/Water Interface level is not always easy to check. Please note neither of these practices are common at the present time.

Slops tanks: tank cleaning residues if not managed according to the regulations could be a common source of pollution.

THE OIL RECORD BOOK (ORB)

ORB's are mandatory for tankers of 150 GTs and above and for any other vessel of 400 GTs and above (MARPOL Annex I Regulation 17).

This document records each machinery space operations that take place in the ship relating to:

- ballasting or cleaning of oil fuel tanks;
- discharge of dirty ballast or cleaning water from oil fuel tanks;
- collection and disposal of oil residues (sludge and other oil residues);
- discharge overboard or disposal otherwise of bilge water which has accumulated in machinery spaces; and,
- bunkering of fuel or bulk lubricating oil.

All completed operations shall be fully recorded without delay in the ORB, and signed by the officer in charge. Each completed page shall be signed by the master of the ship (guidance from MEPC.1/Circ.640).

The lack of records of operations at the ORB, or if the records are manifestly at variance with the evident factual situation, allows for a more detailed inspection of the vessel (including sampling).

A Port State Control Officer (PSCO) may inspect the ORB while the ship is in its port or offshore terminals. Any copy certified by the master of the ship as a true copy of an entry in the ORB shall be made admissible in any judicial proceedings as evidence of the facts stated in the entry.

At any sampling procedure, the ORB shall be checked in order to match its records with soundings of tanks, it can help discover discrepancies.

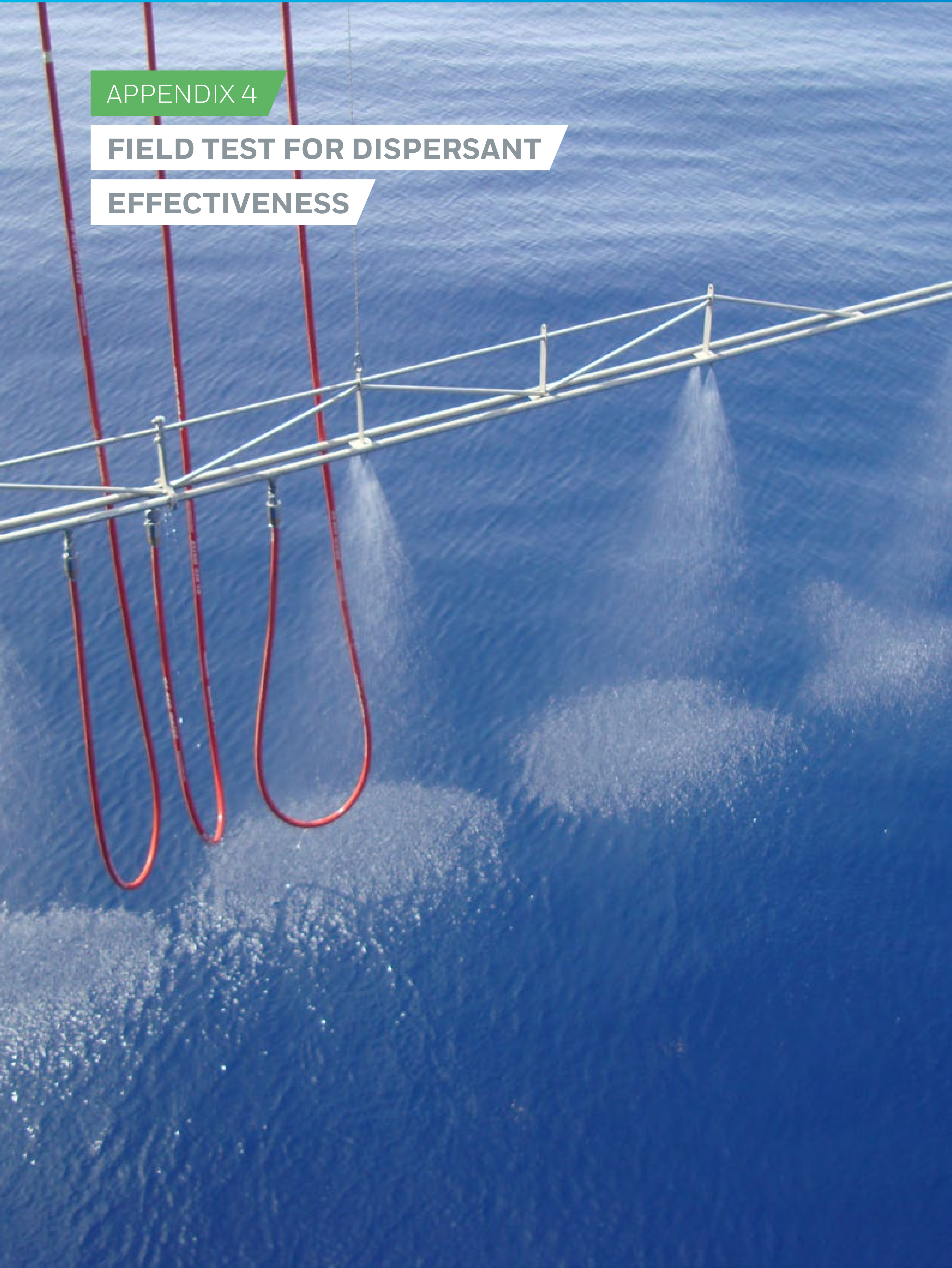
OTHER DATA

Some documents on board are related directly to the slick, while others present ancillary sources of information:

- IOPP certificate;
- Records in the Oil Record Book;
- Records in the Engine Log Book;
- Tank sounding records;
- Piping system and tank plan;
- Data from the OWS, incinerator and/or records of the Oil Discharge Monitoring System (ODMS);
- Data from charts and other Log Books;
- Captain, Chief Engineer, Engineers and ratings interview (some guidance could be found at paragraph 5.2.7 of PMoU Instruction 44/2011/20: Procedure for Investigation under MARPOL); and,
- Receipts from Port Reception Facilities.

APPENDIX 4

FIELD TEST FOR DISPERSANT EFFECTIVENESS



Appendix 4 – Field test for dispersant effecftiveness

During an oil spill response operation, an oil sample can be taken from the sea to check if use of a chemical dispersant is an option. This is often called a Field Effectiveness Test (FET test) or “Shaky Bottle Test”. The on-site test is very useful to check if the oil in question is dispersible and thus provides essential information for the decision makers.

For this test, sampled oil from a spill is added to seawater in two cylinders or bottles. Subsequently, in one of them, a small amount of the chemical dispersant is added. Then, the cylinders/bottles are shaken and finally left standing. After a few minutes, an indication of the dispersant effectiveness can be seen by comparing the cylinders/glasses. While the undispersed oil collects on top of the water phase, the dispersed oil is distributed in the water column.

A detailed procedure should be included in field test equipment kits, a procedure which describes the amounts added and the shaking time.

This field test is an example of oil spill sampling for operational use [41].

See also the document “Overview of national dispersant testing and approval policies in the EU” (developed in 2016 under the CTG MPPR work).

The glass cylinder to the right shows that the oil is dispersible. (Credits: Norwegian Coastal Administration)

APPENDIX 5

WHAT'S WRONG HERE?

PRACTICAL EXAMPLES OF HOW NOT TO SAMPLE

WHAT'S WRONG HERE? PRACTICAL EXAMPLES OF HOW NOT TO SAMPLE

EXAMPLE 1

- Use proper tamper-proof seals or other sealing options (tamper-evidence labels/ security seals or tape /evidence bags).
- Do not use sampling labels, ID stickers etc as security seal!
- Use sampling labels, ID stickers etc. exclusively for their own purpose (they are useless as seals anyway, additionally, you might lose the information on labels when they get ripped etc.)!

(courtesy of BSH, Germany)

EXAMPLE 2

- Don’t put sampling gear (clips, lines) with the samples into the sampling container!

(courtesy of BSH, Germany)

EXAMPLE 3

- Always use dedicated sampling containers!
- Never use containers for food or drink!
- Always pack and transport samples in a way that prevents shattering!
- Always label your samples in a clear and precise way.

(courtesy of IDAEA-CSIC Lab, Spain)

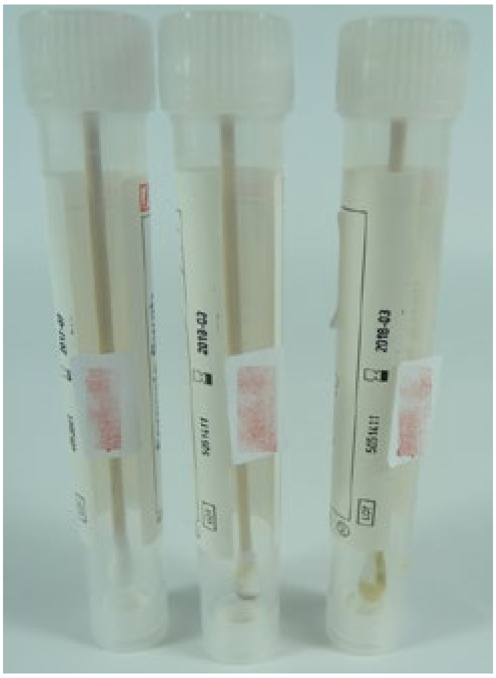
EXAMPLE 4

- Always use dedicated sampling containers!
- Never use containers for food or drink!

(courtesy of BSH, Germany)

EXAMPLE 5

- Always use equipment suitable for oil spill sampling (ETFE net, stainless steel/PTFE spatula).
- Don't use equipment from other forensic disciplines (e.g. cotton swabs from DNA sampling kits - cotton swabs with wood skewers will interfere with the analysis of the sampled oil).



(courtesy of BSH, Germany)

EXAMPLE 6

- Always use equipment suitable for oil spill sampling (ETFE nets!).
- Don't use tissues, handkerchiefs, cloths or cleaning rugs since they will interfere with the analysis of the sampled oil.

(courtesy of IDAEA-CSIC Lab, Spain)

EXAMPLE 7

- Whenever possible, avoid oiling the outside of a sampling container.
- Do not fill containers to their maximum capacity to prevent overflowing of the oil.
- If gotten oiled, always clean the outside of a sampling container directly after sampling and before any further handling.
- Do not apply labels and seals to oiled surfaces (label will become unreadable, seals will not stick).

(courtesy of BSH, Germany)

EXAMPLE 8

- Use proper tamper-proof seals or other sealing options (tamper-evidence labels/ security seals or tape /evidence bags).
- Do not use sampling labels, ID stickers, gloves etc. as security seal.
- Use sampling labels, ID stickers etc. exclusively for their own purpose (they are useless as seals anyway, additionally, you might lose the information on labels when they get ripped etc.).

(courtesy of BSH, Germany)

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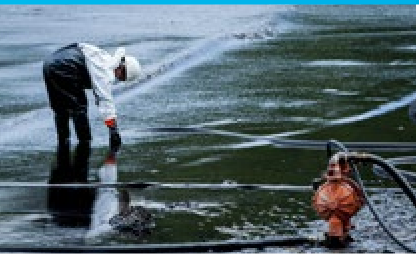
A special mention to Mr Juan Carlos Arbex Sánchez for the technical drawings



ABOUT THE EUROPEAN MARITIME SAFETY AGENCY

The European Maritime Safety Agency is one of the European Union's decentralised agencies. Based in Lisbon, the Agency's mission is to ensure a high level of maritime safety, maritime security, prevention of and response to pollution from ships, as well as response to marine pollution from oil and gas installations. The overall purpose is to promote a safe, clean and economically viable maritime sector in the EU.

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