## 6. OIL SAMPLING

## 6.1 INTRODUCTION

The 17th Meeting of the Combatting Committee of the Helsinki Commission approved the Guidelines on Spill Sampling (the Nordtest Method for Oil Spill Identification; NT CHEM 001, Edition 2; cf. document CC 17/8/1). The Nordtest Method is meanwhile introduced in the Bonn Agreement and the Copenhagen Agreement as well. It contains mainly provisions for securing evidences on discharges from ships and co-operation in investigating violations or suspected violations of discharges regulations. But on the other hand the compensation of spill clean-up costs in major response actions needs carefully secured evidence in order to avoid the refusal of claims for compensation (persistent oil) within the CLC and the IOPC Fund Conventions.

Gas chromatography combined with mass spectrometry analysis (GC/MS) has proved particularly useful for oil identification, compared with, for example, determinations of sulphur, nickel and vanadium.

The GC/MS method has been developed to recognise and take into account the impact of weathering on the identification process. Compounds analysed are common to most types of oil. These compounds are either unaffected or affected in a predictable manner by weathering.

The GC/MS method is not influenced by minor alterations in the sampling or handling techniques, nor is the GC/MS technique of the Nordtest Method limited to the parameters listed here.

Whenever possible it should be avoided, to the greatest possible extend, that the polluter could benefit from the doubts concerning his responsibility.

## 6.2 FIELD OF APPLICATION

The identification system is applicable to oil samples of petroleum origin, containing significant amounts of hydrocarbons with boiling points above 200 °C. Examples are crude oil, fuel oil, lubricating oil and mixtures, such as bilge oil and tank washings. The required sample volume is very small.

The use of this identification system requires that spill samples and samples taken from suspected sources be compared.

When samples from suspected sources are not available, the system may be used to characterise the spill sample. Administrative efforts may then be made to find the source responsible.

### 6.3 CHECKLIST FOR SAMPLING

The annexed checklist (Annexes 1, 2, 3 of the Nordtest Method) shall be applied when collecting and providing samples in order to determine the source of the spill.

For an example of national procedures/routines in oil sampling at sea, see "Oil Sampling at Sea".

## ANNEX

#### I CHECKLIST FOR SPILL SAMPLING

#### General

- 1. Samples should be taken with sampling devices and containers of glass, teflon or stainless steel.
- 2. Preferably, at least 1 ml of oil should be sampled but no samples should be considered too small. If possible, a larger sample volume is recommended for additional analyses (100 ml).
- 3. If contamination of the sample is suspected, take blind samples from the possible contamination.
- 4. To permit cross-checking, three parallel samples should be taken from some of the localities. In larger spills, one sample per locality should be sufficient.
- 5. The sampling container should be properly labelled with all relevant information before sealing.
- 6. A sampling log book with all relevant information from the samples and the sampling environment should be kept.
- 7. The samples should be taken and handled under the supervision of authorised personnel. A chain of custody should be maintained until the identification process is concluded by the relevant authority.
- 8. If stored, all oil samples should be kept under lock and key in darkness at a maximum temperature of +4 °C.
- 9. Oil samples should be taken to an authorised laboratory without unnecessary delay.
- 10. Packing and transport should be carried out in such a way that damage to the samples is avoided. Sorbent material should be used.
- 11. National regulations for transport of flammable materials should be followed.
- 12. Samples should be handled as if they were legal evidence.

#### Collection of samples from the water surface

- 1. Try to concentrate the oil fraction in the sample container by skinning the oil from the water. A bucket with small holes or a conical teflon bag can be used to concentrate the oil into the container.
- 2. Do not fill the container completely. Allow for thermal expansion of the sample.
- 3. If possible, sample oil from the thickest part of the slick.
- 4. In highly contaminated waters, e.g. harbours, take blind samples.
- 5. If a combat action against a waterborne oil spill lasts for several days, take oil samples every day for documentation of weathering and possible additional spills from other sources.
- 6. If other suspicious slicks occur, i.e. their appearance differs, or if slicks are observed a long distance from the expected site, take samples as required in order to ascertain whether more than one spill has occurred in the area.

#### **Collection of samples from beaches**

- 1. Take samples from the geographical edges of the polluted area to document the range of the spill.
- 2. Take samples from different localities within the polluted area to document the spill distribution.
- 3. Old tar balls, earlier oil spills, creosote from pier logs, etc. can contaminate the sample. Take blind samples if contamination is suspected.
- 4. Take relevant samples in order to ascertain whether more than one spill has occurred, whenever anything unusual or suspicious (colour, texture, etc.) is observed in the polluted area.
- 5. When seaweed, small pieces of wood or debris are contaminated by oil, the complete specimen can be placed in the sampling container.

#### Obtaining samples from oiled animals

1. Contaminated feathers and fur may be cut off and placed in the sample container.

- 2. Dead, oiled birds or other animals may be collected in plastic bags, labelled and frozen before sending to a laboratory.
- 3. Before sending any animals, make contact with the relevant authority to make proper arrangements for transport and storage.
- 4. Samples with large amounts of organic materials should be frozen to avoid biological decomposition.

#### II CHECKLIST FOR TAKING SAMPLES IN CARGO SYSTEMS OF OIL TANKERS

- 1. Identify the category of the tanker in relation to MARPOL requirements (i.e. COW, SBT, CBT or a tanker with conventional ballasting (below 4000 tdw). Copy the IOPP Certificate, including Supplement B.
- 2. Identify the loading condition of the ship (loaded, part-loaded or in ballast) and the quality of oil carried (last carried), and copy the bill of lading for the current (latest) voyage.
- 3. Study the oil record book and copy the pages dealing with the operations under investigation.
- 4. Obtain, if possible, a copy of the diagram of the cargo oil and ballast pumping and piping systems on the ship.
- 5. Study the printouts from the oil discharge monitoring and control systems and copy the parts covering the current (latest) ballast voyage.
- 6. Ascertain the current ballast or loading condition and identify tankers carrying ballast and tanks used for ballast during previous phases of the voyage.
- 7. Verify the status of the ship in the load-ballast handling cycle, i.e. whether it carries departure or arrival ballast, whether tank cleaning has been carried out during the voyage and whether water from the slop tanks has been discharged at sea.
- 8. Take oil samples representing the various qualities of cargo oil which the ship has carried during the current (latest) voyage, and mixtures which may have been generated. Take samples of oil remaining on board at locations where these are likely to collect, including (as applicable):
  - 8.1 reference samples carried on board
  - 8.2 all slop tanks (identify also the level of oil/water interface, the quantity of slop oil and the quantity of water in each slop tank)
  - 8.3 tanks which carry or have carried dirty ballast
  - 8.4 pump room bilges
  - 8.5 stripping pumps
  - 8.6 overboard cross-over line, both sides
  - 8.7 deballasting lines to sea chests, both sides
  - 8.8 cargo manifolds on deck

All samples taken must be clearly identified with respect to location and time when the sample was taken.

Take special care to obtain representative samples from slop tanks and bilges, where the composition of the oil may vary from place to place.

9. Note any additional observations which may be of any value in determining the likelihood that a discharge has taken place.

# III CHECKLIST FOR TAKING SAMPLES IN MACHINERY SPACES OF SHIPS

- 1. Verify that the ship carries a valid IOPP Certificate. Note whether the ship is certified as being equipped with a 100 ppm or a 15 ppm oily water separator/filtering equipment. Ascertain whether it has been granted a waiver for any equipment. Copy the certificate, including Supplement A.
- 2. Study the oil record book (machinery part) and copy the pages covering the period under investigation.
- 3. Check levels and contents. Take samples from the following tanks and spaces:
  - 3.1 all bilge wells
  - 3.2 bilge water holding tank (note if no bilge water holding tank is installed)
  - 3.3 waste oil tanks (the ship may have several)
  - 3.4 overflow tank for bunker oil
  - 3.5 fuel and lube oil purifier sludge tanks
  - 3.6 empty bunker tanks which may have been used for water ballast
- 4. Also take samples from:
  - 4.1 service tanks (day tanks) for the engines
  - 4.2 the bilge water separator outlet piping
  - 4.3 the sludge pump outlet piping
- 5. Inspect the bilge water separating/filtering equipment (note the liquid content at the test cocks, request opening of the filtering unit if saturation may be expected).
- 6. Inspect the tank top for accumulation of oil and sludge.
- 7. Note the type of cleaning agent used in the engine room and the claimed rate of consumption.
- 8. If the ship is of 10,000 GRT or above and has a 100 ppm separator, inspect the oil content meter and its recorder. Copy the recorder printout for the period under investigation.