



Designation: D4489 – 95 (Reapproved 2006)

## Standard Practices for Sampling of Waterborne Oils<sup>1</sup>

This standard is issued under the fixed designation D4489; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 These practices describe the procedures to be used in collecting samples of waterborne oils (see Practice D3415), oil found on adjoining shorelines, or oil-soaked debris, for comparison of oils by spectroscopic and chromatographic techniques, and for elemental analyses.

1.2 Two practices are described. Practice A involves “grab sampling” macro oil samples. Practice B can be used to sample most types of waterborne oils and is particularly applicable in sampling thin oil films or slicks. Practice selection will be dictated by the physical characteristics and the location of the spilled oil. These two practices are:

	Sections
Practice A (for grab sampling thick layers of oil, viscous oils or oil soaked debris, oil globules, tar balls, or stranded oil)	9 to 13
Practice B (for TFE–fluorocarbon polymer strip samplers)	14 to 17

1.3 Each of the two practices is designed to collect oil samples with a minimum of water, thereby reducing the possibility of chemical, physical, or biological alteration by prolonged contact with water between the time of collection and analysis.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazards statements, see Section 7.

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

D1129 Terminology Relating to Water

D3415 Practice for Identification of Waterborne Oils

<sup>1</sup> These practices are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

### 3. Terminology

3.1 *Definitions*—For the definitions of terms used in these practices, refer to Terminology D1129.

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *chain of custody*—a documented accountability of each sample, that is, date, time, and signature of each recipient when the sample changes hands, from the time of collection until the requirement for each sample is terminated.

3.2.2 *waterborne oil*—refer to Practice D3415.

### 4. Significance and Use

4.1 Identification of the source of a spilled oil is established by comparison with known oils selected because of their possible relationship to the spill, that is, potential sources. Generally, the suspected source oils are from pipelines, tanks, etc., and therefore pose little problems in sampling compared to the spilled oil. This practice addresses the sampling of spilled oils in particular, but could be applied to appropriate source situations, for example, a ship’s bilge.

### 5. Apparatus

5.1 *Sample Containers*, 100 to 125-mL wide-mouth glass jars that have been thoroughly cleaned. When field expedients must be employed, an empty container of each type used should be included in the shipment to the laboratory, to be used as a blank to measure inadvertent contamination.

5.2 *Closures*—Lids for the glass jars should have TFE-fluorocarbon polymer film or aluminum-coated insert.

5.3 *Strip Samplers*, 5 by 7.5 cm pieces of TFE-fluorocarbon polymer sheets (0.25 mm thickness, or screen or fabric (50–70 mesh)).

5.4 *Wooden Tongue Depressor*.

5.5 *TFE-Fluorocarbon Polymer Net Sampling Kit*.<sup>3</sup>

### 6. Reagents

6.1 *High Purity Solvents*,<sup>4</sup> that must be used for rinsing samplers and sample containers. The solvents which may be

<sup>3</sup> Sampling kit available from General Oceanics, Miami, FL, or equivalent, is suitable.

<sup>4</sup> MCB Spectroquality solvents, available from MCB Manufacturing Chemists, Inc. (Associate of E. Merck, Darmstadt, Germany), 480 Democrat Rd., Gibbstown, NJ 08027, or equivalent are suitable.

used are *n*-hexane, mixed hexanes, cyclohexane, pentane, or dichloromethane, acetone, or chloroform.

## 7. Hazards

7.1 **Precaution:** Extreme care should be exercised so as not to contaminate the samples or cause their integrity to be questioned.

7.2 **Warning:** The rinsing solvents are volatile and, except for dichloromethane, are flammable, and therefore should be handled with appropriate care. Dichloromethane will release toxic vapors when heated.

7.3 Minimize contact with oil even when wearing gloves.

## 8. General Sampling Guidelines

8.1 The objective is to obtain a sample for analysis that is representative of the spilled oil. The most critical factors in sampling are selecting a suitable location, collecting a sample of oil with the least water possible (to minimize possible sample alteration), and maintaining the sample integrity.

8.2 It is recommended that at least three samples be taken of each waterborne oil in order to demonstrate the homogeneity of the spill. These samples should be taken in different regions of the oil slick at points where the accumulation is heaviest. This will increase the volume of oil available for analysis. In the event that multiple samples cannot be collected, then a single sample should be collected from the area where the accumulation of oil visually appears to be the heaviest.

8.3 The following general rules are applicable to sampling of waterborne oils:

8.3.1 Take a sample that contains sufficient oil for the method or methods of analysis to be employed and for any replicate analyses that may be required.

8.3.2 Affix a label or tag to the sample jar in such a manner that it becomes an integral part of the container. The label or tag should contain the following information: sample identification, date and time of collection, location of collection, signature of person collecting the sample, and at least one witness to the collection.

8.3.3 Pack the samples, ship, and manipulate prior to analysis in a manner that maintains a continuous chain of custody and safeguards against tampering or changes in the properties of the samples.

8.4 Store collected samples at refrigerator temperatures (4 to 5°C).

NOTE 1—Storage at lower temperatures (–10°C or lower) may cause irreversible crystallization of waxes. Storage at 4 to 5°C obviates this problem; biological degradation at 4 to 5°C has been found negligible over a 3 to 5 year storage with respect to qualitative identification of oil.

## PRACTICE A—GRAB SAMPLING

### 9. Scope

9.1 This practice is applicable to thick layers of waterborne oil films, viscous oils, oil globules, and tar balls.

9.2 This practice is also applicable to sampling oil stranded on shorelines or oil-soaked debris.

## 10. Summary of Practice

10.1 The sampling consists of collecting the sample directly with the sample container, that is, scooping the sample up in the sample jar and sealing.

## 11. Apparatus

11.1 The sample container serves as the sampling device (see 5.1). The glass jars and lid liners should be rinsed three times with a high purity solvent (see 6.1), allowed to air dry, and assembled prior to use. Sample jars that are precleaned using EPA recommended wash procedures for organics are acceptable.

NOTE 2—To avoid possible sample contamination, do not reuse sample containers, lids, or liners.

11.2 Nitrile gloves are to be worn during sampling.

11.3 A detachable ring for the sample jar and sampling pole may be useful to extend sampling range.

## 12. Procedure for Floating Samples

12.1 Select the sampling site.

12.2 Unscrew the lid from the sample jar. Hold the jar in position for sampling; hold the lid in a free hand or place the lid in a safe position. Gently lower the sample jar into the water and gently skim the oil layer or oil globules from the water surface into the sample container. Continue the process until the sample container is approximately three-quarters full.

12.3 Remove the sample container from the water surface, replace and tighten the lid. Invert the jar and allow the container to stand in this position for 2 to 3 min.

12.4 Gently unscrew the sample jar lid and allow the water layer to drain out of the inverted container. Seal the lid and return the jar to the upright position.

12.5 Repeat 12.2 to 12.4, if necessary, until approximately 60 mL of oil is collected, or until there is no increase in the amount of recovered oil.

12.6 When the collection is complete, invert the jar and allow to stand for 10 min. Gently unscrew the lid to drain off excess water a final time. Tighten the lid and return the jar to the upright position. Wipe excess water and oil from the outside surface of the sample container.

12.7 Attach a sample label or tag to the container, bearing the information cited in 8.3.2.

## 13. Procedure for Shoreline Sampling (Oil on Sand and Debris)

13.1 Select a sampling site where oil accumulation is largest.

13.2 Open the sample jar; hold the jar in one hand and lid in the other. Using either the sample jar or the lid as a scoop, fill the jar three-quarters full with oil-saturated material. Use a wooden tongue depressor to maneuver the sample into the jar, if necessary.

13.3 Replace and tighten the lid. Wipe excess material from the outside surfaces of the sample container and lid.

13.4 Attach the sample label or tag to the container, bearing the information cited in 8.3.2.