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An American National Standard

Standard Practice for Preservation of Waterborne Oil Samples[1]

This standard is issued under the fixed designation D 3325; 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 (ϵ) indicates an editorial change since the last revision or reapproval.

ϵ^1 NOTE—Referenced Documents Section was editorially updated in June 2002.

1. Scope*

1.1 This practice covers the preservation of waterborne oil samples from the time of collection to the time of analysis. Information is provided to ensure sample integrity and to avoid contamination and to minimize microbial degradation.

1.2 The practice is for controlled field or laboratory conditions and specifies thorough preparation of equipment and precise operation. Where these details must be compromised in a field emergency, nonstandard simplifications are recommended that will minimize or eliminate consequent errors.

NOTE 1—Procedures for the analysis of oil spill samples are Practices D 3326, D 3415, D 3650, and D 4489, and Test Methods D 3327, D 3328, and D 3414 . A guide to the use of ASTM test methods for the analysis of oil spill samples is found in Practice D 3415.

1.3 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]
D 3326 Practices for Preparation of Samples for Identification of Waterborne Oils[3]
D 3327 Test Methods for Analysis of Selected Elements in Waterborne Oils [4]
D 3328 Test Methods for Comparison of Waterborne Petroleum Oils by Gas Chromatography[3]
D 3414 Test Method for Comparison of Waterborne Petroleum Oils by Infrared Spectroscopy[3]
D 3415 Practice for Identification of Waterborne Oils [3]
D 3650 Test Method for Comparison of Waterborne Petroleum Oils by Fluorescence Analysis[3]
D 4489 Practices for Sampling Waterborne Oils [3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D 1129.

4. Summary of Practice

4.1 Special types of sample containers and shipping containers are recommended. Samples may be of several types: tar balls, collected oil, oil-water mixtures, emulsions, and oil and water on collecting devices such as silanized glass cloth, TFE-fluorocarbon polymer, or other materials. Instructions are given for the care of samples to minimize changes due to autoxidation and microbial attack between the time of sampling and the time of analysis. Services available for transportation of samples are described.

5. Apparatus

5.1 *Sample Containers*—Borosilicate glass containers that have been thoroughly cleaned are preferable. All glass containers, new or used, must be thoroughly cleaned and washed prior to use. The cleaning steps consist of an initial wash with a warm aqueous detergent mixture followed by six hot tap water rinses, two rinses with reagent water, a rinse with reagent-grade acetone, and a final rinse with a solvent such as pentane, hexane, cyclohexane, dichloromethane, or chloroform followed by drying in a clean oven at 105°C or hotter for 30 min. If the glassware requires cleaning under field conditions, it should be washed with warm aqueous detergent followed by extensive water rinsing. A solvent rinse with acetone should be made, if possible, followed by lengthy air drying to remove residual solvent. (**Warning—**For safety reasons, the use of pentane, hexane, or cylcohexane is recommended over use of dichloromethane or carbon tetrachloride.)

NOTE 2—Hot reagent water rinses are advisable where hot tap water might reintroduce contamination.

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[1] This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.02.

[4] Discontinued; see *1993 Annual Book of ASTM Standards*, Vol 11.02.

***A Summary of Changes section appears at the end of this standard.**

5.1.1 Plastic containers are not acceptable since volatile hydrocarbons diffuse readily through many commercial plastic containers or may be absorbed into the plastic. In addition, the plasticizer may dissolve in the sample causing misleading results.

5.1.2 Metal containers usually should be avoided because the nickel and vanadium determinations could be invalidated by introduction of metal from the can.

5.1.3 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*—Proper choice of closures is critical to avoid contamination and to preserve sample. Use caps with aluminum-coated or TFE-fluorocarbon-coated cardboard inserts. Aluminum foil sheet should not be used. Inserts of TFE-fluorocarbon sheet, however, are acceptable.

5.3 *Refrigerator*, explosion-proof at about 4 to 5°C.

5.4 *Shipping Containers*—Sturdy cartons or wooden boxes should be used. These should be sufficiently large so the sample containers are adequately surrounded by absorbent packing material, such as vermiculite, sufficient to absorb the entire contents should breakage occur and be lined with a grease proof plastic bag.

NOTE 3—After Dec. 31, 1990, packagings used for shipment of dangerous goods such as oil must be tested for compliance with UN Performance Packaging Standards and certified by a marking applied by the packaging manufacturer.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society. [5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Acetone*.

6.3 *N-hexane, Mixed Hexanes, Cyclohexane, Pentane, Dichloromethane, or Chloroform*, spectroquality or equivalent high purity.

7. Sampling

7.1 Collect a representative sample in accordance with Practices D 4489.

7.2 Sample containers should be carefully prepared as described in 5.1.

8. Preservation of Samples

8.1 *Protection Against Autoxidation*—Treat the sample container to displace air and store in a dark area in a refrigerator. Nitrogen or carbon dioxide can be used as inert gases to displace air.

8.2 *Protection Against Microbial Attack*—Maintain refrigeration (4 to 5°C) once samples are received in the laboratory.

9. Shipment of Samples

9.1 The shipping of oil samples is regulated by both Department of Transportation (DOT) and United States Postal Service regulations.

NOTE 4—As of Sept. 18, 1988, the U.S. Postal Service regulations were amended to reflect implementation of the International Civil Aviation Organization (ICAO) Technical Instructions for the Safe Transport of Dangerous Goods by Air. Under these international regulations, the definition of flammable liquid is different from the U.S. DOT definition. Under ICAO rules, a Flammable Liquid—Class 3 is defined as a liquid having a flashpoint of 141°F (60.5°C) or less. In addition, the limited quantity provisions for flammable liquids under U.S. DOT regulations are not applicable under the ICAO rules.

NOTE 5—For more information about the legal requirements for packaging and shipping petroleum oils and other hazardous materials, refer to U.S. Postal Service Publication 52, "Acceptance of Hazardous, Restricted, or Perishable Matter," the Domestic Mail Manual, Part 124, "Nonmailable Matter—Articles and Substances; Special Mailing Rules," and the packaging requirements listed in the Domestic Mail Manual, Part 121.

9.2 Common carriers such as air express or air freight are often employed for transportaiton of oil spill samples. Consult the carrier for current packing and labelling requirements.

9.3 It is good practice to notify the receiving laboratory of shipment arrangements.

10. Procedure

10.1 The prescribed standard procedures for controlled field or laboratory conditions and recommended modification for emergency conditions are summarized in Table 1. Use this table with the specific sections of this practice to choose the proper measures for preservation of the specific waterborne oils under consideration.

11. Keywords

11.1 identification; oil spill; preservation; sample; shipment; storage; waterborne oil

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[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Procedures for the Preservation of Waterborne Oil Samples

Recommended Operation	Procedure for Emergency Conditions	Procedures for Controlled Field or Laboratory
Sample containers	Borosilicate glass jars for high boiling samples.	Borosilicate glass jars for high boiling samples.
Cleaning containers	Wash with warm aqueous detergent followed by water rinsing. Rinse with acetone, if available, followed by air drying.	Wash with warm aqueous detergent followed with hot tap water and reagent water rinses. Rinse with acetone, chloroform, and oven dry.
Closures	TFE-fluorocarbon sheet or TFE-fluorocarbon or aluminum-coated cardboard inserts.	TFE-fluorocarbon sheet or TFE-fluorocarbon or aluminum-coated cardboard inserts.
Protection against autoxidation	Store in dark.	Remove air with nitrogen or cabon dioxide. Store in dark in refrigerator.
Protection against microbial attack	Refrigerate to 4 to 5°C, if possible.	Samples should be stored in laboratory refrigerator at about 4 to 5°C.
Shipment of samples	Pack in ice, if possible, and label appropriately. Notify recipient.	Pack in ice, if possible, and label appropriately. Notify recipient.

SUMMARY OF CHANGES

This section identifies the location of selection changes to this practice that have been incorporated since the last issue. For the convenience of the user, Committee D-19 has highlighted those changes that may impact the use of this practice. This section may also include descriptions of the changes or reasons for the changes or both.

(*1*) Paragraph 5.1 includes the addition of alternative solvents for cleaning sample containers.

(*2*) Note 3, Note 4, and Note 5 contain information on changes to shipping regulations and references useful for determining current regulations in effect by the date of this revision.