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An American National Standard

Standard Practice for Identification of Waterborne Oils[1]

This standard is issued under the fixed designation D 3415; 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.

1. Scope*

1.1 This practice covers the broad concepts of sampling and analyzing waterborne oils for identification and comparison with suspected source oils. Detailed procedures are referenced in this practice. A general approach is given to aid the investigator in planning a program to solve the problem of chemical characterization and to determine the source of a waterborne oil sample.

1.2 This practice is applicable to all waterborne oils taken from water bodies, either natural or man-made, such as open oceans, estuaries or bays, lakes, rivers, smaller streams, canals; or from beaches, marshes, or banks lining or edging these water systems. Generally, the waterborne oils float on the surface of the waters or collect on the land surfaces adjoining the waters, but occasionally these oils, or portions, are emulsified or dissolved in the waters, or are incorporated into the sediments underlying the waters, or into the organisms living in the water or sediments.

1.3 This practice as presently written proposes the use of specific analytical techniques described in the referenced ASTM standards. As additional techniques for characterizing waterborne oils are developed and written up as test methods, this practice will be revised.

1.4 *This standard does not purport to address 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 3325 Practice for Preservation of Waterborne Oil Samples [3]

D 3326 Practice for Preparation of Samples for Identification of Waterborne Oils [3]

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 3650 Test Method for Comparison of Waterborne Petroleum Oils by Fluorescence Analysis [3]

D 4489 Practices for Sampling of Waterborne Oils [3]

D 4840 Guide for Sampling Chain of Custody Procedures [2]

D 5037 Test Method for Comparison of Waterborne Petroleum Oils by High Performance Liquid Chromatography[3]

D 5739 Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry[3]

E 620 Practice for Reporting Opinions of Technical Experts [4]

3. Terminology

3.1 *Definitions:*

3.1.1 *waterborne oil*—any oil, whether or not derived from petroleum, carried by a water system (for example, ocean, bay, lake, river, etc.) usually at the surface but occasionally emulsified or dissolved in the water. The waterborne oil can also be found on beaches or banks edging the water body, in the sediments underlying the water, or in the organisms living in the water or in the sediments.

3.2 For definitions of other terms used in this practice, refer to Terminology D 1129, and to Practices D 3325, D 3326, D 4489, and D 5739, and Test Methods D 3328, D 3650, and D 5037.

4. Significance and Use

4.1 Oil from one crude oil field is readily distinguishable from another, and differences in the makeup of oils from the same crude oil field can often be observed as well. Refined oils are fractions from crude oil stocks, usually derived from distillation processes. Two refined oils of the same type differ because of dissimilarities in the characteristics of their crude oil feed stocks as well as variations in refinery processes and any subsequent contact with other oils mixed in during transfer operations from residues in tanks, ships, pipes, hoses, etc.

[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] *Annual Book of ASTM Standards*, Vol 14.02.

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

Thus, all petroleum oils, to some extent, have chemical compositions different from each other.

4.2 Identification of a recovered oil is determined by comparison with known oils selected because of their possible relationship to the particular recovered oil, for example, suspected sources. Thus, samples of such known oils must be collected and submitted along with the unknown for analysis. Identification of the source of an unknown oil by itself cannot be made without comparison to a known oil. The principles of oil spill identification are discussed in Ref **(1)**.

4.3 Many similarities (within uncertainties of sampling, analysis and weathering) will be needed to establish the identity beyond a reasonable doubt. The analyses described will distinguish many, but not all samples. Examples of weathering of various classes of oils are included in Ref **(2)**.

4.4 This practice is a guide to the use of ASTM test methods for the analysis of oil samples for oil spill identification purposes. The evaluation of results from analytical methods and preparation of an Oil Spill Identification Report are discussed in this practice. Other analytical methods are described in Ref **(3)**.

4.5 A quality assurance program for oil spill identification is specified.

5. Plan for Identification of Waterborne Oils

5.1 *Sampling*—Collect a representative sample of oil according to Practice D 4489. Because of the wide variety of oils carried and used by shipping and because of the possibility of pollution also arising from industrial activity, samples of suspected source oils must be collected at this time so that comparisons can be made between the waterborne oil in question and the suspected source oils. Chain of Custody procedures, such as Practice D 4840, should be followed.

5.2 *Preservation of Sample*—Protect the waterborne oil, as well as the suspected source oils, against possible contamination or microbial degradation, or both, by proper preservation methods as described in Practice D 3325.

5.3 *Preparation of Sample*—Prepare the waterborne oil, as well as the quality control sample (described in 5.4) and any suspected source oils, as described in Practice D 3326.

5.4 *Quality Assurance Procedures*:

5.4.1 In addition to procedures specified within each test method to monitor instrument performance, a quality control sample is analyzed with samples for each spill case. This monitors both sample preparation and instrument performance.

5.4.2 Remove an aliquot of one of the samples for each case prior to sample preparation and treat it as a separate sample. This QC sample should be taken from a spill sample if there is sufficient sample volume to do so. Otherwise, a suspected source sample may be used.

5.4.3 Evaluate the data for the QC sample and its duplicate aliquot according to the guidelines described for each Test Method (refer to 5.5). If the data do not meet the criteria for a match, investigate the problem. If there is a problem with one instrument, the analysis must be repeated for that test method after the problem is corrected. If there is a sample preparation problem, the analysis should be repeated after the problem is identified.

5.5 *Analysis of Samples*:

5.5.1 Analyze spill samples as well as any suspected source oils, by gas chromatography (Test Method A or B of Test Methods D 3328) and by either infrared analysis (Test Method D 3414), or fluorescence spectroscopy (Test Method D 3650). Interpretation of the gas chromatograms and infrared or fluorescence spectra of the waterborne oil and the suspected source oils should provide information as to whether the waterborne oil is from a petroleum source, whether its carbon-number range is similar to distillate, residual, or crude oil, and whether it resembles any of the possible suspected source oils. If the waterborne oil is weathered, it may not be possible to determine if it is a crude oil or a residual oil by gas chromatography. Odor and physical appearance may help to determine if the waterborne oil is actually from a petroleum source. Distillation is not required.

5.5.2 For final identification with a possible source, the samples shall be analyzed by another method in addition to Test Method D 3328. Test Method D 3650 or Test Method D 3414, if there is sufficient sample to do so, may be used for simple cases without significant weathering. If weathering is significant, analysis should include Practice D 5739.

6. Evaluation of Analytical Data

6.1 Data interpretation in oil spill source identification is not straight forward. It is fundamentally different from that of quantitative chemical analyses, in that it involves a qualitative comparison of "fingerprints." The primary difference arises because of a complication brought about by the chemical alteration of a spilled oil. From the moment oil enters the environment, evaporation, dissolution, photochemical oxidation, biodegradation, and other processes begin to alter the oil's characteristics or "fingerprint." The combined effects of these processes are termed weathering and can significantly complicate data interpretation. The experienced oil spill analyst is familiar with the complexities of the weathering processes and their impact on the test methods, and is able to distinguish real differences between two oils from those apparent differences resulting from weathering alterations. Contamination of the spilled oil with other oils or chemical substances is another factor which may have to be considered. Interference from contaminants can usually be recognized as such and discounted when weighing the test results. However, at times, severe weathering or contamination, or both, can mask most of the inherent similarities between oils. In such cases, comparison of test results may be inconclusive. For such cases, biomarker analysis by GC/MS (Practice D 5739) is imperative and may give conclusive results when other methods do not.

6.2 For each standard method, results are specified as Match, Probable Match, Indeterminate and Non-Match. These categories represent standardized degrees of differences between the analyses of two oils. They are not intended, by themselves, to make a statement about the origin of the oil. The analyst interprets the results of all the tests in the light of experience and the existing body of knowledge about oil analysis, and draws conclusions about whether or not certain samples came from the same source. For example, evaporative weathering may affect the comparison of fluorescence or

infrared fingerprints more severely than the gas chromatograms. The overall conclusion should be consistent with the combined results.

7. Reporting Results

7.1 The results and overall conclusions should be reported for the combined results of the test method used. An accepted format similar to Practice E 620 may be used. Copies of the original data and the conditions for each test method should be appended to the report.

8. Keywords

8.1 oil analysis; oil identification; spilled oils; waterborne oils; weathering

REFERENCES

(**1**) Bentz, A. P., "Oil Spill Identification," *Analytical Chemistry*, Vol 48, 1976, pp 454A to 472A.

(**2**) Oil Spill Identification System, U.S. Coast Guard, CG-D-52-77, ADA044750, NTIS, Springfield, VA, June 1977.

(**3**) Butt, J. A., ed., *Characterization of Spilled Oil Samples*, published on behalf of The Institute of Petroleum, London, John Wiley and Sons, New York, NY, 1986.

SUMMARY OF CHANGES

This section identifies the location of selected 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*) The changes in this revision provide an overview of the principles of oil spill identification, reference existing standard methods for oil spill identification, and indicate options for use of the standard methods.