Standard Test Method for
Identification of Oils and Oil Acids in Solvent-Reducible Paints[1]

This standard is issued under the fixed designation D2245; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the identification of oils and oil acids in vehicles that have been separated from solvent-reducible paints. The test method is based on a gas chromatographic technique (of the methyl esters) applicable to products containing both saturated and unsaturated, animal and vegetable, unpolymerized or partially polymerized fatty acids having 8 to 20 carbon atoms.

1.2 This test method is not applicable to products containing fatty acids that have been polymerized or oxidized to such an extent that no characteristic monomeric fatty acids remain.

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:*[2]

D1398 Test Method for Fatty Acid Content of Alkyd Resins and Alkyd Resin Solutions (Withdrawn 2007)[3]

D1983 Test Method for Fatty Acid Composition by Gas-Liquid Chromatography of Methyl Esters (Withdrawn 2003)[3]

D2372 Practice for Separation of Vehicle From Solvent-Reducible Paints

D2800 Test Method for Preparation of Methyl Esters From Oils for Determination of Fatty Acid Composition by Gas-Liquid Chromatography (Withdrawn 2007)[3]

3. Summary of Test Method

3.1 This test method is based upon the differential migration and partitioning of constituent fatty acids in the form of vaporized methyl esters between a flowing gas phase and a supported liquid phase in a gas chromatographic column. The test method is based on isothermal operation of the gas chromatograph and a hot wire, thermal conductivity detector.

3.2 The test method consists in the separation of the vehicle from the paint by centrifugation, extraction of fatty acids from the vehicle after saponification, conversion of fatty acids and a measured addition of margaric acid (internal standard) into methyl esters, preparation of the gas chromatogram, and interpretation of the chromatogram. The amount of each monomeric fatty acid ester is calculated, totaled, subtracted from 100 % to yield polymerized fatty acids, reported as is, and interpreted by comparison with standards as being from specific oils or oil acids.

4. Significance and Use

4.1 This test method provides a procedure to identify the fatty acids present in the vehicle of a paint.

5. Apparatus

5.1 *Centrifuge,*

5.2 *Separatory Funnels,* with PTFE-fluorocarbon stopcocks.

5.3 *Gas Chromatograph and Accessories,* suitable for analysis of fatty acids as methyl esters (see Test Method D1983).

6. Reagent

6.1 *Hydroquinone.*

7. Calibration and Standardization

7.1 Establish optimum operating conditions on the gas chromatograph with known samples of methyl esters as described in Test Method D1983.

7.2 Prepare working standards by running known paints or vehicles through the procedure described in Section 8. Include

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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[2] 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] The last approved version of this historical standard is referenced on www.astm.org.

TABLE 1 Fatty Acid Composition of Oils Used in Paint Products[A]

Caster Oil, %	
Palmitic	2
Stearic	1
Oleic	7
Ricinoleic	87
Linoleic	3
Coconut Oil,%	
Caproic	trace
Caprylic	6
Capric	6
Lauric	44
Myristic	18
Palmitic	11
Stearic	6
Oleic	7
Linoleic	2
Linolenic	trace
Cottonseed Oil,%	
Capric	trace
Lauric	trace
Myristic	1
Myristoleic	trace
Palmitic	29
Palmitoleic	2
Stearic	4
Oleic	24
Linoleic	40
Arachidic	trace
Linseed Oil,%	
Palmitic	6
Palmitoleic	trace
Stearic	4
Oleic	22
Linoleic	16
Linolenic	52
Arachidic	trace
Gadoleic	trace
Menhaden Oil,%	
Lauric	trace
Myristic	7
Myristoleic	trace
Palmitic	16
Palmitoleic	16
Stearic	2
Oleic	15
Linoleic	7
Linolenic	2
Arachidonic	17
Clupanodonic	11
Nisinic	4
Shibic	1
Unidentified unsaturate	2
Oiticica Oil,%	
Palmitic	7
Stearic	5
Oleic	6
Licanic	78
Hydroxy acids	4
Perilla Oil,%	
Palmitic	7
Stearic	2
Oleic	13
Linoleic	14
Linolenic	64
Safflower Oil,%	
Myristic	trace
Myristoleic	trace
Palmitic	8
Palmitoleic	trace
Stearic	3
Oleic	13
Linoleic	75
Linolenic	1
Arachidic	trace
Gadoleic	trace
Soybean Oil,%	
Myristic	trace
Palmitic	11
Stearic	4
Oleic	25
Linoleic	51
Linolenic	9
Arachidic	trace
Gadoleic	trace
Tall Oil,[B] %	
Palmitic	5
Stearic	3
Oleic	46
Linoleic	41
Linolenic	3
Arachidic	2
Tung Oil,%	
Palmitic	4
Stearic	1
Oleic	8
Linoleic	4
Linolenic	3
Eleostearic	80

[A] The acids and percents presented in this table are taken from the "Composition and Constants of Fatty Acids" chart and used by permission of the Archer-Daniels-Midland Co.

[B] The percent rosin acids in tall oil may vary from 0 to 42 %, the percent terpenes from 0 to 13 %. Both variations depend on the grade and refining of the oil.

particularly compositions with chemical or structural modifications that might be expected to alter the fatty acid distribution or the apparent polymer content of the starting raw materials.

8. Procedure

8.1 Separate the vehicle from the paint by direct high-speed centrifuging (see Practice D2372).

8.2 Extract the fatty acids from the separated vehicle after saponification and removal of the dicarboxylate salts and unsaponifiable matter in accordance with Test Method D1398, but substitute separatory funnels with PTFE-fluorocarbon stopcocks when available. In cases involving unsaturated fatty acids, add a crystal or diethyl ether solution of hydroquinone (equivalent to less than 0.05 weight % of the fatty acids to the fatty acid fractions obtained in the Procedure Section, Method B, of Test Method D1398. Swirl the flask containing the fatty acids, some ether solvent, and the hydroquinone until the hydroquinone is well dispersed; evaporate off the remaining ether carefully under vacuum as described in Test Method D1398. Analyze immediately or store for only a limited time in a small tall form vial under nitrogen in a dark cool place.

8.3 Prepare methyl esters of the extracted fatty acids in accordance with the Procedure Section of Test Method D2800.

8.4 Determine the fatty acid composition in accordance with Test Method D1983. (See Appendix, Fig. X1.1, for a typical chromatogram prepared in accordance with Test Method D1983).

8.5 Compare the chromatogram or fatty acid composition, or both, with the chromatograms or fatty acid compositions, or both, of suspected known materials (See Table 1, for typical fatty acid compositions of oils used in paint products). Consider the content of specific fatty acids characteristic of specific oils. Consider the total saturates versus unsaturates and polymer content in relation to what the original starting oil or oil acids might have been.

9. Report

9.1 Report the type of oil or oil acid when the fatty acid distribution approximates a specific known distribution or combination, when the limit of the possibilities is known and when the polymer content can be explained. (See Appendix X1 for some of the considerations in interpreting the analysis results).

9.2 Even when the identification is positive, it is recommended that the actual percent distributions of monomeric fatty acids and the polymer content be reported. In very complex systems where the possible combinations are too numerous to allow an immediate identification, the percent breakdown figures should be recorded. Considered with other data that might subsequently be obtained, the fatty acid and polymer distribution can be important.

10. Precision

10.1 Single-oil types have been correctly identified in collaborative work for seven round-robin samples. Represented were four linseed types, three soya types, one fish oil type, and one coconut type.

11. Keywords

11.1 fatty acids; oils; oil acids; solvent-reducible paints

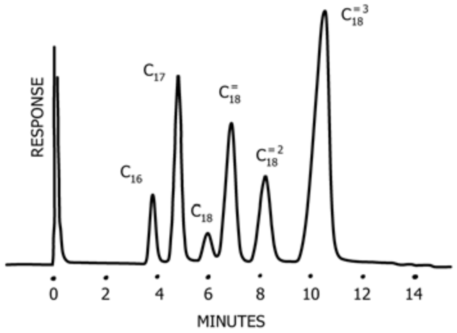
APPENDIX

(Nonmandatory Information)

X1. CONSIDERATIONS IN THE INTERPRETATION OF FATTY ACID COMPOSITION

X1.1 In the determination of the identity of pure natural oils, the percent breakdown of the fatty acids will many times suffice for oil identification. When a mixture of oil or oil-acid types is suspected, one can profit by comparing the content of key fatty acids. For example, a high oleate content in what otherwise appears to be a soya-type composition would suggest soya plus some tall oil. If, however, the palmitate appears a little high, one would conclude that some cottonseed had been added to the soya-type acids. Theoretically, if it can be established that the system under study is derived from a limited number of oil types on which exact fatty acid distributions are known, and polymerization or isomerization is not involved, a strictly mathematical approach to determining the quantity of each oil type is feasible. By means of a set of simultaneous equations involving key acids such as oleate, linoleate, linolenate, and stearate one should expect to be able to estimate mixtures of two or possibly three oil types.

X1.2 In the event that chromatographic analysis suggests the presence of polymer, it must be known that the polymer composition is due solely to oil polymer if the calculation of polymer content is to be of value in oil identification. In such cases the polymer content of the oil-ester sample plus the percent of the polyunsaturated acids obtained from the chromatogram (see Fig. X1.1) can be used to determine the total polyunsaturates present in the original oil. Since the value for total polyunsaturates varies with the oil in question, the experimental value obtained is used as additional evidence for oil identification.



80 % Linseed Type Fatty Acids
20 % Heptadecanoic Acid

FIG. X1.1 Drying-Oil Methyl Esters

X1.3 The presence of oil polymer composed of reaction products of oil acids with cyclo- or dicyclopentadiene, maleic anhydride, styrene vinyltoluene, or other Diels-Alder adducts, or the presence of rosin acids (in the case of tall oil fatty acids with high rosin content), negate the value of percent polymer determinations for assistance in oil identification. In most cases, the presence of these modifications can be detected by running infrared spectra on a portion of the separated fatty acids. As more work is done using the quantitative internal standard technique, it is expected that the data accumulated will aid everyone in interpreting what is involved in some of the observed polymer content results.

X1.4 Another more direct interference, which has not been mentioned, is the presence of carboxylic acid esters other than fatty acids. Acids such as isophthalic and benzoic acid are expected to be present in the fatty acid fraction due to the partial solubility of their potassium salts in the saponification medium. This is the main reason for using anhydrous reagents for the saponification of alkyds or polyesters. When unfamiliar peaks are observed in the chromatogram of fatty acid methyl esters from whole paint, this type of interference should be considered.

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