

# Standard Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints<sup>1</sup>

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  $(\varepsilon)$  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:<sup>2</sup>
- D1398 Test Method for Fatty Acid Content of Alkyd Resins and Alkyd Resin Solutions (Withdrawn 2007)<sup>3</sup>
- D1983 Test Method for Fatty Acid Composition by Gas-Liquid Chromatography of Methyl Esters (Withdrawn 2003)<sup>3</sup>
- 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for Preparation of Methyl Esters From Oils for Determination of Fatty Acid Composition by Gas-Liquid Chromatography (Withdrawn 2007)<sup>3</sup>

# 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 stop-cocks.
- 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

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;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

<sup>&</sup>lt;sup>3</sup> 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<sup>A</sup>

Caster Oil, %	Oiticica Oil,%			
Palmitic	2	Palmitic	,	7
Stearic	1	Stearic		5
Oleic	7	Oleic		6
Ricinoleic	87	Licanic		78
Linoleic	3	Hydroxy acids		4
Coconut Oil,%		,	Perilla Oil,%	
Caproic	trace	Palmitic	•	7
Caprylic	6	Stearic		2
Capric	6	Oleic		13
Lauric	44	Linoleic		14
Myristic	18	Linolenic		64
Palmitic	11		Safflower Oil,%	
Stearic	6	Myristic	,,,,	trace
Oleic	7	Myristoleic		trace
Linoleic	2	Palmitic		8
Linolenic	trace	Palmitoleic		trace
Cottonseed Oil,%		Stearic		3
Capric	trace	Oleic		13
Lauric	trace	Linoleic		75
Myristic	1	Linolenic		1
Myristoleic	trace	Arachidic		trace
Palmitic	29	Gadoleic		trace
Palmitoleic	2		Soybean Oil,%	
Stearic	4	Myristic	20,204 2,70	trace
Oleic	24	Palmitic		11
Linoleic	40	Stearic		4
Arachidic	trace	Oleic		25
Linseed Oil,%		Linoleic		51
Palmitic	6	Linolenic		9
Palmitoleic	trace	Arachidic		trace
Stearic	4	Gadoleic		trace
Oleic	22		Tall Oil, <sup>B</sup> %	
Linoleic	16	Palmitic	, , ,	5
Linolenic	52	Stearic		3
Arachidic	trace	Oleic		46
Gadoleic	trace	Linoleic		41
Menhaden Oil,%		Linolenic		3
Lauric	trace	Arachidic		2
Myristic	7		Tung Oil,%	
Myristoleic	trace	Palmitic		4
Palmitic	16	Stearic		1
Palmitoleic	16	Oleic		8
Stearic	2	Linoleic		4
Oleic	15	Linolenic		3
Linoleic	7	Eleostearic		80
Linolenic	2			
Arachidonic	17			
Clupanodonic	11			
Nisinic	4			
Shibic	1			
Unidentified unsaturate	2			

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.

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 stop-

cocks 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.

<sup>&</sup>lt;sup>B</sup> 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.