Designation: D2245 – 90 (Reapproved 2005)

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 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[3]

D1983 Test Method for Fatty Acid Composition by Gas-Liquid Chromatography of Methyl Esters[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[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*, high-speed, capable of developing in excess of 10 000 *g*.

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*.

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] Withdrawn.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

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 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