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AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Test Methods for Fatty and Rosin Acids in Tall Oil Fractionation Products by Capillary Gas Chromatography¹

This standard is issued under the fixed designation D 5974; 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 These test methods describe the determination of the amounts of the individual fatty acids and rosin acids in fractionated tall oil products using capillary gas chromatographic separation of the volatile methyl esters of these acids.

1.2 Three methods for forming the methyl esters and two methods for determining the amounts of the individual fatty acids and rosin acids are described.

1.3 The methods for forming the methyl esters are through the use of diazomethane, tetramethylammonium hydroxide (TMAH) or trimethylphenylammonium hydroxide (TMPAH).

1.4 The two methods for determining the amount of the individual fatty acids and rosin acids are the “internal standard” method, which yields absolute values and the “area percent” method, which yields relative values.

1.5 *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 509 Test Methods of Sampling and Grading Rosin²

D 804 Terminology Relating to Naval Stores, Including Tall Oil and Related Products²

D 3008 Test Method for Resin Acids in Rosin by Gas Chromatography²

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

3. Significance and Use

3.1 Tall oil fractionated products derived from tall oil are important commercial materials primarily composed of fatty acids and rosin acids but also containing some neutral material (see Terminology D 804). For many applications, it is necessary to know the level of the individual fatty acids and rosin acids present in these products. Gas chromatography has

proven to be a useful tool for such determinations (see Test Methods D 3008, and D 508) and capillary chromatography, described in these test methods, is considered to be the most effective analytical technique currently available. Other techniques may be more suitable than gas chromatography where particular situations exist. For example, the presence of fatty acid esters in the sample would result in transesterification during the derivatization step that may affect the results.

3.2 Due to hydrogen bonding, unmodified tall oil fatty acids and rosin acids cannot be volatilized at atmospheric pressure without undergoing decomposition, and so it is necessary to convert the free acids to the more volatile and more stable derivative, such as the methyl ester, prior to chromatographic separation.

3.3 These test methods describe three ways to prepare methyl esters. Other methylating derivatizing agents (for example, *N,N*-dimethylformamide dimethylacetal), may be used providing it has been demonstrated that such agents yield equivalent results to those described herein.

3.3.1 The classic method is the use of diazomethane. However, diazomethane is a hazardous and toxic material. It should only be used by, or under the supervision of, technically qualified individuals.

3.3.2 TMAH causes the isomerization of a sample's di- and polyunsaturated fatty acids, when it is used in even a slight excess. This leads to inaccurate results for the individual fatty acid components. TMAH should be used for materials containing only rosin acids, or when the identification or quantitation of individual fatty acid components is not important.

3.3.3 TMPAH is the recommended methylating agent when the identification or quantitation of individual di- and polyunsaturated fatty acids is required. TMPAH produces results that are very similar to those of diazomethane, but without the hazards that are normally associated with diazomethane. A considerable excess of TMPAH may cause isomerization of conjugated compounds similar to that encountered with TMAH.

3.4 Two test methods for calculating the amounts of the individual fatty acids and rosin acids are included in this standard. When the actual weight percentage of a given compound is required, the “internal standard” method must be used. This method involves adding a known amount of an internal standard to a known amount of test material and comparing the area of the peak associated with the internal

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.34 on Naval Stores.

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² Annual Book of ASTM Standards, Vol 06.03.

³ Annual Book of ASTM Standards, Vol 14.02.