



Designation: D 890 – 98

Standard Test Method for Water in Liquid Naval Stores¹

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1. Scope

1.1 These test methods cover the quantitative determination of dissolved or occluded water present in any proportion in liquid naval stores, such as turpentine, pinene, dipentene, pine oil, tall oil, and tall oil fatty acids. Three methods of moisture testing are included. The Karl Fisher titration method is the preferred method for testing tall oil, Test Methods D 803.

1.1.1 The Karl Fischer Titration method is based on the reaction between water and a complex reagent² consisting of iodine, sulfur dioxide, pyridine, and methanol, whereby the iodine is converted to a colorless compound. The appearance of a persistent iodine color in the reaction mixture indicates the complete removal of free water by reaction with the reagent, and the endpoint may be measured colorimetrically. Automatic titrators find this endpoint by the restoration of a current strength when the resistance provided by the presence of water is eliminated. Amperometric automatic titrators find this endpoint by detecting the current flow that occurs once water is eliminated.

1.1.2 The coulometric titration method determines water content by electronic integration of a current sufficient to generate the precise amount of iodine from the required reagent to react with the water in the sample.

1.1.3 The azeotropic method utilizes the relatively low boiling point of water, as compared with other sample constituents, in a toluene or xylene matrix so that water is collected in a trap and measured.

1.2 *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.*

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 This procedure has been adapted from the method of Karl Fischer published in *Zeitschrift für Angewandte Chemie*, Vol 48, 1935, p. 395; *Chemical Abstracts*, Vol 29, 1935, p. 6532; as modified by Smith, Bryant, and Mitchell, *Journal*, Am. Chemical Soc., Vol. 61, 1939, p. 2407; and further modified by Axel Johansson, *Svensk Papperstidning*, Vol 50, No. 11B, 1947, p. 124; see also Publication 19 of the Swedish Wood Research Institute, *Wood Chemistry and Paper Technique* (Stockholm) (1947). Karl Fischer reagent is available from various laboratory supplies. Pyridine-free adaptations of the Karl Fischer reagent are available commercially.

2. Referenced Documents

2.1 *ASTM Standards:*

D 803 Test Methods for Tall Oil³

D 1364 Test Method for Water in Volatile Solvents (Fischer Reagent Titration Method)⁴

3. Significance and Use

3.1 Many naval stores products contain water as a result of the processes used for their production. Typically refined products such as terpenes, pine oil, tall oil fatty acids, and distilled tall oil contain only traces of water, but crude tall oil might contain 0.5 to 2.5 % of water. Although the Karl Fischer and coulometric methods are most applicable to low levels of moisture, these can be and are used at higher levels. The azeotropic distillation method is generally used at higher levels.

Moisture By Karl Fischer Titration (Preferred method)

4. Apparatus

4.1 *Titration Vessel*, preferably closed, with stirring capabilities,

4.2 *Buret*, capable of being read at 0.1 mL divisions, or automatic buret, or

4.3 *Automatic Karl Fischer titrator*.

4.4 *Balance*, capable of weighing to the nearest 0.0001 g.

5. Reagents

5.1 *Karl Fischer Reagent, or Other Suitable Reagent, such as Pyridine-free Adaptations of Karl Fischer Reagent*. Reagents vary in strength (titer). This test method is written assuming a titer of 5 mg water/mL reagent. Recommended additions of water in this test method may need to be adjusted depending on the titer of the reagent.

5.2 *Methanol*—ACS grade.

6. Standardization of Iodine Reagent

6.1 Add methanol to the titration vessel, and titrate with reagent until the lemon-yellow color just changes to a red-brown color, or, if an automatic titrator is used, until the

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 06.04.