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Standard Test Methods for Sampling and Testing Plasticizers Used in Plastics¹

This standard is issued under the fixed designation D 1045; 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 cover sampling and testing of liquid plasticizers used in compounding of plastics. Acid number, ester content, specific gravity, color, refractive index, and water content are determined.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.* Specific hazards information is given in Section 5.

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:²

D 70 Test Method for Density of Semi-Solid Bituminous Materials (Pycnometer Method)

D 287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)

D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D 883 Terminology Relating to Plastics

D 1193 Specification for Reagent Water

D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)

D 1218 Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids

D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)

D 1600 Terminology for Abbreviated Terms Relating to Plastics⁴

~~D 1807 Test Methods for Refractive Index and Specific Optical Dispersion of Electrical Insulating Liquids— Terminology for Abbreviated Terms Relating to Plastics~~

~~D 3465 Practice— Test Method for Purity of Monomeric Plasticizers by Gas Chromatography~~

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter

D 5386 Test Method for Color of Liquids Using Tristimulus Colorimetry

E 1 Specification for ASTM Liquid-in-Glass Thermometers

E 203 Test Method for Water Using Volumetric Karl Fischer Titration

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminology D 883 and Terminology D 1600, unless otherwise indicated.

4. Significance and Use

4.1 These test methods ~~may~~^{shall} be used in establishing and confirming quality control standards for liquid plasticizers used in the compounding of plastics.

5. Hazards

5.1 *Chemical Hazard of Reagents*—~~Some~~[—]It is possible that some of the chemicals used in this test method may bear

⁴ These methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.15 on Thermoplastics Materials (Section D20.15.11 on Plasticizers):

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This edition contains changes in Section 1 to include an ISO equivalency statement.

¹ These methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials (Section D20.15.11 on Plasticizers):

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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* Vol 04.03, volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard.

hazardous. Accepted laboratory safety procedures must be followed. See suppliers' material safety data sheets for further information.

6. Sampling

6.1 The method of sampling specified in 6.2 or 6.3 shall be used, according to the special conditions that exist.

6.2 *From Loaded Tank Car or Other Large Vessel*—The composite sample taken shall be not less than 2 L ($\frac{1}{2}$ gal) ~~and should consist gal~~. It is important that the composite sample consists of small samples of not more than 1 L (1 qt) each, taken from near the top and bottom by means of a metal or glass container with removable stopper or top. This device, attached to a suitable pole, shall be lowered to the desired depth, when the stopper or top shall be removed and the container allowed to fill. A bomb sampler attached to a chain is convenient to use; adjust the opening ~~should be adjusted~~ so that the bomb will fill on the way down.

6.3 *From Barrels and Drums*—At least 5 % of the packages in any shipment shall be represented in the sample. ~~The~~ It is permissible for the purchaser may to increase the percentage of packages to be sampled at his discretion; in the case of plasticizers that are purchased in small quantity, ~~each package may be sampled~~ it is permissible to sample and analyzed; analyze each package, if desired. A portion shall be withdrawn from near the center of each package sampled by means of a "thief" or other sampling device and composited. The composite sample thus obtained shall be not less than 1 L (1 qt) and shall consist of equal portions of not less than 250 mL ($\frac{1}{2}$ pt) from each package sampled.

7. Purity of Reagents

7.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. ~~Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.~~³

7.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

8. Thermometers

8.1 All temperature measurements shall be made with ASTM thermometers of suitable range, accurate to within 0.1°C and conforming to the requirements prescribed in Specification E 1.

9. Reagents

9.1 *Alcohol*—Denatured alcohol, Formula No. 3A of the U. S. Bureau of Alcohol, Tobacco, and Firearms.

9.2 *Alkali, Standard Solution (0.01 N)*—Prepare and standardize a 0.01 N aqueous solution of sodium hydroxide (NaOH) or a 0.01 N alcoholic solution of potassium hydroxide (KOH).

9.3 *Alkali, Standard Solution (0.1 N)*—Prepare and standardize a 0.1 N aqueous solution of sodium hydroxide (NaOH) or a 0.1 N alcoholic solution of potassium hydroxide (KOH).

9.4 *Acetone*.

9.5 *Bromthymol Blue Indicator Solution*.

10. Procedure

10.1 Weigh 25 g of the sample into a 125-mL Erlenmeyer flask and dissolve in 50 mL of alcohol. If the sample is not completely soluble in alcohol, use 50 mL of a mixture of equal parts of alcohol and acetone. With certain samples it ~~may~~ will potentially be necessary first to add 25 mL of acetone, warm to effect solution, and then add 25 mL of alcohol.

10.2 Add a few drops of bromthymol blue indicator solution and titrate with 0.01 N NaOH or KOH solution. If the titration exceeds 10 mL, repeat the determination using 0.1 N NaOH or KOH solution.

10.3 *Blank*—Make a blank titration on 50 mL of the solvent used to dissolve the sample.

11. Calculation

11.1 Calculate the acid number, expressed as milligrams of KOH per gram of sample, as follows:

$$\text{Acid number} = [(A - B)N \times 56.1] / C \quad (1)$$

where:

³ *Annual Book of ASTM Standards*, Vol 05.01-Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.