



Designation: D 1045 – 95 (Reapproved 2001)

Standard Test Methods for Sampling and Testing Plasticizers Used in Plastics¹

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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:

- D 70 Test Method for Density of Semi-Solid Bituminous Materials Pycnometer Method²
- D 287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)³
- D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement⁴
- D 883 Terminology Relating to Plastics⁴
- D 1193 Specification for Reagent Water⁵
- D 1600 Terminology for Abbreviated Terms Relating to Plastics⁴
- D 1807 Test Methods for Refractive Index and Specific Optical Dispersion of Electrical Insulating Liquids⁶
- D 3465 Practice for Purity of Monomeric Plasticizers by Gas Chromatography⁷

¹ 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.

² *Annual Book of ASTM Standards*, Vol 04.03.

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ *Annual Book of ASTM Standards*, Vol 08.01.

⁵ *Annual Book of ASTM Standards*, Vol 11.01.

⁶ *Annual Book of ASTM Standards*, Vol 10.03.

⁷ *Annual Book of ASTM Standards*, Vol 08.02.

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter⁸

E 1 Specification for ASTM Thermometers⁹

E 203 Test Method for Water Using Volumetric Karl Fischer Titration¹⁰

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 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 of the chemicals used in this test method may be 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 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; 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 purchaser may 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 and

⁸ *Annual Book of ASTM Standards*, Vol 05.03.

⁹ *Annual Book of ASTM Standards*, Vol 14.03.

¹⁰ *Annual Book of ASTM Standards*, Vol 15.05.

analyzed, 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 (½ 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.¹¹ 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.

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

ACID NUMBER

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 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.

¹¹ *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.

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:

A = NaOH or KOH solution required for titration of the sample, mL,

B = NaOH or KOH solution required for titration of the blank, mL,

N = normality of the NaOH or KOH solution, and

C = sample used, g.

11.2 If desired, in the case of esters, the results may be expressed as a percentage by weight of the appropriate acid, by using the proper factor in the equation in 11.1.

ESTER CONTENT—TITRIMETRIC

12. Reagents

12.1 *Bromthymol Blue Indicator Solution*.

12.2 *Hydrochloric Acid, Standard (0.5 N)*—Prepare and standardize a 0.5 N aqueous solution of hydrochloric acid (HCl).

12.3 *Potassium Hydroxide, Standard Solution (0.5 N)*—Prepare and standardize a 0.5 N alcoholic solution of potassium hydroxide (KOH).

13. Procedure

13.1 Weigh accurately about 2 g of the sample into a 250-mL Erlenmeyer flask with ground-glass joint. By means of a constant delivery pipet or buret, add 50 mL of 0.5 N KOH solution. Connect to a water-cooled condenser with ground-glass joint and reflux for a period of 1 to 4 h, depending on the ester being tested, or until saponification is complete.

13.2 After the apparatus has cooled, wash down the condenser with water and disconnect. Add a few drops of bromthymol blue indicator solution to contents of the flask and titrate with 0.5 N HCl.

13.3 *Blank*—Run a blank, containing 50 mL of the 0.5 N KOH solution, along with the sample.

14. Calculation

14.1 Calculate the ester content, expressed in milligrams of KOH per gram of sample, as follows:

$$\text{Ester content} = [(D - E)N \times 56.1] / G - F \quad (2)$$

where:

D = HCl required for titration of the blank, mL,

E = HCl required for titration of the sample, mL,

F = correction for acidity of sample (Section 11),

N = normality of the HCl, and

G = sample used, g.

14.2 If desired, the results may be expressed as a percentage of the appropriate ester by weight, by using the proper factor in the equation in 14.1.