Designation: D 1045 – 95 (Reapproved 2001)

Standard Test Methods for Sampling and Testing Plasticizers Used in Plastics[1]

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:*

D 70 Test Method for Density of Semi-Solid Bituminous Materials Pycnometer Method[2]

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 Displacement[4]

D 883 Terminology Relating to Plastics[4]

D 1193 Specification for Reagent Water[5]

D 1600 Terminology for Abbreviated Terms Relating to Plastics[4]

D 1807 Test Methods for Refractive Index and Specific Optical Dispersion of Electrical Insulating Liquids[6]

D 3465 Practice for Purity of Monomeric Plasticizers by Gas Chromatography[7]

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter[8]

E 1 Specification for ASTM Thermometers[9]

E 203 Test Method for Water Using Volumetric Karl Fischer Titration[10]

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 (½ 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

[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.

[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.

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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.

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 (HCI).

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.

[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.

ESTER CONTENT—GAS CHROMATOGRAPHY

15. Procedure

15.1 The ester content may be determined using Practice D 3465.

SPECIFIC GRAVITY

16. Selection of Test Method

16.1 Specific gravity may be determined using a hydrometer (Test Method D 287), Westphal balance, pycnometer (Section 17) or Digital Density Meter (Test Method D 4052). Extremely viscous samples may be tested by Test Methods D 792 or Test Method D 70.

17. Procedure Using Pycnometer

17.1 Determine the weight capacity of the pycnometer with water at 23 ± 1°C. Fill this standardized pycnometer with a portion of the sample that has been cooled to approximately 20°C. Insert the thermometer or capillary tube, taking care to avoid introduction of air bubbles. Set the pycnometer in a constant-temperature water bath maintained at 23 ± 1°C (73.4 ± 1.8°F) for a period of at least 30 min. Remove the droplet of sample from the overflow capillary and cover with the glass cap. Clean the outside of the pycnometer and weigh.

18. Calculation

18.1 Calculate the specific gravity as follows:

$$\text{Specific gravity at } 23/23°C = A/B \quad (3)$$

where:
A = grams of sample used, and
B = water capacity of pycnometer in grams.

COLOR

19. Application

19.1 Useful comparisons between the color of relatively light colored plasticizers and platinum-cobalt standards may be made for colors in the range from 0 to 200, as defined in Section 20.

20. Preparation of Color Standards[12]

20.1 Dissolve 1.245 g of potassium chloroplatinate (K_2PtCl_6), containing 0.5 g of platinum, and 1 g of crystallized cobaltous chloride ($CoCl_2 \cdot 6H_2O$), containing about 0.248 g of cobalt, in water containing 100 mL of HCl (sp gr 1.19). Dilute to 1 L with water to prepare a solution having a color of 500. This solution, already prepared, may be purchased from laboratory supply houses.

20.2 To prepare standards having colors of 5, 10, 15, etc., dilute 0.5, 1.0, 1.5 mL, etc., of the solution described in 20.1 with water to 50 mL in standard Nessler tubes. If 100-mL standard Nessler tubes are used, dilute 1.0, 2.0, 3.0 mL, etc., to 100 mL with water to obtain colors of 5, 10, 15, etc. Protect the tubes from evaporation and from dust when not in use. If desired, commercially available platinum-cobalt standards in Nessler tubes or colored glass disks with proper spectral transmission may be used, provided they are first checked against standards prepared from K_2PtCl_6 and $CoCl_2 \cdot 6H_2O$ as described above.

21. Procedure

21.1 Fill a 50 or 100-mL Nessler tube with the sample and compare with the standards by holding side by side and looking down through the columns of liquid at a matte white surface illuminated by northern daylight.

22. Apparatus

22.1 *Refractometer*—An Abbé refractometer with scale graduated directly in terms of refractive index of the D line of sodium at a temperature of 23°C.

22.2 *Water Supply*—A water supply, the temperature of which may be varied.

REFRACTIVE INDEX

23. Procedure

23.1 The refractive index may be measured using the appropriate routine or precision method from Test Method D 1807 or the method given in Sections 22 and 24.

24. Procedure

24.1 Place the refractometer in front of a suitable source of light (either daylight or electric light), insert the thermometer, and adjust the circulation of water so as to bring the prisms to the desired temperature (usually 23°C (73.4°F)). Clean with alcohol and wipe dry. Spread a drop of the liquid to be tested upon the lower prism and clamp the prisms together. Adjust the mirror so that the light enters the telescope. Focus the eyepiece on the cross-hairs and the reading lens of the scale. Upon moving the prism arm, a position can now be found where the lower part of the field is dark and the upper part light. In general, the borderline will be colored. Correct by turning the milled head on the right of the telescope so that a sharp black and white edge is obtained. Move the prism arm until this black edge just crosses the intersection of the cross-hairs. Read the refractive index from the scale, estimating the fourth decimal place.

24.2 The accuracy of the instrument may be checked by a small test plate of known refractive index, which is supplied with the refractometer. Attach this test plate to the upper prism with a liquid of high refractive index (usually monobromonaphthalene). Errors may be corrected by means of a small adjusting screw.

WATER CONTENT

25. Procedure

25.1 The water content may be measured using Test Method E 203.

[12] The preparation of these platinum-cobalt color standards was originally described by Hazen, A., *American Chemical Journal*, Vol 14, 1892, 300. The description given in these methods is substantially identical with that given in the *Standard Methods for the Examination of Water and Sewage*. Am. Public Health Assn., Ninth Edition, p. 14. A description of these standards is also given by Scott, W. W., *Standard Methods of Chemical Analyses.* Fifth Edition, Vol 2, p. 2048.

26. Precision and Bias

26.1 Precision and bias statements are an integral part of referenced test procedures used in this test method. These statements are to be considered applicable to test results obtained by use of this test method.

26.2 Attempts to develop precision and bias statements for Acid Number, Ester Content-Titrimetric, Specific Gravity-Pycnometer, Color, and Refractive Index by the procedure in Section 22 have not been successful. For this reason, data on precision and bias cannot be given. Anyone wishing to participate in the development of precision and bias data should contact the ASTM headquarters.

27. Keywords

27.1 plasticizer; plasticizer acid number; plasticizer color; plasticizer ester content; plasticizer refractive index; sampling; test methods

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