



Designation: E 852 – 94a

Standard Test Methods for C₄–C₁₃ Plasticizer Grade Alcohols¹

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1. Scope

1.1 These test methods cover the chemical and physical analysis of liquid C₄ to C₁₃ alcohols that are manufactured for further processing into plasticizers. The key sections appear in the following order:

Procedure	Sections
Reagents	4
Specific Gravity	5 to 8
Acidity	9 to 12
Color	13 to 16
Water	17 to 20
Sulfuric Acid Color	21 to 26

NOTE 1—Test methods for carbonyl content and hydroxyl number will be added upon successful completion of interlaboratory studies.

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.*

2. Referenced Documents

2.1 ASTM Standards:

- D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals²
- D 1193 Specification for Reagent Water³
- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)⁴
- D 1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products⁴
- E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals²
- E 203 Test Method for Water Using Karl Fischer Reagent²

¹ These test methods are under the jurisdiction of ASTM Committee E-15 on Industrial Chemicals and are the direct responsibility of Subcommittee E15.53 on Alcohols and Polyalcohols.

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² *Annual Book of ASTM Standards*, Vol 15.05.

³ *Annual Book of ASTM Standards*, Vol 11.01.

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

3. Significance and Use

3.1 These test methods may be used with specifications for C₄ to C₁₃ alcohols to determine if the quality of the alcohol is suitable for use in making plasticizers.

4. Reagents

4.1 *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.⁵ 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.

4.2 Unless otherwise indicated, references to water shall be understood to mean Type II or III reagent water conforming to Specification D 1193.

SPECIFIC GRAVITY

5. Summary of Test Method

5.1 The specific gravity of the sample is determined at 20/20°C using the hydrometer method.

6. Procedure

6.1 Determine the specific gravity of the sample at 20/20°C using the hydrometer method in accordance with Test Methods D 891. The determination shall be made at 20.0 ± 0.1°C.

NOTE 2—For precise work the hydrometer must be calibrated at 20°C using liquids of known specific gravities at 20°C. This correction is generally less than 0.001 and may be ignored for most work. Hydrometers are normally calibrated at 60°F or 15.56°C.

⁵ *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.

7. Report

7.1 Report the specific gravity at 20/20°C to the nearest 0.0002 unit.

8. Precision and Bias

8.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 3):

8.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00014 unit at 50 degrees of freedom (DF). The 95 % limit for the difference between 2 such runs is 0.0004 unit.

8.1.2 *Laboratory Precision (Within-Laboratory, Between-Days)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.00024 unit at 25 DF. The 95 % limit for the difference between 2 such averages is 0.0007 unit.

8.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.00066 unit at 4 DF. The 95 % limit for the difference between 2 such averages is 0.0018 unit.

NOTE 3—The precision estimates in 8.1.1 to 8.1.3 are based on an interlaboratory study performed in 1981 on one sample each of *n*-butanol, *n*-hexanol, 2-ethylhexanol, decanol, and tridecanol whose specific gravities ranged from 0.8105 to 0.8370. A total of six laboratories cooperated in the study in which duplicate runs were performed on each of two days.⁶ Practice E 180 was used in developing these precision estimates.

8.2 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

ACIDITY

9. Summary of Test Method

9.1 The total acidity of the sample is measured by titrating the sample with standard caustic solution to the phenolphthalein end point, using isopropanol as the solvent. Any acidic moiety that will titrate under these conditions will be included in the total acidity value which is expressed as percent acetic acid.

10. Procedure

10.1 Determine the total acidity of the sample in accordance with Test Method D 1613 and isopropanol as the solvent.

11. Report

11.1 Report the acidity as acetic acid to the nearest 0.0001 weight %.

12. Precision and Bias

12.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 4):

12.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00049 % absolute at 60 DF. The 95 % limit for the difference between 2 such runs is 0.0014 % absolute.

12.1.2 *Laboratory Precision (Within-Laboratory, Between-Days)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.00034 % absolute at 30 DF. The 95 % limit for the difference between 2 such averages is 0.0010 % absolute.

12.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.00146 % absolute at 5 DF. The 95 % limit for the difference between 2 such averages is 0.0041 % absolute.

NOTE 4—The precision estimates in 12.1.1 to 12.1.3 are based on an interlaboratory study performed in 1981 on one sample each of *n*-butanol, *n*-hexanol, 2-ethylhexanol, decanol, and tridecanol whose acidities ranged from 0.00011 to 0.0092 % by weight as acetic acid. A total of six laboratories cooperated in the study in which duplicate runs were performed on each of two days.⁶ Practice E 180 was used in developing these precision estimates.

12.2 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

COLOR

13. Summary of Test Method

13.1 The color of the sample is visually determined by comparing the sample in a Nessler tube with a series of platinum-cobalt standards in matching Nessler tubes.

14. Procedure

14.1 Determine the color of the sample in accordance with Test Method D 1209.

15. Report

15.1 Report the color of the sample to the nearest 1 platinum-cobalt unit.

16. Precision and Bias

16.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 5):

16.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 1.1 units at 60 DF. The 95 % limit for the difference between 2 such runs is 3 units.

16.1.2 *Laboratory Precision (Within-Laboratory, Between-Days)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.37 unit at 30 DF. The 95 % limit for the difference between 2 such averages is 1 unit.

16.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 2.15 units at 5 DF. The 95 % limit for the difference between 2 such averages is 6 units.

NOTE 5—The precision estimates in 16.1.1 to 16.1.3 are based on an interlaboratory study performed in 1981 on one sample each of *n*-butanol, *n*-hexanol, 2-ethylhexanol, decanol, and tridecanol whose colors ranged from 3.8 to 7.6 platinum-cobalt units. A total of six laboratories cooperated in the study in which duplicate runs were performed on each of two days.

⁶ Supporting data are available from ASTM Headquarters. Request RR: E15-1031.