



Standard Test Methods for Monobasic Organic Acids¹

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1. Scope

1.1 These test methods cover procedures for the analysis of monobasic organic acid.

1.2 The analytical procedures appear in the following order:

	Sections
Freezing Point	7-10
Color	11-13
Specific Gravity	14-16
Water	17-19

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 statements are given in Section 6.*

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 1493 Test Method for Solidification Point of Industrial Organic Chemicals⁴

E 1 Specification for ASTM Thermometers⁵

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

3. Terminology

3.1 There are no terms in these test methods that require new or other than dictionary definitions.

4. Significance and Use

4.1 These test methods reference the analytical procedures that can be used to determine color, water, and specific gravity

¹ These test methods are under the jurisdiction of ASTM Committee E-15 on Industrial Chemicals and are the direct responsibility of Subcommittee E15.51 on Acids.

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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.

⁵ *Annual Book of ASTM Standards*, Vol 14.03.

of monobasic organic acids. In addition, a test method is outlined for the determination of freezing point (and subsequent assay determination) for acetic acid. The results of these procedures may be used to determine conformance to specification limits.

5. Purity of Reagents

5.1 *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.⁶ 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II or Type III reagent water conforming to Specification D 1193.

6. Hazards

6.1 Many organic acids are corrosive and can be extremely hazardous if improperly handled. Avoid personnel exposure. Consult supplier's material safety data sheet for specific hazard information.

FREEZING POINT

7. Scope

7.1 This test method covers the determination of the freezing point of glacial acetic acid. The purity of the acid is estimated from the freezing point by assuming that the only impurity present in the sample is water.

8. Procedure

8.1 Determine the freezing point of the sample as described in Test Method D 1493, using an ASTM Solidification Point Thermometer having a range from 0 to 30°C and conforming to the requirements for Thermometer 90C prescribed in Specification E 1.

⁶ *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 Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

8.2 Refer to Table 1 to estimate the purity of the sample from the freezing point.

9. Report

9.1 Report the freezing point of the sample to the nearest 0.05°C and the purity to the nearest 0.03 %.

10. Precision and Bias

10.1 The following criteria should be used for judging the acceptability of results (Note 1):

10.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be less than 0.001°C at 10 DF. The 95 % limit for the difference between two such runs is 0.05°C.

10.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, formerly called repeatability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.022°C at 5 DF. The 95 % limit for the difference between two such averages is 0.10°C.

10.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.083°C at 4 DF. The 95 % limit for the difference between two such averages is 0.25°C.

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

NOTE 1—These precision estimates are based on an interlaboratory study⁷ performed in 1964 on a single sample of acetic acid. Five laboratories analyzed the sample in duplicate on each of 2 days for a total of 20 analyses. Practice E 180 was used in developing these precision estimates.

COLOR

11. Procedure

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

⁷ Supporting data giving results of cooperative tests are available from ASTM Headquarters. Request RR:E15-0071.

TABLE 1 Purities of Acetic Acid Corresponding to Freezing Points

Freezing Point, °C	Acetic Acid, %	Freezing Point, °C	Acetic Acid, %
16.63	100.00	15.2	99.25
16.6	99.98	15.1	99.20
16.5	99.93	15.0	99.15
16.4	99.88	14.9	99.10
16.3	99.83	14.8	99.04
16.2	99.78	14.7	98.99
16.1	99.72	14.6	98.94
16.0	99.67	14.5	98.89
15.9	99.62	14.4	98.84
15.8	99.57	14.3	98.78
15.7	99.52	14.2	98.73
15.6	99.47	14.1	98.68
15.5	99.41	14.0	98.63
15.4	99.36		
15.3	99.30		

12. Report

12.1 Report the color of the sample to the nearest 5 platinum-cobalt units.

13. Precision and Bias

13.1 The following criteria should be used for judging the acceptability of results (Note 2):

13.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 1.1 platinum-cobalt units at 10 DF. The 95 % limit for the difference between two such runs is 5 units.

13.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, formerly called repeatability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 1.8 platinum-cobalt units at 5 DF. The 95 % limit for the difference between two such averages is 5 units.

13.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 2.2 platinum-cobalt units at 4 DF. The 95 % limit for the difference between two such averages is 10 units.

NOTE 2—These precision estimates are based on an interlaboratory study performed in 1964 on a sample of 2-ethylhexanoic acid with an average color of 25 platinum-cobalt units. One analyst in each of six laboratories performed duplicate determinations and repeated one day later for a total of 20 determinations.⁷ Practice E 180 was used in developing these precision estimates.

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

SPECIFIC GRAVITY

14. Procedure

14.1 Determine the specific gravity of the sample at 20/20°C using the hydrometer method as described in Test Methods D 891, except that the determination shall be made at 20.0 ± 0.1°C.

NOTE 3—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.

15. Report

15.1 Report the specific gravity at 20/20°C to the nearest 0.0005 unit.

16. Precision and Bias

16.1 The following criteria should be used for judging the acceptability of results (Note 4):

16.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00020 unit at 24 DF. The 95 % limit for the difference between two such runs is 0.0005 unit.

16.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, formerly called repeatability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be

0.00016 unit at 12 DF. The 95 % limit for the difference between two such averages is 0.0005 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 0.00057 at 5 DF. The 95 % limit for the difference between two such averages is 0.0015 unit.

NOTE 4—These precision estimates are based on an interlaboratory study⁷ performed in 1964 on one sample each of acetic acid and 2-ethylhexanoic acid. Six laboratories analyzed each sample in duplicate on each of two days for a total of 48 analyses. Practice E 180 was used in developing the precision statements.

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

WATER

17. Procedure

17.1 Determine the water content of the sample in accordance with Test Method E 203 (Note 5). Use a 10-g sample and Karl Fischer reagent having a strength of 2 to 5 mg of water per millilitre.

NOTE 5—For samples such as fumaric acid which are difficult to dissolve, a solvent mixture of 10 % dimethylformamide in methanol has been found suitable.

18. Report

18.1 Report the water content of the sample to the nearest 0.01 %.

19. Precision and Bias

19.1 The following criteria should be used for judging the

acceptability of results (Note 6):

19.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.004 % absolute at 38 DF. The 95 % limit for the difference between two such runs is 0.01 % absolute.

19.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, formerly called repeatability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.006 % absolute at 19 DF. The 95 % limit for the difference between two such averages is 0.02 % absolute.

19.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.018 % absolute at 6 DF. The 95 % limit for the difference between two such averages is 0.05 % absolute.

NOTE 6—These precision estimates are based on an interlaboratory study⁷ performed in 1964 on one sample each of acetic acid, 2-ethylhexanoic acid, and adipic acid. Seven laboratories analyzed the acetic acid, eight analyzed the 2-ethylhexanoic acid, and four analyzed the adipic acid in duplicate on each of two days for a total of 76 analyses. Practice E 180 was used in developing these precision estimates.

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

20. Keywords

20.1 acetic acid; color; freezing point; glacial acetic acid; monobasic organic acids; purity; specific gravity; water

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