

Dehydrated Alcohol

Portions of this monograph that are national *USP* text, and are not part of the harmonized text, are marked with symbols (♦) to specify this fact.



C₂H₆O 46.07
 Ethanol;
 Ethyl alcohol [64-17-5].

DEFINITION

♦Dehydrated Alcohol contains NLT 99.2% by weight, corresponding to NLT 99.5% by volume, at 15.56°, of C₂H₅OH.♦

IDENTIFICATION

- **A.** It meets the requirements of the test for *Specific Gravity* (841).
- **B. INFRARED ABSORPTION** (197S) or (197F): Neat

IMPURITIES

LIMIT OF NONVOLATILE RESIDUE

Sample: 100 mL of Dehydrated Alcohol
Analysis: Evaporate the *Sample* in a tared dish on a water bath, and dry at 100°–105° for 1 h.
Acceptance criteria: The weight of the residue is NMT 2.5 mg.

ORGANIC IMPURITIES

Sample solution A: Substance to be examined
Sample solution B: 300 µL/L of 4-methylpentan-2-ol in *Sample solution A*
Standard solution A: 200 µL/L of methanol in *Sample solution A*
Standard solution B: 10 µL/L of methanol and 10 µL/L of acetaldehyde in *Sample solution A*
Standard solution C: 30 µL/L of acetal in *Sample solution A*
Standard solution D: 2 µL/L of benzene in *Sample solution A*

Chromatographic system

(See *Chromatography* (621), *System Suitability*.)

Mode: GC

Detector: Flame ionization

Column: 0.32-mm × 30-m fused-silica capillary; bonded with a 1.8-µm layer of phase G43

Split ratio: 20:1

Temperatures

Injection port: 200°

Detector: 280°

Column: See *Table 1*.

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
40	0	40	12
40	10	240	10

Flow rate: 35 cm/s
Carrier gas: Helium
Injection volume: 1.0 µL

System suitability

Sample: *Standard solution B*

Suitability requirements

Resolution: NLT 1.5 between the first major peak (acetaldehyde) and the second major peak (methanol)

Analysis

Samples: *Sample solution A*, *Sample solution B*, *Standard solution A*, *Standard solution B*, *Standard solution C*, and *Standard solution D*

Methanol calculation

$$\text{Result} = r_U/r_S$$

r_U = peak area of methanol from *Sample solution A*
 r_S = peak area of methanol from *Standard solution A*

Acetaldehyde calculation (sum of acetaldehyde and acetal)

$$\text{Result} = \left\{ \frac{A_E}{(A_T - A_E)} \times C_A \right\} + \left\{ \frac{D_E}{(D_T - D_E)} \times C_D \times \left(\frac{M_{r1}}{M_{r2}} \right) \right\}$$

A_E = peak area of acetaldehyde from *Sample solution A*

A_T = peak area of acetaldehyde from *Standard solution B*

C_A = concentration of acetaldehyde in *Standard solution B* (µL/L)

D_E = peak area of acetal from *Sample solution A*

D_T = peak area of acetal from *Standard solution C*

C_D = concentration of acetal in *Standard solution C* (µL/L)

M_{r1} = molecular weight of acetaldehyde, 44.05

M_{r2} = molecular weight of acetal, 118.2

Benzene calculation

$$\text{Result} = \left[\frac{B_E}{(B_T - B_E)} \right] \times C_B$$

B_E = peak area of benzene from *Sample solution A*

B_T = peak area of benzene from *Standard solution D*

C_B = concentration of benzene in *Standard solution D* (µL/L)

[NOTE—If necessary, the identity of benzene can be confirmed using another suitable chromatographic system (stationary phase with a different polarity).]

Any other impurity calculation

$$\text{Result} = (r_U/r_M) \times C_M$$

r_U = peak area of each impurity from *Sample solution B*

r_M = peak area of 4-methylpentan-2-ol from *Sample solution B*

C_M = concentration of 4-methylpentan-2-ol in *Sample solution B* (µL/L)

Acceptance criteria: See *Table 2*.

Table 2

Name	Acceptance Criteria
Methanol	NMT 0.5, corresponding to 200 µL/L
Acetaldehyde and acetal	NMT 10 µL/L, expressed as acetaldehyde

^a Disregard any peaks of less than 9 µL/L (0.03 times the area of the peak corresponding to 4-methylpentan-2-ol in the chromatogram obtained with *Sample solution B*).

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Table 2 (Continued)

Name	Acceptance Criteria
Benzene	NMT 2 µL/L
Sum of all other impurities ^a	NMT 300 µL/L

^a Disregard any peaks of less than 9 µL/L (0.03 times the area of the peak corresponding to 4-methylpentan-2-ol in the chromatogram obtained with *Sample solution B*).

SPECIFIC TESTS

- **SPECIFIC GRAVITY (841):** NMT 0.7962 at 15.56°, indicating NLT 99.2% of C₂H₅OH by weight.

Change to read:

• **ULTRAVIOLET ABSORPTION**

Analytical wavelength: 235–340 nm
Cell: 5 cm
Reference: Water
Acceptance criteria
Absorbance: NMT 0.40 at 240 nm; NMT 0.30 between 250 and 260 nm; NMT 0.10 between 270 and 340 nm
Curve: The spectrum shows a steadily descending curve with no observable peaks or shoulders.

• **CLARITY OF SOLUTION**

[NOTE—The *Sample solution* is to be compared to *Standard suspension A* and to water in diffused daylight 5 min after preparation of *Standard suspension A*.]

Hydrazine solution: 10 mg/mL of hydrazine sulfate in water. Allow to stand for 4–6 h.

Methenamine solution: Transfer 2.5 g of methenamine to a 100-mL glass-stoppered flask, add 25.0 mL of water, insert the glass stopper, and mix to dissolve.

Primary opalescent suspension: Transfer 25.0 mL of *Hydrazine solution* to the *Methenamine solution* in the 100-mL glass-stoppered flask. Mix, and allow to stand for 24 h. This suspension is stable for 2 months, provided it is stored in a glass container free from surface defects. The suspension must not adhere to the glass and must be well mixed before use.

Opalescence standard: Transfer 15.0 mL of the *Primary opalescent suspension* to a 1000-mL volumetric flask, and dilute with water to volume. This suspension should not be used beyond 24 h after preparation.

Standard suspension A: Dilute 5.0 mL of the *Opalescence standard* with water to 100.0 mL.

Standard suspension B: Dilute 10.0 mL of the *Opalescence standard* with water to 100.0 mL.

Sample solution A: Substance to be examined

Sample solution B: 1.0 mL of *Sample solution A* diluted with water to 20 mL. Allow to stand for 5 min before testing.

Blank: Water

Analysis

Samples: *Standard suspension A*, *Standard suspension B*, *Sample solution A*, *Sample solution B*, and *Blank*

Transfer a sufficient portion of *Sample solution A* and *Sample solution B* to separate test tubes of colorless, transparent, neutral glass with a flat base and an internal diameter of 15–25 mm to obtain a depth of 40 mm. Similarly transfer portions of *Standard suspension A*, *Standard suspension B*, and *Blank* to separate matching test tubes. Compare samples in diffused daylight, viewing vertically against a black background (see *Spectrophotometry and Light-Scattering (851)*, *Visual Comparison*). The diffusion of light must be such that *Standard suspension A* can be readily distinguished from water, and *Standard suspension B* can be readily distinguished from *Standard suspension A*.

Acceptance criteria: *Sample solution A* and *Sample solution B* show the same clarity as that of water, or their opalescence is not more pronounced than that of *Standard suspension A*.

• **ACIDITY OR ALKALINITY**

Phenolphthalein solution: Dissolve 0.1 g of phenolphthalein in 80 mL of alcohol, and dilute with water to 100 mL.

Sample: 20 mL of Dehydrated Alcohol

Analysis: To the *Sample* add 20 mL of freshly boiled and cooled water and 0.1 mL of *Phenolphthalein solution*. The solution is colorless. Add 1.0 mL of 0.01 N sodium hydroxide.

Acceptance criteria: The solution is pink (30 µg/g, expressed as acetic acid).

• **COLOR OF SOLUTION**

Standard stock solution: Combine 3.0 mL of ferric chloride CS, 3.0 mL of cobaltous chloride CS, 2.4 mL of cupric sulfate CS, and 1.6 mL of dilute hydrochloric acid (10 mg/mL).

Standard solution: 1.0 mL of *Standard stock solution*, diluted with dilute hydrochloric acid (10 mg/mL) to 100 mL. Prepare the *Standard solution* immediately before use.

Sample solution: Substance to be examined

Blank: Water

Analysis

Samples: *Standard solution*, *Sample solution*, and *Blank*
 Transfer a sufficient portion of each of the *Samples* to individual test tubes of colorless, transparent, neutral glass with a flat base and an internal diameter of 15–25 mm to obtain a depth of 40 mm. Compare the *Samples* in diffused daylight, viewing vertically against a white background (see *Spectrophotometry and Light-Scattering (851)*, *Visual Comparison*).

Acceptance criteria: The *Sample solution* has the appearance of water or is not more intensely colored than the *Standard solution*.

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers, protected from light.
- **USP REFERENCE STANDARDS (11)**
 USP Dehydrated Alcohol RS