Dehydrated Alcohol

Add the following:

■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.■1S (*USP37*)

C_2H_6O 46.07
Ethanol;
Ethyl alcohol [64-17-5].

DEFINITION

Change to read:

■◆■1S (*USP37*)Dehydrated Alcohol contains NLT 99.2% by weight, corresponding to NLT 99.5% by volume, at 15.56°, of C_2H_5OH.■◆■1S (*USP37*)

IDENTIFICATION

Change to read:

- **A.** ■It meets the requirements of the test for *Specific Gravity* ⟨841⟩.■1S (*USP37*)
- **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.

Change to read:

- **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■1S (*USP37*) 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■1S (*USP37*)
  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)

$$\text{Result} = \{[A_E/(A_T - A_E)] \times C_A\} + \{[D_E/(D_T - D_E)] \times C_D \times (M_{r1}/M_{r2})\}$$

■1S (*USP37*)

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■1S (*USP37*)
M_{r2} = ■ molecular weight of acetal, 118.2■1S (*USP37*)

  Benzene calculation

$$\text{Result} = (B_E/(B_T - B_E)) \times C_B$$

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
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*).■1S (*USP37*)

SPECIFIC TESTS

Change to read:

• ■◆■1S (*USP37*)**Specific Gravity ⟨841⟩:** NMT 0.7962 at 15.56°, indicating NLT 99.2% of C_2H_5OH by weight■◆ ■1S (*USP37*)

Change to read:

• **Ultraviolet Absorption**
Analytical wavelength: ■235–340 nm■1S (*USP37*)
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: Smooth between 235 and 340 nm

Change to read:

• ■◆■1S (*USP37*)**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*.■◆■1S (*USP37*)

• **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).

Change to read:

• ■◆■1S (*USP37*)**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*.■◆■1S (*USP37*)

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers, protected from light.

- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Dehydrated Alcohol RS