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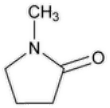
Methylpyrrolidone



C_5H_9NO 99.1

1-Methyl-2-pyrrolidinone;
N-Methyl-2-pyrrolidone;
N-Methylpyrrolidone;
1-Methyl-2-pyrrolidone;
Pyrrolidin, 1-methyl-2-one-;
1-Methylpyrrolidin-2-one;
N-Methyl-γ-butyrolactam;
N-Methyl tetrahydropyrrolone;
1-Methyl-2-oxopyrrolidine;
N-Methyl-1-oxotetramethyleneamine;
2-Methyl-2-azacyclopentanone CAS RN®: 872-50-4.

IDENTIFICATION

Change to read:

• **A.** ▲Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: **197F**▲ (CN 1-May-2020)

IMPURITIES

• **Organic Impurities**

Standard solution: To 1 mL of USP Methylpyrrolidone RS, add 1 mL of pyrrolidone, and dilute with methylene chloride to 20 mL.

Sample solution: Methylpyrrolidone (neat)

Chromatographic system

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

Mode: GC

Detector: Flame ionization

Column: 0.32-mm × 30-m fused-silica capillary; 5-µm layer of phase G2

Temperatures

Injector: 280°

Detector: 280°

Column: See *Table 1*.

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
100	—	100	0
100	3	170	30

Carrier gas: Nitrogen

Linear velocity: 20 cm/s

Injection type: Split ratio about 100:1

Injection volume: 1 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Resolution: NLT 2.0 between pyrrolidone and methylpyrrolidone

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each impurity, excluding any solvent peaks and peaks NMT 0.02%, in the portion of Methylpyrrolidone taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak response of each individual impurity from the *Sample solution*

r_T = sum of the responses of all the peaks from the *Sample solution*

Acceptance criteria: NMT 0.1% of any individual impurity; and NMT 0.3% of total impurities

SPECIFIC TESTS

• **Alkalinity**

Bromothymol blue solution: Dissolve 50 mg of bromothymol blue in a mixture of 4 mL of 0.02 M sodium hydroxide and 20 mL of alcohol, and dilute with water to 100 mL.

Sample: Methylpyrrolidone (neat)

Analysis: Add 0.5 mL of *Bromothymol blue solution* as indicator to 50 mL of water, and adjust with 0.02 M potassium hydroxide or 0.02 M hydrochloric acid until a yellow color is obtained. Add 50 mL of the *Sample*. Titrate with 0.02 M hydrochloric acid to the initial coloration.

Acceptance criteria: NMT 8.0 mL of 0.02 M hydrochloric acid is required.

Change to read:

• **Clarity of Solution**

[Note—The *Sample* is to be compared to the *Reference suspension* in diffused daylight 5 min after preparation of the *Reference suspension*.]

Hydrazine solution: 10 mg/mL of hydrazine sulfate. [Note—Allow to stand 4–6 h before use.]

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

[Note—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.]

Transfer 25.0 mL of the *Hydrazine solution* to the *Methenamine solution* in the 100-mL glass-stoppered flask. [Note—Allow to stand for 24 h.]

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

Reference suspension: Transfer 5.0 mL of the *Opalescence standard* to a 100-mL volumetric flask, and dilute with water to volume.

Sample: Methylpyrrolidone (neat)

Analysis: Transfer a sufficient portion of the *Sample* to a test tube 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 the *Reference suspension* and water to separate matching test tubes. Compare the *Sample*, *Reference suspension*, and water in diffused daylight, viewing vertically against a black background (see ▲*Visual Comparison ⟨630⟩*▲ (CN 1-May-2019)). [Note—The diffusion of light must be such that the *Reference suspension* can readily be distinguished from water.]

Acceptance criteria: The *Sample* shows the same clarity as that of water, or its opalescence is not more pronounced than that of the *Reference suspension*.

Change to read:

• **Color of Solution**

Comparison solution: Mix 3.0 mL of ferric chloride CS, 3.0 mL of cobaltous chloride CS, and 2.4 mL of cupric sulfate CS with 0.3 N hydrochloric acid to make 10 mL. Dilute 1.0 mL of this solution with 0.3 N hydrochloric acid to make 100 mL. [Note—Prepare and use this solution immediately.]

Sample: Methylpyrrolidone (neat)

Analysis: Transfer a sufficient portion of the *Sample* to a test tube 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 a portion of the *Comparison solution* to a separate matching test tube. Compare the color of the *Sample* with that of the *Comparison solution* in diffused daylight, viewing vertically against a white background (see ▲*Visual Comparison ⟨630⟩*▲ (CN 1-May-2019)).

Acceptance criteria: The *Sample* is not more intensely colored than the *Comparison solution*.

• **Water Determination, *Method Ic* ⟨921⟩:** NMT 0.1%, determined on 1.0 g

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in light-resistant containers.

• **USP Reference Standards ⟨11⟩**

USP Methylpyrrolidone RS

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
METHYLPYRROLIDONE	Documentary Standards Support	SE2020 Simple Excipients

Chromatographic Database Information: Chromatographic Database

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