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Benzaldehyde



C_H_O 106.12

Benzoic aldehyde;

Phenyl formaldehyde CAS RN[®]: 100-52-7.

DEFINITION

Benzaldehyde contains NLT 98.0% and NMT 102.0% of benzaldehyde (C₇H₆O).

IDENTIFICATION

Change to read:

• A. <u>Spectroscopic Identification Tests (197), Infrared Spectroscopy:</u> 197F_{▲ (CN 1-May-2020)}

ASSAY

• PROCEDURE

Solution A: Acetonitrile and glacial acetic acid (1000:1, v/v) **Solution B:** Glacial acetic acid and water (1:1000, v/v)

Mobile phase: See <u>Table 1</u>.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	18	82
20	60	40
30	60	40

System suitability solution: 0.1 mg/mL of USP Benzoic Acid RS and 0.06 mg/mL of USP Methylparaben RS in acetonitrile

 $\textbf{Standard solution:} \ 0.15 \ \text{mg/mL of} \ \underline{\text{USP Benzaldehyde RS}} \ \text{in acetonitrile}$

Sample solution: 0.15 mg/mL of Benzaldehyde in acetonitrile

Chromatographic system

(See Chromatography (621), System Suitability.)

Mode: LC

Detector: UV 235 nm

Column: 4.6-mm × 25-cm; 5-µm packing L1

Flow rate: 1.2 mL/min Injection volume: 10 μL System suitability

Samples: System suitability solution and Standard solution

Suitability requirements

Resolution: NLT 2.0 between benzoic acid and methylparaben, *System suitability solution* **Tailing factor:** NMT 2.0 for benzoic acid and methylparaben, *System suitability solution*

Relative standard deviation: NMT 2.0% for benzaldehyde, Standard solution

Analysis

Samples: Standard solution and Sample solution

Calculate the percentage of benzaldehyde (C,H,O) in the portion of Benzaldehyde taken:

Result =
$$(r_{ij}/r_{s}) \times (C_{s}/C_{ij}) \times 100$$

 r_{ij} = peak response of benzaldehyde from the Sample solution

 $r_{\rm s}$ = peak response of benzaldehyde from the Standard solution

C_s = concentration of <u>USP Benzaldehyde RS</u> in the Standard solution (mg/mL)

C, = concentration of Benzaldehyde in the Sample solution (mg/mL)

Acceptance criteria: 98.0%-102.0%

IMPURITIES

• LIMIT OF HYDROCYANIC ACID

Sample solution: Shake 0.5 mL of Benzaldehyde with 5 mL of water, add 0.5 mL of 1 N sodium hydroxide and 0.1 mL of ferrous sulfate TS, and warm the mixture gently.

Acceptance criteria: Upon the addition of a slight excess of hydrochloric acid, no greenish-blue color or blue precipitate is produced within 15 min.

• LIMIT OF NITROBENZENE

Sample solution: Dissolve 1 mL of Benzaldehyde in 20 mL of alcohol, and mix with 10 mL of water.

Analysis: Add 1-g portions of zinc and 1-mL portions of 2 N sulfuric acid, as needed, to maintain a brisk evolution of hydrogen for 1 h. Filter, evaporate the liquid to 20 mL, and boil 10 mL of the concentrated liquid with 1 drop of potassium dichromate TS.

Acceptance criteria: No purplish color is produced.

• CHLORINATED COMPOUNDS

Analysis: Wind a strip of 20-mesh copper gauze 1.5 cm wide and 5 cm long around the end of a copper wire. Heat the gauze in the nonluminous flame of a Bunsen burner until it glows without coloring the flame green. Permit the gauze to cool, and heat several times until a thick coat of oxide has formed. With a medicine dropper, apply 2 drops of Benzaldehyde to the cooled gauze, ignite, and permit it to burn freely in the air. Again cool the gauze, add 2 more drops of Benzaldehyde, and burn as before. Repeat this process until a total of 6 drops have been added and ignited. Then hold the gauze in the outer edge of the Bunsen flame, adjusted to a height of 4 cm.

Acceptance criteria: Not even a transient green color is imparted to the flame.

. LIMIT OF ETHYLBENZENE, CYCLOHEXYLMETHANOL, BENZYL ALCOHOL, AND BENZOIC ACID

Sample solution: Neat Benzaldehyde

Standard solution: 0.1% of <u>USP Ethylbenzene RS</u>, 0.1% of <u>USP Cyclohexylmethanol RS</u>, 0.2% of <u>USP Benzoic Acid RS</u>, and 0.2% of <u>USP</u>

Benzaldehyde RS in USP Benzyl Alcohol RS

Chromatographic system

(See Chromatography (621), System Suitability.)

Mode: GC

Detector: Flame ionization

Column: 0.32-mm × 30-m fused silica capillary, coated with a 0.5-µm layer of phase G16

Temperatures

Detector: 310°
Injection port: 200°
Column: See <u>Table 2</u>.

Table 2

Initial	Temperature	Final	Hold Time at Final	
Temperature	Ramp	Temperature	Temperature	
(°)	(°/min)	(°)	(min)	
50	5	220		

Carrier gas: Helium
Flow rate: 1.2 mL/min
Injection volume: 0.1 µL
Injection type: Splitless injection

System suitability

Sample: Standard solution

[Note—For relative retention times, see <u>Table 3</u>.]

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Component	Relative Retention Time
Ethylbenzene	0.45
Benzaldehyde	1.00
Cyclohexylmethanol	1.03
Benzyl alcohol	1.45
Benzoic acid	2.04

System suitability requirements

Resolution: NLT 3.0 between benzaldehyde and cyclohexylmethanol

Analysis

Samples: Standard solution and Sample solution

Calculate the percentage of each impurity in the portion of Benzaldehyde taken:

Result =
$$(r_{II}/r_{T}) \times 100$$

 r_{ii} = peak response of each impurity from the Sample solution

 r_{τ} = sum of all the peak responses from the Sample solution

Acceptance criteria

Each individual impurity: NMT 1.0% Total impurities: NMT 2.0%

SPECIFIC TESTS

• Water Determination, Method I (921): NMT 1.5%

ADDITIONAL REQUIREMENTS

- PACKAGING AND STORAGE: Preserve in well-filled, tight, light-resistant containers. Store at room temperature, and avoid exposure to excessive heat.
- USP Reference Standards $\langle 11 \rangle$

USP Benzaldehyde RS

USP Benzoic Acid RS

USP Benzyl Alcohol RS

USP Cyclohexylmethanol RS

USP Ethylbenzene RS

USP Methylparaben RS

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

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

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:

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