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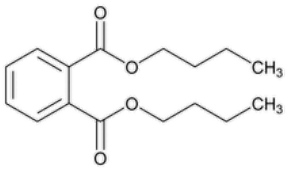
Dibutyl Phthalate



$C_{16}H_{22}O_4$ 278.34

1,2-Benzenedicarboxylic acid, dibutyl ester CAS RN®: 84-74-2.

DEFINITION

Dibutyl Phthalate contains NLT 99.0% and NMT 101.0% of dibutyl phthalate ($C_{16}H_{22}O_4$).

IDENTIFICATION

Change to read:

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

• **B.** It meets the requirements in *Specific Tests* for Refractive Index ⟨831⟩.

ASSAY

• **Procedure**

Sample: 0.75 g

Titrimetric system

(See *Titrimetry* ⟨541⟩.)

Mode: Residual titration

Titrant: 0.5 N alcoholic potassium hydroxide VS

Back-titrant: 0.5 N hydrochloric acid VS

Endpoint detection: Visual

Analysis: Transfer the *Sample* to a flask, add 25.0 mL of *Titrant*, attach a reflux condenser to the flask, and boil in a water bath for 1 h. Add 1 mL of phenolphthalein TS. Titrate immediately with *Back-titrant*. Perform a blank determination. Each mL of *Titrant* is equivalent to 69.59 mg of dibutyl phthalate ($C_{16}H_{22}O_4$).

Acceptance criteria: 99.0%–101.0%

IMPURITIES

• Residue on Ignition ⟨281⟩

Sample: 1.0 g

Acceptance criteria: NMT 0.1%

• **Related Compounds**

Internal standard solution: 3 mg/mL of dibenzyl in methylene chloride

Sample stock solution: 100 mg/mL of Dibutyl Phthalate in methylene chloride

Sample solution A: Dilute 5.0 mL of *Sample stock solution* with methylene chloride to 10 mL.

Sample solution B: Add 5.0 mL of *Sample stock solution*, 1.0 mL of *Internal standard solution*, and dilute with methylene chloride to 10 mL.

Standard solution: Add 1.0 mL of *Sample solution A*, 10.0 mL of *Internal standard solution*, and dilute with methylene chloride to 100 mL.

Chromatographic system

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

Mode: GC

Detector: Flame ionization

Column: 4-mm × 1.5-m; packed with 3% liquid phase G3 on support S1A

Temperatures

Column: 190°

Injection port: 225°

Detector: 225°

Carrier gas: Nitrogen

Flow rate: 30 mL/min

Run time: Three times the retention time of dibutyl phthalate

Injection volume: 1 µL

System suitability

Samples: *Sample solution A* and *Standard solution*

Suitability requirements

[NOTE—The elution order is the internal standard peak followed by the dibutyl phthalate peak.]

Resolution: NLT 12 between the internal standard and the dibutyl phthalate peak, *Standard solution*

Additional requirements: Verify that there is no peak with the same retention time as the internal standard in *Sample solution A*. If such a peak is observed, make any necessary correction for factors of dilution, and then determine the area due to the interfering component that must be subtracted from the area of the internal standard peak appearing in the chromatogram recorded for *Sample solution B*.

Analysis

Samples: *Sample solution B* and *Standard solution*

Calculate the peak area ratio of dibutyl phthalate to the internal standard for the *Standard solution*. Calculate the peak area ratio of the sum of all peaks, excluding the main peak, the solvent peak, and the internal standard peak, to the internal standard peak for *Sample solution B*.

Acceptance criteria: NMT 1.0%; the ratio for *Sample solution B* is NMT the ratio for the *Standard solution*.

SPECIFIC TESTS

- **SPECIFIC GRAVITY ⟨841⟩:** 1.043–1.048 at 20°
- **REFRACTIVE INDEX ⟨831⟩:** 1.490–1.495 at 20°
- **ACIDITY**

Sample: 20.0 g

Analysis: Mix the *Sample* with 50 mL of alcohol that previously has been neutralized to a phenolphthalein endpoint. Add 0.2 mL of phenolphthalein TS, and titrate with 0.1 N sodium hydroxide VS.

Acceptance criteria: NMT 0.50 mL is required to change the color of the indicator.

- **WATER DETERMINATION, *Method I* ⟨921⟩:** NMT 0.2%
- **APPEARANCE**

Sample: Dibutyl Phthalate (neat)

Standard solution: Prepared immediately before use by mixing 2.4 mL of ferric chloride CS and 0.6 mL of cobaltous chloride CS with dilute hydrochloric acid (10 mg/mL) to make 10 mL, and diluting 5 mL of this solution with dilute hydrochloric acid (10 mg/mL) to make 100 mL

Analysis: Compare the *Sample* and the *Standard solution* by viewing the substance and the solution downward in matched color-comparison tubes against a white surface (see *Color and Achromicity* ⟨631⟩).

Acceptance criteria: The *Sample* is clear and not more intensely colored than the *Standard solution*.

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers. No storage requirements specified.
- **USP REFERENCE STANDARDS ⟨11⟩**

USP Dibutyl Phthalate RS

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

Topic/Question	Contact	Expert Committee
DIBUTYL PHTHALATE	Documentary Standards Support	SE2020 Simple Excipients
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SE2020 Simple Excipients

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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