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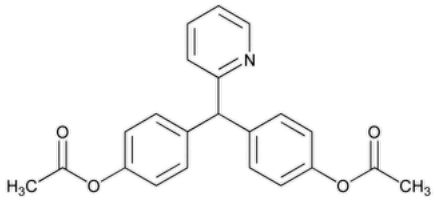
Bisacodyl



$C_{22}H_{19}NO_4$ 361.39

Phenol, 4,4'-(2-pyridinylmethylene)bis-, diacetate (ester);
4,4'-(2-Pyridylmethylene)diphenol diacetate (ester);
4,4'-(Pyridin-2-ylmethylene)diphenyl diacetate CAS RN®: 603-50-9.

DEFINITION

Bisacodyl contains NLT 98.0% and NMT 102.0% of bisacodyl ($C_{22}H_{19}NO_4$), calculated on the dried basis. [**CAUTION—**Avoid inhalation and contact with the eyes, skin, and mucous membranes.]

IDENTIFICATION

Change to read:

• **A.** ▲**SPECTROSCOPIC IDENTIFICATION TESTS ⟨197⟩, *Infrared Spectroscopy*, 197A or 197K**▲ (CN 1-MAY-2020)**:** Meets the requirements.

• **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **PROCEDURE**

Buffer: 1.58 g/L of ammonium formate in water; adjusted with formic acid to a pH of 5.0

Mobile phase: Acetonitrile and *Buffer* (45:55)

Diluent: Acetonitrile, acetic acid, and water (30:4:66)

Standard solution: 0.5 mg/mL of USP Bisacodyl RS in *Diluent*

Sample solution: 0.5 mg/mL of Bisacodyl in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 265 nm

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

Flow rate: 1.5 mL/min

Injection volume: 10 µL

Run time: NLT 2 times the retention time of bisacodyl

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 1.5

Relative standard deviation: NMT 0.73%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of bisacodyl ($C_{22}H_{19}NO_4$) in the portion of Bisacodyl taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response of bisacodyl from the *Sample solution*

r_S = peak response of bisacodyl from the *Standard solution*

C_S = concentration of USP Bisacodyl RS in the *Standard solution* (mg/mL)

C_U = concentration of Bisacodyl in the *Sample solution* (mg/mL)

Acceptance criteria: 98.0%–102.0% on the dried basis

IMPURITIES

- **Residue on Ignition ⟨281⟩:** NMT 0.1%
- **Organic Impurities**

Buffer: 1.58 g/L of ammonium formate in water; adjusted with formic acid to a pH of 5.0

Mobile phase: Acetonitrile and *Buffer* (45:55)

Diluent: Acetonitrile and water (35:5)

System suitability solution: 0.8 mg/mL of USP Bisacodyl RS; 2 µg/mL each of USP Bisacodyl Related Compound A RS, USP Bisacodyl Related Compound C RS, and USP Bisacodyl Related Compound E RS; and 4 µg/mL of USP Bisacodyl Related Compound B RS in *Diluent*

Sensitivity solution: 0.0003 mg/mL of USP Bisacodyl RS in *Diluent*

Standard stock solution: 1.0 mg/mL of USP Bisacodyl RS in *Diluent*

Standard solution: 1.0 µg/mL of USP Bisacodyl RS in *Diluent*

Sample solution: 1.0 mg/mL of Bisacodyl in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 265 nm

Column: 4.6-mm × 25-cm; 4- or 5-µm packing L1

Flow rate: 1.5 mL/min

Injection volume: 20 µL

Run time: NLT 3.5 times the retention time of bisacodyl

System suitability

Samples: *System suitability solution*, *Sensitivity solution*, and *Standard solution*

[Note—See *Table 1* for the relative retention times.]

Suitability requirements

Resolution: NLT 1.5 between the bisacodyl related compound E and bisacodyl peaks, *System suitability solution*

Tailing factor: NMT 2.0 for the bisacodyl peak, *System suitability solution*

Relative standard deviation: NMT 5.0% for the bisacodyl peak, *Standard solution*

Signal-to-noise ratio: NLT 10, *Sensitivity solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each individual impurity in the portion of Bisacodyl taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times (1/F) \times 100$$

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

r_S = peak response of bisacodyl from the *Standard solution*

C_S = concentration of the *Standard solution* (mg/mL)

C_U = concentration of the *Sample solution* (mg/mL)

F = relative response factor (see *Table 1*)

Acceptance criteria: See *Table 1*. The reporting threshold is 0.05%.

Table 1

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Bisacodyl related compound A	0.20	1.7	0.15
Bisacodyl related compound B	0.40	1.5	0.15

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Bisacodyl related compound C	0.45	1.3	0.50
Specified unidentified impurity 1	0.85	1.0	0.20
Bisacodyl related compound E	0.90	1.0	0.50
Bisacodyl	1.0	—	—
Specified unidentified impurity 2	2.6	1.0	0.30
Any individual unspecified impurity	—	1.0	0.10
Total impurities	—	—	1.0

SPECIFIC TESTS

• **Loss on Drying** ⟨731⟩

Analysis: Dry at 105° for 2 h.

Acceptance criteria: NMT 0.5%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers, protected from light. Store at room temperature.

• **USP Reference Standards** ⟨11⟩

USP Bisacodyl RS

USP Bisacodyl Related Compound A RS

4,4'-(Pyridin-2-ylmethylene)diphenol.

$C_{18}H_{15}NO_2$ 277.32

USP Bisacodyl Related Compound B RS

2,4'-(Pyridin-2-ylmethylene)diphenol.

$C_{18}H_{15}NO_2$ 277.32

USP Bisacodyl Related Compound C RS

4-[(4-Hydroxyphenyl)(pyridin-2-yl)methyl]phenyl acetate.

$C_{20}H_{17}NO_3$ 319.35

USP Bisacodyl Related Compound E RS

2-[(4-Acetoxyphenyl)(pyridin-2-yl)methyl]phenyl acetate.

$C_{22}H_{19}NO_4$ 361.39

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

Topic/Question	Contact	Expert Committee
BISACODYL	Documentary Standards Support	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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