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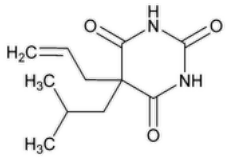
Butalbital



$C_{11}H_{16}N_2O_3$ 224.26

2,4,6(1*H*,3*H*,5*H*)-Pyrimidinetrione, 5-(2-methylpropyl)-5-(2-propenyl)-;

5-Allyl-5-isobutylbarbituric acid CAS RN®: 77-26-9; UNII: KHS0AZ4JVK.

DEFINITION

Butalbital contains NLT 98.0% and NMT 102.0% of butalbital ($C_{11}H_{16}N_2O_3$), calculated on the dried basis.

IDENTIFICATION

Change to read:

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

• **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: 1.0 mL of phosphoric acid diluted with water to 1 L

Mobile phase: Acetonitrile and *Buffer* (25:75)

System suitability solution: 0.1 mg/mL each of USP Butalbital RS and USP Salicylic Acid RS in *Mobile phase*. Sonication may be used to aid in dissolution.

Standard solution: 0.1 mg/mL of USP Butalbital RS in *Mobile phase*. Sonication may be used to aid in dissolution.

Sample solution: 0.1 mg/mL of Butalbital in *Mobile phase*. Sonication may be used to aid in dissolution.

Chromatographic system

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

Mode: LC

Detector: UV 214 nm

Column: 4.6-mm × 10-cm; 5-µm packing L1

Column temperature: 30°

Flow rate: 1 mL/min

Injection volume: 20 µL

System suitability

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

[Note—The relative retention times of salicylic acid and butalbital are 0.86 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 3.0 between salicylic acid and butalbital, *System suitability solution*

Tailing factor: NMT 1.5, *Standard solution*

Relative standard deviation: NMT 1.0%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of butalbital ($C_{11}H_{16}N_2O_3$) in the portion of Butalbital taken:

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

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

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

C_U = concentration of Butalbital 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: 4.1 g/L of monobasic potassium phosphate adjusted with 1 N sodium hydroxide to a pH of 6.0

Mobile phase: Acetonitrile and *Buffer* (22:78)

System suitability solution: 10 µg/mL each of USP Butalbital RS and USP Butabarbital RS in *Mobile phase*. Sonication may be used to aid in dissolution.

Sample solution: 1 mg/mL of Butalbital in *Mobile phase*. Sonication may be used to aid in dissolution.

Chromatographic system

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

Mode: LC

Detector: UV 214 nm

Column: 4.6-mm × 15-cm; 5-µm packing L78

Column temperature: 30°

Flow rate: 1 mL/min

Injection volume: 20 µL

System suitability

Sample: *System suitability solution*

[Note—The relative retention times of butabarbital and butalbital are 0.83 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 2.0 between butabarbital and butalbital

Tailing factor: NMT 1.5 for butalbital

Relative standard deviation: NMT 5.0% for butalbital

Analysis

Sample: *Sample solution*

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

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

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

r_T = sum of the peak responses from the *Sample solution*

Acceptance criteria

Any individual unspecified impurity: NMT 0.10%

Total impurities: NMT 1%

SPECIFIC TESTS

• **Loss on Drying ⟨731⟩**

Analysis: Dry under vacuum at room temperature to constant weight.

Acceptance criteria: NMT 0.2%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers.

• **USP Reference Standards ⟨11⟩**

USP Butabarbital RS

USP Butalbital RS

USP Salicylic Acid RS

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

Topic/Question	Contact	Expert Committee
BUTALBITAL	Documentary Standards Support	SM42020 Small Molecules 4

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 38(4)

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