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Butabarbital Sodium Oral Solution

DEFINITION
Butabarbital Sodium Oral Solution contains NLT 90.0% and NMT 110.0% of the labeled amount of butabarbital sodium ($C_{10}H_{15}N_2NaO_3$).

IDENTIFICATION

Change to read:

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

Sample: Transfer an equivalent to 150 mg of butabarbital sodium from a volume of Oral Solution, to a separator. Render it distinctly alkaline by the addition of 1 N sodium hydroxide, and saturate it with sodium chloride. Extract the mixture with two 15-mL portions of ether, and discard the ether. Acidify the solution with hydrochloric acid, and render it just alkaline to litmus by adding small portions of sodium bicarbonate (carbonate-free). Extract the liberated acid barbiturate using five 20-mL portions of chloroform. Wash the combined chloroform extracts with 10 mL of water acidified with 1 drop of hydrochloric acid, then extract the water with 10 mL of chloroform, adding the latter to the main chloroform solution. Pass the chloroform solution through a pledget of cotton or other suitable filter, previously washed with chloroform, into a tared beaker, and finally wash the separator and the filter with three 5-mL portions of chloroform. Evaporate the combined chloroform solution and washings on a steam bath with the aid of a current of air to dryness, and dry the residue at 105° for 2 h.

Acceptance criteria: Meets the requirements

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

ASSAY

• **Procedure**

Solution A: Dissolve 2.0 mL of bromine and 10 g of potassium bromide in 60 mL of water.

Solution B: Sodium metabisulfite in water (1 in 10)

Internal standard solution: 0.7 mg/mL of secobarbital in chloroform

Standard solution: 1 mg/mL of USP Butabarbital RS and 1.4 mg/mL of secobarbital in chloroform

Sample stock solution: Nominally 0.3 mg/mL of butabarbital sodium from Oral Solution prepared as follows. Transfer a volume of Oral Solution, equivalent to 30 mg of butabarbital sodium, to a separator. Add 1 mL of *Solution A*, and swirl. Allow to stand for 5 min, add 1 mL of *Solution B*, and swirl. Add 300 mg of sodium bicarbonate in small portions, with mixing, and extract with four 10-mL portions of chloroform. Pass the extracts through about 15 g of anhydrous sodium sulfate that is supported on a funnel by a small pledget of glass wool. Collect the combined filtrates in a 50-mL volumetric flask, wash the sodium sulfate with 5 mL of chloroform, collecting the washing with the filtrate, dilute with chloroform to volume, and mix.

[Note—This solution includes a bromination step for elimination of parabens and a carbonate–chloroform extraction for elimination of benzoic acid.]

Sample solution: Combine 2.0 mL of *Sample stock solution* with 2.0 mL of *Internal standard solution* in a suitable container, and reduce the volume to about 1 mL by evaporation, with the aid of a stream of dry nitrogen, at room temperature.

Chromatographic system

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

Mode: GC

Detector: Flame ionization

Column: 4-mm × 0.9-m glass; packed with 3% liquid phase G10 support on 80- to 10-mesh S1A

Temperatures

Injection port: 225°

Detector: 225°

Column: 200 ± 10°

Carrier gas: A suitable gas such as dry nitrogen

Flow rate: 60–80 mL/min

Injection volume: 5 µL

System suitability

Sample: *Standard solution*

[Note—The relative retention times for butabarbital and secobarbital are 0.6 and 1.0, respectively.]

Resolution: NLT 2.4 between butabarbital and secobarbital

Tailing factor: NMT 2.0 each for butabarbital and secobarbital

Relative standard deviation: NMT 1.5% for the peak response ratio of butabarbital to the internal standard

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of butabarbital sodium ($C_{10}H_{15}N_2NaO_3$) in the portion of Oral Solution taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times (M_{r1}/M_{r2}) \times 100$$

R_U = peak response ratio of butabarbital to the internal standard from the *Sample solution*

R_S = peak response ratio of butabarbital to the internal standard from the *Standard solution*

C_S = concentration of USP Butabarbital RS in the *Standard solution* (µg/mL)

C_U = nominal concentration of butabarbital sodium in the *Sample solution* (µg/mL)

M_{r1} = molecular weight of butabarbital sodium, 234.23

M_{r2} = molecular weight of butabarbital, 212.25

Acceptance criteria: 90.0%–110.0%

OTHER COMPONENTS

• **Alcohol Determination,** *Method II*⟨611⟩**:** Between 95.0% and 115.0% of the labeled amount of alcohol (C_2H_5OH)

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers. Store at controlled room temperature.

• **USP Reference Standards** ⟨11⟩

USP Butabarbital RS

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

Topic/Question	Contact	Expert Committee
BUTABARBITAL SODIUM ORAL SOLUTION	Documentary Standards Support	SM42020 Small Molecules 4

Chromatographic Database Information: Chromatographic Database

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