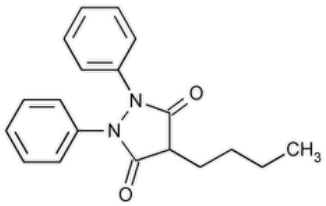
https://trungtamthuoc.com

Status: Currently Official on 16-Feb-2025
Official Date: Official as of 01-May-2020
Document Type: USP Monographs
DocId: GUID-BC01E1BE-6B8A-446A-A415-4D474FAB3324_4_en-US
DOI: https://doi.org/10.31003/USPNF_M64050_04_01
DOI Ref: f4zfk

© 2025 USPC
Do not distribute

Phenylbutazone



$C_{19}H_{20}N_2O_2$ 308.37

3,5-Pyrazolidinedione, 4-butyl-1,2-diphenyl-.

4-Butyl-1,2-diphenyl-3,5-pyrazolidinedione CAS RN®: 50-33-9; UNII: GN5P7K3T8S.

» Phenylbutazone contains not less than 98.0 percent and not more than 102.0 percent of $C_{19}H_{20}N_2O_2$, calculated on the dried basis.

Packaging and storage—Preserve in tight containers.

USP Reference standards ⟨11⟩—

USP Phenylbutazone RS

Identification—

Change to read:

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

Change to read:

B: ▲*Spectroscopic Identification Tests* ⟨197⟩*, Ultraviolet-Visible Spectroscopy: 197U*▲ (CN 1-May-2020)

Solution: 10 µg per mL.

Medium: sodium hydroxide solution (1 in 2500).

Absorptivities at 264 nm, calculated on the dried basis, do not differ by more than 2.0%.

Melting range ⟨741⟩: between 104° and 107°.

Loss on drying ⟨731⟩—Dry it in vacuum at a pressure of 30 ± 10 mm of mercury at 80° for 4 hours: it loses not more than 0.5% of its weight.

Residue on ignition ⟨281⟩: not more than 0.1%, 2.0 g being used for the test.

Chloride ⟨221⟩—Boil 2.0 g with 60 mL of water for 5 minutes, cool, and filter. To a 30-mL portion of the filtrate add 1 mL of 2 N nitric acid and 1 mL of silver nitrate TS: the filtrate shows no more chloride than corresponds to 0.10 mL of 0.020 N hydrochloric acid (0.007%).

Sulfate ⟨221⟩—To a 30-mL portion of the filtrate obtained in the test for *Chloride* add 2 mL of barium chloride TS: the mixture shows no more sulfate than corresponds to 0.10 mL of 0.020 N sulfuric acid (0.01%).

Assay—

Acetate buffer—Transfer 2.72 g of sodium acetate to a 1000-mL beaker, and dissolve in about 700 mL of water. Adjust with glacial acetic acid to a pH of 4.1. Filter through a 0.5-µm filter, dilute with filtered water to 1000 mL, and mix.

Mobile phase—Prepare a filtered and degassed mixture of acetonitrile with 560 mL of *Acetate buffer* (440:560). Make adjustments if necessary (see *System Suitability* under *Chromatography* ⟨621⟩).

Internal standard solution—Dissolve 300 mg of desoxycorticosterone acetate in 200 mL of acetonitrile, and mix.

Standard preparation—Dissolve an accurately weighed quantity of USP Phenylbutazone RS in acetonitrile, with the aid of sonication, and dilute quantitatively with acetonitrile to obtain a solution having a concentration of about 1.4 mg per mL. Pipet 10 mL of this solution into a 50-mL volumetric flask, add 10.0 mL of *Internal standard solution*, dilute with acetonitrile to volume, and mix. [Note—Use this solution within 8 hours of its preparation.]

Assay preparation—Transfer about 140 mg of Phenylbutazone, accurately weighed, to a 100-mL volumetric flask, add 75 mL of acetonitrile, and sonicate to dissolve. Dilute with acetonitrile to volume, and mix. Pipet 10 mL of this solution into a 50-mL volumetric flask, add 10.0 mL of *Internal standard solution*, dilute with acetonitrile to volume, and mix. [Note—Use this solution within 8 hours of its preparation.]

OFFICIAL

www.webofpharma.com

https://trungtamthuoc.com

Chromatographic system (see Chromatography ⟨621⟩)—The liquid chromatograph is equipped with a 254-nm detector and a 4.6-mm × 25-cm column that contains packing L7, preceded by a pre-column that contains packing L2. The flow rate is about 2.4 mL per minute. Chromatograph the *Standard preparation,* and record the peak responses as directed for *Procedure:* the resolution, *R,* of phenylbutazone and the internal standard is not less than 3.5, and the relative standard deviation of the ratio of their peak responses in replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 25 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. The relative retention times are about 1.0 for the internal standard and 0.7 for phenylbutazone. Calculate the quantity, in mg, of $C_{19}H_{20}N_2O_2$ in the portion of Phenylbutazone taken by the formula:

$$500C(R_U/R_S)$$

in which C is the concentration, in mg per mL, of USP Phenylbutazone RS in the *Standard preparation;* and R_U and R_S are the ratios of the peak response of the phenylbutazone to that of the internal standard for the *Assay preparation* and the *Standard preparation,* respectively.

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

Topic/Question	Contact	Expert Committee
PHENYLBUTAZONE	Documentary Standards Support	SM32020 Small Molecules 3
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 28(5)

Current DocID: GUID-BC01E1BE-6B8A-446A-A415-4D474FAB3324_4_en-US
DOI: https://doi.org/10.31003/USPNF_M64050_04_01
DOI ref: f4zfk

OFFICIAL