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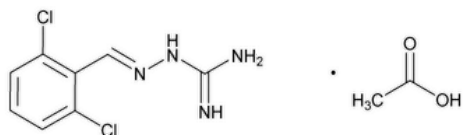
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Guanabenz Acetate

 $C_8H_8Cl_2N_4 \cdot C_2H_4O_2$

291.13

Hydrazinecarboximidamide, 2-[(2,6-dichlorophenyl)methylene]-, monoacetate;

[(2,6-Dichlorobenzylidene)amino]guanidine monoacetate CAS RN®: 23256-50-0; UNII: 443019GK1A.

DEFINITION

Guanabenz Acetate contains NLT 98.0% and NMT 101.5% of the labeled amount of guanabenz acetate ($C_{10}H_{12}N_4O_2Cl_2$).

IDENTIFICATION

Change to read:

- A. [▲SPECTROSCOPIC IDENTIFICATION TESTS \(197\), Infrared Spectroscopy: 197K](#) ▲ (CN 1-MAY-2020)

ASSAY

• PROCEDURE

Sample solution: 4 mg/mL of Guanabenz Acetate in glacial acetic acid**Titrimetric system**(See [Titrimetry \(541\)](#).)**Mode:** Direct titration**Titrant:** 0.1 N perchloric acid VS**Endpoint detection:** Potentiometric**Analysis:** Titrate 50 mL of the *Sample solution* with *Titrant*. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N perchloric acid is equivalent to 29.12 mg of guanabenz acetate ($C_{10}H_{12}N_4O_2Cl_2$).**Acceptance criteria:** 98.0%–101.5%

IMPURITIES

- [RESIDUE ON IGNITION \(281\)](#): NMT 0.2%

• ORGANIC IMPURITIES

Diluent: A mixture of formic acid and methanol (1 in 2000)**Aminoguanidine bicarbonate solution:** Transfer 100 mg of aminoguanidine bicarbonate to a test tube, add 0.05 mL of formic acid, and warm gently to dissolve. Quantitatively transfer the contents of the test tube to a 10-mL volumetric flask, and dilute with methanol to volume.**Standard solution A:** 0.1 mg/mL of [USP Guanabenz Acetate RS](#) in *Diluent* prepared as follows. Transfer a suitable amount of [USP Guanabenz Acetate RS](#) to a suitable volumetric flask, and dissolve in about 50% of total volume of *Diluent*. Add 0.1 mL of *Aminoguanidine bicarbonate solution* per mg of [USP Guanabenz Acetate RS](#), and dilute with *Diluent* to volume.**Standard solution B:** 0.05 mg/mL of [USP Guanabenz Acetate RS](#) in *Diluent* from *Standard solution A***Standard solution C:** 0.02 mg/mL of [USP Guanabenz Acetate RS](#) in *Diluent* from *Standard solution A***Sample solution:** 10 mg/mL of Guanabenz Acetate in *Diluent***Chromatographic system**(See [Chromatography \(621\), Thin-Layer Chromatography](#).)**Mode:** TLC**Adsorbent:** 0.25-mm layer of chromatographic silica gel mixture**Application volume:** 10 µL

Developing solvent system: Chloroform, methanol, and ammonium hydroxide (60:40:1)

Analysis

Samples: *Diluent, Standard solution A, Standard solution B, Standard solution C, and Sample solution*

Allow the chromatographic chamber to equilibrate in *Developing solvent system* for at least 30 min before use. Prewash a plate coated with *Adsorbent* by placing it in the chromatographic chamber, allowing the solvent front to rise to the top of the plate, dry it in air, and activate it by heating at 105° for 20 min. Within 30 min after preparation, separately apply portions of each of the *Standard solutions*, the *Sample solution*, and the *Diluent*. Allow the spots to dry, and place the plate in the chromatographic chamber. When the solvent has moved about three-fourths of the length of the plate, remove the plate, and allow it to air-dry for about 30 min. Examine the plate under short-wavelength UV light. Estimate the amount of any secondary spots, disregarding spots that have the same R_F as those from the *Diluent*, observed in the chromatogram of the *Sample solution* by comparison with each *Standard solution*. Place the plate in a chamber saturated with iodine vapors for about 10 min. Remove and examine the plate. Estimate the amount of any spot in the chromatogram of the *Sample solution* that has an R_F corresponding to the R_F of the spot produced by the aminoguanidine bicarbonate by comparison with each *Standard solution*.

Acceptance criteria: No individual secondary spot is greater in size or intensity than the spot produced by *Standard solution B* (0.5%), and the total of any such spots observed is NMT 1%.

Change to read:

• LIMIT OF 2,6-DICHLOROBENZALDEHYDE

Internal standard solution 1: 1 mg/mL of *p*-chlorobenzaldehyde in chloroform

Internal standard solution 2: 0.1 mg/mL of *p*-chlorobenzaldehyde in chloroform from *Internal standard solution 1*

Standard stock solution: 1 mg/mL of 2,6-dichlorobenzaldehyde in chloroform

Standard solution: 0.4 mg/mL of 2,6-dichlorobenzaldehyde from the *Standard stock solution* and 0.1 mg/mL of *p*-chlorobenzaldehyde from *Internal standard solution 1* in chloroform

Sample solution: Transfer 200 mg of Guanabenz Acetate to a 30-mL glass-stoppered centrifuge tube. Add 10 mL of 0.1 N hydrochloric acid, and shake to dissolve. Add 1.0 mL of *Internal standard solution 2*, and shake. Centrifuge, and transfer a portion of the lower layer to a stoppered container. The lower layer must be removed within 10 min of adding the acid to the centrifuge tube.

Chromatographic system

(See [Chromatography \(621\)](#), [System Suitability](#).)

Mode: GC

Detector: Flame ionization

Column: ▲1.8-m▲ (ERR 1-Jun-2019) × 3-mm; packed with 20% phase G1 on 80–100 mesh S1A support

Temperatures

Injection port: 225°

Detector: 250°

Column: 190°

Carrier gas: Nitrogen

Flow rate: 30 mL/min

Injection volume: 2 µL

System suitability

Sample: *Standard solution*

[NOTE—The relative retention times for *p*-chlorobenzaldehyde and 2,6-dichlorobenzaldehyde are 0.5 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 3.0 between 2,6-dichlorobenzaldehyde and *p*-chlorobenzaldehyde

Analysis

Samples: *Standard solution* and *Sample solution*

Acceptance criteria: The relative peak response ratio from the *Sample solution* does not exceed that from the *Standard solution* (0.2%).

SPECIFIC TESTS

• [pH \(791\)](#)

Sample solution: 7 mg/mL

Acceptance criteria: 5.5–7.0

• [Loss on Drying \(731\)](#)

Analysis: Dry under vacuum at 60° for 2 h.

Acceptance criteria: NMT 1.0%

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight, light-resistant containers.
- **USP REFERENCE STANDARDS** (11).
[USP Guanabenz Acetate RS](#)

Auxiliary Information - Please [check for your question in the FAQs](#) before contacting USP.

Topic/Question	Contact	Expert Committee
GUANABENZ ACETATE	Documentary Standards Support	SM22020 Small Molecules 2

Chromatographic Database Information: [Chromatographic Database](#)

Most Recently Appeared In:

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