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## Secobarbital Sodium Capsules

### DEFINITION

Secobarbital Sodium Capsules contain NLT 92.5% and NMT 107.5% of the labeled amount of secobarbital sodium ( $C_{12}H_{17}N_2NaO_3$ ).

### IDENTIFICATION

*Change to read:*

- A. **▲SPECTROSCOPIC IDENTIFICATION TESTS (197), Infrared Spectroscopy**▲ (CN 1-MAY-2020)

**Sample solution:** Nominally 5 mg/mL of secobarbital sodium in water from the contents of Capsules

**Analysis:** Transfer 10 mL of the filtered *Sample solution* to a separator. Add 2 mL of 3 N hydrochloric acid. Extract the liberated secobarbital with 20 mL of chloroform, and evaporate the extract to dryness. Dissolve the residue in chloroform to prepare a 5-mg/mL solution.

**Acceptance criteria:** The *Sample solution* exhibits maxima only at the same wavelengths as that of a similar preparation of [USP Secobarbital RS](#).

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

### ASSAY

#### • PROCEDURE

**Solution A:** Phosphoric acid and water (80:20)

**Buffer:** 1.4 g/L of monobasic potassium phosphate in water adjusted with *Solution A* to a pH of 3.5

**Mobile phase:** Acetonitrile and *Buffer* (35:65)

**Diluent:** Acetonitrile and water (35:65)

**Standard solution:** 0.4 mg/mL of [USP Secobarbital RS](#) in *Diluent*. Sonication may be used to aid in dissolution.

**Sample solution:** Nominally 0.4 mg/mL of secobarbital sodium in a solution prepared as follows. Transfer the equivalent of 44 mg of secobarbital sodium from the contents of NLT 20 Capsules to a 100-mL volumetric flask. Dilute with *Diluent* to volume. Shaking and sonication may be used to aid in dissolution. Centrifuge a portion of this solution, and use the supernatant.

#### Chromatographic system

(see [Chromatography \(621\), System Suitability](#))

**Mode:** LC

**Detector:** 215 nm

**Column:** 4.6-mm × 15.0-cm; 3.5-μm packing L1

**Flow rate:** 1 mL/min

**Injection volume:** 10 μL

**Run time:** 2 times the retention time of secobarbital

#### System suitability

**Sample:** *Standard solution*

#### Suitability requirements

**Tailing factor:** NMT 1.5

**Relative standard deviation:** NMT 1.0%

#### Analysis

**Samples:** *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of secobarbital sodium ( $C_{12}H_{17}N_2NaO_3$ ) in the portion of Capsules taken:

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

$r_U$  = peak response from the *Sample solution*

$r_S$  = peak response from the *Standard solution*

$C_s$  = concentration of [USP Secobarbital RS](#) in the *Standard solution* (mg/mL)

$C_u$  = nominal concentration of secobarbital sodium in the *Sample solution* (mg/mL)

$M_{r1}$  = molecular weight of secobarbital sodium, 260.26

$M_{r2}$  = molecular weight of secobarbital, 238.28

**Acceptance criteria:** 92.5%–107.5%

#### PERFORMANCE TESTS

- [Dissolution \(711\)](#)

**Medium:** Water; 500 mL

**Apparatus 1:** 100 rpm

**Time:** 60 min

**Standard solution:** [USP Secobarbital RS](#) in 0.1 N sodium hydroxide

**Sample solution:** Solution under test, mixed with sufficient sodium hydroxide to provide a concentration of 0.1 N sodium hydroxide, and diluted with *Medium*, as needed, so that the concentration is similar to that of the *Standard solution*

#### Instrumental conditions

**Mode:** UV

**Analytical wavelength:** 243 nm

**Tolerances:** NLT 75% ( $Q$ ) of the labeled amount of secobarbital sodium ( $C_{12}H_{17}N_2NaO_3$ ) is dissolved.

- [Uniformity of Dosage Units \(905\)](#): Meet the requirements

#### IMPURITIES

- [Organic Impurities](#)

**Solution A, Buffer, Mobile phase, Diluent, and Chromatographic system:** Proceed as directed in the Assay.

**Standard solution:** 0.005 mg/mL of [USP Secobarbital RS](#) in *Diluent*. Sonication may be used to aid in dissolution.

**Sample solution:** Nominally 1 mg/mL of secobarbital sodium in a solution prepared as follows. Transfer the equivalent of 100 mg of secobarbital sodium from NLT 20 Capsules to a 100-mL volumetric flask. Dilute with *Mobile phase* to 50% of the final flask volume. Sonicate and shake. Dilute with *Mobile phase* to volume. Pass through a suitable filter of 0.45- $\mu$ m pore size.

#### System suitability

**Sample:** *Standard solution*

#### Suitability requirements

**Tailing factor:** NMT 1.5

**Relative standard deviation:** NMT 6.0%

#### Analysis

**Samples:** *Standard solution* and *Sample solution*

Calculate the percentage of any individual unspecified degradant in the portion of Capsules taken:

$$\text{Result} = (r_u/r_s) \times (C_s/C_u) \times (M_{r1}/M_{r2}) \times 100$$

$r_u$  = peak response of any individual unspecified degradant from the *Sample solution*

$r_s$  = peak response from the *Standard solution*

$C_s$  = concentration of [USP Secobarbital RS](#) in the *Standard solution* (mg/mL)

$C_u$  = nominal concentration of secobarbital sodium in the *Sample solution* (mg/mL)

$M_{r1}$  = molecular weight of secobarbital sodium, 260.26

$M_{r2}$  = molecular weight of secobarbital, 238.28

**Acceptance criteria:** See [Table 1](#).

**Table 1**

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Iminosecobarbital <sup>a</sup>	0.33	1.0
2-Hydroxypropyl analog <sup>b</sup>	0.38	0.24
Secobarbital	1.0	—
1,3-Dimethylbutyl analog <sup>c</sup>	1.61	0.54
Any individual unspecified degradant	—	0.10
Total impurities	—	1.5

<sup>a</sup> 5-Allyl-4-imino-5-(1-methylbutyl) barbituric acid.

<sup>b</sup> 5-(2-Hydroxypropyl)-5-(1-methylbutyl) barbituric acid.

<sup>c</sup> 5-Allyl-5-(1,3-dimethylbutyl) barbituric acid.

#### ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in tight containers.

• **USP REFERENCE STANDARDS (11).**

[USP Secobarbital RS](#)

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

Topic/Question	Contact	Expert Committee
SECOBARBITAL SODIUM CAPSULES	<a href="#">Documentary Standards Support</a>	SM42020 Small Molecules 4
REFERENCE STANDARD SUPPORT	RS Technical Services <a href="mailto:RSTECH@usp.org">RSTECH@usp.org</a>	SM42020 Small Molecules 4

**Chromatographic Database Information:** [Chromatographic Database](#)

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