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Selegiline Hydrochloride Capsules

DEFINITION
Selegiline Hydrochloride Capsules contain NLT 90.0% and NMT 110.0% of the labeled amount of selegiline hydrochloride ($C_{13}H_{17}N \cdot HCl$).

IDENTIFICATION

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

Add the following:

▲• **B.** The UV spectrum of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.▲ (USP 1-May-2022)

ASSAY

Change to read:

• **Procedure**

Buffer: 11.5 g/L of monobasic ammonium phosphate in water. Adjust with phosphoric acid to a pH of 3.1.

Mobile phase: Acetonitrile and *Buffer* (20:80)

System suitability solution: 0.1 mg/mL each of USP Selegiline Hydrochloride RS and USP Methamphetamine Hydrochloride RS in *Mobile phase*

Standard solution: 0.1 mg/mL of USP Selegiline Hydrochloride RS in *Mobile phase*

Sample stock solution: Nominally, 1 mg/mL of selegiline hydrochloride from the contents of Capsules (NLT 20), prepared as follows. To the weighed portion of the sample in a suitable volumetric flask add *Mobile phase* to fill 80% of the final flask volume. Sonicate for at least 10 min. Dilute with *Mobile phase* to volume, and centrifuge a portion of the solution for at least 10 min.

Sample solution: Nominally, 0.1 mg/mL of selegiline hydrochloride, by diluting a portion of the supernatant from the *Sample stock solution* with *Mobile phase*. Pass a portion of the solution through a suitable filter of 0.45-µm pore size.

Chromatographic system

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

Mode: LC

Detector: UV 205 nm. ▲For *Identification B*, use a diode array detector in the range of 190–400 nm.▲ (USP 1-May-2022)

Column: 3.9-mm × 30-cm; 10-µm packing L1

Flow rate: 1 mL/min

Injection volume: 20 µL

Run time: ▲NLT▲ (USP 1-May-2022) 1.5 times the retention time of selegiline

System suitability

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

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

Suitability requirements

Resolution: NLT 3 between methamphetamine and selegiline, ▲▲ (USP 1-May-2022) *System suitability solution*

Relative standard deviation: NMT ▲1.0%,▲ (USP 1-May-2022) *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of selegiline hydrochloride ($C_{13}H_{17}N \cdot HCl$) in the portion of Capsules taken:

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

r_U = peak response of selegiline from the *Sample solution*

r_S = peak response of selegiline from the *Standard solution*

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

C_U = nominal concentration of selegiline hydrochloride in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• **Dissolution** ⟨711⟩

Medium: Water; 500 mL

Apparatus 1: 50 rpm

Time: 20 min

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

Standard solution: 0.01 mg/mL of USP Selegiline Hydrochloride RS in *Mobile phase*

Sample solution: Pass a portion of the solution under test through a suitable filter.

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of selegiline hydrochloride ($C_{13}H_{17}N \cdot HCl$) dissolved:

$$\text{Result} = (r_U/r_S) \times C_S \times V \times (1/L) \times 100$$

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

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

V = volume of *Medium*, 500 mL

L = label claim (mg/Capsule)

Tolerances: NLT 80% (Q) of the labeled amount of selegiline hydrochloride ($C_{13}H_{17}N \cdot HCl$) is dissolved.

• **Uniformity of Dosage Units** ⟨905⟩**:** Meet the requirements

IMPURITIES

Change to read:

• **Organic Impurities**

Buffer and **Mobile phase:** Prepare as directed in the *Assay*.

Standard stock solution A: 0.2 mg/mL each of USP Selegiline Hydrochloride RS and USP Selegiline Related Compound D RS in *Mobile phase*

Standard stock solution B: 0.2 mg/mL of USP Methamphetamine Hydrochloride RS in *Mobile phase*

Standard solution: ▲0.002 mg/mL▲ (USP 1-May-2022) each of USP Selegiline Hydrochloride RS and USP Selegiline Related Compound D RS, and ▲0.02 mg/mL▲ (USP 1-May-2022) of USP Methamphetamine Hydrochloride RS in *Mobile phase* from *Standard stock solution A* and *Standard stock solution B*, respectively

Sample solution: Nominally, 1 mg/mL of selegiline hydrochloride in *Mobile phase* from the contents of Capsules (NLT 20), prepared as follows. To the weighed portion of the sample in a suitable volumetric flask add *Mobile phase* to fill 60% of the final flask volume. Sonicate for at least 10 min, and dilute with *Mobile phase* to volume. Pass a portion of the solution through a suitable filter of 0.45-µm pore size.

Chromatographic system: Proceed as directed in the *Assay* except for the *Run time*.

Run time: ▲NLT▲ (USP 1-May-2022) 3 times the retention time of selegiline

System suitability

Sample: *Standard solution*

[Note—See *Table 1* for the relative retention times.]

Suitability requirements

Resolution: NLT 6.0 between methamphetamine and selegiline related compound D; NLT 3.2 between selegiline related compound D and selegiline

Relative standard deviation: NMT 10.0% for selegiline related compound D and selegiline ▲▲ (USP 1-May-2022) ; NMT 5.0% for methamphetamine ▲▲ (USP 1-May-2022)

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of methamphetamine and selegiline related compound D in the portion of Capsules taken:

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

r_U = peak response of methamphetamine or selegiline related compound D from the *Sample solution*

r_S = peak response of the corresponding Reference Standard from the *Standard solution*

C_S = concentration of the corresponding Reference Standard in the *Standard solution* (mg/mL)

C_U = nominal concentration of selegiline hydrochloride in the *Sample solution* (mg/mL)

Calculate the percentage of each ▲degradation product▲ (USP 1-May-2022) in the portion of Capsules taken:

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

r_U = peak response of each ▲degradation product▲ (USP 1-May-2022) from the *Sample solution*

r_S = peak response of USP Selegiline Hydrochloride RS from the *Standard solution*

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

C_U = nominal concentration of selegiline hydrochloride in the *Sample solution* (mg/mL)

Acceptance criteria: See *Table 1*. ▲The reporting threshold is▲ (USP 1-May-2022) 0.1%.

Table 1

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Methamphetamine[a]	0.62	2.0
Selegiline related compound D▲▲ (USP 1-May-2022)	0.85	0.50
Selegiline	1.0	—
Any ▲▲ (USP 1-May-2022) unspecified degradation product	—	0.50
Total impurities[b]	—	2.0

[a] Benzeneethanamine, *N*,α-dimethyl-, (*S*)-.

[b] Does not include methamphetamine.

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight, light resistant containers.
- **USP Reference Standards** ⟨11⟩
  USP Methamphetamine Hydrochloride RS

USP Selegiline Hydrochloride RS

USP Selegiline Related Compound D RS

(*R*)-*N*-(1-Phenylpropan-2-yl)prop-2-yn-1-amine hydrochloride.

$C_{12}H_{15}N \cdot HCl$ 209.72

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

Topic/Question	Contact	Expert Committee
SELEGILINE HYDROCHLORIDE CAPSULES	Documentary Standards Support	SM42020 Small Molecules 4
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM42020 Small Molecules 4

Chromatographic Database Information: Chromatographic Database

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