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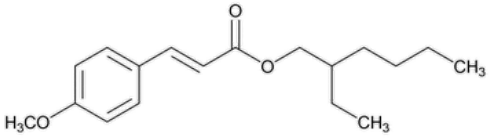
Octinoxate



$C_{18}H_{26}O_3$ 290.40

2-Propenoic acid, 3-(4-methoxyphenyl)-, 2-ethylhexyl ester;

2-Ethylhexyl *p*-methoxycinnamate CAS RN®: 5466-77-3; UNII: 4Y5P7MUD51.

DEFINITION

Octinoxate contains NLT 95.0% and NMT 105.0% of octinoxate ($C_{18}H_{26}O_3$), calculated on the as-is basis.

IDENTIFICATION

Change to read:

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

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

ASSAY

• **Procedure**

System suitability stock solution: 5% (v/v) USP Benzyl Benzoate RS in acetone

System suitability solution: 50 mg/mL of USP Octinoxate RS in *System suitability stock solution*

Standard solution: 50 mg/mL of USP Octinoxate RS in acetone

Sample solution: Transfer 5 mL of Octinoxate to a 100-mL volumetric flask, and dilute with acetone to volume.

Chromatographic system

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

Mode: GC

Detector: Flame ionization

Column: 0.32-mm × 25-m; 0.25-µm thickness of phase G1

Carrier gas: Helium

Temperatures

Injection port: 250°

Detector: 300°

Column: See *Table 1*.

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
80	22	300	10

Flow rate: 2 mL/min

Injection volume: 1 µL

Injection type: Split ratio, 85:1

System suitability

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

[NOTE—The relative retention times for benzyl benzoate and octinoxate are about 0.68 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 20 between benzyl benzoate and octinoxate, *System suitability solution*

Relative standard deviation: NMT 2.0%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of octinoxate ($C_{18}H_{26}O_3$) in the portion of Octinoxate taken:

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

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

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

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

C_U = concentration of Octinoxate in the *Sample solution* (mg/mL)

Acceptance criteria: 95.0%–105.0% on the as-is basis

IMPURITIES

• **ORGANIC IMPURITIES**

System suitability stock solution, System suitability solution, Standard solution, Chromatographic system, and **System suitability:** Proceed as directed in the *Assay*.

Sample solution: Transfer 5 mL of Octinoxate to a 100-mL volumetric flask, and dilute with acetone to volume.

Analysis

Sample: *Sample solution*

Calculate the percentage of each impurity in the portion of Octinoxate taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak response of each impurity from the *Sample solution*

r_T = sum of all the peak responses from the *Sample solution*

Acceptance criteria

Any individual impurity: NMT 0.5%

Total impurities: NMT 2.0%

SPECIFIC TESTS

• **SPECIFIC GRAVITY ⟨841⟩:** 1.005–1.013

• **REFRACTIVE INDEX ⟨831⟩:** 1.542–1.548 at 20°

• **ACIDITY**

Sample: 5 mL of Octinoxate

Analysis: Add 50 mL of alcohol to the *Sample*, and mix. Add 4 drops of phenolphthalein TS, and titrate with 0.1 N sodium hydroxide.

Acceptance criteria: NMT 0.8 mL of 0.1 N sodium hydroxide is required.

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in tight containers, and store in a cool place.

• **USP REFERENCE STANDARDS ⟨11⟩**

USP Benzyl Benzoate RS

Benzoic acid, phenylmethyl ester.

$C_{14}H_{12}O_2$ 212.24

USP Octinoxate RS

Topic/Question	Contact	Expert Committee
OCTINOXATE	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:
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