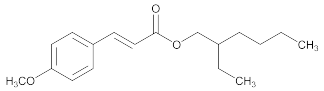
Octinoxate



$C_{18}H_{26}O_3$ 290.40
2-Ethylhexyl 3-(4-methoxyphenyl)-2-propenoate.
2-Propenoic acid, 3-(4-methoxyphenyl)-, 2-ethylhexyl ester. [5466-77-3].

» Octinoxate contains not less than 95.0 percent and not more than 105.0 percent of $C_{18}H_{26}O_3$, calculated on the as-is basis.

Packaging and storage—Preserve in tight containers, in a cool place.

USP Reference standards ⟨11⟩**—**
USP Octinoxate RS
Octyl methoxycinnamate.

Identification—

A: *Infrared Absorption* ⟨197F⟩.

B: *Ultraviolet Absorption* ⟨197U⟩—

Solution: 5 µg per mL.

Medium: alcohol.

Specific gravity ⟨841⟩**:** between 1.005 and 1.013.

Refractive index ⟨831⟩**:** between 1.542 and 1.548 at 20°.

Acidity—Transfer 5 mL of Octinoxate to a suitable container, add 50 mL of alcohol, and mix. Add 4 drops of phenolphthalein TS, and titrate with 0.1 N sodium hydroxide: not more than 0.8 mL is consumed.

Chromatographic purity—

Test solution—Transfer about 5 mL of Octinoxate to a 100-mL volumetric flask, dilute with acetone to volume, and mix.

Chromatographic system (see *Chromatography* ⟨621⟩)—Proceed as directed in the *Assay*.

Procedure—Inject a volume (about 1 µL) of the *Test solution* into the chromatograph, record the chromatogram, and measure the areas for all the peaks. Calculate the percentage of each impurity in the portion of Octinoxate taken by the formula:

$$100(r_i / r_s)$$

in which r_i is the peak area for each impurity; and r_s is the sum of the areas for all the peaks: not more than 0.5% of any individual impurity is found; and not more than 2.0% of total impurities is found.

Assay—

Internal standard solution—Transfer about 25 mL of benzyl benzoate to a 500-mL volumetric flask, dilute with acetone to volume, and mix.

Standard preparation—Dilute an accurately measured quantity of USP Octinoxate RS quantitatively, and stepwise if necessary, with *Internal standard solution* to obtain a solution having a known concentration of about 50 mg per mL.

Assay preparation—Transfer about 5 mL of Octinoxate, accurately measured, to a 100-mL volumetric flask, dilute with *Internal standard solution* to volume, and mix.

Chromatographic system (see *Chromatography* ⟨621⟩)—The gas chromatograph is equipped with a flame-ionization detector, a 0.32-mm × 25-m column that contains coating G1, and a split injection system with a split ratio of about 85:1. The carrier gas is helium, flowing at a rate of about 2 mL per minute. The chromatograph is programmed as follows. Initially the temperature of the column is equilibrated at 80°, then the temperature is increased to 300° over a period of 10 minutes, and maintained at 300° for 10 minutes. The injection port temperature is maintained at 250°, and the detector temperature is maintained at 300°. Chromatograph the *Standard preparation,* and record the peak areas as directed for *Procedure:* the relative retention times are about 0.68 for benzyl benzoate and 1.0 for octinoxate; the resolution, *R,* between benzyl benzoate and octinoxate is not less than 20; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 1 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the areas for the major peaks. Calculate the quantity, in mg, of $C_{18}H_{26}O_3$ in the portion of Octinoxate taken by the formula:

$$100C(R_U / R_S)$$

in which C is the concentration, in mg per mL, of USP Octinoxate RS in the *Standard preparation;* and R_U and R_S are the peak area ratios of octinoxate to benzyl benzoate obtained from the *Assay preparation* and the *Standard preparation,* respectively.

Octisalate

$C_{15}H_{22}O_3$ 250.33
2-Ethylhexyl salicylate.
Benzoic acid, 2-hydroxy-, 2-ethylhexyl ester [118-60-5].

» Octisalate contains not less than 95.0 percent and not more than 105.0 percent of $C_{15}H_{22}O_3$.

Packaging and storage—Preserve in tight containers.

USP Reference standards ⟨11⟩**—**
USP Octisalate RS
Octyl salicylate.

Identification—

A: *Infrared Absorption* ⟨197F⟩.

B: *Ultraviolet Absorption* ⟨197U⟩—

Solution: 5.0 µg per mL.

Medium: alcohol.

Absorptivity at 305 nm, calculated on the as-is basis, does not differ by more than 3.0%.

Specific gravity ⟨841⟩**:** between 1.011 and 1.016.

Refractive index ⟨831⟩**:** between 1.500 and 1.503 at 20°.

Acidity—Transfer 50 mL of alcohol to a suitable container, add 1 mL of phenol red TS, and add sufficient 0.1 N sodium hydroxide to obtain a persistent pink color. Transfer 50 mL of this solution to a suitable container, add about 5.0 mL of accurately measured Octisalate, mix, and titrate with 0.1 N sodium hydroxide: not more than 0.2 mL of 0.1 N sodium hydroxide per mL of Octisalate is required for neutralization.

Chromatographic purity—

Test solution—Use the *Assay preparation.*

Chromatographic system—Proceed as directed in the *Assay.* To evaluate the system suitability requirements, use the *Standard preparation,* as prepared in the *Assay.*

Procedure—Inject a volume (about 1 µL) of the *Test solution* into the chromatograph, record the chromatogram, and measure all of the peak responses. Calculate the percentage of each impurity in the portion of Octisalate taken by the formula:

$$100(r_i / r_s)$$

in which r_i is the peak response for each impurity, and r_s is the sum of the responses of all the peaks: not more than 0.5% of any individual impurity is found; and not more than 2.0% of total impurities is found.

Assay—

Standard preparation—Dissolve an accurately weighed quantity of USP Octisalate RS in *tert*-butyl methyl ether, and dilute quantitatively, and stepwise if necessary, with *tert*-butyl methyl

ether to obtain a solution having a known concentration of about 20.0 mg per mL.

Assay preparation—Transfer about 2 g of Octisalate, accurately weighed, to a 100-mL volumetric flask, dilute with *tert*-butyl methyl ether to volume, and mix.

Chromatographic system (see *Chromatography* ⟨621⟩)—The gas chromatograph is equipped with a flame-ionization detector and a 0.32-mm × 25-m column coated with a 0.1-µm film of phase G1. The carrier gas is helium, flowing at a rate of about 6 mL per minute. The split ratio is 50:1. [NOTE—Split ratio can be modified in order to optimize the performance.] The chromatograph is programmed as follows. Initially the temperature of the column is equilibrated at 60°, then the temperature is increased at a rate of 8° per minute to 240°, and is maintained at 240° for 10 minutes. The injection port temperature is maintained at 240°, and the detector temperature is maintained at 260°. Chromatograph the *Standard preparation,* and record the peak responses as directed for *Procedure:* the resolution, *R,* between octisalate and any other peak is not less than 1.0; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 1 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity, in mg, of $C_{15}H_{22}O_3$ in the portion of Octisalate taken by the formula:

$$100C(r_U / r_S)$$

in which *C* is the concentration, in mg per mL, of USP Octisalate RS in the *Standard preparation;* and r_U and r_S are the peak responses obtained from the *Assay preparation* and the *Standard preparation,* respectively.

Octocrylene

$C_{24}H_{27}NO_2$ 361.48

2-Propenoic acid, 2-cyano-3,3-diphenyl, 2-ethylhexyl ester.

2-Ethylhexyl 2-cyano-3,3-diphenylacrylate [6197-30-4].

» Octocrylene contains not less than 95.0 percent and not more than 105.0 percent of $C_{24}H_{27}NO_2$.

Packaging and storage—Preserve in tight containers.

USP Reference standards ⟨11⟩**—**

USP Octocrylene RS

Identification, *Ultraviolet Absorption* ⟨197U⟩**—**

Solution: 25 µg per mL.

Medium: methanol.

Absorptivities, calculated on the as-is basis, do not differ by more than 3.0%.

Specific gravity ⟨841⟩**:** between 1.045 and 1.055.

Refractive index ⟨831⟩**:** between 1.561 and 1.571 at 20°.

Acidity—Transfer 60 mL of alcohol to a suitable container, add 1 mL of phenolphthalein TS, and add sufficient 0.1 N sodium hydroxide to obtain a persistent pink color. Transfer 60 mL of this solution to a suitable container, add about 6 g of Octocrylene, accurately weighed, mix, and titrate with 0.1 N sodium hydroxide: not more than 0.18 mL of titrant per g of Octocrylene is necessary to obtain a persistent pink endpoint.

Chromatographic purity—

Test solution—Use the *Assay preparation.*

Chromatographic system—Proceed as directed in the *Assay.* To evaluate the system suitability requirements, use the *Standard preparation* prepared as directed in the *Assay.*

Procedure—Inject a volume (about 1 µL) of the *Test solution* into the chromatograph, record the chromatogram, and measure all of the peak responses. Calculate the percentage of each impurity in the portion of Octocrylene taken by the formula:

$$100(r_i / r_s)$$

in which r_i is the peak response for each impurity; and r_s is the sum of the responses of all the peaks: not more than 0.5% of any individual impurity is found; and not more than 2.0% of total impurities is found.

Assay—

Standard preparation—Dissolve an accurately weighed quantity of USP Octocrylene RS in acetone, and dilute quantitatively, and stepwise if necessary, with acetone to obtain a solution having a known concentration of about 21.0 mg per mL.

Assay preparation—Transfer about 2.1 g of Octocrylene, accurately weighed, to a 100-mL volumetric flask, dilute with acetone to volume, and mix.

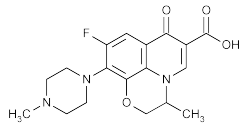
Chromatographic system (see *Chromatography* ⟨621⟩)—The gas chromatograph is equipped with a flame-ionization detector and a 0.32-mm × 60-m column coated with a 0.25-µm film of G1. Helium is used as the carrier gas at a flow rate of about 6 mL per minute. The split ratio is 30:1. The chromatograph is programmed as follows. Initially the temperature of the column is equilibrated at 80°; upon injection, the temperature is increased at a rate of 4° per minute to 280°, and is held at 280° for 10 minutes. The injection port temperature is maintained at 300°, and the detector temperature is maintained at 300°. Chromatograph the *Standard preparation*, and record the peak responses as directed for *Procedure:* the resolution, *R*, between the octocrylene and any other peak is not less than 1.0; and the relative standard deviation for replicate injections is not more than 2.0%.

Procedure—Separately inject equal volumes (about 1 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity, in mg, of $C_{24}H_{27}NO_2$ in the portion of Octocrylene taken by the formula:

$$100C(r_U / r_S)$$

in which *C* is the concentration, in mg per mL, of USP Octocrylene RS in the *Standard preparation;* and r_U and r_S are the peak responses obtained from the *Assay preparation* and the *Standard preparation,* respectively.

Ofloxacin



$C_{18}H_{20}FN_3O_4$ 361.38

7*H*-Pyrido[1,2,3-*de*]-1,4-benzoxazine-6-carboxylic acid, 9-fluoro-2,3-dihydro-3-methyl-10-(4-methyl-1-piperazinyl)-7-oxo-, (±)-.

(±)-9-Fluoro-2,3-dihydro-3-methyl-10-(4-methyl-l-piperazinyl)-7-oxo-7*H*-pyrido[1,2,3-*de*]-1,4-benzoxazine-6-carboxylic acid [82419-36-1].

» Ofloxacin contains not less than 98.5 percent and not more than 101.5 percent of $C_{18}H_{20}FN_3O_4$, calculated on the dried basis.

Packaging and storage—Preserve in well-closed containers, protected from light. Store at 25°, excursions permitted between 15° and 30°.