

Status: Currently Official on 14-Feb-2025  
Official Date: Official as of 01-Jun-2021  
Document Type: Reagents  
DocId: GUID-88A84D5E-C738-429B-8A88-FA111DA6F83A\_5\_en-US  
DOI: [https://doi.org/10.31003/USPNF\\_R7289\\_05\\_01](https://doi.org/10.31003/USPNF_R7289_05_01)  
DOI Ref: jsg86

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## (+)-1-(9-Fluorenyl)ethyl Chloroformate Solution

To view the Notice from the Expert Committee that posted in conjunction with this accelerated revision, please click

<https://www.uspnf.com/rb-plus-1-9-fluorenyl-ethyl-chloroformate-sol-20210528>.

((+)-FLEC),  $C_{16}H_{13}ClO_2$  272.73 CAS RN®: 107474-79-3.—Use a suitable grade with a concentration of NLT 18 mM in acetone.

### Change to read:

#### • ENANTIOMERIC PURITY

**Mobile phase:** [Acetonitrile](#) and [water](#) (1:1)

**Standard solution:** 30 µg/mL of [\(±\)-1-\(9-fluorenyl\)ethanol](#) in alcohol

**Sample solution:** 18 mM solution of (+)-1-(9-fluorenyl)ethyl chloroformate in acetone

**Carbonate buffer solution:** Transfer 338 mg of [sodium carbonate](#) and 152 mg of [sodium bicarbonate](#) to a 100-mL volumetric flask. Dissolve in and dilute with [water](#) to volume.

**Sodium hydroxide solution:** Transfer 30 g of [sodium hydroxide](#) to a 100-mL volumetric flask, add 50 mL of [water](#) to dissolve, and cool and dilute with [water](#) to volume.

**Acetate buffer solution:** Transfer 0.3 mL of [glacial acetic acid](#) to a 100-mL volumetric flask, add 90 mL of [water](#), adjust with *Sodium hydroxide solution* to a pH of 4.2, and dilute with [water](#) to volume.

**Blank:** [Water](#)

**Procedure:** Transfer 20.0 µL each of *Sample solution* and *Blank* to separate 10-mL test tubes. Add 80 µL of *Carbonate buffer solution* to each test tube and cap the tubes tightly. Let each solution react for 24 h at 45° over a water bath. Cool to room temperature, add 5.0 mL of *Acetate buffer solution*, mix by vortex, and transfer to separate vials.

#### Chromatographic System

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

**Mode:** LC

**Detector:** Fluorescence

**Excitation wavelength:** 260 nm

**Emission wavelength:** 315 nm

**Column:** 4.6-mm × 15-cm; 3.0-µm packing [L40](#)

**Column temperature:** 25°

**Flow rate:** 0.5 mL/min

**Injection volume:** 5 µL

**Run time:** 2 times the retention time of (±)-1-(9-fluorenyl)ethanol

#### System Suitability

**Samples:** *Standard solution*, hydrolyzed *Sample solution*, and hydrolyzed *Blank*

##### Suitability requirements

**Relative retention times:** About 0.95 for hydrolysate of (–)-1-(9-fluorenyl)ethyl chloroformate and 1.0 for hydrolysate of (+)-1-(9-fluorenyl)ethyl chloroformate

**Resolution:** NLT 1.5 between hydrolysate of (–)-1-(9-fluorenyl)ethyl chloroformate and hydrolysate of (+)-1-(9-fluorenyl)ethyl chloroformate. Absence of peaks at the retention times for hydrolysate of (–)-1-(9-fluorenyl)ethyl chloroformate and hydrolysate of (+)-1-(9-fluorenyl)ethyl chloroformate in the hydrolyzed *Blank*.

#### Analysis

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

Integrate only peaks at the retention times of (–)-1-(9-fluorenyl)ethanol and (+)-1-(9-fluorenyl)ethanol. Disregard any extraneous peaks in the chromatogram of hydrolyzed *Sample solution*.

Calculate the percentage of (+)-1-(9-fluorenyl)ethyl chloroformate ( $P_A$ ) in the portion of (+)-1-(9-fluorenyl)ethyl chloroformate taken:

$$\text{Result} = r_A / (r_A + r_B) \times 100$$

$r_A$  = peak response of hydrolysate of (+)-1-(9-fluorenyl)ethyl chloroformate from the *Sample solution*

$r_B$  = peak response of hydrolysate of (-)-1-(9-fluorenyl)ethyl chloroformate from the *Sample solution*

Calculate the percentage of (-)-1-(9-fluorenyl)ethyl chloroformate ( $P_B$ ) in the portion of (+)-1-(9-fluorenyl)ethyl chloroformate taken:

$$\text{Result} = 100 - P_A$$

**Acceptance criteria:** NLT ▲99%▲ (RB 1-Jun-2021) of (+)-1-(9-fluorenyl)ethyl chloroformate and NMT ▲1%▲ (RB 1-Jun-2021) of (-)-1-(9-fluorenyl)ethyl chloroformate

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

Topic/Question	Contact	Expert Committee
(±)-1-(9-FLUORENYL)ETHYL CHLOROFORMATE SOLUTION	<a href="#">Margareth R.C. Marques</a> Principal Scientific Liaison	HDQ Headquarters

**Most Recently Appeared In:**

Pharmacopeial Forum: Volume No. PF 43(2)

**Current DocID:** [GUID-88A84D5E-C738-429B-8A88-FA111DA6F83A\\_5\\_en-US](#)

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