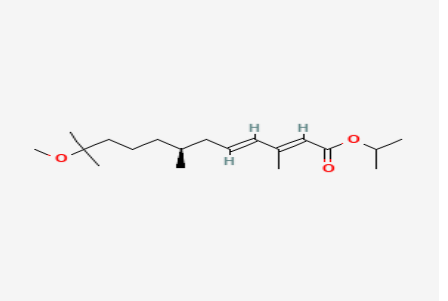
S-METHOPRENE
414



CAS Number: 65733-16-6

IUPAC name: isopropyl (E, E)-(S)-11-methoxy-3,7,11-trimethyldodeca-2,4-dienoate

Chemical name: isopropyl (2E, 4E, 7S)-11-methoxy-3,7,11-trimethyl-2,4-dodecadienoate

Empirical formula: C19H34O3

RMM: 310.5

B.p.: 279.9 °C at atmospheric pressure (97.2 kPa)

Vp.: 0.623 mPa (20 °C), 1.08 mPa (25 °C) (Knudsen effusion)

Kow: logP ≥6

S.g./density: 0.924 (20 °C)

Description: a pale yellow liquid, with a fruity odour

Solubility: In water 6.85 ppm (20 °C). Soluble in most organic solvents, in acetone and hexane >500, methanol >450 (all in g/l, 20±1 °C).

Stability: Stable in water, organic solvents, and in the presence of aqueous acids and alkalis. Sensitive to uv light.

Specific rotation [α] 20D +5.64°

S-METHOPRENE TECHNICAL

As for Methoprene technical **414**/TC/(M)/- (CIPAC H, *p* 115) but add:

4 S-Methoprene Determinations

OUTLINE OF METHOD THE SAMPLE IS DISSOLVED IN N-HEXANE AND THE CONTENT OF S-METHOPRENE IS DETERMINED BY NORMAL PHASE HIGH PERFORMANCE LIQUID CHROMATOGRAPHY ON A CHIRALPAK AD-H SILICA COLUMN WITH DETECTION AT 254 NM (IN COMBINATION WITH CIPAC 414/TC/(M)/-, SECTION 3).

REAGENTS

N-HEXANE HPLC GRADE
2-Propanol HPLC grade
Eluent n-hexane/2-propanol, 100/0.5 (v/v)

Methoprene control sample mixture of stereoisomers, Store refrigerated.

Control sample solution. Prepare a suitable solution of the control sample in mobile phase. This solution is used only for a column efficacy check to demonstrate the separation of the Methoprene stereoisomers and to ensure the correct assignment of the S-Methoprene enantiomer (2E, 4E, 7S) via the relative retention time (solution R).

APPARATUS

HIGH PERFORMANCE LIQUID CHROMATOGRAPH EQUIPPED WITH AN AUTOMATIC LOOP INJECTOR AND AN UV SPECTROPHOTOMETRIC DETECTOR CAPABLE OF MEASURING AT 254 NM
Column stainless steel, 250 × 4.6 mm (i.d.) packed with CHIRALPAK AD-H SILICA, 5µm
Electronic integrator or *data system*

PROCEDURE

(a) Operating conditions (typical):

Column 250 × 4.6 mm (i.d.) packed with CHIRALPAK AD-H SILICA
Mobile phase *n*-hexane/2-propanol*, 100/0.5 (v/v)*
Flow rate 0.5 ml/min
Column temperature 25 °C
Injection volume 10 µl
Detector wavelength 254 nm
Relative retention times

	Methoprene stereoisomer	retention time
	S-cis	10.34
	R-cis	10.82
Methoprene	*S*-methoprene	12.35
	R- methoprene	13.05

(b) Preparation of sample. Prepare samples in duplicate. Weigh 20 mg of S-Methoprene TC (to the nearest 0.1 mg) into a volumetric flask (10 ml). Dissolve, allow to attain room temperature, and make up to volume with mobile phase (Solution S_a and S_b). Transfer 0.1ml Solution S_a and S_b into a 10 ml volumetric flask. Make up to volume with mobile phase. Mix thoroughly (solutions S_1 and S_2). Keep the sample solutions at constant room temperature.

(c) Performance check. Make replicate injections of the control sample solution to check the pattern and the separation of methoprene enantiomer (see Fig.1 and the relative retention times given above). Measure the peak areas and determine the S-methoprene to R-methoprene enantiomer peak area ratios. Repeat until the values of subsequent injections differ by less than 2%.

(d) Determination. Inject duplicate aliquots of each sample solution S_1 and S_2 and measure the peak areas. Repeat the measurement of control sample solution (solution R) after a series of 4 sample runs and at the end of the sequence.

(e) Calculation. Determine for each injection of the peak areas ratio of S-methoprene to the sum of S-methoprene and R--methoprene. (The detector response of each enantiomer is considered to be the same). Calculate the content of S-methoprene using the following formula:

$$\text{Content of S-methoprene} = \frac{A \times B}{100} \text{ g/kg}$$

where:
A = Methoprene content obtained under CIPAC 414/TC/(M)/-*Section* **3**(*f*) (g/kg)
B = peak area ratio of S-methoprene to the sum of S-methoprene and R--methoprene (%)

Fig.1 Representative Chromatogram of Methoprene stereoisomers

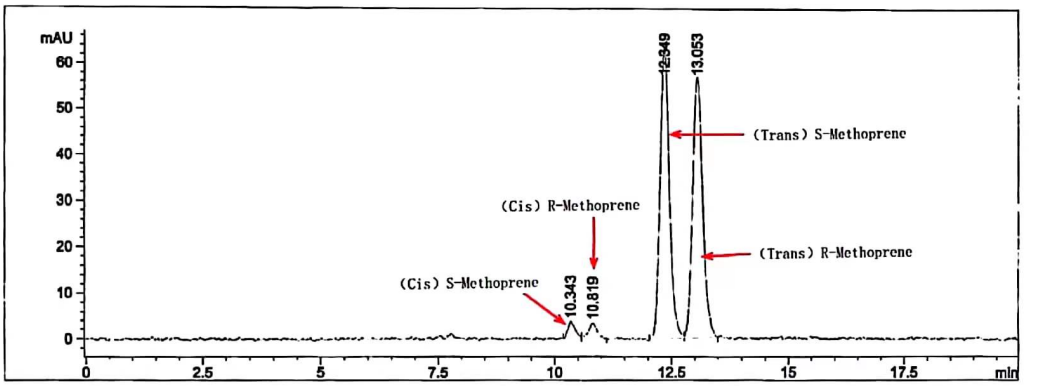
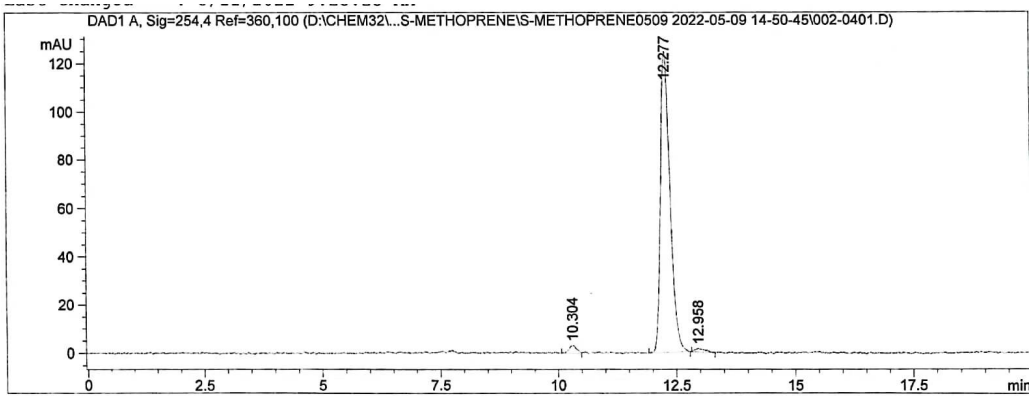


Fig.2 Representative Chromatogram of S-Methoprene TC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mAU
120
100
80
60
40
20
0
10.304
12.277
12.958
0
2.5
5
7.5
10
12.5
15
17.5
min