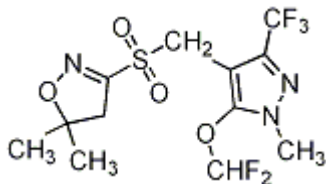


S-metolachlor

607



ISO common name S-metolachlor

Chemical name A mixture of (aRS,1S)-2-chloro-6'-ethyl-N-(2-methoxy-1-methylethyl)acetamide and (aRS,1R)-2-chloro-6'-ethyl-N-(2-methoxy-1-methylethyl)acetamide in the proportion 80–100% to 20–0% (IUPAC); A mixture of (S)-2-chloro-N-(2-ethyl-6-methylphenyl)-N-(2-methoxy-1-methylethyl)acetamide and (R)-2-chloro-N-(2-ethyl-6-methylphenyl)-N-(2-methoxy-1-methylethyl)acetamide in the proportion 80–100% to 20–0% (CA: 447399–55–5)

Empirical formula C₁₅H₂₂ClNO₂

RMM 283.8

Description Clear yellow to brownish liquid

Formulations Emulsifiable Concentrates (EC)

S-METOLACHLOR TECHNICAL

*607/TC/(M)/-

1. Sampling. Take at least 100 g.

2. Identity tests

As for metolachlor technical 400/TC/M/2.

3. Determination of Total isomers Content

As for metolachlor technical 400/TC/M/3.

4. Determination of S-isomers and R-isomers purity

OUTLINE OF METHOD S-isomers and R-isomers percentage in S-metolachlor is determined by normal phase HPLC on a chiral column using UV detector at 230 nm. The S-isomers percentage in S-metolachlor is calculated.

REAGENTS

Metolachlor standard (racemate) of known purity

S-metolachlor standard of known purity

Heptane, HPLC grade

Ethanol HPLC grade

Mobile phase Heptane - Ethanol, 94+6 (v/v). Add by pipette ethanol (60 ml) to heptane (940 ml); degas before use.

Calibration solution (racemate solution). Weigh (to the nearest 0.1 mg) about 25 mg of the racemate metolachlor standard into a volumetric flask (25 ml). Add heptane (about 15 ml) into the flask, place the flask in an ultrasonic bath for 2 min. Allow to cool to ambient temperature. Dilute to volume with heptane. Mix thoroughly. (Solutions Cm).

Calibration solution (S-metolachlor standard solution). Weigh (to the nearest 0.1 mg) about 25 mg of the S-metolachlor standard into a volumetric flask (25 ml). Add heptane (about 15 ml) into the flask, place the flask in an ultrasonic bath for 2 min. Allow to cool to ambient temperature. Dilute to volume with heptane. Mix thoroughly. (Solutions Cs).

APPARATUS

High performance liquid chromatograph equipped with a detector suitable for operation at 230 nm, constant-temperature column compartment and an injector capable of delivering 10 μ l.

Column Daicel CHIRALPAK AY-H (250mm \times 4.6mm \times 5 μ m), or equivalent.

Electronic integrator or data system

Ultrasonic bath

PROCEDURE

(a) *Liquid chromatograph conditions* (typical):

Column Daicel CHIRALPAK AY-H (250mm \times 4.6mm \times 5 μ m), or equivalent.

Mobile phase Heptane – Ethanol, 94+6 (v/v)

<i>Column temperature</i>	30 °C
<i>Injection volume</i>	10 µl
<i>Flow rate</i>	0.6 ml/min
<i>Detector wavelength</i>	230 nm
<i>Run time</i>	25 min
<i>Retention time</i>	S1 isomer: about 14.8 min S2 isomer: about 16.2 min R1 isomer: about 17.8 min R2 isomer: about 19.8 min

(b) System equilibration. Inject 10 µl of the calibration solution C_m and repeat the injections until the retention times and peak areas vary by less than ± 2 % of the mean for three successive injections.

(c) *Preparation of sample solution.* Weigh (to the nearest 0.1 mg) into a volumetric flask (25 ml) sufficient sample to contain approximately 25 mg of S-metolachlor. Add heptane (about 15 ml) into the flask, place the flask in an ultrasonic bath for 2 min. Allow to cool to ambient temperature. Dilute to volume with heptane. Mix thoroughly. (Solutions S).

(d) *Determination.* Inject in duplicate 10 µl portions of the calibration solutions (C_m and C_s) and of the sample solutions (S₁, S₂,) in the following sequence:

C_m, C_s, S₁, S₂, S₃, S₄, C_m, C_s, S₅, S₆.....

Measure the relevant peak areas.

(e) *Calculation.*

$$P_S = \frac{H_S \times 100}{H_S + H_R}$$

$$P_R = (100 - P_S)$$

where:

H_S = peak area of total S-isomers

H_R = peak area of total R-isomers

P_S = Total S-isomers percentage (%)

P_R = Total R-isomers percentage (%)

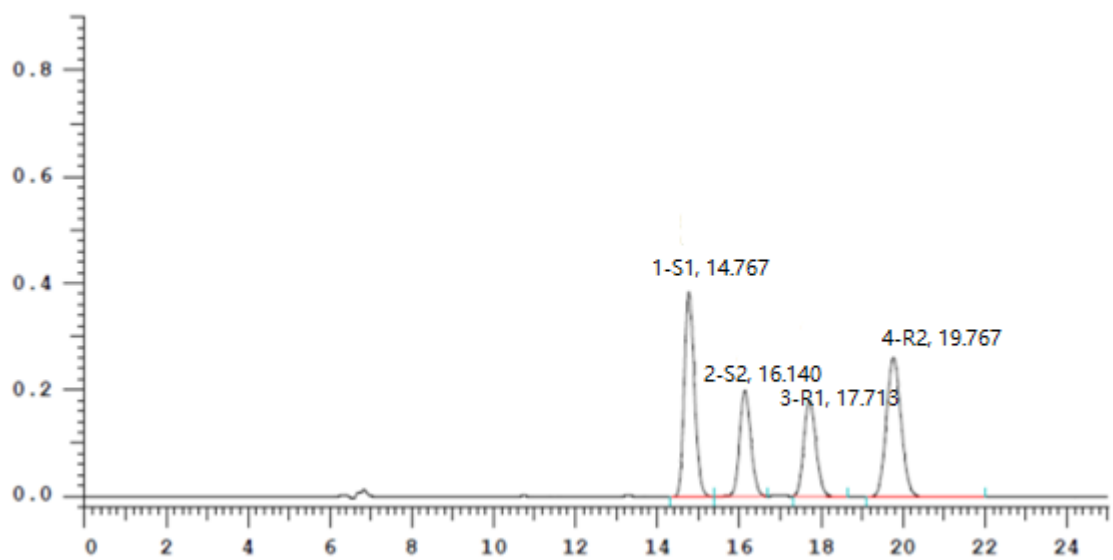


Fig 1 HPLC chromatogram of metolachlor racemate standard

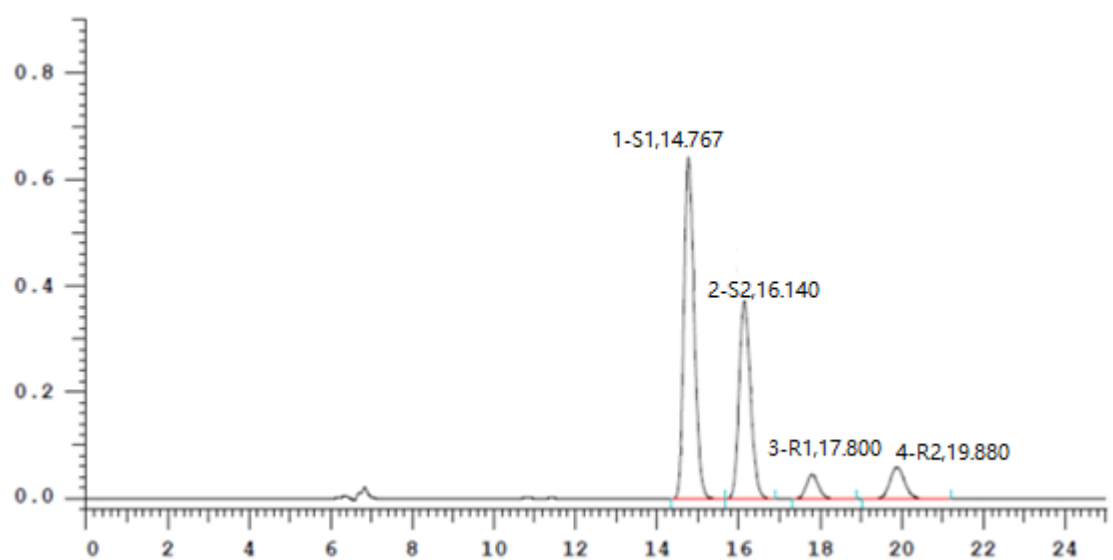


Fig 2 HPLC chromatogram of S-metolachlor standard

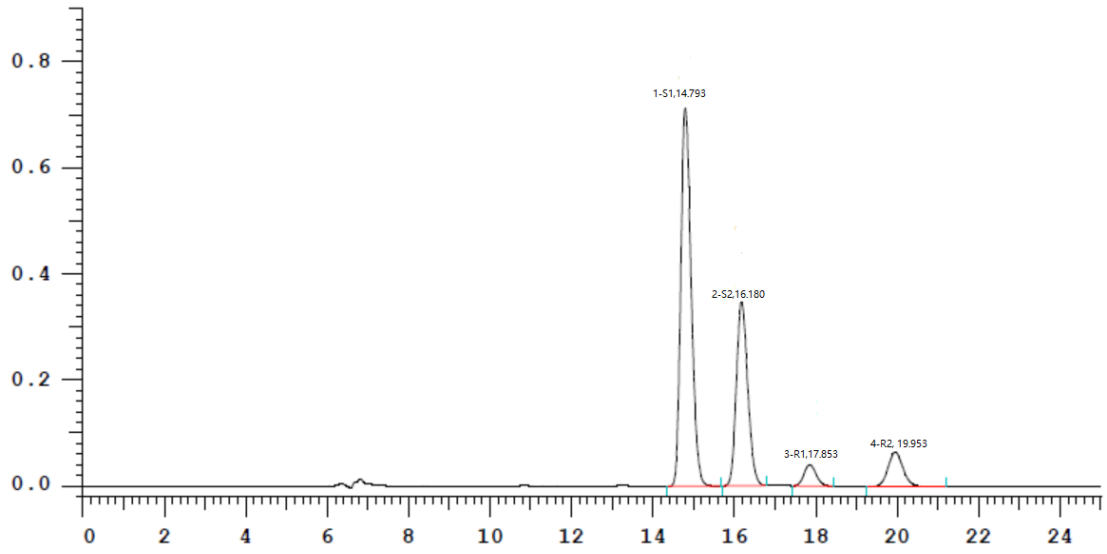


Fig 3 HPLC chromatogram of S-metolachlor TC

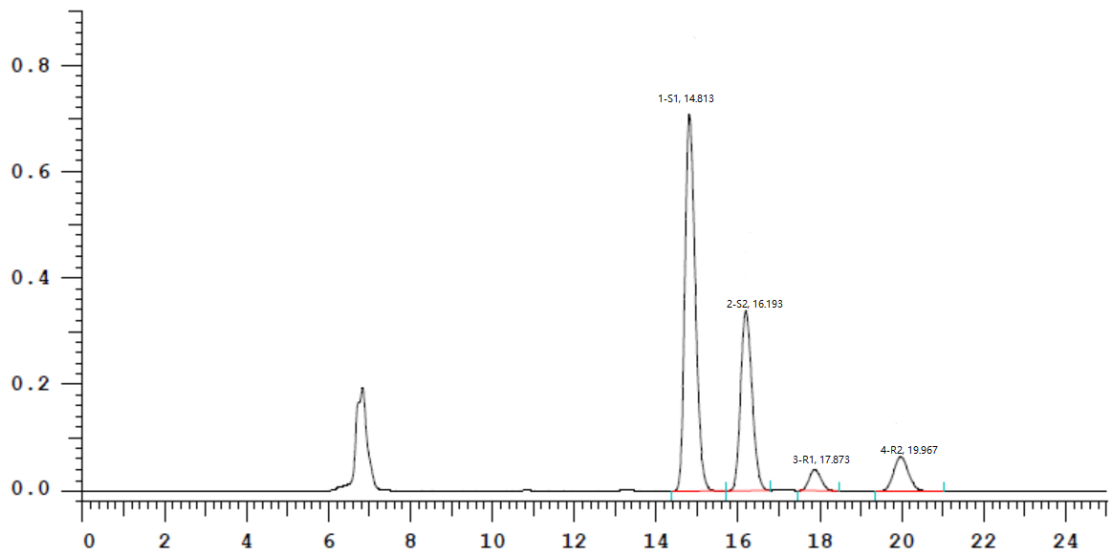


Fig 4 HPLC chromatogram of S-metolachlor EC