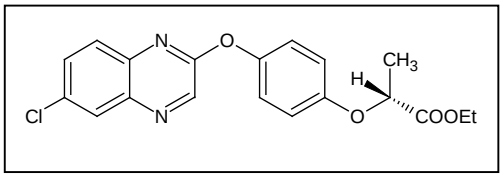
QUIZALOFOP-P-ETHYL

641



ISO common name	Quizalofop-P-ethyl
Chemical name	Ethyl (R)-2-[4-(6-Chloroquinoxalin-2-yloxy)phenoxy]propionate
Empirical formula	$C_{19}H_{17}ClN_2O_4$
RMM	372.8
m.p.	76.1~77.1 °C
v.p.	1.1 x 10^{-4} mPa at 20 °C
Solubility	In water, 6.1×10^{-4} g/l at 20 °C, pH 5.0 – 7.0; Xylene, ethyl acetate and acetone > 250 g/l, 1,2-dichloroethane > 1000 g/l at 22 – 23 °C; methanol 34.87 g/l, n-heptane 7.168 g/l at 20 °C
Description	Off-white powder
Stability	Stable at neutral and acidity condition.
Formulation	Emulsifiable concentrate

QUIZALOFOP-P-ETHYL TECHNICAL
641/TC/M/-

1. Sampling. Take at least 100 g.

2. Identity tests

2.1 GC. Use the GC method below. The relative retention time of Quizalofop-ethyl in the sample solution should not deviate by more than 1.5% from that of calibration solution.

2.2 HPLC. Use the HPLC method below. The relative retention time of Quizalofop-p-ethyl in the sample solution should not deviate by more than 1.5% from that of calibration solution.

2.3 Infrared. Prepare potassium bromide discs for the Quizalofop-p-ethyl technical sample and reference substance. Scan the discs from 4000-400 cm^{-1}. The spectrum produced from the sample should not differ significantly from that of the standard.

3. Quizalofop-ethyl

OUTLINE OF METHOD

Quizalofop-ethyl is determined by gas chromatography using HP-5 15 m X 0.53 mm X 1.5 µm film thickness capillary column using FID detector and di-n-octyl phthalate as internal standard.

REAGENTS

Acetone: HPLC grade
Quizalofop-ethyl reference standard of known purity: mixture of R and S isomers of defined composition.
Di-n-octyl phthalate, must not include impurity affect chromatographic analysis
Internal standard solution: Weigh approximately 4 g di-n-octyl phthalate (accurate to 10 mg) into 1000 ml brown volumetric flask, dissolve and dilute with acetone to volume. Mix thoroughly.
Calibration solution: prepare calibration solutions in duplicate. Weigh approximately (accurate to 0.1 mg) 50 mg of Quizalofop-ethyl working standard into 10 ml volumetric flask. Pipette accurately 10 ml internal standard solution into the flask and mix thoroughly (solution C_{A1} and C_{B1}).

APPARATUS

Gas chromatography equipped with FID detector
Chromatographic work station
Chromatographic column: HP-5 15 m X 0.53 mm X 1.5 µm film thickness

PROCEDURE

(a) Gas chromatographic conditions (typical):

Column HP-5 15 m X 0.53 mm X 1.5 µm film thickness coated with dimethyl polysiloxane (or equivalent)

Injection system

Injector: Split injection

Split Ratio: 10 : 1

Injection volume: 1.0 µl

Temperatures:

Column: 250 °C

Detector: 250 °C

Injector: 250 °C

Gas flow rates

Carrier gas (high purity Nitrogen) flow rate: 15 ml/min.

Hydrogen: approximately 30 ml/min.

Air: approximately 300 ml/min.

Retention time

Di-n-octyl phthalate: approximately 2.5 min,

Quizalofop-ethyl: approximately 3.5 min.

(b) Sample preparation: Weigh (accurate to 0.1mg) sufficient sample to contain about 50 mg Quizalofop-ethyl into 10 ml volumetric flask. Pipette accurately 10 ml internal standard solution into the flask and mix thoroughly. Prepare in duplicate (S_{11} and S_{21})

(c) Equilibration of the chromatographic system. Inject the calibration solution and repeat the injections until retention times and the response factors calculated from the peak areas vary by less than 1% for successive injections.

(d) Determination: Inject in duplicate 1 µl portions of each sample solution bracketing them by injections of the calibration solution as follows: C_{A1}, $S_{11,}$ $S_{11,}$ $C_{B1,}$ $S_{21,}$ $S_{21,}$ $C_{A1,}$ and so on. Measure the relevant peak areas.

(e) Calculation

$$f_i = \frac{I_r \times s \times P}{H_s \times r}$$

$$\text{Content of Quizalofop} - \text{ethyl } (X1) = \frac{H_w \times f \times q}{I_q \times w} \text{ g/kg}$$

where:

f_i = individual response factor

f = mean response factor

H_s = peak areas of quizalofop-ethyl in the calibration solution

I_r = peak areas of di-n-octyl phthalate in the calibration solution

H_w = peak areas of quizalofop-ethyl in the sample solution

I_q = peak areas of di-n-octyl phthalate in the sample solution

s = mass of quizalofop-ethyl in the calibration solution (mg)

r = mass of di-n-octyl phthalate in the calibration solution (mg)

q = mass of di-n-octyl phthalate in the sample solution (mg)

w = mass of sample taken (mg)

P = purity of quizalofop-ethyl standard (g/kg)

4. Quizalofop-P-ethyl

OUTLINE OF METHOD Quizalofop-P-ethyl(R-enantiomer) is separated from the s-enantiomer and determined by normal phase HPLC on chiral column using UV detector at 237 nm.

REAGENTS
n-Hexane: HPLC grade
Isopropanol: HPLC grade
Quizalofop-P-ethyl reference standard of known purity: mixture of R and S isomers of defined composition (50:50).
Preparation of calibration solution in duplicate: Weigh approximately (accurate to 0.1mg) 10 mg Quizalofop-p-ethyl standard into 10 ml volumetric flask. Dissolve to the mark with mobile phase and mix thoroughly (Solution C_{A2} and C_{B2}).

APPARATUS
High-performance liquid chromatography equipped with UV detector
Column stainless steel: 250 mm X 4.6 mm (id), CHIRALPAKAD-H, 5 μm, or equivalent
Chromatographic work station
Ultrasonic bath

PROCEDURES
(a) Liquid Chromatographic Conditions (typical)
Mobile phase: n-hexane + Isopropanol = 90 + 10 (v/v)
Flow rate: 0.6 ml/min
Detector wavelength: 237 nm
Injection volume: 5 μl
Column temperature: 25 °C
Retention time:
S-isomer approximately 18.7 min,
Quizalofop-p-ethyl approximately 17.1 min.

(b) System equilibration. Inject 5 µl portions of C_{A2} until the peak areas for both R-isomer and S-isomer deviate less than 1.0% for the two successive injections and the area ratio of R-isomer and S-isomer should be 50 : 50 and the deviation should be less than 1%.

(c) Preparation of sample solution: Weigh (accurate to 0.1mg) sufficient sample to contain about 10 mg Quizalofop-p-ethyl into 10 ml volumetric flask. Dissolve to the mark with mobile phase and mix thoroughly. Filter through 0.45 µm filter membrane if necessary. Prepare in duplicate (S_{12} and S_{22})

(d) Determination: Inject in duplicate 1 µl portions of each sample solution bracketing them by injections of the calibration solution as follows: C_{A2} $S_{12,}$ $S_{12,}$ C_{B2} $S_{22,}$ $S_{22,}$ C_{A2} and so on. Measure the relevant peak areas.

(e) Calculation

$$K = \frac{H_R}{H_R + H_S} \times 100\%$$

Content of Quizalofop-p-ethyl = $X_1 \times K$

Where:

H_R = mean average of the peak areas of R-isomer in the two sample solutions;

H_S = mean average of the peak areas of S-isomer in the two sample solutions;

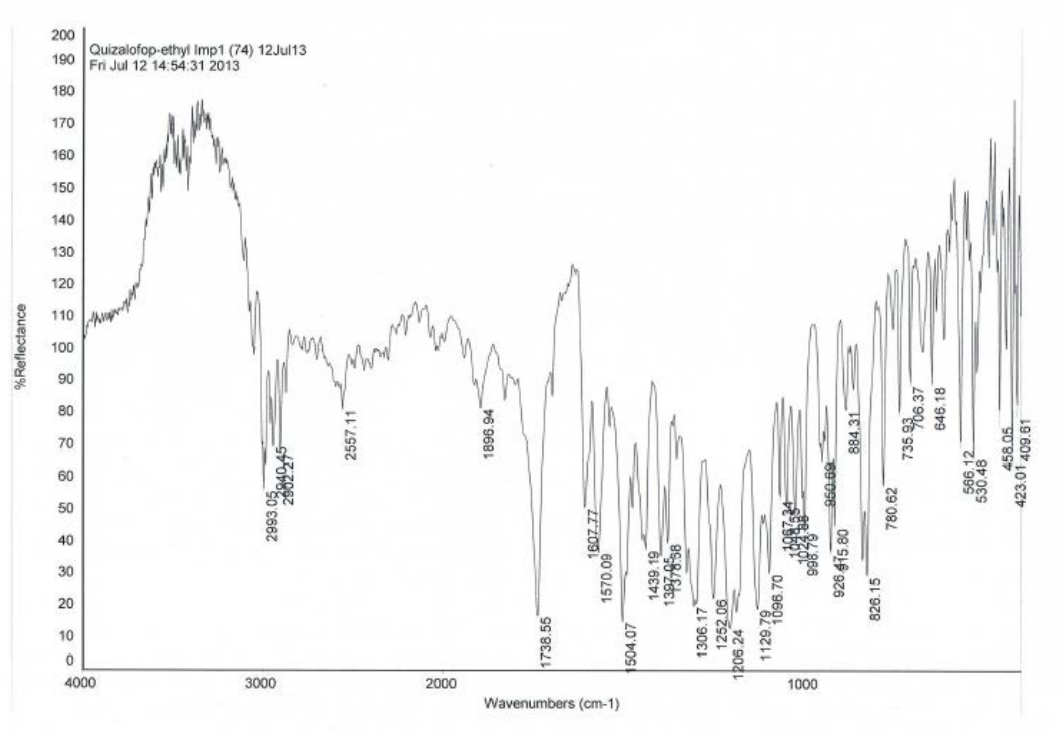


Fig. 1 Infrared spectra of Quizalofop-p-ethyl

QUIZALOFOP-P-ETHYL EMULSIFIABLE CONCENTRATE

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1. **Sampling.** Take at least 1 l.

2. **Identity tests.** As for Quizalofop-p-ethyl technical ******

3. **Quizalofop-p-ethyl.** As for Quizalofop-p-ethyl technical ***** except：

Sample preparation：

Quizalofop-ethyl： Weigh (accurate to 0.1 mg) sufficient sample to contain about 50 mg Quizalofop-ethyl into 10 ml volumetric flask. Pipette accurately 10 ml internal standard solution into the flask and mix throughly. Prepare in duplicate (S_{11} and S_{21})

Quizalofop-P-ethyl： Weigh accurately sufficient sample to contain about 10 mg Quizalofop-p-ethyl into 10 ml volumetric flask. Dissolve to the mark with mobile phase and mix throughly. Use 0.45 μm filter membrane if necessary. Prepare in duplicate (S_{12} and S_{22})

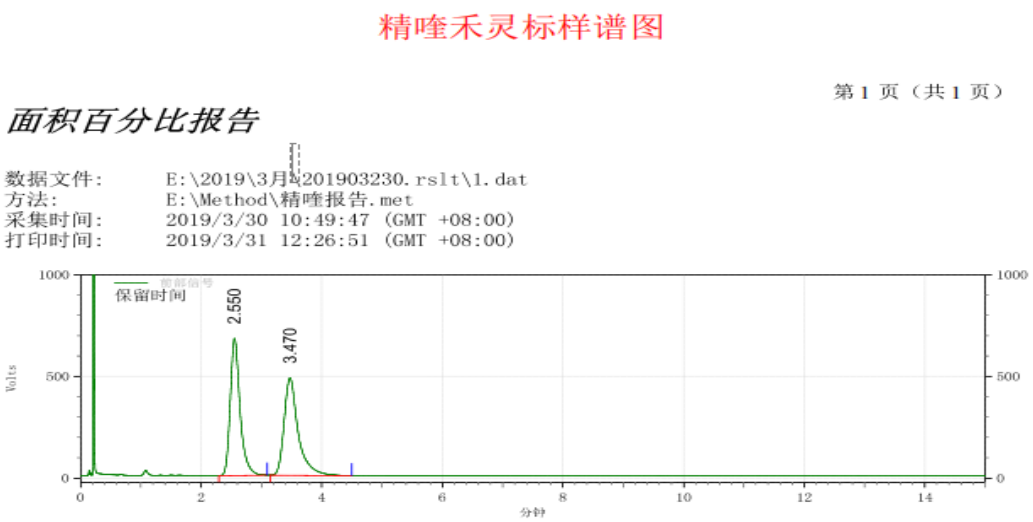

Fig. 2 Chromatogram of Quizalofop-ethyl standard

精喹禾灵原药样品谱图

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面积百分比报告

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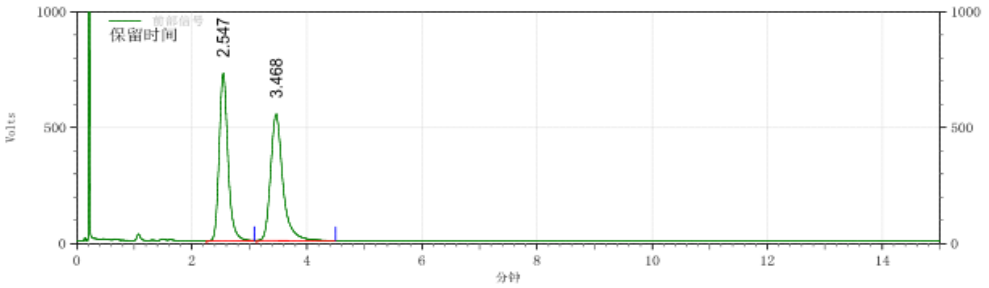

Fig. 3 Chromatogram of Quizalofop-p-ethyl TC sample

精喹禾灵乳油（10%）标样谱图

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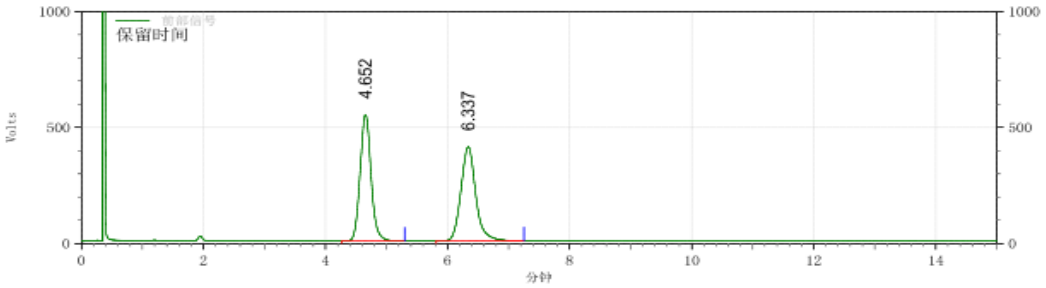

Fig. 4 Chromatogram of Quizalofop-ethyl EC standard

精喹禾灵乳油（10%）样品谱图

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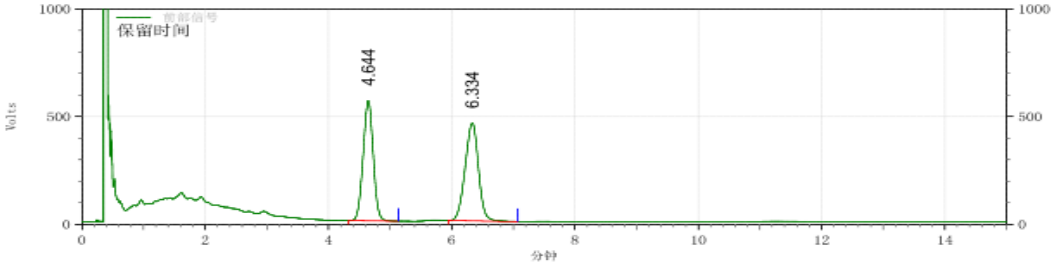

Fig. 5 Chromatogram of Quizalofop-p-ethyl EC sample

标样RS体

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实验者：FL
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使用仪器类型:液相色谱
仪器型号:515/2489
柱温(℃):25
柱型号:AD-H

梯度方式:高压梯度

检测器:紫外
波长(nm):237

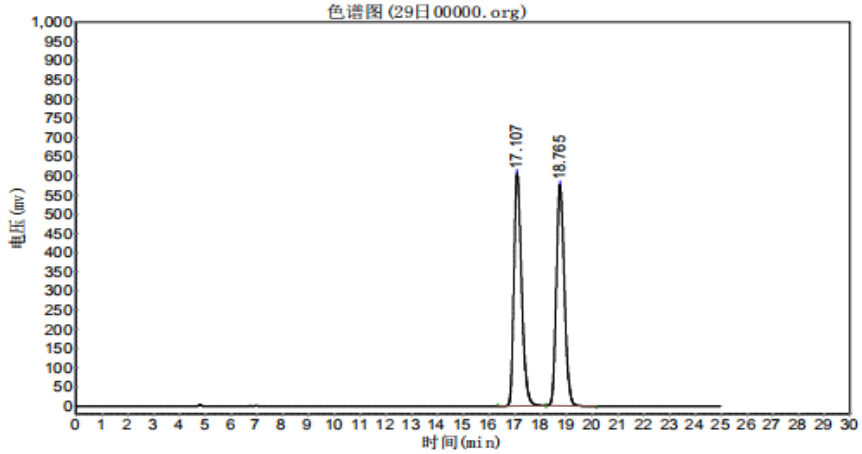

Fig. 6 Chromatogram of Quizalofop-ethyl standard (mixture of R and S)

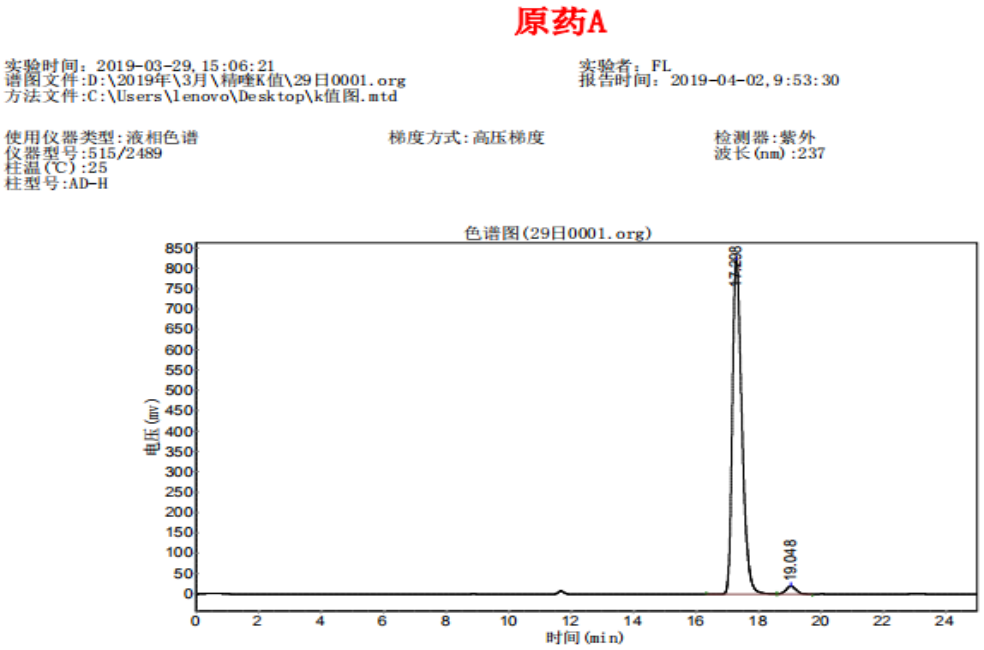

Fig. 7 Chromatogram of Quizalofop-p-ethyl TC sample