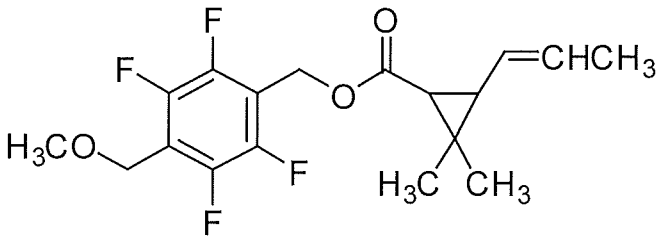
METOFLUTHRIN

993



ISO common name	Metofluthrin
Chemical name	2,3,5,6-tetrafluoro-4-(methoxymethyl)benzyl (1*RS*,3*RS*;1*RS*,3*SR*)-2,2-dimethyl-3-[(*EZ*)-prop-1-enyl]cyclopropanecarboxylate (IUPAC); [2,3,5,6-tetrafluoro-4-(methoxymethyl)phenyl] methyl 2,2-dimethyl-3-(1-propen-1-yl) cyclopropanecarboxylate (CA)
CAS No.	240494-70-6
Empirical formula	$C_{18}H_{20}F_4O_3$
RMM	360.3
Boiling point	340.36°C
v.p.	8.969 x 10^{-4} Pa (20°C)
Solubility	In water, 0.441 µg/l; acetone, >250 g/l; *p*-xylene, >250 g/l; methanol, >250 g/l; ethyl acetate, >250 g/l; 1,2-dichloroethane, >250 g/l; *n*-heptane, >250 g/l
Description	Very pale yellow to pale yellow clear liquid

Note: Metofluthrin is the ISO common name for the mixture of eight isomers. The isomers are designated as follows:
RTZ, *STZ*, *RCE*, *SCE*, *RCE*, *SCE*, *RTE* and *STE* isomers

METOFLUTHRIN
993/TC/m/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 GLC. Use the GLC method below. The relative retention time of metofluthrin (peak A) with respect to the internal standard for the sample solution should not deviate by more than 1% from that for the calibration solution.

2.2 HPLC. Under investigation

3 Metofluthrin

OUTLINE OF METHOD Metofluthrin is determined by capillary gas chromatography using flame ionisation detection and fluoranthene as internal standard.

REAGENTS

Acetone

Metofluthrin standard of known purity. Store refrigerated.

Fluoranthene internal standard. Must not show peaks with the same retention times as metofluthrin (peaks A and B).

Internal standard solution. Dissolve fluoranthene (0.6 g) in acetone (100 mL). Ensure that a sufficient quantity of this solution is prepared for all samples and calibration standards to be analysed.

Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) 90 to 110 mg (*s* mg) of metofluthrin standard into separate vials (50 ml). Add by pipette internal standard solution (10.0 ml) and acetone (about 30 ml). Dissolve the standard and mix well (Solutions C_A and C_B).

APPARATUS

Gas chromatograph equipped with a split/splitless injection and a flame ionisation detector.

Capillary column fused silica, 30 m × 0.25 mm (i.d.), film thickness: 0.25 μm, coated with crosslinked 5% phenyl polysiloxane 95% dimethyl polysiloxane (DB-5 or equivalent)

Electric integrator or *data system*

PROCEDURE

(a) Gas chromatographic conditions (typical):

Column	fused silica, 30 m × 0.25 mm, film thickness: 0.25 µm, coated with crosslinked 5% phenyl polysiloxane 95% dimethyl polysiloxane (DB-5 or equivalent)
Injection system	
Injector	split injection
Split flow	approximately 50 ml/min
Injection volume	1 µl
Detector	flame ionisation
Temperatures	
Column oven	160°C
Injector	300°C
Detector	325°C
Carrier gas	helium, 30 cm/sec
Retention times	metofluthrin:
	peak A: about 26 min
	peak B: about 27 min
	fluoranthene: about 29 min

Note: peak A consists of the *RTZ*, *STZ*, *RCE* and *SCE* isomers
peak B consists of the *RCE*, *SCE*, *RTE* and *STE* isomers

(b) Linearity check. Check the linearity of the detector response by injecting 1 µl of solutions with metofluthrin concentrations 0.5, 1 and 2 times that of the calibration solution before conducting analysis.

(c) System equilibration. Prepare two calibration solutions. Inject 1 µl portions of the first one until the response factors obtained for two consecutive injections differ by less than 1.0%. Then inject a 1 µl portion of the second solution. The response factor for this solution should not deviate by more than 1.0% from that for the first calibration solution, otherwise prepare new calibration solutions.

(d) Preparation of sample solution. Weigh in duplicate (to the nearest 0.1 mg) sufficient sample to contain about 100 mg (*w* mg) of metofluthrin into separate vials (50 ml). Add by pipette internal standard solution (10.0 ml) and acetone (about 30 ml). Dissolve the standard and mix well (Solutions S_A and S_B).

(e) Determination. Inject in duplicate 1 µl portions of each sample solution bracketing them by injections of the calibration solutions as

follows; calibration solution C_A, sample solution S_A, sample solution S_A, calibration solution C_B, sample solution S_B, sample solution S_B, calibration solution C_A, and so on. Measure the relevant peak areas.

(f) Calculation. Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating the metofluthrin contents of the bracketed sample injections.

$$f_i = \frac{I_r \times s \times P}{H_S}$$

$$\text{Metofluthrin content} = \frac{f \times H_w}{I_q \times w} \text{(g/kg)}$$

where:

f_i = individual response factor
f = mean response factor
H_s = total peak area of metofluthrin (peak A+B) in the calibration solution
H_w = total peak area of metofluthrin (peak A+B) in the sample solution
I_r = peak area of internal standard in the calibration solution
I_q = peak area of internal standard in the sample solution
s = mass of metofluthrin standard in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of metofluthrin standard (g/kg)

Repeatability r = 3.7 g/kg at 967 g/kg active ingredient content
Reproducibility R = 5.3 g/kg at 994 g/kg active ingredient content

METOFLUTHRIN EMULSIONS, OIL IN WATER
993/EW/m/-

1 Sampling. Take at least 1L.

2 Identity tests

2.1 GLC. Use the GLC method below. The relative retention time of metofluthrin (peak A) with respect to the internal standard for the sample solution should not deviate by more than 1% from that for the calibration solution.

2.2 HPLC. Under investigation

3 METOFLUTHRIN. As for **993**/TC/m/3 except:

REAGENTS

Sodium chloride

Internal standard solution. Dissolve fluoranthene (30 mg) in acetone (100 mL). Ensure that a sufficient quantity of this solution is prepared for all samples and calibration standards to be analysed.

Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) 90 to 110 mg (*s* mg) of metofluthrin standard into separate volumetric flasks (50 ml). Dissolve and make up to volume with acetone. Transfer by pipette 5.0 ml of these solutions into separate volumetric flasks (20 ml) and make up to volume with acetone. Transfer by pipette 5.0 ml of these solutions into separate vials (20 ml), add by pipette internal standard solution (5.0 ml) and mix well (Solutions C_A and C_B).

APPARATUS

Ultrasonic bath

PROCEDURE

(a) Gas chromatographic conditions (typical)

Injection system

Split flow	approximately 10 ml/min
Injection volume	2 µl

Temperatures

Column oven	160°C (use a short temperature program to remove formulation components, if necessary)

(b) Linearity check. Check the linearity of the detector response by injecting 2 µl of solutions with metofluthrin concentrations 0.5, 1 and 2 times that of the calibration solution before conducting analysis.

(c) System equilibration. Prepare two calibration solutions. Inject 2 µl portions of the first one until the response factors obtained for two consecutive injections differ by less than 2.0%. Then inject a 2 µl portion of the second solution. The response factor for this solution should not deviate by more than 2.0% from that for the first calibration solution, otherwise prepare new calibration solutions.

(d) Preparation of sample solution. Homogenize the sample by vigorous shaking. Weigh in duplicate (to the nearest 0.1 mg) sufficient sample to contain about 2.5 mg (*w* mg) of metofluthrin into separate vials (20 ml). Add by pipette internal standard solution (5.0 ml) and acetone (about 5 ml), and place the vials in an ultrasonic bath for 5 min. Add about 1 g of sodium chloride and mix thoroughly. Filter a portion of each sample solution through a 0.45 µm filter prior to analysis (Solutions S_A and S_B).

(e) Determination. Inject in duplicate 2 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows; calibration solution C_A, sample solution S_A, sample solution S_A, calibration solution C_B, sample solution S_B, sample solution S_B, calibration solution C_A, and so on. Measure the relevant peak areas.

(f) Calculation. Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating the metofluthrin contents of the bracketed sample injections.

$$f_i = \frac{I_r \times s \times P}{H_S}$$

$$\text{Metofluthrin content} = \frac{f \times H_w}{I_q \times w} \times \frac{1}{40} \text{(g/kg)}$$

Repeatability r = 0.032 g/kg at 0.962 g/kg active ingredient content
Reproducibility R = 0.046 g/kg at 0.962 g/kg active ingredient content

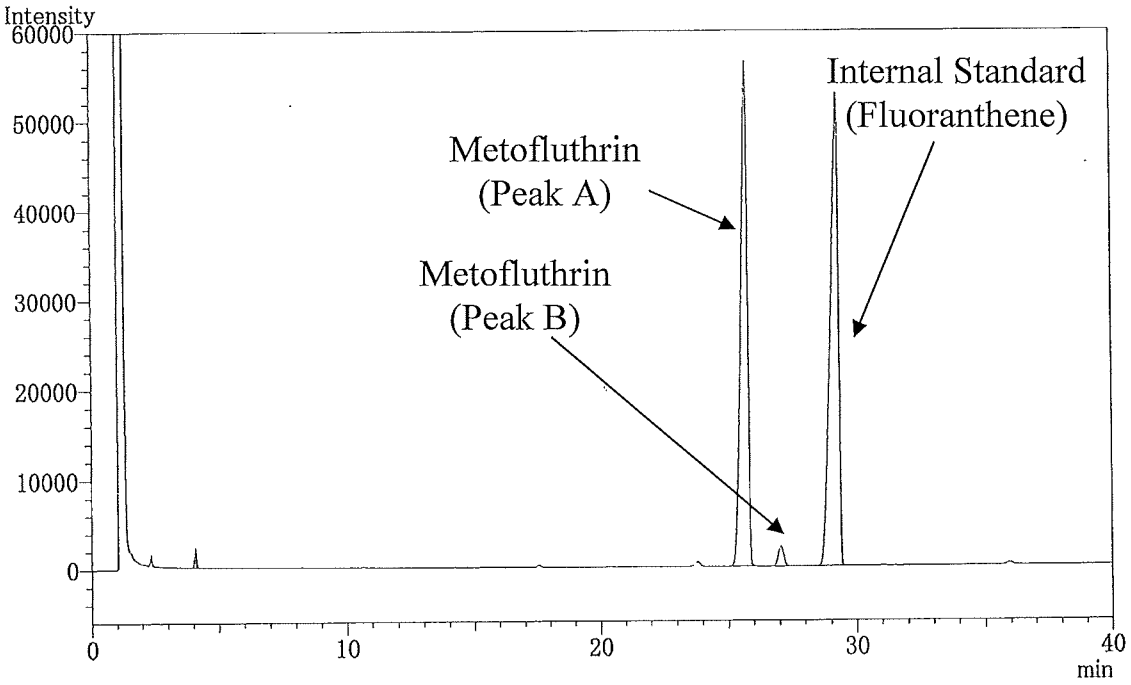


Figure 1 Example of Gas Chromatogram of Metofluthrin TC

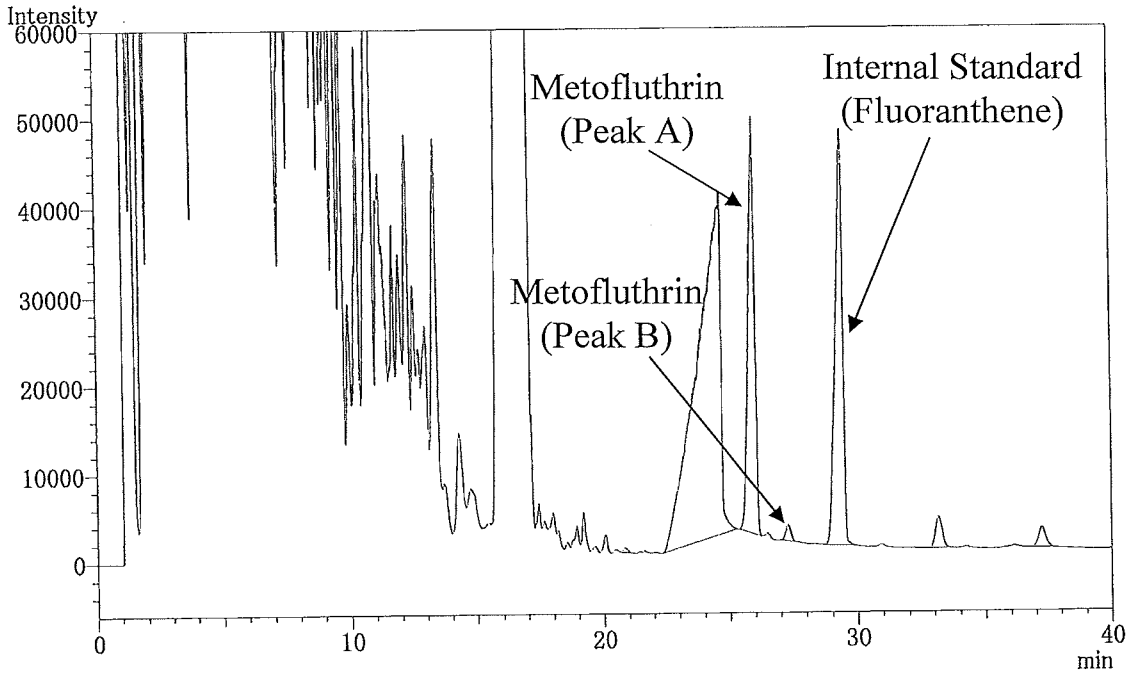


Figure 2 Example of Gas Chromatogram of Metofluthrin EW