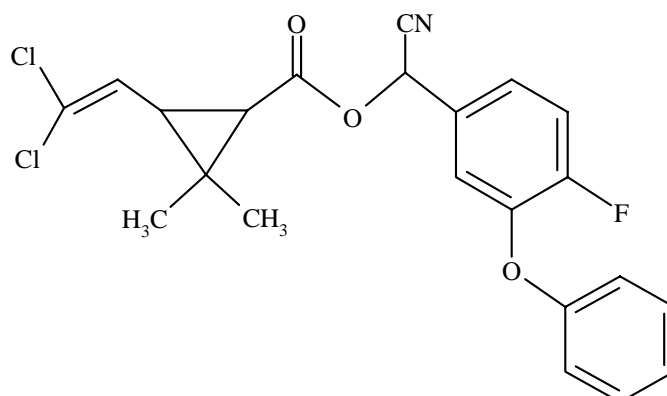


CYFLUTHRIN
385



| | |
|--------------------------|--|
| <i>ISO common name</i> | Cyfluthrin |
| <i>Chemical name</i> | (<i>RS</i>)- α -Cyano-(4-fluoro-3-phenoxybenzyl)- (1 <i>RS</i>)-cis-trans-3-(2,2-dichlorovinyl)-2,2- dimethylcyclopropane carboxylate(IUPAC); (\pm)cis,trans[cyano-(4-fluoro-3-phenoxy- phenyl)-methyl]-3-(2,2-dichloroethenyl)-2,2- dimethyl-cyclopropanecarboxylate(CA; 68359- 37-5) |
| <i>Empirical formula</i> | C ₂₂ H ₁₈ Cl ₂ FNO ₃ |
| <i>RMM</i> | 434.3 |
| <i>m.p.</i> | ca. 60 °C |
| <i>Vapour pressure</i> | Cis I isomer: 1×10^{-5} Pa; cis II isomer: 1×10^{-5} Pa; trans I isomer: 2×10^{-5} Pa; trans II isomer: 9×10^{-5} Pa, all at 20 °C |
| <i>Solubility</i> | Water at 20 °C: 2 μ g/l; readily soluble in toluene, acetone, trichloromethane, heptane and tetrahydrofuran |
| <i>Description</i> | The pure material is a viscous amber partly crystalline oil |
| <i>Stability</i> | Stable under normal storage conditions |
| <i>Formulations</i> | Emulsifiable concentrates, oil in water emulsion and wettable powders |

Note: Cyflutrin is a mixture of four diastereoisomeric forms, each of which is present as a pair of enantiomers. The four diastereoisomers are designated as follows: cis I: 1*R*,3*R*, α *R* + 1*S*,3*S*, α *S*; cis II: 1*R*,3*R*, α *S* + 1*S*,3*S*, α *R*; trans I: 1*R*,3*S*, α *R* + 1*S*,3*R*, α *S*; trans II: 1*R*,3*S*, α *S* + 1*S*,3*R*, α *R*.

CYFLUTHRIN TECHNICAL 385/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The relative retention times of the peaks in the sample solution with respect to isomer *cis* II should not deviate by more than 2 % from that of the calibration solution and the intensities of the four cyfluthrin isomers should give the same pattern as in the calibration solution.

2.2 TLC. Carry out a thin layer chromatography with the sample and the standard using the following conditions:

| | |
|------------------------------|---|
| <i>TLC plate</i> | Coated with silica gel 60 F ₂₅₄ , 0.25 mm (Merck, Darmstadt, art no. 5729 or equivalent) |
| <i>Solvent</i> | <i>n</i> -heptane-acetone, 3 + 2 (v/v) |
| <i>Sample solution</i> | Weigh into a conical flask enough sample to contain about 200 mg cyfluthrin and dissolve in acetone (10 ml). Centrifuge or filter if necessary. |
| <i>Reference solution</i> | Weigh into a conical flask about 200 mg cyfluthrin reference substance and dissolve in acetone (10 ml). |
| <i>Loading</i> | 5 µl (about 100 µg) applied in a line shaped spot |
| <i>Separation</i> | Develop the chromatogram in two steps, drying the plate between each step; first step: 5 cm; second step: 10 cm. |
| <i>Visualisation reagent</i> | Dissolve <i>o</i> -toluidine (0.16 g) in acetic acid (30 ml), add potassium iodide (1 g), dissolve and fill to 500 ml with distilled water. |
| <i>Visualisation</i> | Dry the plate under a hood. Examine the separation in the UV light (254 nm). Put the plate in a chamber filled with chlorine gas (produced by adding concentrated hydrochloric acid to potassium permanganate in a chromatography tank) for 30 s. Dry the plate in a stream of cold air. Then, immerse the plate in the visualisation solution. |
| <i>R_F value</i> | approximately 0.5 (mean value for the double spot). |

A bluish grey coloured double spot is obtained for the active ingredient by reaction with the *o*-toluidine reagent. To distinguish the product from cypermethrin, reference substances of cyfluthrin and cypermethrin should be chromatographed on the same plate. The TLC method is not suitable for differentiating between cyfluthrin and beta-cyfluthrin.

2.3 Infrared. Dissolve 55 mg quantities of the sample and of cyfluthrin reference standard in 1 ml carbon tetrachloride. Transfer the solutions to 100 μm NaCl cells and scan the solutions from 600 to 4000 cm^{-1} . The spectrum obtained from the sample solution should not differ significantly from that of the solution of the standard.

Note: Cyfluthrin (Fig. 10) can not be distinguished from beta-cyfluthrin (Fig. 11) by infrared spectroscopy, but cypermethrin shows a slightly different spectrum (Fig. 12).

2.4 ^1H -NMR spectroscopy. Dissolve 2 mg quantities of the sample and of cyfluthrin reference standard in 2 to 3 ml deuterodichloromethane containing tetramethylsilane as internal standard. Using a 200 MHz instrument, record the NMR-spectrum at 21 $^{\circ}\text{C}$ in a 5 mm NMR-tube. The NMR-spectra of beta-cyfluthrin, cyfluthrin and cypermethrin display the following characteristics (Fig. 13):

a) Beta-cyfluthrin (2 diastereomers)

trans II: doublet at 5.66 ppm

cis I: doublet at 6.18 ppm

b) Cyfluthrin (4 diastereomers)

trans I: doublet at 5.63 ppm

trans II: doublet at 5.66 ppm

cis I: doublet at 6.17 ppm

cis II: doublet at 6.15 ppm

c) Cypermethrin (4 diastereomers)

The aromatic protons of cypermethrin and cyfluthrin (6.95 - 7.05 ppm) have different coupling constants. Cyfluthrin shows a significant doublet at 7.00 ppm, which can be used to distinguish cyfluthrin from cypermethrin.

3 Cyfluthrin

3.1 Normal phase high performance liquid chromatographic method*

SCOPE The method is intended for the determination of total cyfluthrin and for the determination of the diastereoisomer ratio.

OUTLINE OF METHOD Cyfluthrin is determined by normal phase high performance liquid chromatography using UV detection at 235 nm and external standardisation.

* CIPAC method 1996. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, Germany.

REAGENTS

Cyfluthrin working standard with known contents of the four diastereoisomers

n-Heptane HPLC quality

tert-Butyl methyl ether (TBME), HPLC quality

Tetrahydrofuran (THF), HPLC quality

Eluent *n*-heptane-TBME, 950 + 50 (v/v)

Calibration solution. Homogenise the cyfluthrin working standard by warming it in a hot-air cabinet at 60 to 80 °C for about 30 min. After melting, mix the material thoroughly by shaking. Weigh (to the nearest 0.1 mg) in duplicate into two volumetric flasks (50 ml) cyfluthrin working standard (about 100 mg, *s* mg). The quantities weighed should differ about 10 %. Add TBME (15 ml) and place the flasks in an ultrasonic bath for 5 min. Fill the flasks to 1 cm below the mark with *n*-heptane and place them in a water bath at 22 °C for 5 min. Make up to volume with *n*-heptane and mix well (Solutions C₁ and C₂). The solutions are stable for 24 h at room temperature.

APPARATUS

High performance liquid chromatograph equipped with an ultraviolet spectrophotometric detector and an injection system capable of injecting 5 or 10 µl

Column stainless steel, 250 × 4 or 3 mm (i.d.), LiChrospher Si 60, 5 µm or equivalent

Electronic integrator

Ultrasonic bath

Centrifuge

PROCEDURE

(a) *Conditions of chromatography* (typical)

Column temperature 40 °C or room temperature

Flow rate 1.8 ml/min or 1 ml/min for the column with a diameter of 3 mm

Measuring wavelength 235 nm

Injection volume 5 µl and 10 µl respectively

Run time approximately 20 min

Retention times

| | |
|------------------|---------------|
| isomer cis I: | about 6.5 min |
| isomer cis II: | about 5.9 min |
| isomer trans I: | about 8.5 min |
| isomer trans II: | about 7.3 min |

(b) *Sample preparation.* Homogenise the sample by warming it at 60 to 80 °C for about 30 min. After melting, mix the material thoroughly by shaking. Weigh (to the nearest 0.1 mg) in duplicate into two volumetric flasks (50 ml) sufficient sample to contain about 100 mg (w mg) cyfluthrin. The quantities weighed should differ about 10 %. Add TBME (15 ml) and place the flasks in an ultrasonic bath for 5 min. Fill the flasks to 1 cm below the mark with n -heptane and place them in a water bath at 22 °C for 5 min. Make up to volume with n -heptane and mix well (Solutions S_1 and S_2). The solutions are stable for 24 h at room temperature.

(c) *Equilibration of the system.* Pump sufficient eluent through the column to equilibrate the system. When a new column is installed, equilibrate the system at least over night. Inject 5 μ l portions of the calibration solution and repeat the injections until retention times and peak areas vary by less than 0.5 % of the mean for successive injections.

(d) *Determination.* Inject 5 μ l portions of the calibration solutions (C_1 and C_2) and the sample solutions (S_1 and S_2) in the following sequence: C_1 , S_1 , S_2 , C_2 . Determine the peak area of each individual isomer. Calculate the mean of the response factors (f_i) of the calibration solutions bracketing the injections of the sample solutions and calculate the content. If individual measurements vary by more than 0.8 %, prepare new solutions.

(e) *Calculation*

$$f_i = \frac{s \times c_i}{H_{si}}$$

$$\text{Content of the } i\text{th cyfluthrin isomer } (Y_i) = \frac{H_{wi} \times f_i}{w} \text{ g/kg}$$

$$\text{Total cyflythrin content} = \sum Y_i \text{ g/kg } (i=1-4)$$

$$\text{Ratio of the } i\text{th cyfluthrin isomer} = \frac{Y_i}{\sum Y_i} \text{ } (i=1-4)$$

where:

f_i = response factor of the i th cyfluthrin isomer

H_{si} = peak area of the i th cyfluthrin isomer in the calibration solution

H_{wi} = peak area of the i th cyfluthrin isomer in the sample solution

s = mass of cyfluthrin in the calibration solution (mg)

w = mass of sample taken (mg)

c_i = content of the i th isomer in the cyfluthrin standard (g/kg)

Y_i = content of the i th isomer in the sample (g/kg)

Repeatability r = 30 g/kg at 950 g/kg active ingredient content

Reproducibility R = 33 g/kg at 950 g/kg active ingredient content

3.2 Reversed phase liquid chromatographic method*

SCOPE The method is intended for the determination of the total cyfluthrin content only.

OUTLINE OF METHOD Cyfluthrin is separated from other components by reversed phase high performance liquid chromatography and quantified by external standardisation.

REAGENTS

Cyfluthrin working standard with known content of cyfluthrin

Tetrahydrofuran HPLC quality

Acetonitrile HPLC quality

Methanol HPLC quality

Water HPLC quality

n-Heptane HPLC quality

Eluent A acetonitrile-methanol-water, 23 + 49 + 28 (v/v)

Eluent B acetonitrile

Calibration solution. Homogenise the cyfluthrin working standard by warming it in a hot-air cabinet at 60 to 80 °C for about 30 min. After melting, mix the material thoroughly by shaking. Weigh (to the nearest 0.1 mg) in duplicate into two volumetric flasks (100 ml) cyfluthrin working standard (about 100 mg, *s* mg). The quantities weighed should differ about 10 %. Add acetonitrile (40 ml) and place the flasks in an ultrasonic bath for 5 min. Allow to cool to room temperature, make up to volume with acetonitrile and mix well (Solutions C₁ and C₂). The solutions are stable for 24 hours.

APPARATUS

High performance liquid chromatograph equipped with an ultraviolet spectrophotometric or diode array detector and an injection system capable of injecting 10 µl

Column stainless steel, 250 × 4 (i.d.) mm, LiChrospher 100 RP 18, 5 µm or equivalent

Electronic integrator

Ultrasonic bath

Centrifuge

Disposable filter e. g. Chromafil, Macherey and Nagel, art. no. 718005

Extraction column 3 ml, loaded with 500 mg stationary phase SiOH 40 µm, 60 Å, (Bakerbond spe, art.no. 7086-03), or equivalent

* CIPAC method 1996. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, Germany.

PROCEDURE

(a) Operating conditions (typical):

| | | | |
|----------------------------|---|-----|-----------|
| <i>Eluent A</i> | acetonitrile-methanol-water, 23+49+28 (v/v) | | |
| <i>Eluent B</i> | acetonitrile | | |
| <i>Gradient</i> | time (min) | A | B (% v/v) |
| | 0 | 100 | 0 |
| | 25 | 100 | 0 |
| | 26 | 20 | 80 |
| | 28 | 20 | 80 |
| | 29 | 100 | 0 |
| <i>Flow rate</i> | 1.5 ml/min | | |
| <i>Column temperature</i> | 40 °C | | |
| <i>Detector wavelength</i> | 235 nm | | |
| <i>Injection volume</i> | 10 µl | | |

If another injection volume has to be used for technical reasons, quantities weighed or dilutions steps must be adapted in such way that the signal intensity is in the linear range of the detector.

| | | | |
|------------------------|------------------|--------------|--|
| <i>Run time</i> | 32 min | | |
| <i>Retention times</i> | isomer trans II: | about 20 min | |
| | isomer cis I: | about 23 min | |

(b) Sample preparation. Homogenise the sample by warming it in a hot-air cabinet at 60 to 80 °C for about 30 min. After melting, mix the material thoroughly by shaking. Weigh (to the nearest 0.1 mg) in duplicate into two volumetric flasks (100 ml) enough sample to contain about 100 mg (*w* mg) cyfluthrin. The quantities weighed should differ about 10 %. Add acetonitrile (40 ml) and place the flasks in an ultrasonic bath for 5 min. Allow to cool to room temperature, make up to volume with acetonitrile and mix well (Solutions S₁ and S₂). The solutions are stable for 24 hours.

(c) Equilibration of the chromatographic system. Pump sufficient eluent through the column to equilibrate the system. Inject 10 µl portions of the calibration solution and repeat the injections until retention times and peak areas vary by less than 0.5 % of the mean for successive injections.

Note: Under the given conditions cyfluthrin is partly separated into four peaks. For the calculations take the sum of the four peaks.

(d) Determination. Inject 10 µl portions of the calibration solutions (C₁ and C₂) and the sample solutions (S₁ and S₂) in the following sequence: C₁, S₁, S₂, C₂. Determine the sum of the peak areas of the four incompletely separated isomers. Use the mean of the response factors (*f*) of the calibration solutions bracketing the injections of the sample solutions to calculate the content. If individual measurements vary by more than 0.8 %, prepare new solutions.

(e) Calculation

$$f = \frac{s \times P}{H_s}$$

$$\text{Cyfluthrin content} = \frac{H_w \times f}{w} \text{ g/kg}$$

where:

f = mean response factor

H_s = sum of the areas of the cyfluthrin peaks in the calibration solution

H_w = sum of the areas of the cyfluthrin peaks in the sample solution

s = mass of cyfluthrin in the calibration solution (mg)

w = mass of sample taken (mg)

P = content of the sum of the four isomers in the cyfluthrin reference substance (g/kg)

Repeatability r = 15 g/kg at 954 g/kg active ingredient content

Reproducibility R = 16 g/kg at 954 g/kg active ingredient content

CYFLUTHRIN WETTABLE POWDERS 385/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 HPLC. As for cyfluthrin technical **385/TC/M/2.1**.

2.2 TLC. As for cyfluthrin technical **385/TC/M/2.2**.

2.3 Infrared. Extract the sample (containing about 0.5 g cyfluthrin) with dichloromethane (40 ml). Filter, dry the solution with sodium sulphate and evaporate the solvent. Dissolve the residue in dichloromethane (2 ml) and isolate the active ingredient by chromatography over a silica gel column (glass, with sintered glass frit and ground-in stopcock, 15 × 1.3 (i.d.) cm, R_F = 0.89, DC). Evaporate the solvent in a stream of clean dry air and continue as for cyfluthrin technical **385/TC/(M)/2.3**.

2.4 $^1\text{H-NMR}$. Use the extraction procedure as for 2.3, above, and continue as for cyfluthrin technical **385/TC/M/2.4**.

3. Cyfluthrin

3.1 Normal phase high performance liquid chromatographic method*

As for cyfluthrin technical 385/TC/M/3.1 except substitute:

REAGENTS

Calibration solution. Homogenise the cyfluthrin working standard by warming it in a hot-air cabinet at 60 to 80 °C for about 30 min. After melting, mix the material thoroughly by shaking. Weigh (to the nearest 0.1 mg) in duplicate cyfluthrin working standard (about 100 mg, s mg) into two volumetric flasks (50 ml). The quantities weighed should differ about 10 %. Add TBME (15 ml) and place the flasks in an ultrasonic bath for 5 min. Fill the flasks to 1 cm below the mark with n -heptane and place them in a water bath at 22 °C for 5 min. Make up to volume with n -heptane and mix well. Transfer by pipette 5.0 ml of these solutions to two volumetric flasks (50 ml). Add THF (10 ml), fill the flasks to 1 cm below the mark and place them in a water bath at 22 °C for 5 min. Make up to volume with n -heptane and mix well. (Solutions C_1 and C_2). The solutions are stable for 24 h at room temperature.

and:

PROCEDURE

(a) Operating conditions

Injection volume 10 μ l

(b) *Sample preparation.* Weigh (to the nearest 0.1 mg) in duplicate into two ground-glass stoppered Erlenmeyer flasks (100 ml) enough sample to contain about 100 mg (w mg) cyfluthrin. Add by pipette THF (50.0 ml) and place the flasks in an ultrasonic bath for 10 min. Allow to cool to room temperature. Centrifuge the solutions and transfer by pipette 5.0 ml of the clear solutions into volumetric flasks (50 ml). Add to each flask THF (10 ml) and n -heptane (40 ml), mix and allow to cool to room temperature. Fill to the mark with n -heptane (Solutions S_1 and S_2). If the solutions contain any insoluble material, centrifuge before injection.

Repeatability r = 4 g/kg at 103 g/kg active ingredient content

Reproducibility R = 7 g/kg at 103 g/kg active ingredient content

* CIPAC method 1996. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, Germany.

3.2 Reversed phase high performance liquid chromatographic method*

As for cyfluthrin technical **385/TC/(M)/3.2** except:

(b) *Sample preparation.* (For products containing up to 200 g/kg active ingredient). Weigh (to the nearest 0.1 mg) in duplicate enough sample to contain about 100 mg (*w* mg) cyfluthrin into two ground-glass stoppered Erlenmeyer flasks (150 ml). The quantities weighed should differ about 10 %. Add by pipette acetonitrile (100 ml) and place the flasks in an ultrasonic bath for 10 min. Allow to cool to room temperature (Solutions *S*₁ and *S*₂). Before injecting filter the solutions through disposable filters.

Repeatability *r* = 1 g/kg at 102 g/kg active ingredient content

Reproducibility *R* = 1 g/kg at 102 g/kg active ingredient content

4 Suspensibility. Under consideration.

**CYFLUTHRIN EMULSIFIABLE CONCENTRATES
385/EC/M/-**

1 Sampling. Take at least 500 ml.

2 Identity tests

2.1 HPLC. As for cyfluthrin technical **385/TC/M/2.1**.

2.2 TLC. As for cyfluthrin technical **385/TC/M/2.2**.

2.3 Infrared. Take enough sample to contain about 0.5 g cyfluthrin and evaporate to dryness on a water bath at 40 to 50 °C. Suppress any foaming by adding a small amount of sodium chloride. Treat the residue with dichloromethane (40 ml) and continue as for **385/WP/M/2.3** as from: 'Filter, dry the solution.....'

2.4 ¹H-NMR. Use the extraction procedure as for 2.3, above, and continue as for cyfluthrin technical **385/TC/M/2.4**.

3 Cyfluthrin**3.1 Normal phase high performance liquid chromatographic method**

As for cyfluthrin wettable powders **385/WP/M/3.1** except:

* CIPAC method 1996. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, Germany.

(c) *Sample preparation.* Homogenise the sample. Weigh (to the nearest 0.1 mg) in duplicate into two volumetric flasks (50 ml) enough sample to contain about 10 mg (w mg) cyfluthrin. Dissolve in THF (15 ml), add *n*-heptane (30 ml) and mix. Allow to cool to room temperature and fill to the mark with *n*-heptane (Solutions S_1 and S_2).

(e) *Calculation*

$$\text{Content of the } i\text{th cyfluthrin isomer} = \frac{H_{wi} \times f_i}{w \times 10} \text{ g/kg}$$

$$\text{Total cyflythrin content} = \sum Y_i \text{ g/kg } (i=1-4)$$

Repeatability r = 0.7 g/kg at 15.7 g/kg active ingredient content

Reproducibility R = 1.0 g/kg at 15.7 g/kg active ingredient content

3.2 Reversed phase high performance liquid chromatographic method*

As for cyfluthrin technical 358/TC/M/3.2 except add:

REAGENTS

tert-Butylmethylether (TBME) HPLC quality

Extraction mixture TBME + *n*-heptane, 4 + 96 (v/v)

and substitute the following:

Calibration solution (for liquid formulations with more than 2 g/kg cyfluthrin). Homogenise the cyfluthrin working standard by warming it in a hot-air cabinet at 60 to 80 °C for about 30 min. After melting, mix the material thoroughly by shaking. Weigh (to the nearest 0.1 mg) in duplicate into two volumetric flasks (100 ml) cyfluthrin working standard (about 100 mg, s mg). The quantities weighed should differ about 10 %. Add THF (about 40 ml) and place the flasks in an ultrasonic bath for 5 min. Allow to cool to room temperature, make up to volume with THF and mix well. Transfer by pipette 10.0 ml of these solutions to two volumetric flasks (50 ml) and fill to the mark with THF (Solutions C_1 and C_2).

* CIPAC method 1996. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, Germany

*(a) Operating conditions**Injection volume* 10 µl*(b) Sample preparation*

(i) Liquid formulations with more than 10 g/kg and less than 200 g/kg cyfluthrin. Homogenise the sample. Weigh (to the nearest 0.1 mg) in duplicate into two volumetric flasks (50 ml) sufficient sample to contain about 10 mg (*w* mg) cyfluthrin. Dissolve in THF (30 to 40 ml) and fill to the mark with THF (Solutions *S*₁ and *S*₂).

(ii) Formulations containing petroleum and more than 10 g/kg and less than 200 g/kg cyfluthrin. Homogenise the sample. Weigh (to the nearest 0.1 mg) in duplicate onto two extraction columns enough sample to contain about 10 mg (*w* mg) cyfluthrin. Eluate the active ingredient with extraction solvent and collect the effluent in two volumetric flasks (50 ml). Make up to volume with THF and mix well (Solutions *S*₁ and *S*₂).

(e) Calculation

$$f = \frac{s \times P}{H_s \times 10}$$

$$\text{Cyfluthrin content} = \frac{H_w \times f}{w} \text{ g/kg}$$

Repeatability *r* = 0.7 g/kg at 15.8 g/kg active ingredient content

Reproducibility *R* = 1 g/kg at 15.8 g/kg active ingredient content

CYFLUTHRIN EMULSION OF OIL IN WATER* **385/EW/M/-**

1 Sampling. Take at least 500 ml.

2 Identity tests

2.1 HPLC. As for cyfluthrin technical **385/TC/M/2.1**.

2.2 TLC. As for cyfluthrin technical **385/TC/M/2.2**.

2.3 Infrared. Take enough sample to contain about 0.5 g cyfluthrin and evaporate to dryness at a water bath at 40 to 50 °C. Suppress any foaming by adding a small amount of sodium chloride. Treat the residue with dichloromethane (40 ml) and continue as for **385/WP/M/2.3** as from: 'Filter, dry the solution.....'

2.4 ¹H-NMR. Use the extraction procedure as for 2.3, above, and continue as for cyfluthrin technical **385/TC/M/2.4**.

* CIPAC method 1996. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, Germany

3 Cyfluthrin

3.1 Normal phase high performance liquid chromatographic method*

As for cyfluthrin emulsifiable concentrates **358/EC/M/3.1**.

Repeatability r = 3 g/kg at 50.1 g/kg active ingredient content

Reproducibility R = 7 g/kg at 50.1 g/kg active ingredient content

3.2 Reversed phase high performance liquid chromatographic method*

As for cyfluthrin emulsifiable concentrates **358/EC/M/3.2**.

Repeatability r = 3 g/kg at 50 g/kg active ingredient content

Reproducibility R = 6 g/kg at 50 g/kg active ingredient content

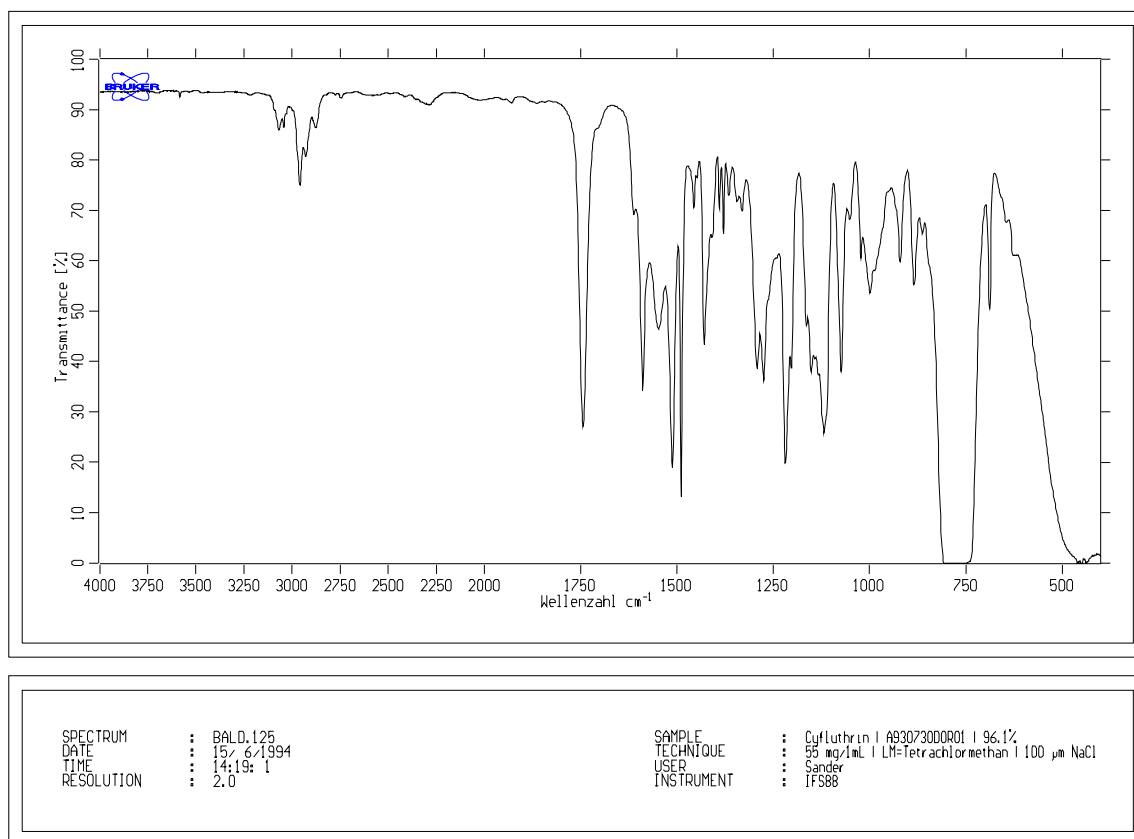


Fig. 10 Infrared spectrum of cyfluthrin

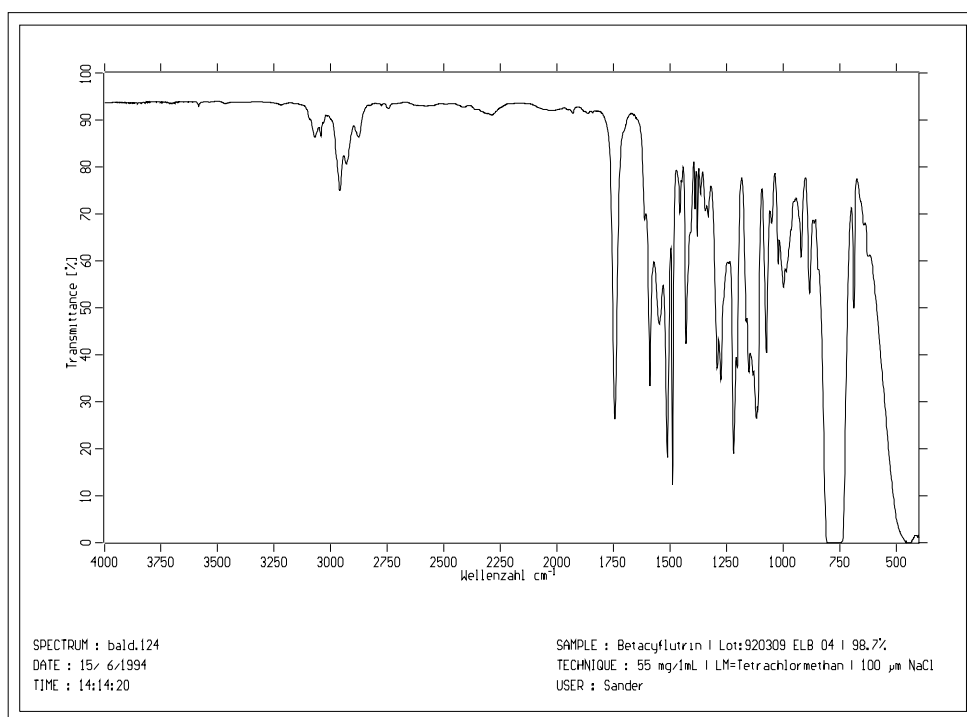


Fig. 11 Infrared spectrum of beta-cyfluthrin

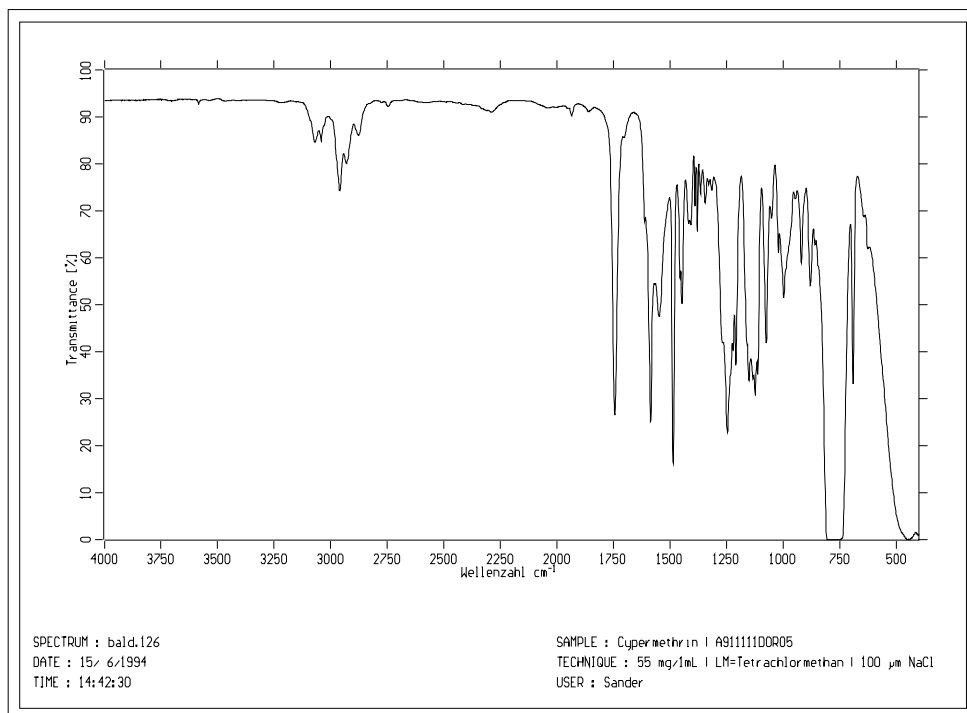


Fig. 12 Infrared spectrum of cypermethrin

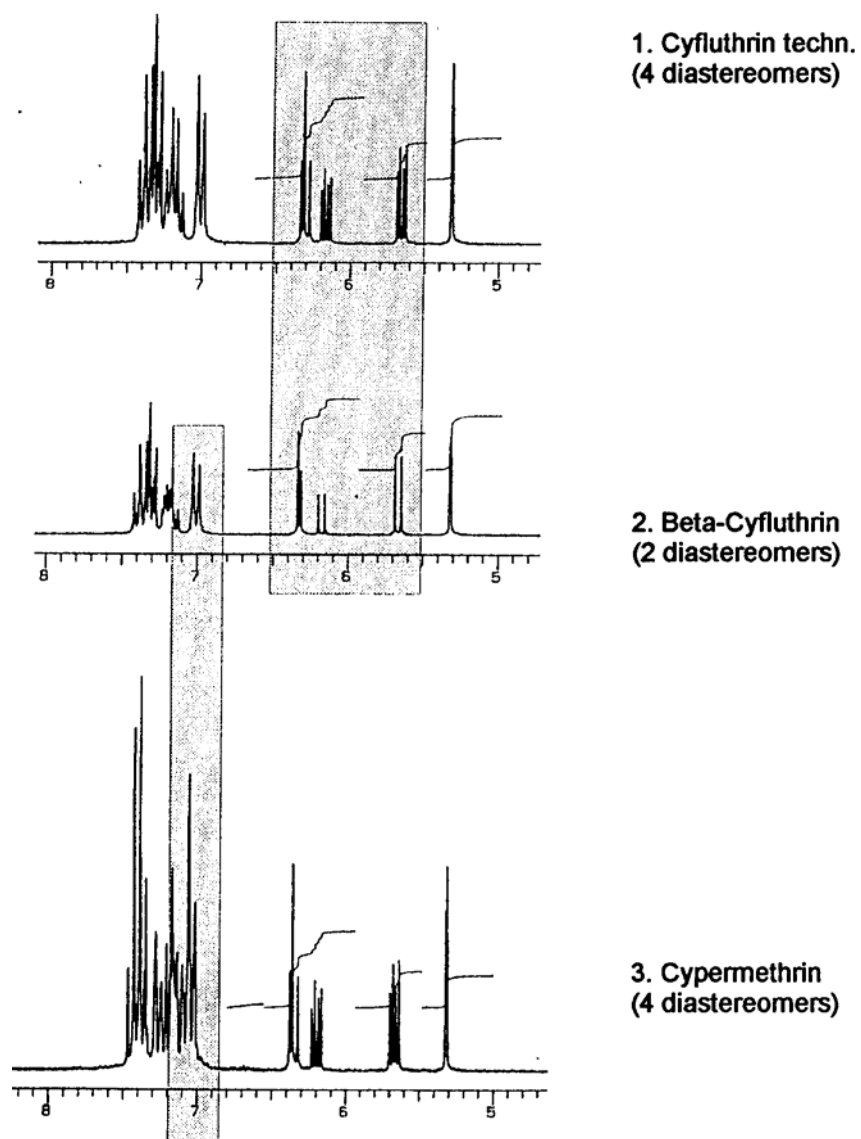


Fig. 13 Identification of Cyfluthrin techn., Beta-Cyfluthrin and Cypermethrin by ^1H -NMR spectroscopy

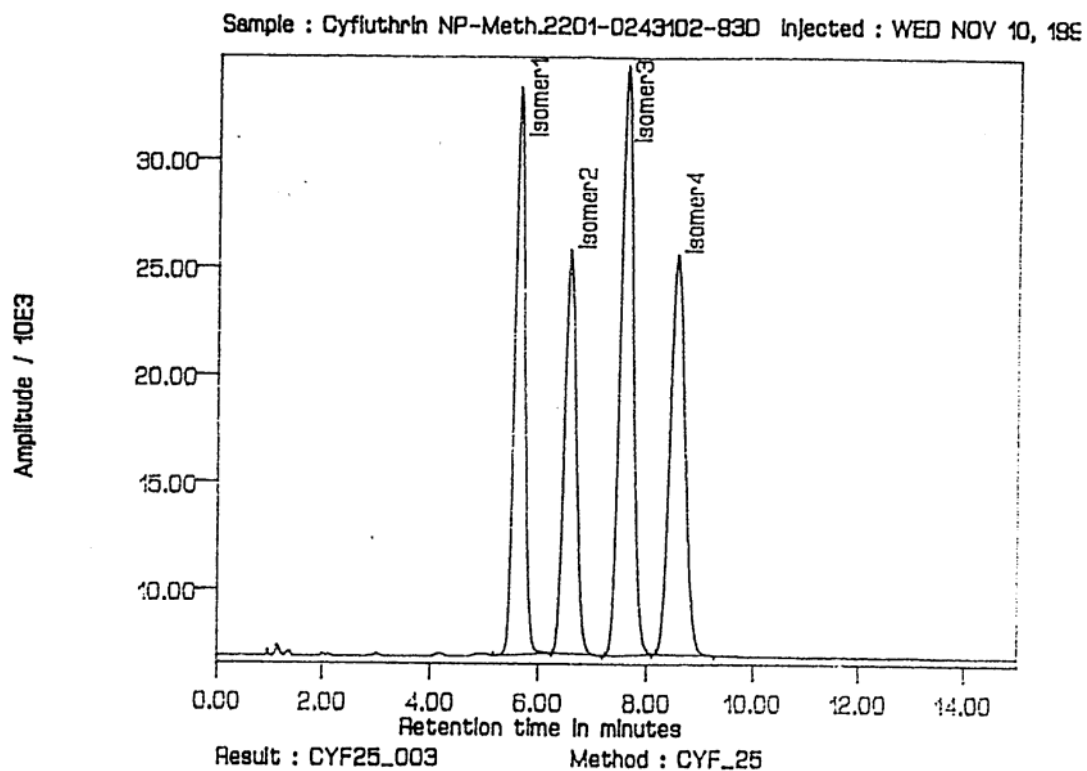


Fig. 14 Normal phase chromatogram of beta-cyfluthrin

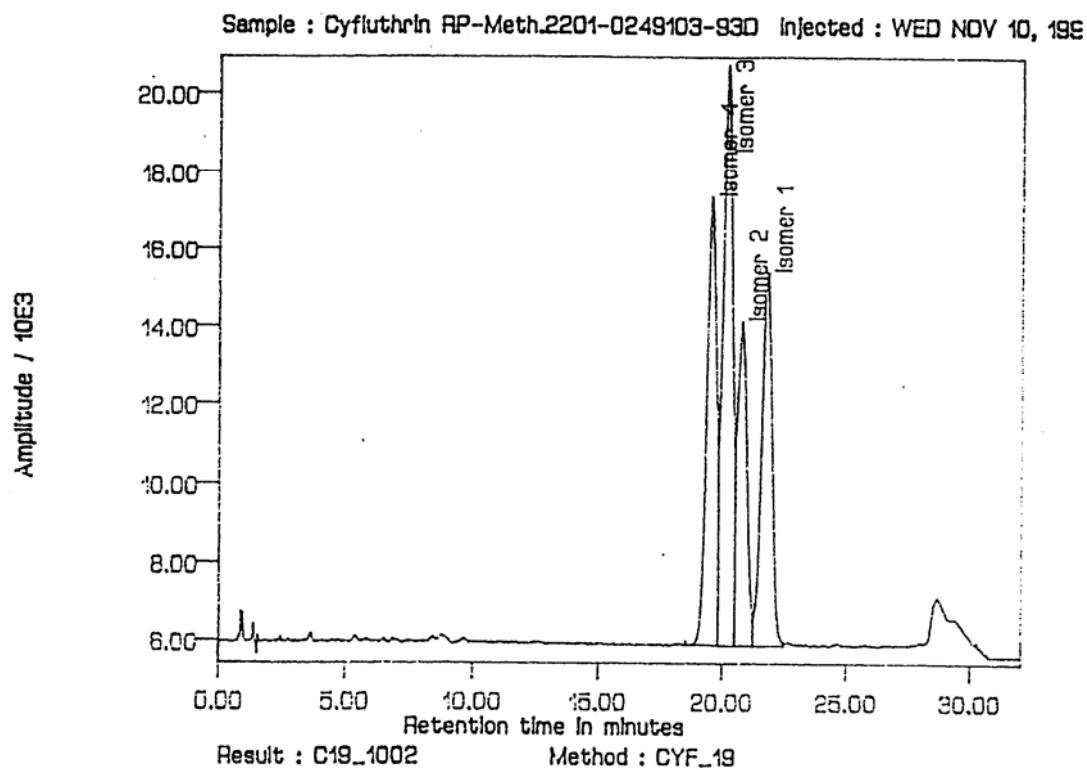


Fig. 15 Reversed phase chromatogram of cyfluthrin