FENVALERATE
334

See fenvalerate, CIPAC D, *p.100*

FENVALERATE EMULSIFIABLE CONCENTRATE
*334/EC/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests. MT 163, CIPAC D, *p.180.*

3 Fenvalerate

SCOPE The method is unsuitable for the determination of the diastereoisomer ratio.

OUTLINE OF METHOD Fenvalerate is separated from emulsifiers by column chromatography. The content of fenvalerate is determined by gas chromatography with flame-ionisation detection (FID) and with diphenyl phthalate as internal standard.

REAGENTS

4-Methylpentan-2-one (methyl isobutyl ketone) MIBK
Diphenyl phthalate (DPP) internal standard
Formic acid purity at least 98%
Dichloromethane
Internal standard solution. Dissolve DPP (1 g) and formic acid (0.5 g) in MIBK (500 ml).
Fenvalerate standard of known purity
Calibration solution. Homogenize the fenvalerate standard by warming the sealed bottle at between 60°C and 65°C until no crystals remain and then mix well.

* CIPAC method 1994. Prepared by the Japanese Committee (JAPAC). Chairman: T Suzuki. Based on a method supplied by Sumitomo Chemical Co, Ltd, Japan.

Weigh in duplicate (to the nearest 0.1 mg) approximately 110 mg of the fenvalerate standard (*s* mg) into volumetric flasks (50 ml), dissolve and dilute to volume with the dichloromethane. Transfer by pipette, 10.0 ml of each solution to a conical flask (30 ml) and add by pipette to each flask 10.0 ml of the internal standard solution (Solutions C_A and C_B).

Silica gel for column chromatography 60 to 80 mesh

APPARATUS

Pasteur pipette glass tube 106 × 5 (i.d.) mm with a 40 mm length capillary outlet

Gas chromatograph equipped with a FID and an on-column injection port

Column glass, 110 × 3 (i.d.) mm, packed with 2% Apiezon L on Chromosorb W-HP, 100 to 120 mesh. Before use, condition a freshly prepared column by purging it with nitrogen for 24 hours at 270 °C. During this operation the column must not be connected to the detector.

Electronic integrator compatible with the gas chromatograph

Microsyringe 10 µl

PROCEDURE

(a) Preparation of the sample solutions. Insert into two Pasteur pipettes small wads of cotton wool and pack the pipettes with a 25 mm layer of silica gel. Weigh in duplicate (to the nearest 0.1 mg) into volumetric flasks (50 ml) sufficient sample (*w* mg) to contain approximately 500 mg of fenvalerate (Solutions S_A and S_B). Dilute to volume with dichloromethane and mix well. Transfer by pipette 2.0 ml of each solution to a separate column. Elute the fenvalerate with 5 portions of about 2 ml of dichloromethane into conical flasks (30 ml). Add by pipette 10.0 ml of the internal standard solution to each of the conical flasks and mix well.

(b) Gas chromatographic conditions (typical):

Column temperature	250 °C
Detector temperature	270 °C
Injection port temperature	270 °C
Carrier gas flow rate, nitrogen	about 50 ml/min
Injection volume	2 µl
Retention times	DPP: 3 min fenvalerate: 10 and 12 min (diastereo-isomers)

(c) Equilibration of the system. Inject 2 µl portions of calibration solution C_A until the peak area ratios of the sum of the two fenvalerate peaks to the DPP peak differ by less than 1.0%.

(d) Analysis of sample. Carry out injections of 2 µl portions of calibration solutions (C_A and C_B) and sample solutions (S_A and S_B) in the following sequence:
C_{A1}, C_{A2}, S_{A1}, S_{A2}, C_{B1}, C_{B2}, S_{B1}, S_{B2}, C_{A1}, C_{A2}, S_{A1}, S_{A2}, C_{B1}, C_{B2}, S_{B1}, S_{B2}
and record the integrated areas of the three peaks. Sum the fenvalerate peak areas in each chromatogram.

(e) Calculation. Calculate the mean value of each pair of calibration factors (*f*) bracketing the injections of the sample injections.

$$f = \frac{s \times P}{R'}$$

where:
- s = mass of fenvalerate working standard in the calibration solution (mg)
- P = purity of the fenvalerate standard (g/kg)
- R' = mean of the ratio of the sum of the areas of the fenvalerate peaks to the DPP peak area for the calibration solution injections

$$\text{Content of fenvalerate} = \frac{f \times R}{w} \text{ g/kg}$$

where:
- f = mean response factor
- R = mean of the ratio of the sum of the areas of the fenvalerate peaks to the DPP peak area for the sample solution injection
- w = mass of sample taken (mg)

Repeatability r = 2.8 g/kg at 100 g/kg active ingredient content
Repeatability r = 2.9 to 3.6 g/kg at 200 g/kg active ingredient content
Reproducibility R = 3.9 g/kg at 100 g/kg active ingredient content
Reproducibility R = 11.9 to 12.5 at 200 g/kg active ingredient content