PIPERONYL BUTOXIDE

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Method Extension of 33/EW/M/-

Determination of Piperonyl butoxide in
Metofluthrin/*d,d-trans*-Cyphenothrin/Piperonyl butoxide EW

by
Makiko Mukumoto
Sumitomo Chemical Co., Ltd.
Environmental Health Science Laboratory
1-98, Kasugade-naka 3-chome, Konohanaku, Osaka
JAPAN

1 Sampling. Take at least 1 l.

2 Identity tests

2.1 GC. Use the GLC method below. The relative retention time obtained from the sample should not deviate by more than 1% from that of the standard under the same conditions.

2.2 GC-MS. (formulations with two or more active substances) Use a GC apparatus connected to a mass spectrometer with an electron impact ion source and separate the components by the GLC method below. Record the mass spectrum of the peak found at the retention time assigned to piperonyl butoxide. The mass spectrum should match that found from the standard.

3 Piperonyl butoxide

SCOPE The method is suitable for the determination of piperonyl butoxide formulations containing piperonyl butoxide as the only active ingredient and in mixtures with metofluthrin and *d,d-trans*-cyphenothrin.

OUTLINE OF METHOD

Piperonyl butoxide is determined by capillary gas chromatography using flame ionisation detection and triphenyl phosphate as internal standard.

REAGENTS

Piperonyl butoxide standard of known purity. Store below 0°C.

Triphenyl phosphate internal standard.

Propan-2-ol

Internal standard solution. Weigh into a volumetric flask (100 ml) triphenyl phosphate (1.8 g). Fill to the mark with propan-2-ol and mix well.

Calibration solutions. Allow piperonyl butoxide to equilibrate to ambient temperature. Then weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) 0. 11 g piperonyl butoxide (*s* mg). Add by pipette internal standard solution (5.0 ml) and make up to volume with propan-2-ol. Mix well (Solution C). Keep tightly closed and store in the dark under refrigeration.

APPARATUS

Gas chromatograph capable of operating over the range 180 to 250°C fitted with a flame ionisation detector, a split injector, and an autosampler

Capillary column fused silica, 30 m x 0.32 mm (i.d.) with 100% methyl polysiloxane, cross-linked, surface bonded stationary phase and 0.25 μm film thickness (Durabond-1 or equivalent)
Electronic integrator or data system

PROCEDURE
(a) Operating conditions (typical):

Column	Fused silica, 30 m x 0.32 mm (i.d.) with 100% methyl polysiloxane, cross-linked, surface bonded stationary phase and 0.25 μm film thickness (Durabond-1 or equivalent)
Injection system	
Injector	Split injection
Injector temperature	250°C
Sprit ratio	20:1
Purge flow	1 ml/min
Injection volume	1 μl
Detector system	
Type	Flame ionisation
Temperature	300°C
Oven temperatures	
Initial	180°C
Program	180°C hold for 11 min → 200°C at 10°C/min, hold for 8 min → 210°C at 10°C/min, hold for 18 min → 245°C at 30°C/min, hold for 4 min
Total run time	45 min
Gas flow rates	
Helium (carrier)	linear velocity: 39 cm/min at 180°C
Helium (make up)	30 ml/min
Hydrogen	40 ml/min
Air	400 ml/min
Total flow	35 ml/min
Retention times	triphenyl phosphate: about 22 min piperonyl butoxide: about 23 min

(b) Preparation of sample. Thoroughly shake the sample container to homogenise the sample before use. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) sufficient sample (*w* mg) to contain 0.11 g piperonyl butoxide. Add by pipette internal standard solution (5.0 ml) and make up to volume with propan-2-ol. Mix wee (Solutions S).

(c) System equilibration. Inject into the gas chromatograph a 1 µl portion of the sample solution to condition the column and to check for the appropriate flow rates and integration events.

(d) Determination. Inject in duplicate into the gas chromatograph 1 µl portions of the calibration and sample solutions in the following sequence:

$$C_1, C_2, S_1, S_2, C_3 \cdots \text{etc.}$$

Determine piperonyl butoxide to internal standard peak area ratio (R and R' for the sample and calibration solutions respectively). Average the ratios (R') of the calibration solution injections bracketing the sample solution injections and the ratios (R) of the bracketed sample solution injections.

(e) Calculation.

$$\text{Piperonyl butoxide content} = \frac{R \times s \times P}{R' \times w} \text{ g/kg}$$

Where:

R = piperonyl butoxide to internal standard peak area ratio of the sample solution

R' = piperonyl butoxide to internal standard peak area ratio of the calibration solution

s = mass of piperonyl butoxide in the sample solution (mg)

w = mass of sample taken (mg)

P = purity of piperonyl butoxide standard (g/kg)

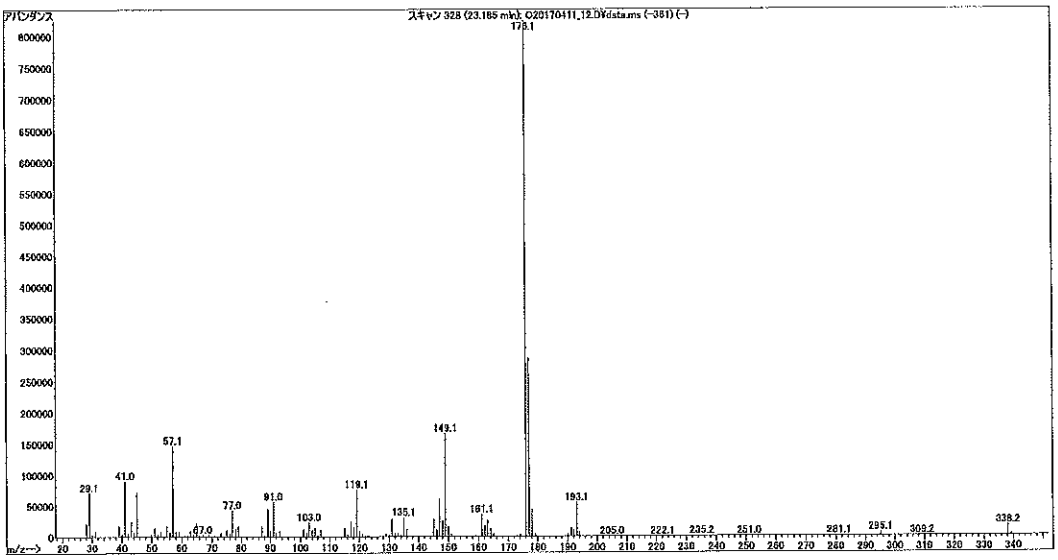

Fig 1 GC-MS Spectrum of piperonyl butoxide standard

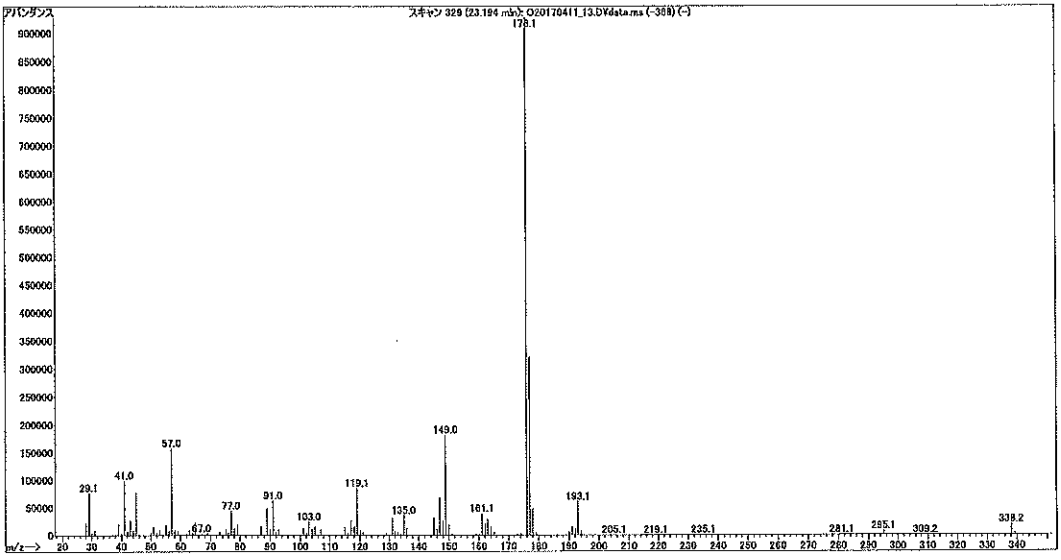

Fig 2 GC-MS Spectrum of piperonyl butoxide in metofluthrin/*d,d-trans*-cyphenothrin/piperonyl butoxide EW

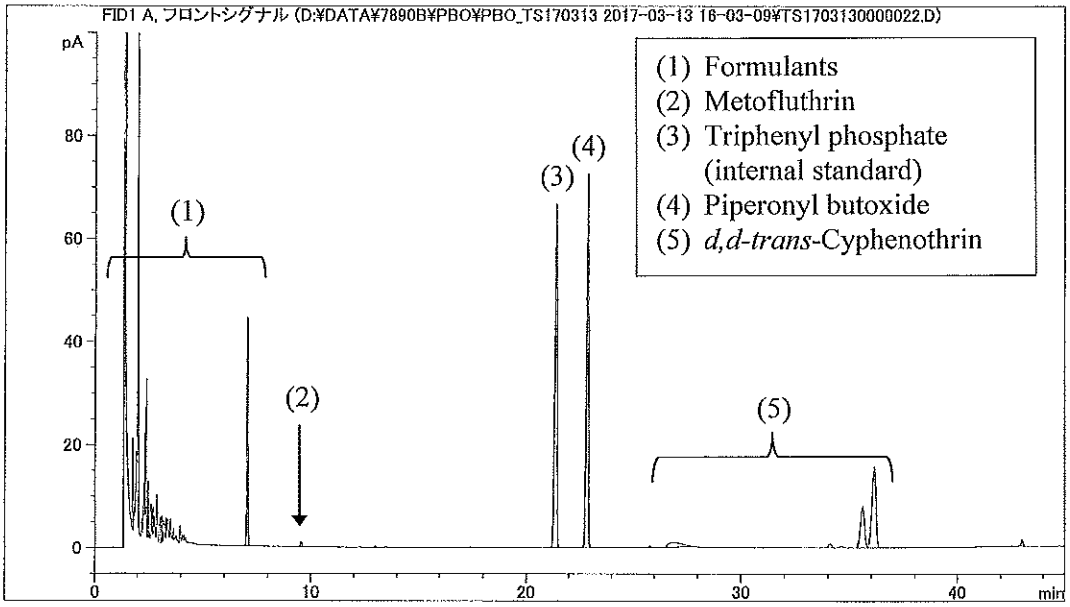

Fig 3 Gas chromatogram of
metofluthrin/*d,d-trans*-cyphenothrin/piperonyl butoxide EW