IMIDACLOPRID
582

See CIPAC H, *p* 185.

IMIDACLOPRID TECHNICAL
*582/TC/M2/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The relative retention time of the imidacloprid peak in the sample solution should not deviate by more than 2% from that of the calibration solution. The UV spectrum measured from this peak should match that obtained from the calibration substance.

2.2 Infrared. As for imidacloprid technical **582**/TC/(M)/2.2, CIPAC H, *p* 186.

2.3 NMR. As for imidacloprid technical **582**/TC/(M)/2.3, CIPAC H, *p* 186.

3 Imidacloprid

OUTLINE OF METHOD. Imidacloprid is determined by reversed phase high performance liquid chromatography using UV detection at 260 nm and external standardisation.

REAGENTS

Imidacloprid reference standard with known content
Water HPLC grade
Acetonitrile HPLC grade
Buffer pH 3 0.1 M sodium citrate - hydrochloric acid (*c*(HCl) = 0.1 mol/l), 40 + 60 (v/v); e. g. Titrisol Merck art no. 109883
Eluent water - buffer solution - acetonitrile 72 + 8 + 20 (v/v/v)
Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) about 75 mg of the imidacloprid standard (*s* mg) into separate volumetric flasks (100 ml). Add acetonitrile (about 30 ml) and place the flasks in an ultrasonic bath for 15 min. Add water to just below the mark and mix. Allow to cool to ambient temperature and fill to the mark with water (Solutions C_1 and C_2).

* CIPAC method 2001. Prepared by the German PAC (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, FRG.

APPARATUS

High performance liquid chromatograph equipped with an ultraviolet spectrophotometric detector and an injection system capable to inject 5 µl

Column stainless steel, 125 × 4 mm (i.d), packed with Lichrospher RP 18, 5 µm, or equivalent material with the same selectivity

Electronic integrator

Ultrasonic bath

Centrifuge or *dispensible filters*, solvent compatible, porosity 0.45 µm (e.g. Gelman GHP ACRODISC or equivalent)

PROCEDURE

(a) Chromatographic conditions (typical)

Column temperature	40 °C
Flow rate	2.0 ml/min
Measuring wavelength	260 nm
Injection volume	5 µl
Run time	approximately 10 min
Retention time	approximately 2.5 min

(b) Equilibration of the system. Pump sufficient eluent through the column to equilibrate the system. Inject 5 µl portions of the calibration solution C_1 and repeat the injections until retention times and peak areas vary by less than ± 0.5 % of the mean for three successive injections.

(c) Sample preparation. Weigh (to the nearest 0.1 mg) sufficient sample to contain about 75 mg imidacloprid (*w* mg) into a volumetric flask (100 ml). Add acetonitrile (about 30 ml) and place the flask in an ultrasonic bath for 15 min. Add water to just below the mark and mix. Allow to cool to ambient temperature and fill to the mark with water (Solution S).

(d) Determination. Inject 5 µl portions of the calibration solutions (C_1 and C_2) and of the sample solutions (S_1, S_2, …,etc.) in the following sequence:

C_1, S_1, C_2, S_2, ...

Determine the peak area of imidacloprid and calculate the response factors (*f*) from the calibration solutions bracketing the injections of the sample solutions. Average the response factors of the calibration solutions preceding and following the sample solution injections. These must agree within ± 0.5 % of the average otherwise repeat the determination. Calculate the content of the sample solutions.

(e) Calculation

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

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

where:
f = mean response factor
H_s = peak area of imidacloprid in the calibration solution
H_w = peak area of imidacloprid in the sample solution
s = mass of imidacloprid in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of imidacloprid standard (g/kg)

Repeatability r = 29 g/kg at 985 g/kg active ingredient content
Reproducibility R = 18 g/kg at 985 g/kg active ingredient content

Based on a study with 22 participants and 22 results

IMIDACLOPRID WATER DISPERSIBLE GRANULES
[*]**582**/WG/M/-

1 Sampling. Take at least 1 kg.

2 Identity tests
2.1 HPLC. As for imidacloprid technical **582**/TC/M2/2.1.
2.2 Infrared. As for imidacloprid wettable powders **582**/WP/(M)/2.2, CIPAC H, *p* 189.
2.3 NMR. As for imidacloprid technical **582**/TC/(M)/2.3, CIPAC H, *p* 186.

3 Imidacloprid. As for imidacloprid technical 582/TC/M2/3, except:
(c) Sample preparation. Weigh (to the nearest 0.1 mg) sufficient sample to contain about 75 mg imidacloprid (*w* mg) into a volumetric flask (100 ml). Add acetonitrile (about 30 ml) and place the flask in an ultrasonic bath for 15 min. Agitate the sediment several times by shaking. Then add water to just below the mark and mix. Allow to cool to ambient temperature and fill to the mark with

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water. Clear the extract by centrifugation or filtration (Solution S).

Repeatability r =.23 g/kg at 703 g/kg active ingredient content
Reproducibility R = 12 g/kg at 703 g/kg active ingredient content

Based on a study with 22 participants and 22 results

4. Suspensibility. (Draft method)

REAGENTS AND APPARATUS As for **582**/TC/M2/3 and MT 168.

PROCEDURE

(a) Preparation of suspension and determination of sedimentation. MT 161.

(b) Determination of imidacloprid in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension transfer the remaining 25 ml of the suspension to a 100 ml volumetric flask. Wash twice with 25 ml tetrahydrofuran and fill up to the mark with a mixture of water/acetonitrile 50+50 (v/v). Determine the mass of imidacloprid (*Q* g) by **582**/TC/M2/3, except:
Prepare the calibration solution with tetrahydrofuran-acetonitrile-water 50+25+25 and dilute the calibration solution with this mixture as appropriate for the imidaclor content of the sample taken for the suspensibility test.

(d) Calculation

$$\text{Suspensibility} = \frac{111(c - Q)}{c} \%$$

where:
c = mass of imidacloprid in sample taken for the preparation of the suspension (g)
Q = mass of imidacloprid in the bottom 25 ml of suspension

IMIDACLOPRID SUSPENSION CONCENTRATES
*582/SC/M2/-

1 Sampling Take at least 1 kg.

2 Identity tests
2.1 HPLC. As for imidacloprid technical **582**/TC/M2/-.
2.2 Infrared. Dry approximately 2 g sample on a clay plate, wash shortly with water and dry again. Then proceed as for imidacloprid suspension concentrates **582**/SC/(M)/2.2, CIPAC H, *p* 189.
2.3 NMR. Dry approximately 2 g sample on a clay plate, wash shortly with water and dry again. Then proceed as for imidacloprid suspension concentrates **582**/SC/(M)/2.3, CIPAC H, *p* 189.

3 Imidacloprid. As for imidacloprid technical **582**/TC/M2/3, except:
(c) Sample preparation. Homogenise the sample by vigorous shaking. Weigh (to the nearest 0.1 mg) sufficient sample to contain about 75 mg imidacloprid (*w* mg) into a volumetric flask (100 ml). Add water (5 ml) and suspend the sample. Add acetonitrile (about 30 ml) and place the flask in an ultrasonic bath for 15 min. Agitate the sediment several times by shaking. Then add water to just below the mark and mix. Allow to cool to ambient temperature and fill to the mark with water. Clear the extract by centrifugation or filtration (Solution S).

Repeatability r = 15 g/kg at 309 g/kg active ingredient content
Reproducibility R = 5.7 g/kg at 309 g/kg active ingredient content

Based on a study with 22 participants and 22 results

4. Suspensibility. (Draft method)

REAGENTS AND APPARATUS As for **582**/TC/M2/3 and MT 168.

PROCEDURE As for **582**/WG/M/4.

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IMIDACLOPRID WATER DISPERSIBLE POWDERS FOR SLURRY SEED TREATMENT

[*]**582**/WS/M/-

1 Sampling. Take at least 500 g

2 Identity tests
2.1 HPLC. As for imidacloprid technical **582**/TC/M2/2.1.
2.2 Infrared. As for imidacloprid wettable powders **582**/WP/(M)/2.2, CIPAC H, *p* 189.
2.3 NMR. As for imidacloprid technical **582**/TC/(M)/2.3, CIPAC H, *p* 186.

3 Imidacloprid. As for imidacloprid water dispersible granules **582**/WG/M/3.

Repeatability r = 38 g/kg at 701 g/kg active ingredient content
Reproducibility R = 40 g/kg at 701 g/kg active ingredient content

Based on a study with 22 participants and 22 results

IMIDACLOPRID FLOWABLE CONCENTRATES FOR SEED TREATMENT

[*]**582**/FS/(M)/-

1 Sampling Take at least 1 kg.

2 Identity tests
2.1 HPLC. As for imidacloprid technical **582**/TC/M2/-.
2.2 Infrared. As for imidacloprid suspension concentrates **582**/SC/M2/2.2.
2.3 NMR. As for imidacloprid suspension concentrates **582**/SC/M2/2.3

3 Imidacloprid. As for imidacloprid suspension concentrates **582**/SC/M2/2/3.

Repeatability r = 12 g/kg at 448 g/kg active ingredient content
Reproducibility R = 40 g/kg at 448 g/kg active ingredient content

Based on a study with 22 participants and 22 results

[*] CIPAC method 2001. Prepared by the German PAC (DAPA). Chairman: W Dobrat. Based on a method supplied by Bayer AG, FRG.

4. Suspensibility. (Draft method)
REAGENTS AND APPARATUS As for **582**/TC/M2/3 and MT 168.
PROCEDURE As for **582**/WG/M/4.

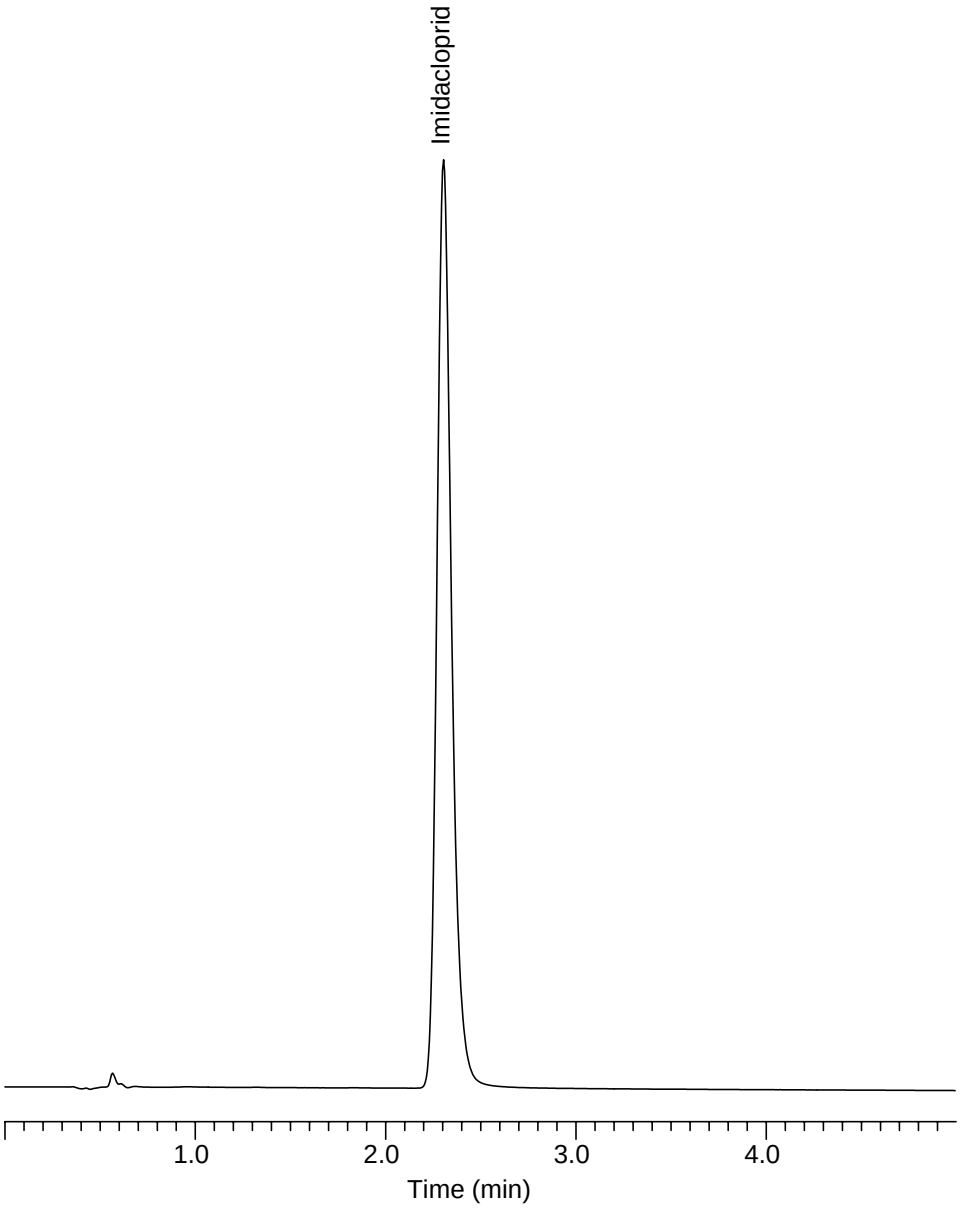

Fig. 24 Chromatogram of imidacloprid technical