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Dimoxystrobin
739

CIPAC collaborative trial, GC Method CIPAC 4710/m

by

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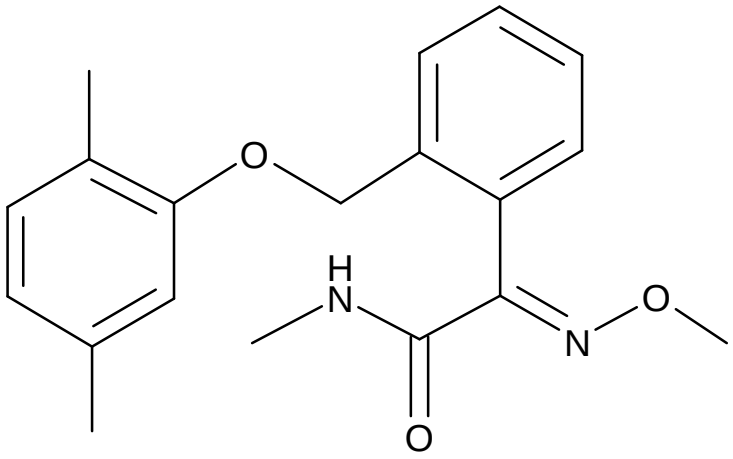


ISO common name	Dimoxystrobin
Chemical name	(E)-o-(2,5-Dimethylphenoxymethyl)-2-methoxyimino-N-methylphenylacetamide
CAS.No.	149961-52-4
Empirical formula	$C_{19}H_{22}N_2O_3$
RMM	326.39
m.p.	138 °C
Solubilty	In water: 4.3 mg/l at 20 °C
Description	Form: off-white odourless powder
Formulations	Suspension Concentrates Suspo-Emulsion

DIMOXYSTROBIN TECHNICAL
*__739__/TC/(M)/-

1 Sampling. Take at least 50 g.

2 Identity tests

2.1 GLC. Use the GLC method below. The relative retention time of the Dimoxystrobin peak in the sample solution should not deviate by more than 0.1 s from that of the calibration solution.

2.2 Infrared. Prepare potassium bromide discs from the sample and from pure Dimoxystrobin using 1 to 1.5 mg material and 300 mg potassium bromide. Scan the discs from 4000 to 400 cm^{-1}. The spectrum obtained from the sample should notdiffer significantly from that of the standard.

3 Dimoxystrobin

OUTLINE OF METHOD. Dimoxystrobin is determined by using a high resolution gas chromatographic procedure that employs an internal standard. The separation is achieved by using a capillary column, temperature programmed. The analyte is detected using an flame ionization detector (FID) and quantified by comparing the specific response ratio of the samples with that of standards of known qualities.

REAGENTS

Dimoxystrobin reference standard of known purity
Water deionized HPLC grade
Acetone GC grade
Tetrahydrofuran GC grade
Tetraphenylethylene purity at least 97 %
Internal standard solution. Weigh into a volumetric flask (100 ml) tetraphenylethylene (1.0 g) and dissolve in tetrahydrofuran.

Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) about 25 mg of the Dimoxystrobin standard (*s* mg) into separate volumetric flasks (25 ml). Add 1ml of the internal standard solution. Then the flask is filled up to the mark with Acetone and placed in an ultrasonic bath for 5 minutes. Two calibration solutions are provided.

APPARATUS

Gas chromatograph equipped with an FID detector, split/splitless injection and an automatic sampler capable to inject 0.2 µl
Column fused silica, 30 m x 0.32 mm (i.d) x 0.25 µm film thickness (DB-1, or equivalent dimethyl polysiloxane phase)
Electronic integrator or data system
Ultrasonic bath

PROCEDURE
(a) Chromatographic conditions (typical)

Column	Fused silica, 30m x 0.32mm x 0.25µm (DB-1, or equivalent dimethyl polysiloxane phase)
Detector	Flame ionisation
Injector	Split/splitless with fused silica liner containing a 1 cm plug of glass wool.
Temperatures	
Column oven	60 °C; hold for 0.5 minutes; to 260 °C at 20 °C/minutes; final hold 9.5 minutes
Injector	280 °C
Detector	280 °C
Flow rates	
Helium (carrier gas)	2 ml/minutes (constant flow)
Helium (make up)	30 ml/minutes or optimum for instrument
Split ratio	approximately 20:1
Injectionvolume	0.2 µl
Run time	approximately 20 minutes
Retention times	Dimoxystrobin approximately 11.1 minutes Tetraphenylethylene approximately 11.4 minutes

Remarks

Flow rates	Necessary to use constant flow
Analytical columns	A list of analytical columns used by the participants in the CIPAC collaborative trial is given in PROCEDURE (f)
Carrier gas	Helium should be used, avoid to use Hydrogen
Internal Standard	alternatively: Weigh into a volumetric flask (100ml) di-n-butyl phthalate (1.0 g) and dissolve in Aceton. Retention time: approximately 9.3 minutes

(b) Equilibration of the system. Inject 0.2 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) Preparation of sample. Weigh (to the nearest 0.1 mg) sufficient sample to contain about 25 mg Dimoxystrobin into a volumetric flask (25 ml). Add 1ml of the internal standard solution. Then the flask is filled up to the mark with Acetone and placed in an ultrasonic bath for 5 minutes (Solutions S_A and S_B)

(d) Determination. Inject each sample solution in duplicate and bracket a series of sample solution injections by injections of the calibration solutions as follows: calibration solution 1, sample solution S_A (double injection), calibration solution 2, sample solution S_B (double injection), calibration solution 1. Measure the relevant peak areas and calculate the response factor (f_i).
Calculate the mean of each pair of response factors bracketing the injections of the two samples and use this value for calculating the Dimoxystrobin contents of the bracketed sample runs. The response factors and retention times for the successive injections should agree within 1 %.

(e) Calculation

$$f_i = \frac{I_r \times s}{H_s}$$

$$\text{Dimoxystrobin content} = \frac{H_w \times f \times P}{I_q \times w} \text{ g/kg}$$

where:

f_i = single response factor
f = average response factor
H_s = area of the Dimoxystrobin peak in the calibration solution
H_w = area of the Dimoxystrobin peak in the sample solution
I_r = area of the internal standard peak in the calibration solution
I_q = area of the internal standard peak in the sample solution
s = mass of Dimoxystrobin in the calibration solution (mg)
w = mass of sample (mg)
P = purity of Dimoxystrobin standard (g/kg)

(f) List of analytical columns. The following analytical columns were used by the participants in the CIPAC collaborative trial:

DB-1, 30 m x 0.32 mm, 0.25µm
HP-1, 25 m x 0.20 mm, 0.33µm
BP-1, 30 m x 0.22 mm, 1µm
VF-1ms, 15 m x 0.25 mm, 0.25µm
DB-1, 30 m x 0.53 mm, 1.5µm
DB-1, 30 m x 0.25 mm, 0.25µm
TR-1, 30 m x 0.25 mm, 0.25µm
Rtx-CLP, 30 m x 0.25 mm, 0.25µm
CP-Sil 13CB, 25 m x 0.32 mm, 0.20µm
HP-5, 30 m x 0.32 mm, 0.25µm
CP-Sil 5CB, 30 m x 0.32 mm, 0.25µm
DB-5, 30 m x 0.32 mm, 0.50µm
HP-1, 25 m x 0.32 mm, 0.25µm
Rtx-1ms, 30 m x 0.25 mm, 0.25µm
Rtx-5, 30 m x 0.25 mm, 0.25µm

Remarks
Due to differences in polarity of the stationary phases of the analytical columns listed above, the retention time of the active substance detected in the chromatographic run may shift. In any case detection and quantification of Dimoxystrobin content in the assay has been done. There was no indication of interferences due to other components.

DIMOXYSTROBIN SUSPO-EMULSIONS
*739/SE/(M)/-

1 Sampling. Take at least 50 ml.

2 Identity test
2.1 GLC. As for Dimoxystrobin technical 739/TC/(M)/2.1.
2.2 Infrared. As for Dimoxystrobin technical 739/TC/(M)/2.2.

3 Dimoxystrobin. As for Dimoxystrobin technical 739/TC/(M)/3 except:

PROCEDURE
change *(c) Preparation of sample solution* to:
Weigh (to the nearest 0.1 mg) sufficient sample to contain about 25 mg Dimoxystrobin into a volumetric flask (25 ml). Add about 1 ml water and shake slightly to advance the solubility. Then 20 ml acetone and 1 ml internal standard solution are added (it is necessary to use this order). Fill up to the mark with acetone and place the flask in an ultrasonic bath for 5 min. Particles which are not dissolved are filtered off by run through a 0.20 µm filter (CHROMAFIL Xtra RC-20/25).

DIMOXYSTROBIN SUSPENSION CONCENTRATES
*739/SC/(M)/-

1 Sampling. Take at least 50 ml.

2 Identity test
2.1 GLC. As for Dimoxystrobin technical 739/TC/(M)/2.1.
2.2 Infrared. As for Dimoxystrobin technical 739/TC/(M)/2.2.

3 Dimoxystrobin. As for Dimoxystrobin suspo-emulsions 739/SE/(M)/3.

4 Suspensibility.

REAGENTS AND APPARATUS. As for 739/TC/(M)/3 and MT 184.

PROCEDURE
(a) Preparation of suspension MT 184.
(b) Determination of sedimentation MT 184.
(c) Determination of Dimoxystrobin in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension add acetonitrile (75 ml) to the 25 ml remaining in the cylinder and mix thoroughly. Place the cylinder in an ultrasonic bath for 5 min. Allow to cool to room temperature and take a suitable aliquot of the solution. Determine the mass of Dimoxystrobin (Q g) by 739/TC/(M)/3.
(d) Calculation

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

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

Typical Chromatograms of Dimoxystrobin

Figure 1 Analytical Standard Dimoxystrobin

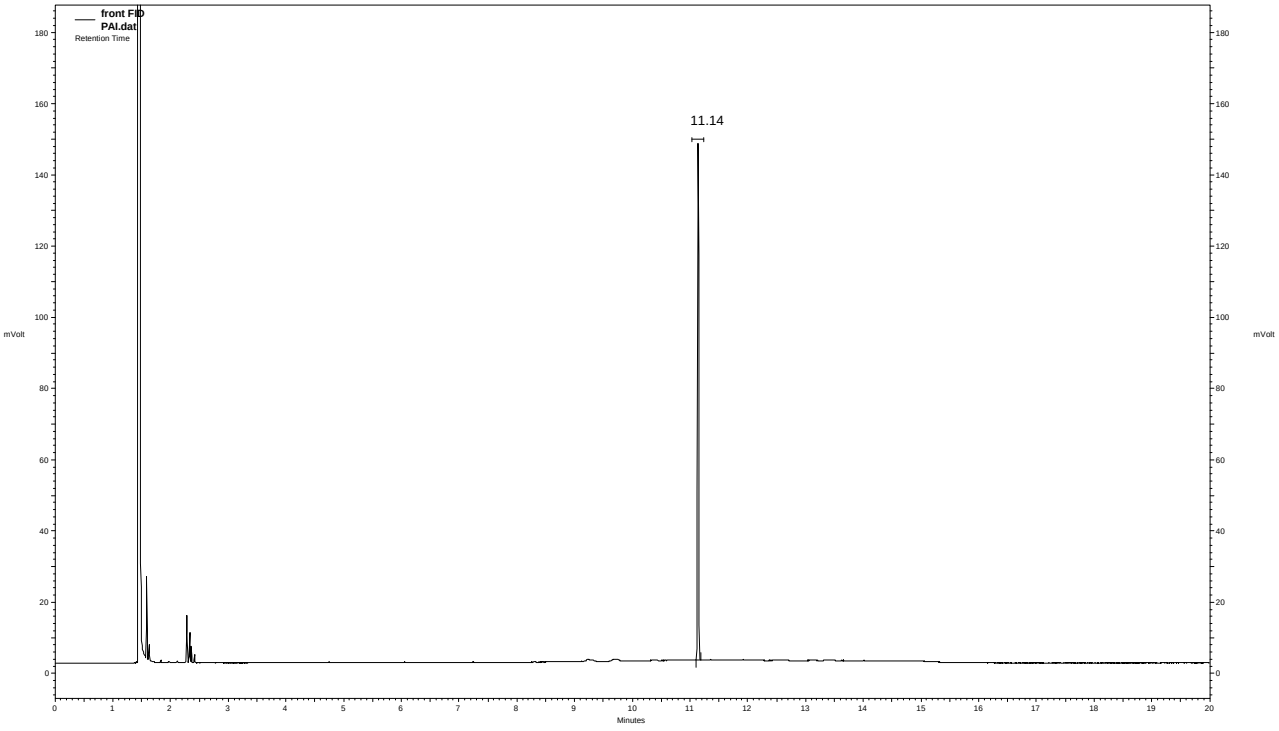


Figure 2 Technical Material TC I

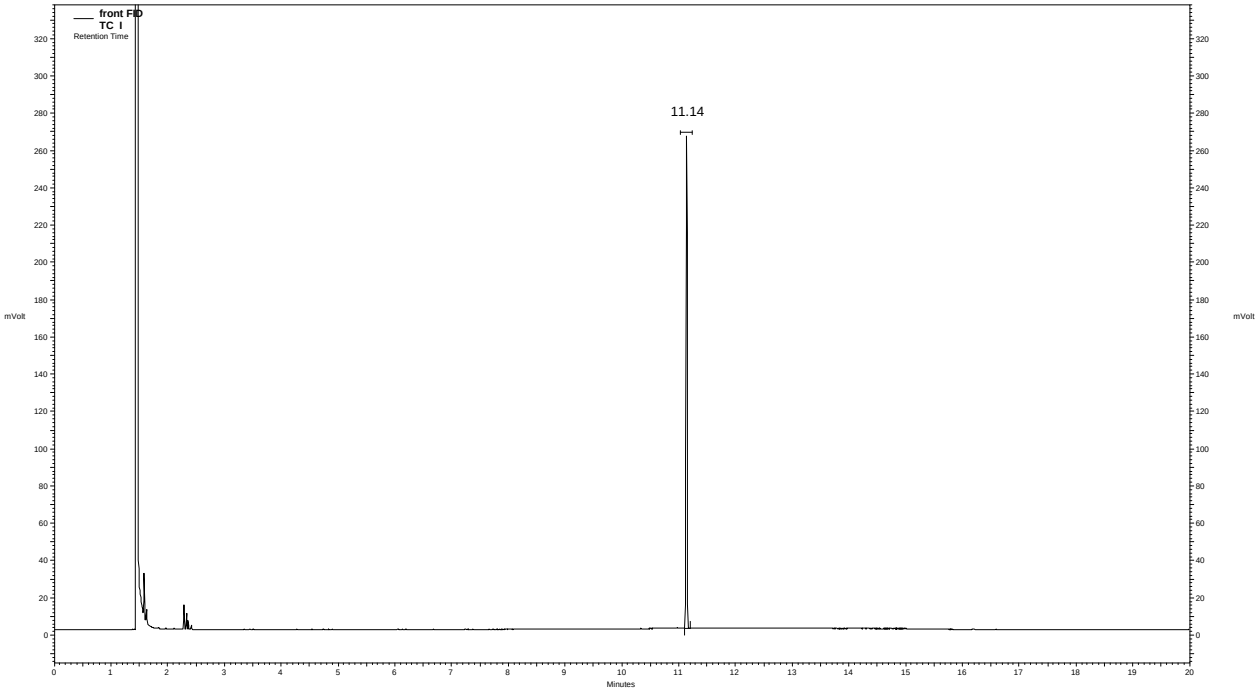


Figure 3 Technical Material TC II

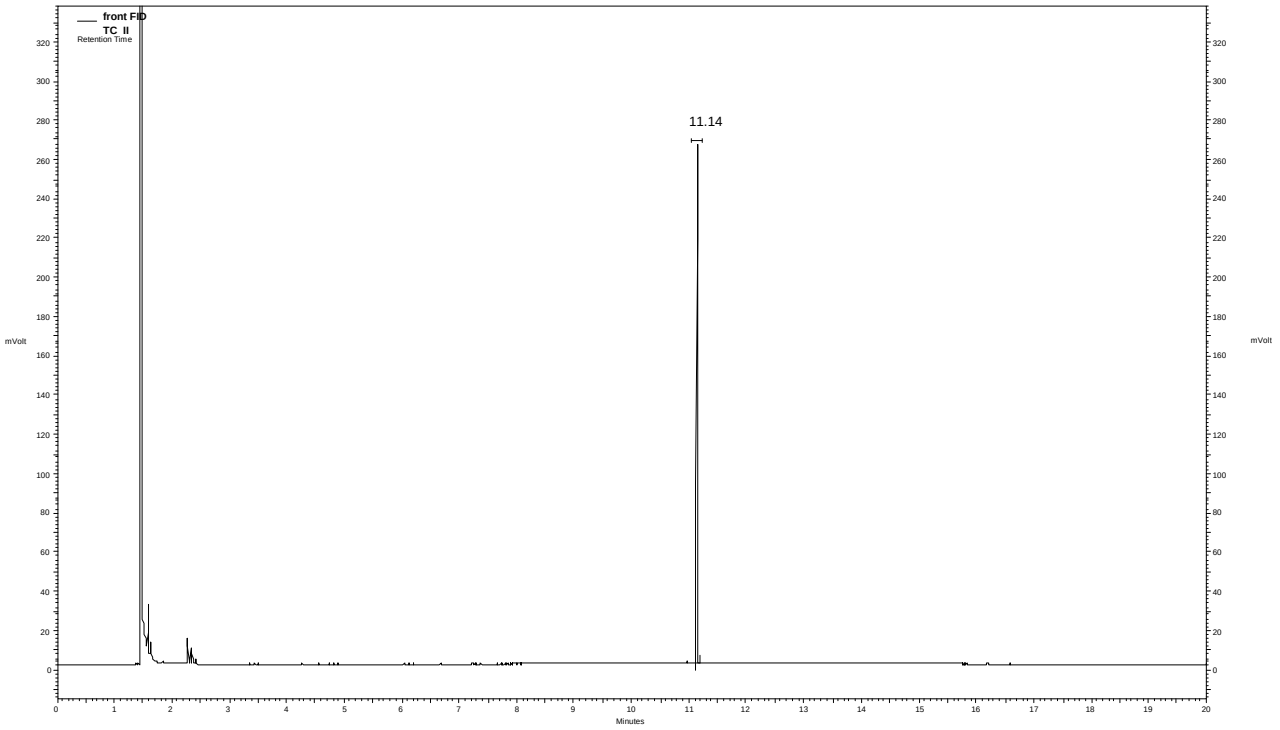


Figure 4 Internal Standard Solution (tetraphenylethylene)

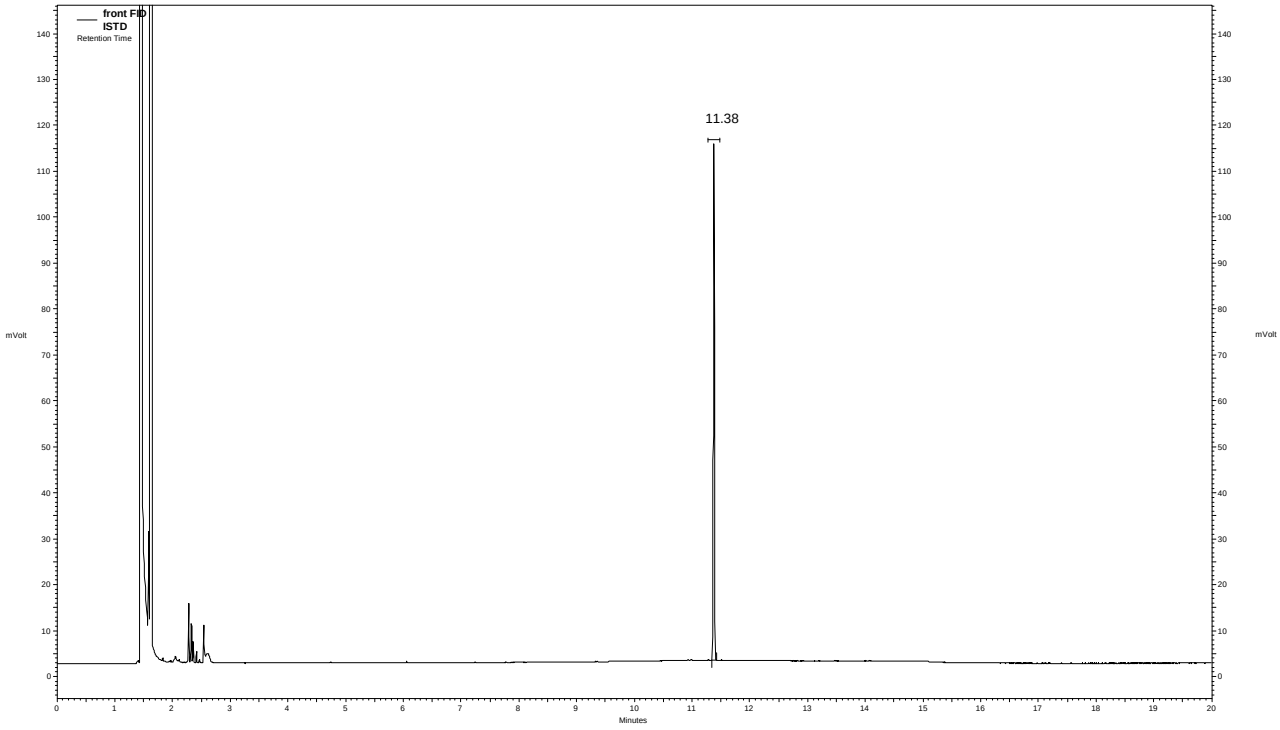
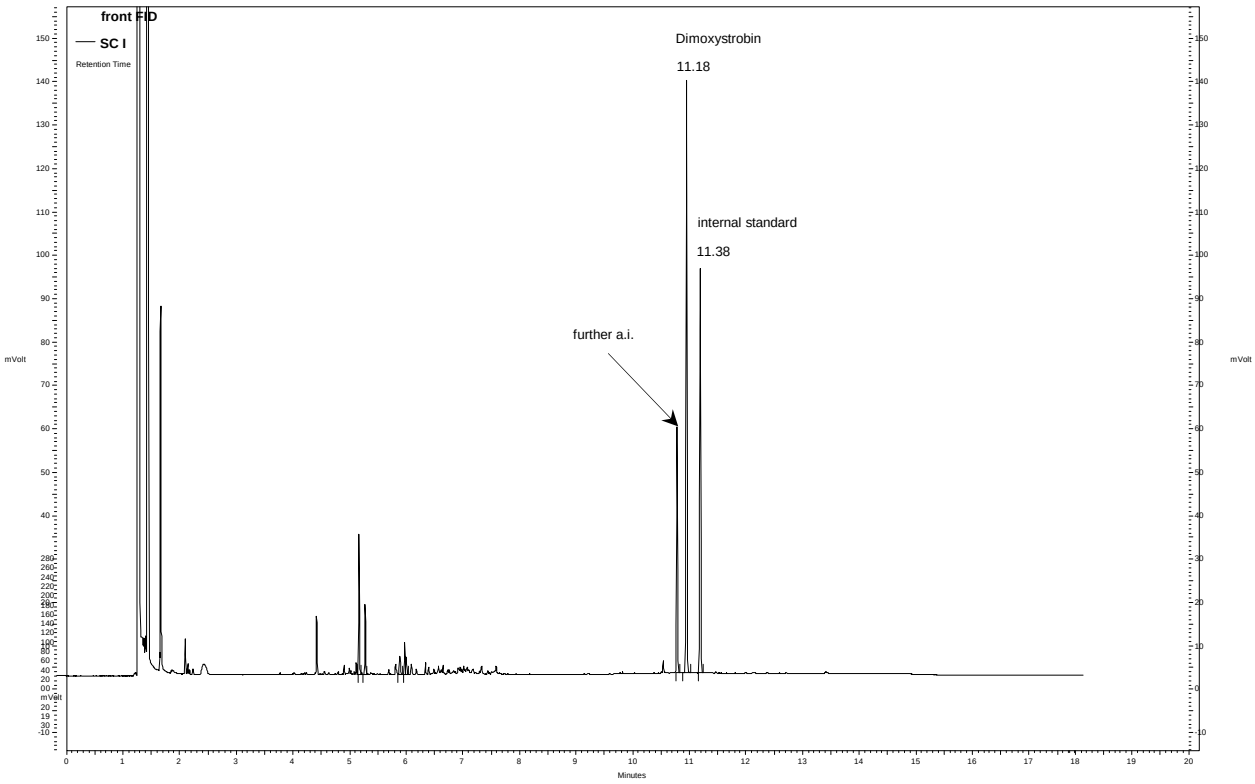


Figure 5 Suspension Concentrate SC I



note: further a.i.: Epoxiconazole

Figure 6 Suspension Concentrate SC I (blank formulation)

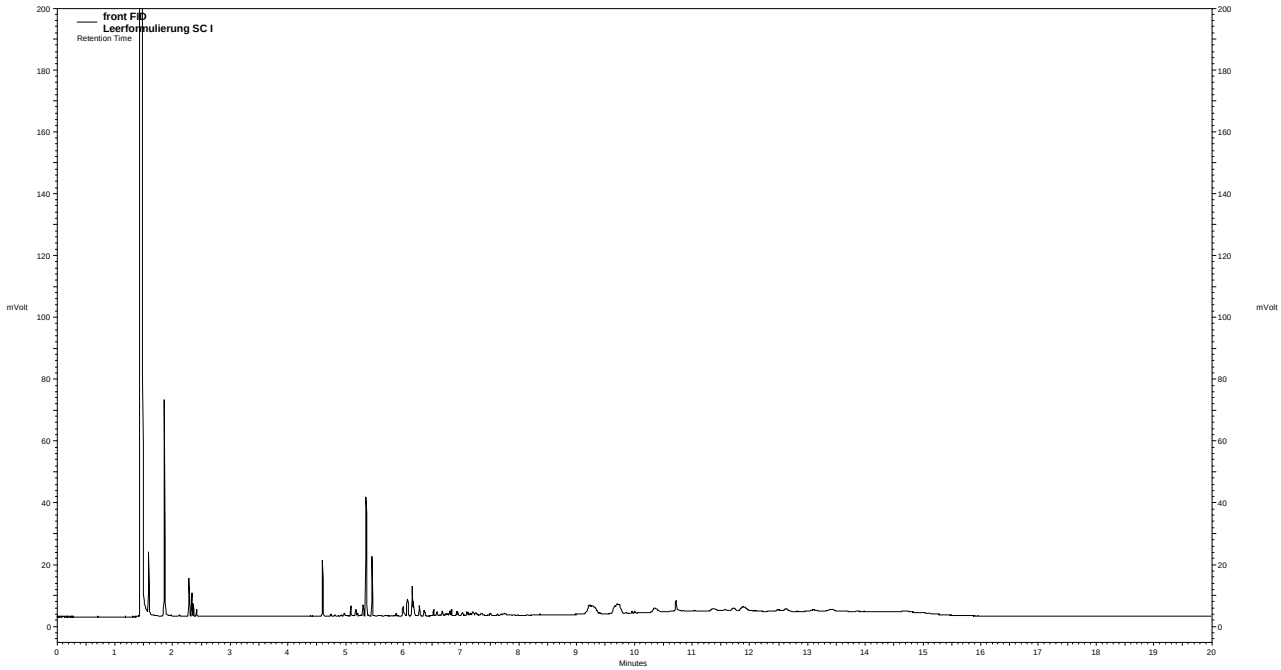
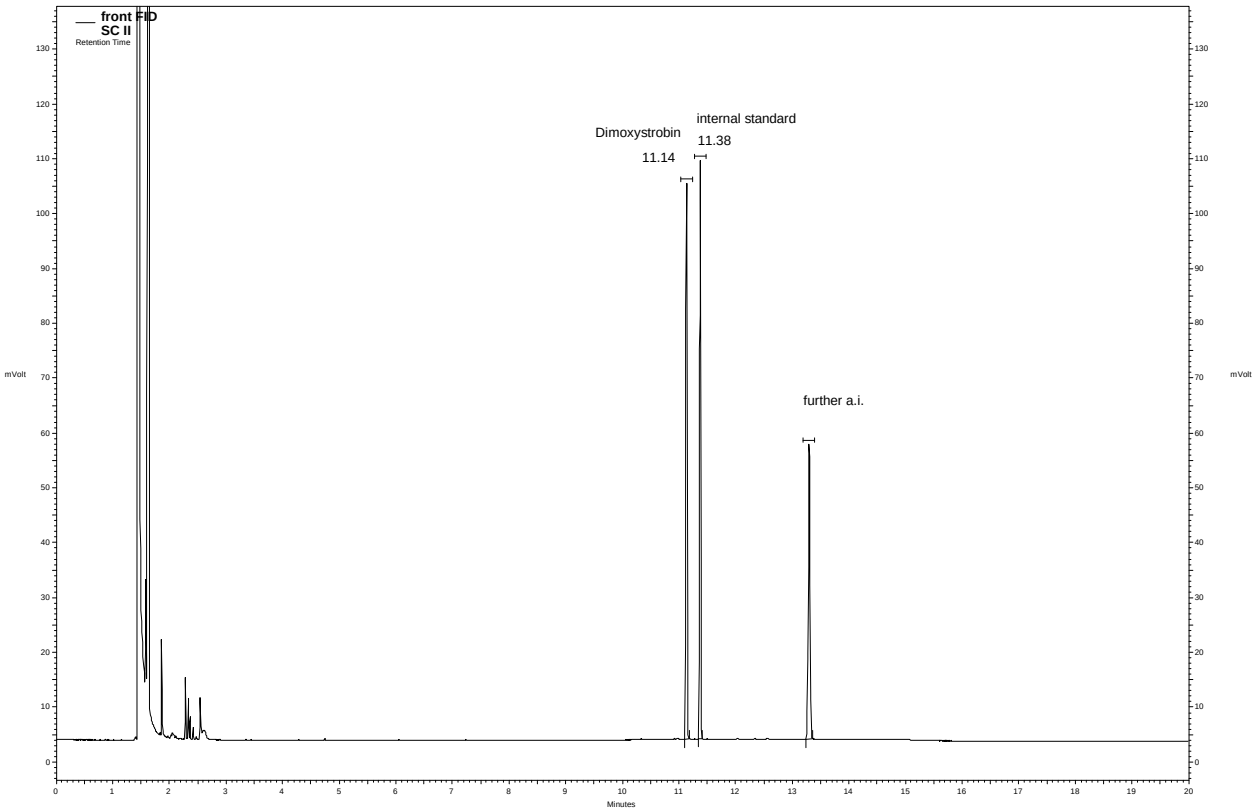


Figure 7 Suspension Concentrate SC II



note: further a.i.: Boscalid

Figure 8 Suspension Concentrate SC II (blank formulation)

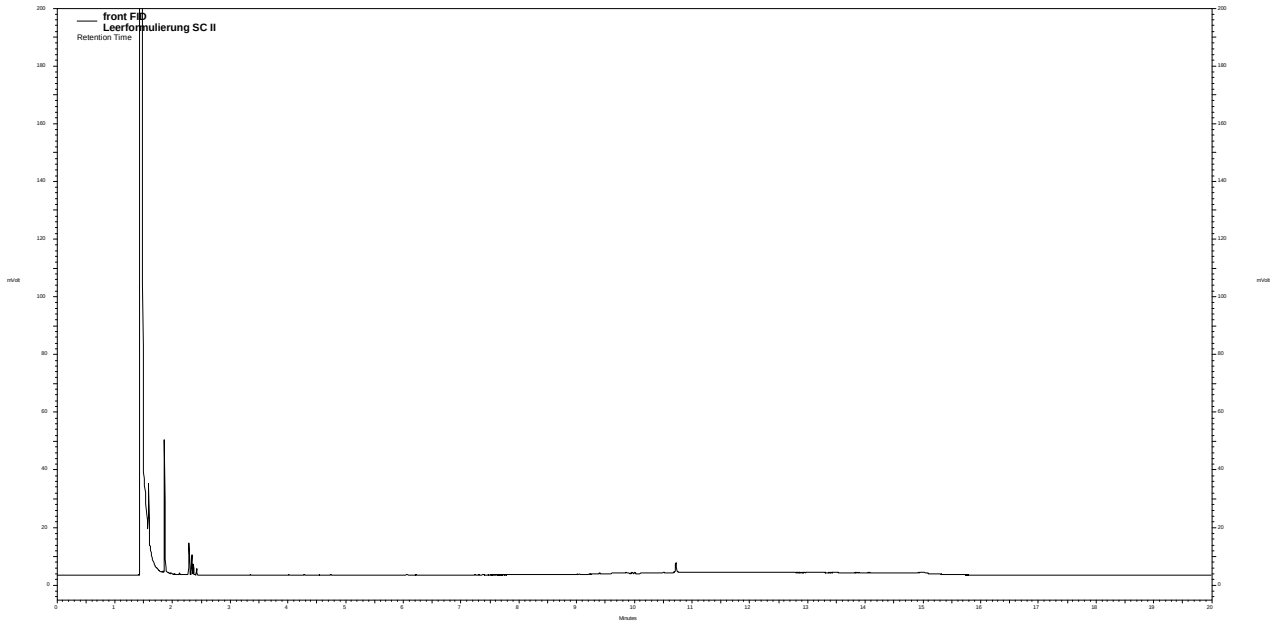
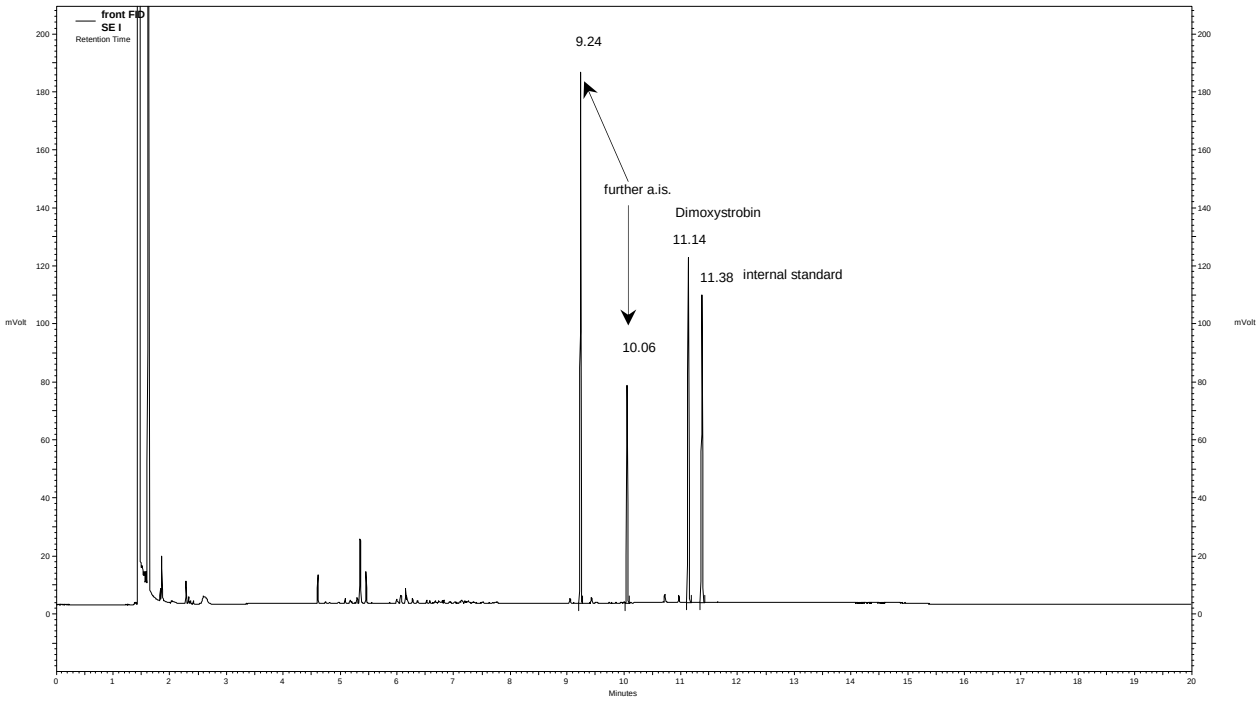


Figure 9 Suspo-emulsion SE



note: further a.is.: Fenpropimorph and Kresoxim-methyl

Figure 10 Suspo-emulsion SE (blank formulation)

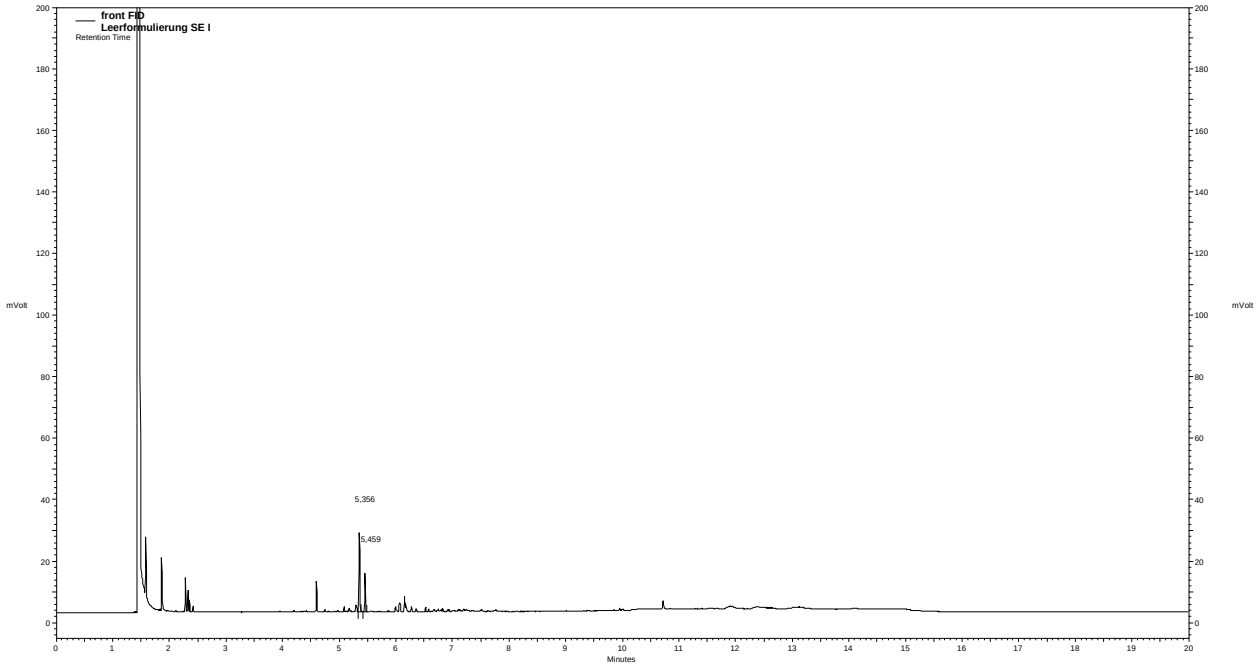


Figure 11 Alternative: Internal Standard Solution (di-n-butyl phthalate)

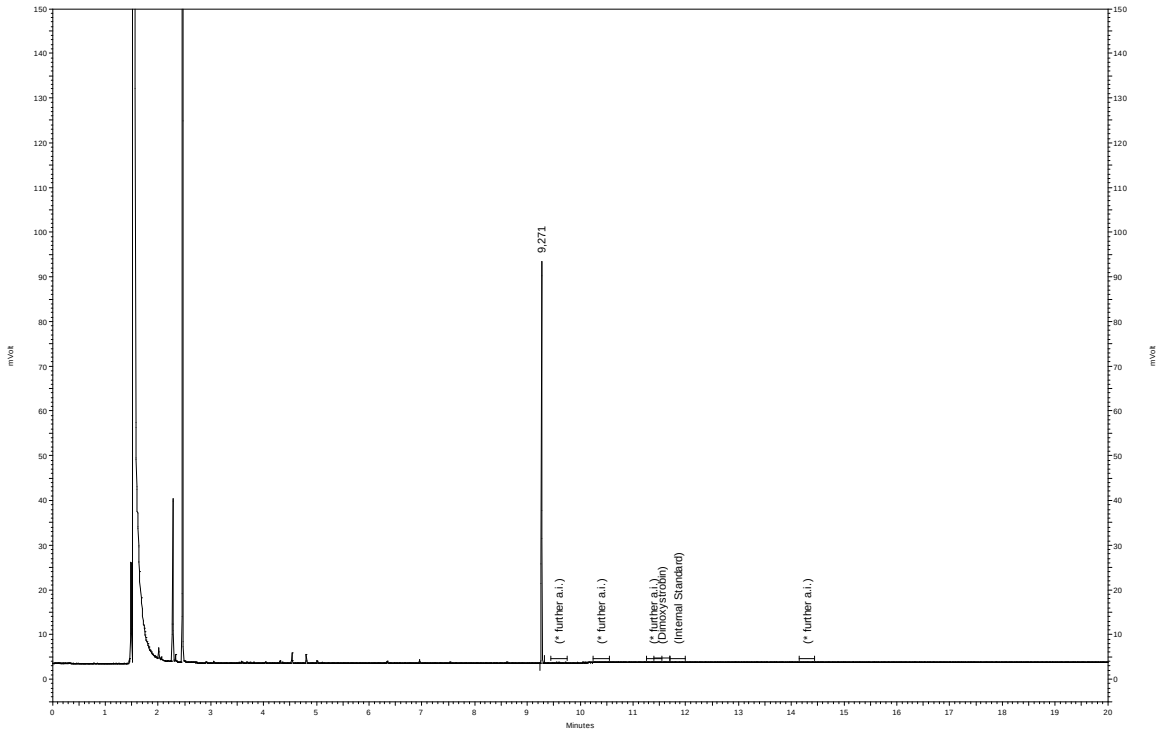


Figure 12 Infrared Spectrum of Dimoxystrobin (Identity test)