



Crop Protection Division
BASF SE, 67114 Limburgerhof, Germany

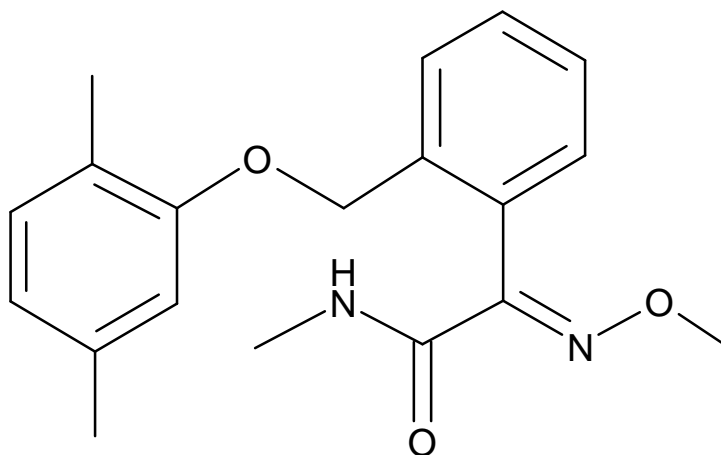
Dimoxystrobin 739

**DAPA collaborative trial, GC Method
CIPAC 4671/m , GC Method**

by

Dr. Jürgen Fries
BASF SE
Agricultural Center Limburgerhof
Crop Protection Division
APR/DA – Li721
D-67114 Limburgerhof
GERMANY

Dimoxystrobin
739



<i>ISO common name</i>	Dimoxystrobin
<i>Chemical name</i>	(E)-o-(2,5-Dimethylphenoxyethyl)-2-methoxyimino-N-methylphenylacetamide
<i>CAS.No.</i>	149961-52-4
<i>Empirical formula</i>	C ₁₉ H ₂₂ N ₂ O ₃
<i>RMM</i>	326.39
<i>m.p.</i>	138 °C
<i>Solubility</i>	In water: 4.3 mg/l at 20 °C
<i>Description</i>	Form: off-white odourless powder
<i>Formulations</i>	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 not differ 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 a 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 μ l

Column fused silica, 30 m x 0.32 mm (i.d) x 0.25 μ m film thickness (DB-1, or equivalent dimethyl polysiloxane phase)

Electronic integrator or data system

Ultrasonic bath

PROCEDURE

(a) *Chromatographic conditions* (typical)

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

Remarks

Flow rates Necessary to use constant flow

Carrier gas Helium should be used, avoid to use Hydrogen

(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 $\pm 0.5\%$ 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)

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.

Typical Chromatograms of Dimoxystrobin

Figure 1 Analytical Standard Dimoxystrobin

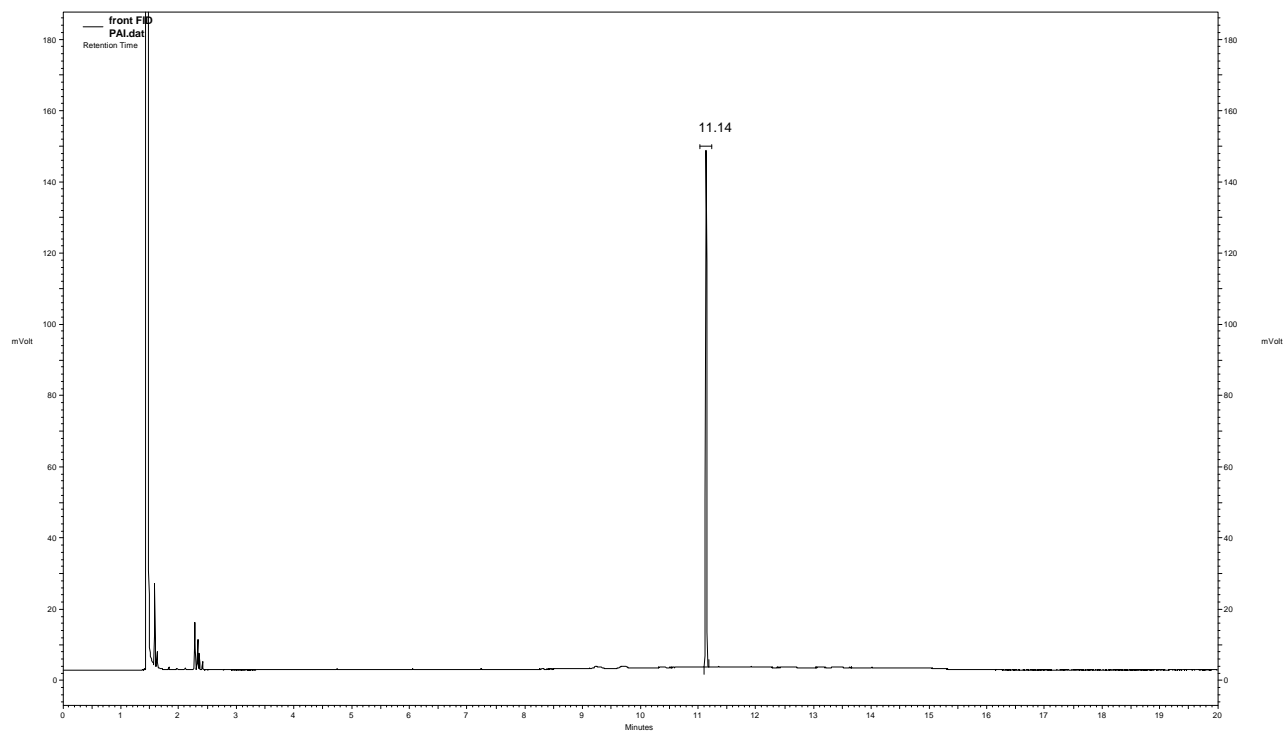


Figure 2 Technical Material TC I

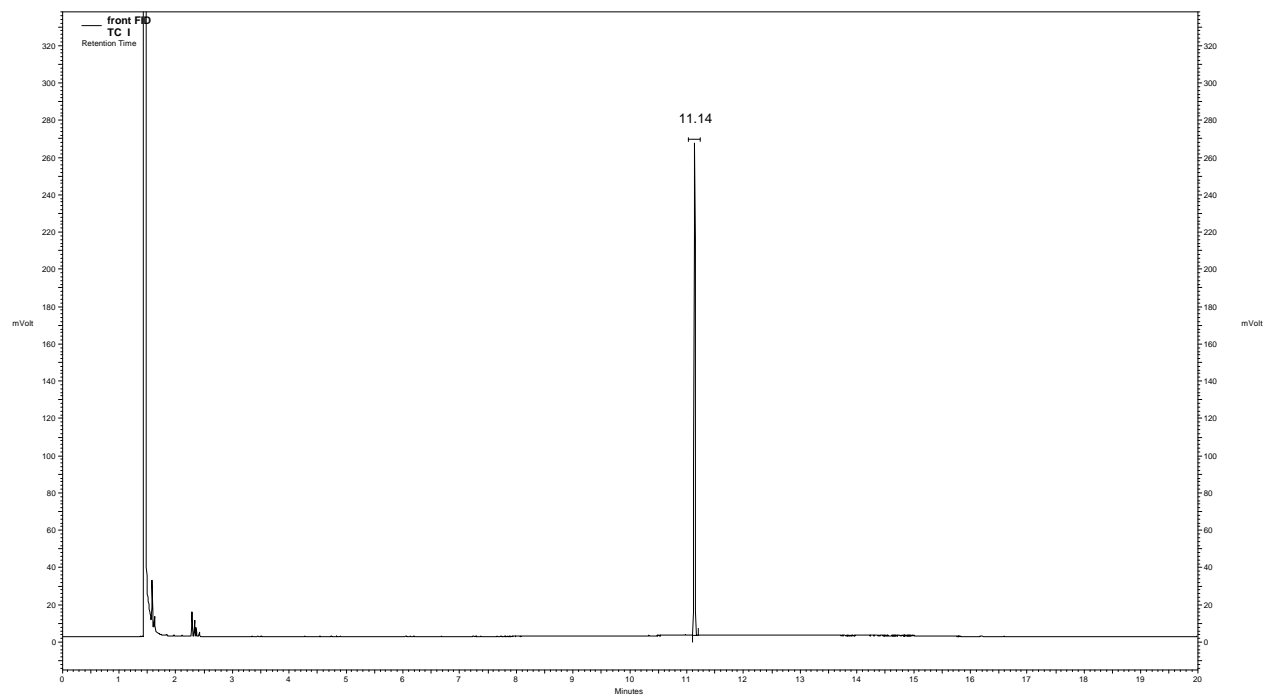


Figure 3 Technical Material TC II

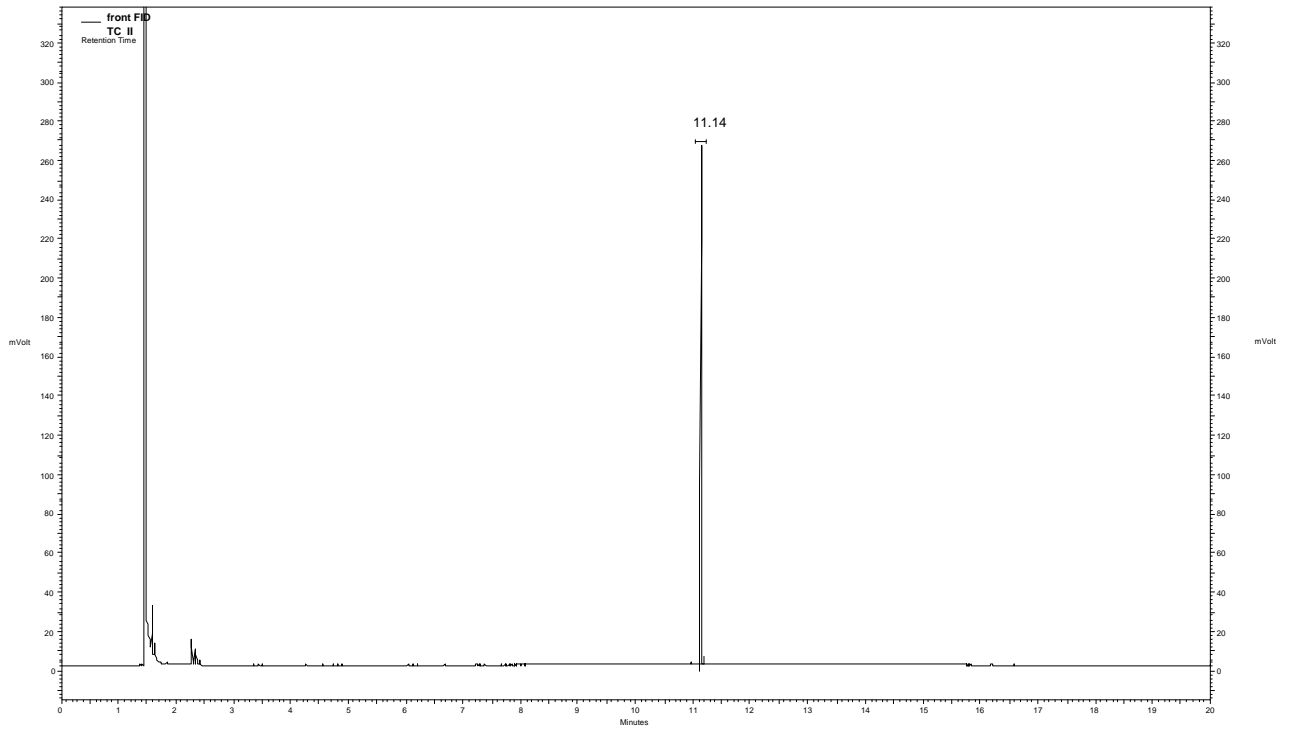


Figure 4 Internal Standard Solution (tetraphenylethylene)

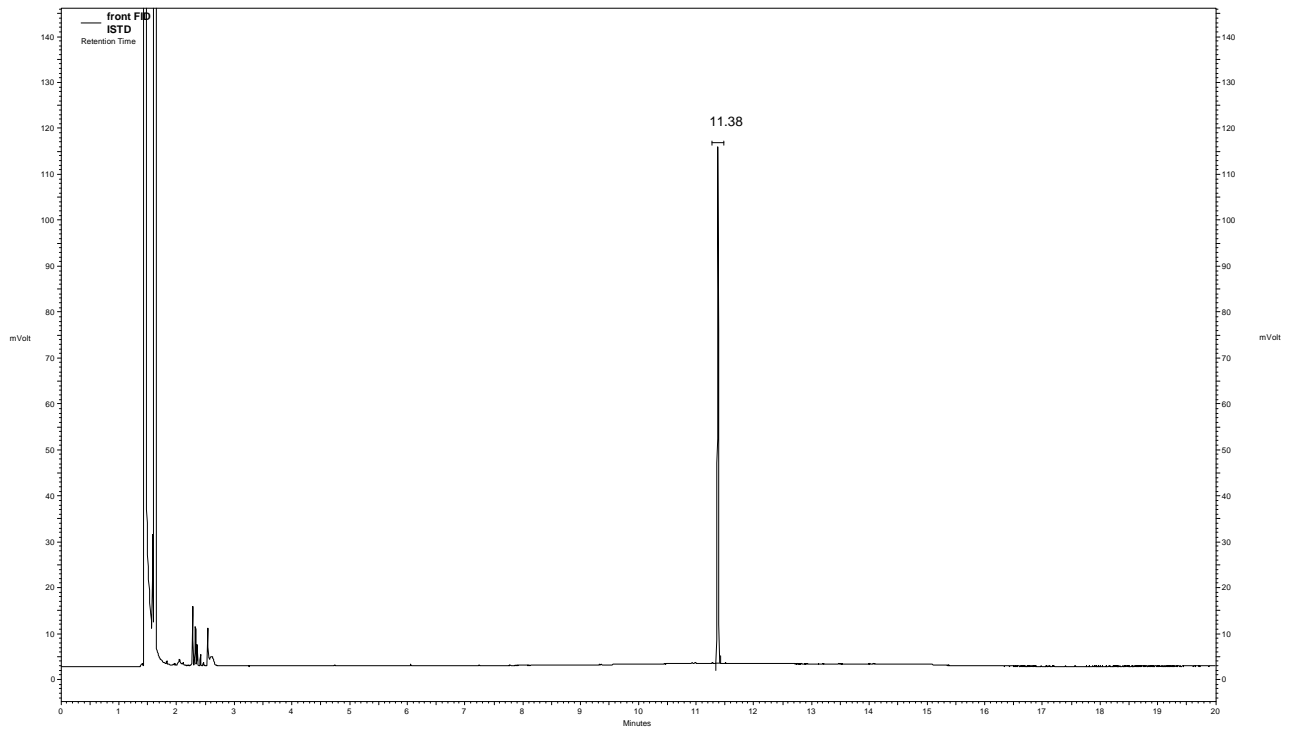
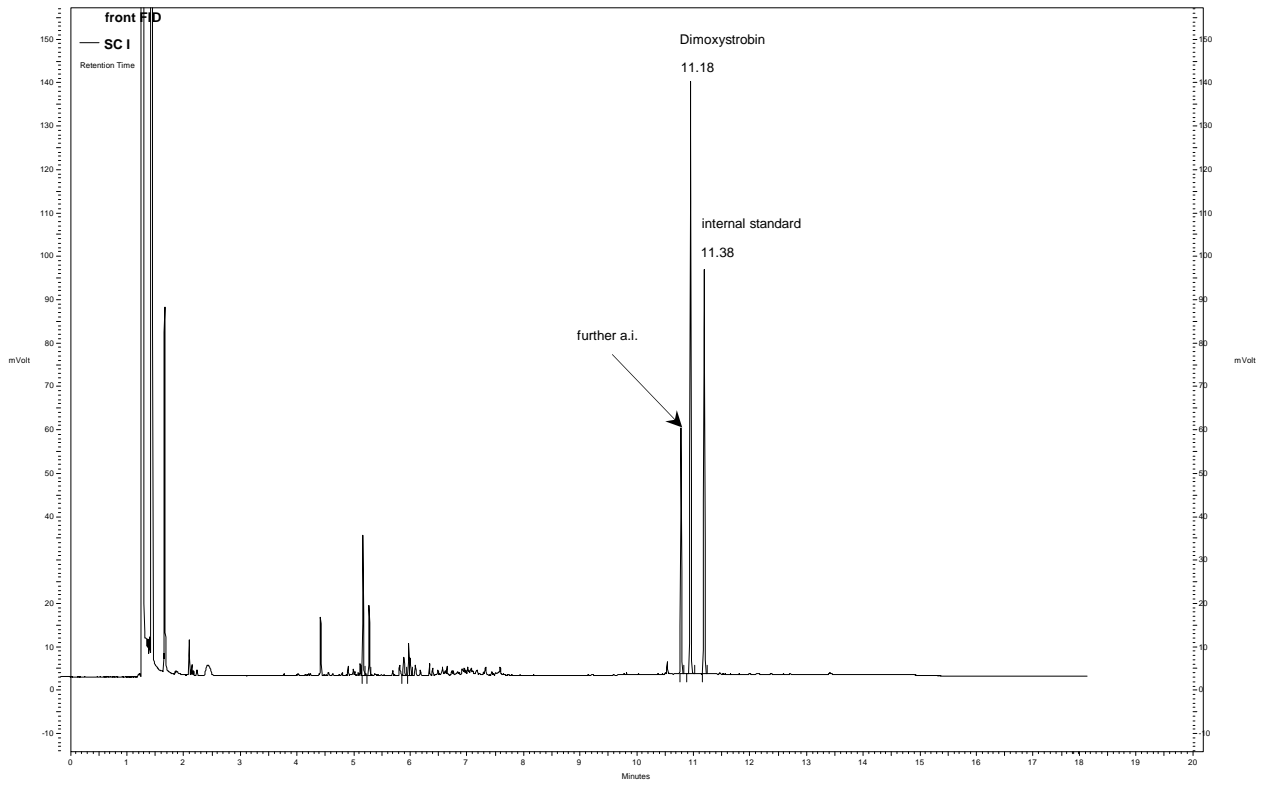


Figure 5 Suspension Concentrate SC I



note: further a.i.: Epoxiconazole

Figure 6 Suspension Concentrate SC I (blank formulation)

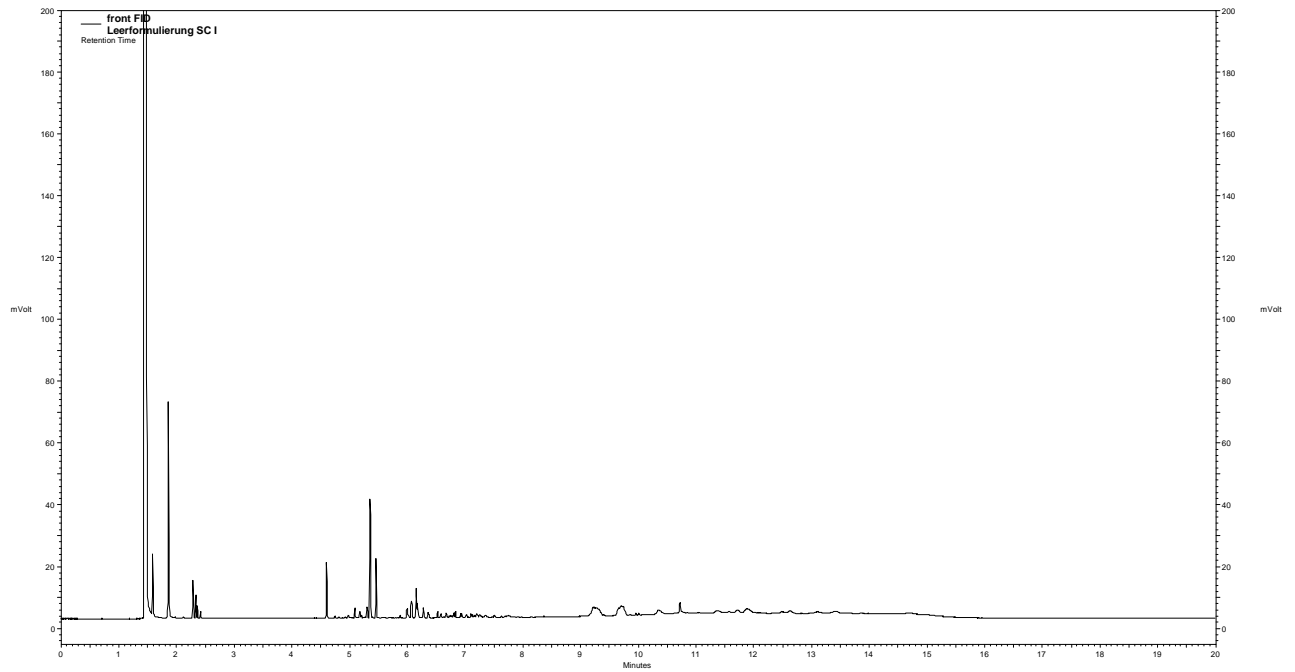
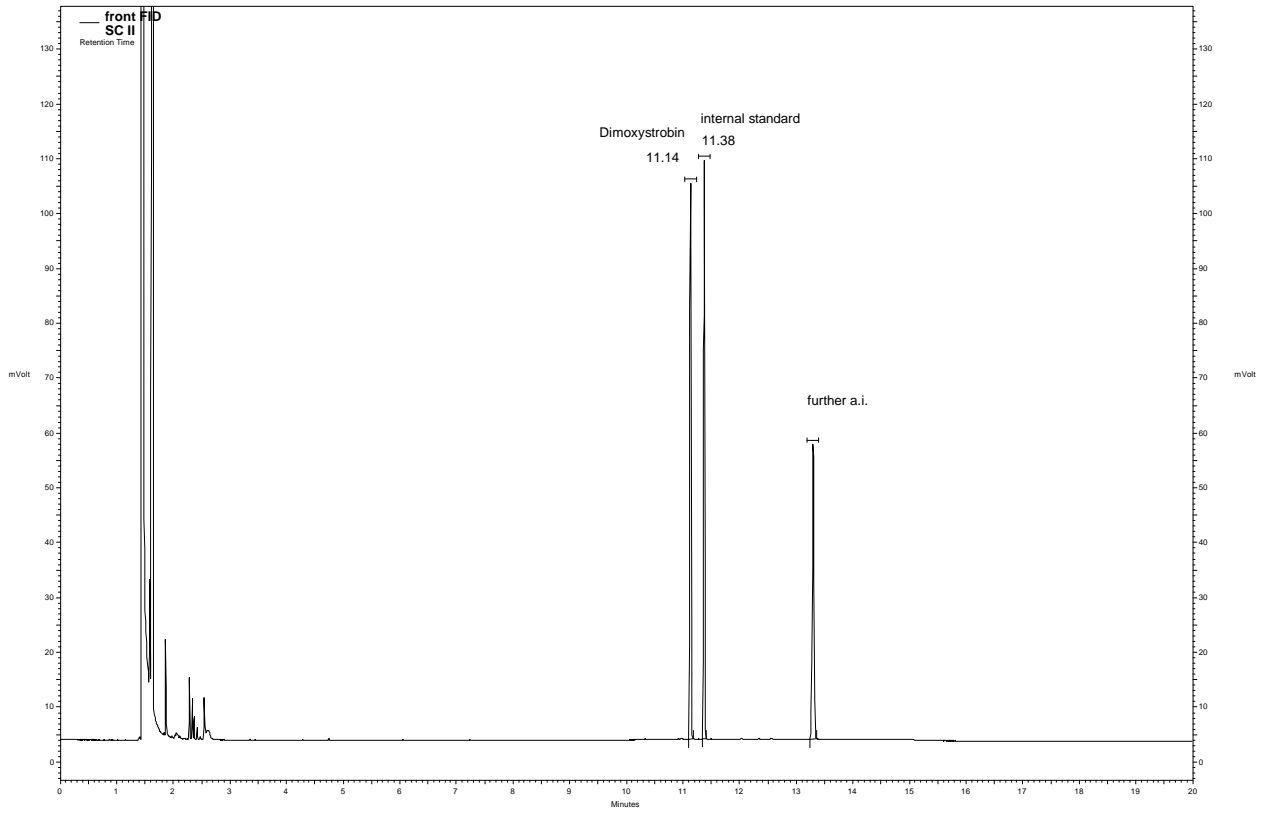


Figure 7 Suspension Concentrate SC II



note: further a.i.: Boscalid

Figure 8 Suspension Concentrate SC II (blank formulation)

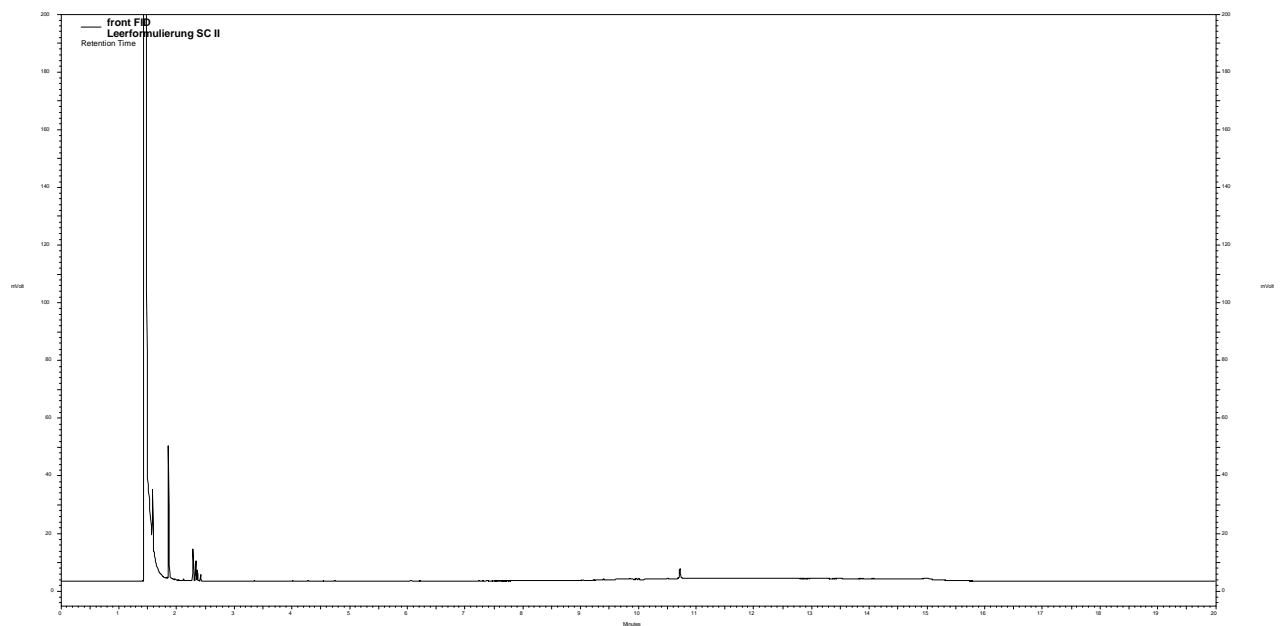
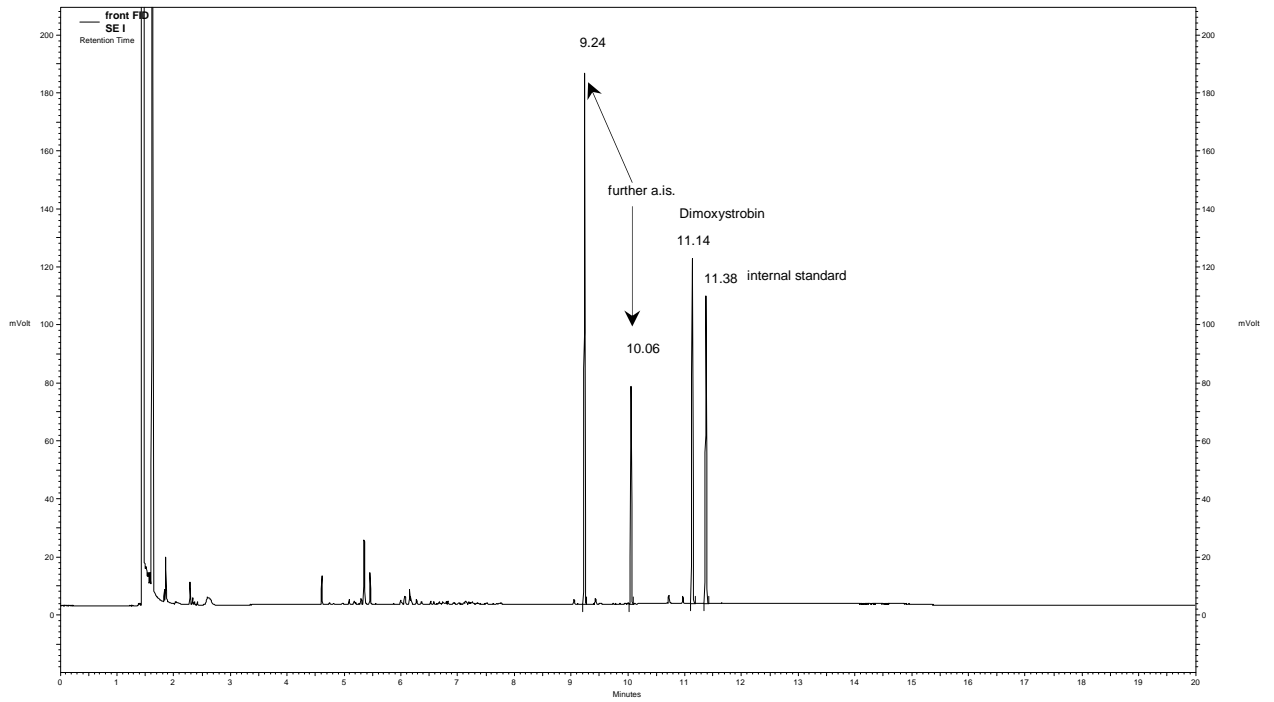


Figure 9 Suspo-emulsion SE



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

Figure 10 Suspo-emulsion SE (blank formulation)

