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Boscalid
673

CIPAC 4611/m, HPLC Method

by

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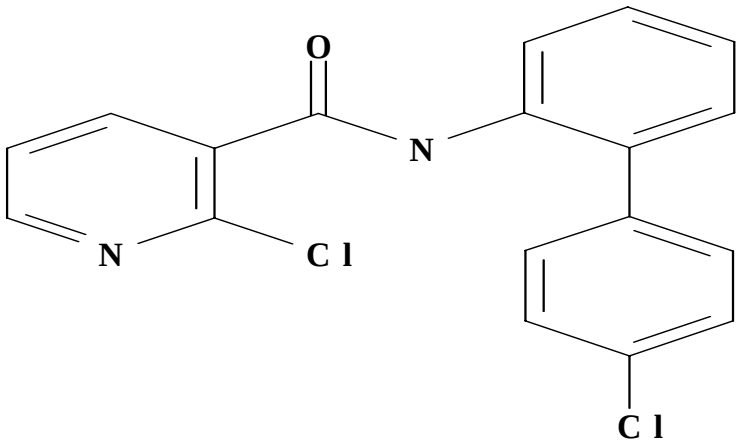


ISO common name	Boscalid
Chemical name	2-chloro-N-(4`-chlorobiphenyl-2-yl)nicotinamide
CAS.No.	188425-85-6
Empirical formula	$C_{18}H_{12}Cl_2N_2O$
RMM	343.2
m.p.	143 to 145 °C
Solubilty	In water: 4.6 mg/l at 20 °C
Description	Form: white odourless powder
Formulations	Water Dispersible Granules Suspension Concentrates Suspo-Emulsions

BOSCALID TECHNICAL
*__673__/TC/(M)/-

1 Sampling. Take at least 50 g.

2 Identity test

2.1 HPLC. Use the HPLC method below. The relative retention time of the Boscalid peak in the sample solution should not deviate by more than 10 s from that of the calibration solution.

2.2 Infrared. Prepare potassium bromide discs from the sample and from pure Boscalid 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 Boscalid

OUTLINE OF METHOD. Boscalid is determined by reversed phase high performance liquid chromatography using UV detection at 260 nm and external standardization (Fig.1).

REAGENTS

Boscalid reference standard of known purity
Water deionized HPLC grade
Acetonitrile HPLC grade
Ammonium acetate 98% (pro analysis)
Mobile phase A: water – acetonitrile – ammonium acetate,
350 ml + 650 ml + 770 mg
Mobile phase B: water – acetonitrile – ammonium acetate,
100 ml + 900 ml + 770 mg

Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) about 100 mg of the Boscalid standard (*s* mg) into separate volumetric flasks (100 ml). Add acetonitrile (about 70 ml) and place the flasks in an ultrasonic bath for 5 min. Allow to cool to ambient temperature and fill to the mark with acetonitrile. Transfer by pipette 10 ml of these solutions into separate volumetric flasks (50 ml), add 5 ml water and fill to the mark with acetonitrile (calibration solutions C_1 and C_2).

APPARATUS

High performance liquid chromatograph equipped with an UV detector and an injection system capable to inject 5 µl
Column stainless steel, 250 x 4.6 mm (i.d), packed with J`sphere ODS-H80, 4 µm, or equivalent material with the same selectivity.
Note: A list of analytical columns used by the participants in the CIPAC collaborative trial is given in the REMARKS.
Electronic integrator
Ultrasonic bath

PROCEDURE

(a) Chromatographic conditions (typical)

Column temperature	room temperature (about 23 °C)
Flow rate of the mobile phase	see gradient
Measuring wavelength	260 nm
Injection volume	5 µl
Run time	approximately 26 min
Retention time	approximately 6.8 min

Gradient

Time in min	% A	% B	Flow in ml/min
0	100	0	1
14	100	0	1
14.5	0	100	1
15	0	100	2
22	0	100	2
22.5	0	100	1
23	100	0	1
26	100	0	1

(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) Preparation of Sample. Weigh (to the nearest 0.1 mg) sufficient sample to contain about 100 mg Boscalid into a volumetric flask (100 ml). Add acetonitrile (about 50 ml) swirl and place the flask in an ultrasonic bath for 5 min. Add water to just below the mark. Allow to cool to ambient temperature and fill to the mark with water and mix. Transfer by pipette 10 ml of these solutions into separate volumetric flasks (50 ml) and mark the volume with acetonitrile (Solution S).

(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 1 (double injection), calibration solution 2, sample solution 2 (double injection), calibration solution 1. Measure the relevant peak areas. Calculate the mean value of each pair of response factors bracketing the injections of the two samples and use this value for calculating the Boscalid contents of the bracketed sample solution injections.

(e) Calculation

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

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

where:

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

REMARKS

List of analytical HPLC columns used by the participants in the CIPAC collaborative trial:

Inertsil ODS 2, 5µm, 250 x 4.6 mm,

Nucleodur Sphinx RP, 5µm, 250 x 4 mm,

Phenomenex Luna C18, 100A, 5µm, 250 x 4.6 mm,

J´sphere ODS-H80, 4µm, 250 x 3.0 mm,

ALLTIMA HP C18 HL, 5µm, 250 x 4.6 mm,

LiChrospher 100 RP-18, 5µm, 250 x 4.0 mm,

Allure C18, 5µm, 250 x 4.6 mm,

J´sphere ODS-M80, 4µm, 250 x 4.6 mm,

Inertsil ODS 2, 5µm, 250 x 4.6 mm,

Phenomenex ODS3 100A, 5µm, 250 x 4.6 mm.

BOSCALID WATER DISPERSIBLE GRANULES
*673/WG/(M)/-

1 Sampling. Take at least 50 g.

2 Identity test
2.1 HPLC. As for Boscalid technical 673/TC/(M)/2.1.

3 Boscalid. As for Boscalid technical 673/TC/(M)/3 except:

PROCEDURE
change *(c) Preparation of sample solution* to:
Weigh (to the nearest 0.1 mg) sufficient sample to contain about 100 mg Boscalid into a volumetric flask (100 ml). Add water (about 10 ml) to form a slurry, acetonitrile (about 50 ml), swirl and place the flask in an ultrasonic bath for 5 min. Add water to just below the mark. Allow to cool to ambient temperature and fill to the mark with water and mix. Transfer by pipette 10 ml of these solutions into separate volumetric flasks (50 ml) and mark the volume with acetonitrile (Solution S).

BOSCALID SUSPENSION CONCENTRATES
*673/SC/(M)/-

1 Sampling. Take at least 50 ml.

2 Identity test
2.1 HPLC. As for Boscalid technical 673/TC/(M)/2.1.

3 Boscalid. As for Boscalid water dispersible granules 673/WG/(M)/3.

BOSCALID SUSPO-EMULSIONS
*673/SE/(M)/-

1 Sampling. Take at least 50 ml.

2 Identity test
2.1 HPLC. As for Boscalid technical 673/TC/(M)/2.1.

3 Boscalid. As for Boscalid water dispersible granules 673/WG/(M)/3.

Typical Chromatograms of Boscalid

Figure 1 Analytical Standard Boscalid

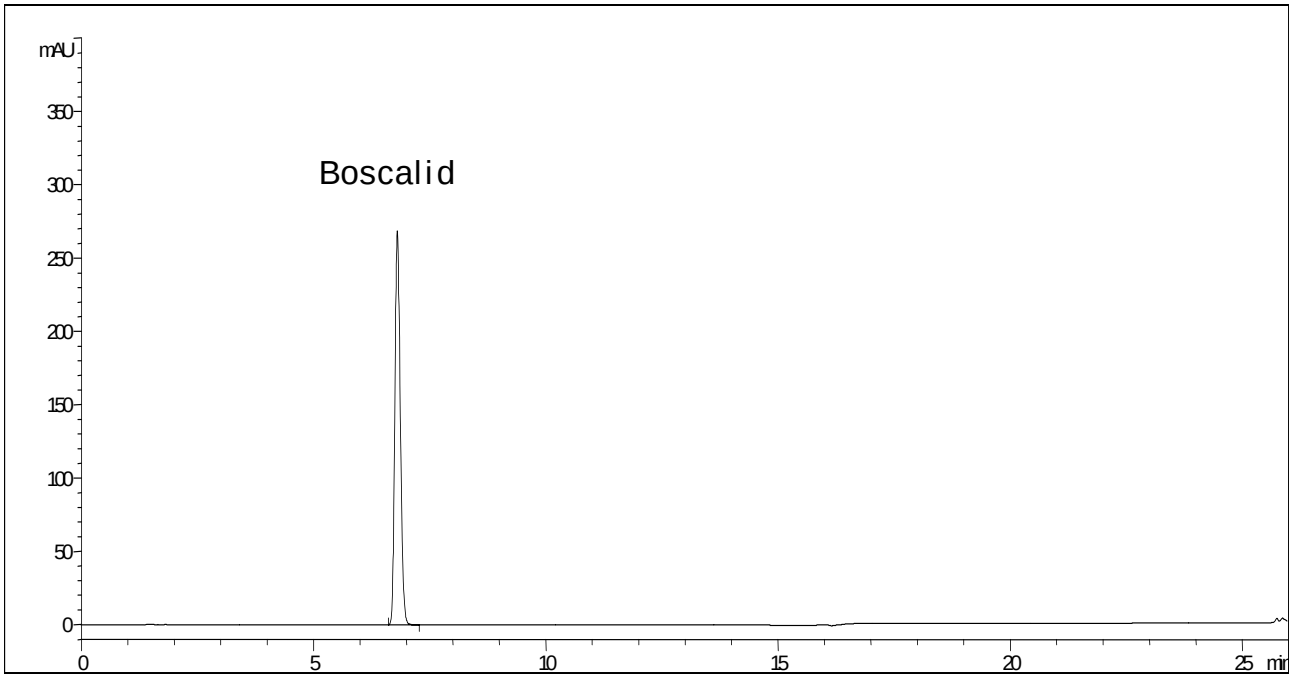


Figure 2 Technical Material TC

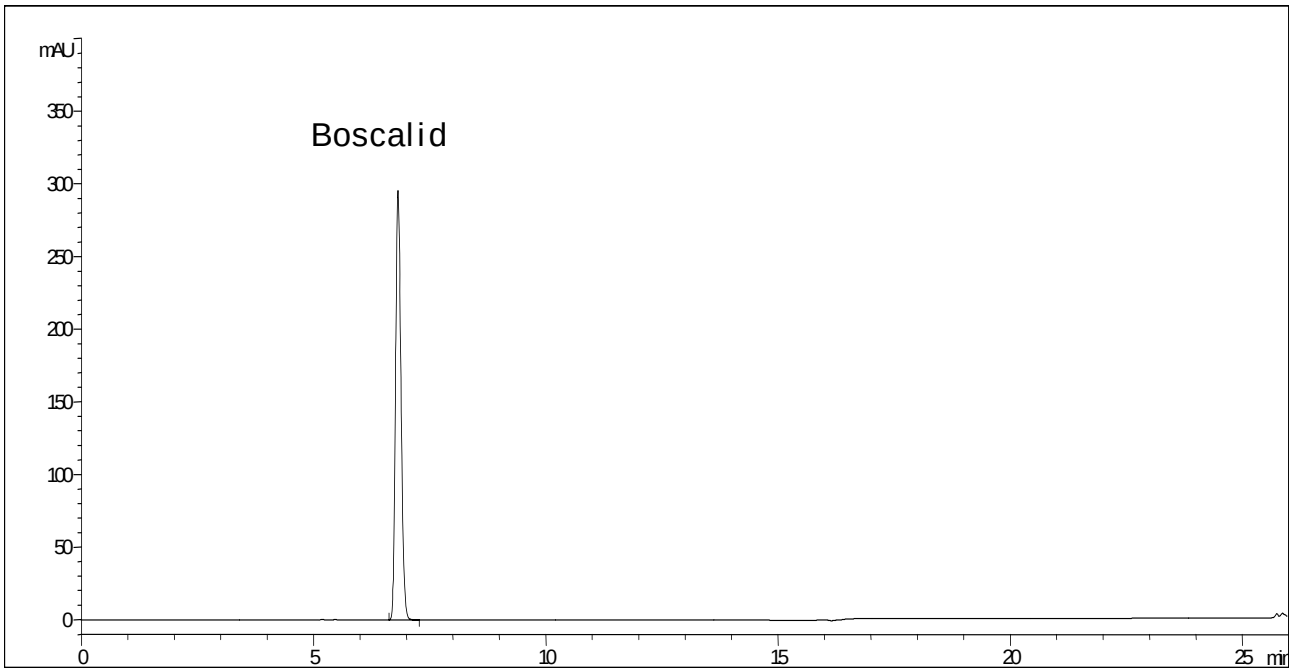


Figure 3 Technical Material TC

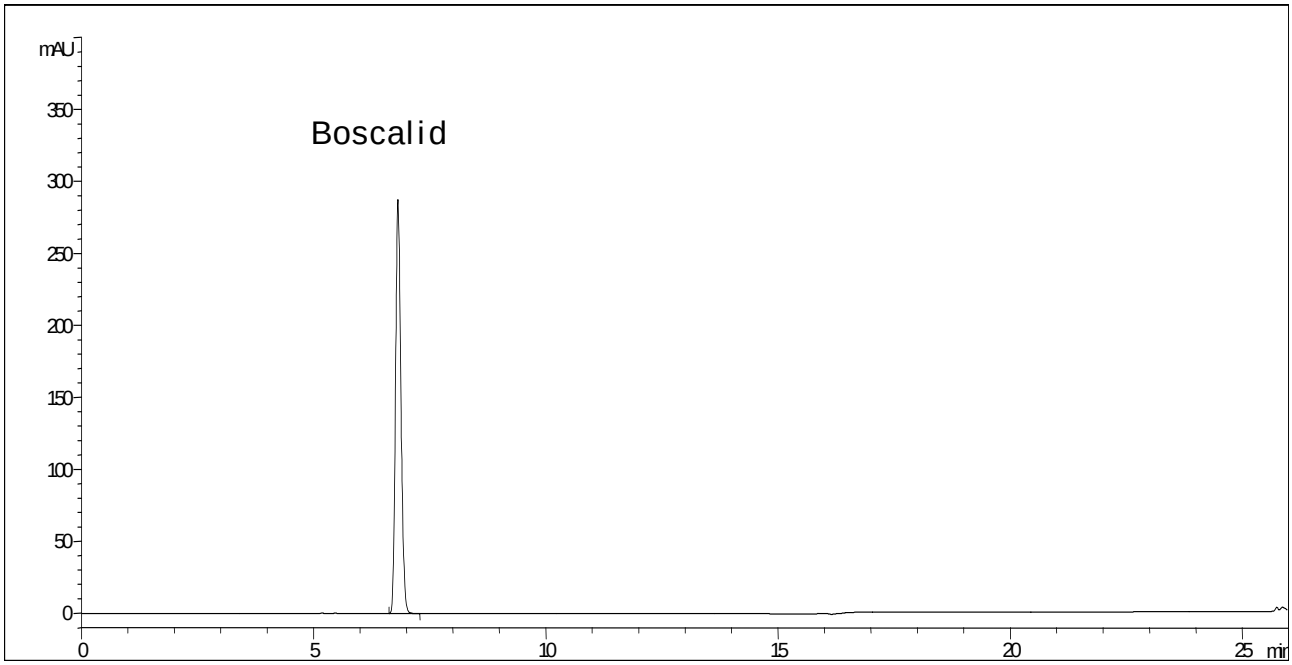


Figure 4 Water Dispersible Granule WG

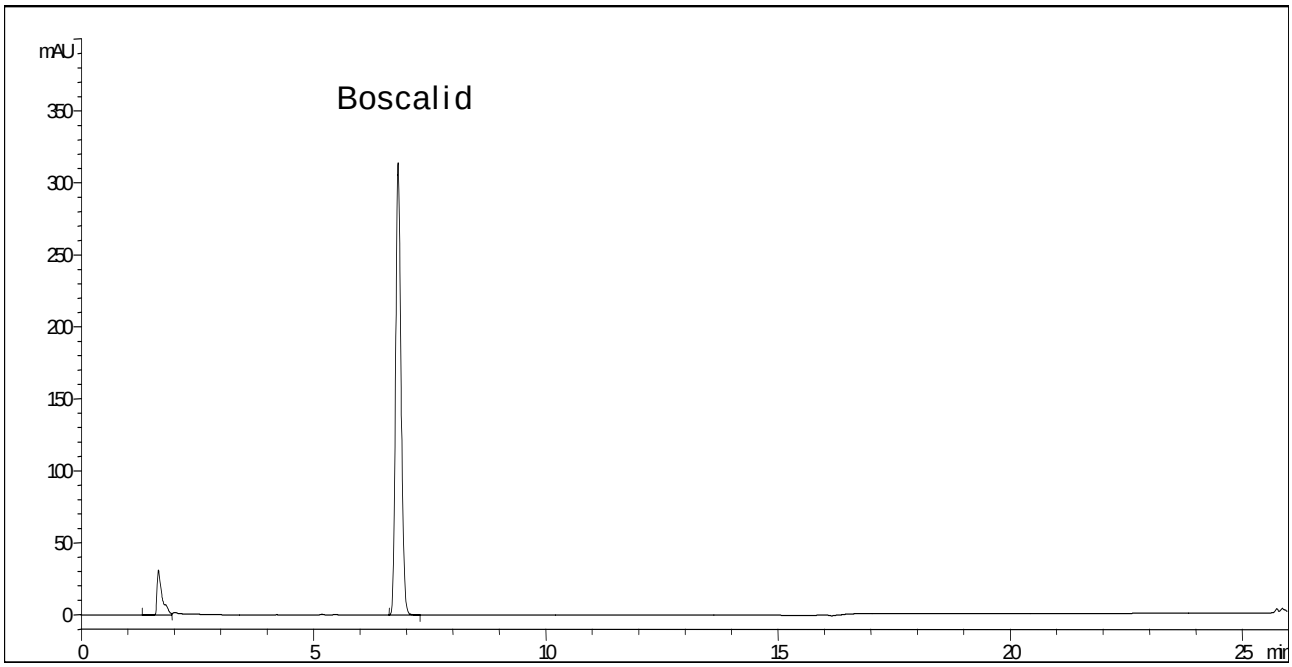


Figure 5 Water Dispersible Granule WG (blank formulation)

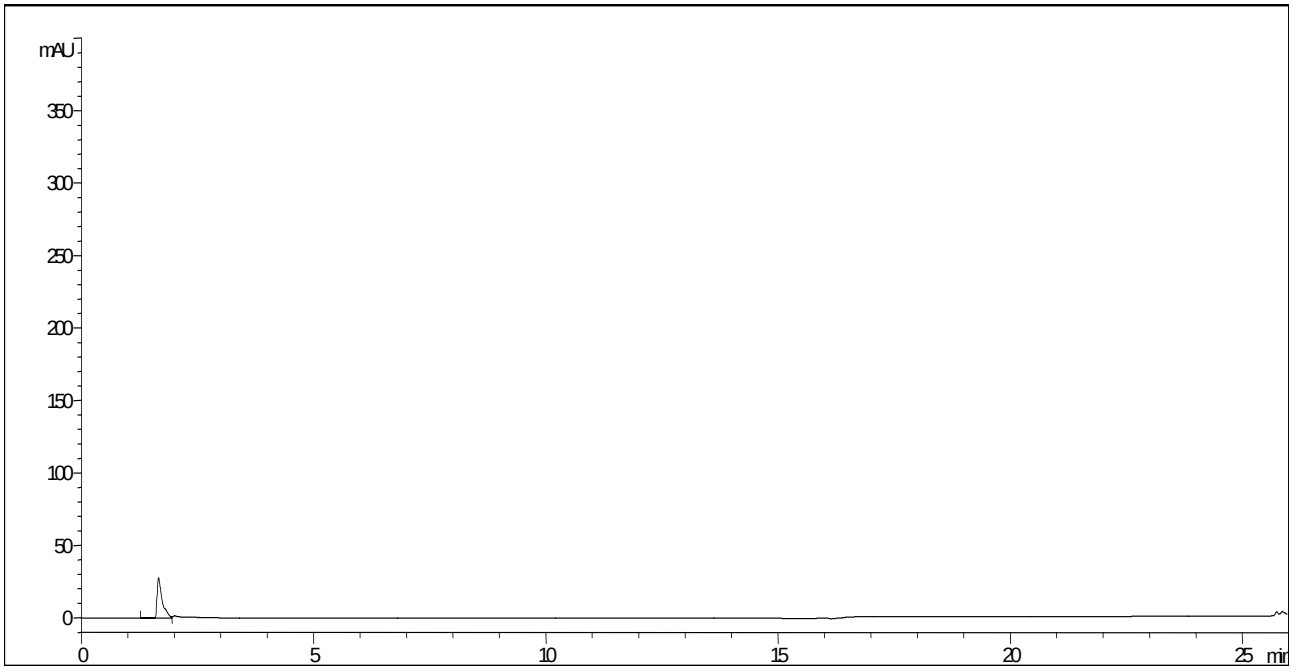


Figure 6 Suspension Concentrate SC

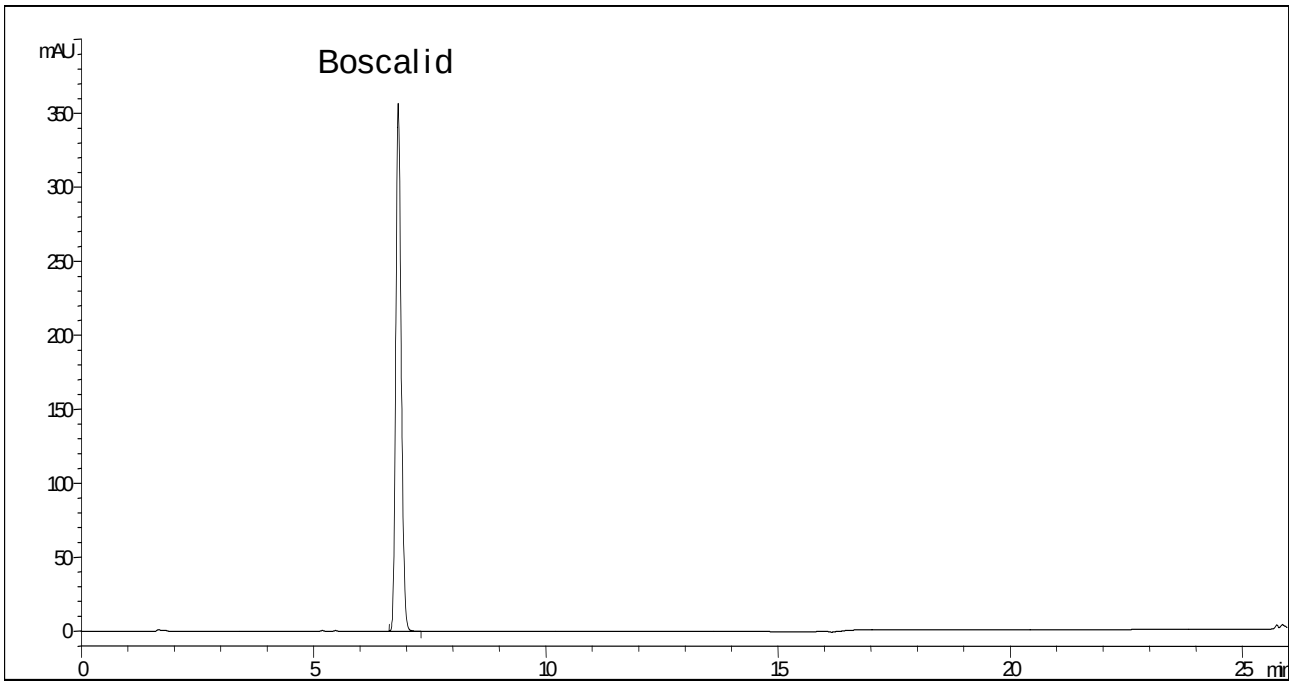


Figure 7 Suspension Concentrate SC (blank formulation)

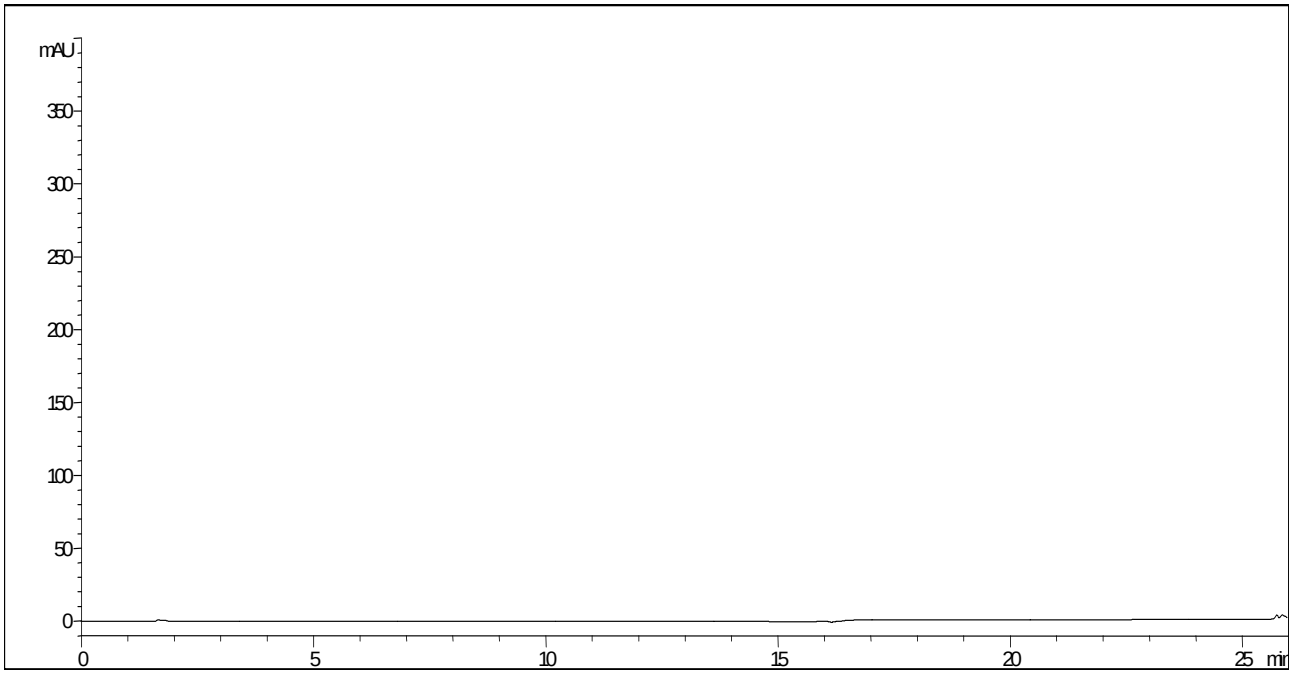
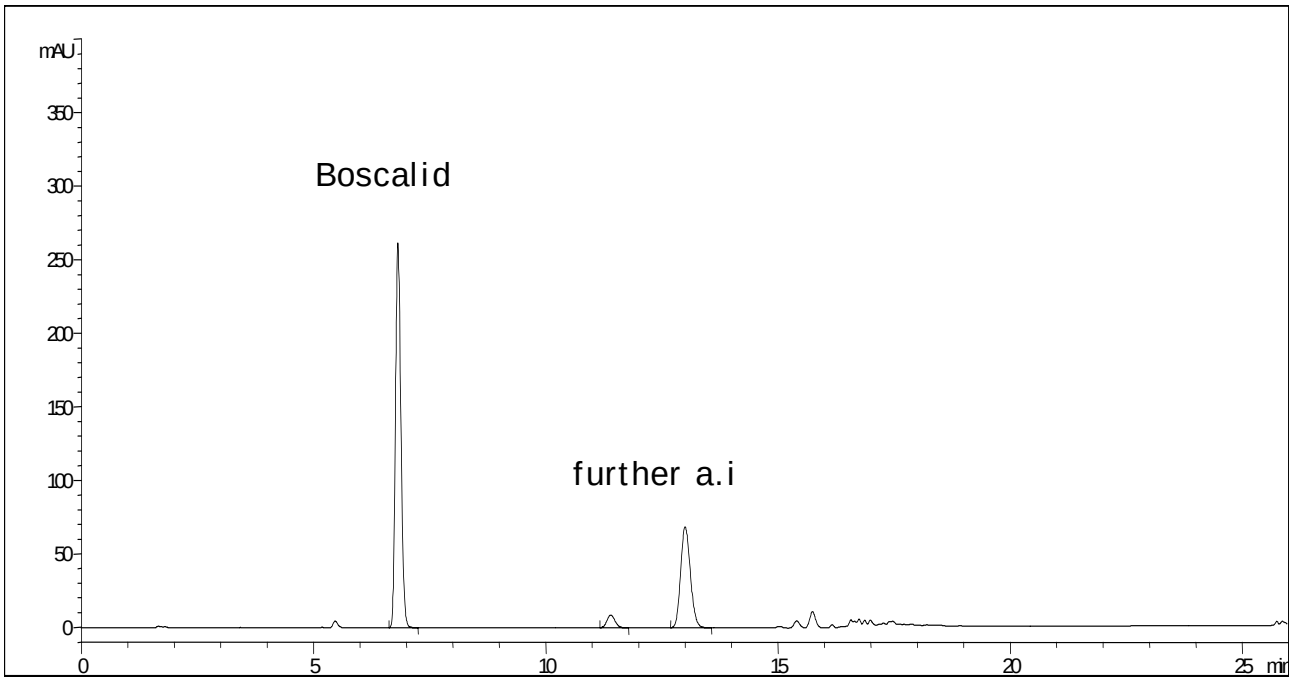


Figure 8 Suspo-emulsion SE



note: further a.i.: Pyraclostrobin

Figure 9 Suspo-emulsion SE (blank formulation)