Tebuconazole (CIPAC 494)

method extension

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Objective

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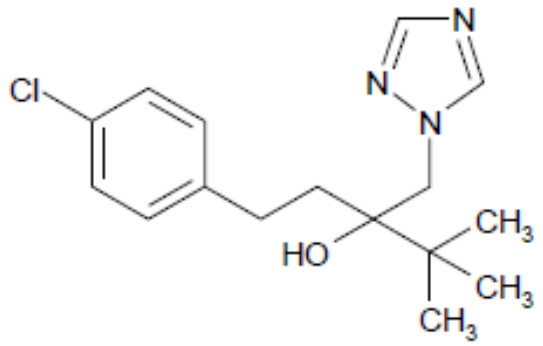
Present the outcome of the method extension to demonstrate that the CIPAC method 494 is suitable for the determination of tebuconazole in emulsifiable concentrate (EC) formulations.

The method extension was conducted by two independent laboratories.

Five batches of a tebuconazole EC formulation were selected to be used for this method extension.

Tebuconazole

Iso common name	tebuconazole
Chemical name	(RS)-1-(4-chlorophenyl)-4,4-dimethyl-3-(1H-1,2,4-triazol-1-ylmethyl)pentan-3-ol
Structural Formula	
Empirical Formula	C16 H22 Cl N3 O
Molecular Weight	307.8 g/mol
CAS No	107534-96-3

Participants, samples

Participants 1.) Dr. Michael Haustein, CURRENTA GmbH & Co. OHG, Dormagen, Germany

2.) Friedhelm Schulz Bayer CropScience AG, Monheim, Germany

Samples:

Tebuconazole, Emulsifiable Concentrate EC 250 g/Declared content: 250 g/L, 26.0 % (w/w)

Batches:
2020-002262
EDFL052343
EDFL052344
EDFL052752
EDFL053105

Determination of Repeatability:

Measurement of each individual batch in two different laboratories

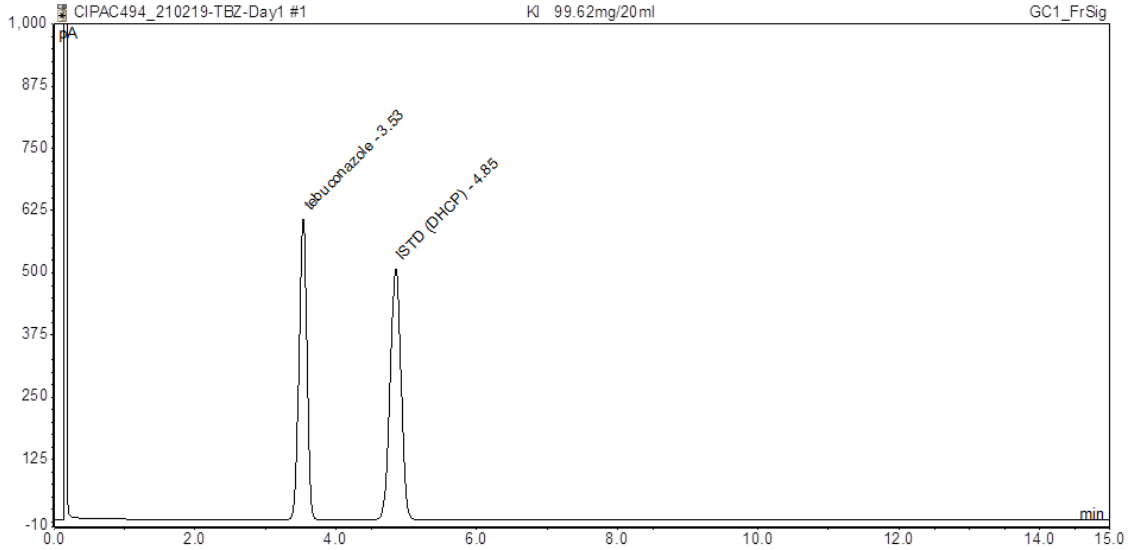
Outline of the method (CIPAC 494)

- **Operation conditions**

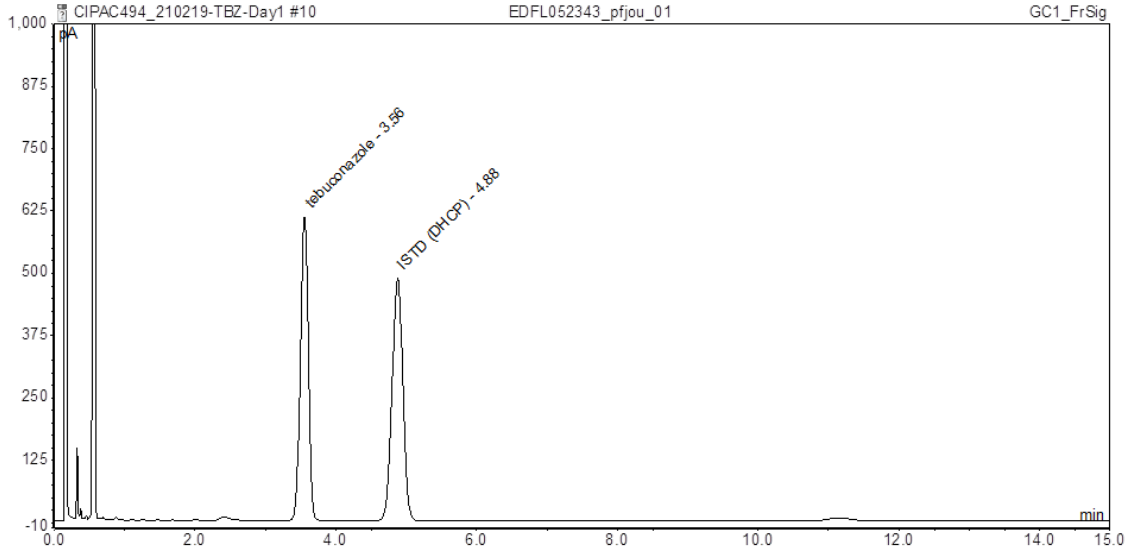
Column :	Fused silica 5 m x 0.53mm (i.d)
Column coating :	methyl silicone
Film thickness :	5 µm
Injector :	300 °C
Split ratio :	1 : 10
Injection volume :	1 µL
Oven :	240 °C
Carrier gas Helium :	approx. 7 mL/min
Temperatures Detector (FID) :	300 °C
Running time :	15 min
Retention times:	tebuconazole: about 3.5 min, dicyclohexyl phthalate about 4.8 min

Chromatograms (reference) and tebuconazole EC formulation

Reference with ISTD



Typical Chromatogram, EC formulation with ISTD



Results for tebuconazole EC 250

Analysis of five different lots in two different laboratories

Batch	2020-002262 Tebuconazole [% w/w]		EDFL052343 Tebuconazole [% w/w]		EDFL052344 Tebuconazole [% w/w]		EDFL052752 Tebuconazole [% w/w]		EDFL053105 Tebuconazole [% w/w]	
	Lab1	Lab2	Lab1	Lab2	Lab1	Lab2	Lab1	Lab2	Lab1	Lab2
	25.90	25.57	26.01	25.62	25.70	25.48	25.85	25.78	25.99	25.93
	25.80	25.61	25.95	25.61	25.43	25.39	25.88	25.78	26.23	25.89
	25.61	25.56	25.55	25.72	25.37	25.45	25.79	25.81	25.82	25.94
	25.39	25.51	25.60	25.74	25.46	25.42	25.78	25.83	25.88	25.87
				25.75						
				25.77						
Mean value	**25.68**	**25.56**	**25.78**	**25.70**	**25.49**	**25.44**	**25.83**	**25.80**	**25.98**	**25.91**
SD	0.2249	0.0400	0.2360	0.0697	0.1449	0.0391	0.0480	0.0234	0.1809	0.0350
RSD [%]	**0.88**	**0.016**	**0.92**	**0.27**	**0.57**	**0.15**	**0.19**	**0.09**	**0.70**	**0.14**

Statistical Summary - Tebuconazole, EC

	Batch 2020-002262	Batch EDFL052343	Batch EDFL052344	Batch EDFL052752	Batch EDFL053105
x [% w/w]	25.62	25.73	25.46	25.81	25.94
L	2	2	2	2	2
s_r [% w/w]	0.1615	0.1547	0.1061	0.0377	0.1303
RSD_r [%]	**0.63**	**0.60**	**0.42**	**0.15**	**0.50**
r [% w/w]	0.4522	0.4330	0.2972	0.1057	0.3649
Horwitz-value RSD $(r)_{max}$	1.64	1.64	1.65	1.64	1.64
Horrat value H_r	**0.38**	**0.37**	**0.25**	**0.09**	**0.30**

Where	
x	= average
L	= number of laboratories
s_r	= repeatability standard deviation
RSD_r	= repeatability relative standard deviation
r	= repeatability (s_r * 2.8)

Summary

- The method extension was conducted by two independent laboratories using five EC formulation batches.
- For the analysis of the EC formulation there was no need to adapt the CIPAC method or the sample preparation
- The analysis was performed in the validated linearity
- Based on the relative standard deviation results (RSD (r)) obtained for the five individual EC batches, the CIPAC method is regarded suitable for the extension to EC formulation types. The repeatability results (ranging from 0.15 to 0.63% relative) are far below the modified Horwitz criterion, the HorRat-value is <=1.

Thank you.