

Tebuconazole

CIPAC SMALL SCALE COLLABORATIVE TRIAL

JUNE 2023, GERMANY

Ms. Yue Wang

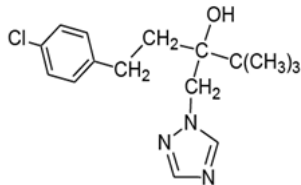
JIANGSU SEVENCONTINENT GREEN CHEMICAL CO., LTD.

Chemical Name (R,S)-1-(4-chlorophenyl-4,4-dimethyl-3-(1*H*-1,2,4-triazole-ylmethyl)pentan-1-ol (IUPAC);
 α -[2-(4-chlorophenyl)ethyl]- α -(1,1-dimethylethyl)-1*H*-1,2,4-triazole-1-ethanol (CA)

CAS No 107534-96-3

Empirical Formula

Structure



GENERAL INFORMATION



RMM	307.8
Vapor Pressure	3.1×10^{-8} mPa at 25°C
Density	1.25 (26°C)
Solubility	In water 32 g/L, (20°C). In Methanol >250g/L, (25°C) In 1,2-dichloroethane >250g/L, (25°C)
Description	White powder.

GENERAL INFORMATION



Stability	Stable to elevated temperatures, and to photolysis and hydrolysis in pure water, under sterile conditions; hydrolysis DT50 >1 y (pH 4-9, 22 °C)
Formulation	Tebuconazole Wettable Powder (WP); Emulsifiable Concentrate (EC); Suspension Concentrate (SC);

PARTICIPANTS

- * **Two technical samples ,WP, EC, SC samples were sent to the following 4 participants on October 2023**
- * **By the end of December 2023, all 4 participants provided their results to us.**

No	Responsible Person	Lab Name	City, Country
1	Ms. Hu Chunhong	Jiangsu Sevencontinent Green Chemical Co.,Ltd.	Jiangsu, China
2	Ms. Tang Huimin	Jiangsu Agro-Product Quality Test Center	Jiangsu, China
3	Ms Hua Lijuan	Nutrichem Laboratory Co., Ltd.	Beijing, China
4	Mr. Shu jun	Jiangsu Eventest Co.,Ltd	Jiangsu, China

*Outline of method

The content of Tebuconazole (g/kg) is determined by capillary gas chromatography with split injection, using dicyclohexyl phthalate as internal standard.

*Recommended Gas Chromatographic Conditions

Carrier Gas: Helium 2.0 ml/min;

Hydrogen 40 ml/min;

Air 400 ml/min;

Oven Temperature: 240°C hold 8min, ramp rate 15°C/min, to 260°C, hold 4 min;

Injector Temperature: 280°C;

Detector Temperature: 300°C ;

Split Ratio: 20:1;

Volume Injected: 0.2µl;

Retention Time: Tebuconazole about 4.8 min.; dicyclohexyl phthalate: about. 6.3min.

***Preparation of calibration solution**

Weigh in duplicate (to the nearest 0.01 mg) approximately 50mg (s in mg) of the Tebuconazole reference standard into separate suitable vessels. Add by pipette internal standard solution (10 ml). Mix thoroughly.

***Preparation of sample solution**

Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.01 mg) sufficient sample (w in mg) (containing approximately 50 mg of Tebuconazole) into separate suitable vessels. Add by pipette internal standard solution (10 ml). Mix thoroughly.

*Determination

Inject in duplicate each sample solution and bracket a series of sample solution injections by injections of the calibration solution as follows: calibration solution C1, calibration solution C2, calibration solution C1, sample solution S1, sample solution S1, calibration solution C1, sample solution S2, sample solution S2, calibration solution C1 ... (C1, C2, C1, S1, S1, C1, S2, S2, C1 ...)

Determine the peak areas of Tebuconazole and dicyclohexyl phthalate.

*Samples

Sample Name	Batch No.
Tebuconazole reference	2022FB9205
Tebuconazole Technical Material (TC-1)	20220601
Tebuconazole Technical Material (TC-2)	20220605
Tebuconazole Suspension Concentrate (SC-1)	20220704
Tebuconazole Suspension Concentrate (SC-2)	20220707
Tebuconazole Emulsifiable Concentrate (EC-1)	20220601
Tebuconazole Emulsifiable Concentrate (EC-2)	20220901
Tebuconazole Wettable Powder (WP-1)	20221011
Tebuconazole Wettable Powder (WP-2)	20221012

***Protocol**

The samples were analyzed on two different days with duplicate injections weighting per sample. Test and calibration solutions were prepared freshly on each day. The samples content was calculated using the mean value of the duplicate injections.

ANALYTICAL CONDITIONS

Conditions	Lab 1	Lab 2	Lab 3	Lab 4
GC-System	Agilent 7890B	Agilent 8890A	Agilent 7890A	Shimadzu GC-2010Plus
Column	HP-5 30 m × 0.32mm × 0.25µm	HP-5 30 m× 0.32 mm× 0.25µm	HP-5 30 m× 0.32 mm× 0.25µm	HP-5 30 m× 0.32 mm× 0.25µm
Detector:	FID	FID	FID	FID
Carrier Gas:	Helium 2.0 ml/min	Nitrogen 2.0 ml/min	Nitrogen 2.0 ml/min	Nitrogen 2.0 ml/min
Oven Temperature:	240 °Chold 8min, Ramp rate 15°C/min, to 260°C, hold 4 min	240 °Chold 8min, Ramp rate 15°C/min, to 260°C, hold 4 min	240 °Chold 8min, Ramp rate 15°C/min, to 260°C, hold 4 min	240 °Chold 8min, Ramp rate 15°C/min, to 260°C, hold 4 min
Injector Temperature:	280°C	280°C	280°C	280°C
Detector Temperature:	300°C	300°C	300°C	300°C
Split Ratio:	20:1	20:1	20:1	20:1
Volume Injected:	0.2µl	0.2µl	0.2µl	0.2µl

RESULTS AND REVIEW

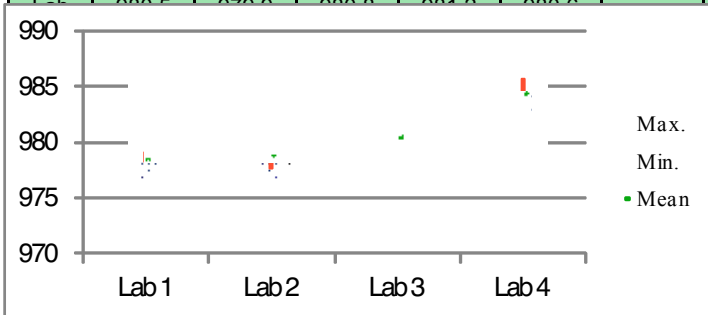
The full results of four labs were included within the statistical assessment. The statistical evaluation of the data was accomplished following the “Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Method” according to DIN ISO 5725.

The assay results obtained by the collaborators and the statistical evaluation are reported in the following tables listed in the next several slides.

ORIGINAL DATA (G/KG)

TC-1							
Lab	Day 1		Day 2		Mean g/kg	Si	Si2
	1	2	1	2			
Lab 1	978.45	979.08	978.95	978.18	978.67	0.42	0.18
Lab 2	977.70	978.85	979.20	980.40	979.04	1.11	1.24
Lab 3	980.50	979.90	980.90	981.90	980.60	0.32	0.92

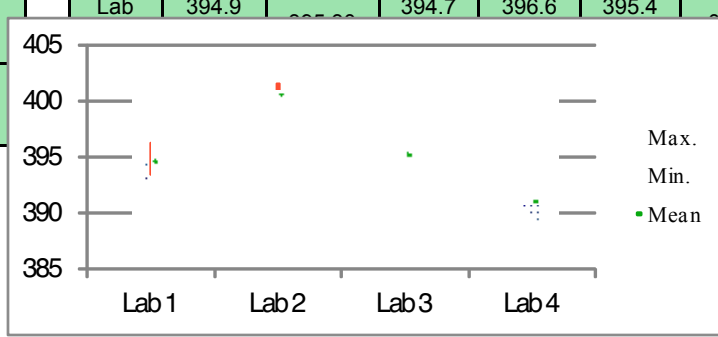
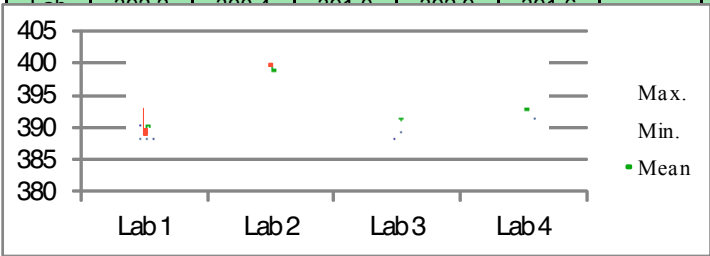
TC-2							
Lab	Day 1		Day 2		Mean g/kg	Si	
	1	2	1	2			
Lab 1	979.02	981.46	980.58	980.33	980.35	1.01	
Lab 2	979.95	977.85	978.20	977.55	978.39	1.08	
Lab 3	980.10	980.00	982.70	981.80	982.10	1.58	1.52



ORIGINAL DATA (G/KG)

SC-1							
Lab	Day 1		Day 2		Mean g/kg	Si	Si2
	1	2	1	2			
Lab 1	393.03	388.77	390.77	390.34	390.73	1.76	3.10
Lab 2	399.40	399.90	398.95	398.25	399.13	0.70	0.49
Lab 3	399.90	399.40	394.90	399.90	394.90	1.30	0.02

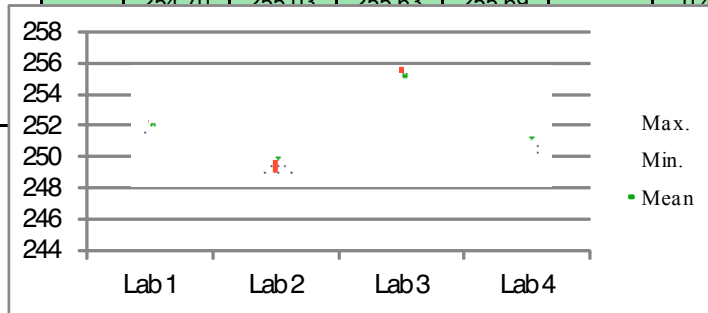
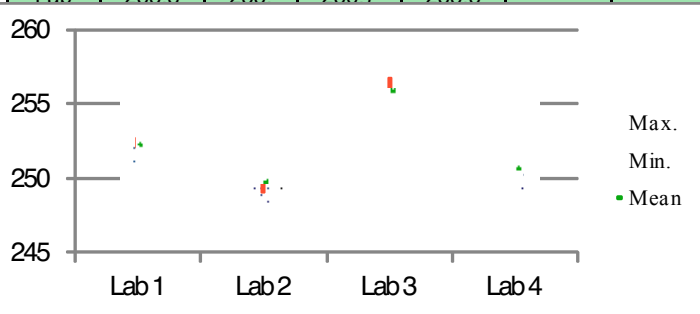
SC-2						
Lab	Day 1		Day 2		Mean g/kg	Si
	1	2	1	2		
Lab 1	394.70	394.90	396.19	393.35	394.79	1.16
Lab 2	398.55	401.45	401.60	401.20	400.70	1.44
Lab 3	394.90	399.90	394.70	396.60	395.40	0.87
Lab 4	394.90	399.90	394.70	396.60	395.40	0.19



ORIGINAL DATA (G/KG)

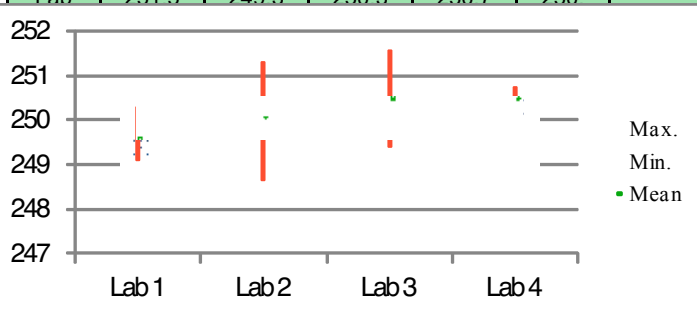
EC-1							
Lab	Day 1		Day 2		Mean g/kg	Si	Si2
	1	2	1	2			
Lab 1	252.16	252.07	252.06	252.56	252.37	0.31	0.10
Lab 2	250.75	250.65	249.40	249.05	249.96	0.86	0.75
Lab	255.8	255	256.7	255.5			

EC-2							
Lab	Day 1		Day 2		Mean g/kg	Si	
	1	2	1	2			
Lab 1	252.17	252.33	252.05	252.21	252.19	0.12	
Lab 2	251.05	250.00	249.05	250.30	250.10	0.83	
Lab	254.70	255.03	255.63	255.69	255.2	0.48	

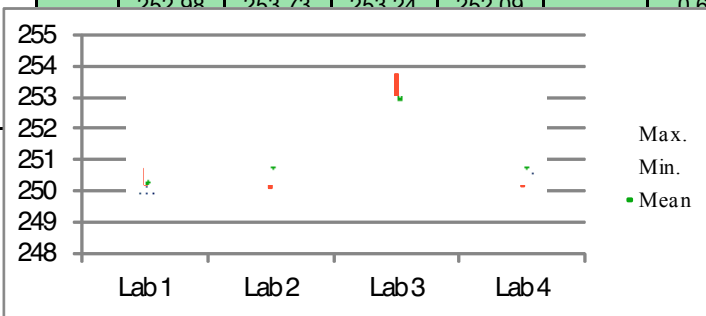


ORIGINAL DATA (G/KG)

WP-1							
Lab	Day 1		Day 2		Mean g/kg	Si	Si2
	1	2	1	2			
Lab 1	250.3 1	249.0 9	249.6 6	249.6 2	249. 67	0.50	0.25
Lab 2	249.7 5	248.6 5	250.7 5	251.3 0	250. 11	1.17	1.36
Lab	251.5	249.3	250.3	250.7	250.		
						0.82	
						0.04	



WP-2							
Lab	Day 1		Day 2		Mean g/kg	Si	
	1	2	1	2			
Lab 1	250.17	250.38	250.72	250.22	250.37	0.25	
Lab 2	250.10	250.80	251.60	250.80	250.83	0.61	
Lab	252.98	253.73	253.24	252.09	253.0	0.69	
							2



SUMMARY



Summary of the statistical evaluation no elimination of any outliers

	TC-1	TC-2	SC-1	SC-2	EC-1	EC-2	WP-1	WP-2	Xm	average
Xm	980.71	981.04	252.29	252.23	393.67	395.53	250.20	251.26	L	Number of laboratories
L	4	4	4	4	4	4	4	4	Sr	Repeatability standard deviation
Sr	0.81	1.66	0.54	0.49	1.11	1.03	0.79	0.60	SL	Pure between laboratory standard deviation
SL	2.62	1.96	2.63	2.18	3.73	3.87	0.06	1.15	SR	Reproducibility standard deviation
SR	2.75	2.57	2.68	2.24	3.90	4.01	0.79	1.30	RSDr	Repeatability relative standard deviation
r	2.28	4.64	1.51	1.39	3.10	2.88	2.20	1.67	RSDR	Reproducibility relative standard deviation
R	7.69	7.20	7.51	6.26	10.91	11.22	2.21	3.63	r	Repeatability
RSDr	0.08	0.17	0.21	0.20	0.28	0.26	0.31	0.24	R	Reproducibility
RSDR	0.28	0.26	1.06	0.89	0.99	1.01	0.32	0.52	RSD (Hor)	Horwitz value calculated from 2

CONCLUSION AND SUGGESTION



For all samples, the values of RSDR (Reproducibility relative standard deviation) were less than Horwitz's value. As a reference, all HorRat values were not greater than 1.0. The proposed method is considered to be appropriate for the determination of Tebuconazole in Technical material; Emulsifiable Concentrate; Suspension Concentrate; Wettable Powder.

We propose to proceed with a large scale collaborative study.

Thanks for your attention

JIANGSU SEVENCONTINENT GREEN CHEMICAL CO., LTD.