PROTHIOCONAZOLE

Collaborative Study

Full Scale Collaborative Study
for the
Determination of Prothioconazole formulations by
High Performance Liquid Chromatography

Report to CIPAC
by
Bayer AG, Crop Science Division
Friedhelm Schulz
Alfred-Nobel-Str. 50
40789 Monheim
Germany

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1. Participants

In September 2017, Information Sheet No. 311 was sent out by the CIPAC Secretary, inviting members to participate in a collaborative study on the determination of prothioconazole by gradient reversed phase High Performance Liquid Chromatography.
By end of March 2018, 20 of the 25 respondents provided their results. Of the remaining 20 laboratories, only 12 participants used the column material described in the CIPAC collaboration trial, 8 laboratories used a different column material.
The results for Prothioconazole FS 258 of one participant were rejected due to an operational error occurred.
The results of all 20 participants were presented and evaluated.

The participating 20 laboratories are listed in alphabetical order, whereas lab numbers in the result tables were assigned, chronologically, based upon receipt of results.

Agence Fédérale pour la Sécurité Alimentaire Rue de Visé, 495 4020 Wandre 04/252.01.58 Belgium
Agroscope Pflanzenschutzchemie Schloss 1 CH-8820 Wädenswil Switzerland
Bayer AG Crop Science Division CS-RD-PD-FT Building 6820 40789 Monheim, Germany
Bayer AG Research & Development, Crop Science Product Chemistry Analytics 3 Building 6530 40789 Monheim, Germany
Bundesamt für Verbraucherschutz und Lebensmittelsicherheit Dept. Plant Protection Products Messeweg 11/12 D-38104 Braunschweig Germany
Currenta GmbH & Co. OHG SEL-ANT-PDA CHEMPARK Dormagen, A559 Germany
Development Laboratory Cerexagri BV Tankhoofd 10/ Harbour no.: 3255 3196 KE Vondelingenplaat/Rt The Netherlands
FRANDESA Co.,Ltd. 1, Bereza district 225209 Brest Region Republic Belarus

Istituto Superiore di Sanità Dipartimento Ambiente e Salute Laboratorio Nazionale di Riferimento per i residui di pesticidi con metodi mono residuo V.le Regina Elena, 299 – 00161 Roma
Jl. Melati Desa Wanaherang Kec. Gunung Putri Cibinong 16965, West Java, Indonesia
Maryland Department of Agriculture 50 Harry S Truman Parkway Annapolis, MD 21401 USA
Ministerio de Desarrollo Agropecuario Dirección Nacional de Sanidad Vegetal Laboratorio de Control de Calidad de Plaguicidas Río Tapia Vía Tocumen, Entrando por Harinas del Istmo 1.5 km Panamá, City
Monsanto Haven 627 Scheldelaan 460 2040 Antwerp Belgium
National Food Chain Safety Office Directorate of Plant Protection, Soil Conservation and Agri-environment Pesticide Analytical Laboratory, Velence H-2481 Velence, Ország út 23. Hungary
National Phytosanitary Autority 11 Voluntari Bvld. 077190, Voluntari, Romania
Pesticide Quality System Development Sub-Group Agricultural Production Science Research and Development Division Department of Agriculture Thailand
Syngenta Crop Protection AG 3120.E74 Breitenloh 5 4333 Münchwilen Switzerland
The Pesticide Control Laboratory, Backweston Laboratory Complex, Backweston, Celbridge, Co. Kildare, Ireland
UKZUZ (Central Institute for Supervising and Testing in Agriculture) National Reference Laboratory Department of Testing Plant Protection Products Zemědělská 1a, 613 00 Brno Czech Republic
Walloon Agricultural Research Centre (CRA-W) Agriculture and Natural Environment Department (D3) Plant Protection Products and Biocides Physico-chemistry and Residues Unit (U10) Carson Building Rue du Bordia, 11 B-5030 Gembloux Belgium

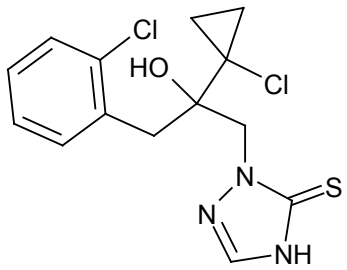
2. Active Ingredient: General Information

Chemical name:	IUPAC	(RS)-2-[2-(1-chlorocyclopropyl)-3-(2-chlorophenyl)-2-hydroxypropyl]-2,4-dihydro-1,2,4-triazole-3-thione
	CAS	3H-1,2,4-Triazole-3-thione, 2-[2-(1-chlorocyclopropyl)-3-(2-chlorophenyl)-2- hydroxypropyl]-2,4-dihydro-

ISO common name: Prothioconazole

CAS-No.: 178928-70-6

Structure:



Molecular mass: 344.3 g/mol

Empirical formula: C14 H15 Cl2 N3 O S

Activity: Fungicide

3. Samples

Five test samples, one analytical standard and one stabilizer were sent to the participants:

1. Prothioconazole tech. sample
2. Prothioconazole EC 250 G
3. Prothioconazole FS 100 G
4. Prothioconazole FS 258 G (8 g/L Prothioconazole)
5. Prothioconazole SC 480 G

Prothioconazole, reference standard purity 99.8 %w/w)
Stabilizer: L-cysteine hydrochloride monohydrate

4. Method

4.1. Scope

The determination of prothioconazole active ingredient content contained within a technical sample (TC) and EC, FS, and SC formulations.

4.2. Principle

The content of prothioconazole (g/kg) is determined by reversed phase high performance liquid chromatography using UV detection at 254 nm and external standard calibration.

4.3. Procedure

Each sample was analyzed using four independent determinations. The samples were analyzed on two different days, each day involving duplicate injections of duplicate weights. Both test and reference solutions were freshly prepared on each day. The four injections of each test solution were bracketed by single injections of the calibration solution. The average response factor, used to calculate the amount of prothioconazole in the test solution, was calculated using the injection before and after the test injections.

5. Remarks of the Participants

Several participants provided comments about the method performance and also made a note of any deviations from the method:

Laboratory 1	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	Filtration instead of centrifugation
Laboratory 2	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	None
Laboratory 3	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	Injection volume: 5µl instead of 3 µL All samples filtrated through a 0.2µm RC filter Retention time of prothioconazole is higher (2.5 min) than reported in the method. At 2.7 min there is a shift in the baseline due to flow change. This results in problems with the integration of the peak. Therefore a blank chromatogram of the respective sequence was substracted from all other chromatograms.
Laboratory 4	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	None
Laboratory 5	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	We have prepared a solution of cysteine: 1.25g/25mL water and added 100 µL to each sample solution instead of weighing 5 mg . We have used THF Emsure instead of HPLC grade. We have filtrated the FS samples, because after centrifugation, the samples were not clear yet. The solutions of the FS formulations are difficult to make up to the mark because the dye is separating from the solution. After addition of water and before making up to the mark, we used brief ultrasonic stirring, to remove air from the solutions (otherwise the stop can fly off).
Laboratory 6	Column:	Zorbax XDB C18 (250 x 3.0 mm, 5.0 µm)
	Remarks:	Flow Rate reduced to 1mL/min Injection volume increased to 5 µL
Laboratory 7	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	We kept a flow rate of 2mL/min throughout the gradient (maximum possible flow on the UPLC)

Laboratory 8	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	In absence of a centrifuge, the samples were filtered through Chromafil disposable filters 0.2µm
Laboratory 9	Column:	Eclipse Plus phenyl-hexyl (100 x 4.6 mm, 3.5 µm)
	Remarks:	We increased the injection volume to 10µL to obtain good injection reproducibility. Injection of 3µL and even 5µL resulted in a poor injection reproducibility (approx. 5%). This should be due to our system, however it was observed on 2 (identical) instruments. The larger injection volume does not appear to be a problem, since the signal is still in the linear part of the detector.
Laboratory 10	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	Prothioconazole 745 test method is a rugged one. The method recommended 3µL, I had to increase to 5µL injection volume to significantly improve signal to noise ratio for low concentration of the analyte in the sample matrix, i.e. FS1 258 G
Laboratory 11	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	In the beginning of the gradient (approx. at 3 min) we observed a higher pressure (approx. 4400 psi)
Laboratory 12	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	Samples requiring centrifugation were filtered through 0.2 µm minisart filter and were not centrifuged.
Laboratory 13	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3 µm)
	Remarks:	Results for 8 g/L were divided by 10.
Laboratory 14	Column:	Zorbax Eclipse XDB-C18 (50 x 4.6 mm, 1.8 µm)
	Remarks:	Sample solutions S5, S6 and S9, S10 were filtrated
Laboratory 15	Column:	Poroshell 120 EC C18, 50 x 4.6 mm, 2.7 µm)
	Remarks:	Too high flow rate and as a result high column pressure. It leads to decrease in the life of the column. For completely to dissolve prothioconazole samples, the time of 3-5 minutes is totally enough. L-cysteine could not be dissolved in pure acetonitrile. Thus in accordance with the method we have to suspend, or to dissolve in acetonitrile? Perhaps it could be better to add aliquot of L-cysteine prepared solution to each flask? There is a discrepancy between the sequence described in Method (part (e) Determination and information in Excel data-sheet.

Laboratory 16	Column:	BEH C18 (50 x 2.1 mm, 1.7 µm)
	Remarks:	I worked in UPLC and thus adapted the flow (to 0.42mL/min) while keeping the equivalence of the gradient of the proposed method. Injection volume: 1 µL
Laboratory 17	Column:	Poroshell 120 EC C18 (50 x 3.0 mm, 2.7 µm)
	Remarks:	The flow was changed from 2.0 mL/min to 1.0 mL/min
Laboratory 18	Column:	Zorbax Extend C18 (50 x 4.6 mm, 3.5 µm)
	Remarks:	None
Laboratory 19	Column:	ODS Hypersil (150 x 4.6 mm, 5µm)
	Remarks:	We used column a from Thermo Scientific, which differs from reference method, but both columns have the same content, only different length and particle size.
Laboratory 20	Column:	Zorbax XDB C18 (75 x 4.6 mm, 3.5 µm)
	Remarks:	S9 and S10, Flowable Concentrate for Seed Treatment FS with Prothioconazole below 1% w/w, were prepared by calculating the concentration from 0.05 mg/mL

6. Evaluation and Discussion

6.1. Data Review

The data obtained from each laboratory was visually reviewed to determine if there were any significant chromatography differences, from what was expected, which might affect the analytical results.

In summary it can be stated that the method deviations, noted by the participating 20 participants, were deemed not to affect the analytical results significantly and therefore all data sets were included within the statistical assessment.

6.2. Determination of Prothioconazole

The statistical evaluation of the data was accomplished following the “Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods”, according to DIN ISO 5725. The testing for outliers/stragglers of the laboratory mean values were performed according to Grubbs test on a 1%/5% significance level, respectively. All results reported by the 20 laboratories are reported and the statistical evaluation of these are listed in Tables 1-2 and displayed in Figures 1-5. These results are reported without any exclusion of outliers and/or stragglers.

In addition, a separate evaluation, listed in Table 3-4 and Figures 6-10, display the results with the exclusion of outliers.

Determination of Prothioconazole – Full set of 20 participants

All results tabulated in table 1 are given in g/kg.

Table 1 - Results

Laboratory	Day	Prothioconazole Tech.	Ø	Prothioconazole EC 250 g/L	Ø	Prothioconazole FS 100 g/L	Ø	Prothioconazole SC 480 g/L	Ø	Prothioconazole FS 258 g/L (8 g/L Prothioconazole)	Ø
1	1	991	991.00	253.2	252.80	86.0	86.00	405	404.00	7.21	7.415
	2	991		252.4		86.0		403		7.62	
2	1	989	990.50	248.8	249.75	83.4	83.65	395	397.00	6.95	6.960
	2	992		250.7		83.9		399		6.97	
3	1	991	987.50	251.6	251.40	86.2	85.70	407	407.50	7.32	7.225
	2	984		251.2		85.2		408		7.13	
4	1	988	989.50	253.0	253.50	85.9	85.65	403	403.50	7.06	7.070
	2	991		254.0		85.4		404		7.08	
5	1	996	988.50	246.5	248.95	83.2	82.90	402	400.50	6.81	6.795
	2	981		251.4		82.6		399		6.78	

Table 1 – Results continued

Laboratory	Day	Prothioconazole Tech.	Ø	Prothioconazole EC 250 g/L	Ø	Prothioconazole FS 100 g/L	Ø	Prothioconazole SC 480 g/L	Ø	Prothioconazole FS 258 g/L (8 g/L Prothioconazole)	Ø
6	1	980	982.00	250.9	251.00	85.1	85.30	404	404.50	7.09	7.100
	2	984		251.1		85.5		405		7.11	
7	1	991	987.50	247.2	249.45	84.4	85.20	400	403.50	7.04	7.035
	2	984		251.7		86.0		407		7.03	
8	1	986	988.00	248.6	250.45	84.3	84.45	405	404.50	7.07	7.065
	2	990		252.3		84.6		404		7.06	
9	1	988	987.50	251.4	250.65	83.6	83.75	394.5	397.00	6.99	6.990
	2	987		249.9		83.9		399.5		6.99	
10	1	989	988.50	258.7	258.65	88.7	88.75	419	422.00	7.27	7.395
	2	988		258.6		88.8		425		7.52	
11	1	985	985.00	249.1	250.25	83.7	84.10	403	403.00	6.97	7.030
	2	985		251.4		84.5		403		7.09	
12	1	1002	1005.50	255.9	256.60	86.8	87.30	410	414.00	7.16	7.175
	2	1009		257.3		87.8		418		7.19	
13	1	987	986.50	250.5	251.85	85.2	84.85	405	407.00	6.98	6.965
	2	986		253.2		84.5		409		6.95	
14	1	984	985.50	252.8	253.00	85.6	85.40	406	404.50	7.03	7.045
	2	987		253.2		85.2		403		7.06	
15	1	995	993.50	254.9	254.65	87.6	87.10	418	411.50	7.32	7.245
	2	992		254.4		86.6		405		7.17	
16	1	994	994.50	255.4	257.45	85.6	85.75	407	409.00	7.17	7.155
	2	995		259.5		85.9		411		7.14	
17	1	974	977.00	255.7	252.30	97.6	96.40[1]	482	477.00[1]	7.35	7.465
	2	980		248.9		95.2		472		7.58	
18	1	987	978.00	253.7	250.60	90.5	86.85	420	413.00	7.02	7.875[1]
	2	969		247.5		83.2		406		8.73	
19	1	974	980.50	248.6	248.70	84.7	85.05	405	403.50	7.32	7.085
	2	987		248.8		85.4		402		6.85	
20	1	1014	1007.50	260.8	258.75	89.6	86.50	415	415.50	n.u.[2]	n.u.[2]
	2	1001		256.7		83.4		416		n.u.[2]	

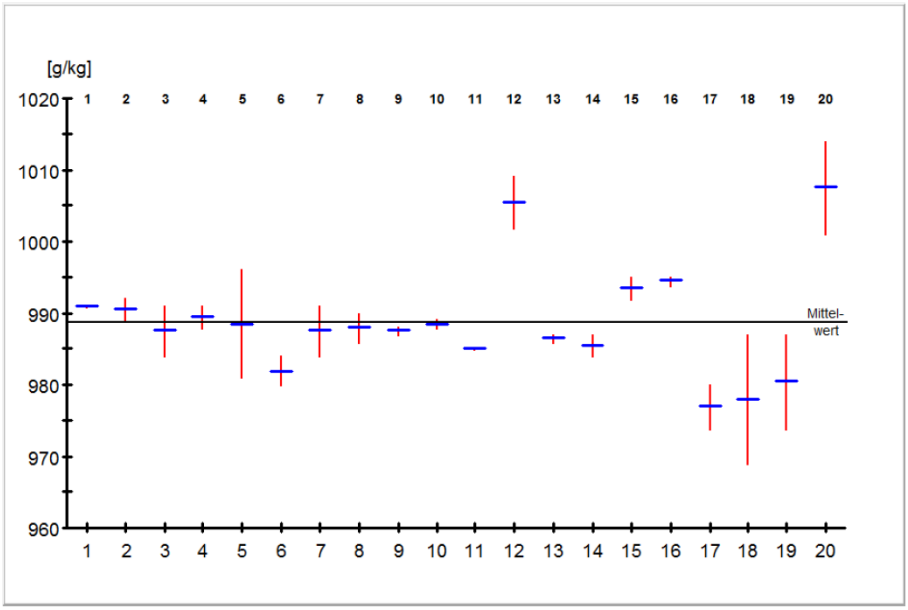
[1] Result was detected as an outlier
[2] Result is not usable

Table 2 – Summary of the statistical evaluation

	Prothioconazole Tech.	Prothioconazole EC 250 g/L	Prothioconazole FS 100 g/L	Prothioconazole SC 480 g/L	Prothioconazole FS 258 g/L (8 g/L Prothioconazole)
x_m [g/kg]	988.7	252.54	86.03	410.1	7.163
x_m [% w/w]	98.87	25.254	8.603	41.01	0.7163
L	20	20	20	20	19
s_r	5.34	2.23	1.63	4.28	0.304
s_R	8.49	3.52	3.03	17.19	0.323
r	14.96	6.24	4.56	11.99	0.851
R	23.78	9.86	8.50	48.12	0.905
RSD_R	0.86	1.39	3.53	4.19	4.511
$RSD_{R\ (Hor)}$	2.00	2.46	2.89	2.29	4.21
HorRat	0.43	0.56	1.22	1.83	1.07

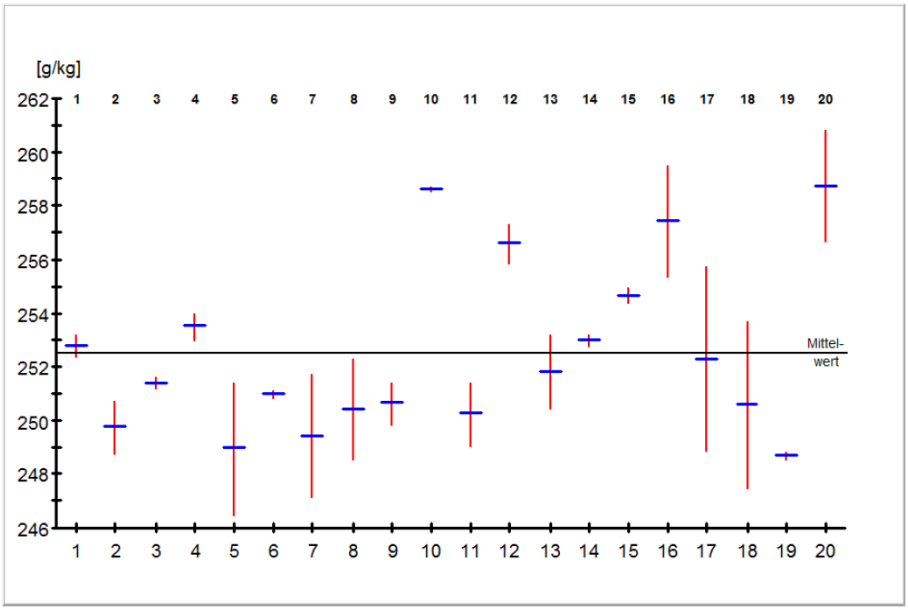
x_m = overall sample mean
L = number of laboratories
s_r = repeatability standard deviation
s_R = reproducibility standard deviation
r = repeatability limit
R = reproducibility limit
RSD_r = relative repeatability standard deviation
RSD_R = relative reproducibility standard deviation
HorRat (Horwitz Ratio) = RSDR/RSDR (Hor)

Fig. 1 – Prothioconazole tech. sample



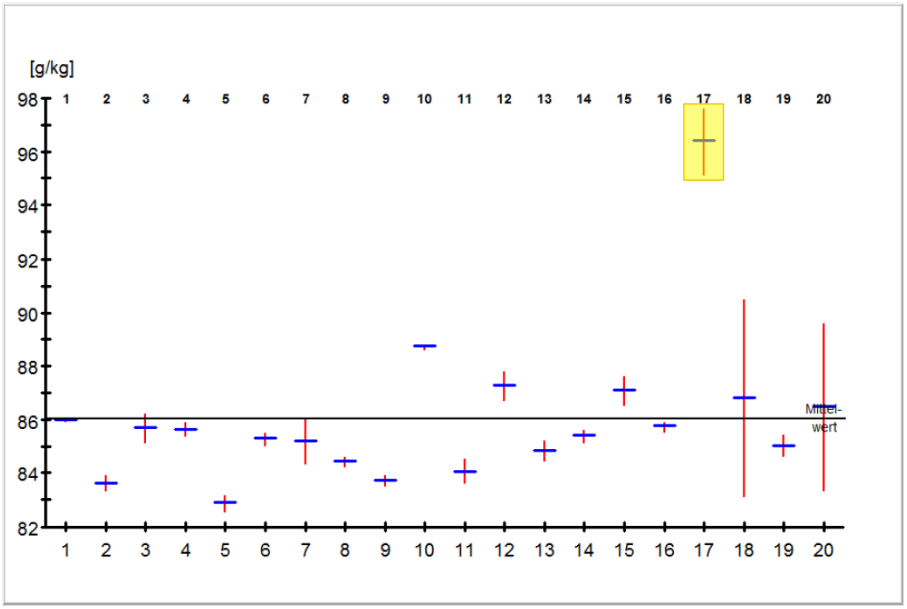
Mean value:	988.7 g/kg
s_r:	5.34
s_R:	8.49
RSD_R:	0.86
$RSD_{R\ (Hor)}$:	2.00
HorRat:	0.43
Outlier (Grubbs):	none

Fig. 2 – Prothioconazole EC 250 g/L



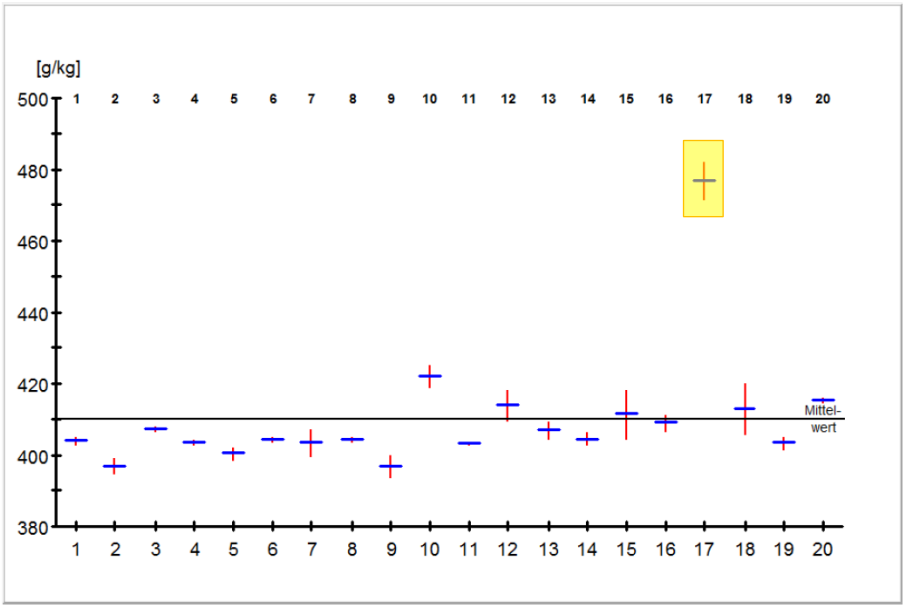
Mean value:	252.54 g/kg
s_r:	2.23
s_R:	3.52
RSD_R:	1.39
$RSD_{R\ (Hor)}$:	2.46
HorRat:	0.56
Outlier (Grubbs):	none

Fig. 3 – Prothioconazole FS 100 g/L



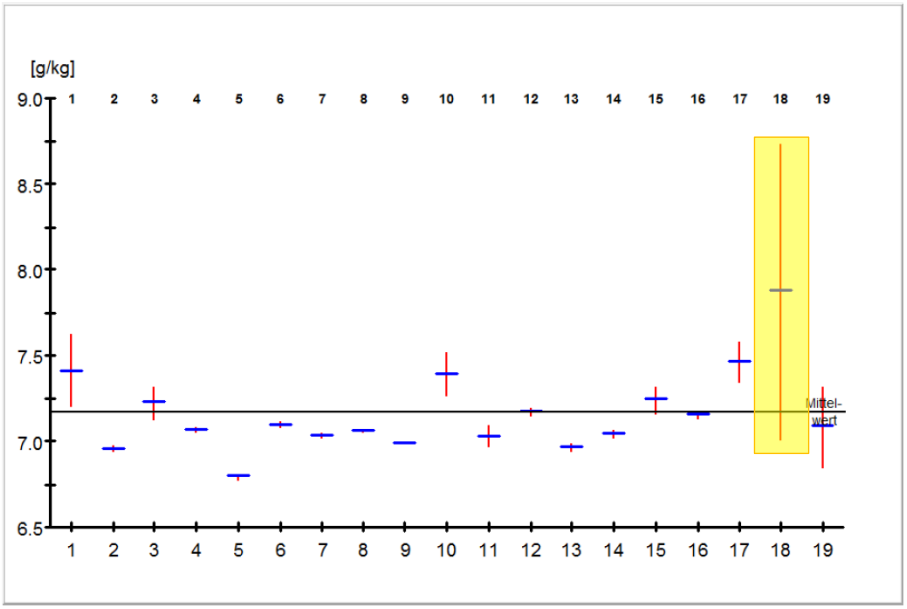
Mean value:	86.03 g/kg
s_r:	1.63
s_R:	3.03
RSD_R:	3.53
$RSD_{R\ (Hor)}$:	2.89
HorRat:	1.22
Outlier (Grubbs):	upper

Fig. 4 – Prothioconazole SC 480 g/L



Mean value:	410.10 g/kg
s_r:	4.28
s_R:	17.19
RSD_R:	4.19
$RSD_{R\ (Hor)}$:	2.29
HorRat:	1.83
Outlier (Grubbs):	upper

Fig. 5 – Prothioconazole FS 258 (8 g/L Prothioconazole)



Mean value:	7.163 g/kg
s_r:	0.304
s_R:	0.323
RSD_R:	4.511
$RSD_{R\ (Hor)}$:	4.21
HorRat:	1.07
Outlier (Grubbs):	upper

Determination of Prothioconazole – Elimination of outliers

All results tabulated in table 3 are given in g/kg.

Table3 – Results without outliers

Laboratory	Day	Prothioconazole Tech.	Ø	Prothioconazole EC 250 g/L	Ø	Prothioconazole FS 100 g/L	Ø	Prothioconazole SC 480 g/L	Ø	Prothioconazole FS 258 g/L (8 g/L Prothioconazole)	Ø
1	1	991	991.00	253.2	252.80	86.0	86.00	405	404.00	7.21	7.415
	2	991		252.4		86.0		403		7.62	
2	1	989	990.50	248.8	249.75	83.4	83.65	395	397.00	6.95	6.960
	2	992		250.7		83.9		399		6.97	
3	1	991	987.50	251.6	251.40	86.2	85.70	407	407.50	7.32	7.225
	2	984		251.2		85.2		408		7.13	
4	1	988	989.50	253.0	253.50	85.9	85.65	403	403.50	7.06	7.070
	2	991		254.0		85.4		404		7.08	
5	1	996	988.50	246.5	248.95	83.2	82.90	402	400.50	6.81	6.795
	2	981		251.4		82.6		399		6.78	

Table 3 – Results without outliers continued

Laboratory	Day	Prothioconazole Tech.	Ø	Prothioconazole EC 250 g/L	Ø	Prothioconazole FS 100 g/L	Ø	Prothioconazole SC 480 g/L	Ø	Prothioconazole FS 258 g/L (8 g/L Prothioconazole)	Ø
6	1	980	982.00	250.9	251.00	85.1	85.30	404	404.50	7.09	7.100
	2	984		251.1		85.5		405		7.11	
7	1	991	987.50	247.2	249.45	84.4	85.20	400	403.50	7.04	7.035
	2	984		251.7		86.0		407		7.03	
8	1	986	988.00	248.6	250.45	84.3	84.45	405	404.50	7.07	7.065
	2	990		252.3		84.6		404		7.06	
9	1	988	987.50	251.4	250.65	83.6	83.75	394.5	397.00	6.99	6.990
	2	987		249.9		83.9		399.5		6.99	
10	1	989	988.50	258.7	258.65	88.7	88.75	419	422.00	7.27	7.395
	2	988		258.6		88.8		425		7.52	
11	1	985	985.00	249.1	250.25	83.7	84.10	403	403.00	6.97	7.030
	2	985		251.4		84.5		403		7.09	
12	1	1002	1005.50	255.9	256.60	86.8	87.30	410	414.00	7.16	7.175
	2	1009		257.3		87.8		418		7.19	
13	1	987	986.50	250.5	251.85	85.2	84.85	405	407.00	6.98	6.965
	2	986		253.2		84.5		409		6.95	
14	1	984	985.50	252.8	253.00	85.6	85.40	406	404.50	7.03	7.045
	2	987		253.2		85.2		403		7.06	
15	1	995	993.50	254.9	254.65	87.6	87.10	418	411.50	7.32	7.245
	2	992		254.4		86.6		405		7.17	
16	1	994	994.50	255.4	257.45	85.6	85.75	407	409.00	7.17	7.155
	2	995		259.5		85.9		411		7.14	
17	1	974	977.00	255.7	252.30	~~97.6~~	~~96.40~~[1]	~~482~~	~~477.00~~[1]	7.35	7.465
	2	980		248.9		~~95.2~~		~~472~~		7.58	
18	1	987	978.00	253.7	250.60	90.5	86.85	420	413.00	~~7.02~~	~~7.875~~[1]
	2	969		247.5		83.2		406		~~8.73~~	
19	1	974	980.50	248.6	248.70	84.7	85.05	405	403.50	7.32	7.085
	2	987		248.8		85.4		402		6.85	
20	1	1014	1007.50	260.8	258.75	89.6	86.50	415	415.50	n.u.[2]	n.u.[2]
	2	1001		256.7		83.4		416		n.u.[2]	

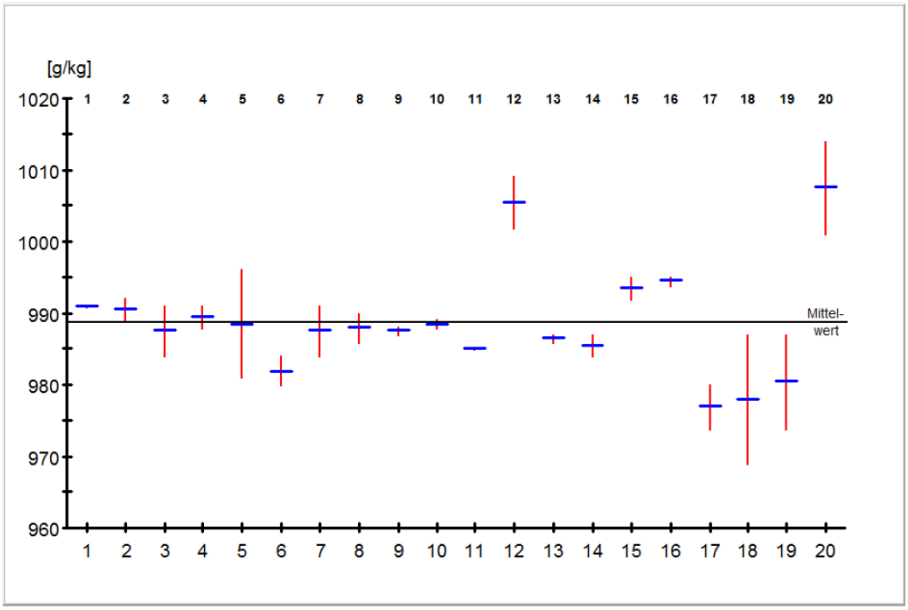
[1] Result was detected as an outlier
[2] Result is not usable

Table 4 – Summary of the statistical evaluation without outliers

	Prothioconazole Tech.	Prothioconazole EC 250 g/L	Prothioconazole FS 100 g/L	Prothioconazole SC 480 g/L	Prothioconazole FS 258 g/L (8 g/L Prothioconazole)
x_m [g/kg]	988.7	252.54	85.49	406.6	7.123
x_m [% w/w]	98.87	25.254	8.549	40.66	0.7123
L	20	20	19	19	18
s_r	5.34	2.23	1.63	4.10	0.127
s_R	8.49	3.52	1.83	7.00	0.196
r	14.96	6.24	4.55	11.4	0.356
R	23.78	9.86	5.13	19.5	0.548
RSD_R	0.86	1.39	2.14	1.72	2.75
$RSD_{R\ (Hor)}$	2.00	2.46	2.90	2.29	4.21
HorRat	0.43	0.56	0.73	0.75	0.65

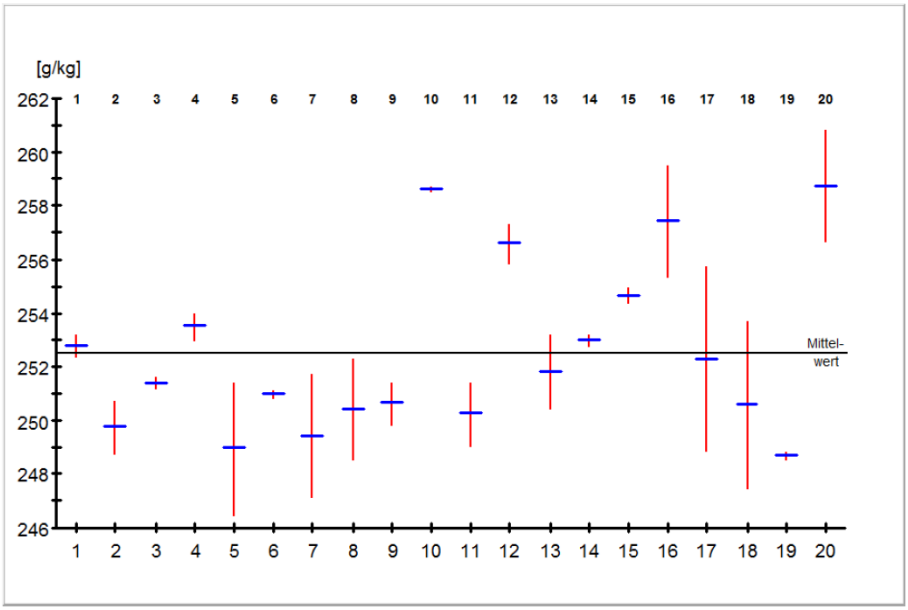
x_m = overall sample mean
L = number of laboratories
s_r = repeatability standard deviation
s_R = reproducibility standard deviation
r = repeatability limit
R = reproducibility limit
RSD_r = relative repeatability standard deviation
RSD_R = relative reproducibility standard deviation
HorRat (Horwitz Ratio) = RSDR/RSDR (Hor)

Fig. 6 – Prothioconazole tech. sample (free of outliers)



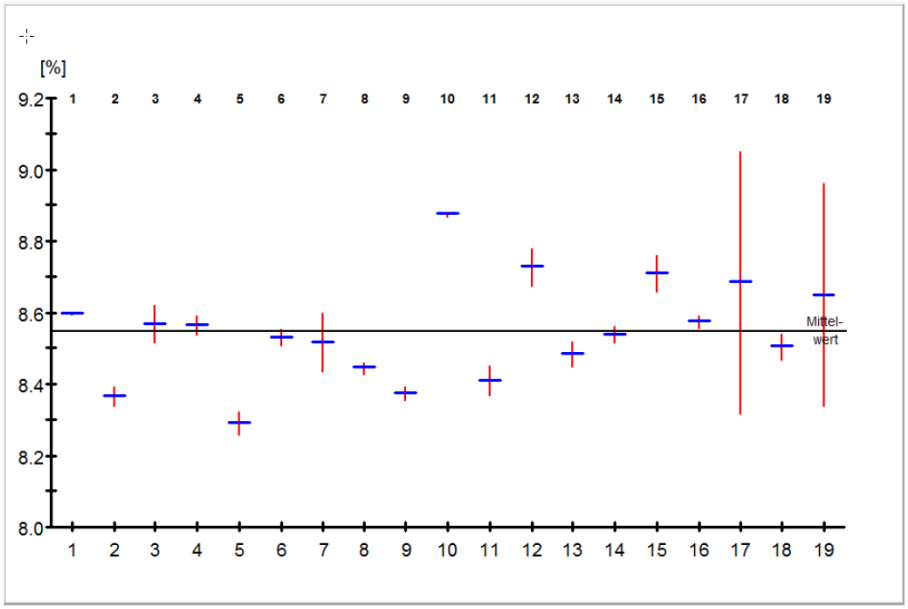
Mean value:	988.7 g/kg
s_r:	5.34
s_R:	8.49
RSD_R:	0.86
$RSD_{R\ (Hor)}$:	2.00
HorRat:	0.43
Outlier (Grubbs):	none

Fig. 7 – Prothioconazole EC 250 g/L (free of outliers)



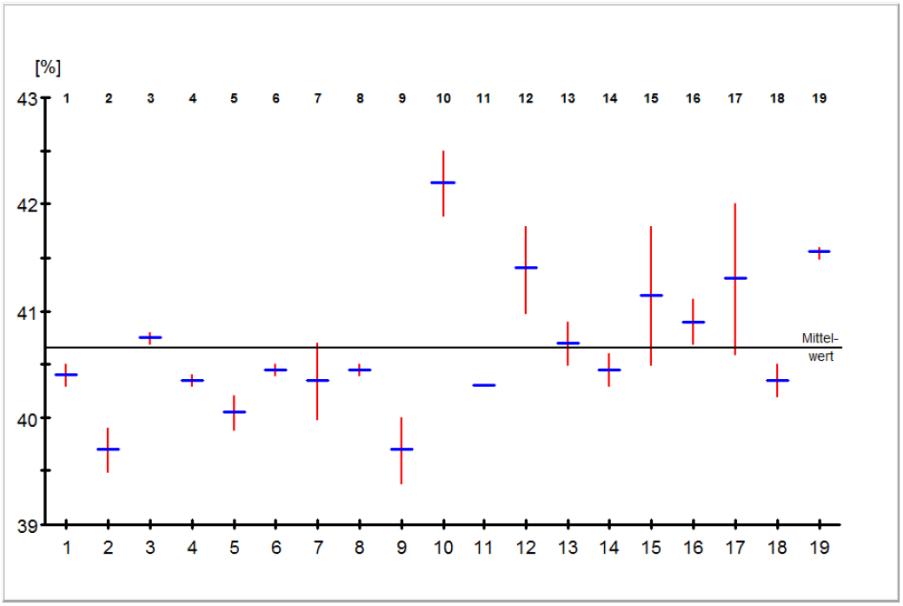
Mean value:	252.54 g/kg
s_r:	2.23
s_R:	3.52
RSD_R:	1.39
$RSD_{R\ (Hor)}$:	2.46
HorRat:	0.56
Outlier (Grubbs):	none

Fig. 8 – Prothioconazole FS 100 g/L (free of outliers)



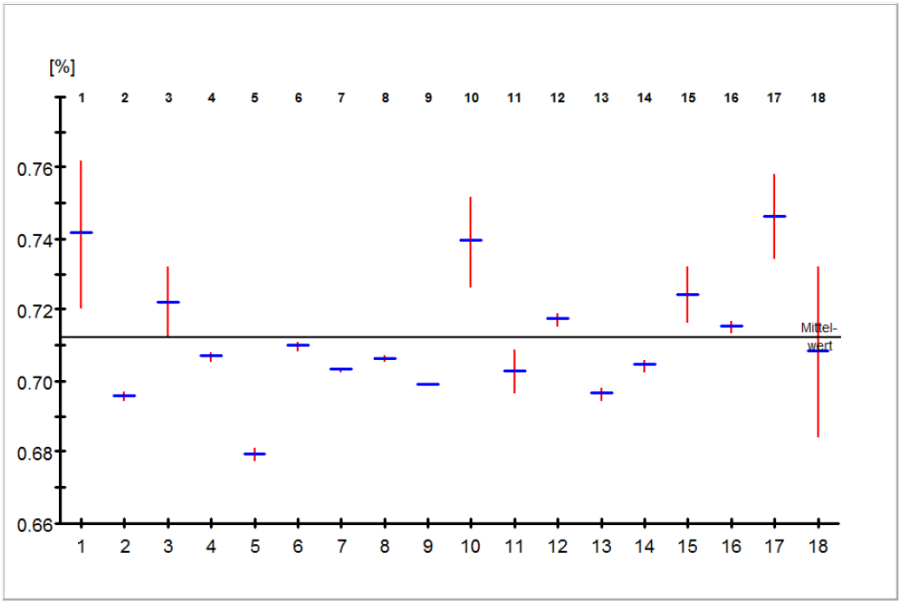
Mean value:	85.49 g/kg
s_r:	1.63
s_R:	1.83
RSD_R:	2.14
$RSD_{R\ (Hor)}$:	2.90
HorRat:	0.74
Outlier (Grubbs):	none

Fig. 9 – Prothioconazole SC 480 g/L (free of outliers)



Mean value:	406.6g/kg
s_r:	4.10
s_R:	7.00
RSD_R:	1.72
$RSD_{R\ (Hor)}$:	2.29
HorRat:	0.74
Outlier (Grubbs):	none

Fig. 10 – Prothioconazole FS 258 (8 g/L Prothioconazole) (free of outliers)



Mean value:	7.123 g/kg
s_r:	0.127
s_R:	0.196
RSD_R:	2.75
$RSD_{R\ (Hor)}$:	4.21
HorRat:	0.65
Outlier (Grubbs):	none

7. Conclusions

The data of 20 different laboratories, who participated in this full scale CIPAC collaboration trial have been used for the statistical evaluation. The data presented in the statistical summary show that the method is suitable to gain acceptable and reproducible results for all samples tested and is therefore regarded to be robust.

Bayer AG, Crop Science Division consider this method to be suitable for the intended purpose, without further changes, and recommend accepting it as a provisional CIPAC method for the determination of Prothioconazole in technical samples and associated formulations: EC, FS and SC.