PYRAOXYSTROBIN

Small Scale Collaborative Trial on the Determination of Pyraoxystrobin in Technical Material and Formulations by HPLC

By

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1. INTRODUCTION

Pyraoxystrobin

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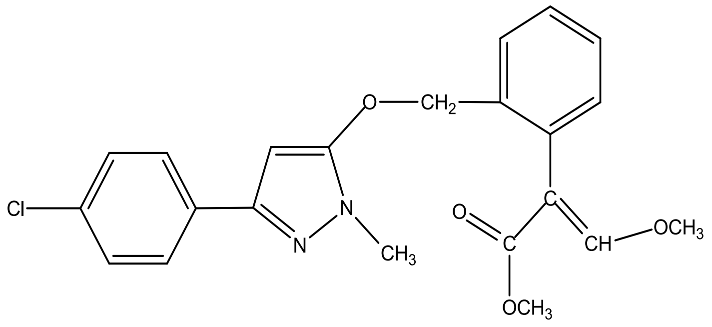

ISO Common name Pyraoxystrobin

CAS No [862588-11-2]

Chemical name:

(*E*)-methyl

2-(2-((3-(4-chlorophenyl)-1-methyl-1*H*-pyrazol-5-yloxy)methyl)phenyl)-

3-methoxyacrylate

Empirical formula $C_{22}H_{21}ClN_2O_4$

RMM 412.87

m.p. 129.6℃

v.p. 1.22E-008Pa

Solubility In water(g/L,20℃)：0.03mg/L，high solubility in DMF, acetone, ethyl acetate, methanol; Low solubility in petro-ether and water.

Description	The pure material is a white, odourless solid
Stability	Not stable in alcohol to heat
Formulations	Suspension concentrates

1.1. Scope

The results of the small scale collaborative study for pyraoxystrobin technical material and formulations are reported.

1.2. Samples

1) Pyraoxystrobin technical (TC-1)

2) Pyraoxystrobin suspension concentrates (SC-1)

3) Pyraoxystrobin suspension concentrates (SC-2)

1.3. Participants

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3) Mr. Zou weibin — JIANGSU ROTAM CHEMISTRY CO., LTD
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2. ANALYTICAL METHODS

2.1. Outline of Methods

Pyraoxystrobin in the test samples is determined by HPLC using uv detection.

Pyraoxystrobin Analytical standard, Batch No.: 20110927, Purity 99.7%;

2.2. program of work

We requested the collaborators to:

1) conduct duplicate determinations on two different days for each of the three samples;

2) inject each sample solution in duplicate and calculate the mean value;

3) describe operating conditions in detail;

4) recommended procedure if the method is deviate .

3. Remarks of participants

3.1. Analytical conditions

Lab	Liquid chromatograph integrator	Column	Mobile phase	Flow rate （ml/min）	Column temp(℃)
1	Waters Empower2	Eclipse XDB-C18 150mm×4.6mm	Acetonitrile/H_2O 60:40	1.3	ambient
2	Agilent HPLC ChemStation	Zorbax XDB-C18 150mm x 4.6 mm	Acetonitrile/H_2O 60:40	1.3	20
3	Agilent HPLC ChemStation	Agilent XDB-C18 150mm×4.6mm	Acetonitrile/H_2O 60:40	1.3	ambient
4	Agilent HPLC ChemStation	Agilent Zorbax C18 150mm x 4.6 mm	Acetonitrile/H_2O 60:40	1.5	ambient
5	Waters Empower2	SB-C18 150mm×4.6mm	Acetonitrile/H_2O 60:40	1.3	ambient

3.2. Remarks

- Lab 1

The column temperature had changed more than 5℃.

- Lab 4

The sample SC-2 was not dissolved completely when it Ultrasonic less than 15min.

4. Results and Discussion

Five data sheets were obtained from the five participants. Summary and detailed statistical evaluations are shown in Tables 1, 2-1 and 2-2. The statistical evaluations were carried out according to ISO 5725.

There is no stragglers and outliers in the small scale collaborative trial on Pyraoxystrobin .

5. CONCLUSION

For all samples, the values of RSD_R(reproducibility relative standard deviation) were smaller than those calculated by Horwitz′ s equation. The proposed method is considered appropriate for the determination of pyraoxystrobin in technical and suspension concentrates product.

We proposes proceeding to a large scale collaborative study.

Table 1 Summary of Statistical Evaluation of Pyraoxystrobin Small Scale Collaborative Study

	TC-1	***SC-1***	***SC-2***
Average(g/kg)	973.78	203.56	199.06
Number of labs.	5	5	5
Repeatability standard deviation(S_r)	2.145	1.200	0.951

"Pure" between laboratory standard variation(S_L)	4.785	0.857	2.210
Reproducibility standard deviation(S_R)	5.244	1.475	2.405
Repeatability(r)	6.006	3.360	2.663
Reproducibility(R)	14.683	4.130	6.734
RSD_r	0.220	0.590	0.478
RSD_R	0.539	0.725	1.208
Horwitz's value	2.008	2.542	2.551

Table 2-1 Statistical Evaluation (Technical -1)

Lab.		*Analytical data (n=4)*		*Yi*	*$(Yi)^2$*	*Si*	**Si^2**
1	Day1	979.7	980.4	980.32	961027.30	0.592	0.350
	Day2	980.1	981.1				
2	Day1	975.0	980.3	977.50	955506.25	3.492	12.194
	Day2	974.0	980.7				
3	Day1	972.0	972.7	972.08	944939.53	0.436	0.190
	Day2	971.7	971.9				
4	Day1	968.6	973.4	969.80	940512.04	2.903	8.427
	Day2	966.6	970.6				
5	Day1	970.8	967.7	969.22	939387.41	1.358	1.844

Day2	969.8 968.6
S1 SUM Yi=	4868.92
S2 SUM Yi^2=	4741372.50
S3 SUM Si^2	23.005

1) Cochran's test (p=5,n=4)

$C=Si^2max/S3 = 0.530 < 0.598$ (p=5,n=4, 5%)

2) Grubb's test (p=5,n=4)

Yi(min) = 969.22 Yi(max) =980.32 Y = S1/p 973.78

S=4.902

Y – Yi(min) = 4.56

Yi(max) –Y = 6.54

Lower = ［Y –Yi(min)］/S = 0.930 ＜ 1.67 (p=5,n=4, 5%)

Upper = ［Yi(max)-Y］/S=1.334 ＜1.67 (p=5,n=4, 5%)

3) Calculation of r and R

Mean; Y= S1/ p = 973.78

$Sr^2 = S3 / p = 4.601$ Sr = 2.145

$SL^2=$ ［$(pS2-S1^2)/p(p-1)$］ $-(Sr^2/n) = 22.900$ SL=4.785

$SR^2= Sr^2 + SL^2 = 27.501$ SR = 5.244

r = 2.8 × Sr = 6.006

R= 2.8 × SR = 14.683

RSDr = (Sr / mean) × 100 = 0.220

RSDR = (SR / mean) × 100 = 0.539

Horwitz's Value = $2^{[1-0.5 \times \log(Y/1000)]}$ = 2.008

RSDR ＜ 2.008 (Horwitz's Value)

Table 2-2 Statistical Evaluation (SC -1)

Lab.		*Analytical data (n=4)*		*Yi*	*(Yi)²*	*Si*	**Si²**
1	Day1	205.7	204.1	203.40	41371.56	1.937	3.752
	Day2	202.6	201.2				
2	Day1	204.7	204.9	205.20	42107.04	1.543	2.381
	Day2	207.4	203.8				
3	Day1	203.8	204.2	203.60	41452.96	0.479	0.229
	Day2	203.4	203.1				
4	Day1	203.3	202.9	203.30	41330.89	0.432	0.187
	Day2	203.9	203.1				
5	Day1	202.7	203.2	202.30	40925.29	0.810	0.656
	Day2	201.5	201.7				
S1 SUM Yi=				1017.80			
S2 SUM Yi²=					207187.74		
S3 SUM Si²							7.205

1) Cochran's test (p=5, n=4)

$C = Si^2max/S3 = 0.521 < 0.598$ (p=5,n=4, 5%)

2) Grubb's test (p=5, n=4)

Yi(min) = 202.30 Yi(max) =205.20 Y = S1/p 203.56

S=1.045

Y – Yi(min) = 1.26

Yi(max) –Y = 1.64

Lower = ［Y –Yi(min)］/S = 1.206 ＜ 1.67 (p=5, n=4, 5%)

Upper = ［Yi(max)-Y］/S= 1.569 ＜1.67 (p=5, n=4, 5%)

3) Calculation of r and R

Mean; Y= S1/ p = 203.56

Sr^2 = S3 / p = 1.441 Sr = 1.200

SL^2= ［(pS2-$S1^2$)/p(p-1)］-(Sr^2/n) = 0.735 SL=0.857

SR^2= Sr^2 + SL^2 = 2.176 SR = 1.475

r = 2.8 × Sr = 3.360

R= 2.8 × SR = 4.130

RSDr = (Sr / mean) × 100 = 0.590

RSDR = (SR / mean) × 100 = 0.725

Horwitz's Value = 2［1- 0.5 × log (Y / 1000)］= 2.542

RSDR ＜ 2.542 (Horwitz's Value)

Table 2-3 Statistical Evaluation (SC -2)

Lab.	*Analytical data (n=4)*		*Yi*	*$(Yi)^2$*	*Si*	**Si^2**
1 Day1	197.4	196.9	196.02	38423.84	1.325	1.756
Day2	194.7	195.1				
2 Day1	196.0	199.5	197.55	39026.00	1.515	2.295

Day2	196.8	197.9				
3 Day1	202.1	201.5	201.62	40650.62	0.320	0.102
Day2	201.5	201.4				
4 Day1	199.5	198.9	199.60	39840.16	0.577	0.333
Day2	199.7	200.3				
5 Day1	200.7	200.6	200.50	40200.25	0.183	0.033
Day2	200.3	200.4				
S1 SUM Yi=			995.29			
S2 SUM Yi^2=				198140.87		
S3 SUM Si^2						4.519

1) Cochran's test (p=5, n=4)

C=Si^{2}max/S3 = 0.508 ＜ 0.598 (p=5,n=4, 5%)

2) Grubb's test (p=5, n=4)

Yi(min) = 196.02 Yi(max) =201.62 Y = S1/p 199.06

S=2.261

Y – Yi(min) = 3.04

Yi(max) –Y = 2.56

Lower = ［Y –Yi(min)］/S = 1.345 ＜ 1.67 (p=5,n=4, 5%)

Upper = ［Yi(max)-Y］/S= 1.132 ＜1.67 (p=5,n=4, 5%)

3) Calculation of r and R

Mean; Y= S1/ p = 199.06

Sr^2 = S3 / p = 0.904 Sr = 0.951

SL^2=［(pS2-S1^2)/p(p-1)］-(Sr^2/n) = 4.882　　SL=2.210

SR^2= Sr^2 + SL^2 = 5.786　　　　SR = 2.405

r = 2.8 × Sr = 2.663

R= 2.8 × SR = 6.734

RSDr = (Sr / mean) × 100 = 0.478

RSDR = (SR / mean) × 100 = 1.208

Horwitz's Value = 2［1- 0.5 × log (Y / 1000)］= 2.551

RSDR ＜ 2.551　　(Horwitz's Value)

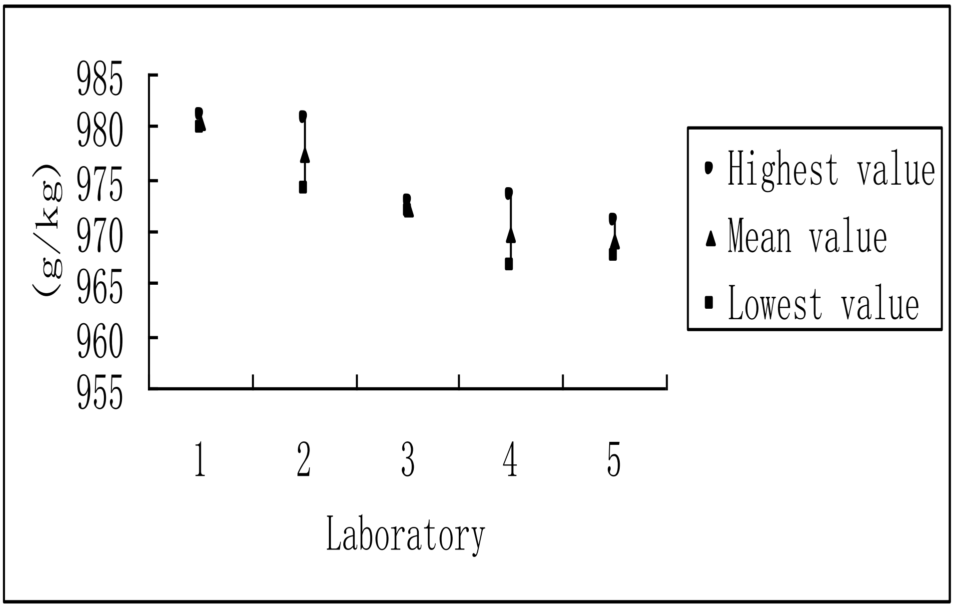

Fig.1　Praoxystrobin Technical-1

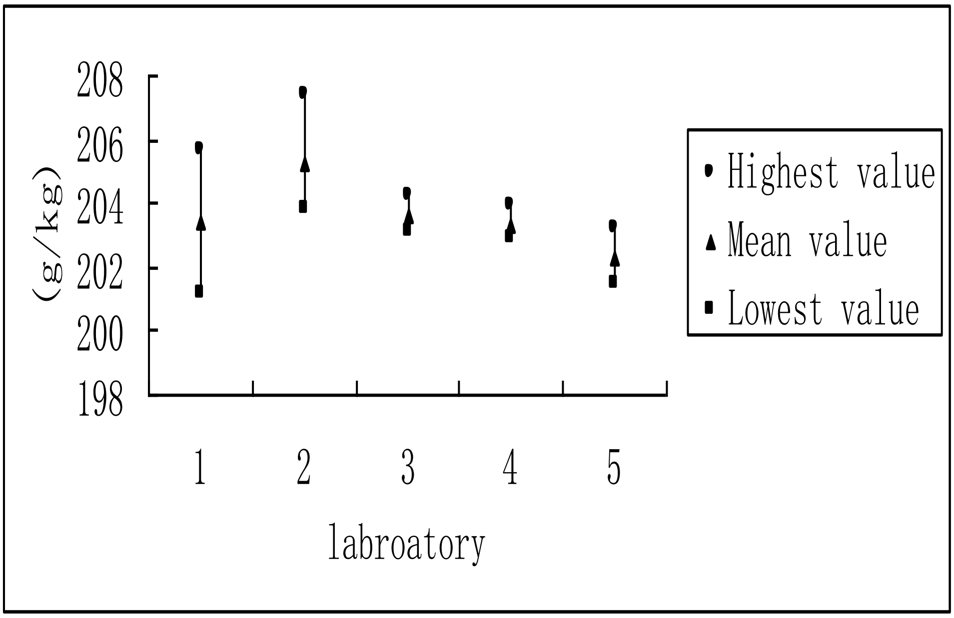

Fig.2 **Praoxystrobin SC-1**

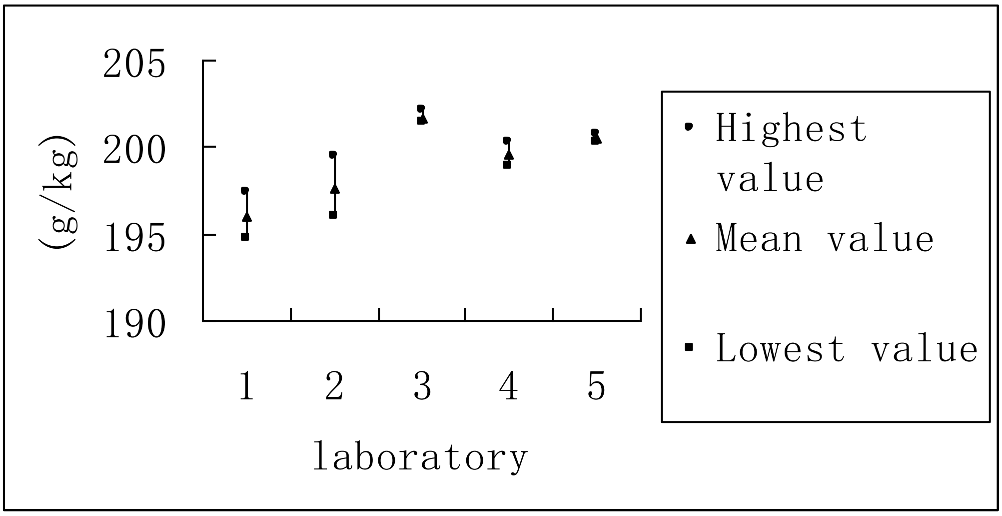

Fig.3 **Praoxystrobin SC-2**