183 Trifluralin

Collaborative Study

Small Scale Collaborative Study for the

Determination of Trifluralin TC and EC formulation by HPLC

Report to CIPAC

by

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Content

1. Participants

Small Scale Collaborative Study for the determination of trifluralin TC and EC formulation by HPLC was organized by Shenyang SYRICI Testing Co., Ltd, and participated by 4 labs. All of the 4 laboratories provided their results, which are presented in the following sections.

Participating laboratories are listed in alphabetical order in the table below whereas lab numbers in the result tables were assigned, chronologically, based upon receipt of results.

No.	Contact	Lab
1	Ling Duoduo	Anhui Fengle Agrochemical Co., LTD. Product Testing Center Hefei Circular Economy Demonstration Park, Feidong County, Hefei Anhui, China
2	Xu Mei	Limin Chemical Co., Ltd. Economic Development Zone, Xinyi Jiangsu,China
3	Wu Peng	National Center for Pesticide Quality Supervision and Inspection (Beijing), Institute for the Control of Agrochemicals, Ministry of Agriculture and Rural Affairs, P. R. China Maizidian Road No.22, Chaoyang District, Beijing, China.
4	Hou Chunqing	Shenyang Research Institute of Chemical Industry Co., Ltd Pesticides Test Laboratory No.8 Shenliao East Road, TieXi District, Shenyang Liaoning, China

2. Active Ingredient: General Information

Chemical name: 2, 6-dinitro-N,N-dipropyl-4-(trifluoromethyl)benzenamine)

ISO common name: Trifluralin

CAS-No.:   1582-09-8
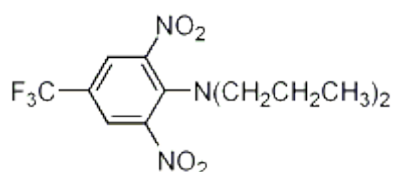
Structure:

Molecular mass: 335.3 g/mol

Empirical formula: $C_{13}H_{16}F_3N_3O_4$

Activity: Herbicide

3. Samples

Five test samples and Trifluralin analytical standard were sent to the participants:

1. Trifluralin tech. sample – 1 (TC-1 Batch number 2020071501)
2. Trifluralin tech. sample – 2 (TC-2 Batch number 2020081903)
3. Trifluralin EC sample – 3 (EC-1 Batch number 2020062401)
4. Trifluralin EC sample – 4 (EC-2 Batch number 2020072401)
5. Trifluralin EC sample – 5 (EC-3 Batch number 2020080701)
Trifluralin, reference standard (purity 99.3 %w/w)

4. Method

4.1 Scope

The determination of Trifluralin active ingredient content in technical grade material (TC) and EC formulation.

4.2 Principle

Trifluralin is determined by reversed phase high performance liquid chromatography using UV detection at 280 nm and external standardization.

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 double injections of the calibration solution. The average response factor, used to calculate the amount of Trifluralin in the test solution, was calculated using the injection before and after the test injections.

5. Remarks of the Participants

Lab	Instrument	Column	Flow Rate	Column Temp.℃	Wavelength	Injection Volume	Mobile phase （V/V）
1	Agilent 1260	ZORBAX SB-C18 4.6× 150mm, 5μm	1.0	25	280	5	Acetonitrile/Water =77/23
2	Agilent 1260	SHIMADZU VP-ODS 150L*4.6	1.0	25	280	5	Acetonitrile/Water =77/23
3	Agilent 1200	Eclipse Zorbax XDB-C8 4.6× 150mm, 5μm	1.0	25	280	5	Acetonitrile/Water =77/23
4	Agilent 1260	Agilent XDB C8 4.6×150mm, 5 μm	1.0	25	280	5	Acetonitrile/Water =77/23

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 participants, were deemed not to affect the analytical results significantly and therefore all data sets were included within the statistical assessment.

Therefore, the report below contains statistical evaluations with the full set of 4 participating laboratories.

6.2 Determination of Trifluralin

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.

In the tables 1-3 and figures 1-5, respectively, the full set of all laboratories (4 participants) are reported. No outliers and stragglers have been found. The Horwitz Ratio (HorRat) was found within the desired range (≥ 0.3 but ≤ 1.0).

Determination of Trifluralin –4 labs

All results are given in g/kg

Table 1 Results

	Trifluralin TC-1		Trifluralin TC-2		Trifluralin EC-1		Trifluralin EC-2		Trifluralin EC-3	
	Day 1	Day 2	Day 1	Day 2	Day 1	Day 2	Day 1	Day 2	Day 1	Day 2
Lab 1	980.4	978.0	979.3	977.5	458.2	459.3	456.4	455.6	454.8	455.5
Lab 2	983.5	983.7	982.7	983.5	457.2	454.1	457.3	458.9	461.4	460.1
Lab 3	980.2	980.4	982.4	981.7	455.7	456.1	452.2	454.0	457.0	457.2
Lab 4	982.5	987.8	980.7	976.6	458.9	454.0	458.9	457.4	458.2	458.8

Table 2 Mean values

	Trifluralin TC-1	Trifluralin TC-2	Trifluralin EC-1	Trifluralin EC-2	Trifluralin EC-3
Lab 1	979.2	978.4	458.7	456.0	455.2
Lab 2	983.6	983.1	455.7	458.1	460.8
Lab 3	980.3	982.0	455.9	453.1	457.1
Lab 4	985.2	978.7	456.5	458.1	458.5

Table 3 Summary of the statistical evaluation

	Trifluralin TC-1	Trifluralin TC-2	Trifluralin EC-1	Trifluralin EC-2	Trifluralin EC-3
x_m [g/kg]	982.06	980.55	456.68	456.33	457.85
x_m [% w/w]	98.21	98.06	45.67	45.63	45.79
L	4	4	4	4	4
S_r	3.09	2.32	2.00	2.04	1.58
S_R	3.88	3.12	2.23	2.96	2.74
r	8.66	6.50	5.61	5.70	4.41
R	10.86	8.72	6.24	8.29	7.66

RSD$_R$	0.40	0.32	0.49	0.65	0.60
RSD$_{R\,(Hor)}$	1.42	1.42	1.59	1.59	1.59
HorRat	0.279	0.224	0.307	0.408	0.376

x$_m$ = total mean value

L = number of laboratories

s$_r$ = repeatability standard deviation

s$_R$ = reproducibility standard deviation
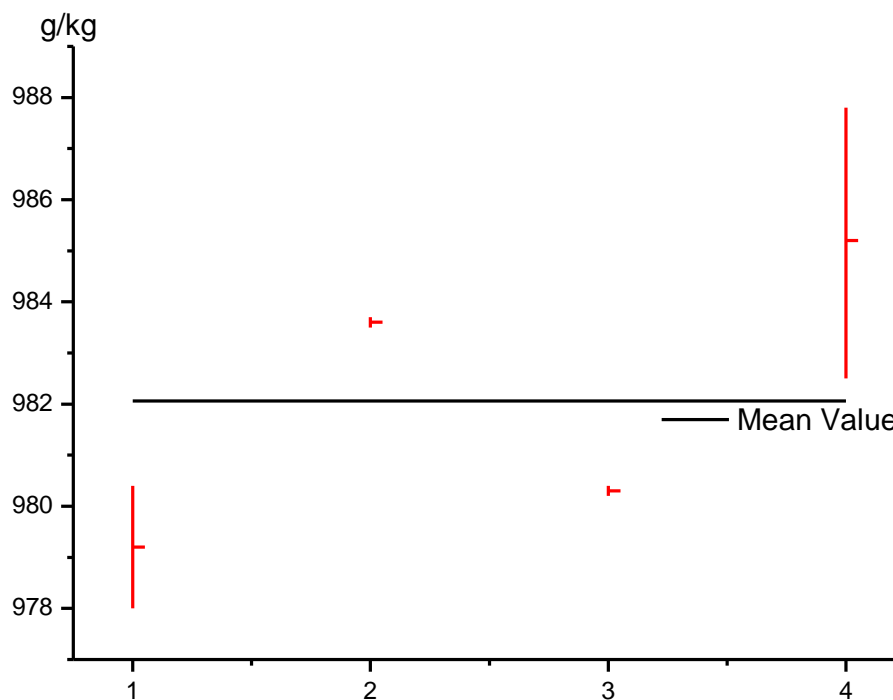
r = repeatability limit

R = reproducibility limit

RSD$_R$ = relative standard deviation of reproducibility

RSD$_{R\,(Hor)}$ = Horwitz Value according to Horwitz equation

HorRat = Horwitz Ratio (RSD$_R$ / RSD$_{R\,(Hor)}$)

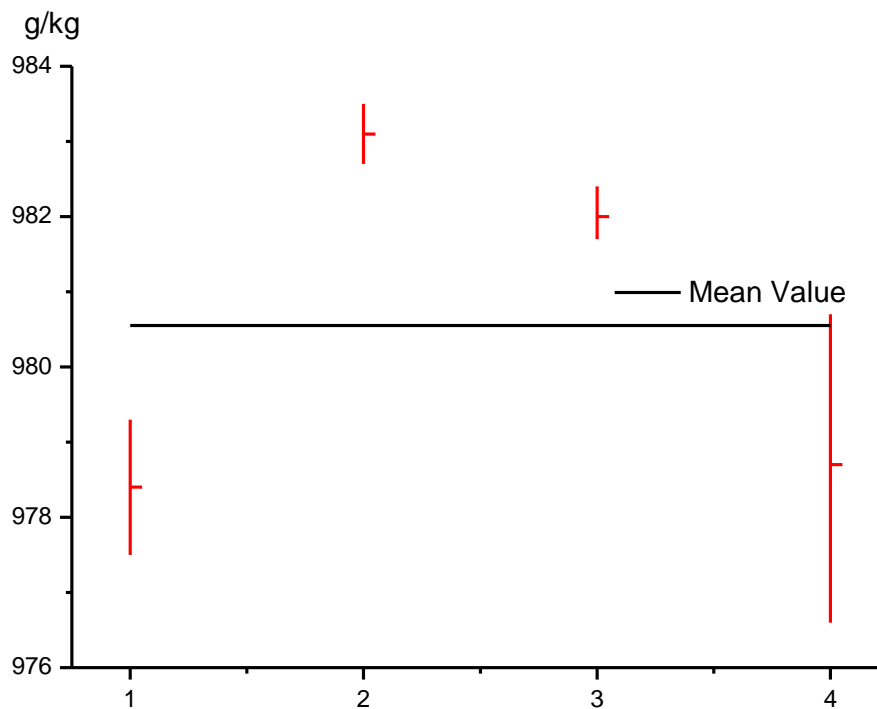Full set of 4 participants:

Fig. 1 Trifluralin tech. sample - 1

Mean value: 982.06 g/kg

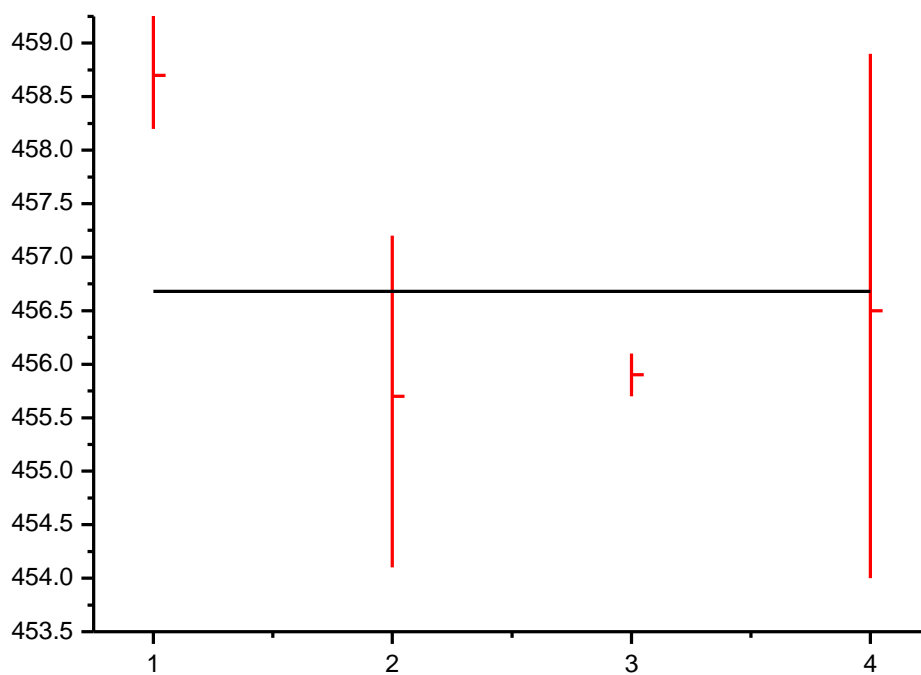S$_r$:   3.09

S$_R$:   3.88

RSD$_R$: 0.40
RSD$_{R(Hor)}$: 1.42
HorRat: 0.279
Outlier (Grubbs): none
Straggler (Grubbs): none

Fig. 2 Trifluralin tech. sample - 2

Mean value: 980.55 g/kg
S$_r$: 2.32
S$_R$: 3.12
RSD$_R$: 0.32
RSD$_{R(Hor)}$: 1.42
HorRat: 0.224
Outlier (Grubbs): none
Straggler (Grubbs): none

Fig. 3 Trifluralin EC sample – 1

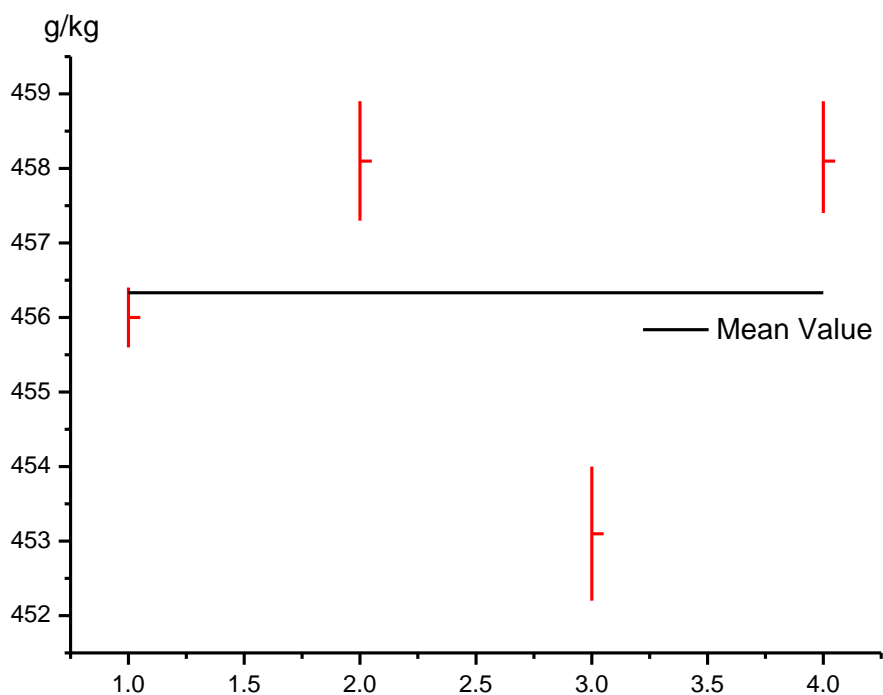|	
Mean value:	456.68 g/kg
S_r:	2.00
S_R:	2.23
RSD_R:	0.49
$RSD_{R\ (Hor)}$:	1.59
HorRat:	0.307
Outlier (Grubbs):	none
Straggler (Grubbs):	none

Fig. 4 Trifluralin EC sample– 2

Mean value: 456.33 g/kg
S_r: 2.04
S_R: 2.96
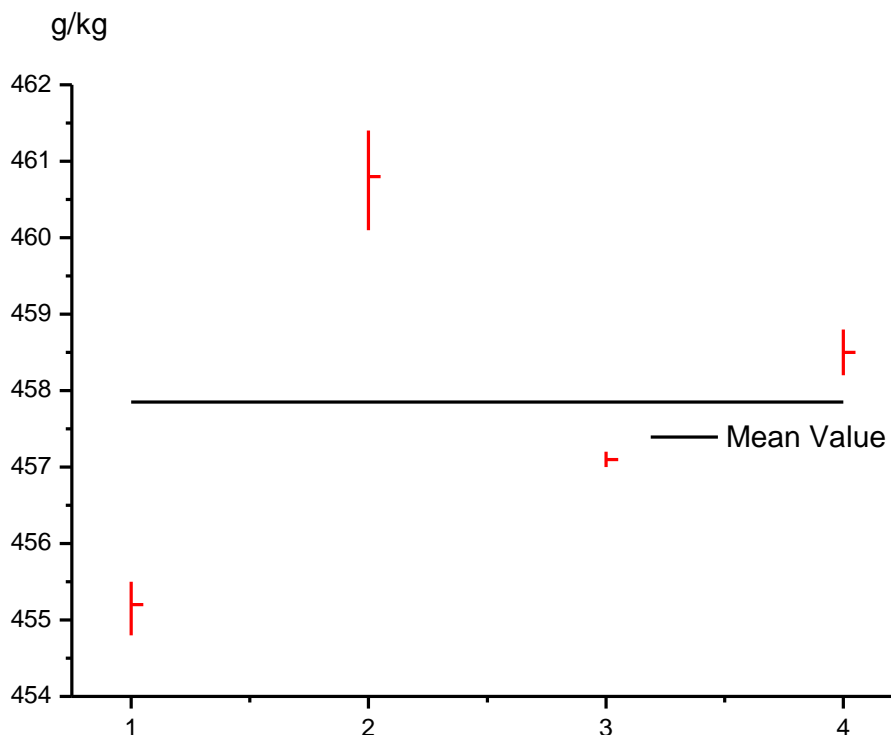RSD_R: 0.65
$RSD_{R (Hor)}$:   1.59
HorRat: 0.408
Outlier (Grubbs): none
Straggler (Grubbs): none

Fig. 5 Trifluralin EC sample– 3

Mean value: 457.85 g/kg
S_r: 1.58
S_R: 2.74
RSD_R: 0.6
$RSD_{R (Hor)}$: 1.59
HorRat: 0.376
Outlier (Grubbs): none
Straggler (Grubbs): none

7. Conclusions

A total of 4 laboratories have participated in the small scale collaborative study.

The data presented in the statistical summary show that this method led to HorRat values all within the required range. That is evidence for the fact that the present HPLC method is acceptable and suitable to produce reproducible results.

SYRICIT considers this method to be suitable for the intended purpose, without further changes, and recommends for performing large-scale collaborative trials.