TRIFLURALIN

Collaborative Study

Full Scale Collaborative Study for the
Determination of Trifluralin in TC and EC formulation by HPLC

Report to CIPAC
by
Shenyang SYRICI Testing Co., Ltd.
No.8, Shenliao East Road,
Tiexi District Shenyang 110021, P.R. China

May 2022

Content

1. Participants

In November 2021, Information Sheet No. 329 was sent out by the CIPAC Secretary inviting members to participate in a large-scale collaborative study on the determination of Trifluralin in TC and EC formulation by HPLC. 22 laboratories responded to participate in the trial.

By mid of April 2022, 20 of the 22 laboratories provided their results, and 2 of them didn't provide due to the failure of customs clearance of the samples. The results of the 20 participants are presented in the following sections.

All the participants are listed in the table below whereas lab numbers in the result tables were assigned, chronologically, based upon receipt of results.

Table 1: list of participants

Contact	Lab	Country/Region
Vanessa Lecocq	Walloon Agricultural Research Centre (CRA-W) Batiment Rachel Carson Rue du Bordia,115030 Gembloux Belgique Belgium	Belgium Europe
Olga Novakova	National Reference Laboratory Department of Testing Plant Protection Products Zemědělská 1a, 613 00 Brno Czech Republic	Czech Republic Europe
Zhang Juntao	Jiangsu EverTest Co., Ltd. 31-1 Hengjing Road, Qixia District, Nanjing, Jiangsu, P.R. China	China Asia
Sagar Patil	Ross Lifescience Dy. TFM, Lead Project Co-Ordinator. Ross Lifescience Pvt. Ltd.	India Asia
Angela Santilio	National Institute of Health (Istituto Superiore di Sanità) Department of Environmental and Health ECASS Section - Pesticide Unit	Italy Europe
Takashi Watanabe	FAMIC Japan	Japan Asia
Maureen Tian	Test Center of Shandong Weifang Rainbow Chemical Co., Ltd. Binhai Economic Development Area, Weifang, Shandong, China China	China Asia
Judy	Shandong Binnong Technology Co., Ltd. No.518, Yongxin Road, Binbei Town, Binzhou, Shandong, China	China Asia

|			
Javier García-Hierro Navas	Agri-Food Arbitration Laboratory Ministry of Agriculture, Fisheries and Food Aguarón, 13. E28023 Madrid Spain	Spain Europe
Elen Karasali	Benaki Phytopathological Institute Benaki Phytopathological Institute 8 Stefanou Delta Street, Kifissia, Athens, 14561 Greece.	Greece Europe
Brenda Checa	Ministerio de Desarrollo Agropecuario Dirección Nacional de Sanidad Vegetal Panama	Panama South America
Lajos Benke	Pesticide Analytical National Reference Laboratory Velence H-2481 Velence, Ország út 23, Hungary	Hungary Europe
Christian Mink	Syngenta Crop Protection AG	Switzerland Europe
Aysel TAKKABULAN	Plant Protection Central Research Institute Gayret Mah. Fatih Sultan Mehmet Bul. No:66 Yenimahalle Ankara, TURKEY	TURKEY Asia
Huang Liang	Anhui Fengle Agrochemical Co., Ltd. Hefei Circular Economy Demonstration Park, Feidong County, Hefei City, Anhui Province 231600 China	China Asia
Shi Kaiwei	National Center for Pesticide Quality Supervision and Inspection (Beijing), Institute for the Control of Agrochemicals, Ministry of Agriculture and Rural Affairs , P. R. China	China Asia
Shang Wei	Laprode （ZHE JIANG）Analysis Co., Ltd.	China Asia
Theo. Derijk	Wageningen Food Safety Research (WFSR) Postbus 230, 6700 AE Wageningen, Akkermaalsbos 2 (gebouw 123), 6708 WB Wageningen	Netherlands Europe
Hou Chunqing	Pesticides Test Laboratory of Shenyang SYRICI Testing Co., Ltd. No.8, Shenliao East Road, Tiexi District Shenyang 110021, P.R. China	China Asia
Estela Bonilha	Federal Laboratory of Animal and Plant Health and Inspection - LFDA/SP Ministry of Agriculture, Livestock and Food Supply Rua Raul Ferrari, sem número, Jardim Santa Marcelina	Brazil South America

2. Active Ingredient: General Information

Chemical name:　　α,α,α-trifluoro-2,6-dinitro-N,N-dipropyl-p-toluidine(IUPAC)

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

ISO common name: Trifluralin

CAS-No.:　　　1582-09-8

Structure:

F_3C—[benzene ring with NO_2 at top, NO_2 at bottom]—$N(CH_2CH_2CH_3)_2$
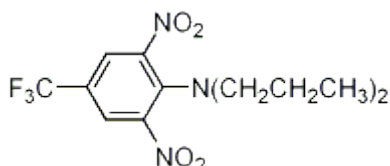
Molecular mass:　　335.3 g/mol

Empirical formula:　C13H16F3N3O4

Activity:　　　　Herbicide

3. Samples

The following 5 batches of test samples and analytical standard were sent to the participants:

1. Trifluralin TC-1, Batch Number: 21090204390

2. Trifluralin TC-2, Batch Number: 21090404393

3. Trifluralin EC-1, Batch Number: 2021090101

4. Trifluralin EC-2, Batch Number: 2021093001

5. Trifluralin EC–3, Batch Number: 2021102701

Trifluralin, reference standard (purity 99.2 %w/w)

4. Method

4.1 Scope

The determination of Trifluralin active ingredient content in TC and EC formulation.

4.2 Principle

Trifluralin is determined by reverse 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 injections of test solution.

5. Remarks of the Participants

5.1 Analytical condition

Table 2: Analytical conditions used by the participants

Lab No.	HPLC Model	Column	FR (mL/min)	CT (ºC)	WL (nm)	Inj. vol. (μL)	Mobile Phase Acetonitrile /Water v/v
1	Agilent 1100 Series	Agilent Zorbax Eclipse XDB-C18, 5 μm, 150 x 4.6 mm i.d.	1.0	25	280	5	77/23
2	Agilent Infinity 1290	Zorbax Extend C18, 150 x 4.6 mm, 5 um	1.0	25	280	5	77/23
3	Agilent 1260 Infinity II	150mm*4.6mm,5μm C18	1.0	25	280	5	77/23
4	Agilent Technologies/ 1260 Infinity	Eclipse Zorbax XDB-C18 4.6 mm x 150	1.0	25	280	5	77/23
5	Perkin Elmer Flexar	Zorbax Eclipse XDB C18 4.6 x 250mm; 5 um	1.0	25	280	10	77/23
6	SHIMADZU / LC-20AD	ZORBAX Eclipse XDB-C18, 150mm x 4.6 mm, 5 μm	1.0	25	280	5	77/23
7	Agilent 1260 InfinIty Ⅱ	Agilent Eclipse XDB-C18 4.6*250mm*5μm	1.0	25	280	5	77/23
8	Agilent Technologies Pte. Ltd DAD: DEAEK00202/G7117C	XBridge Ò C18 5 μm (150 mm x 4.6 mm)	1.0	25	280	5	77/23
9	HPLC Agilent 1260 Infinity II	Phenomenex Gemini 5μ C18 110 A	1.0	25	280	5	77/23
10	HPLC Shimadzu LC-20AB	Nucleodur C18 Gravity (150/4.6 x 5μm)	1.3	30	280	5	77/23
11	1260 Infinity	Eclipse XDB-C18 5μm	1.0	25	280	5	77/23

		4.6x150mm					
12	Dionex HPLC system	Eclipse Zorbax XDB-C18 250 x 3mm, 5µm	0.7	25	280	5	77/23
13	Thermo Scientific / Ultimate 3000	Agilent Zorbax Eclipse XDB	1.5	25	280	5	77/23
14	AGILENT HPLC 1100 SERIES	Agilent Eclipse Zorbax XDB-C18 5 150 x 4.6 mm, 5 um	1.0	25	280	5	77/23
15	Agilent1260	ZORBAX SB-C18 4.6×150mm 5 um	1.0	25	280	5	77/23
16	Agilent 1200 DAD	Eclipse XDB-C18 250*4.6 mm,5 µm	1.0	25	280	5	77/23
17	Agilent 1260 (DAD)	Zorbax SB-C18 150mm*4.6mm*5 µm	1.0	25	280	1.0	77/23
18	Shimadzu LC-40DXR	Eclipse XDB-C18	0.6	25	280	10	75/25
19	Agilent1260 Infinity Ⅱ	Agilent ZORBAX Eclipse XDB-C18 4.6×150mm 5 µm	1.0	25	280	5	77/23
20	Agilent Infinity 1260	Eclipse Zorbax XDB-C18 150 x 4.6 mm, 5 um	1.0	25	280	5	77/23

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

Table 3: Remarks from participants

Lab No	Comments
4	Remarks：The Trifluralin content was found within specification limit for TC and EC. 2. Retention time varied from method given.
6	Remarks：1. Run time was changed from 8.0 to 15.0 min. Because the retention time for trifluralin at our laboratory (approx. 7.2 min) is longer than the typical one (approx. 5.2 min). As a comment on the draft method, "Run time: 8.00 min" is not consistent with the three typical HPLC-chromatograms listed on pages 5 to 6, which were recorded up to 15 min. Those times should be the same. 2. "221/TC/M" referenced in the method of analysis for TRIFLURALIN EC seems to be a typographical error for "183/TC/M". "221/TC/M" is the method of analysis for chlorpyrifos technical.
7	1. Approximately weigh 50mg of trifluralin standard respectively into 50ml volumetric flask. 2. Approximately weigh containing trifluralin 50mg of trifluralin TC or EC

	into 50ml volumetric flask.
10	Remarks：The analytical standard and the technicals (TC1 and TC2) were not powders. They were a single soild mass. A glass rod was used in order to break and homogenize the solid materials.
13	Remarks：Experiments were performed three times. Only two repetitions are shown here. Results of samples TC-1 and TC-2 as well as sample morphology (agglomeration, wet appearance) indicate inhomogeneity of the sample material.
14	Remarks: The samples were studied according to the method. Especially the sample preparation step of the method was very practical and did not take much time. There were no problems in the analysis. Run time was worked as 8 minutes. In the method sent, retention time is given as approximately 5.2 minutes. However, in our all studies, the peak of trifluralin was around 6.3 minutes.
17	Remarks：1.repeated the same exercise on different day(Day 1 on February 11,2022 ,Day 2 on February 14,2022)by different person. 2.The retention time is about 6.5min. 3.Syetem equilibration: calibration solution is injected repeatedly, and the deviation of relative retention time and the response factors is less than 1%.
20	Remarks：The procedure described at "trifluralin analytical method (final)" was followed faithfully. The retention time and peak area obtained from two consecutive CA calibration solution injections deviate was less than 1.0%. The response factor for CB solution deviate was less than 1.0% from that for calibration solution CA. For EC samples, the concentration information 45.4 (w%), indicated on the safety data sheet that accompanied the samples, was considered to calculate the mass necessary to obtain 50mg of trifluralin (s mg) in the volumetric flask (50mL). For the TC samples, the concentration information 96% (w%), indicated on the safety data sheet that accompanied the sample, was considered to calculate the mass necessary to obtain 50mg of trifluralin (s mg) in the volumetric flask (50mL). The retention time was approximately 6,1/6,2min.

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 from 20 participating laboratories were included within the statistical assessment.

6.2 Statistical results

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/ Cochran test on a 1%/5% significance level, respectively.

In the tables 4-11and figures 1-5, respectively, the full set of 20 laboratories are reported. Laboratory 4 three times was identified as outliers of EC-1,EC-2,EC-3 according to Grubbs test. And lab 5 for TC-1, lab 5 and 6 for TC-2, lab 2,5 and 18 for EC-1, lab 5,11 and 18 for EC-2; lab 5, 6 and 18 for EC-3 were identified as outlier according to Cochran test. In the table 12 and figures 6-10, the statistical data without the above outliers was reported. The Horwitz Ratio (HorRat) was found within the desired range (≥ 0.3 but ≤ 1.0).

Results from all the 20 participants
All results are given in g/kg

Table 4 Results of TC-1

Lab	Day 1		Day 2		Mean g/kg	Std. Dev.
	A	B	A	B		
1	989.6	986.2	982.2	986.0	986.0	3.0243
2	981.3	981.3	987.8	986.5	984.2	3.4189
3	980.4	977.6	978.4	978.8	978.8	1.1776
4	965.3	966.2	965.3	966.6	965.9	0.6557
5*	1000.7	1014.9	981.5	982.0	994.8	16.1198
6	984.5	984.6	983.7	991.4	986.1	3.5893
7	978.2	980.6	984.2	984.1	981.8	2.9125
8	987.2	985.6	984.5	985.9	985.8	1.1106
9	980.9	982.5	977.9	972.1	978.4	4.5822
10	978.3	975.8	984.2	984.8	980.8	4.4275
11	987.4	988.5	982.5	986.7	986.3	2.6235
12	980.0	978.3	979.4	981.1	979.7	1.1690
13	979.7	990.6	988.4	981.8	985.1	5.2022
14	967.6	968.3	965.7	962.6	966.1	2.5489

15	983.8	983.0	984.9	982.9	983.7	0.9256
16	986.6	979.0	981.6	983.0	982.6	3.1681
17	984.9	987.1	985.6	986.9	986.1	1.0532
18	976.3	975.7	971.4	975.6	974.8	1.7951
19	982.8	983.1	982.7	983.1	982.9	0.2062
20	987.4	990.8	985.7	985.0	987.2	2.5876

* means Cochran outliers

Table 5 Results of TC-2

Lab	Day 1		Day 2		Mean g/kg	Std. Dev.
	A	B	A	B		
1	987.0	978.4	980.3	983.5	982.3	3.7745
2	973.9	970.7	978.3	974.1	974.3	3.1172
3	976.7	978.5	977.5	979.0	977.9	1.0275
4	967.6	964.0	965.6	964.9	965.5	0.8732
5*	965.0	974.4	995.0	967.1	975.4	13.6895
6*	982.0	980.8	988.2	1001.1	988.0	9.3003
7	981.3	980.3	985.2	981.3	982.3	2.5891
8	985.3	986.7	985.0	986.5	985.9	0.8500
9	982.5	979.1	974.9	970.2	976.7	5.3194
10	976.3	979.7	974.8	978.7	977.4	2.2322
11	990.9	986.5	981.8	982.2	985.4	4.2681
12	978.3	981.3	981.4	981.3	980.6	1.5174
13	994.0	985.4	989.5	991.4	990.1	3.6216
14	962.4	960.9	968.1	967.3	964.7	3.5612
15	982.6	981.4	984.1	982.4	982.6	1.1147
16	983.5	979.1	982.7	983.8	982.3	2.1670
17	984.6	986.8	986.3	984.8	985.6	1.0905
18	979.7	977.0	975.5	976.5	977.2	2.2546
19	982.7	982.9	982.8	982.7	982.8	0.0957
20	985.6	984.2	985.2	980.3	983.8	2.4226

* means Cochran outliers

Table 6 Results of EC-1

Lab	Day 1		Day 2		Mean g/kg	Std. Dev.
	A	B	A	B		
1	460.8	461.3	459.6	462.6	461.1	1.2321
2*	456.7	457.2	462.1	472.7	462.2	7.4276
3	452.6	454.7	453.4	453.8	453.6	0.8732
4	482.5	480.7	481.6	481.2	481.5	0.7375
5*	463.6	457.8	462.2	483.3	466.7	11.3229

6	458.8	459.2	460.5	460.6	459.8	0.8939
7	460.8	462.5	458.5	457.3	459.8	2.3258
8	462.6	461.6	461.1	462.5	462.0	0.7234
9	453.6	455.5	452.0	453.2	453.6	1.4406
10	453.6	454.6	459.6	455.8	455.9	2.6457
11	457.4	458.2	457.4	458.6	457.9	0.6000
12	460.5	457.4	460.3	457.0	458.8	1.8536
13	465.2	466.5	464.1	467.9	465.9	1.6383
14	461.4	461.2	460.2	460.5	460.8	0.5679
15	456.7	454.8	456.9	455.3	455.9	1.0340
16	459.5	454.4	460.6	460.5	458.8	2.9422
17	458.5	460.4	463.7	460.8	460.8	2.1739
18*	477.3	477.8	453.8	454.0	465.7	13.6835
19	460.9	461.5	461.4	462.1	461.5	0.4924
20	454.5	455.5	456.6	457.0	455.9	1.1284

* means Cochran outliers

Table 7 Results of EC-2

Lab	Day 1		Day 2		Mean g/kg	Std. Dev.
	A	B	A	B		
1	459.8	461.3	457.7	458.6	459.4	1.5588
2	460.2	458.8	466.5	460.5	461.5	3.4147
3	452.0	453.8	453.4	453.4	453.2	0.7895
4	479.7	480.2	478.7	476.3	478.7	1.7328
5*	464.6	466.1	475.0	471.4	469.3	4.8037
6	456.7	456.9	459.6	457.4	457.7	1.3329
7	459.1	463.7	460.0	460.8	460.9	1.9916
8	456.9	457.1	459.0	459.8	458.2	1.4259
9	455.5	454.1	452.2	453.9	453.9	1.3525
10	458.8	461.0	460.9	463.0	460.9	1.7154
11*	463.9	460.5	453.7	451.2	457.3	5.8869
12	457.9	458.6	458.0	459.3	458.5	0.6455
13	462.8	462.2	464.2	460.1	462.3	1.7037
14	464.9	458.9	460.5	466.0	462.6	3.4131
15	453.2	453.4	455.5	452.6	453.7	1.2633
16	458.8	458.1	458.7	462.1	459.4	1.8099
17	462.9	461.6	461.8	463.9	462.6	1.0661
18*	479.1	478.8	456.5	455.1	467.4	13.3784
19	460.6	460.4	460.5	460.6	460.5	0.0957
20	455.3	455.1	458.7	457.0	456.5	1.6820

* means Cochran outliers

Table 8 Results of EC-3

Lab	Day 1		Day 2		Mean g/kg	Std. Dev.
	A	B	A	B		
1	461.7	467.9	460.3	463.3	463.3	3.3025
2	458.1	457.5	464.3	463.3	460.8	3.4967
3	455.0	453.4	454.4	454.1	454.2	0.6652
4	479.3	480.2	479.2	480.4	479.8	0.6557
5*	459.5	457.9	465.2	469.0	462.9	5.1335
6*	476.7	460.1	458.9	467.1	465.7	8.1764
7	459.5	461.8	457.4	458.3	459.3	1.9053
8	460.9	459.5	461.5	460.8	460.7	0.8421
9	456.4	456.4	457.0	457.7	456.9	0.6185
10	459.3	457.6	456.8	457.2	457.7	1.0996
11	461.7	463.2	458.4	461.5	461.2	2.0149
12	459.6	459.7	461.3	459.9	460.1	0.7932
13	461.8	467.5	465.3	464.8	464.9	2.3473
14	461.4	461.6	459.8	463.7	461.6	1.6008
15	455.6	457.2	455.0	455.8	455.9	0.9309
16	456.8	465.1	461.1	461.6	461.2	3.4025
17	460.3	460.9	459.1	461.6	460.5	1.0595
18*	479.5	479.1	461.2	461.0	467.4	13.3784
19	463.1	463.0	462.2	462.6	462.7	0.4113
20	459.1	459.3	458.9	461.4	459.7	1.1615

* means Cochran outliers

Table 9 Results of all 5 batches

Lab No.	Trifluralin TC-1		Trifluralin TC-2		Trifluralin EC-1		Trifluralin EC-2		Trifluralin EC-3	
	D 1	D 2	D1	D 2	D 1	D 2	D 1	D 2	D 1	D 2
1	987.9	984.1	982.7	981.9	461.1	461.1	460.6	458.2	464.8	461.8
2	981.3	987.2	972.3	976.2	457.0	467.4	459.5	463.5	457.8	463.8
3	979.0	978.6	977.6	978.3	453.7	453.6	452.9	453.4	454.2	454.3
4	965.8	966.0	965.8	965.3	481.6	481.4	480.0	477.5	479.8	479.8
5	1007.8	981.8	969.7	981.1	460.7	472.8	465.4	473.2	458.7	467.1
6	984.6	987.6	981.4	994.7	459.0	460.5	456.8	458.5	468.4	463.0
7	979.4	984.2	980.8	983.3	461.7	457.9	461.4	460.4	460.7	457.9
8	986.4	985.2	986.0	985.8	462.1	461.8	457.0	459.4	460.2	461.2

9	981.7	975.0	980.8	972.6	454.5	452.6	454.8	453.1	456.4	457.4
10	977.1	984.5	978.0	976.8	454.1	457.7	459.9	462.0	458.5	457.0
11	988.0	984.6	988.7	982.0	457.8	458.0	462.2	452.5	462.5	460.0
12	979.2	980.3	979.8	981.4	459.0	458.6	458.3	458.7	459.7	460.6
13	985.2	985.1	989.7	990.5	465.9	466.0	462.5	462.2	464.7	465.1
14	968.0	964.2	961.7	967.7	461.3	460.4	461.9	463.3	461.5	461.8
15	983.4	983.9	982.0	983.3	455.8	456.1	453.3	454.1	456.4	455.4
16	982.8	982.3	981.3	983.3	457.0	460.6	458.5	460.4	461.0	461.4
17	986.0	986.3	985.7	985.6	459.4	462.3	462.3	462.9	460.6	460.4
18	976.0	973.5	978.4	976.0	477.6	453.9	479.0	455.8	479.3	461.1
19	983.0	982.9	982.8	982.8	461.2	461.8	460.5	460.6	463.1	462.4
20	989.1	985.4	984.9	982.8	455.0	456.8	455.2	457.9	459.2	460.2

Table 10 Results of Mean values of all 5 batches

Lab No	Trifluralin TC-1	Trifluralin TC-2	Trifluralin EC-1	Trifluralin EC-2	Trifluralin EC-3
1	986.0	982.3	461.1	459.4	463.3
2	984.2	974.3	462.2	461.5	460.8
3	978.8	977.9	453.6	453.2	454.2
4	965.9	965.5	481.5	478.7	479.8
5	994.8	975.4	466.7	469.3	462.9
6	986.1	988.0	459.8	457.7	465.7
7	981.8	982.3	459.8	460.9	459.3
8	985.8	985.9	462.0	458.2	460.7
9	978.4	976.7	453.6	453.9	456.9
10	980.8	977.4	455.9	460.9	457.7
11	986.3	985.4	457.9	457.3	461.2
12	979.7	980.6	458.8	458.5	460.1
13	985.1	990.1	465.9	462.3	464.9
14	966.1	964.7	460.8	462.6	461.6
15	983.7	982.6	455.9	453.7	455.9
16	982.6	982.3	458.8	459.4	461.2
17	986.1	985.6	460.8	462.6	460.5
18	977.2	974.8	465.7	470.2	467.4

19	982.9	982.8	461.5	460.5	462.7
20	987.2	983.8	455.9	456.5	459.7

Table 11 Summary of the statistical evaluation (20 labs)

	Trifluralin TC-1	Trifluralin TC-2	Trifluralin EC-1	Trifluralin EC-2	Trifluralin EC-3
x_m [g/kg]	981.96	979.91	460.91	460.86	461.82
x_m [%]	98.20	97.99	46.09	46.09	46.18
L	20	20	20	20	20
S_r	4.52	4.50	4.54	3.33	4.06
S_R	7.79	7.76	7.28	6.74	6.37
r	12.67	12.60	12.71	9.334	11.37
R	21.80	21.72	20.37	18.86	17.82
RSD_R	0.79	0.79	1.58	1.46	1.38
RSD_R （Hor）	1.42	1.42	1.59	1.59	1.59
HorRat	0.56	0.56	0.99	0.92	0.87

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 20 participants:
Fig. 1 Trifluralin tech. sample - 1 (Trifluralin TC-1)
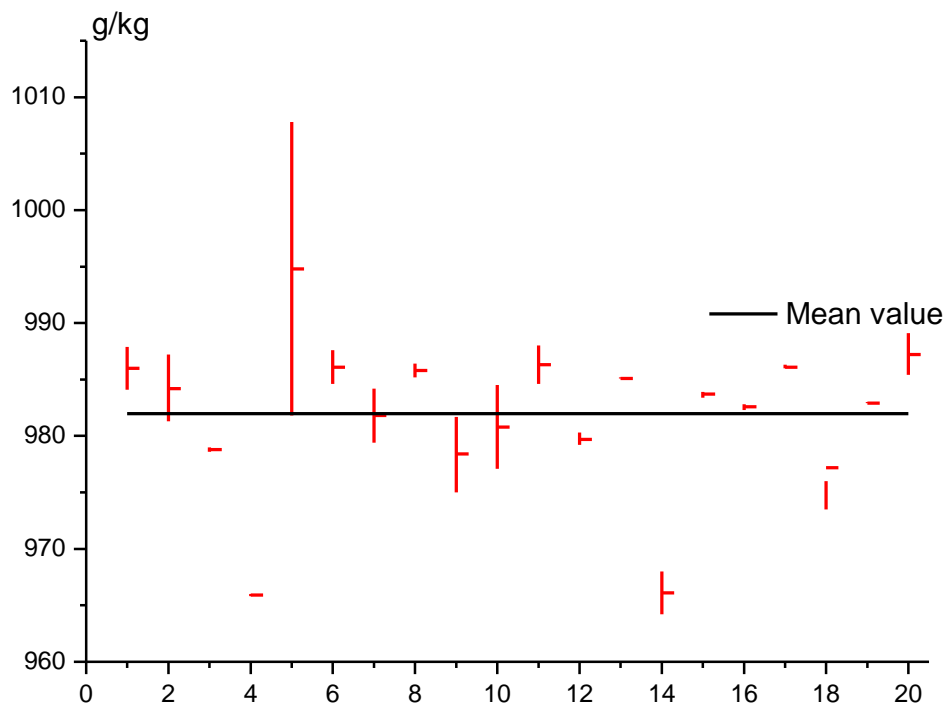
Mean value:	981.96 g/kg
S_r:	4.52
S_R:	7.79
RSD_R:	0.79
$RSD_{R (Hor)}$:	1.42
HorRat:	0.56
Outlier (Grubbs): none	
Outlier (Cochran): lab 5	

Fig. 2 Trifluralin tech. sample - 2 (Trifluralin TC-2)
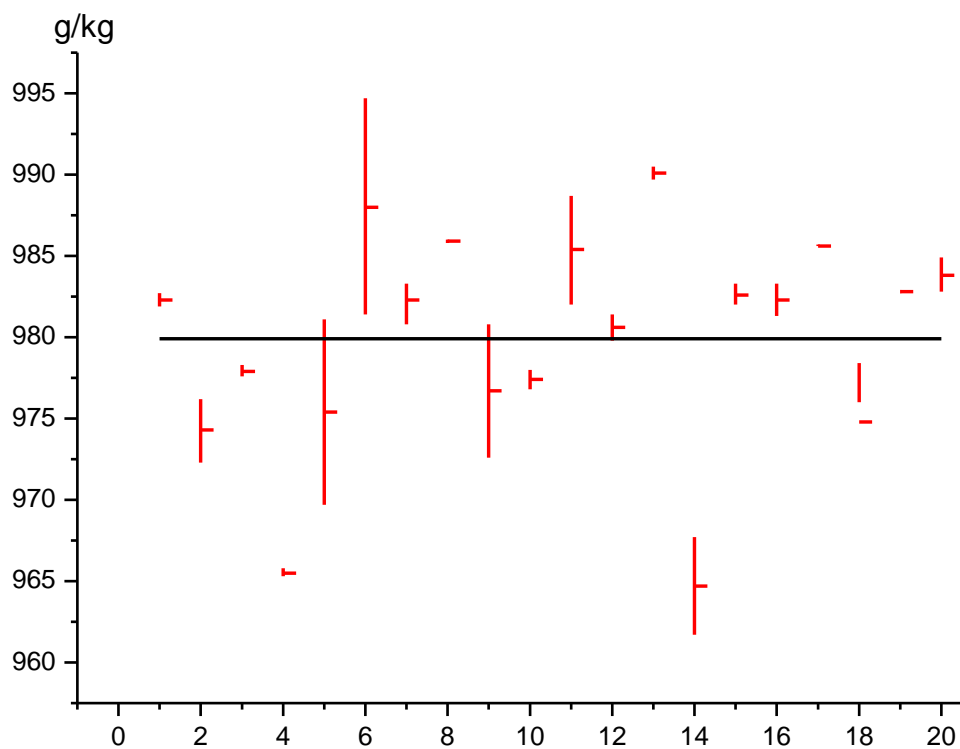
Mean value:	979.91 g/kg
S_r:	4.5
S_R:	7.76
RSD_R:	0.79
$RSD_{R\,(Hor)}$:	1.42
HorRat:	0.56
Outlier (Grubbs): none	
Outlier (Cochran): lab 5 and 6	

Fig. 3 Trifluralin EC sample - 1 (Trifluralin EC-1)
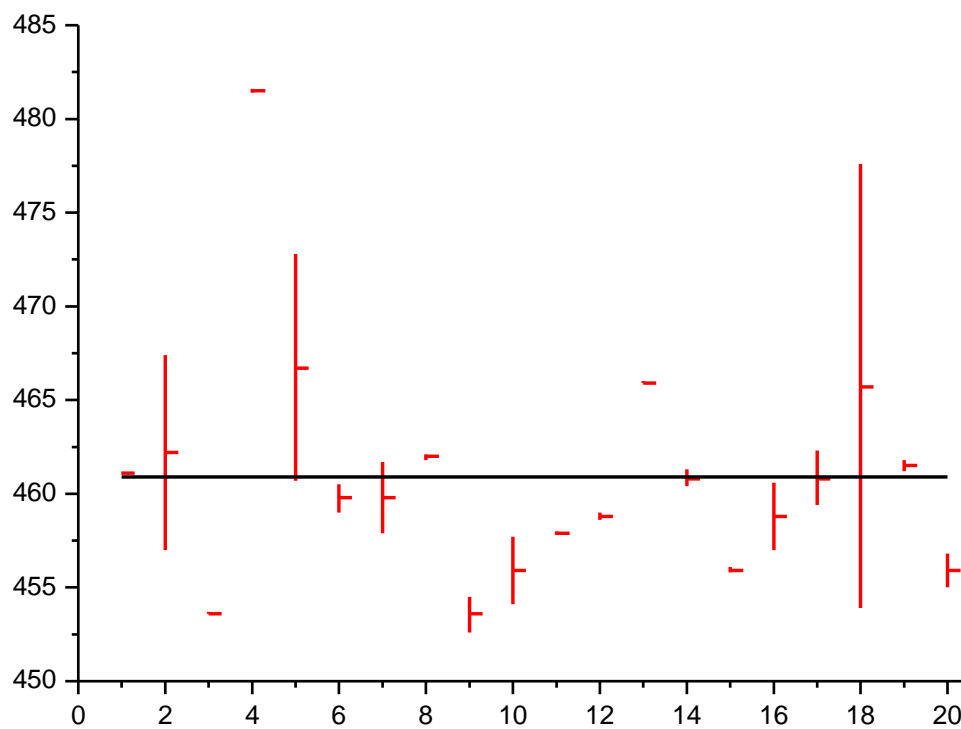
Mean value: 460.91 g/kg
S_r: 4.54
S_R: 7.28
RSD_R: 1.58
$RSD_{R (Hor)}$:   1.59
HorRat: 0.99
Outlier (Grubbs): lab 4
Outlier (Cochran): lab 2, 5 and 18

Fig. 4 Trifluralin EC sample - 2 (Trifluralin EC-2)
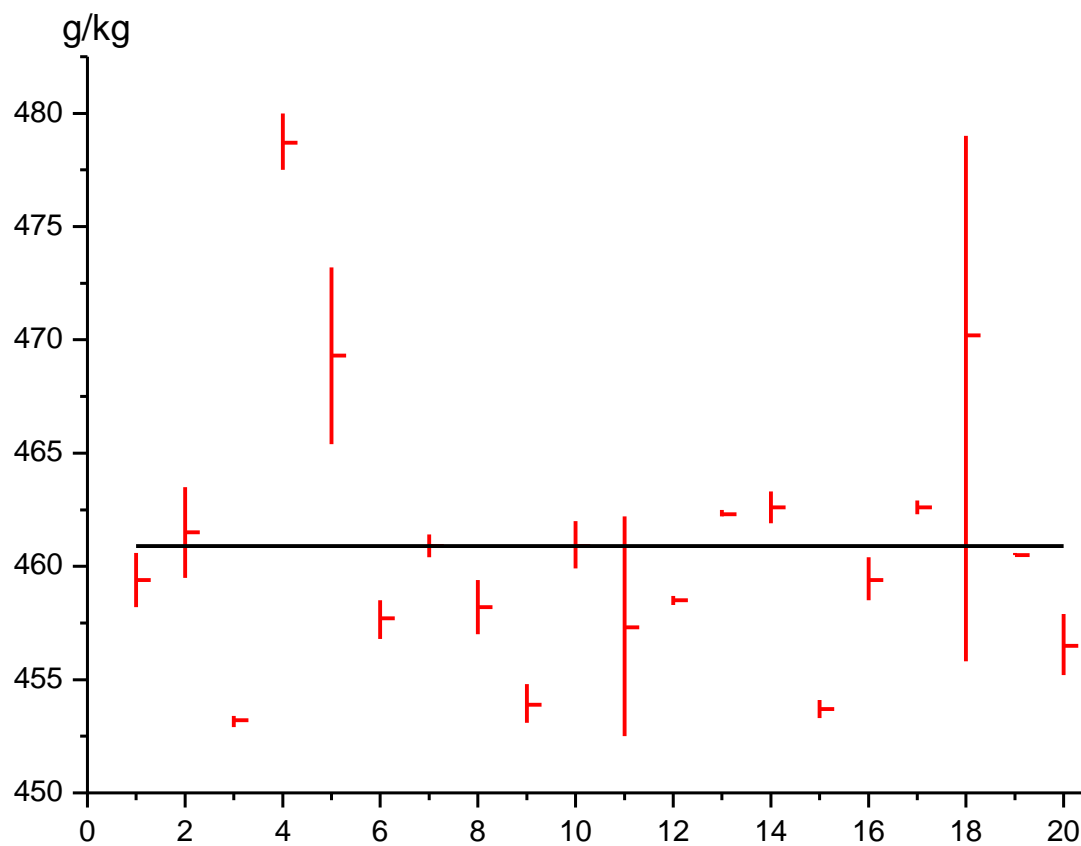
Mean value: 460.86 g/kg

S_r: 3.33

S_R: 6.74

RSD_R: 1.46

$RSD_{R(Hor)}$:   1.59

HorRat: 0.92

Outlier (Grubbs): lab 4

Outlier (Cochran): lab 5, 11 and 18

Fig. 5 Trifluralin EC sample - 3 (Trifluralin EC-3)
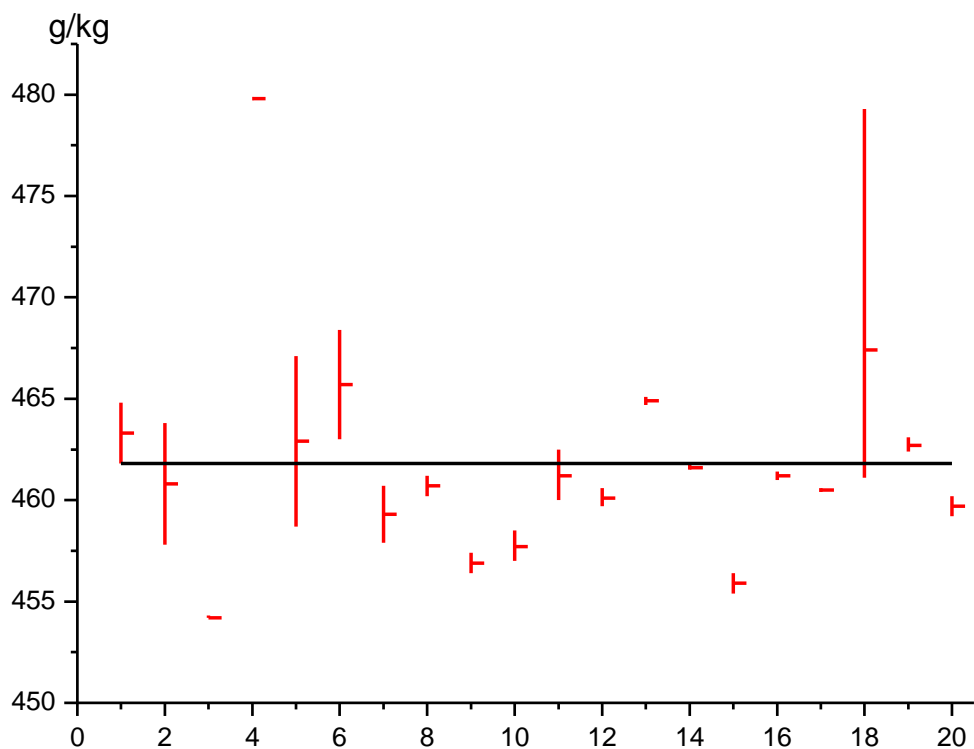
Mean value: 461.82 g/kg
S_r: 4.06
S_R: 6.37
RSD_R: 1.38
$RSD_{R (Hor)}$: 1.59
HorRat: 0.87
Outlier (Grubbs): lab 4
Outlier (Cochran): lab 5, 6 and 18

Table 12 Summary of the statistical evaluation (without outliers)

	Trifluralin TC-1	Trifluralin TC-2	Trifluralin EC-1	Trifluralin EC-2	Trifluralin EC-3
x_m [g/kg]	981.2	979.8	458.9	458.9	460.0
x_m [%]	98.12	97.98	45.89	45.89	46.00
L	19	18	16	16	16
S_r	2.81	2.70	1.60	1.79	1.89
S_R	6.75	7.04	3.58	3.52	3.21
r	7.86	7.55	4.47	5.00	5.30
R	18.89	19.70	10.01	9.84	8.99
RSD_R	0.69	0.72	0.78	0.77	0.70
$RSD_{R (Hor)}$	1.42	1.42	1.59	1.59	1.59
HorRat	0.48	0.51	0.49	0.48	0.44

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

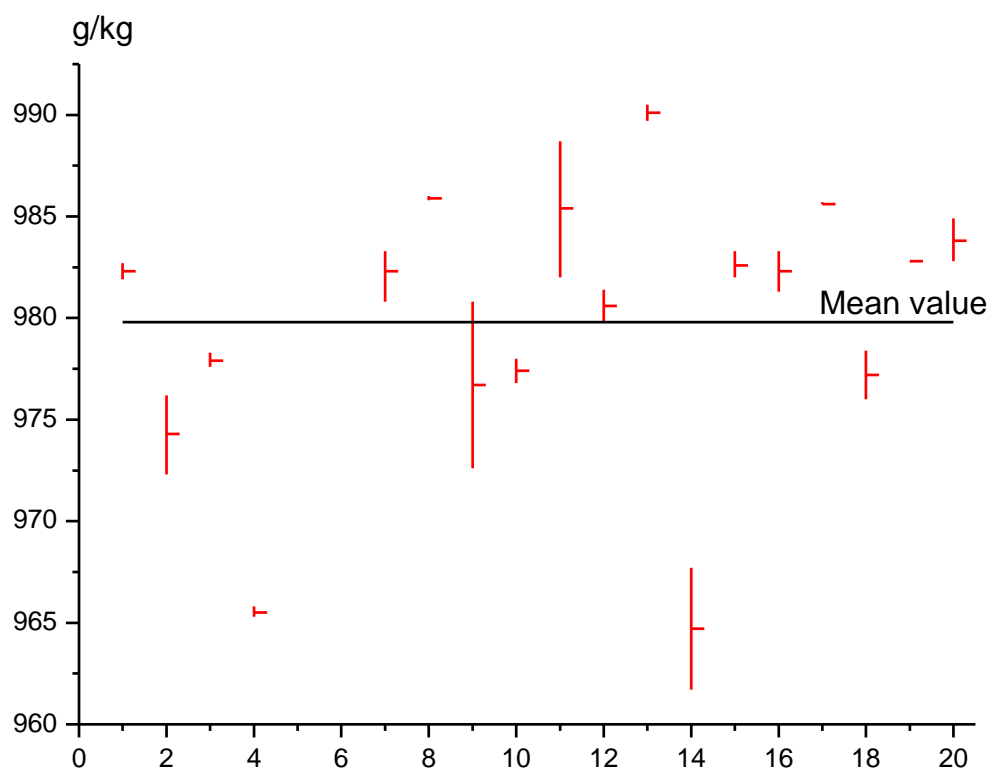$RSD_{R (Hor)}$ = Horwitz Value according to Horwitz equation

HorRat = Horwitz Ratio (RSD_R / $RSD_{R (Hor)}$)

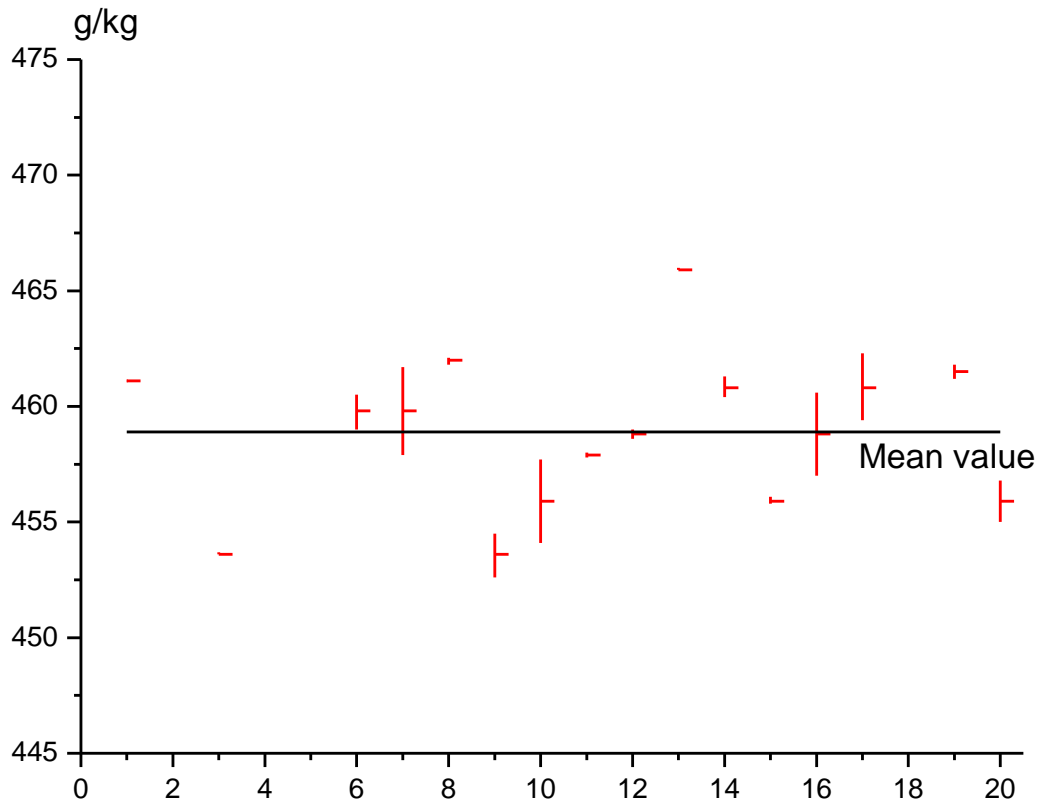Fig. 6 Trifluralin tech. sample - 1 (Trifluralin TC-1)
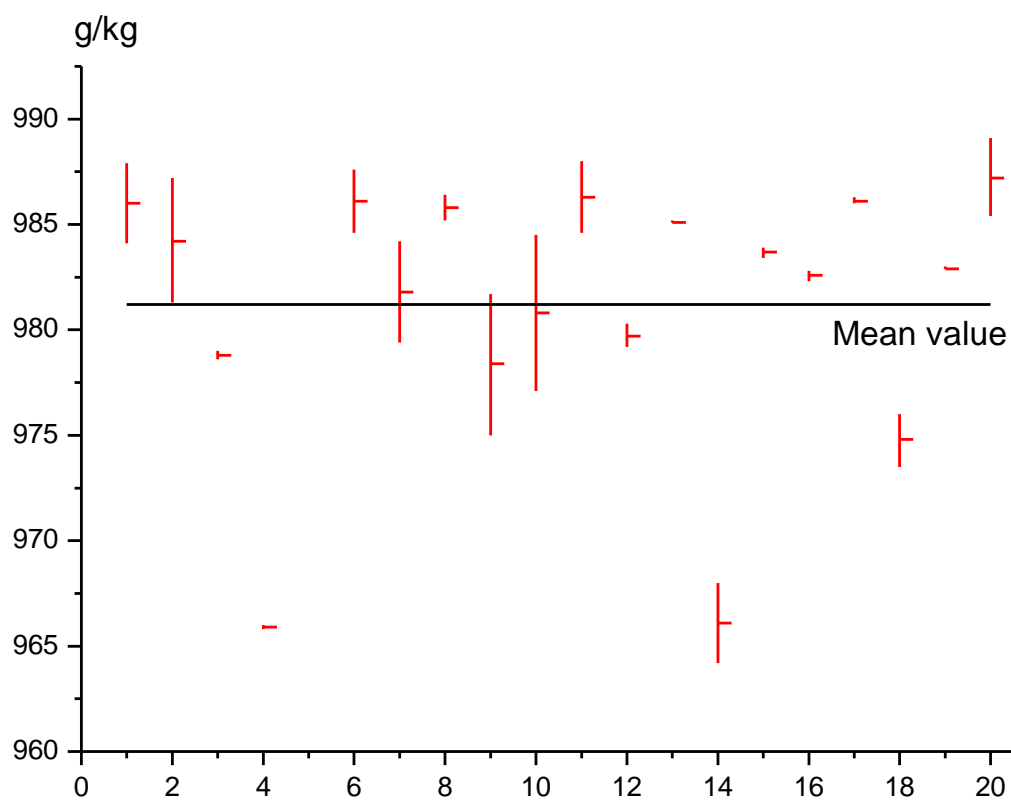
Mean value:	981.2 g/kg
S_r:	2.81
S_R:	6.75
RSD_R:	0.69
$RSD_{R (Hor)}$:	1.42
HorRat:	0.48
Outlier: none	

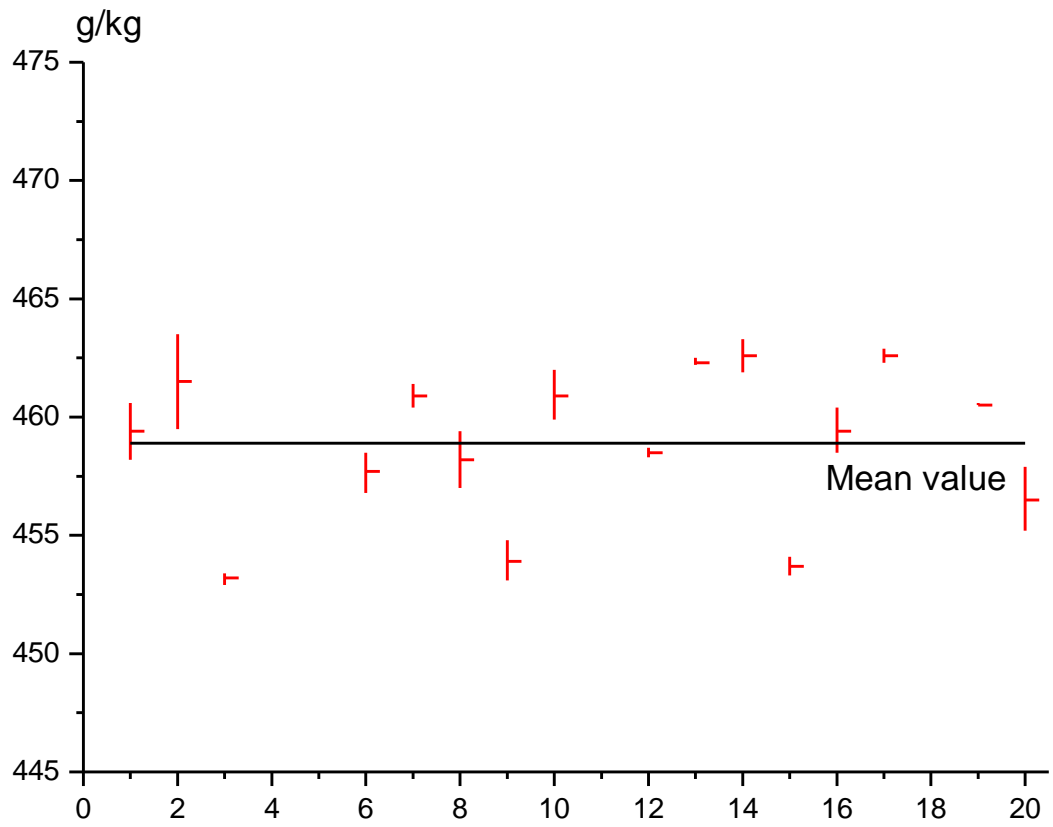Fig. 7 Trifluralin tech. sample - 2 (Trifluralin TC-2)

Mean value: 979.8 g/kg

S_r: 2.70

S_R: 7.04

RSD_R: 0.72

$RSD_{R (Hor)}$: 1.42

HorRat: 0.51

Outlier: none

Fig. 8 Trifluralin EC sample - 1 (Trifluralin EC-1)

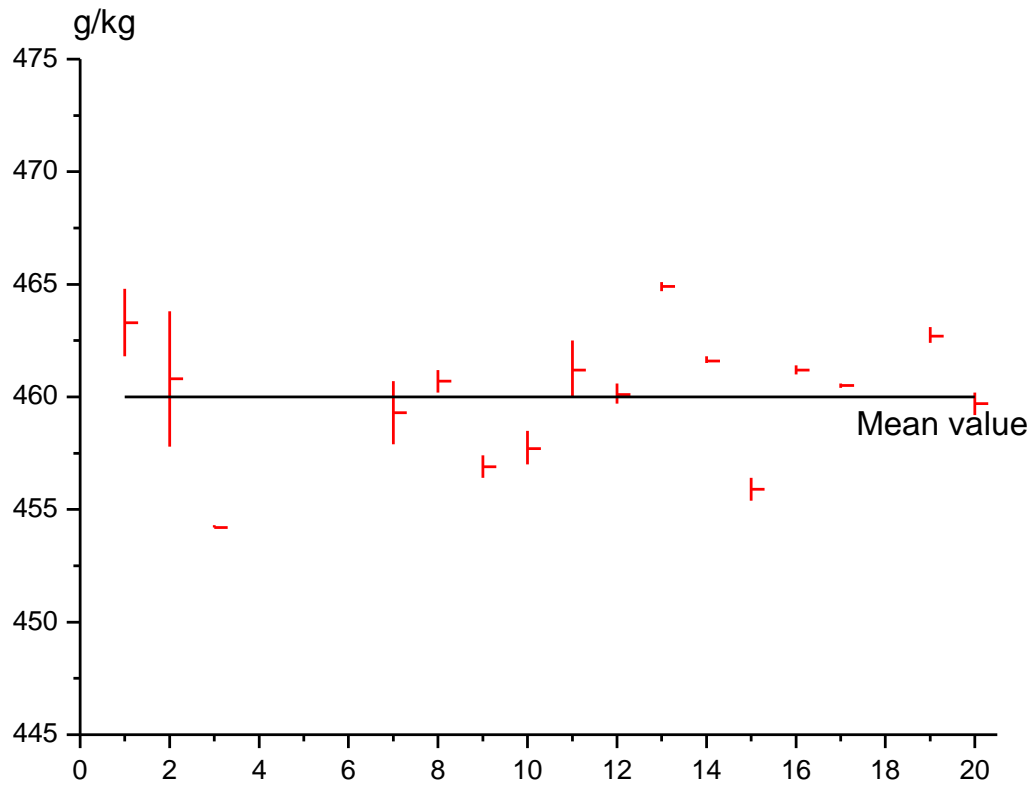Mean value: 458.9 g/kg
S_r: 1.60
S_R: 3.58
RSD_R: 0.78
$RSD_{R (Hor)}$: 1.59
HorRat: 0.49
Outlier: none

Fig. 9 Trifluralin EC sample - 2 (Trifluralin EC-2)

Mean value:	458.9 g/kg
S_r:	1.79
S_R:	3.52
RSD_R:	0.77
$RSD_{R\,(Hor)}$:	1.59
HorRat:	0.48
Outlier: none	

Fig. 10 Trifluralin EC sample - 3 (Trifluralin EC-3)

Mean value: 460.0 g/kg
S_r: 1.89
S_R: 3.21
RSD_R: 0.70
$RSD_{R (Hor)}$: 1.59
HorRat: 0.44
Outlier: none

7. Conclusions

A total of 20 laboratories have participated in this full scale collaborative study. And data from 20 participants have been included in the final data statistical. 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 LC method is acceptable and suitable to produce reproducible results.

SYRICIT considers this method to be suitable for the intended purpose and recommends accepting it as a provisional CIPAC method for the determination of Trifluralin in TC and EC formulation.