Pydiflumetofen

Collaborative study

Small scale collaborative study for the determination of

PYDIFLUMETOFEN

in TC, SC and EC formulated material by LC

Report to CIPAC

By

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1. Participants

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

Company / Lab	Contact	Country
BASF, Limburger Hof	Simone Fuessl	Germany
Bayer, Leverkusen	Dirk Hoffmann	Germany
CRA-W	Marie Baes	Belgium
Eurofins	Christian Hemm	Germany
Fera	Andrew Plumb	UK
Syngenta, Münchwilen	Andreas Herdlitschka	Switzerland
UKZUZ	Hana Slampova	Czech Republic

2. General Information

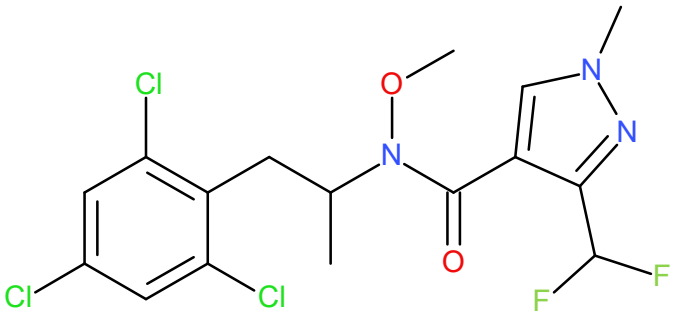
ISO common name: Pydiflumetofen

IUPAC name: 3-(difluoromethyl)-N-methoxy-1-methyl-N-[1-(2,4,6-trichlorophenyl)propan-2-yl]-1H-pyrazole-4-carboxamide

Molecular mass: 426.7 g mol^{-1}

Empirical formula: C16 H16 Cl3 F2 N3 O2

Structure:



3. Samples

In total five samples – one TC, two SC and two EC formulated materials have been shipped together with reference standard.

- Pydiflumetofen TC– sample A
- Pydiflumetofen SC– sample B
- Pydiflumetofen SC– sample C
- Pydiflumetofen EC – sample D
- Pydiflumetofen EC – sample E
- Pydiflumetofen reference standard (purity 99.7 %w/w)

4. Method scope

The method is set up to determine the content of Pydiflumetofen. The sample is dissolved in acetonitrile and quantification is done against external standard, by liquid chromatography using UV detection at 230 nm.

5. Procedure

Each sample was analyzed using four independent determinations: Two sample preparations double injected, analyzed on two different days.

6. Remarks

In table 1 the instruments, columns and chromatographic conditions noted by the participating laboratories are given. Labs 3 and 7 used different dimensions of the column. Lab 3 changed the Flow rate and the injection volume to compensate for the lower inner diameter.

Table 1: Chromatographic conditions used by the participants.

Lab	Instrument	Stationary phase (particle size, type)	length, diameter [mm]	Flow rate [mL/min]	Injection volume [µL]
1	Agilent 1260 Infinity II	Kinetex C18	100 mm x 4.6 mm	1.5	5
2	Thermo Ultimate 3000 RS	Kinetex C18	100 mm x 4.6 mm	1.5	5
3	Agilent 1290 Infinity I	Kinetex C18	150 mm x 2.1 mm	0.4	1
4		Kinetex C18	100 mm x 4.6 mm	1.5	5
5	Waters Alliance	Kinetex C18	100 mm x 4.6 mm	1.5	5
6	Waters Acquity UPLC H Class	Kinetex C18	100 mm x 4.6 mm	1.5	5
7	Agilent 1260 Infinity II	Kinetex C18	50 mm x 4.6 mm	1.5	5

7. Evaluation and Discussion

Data Review

In a first approach all deviations noted by the participating laboratories were deemed not to affect the analytical results. Therefore, all data sets were included within the statistical assessment. In a second attempt only the laboratories using the conditions outlined in the method were considered and in a third approach a statistical outlier has been excluded.

Statistical results

In tables 2 to 6 and the figures 1 to 7 the full set of analytical results of all participating laboratories are shown.

Table 2: Results of the different laboratories for Sample A (TC).

Laboratory no.	**1**	**2**	**3**	**4**	**5**	**6**	**7**
Assay [g/kg] day 1	1000.32	994.71	1002.75	997.99	1000.76	990.83	1018.72
Assay [g/kg] day 2	993.44	999.67	1006.00	999.34	1001.19	990.10	1029.86
mean	996.88	997.19	1004.38	998.67	1000.98	990.47	1024.29

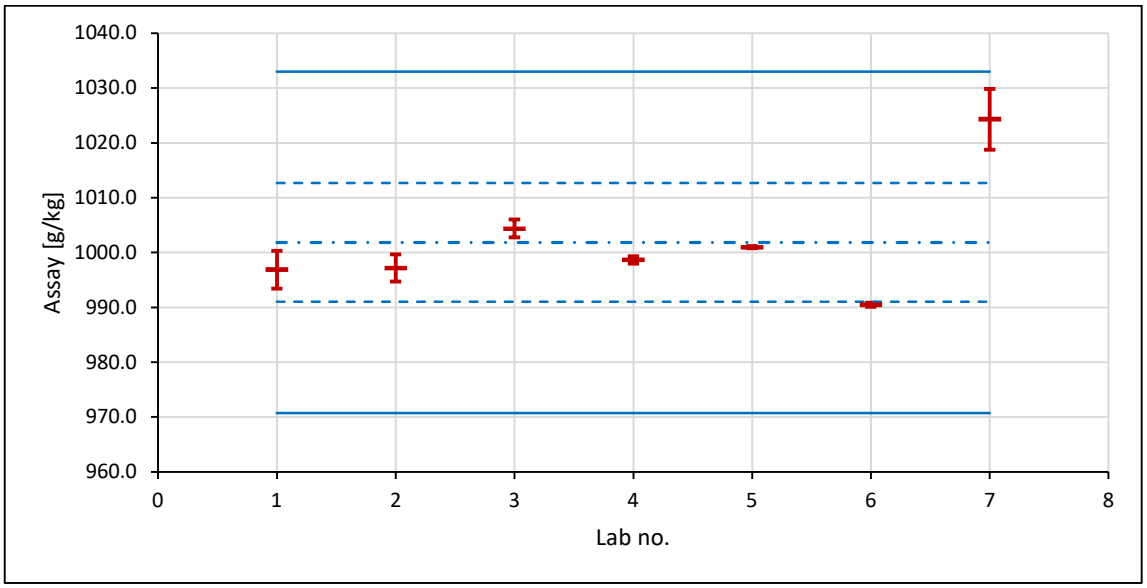


Figure 1: Graphical presentation of the results of the different laboratories for Sample A (TC). For each laboratory (laboratories 1 to 7) the red bars represent day 1, day 2 as well as the average for each lab. Results of laboratory 7 is identified to be an outlier.

Table 3: Results of the different laboratories for Sample B (SC).

Laboratory no.	**1**	**2**	**3**	**4**	**5**	**6**	**7**
Assay [g/kg] day 1	176.58	178.68	179.65	178.00	178.30	173.61	181.82
Assay [g/kg] day 2	174.69	178.95	180.30	178.90	178.60	172.86	184.29
mean	175.63	178.82	179.98	178.45	178.45	173.24	183.05

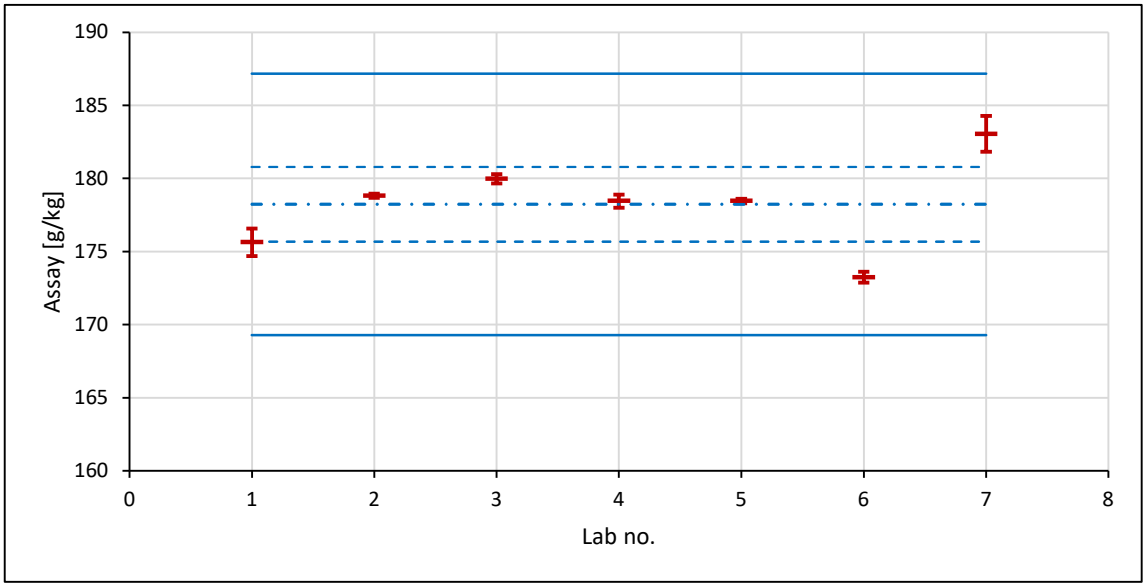


Figure 2: Graphical presentation of the results of the different laboratories for Sample B (SC). For each laboratory (laboratories 1 to 7) the red bars represent day 1, day 2 as well as the average for each lab.

Table 4: Results of the different laboratories for Sample C (SC).

Laboratory no.	**1**	**2**	**3**	**4**	**5**	**6**	**7**
Assay [g/kg] day 1	183.80	184.78	184.60	183.80	184.80	179.65	184.68
Assay [g/kg] day 2	184.65	183.73	185.40	184.80	183.70	179.97	183.72
mean	184.22	184.26	185.00	184.30	184.25	179.81	184.20
s_i	0.36	0.54	0.32	0.50	0.61	0.05	0.47

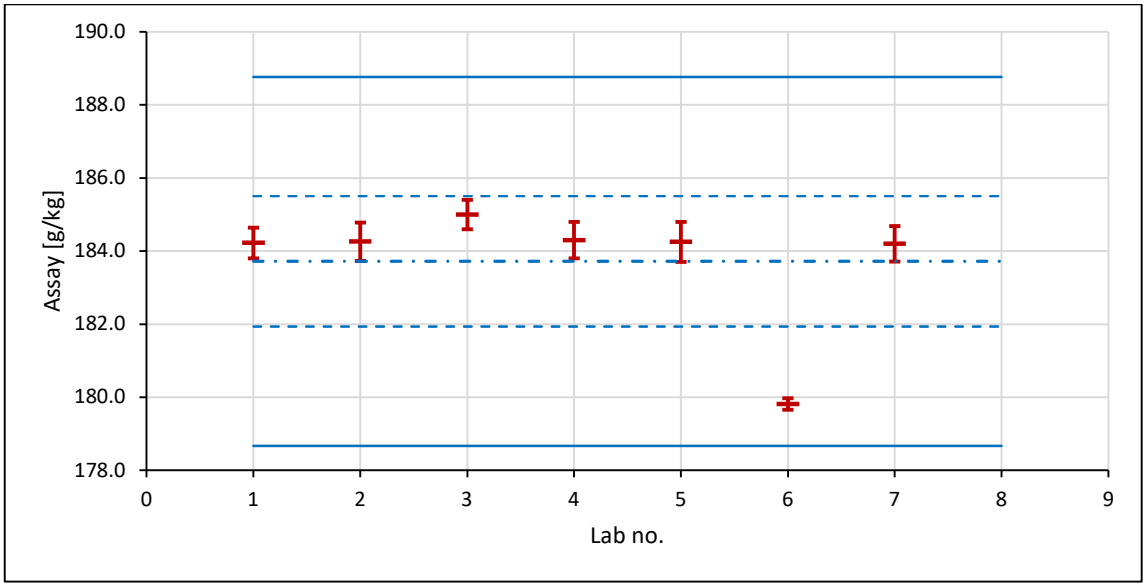


Figure 3: Graphical presentation of the results of the different laboratories for Sample C (SC). For each laboratory (laboratories 1 to 7) the red bars represent day 1, day 2 as well as the average for each lab. Results of laboratory 6 have been identified as outlier.

Table 5: Results of the different laboratories for Sample D (EC).

Laboratory no.	1	2	3	4	5	6	7
ıy [g/kg] day 1	56.86	57.25	60.40	57.30	56.80	57.63	57.38
ıy [g/kg] day 2	57.00	57.30	59.00	57.40	56.40	57.61	56.69
mean	56.93	57.28	59.70	57.35	56.60	57.62	57.04
	0.01	0.00	0.98	0.01	0.08	0.00	0.24

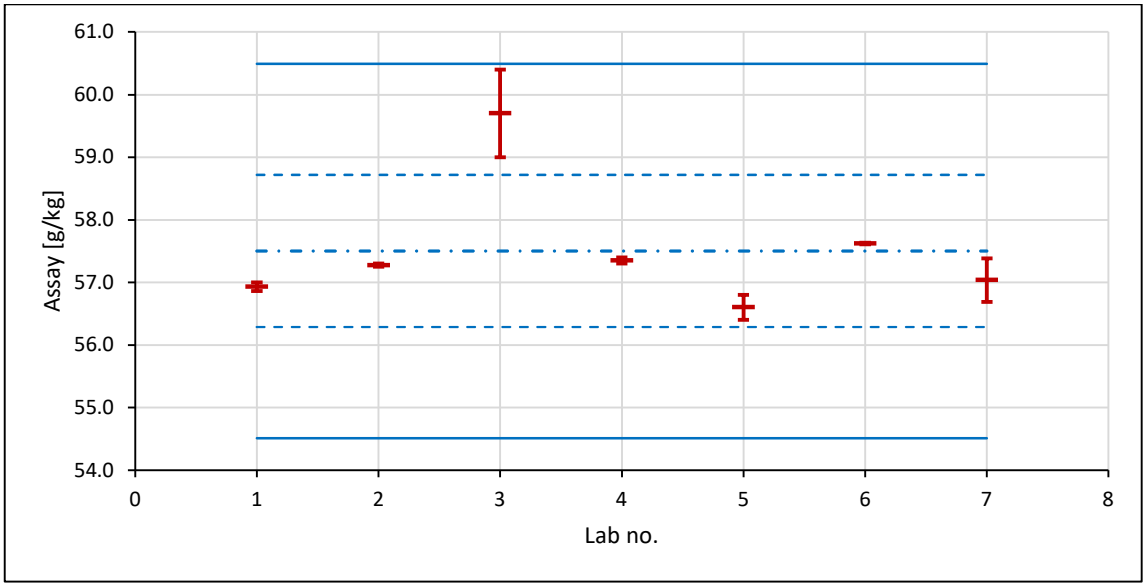


Figure 4: Graphical presentation of the results of the different laboratories for Sample D (EC). For each laboratory (laboratories 1 to 7) the red bars represent day 1, day 2 as well as the average for each lab. Results of laboratory 3 have been identified as outlier.

Table 6: Results of the different laboratories for Sample E (EC)

1	2	3	4	5	6	7
56.58	56.89	58.80	56.90	56.40	57.90	56.34
56.94	56.95	58.50	57.30	56.20	57.70	56.44
56.76	56.92	58.65	57.10	56.30	57.80	56.39
0.06	0.00	0.04	0.08	0.02	0.02	0.01

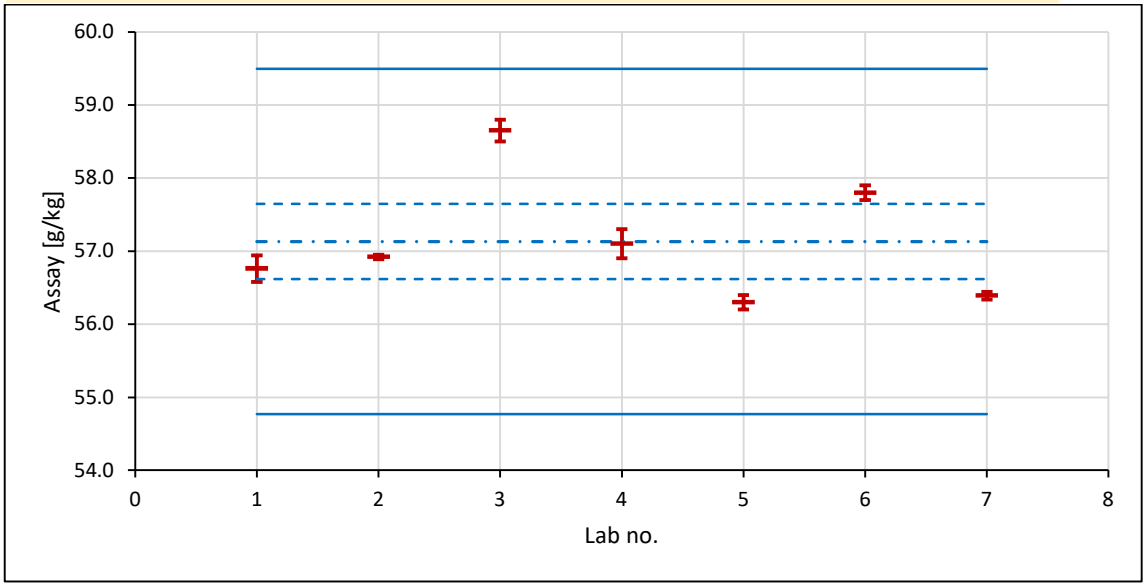


Figure 5: Graphical presentation of the results of the different laboratories for Sample E (EC). For each laboratory (laboratories 1 to 7) the red bars represent day 1, day 2 as well as the average for each lab.

Table 7: Overall statistics on all submitted results:

Statistics	x_m	L	N	s_r	s_L	s_R	r	R	RSD_R	$RSD_{R\ (Hor)}$	HorRat
SAMPLE A	1001.83	7	14	3.87	10.42	11.12	10.82	31.13	1.11	2.00	0.56
SAMPLE B	178.23	7	14	0.91	3.06	3.20	2.56	8.95	1.79	2.59	0.69
SAMPLE C	183.72	7	14	0.64	1.69	1.80	1.78	5.05	0.98	2.58	0.38
SAMPLE D	57.50	7	14	0.43	0.98	1.07	1.21	2.99	1.86	3.07	0.60
SAMPLE E	57.13	7	14	0.18	0.82	0.84	0.52	2.36	1.48	3.08	0.48

Even without elimination of outliers or stragglers, the between laboratory experimental Relative Reproducibility Standard Deviation (RSDR) is below the acceptance limit based on the Horwitz curve calculation (RSDR(Hor)) for all samples.

Table 8: Statistics after elimination of the results of SAMPLE A from laboratory 7, SAMPLE C from laboratory 6, as well as SAMPLE D, laboratory 3:

Statistics	x_m	L	N	s_r	s_L	s_R	r	R	RSD_R	$RSD_{R\ (Hor)}$	HorRat
SAMPLE A	998.09	6	12	2.66	4.26	5.03	7.45	14.07	0.50	2.00	0.25
SAMPLE B	178.23	7	14	0.91	3.06	3.20	2.56	8.95	1.79	2.59	0.69
SAMPLE C	184.37	6	12	0.68	0.00	0.68	1.91	1.91	0.37	2.58	0.14
SAMPLE D	57.14	6	12	0.24	0.32	0.39	0.66	1.11	0.69	3.08	0.22
SAMPLE E	57.13	7	14	0.18	0.82	0.84	0.52	2.36	1.48	3.08	0.48

Xm	Overall mean assay [g/kg]
L	Number of participating labs
N	Number of results
sr	Repeatability standard deviation
sL	Interlaboratory standard deviation
sR	Reproducibility standard deviation
r	Repeatability value, r = 2.8 * sr
R	Reproducibility value, R = 2.8 * sR
RSDR	Reproducibility relative standard deviation
RSDR (Hor)	Horwitz limit
HorRat	Horwitz ratio

8. Summary and Conclusion

A total of 7 laboratories from DAPA and ESPAC participated in the trial, came back in time and provided results. The data sets from all these laboratories have been considered for the statistical evaluation (Figure 1 to 5 and Tables 2 to 7). results of SAMPLE A from laboratory 7, SAMPLE C from laboratory 6, as well as SAMPLE D, laboratory 3. In all cases shown in Tables 7 and 8 the Horrat is well below 1.

Syngenta considers this method to be suitable for the intended purpose and recommends going for a full collaborative trial for the determination of Pydiflumetofen in TC as well as SC and EC formulated material.