Flumetnicam

Collaborative study

Full scale collaborative study for the determination of

FLUMETNICAM

in TC by LC

Report to CIPAC

By

Syngenta Crop Protection
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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 randomized.

Company / Lab	Contact	Country
Agroest	Ioana Minea	Romania
BASF, Limburger Hof	Simone Fuessl	Germany
Bayer, Frankfurt	Frank Ziemer	Germany
Benaki	Helen Karasali	Greece
CRA-W	Marie Baes	Belgium
Currenta	Michael Haustein	Germany
DAFM	Jim Garvey	Ireland
FMC	Mary Ellen McNally	USA
Syngenta, Münchwilen	Radoslaw Bomba	Switzerland
Syngenta, Goa	Jayan Rappai	India
Syngenta, Greensboro	Ramakrishna Mittapalli	USA

2. General Information

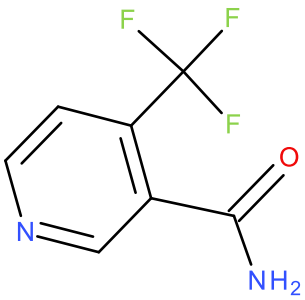
ISO common name: Flumetnicam

IUPAC name: 4-(trifluoromethyl)pyridine-3-carboxamide

Molecular mass: 190.1 g mol^{-1}

Empirical formula: $C_7H_5F_3N_2O$

Structure:



3. Samples

In total five TC samples have been shipped together with reference standard.

- Flumetnicam TC– sample A
- Flumetnicam TC– sample B
- Flumetnicam TC– sample C
- Flumetnicam TC – sample D
- Flumetnicam TC – sample E
- Flumetnicam reference standard (purity 99.5 %w/w)

4. Method scope

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

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 10 and 11 used slightly different columns. Lab 11 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	Kinetex Polar C18, 2.6 µm	150, 4.6	1.0	5
2	Agilent 1100	Kinetex Polar C18, 2.6 µm	150, 4.6	1.0	5
3	Shimadzu prominence	Kinetex Polar C18, 2.6 µm	150, 4.6	1.0	5
4	Agilent 1100	Kinetex Polar C18, 2.6 µm	150, 4.6	1.0	5
5	Shimadzu Nexera 2040	Kinetex Polar C18, 2.6 µm	150, 4.6	1.0	5
6	Agilent 1260 II	Kinetex Polar C18, 2.6 µm	150, 4.6	1.0	5
7	Agilent 1290 II	Kinetex Polar C18, 2.6 µm	150, 4.6	1.0	5
8	Agilent 1290	Kinetex Polar C18, 2.6 µm	150, 4.6	1.0	5
9	Thermo Scientific, DIONEX Ultimate 3000	Kinetex Polar C18, 2.6 µm	150, 4.6	1.0	5
10	Shimadzu LC-20 AB	Phenomenex, Luna C18, 3µm, 100	150, 4.6	1.0	5
11	Thermo Scientific, uHPLC Vanquish Flex	Kinetex Polar C18, 2.6 µm	150, 3.0	0.55	2

Lab 8: 4-(trifluoromethyl)-nicotinamide retained on the column and eluted near the expected RT, but it is eluting near the dead volume.

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 straggler 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	8	9	10	11
Assay [g/kg] day 1	978.62	983.50	980.08	978.70	971.60	983.30	980.70	986.60	976.10	989.20	968.50
Assay [g/kg] day 2	986.23	971.30	970.50	970.60	972.70	982.90	976.83	982.20	980.80	987.40	979.70
mean	982.43	977.40	975.29	974.65	972.15	983.10	978.77	984.40	978.45	988.30	974.10

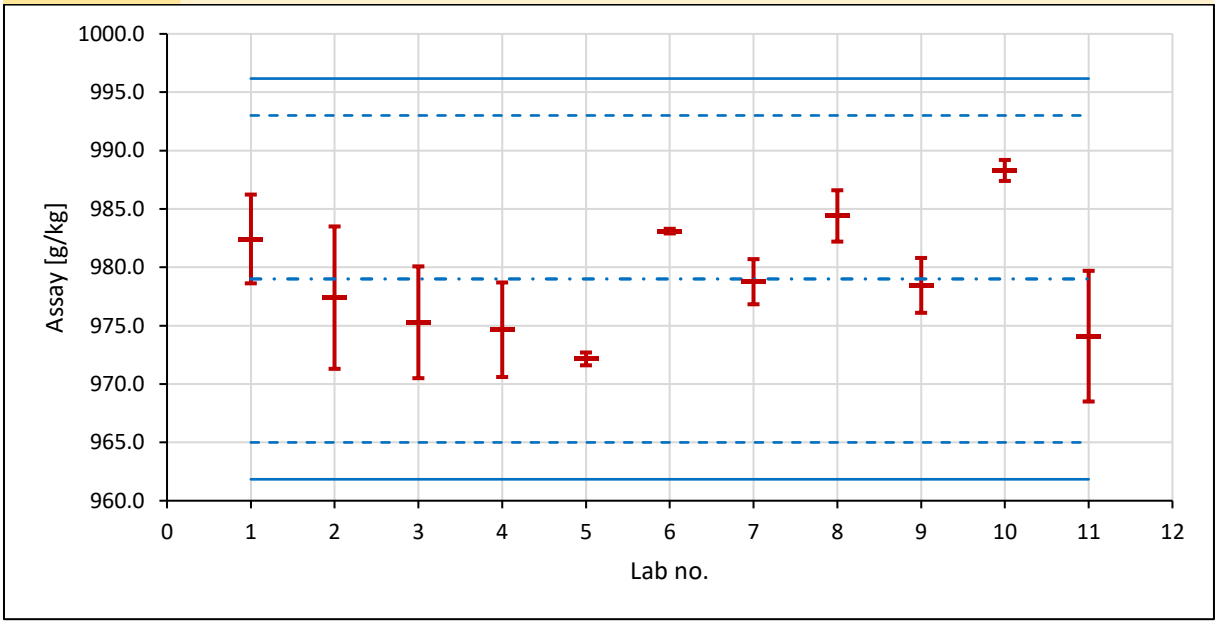


Figure 1: Graphical presentation of the results of the different laboratories for Sample A (TC). For each laboratory (laboratories 1 to 11) the red bars represent day 1, day 2 as well as the average for each lab.

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

Laboratory no.	1	2	3	4	5	6	7	8	9	10	11
Assay [g/kg] day 1	983.72	991.20	984.48	989.80	982.40	988.10	986.80	981.60	988.80	996.90	984.30
Assay [g/kg] day 2	985.90	983.80	992.00	976.60	981.50	986.90	990.25	994.10	989.30	994.60	986.70
mean	984.81	987.50	988.24	983.20	981.95	987.50	988.53	987.85	989.05	995.75	985.50

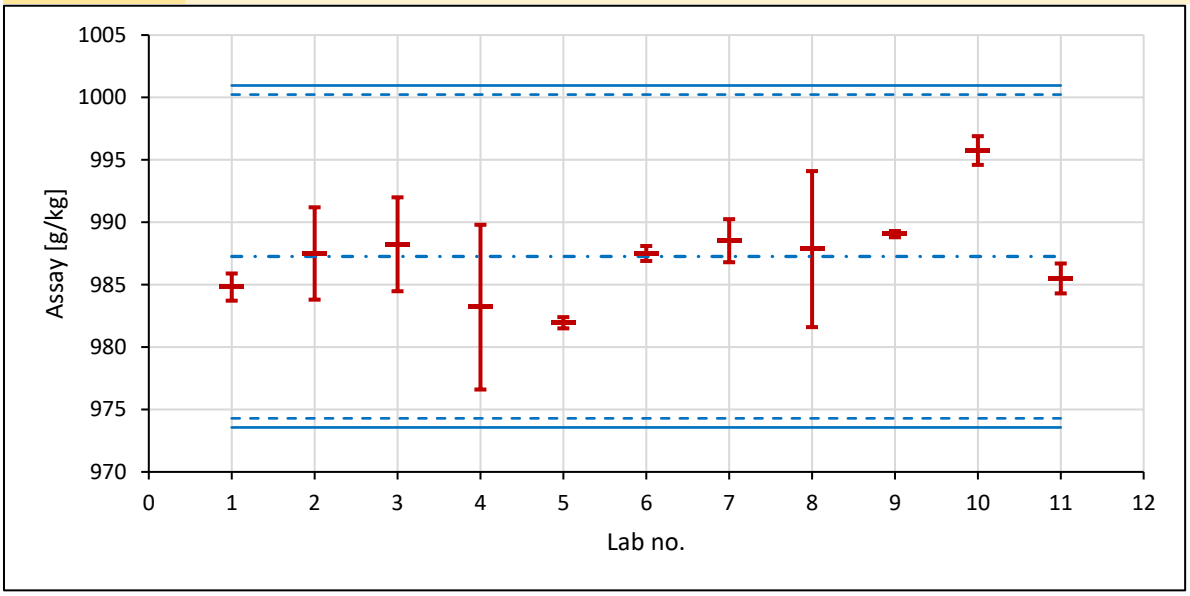


Figure 2: Graphical presentation of the results of the different laboratories for Sample B (TC). For each laboratory (laboratories 1 to 11) the red bars represent day 1, day 2 as well as the average for each lab.

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

Laboratory no.	1	2	3	4	5	6	7	8	9	10	11
Assay [g/kg] day 1	996.41	994.30	995.70	997.20	985.30	997.60	997.80	990.00	999.60	999.60	992.50
Assay [g/kg] day 2	1001.22	990.30	989.80	998.10	984.50	997.20	998.48	994.30	998.00	999.60	998.30
mean	998.81	992.30	992.75	997.65	984.90	997.40	998.14	992.15	998.80	999.60	995.40

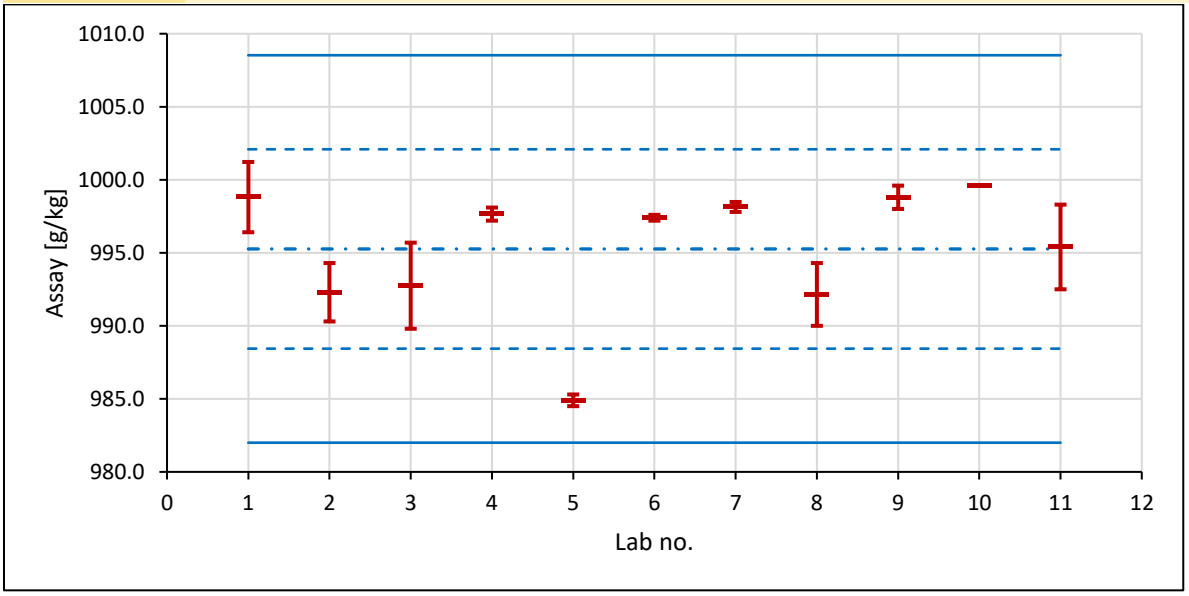


Figure 3: Graphical presentation of the results of the different laboratories for Sample C (TC). For each laboratory (laboratories 1 to 11) the red bars represent day 1, day 2 as well as the average for each lab.

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

Laboratory no.	1	2	3	4	5	6	7	8	9	10	11
Assay [g/kg] day 1	983.32	989.90	990.39	993.30	978.60	993.40	993.80	987.40	996.20	1000.10	982.90
Assay [g/kg] day 2	984.88	991.70	984.80	989.70	979.20	996.00	995.50	986.10	991.70	993.00	987.00
mean	984.10	990.80	987.60	991.50	978.90	994.70	994.65	986.75	993.95	996.55	984.95

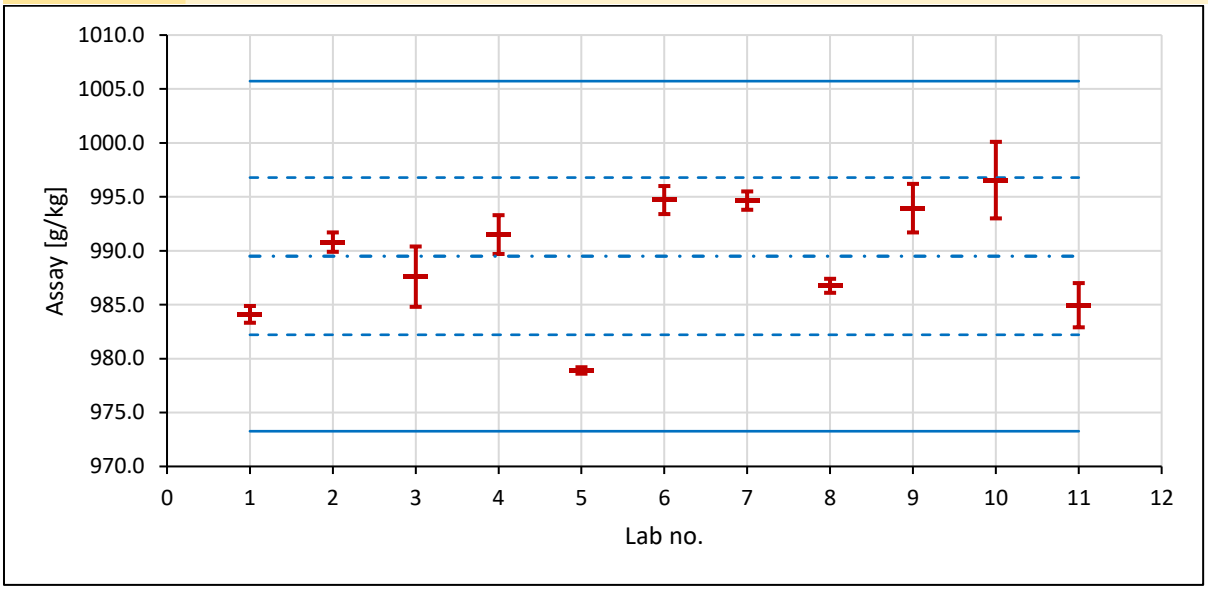


Figure 4: Graphical presentation of the results of the different laboratories for Sample D (TC). For each laboratory (laboratories 1 to 11) the red bars represent day 1, day 2 as well as the average for each lab.

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

Laboratory no.	1	2	3	4	5	6	7	8	9	10	11
Assay [g/kg] day 1	995.58	1006.70	996.61	1003.60	983.30	1003.50	1001.40	996.20	998.50	1010.50	998.16
Assay [g/kg] day 2	1008.29	994.10	995.40	1007.20	986.40	1001.60	1001.00	998.80	999.40	1006.10	997.43
mean	1001.94	1000.40	996.01	1005.40	984.85	1002.55	1001.20	997.50	998.95	1008.30	997.80
s_i	80.72	79.38	0.73	6.48	4.81	1.80	0.08	3.38	0.40	9.68	0.27
Outlier Grubbs											
Straggler Grubbs					X						

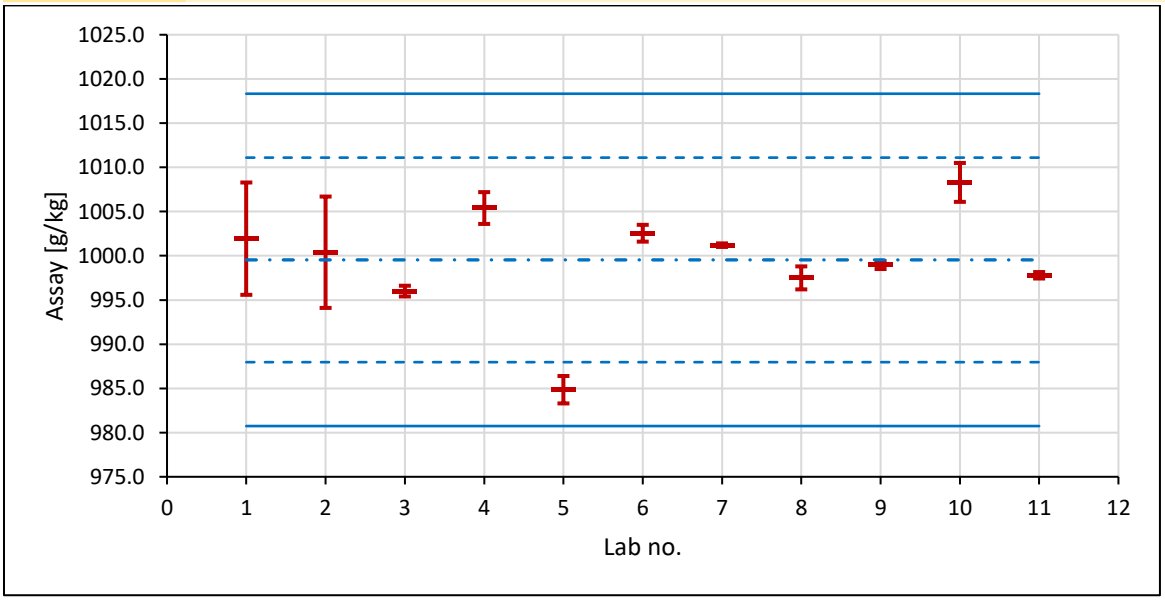


Figure 5: Graphical presentation of the results of the different laboratories for Sample E (TC). For each laboratory (laboratories 1 to 11) the red bars represent day 1, day 2 as well as the average for each lab. Lab 5 is a straggler.

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	979.00	11	22	5.00	3.54	6.13	14.01	17.17	0.63	2.01	0.31
SAMPLE B	987.26	11	22	4.63	1.58	4.89	12.97	13.70	0.50	2.00	0.25
SAMPLE C	995.26	11	22	2.44	4.06	4.74	6.82	13.26	0.48	2.00	0.24
SAMPLE D	989.50	11	22	2.60	5.18	5.80	7.29	16.23	0.59	2.00	0.29
SAMPLE E	999.54	11	22	4.13	5.29	6.71	11.57	18.79	0.67	2.00	0.34

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 E from laboratory 5:

Statistics	x_m	L	N	s_r	s_L	s_R	r	R	RSD_R	$RSD_{R\ (Hor)}$	HorRat
SAMPLE A	979.00	11	22	5.00	3.54	6.13	14.01	17.17	0.63	2.01	0.31
SAMPLE B	987.26	11	22	4.63	1.58	4.89	12.97	13.70	0.50	2.00	0.25
SAMPLE C	995.26	11	22	2.44	4.06	4.74	6.82	13.26	0.48	2.00	0.24
SAMPLE D	989.50	11	22	2.60	5.18	5.80	7.29	16.23	0.59	2.00	0.29
SAMPLE E	1001.00	10	20	4.28	2.27	4.84	11.98	13.55	0.48	2.00	0.24

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 11 laboratories from Asia, Europe and North America 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). The result for Lab 5 SAMPLE E was a straggler for both days. The result was excluded In Table 8. 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 accepting it as a provisional CIPAC method for the determination of Flumetnicam TC.