

REPORT CIPAC/5005/R

CIPAC FULL SCALE COLLABORATIVE STUDY ON THE DETERMINATION OF SILTHIOFAM (N° 635) IN SILTHIOFAM TECHNICAL MATERIAL AND FLOWABLE CONCENTRATE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Progress Report to CIPAC on method Validation Work conducted by Monsanto

By

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SILTHIOFAM LARGE SCALE COLLABORATIVE TRIAL

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1 INTRODUCTION

The results of the CIPAC full scale collaborative study for Silthiofam technical product and Silthiofam Flowable concentrate are reported in this present study for the content of Silthiofam.

In September 2014, CIPAC information sheet 303 was sent out by the CIPAC Secretary inviting members to participate in a collaborative study to validate the high performance liquid chromatography assay method for Silthiofam in Technical material and Flowable concentrate. A copy of the analytical method, protocol for the performance of the study, analysis report form, samples and standards required for the analysis were sent to the respondents. The participants who completed the study are listed in Section 1.2.

The analytical method assessed in this study took into consideration CIPAC comments made in the frame of a previous small collaborative trial (report presented at CIPAC meeting June 2014)

1.1 SAMPLES

8 test samples, homogenized and analytical standards were sent to the participants. All samples came from different batches of Technical material and Flowable concentrate.

Test sample	Identification code
Silthiofam Technical lot 10-254	TC A
Silthiofam Technical lot 13-396	TC B
Silthiofam Technical lot SIL-test2	TC C
Silthiofam Flowable conc. AG024ES207/1	FS A
Silthiofam Flowable H14507001	FS B
Silthiofam Flowable conc. AG024ES207/2	FS C
Silthiofam Flowable H14507023	FS D
Silthiofam Flowable conc. AG024ES206/1	FS E

Silthiofam Analytical Reference standard (GLP-0911-20072-A) was provided by Monsanto, St Louis.

Di-ethyl phthalate internal standard (A0336406) was purchased at Acros, cat 11452.

1.2 PARTICIPANTS

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Lab 16	<p>Florentina Ciotea Laboratory for Quality Control of Pesticide Central Phytosanitary Laboratory 11 Voluntari St Code: 077190, Voluntari Romania</p>
Lab 17	<p>Kujala Jouko CABB OY PO box 566 67701 Kokkola Finland</p>
Lab 18	<p>Dr. Angela Santilio Italian National Institute of Health Department of Environmental and Primary Prevention pesticide unit V.le Regina Elena, 299 Rome 00162 Italy</p>

2 ANALYTICAL METHOD

2.1 SCOPE

This method is applicable for the determination of Silthiofam in Silthiofam Technical and Silthiofam Flowable concentrate. In the small scale trial CIPAC/4960/R June 2014 the effect of both internal and external standard analysis was evaluated, concluding that the internal standard analysis was the preferred calculation method.

This approach was, as requested at the ESPAC meeting in Dublin 2015, repeated in the full scale trial to give further confidence in the evaluation of the preferred calculation method.

2.2 OUTLINE OF THE METHOD

Silthiofam was determined by reversed-phase high performance liquid chromatography using UV detection at 260nm.

For evaluation purposes as requested by ESPAC team, the collaborative trial was set-up to provide quantification by both external standardization and internal standard (di-ethyl phthalate) calculation.

The following table indicates all relevant suggested instrumental parameters.

Column	LiChroCart C18, LiChrospher 100 (125 x 4 mm; 5 μ particle size)
	Merck part n ^o 017011
Pump Program	Isocratic – 50% Milli-Q water / 50% Acetonitrile
	Flow Rate: 1.2ml / min
Detector	UV / visible detector
	Wavelength: 260nm
Auto sampler	Injection Volume: 5 μ l
Run Time	Total Run Time: 10 minutes
	Retention Time: Silthiofam: 7.5 minutes
	DEP (internal standard): 3.5 minutes
Column temperature	40 °C

2.3 PROCEDURE

Each sample was analyzed by six individual determinations ($n_1 = 6$). The samples were analyzed in a first run on day 1 by duplicate injection of three weightings for each sample. The sequence was repeated on a second day with

three other weightings with duplicate injection of each sample. The Silthiofam content for each injection was calculated using the mean response factor from the calibration solutions bracketing the injections of the sample solutions. For the calculation of the content of a sample, the mean value of the duplicate injections was used. For further details, please see method CIPAC/5004/m.

3 REMARKS OF PARTICIPANTS

3.1 ANALYTICAL CONDITIONS

LAB	Liquid chromatograph, detector	Column	Column dimensions	part. Size	Mobile phase	flow rate	detector wavelength	inj. Vol.	temp.
1	Jasco PU-2089 plus	Symmetry C18	150 x 3	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
2	Agilent 1100, PDA	Zorbax Eclipse XDB C18	125 x 4	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
3	Dionex UltiMate 3000, PDA	lichrospher C18	125 x 4	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
4	Dionex HPLC System, UV	purospher RP	125 x 4	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	25 °C
5	Waters ACQUITY UPLC, PDA	lichrospher C18	125 x 4	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
6	Agilent 1200, PDA	Zorbax Eclipse XDB C18	?	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	ambient
7	H-Class Waters (QSM), PDA	BEH C18	100 x 2.1	1.7µm	50%water/50%ACN	1.2 ml/min	260 nm	1 µl	40 °C
8	Agilent 1200 Series, UV	lichrospher C18	125 x 4	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
9	SHIMADZU LC-20A, PDA	Inertsil ODS-3	4.6 mm x 150 mm	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
10	Agilent 1100, PDA	Phenomenex Gemini C18 110 A	150 x 4.6 mm	5µm	50%water/50%ACN	1.4 ml/min	260 nm	5 µl	40 °C
11	Simadzu, pump: LC-20AD; detector:SPD-M10Avp	Supelcosil LC-18DB	100x4,6 mm	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
12	Agilent 1200 Series, UV	Hypersil ODS	150x4.6mm	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
13	HPLC Waters Alliance 2695, PDA	Hypersil ODS C18	125 x 4 mm	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
14	Agilent 1100, PDA	lichrospher C18	125 x 4	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
15	Agilent 1100, UV	Lichrospher 100-5RP-18	125 x 4.6	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
16	Varian Prostar, UV	Kinetex C18	100 x 4.6	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
17	Agilent 1200, PDA	Alltech, Alltima C18	150 x 3.2	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C
18	Perkin Elmer serie 200, PDA	Lichrospher C18	250 x 4	5µm	50%water/50%ACN	1.2 ml/min	260 nm	5 µl	40 °C

3.2 REMARKS

Several remarks were received listing deviation from the suggested method:

LAB	Remark
1	No remarks
2	No remarks
3	No remarks
4	No remarks
5	Bad inj technical C2 Day2
6	No remarks
7	UPLC train used dilution 1ml/25ml in stead of 5ml/25ml
8	A 10% DEP solution was prepared. To each 50 mL volumetric flask 4 mL of this solution was added.
9	Instead of weighing DEP, I prepared DEP solution and used it for preparing samples (detail is following). 20 g DEP is weighed into a 250 ml volumetric flask and diluted with acetonitrile to the mark. 5 ml of this solution is transferred into a 50 ml volumetric flask instead of 0.4 g DEP being weighed into the flask. # In day 1, 19.9965 g DEP was weighed into 250 ml volumetric flask. # In day 2, 20.0449 g DEP was weighed into 250 ml volumetric flask.
10	Less amount of analytical standard and samples have been weighed to avoid dilutions. Concentration results (g/100mL) in the table are divided by 5. 10 mL (day 1) and 20 mL (day 2) 0.8% internal standard solution has been added to each sample.
11	No remarks
12	Tailing peaks
13	Add istd volumetric Sonicate 15min in stead of shake 15min
14	Odd results day 2 : pressure fluctuations Suggest to use volumetric ISTD addition Suggest to use more water to dissolve sample before ACN
15	add istd volumetric
16	No remarks
17	Sonicate 15min in stead of shake 15min Part of day 2 sequence was carried out on day 3
18	No results for Day2

4 RESULTS & DISCUSSION

4.1 RESULTS

4.1.1 LINEARITY

The following table summarizes for all the labs the obtained coefficient of determination (R^2) using a three level calibration curve with both internal and external standard analysis.

Using a paired T-test no significant difference could be observed between internal and external standard analysis.

	Internal Standard	External Standard
Lab 1	0.99988	0.99958
Lab 2	0.99998	0.99996
Lab 3	0.99978	0.99975
Lab 4	0.99993	0.99999
Lab 5	1.00000	0.99998
Lab 6	0.99840	0.99820
Lab 7	1.00000	0.99997
Lab 8	1.00000	0.99999
Lab 9	1.00000	1.00000
Lab 10	1.00000	1.00000
Lab 11	0.99996	0.99987
Lab 12	1.00000	0.99995
Lab 13	0.99998	0.99997
Lab 14	0.99974	0.99997
Lab 15	1.00000	0.99989
Lab 16	0.99999	0.99999
Lab 17	0.99999	0.99997
Lab 18	0.99763	0.99892
average	099974	099978
min	0.997627	0.998200
max	1.000000	1.000000

4.1.2 CARRY OVER / SYSTEM INTERFERENCE

No lab reported the observation of significant interferences in the first injection of a blank sample injection after injection of the highest standard tested in the linearity test, thus no carry-over was reported.

4.1.3 REPEATABILITY

The following table summarizes the results of 6 repeated injections of one calibration solution. A paired T-test showed a significant difference between the repeatability using internal standard versus external standard analysis. As can be seen relative standard deviation values (RSD) obtained using an internal standard are better for all but one lab compared to the RSDs obtained without internal standard.

	ISTD	ESTD
Lab 1	0.082	0.429
Lab 2	0.157	0.215
Lab 3	0.012	0.085
Lab 4	0.571	0.719
Lab 5	0.056	0.064
Lab 6	0.137	0.123
Lab 7	0.212	0.214
Lab 8	0.357	0.362
Lab 9	0.087	0.127
Lab 10	0.091	0.141
Lab 11	0.158	0.603
Lab 12	0.195	0.216
Lab 13	0.131	0.237
Lab 14	0.017	0.429
Lab 15	0.080	0.168
Lab 16		
Lab 17	0.091	0.143
Average	0.152	0.276
min	0.012000	0.064000
max	0.571000	0.719000

4.1.4 STATISTICAL ANALYSIS

The statistical evaluation of the collaborative trial was performed according to DIN ISO 5725. The samples were sent to 18 laboratories. All labs sent back results. The results from lab 18 were not included because no data for the day 2 analysis could be provided. The assay results reported by the laboratories are listed in tables 1 - 10, and are presented in figures 1 - 24.

Statistical evaluation of the data was done following "CIPAC guidelines for Collaborative Study Procedures for Assessment of Performance of Analytical Methods". Both data sets for external standard calculation and internal standard calculation were examined for outliers and stragglers using first visual inspection of the box plot diagram for strong outliers, next Cochran's test on the within-lab variability, followed by a Grubbs test on the laboratory means, and using iteration and interpretation were necessary. Where deemed, tests were performed at an alpha level of 0.01 for outlier detection and 0.05 for straggler detection. Based on this procedure, the Cochran and

Grubbs test identified the following potential outlier and straggler lab data from the sample sets. Straggler and outlier values are reported below.

Internal standard	Box-plot	Cochran Straggler	Cochran outlier	Grubbs straggler	Grubbs outlier
TC A	14	3	12	1, 14	6
TC B	14	None	6, 14	3, 16	4, 6
TC C	7, 14	3	6, 12	3	None
FS A	6	6	7, 15	None	None
FS B	2, 4, 7	None	15, 16	None	None
FS C	6, 15, 17	None	1, 16	None	None
FS D	6, 11, 17	None	none	None	None
FS E	2, 17	1	1, 6	4	None

External standard	Box-plot	Cochran Straggler	Cochran outlier	Grubbs straggler	Grubbs outlier
TC A	14, 16	2	12, 15	4, 7	6, 12, 14
TC B	5, 14	12	1, 15	4, 16	6, 7, 14
TC C	5, 6, 14	None	1, 15, 16	None	None
FS A	2, 6	None	15	None	7
FS B	2	2	1, 14, 15, 16	None	7
FS C	15, 16, 17	15	1, 2	None	None
FS D	2, 16, 17	6	1, 6, 15	None	None
FS E	2, 4, 5, 14	None	17	1, 16	None

A summary of the statistical evaluation for the labs is given in table 1 & 2, which includes the repeatability and reproducibility values, as well as the between-lab experimental Reproducibility Relative Standard Deviation, $\%RSD_R$ and the calculated accepted value $\%RSD_{R(HOR)}$, based on the Horwitz curve calculation.

4.2 DISCUSSION

4.2.1 INTERNAL STANDARD EVALUATION

TC-A Internal standard

Lab 14 reported odd results on the day 2 analysis caused by pressure fluctuations. These outliers were identified as outliers by visual analysis of the box-plot (fig. 1 - 8). 1 additional outlier was detected by the Cochran test. The Grubbs test revealed lab 6 as an outlier lab with 100.26 %(m,m) as significant higher mean value. Lab 1 and 14 were identified as straggler by the Grubbs test.

TC-B Internal standard

Lab 14 reported odd results on the day 2 analysis caused by pressure fluctuations. Also day 1 analyses by lab 14 were also identified as outlier by visual analysis of the box-plot (fig. 1 - 8). 3 additional outliers and 1 straggler were detected by the Cochran test. The Grubbs test revealed lab 4 and 6 as an outlier lab with rep. 98.67 %(m,m) and 99.16 %(m,m) as significant lower mean value. Lab 1 and 14 were identified as straggler by the Grubbs test.

TC-C Internal standard

Again, Lab 14 reported odd results on the day 2 analysis caused by pressure fluctuations. 1 value from day 1 analyses by lab 14 and 2 values from lab 7 were also identified as outliers by visual analysis of the box-plot (fig.). 2 additional outliers and 1 straggler were detected by the Cochran test. The Grubbs test revealed lab 3 as straggler by the Grubbs test.

FS-A Internal standard

1 visual outlier was detected, 3 Cochran outliers were identified with no stragglers. No Grubbs outliers or stragglers were identified.

FS-B Internal standard

3 visual outliers were detected, 2 Cochran outliers were identified with no stragglers. Cochran statistic remains significant at 0.01 alpha level. However no clear outliers could be identified so further data was retained. No Grubbs outliers or stragglers were identified.

FS-C Internal standard

4 visual outliers were detected, 2 Cochran outliers were identified, also lab 16 day 2 dataset was identified as outliers by Cochran with no stragglers. No Grubbs outliers or stragglers were identified.

FS-D Internal standard

5 visual outliers were detected; no Cochran outliers were identified with no stragglers. Cochran statistic remains significant at 0.01 alpha level. However no clear outliers could be identified so further data was retained. No Grubbs outliers or stragglers were identified.

FS-E Internal standard

2 visual outliers were detected, 3 Cochran outliers were identified with 1 straggler. Lab 4 was identified as Grubbs stragglers.

4.2.2 EXTERNAL STANDARD EVALUATION**TC-A External standard**

Lab 14 reported odd results on the day 2 analysis caused by pressure fluctuations. These and one additional result were identified as outliers by visual analysis of the box-plot (fig. 1 - 8). The entire dataset of Lab 15 was deleted due to the clear presence of a bimodal (day1 – day2) distribution. 1 additional outlier was detected by the Cochran test. The Grubbs test revealed lab 6 and 12 as an outlier lab with resp. 100.52 %(m,m) and 100.13 %(m,m) as significant higher mean value and lab 14 as an outlier lab with 98.34 %(m,m) as significant lower mean value. Lab 3 and 16 were identified as straggler by the Grubbs test.

TC-B External standard

Lab 14 reported odd results on the day 2 analysis caused by pressure fluctuations. These and 3 additional results were identified as outliers by visual analysis of the box-plot (fig. 1 - 8). The entire dataset of Lab 1 and 15 was deleted due to the clear presence of a bimodal (day1 – day2) distribution. No additional outlier was detected by the Cochran test. The Grubbs test revealed lab 7 as an outlier lab with 100.80 %(m,m) as significant higher mean value and 6 and 14 as an outlier lab with resp. 98.84 %(m,m) and 97.24 %(m,m) as significant lower mean value. Lab 4 and 16 were identified as straggler by the Grubbs test.

TC-C External standard

Lab 14 reported odd results on the day 2 analysis caused by pressure fluctuations. These and 2 additional results were identified as outliers by visual analysis of the box-plot (fig. 1 - 8). The entire dataset of Lab 1 and 15 was deleted due to the clear presence of a bimodal (day1 – day2) distribution. 1 additional outlier was detected by the Cochran test. The Grubbs test revealed no outliers and stragglers with the remaining data.

FS-A External standard

2 results were identified as outliers by visual analysis of the box-plot (fig. 1 - 8). 1 additional outlier was detected by the Cochran test. The Grubbs test revealed lab 7 as an outlier lab with 11.746 %(m,m) as significant higher mean value. No straggler was detected with the Grubbs test.

FS-B External standard

1 result was identified as outlier by visual analysis of the box-plot (fig. 1 - 8). The entire dataset of Lab 1, 15 and 16 was deleted due to the clear presence of a bimodal (day1 – day2) distribution. 2 additional outliers were detected by the Cochran test. The Grubbs test revealed lab 7 as an outlier lab with 11.855 %(m,m) as significant higher mean value. No straggler was detected with the Grubbs test.

FS-C External standard

3 results were identified as outlier by visual analysis of the box-plot (fig. 1 - 8). 3 additional outliers were detected by the Cochran test. No outlier or straggler was detected with the Grubbs test.

FS-D External standard

3 results were identified as outlier by visual analysis of the box-plot (fig. 1 - 8). The entire dataset of Lab 1 and 15 was deleted due to the clear presence of a bimodal (day1 – day2) distribution. 1 additional outlier was detected by the Cochran test. No outlier or straggler was detected with the Grubbs test.

FS-E External standard

4 results were identified as outlier by visual analysis of the box-plot (fig. 1 - 8). The entire dataset of Lab 1 and 15 was deleted due to the clear presence of a bimodal (day1 – day2) distribution. 1 additional outlier was detected by the Cochran test. No outlier , 2 stragglers were detected with the Grubbs test.

4.2.3 INTERNAL STANDARD VS EXTERNAL STANDARD

Using Internal Standard Analysis a total of 65 data points were removed as outlier by either visual inspection of the box-plot diagram and Cochran and Grubbs, representing 3.8 % of all data.

Using External Standard Analysis a total of 136 data points were removed as outlier by either visual inspection of the box-plot diagram and Cochran and Grubbs, representing 7.8 % of all data.

5 CONCLUSION

A considerable number of outliers were identified for several labs. Careful examination of the original data using a box-plot diagram (fig. 1 - 8) led to the exclusion of several obvious outliers, both for internal and external standard analysis. Statistical analysis using the data-set without the visual outliers showed for both Internal and External Standard Analysis that the between-lab Reproducibility Relative Standard Deviation ($\%RSD_R$) and the Repeatability Relative Standard deviation ($\%RSD_r$) were below the calculated limit ($\%RSD_{R(HOR)}$) using the Horwitz equation for the determination of Silthiofam in Silthiofam Technical and Silthiofam Flowable Concentrate (table 1).

After further refining the data using Cochran and Grubbs outlier tests to identify and remove outliers, the $\%RSD_R$ and $\%RSD_r$ improved further both for Internal and External standard analysis to well below the calculated limit ($\%RSD_{R(HOR)}$) using the Horwitz equation for the determination of Silthiofam in Silthiofam Technical and Silthiofam Flowable Concentrate.

When compared to internal standard analysis significantly more data was identified as outlier using Cochran and Grubbs outlier tests for external standard analysis. In general performance characteristics are superior for internal standard analysis when compared to external standard analysis. (fig.25)

The acceptance of the Internal Standard Analysis method as an approved CIPAC assay method for Silthiofam in Silthiofam Technical and Silthiofam Flowable Concentrate is recommended.

6 ACKNOWLEDGEMENTS

Monsanto company wishes to thank all the laboratories and their staff who participated in this study.

7 TABLES & FIGURES

Table 1A – Silthiofam Internal Standard Analysis– Summary of the statistical evaluation of the collaborative study data. Only strong outliers removed (box-plot diagram)

level	TC-A	TC-B	TC-C	FS-A	FS-B	FS-C	FS-D	FS-E
N° of Labs	17	17	17	17	17	17	17	17
N° of Outliers	3	5	6	1	2	4	5	2
N° of Labs retained	17	16	17	17	17	17	17	17
N° of Results	99	97	96	101	100	98	97	100
X %(m,m)	99.44	99.64	99.03	11.37	11.66	11.09	11.96	12.23
S _r	0.43	0.31	0.46	0.08	0.10	0.10	0.10	0.09
S _L	0.31	0.44	0.30	0.07	0.06	0.11	0.00	0.10
S _R	0.52	0.54	0.55	0.11	0.12	0.15	0.12	0.13
r	1.19	0.86	1.28	0.23	0.29	0.27	0.28	0.26
R _L	0.86	1.24	0.83	0.20	0.17	0.32	0.20	0.27
R	1.47	1.51	1.53	0.30	0.33	0.42	0.34	0.37
%RSD _r	0.43	0.31	0.46	0.73	0.89	0.88	0.82	0.75
%RSD _R	0.53	0.54	0.55	0.96	1.03	1.35	1.02	1.08
%RSD _{R(HOR)}	2.00	2.00	2.00	2.77	2.76	2.78	2.75	2.74

Limits %(m,m)

X+R	100.91	101.15	100.56	11.68	11.99	11.51	12.31	12.60
X-R	97.97	98.13	97.50	11.07	11.32	10.67	11.62	11.86
X+r	100.63	100.50	100.31	11.60	11.95	11.37	12.24	12.49
X-r	98.25	98.78	97.75	11.14	11.37	10.82	11.69	11.97

Where:

X = average

S_r = repeatability standard deviationS_L = pure between laboratory standard deviationS_R = reproducibility standard deviation $(S_r^2 + S_L^2)^{0.5}$ r = repeatability within-lab (2.8*S_r)R = reproducibility between labs (2.8*S_R)R_L = reproducibility within lab on different days (2.8*S_L)%RSD_r = repeatability relative standard deviation (100*S_r/X)%RSD_R = reproducibility relative standard deviation between labs (100*S_R/X)%RSD_{R(HOR)} = Horwitz value calculated from $2^{(1-0.5 \log c)}$

Where c is the concentration of the analyte as a decimal fraction (e.g. for 100% concentration c=1)

Table 1B – Silthiofam External Standard Analysis– Summary of the statistical evaluation of the collaborative study data. Only strong outliers removed (box-plot diagram)

level	TC-A	TC-B	TC-C	FS-A	FS-B	FS-C	FS-D	FS-E
N° of Labs	17	17	17	17	17	17	17	17
N° of Outliers	4	6	6	7	1	3	3	3
N° of Labs retained	17	17	16	17	17	17	17	17
N° of Results	98	96	95	100	101	99	99	100
X %(m,m)	99.54	99.67	99.05	11.35	11.64	11.07	11.96	12.20
S _r	0.83	0.76	0.81	0.11	0.20	0.13	0.20	0.15
S _L	0.36	0.70	0.12	0.12	0.06	0.10	0.08	0.17
S _R	0.91	1.03	0.81	0.16	0.21	0.17	0.21	0.22
r	2.33	2.12	4.28	0.30	0.57	0.37	0.55	0.42
R _L	1.01	1.95	0.31	0.33	0.18	0.29	0.21	0.46
R	2.54	2.88	4.46	0.44	0.60	0.47	0.59	0.62
%RSD _r	0.84	0.76	0.81	0.94	1.76	1.18	1.63	1.22
%RSD _R	0.91	1.03	0.82	1.39	1.84	1.51	1.75	1.83
%RSD _{R(HOR)}	2.00	2.02	2.00	2.78	2.79	2.81	2.78	2.75

Limits %(m,m)

X+R	102.08	102.55	101.32	11.80	12.23	11.54	12.54	12.82
X-R	97.00	96.78	96.77	10.91	11.04	10.60	11.38	11.57
X+r	101.87	101.79	101.30	11.65	12.21	11.44	12.51	12.62
X-r	97.21	97.54	96.79	11.05	11.06	10.70	11.41	11.78

Where:

X = average

S_r = repeatability standard deviation

S_L = pure between laboratory standard deviation

S_R = reproducibility standard deviation $(S_r^2 + S_L^2)^{0.5}$

r = repeatability within-lab $(2.8 * S_r)$

R = reproducibility between labs $(2.8 * S_R)$

R_L = reproducibility within lab on different days $(2.8 * S_L)$

%RSD_r = repeatability relative standard deviation $(100 * S_r / X)$

%RSD_R = reproducibility relative standard deviation between labs $(100 * S_R / X)$

%RSD_{R(HOR)} = Horwitz value calculated from $2^{(1-0.5 \log c)}$

Where c is the concentration of the analyte as a decimal fraction (e.g. for 100% concentration c=1)

Table 2A – Silthiofam Internal Standard Analysis– Summary of the statistical evaluation of the collaborative study data. Selected Outlier Test Results Removed

Level	TC-A	TC-B	TC-C	FS-A	FS-B	FS-C	FS-D	FS-E
N° of Labs	17	17	17	17	17	17	17	17
N° of Stragglers	1 ^(c) 2 ^(g)	2 ^(g)	1 ^(c) 1 ^(g)	1 ^(c)	1 ^(c)	none	1 ^(c)	1 ^(c) 1 ^(g)
N° of Outliers	4 ^(c) 1 ^(g)	9 ^(c) 2 ^(g)	8 ^(c)	4 ^(c)	6 ^(c)	9 ^(c)	6 ^(c)	5 ^(c)
N° of Labs retained	16	14	17	17	17	17	17	17
N° of Results	92	84	94	98	96	93	97	97
X %(m,m)	99.37	99.82	99.02	11.37	11.68	11.09	11.96	12.24
S _r	0.35	0.28	0.38	0.07	0.08	0.06	0.10	0.08
S _L	0.25	0.17	0.30	0.07	0.08	0.11	0.00	0.09
S _R	0.43	0.32	0.49	0.10	0.11	0.12	0.12	0.12
R	0.98	0.77	1.07	0.19	0.21	0.16	0.28	0.22
R _L	0.69	0.47	0.85	0.19	0.22	0.30	0.20	0.26
R	1.20	0.91	1.37	0.27	0.31	0.34	0.34	0.34
%RSD _r	0.35	0.28	0.38	0.61	0.66	0.50	0.82	0.65
%RSD _R	0.43	0.32	0.49	0.86	0.94	1.09	1.02	0.99
%RSD _{R(HOR)}	2.00	2.00	2.00	2.77	2.76	2.78	2.75	2.74

Limits %(m,m)

X+R	100.57	100.73	100.39	11.64	11.98	11.42	12.31	12.57
X-R	98.17	98.92	97.66	11.09	11.37	10.75	11.62	11.90
X+r	100.35	100.60	100.09	11.56	11.89	11.24	12.24	12.46
X-r	98.39	99.05	97.96	11.17	11.46	10.93	11.69	12.01

Where:

X = average

S_r = repeatability standard deviation

S_L = pure between laboratory standard deviation

S_R = reproducibility standard deviation $(S_r^2 + S_L^2)^{0.5}$

r = repeatability within-lab (2.8*S_r)

R = reproducibility between labs (2.8*S_R)

R_L = reproducibility within lab on different days (2.8*S_L)

%RSD_r = repeatability relative standard deviation (100*S_r/X)

%RSD_R = reproducibility relative standard deviation between labs (100*S_R/X)

%RSD_{R(HOR)} = Horwitz value calculated from $2^{(1-0.5\log c)}$

Where c is the concentration of the analyte as a decimal fraction (e.g. for 100% concentration c=1)

Table 2B – Silthiofam External Standard Analysis– Summary of the statistical evaluation of the collaborative study data. Selected Outlier Test Results Removed

Level	TC-A	TC-B	TC-C	FS-A	FS-B	FS-C	FS-D	FS-E
N° of Labs	17	17	17	17	17	17	17	17
N° of Stragglers	1 ^(c) 2 ^(g)	1 ^(c) 2 ^(g)	none	none	1 ^(c)	1 ^(c)	1 ^(c)	2 ^(g)
N° of Outliers	11 ^(c) 3 ^(g)	18 ^(c) 3 ^(g)	18 ^(c)	3 ^(c) 1 ^(g)	21 ^(c) 1 ^(g)	6 ^(c)	16 ^(c)	3 ^(c)
N° of Labs retained	13	12	15	16	13	17		
N° of Results								
X %(m,m)	99.56	99.88	99.01	11.34	11.63	11.09	11.98	12.20
S _r	0.60	0.44	0.58	0.09	0.07	0.09	0.11	0.14
S _L	0.00	0.18	0.21	0.06	0.03	0.10	0.10	0.18
S _R	0.59	0.48	0.62	0.11	0.08	0.13	0.15	0.22
r	1.68	1.24	1.63	0.26	0.21	0.25	0.31	0.38
R _L	0.00	0.50	0.59	0.17	0.09	0.28	0.27	0.50
R	1.66	1.34	1.73	0.31	0.23	0.38	0.41	0.63
%RSD _r	0.60	0.44	0.59	0.81	0.64	0.81	0.92	1.12
%RSD _R	0.60	0.48	0.62	0.97	0.70	1.21	1.23	1.84
%RSD _{R(HOR)}	2.00	2.00	2.00	2.78	2.76	2.78	2.75	2.75

Limits %(m,m)

X+R	101.22	101.22	100.74	11.64	11.86	11.47	12.39	12.83
X-R	97.90	98.55	97.28	11.03	11.41	10.71	11.57	11.57
X+r	101.24	101.12	100.64	11.59	11.84	11.34	12.29	12.58
X-r	97.89	98.64	97.38	11.08	11.42	10.84	11.67	11.82

Where:

X = average

S_r = repeatability standard deviation

S_L = pure between laboratory standard deviation

S_R = reproducibility standard deviation $(S_r^2 + S_L^2)^{0.5}$

r = repeatability within-lab $(2.8*S_r)$

R = reproducibility between labs $(2.8*S_R)$

R_L = reproducibility within lab on different days $(2.8*S_L)$

%RSD_r = repeatability relative standard deviation $(100*S_r/X)$

%RSD_R = reproducibility relative standard deviation between labs $(100*S_R/X)$

%RSD_{R(HOR)} = Horwitz value calculated from $2^{(1-0.5\log c)}$

Where c is the concentration of the analyte as a decimal fraction (e.g. for 100% concentration c=1)

Table 3A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-A (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	99.92	99.69	99.81	99.76	99.81	99.91	99.82	0.09	G
2	98.05	99.10	98.93	99.61	99.40	99.50	99.10	0.57	
3	100.36	100.57	99.82	99.24	99.20	98.99	99.70	0.66	C
4	99.33	98.84	99.80	99.53	99.02	99.26	99.30	0.34	
5	98.90	99.17	98.96	98.88	99.00	99.08	99.00	0.11	
6	100.72	100.56	100.79	100.13	99.31	100.04	100.26	0.56	G
7	99.54	99.68	99.32	98.79	99.35	99.36	99.34	0.30	
8	99.77	99.32	99.54	99.63	99.14	99.74	99.52	0.25	
9	99.00	99.19	98.73	99.42	99.65	99.71	99.28	0.38	
10	99.45	99.74	99.63	99.13	99.07	99.12	99.36	0.29	
11	99.11	99.79	100.58	99.44	99.65	100.22	99.80	0.53	
12	101.39	98.87	99.10	99.75	99.25	99.27	99.60	0.92	C
13	99.03	99.29	99.21	99.19	99.33	98.97	99.17	0.14	
14	98.58	99.23	98.83	99.37	94.54	115.43	101.00	7.30	G
15	99.25	99.53	99.70	99.47	99.06	99.32	99.39	0.23	
16	99.44	99.18	99.36	99.55	98.53	99.14	99.20	0.36	
17	99.62	99.54	99.58	99.74	100.11	100.04	99.77	0.25	

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 3B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-A (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	99.56	98.81	100.36	98.30	100.28	100.23	99.59	0.87	
2	97.73	99.40	99.01	100.66	100.45	100.23	99.58	1.11	C
3	100.51	100.33	99.93	98.75	98.86	99.00	99.56	0.79	
4	98.60	99.49	99.60	99.54	98.56	99.12	99.15	0.48	C
5	99.49	99.73	99.36	99.01	99.19	99.47	99.38	0.25	
6	100.34	100.66	100.67	100.63	101.06	99.78	100.52	0.43	G
7	99.88	100.65	99.32	100.31	100.26	99.68	100.01	0.48	C
8	100.13	99.85	99.73	99.59	98.83	99.84	99.66	0.44	
9	99.56	99.22	98.67	99.51	99.69	99.69	99.39	0.39	
10	99.57	99.41	99.26	99.52	99.49	99.34	99.43	0.11	
11	101.31	100.62	100.30	99.02	98.99	99.34	99.93	0.96	
12	101.34	98.78	98.45	100.96	100.47	100.80	100.13	1.22	C, G
13	99.34	99.59	100.16	99.17	99.43	99.61	99.55	0.34	
14	98.16	98.44	98.44	99.74	100.21	114.83	101.64	6.52	C, G
15	101.70	100.38	100.40	97.56	96.90	95.87	98.80	2.33	C
16	99.63	99.04	99.36	99.32	99.74	103.01	100.02	1.49	C
17	99.29	100.05	99.70	99.60	99.52	99.80	99.66	0.26	

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 4A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-B (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	99.88	99.92	99.69	99.76	99.97	99.74	99.83	0.11	
2	99.70	99.79	99.75	100.07	100.08	100.49	99.98	0.30	
3	100.42	100.60	100.62	99.70	99.73	99.70	100.13	0.47	G
4	99.11	99.00	99.38	99.00	99.07	99.30	99.14	0.16	G
5	99.52	99.75	99.60	99.74	99.59	99.46	99.61	0.11	
6	97.93	99.51	98.29	98.03	99.53	98.71	98.67	0.72	C, G
7	100.07	99.86	99.74	99.69	99.24	100.09	99.78	0.31	
8	99.89	99.47	99.78	99.77	99.15	99.27	99.56	0.30	
9	99.07	99.66	99.71	100.01	100.08	99.90	99.74	0.37	
10	99.98	99.60	100.09	100.06	99.98	100.02	99.95	0.18	
11	100.36	100.33	99.98	100.14	100.00	99.69	100.08	0.25	
12	99.43	99.25	99.70	99.87	99.97	99.98	99.70	0.30	
13	99.87	99.68	99.65	99.53	99.50	99.87	99.68	0.16	
14	94.89	97.11	98.54	108.80	107.67	102.07	101.51	5.71	C
15	99.99	100.01	99.99	99.69	99.68	100.06	99.90	0.17	G
16	99.72	98.93	99.77	99.89	99.43	99.27	99.50	0.36	
17	99.78	99.95	100.06	100.31	100.14	100.25	100.08	0.20	

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 4B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-B (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	98.19	98.18	98.24	100.56	100.47	99.68	99.22	1.15	C
2	100.17	100.18	99.76	100.02	99.84	100.80	100.13	0.37	
3	100.25	100.81	100.32	100.11	99.31	99.61	100.07	0.54	
4	99.40	99.59	99.65	99.06	99.85	99.34	99.48	0.28	
5	99.66	99.99	99.82	96.47	99.58	99.49	99.17	1.33	C
6	98.15	99.42	98.92	99.20	98.09	99.26	98.84	0.58	C
7	99.85	100.72	100.12	101.50	101.60	100.97	100.79	0.71	G
8	99.93	99.59	99.89	99.94	98.80	99.14	99.55	0.48	
9	99.66	99.64	99.95	100.22	100.37	100.44	100.05	0.35	
10	99.73	99.87	99.96	100.12	100.12	99.98	99.96	0.15	
11	99.99	100.04	99.87	98.87	99.72	99.33	99.64	0.45	
12	99.52	98.93	98.75	99.73	99.99	101.09	99.67	0.84	c
13	100.29	99.79	99.97	100.32	99.88	99.91	100.03	0.22	
14	95.66	97.54	98.52	97.63	97.88	99.23	97.74	1.20	C, G
15	100.57	100.59	102.26	98.75	98.29	97.51	99.66	1.78	C
16	100.41	100.34	99.65	100.54	101.31	101.32	100.59	0.64	C
17	99.32	99.81	99.71	100.71	100.68	100.11	100.06	0.55	

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 5A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-C (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	99.72	99.70	99.85	99.71	99.71	99.89	99.77	0.08	g
2	99.05	99.06	99.20	99.68	99.49	99.41	99.32	0.25	
3	100.38	99.95	99.62	98.85	98.25	99.13	99.36	0.77	c
4	98.90	98.57	99.17	98.35	98.48	98.53	98.67	0.31	
5	98.78	98.75	98.73	98.60	98.70	98.65	98.70	0.07	
6	98.41	98.43	97.63	99.73	99.86	99.55	98.93	0.90	C
7	130.39	132.79	99.00	98.49	98.93	99.07	109.78	16.91	C
8	99.38	99.31	99.01	99.64	99.39	98.35	99.18	0.46	
9	98.28	98.64	98.51	98.83	99.17	99.35	98.79	0.40	
10	99.37	99.62	99.45	99.15	98.89	99.09	99.26	0.27	
11	99.72	100.41	98.77	98.96	99.37	98.73	99.33	0.65	
12	98.86	98.71	98.99	99.10	99.37	101.21	99.37	0.93	C
13	98.79	98.92	98.97	98.54	98.49	98.49	98.70	0.22	
14	98.51	98.57	92.15	99.31	101.52	96.32	97.73	3.20	C
15	98.88	99.18	98.96	99.25	99.09	99.02	99.06	0.14	
16	98.42	98.82	98.76	98.65	98.11	98.30	98.51	0.28	
17	98.94	99.29	99.23	99.20	98.81	99.29	99.13	0.20	

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 5B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-C (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	99.21	98.12	98.37	99.39	100.09	100.37	99.26	0.90	C
2	99.42	99.02	98.66	99.62	99.93	99.79	99.40	0.48	
3	99.65	99.57	99.20	98.36	97.32	98.35	98.74	0.90	
4	98.67	99.04	99.23	98.24	98.15	98.31	98.61	0.45	G
5	98.99	99.09	99.10	98.67	91.44	97.62	97.48	3.01	C
6	98.31	98.16	97.54	99.19	98.62	99.29	98.52	0.66	
7	98.98	98.95	98.79	99.93	98.57	100.21	99.24	0.67	
8	99.73	99.45	99.31	99.48	99.56	98.77	99.38	0.33	
9	98.47	98.47	98.58	99.34	99.43	99.48	98.96	0.50	
10	99.24	99.32	98.96	99.33	99.38	99.77	99.33	0.26	
11	98.85	99.72	99.98	98.75	99.82	98.86	99.33	0.56	
12	98.20	98.30	98.86	98.96	99.88	100.03	99.04	0.77	
13	97.90	98.92	98.92	98.89	99.13	99.12	98.81	0.46	
14	98.90	97.88	92.31	98.62	100.70	95.82	97.37	2.94	C
15	100.98	99.66	100.53	96.49	96.90	97.14	98.62	2.00	C
16	98.47	98.89	99.52	99.19	100.03	102.09	99.70	1.29	C, g
17	98.92	98.89	99.07	98.49	99.17	98.98	98.92	0.23	

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 6A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-A (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	11.48	11.41	11.48	11.29	11.37	11.43	11.41	0.07	
2	11.36	11.34	11.33	11.46	11.40	11.43	11.39	0.05	
3	11.45	11.44	11.44	11.34	11.34	11.32	11.39	0.06	
4	11.55	11.61	11.47	11.42	11.37	11.38	11.47	0.10	
5	11.25	11.26	11.25	11.28	11.28	11.29	11.27	0.02	
6	11.40	11.32	11.20	11.56	10.73	11.48	11.28	0.30	C
7	11.50	11.34	11.35	11.41	11.71	11.70	11.50	0.17	C, c
8	11.19	11.17	11.12	11.30	11.24	11.26	11.21	0.06	
9	11.24	11.27	11.26	11.38	11.38	11.40	11.32	0.07	
10	11.37	11.39	11.37	11.20	11.22	11.27	11.30	0.08	
11	11.29	11.49	11.52	11.46	11.52	11.56	11.47	0.10	
12	11.38	11.33	11.34	11.42	11.44	11.42	11.39	0.05	
13	11.26	11.26	11.28	11.36	11.34	11.33	11.30	0.04	
14	11.27	11.30	11.24	11.38	11.38	11.38	11.32	0.06	
15	11.48	11.47	11.49	11.52	11.50	11.21	11.44	0.12	C
16	11.28	11.31	11.34	11.32	11.33	11.35	11.32	0.03	
17	11.38	11.38	11.38	11.42	11.41	11.40	11.39	0.02	

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 6B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-A (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	11.21	11.37	11.14	11.45	11.18	11.16	11.25	0.13	
2	11.44	10.77	11.15	11.36	11.40	11.33	11.24	0.25	C
3	11.41	11.44	11.38	11.23	11.20	11.23	11.31	0.11	
4	11.44	11.43	11.40	11.33	11.43	11.44	11.41	0.04	
5	11.30	11.31	11.29	11.25	11.34	11.27	11.29	0.03	
6	11.48	11.35	11.27	11.51	10.90	11.42	11.32	0.23	C
7	11.74	11.82	11.72	11.68	11.66	11.85	11.75	0.08	G
8	11.23	11.22	11.17	11.26	11.24	11.22	11.22	0.03	
9	11.26	11.21	11.22	11.31	11.32	11.33	11.28	0.05	
10	11.33	11.38	11.36	11.30	11.29	11.31	11.33	0.04	
11	11.49	11.31	11.48	11.38	11.36	11.46	11.41	0.07	
12	11.20	11.28	11.34	11.63	11.49	11.53	11.41	0.16	
13	11.20	11.28	11.26	11.31	11.40	11.38	11.31	0.07	
14	11.17	11.31	11.32	11.17	11.29	11.27	11.26	0.07	
15	11.46	11.40	11.43	11.27	11.10	10.75	11.23	0.27	C
16	11.63	11.55	11.54	11.34	11.34	11.47	11.48	0.12	
17	11.32	11.32	11.31	11.34	11.38	11.28	11.33	0.03	

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 7A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-B (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	11.53	11.41	11.31	11.63	11.74	11.63	11.54	0.16	C
2	11.66	11.69	11.67	11.91	11.62	11.68	11.71	0.10	C
3	11.79	11.79	11.76	11.67	11.69	11.65	11.72	0.06	
4	11.79	11.56	11.80	11.72	11.71	11.61	11.70	0.10	
5	11.58	11.58	11.59	11.60	11.57	11.59	11.59	0.01	
6	11.79	11.62	11.73	11.84	11.49	11.65	11.69	0.13	
7	11.63	11.64	11.63	12.02	11.65	11.65	11.70	0.16	C
8	11.51	11.52	11.52	11.59	11.53	11.56	11.54	0.03	
9	11.56	11.59	11.61	11.71	11.75	11.74	11.66	0.08	
10	11.65	11.68	11.69	11.60	11.61	11.65	11.65	0.04	
11	11.90	11.90	11.86	11.80	11.75	11.78	11.83	0.07	
12	11.61	11.63	11.61	11.69	11.71	11.75	11.67	0.06	
13	11.65	11.64	11.61	11.65	11.60	11.63	11.63	0.02	
14	11.61	11.50	11.58	11.68	11.58	11.70	11.61	0.07	
15	11.84	11.84	11.84	11.74	11.51	11.45	11.70	0.18	C
16	11.68	11.98	11.34	11.67	11.25	11.72	11.61	0.27	C
17	11.72	11.71	11.72	11.73	11.76	11.74	11.73	0.02	

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 7B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-B (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	11.32	11.05	11.14	11.86	11.84	11.71	11.49	0.36	C
2	10.95	11.58	11.68	11.87	11.49	11.63	11.53	0.31	C, c
3	11.79	11.77	11.73	11.60	11.66	11.54	11.68	0.10	
4	11.58	11.54	11.55	11.59	11.50	11.52	11.55	0.04	
5	11.62	11.63	11.64	11.62	11.58	11.59	11.61	0.02	
6	11.72	11.66	11.63	11.80	11.60	11.55	11.66	0.09	
7	11.93	11.82	11.82	12.10	12.01	11.44	11.86	0.23	C, G
8	11.58	11.54	11.59	11.60	11.46	11.58	11.56	0.05	
9	11.58	11.51	11.57	11.72	11.65	11.68	11.62	0.08	
10	11.63	11.68	11.67	11.67	11.68	11.72	11.68	0.03	
11	11.96	11.94	11.93	11.57	11.69	11.71	11.80	0.16	
12	11.62	11.57	11.72	11.57	11.69	11.68	11.64	0.07	
13	11.63	11.63	11.66	11.66	11.69	11.72	11.66	0.03	
14	11.72	11.46	11.54	11.65	11.62	10.98	11.49	0.27	C
15	11.82	11.67	11.86	11.24	11.12	11.07	11.46	0.36	C
16	11.96	12.45	11.31	11.64	11.06	11.86	11.71	0.49	C
17	11.65	11.66	11.61	11.66	11.76	11.69	11.67	0.05	

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 8A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-C (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	10.80	11.16	10.81	11.00	11.16	11.18	11.02	0.18	C
2	10.99	11.06	11.12	11.07	11.12	11.13	11.08	0.05	
3	11.15	11.13	11.12	11.08	11.05	11.05	11.10	0.04	
4	11.41	11.36	11.45	11.44	11.41	11.23	11.38	0.08	
5	11.01	11.00	10.96	10.99	10.97	10.97	10.99	0.02	
6	11.01	10.95	11.02	10.61	11.42	11.05	11.01	0.26	C
7	11.05	11.03	11.01	11.01	11.04	11.02	11.03	0.02	
8	10.92	10.90	10.88	11.01	10.96	10.91	10.93	0.05	
9	10.92	10.95	10.97	11.10	11.13	11.12	11.03	0.09	
10	11.13	11.10	11.15	11.07	11.04	11.14	11.11	0.04	
11	11.31	11.30	11.29	11.23	11.15	11.22	11.25	0.06	
12	11.02	11.08	11.05	11.12	11.09	11.13	11.08	0.04	
13	11.02	10.99	10.97	11.08	11.05	11.03	11.03	0.04	
14	10.96	11.07	11.04	11.05	11.02	11.03	11.03	0.04	
15	11.19	11.22	11.24	11.20	11.22	10.66	11.12	0.23	C
16	11.06	11.09	11.05	11.81	11.36	11.31	11.28	0.29	C
17	11.10	11.11	11.08	10.92	10.92	11.79	11.16	0.33	C

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 8B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-C (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	10.58	11.06	10.60	11.23	11.20	11.15	10.97	0.30	C
2	10.65	10.57	11.08	10.98	11.01	11.10	10.90	0.23	
3	11.13	11.16	11.11	11.01	11.00	10.92	11.05	0.09	
4	11.22	11.26	11.25	11.20	11.24	11.27	11.24	0.03	
5	11.04	11.06	10.97	11.01	10.96	11.00	11.01	0.04	
6	11.04	10.98	11.13	11.07	11.05	11.07	11.06	0.05	
7	11.21	11.35	11.28	11.31	11.45	11.29	11.31	0.08	
8	10.99	10.99	10.91	11.01	10.95	10.84	10.95	0.06	
9	10.89	10.94	10.99	11.04	11.09	11.03	11.00	0.07	
10	11.16	11.11	11.16	11.16	11.08	11.16	11.14	0.03	
11	11.36	11.34	11.30	11.17	11.14	11.01	11.22	0.14	
12	11.00	10.99	10.97	10.93	10.97	11.04	10.98	0.04	
13	10.90	10.96	10.91	11.07	11.11	11.06	11.00	0.09	
14	11.06	11.05	11.00	10.92	10.86	10.91	10.97	0.08	
15	11.19	11.29	11.20	10.90	10.93	10.26	10.96	0.38	C, c
16	11.15	11.20	11.14	12.17	11.45	10.97	11.35	0.43	C
17	11.02	11.02	11.03	11.22	11.28	12.28	11.31	0.49	C

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 9A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-D (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	11.61	11.96	11.67	11.92	12.04	11.80	11.83	0.17	C
2	11.97	11.97	11.99	12.03	11.96	12.02	11.99	0.03	
3	12.10	12.08	12.11	11.98	11.97	11.95	12.03	0.07	
4	11.97	12.06	12.06	12.06	12.17	12.23	12.09	0.09	
5	11.89	11.91	11.87	11.90	11.87	11.92	11.89	0.02	
6	11.96	11.86	11.87	12.35	11.38	11.85	11.88	0.31	
7	11.91	11.95	11.93	11.91	11.95	11.96	11.93	0.02	
8	11.81	11.82	11.83	11.90	11.85	11.85	11.84	0.03	
9	11.81	11.88	11.89	12.19	12.06	12.05	11.98	0.15	
10	12.07	12.05	12.07	11.91	11.94	11.95	12.00	0.07	
11	12.18	12.16	12.21	11.37	12.12	12.10	12.03	0.32	C
12	11.91	11.96	11.94	12.04	11.95	11.99	11.97	0.04	
13	11.97	11.92	11.91	11.96	11.92	11.84	11.92	0.04	
14	11.66	11.91	11.91	12.02	12.09	12.04	11.94	0.16	
15	12.15	12.16	12.14	11.72	12.12	11.76	12.01	0.21	C
16	11.96	11.95	11.97	11.75	11.99	11.98	11.93	0.09	
17	12.02	12.01	12.04	12.43	11.23	11.85	11.93	0.39	C

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 9B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-D (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	11.47	11.65	11.36	12.68	12.40	11.81	11.90	0.53	C
2	11.84	10.31	11.99	11.82	11.83	11.88	11.61	0.64	C
3	12.10	12.03	12.09	11.80	11.85	11.87	11.96	0.13	
4	11.90	11.87	11.92	12.11	11.83	11.92	11.93	0.10	
5	11.88	11.90	11.88	11.98	11.98	11.97	11.93	0.05	
6	12.07	12.05	11.95	12.37	11.50	11.80	11.96	0.29	C, c
7	12.19	12.20	12.32	12.01	12.43	12.37	12.25	0.15	
8	11.82	11.89	11.87	11.86	11.87	11.88	11.87	0.02	
9	11.84	11.86	11.86	12.10	11.96	12.02	11.94	0.11	
10	12.02	12.11	12.14	11.98	12.01	12.00	12.04	0.06	
11	12.13	12.12	12.21	11.98	11.98	12.08	12.08	0.09	
12	11.86	11.86	11.98	11.90	11.92	11.86	11.90	0.05	
13	11.95	11.89	11.87	11.97	11.97	11.93	11.93	0.04	
14	12.04	11.91	11.85	11.57	11.93	11.96	11.88	0.16	
15	12.18	12.08	12.04	11.30	11.77	11.38	11.79	0.38	C
16	12.18	12.09	11.95	10.91	12.00	12.26	11.90	0.50	
17	11.98	11.89	11.93	12.18	12.03	12.77	12.13	0.33	C

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 10A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-E (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	11.86	12.02	12.05	12.22	12.23	11.79	12.03	0.18	C, c
2	12.19	12.19	12.20	12.27	12.28	11.83	12.16	0.17	C
3	12.34	12.31	12.30	12.21	12.20	12.20	12.26	0.06	
4	12.36	12.36	12.40	12.35	12.53	12.59	12.43	0.10	G
5	12.14	12.11	12.10	12.10	12.09	12.14	12.11	0.02	
6	12.08	12.35	12.23	12.46	11.85	12.03	12.16	0.22	C
7	12.18	12.18	12.19	12.17	12.21	12.21	12.19	0.02	
8	12.07	12.06	12.07	12.12	12.11	12.12	12.09	0.03	
9	12.11	12.13	12.15	12.29	12.29	12.28	12.21	0.09	
10	12.35	12.34	12.31	12.21	12.22	12.15	12.26	0.08	
11	12.37	12.38	12.41	12.38	12.37	12.40	12.38	0.01	
12	12.19	12.21	12.20	12.26	12.31	12.25	12.24	0.04	
13	12.16	12.21	12.21	12.20	12.19	12.16	12.19	0.02	
14	12.22	12.20	12.25	12.37	12.37	12.38	12.30	0.08	
15	12.39	12.21	12.23	12.40	12.39	12.35	12.33	0.09	
16	12.15	12.26	12.19	12.28	12.25	12.24	12.23	0.05	
17	12.25	12.25	12.29	12.27	12.49	10.76	12.05	0.64	C

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Table 10B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-E (all values in %(m,m))

Lab	DAY 1			DAY 2			Mean	SD	Notes
	A	B	C	A	B	C			
1	11.73	11.73	11.71	12.02	11.75	11.71	11.78	0.12	g
2	12.05	12.35	12.19	12.28	12.21	12.08	12.19	0.12	
3	12.28	12.27	12.32	12.07	12.05	12.06	12.18	0.13	
4	12.10	12.05	12.12	12.06	12.26	12.06	12.11	0.08	
5	12.10	12.15	12.10	12.12	11.62	12.11	12.03	0.20	C
6	12.17	12.27	12.29	12.60	11.66	12.05	12.17	0.31	
7	12.32	12.42	12.29	12.45	12.54	12.71	12.46	0.15	g
8	12.13	12.10	12.10	12.18	12.09	12.10	12.12	0.03	
9	12.08	12.08	12.09	12.20	12.18	12.19	12.14	0.06	
10	12.40	12.38	12.34	12.24	12.23	12.22	12.30	0.08	
11	12.39	12.46	12.56	12.26	12.31	12.26	12.37	0.12	
12	12.08	12.10	12.19	12.12	12.11	12.10	12.12	0.04	
13	12.02	12.13	12.22	12.19	12.26	12.27	12.18	0.09	
14	12.14	12.23	12.12	11.69	12.17	12.20	12.09	0.20	C
15	12.15	12.26	12.28	11.96	11.93	11.98	12.09	0.16	
16	12.72	12.68	12.93	12.30	12.56	12.38	12.60	0.23	
17	12.21	12.28	12.15	12.52	12.73	11.86	12.29	0.30	C

SD = standard deviation

c = Cochran Straggler, C = Cochran outlier, g = Grubbs straggler, G = Grubbs outlier

Figure 1A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-A - Box-plot diagram showing strong outliers.

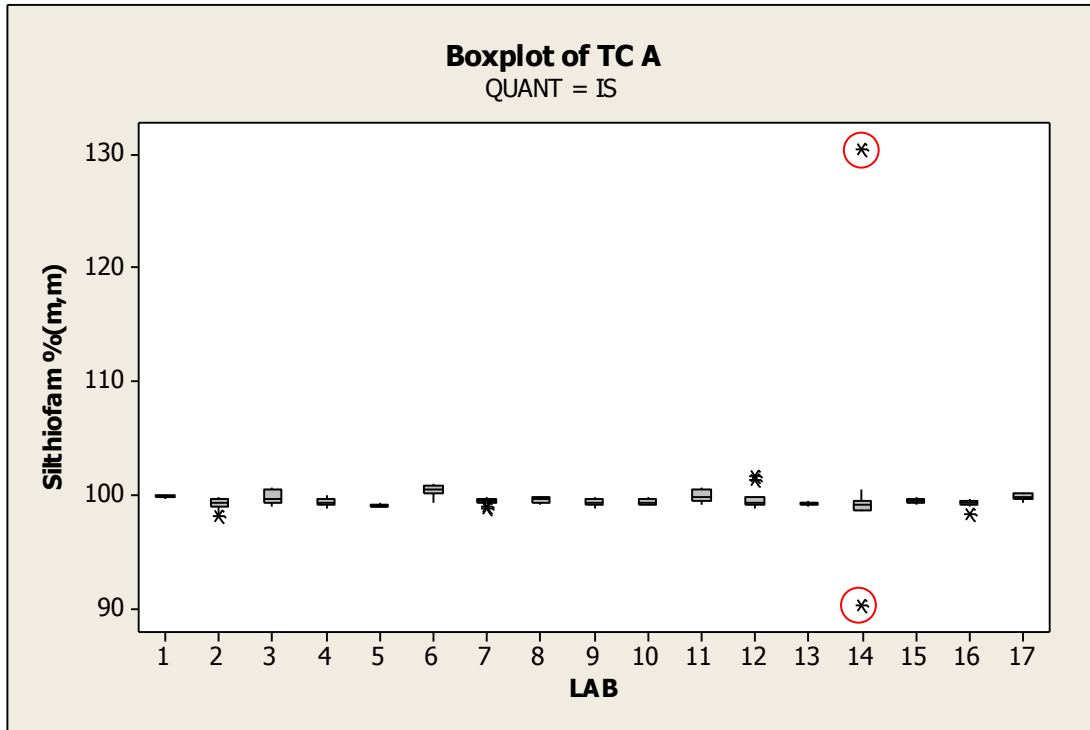


Figure 1B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-A - Box-plot diagram showing strong outliers.

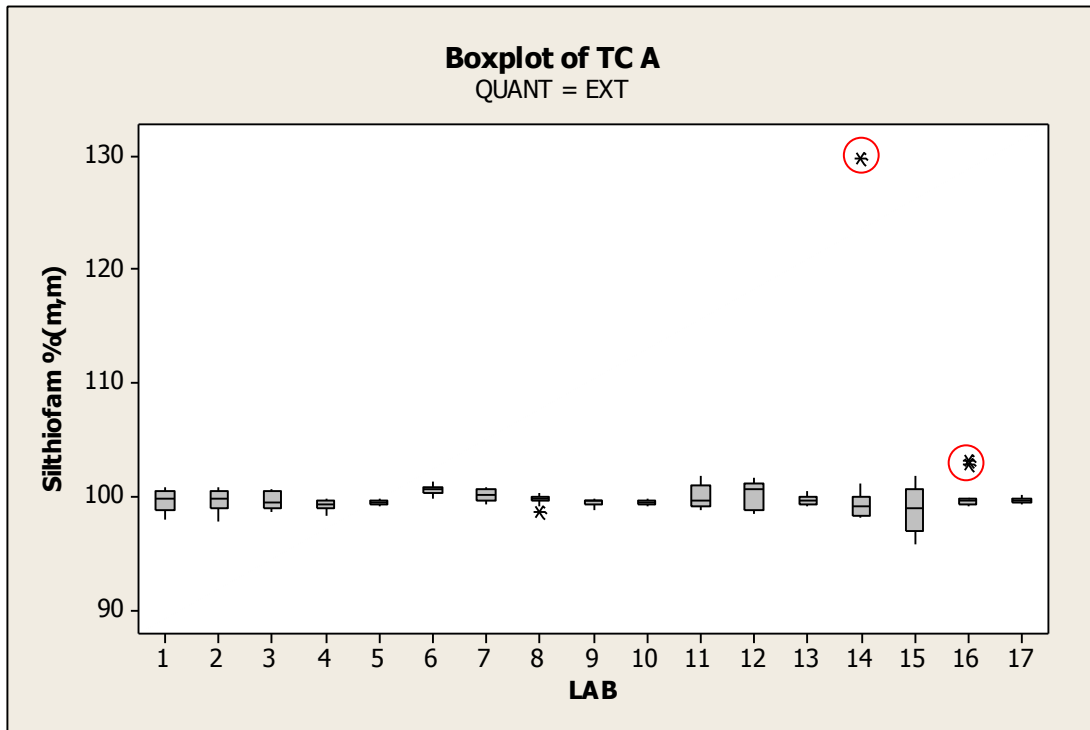


Figure 2A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-B - Box-plot diagram showing strong outliers.

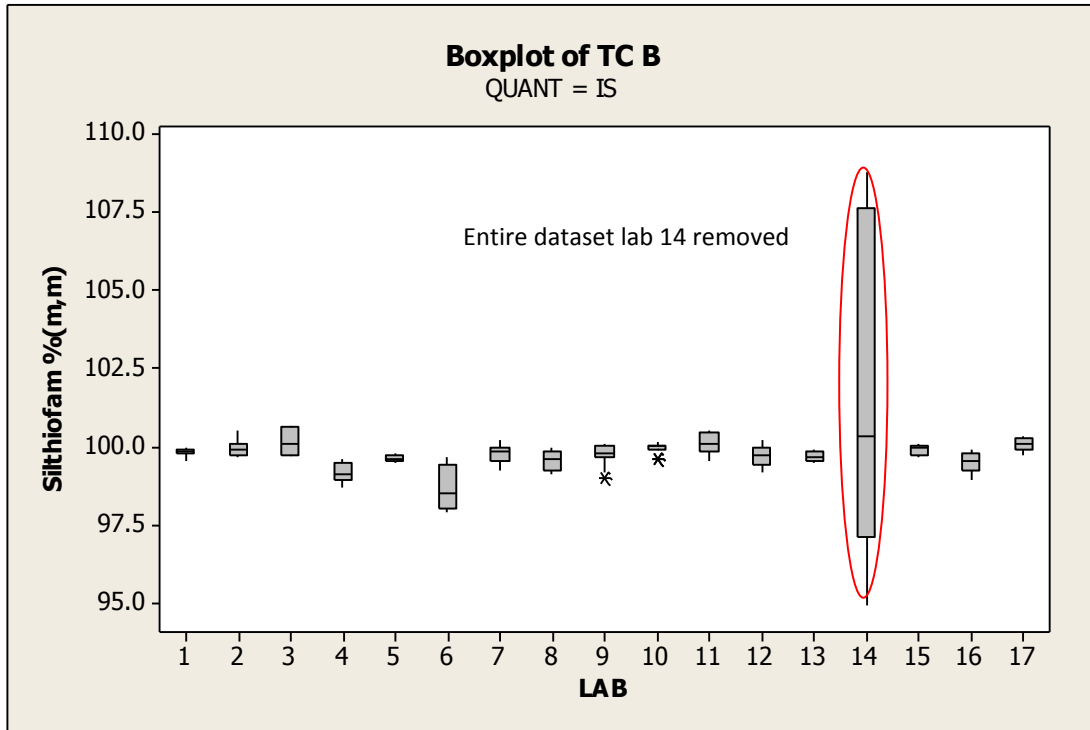


Figure 2B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-B - Box-plot diagram showing strong outliers.

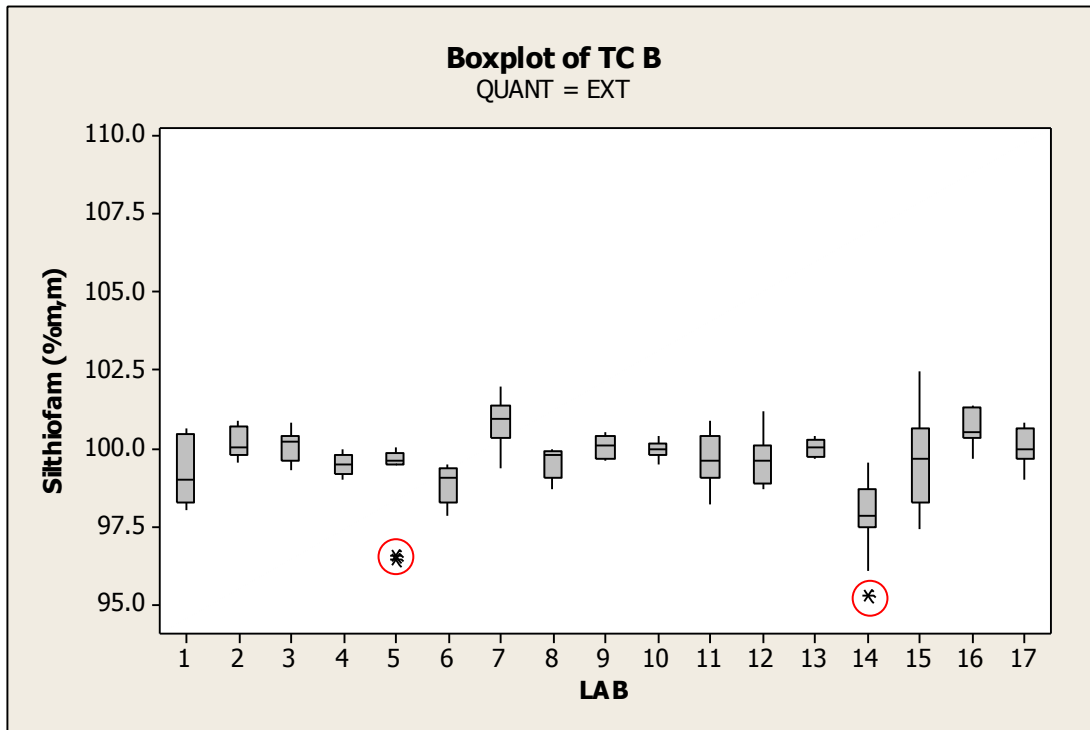


Figure 3A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-C - Box-plot diagram showing strong outliers.

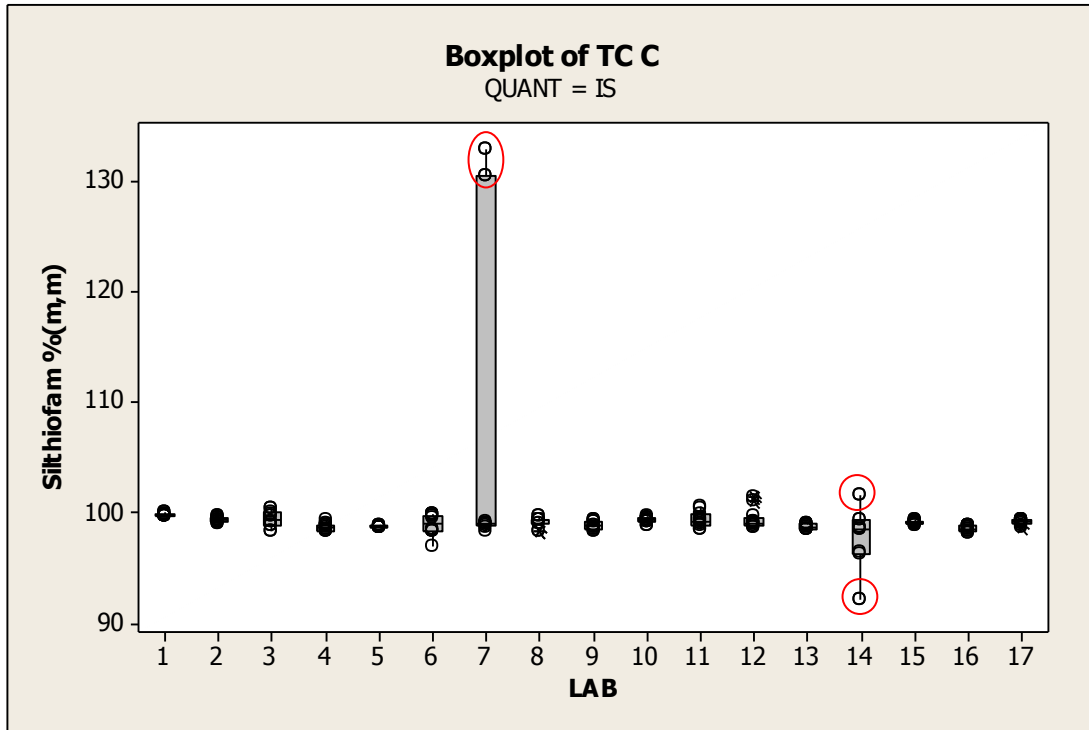


Figure 3B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-C - Box-plot diagram showing strong outliers.

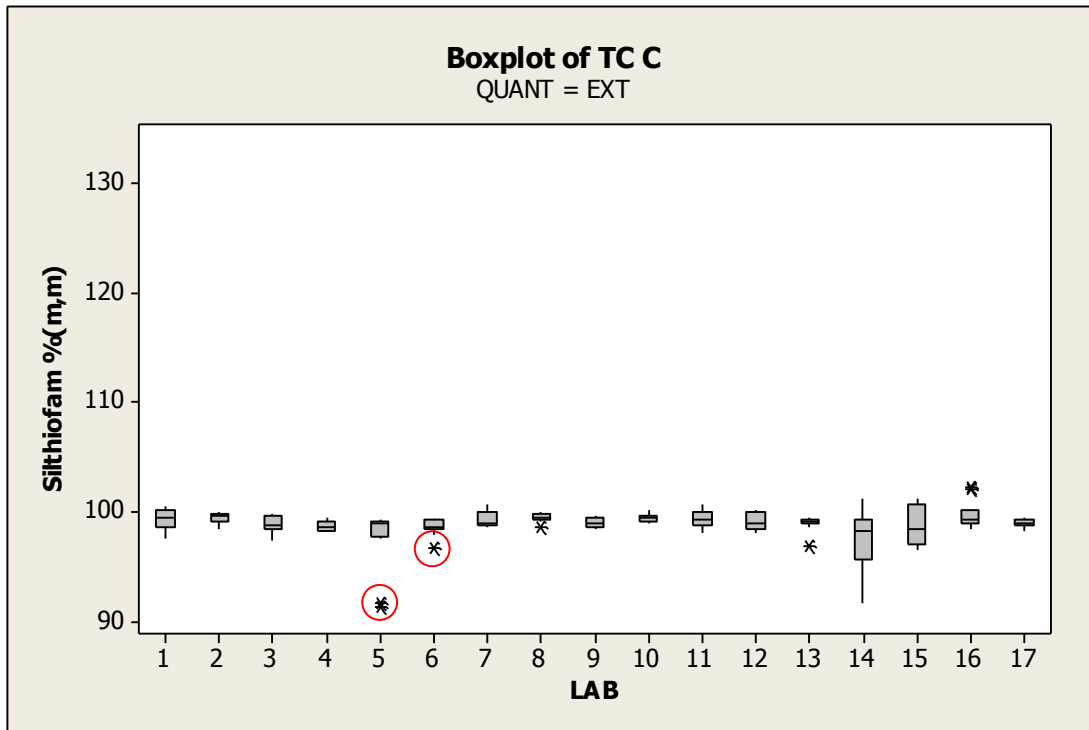


Figure 4A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-A - Box-plot diagram showing strong outliers.

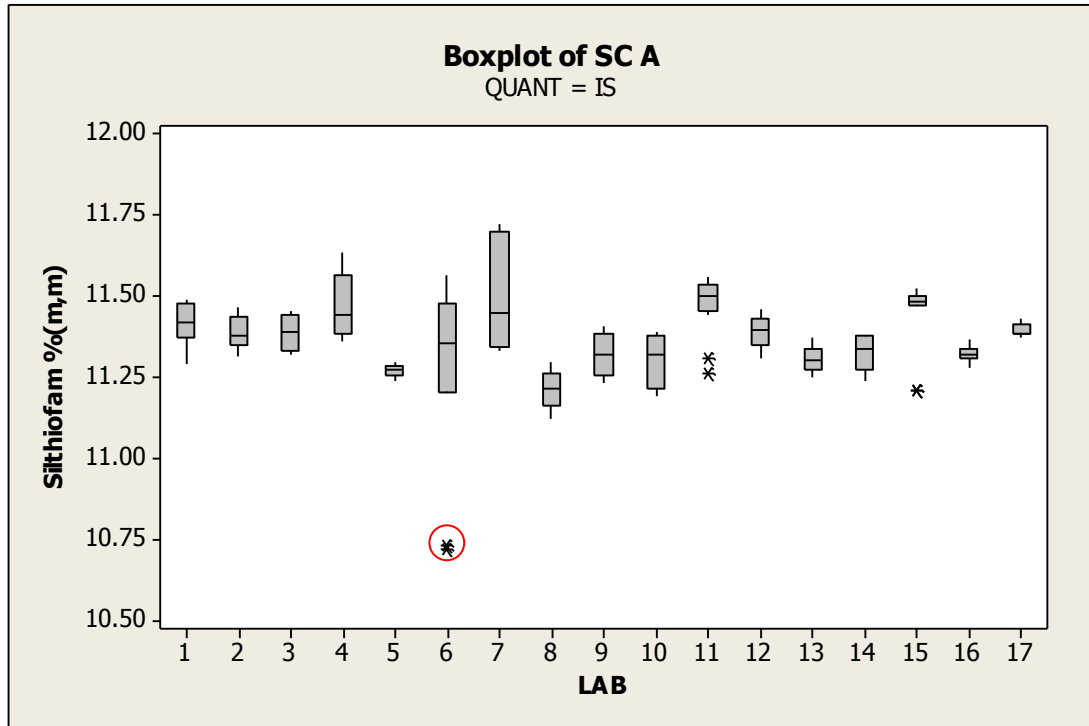


Figure 4B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-A - Box-plot diagram showing strong outliers.

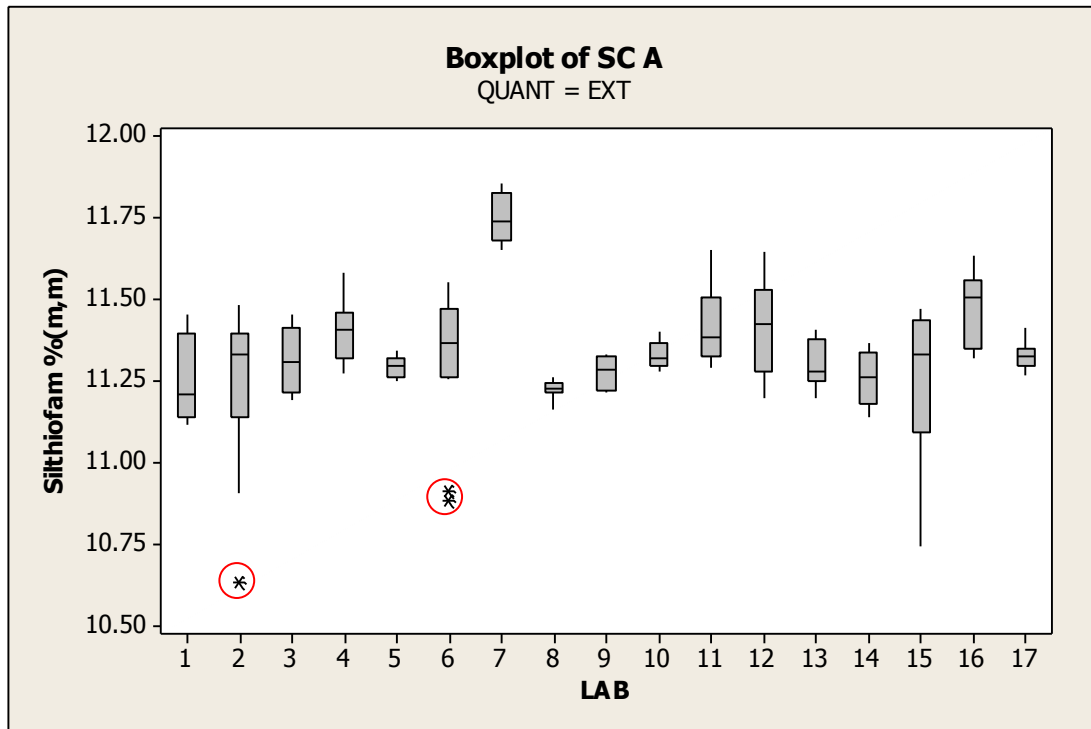


Figure 5A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-B - Box-plot diagram showing strong outliers.

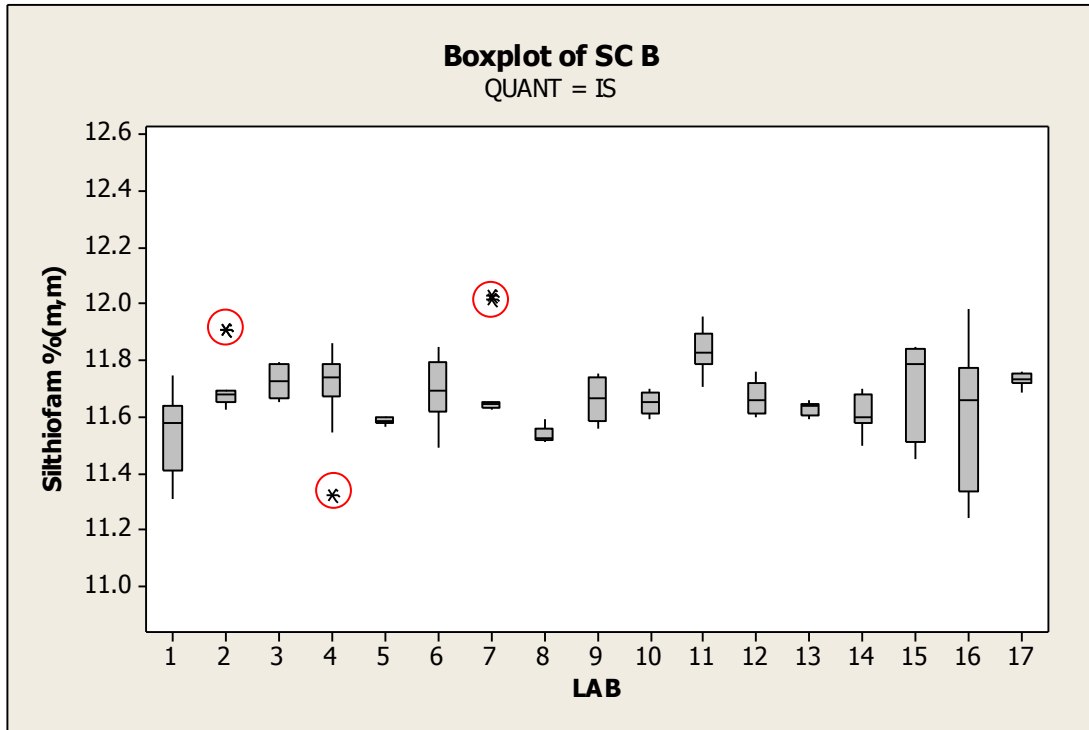


Figure 5B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-B - Box-plot diagram showing strong outliers.

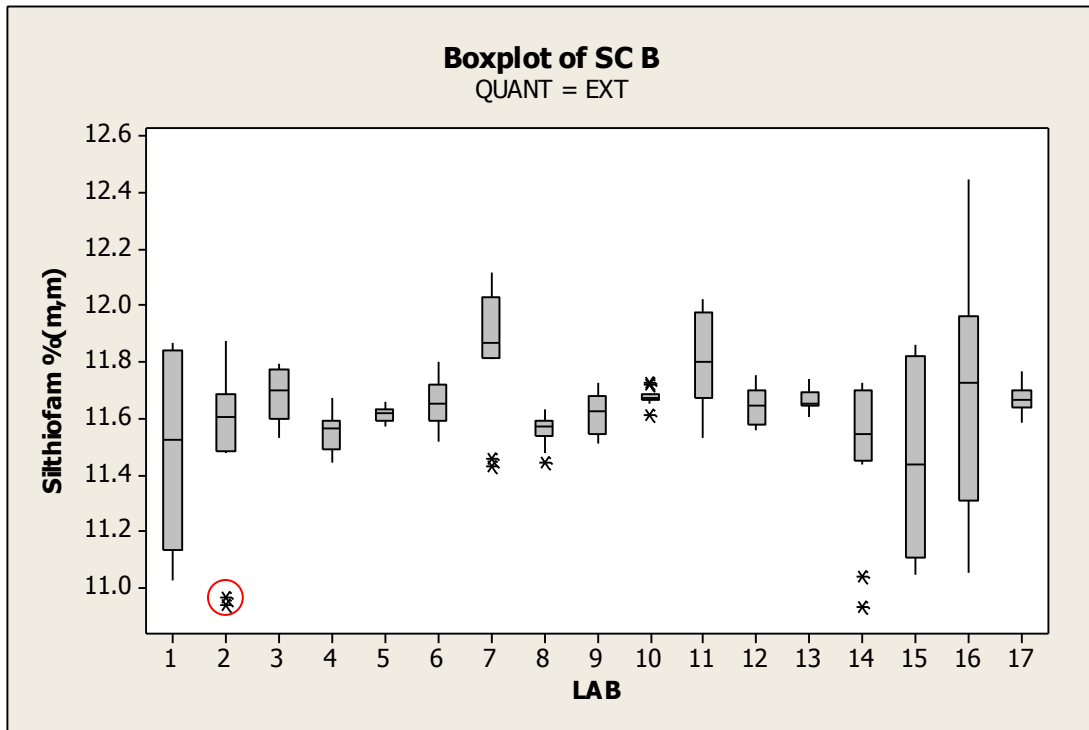


Figure 6A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-C - Box-plot diagram showing strong outliers.

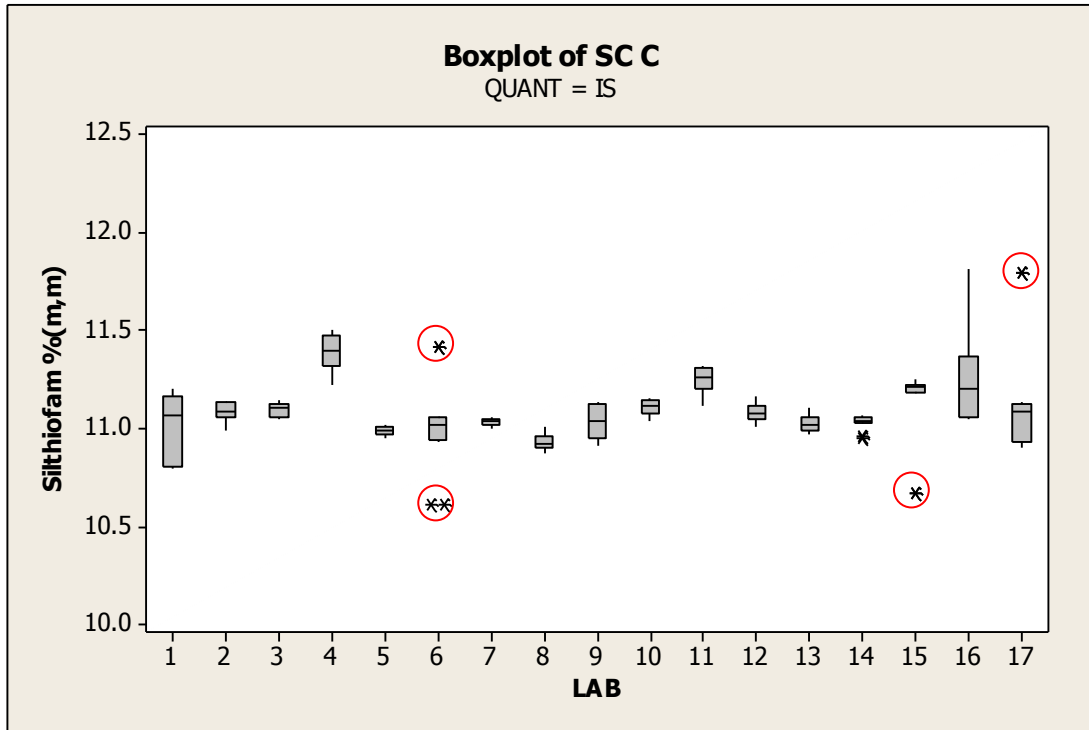


Figure 6B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-C - Box-plot diagram showing strong outliers.

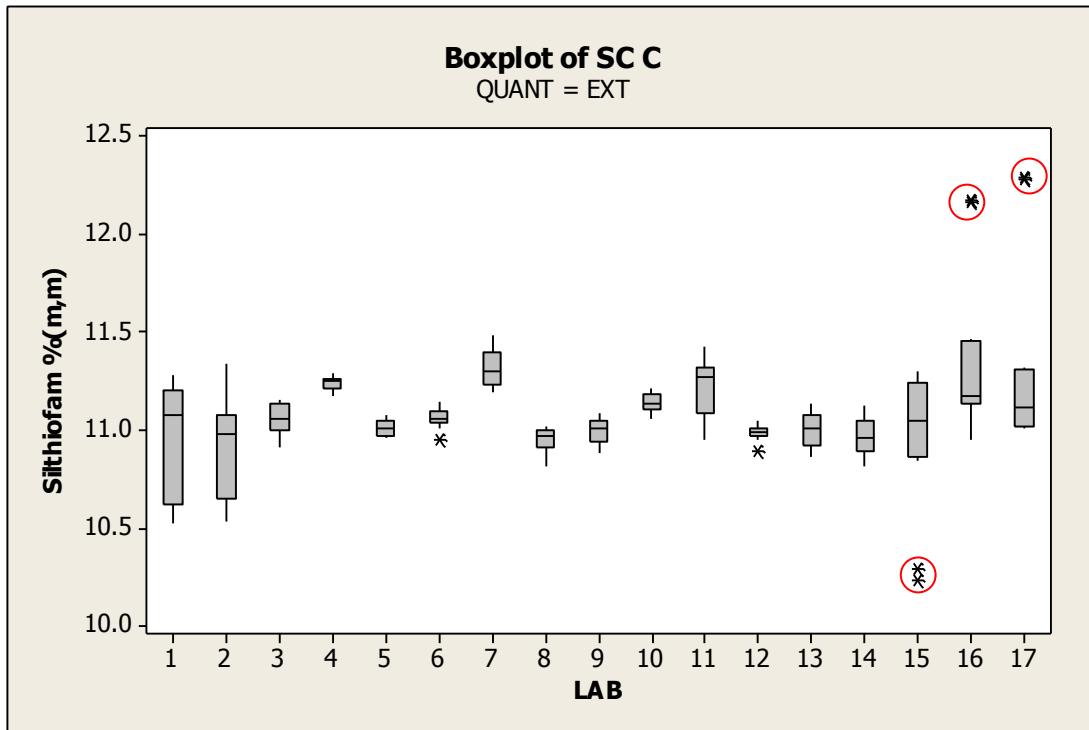


Figure 7A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-D - Box-plot diagram showing strong outliers.

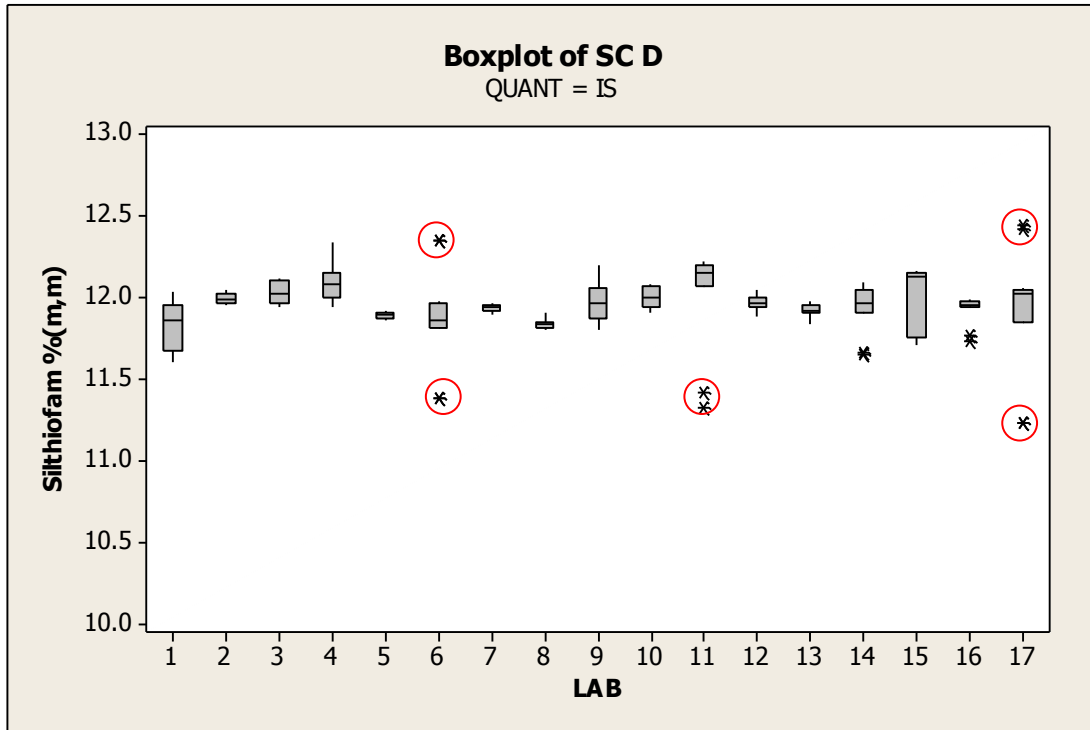


Figure 7B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-D - Box-plot diagram showing strong outliers.

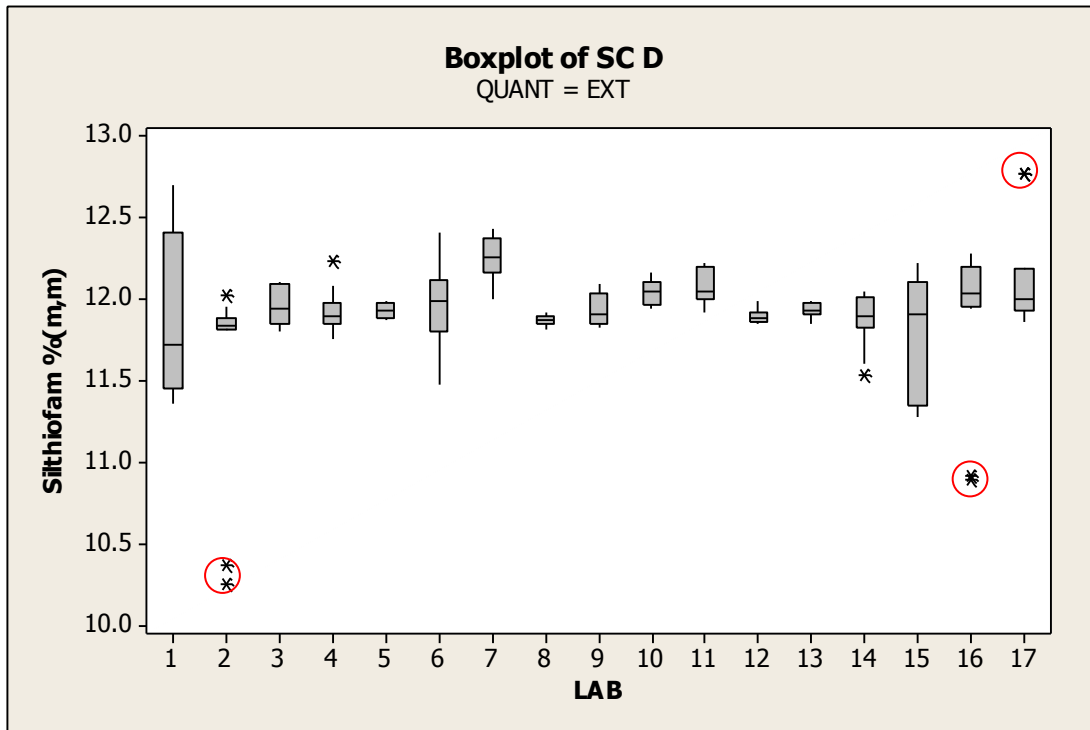


Figure 8A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-E - Box-plot diagram showing strong outliers.

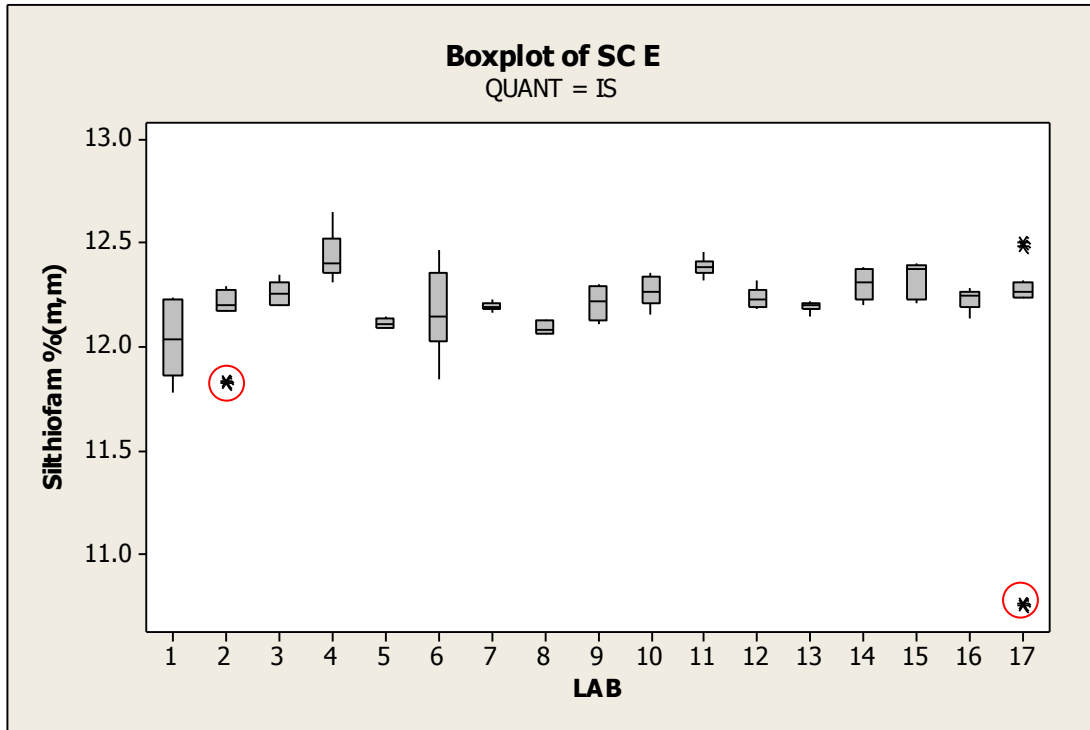


Figure 8B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-E - Box-plot diagram showing strong outliers.

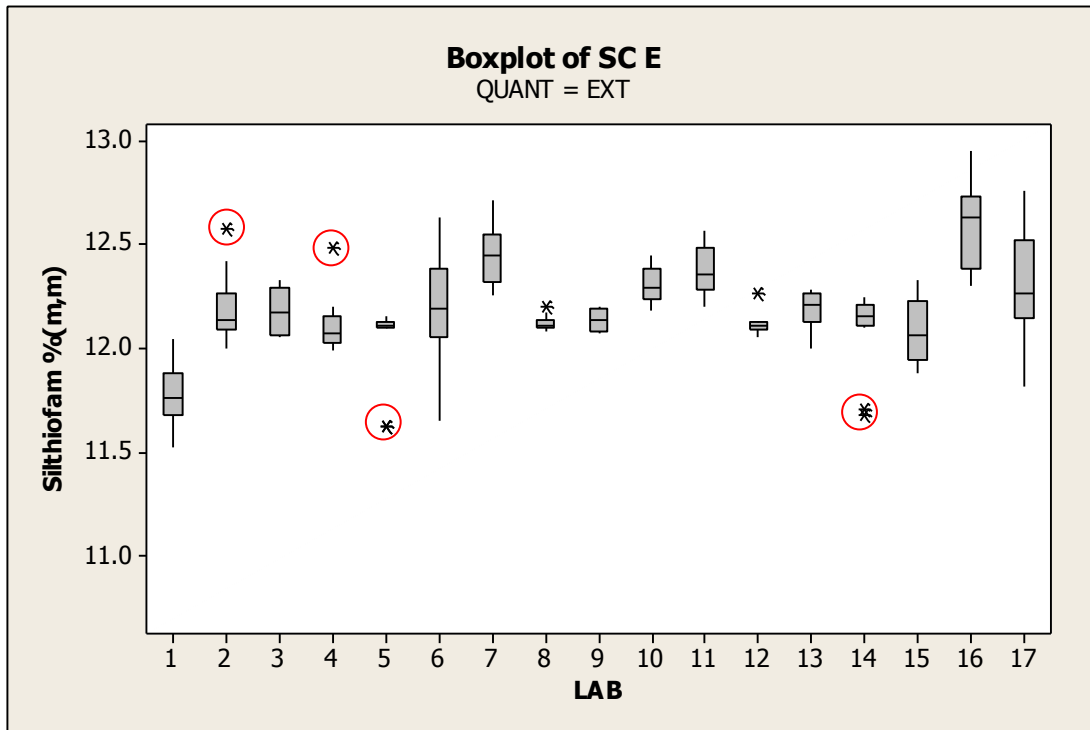


Figure 9A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-A - only strong outliers removed (box-plot diagram)

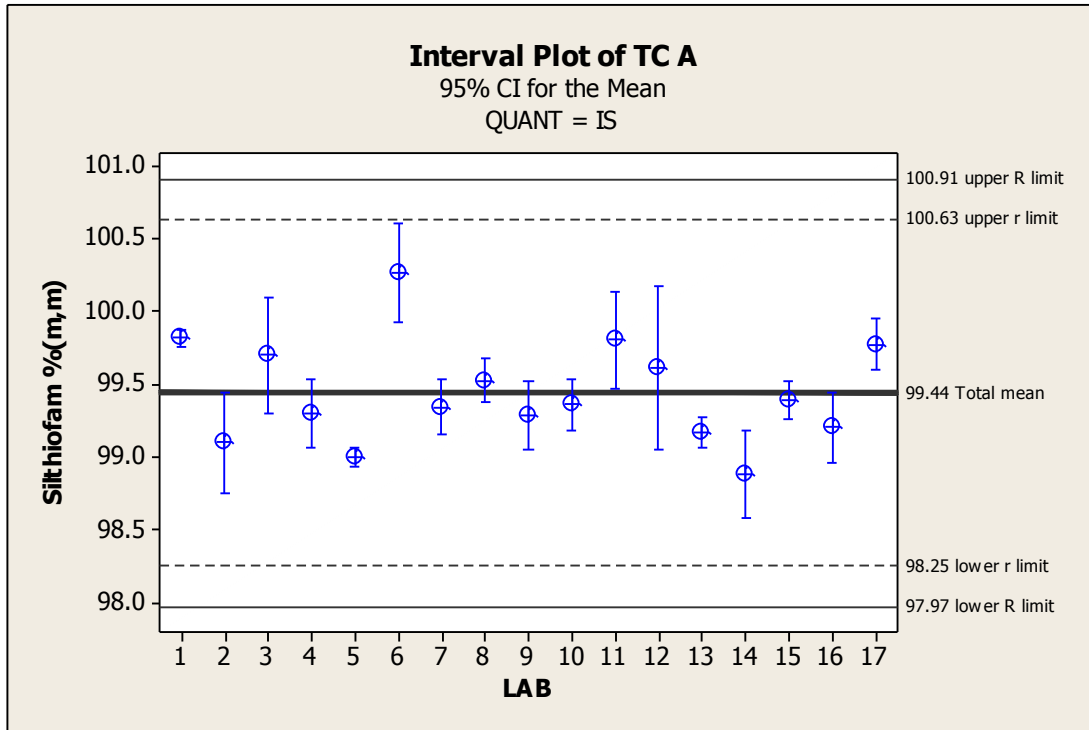


Figure 9B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-A - only strong outliers removed (box-plot diagram)

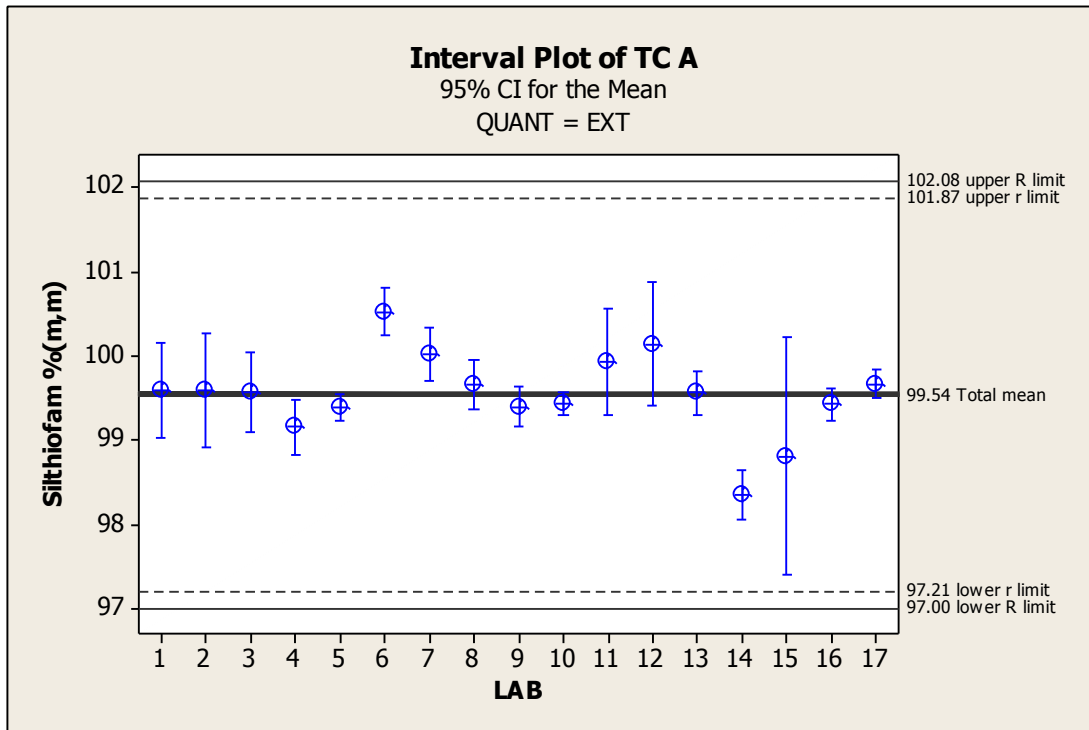


Figure 10A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-B - only strong outliers removed (box-plot diagram)

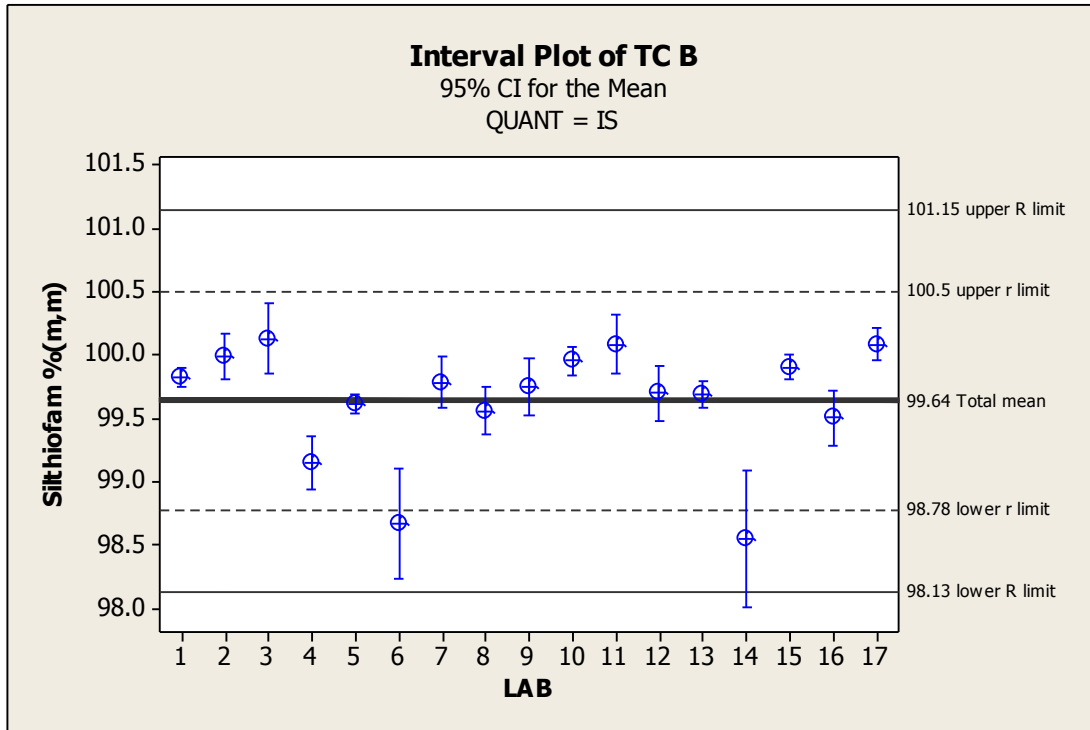


Figure 10B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-B - only strong outliers removed (box-plot diagram)

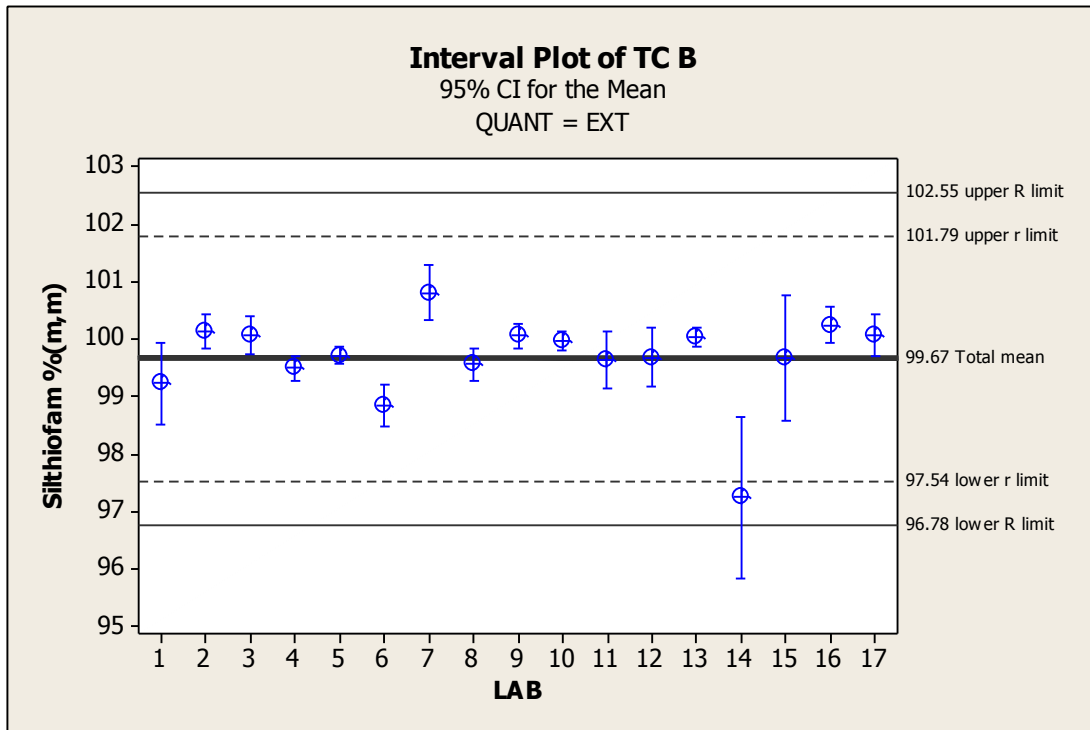


Figure 11A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-C - only strong outliers removed (box-plot diagram)

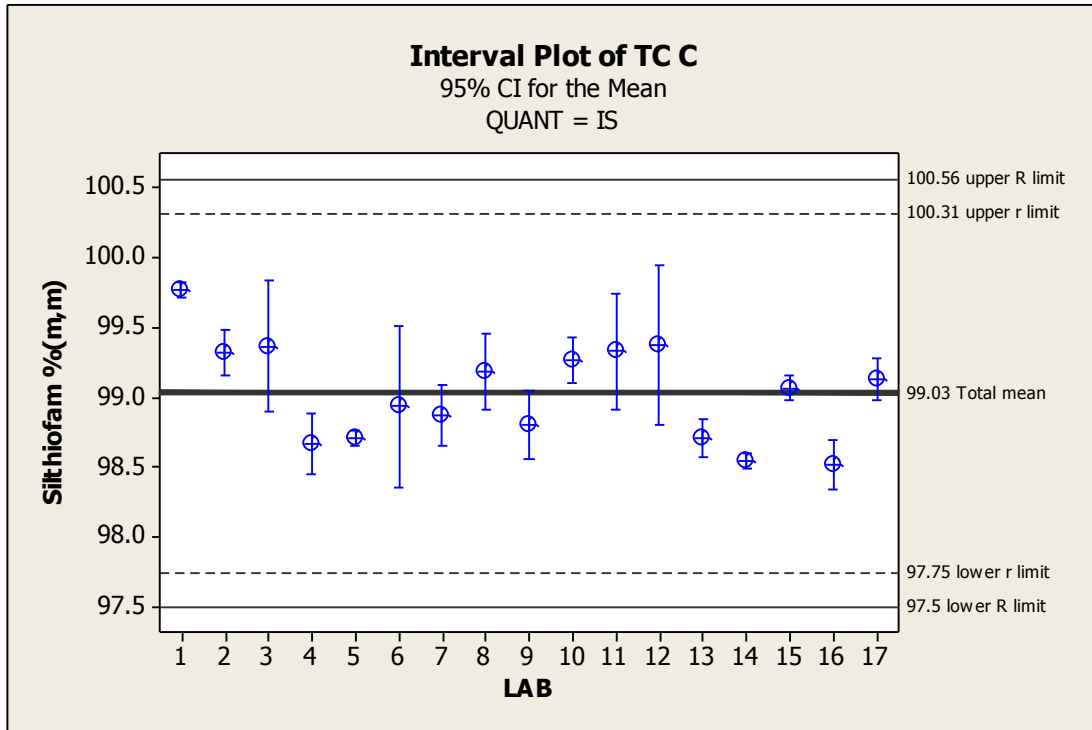


Figure 11B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-C - only strong outliers removed (box-plot diagram)

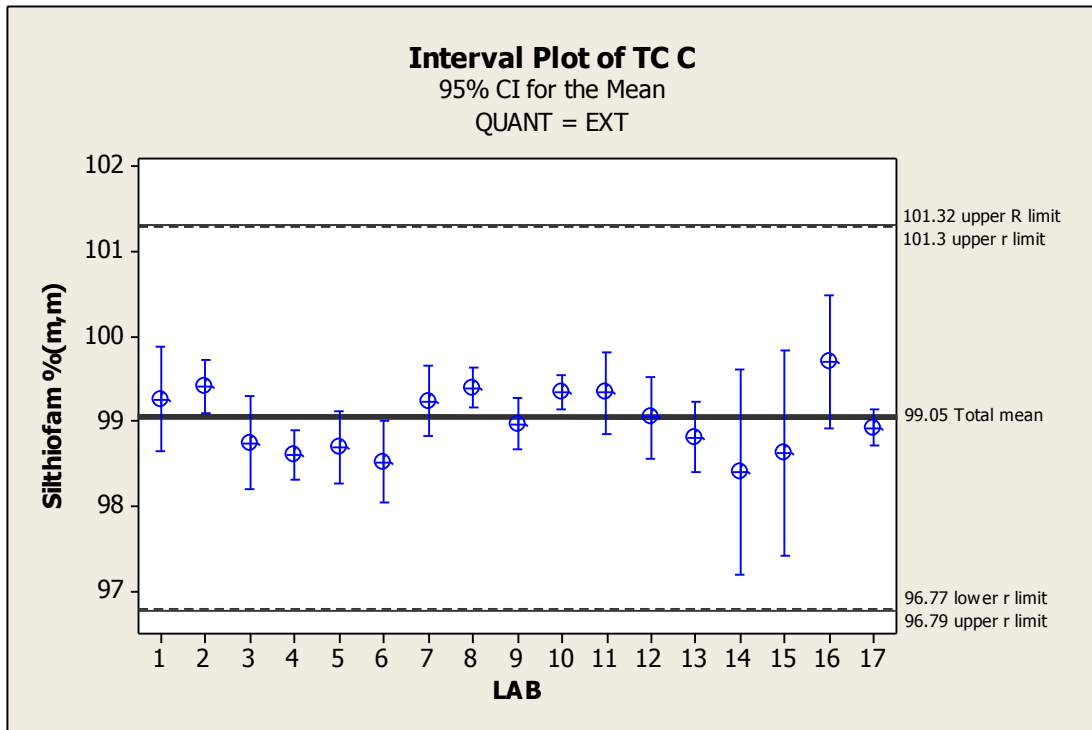


Figure 12A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-A - only strong outliers removed (box-plot diagram)

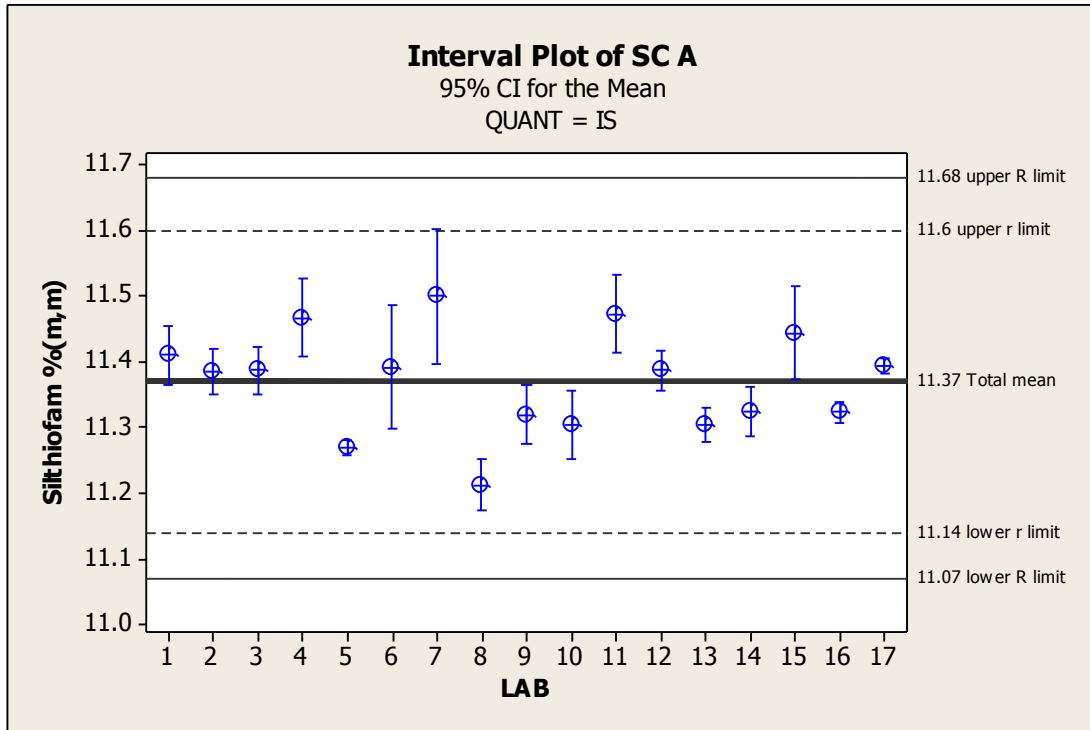


Figure 12B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-A - only strong outliers removed (box-plot diagram)

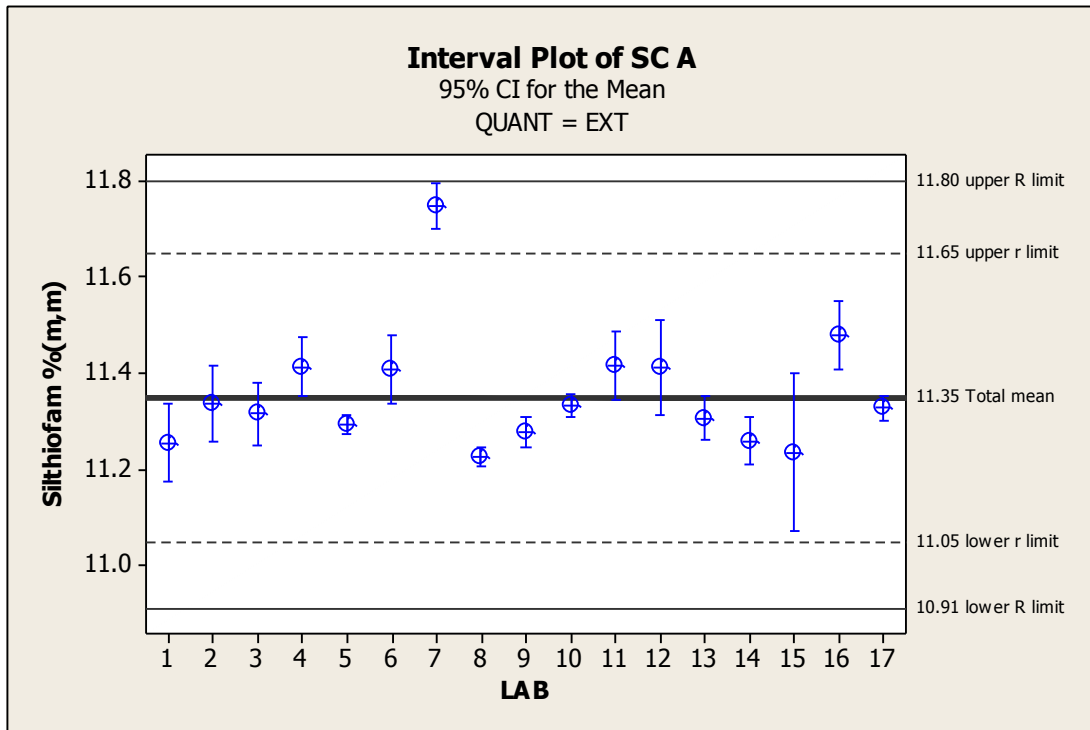


Figure 13A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-B - only strong outliers removed (box-plot diagram)

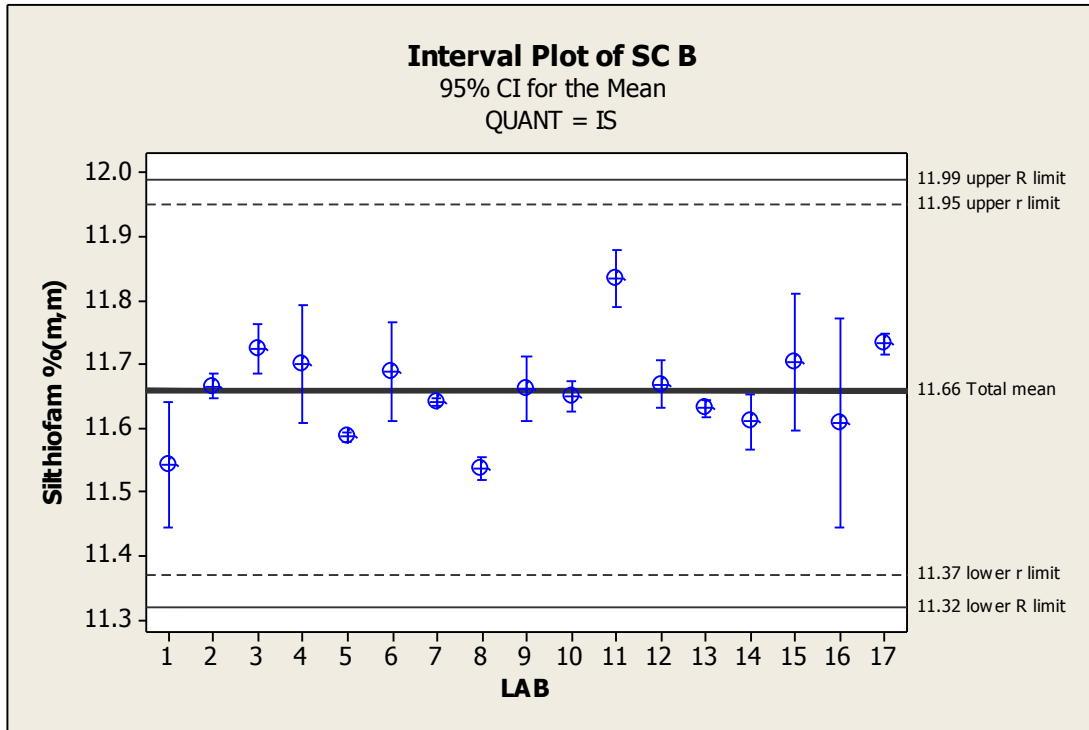


Figure 13B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-B - only strong outliers removed (box-plot diagram)

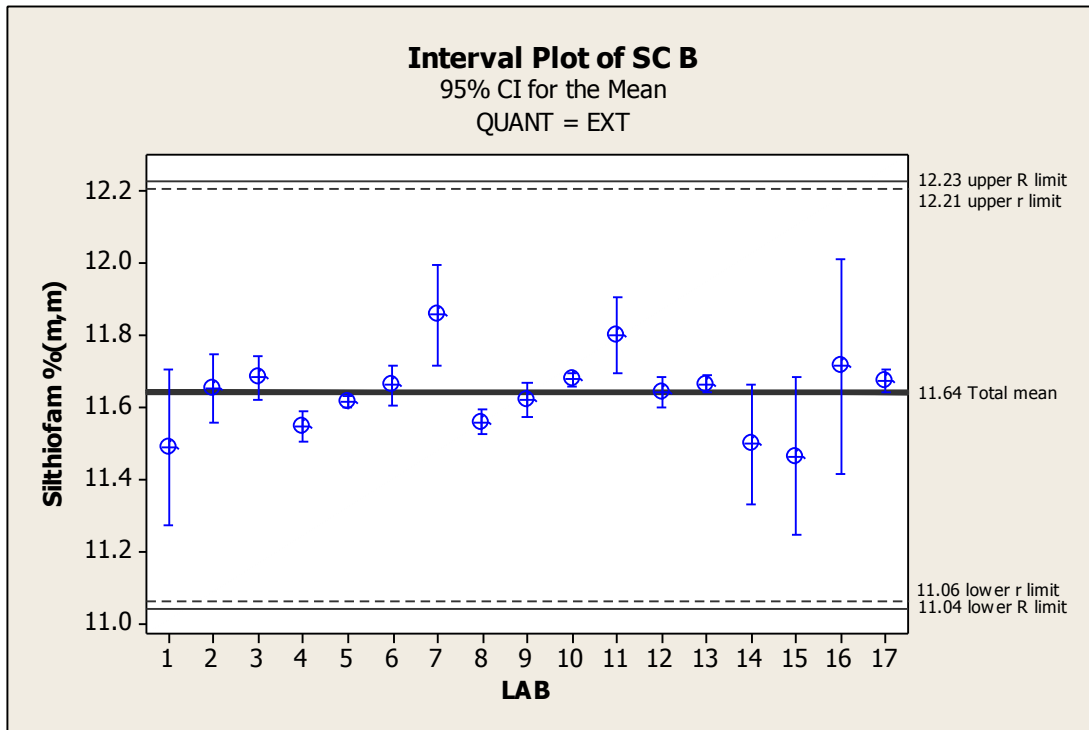


Figure 14A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-C - only strong outliers removed (box-plot diagram)

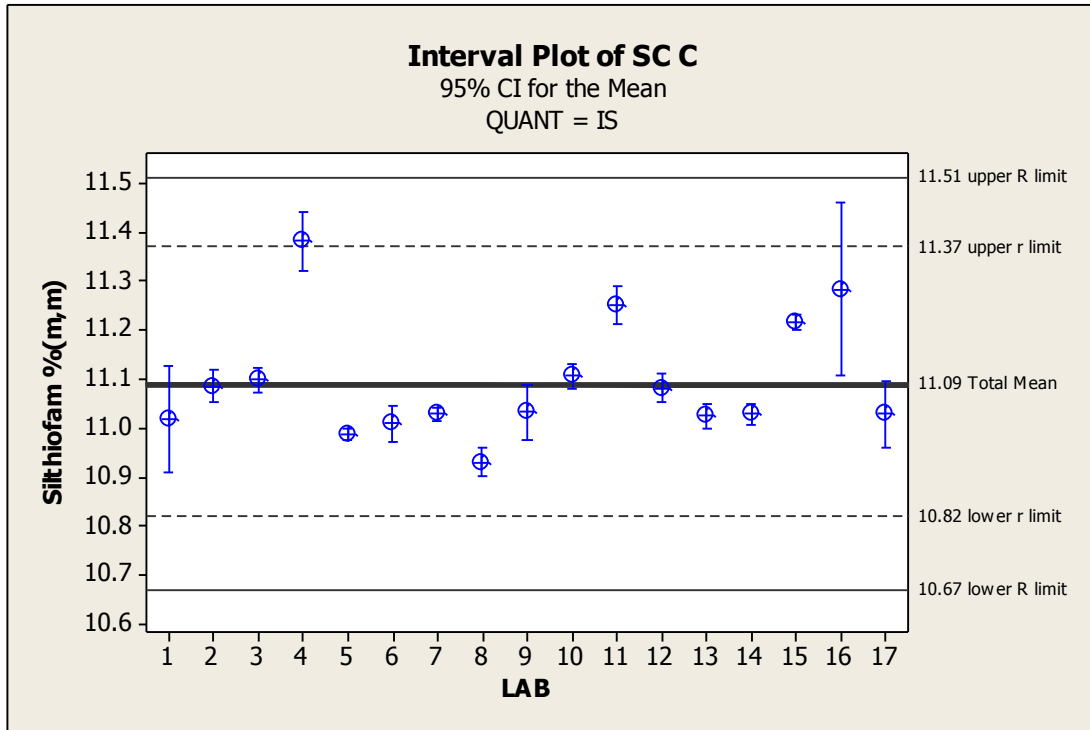


Figure 14B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-C - only strong outliers removed (box-plot diagram)

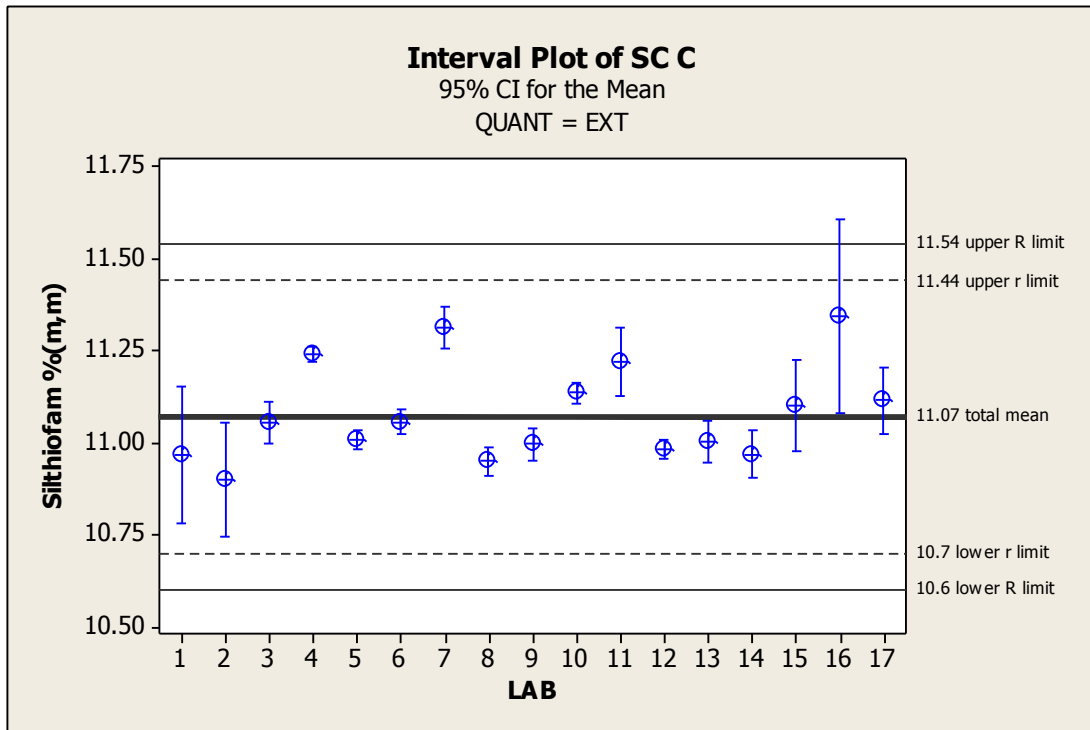


Figure 15A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-D - only strong outliers removed (box-plot diagram)

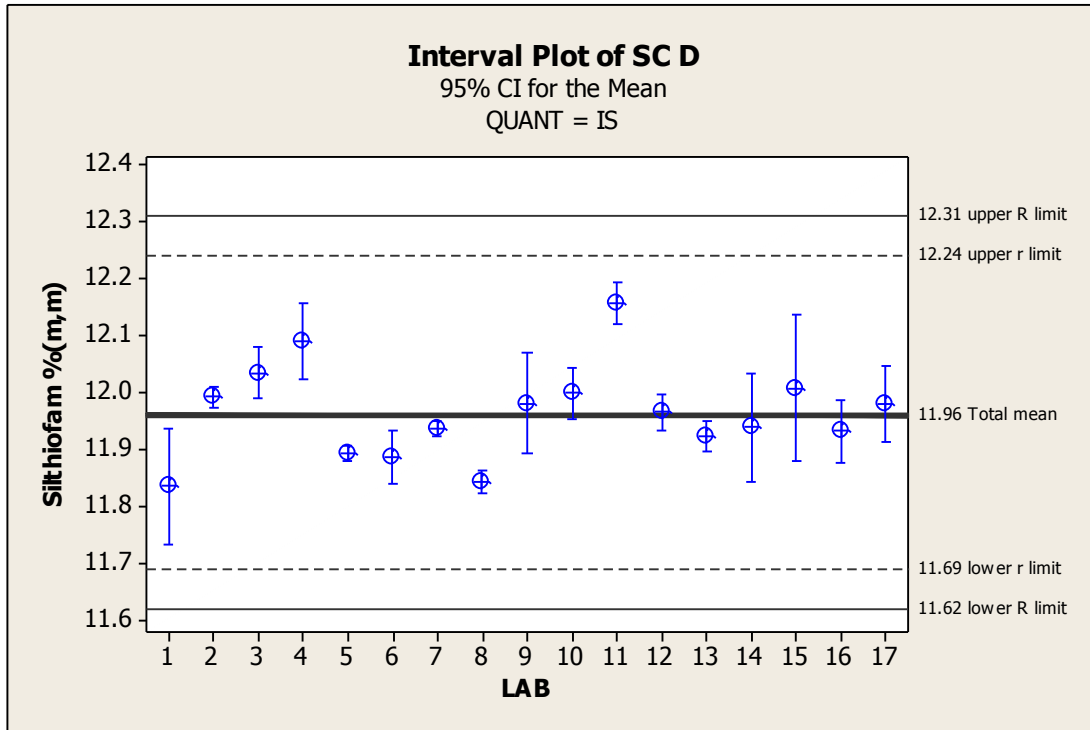


Figure 15B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-D - only strong outliers removed (box-plot diagram)

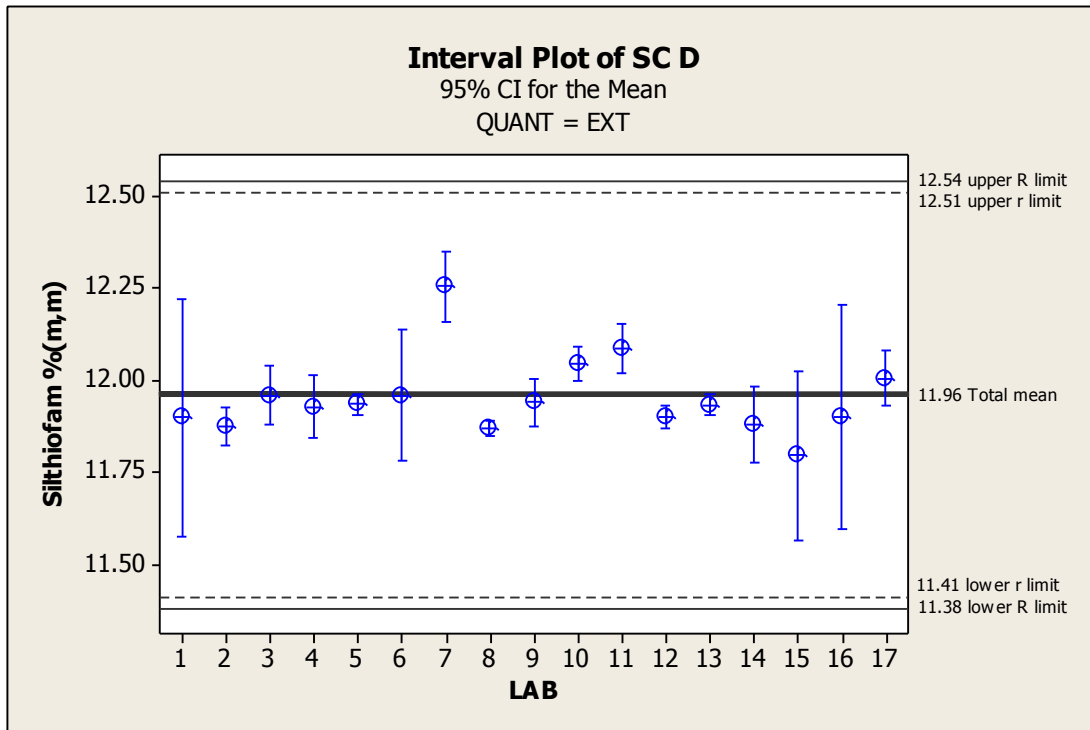


Figure 16A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-E - only strong outliers removed (box-plot diagram)

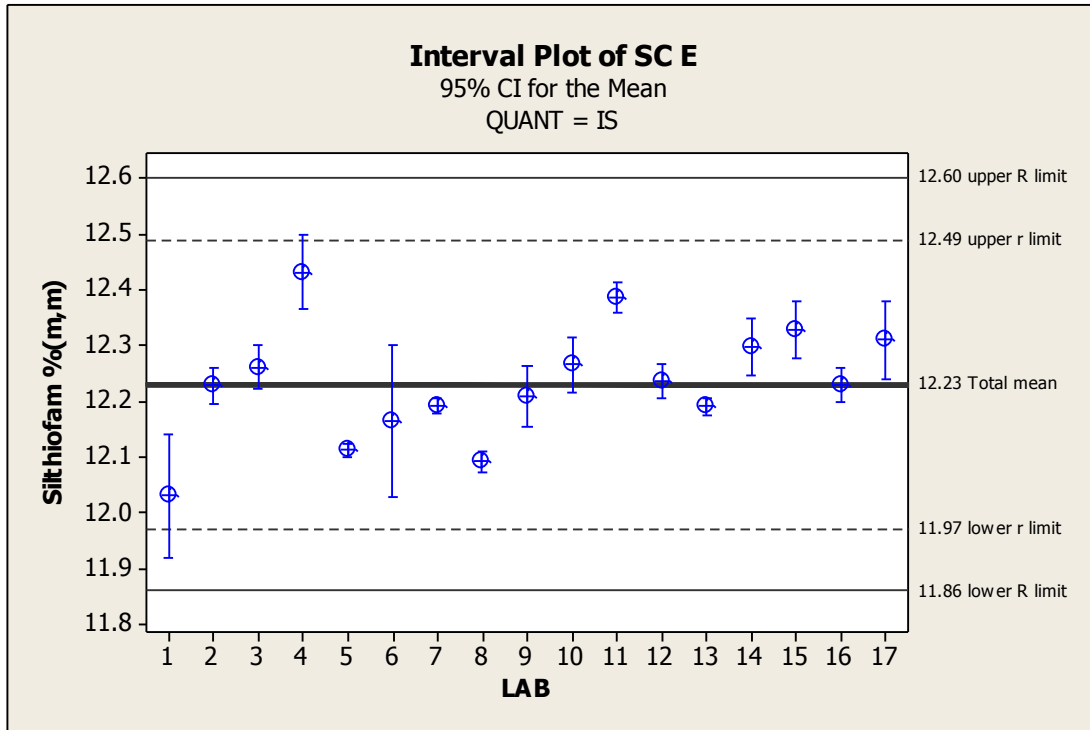


Figure 16B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-E - only strong outliers removed (box-plot diagram)

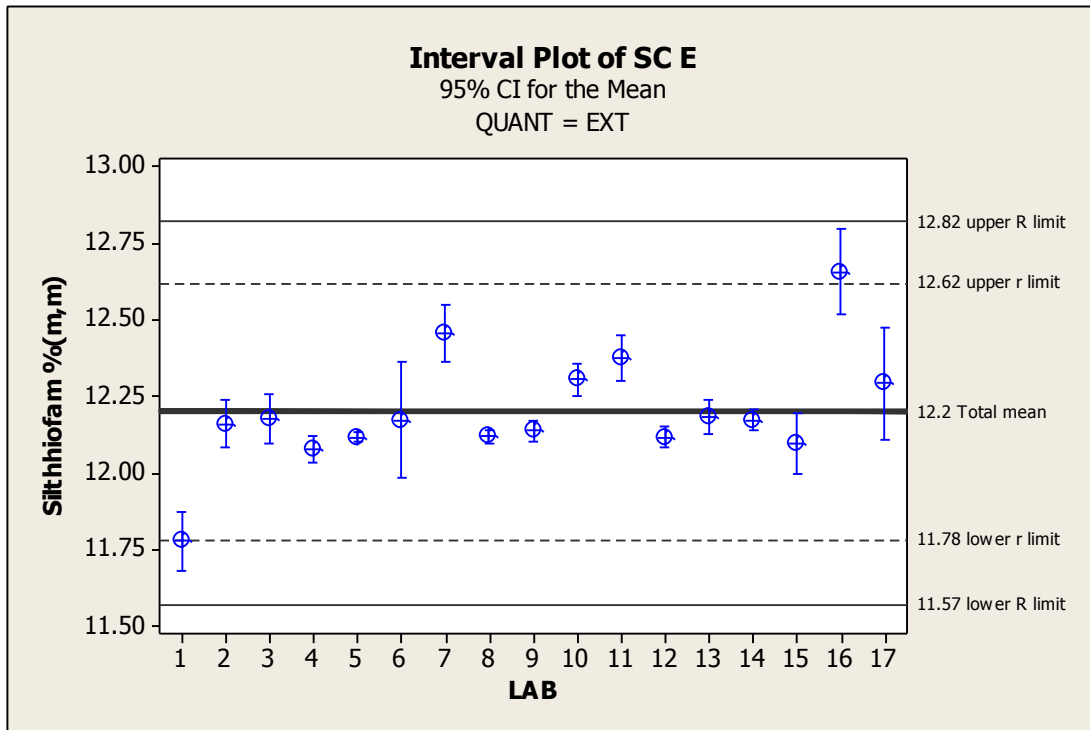


Figure 17A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-A – selected outliers test results removed

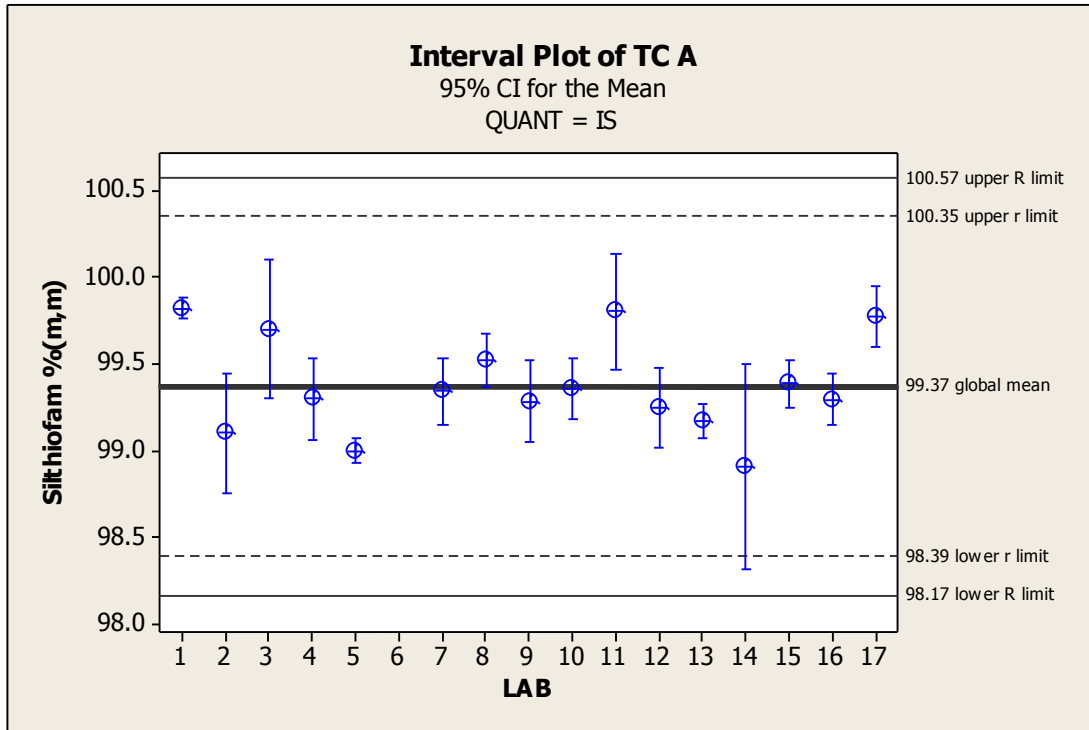


Figure 17B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-A – selected outliers test results removed

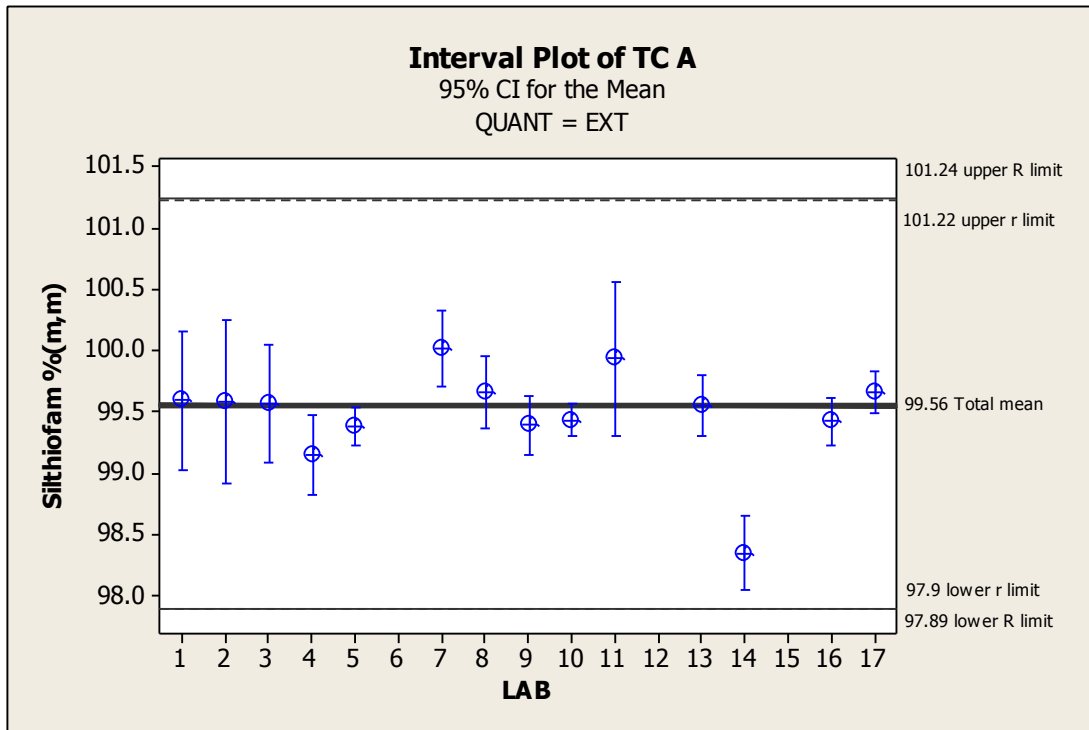


Figure 18A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-B – selected outliers test results removed

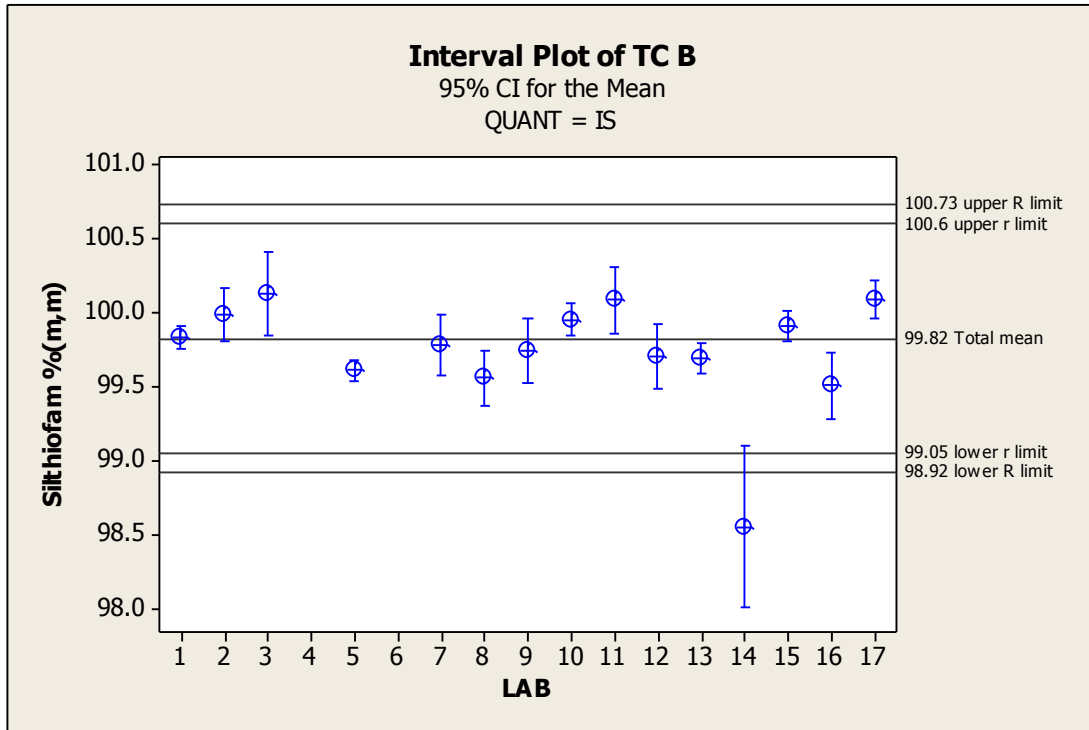


Figure 18B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-B – selected outliers test results removed

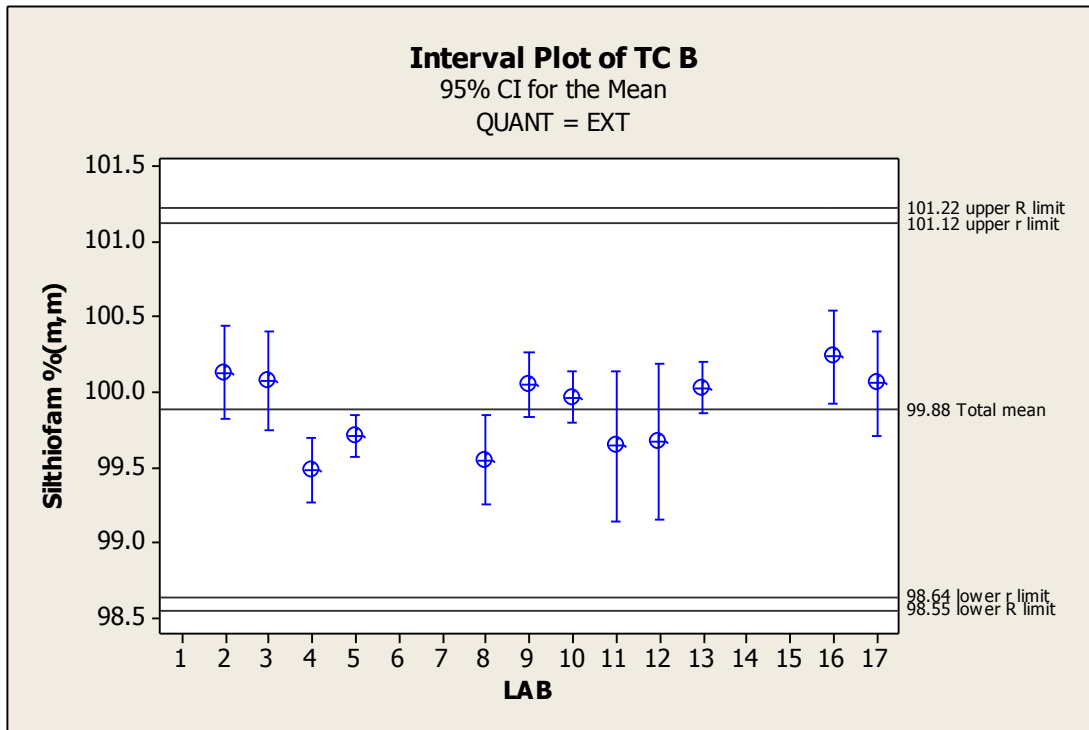


Figure 19A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Technical Sample TC-C – selected outliers test results removed

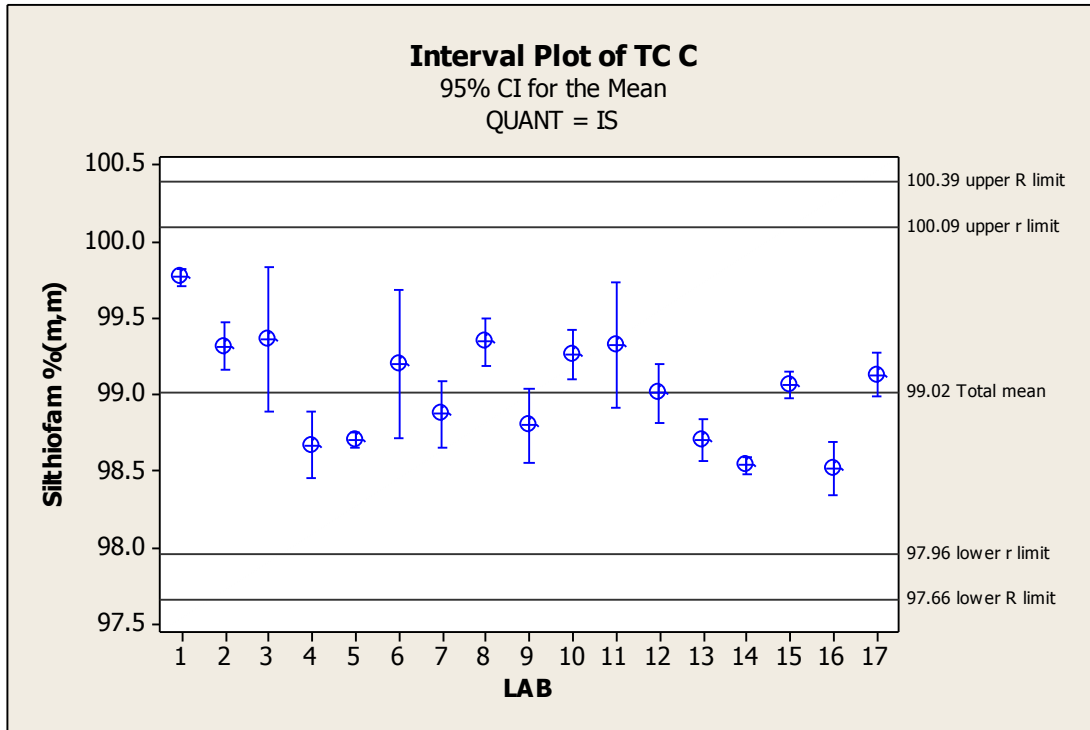


Figure 19B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Technical Sample TC-C – selected outliers test results removed

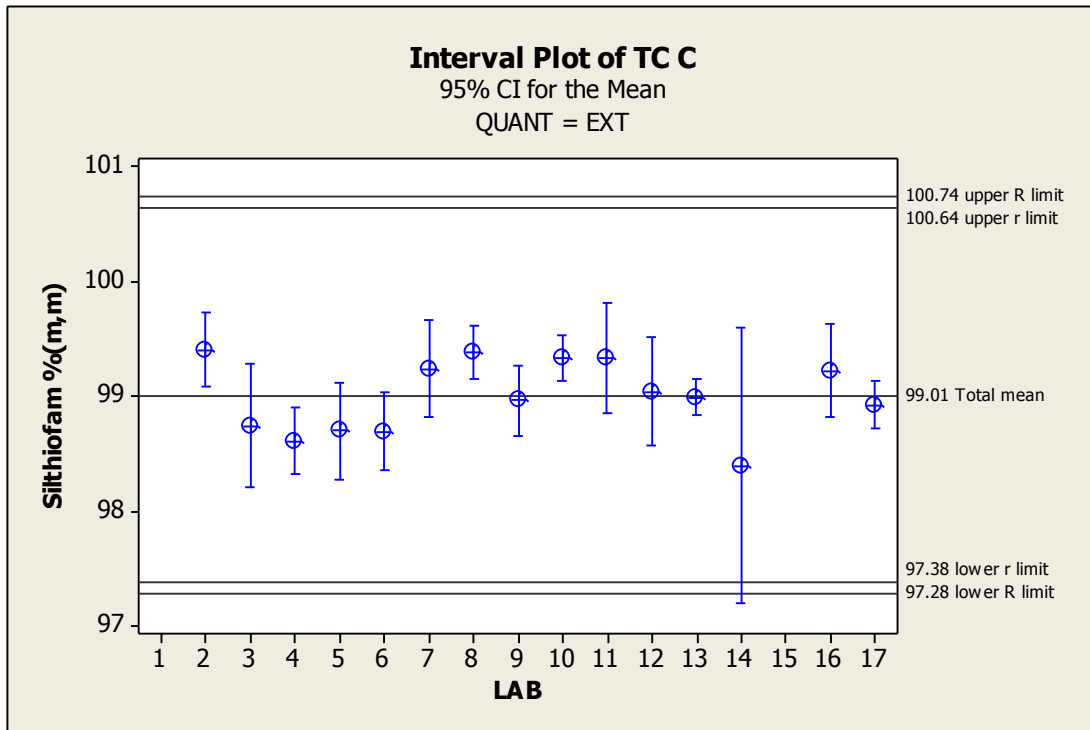


Figure 20A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-A – selected outliers test results removed

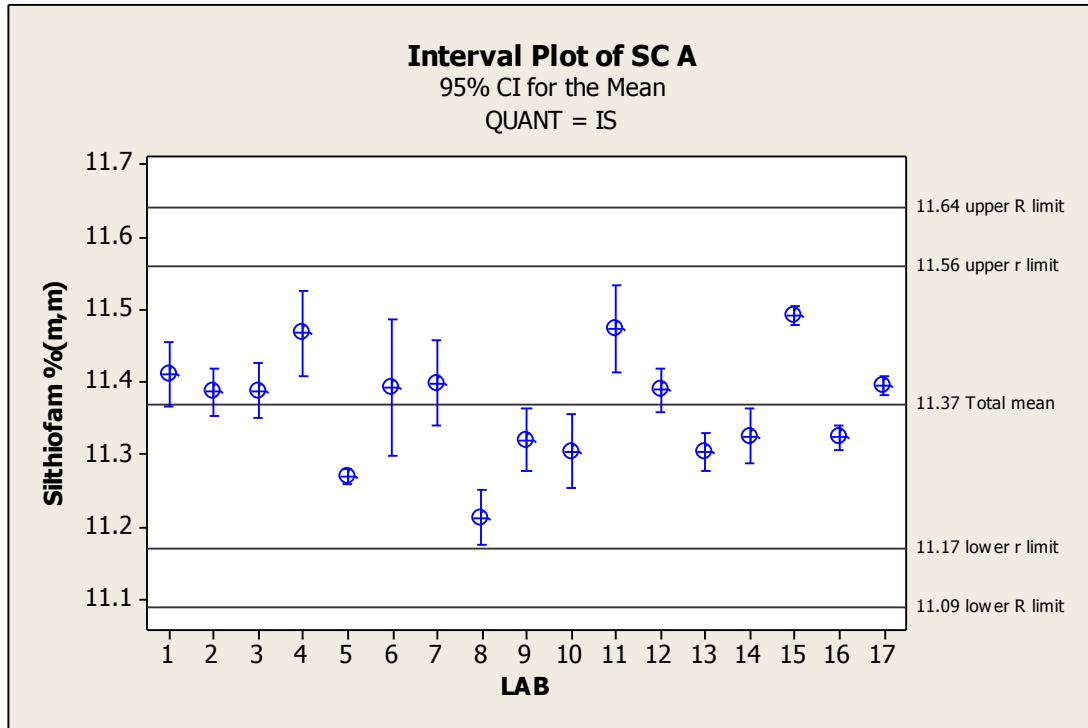


Figure 20B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-A – selected outliers test results removed

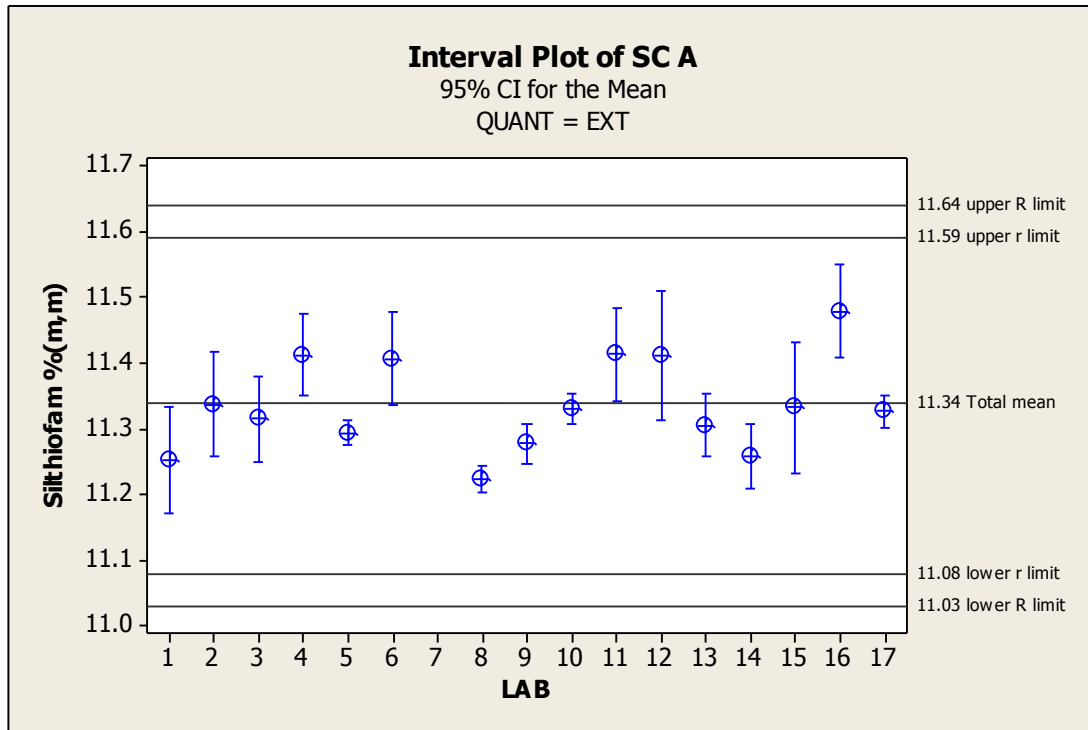


Figure 21A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-B – selected outliers test results removed

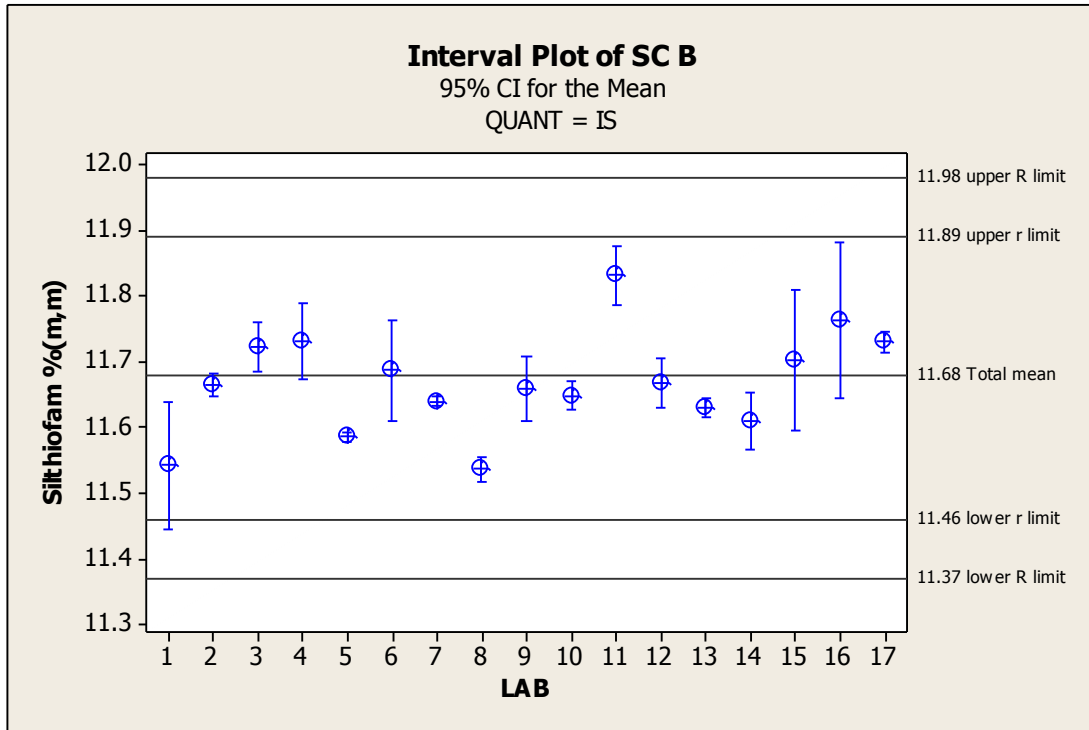


Figure 21B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-B – selected outliers test results removed

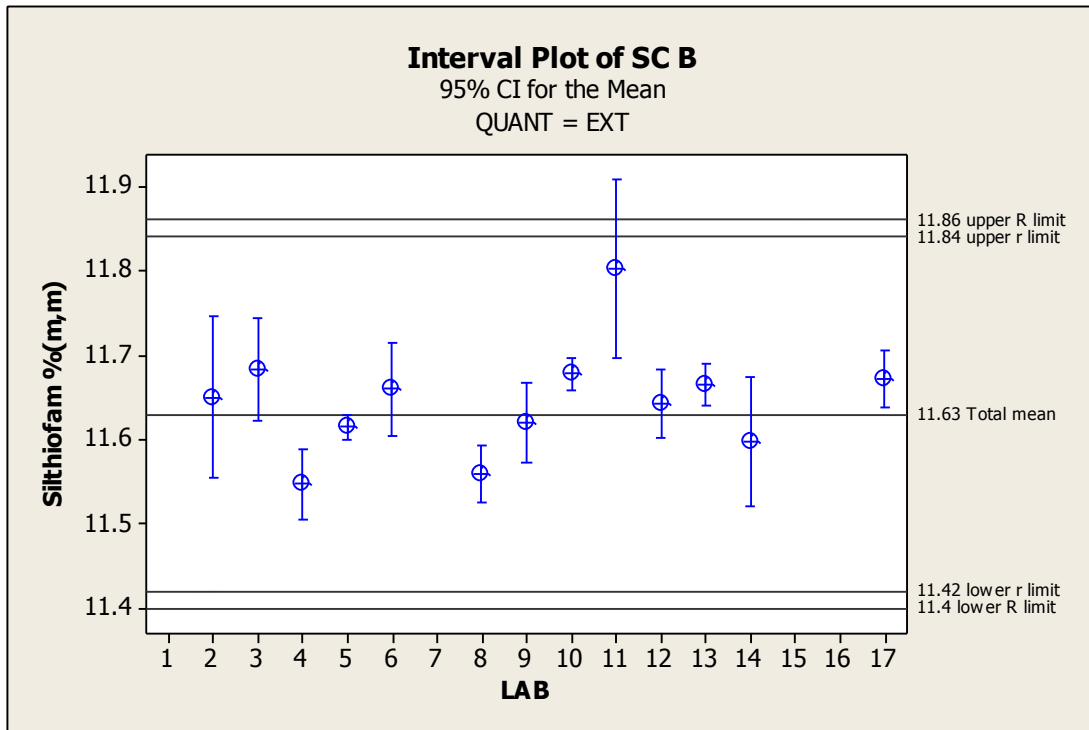


Figure 22A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-C – selected outliers test results removed

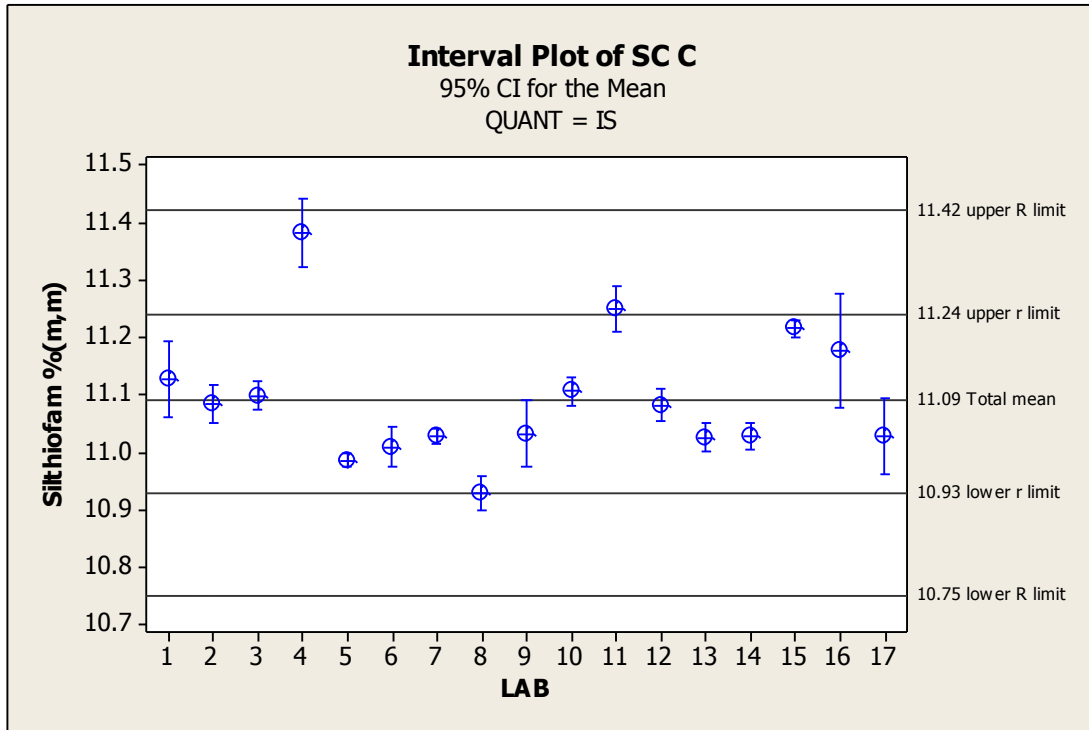


Figure 22B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-C – selected outliers test results removed

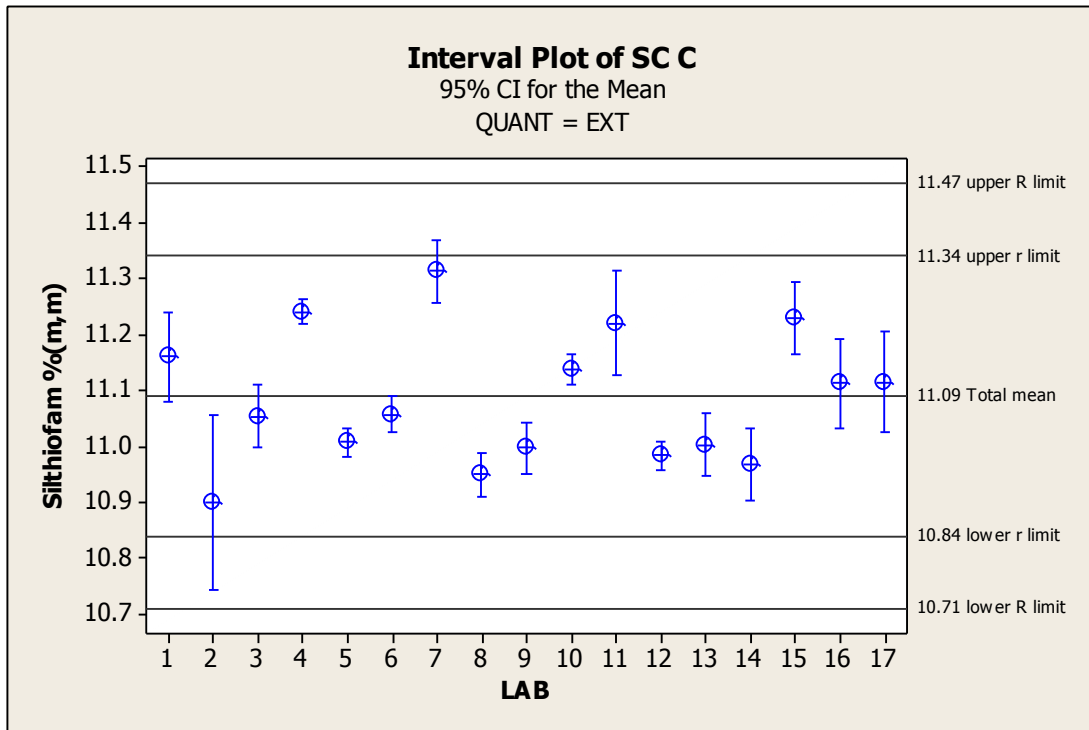


Figure 23A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-D – selected outliers test results removed

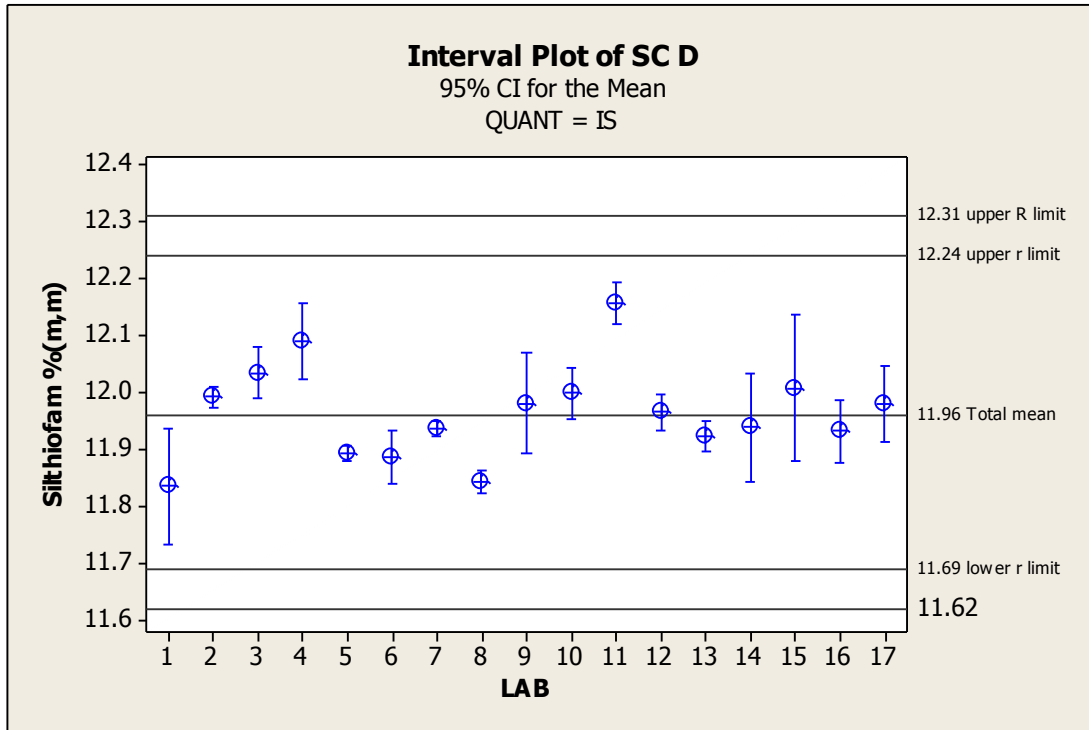


Figure 23B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-D – selected outliers test results removed

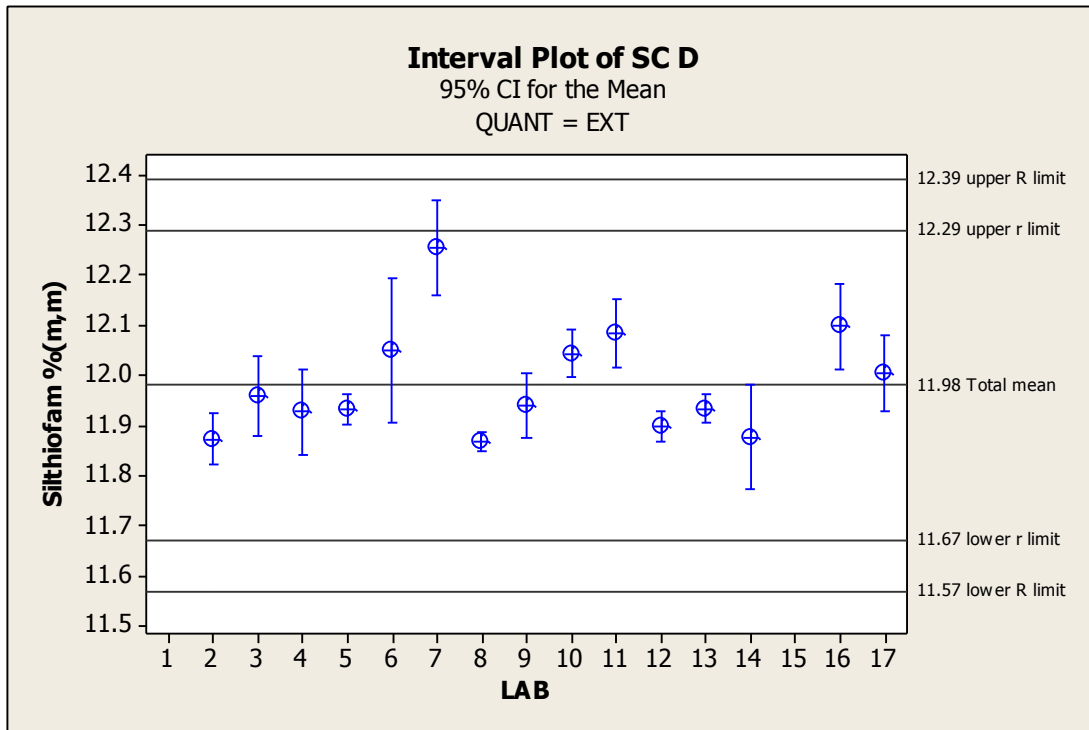


Figure 24A – Assay Results Summary by Lab for Internal Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-E – selected outliers test results removed

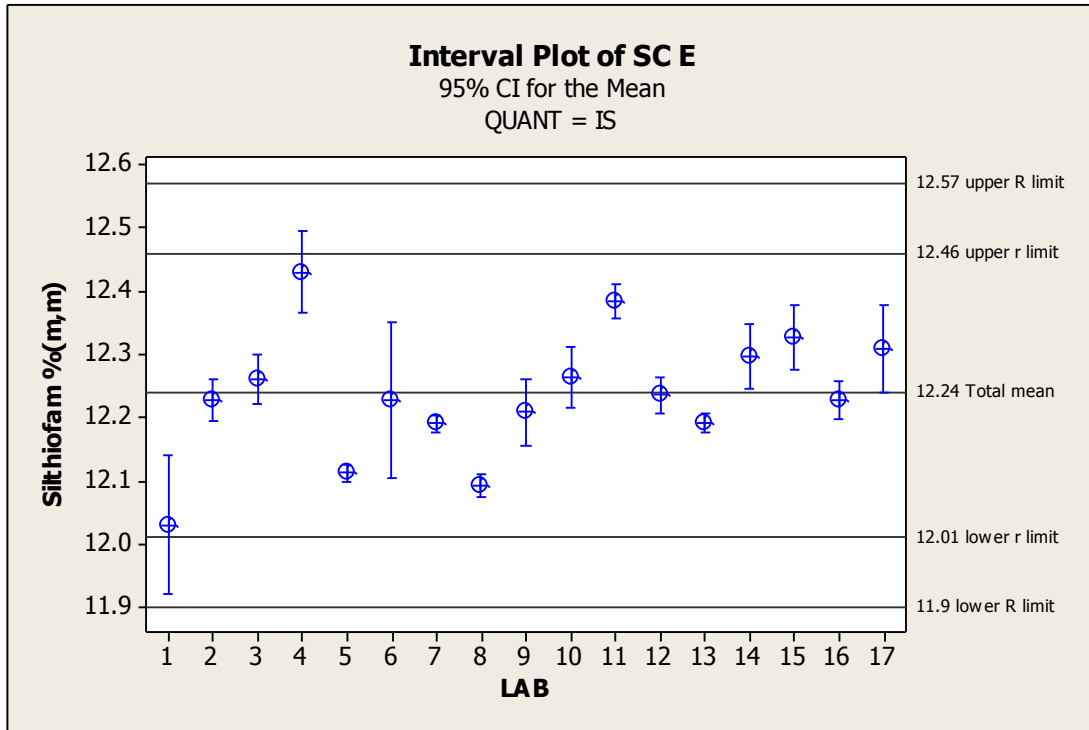


Figure 24B – Assay Results Summary by Lab for External Standard analysis of Silthiofam in Silthiofam Flowable Concentrate Sample FS-E – selected outliers test results removed

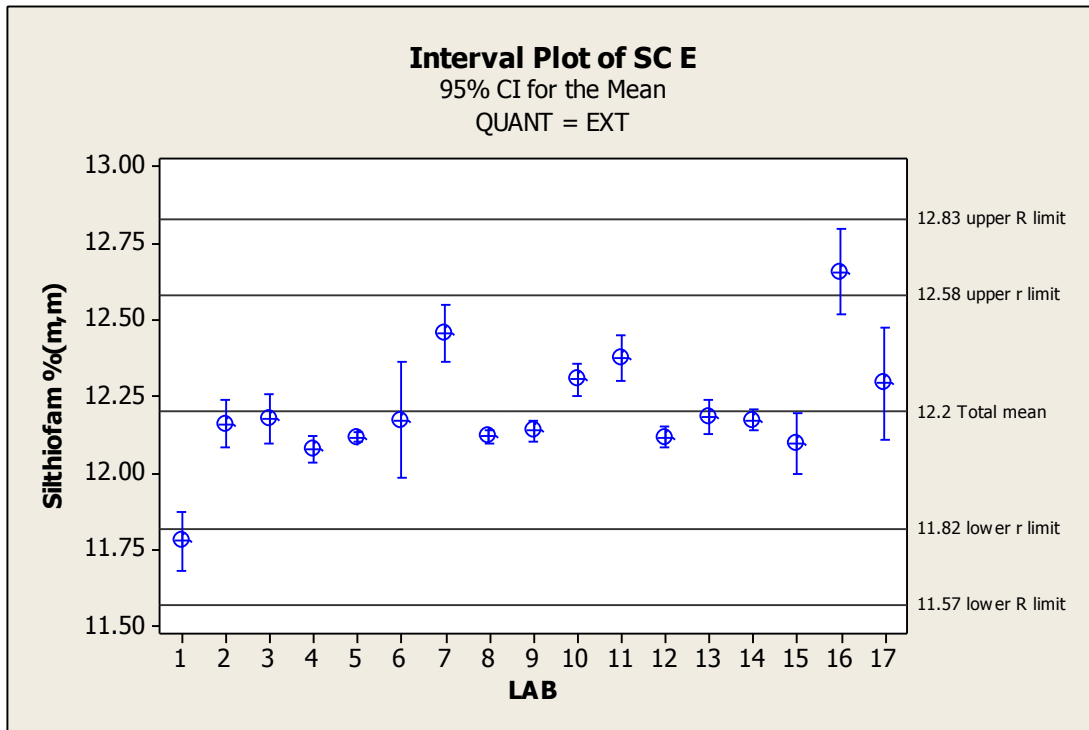


Figure 25 – Comparison %RSDR by Internal and External Standard Analysis Lab for visual outlier removal and selected outlier removal after visual, Cochran and Grubbs investigation.

