Pyroxasulfone
Collaborative Trial Report

Small scale collaborative trial for the determination of Pyroxasulfone in TC, SC and WG

Report to CIPAC by Shandong Binnong Technology Co., Ltd
No. 518, Yongxin Road, Binbei Town, Binzhou, Shandong, China.

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1. Participants and sample distribution

Index	NAME	ORGANIZATION	Address
Lab1	Dong Jie	Shandong Binnong Technology Co., Ltd.	No.518, Yongxin Road, Binbei Town, Binzhou, Shandong, China
Lab2	Wendy Wang	Jiangsu Agrochem Laboratory Co., Ltd.	No.98, Minjiang Road, Hi-Tech Development Zone Changzhou, Jiangsu, China
Lab3	Shen Peng	JiangSu EverTest Co., Ltd.	No. 31-1, Hengjing Road, Economic and Technological Development Zone, Nanjing, Jiangsu, China

2. Sample information

Sample	Quantity	Batch	Declared Content of AI
Pyroxasulfone TC-1	100 g	20240127	980 g/kg
Pyroxasulfone TC-2	100 g	20249223	980 g/kg
Pyroxasulfone SC-1	100 g	20240116	980 g/kg
Pyroxasulfone SC-2	100 mL	20240210	410 g/kg
Pyroxasulfone SC-3	100 mL	20240225	410 g/kg
Pyroxasulfone WG-1	100 mL	20240207	410 g/kg
Pyroxasulfone WG-2	100 mL	20240221	850 g/kg
Pyroxasulfone WG-3	100 mL	20240309	850 g/kg

Pyroxasulfone analytical standard, 99.4 % purity.

3. Method description

3.1 Outline of method

Pyroxasulfone is determined by reversed phase high-performance liquid chromatography using UV detection at 225 nm and external standardization.

3.2 Apparatus and reagents

HPLC system with UV
Column, XBridge® C18, 150 × 4.6 (i.d.) mm, 5 µm particle size, or equivalent.
Electronic integrator or data system
Ultrasonic bath
Pyroxasulfone standard of known purity
Acetonitrile, HPLC grade
Water HPLC grade
Mobile phase Acetonitrile – water, 45+55 (v/v)
Calibration solutions. Weigh in duplicate (to the nearest 0.1 mg) into a volumetric

flask (25 ml) about 25 mg of Pyroxasulfone standard (s mg). Add acetonitrile (about 20 ml) into the flask, place the flask in an ultrasonic bath for 2 min. Allow to cool to ambient temperature. Dilute to volume with acetonitrile. Mix thoroughly. (Solutions C_A and C_B).

3.3 HPLC condition

Column temperature	35°C
Detector wavelength	225 nm
Injection volume	10 µl
Flow rate	1.0 ml/min
Run time	20 min
Retention time	Pyroxasulfone: about 8.7 min

3.4 Procedure

(i) Preparation of Pyroxasulfone sample. Prepare sample solutions in duplicate for each sample. Weigh in duplicate (to the nearest 0.1 mg) into a volumetric flask (25 ml) sufficient sample to contain approximately 25 mg of Pyroxasulfone (w mg). Add acetonitrile (about 20 ml) into the flask, place the flask in an ultrasonic bath for 2 min. Allow to cool to ambient temperature. Dilute to volume with acetonitrile. Mix thoroughly. (Solutions S_A and S_B).

(ii) Determination of Pyroxasulfone. Inject in duplicate 10µl portions of each sample solution bracketing them by injections of the calibration solutions as follows: C_{A1}, S_{A1}, S_{A2}, C_{B1}, S_{B1}, S_{B2}, C_{A2}, and so on. Measure the relevant peak areas.

(iii) Calculation. Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating the pyroxasulfone contents of the bracketed sample injections. The pyroxasulfone content is the mean value of two sample solutions.

$$f_i = \frac{s \times P}{H_S}$$

$$\text{Pyroxasulfone content} = \frac{s \times Hw}{w} \quad \text{g/kg}$$

where:

fi = individual response factor

f = mean response factor

Hs = peak area of pyroxasulfone in the calibration solution

Hw =peak area of pyroxasulfone in the sample solution

s = mass of pyroxasulfone standard taken (mg)

w = mass of sample taken (mg)

P = purity of the of pyroxasulfone standard (g/kg)

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.

4. Deviations and remarks

Lab	Lab 1	Lab 2	Lab 3
HPLC Model	Hitachi Primaide 1430	Agilent 1260	Agilent 1260
Column Information	Waters X-bridge C18, 150mm×4.6mm×5μm	Waters X-bridge C18, 150mm×4.6mm×5μm	SHIMADZU C18, 150mm×4.6mm×5μm
Column Temperature (ºC)	35	35	35
Detector Wavelength (nm)	225	225	225
Flow rate, ml/min	1.0	1.0	1.0
Injection Volume (μL)	10	10	10
Mobile phase Acetonitrile-water (v/v)	45+55	45+55	45+55
Retention Time, min	8.7	8.7	15.2

Note: Lab 3 used a different column of SHIMADZU C18, obtained different retention time.

5. Statistical evaluation

Table 1 Results of the analysis of AI content in the TC-1

	DAY -1		DAY -2		Average Yi (g/kg)	Yi^2	Standard Deviation，Si	Si^2
LAB 1	986.06	983.96	983.76	982.41	984.0	968349.18	1.5049	2.26
LAB 2	981.79	977.72	982.75	978.89	980.3	960961.30	2.3718	5.63
LAB 3	987.63	981.90	984.31	983.09	984.2	968713.92	2.4707	6.10

Table 2 Results of the analysis of Al content in the TC-2

	DAY -1		DAY -2		Average Yi (g/kg)	Yi^2	Standard Deviation, Si	Si^2
LAB 1	985.80	982.21	980.49	981.89	982.6	965497.85	2.2619	5.12
LAB 2	983.32	979.79	981.20	977.87	980.5	961466.41	2.2997	5.29
LAB 3	985.22	980.13	983.50	985.80	983.7	967592.08	2.5482	6.49

Table 3 Results of the analysis of Al content in the SC-1

	DAY -1		DAY -2		Average Yi (g/kg)	Yi^2	Standard Deviation，Si	Si^2
LAB 1	416.06	417.56	419.04	417.85	417.6	174412.73	1.2256	1.50
LAB 2	418.45	416.61	415.72	418.74	417.4	174206.06	1.4541	2.11
LAB 3	425.80	419.76	419.33	426.43	422.8	178784.66	3.8084	14.50

Table 4 Results of the analysis of Al content in the SC-2

	DAY -1		DAY -2		Average Yi (g/kg)	Yi^2	Standard Deviation，Si	Si^2
LAB 1	411.77	415.50	415.28	414.07	414.2	171524.36	1.7098	2.92
LAB 2	417.34	413.66	423.18	421.52	418.9	175497.31	4.2862	18.37
LAB 3	420.16	424.34	421.50	420.08	421.5	177679.50	1.9894	3.96

Table 5 Results of the analysis of Al content in the SC-3

	DAY -1		DAY -2		Average Yi (g/kg)	Yi^2	Standard Deviation, Si	Si^2
LAB 1	416.40	410.63	419.00	418.76	416.2	173220.36	3.8927	15.15
LAB 2	412.79	412.91	417.73	419.04	415.6	172737.91	3.2404	10.50
LAB 3	419.18	423.05	421.96	420.72	421.2	177430.54	1.6665	2.78

Table 6 Results of the analysis of Al content in the WG-1

	DAY -1		DAY -2		Average Yi (g/kg)	Yi^2	Standard Deviation，Si	Si^2
LAB 1	850.18	849.89	847.91	849.38	849.3	721378.44	1.0091	1.02
LAB 2	854.21	848.60	861.93	872.13	859.2	738255.14	10.1963	103.96
LAB 3	865.17	864.52	862.93	866.91	864.9	748025.64	1.6453	2.71

Table 7 Results of the analysis of Al content in the WG-2

	DAY -1		DAY -2		Average Yi (g/kg)	Yi^2	Standard Deviation, Si	Si^2
LAB 1	850.57	851.63	853.31	853.16	852.2	726189.45	1.3078	1.71

LAB 2	853.47	857.31	865.53	866.23	860.6	740693.50	6.2626	39.22
LAB 3	859.12	862.91	862.97	861.47	861.6	742389.38	1.8033	3.25

Table 8 Results of the analysis of Al content in the WG-3

	DAY -1		DAY -2		Average Yi (g/kg)	Yi^2	Standard Deviation，Si	Si^2
LAB 1	851.35	851.19	852.61	853.88	852.3	726342.85	1.2543	1.57
LAB 2	857.37	853.01	866.44	866.66	860.9	741097.16	6.7965	46.19
LAB 3	866.64	863.65	868.02	865.00	865.8	749657.68	1.9054	3.63

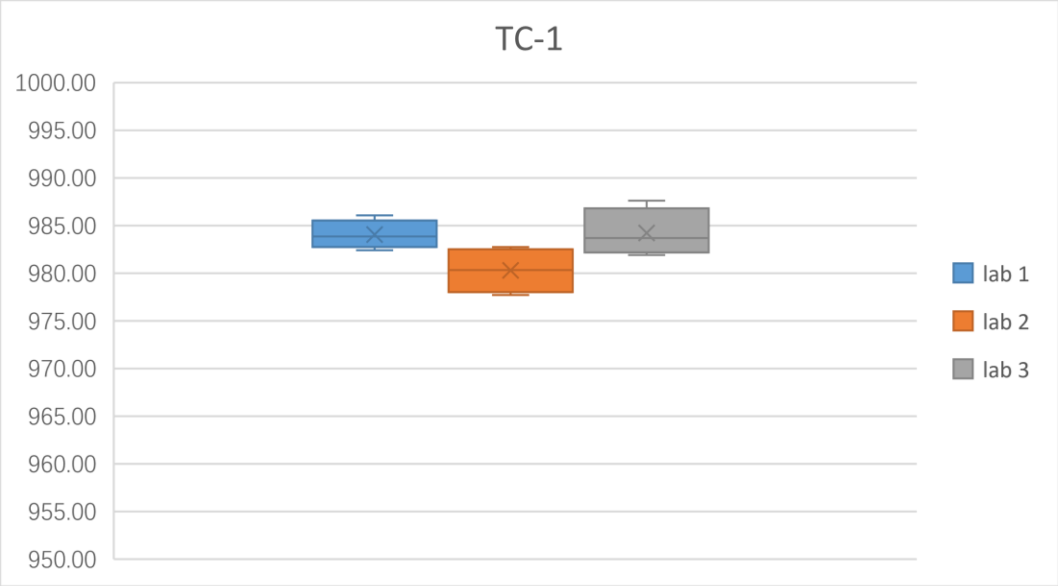

Figure 1 Graphical presentation of TC-1 data

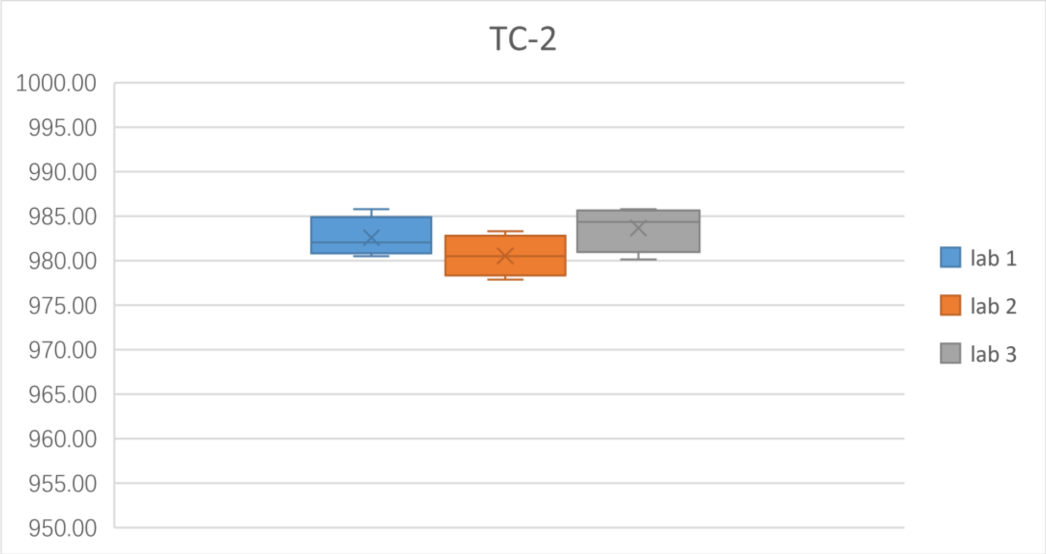

Figure 2 Graphical presentation of TC-2 data

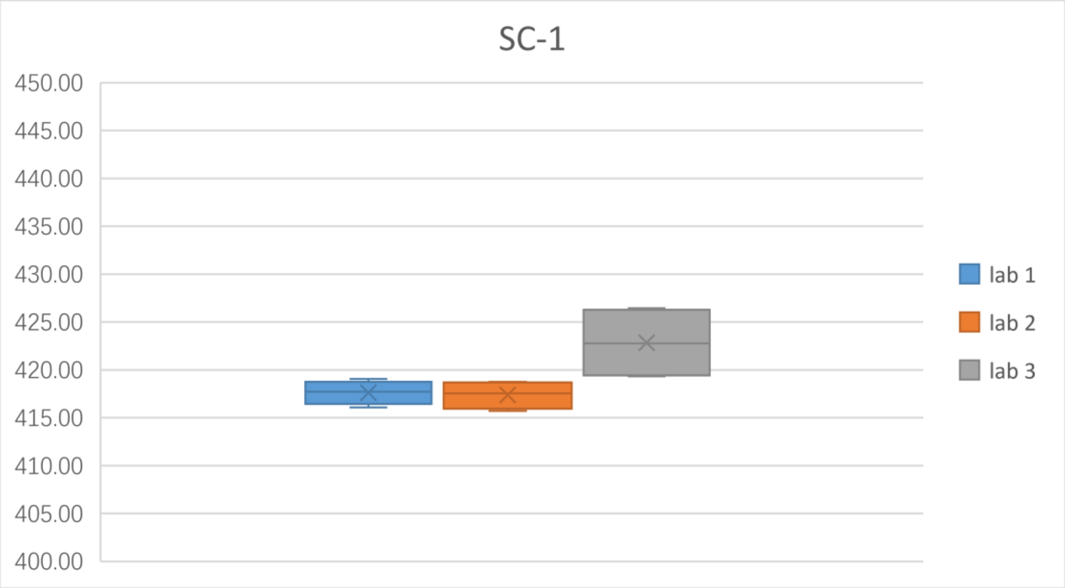

Figure 3 Graphical presentation of SC-1 data

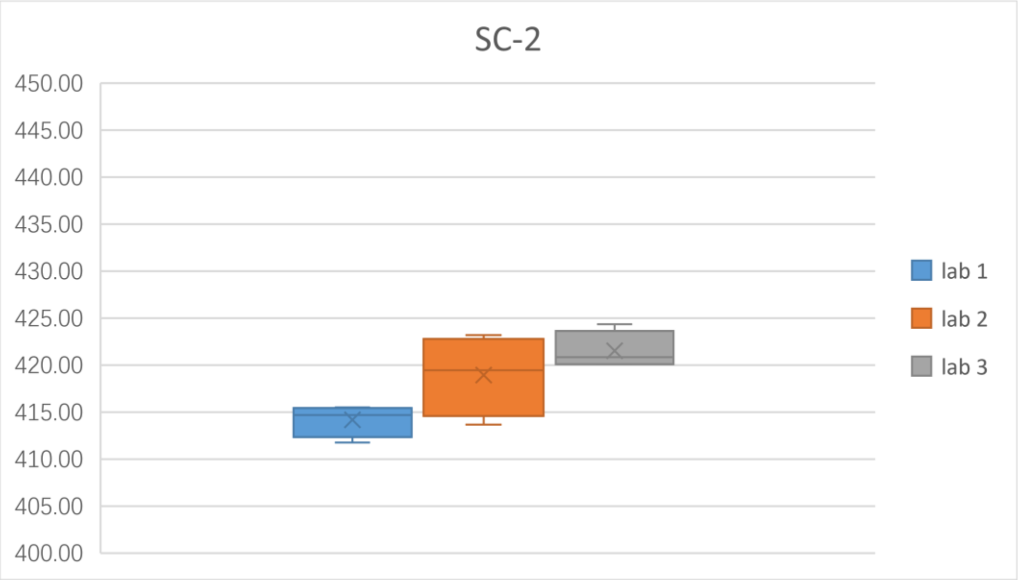

Figure 4 Graphical presentation of SC-2 data

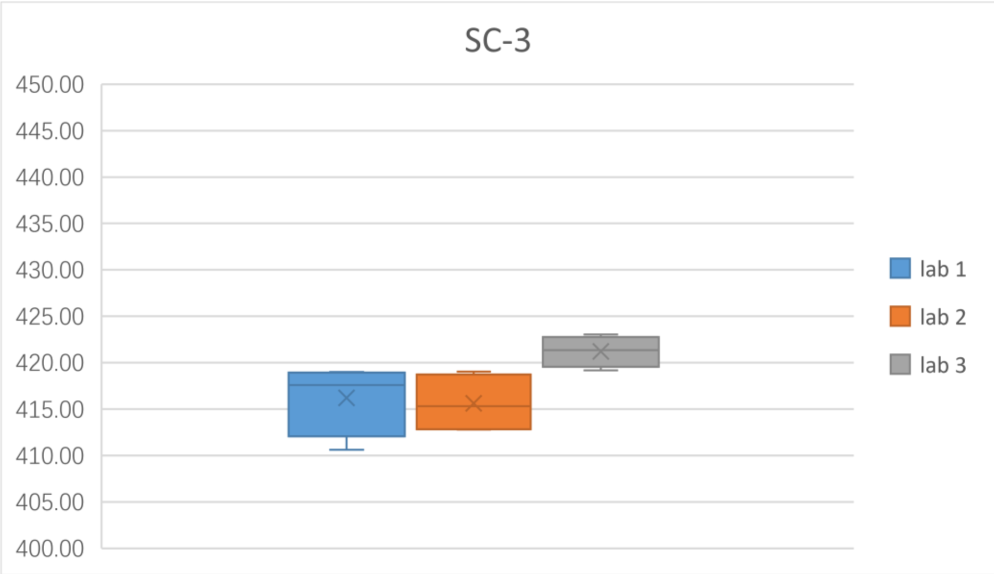

Figure 5 Graphical presentation of SC-3 data

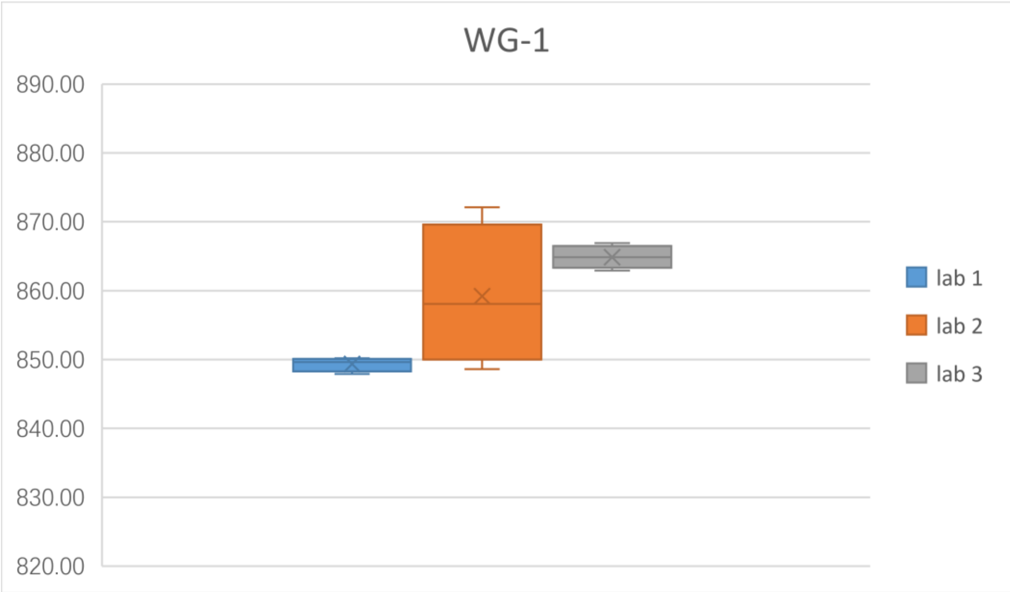

Figure 6 Graphical presentation of WG-1 data

Figure 7 Graphical presentation of WG-2 data

Figure 8 Graphical presentation of WG-3 data

Table 9 Statistics of the results of TC-1

S_1=sum Yi	2948.566339		
S_2=sum Yi^2	2898024.403		
S_3=sum Si^2	13.99467221		
No. Lab P	3		
No. Determination n	4		
Average $Y=S_1/P$	982.8554463		
$S_r^2=S_3/P$	4.664890737	**Standard Deviation of Repeatability, S_r**	2.159835813
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n$	3.79260851	**S_L**	1.947462069
$S_R^2=S_r^2+S_L^2$	8.457499248	**Standard Deviation Reproducibility, S_R**	2.908177995
Repeatability, r=2.8*Sr	6.047540275		
Reproducibility, $R=2.8*S_R$	8.142898385		
Relative Standard Deviation of Repeatability, $RSD_r=S_r*100/Y$	0.219751116		
Relative Standard Deviation of Reproducibility, $RSD_R=S_R*100/Y$	0.295890714		
Horwitz RSD_R (Hor)=2^[1-0.5*log(Y/1000)]	2.00521256		

HorRat	0.147560772		

Table 10 Statistics of the results of TC-2

S_1=sum Yi	2946.80402		
S_2=sum Yi^2	2894556.337		
S_3=sum Si^2	16.89808505		
No. Lab P	3		
No. Determination n	4		
Average $Y=S_1/P$	982.2680067		
$S_r^2=S_3/P$	5.632695017	**Standard Deviation of Repeatability, S_r**	2.373329943
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n$	1.104745041	**S_L**	1.051068523
$S_R^2=S_r^2+S_L^2$	6.737440058	**Standard Deviation Reproducibility, S_R**	2.595657924
Repeatability, r=2.8*Sr	6.64532384		
Reproducibility, R=2.8*SR	7.267842187		
Relative Standard Deviation of Repeatability, RSDr=Sr*100/Y	0.241617352		
Relative Standard Deviation of Reproducibility, RSDR=SR*100/Y	0.264251498		
Horwitz RSDR (Hor)=2^[1-0.5*log(Y/1000)]	2.005393012		
HorRat	0.131770429		

Table 11 Statistics of the results of SC-1

S_1=sum Yi	1257.836854		
S_2=sum Yi^2	527403.4555		
S_3=sum Si^2	18.11996568		
No. Lab P	3		
No. Determination n	4		
Average $Y=S_1/P$	419.2789512		
$S_r^2=S_3/P$	6.039988561	**Standard Deviation of Repeatability, S_r**	2.457638818
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n$	7.959335023	**S_L**	2.821229346

$S_R^2=S_r^2+S_L^2$	13.99932358	**Standard Deviation Reproducibility, S_R**	3.741566996
Repeatability, r=2.8*Sr	6.881388691		
Reproducibility, R=2.8*SR	10.47638759		
Relative Standard Deviation of Repeatability, RSDr=Sr*100/Y	0.586158406		
Relative Standard Deviation of Reproducibility, RSDR=SR*100/Y	0.89238131		
Horwitz RSDR (Hor)=2^[1-0.5*log(Y/1000)]	2.279549077		
HorRat	0.391472734		

Table 12 Statistics of the results of SC-2

S_1=sum Yi	1254.599443		
S_2=sum Yi^2	524701.1656		
S_3=sum Si^2	25.2525397		
No. Lab P	3		
No. Determination n	4		
Average $Y=S_1/P$	418.1998143		
$S_r^2=S_3/P$	8.417513234	**Standard Deviation of Repeatability, S_r**	2.901295096
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n$	11.8514314	**S_L**	3.442590798
$S_R^2=S_r^2+S_L^2$	20.26894463	**Standard Deviation Reproducibility, S_R**	4.502104467
Repeatability, r=2.8*Sr	8.123626269		
Reproducibility, R=2.8*SR	12.60589251		
Relative Standard Deviation of Repeatability, RSDr=Sr*100/Y	0.693758102		
Relative Standard Deviation of Reproducibility, RSDR=SR*100/Y	1.076543871		
Horwitz RSDR (Hor)=2^[1-0.5*log(Y/1000)]	2.280433472		

HorRat	0.472078613

Table 13 Statistics of the results of SC-3

S_1=sum Yi	1253.040052		
S_2=sum Yi^2	523388.8098		
S_3=sum Si^2	28.43052398		
No. Lab P	3		
No. Determination n	4		
Average Y=S_1/P	417.6800173		
$S_r^2=S_3/P$	9.476841326	**Standard Deviation of Repeatability, S_r**	3.078447876
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]- S_r^2/n$	7.140342862	**S_L**	2.672141999
$S_R^2=S_r^2+S_L^2$	16.61718419	**Standard Deviation Reproducibility, S_R**	4.076418059
Repeatability, r=2.8*Sr	8.619654053		
Reproducibility, R=2.8*SR	11.41397056		
Relative Standard Deviation of Repeatability, RSDr=Sr*100/Y	0.737034991		
Relative Standard Deviation of Reproducibility, RSDR=SR*100/Y	0.975966742		
Horwitz RSDR (Hor)=2^[1-0.5*log(Y/1000)]	2.280860403		
HorRat	0.427894115		

Table 14 Statistics of the results of WG-1

S_1=sum Yi	2573.442502		
S_2=sum Yi^2	2207659.212		
S_3=sum Si^2	107.689997		
No. Lab P	3		
No. Determination n	4		
Average Y=S_1/P	857.8141674		
$S_r^2=S_3/P$	35.89666568	**Standard Deviation of Repeatability, S_r**	5.991382618
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]- S_r^2/n$	52.91320822	**S_L**	7.274146563

$S_R^2=S_r^2+S_L^2$	88.8098739	**Standard Deviation Reproducibility, S_R**	9.423899082
Repeatability, r=2.8*Sr	16.77587133		
Reproducibility, R=2.8*SR	26.38691743		
Relative Standard Deviation of Repeatability, RSDr=Sr*100/Y	0.698447618		
Relative Standard Deviation of Reproducibility, RSDR=SR*100/Y	1.098594479		
Horwitz RSDR (Hor)=2^[1-0.5*log(Y/1000)]	2.046705308		
HorRat	0.536762413		

Table 15 Statistics of the results of WG-2

S_1=sum Yi	2574.423228		
S_2=sum Yi^2	2209272.328		
S_3=sum Si^2	44.18225586		
No. Lab P	3		
No. Determination n	4		
Average $Y=S_1/P$	858.1410759		
$S_r^2=S_3/P$	14.72741862	**Standard Deviation of Repeatability, S_r**	3.837631903
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]- S_r^2/n$	23.32325399	**S_L**	4.829415491
$S_R^2=S_r^2+S_L^2$	38.05067261	**Standard Deviation Reproducibility, S_R**	6.168522725
Repeatability, r=2.8*Sr	10.74536933		
Reproducibility, R=2.8*SR	17.27186363		
Relative Standard Deviation of Repeatability, RSDr=Sr*100/Y	0.447202915		
Relative Standard Deviation of Reproducibility, RSDR=SR*100/Y	0.718823851		
Horwitz RSDR (Hor)=2^[1-0.5*log(Y/1000)]	2.046587933		

HorRat	0.351230377

Table 16 Statistics of the results of WG-3

S_1=sum Yi	2578.955246		
S_2=sum Yi^2	2217097.688		
S_3=sum Si^2	51.39673318		
No. Lab P	3		
No. Determination n	4		
Average $Y=S_1/P$	859.6517485		
$S_r^2=S_3/P$	17.13224439	**Standard Deviation of Repeatability, S_r**	4.139111546
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n$	42.86793256	**S_L**	6.547360732
$S_R^2=S_r^2+S_L^2$	60.00017695	**Standard Deviation Reproducibility, S_R**	7.745978115
Repeatability, r=2.8*Sr	11.58951233		
Reproducibility, R=2.8*SR	21.68873872		
Relative Standard Deviation of Repeatability, RSDr=Sr*100/Y	0.481487015		
Relative Standard Deviation of Reproducibility, RSDR=SR*100/Y	0.901060008		
Horwitz RSDR (Hor)=2^[1-0.5*log(Y/1000)]	2.046046204		
HorRat	0.440390841		

A Grubbs test were run on all determinations, and no outlier was found.

6. Conclusion

No outlier was found in the data set.

All RSD_R values were lower than the criteria calculated using the Horwitz equation. All HorRat values were smaller than 1.0, while 2 values fell below 0.3.

From the results shown above, the method can be considered applicable for the determination of pyroxasulfone contents in TC, SC and WG. We propose that a full scale collaborative trial might be conducted.

7. Figures

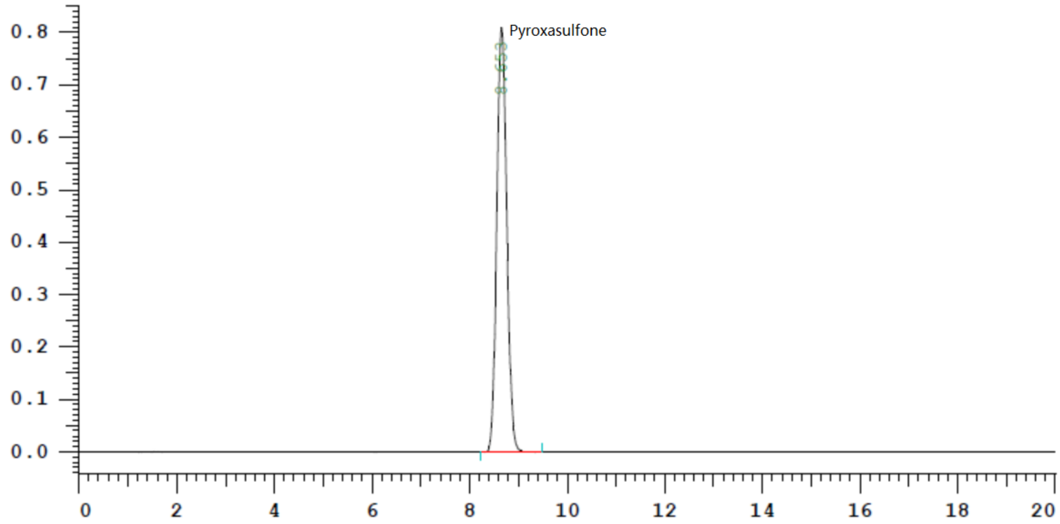

Fig.1 HPLC chromatogram of Pyroxasulfone standard

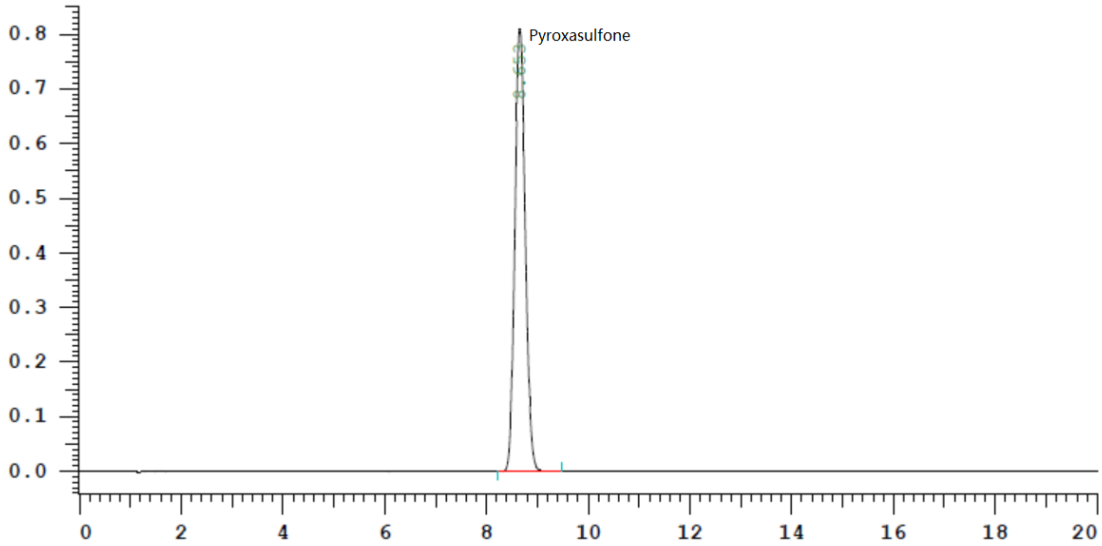

Fig.2 HPLC chromatogram of Pyroxasulfone TC

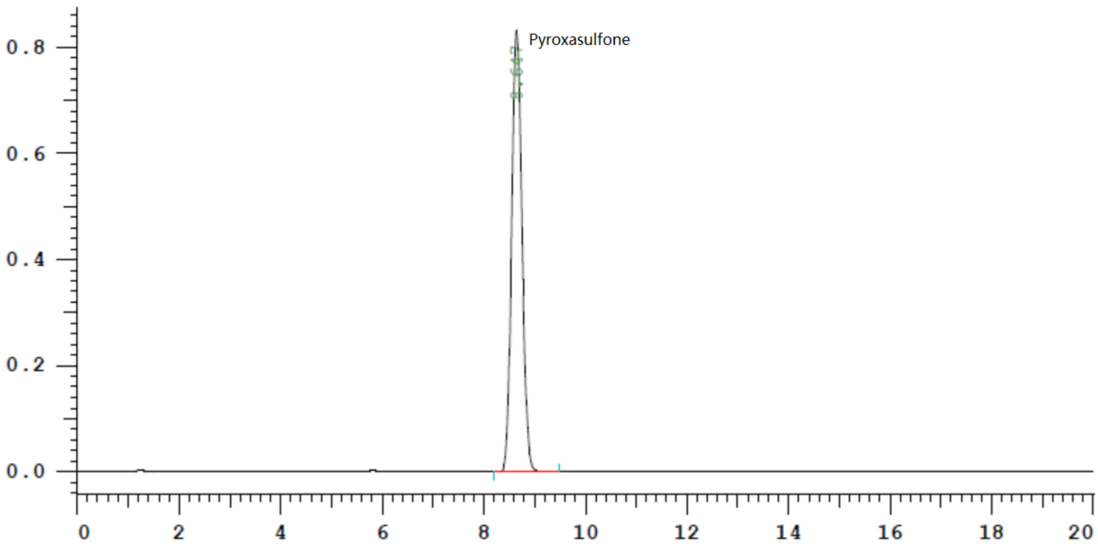

Fig.3 HPLC chromatogram of Pyroxasulfone SC

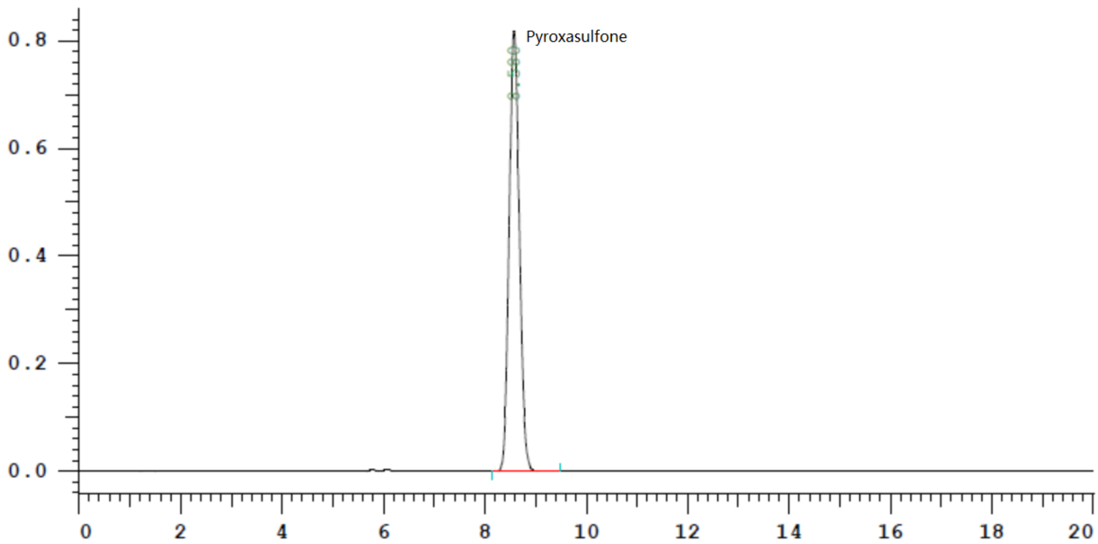

Fig.4 HPLC chromatogram of Pyroxasulfone WG