

# Method Extension of CIPAC 578 in Flumioxazin WG

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Test Guideline: CIPAC No. 3807, CIPAC 578



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# STATEMENT

Test substance: Flumioxazin WG Study Title: Method Extension of CIPAC 578 in Flumioxazin WG Study No.: BNS20220053

This study was designed and performed by QC Center of Shandong Binnong Technology Co., Ltd. To the best of our knowledge, there were no deviations from the study protocol affecting the quality or integrity of the study.

This report accurately reflects the raw data and accurately describes the methods and standard operation procedures used in this study.

Chief Analyzer: Sun Jing

Signature:

Sun Jong

Date: 2022-06-09

Shandong Binnong Technology Co., Ltd.



### 1. Summary

For Flumioxazin WG, there is no CIPAC method available. To determine whether the method (CIPAC 578) can be applied for the new formulation of Flumioxazin WG, a specificity test was performed according to the procedure of the extension of the scope of methods. The results are summarized in the following Table 1 and Table 2.

Solution tested	Conclusion
Acetonitrile (Solvent)	
Blank formulation	No interferences were detected at the
Test item solution	retention time of the active ingredient.
Reference item solution	

Table 1 The specificity test results

Table 2 Peak purity analysis results

Sample solution name	Purity threshold	Purity factor
Test item solution	990.000	999.959
Reference item solution	990.000	999.951

According to the specificity test and peak purity analysis results, the method (CIPAC 578) was considered specific applied for the Flumioxazin WG.

And the concentration of flumioxazin analyte is inside the acceptability range covered by the sample, so the method (CIPAC 578) can be applied for Flumioxazin WG.



### 2. Introduction

The objective of the study is to evaluate whether the method (CIPAC 578) is suitable for the determination of flumioxazin in WG formulation.

### 3. Material

#### 3.1 Specification of test item

The related information and analytical identification provided by the sponsor was the responsibility of the sponsor. The details of the test items are given as follow:

Product name	Flumioxazin WG
Molecular formula of Al	C <sub>19</sub> H <sub>15</sub> FN <sub>2</sub> O <sub>4</sub>
Molecular weight of Al	354.3 g/mol
Structure formula of AI	
Chemical name (IUPAC) of	N-(7-fluoro-3,4-dihydro-3-oxo-4-prop-2-ynyl-2H-1,4-
AI	benzoxazin-6-yl) cyclohex-1-ene-1,2-dicarboxamide
CAS No. of Al	103361-09-7
Physical state	Light brown granule
Batch No.	202205098
Active ingredient, % (w/w)	52.0
Moisture, % (w/w)	0.62
Manufacture date	May 25, 2022
Retest date	May 25, 2024
Storage condition	Store in original container, in a well-ventilated, cool, dry,
Storage condition	secure area.
Manufacturer	Shandong Binnong Technology Co., Ltd.
Address of manufacturer	No.518, Yongxin Road, Binbei Town, Binzhou, Shandong,
	China

Certificate of analysis for test item is presented in Appendix B.

#### 3.2 Specification of reference item

Product Name	Flumioxazin
CAS Number	103361-09-7
Lot No.	G140220
Purity	99.47%
Certified date	May 26, 2017
Expiry Date	February 16, 2023
Storage Condition	<b>20±4</b> ℃
Manufacturer	Dr. Ehrenstorfer

Certificate of analysis for reference item is presented in **Appendix C**.

### 3.3 Storage conditions

The test item was stored at 20±10  $\,\,{}^\circ\!{}^\circ\!{}^\circ$  in Test Substance Control Office of BNS QC Center.

The reference item was stored at  $20\pm4$  °C in refrigerator in BNS QC Center. They are treated according to relevant SOP for reception, storage and distribution.

# 4. Experimental Procedure

# 4.1 Reagents

Flumioxazin reference item, purity 99.47%, Lot No.: G140220, supplied by Dr. Ehrenstorfer. Acetonitrile, HPLC grade, Lot No.: 10972907839, supplied by Merck KGaA, Germany.

Ultrapure water, resistivity:  $\geq 10 \text{ M}\Omega$  cm, prepared by ultrapure water machine.

### 4.2 Equipment

Agilent 1260 Infinity II LC system, with DAD detector, supplied by Agilent Technologies Pte., Ltd, USA;

Electronic balance, type: XSE205DU, supplied by Mettler-Toledo Instrument Co., Ltd, Switzerland.

Ultrapure water machine, type: UPR-II-20L, supplied by ULUPURE Ultrapure Technology Co., Ltd, China.

Ultrasonic cleaner, type: AS-3120, supplied by Tianjin Automatic Science Instrument Co., Ltd, China.

Volumetric flask, 100 ml, supplied by Tianjin Glass Instrument Manufacturing Co., Ltd, China. Nylon syringe filters, 0.22 µm, supplied by Tianjin Heshi Science and Technology Development Ltd., China.

Disposable syringe, 5 mL, supplied by Henan Shu Guang Jian Shi Medical Apparatus and Instruments Co., Ltd., China.

Detector:	DAD	DAD				
Wavelength:	288 nm	288 nm				
Column:	Phenomenex (	Phenomenex Gemini (250mm x 4.6mm x 5µm)				
Column temperature:	<b>40</b> °C					
Injection volume:	10.0 µl	10.0 µl				
Mobile phase:	Solvent A: Ultra	Solvent A: Ultrapure Water				
	Solvent B: Ace	Solvent B: Acetonitrile				
Stop time:	15 min	15 min				
Post time	0 min	0 min				
Time (min)	Flow (mL/min)	A (%)	B (%)			
0	1.000	1.000 50 50				

### 4.3 Analytical condition

# 4.4 Specificity test

The specificity of the method was evaluated by injecting Acetonitrile (solvent used for solution preparation), blank formulation, test item solution and reference item solutions to check for any interfering peak at the retention time of the peak of interest.

**Blank formulation solution preparation:** 47.74mg of blank formulation was weighed into a 100 mL volumetric flask. About 80 mL Acetonitrile was added into flask and the flask was shaken to dissolve. The flask was placed in ultrasonic bath for 10 min, then cool to ambient temperature. The solution was made up to the mark using Acetonitrile and mixed well.



**Test item solution preparation:** 96.66mg of test item was weighed into a 100 mL volumetric flask. About 80 mL Acetonitrile was added into flask and the flask was shaken to dissolve. The flask was placed in ultrasonic bath for 10 min, then cool to ambient temperature. The solution was made up to the mark using Acetonitrile and mixed well.

**Reference item solution preparation**: 50.63mg of reference item was weighed into a 100 mL volumetric flask. About 80 mL Acetonitrile was added into flask and the flask was shaken to dissolve. The flask was placed in ultrasonic bath for 10 min, then cool to ambient temperature. The solution was made up to the mark using Acetonitrile and mixed well.

All the solutions were filtered through a 0.22  $\mu$ m filter prior to analysis.

And inject at least 2 portions of Reference item solutions for system equilibration before performing the specificity test.

#### 4.5 Linearity check

Check the linearity of the detector response by injecting 10  $\mu$ l of solutions with flumioxazin concentrations 0.5, 1 and 2 times that of the Reference item solution to determine whether the concentration of the flumioxazin is inside the acceptability range covered by the samples applied in the method (CIPAC 578).

**Lin-1**: 25.09mg of reference item was weighed into a 100 mL volumetric flask. About 80 mL Acetonitrile was added into flask and the flask was shaken to dissolve. The flask was placed in ultrasonic bath for 10 min, then cool to ambient temperature. The solution was made up to the mark using Acetonitrile and mixed well.

**Lin-2**: 50.03mg of reference item was weighed into a 100 mL volumetric flask. About 80 mL Acetonitrile was added into flask and the flask was shaken to dissolve. The flask was placed in ultrasonic bath for 10 min, then cool to ambient temperature. The solution was made up to the mark using Acetonitrile and mixed well.

**Lin-3**: 100.19mg of reference item was weighed into a 100 mL volumetric flask. About 80 mL Acetonitrile was added into flask and the flask was shaken to dissolve. The flask was placed in ultrasonic bath for 10 min, then cool to ambient temperature. The solution was made up to the mark using Acetonitrile and mixed well.

All the solutions were filtered through a 0.22  $\mu$ m filter prior to analysis.

### 5. Result and Conclusion

### 5.1 Specificity test results

Solution tested	Conclusion
Acetonitrile (Solvent)	
Blank formulation	No interferences were detected at the
Test item solution	retention time of the active ingredient.
Reference item solution	

#### Table 1 Specificity of Flumioxazin WG

The specificity relevant chromatograms, are listed in Appendix A, figure 1-4.

Peak purity analysis was performed using a DAD detector to detect the presence of any other substance co-eluting with the analyte peak. The peak purity factor of the sample solutions was



greater than the peak purity threshold, indicating that there was no interference. That shows a good peak purity.

Sample solution name	Purity threshold	Purity factor
Test item solution	990.000	999.959
Reference item solution	990.00	999.951

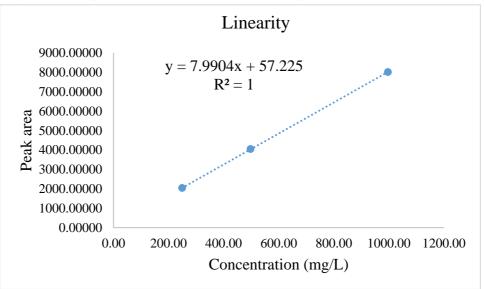
The peak purity analysis related chromatograms, are listed in Appendix A, figure 5-6. From the above specificity test, the method (CIPAC 578) used for flumioxazin WG formulation is specific.

### 5.2 Linearity check results

From the CIPAC guideline "EXTENSION OF THE SCOPE OF METHODS", the acceptability range is from 200 % to 50 % of the concentration of an analyte in a sample studied.

The 3 concentrations (0.5, 1, 2 times) of the Flumioxazin Reference item solution, were injected with duplicate for each. The line was generated with slope, intercept and correlation coefficient data.

The linearity chromatograms, are listed in Appendix A, figure 7-12.



The correlation coefficient (r) is > 0.99. over the range (200% ~ 50% of the nominal). The test item solution injected for specificity test with peak area 4082.98804, is in the range of the linearity concentration.

Sample solution name	Lin-1	Lin-2	Lin-3		
Average peak area for 2 injections	2041.79016 4047.97559 8015.5710				
Peak area of Test item solution for	4082.98804				
specificity					
Peak area of Reference item solution	4110 91250				
for specificity	4110.81250				

#### Table 3 Peak area of Flumioxazin reference and test item solutions

From the table 3, the peak area of Test item solution for specificity is nearly with the peak area



of Lin-2, which indicate the concentration of analyte in test item solution for specificity is nearly with the concentration of analyte in Lin-2(100% of nominal analyte concentration).

The other hand, the sample content is 52.0% (indicated in sponsor COA), which contains the analyte flumioxazin concentration as follows: 96.66 mg/100 ml \*1000\* 52.0% = 502.6 mg/I, and in the method (CIPAC 578), the nominal concentration of flumioxazin used as (50 mg/100 ml = 500 mg/I), so, the acceptability range is 502.6/500\*100% = 100.52%, which is also indicate the concentration of analyte in test item solution for specificity is in the acceptable range between 200% and 50%.

### 6. Amendment and deviation

The test is focus on the extension of the flumioxazin CIPAC method to flumioxazin WG formulation. The specificity test was conducted completely follow the CIPAC method without any change.

### 7. Reference

- (1) CIPAC No.3807 Guidelines on method validation to be performed in support of analytical methods for agrochemical formulations.
- (2) CIPAC Method (578/TC/M/3, 578/WP/M/3).
- (3) CIPAC guideline 'Extension of the scope of methods'.

### 8. Appendix

APPENDIX A: COPY OF CHROMATOGRAM APPENDIX B: CERTIFICATE OF ANALYSIS FOR TEST ITEM APPENDIX C: CERTIFICATE OF ANALYSIS FOR REFERENCE ITEM



#### APPENDIX A: COPY OF CHROMATOGRAM

Data File D:\HPLC\DA...LUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\016-P2-B4-Acetonitrile.D Sample Name: Acetonitrile

Acq. Operator :	SYSTEM	Seq. Line : 16
Acq. Instrument :	LC 1260 DAD	Location : P2-B-04
1 P	6/7/2022 9:17:02 PM	Inj: 1
	. No • 20. Contractor new residences 5 sector 15 Hope	Inj Volume : 10.000 μl
Acq. Method :	D:\HPLC\Data\Flumioxazir	n\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M
	11/17/2021 8:30:46 AM by	
	D:\HPLC\METHOD\FLUMIOXAZ	
	6/8/2022 4:13:11 PM by S	
Ų.		VFLUMIOXAZIN 2022-06-07 17-17-09\016-P2-B4-Acetonitrile.D)
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Sorted By	: Signal	
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Dilution	: 1.0000	
ose Multipiien &	Dilution Factor with ISTD	75
No peaks found		
	*** End of Repor	

LC 1260 DAD 6/8/2022 4:19:36 PM SYSTEM

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#### FIGURE 1: CHROMATOGRAM OF ACETONITRILE, SOLVENT)



Data File D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\017-P2-B5-Blank-WDG.D Sample Name: Blank-WDG

			***	End of Rep					
peaks									
orted By Itiplie Iution Ge Multi	r	Dilu	: 1 : 1	Signal 1.0000 1.0000 Cor with IS	STDs				
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100 -									
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-									
300 -									
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			/2022 9:3			Volume : 10	.000 µl		
			1260 DAD	2.50 DM	L	ocation : 『 Inj : 〔	P2-B-05		
	rator	: SYS	I EM		Se	q. Line : 17	/		

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#### FIGURE 2: CHROMATOGRAM OF BLANK FORMULATION



Data File D:\HPLC\DA...MIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\018-P2-B6-Reference item.D Sample Name: Reference item

Acq. Operator : SYSTEM		Seq. Line :	18		
Acq. Instrument : LC 1260	DAD	Location :	P2-B-06		
Injection Date : 6/7/202	<pre>iection Date : 6/7/2022 9:48:54 PM Inj : 1 Inj Volume : 10.000 µl inj Volume : 10.0000 µl inj Volume : 10.000</pre>				
		Inj Volume :	10.000 µl		
<pre>cq. Operator : SYSTEM Seq. Line : 18 .cq. Instrument : LC 1260 DAD Location : P2-B-06 .njection Date : 6/7/2022 9:48:54 PM Inj : 1 Inj Volume : 10.000 µl .cq. Method : D:\HPLC\Data\Flumioxazin\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M ast changed : 11/17/2021 8:30:46 AM by SYSTEM malysis Method : D:\HPLC\DMETHOD\FLUMIOXAZIN\flumioxazin-1.M ast changed : 6/8/2022 4:13:11 PM by SYSTEM DAD1 A, SIg=288.4 Ref=off (D:\HPLC\DAXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\018-P2-B6-Reference item.D) mAU 400 - 0 - 0 - 0 - 0 - 0 - 0 - 0 - 0 - 0</pre>					
Last changed : 11/17/2	Pection Date : 6/7/2022 9:48:54 PM Inj: 1 Inj Volume : 10.000 µl I. Method : D:\HPLC\Data\Flumioxazin\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M it changed : 11/17/2021 8:30:46 AM by SYSTEM DADI A, Sg=288.4 Ref=off (D\HPLC\DAXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\018-P2-B6-Reference item.D) mAU 400- 300- 100-				
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cq. Operator : SYSTEM Seq. Line : 18 cq. Instrument : LC 1260 DAD Location : P2-B-06 njection Date : 6/7/2022 9:48:54 PM Inj : 1 Inj Volume : 10.000 µl cq. Method : D:\HPLC\Data\Flumioxazin\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M ast changed : 11/17/2021 8:39:46 AM by SYSTEM DAD1 A. SIg=288.4 Ref=off (D:\HPLC\DAL_XAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\018-P2-B8-Reference item.D) MAU 400 					
-	q. Operator       : SYSTEM       Seq. Line : 18         q. Instrument : LC 1260 DAD       Location : P2-B-06         jection Date       : 6/7/2022 9:48:54 PM       Inj : 1         Inj Volume : 10.000 µl       Inj Volume : 10.000 µl         q. Method       : D:\HPLC\Data\Flumioxazin\Flumioxazin\2022-06-07 17-17-09\flumioxazin.M         st changed       : 11/17/2021 8:30:46 AM by SYSTEM         alysis Method       : D:\HPLC\METHOD\FLUMIOXAZIN\flumioxazin-1.M         st changed       : 6/8/2022 4:13:11 PM by SYSTEM         DAD1 A, Sig=288.4 Ref=off (D\HPLC\DA_XAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\018-P2-B6-Reference item.D)         mAU				
200	q. Operator       : SYSTEM       Seq. Line : 18         q. Instrument : LC 1260 DAD       Location : P2-B-06         jection Date       : 6/7/2022 9:48:54 PM       Inj : 1         Inj Volume : 10.000 µl       Inj Volume : 10.000 µl         q. Method       : D:\HPLC\Data\Flumioxazin\Flumioxazin\2022-06-07 17-17-09\flumioxazin.M         st changed       : 11/17/2021 8:30:46 AM by SYSTEM         alysis Method       : D:\HPLC\METHOD\FLUMIOXAZIN\flumioxazin-1.M         st changed       : 6/8/2022 4:13:11 PM by SYSTEM         DAD1 A, Sig=288.4 Ref=off (D:\HPLC\DA_XAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\018-P2-B6-Reference item.D)         mAU				
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,					
	<del> </del>				
2	4 6	8	10	12 14	min
Sorted By :	Signal				
Multiplier :	1.0000				
Dilution :	1.0000				
Use Multiplier & Dilution	Factor with ISTDs				
Signal 1: DAD1 A, Sig=288	,4 Ref=off				
Peak RetTime Type Width	Area Height	Area			
	[mAU*s] [mAU]	%			
1 12.720 BB 0.2578	4110.81250 244.96487	100.0000			
Totals :	4110.81250 244.96487				
	*** End of Report ***				
	end of kepore				

LC 1260 DAD 6/8/2022 4:20:41 PM SYSTEM

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#### FIGURE 3: CHROMATOGRAM OF REFERENCE ITEM



Data File D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\019-P2-B7-Sample-WDG.D Sample Name: Sample-WDG

------Acq. Operator : SYSTEM Seq. Line : 19 Acq. Instrument : LC 1260 DAD Location : P2-B-07 Inj : 1 Injection Date : 6/7/2022 10:04:50 PM INJ . \_ Inj Volume : 10.000 μl Acq. Method : D:\HPLC\Data\Flumioxazin\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M Last changed : 11/17/2021 8:30:46 AM by SYSTEM Analysis Method : D:\HPLC\METHOD\FLUMIOXAZIN\flumioxazin-1.M Last changed : 6/8/2022 4:13:11 PM by SYSTEM DAD1 A, Sig=288,4 Ref=off (D:\HPLC\DA...UMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\019-P2-B7-Sample-WDG.D) mAU 400 300-12.714 200 -100 -0 10 12 14 min 2 8 \_\_\_\_\_\_ Area Percent Report Sorted By : Signal Multiplier : 1.0000 Dilution 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=288,4 Ref=off Peak RetTime Type Width Area Height Area [min] [mAU\*s] [mAU] % # [min] 1 12.714 BB 0.2576 4082.98804 243.51692 100.0000 Totals : 4082.98804 243.51692 \*\*\* End of Report \*\*\*

LC 1260 DAD 6/8/2022 4:21:15 PM SYSTEM

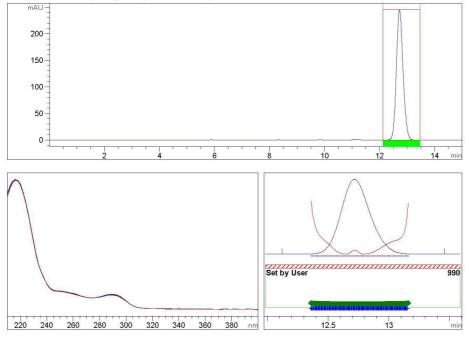
Page 1 of 1

#### FIGURE 4: CHROMATOGRAM OF TEST ITEM



Data File D:\HPLC\DA...MIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\018-P2-B6-Reference item.D Sample Name: Reference item

Purity results peak 1 at 12.720 min.



Signal DAD1 A, Sig=288,4 Ref=off (D:\HPLC\DA...XAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\018-P2-

-> The purity factor is within the threshold limit. <-

Purity factor : 999.951 (121 of 121 spectra are within the threshold limit.)Threshold : 990.000 (Set by user)Reference : Nearest baseline spectrum (stored) (0.000)Spectra : 121 (Selection automatic, All)Warning : Spectral absorbances > 1000 mAU (see help for more information)

\*\*\* End of Report \*\*\*

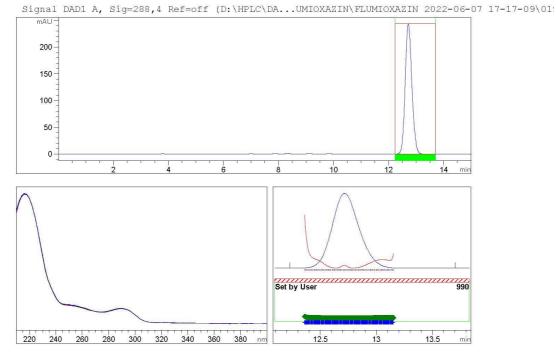
LC 1260 DAD 6/8/2022 10:39:40 AM SYSTEM

Page 1 of 1

### FIGURE 5: PEAK PURITY OF REFERENCE ITEM



Data File D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\019-P2-B7-Sample-WDG.D Sample Name: Sample-WDG



Purity results peak 1 at 12.714 min.

-> The purity factor is within the threshold limit. <-

Purity factor : 999.959 (120 of 120 spectra are within the threshold limit.)Threshold : 990.000 (Set by user)Reference : Nearest baseline spectrum (stored) (0.000)Spectra : 120 (Selection automatic, All)Warning : Spectral absorbances > 1000 mAU (see help for more information)

\*\*\* End of Report \*\*\*

LC 1260 DAD 6/8/2022 10:39:59 AM SYSTEM

Page 1 of 1

#### FIGURE 6: PEAK PURITY OF TEST ITEM



Data File D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\020-P2-B1-Lin-1.D Sample Name: Lin-1

------Acq. Operator : SYSTEM Seq. Line : 20 Location : P2-B-01 Inj : 1 Acq. Instrument : LC 1260 DAD Injection Date : 6/7/2022 10:20:46 PM Inj Volume : 10.000 μl : D:\HPLC\Data\Flumioxazin\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M Acq. Method Last changed : 11/17/2021 8:30:46 AM by SYSTEM Analysis Method : D:\HPLC\METHOD\FLUMIOXAZIN\flumioxazin 1.M : 6/8/2022 9:14:04 AM by SYSTEM Last changed DAD1 A, Sig=288,4 Ref=off (D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\020-P2-B1-Lin-1.D) mAU 600 500 -400 -300-200 -12.722 100 0 10 12 14 mir \_\_\_\_\_\_ Area Percent Report \_\_\_\_\_ Sorted By Signal : Calib. Data Modified : 6/8/2022 8:32:43 AM Multiplier : 1.0000 Dilution : 1.0000 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1 A, Sig=288,4 Ref=off Peak RetTime Type Width Area Area Name [min] [mAU\*s] % # [min] 1 12.722 BB 0.2577 2040.47607 100.0000 Flumioxazin Totals : 2040.47607 \*\*\* End of Report \*\*\*

LC 1260 DAD 6/8/2022 9:37:42 AM SYSTEM

Page 1 of 1

#### FIGURE 7: LINEARITY CHECK FOR LIN-1, injection 1



Data File D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\021-P2-B1-Lin-1.D Sample Name: Lin-1

======================================	: SYSTEM		
	: LC 1260 DAD	Location : P2-B-01	
	: 6/7/2022 10:36:42 PM	Inj: 2	
		Inj Volume : 10.000 µl	
Acq. Method	: D:\HPLC\Data\Flumioxazin	\Flumioxazin 2022-06-07 17-17-09\1	lumioxazin.M
Last changed	: 11/17/2021 8:30:46 AM by		
Analysis Method	: D:\HPLC\METHOD\FLUMIOXAZ	IN\flumioxazin 1.M	
Last changed	: 6/8/2022 9:14:04 AM by S		
	Sig=288,4 Ref=off (D:\HPLC\DATA\FLUMIO	XAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\021-P2-	B1-Lin-1.D)
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Multiplier	: 1.0000	.45 AM	
Dilution	: 1.0000		
	& Dilution Factor with ISTD:	5	
<b>F</b>			
Signal 1: DAD1	A, Sig=288,4 Ref=off		
Peak RetTime 1	Type Width Area A	rea Name	
# [min]	[min] [mAU*s]	%	
1 12.747 BE			
Totals :	2042 10425		
TOTALS .	2043.10425		
	*** 5.4.5 0		
	*** End of Repor		
			Page 1 of 1

# FIGURE 8: LINEARITY CHECK FOR LIN-1, injection 2



Data File D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\022-P2-B2-Lin-2.D Sample Name: Lin-2

Acq. Operator	: SYSTEM Seq. Line : 22
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-	<pre>q. Instrument : LC 1260 DAD Location : P2-B-02 jection Date : 6/7/2022 10:52:38 PM Inj : 1 Inj Volume : 10.000 μl q. Method : D:\HPLC\Data\Flumioxazin\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M st changed : 11/17/2021 8:30:46 AM by SYSTEM alysis Method : D:\HPLC\METHOD\FLUMIOXAZIN\flumioxazin 1.M st changed : 6/8/2022 9:14:04 AM by SYSTEM DAD1 A, Sig=288.4 Ref=off (D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\022-P2-B2-Lin-2.D)</pre>
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Multiplier	: 1.0000
Dilution	: 1.0000
Use Multiplie	r & Dilution Factor with ISTDs
Signal 1: DAD	L A, Sig=288,4 Ref=off
Peak RetTime	Type Width Area Area Name
# [min]	[min] [mAU*s] %
1 12.735	3B 0.2582 4043.69287 100.0000 Flumioxazin
Totals :	4043.69287
	*** End of Report ***
100 DAD 6/8/2	22 9:38:26 AM SYSTEM Page 1 of 1

# FIGURE 9: LINEARITY CHECK FOR LIN-2, injection 1



Data File D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\023-P2-B2-Lin-2.D Sample Name: Lin-2

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Acq. Operator	SYSTEM Seq. Line : 23	
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Injection Date		
Acq. Method		-1/-09\+1umloxazin.M
	MAL PARTNERS AND	
Last changed		
		09\023-P2-B2-Lin-2.D)
mAU		
-		
600		
-	Operator       : SYSTEM       Seq. Line : 23         Instrument       : LC 1260 DAD       Location : P2-B-02         tion Date       : 6/7/2022 11:08:34 PM       Inj : 2         Inj Volume       : 10.000 µl         Method       : D:\HPLC\Data\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M         changed       : 11/17/2021 8:30:46 AM by SYSTEM         sis Method :       D:\HPLC\METHOD\FLUMIOXAZIN\Flumioxazin 1.M         changed       : 6/8/2022 9:14:04 AM by SYSTEM         DAD1 A, Sig=288.4 Ref=off (D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\023-P2-B2-Lin-2.D)         AU	
500 -		
-	Instrument : LC 1260 DAD Location : P2-B-02 tion Date : 6/7/2022 11:08:34 PM Inj : 2 Inj Volume : 10.000 µl Method : D:\HPLC\Data\Flumioxazin\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M changed : 11/17/2021 8:30:46 AM by SYSTEM /// // // // // // // // // // // // //	
-	perator : SYSTEM Seq. Line : 23 hstrument : LC 1260 DAD Location : P2-B-02 ion Date : 6/7/2022 11:08:34 PM Inj : 2 Inj Volume : 10.000 µl ethod : D:\HPLC\Data\Flumioxazin\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M hanged : 11/17/2021 8:30:46 AM by SYSTEM is Method : D:\HPLC\METHOD\FLUMIOXAZIN\flumioxazin 1.M DAD1 A_SIG=288.4 Ref=off (D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\023-P2-B2-Lin-2.D) DAD1 A_SIG=288.4 Ref=off (D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\023-P2-B2-Lin-2.D)	
400 -		
300		15
300		2.74
-	ator : SYSTEM Seq. Line : 23 nument : LC 1260 DAD Location : P2-B-02 Date : 6/7/2022 11:08:34 PM Inj : 2 Inj Volume : 10.000 μl od : D:\HPLC\Data\Flumioxazin\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M ged : 11/17/2021 8:30:46 AM by SYSTEM Method : D:\HPLC\METHOD\FLUMIOXAZIN\flumioxazin 1.M ged : 6/8/2022 9:14:04 AM by SYSTEM AD1 A, Sig=288,4 Ref=off (D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\023-P2-B2-Lin-2.D)	7
200 -	serator : SYSTEM Seq. Line : 23 hstrument : LC 1260 DAD Location : P2-B-02 Lion Date : 6/7/2022 11:08:34 PM Inj : 2 Inj Volume : 10.000 µl ethod : D:\HPLC\Data\Flumioxazin\Flumioxazin 2022-06-07 17-17-09\flumioxazin.M hanged : 11/17/2021 8:30:46 AM by SYSTEM DAD1 A, Sig=288.4 Ref=off (D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 1.M DAD1 A, Sig=288.4 Ref=off (D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\023-P2-B2-Lin-2.D)	
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Multiplier	: 1.0000	
Dilution	: 1.0000	
	r & Dilution Factor with ISTDs	
Signal 1: DAD:	1 A, Sig=288,4 Ref=off	
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Peak RetTime # [min]	Type Width Area Area Name [min] [mAU*s] %	
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1 12.745		
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Totals :	4052.25830	
		=
	www.Fullet.F.Densel	
	*** End of Report ***	
	*** End of Report ***	
:60 DAD 6/8/20	*** End of Report *** 022 9:38:57 AM SYSTEM	Page 1 of 1

# FIGURE 10: LINEARITY CHECK FOR LIN-2, injection 2



Data File D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\024-P2-B3-Lin-3.D Sample Name: Lin-3

Acq. Operator	: SYSTEM	Seq. Line :		
	t : LC 1260 DAD	Location :		
Injection Date	: 6/7/2022 11:24:31 PM	Inj:		
Aca Mothod		Inj Volume :		umiovozin M
Acq. Method Last changed	: D:\HPLC\Data\Flumioxazi : 11/17/2021 8:30:46 AM b		0-0/ 1/-1/-09(TI	umitoxaziti'M
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Last changed	: 6/8/2022 9:14:04 AM by			
	Sig=288,4 Ref=off (D:\HPLC\DATA\FLUM		06-07 17-17-09\024-P2-B3	-Lin-3.D)
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	& Dilution Factor with IST	Ds		
Signal 1: DAD1	A, Sig=288,4 Ref=off			
Peak RetTime		Area Name		
# [min] !!-	[min] [mAU*s]	% 		
1 12.749 B				
Totals :	8015.36426			
	*** [nd of Dono			
	*** End of Repo	ru		
60 DAD 6/8/20	22 9:39:17 AM SYSTEM			Page 1 of 1

# FIGURE 11: LINEARITY CHECK FOR LIN-3, injection 1



Data File D:\HPLC\DATA\FLUMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\025-P2-B3-Lin-3.D Sample Name: Lin-3

			-
Acq. Operator	: SYSTEM	Seq. Line : 25	
	t : LC 1260 DAD	Location : P2-B-0	93
Injection Date	: 6/7/2022 11:40:27 PM	Inj: 2	
		Inj Volume : 10.000 µ	
Acq. Method Last changed	: D:\HPLC\Data\Flumioxazin		τ/-W>\tiumioxazin.M
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Last changed	: 6/8/2022 9:14:04 AM by S		
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Sorted By	: Signal		
Calib. Data Mc	dified : 6/8/2022 8:32	:43 AM	
Multiplier	: 1.0000		
Dilution	: 1.0000 & Dilution Factor with ISTD	-	
ose muitipiier	a Difuction Factor with ISID.	5	
Signal 1: DAD1	A, Sig=288,4 Ref=off		
Peak RetTime	Type Width Area A	rea Name	
# [min]	[min] [mAU*s]	%	
1 12.750 B			
Totals :	8015.77783		
	3013.77703		
	*** End of Repor		
		-	
260 DAD 6/8/20	22 9:39:34 AM SYSTEM		Page 1 of 1

FIGURE 12: LINEARITY CHECK FOR LIN-3, injection 2



#### APPENDIX B: CERTIFICATE OF ANALYSIS FOR TEST ITEM



#### **CERTIFICATE OF ANALYSIS**

		F	lumioxazin V	WG	
Manufacture Date	May 25, 20	022	Test Date	Ma	ay 25, 2022
Batch No.	20220509	8	Expiry Date	Ma	ay 25, 2024
	Test R	esult & Con	clusion		
Analysis Items		Specifica	tion	Tes	t results
Appearance	Ι	_ight brown	granule	Light bi	own granule
Active ingredient, %	(w/w)	≥ 51	0		52.0
Moisture, % (w/v	7)	≤ 2.	)		0.62
pH Value		5 -8			7.40
Emulsion stabilit	y	Qualifi	ed	Qı	alified
Storage condition	n Stor	re in origina		n a well-vent e area.	ilated, cool, dry,
				Report Date	May 25, 2022
Chief Analyzer: Sun Jing	Exami	ner: Song S	nuyan	Inspector:	Geng Diangang
_	-				
SHANDONG BINNONG Stamp: Date: May 25, 2022	H	GY CO., LT	D.		



# APPENDIX C: CERTIFICATE OF ANALYSIS FOR REFERENCE ITEM

LGC				. 1	
			Dr. Ehrensto	ference Materials for	
Certific	ate of Analysis			Residue Analysie	
Product Identi	DRE-C13725000 Flumioxazin C19H15FN2O4		Lot Number: Expiry Date: Storage Temp	G140220 16.02.2023 erature: 20°C ± 4°C	
Storage and hand	ling: The RM should be stored in the original sealed riginal sealed bottles under recommended storage c	bottle at the temperatur given above. After u	se the bottle should be tightly closed and prot	ected from moisture and light. The e	expiry
date is valid for of		onarions only.			
-	Purity: 99.47% (g/g)				
	Expanded Uncertainty U= 1.05% (g/g)				
Analytical Data					
	omatography: To the International System of Units HPL/CDAD DAD Reprofil 100 C18 5 µm 250 x 3 mm 10 µl 1.0 ml/min 1.62 min	(SI). Method Datails Acetonitrile:Water 4:1			
Intrument: Detection: Column: InjVol.: Flow: Ret.Time: Comment Traceability: The I Calibrated class A Cartificate Revisio	HPLC/DAD DAD Reprofil 100 C18 5 µm 250 x 3 mm 10 µl 1.0 ml/min 1.5z min balances used are calibrated with weights traceable glassware is used for volumotric measurements.	Method Details Acetonitrile:Water 4:1			
Intrument: Detection: Column: InjVol.: Flow: Ret.Time: Comment Traceability: The I Calibrated class A Cartificate Revisio Purity was detern Identity: EA, NMR	HPLC/DAD DAD DAD 10 Jul 1.0 m/min 1.52 min balances used are calibrated with weights traceable glassware is used for volumetric measurements. in 1 inned by quantitative NMR.	Method Details Acetonitrile:Water 4:1			
Intrument: Detaction: Column: InjVol.: Flow: Ret.Time: Comment Traceability: The I Calibrated class A Cartificate Revisic Purity was detern Identity: EA, NMR Certified on:	HPLC/DAD DAD DAD 10 Jul 1.0 ml/min 1.62 min balances used are calibrated with weights traceable glassware is used for volumetric measurements. in 1 Inned by quantitative NMR.	Method Details Acetonitrile:Water 4:1			
Intrument: Detection: Column: InjVol.: Flow: Ret.Time: Comment Traceability: The t Calibrated class A Calibrated class A Calibrate Revisic Purity was detern	HPLC/DAD DAD Reprofil 100 C18 5 µm 250 x 3 mm 10 µl 1.0 ml/min 1.6z min balances used are calibrated with weights traceable glassware is used for volumetric measurements. on 1 nined by quantitative NMR. , RT, IR, UV 26.05.2017	Method Details Acetonitrile:Water 4:1	from t	rized copy he original JUNI 2017	
Intrument: Detaction: Column: InjVol.: Flow: Ret.Time: Comment Traceability: The I Calibrated class A Cartificate Revisic Purity was detern Identity: EA, NMR Certified on:	HPLC/DAD DAD Reprofil 100 C18 5 µm 250 x 3 mm 10 µl 1.0 ml/min 1.82 min balances used are calibrated with weights traceable glassware is used for volumetric measurements. on 1 nined by quantitative NMR. k, RT, IR, UV 26.05.2017 N. Müller The LGC Labor GmbH, accrete has shown competence based or reference materials in form of organ	Method Details Acetonitrile:Water 4:1	from t 1 6. Sign.: ation number D-RM-19883-01 & D-Pi-19883- N EN ISO/IIC 37025-2005 for production of o multi-component solutions of organic pure to	he original JUNI 2017	
Intrument: Detaction: Column: InjVol.: Flow: Ret.Time: Comment Traceability: The I Calibrated class A Cartificate Revisic Purity was detern Identity: EA, NMR Certified on:	HPLC/QAD DAD Reprofil 100 C18 5 µm 250 x 3 mm 10 µl 1.0 ml/min 1.82 min balances used are calibrated with weights traceable glassware is used for volumetric measurements. on 1 nined by quantitative NMR. k, KT, IR, UV 26,05.2017 N. Möller The LGC Labor GmbH, accret has shown competence based or reference materials in form of organ	Method Details Acetonitrile:Water 4:1 to the national standards (DKD).	tion number D-RM-19883-01 & D-PL-19883- NI EN ISO/ICC 37025-2005 for production of o multi-component solutions of organic pure to 199 Augsburg - Germany USPAugsburg - Germany	he original JUNI 2017	
Intrument: Detection: Column: InjVol.: Flow: Ret.Time: Comment Traceability: The I Calibrated class A Cartificate Revisic Purity was detern Identity: EA, NMR Certified on:	HPLC/DAD DAD Reprofil 100 C18 5 µm 250 x 3 mm 10 µl 1.0 ml/min 1.82 min balances used are calibrated with weights traceable glassware is used for volumetric measurements. on 1 nined by quantitative NMR. k, KT, IR, UV 26,05.2017 N. Möller The LGC Labor GmbH, accred has shown competence based or reference materials in form of organ	Method Details Acetonitrile:Water 4:1 to the national standards (DKD).	tion number D-RM-19883-01 & D-PL-19883- NI EN ISO/ICC 37025-2005 for production of o multi-component solutions of organic pure to 199 Augsburg - Germany USPAugsburg - Germany	he original JUNI 2017	

To be continue



Acq. Operator : SYSTEM Seq. Line : 28 Acq. Instrument : LCMS Location : 54 Injection Date : 13.02.2017 21:49:12 Inj Volume : 10.000 µl Acq. Method : C:\Chem32\1\Data\2017RW07\130217-2 2017-02-13 15-53-28\41K.M Last changed : 10.11.2015 09:04:07 by DAD3_Admin Method Info : Acetonitrile : Water 4:1 Sample Info : Flumioxazin Additional Info : Peak(s) manually integrated TOAD, Sg-21200, 200 Ref-off, EXT 507ted By : Retention Time Multiplier & Dilution Factor with ISTDs Signal 1: DADL, Sig=212.00, 2.00 Ref-off, EXT Signal has been modified after loading from rawdata file!
Last changed : 13.02.2017 15:53:29 by SYSTEM Analysis Method : L:\GERATE BACKUP\DAD3\METHODS\41K.M Last changed : 10.11.2015 09:04:07 by DAD3_Admin Method Info : Reetonitrile : Water 4:1 Sample Info : Flumioxazin Additional Info : Peak(s) manually integrated TADDI, Sig=212.00, 200 Nem-off, EXT of 19725000.D TADDI, Sig=212.00, 200 Nem-off, EXT of 19725000.D TATE Percent Report Area Percent Report Sorted By : Retention Time Multiplier : 1.0000 Diution : 1.0000 Diution : 1.0010, Sig=212.00, 2.00 Ref=off, EXT
Additional Info : Peak(s) manually integrated "DADI, Stg=212.00, 2.00 Ref=off, EXT "DADI, Stg=212.00, 2.00 Ref=off, EXT Additional Info : Peak(s) manually integrated "DADI, Stg=212.00, 2.00 Ref=off, EXT Additional Info : Peak(s) manually integrated "DADI, Stg=212.00, 2.00 Ref=off, EXT Additional Info : Peak(s) manually integrated "DADI, Stg=212.00, 2.00 Ref=off, EXT
************************************
175       180       125       100       75       50       25       0       25       0       2       3       4       min       Sorted By       :       Retention Time       Multiplier       :       1.0000       Use Multiplier & Dilution Factor with ISTDs       Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
150         125         100         75         50         25         0         25         0         25         0         25         0         25         0         25         3         4         min         Area Percent Report
Area Percent Report Area Percent Report Sorted By : Retention Time Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
100       75       50       25       0       25       0       2       3       Area Percent Report
75         50         25         0         26         0         27         3         4         min         Area Percent Report         Area Percent Report         Sorted By       : Retention Time         Multiplier       : 1.0000         Dilution       : 1.0000         Use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
75         50         25         0         25         0         26         0         27         3         4         min         Area Percent Report         Area Percent Report         Sorted By       : Retention Time         Multiplier       : 1.0000         Dilution       : 1.0000         Use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
Sorted By : Retention Time Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
25       0       1       2       3       4       min       Area Percent Report
Area Percent Report 
Area Percent Report Area Percent Report Sorted By : Retention Time Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
Area Percent Report         Sorted By       :         Multiplier       :         1.0000         Dilution       :         1.0000         Use Multiplier & Dilution Factor with ISTDs         Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
Sorted By : Retention Time Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
Multiplier : 1.0000 Dilution : 1.0000 Use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
Use Multiplier & Dilution Factor with ISTDs Signal 1: DAD1, Sig=212.00, 2.00 Ref=off, EXT
Peak RetTime Sig Type         Area         Height         Area           # [min]         [mAU*s]         [mAU]         %                        1         1.618         1         BB         717.47504         196.52742         100.0000
Totals : 717.47504 196.52742