Method Extension of CIPAC 578 in Flumioxazin WG

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Test Guideline: CIPAC No. 3807, CIPAC 578

Content

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:

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.

Table 1 The specificity test results

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

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.
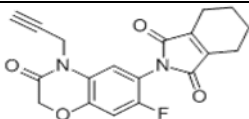
2. Introduction

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 AI	$C_{19}H_{15}FN_2O_4$
Molecular weight of AI	354.3 g/mol
Structure formula of AI	
Chemical name (IUPAC) of AI	N-(7-fluoro-3,4-dihydro-3-oxo-4-prop-2-ynyl-2H-1,4-benzoxazin-6-yl) cyclohex-1-ene-1,2-dicarboxamide
CAS No. of AI	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, 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	20±4℃
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 ℃ in Test Substance Control Office of BNS QC Center.

The reference item was stored at 20±4℃ 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.: I0972907839, supplied by Merck KGaA, Germany.

Ultrapure water, resistivity: ≥10 MΩ·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.

4.3 Analytical condition

Detector:	DAD
Wavelength:	288 nm
Column:	Phenomenex Gemini (250mm x 4.6mm x 5μm)
Column temperature:	40℃
Injection volume:	10.0 μl
Mobile phase:	Solvent A: Ultrapure Water
	Solvent B: Acetonitrile
Stop time:	15 min
Post time	0 min

Time (min)	Flow (mL/min)	A (%)	B (%)
0	1.000	50	50

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 μm 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 μl 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 μm filter prior to analysis.

5. Result and Conclusion

5.1 Specificity test results

Table 1 Specificity of Flumioxazin WG

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

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.

Table 2 Peak purity of Flumioxazin in the method

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.

$$y = 7.9904x + 57.225$$
$$R^2 = 1$$
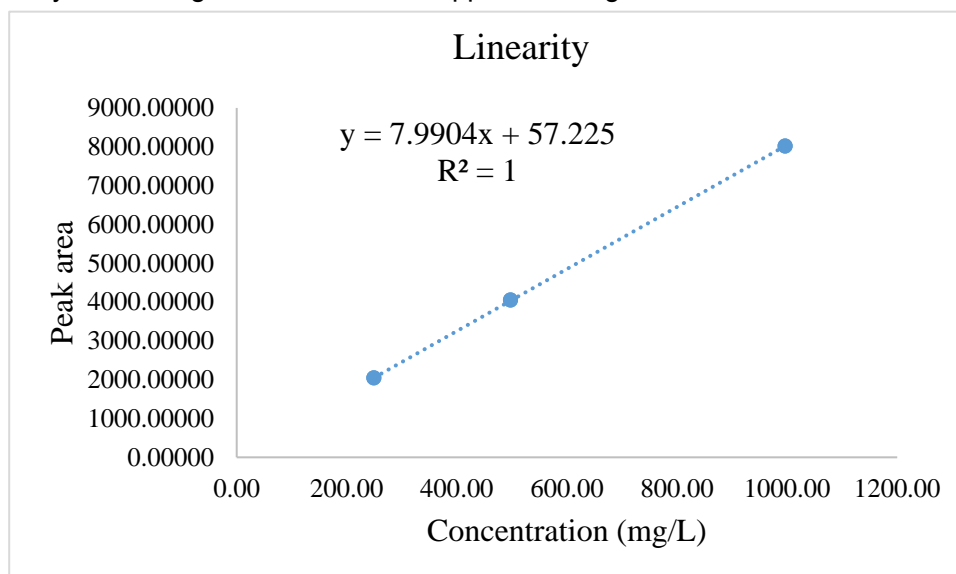
The correlation coefficient (r) is $>$ 0.99. over the range (200% ~ 50% of the nominal).

The test item solution injected for specificity test with peak area 4082.98804, is in the range of the linearity concentration.

Table 3 Peak area of Flumioxazin reference and test item solutions

Sample solution name	Lin-1	Lin-2	Lin-3
Average peak area for 2 injections	2041.79016	4047.97559	8015.57105
Peak area of Test item solution for specificity	4082.98804		
Peak area of Reference item solution for specificity	4110.81250		

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: 96.66mg /100ml *1000* 52.0%= 502.6mg/l, and in the method (CIPAC 578), the nominal concentration of flumioxazin used as (50mg /100ml =500mg/l), 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
Injection Date  : 6/7/2022 9:17:02 PM             Inj :   1
                                          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
```
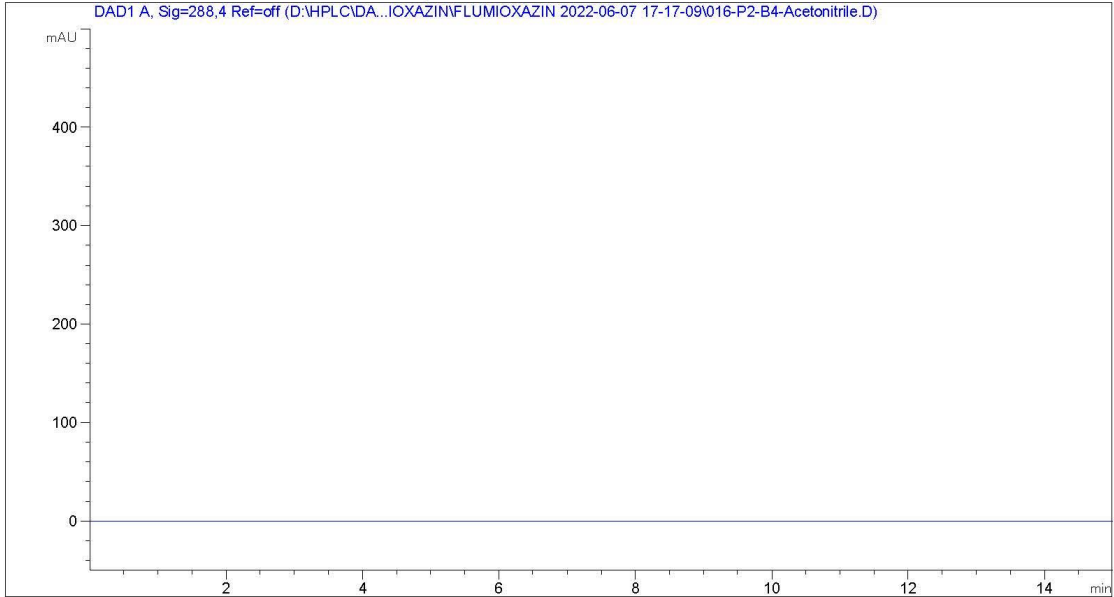


```
==================================================================
                    Area Percent Report
==================================================================


Sorted By              :      Signal
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

No peaks found


==================================================================
                    *** End of Report ***
```

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

```
==================================================================
Acq. Operator   : SYSTEM                    Seq. Line :  17
Acq. Instrument : LC 1260 DAD               Location :   P2-B-05
Injection Date  : 6/7/2022 9:32:58 PM            Inj :   1
                                          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
```



```
==================================================================
                    Area Percent Report
==================================================================


Sorted By            :      Signal
Multiplier           :      1.0000
Dilution             :      1.0000
Use Multiplier & Dilution Factor with ISTDs


No peaks found


==================================================================
                    *** End of Report ***
```

LC 1260 DAD 6/8/2022 4:20:15 PM SYSTEM   Page   1 of 1

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  : 6/7/2022 9:48:54 PM          Inj :   1
                                          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
```



```
==================================================================
                      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]        [min]   [mAU*s]      [mAU]         %
----|-------|----|-------|----------|----------|--------|
  1  12.720 BB    0.2578 4110.81250  244.96487 100.0000

Totals :                 4110.81250  244.96487


==================================================================
                      *** End of Report ***
```
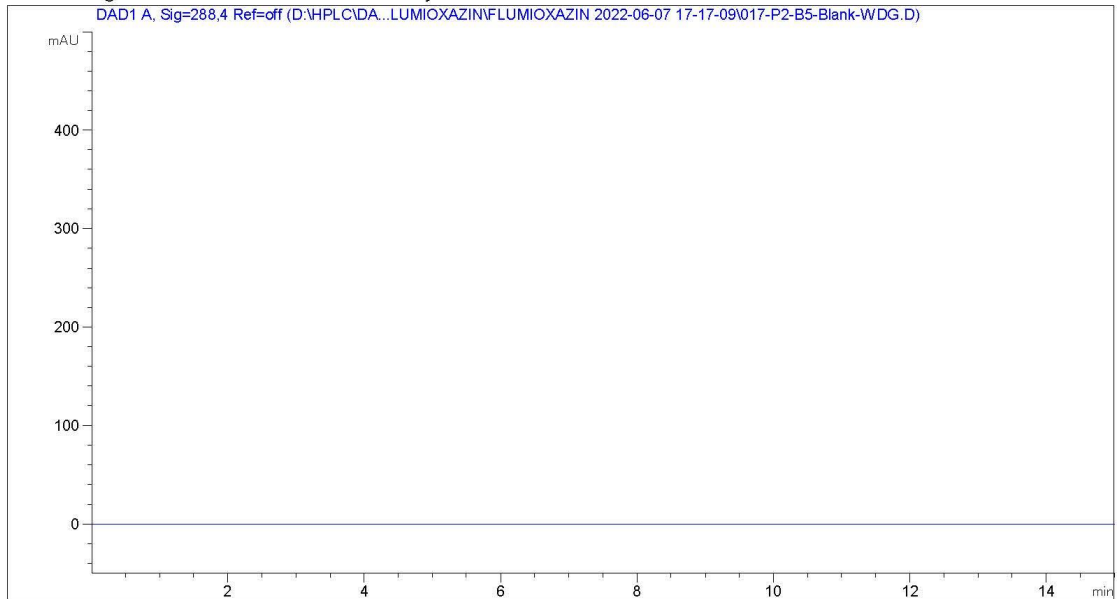
LC 1260 DAD 6/8/2022 4:20:41 PM SYSTEM   Page   1 of 1

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
Injection Date  : 6/7/2022 10:04:50 PM           Inj :   1
                                           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
```



```
===================================================================
                     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]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  12.714 BB    0.2576 4082.98804  243.51692 100.0000

Totals :                  4082.98804  243.51692


===================================================================
                      *** End of Report ***
```

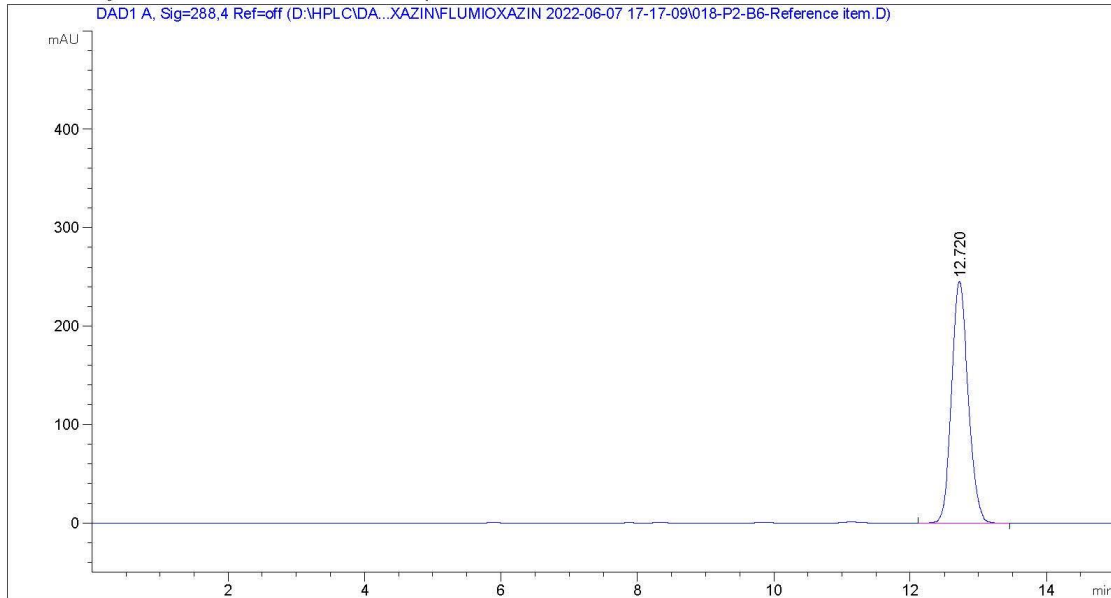LC 1260 DAD 6/8/2022 4:21:15 PM SYSTEM   Page   1 of 1

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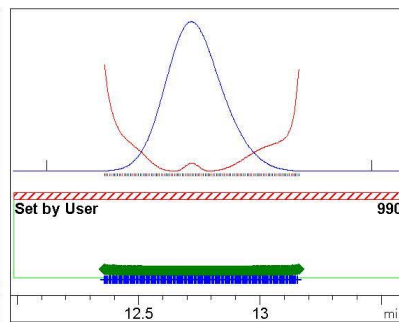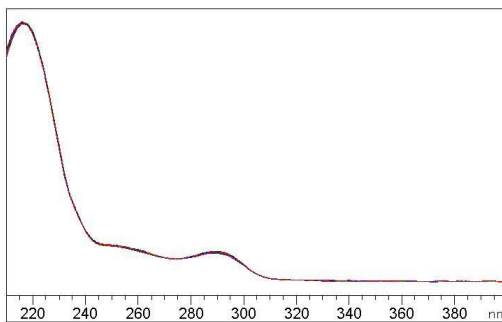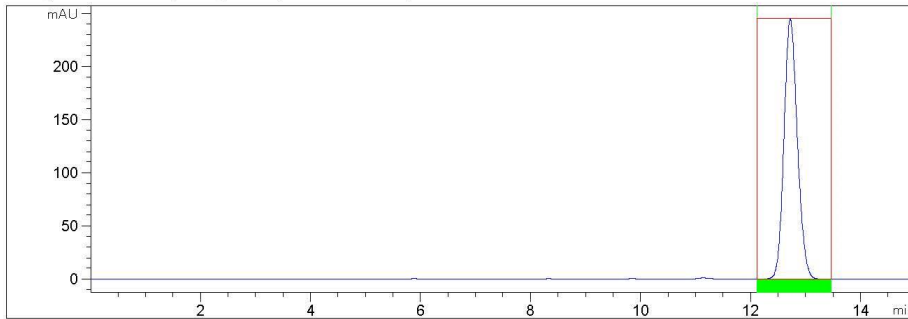
-> The purity factor is within the threshold limit. <-

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 ***

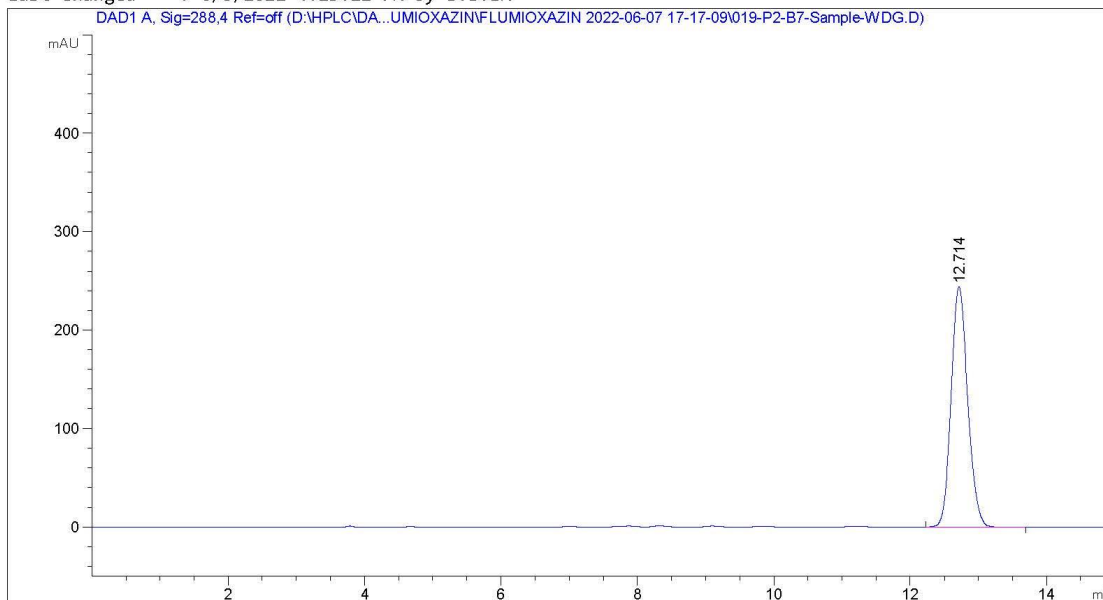LC 1260 DAD 6/8/2022 10:39:40 AM SYSTEM   Page   1 of 1

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.

Signal DAD1 A, Sig=288,4 Ref=off (D:\HPLC\DA...UMIOXAZIN\FLUMIOXAZIN 2022-06-07 17-17-09\01
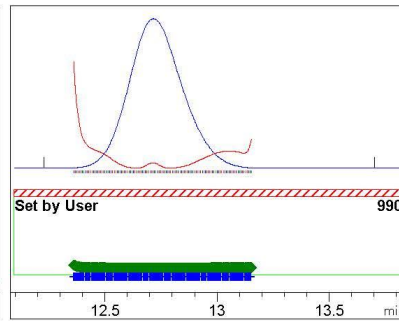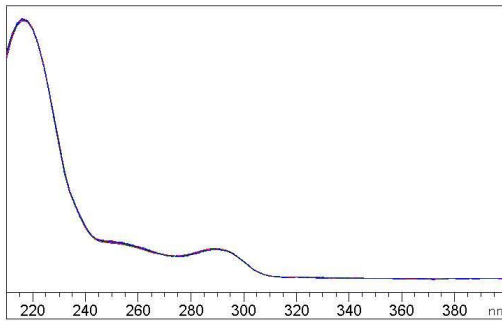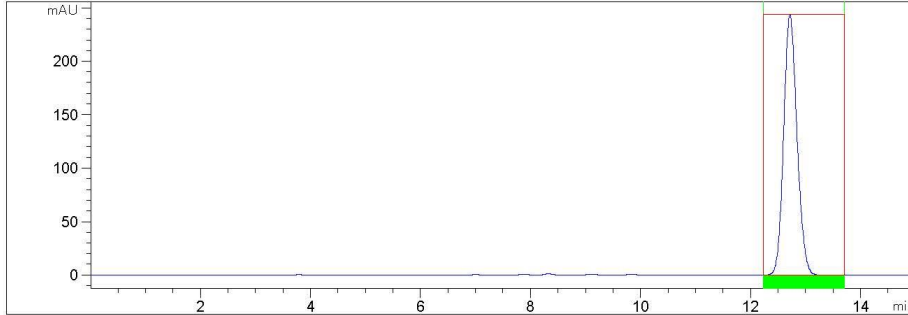
-> The purity factor is within the threshold limit. <-

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
Acq. Instrument : LC 1260 DAD                Location :   P2-B-01
Injection Date  : 6/7/2022 10:20:46 PM              Inj :    1
                                             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 9:14:04 AM by SYSTEM
```



```
=================================================================
                      Area Percent Report
=================================================================

Sorted By               :      Signal
Calib. Data Modified    :      6/8/2022 8:32:43 AM
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       Area      Name
  #   [min]         [min]    [mAU*s]       %
----|-------|------|-------|----------|--------|---------------------
  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
```

```
=====================================================================
Acq. Operator   : SYSTEM                      Seq. Line :   21
Acq. Instrument : LC 1260 DAD                  Location :   P2-B-01
Injection Date  : 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\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 9:14:04 AM by SYSTEM
```
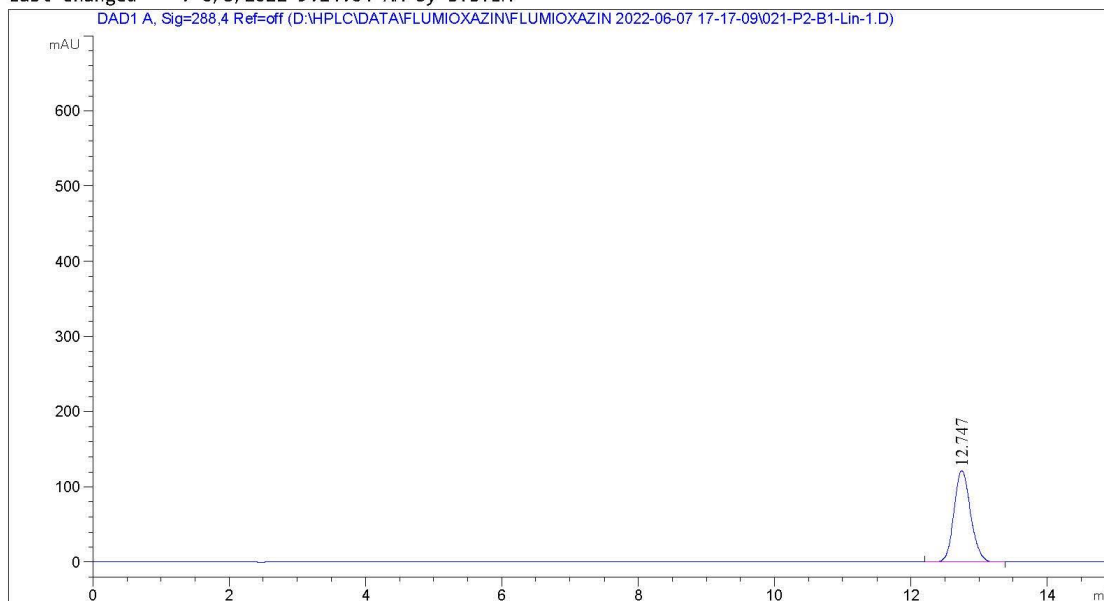


```
=================================================================
                     Area Percent Report
=================================================================

Sorted By            :        Signal
Calib. Data Modified :        6/8/2022 8:32:43 AM
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       Area     Name
  #   [min]          [min]   [mAU*s]       %
----|-------|------|-------|----------|--------|--------------------
  1  12.747 BB     0.2583 2043.10425 100.0000 Flumioxazin

Totals :                   2043.10425


=================================================================
                     *** End of Report ***
```

LC 1260 DAD 6/8/2022 9:38:06 AM SYSTEM   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
Acq. Instrument : LC 1260 DAD                  Location :   P2-B-02
Injection Date  : 6/7/2022 10:52:38 PM            Inj :    1
                                           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 9:14:04 AM by SYSTEM
```



```
======================================================
                  Area Percent Report
======================================================

Sorted By            :      Signal
Calib. Data Modified :      6/8/2022 8:32:43 AM
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       Area      Name
  #   [min]          [min]    [mAU*s]       %
----|-------|------|-------|----------|--------|--------------------
   1  12.735 BB     0.2582 4043.69287 100.0000 Flumioxazin

Totals :                    4043.69287


======================================================
                  *** End of Report ***
```
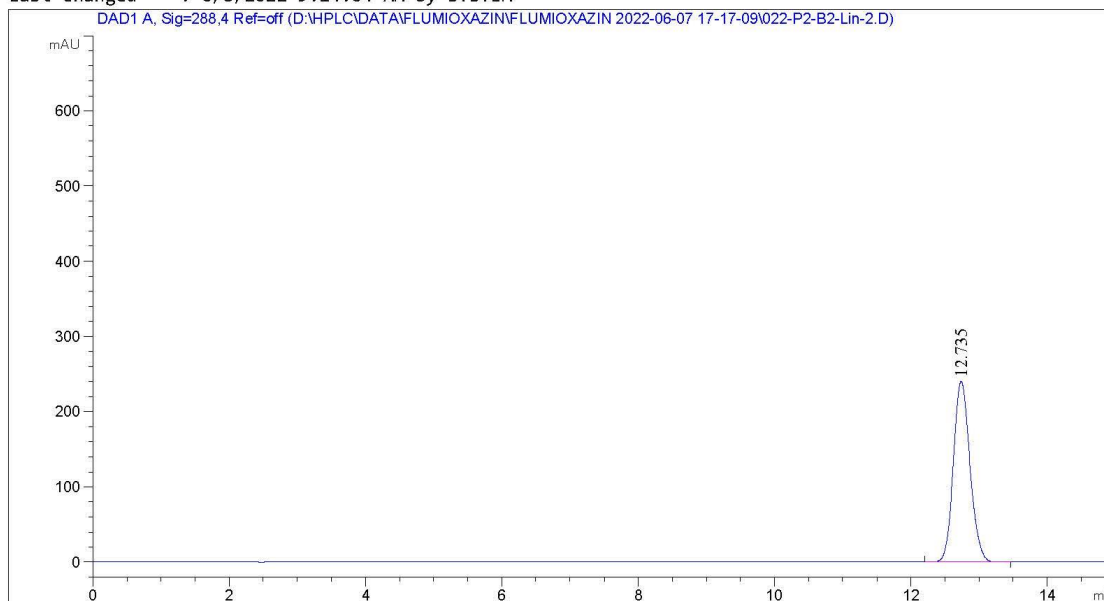
FIGURE 9: LINEARITY CHECK FOR LIN-2, injection 1

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

```
=====================================================================
Acq. Operator   : SYSTEM                      Seq. Line :  23
Acq. Instrument : LC 1260 DAD                 Location :  P2-B-02
Injection Date  : 6/7/2022 11:08:34 PM             Inj :   2
                                         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 9:14:04 AM by SYSTEM
```



```
=====================================================================
                       Area Percent Report
=====================================================================

Sorted By             :      Signal
Calib. Data Modified  :      6/8/2022 8:32:43 AM
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       Area      Name
  #   [min]          [min]    [mAU*s]       %
----|-------|------|-------|----------|--------|--------------------
  1  12.745 BB     0.2579 4052.25830 100.0000 Flumioxazin

Totals :                    4052.25830


=====================================================================
                       *** End of Report ***
```

LC 1260 DAD 6/8/2022 9:38:57 AM SYSTEM   Page   1 of 1
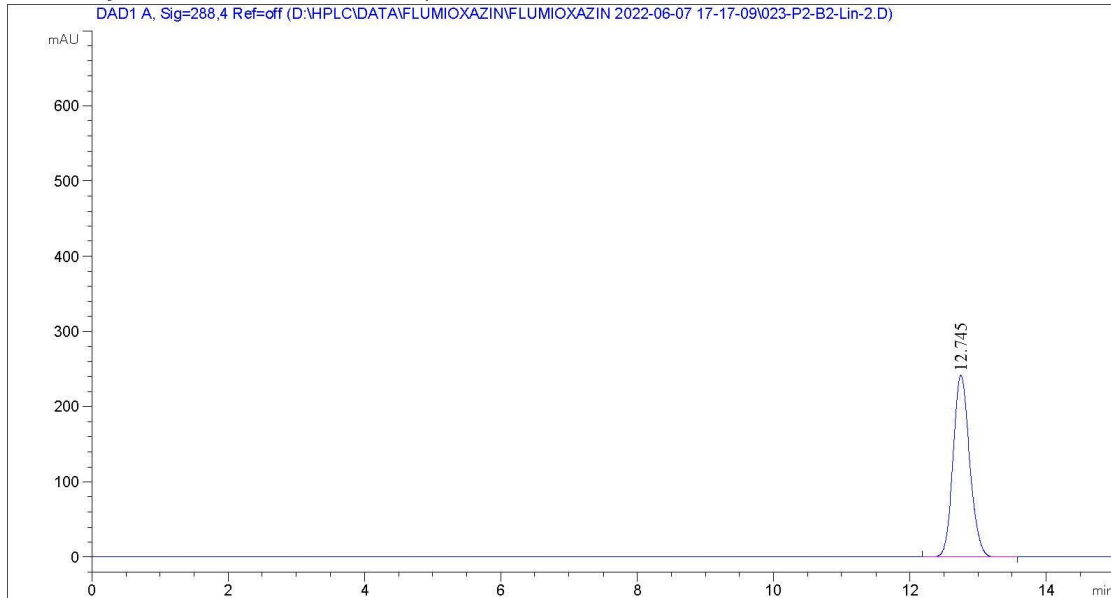
FIGURE 10: LINEARITY CHECK FOR LIN-2, injection 2

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 :   24
Acq. Instrument : LC 1260 DAD                  Location :   P2-B-03
Injection Date  : 6/7/2022 11:24:31 PM            Inj :    1
                                           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 9:14:04 AM by SYSTEM
```



```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By            :        Signal
Calib. Data Modified :        6/8/2022 8:32:43 AM
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      Area      Name
  #   [min]        [min]   [mAU*s]      %
----|-------|------|-------|----------|--------|--------------------
  1  12.749 BB     0.2580 8015.36426 100.0000 Flumioxazin

Totals :                  8015.36426


=====================================================================
                      *** End of Report ***
```

LC 1260 DAD 6/8/2022 9:39:17 AM SYSTEM   Page   1 of 1
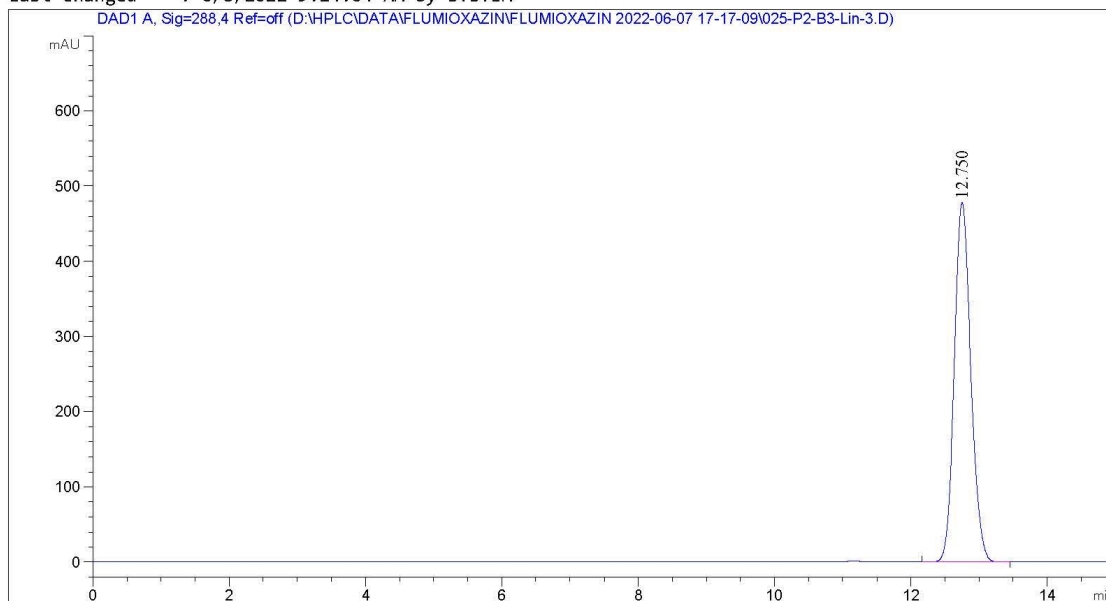

FIGURE 11: LINEARITY CHECK FOR LIN-3, injection 1

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
Acq. Instrument : LC 1260 DAD               Location :   P2-B-03
Injection Date  : 6/7/2022 11:40:27 PM         Inj :    2
                                         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 9:14:04 AM by SYSTEM
```



```
==================================================================
                    Area Percent Report
==================================================================

Sorted By             :      Signal
Calib. Data Modified  :      6/8/2022 8:32:43 AM
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       Area      Name
  #   [min]         [min]    [mAU*s]       %
----|-------|------|-------|----------|--------|--------------------
  1  12.750 BB     0.2577 8015.77783 100.0000 Flumioxazin

Totals :                   8015.77783


==================================================================
                    *** End of Report ***
```

LC 1260 DAD 6/8/2022 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

山东滨农科技有限公司
SHANDONG BINNONG TECHNOLOGY CO.,LTD

CERTIFICATE OF ANALYSIS

Product Name	Flumioxazin WG		
Manufacture Date	May 25, 2022	Test Date	May 25, 2022
Batch No.	202205098	Expiry Date	May 25, 2024
Test Result & Conclusion			
Analysis Items	Specification		Test results
Appearance	Light brown granule		Light brown granule
Active ingredient, % (w/w)	$\geqslant$ 51.0		52.0
Moisture, % (w/w)	$\leqslant$ 2.0		0.62
pH Value	5 -8		7.40
Emulsion stability	Qualified		Qualified
Storage condition	Store in original container, in a well-ventilated, cool, dry, secure area.		
All the items are qualified for use and transport			

Report Date: May 25, 2022

Chief Analyzer: Sun Jing   Examiner: Song Shuyan   Inspector: Geng Diangang

SHANDONG BINNONG TECHNOLOGY CO., LTD.

Stamp:

Date: May 25, 2022

APPENDIX C: CERTIFICATE OF ANALYSIS FOR REFERENCE ITEM

LGC

Dr. Ehrenstorfer

Reference Materials for
Residue Analysis

Certificate of Analysis

ISO Guide 34 Reference Material

Product Identification

Article Code:	DRE-C13725000	Lot Number:	G140220
Article Name:	Flumioxazin	Expiry Date:	16.02.2023
Formula:	C19H15FN2O4	Storage Temperature:	20°C ± 4°C
Mol. Weight:	354.33		
CAS No.:	103361-09-7		

Storage and handling: The RM should be stored in the original sealed bottle at the temperatur given above. After use the bottle should be tightly closed and protected from moisture and light. The expiry date is valid for original sealed bottles under recommended storage conditions only.

Purity:	99.47% (g/g)
Expanded Uncertainty U=	1.05% (g/g)

The uncertainty of this standard is calculated in accordance with the ISO Guide 34 and EURACHEM/CITAC Guide - Quantifying Uncertainty in Analytical Measurement, Second Edition. The expanded uncertainty is $U(exp) = u(RM) \times k$, where k is the coverage factor at the 95% confidence level (k=2). Uncertainty u(RM) is based on the combination of the uncertainties associated with each individual operation involved in the analysis of the product: $u(RM) = \sqrt{u(char)^2 + u(bb)^2 + u(lts)^2 + u(sts)^2}$; u(char) is the uncertainty of purity determination; u(bb) uncertainty of homogeneity test; u(lts) uncertainty of stability test long-term; u(sts) uncertainty of stability test short-term. u(lts) and u(sts) are not included in the calculation as the stability statement is based on real evidence opposed to simulation. Minimum sample: 1 mg is recommended as the minimal sample amount. If less material is used, it is recommended to increase the certified uncertainty by a factor of two for half sample and a factor of four for a quarter of sample.
Intended use: Use this RM as calibrant for chromatography or any other analytical technique.

Analytical Data

Traceability of chromatography: To the International System of Units (SI).

Intrument:	HPLC/DAD	**Method Details**
Detection:	DAD	Acetonitrile:Water 4:1
Column:	ReproSil 100 C18 5 µm 250 x 3 mm	
Inj.-Vol.:	10 µl	
Flow:	1.0 ml/min	
Ret.Time:	1.62 min	

Comment
Traceability: The balances used are calibrated with weights traceable to the national standards (DKD).
Calibrated class A glassware is used for volumetric measurements.
Certificate Revision 1
Purity was determined by quantitative NMR.

Identity: EA, NMR, RT, IR, UV

Certified on:	26.05.2017
Certified by:	N. Müller

Authorized copy
from the original
1 6. JUNI 2017
Sign.:

To be continue

Data File L:\GERÄTE BACKUP\DAD4\2017\2017KW07\130217-2 2017-02-13 15-53-28\13725000.D

14.02.17
14

```
===================================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  28
Acq. Instrument : LCMS                          Location  :  54
Injection Date  : 13.02.2017 21:49:12               Inj   :   1
                                                Inj Volume : 10.000 µl
Acq. Method     : C:\Chem32\1\Data\2017KW07\130217-2 2017-02-13 15-53-28\41K.M
Last changed    : 13.02.2017 15:53:29 by SYSTEM
Analysis Method : L:\GERÄTE BACKUP\DAD3\METHODS\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
```
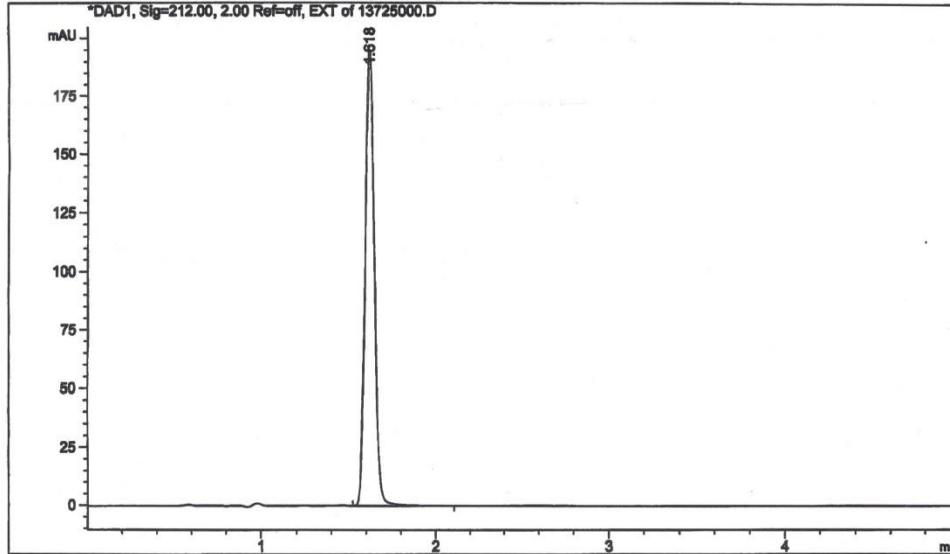

*DAD1, Sig=212.00, 2.00 Ref=off, EXT of 13725000.D

```
===================================================================================
                         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
 Signal has been modified after loading from rawdata file!

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
```

```
===================================================================================
                         *** End of Report ***
```