„"INDUSTRIAL TRAINING""

A REPORT SUBMITTED TO

SAVITRIBAI PHULE PUNE UNIVERSITY, PUNE

FOR THE DEGREE OF

MASTER OF SCIENCE

IN

ORGANIC CHEMISTRY

UNDER THE FACULTY OF SCIENCE

BY

Mr. Aniket Shivaji Tungar

Department of Chemistry, G. M. D.Arts,B.W. Commerce and Science College, Sinnar

UNDER THE GUIDANCE OF

Prof. :- Dr. M.R. Gaware

Head of

DEPARTMENT OF CHEMISTRY

G.M.D.ARTS, B.W.COMMERCE AND SCIENCE COLLEGE,

SINNAR 422103

APRIL 2023

Maratha Vidya Prasarak Samaj's

G.M.D. ARTS, COMMERCE AND SCIENCE COLLEGE,

SINNAR, DISTRICT- NASHIK

DEPARTMENT OF CHEMISTRY (PG)

CERTIFICATE

This is to certify that **Mr. Aniket Shivaji Tungar** studying in M.Sc.-II (Organic Chemistry) at **M.V.P. Samaj's G.M.D. Arts, B.W. Commerce and Science College , Sinnar** has successfully completed "Pharmaceutical Training Course in Analytical Techniques" **(CHO-453-Industrial Training)** from 07/12/2022 to 07/01/2023 conducted by Arni Analyticals, Nashik during the semester IV of academic year 2022-2023.

HOD Chemistry

HEAD
DEPARTMENT OF CHEMISTRY
M.D. Arts, B.W. Commerce
Science college, Sinnar

Examiner

12-05-2023

Principal

PRINCIPAL
G.M.D.Arts, B.W.Commerce and
Science College, Sinnar, Dist. Nashik

ISO 9001 CERTIFIED

ARNI ANALYTICAL

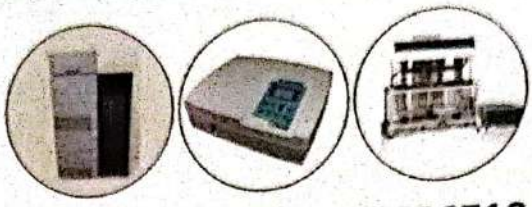
Add.: Pushpak Apartment, Flat No. 102, Lane No. 3, Near Neurocare Hospital, Pandit Colony, Nashik.
e-mail : arnianalyticals@gmail.com | Web Site : www.arnianalyticals.com

Certificate

This is to Certify that ..... *Aniket Shivaji Tungar* .....
has Successfully Completed Pharmaceutical Training Course in
Analytical Techniques includes Practically Handling the
Instruments Like HPLC, UV - Spectrophotometer,
Dissolution Test Apparatus & Pharmaceutical
Instruments in the Training Period From *7 Dec. 2022* To *7 Jan. 2023*

Director

ARNI
ANALYTICALS



: 9307686710

Certificate

"Pharmaceutical Training Course in Analytical Techniques"

This is to certify that Mr./Miss/ Mrs. **Aniket Shivaji Tungar** studying in **M. Sc.-II (Organic Chemistry)** at **M. V. P. Samaj's G. M. D. Arts, B. W. Commerce and Science College, Sinnar** has successfully completed **"Pharmaceutical Training Course in Analytical Techniques"** from 07/12/2022 to 07/01/2023 conducted by **Arni Analyticals, Nashik** and has obtained **"B+"** grade.

Mr. Masum Deshmukh
Director

Add.: Pushpak Apartment, Flat No. 102, Lane No. 3, Near Neurocare Hospital, Pandit Colony, Nashik. | **e-mail :** arnianalyticals@gmail.com

ACKNOWLEDGEMENT

The success and final outcome of this training required a lot of guidance and assistance from many people. All that I have done is only due to such supervision and assistance and I would never forget to thank them.

I respect and thank Respected Dr. P.V. Rasal Sir for providing me an opportunity to do the training and giving all the support and guidance which made me complete the training successfully. I am extremely thankful to him for providing such a nice support and guidance.

I owe my deep gratitude to Prof. Manoj Gaware Sir (Head of Chemistry Department) who took interest on my training and guided me all along, till the completion of training by providing all the necessary information .

I am thankful to Mr. Masum Deshmukh Sir for his guidance and suggestions during the training and thankful for giving all the knowledge during the training.

I am thankful to and fortunate enough to get constant encouragement, support and guidance from all Teaching Staffs of Department of Chemistry which helped me in successfully completing my training.

Sign:- Tungar

Name:- Aniket Shivaji Tungar

INDEX

TENELIGLIPTIN

Introduction-

- Teneligliptin is a pharmaceutical drug for the treatment of type-2 diabetes mellidus.
- Teneligliptin belongs to the category of medicines called "anti-diabetic".
- It is used along or in combination with other drugs to lower blood sugar levels.
- Teneligliptin tablet contains the teneligliptin which belongs to class of dipeptidyl peptidase-4 inhibitors.
- It works by blocking the action of DPP-4 (an enzyme that destroys the harmone 'Incretin'). The enzyme 'Incretins' helps to produce more insulin only when required and reduces the liver's blood sugar level when not needed.

Chemical Formula- $C_{22}H_{30}N_6O_5$

Molar Mass- 426.58 gm/mol

- Teneligliptin significantly controls glycemic parameters with safety. No dose adjustment is required.
- As we all know that teneligliptin tablet contains only 20 mg active ingredient i.e. teneligliptin. Other layers or coatings are excepients.
- Once a tablet is formulated then directly it doesn't comes to market. First of all some of the random tablets are collected and forwarded for testing.

Testing have 2 types-

1. Physical
2. Chemical

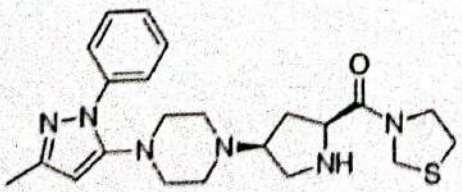
Physical Testing-

1. Average weight test
2. Uniformity of weight
3. Thickness
4. Dimensions
5. **Hardness**

Chemical Tests-

1. Dissolution Test
2. Separation Technique (HPLC)
3. Absorbance

Structure of Teneligliptin-



ARNI ANALYTICALS

TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-	ANALYTICAL BALANCE	Page No.
Instrument Make :-	WENSAR	
Instrument Model No. :-	DS 8000	1 of 3
Instrument ID : -	ARNI/INS-004	

NAME OF STUDENT:- Tungar Aniket Shivaji

MONTHLY CALIBRATION RECORD

1. **Calibration by using Standard certified weights:**

Observation Table:

Sr. No.	Reference Weight in g	Observed Weight in g	Weight in g (Limit: ± 0.1%)
1	200.0000	200.032	199.8000 to 200.2000
2	100.0000	98.773	99.9000 to 100.1000
3	50.0000	50.702	49.9500 to 50.0500
4	20.0000	19.049	19.9800 to 20.0200
5	10.0000	9.998	9.9900 to 10.0100
6	5.0000	5.085	4.9950 to 5.0050
7	2.0000	1.230	1.9980 to 2.0020
8	1.0000	0.922	0.9990 to 1.0010
9	0.5000	0.152	0.4995 to 0.5005
10	0.2000	0.104	0.1998 to 0.2002
11	0.1000	0.058	0.0999 to 0.1001
12	0.0500	0.039	0.0499 to 0.0501
13	0.0200	0.016	0.0199 to 0.0200
14	0.0100	0.019	0.0099 to 0.0100
15	0.0050	0.013	0.0049 to 0.0051

Conclusion: The observed weights are **within limit/~~out-of-limit~~.**

ANALYSED BY

CHECKED BY:-

	ARNI ANALYTICALS	
TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-	ANALYTICAL BALANCE	Page No.
Instrument Make :-	WENSAR	
Instrument Model No. :-	DS 8000	2 of 3
Instrument ID : -	ARNI / INS - 004	

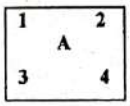
2. Test for Linearity:

Sr. No.	Selected Weights in g	Observed Weight in g
1	20	19.048
2	50	50.696
3	100	98.754

Conclusion: The observed weights are **Consistent/~~not Consistent~~.**

ANALYSED BY **CHECKED BY:-**

3. Test for Eccentricity:



Sr. No.	Weight Observed in g		Difference in g	Limit
1.	At Centre- (A)	19.048		
2.	At Corner 1 (B)	19.049	B-A = 0.001	
3.	At Corner 2 (C)	19.050	C-A = 0.002	± 0.1 %
4.	At Corner 3 (D)	19.049	D-A = 0.001	
5.	At Corner 4 (E)	19.049	E-A = 0.001	

Conclusion: The maximal Differential Eccentricity error is **within limit/~~out of limit~~** of Std. deviation.

ANALYSED BY **CHECKED BY:-**

	ARNI ANALYTICALS	
TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-	ANALYTICAL BALANCE	Page No.
Instrument Make :-	WENSAR	
Instrument Model No. :-	DS 8000	3 of 3
Instrument ID : -	ARNI/INS - 004	

4. Test for Repeatability :

Selected Weight in g: 50

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	50.693	6	50.694	± 0.1 %
2	50.693	7	50.692	
3	50.692	8	50.693	
4	50.690	9	50.692	
5	50.693	10	50.693	

Conclusion: Individual measurement deviation from average value **exceeds/ ~~does not exceed~~** standard deviation.

Remark: The instrument is found **Satisfactory/ ~~unsatisfactory~~** for its use.

ANALYSED BY

CHECKED BY:-

	ARNI ANALYTICALS	
TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-		Page No
Instrument Make :-		
Instrument ID : -		1 of 3

MONTHLY CALIBRATION RECORD

1. **Calibration by using Weights:**

Observation Table:

Sr. No.	Reference Weight in g	Observed Weight in g	Weight in g (Limit: ± 0.1%)
1	200.0000	200.000 g	199.8000 to 200.2000
2	100.0000	98.751 g	99.9000 to 100.1000
3	50.0000	50.692 g	49.9500 to 50.0500
4	20.0000	19.049 g	19.9800 to 20.0200
5	10.0000	9.997 g	9.9900 to 10.0100
6	5.0000	5.081 g	4.9950 to 5.0050
7	2.0000	1.230 g	1.9980 to 2.0020
8	1.0000	0.922 g	0.9990 to 1.0010
9	0.5000	0.152 g	0.4995 to 0.5005
10	0.2000	0.104 g	0.1998 to 0.2002
11	0.1000	0.058 g	0.0999 to 0.1001
12	0.0500	0.039 g	0.0499 to 0.0501
13	0.0200	0.016 g	0.0199 to 0.0200
14	0.0100	0.019 g	0.0099 to 0.0100
15	0.0050	0.013 g	0.0049 to 0.0051

Conclusion: The observed weights are **within limit/** ~~**out of limit.**~~

ANALYSED BY

CHECKED BY

	ARNI ANALYTICALS	
TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-		Page No
Instrument Make :-		
Instrument ID : -		2 of 3

2. Test for Linearity:

Sr. No.	Selected Weights in g	Observed Weight in g
1	20g	19.048g
2	50g	50.696g
3	100g	98.754

Conclusion: The observed weights are **Consistent/not Consistent.**

ANALYSED BY **CHECKED BY**

3. Test for Eccentricity:

1	2
A	
3	4

Sr. No.	Weight Observed in g		Difference in g	Limit
1.	At Centre- (A)		19.048g	
2.	At Corner 1 (B)	19.049	B-A = 0.001	
3.	At Corner 2 (C)	19.050	C-A = 0.002	± 0.1 %
4.	At Corner 3 (D)	19.049	D-A = 0.001	
5.	At Corner 4 (E)	19.049	E-A = 0.001	

Conclusion: The maximal Differential Eccentricity error is **within limit/out of limit** of Std. deviation.

ANALYSED BY **CHECKED BY**

	ARNI ANALYTICALS	
TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-		Page No
Instrument Make :-		3 of 3
Instrument ID : -		

4. Test for Repeatability :

Selected Weight in g: 50g

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	50.693 g	6	50.694 g	± 0.1 %
2	50.693 g	7	50.692 g	
3	50.692 g	8	50.693 g	
4	50.690 g	9	50.692 g	
5	50.693 g	10	50.693 g	

Conclusion: Individual measurement deviation from average value **exceeds/ does not exceed** standard deviation.

Remark: The instrument is found **Satisfactory/ unsatisfactory** for its use.

ANALYSED BY

CHECKED BY

	ARNI ANALYTICALS	
TITLE	DISSOLUTION TEST APPARATUS WORKSHEET	
Instrument Name :-		Page No.
Instrument ID : -		
Instrument Model No. :-		1 of 1
Name Of Students		

NAME OF TEST :-

TRIAL FOR DISSOLUTION TEST.

DISSOLUTION CONDITIONS:-

Dissolution Media	WATER
Media Volume	900 mL
Apparatus	USP TYPE II PADDLE
RPM	100
Temperature	37.0 ± 0.5°C
Time	45 Minutes

PREPARATIONS:-

Pour 900 mL of dissolution medium in each vessel. Allow sufficient time for the dissolution medium to equilibrate at 37°C ± 0.5°C. Adjust stirring element speed to 100 rpm. Place one capsule in each of six paddle and adjust the paddle in the dissolution medium so that there is a distance of 25 mm ± 2 mm between the bottom of the paddle and inside bottom of the vessel. Start the apparatus. At the end of specified time interval, withdraw 10 mL aliquot from a zone midway between the surface of the dissolution medium and at top of the rotating paddle. Further dilute 2ml of the above solution to 25ml with dissolution medium.

ANALYSED BY

CHECKED BY:-

	ARNI ANALYTICALS	
TITLE	DISSOLUTION TEST APPARATUS WORKSHEET	
Instrument Name :-	DISSOLUTION TEST APPARATUS	Page No.
Instrument ID : -	ARNI / INS - 003	
Instrument Model No. :-	DS - 8000	1 of 1
Name Of Students	Tungar Aniket Shivaji	

NAME OF TEST :-

TRIAL FOR DISSOLUTION TEST.

DISSOLUTION CONDITIONS:-

Dissolution Media	WATER
Media Volume	900 mL
Apparatus	USP TYPE II PADDLE
RPM	100
Temperature	37.0 ± 0.5°C
Time	45 Minutes

PREPARATIONS:-

Poured 900 mL of dissolution medium in each vessel. Allowed sufficient time for the dissolution medium to equilibrate at 37°C ± 0.5 °C. Adjusted stirring element speed to 100rpm. Placed one capsule in each of six paddle and adjusted the paddle in the dissolution medium so that there is distance of 25mm ±2mm. Started the apparatus. At the end of specified time interval, withdrawed 10 ml aliquot. Further diluted 2ml of the above solution to 25ml with dissolution medium.

ANALYSED BY

CHECKED BY:-

TEST METHOD

1) **Description:** White coloured, round shaped, film coated tablets, plain on both sides.

2) **Identification:**
The retention time of the principal peak in the chromatogram of sample preparation should correspond to that of the standard preparation as obtained in the "Assay".

3) **Average weight:**

TABLETS-1	:	285	TABLETS-6	:	285
TABLETS-2	:	285	TABLETS-7	:	292
TABLETS-3	:	286	TABLETS-8	:	285
TABLETS-4	:	282	TABLETS-9	:	280
TABLETS-5	:	279	TABLETS-10	:	295

AVERAGE WEIGHT:- 286.8 mg **LIMIT: 283 MG ± 7.5%**

) **Uniformity of Weight:**
Select randomly 10 tablets and weigh individual tablet. Calculate average, the minimum and maximum value.

TABLETS-1	:	285	TABLETS-6	:	285
TABLETS-2	:	285	TABLETS-7	:	292
TABLETS-3	:	286	TABLETS-8	:	285
TABLETS-4	:	282	TABLETS-9	:	280
TABLETS-5	:	279	TABLETS-10	:	295

MINIMUM WEIGHT :-
MAXIMUM WEIGHT :-

LIMIT: 283 MG ±7.5% (BETWEEN 261.8 MG AND 304.2 MG)

5) Dissolution (By HPLC):

Dissolution Parameters :					
Medium	:	Water	Rotatory Speed	:	75 RPM
Volume	:	900ml	Temperature	:	37°C ± 0.5°C
Apparatus	:	USP Type-II (paddle)	Time	:	45 minutes

Standard Weight :-

Potency:-

Calculations: Teneligliptin (% Drug Release) $= \frac{At}{As} \times \frac{Ws}{100} \times \frac{900}{LC} \times \frac{P}{100} \times \frac{426.57}{628.86} \times 100$

Tablet 1= $\frac{0.6045}{0.6642} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 89.95\%$

Tablet 2= $\frac{0.5994}{0.6642} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 89.19\%$

Tablet 3= $\frac{0.6237}{0.6642} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 92.81\%$

Tablet 4= $\frac{0.5951}{0.6642} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 88.55\%$

Tablet 5= $\frac{0.5808}{0.6642} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 86.43\%$

Tablet 6= $\frac{0.6084}{0.6642} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 90.53\%$

Average:- $\frac{89.95 + 89.19 + 92.81 + 88.55 + 86.43 + 90.53}{6} = 89.57\%$

Limits: Not less than 80.00 % of labeled amount is dissolved in 45 minutes

ARN ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG | PAGE NO.:- Page 4 of 4

6) Assay (By HPLC):

Chromatographic Conditions:

Column	:	C18 , (150 mm × 4.6 mm), 5 µm
Pump mode	:	Isocratic
Mobile Phase	:	Buffer:- Acetonitrile (65:35)
Flow rate	:	1.00 mL /min
Injection volume	:	20 µl
Column Temperature	:	30°C
Wavelength	:	1.5 times of retention time of principle peak

Preparation of solutions:

- **Standard preparation:**

Dissolved 0.1M potassium dihydrogen orthophosphate in 300 ml of water. Prepare a mix. of buffer, acetonitrile (65 : 30 v/v). filter through 0.45 µ filter & degas

- **Sample preparation:**

Standard Weight :- 20 mg
Sample Weight :- 286.8 mg
Average Weight :- 286.8 mg
Potency :- 99.85%

- **Calculations:**

$$\% \text{ of Teneligliptin} = \frac{At}{As} \times \frac{Ws}{100} \times \frac{100}{Wt} \times \frac{P}{100} \times Aw \times \frac{426.57}{628.86} \times \frac{100}{LC}$$

1) $$\frac{1196080}{1135986} \times \frac{29.43}{100} \times \frac{100}{286.8} \times \frac{P}{100} \times 286.8 \times \frac{426.57}{628.86} \times \frac{100}{20}$$

$= 104.93\%$

2) $$\frac{1194752}{1135986} \times \frac{29.43}{100} \times \frac{100}{286.8} \times \frac{P}{100} \times 286.8 \times \frac{426.57}{628.86} \times \frac{100}{20}$$

$= 105.92\%$

Average :-

Limit: Not less than 90.00 % and not more than 110.00 % of the label claim

	ARNI ANALYTICALS	
TITLE	HPLC DATA SHEET	
Instrument Name :-	HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHI	1 of 1
Instrument ID : -	ARNI / INS- 001	

DATA SHEET

NAME OF TEST :- SYSTEM SUITABILITY

SYSTEM SUITIBILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINCE

- **Chromatographic Conditions:**

Column	A stainless steel column **Dimentions :-** **Length** :- 15 cm × **Diameter:-** 4.6 mm ; **Particle size** :-5µm **Length** :- 150 mm × **Diameter:-** 4.6 mm ; **Particle size** :-5µm **Stationary Phase** :- Packed with octadecylsilyl (C18) silica gel
Data Aquisitation Time	7 Minutes
Pump (Flow Rate)	1.00 ml/min
Port	A
Detector (Wavelength)	273nm
Column Oven Temperature	30°C
Degasser	Off
Autosampler Temperature	Off

- **MOBILE PHASE PREPARATION :-**

Prepare a Mixture of ~~80~~ 70 volumes of Water and ~~20~~ 30 volumes of ~~Methanol~~ Acetonitrile. Mix well.

2/0 90

- **STANDARD PREPARATION :-**

Weigh accurately 20mg of Caffeine standard to a 100ml volumetric flask. Add 60ml of HPLC grade water and shake to dissolve completely. Slowly makeup the volume upto the mark. Mix well. Further dilute 5ml of the above solution to 50ml volumetric flask, dilute with water to makeup volume.

	ARNI ANALYTICALS	
TITLE	HPLC DATA SHEET	
Instrument Name :-		Page No
Instrument Make :-		
Instrument Model No. :-		1 of 1
Instrument ID : -		

NAME OF STUDENT :- Tungar Aniket Shivaji

DATA SHEET

NAME OF TEST :- SYSTEM SUITABILITY

SYSTEM SUITIBILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINCE

- **Chromatographic Conditions:**

Column	A stainless steel column Dimensions: Length :- 15cm x dia :- 4.6mm particle size :- 5um
Data Aquisitation Time	7 minutes
Pump (Flow Rate)	1.00 ml /min
Port	A
Detector (Wavelength)	273 nm
Column Oven Temperature	30°C
Degasser	Off
Autosampler Temperature	Off

- **MOBILE PHASE PREPARATION :-**

  Prepared a mixture of 70 volumes of water and 20 volumes of Acetonitrile and mixed well.

- **STANDARD PREPARATION :-**

  Weigh accurately 20mg of caffiene standard to 100 ml volumetric flask. Add 60 ml HPLC grade water and shake it. Make up the volume & further dilute 5ml of above solution to 5ml volumetric flask & dilute to make up the volume

- **SEQUENCE OF INJECTION :-**

Name of Solution	No. Of Injection
Blank	1
Standard	2

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	ARNI ANALYTICALS	
TITLE	HPLC DATA SHEET	
Instrument Name :-	High Performance Liquid Chromatography	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2d0 CHI	1 of 3
Instrument ID : -	ARNI/INS-001	
Name Of Student :-	Tungar Aniket Shivaji	

HPLC DATA SHEET

- **HPLC Parameter settings:**
  - **Make a purging of the mobile phase of all ports & injection port to remove the air bubble from the line.**
  - **Create a new method by using below parameter.**
  - **Save the Method Parameters with a file name.**
  - **Download the method to the instruments.**

- **CHROMATOGRAPHIC PARAMETERS-1**

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisitation Time	:	13 min
Pump	:	0.80 ml/min
Port	:	C
Detector (Wavelength)	:	222 nm
Column Oven Temperature	:	40 °C
Degasser	:	OFF
Autosampler Temperature	:	7° C

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TITLE	HPLC DATA SHEET	
Instrument Name :-		Page No
Instrument Make :-		
Instrument Model No. :-		2 of 3
Instrument ID : -		
Name Of Student :-		

- **CHROMATOGRAPHIC PARAMETERS-2**

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisitation Time	:	22 min
Pump	:	1.20 ml/min
Port	:	A
Detector (Wavelength)	:	260 nm
Column Oven Temperature	:	30°C
Degasser	:	OFF
Autosampler Temperature	:	15°C

- **CHROMATOGRAPHIC PARAMETERS-3**

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisitation Time	:	30 min
Pump	:	1.50 ml/min
Port	:	B
Detector (Wavelength)	:	260 nm
Column Oven Temperature	:	30°C
Degasser	:	OFF
Autosampler Temperature	:	15°C

ANALYSED BY

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	ARNI ANALYTICALS	
TITLE	HPLC DATA SHEET	
Instrument Name :-		Page No
Instrument Make :-		
Instrument Model No. :-		3 of 3
Instrument ID : -		
Name Of Student :-		

- **CHROMATOGRAPHIC PARAMETERS-4**

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisitation Time	:	10 min
Pump	:	1.00 mL/min
Port	:	A
Detector (Wavelength)	:	210 nm
Column Oven Temperature	:	30°C
Degasser	:	ON
Autosampler Temperature	:	10 °C

- **CHROMATOGRAPHIC PARAMETERS-5**

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisitation Time	:	20 min
Pump	:	0.00 ml/min
Port	:	A
Detector (Wavelength)	:	OFF
Column Oven Temperature	:	OFF
Degasser	:	OFF
Autosampler Temperature	:	OFF

ANALYSED BY

CHECKED BY

SPECIFICATION AND TESTS OF TENELIGLIPTIN TABLETS 20 MG

Sr. No.	Tests	Specification
1	Description	Yellow coloured, round shaped, film coated tablets, plain on both sides.
2	Identification	The retention time of the major peak in the chromatogram of assay preparation should correspond to that in the chromatogram of the standard preparation, as obtained in the "Assay".
3	Average weight of Tablet	283 mg ± 7.5%
4	Uniformity of weight	283 mg ± 7.5% (Between 261.8 mg and 304.2 mg)
5	Dissolution	Not less than 80.00 % of labeled amount is dissolved in 45 minutes
6	Assay	Not less than 90.00% and Not more than 110.00% of Label Claim (Between 18.00 mg and 22.00 mg per tablet)

TEST METHOD

1) **Description:** White coloured, round shaped, film coated tablets, plain on both sides.

2) **Identification:**
The retention time of the principal peak in the chromatogram of sample preparation should correspond to that of the standard preparation as obtained in the "Assay".

3) **Average weight:**
Weigh together 10 tablets selected at random and calculate the average weight.

Calculation:

$$\text{Average weight (mg)} = \frac{W}{10}$$

Where, W= Weight of 10 tablets in mg

Limit: 283 mg ± 7.5%

4) **Uniformity of Weight:**
Select randomly 10 tablets and weigh individual tablet. Calculate average, the minimum and maximum value.

Limit: 283 mg ±7.5% (Between 261.8 mg and 304.2 mg)

5) Dissolution (By HPLC):

Dissolution Parameters :					
Medium	:	Water	**Rotatory Speed**	:	75 rpm
Volume	:	900 mL	**Temperature**	:	37°C ± 0.5°C
Apparatus	:	USP Type II (Paddle)	**Time**	:	45 Minutes

11.1 Preparation of Solutions :

Standard preparation :

Weigh and transfer accurately about 22 mg of Teneligliptin (Equivalent to 32.43 mg Teneligliptin Hydrobromide Hydrate) working standard to a 100 mL volumetric flask add 70 mL of water and sonicate to dissolve and make up the volume with water. 5/50

- **Sample preparation:**

Pour 900 mL of dissolution medium in each vessel. Allow sufficient time for the dissolution medium to equilibrate at 37°C ± 0.5°C. Adjust stirring element speed to 75 rpm. Place one tablet in each of the six vessels and immerse the paddles in the dissolution medium so that there is a distance of 25mm ± 2mm between the bottom of the paddle and inside bottom of the vessel. Start the apparatus.

At the end of specified time intervals (after 45 minutes), withdraw 10 mL aliquot from a zone midway between the surface of the dissolution medium and the top of the rotating paddle and filter through 0.45µ filter paper discarding first few mL of the filtrate. Inject directly.

11.2 Procedure:

Measure the absorbance of the resulting solution at 210nm.

Calculations:

Teneligliptin

$$(\%\ \text{Drug Release}) = \frac{At}{As} \times \frac{Ws}{100} \times \frac{900}{LC} \times \frac{P}{100} \times \frac{426.57}{628.86} \times 100$$

Where,

A_T	=	Absorbance due to Teneligliptin in the sample preparation.
A_S	=	Absorbance due to Teneligliptin in the standard preparation.
W_S	=	Weight of Teneligliptin hydrobromide hydatre working standard taken for standard preparation, in mg.
P	=	Purity of Teneligliptin hydrobromide hydatre working standard used for standard
LC	=	Label claim of a tablet, in mg.
426.57	=	Molecular weight of Teneligliptin
628.86	=	Molecular weight of Teneligliptin hydrobromide hydatre

Limits: Not less than 80.00 % of labeled amount is dissolved in 45 minutes

13) Assay (By HPLC):

- **Reagents Required:**

Sr.No.	Name of Reagent	Grade
1	Water	HPLC grade
2	Acetonitrile	HPLC grade
3	Octane-1-sulphonic acid sodium salt	AR grade
4	O-Phosphoric acid	AR grade

13.1 Chromatographic Conditions:

Column	:	C18, (150 mm X 4.6 mm), 5µm
Pump mode	:	Isocratic
Mobile Phase	:	Buffer : Acetonitrile (60:40)
Flow rate	:	1.0 mL/min
Injection volume	:	20 µl
Column Temperature	:	30°C
Wavelength	:	UV, 210 nm
Run time	:	1.5 times of the retention time of principle peak

13.2 Preparation of Mobile Phase:

- **Preparation of Buffer:** 2.6g 195ml 240ml 195 - 105

Dissolved 0.1M Potassium dihydrogen orthophosphate in 1000 mL of water;

Prepare a mixture of Buffer, Acetonitrile (60:40 v/v), filter through 0.45 µ filter and degas.

13.3 Preparation of solutions: 105ml 65-35 80-20 240-60

- **Standard preparation:**

Weigh and transfer accurately about 20 mg of Teneligliptin (29.48 mg Teneligliptin Hydrobromide Hydrate) working standard to a 100 mL volumetric flask add 70 mL of water and sonicate to dissolve and make up the volume with water.

- **Sample preparation:**

Weigh 10 tablets and determine average weight. Crush the tablets to a fine powder. Weigh and transfer powder equivalent to 20 mg of Teneligliptin to a 100 mL dry volumetric flask. Add 70 mL of water, sonicate for not less than 20 minutes with intermittent shaking. Make up the volume with water. Filter through 0.45 µ Nylon filter discarding first few mL of the filtrate.

13.4 Evaluation of System Suitability:

Equilibrate the column with mobile phase with the chromatographic conditions for stable baseline. Inject blank and record the chromatogram. Inject standard preparation in five replicates and record the chromatograms. It should comply with the system suitability criteria as mentioned.

- Theoretical plate for Teneligliptin peak should not be less than 2000
- The relative standard deviation for area of Teneligliptin peak should not be more than 2.0 %
- The tailing factor for Teneligliptin should not be more than 2.0.

13.5 Procedure:

Inject sample preparation in duplicate and record the chromatogram. Inhibit the integration due to blank peak in the chromatogram of sample preparation.

- **Calculations:**

$$\text{mg/tab of Teneligliptin} = \frac{At}{As} \times \frac{Ws}{100} \times \frac{100}{Wt} \times \frac{P}{100} \times Aw \times \frac{426.57}{628.86} \times \frac{100}{LC}$$

Where,

A_T	=	Area of the peak due to Teneligliptin obtained in the chromatogram of sample preparatior
A_S	=	Mean area of the peak due to Teneligliptin obtained in the chromatogram of standard preparation.
W_S	=	Weight of Teneligliptin hydrobromide hydatre working standard taken for standard preparation, in mg.
P	=	Purity of Teneligliptin hydrobromide hydatre working standard, on as is basis.
LC	=	Label claim in mg.
A_W	=	Average weight in mg.
W_T	=	Weight of sample taken for sample preparation, in mg.
426.57	=	Molecular weight of Teneligliptin.
628.86	=	Molecular weight of Teneligliptin Hydrobromide Hydrate

Limit: Not less than 90.00 % and not more than 110.00 % of the label claim