

„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

Miss. Nikita Angad Pandey

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 **Miss. Nikita Angad Pandey** 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.

Gaware
HOD Chemistry
HEAD

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

P. J. Jadhav
2-05-2023
Examiner

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



ARNI ANALYTICAL

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



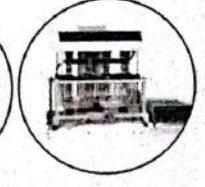
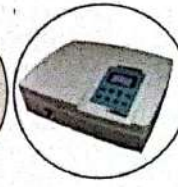
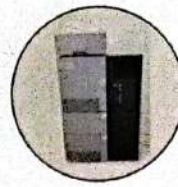
Certificate

This is to Certify that *Nikita Angad Pandey*
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. **Nikita Angad Pandey** 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 “**A**” grade.

Mr. Masum Deshmukh
Director

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:-



Name:- Nikita Angad Pandey

INDEX

Sr.No	Description	Page No.
1	Specification and Tests of Teneiglipatin Tablets 20 mg	1
2	HPLC Data Sheet	9
3	Monthly calibration record of analytical balance	14
4	Daily calibration record of pH- Meter	20
5	Dissolution Test Apparatus Worksheet	22
6	UV- Spectrophotometer Worksheet	24

TENELIGLIPTIN

Introduction-

- Teneligliptin is a pharmaceutical drug for the treatment of type-2 diabetes mellitus.
- 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 hormone '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- C₂₂H₃₀N₆O₅

Molar Mass- 426.58 gm/mol

- Teneligliptin significantly controls glycemc 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 excipients.
- 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

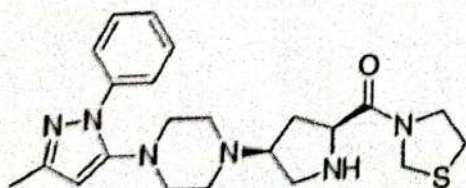
Physical Testing-

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-





ARN ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 1 of 4

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 \pm 7.5%
4	Uniformity of weight	283 mg \pm 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)

Area - STD - ① 14504216

② 14631085

③ 17812119

1st tray 41
61

Sample - ① 12137379

② 10902060

③ 10958003

④ 12445266

15649140



ARNI ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 2 of 5

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)



ARNI ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 3 of 5

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.

• 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

$$(\% \text{ Drug Release}) = \frac{A_t}{A_s} \times \frac{W_s}{100} \times \frac{900}{LC} \times \frac{P}{100} \times \frac{426.57}{628.86} \times 100$$

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 hydrate working standard taken for standard preparation, in mg.

P = Purity of Teneligliptin hydrobromide hydrate 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 hydrate

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



ARNI ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 4 of 5

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

65 : 35
 195 ml, 105 ml
 Buff. Aceton

13.2 Preparation of Mobile Phase:

1M = 136.09 = 1000 0.1M = 13.609 = 1000

• Preparation of Buffer:

Dissolved 0.1M Potassium dihydrogen orthophosphate in 1000 mL of water; $0.1 = 195$
 Prepare a mixture of Buffer, Acetonitrile (60:40 v/v), filter through 0.45 µ filter and degas. 2.653 gm

13.3 Preparation of solutions:

• 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.

A.V.
 $Eq = 20 \text{ mg}$ (Weight of 1 tablet)

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.

13.609 = 1000
 $x = 195$



ARNI ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 5 of 5

- 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:

$$\text{mg/tab of Teneligliptin} = \frac{A_t}{A_s} \times \frac{W_s}{100} \times \frac{100}{W_t} \times \frac{P}{100} \times A_w \times \frac{426.57}{628.86} \times \frac{100}{LC}$$

Where,

A_T = Area of the peak due to Teneligliptin obtained in the chromatogram of sample preparation.

A_S = Mean area of the peak due to Teneligliptin obtained in the chromatogram of standard preparation.

W_S = Weight of Teneligliptin hydrobromide hydrate working standard taken for standard preparation, in mg.

P = Purity of Teneligliptin hydrobromide hydrate 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

Example:

Dissolutⁿ.

500 mg → 900 ml.

Sample.

$$\frac{500}{900} \times \frac{2}{50} = 22.2 \text{ ppm}$$

- Abs = 1) 0.478
- 2) 0.473
- 3) 0.485
- 4) 0.457
- 5) 0.469
- 6) 0.472.

Stand.

$$\frac{22}{100} \times \frac{5}{50} = 22 \text{ ppm}$$

Absorbance = 0.476.

Formula:-

$$\text{①} \frac{\text{Sam. Abs}}{\text{Stand. Abs}} \times \frac{\text{Std. wt}}{\text{dil}^n} \times \frac{\text{dil}^n}{\text{L.C}} \times \text{dil}^n \times \frac{P}{100} \times 100.$$

$$= \frac{0.478}{0.476} \times \frac{22.5}{100} \times \frac{5}{50} \times \frac{900}{500} \times \frac{50}{2} \times \frac{99.5}{100} \times 100$$

$$= 101.16 \%$$

$$\text{②} \frac{0.473}{0.476} \times \frac{22.5}{100} \times \frac{5}{50} \times \frac{900}{500} \times \frac{50}{2} \times \frac{99.5}{100} \times 100.$$

$$= 100.1 \%$$

$$\text{③} \frac{0.485}{0.476} \times \frac{22.5}{100} \times \frac{5}{50} \times \frac{900}{500} \times \frac{50}{2} \times \frac{99.5}{100} \times 100.$$

$$= 102.6 \%$$

$$\text{④} \frac{0.457}{0.476} \times \frac{22.5}{100} \times \frac{5}{50} \times \frac{900}{500} \times \frac{50}{2} \times \frac{99.5}{100} \times 100$$

$$= 96.72 \%$$

$$\text{⑤} \frac{0.469}{0.476} \times \frac{22.5}{100} \times \frac{5}{50} \times \frac{900}{500} \times \frac{50}{2} \times \frac{99.5}{100} \times 100$$

$$= 99.26 \%$$

Calculations..

① for Raw Material.

Ex.	standard	sample.
	10.3mg	10mg
	100ml	100ml.
	Abs:- 0.576	0.570

formula.

$$= \frac{\text{Sample Abs}}{\text{Stand. Abs}} \times \frac{\text{Std. wt}}{100} \times \frac{100}{\text{Sample wt}} \times \frac{\text{Potency}}{100} \times 100.$$

$$= \frac{0.570}{0.576} \times \frac{10.3}{100} \times \frac{100}{10} \times \frac{99.5}{100} \times 100.$$

$$= 101.39\%$$

② for finished.

Ex. Paracetamol :-

Paxa - 500 mg

1 - 100

2 - 50

3 - 100

750 mg.

20 tablets

↓ average
wt.

752.3 mg.

Std. ⇒ 50.2 mg.

↓
100 ml

5 ml → 50 ml

Sample.

500mg → 100 ml

1 → 100 ml

concⁿ = 50 ppm

Abs = 0.379.

Concⁿ = 50 ppm

Abs = 0.382

formula.

$$= \frac{\text{Sample Abs}}{\text{Stand Abs}} \times \frac{\text{Std wt}}{100} \times \frac{5}{50} \times \frac{100}{\text{Sam. wt}} \times \frac{100}{1} \times \text{Av. wt} \times \frac{P}{100} \times \frac{100}{\text{L.C.}}$$

$$= \frac{0.379}{0.382} \times \frac{50.2}{100} \times \frac{5}{50} \times \frac{100}{752.3} \times \frac{100}{1} \times 752.3 \times \frac{99.5}{100} \times \frac{100}{500}$$

$$= 99.06\%$$



ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	High Performance liquid chromatography	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	1 of 3
Instrument ID :-	ARNI/INS-001	
Name Of Student :-	Pandey Nikita Angad.	

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 Aquisition 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

Pandey
ANALYSED BY

K. Singh
CHECKED BY



ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	Page No
Instrument Name :-	HPLC	
Instrument Make :-	SHIMADZU	2 of 3
Instrument Model No. :-	LC 2010 CHT	
Instrument ID :-	ARNI/INS -001	
Name Of Student :-	Pandey Nikita Angad .	

• CHROMATOGRAPHIC PARAMETERS-2

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

• CHROMATOGRAPHIC PARAMETERS-3

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

Pandey
ANALYSED BY

J. K. Das
CHECKED BY



ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	HPLC	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC2010 CHT	3 of 3
Instrument ID :-	ARNI/INS-001	
Name Of Student :-	Pandey Nikita Angad	

• CHROMATOGRAPHIC PARAMETERS-4

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

• CHROMATOGRAPHIC PARAMETERS-5

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

Pandey
ANALYSED BY

Makrop
CHECKED BY



ARNI ANALYTICALS

TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-	ANALYTICAL BALANCE	Page No
Instrument Make :-	WENSAR	
Instrument ID :-	ARNI / INS - 004	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	199.986 g	199.8000 to 200.2000
2	100.0000	98.750 g	99.9000 to 100.1000
3	50.0000	50.689 g	49.9500 to 50.0500
4	20.0000	19.048 g	19.9800 to 20.0200
5	10.0000	9.996 g	9.9900 to 10.0100
6	5.0000	5.080 g	4.9950 to 5.0050
7	2.0000	1.326 g	1.9980 to 2.0020
8	1.0000	0.923 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	0.069 g	0.0499 to 0.0501
13	0.0200	0.016 g	0.0199 to 0.0200
14	0.0100	0.015 g	0.0099 to 0.0100
15	0.0050	0.000 g	0.0049 to 0.0051

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

Wander
ANALYSED BY

[Signature]
CHECKED BY



ARNI ANALYTICALS

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

2. Test for Linearity:

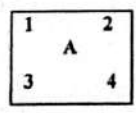
Sr. No.	Selected Weights in g	Observed Weight in g
1	200	199.986 g
2	20	19.049 g
3	5	5.080 g

Conclusion: The observed weights are Consistent/not Consistent.

S. Pandey
ANALYSED BY

M. B. B.
CHECKED BY

3. Test for Eccentricity:



Sr. No.	Weight Observed in g	Difference in g	Limit
1.	At Centre- (A) 5.082 g	5.082 <u>5.082</u> <u>S. Pandey 15/12/22</u>	± 0.1 %
2.	At Corner 1 (B) 5.082	B-A = 0.000	
3.	At Corner 2 (C) 5.082	C-A = 0.000	
4.	At Corner 3 (D) 5.081	D-A = 0.001	
5.	At Corner 4 (E) 5.081	E-A = 0.001	

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

S. Pandey
ANALYSED BY

M. B. B.
CHECKED BY



ARNI ANALYTICALS

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

4. Test for Repeatability :

Selected Weight in g: 5 gm

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	5.081	6	5.081	± 0.1 %
2	5.082	7	5.082	
3	5.081	8	5.080	
4	5.082	9	5.080	
5	5.081	10	5.081	

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

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

Handey.
ANALYSED BY

Handey.
CHECKED BY



ARNI ANALYTICALS

TITLE

DAILY CALIBRATION RECORD OF pH-METER

Instrument Name :-

PH-meter

Page No

Instrument Make :-

LABMAN

Instrument Model No. :-

LMIPH-10

1 of 1

Instrument ID :-

ARNI/INS-005

DAILY CALIBRATION RECORD

Procedure: Refer SOP No. : SOP/ARN/INS-005

Preparation Of Solutions:

pH-4.01 :-

- Transfer the capsule content in a 100ml volumetric flask using a funnel.
- Dissolve the contents in 10 ml of distilled water and then make it up to 100 ml with distilled water.
- This solution will have a pH of 4.0 ± 0.05 at 25°C .

pH-7.00 :-

- Transfer the capsule content in a 100ml volumetric flask using a funnel.
- Dissolve the contents in 10 ml of distilled water and then make it up to 100 ml with distilled water.
- This solution will have a pH of 7.0 ± 0.05 at 25°C .

pH-9.20 :-

- Transfer the capsule content in a 100ml volumetric flask using a funnel.
- Dissolve the contents in 10 ml of distilled water and then make it up to 100 ml with distilled water.
- This solution will have a pH of 9.20 ± 0.05 at 25°C .

Observation Table:

Sr. No.	Date	pH	
		4.00 (± 0.05)	7.00 (± 0.05)
1	11/12/22	4.02	6.78

Slope = 87 %

Mandey
PERFORMED BY

Mandey
CHECKED BY



ARNI ANALYTICALS

TITLE

MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE

Instrument Name :-

ANALYTICAL BALANCE

Page No.

Instrument Make :-

LIENSAR

Instrument Model No. :-

DS 8000

1 of 3

Instrument ID :-

ARNI/INS-004

NAME OF STUDENT:- Pandey Nikita Angad

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: $\pm 0.1\%$)
1	200.0000	200.011	199.8000 to 200.2000
2	100.0000	98.770	99.9000 to 100.1000
3	50.0000	50.703	49.9500 to 50.0500
4	20.0000	19.055	19.9800 to 20.0200
5	10.0000	10.001	9.9900 to 10.0100
6	5.0000	5.085	4.9950 to 5.0050
7	2.0000	1.327	1.9980 to 2.0020
8	1.0000	0.926	0.9990 to 1.0010
9	0.5000	0.151	0.4995 to 0.5005
10	0.2000	0.105	0.1998 to 0.2002
11	0.1000	0.056	0.0999 to 0.1001
12	0.0500	0.036	0.0499 to 0.0501
13	0.0200	0.014	0.0199 to 0.0200
14	0.0100	0.016	0.0099 to 0.0100
15	0.0050	0.000	0.0049 to 0.0051

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

S. Pandey
ANALYSED BY

M. Singh
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 :-

ARNJ/7NS-004

2. Test for Linearity:

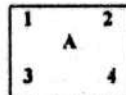
Sr. No.	Selected Weights in g	Observed Weight in g
1	200	200.0012
2	100	98.773
3	50	50.702

Conclusion: The observed weights are Consistent/not Consistent.

Handwritten Signature
ANALYSED BY

Handwritten Signature
CHECKED BY:-

3. Test for Eccentricity:



Sr. No.	Weight Observed in g	Difference in g	Limit
1.	At Centre- (A) 5.083		± 0.1 %
2.	At Corner 1 (B) 5.082	B-A = -0.001	
3.	At Corner 2 (C) 5.082	C-A = -0.001	
4.	At Corner 3 (D) 5.083	D-A = 0.000	
5.	At Corner 4 (E) 5.082	E-A = -0.001	

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

Handwritten Signature
ANALYSED BY

Handwritten Signature
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/JNS-004

4. Test for Repeatability :

Selected Weight in g: 5g

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	5.079	6	5.082	± 0.1 %
2	5.080	7	5.082	
3	5.082	8	5.083	
4	5.082	9	5.082	
5	5.081	10	5.082	

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

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

Nandey
ANALYSED BY

Mishra
CHECKED BY:-

ARNI ANALYTICALS

TITLE	DAILY CALIBRATION RECORD OF pH-METER	
Instrument Name :-	PH - METER	Page No
Instrument Make :-	LABMAN	1 of 1
Instrument Model No. :-	LMPH-10	
Instrument ID :-	ARNI/INS-005	

DAILY CALIBRATION RECORD

- **Procedure: Refer SOP No. : SOP/ARN/INS-005**
- **Preparation Of Solutions:**

- **pH-4.01 :-**
 - Transfer the capsule content in a 100ml volumetric flask using a funnel.
 - Dissolve the contents in 10 ml of distilled water and then make it up to 100 ml with distilled water.
 - This solution will have a pH of 4.0 ± 0.05 at 25°C .
- **pH-7.00 :-**
 - Transfer the capsule content in a 100ml volumetric flask using a funnel.
 - Dissolve the contents in 10 ml of distilled water and then make it up to 100 ml with distilled water.
 - This solution will have a pH of 7.0 ± 0.05 at 25°C .
- **pH-9.20 :-**
 - Transfer the capsule content in a 100ml volumetric flask using a funnel.
 - Dissolve the contents in 10 ml of distilled water and then make it up to 100 ml with distilled water.
 - This solution will have a pH of 9.20 ± 0.05 at 25°C .

- **Observation Table:**

Sr. No.	Date	pH	
		4.00 (± 0.05)	7.00 (± 0.05)
1	15/12/22	3.90	6.80

Slope = 97% .

Pandey
PERFORMED BY

Mishra
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	Pandey Nikita Angad.	

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.

Pandey
ANALYSED BY

[Signature]
CHECKED BY:-

ARNI ANALYTICALS

TITLE	DISSOLUTION TEST APPARATUS WORKSHEET	
Instrument Name :-	Dissolution Test Apparatus	Page No.
Instrument ID :-	ARN21 JNS-003	
Instrument Model No. :-	DS-8000	1 of 1
Name Of Students	Pandey Nikita Angad	

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°C
Time	45 minutes

PREPARATIONS:-

Pour 900ml of dissolution medium in each bowl.
 Maintain the temperature of water bath at 37.5°C
 Adjust the stirring speed at 100 RPM.
 Adjust the paddle with shaft in dissolution medium
 Adjust the distance between paddle and inside bottom of the bowl. Start the Apparatus. End of specific time interval withdraw 10 ml aliquot from dissolution medium.
 further dilute 2 ml of solution to 25 ml with dissolution medium.

Pandey
ANALYSED BY

Nikita
CHECKED BY:-

ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	High Performance liquid chromatograph	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	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 Aquisition 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 volumes of Water and 20 volumes of Methanol. Mix well.

• **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 :-	High Performance liquid chromatogr	Page No 1 of 1
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	
Instrument ID :-	ARNI/INS-001	

NAME OF STUDENT :- Pandey Nikita Angad .

DATA SHEET

NAME OF TEST :- SYSTEM SUITABILITY

SYSTEM SUITIBILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINCE

• **Chromatographic Conditions:**

Column	
Data Aquisition Time	10 min
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 :-**

Mixture of water & Ethanol ^{Aceto nitrile} prepare in proportion of 7:3 ^{S.Pandey .15/12/22}

• **STANDARD PREPARATION :-**

Weigh accurately 20mg of caffeine standard to a 100ml Volumetric flask. Add 60ml of HPLC grade water & 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.

• **SEQUENCE OF INJECTION :-**

Name of Solution	No. Of Injection
Blank	1
Standard	3

S.Pandey
ANALYSED BY

M. B. Singh
CHECKED BY



ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV - SPECTROPHOTOMETER	Page No.
Instrument ID :-	ARNI/INS-002	
Instrument Model No. :-	LMSP-W1008	1 of 1
Name Of Students	Pandey Nibita Angad	

NAME OF TEST :-

- 1) PHOTOMETRIC ANALYSIS
- 2) WAVELENGTH SCAN

REPARATIONS:-

STANDARD PREPARATION :-

Weigh accurately 10mg of Caffeine standard in a 100ml volumetric flask, add 60ml of water sonicate for 5 minutes to completely dissolve, makeup the volume with water.
Further dilute 5ml of the above solution to 50ml with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- 273nm

Pandey
ANALYSED BY

[Signature]
CHECKED BY:-



ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV-SPECTROPHOTOMETER	Page No.
Instrument ID :-	ARNI/INS-002	
Instrument Model No. :-	LMSP - UV100B	1 of 1
Name Of Students	Pandey Nikita Angad.	

Date:- 17/12/22

NAME OF PRODUCT	:	Caffeine
WORKING STANDARD NO.	:	-
POTENCY	:	-
INSTRUMENT ID	:	ARNI/INS-002.

NAME OF TEST :- Wavelength scan.

PREPARATIONS:-

STANDARD PREPARATION :-

weigh accurately 10mg of caffeine standard in 100 ml volumetric flask, add 60ml of water sonicate for 5 min to completely dissolve, makeup the volume with water. further dilute 5ml of the above solution to 50 ml with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- 274 nm - maximum
206 nm - minimum

OBSERVATIONS:-

MAXIMUM ABSORPTION WAVELENGTH - 274nm

N. Pandey
ANALYSED BY

N. Pandey
CHECKED BY:-



ARN ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 2 of 4

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

AVERAGE WEIGHT:- 285.4 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.

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

MINIMUM WEIGHT :- 279 mg

MAXIMUM WEIGHT :- 295 mg

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



ARN ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 3 of 4

5) Dissolution (By HPLC):

Dissolution Parameters :

Medium	:	Water	Rotatory Speed	:	75 rpm
Volume	:	900ml	Temperature	:	37°C
Apparatus	:	USP Type II	Time	:	45 Minutes

Standard Weight :- 32.43 mg

Potency:- 99.85 %

Calculations: Teneligliptin (% Drug Release) = $\frac{A_t}{A_s} \times \frac{W_s}{100} \times \frac{900}{LC} \times \frac{P}{100} \times \frac{426.57}{628.86} \times 100$

Tablet 1 = $\frac{0.6682}{0.6642} \times \frac{32.43}{100} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 \times \frac{5}{50} = 99.43\%$

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

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

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 = $\frac{0.6089}{0.6642} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 90.53\%$

91.15%

Average:- 91.15%

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, (150mm x 4.6 mm), 5 μm
Pump mode	: Isocratic
Mobile Phase	: Buffer: Acetonitrile (65:35)
Flow rate	: 1.0 ml/min
Injection volume	: 20 μl
Column Temperature	: 30 °C
Wavelength	: 210 nm

Preparation of solutions:

• Standard preparation:

20 mg of Teneligliptin (29.48 mg Teneligliptin Hydrobromide hydrate) dissolve in 100ml volumetric flask.

• Sample preparation:

Weigh 10 tablets & determine average weight. Crush the tablets to a fine powder. weigh & transfer powder eqⁿ to 20 mg of Teneligliptin to 100ml volumetric flask. filter through 0.45 μ Nylon filter discarding first few ml of filtrate.

- Standard Weight :- 29.48
- Sample Weight :- 286
- Average Weight :- 286.9
- Potency :- 99.85%

• Calculations:

$$\% \text{ of Teneligliptin} = \frac{A_t}{A_s} \times \frac{W_s}{100} \times \frac{100}{W_t} \times \frac{P}{100} \times A_w \times \frac{426.57}{628.86} \times \frac{100}{LC}$$

$$1) \frac{11452512}{14939435} \times \frac{29.48}{100} \times \frac{100}{286} \times \frac{99.85}{100} \times 286.9 \times \frac{426.57}{628.86} \times \frac{100}{20} = 76.77\%$$

$$2) \frac{12414022}{14939435} \times \frac{29.48}{100} \times \frac{100}{286} \times \frac{99.85}{100} \times 286.9 \times \frac{426.57}{628.86} \times \frac{100}{20} = 83.21\%$$

Average :- 79.99%

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