„"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. Sanjivani Ganesh Dube

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. Sanjivani Ganesh Dube** 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.

Bawane

HOD Chemistry

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

12-05-2023

Examiner

Principal

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

ISO 9001 CERTIFIED

ARNI ANALYTICAL

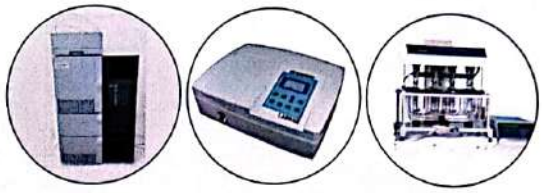
Add.: Pushpak Apartment, Flat No. 102, Lane No. 3, Near Neurocare Hospital, Pandit Colony, Nashik.
e-mail : arnianalyticals@gmail.com | Web Site : www.arnianalyticals.com

Certificate

This is to Certify that ..... *Sanjivini Ganesh Dube* .....
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. **Sanjivani Ganesh Dube** 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

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

ACKNOWLEDGEMENT

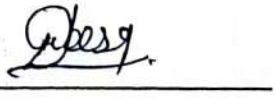
The success and final outcome of this training required a lot of guidance and assistance from many people. All that I have done is only due to such supervision and assistance and I would never forget to thank them.

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:- 

Name:- Sanjivani Ganesh Dube

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

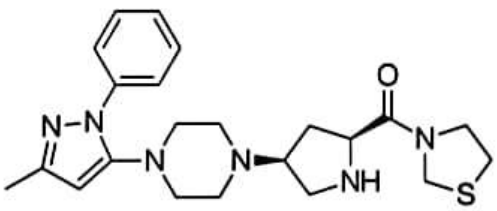
Physical Testing-

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-



- 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 preparation
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

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FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG |

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.

- **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:**

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:

- **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.

$\frac{A.wt}{10} =$

- **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.

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	:	290	TABLETS-6	:	286
TABLETS-2	:	285	TABLETS-7	:	286
TABLETS-3	:	284	TABLETS-8	:	281
TABLETS-4	:	286	TABLETS-9	:	279
TABLETS-5	:	291	TABLETS-10	:	297

AVERAGE WEIGHT:- $\frac{2866}{10} = 286$ **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	:	290	TABLETS-6	:	286
TABLETS-2	:	285	TABLETS-7	:	286
TABLETS-3	:	284	TABLETS-8	:	281
TABLETS-4	:	286	TABLETS-9	:	279
TABLETS-5	:	291	TABLETS-10	:	297

MINIMUM WEIGHT :- 281
MAXIMUM WEIGHT :- 297

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 |

5) Dissolution (By HPLC):

Dissolution Parameters :					
Medium	:	water	Rotatory Speed	:	75
Volume	:	900 mL	Temperature	:	37
Apparatus	:	USP-II (Paddle)	Time	:	45 min

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= $\frac{0.7201}{0.6774} \times \frac{82.43}{100} \times \frac{5 \times 900}{50 \quad 20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 105.07$

Tablet 2= $\frac{0.7492}{0.6774} \times \frac{82.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 109.31$

Tablet 3= $\frac{0.6778}{0.6774} \times \frac{82.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 98.90$

Tablet 4= $\frac{0.7396}{0.6774} \times \frac{82.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 107.91$

Tablet 5= $\frac{0.7459}{0.6774} \times \frac{82.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 108.83$

Tablet 6= $\frac{0.7601}{0.6774} \times \frac{82.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 110.90$

Average:- $\frac{105.07 + 109.31 + 98.90 + 107.91 + 108.83 + 110.90}{6} = \frac{640.92}{6} = 106.82$

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, C150 mm x 4.6 mm), 5 µm
Pump mode	:	Isomatic
Mobile Phase	:	Buffer: Acetonitrile (60:40)
Flow rate	:	1.0 mL/min
Injection volume	:	20 µL
Column Temperature	:	30°C
Wavelength	:	UV, 210 nm.

Preparation of solutions:

- **Standard preparation:** Wt. and transfer accurately about 20mg. of Teneligliptin Hydrobromide Hydrate) working Std. (29.48 mg Teneligliptin to a 100ml volumetric flask and 70 ml of water and sonicate to dissolve and make up the volume.
- **Sample preparation:** Wt. 10 tablet and determine average wt. crush the tablet to a fine powder. wt and transfer powder equivalent to 20mg teneligliptin to a 100 ml dry vol. flask.

Standard Weight :- 29.48
Sample Weight :- 282
Average Weight :- 282.33
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) $\frac{11688085}{14490846.5} \times \frac{29.48}{100} \times \frac{100}{282} \times \frac{99.85}{100} \times 282.33 \times \frac{426.57}{628.86} \times \frac{100}{20}$

$= 80.61\%$

2) $\frac{11546288}{14490846.5} \times \frac{29.48}{100} \times \frac{100}{282} \times \frac{99.85}{100} \times 282.33 \times \frac{426.57}{628.86} \times \frac{100}{20}$

$= 79.64\%$

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 :-	HPLC	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	1 of 3
Instrument ID : -	ARNI/ INS -001	
Name Of Student :-	Dube Sanjivani Ganesh.	

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	:	10 min
Pump	:	1.00 ml/min
Port	:	A
Detector (Wavelength)	:	210 nm
Column Oven Temperature	:	30°C
Degasser	:	On
Autosampler Temperature	:	10°C

ANALYSED BY

CHECKED BY

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

NAME OF STUDENT :- Dube Sanjivani Ganesh

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 - 15cm x Diameter - 4.6mm; particle size - 5um length - 150 mm x Diameter - 4.6mm; particle size - 5um Stationary phase - packed with octadecylsilyl (C18) silica gel.
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 :-**
  prepare a mixtuee 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 60 ml of HPLC grade water and shake to dissolve completly. Slowly makeup the volume upto the mark. Mix well. further dilute 5ml of the above soln to 50 ml volumetric flask, dilute with watee to make up volume.

- **SEQUENCE OF INJECTION :-**

Name of Solution	No. Of Injection
Blank	-
Standard	3

16/12/22

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ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	HPLC	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	2 of 3
Instrument ID : -	ARNI INS-001	
Name Of Student :-	Dube Sanjivani Ganesh.	

- **CHROMATOGRAPHIC PARAMETERS-2**

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

- **CHROMATOGRAPHIC PARAMETERS-3**

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.

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ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	HPLC	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	3 of 3
Instrument ID : -	ARNI/INS-001	
Name Of Student :-	Dube Sanjivani Ganesh.	

- **CHROMATOGRAPHIC PARAMETERS-4**

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisitation Time	:	30min
Pump	:	1.50 ml/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 Aquisitation Time	:	20 min
Pump	:	0.00 ml/min
Port	:	A
Detector (Wavelength)	:	OFF
Column Oven Temperature	:	OFF
Degasser	:	OFF
Autosampler Temperature	:	OFF

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Name- Dube Sanjivani Ganesh

ARNI ANALYTICALS

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

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	199.8000 to 200.2000
2	100.0000	98.760	99.9000 to 100.1000
3	50.0000	50.698	49.9500 to 50.0500
4	20.0000	19.052	19.9800 to 20.0200
5	10.0000	10.000	9.9900 to 10.0100
6	5.0000	5.082	4.9950 to 5.0050
7	2.0000	1.280	1.9980 to 2.0020
8	1.0000	0.923	0.9990 to 1.0010
9	0.5000	0.150	0.4995 to 0.5005
10	0.2000	0.104	0.1998 to 0.2002
11	0.1000	0.057	0.0999 to 0.1001
12	0.0500	0.037	0.0499 to 0.0501
13	0.0200	0.015	0.0199 to 0.0200
14	0.0100	0.014	0.0099 to 0.0100
15	0.0050	0.005	0.0049 to 0.0051

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

11/12/22

ANALYSED BY

CHECKED BY

Name - Dube Sanjivani Ganesh

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

2. Test for Linearity:

observed selected

Sr. No.	~~Selected~~ Weights in g	~~Observed~~ Weight in g
1	200.001	200
2	98.760	100
3	50.693	50

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

ANALYSED BY **CHECKED BY**

3. Test for Eccentricity:

1	2
A	
3	4

wt = 100 gm

Sr. No.	Weight Observed in g	Difference in g	Limit
1.	At Centre- (A) 98.756		
2.	At Corner 1 (B) 98.759	B-A = 0.003	
3.	At Corner 2 (C) 98.758	C-A = 0.002	± 0.1 %
4.	At Corner 3 (D) 98.755	D-A = -0.001	
5.	At Corner 4 (E) 98.757	E-A = 0.001	

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

01/12/22

ANALYSED BY **CHECKED BY**

Name. Dube Sanjivani Ganesh.

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

4. **Test for Repeatability :**

Selected Weight in g: 50 gm

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	50.692	6	50.690	± 0.1 %
2	50.692	7	50.691	
3	50.690	8	50.692	
4	50.694	9	50.692	
5	50.693	10	50.693	

1) max = $\frac{50.694 \times 0.1}{100}$ = 0.0506

2) 50.694 + 0.0506
= ~~50.747~~ 50.7446

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

mini = 3) 50.690 - 0.0506
= 50.6894

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

11/12/22

ANALYSED BY

CHECKED BY

Name- Dube Sanjivani Ganesh

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

DAILY CALIBATION 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)	9.1 (± 0.05)
1	11/12/22	4.02	6.89	9.09

Slope - 96%

11/12/22
PERFORMED 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. :-	DS8000	1 of 1
Name Of Students	Dube Sanjivani, Ganesh.	

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.

18/12/22

ANALYSED BY

CHECKED BY:-

	ARNI ANALYTICALS	
TITLE	DISSOLUTION TEST APPARATUS WORKSHEET	
Instrument Name :-	DISSOLUTION TEST APPARATUS	Page No.
Instrument ID : -	ARNT/INS - 003	
Instrument Model No. :-	DS 8000	1 of 1
Name Of Students	Dube Sanjivani Ganesh	

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 900mL 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 100rpm. place one capsule in each of six paddle and adjust the paddle in the dissolution medium so that there is a distance of 25mm ± 2mm betn 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

18/12/22

ANALYSED BY

CHECKED BY:-

ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV-Spectrophotometer	Page No.
Instrument ID : -	ARNI/INS-002	
Instrument Model No. :-	LMSP-UV-100B	1 of 1
Name Of Students	Dube Sanjivani Ganesh	

NAME OF TEST :-

1) **PHOTOMETRIC ANALYSIS**
2) **WAVELENGTH SCAN**

PREPARATIONS:-

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 5mlof the above solution to 50ml with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- **273nm**

ANALYSED BY

CHECKED BY:-

ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	Page No.
Instrument Name :-	UV- Spectrophotometer	
Instrument ID : -	ARNI/INS-002	1 of 1
Instrument Model No. :-	LMSP-UV-100B	
Name Of Students	Dube Sanjivani Ganesh	

Date:-

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

NAME OF TEST :- wavelength scan

PREPARATIONS:-

STANDARD PREPARATION :-
wt. accurately 10 mg. of caffeine standard in a 100ml. volumetric flask, add 60ml of water sonicate for 5min, to completely dissolve, makeup the volume with water- further dil. 5ml of the above soln, to 50ml with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- 273nm

OBSERVATIONS:-

MAXIMUM ABSORPTION WAVELENGTH

273nm - max
206nm - min

ANALYSED BY

CHECKED BY:-

Maratha Vidya Prasark Samaj, Nashik

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

A

Internship Report

On

Research And Development

Submitted By-

Miss.Sujata Pandit Nirmal

M.Sc. (Organic Chemistry)

Dept. Of Chemistry

Year: 2022-2023

Maratha Vidya Prasark Samaj,Nashik

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

Examination Seat No:

Certificate

This is to Certified that the internship report

"Research And Development"

Has been carried out in our premises by-

Nirmal sujata pandit

Under my guidance in partial fulfilment of the requirement for

Second year of M.Sc. during the academic year 2022-23

In

SAVITRIBAI PHULE PUNE UNIVERSITY, PUNE-411007

Prof. M.Gaware

Head Of the Department

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

12.05.2023

External examiner

Dr .P.V Rasal

Principal

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

ARSS BIOFUEL PRIVATE LIMITED
CIN-U24119OR2003PTC007115
FACTORY ADD: GAT NO: 196, WADIVARHE, DARNA ASWALI ROAD, TALUKA - IGATPURI, NASHIK-1
EMAIL ID – arss112000@yahoo.com PH +91 9422271148 / 8830665543.

APPRENTICESHIP LETTER

To whom it may concern

This is to certify that **Ms. Sujata Pandit Nirmal worked with ARSS Biofuel Pvt. Ltd.** as a Chemist from Novomber2022.

She has a wide knowledge in chemical process intensification and optimization and has worked on several projects regarding the same for our company.

Her efforts and skills are commendable and have contributed towards the successful operation of the company in the research and operational fronts. She has performed her duties in diligent manner and is a valuable resource of our company.

Her job role included:

- Assessing existing processes and taking measurements
- Perform process Simulation and troubleshooting issues
- Assisting optimizing and upgrading systems

wishing her good luck for future endeavors.

For ARSS BIOFUEL PVT LTD

Authorized Signatory

ARSS BIOFUEL PRIVATE LIMITED CIN U24119 OR2003 PTC007115 NASHIK

Place: Nashik

Date:

ACKNOWLEDGEMENT

I would like to thank the owner of **ARSS Pingle Mohan** for giving me permission to use all the facilities available in the industry for my internship work. I would also like to thank the all the worker of **ARSS** for their cooperation . I am grateful to his encouragement, guidance and supervision of my internship work during the year. I must acknowledge the financial support given to my training by my parents. I would also like to thank principle of G.M.D college **Dr. P.V Rasal** and head of the department **Dr. Manoj Gaware** and all the teaching and non-teaching staff for their help.

Nirmal Sujata Pandit

M.Sc. II (Organic Chemistry)

INDEX

Abstract

I have undergone my industrial training in ARSS Biofuel in Igatpuri in November 2022. ARSS bio fuel is company that specialized in manufacturing anhydrous ethanol and sanitizer. I was placed in sanitizer department as a lab technician under a senior supervisor.

During my one month internship I have learnt the basics knowledge of doing maintenance of machine. Furthermore, I have learnt about the sanitizers manufacturing.

They always focus more on safety of worker as they are dealing with chemical. All ine this industrial training has given me the valuable insight of being a professional chemist.

Introduction

Internship program has become the bridge for those who want to enter to corporate level from the college life, through internship I got to know the real working environment that was very much different from my course study.

The primary object objective of the internship is to generate a through understanding of the work place relationship, performing of the activities and engaging oneself in the working environment. It was more to get practical implication of all the studies, theories that I had acquired so far. This would help me to pave a way towards growth in my academic as well as personal development.

- To learn and apply theoretical knowledge practically.
- To develop interpersonal managerial and communication skill.
- To come up with possible strategies to gain competitive advantage.
- To fulfill the partial requirement for the Master of Chemistry of SPPU.

A R S S
BIOFUEL PVT. LTD.

About Company

Name : ARSS BIOFUEL PRIVATE LIMITED

CIN – U24119OR2003PTC007115

Add : GATE.NO : 196 , WADIVARHE,DARNA ASWALI ROAD , TAL,

IGTAPURI NASHIK-1

Email ID : arss112000@yahoo.com

Ph : +91 9422271148 / 8830665543

It is a non government company ,incorporated on 17 April 2003 . it's a private unlisted company and is classified as company limited by shares.

The company authorised capital stands at Rs 100 lakhs and 62.90% paid –up

capital which is Rs 69.3 lakhs. ARSS Biofuel Private limited last annual general

meet happened on 2017.

Arss Biofuel is majorly in manufacturing (Metal & Chemicals) .

Bio-Fuel

- Unlike other renewable energy sources, biomass can be converted directly into liquid fuel called "Bio fuel".
- It helps to meet transportation fuel need.
- Bio fuel is commonly advocated as a cost effective and environment friendly.
- Bio fuel is the fuel which produce from organic product and wastage.
- The common commercially used bio fuel are Bio Ethanol, Bio Diesel and Bio Methane.
- Bio ethanol is made from sugar, alga, wheat and sugar beet .
- Bio diesel is made from vegetable oils, algal lipids and animal fats.

Company Product

1.Ethanol

- Ethanol is an organic compound.
- It is an alcohol with the chemical formula C_2H_5OH.
- Ethanol is volatile, flammable, colourless liquid with a characteristic wine like odour and pungent test.

Molar mass: 46.07 gm/mo

Boiling Point: 78.37°C

Density: 789 kg/m³

2. Denatured Anhydrous Ethanol

- Production of Ethanol is large scale has been made by extractive distillation using conventional solvent like ethylene-glycol.
- Extractive distillation process is done to obtain pure ethanol using ethylene-glycol as solvent. Residue curve maps arcused to analyse the proposed distillation process in interpreting mixture behaviour and feasibility of distillation column.
- Ethanol forms a minimum – boiling azeotrope with water at about 90mol% at 1 atm.
- This azeotrope must be broken to achieve anhydrous ethanol.
- The usual solvent applied in the industries to promote ethanol and water separation is the ethylene-glycol.

Posted on May 10, 2018

We are manufacturer and exporter of Denatured Ethanol.

Application Of Anhydrous Ethanol

- Chemical Reagent
- Organic Solvent
- Raw Material for drugs
- Raw material for cosmetics
- It also used to remove the paint, ink from fabrics.

3. Ethanolic HCL

Hydrochloric acid in ethanol for microscopy is used for human medical cell diagnosis and histological investigation of sample material of human origin. Acid fast bacteria are difficult to stain because of the high proportion of lipid and wax in their cell wall.

Structure of ethanolic HCL: C_2H_7ClO

Price of Ethanolic HCL : pure grade Ethanolic HCL for ARSS biofuel liquid at is 550/kg in Igatpuri Nashik-1.

4.Sanitizers:

- Hand Sanitizer (also known as hand antiseptic) hand disinfectant hand rub or hand.
- Alcohol based hand sanitizer that is at least 60% alcohol in water (specially ethanol) or isopropyl alcohol or isopropanol is recommended by the united state centre for Disease Control and Prevention (CDC).
- The CDS recommends the following step's when using an alcohol based hand sanitizers.

1. Apply Product to palm on hand.
2. Rub hand together.
3. Rub product over all surface of hand and finger's until hand are dry.
4. Do not go near flame or gas banned or any burning object during application of hand sanitizer.

Preparation of institizer (sanitizer)

Steps:

(Isopropyl alcohol) (95%)

Hydrogen peroxide (3%)

⇓

Glycerin $C_3H_8O_3$

⇓

Distilled water

⇓

Shake or stir well

⇓

Clear or contain in plastic

Many hand sanitizer must be stored at below 25°C.

Do not store hand sanitizer in car or anywhere it will get too hot hand sanitizer is flammable so don't store it next to heat source. Spark or open flames.

- Instead stored in following plastic bottles:

  Plastic used for hand sanitizers.

  PET- polyethylene terephthalate

  HDPE- high density polyethylene

  LDPE- low density polyethylene

  PP - polypropylene

Job Description:

Job title and grade: Internship-Laboratory chemist

Job purpose: Laboratory

Key Responsibility:

- Conduct testing under supervision of either chemist/senior or under laboratory technician.
- Evaluation /test quality of specially chemicals for sanitizer.
- Handling and cleaning laboratory and apparatus (laboratory hygiene).
- Packaging sample requested by customer.

Conclusion

The experience and knowledge during the internship at ARSS Bio fuel was great. This industry has a superb work culture, great mind and very high quality of work. At the laboratory, they provide many instrument and their worker will conduct the entire instrument by their own. During the internship, I was introduced and learned to handle equipment such a high machine used to manufacture ethanol and other product. The knowledge and skill get from the internship will be use to apply a worker and do the final year project. Working with their people was a rare chance and it was another opportunity to make friends and share ideas. Overall experience is very helpful.

Reference

- https:/m.indianmart.com
- http:/arss-biofuel-pvt-ltd.business
- http:/www.thecompanycheck.com

„"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. Ajinkya Dattatray Lonare

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. Ajinkya Dattatray Lonare** 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
G.M.D. Arts, B.W. Commerce
and Science college. Sinnar

Examiner

12-05-2023

Principal

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

CERTIFIED ISO 9001 CERTIFIED

ARNI ANALYTICAL

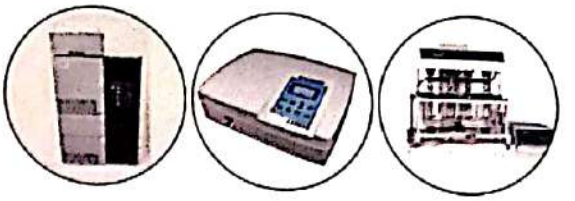
Add.: Pushpak Apartment, Flat No. 102, Lane No. 3, Near Neurocare Hospital, Pandit Colony, Nashik.
e-mail : arnianalyticals@gmail.com | Web Site : www.arnianalyticals.com

Certificate

This is to Certify that ..... *Ajinkya Dattatraya Lonare* .....
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. **Ajinkya Dattatray Lonare** 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:- 

Name:- Ajinkya Dattatray Lonare

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

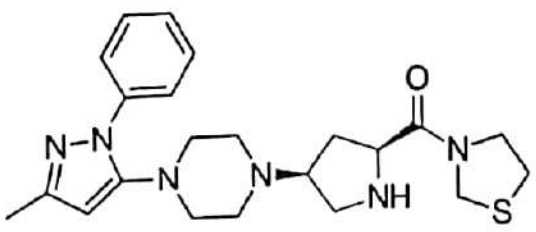
Physical Testing-

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-



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:**

TABLETS-1	:	285 mg	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%**

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 :-
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	:	900 ml	Temperature	:	37°C ± 0.5°C
Apparatus	:	USP Type II (Paddle)	Time	:	45 min

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

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

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

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

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

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

$89.95 + 89.19 + 92.81 + 88.55 + 86.43 + 90.53 = 89.57$ %

6

Average:- 89.57 %

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

6) Assay (By HPLC):

Chromatographic Conditions:

Column	:	C_{18}, (150mm X 4.6mm), 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 (2.5 min) (5 min)

Preparation of solutions:

- **Standard preparation:**

Dissolved 0.1M potassium dihydrogen orthophosphate in 300ml of wate

Prepare a mix. of Buffer, Acetonitrile (65:30 v/v). filter through 0.45 µ filter & degas.

- **Sample preparation:**

Standard Weight :- 20mg
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) $\frac{11960800}{11135986} \times \frac{29.43}{100} \times \frac{100}{286.8} \times \frac{P}{100} \times 286.8 \times \frac{426.57}{628.86} \times \frac{100}{20} = 104$

=

2) $\frac{11947512}{11135986} \times \frac{29.43}{100} \times \frac{100}{286.8} \times \frac{P}{100} \times 286.8 \times \frac{426.57}{628.86} \times \frac{100}{20} = 106$

=

Average :- 105.92%.

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 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 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 volumes of Water and 20 volumes of Methanol. Mix well.

70 30 Acetonitrile

- **STANDARD PREPARATION :-**

Weigh accurately 20mg of Caffeine standard to a 100ml volumetric flask. Add 60ml of HPLC grade wate 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 :-

DATA SHEET

NAME OF TEST :- SYSTEM SUITABILITY

SYSTEM SUITIBILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINCE

- **Chromatographic Conditions:**

Column	A stainless steel
Data Aquisitation Time	5 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 :-**

Prepare a mixture of 70 volumes of water & 20 volumes of Acetonitrile & Mix well.

- **STANDARD PREPARATION :-**

Weigh accurately 20 mg of caffiene standard to 100ml volumetric flask. Add 60 ml HPLC grade water & shake it make up the volume & further dil. 5 ml of above soln to 50ml vol. flask & dilute to make up volume.

- **SEQUENCE OF INJECTION :-**

Name of Solution	No. Of Injection
Blank	1
Standard	2

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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	:	10 min
Pump	:	1.00 ml/min
Port	:	A
Detector (Wavelength)	:	210 nm
Column Oven Temperature	:	30°C
Degasser	:	On
Autosampler Temperature	:	10°C

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- **CHROMATOGRAPHIC PARAMETERS-2**

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	:	4°C

- **CHROMATOGRAPHIC PARAMETERS-3**

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

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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	:	15 min
Pump	:	0.70 ml/min
Port	:	B
Detector (Wavelength)	:	225 nm
Column Oven Temperature	:	35°C
Degasser	:	on
Autosampler Temperature	:	12°C

- **CHROMATOGRAPHIC PARAMETERS-5**

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

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	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	200.065	199.8000 to 200.2000
2	100.0000	98.790	99.9000 to 100.1000
3	50.0000	50.714	49.9500 to 50.0500
4	20.0000	19.055	19.9800 to 20.0200
5	10.0000	10.002	9.9900 to 10.0100
6	5.0000	5.083	4.9950 to 5.0050
7	2.0000	1.329	1.9980 to 2.0020
8	1.0000	0.925	0.9990 to 1.0010
9	0.5000	0.152	0.4995 to 0.5005
10	0.2000	0.105	0.1998 to 0.2002
11	0.1000	0.058	0.0999 to 0.1001
12	0.0500	0.088	0.0499 to 0.0501
13	0.0200	0.014	0.0199 to 0.0200
14	0.0100	0.013	0.0099 to 0.0100
15	0.0050	0.008	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	50	50.714
2	100	98.790
3	200	200.067

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)	50.714		± 0.1 %
2.	At Corner 1 (B)	50.710.	B-A = −0.004	
3.	At Corner 2 (C)	50.710	C-A = −0.004	
4.	At Corner 3 (D)	50.708	D-A = −0.006	
5.	At Corner 4 (E)	50.709	E-A = −0.005	

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 :-		
Instrument ID : -		3 of 3

4. Test for Repeatability :

Selected Weight in g: 100 g

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	98.790	6	98.796	± 0.1 %
2	98.792	7	98.793	
3	98.794	8	98.794	
4	98.795	9	98.794	
5	98.793	10	98.794	

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

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

Aljikya
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	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:-

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	200.022	199.8000 to 200.2000
2	100.0000	98.770	99.9000 to 100.1000
3	50.0000	50.699	49.9500 to 50.0500
4	20.0000	19.051	19.9800 to 20.0200
5	10.0000	9.997	9.9900 to 10.0100
6	5.0000	5.080	4.9950 to 5.0050
7	2.0000	1.280	1.9980 to 2.0020
8	1.0000	0.923	0.9990 to 1.0010
9	0.5000	0.191	0.4995 to 0.5005
10	0.2000	0.106	0.1998 to 0.2002
11	0.1000	0.057	0.0999 to 0.1001
12	0.0500	0.035	0.0499 to 0.0501
13	0.0200	0.014	0.0199 to 0.0200
14	0.0100	0.008	0.0099 to 0.0100
15	0.0050	0.005	0.0049 to 0.0051

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

Ajinkya
ANALYSED BY

CHECKED BY:-

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

2. Test for Linearity:

Sr. No.	Selected Weights in g	Observed Weight in g
1	5	5·081
2	10	10·002
3	20	18·375

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) 10·000		
2.	At Corner 1 (B) 9·999	B-A = −0·001	
3.	At Corner 2 (C) 9·998	C-A = −0·002	± 0.1 %
4.	At Corner 3 (D) 10·000	D-A = 0·000	
5.	At Corner 4 (E) 9·997	E-A = −0·003	

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 :-		
Instrument Model No. :-		3 of 3
Instrument ID : -		

4. Test for Repeatability :

Selected Weight in g: 10 gm

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	9.997	6	9.998	± 0.1 %
2	9.998	7	9.996	
3	10.000	8	9.997	
4	9.999	9	9.997	
5	9.998	10	9.998	

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

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

Alfiikya

ANALYSED BY

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-00	

DAILY CALIBATION 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	16/12/2022	3.95	6.77

Slope = 95 %.

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	ARNI ANALYTICALS	
TITLE	DAILY CALIBRATION RECORD OF pH-METER	
Instrument Name :-		Page No
Instrument Make :-		1 of 1
Instrument Model No. :-		
Instrument ID : -		

DAILY CALIBATION 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)
01.	10/12/2022	3.99	6.88

Slope = 97 %

Alijkya
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	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	Lonare Ajinkya Dattatraya	

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

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	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 min

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 at 100 rpm. Place one capsule in each of six paddle & adjust it in medium so the dist of 25 mm ± 2 mm betn bottom of paddle & inside bottom of vessel. Start the apparatus. At end of specified time, withdrawn 10 ml aliquot from zone midway. Further diluted 2 ml above solution to 25 ml dissolution medium.

Aijaya
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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	Lonare Ajinkya Dattatraya	

NAME OF TEST :-

1) **PHOTOMETRIC ANALYSIS**
2) **WAVELENGTH SCAN**

PREPARATIONS:-

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 5mlof the above solution to 50ml with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- **273nm** (Maxima)

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ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-		Page No.
Instrument ID : -		
Instrument Model No. :-		1 of 1
Name Of Students		

Date:-

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

NAME OF TEST :- 1) Photometric Analysis
2) Wavelength Scan

PREPARATIONS:-

STANDARD PREPARATION :-

Weigh accurately 10 mg of caffiene standard in 100 ml volumetric flask, add 60 ml water & sonicate for 5 min. dissolve it & makeup volume. Further dilute 5 ml of soln to 50 ml of water.

UV-SPECTROPHOTOMETER WAVELENGTH :- 273 nm (maxima)
246 nm (minimum)

OBSERVATIONS:-

MAXIMUM ABSORPTION WAVELENGTH

273 nm.

Ajitkya
ANALYSED BY

CHECKED BY:-

„"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

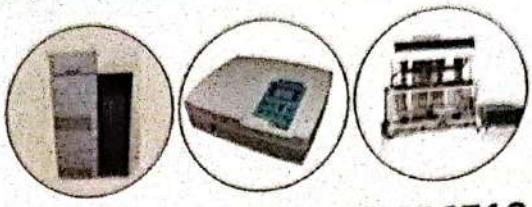
Add.: Pushpak Apartment, Flat No. 102, Lane No. 3, Near Neurocare Hospital, Pandit Colony, Nashik.
e-mail : arnianalyticals@gmail.com | Web Site : www.arnianalyticals.com

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

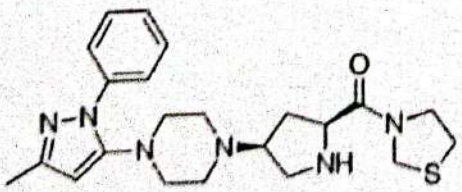
Physical Testing-

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	

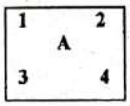
2. Test for Linearity:

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

ANALYSED BY

CHECKED BY

	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

ANALYSED BY

CHECKED BY

ARNI ANALYTICALS

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

CHECKED BY

	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

„"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. Ashwini Raosaheb Thorat

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,

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APRIL 2023

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

SINNAR, DISTRICT- NASHIK

DEPARTMENT OF CHEMISTRY (PG)

CERTIFICATE

This is to certify that **Miss. Ashwini Raosaheb Thorat** 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
G.M.D. Arts, B.W. Commerce
and 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 ..... *Ashwini Raosaheb Thorat* .....
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. **Ashwini Raosaheb Thorat** 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:- 

Name:- Ashiwini Raosaheb Thorat

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

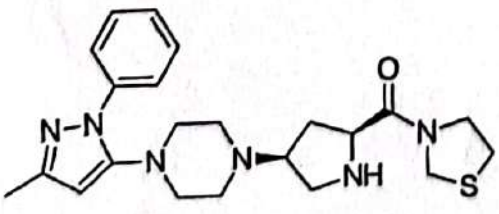
Physical Testing-

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-



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. after 5 → 50 ml.

- **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 ~~210~~ 230nm.

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. 20 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, 230 nm
Run time	:	1.5 times of the retention time of principle peak

13.2 Preparation of Mobile Phase:

- **Preparation of Buffer:**

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:

- **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

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)

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FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG |

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	:	286	TABLETS-6	:	287
TABLETS-2	:	283	TABLETS-7	:	284
TABLETS-3	:	282	TABLETS-8	:	286
TABLETS-4	:	292	TABLETS-9	:	291
TABLETS-5	:	289	TABLETS-10	:	286

AVERAGE WEIGHT:- $\frac{2876}{10} = 287.6$ g **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	:	283	TABLETS-6	:	286
TABLETS-2	:	285	TABLETS-7	:	293
TABLETS-3	:	283	TABLETS-8	:	289
TABLETS-4	:	285	TABLETS-9	:	292
TABLETS-5	:	293	TABLETS-10	:	288

MINIMUM WEIGHT :- 283 g
MAXIMUM WEIGHT :- 293 g.

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	:	0.1N HCl	Rotatory Speed	:	75
Volume	:	900	Temperature	:	37°C
Apparatus	:	Paddle USP-II	Time	:	45 min.

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= $\frac{0.6364}{0.5531} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 113.72 %

Tablet 2= $\frac{0.6261}{0.5531} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 111.88 %

Tablet 3= $\frac{0.5401}{0.5531} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 96.52 %

Tablet 4= $\frac{0.6254}{0.5531} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 111.79 %

Tablet 5= $\frac{0.6145}{0.5531} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 109.84 %

Tablet 6= $\frac{0.5468}{0.5531} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 97.71 %

Average:- $\frac{646.24}{6} = 107.70$ %

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 x 4.6 mm) 5 µm
Pump mode	:	Isocratic
Mobile Phase	:	Buffer : Acetonitrile (80:20)
Flow rate	:	1.0 ml/min
Injection volume	:	20 µl
Column Temperature	:	30°C
Wavelength	:	210 nm

Preparation of solutions:

- **Standard preparation:**
  Wt. accurately 20 mg Teneligliptin. dissolve in 100 ml Volumetric flask make up the volm with water.

- **Sample preparation:**
  of 10 tablet and determine average wt. cut. transfer powder equivalent 20 mg Teneligliptin. dissolve 100 volumetric flask.

  Standard Weight :- 20 mg
  Sample Weight :- 282
  Average Weight :- 282.33
  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) $\frac{1373685}{1436757} \times \frac{29.48}{100} \times \frac{100}{282} \times \frac{P}{100} \times 282.33 \times \frac{426.57}{628.86} \times \frac{100}{20}$

= 95.57%

2) $\frac{1270685}{1436757} \times \frac{29.48}{100} \times \frac{100}{282} \times \frac{P}{100} \times 282.33 \times \frac{426.57}{628.86} \times \frac{100}{20}$

= 88.41%

Average :- 91.99%

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 CHT	1 of 1
Instrument ID : -	ARNT/IIMS -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 10 min
Pump (Flow Rate)	~~1.00~~ ml/min 1.50 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. 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 Performace liquid chromotography	Page No
Instrument Make :-	Shimadzu	
Instrument Model No. :-	LC2010 CHT	1 of 1
Instrument ID : -	ARNI / IMS 001	

NAME OF STUDENT :-

DATA SHEET

NAME OF TEST :- SYSTEM SUITABILITY

SYSTEM SUITIBILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINCE

- **Chromatographic Conditions:**

Column	Stainless Steel Column Dimension L 15 cm x ID - 4.6 mm : P.S. 5µm ~~L 250 mm x ID - 4.6 mm : P.S. 5µ~~
Data Aquisitation Time	10 min
Pump (Flow Rate)	1.50 ml/min
Port	A
Detector (Wavelength)	273 nm
Column Oven Temperature	36°C
Degasser	off
Autosampler Temperature	off

- **MOBILE PHASE PREPARATION :-**

Prepare a mixture 70 vol^{n} of water and 30ml vol^{n} of methanol. Mix well.

- **STANDARD PREPARATION :-**

wt. accrately 20 mg caffeine standard to 100ml Volumetric flask. dissolve completely & make up vol^{n} upto the mark. mix well. farther dilute 5ml sol^{n} to 50 ml volumetric flask. make up vol^{n}.

- **SEQUENCE OF INJECTION :-**

Name of Solution	No. Of Injection
Blank	
Standard	

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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 2010 CHT	1 of 3
Instrument ID : -	ARNI/INS - 001	
Name Of Student :-	Thorat Ashwini Raosaheb	

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	:	10 min
Pump	:	1.00 ml/min
Port	:	A A
Detector (Wavelength)	:	210 nm
Column Oven Temperature	:	30°C
Degasser	:	on
Autosampler Temperature	:	10°C

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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 2010 CHT	2 of 3
Instrument ID : -	ARNI / IMS -001	
Name Of Student :-	Thorat Ashwini Raosaheb	

- **CHROMATOGRAPHIC PARAMETERS-2**

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisitation Time	:	13
Pump	:	0.80 ml/min.
Port	:	C
Detector (Wavelength)	:	222 nm
Column Oven Temperature	:	20°C
Degasser	:	off
Autosampler Temperature	:	7°C

- **CHROMATOGRAPHIC PARAMETERS-3**

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

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ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	High Performace liquid chromatography	Page No
Instrument Make :-	Shimadzu	
Instrument Model No. :-	LC 2010 CHT	3 of 3
Instrument ID : -	ARNI/INS/001	
Name Of Student :-	Thorat Ashwini Pandurang	

- **CHROMATOGRAPHIC PARAMETERS-4**

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	:	5°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

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	ARNI ANALYTICALS	
TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-	ANALYATICAL BALANCE	Page No
Instrument Make :-	HENSER	
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	199.999	199.8000 to 200.2000
2	100.0000	99.758	99.9000 to 100.1000
3	50.0000	50.680	49.9500 to 50.0500
4	20.0000	20.049	19.9800 to 20.0200
5	10.0000	9.999	9.9900 to 10.0100
6	5.0000	5.083	4.9950 to 5.0050
7	2.0000	1.303	1.9980 to 2.0020
8	1.0000	0.926	0.9990 to 1.0010
9	0.5000 500mg	0.150	0.4995 to 0.5005
10	0.2000	0.103	0.1998 to 0.2002
11	0.1000	0.057	0.0999 to 0.1001
12	0.0500	0.0377	0.0499 to 0.0501
13	0.0200	0.017	0.0199 to 0.0200
14	0.0100	0.014	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.**

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

2. **Test for Linearity:**

Sr. No.	Selected Weights in g	Observed Weight in g
1	50.0000 gm	50.0005 gm
2	200.0000 gm	199.9994 gm
3	100.0000 gm	99.9998 gm

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

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3. **Test for Eccentricity:**

1	2
A	
3	4

Sr. No.	Weight Observed in g	Difference in g	Limit
1.	At Centre- (A) 199.9999		
2.	At Corner 1 (B) 200.0011	B-A = 0.0012	
3.	At Corner 2 (C) 200.0000	C-A = 0.0011	± 0.1 %
4.	At Corner 3 (D) 199.9990	D-A = -0.0009	
5.	At Corner 4 (E) 199.9987	E-A = -0.0012	

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

0.199 Limit not increase

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

4. **Test for Repeatability :**

Selected Weight in g:

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	10.0001	6	10.0000	± 0.1 %
2	9.996	7	9.998	
3	9.997	8	10.0011	
4	9.998	9	9.99999	
5	10.0011	10	9.9998	

10.010 ↑
9.990 ↓

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

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

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

NAME OF STUDENT:-

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	200.3555	199.8000 to 200.2000
2	100.0000	99.778 179	99.9000 to 100.1000
3	50.0000	50.708 708	49.9500 to 50.0500
4	20.0000	19.056	19.9800 to 20.0200
5	10.0000	10.0000	9.9900 to 10.0100
6	5.0000	5.082 2	4.9950 to 5.0050
7	2.0000	1.233 3	1.9980 to 2.0020
8	1.0000	0.925	0.9990 to 1.0010
9	0.5000	0.154	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	0.035	0.0499 to 0.0501
13	0.0200	0.019	0.0199 to 0.0200
14	0.0100	0.013	0.0099 to 0.0100
15	0.0050	0.012	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 :-	ANLYTICAL BALANCE	Page No.
Instrument Make :-	HEMSAR	
Instrument Model No. :-	DS 8000	2 of 3
Instrument ID : -	ARNI IINS 004	

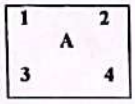
2. **Test for Linearity:**

Sr. No.	Selected Weights in g	Observed Weight in g
1	200	200.036
2	100	98.785 98.785
3	50	50.706 50.706

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

ANALYSED BY **CHECKED BY:-**

3. **Test for Eccentricity:**



diff = ± 0.09528

Sr. No.	Weight Observed in g	Difference in g	Limit
1.	At Centre- (A) 19.056		± 0.1 %
2.	At Corner 1 (B) 19.057	B-A = +0.001	
3.	At Corner 2 (C) 19.056	C-A = 0	
4.	At Corner 3 (D) 19.057	D-A = +0.001	
5.	At Corner 4 (E) 19.056	E-A = 0	

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 :-	ANLYTICAL BALANCE	Page No.
Instrument Make :-	WENSER	
Instrument Model No. :-	BS - 8000	3 of 3
Instrument ID :-	ARNI ITNS 004	

4. Test for Repeatability :

Selected Weight in g: d.11 : 0.1002

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	10.002	6	9.999	±0.1 %
2	10.008	7	10.001	
3	9.997	8	10.001	
4	10.000	9	10.002	
5	10.001	10	10.005	

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	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 004	

DAILY CALIBATION 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	14/12/22	3.91	6.81

PERFORMED BY

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 004	

DAILY CALIBATION 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.

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  - 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	4.011	6.95

slope 97%
slope 97%

PERFORMED 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	Thorat Ashwini Raosaheb	

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 / IHS 003	
Instrument Model No. :-	DS 8000	1 of 1
Name Of Students	Thorat Ashvini Raosaheb	

NAME OF TEST :-

TRIAL FOR DISSOLUTION TEST.

DISSOLUTION CONDITIONS:-

Dissolution Media	Water
Media Volume	900 ml
Apparatus	USP -II
RPM	100
Temperature	37.0 ± 0.5°C
Time	45 minutes

PREPARATIONS:-

Pour 900 ml of dissolution medium in each vessel. dissolution medium equilibrate at 37°C .. Adjust the speed of stiring element at 100 rpm. Place one capsule in each six paddle & adjust the distance 25 mm . bet^ the bottom of paddle & inside the bottom of vessel. start the Apparatus end of specific time interval , withdraw 10 ml aliquote.

ANALYSED BY

CHECKED BY:-

ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV - SPECTROPHOTOMETER	Page No.
Instrument ID :-	ARNI/INS-002	
Instrument Model No. :-	LABSP-UV 1900	1 of 1
Name Of Students	Thota Ashwini Prasad	

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

Date:-

NAME OF TEST :- Wavelength Scan

PREPARATIONS:-

STANDARD PREPARATION :-

Wt. of accurately 10 mg caffeine standard in volumetric flask. dissolve substance & make up the Vol^m with water. further dilute 5 ml of above Sol^n to 50 ml with water.

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

OBSERVATIONS:-

MAXIMUM ABSORPTION WAVELENGTH - 274 nm

ANALYSED BY

CHECKED BY:-

	ARNI ANALYTICALS	
TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV -SPECTPHTOMETER	Page No.
Instrument ID : -	ARIYI / INS -004	
Instrument Model No. :-	LMSP -UV 100B	1 of 1
Name Of Students	Thorat Ashwini Raosaheb	

NAME OF TEST :-

1) **PHOTOMETRIC ANALYSIS**
2) **WAVELENGTH SCAN**

PREPARATIONS:-

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 5mlof the above solution to 50ml with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- **273nm**

Wt. accurately 10 mg of affinex ~~standard~~ in a 100 ml volumetric flask. dissolve complety and make up voln. further dilute 5 ml dissolve 50 ml with water.

ANALYSED BY

CHECKED BY:-

Savitribai Phule Pune University

Internship Report

P.G DEPARTMENT OF CHEMISTRY

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

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

CERTIFICATE

This is to certify the work incorporated in the internship was satisfactorily out by MS Gaware Puja Baban of M.Sc. Organic Chemistry. She is a Bonafide student at this college. She has completed this Internship under supervision and guidance during the academic year 2022-2023. This project work submitted by her original and the scientific information obtained from other sources have been duly acknowledged. (CHO-453)

Gaware 12/5/23

Prof. Dr. Manoj Gaware
Head Department of Chemistry
HEAD
DEPARTMENT OF CHEMISTRY
G.M.D. Arts, B.W. Commerce
and Science College, Sinnar

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

12.05.2023
Examiner

REVE PHARMA

Works : Plot No. 78, STICE, A/p Musalgaon,Tal. Sinnar, Dist. Nashik 422 112.
Telephone : 02551-240138/39. **Telefax** : 02551-240127.
Email : revepharma@gmail.com, info@revepharma.com. **Website** : www.revepharma.com

Date: 11.05.2023

TO WHOMSOEVER IT MAY CONCERN

This to certify that **Miss Pooja Baban Gaware** has completed the training from dated on 10.04.2023 to 10.05.2023.

She has undergone training of **Tablet, Capsule, Liquid, Oil, Cream & Ointment Manufacturing, Filling & Packing operations.**

We wish good luck for her future life.

For **REVE PHARMA**

Authorized Signatory

HR Executive

REVE PHARMA

ACKNOWLEDGEMENT

First, I would like to thank Miss. Sonali Satpute for giving me the opportunity to do an internship within the organization.

I would like to thank my Head of the Department **Prof. Dr. Manoj Gaware** for his constructive help throughout my internship.

I am extremely great full to my department staff members and friends who helped me in successful completion of this internship.

Organization information:

It was the dream of two professionals to have their own pharmaceutical manufacturing unit. They always dreamed of manufacturing Standardized Ayurvedic Products. They wanted to be a media to spread this vast ancient knowledge to mankind in the form of effective products.

Reve Pharma came into existence in the year 2007. They were inspired by their mentors, Mr. D.J.Dhamne and Mr. Yogesh Deore, both with experience of about 45 years.

People
These two people, Milind Katariya and Anjali Katariya, after an experience of about 10 years, came together to build this dream and started with "Reve Pharma".

Reve Pharma has a board of technical directors, Dr. Anil Ghogre, Dr. C.L.Bhingare and Dr. Santosh Tambe.

Introduction

Location: Plot - 78 STICE Musalgaon Tal- Sinnar Dist , Nashik, Maharashtra, India.

Site Capabilities: Manufacturing Facility- Vitamins & Minerals Premixes

Processing, Primary Packing, Secondary Packing, and

Sachet filling machine

- General Tablets Granulation Area, Three Compression
- cubicles and three packing lines
- State of art Quality laboratory-
- Qualified and competent staff
- Separate Wet and Instrument Lab
- Class 100,000 Microbiology Setup
- Labs are well equipped with latest infrastructure.
- USFDA, WHO-GMP, ISO 9001, 14001,

  22000,HACCP,SQF,Etc certifications.

Mission:

We Shall ensure the Quality, reliability, and innovation thereby enhancing the sustainability and values for all stakeholders.

Values:

Knowledge- Expertise and Innovation

Action- Entrepreneurship and Integrity

Care- Trusteeship and Humility

Impact- Performance and Resilience

The values that guide our culture are embodied in our purpose-

"Doing Well and Doing Good "

Departments in the manufacturing of Tablets:

Here are the departments in which the manufacturing of tablets take place:

Store

Quality Assurance

Quality Control

Production

PPIC (Production planning and Inventory control)

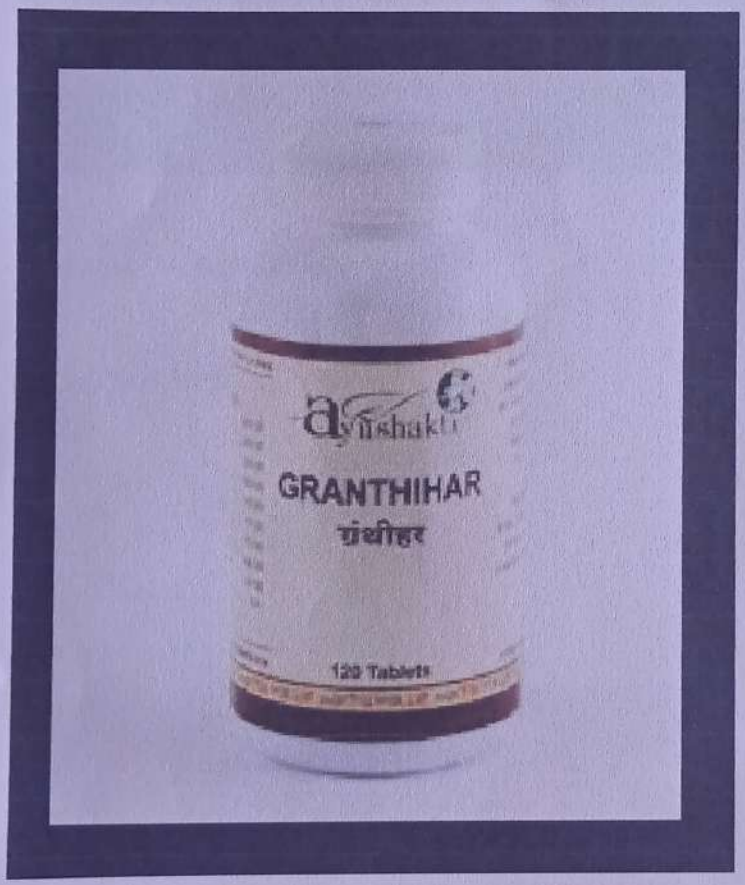
Account

R&D (Research and Development)

LIST OF PRODUCTS

Here are the products I got a chance to work on. From the initial phase to the Packaging phase.

1. GRANTHIHAR Tablet:



Usage: Adenitis, ovarian cyst, tube blocks, inflammation, uterine bulkiness,any block caused by inflammation or fibrosis.

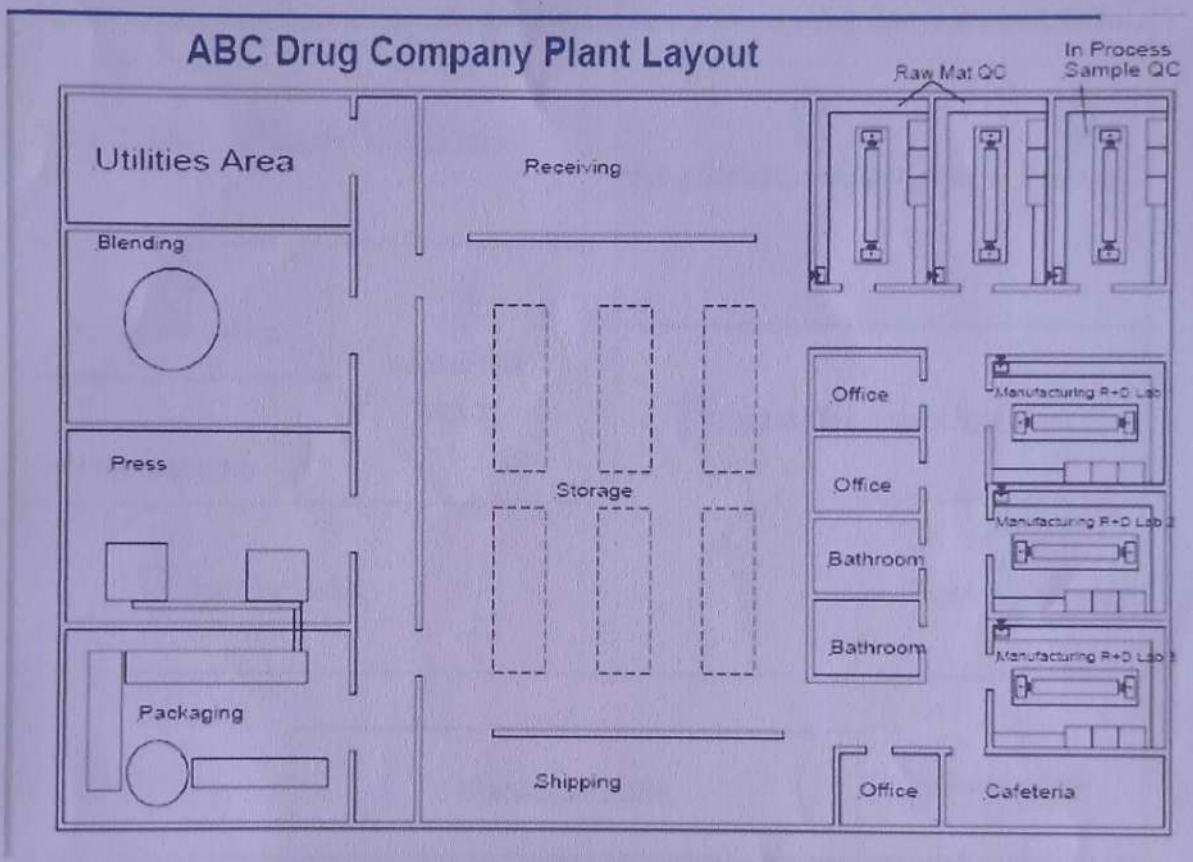
2. Manas Kesharogya vati tablet:

Usage: Used for the health of hair.

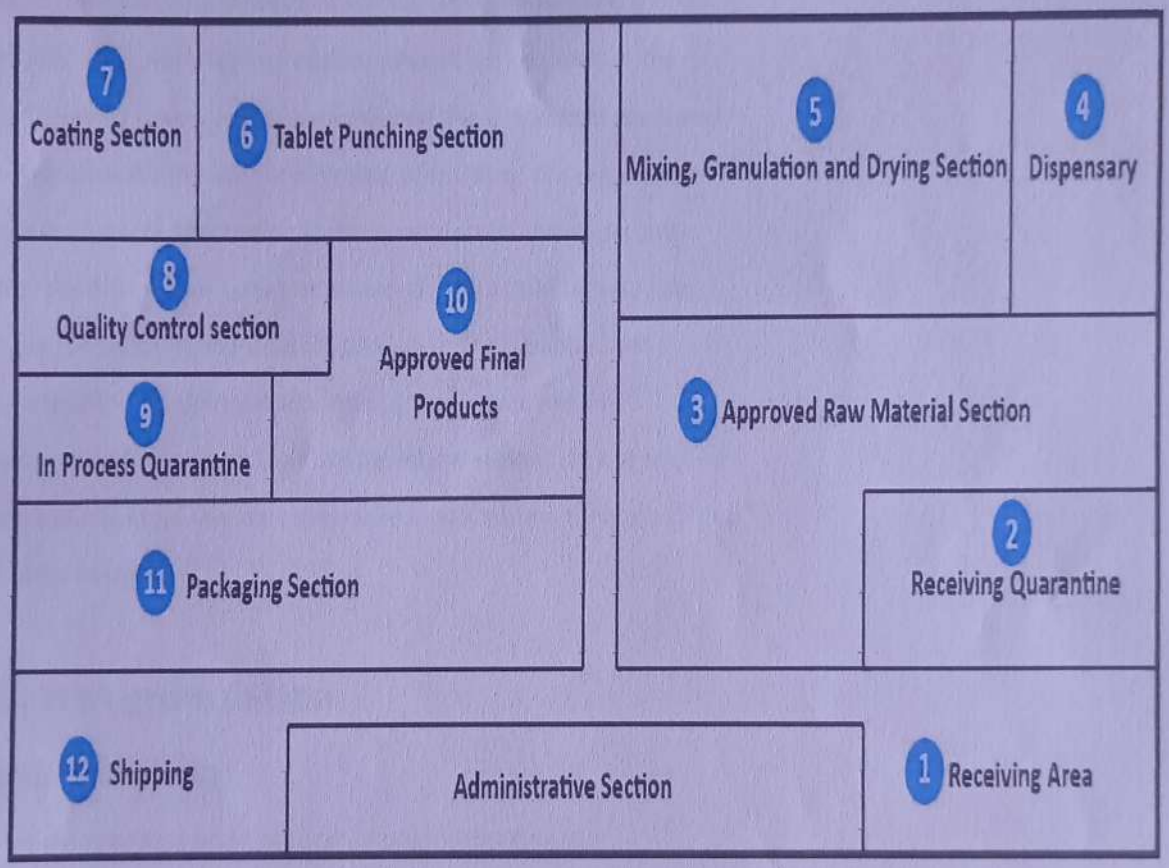
3. Livo fine Tablets:

Usage: Used for the well being of Liver.

Layout


ABC Drug Company Plant Layout
Raw Mat QC
In Process Sample QC
Utilities Area
Receiving
Blending
Press
Storage
Office
Office
Bathroom
Bathroom
Packaging
Shipping
Office
Cafeteria

Layout Of Tablet Manufacturing Section:



Tablet Manufacturing:

Manufacturing of the tableting blend:

In the tablet pressing process, the main guideline is to ensure that the appropriate amount of the active ingredient is in each tablet. Hence all the ingredients should be mix well. If the sufficiently homogeneous mixture of the components cannot be obtained with simple blending processes, the ingredients must be granulated prior to compression to assure an even distribution of the active compound in the final tablet. Two basic techniques are used to granulate the powders for granulation into the tablets. Wet granulation and Dry granulation. Powders that can be mixed welled do not require granulation and can be compressed into tablets through direct Compression.

1. Wet granulation

Introduction:

The most widely used process of agglomeration in a pharmaceutical industry is wet granulation. Wet granulation process simply involve the wet mass of the powder blend with a granulating liquid. Wetting size and drying are important steps in involved in the wet granulation.

Process:

1. Mixing of the drugs and excipients.
2. Preparation of the binder solution

3. Mixing of the binder solution with powder solution to form wet mass.

4. Drying of the moist granules.

5. Mixing of the screened granules with disintegrant, lubricant and glidant.

The wet granulation technique has some limitations.

2. Dry granulation:

Introduction:

In dry granulation process the powder mixture is compressed without the use of solvent and heat. It is the least desirable of all methods of granulation. The two basic procedures are to form compact of material by compression and then mill to the compact to obtain a granules. Two methods are used for dry granulation. The most widely used method is slugging where the powder

is recompressed and the resulting tablet or slug are milled to yield the granules. The other method is to precompress the powder with the powder with pressure rolls using a machine such as Chilosonator.

Roller compaction:

The roller compaction of powder by means of pressure roll can also be accompanied by machine called Chilosonator. Unlike tablet machine the chilosonator turns out to be a compacted mass in a steady continuous flow. The powder is fed down between the powder into the

compaction zone like slugs. The aggregates are milled or screened out for the production into granules.

Processing Steps:

1. Selection of raw materials
2. Weighing
3. Size Reduction
4. Mixing (Precompression or slugging)
5. Screening
6. Lubrication
7. Compression

This method has also some advantages and disadvantages too.

3. *Direct compression:*

This method is used when a group of ingredients can be blended and placed in a tablet press to make tablet without any of the ingredients having to be changed. This is not very common because many tablets have active pharmaceutical ingredients which will not allow for direct compression due to their concentration or excipients used in formulations are not conductive to direct compression. Granulation is the process of collecting particles together by creating bonds between them.

This method is utilize simple operation it requires mixed all the ingredients then go for the direct compression using compressor machine. This method used when the

small dose of drug is directly used with diluent.

Manufacturing of Tablet:

First the powder is filled into the die from above. The mass of powder is determined by the position of the lower punch in the die, the cross section area of the die, and the powder density. At this stage adjustment to the tablet weight are normally made by repositioning the lower punch. After the die filling upper punch is lowered into the die and the powder is uniaxially compressed to a porosity of between 5 and 20%. The compression can takes place in one or two stages and for commercial production occurs very fast. Finally the upper punch is pulled up and out of the die and the tablet is ejected from the die by lifting the lower punch until its upper surface . is flush with the top face of the die. This process is repeated for each tablet.

Common problems encounter in during tablet manufacturing operation include:

- Fluctuations in tablet weight, usually caused by uneven powder flow into the die due to poor powder flow properties.
- Fluctuations in dosage of the active pharmaceutical ingredient, caused by uneven distribution of the API in the tableting blend.
- Sticking, mottling ,orange pill effect ,capping,

lamination, etc., are the problems were encounter in the tablet manufacturing.

Tablet Coating:

An application of coating material to the exterior of tablet with the intension of conferring benefit and properties to the dosage form over uncoated variety.

Objective:

To mask color, odor and taste of drug.

To provide physical and chemical protection to drug.

To control release of drug from the tablet.

To provide physical elegance.

Types of Tablet Coating

- Sugar coating.
- Film coating.
- Press coating.

The materials used for coating may largely comprise sucrose, water soluble film coating polymers or substances which are soluble in intestinal secretions but not in the stomach. These types of coating can be applied by the pan or fluid bed processes. The compression coating technique is suitable for sugar and enteric coatings but not for film. The tablet coating contains use of polymer , coloring agent, etc.

Quality Control Section

It is the essay method in substance such as drugs, packing, material, raw material, adjuvant, containers are checked according to the monograph as per standards given to the *pharmacopoeia*.

Following are the equipment's used in QC section:

1. Magnetic stirrer
2. Electronic and simple balance
3. Capsule disintegration tester
4. Dissolution test apparatus
5. pH meter
6. Autoclave
7. UV and visible spectrophotometer
8. Leaker test apparatus.

1. Autoclave: Autoclave is a device used to sterilize the equipment and supply by subjecting them to high pressure saturated at 121 degree Celsius for around 10-15 minutes.

2. pH meter: A pH meter is an electronic device used for the measuring the pH of liquid formulation. A typical pH meter consist of a special measuring probe connected to an electronic meter that measure the display the pH Reading.

3. Dissolution Test Apparatus: In this apparatus the dissolution study of tablet is carried out . A single tablet is taken and placed in wire mesh basket connected to variable speed motor by means of a shaft this basket is immersed in the dissolution medium contain in 100ml; flask. The flask is maintained at 37+-0.5 degree Celsius by means of constant temp bath. Motor is adjusted to specified speed and samples of fluid are withdrawn at regular time interval to determine the amount of drug in the solution.

4.UV-Visible Spectrophotometer: UV Visible spectrophotometer is used in pharmaceutical industry due to its various applications. It is one useful in detection of impurities, Food industry, forensic science, qualitative

and quantitative analysis are carried out with the help of the same.

CONCLUSION

In the end I am glad to tell you that training in REVE PHARMA, Sinnar Dist- Nashik was an excellent and fabulous experience. During the training I learned about the Pharmaceutical company and above its working the theoretical knowledge is worth for getting a degree, and it is accessible in the book. We can only imagine about the thing we read, but practical life is always different and excellent one. During My training period, I had seen the various instruments and apparatus in the industry. The highly sophisticated instruments that work precisely must be operated with intense care for optimum use. We could acquire a lot of information regarding the latest instruments and their working procedures.

Similarly, from practical point of view a pharmaceutical company is very difficult. During the training session I tried to my level best to gain practical

knowledge as much as I can. I improved my basic classified doubts and also understood the importance of maintaining of quality of products at pharmaceutical company.

I was successfully able to complete my short venture of training. Lastly, I hope that my training report fulfill the intended requirements.

„"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. Jalindar Raybhan Kalokhe

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. Jalindar Raybhan Kalokhe** 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.

Pawar

HOD Chemistry

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

11.05.2023

Examiner

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 : arnianalyticals@gmail.com | Web Site : www.arnianalyticals.com

Certificate

This is to Certify that *Jalindar Raybhan Kalokhe*
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. **Jalindar Raybhan Kalokhe** 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

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:- 

Name:- Jalindar Raybhan Kalokhe

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 exceplents.
- 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

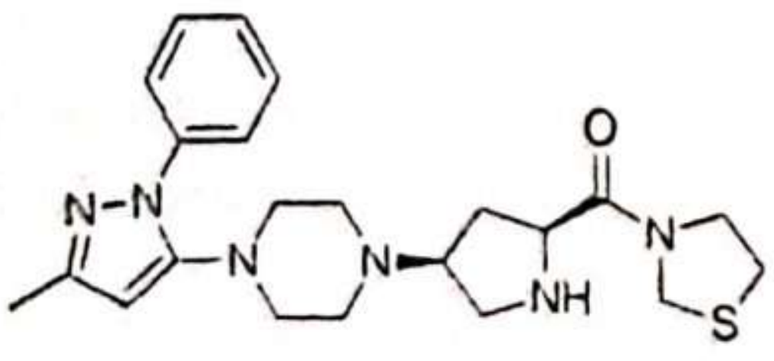
Physical Testing-

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	2 of 3
Instrument ID : -	ARNI / INS - 004	

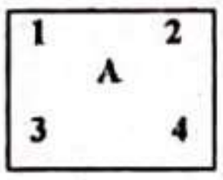
2. Test for Linearity:

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	20 g	19.048 g
2	50 g	50.696 g
3	100 g	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)	19.048 g	
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 :-		
Instrument ID : -		3 of 3

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

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%**

4) **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)

ARN ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

6) Assay (By HPLC):

Chromatographic Conditions:

Column	:	C18 , (150 mm x 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 :- 20mg
Sample Weight :- 286.8mg
Average Weight :- 286.8mg
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) $$\frac{11960800}{11135986} \times \frac{29.43}{100} \times \frac{100}{286.8} \times \frac{P}{100} \times 286.8 \times \frac{426.57}{628.86} \times \frac{100}{20}$$

$= 104.93\%$

2) $$\frac{11947512}{11135986} \times \frac{29.43}{100} \times \frac{100}{286.8} \times \frac{P}{100} \times 286.8 \times \frac{426.57}{628.86} \times \frac{100}{20}$$

$= 105.92\%$

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.

- **STANDARD PREPARATION :-** 2/0 90

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 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 9 0

- **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. :-		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

CHECKED BY

	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

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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 ± 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 175 - 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.

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 175 - 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

„"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. Jyoti Ganesh Chavan

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. Jyoti Ganesh Chavan** 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.

Gawade

HOD Chemistry

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

12-05-2023

Examiner

Principal

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

CERTIFIED 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 ..... *Jyoti Ganesh Chavan* .....
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. **Jyoti Ganesh Chavan** 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:- _____

Name:- Jyoti Ganesh Chavan

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

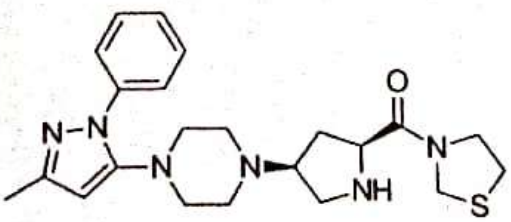
Physical Testing-

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-



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 ± 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.

- **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:**

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:

- **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 preparation
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

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	:	290	TABLETS-6	:	286
TABLETS-2	:	285	TABLETS-7	:	286
TABLETS-3	:	284	TABLETS-8	:	281
TABLETS-4	:	286	TABLETS-9	:	279
TABLETS-5	:	291	TABLETS-10	:	297

AVERAGE WEIGHT:- $\frac{2866}{10} = 286$ **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	:	290	TABLETS-6	:	286
TABLETS-2	:	285	TABLETS-7	:	286
TABLETS-3	:	284	TABLETS-8	:	281
TABLETS-4	:	286	TABLETS-9	:	279
TABLETS-5	:	291	TABLETS-10	:	297

MINIMUM WEIGHT :- 281
MAXIMUM WEIGHT :- 297

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 |

5) Dissolution (By HPLC):

Dissolution Parameters :					
Medium	:	water	Rotatory Speed	:	75.
Volume	:	900mL	Temperature	:	37
Apparatus	:	USP-II (Paddle)	Time	:	45 min.

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= $\frac{0.7201}{0.6774} \times \frac{32.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 105.07$

Tablet 2= $\frac{0.7492}{0.6774} \times \frac{32.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 109.31$

Tablet 3= $\frac{0.6778}{0.6774} \times \frac{32.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 98.90$

Tablet 4= $\frac{0.7396}{0.6774} \times \frac{32.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 107.91$

Tablet 5= $\frac{0.7459}{0.6774} \times \frac{32.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 108.83$

Tablet 6= $\frac{0.7601}{0.6774} \times \frac{32.43}{100} \times \frac{5}{50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 110.90$

Average:- $\frac{105.07 + 109.31 + 98.90 + 107.91 + 108.83 + 110.90}{6} = \frac{640.92}{6}$

$= 106.82$

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, (150mmX4.6mm), 5-µm
Pump mode	:	Isocratic
Mobile Phase	:	Buffer : Acetonitrile (60:40)
Flow rate	:	1.0 ML/min
Injection volume	:	20µl
Column Temperature	:	30°C
Wavelength	:	UV, 250 nm

Preparation of solutions:

- **Standard preparation:**

wt. and transfer accurately about 20mg. of Teneligliptin (29.48mg teneligliptin Hydrobromide Hydrate) working std. to a 100ml volumetric flask & 70ml of water & sonicate to dissolve & make up the volume.

- **Sample preparation:**

wt. 10 tablet & determine average wt. crush the tablet to a fine powder. wt. & transfer powder equivalent to 20mg teneligliptin to a 100ml dry vol. flask.

Standard Weight :- 29.48
Sample Weight :- 287
Average Weight :- 286.
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) $\frac{13736385}{14490846.5} \times \frac{29.48}{100} \times \frac{100}{287} \times \frac{P}{100}_{(99.85)} \times 286 \times \frac{426.57}{628.86} \times \frac{100}{20}$

$= 94.30\%$

2) $\frac{11546288}{14490846.5} \times \frac{29.48}{100} \times \frac{100}{287} \times \frac{P}{100}_{(99.85)} \times 286 \times \frac{426.57}{628.86} \times \frac{100}{20}$

$= 79.27\%$

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 chromato-	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	1 of 3
Instrument ID : -	ARNI / INS-001	
Name Of Student :-	Chavan Jyoti Ganesh.	

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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	ARNI ANALYTICALS	
TITLE	HPLC DATA SHEET	
Instrument Name :-	HPLC	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	2 of 3
Instrument ID : -	ARNI/INS-001	
Name Of Student :-	Chavan Jyoti Ganesh	

- **CHROMATOGRAPHIC PARAMETERS-2**

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-3**

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisitation Time	:	22 min.
Pump	:	1.20 ml/min
Port	:	A
Detector (Wavelength)	:	250 nm
Column Oven Temperature	:	30°C
Degasser	:	off
Autosampler Temperature	:	15°C.

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	ARNI ANALYTICALS	
TITLE	HPLC DATA SHEET	
Instrument Name :-	HPLC	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	3 of 3
Instrument ID : -	ARNI /INS-001	
Name Of Student :-	chavan Jyoti Ganesh	

- **CHROMATOGRAPHIC PARAMETERS-4**

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.

- **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.

Chavan.

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	ARNI ANALYTICALS	
TITLE	HPLC DATA SHEET	
Instrument Name :-	High Performance liquid chromatog.	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 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 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	Page No
Instrument Name :-	High Performance liquid chromato	
Instrument Make :-	SHIMADZU	1 of 1
Instrument Model No. :-	LC 2010 CHT	
Instrument ID : -	ARNI / INS - 001	

NAME OF STUDENT :- Chavan Jyoti Ganesh.

DATA SHEET

NAME OF TEST :- SYSTEM SUITABILITY

SYSTEM SUITIBILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINCE

- **Chromatographic Conditions:**

Column	
Data Aquisitation Time	10 min
Pump (Flow Rate)	1.00ml /min
Port	A
Detector (Wavelength)	273 nm
Column Oven Temperature	30°c
Degasser	off
Autosampler Temperature	off.

- **MOBILE PHASE PREPARATION** :- Acetonitrile Chavan 19/12/22
  Mix. of water and ~~ethanol~~ prepare in ratio of 7:3

- **STANDARD PREPARATION** :-
  wt. accurately 20mg. of caffeine standard to a 100ml vol. flask, Add 60 ml. of HPLC grade water and shake to dissolve completely. slowly makeup the volume upto the mark. mix well. Further dilute 5ml of the above sol? to 50ml. volumetric flask, dilute with water to makeup volume.

- **SEQUENCE OF INJECTION** :-

Name of Solution	No. Of Injection
Blank	—
Standard	3

Chavan.
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ARNI ANALYTICALS

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

NAME OF STUDENT:- chavan Jyoti Ganesh.

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	200.015g.	199.8000 to 200.2000
2	100.0000	98.772g.	99.9000 to 100.1000
3	50.0000	50.702g.	49.9500 to 50.0500
4	20.0000	19.055g.	19.9800 to 20.0200
5	10.0000	10.000g.	9.9900 to 10.0100
6	5.0000	5.081g.	4.9950 to 5.0050
7	2.0000	1.231g.	1.9980 to 2.0020
8	1.0000	0.925g.	0.9990 to 1.0010
9	0.5000	0.151mg.	0.4995 to 0.5005
10	0.2000	0.105.	0.1998 to 0.2002
11	0.1000	0.057	0.0999 to 0.1001
12	0.0500	0.037	0.0499 to 0.0501
13	0.0200	0.016	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.

Chavan
ANALYSED BY

CHECKED BY:-

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

4. Test for Repeatability :

Selected Weight in g: 20g.

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	19.054 g.	6	19.054g.	± 0.1 %
2	19.055g.	7	19.053	
3	19.055g.	8	19.054	
4	19.054g.	9	19.052	
5	19.055g.	10	19.053	

1) max. = $\frac{19.055 \times 0.1}{100}$ = 0.019055

2) 19.055 + 0.019055
= 19.0740

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

3) 19.052 - 0.019055
= 19.0329

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

Bhavan.

ANALYSED BY

CHECKED BY:-

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

2. Test for Linearity:

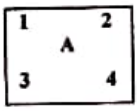
Sr. No.	Selected Weights in g	Observed Weight in g
1	50g.	50.706
2	20g.	19.055
3	10g.	10.002.

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

ANALYSED BY

CHECKED BY:-

3. Test for Eccentricity:



20g.

Sr. No.	Weight Observed in g		Difference in g	Limit
1.	At Centre- (A)	19.056	-	± 0.1 %
2.	At Corner 1 (B)	19.058	B-A = 0.002	
3.	At Corner 2 (C)	19.057	C-A = 0.001	
4.	At Corner 3 (D)	19.057	D-A = 0.001	
5.	At Corner 4 (E)	19.056	E-A = 0	

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

Bhavan

ANALYSED BY

CHECKED BY:-

	ARNI ANALYTICALS	
TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-	Analytical Balance	Page No
Instrument Make :-	WENSAR	
Instrument ID : -	DS 8000	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	199.992 g.	199.8000 to 200.2000
2	100.0000	98.755 g.	99.9000 to 100.1000
3	50.0000	50. 594 g.	49.9500 to 50.0500
4	20.0000	19.050 g.	19.9800 to 20.0200
5	10.0000	9.997 g.	9.9900 to 10.0100
6	5.0000	5.080 g.	4.9950 to 5.0050
7	2.0000	1.230 g.	1.9980 to 2.0020
8	1.0000	0.924 g.	0.9990 to 1.0010
9	0.5000	0.149 g.	0.4995 to 0.5005
10	0.2000	0.104 g.	0.1998 to 0.2002
11	0.1000	0.055 g.	0.0999 to 0.1001
12	0.0500	0.069 g.	0.0499 to 0.0501
13	0.0200	0.015 g.	0.0199 to 0.0200
14	0.0100	0.006 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.**

Bharat

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:

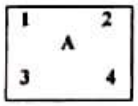
Sr. No.	Selected Weights in g	Observed Weight in g
1	20	19.049
2	10	10.006
3	5	5.079

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

ANALYSED BY **CHECKED BY**

3. Test for Eccentricity:

10g.



Sr. No.	Weight Observed in g		Difference in g	Limit
1.	At Centre- (A)		9.998g.	
2.	At Corner 1 (B)	9.998	B-A = 0	
3.	At Corner 2 (C)	9.999	C-A = 0.001	± 0.1 %
4.	At Corner 3 (D)	9.998	D-A = 0	
5.	At Corner 4 (E)	9.999	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	50.691	6	50.691	± 0.1 %
2	50.691	7	50.693	
3	50.694	8	50.689	
4	50.694	9	50.695	
5	50.691	10	50.692	

1) max = $\frac{50.695 \times 0.1}{100} = 0.0506$

2) $50.695 + 0.0506$
$= 50.7456$

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

mini. = 3) $50.689 - 0.0506$
$= 50.6384$

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

Bhavan.
ANALYSED BY

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-004	

DAILY CALIBATION 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	12-12-22	4.29	6.81

Slope = 84%.

PERFORMED BY

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 CALIBATION 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	3.91	6.82

Slope = 97 %.

Rhawan.
PERFORMED 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	chavan Jyoti Ganesh.	

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	Chavan Jyoti Ganesh	

NAME OF TEST :-

TRIAL FOR DISSOLUTION TEST.

DISSOLUTION CONDITIONS:-

Dissolution Media	water
Media Volume	900 ml
Apparatus	USP type-II Paddle
RPM	100
Temperature	
Time	45 min.

PREPARATIONS:-

- Powr 900 mL of dissolution medium in each vessel.
- Allow sufficient time for dissolution medium
- Adjust stirring element speed to 100 rpm.
- Adjust the paddle in the dissolution medium and the distance bet^{n}. bottom of the paddle and inside bottom of the vessel. start the appratus. at the end of specified time interval, withdraw 10 mL aliquot from ozone.
- Fwether dil. 2ml. of the above solution to 25 ml with dissolution medium.

Chavan:
ANALYSED BY

CHECKED BY:-

	ARNI ANALYTICALS	
TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV - spectrophotometer.	Page No.
Instrument ID : -	ARNI / INS - 002	
Instrument Model No. :-	IMSP - UV - 100B	1 of 1
Name Of Students	chavan Jyoti Ganesh.	

NAME OF TEST :-

1) **PHOTOMETRIC ANALYSIS**
2) **WAVELENGTH SCAN**

PREPARATIONS:-

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 5mlof the above solution to 50ml with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- **273nm**

ANALYSED BY

CHECKED BY:-

ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV- Spectrophotometer.	Page No.
Instrument ID : -	ARNI/INS-002	
Instrument Model No. :-	LMSP-UV 100B	1 of 1
Name Of Students	Chavan Jyoti Ganesh.	

Date:-

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

NAME OF TEST :- wavelength scan

PREPARATIONS:-

STANDARD PREPARATION :-

Wt. Accurately 10mg. of caffeine standard in a 100ml. volumetric flask, add 60ml of water sonicate for 5min. to completely dissolve, makeup the volume with water. Further dil. 5ml of the above sol? to 50ml with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- 273nm

OBSERVATIONS:-

MAXIMUM ABSORPTION WAVELENGTH 273 nm - max.

206nm - min.

Chavan

ANALYSED BY

CHECKED BY:-

„"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.

HOD Chemistry

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

Examiner

Principal

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

ISO 9001 CERTIFIED

ARNI ANALYTICAL

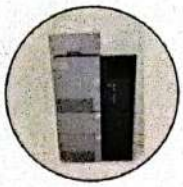
Add.: Pushpak Apartment, Flat No. 102, Lane No. 3, Near Neurocare Hospital, Pandit Colony, Nashik.
e-mail : arnianalyticals@gmail.com | Web Site : www.arnianalyticals.com

Certificate

This is to Certify that ..... *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

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:- Pandey

Name:- Nikita Angad Pandey

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

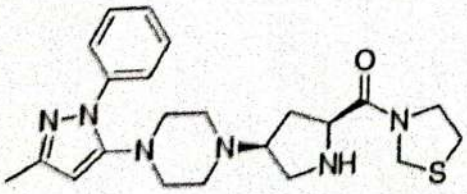
Physical Testing-

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-



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 ± 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)

Area - Std ① 14506216
② 14631085
③ 17812119

1st tray 41
61

Samp ① 12137379
② 10902060
③ 10958003
④ 12445265

15649140

ARNI ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG |

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 |

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

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

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

ARNI ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG |

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 = 3.26 = 1000

- **Preparation of Buffer:** 0.1M = 13.609 = 1000 0.1 = 195

Dissolved 0.1M Potassium dihydrogen orthophosphate in 1000 mL of water; 2.653 gm

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

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:** Eq = 20mg (A.V. Weight of tablet).

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.

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:**

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

Where,

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

Example :

Dissolutⁿ.

500 mg ⟶ 900 ml.

Sample.

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

Stand.

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

Absorbance = 0.476.

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

Formula :-

(1) $= \frac{\text{Sam. Abs}}{\text{Stand. Abs}} \times \frac{\text{Std. wt}}{\text{dilⁿ}} \times \frac{\text{dilⁿ}}{\text{L.C}} \times \text{dilⁿ} \times \frac{P}{100} \times 100.$

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

$= 101.16\ \%$.

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

$= 100.1\ \%$.

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

$= 102.6\ \%$.

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

$= 96.72\ \%$

(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.
	10.3 mg.	10 mg
	100 ml	100 ml.
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 :

Para - 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

Conc^n = 50 ppm
Abs = 0.382

Sample.
500 mg ⟶ 100 ml
1 ⟶ 100 ml

Conc^n = 50 ppm
Abs = 0.379.

formula.

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

$$= \frac{0.379}{0.382} \times \frac{50.2}{100} \times \frac{5}{50} \times \frac{100}{752.7} \times \frac{100}{1} \times 752.3 \times \frac{99.5}{100} \times \frac{100}{500}$$

$$= 99.06\ \%.$$

	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 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

Pandey.

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

- **CHROMATOGRAPHIC PARAMETERS-2**

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisitation 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 Aquisitation 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

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 Aquisitation 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 Aquisitation Time	:	20min
Pump	:	0.00ml/min
Port	:	A
Detector (Wavelength)	:	OFF
Column Oven Temperature	:	OFF
Degasser	:	OFF
Autosampler Temperature	:	OFF.

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	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	5.080g	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	0.152g	0.4995 to 0.5005
10	0.2000	0.104 g	0.1998 to 0.2002
11	0.1000	0.058g	0.0999 to 0.1001
12	0.0500	0.069g	0.0499 to 0.0501
13	0.0200	0.016 g	0.0199 to 0.0200
14	0.0100	0.015g	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.**

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	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.**

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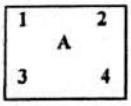
3. Test for Eccentricity:



Sr. No.	Weight Observed in g		Difference in g	Limit
1.	At Centre- (A)	5.082 g	~~5.082 g~~ Nandey 15/12/22	
2.	At Corner 1 (B)	5.082	B-A = 0.000	
3.	At Corner 2 (C)	5.082	C-A = 0.000	± 0.1 %
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.

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	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.

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(90)

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

DAILY CALIBATION 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 %.

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	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:- 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: ± 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.

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	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	200	200.0012
2	100	98.773
3	50	50.702

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

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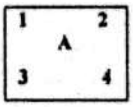
3. Test for Eccentricity:



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

Mandeep
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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 : -	ARNJ/JNS-004	

4. Test for Repeatability :

Selected Weight in g: 5 g

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.

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	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 CALIBATION 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 %.

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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.

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	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°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 25ml with dissolution medium.

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	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 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 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
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	1 of 1
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 Aquisitation 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 :-** S.Pandey 19/12/22

Mixture of Water & ~~Ethanol~~ Aceto nitrile prepare in Proportion of 7:3

- **STANDARD PREPARATION :-**

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

- **SEQUENCE OF INJECTION :-**

Name of Solution	No. Of Injection
Blank	—
Standard	3

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	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 Nibita Angad	

NAME OF TEST :-

1) **PHOTOMETRIC ANALYSIS**
2) **WAVELENGTH SCAN**

PREPARATIONS:-

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 5mlof the above solution to 50ml with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- **273nm**

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

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

OBSERVATIONS:-

MAXIMUM ABSORPTION WAVELENGTH - 274 nm

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FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG |

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)

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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) $= \frac{At}{As} \times \frac{Ws}{100} \times \frac{900}{LC} \times \frac{P}{100} \times \frac{426.57}{628.86} \times 100$

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

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

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

Tablet 6= $\frac{0.6089}{0.6642} \times \frac{32.43 \times 5}{100} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 90.53\%$

91.15 %

Average:- 91.15 %.

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

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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	:	C_{18}, (150mm x 4.6mm), 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 eq^n to 20 mg of Teneligliptin to 100 ml 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:**

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

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

$= 76.77\%$

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

„"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. Snehal Radhakisan Rahane

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. Snehal Radhakisan Rahane** 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

12-05-2023

Examiner

Principal

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

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ARNI ANALYTICAL

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e-mail : arnianalyticals@gmail.com | Web Site : www.arnianalyticals.com

Certificate

This is to Certify that ..... *Snehal Radhakisan Rahane* .....
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. **Snehal Radhakisan Rahane** 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

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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:- Rahane

Name:- Snehal Radhakisan Rahane

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

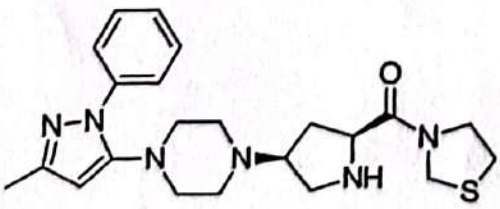
Physical Testing-

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-



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 ± 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.

- **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

ARNI ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG |

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

100 ⇒ 65 : 35
300 ⇒ 195 : 105
Buffer : ACN

13.2 Preparation of Mobile Phase:

- **Preparation of Buffer:**

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.

1M = 136.09 = 1000
0.1M = 13.60 = 1000
0.1 = 13.60 → 1000
0.1 = 2.653 gm → 195
0.1 = 13.60
0.1 = 2.653

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:**

Eq = 20mg (Av weight of 1 tablet)

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.

2 tray → 61
2 tray → 51

- 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

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	:	27	TABLETS-10	:	295

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	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 :- 279 mg
MAXIMUM WEIGHT :- 295 mg

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

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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 ppm
Volume	:	900 ml	Temperature	:	37°C ± 0.5°C
Apparatus	:	USP Type II (Paddle)	Time	:	45 minutes

Standard Weight :- 32.43 mg

Potency:- 99.85

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= $\frac{0.6682}{0.2316} \times \frac{32.43}{100} \times \frac{900}{\frac{99.85}{20}} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = \times \frac{S}{50} =$

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

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

Tablet 4= $\frac{0.5951}{0.6642} \times \frac{32.43}{100} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = \times \frac{S}{50} =$ ~~86.43%~~ 88.55%

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

Tablet 6= $\frac{0.6084}{0.6642} \times \frac{32.43}{100} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 \times \frac{S}{50} =$ 90.55% / 91.15%

Average:- 91.15%

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

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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 X4.6mm), 5um
Pump mode	:	Isocratic
Mobile Phase	:	Buffer : Acetonitrile (65:35)
Flow rate	:	1.0 ml/min
Injection volume	:	20 µl
Column Temperature	:	30°C
Wavelength	:	210nm

Preparation of solutions:

- **Standard preparation:** 20mg of Teneligliptin (29.48mg teneligliptin Hydrobromide hydrate) dissolve in 100ml volumetric flask.
- **Sample preparation:** Weigh 10 tablets & determine average weight Crush tablets to fine powder. Weigh & transfer powder eq. to 20mg of teneligliptin to 100ml volumetric flask. filter through 0.45u nylon filter discarding first few ml of filtrate

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

- **Calculations:**

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

1) $\frac{11145252}{14939435} \times \frac{29.48}{100} \times \frac{100}{286} \times \frac{99.85}{100} \times 286.9 \times \frac{426.57}{628.86} \times \frac{100}{20}$

$= 76.77\%$

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

	ARNI ANALYTICALS	
TITLE	HPLC DATA SHEET	
Instrument Name :-	High Performance Liquid Chromatography (HPLC)	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	1 of 3
Instrument ID : -	ARNI /INS-001	
Name Of Student :-	Rahane Snehal Radhakisan	

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	:	10min
Pump	:	1.00 ml/min
Port	:	A
Detector (Wavelength)	:	210 nm
Column Oven Temperature	:	30°C
Degasser	:	On
Autosampler Temperature	:	10°C

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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 2010 CHT	2 of 3
Instrument ID : -	ARNI / INS-001	
Name Of Student :-	Rohane Snehal Radhakisan	

- **CHROMATOGRAPHIC PARAMETERS-2**

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

- **CHROMATOGRAPHIC PARAMETERS-3**

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

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ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	High Performance Liquid chromatography	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC2010 CHT	3 of 3
Instrument ID : -	ARNI / INS-001	
Name Of Student :-	Rahane Snehal Radhakisan	

- **CHROMATOGRAPHIC PARAMETERS-4**

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

- **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

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	ARNI ANALYTICALS	
TITLE	HPLC DATA SHEET	
Instrument Name :-	High Performance Liquid chromatography	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC2010 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 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 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.

Name:- Rahane Snehal Radhakisan

	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	199.98 g	199.8000 to 200.2000
2	100.0000	98.75 g	99.9000 to 100.1000
3	50.0000	50.69 g	49.9500 to 50.0500
4	20.0000	19.05 g	19.9800 to 20.0200
5	10.0000	10.00 g	9.9900 to 10.0100
6	5.0000	5.080 g	4.9950 to 5.0050
7	2.0000	1.323 g	1.9980 to 2.0020
8	1.0000	0.917 g	0.9990 to 1.0010
9	0.5000	0.151 g	0.4995 to 0.5005
10	0.2000	0.103 g	0.1998 to 0.2002
11	0.1000	0.056 g	0.0999 to 0.1001
12	0.0500	0.037 g	0.0499 to 0.0501
13	0.0200	0.017 g	0.0199 to 0.0200
14	0.0100	0.015 g	0.0099 to 0.0100
15	0.0050	0.005 g	0.0049 to 0.0051

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

Rahane 11/12/22

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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

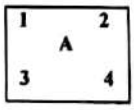
2. Test for Linearity:

Sr. No.	Selected Weights in g	Observed Weight in g
1	50.00 g	50.70 g
2	100 g	98.76 g
3	200 g	200 g

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)	50.70 g		
2.	At Corner 1 (B)	50.69 g	B-A = - 0.01	
3.	At Corner 2 (C)	50.69 g	C-A = - 0.01	± 0.1 %
4.	At Corner 3 (D)	50.69 g	D-A = - 0.01	
5.	At Corner 4 (E)	50.69 g	E-A = - 0.01	

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

11-12-22

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ARNI ANALYTICALS

TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-	Analytical Balance	Page No
Instrument Make :-	WENSAR	
Instrument ID : -	ARNI/ IN6 – 004	3 of 3

4. Test for Repeatability :

Selected Weight in g: 200 gm

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	200.00 gm	6	199.97 g	± 0.1 %
2	199.99 gm	7	199.99 g	
3	199.99 g	8	199.97 g	
4	199.98 g	9	199.98 g	
5	199.98 g	10	199.98 g	

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

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

11-2-22

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	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:-

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	199.96	199.8000 to 200.2000
2	100.0000	99.92	99.9000 to 100.1000
3	50.0000	50.09	49.9500 to 50.0500
4	20.0000	19.06	19.9800 to 20.0200
5	10.0000	10.01	9.9900 to 10.0100
6	5.0000	5.085	4.9950 to 5.0050
7	2.0000	1.9980	1.9980 to 2.0020
8	1.0000	0.982	0.9990 to 1.0010
9	0.5000	0.4992	0.4995 to 0.5005
10	0.2000	0.2000	0.1998 to 0.2002
11	0.1000	0.057	0.0999 to 0.1001
12	0.0500	0.0498	0.0499 to 0.0501
13	0.0200	0.017	0.0199 to 0.0200
14	0.0100	0.0091	0.0099 to 0.0100
15	0.0050	0.005	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 gm	19.057 gm
2	50 gm	50.700 gm
3	100 gm	98.775 gm

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

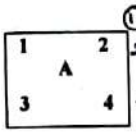
ANALYSED BY

CHECKED BY:-

3. Test for Eccentricity:

B = 98.772
C = 98.773
D = 98.770
E = 98.767

calculation ⇒



① B - A = 98.772 - 98.769 = 0.003
② C - A = 98.773 - 98.769 = 0.004
③ D - A = 98.770 - 98.769 = 0.001
④ E - A = 98.767 - 98.769 = -0.002

Sr. No.	Weight Observed in g	Difference in g	Limit
1.	At Centre- (A)	98.769	= ± 0.1 %
2.	At Corner 1 (B)	B-A = 0.003	
3.	At Corner 2 (C)	C-A = 0.004	± 0.1 %
4.	At Corner 3 (D)	D-A = 0.001	
5.	At Corner 4 (E)	E-A = -0.002	

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. :-	DS8000	3 of 3
Instrument ID : -	ARNI /INS- 004	

4. Test for Repeatability :

Selected Weight in g:

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	98.769	6	98.770	± 0.1 %
2	98.771	7	98.769	
3	98.772	8	98.768	
4	98.771	9	98.770	
5	98.770	10	98.771	

Calculations = $\frac{98.772 \times 0.1}{100} = 0.098772$

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

1> Max value = 98.772 + 0.098772
= 98.8707

2> Min value = 98.768 - 0.098772 = 98.6698

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

ANALYSED BY

CHECKED BY:-

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

DAILY CALIBATION 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>		4.01	6.86

slope ⇒ 98%.

PERFORMED BY

CHECKED BY

Name : Rahane Snehal Radhakisan

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

DAILY CALIBATION 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)	9.2 (± 0.05)
2>	10/12/22	4.02	6.87	9.15

slope ⟹ 99 %.

PERFORMED BY

CHECKED BY

ARNI ANALYTICALS

TITLE	DISSOLUTION TEST APPARATUS WORKSHEET	
Instrument Name :-	DISSOLUTION TEST	Page No.
Instrument ID : -	ARNI / INS - 003	
Instrument Model No. :-	DS - 8000	1 of 1
Name Of Students	Rahane Snehal Radhakisan	

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	Page No.
Instrument ID : -	ARNI / INS - 003	
Instrument Model No. :-	DS-8000	1 of 1
Name Of Students	Rahane Snehal Radhakisan	

NAME OF TEST :-

TRIAL FOR DISSOLUTION TEST.

DISSOLUTION CONDITIONS:-

Dissolution Media	Water
Media Volume	900ml
Apparatus	USP TYPE II (PADDLE)
RPM	100
Temperature	37.0 ± 0.5°C
Time	45 minutes

PREPARATIONS:-

i) Pour 900mL of dissolution medium in each vessel
ii) Allow sufficient time to dissolution medium at equilibrate at 37°C ± 5°C.
iii) Adjust stirring element speed to
iv) Place one capsule in each of six paddle and adjust paddle in dissolution medium, so distance 25mm ±2mm. between bottom of paddle and inside bottom of vessel
v) Start the apparatus.
vi) At end of, withdraw 10mL aliquot from a zone between surface of dissolution medium.

ANALYSED BY

CHECKED BY:-

ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV- SPECTROPHOTOMETER WORKSHEET	Page No.
Instrument ID : -	ARNI /JNG-002-	
Instrument Model No. :-	LMSP - UV - 100 B	1 of 1
Name Of Students	Rahane Snehal Radhakisan	

NAME OF TEST :-

1) **PHOTOMETRIC ANALYSIS**
2) **WAVELENGTH SCAN**

PREPARATIONS:-

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 5mlof the above solution to 50ml with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- **273nm**

ANALYSED BY

CHECKED BY:-

ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV - Spectrometer Worksheet	Page No.
Instrument ID : -	ARNI / INS - 002	
Instrument Model No. :-	UVSP - UV - 100B	1 of 1
Name Of Students	Rahane Shehal Radhakisan	

Date:-

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

NAME OF TEST :- Absorbance of Caffiene

PREPARATIONS:-

STANDARD PREPARATION :-

Weight accurately 10mg of caffine standard in 100 ml volumetric flask. Add 60mL of water sonicate for 5 min to completely dissolve, make up volume with water Further dilute 5mL of above solution to 50 mL with water.

UV-SPECTROPHOTOMETER WAVELENGTH :- 273 nm

1> Weight Caffiene = 10mg

2> Wavelength - 273nm

OBSERVATIONS:- Caffiene Wavelengths - 273nm

MAXIMUM ABSORPTION WAVELENGTH

ANALYSED BY

CHECKED BY:-

„"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. Rohit Vilas Chitalkar

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. Rohit Vilas Chitalkar** 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
G.M.D. Arts, B.W. Commerce
and Science college, Sinnar

Examiner

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 ..... *Rohit Vilas Chitalkar* .....
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. **Rohit Vilas Chitalkar** 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

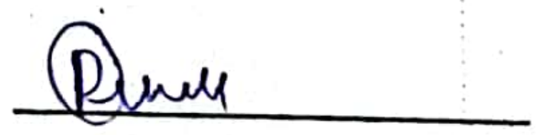
The success and final outcome of this training required a lot of guidance and assistance from many people. All that I have done is only due to such supervision and assistance and I would never forget to thank them.

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:- 

Name:- Rohit Vilas Chitalkar

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

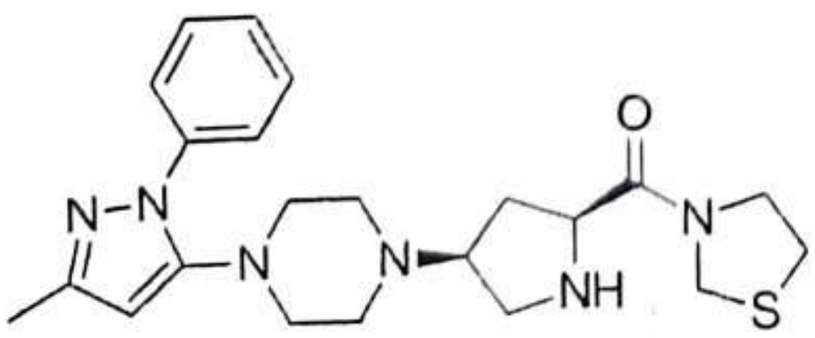
Physical Testing-

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-



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 ± 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.

- **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:**

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:

- **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

ARN ANALYTICAL

FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG |

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
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%**

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 :-
MAXIMUM WEIGHT :-

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 |

5) Dissolution (By HPLC):

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

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= $\frac{0.6045}{0.6642} \times \frac{32.43}{100} \times \frac{5 \times 900}{5020} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 89.95 %.

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

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

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

Tablet 5= $\frac{0.5808}{0.6642} \times \frac{3243 \times 5}{100\ 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 86.43 %.

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

$$\frac{89.95 + 89.19 + 92.81 + 88.55 + 86.43 + 90.53}{6} = 89.57$$

Average:-

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

6) Assay (By HPLC):

Chromatographic Conditions:

Column	:	C_{18} (150mm x 4.6mm)
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 retent^n time of principle Peak

Preparation of solutions:

- **Standard preparation:**

- **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) $\frac{11960800}{11135986} \times \frac{29.43}{100} \times \frac{100}{286.8} \times \frac{P}{100} \times 286.8 \times \frac{426.57}{628.86} \times \frac{100}{20}$ = 104.93%.

=

2) $\frac{11947512}{11135986} \times \frac{29.43}{100} \times \frac{100}{286.8} \times \frac{P}{100} \times 286.8 \times \frac{426.57}{628.86} \times \frac{100}{20}$ 166.92%

=

Average :- 165.92 %.

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

AN	**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	200.0000 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

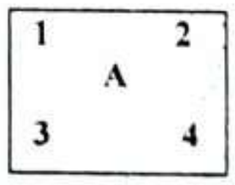
2. Test for Linearity:

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

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 g	
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 :-		
Instrument ID : -		3 of 3

4. Test for Repeatability :

Selected Weight in g: 50 g

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	50.691 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	**DAILY CALIBRATION RECORD OF pH-METER**	
Instrument Name :-		**Page No**
Instrument Make :-		1 of 1
Instrument Model No. :-		
Instrument ID : -		

DAILY CALIBATION 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)		4.02	6.72

slope = 92%

PERFORMED BY

CHECKED BY

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

4. **Test for Repeatability :**

Selected Weight in g:

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

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

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

ANALYSED BY

CHECKED BY:-

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

NAME OF STUDENT:-

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 g	199.8000 to 200.2000
2	100.0000	98.773 g	99.9000 to 100.1000
3	50.0000	50.702 g	49.9500 to 50.0500
4	20.0000	19.053 g	19.9800 to 20.0200
5	10.0000	10.001 g	9.9900 to 10.0100
6	5.0000	5.084 g	4.9950 to 5.0050
7	2.0000	~~1.231~~ g 2.084	1.9980 to 2.0020
8	1.0000	~~1.231~~ g 0.925g	0.9990 to 1.0010
9	0.5000	0.923 g	0.4995 to 0.5005
10	0.2000	0.109 g	0.1998 to 0.2002
11	0.1000		0.0999 to 0.1001
12	0.0500		0.0499 to 0.0501
13	0.0200		0.0199 to 0.0200
14	0.0100		0.0099 to 0.0100
15	0.0050		0.0049 to 0.0051

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

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	Chitalkar Rohit vilas	

NAME OF TEST :-

TRIAL FOR DISSOLUTION TEST.

DISSOLUTION CONDITIONS:-

Dissolution Media	water
Media Volume	900 ml
Apparatus	Use type II Paddle
RPM	100
Temperature	37.0 ± 0.5°C
Time	45 minutes

PREPARATIONS:-

ANALYSED BY

CHECKED BY:-

BIO-INSPIRED SYNTHESIS OF MIXED-PHASE Fe_2O_3 MAGNETIC NANORODS AS A NOVEL RECOVERABLE HETEROGENEOUS CATALYST FOR BIGINELLI SYNTHESIS

THE PROJECT SUBMITTED TO

SAVITRIBAI PHULE PUNE UNIVERSITY, PUNE (M.S)

FOR THE AWARD OF M.Sc. ORGANIC CHEMISTRY IN THE FACULTY OF SCIENCE & TECHNOLOGY

SUBMITTED BY

MR. ROHIT GANESH KOKATE

UNDER THE GUIDANCE OF

MR. DNYANESHWAR SANAP

ASSISTANTS PROFESSOR , DEPARTMENT OF CHEMISTRY
G.M.D. ARTS, B.W. COMMERCE AND SCIENCE COLLEGE, SINNAR, TAL-SINNAR, DIST-NASHIK - 422103

WORK PLACE

POST GRADUATE DEPARTMENT OF CHEMISTRY AND RESEARCH CENTER,
G.M.D. ARTS, B.W. COMMERCE AND SCIENCE COLLEGE, SINNAR, TAL-SINNAR, DIST-NASHIK - 422103

APRIL -MAY 2023

MARATHA VIDYA PRASARAK SAMAJ, NASHIK

G.M.D. ARTS, B.W. COMMERCE AND SCIENCE
COLLEGE, SINNAR, TAL-SINNAR, DIST-NASHIK - 422103

CERTIFICATE

DEPARTMENT OF CHEMISTRY

This is to certify that the work incorporated in the project entitled

Bio-inspired synthesis of mixed-phase Fe_2O_3 magnetic nanorods as a novel recoverable heterogeneous catalyst for Biginelli Synthesis

Was satisfactorily carried out by **Mr. Rohit Ganesh Kokate** of **M.Sc. Organic Chemistry.** He has completed this project under my supervision and guidance during **Academic Year 2022-2023**. This project work submitted by his original and the scientific information obtain from other sources have been duly acknowledged.

Mr. Dnyaneshwar Sanap

(Project Guide)

Maratha Vidya Prasarak Samaj's

G.M.D. ARTS, COMMERCE AND SICENCE COLLGE,

SINNAR DIST. NASHIK

DEPARTMENT OF CHEMISTRY (PG)

CERTIFICATE

This is to certify that **Mr.Kokate Rohit Ganesh** of M.Sc. II (Organic Chemistry) has satisfactorily completed the project work in **Organic Chemistry** during the semester IV of academic year 2022-2023.

Date : 12-05-2023

Place : Sinnar

ARTS, B.W.-COM. & SCIENCE COLLEGE * G.M.D. SINNAR 422 103

Project Incharge

12-05-2023

Examiner

HoD Chemistry

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

Principal

PRINCIPA
G.M.D. Arts, B.W. Com
Science College, Sinnar

ACKNOWLEDGEMENT

- I would like to thank M.V.P. Samaj's G.M.D. Arts B.W. Commerce and Science College Sinnar, for providing me with the necessary resources, infrastructural, and facilities to carry out this project.

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- I would like to thank, HoD Prof. M.R. Gaware for his constructive help during project.

- I would like to thank, **Mr. Dnyaneshwar Sanap** (Project Guide), for their constant encouragement, insightful feedback, and expert guidance that helped me to navigate through the various challenges of this project.

- I would like to thank all the faculty members of the Department of Chemistry, for imparting me with the theoretical and practical knowledge necessary to complete this project.

- I would like to thank my classmates and colleagues, for their feedback, suggestions, and stimulating discussions that enriched my understanding of the subject matter.

Bhokarte.

Table of Content

Bio-inspired synthesis of mixed-phase Fe_2O_3 magnetic nanorods as a novel recoverable heterogeneous catalyst for Biginelli synthesis

Abstract

For the first time, mixed-phase (Hematite and Maghemite) magnetic Fe_2O_3 nanorods were successfully biosynthesis by sol-gel auto-combustion method using the 1:1 mixture of *Eucalyptus citriodora* and *Murraya koenigii* leaf extract as a capping agent, and its catalytic effect on synthesis of ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate (THPMs) derivatives were investigated. Further, the phase formation, surface topography, and crystallinity of biosynthesized Fe_2O_3 nanorods (NRs) were explored using powder XRD (X-Ray Diffraction), UVDRS (UV-Visible Reflectance Spectroscopy), FTIR (Fourier Transform Infrared Spectroscopy), FESEM (Field Emission Scanning Electron Microscopy), EDX (Energy Dispersive X-Ray), and VSM (Vibrating Sample Magnetometry). Furthermore, the catalytic activity of biosynthesized Fe_2O_3 NRs was examined for one-pot synthesis of ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate *via* Biginelli reaction. To achieve high yields (93-99 %) of ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate derivatives, this heterogeneous catalytic method is used with a wide range of aromatic aldehydes within a minimum reaction time, simple reaction work-up, and easily recoverable catalyst by an external magnet. The recovered catalyst is then employed for five successive cycles without non-noticeable loss of catalytic activity. We believe that this protocol presents a broad scope for Biginelli reaction through greenly produced and magnetically separable heterogeneous catalysts.

Keywords: Green synthesis; Biginelli reaction; Fe_2O_3 NRs; Heterogeneous catalysis

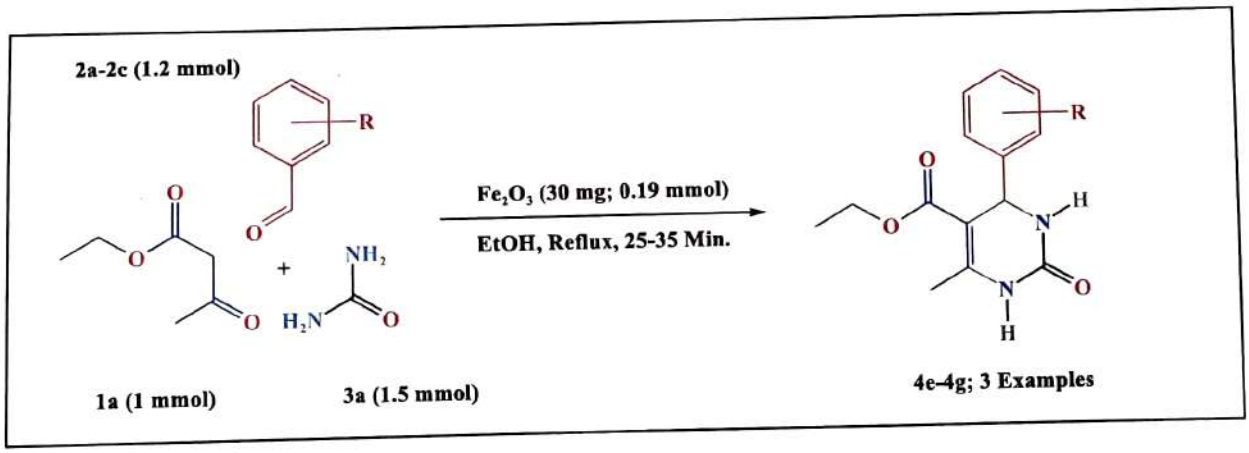
1. Introduction

The 2-oxo-1,2,3,4-tetrahydropyrimidines-THPMs (The former name is 3,4-dihydropyrimidin-2(1H)-ones-DHPMs) Safford's are an important class of heterocyclic compounds known for a wide range of biological activities including anti-HIV [1], antibacterial, antifungal, anti-tubercular, antioxidant, anti-inflammatory [2], and anticancer [3] as well as potential agents in the treatment of many diseases including diabetes, Alzheimer's disease [4, 5], and other neuros diseases [3].

THPMs are a unique scaffold for constructing and creating biologically important medicines and agents for anti-tumour [3], and Alzheimer's disease [4, 5] by changing the substituent on its C6 position.

Conventionally, the THPMs Safford's were synthesized by the well-known Biginelli reaction by three components reaction between aldehyde, ethyl acetoacetate, and urea assisted by hydrochloric acid as a homogeneous catalyst [6] with the yield of 72-92 %.

However, there are five major challenges embedded in the synthesis of THPMs Safford's: 1) use of hazardous solvents; 2) use of hazardous reagents; 3) low to moderate yield; 4) long reaction time; 5) recovery of homogenous catalyst. Herein, we report the successful synthesis strategy for the THPMs Safford's synthesis, using heterogeneous - magnetically separable biosynthesized Fe_2O_3 catalyst, with the yield in the range of 93-99 % (**Scheme 1**). The structure of the synthesized products was confirmed by ^1H NMR and ^{13}C NMR.



Scheme 1: Strategies for the construction of ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate

2. Experimental

2.1 Chemical/materials

Eucalyptus citriodora (Nilgiri Plant Leaf), *Murraya koenigii* leaf (Curry Leaf), Ferric nitrate nonahydrate ($Fe(NO_3)_3.9.H_2O$), substituted aldehydes, ethyl alcohol, ethyl acetate, and n-hexane were purchased from S.D. Fine Chemical Limited. Before being used in this investigation, all the solvents were distilled off.

2.2 *Preparation of leaves extracts*

The fresh leaves of *Eucalyptus citriodora* and *Murraya koenigii* were collected from the campus of G.M.D. Arts B.W. Commerce and Science College Sinnar, Nashik (Maharashtra), were washed thoroughly with distilled water to remove mud, dirt, impurities, and surface contamination. The wash leaves were weighed accurately at 8 gm each, cut into small pieces and pulverized using the mixer in 10 mL distilled water separately. Transfer the pest to a 100 mL RB flask and add 30 mL of distilled water to each pest mixture, then gently heat at about 80-90 ^{0}C for 1 h, then cool it, and filter it with Whatman filter paper (No. 41). The obtained filtrates use for the synthesis of Fe_2O_3 NRs after mixing.

2.3 *Synthesis of Fe_2O_3 Magnetic NRs*

Fe_2O_3 Magnetic NRs were successfully fabricated by a simple sol-gel auto-combustion technique using the $Fe(NO_3)_3.9.H_2O$, *Eucalyptus citriodora,* and *Murraya koenigii* leaves extract precursors.

5 g of $Fe(NO_3)_3.9.H_2O$ was dissolved in 15 mL of distilled water, and stir it for the next 30 min at 70-80 ^{0}C, to the same homogeneous salt solution, 50 mL of the mixed leaves extract (1:1) added drop by drop over 40 min at 70-80 ^{0}C, stirring of mixture was continued for next 30 min upon addition of leaves extract is over. After that, the resulting solution was kept on a hot plate for the next 3 h at about 70-80 ^{0}C to get a thick paste. The paste obtained was heated to 100-120 ^{0}C for 5 h in a hot air oven for auto-combustion to get a dried thick mass. Further dried mass, grind in mortar-pestle for 30 min to get dry powder. The obtained dried Fe_2O_3 powder was calcinated at 600 ^{0}C for 3 h. Finally, the Gray Fe_2O_3 powder obtained. The obtained nanocrystalline Fe_2O_3 screen for the synthesis of ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate-THPMs derivatives.

2.4 *General procedure for* **ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate** *synthesis*

A mixture of freshly distilled Ethyl acetoacetate (1 mmol), aromatic carbaldehyde (1.2 mmol), Urea (1.5 mmol), and biosynthesized Fe_2O_3 NRs (0.19 mmol) in ethyl alcohol (3 mL) was well refluxed in a 25 mL RB flask under dark condition (**Table No. 3,** Entry 1 to 3). TLC was used to track the development of the reaction (using n-Hexane and Ethyl acetate in 1:1 proportion). Upon the completion of the reaction, the reaction mixture is kept near an external magnet for

5 to 7 min to separate the Fe_2O_3 catalyst, and then the ethanolic layer of the reaction mixture is separated by decanting into a beaker through Whatman filter paper. In the end, the catalyst in RB was washed using 1 mL ethanol every time thrice, dried, activated, and used for a sequential cycle. After column chromatography, the pure product was obtained using n-hexane and ethyl acetate (85:15 to 40:60) solvent as a mobile phase. By using ^1H NMR, and ^{13}C NMR techniques, all the organic products were confirmed.

2.5 Fe_2O_3 MNRs characterization technique

Model V-770 - Jasco Spectrophotometer was used to get a UV-DRS of Fe_2O_3 NRs. The UV-DRS absorption spectra were measured from 200 to 800 nm. A JASCO-4600, Type-A model spectrophotometer was used to acquire FT-IR spectra of NRs in the 400 to 4000 cm^{-1} range. Bruker D8 diffractometer having Cu-Kα radiation (having $\lambda = 1.54060\ A^0$) with an angle between 20^0 to 80^0, with a minimum step size of 2θ is 0.020^0 were carried out for crystal structure analysis of biosynthesized NRs. 10 kV accelerating voltage FEI Nova Nano SEM 450 instrument was used to examine cross-section morphology or surface structure and element mapping of NRs. Quanta-Chrome NOVA 1000e model instrument was used to acquire the data of N_2 adsorption-desorption, pore-sized distribution, and pore diameter at 77 K. The magnetic behavior of biosynthesized NRs was investigated using Vibrating Sample Magnetometry. Thiele's tube assembly measured the melting points of the synthesized ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate scaffold. Finally, the Bruker Advance NEO 500 MHz Spectrometer was used to confirm the molecular structure of synthesized ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate scaffold with the instrument strength of 500 MHz and 126 MHz for ^1H and ^{13}C nuclei, respectively.

3. Result and discussion

3.1 Physical, chemical, and structural characterization of the catalyst

The powder X-Ray Diffraction pattern of biosynthesized Fe_2O_3 demonstrates that the sample contains Hematite and Maghemite mixed phase, with rhombohedral and cubic crystal systems [8]. **Figure 1** revealed characteristic X-Ray Diffraction peaks at 2θ: 24.18 (110), 33.20 (211), 35.68 (101), 40.92 (210), 49.53 (202), 54.15 (312), 64.10 (211), 72.01 (433), and 26.18 (211), 30.23 (202), 35.72 (311), 43.42 (400), 46.20 (411), 57.44 (511), 63.01 (404), 74.66 (533) for Hematite and Maghemite phase respectively. This result of the mixed phase is in good agreement with previous studies for Hematite [9, 10] and Maghemite [11], respectively. The

average crystalline size of mixed-phase biosynthesized NRs was calculated using Scherrer's equation by Full Width at Half Maximum (FWHM) for prominent peaks, which were found to be 28.48 nm (**Table 1**).

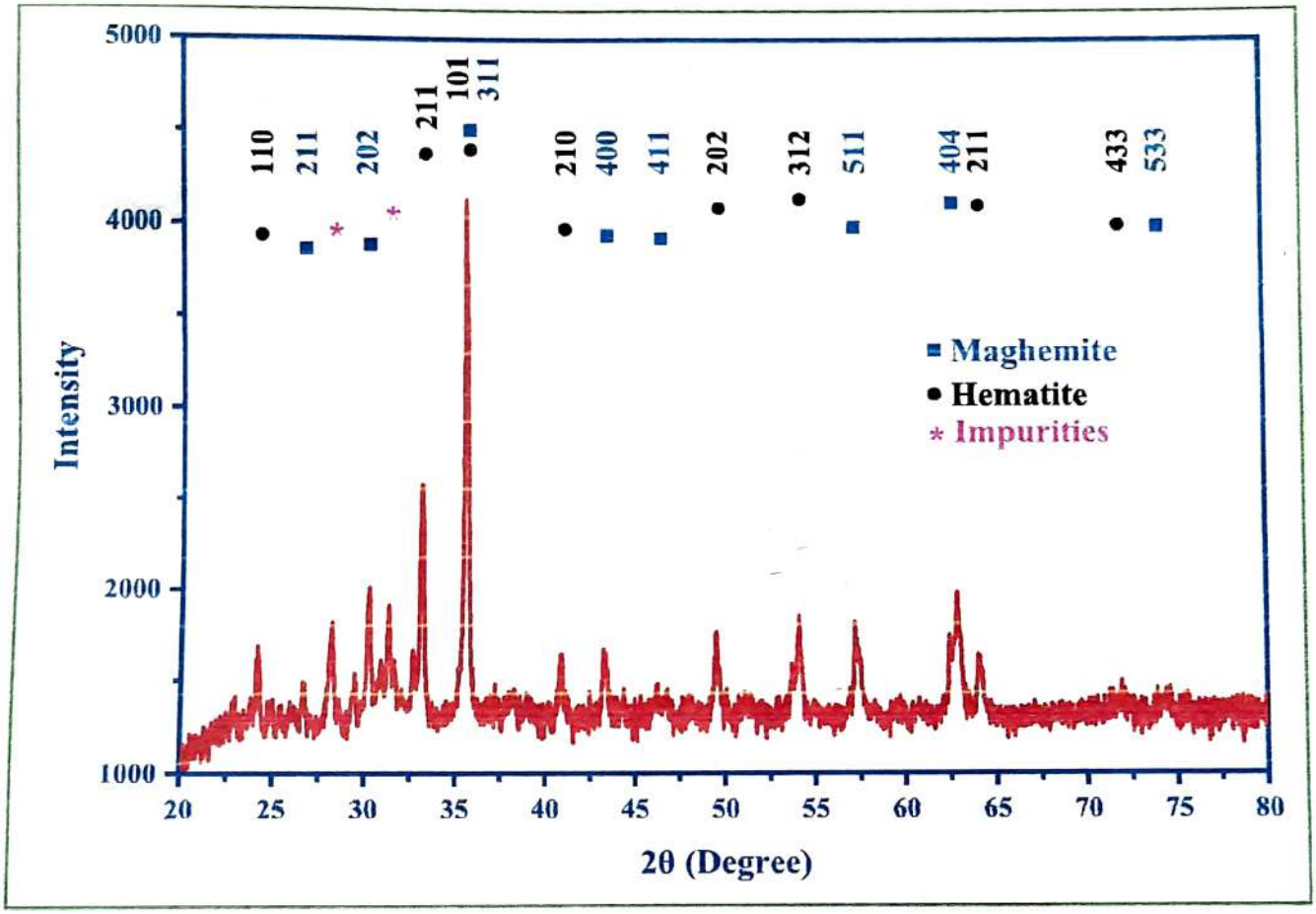


Figure 1: XRD patterns of biosynthesized Fe_2O_3 NRs

2θ (Degree)	FWHM	Crystalline Size D (nm)	Average D (nm)
24.17	0.2336	34.79	**28.48**
33.18	0.2417	34.29	
35.71	0.2772	30.10	
40.90	0.2969	28.55	
43.37	0.2988	28.61	
49.51	0.3114	28.09	
54.14	0.5161	17.28	
57.41	0.4408	20.54	
62.95	0.2507	37.06	
64.07	0.3672	25.52	

Table 1: Calculation of average crystal size of biosynthesized Fe_2O_3 NRs by Scherer equation using FWHM for listed prominent peaks

Further morphological characteristics, sizes, and element mapping of fabricated Fe_2O_3 NRs were studied by FESEM analysis, as shown in **Figure 2.** According to FESEM images, the rod-shaped morphology and uniform distribution were obtained Fig. 2(a-b) as-prepared NRs.

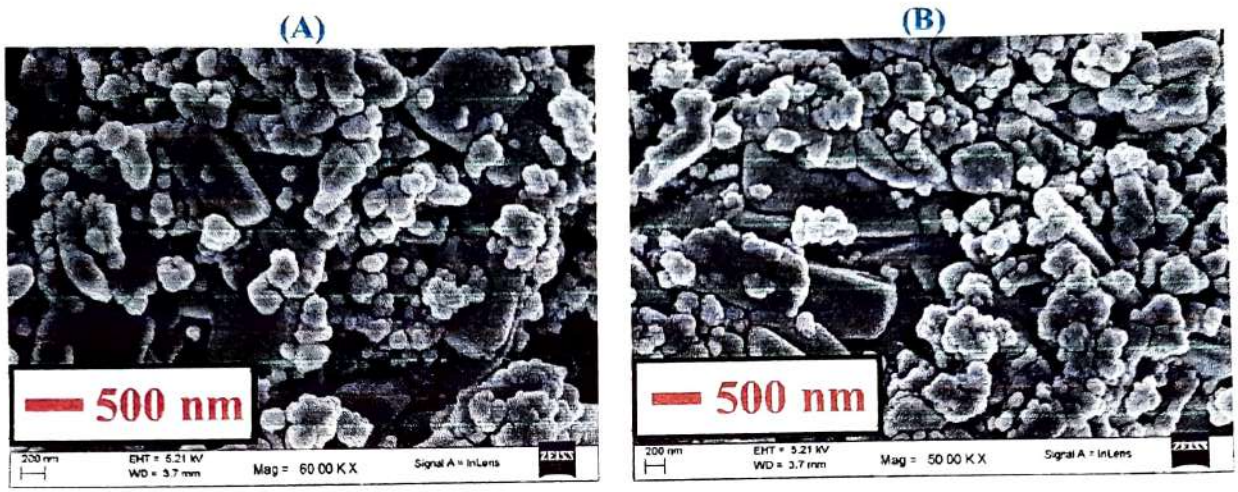
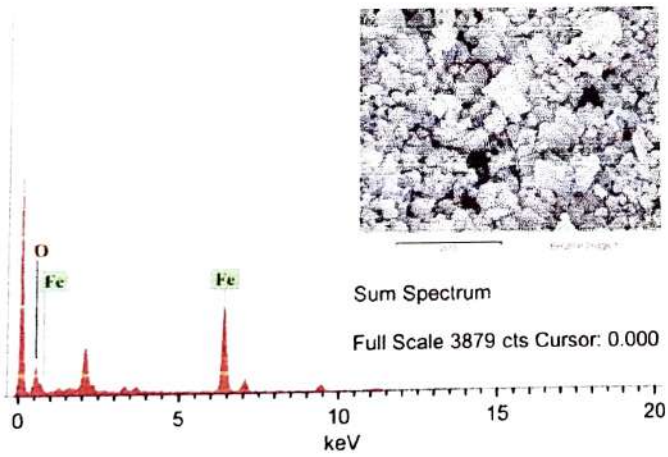


Figure 2: FE-SEM images of as-prepared Fe_2O_3 NRs (a-b).

Additionally, as demonstrated in **Figure 3a,** the EDX spectrum indicated the Fe_2O_3 NRs elemental composition. This EDX analysis (**Fig. 3b**) shows that the material contains Iron (Fe) and Oxygen (O), demonstrating the formation of Fe_2O_3 NRs.



(B) Element	Weight %	Atomic %
Fe	79.49	52.61
O	20.51	47.39

Figure 3: EDX spectrum of synthesized Fe_2O_3 NRs (a); Percent element composition of Fe_2O_3 NRs (b).

The FTIR spectrum was used to investigate the nature of chemical bonding in the molecule. **Figure 4**, FTIR spectrum of biosynthesized Fe_2O_3 NRs, shows intense absorption peaks at 1581, 1095, 594, and 532 cm^{-1}. The peak at 1581 cm^{-1} may correspond to the amide bending vibration of proteins N-H bond or may be due to the stretching vibration of aromatic C=C bonds [12]. The next observed peaks at 1095 cm^{-1} could be caused by aliphatic ether C-O

stretching [12]. The two strong peaks of stretching vibration at 594 and 532 cm^{-1} correspond to Fe-O bond formation in Fe_2O_3 NRs [13-14].

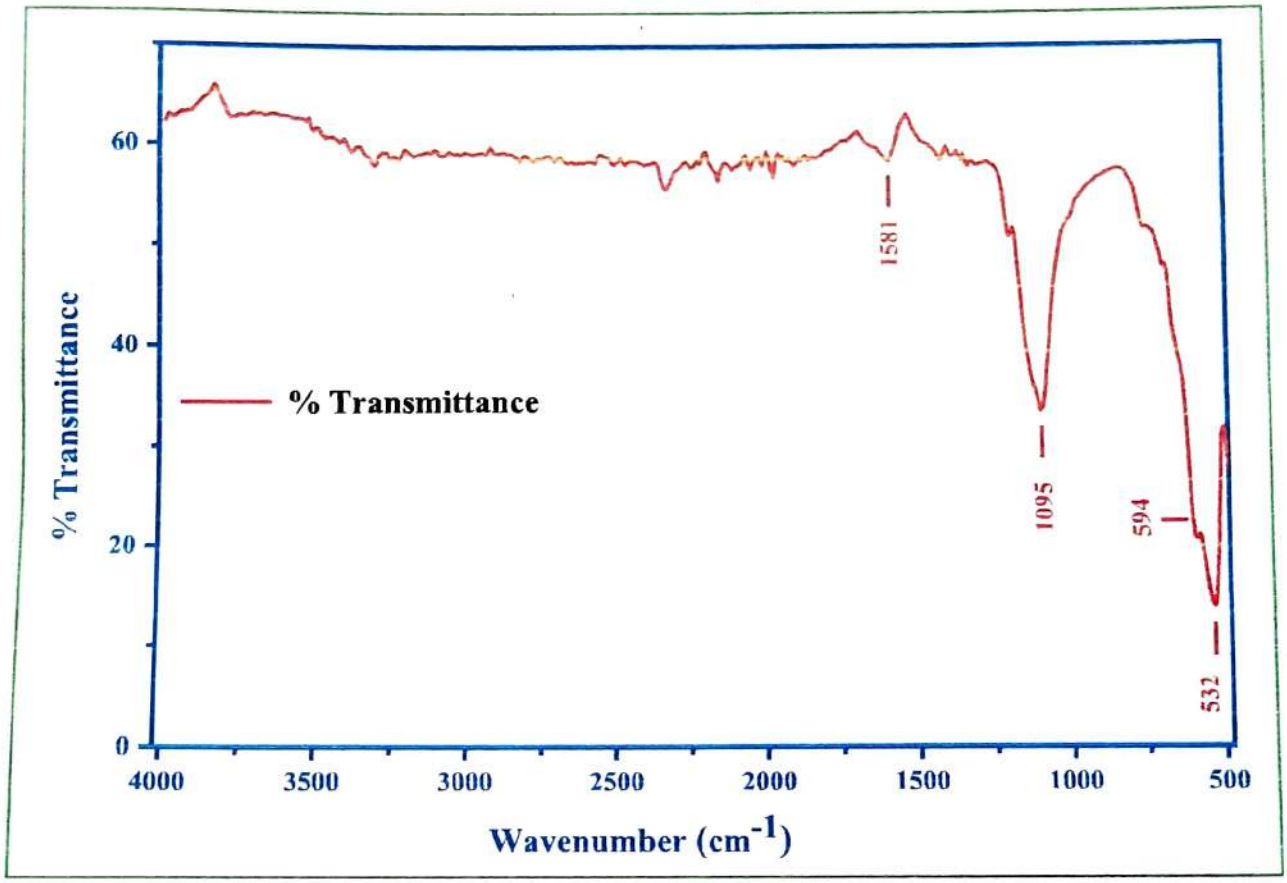


Figure 4: FTIR spectrum of biosynthesized Fe_2O_3 NRs

Figure 5 illustrates the main active phytochemicals found in *Eucalyptus citriodora* (**Fig. 5a**), and *Murraya koenigii* (**Fig. 5b**) leaves to explain how the metal precursor salt $Fe(NO_3)_2$ 9 H_2O can change into Fe_2O_3 nanorods. The plausible reaction mechanism demonstrates how different active phytochemicals act as stabilizing and reducing agents. Diverse active phytochemicals are found in *Eucalyptus citriodora* and *Murraya koenigii*, including saponins, carbohydrates, sterols, glycosides, alkaloids, flavonoids, terpenoids, and polyphenols [15-16]. For a possible reaction mechanism, a flavonoid has been chosen as a sample molecule to suggest the mechanism. Because of the electrostatic attraction between hydroxyl groups of flavonoid and cation of metal precursor, aromatic hydroxyl groups cling to ferric ions (Fe^{3+}), and ferric ions and flavonoids form a stable complex. After the treatment of calcination, the complex decomposes and forms Fe_2O_3 nanorods. As a result, the single-phase formation of Fe_2O_3 is not selective. Therefore, two phases were observed in the XRD analysis corresponding to α-Fe_2O_3 and γ-Fe_2O_3 (**Figure 6**).

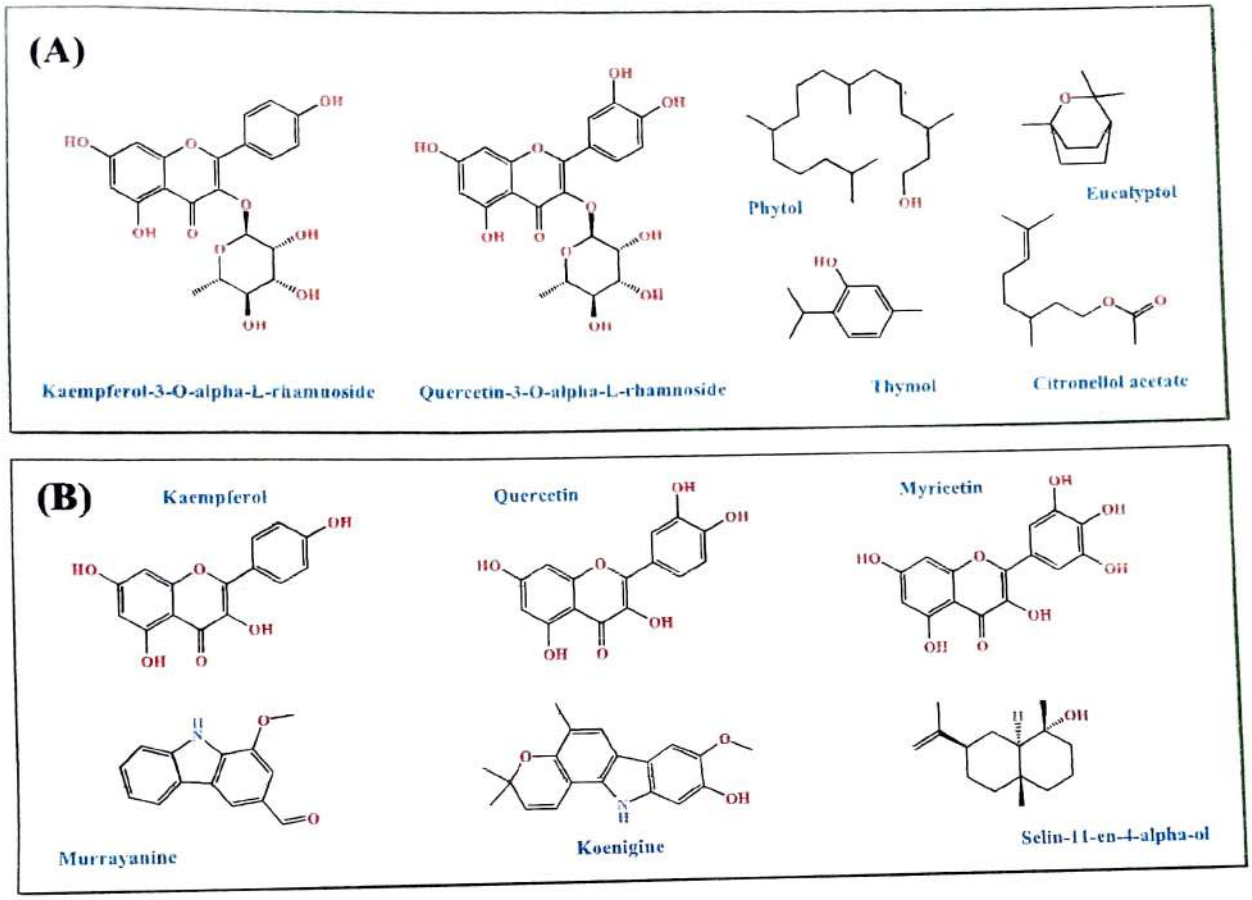


Figure 5: Biologically active phytochemicals present in a) *Eucalyptus citriodora* and b) *Murraya koenigii*

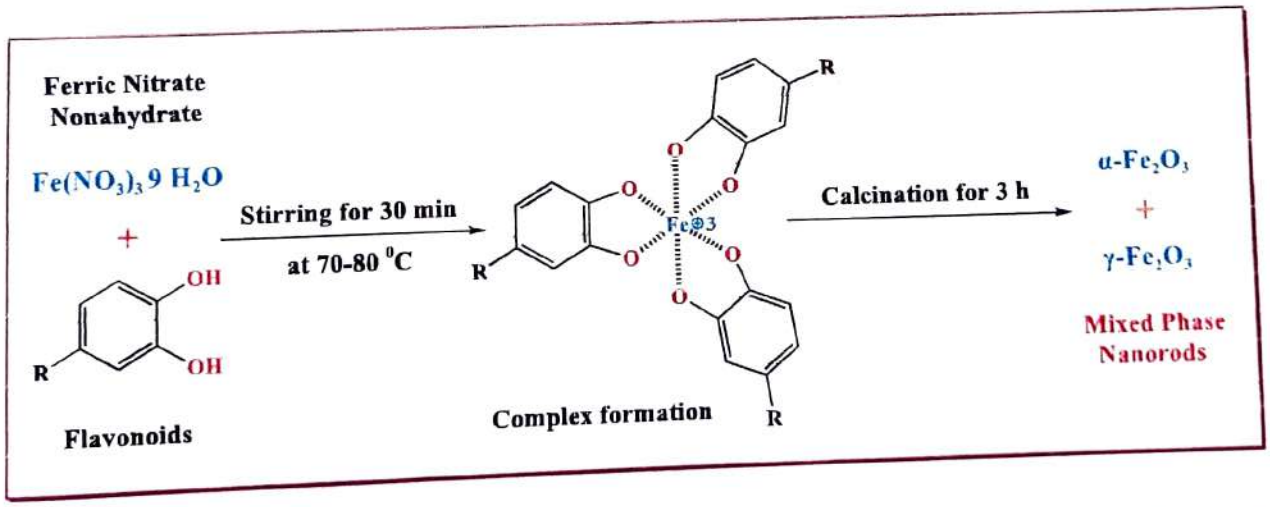


Figure 6: A plausible reaction mechanism for the formation of Fe_2O_3 NRs using metal salt and leaf extracts

The optical absorbance of the fabricated mixed-phase Fe_2O_3 NRs (α-Fe_2O_3-Hematite and γ-Fe_2O_3-Maghemite) was investigated in the range of 200 to 800 nm [17]. The corresponding spectrum is shown in **Figure 7a**. The absorption spectra of a Fe_2O_3 NRs show the presence of

three distinct adsorption zones [18], namely, the first region (200-400 nm) due to charge-transfer from ligands to Fe^{3+} metal ion and because of the Fe^{3+} ligand field transitions contributions, in part from $^6A_1 \rightarrow {}^4T_1(^4P)$ at 290-310 nm, $^6A_1 \rightarrow {}^4E(^4D)$ and $^6A_1 \rightarrow {}^4T_2(^4D)$ at 360-380 nm. The second region (400-600 nm) represents the end consequence of pair excitation of processes $^6A_1 + {}^6A_1 \rightarrow {}^4T_1(^4G) + {}^4T_1(^4G)$ at 485-550 nm and overlapped contribution of $^6A_1 \rightarrow {}^4E, {}^4A_1(^4G)$ ligand field transitions at 430 nm and the charge-transfer band tail. Region third (600-750 nm) is attributed to 640 nm for the $^6A_1 \rightarrow {}^4T_2(^4G)$ transition. Furthermore, according to the selection rules, the area's first and second absorption intensity is significantly higher than that of the third region, indicating that the absorption from charge-transfer transitions or pair excitations is much stronger than that from ligand field transitions [19]. The bandgap energy of biosynthesized Fe_2O_3 NRs is calculated using Tauc's plot (direct method) **(Figure 7b)** and was estimated to be 1.98 eV.

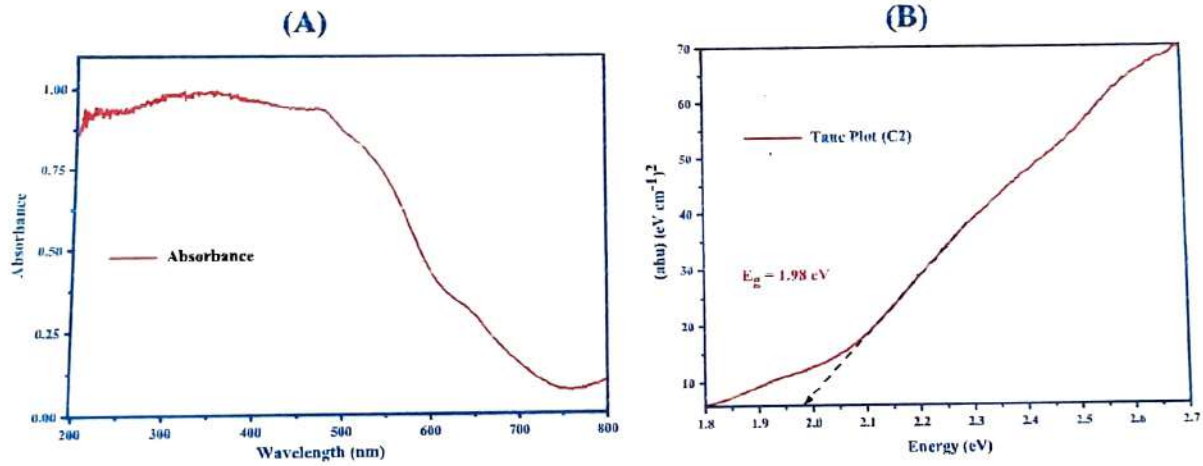


Figure 7: a) UV-DRS spectrum of bio-synthesized Fe_2O_3 NRs; Band-gap energy by Tauc plot (b)

Surface area and porosity of Fe_2O_3 NRs were calculated by Brunauer-Emmett-Teller (BET) N_2 adsorption/desorption studies to get an idea of the absorbance capacity of biosynthesized Fe_2O_3 NRs, and Pore Size distribution (PSD) was obtained by Barrett-Joyner-Halenda (BJH) plot **(Figure 8)**. The surface area of the as-formulated sample was observed to be 3.2097 $m^2 g^{-1}$.

The magnetic behavior of biosynthesized Fe_2O_3 NRs was investigated by Vibrating Sample Magnetometry (VSM). **Figure 9** shows the hysteresis loop for synthesized Fe_2O_3 NRs. The value of Ms, Hc, and Mr **(Table 2)** were estimated based on the hysteresis loop by applying the applied field in the range of -15 kOe to 15 kOe to study the magnetic characteristics of NRs.

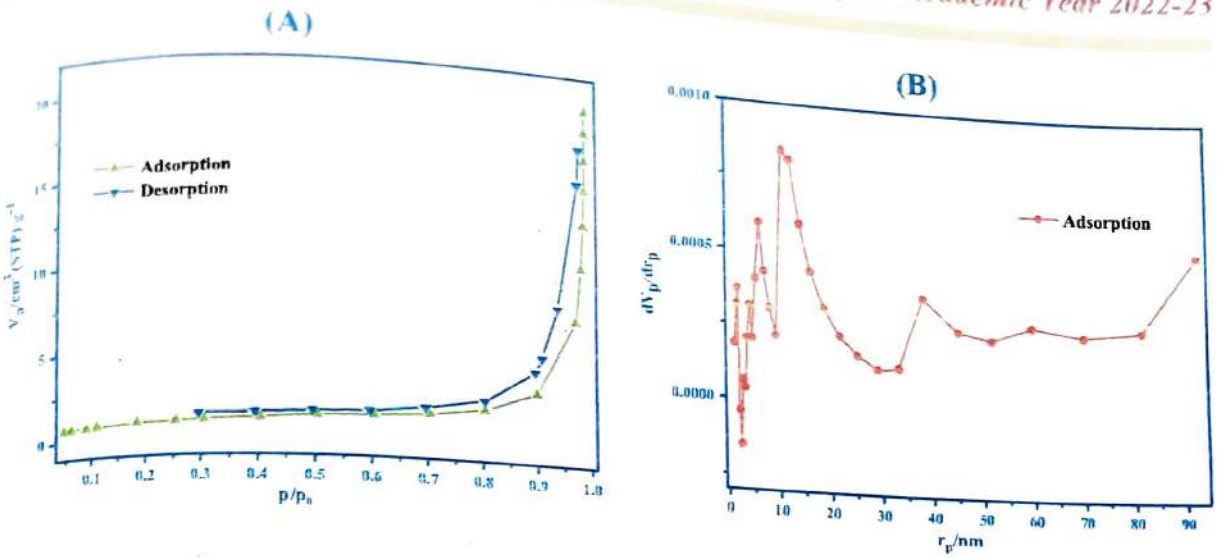


Figure 8: a) Nitrogen adsorption-desorption isotherms at -196 ^{0}C (BET plot); b) Pore size distribution plot of Fe_2O_3 NRs (BJH plot)

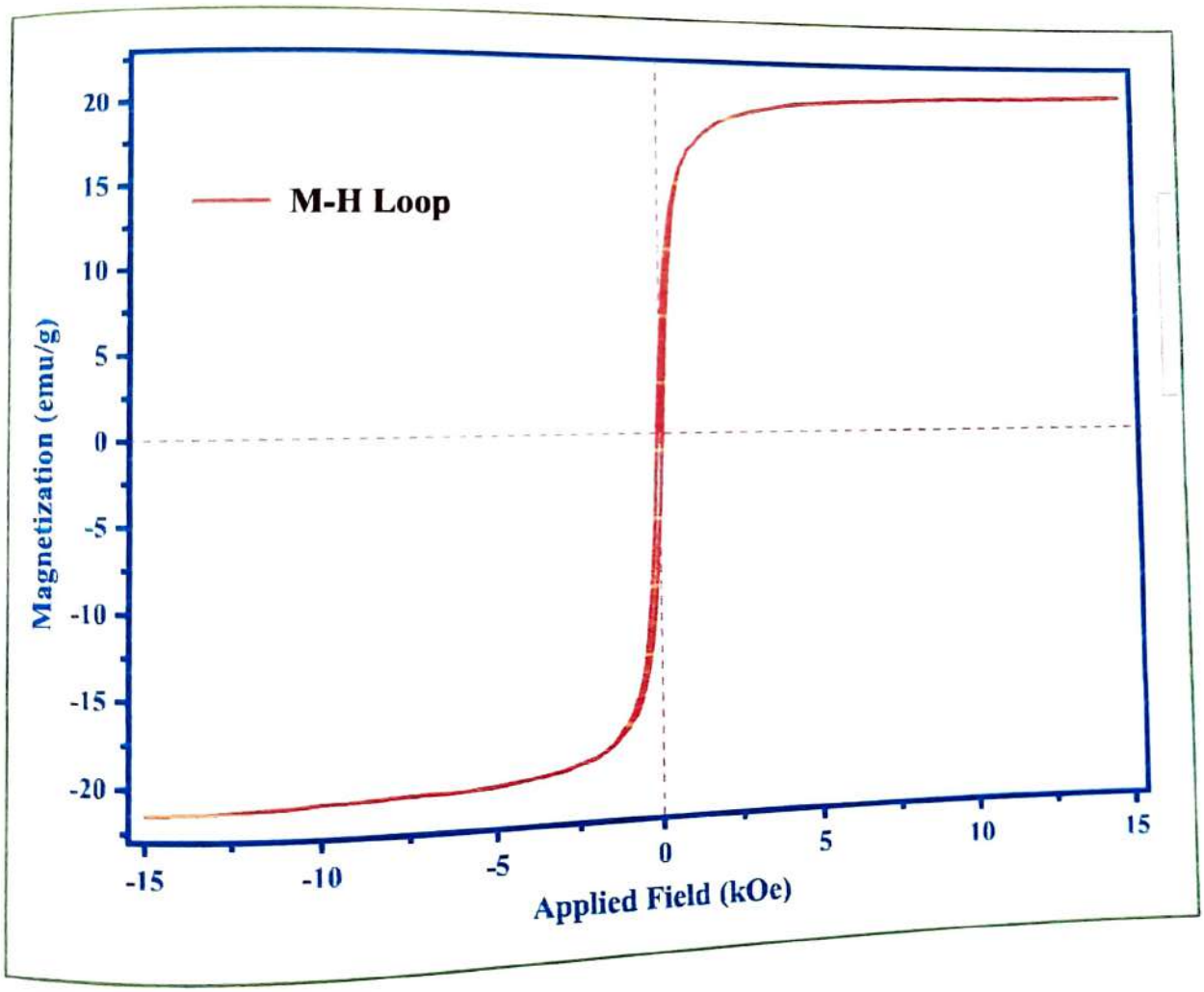


Figure 9: VSM plot of biosynthesized Fe_2O_3 NRs

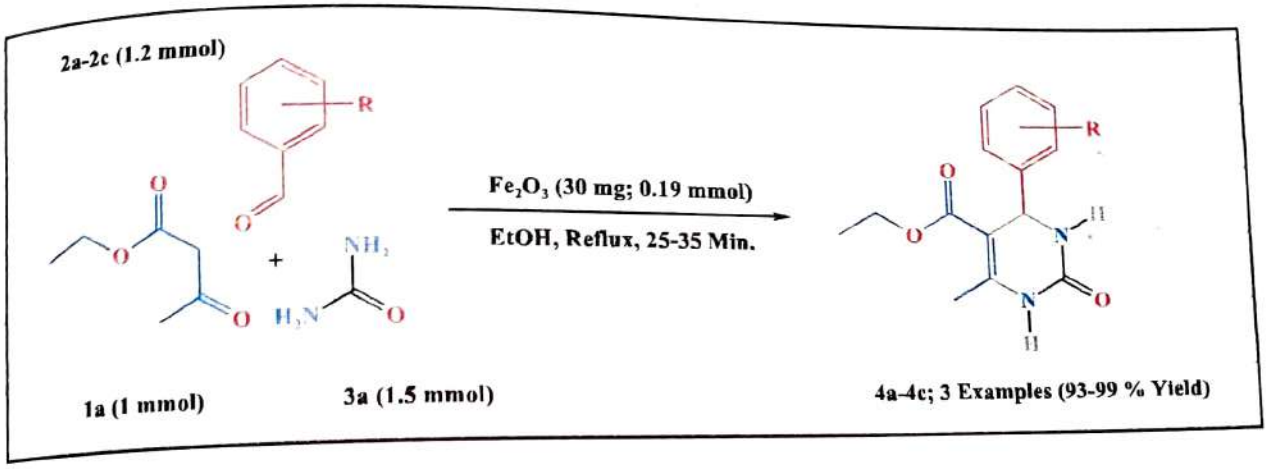
Saturation magnetization (Ms)	Coercivity (Hc)	Remnant field (Mr)
21.16 emu/g	86.98 Oe	4.44 emu/g

Table 2: Summery for Magnetic properties of biosynthesized Fe_2O_3 NRs

3.2 *Synthesis of ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate derivative*

Synthesis and purification of ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate derivative was carried out according to section 2.4 procedure.

Table 3: Synthesis of ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate derivative



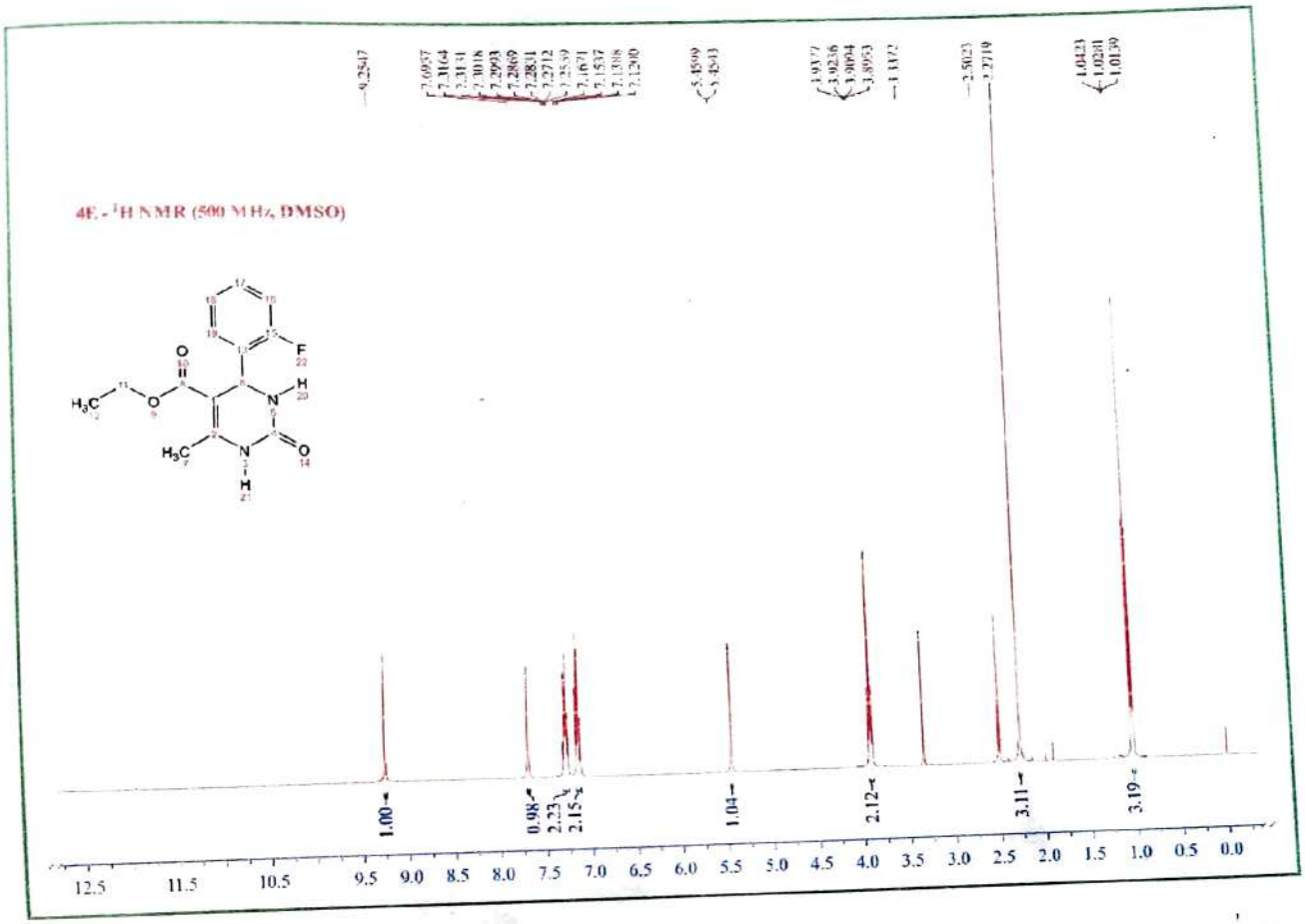
Entry [a]	Aryl Carbaldehyde	Product Code	Time (Min)	Yield (%) [b]
1	2-fluoro Phenyl	4e	35	93
2	2-Bromo Phenyl	4f	35	99
3	4-Hydroxy Phenyl	4g	35	96

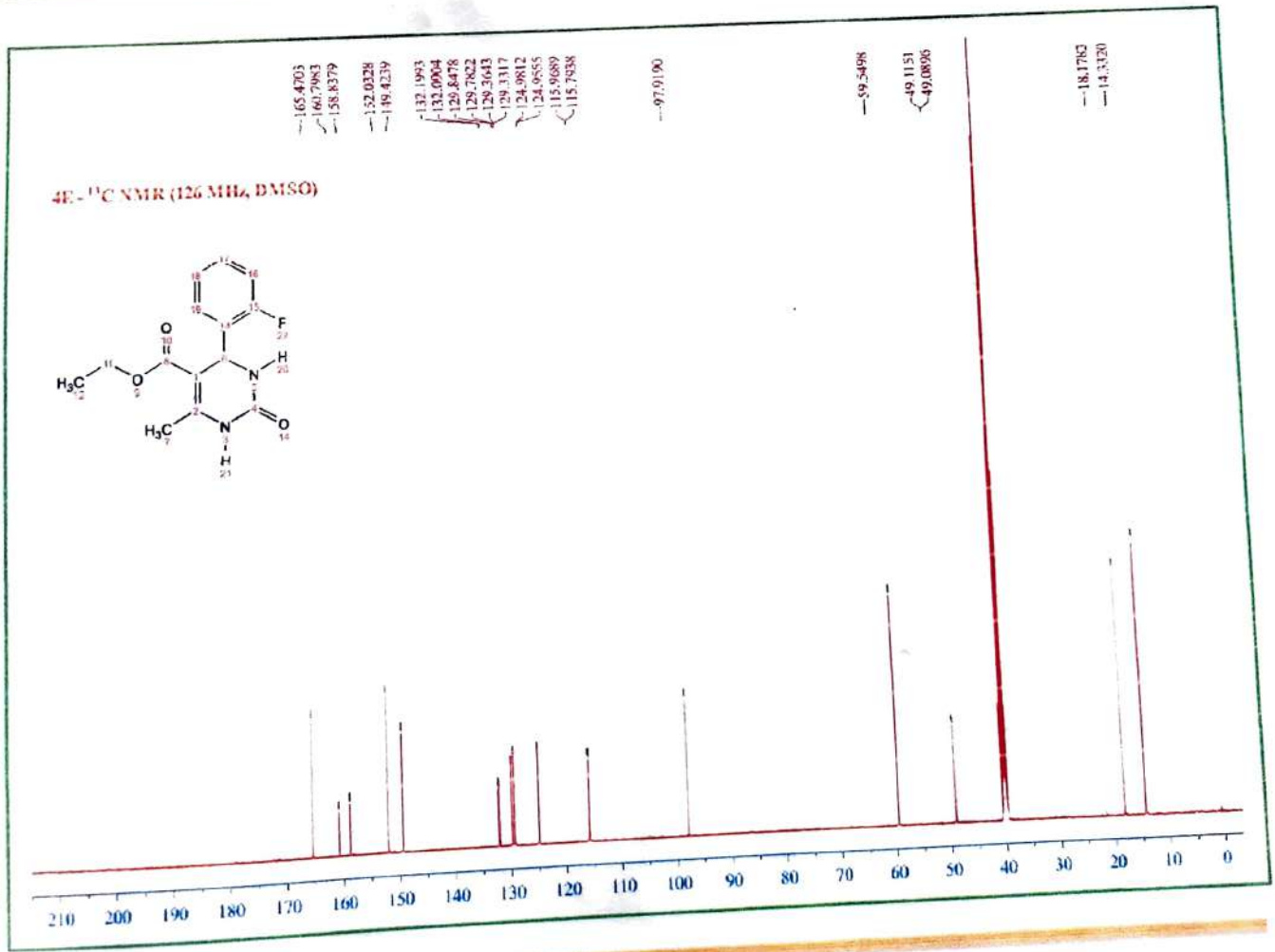
[a] Reaction Condition: Ethyl acetoacetate (1 mmol), Aromatic aldehyde (1.2 mmol), Urea (1.5 mmol), Ethanol (3 ml), Catalyst-Fe_2O_3 NRs (30 mg; 0.19 mmol; 4.83 mol %) under reflux condition.

[b] Isolable yield

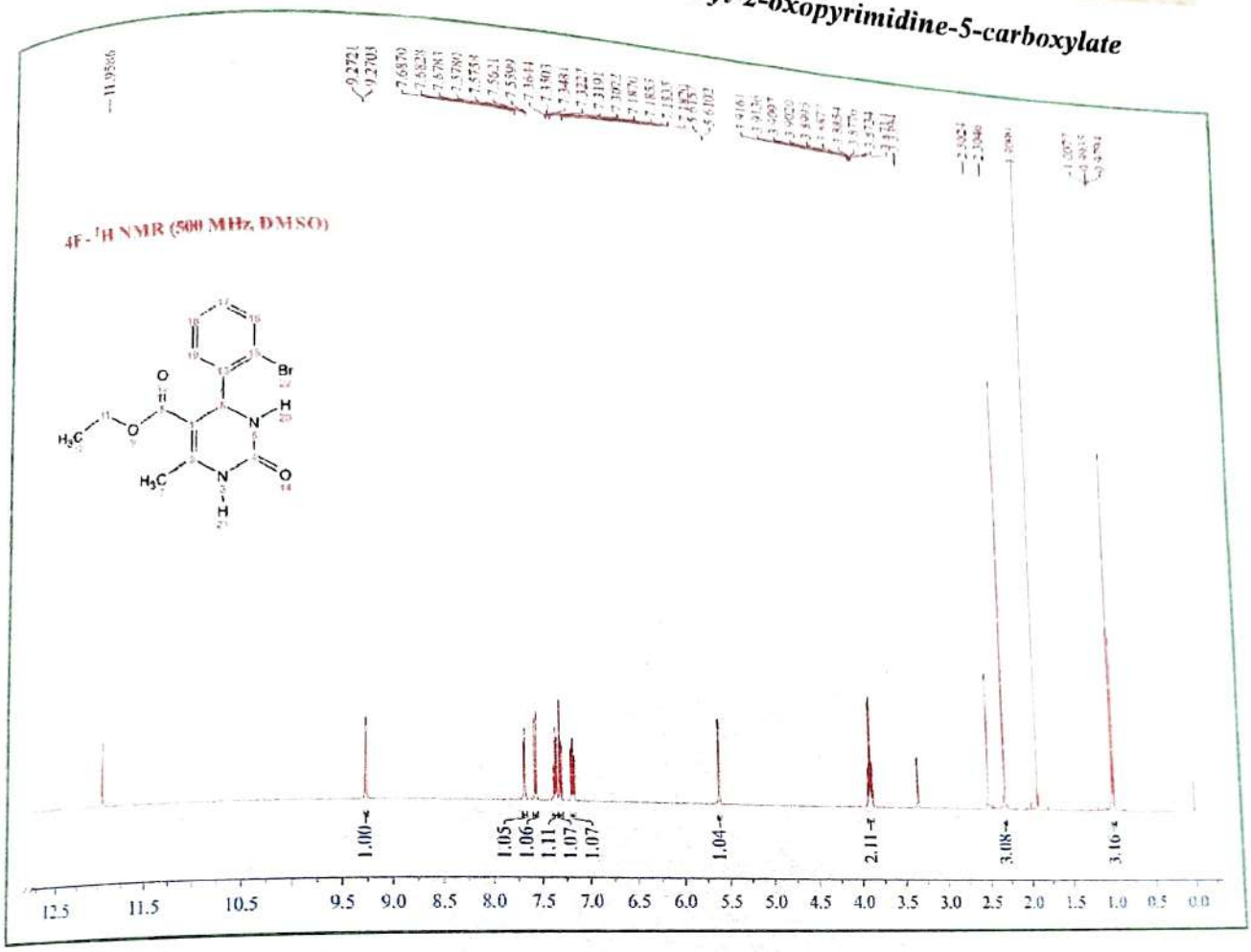
follows:

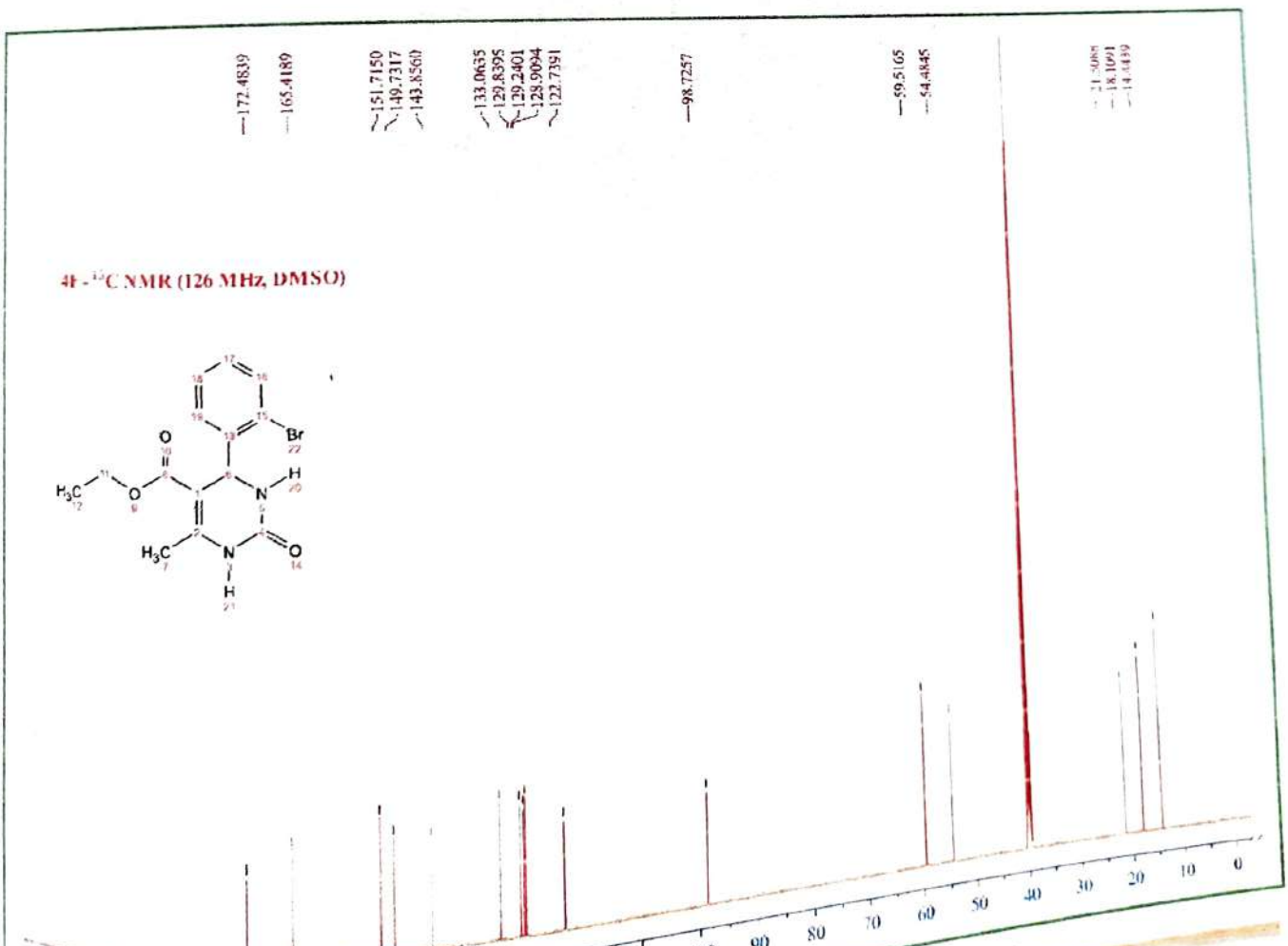
Ethyl 4-(2-fluorophenyl)-1,2,3,4-tetrahydro-6-methyl-2-oxopyrimidine-5-carboxylate

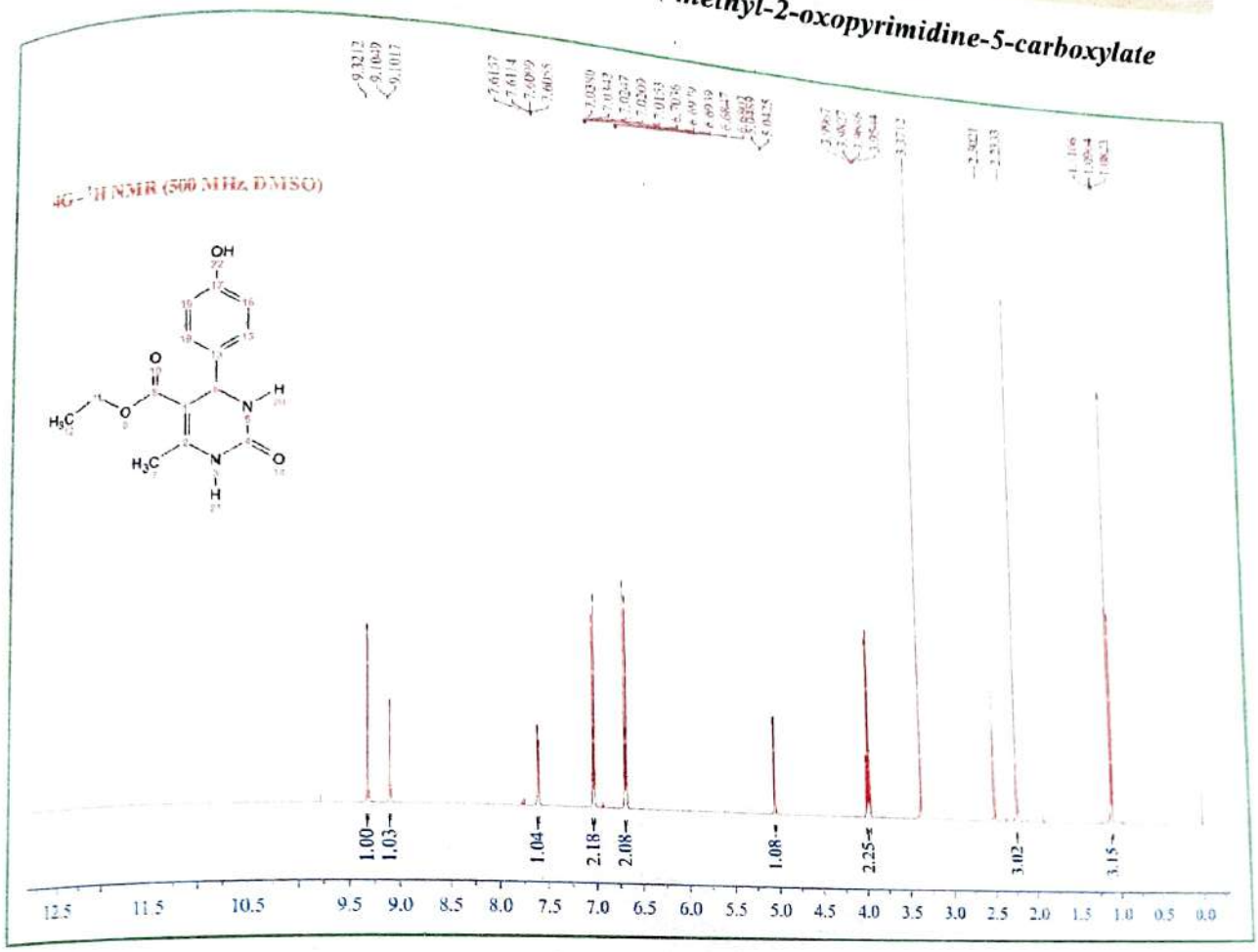


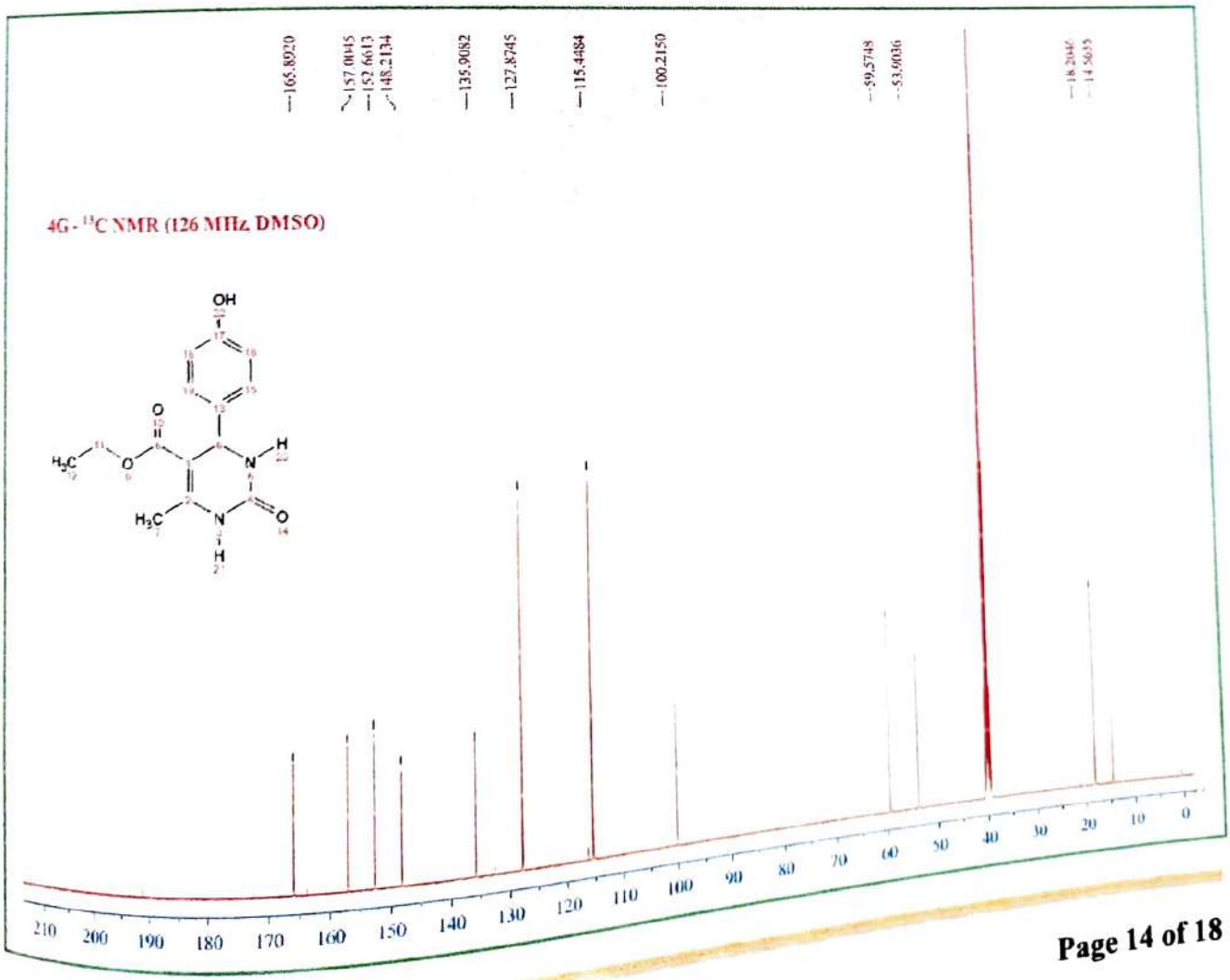


Ethyl 4-(2-bromophenyl)-1,2,3,4-tetrahydro-6-methyl-2-oxopyrimidine-5-carboxylate






4G - ^{1}H NMR (500 MHz, DMSO)
9.3212
9.1049
9.1017
7.6157
7.6114
7.6099
7.6055
3.3712
2.5233
1.1863
1.00
1.03
1.04
2.18
2.08
1.08
2.25
3.02
3.15
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


4G - ^{13}C NMR (126 MHz, DMSO)
165.8920
157.0045
152.6613
148.2134
135.9082
127.8745
115.4484
100.2150
59.5748
53.6036
18.2046
14.5655
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

subsequently undergoes nucleophilic addition on aldehyde to get THPMs. The third mechanistic path involves a Knoevenagel-type reaction (**Figure 10c**) [22]. Here firstly, the condensation of an aldehyde with ethyl 3-hydroxybut-2-enoate (enol generated in-situ form Ethyl acetoacetate) takes place, which on reaction with urea result in the formation of THPMs. In the last decade, De Souza et al. [23], Neto et al. [24], and Puripat et al. [25] did a comparative study on the mechanistic path of the Biginelli reaction. They found that the Iminium intermediate route is favored over the Enamine and Knoevenagel route. According to Puripat et al. [25], based on AFIR and DFT calculation, the order of reactive intermediate formation is Imine > Enamine > Knoevenagel route.

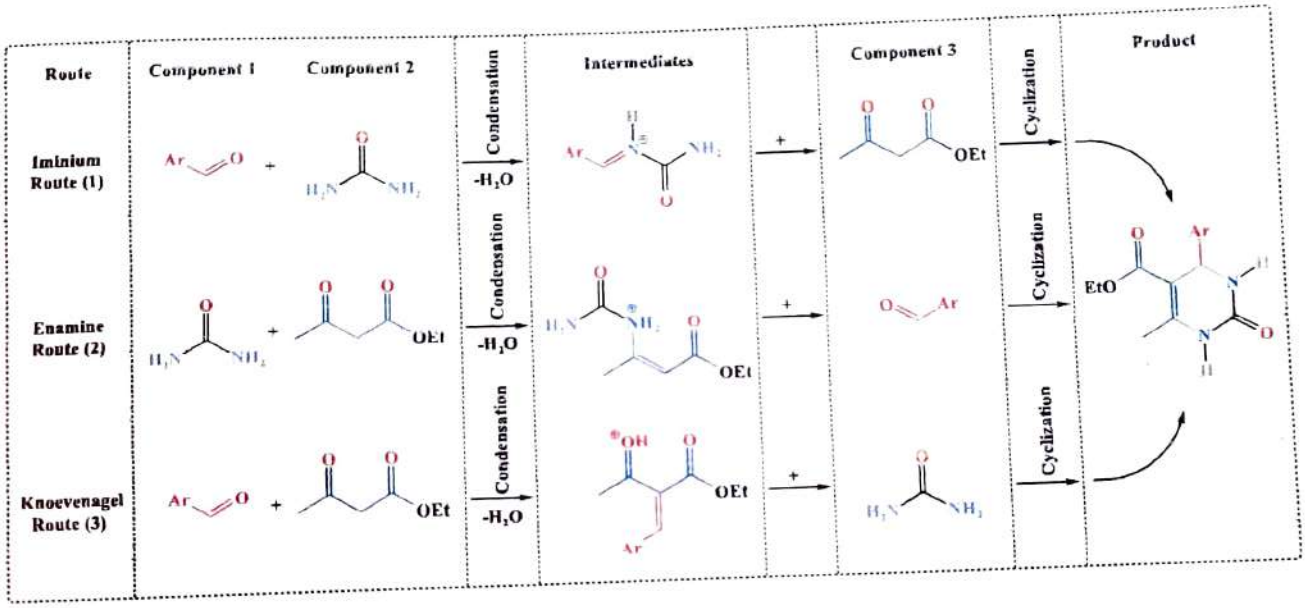


Figure 10. Possible reaction mechanism for the Fe_2O_3 NRs catalyzed THPMs synthesis

4. Conclusion

In this study, for the first time, mixed-phase Fe_2O_3 NRs were biologically synthesized using a mixture of *Eucalyptus citriodora* and *Murraya koenigii* leaf extracts and its application as a heterogeneous magnetic nanocatalyst in the multicomponent Biginelli reaction was reported. The catalyst was synthesized through an utterly green approach and then characterized by XRD, UVDRS, FTIR, FESEM, EDX, and VSM analysis. Then, the practical synthesis of ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-arylpyrimidine-5-carboxylate was prepared with an ethyl acetoacetate, aromatic aldehydes, urea with a catalytic amount of the magnetically recoverable Fe_2O_3 NRs in ethanol as a green solvent. The notable merits of this protocol are easy workup procedure, reusability of catalyst, being clean and safe and having excellent yields of the final

products. This eco-friendly approach for synthesizing Fe_2O_3 NRs can open new horizons for exploring their possible role in organic transformation.

References

1. Kim, J., et al., *A novel 3, 4-dihydropyrimidin-2 (1H)-one: HIV-1 replication inhibitors with improved metabolic stability.* Bioorganic & medicinal chemistry letters, 2012. **22**(7): p. 2522-2526.
2. de Fátima, Â., et al., *A mini-review on Biginelli adducts with notable pharmacological properties.* Journal of advanced research, 2015. **6**(3): p. 363-373.
3. Janković, N., et al., *Discovery of the Biginelli hybrids as novel caspase-9 activators in apoptotic machines: Lipophilicity, molecular docking study, influence on angiogenesis gene and miR-21 expression levels.* Bioorganic chemistry, 2019. **86**: p. 569-582.
4. Canto, R.F., et al., *Design, synthesis and evaluation of seleno-dihydropyrimidinones as potential multi-targeted therapeutics for Alzheimer's disease.* Organic & biomolecular chemistry, 2014. **12**(21): p. 3470-3477.
5. Barbosa, F.A., et al., *Synthesis and evaluation of dihydropyrimidinone-derived selenoesters as multi-targeted directed compounds against Alzheimer's disease.* Bioorganic & Medicinal Chemistry, 2016. **24**(22): p. 5762-5770.
6. Mostafa, A.S. and K.B. Selim, *Synthesis and anticancer activity of new dihydropyrimidinone derivatives.* European journal of medicinal chemistry, 2018. **156**: p. 304-315.
7. Sana, S., et al., *Design and synthesis of substituted dihydropyrimidinone derivatives as cytotoxic and tubulin polymerization inhibitors.* Bioorganic Chemistry, 2019. **93**: p. 103317.
8. Tungittiplakorn, W., C. Cohen, and L.W. Lion, *Engineered polymeric nanoparticles for bioremediation of hydrophobic contaminants.* Environmental Science & Technology, 2005. **39**(5): p. 1354-1358.
9. Pauling, L. and S.B. Hendricks, *The crystal structures of hematite and corundum.* Journal of the American Chemical Society, 1925. **47**(3): p. 781-790.
10. Hmamouchi, S., A. El Yacoubi, and B.C. El Idrissi, *Using egg ovalbumin to synthesize pure α-Fe2O3 and cobalt doped α-Fe2O3: structural, morphological, optical and photocatalytic properties.* Heliyon, 2022. **8**(2): p. e08953.
11. Pecharromán, C., T. Gonzalez-Carreno, and J.E. Iglesias, *The infrared dielectric properties of maghemite, γ-Fe2O3, from reflectance measurement on pressed powders.* Physics and Chemistry of Minerals, 1995. **22**(1): p. 21-29.
12. Fouda, A., et al., *An eco-friendly approach to textile and tannery wastewater treatment using maghemite nanoparticles (γ-Fe2O3-NPs) fabricated by Penicillium expansum strain (Kw).* Journal of Environmental Chemical Engineering, 2021. **9**(1): p. 104693.
13. Ali, H.R., H.N. Nassar, and N.S. El-Gendy, *Green synthesis of α-Fe2O3 using Citrus reticulum peels extract and water decontamination from different organic pollutants.* Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, 2017. **39**(13): p. 1425-1434.

14. Miri, A., M. Khatami, and M. Sarani, *Biosynthesis, magnetic and cytotoxic studies of hematite nanoparticles*. Journal of Inorganic and Organometallic Polymers and Materials, 2020. **30**(3): p. 767-774.
15. Zhou, Z.-L., et al., *Flavonoid glycosides and potential antivirus activity of isolated compounds from the leaves of Eucalyptus citriodora*. Journal of the Korean Society for Applied Biological Chemistry, 2014. **57**: p. 813-817.
16. Ibrahim, J., et al., *Comparative pharmacognostic and chemical analyses of Eucalyptus camaldulensis Dehnh and Eucalyptus citriodora (Hook)*. Journal of Chemical Society of Nigeria, 2018. **43**(3).
17. Nejres, A.M., et al., *Potential effect of ammonium chloride on the optical physical properties of polyvinyl alcohol*. Systematic Reviews in Pharmacy, 2020. **11**(6): p. 726-732.
18. He, Y., et al., *Size and structure effect on optical transitions of iron oxide nanocrystals*. Physical review B, 2005. **71**(12): p. 125411.
19. Apriandanu, D.O.B., et al., *Effect of two-step annealing on photoelectrochemical properties of hydrothermally prepared Ti-doped Fe2O3 films*. Catalysis Today, 2022.
20. Kappe, C.O., *A reexamination of the mechanism of the Biginelli dihydropyrimidine synthesis. Support for an N-Acyliminium ion intermediate1*. The Journal of organic chemistry, 1997. **62**(21): p. 7201-7204.
21. Folkers, K. and T.B. Johnson, *Researches on pyrimidines. CXXXVI. the mechanism of formation of tetrahydropyrimidines by the biginelli reaction1*. Journal of the American Chemical Society, 1933. **55**(9): p. 3784-3791.
22. Sweet, F. and J.D. Fissekis, *Synthesis of 3, 4-dihydro-2 (1H)-pyrimidinones and the mechanism of the Biginelli reaction*. Journal of the American Chemical Society, 1973. **95**(26): p. 8741-8749.
23. De Souza, R.O., et al., *The three-component Biginelli reaction: a combined experimental and theoretical mechanistic investigation*. Chemistry–A European Journal, 2009. **15**(38): p. 9799-9804.
24. dos Santos, R.L.T.A., *MR de Oliveira HCB Gomes AF Gozzo FC de Oliveira AL Neto BAD J*. Org. Chem, 2012. **77**: p. 10184-10193.
25. Puripat, M., et al., *The Biginelli reaction is a urea-catalyzed organocatalytic multicomponent reaction*. The Journal of Organic Chemistry, 2015. **80**(14): p. 6959-6967.

Maratha Vidya Prasark Samaj, Nashik

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

A

Internship Report

On

Research And Development

Submitted By-

Mr. Hire Sagar Kailas

M.Sc. (Organic Chemistry)

Dept. Of Chemistry

Year : 2022-2023

Maratha Vidya Prasark Samaj,Nashik

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

Examination Seat No:

Certificate

This is to Certified that the internship report

"Research And Development"

Has been carried out in our premises by-

Hire Sagar Kailas

Under my guidance in partial fulfilment of the requirement for

Second year of M.Sc. during the academic year 2022-23

In

SAVITRIBAI PHULE PUNE UNIVERSITY, PUNE-411007

Prof. M.Gaware

Head Of the Department

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

External Examinar

Dr .P.V Rasal

Principal

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

ARSS BIOFUEL PRIVATE LIMITED
CIN- U24119OR2003PTC007115
FACTORY ADD: GAT NO: 196, WADIVARHE, DARNA ASWALI ROAD. TALUKA - IGATPURI, NASHIK-1
EMAIL ID – arss112000@yahoo.com PH +91 9422271148 / 8830665543.

APPRENTICESHIP LETTER

To whom it may concern

This is to certify that **Mr. Sagar Kailas Hire worked with ARSS Biofuel Pvt. Ltd.** as a Chemist from Novomber2022.

He has a wide knowledge in chemical process intensification and optimization and has worked on several projects regarding the same for our company.

His efforts and skills are commendable and have contributed towards the successful operation of the company in the research and operational fronts. He has performed his duties in diligent manner and is a valuable resourceof our company.

His job role included:

- Assessing existing processes and taking measurements
- Perform process Simulation and troubleshooting issues
- Assisting optimizing and upgrading systems

wishing him good luck for future endeavors.

For ARSS BIOFUEL PVT LTD

Authorized Signatory

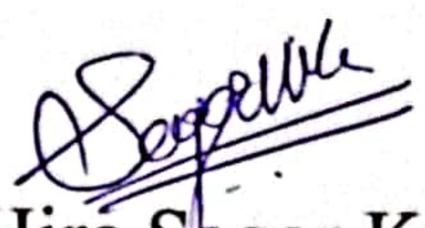
ARSS BIOFUEL PRIVATE LIMITED CIN U24119 OR2003 PTC007115 NASHIK

Place: Nashik

Date:

ACKNOWLEDGEMENT

I wish like to thank the owner of **ARSS Pingle Mohan** for permitting me to use all the facilities available in the industry for my internship work. I would also like to thank the all the worker of **ARSS** for their cooperation . I am grateful to his encouragement, guidance and supervision of my internship work during the year. I must acknowledge the financial support given to my training by my parents. I would also like to thank principle of G.M.D college **Dr. P.V Rasal** and head of the department **Dr. Manoj Gaware** and all the teaching and non-teaching staff for their help.



Hire Sagar Kailas

M.Sc. II (Organic Chemistry)

INDEX

Abstract

I have undergone my industrial training in ARSS Biofuel in Igatpuri in November 2022. ARSS bio fuel is company that specialized in manufacturing anhydrous ethanol and sanitizer. I was placed in sanitizer department as a lab technician under a senior supervisor.

During my one month internship I have learnt the basics knowledge of doing maintenance of machine. Furthermore, I have learnt about the sanitizers manufacturing.

They always focus more on safety of worker as they are dealing with chemical. All ine this industrial training has given me the valuable insight of being a professional chemist.

Introduction

Internship program has become the bridge for those who want to enter to corporate level from the college life, through internship I got to know the real working environment that was very much different from my course study.

The primary object objective of the internship is to generate a through understanding of the work place relationship, performing of the activities and engaging oneself in the working environment. It was more to get practical implication of all the studies, theories that I had acquired so far. This would help me to pave a way towards growth in my academic as well as personal development.

- To learn and apply theoretical knowledge practically.
- To develop interpersonal managerial and communication skill.
- To come up with possible strategies to gain competitive advantage.
- To fulfill the partial requirement for the Master of Chemistry of SPPU.

A R S S
BIOFUEL PVT. LTD.

About Company

Name : ARSS BIOFUEL PRIVATE LIMITED

CIN – U24119OR2003PTC007115

Add : GATE.NO : 196 , WADIVARHE,DARNA ASWALI ROAD , TAL,

IGTAPURI NASHIK-1

Email ID : arss112000@yahoo.com

Ph : +91 9422271148 / 8830665543

It is a non government company, incorporated on 17 April 2003 . it's a private unlisted company and is classified as company limited by shares.

The company authorised capital stands at Rs 100 lakhs and 62.90% paid –up

capital which is Rs 69.3 lakhs. ARSS Biofuel Private limited last annual general

meet happened on 2017.

Arss Biofuel is majorly in manufacturing (Metal & Chemicals) .

Bio-Fuel

- Unlike other renewable energy sources, biomass can be converted directly into liquid fuel called "Bio fuel".
- It helps to meet transportation fuel need.
- Bio fuel is commonly advocated as a cost effective and environment friendly.
- Bio fuel is the fuel which produce from organic product and wastage.
- The common commercially used bio fuel are Bio Ethanol, Bio Diesel and Bio Methane.
- Bio ethanol is made from sugar, alga, wheat and sugar beet .
- Bio diesel is made from vegetable oils, algal lipids and animal fats.

Company Product

1.Ethanol

- Ethanol is an organic compound.
- It is an alcohol with the chemical formula C_2H_5OH.
- Ethanol is volatile, flammable, colourless liquid with a characteristic wine like odour and pungent test.

Molar mass: 46.07 gm/mo

Boiling Point: 78.37°C

Density: 789 kg/m³

2. Denatured Anhydrous Ethanol

- Production of Ethanol is large scale has been made by extractive distillation using conventional solvent like ethylene-glycol.
- Extractive distillation process is done to obtain pure ethanol using ethylene-glycol as solvent. Residue curve maps arcused to analyse the proposed distillation process in interpreting mixture behaviour and feasibility of distillation column.
- Ethanol forms a minimum – boiling azeotrope with water at about 90mol% at 1 atm.
- This azeotrope must be broken to achieve anhydrous ethanol.
- The usual solvent applied in the industries to promote ethanol and water separation is the ethylene-glycol.

Posted on May 10, 2018

We are manufacturer and exporter of Denatured Ethanol.

Application Of Anhydrous Ethanol

- Chemical Reagent
- Organic Solvent
- Raw Material for drugs
- Raw material for cosmetics
- It also used to remove the paint, ink from fabrics.

3. Ethanolic HCL

Hydrochloric acid in ethanol for microscopy is used for human medical cell diagnosis and histological investigation of sample material of human origin. Acid fast bacteria are difficult to stain because of the high proportion of lipid and wax in their cell wall.

Structure of ethanolic HCL: C_2H_7ClO

Price of Ethanolic HCL : pure grade Ethanolic HCL for ARSS biofuel liquid at is 550/kg in Igatpuri Nashik-1.

4.Sanitizers:

- Hand Sanitizer (also known as hand antiseptic) hand disinfectant hand rub or hand.
- Alcohol based hand sanitizer that is at least 60% alcohol in water (specially ethanol) or isopropyl alcohol or isopropanol is recommended by the united state centre for Disease Control and Prevention (CDC).
- The CDS recommends the following step's when using an alcohol based hand sanitizers.

1. Apply Product to palm on hand.
2. Rub hand together.
3. Rub product over all surface of hand and finger's until hand are dry.
4. Do not go near flame or gas banned or any burning object during application of hand sanitizer.

Preparation of institizer (sanitizer)

Steps:

Isopropyl alcohol (95%)

⇓

Hydrogen peroxide (3%)

⇓

Glycerin ($C_3H_8O_3$)

⇓

Distilled water

⇓

Shake or stir well

⇓

Clear or contain in plastic

Many hand sanitizer must be stored at below 25°C.

Do not store hand sanitizer in car or anywhere it will get too hot hand sanitizer is flammable so don't store it next to heat source. Spark or open flames.

- Instead stored in following plastic bottles:

  Plastic used for hand sanitizers.

  PET- polyethylene terephthalate

  HDPE- high density polyethylene

  LDPE- low density polyethylene

  PP - polypropylene

Job Description:

Job title and grade: Internship-Laboratory chemist

Job purpose: Laboratory

Key Responsibility:

- Conduct testing under supervision of either chemist/senior or under laboratory technician.
- Evaluation /test quality of specially chemicals for sanitizer.
- Handling and cleaning laboratory and apparatus (laboratory hygiene).
- Packaging sample requested by customer.

Conclusion

The experience and knowledge during the internship at ARSS Bio fuel was great. This industry has a superb work culture, great mind and very high quality of work. At the laboratory, they provide many instrument and their worker will conduct the entire instrument by their own. During the internship, I was introduced and learned to handle equipment such a high machine used to manufacture ethanol and other product. The knowledge and skill get from the internship will be use to apply a worker and do the final year project. Working with their people was a rare chance and it was another opportunity to make friends and share ideas. Overall experience is very helpful.

Reference

- https:/m.indianmart.com
- http:/arss-biofuel-pvt-ltd.business
- http:/www.thecompanycheck.com