

„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

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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. Swati Sahebrao 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**.

*Gaware*

**HOD Chemistry**

**HEAD**

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

*Arni*  
*12-01-2023*

**Examiner**

*Principals*

**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.  
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**Certificate**

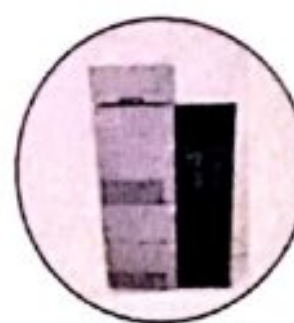
This is to Certify that ..... ***Swati Sahebrao Thorat*** .....  
has Successfully Completed Pharmaceutical Training Course in  
Analytical Techniques includes Practically Handling the  
Instruments Like HPLC, UV - Spectrophotometer,  
Dissolution Test Apparatus & Pharmaceutical  
Instruments in the 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. **Swati Sahebrao 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:- Swati Sahebrao Thorat



# INDEX

Sr.No	Description	Page No.
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# TENELIGLIPTIN

## Introduction-

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

**Chemical Formula-** C<sub>22</sub>H<sub>30</sub>N<sub>6</sub>O<sub>5</sub>

**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 excipients.
- Once a tablet is formulated then directly it doesn't come to market. First of all some of the random tablets are collected and forwarded for testing.

## Testing have 2 types-

1. Physical
2. Chemical

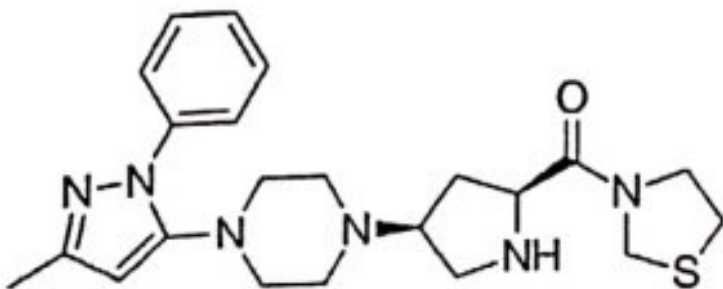
## Physical Testing-

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

## Chemical Tests-

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

## Structure of Teneligliptin-







# ARN ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 1 of 4

### SPECIFICATION AND TESTS OF TENELIGLIPTIN TABLETS 20 MG

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





# ARNI ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 2 of 5

### TEST METHOD

- 1) **Description:** White coloured, round shaped, film coated tablets, plain on both sides.
- 2) **Identification:**  
The retention time of the principal peak in the chromatogram of sample preparation should correspond to that of the standard preparation as obtained in the "Assay".
- 3) **Average weight:**  
Weigh together 10 tablets selected at random and calculate the average weight.

**Calculation:**

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

Where, W= Weight of 10 tablets in mg

Limit: 283 mg  $\pm$  7.5%

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

Limit: 283 mg  $\pm$  7.5% (Between 261.8 mg and 304.2 mg)





# ARNI ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 3 of 5

### 5) Dissolution (By HPLC):

#### Dissolution Parameters :

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

potency = 99.85%

### 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 ml → 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 270nm.

L.C. = 20 mg

#### Calculations:

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

*↓ T.H.H → Teneligliptin Hydrobromide Hydrate*

Where,

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

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





# ARNI ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 4 of 5

### 13) Assay (By HPLC):

#### • Reagents Required:

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

#### 13.1 Chromatographic Conditions:

Column	:	C18, (150 mm X 4.6 mm), 5 $\mu$ m
Pump mode	:	Isocratic
Mobile Phase	:	Buffer : Acetonitrile (60:40)
Flow rate	:	1.0 mL/min
Injection volume	:	20 $\mu$ l
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  $\mu$  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  $\mu$  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.





# ARNI ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 5 of 5

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

### 13.5 Procedure:

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

#### • Calculations:

$$\text{mg/tab of Teneligliptin} = \frac{A_T}{A_S} \times \frac{W_S}{100} \times \frac{100}{W_T} \times \frac{P}{100} \times A_W \times \frac{426.57}{628.86} \times \frac{100}{LC}$$

Where,

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

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

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

$P$  = Purity of Teneligliptin hydrobromide hydrate working standard, on as is basis.

$LC$  = Label claim in mg.

$A_W$  = Average weight in mg.

$W_T$  = Weight of sample taken for sample preparation, in mg.

426.57 = Molecular weight of Teneligliptin.

628.86 = Molecular weight of Teneligliptin Hydrobromide Hydrate

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





# ARN ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 2 of 4

### TEST METHOD

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

2) **Identification:**

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

3) **Average weight:**

TABLETS-1	:	292	TABLETS-6	:	287
TABLETS-2	:	291	TABLETS-7	:	286
TABLETS-3	:	287	TABLETS-8	:	287
TABLETS-4	:	283	TABLETS-9	:	281
TABLETS-5	:	300	TABLETS-10	:	292

AVERAGE WEIGHT:-

288.6%

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	:	291	TABLETS-6	:	286
TABLETS-2	:	287	TABLETS-7	:	300
TABLETS-3	:	292	TABLETS-8	:	292
TABLETS-4	:	287	TABLETS-9	:	281
TABLETS-5	:	283	TABLETS-10	:	287

MINIMUM WEIGHT :- 281 mg.

MAXIMUM WEIGHT :- 300 mg.

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





# ARN ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 3 of 4

### 5) Dissolution (By HPLC):

Dissolution Parameters :					
Medium	:	water	Rotatory Speed	:	75 rpm.
Volume	:	900 ml	Temperature	:	37°C
Apparatus	:	Paddle-usp.2.	Time	:	45 min

Standard Weight :-

Potency:-

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

$$\text{Tablet 1} = \frac{0.6364}{0.5531} \times \frac{32.43}{100} \times \frac{5 \times 900}{50 \times 20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 113.72$$

$$\text{Tablet 2} = \frac{0.6260}{0.5531} \times \frac{32.43}{100} \times \frac{5 \times 900}{50 \times 20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 111.87$$

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

$$\text{Tablet 4} = \frac{0.6256}{0.5531} \times \frac{32.43}{100} \times \frac{5 \times 900}{50 \times 20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 111.79$$

$$\text{Tablet 5} = \frac{0.6415}{0.5531} \times \frac{32.43}{100} \times \frac{5 \times 900}{50 \times 20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 114.64$$

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

$$\text{Average:- } \frac{113.72 + 111.87 + 96.52 + 111.79 + 114.64 + 97.71}{6} = 107.70\%$$

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	:	C <sub>18</sub> , (150 nm 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	:	UV. 210 nm.

#### Preparation of solutions:

##### • Standard preparation:

weigh accurately 20 mg of teneligliptin (29.48 mg) THH) working standard to 100 ml volumetric flask, add 60 ml of water & sonicate to dissolve & make up the volume with water.

##### • Sample preparation:

wt. 10 tablet & determine av. wt. crush it. wt. the & transfer 20 mg of teneligliptin (282 mg) of teneligliptin to 100 ml volumetric flask. add water & sonicate & make up the volume with water. & Fil. 0.45 μm fil. discarding first few ml of filtrate.

Standard Weight :- 29.48 mg

Sample Weight :- 282.0 mg

Average Weight :- 282.33 %

Potency :- 99.85 %

##### • Calculations:

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

$$1) \frac{13736385}{14490846.5} \times \frac{29.48}{100} \times \frac{100}{282} \times \frac{99.85}{100} \times 282.33 \times \frac{426.57}{628.86} \times \frac{100}{20}$$

$$= 94.74 \%$$

$$2) \frac{12688085}{14490846.5} \times \frac{29.48}{100} \times \frac{100}{282} \times \frac{99.85}{100} \times 282.33 \times \frac{426.57}{628.86} \times \frac{100}{20}$$

$$= 87.57 \%$$

Average :-  $\frac{94.74 + 87.57}{2} = 91.28 \%$

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 3
Instrument ID :-	ARNI / INB - 001	
Name Of Student :-	Thorat Swati Sahebrao	

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

Thorat  
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Prabhu  
CHECKED BY





# ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	High Performance Liquid chromatography	Page No
Instrument Make :-		
Instrument Model No. :-	Shimadzu	2 of 3
Instrument ID :-	L-2010 CHT	
Name Of Student :-	ARNI / INS - 001 Thorat Swati Sahabrao	

## • CHROMATOGRAPHIC PARAMETERS-2

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

## • CHROMATOGRAPHIC PARAMETERS-3

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisition Time	:	22
Pump	:	1.20
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. :-	LC 2010 CHT	3 of 3
Instrument ID :-	ARN/INS - 001	
Name Of Student :-	Thorat Swati Sahebrao	

## • CHROMATOGRAPHIC PARAMETERS-4

INSTRUMENT PARAMETERS		Set Parameters
Data Acquisition Time	:	30 min
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 Acquisition 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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Thorat  
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# ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	High performance Liquid Chromatography	Page No 1 of 1
Instrument Make :-	shimadzu	
Instrument Model No. :-	LC 2010 CHT	
Instrument ID :-	ARNI/IMS -001	

## DATA SHEET

NAME OF TEST :- SYSTEM SUITABILITY

SYSTEM SUITABILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINE

• **Chromatographic Conditions:**

Column	A stainless steel column Dimensions :- 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 Acquisition Time	7 Minutes
Pump (Flow Rate)	<del>1.00</del> 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 <sup>70</sup>~~80~~ volumes of Water and <sup>30</sup>~~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 chromatography.

Page No

Instrument Make :-

Shimadzu

Instrument Model No. :-

LC 2010 CHT

1 of 1

Instrument ID :-

ARNI / INS - 001.

NAME OF STUDENT :-

## DATA SHEET

NAME OF TEST :- SYSTEM SUITABILITY

SYSTEM SUITABILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINE

• Chromatographic Conditions:

Column	
Data Acquisition Time	15 min
Pump (Flow Rate)	1.50 ml/min
Port	A
Detector (Wavelength)	273 nm.
Column Oven Temperature	30°C
Degasser	off.
Autosampler Temperature	off

• MOBILE PHASE PREPARATION:- Prepare mix. of 140 ml of water & 60 ml of methanol.

• STANDARD PREPARATION:- wt. 20 mg of caffeine standard to 100 ml volumetric flask, Add 60 ml HPLC grade water & shake to dissolve completely. make up volume up to mark. further dilute 5 ml of the above sol<sup>n</sup> to 50 ml volumetric flask. dilute up to mark

• SEQUENCE OF INJECTION:-

Name of Solution	No. Of Injection
Blank	
Standard	

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# ARNI ANALYTICALS

TITLE

MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE

Instrument Name :-

Analytical Balance

Page No

Instrument Make :-

Wensar

Instrument ID :-

~~DS-8000~~ 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: $\pm 0.1\%$ )
1	200.0000	199.98	199.8000 to 200.2000
2	100.0000	98.751	99.9000 to 100.1000
3	50.0000	50.692	49.9500 to 50.0500
4	20.0000	19.048	19.9800 to 20.0200
5	10.0000	9.993	9.9900 to 10.0100
6	5.0000	5.078	4.9950 to 5.0050
7	2.0000	1.231	1.9980 to 2.0020
8	1.0000	0.911	0.9990 to 1.0010
9	0.5000 500mg	0.138	0.4995 to 0.5005
10	0.2000 200	0.092	0.1998 to 0.2002
11	0.1000 100	0.058	0.0999 to 0.1001
12	0.0500 50	0.038	0.0499 to 0.0501
13	0.0200 20	0.017	0.0199 to 0.0200
14	0.0100 10	0.014	0.0099 to 0.0100
15	0.0050 5	0.006	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 :-

Mensar

Instrument ID :-

ARNZ/INS-004

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## 2. Test for Linearity:

Sr. No.	Selected Weights in g	Observed Weight in g
1	50 gm	50.697
2	100 gm	98.756
3	200 gm	199.999

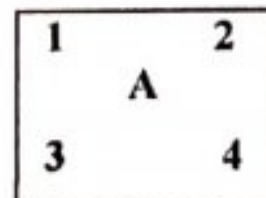
Conclusion: The observed weights are **Consistent/not Consistent**.

*Mensar*

ANALYSED BY

CHECKED BY

## 3. Test for Eccentricity:



Sr. No.	Weight Observed in g	Difference in g	Limit	
1.	At Centre- (A)	199.998	± 0.1 % 0.199%	
2.	At Corner 1 (B)	200.007		B-A = 0.009
3.	At Corner 2 (C)	199.995		C-A = -0.003
4.	At Corner 3 (D)	199.991		D-A = -0.007
5.	At Corner 4 (E)	200.00		E-A = -0.002

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

*Shank*  
ANALYSED BY

*Mensar*  
CHECKED BY





# ARNI ANALYTICALS

TITLE

MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE

Instrument Name :-

Analytical Balance

Page No

Instrument Make :-

WENSAR

Instrument ID :-

ARNI/INS-004

3 of 3

#### 4. Test for Repeatability :

Selected Weight in g: 10 g.

Sr. No.	Observed Weight in g	Sr. No.	Observed Weight in g	Limit
1	10.003	6	9.996	± 0.1 %
2	9.997	7	9.998	
3	9.998	8	9.995	
4	9.996	9	9.995	
5	9.999	10	9.997	

+ 10.01 g

- 9.99 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  
*STB*

CHECKED BY  
*STB*





# ARNI ANALYTICALS

<b>TITLE</b>	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
<b>Instrument Name :-</b>	ANALYTICAL BALANCE	<b>Page No.</b>
<b>Instrument Make :-</b>	WENSAR	
<b>Instrument Model No. :-</b>	DS-8000	1 of 3
<b>Instrument ID :-</b>	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: $\pm 0.1\%$ )
1	200.0000	200.035	199.8000 to 200.2000
2	100.0000	98.778	99.9000 to 100.1000
3	50.0000	50.708	49.9500 to 50.0500
4	20.0000	19.056	19.9800 to 20.0200
5	10.0000	10.00	9.9900 to 10.0100
6	5.0000	5.082	4.9950 to 5.0050
7	2.0000	1.233	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  
S. Sreed

CHECKED BY:-  
M. Sreed





# ARNI ANALYTICALS

TITLE

MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE

Instrument Name :-

ANALYTICAL BALANCE

Instrument Make :-

WENSAR

Instrument Model No. :-

DS-8000

Instrument ID :-

ARNI/INS - 004

Page No.

2 of 3

## 2. Test for Linearity:

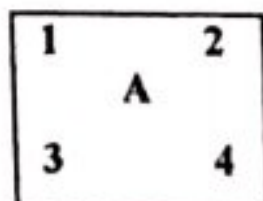
Sr. No.	Selected Weights in g	Observed Weight in g
1	200	200.035
2	100	98.785
3	50	50.706

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) 19.056		± 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	

diff = 0.09528

0.944

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 :-

Instrument Make :-

Instrument Model No. :-

Instrument ID :-

Page No.

3 of 3

#### 4. Test for Repeatability :

Selected Weight in g: 10 gm,

diff = 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	9	10.001	
5	10.001	10	10.003	

**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

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# ARNI ANALYTICALS

TITLE

DAILY CALIBRATION RECORD OF pH-METER

Instrument Name :-

pH meter

Page No

Instrument Make :-

LABMAN

Instrument Model No. :-

LMPH-10

1 of 1

Instrument ID :-

ARNI / INS -004

## DAILY CALIBRATION RECORD

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

- Preparation Of Solutions:

- pH-4.01 :-

- Transfer the capsule content in a 100ml volumetric flask using a funnel.
- Dissolve the contents in 10 ml of distilled water and then make it up to 100 ml with distilled water.
- This solution will have a pH of  $4.0 \pm 0.05$  at  $25^{\circ}\text{C}$ .

- 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  $7.0 \pm 0.05$  at  $25^{\circ}\text{C}$ .

- 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  $9.20 \pm 0.05$  at  $25^{\circ}\text{C}$ .

- Observation Table:

Sr. No.	Date	pH	
		4.00 ( $\pm 0.05$ )	7.00 ( $\pm 0.05$ )
		4.01	6.95

SLOP = 97%

*S. Shrestha*  
PERFORMED BY

*M. B. K. S.*  
CHECKED BY





# ARNI ANALYTICALS

TITLE	DAILY CALIBRATION RECORD OF pH-METER	
Instrument Name :-	pH meter	Page No 1 of 1
Instrument Make :-	LABMAN	
Instrument Model No. :-	LMPH-10	
Instrument ID :-	ARNI/INS-004	

## DAILY CALIBRATION RECORD

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

- **pH-4.01 :-**
  - Transfer the capsule content in a 100ml volumetric flask using a funnel.
  - Dissolve the contents in 10 ml of distilled water and then make it up to 100 ml with distilled water.
  - This solution will have a pH of  $4.0 \pm 0.05$  at  $25^{\circ}\text{C}$ .
- **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  $7.0 \pm 0.05$  at  $25^{\circ}\text{C}$ .
- **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  $9.20 \pm 0.05$  at  $25^{\circ}\text{C}$ .

### Observation Table:

Sr. No.	Date	pH	
		4.00 ( $\pm 0.05$ )	7.00 ( $\pm 0.05$ )
1	14-12-2022	3.88	6.81

Slope = 98 %

*CS Theend*  
PERFORMED BY

*Trace*  
CHECKED BY



# ARNI ANALYTICALS

TITLE

DISSOLUTION TEST APPARATUS WORKSHEET

Instrument Name :-

DISSOLUTION TEST APPARATUS

Page No.

Instrument ID :-

ARNI / IN9 - 003

Instrument Model No. :-

DS 8000

Name Of Students

Thorat swati sahebrao

1 of 1

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/TNS 003	
Instrument Model No. :-	DS 8000	1 of 1
Name Of Students	Tharret Swati Sahebrao	

## NAME OF TEST :-

TRIAL FOR DISSOLUTION TEST.

## DISSOLUTION CONDITIONS:-

Dissolution Media	water
Media Volume	900 ml.
Apparatus	USP TYPE II PADDLE
RPM	100
Temperature	$37.0 \pm 0.5^{\circ}\text{C}$
Time	45 min.

## PREPARATIONS:-

Pour 900 ml of dissolution medium in each vessel. dissolution medium equilibrate at  $37^{\circ}\text{C}$ , Adjust the speed of stirring element at 100 rpm. & place one capsule in each six paddle & adjust the distance 25 mm. bet<sup>n</sup> the bottom of Paddle & inside the bottom of vessel. start the Apparatus. end of specific time interval, withdraw 10 ml aliquot

ANALYSED BY  
Tharret

CHECKED BY:-  
Mhanna





# ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV - spectrophotometer.	Page No.
Instrument ID :-	Labman	
Instrument Model No. :-	1. MSP - UV - 100B	1 of 1
Name Of Students	A ARNI / INS - 002	

## 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 5ml of the above solution to 50ml with water.

10 ppm

### UV-SPECTROPHOTOMETER WAVELENGTH :- 273nm

$$\frac{10}{100} \times \frac{5}{50} = 0.01 \times 1000 \text{ } \underline{\underline{274 \text{ nm}}}$$
$$= \underline{\underline{10 \text{ ppm}}}$$

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	Thorat Swati Satebrao	

Date:-

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

NAME OF TEST :- Wavelength scan.

### PREPARATIONS:-

#### STANDARD PREPARATION :-

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

#### UV-SPECTROPHOTOMETER WAVELENGTH :-

maximum wavelength - 274 nm

minimum wavelength - 206 nm.

### OBSERVATIONS:-

MAXIMUM ABSORPTION WAVELENGTH - 274 nm.

ANALYSED BY  
S. Sheel

CHECKED BY:-  
M. B. B.