

„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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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. 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**.

*Gaware*  
**HOD Chemistry**

**HEAD**

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

*R. D. ...  
12-05-2023*  
**Examiner**

*...*  
**Principal**

**PRINCIPAL**

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# ARNI ANALYTICAL

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## 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,  
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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:-



Name:- Ashiwini Raosaheb Thorat

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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

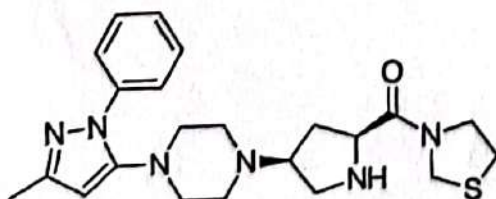
### Physical Testing-

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-







# ARNI ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 2 of 5

### TEST METHOD

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

2) **Identification:**

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

3) **Average weight:**

Weigh together 10 tablets selected at random and calculate the average weight.

**Calculation:**

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

Where, W= Weight of 10 tablets in mg

**Limit: 283 mg ± 7.5%**

4) **Uniformity of Weight:**

Select randomly 10 tablets and weigh individual tablet. Calculate average, the minimum and maximum value.

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





# ARNI ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 3 of 5

### 5) Dissolution (By HPLC):

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

### 11.1 Preparation of Solutions :

#### • Standard preparation :

Weigh and transfer accurately about 22 mg of Teneligliptin (Equivalent to 32.43 mg Teneligliptin Hydrobromide Hydrate) working standard to a 100 mL volumetric flask add 70 mL of water and sonicate to dissolve and make up the volume with water. *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 ~~210nm~~ *230nm*.

#### Calculations:

Teneligliptin

$$(\% \text{ Drug Release}) = \frac{A_T}{A_S} \times \frac{W_S}{100} \times \frac{900}{LC} \times \frac{P}{100} \times \frac{426.57}{628.86} \times 100$$

Where,

A<sub>T</sub> = Absorbance due to Teneligliptin in the sample preparation.

A<sub>S</sub> = Absorbance due to Teneligliptin in the standard preparation.

W<sub>S</sub> = 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. *20 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: 15

##### • 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: 80.60

##### • 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  $\mu$  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 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)



# 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	:	289	286	TABLETS-6	:	287
TABLETS-2	:	281	283	TABLETS-7	:	284
TABLETS-3	:		282	TABLETS-8	:	286
TABLETS-4	:		292	TABLETS-9	:	291
TABLETS-5	:		299	TABLETS-10	:	286

AVERAGE WEIGHT:-  $\frac{2876}{10} = 287.6 \text{ g}$

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	283	TABLETS-6	:	286
TABLETS-2	:	285	285	TABLETS-7	:	293
TABLETS-3	:	283	283	TABLETS-8	:	289
TABLETS-4	:	285	285	TABLETS-9	:	282
TABLETS-5	:	293	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	:	Water	Rotatory Speed	:	75
Volume	:	900	Temperature	:	37°C
Apparatus	:	Paddle USP-TF	Time	:	45 min.

Standard Weight :-

Potency:-

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

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

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

$$\text{Tablet 3} = \frac{0.5401}{0.5531} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 95.52\%$$

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

$$\text{Tablet 5} = \frac{0.615}{0.5531} \times \frac{32.43 \times 5}{100 \times 50} \times \frac{900}{20} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 = 111.52\%$$

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

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

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 nm x 4.6 nm) 5 $\mu$ m
Pump mode	: Isochratic
Mobile Phase	: Buffer : Acetonitrile (80:20)
Flow rate	: 1.0 ml/min
Injection volume	: 20 $\mu$ l
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 vol<sup>m</sup> with water.
- **Sample preparation:**  
of 10 tablet and determine average wt. est. 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:

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

$$1) \frac{13736385}{14364757} \times \frac{29.48}{100} \times \frac{100}{282} \times \frac{P}{100} \times 282.33 \times \frac{426.57}{628.86} \times \frac{100}{20}$$

$$= 95.57\%$$

$$2) \frac{12706085}{14364757} \times \frac{29.48}{100} \times \frac{100}{282} \times \frac{P}{100} \times 282.33 \times \frac{426.57}{628.86} \times \frac{100}{20}$$

$$= 88.41\%$$

Average :- 91.99%

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

# ARNI ANALYTICALS

<b>TITLE</b>	HPLC DATA SHEET	
<b>Instrument Name :-</b>	High performance liquid chromatography	Page No 1 of 1
<b>Instrument Make :-</b>	Shimadzu	
<b>Instrument Model No. :-</b>	LC 2010 CH	
<b>Instrument ID :-</b>	ARNI / DMS - 001	

## DATA SHEET

**NAME OF TEST :- SYSTEM SUITABILITY**

**SYSTEM SUITABILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINE**

• **Chromatographic Conditions:**

<b>Column</b>	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
<b>Data Acquisition Time</b>	7 Minutes 10 min
<b>Pump (Flow Rate)</b>	100 ml/min 150 ml/min
<b>Port</b>	A
<b>Detector (Wavelength)</b>	273nm
<b>Column Oven Temperature</b>	30°C
<b>Degasser</b>	Off
<b>Autosampler Temperature</b>	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<sup>®</sup>

<b>TITLE</b>	HPLC DATA SHEET	
<b>Instrument Name :-</b>	High Performance liquid chromatography	Page No 1 of 1
<b>Instrument Make :-</b>	Shimadzu	
<b>Instrument Model No. :-</b>	LC2010 CHT	
<b>Instrument ID :-</b>	ARNT / DMS 001	

NAME OF STUDENT :-

DATA SHEET

NAME OF TEST :- SYSTEM SUITABILITY

SYSTEM SUITABILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINE

• Chromatographic Conditions:

Column	Stainless Steel Column Dimension L 15 cm x $\phi$ - 4.6 mm : P.S. 5 $\mu$ m L 250 mm x $\phi$ - 4.6 mm : P.S. 5 $\mu$ m
Data Acquisition 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 20 vol<sup>m</sup> of water and 30ml vol<sup>n</sup> of methanol. Mix well.

• STANDARD PREPARATION :-

wt. accurately 20 mg caffeine standard to 100ml volumetric flask. dissolve completely & make up vol<sup>m</sup> upto the mark. mix well. further dilute 5ml sol<sup>n</sup> to 50 ml volumetric flask. make up vol<sup>m</sup>.

• 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/IMS-001	
Name Of Student :-	Thorad Ashwini Raosahab	

## 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	:	100min
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  2 of 3
Instrument Make :-	Shimadzu	
Instrument Model No. :-	LC 2010 CH1	
Instrument ID :-	ARNI / JMS -001	
Name Of Student :-	Thorad Ashwini Paosach	

## • CHROMATOGRAPHIC PARAMETERS-2

INSTRUMENT PARAMETERS		Set Parameters
Data Acquisition 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 Acquisition 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  3 of 3
Instrument Make :-	Shimadzu	
Instrument Model No. :-	LC 2010 CH11	
Instrument ID :-	ARNI JNS 001	
Name Of Student :-	Thorad Ashwini Puosateb	

## • CHROMATOGRAPHIC PARAMETERS-4

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

*Apant*

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*7/12/2020*

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# ARNI ANALYTICALS

TITLE

MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE

Instrument Name :-

ANALYTICAL 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: $\pm 0.1\%$ )
1	200.0000	199.9999	199.8000 to 200.2000
2	100.0000	99.7588	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	0.150	0.4995 to 0.5005
10	0.2000 <sup>500mg</sup>	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	0.0177	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 2 of 3
Instrument Make :-	LIENSAR	
Instrument ID :-	ARNI IZHS -004	

## 2. Test for Linearity:

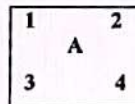
Sr. No.	Selected Weights in g	Observed Weight in g
1	50.0000 gm	50.695 gm
2	20.0000 gm	19.954 gm
3	10.0000 gm	9.998 gm

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) 199.9999		± 0.1 %
2.	At Corner 1 (B) 200.0011	B-A = 0.002	
3.	At Corner 2 (C) 200.0000	C-A = 0.0011	
4.	At Corner 3 (D) 199.9990	D-A = -0.0009	
5.	At Corner 4 (E) 199.9997	E-A = -0.0003	

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

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0.199 Limit  
not increase



# ARNI ANALYTICALS

TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-	ANALYTICAL BALANCE	Page No
Instrument Make :-	LENSAR	
Instrument ID :-	ARNI ITHS - 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	9.9996	7	9.9988	
3	9.9997	8	10.0011	
4	9.9988	9	9.9999	
5	10.0011	10	9.9998	

10.0101  
9.9904

**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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A. P. S.

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M. B. S.





# ARNI ANALYTICALS

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

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	200.8535	199.8000 to 200.2000
2	100.0000	99.778179	99.9000 to 100.1000
3	50.0000	50.708708	49.9500 to 50.0500
4	20.0000	19.05656	19.9800 to 20.0200
5	10.0000	10.0000	9.9900 to 10.0100
6	5.0000	5.08222	4.9950 to 5.0050
7	2.0000	1.12333	1.9980 to 2.0020
8	1.0000	0.92525	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	0.0355	0.0499 to 0.0501
13	0.0200	0.019	0.0199 to 0.0200
14	0.0100	0.0135	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.

Anal.  
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Mary  
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# ARNI ANALYTICALS

TITLE	MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE	
Instrument Name :-	ANLYTICAL BALANCE	Page No.
Instrument Make :-	HENSAR	
Instrument Model No. :-	DS 8000	2 of 3
Instrument ID :-	ARNI IHS 004	

## 2. Test for Linearity:

Sr. No.	Selected Weights in g	Observed Weight in g
1	2000	2000.036
2	1000	987.85
3	500	507.06

Conclusion: The observed weights are Consistent/not Consistent.

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## 3. Test for Eccentricity:

1	2
A	
3	4

$$\text{diff} = \pm 0.09528$$

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.

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# ARNI ANALYTICALS

TITLE: MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE

Instrument Name :-	ANALYTICAL BALANCE	Page No.
Instrument Make :-	LIENSER	
Instrument Model No :-	DS-8000	3 of 3
Instrument ID :-	MHT ITHS 7004	

**4. Test for Repeatability:**

Selected Weight in g

diff = 0.1002

Sl. No.	Observed Weight in g	Sl. 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.

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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 ( $\pm 0.05$ )
1	14/12/22	3.91	6.81

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# ARNI ANALYTICALS

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

## DAILY CALIBRATION RECORD

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

• pH-4.01 :-

- Transfer the capsule content in a 100ml volumetric flask using a funnel.
- Dissolve the contents in 10 ml of distilled water and then make it up to 100 ml with distilled water.
- This solution will have a pH of  $4.0 \pm 0.05$  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	15-12-22	4.01	6.95

slope 97%  
slope 97%

*Arif*  
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*M. K. K.*  
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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	Thorad Ashwini Raosateb	

## 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

	<b>ARNI ANALYTICALS</b>	
<b>TITLE</b>	DISSOLUTION TEST APPARATUS WORKSHEET	
<b>Instrument Name :-</b>	DISSOLUTION TEST APPARATUS	<b>Page No.</b>
<b>Instrument ID :-</b>	ARNI / IMS 003	
<b>Instrument Model No. :-</b>	DS 8000	1 of 1
<b>Name Of Students</b>	Thorad Ashwini Raosateh	

## 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 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 aliquote.

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Ara

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Ara



# ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV - SPECTROPHOTOMETER	Page No.
Instrument ID :-	ARNI / JHS - 002	
Instrument Model No. :-	LN5P - UV 100B	
Name Of Students	Tharal Ashwini Paosaleb	1 of 1

NAME OF PRODUCT	1	caffeine
WORKING STANDARD NO.	1	-
POTENCY	1	-
INSTRUMENT ID	1	ARNI / JHS - 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<sup>m</sup> with water. further dilute 5 ml of above sol<sup>n</sup> to 50 ml with water.

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

OBSERVATIONS:-

MAXIMUM ABSORPTION WAVELENGTH - 274 nm

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# ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV -SPECTPHOTOMETER	Page No.
Instrument ID :-	ARNI / INS -002	
Instrument Model No. :-	LNSP -UV 100B	1 of 1
Name Of Students	Thorat Ashwini Paosateb	

## 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.

### UV-SPECTROPHOTOMETER WAVELENGTH :- 273nm

Wt. accurately 10 mg of caffeine standard in a 100 ml volumetric flask. dissolve completely and make up vol<sup>n</sup>. further dilute 5 ml dissolve 50 ml with water.

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