

„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 **Mr. Akshay Anil Nannaware** 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**.

*A. Nannaware*  
**HOD Chemistry**  
**HEAD**

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

*A. Nannaware*  
**Examiner**

*A. Nannaware*  
**Principal**

**PRINCIPAL**  
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This is to Certify that ..... **Akshay Anil Nannaware**.....  
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

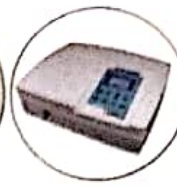
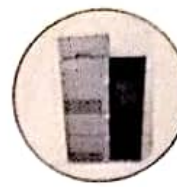
Director







**ARNI**  
ANALYTICALS



☎ : 9307686710

## Certificate

### “Pharmaceutical Training Course in Analytical Techniques”

This is to certify that Mr./Miss/ Mrs. **Akshay Anil Nannawarer** 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



## 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:- Akshay Anil Nannaware

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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

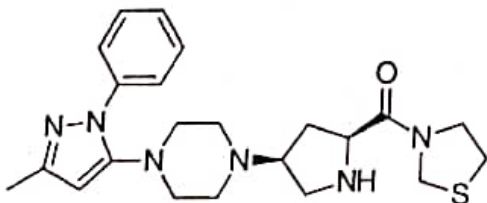
### Physical Testing-

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 2 of 4

24/12/2022

### 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	:	287	TABLETS-6	:	291
TABLETS-2	:	285	TABLETS-7	:	281
TABLETS-3	:	286	TABLETS-8	:	284
TABLETS-4	:	285	TABLETS-9	:	287
TABLETS-5	:	286	TABLETS-10	:	298

**AVERAGE WEIGHT:-**  $\frac{2868}{10} = 286.8$

**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.

TABLETS-1	:	287	TABLETS-6	:	291
TABLETS-2	:	285	TABLETS-7	:	281
TABLETS-3	:	286	TABLETS-8	:	284
TABLETS-4	:	285	TABLETS-9	:	287
TABLETS-5	:	286	TABLETS-10	:	298

**MINIMUM WEIGHT :-** ~~298~~ 281

**MAXIMUM WEIGHT :-** ~~281~~ 298

**LIMIT: 283 MG  $\pm$  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 ± 0.5°C
Apparatus	:	USP Type II (paddle)	Time	:	45 minutes.

#### Standard Weight :-

#### Potency:-

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

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

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

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

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

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

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

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

#### Average:-

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



# ARN ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 4 of 4

### 6) Assay (By HPLC):

#### Chromatographic Conditions:

Column	: C18 (150mm X 4.6 mm) 5µm
Pump mode	: Isocratic
Mobile Phase	: Buffer : acetonitrile (60:40)
Flow rate	: 1.0 mL/min.
Injection volume	: 20µl
Column Temperature	: 30°C
Wavelength	: UV, 210 nm

#### Preparation of solutions:

##### • Standard preparation:

Dissolve 0.1M potassium dihydrogen orthophosphate in 1000 ml of water. prepare mixture Buffer acetonitrile (65:35), filter through 0.45µm filter and degas.

##### • Sample preparation:

Weight accurately 286mg teneligliptin.

① 10 Tablet crushed in mortar pestle, accurately 286mg powder teneligliptin. Then add 70 ml water in volumetric flask, & the 20 min shaking in sonicator & then make up the volume

Standard Weight :-

Sample Weight :-

Average Weight :-

Potency :-

##### • Calculations:

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

$$1) \frac{15649140}{1237379} \times \frac{29.48}{100} \times \frac{100}{286} \times \frac{99.85}{100} \times 286 \times \frac{426.57}{628.86} \times \frac{100}{20}$$

$$= 128.72$$

$$2) \frac{15649140}{12445266} \times \frac{29.48}{100} \times \frac{100}{286} \times \frac{99.85}{100} \times 286 \times \frac{426.57}{628.86} \times \frac{100}{20}$$

$$= 128.53$$

Average :-

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)

$$\textcircled{1} \frac{15649140}{12137379} \times \frac{29.68}{100} \times \frac{100}{285} \times \frac{99.81}{100} \times 286 \times \frac{426.57}{628.86} \times \frac{100}{20} = 128.72$$

$$\textcircled{2} \frac{15649140}{10902060} \times \frac{29.68}{1}$$

$$\frac{12448266}{}$$

Start





# ARNI ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 2 of 5

### TEST METHOD

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

2) **Identification:**

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

3) **Average weight:**

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

**Calculation:**

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

Where, W= Weight of 10 tablets in mg

**Limit: 283 mg ± 7.5%**

4) **Uniformity of Weight:**

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

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



# ARNI ANALYTICAL

## FINISHED PRODUCT SPECIFICATION AND TEST METHOD

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG

PAGE NO.:- Page 3 of 5

### 5) Dissolution (By HPLC):

#### Dissolution Parameters :

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

### 11.1 Preparation of Solutions :

#### • Standard preparation :

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

#### • Sample preparation:

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

At the end of specified time intervals (after 45 minutes), withdraw 10 mL aliquot from a zone midway between the surface of the dissolution medium and the top of the rotating paddle and filter through 0.45µ filter paper discarding first few mL of the filtrate. Inject directly.  $\rightarrow \frac{20}{300} \rightarrow 0.0222 \times 1000 = 22.2$

### 11.2 Procedure:

Measure the absorbance of the resulting solution at 210nm. *Photometric scan*

#### Calculations:

Teneligliptin

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

Where,

$A_T$  = Absorbance due to Teneligliptin in the sample preparation.

$A_S$  = Absorbance due to Teneligliptin in the standard preparation.

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

$P$  = Purity of Teneligliptin hydrobromide hydrate working standard used for standard

$LC$  = Label claim of a tablet, in mg. *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:

##### • 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.

$$\begin{aligned} 1M &= 136.086 - 1000 \\ 0.1M &= 13.609 - 1000 \end{aligned} \quad \text{m.m.l} = 136.09 \times 0.2 \times 2 = 54.436$$

Handwritten calculation:  $136.09 \times 0.2 \times 2 = 54.436$

#### 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.

$$\frac{20}{100} \times \frac{5}{50} = 0.2 \text{ ppm}$$

##### • 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 preparator
- $A_S$  = 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. 20mg
- $A_W$  = 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**

# ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	High pressure liquid chromatography	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CH	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	A stainless steel column (C <sub>18</sub> ) Diameter - length 15 cm x diameter - 4.6 mm particle size 5 μm, Length 150 mm x diameter - 4.6 - 5 μm. (C <sub>18</sub> ) column
Data Acquisition Time	10 min
Pump (Flow Rate)	1.00 ml/min
Pct	A
Detector (Wavelength)	273 nm
Column Oven Temperature	30°C
Degasser	OFF
Autosampler Temperature	OFF

• MOBILE PHASE PREPARATION :-

- prepare mixture of ~~80~~ 140 ml of water and 60 ml of methanol. mix well.

• STANDARD PREPARATION :-

- Weigh accurately 20 mg of caffeine standard to a 100 ml volumetric flask. Add 60 ml of HPLC grade water and shake to dissolve completely. Now make up the volume up to mark. mix well. Further dilute 5 ml of the above solution to 50 ml volumetric flask, dilute with water to make up volume.

• SEQUENCE OF INJECTION :-

Name of Solution	No. Of Injection
Blank	0
Standard	3

ANALYSED BY

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# ARNI ANALYTICALS

<b>TITLE</b>	<b>HPLC DATA SHEET</b>	
<b>Instrument Name :-</b>	High pressure liquid chromatography	<b>Page No</b>
<b>Instrument Make :-</b>	SHIMADZU	
<b>Instrument Model No. :-</b>	LC 2010 CHT	<b>1 of 1</b>
<b>Instrument ID :-</b>	ARNI/INS-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 <b>Dimensions :-</b> 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
<b>Pump (Flow Rate)</b>	1.00 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 80 volumes of Water and 20 volumes of Methanol. Mix well. 200 ml

$$\begin{array}{r} 70 \\ + 30 \\ \hline 100 \end{array}$$

• **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 make up the volume upto the mark. Mix well. Further dilute 5ml of the above solution to 50ml volumetric flask, dilute with water to make up volume.



<b>AN</b>	<b>ARNI ANALYTICALS</b>	
<b>TITLE</b>	<b>HPLC DATA SHEET</b>	
<b>Instrument Name :-</b>	High Pressure Liquid chromatography <i>(performance)</i>	<b>Page No</b>
<b>Instrument Make :-</b>	SHIMADZU	
<b>Instrument Model No. :-</b>	LC 2010 CHT	<b>1 of 3</b>
<b>Instrument ID :-</b>	ARNI/INS-001	
<b>Name Of Student :-</b>	Nannawate Akshay Anil	

### 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
<b>Data Aquisition Time</b>	:	.15 min
<b>Pump</b>	:	1.00 ml/min
<b>Port</b>	:	A
<b>Detector (Wavelength)</b>	:	210 nm
<b>Column Oven Temperature</b>	:	30°C
<b>Degasser</b>	:	on
<b>Autosampler Temperature</b>	:	10°C

*Ashay*  
**ANALYSED BY**

*M. B. U.*  
**CHECKED BY**



# ARNI ANALYTICALS

<b>TITLE</b>	HPLC DATA SHEET	
Instrument Name :-	High <sup>(performance)</sup> pressure liquid chromatography	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010 CHT	2 of 3
Instrument ID :-	ARNI/INS-001	
Name Of Student :-	Nannaware Akshay Anil	

### CHROMATOGRAPHIC PARAMETERS-2

INSTRUMENT PARAMETERS		Set Parameters
Data Acquisition Time	:	8 min
Pump	:	0.80 ml/min
Port	:	B
Detector (Wavelength)	:	222 nm
Column Oven Temperature	:	40°C
Degasser	:	on
Autosampler Temperature	:	7°C

### CHROMATOGRAPHIC PARAMETERS-3

INSTRUMENT PARAMETERS		Set Parameters
Data Acquisition Time	:	17 min
Pump	:	1.20 ml/min
Port	:	C
Detector (Wavelength)	:	270 nm
Column Oven Temperature	:	43°C
Degasser	:	on
Autosampler Temperature	:	15°C

*Akshay*  
**ANALYSED BY**

*Trace*  
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# ARNI ANALYTICALS

TITLE	HPLC DATA SHEET	
Instrument Name :-	High pressure Liquid chromatography	Page No
Instrument Make :-	SHIMADZU	
Instrument Model No. :-	LC 2010CHT	3 of 3
Instrument ID :-	ARNI/INC-001	
Name Of Student :-	Nannaware Akshay Anil	

## • CHROMATOGRAPHIC PARAMETERS-4

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisition Time	:	47 min
Pump	:	1.50 ml/min
Port	:	D
Detector (Wavelength)	:	260 nm
Column Oven Temperature	:	40°C
Degasser	:	on
Autosampler Temperature	:	13°C

## • CHROMATOGRAPHIC PARAMETERS-5

INSTRUMENT PARAMETERS		Set Parameters
Data Aquisition Time	:	31 min
Pump	:	1.20 ml/min
Port	:	A
Detector (Wavelength)	:	265 nm
Column Oven Temperature	:	45°C
Degasser	:	OFF
Autosampler Temperature	:	8°C

Akshay  
ANALYSED BY

Akshay  
CHECKED BY



# ARNI ANALYTICALS

TITLE

MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE

Instrument Name :-

Analytical balance.

Page No

Instrument Make :-

WENSAR

1 of 3

Instrument ID :-

ARNI / JNS-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: $\pm 0.1\%$ )
1	200.0000	200.0 g.	199.8000 to 200.2000
2	100.0000	98.754 g.	99.9000 to 100.1000
3	50.0000	50.690 g.	49.9500 to 50.0500
4	20.0000	19.046 g.	19.9800 to 20.0200
5	10.0000	09.999 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	0.922 g.	0.9990 to 1.0010
9	0.5000	0.153 g.	0.4995 to 0.5005
10	0.2000	0.105 g.	0.1998 to 0.2002
11	0.1000	0.056 g.	0.0999 to 0.1001
12	0.0500	0.025 g.	0.0499 to 0.0501
13	0.0200	0.013 g.	0.0199 to 0.0200
14	0.0100	0.012 g.	0.0099 to 0.0100
15	0.0050	0.008 g.	0.0049 to 0.0051

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

ANALYSED BY

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# ARNI ANALYTICALS

TITLE		MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE
Instrument Name :-	Analytical balance	Page No 2 of 3
Instrument Make :-	WENCAP	
Instrument ID :-	ARNI/JNS-004	

## 2. Test for Linearity:

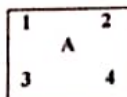
Sr. No.	Selected Weights in g	Observed Weight in g
1	20 gm	19.049 g.
2	50 gm	50.692 g.
3	100 gm	98.755 g.

Conclusion: The observed weights are Consistent/not Consistent.

ANALYSED BY  
Abhay

CHECKED BY  
M. N. N.

## 3. Test for Eccentricity:



Sr. No.	Weight Observed in g	Difference in g	Limit
1.	At Centre- (A) 50 gm.	50.696	±0.1% = 0.0506
2.	At Corner 1 (B) 50.694	B-A = -0.002	
3.	At Corner 2 (C) 50.695	C-A = -0.001	
4.	At Corner 3 (D) 50.691	D-A = -0.005	
5.	At Corner 4 (E) 50.695	E-A = -0.001	

$$\frac{50.696 \times 0.1}{100} = 0.0506$$

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

ANALYSED BY  
Abhay

CHECKED BY  
M. N. N.



# ARNI ANALYTICALS

TITLE

MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE

Instrument Name :-

pH meters. Analytical balance.

Page No

Instrument Make :-

WENSHAR

Instrument ID :-

ARNI/INLS-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	98.754 gm	6	98.757 gm	± 0.1 %
2	98.756 gm	7	98.758 gm	
3	98.755 gm	8	98.757 gm	
4	98.755 gm	9	98.759 gm	
5	98.756 gm	10	98.754	

$$0.1\% \text{ Formula} = \frac{98.759 \times 0.1}{100} = 0.098759 \pm 98.759 \pm 0.098759 = 98.8577$$

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

$$\text{minimum value} = 98.759 - 0.098759 = 98.6602$$

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

ANALYSED BY  
Abhay

CHECKED BY  
Bhargava



# ARNI ANALYTICALS

25/12/2022

## MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE

TITLE	Analytical Balance		Page No. 1 of 3
Instrument Name :-	WIENSAR		
Instrument Make :-	DS8000		
Instrument Model No. :-	ARNI/INS-004		
Instrument ID :-			

NAME OF STUDENT:-

### MONTHLY CALIBRATION RECORD

1. Calibration by using Standard certified weights:

Observation Table:

No.	Reference Weight in g	Observed Weight in g	Weight in g (Limit: $\pm 0.1\%$ )
1	200.0000	200.016	199.8000 to 200.2000
2	100.0000	99.770	99.9000 to 100.1000
3	50.0000	50.700	49.9500 to 50.0500
4	20.0000	19.052	19.9800 to 20.0200
5	10.0000	9.998	9.9900 to 10.0100
6	5.0000	5.082	4.9950 to 5.0050
7	2.0000	1.230	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	0.103	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	0.016	0.0199 to 0.0200
14	0.0100	0.018	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 :-

Analytical Balance

Page No.

Instrument Make :-

WENSTAR

Instrument Model No. :-

DS8000

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	19.054 gm.
2	50 gm	50.700 gm
3	100 gm	98.768 gm

Conclusion: The observed weights are Consistent/not Consistent.

ANALYSED BY  
Abhay

CHECKED BY:-  
Abhay

## 3. Test for Eccentricity:

1	2	$\beta = 98.772$
	A	$c = 98.773$
3	4	$D = 98.770$
		$E = 98.767$

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	
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.

Calculation -  $\beta - A = 98.772 - 98.769 = 0.003$   
 $C - A = 98.773 - 98.769 = 0.004$   
 $D - A = 98.770 - 98.769 = 0.001$   
 $E - A = 98.767 - 98.769 = -0.002$

} ± 0.1%

ANALYSED BY  
Abhay

CHECKED BY:-  
Abhay





# 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.

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	98.769	6	98.771	± 0.1 %
2	98.772	7	98.770	
3	98.771	8	98.769	
4	98.770	9	98.768	
5	98.771	10	98.770	

Calculations =  $\frac{98.772 \times 0.1}{100} = 0.098772$  | 1) max value -  $98.772 + 0.098772 = 98.8707$

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

2) min value -  $98.768 - 0.098772 = 98.6692$

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

*Ashay*

ANALYSED BY

*Abhi*

CHECKED BY:-



# ARNI ANALYTICALS

## 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-004005	

### 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.	20/12/2022	3.93	6.78

slope - 96%

Ashay  
PERFORMED BY

Nancy  
CHECKED BY



# ARNI ANALYTICALS

## DISSOLUTION TEST APPARATUS WORKSHEET

Instrument Name :-	DISSOLUTION TEST	Page No.
Instrument ID :-	ARNI/JNS-003	
Instrument Model No. :-	DS 8000	1 of 1
Name Of Students	Nannaware Akshay Adil	

### 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.

Akshay  
**ANALYSED BY**

**CHECKED BY:-**

# ARNI ANALYTICALS

TITLE	DISSOLUTION TEST APPARATUS WORKSHEET	
Experiment Name :-	DISSOLUTION TEST	Page No.
Instrument ID :-	ARNI/INS-003	
Instrument Model No. :-	DS-8000	1 of 1
Name Of Students	Nannware Akshay Anil	

## NAME OF TEST :-

TRIAL FOR DISSOLUTION TEST.

## DISSOLUTION CONDITIONS:-

Dissolution Media	Water
Media Volume	900 ml.
Apparatus	USP TYPE II PADDLE
RPM	100
Temperature	$37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$
Time	45 minutes.

## PREPARATIONS:-

900 ml of volume of water in bowl. Allow sufficient time for the dissolution medium to equilibrate at  $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ . Adjust stirring element speed 100 rpm. Each one capsule in each of six paddle and adjust the paddle in the dissolution so that there is a distance  $25\text{mm} \pm 2\text{mm}$  between 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 rotating paddle. Further dilute 2 ml of the above solution to 25 ml with dissolution medium.

Akshay  
ANALYSED BY

K. B. B. B.  
CHECKED BY:-





# ARNI ANALYTICALS

TITLE	UV-SPECTROPHOTOMETER WORKSHEET	
Instrument Name :-	UV - Spectrophotometer.	Page No.
Instrument ID :-	ARNI / JNS - 002	
Instrument Model No. :-	LMSP - UV - 100B	1 of 1
Name Of Students	Nannawate Akshay Anil	

## 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

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

ANALYSED BY

CHECKED BY:-





# ARNI ANALYTICALS

TITLE

UV-SPECTROPHOTOMETER WORKSHEET

Instrument Name :-

UV-spectrophotometer worksheet

Page No.

Instrument ID :-

ARNI/INS-002

Instrument Model No. :-

UMSP-UV-100B

Name Of Students

Nannaware Akshay ANI

1 of 1

Date:-

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

**NAME OF TEST :-** absorbance of caffeine (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 solution to 50 ml with water.

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

Weight caffeine - 10 mg

Wavelength - 273 nm - maximum

206 - minimum.

**OBSERVATIONS:-** caffeine wavelength - 273 nm

**MAXIMUM ABSORPTION WAVELENGTH** - .

273 nm

Akshay  
**ANALYSED BY**

M. S. S.  
**CHECKED BY:-**

