„"INDUSTRIAL TRAINING""

A REPORT SUBMITTED TO

**SAVITRIBAI PHULE PUNE UNIVERSITY, PUNE**

FOR THE DEGREE OF

**MASTER OF SCIENCE**

**IN**

**ORGANIC CHEMISTRY**

UNDER THE FACULTY OF SCIENCE

**BY**

Miss. Seema Kailas Bhise

Department of Chemistry, G. M. D.Arts,B.W. Commerce and

Science College, Sinnar

UNDER THE GUIDANCE OF

Prof. :- Dr. M.R. Gaware

Head of

**DEPARTMENT OF CHEMISTRY**

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

SINNAR 422103

**APRIL 2023**

**Maratha Vidya Prasarak Samaj's**

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

SINNAR, DISTRICT- NASHIK

DEPARTMENT OF CHEMISTRY (PG)

# CERTIFICATE

This is to certify that **Miss. Seema Kailas Bhise** 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.

Bawaze

**HOD Chemistry**

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

Examined on 12-05-2023

**Examiner**

**Principal**

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

ARNI
ANALYTICAL

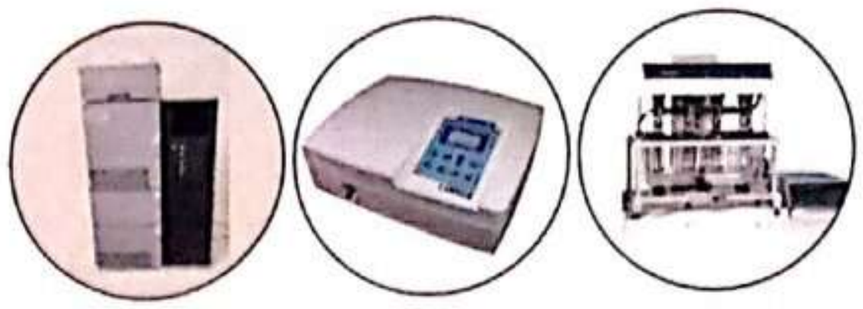
**Add.:** Pushpak Apartment, Flat No. 102, Lane No. 3, Near Neurocare Hospital, Pandit Colony, Nashik.
**e-mail :** arnianalyticals@gmail.com | Web Site : www.arnianalyticals.com

# Certificate

This is to Certify that ..... *Seema Kailas Bhise* .....
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. **Seema Kailas Bhise** 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:- Seema Kailas Bhise**

# INDEX

# TENELIGLIPTIN

**Introduction-**

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

**Chemical Formula-** $C_{22}H_{30}N_6O_5$

**Molar Mass-** 426.58 gm/mol

- Teneligliptin significantly controls glycemic parameters with safety. No dose adjustment is required.
- As we all know that teneligliptin tablet contains only 20 mg active ingredient i.e. teneligliptin. Other layers or coatings are excepients.
- Once a tablet is formulated then directly it doesn't comes to market. First of all some of the random tablets are collected and forwarded for testing.

**Testing have 2 types-**

1. Physical
2. Chemical

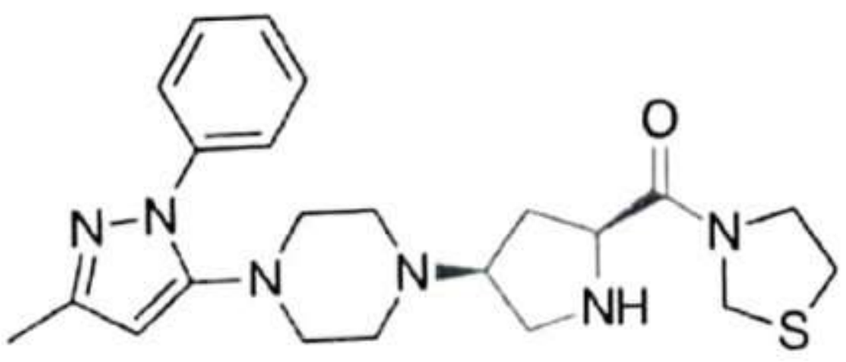
**Physical Testing-**

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

**Chemical Tests-**

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

**Structure of Teneligliptin-**



## SPECIFICATION AND TESTS OF TENELIGLIPTIN TABLETS 20 MG

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

## TEST METHOD

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

**2) Identification:**

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

**3) Average weight:**

| | | | | | |
|---|---|---|---|---|---|
| TABLETS-1 | : | 290 | TABLETS-6 | : | 286 |
| TABLETS-2 | : | 285 | TABLETS-7 | : | 286 |
| TABLETS-3 | : | 284 | TABLETS-8 | : | 281 |
| TABLETS-4 | : | 286 | TABLETS-9 | : | 279 |
| TABLETS-5 | : | 291 | TABLETS-10 | : | 297 |

**AVERAGE WEIGHT:-** $\frac{2866}{10} = 286$ **LIMIT: 283 MG ± 7.5%**

**4) Uniformity of Weight:**

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

| | | | | | |
|---|---|---|---|---|---|
| TABLETS-1 | : | 290 | TABLETS-6 | : | 286 |
| TABLETS-2 | : | 285 | TABLETS-7 | : | 286 |
| TABLETS-3 | : | 284 | TABLETS-8 | : | 281 |
| TABLETS-4 | : | 286 | TABLETS-9 | : | 279 |
| TABLETS-5 | : | 291 | TABLETS-10 | : | 297 |

**MINIMUM WEIGHT :-** 281
**MAXIMUM WEIGHT :-** 297

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

# ARN ANALYTICAL

**FINISHED PRODUCT SPECIFICATION AND TEST METHOD**

NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG | 

**5) Dissolution (By HPLC):**

| **Dissolution Parameters :** | | | | | |
|---|---|---|---|---|---|
| **Medium** | : | Water | **Rotatory Speed** | : | 75 rpm |
| **Volume** | : | 900ml | **Temperature** | : | 37°c ± 0.5°c |
| **Apparatus** | : | USP Type II (paddle) | **Time** | : | 45 minutes |

**Standard Weight :-**

**Potency:-**

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

**Tablet 1**= $\frac{0.6712}{0.5566} \times \frac{32.43}{100} \times \frac{900}{20} \times \frac{5}{50} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 119.1%.

**Tablet 2**= $\frac{0.6352}{0.5566} \times \frac{32.43}{100} \times \frac{900}{20} \times \frac{5}{50} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 112.8%.

**Tablet 3**= $\frac{0.5753}{0.5566} \times \frac{32.43}{100} \times \frac{900}{20} \times \frac{5}{50} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 102.1%.

**Tablet 4**= $\frac{0.6360}{0.5566} \times \frac{32.43}{100} \times \frac{900}{20} \times \frac{5}{50} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 113.9%.

**Tablet 5**= $\frac{0.7426}{0.5566} \times \frac{32.43}{100} \times \frac{900}{20} \times \frac{5}{50} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 131.7%.

**Tablet 6**= $\frac{0.5585}{0.5566} \times \frac{32.43}{100} \times \frac{900}{20} \times \frac{5}{50} \times \frac{99.85}{100} \times \frac{426.57}{628.86} \times 100 =$ 99.19 %.

**Average:-** $\frac{119.1\% + 112.8\% + 102.1\% + 113.9\% + 131.7\% + 99.19}{6} = 113.00\ \%.$

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

**6) Assay (By HPLC):**

**Chromatographic Conditions:**

| | | |
|---|---|---|
| Column | : | C18, (150mm x 4.6mm), 5um |
| Pump mode | : | Isocratic |
| Mobile Phase | : | Buffer : Acetonitrile (60:40) |
| Flow rate | : | 1.0 ml/min |
| Injection volume | : | 20 ul |
| Column Temperature | : | 30°C |
| Wavelength | : | UV, 250nm |

**Preparation of solutions:**

- **Standard preparation:** weigh & transfer accurately about 20mg of teneligliptin (29.48 mg) Teneligliptin Hydrobromide Hydrate) working standared 100ml Volumetric flask & 70ml of water & sonicate to dissolve make up Volume.
- **Sample preparation:** weigh 10 tablets & determine average weight. Crush the tablets to fine powder. weigh & transfer powder equvalent to 20mg of Teneligliptin to 100 ml V. Flask.

Standard Weight :- 29.48
Sample Weight :- 287
Average Weight :- 286.9
Potency :- 99.85

- **Calculations:**

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

1) $\frac{13736385}{14490846.5} \times \frac{29.48}{100} \times \frac{100}{287} \times \frac{99.85}{100} \times 286.9 \times \frac{426.57}{628.86} \times \frac{100}{20} = 94.34\%$

=

2) $\frac{11688085}{11490846.5} \times \frac{29.48}{100} \times \frac{100}{287} \times \frac{99.85}{100} \times 286.9 \times \frac{426.57}{628.86} \times \frac{100}{20} = 80.49\%$

=

Average :- $\frac{94.34 + 80.49}{2} = 87.41\%$

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

## TEST METHOD

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

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

3) **Average weight:**
Weigh together 10 tablets selected at random and calculate the average weight.

**Calculation:**

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

Where, W= Weight of 10 tablets in mg

**Limit: 283 mg ± 7.5%**

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

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

**5) Dissolution (By HPLC):**

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

**11.1 Preparation of Solutions :**

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

- **Sample preparation:**
  Pour 900 mL of dissolution medium in each vessel. Allow sufficient time for the dissolution medium to equilibrate at 37°C ± 0.5°C. Adjust stirring element speed to 75 rpm. Place one tablet in each of the six vessels and immerse the paddles in the dissolution medium so that there is a distance of 25mm ± 2mm between the bottom of the paddle and inside bottom of the vessel. Start the apparatus.
  At the end of specified time intervals (after 45 minutes), withdraw 10 mL aliquot from a zone midway between the surface of the dissolution medium and the top of the rotating paddle and filter through 0.45µ filter paper discarding first few mL of the filtrate. Inject directly.

**11.2 Procedure:**

Measure the absorbance of the resulting solution at 210nm.

**Calculations:**

Teneligliptin

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

Where,

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

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

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

P = Purity of Teneligliptin hydrobromide hydatre working standard used for standard

LC = Label claim of a tablet, in mg.

426.57 = Molecular weight of Teneligliptin

628.86 = Molecular weight of Teneligliptin hydrobromide hydatre

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

Normal phase
Reverse phase

# ARNI ANALYTICAL

**FINISHED PRODUCT SPECIFICATION AND TEST METHOD**

| NAME OF PRODUCT : TENELIGLIPTIN TABLETS 20 MG | PAGE NO.:- Page 4 of 5 |
|---|---|

**13) Assay (By HPLC):**

- **Reagents Required:**

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

**13.1 Chromatographic Conditions:**

| | | |
|---|---|---|
| Column | : | C18, (150 mm X 4.6 mm), 5µm |
| Pump mode | : | Isocratic |
| Mobile Phase | : | Buffer : Acetonitrile (60:40) |
| Flow rate | : | 1.0 mL/min |
| Injection volume | : | 20 µl |
| Column Temperature | : | 30°C |
| Wavelength | : | UV, 210 nm |
| Run time | : | 1.5 times of the retention time of principle peak |

**13.2 Preparation of Mobile Phase:**

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

**13.3 Preparation of solutions:**

- **Standard preparation:**
  Weigh and transfer accurately about 20 mg of Teneligliptin (29.48 mg Teneligliptin Hydrobromide Hydrate) working standard to a 100 mL volumetric flask add 70 mL of water and sonicate to dissolve and make up the volume with water.
- **Sample preparation:** - 1 tablet weight / 3-tablet
  Weigh 10 tablets and determine average weight. Crush the tablets to a fine powder. Weigh and transfer powder equivalent to 20 mg of Teneligliptin to a 100 mL dry volumetric flask. Add 70 mL of water, sonicate for not less than 20 minutes with intermittent shaking. Make up the volume with water. Filter through 0.45 µ Nylon filter discarding first few mL of the filtrate.

**13.4 Evaluation of System Suitability:**

Equilibrate the column with mobile phase with the chromatographic conditions for stable baseline. Inject blank and record the chromatogram. Inject standard preparation in five replicates and record the chromatograms. It should comply with the system suitability criteria as mentioned.

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

**13.5 Procedure:**

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

- **Calculations:**

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

Where,

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

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

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

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

LC = Label claim in mg.

$A_W$ = Average weight in mg.

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

426.57 = Molecular weight of Teneligliptin.

628.86 = Molecular weight of Teneligliptin Hydrobromide Hydrate

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

Name :- Bhise Seema Kailas.

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

**NAME OF STUDENT :-**

**DATA SHEET**

**NAME OF TEST :- SYSTEM SUITABILITY**

**SYSTEM SUITIBILITY CHECK BY INJECTING 3 REPLICATE INJECTIONS OF CAFFEINCE**

- **Chromatographic Conditions:** A Stainless Steel Column

| | |
|---|---|
| **Column** | |
| **Data Aquisitation Time** | 7 min |
| **Pump (Flow Rate)** | 1.00 ml / min |
| **Port** | A |
| **Detector (Wavelength)** | 273 nm |
| **Column Oven Temperature** | 30° C |
| **Degasser** | off |
| **Autosampler Temperature** | off |

- **MOBILE PHASE PREPARATION :-**

  Prepare a Mixture of 80 Volumes of water and 20ml Volumes of Methonol. Mix well.

- **STANDARD PREPARATION :-**

  Weigh Accurately 20mg of Caffeine standared to 100ml Volumetric flask. Add 60 ml of Hplc grade water and shake to dissolve Completely. slowly make up Volume to mark. Mix well. dilute 5ml of above solution to 50 ml Volumetric flask, dilute with water to makeup Volume.

- **SEQUENCE OF INJECTION :-**

| Name of Solution | No. Of Injection |
|---|---|
| Blank | — |
| Standard | 3 |

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| | ARNI ANALYTICALS | |
|---|---|---|
| TITLE | HPLC DATA SHEET | |
| Instrument Name :- | HPLC | Page No |
| Instrument Make :- | SHIMADZU | |
| Instrument Model No. :- | LC 2010 CHT | 1 of 3 |
| Instrument ID : - | ARNI / INS - 001 | |
| Name Of Student :- | Bhise seema kailas | |

## HPLC DATA SHEET

- HPLC Parameter settings:
  - Make a purging of the mobile phase of all ports & injection port to remove the air bubble from the line.
  - Create a new method by using below parameter.
  - Save the Method Parameters with a file name.
  - Download the method to the instruments.

- CHROMATOGRAPHIC PARAMETERS-1

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

ANALYSED BY

CHECKED BY

| | ARNI ANALYTICALS | |
|---|---|---|
| TITLE | HPLC DATA SHEET | |
| Instrument Name :- | Hplc | Page No |
| Instrument Make :- | SHI-MAD2U | |
| Instrument Model No. :- | LC-2010 CHT | 2 of 3 |
| Instrument ID : - | ARNT / TNS - 001 | |
| Name Of Student :- | Bhise seema kailas | |

- **CHROMATOGRAPHIC PARAMETERS-2**

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

- **CHROMATOGRAPHIC PARAMETERS-3**

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

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| | ARNI ANALYTICALS | |
|---|---|---|
| **TITLE** | **HPLC DATA SHEET** | |
| **Instrument Name :-** | HPLC | **Page No** |
| **Instrument Make :-** | SHIMADZU | |
| **Instrument Model No. :-** | LC-2010 CHT | **3 of 3** |
| **Instrument ID : -** | ARNI/INS-001 | |
| **Name Of Student :-** | Bhise seema kailas. | |

- **CHROMATOGRAPHIC PARAMETERS-4**

| INSTRUMENT PARAMETERS | | Set Parameters |
|---|---|---|
| **Data Aquisitation Time** | : | 30min |
| **Pump** | : | 1.50ml/min |
| **Port** | : | B |
| **Detector (Wavelength)** | : | 260nm |
| **Column Oven Temperature** | : | 30°c |
| **Degasser** | : | off |
| **Autosampler Temperature** | : | 15°c |

- **CHROMATOGRAPHIC PARAMETERS-5**

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

Bhise

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**CHECKED BY**

| | ARNI ANALYTICALS | |
|---|---|---|
| TITLE | MONTHLY CALIBRATION RECORD OF ANALYTICAL BALANCE | |
| ~~Instrument~~ Name :- | Bhise Seema Kailas. | Page No |
| Instrument Make :- | Wensar | |
| Instrument ID : - | ARNI/INS-004 | 1 of 3 |

Instrument Name- Analytical Balance

**MONTHLY CALIBRATION RECORD**

**1. Calibration by using Weights:**

**Observation Table:**

| Sr. No. | Reference Weight in g | Observed Weight in g | Weight in g (Limit: ± 0.1%) |
|---|---|---|---|
| 1 | 200.0000 | 199.989 | 199.8000 to 200.2000 |
| 2 | 100.0000 | 98.754 | 99.9000 to 100.1000 |
| 3 | 50.0000 | 50.696 | 49.9500 to 50.0500 |
| 4 | 20.0000 | 19.049 | 19.9800 to 20.0200 |
| 5 | 10.0000 | 9.999 | 9.9900 to 10.0100 |
| 6 | 5.0000 | 5.080 | 4.9950 to 5.0050 |
| 7 | 2.0000 | 1.326 | 1.9980 to 2.0020 |
| 8 | 1.0000 | 0.925 | 0.9990 to 1.0010 |
| 9 | 0.5000 | 0.152 | 0.4995 to 0.5005 |
| 10 | 0.2000 | 0.103 | 0.1998 to 0.2002 |
| 11 | 0.1000 | 0.058 | 0.0999 to 0.1001 |
| 12 | 0.0500 | 0.040 | 0.0499 to 0.0501 |
| 13 | 0.0200 | 0.016 | 0.0199 to 0.0200 |
| 14 | 0.0100 | 0.012 | 0.0099 to 0.0100 |
| 15 | 0.0050 | 0.005 | 0.0049 to 0.0051 |

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

10/12/22

**ANALYSED BY**

**CHECKED BY**

Bhise seema kailas

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

**2. Test for Linearity:**

| Sr. No. | Selected Weights in g | Observed Weight in g |
|---|---|---|
| 1 | 10 gm | 9.999 gm |
| 2 | 20 gm | 19.050 gm |
| 3 | 50 gm | 50.695 gm |

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

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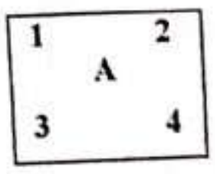
**3. Test for Eccentricity:**




| Sr. No. | Weight Observed in g | | Difference in g | Limit |
|---|---|---|---|---|
| 1. | At Centre- (A) | 19.052 | | |
| 2. | At Corner 1 (B) | 19.053 | B-A = 0.001 | |
| 3. | At Corner 2 (C) | 19.050 | C-A = -0.002 | ± 0.1 % |
| 4. | At Corner 3 (D) | 19.048 | D-A = -0.004 | |
| 5. | At Corner 4 (E) | 19.049 | E-A = -0.003 | |

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

10/12/22

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AN

Name : Bhise Seema kailas

| | | |
|---|---|---|
| | **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:** 50 gm

| Sr. No. | Observed Weight in g | Sr. No. | Observed Weight in g | Limit |
|---|---|---|---|---|
| 1 | 50.695 | 6 | 50.697 | ± 0.1 % |
| 2 | 50.696 | 7 | 50.696 | |
| 3 | 50.696 | 8 | 50.697 | |
| 4 | 50.697 | 9 | 50.697 | |
| 5 | 50.696 | 10 | 50.695 | |

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

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

S K Bhise
10/12/22
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Name :- Bhise Seema kailas

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

**Page No** : 1 of 1

## DAILY CALIBATION RECORD

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

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

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

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

- **Observation Table:**

| Sr. No. | Date | pH | | |
|---|---|---|---|---|
| | | 4.00 (± 0.05) | 7.00 (± 0.05) | 9.1 (±0.05) |
| 1. | 11/12/22 | 4.01 | 6.87 | 9.25 |

slope = 96%

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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. :- | DS8000 | 1 of 1 |
| Name Of Students | Bhise seema kailas | |

**NAME OF TEST :-**

**TRIAL FOR DISSOLUTION TEST.**

**DISSOLUTION CONDITIONS:-**

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

**PREPARATIONS:-**

Pour 900mL of dissolution medium in each vessel.
Allow sufficient time for dissolution medium to a equlibrate at 37°C ± 0.5°C.
Adjust stirring element speed to 100rpm. Place one capule in each six paddle & adjust the paddle in the dissolutionmedium so that there is distance of 25mm ± 2mm between the bottom of the paddle & inside bottom of vessel. Start the apparatus.
At end specified time interval, withdraw 10mL aliquot from a zone midway between the surface of dissolution medium & top of rotating paddle.

Bhise
18/12/22

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| | ARNI ANALYTICALS | |
|---|---|---|
| TITLE | UV-SPECTROPHOTOMETER WORKSHEET | |
| Instrument Name :- | UV - Spectrophotometer | Page No. |
| Instrument ID : - | ARNI / INS - 002 | |
| Instrument Model No. :- | LMSP - UV100B | 1 of 1 |
| Name Of Students | Bhise seema kailas | |

Date:-

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

**NAME OF TEST :-** 1) Photometric Analysis
2) Wavelength scan

**PREPARATIONS:-**

**STANDARD PREPARATION :-**

Weigh accurately 10mg of caffeine standored in a 100ml Volumetric flask. add 60 ml of water sonicate for 5 minutes to Completely dissolve. make up the Volume with water. further dilute 5ml of sol^n to 50ml with water

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

1) Weight caffiene - 10mg.
2) wavelength - 273 nm.

**OBSERVATIONS:-** Caffeiene wavelength - 273nm.

**MAXIMUM ABSORPTION WAVELENGTH**

Maximum - 273 nm
minimum - 246 nm

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**CHECKED BY:-**