**CONTENTS**

0.0 version control history 3

1.0 what 4

2.0 why 4

3.0 when 4

4.0 RESPONSIBILITY and accountability 4

5.0 Dissolution Tester consist of 5

5.1 GENERAL INSTRUCTION 5

5.2 PRECAUTIONS 5

5.3 CLEANING 6

5.4 OPERATION 6

5.5 PROTOCOL SETTING 7

5.6 RPM SETTING 7

5.7 TEMP SETTING 7

5.8 TIME SETTING 9

5.9 GRADIENT TEMPERATURE 10

5.10 SYSTEM SETTINGS 10

5.11 POWER FAILURE DURING OPERATION 15

5.12 BATH SET UP 16

6.0 CALIBRATION PROCEDURE 22

6.1 pHYSICAL PARAMETERS CALIBRATION 23

6.2 CHEMICAL CALIBRATION CHECKS 25

7.0 GENERATION OF INSTRUMENT CALIBRATION NUMBER 27

8.0 GUIDELINES AND PRECAUTIONS 27

9.0 ABBREVIATIONS 28

APPENDIX I LOGBOOK OF DISSOLUTION TEST APPARATUS (OFFLINE SAMPLING) 29

APPENDIX II CALIBRATION DATA SHEET 30

APPENDIX III SUMMARY SHEET 42

# version control history

|  |  |  |  |
| --- | --- | --- | --- |
| Date | *Issue No.* | *CCR* | *Change description / reason / supersedes* |
| 01-Feb -2010 | 01 | Nil | New document. |

# what

This document details the procedure for the operation and calibration of Dissolution Test Apparatus (offline sampling).

# why

It is the policy of XYZ Limited that the written procedure shall be followed for the operation and calibration of Dissolution Test Apparatus in order to ensure smooth operation and its use monitored to obtain consistent and reproducible results and to minimise down time, in compliance with regulatory requirements.

# when

This procedure is to be applied at the time of operation and calibration of Dissolution test apparatus (offline sampling).

# 4.0 RESPONSIBILITY and accountability

Persons along with their responsibilities and accountability are given below:

|  |  |  |
| --- | --- | --- |
| S. No | Designation | Responsibility |
| 01 | Executive - Corporate  Quality Assurance | To Prepare the SOP. |
| 02 | Trainee Analyst/ Jr. Analyst/ Analyst/ Sr. Analyst | To follow the SOP accordingly |
| 03 | Manager - Quality  Control | To ensure implementation of SOP |
| 04 | Manager - Quality  Assurance | To ensure implementation of SOP |
| 05 | Manager - Corporate  Quality Assurance | To ensure implementation of SOP |

# 5.0 DISSOLUTION TESTER CONSISTS OF:

# 5.0.1 Basic instrument

# 5.0.2 Water bath with temperature controller

# 5.0.3 Set of glass / merlon jars (8 nos.)

# 5.0.4 Set of basket assembly (6 nos.)

# 5.0.5 Set of paddle assembly (6 nos.)

**5.1** **GENERAL INSTRUCTIONS**

Check and ensure that:

5.1.1 The instrument is clean and free from dust. If not, then clean the instrument using lint free duster.

5.1.2 The instrument is connected to 230 volts stabilised current (UPS).

5.1.3 The purified water in the bath is clear and up to the mark.

5.1.4 The level of the instrument using level meter.

**5.2 PRECAUTIONS:**

5.2.1. Replace the water in water-bath once in 8 days or whenever it is found to be turbid and clean the water-bath before adding the fresh water.

5.2.2 Preferably add Cetylpyridinium Chloride in bath of water to get 0.01% concentration after adding fresh purified water or add any other suitable anti microbial agent.

5.2.3 Disconnect the power supply while cleaning the water bath.

5.2.4 Do not pull or force the paddle or basket.

5.2.5 Do not stop the stirrer while in operation.

* + 1. Do not push the shafts forcefully.

5.2.7 Check the integrity of basket mesh prior to use.

5.2.8 Baskets & paddles must be certified for any damage prior to use. Deformed baskets or dented paddle must be taken out from use & discarded.

5.2.9 Connect the mains with proper earth point.

5.2.10 Oil the lifting arrangement regularly for smooth operation.

5.2.11 Check the lids of the jars are not kept on the jars while lowering down the paddle or basket assembly.

5.2.12 After completion of the work removes and cleans the jars, paddles, baskets and keeps it at its designated place.

5.2.13 Always lower the shaft assembly to lowest position, at the end of the day and when not in use.

5.2.14 Switch ‘OFF’ the instrument after use.

## 5.3 CLEANING

### 5.3.1 Disconnect the Instrument from main power supply.

### 5.3.2 Clean the instrument externally with clean dry cloth daily.

### 5.3.3 Clean the dissolution vessels after every dissolution test.

### 5.3.4 Remove the water from tank weekly and clean with wet cloth soaked in hot water. After cleaning of tank fill the tank with water. Also clean externally with wet cloth.

## 

## 5.4 OPERATION

### Connect the whole system with main power supply & switch on.

### Switch on the power switch provided on rear side of panel board.

### Switch on the power switch provided on front panel of fraction collector (FC-12).

### Switch on the power switch provided on the front panel of the temperature controller.

### Now, Switch on the Power switch provided on the rear side of TDT – 08L. The instrument shall initialise by displaying a power flash screen, which shall flash once as for a moment:

ELECTROLAB

TDT – 08L

DISSOLUTION

TEST APPARATUS

### After the power flash screen, an Idle screen shall be displayed showing the last protocol no. selected, RPM, Bath temperature , and Temp by external probe

PROTOCOL = 01 ⏶⏷

RPM = 000

BATH = 25.6° EXT = 24.2° C

PREPARE JAR TEMP.

### Load the cassetes into the pump head , ensuring that they are engaged on to the cassette guides at each end.

### Squeeze the knob until a click sound is heard repeat the same for remaining cassettes.

### Place the test vessels filled with the media, in such a way that the jar clamp are settle in the groove of the jar provided at jar flange, and rotate the jar clockwise or anticlockwise.

## PROTOCOL SETTING

### Now prepare the protocol by using four keys i.e RPM, TEMP, TIME & OPTN. Total 20 protocols can be prepared in software and all protocols can be prepared having different parameters such as RPM, TEMP, RUN TIME & other OPTN.

### Select the desired protocol 01, 02, 03, …………, 20.

## RPM SETTING

### Set the RPM (25 to 200) by pressing RPM key provided on front panel, RPM screen shall be displayed.

### Set the required RPM using UP / DOWN / DIGIT scroll key.

### Press F1 to start the spindle, the screen shall display as below:

SET RPM = 050 ⏶ ⏷⏴↵

STIRRER

< ON > OFF

F1

F2

### Press F2 to stop the spindle, the screen shall display as below.

SET RPM = 050 ⏶⏷⏴↵

STIRRER

< ON > OFF

F1

F2

### Press Enter key to register the value and come out of the RPM mode.

## TEMP SETTING

### Temperature shall be set up from 30° C to 40° C.

### Press TEMP key on front panel, the screen shall display as follows.

SET TEMP = 37.5° C ⏶⏷⏴↵

HEATER

ON < OFF >

F1

F2

### Set the required temperature by using UP / DOWN / DIGIT scroll keys.

### Press F1 key to ON the heater and F2 key to OFF the heater as per requirement.

### Press Enter key to register the value and come out from the TEMP mode. In OFF condition the screen shall display as above and in ON position, the screen shall display as below:

SET TEMP = 37.5° C ⏶⏷⏴↵

HEATER

< ON > OFF

F1

F2

### In the ON condition of Temperature controller, the Temp. ON LED shall glow on the front panel and ETC-11L shall also glow.

### If heater is ON, then the circulating pump shall start by default.

### If heater is ON, then BATH & EXT probe on the idle screen shall show the rise in temperature simultaneously till attain the set temperature.

PROTOCOL = 01 ⏶⏷

RPM = 000

BATH = 37.5° EXT = 37.3° C

PREPARE JAR TEMP.

F1

F2

### If Heater is in OFF condition, on the screen, BATH & EXT probes shall display the room temp. or surrounding temp.

PROTOCOL = 01 ⏶⏷

RPM: 000

BATH = 27.5° EXT = 26.9° C

PREPARE JAR TEMP.

F1

F2

## TIME SETTING

### 12 Sampling Steps in each protocol can be stored and sampling time interval can be set 23 Hr, 59 Mn.

### Press TIME key from the front panel, a screen shall display as follows:

SAMP. No. = 01 OF 04 ⏶⏷⏴↵

SAMP. INTV. = 01Hr : 00 Mn.

### Press UP / DOWN / DIGIT scroll key and set the total STEPS (SAMP. NO. = 01 OF 01, 01 OF 02, 01 OF 12) to be programmed. By default the no. of steps displayed shall be 01 OF 12.

### Using UP / DOWN / DIGIT scroll key select the STEPS nos. to be programmed by default the step no displayed shall be 01

SAMP No = 02 OF 04 ⏶⏷⏴↵

SAMP. INTV. = 04Hr: 00 min

### Set the desired time intervals for sampling using UP / DOWN / DIGIT scroll key.

SAMP No = 03 OF 04 ⏶⏷⏴↵

SAMP. INTV. = 08Hr: 00 min

### Repeat above four steps for the entire total no. of STEPS.

### Press Enter key to register the value for the selected protocol and to come out of the Time mode.

PROTOCOL = 01 ⏶⏷

RPM = 000

BATH = 37.5 EXT = 37.3

PREPARE JAR TEMP.

F2

F1

Warning– sample interval timing should not be 00 min otherwise that particular step shall be skipped.

### Sampling interval should be more than 5.0 min., as time taken by auto-sampler for withdrawing and replenishing sample is approximately 2.0 min.

## 5.9 GRADIENT TEMPERATURE

### 5.9.1 Gradient temperature is the difference in temperatures of BATH and JAR. Pressing TEMP and DIGIT scroll key simultaneously from the front panel. Gradient Temperature screen shall be displayed as below. Here gradient temperature can set up.

Temperature Gradient

Jar to Bath = 0.50

### 5.9.2 By using UP / DOWN / DIGIT scroll key set the required Gradient TEMP.

### 5.9.3 Press Enter key to register the value and come out of the Gradient TEMP Screen.

## 5.10 SYSTEM SETTINGS

### 5.10.1 Now press OPTN key to set the system. There are six options (A – G) to set the instrument as desired and the screen shall be displayed as:

→ A. Configure System ↵

B. Set Protocol

NEXT PREV

F1

F2

### Press Enter, the screen shall be displayed as:

→ A. Auto Sampler ↵

B. Manifold

NEXT PREV

F1

F2

### Press Enter, the screen shall be displayed as:

AUTO SAMPLING SYSTEM

CONNECTED

< YES > NO

F1

F2

### To connect the auto sampler press F1 and for NO Connection press F2. If yes press enter, the screen shall be displayed as:

SAMPLING MODE

ON LINE < OFF LINE>

F1

F2

### PRESS enter to register the values. The screen shall be displayed

→A. Auto Sampler ↵

B. Manifold

NEXT ESC

F1

F2

### Press F1 to Manifold Selection. The screen shall be displayed

→ B. Manifold ↵

C. Temperature

NEXT PREV

F1

F2

### Press Enter, the screen shall be displayed as:

SAMPLING MANIFOLD

MOTORISED

< YES > NO

F1

F2

### Press F1 for connection of motorized Manifold, the screen shall display as above.

### For Manual or without manifold operation press F2, the screen shall be displayed as:

SAMPLING MANIFOLD

MOTORISED

YES < NO >

F1

F2

### 5.10.9 Press Enter, the screen shall display as:

→ B. Manifold ↵

C. Temperature

NEXT ESC

F1

F2

### 5.10.10 Now press F1 key to select the temperature, the screen shall display as:

→C. Temperature ↵

NEXT ESC

F1

F2

### 5.10.11 Press enter, the screen shall display as:

Jar. Probe = 06 ⏶⏷↵

After Test

Temperature Controller

< ON > OFF

F2

F1

### 5.10.12 By using UP/ DOWN arrow key, select desired number of probes i.e. 1, 6 & 8. In the instruments we shall select 6 probes.

### 5.10.13 If we want the heater in ON condition after the test is over, press F1 key.

Jar. Probe = 06 ⏶⏷↵

After Test

Temperature Controller

< ON > OFF

F1

F2

### 5.10.14 For heater to be OFF position after the test is over, press F2

Jar. Probe = 06 ⏶⏷↵

After Test

Temperature Controller

ON < OFF >

F1

F2

### Press Enter key, the Screen shall display as follows:

→C. Temperature ↵

NEXT ESC

F1

F2

### 5.10.16 Press F2 key, the screen shall be displayed as:

→A. Configure System ↵

B. Set Protocol

NEXT PREV

### 

F2

F1

### 5.10.17 For protocol setting Press F1 key, the screen shall display as:

→B. Set Protocol ↵

C. Clock

< NEXT > PREV

F2

F1

### 5.10.18 Press Enter key, the screen shall display as below:

→A. Sample Volume ↵

B. Pfail Duration

NEXT ESC

F1

F2

### 5.10.19 Press Enter key, the screen shall display as:

Sample Vol.: C1 = 00 ml

Replenishment

< YES > NO

F1

F2

### 5.10.20 Select desired sampling volume protocol which is prepared in calibration option (C0 to C9) by using Shift / UP / Down arrow keys if replenishment is required, press F1 key to YES otherwise F2 for NO. Press Enter.

## 5.11 POWER FAILURE DURING OPERATION

### 5.11.1 For setting power failure during operation, press F1 key, the screen shall display as:

→B. Pfail Duration ↵

C. Bath set up

NEXT ESC

F1

F2

### Press Enter, the screen shall display as:

Power fail Delay = 00 Min. ⏶⏷⏴↵

Allowed during test

<NEXT> ESC

F1

F2

### Set power fail duration time using UP / DOWN arrow key. Press Enter to register the value. The screen shall display as:

→B. Pfail Duration ↵

C. Batch setup

NEXT ESC

F1

F2

### If power is resumed back within set time the test shall start automatically and if power is resumed back after the set time the screen shall show as:

Cumulative power fail

Delay is more than set

Want to continue

< START > STOP

F1

F2

### Now we can START /STOP the test by pressing the corresponding F1/ F2 keys respectively.

## BATH SET UP

(This parameter shall set the sampling height and stirrer unit height automatically according to volume of media).

### Press F1 key, the screen shall show as:

→ C. Bath Setup ↵

NEXT ESC

F1

F2

### Press Enter, the screen shall show

MEDIA VOLUME: 900 ml

TEST METHOD: PADDLE 25

FILTER IMMERSED

< YES > NO

F1

F2

### Using Shift / UP / Down arrow key, select the media volume (media can be Selected 500, 750, 900, 1000 ml).

### After media select the method by UP / Down arrow keys, we can select either one these methods Paddle 45, Paddle 25, Basket, Paddle over disk, Trans dermal 1, Trans dermal 2.

### Press F1 for filter to be immersed and F2 for filter not immersed. If press F1 the screen shall show as:

MEDIA VOLUME: 900 ml

TEST METHOD: PADDLE 25

FILTER IMMERSED

< YES > NO

F1

F2

### Press Enter, the screen shall show

→ C. Bath Setup ↵

NEXT ESC

F1

F2

### Press ESC, the screen shall show

→ B. Set Protocol ↵

C. Clock

NEX T PREV

F1

F2

### Now press F1, the screen shall display as

→ C. Clock ↵

D. Manual operation

NEXT PREV

F1

F2

### Press Enter, the screen shall display as:

→ A. Time ↵

B. Date

C. Wake Up

NEX T ESC

F1

F2

### Now press Enter, the screen shall display as:

CLOCK = # # : # # : # # ⏷⏴⏶↵

ENTER TO REGISTER

F2

F1

### Using UP / Down / Digit Scroll key set the current time. Press enter to register. The screen shall displayed

→ A. Time ↵

B. Date

C. Wake Up

NEXT ESC

F1

F2

### Press F1 the screen shall display as:

A. Time ↵

→ B. Date

C. Wake Up

NEXT ESC

F1

F2

### Press Enter, the screen shall display as:

DATE = # #: # #: # # ⏷⏴⏶↵

ENTER TO REGISTER

F1

F2

### Using UP / DOWN / DIGIT scroll key set the current date and press Enter. The screen shall display as:

A. Time

→ B. Date ↵

C. Wake Up

NEXT ESC

F1

F2

### Press F1 Key, the screen shall display as:

A. Time

B. Date

→ C. Wake Up ↵

NEXT ESC

F1

F2

### In this parameter if we want to start the instrument before the shift start on next day, wake up time for next day can be set up as follows:

### Press Enter at above screen, the screen shall display as:

TIME = 08: 00: 15 ⏶⏷⏴↵

DATE = 28 JUNE. 2004

SLEEP

< ON > OFF

F1

F2

### Set the desired time and date by using UP / Down / Digit scroll key and press F1 key and press Enter. The display screen shall be off and the instrument shall start on next day at the set time. But at the time of settings press F2 on above screen and do not press F1 because the screen shall off.

### On pressing F2 key followed by Enter and ESC, the screen shall display as:

→ C. Clock ↵

D. Manual operation

NEXT PREV

F1

F2

### Now press F1 key the screen shall display as:

→ D. Manual operation ↵

E. Printer set up

NEXT PREV

F1

F2

### Now set manual operation. In manual operation we can move the sampling manifold UP / Down or ON / OFF the circulating pump manually.

### Press Enter on above displayed screen, the screen shall display as:

→ A. Circulating Pump ↵

B. Diving Manifold

NEXT ESC

F1

F2

### Press Enter, a circulating pump screen shall display as:

BATH WATER CIRCULATE

ON <OFF>

F1

F2

### Select pump ON /OFF using F1 / F2 keys respectively. Press Enter followed by F1 key, the screen shall display as:

A. Circulating Pump

→ B. Diving Manifold ↵

NEXT ESC

F1

F2

### Press enter the screen shall display as:

SAMPLER MANIFOLD

MOVE

DOWN UP

F1

F2

### Press F1 / F2 key to move manifold DOWN / UP respectively. If the manifold in down position, then press F2 to move manifold up and the screen shall be displayed as:

SAMPLER MANIFOLD

MOVE

DOWN STOP

F1

F2

### Now we can stop the manifold by pressing F2 key.

### Press Enter the screen shall display as:

A. Circulating Pump

→ B. Diving Manifold ↵

NEXT ESC

F1

F2

### Press ESC the screen shall display as:

→ D. Manual Operation ↵

E. Printer Set Up

NEXT PREV

F1

F2

### After that we set the printer setting. Press F1 key, the screen shall display as:

→ E. Printer Set Up ↵

F. Sampler cal.

NEXT PREV

F1

F2

### Press Enter, the screen shall display as:

Print Last Test ≡ Yes ⏶⏷⏴↵

PRINTER

< ON > OFF

F1

F2

### Using Up / Down arrow keys, select last test report to be print Yes or No. Select printer ON / Off using F1 / F2 keys and press enter followed by F1. The screen shall be displayed as:

→ F. Sampler Cal. ↵

G. Cleaning Cycle

NEXT PREV

F1

F2

### As the dissolution test apparatus is offline so we are not required Sampler Cal. and Cleaning Cycle then press Enter key to register the value and come out of the Bath set up mode.

### Now operate the system to start the test-

* + - 1. Lift the stirrer unit
      2. Insert the shaft rod as per the method selected
      3. Place the sample in the test vessel
      4. Press down arrow key from the front panel to lower the stirrer unit. The stirrer unit shall adjust automatically to required height depending on the test method selected.
      5. Press start key from the front panel to start the test. The screen shall show as:

PROTO: #### STEP: ####

ELAPSE TIME (T): ##: ##: ##

RPM: ###

BATH: ##. # EXT: ##. #

### Record the observation of instrument on XYZ/SOP-037/FR-01 Logbook of Dissolution Test Apparatus (Offline Sampling) (Appendix I).

### Report any discrepancy observed during Operation of instrument to Section In - charge or his authorized representative for corrective and preventive action.

### Section In - charge or his authorized representative shall report the same to Manager-Quality Assurance and notify the defect to Engineering Department service engineer to rectify the defect.

### Affix ‘Under Maintenance’ label on the instrument.

**6.0 CALIBRATION PROCEDURE:**

6.1 Carry out the calibration checks for the following physical parameters on quarterly basis**:**

6.1.1 Rotational speed

6.1.2 Water bath temperature

6.1.3 Timer

6.1.4 Shaft wobbles

6.1.5 Distance from paddle / basket to the bottom of jar.

6.1.6 Distance between the shaft axis & vertical axis of the vessel.

6.1.7 Head co-planarity.

6.1.8 Integrity checks & mesh size of the basket.

6.2 Carry out the following additional chemical calibration checks on half yearly basis:

6.2.1 Dissolution with disintegrating type tablets (Prednisone Tablets 10 mg)

6.3 Carry out the calibration checks for the following physical parameters on yearly basis by the instrumentation department / manufacturer / supplier:

6.3.1 Head plate co-planarity

6.3.2 Shaft perpendicularity

6.3.3 Shaft wobble

6.3.4 Vessel centering

6.3.5 Rotational speed

6.3.6 Water bath temperature

6.3.7 Distance from paddle / basket to the bottom of jar.

6.3.8 Belt tension

6.3.9 Digital display for temperature, rpm and time.

**6.1 PHYSICAL PARAMETERS CALIBRATION (QUARTERLY):**

6.1.1 Procedure for number of revolutions check:

Select any one paddle and check the number of revolutions using a tachometer. Repeat the same procedure for other paddles. The above results should fall within the limits specified below:

Acceptance criteria:

Number of revolutions (rpm) Limits (rpm)

50 48 - 52

100 96 - 104

125 120 - 130

6.1.2 Procedure for temperature check:

Set the temperature of the bath to 37.5°C and wait for the display of the bath to show 37.5°C. Record the temperature in all the six jars. Check the set temperature for intervals 10, 20, 30, 40, 50 and 60 minutes with calibrated thermometer.

Acceptance criteria:

The observed temperature in each position of jars is in the range of 36.5o C to 37.5o C

6.1.3 Calibration of timer:

Adjust the ‘SET TIME’ to 30 minutes. Press the start button and simultaneously start the stopwatch. When alarm starts, check the time on stopwatch. Follow the procedure for 60 minutes as above.

Acceptance criteria:

Set time Tolerance

30 minutes Between 29 minutes 24 seconds And 30 minutes 36 seconds.

60 minutes Between 58 minutes 48 seconds And 61 minutes 12 seconds.

6.1.4 Procedure for wobble check:

6.1.4.1 Check the calibration for wobble for paddles and baskets using wobble meter. Empty all the jars and keep them without the jar lid in respective position. Lower the stirrer unit down so as the blades of paddle/ baskets just enter the jars. Ensure that the paddle or basket rod is properly tightened with the spindle use the key provided to tighten it.

6.1.4.2 Fix the wobble meter in the stand and attach it into the jar in such a way that the censor of the dial gauge is just touching the body of the paddle without any obstruction.

6.1.4.3 Start the rotation of paddle / basket rod check the maximum deviation of the pointer of device on both sides of ‘0’ and add the reading carry out this procedure for 50 and 100 rpm and measure the wobble (in mm) from dial gauge.

Acceptance criteria:

The tolerance for wobble is not more than 0.5 mm for paddles and 1 mm for basket. Check the wobble of all the 6 paddles and baskets.

6.1.5 Measurement of distance from paddle bottom to jar:

6.1.5.1 Keep all the jars empty and remove all jar lids. Clamp all the jars using jar holder provided on jar plate, and lower the stirrer unit to the bottom most position till it stops automatically. Hold depth gauge apparatus vertical and touching parallel to the paddle shaft. Touch the reference bracket of the depth gauge apparatus at the bottom face of paddle. Slide the depth gauge apparatus scale down till the point of stylus touches the jar bottom. Lock the depth gauge apparatus at this position and note the reading Repeat the procedure for remaining five paddles.

Acceptance criteria:

Distance should be 25 mm ± 2 mm.

6.1.5.2 Follow the same procedure given in Measurement of distance from paddle bottom to jar (1.5) using basket instead of paddle.

Acceptance criteria:

Distance should be 25 mm ± 2 mm.

6.1.6 Distance between the shaft axis and vertical axis of the vessel:

Empty all the jars and keep them without the jar lid in respective position. Lower the stirrer unit down so as the blades of paddle just enter the jars. Ensure that the paddle or basket rod is properly tightened with the spindle, use the key provided to tighten it.

6.1.6.1 Fix wobble meter on the jar so that the pointer is just pressed against the paddle shaft.

6.1.6.2 Rotate the device with both hands around the shaft of the paddle in the jar. Ensure that the bottom plate is not lifted from the jar or is in contact with the jar properly.

6.1.6.3 Make a note of the deflection of the pointer on both sides of `0`. Add both the readings and divide the sum by two.

Acceptance Criteria:

Cantering should be ≤ 2 mm.

6.1.7 Head co-planarity

6.1.7.1 A spirit level should be used to ensure that both the head plate and the base plate are perfectly horizontal.

6.1.7.2 A spirit level should be kept on the head plate to check that the head plate is perfectly horizontal. If it is not horizontal, the bath should be removed and the base plate under the bath should be checked with the spirit level. A base plate should be brought to the planarity and then the bath should be kept in its place and the head plate should be checked for the planarity.

6.1.8 Integrity check of the basket:

6.1.8.1 Check the integrity of SS wire mesh by holding the basket under illuminated magnifier and visually check for any damages.

6.1.8.2 Repeat the procedure for remaining five baskets.

(Note: If SS wire mesh is found to be damaged, discard the basket).

Acceptance criteria: The mesh should be intact.

6.1.8.3 Mesh size of basket:

6.1.8.3.1 Use a magnifying lens to check the number of openings of mesh of the basket.

6.1.8.3.2 Keep the basket in vertical position. Place the lens on the portion of the mesh of which the mesh number is to be determined.

6.1.8.3.3 Keep the basket in horizontal position. Place the lens on the portion of the mesh of which the mesh number is to be determined.

6.1.8.3.4 Observe through the lens and count the number of opening.

6.1.8.3.5 Repeat the procedure for remaining five baskets.

Acceptance criteria:

The number of opening should be 40 per linear inch.

**6.2 CHEMICAL CALIBRATION CHECKS** **(CALIBRATION OF THE INSTRUMENT USING PREDNISONE TABLETS) (HALF - YEARLY)**

**6.2.1 DISSOLUTION - For Prednisone Tablets 10mg (Disintegrating Type)**

This USP Dissolution Calibrator is provided for the Apparatus Suitability Test in the General chapter. Do not expose to excessive humidity.

6.2.1.1 USP APPARATUS 1 (Basket)

Dissolution medium : Purified water

Duration : 30 minutes

Temperature : 37°C ± 0.5°C

Volume : 500 ml

Speed : 50 rpm

6.2.1.1.1 STANDARD PREPARATION:

20 mg of Prednisone USPRS standard is dissolved in 5 ml of alcohol and diluted to 200 ml with the purified water. Further 5 ml is diluted to 100 ml with purified water.

Note: An amount of alcohol not to exceed 5 % of the total volume of the standard solution to be used to bring the Prednisone standard into solution prior to dilution with Dissolution Medium.

6.2.1.1.2 SAMPLE PREPARATION:

One tablet is placed in each of the baskets. At the end of 30 minutes withdraw about 20 ml of the aliquot and filter through Whatman filter paper No. 41. Further 5 ml is diluted to 10 ml with purified water.

6.2.1.1.3Record the absorbance of the standard and sample solutions at 242 nm using purified water as the blank.

* + - * 1. Determine the quantity of Prednisone C21H26O5, dissolved in 30 minutes, for each spindle expressed as percent of labelled amount by following formula:

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
|  | AT |  | W |  | 5 |  | 500 |  | 10 |  | P |  |  |
| = | ------- | × | ------- | × | ------ | × | ------ | × | ----- | × | ---- | × | 100 |
|  | AS |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  |

% Released

Where,

AT = Absorbance of sample preparation

AS = Absorbance of the standard preparation

W = Weight of the standard preparation (in mg)

P = %Purity of Prednisone USPRS (on as is basis)

Acceptance criteria: % release between 47 to 82% of labelled amount of prednisone.

6.2.1.2 USP APPARATUS 2 (Basket)

6.2.1.2.1All the conditions are same as that in USP APPARATUS 1 above except that paddles are used instead of baskets with 50 rpm. Repeat the procedure as in USP APPARATUS 1 and note down the absorbance at 242 nm.

6.2.1.2.2Determine the quantity of Prednisone C21H26O5, dissolved in 30 minutes, for each spindle expressed as percent of labelled amount by following formula:

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
|  | AT |  | W |  | 5 |  | 500 |  | 10 |  | P |  |  |
| = | ----- | × | ----- | × | ------ | × | ------ | × | ----- | × | ------ | × | 100 |
|  | AS |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  |

% Released

Where,

AT = Absorbance of sample preparation

AS = Absorbance of the standard preparation

W = Weight of the standard preparation (in mg)

P = %Purity of Prednisone USPRS (on as is basis)

Acceptance criteria: % release between 30 to 57% of labelled amount of prednisone

* + 1. Record all the observations of Calibration as per XYZ/SOP-037/FR-02, Calibration Data Sheet (Appendix II)
    2. Compiled all the Calibration data as per XYZ/SOP-037/FR-03, Summary Sheet (Appendix III).

**7.0 GENERATION OF INSTRUMENT CALIBRATION NUMBER**

Generate Instrument Calibration number on calibration data sheet as INSCALXXYYZZZ

Where, INS denotes Instrument, CAL denotes Calibration, XX denotes year, YY denotes Month ZZZ denotes sequence number.

**8.0** **Guidelines and Precautions**

Guidelines for the usage of the dissolution test apparatus and precautions to be taken for using Dissolution accessories are given below:

8.1 Ensure that the apparatus and accessories are cleaned before starting the analysis.

8.2 Clean the dissolution vessels and the dissolution accessories like Paddles / Basket shafts, baskets, sinkers using the purified water and the soap solution and tap water followed by purified water. To remove the traces of the tablets or the capsules from the basket, paddles and blades use the soft brush.

8.3 Ensure that the required temperature of dissolution medium in jar is achieved before you start the analysis by using calibrated thermometer.

8.4 Degas the dissolution medium before it is charged in the apparatus by filtering the medium through the 0.45 µm membrane filter under vacuum and stirring. Do not allow dissolution medium to cool down to the 37°C before starting of the dissolution test.

8.5 Dissolution medium should pour in the vessels from sidewalls of vessels to avoid aeration.

8.6 Clean the auto pipette after every analysis or clean the auto sampler line / tubing before and after every analysis.

8.7 During filtration discard the first few ml of the filtrate before it is taken for the further analysis.

8.8 Use fresh specified filters for the every sample filtration.

8.9 Use only specified filters for the filtration of the dissolution medium.

8.10 In case of dissolution profiling studies sample pooling and replenishing of the dissolution medium should be done within the 1 minute of specified time point.

8.11 Keep the cleaned accessories in the self sealing polythene bags.

8.12 Do not use the cracked baskets / damaged shafts for the analysis. Destroy them immediately as soon as noticed and report the same to the supervisors for replacements.

8.13 Change the purified water from the water bath once in a week.

## 9.0 ABBREVIATIONS

INS CAL : Instrument Calibration

RPM : Rotation per minute

USPRS : United State Pharmacopoeia Reference Standard

A. R. No. : Analytical report number

**APPENDIX I** **XYZ/SOP-037/FR-01**

LOG BOOK OF DISSOLUTION TEST APPARATUS (offline sampling)

**Instrument Id. No.: …………………………………….**

**Location: ……………………………………..**

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Sr. No.** | **Date** | **Start Time** | **Name of Sample** | **Batch No/ A. R. No.** | **End Time** | **Done by** | **Checked By** | **Remarks** |
|  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |

**APPENDIX II XYZ/SOP-037/FR-02**

**CALIBRATION DATA SHEET INSCAL \_\_\_\_\_\_\_\_\_\_\_\_\_\_**

**1.0 Instrument Id.: ……………………………………..**

**2.0 Location: ……………………………………..**

**3.0 Date of Calibration: ………………… (Quarterly/ Half Yearly)**

**4.0 Next due date of Calibration: ……………………………………..**

**5.0 PHYSICAL PARAMETERS CALIBRATION (QUARTERLY)**

5.1 Rotational Speed

Tachometer Id. ……………………………………..

Validity of Calibration: ……………………………………..

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sr. No. | RPM | Number of Revolutions | | | | | | Limit |
| Paddle I | Paddle II | Paddle III | Paddle IV | Paddle V | Paddle VI |
| 1. | 50 |  |  |  |  |  |  | 48-52 |
| 2. | 100 |  |  |  |  |  |  | 96-104 |
| 3. | 125 |  |  |  |  |  |  | 120-130 |

Remarks: Complies/Does not Complies

Done By: Checked By: Approved By:

Date: Date: Date:

5.2 Water bath Temperature

Thermometer Id. ………………………………

Validity of Calibration: ………………………………

Set Temperature: ………………………………

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sr.No. | Time Interval  (min) | Observed Temperature | | | | | | Limit  (˚C) |
| Jar I | Jar II | Jar III | Jar IV | Jar V | Jar VI |
| 1. | 10 |  |  |  |  |  |  | 36.5 -37.5 |
| 2. | 20 |  |  |  |  |  |  |
| 3. | 30 |  |  |  |  |  |  |
| 4. | 40 |  |  |  |  |  |  |
| 5. | 50 |  |  |  |  |  |  |
| 6. | 60 |  |  |  |  |  |  |

Remarks: Complies/Does not Complies.

Done By: Checked By: Approved By:

Date: Date: Date:

5.3. Timer

Stopwatch Id. ……………………………….

Validity of Calibration: ……………………………….

|  |  |  |  |
| --- | --- | --- | --- |
| Sr. No. | Set Time (min) | Observed Time (min) | Limit |
| 1. | 30 |  | Between 29 min 24 sec. and 30 min 36 sec. |
| 2. | 60 |  | Between 58 min 48 sec. and 61 min 12 sec. |

Remarks: Complies/Does not Complies.

Done By: Checked By: Approved By:

Date: Date: Date:

5.4 Shaft Wobble

Wobble meter Id. ……………………………..

Validity of Calibration: ……………………………..

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Sr. No. | Observed Tolerance (50 rpm) | | Observed Tolerance (100 rpm) | | Limit |
| Paddle | Basket | Paddle | Basket |
| 1. |  |  |  |  | For Paddle : NMT 0.5 mm |
| 2. |  |  |  |  |  |
| 3. |  |  |  |  | For Basket : NMT 1.0 mm |
| 4. |  |  |  |  |  |
| 5. |  |  |  |  |  |
| 6. |  |  |  |  |  |

Remarks: Complies / Does not Complies

Done By: Checked By: Approved By:

Date: Date: Date:

5.5 Distance from paddle/basket to the bottom of the jar

Depth gauge Apparatus Id.: …………………………………

Validity of Calibration: ………………………………….

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Sr. No. | No. of Paddles | No. of Basket | Distance Observed (mm) | | Limit (mm) |
| Paddle | Basket |
| 1. | I | I |  |  | 25 + 2 |
| 2. | II | II |  |  |
| 3. | III | III |  |  |
| 4. | IV | IV |  |  |
| 5. | V | V |  |  |
| 6. | VI | VI |  |  |

Remarks: Complies/ Does not Complies

Done By: Checked By: Approved By:

Date: Date: Date:

5.6 Distance between the shaft axis and vertical axis of the vessel

Wobble meter Id. ………………………………….

Validity of Calibration: ………………………………….

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Sr. No. | No. of Paddles | No. of Basket | Distance Observed (mm) | | Limit (mm) |
| Paddle | Basket |
| 1. | I | I |  |  | For Paddle = NMT 2.0 mm |
| 2. | II | II |  |  |
| 3. | III | III |  |  |
| 4. | IV | IV |  |  | For Basket = NMT 2.0 mm |
| 5. | V | V |  |  |
| 6. | VI | VI |  |  |

Remarks: Complies/Does not Complies

Done By: Checked By: Approved By:

Date: Date: Date:

5.7. Head Co-Planarity

Spirit level Id. ………………….……………….

Validity of Calibration: ……………………………………

Acceptance Criteria: The head plate should be planar.

Remarks: Complies / Does not Complies

Done By: Checked By: Approved By:

Date: Date: Date:

5.8 Integrity check / Mesh size of basket

Illuminated Magnifier Id: ……………………………………

Validity of Calibration: ……………………………………

5.8.1 Integrity Check

Acceptance Criteria: The mesh should be intact.

5.8.2 Mesh Size

Magnifying lens Id: ……………………………………

Validity of Calibration: ……………………………………

|  |  |  |  |
| --- | --- | --- | --- |
| Sr.No. | No. of Basket | Observed No. of Opening | Limit |
| 1. | I |  | 40 per linear inch. |
| 2. | II |  |
| 3. | III |  |
| 4. | IV |  |
| 5. | V |  |
| 6. | VI |  |

Remarks: Complies/ Does not Complies

Done By: Checked By: Approved By:

Date: Date: Date:

**6.0. CHEMICAL PARAMETERS CALIBRATION (HALF YEARLY)**

Balance Id.: ………………………………….

Validity of Calibration: ………………………………….

* + 1. Prednisone Tablets 10 mg (Disintegrating type)

USP Apparatus I (Basket)

Dissolution medium: Distilled Water

Duration: 30 minutes

Temperature: 37˚ C + 5 ˚ C

Volume: 500 ml

Speed: 50 rpm

Lot No: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Standard Preparation:

………………..mg of Prednisone USPRS …………….. ml with dissolution media.

Dilute …………….. ml ………….. ml with dissolution medium.

Sample Preparation:

……………. mg (1 tablet) …………….. ml with dissolution medium.

Record the maximum absorbance of standard and sample solutions at 242 nm using dissolution medium as blank.

Observation Table:

Standard absorbance: ……………..

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sr. No. | | Test Abs. | | Calculations | | Result  (%release) | | Limit (%) | |
| 1. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | | 47 to 82 | |
| 2. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | |
| 3. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | |
| 4. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | |
| 5. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | |  | |
| 6. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | |  | |

Remarks: Complies/ Does not Complies.

Done By: Checked By: Approved By:

Date: Date: Date:

6.2 USP Apparatus II (Paddle)

Dissolution medium: Distilled Water

Duration: 30 minutes

Temperature: 37˚ C + 5 ˚ C

Volume: 500 ml

Speed: 50 rpm

Lot No: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Standard Preparation:

………………..mg of Prednisone USPRS …………….. ml with dissolution media.

Dilute …………….. ml ………….. ml with dissolution medium.

Sample Preparation:

……………. mg (1 tablet) …………….. ml with dissolution medium.

Record the maximum absorbance of standard and sample solutions at 242 nm using dissolution medium as blank.

Observation Table:

Standard absorbance: ……………..

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sr. No. | | Test Abs. | | Calculations | | Result  (%release) | | Limit (%) | |
| 1. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | | 30 -57 | |
| 2. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | |
| 3. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | |
| 4. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | |
| 5. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | |  | |
| 6. | |  | | |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | |  |  |  |  | 5 |  | 500 |  | 10 |  |  |  |  | | ---- | × | ----- | × | --- | × | ----- | × | ---- | × | ----- | × | 100 | |  |  | 200 |  | 100 |  | 10 |  | 5 |  | 100 |  |  | | |  | |  | |

Remarks: Complies/ Does not Complies.

Done By: Checked By: Approved By:

Date: Date: Date:

**APPENDIX III XYZ/SOP-037/FR-03**

**SUMMARY SHEET INSCAL\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_**

**Location: ……………………………………..**

**Instrument Id.: ……………………………………..**

**Physical Parameters**

|  |  |  |  |
| --- | --- | --- | --- |
| **Test Name** | **Observations** | **Acceptance Criteria** | **Status** |
| Rotational Speed | 50 rpm = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. | 48 - 52 | Meet / Does not meet |
|  | 100 rpm = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. | 96 - 104 |  |
|  | 125 rpm = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. | 120 - 130 |  |
| Water bath temperature | 10 min = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. | 36.5 ˚ C to 37.5 ˚ C | Meet / Does not meet |
|  | 20 min = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. |  |  |
|  | 30 min = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. |  |  |
|  | 40 min = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. |  |  |
|  | 50 min = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. |  |  |
|  | 60 min = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. |  |  |
| Timer | 30 min = \_\_\_\_\_\_\_\_ | 29 min 24 sec to 30 min 36 sec | Meet / Does not meet |
|  | 60 min = \_\_\_\_\_\_\_\_ | 58 min 48 sec to 61 min 12 sec |  |

|  |  |  |  |
| --- | --- | --- | --- |
| **Test Name** | **Observations** | **Acceptance Criteria** | **Status** |
| Shaft Wobbles | For paddle (50 rpm) = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. | For paddle = NMT 0.5 mm | Meet / Does not meet |
|  | 100 rpm = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. |  |  |
|  | For basket (50 rpm) = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. | For basket = NMT 1mm |  |
|  | 100 rpm = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. |  |  |
| Distance from paddle / basket to the bottom of jar. | Paddle = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. | 25 + 2 mm | Meet / Does not meet |
|  | Basket = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. |  |  |
| Distance between shaft axis and vertical axis | Paddle = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. | Cantering should be < 2 mm | Meet / Does not meet |
|  | Basket = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. |  |  |
| Head Co-planarity |  | The head plate should be planar. | Meet / Does not meet |
| a) Integrity check |  | The mesh should be intact | Meet /Does not meet |
| b) Mesh Size | Basket = \_\_\_\_, \_\_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_, \_\_\_\_. | 40 per linear inch | Meet /Does not meet |

**Chemical Parameters**

|  |  |  |  |
| --- | --- | --- | --- |
| **Test Name** | **Observations** | **Acceptance Criteria** | **Status** |
| Prednisone tablet 10 mg |  |  |  |
| USP Apparatus I (Basket) | % release range = \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ | 47 to 82% | Meet /Does not meet |
| USP Apparatus II (Paddle) | % release range = \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ | 30 to 57% | Meet /Does not meet |

Remarks: All the parameters for the calibration as per predefined acceptance criteria are under limit hence the instrument is suitable for routine analysis

Done By: Checked By: Approved By:

Date: Date: Date: