PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

STANDARD OPERATING PROCEDURE	
Department: Quality Control	**SOP No.:**
Title: Operation & Calibration of Dissolution Test Apparatus	**Effective Date:**
Supersedes: Nil	**Review Date:**
Issue Date:	**Page No.:**

1.0 OBJECTIVE:

To lay down a procedure for operation & calibration of Dissolution Test Apparatus.

2.0 SCOPE:

This SOP is applicable for operation & calibration of Dissolution Test apparatus; Make: Electro lab; Model: EDT-08 Lx.

The scope of this SOP covers the operation and calibration of dissolution test apparatus as per the current Pharmacopoeias (IP/BP/USP) & In-House requirements.

3.0 RESPONSIBILITY:

Executive/ Officer/Section Head – Quality Control

Head of Department – Quality Control

4.0 PROCEDURE:

4.1 Operation Procedure

4.1.1 Preliminary check:

4.1.1.1 Ensure that the instrument is clean and free from dust, if not then clean with dry cloth.

4.1.1.2 Ensure the water level in the water bath (Filling tank).

4.1.1.3 Ensure that Instrument is Calibrated.

4.1.1.4 Clean the Paddle shaft, Basket shaft and Jars with purified water before start of the operation.

4.1.1.5 Before start the operation ensure that temperature of all the vessels achieved as per requirements.

4.1.2 Operation:

4.1.2.1 Switch on the power switch on the rear side of the panel board and heater switch at the rear hand side of panel board.

4.1.2.2 After switch On, The instrument will initialized itself the logo screen will flash for 3 seconds and then idle screen will be displayed.

ELECTROLAB DISSOLUTION TESTER

MODEL : EDT-08LX VER-4.57.E

4.1.2.3 The Idle screen displays the current status of the instrument.

Date:##-###-#### ##:##:##
RPM:###.# BATH:##.# ºC EXT:##.# ºC
PROTOCOL= ##
MENU PREPARE

4.1.2.4 It indicates the Date, Time, RPM, Bath Temperature, External probe and loaded protocol number.

4.1.2.5 Press F1 key from front panel to enter menu screen.

1.Configure	**4.Reports**	**7.Version**
2. Protocol	**5.Run**	
3. Settings	**6.Jar Temp**	
Press number for selection		**<BACK>**

4.1.2.6 **MENU<Configure)**

Press 1 from alphanumeric keypad to enter configuration menu.

1. Sampling	**4.Commu**	**7.Auto Disp.**
2. Indv.Prb	**5.Stirrer**	**8.Instru.No.**
3. Tem.Grad.	**6.Calibr.**	**9.Disb.LED.**
Press number for selection		**<BACK>**

4.1.2.7 **MENU<Configure)|Sampling|**

Press 1 from alphanumeric keypad to enter sampling menu.

1.Auto	
2.Manual	
Press number for selection	**<BACK>**

4.1.2.7 **MENU<Configure)|Sampling|[Manual]**

Press 1 to select manual sampling through syringe manifold or 2 to select manual sampling through pipette.

1.Syringe Manifold

2.Pipette

Press number for selection **<BACK>**

4.1.2.9 **MENU<Configure>|Individual Probe|**

Press 2 to enter Individual Probe Screen.

Press 1from alphanumeric keypad to select the individual probes.

1.Yes **No. of Probes = 08**

2.No

Press number for selection **<BACK>**

4.1.2.10 **MENU<Configure>|Temp Gradient|**

Press 3 to enter Temp Gradient.

Temperature Controller set.

Key: To “On” the Temp Controller.

F2 Key :To “OFF “the Temp Controller

F3 Key: To “SAVE” & to return back to the Configure screen.

Gradient Temp:#.#ºCΔv

TEMP control ON after test over ?

ON **OFF** **SAVE**

4.1.2.16 **MENU<Configure>|Communication|**

Press 4 from front panel to enter Communication.

Select RS232 for Printer /PC and ETHERNET for LAN Connection using 1 or 2 numeric Keys.

Press F3 key to go back to configure screen

1.<RS232>

2. ETHERNET

Press number for selection

<BACK>

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4.1.2.17 **MENU<Protocol>**

Press 2 to enter protocol menu.

Press 1 to enter Load Protocol.20 programmable protocol can be loaded. Protocol No. can be loaded using Up/ Down key & No .Enter key needs to be pressed.

Press F1 to load next protocol

Press F2 to load previous Protocols

Press F3 to back to the Protocol screen

1.Load Protocol
2.Edit Protocol
3.View Protocol
Press number for selection **<BACK>**

Press 2 to enter edit protocol. Select the protocol which needs to be edited using Up/ Down arrow and press “Enter” key.

The respective protocol details such as Drug Name, Media Name, pH Value, Temperature, Media Vol, Power fail, Apparatus, Time Table, Sampling info details need to be entered.

The protocol details can be edited using alphanumeric keypad. The next parameter can be selected for editing before saving the previous parameter by pressing F3 key from front panel.

To select different apparatus press enter key and select the desired apparatus using numeric keys.

4.1.2.18 **To set RPM**

Press RPM key from front panel, press numeric key to register the rpm range from 20 to 300 RPM.

Press F1 key to Turn –ON the motor & F2 key to Turn off.

If RPM is out of range on error screen will be displayed.

SET RPM :### (20-300 RPM)
Motor is stopped
Turn on **Turn off**

4.1.2.19 To set Temperature

Press Temp key from front panel press numeric key to register the temperature range from 20 to 40 °C

Press F1 or F3 to Turn On/ off heater.

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SET TEMP: ##.# °C (20.0-40.0°C)

Heater is on
Turn On **Pump On** **Turn off**

4.1.2.20 TO PREPARE THE TEST

Load the appropriate no. & Press F2 key to start preparing the test.

Batch Number & A.R Number will be displayed.

Using down arrow and enter key, the Batch No & A.R.no. up to ten digit can added. Press Enter key to register the Numbers.
Ready Indication will glow on front panel when jar temperature reach to set point & Start indication will be displayed.
Press START key from front panel to start the test.

4.2 Calibration:

Before calibration, ensure the level of instrument with the help of Digital protector and recorded in format as per Annexure – I OK/Not OK.

4.2.1 Physical calibration:

4.2.1.1 For Vessels Shaft Centering :

4.2.1.1.1 Each vessel must be free of scratches, cracks, pits and residue. It should be recorded.

4.2.1.1.2 Each vessels centering measuring by venire caliper of all four location (X1, X2, X3 and X4) equally space around the vessel and recorded all the results in format as per Annexure - I.

Calculate the centering in mm by using formula: $\Delta X = X_{Max} - X_{Min}$

4.2.1.1.3 Acceptance criteria: Less than 2.0 mm

4.2.1.2 For Temperature:

4.2.1.3.1 Fill the water as a medium in each jar and set the temperature of the instrument 37.7°C for marlon jar and 37.5°C for glass jar. After half an hour the jar temperature will be reached at 37°C.

4.2.1.3.2 After flash will glow on the LHS against ready, place a calibrated thermometer in each jars for five minutes and note down the reading in the calibration format as per Annexure - I.

The acceptance criteria is 37°C ± 0.5° C.

4.2.1.4 For RPM:

4.2.1.4.1 Lower the basket /paddle by pressing "Down".

4.2.1.4.2 Set 50 RPM on the instruments and press start RPM and rotation will start.

4.2.1.4.3 Measure the rotation by calibrated tachometer.

4.2.1.4.4 Again set the RPM at 100, 150 & then at 200 and measure the rotations by tachometer and note down the reading in the calibration format as per Annexure - I.

The acceptance criteria ± 4 % of set RPM

4.2.1.5 For Basket & Paddle:(Wobble & Depth)

4.2.1.5.1 **Physical observation:**

Each basket visually examined for defects such as rusting, corrosion, wires sticking out beyond the basket, clogged mesh holes or deformed mesh sides. It should be recorded.

4.2.1.5.2 Each paddle visually examined for defects such as rusting, corrosion or loose pieces of coating on the paddles (for paddles coated with Teflon or another coating). It should be recorded.

4.2.1.5.3 **Wobbling:**

Set the wobble meter in the basket or paddle individually and adjust the position of pointer to zero. Start the rotation of the instrument at 200 RPM and determine the distance left and right side of pointer from the set point.

4.2.1.5.4 Measure the reading individually for six paddles & basket and note down the reading in the calibration format as per Annexure - I.

The acceptance criteria for Basket not more than 1 mm and for paddle not more than 0.5 mm.

4.2.1.5.5 **Distance:**

Measure the distance between inside bottom of the vessel & basket or paddle for six vessels and note down the observation in calibration format as per Annexure - I.

The acceptance criteria are 25 ± 2 mm for basket & paddle.

4.2.2 Dissolution Performance Verification Test:

(By Prednisone Tablets 10mg)

4.2.2.1 **Dissolution parameters:**

Apparatus	: Type-1 (Basket) & Type-2 (Paddle)
Medium	: Purified water
Volume	: 500 ml

Medium Temperature : 37° ± 0.5°C

Time : 30 Minutes

RPM : 50

4.2.2.2 **Standard Preparation:**

Weigh accurately 25 mg of Prednisone reference/working standard in 50 ml volumetric flask, add 2.5 ml ethanol to dissolve and make the volume with purified water. Dilute 4 ml of above solution to 100 ml with purified water.

4.2.2.3 **Sample preparation:**

Ensure the lot No. of tablet is current. Place one tablet in each jar (vessel) having 500mL Deaerated Purified water, after instrument get ready and start the instrument.

4.2.2.4 After the set time is over a long beep will be heard and the instrument will stop automatically. At the end of the specified time, withdraw 25 ml of aliquot from a zone midway between the surface of the dissolution medium and the top of the basket or top of the paddle blade.

4.2.2.5 Filter the each sample immediately through a syringe filter (0.45 µm PVDF type or equivalent), discarding first 5 ml portion of the filtrate. Collect the subsequent filtrate and cool at room temperature.

4.2.2.6 Measure the absorbance of standard and sample simultaneously on UV-Visible spectrophotometer at 242 nm and calculate the % of Prednisone dissolved. The % RSD of the five replicate absorbance of the standard absorbance should not be more than 2.0. Transform the percent dissolved results, to the natural log scale, and determine the mean and variance.

4.2.2.7 Convert the results to a GM and % CV. Compare the results with COA of tablets current lot No.

Acceptance Criteria: As per current tablet lot No. COA

4.2.2.8 Results of 1st stage satisfy both acceptance criteria, stop the assembly. If the results of 1st stage not meet the acceptance criteria continued 2nd stage.

4.2.2.9 Repeat the step no. 4.2.2.1 to 4.2.2.6 for additional set of the tablets.

4.2.2.10 Average the two means and two variances obtained.

4.2.2.11 Convert the results to a GM and % CV. Record the results in the calibration format as per annexure – II. Compare the results with COA of tablets current lot No.

Acceptance Criteria: As per current tablet lot No. COA

4.3 **Frequency:**

i) Physical Calibration - Quarterly

ii) Dissolution Performance Verification Test – Half Yearly

4.4 Record the calibration report as per Annexure – I and Annexure – II.

4.5 If the instrument is out of calibration, Affix Out of calibration label and proceed as per SOP No. QAD/055.

4.6 Note the Calibration activity in the instrument logbook.

4.7 Cleaning:

4.7.1 Before cleaning switch off the instrument.

4.7.2 Wipe main body of the instrument with a clean dry cloth.

4.7.3 After completion of the operation, first clean the Paddles, Baskets with shaft, jars (vessels) with lid in running tap water using 0.1 % v/v solution of neutral detergent Extran (Merck). Finally rinse with purified water and dry.

4.7.4 If required basket shall be dipped in purified water and sonicate.

4.7.5 When the bath water is shown hazy, drain the water from bath (Tank) and shall be cleaned with purified water.

4.7.6 Fill the purified water up to the level and add 100 ml of 0.01% cetyl pyridinium chloride or sodium benzoate solution in a dissolution bath (Tank).

4.8 Precautions:

4.8.1 Do not put the heater ‘ON’ if there is no water in the tank up to the mark.

4.8.2 Check water level in the water bath (Filling tank).

5.0 ANNEXURE (S):

Annexure I : Physical Calibration Record of Dissolution Test Apparatus

Annexure II: Performance Verification Record of Dissolution Test Apparatus

6.0 REFERENCE (S):

SOP: Handling of out of calibration instrument.

SOP: Preparation, Approval, Distribution control, revision and destruction of Standard operating Procedure (SOP).

USP/BP/Ph. Eur./IP

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7.0 ABBREVIATION (S)/DEFINITION (S):

RPM : Rotation per minute

GM : Geometric mean

% CV : Percent coefficient of variation

IP : Indian Pharmacopoeia

USP : United States Pharmacopoeia

PVDF : Polyvinylidene difluoride

REVISION CARD

S. No.	REVISION No.	REVISION DATE	DETAILS OF REVISION	REASON (S) FOR REVISION	REFERENCE CHANGE CONTROL No.
1	00	---	---	New SOP	-

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ANNEXURE I

PHYSICAL CALIBRATION RECORD OF DISSOLUTION TEST APPARATUS Reference SOP No.			
Location		**Page No.**	10 of 4
Manufactured By	Electro lab	**Model No.**	
Frequency	Quarterly	**Identification No.**	
Date of Calibration		**Next Due Date of Calibration**	

1.0) Instrument level:

Digital Protector ID No.: Calibration Valid Up to:

OK/Not OK

2.0) For Vessels Shaft Centering:

Vernier Caliper ID No.: Calibration Valid Up to:

$\Delta X = X_{Max} - X_{Min}$

S.No.	Physical observation	Centering (< 2.0 mm)					Remarks
		X1	X2	X3	X4	ΔX	
1.							
2.							
3.							
4.							
5.							
6.							
7.							
8.							

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3.0) Temperature Calibration:

Thermometer ID No.: Calibration Valid Up to:

Set Temperature °C	
Bath Temperature (Temperature probe) °C	
Bath Temperature (External thermometer) °C	

Temperature °C (LIMIT: 37°C ± 0.5°C)							
1 JAR	**2 JAR**	**3 JAR**	**4 JAR**	**5 JAR**	**6 JAR**	**7 JAR**	**8 JAR**
Remarks:							

4.0) RPM Calibration:

Tachometer ID No. : Calibration Valid Up to:

TEST	SET RPM	OBSERVED	ACCEPTANCE CRITERIA ± 4 % OF SET RPM	REMARKS
RPM	50 RPM			Complies /does not Comply
	100 RPM			
	150 RPM			
	200 RPM			

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5.0) Basket: (Wobble &Depth)

Wobble meter ID No.: Calibration Valid Up to:

Depth gauge ID No.: Calibration Valid Up to:

Test position	Physical observation	Wobble (± 1 mm) Wobble	Distance Between Bottom Edge (25 ± 2 mm)	Remarks
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				

Result: Complies /does not comply

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6.0) Paddle: (Wobble &Depth)

Test position	Physical observation	Wobble (± 1 mm) Wobble	Distance Between Bottom Edge (25 ± 2 mm)	Remarks
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				

Result: Complies /does not comply

Remarks: The Instrument Calibration is **OK/ Not OK** as per **IP/BP/USP/In-House** requirements.

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ANNEXURE II

PERFORMANCE VERIFICATION RECORD OF DISSOLUTION TEST APPARATUS Reference SOP No.			
Location		**Page No.**	14 of 6
Manufactured By	Electro lab	**Model No.**	
Frequency		**Identification No.**	
Date of Calibration		**Next Due Date of Calibration**	

Lot No. of tablets :	RS/WS No.:
Balance ID: EQ/QCD/	UV Spectrophotometer ID: EQ/QCD/

Dissolution Conditions

Parameters		**Stage I**	**Stage II**
Medium (Deareted water)	:		
Volume (500 ml)	:		
Temperature (37 ± 0.5°C)	:		
Rotatory speed (50 RPM)	:		
Time (30 minutes)	:		

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Stage I:

S.No.	Basket ID No.	Vessel ID No.	Temperature (°C)	Tablet dropping time (min)	Sample Withdrawal time (min)
1					
2					
3					
4					
5					
6					

Stage II

S.No.	Basket ID No.	Vessel ID No.	Temperature (°C)	Tablet dropping time (min)	Sample Withdrawal time (min)
1					
2					
3					
4					
5					
6					

Standard preparation:

Weighed _____ mg (about 25 mg) of Prednisone reference/working standard in _____ ml (50 ml) volumetric flask, added _____ ml (2.5 ml) ethanol to dissolve and made the volume with purified water. Diluted _____ ml (4 ml) of above solution to _____ ml (100 ml) with purified water.

Sample preparation:

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1 Tablet → _____ ml (500 ml) of Deareted Purified water. At the end of the specified time, withdrawn _____ ml (25 ml) of aliquot from a zone midway and filtered the each sample immediately through a syringe filter (0.45 µm PVDF type or equivalent), discarding first 5 ml portion of the filtrate. Collect the subsequent filtrate and cool at room temperature.

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Apparatus USP Type I (Basket); Stage II:					
Stage	**Vessel No.**	**Sample Absorbance**	**%**	**GM**	**% CV**
Stage 1	1				
	2				
	3				
	4				
	5				
	6				
Stage 2	7				
	8				
	9				
	10				
	11				
	12				
Mean of stage 1 and stage 2					
			Limits:		

% RSD of Standard absorbance: _____ (Not more than 2.0)

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Stage I:

S. No.	Paddle ID No.	Vessel ID No.	Temperature (°C)	Tablet dropping time (min)	Sample Withdrawal time (min)
1					
2					
3					
4					
5					
6					

Stage II

S. No.	Paddle ID No.	Vessel ID No.	Temperature (°C)	Tablet dropping time (min)	Sample Withdrawal time (min)
1					
2					
3					
4					
5					
6					

Standard preparation:

Weighed _____ mg (about 25 mg) of Prednisone reference/working standard in _____ ml (50 ml) volumetric flask, added _____ ml (2.5 ml) ethanol to dissolve and made the volume with purified water. Diluted _____ ml (4 ml) of above solution to _____ ml (100 ml) with purified water.

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Sample preparation:

1 Tablet → _____ ml (500 ml) of Deareted Purified water. At the end of the specified time, withdrawn _____ ml (25 ml) of aliquot from a zone midway and filtered the each sample immediately through a syringe filter (0.45 µm PVDF type or equivalent), discarding first 5 ml portion of the filtrate. Collect the subsequent filtrate and cool at room temperature.

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Apparatus USP Type II (Paddle); Stage II:

Stage	Vessel No.	Sample Absorbance	%	GM	% CV
Stage 1	1				
	2				
	3				
	4				
	5				
	6				
Stage 2	7				
	8				
	9				
	10				
	11				
	12				
Mean of stage 1 and stage 2					
			Limits:		

Remarks: The Instrument Calibration is **OK/ Not OK** as per **USP** requirements.

Calibrated By: **Date**	**Checked By:** **Date**	**Approved By:** **Date**