



**STANDARD OPERATING PROCEDURE**

<b>Department:</b> Quality Control	<b>SOP No.:</b>
<b>Title:</b> Operation and Calibration of Tap Density Tester	<b>Effective Date:</b>
<b>Supersedes:</b> Nil	<b>Review Date:</b>
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**1.0 OBJECTIVE:**

To lay down a procedure for operation & calibration of Tap Density Tester.

**2.0 SCOPE:**

This SOP is applicable for operation & calibration of Tap Density Tester in Quality control department.

Model: ETD-1020

Make: Electro lab

**3.0 RESPONSIBILITY:**

Officer, Executive – Quality Control.

Head of Department - Quality Control.

**4.0 PROCEDURE:**

**4.1 Start up (for first time)**

- 4.1.1 Place the instrument on a leveled surface
- 4.1.2 Connect the instrument to a stabilized power supply.
- 4.1.3 Connect the printer port with the printer if required.
- 4.1.4 Switch ON the instrument.
- 4.1.5 Allow the instrument to initialize itself.
- 4.1.6 Use the scroll bar ⇐ to set USP in mode menu.
- 4.1.7 Press set key and enter key till the clock menu displays.
- 4.1.8 Set the time using the scroll bars to increase or decrease the value in each digit.
- 4.1.9 Press ENTER key to go to DATE menu.
- 4.1.10 Set the date using scroll bar to change the date and enter.
- 4.1.11 Press SET to exit the menu.

**4.2 Operation using USP Mode**

- 4.2.1 Switch on the instrument.
- 4.2.2 Ensure that the instrument is in validity period of calibration.



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- 4.2.3 After initialization, select USP 1 or USP 2 by pressing the method toggles key.
- 4.2.4 Press SET key and set the tap counts 1 using up/down and digit scroll key.
- 4.2.5 Set the counts 1 as 10 taps.
- 4.2.6 Similarly, Set the count 2 as 500 and third count as 1250.
- 4.2.7 Press the SET key to exit the menu or press the ENTER key to scroll back to set menu.
- 4.2.8 Pass a quantity of material sufficient to perform the test through 20-mesh screen (or as specified in the individual STP) to break up any agglomerates.
- 4.2.9 Transfer a quantity sifted (about 100 g or as specified in the individual STP or as per GTP) into a dry 250 ml graduated cylinder with 2 mL accuracy.
- 4.2.10 Alternatively transfer a quantity of material into a 100 ml cylinder with 1 ml accuracy to minimize the sample quantity.
- 4.2.11 Mention the modified condition in the results when using modified condition.
- 4.2.12 Without compacting, measure the volume of the sample ( $V_0$ ) to the nearest graduation mark.
- 4.2.13 Press START key and follow the following instructions displayed on the displayed window.
- 4.2.14 Enter sample weight in gram using up/down/digit scroll keys in the keypad.
- 4.2.14. Enter the volume ( $V_0$ ) of the untapped material using up/down/digit scroll keys.  
1
- 4.2.15 Keep the cylinder in the respective holder and lock the assembly.
- 4.2.16 Press the start key again to run the test.
- 4.2.17 After programmed count 1 is over, measure the volume ( $V_{10}$ ) and enter the tapped volume.
- 4.2.18 Press the START key to resume count 2 programs.
- 4.2.19 Measure the volume at the end of count 2 program and enter the value ( $V_{500}$ ) using up/down/ digit scroll keys.
- 4.2.20 Press the START key to resume count 3 programs.
- 4.2.21 Measure the volume at the end of count 3 program and enter the value ( $V_{1250}$ ) using up/down/ digit scroll keys.



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- 4.2.22 The difference between the two counts ( $V_{500}$ ) and ( $V_{1250}$ ) will be displayed as a result.
- 4.2.23 If the result, after the completion of count 3 is less than 2 mL, press STOP key to end the operation and to get the results.
- 4.2.24 If the result is more than 2 mL then press START key again press start key to start the count 3 to get a new ( $V_{1250}$ ) value.
- 4.2.25 Repeat the operation till the difference is less than 2 mL
- 4.2.26 Press stop key once the desired difference is obtained.
- 4.2.27 The results of bulk density or initial density, tapped density and Hausner ratio and compressibility index will be displayed one after another by using down key.
- 4.2.28 The results shall be recorded separately or taken as a print out.
- 4.2.29 The print out can be obtained directly, if the printer is attached online.
- 4.2.30 Pressing the set key will initialize the instrument for the new test.

**4.3 Measurement using User method:**

- 4.3.1 Select the user method using scroll key.
- 4.3.2 Set the required taps using up/down/digit scroll keys.
- 4.3.3 Set the sample in the required holder and note the volume.
- 4.3.4 Start the run using START key.
- 4.3.5 The test stops at the end of the set taps.
- 4.3.6 Record the volume.

**4.4 Measuring the density for In house Analysis:**

- 4.4.1 Prepare the samples as per the standard test procedure.
- 4.4.2 Set the instrument as given in the individual test procedure and operate the instrument.
- 4.4.3 Record the reading either as a print out or as a manual recording.

**4.5 Calibration:**

- 4.5.1 Operate the instrument as mentioned in the above procedure.

**4.5.2 USP Type I (300 Drops( Taps) per minute)**

- 4.5.2.1 Set the method to **USP Type 1 (300 Drops/minute)** with help of METHOD key.
- 4.5.2.2 Set the mode to **user** mode with help of cursor key.

**4.5.3 USP Type II (250 Drops(Taps) per minute)**



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4.5.3.1 Similarly, Set the method to **USP Type 2 (250 Drops/minute)** with help of method key.

4.5.3.2 Set the mode to **user** mode with help of cursor key.

**4.5.4 Number of Drops( Taps) USP 1**

4.5.4.1 Set the tap count to 100 on display with help of cursor on keyboard.

4.5.4.2 Start the instrument by pressing START key and also start calibrated stopwatch simultaneously.

4.5.4.3 Note the time taken (in second) for 100 taps.

4.5.4.4 Similarly set tap count to 300, 500, 750, 1250. Observe the time taken (in second) and calculate taps by following formula.

$$\text{Taps} = \frac{\text{Observed time of set tap (in Second)} \times 300}{60}$$

$$\text{Taps/minute} = \frac{\text{Set tap} \times 60}{\text{Observed time of set tap (in Second)}}$$

**4.5.5 Number of Drops( Taps) USP 2**

4.5.5.1 Set the tap count to 100 on display with help of instrument keyboard.

4.5.5.2 Start the instrument by pressing START key and also start calibrated stopwatch simultaneously.

4.5.5.3 Note the time taken in second for 100 taps.

4.5.5.4 Similarly set tap count to 250, 500, 750, 1250. Observe the time taken (in second) and calculate taps by following formula.

$$\text{Taps} = \frac{\text{Observed time of set tap (in Second)} \times 250}{60}$$

$$\text{Taps/minute} = \frac{\text{Set tap} \times 60}{\text{Observed time of set tap (in Second)}}$$

**4.5.5.6 Limit for Taps (USP 1 & USP 2):**

For 100 taps -  $100 \pm 5\%$  (95 - 105)

For 250 taps -  $250 \pm 5\%$  (237.5 - 262.5)

For 500 taps -  $500 \pm 5\%$  (475 - 525)

For 750 taps -  $750 \pm 5\%$  (712.5 - 787.5)



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For 1250 taps -  $1250 \pm 5\%$  (1187.5 - 1312.5)

**Limit for Drops (Taps) per minute (USP 1 & USP 2):**

USP 1  $300 \pm 15$

USP 2  $250 \pm 15$

**4.5.6 Drop height of the Big /Small holder:**

4.5.6.1 Open the top cover by removing bottom screws.

4.5.6.2 Turn the cam attached to the motor shaft clockwise so that the cylinder holder shaft is at the minimum position.

4.5.6.3 Make sure that the bottom plate of the cylinder holder is in perfect contact with the tapping platform.

4.5.6.4 Measure the distance using vernier caliper between the platform and the clamp plate of the cylinder holder. ( $H_R$ ).

4.5.6.5 Rotate the cam further by hand clockwise till the cylinder holder shaft reaches the maximum height. At this position the shaft tip should be on the falling edge of the cam arm. Mark this arm as no. 1.

4.5.6.6 With a digital vernier caliper measure the distance between the top plate of the frame and the clamp plate of the cylinder holder. ( $H_I$ ).

4.5.6.7 Make a mark on the point of measurement on the top plate of the frame and on the cylinder plate.

4.5.6.8 The difference between the two readings is the drop height of the cylinder.

4.5.6.9 Repeat this procedure for the remaining arms of the cam.

4.5.6.1 Make sure that the point of measurement on the platform and on the cylinder holder remains same for all the measurements.

**4.5.6.1 Acceptance criteria:**

**1** Drop height of the Big holder of USP Type I should be  $14 \pm 2$  mm.

Drop height of the Small holder of USP Type II should be  $3 \pm 0.2$  mm.

**4.5.7 Frequency – Quarterly of USP Type I (300 Drops/minute), USP Type II (250 Drops/minute) & Taps**

**Frequency- Six Month of Drop height of the Big /Small holder:**



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**4.5.8** If the instrument is out of calibration, put Out of calibration label and proceed as per SOP.

**5.0 ANNEXURE (S) :**

Annexure - I: Calibration Record of Tap Density Apparatus.

**6.0 REFERENCE (S):**

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

**7.0 ABBREVIATION (S)/DEFINITION (S):**

USP – United State Pharmacopoeia

GTP – General Test Procedure

SOP – Standard Operating Procedure

**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**

**CALIBRATION RECORD TAP DENSITY APPARATUS**

<b>Location</b>		<b>Page No.</b>	7 of 8
<b>Manufactured</b>		<b>Model No.</b>	
<b>Date of Calibration By</b>		<b>Instrument ID No.</b>	
<b>Next Calibration Due on</b>		<b>Frequency</b>	

**1.0) DROPS(TAPS ) PER MINUTE (QUATERLY)**

**STOP WATCH ID:**

**CALIBRATION DUE DATE:**

<b>USP 1 DROPS(TAPS ) / MINUTE</b>				
<b>S.No.</b>	<b>Set Tap</b>	<b>Time in Second</b>	<b>Observed Value</b>	<b>Limit (300±15)</b>
1.	300			
2.	300			
3.	300			

  

<b>USP 2 DROPS / MINUTE</b>				
<b>S.No.</b>	<b>Set Tap</b>	<b>Time in Second</b>	<b>Observed Value</b>	<b>Limit (250±15)</b>
1.	250			
2.	250			
3.	250			

$$\text{Taps/minute} = \frac{\text{Set tap} \times 60}{\text{Observed time of set tap (in Second)}}$$

**2.0) NUMBER OF DROPS(TAPS)**

<b>USP 1</b>				
<b>S.No.</b>	<b>SET TAPS</b>	<b>OBSEREVED TIME</b>	<b>OBSEREVED TAPS</b>	<b>TAPS(Limit)</b>
1.	100			95 - 105
2.	300			237.5 - 262.5
3.	500			475 - 525
4.	750			712.5 - 787.5
5.	1250			1187.5 - 1312.5

  

<b>USP 2</b>				
<b>S.No.</b>	<b>SET TAPS</b>	<b>OBSEREVED TIME</b>	<b>OBSEREVED TAPS</b>	<b>TAPS(Limit)</b>
1.	100			95 - 105
2.	250			237.5 - 262.5



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3.	500			475 - 525
4.	750			712.5 - 787.5
5.	1250			1187.5 - 1312.5

$$\text{USP1 (Taps)} = \frac{\text{Observed time of set tap (in Second)} \times 300}{60}$$

$$\text{USP 2 (Taps)} = \frac{\text{Observed time of set tap (in Second)} \times 250}{60}$$

**3.0) Drop Height (HALF YEARLY)**

**VERNIER CALLIPER ID:**

**CALIBRATION DUE DATE:**

	USP 1				USP 2				
Big Holder	Arm 1 H <sub>I</sub>	Arm 2 H <sub>2</sub>	Arm 3 H <sub>3</sub>	USP Standard	Arm 1 H <sub>I</sub>	Arm 2 H <sub>2</sub>	Arm 3 H <sub>3</sub>	Arm 4 H <sub>3</sub>	USP Standard
H <sub>I</sub>				-					-
H <sub>R</sub>				-					-
Drop Ht (H <sub>I</sub> - H <sub>R</sub> )				14±2 mm					3±0.2 mm
	USP 1				USP 2				
Small Holder	Arm 1 H <sub>I</sub>	Arm 2 H <sub>2</sub>	Arm 3 H <sub>3</sub>	USP Standard	Arm 1 H <sub>I</sub>	Arm 2 H <sub>2</sub>	Arm 3 H <sub>3</sub>	Arm 4 H <sub>3</sub>	USP Standard
H <sub>I</sub>				-					-
H <sub>R</sub>				-					-
Drop Ht (H <sub>I</sub> - H <sub>R</sub> )				14±2 mm					3±0.2 mm

**Status :** The Instrument Calibration is **OK/ Not OK**

**Calibrated By :**

**Checked By :**

**Approved By :**

**Date :**

**Date :**

**Date :**