STANDARD OPERATING PROCEDURE	
Department: Quality Control	**SOP No.:**
Title: Process Validation and Equipment Qualification	**Effective Date:**
Supersedes: Nil	**Review Date:**
Issue Date:	**Page No.:**

1.0 OBJECTIVE:

To lay down a procedure for operation & calibration of Visual Melting Range Apparatus. (Make: Labindia).

2.0 SCOPE:

This SOP is applicable to carry out visual Melting range of material in the Quality control department. The Scope of this SOP covers the operation and calibration of melting range apparatus as per the current pharmacopoeias (IP/BP/USP) and In-House requirements.

3.0 RESPONSIBILITY:

Officer, Executive – Quality Control.

Head - Quality Control.

4.0 PROCEDURE :

4.1 Operation:

4.1.1 Ensure that instrument is clean and free from dust.

4.1.2 Check the calibration status of instrument.

4.1.3 Connect the instrument power plug to mains supply socket.

4.1.4 Connect the monitor power mains plug to mains supply socket.

4.1.5 Switch ‘ON’ the Instrument.

4.1.6 Connect the printer power plug to mains supply socket.

4.1.7 Carefully remove the lens cap from the CCD camera lens.

4.1.8 Switch on the monitor by pressing the power switch.

4.1.9 Switch ON the instrument, the illuminator lamp shall blink on / off and the 20 character, two line display shall read

4.1.10 After 2 to 3 Seconds, the display shall be

LAB INDIA MELTING RANGE APPARATUS
MODEL: MR-VIS Version: 2.2.2

4.1.11 Again after 2 to 3 seconds, the display shall be

1) Prog 2) Run 3) Print 4) Clock 5) Fur.Temp:

4.1.12 Keep the instrument 'ON' for 15 to 20 minutes for warming before actually starting the sample analysis.

4.1.13 When the required operation is completed, allow the apparatus to cool to 25 – 30° C.

4.1.14 Switch 'Off' the instrument.

4.2 Sample Preparation :

4.2.1 **Preparation on Glass Slide :**

4.2.1.1 Place the 'jig' on one side of the glass slide and finally spread the sample powder with the help of a spatula. Place the round cover slip on it. Observe that the sample is homogeneously spread in a well-defined circle.

4.2.2 **Preparation in Capillary Tube:**

4.2.2.1 Fill the sample in a One end sealed capillary tube upto 5 mm height by gently tapping against a hard surface.

4.3 Program Entry:

4.3.1 Select 'PROG' from main menu options by pressing 1,display reads

PROGRAM:1>ADD2>VIEW 3>COPY4>DELETE: _

4.3.2 Select 'ADD' from menu by pressing '1' & 'ENTER' keys**.**

4.3.3 The display shall be

Prog. No. (1-20):1 ADD / EDIT Mode	Sample Name: ___

4.3.4 Use Alphanumeric keys to enter Sample Name & press "ENTER" key. (Eg. ABC, continuously press button till required character is displayed, one time for A, 2 times for B, 3 times for C, The display shall be

4.3.5 Use Alphanumeric keys to enter Sample ID & press "ENTER" key. (Eg. MPL/070227, continuously press button till required character is displayed, one time for A, 2 times for B, 3 times for C, The display shall be

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Sample Name:
ABC __

4.3.6 Again press 'ENTER' key**,** Display shall read the type of sample preparation

Sample preparation:
1> slide 2> Capillary

4.3.7 Select '1' for Slide & '2' for capillary (one end sealed) and press 'ENTER' key, display shall read the 'HEAT' mode of melting range determination**.**

Mode:
1>Heat 2>Heat & Cool

4.3.8 Press 1 or 2 for the required mode and enter.

4.3.9 The display shall read Melting range Process / Detection.

Operation:
1> Auto 2 > Manual.

4.3.10 Press 1 or 2 for required operation and press "ENTER". (In the "Heat & Cool" mode only manual operation is possible)

4.3.11 The display shall wait for set temperature entry to be made.

Set temperature °C:

4.3.12 Use numeric keys to enter set temperature value between ambient to 310°C and press "ENTER" key.

4.3.13 If manual operation is selected the display shall be

Furnace temperature Cut Off Limit:

4.3.14 Enter the cutoff temperature between Ambient to 325°C and more than set temperature and press: ENTER" key. The display shall be

Rate: "SET Rate" to select

4.3.15 Press "SET RATE" key to select the heating rate. The display shall be

Rate: 0.2°C/MIN "ENTER" to select…

4.3.16 Press “SET RATE” key, repeat till the desired rate is displayed

Rate: _____°C/min “ENTER” to select…

Press “ENTER” key.

4.3.17 The instrument shall give beep and returns to program screen.

4.3.18 Now another program can be added or modified.

4.4 Viewing Program Parameters:

4.4.1 Repeat steps up to

PROGRAM: 1>ADD 2 >VIEW 3>copy4>DELETE…..

4.4.2 Press 2 and enter. Display shall be

Prog No (1-20): VIEW Mode

4.4.3 Enter the program No. Whose parameters are to be viewed.

4.4.4 The program parameters one after another and display screen returns to program entry menu by pressing “Esc” key.

4.5 Finding Melting Point of a Sample:

4.5.1 Repeat steps up to

1) PROG 2) RUN 3) Press 4) Clock 5) Fur. Temp

4.5.2 Press 2. Display reads

Prog No. 1

4.5.3 The program No. of LAST RUN is displayed

4.5.4 Enter the desired program No. and press “ENTER” key.

4.5.5 If that program does not exist the message displayed is

Prog No.: Prog Does Not Exit!

4.5.6 If program exists then

Prog No.: R.No. 1 1) Run 2) View: 1

R. No. Indicates the run no. of the day.

4.5.7 Press 1 and Enter. The display shall be

Sample No:_____

4.5.8 Enter sample No. using Alphanumeric keys.(Eg. X)

Sample No:_____ X_

4.5.9 Press Enter

Id. No:_____ __

4.5.10 Enter Id. No. using Alphanumeric keys. Eg.M2

Id. No:_____ M2_

4.5.11 Press enter to start “RUN”

4.5.12 The furnace shall start heating and the display shall be

T°C :100.0 SET : 114.0 Wait cooling ! : 99.0

4.5.13 The furnace temperature rises fast tills the sample insertion temperature (In display it is 99.0)

Note: The furnace lid should be placed on the furnace.

4.5.14 If the furnace temperature is more than the sample insertion temperature, then.

T°C :26.5 SET : 114.0 Wait Heating! : 99.0

(*Furnace Temp: 100°, Set temp: 114.0°.*)

4.5.15 As soon as the furnace temperature reaches sample insertion temperature, the display reads

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T°C: 99.0 SET: 114.0
Keep sample& "ENTER"

4.5.16 Remove the furnace lid and place the sample such that its edge (Round) is not visible on the monitor.

4.5.17 If image is not clear, refocus the camera lens by rotating the focusing – Ring

4.5.18 Now press 'ENTER", display shall show, The furnace will heat up to 5°C below set temperature.

T°C: 26.5 SET: 114.0
Wait Heating! 109.0

4.5.19 The Lamp will be switched ON automatically as soon as the furnace temperature reaches 5°C below set temp. Display shall read,

Switching Lamp On…

4.5.20 Now, the furnace temp. rises as per user specified rate and the display shall be

(For Manual Operation, "MANUAL" will appear instead of "AUTO")

T°C : 109.0 ST°C:____
AUTO ET°C:____

4.5.21 In auto operation as soon as the sample starts melting, the photo sensor detects the start and the instrument automatically shall record it and displays this temperature as 'ST.'

T°C : 114 ST°C: 114.5
AUTO ET°C:____

4.5.22 The furnace temp. continues heating as per the heating rate, when the sample completely melts, the end temperature "ET" shall be displayed. The Display shall read,

T°C : 115 ST°C: 114.5
AUTO ET°C : 115.5

4.5.23 In manual operation, record ST & ET by using the lock key.

4.5.24 To print the result report / view the result. Press "Esc" key to enter into main menu. Record the

details as given in log book.

4.6 **Heating Rates Suggested:**

Sample Description	**Suggested Heating Rate**
Unknown range Sample	6 /12°C / min.
Plastic / Fiber Sample	12°C / min.
Dark coloured Samples	Manual Operation 12°C / min
Samples which shrink / become - Transparent on Heating.	3/6/12°C / min.

4.7 **Calibration:**

4.7.1 Repeat all the steps mentioned in 4.1 and 4.2.

4.7.2 Enter programs separately for, Vanillin, Acetanilide, Sulfapyridine, Caffeine and as described in 4.3.

4.7.3 Find the Melting points of Vanillin, Acetanilide, Sulfapyridine, and Caffeine and as described in 4.5 individually.

4.7.4 Press “Esc” key to enter into main menu. Select print by press ‘3’ and Enter.

4.7.5 The display shall be

```
1>View Prev. Result
2>Print
```

4.7.6 Press 2 and Enter, the printout of the calibration shall be given. Enter the calibration details as given in Annexure – I.

4.8 Obtaining Hard Copy of Run Report:

4.8.1 Main Menu – Display shall read,

```
1>Prog 2>Run 3>Print
4> Clock 5>Fur. Temp:
```

4.8.2 Select “PRINT” from Main menu options by pressing “3”.

```
1> View Prev. Result
2> Print:
```

4.8.3 **Frequency:** Quarterly

4.8.4 If the instrument is out of calibration, put an "OUT OF CALIBRATION" tag, and proceed as per SOP No.

4.9 **Precaution:** Never switch off the apparatus above 50°C.

5.0 ANNEXURE (S) :

Annexure – I: Calibration record of visual melting range apparatus

6.0 REFERENCE (S):

Preparation, approval, distribution control, revision and destruction of Standard Operating Procedure (SOP).

SOP Handling of Out of Calibration

USP/BP/Ph. Eur.

7.0 ABBREVIATION (S)/DEFINITION (S):

mm - milli meter

ml - milliliter

temp. – Temperature.

°C – Degree Celsius

min. - Minute

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ANNEXURE I

CALIBRATION RECORD OF VISUAL MELTING RANGE APPARATUS

Location		**Page No. :**	9 of 1
Manufactured By	Lab India	**Model No.**	
Frequency	Quarterly	**Identification No.**	
Date of Calibration		**Next Calibration Due on**	

S.No.	Reference Material	Batch/ Lot no.	Observed Melting range	Limit*	Remarks.
1.	Vanillin				
2.	Acetanilide				
3.	Sulfapyridine				
4.	Caffeine				

* Limit will be assign based on Melting Points standards certificates.

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

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

REVISION CARD

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