**TITLE: OPERATION AND CALIBRATION OF MELTING POINT APPARATUS**

## 1.0 PURPOSE:

To describe the Operation and Calibration of Melting Point Apparatus.

## 2.0 SCOPE:

This procedure is applicable to Melting Point Apparatus in Laboratory.

Make : Datla instruments

Model No : DMP-630

ID No : DIPL/QC/INS/MR/002

## 3.0 RESPONSIBILITY:

3.1 Analyst-QC is responsible to follow this SOP.

3.2 Head-QC/Designee is responsible for ensuring implementation of this SOP.

3.3 Head-QA/Designee is responsible for monitoring overall compliance of this SOP.

## 4.0 DEFINITIONS:

Nil.

## 5.0 PROCEDURE:

### 5.1 Precaution:

5.1.1 Ensure that the power supply to the instrument is switched 'OFF' before cleaning.

5.1.2 Clean the instrument with a clean dry cloth every day.

5.1.3 Occasionally clean with wet cloth dipped in dilute soap solution and immediately remove moisture with dry cloth.

5.1.4 Check the melting point apparatus and silicone oil used in the apparatus.

5.1.5 If color that hinders observation of melting or polymerization is observed, Change the oil.

### 5.2 Selection and Packing of Capillary Tube:

5.2.1 Take dry material as recommended in the individual procedure or monograph

5.2.2 Select a capillary tube with following dimensions:

5.2.3 Length 70mm to 80 mm

5.2.4 Internal Diameter 0.9 mm to 1.1 mm

5.2.5 Wall thickness 0.10 mm to 0.15 mm

5.2.6 One end sealed

5.2.7 Introduce sufficient quantity of dry fine powder in the capillary

5.2.8 Take a glass tube about 180cm long & dry 1 cm ID and place vertically on a hard

5.2.9 Surface

5.2.10 Hold the capillary vertically and drop from the top of tube.

5.2.11 Repeat the operation till the material packed shall be between 4 to 6 mm

5.2.12 Drop the capillary from the top six times

5.2.13 Check the material length .It shall be between 4 to 6 mm

5.3 **Operation:**

5.3.1 Switch on the power supply by switching on main Supply and switch provided on back side of the instrument.

5.3.2 Switch on the mains and light of the Instrument.

5.3.3 Start the stirrer.

5.3.4 Switch on the Heater.

5.3.5 Insert the capillary tube in both.

5.3.6 Start the heating temperature 120°C to 150°C below the actual melting temperature of the sample using high heater operations.

5.3.7 After reach the temperature 120°C to 150°C of the actual melting temperature of the sample then increase the temperature using the low heater option.

5.3.8 Watch the sample through magnifying lens.

5.3.9 Record the results.

5.4 **Calibration:**

5.4.1 Frequency: Once in 3 Months (±3 Days).

5.4.2 Items Required for Calibration: Digital Temperature Indicator.

5.4.3 Melting point standards are:

5.4.3.1 Vanillin AR.

5.4.3.2 Sulphanilamide AR.

5.4.3.3 Caffeine AR.

5.5 **Calibration Procedure:**

5.5.1 Reduce the sample substance to a very fine powder and dry it at temperature below its Melting Temperature.

5.5.2 Take the sample substance (dried) into a capillary tube to from compact column about 3 mm height.

5.5.3 Switch on the power supply by switching on main Supply and switch provided on back side of the Instrument.

5.5.4 Start the stirrer.

5.5.5 Switch on the Heater.

5.5.6 Insert the capillary tube in both.

5.5.7 Start the heating temperature to 120°C to 150°C below the actual melting temperature of the sample using high heater operations.

5.5.8 After reach the temperature 120°C to 150°C of the actual melting temperature of the sample then increase the temperature using the low heater option.

5.5.9 Watch the sample through magnifying lens.

5.5.10 Record the results.

5.6 **Qualification of Melting Point Standards:**

5.6.1 Qualify the Melting point standard with against USP standards in in-house when received the new standards.

5.6.2 Record the observation in current version of QC-035-F-02.

| S.No | Melting Point Standards | Melting Range |
|---|---|---|

| | | |
|---|---|---|
| 1. | Vanillin AR | 81°C to 83°C |
| 2. | Sulphanilamide AR | 164°C to 166°C |
| 3. | Caffeine AR | 234°C to 239°C |

**6.0 FORMATS / ANNEXURE(S):**

6.1 Melting point apparatus calibration : QC035-FM071

6.2 Melting Point standards Qualification Record : QC035-FM050

6.3 Instrument Usage log book : QC048-FM086

**7.0 CHANGE HISTORY:**

| Revision No. | Effective Date | Details of Revision | Ref CCF No. |
|---|---|---|---|
| 00 | 15.07.2014 | New SOP introduced | -- |
| 01 | 09.02.2015 | 1. Replace the thermometer to Digital temperature indicator for calibration.<br>2. Replace 4-Nitroanaline to Sulphanilamide for calibration.<br>3. Caffeine Melting range acceptance criteria changed as 234°C and 239°C. | -- |
| 02 | 01.08.2016 | Include the qualification MR standards. | -- |
| 03 | 01.01.2017 | SOP format changed make to line with SOP-QA-001-04. | QC-CRF-025/16 |

| | Prepared by | Reviewed by | Checked by |
|---|---|---|---|
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