

# DIGITAL MELTING POINT APPARATUS

Mode-CDMP

## Operating Manual



**CONTECH**<sup>®</sup>  
**Instruments Ltd.**

AN ISO 9001,14001 & OHSAS 18001 COMPANY

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## INTRODUCTION

Melting point of a substance is the temperature at which the substance changes from solid state to liquid state. The measurement of a melting point is a basic means of understanding the nature of substance and also is one of the important parameter in the chemical industry and medicinal research. The melting point apparatus is an essential instrument in Pharmaceuticals, Perfumes, Dyestuff, Food products & any other organic crystal substances.

**Model CDMP-300** Melting Point Apparatus is based on Photo Electric Detection Technique and the reading of temperature at the point of melting by the temperature sensor Pt-100.

This instrument can display initial and final melting points, record the melting curve and display average value. The melting point apparatus uses the capillary sample tubes as per the specifications of Pharma Copia.

## SPECIFICATIONS

Melting point measurement range : Ambient to 300 deg C

Starting melting point temperature : Settable from 50 to 300 deg C

Heating Time : 3 Minutes (Up to 300deg C)

Cooling Time : 5 Minutes (From 300 deg C)

Accuracy : +/- 0.5 deg up to 200

+/- 0.8 deg from 200 to 300

Temperature display resolution : 0.1 deg C

Rate of heating (deg C per minute) : 0.2, 0.5, 1, 1.5, 2,3,4,5

Deviation of heating rate : Less than 10% of set value.

Accuracy of melting point : +/- 0.5 deg C (up to 200 deg C)

+ / - 0.8 deg C ( from 200 to 300 deg C)

Repeatability of melting point reading : +/- 0.2 deg C when heating rate is 0.2 deg C

+/- 0.3 deg C when heating rate is 1 deg C

Capillary sample tube size : O.D. 1.4 mm, I.D. 1.0 mm, length 80 mm

Height of sample loading in capillary tube : 3mm

Serial interface : RS-232, 8 bits data, 1 stop bit, 9600 baud rate.

Temperature Sensor : Pt-100.

Power : 220 +/- 10% V 50/60 Hz 100 Watt

Dimension : 280 L X 400 D X 215 H mm

Weight : 12 kg

## PRINCIPLE OF OPERATION

When a crystalline substance melts, there is a transition from solid to liquid state which results into transition from reflection to the transmittance of light through the capillary containing the sample. This light is detected by the photo electric technique and the temperature at that exact point is measured by the high precision RTD sensor Pt-100 and is displayed.

$t(I)$  is the initial melting point, the temperature of the sample at the point it starts melting and  $t(F)$  is the final melting point, the temperature of the sample when it is fully melted. The interval  $t(F) - t(I)$  is the melting range.

These points are detected automatically and saved on the display until cleared for the next measurement.

The light from the incandescent source passes through the optical fibre, electric furnace in which the capillary is housed and the capillary tube socket before falling on the sample placed in the capillary. The light penetrated through the molten sample in the capillary is then received by the photo electric detector. As the sample melts, the light flux increases and the micro computer records the temperature readings from the platinum resistance detector inserted in the capillary tube socket placed directly below the capillary. The instrument displays initial and final melting point readings as well as the melting curve. Cooling fan is provided to cool the furnace after the completion of the test.

## INSTALLATION

### 1. Unpacking :

Unpack the Melting Point Apparatus. Save the packing container for future use.

### 2. Electrical Requirements :

Melting Point Apparatus requires very stable power. It works on 230V AC supply with proper earthing. The power outlet used for the Melting Point Apparatus should not be shared with any other devices which draws current in inconsistent manner like Air Conditioner or Refrigerator, etc.

### 3. Environmental Requirements :

For best results, Melting Point Apparatus should be placed on a level surface which is free from drafts. It should not be exposed to direct sunlight or radiated heat. Table used for Melting Point Apparatus should be sturdy and should not transmit vibration from other equipments and free from the movement of people. No vibration producing equipment should be operated on the same platform as Melting Point Apparatus.

## OPERATION

Turn Power ON through the power switch on the back panel. The screen displays the starting temperature and rate of heating as per the previous setting(setting before the instrument was switched off last time). Cursor is as "Start T" (initial temperature of the test).

Initial temperature can be changed by entering required temperature through the keyboard & followed by ENTER key (↵).

Cursor moves to "Rate" (heating rate). Input the rate of heating in deg C per minute followed by Enter key (↵).

While entering numeric data, any editing in the previous digit can be done by using back arrow key (←).

The temperature starts raising at a higher heating rate till the set value of starting temperature & settles at the initial temperature value.

At this stage insert the capillary filled with sample upto 3mm height (for consistency of melting point results loading height should be consistently maintained) & press warming key.

Now the temperature starts raising slowly as per the set value of rate of heating. The screen displays the graph of light transmittance(y axis) v/s melting point temperature (x axis) with initial melting point (S Melt) & Final melting point (F Melt) in deg C. The current value of temperature at capillary is also displayed on the top of the screen with ↑ indicating increase in the temperature. The screen records the initial and final melting values along with the graph. After the detection of final melting value the heating stops & the temperature settles at the set initial temperature.

Incase the capillary is not inserted while starting the test ( pressing warming key) the screen displays an error.

This instrument allows maximum 3 tests to be carried for finding average value of melting point results.

For repeating the test with fresh sample, press warming key. New graph next to the 1<sup>st</sup> graph corresponding to the new sample is displayed in the screen. Similarly the 3<sup>rd</sup> sample can also be tested. Average value of S Melt and F Melt is displayed on the screen. However, incase any of the test readings to be discarded (results not as desired) the same can be done by clearing the results.

Clearing any particular result can be achieved by pressing Clear Key, Screen displays 1 2 3 C. 1

corresponds to the left most graph and 3 corresponds to the right most graph on the screen. Press the number of the graph to be deleted by inputting the corresponding number in the keyboard. Now the new average values are displayed. Discarded result can be replaced by conducting a new test. Maximum upto 3 results can be considered for the average values.

A new test after the 3 results on the screen will automatically clear all the previous results and the results of the new sample displayed.

Incase the test has to be carried for a different sample altogether fresh start temperature is entered with the cursor on Start T followed by new heating rate.

Use Reset key on the top right hand side corner for resetting all the results in the memory.

## **CALIBRATION**

Instrument may need Calibration if it is not used for long time or extreme changes in the ambient temperature.

Calibration of the equipment is carried with known samples (either 1 point or 2 point or 3 point).

For Calibration, setting temperature should be 5 deg less than the final melting point of the standard sample and the rate of heating is 1°C/minute

Press CAL Key. Display shows '0' or '1'.

Press '0' for recovering factory default calibration values and press '1' for new calibration.

User can select either 1 or 2 or 3 point calibration. Calibration is carried by using known melting point standards to cover the entire range. Generally standards naphthalene (melting point 81.00°C) Adipic acid (melting point 152.89°C) & Anthraquinone (melting point 285.96°C) are used to cover the range.

## **DISPLAY PARAMETERS**

'S' Melt : Average of initial melting point.

'F' Melt : Average of final melting point.

↑ : Heating

↓ : Cooling

1 2 3 C Press Clear key followed by Number 1, 2 or 3 to delete the particular melting curve.

## **MATTERS REQUIRING ATTENTION**

1. Preparation of sample : Sample must be baked in accordance with the requirements and pulverized in a dry and clean bowl. Capillary should be knocked to fill by “free-falling” method so that the sample is firmly loaded. Loading height of the sample must be consistent (approx 3 mm). It is recommended to load multiple capillary tubes at the same time to maintain the consistency of loading for repeating the tests and to select best 3 results to get the average value.
2. Repeatability of measurement : For achieving consistency in the measurement , set value of initial temperature and heating rate should not vary with tests. For better accuracy of result, heating should be kept low. When the heating rate is selected at “1°C/m” the initial temperature setting should be atleast 3-5° less than the melting point of the sample and if the heating rate is selected at 3°C/m , the initial temperature setting should be atleast 9-15° less than the melting point of the sample. For unknown samples (melting point of the sample is not known), it is better to try with higher heating rate for the speedy determination of approximate melting point value and then use lower heating rate & repeat the test to get accurate result.
3. Capillary must be wiped off stains and dirt on the outer surface with a piece of soft cloth before inserting into the instrument to avoid accumulation of dirt on the bottom of the socket which will lead to wrong results (no transmittance of light resulting in test failure).
4. When the instrument is turned ON the temperature of the furnace would raise upto the value of initial temperature setting of the previous test. If the desired temperature is different set a fresh value.

## **MAINTENANCE**

The capillary used in the instrument must be from reputed manufacturer. Never use capillary drawn manually as they are easy to breakdown.

Incase the capillary is broken in the tube socket , power source to the instrument must be switched off and the furnace should be allowed to cool down. Insert a 1mm drill into the broken capillary and lift the drill slowly to take out the capillary. If there are still some broken glasses left in the tube socket, tube socket can be pulled out to clear the glasses from the socket. Using the drill break the glass pieces and pour them out. Place the tube socket in the earlier position (make sure the notch matches the slot). Check the instrument to confirm the socket is cleared off broken capillary.

Incase of error display “En” look for the following cause :

1. Starting temperature too high.
2. Capillary is not inserted in the socket.
3. Sample loading in the capillary is not proper.



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