

APARATO AUTOMÁTICO PARA PUNTO DE FUSIÓN
AUTOMATIC MELTING POINT APPARATUS
APPAREIL AUTOMATIQUE POUR LE POINT DE FUSION

REF.- CODE - RÉF. KPC001
MODELO - MODEL - MODÈLE 440



Este manual es parte inseparable del aparato por lo que debe estar disponible a todos los usuarios del equipo. Le recomendamos leer atentamente el presente manual y seguir rigurosamente los procedimientos de uso para obtener las máximas prestaciones y una mayor duración del mismo.

This manual should be available for all users of these equipments. To get the best results and a higher duration of this equipment it is advisable to read carefully this manual and follow the processes of use.

Ce manuel est une partie indissociable de l'appareil et doit être mis à la disposition de tous les utilisateurs de l'équipement. Nous vous recommandons de lire attentivement ce manuel et de suivre scrupuleusement les procédures d'utilisation afin d'obtenir des performances maximales et une plus longue durée de vie de l'appareil.

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1. APPLICATIONS AND FEATURES

According to the definition in physical chemistry, the melting point of a substance is the temperature at which the material changes from a solid to a liquid state. In organic chemistry, melting point determination is not only the basic approach to determine the nature of material, but also an important method to obtain the purity of a substance. Therefore, melting point apparatus is widely used in chemical industry and medical research, and it's a must-have instrument in the manufacturing of medicine, spices, dyes and other organic crystal substances.

The model 440 melting point apparatus adopts automatic photoelectric detection, dot-matrix graphic 6.9-inch LCD and touch screen buttons. It's able to record melting curve automatically, and display start temperature and stop temperature. The application of platinum resistance thermometer, digital PID adjustment and PWM temperature control technology in the temperature control system secures accuracy and reliability of melting point determination. The instrument will automatically save working parameters and measurement results. It's also able to communicate with PC through USB or RS232 interface. Can be connected to the micro printer which is supplied with the device.

2. SPECIFICATIONS

Rango de temperatura	Temperatura ambiente - 360 °C
Measurement method	Automatic
Number of capillaries	3
Temperature resolution	0.1 °C
Linear temperature rate	From 0.1 °C/min to 20.0 °C/min
Temperature accuracy	≤ 200 °C: ±0,4 °C > 200 °C: ±0,7 °C
Repeatability	0.3 °C (Linear temperature rate: 1 °C/min)
Measurement modes	8
Dimensions of capillaries	Outside diameter: 1.4mm Inside diameter: 1.0mm Length: 90mm
Capillary fill height	3mm
Data storage	1000 sets
Interface	USB and RS232
Power	100V-240V, 50/60Hz, 120W
Dimensions (L×W×H)	370mm×285mm×175mm
Net weight	5kg

3. PRINCIPLE OF OPERATION

The working principle of the instrument is based on the following fact: when a substance is in its crystal state, it reflects the light, when is in melted state, it transmits light. Therefore, in the process of melting, a transition of the transmissivity will occur with the rise of the temperature.

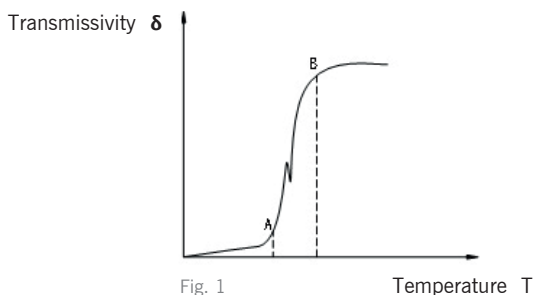


Fig. 1

Fig.1 shows a typical melting curve (the temperature-transmissivity curve). In Fig.1, the temperature T_a (to which point A is corresponding) is called initial melting point; and the temperature T_b (to which point B is corresponding) is called final melting point (or full melting point). The length of T_a - T_b is called melting range (or melting interval).

The principle of the instrument is shown in figure 2. Light from the light source goes through the condenser, the oven and a light transmission hole on the holder of capillary and concentrates on the capillary tube. As passing through the sample to be tested, it is received by silicon photocell. After amplification and A/D conversion, the photoelectric signal is sent to MCU to process. The temperature is detected by the platinum resistance thermometer which is directly inserted on the bottom of capillary holder. The temperature signal is processed with non-linear correction, voltage amplification, and A/D conversion and then sent to be further processed in MCU. MCU collects data such as start temperature and ramp rate. After calculation, the output control signal drives executor with temperature controller. When the temperature of oven is higher than the start temperature, cooling fan works and the oven starts cooling. When the temperature of oven is lower than the set one, electric wire of the oven starts heating. Through such a close cycle system, the temperature of oven is controlled by MCU at the set value, therefore, full-speed heating and cooling or linear ramping of oven is realized.

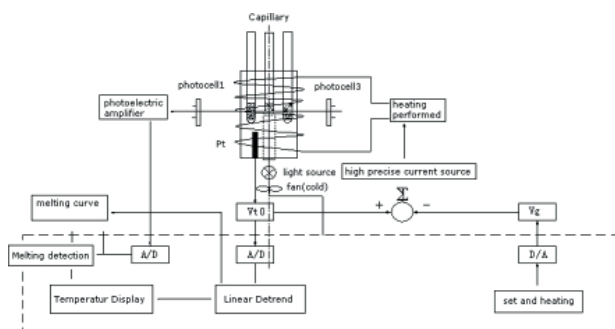


Fig. 2

4. APPAREANCE

1. Front view of the instrument



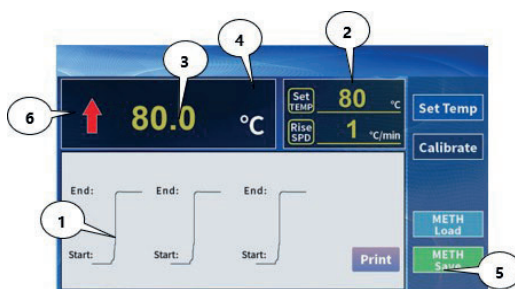
2. Back view of the instrument



5. OPERATION

This melting point apparatus is a new model with full touch screen button. Compared with the previous one, this model has enhancement in circuit, increase the storage of measurement data, and displays real-time clock. At the same time, it can be calibrated in the program with software, and the ramp rate can be set at any value from 0.1 °C/min to 20 °C/min.

1. Turn on the machine



1) Instructions of display

1. Showing the melting curves of the last three measurements.	2. Temperature and heating rate setting. Click on the number area to set the required parameters.
3. Real-time oven temperature.	4. Heat preservation and multi-melting mode prompt (on demand).
5. Timer.	6. Red up arrow indicates heating, green down arrow indicates cooling.

2) Functions of keys

Set TEMP	Preset temperature
Rise TEMP	Only display until the preset temperature of the instrument is reached. Press this button and the instrument starts to heat steadily according to the set heating rate.
Calibrate	Calibration of temperature (Operate cautiously!).
Record	Show stored records, maximum 1000 sets.
Methods Load	8 measuring methods can be set and stored (preset temperature and heating rate).
Methods Save	Save the methods that are set.
Print	Print the latest measurements and melting curves.

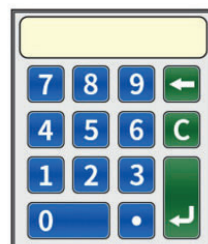
2. Preset

1) Click on the preset temperature number on the screen, and the screen displays the digital keyboard, see the figure on the right.

2) Press the number key to enter the required preset temperature□ click "Enter".

3) Note: The maximum input temperature cannot be exceeded, otherwise the input is invalid.

4) Click on [Set TEMP] to start preheating.



3. Rise the temperature

- 1) When the temperature of the instrument reaches the preset temperature and is stable for 30 seconds, the device will beep, indicating the preset is completed. At the same time, [Rise TEMP] will appear to perform the next operation.
- 2) Click on [Rise SPD] number to set the heating rate.
- 3) Insert the capillary with testing sample into the oven, press [Rise TEMP] and the display shows as in Figure 3.

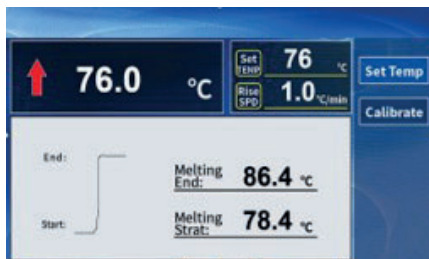


Fig. 3

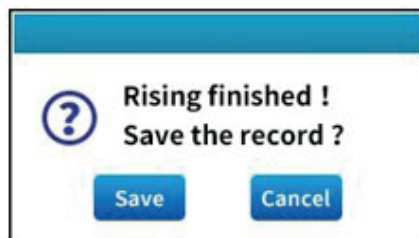


Fig. 4

4) If the capillary wasn't inserted or the capillary has no sample inside, the device will beep and show error in 5 seconds, then it will return to preset state.

5) During the heating process, you can press [Set TEMP] to exit the heating at any time.

6) As the temperature rises, the melting curve of the sample will be depicted on the display screen, and the initial and final melting temperatures will be displayed, as shown in Figure 3.

7) Press the [Hold] key, the temperature window has the letter "H", the instrument will be in the heat preservation mode. And the current temperature will no longer rise (according to the different heating rate, there may be a slight deviation). It can be used to measure special samples. User can press the [Hold] button again to exit.

8) Press the [Double Melt] key, the letter "M" is displayed in the temperature window, and the instrument will be in the multi-melting point measurement mode. When the instrument detects the first final melting-point, it will not stop measuring but will continue heating until the second final melting-point is detected. It can be used to measure special samples. Users can press the [Double Melt] button again to exit.

9) When the instrument detects the final melting-point of the sample, it will beep and automatically return to the preset temperature. The screen prompts whether to save the measurement results, as shown in Figure 4, the user can choose to save or abandon the measurement values.

10) When measurement is finished, press the [Print] key to print out the measured data and melting curve.

4. Measurement modes

The instrument can store 8 common measurement modes (preset temperature and heating rate). Users can load the stored data at any time or save the set parameters to the instrument.

1. Load: Press the [METH Load] key to display the right-hand interface, click the number 1-8 to test with the required measurement mode.
2. Save: Press the [METH Save] key to display the right-hand interface, click the number 1-8 to save the current set measurement mode.
3. If no need of these two modes, press the [Cancel] key to exit.



5. Records

Press the [Record] key to display the interface as shown below. The instrument can store 1000 records, including date, time, initial temperature, heating rate, initial melting-point temperature, final melting-point temperature, etc.

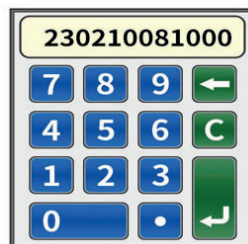
When the number of records exceeds 1000 sets, the instrument will automatically delete the oldest records and keep new records.

[illegible]

6. Time setting up

Users can click on the upper right of the main display screen to change the time.

Enter the time in order of “year-month-day-hour-minute-second” (in this order necessarily). For example, to get the time “2023-02-10 08:10:00”, input the number “230210081000” in order.



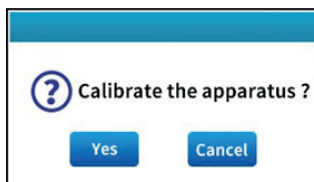
6. CALIBRATION OF TEMPERATURE

- Please note that the melting point apparatus does not need to be calibrated after it is manufactured. Users should read the following carefully and operate with caution.
- If the parameters are incorrect due to improper operation, please resume to default setting according to part two in below.
- **Warning:** the standard substance which meets the requirements of national standards must be used to calibrate the instrument, along with the correct calibration method; otherwise, the accuracy of the instrument will be greatly influenced.

1. Calibrate the temperature

If the measurement accuracy is poor, the instrument can be calibrated with standard materials. Users can use one-point correction or multi-point correction according to their needs.

1) Press [Calibrate] to indicate whether the temperature really needs to be corrected. Press [Cancel] to quit.



2) After entering the correction mode, the display screen shows the correction point selection interface (Figure 5). There are three correction points in total.

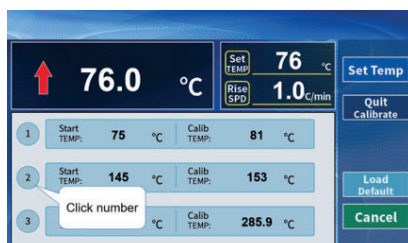


Fig. 5

3) The default calibration samples used by melting point apparatus are naphthalene (80.7 °C), adipic acid (153.0 °C) and anthraquinone (285.9 °C). If the user does not use the above samples, click on the number to input the starting temperature and correction temperature according to their standard sample melting point.

4) After setting the starting temperature and correction temperature, press 1-3 digital key to select the point to be corrected. The instrument automatically starts the temperature preset according to the preset starting temperature.

5) When the preset is done, insert the capillary and press the [Rise TEMP] to heat up.

6) When calibrating, the heating rate is set at 1.0 °C/min.

7) After the instrument detects the final melting-point, the device will beep automatically return to the preset starting temperature. The screen displays the correction confirmation interface, Figure 6.

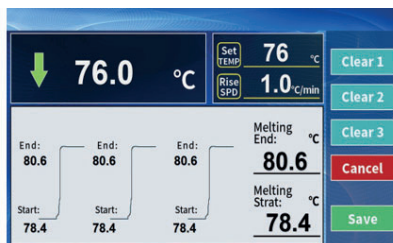


Fig. 6

8) Three capillaries are used in instrument calibration. Because of the difference of capillary sample and filling capacity, there may be a big difference between the calibration values of one capillary and the other two capillaries. At this point, the capillary data with large correction difference can be deleted by pressing the key of “Clear 1” or “Clear 2” or “Clear 3”.

9) The average calibration value of initial and final melting-point will also be updated in real time according to user's operation.

10) If the calibration is good, press the [Save] key to store the data, otherwise press the [Cancel] to abandon the result and recalibrate it.

11) After one point correction is completed and saved, if other calibrations are needed, then re-select other points to calibration. You can also press [Quit Calibrate] to return to normal measurement mode.

12) The new calibration parameters will take effect immediately after returning to the measurement mode.

2. Resume to default setting

When the user is calibrating, if the improper operation causes parameter confusion and cannot be measured normally, the factory calibration parameters can be restored according to the following method:

1. Press [Calibrate] to enter the mode.
2. Press [Load Default], enter the password on the digital keyboard, press the Enter key to confirm.
3. The instrument will derive the factory calibration parameters stored internally and refresh the calibration data.

7. CONNECT WITH COMPUTER

1. Through RS232 or USB cable to connect melting point apparatus and computer.
2. Install the software and operate according to instructions.

8. CAUTIONS

1. Any substance being loaded into a melting point capillary must be fully dry and be crushed into fine power in a dry and clean mortar. With free fall method, tap the capillary tube and make the sample fill in a tight packing. The packing level is 3mm. For the same batch of samples, a fixed level in the fill must be the same to ensure the measurement results are consistent.
2. When the instrument is turned on, the screen shows the current temperature of the oven. Press "Preset" key to preheat the oven to the start temperature. The start temperature shall not be higher than the maximum measurement temperature, 360°C; otherwise, the instrument will be broken.
3. The start temperature of the sample has influence on the result of measurement. Therefore, it's necessary to follow a certain operation procedure. Suggestion: insert the capillary prior to 3 to 5mins. If the linear ramp rate is 1°C/min, the start temperature shall be lower than the melting point for 3°C-5°C. For the ramp rate 3°C/min, it shall be for 9°C-15°C. Generally, it shall be determined according to the optimal testing conditions.
4. The difference of linear ramp rate, the measurement results will not be the same. It's necessary to have a rule. Generally, the higher the rate is, the higher the reading will be. For each rate, the melting point reading can be unified through experiment correction. For the melting point of unknown sample, the initial range can be determined through rapid ramping temperature or higher rate.
5. If there is a reference sample, its melting point can be determined first. According to the selection of start temperature and ramp rate, comparison measurement can be conducted with the onset point of reference sample as the reference. With the melting point of standard sample as temperature transfer standard, the results can be amended according to the reading of the adjacent standard sample.
6. It's better to fill the sample to be tested into 5 capillary tubes. After measurement, reject the max and min value, and use the average value of the middle three values as the measurement result, which can eliminate the accidental error caused by the capillary and during filling the sample.
7. When measuring two samples separately with high and low melting point, test the one with high melting point first, then enter the lower start temperature and the oven will automatically cool down to prepare for the next measurement.
8. For few colored samples with poor melting characteristics or completely carbonized samples, plastic slice samples and trace samples, microscopic analysis instrument can be used for measurement. For polymer products, softening point tester can be used.
9. Before the capillary tube is inserted into the instrument, it shall be wiped clean with soft cloth. Otherwise, dust from dirty tubes can slowly accumulate on the heating stand and influence the measurement or fail to work.

9. MAINTENANCE AND CHECK

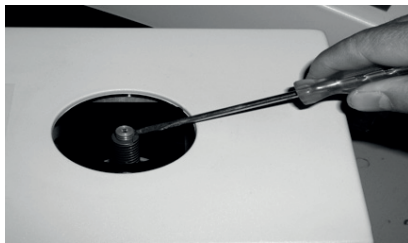


Fig. 7

1. The instrument shall be operated in dry and ventilated room, away from water sources to avoid damp. The instrument uses three-pin plug, the ground line shall be connected to the earth and shall not be replaced by common midline.
2. The capillary tubes to be used must be the ones supplied by the instrument manufacturer, which have been carefully selected to avoid too tight to break. Please don't use handmade capillaries.
3. If a capillary tube is broken in the holder, shut down the power first. After the oven is cooled down, follow the illustration in Figure 7. Dismantle the capillary holder from the oven and remove the glass scrap with a needle (accessory). Then put the holder back in the oven. When inserting the holder, make sure the holder is fastened with the oven. Finally, check the instrument to see whether it works normally. Contact the supplier Technical Service in case of any unfamiliar situation.
4. Accuracy test: The instrument selects three substances: Naphthalene (clear point 80.6°C), Adipic acid (clear point 152.89°C), Anthraquinone (clear point 285.7°C), ramp rate is 1.0°C/min. The start temperature is lower than clear point by 5°C. According to the testing requirements, measure five times, discard the maximum and minimum values, and calculate the average of the rest three values. The offset shall be less than the specified accuracy.
5. Reproducibility test: for the same sample preparation, with the ramp rate 0.2°C/min or 1.0°C/min, test the standard substance Benzoic acid for five times. Calculate the standard offset with the following equation:

$$S = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}}$$

Where:

x_i : value from each measurement (°C)

$\bar{x}$: average value (°C)

n : measurement times (=5)

10. COMMON FAILURES AND REMEDIES

Failure	Possible reasons	Remedies
No display when it's turned on	1- Not connect with power. 2- The fuse is broken. 3- Others.	1- Get connected with the power and make sure it's well connected. 2- Replace with the same type of use. 3- Send back to supplier for repair.
Incorrect indication	1- The parameter settings changed. 2- The temperature change of environment is too big.	1- Calibrate the instrument in accordance with the calibration procedures.
Failure communication with PC	1- The connection wire is broken. 2- The communication program is wrong.	1- Check the connection wire. 2- Contact the supplier.
Poor reproducibility	1- The fill level is not consistent. 2- The size of capillary is not consistent. 3- The purity of sample is poor.	1- Follow the sample filling rule. 2- Select the right capillary. 3- Use high purity sample and make sure the instrument works normally.