Laboratory Equipment Manufacturer
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INE-WRS-1C

DIGITAL MELTING POIINT APPARATUS

OPERATING INSTRUCTION

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Contents

I. 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

INE-WRS-1C melting point apparatus adopts automatic photoelectric detection, dot-matrix graphic 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 provided by our company. The instrument uses the capillary which is in compliance with the provision of pharmacopoeia as sample tube.

II. Specifications and Main Technical Parameters

1. Temperature range:	Ambient□ 400℃
2. Measurement method□	Automatic
3. Display min. indication□	0.1℃
4. Linear temp rates□	Between 0.1℃/min and 20.0℃/min
5. Temperature accuracy□	Up to 200℃□ ± 0.4℃ Above 200℃□ ± 0.7℃
6. Repeatability□	0. (Linear temp rate: 1℃/min)
7. Measurement modes□	8
8. Standard capillary dimensions□	Φ 1.4mm outside diam, Φ 1.0mm inside diam, 90mm lengths
9. Fill height□	3mm
10. Storage□	1000 sets
11. Interface□	USB or RS232
12. Power□	220V± 22V□ 50Hz□ 100W
13. Dimensions (L×W×H) □	360 mm× 290mm× 170mm
14. Weight□	8kg

III. 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 it is melted state, it transmits light. Therefore, in the process of melting, a transition of the transmissivity will occur with the rise of the temperature.

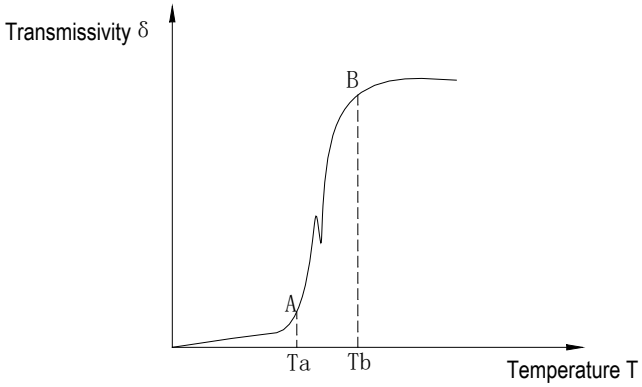


Fig.1

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

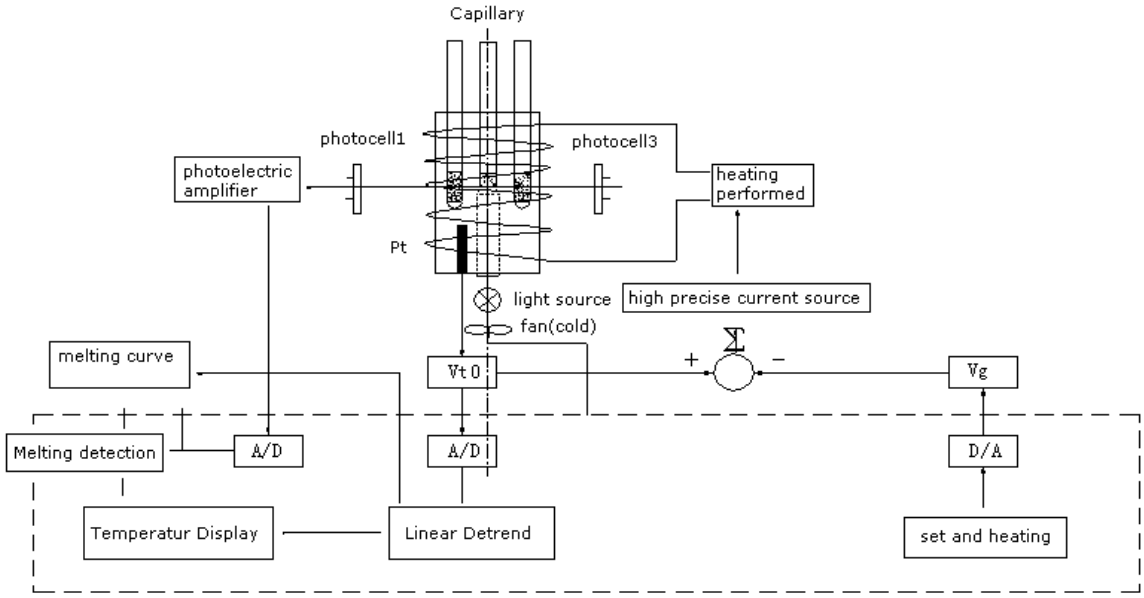


Figure 2

IV. Instrument Overview

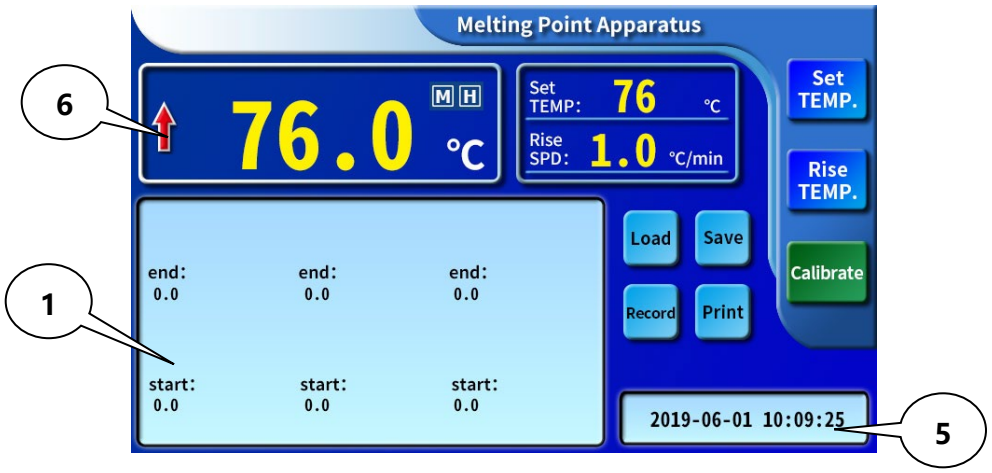
i. Front view of the instrument

ii. Back view of the instrument

V. Operations

INE-WRS-1C 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 rate from 0.1℃/min to 20℃/min.

i. Turn on the machine



1. Instructions of display

1. Showing the melting curves of the last three measurements.	2. Temperature preset and heating rate setting. Click on the number section to set the required parameters.
3. Real-time furnace 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

【 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□□	Calibrate the temperature□ **Operate cautiously!**□ .
□ Record□□	Show stored records, maximum 1000 sets.
□ Methods Load□□	8 measure methods can be set and stored (Preset temperature and heating rate).
□ Methods Save□□	Save the methods that is set.
□ Print□□	Print the latest measurements and melting curves.

ii. 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 is 400 ℃, otherwise the input is invalid.**

4. Click on “Set TEMP” to start preheating.

iii. Rise the temperature

1. When the temperature of the instrument reaches the preset temperature and is stable for 30 seconds, the device will beep that indicating the preset is completed. At the same time, [Rise TEMP] will appear to perform the next operation.

2. Click on [Rise SPD] 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.

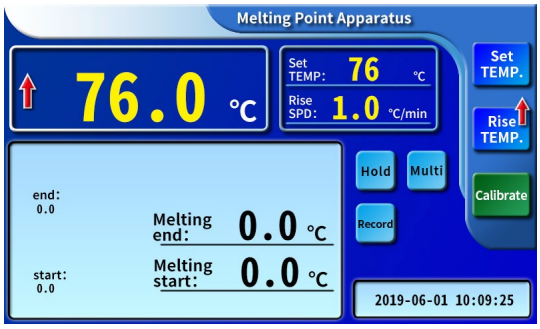


Figure 3

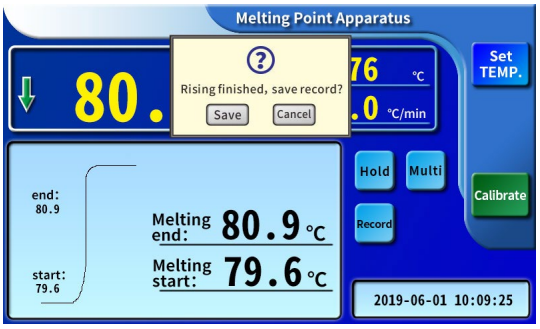


Figure 4

4. <u>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.</u>

5. <u>During the heating process, you can press [Set TEMP] to exit the heating at any time.</u>

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. Figure 4.

7. Press the [Hold] button, 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. Users can press the [Hold] button again to exit.

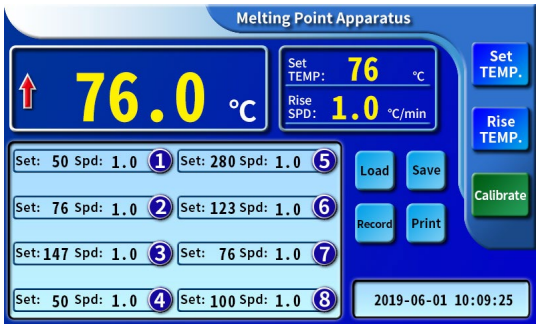
8. Press the [Multi] button, the letter "M" is displayed in the temperature window, and the instrument will be in the multi-melting point measurement mode. When the instrument detects the first final melting-point, it will not stop measuring, but will continue heating until the second final melting-point is detected. It can be used to measure special samples. Users can press the [Multi] 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] button to print out the measured data and melting curve (The printer is optional).

iv. 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.



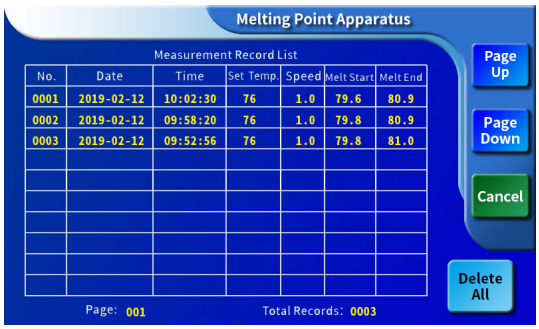
1. Load： Press the [Load] button to display the right-hand interface, click the number 1-8 to test with the required measurement mode.

2. Save： Press the [Save] button 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 [Load] or [Save] key again to exit.

v. Records

Press the [Record] button 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.

vi. Time setting up

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

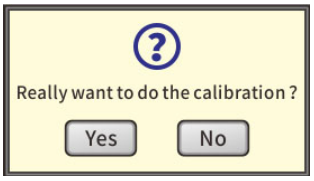
Enter the time in order of "year-month-day-hour-minute-second" (in this order necessarily). For example, to get the time “2018-09-01 12:30:00”, input the number “180901123000” in order.

VI. 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 parts 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 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.**

i. Calibrate the temperature

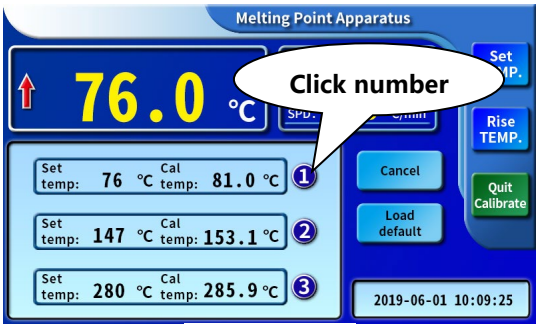
If the measurement accuracy of the sample is not very accurate, the instrument can be calibrated with standard materials. Users can use one-point correction or multi-point correction according to their needs.



1. Press the [Calibration] to indicate whether the temperature really needs to be corrected. Or press the [Calibration] again 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.

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



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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 & press the [Rise TEMP] to heat up.

6. **When calibrating, the heating rate is fixed at 1.0 ℃/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.

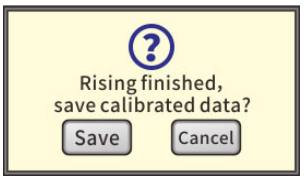

Figure 6

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

9. 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 Calibration] return to normal measurement mode.

10. **The new calibration parameters will take effect immediately after returning to the measurement mode.**

ii. Resume to default setting

1. Press the [Calibration] to enter the mode.

2. Press the [Load Defaults], 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.

VII. Connect with computer

1. Through RS232 or USB cable to connect melting point apparatus and computer.

2. Put the CD into CD-ROM of the compute, install and operate according to instruction.

VIII. 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 max measurement temperature, 400℃. 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 following a certain operation procedures. Suggestion: insert the capillary prior to 3 to 5mins. If the linear ramp rate is 1℃／min, the start temperature shall be lower than the melting point for 3℃□5℃. For the ramp rate 3℃□ min, it shall be for 9℃□15℃. Generally, it shall be determined according to the optimal testing conditions.

4. The difference of linear ramp rate, the measurement results will not 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, INE-WRX-1S microscopic analysis instrument manufactured by our factory can be used for measurement. For polymer products such as polymer, INE-WQD-1A softening point tester produced by our factory can be sued.

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.

IX. Maintenance and check

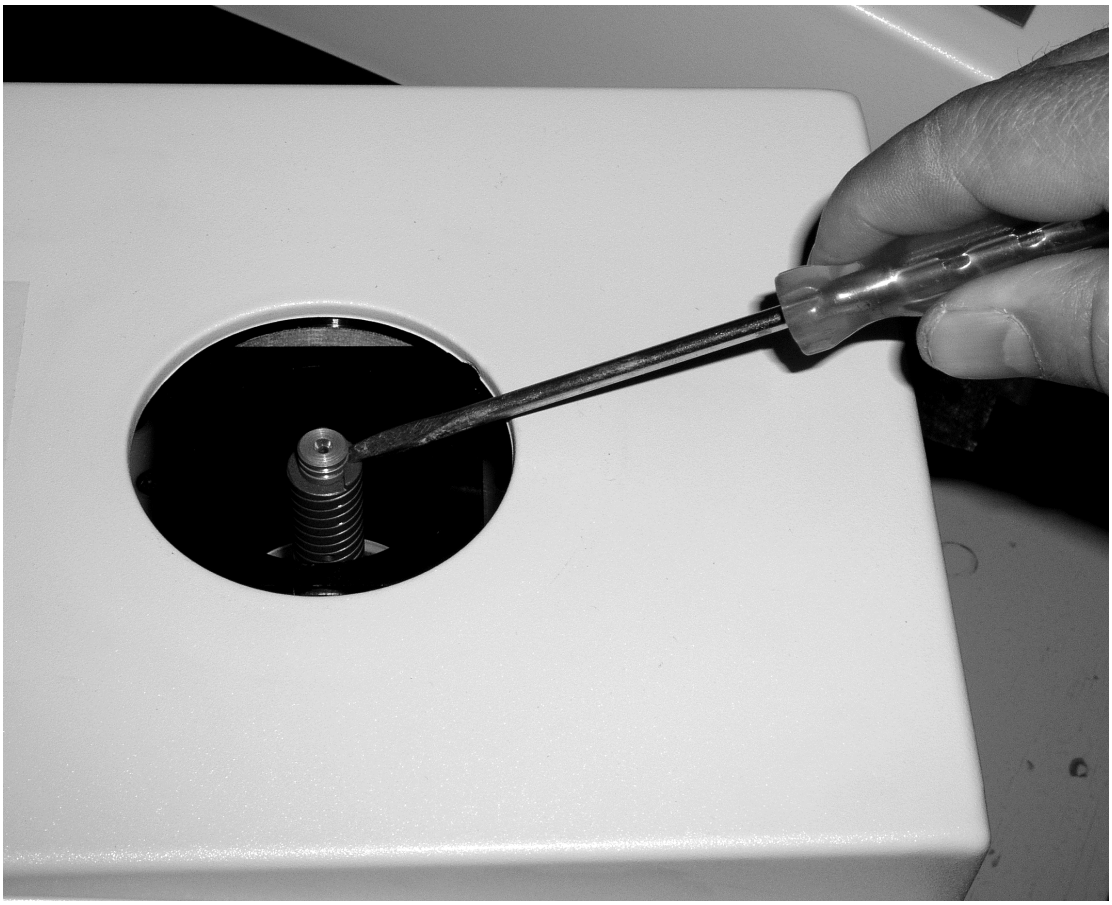

Figure 7

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 tube can only accept the ones produced by our factory, which have been carefully selected and can avoid too tight to break. Please don't use handmade capillary as a replacement.

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 insert the holder, make sure the holder is fastened with the oven. Finally, check the instrument whether it works normally. Contact the technical support department of our factory in case of any unfamiliar situations.

4. Acceptability test: In accordance with the melting point of GBW13238 standard substance approved by National Bureau of Technical Supervision in 1989 (File No. 335). The instrument select three substances: Naphthalene (clear point 80.6℃), Adipic acid (clear point 152.89℃), Anthraquinone (clear point 285.7℃), ramp rate is 1.0℃／min. The start temperature is lower than clear point by 5℃. According to the testing requirements, measure five times, discard the max and min value, and calculate the average of the rest three values. The offset shall be less than the specified accuracy. (The standard substance can be purchased from our factory.)

X. Common breakdown and handing

Failure phenomenon	**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 factory 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 factory.
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.