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# Operation Instructions for INE-WRS-1B Digital Melting Point Apparatus



**PLEASE READ THIS MANUAL CAREFULLY BEFORE OPERATION**

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# 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 the chemical industry and the medical research, and it's a must-have instrument in the manufacturing of medicine, spices, dyes and other organic crystal substances

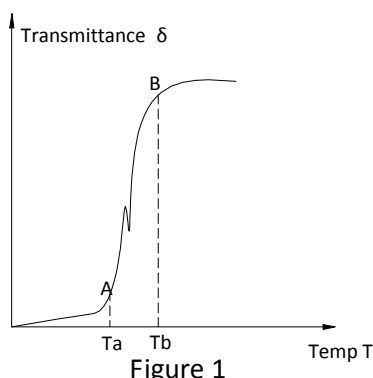
The INE-WRS-1B digital melting point apparatus applies a "humane" design concept, adopting photoelectric detection, dot-matrix liquid crystal display, and digital keyboard input. The instrument can measure one standard sample at a time, and measure three times to calculate the melting point average automatically. It can display the initial and finish melting and record the melting curve automatically. The temperature system applies the high linearity platinum resistance as the detection element, using PID control technology to improve the accuracy and reliability of the melting point. Besides, it employs the integrated electronic circuits to achieve fast "initial temperature" setup and has eight rates to choose from the linear "ramp rate" automatic control. The instrument establishes communications via USB with the PC, and can save and print graphs. The sample tube adopts the capillary as prescribed in the pharmacopoeias.

## II. Technical Specifications

Temperature range:	Ambient ~320°C
"Start temperature" setup time:	50°C ~ 320°C: not greater than 4min 320°C ~ 50°C: not greater than 6min
"Start temperature" accuracy:	±0.4°C
Temperature resolution:	0.1°C
Linear "ramp rate":	0.2°C/min, 0.5°C/min, 1°C/min, 1.5°C/min, 2°C/min, 3°C/min, 4°C/min, and 5°C/min (eight rate selections in total)
Linear "ramp rate" accuracy:	Not greater than 10% of the setup value
Temperature accuracy	Less than 200°C: ±0.4°C 200°C ~ 320°C: ±0.7°C
Reproducibility:	0.3°C when "ramp rate" is 1.0°C/min
Capillary dimensions:	Φ1.4mm outside dia., Φ1.0mm inside dia., 80mm lengths.
Fill height:	>3mm
Communication interface:	USB and RS232 serial interface (9600baud, 1 stop bit, 8 data bits)
Electrical supply:	220V±22V, 100W, 50Hz
Dimensions (h ×d×w):	365mm×290mm×176mm
Weight:	10Kg

### III. Working Principle

The working principle of the instrument is based on the following fact: substances in the crystalline state reflect light, and transmit light in the liquid state. Thus, during the melting process, as the temperature increases, the transmittance of the substance will jump. Figure 1 is a typical melting curve. The temperature  $T_a$  corresponding to the point A is called the initial melting point; the temperature  $T_b$  corresponding to point B is called the final melting point (or full melting point); AB is called the melting range, i.e., melting interval or melting range.



The instrument uses an optical mode to automatically detect changes in the melting curve. When the temperature reaches the initial melting point and final melting point, the instrument shows the temperature of the initial and final melting point, and stores to detect the next sample.

The principle of the instrument is shown in Figure 2. The light from LED lamp goes through the optical fiber, an electric oven and the aperture on the capillary holder to converges in the capillary. The light transmitted through the melted sample is received by a silicon photocell. When the temperature rises, during the melting process, the flux of the sample becomes larger. The computer will record and display the melting curve and the temperature of the initial and final melting points.

Temperature detection uses the platinum resistance which is directly inserted into the bottom of the capillary tube holder as the probe. The resulting temperature signal is amplified by voltage and sent to A/D converter. After having been calculated by the software, the temperature is displayed. The corresponding "ramp rate" can be obtained through the keyboard input. Enter "ramp rate", and the temperature analog voltage generated from the D/A converter and the temperature unit is sent to the adder. The output of the error signal will be driven and heated by the regulator.

When the electric oven's actual temperature is higher than the temperature of the analog D/A converter, electric oven will cool down. When the actual temperature is below the temperature of the analog D/A converter or fails to reach the set "initial temperature", increase the heating current. Through such close-loop system and software-driven temperature calibration to empower the electric oven to achieve the follow-up function, as well as to eliminates the effect of seasonal temperature on the preset temperature.

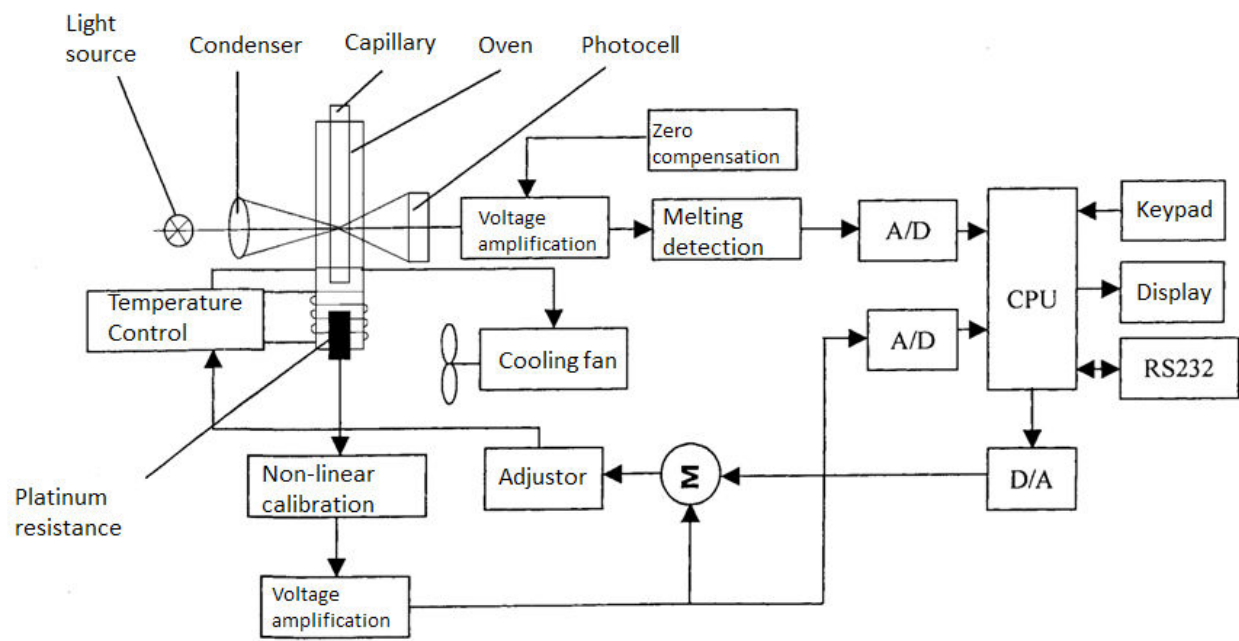


Figure 2

## IV. Instrument Overview

### 1. Front Panel Overview (See Figure 3)

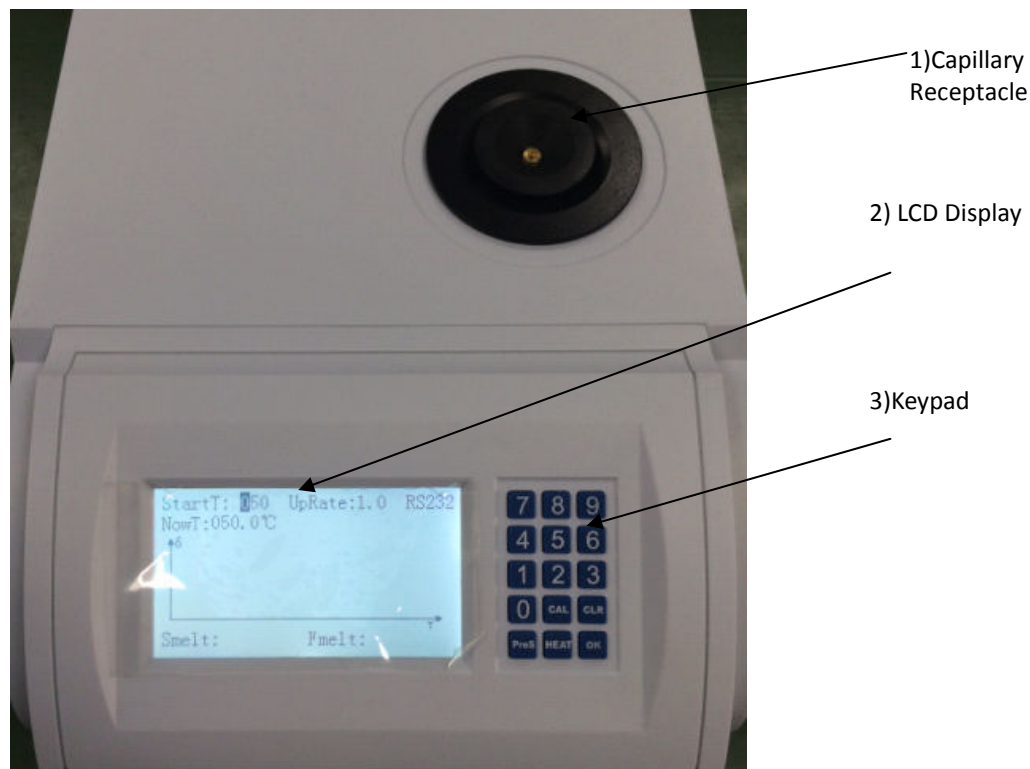


Figure 3

### 2. LCD Display (See Figure 4)

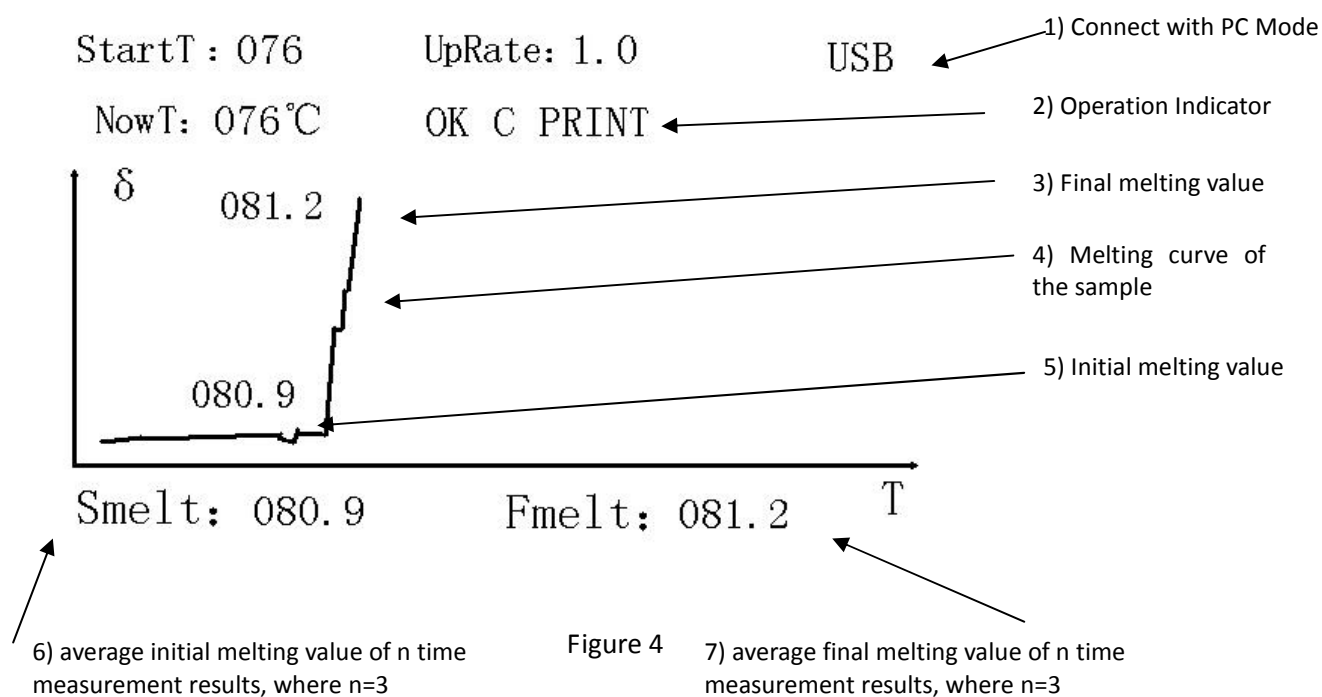


Figure 4

Note: Operation indicators remarks:

- 1) "E 1 2 3" means the user press **Heat** without the capillary on the holder or "Start Temperature" is too high, the powder is melted down when just put in.
- 2) **↑** means heating-up; **↓** means cooling-down.
- 3) "OK C print" means sample test is finished, and you can decide whether to print the result; To print, click **OK**, or to cancel, click **Clear**.
- 4) "Calibration" means starting calibration;
  - a) "OK C" means: during calibration, the measurement is valid (three final melting values are close), press **OK**; or the measurement is invalid (individual final melting value is too dispersed), press **Clear**.
  - b) "A" means the instrument is running calibration now.
- 5) "1 2 3 C" means after test, press **Clear**: 1, 2 and 3 refers to the serial number of the curve to be cleared, the left curve is 1, the middle one is 2, and the left is 3. To cancel clear operation, press **Clear**.

### 3. Keypad (See Figure 5)

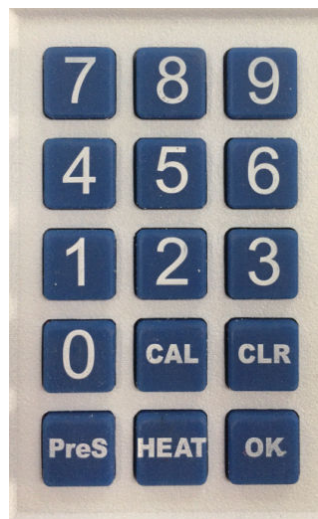


Figure 5

### 4. Back Panel Overview (See Figure 6)



Figure 6



## V. Operation Instructions

1. Turn on the power switch, the display shows "start temperature", "ramp rate" and "communication", during this time, the instrument is preheating; If the default settings are the last parameters, simply press the "**Preset**" key to enter the preset state; If you need to set new parameters, use the keypad to input.
2. The initial cursor will stop at the first digit of "start temperature". Modify the "start temperature" with keypad. Press "**OK**" key when the "start temperature" is successfully set; or press the "**OK**" to apply the default current "start temperature". Move the cursor to the first digit of "ramp rate", press "**Preset**" key to enter the preset state, and the cursor will go back to the first digit of the "start temperature".
3. Modify the "ramp rate" with keypad. Press the "**OK**" key when the "ramp rate" is set successfully; or press "**OK**" key to apply the default current "ramp rate". Move the cursor to "start temperature" settings, press the "**Preset**" key to enter the preset state, the cursor back to the first digit of "start temperature".  
**Note:** If the entered "ramp rate" value is not among the eight options, the system will use 1.0°C/min by default.
4. Using keypad to input is to change the digit where the cursor stops, and then the cursor moves to the next digit. When you change the last digit, the cursor returns to the first digit, in this way, each digit can be modified.
5. When the actual oven temperature remains at the preset temperature  $\pm 0.2$  and the buzzer is finished alarming (prompt preset must be finished before heating up), you can insert the sample capillary, press the "Ramp" key to start heating (the instrument INE-WRS-1B allows the user to put one capillary).  
**Note:** If the actual oven temperature doesn't remains at the preset temperature  $\pm 0.2$ , it doesn't work to press the temperature key.
6. Press "**Heat**" key, and the operation indicator displays "↑". At this time the instrument will follow the preset parameters to measure the samples.  
**Note:** Press "**Heat**" key, if the oven has no capillaries, the display will appear E123 alarm without showing "↑".
7. When reaching the initial melting point, the display shows the initial melting temperature; when reaching the final melting point, the final melting temperature will be shown, as well as the melting curves. (See Figure 4 for specific data meaning).
8. The instrument can only measure one sample at a time, and save up to three samples' test data. It will automatically calculate the average value of the initial and final melting temperature.
9. After each sample test, the instrument operation indicates "OK C Print", waiting for the user to decide to print or not. Press "**OK**" key to execute print, or "**Clear**" key to cancel printing. If the instrument is not connected to a printer, after pressing "**OK**", the buzzer will ring for four seconds, suggesting that the printer is not connected. If the printer is reconnected, press "**OK**" to print.

10. When the sample test is completed and printing is performed, if new samples are tested, last curves and data will be cleared automatically.
11. As long as the power is not cut off, the above readings will remain in the instrument.  
**Note:** The quality of sample loading and consistency will directly affect the accuracy of the readings, the "poor sample loading" could cause the melting curves to appear trough or discontinuous over long distance. Now, the measured values serve for reference only.
12. If you want to measure a new sample, enter "start temperature" and press **"OK"** key, the original curve will be cleared and the new measurement will start.
13. **"Clear"** key: when you finish testing a sample, the display will show the melting curves corresponding to three capillaries. If there is any curve discontinuous over long distance or the measurement error is too big, due to poor sample loading, you can clear the curve with the following steps:
  - 1) Press **"Clear"** key, the operation indicator shows "2 3 C";
  - 2) Press the corresponding number key to clear the curve, or press "Clear" key again to cancel the operation.**Note:**
  - a) From left to right, the serial number of curve is 1,2 and 3. Press the number key "1" to clear Curve 1 (i.e., the left curve), and so on;
  - b) As one curve is cleared, the average melting point will change, i.e., the cleared curve will not be included in the average calculation.
  - c) Re-test the sample and a new curve will be generated in the empty position, and the instrument will automatically calculate the average of the initial and final melting temperature.
14. **"Calibration"** key:
  - 14.1 Use of Calibration key:
    - 1) When the instrument has been working for a long time, or the measurement error is too big due to the seasonal temperature difference, you can calibrate the instrument to ensure the measurement accuracy;
    - 2) When the instrument is shipped from the factory, it has been calibrated with our standard samples including naphthalene (81.0°C), adipic acid (153.1°C), and quinone anthracene (285.7°C). If you want to use your own standard sample, it can also be calibrated.**Note: If you do not encounter either of the above case, please do not perform calibration! ! !**

#### 14.2 Calibration Steps:

- 1) Press "Calibration" key, and the instrument shows calibration page (Figure 8);

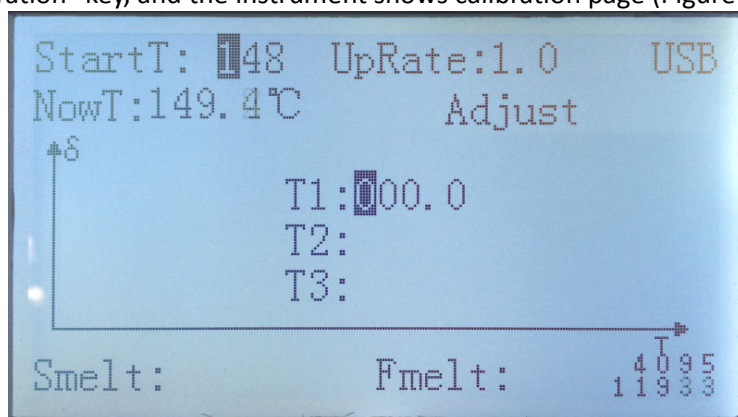


Figure 8

- 2) The user can press the corresponding key according to the operation indicator (it displays "calibration");
  - a) Press the "0" key or press the "Calibration" key again, which means exiting calibration.
  - b) Press the "1" key, which means re-calibrating the parameters, and the instrument will show the temperature input page (Figure 9):

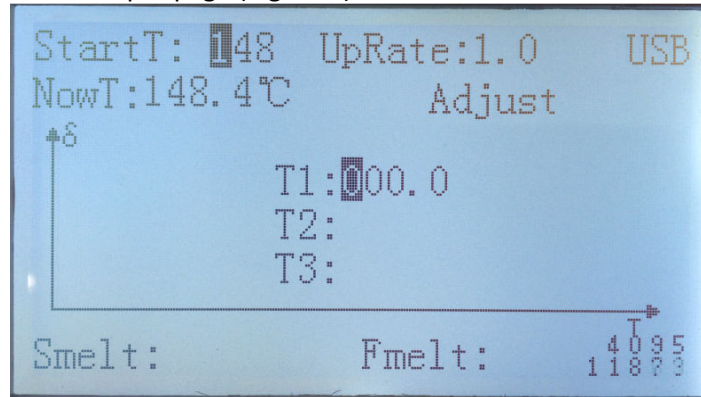


Figure 9

**Temp 1, Temp 2, and Temp 3** refer to the final melting temperature of the standard sample, can perform the **first order calibration, second order calibration** and **third order calibration**.

- a) In **Temp 1**, enter the final melting value, press the "OK" key, and the cursor jumps to **Temp 2**; In **Temp 2**, enter the final melting value, press the "OK" key, and the cursor jumps Temp 3; In **Temp 3**, enter the final melting value, and press the "OK" key. Now, one point third order calibration setup is completed, and start the calibration.
- b) In **Temp 1**, enter the final melting value, press the "OK" key, and the cursor jumps to **Temp 2**; In **Temp 2**, enter the final melting value, press the "OK" key, and the cursor jumps Temp 3; In **Temp 3**, don't enter the final melting value, and press the "OK" key directly. Now, one point second order calibration setup is completed, and start the calibration.
- c) In **Temp 1**, enter the final melting value, press the "OK" key, and the cursor jumps to **Temp 2**; In **Temp 2** and **Temp 3**, don't enter the final melting value, and press the "OK" key directly. Now, one point first order calibration setup is completed, and start the calibration.

- 3) The first point third order calibration (See Figure 10):

If the user uses one standard sample to perform third order calibration, enter the final melting temperature of the sample in the corresponding position, e.g., in Temp 1, 2, and 3, enter 081.0°C, then press "OK" key.

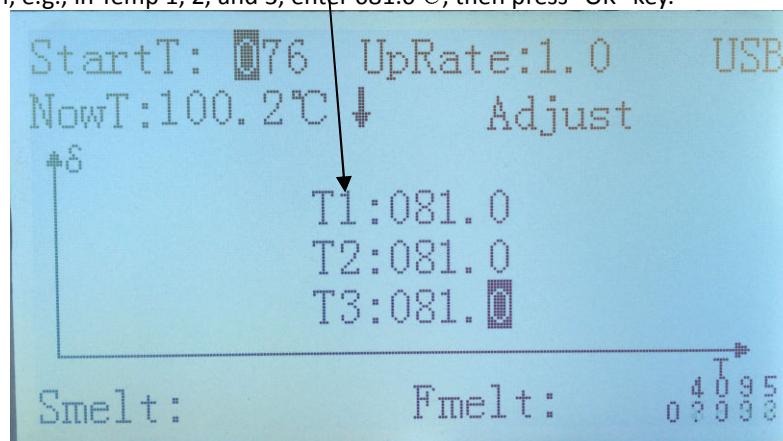


Figure 10

4) The second point third order calibration (See Figure 11):

If the user uses one standard sample to perform third order calibration, enter the final melting temperature of the sample in the corresponding position, e.g., in Temp 1, 2, and 3, enter 153.1°C, then press "OK" key.

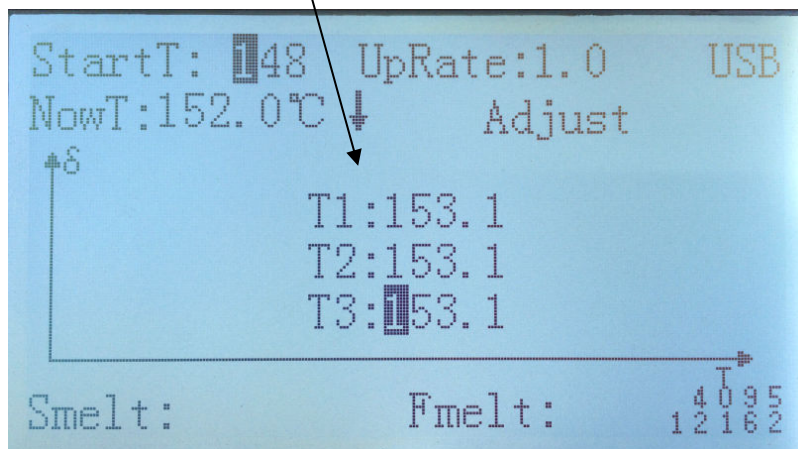


Figure 11

5) The third point third order calibration (See Figure 12):

If the user uses one standard sample to perform third order calibration, enter the final melting temperature of the sample in the corresponding position, e.g., in Temp 1, 2, and 3, enter 285.7°C, then press "OK" key.

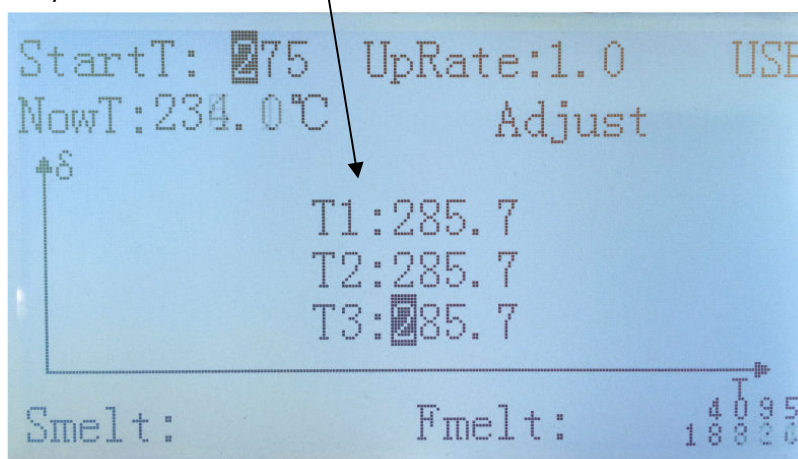


Figure 12

The user can set the final melting temperature of the standard sample according to the actual situation. After entering the value in Temp 3, press "OK" to start calibration. The instrument will clear the temperature entry page to return to the measurement page. Then follow the above measurement procedures to test standard samples.

**Note:** The user can decide the number of points and times of order for calibration. If enter "0" to temperature, it means this point doesn't need to calibrate. During the process of setting temperature, press "Calibrate" key to exit the function.

14.3."OK C" selection:

- 1) As finishing one standard sample measurement, the operation indicator will display "OK C". The user can determine whether this measurement is valid or not; Press "OK" key to confirm it's valid, or press "Clear" key if it's invalid.
- 2) If you think the measurement error is too large (melting range is not normal) and want to measure the sample again, you can only choose C and press the "Clear" key. (See Figure 13 and

Figure 14)

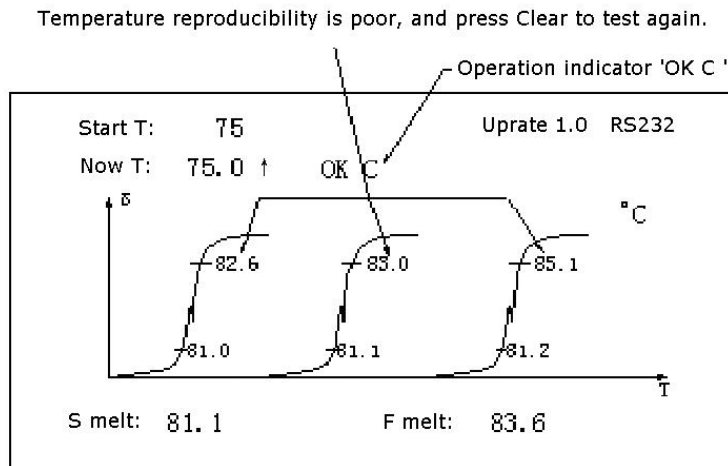


Figure 13

The measurement data are discontinuous and cause the melting range too long, exceeding the normal range. The melting range of Na is less than 1.5 °C. It should be cleared and measured again.

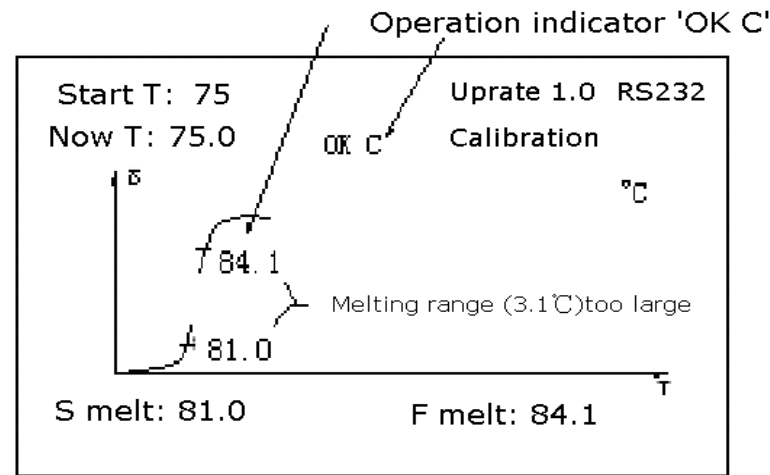


Figure 14

- 3) If the melting range of three curves are close, i.e., the reproducibility is good, the measurement can be deemed valid. (See Figure 15)

The final melting value exceeds the error ( $81.0 \pm 0.4^\circ\text{C}$ ), but reproducibility is fine. Press "Ok" key.

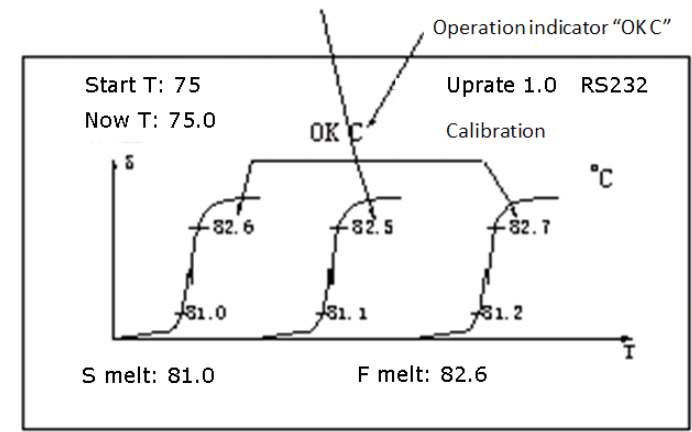


Figure 15

Note:

- a) During calibration, the accuracy of the final melting point may be ignored, just focus on the reproducibility of the sample. As long as the reproducibility of the sample is good, and for the melting point which is not in the allowable range, the measurement is also considered effective. To confirm, press the "OK" key;
  - b) Select OK and C is very critical to the success of calibration, which might lead to that the instrument does not work. If you take this measurement as effective and press the "OK" key, the sample cannot be measured again, and should proceed to measure next standard sample; only the user take this measurement as invalid and press the "Clear" key, before the sample is measured again until the measured effective and press the "OK" key is pressed.
- 4) The number of final melting temperatures have been entered should be consistent to the number of standard samples to be measured. When the last standard sample is regarded as valid, press "OK" key. The operation indicator displays "A", which means the instrument is under calibration status. When "A" disappeared, it means the calibration is finished (See Figure 16), and you can start with sample detection.

When the last standard sample is finished with testing, press "OK" key, the indication "A" shows for one second and then will disappear. When "Calibration" also disappears, it means the point is finished with calibration.

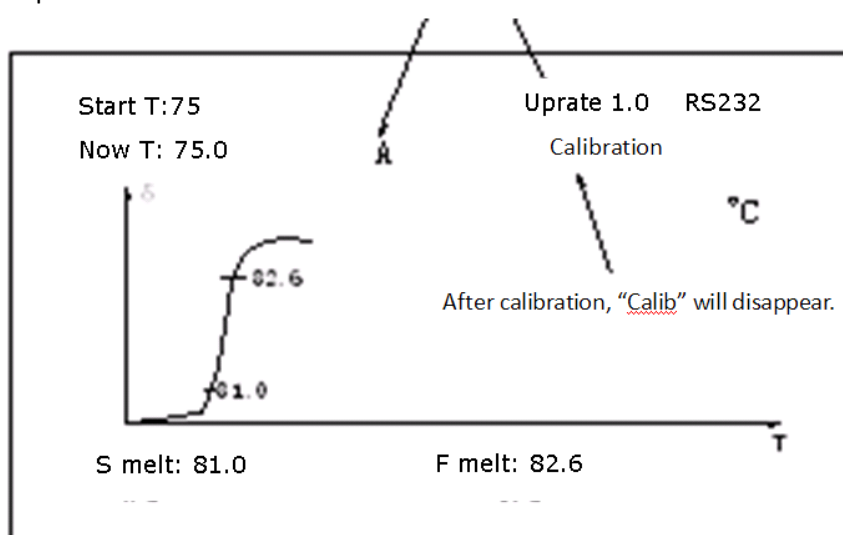


Figure 16

Note:

If the default "preset temperature" is lower than the standard melting point by 5°C, the "ramp rate" must be set as 1°C/min. If the default "preset temperature" is not suitable, you can set the "preset temperature" during calibration.

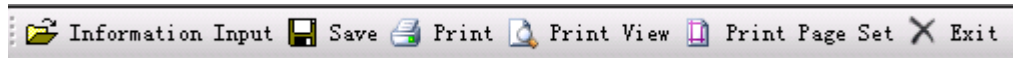
#### 15. Communication with PC

- 1) With USB cable to connect melting point apparatus and computer.
- 2) Put CD into the CD-ROM of the computer to install USB drive before using USB to communicate.
- 3) The computer runs Windows XP program (see details in Readme.txt).



## VI. PC Operation

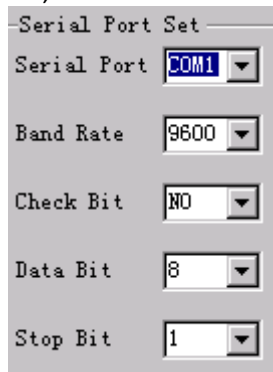
### 1. Toolbar Menu



- 1) **"Information Input"**: input the sample type, tester and test time.
- 2) **"Save"**: save the curve of the sample to the computer as jpg format;
- 3) **"Print"**: print the curve of the sample;
- 4) **"Print view"**: see how the file looks like when it were printed out;
- 5) **"Print Page Set"**: set the print pages, such as horizontal or vertical print;
- 6) **"Exit"**: shut down the software on PC.

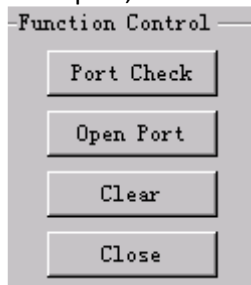
### 2. Serial Port Settings

The default parameters cannot be configured, and the COM port can be selected from the drop-down list, as shown below:



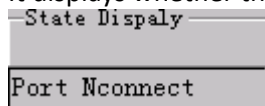
### 3. Function Control

**Serial detection** can automatically identify which serial port is available on your computer. You can turn on or off the port, clear the last curve, or shut down the software, as shown below



### 4. Status

It displays whether the serial port connection is successful, as shown below:



### 5. Operation Steps

Step 1, click **operation control** → **serial detection**, as shown below;

Step 2, select COM, **Serial Port Settings** → **COM**, as shown below;

Step 3, click **Open Serial Port** key:

If the status shows "**Serial port is not connected**", it means the COM is not correctly selected. Go back to Step 2 and select COM again.

If the status shows "**USB connection is successful**", it means the COM is correctly selected, and the connection is successful, as shown below.

Step 4, Successful Connection, the software will display the current oven temperature, start temperature and ramp rate.



## VII. 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, it automatically preset as the last "start temperature". If the oven is higher or lower than the temperature, it can be set with keypad.
3. The start temperature shall be set within 300°C. The instrument has self-protection function. The max start temperature is 300°C.
4. 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°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.
5. 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.
6. 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.
7. It's better to fill the sample to be tested into 5 capillary tubes, 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 sample loading.
8. 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.
9. 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.
10. 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.

## VIII. Maintenance and Check

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.
3. If a capillary tube is broken in the holder, shut down the power first. After the oven is cooled down, 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. If the indication shows "E123" during testing samples, following are the possible reasons: 1) "initial temperature" are too high and the sample power melts down too quickly; 2) the capillary tube is empty without sample power to be tested.
5. **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°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 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. )
6. **Reproducibility test:** for the same sample preparation, with the ramp rate 1.0°C / min, test the standard substance Benzoic acid for five times. Calculate the standard offset with the following equitation:

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

Where:

Xi: value from each measurement

X: average value

n: measurement times (=5)

7. **Indicator Calibration:** See Page 6 for the Operation of "Calibration" key.  
**Note to user:** (1) If no significant changes in the final melting point of standard samples are found during the periodic inspection, it's generally unnecessary to perform indicator calibration. (2) When the precision test is conducted during calibration, the sample must be handled very seriously and sample loading shall be paid special attention. Otherwise, it is difficult to control the calibration value.

## **IX. Complete set of Instrument**

(see the packing list)

## X. Malfunction and Troubleshooting

Failure phenomenon	Possible reasons	Remedies
No display when it's turned on	Not connect with power; The fuse is broken; Others.	Get connected with the power and make sure it's well connected. Replace with the same type of fuse. Send back to factory for repair.
Incorrect indication	1. Component aging. 2. The ambient temperature changes too dramatic.	1. Recalibration (See Page 7 for details)
Failure communication with PC	1. Whether USB drive is correctly installed; 2. Whether USB cable is not broken; 3. Whether USB cable and PC is well connected; 4. Whether the software is installed on computer.	1. Check the connection wire; 2. Disconnect all connections and connect again; 3. Contact the factory.
The instrument cannot work properly after performing calibration.	Consequences of incorrect parameters caused by calibration failure.	Read the user's manual carefully, and perform calibration again.

## **XI. After Sales Service and Guarantee**

1. We follow three guarantees for product(s): "repair, replace or return", i.e., repair or replace the defective products, or refund the original purchase price.
2. We provide twelve-month warranty from the date of invoice for product(s).