广东纽格尔仪器有限公司
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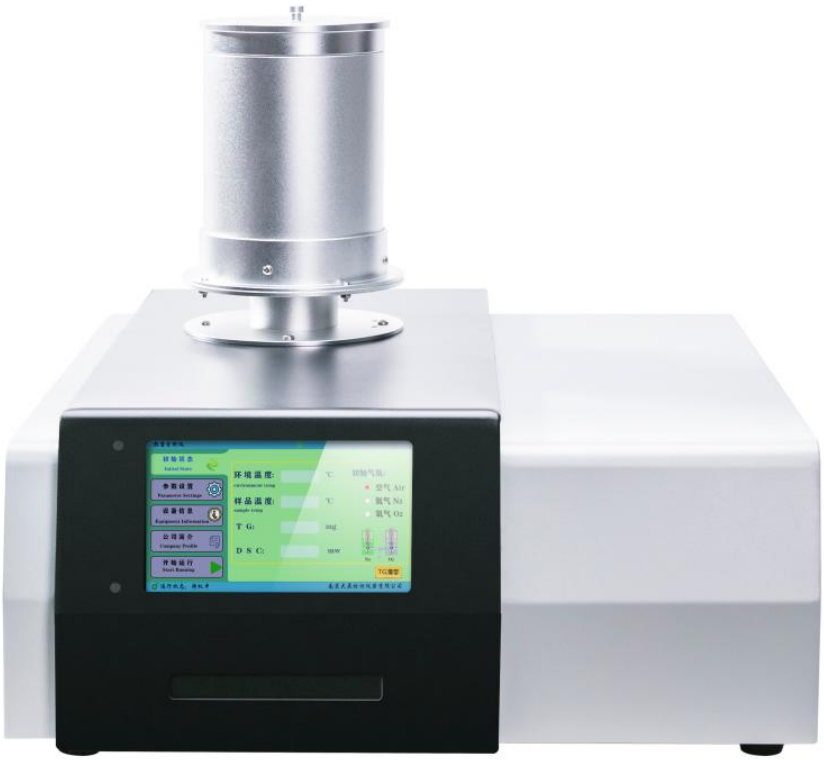
Operation Manual of

Differential Thermal Analyzer



Read before Operation

Before operation this machine, please read this manual thoroughly, and keep a copy with the machine for future reference. This manual is to be considered part of your machine.

Catalogue

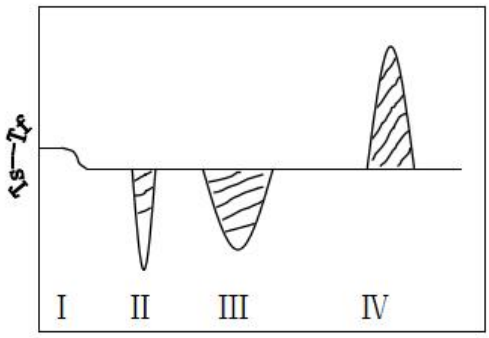
1. Brief introduction of instrument

DTA has been widely used. Differential scanning calorimeter is not only a routine quality testing tool but also a research tool. The relationship between temperature and heat flow related to the internal thermal transformation of materials is measured. Our instrument is a heat flow differential scanning calorimeter with good repeatability and high accuracy.Especially suitable for accurate measurement of specific heat. The equipment is easy to calibrate, easy to use, fast and reliable, and widely used, especially in material research and development, performance testing and quality control. The characteristics of the material, such as glass transition temperature, cold crystallization, phase transition, melting, crystallization, product stability, curing/crosslinking, oxidation induction period, etc.It is the research field of differential scanning calorimeter. Our company has many types of differential scanning calorimeters, and customers choose different models according to experimental parameters and experimental requirements.

The application range of differential scanning calorimeter includes: curing reaction temperature and thermal effect of polymer materials, phase change temperature and thermal effect measurement of materials, crystallization, melting temperature and thermal effect measurement of polymer materials, glass transition temperature of polymer materials, etc. Different types of instruments, testing different indicators.

Put the sample and reference into the crucible respectively, and put them in the furnace for programmed heating to change the temperature of the sample and reference. If the heat capacity of the reference substance and the sample is the same, and the sample has no thermal effect, the temperature difference between them is almost "zero", and then a smooth curve is obtained. With the increase of temperature, the sample produced thermal effect, but the reference did not.There is a temperature difference between them, which is shown as a peak in DSC curve. The larger the temperature difference, the larger the peak, and the more times the temperature difference changes, the more the number of peaks. The peak with upward peak is called exothermic peak, and the peak with downward peak is called endothermic peak.

The following figure is a typical DSC curve, showing four types of transitions:



i is a secondary change, which is the change of horizontal baseline.

ii is the endothermic peak, which is caused by the melting or melting transition of the sample.

iii is the endothermic peak, which is caused by the decomposition or cracking reaction of the sample.

Temperature coefficient → the exothermic peak, which is the result of the crystal phase transition of the sample.

2. Instrument principle

The physical and chemical changes of substances are often accompanied by thermal effects. Exothermic and endothermic phenomena reflect the change of enthalpy of substances. Differential scanning calorimeter is to measure the functional relationship of temperature difference between the measured sample and reference material to temperature or time under the same heating condition.

DTA is a technique to measure the relationship between the power difference between the output substance and the reference substance and the temperature under the condition of programmed temperature control. Our instrument is a heat flow differential scanning calorimeter, and the ordinate is the heat flow difference between the sample and the reference in mw. The abscissa is the time (t) or the temperature (t), and it increases from left to right (if it does not meet this requirement, please indicate it).

After the sample and the reference are put into the crucible, the temperature is increased at a certain rate. If the heat capacities of the reference and the sample are approximately the same, the ideal scanning calorimetry diagram can be obtained.

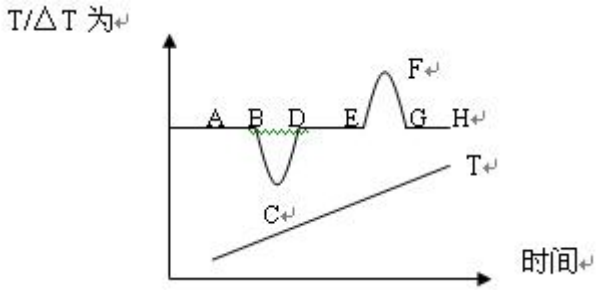


Figure T is the temperature curve reflected by the thermocouple inserted on the reference object. Temperature difference curve between linear reaction sample and reference substance. If there is no thermal effect on the sample, then △T=0 between the sample and the reference, and the baseline as smooth as AB, DE and GH on the curve will appear. When the temperature of the sample is lower than that of the reference due to the thermal effect, an endothermic peak such as BCD peak downward appears.On the contrary, the EFG exothermic peak with upward peak appears.

The number, position, peak area, direction, height, width and symmetry of the peaks in the figure reflect the times of physical and chemical changes, the temperature range in which the transition occurs, the magnitude and positive and negative of the thermal effect of the sample within the measured temperature range.The peak height, width and symmetry are not only related to the test conditions, but also related to the dynamic factors in the process of sample change. The measured results are much more complicated than the ideal curve.

3. Instrument characteristics

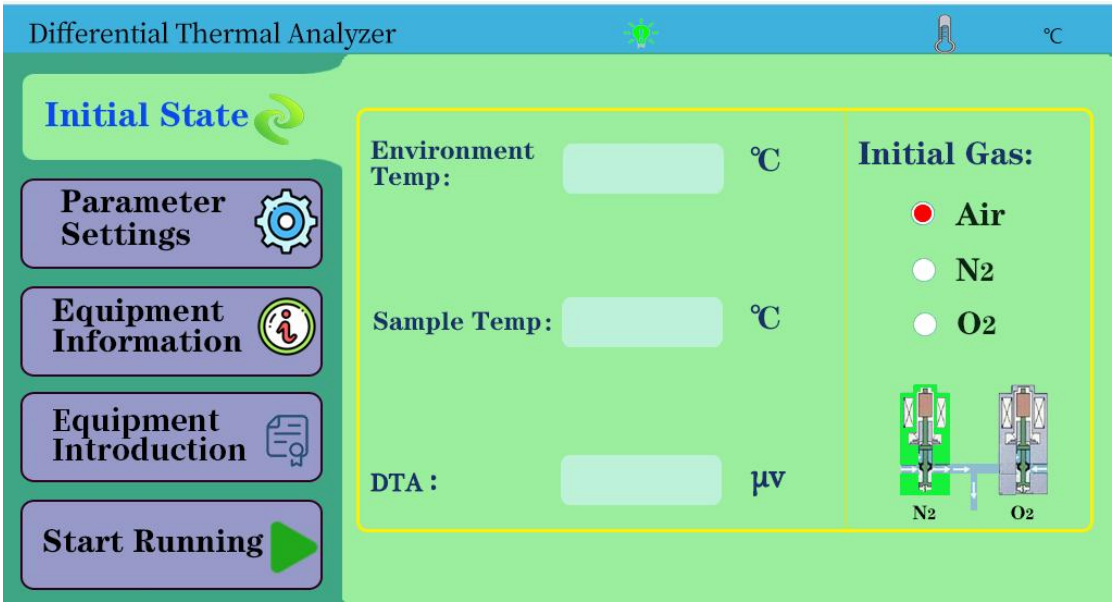
3.1 brand-new furnace structure, better resolution and resolution and baseline stability;

3.2 Real-time data transmission of the lower computer of the instrument, friendly interface and easy operation.

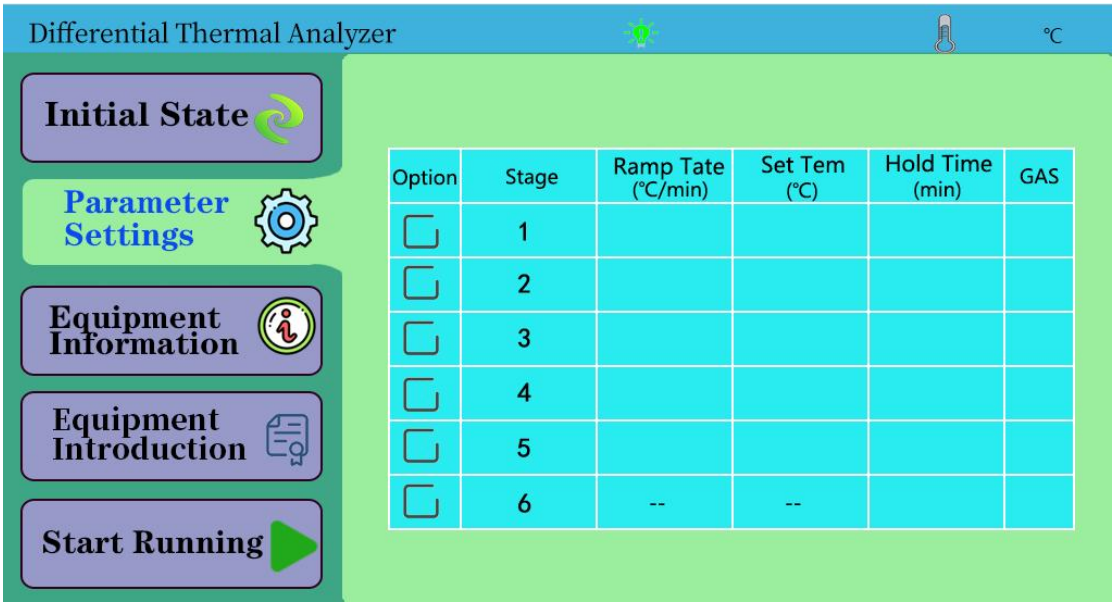
DTA	Differential Thermal Analyzer
DTA range	0～±2000μ V
temperature range	Room temperature ~1450℃
heating rate	0.1～80℃/min
temperature resolution	0.1℃
Temperature accuracy	±0.1℃
Temperature repeatability	±0.1℃
Accuracy DTA	0.01μ V
Sensitivity of DTA	0.01μV
Temperature control mode	Heating and constant temperature (full program automatic control)
Curve scanning	Heating scanning
Atmosphere control	Automatic instrument switching
display mode	24bit color 7-inch LCD touch screen display
data interface	Standard USB interface Supporting data line and operation software
Instrument standard	Equipped with standard substance (tin), users can correct the temperature and enthalpy by themselves.
remarks	All technical indicators can be adjusted according to user requirements.

4. Instrument interface

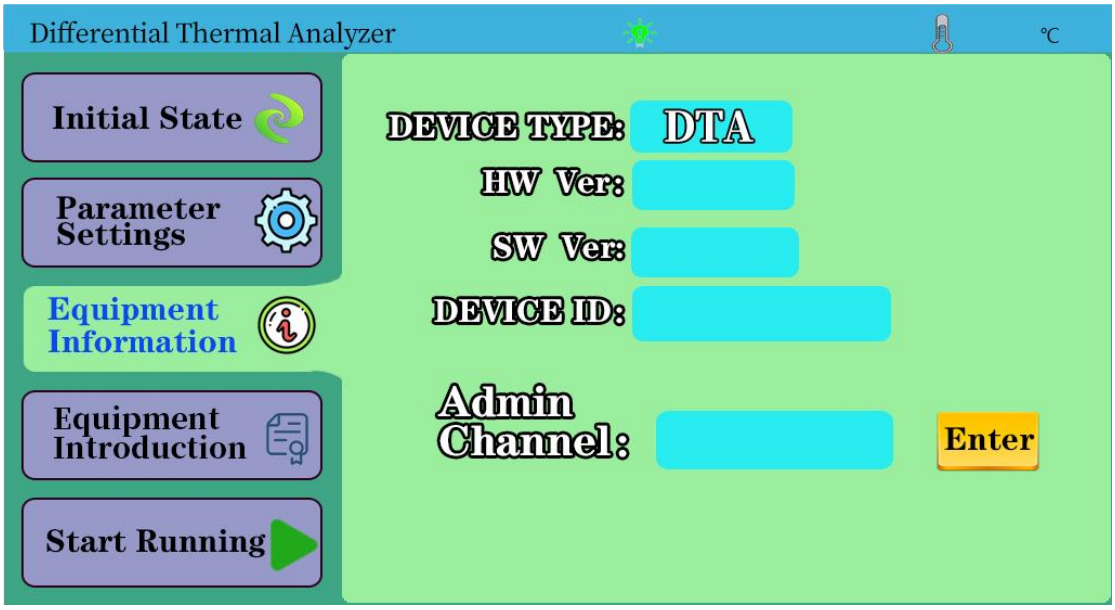
4.1 "Initial state" key, which is used to view information such as ambient temperature and sample temperature.



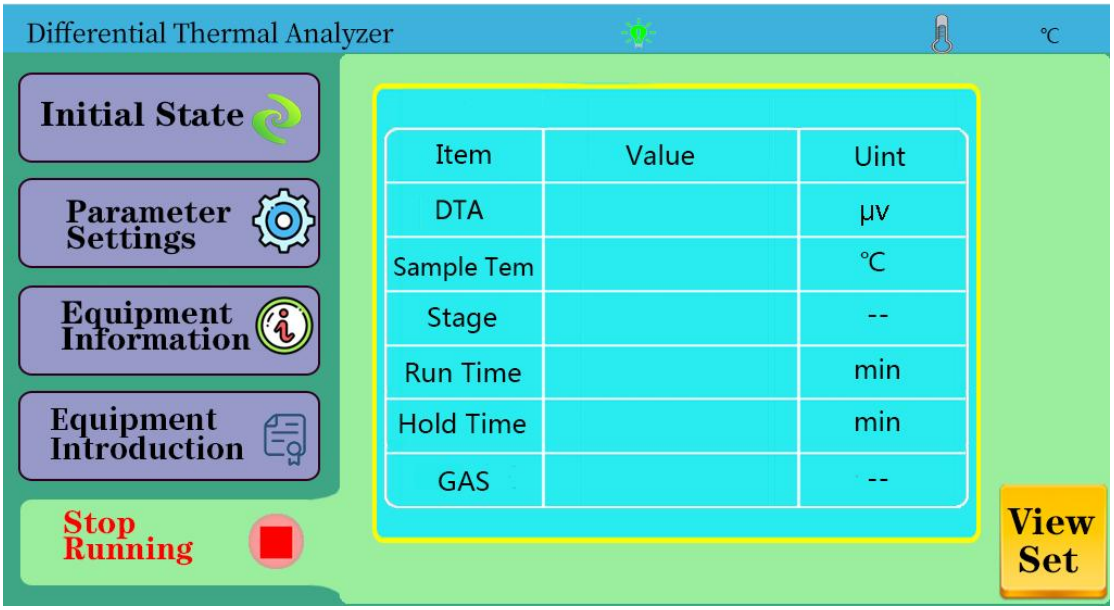
4.2 "Parameter Setting" key, which is used to set experimental parameters, is generally set in software.



4.3 "Equipment Information" key to display equipment information. Used by the personnel inside the administrator channel to calibrate the temperature.

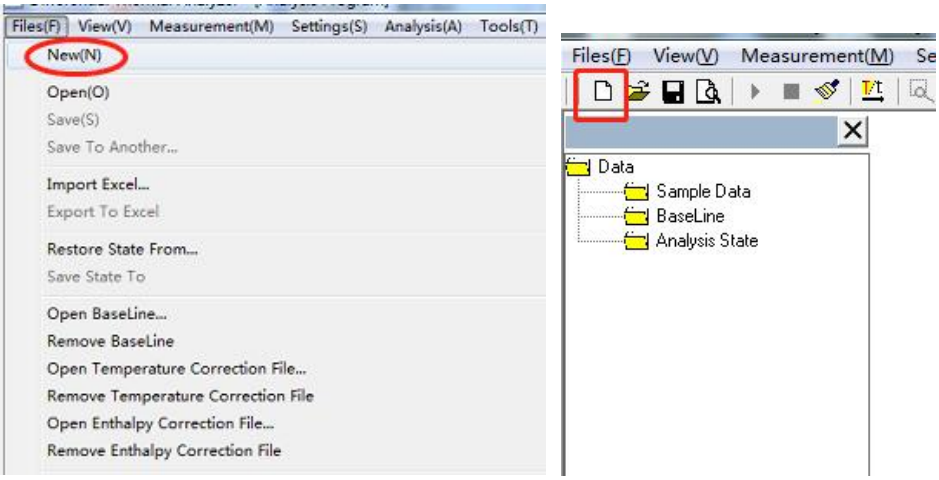


4.4 "Start Operation" button, which displays the current data information after the operation on the computer software is started.

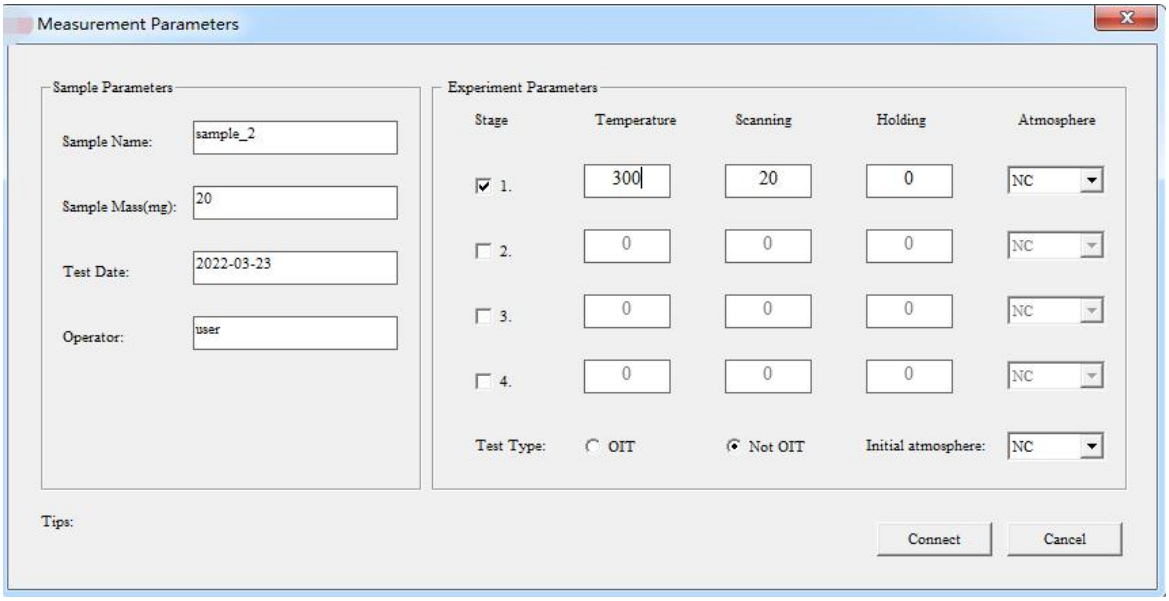


5. Software description

5.1 Open the software and click [New] under the "File" menu bar, or the [New] shortcut key is as follows:

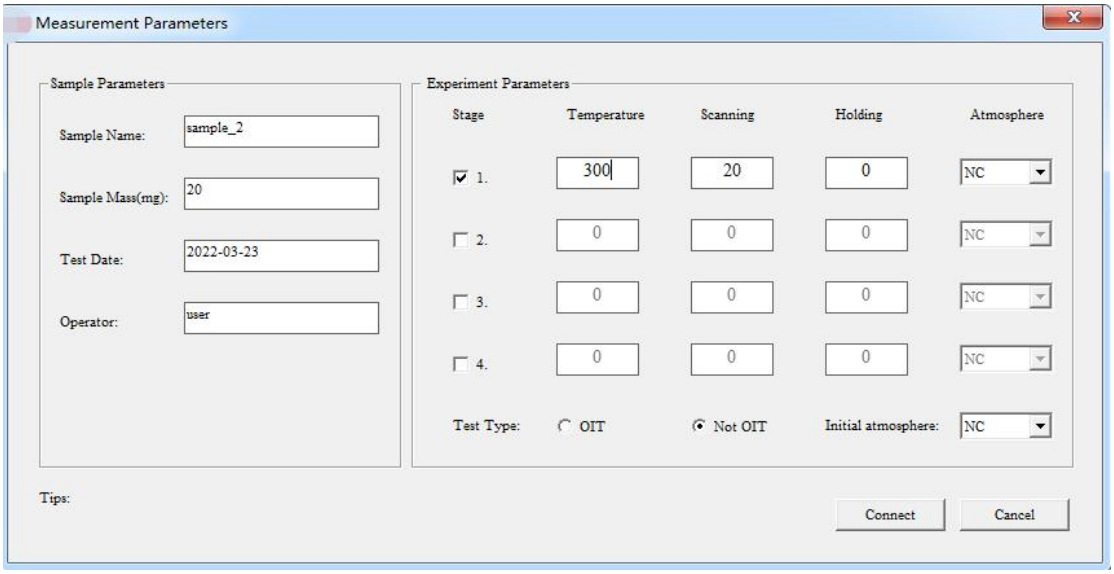


5.2 After clicking "New", you will be transferred to a new window. In the new window, enter information such as [sample name], [sample quality], [operator], [experimental parameters] and [atmosphere]. Select [OIT] or [non-OIT] as the test type according to the customer's requirements. Click [Connect instrument] and you will hear a beep. Pay attention to the two experiments, the sample names can't be the same,Otherwise, the last data will be overwritten, resulting in the loss of the last data. The following figure:

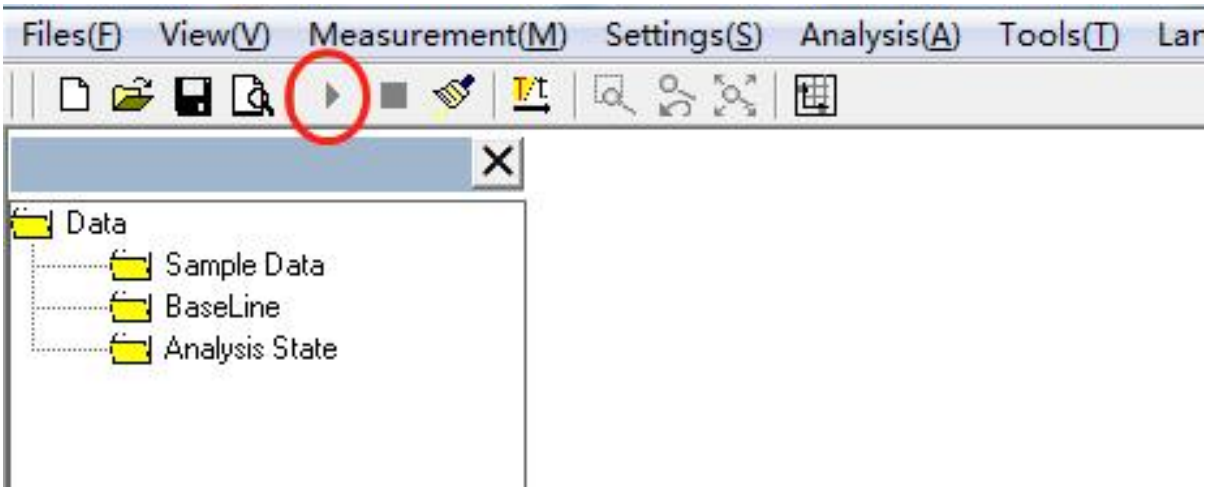


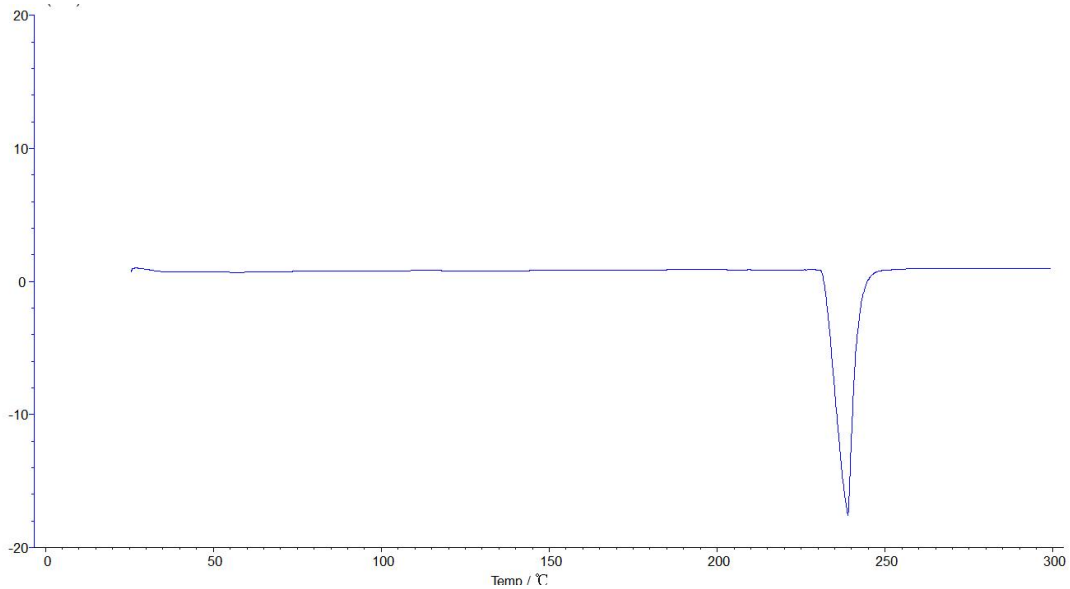
The experimental parameters are set as follows:

"Parameter setting of melting point and phase transition temperature experiment" (according to the sample estimation parameter setting, the test type is non-OIT.) as shown below:

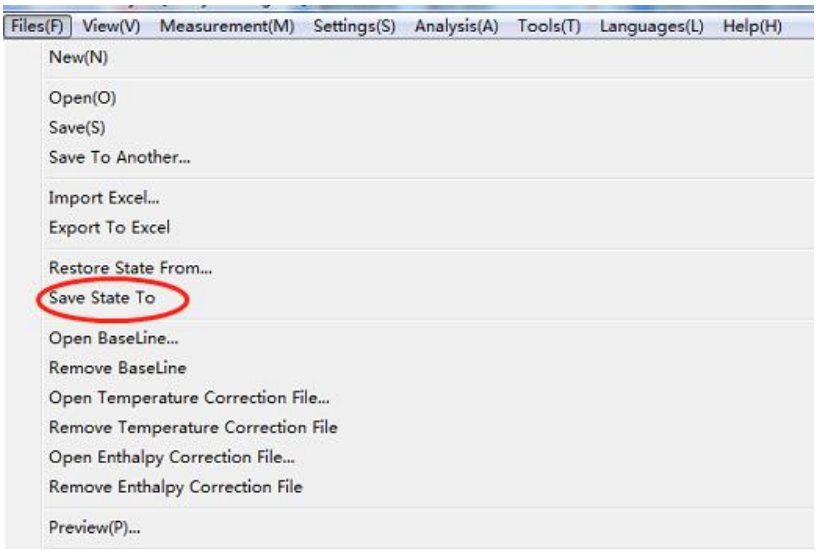


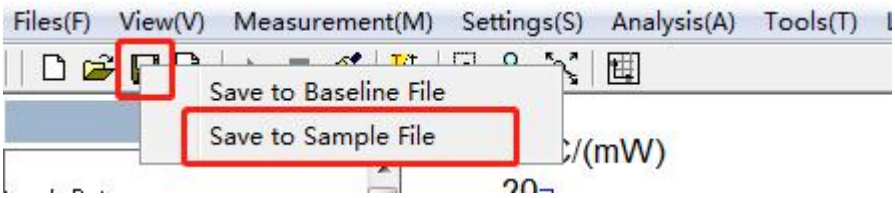
5.3 After all software settings are completed, click [Connect Instrument] and click "in the upper left corner of the software"  "Start button (pictured below), the equipment will heat up according to the set program, and the software will record data in real time. When the temperature is set, the instrument will automatically stop, and the following chart will appear (this chart is the melting point and phase transition temperature chart).



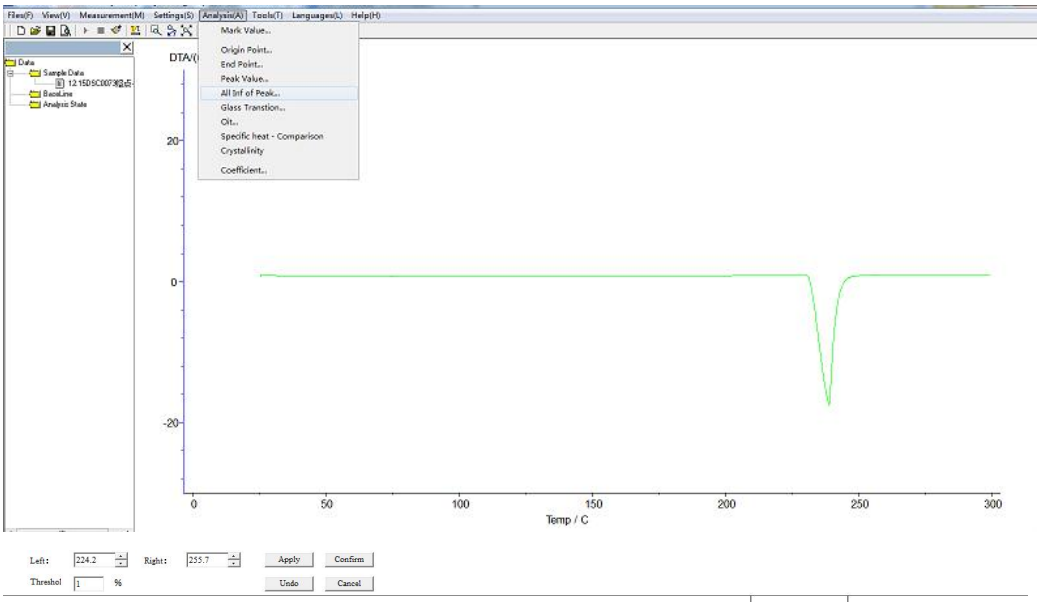


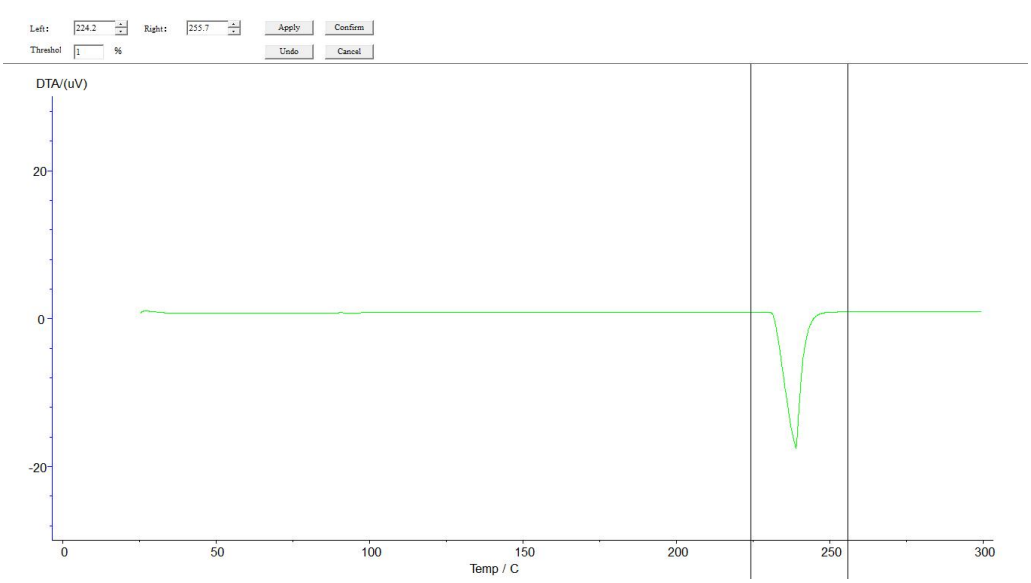
5.4 First, save the atlas to prevent loss. You can also use the shortcut key and select [Save as sample]. And then analyze it. The following figure:

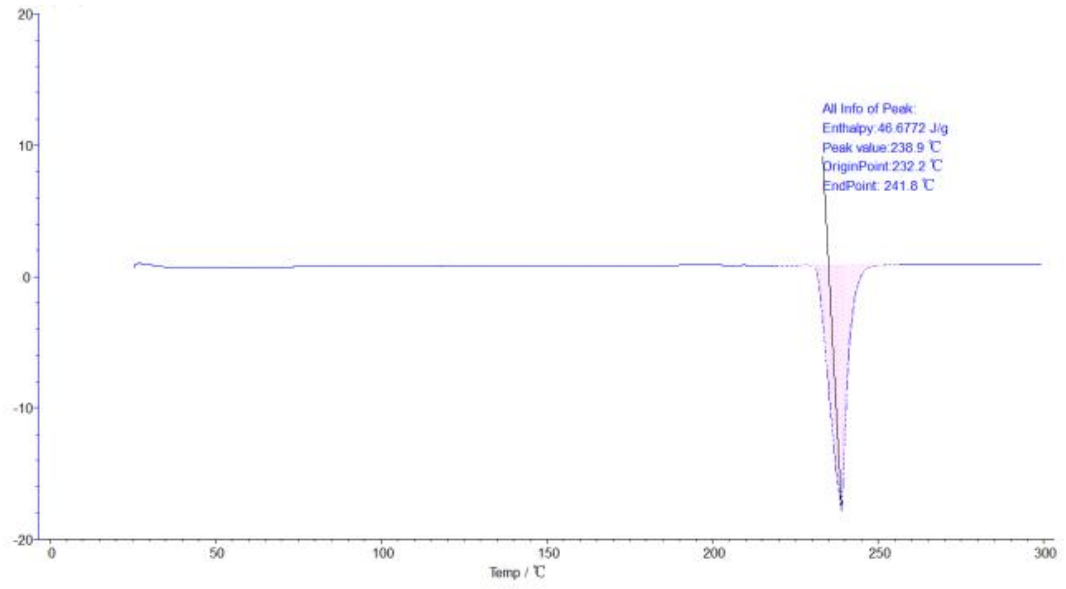




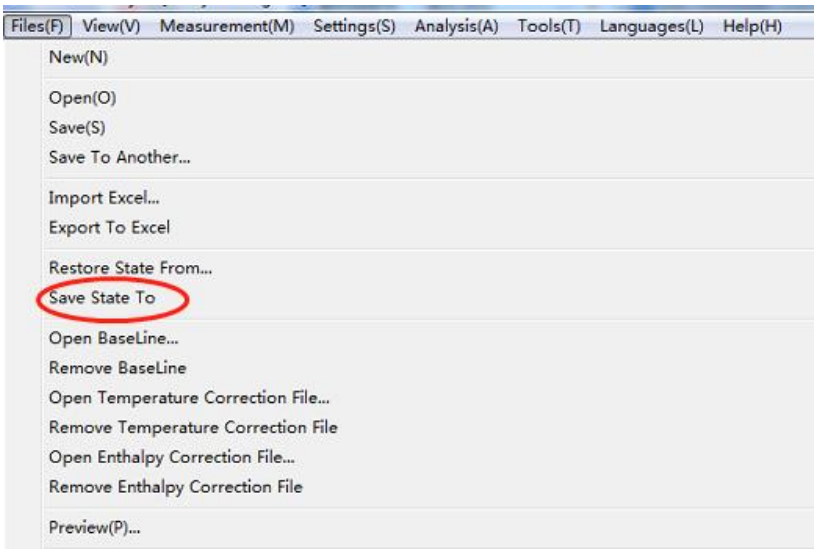
5.4.1 Melting point, enthalpy and phase change temperature analysis process: click the atlas to turn it green, that is, select the atlas, click Analysis-Peak Comprehensive Analysis in the taskbar, two black lines on the left and right will appear, drag the left analytical line at the front end of the change and the right analytical line at the back end of the change, click Apply and OK after selection, and then click the curve to turn it blue.Analysis is complete. The analyzed atlas is as follows:


Mark Value...
Origin Point...
End Point...
Peak Value...
All Inf of Peak...
Glass Transtion...
Oit...
Specific heat - Comparison
Crystallinity
Coefficient...
DTA/(
20
0
-20
0
50
100
150
200
250
300
Temp / C

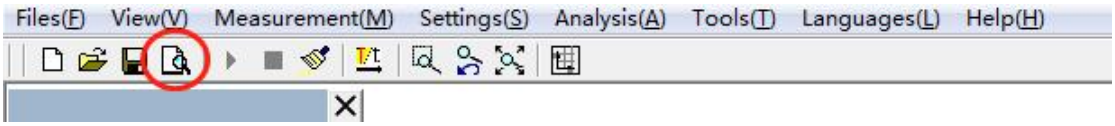

Left: 224.2
Right: 255.7
Apply
Confirm
Threshol 1 %
Undo
Cancel
DTA/(uV)
20
0
-20
0
50
100
150
200
250
300
Temp / C

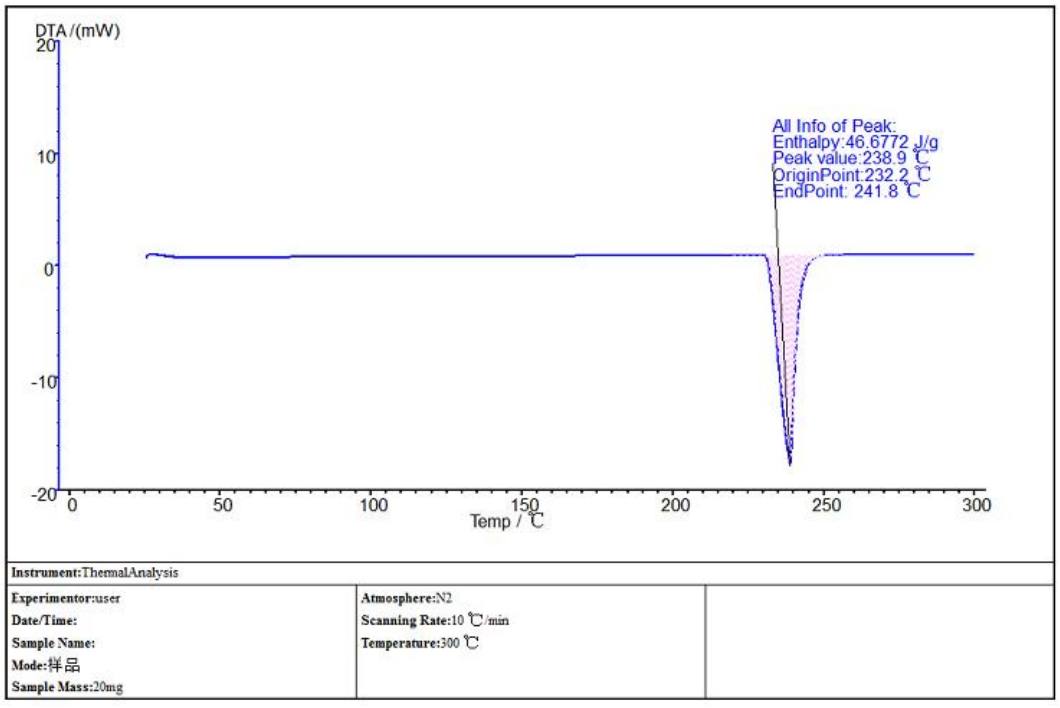

All Info of Peak:
Enthalpy:46.6772 J/g
Peak value:238.9 ℃
OriginPoint:232.2 ℃
EndPoint: 241.8 ℃
20
10
0
-10
-20
0
50
100
150
200
250
300
Temp / ℃

5.5 For all the analyzed maps, click File-Save as Status T to save the analysis data. The following figure:



5.6 All maps can be reported, and click Print Preview, as shown in the following figure:





6. Selection of calibration objects and temperature correction

6.1 Selection of calibration objects

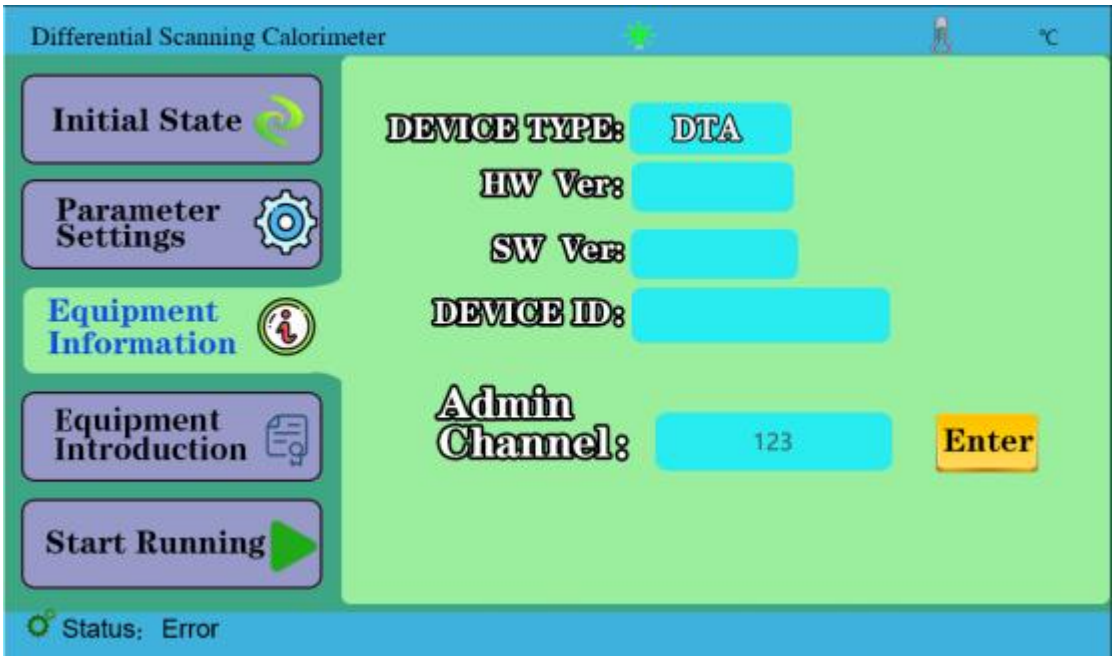
Temperature correction is carried out irregularly to ensure the test accuracy. Select the calibrator according to the actual test temperature of the sample. Principle of calibration object selection: the extrapolated temperature of calibration object should be close to the temperature of the sample to be measured to ensure the accuracy of the test. Our company only provides tin calibrators.

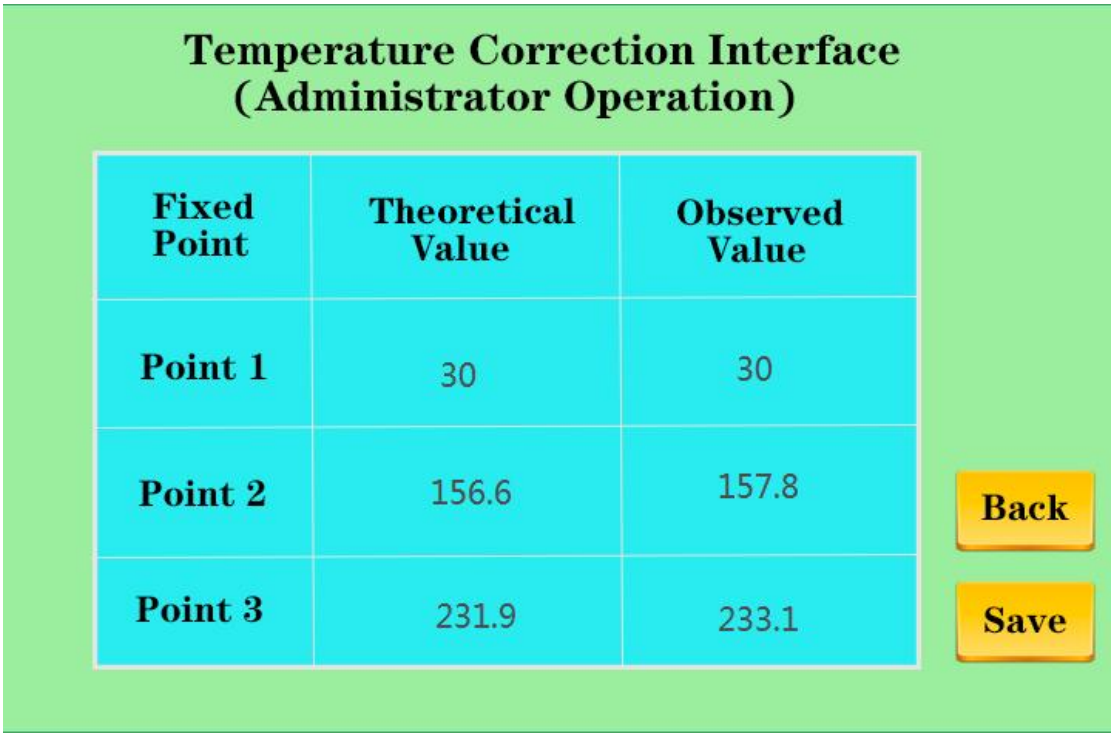
The following table shows the melting point and theoretical enthalpy values of commonly used calibrators.

standard substance	Theoretical melting point℃	Theoretical melting enthalpy J/g
Indium	156.6	28.6
Xi Xi	231.9	60.5
Zinc	419.5	107.5

6.2 Temperature calibration operation steps:

Information-administrator channel-123 access-input theory and measurement value-save-shutdown and restart (the measurement value is the starting temperature obtained by the melting point test of the calibration object)

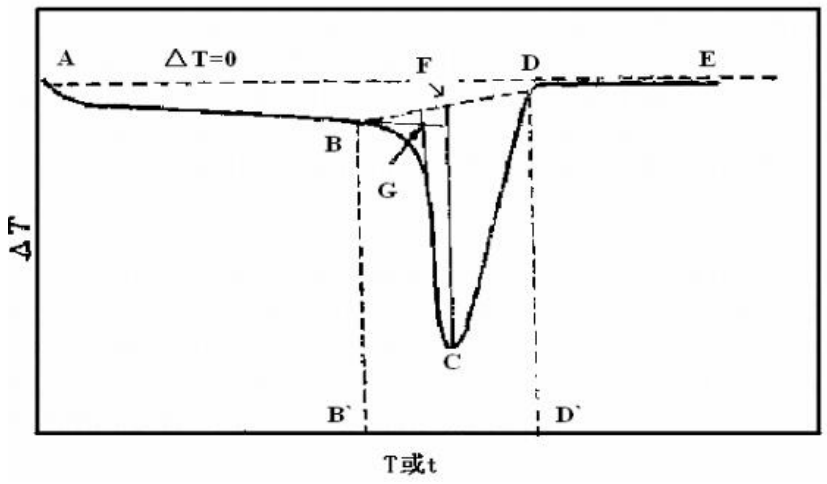

Differential Scanning Calorimeter
℃
Initial State
Parameter Settings
Equipment Information
Equipment Introduction
Start Running
DEVICE TYPE:
DTA
HW Ver:
SW Ver:
DEVICE ID:
Admin Channel:
123
Enter
Status: Error


Temperature Correction Interface
(Administrator Operation)
Fixed Point
Theoretical Value
Observed Value
Point 1
30
30
Point 2
156.6
157.8
Point 3
231.9
233.1
Back
Save

7. Instrument application

7.1 Measurement of melting point (enthalpy)

It is the transition temperature of melting point substance from crystal phase to liquid phase, and it is one of the most commonly measured physical data in thermal analysis. The error between the measurement accuracy and the thermodynamic equilibrium temperature can reach about 1℃. At present, the method recommended by ICTA is used to measure the melting endothermic bee of a solid substance. As shown in the figure below, B' corresponding to point B in the figure is the starting temperature Ti,The temperature corresponding to point G is the extrapolation starting temperature Teo, that is, the intersection point of the tangent line at the maximum slope of the front edge of the peak and the extension line of the front baseline, the temperature corresponding to point C is the peak temperature TM, and the temperature corresponding to point D is the ending temperature Tf.



Enthalpy is a state function representing the energy of a material system, which is numerically equal to the internal energy u of the system plus the product of pressure p and volume v, that is, H=U+PV. Under certain conditions, the change of internal energy and enthalpy of the system can be measured from the heat transfer between the system and the environment. Under the condition of no other work, all the heat absorbed by the system in the process of constant volume is used to increase the internal energy.All the heat absorbed by the system in the process of isobaric pressure is used to increase enthalpy. Since most general chemical reactions are carried out under isobaric pressure, enthalpy is more practical. In DSC curve, we can get the melting enthalpy of the sample by calculating the peak area, that is, BCD in the figure.

7.2 Determination of instrument coefficient

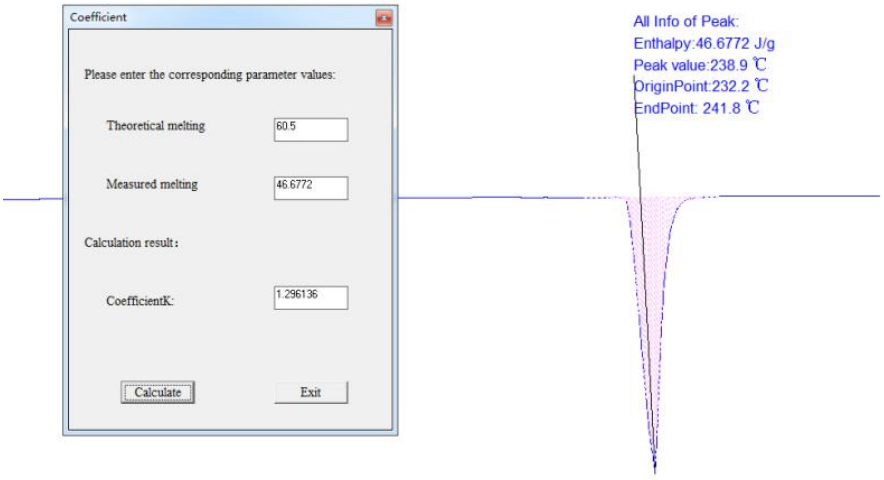
Because the instrument coefficient may change according to the change of environment, temperature, humidity and so on will have greater or lesser influence on it. In order to ensure the accuracy of the experimental results, the coefficient of the instrument should always be measured. Tin, zinc and indium are usually used to calibrate the instrument and measure the instrument coefficient.

The instrument coefficient is to test the enthalpy of the calibration object on the premise of calibrating the temperature, and then calculate the instrument coefficient according to the theoretical enthalpy of the calibration object and the calculation formula of the instrument coefficient.

In the [Data Analysis] column, select [Instrument coefficient] to open the dialog box below, fill the theoretical melting enthalpy and measured melting enthalpy in the corresponding columns respectively, and click the calculation button to get the instrument coefficient. The instrument coefficient is also used in calculating crystallinity. If the experiment is not continuous, the instrument coefficient should be recorded for later use.

Taking the pure tin sample experiment as an example, the theoretical enthalpy of input tin is 60.5J/g, the measured enthalpy is 36.3326J/g, and the instrument coefficient K calculated by the system is 60.5/36.3326. The instrument coefficient is automatically generated on the software interface.

Usually, the instrument coefficient can be measured after the instrument calibration. When calibrating the instrument, weigh the quality of the reference material and fill it in the quality column of the real-time data column. If the phase change temperature measured by calibration is close to the actual temperature of the sample, the enthalpy value of this time can be recorded and the instrument coefficient can be calculated as the coefficient of the instrument. Set as follows:



8 Matters needing attention in the use of instruments

1. In order to ensure the normal use of the instrument, the sample can't be thermally decomposed within the test temperature range, can't react with aluminum metal and has no corrosion. If the measured sample generates a large amount of gas during the heating process, or can cause explosion, the instrument cannot be used. Therefore, we should have a general understanding of the nature of samples before testing.

2. Check whether all connections of the instrument are correct, whether the gas used is sufficient and whether the tools are complete.

3. In the test, if the aluminum crucible is selected as the sample dish, the maximum temperature of the test shall not exceed 550℃.

4. The laboratory room temperature is controlled between 20℃ and 30℃, and the accuracy and repeatability of the experimental results are high when the temperature is relatively constant. When the room temperature is high, the air conditioner should be turned on to ensure that the ambient temperature is relatively constant in a short time. After each experiment, you can do the second experiment only when the temperature drops below 40 degrees.

5. The crucible bottom should be flat, without sawtooth or bending, otherwise the heat transfer is poor.

6. When preparing DSC samples, don't spill the samples on the edge of the crucible, so as not to pollute the sensor and damage the instrument. Samples and impurities can't be attached to the bottom of the crucible and all external surfaces to avoid affecting the experimental results.

7. The sample dosage should be appropriate, neither too much nor too little. Generally, the solid sample is about 10mg. The sample does not exceed one third of the crucible capacity. If the sample dosage is otherwise required, determine the dosage according to the requirements.

8. The inorganic samples can be ground and sieved in advance; The polymer sample should be as uniform as possible; The fiber can be made into the same length of 1~2mm; Powder samples should be compacted.

9. Put the crucible in a fixed position in the sensor. When the amount of sample is small, it should be evenly spread on the bottom of the crucible, and not piled on one side. If the sample is a particle, it needs to be placed in the center of the crucible.

10. The heating rate is usually 10℃/min. Too much will make the curve drift and reduce the resolution; Too small measurement time is long.

11. Do not use hard objects to clean the sample holder and experimental area to avoid irreversible damage to the instrument.

12. If there is dust or other powdery sundries in the experimental area, use ear-washing balls to blow them clean, and it is forbidden to blow them with your mouth to avoid accidents.

13. In the process of data collection, obvious vibration around the instrument should be avoided. It is forbidden to open the upper cover, and slight collision with the front of the instrument will produce obvious peaks and valleys on the DSC curve.

14. Don't adjust the flow rate of purified gas during data collection, because slight change of gas flow rate will affect DSC curve.

15. After the experiment, be careful about the furnace cover of DSC. When the temperature drops below 100℃, handle it gently with tweezers to avoid scalding or damage to the furnace cover.

16. power supply: AC220V,50HZ, power consumption ≤2000W.

17. Disconnect the data cable and turn off the software before turning off the instrument. To prevent online and communication errors. (This problem will be found in XP and SP3 systems, and other systems have not been tested).

Solution:

1. If the online connection is successful and no data is returned, you need to restart the computer.

2. If you encounter online failure, you need to uninstall and reload the USB device with exclamation point in the device manager, without restarting the computer.

9 packing list

Main machine	1 set
USB flash disk	1 only
data wire; data cable	2 root
Power cord	1 root
Aluminum crucible	200
Ceramic crucible	100
Metal cover	1
Ceramic cover	2
Raw tape	1 volume
Pure tin grain	1 bag
10A fuse	5 only
Sample spoon/sample pressure bar/tweezers	1 each
Ear ball	one
trachea	2 root
Instructions	1 copy

Remarks: If other accessories are needed, please discuss separately.

10 Quality Assurance

I 、The guarantee period is 12 months (consumer goods and travel cost are not included).

II、The main quality warranty maintenance certificate

1. Please keep this guarantee letter, if you lost it, please connect with us in a month.

2. If this guarantee letter has been altered or it has no our stamp, it is useless.

III、The following conditions need to pay reasonably even in the assurance period.

1. Natural reasons

2. Operating mistakes

3. Voltage is not fit for our operation instruction

4. Repack it without our guides

5. Damaged for borrowing to others

6. Damaged for authorized machine modification

7. Damaged for authorized calibration

8. Authorized transshipment mistake

9. Serve for long distance area

IV、Notes:

1. Where the original in Guangdong province installation of our products, if the product moved to outside Guangdong-area use, whether or not within the effective warranty period, the transportation and travel expenses of the service personnel should be paid by the client.

2. All customers outside of Guangdong province, whether or not in the warranty period, the service of transportation and travel expenses paid by the customer.