SSA-7000

Aperture and specific surfacearea analyzer

Operation manual

☎ 400-6698981

Dearusers:

First of all,thank you for purchasing our products,our company independent research and development,generation,sales of products mainly:

1. specific surface area and aperture analyzer series
2. Temperature programmed chemisorbed instrument series
3. Gas method true density instrument series
4. Volume method steam adsorption instrument series
5. Volumetric gas adsorption instrument series
6. Capacity method high pressure adsorption instrument series
7. Bubble point method membrane aperture analyzer series
8. Laboratory valve series (intake solutions for IR/ MS etc.)
9. Customized series of pilot catalytic evaluation devices (including VOCs generation and treatmentseries,SCR denitrification and desulfurization series,etc.)
10. On-line mass spectrometer series

The company was founded on January 9,2003,specializing in the technical research and development of material characterization instruments,is a collection of project research and development,product generation,testing and consulting in one of the technical serviceenterprises..In the development process of nearly 20 years,the company has accumulated a group of high-quality R & D and application technical personnel,under the guarantee ofstrong technical support and personnel infrastructure,Piode can independently complete the generation of products and core partsR & D and manufacturing,for all levels of scientific research unit s and enterprises to provide technologydevelopment services outside the scope of the main product.

This manual describes all the functions of the SSA-7000. Your product may not have all the features in this manual. The actual product shall prevail.

It is not covered by the warranty because it is contrary to its proper use or is clearly defined as a predictable misuse.

1. This document refers to the identification of the StencilWizard software version and the identification of the firmware software version.
2. Please read this manual carefully before using it for the first time.If you have any questions or suggestions,please contact 400-669-8981 and email to service@bjbiaode.com.

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lMore outlets are under construction

catalogue

Chapter 1 Installation

(1) Installation of gas cylinders and pressure reducing valves

1. Install the pressure reducing valve adapter

As shown in Figure 1-1, insert the yellow spherical end of the pressure reducing valve joint into the gas cylinder outlet and tighten the sealing nut, as shown in Figure 1-2.

Figure 1-1

Figure 1-2

2. Install pressure reducing valve switch valve

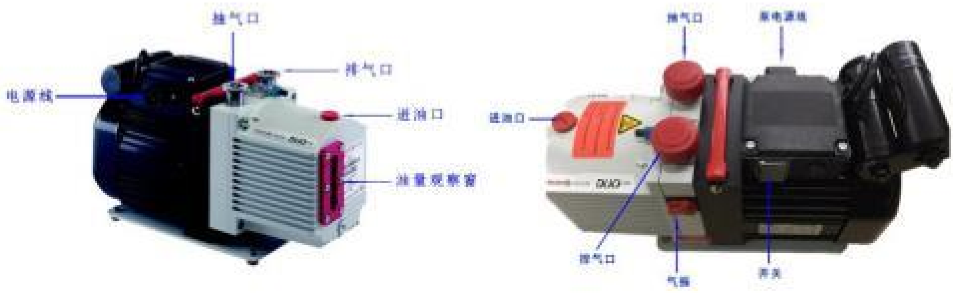
The black knob (i.e., the switch valve of the pressure reducing valve) is installed on the pressure reducing valve, as shown in Figure 1-3

graph 1-3

(2) Installation of vacuum pump

1. Installation and use of host vacuum pump

The SSA-7000 is equipped with a dual-stage rotary vane mechanical pump (Figure 1-4) and a turbine molecular pump (Figure 1-5). The molecular pump is integrated within the analytical main unit and does not require separate installation. Note that the suction port and exhaust port must not be mistakenly installed, as this can cause the pump body to rupture and become irreparable or result in more serious safety accidents. The pumps air vibration should always remain at the factory head, and must not be touched.



graph 1-4

graph 1-5

(1) Power line connection

Insert the other end of the power cable of the mechanical pump into the 220V power interface, as shown in Figure 1-6

graph 1-6

(2) Installation of KF16 clamp

graph 1-7

As shown in Figure 1-7, fit the KF 16 sealing ring over the bell mouth, then fit it into the clamp and tighten the nut. When in contact with.When connecting the instrument, align the mouth of the corrugated tube with the instrument interface and press it tightly, then put in KF 16 clamp and tighten the nut.

(3) Pump oil addition

Turn the inlet of the mechanical pump (see Figure 1-4), slowly pour the pump oil into it, and observe the oil level at 2/3 of the pumps front end oil level observation port (Figure 1-8), then tighten the inlet oil screw cap. If too much pump oil is poured, use a hex key to open the drain port next to the oil level observation port to release the excess oil and then tighten the screw cap.

graph 1-8

(4) Pump usage

① Use of mechanical pump

The opening and closing of the pump is controlled by the vacuum pump power switch.

When the instrument is working, the real pump switch should be opened, and the vacuum pump switch should be closed when it is finished.

2. Installation and use of vacuum pump in degassing station

(1) Understand the vacuum pump

As shown in Figure 1-10, the instrument is equipped with a dual-pole rotary vane vacuum pump. Before installation, please familiarize yourself with the functions of each joint/switch on the pump to avoid incorrect installation. Note, do not confuse the suction port and exhaust port installation, as this can cause the pump body to explode and become irreparable or result in more serious safety accidents.

注意：

切勿将排气

口当成抽气

口安装！！！

graph 1-10

(2) Install bellows

① Install KF 16 card sleeve

As shown in Figure 1-11, the KF 16 sealing ring is placed over the bellows opening, then the clamp is placed on it, and the nut is tightened.

graph 1-11

② When connected to the instrument, align the bellows section with the vacuum pump suction port, then fit the KF 16 clamp and tighten the clamp, and connect the other end of the bellows to the instruments vacuum port in the same way, and tighten the clamp.

graph 1-12

(3) Add pump oil

Turn the oil inlet of the vacuum pump (see Figure 1-13), slowly pour the pump oil into the vacuum pump, and observe the oil level at or above the midpoint through the oil level observation port at the front end of the vacuum pump (as shown in Figure 7), then tighten the oil inlet. If too much pump oil is poured in, use a hex key to open the drain port next to the oil level observation port and release the excess pump oil, then tighten the nut.

graph 1-13

(4) Power line for pump installation

① One end of the power cable of the general vacuum pump is already connected to the pump body, and the other end is inserted into the three-phase power socket. After opening the power switch of the vacuum pump, the pump starts to work.

② When the pump body is not connected to the power cable, insert the matching power cable into the power socket of the pump body, and insert the other end into the three-phase power socket. After opening the power switch of the vacuum pump, the pump starts to work.

(3) Connection of gas line

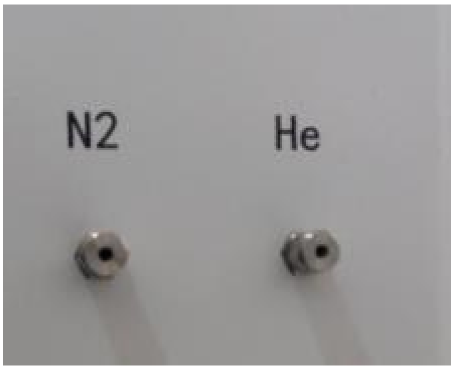
1. Install the air line sealing ring

Remove one Φ3 stainless steel external air line, and sequentially fit one nut and 2 or 3 Φ3 seals to each end of the air line

Circle. As shown in Figure 1-14

graph 1-14

2. Connection of gas cylinder to instrument gas line



graph 1-15

(1) Connect the nitrogen line

Insert one end of the gas line into the "N 2" gas interface of the instrument (see Figure 1-15 for N 2 interface), tighten the nut, and connect the other end to the outlet of the pressure reducing valve of the nitrogen cylinder and tighten the nut.

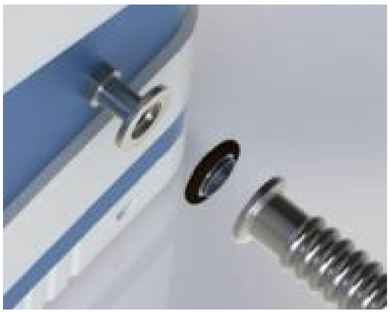
(2) Connect the helium line

Insert one end of the gas line into the "He" gas interface of the instrument (He interface is shown in Figure 1-15), tighten the nut, and connect the other end to the outlet of the pressure reducing valve of the helium cylinder and tighten the nut.

3. Vacuum pump and instrument air line connection

(1) The main unit is connected to the mechanical pump air circuit

As shown in Figure 1-16, according to the installation method of the bellows, one end of the bellows is connected to the air extraction port of the main engine, and the other end is connected to



Mechanical pump suction port connection.

graph 1-16

4. Gas line connection of degassing system

Insert one end of φ3 gas line into the left exhaust port of SSA-7000 host (see Figure 1-17), tighten the nut, and connect the other end to the suction port of vacuum pump and tighten the nut.

graph 1-17

(4) Installation of the main unit

1. Install the degassing station electric furnace

(1) Place the electric furnace on the lifting tray on the left side of the main unit.

(2) Insert the electric furnace power cable interface into the "Electric Furnace" port on the left side of the SSA-7000 main unit, and tighten the nut. Align the heating furnace power cable groove with the protruding part of the port and insert it, then tighten the nut; when pulling out, loosen the nut, grip the end of the power cable interface firmly to pull out, do not pull directly by holding the wire. As shown in Figure 1-18

graph 1-18

2. Connect the host power cable

Remove the power cable, one end inserted into the instrument power interface, the other end inserted into 220V power interface.

3. Start the host

Turn on the power switch on the left side of the main unit, turn on the instrument and enter the operation interface after the control system is started, as shown in Figure 1-19

graph 1-19

(5) Software installation

1. Installation of PC computer analysis software

Double-click to open the "StencilWizard Setup" installation package, open the software installation guide, click "Next" according to the installation prompt, you can use the default installation path, or click "Change" to customize the installation path, until the installation is completed

A shortcut icon appears on ".
the back desktop

StencilWizard - InstallShield Wizard

正在准备安装...

StencilWizard 安装程序正在准备 InstallShield Wizard，它将引导您完成剩余的安装过程。请稍候。

正在解压缩: StencilWizard.msi

StencilWizard InstallShield Wizard

欢迎使用 StencilWizard InstallShield Wizard

InstallShield(R) Wizard 允许修改、修复或删除 StencilWizard。要继续，请单击"下一步"。

StencilWizard InstallShield Wizard

目的地文件夹

单击"下一步"安装到此文件夹，或单击"更改"安装到不同的文件夹。

将 StencilWizard 安装到:
H:\StencilWizard\

StencilWizard InstallShield Wizard

已做好安装程序的准备

向导准备开始安装。

若要查看或更改任何安装设置，请单击"上一步"。单击"取消"退出向导。

当前设置:

安装类型:
典型

目的地文件夹:
H:\StencilWizard\

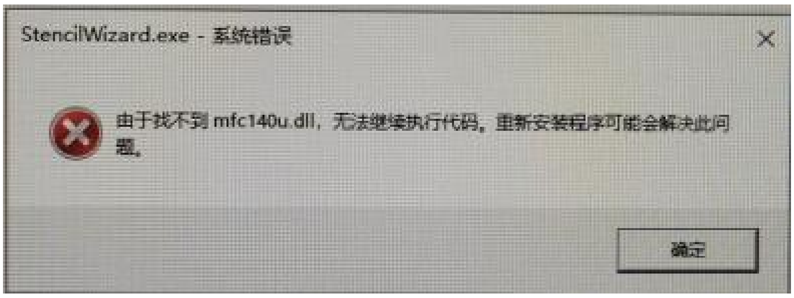
用户信息:
姓名: Microsoft
公司: Microsoft

graph 1-20

This software is for data analysis only and can be installed on any computer

2. The analysis software is not available

When the prompt software fails to open and there is an error as shown in Figure 1-21, you need to install "mfc 140u.dll" in the C disk system. The installation method is as follows.



StencilWizard.exe - 系统错误

由于找不到 mfc140u.dll，无法继续执行代码。重新安装程序可能会解决此问题。

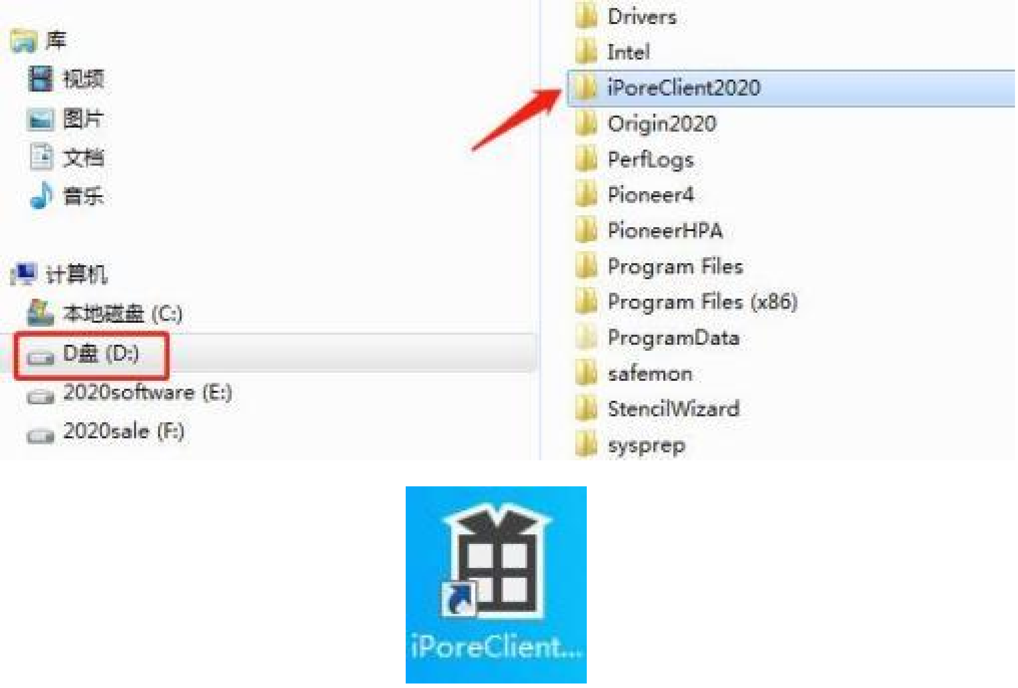
graph 1-21

The system is windows 10 X64

Step 1: Put the mfc 140u. dll in the C drive windows under the SysWOW 64. Step 2: Run the setup. exe

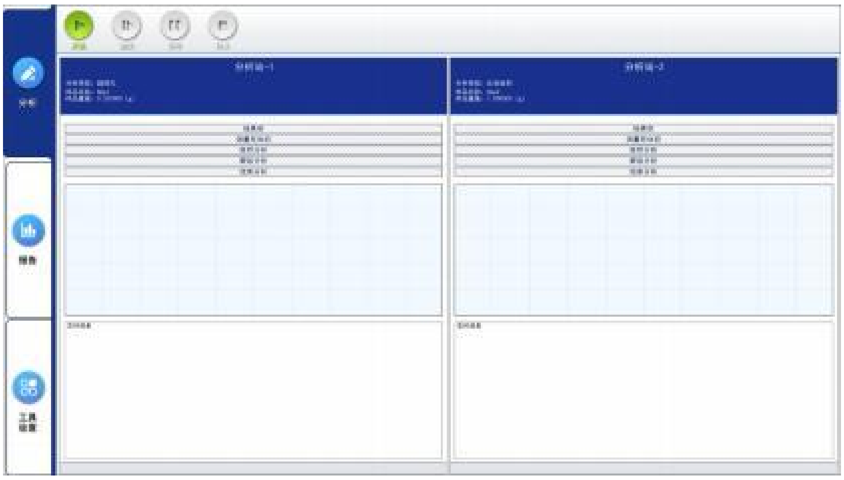
3. Connection between computer control end and SSA-7000 host

(1) First, use USB data cable to connect the external computer and SSA 7000 host, copy the computer control software to the root directory of D disk, and create a shortcut on the desktop. As shown in Figure 1-22



graph 1-22

(2) Open the client, as shown in Figure 1-23.



graph 1-23

(3) Click the tool Settings module, click "Load from Report", select any library file, and then click Load to copy the system parameters from the SSA 7000 host to the current computer client.

界面语言

简体中文

本地大气压[KPa]: 102.000

声音设定

☑ 步骤提醒

☑ 完成分析提醒

分析计量平衡[秒]: 10

建立Po时间[秒]: 8

采样延迟[秒]: 10

卸载压力时间[秒]: 120

最大分析时间[时]: 18

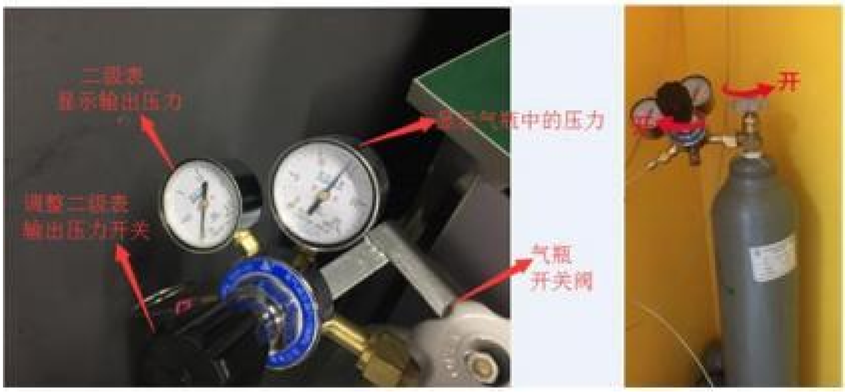
☑ 自动卸载压力

graph 1-24

(4) After the above operation is completed, the measurement parameters can be set up by the external computer, data can be collected and reports can be saved.

Chapter II Use

(一) Use of gas cylinders and decompressors



graph 2-1

1. Understand the pressure reducer and the main valve of the cylinder

The display table of the pressure reducer, switch and switch of the gas cylinder are shown in Figure 2-1.

2. Adjust output pressure

Open the main valve of the cylinder, then turn the black screw to adjust the output pressure (open the output when adjusting clockwise, and close the output when adjusting counterclockwise), and adjust the output pressure of nitrogen and helium to 0.3 MPa respectively.

3. Use of gas cylinders

When not working, just close the main valve of the cylinder. When used next time, just open the main valve of the cylinder without adjusting the output pressure again.

4. Use of gas

In the conventional nitrogen adsorption experiment, high purity nitrogen (purity 99.999%) is used for adsorbate/gas, and high purity helium (purity 99.999%) is used for dead volume quantitative gas.

(二) Operation status check and calibration

1. Check the operating status of the machine

After power-on, observe whether the control system can enter the control interface normally. The start of the control system is generally within 1 minute.

2. Check the intake pressure and vacuum state

(1) Click instrument control 控制仪器 "Click the icon to enter the airway control interface, as shown in Figure 2-2

After the host is preheated, open the airway control interface and observe whether the temperature in the lower right corner is displayed as 35℃.

4. Exit the computer control end

In the tool Settings interface, select background maintenance" 后台维护 "Close the computer control end. Remember not to choose to close the instrument

impl 关闭仪器 ", otherwise the computer will be shut down immediately.

(3) Use of degassing stations

1. Sampling and weighing

(1) Determine the sample amount

Usually, the sample to be analyzed provides a surface area of 15 to 150 square meters, suitable for nitrogen adsorption analysis. Less than this will result in unstable analytical results, while more than this will prolong the analysis time. For samples with large specific surface areas, the sample amount should be small. For small amounts of samples, weighing after degassing becomes crucial, as even minor weighing errors can account for a significant proportion of the total weight. Thus, weighing techniques are particularly important. The sample weight should not be less than 30 milligrams. For powdered samples, use a long-necked funnel, add the sample to the bottom of the sample tube, and when removing the funnel, tilt the sample tube slightly to prevent residual sample from falling onto the walls of the sample tube. For larger particle samples, use tweezers for addition (note, do not touch the sample with your fingers to avoid adhering grease to the sample surface).

Sample usage reference table:

specific surface [㎡/g]	<1	1-10	10-100	100-300	＞300	remarks
volume of sample loading [g]	＞2	1-2	＞0.5	＞0.1	<0.1	The minimum should not be <0.01

(2) Sample weighing (weight of water-containing samples)

Since the analysis results are expressed as surface area per unit mass, the true mass of the sample is required. The sample tube and sample should be weighed carefully.

① Remove the sample tube and stopper with labels, plug the sample tube with the stopper, and record the sample tube number and stopper number in the notebook.

② Place the tray (the tray can be a container such as a measuring cylinder or glass cup, used to place the round bottom sample tube) on the balance and weigh it to remove the skin so that the balance is stable at zero.

③ Place the sample tube and stopper together with the tray on the balance to weigh. After the balance value is stable, record the weight m1 of the empty sample tube and stopper (see Figure 2-3).

graph 2-3

④ Use a funnel to fill the bottom of the sample tube with the sample. The height of the accumulated sample should be less than 15 mm (see Figure 2-4).

graph 2-4

⑤ Replace the stopper, weigh the sample tube and stopper containing the sample, and record the total weight m2 of the sample tube before degassing.

⑥m2 minus m1 gives the weight of the sample before degassing and drying m3.

2. Install sample tube

Most samples adsorb a large number of pollutants and impurities on their surfaces at room temperature, which should be removed before analysis. The sample surface is cleaned by heating the sample under vacuum to remove the dirt on the sample surface, which is called sample degassing.

(1) If there is a plug or sample tube at the degassing port of the degassing system, loosen the nut first and remove the plug (see Figure 2-5 left); see Figure 2-5 right for the installation of the plug.

graph 2-5

(2) A nut, 2 sealing rings and 1 filter plug are installed at the sample tube opening (the filter plug is not required for non-powder samples), as shown in Figure 2-7

graph 2-6

(3) Install the sample tube on the degassing port, keep the sample tube perpendicular to the ground, push it up tightly, and then tighten the nut by hand.

(4) According to the above method, all the degassing ports are connected to the sample tube or plug.

(5) Raise the heating furnace to the specified position, adjust the position of the heating furnace, so that the sample tube ball is exactly buried in the heating furnace. See graph 2-7

graph 2-7

(6) If the heating furnace power cable has not been installed, install the heating furnace first before proceeding with (5). Pass the heating furnace power cable through the hole in the center of the tray and insert it into the power socket of the electric furnace. Place the heating furnace on the lifting tray as shown in Figure 2-8. The heating furnace power cable groove should align with the protruding interface, then insert the needle hole and tighten the nut; when removing, loosen the nut, grip the power cable interface end firmly to pull out, do not pull directly by holding the wire.

Note: When connecting the power supply of the heating furnace, the instrument should be in a state of power off.

graph 2-8

3. Set the degassing mode


300.0℃
-0.01 KPa
真空 1
预处理中
80%
300 温度 ℃
注气 3
120 时间 min

graph 2-9

(1) Click on the "Temperature" and "Time" checkboxes, and input the temperature and time to be processed respectively. For samples with unknown processing time, you can check the "AI" key at the same time, and the instrument will automatically determine the degree of dehydration completion and record and display the time.

(2) Select sample processing scheme: There are three types ("water", "light", "standard"), and the instrument will run the corresponding processing scheme according to the sample type and condition. After selection, the instrument will run the corresponding processing scheme according to the sample type.

(3) Click the "S" key to start degassing, and the heating furnace will automatically rise to the target temperature.

4. Weigh again after degassing

(1) After degassing is completed, there will be a "beep" alarm sound and a pop-up prompt. At this point, lower the heating furnace to cool the sample tube to room temperature, but be aware that the heating furnace is still in a high-temperature (cooling process) state; do not touch the heating furnace walls or openings with your hands to avoid burns from high temperatures.

(2) Click the "inflate" button to inject inert gas protection gas, usually high purity nitrogen is used.

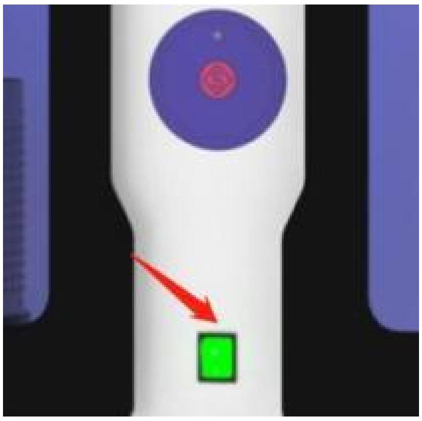
(3) Reweigh (net weight of sample)

Remove the sample tube and quickly insert the corresponding labeled sample tube stopper into the tube opening to prevent the dried sample from coming into contact with air again then place the sample tube on a balance to weigh it and record it as m4. Subtract m4 from m1 to obtain the net weight of the dried sample m.

Note that if m(mass of dried samples) is greater than m3(mass of samples before drying), the effect of weighing error or operational error should be considered. If the difference is large, the samples should be reprocessed according to the above steps.

(4) Start the instrument

1. Turn on the power switch of the instrument (on the front and middle-lower position of the instrument), as shown in Figure 2-10

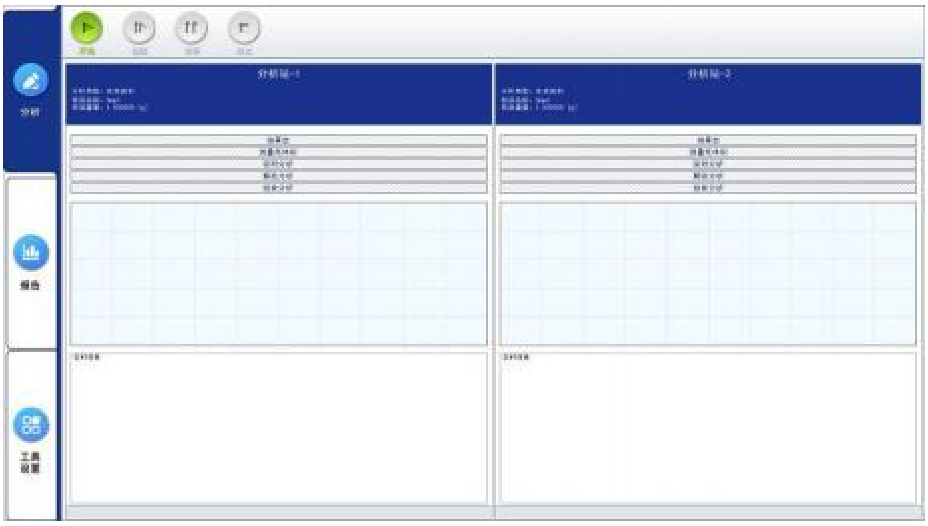


graph 2-10

2. The LCD computer touch screen on the instrument control panel will show a startup page, as shown in Figure 2-11

graph 2-11

3. After waiting for about ten seconds, the instrument system enters the operation page, as shown in Figure 2-12. Users can operate through the touch screen.



graph 2-12

(5) Use of touch computer systems

After the computer is turned on, the screen of the touch computer system is divided into three modules: "Analysis", "Report" and "Tool Settings". When the instrument is used for the first time, the "Tool Settings" module should be entered first, and the basic information should be set in the "Instrument Parameters" interface.

1. Tool Settings module

In this module, you should set up basic parameters under the "User Operable" module, and do not operate other modules by yourself, otherwise the loss caused by you shall be borne by the user.

1.1 User operable modules (left sidebar)				
1.2 User operability module (tool group module)				
	The instrument air circuit can be opened to check the status of each component	Exhausting is required when a new unit is installed or when the cylinder is replaced. The number of times nitrogen and helium are exhausted uses the default value	Undertheguidanceofthe manufacturer, updatethe system and put the update file into the root directory of E disk	Check the pressure values of each analysis station

1.3 Operation modules are strictly prohibited (if operation is required, it should be carried out under the guidance of the manufacturer, phone 400-669-8981)	校准传感器 测量计量舱 校准PFC

(1) Software parameters-conventional parameters, as shown in Figure 2-13


远程控制
☑ 允许远程控制
仪器名：SSA-7000
密　码：●●●●●●●●
本地IP
192.168.0.147
界面语言
简体中文
本地大气压[KPa]
102.000
声音设定
☑ 步骤提醒
☑ 完成分析提醒

时间参数

快速分析模式
抽真空[分]：5
死体积计量平衡[秒]：30
死体积热平衡[秒]：300
清除氦气[秒]：300
分析计量平衡[秒]：10

标准分析模式
抽真空[分]：7
死体积计量平衡[秒]：300
死体积热平衡[秒]：600
清除氦气[秒]：600
分析计量平衡[秒]：10

高精度分析模式
抽真空[分]：20
死体积计量平衡[秒]：600
死体积热平衡[秒]：1200
清除氦气[秒]：1200
分析计量平衡[秒]：60

建立Po时间[秒]：8
采样延迟[秒]：10
卸载压力时间[秒]：120
最大分析时间[时]：18
☑ 自动卸载压力

graph 2-13

① Remote control: After selection, you can use the computer to access the instrument.

② Time parameter: divided into fast, standard and high precision analysis mode, generally use default value (do not change it yourself).

(2) Instrument parameters, as shown in Figure 2-14

仪器参数
仪器型号：iPore600
分析站数量：2
气路温度[°C]：40.0
生产日期：0 年 0 月 0 日
仪器编号：
固件版本
版本号：1.5.43

graph 2-14

① Configuration of instrument: set the model number of instrument, number of analysis stations, generation date, instrument number, etc., and click apply.

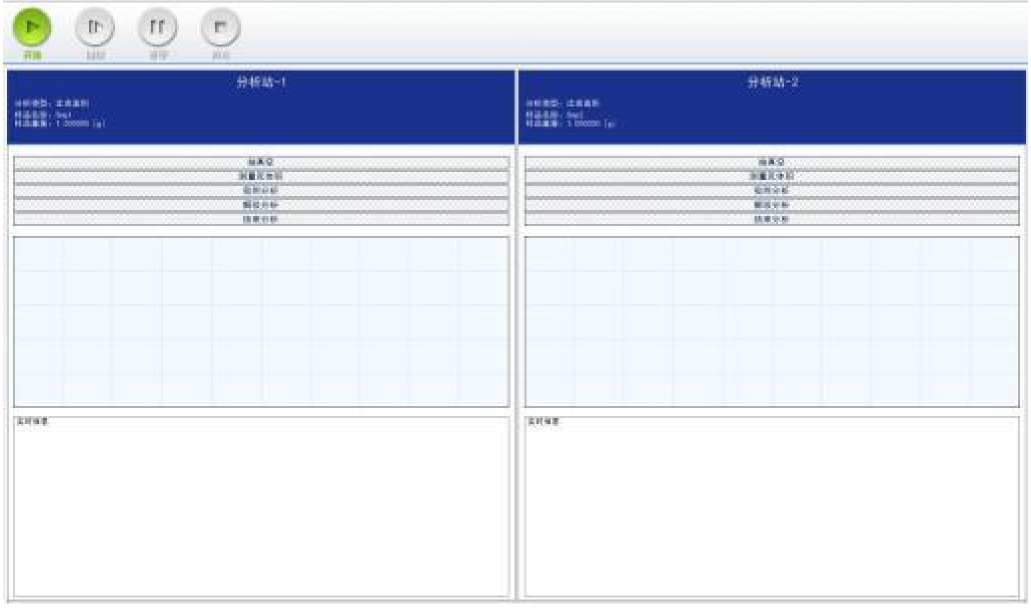
② Measurement tank volume: default parameters at factory, no user setting required.

③ Calibration parameters: factory default parameters, no user setting required.

④ Set PFC: factory default parameters, no user setting required.

2. Analysis module

As shown in Figure 2-15, this is the commonly used interface for the test sample.



graph 2-15

(1) Set the analysis station analysis parameters

As shown in Figure 2-16, click on the blue area of the analysis station to enter the "Set Measurement Parameters" interface. After opening, you can set the measurement parameters of each analysis station; the common parameters are generally set at the time of delivery and do not need to be modified.

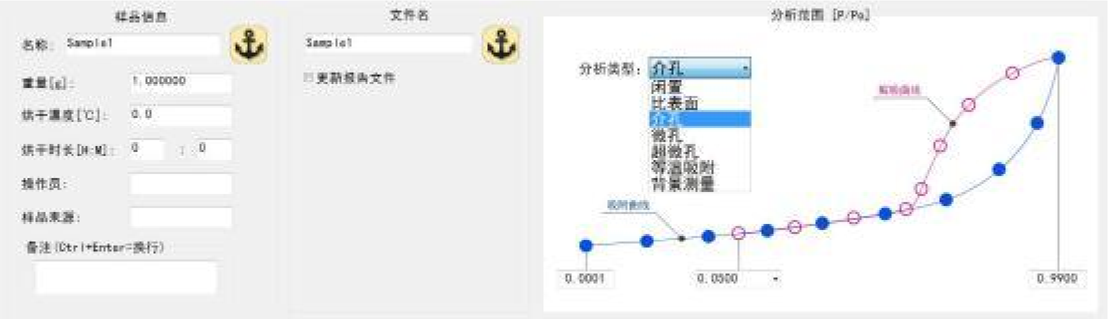
Analysis station measurement parameter setting	**Public parameter setting**
	

分析站-1

分析类型：比表面积
样品名称：Smpl
样品重量：1.000000 [g]

graph 2-16

① Test mode setting, as shown in Figure 2-17



样品信息
名称：Sample1
重量[g]：1.000000
烘干温度[℃]：0.0
烘干时长[H:M]：0 : 0
操作员：
样品来源：
备注(Ctrl+Enter=换行)

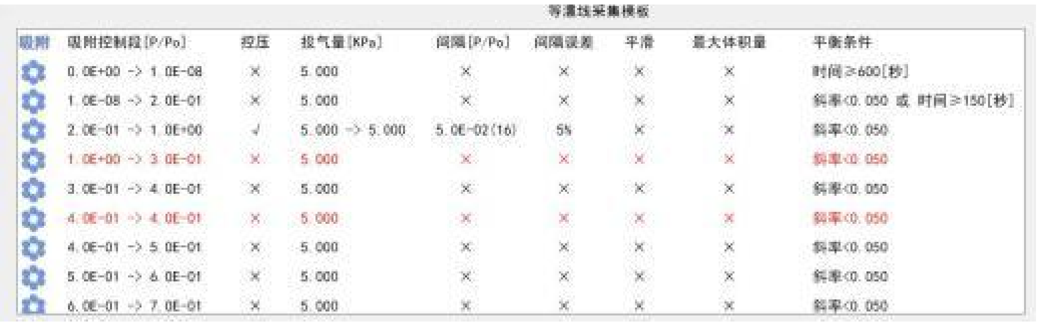
文件名
Sample1
☐ 更新报告文件

分析范围 [P/Po]
分析类型：介孔

graph 2-17

Five types of analysis can be set: "specific surface/mesoporous/microporous/ultra-microporous/isothermal adsorption", and different types of analysis match different P/ Po analysis range.

Under different test modes, the range of isothermal line collection and the number of points can be set. Do not modify it by yourself without knowing the sample and the operating system. If you need to modify it, please contact the manufacturers engineer. As shown in Figure 2-18



等温线采集模板

吸附	吸附控制段[P/Po]	控压	投气量[KPa]	间隔[P/Po]	间隔误差	平滑	最大体积量	平衡条件
	0.0E+00 -> 1.0E-08	×	5.000	×	×	×	×	时间≥600[秒]
	1.0E-08 -> 2.0E-01	×	5.000	×	×	×	×	斜率<0.050 或 时间≥150[秒]
	2.0E-01 -> 1.0E+00	√	5.000 -> 5.000	5.0E-02(16)	5%	×	×	斜率<0.050
	1.0E+00 -> 3.0E-01	×	5.000	×	×	×	×	斜率<0.050
	3.0E-01 -> 4.0E-01	×	5.000	×	×	×	×	斜率<0.050
	4.0E-01 -> 4.0E-01	×	5.000	×	×	×	×	斜率<0.050
	4.0E-01 -> 5.0E-01	×	5.000	×	×	×	×	斜率<0.050
	5.0E-01 -> 6.0E-01	×	5.000	×	×	×	×	斜率<0.050
	6.0E-01 -> 7.0E-01	×	5.000	×	×	×	×	斜率<0.050

graph 2-18

② Sample information, as shown in Figure 2-19

In this interface, the sample name and weight (weight after degassing) should be entered at least, and other information can be filled in optionally.

样品信息

名称：Smp1

重量[g]：1.000000

烘干温度[℃]：110.0

烘干时长[H:M]：12 ： 0

操作员：

样品来源：

备注(Ctrl+Enter=换行)

graph 2-19

The sample name can be modified at will, but special characters are not allowed, such as:? / * # $!

③ **Create a new file name**

文件名

Port1

□ 更新报告文件

graph 2-20

This is the file name automatically generated after the analysis ends. When there is no modification, the format saved in the database is "Port1-2020-12-18-1" (i.e., analysis station 1-current time-day test times).

Select the lock ⚓ " key"	**Release the lock ⚓ " key"**
The background of the lock key becomes yellow: the file name and sample name are stored in consistency	The background of the lock key becomes blue: The file name can be modified at will, but there are no special characters, such as:?/ *#$!

(2) Set public parameters

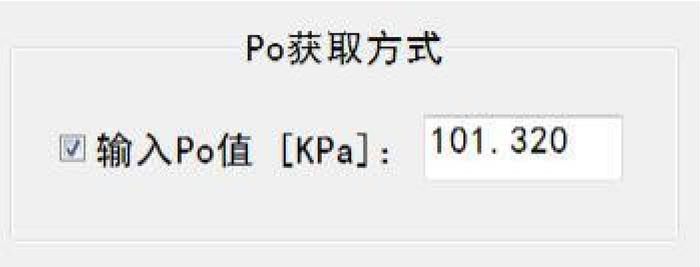
graph 2-21

① Here, you only need to select the type of adsorbate. The system defaults to nitrogen at 77K. When using other adsorbates, select from the drop-down menu as shown in Figure 2-22

吸附质选择： Nitrogen-77.35K

graph 2-22

② When "room temperature adsorption" is needed, only the Po acquisition mode needs to be checked. The adsorption quality parameters and adsorption isothermal parameters can be modified by default, and can be saved as a library file for direct use next time.

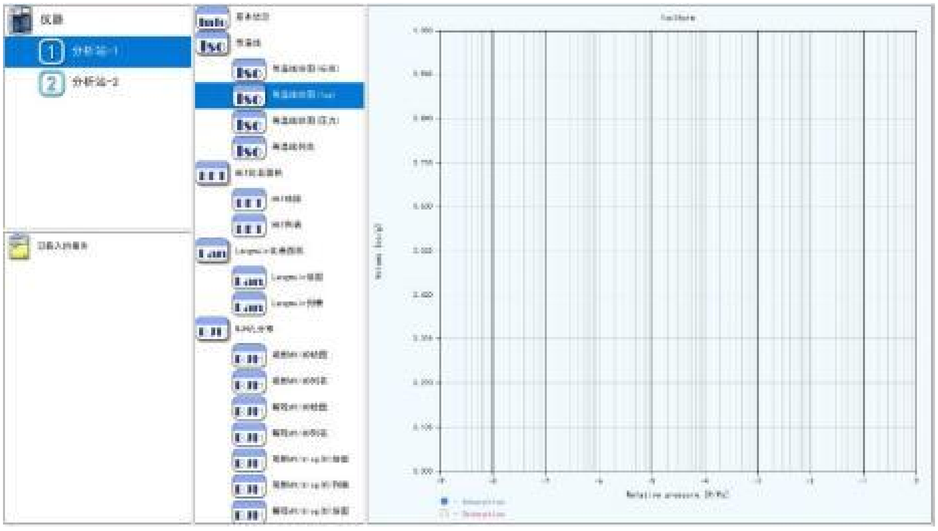


Po获取方式

☑ 输入Po值 [KPa]： 101.320

graph 2-23

3. Report module



graph 2-24

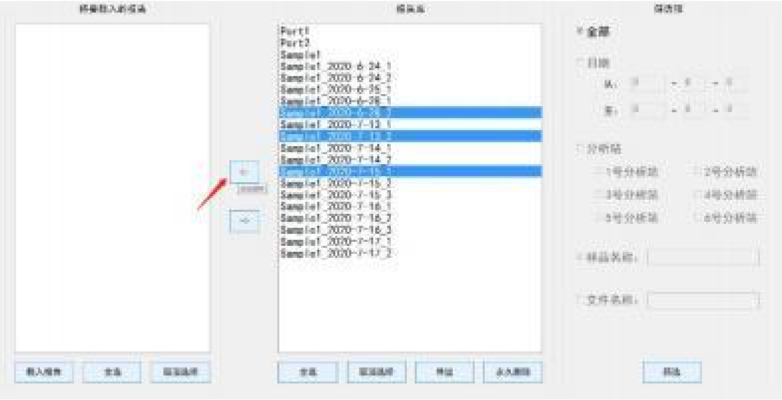
(1) Instrument-analytical station

Show the model spectrum of each analysis station in the current main interface test window. For example, sample basic information, isotherm, BET, Langmuir, BJH, etc.

(2) Review previous test reports

Click the lower left corner of the interface to load" "Buttons can load previous files from the database.

① In the selected state, click "←" to add selection, and add the target file to the report window that is being loaded. Click "载入报告", the target files are displayed below the loaded report, and you can view them by clicking on each one.



Port1
Port2
Sample1
Sample1_2020-6-24_1
Sample1_2020-6-24_2
Sample1_2020-6-25_1
Sample1_2020-6-28_1
Sample1_2020-6-28_2
Sample1_2020-7-13_1
Sample1_2020-7-13_2
Sample1_2020-7-14_1
Sample1_2020-7-14_2
Sample1_2020-7-15_1
Sample1_2020-7-15_2
Sample1_2020-7-15_3
Sample1_2020-7-16_1
Sample1_2020-7-16_2
Sample1_2020-7-16_3
Sample1_2020-7-17_1
Sample1_2020-7-17_2

graph 2-25


已载入的报告

Sample1_2020-6-28_2

Sample1_2020-7-13_2

Sample1_2020-7-15_1

graph 2-26

(3) Set up model related parameters

Click Settings Press the key to open the model Settings window, you can re-edit the parameters of each model, and then click Hit save and close.

P/Po: 0.000000 – 0.000000

厚度[nm]: –

厚度方程: Halsey

比表面积校准系数: 0.000000

graph 2-27

Chapter 3 Analysis of samples

(一) Preparations before analysis

To ensure the accuracy and repeatability of the analysis, preparations should be made in the following order and steps.

Clean and label

sample tubes for

sample weighing

Sample degassing

Install sample tubes

1. Clean and label the sample tube

The sample tube and filling rod should be cleaned and dried before use.

⑴ First confirm that the ultrasonic cell is clean, add about 500 ml of hot water, add 5 ml of detergent or surfactant, put the sample tube and filling rod in and ultrasonically clean for about 15 minutes (see Figure 3-1).

graph 3-1

⑵ Put on rubber gloves to remove the sample tube and clean the inside of the tube with a dedicated brush. ⑶ Clean the sample tube with alcohol or acetone.

⑷ Clean the sample tube with distilled or deionized water (see Figure 3-2).

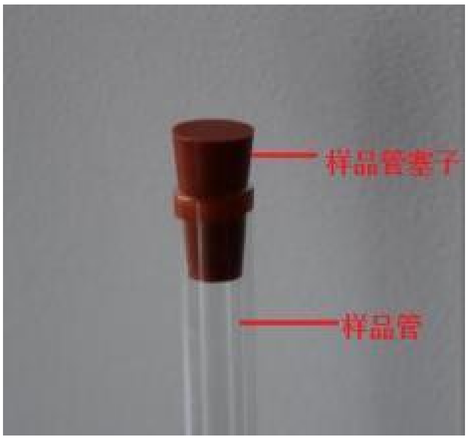
graph 3-2

⑸ Place the sample tube and filling rod on the shelf to dry naturally or use an oven or vacuum oven, set 110 degrees and 2 hours of drying for standby.

⑹ Clean the plug with a clean silk cloth and install it on the sample tube.

⑺ Label the sample tube and stopper.

⑻ Use a plug to block the sample tube (see Figure 3-3).



graph 3-3

2. Sample weighing and dehydration

Refer to "Use of degassing stations" (see page 9) for the weighing and dehydration of samples.

3. Install sample tube

⑴ Remove the plug from the tube.

⑵ Fill the rod and place the sample tube and rod horizontally (see Figure 3-3).

graph 3-3

Installing a filling rod vertically downward will break the sample tube.

⑶ After loading the filler rod, install the nut, two seals and the filter plug (not required for powder samples) (see Figure 3-4)

graph 3-4

⑷ Put the sample tube on the analysis station, push it up tightly, keep the sample tube perpendicular to the ground, hold the bottom of the sample tube with one hand, tighten the nut with one hand, and then turn it half a circle to one circle with a wrench.

(二) Measurement parameter setting

Refer to section (5) of Chapter II, "2. Analysis module" for use.

1. Use system templates

(1) Open the software, click each analysis station to enter the parameter setting interface, click load to open the system template, as shown in Figure 3-5

等温线采集模板

吸附	吸附控制段[P/Po]	控压	投气量[KPa]	间隔[P/Po]	间隔误差	平滑	最大体积
⚙	0.0E+00 -> 1.0E-08	×	3.000	×	×	×	×
⚙	1.0E-08 -> 2.0E-01	×	3.000	×	×	×	×
⚙	2.0E-01 -> 3.0E-01	×	3.000	×	×	×	×
⚙	3.0E-01 -> 4.0E-01	×	3.000	×	×	×	×
⚙	4.0E-01 -> 5.0E-01	×	3.000	×	×	×	×
⚙	5.0E-01 -> 6.0E-01	×	3.000	×	×	×	×
⚙	6.0E-01 -> 7.0E-01	×	3.000	×	×	×	×
⚙	7.0E-01 -> 8.0E-01	×	3.000	×	×	×	×
⚙	8.0E-01 -> 9.0E-01	×	3.000	×	×	×	×

graph 3-5

(2) You can choose different configuration files, load parameters, and apply analysis parameters to each analysis station. After setting up, click the "load" item in the lower right corner to complete the loading. As shown in Figure 3-6

配置文件列表

☐ 从U盘载入

☐ 从报告文件中载入

活性炭全分析.ASCC
炭素比表面.ASCC
甲醛等温吸附.ASCC
电池正极比表面积测试.ASCC
电池负极比表面积测试.ASCC
白炭黑全分析.ASCC
石墨烯全分析.ASCC
硬脂酸镁比表面.ASCC
碳酸钙比表面积测试.ASCC
药品比表面积测试.ASCC
超微孔全分析.ASCC
金属氢化物全分析.ASCC
钛白粉比表面.ASCC
陶瓷材料模板.ASCC

载入项

- ☐ 全选
  - ☐ 分析参数
  - ☐ 样品信息
  - ☐ 输出文件名
  - ☑ 等温线采集控制
  - ☐ 吸附选项
  - ☐ 报告设置

选择配置的分析站

- ☑ 全选
  - ☑ 分析站-1
  - ☑ 分析站-2
  - ☑ 分析站-3
  - ☑ 分析站-4

graph 3-6

(3) Modify the basic parameters of the sample, such as the name and weight of the sample (the weight of the sample after degassing), and create a new file name for this sample storage, as shown in Figure 3-7. If you need to keep the sample name and file name consistent, you can choose the lock key as shown in Figure 3-8

The file name is automatically saved according to the current test date when saving, and the sample name and file name should not contain special characters, such as: ? /

*** # $!**

样品信息

名称： Sample1

重量[g]： 1.000000

烘干温度[℃]： 0.0

烘干时长[H:M]： 0 ： 0

操作员：

样品来源：

备注(Ctrl+Enter=换行)

文件名

Sample1

☐ 更新报告文件

graph 3-7

样品信息

名称： Sample1

重量[g]： 1.000000

文件名

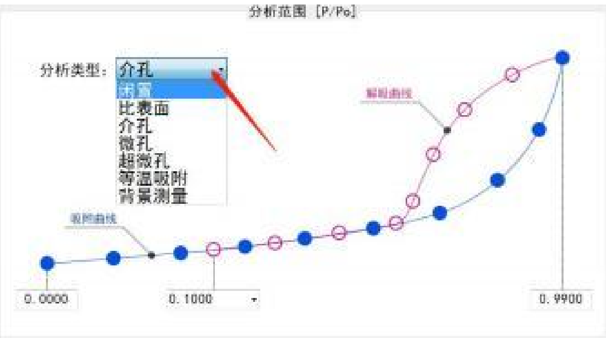
Sample1

报告文件

graph 3-8

Select the lock ⚓ " key"	Release the lo⚓ " key"
The background of the lock key becomes yellow: the file name and sample name are stored in the same way	The background of the lock key becomes blue: the file name can be modified arbitrarily, but special characters cannot be used, such as?/ * #$!

(4) Select the type of analysis, as shown in Figure 3-8



分析范围 [P/Po]

分析类型： 介孔

比表面

介孔

微孔

超微孔

等温吸附

背景测量

graph 3-9

(5) According to the above method, set up the corresponding parameters of each analysis station that needs to be worked, click save and close to return the main interface. After the preparation is completed, click the "start" key to start.

分析站-1

分析类型：介孔
样品名称：Sample1
样品重量：1.000000 [g]

graph 3-10

2. Custom templates

(1) Open the software and click Analysis Station 1 to enter the parameter setting interface, as shown in Figure 3-11

graph 3-11

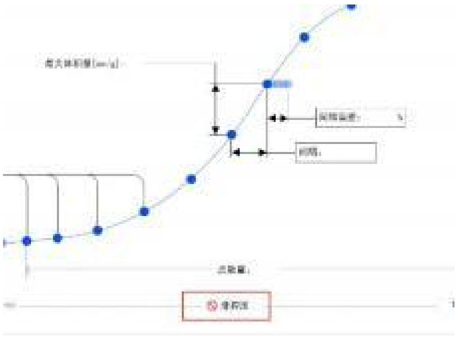
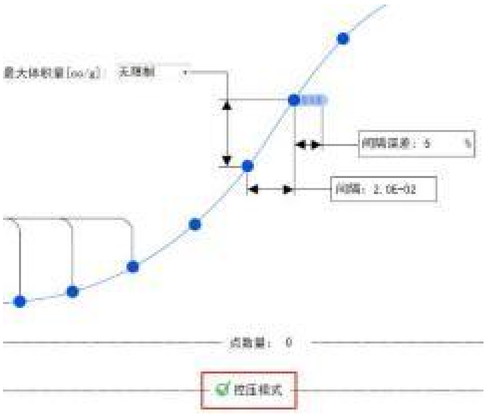
(2) Set the basic parameters and analysis type of samples, and set them according to the above system template.

(3) Set up isothermal line collection template, and add, modify or delete selected segments according to the analysis type.

等温线采集模板

吸附	吸附控制段[P/Po]	控压	投气量[KPa]	间隔[P/Po]	间隔误差	平滑	最大体积量	平衡条件
	0.0E+00 -> 1.0E-08	×	5.000	×	×	×	×	时间≥600[秒]
	1.0E-08 -> 2.0E-01	×	5.000	×	×	×	×	斜率<0.050
	2.0E-01 -> 3.0E-01	×	5.000	×	×	×	×	斜率<0.050
	3.0E-01 -> 4.0E-01	×	5.000	×	×	×	×	斜率<0.050
	4.0E-01 -> 5.0E-01	×	5.000	×	×	×	×	斜率<0.050
	5.0E-01 -> 6.0E-01	×	5.000	×	×	×	×	斜率<0.050
	6.0E-01 -> 7.0E-01	×	5.000	×	×	×	×	斜率<0.050
	7.0E-01 -> 8.0E-01	×	5.000	×	×	×	×	斜率<0.050
	8.0E-01 -> 9.0E-01	×	5.000	×	×	×	×	斜率<0.050

graph 3-12

Non-pressure control mode (Constant air supply increment/decrement control)	**Pressure control mode** (Po incremental/Po precise control)
 最大体积量[cc/g] 间隔误差： % 间隔： 点数量： 非控压	 最大体积量[cc/g]： 无限制 间隔误差： 5 % 间隔： 2.0E-02 点数量： 0 控压模式
Satisfy most material analysis	**The number of data points of isothermal line can be customized to accurately P/Po** **margin**

① Double-click any isothermal line section and set adsorption/desorption parameters. Two test modes are provided to meet the testing requirements of different materials.

② Non-pressure control mode setting. As shown in Figure 3-13

According to the characteristics of the adsorption performance of the target material, it is customized to be put in, and the initial air input is set to 5 KPa, and the maximum air input does not exceed 15 KPa.

投气量[KPa]

□自动设置投气量

初始：5.000

最大：5.000

吸附与解吸平衡条件

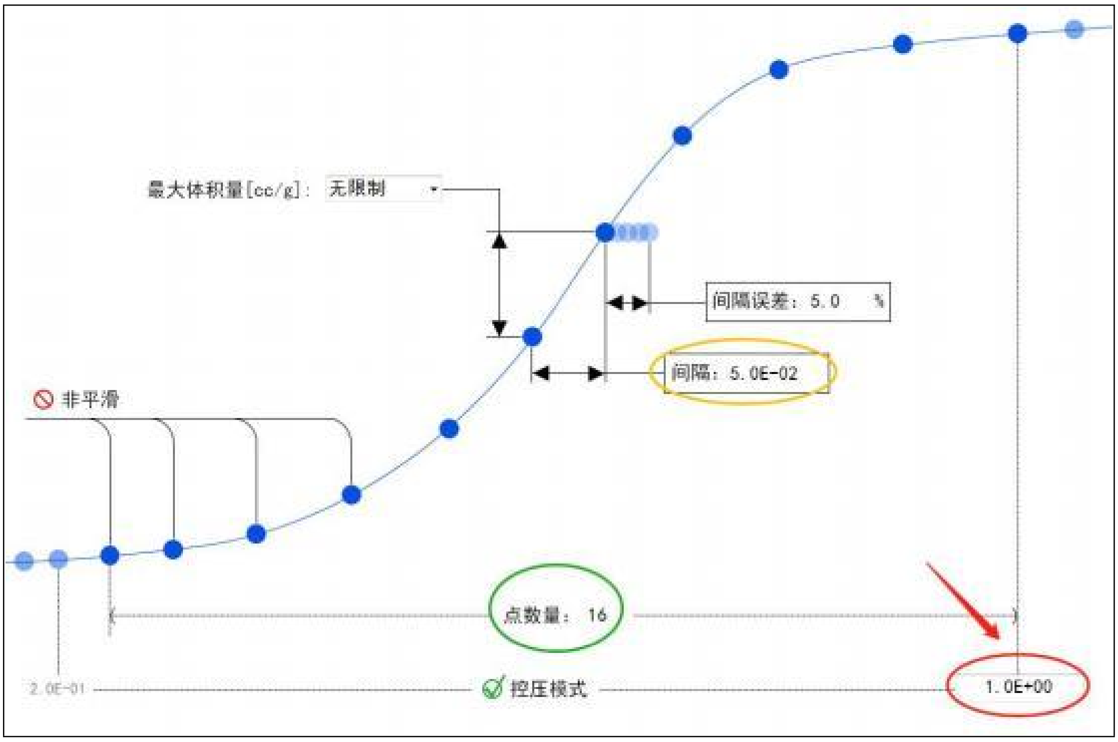
☑自动斜率控制：0.050000

☑平衡时间[秒]：200

□斜率与时间必须同时满足

graph 3-13

③ Pressure control mode setting. The interval error is set to 3-5%, as shown in Figure 3-14.



graph 3-14

- Enter the P/ Po end point (red circle part), such as "1.0E+00" in the figure, and enter the number of input points (green circle part) "16", then the P/ Po interval is automatically calculated.
- Enter the P/Po end point (red circle part), such as "1.0E+00" in the figure, and enter the interval (yellow circle part) P"5.0E-02, and the number of points will be automatically calculated.
- Check automatic setting of air supply and automatic slope control

投气量[KPa]

☑自动设置投气量

初始：5.000

最大：5.000

吸附与解吸平衡条件

☑自动斜率控制：0.050000

☐平衡时间[秒]：150

☐斜率与时间必须同时满足

- As above, set up the selected segment and save it

(4) Save custom templates. Select Save as and store custom templates.

吸附	吸附控制段[P/Po]	控压
	0.0E+00 -> 1.0E-08	×
	1.0E-08 -> 2.0E-01	×
	2.0E-01 -> 3.0E-01	×
	3.0E-01 -> 4.0E-01	×
	4.0E-01 -> 5.0E-01	×
	5.0E-01 -> 6.0E-01	×
	6.0E-01 -> 7.0E-01	×
	7.0E-01 -> 8.0E-01	×
	8.0E-01 -> 9.0E-01	×

保存选项

☐保存至U盘

文件名

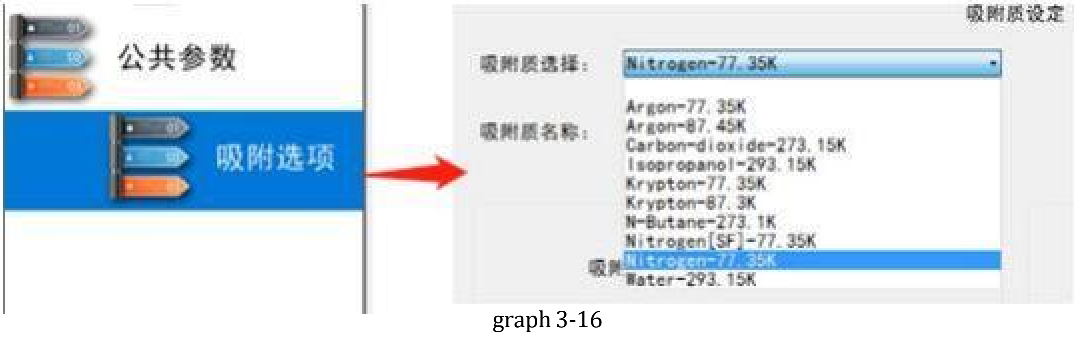
氧化物比表面积

配置文件列表

6.ASCC
实验.ASCC
标样测试.ASCC
氧化物全孔分析.ASCC

graph 3-15

(5) Public parameter setting. Open the public parameter interface, select the adsorbate and adsorption temperature, save and close.



吸附质设定

吸附质选择：Nitrogen-77.35K

吸附质名称：

Argon-77.35K
Argon-87.45K
Carbon-dioxide-273.15K
Isopropanol-293.15K
Krypton-77.35K
Krypton-87.3K
N-Butane-273.1K
Nitrogen[SF]-77.35K
Nitrogen-77.35K
Water-293.15K

graph 3-16

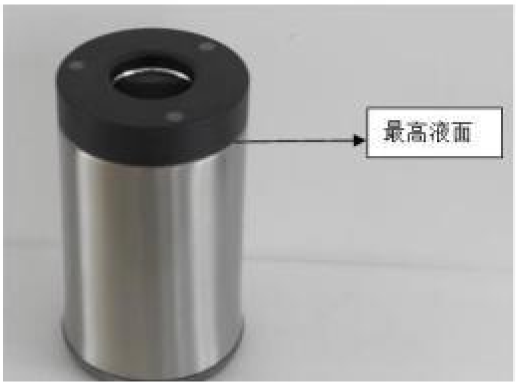
(6) Return to the main interface, and after the preparation is completed, click "Start" to start.

3. Add liquid nitrogen to the Dewar flask

(1) Wear protective equipment, wear protective glasses and put on thermal gloves if conditions permit.

(2) When adding liquid nitrogen to the Dewar flask, add it slowly to reduce the "thermal shock" of the Dewar flask and prevent the splashing of liquid nitrogen, so that the height of the liquid nitrogen in the liquid nitrogen cup is as shown in Figure 3-16. Note that you must be careful when adding liquid nitrogen to prevent low temperature

empyrosis.



graph 3-16

(3) Place the Dewar bottle on the lifting tray.

(3) Start the analysis

1. Air cylinder

Open the main valve of the helium and nitrogen cylinders, the output pressure of the pressure reducing valve is 0.3 MPa. Note that when the output pressure of the pressure reducing valve exceeds 0.5 MPa, the gas line should be removed under the condition that the main pressure valve of the cylinder is closed, and the gas in the pressure reducing valve should be completely drained. Then reinstall the gas line and readjust the pressure distribution (the output pressure should be 0.3~0.5 MPa).

2. Open the vacuum pump

Turn on the power switch of the mechanical pump and molecular pump.

3. Start analysis

Click the "Start" button on the main screen to start the analysis. The isotherm drawing and real -time parameters during the test process will be displayed on the main screen.

graph 3-17

graph 3-18

(4) End analysis

1. End operation

After the analysis is completed, the default data storage location is:

计算机 ▸ D盘 (D:) ▸ iPoreClient2020 ▸ SAVE

After sample testing, the total valve of the gas cylinder and the vacuum pump should be closed.

(1) Close the total pressure valve of the gas cylinder. If the gas continues to be used, the pressure dividing valve does not need to be closed when the gas line is not removed.

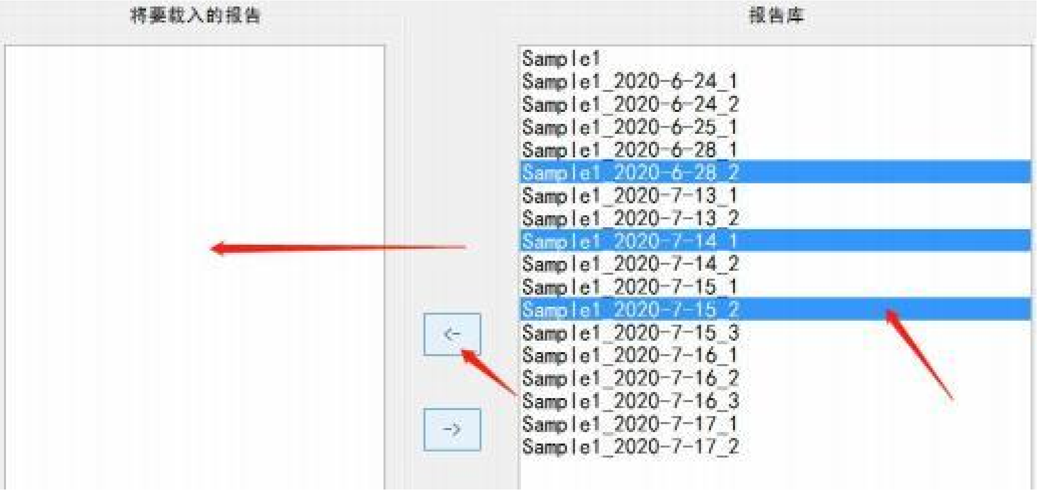
(2) Turn off the power of the instrument and recover the remaining liquid nitrogen (discard the ice).

(3) Put clean sample tubes or plugs on the instrument analysis station to prevent air from entering the instrument and causing contamination.

2. View the test report on the computer control end

(1) In the report tool group, click the load report butt "Open the report library, select the
the bottom left corner target report and

Press "←" to move the report to the load report window, then click load report; if you cancel the load, select the report and click "→" as shown in Figure 3-19 and 3-20


将要载入的报告

报告库

Sample1
Sample1_2020-6-24_1
Sample1_2020-6-24_2
Sample1_2020-6-25_1
Sample1_2020-6-28_1
Sample1_2020-6-28_2
Sample1_2020-7-13_1
Sample1_2020-7-13_2
Sample1_2020-7-14_1
Sample1_2020-7-14_2
Sample1_2020-7-15_1
Sample1_2020-7-15_2
Sample1_2020-7-15_3
Sample1_2020-7-16_1
Sample1_2020-7-16_2
Sample1_2020-7-16_3
Sample1_2020-7-17_1
Sample1_2020-7-17_2

graph 3-19


将要载入的报告

Sample1_2020-7-14_1
Sample1_2020-7-15_3
Sample1_2020-7-17_1

graph 3-20

(2) You can screen the target report according to different selection criteria such as analysis station/test date/sample name/file name, etc., as shown in Figure 3-21

筛选项

☐ 全部

☐ 今天

☐ 日期

从：0 - 0 - 0

至：0 - 0 - 0

☐ 分析站

☐ 1号分析站 ☐ 2号分析站

☐ 3号分析站 ☐ 4号分析站

☐ 5号分析站 ☐ 6号分析站

☐ 样品名称：

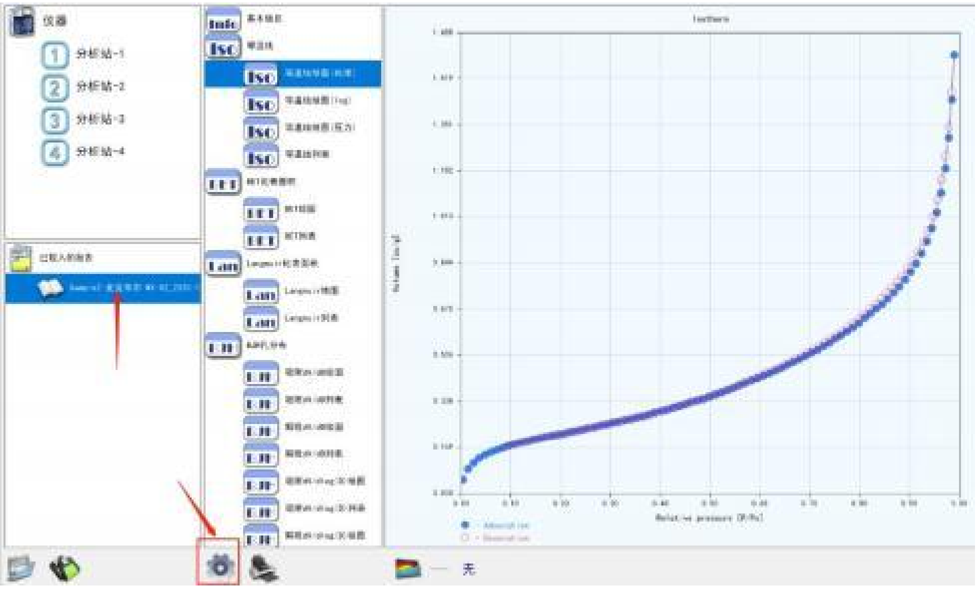
☐ 文件名称：

筛选

graph 3-21

(3) In the loaded report interface, you can view the results of each model. If you need to modify the model parameters, you can click the Settings button to enter the modification. As shown in Figure 3-22

Note: This step can be ignored and data can be processed using data processing software according to "Chapter 4 Data Management".



graph 3-22

Chapter IV Data Management

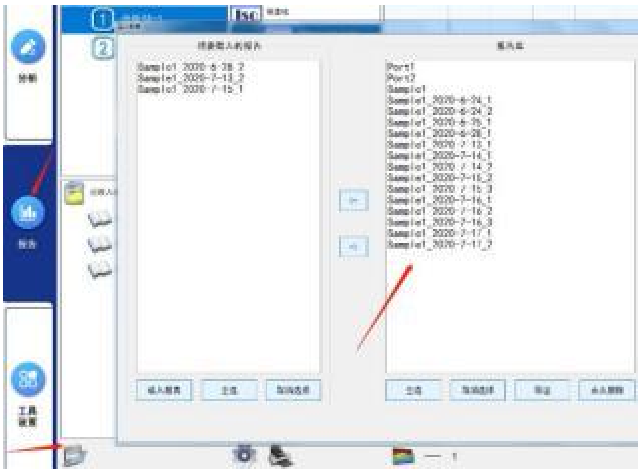
(1) Data export

1. Create a target folder in the USB drive

Before exporting, you need to create a folder named "iPoreReport" in the USB drive that copies the data, i.e., "lip oree port", and insert the USB drive into the USB port of the instrument.

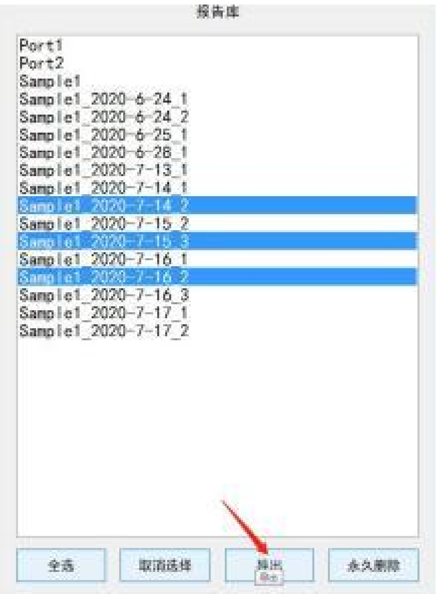
2. Export the source file under the report module

(1) Click the report module, click the "Load report" key in the lower left corner to open the report library (see Figure 4-1).



graph 4-1

(2) You can select a single or multiple library files. Then click the "Export" button as shown in Figure 4-2



Port1
Port2
Sample1
Sample1_2020-6-24_1
Sample1_2020-6-24_2
Sample1_2020-6-25_1
Sample1_2020-6-28_1
Sample1_2020-7-13_1
Sample1_2020-7-14_1
Sample1_2020-7-14_2
Sample1_2020-7-15_2
Sample1_2020-7-15_3
Sample1_2020-7-16_1
Sample1_2020-7-16_2
Sample1_2020-7-16_3
Sample1_2020-7-17_1
Sample1_2020-7-17_2

graph 4-2

(3) You can screen the target files according to date, analysis station, sample name, file name, etc. from the database. After the source file is exported successfully, there will be relevant prompts.

graph 4-3

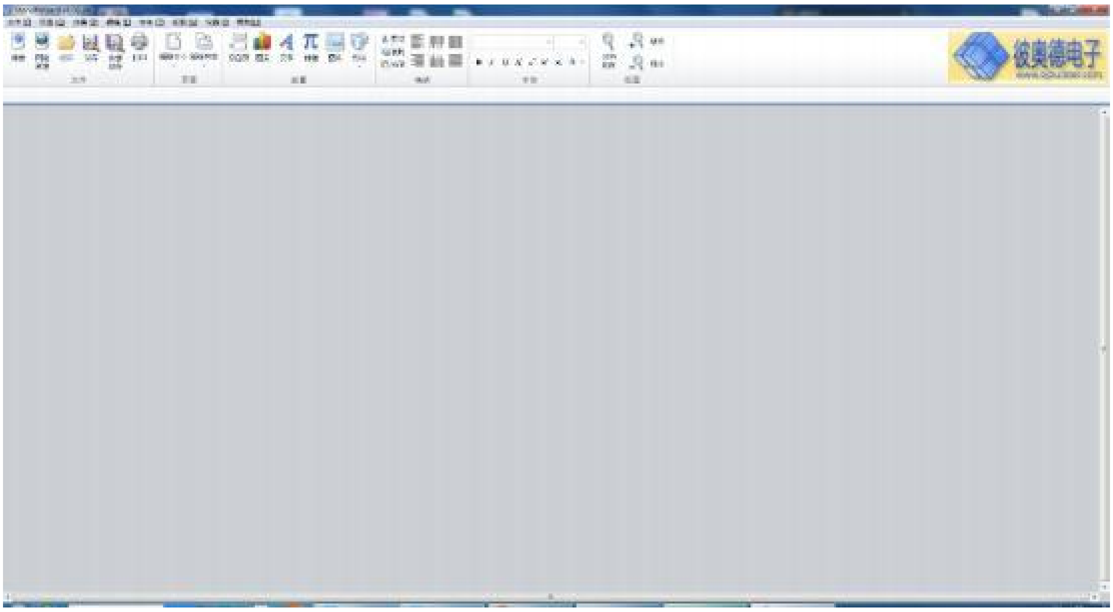
(4) Solutions to failed file export.

 找不到目标文件夹	 导出文件失败！！！
In the USB drive, there is no newly created folder named "iPoreReport" or the file name is wrong. Create or modify it again.	There are source files with the same file name in the USB drive, Modify the file name in the U drive or check "Replace existing files"  □ 替换已存在的文件

(二) Data extraction

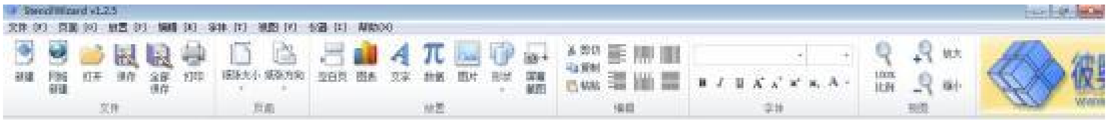
1. Open the data processing software

(1) Double-click the desktop analysis software shortcut icon "", Open the software interface. Figure 4-4



graph 4-4

(2) Menu bar introduction



graph 4-5

lNew: Search for newly generated test files and open or edit them; you can select the "New" shortcut button or select File-New

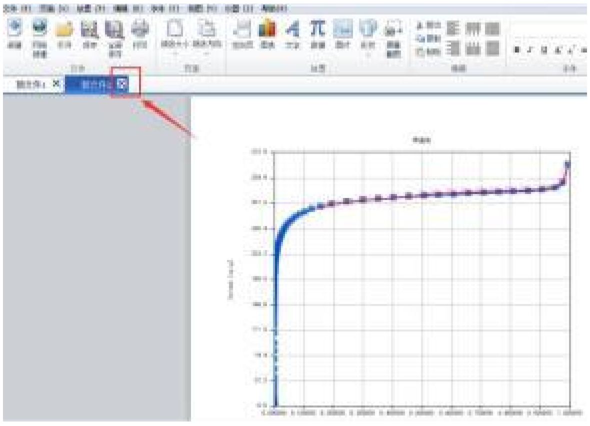
lOpen: retrieve the edited file and open it again; you can choose the "New" shortcut button or file-open

lNew network: Internet remote control is realized through fixed IP setting

l Save: Save the current edited file as a new file, which can be saved as ".MFAR", ".RAW", "excel" format

lSave all: save the current edited file as a new file in sequence and continuously

lClose: Close the current file, Figure 4-6



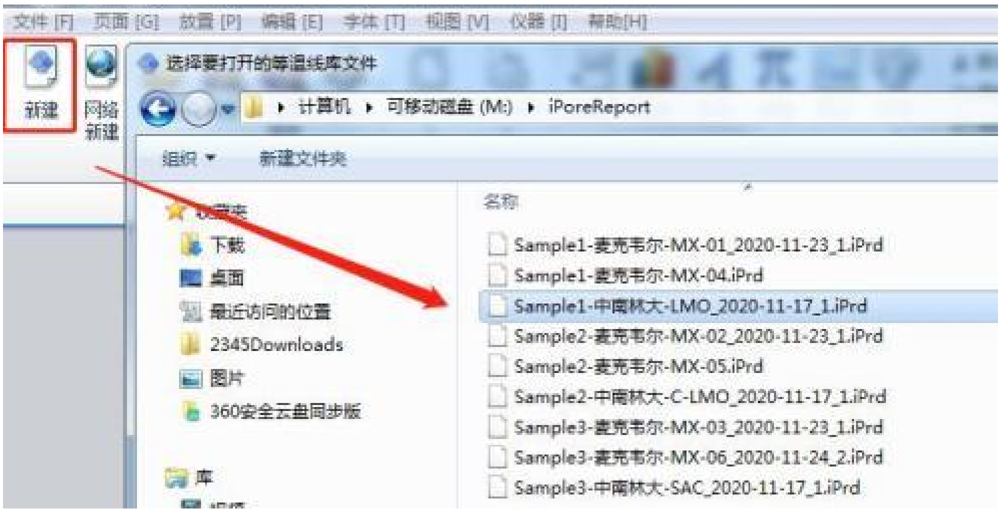
graph 4-6

lPrint: Allows you to print table data, charts or entire reports

lPage: Set the paper size and direction

2. Open the target source file

Click the "New" button on the software, find and open the "iPoreReport" folder in the U disk, and double-click the source file to be processed with the left mouse button, "XXX. iPrd" Figure 4-7.



graph 4-7

(3) Data editing

After clicking "New" to import the source file, the report settings interface will automatically open. At this point, you will enter the "Basic Parameters" window in the report settings. Here, you can switch between different analytical models, edit all types of sample parameters in the physical adsorption instrument, and select to generate analytical models. After completing the settings of each window model, please click "OK"; otherwise, you will need to re-edit.

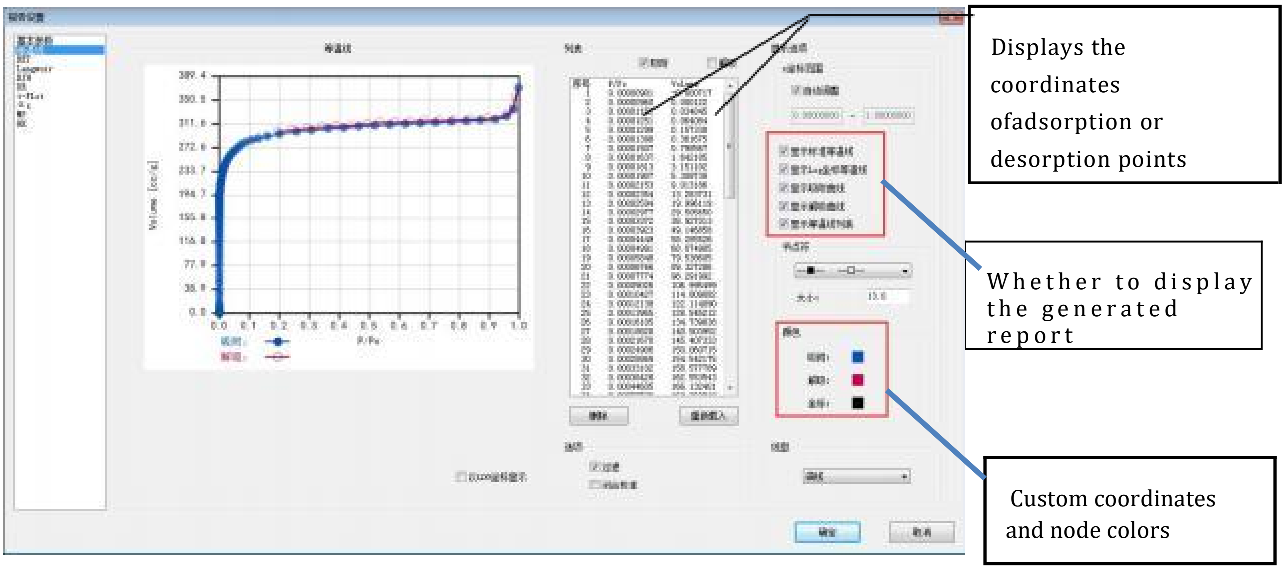
1. Basic parameters window

Here you can edit the basic parameters of the sample, edit the header and footer, etc. When the low temperature nitrogen adsorption method is used by default, please do not modify the three parameters "adsorbate, adsorbate area, gas-liquid volume conversion coefficient". See Figure 4-8

graph 4-8

2. Isothermal window

You can view the values of adsorption and desorption nodes on isotherms; edit the coordinates of isotherms, adsorption, and desorption lines; generate or remove isotherm line plots; select connection lines for isotherm line types, and also filter adsorption and desorption data nodes to generate new isotherm line spectra. Figure 4-9



Displays the coordinates ofadsorption or desorption points

Whether to display the generated report

Custom coordinates and node colors

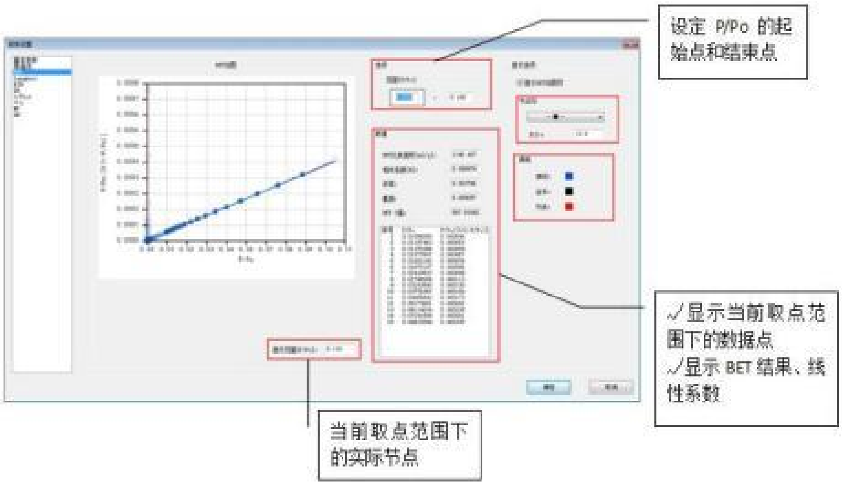
graph 4-9

(1) The x-coordinate of the isotherm: relative pressure [P/Po], P is the actual test pressure; Po is the saturated vapor pressure of liquid nitroge

(2) Isothermal line y-coordinate: adsorption amount of material per unit mass [cc/g]

3.BET drawing window

The selectable range, node symbol and line color can be edited. The selectable range is different for different materials. Please select the point according to the actual material. Do not choose too high or too low P/ Po, otherwise the calculation result will be greatly deviated. Figure 4 -10



设定 P/Po 的起始点和结束点

✓显示当前取点范围下的数据点
✓显示 BET 结果、线性系数

当前取点范围下的实际节点

graph 4-10

(1) P/Po is in the range of 0 to 0.15

The measurement and analysis of micropore distribution can be carried out. For ultra-micropore and micropore samples, the range of P/ Po is 0~0.1, and it is necessary to have a vacuum condition of 3×10-8 pa and a high-precision pressure sensor with multiple different ranges to ensure the number of data points and accuracy in this range.

(2) P/Po is in the range of 0.05~0.30

Multi-point BET specific surface area can be tested and calculated. Multi-point BET is suitable for sample characterization under multi-layer adsorption theory, and Laugmuir specific surface area data is used for characterization under single-layer adsorption theory.

P/ Po is only applicable to mesoporous materials in the range of 0.05~ 0.30; for samples with micropores, the value range needs to be appropriately adjusted: for X zeolites, the value range is 0.005~ 0.01; for microporous materials, it is 0.005~ 0.1; for meso-microporous composite materials, it is 0.01~ 0.2; since microporous materials are closer to monolayer adsorption characteristics, Laugmuir specific surface area data can more accurately characterize microporous materials.

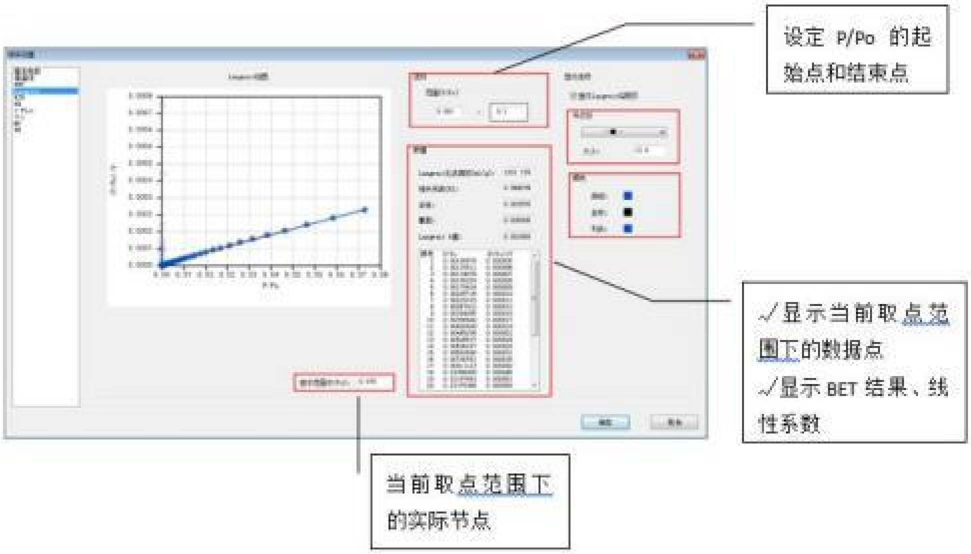
(3) P/ Po is in the range of 0.15~0.996

The mesoporous and macroporous measurements and analysis were carried out. In this relative pressure range, capillary condensation phenomenon occurred on the surface of the adsorbent channel, and the obvious characteristic of the adsorption-desorption isothermal line was the generation of "lag ring".

4.Langmuir Drawing window

The editable Langmuir algorithm can take the range of points, node symbol and line color, and Langmuir is more suitable for the characterization of microporous material

4-11



设定 P/Po 的起始点和结束点

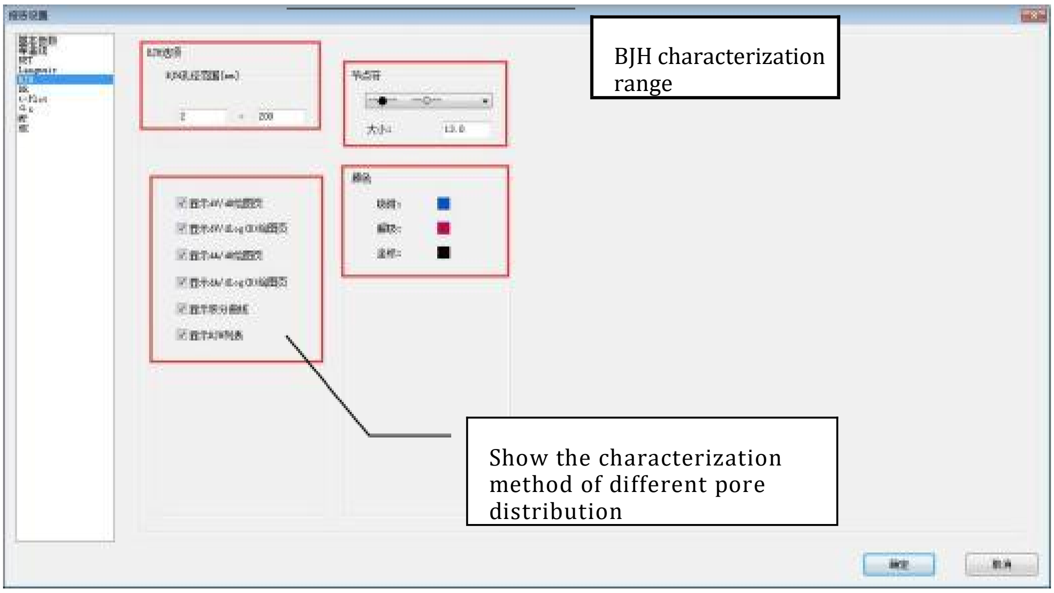
√显示当前取点范围下的数据点
√显示 BET 结果、线性系数

当前取点范围下的实际节点

graph 4-11

5.BJH selection window

- BJH can only be used for mesopores, and the columnar model is used in theoretical calculations.
- The pores are rigid, have a regular shape (such as cylindrical or slit-shaped), and the pore size distribution is narrow and well defined (i.e., H1 type hysteresis loop); there are no micropores or very large pores (a clear IV type isothermal line).
- When the aperture is <10 nm, the aperture is underestimated, and there is a 20% error when the aperture is <5 nm.



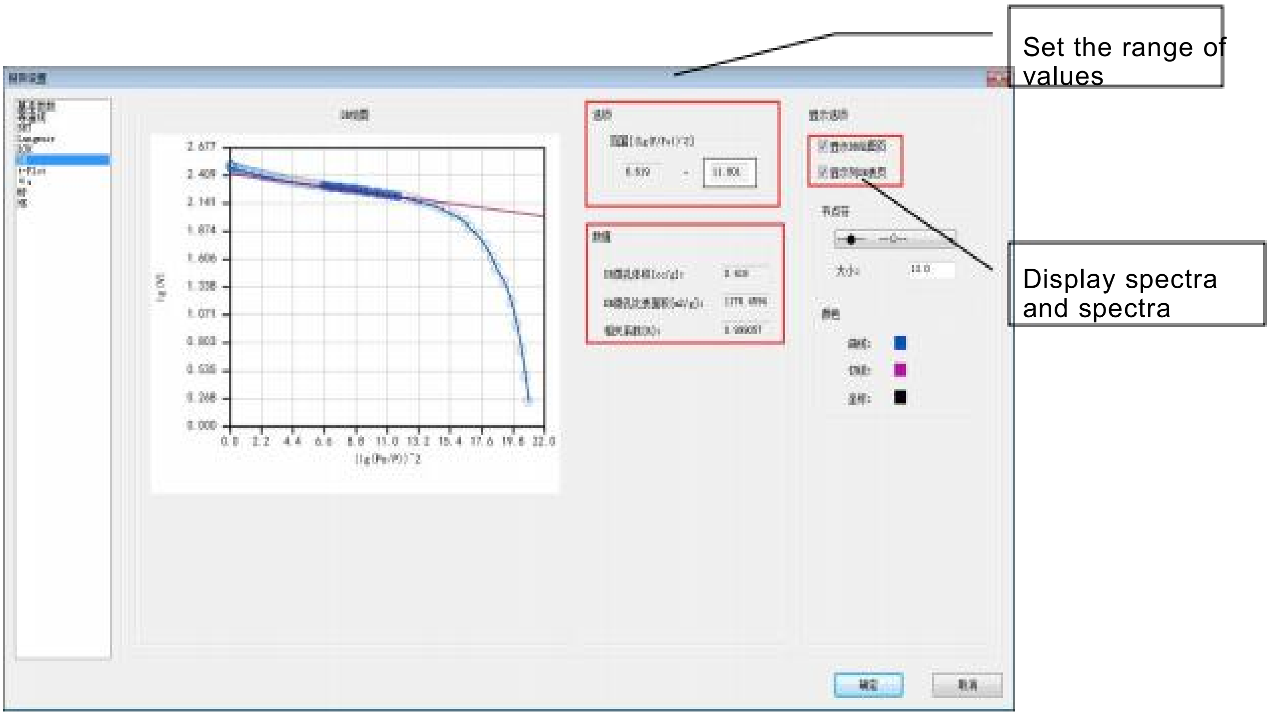
BJH characterization range

Show the characterization method of different pore distribution

graph 4-12

6.DR (Dubinin-Radushkevich) window

The DR filling theory suggests that molecules are not adsorbed on the wall of the pore layer by layer, but are It is a method for calculating the pore volume of microporous materials such as microporous activated carbon and molecular sieves. It is also used for analyzing other microporous materials.



Set the range of values

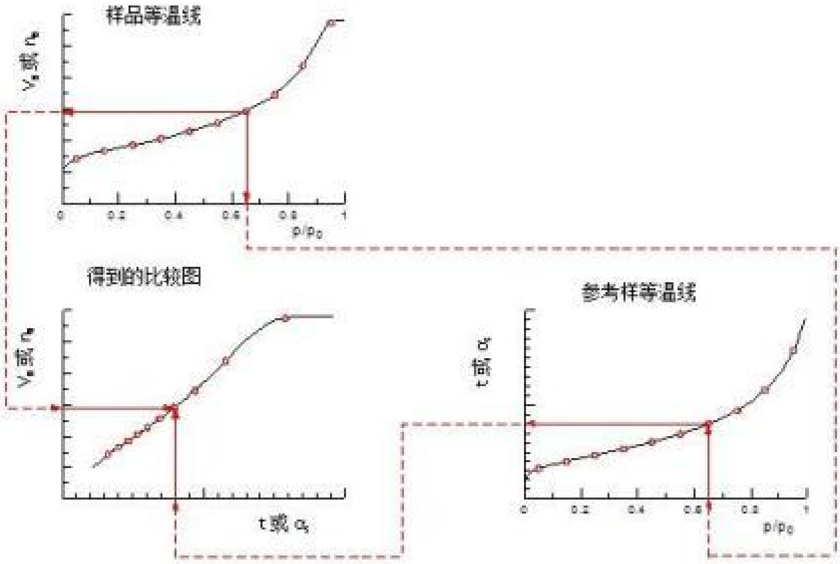
Display spectra and spectra

graph 4-13

7. t-plot/t plot window

Assumption: The chemical properties of the surface are uniform with no strong specific adsorption sites. To estimate whether micropores, mesopores, or both are present in the test samples (mainly used to characterize the presence of micropores), the adsorption isotherm extrapolation method is often applied to estimate the micropore volume, micropore volume, and surface area. The two main methods are the t-plot method and the αs-plot method, with the t-plot method being the most commonly used. The specific method involves plotting the adsorption layer thickness t against the adsorption amount V and comparing it with the non-porous adsorbent V-t curve.

It compares the experimental isotherm with the reference isotherm obtained from a non-porous solid.The reference isotherm is calculated by a mathematical expression where the adsorbent must have chemical properties similar to those of the test sample.Therefore, for different samples, non-porous reference curves, i.e., different models, should be selected.For example, for high silica zeolite, a non-porous silicon dioxide reference curve should be used.

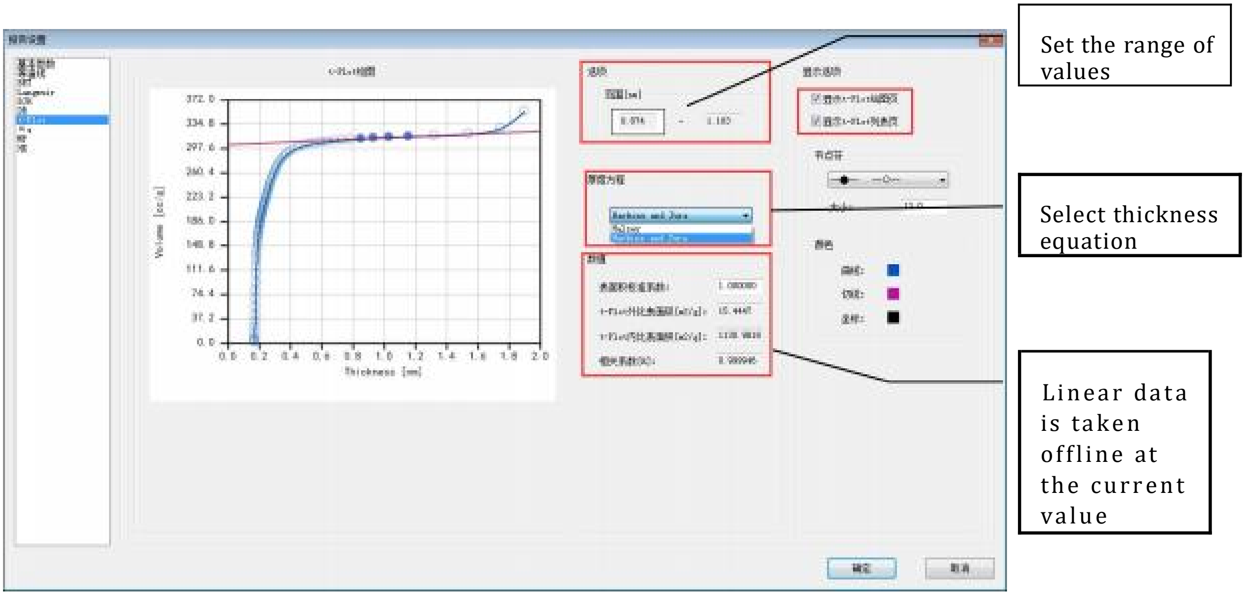


✷How to judge the rationality of t-method results?

After selecting the model and calculation points, the instrument can give the calculation value and figure. However, whether this result is reasonable and reliable is often the most easily overlooked and common analysis error. The principle of judging the result:

A, the regression line intersects with the t-curve instead of intersecting with it. If they intersect, the pressure values should be adjusted and recalculated to meet the above requirements.

B. The regression coefficient of the straight line has at least 3 nines (>0.99 9)



Set the range of values

Select thickness equation

Linear data is taken offline at the current value

graph 4-14

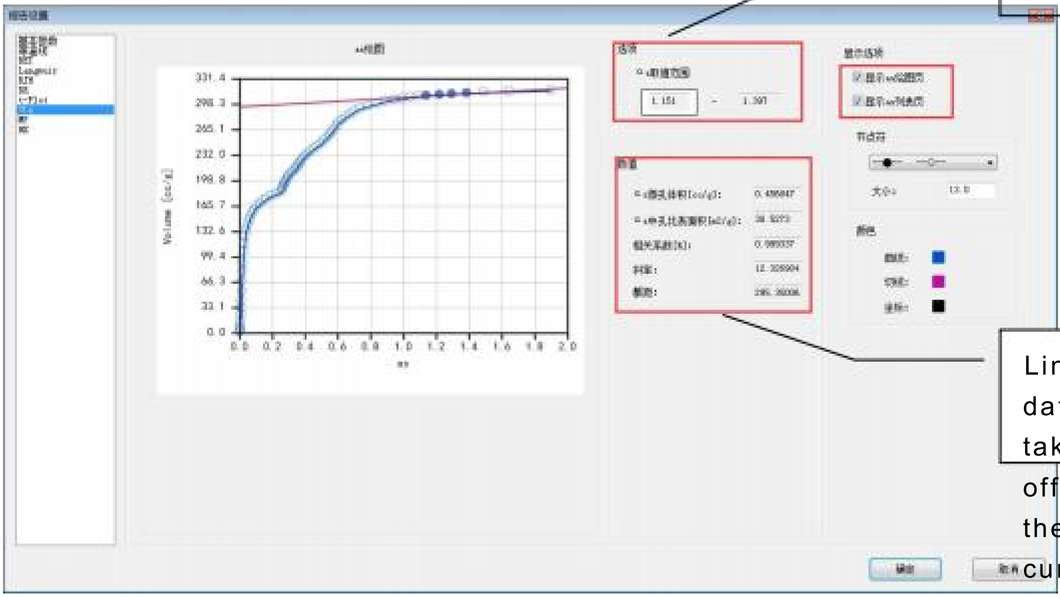
✷ Thickness equation:

A, Halsey: Similar to the application of de Boer model, it is used for the calculation model of molecular sieve and oxide. The range of pressure point value calculation is 0.4~0.6 (P/P0).

8.An as window

At p/p0=0.4, the corresponding adsorption amount V0.4 is derived from a s calculation, i.e., αs = V/V0.4. Similar to the t graph method, the difference lies in using the V-αs graph instead of the V-t graph, which can be used even when the adsorption layer thickness is unknown and can also measure adsorption beyond nitrogen

Attached matter.



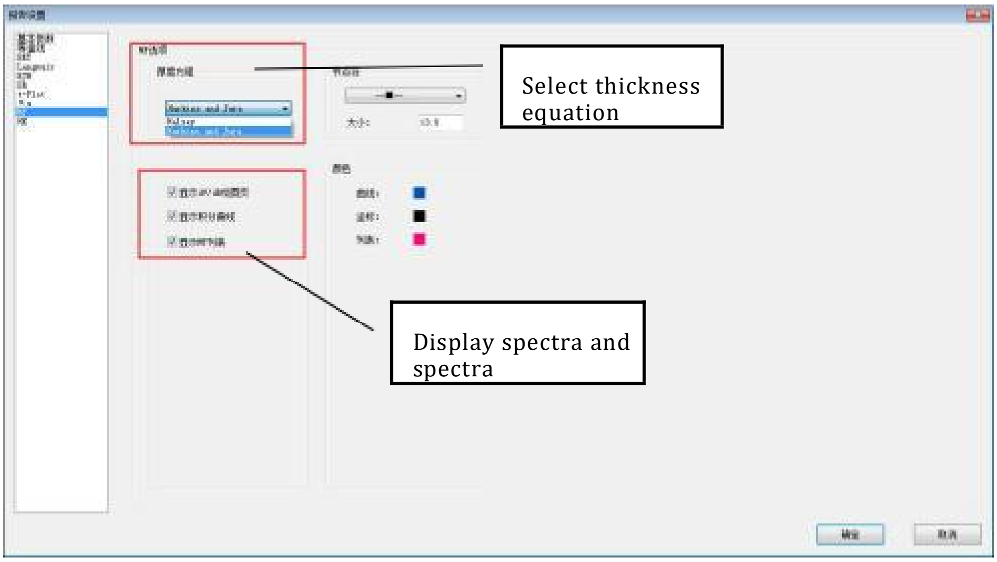
Set the range

Linear data is taken offline at the current

graph 4-15

9.MP map window

The MP method is to obtain the surface area, micropore volume and micropore distribution of microporous adsorbents by analyzing the V-t curve.



Select thickness equation

Display spectra and spectra

graph 4-16

10.HK Spectrum window

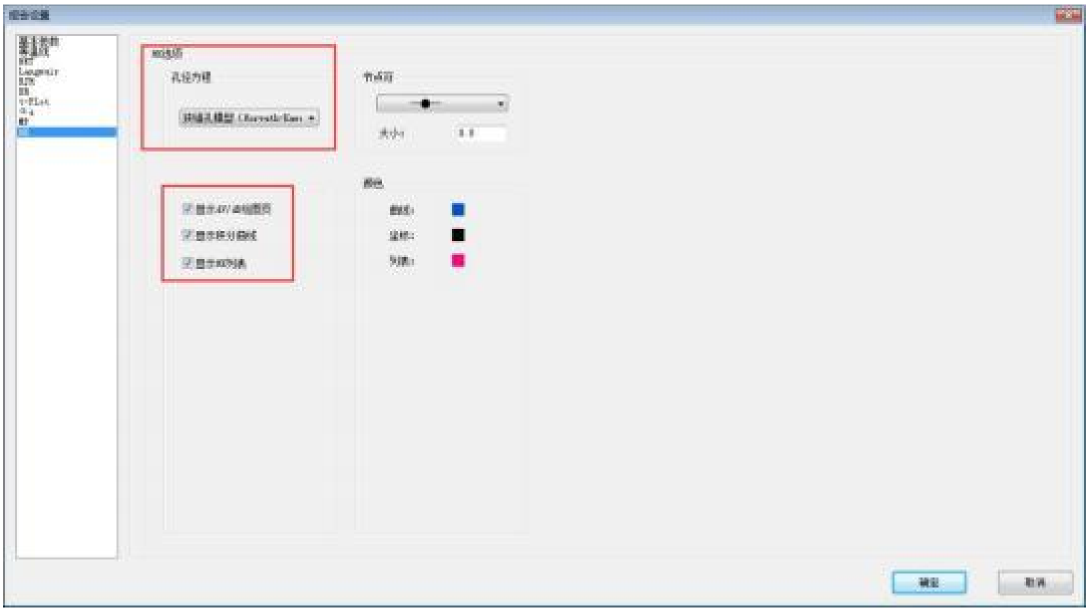
HK micropore model (including Saito & Foley correction, Cheng & Yang correction), used for micropore aperture distribution, including the following three:

Horvath-Kawazoe (HK) model: it is assumed that the pore type is slit-type micropore. It mainly targets nitrogen adsorption in slit pores within carbon molecular sieves and activated carbon.

Saito-Foley (SF) model: assumes cylindrical pore shape. Saito and Foley extended HK method to calculate effective pore size distribution by calculating isothermal line of adsorption on cylindrical pores of zeolite molecular sieve at 87K by argon.

Cheng/Yang model: spherical pore distribution model. It can be used for the spherical pores of octahedrite and 5A molecular sieve.

The HK and improved HK equations take into account the interaction between adsorbate and adsorbent but this method is significantly influenced by the selection of pore models and the values of physical parameters in the formulas. Therefore, when using these methods, it is necessary to reasonably choose pore models and equation parameters based on the types of adsorbates and microporous samples.



graph 4-17

11. Data summary

Here, you can selectively generate the display results, and check to generate them. Figure 4-18

graph 4-18

Chapter V theoretical overview

(一) Definition overview

1. Overview of surface characteristics

The surface characteristics of powder materials are very complex, especially for porous nanoscale powder materials, as the state of atoms on the surface differs significantly from that within the bulk. Therefore, the surface characteristics encompass not only the conventional surface but also the inner surfaces connected to the surface through pores. To better characterize and analyze the surface properties of materials, data analysis is categorized into two main types: specific surface area and porosity.

2. Surface feature definition

(1) Concept of specific surface area

The specific surface area refers to the surface area of a porous solid substance per unit mass, commonly measured in square meters per gram.

(2) Overview of the hole

① The concept of holes

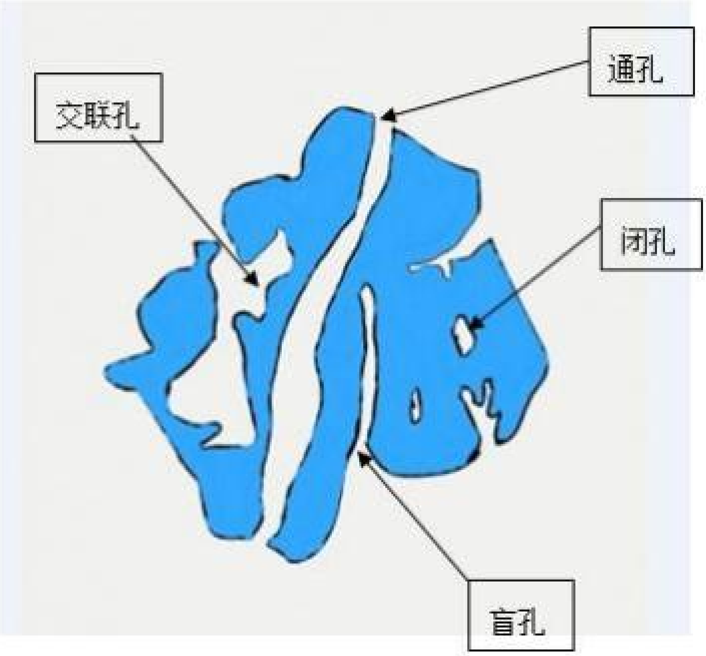
Channels or cavities within solid materials can be called holes. In addition to holes within solid materials, spaces between aggregates of solid particles (such as cracks or voids) can also be called holes.

② Classification of holes

The size of pores in porous solids (adsorbents and catalysts) is directly related to their specific surface area and the diffusion of adsorbate molecules. For characterization and analysis, the International Union of Pure and Applied Chemistry (IUPAC) later recognized that pore sizes can be classified into three categories based on their aperture width: macropores (>50nm); mesopores (2~50nm); and micropores (<2nm). Micropores are further divided into submicropores and ultra-micropores; dubinin further classifies micropores into ultra-micropores <0.7nm and submicropores between 0.7-2.0nm.

In addition to the quantitative classification in scientific research, according to the characteristics of the hole type, it is divided into: through hole, closed hole, cross-linked hole, blind hole, etc., among which cross-linked hole and blind hole include cylindrical hole, slit hole, spherical hole, etc. (see Figure 5-1)

In order to facilitate the study, the shape of the hole is simplified into two categories: slit shape and cylindrical shape.



graph 5-1

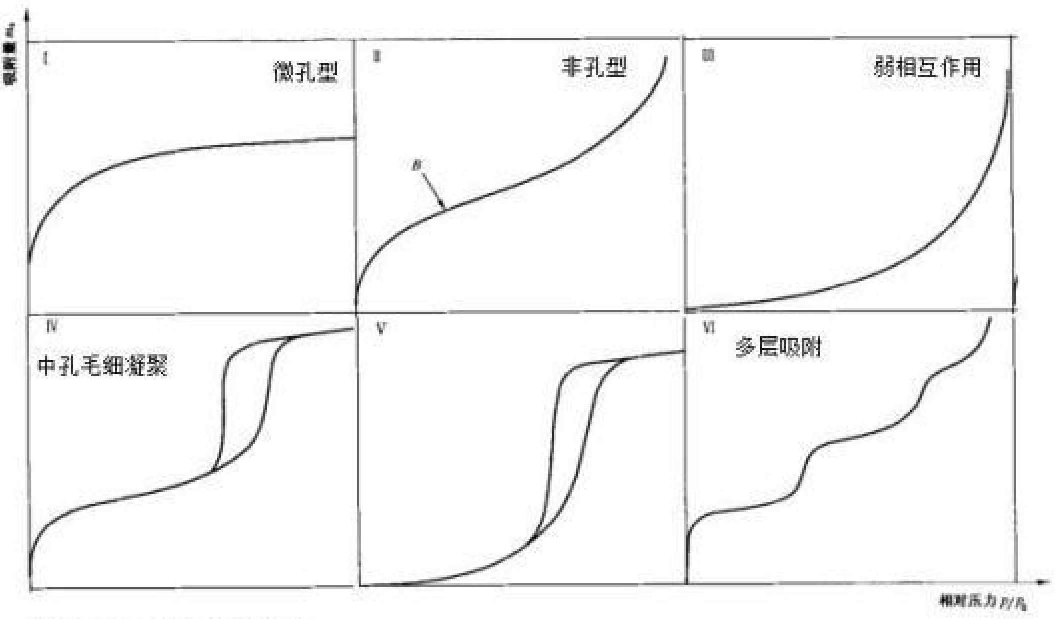
(二) Analysis methods

1. Analysis method

The characterization of surface properties is often divided into two major categories: specific surface area and porosity, among which there are various methods for analyzing and testing specific surface area, such as gas adsorption method, gas permeation method, mercury intrusion porosimetry, bubble point method, small angle X-ray diffraction, etc. Among these, the gas adsorption method is widely adopted in various industries both domestically and internationally due to its scientific testing principles, reliable testing process, and consistent test results, gradually replacing other specific surface area testing methods and becoming the most authoritative testing method recognized. The gas adsorption method (also known as static volumetric method) requires data to be calculated based on the adsorption and desorption amounts of adsorbate gases under different relative pressures (adsorption-desorption isotherms). Simply put, the gas adsorption method uses gas molecules as a measurement "scale" throughThe consumption of adsorbate molecules and the state of adsorption-desorption curve are analyzed to describe the surface characteristics of materials.

The commonly used gas adsorption method is nitrogen adsorption, under constant temperature (usually the boiling point of the adsorbate gas, with nitrogen being 77.35k), the curve showing that the adsorption amount varies with relative pressure (P/P0) is called the adsorption-desorption isotherm, which is the most important manifestation of solid material adsorption characteristics. Based on the data points of the adsorption-desorption isotherm at different relative pressure ranges, different theoretical models can be used for various surface property analyses

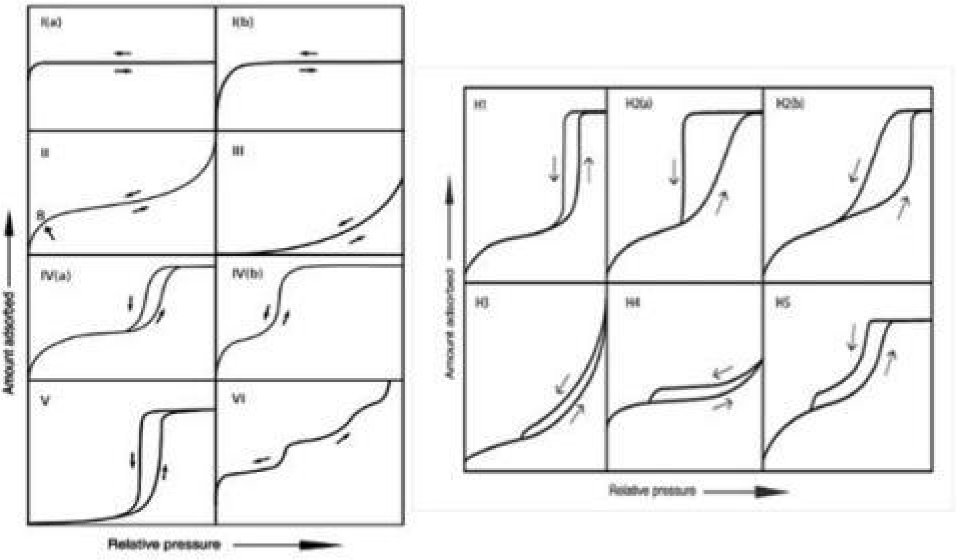
(1) Classification of isothermal lines of adsorption and desorption (see Figure 5-2 and 5-3)



注：B点为单层吸附容量结束的标记。

graph 5-2

Figure 5-2 presents the classification of physical adsorption isotherms proposed by the International Union of Pure and Applied Chemistry (IUPAC). In 2015, the IUPAC, an authoritative body in international chemistry, published new norms for physical adsorption analysis, as shown in Figure 5-3. Over the past 30 years, with the continuous synthesis of new materials such as various ordered mesoporous molecular sieves, microporous molecular sieves, metal-organic frameworks (MOFs), etc., the existing norms have become inadequate to meet current research requirements. In the new norms, the types of adsorption isotherms have been expanded from 6 to include 2 subcategories, resulting in a total of 8 types of adsorption isotherms, refining the classifications of microporous and mesoporous materials; additionally, 2 types of desorption hysteresis loops have been added.



新的吸附等温线和脱附迟滞类型

graph 5-3

The characteristic of the I-type is that in the low relative pressure region, there is a rapid increase in gas adsorption which is due to the occurrence of a micropore filling process. The subsequent horizontal or nearly horizontal plateau indicates that the micropores are already filled with no or almost no further adsorption occurring. When saturation pressure is reached, adsorbate aggregation may occur. Microporous solids with relatively small external surface areas, such as activated carbon, zeolites, and certain porous oxides, exhibit this isotherm.

Type II isotherms are generally produced by nonporous or macroscopic solids. Point B is usually taken as the end point of monolayer adsorption capacity.

Type III isothermal lines are characterized by a protrusion in the direction of the relative pressure axis. These are rare when weak gas-solid interactions occur on nonporous or macroscopic solids.

The IV-type isothermals are produced by mesoporous solids. A typical feature is that the adsorption branch of the isothermals is inconsistent with the desorption branch, and a hysteresis loop can be observed. A plateau can be observed in regions with higher P/P0 values, sometimes ending with the final turn of the isothermals turning upward.

The characteristic of V-shaped isotherms is that they bulge towards the relative pressure axis. Unlike III-shaped isotherms, there is a inflection point at higher relative pressures. V-shaped isotherms originate from weak gas-solid interactions on microporous and mesoporous solids, and water vapor adsorption in microporous materials often results in such line shapes.

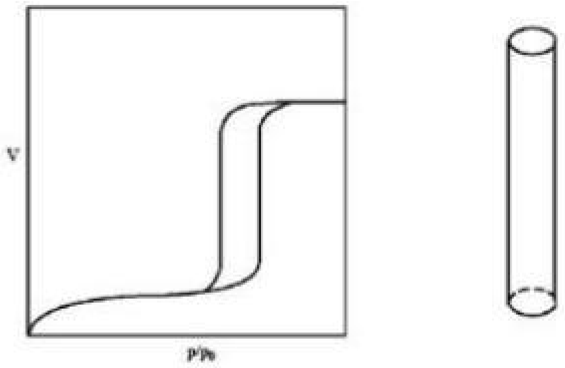
The VI isotherm is renowned for its stepped characteristics in the adsorption process.These steps originate from successive multilayer adsorption on a uniformly nonporous surface.Nitrogen adsorption at liquid nitrogen temperature does not yield the complete form of this isotherm, whereas argon adsorption at liquid argon temperature can achieve it.

The above six common adsorption patterns can be used to identify different materials by observing the adsorption amount and adsorption rate under different relative pressures. During pore size analysis, the adsorption curve and desorption curve do not coincide, so the isothermal curves for adsorption and desorption are used

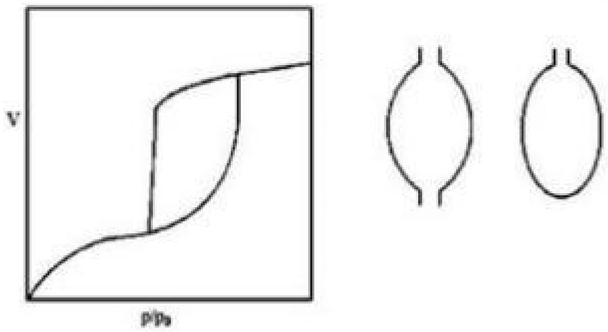
It will form a ring, which means that during adsorption, multi-molecular layer adsorption occurs first, and only when the adsorption layer on the pore wall reaches a sufficient thickness can coalescence occur. During adsorption, there are two factors involved: multi-molecular layer adsorption on the pore wall and coalescence within the pore, while desorption is solely caused by capillary coalescence. When desorption occurs at the same P/P0 pressure as adsorption, only the vapor on the liquid surface within the capillary can occur, and it cannot cause the molecules adsorbed under P/P0 to desorb. To achieve desorption, a smaller P/P0 is required, hence the lag phenomenon in desorption, which is actually due to the irreversibility of adsorption under the same P/P0.

(2) The type of material pores is judged from the lag loop of nitrogen adsorption isotherm

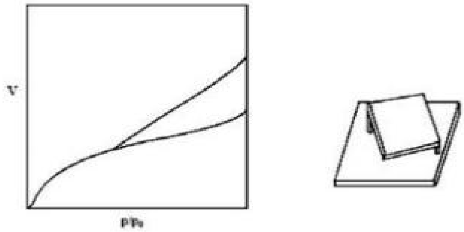
① The hysteresis loop corresponding to the open tubular hole structure at both ends



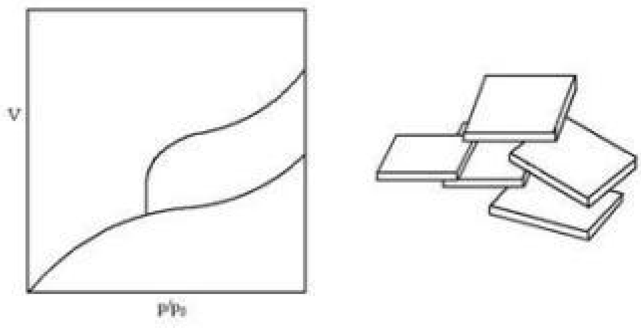
② The hysteresis loop corresponding to the "ink bottle" hole



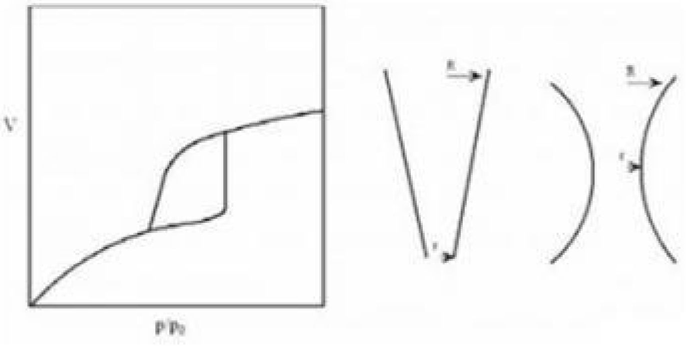
③ The inclined plate with four sides open and overlapping slit structure corresponds to the hysteresis ring



④ Flat slit hole structure corresponds to the hysteresis ring (it is difficult to form a concave liquid surface during adsorption)



⑤ The hysteresis ring corresponding to the conical or double-conical tubular hole structure (gradually evaporated during desorption)



2. Specific surface area and range of values for samples with different pore sizes

(1) P/ Po relative pressure point

The BET formula is applied to calculate the specific surface area, and the BET linear relationship obtained for microporous materials at relative pressures of 0.01-0.1 is better than that at 0.05-0.2. For mesoporous materials, the BET results at 0.05-0.2 are reasonable. Most microporous materials have a specific surface area smaller than that at 0.01-0.1 when calculated at relative pressures of 0.05-0.2, and the difference in calculated values increases with higher content of microporous materials in the catalyst within these two relative pressure ranges.

For the BET surface calculations of microporous materials, it is essential to select the correct relative pressure range, and the C constant is a simple indicator for judgment. Any negative intercept on the BET plot indicates that the value exceeds the effective application range of the BET equation. The classic range for microporous materials in the BET plot is 0.05-0.30, and linear data cannot be obtained; moreover, a negative C constant has no physical meaning. For selecting the linear range of the microporous materials BET plot, any subjectivity should be avoided. The correct procedure is: the C constant must be positive, and the adsorption capacity should continuously increase with changes in relative pressure.

(2) The BET equation should not be used at too low a pressure point

When some data bend towards the origin these points cannot be used to calculate the specific surface area because the pressure points are too low to form a monolayer and when the C value is very small it results in a very high intercept. In this case conventional values are often obtained

The lower limit of 0.05 or more curvature is very obvious in the BET graph, which indicates that the upper line of the BET pressure point is compressed, and the data points with obvious bending below 0.3 should be deleted.

(3) Do not choose the relative pressure point of the BET equation too high

Incorrect sampling leads to poor correlation coefficients and negative intercepts in linear regression, meaning the C constant is negative. The upper limit of BET sampling can be determined by calculating the maximum single-point BET value. However, not all samples exhibit this behavior. Some samples do not show a maximum point in their single-point BET calculations and instead increase as pressure increases, indicating that there will be no short linear region below relative pressure 0.15. In such cases, the BET equation is not suitable for these samples.

(3) Physical adsorption theory

gas adsorptionTheories mainly includeLangmuirmonolayer adsorptiontheory,PolanyiAdsorption potential energy theory, BET multilayer adsorption theory (see Multimolecular layer adsorption), two-dimensional adsorptionfilm theoryPolarization theory, etc., the first three theories are most widely used. These

Adsorption theory starts from different physical models, comprehensively examines a large number of experimental results, and after certain mathematical processing, it is applied to a certain kind

(Or several) typesadsorption isothermThe limiting part is explained and the description of the

isothermal line of adsorption is givenequationResearch and development

-The most commonly used method for gas adsorption is to obtain the adsorption isotherm from isothermal adsorption experiments. Different types of adsorption isotherms represent different adsorption systems and can be explained by different adsorption theories and adsorption models.

Coal adsorption of methane mostly conforms to the isotherm of type I, and describing the coal adsorption process using the langmuir equation generally yields satisfactory results. It is also one of the most frequently used models in the coalbed methane field. The reasons are: firstly, the langmuir equation has only two parameters, making it easy to convert into a univariate equation for solving, with a simple form and convenient use. Secondly, the two parameters of the langmuir equation have clear physical meanings. Thirdly, after long-term engineering practice, it is believed that the langmuir equation can meet engineering needs.

1. Material specific surface area analysis

(1) Characterization of specific surface area

In 1938, Brumauer, Emmett, and Teller discovered the relationship between the actual adsorption amount V and the monolayer saturation adsorption amount Vm through thermodynamic and kinetic analysis of gas adsorption processes, leading to the famous BET equation. This equation is applicable for specific surface area testing under multi-layer theory and is currently the standard method for specific surface area testing of most materials.

In the characterization of the specific surface area of systems, there is also a portion of materials that conform to the characteristics of single-layer adsorption. Laugmuir derived the corresponding specific surface area testing formula based on the assumption of single-layer adsorption, which is used for characterizing materials with single-layer adsorption characteristics. This specific surface area is also referred to as Laugmuir specific surface area.

In addition, in some fields, the inner surface area of the materials micropores does not play a role, so the concept of external specific surface area is put forward again.

The test model of specific surface area was t-plotted.

(2) Basic assumptions of Langmuir monolayer adsorption theory

(i) There is a certain number of adsorption sites on the surface of the solid adsorbent, and each adsorption site can only adsorb one molecule or atom; (ii) The adsorption capacity of all adsorption sites on the surface is the same, that is, the adsorption heat on all adsorption sites is equal;

(iii) There is no interaction between adsorbed molecules. Thus, it follows that:

Pa/V=1/ Vmb+ Pa/ Vm

Pa—— adsorption equilibrium gas phase pressure
Vm—— monomolecular layer saturation adsorption amount
V—— volume of adsorbed gas
b—— Total adsorption constant

Using P/V-P as the graph, it is a straight line, and b and Vm can be obtained according to the slope and intercept.

(3) Multimolecular layer adsorption theory

①BET algorithm

The BET theory is hereLangmuir theoryBased on the single molecule adsorption model, the multi-layer adsorption is extended based on the following three assumptions

The situation:

(i) Gas molecules can be adsorbed on the solid in countless layers (ii) there is no interaction between the layers of adsorption

(iii) The Langmuir adsorption theory holds for each monomolecular layer. Thus, the BET adsorption isothermal equation is as follows:

$$\frac{1}{v\left[(P_0/P)-1\right]}=\frac{c-1}{v_m c}\left(\frac{P}{P_0}\right)+\frac{1}{v_m c} \qquad (1)$$

②t-plot method

T-plot, also known as t-curve, is an isothermal curve in which the adsorption amount is plotted against the relative pressure P/P0 as the x-axis and the adsorption amount as the y-axis

The line,

$$(1)\quad t = \frac{v}{v_m} \bullet t_m$$

$$(2)\quad \frac{v}{v_m} = \frac{C \bullet P/P_0}{(1 - P/P_0)[1 + (C-1)P/P_0]}$$

In this case, tm—— is the thickness of a single attached layer, t—— is the thickness of the adsorption layer, V—— is the adsorption amount of the sample under test Vm—— Saturation monolayer adsorption capacity

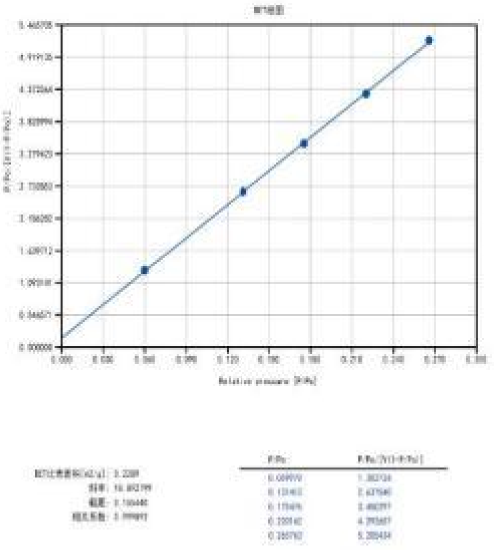
Tm——77.4K The thickness of adsorbate (nitrogen molecules) at Tm——77.4K temperature, assuming that the nitrogen molecules in the adsorbate film are arranged in hexagonal close packing, gives the thickness of a single layer of nitrogen, which is 0.354 nm.

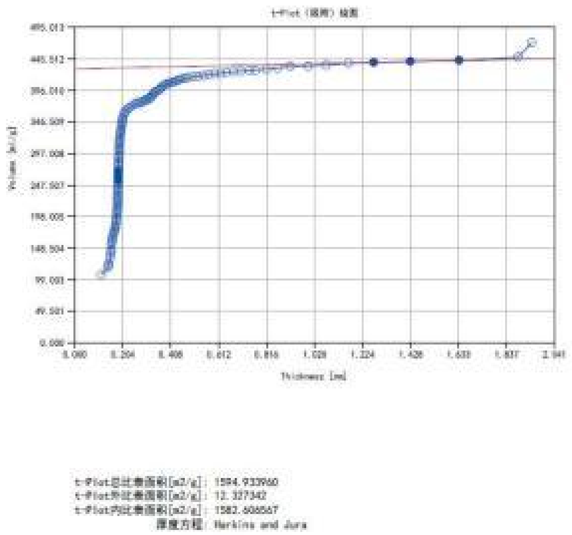
*** Preceding conditions for the application of t-plot method:**

(i) The samples have a smooth surface within the mesoporous range

(ii) The relative pressure range of the occurrence of micropore filling and capillary condensation does not overlap.

(iii) The deviation from linearity of the linear part of the figure corresponds to multi-layer adsorption (non-pore sample), which is caused by the pores contained in the sample. The latter can be evaluated by the volume of pores within a selected pore size range, and the specific surface area of the linear part, i.e., the unfilled part of the line, can be calculated from the slope of the line.





t-Plot总比表面积[m2/g]: 1594.933960
t-Plot外比表面积[m2/g]: 12.327342
t-Plot内比表面积[m2/g]: 1582.606567
厚度方程: Harkins and Jura

2. Characterization of porosity

(1) The concept of porosity

Porosity (Porosity) is the specific term used to characterize the surface properties of powders and particles: including total pore volume, pore size distribution, average pore size and other connotations.

(2) Characterization of porosity

According to the characteristics of material surface, the characterization of porosity can be divided into mesoporous, macroporous and microporous aspects.

Characterization of ① mesoporous and macroporous

Testing methods for mesoporous and macroporous materials

The determination and analysis of mesoporous and macroporous pores were carried out using BJH (Barrett-Joyner-Halenda). BJH pore size analysis was the main

The theoretical basis is the capillary condensation theory, and the main calculation method is to determine the pore size corresponding to the pressure by Kelvin equation, and assume that the nitrogen adsorbed in the pore exists at the density of liquid nitrogen.

According to the data points of isothermal line of gas adsorption and desorption, BJH model can calculate the pore volume, pore size distribution, total pore volume and average pore size step by step.

The characterization range of mesoporous and macroporous

Currently, the control range of nitrogen partial pressure for all nitrogen adsorption instruments is almost wide: the minimum value is close to 0, and the maximum value is close to 1. The lower limit of mesopores, i.e., 2 nm, corresponds to a nitrogen partial pressure of 0.14; when the pressure is 0.996, the pore diameter can reach 500 nm, so the pore size analysis range obtained by the BJH method includes mesopores and some large pores, generally it is believed that the upper limit of pore size measured by nitrogen adsorption methods is 500 nm.

② Characterization of total pore volume I adsorption of total pore volume

The total pore volume is calculated as the total pore volume, which is regarded as all the adsorbed and filled in the pores under the maximum relative pressure of nitrogen.

Capillary condensation theory and Kelvin equation

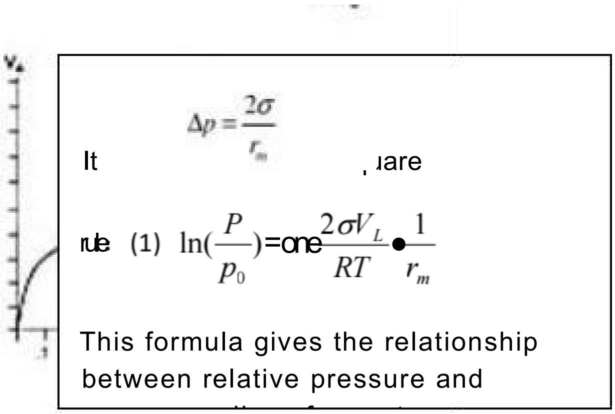
Capillary condensation refers to the phenomenon where, in a capillary pore, if adsorption must form a concave liquid meniscus, the equilibrium vapor pressure P must be less than the saturation vapor pressure P0 of the same temperature for the flat liquid meniscus. The smaller the capillary pore diameter, the smaller the curvature radius of the concave liquid meniscus, and the lower the equilibrium vapor pressure. This means that the smaller the pore size, the lower the pressure required for capillary condensation. Due to capillary condensation, the adsorption capacity of the sample increases sharply. When all pores are filled with liquid adsorbate, the adsorption capacity reaches its maximum, and the relative pressure P/P0 also reaches its maximum value of 1; as the pressure decreases, the condensed liquid in larger pores desorbs first, and with the gradual decrease in pressure, the condensed liquid in smaller pores also desorbs gradually.

Interpretation of adsorption-desorption curve

The adsorption-desorption process curve of mesoporous materials. At the critical temperature, when gas adsorbs onto the mesoporous adsorbent, it first forms a monomolecular adsorption layer, corresponding to segment AB in the figure. When the monomolecular layer adsorption approaches saturation, multi-molecular layer adsorption begins. When the relative pressure reaches a specific value corresponding to the Kelvin radius at which capillary condensation occurs, capillary condensation begins. Capillary condensation continues until the mesopores are fully filled, reaching adsorption saturation. The desorption process is the reverse, starting with capillary condensation within the capillary, followed by multi-layer desorption, and finally monomolecular desorption.

Assuming the capillaries are cylindrical pores divide all micropores into several zones according to their diameter these pore zones are arranged in order of size. Pores of different diameters produce different pressure conditions for capillary condensation during the desorption process when the pressure decreases from its maximum value 1 the condensed liquid gradually desorbs from the larger pores and then from the smaller pores. Clearly pores that can produce condensation phenomena or desorb from a condensed state can be identified

There is a certain relationship between the diameter and the pressure of the adsorbate, which is given by kelvin equation. As shown in the figure:



$\Delta p = \frac{2\sigma}{r_m}$

It uare

rule (1) $\ln(\frac{P}{p_0})$=one$\frac{2\sigma V_L}{RT}\bullet\frac{1}{r_m}$

This formula gives the relationship between relative pressure and

However, the aperture at which capillary condensation occurs is not the radius of curvature. Kelvin Equation:

① $$\ln(\frac{P}{p_0}) = -\frac{2\sigma V_L}{RT}\bullet\frac{1}{r_m}$$

As shown in the figure, the average radius of curvature has the following relationship with Kelvin radius:

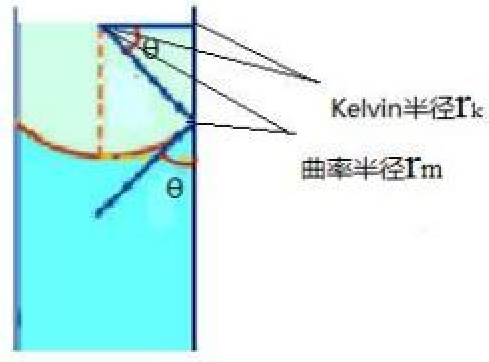
$r_k=\cos\theta r_m$

② Kelvin

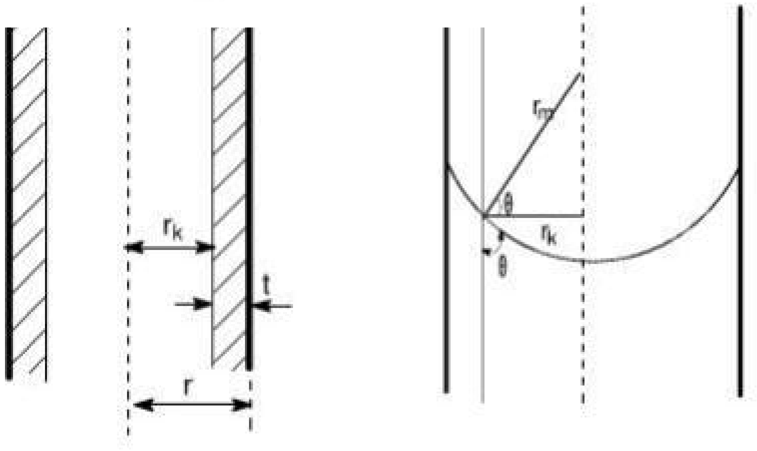
The equation gives the relationship between pore size and relative pressure when capillary condensation occurs in fact during Kelvin condensation multiple layers of adsorption have already occurred on the pore walls which means that the adsorption film occurs within the pore core surrounded by the adsorption film According to the t-P plot method it is known that the film thickness is related to P/P0 when the adsorption film thickness increases to a certain extent capillary condensation will occur within the adsorption film that is there is the following relationship:

③r= rk+t,

Here t is the membrane thickness and r is the radius of the hole.



The relationship between the curvature radius and the Kelvin radius is shown in the figure



The above ①②③ equation suggests: $r = \dfrac{2\sigma V_L}{RT\ln(P/P_0)} + t(P/P_0) + 0.3$

The r-pore radius; t-the thickness of the statistical film adsorbed on the pore wall, can be obtained from the t curve.

The BJH method was used to determine the pore size distribution

The Kelvin equation can better explain the IV-type isotherm, which is the general isotherm of adsorption by mesoporous materials. Using the Kelvin equation, the pore radius at any point on the isotherm can be calculated. According to the assumption of capillary condensation theory, all pores with radii smaller than a certain size will be filled at the corresponding adsorption pressure when the pore size is changed. Therefore, we can obtain the cumulative void volume as a function of pressure. Then, by differentiating the cumulative pore volume with respect to the pore radius function, we can obtain the pore size distribution.

According to the isothermal nitrogen method, the pore size distribution is commonly calculated using the Kelvin equation. From the BJH method proposed by Barrett, joyner and halenda to the widely used method at present.

The basic assumption of this method is:

All the pores are non-crossing cylindrical holes.

The contact angle of the hemispherical curved liquid surface is zero or completely wet.

iii Application of simple Kelvin equation.

To perform pore size distribution calculations, pores must be divided into several groups according to their pore size, and the r values and t values for a series of different relative pressures P/P0 must be calculated using the aforementioned formulas. Then, based on the basic formula for calculating pore size distribution using the BJH method, the volume ΔV of each pore size segment in the desorption stage must be calculated for each pore size. Plotting ΔV/Δr against the average pore radius r yields the pore size distribution curve. The Kelvin equation does not account for the superposition effect of adsorption potential in micropores, making it unsuitable for evaluating microporous carbon adsorbents but suitable for evaluating mesoporous materials. As the pore size decreases, its accuracy deteriorates, and when the micropore size equals the diameter of an adsorbate gas molecule, the Kelvin equation becomes completely inapplicable.

Using the BJH method, the total pore volume obtained by accumulating the pore volumes of different pore sizes calculated from isothermal adsorption or isothermal desorption processes is determined. It has clear upper and lower limits for pore size, generally ranging from 2 nm at the lower limit to 200-400 nm at the upper limit. When comparing various data, attention should be paid to the differences in pore size ranges. The measurement range of pore sizes should match the material being tested; blindly pursuing a high upper limit for pore size measurement is not necessarily beneficial. For example, for nanoscale powders, particle sizes are less than 100 nm, making it difficult to imagine particles having pores larger than a few dozen nm in diameter. In fact, the "large pores" measured are gaps between particles, which is incorrect for characterizing the surface and bulk properties of powders. Theoretically, capillary condensation occurs when P/P0 ≥ 0.4, but the lower limit of pore size analysis using BJH has consistently been much lower than 0.4, which has yet to be adequately explained.

There are three different ways to represent average aperture:

(1) Average pore size: The average pore size, which is calculated by the total adsorption volume and the BET specific surface area, includes all pores and defines only the upper limit of pore size.

(2) Average pore size of BJH adsorption: the average pore size is calculated from the total pore volume and total inner surface area of BJH adsorption accumulation, with upper and lower limits of pore size.

(3) BJH desorption average pore size: the average pore size obtained by BJH desorption cumulative total pore volume and BJH desorption cumulative total pore inner surface area, with upper and lower limits of the pore size.

Extending the BJH pore size analysis method to micropores is incorrect for two reasons: firstly, the Kelvin equation is not applicable when the pore size is <2 nm; secondly, the adsorbate described by capillary condensation is in a liquid state within micropores, whereas in micropores, due to the interactions between densely packed pore walls, the adsorbate filled in micropores is in a non-liquid state, thus the rules governing pore size distribution must be based on new theories and computational methods, macroscopic thermodynamic methods are far from sufficient.

3. Analysis and theory of microporous structure

(1) Introduction to Polanyi adsorption potential theory

The potential energy theory of Polanyi indicates that a potential energy field exists near the adsorbed surface, with the potential energy increasing as one approaches the surface. When a molecule is adsorbed, it enters the zero potential energy surface and is captured on an equipotential surface between the surface and the zero potential energy surface.

According to the theory of potential energy, the volume V adsorbed in different temperature ranges is determined by the surface of the adsorbent and the equipotential surface En.

When adsorption occurs above the critical temperature of the adsorbate, the adsorbate cannot be liquefied and remains in a gaseous state, but its density is higher at the surface; near the critical temperature, the adsorbed adsorbate appears as a liquid near the surface but becomes gaseous farther from the surface; if the adsorption temperature is much lower than the critical temperature of the adsorbate, the adsorbed adsorbate is considered to be entirely in a liquid state, and the adsorption volume can be expressed as

$$V = \frac{\mathrm{W}}{\rho}$$

Here, W is the mass of adsorption (g), and ρ is the density of the adsorbate liquid (g/cm3). The potential energy theory suggests that when the adsorbate is in the liquid state, its adsorption potential energy is:

$$E = \mathrm{RT} \ln \frac{\mathrm{p}_0}{\mathrm{p}}$$

According to this equation, the potential energy E is the work done in the adsorption region when the steam at equilibrium pressure p is compressed to the saturated vapor pressure p0 under isothermal conditions.

For any adsorption system, the characteristic curve is unique, therefore by measuring the isotherm of adsorption at one temperature and constructing the characteristic curve, the isotherm at any temperature can be obtained, which is precisely the advantage of adsorption potential theory. Activated carbon is a typical non-polar adsorbent, and the interaction with activated carbon mainly relies on dispersion forces, thus the adsorption potential theory provides a very successful explanation for activated carbon adsorption systems.

(2) Micro-pore filling theory and DR equation

The adsorbents used in industry are mostly microporous adsorbents, the adsorption behavior of gases in micropores differs from that in mesopores and macropores. The microporous physical adsorption theory was established by polanyi adsorption potential theory, which was later introduced to microporous adsorption studies by dubinin, leading to the creation of microporous filling theory. This theory is also known as dubinin-polanyi adsorption theory.

The DR equation is based on three assumptions: 1: θ is a function of adsorption potential; 2: β is a constant; 3: pore distribution is gaussian type.

For ease of use, the DR equation is usually converted to the following form:

$$\lg(\frac{V}{V_0}) = -D\lg^2(\frac{p_0}{P})$$

In order to expand the application range of DR method, Dubinin and Astakhov put forward a more general equation, namely DA equation

$$\theta = \exp[-(\frac{A}{E})^n]$$

The equation is based on the aperture distribution rather than the Gaussian aperture distribution, n is a small integer and E is the characteristic adsorption free energy.

$$\lg(\frac{V}{V_0}) = -D\lg^2(\frac{p_0}{P})$$

$$D = 2.303^{n-1}(\frac{RT}{\beta E_0})^n$$

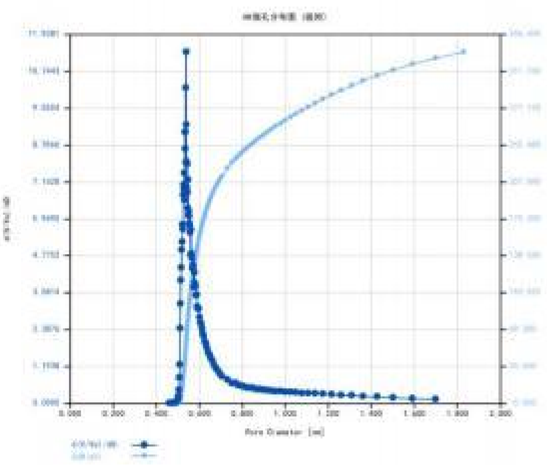
The value of n can be calculated as follows:

(1) V0 can be known as V/V0 from the platform of the adsorption isotherm.

(2) When A = E, θ = 1, θ = V/V0 = 1/e = 0.368, and P0/P corresponding to θ =0.368 on the isothermal line is calculated by RTln(P0/P).

(3) The value of n can be obtained by finding a set of data on the pressure and adsorption isotherm.

The above θ-micropore filling factor; V-at the relative pressure, which is the volume of adsorbed gas at that pressure, i.e., the volume of micropores already filled; V0-saturated adsorption volume, which is the total volume of micropores; A-Gibbs free adsorption energy; A=-RTln(P/P0); E-characteristic adsorption energy, E=βE0. Where E0 is the characteristic adsorption energy of the reference fluid (benzene), β is the affinity coefficient indicating the degree of similarity to the reference fluid.



HK equation

The HK equation is a semi-empirical method for characterizing the structure of microporous materials and calculating pore size distribution. Initially based on the slit pores of carbon molecular sieves and activated carbon, the adsorption potential of slit pores is expressed as a function of pore size, and the adsorption amount is calculated using the adsorption isotherm, followed by calculating the pore distribution.

The main basic framework of the HK model is the same, described by the following equation:

$$RT\ln(\frac{P}{p_0}) = U_0 + P_0$$

Among them: U0 adsorbate↔Adsorbent

interaction energy P0-adsorbate

and pore wall interaction energy

In the case of micropores, the interaction potential energy between the walls overlaps with each other, and the adsorption in micropores is larger than that in mesopores, so the phase is different

When the pressure is <0.01, filling occurs in micropores. Pores with diameters of 0.5~1 nm can even adsorb substances at relative pressures of 10^-5~ to 10^-7, making the measurement and analysis of micropores much more complex than that of mesopores.

The HK and SF methods have introduced semi-empirical analytical approaches for calculating the effective pore size distribution from isothermal adsorption lines for microporous samples. They are respectively applied to nitrogen/carbon (slit) and argon/zeolite (cylindrical pore) systems, treating the filler liquid as a kind of loose fluid. These calculations require introducing a series of relevant parameters between adsorbents and adsorbates, and the selection of these parameters significantly impacts the computational results. This method proposes a new relationship between micropore diameter and filling pressure but does not address the density model of nitrogen molecules in micropores. Therefore, it can represent the adsorption patterns of micropores and the distribution of micropore pore sizes, but it still lacks quantitative analytical significance.

According to the t-graph method proposed by Lippens and deBoer, it is one of the most widely used methods in microporous analysis, where the adsorption capacity is defined as a function of statistical layer thickness t, which is calculated by the standard isotherm. The t-graph can be used to calculate the specific surface area of the inner surface of micropores, the outer surface area, and the total volume of micropores. The t-graph method, also known as the MP method, is used for pore size distribution analysis, which can analyze the pore size distribution of micropores. However, the disadvantages of the t-graph method and the MP method are that they still treat the adsorbate filled in the micropores as a liquid, and the pore size calculation still uses the Kelvin equation, thus the total pore volume obtained is only relatively meaningful, and the pore size distribution range cannot represent the true micro-pore region.

In the past decade, Non-Linear Density Functional Theory (NLDFT) and computer simulation methods (such as Monte Carlo fitting) have developed into effective methods for describing the adsorption and phase behavior of confined fluids in porous materials. NLDFT methods are applicable to various adsorbent/adsorbate systems and provide a molecular-level description of fluid behavior confined within pores compared to classical thermodynamic and microscopic modeling methods. Their application can correlate the molecular properties of adsorbate gases with their adsorption performance in pores of different sizes, making NLDFT suitable for characterizing pore size distributions across the full range of micropores and mesopores. Currently, NLDFT methods are highly regarded in foreign instruments and are used as standards to compare the effectiveness of other methods. However, the computational methods for different adsorbent/adsorbate systems vary, making it inconvenient to apply them universally. The new theory aims to address two main issues: first, to develop a more reasonable relationship between micropore filling pressure and pore size, and second, to propose the state and density of adsorbate within micropores. There is reason to expect a simpler and more universal density functional theory that would make the analysis of ultra-micropore pore size distributions more extensive and comparable.

Chapter 6 Common faults and solutions

(1) degassing system failure

1. The temperature display is "HHHH"

This is a heating furnace temperature detector failure, and the heating furnace should be sent back to the manufacturer for repair.

2. Vacuum abnormally

(1) The vacuum cannot reach minus 0.1 Pa

If the pressure is stable when the pressure is drawn, it is the error of the pressure gauge system; if the pointer jumps, the new pressure gauge needs to be replaced.

(2) Unable to evacuate

(3) Check whether all the inspection and treatment ports are installed with sample tubes.

The motherboard chip is faulty and needs to be sent back to the manufacturer for repair.

(2) Vacuum pump failure

1. The vacuum pump emits "white smoke"

The fault is mainly caused by the air leakage:

(1) Check whether the air line is damaged; check whether there is gas leakage at the connection.

(2) Check whether the sample tube nut is "tightened" and whether the sample tube opening is damaged / "sand eye".

2. Abnormal noise

If the pump is contaminated by entering the pump, the oil should be replaced. If the oil replacement does not improve, the pump body has been worn by contaminants and can continue to be used without affecting the vacuum effect.

3. The vacuum pump is not working

(1) The vacuum pump power plug is damaged during the moving of the instrument, and the vacuum pump power plug needs to be replaced or repaired.

(2) The relay controlling the operation of the vacuum pump is damaged. When the instrument controls the opening of the vacuum pump, the power supply of the vacuum pump at the main unit end is connected

There should be 220v AC output at the port, which needs to be determined with a multimeter.

(3) The vacuum pump is damaged. Connect the vacuum pump to the 220v power supply, and the vacuum pump does not work.

(3) The program runs slowly or stops

1. "Vacuum" operation pause

(1) Due to the failure to end the last test normally or the sudden power failure during the test. In this case, the program needs to be run first to "finish"

When the "bund analysis" can be operated, click the end analysis.

(2) If the sample tube has cracks or is broken during installation, the sample tube should be replaced.

(3) Caused by insufficient intake pressure, the intake pressure needs to be adjusted (helium 1 15 KPa, nitrogen 140 KPA) 2. "dead volume calibration" operation pause

Insufficient helium inlet pressure or helium source not opened

3. The operation of "adsorption analysis" is suspended or not finished

(1) No nitrogen, check the nitrogen cylinder

(2) Excessive sample quantity

4. The operation of "desorption analysis" is suspended or not finished

(1) The sample tube is damaged

(2) No nitrogen, check the nitrogen cylinder

(3) Vacuum pump failure

(4) The analysis time is too long, and there is no liquid nitrogen in the Dewar flask. "End analysis" does not end

(1) The sample tube is damaged

(2) Insufficient helium inlet pressure

(3) Vacuum pump failure

(4) Electromagnetic valve failure

(4) Instrument malfunction or abnormality

1. Vacuum calibration failure / pressure sensor calibration failure

(1) Check helium pressure

(2) Check whether the sample tube is damaged

(3) The last test did not end normally or the test was suddenly cut off by power failure 2. crash

WindowsCE motherboard failure of the program

3. Isothermal line anomaly

(1) Helium quantification error

(2) The sample quantity is too small

(3) The sample tube is damaged

Chapter VII Instrument Accessories

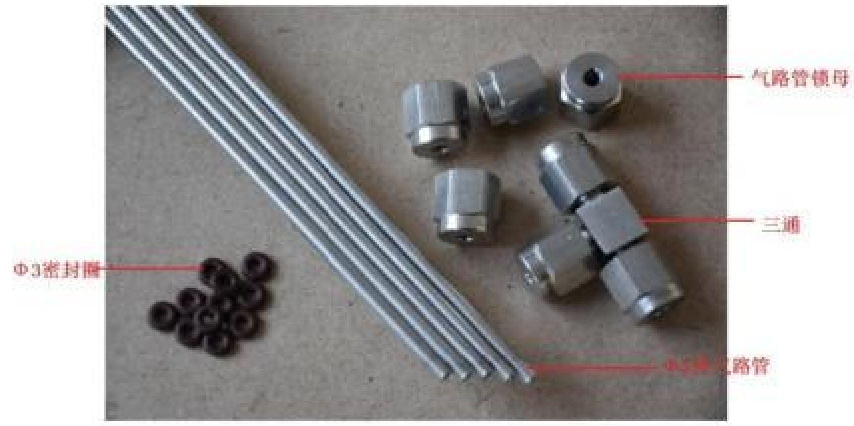
(一) Annex table

order number	Name of the annex	specifications	code

1	Two-stage rotary vane mechanical pump	4X10-2 Pa	1100-PF-D3
2	turbomolecular pump	1X10-6 Pa	1100-PF-H80
3	liquid nitrogen container	10L	1300-YX-10
4	High purity helium and cylinder pressure reducing valve	10L	1200-GAS-10H
5	High purity nitrogen and gas cylinder pressure reducing valve	10L	1200-GAS-10N
6	Stainless steel Dewar flask	3000 ml	1320-HS-3L
7	BET sample cell	270 mm	2000-Q-270
8	Sample funnel	270 mm	2020-L-270
9	Sample filter plug	Φ10	2110-M-F10
10	Plug column	260 mm	2030-T-260
11	P0 pipe	270 Smm	2040-P0-270S
12	standard	——	2500-AL-180
13	Pressure reducer (including air line connectors)	YQY-12	1210-GAS-PCV
14	Tracheal lock	M8*3	2150-M-M8*3
15	External gas line	Φ3	1220-GAS-3163
16	Airway pipe seal	Φ3	2100-M-3
17	P0 pipe seal	Φ6	2100-M-6
18	Sample tube seal	Φ8	2100-M-8
19	built-for purpose tools	20 mm	2220-SATA-6
20	sylphon bellows	KF16(150 cm)	1230-GAS-KF 150
21	KF 16 clamp, sealing ring	KF-16	2130-KF-16
22	power line	1.5m	2330-PW-200
23	network adapter	4	2310-T-2
25	Software, manual and certificate of conformity		
26	convenienve receptacle		
27	Liquid crystal touch pen		
28	Storage USB drive		

(二) Introduction to the annex

1. External gas line sealing accessories, as shown in Figure 7-1



graph 7-1

2. Vacuum pump accessories

1. The vacuum pump is mainly a turbine molecular pump (Figure 7-2) and a bipolar rotary mechanical pump (Figure 7-3), and the molecular pump is located inside the instrument host

2. Please refer to Figure 7-4 for the sealing parts and Figure 7-5 for the sealing method

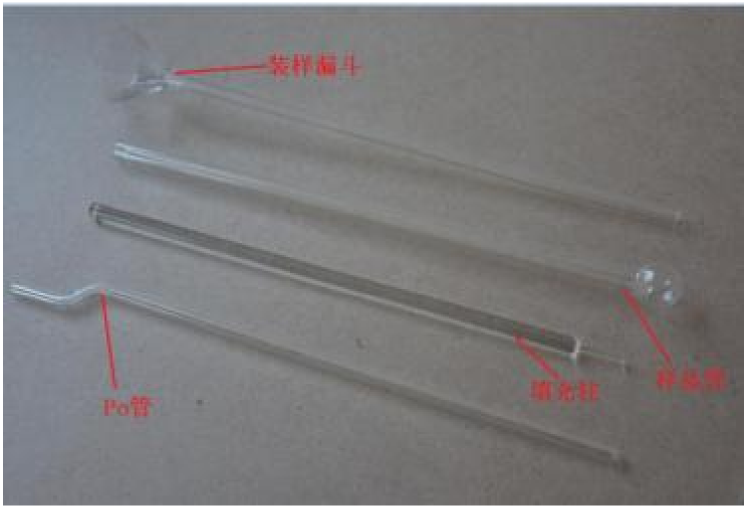
graph 7-2

graph 7-3

graph 7-4

graph 7-5

3. Sample tubes and Po tube accessories, as shown in Figure 7-6, sealing accessories as shown in Figure 7-7 and Figure 7-8



graph 7-6

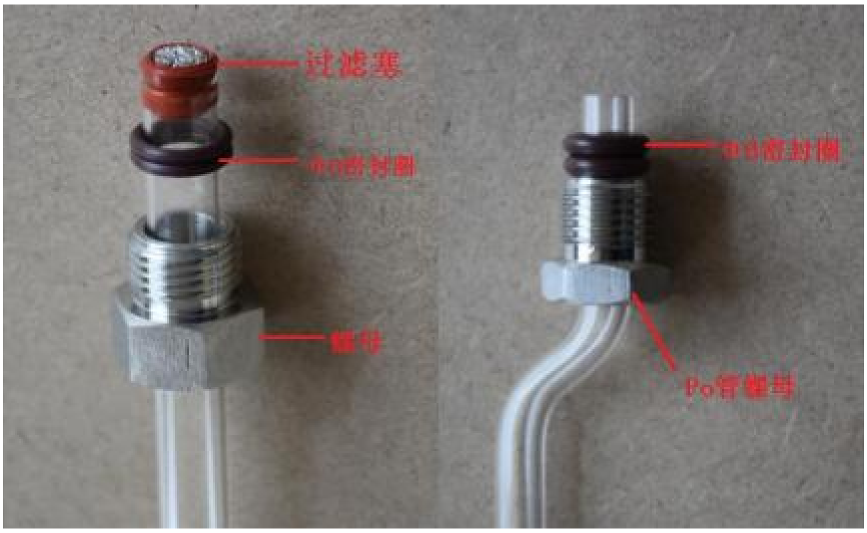


Figure 7-7 Sample tube seal Figure 7-8 Po tube seal

When loading the sample tube and Po tube into the instrument, it is also necessary to seal it with a special sealing tool, as shown in Figure 7-9.

graph 7-9

4. Liquid nitrogen container accessories, mainly including Dewar bottles (Figure 7-10) and liquid nitrogen tanks (Figure 7-11)

Figure 7-10 Figure 7-11

Chapter VIII After-sales Service Commitment

The service includes technical service commitment and product maintenance commitment. The following is a detailed explanation of the two parts:

(一) Technical services

This commitment applies to the warranty period of the product, which is 1 year from the date when the equipment arrives at the user and is qualified after opening and acceptance. After the warranty period ends, we will continue to provide technical services at the most favorable price.

Pete Companys confidence and determination to achieve success in the inspection and analytical instrument manufacturing industry in China are entirely due to our possession of "excellent talent", "rich experience", "advanced technology", and a "unified command organizational system". We will organize our most outstanding and experienced technical personnel to provide your company with the highest quality technical services.

1. Content of technical services

Piod Electronic Technology Co., Ltd. is willing to provide users with lifetime technical consulting services. Its contents include: notification of new products and technologies, hardware and software technology consultation, providing technical solutions and research and solution of technical problems.

The environment at the equipment installation site is a fundamental condition for the normal operation of the equipment and also serves as the basic guarantee for the good operation of the entire system. Therefore, out of responsibility to our users, we provide free support and guidance on requirements such as temperature, humidity, and equipment connections at the installation site. To ensure that users can manage all equipment uniformly and reasonably, we will provide specific requirements for the installation site based on the users actual situation to meet the needs of various on-site environments.

(二) Product services

1. Hardware warranty service

The Potevio Technical Support Center provides 5 days 8 hours of service, and in special cases can provide 7 days 24 hours of service. During the warranty period, we will provide users with comprehensive troubleshooting technical services and full-process monitoring of troubleshooting (from the start of the fault to the complete resolution of the fault).

When the equipment fails and cannot work, the user needs to notify the company through real -time notification and explain the cause and condition of the fault as much as possible. The company will solve the problem in the following way.

① General faults:

General faults refer to the failure or improper configuration of non-important links and equipment, which affects individual functions or operations. For such faults, we first use telephone support to guide the on-site solution until the problem is eliminated.

② Severity failure:

Serious failure refers to the failure or improper configuration of important equipment, which greatly affects the use of customers. For serious failure, we first use the method of telephone support to find out the cause of the failure, and make corresponding preparations before going to the site to solve it.

③ Emergency fault:

An emergency failure is when the equipment is completely down and unable to do any work. For emergency failures, we will commit to two simultaneous solutions: telephone support and on-site resolution to ensure that the problem is solved in a timely and effective manner.

To better serve our users please provide the product serial number when the device malfunctions. We will document any issues that arise to facilitate analysis and reduce the likelihood of similar incidents. The fault response time is within 12 hours of receiving a written fault notification from the customer. We will respond accordingly within this timeframe to ensure prompt handling of the fault and minimize any losses caused by the malfunction.

(三) Software upgrade

Software updates include the installation of new versions of software or maintenance of software versions. Pioder will provide users with timely software update technical services and provide free software upgrades for life.

(四) Technical training

In order to enable the technical personnel of the client side to proficiently master the operation and maintenance of the equipment for the normal operation of the entire system understand the new development trends in the involved technology and strengthen cooperation between both parties after the cooperation agreement between both parties takes effect Piod Company will dispatch dedicated personnel to arrange a series of training sessions based on user requirements covering specific technical and instrumental equipment fundamentals so as to facilitate the client sides technical personnel to be most proficient

Learn how to use the instrument in a short period of time so that the instruments and equipment of Peter Company can be used to the maximum in your enterprise.

Chapter IX Appendix

(一) Operation process

1. Sampling and weighing

(1) Experimental equipment: BET sample tube, funnel, sample tube stopper, balance (precision of 0.1 mg or higher).

(2) Sampling: The diameter of the sample is <5 mm. The amount of sampling depends on the size of the adsorption capacity of the sample. The larger the specific surface area and the more developed the pores, the greater the adsorption capacity, and the less amount of sample is needed for analysis. The recommended values of sampling amount are listed in the table below for reference.

specific surface [㎡/g]	<1	1-10	10-100	100-300	>300	remarks
volume of sample loading [g]	>2	1-2	>0.5	>0.1	<0.1	The minimum should not be <0.01

(3) Loading:

① Put the stopper into the mouth of the empty tube, weigh the mass of the empty tube and stopper, recorded as M1, and then peel it off.

Note: If you weigh multiple samples at one time, you need to ensure that the empty tube corresponds to the rubber stopper. You can choose to mark the empty tube and rubber stopper.

② Remove the stopper, put the funnel in place, fill the sample into the empty tube and plug it with the corresponding stopper, weigh the sample and record it as M2.

2. Sample pretreatment

(1) Experimental equipment: degassing system, sample filter plug, Φ10 sealing ring.

(2) Sample tube into the processor: remove the rubber stopper for use, and sequentially install 1 nut and 2 Φ10 sealing rings into each sample tube

1 filter plug is connected to the degassing port and tightened. The heating furnace is raised so that the sample tube belly is exactly inserted into the heating port. The remaining treatment position is connected to an empty tube.

(3) Set conditions: set the temperature and time to be processed, then click start. After the processing is completed, the processor will automatically alarm and prompt. At this time, lower the heating furnace and cool the sample tube to room temperature.

(4) Processing completion: After the sample tube is cooled to room temperature, click the "gas injection" button and charge nitrogen as a protective gas.

(5) Secondary weighing: Remove the sample tube after processing, remove the filter plug and insert the corresponding rubber plug to weigh and record the weight as M3. After processing, the mass of the sample is M=M3-M1. Note that if M>M2, the sample should be sampled again for processing.

3. Test on machine

(1) Experimental equipment: SSA-7000 analysis host, liquid nitrogen, high purity nitrogen and helium, vacuum pump, Dewar flask, Φ8 sealing ring, filter plug, packing column.

(2) Open the valve of helium cylinder and nitrogen cylinder, adjust the output pressure to 0.3 MPa, and start the vacuum pump and main electric machine

Source, instrument preheating 30 min.

(3) Sample tube mounting: Remove the rubber stopper, and sequentially install 1 nut, 2 Φ8 sealing rings and 1 filter stopper into each sample tube, with the sealing ring 1-2 cm away from the mouth of the sample tube. Make the sample tube vertical to the ground, and tighten the nut.

(4) Set parameters: In the parameter setting interface corresponding to the analysis station, input the sample mass, name, drying temperature, and time, and set the file name for generating the report; in the analysis control interface, set the analysis type, generally choose "Microporous", and select idle for remaining untested samples analysis stations. After setting is completed, click OK, close the window, return to the main interface, and click Prepare Complete.

Note: The current file name cannot be duplicated with the previous file name, otherwise the previous file data will be overwritten.

(5) Start Test: When the start test button turns green, it indicates that all analysis stations have been prepared. At this point, click to start the test.

(6) Add liquid nitrogen: Fill the Dewar flask with liquid nitrogen, and the liquid nitrogen surface is 1-2 cm away from the mouth of the Dewar flask. Then place the Dewar flask on the lifting tray of the analytical host.

(二) Precautions

1. Installation

(1) After the gas line is installed, a leak test must be conducted first. Open the main pressure valve of the gas cylinder and adjust the pressure reducing valve to 0.3 MPa, then close the main pressure valve of the gas cylinder and wait for 10 minutes. Observe the pressure on the pressure reducing valve gauge. If the pressure does not drop, there is no leakage; if the pressure on the gauge drops, apply soapy water to the threads or other gas line connections, where bubbles indicate the leak point; if the pressure reducing valve pointer fluctuates, a new pressure reducing valve should be replaced.

(2) Before the first installation or replacement of the cylinder, the instrument should be vented before use. Follow the system prompts to do so.

(3) Correctly install and use the vacuum pump, do not confuse the suction port and exhaust port of the vacuum pump during installation, otherwise it may cause the pump body to explode or more serious safety accidents. The vibration of the vacuum pump should always remain in the factory state and must not be touched. In case of any problems, contact the manufacturer promptly.

(4) The pump oil in the vacuum pump should always be clean (crude oil color), and the oil quantity should be at the position of 1/2 or above in the oil quantity display window. The pump oil should be added in time when it is too little, discharged in time when it is too much, and replaced in time when it is changed color.

2. Sample pretreatment

(1) When sampling and weighing, the sample tube should be matched with the rubber stopper and not confused.

(2) If multiple samples are processed at the same time, the sample tubes and stoppers should be marked to facilitate weighing and sample recovery.

(3) The heating furnace needs a certain time to heat up, and the influence of heating time should be considered when setting the processing temperature.

(4) If there is any remaining degassing position on the degassing station, an empty sample tube or plug must be connected.

(5) The samples must be cooled to room temperature before nitrogen can be charged to protect them.

3. Test on machine

(1) The output pressure of the pressure reducing valve should be 0.3-0.5 MPa, not too high or too low. If it is too high, the gas line should be removed first, and the gas in the pressure reducing valve should be drained, and then the gas line should be reinstalled to adjust the output pressure of the pressure reducing valve.

(2) The sample tube to be tested should contain a packing column, and the weight of the sample on the software should be input as the net weight of the sample after degassing and drying.

(2) For ultra-light powder analysis, the sample tube must be fitted with a filter plug.

(3) If there is no idle station, an empty sample tube must be installed.

(4) When setting the file name of the current sample, it cannot be repeated with the previous file name, otherwise the previous file data will be overwritten; the sample name and file name cannot have special symbols, such as ￥@ * &?!% etc.

(5) When the specific surface area of the test sample is very small, the incremental pressure can be set to 0.02 or lower.

(7) If the power supply is unexpectedly cut off during the test process, the analysis should be repeated after the power supply is turned on. When the analysis is terminated, the termination button should be clicked to restore the default state of the system parameters.

(8) After the test is completed, it is necessary to wait until the analysis process display bar is completely completed before disassembling or disconnecting the sample tube.

(9) When exporting data, if the export fails, you should first determine that a new "iPoreReport" folder has been created in the U disk. If it still fails, you can check the option to replace existing files in the export interface, and the export will be successful.

(三) Maintenance guidance

Clean the instrument housing	**once a week**
Replace the filter plug of the sample tube	Every three months
Clean the Duv bottle	once a week
Replace the sample tube seal	Every three months
Check and change vacuum pump oil	Every 3 months
Run the instrument to check for leaks	Every year

1. Clean the instrument housing once a week

Wipe the instrument housing with a clean cloth dipped in isopropanol or water. Avoid washing the cleaning solution into the interior of the instrument housing.

2. Replace the sample tube filter plug every 3 months

If the filter plug used in the sample tube is contaminated, adsorption and desorption will occur during the analysis process, thus affecting the analysis results. A batch of filter plugs should be replaced every three months.

3. Clean the Dewar bottle once a week

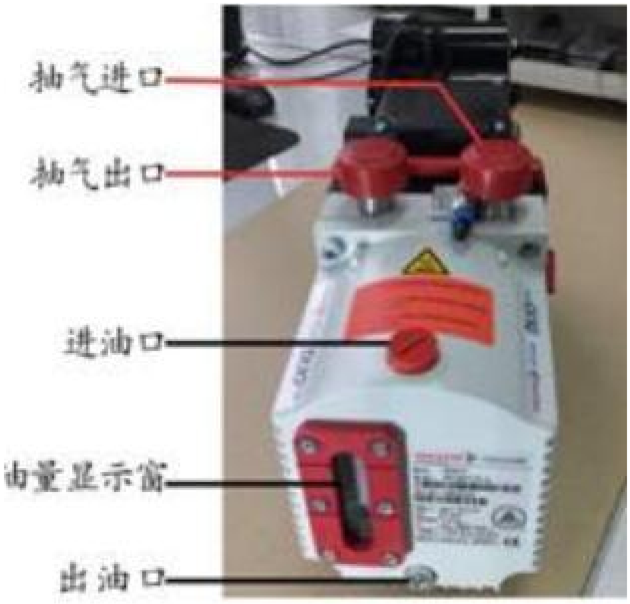
Check whether the interior of the Dewar bottle is clean. The sediment in the Dewar bottle will pollute the liquid nitrogen and affect the normal lifting and lowering of the Dewar bottle. Wash the interior of the Dewar bottle with water and dry it naturally.

4. Replace the sample tube seal every 3 months

The brown sealing ring on the sample tube needs to be replaced after aging or damage to avoid affecting the vacuum seal of the sample tube.

5. Check and change vacuum pump oil every 3 months

Observe whether the oil level in the pump is normal (the oil level should be at the middle position) from the oil level display window. Check if the oil color is clear and transparent (the oil should be clear and transparent). If not, drain the oil from the pumps outlet and fill the pump to the normal oil level from the fueling port. The pumps outlet is at the bottom, so it is best to place the pump flat on a table or other high surface, and use a container to collect the waste oil. The pumps inlet and outlet are shown in the following figure.



6. Run the instrument to check for leaks, and check the instrument every 1 year

The instrument is checked for leak and calibration every two years using the standard sample to check the performance of the instrument.

(四 Experimental record sheet

The following is a reference format for the record sheet to facilitate data management and viewing.

Source: Pipe Sealing Number: Date:

Sample tube number	Analysis station I	Analysis Station II	Analysis station iii	experiment condition
Sample number				Pre-treatment conditions: Experimental parameters:
Sample tube weight (g)				
Sample weight before drying (g)				
Weight of sample tube after drying (g)				
Weight of sample after drying (g)				

Source: Pipe Sealing Number: Date:

Sample tube number	Analysis station I	Analysis Station II	Analysis station III	experiment condition
Sample number				Pre-treatment conditions: Experimental parameters:
Sample tube weight (g)				
Sample weight before drying (g)				
Weight of sample tube after drying (g)				
Weight of dried samples (g)				