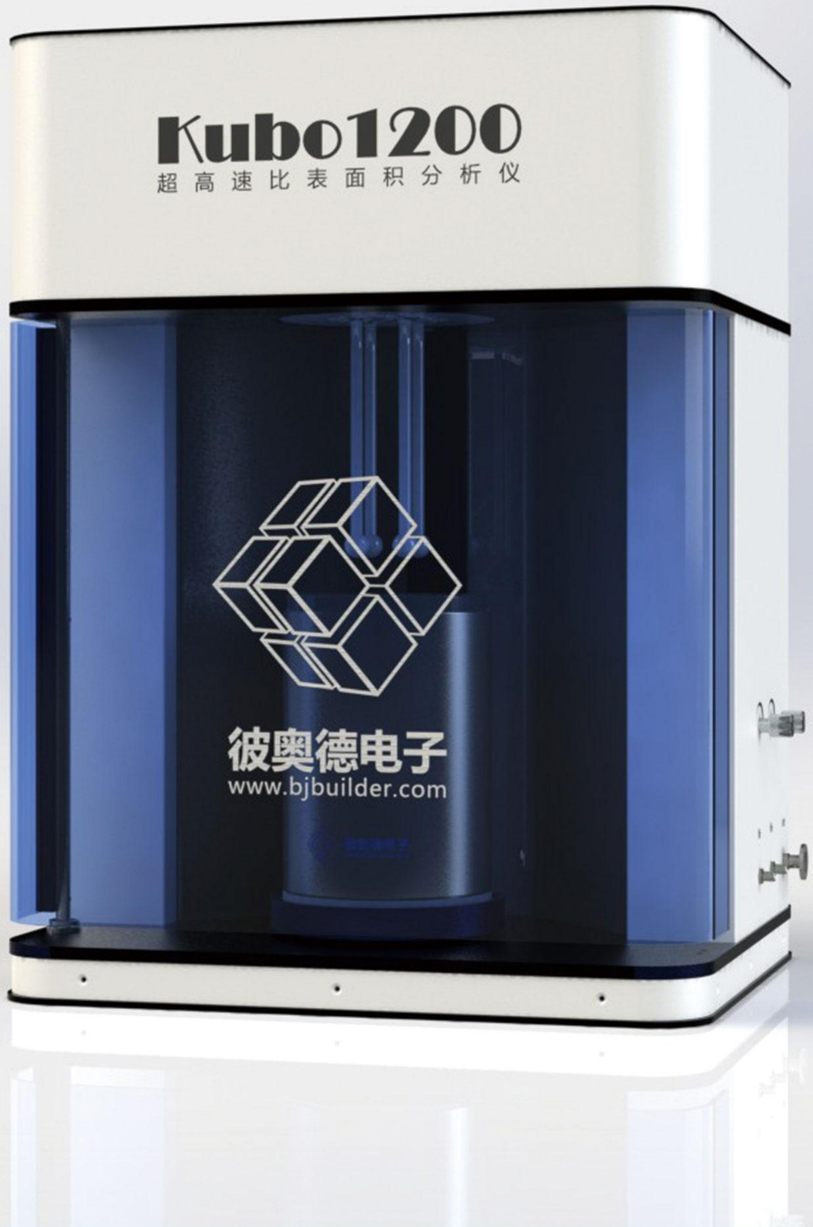

Kubo-1200

Ultra-high-speed specific surface area

Operating Manual

彼奥德电子
www.bjbuilder.com

400-6698981

Dear Users:

First of all, thank you for choosing our products. Our products are mainly developed, generated and sold by ourselves:

1. **Series of specific surface area and pore size analyzer**
2. **Series of programmed temperature chemical adsorption instruments**
3. **Gas true density meter series**
4. **Capacity method steam adsorption instrument series**
5. **Capacitance gas adsorption instrument series**
6. **Capacitance method high pressure adsorption instrument series**
7. **Bubble point membrane pore size analyzer series**
8. **Laboratory gas distribution instrument series (providing gas inlet scheme for infrared/mass spectrometry, etc.)**
9. **Customized series of small-scale catalytic evaluation devices (including VOCs generation and treatment series, SCR denitration and desulfurization series, etc.)**
10. **Online mass spectrometer series**

Beijing Builder Electronic Technology Co., Ltd. was established on January 9, 2003, specializing in the technical research and development of material characterization instruments. It is a technology service company that integrates project R&D, product development, and testing consulting. Over nearly two decades of development, the company has accumulated a team of highly skilled R&D and application technology professionals. With strong technical support and robust personnel infrastructure, Poid can independently complete product development and the R&D and manufacturing of core components, providing technical development services beyond its main product range to various research institutions and enterprises.

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This manual describes all the functions of the instrument. Your product may not have all the features in this manual. Please refer to the actual goods.

Failure to use it correctly or misuse that is clearly defined as foreseeable is not covered by the warranty.

1. For the version number of data processing software and firmware in this manual, see the software.
2. Please read the product manual in detail before first use. If you have any questions or suggestions, please call 400-669-8981 or email to service@bjbiaode.com.
3. The company will continue to improve and update the performance and variety of various instruments. For this reason, we must reserve the right to change the scope of supply, configuration and technical description, and we hope users can understand.
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- More 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 connector into the gas cylinder outlet and screw on the sealing nut, as shown in Figure 1-2.

graph 1-1

graph 1-2

2. Install the pressure reducing valve and switch valve

Install the black rotary switch (i.e., the switching valve of the pressure reducing valve) on the pressure reducing valve, as shown in Figure 1-3

graph 1-3

(2) Installation of degassing station

1. Understand the vacuum pump

As shown in Figure 1-4, the instrument is equipped with a bipolar rotary vacuum pump. Before installation, please familiarize yourself with the function of each joint/switch on the pump to avoid incorrect installation. Note that do not confuse the suction port and exhaust port, otherwise it will cause the pump body to burst and cannot be repaired or more serious safety accidents.

pay attention to: Do not install the exhaust port as a suction port!!!!

graph 1-4

2. Install the external air pipe

① Install KF16 to 3mm connector

The KF16 seal ring is fitted over the vacuum pump suction port, and then the KF16 turn φ3 adapter is installed as shown in Figure 1-5. After the adapter is installed, the KF16 clip is inserted into the connection and the nut is tightened.

graph 1-5

② Insert one end of the φ3 stainless steel gas line into the "vacuum pump gas line interface" of the degassing station, and tighten the nut. The other end is connected to the vacuum pump adapter, and tighten the nut.

3. Use of pump oil

(1) Add pump oil

Note: Be sure to use the pump oil of the vacuum pump, not the oil!

Loosen the oil inlet of the vacuum pump (see Figure 1-6), and slowly pour the pump oil into the vacuum pump. Observe the oil level through the oil gauge at the front end of the vacuum pump until it reaches the middle or higher position, then tighten the oil inlet. If too much oil has been poured, use a hex key to open the drain port next to the oil gauge, release the excess oil, and tighten the nut.

graph 1-6

(2) Inspection and replacement of oil in the pump

Observe the oil level in the tank to ensure it is normal (the oil should be at the center; if too low, add more promptly); check if the oil color is clear and transparent (the oil should be clear and transparent). If not, drain the dirty oil from the pump outlet and refill it to the normal oil level using the filling port. The pump's outlet is at the bottom, so it is best to place the pump flat on a table or other elevated surface. Use a container to collect the drained waste oil. See Figure 1-7 for the pump's inlet and outlet.

graph 1-7

4. Install the power cable of the pump

① One end of the power cable of the general vacuum pump has been connected to the pump body. At this time, the other end can be 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.

5. Install the heating furnace

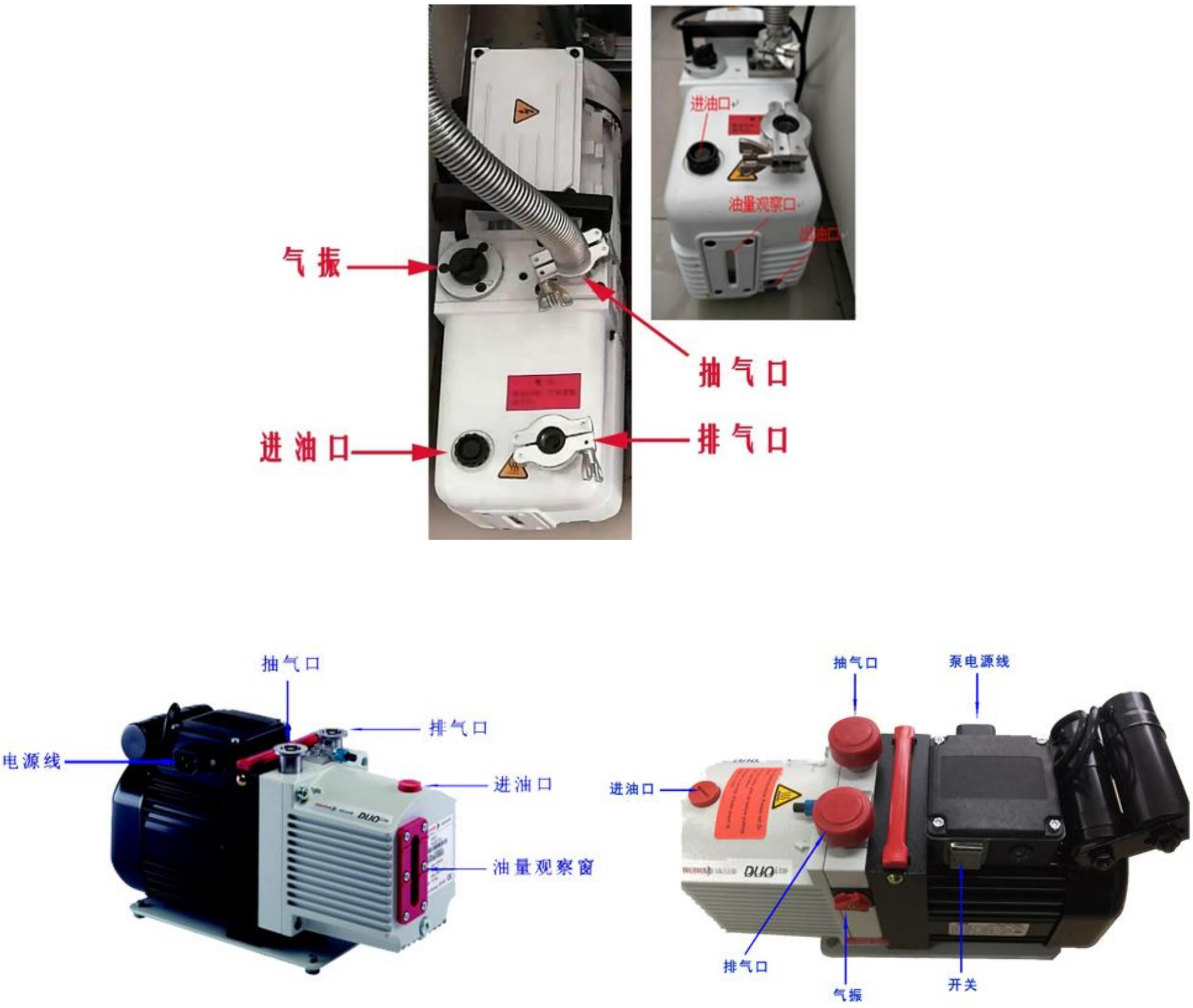
graph 1-8

The electric furnace interface is located on the left side of MD200S. According to the figure, align the groove of the heating furnace power line with the protrusion of the interface, and then insert the "heating power" and tighten the nut; when pulling out, loosen the nut, hold the power line interface end and pull out, do not directly hold the wire to pull out.

Note: When connecting the heating furnace power supply by "plug and play", the front processor must be in the state of power off.

(3) Installation of the main vacuum pump

The Kubo-1108 is equipped with a two-stage rotary vane mechanical pump, as shown in (Figure 1-9). Note that the suction port and exhaust port should not be confused and installed incorrectly, otherwise it will cause the pump body to burst and cannot be repaired or cause more serious safety accidents. The vibration of the pump should always be at the factory head, do not touch it.



graph 1-9

1. Power cable connection

Insert the other end of the power cable of the mechanical pump into the 220V power interface, as shown in Figure 1-10

graph 1-10

2. Installation of KF16 clamp

graph 1-11

As shown in Figure 1-11, fit the KF16 seal ring over the bellows port, then fit the clamp and tighten the nut. When connecting to the instrument, align the bellows port with the instrument interface and press it tightly, then fit the KF16 clamp and tighten the nut.

3. Add oil to the pump

Loosen the oil inlet of the mechanical pump (see Fig. 1-9), and slowly pour the pump oil into it. Observe the oil level at the front end of the pump through the oil gauge port until it reaches two-thirds full, then tighten the oil inlet nut. If too much oil has been poured, use a hex key to open the drain port next to the oil gauge port, release the excess oil, and tighten the nut.

graph 1-12

4. Start and stop of the pump

The opening and closing of the pump are controlled by the vacuum pump power switch. When the instrument is working, the vacuum pump switch should be opened, and when the work is finished, the vacuum pump switch should be closed.

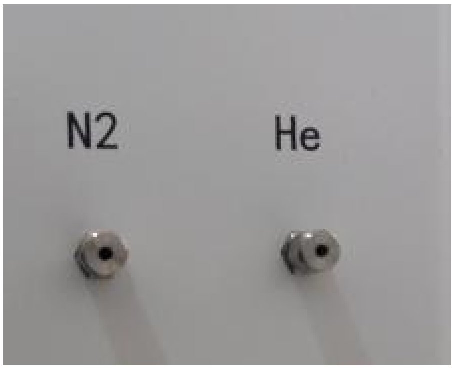
(4) Connection of the external air circuit of the main engine

1. Install the air line seal ring

Take out one Φ3 stainless steel external air pipe, and put a nut and 2 or 3 Φ3 seals on each end of the air pipe in turn. See Figure 1-13

graph 1-13

2. Connection of gas cylinder and instrument gas line



graph 1-14

(1) Connect the nitrogen line

Insert one end of the gas line into the "N2" gas interface of the instrument (see Figure 1-14 for N2 interface), tighten the nut, and connect the other end to the outlet of the pressure reducer 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 (see Figure 1-14 for the He interface), tighten the nut, and connect the other end to the outlet of the pressure reducer of the helium cylinder, and tighten the nut.

3. Connection of vacuum pump and instrument air line

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

As shown in Figure 1-15, according to the installation method of the bellows, one end of the bellows is connected to the air intake port of the main engine, and the other end is connected to the air intake port of the mechanical pump.

graph 1-15

(5) Installation of the main engine

1. Install Po pipe

(1) Take out the Po tube, and fit one Po tube nut and two Po tube seals in turn. The sealing ring is about 5mm away from the Po tube opening, as shown in Figure 1-16

graph 1-16

(2) Insert the Po tube into the Po analysis station interface of the instrument, and tighten the nut. The corresponding positions of the Po tube and each sample tube are installed according to the instrument identification.

graph 1-17

(6) Software installation

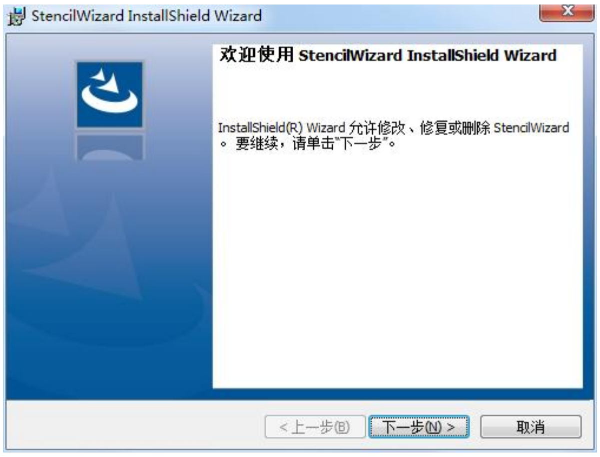
1. Installation of PC analysis software

Double-click to open the "StencilWizard Setup" installation package, open the software installation wizard, 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 complete, after completion, the desktop appears a shortcut icon "".

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



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

已做好修复程序的准备

向导准备开始安装。

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。

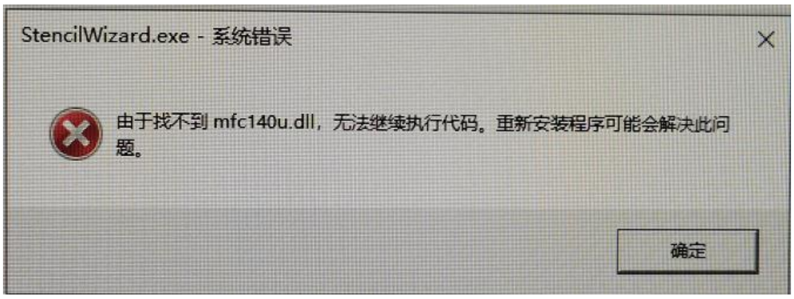
当前设置:
安装类型:
典型
目的地文件夹:
H:\StencilWizard\
用户信息:
姓名: Microsoft
公司: Microsoft

graph 1-18

This software is only used for data analysis 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 1-19, you need to install "mfc140u.dll" in the C drive 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 1-19

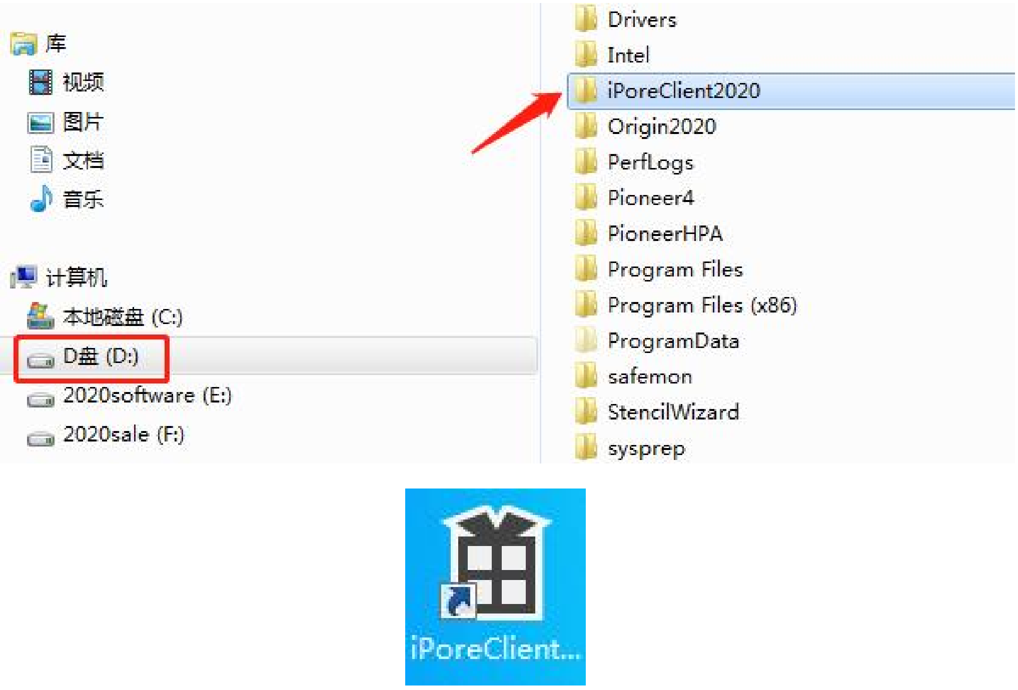
The system is windows10 X64

Step 1: Put mfc140u.dll in the SysWOW64 under C drive windows

Step 2: Run setup.exe

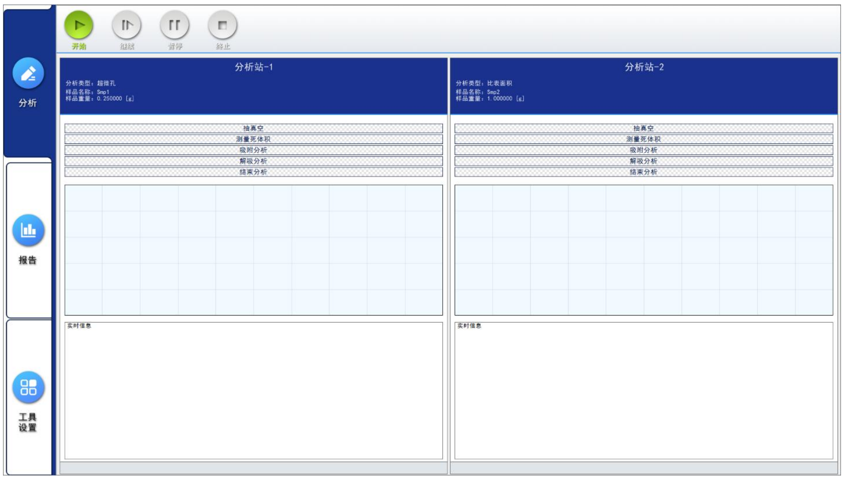
3. Installation of computer control terminal software

(1) First, connect the external computer and SSA7000 host with USB data cable, copy the computer control terminal software to the root directory of D disk, and create a shortcut on the desktop. As shown in Figure 1-20



graph 1-20

(2) Open the client, as shown in Figure 1-21.



分析类型：超微孔
样品名称：Smp1
样品重量：0.250000 [g]

分析类型：比表面积
样品名称：Smp2
样品重量：1.000000 [g]

graph 1-21

(3) Click the tool setting module, click "Load from the report", select any library file, and then click load to copy the system parameters to the current software control end.

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

从文件中载入设置

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 1-22

(4) After the above operation is completed, you can use an external computer to set measurement parameters, collect data, save reports, etc.

(7) Drive installation

To install the driver, follow these steps.

1.3.1 Installation of XP/Win7 system drivers

Before installing the driver, make sure that the data cable between the computer and the analysis host is correctly connected and turn on the computer.

- Find "My Computer" right-click to select properties, open device manager, then find "Universal Serial Bus Controller" and open, or directly find the position of the "!", as shown in the figure below.

通用串行总线控制器
Generic USB Hub
Generic USB Hub
Intel(R) 8 Series/C220 Series USB Enhanced Host
Intel(R) 8 Series/C220 Series USB Enhanced Host
USB Composite Device
USB Root Hub

- Find the location of the "!", double-click it, and select Custom Update Driver.
- Select the driver in the installation program "32-bit system driver /64-bit system driver" or update the "Drive folder" in the installation package.

1.3.2 Installation of Win10 system drivers

- Before installing the driver, you need to remove the prohibition program of win10 system first. The operation method is as follows:

** Precautions: Do not plug in the U disk when doing the following steps **

I. Open window Settings. There are three ways to open it:

A. Click the "window button" in the lower left corner, and then click the icon "gear"

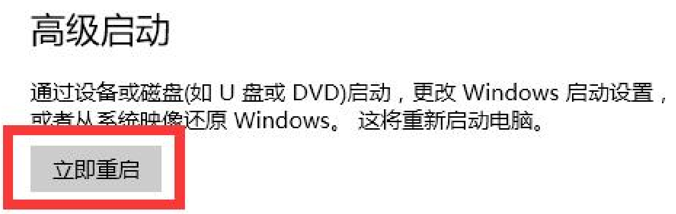
B. Right-click on the desktop, click "Show Settings", and then click "Home"

C. Click notifications, find and go to "All Settings."

2. Click "Update and Security"

3. Click "Restore"

4. Click "Start Up" under "Advanced Start Up". After clicking, the computer will restart.



通过设备或磁盘(如 U 盘或 DVD)启动，更改 Windows 启动设置，或者从系统映像还原 Windows。 这将重新启动电脑。

5. After restarting, select "Troubleshooting"

6. Select "Advanced Options" again

7. Select "Start Settings"

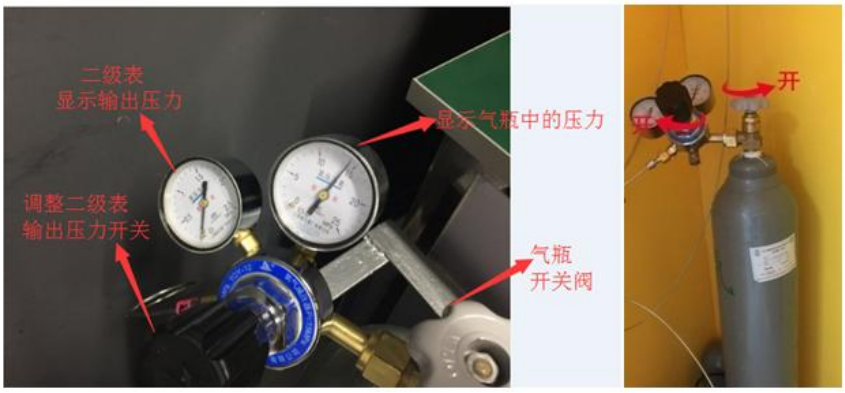
8. Click "Restart" and the computer will restart.

9. After the computer starts, press "7" to complete the setting.

After removing the ban program of win10 system, complete the driver installation by following the method of installing the driver according to XP/win7 system.

Chapter II Use

(1) Use of gas cylinders and pressure reducers



graph 2-1

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

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

2. Adjust the output pressure

Open the main valve of the gas cylinder, then turn the black knob to adjust the output pressure (clockwise adjustment opens the output, counterclockwise adjustment closes the output). Set the nitrogen and helium output pressures to 0.3MPa each. If the pressure adjustment exceeds 0.5 MPa, first close the main valve of the gas cylinder, disconnect the gas line from the pressure reducing valve, and purge the gas from the gauge. Then readjust the output pressure.

3. Use of gas cylinders

When not working, only the main valve of the cylinder needs to be closed. When using it again, the main valve of the cylinder can be opened directly without adjusting the output pressure again, so as to avoid the output pressure of the pressure divider valve from exceeding 0.5MPa.

4. Use of gas

In conventional low temperature nitrogen adsorption experiments, high purity nitrogen (purity 99.999%) is generally used for adsorbent/gas, and high purity helium (purity 99.999%) is used for dead volume quantitative gas.

(2) Use of degassing station

1. Sampling and weighing

(1) Determine the sample quantity

Typically, the sample to be analyzed should provide a surface area of 15 to 150 square meters, suitable for nitrogen adsorption analysis. Less than this will lead to unstable results, while more will prolong the analysis time. For samples with large specific surface areas, the sample size should be small. For small samples, weighing after degassing becomes crucial; even minor weighing errors can significantly impact the total weight. Thus, weighing techniques are extremely important. The sample weight should not be less than 30 milligrams. For powdered samples, use a long-necked funnel and add the sample to the bottom of the sample tube. When removing the funnel, slightly tilt the sample tube horizontally to prevent residual sample from falling onto the walls of the sample tube. For large particle samples, use tweezers for sampling (note, do not touch the sample with your fingers to avoid adhering oils to the sample surface).

Sample dosage 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 is less than 0.01

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

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

① Take the sample tube and stopper with the label, plug the sample tube with the stopper, and record the sample tube number and stopper number in the record book.

② Place the container (which can be a graduated cylinder or glass cup, etc., used to place the round-bottomed sample tube) on the balance to weigh and remove the skin so that the balance is stable at zero.

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

graph 2-2

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 15mm. (See Figure 2-4)

graph 2-3

⑤ Add the plug again, weigh the sample tube and the plug containing the sample, and record the total weight of the sample tube before degassing m2.

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

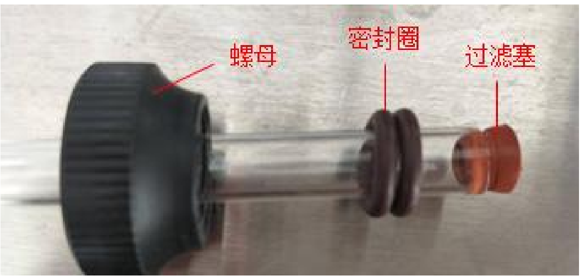
2. Install the sample tube

Most of the sample surface adsorbs a large number of pollutants and impurities in the room temperature environment, before the analysis to remove these dirty things, the sample surface cleaning. The sample is heated under vacuum, so as to remove the dirty things on the sample surface, called sample degassing.

(1) If there is a plug or sample tube at the degassing port of the degassing system, first loosen the nut and remove the plug as shown in Figure 2-4 left; see Figure 2-4 right for the installation of the plug.

graph 2-4

(2) Install 1 nut, 2 sealing rings and 1 filter plug on the sample tube opening (for non-powder samples, the filter plug can be omitted), as shown in Figure 2-5

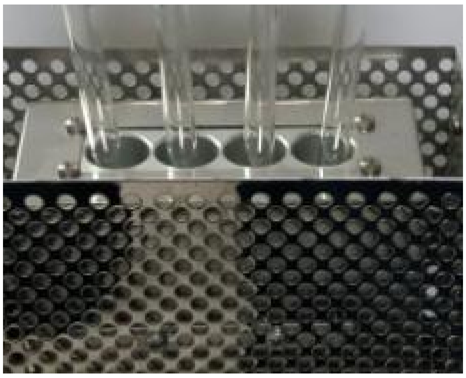


graph 2-5

(3) Install the sample tube on the degassing position. The sample tube should be kept vertical to the ground, pushed up tightly, and then tightened with hand.

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

(5) Put all the sample tubes to be processed into the heating tank, so that the ball belly of the sample tube is exactly buried in the heating furnace. See Figure 2-6.



graph 2-6

3. Set the degassing mode

300.0℃

-0.01 KPa

真空 1

注气 3

300 温度 ℃

120 时间 min

预处理中 80%

graph 2-7

(1) Click the "Temperature" and "Time" selection boxes to input the temperature and time to

be processed respectively. For samples with unknown processing duration, you can check the "AI" key at the same time. 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"), which can be selected according to the type and state of the sample. After selection, the instrument will run the corresponding processing scheme according to the sample type.

(3) Click the "S" key to start degassing. At this time, the heating furnace will automatically heat up to the target temperature.

4. Deaeration operation

(1) Start of vacuum pump: open the switch on the pump, the pump starts to work; close the switch, the pump stops working.

(2) Sample processing: Click the "S" key on the touch screen to start degassing. At this time, the heating furnace begins to heat up.

Note: Some samples are too light (such as graphene, ultrafine silica, boehmite, etc.) and may enter the gas line, resulting in sample quality loss and processor gas line contamination. When handling such samples, a filter plug should be inserted into the sample tube opening.

(3) In the process of treatment, it gradually shows negative pressure with the decrease of actual vacuum.

5. Weigh again after degassing

(1) After degassing is complete, there will be a "beep" alarm and a pop-up prompt. At this point, remove the sample tube from the heating bath and place it in the cooling bath to cool down to room temperature. However, note that the heating furnace is still at high temperature (during the cooling process). Do not touch the walls or opening of the heating furnace to avoid burns from the high temperature.

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

(3) Weigh again (net weight of samples)

Remove the sample tube and quickly insert the plug of the corresponding sample tube into the tube opening to prevent the dried sample from contacting with air again. Then weigh the sample tube on the balance and record it as m4. Subtract m1 from m4 to obtain the net weight of the dried sample m.

Note that if m (type of sample after drying) is greater than m3 (weight of sample before drying), the influence of weighing error or operation error should be considered. If the difference is large, the sample should be treated again according to the above steps.

(3) Use of instrument control terminal software

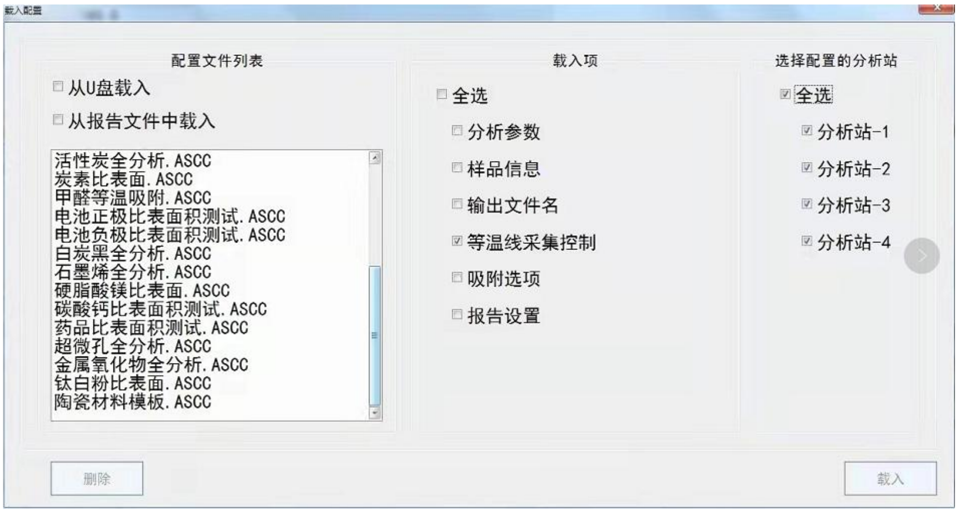
1. Use the system template directly

(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 2-8

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 2-8

(2) You can select different configuration files, load parameters, and apply the 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 2-9


载入配置

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
金属氧化物全分析.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 2-9

(3) Modify the basic parameters of the sample, such as the sample name and weight (the weight of the sample after degassing), and create a new file name for the sample storage, as shown in Figure 2-10. If you need to keep the sample name and file name consistent, you can select the lock key, as shown in Figure 2-11

The file name is automatically saved according to the current test date. 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[°C]：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 2-10

名称：Sample1

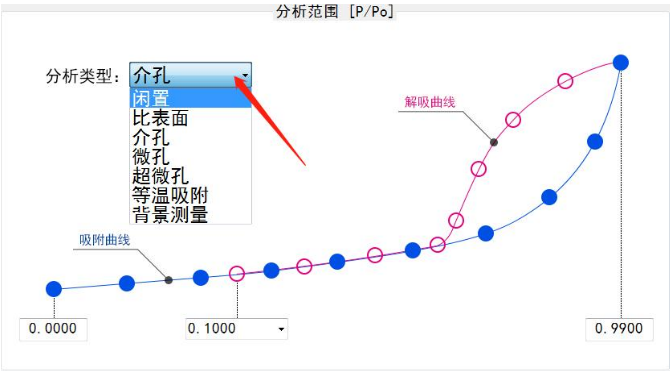
重量[g]：1.000000

Sample1

graph 2-11

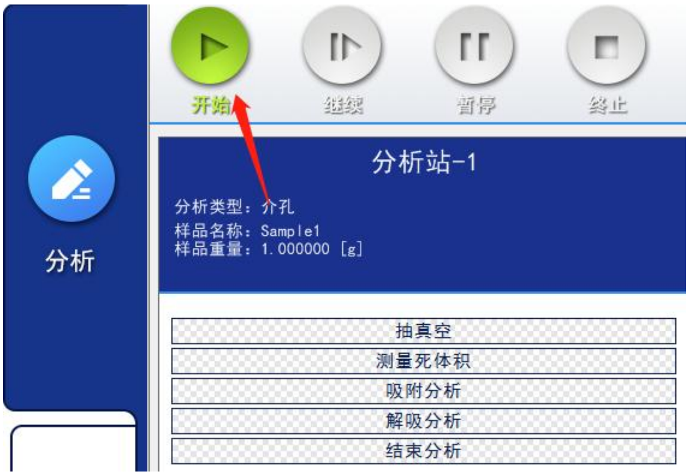
Select the lock key ""	**Unlock the lock key ""**
The background of the lock key becomes yellow: the file name is stored in accordance with the sample name	The background of the lock key becomes blue: The file name can be modified at will, but it cannot have special characters, such as:? / * # $!

(4) Select the analysis type, as shown in Figure 2-12



分析类型：介孔

graph 2-12

(5) According to the above method, set the corresponding parameters of each analysis station that needs to work, click save and close to return to 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 2-13

Non-pressurized mode	**Pressure control mode**
(Incremental control of fixed gas injection)	(P/Po incremental/precise control)

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

分析类型: 超微孔

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

2. Custom templates

(1) Open the software and click Analysis Station 1 to enter the parameter setting interface, as shown in Figure 2-14

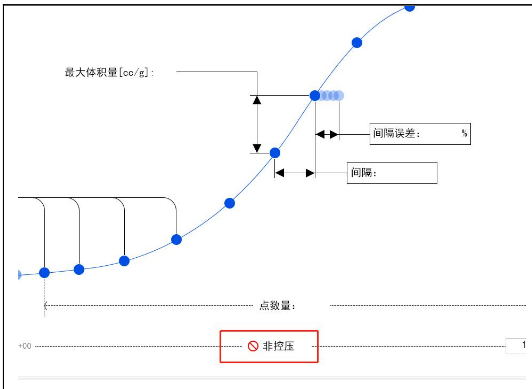
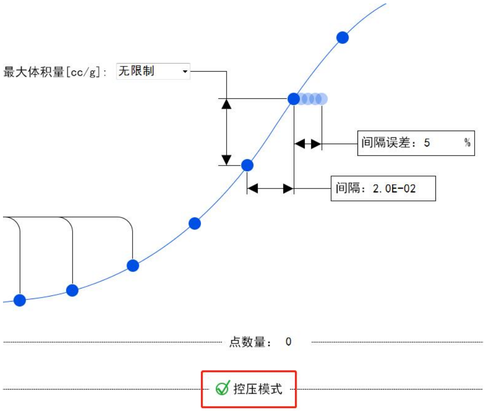
graph 2-14

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

(2) Set the basic parameters and analysis types of samples, referring to the "System Template" in this chapter.

(3) Set the isotherm line acquisition template, and add, modify or delete selected sections according to the analysis type.

graph 2-15

 最大体积量[cc/g]: 间隔误差: % 间隔: 点数量: +00 非控压 1	 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 isotherm data points can be customized to accurately determine the P/Po interval

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

② Non-pressure control mode setting, as shown in Figure 2-16

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

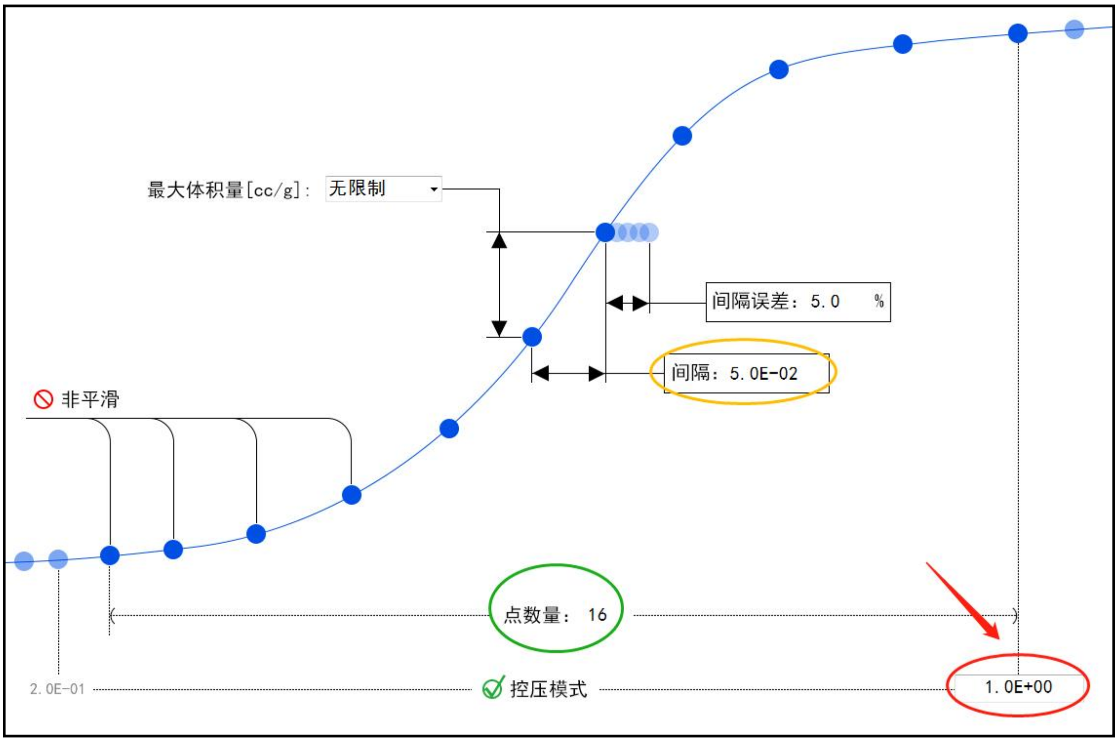
☑ 自动斜率控制: 0.050000

☑ 平衡时间[秒]: 200

□ 斜率与时间必须同时满足

graph 2-16

③ Control pressure mode setting. Interval error is set to 3-5%, as shown in Figure 2-17.



graph 2-17

- 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", then the number of points is automatically calculated.
- Check automatic gas flow setting 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

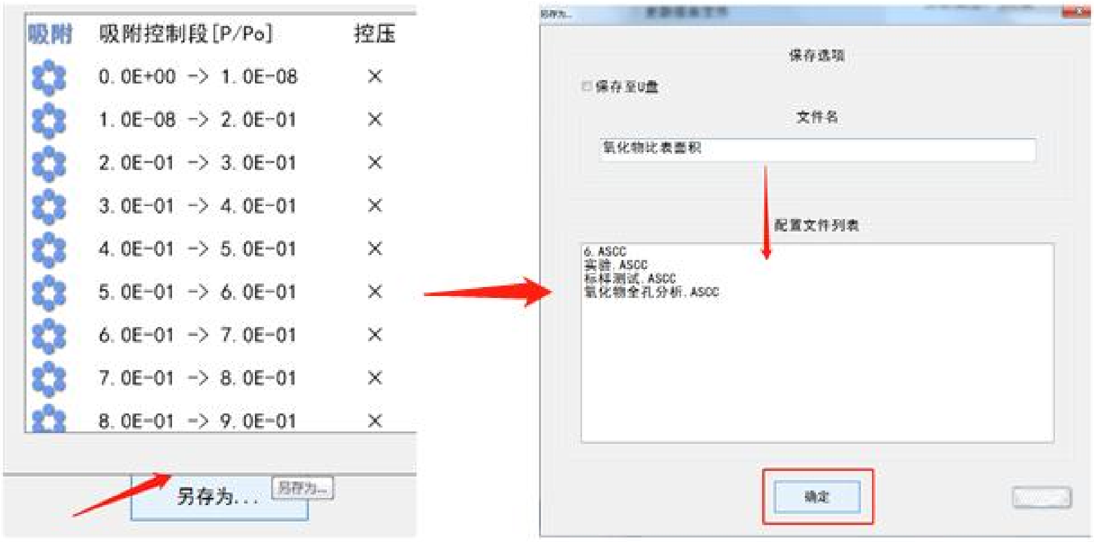
  ☑ 自动斜率控制：0.050000

  ☐ 平衡时间[秒]：150

  ☐ 斜率与时间必须同时满足

- As above, set the selected paragraph and save it

(4) Save the custom template. Select Save as to store the custom template.



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 2-18

(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 2-19

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

Chapter three Analytical samples

(1) 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

Sample weighing

Sample degassed

Install sample tube

Add liquid nitrogen

Analysis preparation

EOT

1. Clean and label the sample tube

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

(1) First, confirm that the ultrasonic tank is clean. Add about 500 ml of hot water and 5 ml of detergent or surfactant. Put the sample tube and filling rod into the ultrasonic cleaning for about 15 minutes and take it out (see Figure 3-1).

graph 3-1

(2) Put on rubber gloves to remove the sample tube and clean the inside of the tube with a special brush.

(3) Clean the sample tube with alcohol or acetone.

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

graph 3-2

(5) Place the sample tube and filling rod on the shelf to dry naturally or use the oven or vacuum oven, set 110 degrees for 2 hours to dry for standby.

(6) Clean the plug with clean silk cloth and install it on the sample tube.

(7) Label the sample tube and stopper.

(8) Plug the sample tube with a plug (see Figure 3-3).

graph 3-3

2. Sample weighing

(1) Determine the sample analysis amount before weighing the sample.

Typically, the sample to be analyzed can provide an area of 15 to 150 square meters, suitable for nitrogen adsorption analysis. Less than this will lead to unstable results, while more will prolong the analysis time. For samples with large specific surface areas, the sample size should be small. For small samples, weighing after degassing becomes crucial, as even minor weighing errors can significantly impact the total weight. This is the weighing technique

The technique is crucial. The sample weight should not be less than 30 milligrams. For powdered samples, use a long-necked funnel to add the sample to the bottom of the sample tube. When removing the funnel, slightly tilt the sample tube to prevent any residual sample from falling onto the walls of the tube. For larger particle samples, use tweezers to add the sample. (Refer to Appendix 8 for analytical quantities)

⚠ **Do not touch the sample with your fingers to avoid sticking grease to the surface of the sample.**

(2) Weigh the sample.

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

① Take the sample tube and stopper with the label, plug the sample tube with the stopper, and record the sample tube number and stopper number in the notebook.

② Place the container (which can be a graduated cylinder or glass cup, etc., used to place the round-bottomed sample tube) on the balance to weigh and remove the skin so that the balance is stable at zero.

③ Place the sample tube and stopper together with the support on the balance to weigh, and record the weight m1 of the empty sample tube and stopper after the balance value is stable. (See Figure 3-4)

graph 3-4

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 15mm. (See Figure 3-5)

graph 3-5

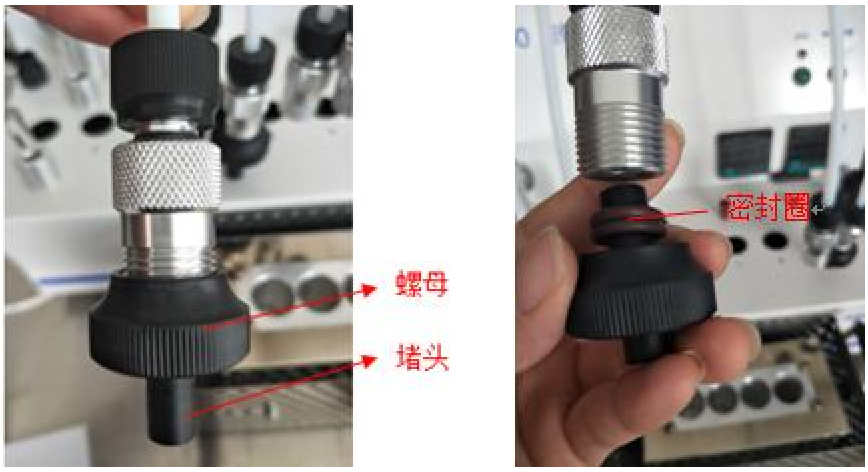
⑤ Add the plug again, weigh the sample tube and the plug containing the sample, and record the total weight of the sample tube before degassing m2.

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

3. Sample degassing and drying preparation

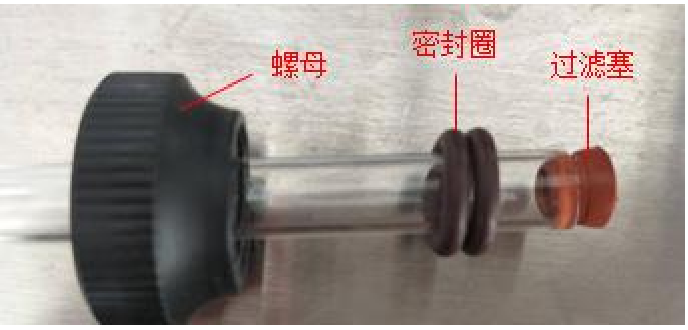
Most of the sample surface adsorbs a large number of pollutants and impurities in the room temperature environment, before the analysis to remove these dirty things, the sample surface cleaning. The sample is heated under vacuum, so as to remove the dirty things on the sample surface, called sample degassing.

⑴ If the vent of the preprocessor is blocked, first loosen the nut and remove the plug (see Figure 3-6 left); see Figure 3-6 right for the installation of the plug.



graph 3-6

(2) Remove the rubber plug from the sample tube, and install the nut, sealing ring and filter plug (if the sample is not powder, it can be omitted) (see Figure 3-7).



graph 3-7

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

(4) Insert the connected sample tube into the heating furnace. (See Figure 3-8)

graph 3-8

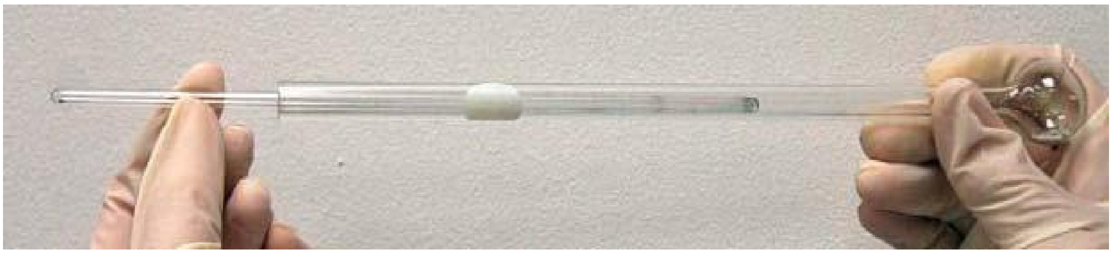
(5) Set the temperature of pretreatment according to the characteristics of the sample. The upper limit of the temperature is 400℃ (refer to the preprocessor manual for the setting method).

(6) After completing the pretreatment, remove the sample from the interface of the preprocessor and quickly seal the sample tube opening with the original stopper to prevent the dried sample from coming into contact with air again. Place the sample tube on a balance to weigh it, and record m4. Subtract m1 from m4 to obtain the net weight of the sample after drying.

4. Install the sample tube

(1) Remove the plug from the pipe;

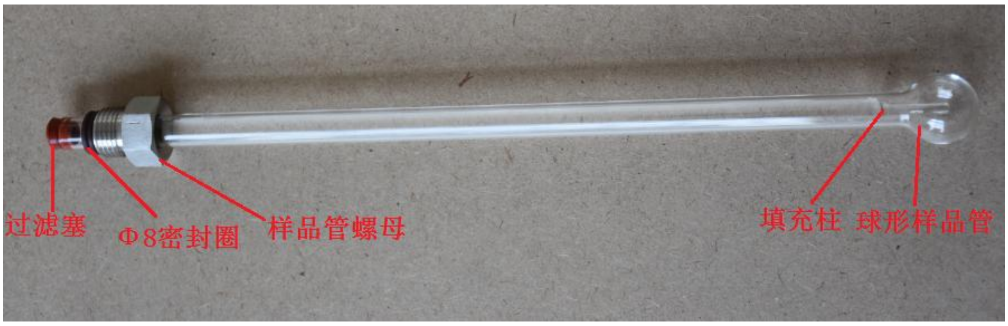
(2) Load the filling rod and put the sample tube and filling rod horizontally; (see Figure 3-9)



graph 3-9

Vertical downward installation of the filling rod will break the sample tube.

(3) After loading the filling rod, install the nut, 2 sealing rings and the filter plug (if not powder sample, the filter plug can be omitted) (see Figure 3-10)



graph 3-10

(4) Put the sample tube on the analysis port, push it up tightly, hold the bottom of the sample tube with one hand, and tighten the nut with the other hand, then turn it half a circle to one circle with a wrench.

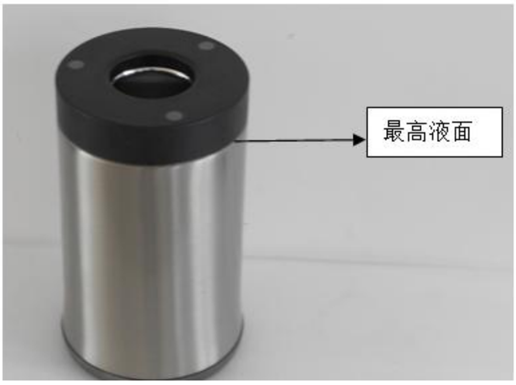
5. Add liquid nitrogen to the Dewar flask

B⚠e careful when adding liquid nitrogen to avoid scalding from low temperatures.

(1) Wear protective equipment, wear protective goggles and warm gloves.

(2) When adding liquid nitrogen to the Dewar bottle, reduce the thermal shock of the Dewar bottle bit by bit

Strike and prevent the liquid nitrogen from splashing. Make the height of the liquid nitrogen in the liquid nitrogen cup as shown below (see Figure 3-11).



graph 3-11

(3) Place the liquid nitrogen cup on the lifting tray of the instrument.

6. Analysis preparation

Make sure everything is ready to start analyzing the samples.

(1) Open the total pressure gauge of helium and nitrogen cylinders, adjust the pressure reducing valve to the output pressure 0.3-0.5MPa.

(2) Turn on the power switch of the instrument host and mechanical pump.

(2) Setting measurement parameters

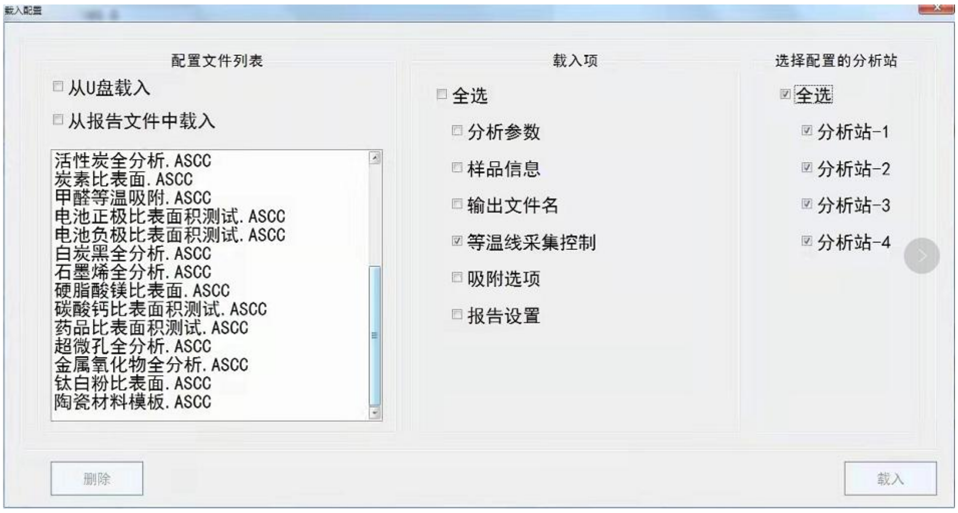
1. Use system templates

(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 3-12

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 3-12

(2) You can select different configuration files, load parameters, and apply the analysis parameters to each analysis station. After setting, click the "Load" item in the lower right corner to complete the loading. As shown in Figure 3-13



□ 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
金属氧化物全分析. ASCC
钛白粉比表面. ASCC
陶瓷材料模板. ASCC

□ 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

☑ 全选
☑ 分析站-1
☑ 分析站-2
☑ 分析站-3
☑ 分析站-4

graph 3-13

(3) Modify the basic parameters of the sample, such as the sample name, weight (the weight of the

sample after degassing), and create a new file name for the sample storage, as shown in Figure 3-14. If you need to keep the sample name and file name consistent, you can select the lock key, as shown in Figure 3-15

The file name is automatically saved according to the current test date, and the sample name and file name cannot 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 3-14

样品信息

名称： Sample1

重量[g]： 1.000000

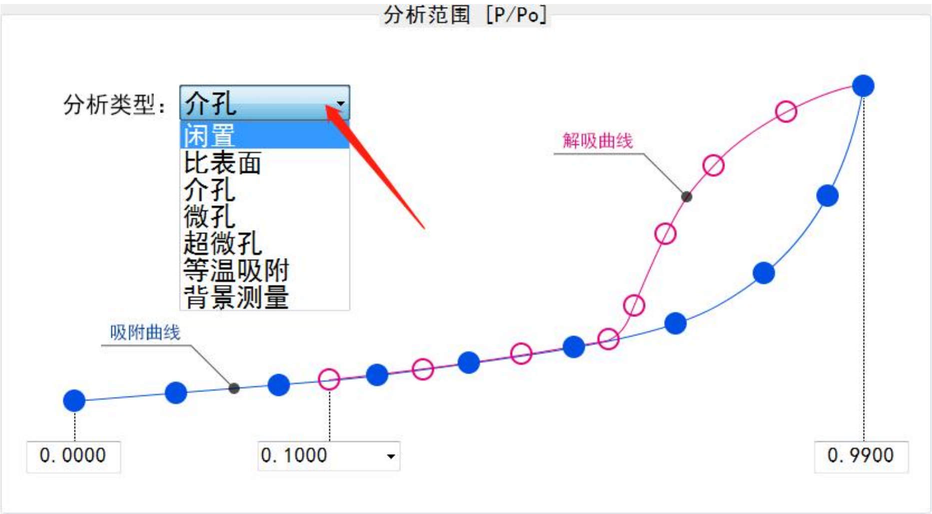
文件名

Sample1

graph 3-15

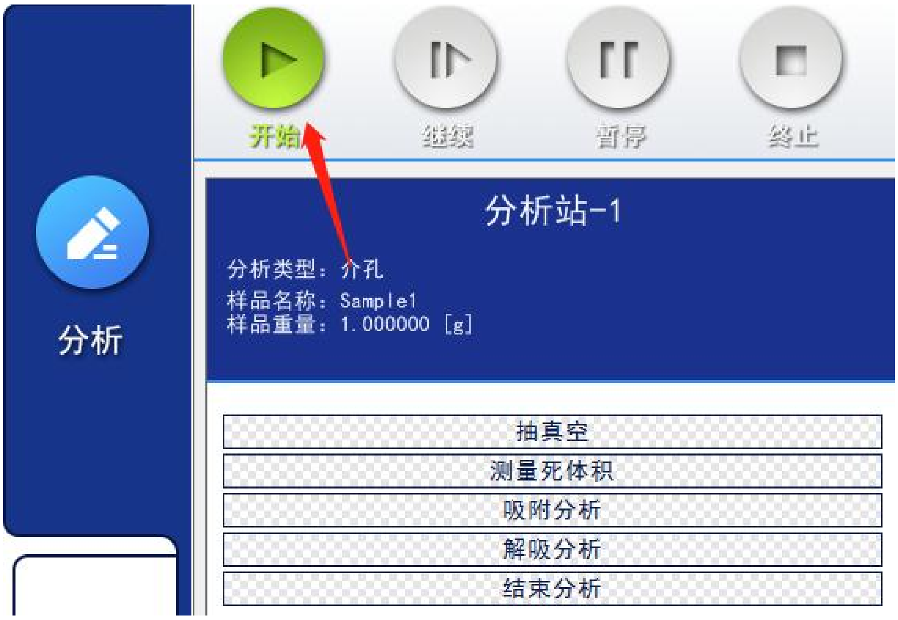
Select the lock key ""	**Unlock the lock key ""**
The background of the lock key becomes yellow: the file name is stored in accordance with the sample name	The background of the lock key becomes blue: The file name can be modified at will, but it cannot have special characters, such as:? / * # $!

(4) Select the analysis type (set the P/Po analysis range), as shown in Figure 3-16



分析类型：介孔

graph 3-16

(5) According to the above method, set the corresponding parameters of each analysis station that needs to work, click save and close to return to the main interface. Check whether the gas cylinder and liquid nitrogen are in place, click the "Start" button to run the analysis.

分析站-1

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

graph 3-17

2. Custom templates

(1) Open the software and click Analysis Station 1 to enter the parameter setting interface, as shown in Figure 3-18

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)

Sample1

□ 更新报告文件

分析类型：超微孔

2.0E-08 0.0500 0.9900

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 3-18

(2) Set the basic parameters and analysis types of samples, as described above.

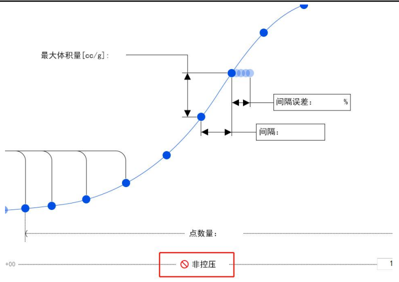
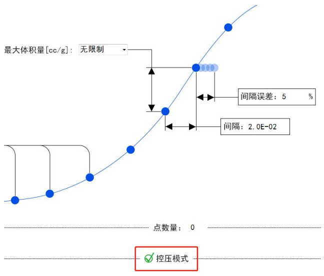
(3) Set the isotherm line acquisition template, and add, modify and delete selected sections according to the analysis type.

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 3-19

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

Non-pressurized mode (Incremental control of fixed gas injection)	**Pressure control mode** (P/Po incremental/decreasing precision control)
	
Satisfy most material analysis	The number of isotherm data points can be customized to accurately determine the P/Po interval

② Non-pressure control mode setting. See Figure 3-20

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

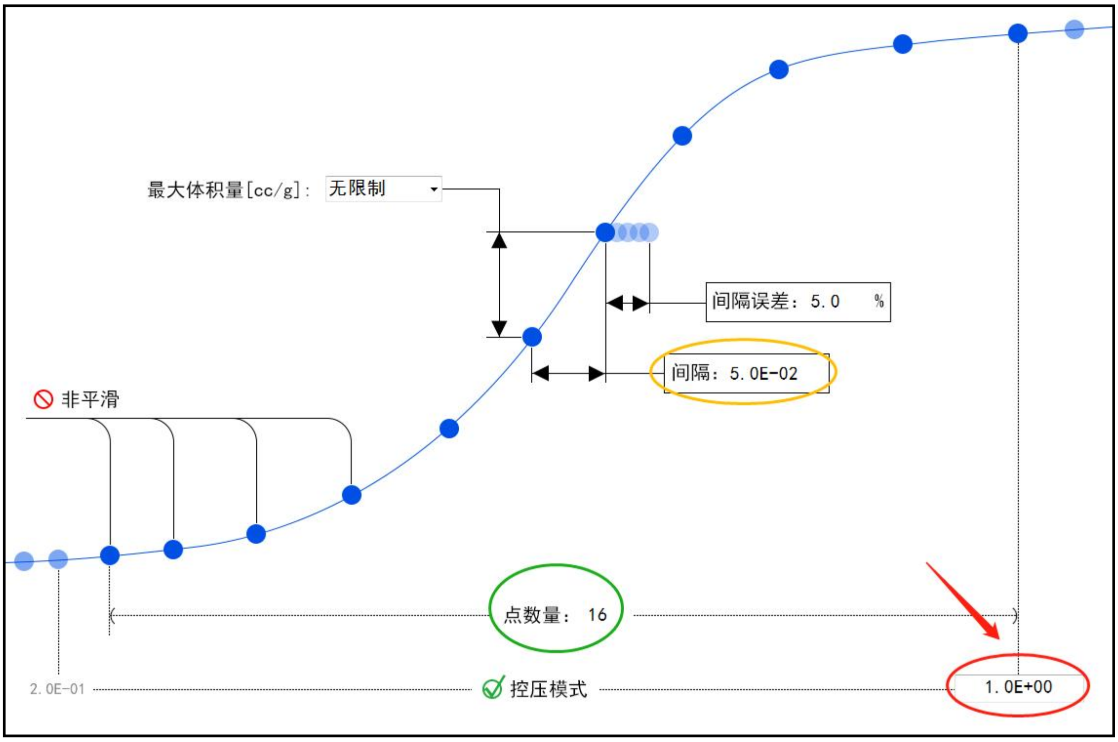
☑ 自动斜率控制： 0.050000

☑ 平衡时间[秒]： 200

□ 斜率与时间必须同时满足

graph 3-20

③ Control pressure mode setting. Interval error is set to 3-5%, as shown in Figure 3-21.



最大体积量[cc/g]： 无限制

间隔误差： 5.0 %

间隔： 5.0E-02

点数量： 16

graph 3-21

- 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", then the number of points is automatically calculated.
- Check automatic gas flow setting 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

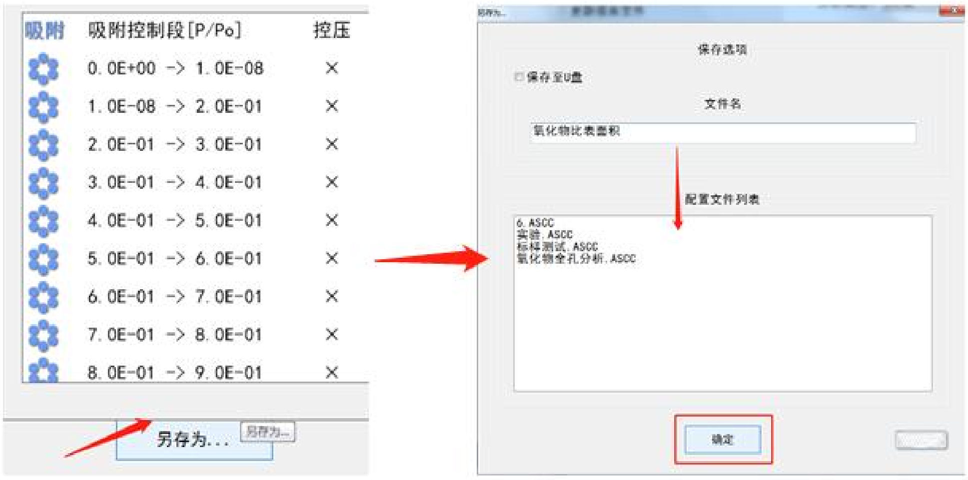
☑ 自动斜率控制： 0.050000

☐ 平衡时间[秒]： 150

☐ 斜率与时间必须同时满足

- As above, set the selected paragraph and save it

(4) Save the custom template. Select Save as to store the custom template.



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 3-22

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

吸附质选择：Nitrogen-77.35K

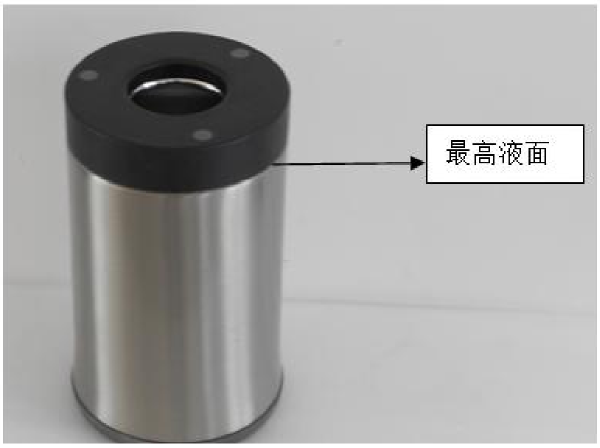
graph 3-23

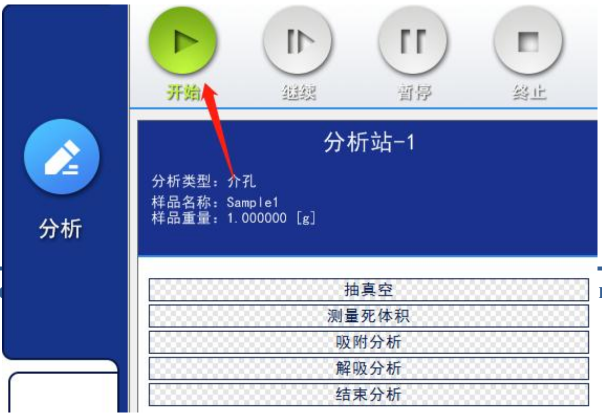
(6) Return to the main interface, after the preparation is completed, click "Start" to start.

(3) Automatic analysis

1. Start analysis

After the preparation of the sample is completed, turn on the vacuum pump and gas cylinder, add liquid nitrogen to the Dewar bottle, and pour liquid nitrogen to 4-5cm away from the mouth of the Dewar bottle. After filling the liquid nitrogen, place the Dewar bottle on the instrument lifting tray, click start analysis, and the instrument will automatically start analysis.



graph 3-24

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

graph 3-25

2. Automatic operation process

Step 1: Vacuum preparation.

Step 2: Measure the dead volume/cold free space. At this time, the Dewar bottle will rise and the sample tube will be immersed in liquid nitrogen. When the dead volume test is completed, the Dewar bottle will drop to the initial position.

Step 3: Adsorption analysis. In this process, the instrument will inject the adsorbent gas and complete the drawing of the adsorption isotherm and the calculation of BET\Langmuir\t-Plot and other models. After completion, the data will be displayed in the dialog box of each analysis station.

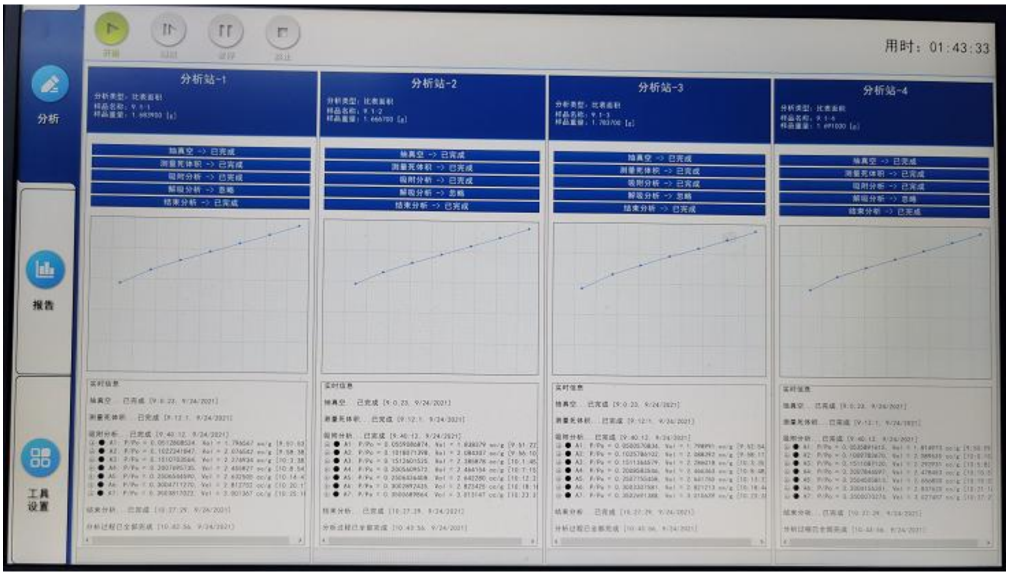
Step 4: Analysis. In this process, the instrument will extract the adsorbed gas and complete the drawing of desorption isotherm and calculation of BET\Langmuir\t-Plot and other models. After completion, the data will be displayed in the dialog box of each analysis station.

Step 5: End the analysis and restore the instrument to its original state.

Its function is: to unload and compensate for the pressure inside the sample tube. Due to the large amount of nitrogen injected during the adsorption process, this process uses a vacuum method to expel the large amount of nitrogen adsorbed. After the process is complete, the Dewar flask automatically descends and pours the remaining liquid nitrogen into the liquid nitrogen tank; if continuous testing is not required, simply close the main unit, vacuum pump, and gas cylinder valve; do not disassemble the sample tube, if disassembled, install a plug or clean empty tube to prevent contamination of the gas line; close the software after data is saved and processed.

3. End the analysis

(1) During the test, the software interface automatically displays the test results of each analysis station in real time, as shown in Figure 3-25



graph 3-26

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


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

(2) Check the test report on the computer control end

① In the report tool group, click Report Library to open all test files in the library. You can select, cancel, display, export and other operations here as shown in the figure

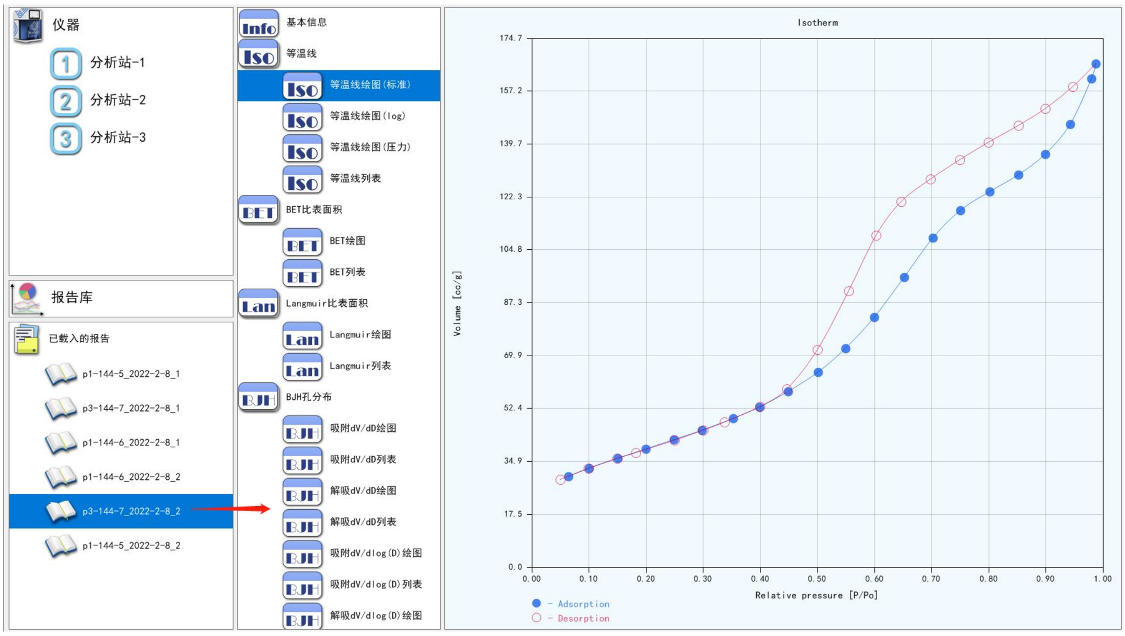
201	9.1-2	8.7595 m2/g	1.261200 g	0.999936	9.1-2_2022-1-20_1.iPrd	2022/01/20, 11:36
202	9.1-3	8.7151 m2/g	1.433300 g	0.999938	9.1-3_2022-1-20_1.iPrd	2022/01/20, 11:36
203	9.1-1	8.7225 m2/g	1.230900 g	0.999941	9.1-1_2022-1-20_1.iPrd	2022/01/20, 11:36
204	5.78-2	5.7048 m2/g	2.017300 g	0.999946	5.78-2.iPrd	2022/01/21, 09:18
205	5.78-3	5.7321 m2/g	2.064500 g	0.999922	5.78-3_2022-1-21_1.iPrd	2022/01/21, 11:34
206	5.78-1	5.7380 m2/g	2.058900 g	0.999931	5.78-1_2022-1-21_1.iPrd	2022/01/21, 11:34
207	5.78-2	5.8167 m2/g	2.017300 g	0.999982	5.78-2_2022-1-21_1.iPrd	2022/01/21, 11:34
208	5.78-3	5.7207 m2/g	2.064500 g	0.999953	5.78-3_2022-1-21_2.iPrd	2022/01/21, 13:58
209	5.78-2	5.7384 m2/g	2.017300 g	0.999957	5.78-2_2022-1-21_2.iPrd	2022/01/21, 13:58
210	5.78-1	5.7115 m2/g	2.058900 g	0.999960	5.78-1_2022-1-21_2.iPrd	2022/01/21, 13:58
211	144-5	140.3571 m2/g	0.195800 g	0.999991	p1-144-5_2022-2-8_1.iPrd	2022/02/08, 08:57
212	144-7	142.5706 m2/g	0.197100 g	0.999989	p3-144-7_2022-2-8_1.iPrd	2022/02/08, 08:57
213	144-6	141.1817 m2/g	0.192600 g	0.999987	p1-144-6_2022-2-8_1.iPrd	2022/02/08, 08:57
214	144-6	141.4262 m2/g	0.192600 g	0.999986	p1-144-6_2022-2-8_2.iPrd	2022/02/08, 14:02
215	144-7	142.4382 m2/g	0.197100 g	0.999986	p3-144-7_2022-2-8_2.iPrd	2022/02/08, 14:02
216	144-5	140.4141 m2/g	0.195800 g	0.999984	p1-144-5_2022-2-8_2.iPrd	2022/02/08, 14:02
217	2022-05-05-HYQ-01-01-...	850.9531 m2/g	0.069200 g	0.996248	2022-05-05-HYQ-01-01-...	2022/05/05, 10:43
218	2022-05-05-HYQ-01-01-...	604.6510 m2/g	0.053500 g	0.996630	2022-05-05-HYQ-01-01-...	2022/05/05, 10:43

graph 3-27

② Select the data to be viewed, click load, and the target file will be displayed in the report column on the left. Here you can quickly view isotherms and BET models, and more model analysis should be used with data processing software.

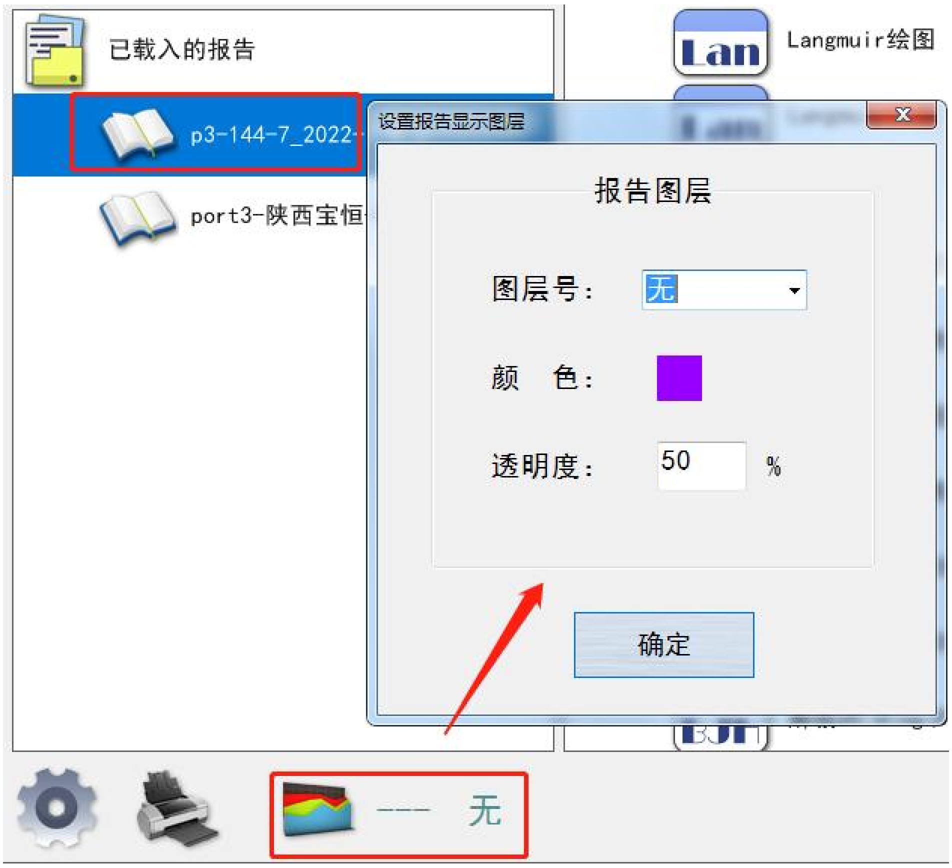
203	9.1-1	8.7225 m2/g	1.230900 g	0.999941	9.1-1_2022-1-20_1.iPrd	2022/01/20, 11:36
204	5.78-2	5.7048 m2/g	2.017300 g	0.999946	5.78-2.iPrd	2022/01/21, 09:18
205	5.78-3	5.7321 m2/g	2.064500 g	0.999922	5.78-3_2022-1-21_1.iPrd	2022/01/21, 11:34
206	5.78-1	5.7380 m2/g	2.058900 g	0.999931	5.78-1_2022-1-21_1.iPrd	2022/01/21, 11:34
207	5.78-2	5.8167 m2/g	2.017300 g	0.999982	5.78-2_2022-1-21_1.iPrd	2022/01/21, 11:34
208	5.78-3	5.7207 m2/g	2.064500 g	0.999953	5.78-3_2022-1-21_2.iPrd	2022/01/21, 13:58
209	5.78-2	5.7384 m2/g	2.017300 g	0.999957	5.78-2_2022-1-21_2.iPrd	2022/01/21, 13:58
210	5.78-1	5.7115 m2/g	2.058900 g	0.999960	5.78-1_2022-1-21_2.iPrd	2022/01/21, 13:58
√211	144-5	140.3571 m2/g	0.195800 g	0.999991	p1-144-5_2022-2-8_1.iPrd	2022/02/08, 08:57
√212	144-7	142.5706 m2/g	0.197100 g	0.999989	p3-144-7_2022-2-8_1.iPrd	2022/02/08, 08:57
√213	144-6	141.1817 m2/g	0.192600 g	0.999987	p1-144-6_2022-2-8_1.iPrd	2022/02/08, 08:57
√214	144-6	141.4262 m2/g	0.192600 g	0.999986	p1-144-6_2022-2-8_2.iPrd	2022/02/08, 14:02
√215	144-7	142.4382 m2/g	0.197100 g	0.999986	p3-144-7_2022-2-8_2.iPrd	2022/02/08, 14:02
√216	144-5	140.4141 m2/g	0.195800 g	0.999984	p1-144-5_2022-2-8_2.iPrd	2022/02/08, 14:02
217	2022-05-05-HYQ-01-01-...	850.9531 m2/g	0.069200 g	0.996248	2022-05-05-HYQ-01-01-...	2022/05/05, 10:43
218	2022-05-05-HYQ-01-01-...	604.6510 m2/g	0.053500 g	0.996630	2022-05-05-HYQ-01-01-...	2022/05/05, 10:43

graph 3-28



graph 3-29

③ If you need more than one isotherm, you can select the target file, click display, and report multiple reports in the same layer number.

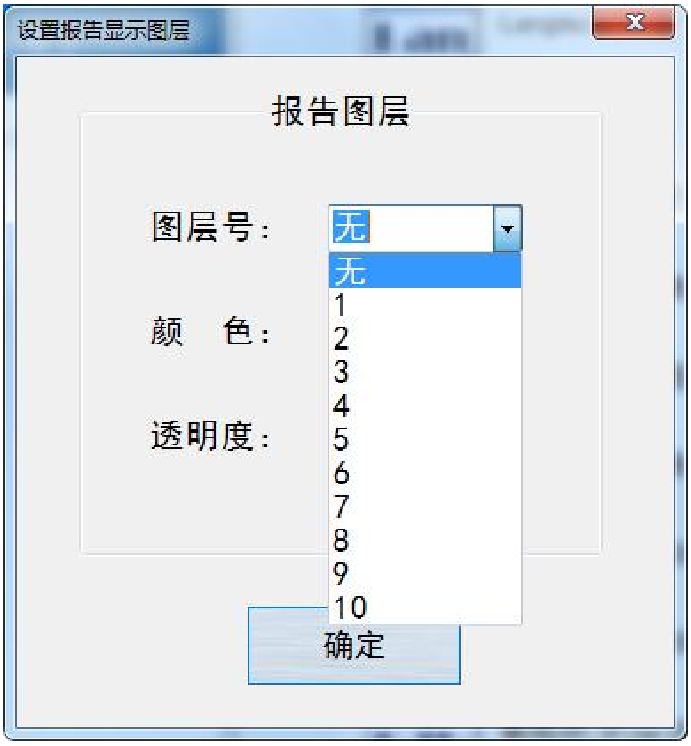


图层号：无

颜　色：

透明度：50 %

graph 3-30



图层号：无

颜　色：

透明度：

graph 3-31

Chapter IV. Theoretical overview

(I) Definition and overview

4.1.1 Overview of surface features

The surface characteristics of powder materials are extremely complex, especially for porous nanoscale powders. Due to the significant difference between the state of atoms on the surface and those within the material, the surface features encompass not only the general surface but also the inner surfaces connected to the pores. To better characterize and analyze the surface features of materials, data analysis is divided into two main categories: specific surface area and porosity.

4.1.2 Definition of surface features

4.1.2.1 Concept of specific surface area

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

4.1.2.2 Overview of holes

(1) The concept of holes

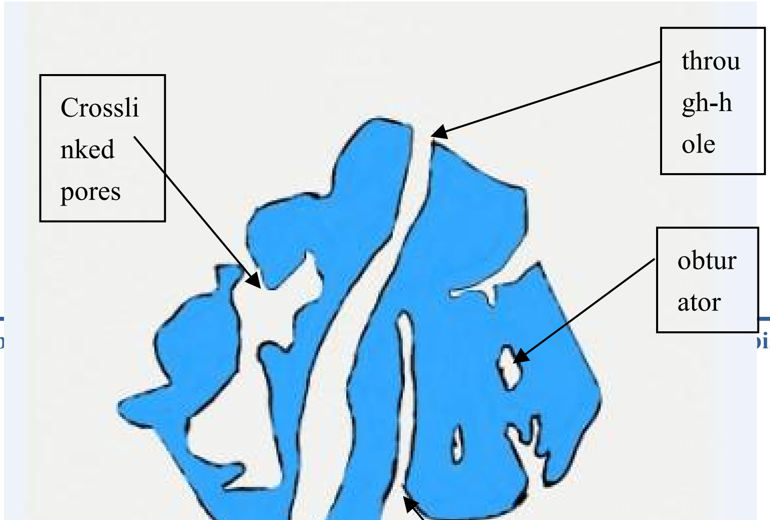
Channels or cavities within a solid material can be referred to as pores. In addition to the pores within a solid material, the space between agglomerates of solid particles (such as cracks or voids) can also be referred to as pores.

(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 ease of characterization and analysis, the International Union of Pure and Applied Chemistry (IUPAC) has recognized the classification of pore sizes based on their slit width into three categories: macropores (macropores):>50nm; mesopores (mesopores): 2~50nm; micropores (micropores): <2nm. Micropores are further divided into submicropores and ultra-micropores; dubinin further classifies micropores into ultra-micropores smaller than 0.7nm and submicropores between 0.7-2.0nm.

In addition to the quantitative classification in scientific research, according to the characteristics of the pore type, it is divided into: through-hole, closed hole, cross-linked hole, blind hole, etc., among which the cross-linked hole and blind hole include cylindrical hole, slit hole, spherical hole, etc. (see Figure 4-1)

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



graph 4-1

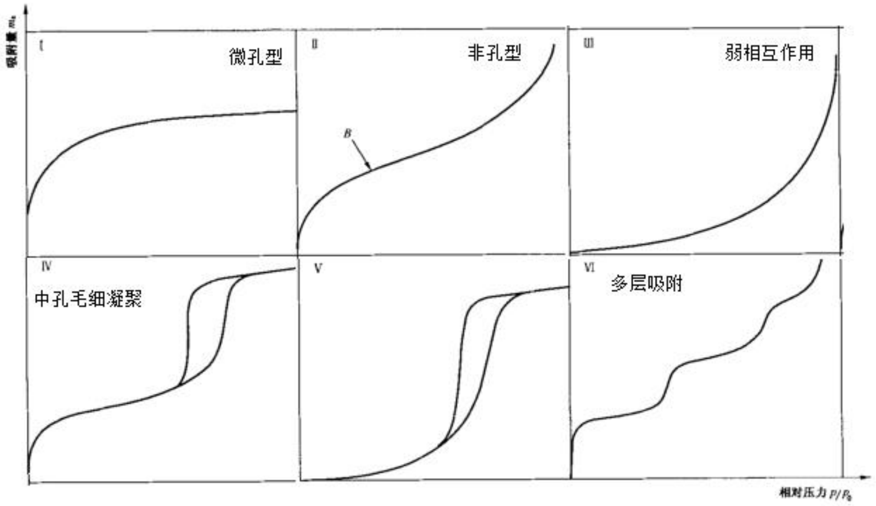
(2) Analysis methods

4.2.1 Analysis method

The characterization of surface properties is typically divided into two major categories: specific surface area and porosity. Various methods are used for specific surface area analysis, including gas adsorption, gas permeation, mercury porosimetry, bubble point method, and small-angle X-ray diffraction, among others. Among these, the gas adsorption method is widely adopted across various industries both domestically and internationally due to its scientific testing principles, reliable testing process, and consistent test results. It has gradually replaced other specific surface area testing methods and is recognized as the most authoritative method. The gas adsorption method (also known as static capacity method) involves measuring the adsorption and desorption amounts of adsorbate gases at different relative pressures (isotherms). Simply put, the gas adsorption method uses gas molecules as a "yardstick," analyzing the consumption of adsorbate molecules and the state of the adsorption-desorption curve to describe the surface characteristics of materials.

The commonly used gas adsorption method is nitrogen adsorption. At a constant temperature (usually the boiling point of the adsorbate gas, for nitrogen it is 77.35k), the curve showing how the adsorption capacity varies with relative pressure (P/P0) is called the adsorption-desorption isotherm. This is the most important manifestation of the adsorption characteristics of solid materials. Based on data points from different relative pressure ranges on the adsorption-desorption isotherm, various theoretical models can be used to analyze different surface properties

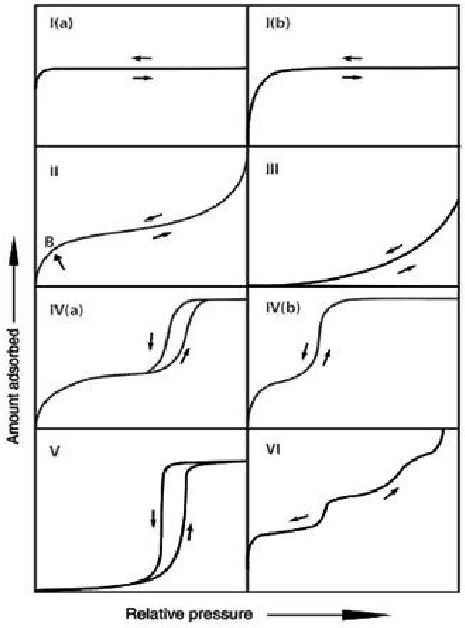
4.3.1.1 Classification of adsorption and desorption isotherms (FIG. 4-2, FIG. 4-3)

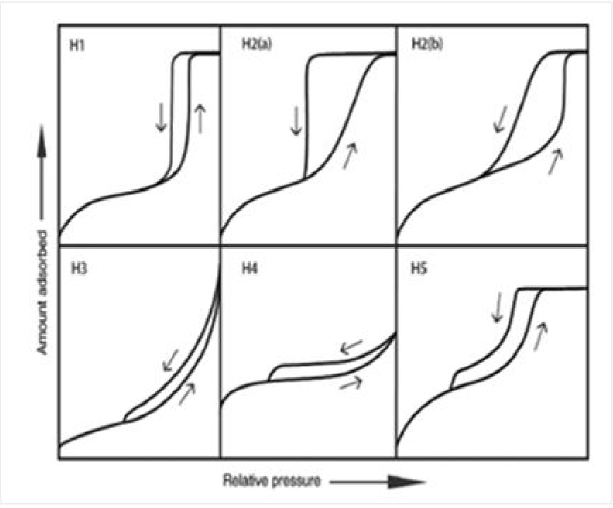


注：B 点为单层吸附容量结束的标记。

graph 4-2

Figure B presents the classification of physical adsorption isotherms proposed by the International Union of Pure and Applied Chemistry (IUPAC), which consists of six types. In 2015, the IUPAC, an authoritative body in the international chemistry community, published new guidelines for physical adsorption analysis. Over the past three decades, with the continuous synthesis of new materials such as various ordered mesoporous molecular sieves, microporous molecular sieves, and metal-organic frameworks (MOFs), the existing guidelines have become inadequate to meet current research requirements. The new guidelines add two subcategories to





the original six types of adsorption isotherms, bringing the total to eight types, thus refining the classification of microporous and mesoporous materials; the types of desorption hysteresis loops have also been expanded

graph 4-3

The characteristic of the I-type isothermal line is that there is a rapid increase in gas adsorption in the low relative pressure region. This is due to the 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. At saturation pressure, coagulation of the adsorbate may occur. Microporous solids with relatively small external surfaces, such as activated carbon, molecular sieve zeolites, and certain porous oxides, exhibit this isothermal line.

Type II isotherms are generally produced by non-porous or macroscopic solids. Point B is usually used as a marker for the end of single-layer adsorption capacity.

Type III isotherms are characterized by a protrusion of the relative pressure axis. This type of isotherm occurs when weak gas-solid interactions occur on non-porous or macroscopic solids and is not common.

Type IV isotherms are produced by mesoporous solids. A typical feature is that the adsorption branch of the isotherm does not match the desorption branch of the isotherm, and hysteresis loops can be observed. A plateau can be observed in regions with higher P/P0 values, sometimes ending with a final upward turn of the isotherm.

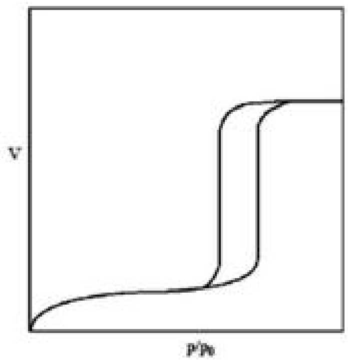
The characteristic of the V-shaped isotherm is that it bulges toward the relative pressure axis. Unlike the III-shaped isotherm, there is an inflection point at higher relative pressures. The V-shaped isotherm originates from weak gas-solid interactions on microporous and mesoporous solids, and this type of line is common in water vapor adsorption on microporous materials.

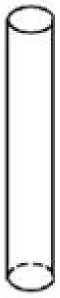
VI-type isotherms are known for their step-like characteristics of the adsorption process. These steps are derived from sequential multilayer adsorption on a uniform non-porous surface. Nitrogen adsorption at liquid nitrogen temperature does not yield the complete form of this isotherm, whereas argon adsorption at liquid argon temperature can be achieved.

The above six common adsorption graphs can be used to identify different materials by observing the adsorption amount and rate at various relative pressures. When conducting pore analysis, the adsorption curve and desorption curve do not coincide, so the isotherm for adsorption and desorption forms a ring. This means that during adsorption, multilayer adsorption occurs first, and only when the adsorption layer on the pore wall reaches a sufficient thickness does coagulation occur. Adsorption involves both multilayer adsorption on the pore wall and coagulation within the pores, while desorption is solely caused by capillary condensation (see Figure D). When desorption occurs at the same P/P0 pressure as adsorption, only the vapor above the liquid surface in the capillary condenses, which cannot desorb molecules adsorbed at P/P0. To desorb these molecules, a smaller P/P0 is required, leading to a lag in desorption. This is actually due to the irreversibility of adsorption at the same P/P0.

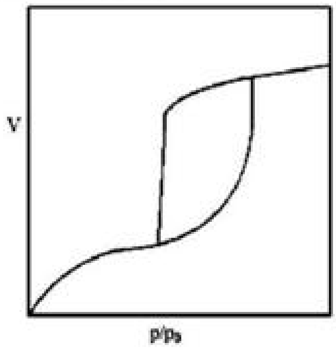
4.2.2 The type of material pore is determined from the lagging ring of nitrogen adsorption isotherm

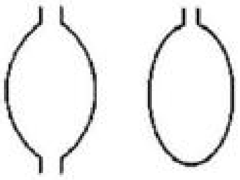
4.2.2.1 The hysteresis ring corresponding to the tubular hole structure at both ends



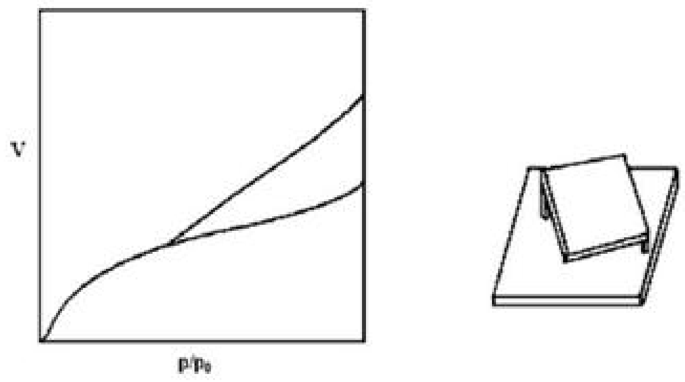


4.2.2.2 The hysteresis loop corresponding to the "ink bottle" hole





4.2.2.3 The four-sided open inclined plate overlapping slit structure corresponds to the backflow ring



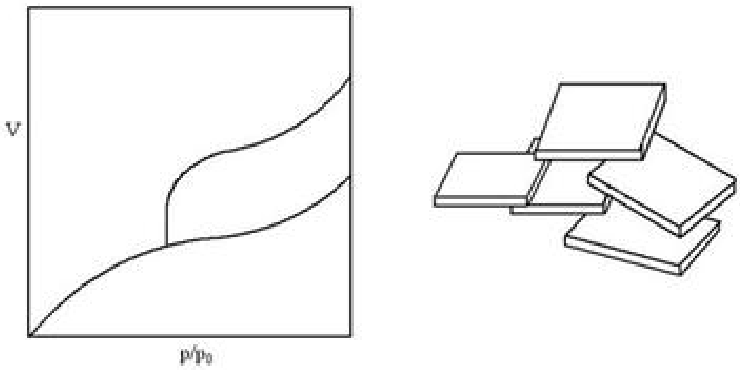
4.2.2.4 The structure of the flat slit hole corresponds to the stagnation ring (it is difficult to form a concave liquid surface when adsorbed)

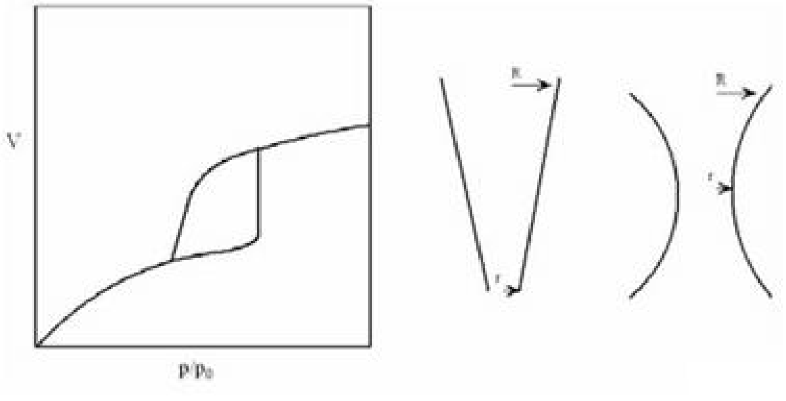
4.2.2.5 Retardation rings corresponding to conical or double-cone tubular pore structures (evaporated gradually during desorption)

4.2.3 Specific surface area and the range of different pore size samples

4.2.3.1 Relative pressure sampling point

The BET formula is used to calculate the specific surface area. For microporous materials, the BET line relationship obtained at relative pressures of 0.01-0.1 is better than that at 0.05-0.2.





For mesoporous materials, the BET results obtained at 0.05-0.2 are reasonable. Most microporous materials have a specific surface area smaller than 0.01-0.1 when calculated at relative pressures of 0.05-0.2, and the higher the content of microporous materials in the catalyst, the greater the difference in calculated values within these two relative pressure ranges.

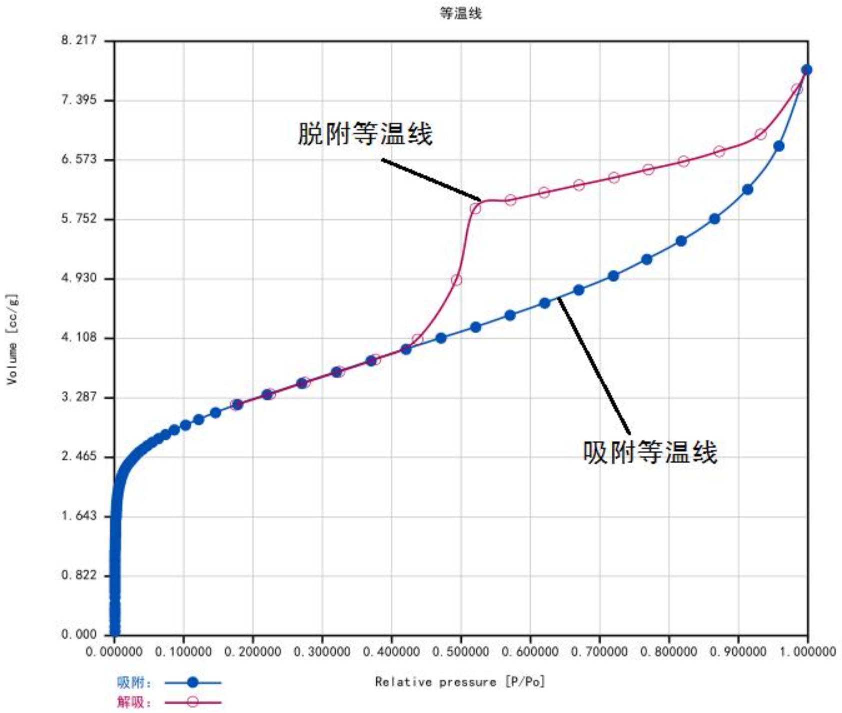
The BET surface area calculation for microporous materials requires selecting the correct relative pressure range; the C constant is a simple criterion for judgment. Any negative intercept on the BET plot indicates that the value exceeds the effective application range of the BET equation. The classic BET range for microporous materials is 0.05-0.30, where linear data cannot be obtained, and a negative C constant has no physical significance. When choosing the linear range for the BET plot of microporous materials, any subjectivity should be avoided. The correct process is: the C constant must not only be positive but also the adsorption amount should continue to increase with the relative pressure.

4.2.3.2 Do not use too low pressure point in BET equation

When some data curves back to the origin, these points cannot be used for calculating specific surface area. This is because pressure points that are too low are insufficient to form a monolayer. When the C value is very small, it results in a very high intercept. In such cases, BET plots often show a significant curvature above the conventional lower limit of 0.05, indicating that the BET pressure curve has been compressed. Data points with a clear bend below 0.3 should be removed.

4.2.3.3 BET Equation Do not choose a relatively high pressure point

Incorrect sampling points can lead to poor correlation coefficients and negative intercepts in linear regression, meaning the C constant is negative. The upper limit for BET sampling can be determined by calculating the maximum single-point BET value. However, this is not always the case for all samples. Some samples do not show a single-point BET peak but increase with pressure, indicating that there will be no short linear region below relative pressure 0.15. In such cases, the BET equation is not applicable to these samples.



graph 4-4

(3) Physical adsorption theory

The theory of gas adsorption mainly includes the Langmuir monolayer adsorption theory, the Porani adsorption potential energy theory, the BET multilayer adsorption theory (see multilayer adsorption), the two-dimensional adsorption film theory, and the polarization theory. The first three theories are the most widely applied. These adsorption theories all start from different physical models, comprehensively examine a large number of experimental results, undergo certain mathematical processing, and explain the limiting parts of the isotherms for one (or several) types of adsorption, providing equations to describe the isotherms. The most common method for studying solid-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 using different adsorption theories and models.

Coal adsorption of methane mostly conforms to the I-type isotherm. Using the langmuir equation to describe the coal adsorption process often yields satisfactory results, making it one of the most frequently used models in the coalbed methane field. The reasons are as follows: first, the langmuir equation has only two parameters, which makes it easy to convert into a linear equation for solving, with a simple form that is convenient to use. Second, the two parameters of the langmuir equation have clear physical significance. Third, the langmuir equation has been validated through extensive engineering practice and is considered sufficient to meet engineering needs.

4.3.1 Material specific surface area analysis

4.3.1.1 Characterization of specific surface area

In 1938, Brumauer, Emmett, and Teller discovered the relationship between the actual adsorption capacity V and the monolayer saturated adsorption capacity Vm through thermodynamic and kinetic analysis of gas adsorption processes. They derived the famous BET equation, which is applicable to specific surface area measurements under multi-layer theory. Currently, this method is used as the standard for specific surface area testing of most materials.

In the characterization of specific surface area of the system, there are also some materials that conform to the characteristics of single layer adsorption. Laugmuir also deduced the corresponding specific surface area test formula based on the assumption of single layer adsorption, which is used to characterize the materials with single layer adsorption characteristics. This specific surface area is also called Laugmuir specific surface area.

In addition, in some fields, the internal surface area of micropores in materials does not play a role, so the concept of external specific surface area is put forward again. The test model of external specific surface area adopts t-plot method.

4.3.1.2 Basic assumptions of Langmuir single layer adsorption theory

(i) there are a certain number of adsorption sites on the surface of the solid adsorbent, each of which can only adsorb one molecule or atom; (ii) all adsorption sites on the surface have the same adsorption capacity, that is to say, the adsorption heat on all adsorption sites is equal;

(Iii) There is no interaction between adsorbed molecules.

The following results are obtained:

$$Pa/V=1/Vmb+Pa/Vm$$

Pa—— The gas phase pressure of adsorption equilibrium

Vm—— Saturated adsorption capacity of monolayer

V—— The volume of adsorbed gas

b—— Total adsorption constant

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

4.3.1.3 Multilayer adsorption theory

1.BET algorithm

On the basis of the single-molecule adsorption model of Langmuir theory, BET theory is extended to the case of multi-layer adsorption based on the following three assumptions:

(i) Gas molecules can be adsorbed on a solid in countless layers

(ii) There is no interaction between the layers of adsorption

(iii) Langmuir adsorption theory holds for each monolayer. Thus the BET adsorption isotherm is obtained as follows:

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

2. T-chart method

T-plot, also known as t-curve, is an isotherm represented by t replacing the relative pressure P/P0 as the horizontal coordinate and adsorption amount as the vertical coordinate,

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

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

The thickness of the single layer of tm—— is t——, the thickness of the adsorption layer is V——, and the adsorption amount of the measured sample is Vm——

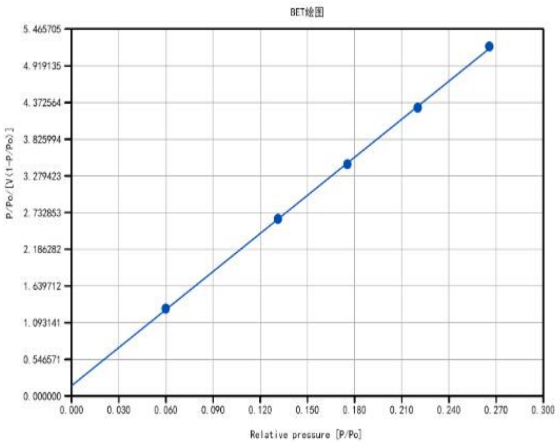
Tm——77.4K The thickness of the adsorbate (nitrogen molecule) at temperature, assuming that the nitrogen molecules in the adsorption film are arranged in a hexagonal close packing, the thickness of the single layer of nitrogen is given, which is 0.354nm.

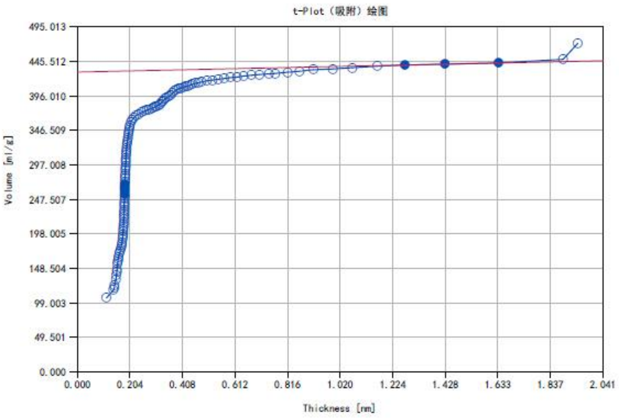
* The premise of the application of t-plot method:

(I) The samples have a smooth surface in the mesoporous range

(ii) The range of relative pressures for micro-pore filling and capillary condensation does not overlap.

(Iii) The straight part of the comparison graph corresponds to (non-pore sample) multi-layer adsorption, and the upward deviation from the line is caused by the pores contained in the sample. The latter can evaluate the pore volume of a selected range of pore size, and the specific surface area of the straight part, which is the unfilled pore part, can be calculated by the slope of the straight part.





BET比表面积[m2/g]: 0.2289
斜率: 18.892799
截距: 0.155440
相关系数: 0.999892

P/Po	P/Po/[V(1-P/Po)]
0.059970	1.302724
0.131412	2.637540
0.175476	3.450297
0.220162	4.292657
0.265763	5.205434

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

4.3.2 Characterization of porosity

4.3.2.1 The concept of porosity

Porosity (Porosity) is a special term for the characterization of surface properties of powders and particles: including total pore volume, pore size distribution, average pore size and so on.

4.3.2.2 Characterization of porosity

According to the surface characteristics of the material, the characterization of porosity can be divided into mesopore, macropore and micropore.

4.3.2.2.1 Characterization of mesopores and macropores

① Test methods for mesopores and macropores

The BJH (Barrett-Joyner-Halenda) method is used for the determination and analysis of mesopores and macropores. The main theoretical basis for BJH pore size analysis 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 gas adsorption and desorption isotherm, BJH model can calculate pore volume, pore size distribution, total pore volume and average pore size successively.

② The characterization range of mesopores and macropores

Currently, the control range of nitrogen partial pressure in all nitrogen adsorption instruments is almost very wide: the minimum value is close to 0, and the maximum value is close to 1. The lower limit of mesopores, at 2 nm, corresponds to a nitrogen partial pressure of 0.14; when the pressure is 0.996, the pore diameter can reach 500 nm. Therefore, the pore size analysis range conducted by the BJH method includes mesopores and some macropores, and it is generally believed that the upper limit for pore measurement using nitrogen adsorption methods is 500 nm.

4.3.2.2.2 Characterization of total pore volume

① Total pore volume of adsorption:

The total pore volume calculated by taking the adsorption amount at the highest relative pressure of nitrogen as being fully absorbed and filled in the pores is called the total pore volume of adsorption.

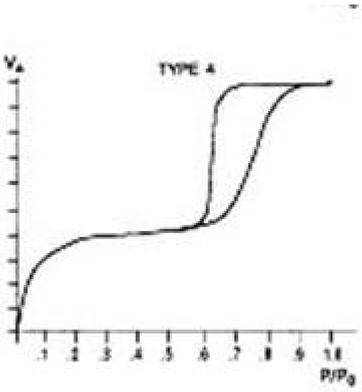
Capillary condensation theory and Kelvin equation

Capillary condensation refers to the phenomenon where, in capillary pores, if adsorption is required to form a concave liquid surface, the equilibrium vapor pressure P must be lower than the saturated vapor pressure P0 of a flat liquid surface at the same temperature. The smaller the diameter of the capillary pore, the smaller the radius of curvature of the concave liquid surface, and the lower the equilibrium vapor pressure. In other words, 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 is desorbed first, and as the pressure continues to decrease, the condensed liquid in smaller pores is also desorbed gradually.

Interpretation of 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 monolayer of adsorption, corresponding to segment AB in the figure. When the monolayer adsorption approaches saturation, multilayer adsorption begins. When the relative pressure reaches a specific value corresponding to the radius Kelvin of capillary condensation, capillary condensation starts. Capillary condensation continues until the mesopores are filled, reaching saturation. The desorption process is the reverse: it begins with capillary condensation within the capillary, followed by multilayer desorption, and finally monolayer desorption.

Assuming the capillary pores are cylindrical, divide all micropores into several zones based on their diameter, and arrange these zones in order of size. Pores of different diameters produce varying capillary condensation pressure conditions. During desorption, as the pressure decreases from its maximum value, the condensed liquid first gradually desorbs from larger pores and then from smaller ones. Clearly, there is a certain correspondence between pore size and the pressure of the adsorbate that can lead to condensation or desorption from the condensed state, as given by Equation kelvin. As shown in the figure:



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

It can be derived that: Kelvin

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

However, the pore size when capillary condensation occurs is not the radius of curvature.

Kelvin Equation:

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

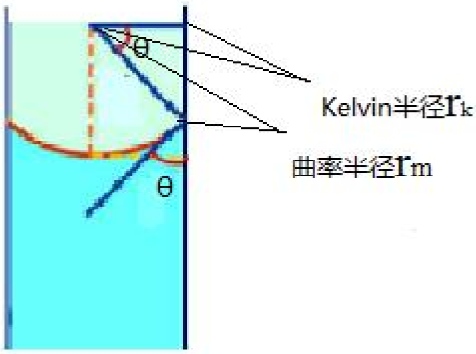
As shown in the figure, the relationship between the average radius of curvature and Kelvin radius is as follows: $r_k = \cos\theta r_m$

②Kelvin

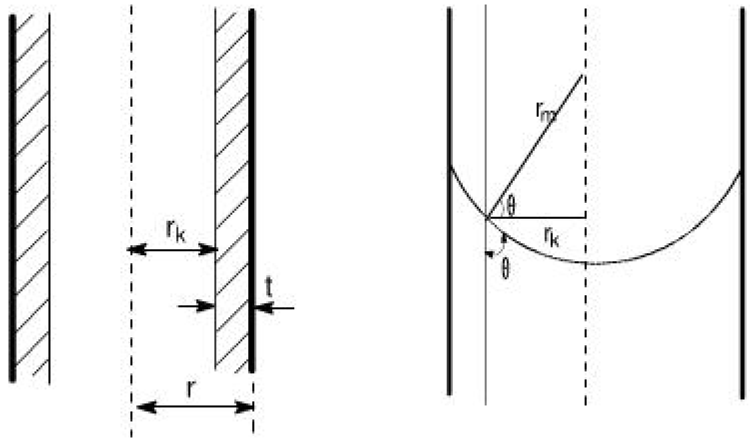
The equation provides the relationship between pore size and relative pressure when capillary condensation occurs. In fact, during Kelvin condensation, multiple layers of adsorption have already occurred on the pore walls, meaning that the adsorption film forms within the pore core it surrounds. According to the t-plot method, the film thickness is related to P/P0. When the thickness of the adsorption film increases to a certain level, capillary condensation will occur within the adsorption film. The following relationship holds:

③ r=rk+t,

T is the thickness of the membrane and r is the radius of the hole.



The relationship between the radius of curvature and



The above ①②③ equation is proposed as follows:

$$r = \frac{2\sigma V_L}{RT\ln(P/P_0)} + t(P/P_0) + 0.3$$

Among them, r is the pore radius; t is the thickness of the statistical film adsorbed on the pore wall, which can be obtained from the t curve.

The BJH method was used to determine the pore size distribution

The Kelvin equation can effectively explain the IV-type isotherm, which represents the general isotherm for adsorption in mesoporous materials. Using the Kelvin equation allows us to calculate the pore radius at any point on the isotherm. According to the capillary condensation theory, all pores with radii less than a certain size will be filled under the corresponding adsorption pressure at that size. 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 derive the pore size distribution.

It is a common method to calculate pore size distribution using Kelvin equation in nitrogen isothermic counties. The BJH method proposed by Barrett, joyner and halenda has been widely used at present.

The basic assumptions of this approach are:

All the pores are non-intersecting cylindrical pores.

The contact angle of the semi-spherical curved liquid surface is zero or completely wet.

iii Apply the simple Kelvin equation.

To calculate the pore size distribution, pores must be grouped according to their size. Using the formulas mentioned above, a series of r and t values at different relative pressures P/P0 are derived. Then, based on the basic formula for pore size distribution in BJH, the pore volume ΔV for each desorption stage with various pore sizes is calculated. Plotting $\Delta V/\Delta r$ against the average pore radius r yields the pore size distribution curve. The Kelvin equation does not account for the superposition effect of adsorption potentials in micropores, making it unsuitable for evaluating microporous carbonaceous adsorbents; it is only appropriate for mesoporous materials. As pore size decreases, its accuracy diminishes, and when the micropore size equals the diameter of an adsorbate gas molecule, the Kelvin equation becomes entirely inapplicable.

Using the BJH method, the total pore volume is obtained by accumulating the pore volumes of different pore sizes calculated step by step from isothermal adsorption or desorption processes. It has clear upper and lower limits for pore size, with the lower limit generally being 2nm and the upper limit ranging from 200 to 400 nm. When comparing various data, attention should be paid to the differences in pore size ranges. The measurement range of pore size 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 several dozen nm in diameter. In fact, the "large pores" measured are gaps between particles, which incorrectly characterizes the surface and surface properties of the powder. Theoretically, capillary condensation occurs when $P/P0 \geq 0.4$, but the lower limit of pore size analysis using the BJH method has always been far below 0.4, and there has yet to be an appropriate explanation for this discrepancy.

There are three different ways to express the average aperture:

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

(2) BJH adsorption average pore size: the average pore size calculated by BJH adsorption cumulative total pore volume and BJH adsorption cumulative total pore surface area, with upper and lower limits of pore size.

(3) BJH desorption average pore size: the average pore size calculated by BJH desorption

cumulative total pore volume and BJH desorption cumulative total pore surface area, with upper and lower limits of pore size.

Extending the BJH pore size analysis method to micropores is incorrect for two reasons: First, the Kelvin equation does not apply when pore sizes are <2 nm; Second, in capillary condensation, the adsorbate in pores is liquid, whereas in micropores, due to the interaction between densely packed pore walls, the adsorbate becomes non-liquid. Therefore, new theories and computational methods are required for the pore size distribution, as macroscopic thermodynamic approaches are far from sufficient.

Analysis and theory of microporous pore structure

Introduction to Polanyi adsorption potential theory

The potential energy theory of Polanyi points out that there is a potential energy field near the surface where adsorption occurs, and the potential energy is greater as it approaches the surface. When a molecule is adsorbed, it enters the zero potential energy surface and is captured on a certain potential energy surface between the surface and the zero potential energy surface.

According to the potential energy theory, the volume V of adsorption at 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 near the surface; near the critical temperature, the adsorbate is considered a liquid near the surface but becomes a gas farther from the surface; if the adsorption temperature is much lower than the critical temperature of the adsorbate, the adsorbate is assumed to be entirely in a liquid state, and the adsorption volume can be expressed as

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

Among them, W is the mass of adsorption (g), and ρ is the density of adsorbate liquid (g/cm3).

According to the potential energy theory, when the adsorbate is in 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 required to compress the steam at equilibrium pressure p into the liquid in the adsorption region under the saturated vapor pressure p0 under isothermal conditions.

For any adsorption system, the characteristic curve is unique. Therefore, by measuring the isotherm at one temperature and constructing the characteristic curve, the isotherms at any other temperatures can be obtained. This is precisely the advantage of adsorption potential theory. Activated carbon is a typical nonpolar adsorbent, and its interactions mainly rely on dispersive forces. Thus, the adsorption potential theory has been very successful in explaining activated carbon adsorption systems.

Microporous filling theory and DR equation

Most adsorbents used in industry are microporous. The adsorption behavior of gases in micropores differs from that in mesopores and macropores. The theory of physical adsorption in micropores is based on the polanyi adsorption potential theory, which was later introduced to microporous adsorption studies by dubinin, leading to the establishment of the microporous packing theory. This theory is also known as the 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. The 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 proposed a more general equation, namely DA equation

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

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

approach

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

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

The value of n can be calculated according to the following process:

(1) V0 can be obtained from the plateau of the adsorption isotherm, and V/V0 can be known.

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

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

The above θ-micropore filling rate; V-atmospheric pressure adsorption volume, i.e., the volume of micropores already filled; V0-saturated adsorption volume, i.e., the total volume of micropores; A-Gibbs free adsorption energy; A=-RTln(P/P0); E-characteristic adsorption energy, E=βE0. Here, 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 to characterize the structure of microporous materials and calculate the pore size distribution. It was originally based on the slit pores of carbon molecular sieves and activated carbon, and the adsorption potential of slit pores was expressed as a function of pore size, and then the adsorption amount was calculated by using the adsorption isotherm line, and the pore distribution was calculated.

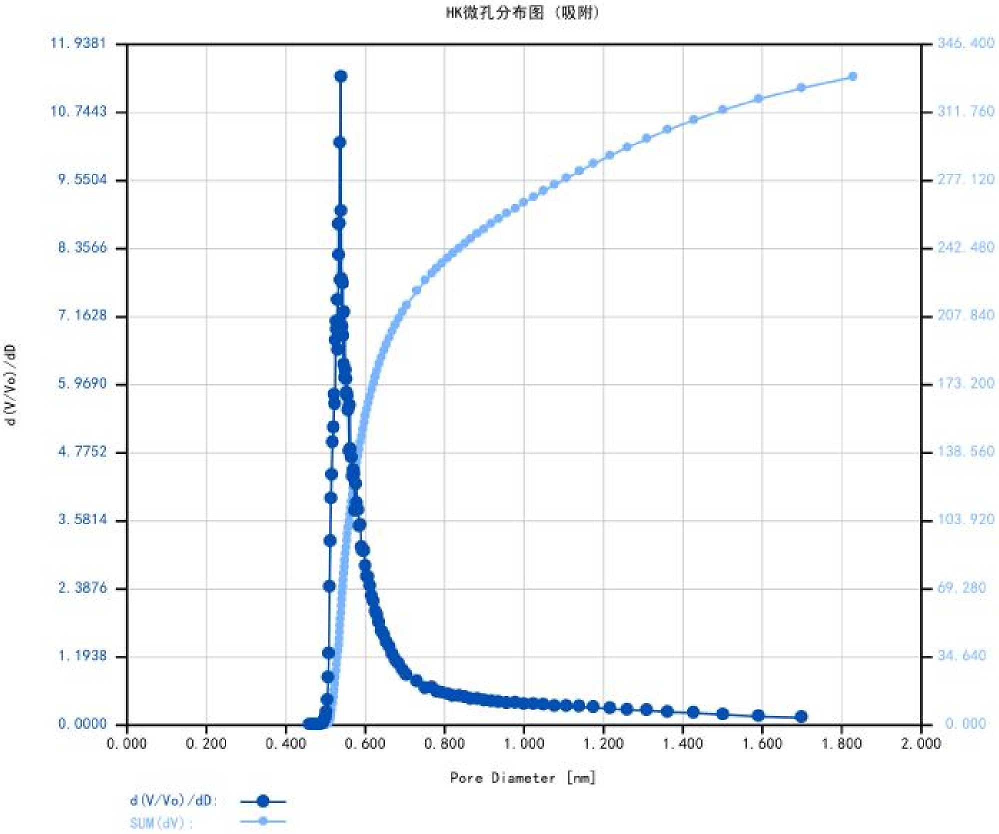
The main basic framework of the HK model is the same, described by the following equation:

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

Among them: U0--adsorbent interaction energy

P0-adsorbent and pore wall interaction energy

In the case of micropores, the interaction potential energy between pore walls overlaps, and the adsorption in micropores is greater than in mesopores. Therefore, filling occurs in micropores when the relative pressure is <0.01. Pores with diameters between 0.5 to 1 nm can even be filled with adsorbates at relative pressures of 10^-5 to 10^-7, making the measurement and analysis of micropores much more complex than that of mesopores.



HK and SF proposed a semi-empirical analytical method for calculating the effective pore size distribution using isotherms of microporous samples. This method was applied to nitrogen/carbon (slit) and argon/zeolite (cylindrical pore) systems, treating the packing fluid as a loose fluid. A series of parameters related to the adsorbent and adsorbate must be introduced in these calculations, and the selection of these parameters significantly affects the results. The method introduces a new relationship between micropore diameter and packing pressure but does not address the density model of nitrogen molecules in micropores. Therefore, it can represent the adsorption rules and pore size distribution patterns of micropores, but it still lacks quantitative analytical significance.

According to the t-chart method proposed by Lippens and deBoer, which is widely used in micropore analysis, the adsorption capacity is defined as a function of the statistical layer thickness t, which is calculated using standard isotherms. The t-chart can be used to calculate the specific surface area, total surface area, and total volume of micropores within the inner surface. The T-chart method, also known as the MP method, is used for analyzing pore size distribution and can analyze the pore size distribution of micropores. However, the drawbacks of the T-chart and MP methods are that they still treat the adsorbate in the micropores as a liquid and use the Kelvin equation for pore size calculation. Therefore, the total pore volume of micropores obtained by these methods only has relative significance, and the pore size distribution range cannot represent the true micro-pore region.

In the past decade, Non-Dimensional Density Function Theory (NLDFT) and computer simulation methods (such as Monte Carlo fitting) have developed into effective approaches for describing the adsorption and phase behavior of confined non-uniform fluids in porous materials. The NLDFT method is applicable to various adsorbent/adsorbate systems. Compared with classical thermodynamics and microscopic models, the NLDFT method describes the behavior of fluids confined within pores at the molecular level. Its application can link the molecular properties of adsorbed gases with their adsorption performance in different pore sizes, making the NLDFT method suitable for characterizing pore size distributions across the entire range of micropores and mesopores. Currently, the NLDFT method is highly favored in foreign instruments and used as a standard to compare the effectiveness of other methods. However, the computational methods for different adsorbent/adsorbate systems vary, making it difficult to apply universally across different systems. New theories aim to address two main issues: first, to derive a more reasonable relationship between micropore filling pressure and pore size; second, to propose the state and density of adsorbates within micropores. There is reason to expect a simpler and more universal density function theory that would make the analysis of ultra-micropore pore size distributions more extensive and comparable.

Chapter 5 Fault Troubleshooting

- If the power is suddenly cut off or an unexpected situation occurs, causing the test to stop and cannot be continued, after the unexpected situation is eliminated, you need to open the power supply and gas source, open the program, click the "Start analysis" button under the measurement direction guide interface, run for 2-5min, click the "Terminate analysis" button, and reset the software status to complete the analysis before measuring.
- If only one sample needs to be tested, simply select "Idle" in the "Analysis Control" of each analysis port, and install the idle analysis port into a dry and clean empty tube.
- During the operation of the software, if the calibration sensor has not finished for more than 20 minutes, it may be that the cylinder valve is not opened. Please check the status of the nitrogen and helium cylinder valves. If the cylinder is not opened, please open the cylinder valve counterclockwise, click the "Terminate analysis" button, wait for the software to run out, and then start testing samples again.
- During the operation of the software, if the vacuum is extracted for more than 20 minutes and it is not finished, it may be that some connection parts are not connected well, resulting in the possibility of gas leakage. At this time, we need to check all the connection parts; for example: the connection of sample tube, the connection of gas cylinder and instrument gas path, etc.
- During the test, liquid nitrogen should be added to the designated height of the Dewar bottle after the first and second step analysis.
- If there are other matters that have not been completed or the fault cannot be determined, please contact us in time. We will try our best to eliminate it for you.

Chapter VI Instrument accessories

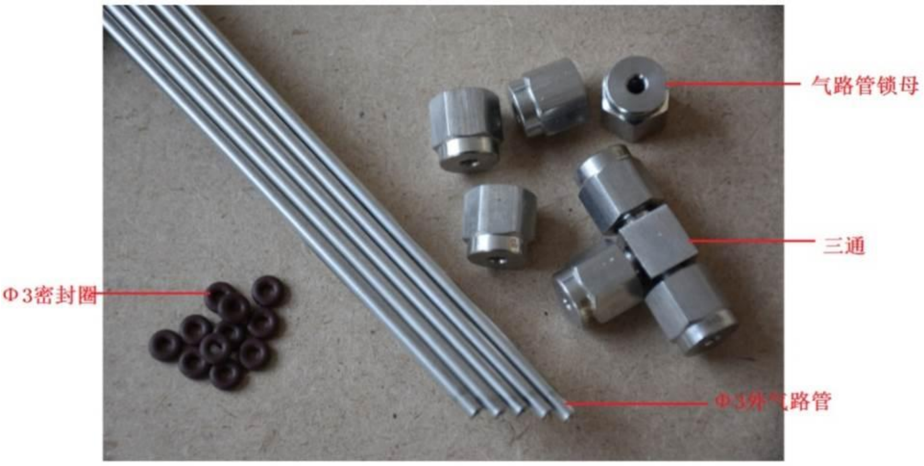
(1) Annex table

(Subject to actual supply)

order	Title of annex	specifications	remarks
1	Two-stage rotary vane vacuum	Take random goods as	
2	nitrogen canister	Take random goods as	Liquid nitrogen is
3	High purity helium	Take random goods as	User provided
4	High purity nitrogen	Take random goods as	User provided
5	Dewar flask	Take random goods as	
6	Pressure reducer (including	Take random goods as	User provided
7	Tracheal lock nut	M8*3	
8	External air line	Φ3, stainless steel	
9	sample cell	Take random goods as	
10	Sample tube filter plug	Φ10	
11	Sample funnel	Take random goods as	
12	filler rod	200mm	
13	standard	Take random goods as	
14	P0 tube	Take random goods as	
15	P0 pipe seal ring	5x2.5mm	
16	Spherical tube seal	8x2mm	
17	O-ring	2.2x1.9mm	
18	Sample tube holder	20mm	
19	convenienve receptacle	S1-12	
20	Power cable for the host	1.5m	
21	sylphon bellows	KF16(150cm)	
22	KF16 adapter	KF16-3	
23	built-for purpose tools	Take random goods as	
24	U tray	Take random goods as	
25	Sample tube stopper	Φ10	
26	data wire	1.8 meters	
27	software	Take random goods as	
28	certificate	Take random goods as	
29	manual of operation	Take random goods as	

(2) Introduction of annexes

6.2.1. External air pipe sealing accessories, as shown in Figure 6-1

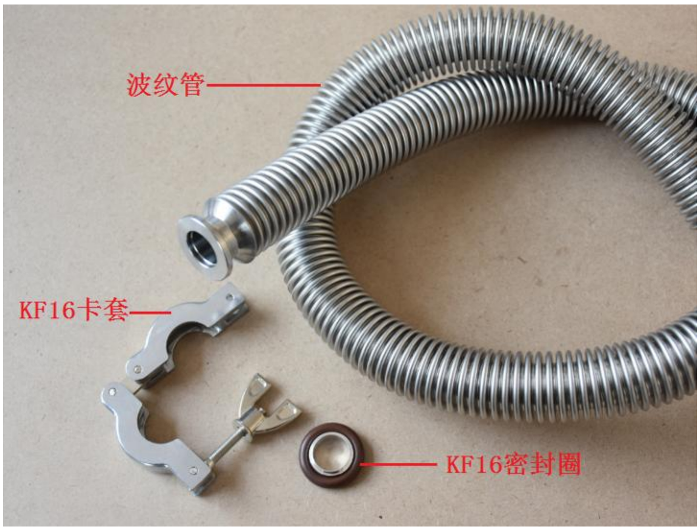


graph 6-1

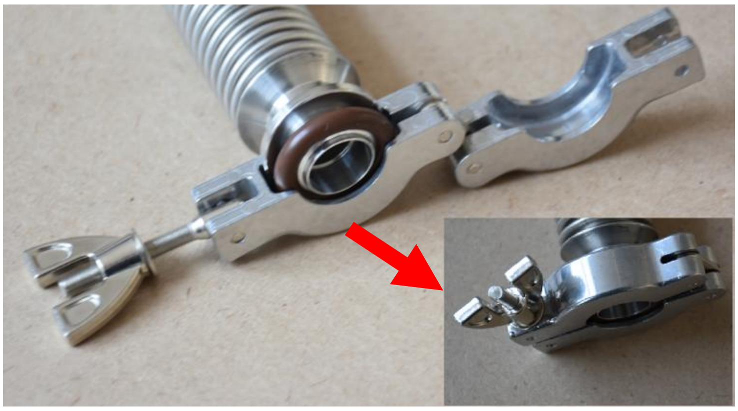
6.2.2 Vacuum pump accessories

1. Double click the rotary vane mechanical pump 2 (Figure 6-2)
2. See Figure 6-3 for the sealing parts, and figure 6-4 for the sealing method

graph 6-2

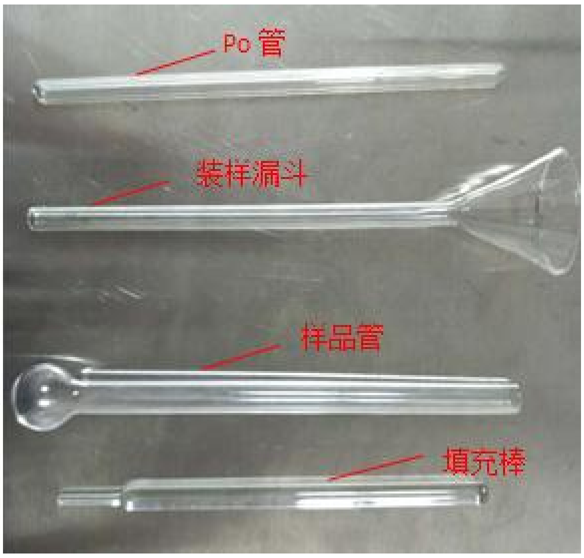


graph 6-3



graph 6-4

6.2.3 Sample tube and Po tube accessories, as shown in Figure 6-5, sealing accessories Figure 6-6 and Figure 6-7



graph 6-5

Figure 6-6 Sample tube seal Figure 6-7 Po tube seal

6.2.4. Liquid nitrogen container accessories, mainly include Dewar bottle (Figure 6-8) and liquid nitrogen tank (Figure 6-9)

graph 6-8

graph 6-9

Chapter VII After-sales Service Commitment

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

technical service

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

The reason why Poid Company has confidence and determination to make achievements in the inspection and analysis instrument manufacturing industry in China is that we have "excellent talents", "rich experience", "advanced technology" and "unified command organization system". We will organize the most excellent and experienced technical personnel of the company to provide you with the best technical services.

Technical services content:

Pheid Electronic Technology Co., Ltd. is willing to provide users with lifetime technical consulting services, including: notification of new products and technologies, hardware and software technical consulting, providing technical solutions and researching and solving technical problems.

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

product service

Hardware warranty service:

The technical support center of Peode Company provides 5 days and 8 hours of service, and can provide 7 days and 24 hours of service under special circumstances. During the warranty period, we will provide users with comprehensive troubleshooting technical services and monitoring of the whole troubleshooting process (from the start of the fault to the complete troubleshooting).

When the equipment fails and cannot work, the user needs to inform the company of the fault by sending a true form and explain the cause and condition of the fault as much as possible. The company will solve the problem in the following ways.

General faults:

General faults refer to the failure or improper configuration of non-important equipment, which affects individual functions or makes it inconvenient to operate. For such faults, we first use telephone support to guide the on-site solution until the problem is resolved.

Serious faults:

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

Urgent faults:

An emergency failure is when the equipment is completely disabled and unable to do any work. For emergency failures, we will promise to provide both telephone support and on-site solutions to ensure that the problem is solved in a timely and effective manner.

To better serve our users, please provide us with the product serial number when a device malfunctions. We will document any issues that arise to facilitate analysis and reduce the likelihood of similar incidents. The response time for faults is within 12 hours of receiving a written notification from the customer. We will respond within this timeframe to address the issue promptly, minimizing any losses caused by the malfunction.

Software upgrades

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

technically training

To enable the technical personnel on the client side to proficiently operate and maintain the equipment, ensuring the smooth operation of the entire system, understand new directions in technology development, and enhance cooperation between both parties. After the cooperation agreement takes effect, Poider Company will dispatch specialists to arrange a series of training sessions based on user needs, covering specific technical and instrumental knowledge. This is aimed at helping the client's technical staff master the usage methods of the instruments as quickly as possible, so that Poider's equipment can be utilized to its fullest potential in your company.

Chapter VIII. Appendix

(1) Operation process

1. Sampling and weighing

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

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

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 is less than 0.01

(3) Sample loading:

A) Insert the rubber stopper into the air pipe opening, weigh the mass of the empty pipe and the rubber stopper, record it as M1, and then peel off the skin.

Note: If multiple samples are weighed at one time, it is necessary to ensure that the empty tube and rubber stopper correspond to each other. You can choose to mark the empty tube and rubber stopper.

B) Remove the rubber stopper, put it into the funnel, put the sample into the empty tube and plug it with the corresponding rubber stopper, weigh the sample and record it as M2.

2. Sample degassing and drying preparation

(1) Experimental equipment: sample preprocessor, sample filter plug, Φ8 sealing ring.

(2) Sample tube loading into the processor: remove the rubber stopper for later use. Load each sample tube with one nut, two Φ8 sealing rings and one filter plug in turn, connect it to the processor interface and tighten it, and place the sample tube on the heating position. The remaining processing position of the processor is connected to an empty tube.

(3) Setting conditions: input the temperature and time to be processed, and then click start. After

the processing is completed, the processor will automatically alarm and prompt you to take the sample tube out to the cooling position and cool it to room temperature.

(4) Processing is complete: after the sample tube is cooled to room temperature, click the "inject gas or stop" button to fill nitrogen protective gas.

(5) Secondary weighing: take the sample tube after treatment, remove the filter plug and insert the corresponding rubber plug to weigh, and record it as M3. The mass of the sample after treatment is M = M3-M1.

3. On-machine testing

(1) Experimental equipment: Kubo-1108 analysis host, liquid nitrogen, high purity nitrogen and helium, vacuum pump, Dewar bottle, Φ8 sealing ring, filter plug, filling column.

(2) Start the gas: open the valve of helium cylinder and nitrogen cylinder, adjust the output pressure to 0.3MPa, and start the vacuum pump and host power supply.

(3) Sample tube machine: remove the rubber plug, and install 1 nut, 2 Φ8 sealing rings and 1 filter plug into each sample tube in turn. The sealing ring is 1-2cm 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 bit, input the sample quality and name, and set the file name of the generated report; select the analysis mode, and select idle for the remaining unmeasured samples. Click OK after setting, close the window, and return to the main interface.

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

(5) Start testing: When the start test button turns green, it indicates that all analysis bits have been prepared. Click "Start Test" at this time.

(6) Add liquid nitrogen: Fill the Dewar bottle with liquid nitrogen, and the liquid surface of liquid nitrogen is 1-2cm away from the mouth of the Dewar bottle (it is necessary to ensure that the liquid surface of liquid nitrogen is fixed every time). Then put the Dewar bottle on the lifting tray of the analysis host.

(2) Operation precautions

1. Sample degassing and drying treatment

(1) When sampling and weighing, the sample tube and stopper should correspond to each other and should not be confused.

(2) If multiple samples are processed at the same time, the sample tube and stopper should be marked for easy weighing and sample recovery.

Clean the instrument housing	once a week
Replace the sample tube filter plug	Every three months
Clean the Dewar flask	once a week
Replace the sample tube seal	Every three months
Check and replace vacuum pump oil	Every three months
Run the instrument to detect leakage and check the instrument	Every year

(3) It takes a certain amount of time for the processor to heat up, so the effect of heating time should be taken into account when setting the processing temperature.

(4) The remaining processing bits on the processor must be connected to an empty sample tube.

(5) The treated samples must be cooled to room temperature before nitrogen protection gas can be injected.

2. On-machine testing

(1) The sample tube to be measured should be filled with a column.

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

(3) If the analysis position is idle, it must be filled with an empty sample tube.

(4) When setting the file name of the current sample, it cannot be duplicated with the previous file name, otherwise the data of the previous file will be overwritten.

(5) When the specific surface area of the test sample is very small, the decreasing pressure can be set to 0.5~8KPa.

(3) Maintenance guidance

1. Clean the instrument housing once a week

Wipe the instrument shell with a clean cloth dipped in isopropanol or water. Avoid cleaning liquid entering the instrument shell.

2. Replace the sample tube filter plug every 3 months

If the filter plug used in the sample tube is contaminated, it will be adsorbed and desorbed during the analysis process, thus affecting the analysis results. The filter plug 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 of the Dewar bottle. Rinse the interior of the Dewar bottle with clean 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, so as not to affect the vacuum seal of the sample tube.

5. Check and replace vacuum pump oil every 3 months

Observe the oil level in the tank to ensure it is normal (the oil should be at the center). Check if the oil color is clear and transparent (the oil should be clear and transparent). If not, drain the oil from the pump's outlet and refill it to the normal oil level. The pump's outlet is at the bottom, so it's best to place the pump flat on a table or other elevated surface. Use a container to catch the drained waste oil. See the diagram below for the pump's inlet and outlet.

6. Run the instrument to check the leak every 1 year

The instrument leakage and calibration are carried out every other year with the standard sample to check the instrument performance.

(4) Format of experimental data record form

Kubo series BET experimental data record sheet

Sample sending unit: (Ser:) date:

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

Sample sending unit: (Ser:) date:

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

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