SSA-4000

Specific surface area and Pore size analyzer

Operation manual

Dear user

Firstly, thank you for choosing our company's products. Our independently developed, generated, and sold products mainly include:

1. Specific surface area and pore size analyzer series
2. Programmed temperature rise chemical adsorption instrument series
3. Gas method true density instrument series
4. Capacity based steam adsorption instrument series
5. Capacity based gas adsorption instrument series
6. Capacity based high-pressure adsorption instrument series
7. Bubble point method membrane pore size analyzer series
8. Laboratory gas distribution instrument series (providing intake solutions for infrared/mass spectrometry, etc.)
9. Customized series of small-scale catalytic evaluation devices (including VOCs generation and treatment series, SCR denitrification and desulfurization series, etc.)
10. Online mass spectrometer series

was established on January 9, 2003, specializing in the technical research and development of material characterization instruments. It is a technical service enterprise that integrates project research and development, product generation, and testing consulting. Over the course of more than 20 years of development, the company has accumulated a group of high-quality R&D and application technology talents. With strong technical support and personnel infrastructure, Biotech is able to independently complete the generation of products and the research and manufacturing of core components. It can provide technical development services beyond the main product scope for scientific research units and enterprises at all levels.

CAUTIONS

This operation manual describes all the functions that Specific surface area analyzer has. Your product may not have all the features in this manual. Please refer to the actual goods for accuracy.

Any violation of its correct use or clearly defined as foreseeable misuse is not covered by the warranty.

1. This manual involves the analysis software version number StencilWizard as indicated by the label, and the firmware software version as indicated by the label. Before the first use, please read this product operation manual carefully. If you have any questions or suggestions, please call+86-400-669-898.
2. Our company will continuously improve and update the performance and variety of various instruments. Therefore, we must reserve the right to make changes to the scope of supply, configuration, and technical specifications. We hope users can understand.
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- More network construction in progress

Catalogue

Chapter 1 Installation

（1） Installation of Gas Cylinders and Pressure Reducing Valves

1. Install the Pressure Reducing Valve Adapter

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

Figure 1-1

Figure 1-2

2. Install the Pressure Reducing Valve on/off Valve

Install the black knob (i.e. the on/off valve of the pressure reducing valve) on the pressure reducing valve, as shown in Figure 1-3

Figure 1-3

（2） Installation of Vacuum Pump

1. Installation and Use of Main Vacuum Pump

The SSA-4000 specific surface area and aperture analyzer comes standard with two two-stage rotary vane mechanical pumps (Figure 1-4). Be careful not to mix and install the suction port and exhaust port incorrectly, otherwise it will cause the pump body to explode and cannot be repaired or cause more serious safety accidents. The pneumatic vibration of the pump should always be in the factory state and should not be touched.

Figure 1-4

(1) Power Cord Connection

Insert the other section of the power cord on the mechanical pump into the 220V power interface, as shown in Figure 1-5

Figure 1-5

(2) Installation of KF16 Clamp

Figure 1-6

As shown in Figure 1-6, place the KF16 sealing ring around the corrugated pipe opening, then insert the clamp and tighten the screw cap. When connecting to the instrument, align the corrugated tube mouth that is inserted into the sealing ring with the instrument interface and tighten it, then insert the KF16 clamp and tighten the nut.

(3) Pump Oil Addition

Unscrew the oil inlet of the mechanical pump (see Figure 1-4), slowly pour in the pump oil, observe the oil volume observation port at the front of the pump, and add the pump oil to 2/3 of the position (Figure 1-7), then tighten the oil inlet nut. If the pump oil is poured too much, use an Allen wrench to open the oil drain port next to the oil quantity observation port, drain the excess pump oil, and tighten the nut.

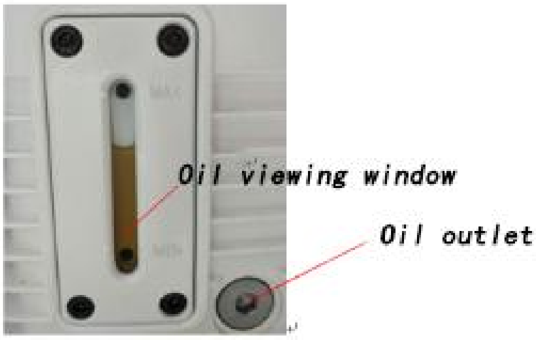


Figure 1-7

(4) Use of Pumps

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

When the instrument is in operation, the true pump switch needs to be turned on. When the operation is completed, the vacuum pump switch needs to be turned off.

2. Installation and Use of Vacuum Pump in Degassing Station

（1）Understanding Vacuum Pumps

As shown in Figure 1-8, the instrument comes standard with a bipolar rotary vane vacuum pump. Before installation, please familiarize yourself with the functions of each connector/switch on the pump to avoid incorrect installation. Be careful not to mix and install the suction port and exhaust port incorrectly, otherwise it will cause the pump body to explode and cannot be repaired or cause more serious safety accidents.

Attention:

Do not install the exhaust port as the suction port!!!

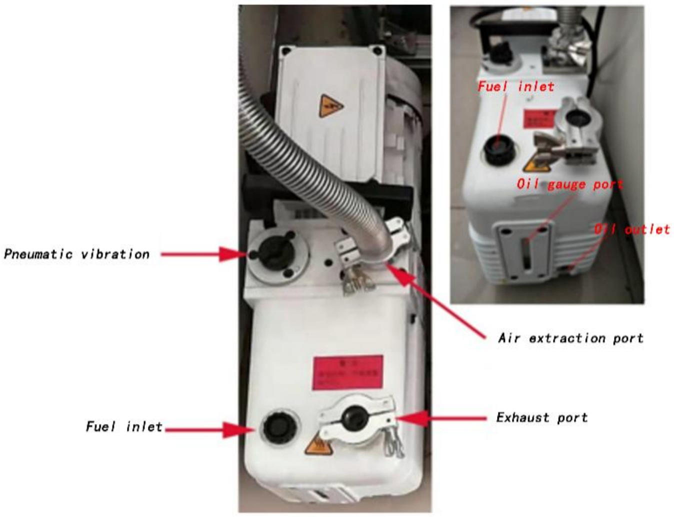


Figure 1-8

（2）Installing Corrugated Pipes

①Install KF16 card sleeve

As shown in Figures 1-9, place the KF16 sealing ring around the corrugated pipe opening, then insert the clamp and tighten the screw cap.

Figure 1-9

②When connecting to the instrument, align a section of the corrugated pipe that is inserted into the sealing ring with the vacuum pump suction port, then insert the KF16 clamp and tighten the clamp. Connect the other end of the corrugated pipe to the vacuum interface of the instrument using the same method and tighten the clamp.

Figure 1-10

(3) Add pump oil

Unscrew the oil inlet of the vacuum pump (see Figure 1-11), slowly pour the pump oil into the vacuum pump, observe the oil level through the oil observation port at the front end of the vacuum pump, and then tighten the oil inlet. If the pump oil is poured too much, use an Allen wrench to open the oil drain port next to the oil quantity observation port, drain the excess pump oil, and tighten the nut.

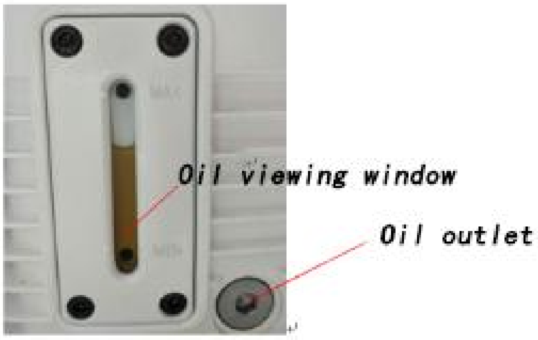


Figure 1-11

(4) Install the power cord for the pump

①Generally, one end of the power cord of a vacuum pump is already connected to the pump body. At this time, insert the other end into the three-phase power socket. After turning on the power switch of the vacuum pump, the pump will start working.

②When the pump body is not connected to the power cord, insert the matching power cord into the pump body power socket, and the other end into the three-phase power socket. After turning on the power switch of the vacuum pump, the pump starts working.

（3） Connection of Gas Path

1. Install the Sealing Ring for the Gas Pipeline

Take out one stainless steel external gas pipeline and insert one nut and two or three sealing rings at each end of the pipeline in sequence. As shown in Figure 1-12

Figure 1-12

2. Gas pipeline connection between gas cylinders and instruments

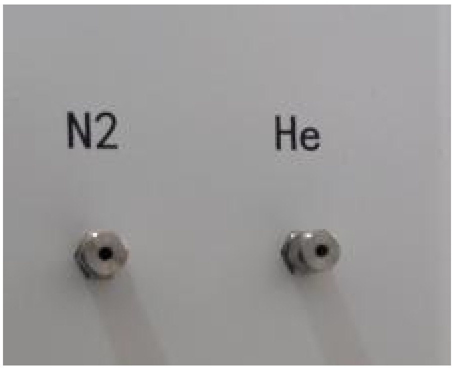


Figure 1-13

(1) Connect the nitrogen gas path

Insert one end of the gas pipeline into the "N2" gas interface of the instrument (see Figure 1-13 for the N2 interface), tighten the nut, and connect the other end to the pressure reducer outlet of the nitrogen cylinder, and tighten the nut.

(2) Connecting the helium gas path

Insert one end of the gas pipeline into the "He" gas interface of the instrument (see Figure 1-13 for the He interface), tighten the nut, and connect the other end to the pressure reducer outlet of the helium cylinder, and tighten the nut.

3. Gas Path Connection between Vacuum Pump and Instrument

(1) Connection between the host and mechanical pump air circuit

As shown in Figure 1-14, referring to the installation method of the corrugated pipe, connect one end of the corrugated pipe to the right suction port of the host, and the other end to the mechanical pump suction port.

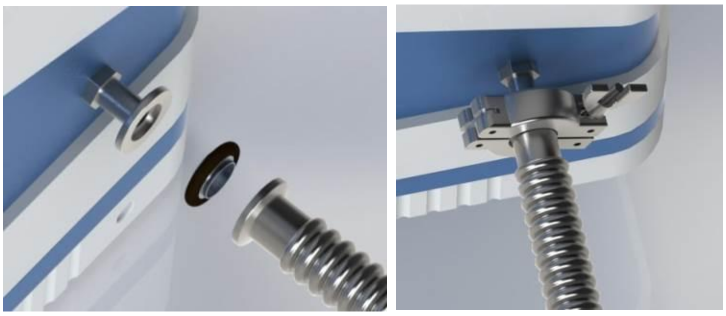


Figure 1-14

4. Gas circuit connection of degassing system

As shown in Figures 1-14, referring to the installation method of corrugated pipes, connect one end of the corrugated pipe to the left suction port of the host, and the other end to the mechanical pump suction port.

（4） Installation of the Host

1. Installation of Degassing Station Electric Furnace

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

(2) Insert the power cord interface of the electric furnace into the left "electric furnace" interface of the SSA-4000 host and tighten the nut. Align the groove of the heating furnace power cord with the protruding interface and insert it, then tighten the nut; When pulling out, release the nut, grip the power cord interface end tightly, and do not directly pull out the cable. As shown in Figure 1-15

Figure 1-15

Figure 1-16

2. Connect the Host Power Cord

Take out the power cord and insert one end into the instrument power interface, and the other end into the 220V power interface.

3. Host Turned on

Turn on the power switch of the host and turn on the instrument, as shown in Figure 1-17

Figure 1-17

(5) Software Installation

1. Installation of PC Analysis Software

Double click to open the "StencilWizard Setup" installation package, open the software installation wizard, follow the installation prompts, click "Next", click "Change" to customize the installation path to drive D, until the installation is completed. After completion, a shortcut icon will appear on the desktop.

StencilWizard - InstallShield Wizard

正在准备安装...

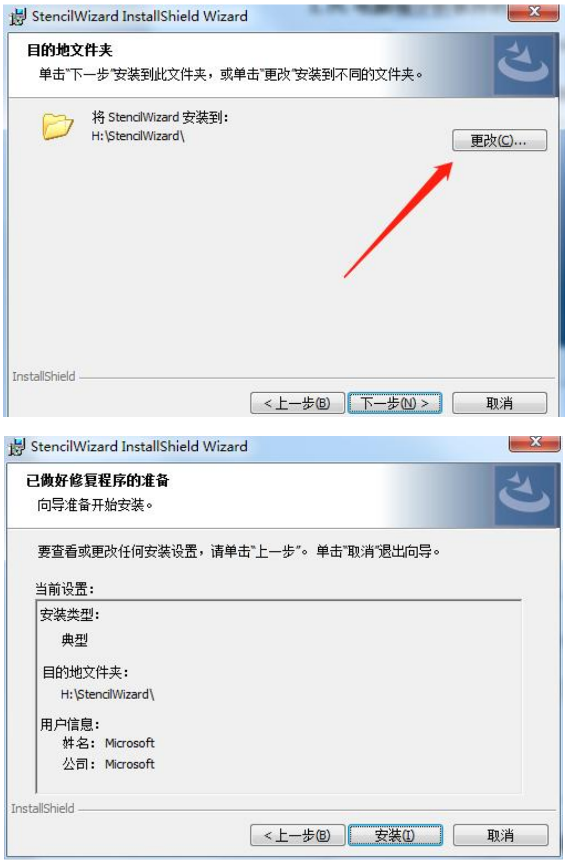
StencilWizard 安装程序正在准备 InstallShield Wizard，它将引导您完成剩余的安装过程。请稍候。

正在解压缩: StencilWizard.msi

StencilWizard InstallShield Wizard

欢迎使用 StencilWizard InstallShield Wizard

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



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\

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。

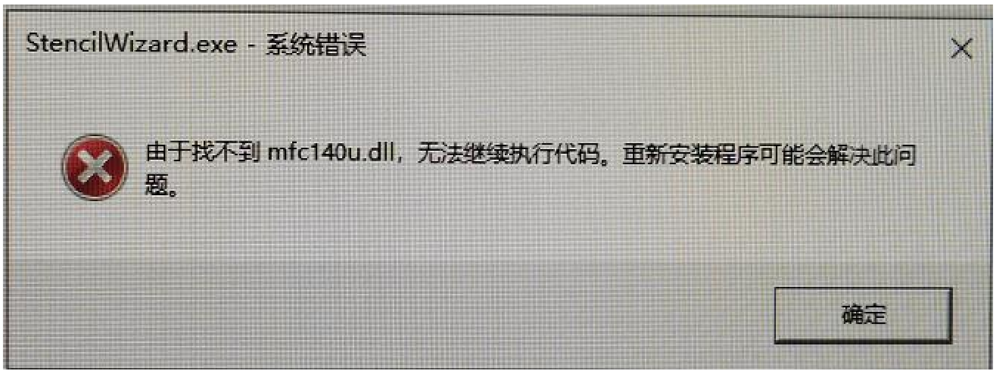
当前设置：
安装类型：
典型
目的地文件夹：
H:\StencilWizard\
用户信息：
姓名：Microsoft
公司：Microsoft

Figure 1-18

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

2. Analysis software cannot be used

When the software fails to open and there is an error as shown in Figure 1-19, it is necessary to install "mfc140u. dl" 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。

Figure 1-19

The system is Windows 10 X64

Step 1: Put mfc140u.dll into SysWOW64 on C drive Windows

Step 2: Run setupexe

3. Driver Installation

* To install the driver, please follow the steps below *

（1） Installation of XP/Win7 System Drivers

Before installing the driver, ensure that the data cable between the computer and the analysis host is correctly connected and turned on.。

① Right click on "My Computer" and select Properties, open Device Manager, then find "Universal Serial Bus Controller" and open it, or directly locate the location where "!" appears. As shown in Figure 1-20.

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

Figure 1-20

② Find the location where "!" appears, double-click, and select Custom Update Driver.

③ Select the driver installation package "Drive folder" in the installation program to update.

（2） Installation of Win10 System Drivers

Before installing the driver, it is necessary to lift the ban program on the Win10 system. The operation method is as follows:

** Attention: Please do not insert a USB drive when performing the following steps **

①Open window settings in three ways:

A. Click on the "window button ⊞" in the bottom left corner, then click on the icon "gear ⚙".

B. Right click the mouse on the desktop, click "Display Settings", and then click "Home".

Figure 1-21

C. Click on the notification, find and enter "All Settings".

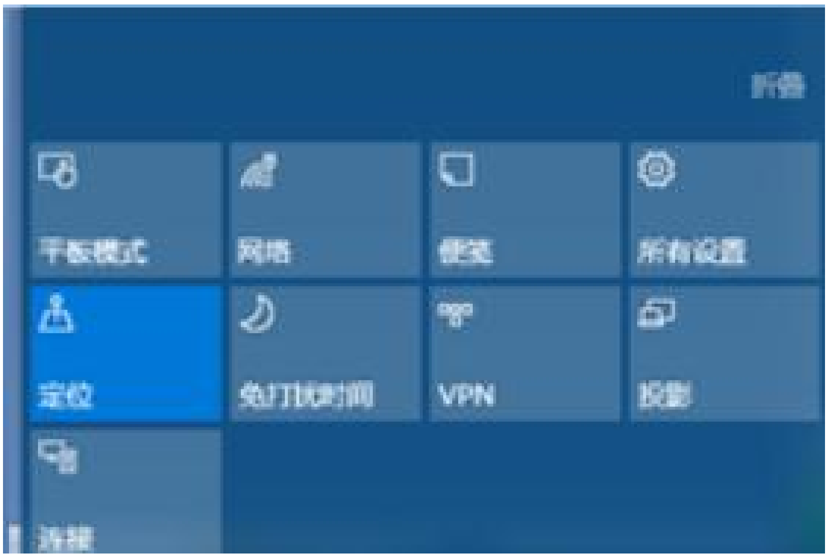


Figure 1-22

②Click on "Update and Security"

Figure 1-23

③ Click on "Restore"

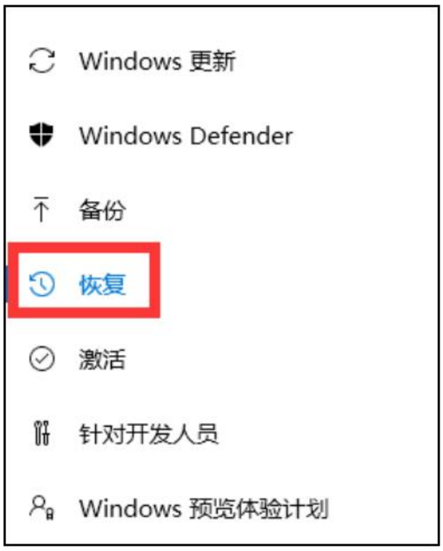
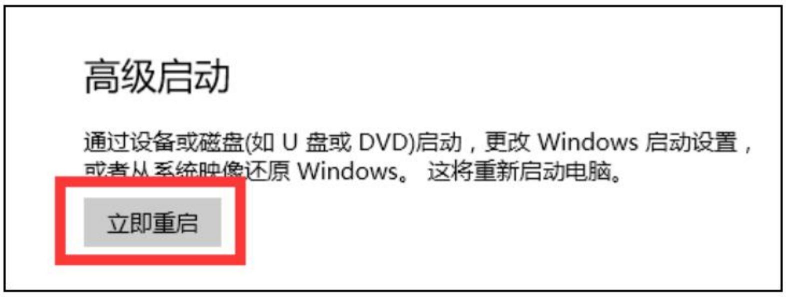


Figure 1-24

④ Click "Start Now" under "Advanced Startup", and after clicking, the computer will restart.



高级启动

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。

Figure 1-25

⑤ After restarting, select "Troubleshooting"

Figure 1-26

⑥ Select "Advanced Options" again

Figure 1-27

⑦ Select "Startup Settings"

Figure 1-28

⑧ Click "Restart" and the computer will restart.

Figure 1-29

⑨ After starting the computer, press "7" to complete the settings.

After lifting the ban program on the Win10 system, complete the driver installation using the XP/Win7 system installation method.

4. Connection between Computer Control end and SSA-4000 Host

Firstly, connect the external computer to the SSA4000 host using a USB cable, copy the computer control software to the root directory of the D drive, and create a shortcut on the desktop. As shown in Figure 1-30.

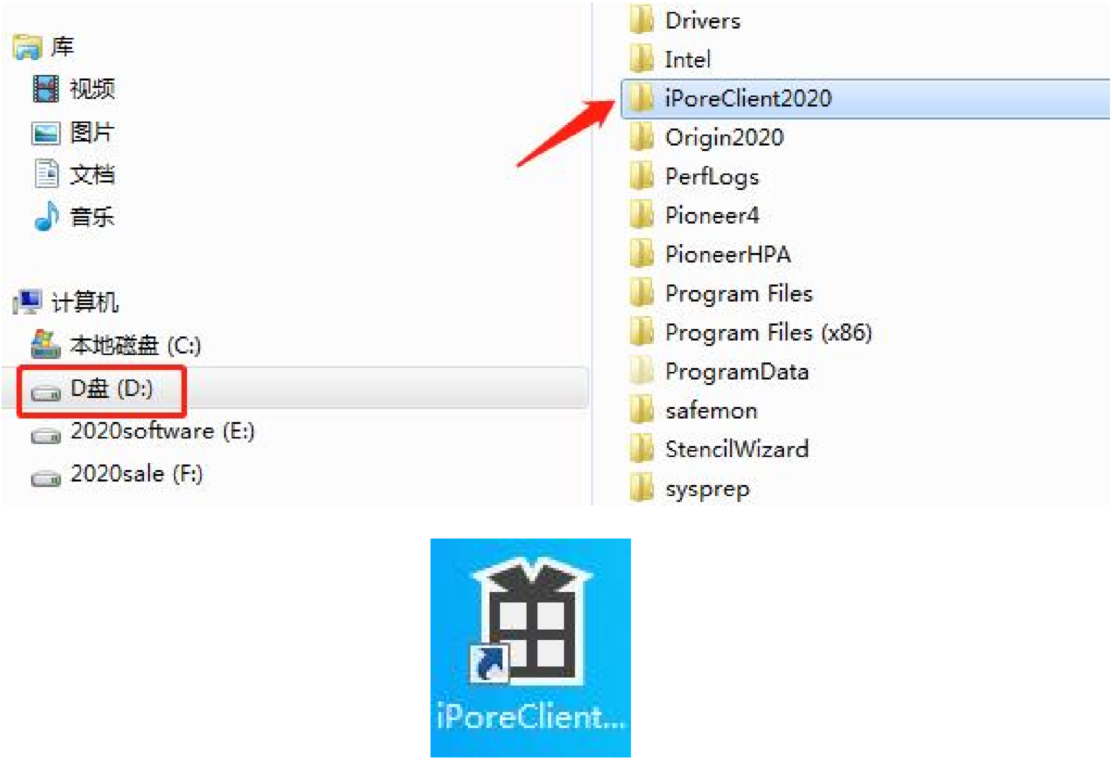


Figure 1-30

Chapter 2 Usage

(1) The Use of Gas Cylinders and Pressure Reducers

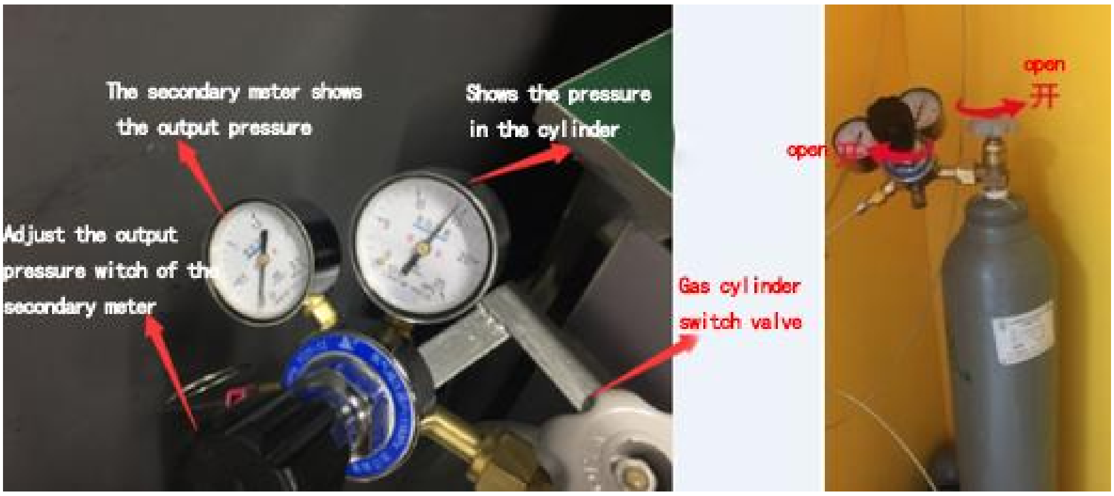


Figure 2-1

1. Understanding Pressure Reducers and Gas Cylinder Main Valves

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

2. Adjusting output Pressure

Open the main valve of the gas cylinder, then turn the black knob to adjust the output pressure (clockwise adjustment opens output, counterclockwise adjustment closes output), and adjust the nitrogen and helium output pressures to 0.3MPa respectively.

3. The Use of Gas Cylinders

When not in operation, simply close the main valve of the gas cylinder. Next time you use it, simply open the main valve of the gas cylinder without adjusting the output pressure again.

4. Gas Usage

Conventional nitrogen adsorption experiments typically use high-purity nitrogen gas (purity 99.999%) for adsorbate/gas, and high-purity helium gas (purity 99.999%) for dead volume quantitative gas.

(2) Operation Status Inspection and Calibration

1. Power on Operation Status Check

After powering on, open the analysis software.

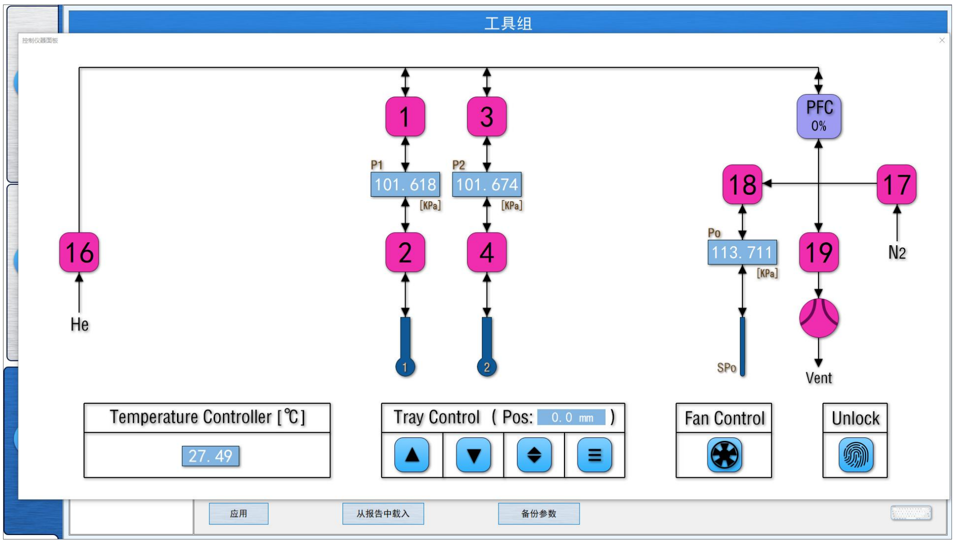


Figure 2-2

2. Inspection of Intake Pressure and Vacuum Status

(1) Click on the tool settings, control the instrument icon 控制仪器 , and enter the pneumatic control interface, as shown in Figure 2-2

(2) Check the intake pressure of helium and nitrogen gas

①Open the nitrogen and helium cylinders, with an output pressure of 0.3MPa; Turn on the vacuum pump.

②Click on valve 1, PFC, and valve 19 to vacuum and observe if the pressure value of "P1..." decreases. After about 5 minutes, check the displayed value of "P1...". At this time, the pressure value should be less than 0.04KPa.

③Close valve 19, open valve 16, and inject helium. At this time, the pressure in the system will rapidly increase. Observe the instantaneous reading of "P1..." within 5-10 seconds, and the pressure value should display 115-120 KPa; Then close valve 16, open valve 19, and continue to vacuum until the reading of "P1..." is less than 0.04KPa. Close valve 19, open valve 17, and inject nitrogen. At this time, the pressure in the system will rapidly increase. Observe the instantaneous reading of "P1..." within 5-10 seconds, and the pressure value should display 115-120KPa.

④According to the above method, if the intake pressure values of helium and nitrogen are too high or too low, it needs to be adjusted. At this point, it is necessary to find and adjust the stabilizing valve that controls the helium and nitrogen intake air paths respectively (using the stabilizing valve: increase the intake pressure clockwise and decrease the intake pressure counterclockwise).

3. Inspection of the Working Status of the Temperature Measurement System

After preheating the host, open the air circuit control interface and observe whether the temperature in the lower right corner is displayed as room temperature.

4. Exit the Computer Control Terminal

In the tool settings interface, select "background maintenance 后台维护 " to close the computer control end. **Remember not to choose to turn off the instrument 关闭仪器 , otherwise the computer will be immediately shut down.**

(3) The Use of Degassing Stations

1. Sampling and Weighing

(1) Determine Sample Dosage

Usually, the sample to be analyzed can provide a total surface area of 15 to 150 square meters, suitable for nitrogen adsorption analysis. Less than it will bring instability to the analysis results, while more than it will prolong the analysis time. For samples with a large specific area, the sample size should be small. For a small number of samples, weighing after degassing becomes very important, and a small amount of weighing error will account for a large proportion of the total weight. This weighing technique is crucial. The sample weight should not be less than 30 milligrams. The powder sample is collected using a long necked funnel, and the sample is added to the bottom of the sample tube. When the funnel is withdrawn, the sample tube should be slightly tilted horizontally to prevent residual samples from falling on the wall of the sample tube. Large particle samples should be added with tweezers (be careful not to touch the sample with your fingers to prevent oil from sticking to the surface of the sample).

Sample Dosage Reference Table:

Specific Surface [㎡/g]	< 1	1-10	10-100	100-300	> 300	notes
Sample Size [g]	> 2	1-2	> 0.5	> 0.1	< 0.1	Minimum cannot < 0.01

(2) Sample Weighing (including Water sample Weight)

Since the analysis results are expressed as surface area per unit mass, it is necessary to know the true mass of the sample. Carefully weigh the sample tube and sample.

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

② 将 A tray (which can be a measuring cylinder or glass cup, used to place a round bottomed sample tube) is weighed and peeled on a one in ten thousand scale to stabilize the scale at zero.

③ Weigh the sample tube and sample tube stopper together with the tray on the balance. After the balance values are stable, record the weight m1 of the empty sample tube and sample tube stopper. (See Figure 2-3)

Figure 2-3

④ Using a funnel, insert the sample into the bottom of the sample tube, and the stacking height of the loaded sample should be less than 15mm. (See Figure 2-4).

Figure 2-4

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

⑥Subtracting m1 from m2 yields the weight m3 of the sample before degassing and drying.

2. Installing Sample Tubes

The vast majority of sample surfaces adsorb a large amount of pollutants and impurities at room temperature. These impurities need to be removed before analysis and the sample surface needs to be cleaned. The sample is heated under vacuum to remove impurities from the surface, which is 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 (see Figure 2-5 on the left); The installation of the plug is shown on the right side of Figure 2-5.

Figure 2-5

（2）Install one nut, two sealing rings, and one filter plug at the sample tube mouth (for non powdered samples, the filter plug may not be installed), as shown in Figure 2-7

Figure 2-6

(3) Install the sample tube onto the degassing port, keeping it perpendicular to the ground, pressing it upwards, and then tightening the nut by hand.

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

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

Figure 2-7

(6) If the power cord of the heating furnace is not installed, the heating furnace should be installed first before proceeding with (5). Thread the power cord of the heating furnace through the hole in the center of the tray, insert the power cord into the electric furnace socket, and place the heating furnace on the lifting tray. As shown in Figure 2-8, align the groove of the heating furnace power cord with the interface protrusion, then insert the needle hole and tighten the nut; When pulling out, release the nut, grip the power cord interface end tightly, and do not directly pull out the cable.

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

Figure 22-8

3. Set Degassing Mode

300.0 ℃

-0.01 KPa

真空 1

注气 3

温度 300 ℃

时间 120 min

预处理中 80%

Figure 2-9

(1) Click on the "Temperature" and "Time" checkboxes, and enter the temperature and time that need to be processed separately. For samples with unknown processing time, the "AI" button can be checked simultaneously, and the instrument will automatically determine the degree of dehydration completion and record and display the time.

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

（3）Click the "S" button to start degassing, and the heating furnace will automatically heat up to the target temperature.

4. Weigh again after Degassing

（1）After degassing is completed, there will be a "drip" alarm sound and a pop-up window prompt. At this time, lower the heating furnace to cool the sample tube to room temperature, but be careful that the heating furnace is still in a high temperature (cooling process) state, and hands should not touch the furnace wall and mouth to avoid high-temperature burns.

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

（3）Reweigh (net weight of sample)

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

Note that if m (weight of sample after drying) is greater than m3 (weight of sample before drying), the influence of weighing or operating errors should be considered. If the difference is significant, the sample should be reprocessed according to the above steps.

（4） Start the Instrument

1. Turn on the power switch of the instrument, as shown in Figure 2-10

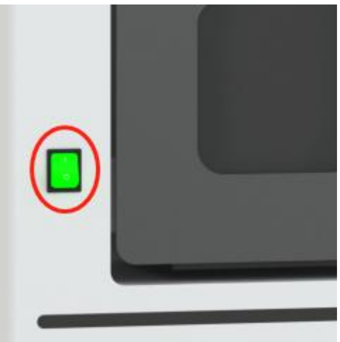
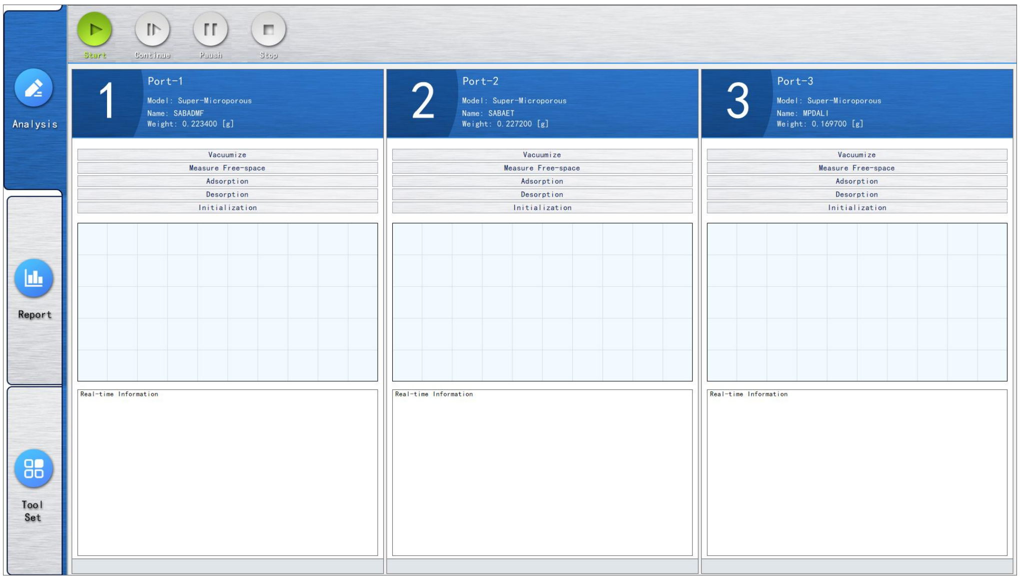


Figure 2-10

2. Open the computer and double-click the analysis software icon, as shown in Figure 2-11

Figure 2-11

3. The computer enters the analysis software operation page, as shown in Figure 2-12.



Port-1
Model: Super-Microporous
Name: SABADMF
Weight: 0.223400 [g]

Port-2
Model: Super-Microporous
Name: SABAET
Weight: 0.227200 [g]

Port-3
Model: Super-Microporous
Name: MPDALI
Weight: 0.169700 [g]

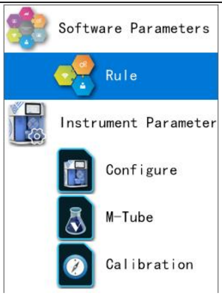
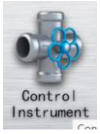
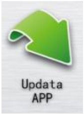
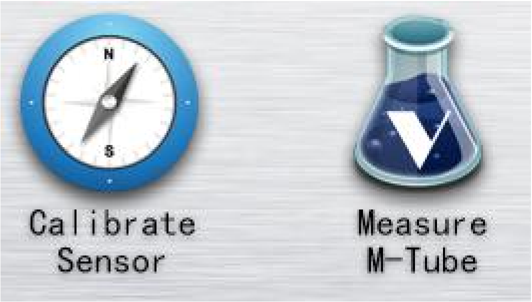
Figure 2-12

(5) Use of Analysis Software

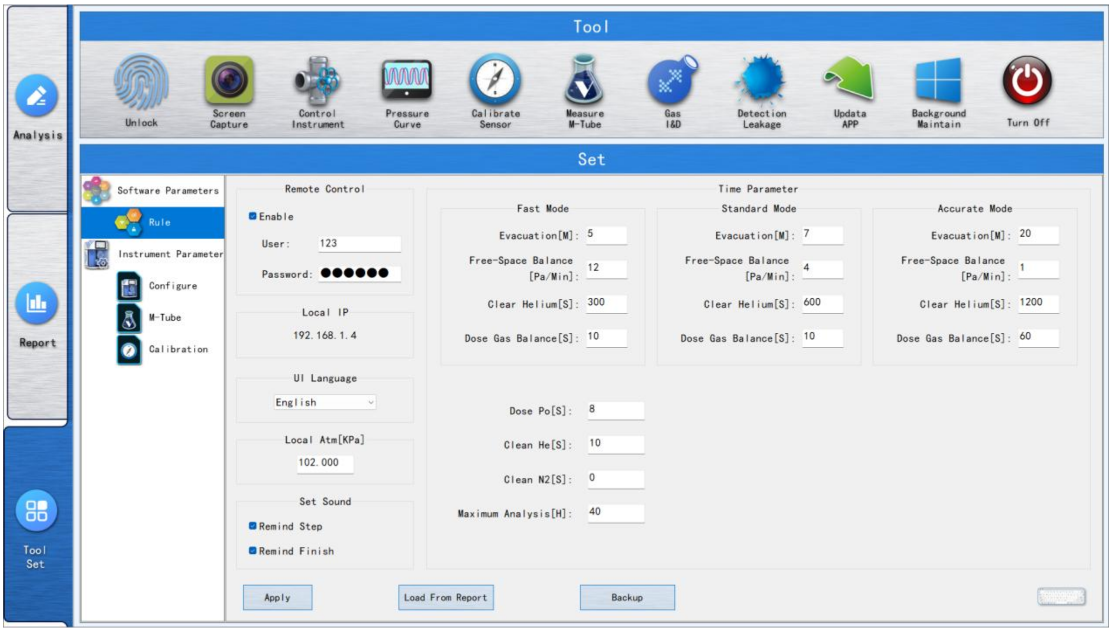
The computer screen is divided into three modules: "Analysis", "Report", and "Tool Settings". When using the instrument for the first time, you should first enter the "Tool Settings" module and set basic information on the "Instrument Parameters" interface.

1. Tool Settings Module

In this module, you should set basic parameters under the "User Operable" module. Do not operate other modules on your own, otherwise any losses caused should be the responsibility of the user.

<table>
<tr>
<td>1.1 User Operable Modules (left column)</td>
<td colspan="4"> </td>
</tr>
<tr>
<td rowspan="2">1.2 User Operability Module (Tool Group Module)</td>
<td> </td>
<td> </td>
<td> </td>
<td> </td>
</tr>
<tr>
<td>You can open the instrument's gas circuit to check the status of each component</td>
<td>When installing a new machine or replacing a gas cylinder, it is necessary to exhaust. Use default values for nitrogen and helium exhaust times</td>
<td>Under the guidance of the manufacturer, the system update should be carried out by placing the update file in the root directory of the E drive</td>
<td>View the pressure values of each analysis station</td>
</tr>
<tr>
<td colspan="2">1.3 It is strictly prohibited to operate modules (if operation is required, it should be carried out under the guidance of the manufacturer, phone number 400-669-8981)</td>
<td colspan="3"> </td>
</tr>
</table>

(1) Software Parameters - General Parameters, as shown in Figure 2-13



Remote Control
☑ Enable
User: 123
Password: ●●●●●●

Local IP
192.168.1.4

UI Language
English

Local Atm[KPa]
102.000

Set Sound
☑ Remind Step
☑ Remind Finish

Time Parameter

	Fast Mode	Standard Mode	Accurate Mode
Evacuation[M]:	5	7	20
Free-Space Balance [Pa/Min]:	12	4	1
Clear Helium[S]:	300	600	1200
Dose Gas Balance[S]:	10	10	60

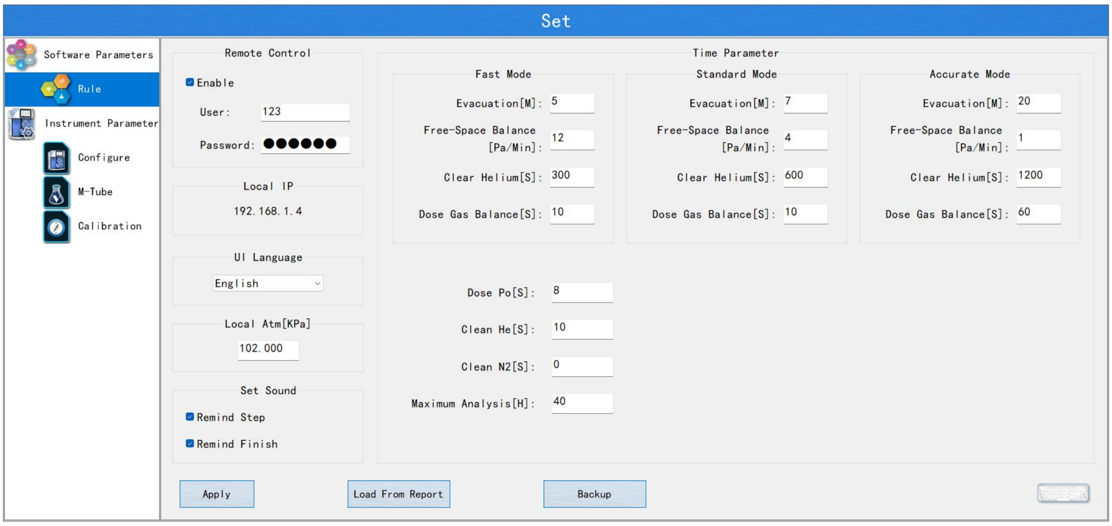
Dose Po[S]: 8
Clean He[S]: 10
Clean N2[S]: 0
Maximum Analysis[H]: 40

Figure 2-13

① Remote control: After selection, the instrument can be accessed using a computer.

②Time parameters: divided into fast, standard, and high-precision analysis modes, generally using default values (do not change them on your own).

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



Remote Control
☑ Enable
User: 123
Password: ●●●●●●

Local IP
192.168.1.4

UI Language
English

Local Atm[KPa]
102.000

Set Sound
☑ Remind Step
☑ Remind Finish

Time Parameter

	Fast Mode	Standard Mode	Accurate Mode
Evacuation[M]:	5	7	20
Free-Space Balance [Pa/Min]:	12	4	1
Clear Helium[S]:	300	600	1200
Dose Gas Balance[S]:	10	10	60

Dose Po[S]: 8
Clean He[S]: 10
Clean N2[S]: 0
Maximum Analysis[H]: 40

Figure 2-14

① Configure instruments: Set instrument model, number of analysis stations, generation date, instrument number, etc., and click apply.

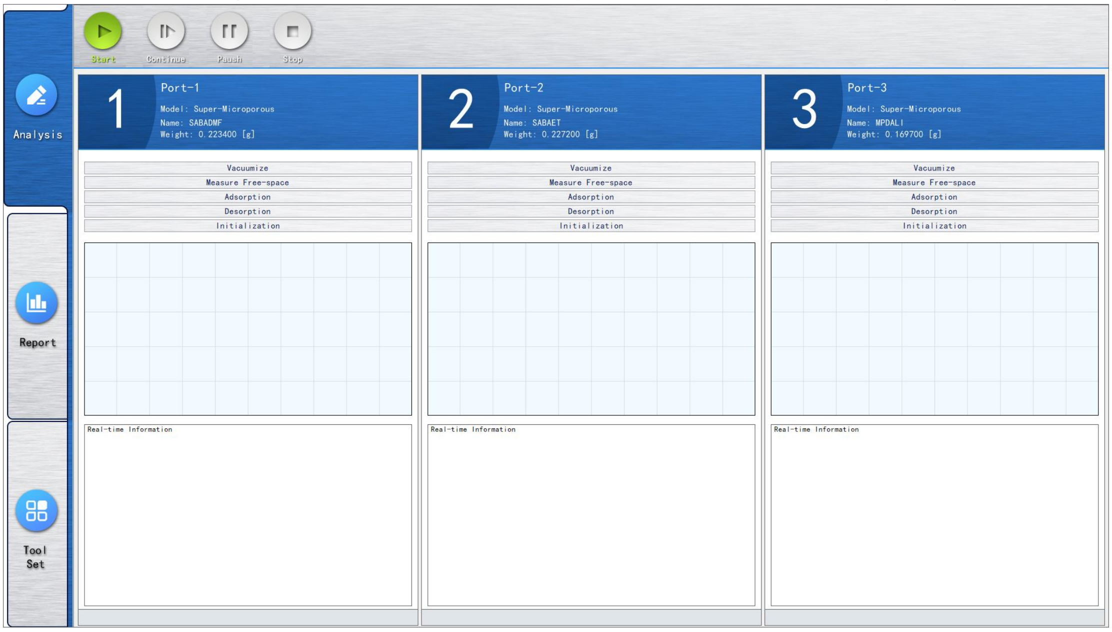
② Measurement cabin volume: Factory default parameters, no need for user settings.

③ Calibration parameters: Factory default parameters, no need for user settings.

④Set PFC: Factory default parameters, no need for user settings.

2. Analysis Module

As shown in Figure 2-15, this is a commonly used interface for testing samples.



Port-1
Model: Super-Microporous
Name: SABADMF
Weight: 0.223400 [g]

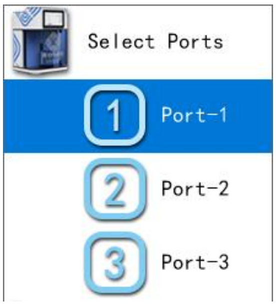
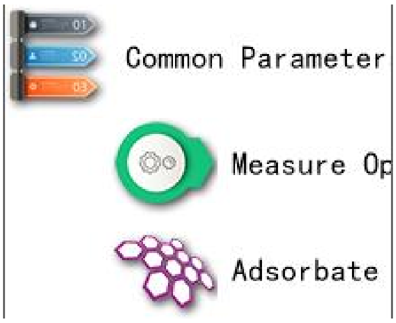
Port-2
Model: Super-Microporous
Name: SABAET
Weight: 0.227200 [g]

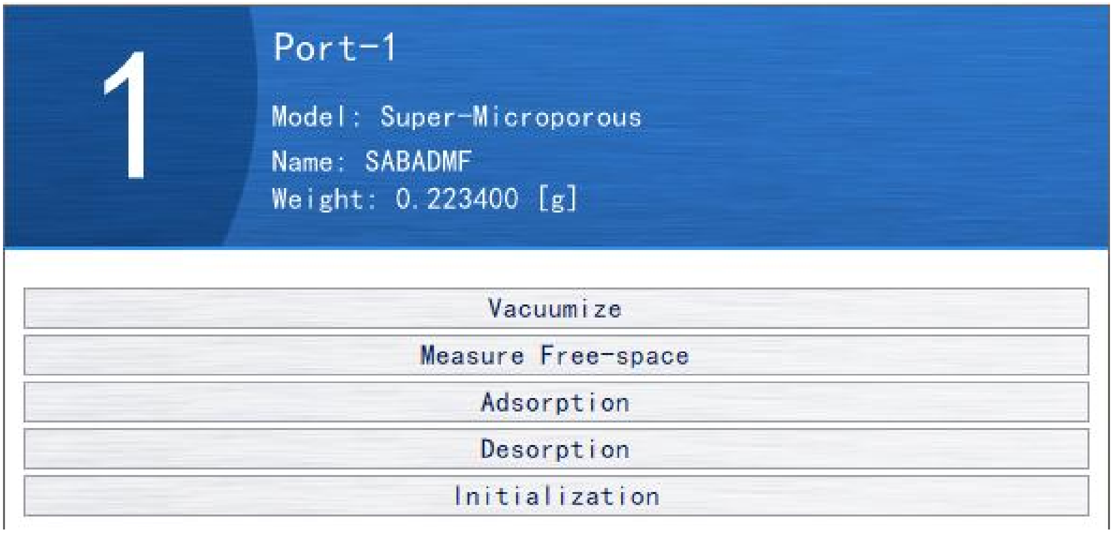
Port-3
Model: Super-Microporous
Name: MPDALI
Weight: 0.169700 [g]

Figure 2-15

（1）Set Analysis Station Analysis Parameters

As shown in Figure 2-16, click on the blue area of the analysis station to enter the "Set Measurement Parameters" interface. After opening, you can set the measurement parameters of each analysis station; Common parameters are generally set at the factory and do not require modification.

分析站测量参数设定	公共参数设定
	



1

Port-1

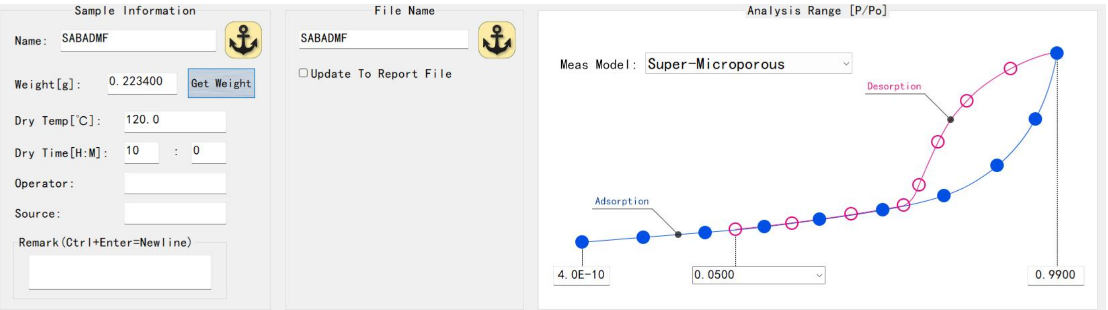
Model: Super-Microporous

Name: SABADMF

Weight: 0.223400 [g]

Figure 2-16

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



Sample Information

Name: SABADMF

Weight[g]: 0.223400

Dry Temp[℃]: 120.0

Dry Time[H:M]: 10 : 0

Operator:

Source:

Remark(Ctrl+Enter=Newline)

File Name

SABADMF

☐ Update To Report File

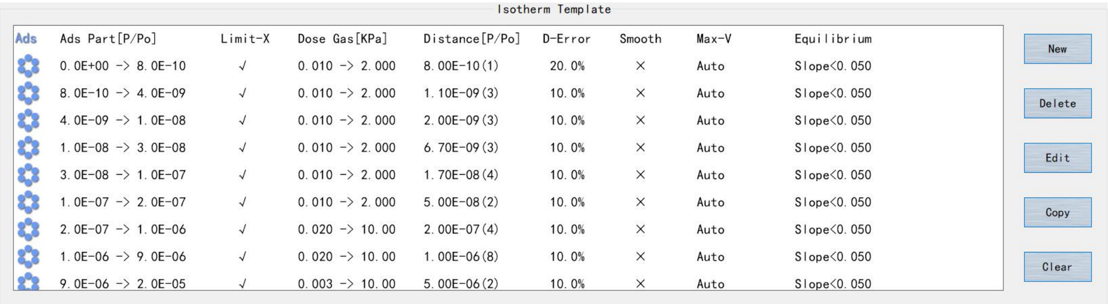
Analysis Range [P/Po]

Meas Model: Super-Microporous

Figure 2-17

Five analysis types can be set, including specific surface/mesoporous/microporous/ultra microporous/isothermal adsorption, and different analysis types can be matched with different P/Po analysis ranges.

In different testing modes, the isotherm collection range and number of points can be set. Please do not make any modifications on your own without understanding the sample and operating system. If any modifications are needed, please contact the manufacturer's engineer. As shown in Figure 2-18



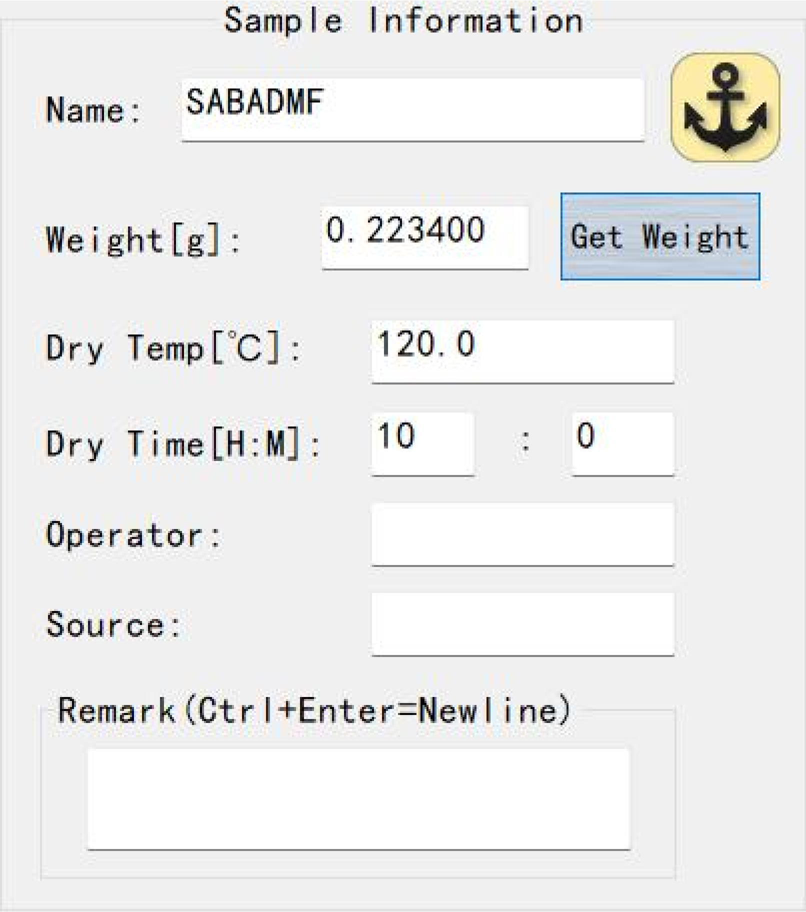
Isotherm Template

Ads	Ads Part[P/Po]	Limit-X	Dose Gas[KPa]	Distance[P/Po]	D-Error	Smooth	Max-V	Equilibrium
	0.0E+00 -> 8.0E-10	√	0.010 -> 2.000	8.00E-10(1)	20.0%	×	Auto	Slope<0.050
	8.0E-10 -> 4.0E-09	√	0.010 -> 2.000	1.10E-09(3)	10.0%	×	Auto	Slope<0.050
	4.0E-09 -> 1.0E-08	√	0.010 -> 2.000	2.00E-09(3)	10.0%	×	Auto	Slope<0.050
	1.0E-08 -> 3.0E-08	√	0.010 -> 2.000	6.70E-09(3)	10.0%	×	Auto	Slope<0.050
	3.0E-08 -> 1.0E-07	√	0.010 -> 2.000	1.70E-08(4)	10.0%	×	Auto	Slope<0.050
	1.0E-07 -> 2.0E-07	√	0.010 -> 2.000	5.00E-08(2)	10.0%	×	Auto	Slope<0.050
	2.0E-07 -> 1.0E-06	√	0.020 -> 10.00	2.00E-07(4)	10.0%	×	Auto	Slope<0.050
	1.0E-06 -> 9.0E-06	√	0.020 -> 10.00	1.00E-06(8)	10.0%	×	Auto	Slope<0.050
	9.0E-06 -> 2.0E-05	√	0.003 -> 10.00	5.00E-06(2)	10.0%	×	Auto	Slope<0.050

Figure 2-18

② **Sample information, as shown in Figure 2-19**

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



Sample Information

Name: SABADMF

Weight[g]: 0.223400

Dry Temp[℃]: 120.0

Dry Time[H:M]: 10 : 0

Operator:

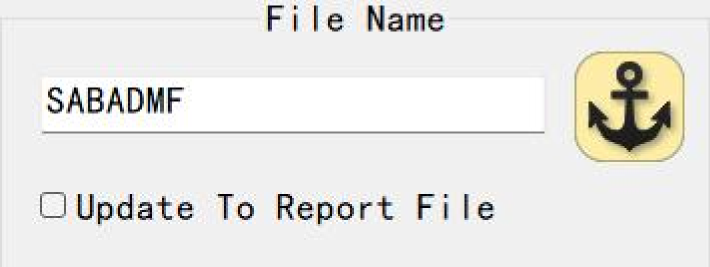
Source:

Remark(Ctrl+Enter=Newline)

Figure 2-19

The sample name can be modified arbitrarily, but it cannot have special characters, such as:?/* # $!

③ **Create a new File Name**



File Name

SABADMF

☐ Update To Report File

Figure 2-20

This is the file name that automatically generates the current data report after the analysis is completed. When there are no modifications, the save format in the database is "Port1-2020-12-18-1" (i.e. analysis station 1-current time - number of tests on that day).

Select the Lock Key ⚓	**Unlock Key** ⚓
Lock key background turns yellow: file name matches sample name for storage	Lock key background turns blue: The file name can be modified arbitrarily, but it cannot have special characters, such as:?/* # $!

(2) Set Public Parameters

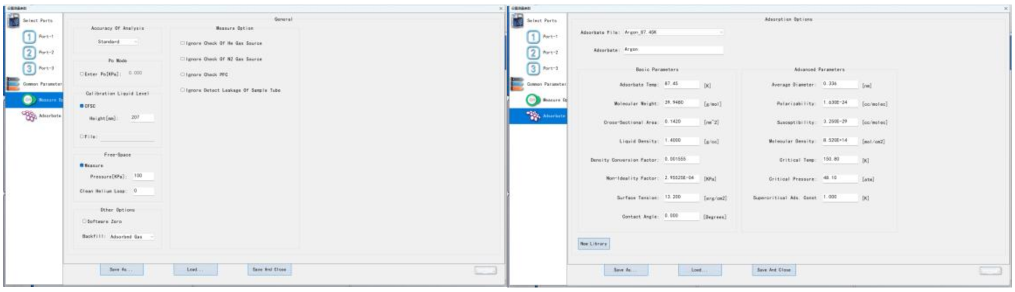
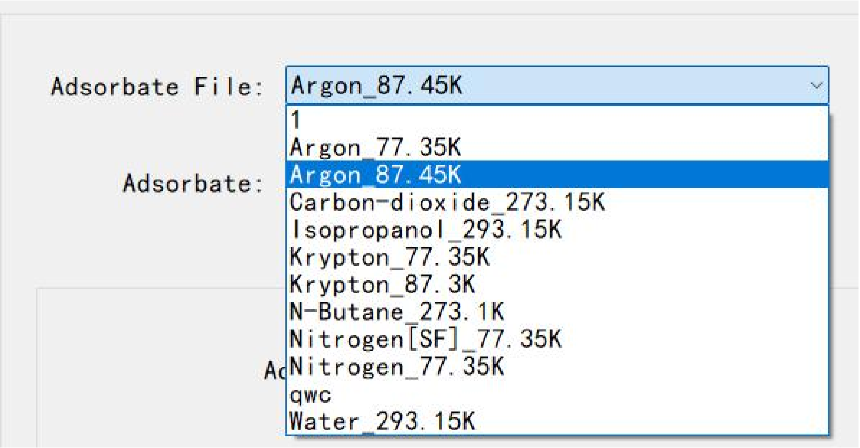


Figure 2-21

① Here, all you need to do is select the type of adsorbent. The system defaults to 77K nitrogen. When using other adsorbents, simply select from the drop-down menu. As shown in Figure 2-22

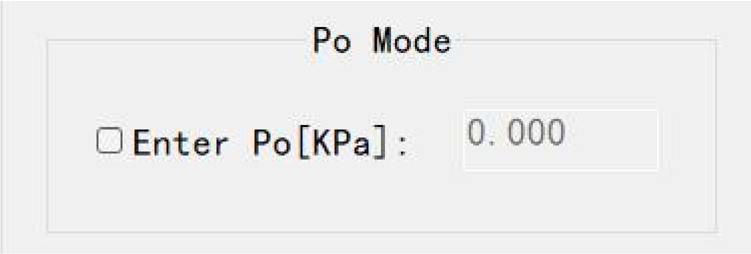


Adsorbate File: Argon_87.45K

Adsorbate:

Figure 2-22

② When "room temperature adsorption" is required, simply select the Po acquisition method. The adsorption mass parameters, adsorption isotherms, etc. can be customized and modified, and can be saved as a library file, which can be directly called next time.


Po Mode

☐ Enter Po[KPa]: 0.000

Figure 2-23

3. Reports Module

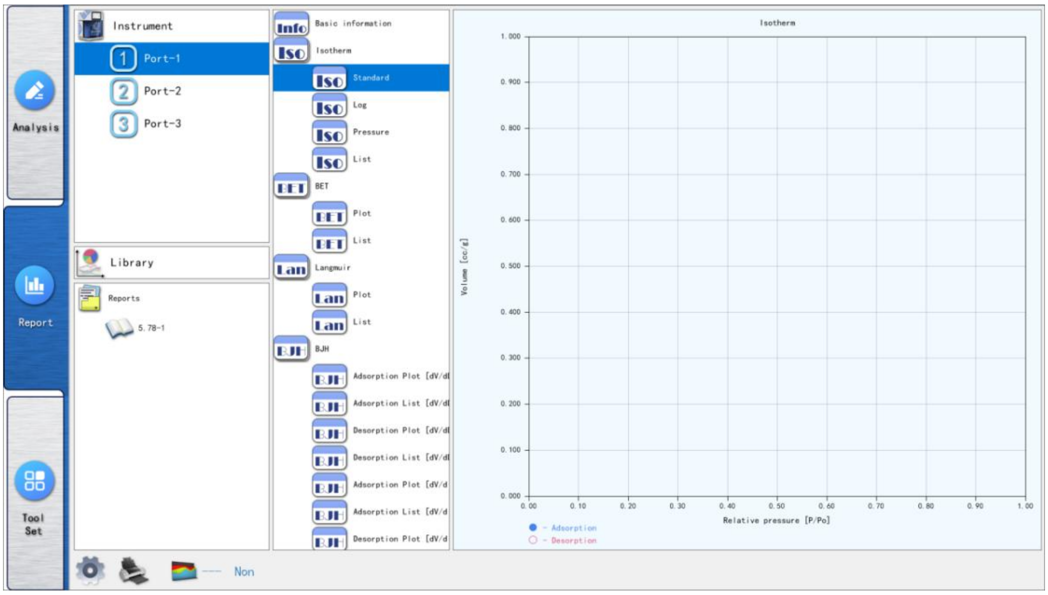


Figure 2-24

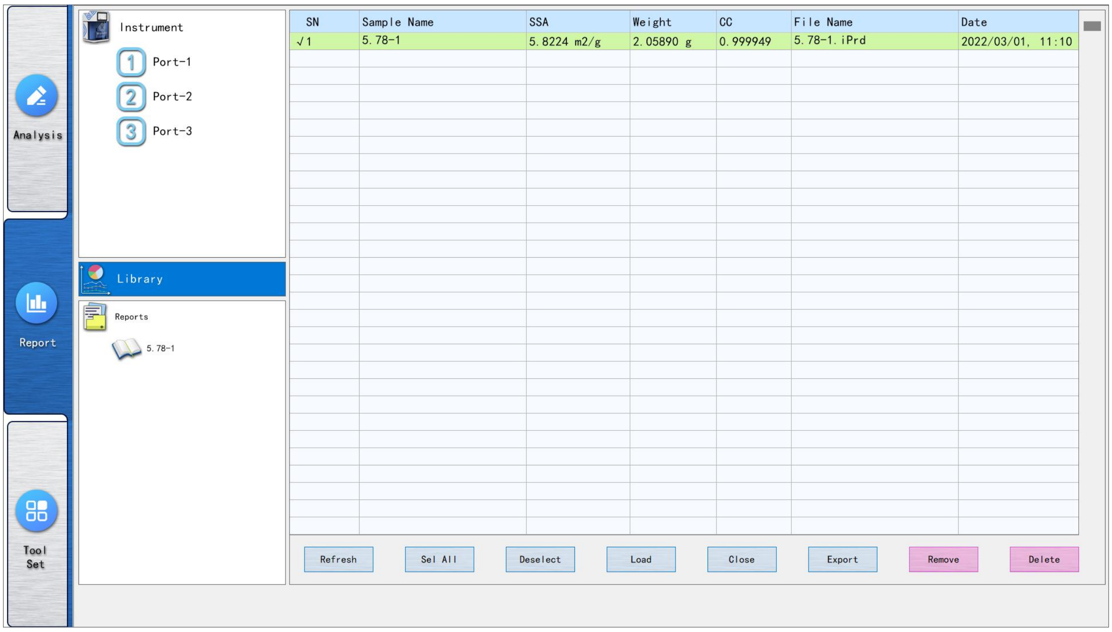
(1) Instrument - Analysis Station

Display the model graphs of each analysis station under the current main interface test window. For example, basic sample information, isotherms, BET, Langmuir, BJH, etc.

(2) View Past Test Reports

Click on "Report Library" to load previous files.

① In the selected state, click "Load Report Load " to add the target file to the "Loaded Report Reports ". Each target file is displayed below the loaded report, and you can click to view it separately.



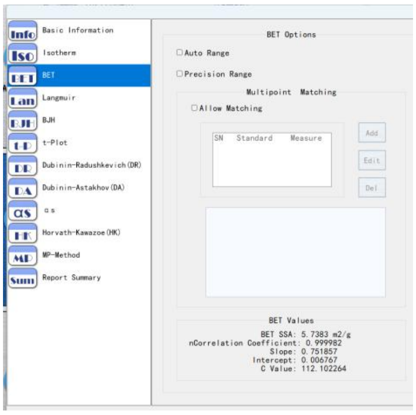
SN	Sample Name	SSA	Weight	CC	File Name	Date
√1	5. 78-1	5. 8224 m2/g	2. 05890 g	0. 999949	5. 78-1. iPrd	2022/03/01, 11:10

Figure 2-25

Figure 2-26

(3) Set Model Related Parameters

Click the "Settings " button to open the model settings window, where you can re edit the relevant parameters of each model. After making the changes, click "Save" and close.



☐ Auto Range

☐ Precision Range

☐ Allow Matching

BET Values

BET SSA: 5. 7383 m2/g
nCorrelation Coefficient: 0. 999982
Slope: 0. 751857
Intercept: 0. 006767
C Value: 112. 102264

Figure 2-27

（6） Isothermal Adsorption Test (Gas Adsorption Performance Analysis)

1. Install a Constant Temperature Dewar Jar and Controller

The inlet and outlet of the Dewa can are marked with in and out. The inlet of the Dewa can is connected to the outlet of the controller (high and low temperature circulator), and the outlet of the Dewa can is connected to the inlet of the controller. The tower joint is directly inserted into the water bath connection pipe, as shown in Figure 2-28. Select the corresponding liquid medium to add to the Dewar tank and controller.

According to the measured temperature, fill the controller box with recyclable liquids such as water/oil, and fill it with the same liquid (1-3cm below the tank opening) in a constant temperature Dewar tank. Afterwards, place the constant temperature Dewar jar on the lifting plate of the analysis station.

Figure 2-28

2. Install Adsorbed Gas

（1）Replacing gas cylinders

Connect the required replacement adsorbed gas to the N2 interface of the instrument, adjust the output pressure to 0.2-0.3MPa, and check if there is any gas leakage at the connection.

（2）Open the air Circuit Control

Replace the analysis port with a clean empty sample tube and tighten the seal as required.

Open the vacuum pump, open the software, click on "Tool Settings", "Control Instrument", and the air circuit control interface will pop up, as shown in the following figure (red represents valve closed, green represents open).

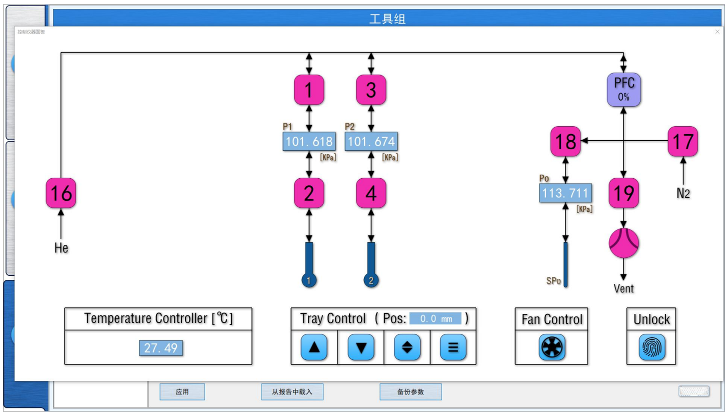


Figure 2-29

(3) Adjusting Intake Pressure

①Click "Unlock" in the bottom right corner and enter the password (system 1) to unlock. Open valves 19, PFC (pop up dialog box, click open), 18, 1, and 3 in sequence, as shown in Figure 2-30, and vacuum for about 5 minutes.

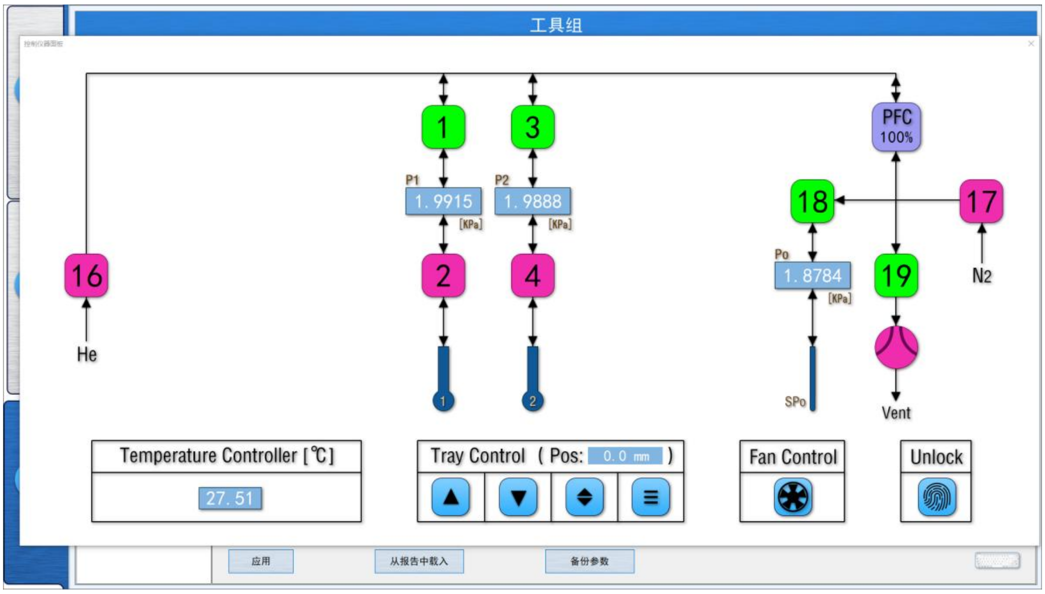


Figure 2-30

②Close valve 19 and open valve 17, as shown in Figure 2-31, while observing whether the pressure sensor values of P0, P1, P2, and P3 reach between 115MPa-120KPa. If the pressure is insufficient, the corresponding stabilizing valve knob on the instrument can be adjusted clockwise to the desired pressure; If the pressure is too high, first close valve 17, turn the pressure stabilizing valve knob counterclockwise to reduce it, then open valve 19 to vacuum, and after 1 minute, close valve 19 and open valve 17 to inject air. At this time, slowly adjust the pressure stabilizing valve knob clockwise and observe the intake pressure until it is adjusted to the required pressure.

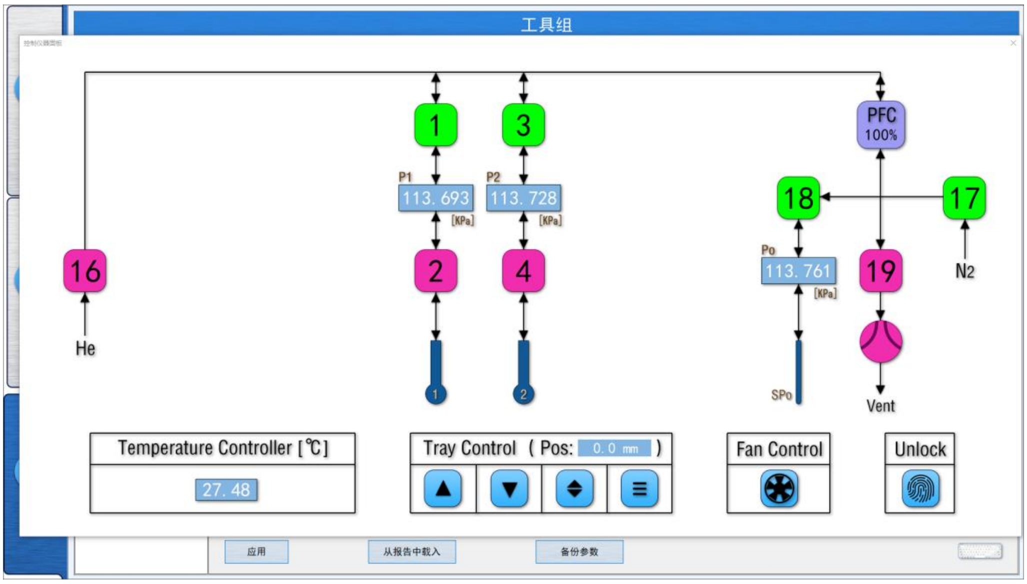


Figure 2-31

(4) Purification Gas Path

After adjusting the pressure, close the main valve of the gas cylinder and open valves 2, 4, 17, and 19 to vacuum for more than 12 hours, as shown in Figure 2-32.

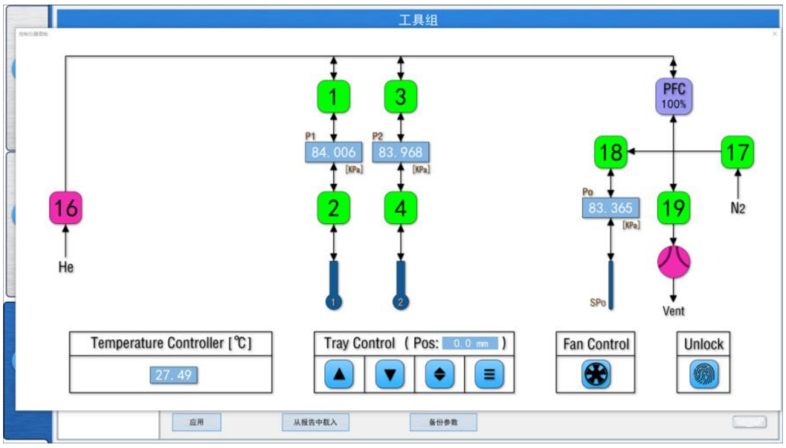
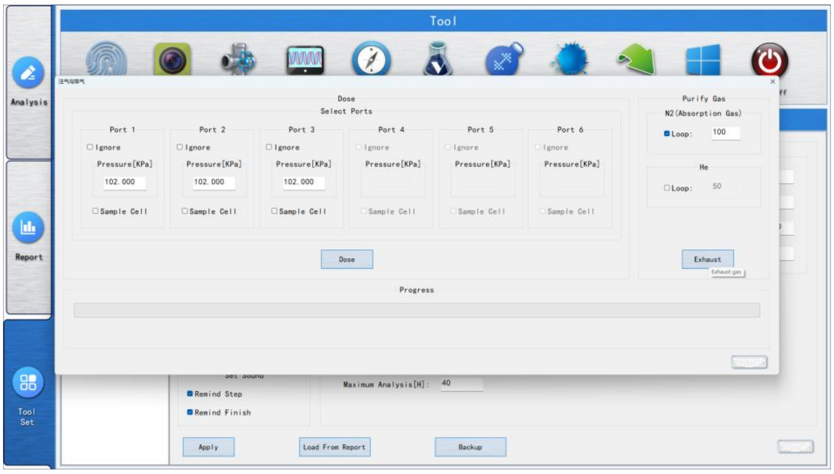


Figure 2-32

(5) Replacement Gas

After 12 hours, close valve 19, open the gas cylinder, close the gas path control interface, click "Gas injection and exhaust", and the gas injection and exhaust interface will pop up, as shown in Figure 2-33. Click "Start exhaust", and when the progress is completed, the gas replacement is completed. (This interface setting can refer to the following figure for the same settings)



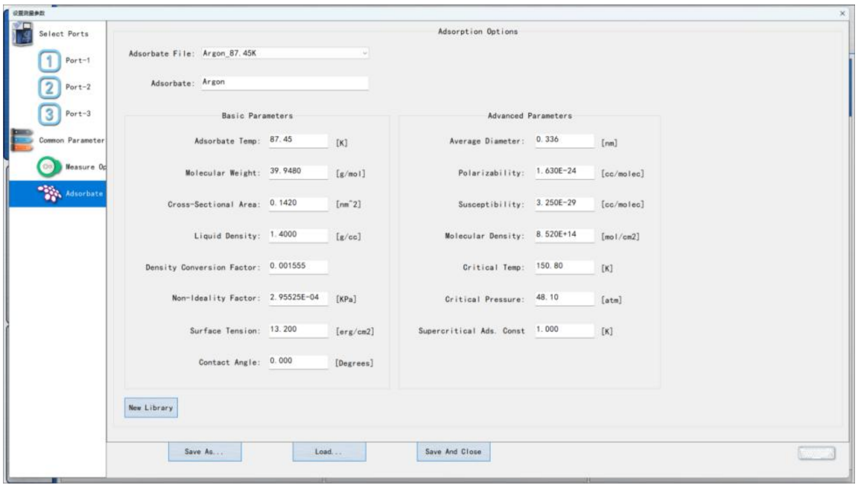
Port 1: ☐ Ignore; Pressure[KPa] 102.000; ☐ Sample Cell
Port 2: ☐ Ignore; Pressure[KPa] 102.000; ☐ Sample Cell
Port 3: ☐ Ignore; Pressure[KPa] 102.000; ☐ Sample Cell
Port 4: ☐ Ignore; Pressure[KPa]; ☐ Sample Cell
Port 5: ☐ Ignore; Pressure[KPa]; ☐ Sample Cell
Port 6: ☐ Ignore; Pressure[KPa]; ☐ Sample Cell
N2(Absorption Gas): ☑ Loop: 100
He: ☐ Loop: 50
☑ Remind Step
☑ Remind Finish
Maximum Analysis[H]: 40

Figure 2-33

3、Software Settings

(1) Adsorbent Selection

Click on "Adsorbent Settings" as shown in Figure 2-34, select the corresponding adsorption gas parameters. If there is no corresponding gas in the library, you can manually input the parameters and click "Generate New Library".



Adsorbate File: Argon_87.45K

Adsorbate: Argon

Adsorbate Temp: 87.45 [K]

Molecular Weight: 39.9480 [g/mol]

Cross-Sectional Area: 0.1420 [nm^2]

Liquid Density: 1.4000 [g/cc]

Density Conversion Factor: 0.001555

Non-Ideality Factor: 2.95525E-04 [KPa]

Surface Tension: 13.200 [erg/cm2]

Contact Angle: 0.000 [Degrees]

Average Diameter: 0.336 [nm]

Polarizability: 1.630E-24 [cc/molec]

Susceptibility: 3.250E-29 [cc/molec]

Molecular Density: 8.520E+14 [mol/cm2]

Critical Temp: 150.80 [K]

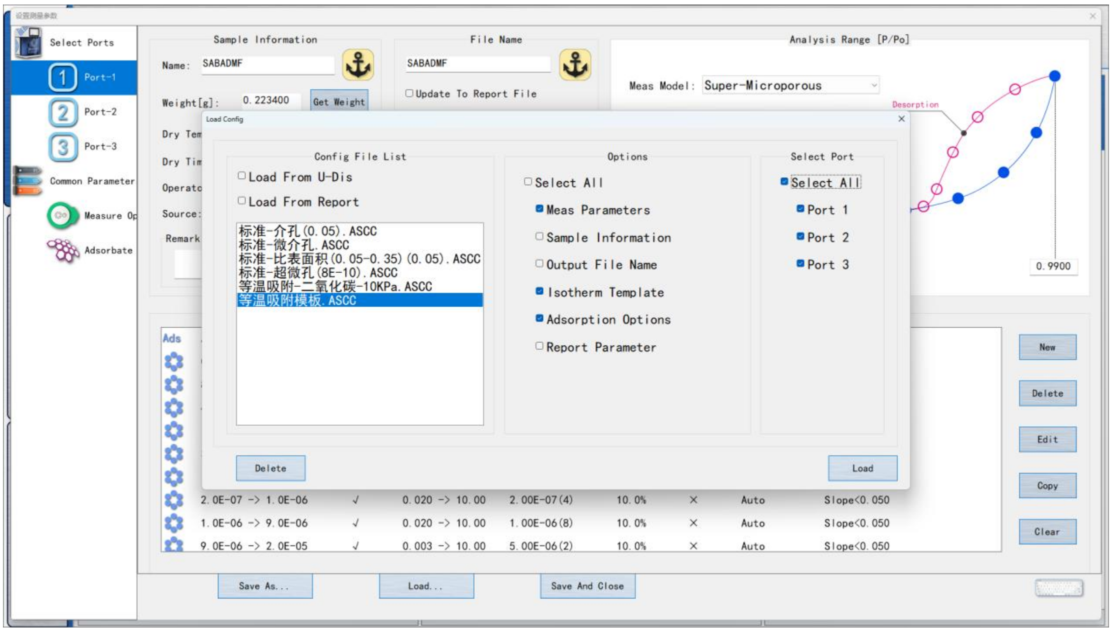
Critical Pressure: 48.10 [atm]

Supercritical Ads. Const 1.000 [K]

Figure 2-34

（2）Load Template

Enter the sample name, weight, and other parameters, click "Load", and select the isothermal adsorption template.



Name: SABADMF

Weight[g]: 0.223400

File Name: SABADMF

☐ Update To Report File

Meas Model: Super-Microporous

☐ Load From U-Dis

☐ Load From Report

标准-介孔(0.05).ASCC
标准-微介孔.ASCC
标准-比表面积(0.05-0.35)(0.05).ASCC
标准-超微孔(8E-10).ASCC
等温吸附-二氧化碳-10KPa.ASCC
等温吸附模板.ASCC

☐ Select All
☑ Meas Parameters
☐ Sample Information
☐ Output File Name
☑ Isotherm Template
☑ Adsorption Options
☐ Report Parameter

☑ Select All
☑ Port 1
☑ Port 2
☑ Port 3

2.0E-07 -> 1.0E-06	√	0.020 -> 10.00	2.00E-07(4)	10.0%	×	Auto	Slope<0.050
1.0E-06 -> 9.0E-06	√	0.020 -> 10.00	1.00E-06(8)	10.0%	×	Auto	Slope<0.050
9.0E-06 -> 2.0E-05	√	0.003 -> 10.00	5.00E-06(2)	10.0%	×	Auto	Slope<0.050

Figure 2-35

Chapter 3 Analysis of Samples

(1) Preparation before Analysis

To ensure analysis accuracy and repeatability, preparation should be carried

out in the following order and steps.

Cleaning and labeling sample tubes

Sample weighing

Sample degassing

Installing sample tubes

1. Cleaning and Labeling of Sample Tubes

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

(1)Firstly, make sure that the ultrasonic pool is clean. Add about 500 milliliters of hot water, 5 milliliters of detergent or surfactant, and place the sample tube and filler rod in it. Then, ultrasonically clean for about 15 minutes and remove it (see Figure 3-1).

Figure 3-1

(2)Wear rubber gloves and remove the sample tube, then clean the inside of the tube with a specialized 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).

Figure 3-2

(5)Place the sample tube and filler rod on a rack to dry naturally or use an oven or vacuum oven, set it to 110 degrees Celsius for 2 hours to dry for later use.

(6)Wipe the sample tube stopper clean with a clean silk cloth and install it on the sample tube.

(7)Label the sample tube and the sample tube with rubber plugs.

(8)Use a sample tube stopper to plug the sample tube (see Figure 3-3).

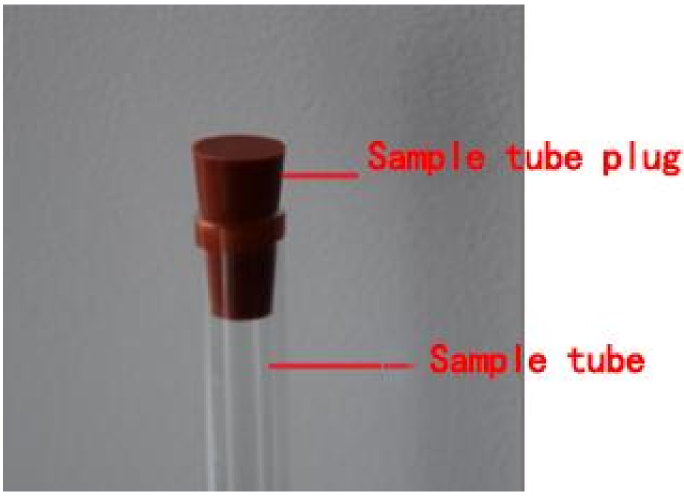


Figure 3-3

2. Sample Weighing and Dehydration

Refer to "Usage of Degassing Station" (see page 9), weigh and dehydrate the sample.

3. Installing Sample Tubes

(1)Remove the sample tube plug from the nozzle.

(2)Insert the filling rod and place the sample tube and filling rod horizontally. (See Figure 3-3)

Figure 3-3

Installing the filler rod vertically downwards will break the sample tube.

(3)After installing the filler rod, install a nut, 2 sealing rings, and filter plug (if it is not a powder sample, filter plug can be omitted) (see Figure 3-4)

Figure 3-4

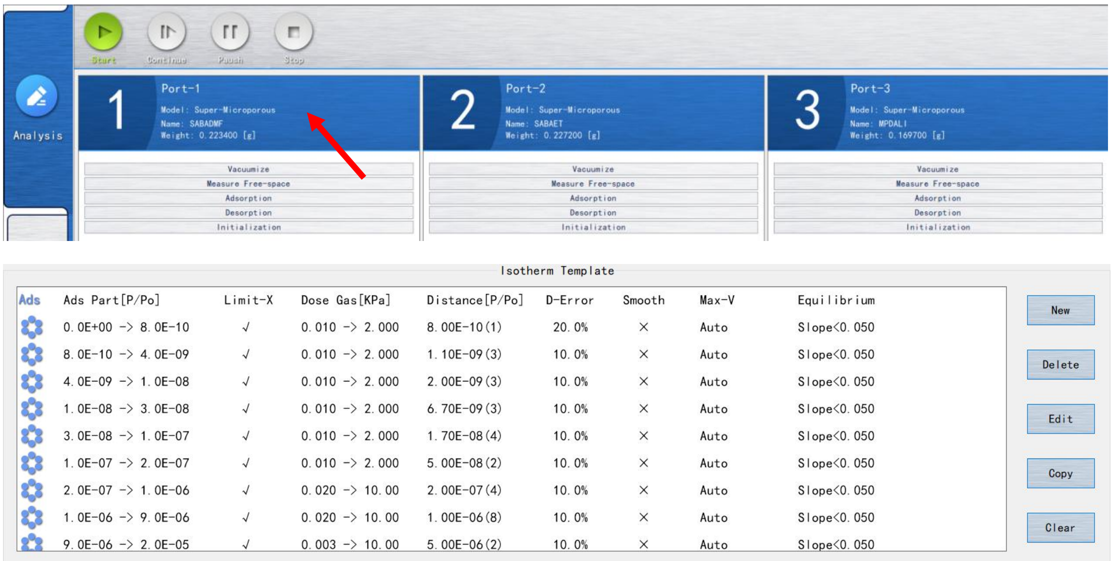
(4)Install the sample tube onto the analysis station, tighten it upwards, keep the sample tube perpendicular to the ground, hold the bottom of the sample tube with one hand, tighten the nut with the other hand, and then use a wrench to tighten half a turn to one turn.

(2) Measurement Parameter Settings

Refer to Chapter 2, Section (5) for the use of "2. Analysis Module".

1. Using System Templates

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



1 Port-1
Model: Super-Microporous
Name: SABADMF
Weight: 0.223400 [g]

2 Port-2
Model: Super-Microporous
Name: SABAET
Weight: 0.227200 [g]

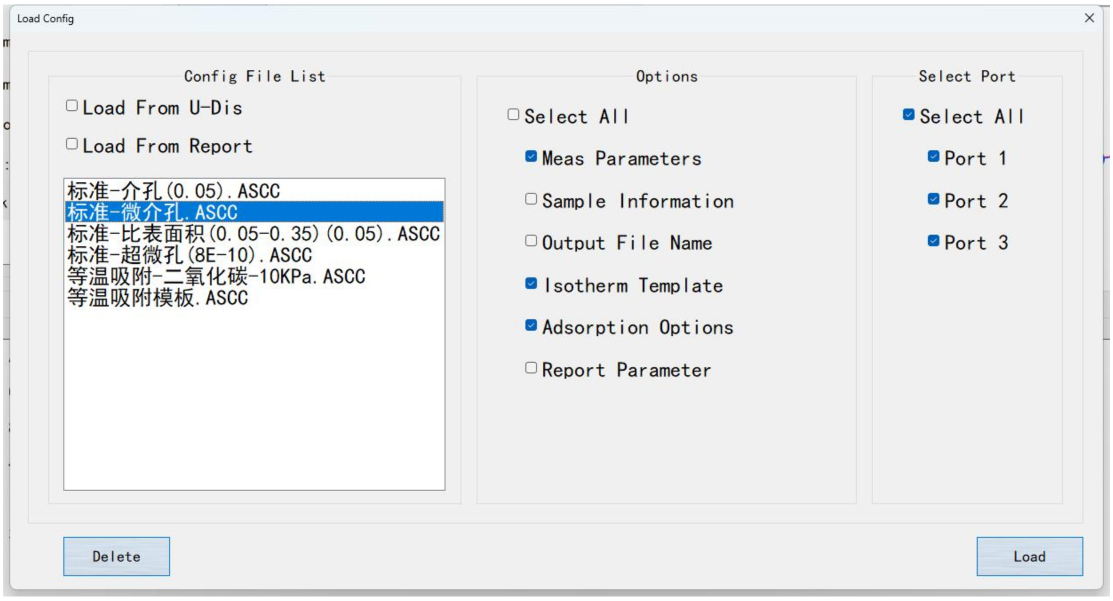
3 Port-3
Model: Super-Microporous
Name: MPDALI
Weight: 0.169700 [g]

Isotherm Template

Ads	Ads Part[P/Po]	Limit-X	Dose Gas[KPa]	Distance[P/Po]	D-Error	Smooth	Max-V	Equilibrium
	0.0E+00 -> 8.0E-10	√	0.010 -> 2.000	8.00E-10(1)	20.0%	×	Auto	Slope<0.050
	8.0E-10 -> 4.0E-09	√	0.010 -> 2.000	1.10E-09(3)	10.0%	×	Auto	Slope<0.050
	4.0E-09 -> 1.0E-08	√	0.010 -> 2.000	2.00E-09(3)	10.0%	×	Auto	Slope<0.050
	1.0E-08 -> 3.0E-08	√	0.010 -> 2.000	6.70E-09(3)	10.0%	×	Auto	Slope<0.050
	3.0E-08 -> 1.0E-07	√	0.010 -> 2.000	1.70E-08(4)	10.0%	×	Auto	Slope<0.050
	1.0E-07 -> 2.0E-07	√	0.010 -> 2.000	5.00E-08(2)	10.0%	×	Auto	Slope<0.050
	2.0E-07 -> 1.0E-06	√	0.020 -> 10.00	2.00E-07(4)	10.0%	×	Auto	Slope<0.050
	1.0E-06 -> 9.0E-06	√	0.020 -> 10.00	1.00E-06(8)	10.0%	×	Auto	Slope<0.050
	9.0E-06 -> 2.0E-05	√	0.003 -> 10.00	5.00E-06(2)	10.0%	×	Auto	Slope<0.050

Figure 3-5

（2） You can choose different configuration files, load parameters, and apply analysis parameters to various analysis stations. After setting up, click on the "Load" option in the bottom right corner to complete the loading. As shown in Figures 3-6



Config File List

☐ Load From U-Dis

☐ Load From Report

标准-介孔(0.05).ASCC
标准-微介孔.ASCC
标准-比表面积(0.05-0.35)(0.05).ASCC
标准-超微孔(8E-10).ASCC
等温吸附-二氧化碳-10KPa.ASCC
等温吸附模板.ASCC

Options

☐ Select All
☑ Meas Parameters
☐ Sample Information
☐ Output File Name
☑ Isotherm Template
☑ Adsorption Options
☐ Report Parameter

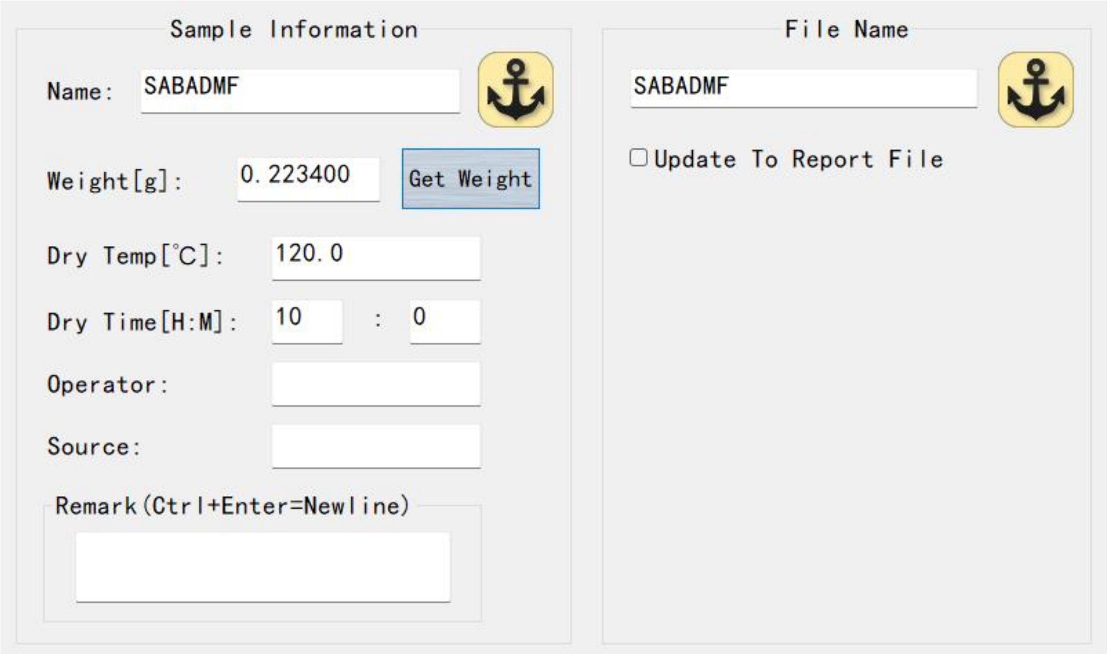
Select Port

☑ Select All
☑ Port 1
☑ Port 2
☑ Port 3

Figure 3-6

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

When saving the file name, it will be automatically saved according to the current test date. The sample name and file name cannot have special characters, such as:?/* # $!



Sample Information

Name: SABADMF

Weight[g]: 0.223400

Dry Temp[℃]: 120.0

Dry Time[H:M]: 10 : 0

Operator:

Source:

Remark(Ctrl+Enter=Newline)

File Name

SABADMF

☐ Update To Report File

Figure 3-7

Sample Information

Name: SABADMF

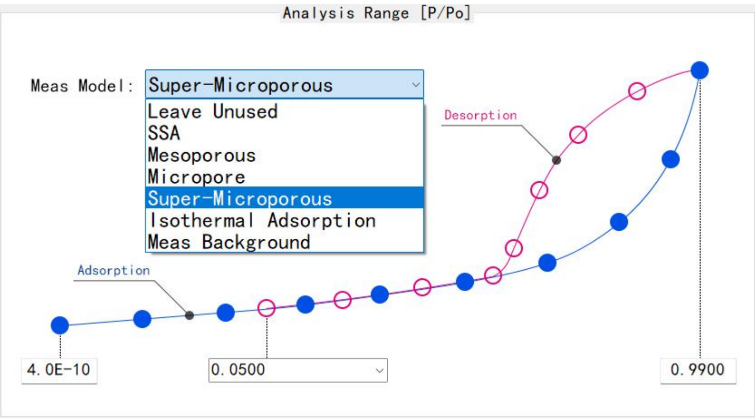
File Name

SABADMF

Figure 3-8

Select the Lock Key ⚓	**Unlock Key** ⚓
Lock key background turns yellow: file name matches sample name for storage	Lock key background turns blue: The file name can be modified arbitrarily, but it cannot have special characters, such as:?/* # $!

(4) Select the analysis type, as shown in Figure 3-9

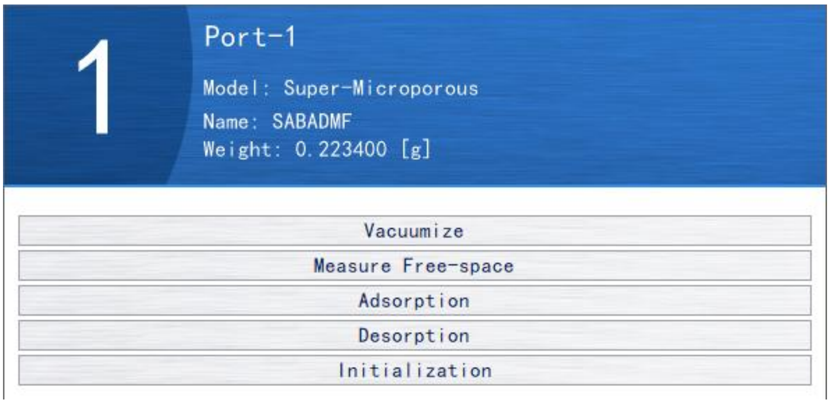


Analysis Range [P/Po]

Meas Model: Super-Microporous

Figure 3-9

(5) According to the above method, set the corresponding parameters for each analysis station that needs to work, click save and close to return to the main interface. After completing the preparation work, click the "Start" button to start.



1

Port-1

Model: Super-Microporous

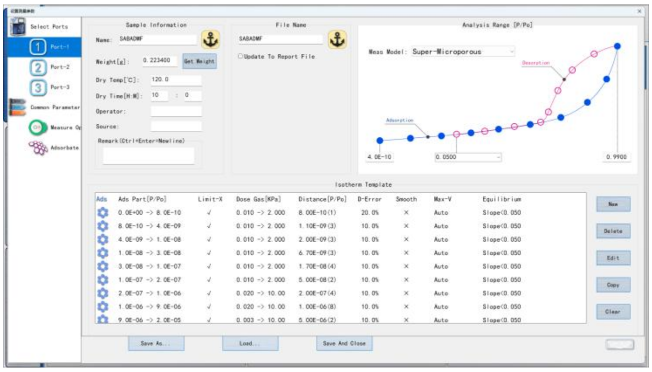
Name: SABADMF

Weight: 0.223400 [g]

Figure 3-10

2. Custom Template

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



Name: SABADMF

Weight[g]: 0. 223400

Dry Temp[℃]: 120. 0

Dry Time[H:M]: 10 : 0

Operator:

Source:

Remark(Ctrl+Enter=Newline)

File Name: SABADMF

☐Update To Report File

Meas Model: Super-Microporous

<table>
<tr>
<th>

Non Pressure Control Mode

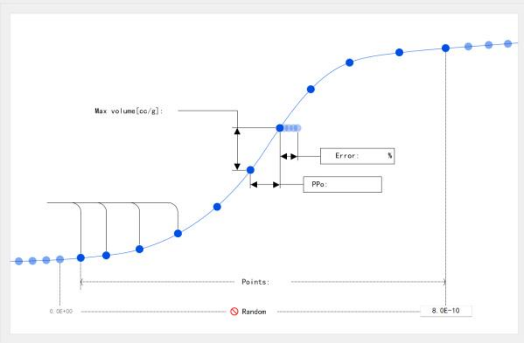
（ Fixed gas injection rate increase/decrease control ）

</th>
<th>

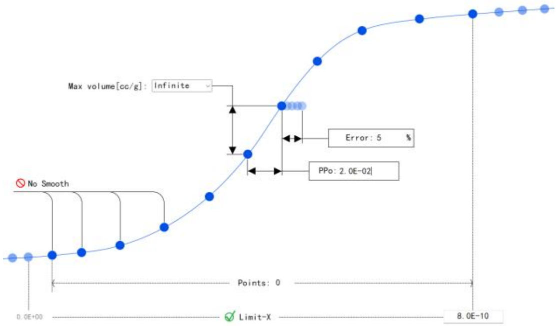
Pressure Control Mode

（ P/Po increase/decrease precise control ）

</th>
</tr>
<tr>
<td>



</td>
<td>

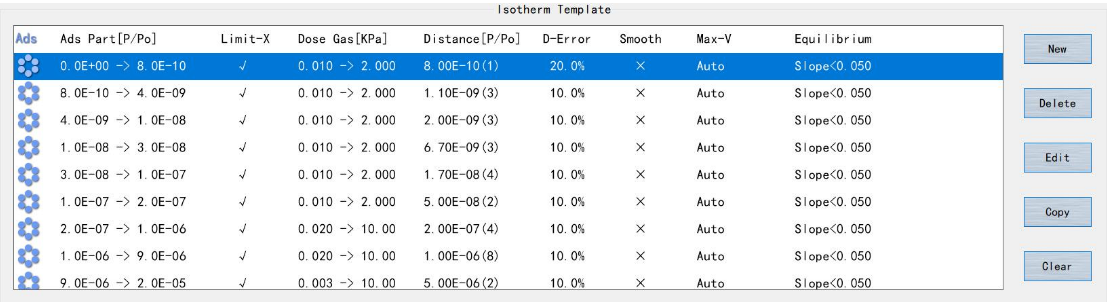


</td>
</tr>
<tr>
<td>Satisfy most material analysis</td>
<td>Customizable number of isotherm data points, precise P/Po interval</td>
</tr>
</table>

Figure 3-11

（2）Set the basic parameters and analysis type of the sample, referring to the system template settings above.

（3）Set up an isotherm collection template, which can add, modify, or delete segments based on the analysis type.



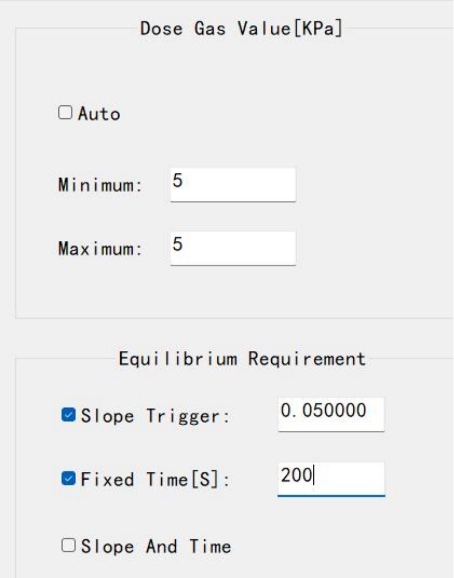
Ads	Ads Part[P/Po]	Limit-X	Dose Gas[KPa]	Distance[P/Po]	D-Error	Smooth	Max-V	Equilibrium
	0. 0E+00 -> 8. 0E-10	√	0. 010 -> 2. 000	8. 00E-10(1)	20. 0%	×	Auto	Slope<0. 050
	8. 0E-10 -> 4. 0E-09	√	0. 010 -> 2. 000	1. 10E-09(3)	10. 0%	×	Auto	Slope<0. 050
	4. 0E-09 -> 1. 0E-08	√	0. 010 -> 2. 000	2. 00E-09(3)	10. 0%	×	Auto	Slope<0. 050
	1. 0E-08 -> 3. 0E-08	√	0. 010 -> 2. 000	6. 70E-09(3)	10. 0%	×	Auto	Slope<0. 050
	3. 0E-08 -> 1. 0E-07	√	0. 010 -> 2. 000	1. 70E-08(4)	10. 0%	×	Auto	Slope<0. 050
	1. 0E-07 -> 2. 0E-07	√	0. 010 -> 2. 000	5. 00E-08(2)	10. 0%	×	Auto	Slope<0. 050
	2. 0E-07 -> 1. 0E-06	√	0. 020 -> 10. 00	2. 00E-07(4)	10. 0%	×	Auto	Slope<0. 050
	1. 0E-06 -> 9. 0E-06	√	0. 020 -> 10. 00	1. 00E-06(8)	10. 0%	×	Auto	Slope<0. 050
	9. 0E-06 -> 2. 0E-05	√	0. 003 -> 10. 00	5. 00E-06(2)	10. 0%	×	Auto	Slope<0. 050

Figure 3-12

① Double click on any isotherm segment to set adsorption/desorption parameters. There are two testing modes provided here to meet the testing needs of different materials.

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

According to the adsorption performance characteristics of target materials, the gas injection rate is customized. Generally, the initial gas injection rate is set to 5KPa, and the maximum gas injection rate is not more than 15KPa. For special samples such as aerogel, the gas injection rate can be appropriately increased.



Dose Gas Value[KPa]

☐ Auto

Minimum: 5

Maximum: 5

Equilibrium Requirement

☑ Slope Trigger: 0.050000

☑ Fixed Time[S]: 200

☐ Slope And Time

Figure 3-13

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

Figure 3-14

- Enter the P/Po end point (circled in red), as shown in "1.0E+00" in the figure. After entering the number of points (circled in green), "16", the P/Po interval will be automatically calculated.
- Enter the P/Po end point (circled in red), as shown in "1.0E+00" in the figure. After entering the interval (circled in yellow) P "5.0E-02", the number of points will be automatically calculated.
- Check the automatic setting of gas injection volume and automatic slope control

Dose Gas Value[KPa]

☑ Auto

Minimum: 5

Maximum: 5

Equilibrium Requirement

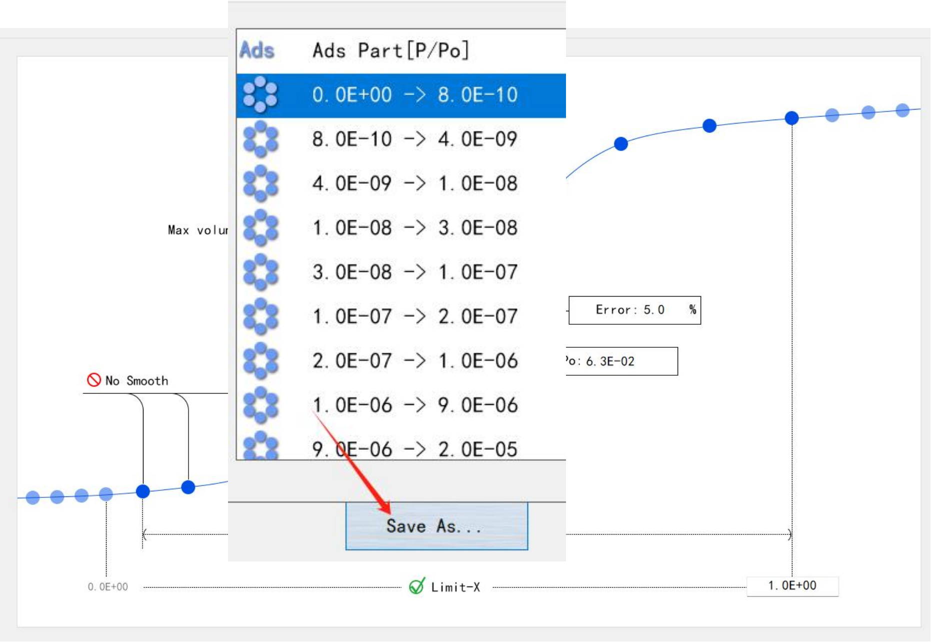
☑ Slope Trigger: 0.050000

☐ Fixed Time[S]: 200

☐ Slope And Time

- As above, complete and save the selected segment settings

（4）Save the custom template. Choose Save As to store the custom template.

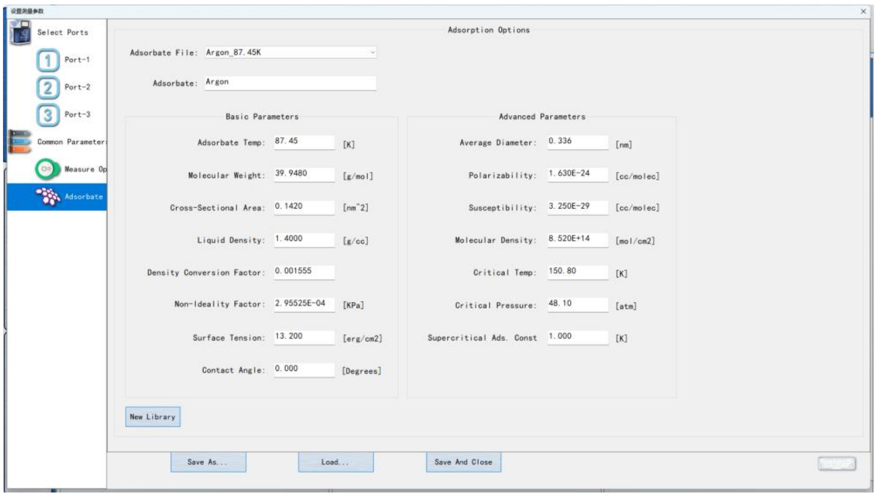


Ads Part[P/Po]

0.0E+00 -> 8.0E-10
8.0E-10 -> 4.0E-09
4.0E-09 -> 1.0E-08
1.0E-08 -> 3.0E-08
3.0E-08 -> 1.0E-07
1.0E-07 -> 2.0E-07
2.0E-07 -> 1.0E-06
1.0E-06 -> 9.0E-06
9.0E-06 -> 2.0E-05

Figure 3-15

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



Adsorbate File: Argon_87.45K

Adsorbate: Argon

Adsorbate Temp: 87.45 [K]

Molecular Weight: 39.9480 [g/mol]

Cross-Sectional Area: 0.1420 [nm^2]

Liquid Density: 1.4000 [g/cc]

Density Conversion Factor: 0.001555

Non-Ideality Factor: 2.95525E-04 [KPa]

Surface Tension: 13.200 [erg/cm2]

Contact Angle: 0.000 [Degrees]

Average Diameter: 0.336 [nm]

Polarizability: 1.630E-24 [cc/molec]

Susceptibility: 3.250E-29 [cc/molec]

Molecular Density: 8.520E+14 [mol/cm2]

Critical Temp: 150.80 [K]

Critical Pressure: 48.10 [atm]

Supercritical Ads. Const 1.000 [K]

Figure 3-16

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

3. Add Liquid Nitrogen to the Dewar Bottle

（1） If conditions permit, wear protective equipment, protective goggles, and insulated gloves.

（2）When adding liquid nitrogen to a Dewar bottle, it should be slowly added to reduce the "thermal shock" of the bottle and prevent liquid nitrogen splashing, so that the height of the liquid nitrogen level in the liquid nitrogen cup is as shown in Figure 3-17. **Be careful when adding liquid nitrogen to prevent low-temperature burns.**

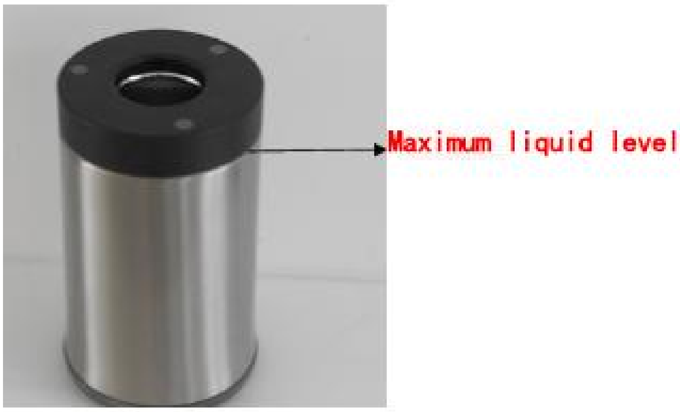


Figure 3-17

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

(3) Starting Analysis

1. Open Gas Cylinder

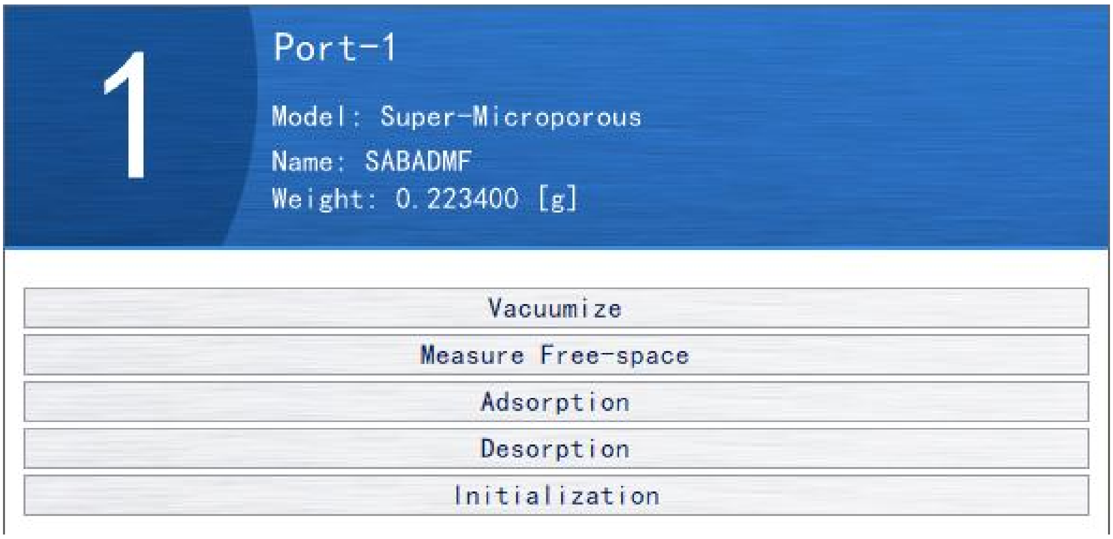
Open the main valve of the helium and nitrogen steel cylinder, and the output pressure of the pressure reducing valve is 0.3MPa. **Note that when the output pressure of the pressure reducing valve is higher than 0.5MPa, the gas pipeline should be removed while the total pressure valve of the gas cylinder is closed, and the gas in the pressure reducing valve should be completely discharged. Then, the gas pipeline should be reinstalled and the partial pressure should be readjusted (the output pressure should be 0.3-0.5MPa).**

2. Turn on the Vacuum Pump

Turn on the power switch of the mechanical pump.

3. Restarting Analysis

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



1

Port-1

Model: Super-Microporous

Name: SABADMF

Weight: 0.223400 [g]

Figure 3-18

(4) End Analysis

1. End Operation

After completing the analysis, the default location for saving data is:

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

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

(1) Close the total pressure valve of the gas cylinder. If the gas is still in use and the gas circuit is not removed, the pressure valve does not need to be closed.

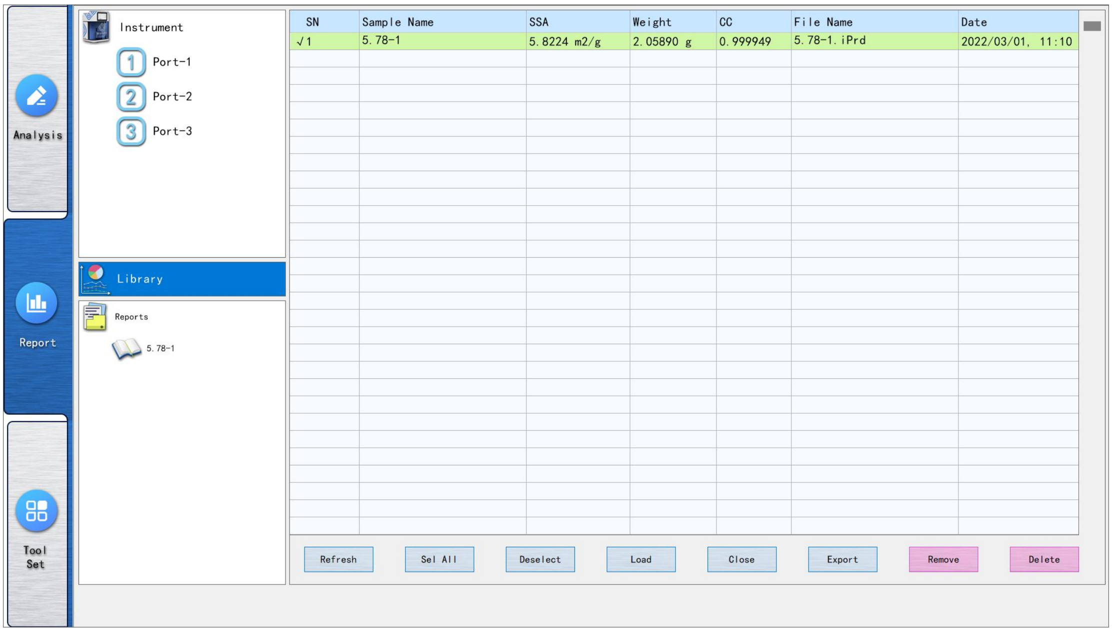
（2） Turn off the power of the instrument host and recover the remaining liquid nitrogen (discard the ice residue).

（3） Turn off the vacuum pump.

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

2. Viewing Test Reports on the Computer Control end

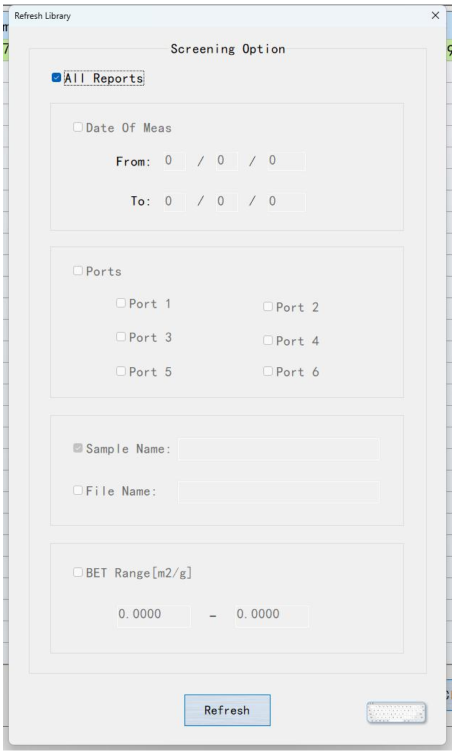
（1） In the report tool group, click "Report Library", select the target report, click "Load Report", and then click "Load Report", as shown in Figure 3-19.



SN	Sample Name	SSA	Weight	CC	File Name	Date
√1	5.78-1	5.8224 m2/g	2.05890 g	0.999949	5.78-1.iPrd	2022/03/01, 11:10

Figure 3-19

（2） You can select and filter target reports based on different factors such as analysis station/testing date/sample name/file name. As shown in Figure 3-20



Refresh Library

Screening Option

☑ All Reports

☐ Date Of Meas

From: 0 / 0 / 0

To: 0 / 0 / 0

☐ Ports

☐ Port 1 ☐ Port 2

☐ Port 3 ☐ Port 4

☐ Port 5 ☐ Port 6

☑ Sample Name:

☐ File Name:

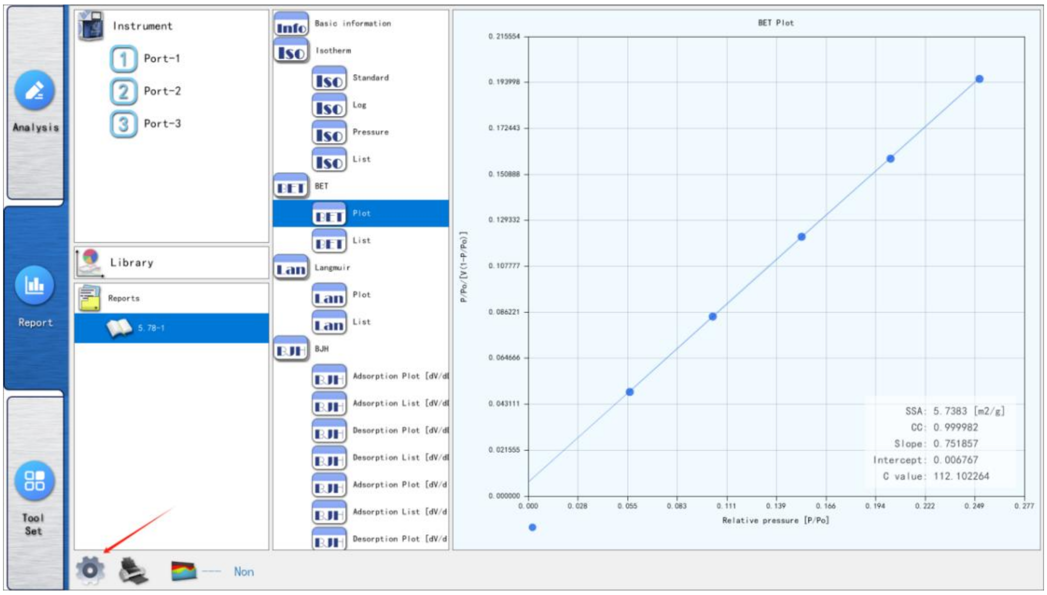
☐ BET Range[m2/g]

0.0000 – 0.0000

Figure 3-20

(3) In the loaded report interface, you can view the results of each model. If you need to modify model parameters, you can click the settings button to enter the modification process. As shown in Figure 3-21

Tip: This step can be ignored. Follow "Chapter 4 Data Management" and use data processing software to process the data.



SSA: 5.7383 [m2/g]
CC: 0.999982
Slope: 0.751857
Intercept: 0.006767
C value: 112.102264

Figure 3-21

Chapter 4 Data Management

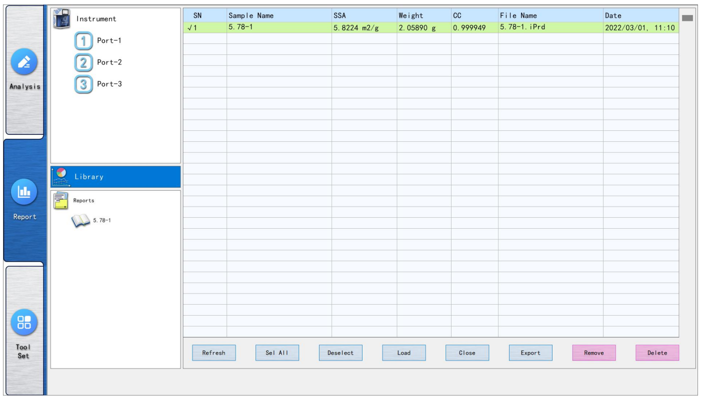
(1) Data Export

1. Create a new Target Folder in the USB Drive

Before exporting, first create a folder named "iPoreReport iPoreReport " in the USB drive where the copied data is located, and insert the USB drive into the instrument's USB interface.

2. Export Source Files under the Report Module

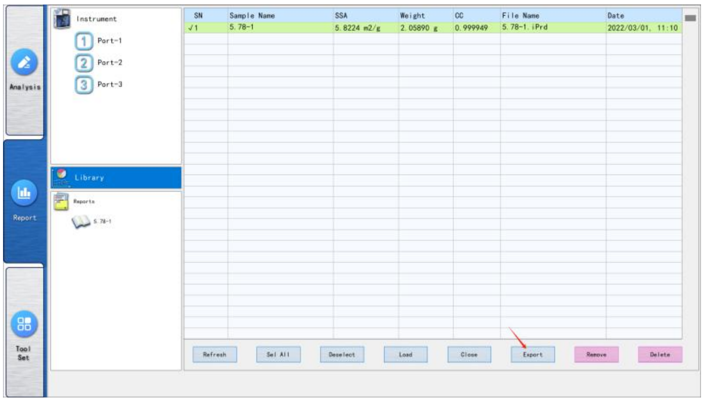
(1) Click on the report module to open the report library (as shown in Figure 4-1).



SN	Sample Name	SSA	Weight	CC	File Name	Date
√1	5.78-1	5.8224 m2/g	2.05890 g	0.999949	5.78-1.iPrd	2022/03/01, 11:10

Figure 4-1

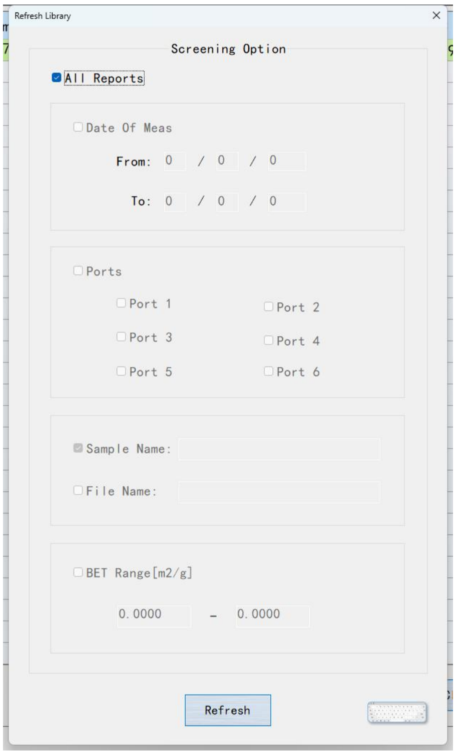
(2) You can select single or multiple library files. Then click on "Export Report". As shown in Figure 4-2



SN	Sample Name	SSA	Weight	CC	File Name	Date
√1	5.78-1	5.8224 m2/g	2.05890 g	0.999949	5.78-1.iPrd	2022/03/01, 11:10

Figure 4-2

(3) You can filter the required target files from the database by date, analysis station, sample name, file name, etc. After successfully exporting the source file, there will be relevant prompts.



Refresh Library

Screening Option

☑ All Reports

☐ Date Of Meas

From: 0 / 0 / 0

To: 0 / 0 / 0

☐ Ports

☐ Port 1 ☐ Port 2

☐ Port 3 ☐ Port 4

☐ Port 5 ☐ Port 6

☐ Sample Name:

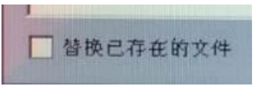
☐ File Name:

☐ BET Range[m2/g]

0.0000 – 0.0000

Figure 4-3

(4) Solution to file export failure.

 错误！ 找不到目标文件夹 确定	 错误！ OK 导出文件失败！！！
There is no new folder named "iPoreReport" in the USB drive or the file name is incorrect. Simply create or modify it again.	There are source files with the same file name in the USB drive. Modify the file name in the USB drive or check "Replace existing files"  ☐ 替换已存在的文件

(2) Data Extraction

1. Open Data Processing Software

（1）Double click on the desktop analysis software shortcut icon to open the software interface. Figure 4-4

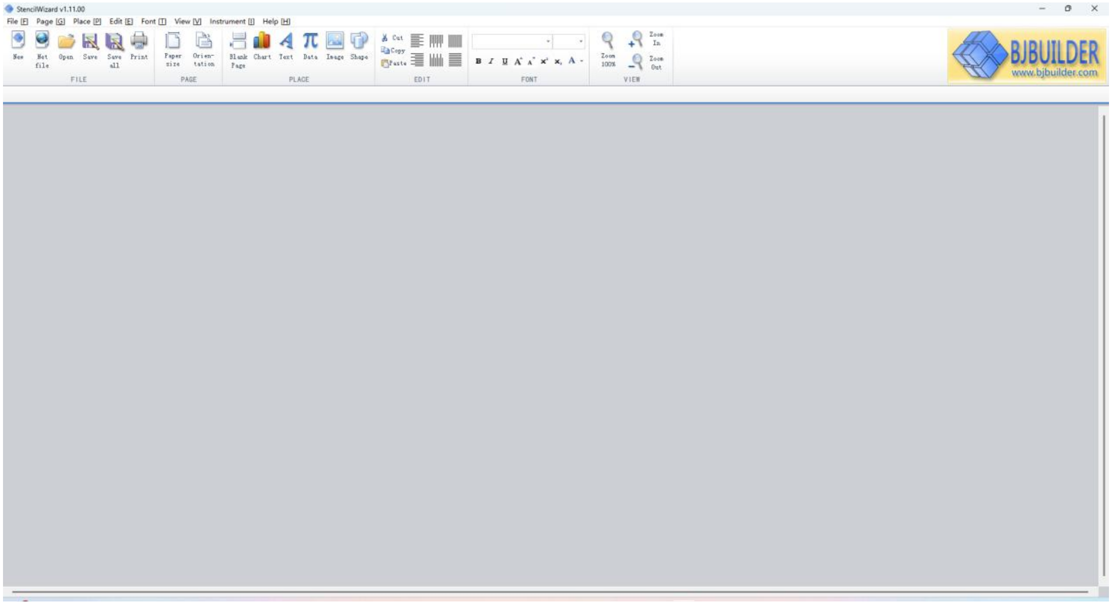
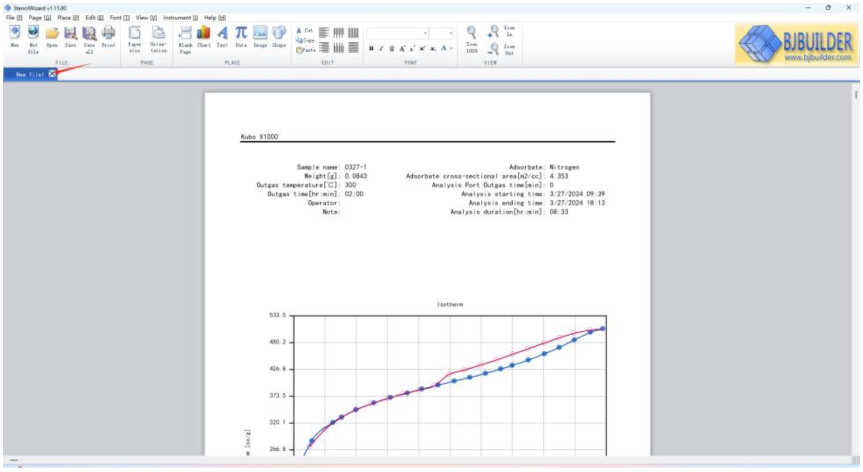


Figure 4-4

（2） Menu bar introduction

Figure 4-5

- New: Retrieve the newly generated test file, and open and edit it; You can choose the "New" shortcut button or choose File - New
- Open: Retrieve the edited file, and open and re edit it; You can choose the "New" shortcut button or choose File - Open
- Network creation: Implementing remote Internet control through fixed IP settings
- Save: Save the currently edited file as a new file, which can be saved in ". MFAR", ". RAW", or "Excel" formats
- Save All: sequentially and consecutively save the currently edited file as a new file
- Close: Close the current file, as shown in Figure 4-6



Kubo X1000

Sample name: 0327-1
Weight[g]: 0.0843
Outgas temperature[°C]: 300
Outgas time[hr:min]: 02:00
Operator:
Note:

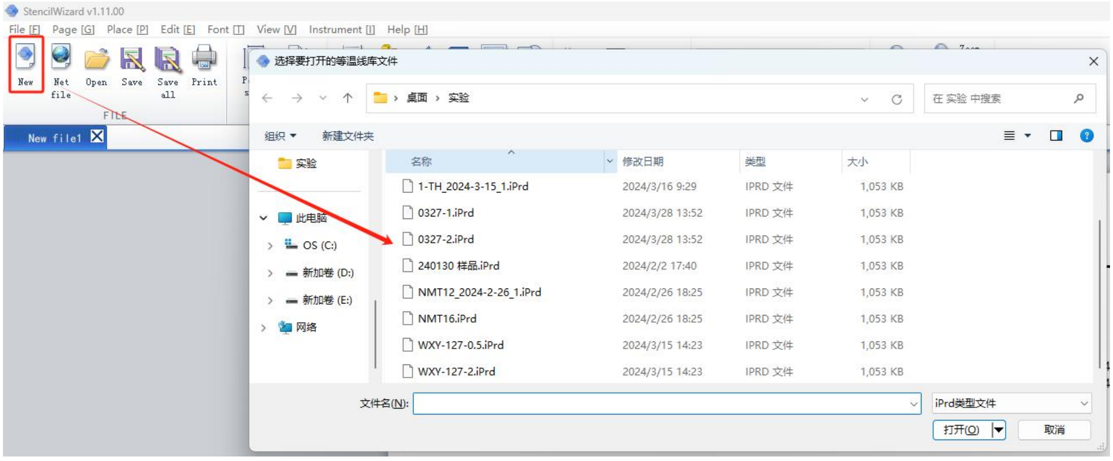
Adsorbate: Nitrogen
Adsorbate cross-sectional area[m2/cc]: 4.353
Analysis Port Outgas time[min]: 0
Analysis starting time: 3/27/2024 09:39
Analysis ending time: 3/27/2024 18:13
Analysis duration[hr:min]: 08:33

Figure 4-6

- Printing: allows you to print table data, charts, or entire reports
- Page: Set paper size and orientation

2. Open Target Source File

Click the "New" button on the software, find the "iPoreReport" folder in the USB drive and open it. Double click the source file that needs to be processed with the left mouse button, as shown in Figure 4-7 of "XXX. iprd".



名称	修改日期	类型	大小
1-TH_2024-3-15_1.iPrd	2024/3/16 9:29	IPRD 文件	1,053 KB
0327-1.iPrd	2024/3/28 13:52	IPRD 文件	1,053 KB
0327-2.iPrd	2024/3/28 13:52	IPRD 文件	1,053 KB
240130 样品.iPrd	2024/2/2 17:40	IPRD 文件	1,053 KB
NMT12_2024-2-26_1.iPrd	2024/2/26 18:25	IPRD 文件	1,053 KB
NMT16.iPrd	2024/2/26 18:25	IPRD 文件	1,053 KB
WXY-127-0.5.iPrd	2024/3/15 14:23	IPRD 文件	1,053 KB
WXY-127-2.iPrd	2024/3/15 14:23	IPRD 文件	1,053 KB

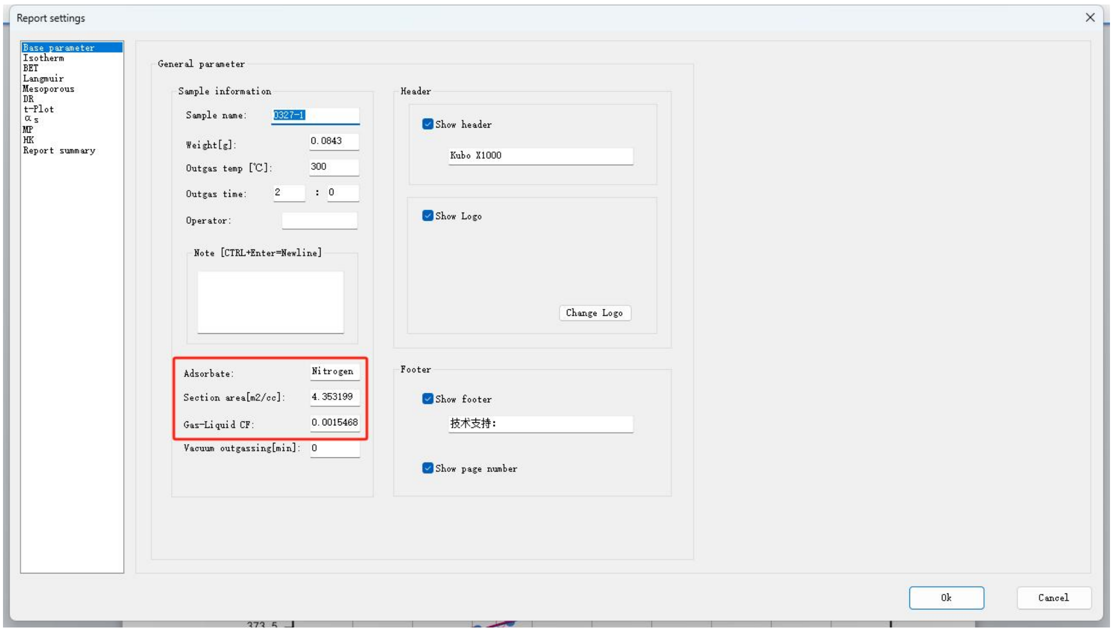
Figure 4-7

（3） Data Editing

After clicking "New" to import the source file, the report settings interface will automatically open. At this point, you will enter the "Basic Parameters" window in the report settings. Here, you can switch between different analysis models, edit all types of sample parameters in the physical adsorption instrument, choose to generate analysis models, and so on. After setting up the models for each window, please click "OK". Otherwise, you will need to edit them again.

1. Basic Parameter Window

Here, you can edit the basic parameters of the sample, edit the header and footer, and so on. When using the low-temperature nitrogen adsorption method by default for testing, do not modify the three parameters of "adsorbate, adsorbate area, and gas-liquid volume conversion coefficient". As shown in Figure 4-8



Sample name: 0327-1
Weight[g]: 0.0843
Outgas temp [℃]: 300
Outgas time: 2 : 0
Operator:
Adsorbate: Nitrogen
Section area[m2/cc]: 4.353199
Gas-Liquid CF: 0.0015468
Vacuum outgassing[min]: 0
☑ Show header
Kubo X1000
☑ Show Logo
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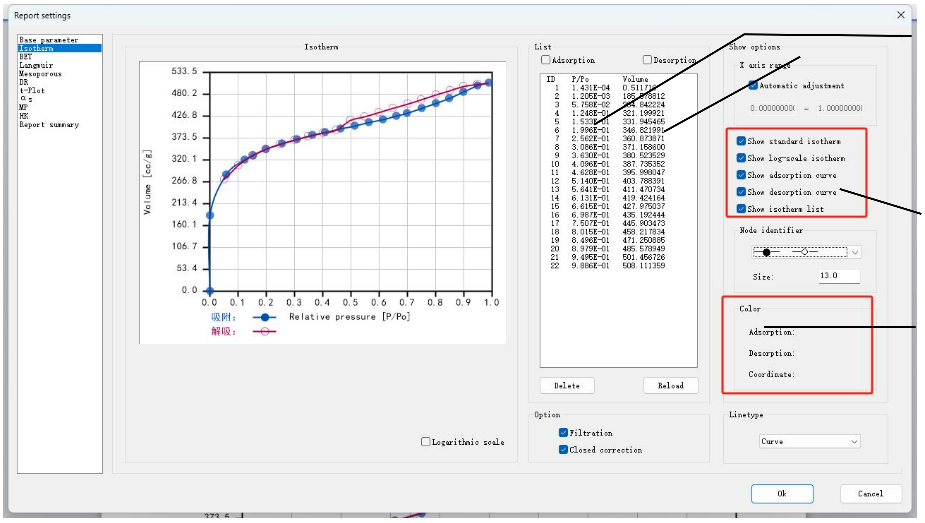
技术支持:
☑ Show page number

Figure 4-8

2. Isotherm window

You can view the adsorption and analytical node values on the isotherm; Edit isotherm coordinates, adsorption, and analyze linear colors; Generate or remove isotherm plots; Selecting the isotherm linear connection line can also filter isotherm adsorption and analysis data nodes and generate new isotherm graphs. Figure 4-9



List

☐ Adsorption ☐ Desorption

ID	P/Po	Volume
1	1.431E-04	0.511710
2	1.205E-03	185.[illegible]78812
3	5.758E-02	284.842224
4	1.248E-01	321.199921
5	1.533E-01	331.945465
6	1.996E-01	346.821991
7	2.562E-01	360.873871
8	3.086E-01	371.158600
9	3.630E-01	380.523529
10	4.096E-01	387.735352
11	4.628E-01	395.998047
12	5.140E-01	403.788391
13	5.641E-01	411.470734
14	6.131E-01	419.424164
15	6.615E-01	427.975037
16	6.987E-01	435.192444
17	7.507E-01	445.903473
18	8.015E-01	458.217834
19	8.496E-01	471.250885
20	8.979E-01	485.578949
21	9.495E-01	501.456726
22	9.886E-01	508.111359

☐ Logarithmic scale

Option

☑ Filtration

☑ Closed correction

Show options

X axis range

☑ Automatic adjustment

0.000000000 – 1.000000000

☑ Show standard isotherm

☑ Show log-scale isotherm

☑ Show adsorption curve

☑ Show desorption curve

☑ Show isotherm list

Node identifier

Size: 13.0

Color

Adsorption:

Desorption:

Coordinate:

Linetype

Curve

Display the coordinates of adsorption or analysis points

Do you want to display the generated report

Custom coordinates and node colors

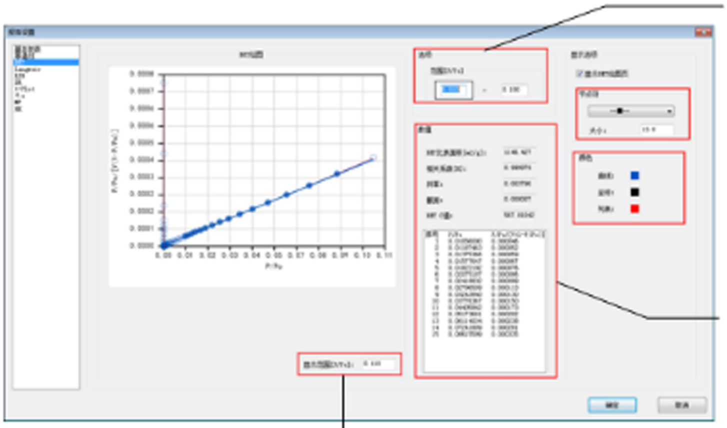
Figure 4-9

（1）Horizontal axis of isotherm: relative pressure [P/Po], where P is the actual test pressure; Po is the saturated vapor pressure of liquid nitrogen

（2）Isotherm ordinate: adsorption capacity of material per unit mass [cc/g]

3. BET Drawing Window

Editable point range, node symbols, and line colors. The point range varies for different materials. Please select points based on the actual situation of the materials. Do not choose P/Po that is too high or too low, as it may cause significant deviation in the calculation results. Figure 4-10



Set the start and end points of P/P0

√Displays the data points in the current fetch point range

√The BET result and linear coefficient are displayed

The actual node in the current fetch point range

Figure 4-10

（1）P/Po within the Range of 0 to 0.15

Measurement and analysis of micropore distribution can be carried out. For ultra microporous and microporous samples, the value range of P/Po is 0~0.1, and a vacuum condition of 3 × 10-8pa and multiple high-precision pressure sensors with different ranges are required to ensure the number of data points and accuracy within this range.

（2）P/Po within the Range of 0.05 to 0.30

Multi point BET can be used for testing and calculation of specific surface area, which is suitable for sample characterization under multi-layer adsorption theory. For characterization under single-layer adsorption theory, Laughmuir specific surface area data can be used.

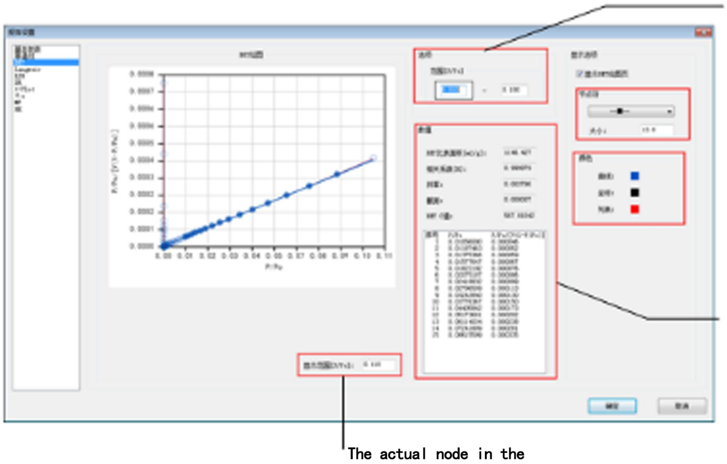
P/Po between 0.05 and 0.30 is only applicable to mesoporous materials. For samples with micropores, the value range needs to be adjusted appropriately. For X molecular sieve, the value range is 0.005 to 0.01; Take 0.005-0.1 for microporous materials; Take 0.01-0.2 for mesoporous composite materials; Due to the closer monolayer adsorption characteristics of microporous materials, the specific surface area data of Laughmuir can more accurately characterize microporous materials.

（3）P/Po within the Range of 0.15 to 0.996

Measurement and analysis of mesopores and macropores show that within this relative pressure range, capillary condensation occurs on the surface of adsorbent pores, and the obvious feature on the adsorption desorption isotherm is the formation of a hysteresis loop.

4. Langmuir drawing window

The editable Langmuir algorithm takes point ranges, node symbols, and line colors, making Langmuir more suitable for characterizing microporous materials. Figure 4-11



Set the start and end points of P/P0

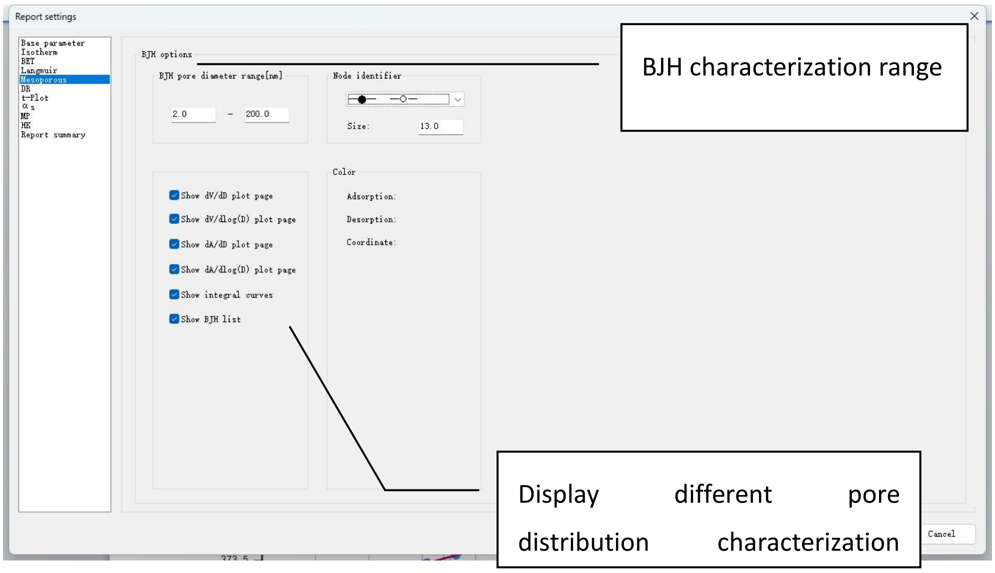
√Displays the data points in the current fetch point range
√The BET result and linear coefficient are displayed

The actual node in the current fetch point range

Figure 4-11

5. BJH Selection Window

- BJH can only be used for mesopores, and cylindrical models are used in theoretical calculations.
- Pores are rigid and have a regular shape (such as cylindrical or slit), with a narrow pore size distribution and a clear range (i.e. H1 hysteresis loop); There are no micropores or large pores (which is a clear type IV isotherm).
- When the aperture is less than 10nm, the aperture is underestimated, and there is a 20% error when the aperture is less than 5nm.



Report settings

BJH options

BJH pore diameter range[nm]: 2.0 – 200.0

Node identifier

Size: 13.0

Color

Adsorption:

Desorption:

Coordinate:

☑ Show dV/dD plot page

☑ Show dV/dlog(D) plot page

☑ Show dA/dD plot page

☑ Show dA/dlog(D) plot page

☑ Show integral curves

☑ Show BJH list

BJH characterization range

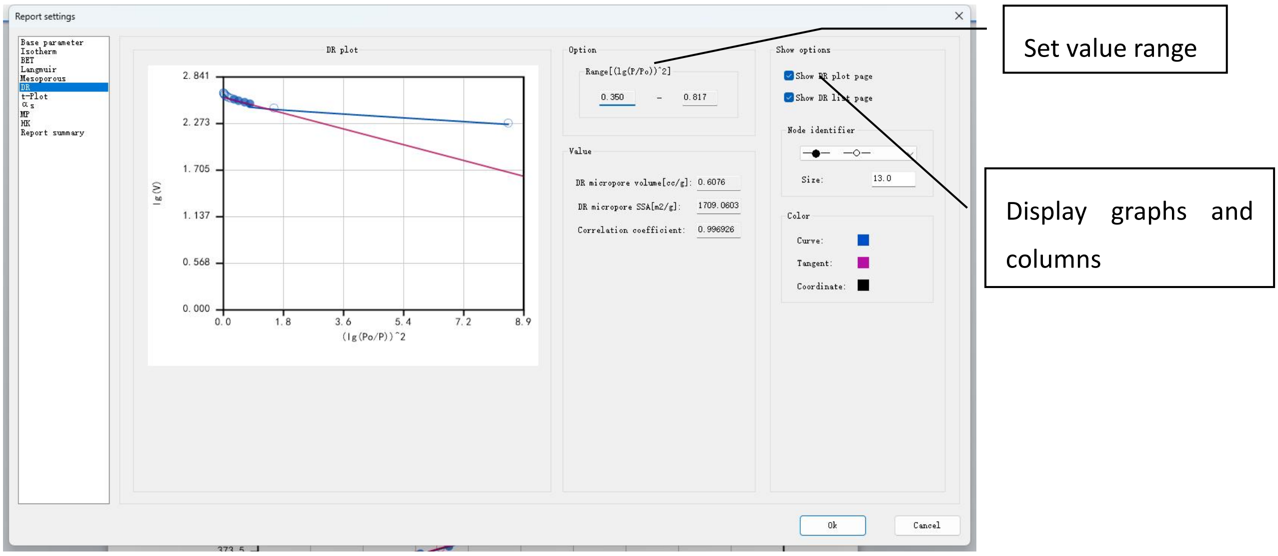
Display different pore distribution characterization

Figure 4-12

6. DR (Dubin Radushkevich) window

The DR filling theory suggests that molecules are not adsorbed layer by layer on the pore wall, but rather undergo varying degrees of volume filling.

It is suitable for calculating the pore volume of microporous materials such as microporous activated carbon and molecular sieve. It is a method developed based on the research of microporous porosity of activated carbon and can also be used to analyze other microporous materials.



Range[(lg(P/Po))^2] 0. 350 – 0. 817

DR micropore volume[cc/g]: 0. 6076

DR micropore SSA[m2/g]: 1709. 0603

Correlation coefficient: 0. 996926

☑ Show DR plot page

☑ Show DR list page

Size: 13. 0

Curve: 🟦

Tangent: 🟪

Coordinate: ⬛

Set value range

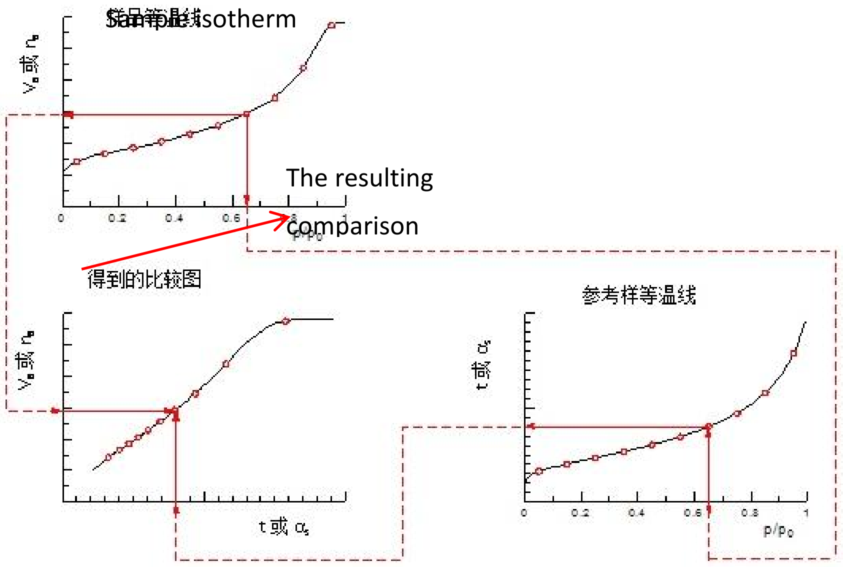
Display graphs and columns

Figure 4-13

7. T-plot/t graphical window

Assumption: The surface has uniform chemical properties and no strong specific adsorption sites. In order to estimate the presence of micropores, mesopores, or both in the test sample (mainly used to characterize the presence of micropores), the adsorption isotherm extrapolation method is often used to estimate the micropore area, micropore volume, and outer surface area. The two main methods are t-plot method and α s-plot method, and t-plot method is the most commonly used. The specific method is to use the adsorption layer thickness t and adsorption capacity V to plot and compare the V-t curve of non porous adsorbents.

It compares the experimental isotherm with the reference isotherm obtained from non porous solids. The reference isotherm is calculated using mathematical expressions, where the adsorbent must have chemical properties similar to the test sample. Therefore, for different samples, non porous reference curves, i.e. different models, should be selected. For example, for high silica zeolites, a reference curve of non porous silica should be used.



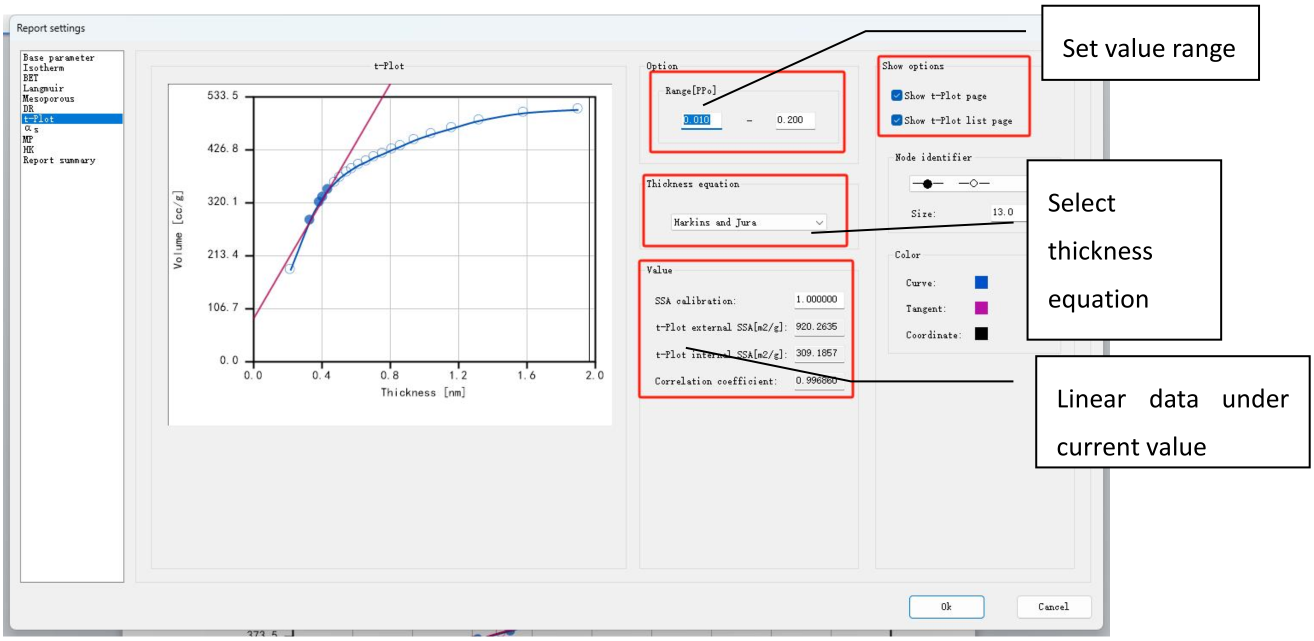
Reference sample isotherm

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

After selecting the model and calculation points, the instrument can provide the calculated values and graphs. But whether this result is reasonable and reliable is often the most easily overlooked and common analytical error. Principles for judging results:

A、The regression line is tangent to the t-curve rather than intersecting. If they intersect, the pressure value point needs to be adjusted and recalculated to meet the above requirements.

B、Linear regression coefficients of at least 3 9 (>0.999)



Set value range

Option
Range[PPo] 0.010 – 0.200

Show options
☑ Show t-Plot page
☑ Show t-Plot list page

Thickness equation
Harkins and Jura

Select thickness equation

Value
SSA calibration: 1.000000
t-Plot external SSA[m2/g]: 920.2635
t-Plot internal SSA[m2/g]: 309.1857
Correlation coefficient: 0.996860

Size: 13.0

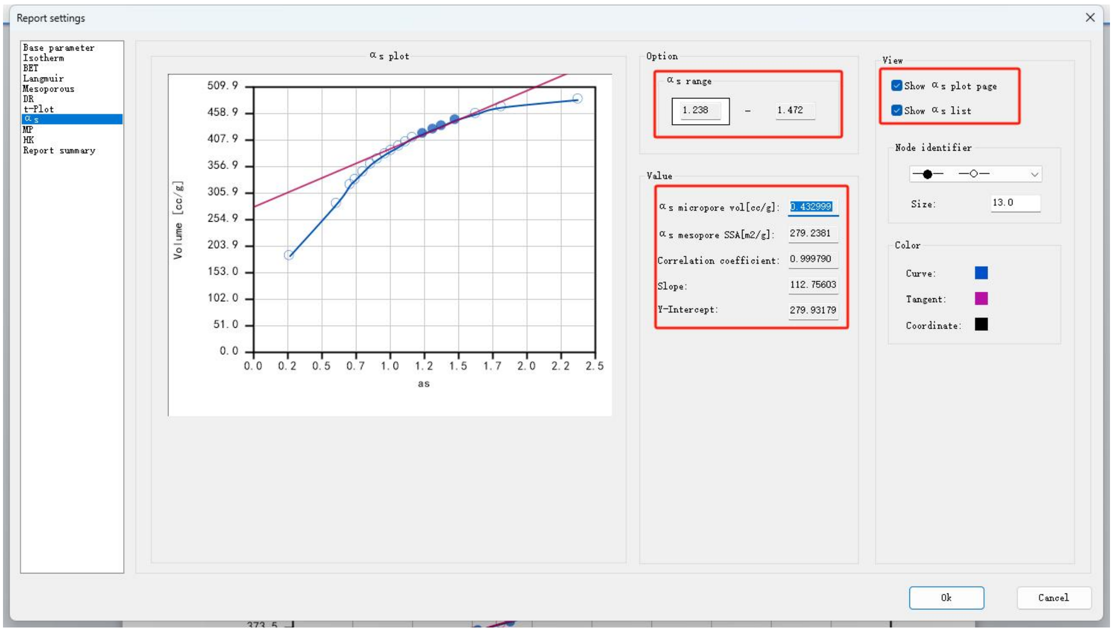
Linear data under current value

Figure 4-14

✶ Thickness equation:

A、Halsey: Similar to the application of the de Boer model, it is used as a computational model for molecular sieves and oxides, with a pressure point value calculation range of 0.4-0.6 (P/P0)

8. As image window



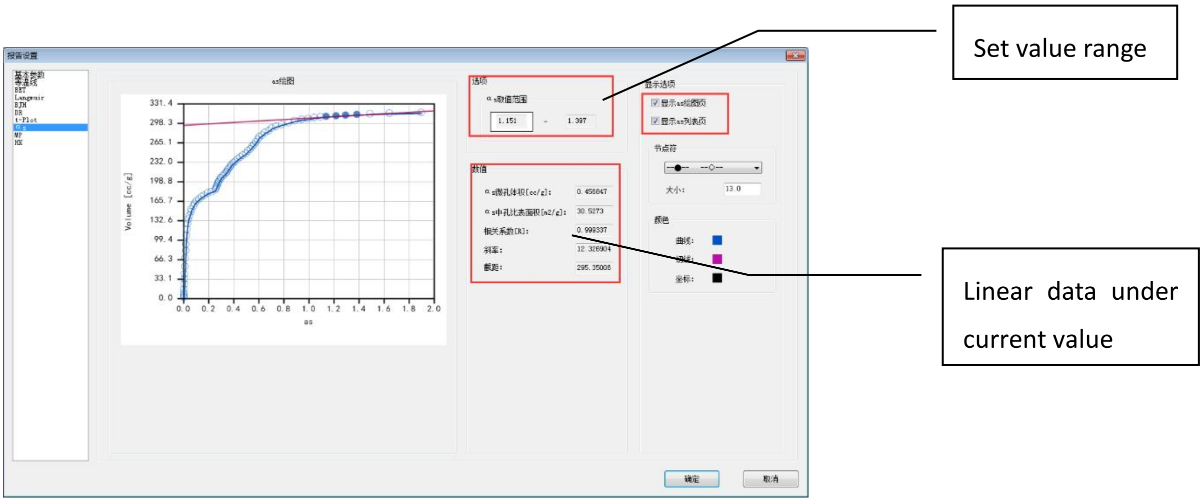
Option
α s range 1.238 – 1.472

View
☑ Show α s plot page
☑ Show α s list

Value
α s micropore vol[cc/g]: 0.432999
α s mesopore SSA[m2/g]: 279.2381
Correlation coefficient: 0.999790
Slope: 112.75603
Y-Intercept: 279.93179

Size: 13.0

At p/p0=0.4, the corresponding adsorption amount V0.4 is derived from the as calculation company, which is α s=V/V0.4. Similar to the t-plot method, the difference is that the V-alpha s plot is used instead of the V-t plot, which can be used even if the thickness of the adsorption layer is not known, and can also determine adsorbates other than nitrogen.



α s取值范围 1.151 - 1.397

☑ 显示as绘图页

☑ 显示as列表页

α s微孔体积[cc/g]: 0.458847

α s中孔比表面积[m2/g]: 30.5273

相关系数[R]: 0.999337

斜率: 12.326904

截距: 295.35008

大小: 13.0

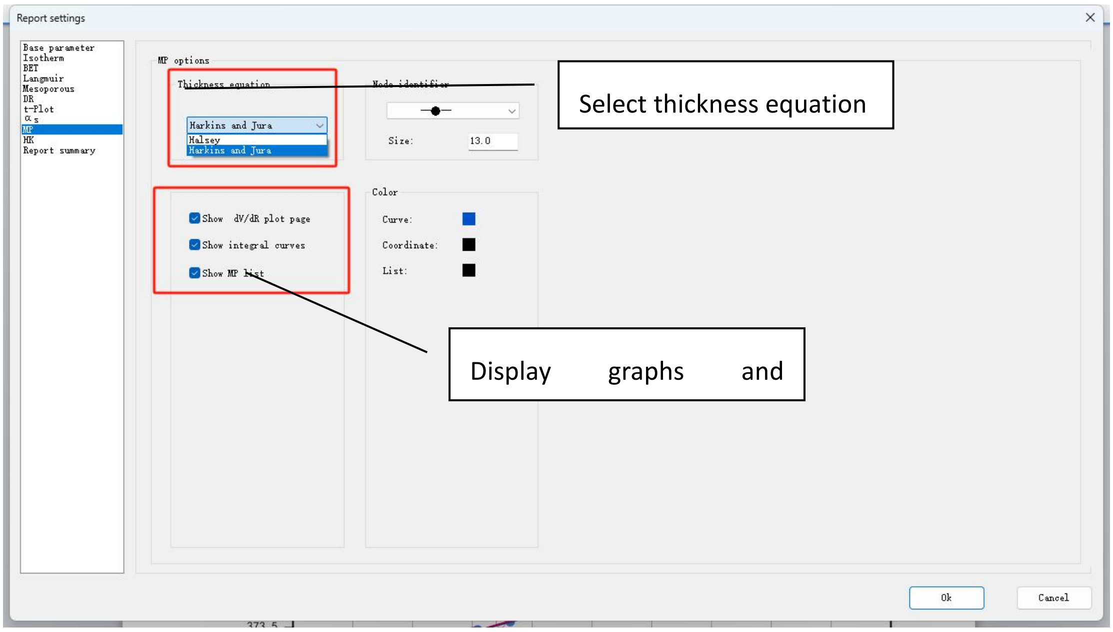
Set value range

Linear data under current value

Figure 4-15

9. MP Graph Window

The MP method obtains information on the surface area, micropore volume, and micropore distribution of microporous adsorbents by analyzing the V-t curve.



Thickness equation: Harkins and Jura

Halsey

Harkins and Jura

Size: 13.0

☑ Show dV/dR plot page

☑ Show integral curves

☑ Show MP list

Select thickness equation

Display graphs and

Figure 4-16

10. HK Graph Window

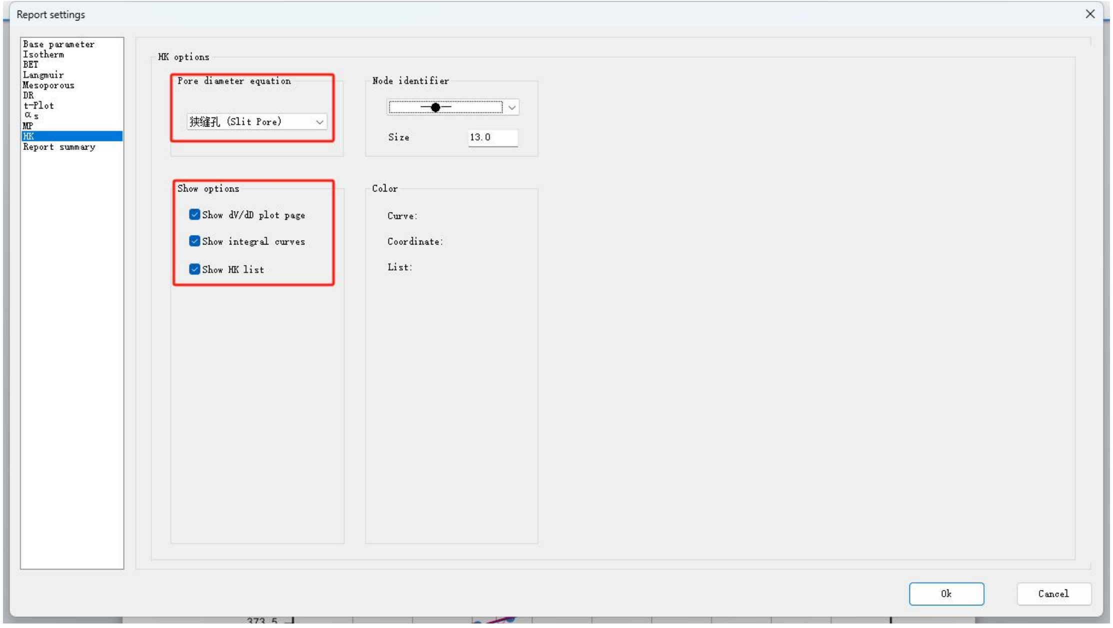
The HK microporous model (including Saito&Foley correction, Cheng&Yang correction) is used for microporous pore size distribution, including the following three:

Horvath-Kawazoe(HK)模型: Assuming the pore type is a narrow slit type micropore. Mainly aimed at nitrogen adsorption in the narrow pores of carbon molecular sieves and activated carbon.

Saito-Foley (SF)模型: Assuming the hole type is cylindrical. Saito and Foley extended the HK method to calculate the effective pore size distribution through adsorption isotherms on cylindrical pores of zeolite molecular sieves at argon 87K.

Cheng/Yang 模型: Spherical microporous distribution model. Can be used for spherical pores of octahedral zeolite and 5A molecular sieve.

The HK and improved HK equations consider the interaction between adsorbate and adsorbent, but this method is greatly influenced by the choice of pore model and the values of physical parameters in the formula. Therefore, when using, it is necessary to choose the pore model and equation parameters reasonably based on the type of adsorbent and microporous sample.



Pore diameter equation: 狭缝孔 (Slit Pore)

Size: 13.0

Show options

☑ Show dV/dD plot page

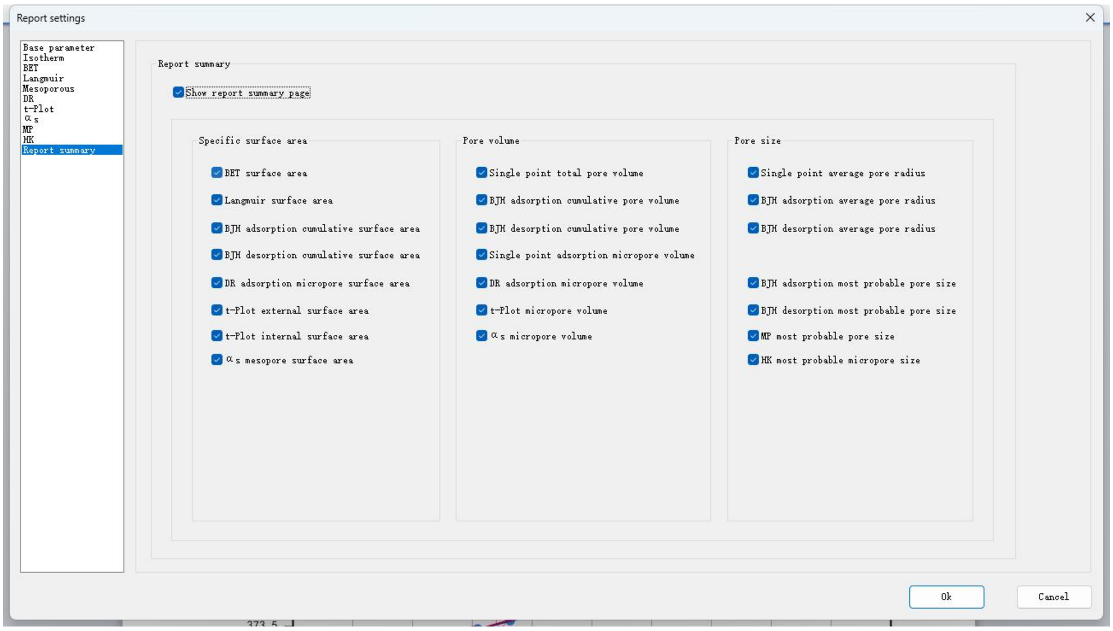
☑ Show integral curves

☑ Show HK list

Figure 4-17

11. Data Digest

Here, the displayed results can be selectively generated, and once checked, they can be generated. Figure 4-18



Report summary

☑ Show report summary page

Specific surface area

☑ BET surface area
☑ Langmuir surface area
☑ BJH adsorption cumulative surface area
☑ BJH desorption cumulative surface area
☑ DR adsorption micropore surface area
☑ t-Plot external surface area
☑ t-Plot internal surface area
☑ α s mesopore surface area

Pore volume

☑ Single point total pore volume
☑ BJH adsorption cumulative pore volume
☑ BJH desorption cumulative pore volume
☑ Single point adsorption micropore volume
☑ DR adsorption micropore volume
☑ t-Plot micropore volume
☑ α s micropore volume

Pore size

☑ Single point average pore radius
☑ BJH adsorption average pore radius
☑ BJH desorption average pore radius
☑ BJH adsorption most probable pore size
☑ BJH desorption most probable pore size
☑ MP most probable pore size
☑ HK most probable micropore size

Figure 4-18

Chapter 5 Theoretical Overview

(1) Definition Overview

1. Overview of Surface Features

The surface characteristics of powder materials are very complex, especially porous nano powder materials. Because the state of the surface atoms is very different from the state of the internal atoms, the surface characteristics include both the general surface and the internal surface with holes connected to the surface. To better characterize and analyze the surface characteristics of materials, data analysis is divided into two categories: specific surface area and porosity.

2. Definition of Surface Features

(1) The concept of specific surface area

Specific surface area refers to the surface area per unit mass of a porous solid substance, commonly measured in m^2/g.

(2) Overview of Holes

① The Concept of Holes

Channels or cavities within solid materials can be referred to as pores, and in addition to pores within solid materials, spaces 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 the size of their specific surface area and the diffusion of adsorbate molecules. For the convenience of characterization and analysis, the International Union of Pure and Applied Chemistry (IUPAC) later recognized that the size of pores can be divided into three categories based on their gap width: macropores:>50nm; Mesopores: 2-50nm; Micropores:<2nm. Micropores are further divided into sub micropores and super micropores; Dubinin further divides micropores into ultra micropores smaller than 0.7nm and sub micropores between 0.7-2.0.

In scientific research, in addition to quantitative classification, pores can be further classified according to their characteristics, including through pores, closed pores, cross-linked pores, blind pores, etc. Cross linked pores and blind pores include cylindrical pores, slit pores, spherical pores, etc. (See Figure 5-1)

For the convenience of research, the shape of holes is generally simplified into two categories: gap shaped and cylindrical.

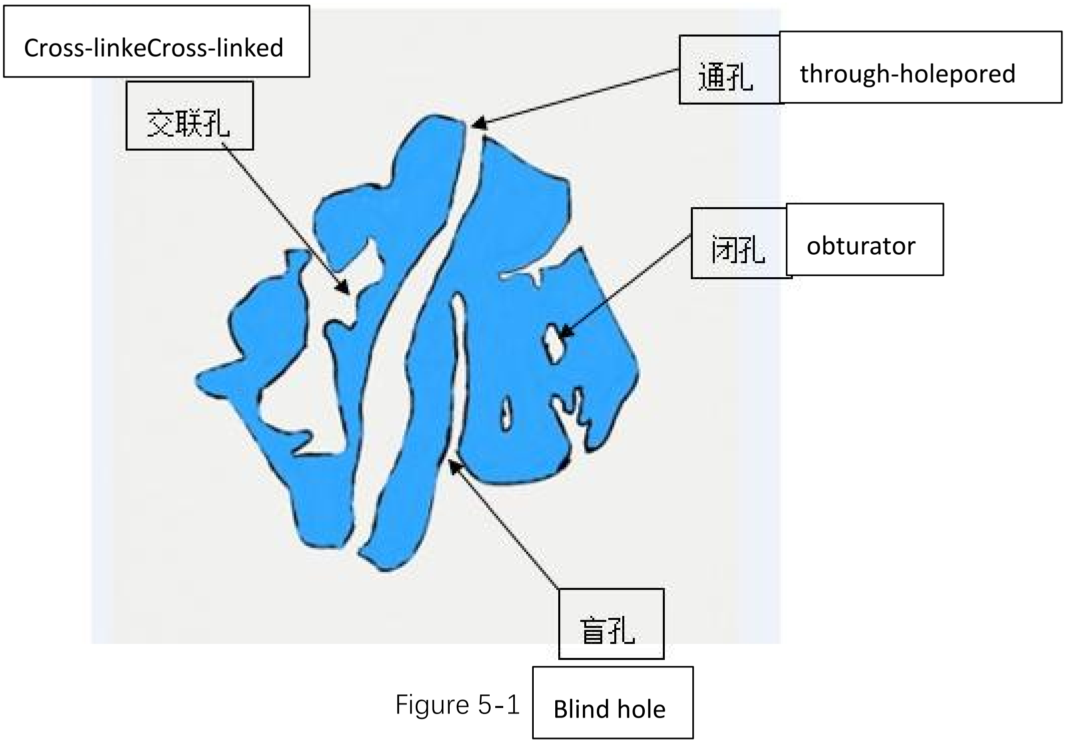


Figure 5-1

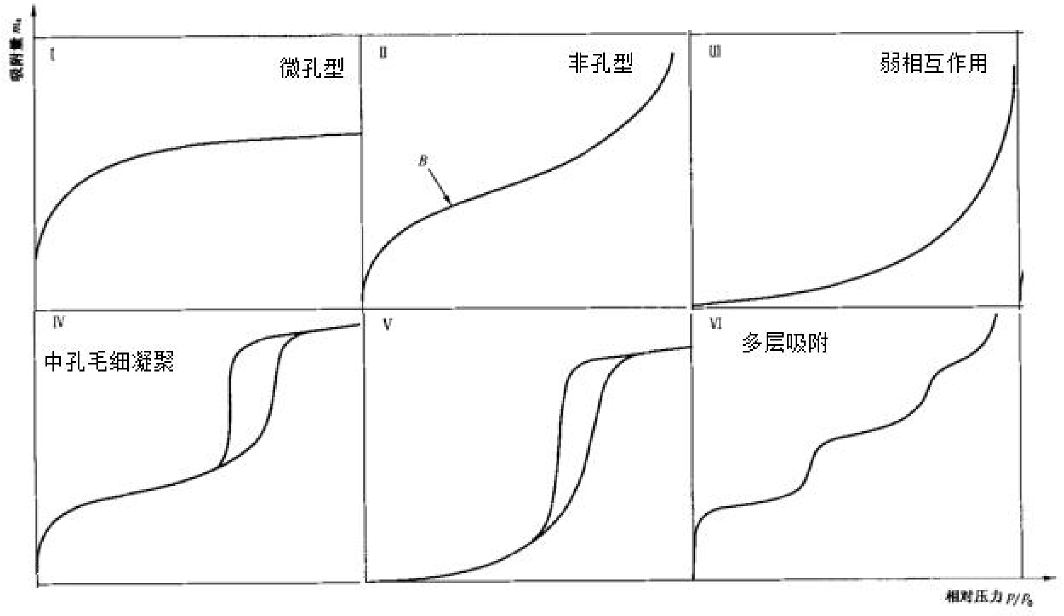
（2） Analysis Methods

1. Analysis Methods

The characterization of surface characteristics is often divided into two categories: specific surface area and porosity. There are various methods for analyzing and testing specific surface area, such as gas adsorption, gas permeation, mercury intrusion, bubble point, small angle X-ray diffraction, and so on. Among them, gas adsorption method is widely used in various industries both domestically and internationally due to its scientific testing principle, reliability of testing process, and consistency of testing results. It has gradually replaced other specific surface area testing methods and become the recognized most authoritative testing method. The gas adsorption method (also known as static capacity method) requires data to be calculated based on the adsorption and desorption amount of adsorbate gas by the sample at different relative pressures (adsorption desorption isotherms),. Simply put, gas adsorption method uses gas molecules as a measuring "ruler" to describe the characteristics of the material's surface by analyzing the consumption of adsorbate molecules and the state of the adsorption desorption curve

The commonly used gas adsorption method is nitrogen adsorption. At a constant temperature (usually the boiling point of the adsorbate gas, nitrogen is 77.35k), the curve of adsorption capacity changing with relative pressure (P/P0) is called the adsorption desorption isotherm, which is the most important manifestation of the adsorption characteristics of solid substances. Based on the data points of adsorption desorption isotherms in different relative pressure ranges, different theoretical models can be used to analyze different surface characteristics.

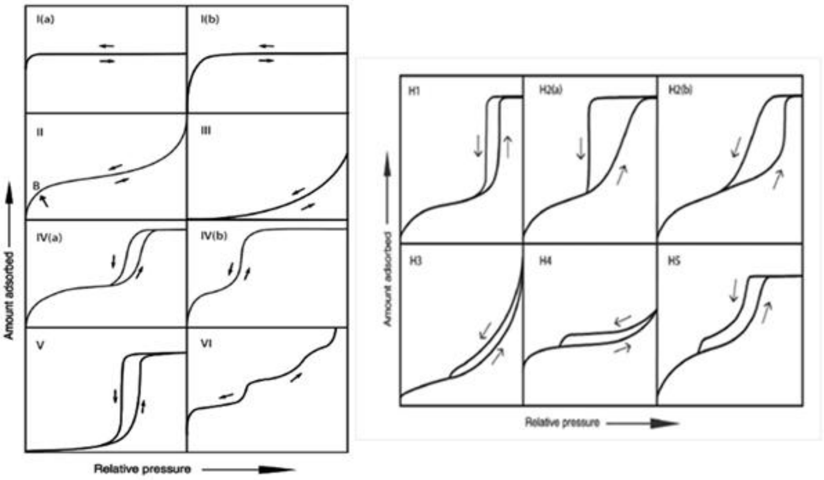
(1) Classification of Adsorption Desorption Isotherms (see Figure 5-2 and 5-3)



注：B 点为单层吸附容量结束的标记。

Figure 5-2

Figure 5-2 shows the classification of physical adsorption isotherms proposed by the International Union of Pure and Applied Chemistry (IUPAC). In 2015, the International Union of Pure and Applied Chemistry (IUPAC), an authoritative organization in the field of chemistry, released a new standard for physical adsorption analysis, as shown in Figures 5-3. In the past 30 years, with the continuous synthesis of new materials such as various ordered mesoporous molecular sieves, microporous molecular sieves, metal organic frameworks (MOFs), etc., the original specifications can no longer meet the requirements of current scientific research. In the new standard, the types of adsorption isotherms have increased from 6 to 2 sub branches, and now there are a total of 8 types of adsorption isotherms, improving the types of micropores and mesopores; The types of detachment hysteresis loops have also increased by two.



新的吸附等温线和脱附迟滞类型

Figure 5-3

The characteristics of Type I isotherms are: In the low relative pressure region, there is a rapid increase in gas adsorption capacity. This is due to the occurrence of a microporous filling process. Subsequent horizontal or near horizontal platforms indicate that the micropores have been filled with no or almost no further adsorption has occurred. When reaching saturation pressure, adsorbate condensation may occur. Microporous solids with relatively small outer surfaces, such as activated carbon, molecular sieve zeolites, and certain porous oxides, exhibit this isotherm.

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

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

Type IV isotherms are generated by mesoporous solids. A typical feature is that the adsorption branch of the isotherm is inconsistent with the desorption branch of the isotherm, and a hysteresis loop can be observed. In areas with higher P/P0 values, a plateau can be observed, sometimes ending with the final turning of the isotherm upwards.

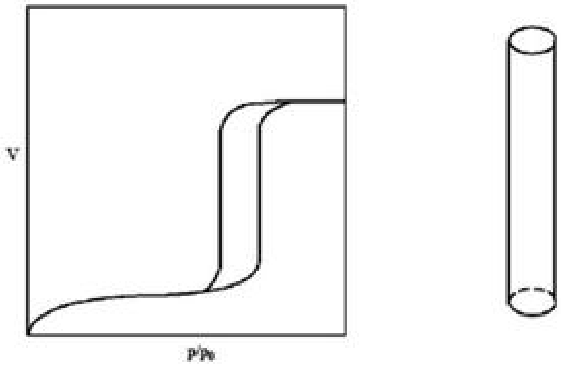
The characteristic of a V-shaped isotherm is that it protrudes towards the relative pressure axis. Unlike Type III isotherms, there is a turning point at higher relative pressures. The V-shaped isotherm originates from weak gas-solid interactions on microporous and mesoporous solids, and this type of isotherm is commonly observed in the water vapor adsorption of microporous materials.

The VI isotherm is known for its step like characteristics in the adsorption process. These steps originate from the sequential multilayer adsorption of uniform non porous surfaces. The complete form of this isotherm cannot be obtained by nitrogen adsorption at liquid nitrogen temperature, while argon adsorption at liquid argon can be achieved.

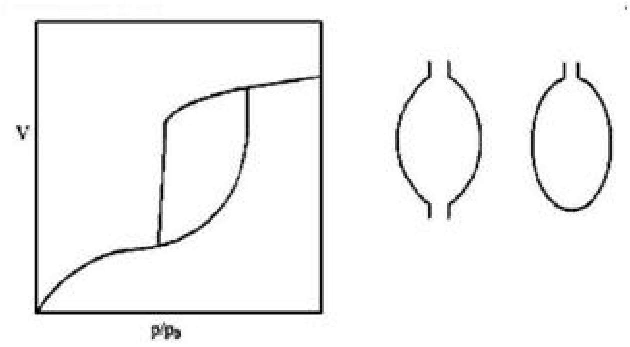
The above six common adsorption charts can be used to determine the corresponding materials by observing the adsorption amount and rate under different relative pressures. When conducting porosity analysis, the adsorption curve and desorption curve cannot overlap, so a ring shape will be formed in the isotherm curve of adsorption and desorption. This means that multi molecular layer adsorption first occurs during adsorption, and agglomeration can only occur when the adsorption layer on the pore wall reaches a sufficient thickness. There are two factors in adsorption: multi molecular layer adsorption on the pore wall and agglomeration in the pore, while desorption is only caused by capillary condensation. When desorption occurs at the same P/P0 pressure as adsorption, the vapor that only occurs on the liquid surface in the capillary cannot cause the molecules adsorbed under P/P0 to desorb. To desorb, a smaller P/P0 is required, so the hysteresis phenomenon of desorption is actually caused by the irreversibility of adsorption at the same P/P0.

(2) Judging the type of material pores from the hysteresis loop of nitrogen adsorption isotherms

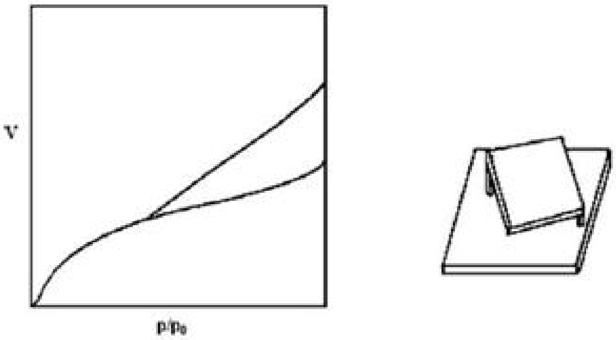
① The hysteresis loop corresponding to the tubular hole structure with open ends



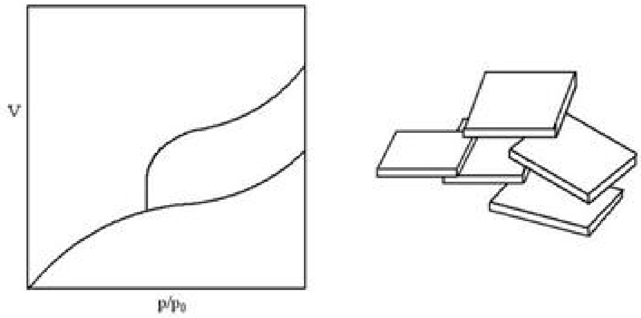
② The hysteresis loop corresponding to the "ink bottle" hole



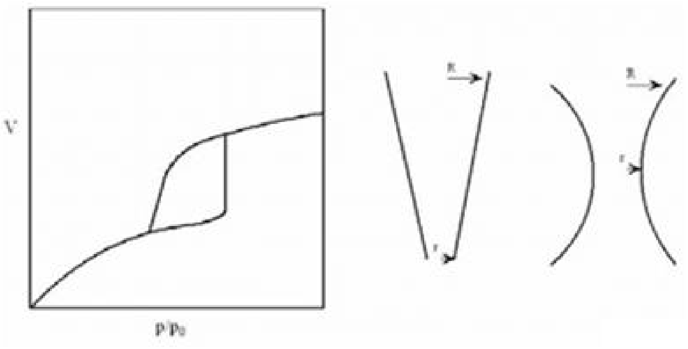
③ The hysteresis loop corresponding to the overlapping narrow slit structure of inclined plates with four open sides



③ The structure of flat slit holes corresponds to hysteresis loops (it is difficult to form concave liquid surfaces during adsorption)



④ Hysteresis loop corresponding to conical or double conical tubular pore structures (gradually evaporating during desorption).



2. Value Range of Specific Surface Area and Samples with different Pore Sizes

(1) P/Po Relative Pressure Point Selection.

The BET formula is used to calculate the specific surface area, and the BET linear relationship obtained by comparing the relative pressure of microporous materials between 0.01-0.1 and 0.05-0.2 is good. The BET results obtained by mesoporous materials at 0.05-0.2 are reasonable. Most microporous materials have a smaller specific surface area ratio of 0.01-0.1 calculated at a relative pressure range of 0.05-0.2, and the more microporous material content in the catalyst, the greater the difference calculated within these two relative pressure ranges.

The BET surface calculation of microporous materials requires selecting the correct relative pressure range, and the C constant is a simple indicator for judgment. Any negative intercept in the BET plot indicates that the value exceeds the effective application range of the BET equation. The classical range of BET values for microporous materials is 0.05-0.30, and linear data cannot be obtained, and the C constant is negative and has no physical significance. The selection of linear range for BET diagrams of microporous materials should avoid any subjectivity. The correct process is that the C constant must not only be positive, but the adsorption capacity should continue to increase in relation to the relative pressure graph.

(2) Do not Use Low Pressure Points in the BET Equation.

When some data is bent towards the origin, these points cannot be used to calculate the specific surface area. Because a low pressure point is not enough to form a monolayer, a very low C value will result in a very high intercept. In this case, it is common to obtain BET plots with significant curvature above the conventional lower limit of 0.05, indicating that the upper line of the BET pressure point has been compressed, and data points with obvious curvature below 0.3 should be removed.

(3) Do not Choose Excessively High Relative Pressure Points for the BET Equation

Incorrect point selection leads to a negative correlation coefficient difference and negative intercept in linear regression, i.e. the C constant is negative. The upper limit of BET can be calculated by the maximum value of BET at a single point. But not all samples are like this. Some samples cannot find the maximum point in single point BET calculation and increase with increasing pressure, which means that there will be no short linear region below the relative pressure of 0.15. In this case, the BET equation is not applicable to such samples.

（3） Physical Adsorption Theory

The gas adsorption theory mainly includes Langmuir monolayer adsorption theory, Polani adsorption potential energy theory, BET multilayer adsorption theory (see multi-layered adsorption), two-dimensional adsorption film theory, and polarization theory. The first three theories are the most widely used. These adsorption theories start from different physical models, comprehensively examine a large number of experimental results, and after certain mathematical processing, explain the limited parts of certain (or several) types of adsorption isotherms and provide equations to describe the adsorption isotherms. The most commonly used method for studying the adsorption capacity of solid gas is to obtain the adsorption isotherm through isothermal adsorption experiments. Different types of adsorption isotherms represent different adsorption systems and can be explained by different adsorption theories and models.

The adsorption of methane by coal mostly conforms to the Type I isotherm, and using the Langmuir equation to describe the adsorption process of coal can mostly obtain satisfactory results. It is also one of the most frequently used models in the field of coalbed methane. The first reason is that the Langmuir equation has only two parameters, making it easy to convert into a univariate equation for solution, with a simple form and convenient use. The second is that the two parameters of the Langmuir equation have clear physical meanings. Thirdly, the Langmuir equation has undergone long-term engineering practice and is believed to meet engineering needs.

1. Material Specific Surface Area Analysis

(1) Characterization of Specific Surface Area

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

In the characterization of the specific surface area of the system, there is also a portion of materials that conform to the monolayer adsorption characteristics. Based on the assumption of monolayer adsorption, Laughmuir has also inferred the corresponding specific surface area testing formula for characterizing monolayer adsorption characteristic materials, which is also known as the Laughmuir 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 has been proposed, and the t-plot method is used to test the external specific surface area.

(2) Basic Assumptions of Langmuir Monolayer Adsorption Theory

(i) There are a certain number of adsorption sites on the surface of solid adsorbents, each of which can only adsorb one molecule or atom;

(ii) The adsorption capacity of all adsorption sites on the surface is the same, which means that the adsorption heat on all adsorption sites is equal;

(iii) There is no interaction between adsorbed molecules.

Thus, the following can be obtained::

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

Pa——Gas phase pressure for adsorption equilibrium

Vm——Single molecular layer saturated adsorption capacity

V——Volume of adsorbed gas

b——Total adsorption constant

Drawing P/V-P as a straight line, b and Vm can be calculated based on slope and intercept.

(3) Multimolecular Layer Adsorption Theory.

① BET Algorithm

The BET theory is based on the single-molecule adsorption model of Langmuir's theory and is extended to the case of multi-layer adsorption based on the following three assumptions:

(i) Gas molecules can adsorb countless layers on solids

(ii) There is no interaction between the adsorbed layers

(iii) The Langmuir adsorption theory holds for each monolayer. The BET adsorption isotherm can be 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-plot method

T-plot, also known as t-curve, is an isotherm where t represents the relative pressure P/P0 as the x-axis and adsorption capacity as the y-axis,

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

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

Among them, tm - thickness of the single adsorption layer t - thickness of the adsorption layer V - adsorption capacity of the tested sample Vm - saturated monolayer adsorption capacity

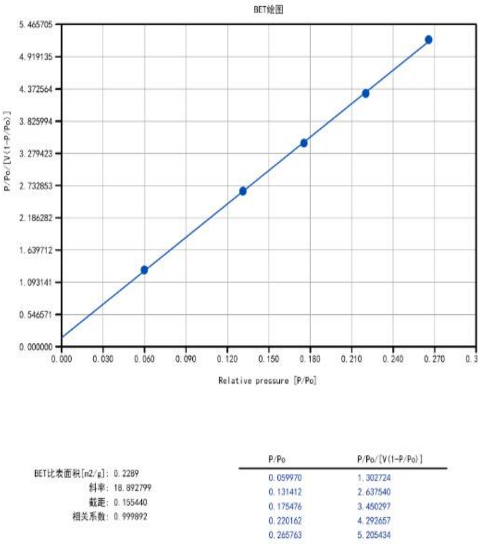
Tm - The thickness of the adsorbate (nitrogen molecules) at a temperature of 77.4K. Assuming that the nitrogen molecules in the adsorption membrane are arranged in a hexagonal dense packing pattern, the thickness of the nitrogen monolayer is given, with a value of 0.354nm.

*** The prerequisite for applying the t-plot method is:**

(i) The sample has a smooth surface within the mesoporous range

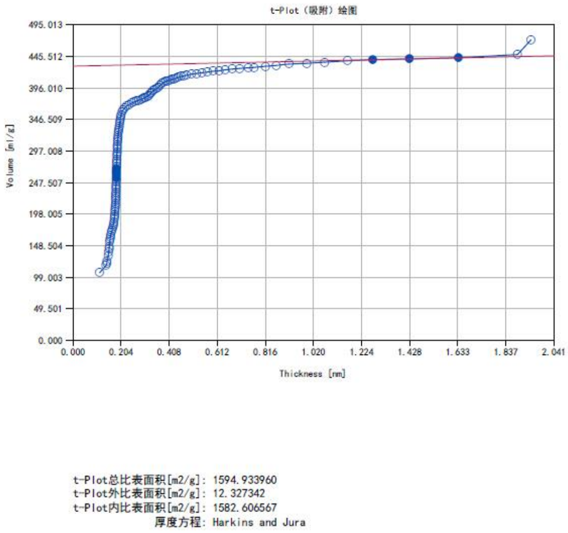
(ii) The relative pressure range of microporous filling and capillary condensation does not overlap.

(iii) The straight line on the comparison graph corresponds to multi-layer adsorption (non porous sample), and the upward deviation from the linearity is caused by the pore channels in the sample. The latter can evaluate the pore volume within a selected pore size range. The specific surface area of the straight line, i.e. the unfilled pore area, can be calculated from the slope of this straight line.



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

2. On the Characterization of Porosity

(1) The Concept of Porosity

Porosity is a proprietary term for characterizing the surface characteristics of powders and particles, including total pore volume, pore size distribution, average pore size, and other related connotations.

(2) Characterization of Porosity

According to the surface characteristics of materials, the characterization of porosity can be divided into several aspects: mesopores, macropores, and micropores.

① Characterization of Mesopores and Macropores

Test methods for mesopores and macropores

The determination and analysis of mesopores and macropores were carried out using the BJH (Barrett Joyner Helenda) method. The main theoretical basis for BJH pore size analysis is capillary condensation theory, and the main calculation method is to determine the pore size corresponding to pressure using the Kelvin equation, assuming that the nitrogen adsorbed in the pores exists at the density of liquid nitrogen.

The BJH model can calculate pore volume, pore size distribution, total pore volume, and average pore size step by step based on the data points of gas adsorption desorption isotherms.

Characterization range of mesopores and macropores

At present, the control range of nitrogen partial pressure for all nitrogen adsorbers is almost wide: the minimum value is close to 0, the maximum value is close to 1, and the lower limit of mesopores, that is, 2nm, corresponds to a nitrogen partial pressure of 0.14; When the pressure is 0.996, the diameter of the pores can reach 500nm. Therefore, the pore size analysis range conducted by the BJH method includes mesopores and some macropores. It is generally believed that the upper limit for nitrogen adsorption method to measure pores is 500nm.

② Characterization of Total Pore Volume

Total pore volume of adsorption

The total pore volume of adsorption is calculated by considering the adsorption capacity at the highest relative pressure of nitrogen as being fully adsorbed and filled in the pores.

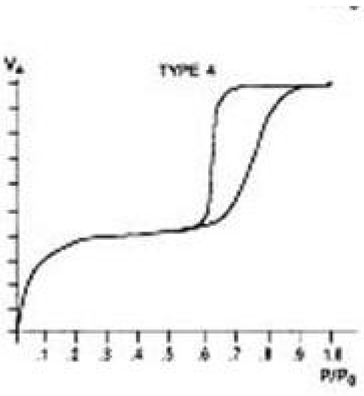
Capillary Condensation Theory and Kelvin Equation

The phenomenon of capillary condensation refers to the formation of a concave liquid surface in capillary pores due to adsorption, and the equilibrium vapor pressure P formed with the liquid surface must be lower than the saturated vapor pressure P0 of the flat liquid surface at the same temperature. The smaller the diameter of the capillary hole, the smaller the curvature radius of the concave liquid surface, and the lower the equilibrium vapor pressure. That is to say, the smaller the pore size, the lower the pressure to form capillary condensation. Due to the occurrence of capillary condensation, the adsorption capacity of the sample increases sharply. When all the 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; When reducing pressure, the condensed liquid in the large hole first desorbs, and as the pressure gradually decreases, the condensed liquid in the small hole also gradually desorbs.

Interpretation of Adsorption Desorption Curve

The adsorption desorption process curve of mesoporous materials. At critical temperature, when gas adsorbs on mesoporous adsorbents, a single-molecule adsorption layer is first formed, corresponding to the AB segment in the figure. When the single-molecule layer adsorption approaches saturation, multi-molecule layer adsorption begins. When the relative pressure reaches a specific value corresponding to the Kelvin radius at which capillary condensation occurs, capillary condensation begins to occur. Capillary condensation continues until the mesopores are filled and adsorption reaches saturation. The desorption process is exactly the opposite, starting with capillary desorption, followed by multilayer desorption, and finally proceeding with monolayer desorption.

Assuming that the capillary pores are cylindrical pores, divide all micropores into several zones according to their diameter, and arrange these pore zones in order of size. The pressure conditions for capillary condensation are different for pores with different diameters. During the desorption process, when the pressure decreases from the highest value of 1, the condensed liquid gradually desorbs from the large pores and then from the small pores. Obviously, there is a certain correspondence between the pore size that can produce condensation or desorb from the condensed state and the pressure of the adsorbate, which is given by the Kelvin equation. As shown in the figure:



Derived from: $\Delta p = \frac{2\sigma}{r_m}$ Kelvin equation: (1) $\ln(\frac{P}{p_0}) = -\frac{2\sigma V_L}{RT} \bullet \frac{1}{r_m}$

This equation provides the relationship between relative pressure and average curvature radius.

However, the aperture at which capillary condensation occurs is not the curvature radius.

Kelvin equation:

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

As shown in the figure, there is a relationship between the average curvature radius and the Kelvin radius as follows:

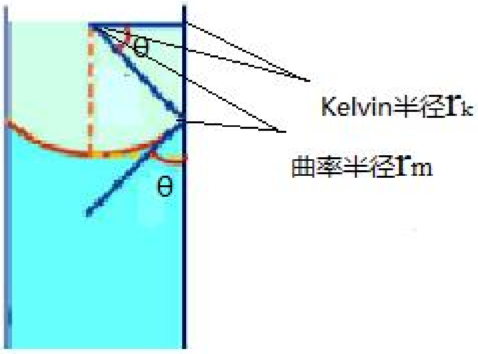
$$r_k = \cos\theta r_m$$

②Kelvin

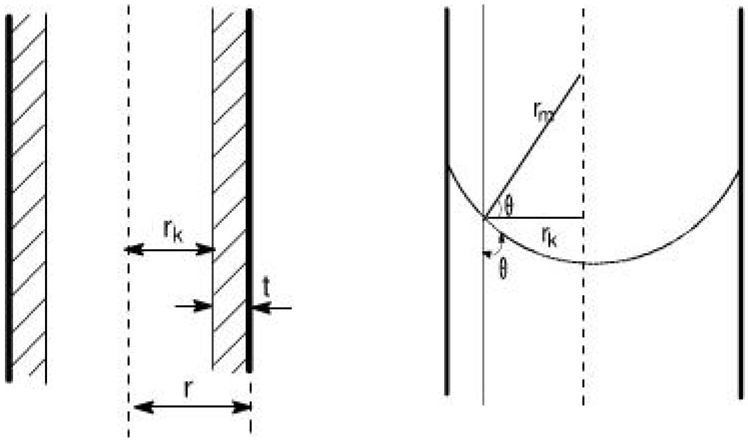
The equation provides the relationship between pore size and relative pressure when capillary condensation occurs. In fact, when Kelvin condensation occurs, multiple layers of adsorption have already occurred on the pore wall, which means that the adsorption film occurs within the pore core surrounded by the adsorption film. 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 extent, capillary condensation will occur within the adsorption film. There are the following relationships:

③ r=rk+t,

Where t is the film thickness and r is the pore radius.



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



Proposed by equations ①, ②, and ③ above:

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

Where r - pore radius; The statistical membrane thickness adsorbed on the pore wall can be obtained from the t-curve.

Determination of Mesopore Size Distribution by BJH Method

The Kelvin equation can better explain the Type IV isotherm, which is a general isotherm for the adsorption of mesoporous materials. The Kelvin equation can be used to calculate the pore radius at any point on the isotherm. According to the assumption of capillary condensation theory, all pores with a radius smaller than a certain size will be filled under the adsorption pressure corresponding to the size change. Therefore, we can obtain the cumulative empty volume as pressure. Then, by differentiating the cumulative pore volume as a function of pore radius, the pore size distribution is obtained.

Using the Kelvin equation to calculate pore size distribution based on nitrogen isotherm is a commonly used method. The BJH method proposed by Barrett, Joyner, and Halenda has now become a widely used method.

The basic assumption of this method is:

i All pores are non intersecting cylindrical pores.

ii The contact angle of the hemispherical meniscus is zero or completely wetted.

iii Apply the simple Kelvin equation.

In order to calculate the pore size distribution, it is necessary to divide the pores into several groups according to their size, and solve for a series of r and t values under different relative pressures P/P0 according to the above formulas. Then, according to the basic formula of BJH for calculating the pore size distribution, calculate the pore volume Δ V for each desorption section with different pore sizes. Plot the average pore radius r with Δ V/Δ r to obtain the pore size distribution curve. The Kelvin equation does not consider the superposition effect of adsorption potential in micropores, so it is not suitable for evaluating microporous carbon based adsorbents, only suitable for evaluating mesoporous materials. When the pore size decreases, its accuracy deteriorates. When the pore size of a micropore is equal to the diameter of an adsorbate gas molecule, the Kelvin equation is no longer applicable.

Using the BJH method, the total pore volume obtained by accumulating the pore volumes of different pore sizes obtained step by step from isothermal adsorption or isothermal desorption processes. He has clear upper and lower limits on aperture, with a lower limit of generally 2nm and an upper limit of 200-400nm. When comparing various data, attention should be paid to the differences in aperture ranges. The measurement range of aperture should match the tested material, and blindly pursuing a high upper limit for aperture measurement may not be a good thing. For example, for nano powder, its particle size is less than 100nm, and it is hard to imagine that the particle itself has a hole with a diameter of more than tens of nm. In fact, the measured "big hole" is the gap between particles, so it is wrong to characterize the surface and surface characteristics of the powder. In theory, it is believed that capillary condensation occurs when $P/P0 \geqslant 0.4$, but the lower limit of the BJH pore size analysis that has been used is much lower than 0.4, and no corresponding explanation has been provided for this.

There are three different ways to represent the average aperture:

（1） Adsorption average pore size: The average pore size calculated from the total adsorption pore volume and BET specific surface area includes all pores, with only the upper limit of pore size defined.

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

(3) BJH desorption average pore size: The average pore size calculated from the cumulative total pore volume of BJH desorption and the cumulative total pore surface area of BJH desorption, with upper and lower limits on pore size.

Extending the BJH pore size analysis method to the microporous region is incorrect for two reasons: firstly, the Kelvin equation is not applicable when the pore size is less than 2nm; Secondly, the phenomenon of capillary condensation describes that the adsorbate in the pores is liquid, while in micropores, due to the interaction of dense pore walls, the adsorbate filled in the micropores is in a non liquid state. Therefore, new theories and calculation methods are necessary for the distribution of pore sizes, and macroscopic thermodynamic methods are far from sufficient.

3. Analysis and Theory of Microporous Structure

(1) Introduction to Polanyi Adsorption Potential Theory

Polanyi's potential energy theory suggests that there is a potential energy field near the surface near adsorption, and the closer the surface is, the greater the potential energy. When a molecule is adsorbed, it enters the zero equipotential surface and is trapped on an equipotential surface between the surface and the zero equipotential surface.

According to the potential energy theory, the volume V adsorbed at different temperature ranges is determined by the surface of the adsorbent and the equipotential energy surface En. When adsorbing above the critical temperature of the adsorbate, the adsorbate cannot be liquefied, and the adsorption remains gas, but its density is relatively high on the surface; Near the street temperature, the adsorbed adsorbate is considered liquid near the surface, but gas far from the surface; If the adsorption temperature is much lower than the critical temperature of the adsorbate, the adsorbed adsorbate is considered to be entirely liquid, and the adsorption volume can be written as

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

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

The potential energy theory proposes that when the adsorbate is in a 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 vapor at equilibrium pressure p to saturation vapor pressure p0 under isothermal conditions to obtain the liquid in the adsorption region.

For any adsorption system, the characteristic curve is unique. Therefore, as long as the adsorption isotherm at a certain temperature is measured and the characteristic curve is constructed, the adsorption isotherm at any temperature can be obtained. This is precisely the advantage of the adsorption potential theory. Activated carbon is a typical non-polar adsorbent, and its interaction with activated carbon mainly relies on dispersion force. Therefore, the adsorption potential theory has been very successful in explaining the adsorption system of activated carbon.

(2) Micro Pore Filling Theory and DR Equation

Most adsorbents used in industry are microporous adsorbents, and the adsorption behavior of gases in micropores is different from that in mesopores and macropores. The microporous physical adsorption theory is based on the Polanyi adsorption potential theory, followed by the introduction of adsorption potential theory by Dubin to the study of microporous adsorption, which established the microporous filling theory. This theory is also known as the Dubin 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 transformed into the following form:

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

In order to expand the application scope of the DR method, Dubin and Astakhov proposed a more general equation, namely the DA equation

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

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

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

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

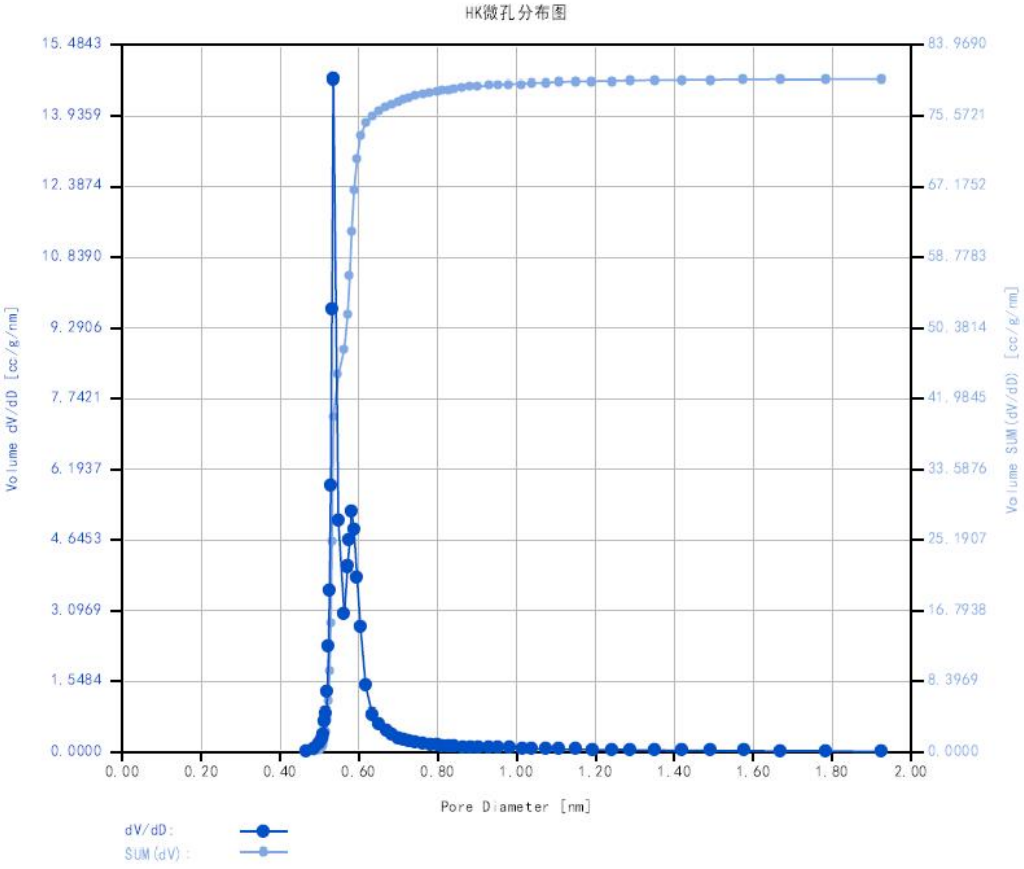
The value of n can be calculated according to the following process:

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

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

（3）Find a set of data on adsorption capacity versus pressure from the adsorption isotherm line to obtain the value of n.

The above θ - microporous filling rate; V - adsorption volume at a certain relative pressure, 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=β E0. Where E0 is the characteristic adsorption energy of the reference fluid (benzene), and β is the affinity coefficient, indicating the degree of similarity with the reference fluid.



HK Equation

The HK equation is a semi empirical method for characterizing the structure of microporous materials and calculating pore size distribution. Initially, it was based on the narrow pores of carbon molecular sieves and activated carbon, and the adsorption potential of the narrow pores was expressed as a function of pore size. Then, the adsorption amount was calculated using adsorption isotherms, and the pore distribution was calculated.

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

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

Among them: U0- interaction energy between adsorbate and adsorbent

P0- Interaction energy between adsorbate and pore wall

In the case of micropores, the interaction potential energy between the pore walls overlaps with each other, and the adsorption in micropores is greater than that in mesopores. Therefore, filling in micropores occurs when the relative pressure is less than 0.01. Pores with a pore size of 0.5~1nm can even generate adsorbate filling at relative pressures of 10~5~10~7, making the determination and analysis of micropores much more complex than mesopores.

The HK and SF methods have developed a semi empirical analysis method for calculating the effective pore size distribution from isothermal adsorption lines of microporous samples. They are respectively used for nitrogen/carbon (slit) and argon/zeolite (cylindrical pore) systems, treating the filling fluid as a certain type of loose fluid. These calculations require the introduction of a series of parameters related to adsorbents and adsorbates, and the selection of these parameters has a significant impact on the calculation results. This method proposes a new relationship between micropore diameter and filling pressure, but does not solve the density model of nitrogen molecules in micropores. Therefore, he can represent the adsorption law and pore size distribution law of micropores, but it also cannot have quantitative analysis significance.

According to Lippens and deBoer's t-plot method, which is commonly used in micropore analysis, adsorption capacity is defined as a function of statistical layer thickness t, which is calculated from standard isotherms. The t-plot can be used to calculate the specific surface area, outer surface area, and total volume of micropores containing inner surfaces. The T-plot is used to analyze the pore size distribution using the MP method, which can analyze the pore size distribution of micropores. The disadvantage of the T-plot method and the MP method is that they still consider the adsorbate filled in the micropores as a liquid, and use the Kelvin equation to calculate the pore size. Therefore, the total pore volume obtained by him only has relative significance, and the pore size distribution range cannot represent the true small pore area.

In the past decade, non local density function theory (NLDFT) and computer simulation methods (such as Monte Carlo fitting) have developed into effective methods for describing the adsorption and phase behavior of restricted non-uniform fluids in porous materials. The NLDFT method is applicable to various adsorbent/adsorbent systems. Compared with classical thermodynamic and microscopic modeling methods, the NLDF method describes the behavior of fluids confined to pores at the molecular level. Its application can associate the molecular properties of adsorbate gases with their adsorption performance in pores of different sizes. Therefore, the NLDFT method for characterizing pore size distribution is suitable for the full range of micropores and mesopores. At present, the NLDFT method is highly respected in foreign instruments and is used as a standard to compare the effectiveness of other methods. However, the NLDFT method has different calculation methods for different adsorbent/adsorbate systems, making it difficult to apply universally in different systems. The new theory aims to solve two problems. Firstly, it proposes a more reasonable correspondence between the filling pressure and pore size of micropores. Secondly, it proposes the state and density of the adsorbate filled in micropores. It is reasonable to expect a simpler and more universal density function theory, which will make the analysis of the pore size distribution of ultra micropores more extensive and comparable.

Chapter 6 Common Faults and Solutions

（1） Degassing System Malfunction

1. Temperature Display "HHHH"

(1) The temperature detector of the heating furnace is faulty and needs to be sent back to the manufacturer for repair.

(2) The power connector of the heating furnace is not connected to the host.

2. Abnormal Vacuum Pumping

Unable to vacuum

(1) Check if all sample tubes are installed at the processing ports.

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

(3) Check if the oil level of the vacuum pump is normal.

（2） Vacuum Pump Malfunction

1. Vacuum Pump Emits "White Smoke"

The fault is mainly caused by air leakage in the air circuit:

(1) Check if the air duct is damaged; Check for air leakage at the connection.

(2) (2) Check if the sample tube nut is "tightened" and if the sample tube mouth is damaged/"trachoma".

2. Abnormal Sound

(1) Due to the entry of dirt into the pump, replace the pump oil. If the pump oil cannot be improved after replacement, and the pump body has been worn by pollutants, it can continue to be used without affecting the vacuum effect.

3. Vacuum Pump not Working

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

(2) The relay that controls the operation of the vacuum pump is damaged. When the instrument controls the vacuum pump to start, there should be a 220V AC output at the power interface of the main vacuum pump, which needs to be determined using a multimeter.

(3) The vacuum pump is damaged. Connect the vacuum pump to a 220V power supply, but the vacuum pump does not work.

（3） Program Running Stuttering/Pausing

1. "Vacuum Pumping" Operation Pauses

(1) "Vacuum pumping" operation pause (1) caused by the last test not ending normally or sudden power outage during the testing process. At this point, you need to run the program first and click "Restore" when the software is operational.

(2) If the sample tube has cracks or ruptures during installation, it needs to be replaced.

(3) Due to insufficient intake pressure, it is necessary to adjust the intake pressure to 118KPa.

2. "Measure Dead Volume" Operation Pause

(1) Insufficient helium intake pressure or helium cylinder not opened.

3. "Adsorption Analysis" Operation Pauses or does not End

(1) Check if the nitrogen cylinder is open.

(2) Excessive sample size.

4. "Desorption Analysis" Operation Pauses or does not End

(1) The sample tube is damaged.

(2) Check if the nitrogen cylinder is open.

(3) Vacuum pump malfunction.

(4) Analysis time is too long, there is no liquid nitrogen in the Dewar bottle.

5. "End Analysis" does not End

(1) The sample tube is damaged.

(2) Insufficient helium intake pressure.

(3) Vacuum pump malfunction.

(4) Electromagnetic valve malfunction.

（4） Instrument Malfunction or Abnormality

1. Inconsistent Readings of Pressure Sensors

(1) Failure to shut down for a long time caused the sensor to crash.

(2) Sensor zero deviation.

(3) Sensor malfunction.

2. Crash

(1) Windows CE program motherboard malfunction.

3. Isotherm Anomaly

(1) Helium quantification error.

(2) The sample size is too small.

(3) The sample tube is damaged.

4. Software Report "Analysis Station 1, 2, and 3 Leaked"

(1) Helium is not turned on or the intake pressure is low.

(2) The sample tube is damaged.

5. Display Unconnected Instrument

(1) The instrument is not turned on.

(2) The communication line is not connected properly.

(3) Replacing or updating the computer may result in driver loss.

(4) Open multiple software.

6. Software Report "Analysis Station 1 Dead Volume Abnormality"

(1) The quantitative warehouse parameter shows 0, and the parameter is missing.

(2) Valve contamination caused the solenoid valve to open normally.

7. Software Report "Analysis Station 1 Needs Cleaning and Maintenance"

(1) The analysis port solenoid valve is contaminated.

Chapter 7 Instrument Accessories

（1） Attachment Table

Based on Actual Goods

	Code	**Packing Details**	**Specification/Model**
Main goods	/	Specific surface area and pore size analyzer host	SSA-4000
	032-0003-000	Double stage rotary vane vacuum pump	VRD-4
	005-0017-001	liquid nitrogen tank	10L
	005-0016-004Z	Dewar flask	V3
	005-0018-002	Standard substance for specific surface area of aluminum trioxide	GBW13913
Consumables	901-0002-007	Flat bottomed sample tube	Z240H
	905-0001-001	Metal funnel	L-240HJ
	903-0001-002	P0 tube	P0-240
	904-0001-008	Packing column	T-220HJ
	005-0019-000	Sample tube brush	300mm
spare parts	029-0003-001	316L gas pipeline	2 m/root
	029-0001-006	316L corrugated pipe	1.5m/KF16
	033-0008-002	Clamp	KF16
	001-0003-002	Fluorine rubber ring (with	KF16

		bracket)	
	001-0001-005	Fluorine rubber sealing ring	2.2*1.9mm
	001-0001-003	Fluorine rubber sealing ring	5*2.5mm
	001-0001-006	Fluorine rubber sealing ring	8*2mm
	005-0005-003	Sample tube filter plug	7mm
	005-0005-002	Sample tube plug	10mm
	011-0002-001	nut	M8*1
	005-0004-001	U disk	16G
	005-0003-001	test tube rack	6 stations
	power line	power line	be current
	005-0008-001	patch panel	be current
	028-0001-006	USB data cable	be current
File	/	PC testing software iPoreClient2020	The firmware version can be found in the software
	/	PC data processing software	StencilWizard
	/	operating manual	/

(2) Attachment Introduction

1. Sealing accessories for external gas pipeline, as shown in Figure 7-1

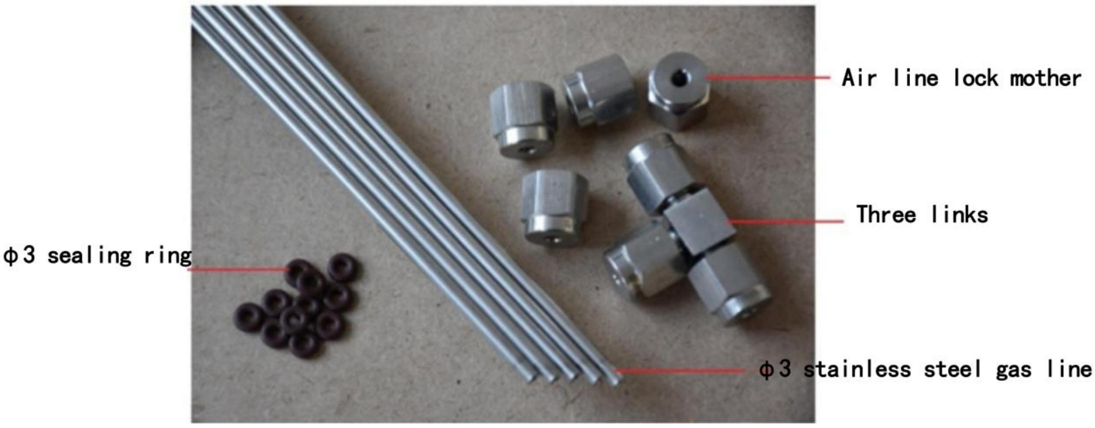


Figure 7-1

2. Vacuum pump accessories

1、The vacuum pump mainly consists of two bipolar rotary vane mechanical pumps (Figure 7-2)

2、The sealing element is shown in Figure 7-3, and the sealing method is shown in

Figure 7-4

Figure 7-2

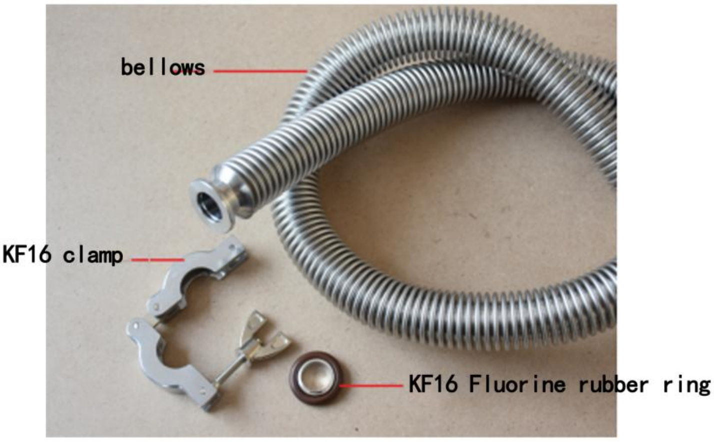


Figure 7-3

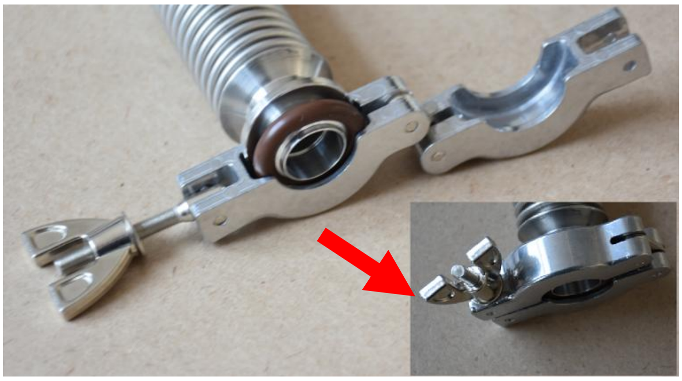


Figure 7-4

3. Sample tube and Po tube accessories, as shown in Figure 7-5, sealed accessories Figure 7-6 and Figure 7-7

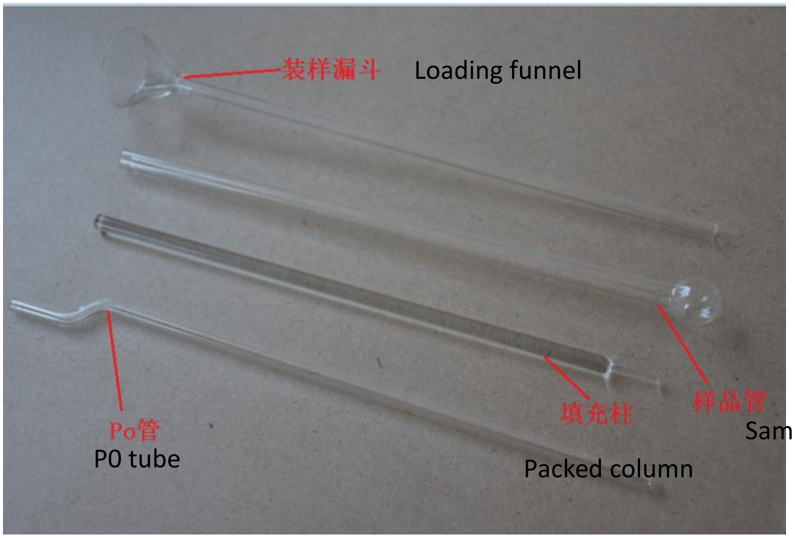


Figure 7-5

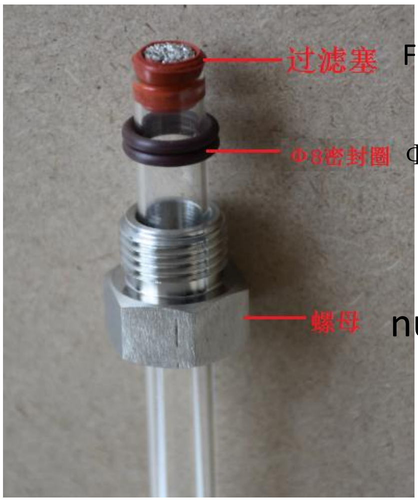


Figure 7-6 Sample tube seal

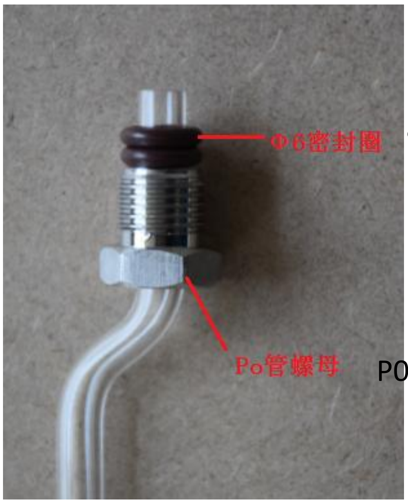


Figure 7-7 Po tube seal

4. Liquid nitrogen container accessories, mainly including Dewar bottles (Figure 7-8) and liquid nitrogen tanks (Figure 7-9)

Figure 7-8

Figure 7-9

Chapter 8 After Sales Service Commitment

The service includes two parts: technical service commitment and product maintenance commitment. Below, we will provide a detailed explanation of these two parts:

（1） Technical Services

This commitment applies to the product warranty period, which is one year from the date of equipment arrival at the user's location and acceptance upon unpacking. After the warranty period ends, we will continue to provide technical services at the most favorable price.

The reason why Biotech has the confidence and determination to make some achievements in China's inspection and analysis instrument manufacturing industry is entirely because we have "excellent talents", "rich experience", "advanced technology", and "unified command organizational system". We will organize the best and most experienced technical personnel of the company to provide the highest quality technical services for your enterprise.

1. The Content of Technical Services

Is willing to provide lifelong technical consulting services to users. Its content includes: notification of new products and technologies, software and hardware technology consulting, providing technical solutions, and researching and solving technical problems.

The environment of the equipment installation site is the basic condition for the normal operation of the equipment, and it is also the basic guarantee for the good operation of the entire system. To this end, we will provide free support and guidance for equipment installation site requirements such as temperature, humidity, and equipment connection, in line with our responsibility to users. In order for users to have a unified and reasonable management of all devices, we will provide specific requirements for the installation site of devices based on the specific actual situation of users, to meet the needs of various equipment on-site environments.

（2） Product Services

1. Hardware Maintenance

The Technical Support Center of Biotech provides 5-day, 8-hour service, and in special circumstances, it can provide 7-day, 24-hour service. During the warranty period, we will provide users with comprehensive troubleshooting technical services and monitoring of the entire troubleshooting process. (Starting from the fault until the fault is completely eliminated)

When the device malfunctions and becomes inoperable, the user needs to formally notify our company by fax and explain the cause and condition of the malfunction as much as possible. Our company will solve the problem in the following ways.

① General Faults:

General faults refer to the inconvenience caused by the failure or improper configuration of non critical equipment, which affects individual functions or operations. For such malfunctions, we first use telephone support to guide on-site solutions until the problem is resolved.

② Severity Fault:

Severe failure refers to the failure or improper configuration of important equipment, which greatly affects the use of customers. For severe faults, we first use telephone support to identify the cause of the fault, make corresponding preparations, and then go to the site to solve it.

④ Urgent Malfunction:

Emergency failure refers to the complete paralysis of the equipment, making it unable to perform any work. For urgent malfunctions, we will promise to provide telephone support and on-site resolution through two synchronized approaches to ensure timely and effective problem-solving.

In order to better serve users, please provide the product serial number when the device malfunctions. We will keep a record of any issues that arise, in order to analyze them based on this and reduce the likelihood of similar situations occurring. The time limit for fault response is: within 12 hours of receiving written fault notification from the customer. We will provide corresponding responses within the time limit in order to quickly handle the fault and minimize the losses caused by the fault.

(3) Software Upgrades

Software updates include the installation of new versions of software or the maintenance of software versions. Biotech will provide users with timely software update technology services and lifetime free software upgrades.

(4) Technical Training

In order to enable the technical personnel of the client to proficiently master the operation of the equipment and maintain the normal operation of the entire system, understand the new development direction of the involved technology, and strengthen cooperation between both parties. After the cooperation agreement between the two parties takes effect, Biotech will send dedicated personnel to arrange a series of training on specific technical and instrument equipment basic knowledge according to user needs. In order to enable the technical personnel of the client to master the usage of the instrument in the shortest possible time, and to enable the instruments and equipment of Biotech Company to maximize their use in your enterprise.

Chapter 9 Appendix

(1) Operation Process

1. Sampling and Weighing

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

（2）Sampling: Sample diameter<5mm. The amount of sample to be taken depends on the adsorption capacity of the sample. If the specific surface area is large and the pores are well-developed, the adsorption capacity will be greater, and the amount of sample required for analysis will be smaller. The following table lists the recommended values for sampling amount for reference only.

Specific Surface [㎡/g]	< 1	1-10	10-100	100-300	> 300	**Notes**
Sample Size [g]	> 2	1-2	> 0.5	> 0.1	< 0.1	Minimum cannot < 0.01

（3）Sample Installation:

①Insert the rubber plug into the empty tube mouth, weigh the mass of the empty tube and the rubber plug, record it as M1, and then peel it off.

Attention: If multiple sets of samples are weighed at once, it is necessary to ensure that the empty tube corresponds one-to-one with the rubber plug. It is optional to mark the empty tube and the rubber plug.

②Remove the rubber stopper, load it into a funnel, place the sample into an empty tube and insert the corresponding rubber stopper. Weigh the sample mass and record it as M2.

2. Sample Pretreatment

（1）Experimental equipment: degassing system, sample filter plug, and 10 diameter sealing ring.

（2）Load the sample tube into the processor: Remove the rubber plug for later use. Install one nut, two M10 sealing rings, and one filter plug into each sample tube in sequence, connect them to the degassing interface and tighten them. Lift the heating furnace so that the sample tube belly is just inserted into the heating port. The remaining processing bits are connected to air traffic control.

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

（4）Processing completed: After the sample tube cools to room temperature, click the "gas injection" button and fill it with nitrogen protection gas.

（5）Secondary weighing: Remove the processed sample tube, remove the filter plug, insert the corresponding rubber plug, and weigh it. Record it as M3. The quality of the sample after processing is M=M3-M1. Note that if M>M2, resampling is required.

3. On Machine Testing

（1）Experimental equipment: SSA-4000 analysis host, liquid nitrogen, high-purity nitrogen and helium, vacuum pump, Dewar bottle, diameter 8 sealing ring, filter plug, and filling column.

（2）Power on: Open the valves of the helium and nitrogen cylinders, adjust the output pressure to 0.3MPa, and turn on the vacuum pump and main power supply.

（3）Sample tube machine: Remove the rubber plug, and install one nut, two sealing rings with a diameter of 8, and one filter plug into each sample tube in sequence. The sealing ring should be 1-2cm away from the sample tube mouth. Make the sample tube vertical to the ground and tighten the nut.

（4）Set parameters: In the corresponding parameter setting interface of the analysis station, enter the sample quality, name, drying temperature, time, and set the file name for generating the report; Set the analysis type in the analysis control interface, generally select "micropores", and select idle for the remaining unmeasured analysis stations. After setting up, click OK, close the window, return to the main interface, and click Prepare to complete.

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

（5）Start testing: When the start testing button turns green, it indicates that all analysis stations are ready. At this point, click to start the test.

（6）Add liquid nitrogen: Fill the Dewar bottle with liquid nitrogen, with the liquid nitrogen level 1-2cm away from the mouth of the Dewar bottle. Then place the Dewar bottle on the lifting tray of the analysis host.

（2） Precautions

1. Install

（1）After the installation of the air circuit, it is necessary to first check for leaks. Open the total pressure valve of the gas cylinder, adjust the pressure reducing valve to 0.3MPa, then close the total pressure valve of the gas cylinder, wait for 10 minutes, and observe the pressure on the pressure gauge of the pressure reducing valve. If there is no pressure drop, there is no air leakage; If the pressure of the pressure gauge drops, soap water should be brushed onto the gas circuit interface such as the nut, and the bubbling point is the leakage point; If the pointer of the pressure gauge jumps back and forth, a new pressure reducing valve should be replaced.

（2） After the first installation or replacement of gas cylinders, the instrument should be vented before use. Follow the system prompts.

（3） Install and use the vacuum pump correctly, do not mix the suction port and exhaust port of the vacuum pump, otherwise it may cause the pump body to explode or more serious safety accidents. The air vibration on the vacuum pump should always be in the factory state and should not be touched. When encountering problems, the manufacturer should be contacted in a timely manner.

（4) The pump oil in the vacuum pump should always be kept clean (in its original color) and the oil level should be at 1/2 or more of the oil level display window. If the pump oil is too low, it should be added in a timely manner. If there is too much pump oil, it should be discharged in a timely manner. If the pump oil changes color, it should be replaced in a timely manner.

2. Sample Pretreatment

（1) When sampling and weighing, the sample tube should correspond one-to-one with the rubber stopper and should not be confused.

（2） If multiple sets of samples are processed simultaneously, it is necessary to label the sample tubes and stoppers for easy weighing and sample recovery.

（3） Heating up the furnace requires a certain amount of time, and the effect of heating up time should be considered when setting the processing temperature.

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

（5） The processed sample must be cooled to room temperature before being filled with nitrogen protective gas.

3. On Machine Testing

（1）The output pressure of the pressure reducing valve should be between 0.3 and 0.5 MPa, and should not be too high or too low. If it is too high, the gas pipeline should be removed first, and the gas in the pressure reducing valve should be drained. Then, reinstall the gas pipeline and adjust the output pressure of the pressure reducing valve.

（2） The sample tube to be tested needs to be filled with a column, and the weight of the sample on the software should be entered as the net weight of the sample after degassing and drying.

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

（3） If the analysis station is idle, it must be filled with empty sample tubes.

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

（5） When the specific surface area of the tested sample is small, the equal spacing progressive pressure can be set to 0.02 or lower.

（7） If there is an unexpected power outage during the testing process, the system parameters should be restored to the default state by clicking on the start analysis button again after power on. When the analysis can be terminated, click on the terminate button.

Cleaning the Instrument Casing	**weekly**
Replace the sample Tube Filter Plug	Every 3 months
Cleaning Dewar Bottles	weekly
Replace the Sealing Ring of the Sample Tube	Every 3 months
Check and Replace the Vacuum Pump Oil	Every 3 months
Run the Instrument for Leak Detection and Check the Instrument	every year

（8） After the test is completed, be sure to wait for the analysis process to display the complete completion of the bar before disassembling or powering off the sample tube.

（9） When exporting data, if the export fails, you should first make sure that a new "iPoreReport" folder has been created in the USB drive. If the export still fails, you can select the option to replace existing files on the export interface, and the export will be successful.

（3） Maintenance Guidance

1. Clean the Instrument Casing once a week

Wipe the instrument casing with a clean cloth dipped in isopropanol or clean water. Avoid cleaning solution from entering the interior of the instrument casing.

2. Replace the Sample Tube Filter Plug every 3 months

If the filter plug used in the sample tube is contaminated, adsorption and desorption may occur during the analysis process, thereby affecting the analysis results. Replace a batch of filter plugs every three months.

3. Clean the Dewar Bottle once a week

Check if the inside of the Dewar bottle is clean. Sediments inside the Dewar bottle can contaminate liquid nitrogen and affect the normal lifting of the Dewar bottle. Rinse the inside of the Dewar bottle with clean water and let it dry naturally.

4. Replace the Sealing Ring of the Sample Tube every 3 months

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

5. Check and Replace Vacuum Pump Oil every 3 months

Observe whether the oil level of the pump is normal from the oil level display window (the oil level should be in the middle position). Is the color of the oil clear and transparent (the oil should be clear and transparent),. Otherwise, drain the oil from the pump outlet and add oil to the normal oil level at the oil filling port. The oil outlet of the pump is at the bottom, so it is best to place the pump flat on a table or other high place, and use a container to collect the replaced waste oil.

6. Run the Instrument for Leak Detection and Check the Instrument every

year

Perform instrument leakage testing and calibration with standard samples every year to check instrument performance.

（4） Experimental Record Form

The following is the reference format for the record table, which is convenient for data management and viewing.

Source： Pipe Plug Number： Date：

Sample Tube Number	Analysis Station Ⅰ	Analysis Station Ⅱ	Analysis Station Ⅲ	Experimental Condition
Sample Number				Pre processing conditions:： Experimental parameters：
Sample Tube Weight (g)				
Sample Weight before Drying (g)				
Sample Tube Weight after Drying (g)				
Sample Weight after Drying (g)				

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