



# PCA-1200

## Chemisorbed instrument

### *Operation manual*



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400-6698981



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3. Gas method true density instrument series
4. Capacity based steam adsorption instrument series
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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.)
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## Attention:

*This operation manual describes all the functions of the PCA-1200 series chemical adsorption instrument. Your product may not have all the characteristics described 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 software version numbers 2018-2.0.2.0.
2. Before the first use, please read the operation manual of this product in detail. If you have any questions or suggestions, please feel free to contact 400-6698981 or service@bjbiaode.com .
3. 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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## Chapter 1 Main Technical Parameters

<b>Analysis Method:</b>	Continuous flow program heating method
<b>Analysis Method:</b>	H <sub>2</sub> 、O <sub>2</sub> 、CO、CO <sub>2</sub> 、NO、SO <sub>2</sub> 、H <sub>2</sub> S、NH <sub>3</sub>
<b>Detector:</b>	Gold plated TCD detector
<b>Output Report:</b>	TPD\TPR\TPO\TPS\TPSR
	Metal dispersion
	Active metal surface area
	Oxygen storage capacity/hydrogen storage capacity
<b>Analysis Temperature:</b>	RT-1000 °C
<b>Analyze Pressure:</b>	Atmospheric pressure
<b>Security Control System:</b>	High precision combustible gas warning
<b>Flow Control:</b>	Mass flow controller
<b>Pneumatic Control System:</b>	High precision twelve in one solenoid valve control module
<b>Sampling System:</b>	Two position six way valve
<b>Gas Input Port:</b>	6
<b>Exhaust Port:</b>	4
<b>Reactor:</b>	U-shaped bifurcated quartz reactor
<b>Report Output:</b>	TXT, PDF, Excel, etc
<b>Specifications and Dimensions:</b>	L600 x W420 x H700 mm
<b>Power Supply:</b>	AC220V 、800W



## Chapter2 Equipment Diagram





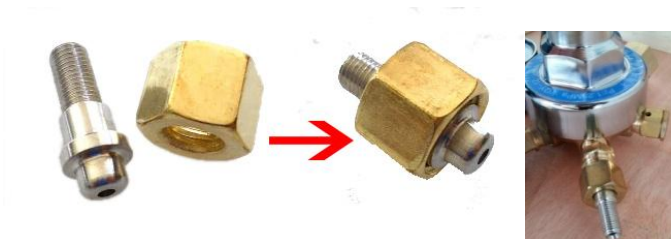
## Chapter 3 Basic Structure of Instrument System

- One main unit of chemical adsorption instrument
- Analyze a set of computers and software
- Experimental gas, please refer to Section 1 of Chapter 5 for details
- Programmed heating electric furnace
- Low temperature cold trap
- Gas connection pipe

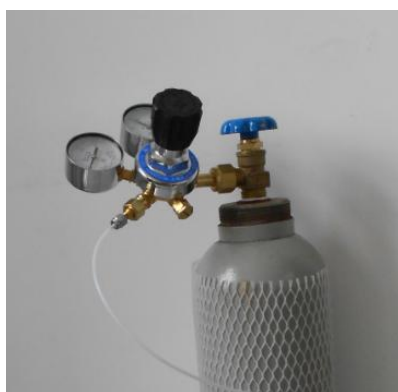
## Chapter 4 Installation and Inspection of Instruments

### 1、Installation and Requirements of Pressure Reducers

1. Install the air circuit adapter.



2. Connect the pressure reducer to the gas cylinder, wrap the raw material belt tightly at the interface of the pressure reducer, and then tighten the nut.



3. Adjust the output pressure of the secondary gauge to 0.3MPa.

### 2、Installation of Pneumatic Circuit





Insert one nut and two sealing rings with a diameter of 3 at each end of each gas pipeline in sequence. Note: During installation, the gas cylinder should be placed on the right side of the instrument, and the main engine should be placed as close to the window as possible. The exhaust pipe should not be too long.



#### 1. Installation of carrier gas path

When the length allows, the intake pipe can be bent into a spiral shape, which can effectively reduce baseline jumping caused by unstable output pressure of the pressure reducer.

#### 2. Installation of processing gas path

Be careful not to connect easily reactive gases such as hydrogen and oxygen simultaneously.

#### 3. Installation of pulse air circuit

#### 4. Installation of exhaust gas path

Pay attention to separating each exhaust gas path. In special circumstances, the reference and testing can be connected in parallel. The exhaust gas should be connected in parallel with the



pulse gas, and the exhaust air should not be connected in parallel with the reference and testing outlets to avoid polluting the TCD.

### 3、 Installation and use of instruments

#### 1. Installation and Use of Programmed Heating Electric Stove

##### (1) Installation of power cord

The electric furnace interface is located on the left side of the host, and when inserting the furnace power cord, it needs to be "point-to-point"; When unplugging, simply grip the power cord interface and unplug it, not the cable.



##### (2) Installation of electric furnace



Attention: If the quartz tube core on the inner wall of the furnace core is movable, the furnace mouth cannot be lowered when the electric furnace is taken out and moved.

#### 2. Installation and Use of Reactors

##### (1) Installation of reactors



①



②



③



④



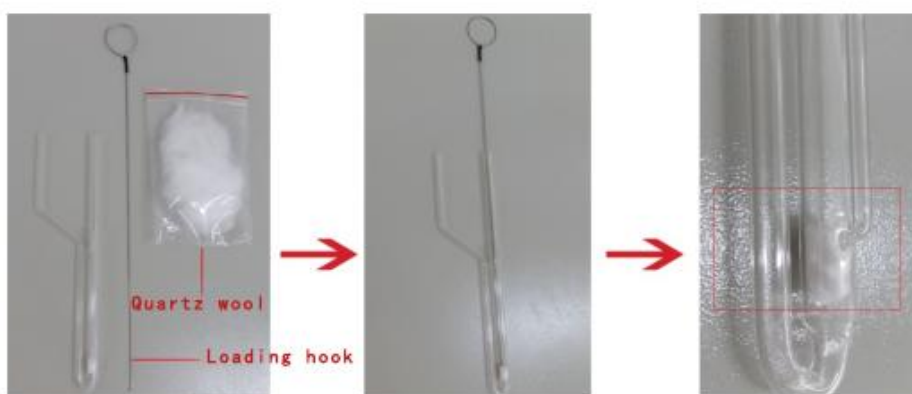
⑤



⑥

Attention: When installing the reactor, it should be kept perpendicular to the ground; When tightening the nut, hold the branch pipes on both sides with one hand and tighten the nut separately with the other hand.

## (2) The use of reactors



Take a small portion of quartz cotton and insert it into the thick tube at the right end of the reactor. Use a sampling hook to move the quartz cotton to the bottom of the bypass tube, with the upper end of the quartz cotton aligned with the bottom of the bypass tube. (PS: The



bottom position of the branch pipe is the temperature measurement position)

### 3. Installation and Use of Cold Traps



Cold trap

(1) Impurity removal: mainly used for TPR experiments, using glass cold trap tubes, adding color changing silica gel to the glass cold trap tubes, and adding ice water mixture to the cold trap tubes. It cannot be used in  $\text{NH}_3$ -TPD experiments, and the desorbed ammonia gas will be adsorbed by the color changing silica gel, making it impossible to obtain the desorption peak.

(2) Cooling: In addition to the TPR experiment, use a metal cold trap tube and add an ice water mixture to the cold trap.

### 4. Installation of software and drivers

#### (1) Software installation

Open the installation program "PCA-1200setup" and double-click to install. Follow the instructions to install.

#### (2) Installation of Drivers

##### 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.



- Right click on "My Computer" and select Properties, open Device Manager, then



find "Universal Serial Bus Controller" or locate the location where "!" appears. Double click, select Custom Update Driver, and find the driver folder in the USB software provided by Biod.

- According to the computer system configuration, select the driver "32-bit system driver" or "64 bit system driver" in the software installation program to update.

## ②Installation 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 \*\***

I、Open 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"



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



II、Click on "Update and Security"



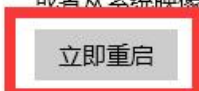
III、Click on "Restore"



IV、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。







V、After restarting, select "Troubleshooting"



VI、Select "Advanced Options" again



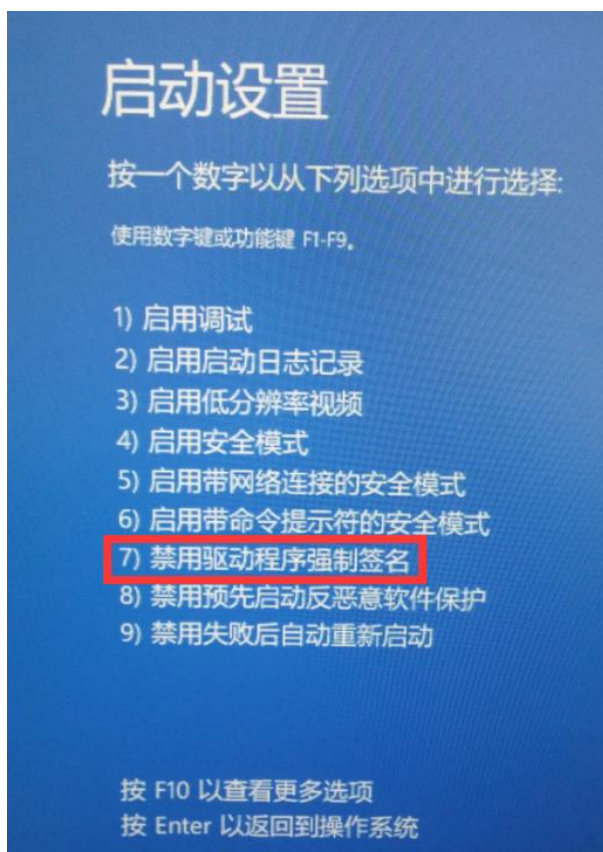
VII、Select "Startup Settings"



VIII、Click "Restart" and the computer will restart.



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



- After completing the setup, install the driver according to the installation method of the XP/Win7 system driver.

## Chapter 5 Use of Instruments

### 1、 Selection of Experimental Gases

When selecting carrier gas, try to use two components of gas with significantly different thermal conductivity (commonly used gas thermal conductivity is shown in the table below) to avoid TCD being unable to identify concentration changes in the components.

Gas	Type	Thermal	Conductivity
-----	------	---------	--------------





H2	0.163
He	0.144
Ar	0.0173
Ne	0.0455
O2	0.0240
N2	0.0228
NH3	0.0215
air	0.0233
CO	0.0226
CO2	0.0137
CH4	0.0300
C2H6	0.0180
C3H8	0.0148
C2H4	0.0164
C2H2	0.0184

2. The use of different experimental gases (note: the purity of all experimental gases below must be 99.999%)

The listed experiments are frequently conducted. Please contact our company before preparing for other experiments; After the gas is ready, a matching pressure reducer needs to be prepared, and the output pressure can be adjusted to 0.3-0.5MPa.

Purpose Experiment	Carrier Gas	Blowing/Treatment/Reaction Gas	Pulse Gas
NH <sub>3</sub> -TPD	N <sub>2</sub> /Ar	Low concentration NH <sub>3</sub> (1%-5%)	——
CO <sub>2</sub> -TPD	He	CO <sub>2</sub>	——
O <sub>2</sub> -TPD	He/Ar	O <sub>2</sub> or low concentration O <sub>2</sub>	——
N <sub>2</sub> -TPD	He/Ar	N <sub>2</sub>	——
O <sub>2</sub> -TPO	He/O <sub>2</sub> mixture (5%-10% O <sub>2</sub> )	He/N <sub>2</sub>	——
H <sub>2</sub> -TPR	Ar/H <sub>2</sub> mixture (5%-10% H <sub>2</sub> )	Ar/N <sub>2</sub>	——



	N2/H2 mixture (5% -10% H2)	Ar/N2	——
CO-TPR	Low concentration CO	He/Ar	——
Metal dispersion	Ar/N2	Ar/N2 O2 (optional steps)	H2 or low concentration H2 (If the metal load is very low, it is advisable to use low concentrations of H2)
Oxygen storage capacity	He	H2	O2
Hydrogen storage capacity	Ar/N2	O2	H2

## 2、 Pre Processing of Samples before Machine Analysis

Due to the uncontrollable state before sample analysis, the sample may contain a lot of moisture, organic matter, or corrosive substances inside. Before analysis, in order to ensure the impurities in the analysis sample, do not contaminate the instrument, and do not damage or corrode the instrument pipeline, pre-treatment is usually carried out before machine analysis.

## 3、 Determine the Amount of Sample Analysis Used

To achieve optimal accuracy and repeatability, you should pay attention to:

1. Use an equivalent sample size for each analysis, especially when comparing samples.
2. The sample is well dispersed inside the tube, and there should be no accumulation of samples on one side of the tube or many samples on the tube wall.
3. Ensure that the particle distribution of the sample remains unchanged (the distribution of large and small particles in the same material will affect the reaction



rate). For samples with a wide range of distribution, it is important to ensure that the sampling is representative.

4. The recommended sample weight is between 20 mg and 2 g. The sample should be within approximately 20 mm of the bottom of the sample tube. When determining the sample size, the percentage content of active metals should be considered. For low content samples, the sample size should be increased. The powder sample is added to the bottom of the sample tube using a long neck funnel. Large particle samples should be added using tweezers.

## 4、 Installing Sample Tubes

1、 It is best to wear gloves to hold the quartz tube, because the salt on your hands can lower the melting point of the quartz tube, and the oil on your skin can get stuck on the quartz tube, which can damage it during heating.

1) Install two sets of fixed sealing clamps, one large and one small, onto the thick and thin sample tubes. Be careful not to let the sleeve slide down the tube wall from the top and break the sample tube.

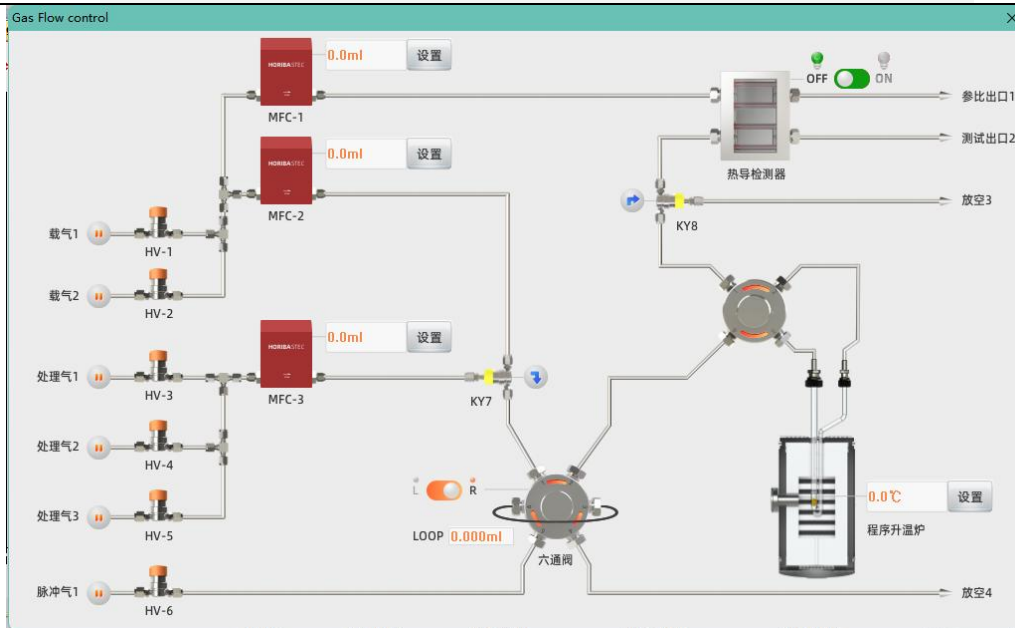
2) Install an O-ring seal at the opening of the sample tube.

3) Tighten the sample tube to the analysis port by tightening the nut.

## 5、 Gas Path Control

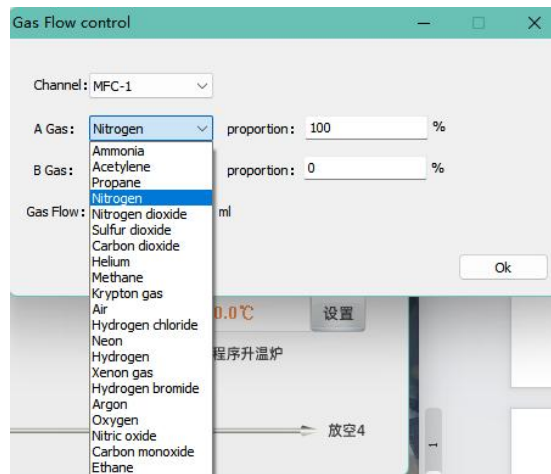
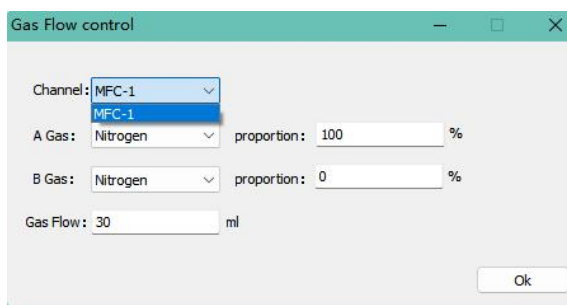
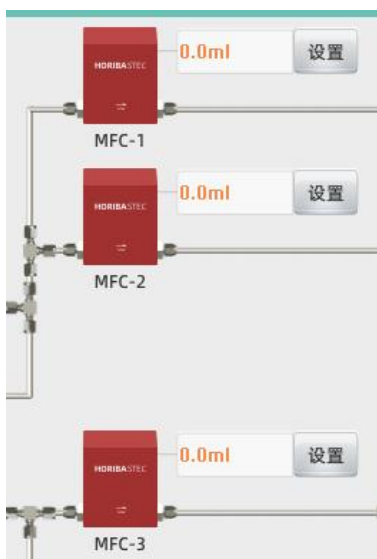
Control the pneumatic circuit through software, and open and close the corresponding numbered solenoid valves and pneumatic channels according to the analysis steps.





## 6、Flow Aetting

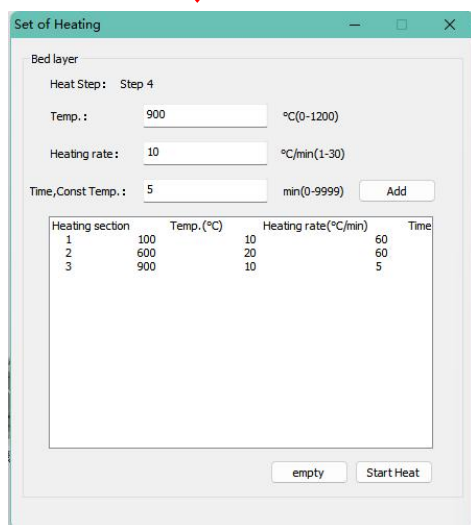
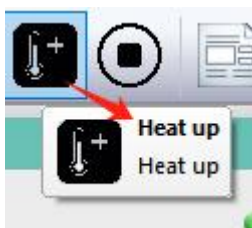
Adjust the flow rate of all test gases; Open the gas path control window to select the type of gas in each channel and set the flow rate. Click the corresponding channel setting button, enter the flow value, then confirm the button to end the flow setting, and close the gas flow setting window.





## 7、 Program Temperature Setting During the Testing Process

Set the program temperature according to the experimental conditions, click the temperature rise button, and open the setting window. After setting the temperature and heating rate, click "Add" to complete the setting, and click "Start program heating" to complete the heating control. (Note: Before clicking to start the program heating, please ensure that the thermocouple and program heating electric furnace have entered the operating position.)



## 8、 Instructions for Using TCD Temperature Control Meter

- 1、 Set temperature sensor type and temperature unit (Celsius or Fahrenheit)



On the first page of the initial 0 screen group (which is the default window displayed after power on), long press and hold for three seconds to enter screen group 4, Press to switch to (select temperature sensor type), Select 05 (K-type hot spot couple) for the chemical adsorption



instrument and confirm by pressing **ENT**. Press **□** to switch to **Unit** (select temperature measurement unit), press confirm. C °C, F °F, The initial value is C.

Exit screen group 4, long press **□**, Until displayed **SEt**, Long press again **□** Three seconds Exit screen group 4, Return to initial screen group 0.

## 2、Set manual or programmed temperature control



Switch from the basic screen button **ENT** of screen group 0 to the "1-0 initial screen" of screen group 1.

FIX control ON/OFF setting screen.

Initial setting: ON

Setting options: ON, OFF

ON: Manual constant temperature rise control; OFF: Program temperature control

Exit screen group 1, press **□** to switch to **F C** **SEt** initial screen 1-0, then press **ENT** to switch to screen group 0-0 initial screen.

## 3、Program heating setting



Switch **ENT** to the initial screen of screen group 3 (program control) by pressing the "2-0 Initial Screen" button in screen group 2. Programming instructions:

Press **□** switch to **SEtP** **SEt** press the key **▲** or **▼** select **SEtP** **i**, press **□** switch to **5.01** **0.0**, this code is to set the temperature value for heating up, press **▲** the key to set the temperature value, press **ENT** confirm. Press **□** to switch to **5.01** **0.00**, this code is used to set the heating time (for example, if the temperature rises to 500 degrees per minute at 10 degrees, the time value of this code needs to be set to 50 minutes), press the key **▲** to set the time value you need, and press **ENT** confirm. Press **SEtP** **i** switch back, press or hold the key to switch to code, then long press **□** to switch back **PrOG** **Ptn1** 初始屏幕 (曲线1), and then press **ENT** to switch back to screen group 0-0



## Chapter 6 Experimental Steps

### Notice before Experiment:

- This version of the manual and instrument has achieved fully automated analysis, data reading, and automatic saving of program temperature rise and pulse.
- You can choose between fully automated analysis modules or semi automated analysis modules.

### 1、 Fully Automatic Analysis Mode

#### 1. Preparation before the experiment

1.1 Connect the experimental gas, open the gas cylinder, and adjust the output pressure to 0.3Mpa.

1.2 Install the reactor containing the sample into the instrument analysis position, insert the thermocouple into the corresponding position, correctly place the cold trap, raise the programmed heating furnace, and ensure that the four-way valve is pointing correctly.

1.3 Open the software and find the "Automation Experiment" button.

2. According to the experimental requirements, after selecting TPR/TPD/TPO/pulse, the gas (cylinder) connection position will be automatically prompted. Before testing, please check if the gas connection is correct.

3. After clicking OK, the automation experiment settings interface will pop up, as shown in the following figure.

#### 3.1 Program Temperature Recovery (TPR) Automation Settings Window



TPR Set

Valve

Carrier ( Value1 ) Purge sample : Value3

Temperature Programmed Heating Treatment (Dehydration)

Temp.Prog.Heat: Heat setting

Channel 3: Gas Flow control

Program heating restoration

Start temp.: 0 °C

Channel 1、2: Gas Flow control

BaseLine

☒ Times of(>2): 0

☐ Voltage variation range(>5): ± 0 uv

Temp.Prog.Heat: Set of Heating

Open Save Ok

### 3.2 Automated setup of temperature programmed desorption (TPD)

TPD Set

Value

Carrier Gas: Value1 Purging Gas: Value3 Adsorption Gas: Value3

Temperature Programmed Heating Treatment (Dehydration)

Temp.Prog.Heat: Temp. Setting Channel 3: Gas Flow control

Adsorption

Temp.: 0 °C Time,Const Temp.: 0 min Channel 3: Gas Flow control

Purging

Temp.: 0 °C Time,Const Temp.: 0 min

Temp.Prog.Heat Desorption

Start Temp.: 0 °C Channel 1、2: Gas Flow control

Baseline

☒ Times of Zeroing(>2): 0 ☐ Voltage variation range(>5): ± 0 uv

Temp.Prog.Heat: Set of Heating

Open Save Ok





### 3.3 Automatic Settings Window for Temperature programmed Oxidation (TPO)

TPO Set

Valve

Carrier ( Value1 ) Purge sample: Value3

Temperature Programmed Heating Treatment (Dehydration)

Temp.Prog.Heat: Heat setting

Channel 3: Gas Flow control

Program heating oxidation

Start temp.: 0 °C

Channel 1 \ 2: Gas Flow control

BaseLine

☒ Times of(>2): 0

☐ Voltage variation range(>5): ± 0 uv

Temp.Prog.Heat: Set of Heating

Open Save Ok

### 3.4 Automated setup window for pulse experiment (H2-O2)

Pulse Set

Value

Carrier Gas: Value1 Purging air: Value3 Adsorption Gas: Value3

Pulse Gas: Value6

Temperature Programmed Heating Treatment (Dehydration)

Temp.Prog.Heat: Temp. Setting Channel 3: Gas Flow control

Adsorption

Temp.: 0 °C Time, Const Temp.: 0 min Channel 3: Gas Flow control

Temp.Prog.Heat

Start Temp.: 0 °C Channel 1 \ 2: Gas Flow control

Temp.Prog.: Temp. Setting

Baseline

☒ Times of Zeroing(>2): 0 ☐ Voltage variation range(>5): ± 0 uv

Pulse

Times: 0 Waiting time: 0 min Channel 3: Gas Flow control

Open Save Ok



4. Set up an automated running program (using NH<sub>3</sub>-TPD experiment as an example, divided into the following four experimental stages), in the order of I → II → III → IV.

Before instrument operation or automatic program execution, it is necessary to ensure that the gas is correctly connected, the gas cylinder is open, and the valves are in their respective states.

The screenshot shows the 'TPD Set' window with four main sections, each outlined in red and labeled with a Roman numeral:

- I Value:** Contains three dropdown menus: 'Carrier Gas: Value1', 'Purging Gas: Value3', and 'Adsorption Gas: Value3'.
- II Temperature Programmed Heating Treatment (Dehydration):** Contains 'Temp.Prog.Heat: Temp. Setting' and 'Channel 3: Gas Flow control'.
- III Purging:** Contains 'Temp.: 0 °C', 'Time, Const Temp.: 0 min', and 'Channel 3: Gas Flow control'.
- IV Temp.Prog.Heat Desorption:** Contains 'Start Temp.: 0 °C', 'Channel 1 \ 2: Gas Flow control', a 'Baseline' section with radio buttons for 'Times of Zeroing(>2): 0' (selected) and 'Voltage variation range(>5): ± 0 uv', and 'Temp.Prog.Heat: Set of Heating'.

At the bottom are buttons for 'Open', 'Save', and 'Ok'.

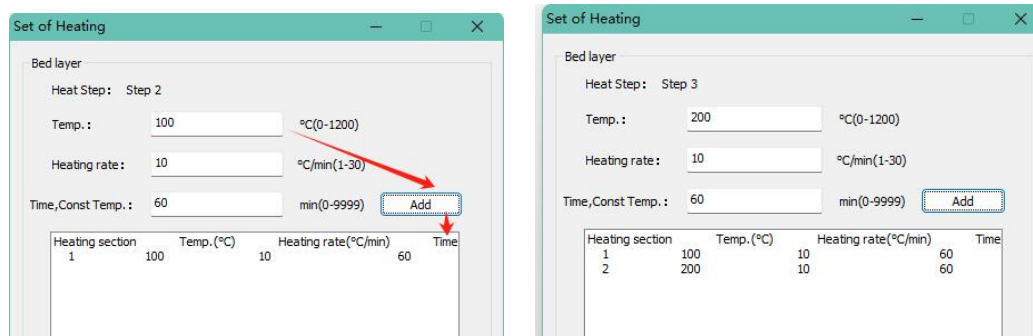
4.1(i.e., I) Valve: Select the correct working air path, and the intake ports 1-6 correspond to valves 1-6 respectively.

4.2(i.e. II) Program heating and dehydration setting

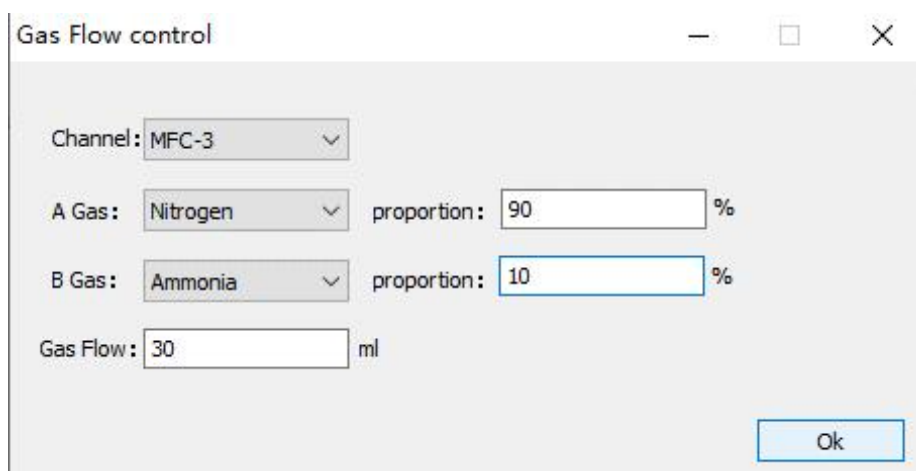
(1) Set target temperature: Click on "Heating Settings" to bring up a dialog box. You can

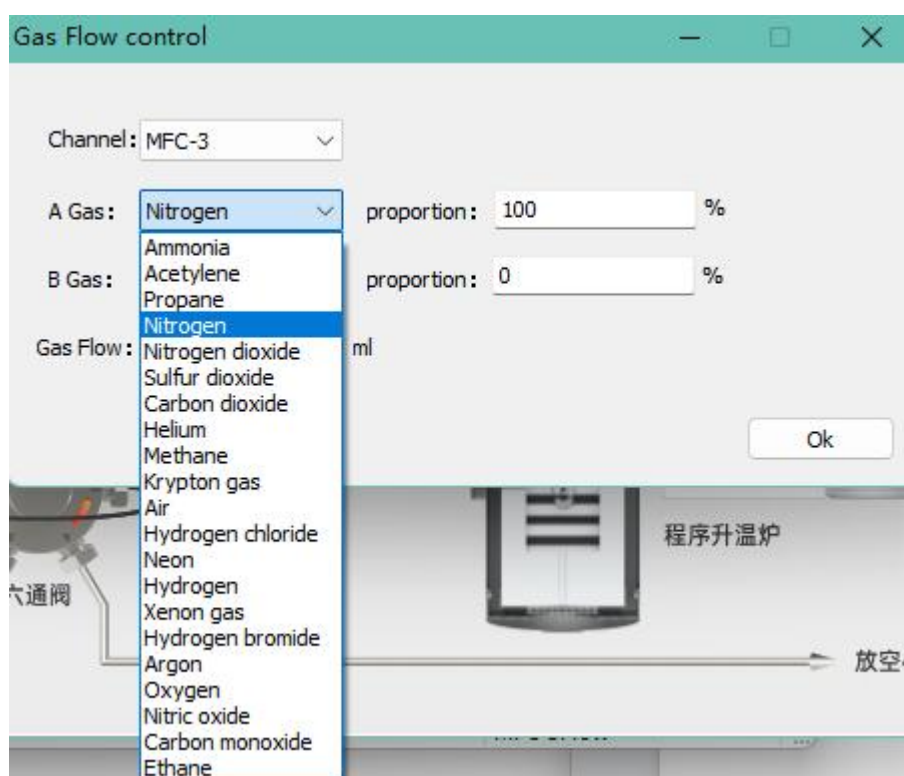


add 1-10 heating programs here.



(2) Set the type and flow rate of the blowing gas: Click on "Channel 3 Flow Setting" to bring up the dialog box. Channel 3 can control the intake of valves 3-5.

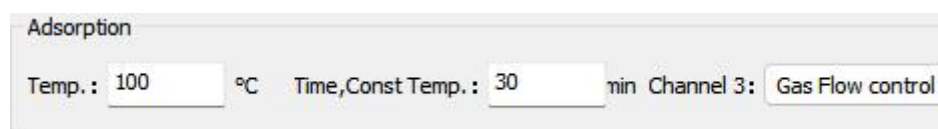




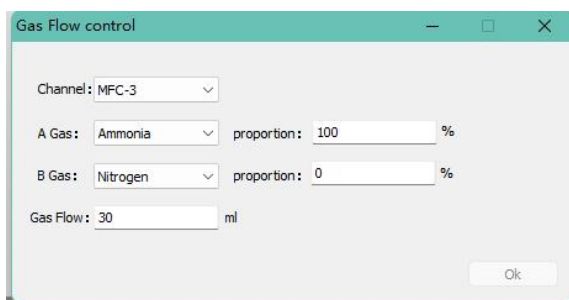
Note that if it is a single component gas, after selecting gas A, the proportion should be set to 100%, and the proportion of gas B should be set to 0%. Click OK to proceed; If it is a two-component mixture, after selecting components A and B, it is necessary to correctly input the percentage ratio corresponding to that gas component.

#### 4.3 (i.e. III) NH<sub>3</sub> saturation adsorption setting

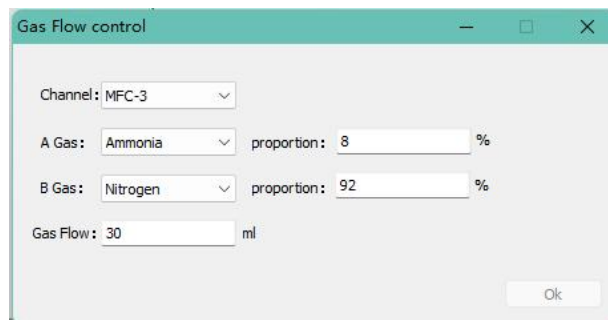
(1) NH<sub>3</sub> adsorption setting: Set the target temperature, adsorption duration, and adsorption gas in channel 3.



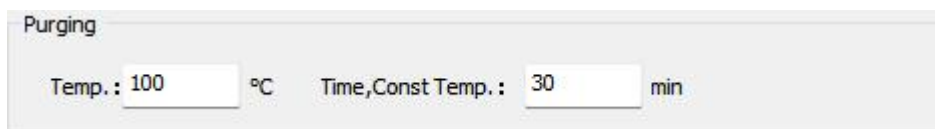
Attention: If the adsorbed gas is high-purity ammonia, select ammonia in the A gas drop-down window, set the proportion to 100%, and set the proportion of the other B gas to 0%. Click OK to proceed; If it is a mixture of ammonia and another component, after selecting components A and B, it is necessary to correctly input the corresponding percentage ratio of ammonia and the other component. As shown in the following figure



Or

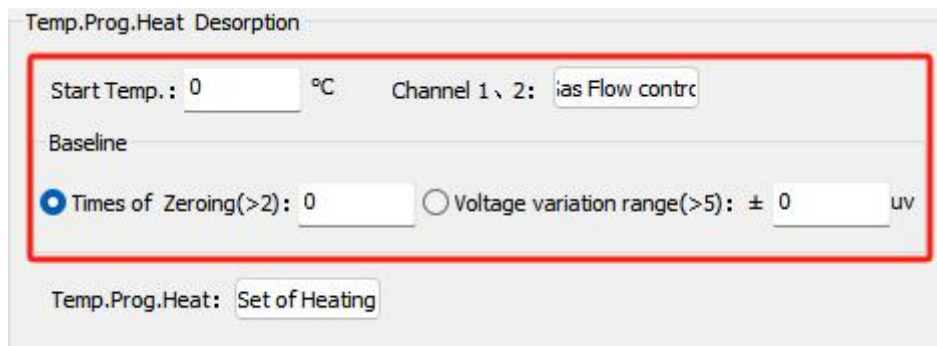


(2) Removing physically adsorbed ammonia: The blowing temperature is generally the same as the adsorption temperature, and the blowing time is at least 30 minutes

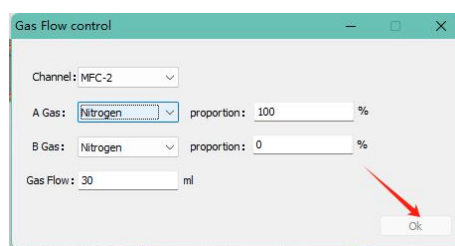
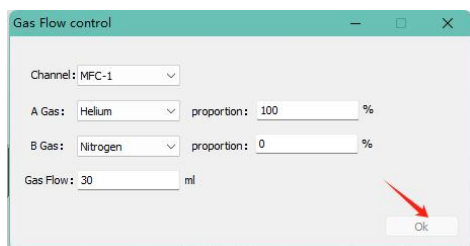


#### 4.4 (i.e. IV) NH<sub>3</sub> programmed temperature desorption: obtaining experimental results

##### (1) Baseline program settings



- ① The starting temperature is set to  $\geq 50$  °C, and channels 1 and 2 respectively control the TCD reference and test gas paths.



- ② Baseline stabilization standard (two options): Zero adjustment frequency  $\geq 10$  times or voltage fluctuation range 20-100  $\mu$  V



Temp.Prog.Heat Desorption

Start Temp.: 80 °C Channel 1、2: Gas Flow control

Baseline

☒ Times of Zeroing(>2): 2 ☐ Voltage variation range(>5): ± 0 uv

- ③ Set up the detachment program: Click on "Heating Settings" to bring up a dialog box.

Temp.Prog.Heat Desorption

Start Temp.: 80 °C Channel 1、2: Gas Flow control

Baseline

☒ Times of Zeroing(>2): 2 ☐ Voltage variation range(>5): ± 0 uv

Temp.Prog.Heat: Set of Heating

Set of Heating

Bed layer

Heat Step: Step 2

Temp.: 1000 °C(0-1200)

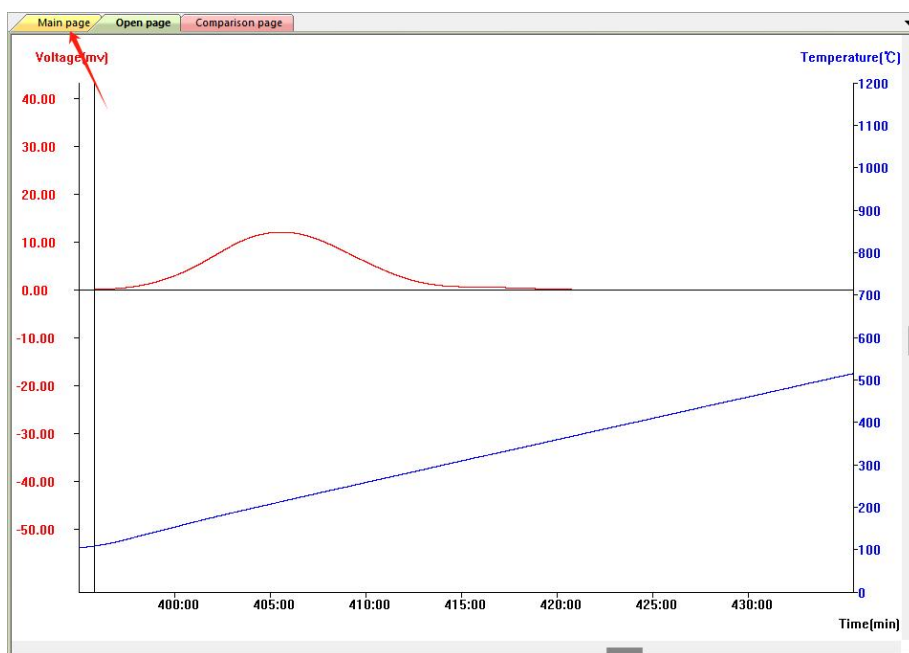
Heating rate: 10 °C/min(1-30)

Time, Const Temp.: 5 min(0-9999)

Heating section	Temp.(°C)	Heating rate(°C/min)	Time
1	1000	10	5



After setting up, click OK to close the current window and return to the TPD main window. After clicking the "OK" button at the bottom, the TCD main window will automatically disappear. After a few seconds, hearing the instrument make a "beep" sound indicates the start of the automatic program execution. Real time spectrograms can be viewed in the software's main page window.



5. After the experiment, close the instrument and gas cylinder valves, and install the analysis position into a clean reactor.

## 2、Semi Automatic Analysis Mode

### (1) Catalyst acidity analysis (NH<sub>3</sub>-TPD)

#### 1. Preparation before the experiment

1.1 Connect the carrier gas 1 (valve 1) interface to N<sub>2</sub> or He, and the processing gas 1 (valve 3) interface to NH<sub>3</sub>.

1.2 Fill the bed layer, load the sample into the reactor, about 0.05-0.5g (experimental temperature < 600 °C, use a glass reactor; < 1000 °C, use a quartz reactor), and connect to the instrument interface.

#### 2. Conduct experiments

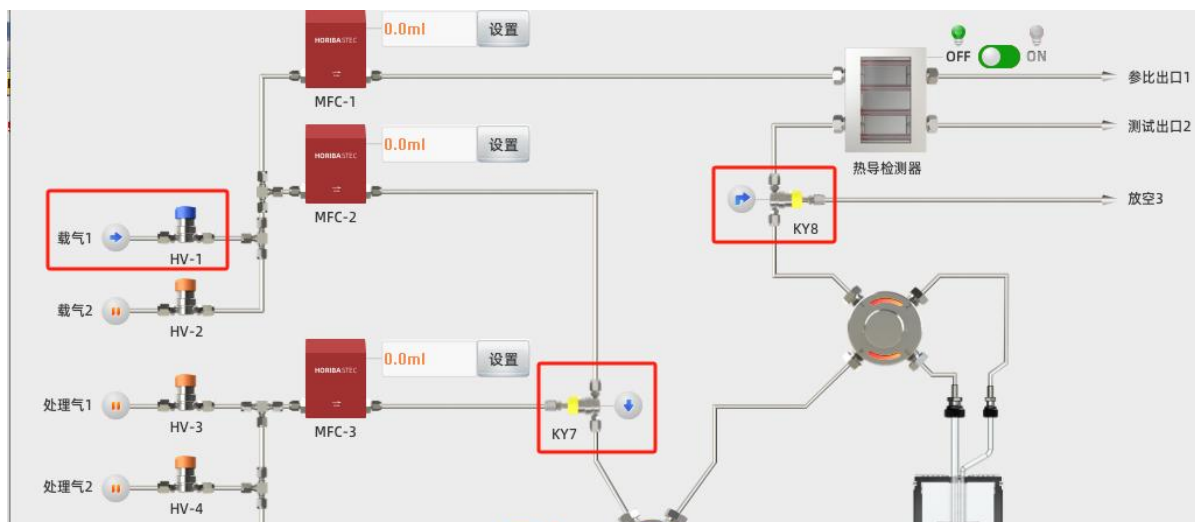
##### 2.1 Remove residual gases from the gas path



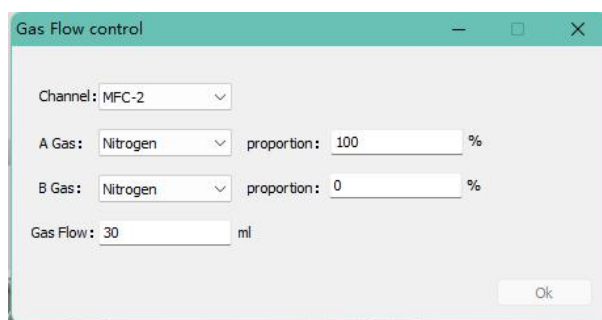


(1) Open the host, software, and N2 gas cylinder valve (adjust the output pressure to 0.3-0.4 MPa).

(2) Adjust the air path: Control the air path by adjusting the direction of valve 1/valve 7/valve 8 as shown in the diagram below.

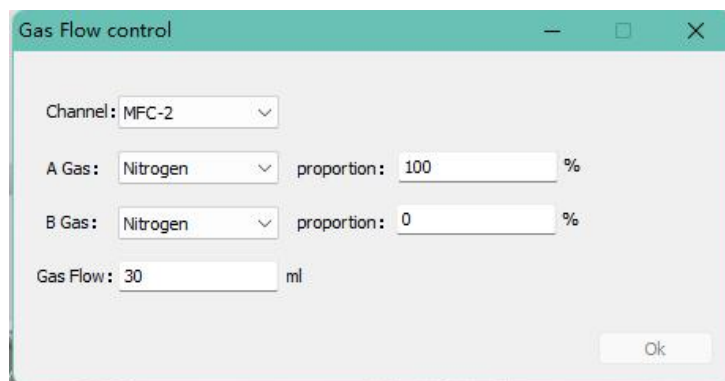


(3) Adjust flow rate: Gas path control → Select channel 2 # gas and set it to 30ml/min → Confirm and close the window.



Here, channels 1 # and 2 # refer to the instrument carrier gas channel (1 # is the reference port flow rate; 2 # is the test port channel)

This step requires selecting an inert gas to blow the gas. For example, if nitrogen is used, the type of gas A should be nitrogen with a ratio of 100%, and the type of gas B does not need to be selected with a ratio of 0%. Click OK to proceed.







(4) Blow the sample in an inert gas environment at room temperature for at least 10 minutes.

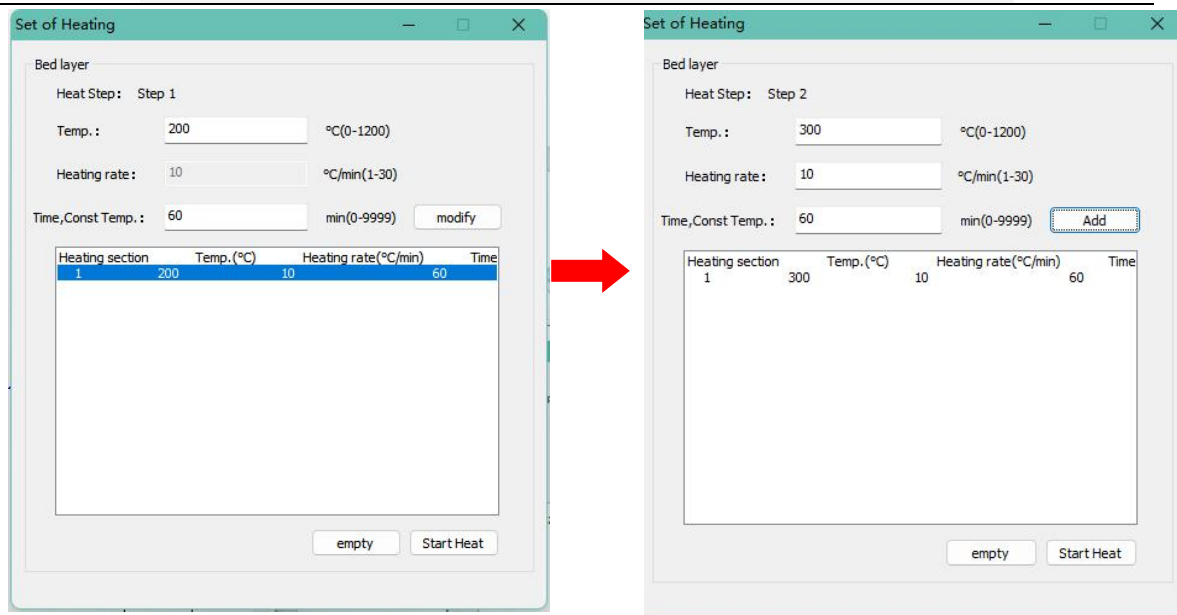
## 2.2 Programmed heating pretreatment (dehydration)

(1) Raise the furnace and insert the thermocouple into the bottom of the reactor branch pipe.

(2) Setting conditions: Multiple program temperature settings can be performed, and relevant parameters can be set according to the experimental conditions: temperature control → set the upper temperature limit, heating rate, and constant temperature time → add to the temperature control section → click to start heating → close the interface.

Heating section	Temp.(°C)	Heating rate(°C/min)	Time
1	200	10	60

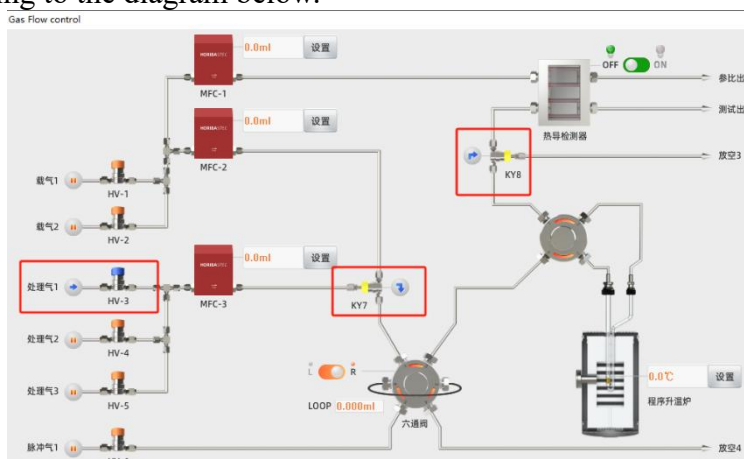
If it is necessary to increase or decrease the target temperature during program execution, you can left click to select the target segment that needs to be modified. At this time, the "Add" button will change to "Modify". After resetting the target temperature, click the "Modify" button.



(3) After dehydration is completed, when the furnace temperature drops below 200 °C, lower the furnace (to avoid thermocouple damage), and continue to use N<sub>2</sub> for blowing. The next operation can be carried out after the temperature of the sample position returns to room temperature (generally, the strong cooling system requires 30-40 minutes for the furnace body to cool down to below 50 °C)

### 2.3 Saturated adsorption of NH<sub>3</sub>

- (1) Open the valve of the NH<sub>3</sub> gas cylinder.
- (2) Adjust the air circuit: Control the air circuit and adjust the direction of valve 3/valve 7/valve 8 according to the diagram below.

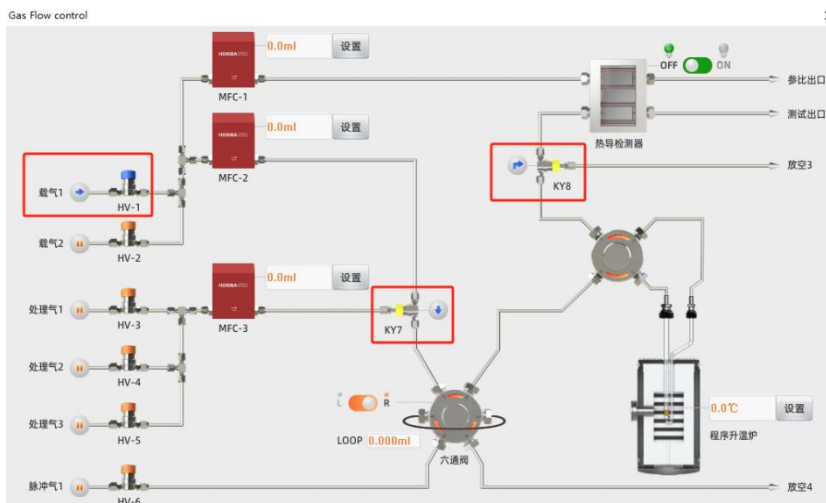


- (3) Channel 3 # selects ammonia and sets the flow rate: usually 30ml/min.
  - (4) The adsorption temperature of pure NH<sub>3</sub> is generally between 50-150 °C, and the adsorption time is 15-20 minutes. After completion, close the gas cylinder.
  - (5) Removing physically adsorbed NH<sub>3</sub>
- At a certain temperature (usually NH<sub>3</sub> adsorption temperature), use N<sub>2</sub> for blowing, and



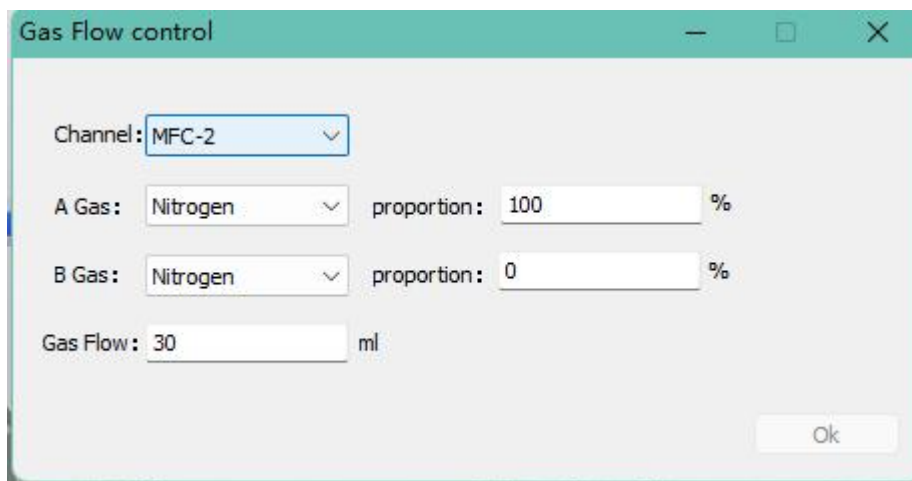
the steps are as follows:

- ①Open the N<sub>2</sub> gas cylinder valve (adjust the output pressure to 0.3-0.4 MPa).
- ②Adjust the air circuit: Control the air circuit and adjust the direction of valve 1/valve 7/valve 8 according to the diagram below.



- ③Adjusting flow rate: Air path control, channel 2 # set to 30ml/min → complete and close window.

Here, channels 1 # and 2 # refer to the instrument carrier gas channel (1 # is the reference port flow rate; 2 # is the test port channel).



- ④Setting conditions: Temperature control → Set temperature upper limit, heating rate, and constant temperature time → Add to temperature control section → Click to start heating → Close the interface (longer constant temperature time can also be set in step (4) above, and after reaching the adsorption time, switch the gas path to complete the physical adsorption of NH<sub>3</sub> removal. Step (5) is omitted, and generally at least 30 minutes of NH<sub>3</sub> removal is required).



Set of Heating

Bed layer

Heat Step: Step 1

Temp.: 0 °C(0-1200)

Heating rate: 0 °C/min(1-30)

Time, Const Temp.: 0 min(0-9999) Add

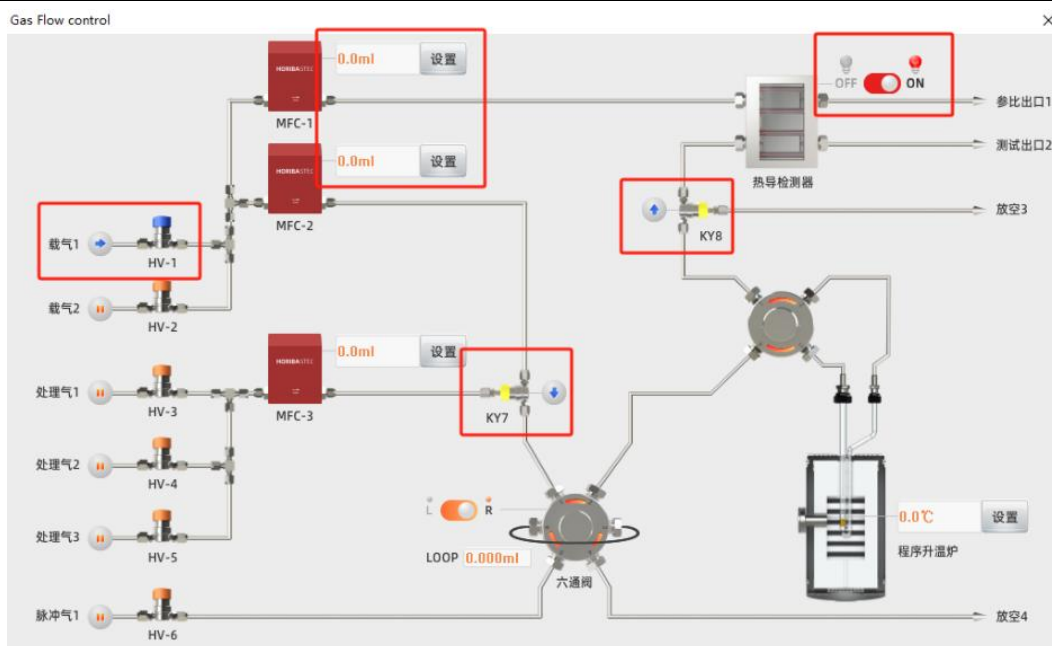
Heating section	Temp.(°C)	Heating rate(°C/min)	Time
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empty Start Heat

## 2.5 Programmed Temperature Rise Desorption

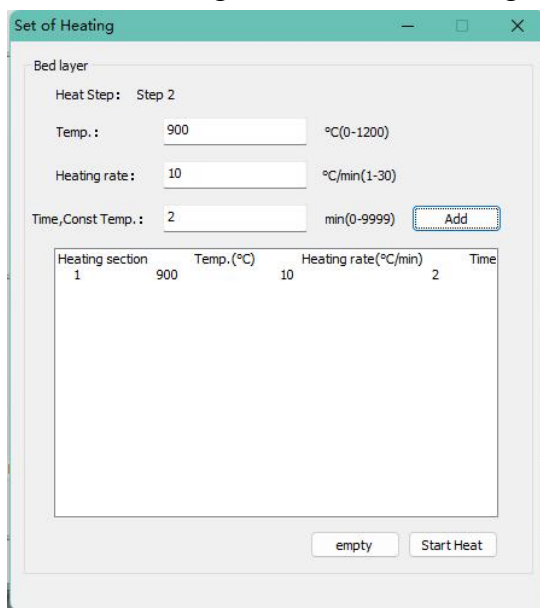
- (1) Open the N<sub>2</sub> gas cylinder valve (adjust the output pressure to 0.3-0.4 MPa).
- (2) Adjusting the air circuit: Air circuit control, adjust the direction of valve 1/valve 7/valve 8 according to the diagram below.
- (3) Adjust the flow rate (reference and test flow rate): Channel 1 # selects nitrogen and sets it to 30ml/min, while Channel 2 # selects nitrogen and sets it to 30ml/min, and switch the TCD indicator light to on.

Here, channels 1 # and 2 # refer to the instrument carrier gas channel (1 # is the reference port flow rate; 2 # is the test port channel).



(4) Adjust the polarity according to the TCD polarity, turn on the TCD, and wait for the baseline to level (multiple zeros are required during the stabilization process until the voltage fluctuation range is  $\pm 50 \mu V$ ). Raise the furnace to the designated position (due to the previous heating operation, there may be residual temperature in the furnace body, and the strong cooling system usually takes 30-40 minutes for the furnace body to cool down to below  $50^\circ C$ . After the temperature stabilizes, zero the baseline and proceed to the next step).

(5) Set up desorption program: Open the heating window, set the maximum desorption temperature, heating rate, etc. After adding, click the start heating button to start desorption.



### 3.Experimental completion

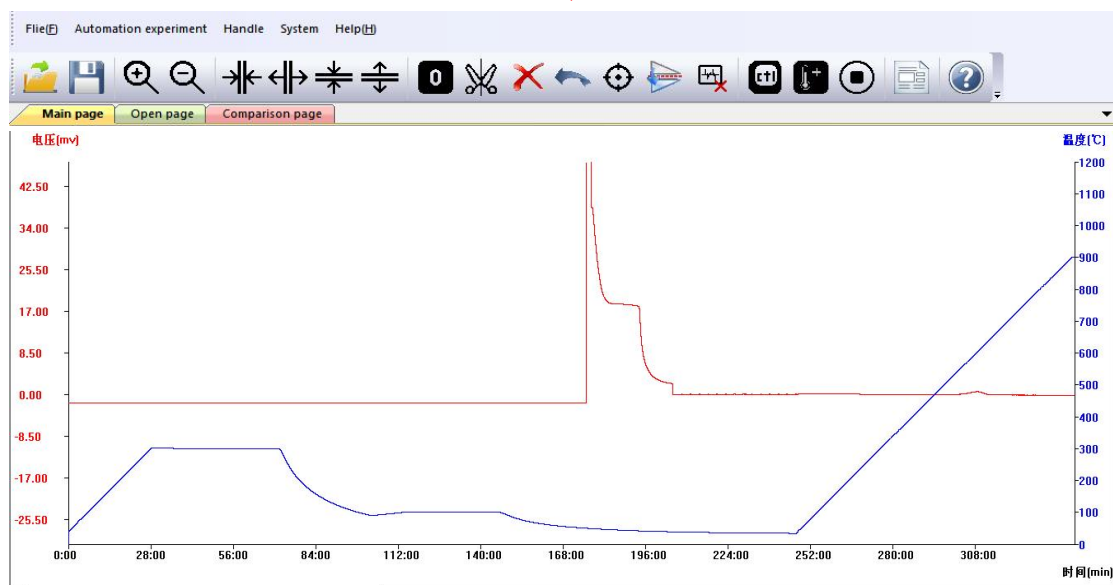
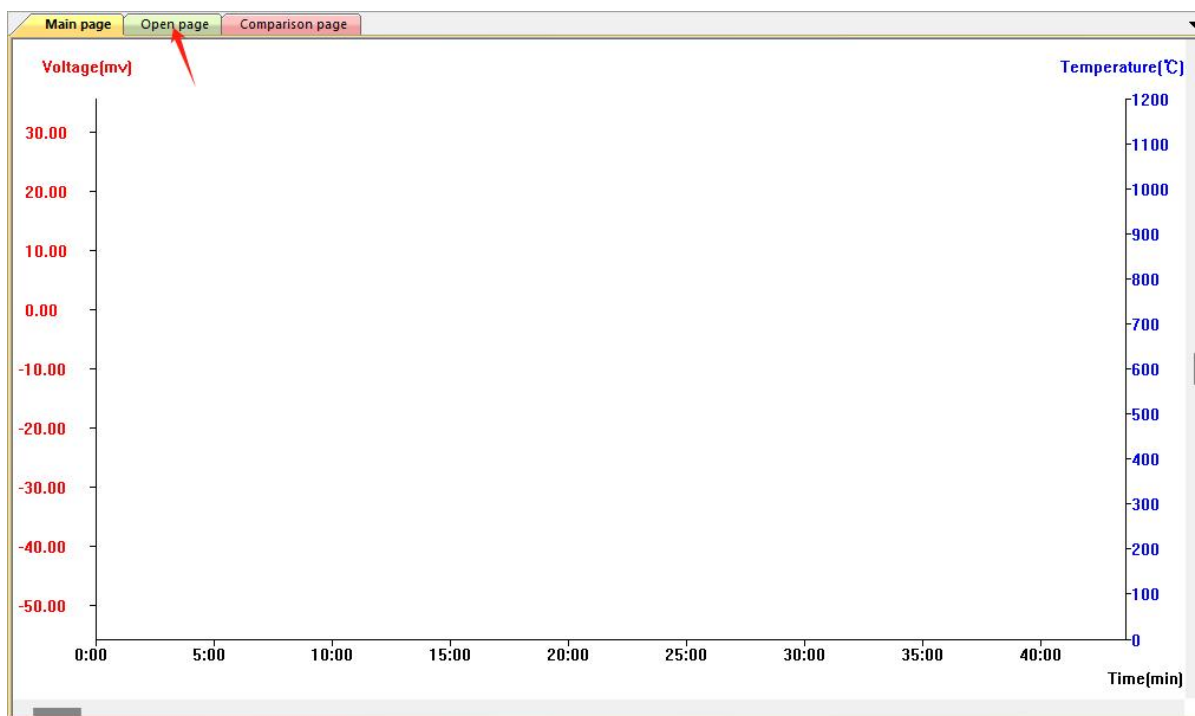
After the program heating program is completed, save the data, close the TCD thermal conductivity cell, turn off the gas, turn off the chemical adsorption instrument host, recover or dispose of the sample, and load the analysis position into a clean reactor.



## 4.data processing

### 4.1 Open the map

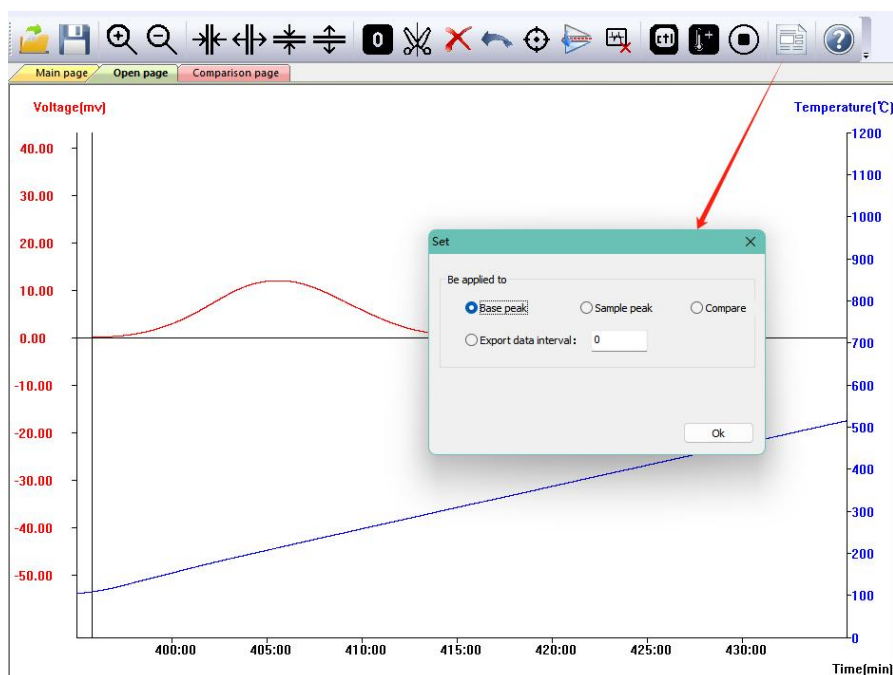
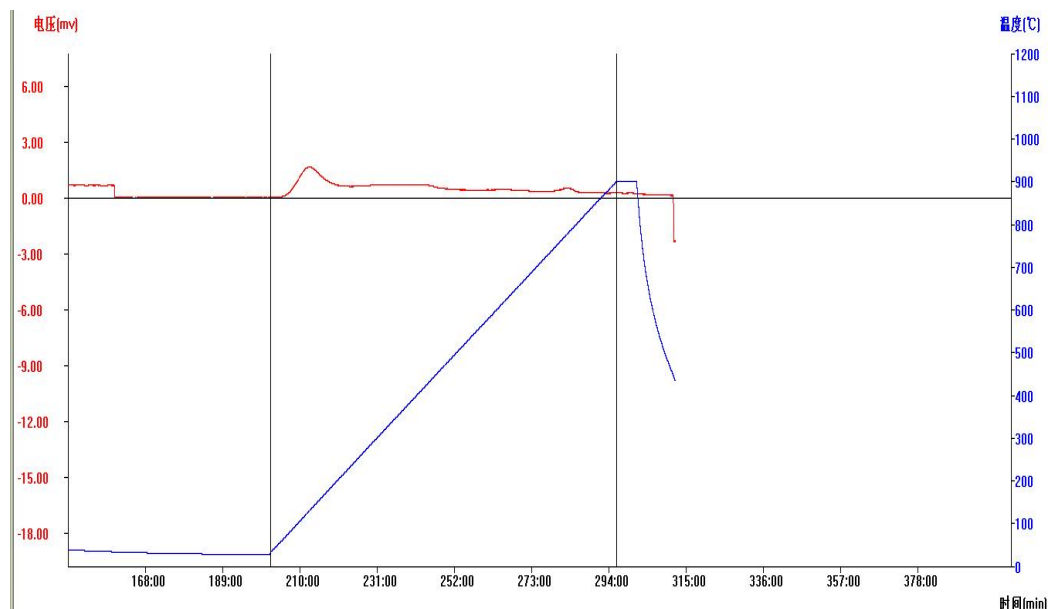
Click to switch to the open page, click on the open icon, find the file that needs to be processed, and open it.



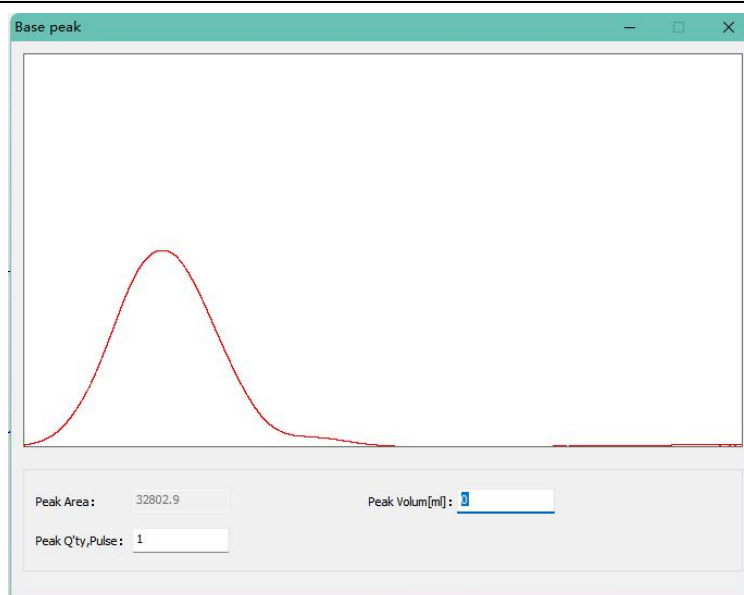
### 4.2 data processing



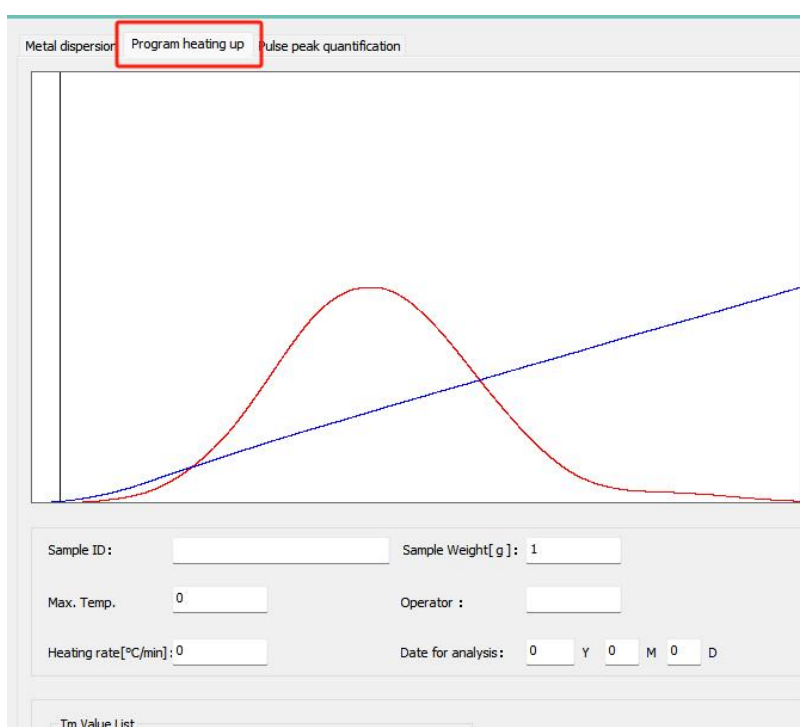
Click the peak cutting button to identify the required desorption segment map, then click the report button to pop up the data usage window.



- a) Quantitative: Integrating the recognition graph can obtain the peak area, as shown in the figure below.

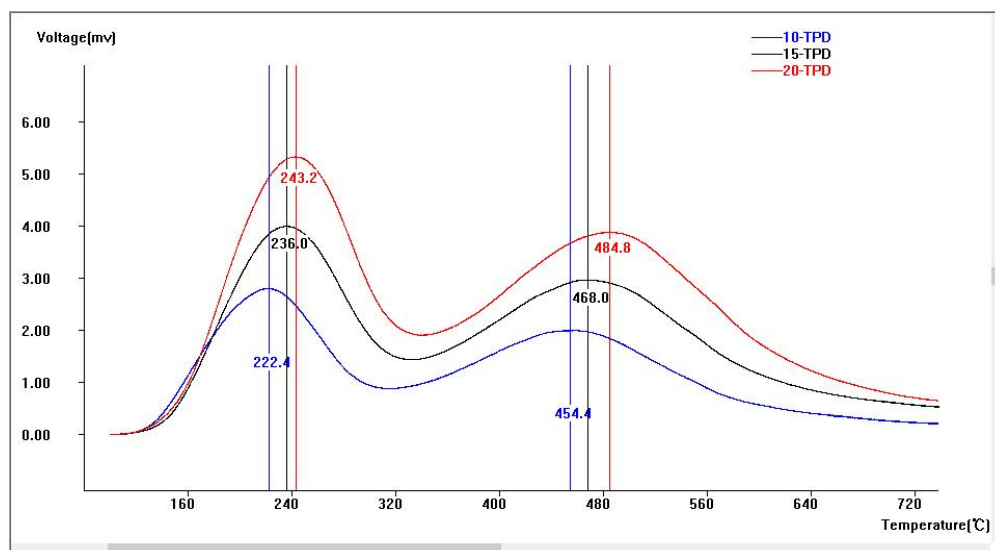
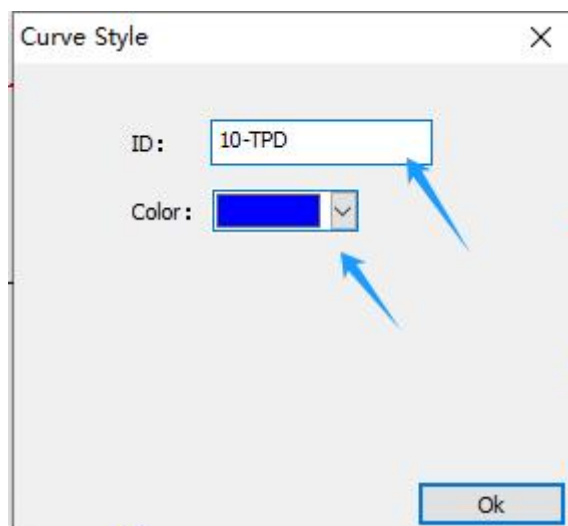
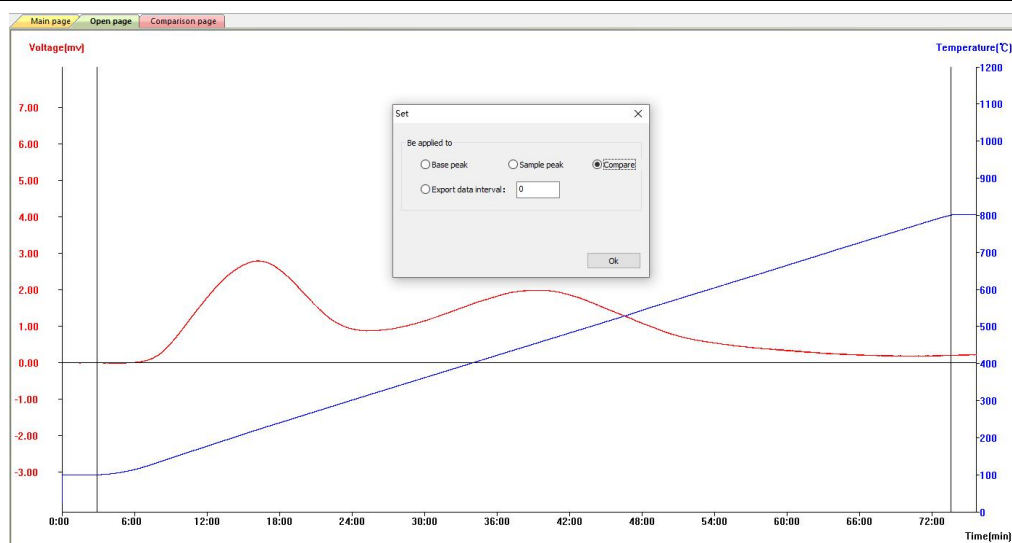


② Sample: It can generate "adsorption report", "temperature rise report", and "desorption report", and output different report types of graphs and data according to different needs.



④ Comparison: Different graph data can be added to the comparison window.







## (2) Programmed Temperature Reduction (H2-TPR)

### 1.Preparation Before the Experiment

1.1 Connect the carrier gas 1 (valve 1) interface to Ar/H<sub>2</sub> or N<sub>2</sub>/H<sub>2</sub> mixture (H<sub>2</sub> 5% -5%); Process gas 1 (valve 3) interface connected to N<sub>2</sub> or Ar.

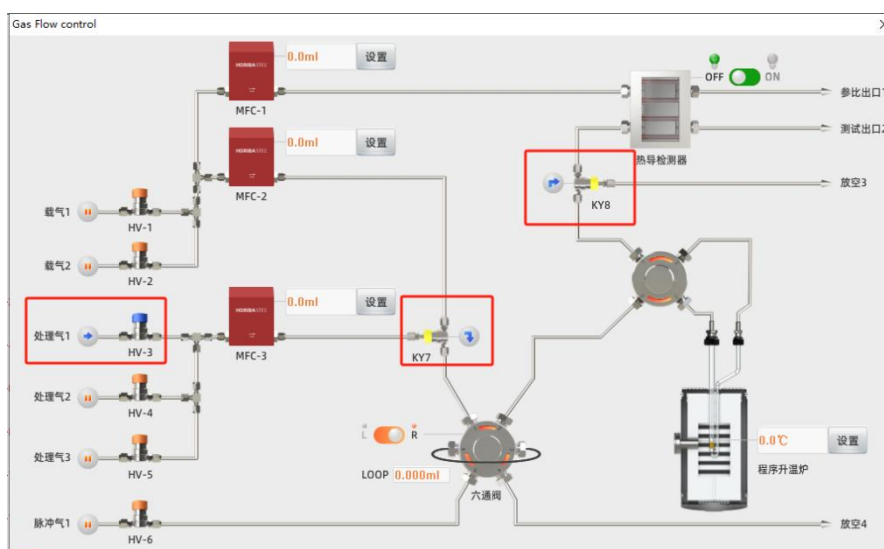
2.2 Fill the bed layer, load the sample into the reactor, about 0.05-0.5g (experimental temperature < 600 °C, use a glass reactor; < 1000 °C, use a quartz reactor), and connect to the instrument interface.

### 2.Conduct Experiments

#### 2.1 Remove Residual Gases from the Gas Path

(1) Open the host, software, and N<sub>2</sub> gas cylinder valve (adjust the output pressure to 0.3-0.4 MPa).

(2) Adjust the air circuit: Control the air circuit, adjust the direction of valve 3/valve 7/valve 8, and close the window.



(3) Adjust flow rate: Select nitrogen in channel 3 # and set the flow rate to 30ml/min.

(4) Blow the sample under normal temperature gas environment for at least 10 minutes.

#### 2.2 Programmed Heating Pretreatment (Dehydration)

(1) Raise the furnace and insert the thermocouple into the bottom of the reactor branch pipe

(2) Setting conditions: Multiple program heating settings can be performed, and relevant parameters can be set according to experimental conditions: program heating → set temperature upper limit, heating rate, and constant temperature time → add → click to start heating → close the interface

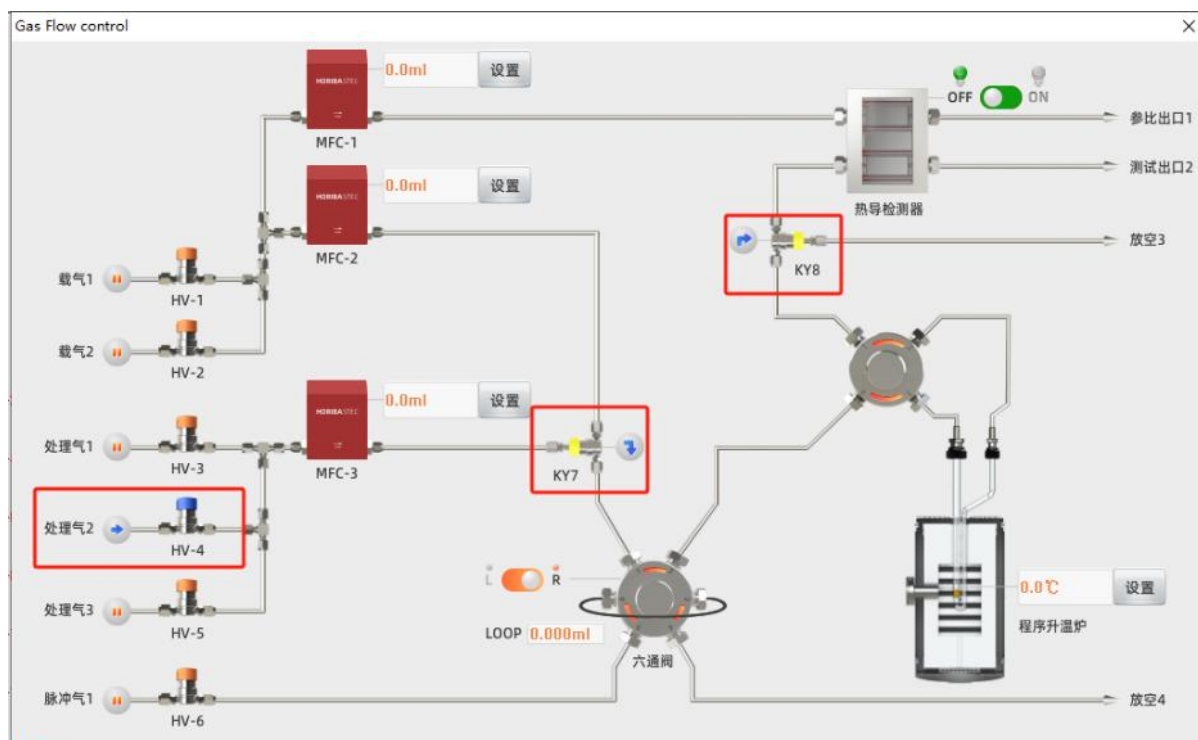


Heating section	Temp.(°C)	Heating rate(°C/min)	Time
1	300	10	60

(3) After the program heating program is completed, when the furnace temperature drops below 200 °C, lower the furnace (to avoid thermocouple damage), continue to use N2 for blowing, and proceed to the next step after the temperature at the sample location returns to room temperature.

Optional steps: The sample needs to be reduced according to its condition. The steps are as follows

- (1) Connect O2 to the processing gas 2 interface, open the O2 cylinder valve (adjust the output pressure to 0.3-0.4 MPa)
- (2) Adjusting the air circuit: Air circuit control, valve 4/valve 7/valve 8 pointing to switch, closing the window



(3) Adjust flow rate: Select oxygen in channel 3 # and set the flow rate to 30ml/min.

(4) Raise the furnace and insert the thermocouple into the bottom of the reactor branch pipe.

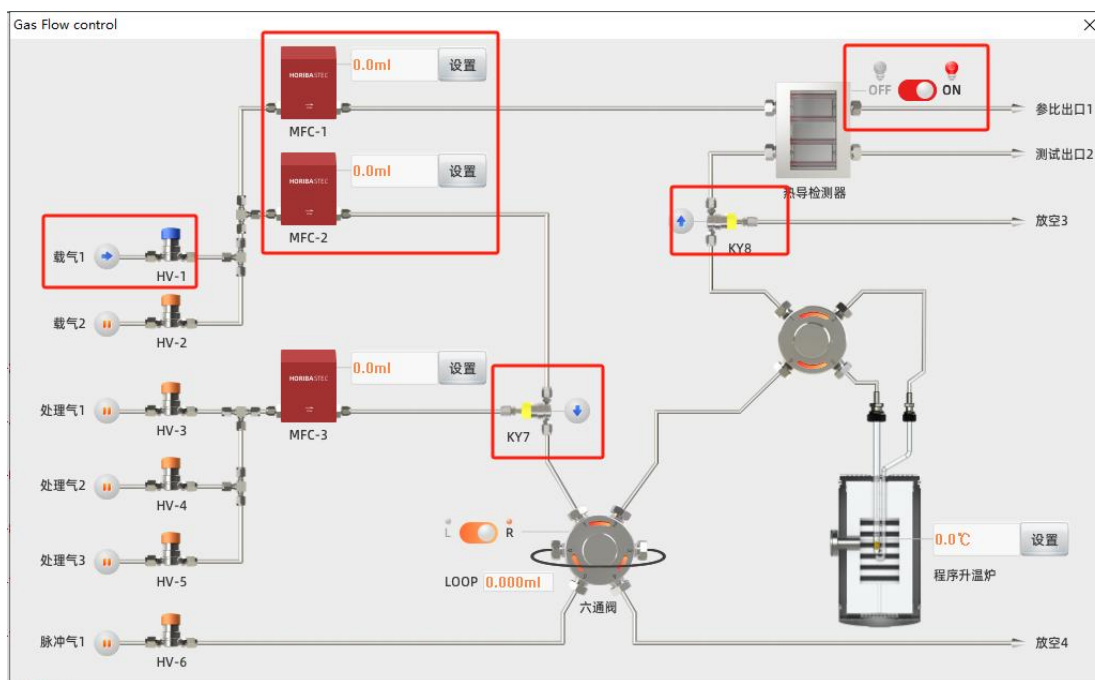
(5) Setting conditions: Multiple program heating settings can be performed, and relevant parameters can be set according to experimental conditions: program heating → set temperature upper limit, heating rate, and constant temperature time → add → click to start heating → close the interface.

(6) After the program heating program is completed, when the furnace temperature drops below 200 °C, lower the furnace (to avoid thermocouple damage), continue to use O<sub>2</sub> for blowing, and switch to treatment gas 1 after the sample position temperature returns to room temperature. After using N<sub>2</sub> to blow the gas path for 10 minutes, the next operation can be carried out.

### 2.3 Program Heating Reduction

(1) Open the valve of the Ar/H<sub>2</sub> mixed gas cylinder (adjust the output pressure to 0.3-0.4 MPa).

(2) Adjust the air circuit: Control the air circuit, adjust the direction of valve 1/valve 7/valve 8.



(3) Adjust flow rate: Channel 1 # is set to argon gas with a flow rate of 30ml/min, Channel 2 # is set to argon gas with a flow rate of 30ml/min, complete and close the window, and open TCD

Here, channels 1 # and 2 # refer to the instrument carrier gas channel (1 # is the reference port flow rate; 2 # is the test port channel)

Attention: When using a two-component mixture, after selecting components A and B, it is necessary to correctly input the percentage ratio corresponding to the gas component, and the A and B components set in channel 1 # and channel 2 # should be the same.

(4) Adjust the polarity according to the TCD polarity, turn on the TCD, wait for the baseline to level (multiple manual zeros are required during the stabilization process to obtain a stable baseline), raise the furnace to the designated position (due to the previous heating operation, there may be residual temperature in the furnace body, and the strong cooling system usually takes 30-40 minutes for the furnace body to cool down to below 50 °C. After the temperature stabilizes, zero the baseline and proceed to the next step).



(5) Setting conditions: Program heating → Set temperature upper limit, heating rate, and constant temperature time → Add to temperature control section → Click to start heating → Close the interface.

Heating section	Temp.(°C)	Heating rate(°C/min)	Time
1	900	10	5

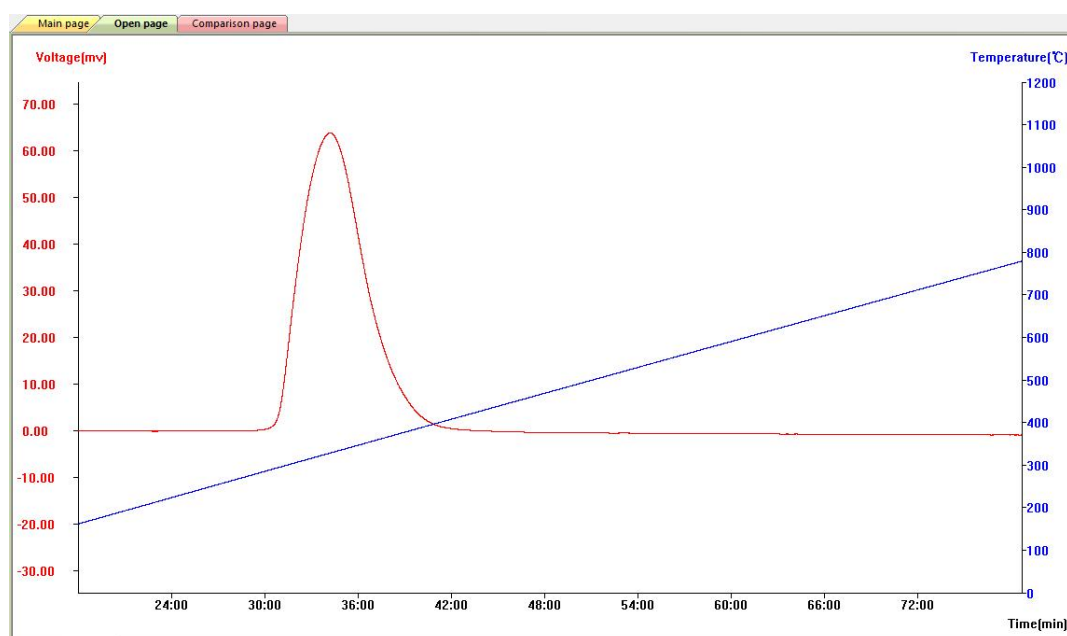
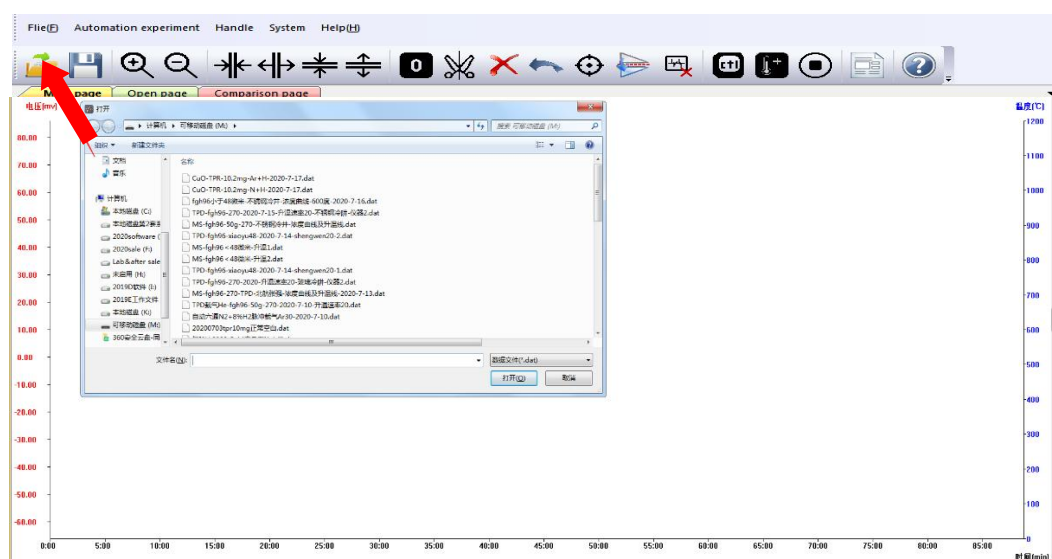
### 3.Experimental Completion

After the program heating program is completed, save the data, close the TCD thermal conductivity cell, turn off the gas, turn off the chemical adsorption instrument host, recover or dispose of the sample, and load the instrument analysis position into a clean reactor.

### 4.Data Processing

#### 4.1 Open the map

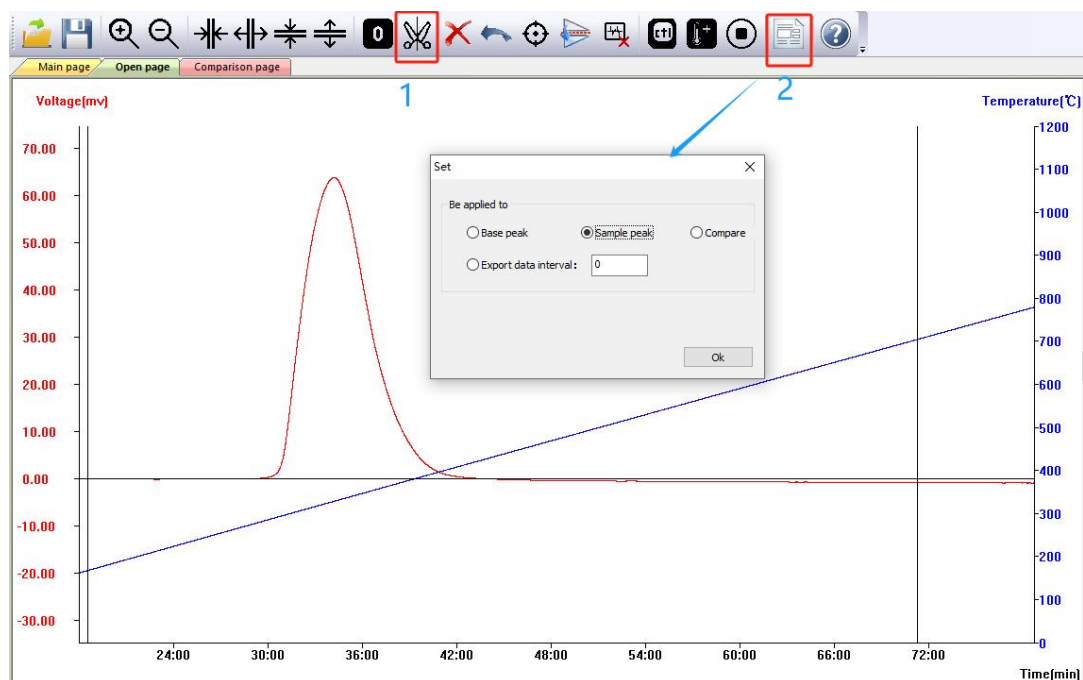
Click to switch to the open page, click on the open icon, find the file that needs to be processed, and open it.



## 4.2data processing

Click the peak cutting button to identify the desired desorption segment map, then click the report button to pop up a data usage window. According to the requirements, the map can be processed for "quantitative", "sample", and "comparison". The operation method can be found in NH<sub>3</sub>-TPD data processing.





### (3) Temperature Programmed Oxidation (O2-TPO)

#### 1.Preparation before the Experiment

1.1 Connect the interface of carrier gas 1 (valve 1) to He/O<sub>2</sub> mixture (O<sub>2</sub> is 5% -10%); Process gas 1 (valve 3) interface connected to N<sub>2</sub> or Ar.

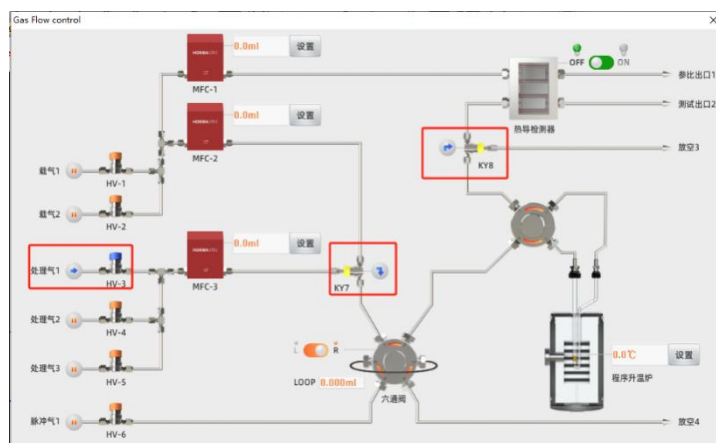
1.2 Fill the bed layer, load the sample into the reactor, about 0.05-0.5g (experimental temperature < 600 °C, use a glass reactor; < 1000 °C, use a quartz reactor), and connect to the instrument interface.

#### 2.Conduct Experiments

##### 2.1 Remove Residual Gases from the Gas Path

(1) Open the host, software, and N<sub>2</sub> gas cylinder valve (adjust the output pressure to 0.3-0.4 MPa).

(2) Adjust the air circuit: Control the air circuit, adjust the direction of valve 3/valve 7/valve 8, and close the window.





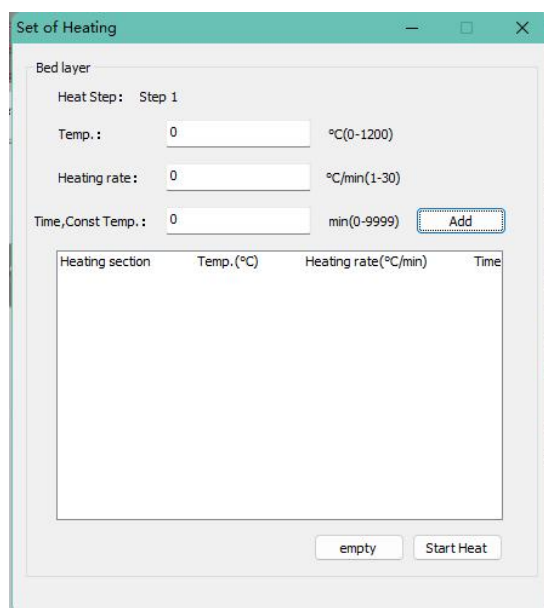
(3) Adjust flow rate: Select nitrogen in channel 3 # and set the flow rate to 30ml/min.

(4) Blow the sample under normal temperature gas environment for at least 10 minutes.

## 2、Programmed Heating Pretreatment (Dehydration)

(1) Raise the furnace and insert the thermocouple into the bottom of the reactor branch pipe.

(2) Setting conditions: Multiple program heating settings can be performed, and relevant parameters can be set according to experimental conditions: program heating → set temperature upper limit, heating rate, and constant temperature time → add → click to start heating → close the interface.

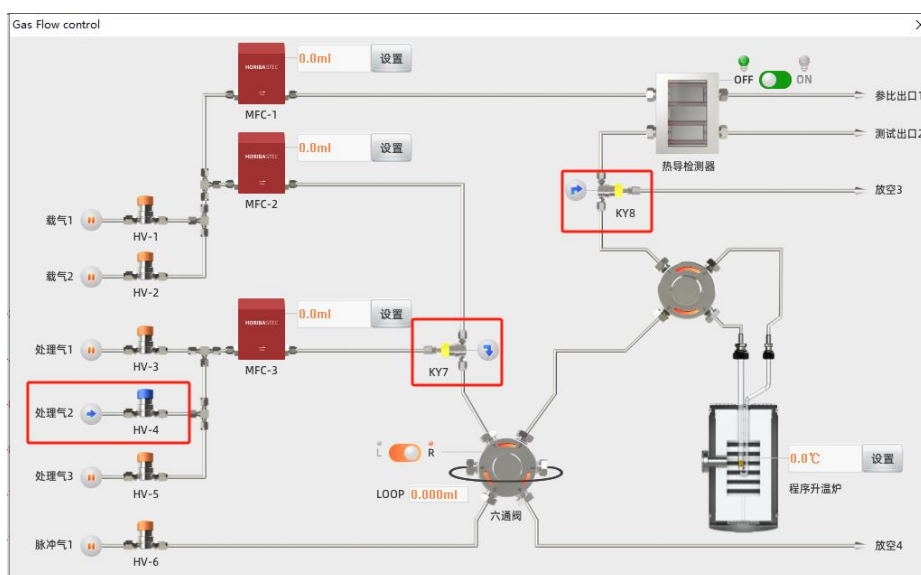


(3) After the program heating program is completed, when the furnace temperature drops below 200 °C, lower the furnace (to avoid thermocouple damage), continue to use N2 for blowing, and proceed to the next step after the temperature of the sample position returns to room temperature (generally, the strong cooling system requires 30-40 minutes for the furnace body to cool down to below 50 °C).

Optional steps: The sample needs to be reduced according to its condition. The steps are as follows

(1) Connect H2 to the processing gas 2 interface and open the H2 cylinder valve (adjust the output pressure to 0.3-0.4 MPa)

(2) Adjusting the air circuit: Air circuit control, regulating valve 4/valve 7/valve 8 pointing, closing the window



(3) Adjust flow rate: Channel 3 # is set to hydrogen gas, and the flow rate is set to 30ml/min

(4) Raise the furnace and insert the thermocouple into the bottom of the reactor branch pipe

(5) Setting conditions: Multiple program heating settings can be performed, and relevant parameters can be set according to experimental conditions: program heating → set temperature upper limit, heating rate, and constant temperature time → add temperature control section → click to start heating → close the interface

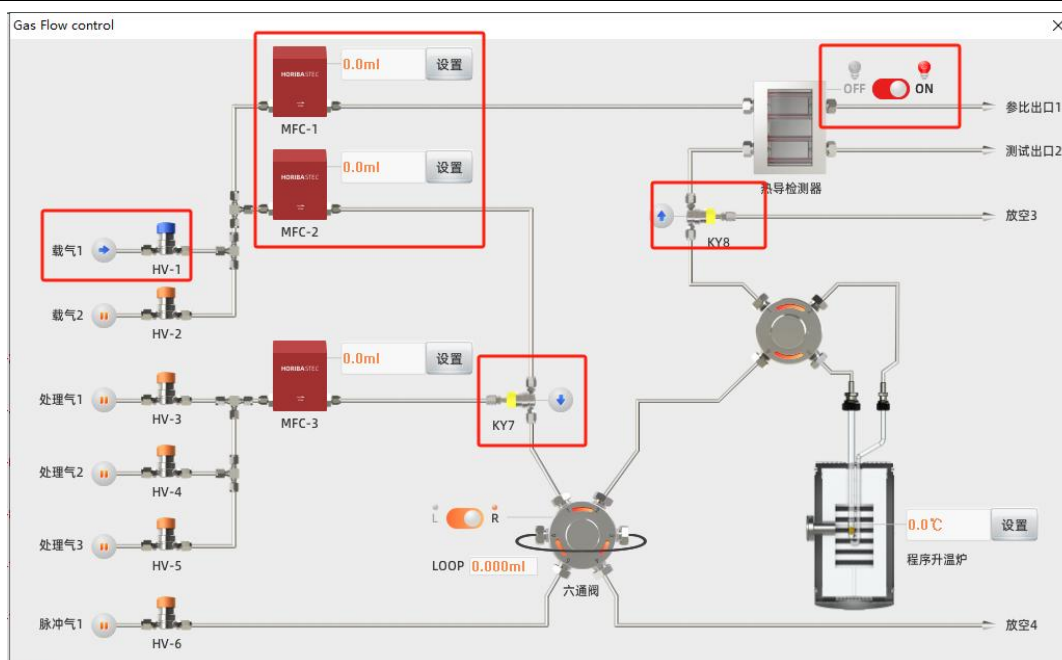
(6) After the program heating program is completed, when the furnace temperature drops below 200 °C, lower the furnace (to avoid thermocouple damage), continue to use H<sub>2</sub> for blowing, and switch to treatment gas 1 after the sample position temperature returns to room temperature. After using N<sub>2</sub> to blow the gas path for 10 minutes, the next operation can be carried out.

### 2.3 Programmed Heating Oxidation

(1) Open the valve of the He/O<sub>2</sub> mixed gas cylinder (adjust the output pressure to 0.3-0.4 MPa)

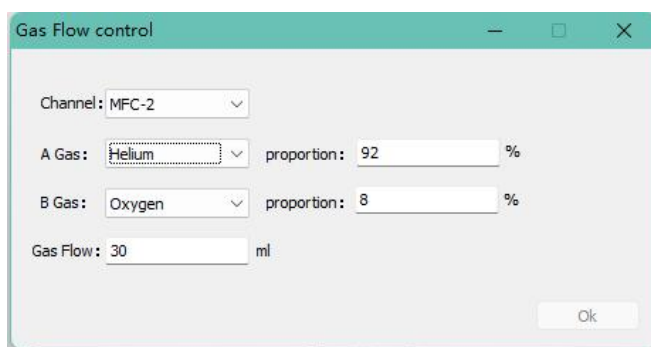
(2) Adjusting air circuit: Air circuit control, regulating valve 1/valve 7/valve 8 pointing.

(3) Adjust flow rate: Channel 1 # is set to helium oxygen mixture with a flow rate of 30ml/min, Channel 2 # is set to helium oxygen mixture with a flow rate of 30ml/min, complete and close the window, opening TCD, closing window.



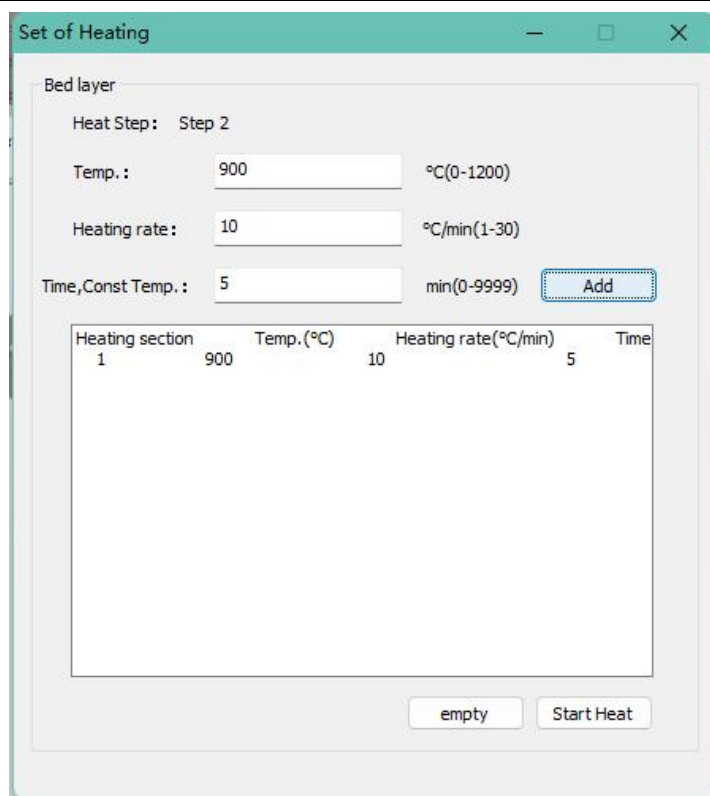
Here, channels 1 # and 2 # refer to the instrument carrier gas channel (1 # is the reference port flow rate; 2 # is the test port channel)

Note: When using a two-component mixture, after selecting components A and B, it is necessary to correctly input the percentage ratio corresponding to the gas component, and the A and B components set in channel 1 # and channel 2 # should be the same.



(4) Adjust the polarity according to the TCD polarity, turn on the TCD, wait for the baseline to level (multiple manual zeros are required during the stabilization process to obtain a stable baseline), and raise the furnace to the designated position (due to the previous heating operation, there may be residual temperature in the furnace body, and the strong cooling system usually takes 30-40 minutes for the furnace body to cool down to below 50 °C. After the temperature stabilizes, zero the baseline and proceed to the next step).

(5) Setting conditions: Program heating → Set temperature upper limit, heating rate, and constant temperature time → Add temperature control section → Click to start heating → Close the interface



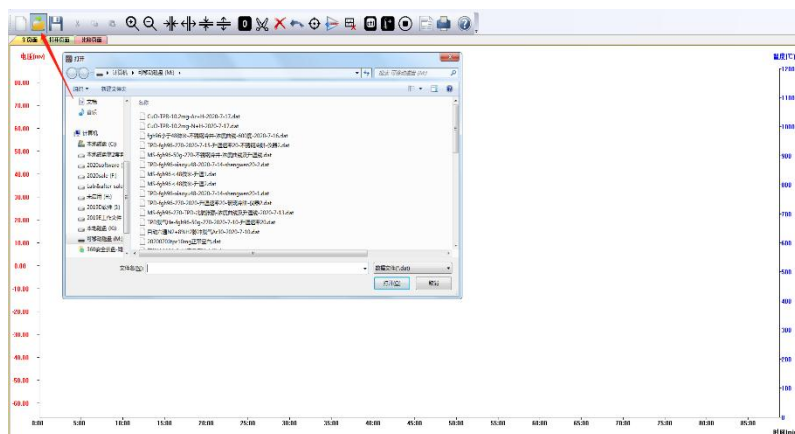
### 3.Experimental completion

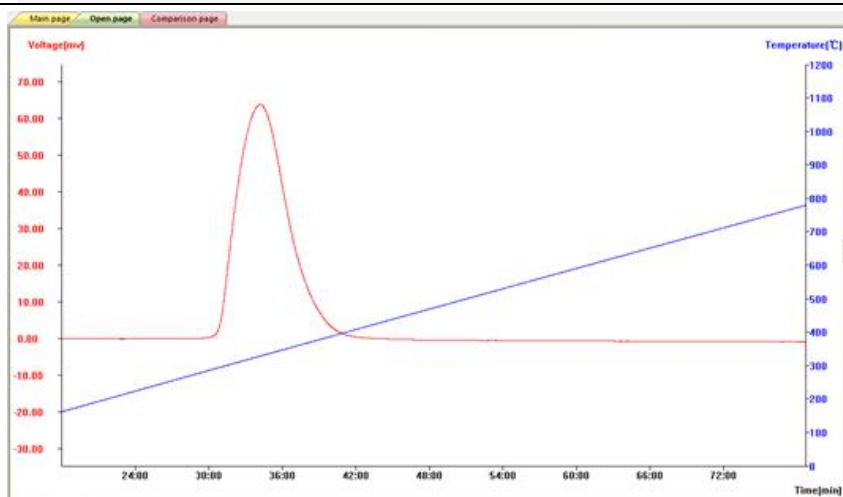
After the program heating program is completed, save the data, close the TCD thermal conductivity cell, turn off the gas, turn off the chemical adsorption instrument host, recover or dispose of the sample, and load the analysis position into a clean reactor.

### 4.data processing

#### 4.1Open the map

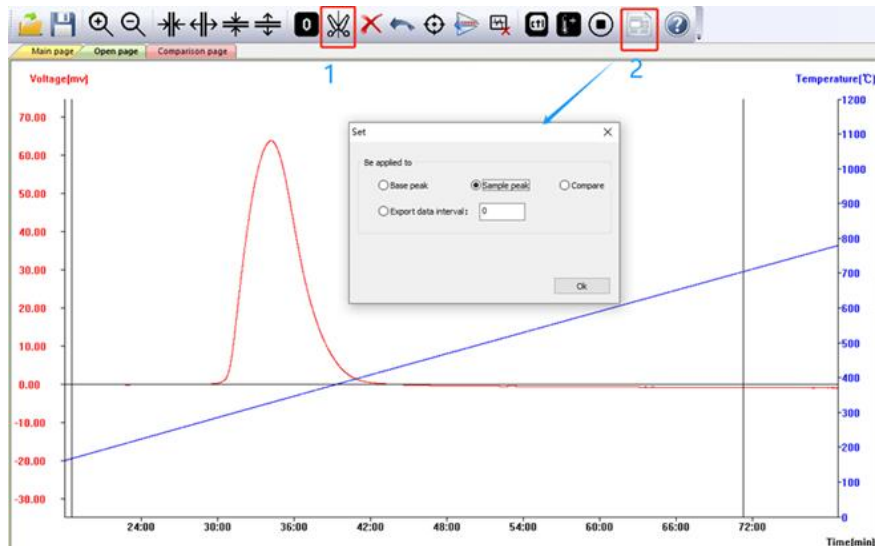
Click to switch to the open page, click on the open icon, find the file that needs to be processed, and open it





## 4.2 data processing

Click the peak cutting button to identify the desired desorption segment map, then click the report button to pop up a data usage window. According to the requirements, the map can be processed for "quantitative", "sample", and "comparison". The operation method can be found in NH3-TPD data processing.



## (4) Pulse H2-O2 Injection

### 1.Preparation before the Experiment

1.1 Connect the carrier gas 1 (valve 1) interface to Ar; Process gas 1 (valve 3) interface connected to O2; Process gas 2 (valve 4) interface connected to H2; Pulse gas 1 (valve 6) interface is connected to H2.

1.2 Fill the bed layer, load the sample into the reactor, about 0.05-0.5g (experimental temperature < 600 °C, use a glass reactor; < 1000 °C, use a quartz reactor), and connect to the

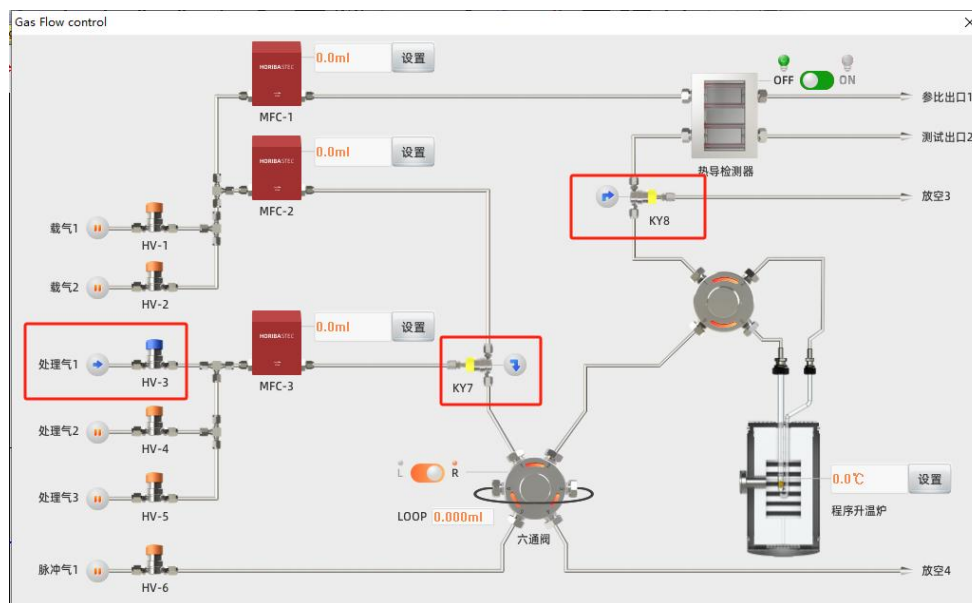


instrument interface.

## 2. Conduct Experiments

### 2.1 Remove residual gases from the gas path

- (1) Open the host, software, and Ar bottle valve (adjust the output pressure to 0.3-0.4 MPa)
- (2) Adjusting the air circuit: Air circuit control, regulating valve 1/valve 7/valve 8 pointing, closing the window



- (3) Adjust flow rate: Channel 2 # is set to argon gas, and the flow rate is set to 30 ml/min. Here, channels 1 # and 2 # refer to the instrument carrier gas channel (1 # is the reference port flow rate; 2 # is the test port channel)

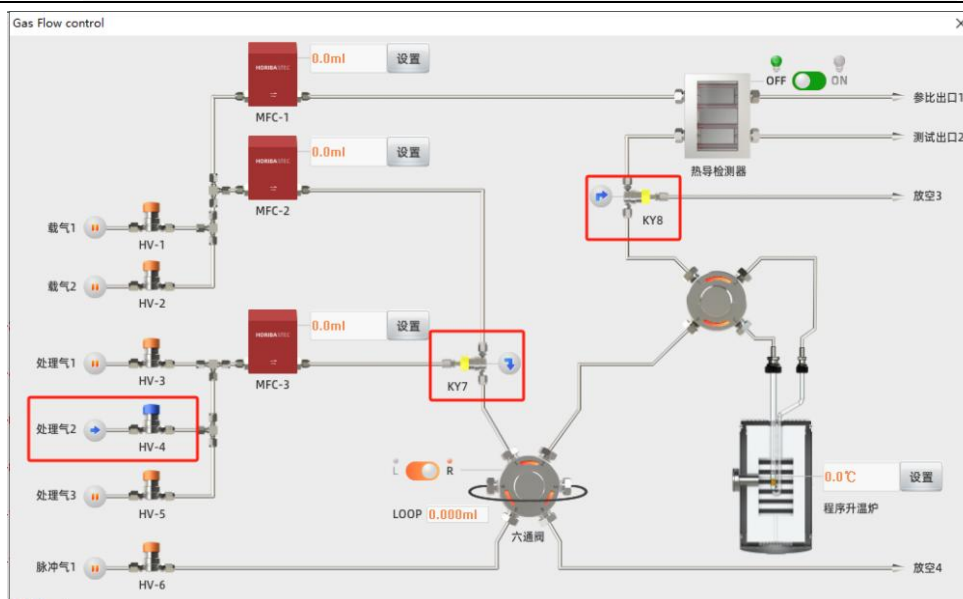
*Note that if it is a single component gas, after selecting gas A, the proportion should be set to 100%, and the proportion of gas B should be set to 0%. Click OK to proceed; If it is a two-component mixture, after selecting components A and B, it is necessary to correctly input the percentage ratio corresponding to that gas component.*

- (4) Blow the sample in an inert gas environment at room temperature for at least 10 minutes.

### 2. Program heating and H<sub>2</sub> reduction

- (1) Raise the furnace to the designated position, insert the thermocouple into the bottom of the reactor branch pipe, open the H<sub>2</sub> bottle valve, set channel 3 # to hydrogen, and set the flow rate to 30ml/min.
- (2) Adjust the air circuit: close valve 1, adjust valve 4/valve 7/valve 8 to point, and close the window.

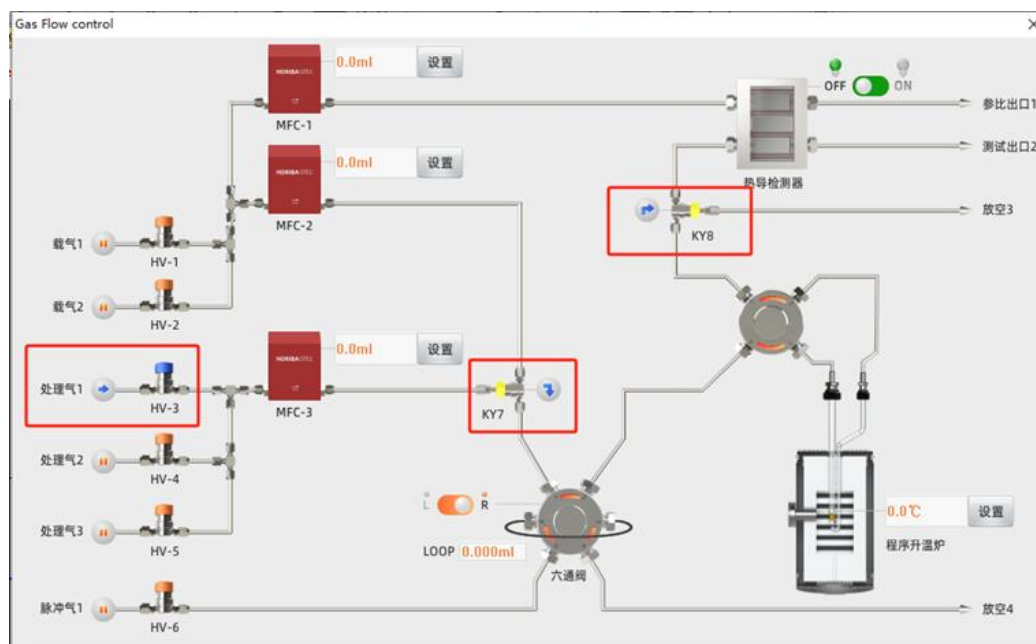




(3) Set program heating conditions: program heating → set temperature upper limit, heating rate, and constant temperature time → add temperature control section → click to start heating → close the interface.

(4) After the program heating program is completed, when the furnace temperature drops below 200 °C, lower the furnace (to avoid thermocouple damage), continue to use Ar gas for blowing, and proceed to the next step after the temperature of the sample position returns to room temperature (generally, the strong cooling system requires 30-40 minutes for the furnace body to cool down to below 50 °C).

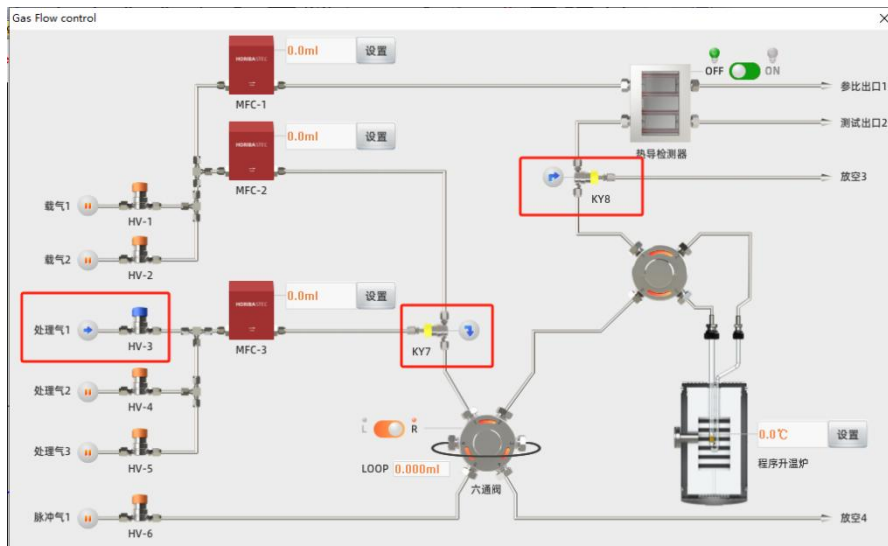
(5) Remove H<sub>2</sub> from the gas path: Close valve 3, adjust valve 1/valve 7/valve 8 to point, and blow Ar gas for at least 10 minutes.





### 2.3 At a certain temperature, saturated adsorption of O<sub>2</sub>

(1) Adjust the air circuit: close valve 1, adjust valve 3/valve 7/valve 8 to point, and close the window.



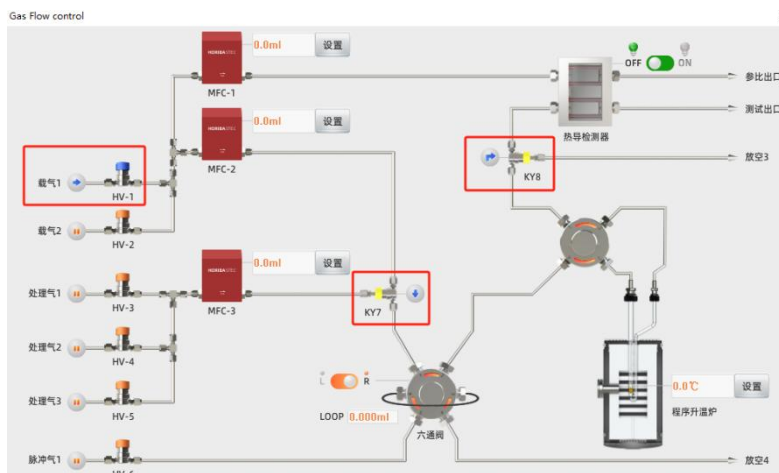
(2) Set O<sub>2</sub> adsorption temperature:

① Set temperature: Program heating → Set upper temperature limit, heating rate, and constant temperature time → Add temperature control section → Click to start heating → Close the interface.

② When the set temperature is reached, open the O<sub>2</sub> bottle valve, set channel 3 # to oxygen, set the flow rate to 30mL/min, and allow pure O<sub>2</sub> to adsorb for about 30min.

(3) After the program heating program is completed, when the furnace temperature drops below 200 °C, lower the furnace and continue to use Ar gas for blowing. After the temperature of the sample position returns to room temperature, the next operation can be carried out.

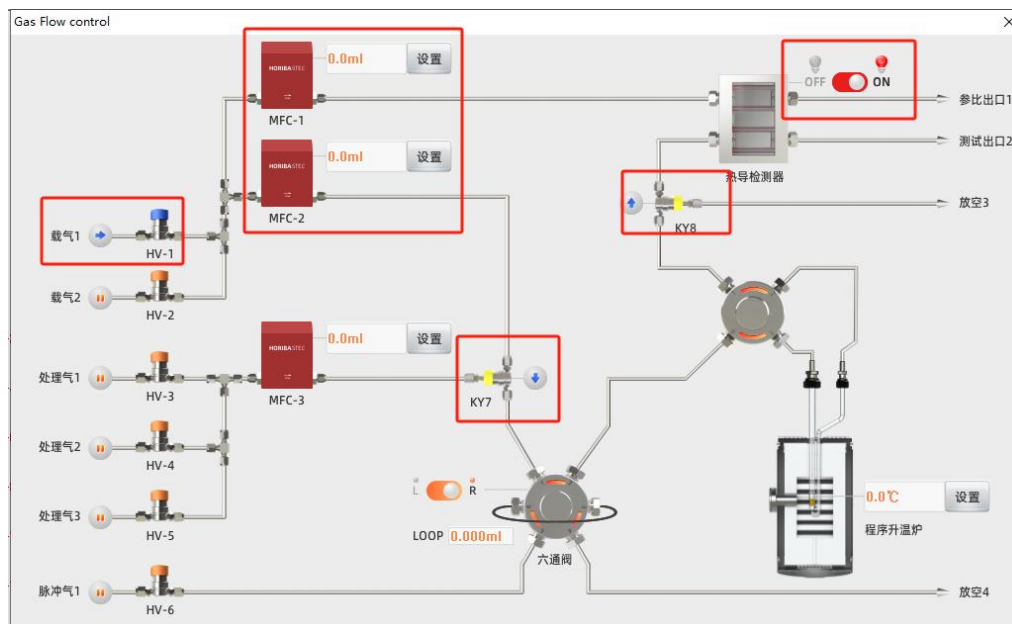
(4) Remove O<sub>2</sub> from the gas path: Close valve 3, adjust valve 1/valve 7/valve 8 to point, and blow Ar gas for at least 10 minutes.





## 2.4 Pulse H<sub>2</sub> titration at a certain temperature

(1) Adjust the air circuit: Adjust valve 1/valve 7/valve 8 to point and open TCD.



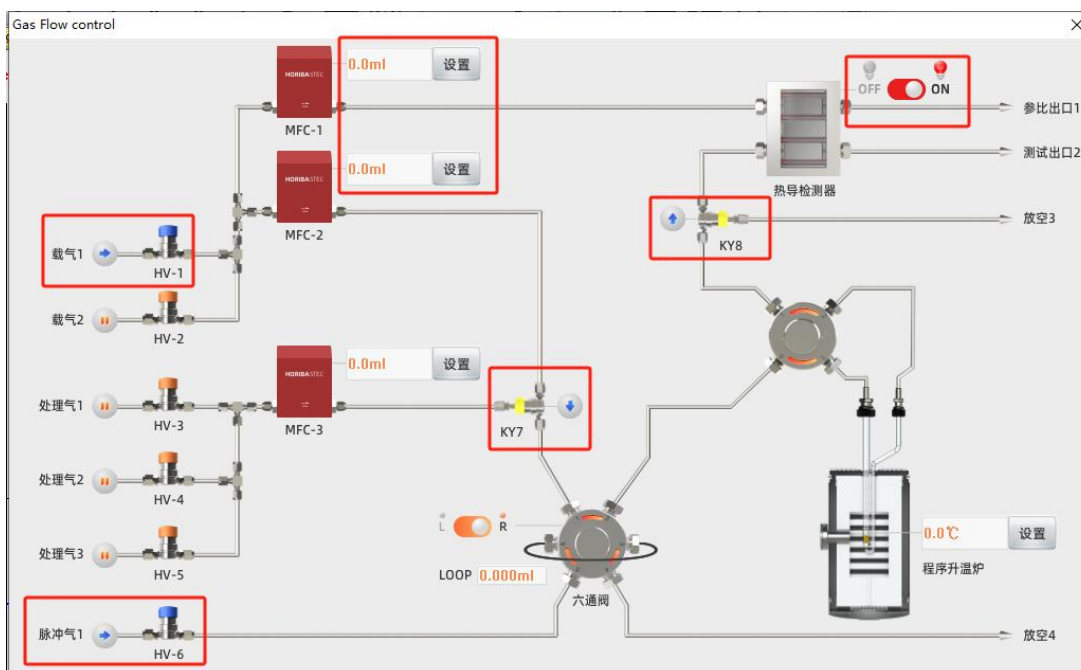
(2) Adjust flow rate: Channel 1 # is set to argon gas with a flow rate of 30ml/min, Channel 2 # is set to argon gas with a flow rate of 30ml/min, complete and close the window.

(3) Raise the furnace to the designated position.

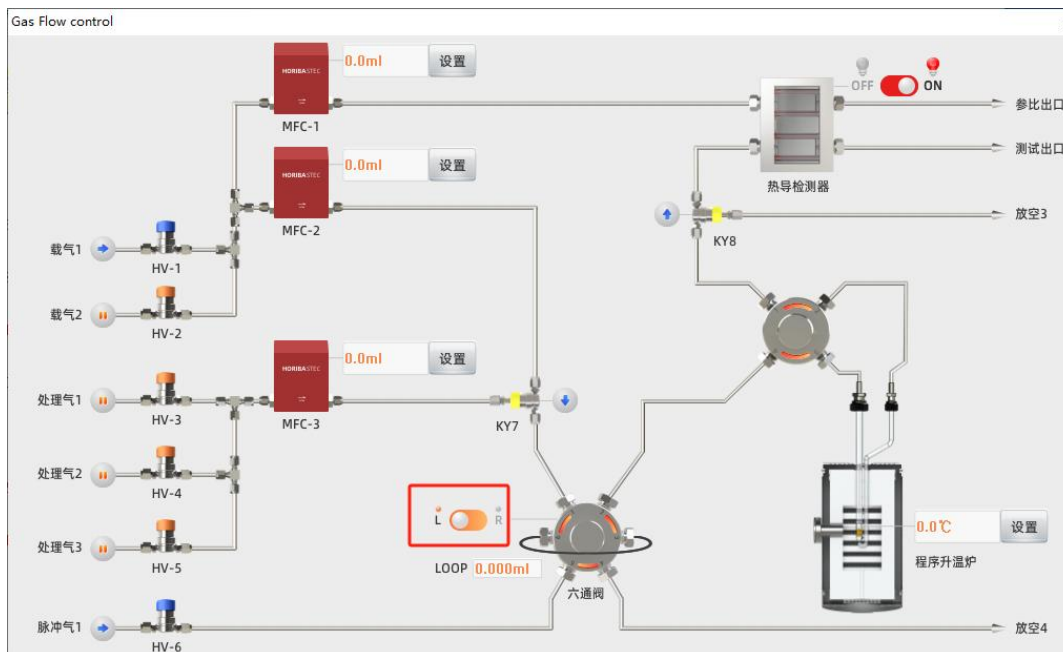
(4) Set H<sub>2</sub> titration temperature: program heating → set upper temperature limit, heating rate, and constant temperature time → add temperature control section → click to start heating → close the interface.

(5) **When the H<sub>2</sub> titration temperature is reached, adjust the polarity of TCD to start TCD, and stabilize the baseline leveling (manual zeroing is required for many times during the stabilization process).**

(6) Adjust the gas path: Maintain the current gas path, open valve 6, adjust the pulse rotor flowmeter to 40ml/min, and stabilize for 10 minutes.



(7) Pulse titration: inject the first H<sub>2</sub> pulse with the six way valve, adjust the zero point after the baseline is leveled, and inject the second H<sub>2</sub> pulse... until the last pulse peak and the peak area of the previous pulse peak are the same.



### 3.Experimental Ended

Save experimental data, close the TCD thermal conductivity cell, close the gas cylinder valve, close the chemical adsorption instrument host, recover or dispose of samples, and load the analysis position into a clean reactor.



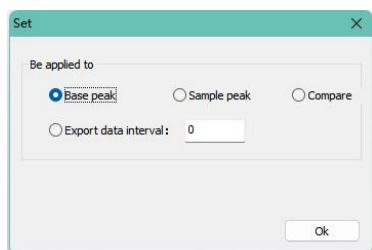
## 4.Data Processing

### 4.1 Open the application

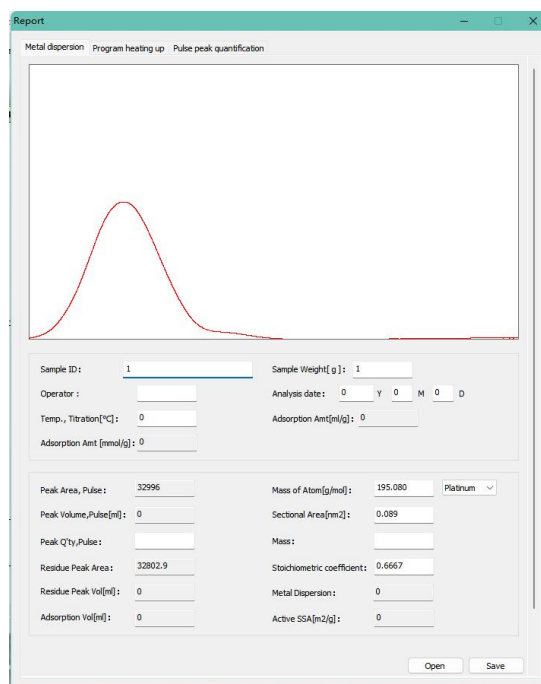
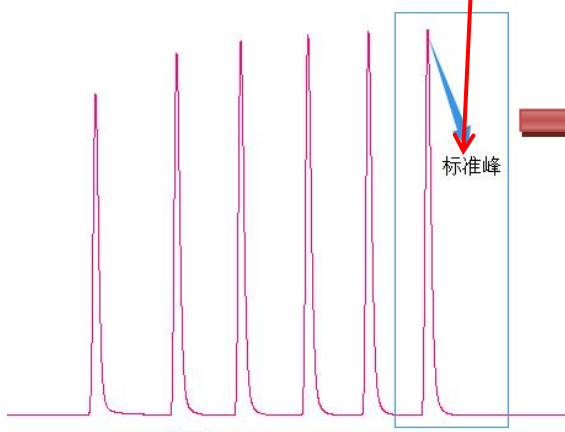
Click to switch to the open page, click on the open icon, find the file that needs to be processed, and open it.

### 4.2 data processing

Click the peak cutting button to identify the desired pulse peak spectrum, then click the report button to pop up a data usage window. According to the requirements, the spectrum can be processed for "quantitative", "sample", and "comparison". The operation method can be found in NH3-TPD data processing.



Standard peak



## 5. Data Analysis

This device analysis software provides a data analysis module for characterizing catalyst indicators such as metal dispersion, active surface area, and average grain size. For specific experimental data acquisition and calculation applications, please refer to the following instructions:



## 1. Metal dispersion:

1) Definition: Metal dispersion refers to the ratio of the number of active metal atoms on the catalyst surface to the total number of metal atoms in the catalyst.

2) Application formula:

$$D = \frac{n \times V_g \times M_M}{W \times P \times 22414} \quad (1)$$

In the formula:

D : D: Catalyst metal dispersion;

n : Analyzing the stoichiometric coefficients of gases during actual reaction processes (for the stoichiometric formula of hydrogen oxygen titration, different literature provides different results. For example, when measuring the chemical adsorption of oxygen on Pt by hydrogen titration, one oxygen atom on Pt consumes two hydrogen atoms and reacts to produce water, which is adsorbed on the carrier); Meanwhile, due to the fact that Pt atoms themselves need to adsorb a hydrogen atom after deoxidation, the amount of hydrogen used to titrate the oxygen atoms adsorbed on Pt is only 2/3 of the total hydrogen consumption. That is, if the number of surface Pt atoms is 2/3 of the number of H<sub>2</sub> molecules consumed, the stoichiometric coefficient is taken as 2/3. In fact, the choice of chemical stoichiometry and coefficients must be determined by supplementary experiments

M<sub>M</sub>: Relative atomic mass of catalyst metal, unit: g/mol (g/mol)

W : Mass of the tested sample, in grams

P : Mass fraction of active metals in catalyst, %

3) Calculation of V<sub>g</sub>:

$$V_g = \frac{NA_i - A_{II}}{A_i} \times V \quad (2)$$

In the formula:

V<sub>g</sub> Indicates the volume of analytical gas (H<sub>2</sub> or O<sub>2</sub>) consumed by the catalyst sample that has been completely oxidized (or reduced) during the experimental process, in milliliters;



Ai The chromatographic peak area caused by injecting an analytical gas pulse when unloaded (without sample) (please refer to the software instructions for peak cutting operations);

AII The chromatographic peak area caused by the overflow of the analytical instrument from the beginning of pulse titration to the saturation of titration (please refer to the software instructions for peak cutting operation);

V Instrument pulse quantitative tube volume, in milliliters, with built-in parameters of the instrument;

N The number of pulses injected from the beginning of pulse titration to the saturation of titration.

## 2.Active specific surface area:

1) Definition: The surface area of surface active metal atoms in a unit mass catalyst.

2) Application formula:

$$S = \frac{2 \times V_g \times N_0 \times \sigma_M}{W \times P \times 22414} \times 10^{-18}$$

3) In the formula:

S : Catalyst activity specific surface area, in units  $\text{m}^2/\text{g}$

$V_g$  : Indicates the volume of analytical gas ( $\text{H}_2$  or  $\text{O}_2$ ) consumed by the completely oxidized (or reduced) catalyst sample during the experimental process, in ml

$N_0$  :  $N_A$ ,  $6.02 \times 10^{23} / \text{mol}$

$\sigma_M$  : The cross-sectional area of catalyst metal atoms,  $0.089 \text{ nm}^2$

W : : Mass of the tested sample, in g

P : Mass fraction of active metals in catalyst, %

## 3. Average Grain Size

1) Definition: Average particle size of catalyst active metal (The average grain size of Pt is estimated based on the Hughes model. Its basic assumption is that all Pt grains are ideal, a cube of the same size, with one face in contact with the support and the other five faces exposed. The length d of one side of the cube is related to the surface area S, volume V, or density rho.)

2) Application formula:

$$d = \frac{5 \times 10^4}{\rho_M \times S} \quad (4)$$





In the formula:

$d$ : Average grain size of catalyst metal, unit:  $\text{\AA}$

$S$ : Catalyst activity specific surface area, in units  $\text{m}^2/\text{g}$

$\rho_M$ : Catalyst metal density, in units  $\text{g}/\text{cm}^3$

#### 4、Table of Common Catalytic Metal Parameters

No.	Metal Name	M (g/mol)	S (nm <sup>2</sup> )	$\rho$ (g/ml)
1	Pt	195.08	0.089	21.45
2	Pd	106.42	-	12.02
3	Re	186.207	-	21.04
4	Ru	101.07	-	12.37
5	Ni	58.69	-	8.9
6	Co	58.933	-	8.9
7	Fe	55.84	-	7.874
8	Ag	107.868	-	10.5
9	Cu	63.54	-	8.96
10	Zn	65.38	-	7.13

(Generally, only calculations are made for precious metals 1-4)

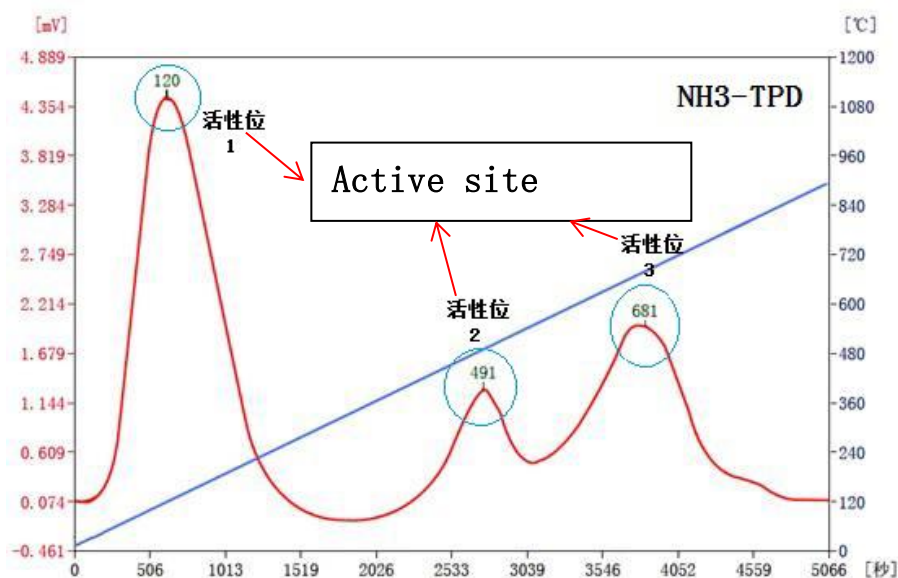


## Chapter 7 Experimental Analysis

### 1、Experimental Analysis of NH<sub>3</sub>-TPD (Temperature Programmed Desorption of Ammonia)

TPD (temperature programmed desorption)

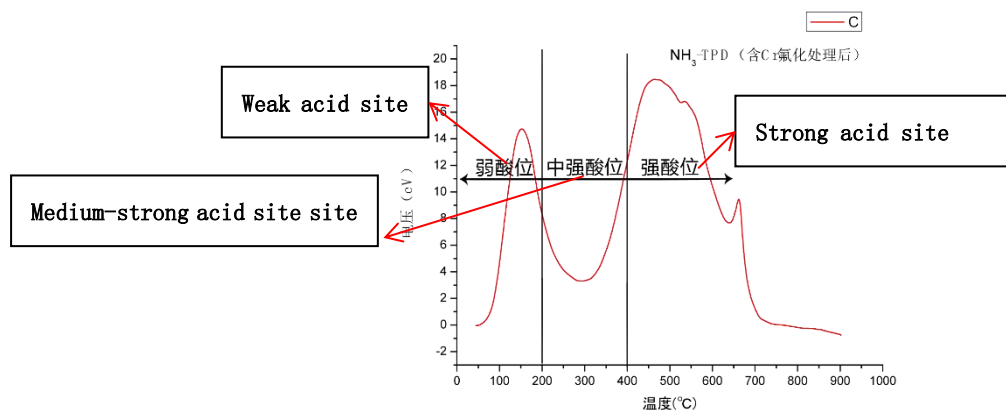
NH<sub>3</sub>-TPD is an experimental method for characterizing the acidity of catalysts. The experiment uses a carrier gas (usually an inert gas such as N<sub>2</sub> \ Ar \ He) under certain programmed heating conditions, passes through the catalyst bed, and uses a thermal conductivity (or mass spectrometry) detector to measure the concentration signals of NH<sub>3</sub> desorbed by the catalyst in the carrier gas at various temperature points (different acidic sites, adsorbents usually desorb at different temperatures). From the obtained TPD spectrum, information such as desorption peaks and temperature points can be obtained, such as the number of acidic sites, acidic center temperature, and acid strength. Acid value can also be obtained through different quantitative methods in the experiment.



There is a linear relationship between the adsorption force of NH<sub>3</sub> on the sample and the acidity of the adsorption sites. The stronger the acidity of the adsorption site, the greater the adsorption force of NH<sub>3</sub>, and the higher the temperature required for desorption. Characterize the acidity distribution of the sample by analyzing the NH<sub>3</sub> content in the desorption gas at different temperatures. In the field of catalysis, it is



generally believed that desorption peaks below 200 °C correspond to weak acid sites, desorption peaks in the temperature range of 200-400 °C correspond to medium strong acid sites, and desorption peaks above 400 °C correspond to strong acid sites.



How should NH<sub>3</sub>-TPD experiments be conducted and what problems may arise during this process?

#### 1. Selection of gas usage

Processing gas: Select high-purity NH<sub>3</sub>

PS: Why not use a mixture of low concentration inert gas and NH<sub>3</sub>?

At 20 °C, the liquefaction pressure of NH<sub>3</sub> is about 0.86Mpa, and the pressure of the mixed gas cylinder is generally around 10Mpa. At this time, NH<sub>3</sub> in the steel cylinder accumulates in a liquefied state at the bottom of the cylinder, and the actual output concentration of NH<sub>3</sub> is much lower than the percentage of the ratio; Due to the low output concentration, during a certain period of adsorption, it is possible that the adsorption saturation state may not be reached, resulting in significant deviation in the test results.

Carrier gas (reaction gas): high-purity Ar, high-purity N<sub>2</sub>, and high-purity He can be selected

#### 2、experimental condition

The experimental conditions mainly include the following points:

(1) Sample processing and loading amount: For chemical adsorption testing, in order to ensure the passing of the test gas, the sample needs to be compressed and sieved through a 40-60 mesh screen (if conditions are not met, ensure consistent particle size for each test); Comparative experiments need to ensure consistency in the sample size, which is generally 50mg-100mg. For samples with unsatisfactory



testing results or low content of the tested substance, the sample size can be appropriately increased.

(2) Dehydration temperature: Choose an appropriate processing temperature based on the type of sample to avoid changes in the composition or structure of the sample caused by high temperature; The most common processing temperature is 200-300 °C. For some samples with well-developed pores, a higher processing temperature (300-500 °C) may be chosen. For high-temperature processing, attention should be paid to avoiding the precipitation and decomposition of the load.

(3) The adsorption temperature and physical adsorption removal of NH<sub>3</sub>: The adsorption temperature is selected between 50-100 °C based on the sample situation. Currently, the most commonly used adsorption temperature condition is 100 °C, and the adsorption process adopts two methods: pulse quantitative adsorption or saturation adsorption.

PS: How to determine if adsorption is saturated?

① Pulse adsorption until there is no change in the peak area of the pulse to determine adsorption saturation. Some experimenters also determine the amount of chemically adsorbed or desorbed ammonia based on the number and area of adsorption peaks, but this method is not rigorous. Firstly, there will be significant physical adsorption during the adsorption process. Secondly, the peak emergence process is a concentration change curve under the influence of heating rate, which has poor consistency with the pulse peak area.

② The sample undergoes saturation adsorption under a certain flow rate of high-purity NH<sub>3</sub> gas flow, generally requiring at least 20 minutes to ensure sufficient adsorption of ammonia by the sample. NH<sub>3</sub> adsorption is still relatively easy at a certain temperature, but concentration and adsorption time need to be ensured.

(4) Program heating: The program heating conditions are quite critical, and different conditions will lead to different experimental results. For example, differences in peak temperatures and whether signal peaks at different acidic sites are "separated". So what are the conditions included?

① Heating rate: The commonly used heating rate is 10 °C/min. Many experiments have unclear peak



separation and even become a sample peak, and the rapid heating rate is one of the factors that cause this problem. In addition, a too fast heating rate can also cause a lag in the peak temperature.

② Carrier gas flow rate: The commonly used carrier gas flow rate is 30ml/min; The carrier gas flow rate, as a reaction condition, directly affects the peak temperature. Generally, when the flow rate decreases, the peak temperature will increase.

③ Temperature upper limit: Generally, the testing temperature for this experiment is within 700 °C, and excessively high temperatures are often meaningless.

### 3. Common experimental questions

(1) Calculation of acidity: The experimental process of "acidity" in NH<sub>3</sub>-TPD testing is quite complex, and there are mainly the following methods

① Standard pulse peak calibration method: Compare the peak area of the sample signal with the peak area of the standard pulse, calculate the volume of NH<sub>3</sub> in the peak area of the sample, and then calculate the "acidity". But the peak process is a concentration change curve under the influence of heating rate, which has poor consistency with the pulse peak area.

② Tail gas absorption titration method: This method is currently recognized as a relatively reliable method, which uses diluted acid solution for tail gas absorption, determines the amount of NH<sub>3</sub> desorbed through reverse titration, and then calculates the "acid amount". It is worth noting that after the adsorption is completed, the exhaust pipe should be promptly removed from the absorption tank to avoid acid solution backsuction after gas interruption. Backsuction will have a certain impact on the detector and other components of the instrument, and even damage them.

③ Solid acid calibration method: This method is theoretically feasible and has not yet been reported. The experimental process is the same as the calibration method of TPR, using standard substances to obtain the NH<sub>3</sub> amount corresponding to the peak area, and then comparing it with the test peak area to calculate the amount of NH<sub>3</sub> removed.

(2) What data processing problems will be encountered during the experiment?



① When multiple acidic sites exist simultaneously, the test peak is not completely separated. How to determine the acidity of a certain acidic site?

PS: You can use Origin software to simulate peak division, obtain the corresponding peak area, and then perform calculations.

② How to choose the conditions such as peak area and sample volume for standard sample calibration?

PS: Ensure that the sample size and testing conditions are consistent with the tested sample.

③ How is the amount of  $\text{NH}_3$  determined when calibrating the peak area for pulse peaks and solid acids?

PS: The amount of  $\text{NH}_3$  in the pulse peak is obtained through the volume of the quantitative ring, which ranges from 50 microliters to 200 microliters. Generally, each instrument is equipped with a fixed volume quantitative ring before leaving the factory; The peak area consumption  $\text{NH}_3$  of the standard sample calibration is calculated based on the theoretical consumption  $\text{NH}_3$  of the standard sample.

(3) What is the "tailing" of a peak?

The main reason for this is the rise in gas temperature, which causes the TCD signal to experience temperature drift. The influence of gas temperature on TCD signals is generally not significantly different, and the decomposition and extraction of samples often exhibit sustained and strong effects.

(4) What is the "negative peak" in the test results?

"Negative peak" refers to the peak shape that is opposite to the result of the tested substance in the experiment; This phenomenon is relatively rare in TPD experiments, and the opposite peak shape is mainly caused by the following situations, which need to be determined based on actual experimental conditions.

① The extraction of sample  $\text{H}_2\text{O}$ , such as the incomplete removal of  $\text{H}_2\text{O}$  adsorbed by the sample

②  $\text{NH}_3$  undergoes decomposition under high-temperature catalytic action

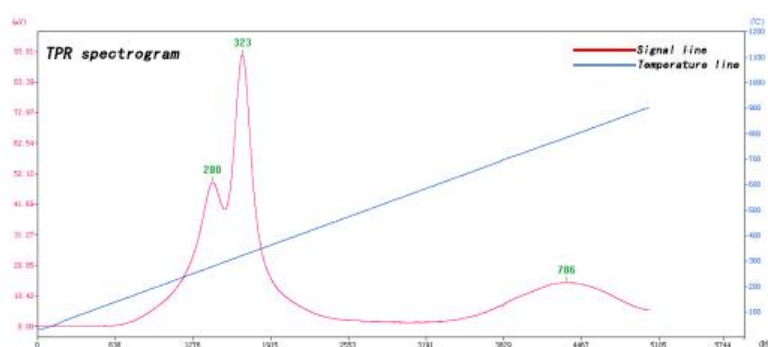
③ Decomposition of sample load or carrier under high temperature



## 2、 Analysis of TPR (Programmed Temperature Reduction) Experiment

TPR (temperature rammed)。

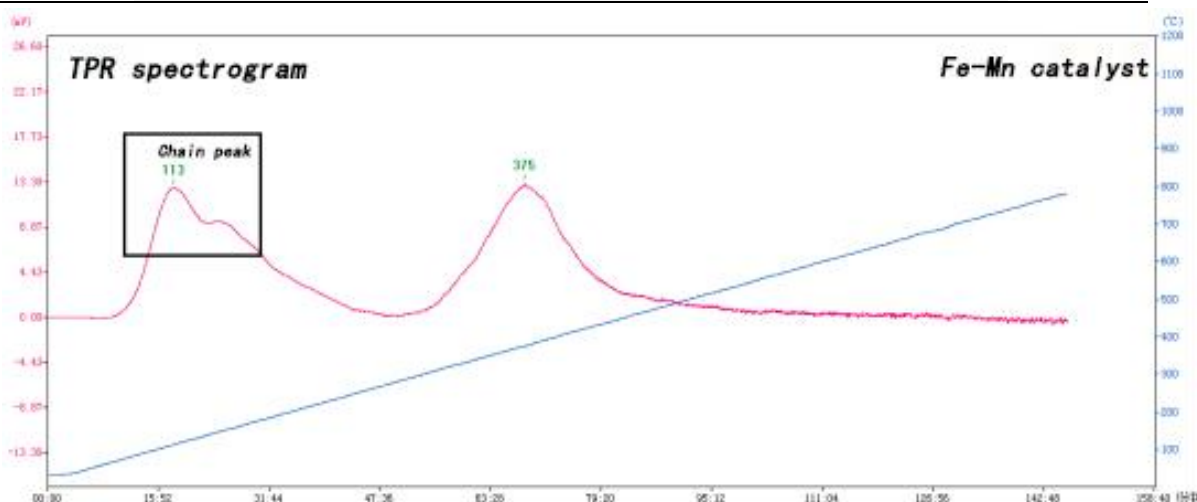
This experiment uses reduced gas (Ar/H<sub>2</sub> or N<sub>2</sub>/H<sub>2</sub>) under certain programmed heating conditions, passes through the catalyst bed, and measures the hydrogen concentration signals at various temperature points using a thermal conductivity (or mass spectrometry) detector to obtain the reduction performance of the catalyst.



The reduction temperature of metal oxides and metal salts depends on their chemical properties. If other substances are doped into a single substance and there are interactions between the substances that change the properties of the original substance, the reduction temperature will also change accordingly. Based on the degree to which the reduction temperature of the mixture deviates from the reduction temperature of the pure compound (this deviation mainly refers to the changes in the peak temperature and peak temperature range), we can infer the interactions between the components.

In TPR experiments, a "standard sample" can be used to calibrate the peak area. After calibration, we can obtain the amount of "hydrogen" consumed based on the peak area of the sample test, and then calculate the amount of reduced substance; In addition to obtaining the amount of reducing substance, we can also obtain the valence state of the reduced substance. When different valence states coexist in the same substance, they often appear as two inseparable "continuous peaks" in the spectrum.





How should TPR experiments be conducted and what problems may arise during this process?

### 1. Selection of gas usage

Processing gas: Select high-purity Ar and high-purity He

Carrier gas (reaction gas): Select high-purity Ar/H<sub>2</sub> (H<sub>2</sub> is 5% -10%) mixture gas

When conditions permit, try not to choose N<sub>2</sub> or N<sub>2</sub>/H<sub>2</sub> mixture as much as possible, as some samples may react or adsorb with N<sub>2</sub>, such as Ag<sub>3</sub>N

### 2. experimental condition

The experimental conditions mainly include the following points:

- (1) Sample processing and loading amount: For chemical adsorption testing, in order to ensure the passing of the test gas, the sample needs to be compressed and sieved through a 40-60 mesh screen (if conditions are not met, ensure consistent particle size for each test); Comparative experiments need to ensure consistency in the sample size, which is generally 50mg-100mg. For samples with unsatisfactory testing results or low content of the tested substance, the sample size can be appropriately increased.
- (2) Dehydration temperature: Choose an appropriate processing temperature based on the type of sample to avoid changes in the composition or structure of the sample caused by high temperature; The most common processing temperature is 200-300 °C. For some samples with well-developed pores, a higher processing temperature (300-500 °C) may be chosen. For high-temperature processing, attention should



be paid to avoiding the precipitation and decomposition of the load.

(3) Program heating: The program heating conditions are quite critical, and different conditions will lead to different experimental results. For example, the difference in peak temperature and whether the reduction peaks of the two substances are "separated". So what are the conditions included?

① Heating rate: The commonly used heating rate is 10 °C/min. Many experiments have unclear peak separation and even become a sample peak, and the rapid heating rate is one of the factors that cause this problem.

② Carrier gas flow rate: The commonly used carrier gas flow rate is 30ml/min; The carrier gas flow rate, as a reaction condition, directly affects the peak temperature. Generally, when the flow rate decreases, the peak temperature will increase.

### 3. Common experimental questions

(1) How to determine the hydrogen consumption in the experiment?

① Peak area calibration using standard pulse peaks

② Peak area calibration using standard copper oxide

What problems will be encountered during the use of the above methods?

When multiple substances or a single substance has multiple valence states, the test peaks are not completely separated. To determine the hydrogen consumption of each component, it is necessary to simulate the peak separation using Origin software, obtain the corresponding peak area, and then perform calculations.

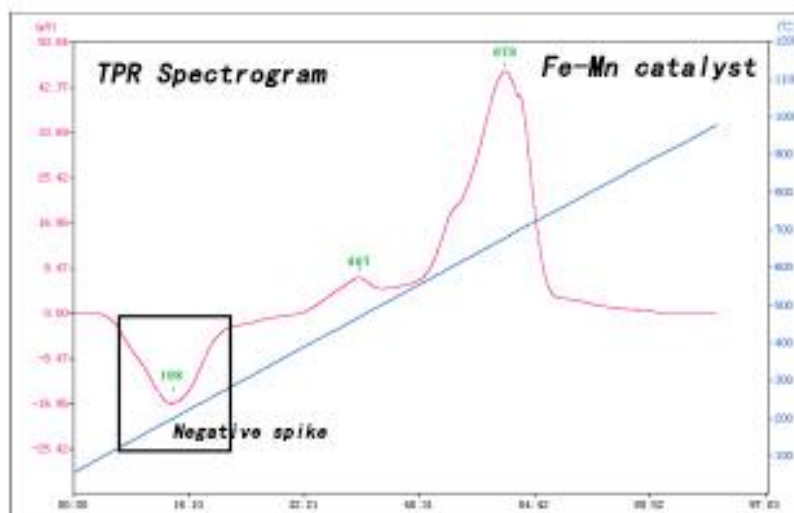
How to choose the conditions such as peak area and sample volume for standard sample calibration? It is necessary to ensure that the sample size and testing conditions are consistent with the tested sample.

How is the amount of H<sub>2</sub> determined when calibrating the peak area of a pulse peak? The amount of H<sub>2</sub> in the pulse peak is obtained through the volume of the quantitative ring, which ranges from 50 microliters to 200 microliters. Generally, each instrument is equipped with a fixed volume quantitative ring before leaving the factory; The peak area hydrogen consumption calibrated by the standard sample is obtained by



calculating the theoretical hydrogen consumption of the standard sample.

(2)What is the "negative peak" in the test results?



"Negative peak" refers to the peak shape that is opposite to the result of the tested substance in the experiment; The appearance of opposite peaks in TPR experiments is mainly caused by the following situations, which need to be determined based on actual experimental conditions.

- ① H<sub>2</sub>O generated by reaction
- ② HCl generated by reaction
- ③ CH<sub>4</sub> generated by reaction
- ④ Material undergoes dehydrogenation phenomenon (hydrogen overflow)
- ⑤ H<sub>2</sub>O adsorbed by the material itself

How to judge and solve the above situation?

- ① Negative peak generated by H<sub>2</sub>O

If there is a negative peak in the low-temperature range, it is likely to be an adsorbed water peak; The water peak can be divided into two situations: adsorption of H<sub>2</sub>O and reaction of H<sub>2</sub>O. We can replace the carrier gas with Ar and conduct experiments on samples treated under the same conditions. If the negative peak still exists, it proves that it is adsorption of H<sub>2</sub>O. If it disappears, it indicates reaction of H<sub>2</sub>O; The negative peak appearing in the reaction of H<sub>2</sub>O often occurs when the content of the loaded substance is



too low (usually manifested as weakening the peak signal); The H<sub>2</sub>O peak signal can be driven out by setting a cold trap before TCD.

② Negative peak generated by hydrogen overflow

After excluding the influence of H<sub>2</sub>O peak through cold trap, some negative peaks may also appear, which may be hydrogen overflow; Hydrogen overflow is a phenomenon in which hydrogen chemical bonds in a catalyst decompose, resulting in dehydrogenation and an increase in hydrogen concentration in the mixed gas. This phenomenon occurs at the application temperature of the catalyst and is considered beneficial for catalytic reactions. The concentration of H<sub>2</sub> in TPR decreases, and the test results show a positive peak; On the contrary, it will exhibit a negative peak. Hydrogen overflow occurs in numerous catalytic reactions. In addition, hydrogen reacts with precious metals such as Pt to form hydrides, which also decompose at high temperatures. These negative peaks often appear at higher temperatures; There is also a dehydrogenation phenomenon, which is common when the specific surface area of the sample is large. At low temperatures, the sample adsorbs hydrogen gas, and at high temperatures, it desorbs, leading to negative peaks. This situation is relatively rare because the desorption temperature of hydrogen gas is lower.

③ The negative peak generated by the compound generated by the reaction

Carbon deposits exist during sample preparation or in used catalysts, and methane is generated during the TPR process. On the one hand, the existence of carbon deposits is demonstrated, and on the other hand, the temperature at which methane occurs is also demonstrated; There are also some metal salts that have the effect of producing products during TPR experiments, such as HCl produced after CuCl reduction; The above products can all be removed by filling the cold trap tube with adsorbent to eliminate the negative peak and its impact on the experimental results.

(3) After the experiment is completed, there is material sintering on the reactor tube wall.

This phenomenon is caused by the precipitation of metal elements in the load due to the high reduction temperature, which are sintered on the pipe wall; Therefore, when conducting TPR experiments, attention



should be paid to the experimental temperature, as excessively high temperatures may not necessarily be meaningful.

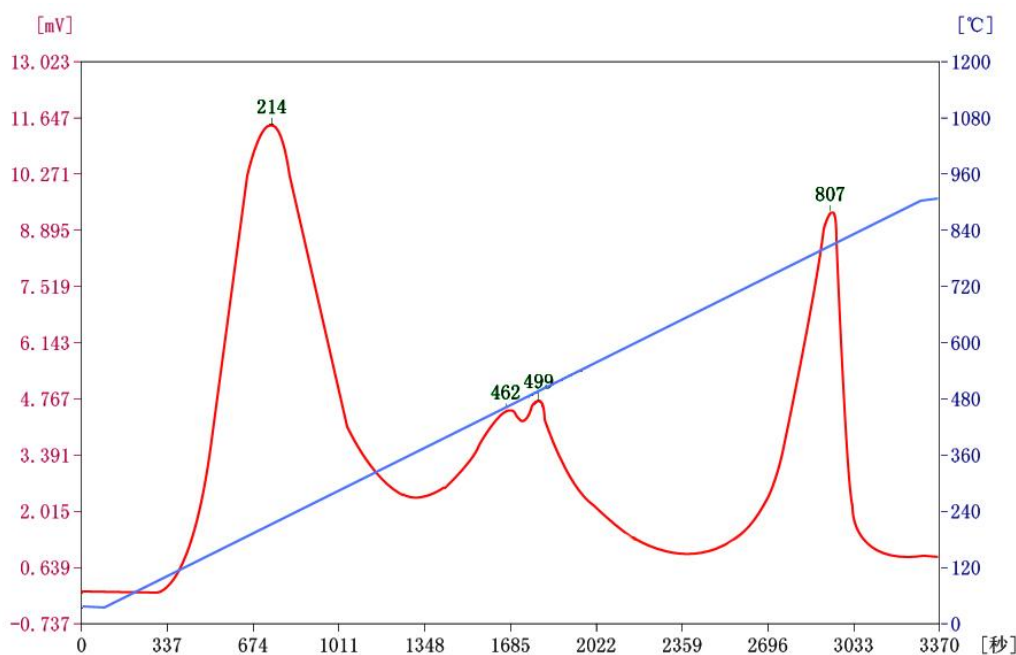
(4) TCD signal cannot return to "zero point".

The problem is mainly caused by two factors. One is that high temperature increases the gas temperature, causing a difference in gas temperature between the TCD testing port and the reference port, resulting in a signal value of "temperature drift"; Another reason is that there is continuous gas consumption or reactant generation in the test sample.

### **3、Experimental Analysis of O<sub>2</sub> TPD (Temperature Programmed Desorption of Oxygen)**

O<sub>2</sub>-TPD (oxygen temperature programming desorption) .

The O<sub>2</sub>-TPD experiment is currently the main method used to study the oxygen storage performance of catalysts (nucleation oxygen and adsorption oxygen). This experiment uses a thermal conductivity detector under certain programmed heating conditions to measure the oxygen concentration signals desorbed by the catalyst at various temperature points, and obtain the oxygen storage capacity and active sites of the catalyst. In addition, the oxygen storage capacity in ternary catalysis is measured using the method of oxygen pulse titration.



O<sub>2</sub>-TPD experiment

How to operate the O<sub>2</sub>-TPD experiment and what problems may exist during this process?

### 1、 Selection of gas usage

Processing gas: Select high-purity oxygen gas O<sub>2</sub>

Carrier gas: Select high-purity helium He

### 2、 experimental condition

The experimental conditions mainly include the following points:

- (1) Sample processing and loading amount: For chemical adsorption testing, in order to ensure the passing of the test gas, the sample needs to be compressed and sieved through a 40-60 mesh screen (if conditions are not met, ensure consistent particle size for each test); Comparative experiments need to ensure consistency in the sample size, which is generally 50mg-100mg. For samples with unsatisfactory testing results or low content of the tested substance, the sample size can be appropriately increased.
- (2) Dehydration temperature: In oxygen desorption testing, nucleation oxygen is the focus of the test, so the dehydration temperature should not be too high to avoid the precipitation of nucleation oxygen caused by excessive temperature. Therefore, it is recommended to set the dehydration temperature within 200 °C.

If there is crystal water or other conditions in the sample that cannot be completely removed at 200 °C, a



low-temperature cold well can be used for condensation removal in the experiment to avoid water signals affecting the test signal results.

(3) Oxygen adsorption: In order to test the oxygen adsorption capacity (amount of desorbed oxygen) of the sample during testing, it is necessary to perform oxygen adsorption on the catalyst. Therefore, the adsorption temperature is crucial. Oxygen has a certain oxidation capacity, and too high a temperature can change the valence state of the catalyst load. At the same time, too high a temperature can also cause the precipitation of crystal nucleus oxygen. It is recommended to set the oxygen adsorption temperature below 200 °C, with the most common conditions being 100 °C and 200 °C.

(4) Program heating: The program heating conditions are quite critical, and different conditions will lead to different experimental results. The general testing conditions for the PCA-1200 chemical adsorption instrument are: carrier gas flow rate 30ml/min, heating rate 10 °C/min. The decrease in heating rate will cause the peak temperature to decrease, and the decrease in carrier gas flow rate will cause the peak temperature to increase.

### 3. Common experimental questions

(1) How to determine the source of oxygen, whether it is nucleation oxygen or adsorption oxygen?

#### ① Isotope tracing can be used to label catalysts

It is a microanalysis method that uses radioactive isotopes as tracers to label research objects. Tracer atoms, also known as labeled atoms, are atoms with easily detectable nuclear physical characteristics.

#### ② X-ray photoelectron spectroscopy, also known as XPS

It is a spectroscopic method that uses high-energy photons with wavelengths in the X-ray range to irradiate the measured sample and measure the resulting photoelectron energy distribution.

#### ③ Judging by desorption temperature

The desorption of crystal nucleus oxygen generally requires a certain amount of energy, so the temperature of the desorption peak is often above 300 °C, while the adsorbed oxygen is often within 200 °C.





## 4、Experimental Analysis of Metal Dispersion

Metal dispersion refers to the ratio of the number of metal atoms distributed on the surface of the carrier to the total number of atoms on the carrier. For metal dispersion testing, the active components tested mainly include Pt, Pd, Rh, Ni, Cu, etc. The most commonly used testing methods for metal dispersion are chemical adsorption, namely pulse titration and static volumetric method.

### 1、The testing method and principle of metal dispersion

#### (1) The principle of chemical adsorption method for testing metal dispersion

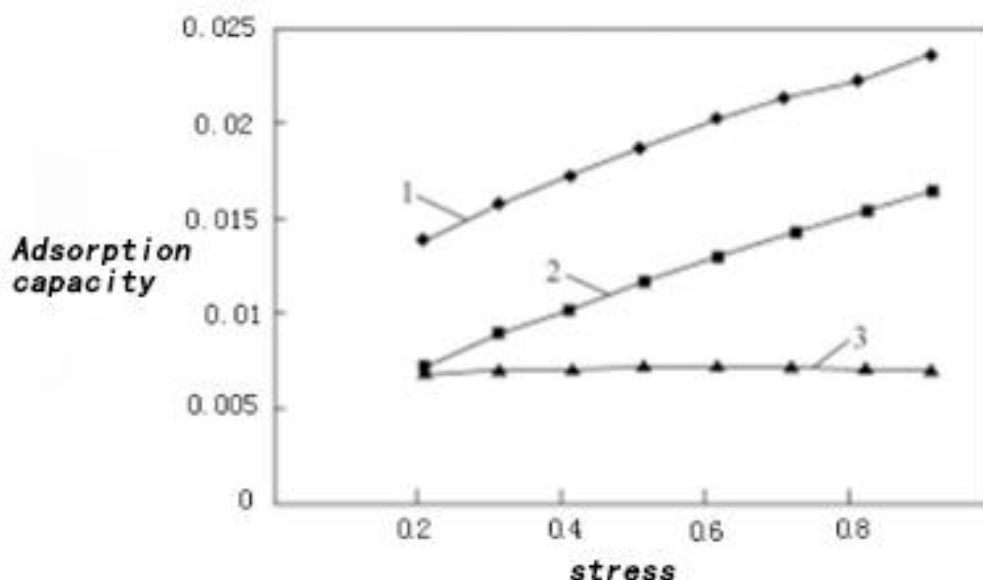
Chemical adsorption method is the use of certain probe molecules (such as  $H_2$ ,  $O_2$ ,  $CO$ , etc.) to adsorb on metal surfaces in a chemically constructed form. By measuring the chemical adsorption capacity of the probe molecules, the dispersion and active surface area of the metal can be calculated. During the testing process, the pulse titration method requires that the probe molecules do not have any adsorption with the carrier, while the static volumetric method only requires that the probe molecules do not have any chemical adsorption with the carrier. Similarly, the chemical adsorption form of the probe molecules with the tested metal conforms to a certain chemical stoichiometry coefficient.

#### (2) The main method for testing the dispersion of metals using chemical adsorption method

##### ① Static capacity method

The static capacity method uses a physical adsorption instrument (specific surface area and pore size analyzer) to conduct two consecutive isothermal adsorption tests of  $H_2$  on the tested sample, obtaining two isothermal adsorption lines of  $H_2$ . Due to the irreversibility of chemical adsorption, the fitting curve of chemical adsorption capacity can be obtained by comparing the difference in adsorption capacity between two curves at different partial pressures, and then the  $H_2$  chemical adsorption capacity can be obtained. Data such as metal dispersion and active surface area can be calculated.

As shown in the figure:



#### Isothermal adsorption test of H<sub>2</sub> (static capacity method)

There are two isotherm adsorption curves for H<sub>2</sub> in the figure:

The first isothermal adsorption curve represents the reversible chemical adsorption, irreversible chemical adsorption, and physical adsorption isotherms of H<sub>2</sub> on the metal surface.

The second isothermal adsorption curve represents the irreversible chemical and physical adsorption isotherms of H<sub>2</sub> on the metal surface.

The third curve is a fitting curve for chemical adsorption capacity, and its intercept from the y-axis is the chemical adsorption capacity of the probe gas on the surface metal.

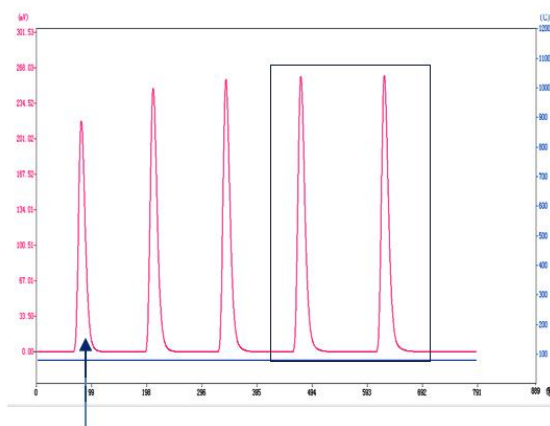
So how was curve 3 obtained? Why is this curve the chemical adsorption capacity of the probe gas? Due to the irreversibility of chemical adsorption, after the completion of curve 1 testing, the reversible chemical adsorption and physical adsorption of H<sub>2</sub> can be removed through vacuum treatment, retaining the irreversible chemical adsorption of H<sub>2</sub> on the metal surface. Under the same experimental conditions, the second isothermal adsorption curve was measured. Due to the occupation of irreversible chemical adsorption sites, only reversible chemical adsorption and physical adsorption can occur this time, resulting in a difference in H<sub>2</sub> adsorption capacity. The difference between the two adsorption isotherms is called



irreversible chemical adsorption.

## ② Pulse titration method (dynamic chemical adsorption method)

Pulse titration method is an experiment that uses a dynamic chemical adsorption instrument to perform O<sub>2</sub> titration and H<sub>2</sub> titration on a catalyst that has been reduced by H<sub>2</sub>. Finally, data such as the dispersion and active surface area of the metal are obtained by calculating the consumption of H<sub>2</sub>.



Residual peak of pulsed gas

When the peak area does not change (no consumption), it can be considered as the standard peak

### Pulse titration method (dynamic chemical adsorption method)

It should be noted that during the experiment, the signal displayed in the spectrum is the residual quantity, not the consumption quantity. When the sample no longer consumes pulse gas, the peak area will not change, and we consider the unchanged peak area as the standard peak (corresponding to a certain volume of pulse gas). We can obtain the consumption volume of H<sub>2</sub> by the difference in peak area between the remaining peak and the standard peak.

## ③ Calculation formula

- a) Metal dispersion: Metal dispersion refers to the ratio of the number of active metal atoms on the catalyst surface to the total number of metal atoms in the catalyst.

Application formula:

$$D = \frac{n \times V_g \times M_M}{W \times P \times 22414}$$

Explanation: In the actual reaction process, the stoichiometric coefficient of the gas is analyzed. For the stoichiometric formula of hydrogen oxygen titration, different literature provides different results. For



example, when the hydrogen titration method is used to determine the chemical adsorption of oxygen on Pt, one oxygen atom on Pt consumes two hydrogen atoms to react and produce water, which is adsorbed on the carrier; Meanwhile, due to the fact that Pt atoms themselves need to adsorb a hydrogen atom after deoxidation, the amount of hydrogen used to titrate the oxygen atoms adsorbed on Pt is only 2/3 of the total hydrogen consumption. That is, if the number of surface Pt atoms is 2/3 of the number of H<sub>2</sub> molecules consumed, the stoichiometric coefficient is taken as 2/3. In fact, the choice of chemical stoichiometry and coefficients must be determined by supplementary experiments.

**V<sub>g</sub>**: Represents the volume of analytical gas consumed by the catalyst sample that has been completely oxidized (or reduced) during the experimental process, in ml;

**n** : Stoichiometric coefficient

**M<sub>M</sub>**: Relative atomic mass of catalyst metal, unit: g/mol

**W** : Mass of the tested sample, in grams

**P** : Mass fraction of active metals in catalyst, %

**b)** Active specific surface area: The surface area of surface active metal atoms in a unit mass catalyst.

Application formula: 
$$S = \frac{2 \times V_g \times N_0 \times \sigma_M}{W \times P \times 22414} \times 10^{-18}$$

**S** : Catalyst activity specific surface area, in units **m<sup>2</sup>/g**

**V<sub>g</sub>** : The volume of analytical gas (H<sub>2</sub> or O<sub>2</sub>) consumed by the catalyst sample that has been completely oxidized (or reduced) during the experimental process, expressed in ml;

**N<sub>0</sub>** : NA, 6.02 × 10<sup>23</sup> /mol

**σ<sub>M</sub>** : Cross section area of catalyst metal atoms, 0.089 nm<sup>2</sup>

**W** : Mass of the tested sample, in g

**P** : Mass fraction of active metals in catalyst, %

2、Experimental conditions and issues of dynamic chemical adsorption (using PCA-1200 as an example)



(1) Selection of gas usage

- a) Pulse gas: high-purity O<sub>2</sub>, high-purity H<sub>2</sub>; Choose high-purity CO when testing metal Ni
- b) Carrier gas: Select high-purity N<sub>2</sub> and high-purity Ar; Select high-purity He when testing metal Ni

Process gas and reaction gas should be selected with a purity of 99.999% or higher as much as possible.

(2) experimental condition

The experimental conditions mainly include the following points:

① Sample processing and sample volume

For metal dispersion testing, to ensure consistency in testing, the sample must ensure consistent particle size during each test; The general sample size is 50mg-100mg. For samples with unsatisfactory testing results or low content of the tested substance, the sample size can be increased appropriately, but it should not be too much to avoid hydrogen overflow, which may lead to larger testing errors and poor repeatability.

② Dehydration temperature

For the vast majority of catalysts, the dehydration temperature depends on the material's tolerance temperature and the form of water present in the tested sample. When the sample can withstand higher temperatures, it is recommended to have a dehydration temperature of 300-400 °C;

③ Reduction temperature

It is necessary to choose an appropriate reduction temperature based on the situation of the sample to ensure that the tested component is in a elemental state before the pulse titration experiment. Many Pt/Rh/Pd catalysts often require a higher reduction temperature, typically between 300-500 °C.

④ Oxidation temperature and titration temperature

For the vast majority of samples, 100 °C is relatively reasonable; Of course, if the activity of the catalyst is strong enough, there is no problem at room temperature. For supported catalysts such as Ni/Cuo, this temperature may need to be appropriately increased (the metal dispersion test of Ni based catalysts cannot



oxidize).

### (3) Common experimental questions

#### ① What is the significance of using cold traps in pulse experiments

The function of a cold trap is to cool down and remove impurities; It needs to be filled with adsorbent and placed in the coolant. So what important role can be played in pulse titration experiments? Most metal dispersion tests use hydrogen oxygen titration, where hydrogen produces  $H_2O$  during the reduction of oxides. The response signal value of  $H_2O$  in TCD is opposite to the corresponding signal value of  $H_2$ , which means that  $H_2O$  will cancel out the intensity of the remaining  $H_2$  signal peak; When the sample does not consume  $H_2$ , the standard peak is not affected by  $H_2O$ , resulting in an overestimation of the calculated amount of  $H_2$  consumption. The final calculated metal dispersion will also be overestimated, and even the calculated activity specific surface area value will be large and off spectrum.

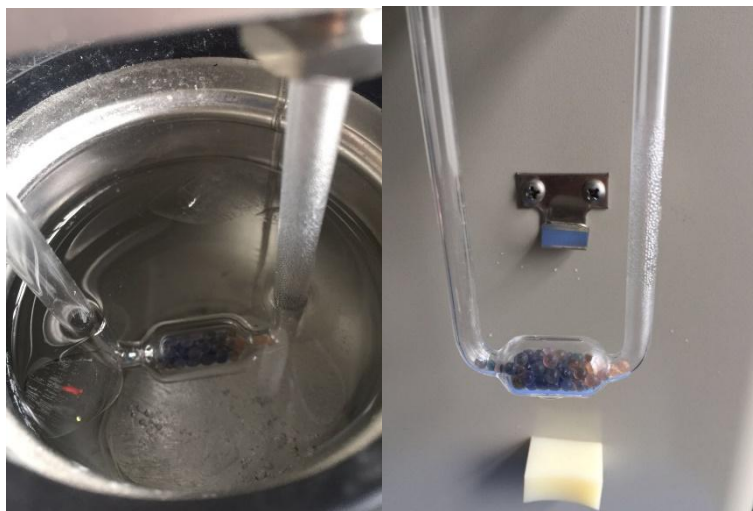


Image: Water in a cold trap

#### ② Catalysts such as palladium and carbon supported on carbon are prone to "burning", what should be done?

Catalysts supported by carbon materials such as palladium and carbon will experience combustion during the process of oxygen absorption. How should we avoid it during testing? Firstly, it is necessary to control the oxygen absorption temperature and titration temperature reasonably. The temperature should not be too high. If the reaction activity is allowed, room temperature can be chosen. Secondly, if the



reaction is not easy at room temperature, low concentration oxygen can be used or pulse injection can be used to control the oxygen content, but the temperature should also be controlled within 100 °C as much as possible.

③ How to conduct the metal dispersion test for metallic Ni

The testing of metallic Ni often does not require oxygen absorption, which can lead to the failure of the testing experiment. The testing process of Ni should not refer to the testing process of Pt/Pd/Rh and other metals, and the H-O reaction method should not be used. The biggest purpose of this method is to increase the consumption of H<sub>2</sub>. If the hydrogen consumption is too small and the quantitative ring volume is too large, TCD may not be able to detect it, so many low load precious metal samples use hydrogen oxygen titration. However, for the testing of Ni, it is difficult to reduce NiO after oxygen absorption by H<sub>2</sub> or CO, so the method of directly titrating Ni in H<sub>2</sub> or CO should be chosen. It is worth noting that the testing of Ni requires a certain temperature, and it is recommended to operate at 100-200 °C. Some samples may even require 300 °C. In actual testing, CO is easier to obtain effective results than H<sub>2</sub>.

④ How to conduct the metal dispersion test for metallic Cu

The metal dispersion test of Cu is similar to the testing process of Pt/Pd/Rh and other metals, using H<sub>2</sub> titration. However, due to the multivalent state of metal Cu, it is difficult to determine the stoichiometric coefficient. Therefore, the oxygen absorption process of Cu cannot use oxygen with higher oxidation performance, and requires the use of N<sub>2</sub>O with relatively weaker oxidation ability. This gas can only oxidize Cu to monovalent at lower temperatures, thereby solving the problem of stoichiometric coefficient. It is recommended to choose the oxidation temperature and titration temperature between 100-200 °C in the experiment.

## Chapter 8 Precautions

### 1、 Preparation before the Experiment

1. Correct use of gas, refer to the following table





Target Experiment	Carrier Gas	Treatment/Reaction Gas	Pulse Gas
NH <sub>3</sub> -TPD	N <sub>2</sub> /Ar	Low concentration NH <sub>3</sub> (1% -5%)	/
CO <sub>2</sub> -TPD	He/Ar	CO <sub>2</sub>	/
O <sub>2</sub> -TPD	He/Ar	O <sub>2</sub> or low concentration O <sub>2</sub>	/
N <sub>2</sub> -TPD	He/Ar	N <sub>2</sub>	/
O <sub>2</sub> -TPO	He/O <sub>2</sub> mixture (5% -10% O <sub>2</sub> )	He/N <sub>2</sub>	/
H <sub>2</sub> -TPR	Ar/H <sub>2</sub> mixture (5% -10% H <sub>2</sub> )	Ar/N <sub>2</sub>	/
	N <sub>2</sub> /H <sub>2</sub> mixture (5% -10% H <sub>2</sub> )	Ar/N <sub>2</sub>	/
CO-TPR	Low concentration CO monoxide	He/Ar	/
Metal dispersion	Ar/N <sub>2</sub>	Ar/N <sub>2</sub> O <sub>2</sub> (optional steps)	H <sub>2</sub> or low concentration H <sub>2</sub>
Oxygen storage capacity	He	H <sub>2</sub>	O <sub>2</sub>
Hydrogen storage capacity	Ar/N <sub>2</sub>	O <sub>2</sub>	H <sub>2</sub>

First, check if the gas circuit connection is correct. After the gas is turned on, set the output pressure to 0.2-0.5MPa and ensure that the software and instrument are successfully connected.

## 2. Proper use of reactors

The experimental temperature is less than 600 °C using a glass reactor.

When the experimental temperature is above 600 °C, a quartz reactor is used, and the nozzle of the quartz reactor is marked as "1200 °C".

When installing the reactor, first try installing (comparing the position), keep the reactor perpendicular to the ground, and then tighten the nuts; The inlet and outlet nuts should be tightened simultaneously to avoid reactor damage.

## 3. Proper loading of bed and catalyst

Quartz cotton should be used as the bed layer, and the top of the bed layer should be laid flat and level with the bottom of the manifold.

Install an appropriate amount of catalyst according to the operation manual, and the catalyst should be laid



flat on the bed, with a general bed height of a few millimeters; If the catalyst is relatively fluffy, the filling height should be less than 1cm; The catalyst cannot be too fine, generally not less than 80 mesh.

#### 4. Proper use of four-way and six way valves

Except for the pulse experiment, the six way valve switch needs to be directed towards the "↗" injection pressure.

In all experiments, the four-way valve needs to be directed towards“ ↑ ”。

#### 5. Proper use of thermocouples

The temperature detector (thermocouple) should be inserted into the bottom of the manifold.

When raising the electric furnace, the reactor manifold should be tightly attached to the furnace wall, and the furnace mouth should be filled with quartz cotton.

#### 6. Correct use of cold hydrazine

When NH<sub>3</sub>-TPD and CO<sub>2</sub>-TPD are used, adsorbents such as silica gel cannot be added to the cold hydrazine tube.

When H<sub>2</sub>-TPR, use a glass cold hydrazine tube, fill the tube with dried discolored silicone gel, and place ice water or a mixture of liquid nitrogen/isopropanol in a cold hydrazine cup.

#### 7. Proper installation of exhaust pipes

Exhaust pipes 1-4 should be installed independently and discharged outdoors; Avoid connecting exhaust 1/2 and exhaust 3/4, and do not use the same exhaust pipe.

When using hazardous gases such as CO, NH<sub>3</sub>, H<sub>2</sub>, etc., exhaust pipes must be installed and discharged outdoors.

When using O<sub>2</sub> and H<sub>2</sub> simultaneously, the same exhaust pipe cannot be used.

## 2、 During the Experiment

1. During the degassing and adsorption stages, TCD does not need to be activated.
2. When heating up the program, do not touch or shake the high-temperature furnace.



3. To protect the thermocouple and extend its service life, wait for the furnace temperature to drop below 200 °C before lowering the electric furnace.
4. Start TCD constant temperature in advance, usually at 80 °C or 100 °C; When running the benchmark, it is necessary to first turn on the reference and test air circuit of TCD, set the flow rate to be consistent, and then turn on TCD (i.e. the TCD indicator light is on).
5. Manual operation experiment, after TCD is turned on, let it run automatically for 10-15 minutes before manually zeroing; Adjust to zero every 2-3 minutes until the requirements are met.
6. During H<sub>2</sub>-TPR, the coolant (mixture of ice water or liquid nitrogen/isopropanol, etc.) is loaded into a cold hydrazine cup while starting TCD.
7. During the experiment, if black/brown/yellow/oily condensate appears on the wall of the reactor tube, it may be due to sample decomposition or incomplete treatment. At this time, the generated signal line may have interfered with the target signal line, and excessive ejecta can contaminate the instrument, causing unnecessary property damage. This situation should be avoided as much as possible before testing. Especially, the sample should not contain untreated organic matter.
8. When using automatic operation mode, after preparing according to the requirements in the operation manual, set the operating parameters correctly.

### **3、 End of Experiment**

1. After each use of gas, especially corrosive gases such as ammonia and hydrogen sulfide, the main valve and pressure divider valve of the gas cylinder should be closed, and the gas between the gas cylinder and the instrument inlet should be completely discharged, and the gas path should be cleaned with inert gas.
2. After each experiment, all gas between the connected instruments and the gas cylinder should be drained to avoid damaging the valve without the instrument for a long time.
3. After the experiment, the TCD should be turned off first (i.e. the TCD indicator light should be turned off), and then the gas cylinder should be turned off.



4. After the manual testing is completed, pay attention to saving the data.

## **Chapter 9 Common Problems and Solutions**

### **1、 Security Alarm**

1. Combustible gas alarm

The high-frequency and rapid alarm sound of "Didi Didi".

2. High temperature safety alarm

A low-frequency alarm sound with a certain time interval.

### **2、 Signal Line Faults and Trouble Shooting Methods**

#### **1. The signal line is always drifting in a certain direction and cannot be stable**

(1) Determine if the flow rate of the No. 2 mass flow meter is normal. If it is not normal, check the operation status of valve 7. After normal operation, if the flow rate is still abnormal, check if there is any blockage in the air circuit. If the No. 2 flow meter is working normally, it is necessary to check the working status of valve 8. (First, ensure that the operation of the No.1 flow meter is normal.)

(2) Due to air leakage in a certain pathway, this situation often occurs in the testing gas path. Specific exclusion methods refer to

Summary: There are two main reasons for the occurrence of this problem: first, there is no gas in one of the two channels of TCD, and second, there is gas leakage in one channel or the presence of ejecta in the sample.

#### **2. Irregular jitter (burrs) in signal lines**

(1) The intake pressure is unstable, replace the pressure reducing valve and pressure stabilizing valve to eliminate it.

(2) The air outlet is blocked and blocked. Check whether there are foreign matters at the



outlet of the exhaust pipe and in the exhaust pipe. Raindrops in rainstorm weather may cause poor exhaust, which may also occur.

(3) TCD pollution requires the use of gas to clean the TCD gas circuit. After blowing, open the TCD heating device, first raise the temperature to 200 °C and blow for 20 minutes, then lower it to 100 °C and blow for 2 hours. If the TCD signal still cannot be stable during the constant temperature process, it is recommended to replace the TCD. Note: To clean TCD, it is necessary to ensure that there is always gas passing through the reference and test gas paths, and the flow rate can be set to 80-90ml/min.

### 3. The peak shape is not smooth and has serrations

(1) Determine the signal strength (peak voltage value). If the signal strength is relatively weak (1-2mv), this phenomenon is normal. If the signal value is still high, it is likely due to sample decomposition or the influence of extracted substances. Try to drive it away through a cold well as much as possible.

(2) Empty the sample tube and run the baseline. If the baseline is stable, it can be determined that it is a sample problem. If it is abnormal, check for air leakage and clean the TCD with gas.

### 3、 Non Peak Fault and Troubleshooting Methods

1. Check the working status of the four-way valve and confirm that the position of the four-way valve is "upward" and not in the "empty" state.



Emptying condition

Working condition



2. Check the coordinates of the software's horizontal axis. If the coordinates are reduced too much, make changes. The first scale on the "0" coordinate is 5-10mv.

3. Check if the test gas is reasonable, such as using a mixture of argon and hydrogen gas for TPR testing, which cannot be achieved using pure hydrogen gas; The carrier gas for O2-tpd should be argon or helium, and nitrogen cannot peak (the thermal conductivity coefficients of nitrogen and oxygen are too close, and the TCD detector cannot separate the signal).

4. Check the working status of TCD and whether the air circuit is normal



Confirm that TCD is in the on state (the indicator switch light is red); Check if the test gas circuit is correct, manually click on each valve to confirm if the valve is working properly. The common problem is that valve 8 does not switch to the TCD test outlet; Check if the output flow rate of the quality flow meter is normal, and make adjustments to confirm that the flow meter is working properly and there is no "dead state".

### Correct Operation Method for TCD Temperature Control:

- Enable TCD
- Long press the "run" button until the run indicator light turns green
- Press the up/down keys to set the constant temperature, usually set to 80 °C

### Be careful:

- Before turning off TCD, first turn off the TCD temperature control (i.e. long press the "run" button until the run indicator light goes out), and then turn off TCD
- Except for commonly used temperature control buttons, do not press other control buttons randomly; If the garbled code is pressed, you can wait for a few minutes until the garbled code on the temperature control meter disappears before continuing the operation.



5. Check if the use of the cold trap is correct. NH<sub>3</sub>-TPD cannot add adsorbents to the cold trap, and the desorbed NH<sub>3</sub> will be adsorbed by the adsorbent, and the signal cannot be detected; In TPR testing, reaction products such as H<sub>2</sub>O, CH<sub>4</sub>, and HCl often cancel out signal peaks or even exhibit negative peaks, which requires the addition of adsorbents in the cold trap tube to drive them away.

#### **4、 The reasons for not dividing peaks and improvement methods**

1. The heating rate is too fast, resulting in a "hump" and unclear separation of the two peaks. It is necessary to reduce the heating rate, but not too much.

2. The heating rate is too slow, causing the peak to be not sharp enough, too flat and wide.

It is necessary to increase the heating rate appropriately.

3. The peak temperatures of the two signal peaks are too close and the consumption or removal amount is large, resulting in peak superposition. It is necessary to increase the carrier gas flow rate.

Note: If the above methods cannot be solved, more reliable data can only be obtained through external mass spectrometry detection.

#### **5、 Heating fault and troubleshooting methods**

##### **1. Software error "Thermocouple damaged or not placed in designated location"**

(1) Program heating electric furnace not placed in designated position or damaged

Due to the self-test program set up by the software, the electric furnace was not placed correctly. The software controls the continuous power supply to the electric furnace, and if the temperature does not rise or fall, the software will determine the fault.

(2) The heating program has not ended, remove the programmed heating electric furnace.





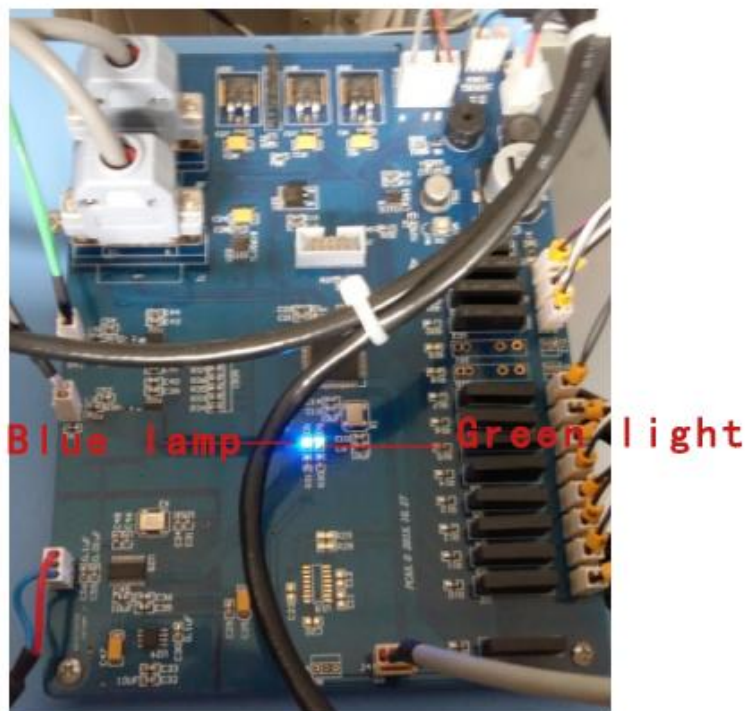
### (3) Thermocouple damage

If the thermocouple is damaged, the software will display a temperature of 1024 °C. If the display is normal, you can observe whether there is a temperature change in the thermocouple by measuring the body temperature when the temperature is displayed as room temperature.

### (4) Program heating electric furnace damaged

The electric furnace needs to use a multimeter to measure whether there is resistance (whether it is damaged). If it is damaged, replace the programmed heating electric furnace.

( 5 ) Motherboard malfunction: After setting the heating program, the motherboard indicator light does not flash.

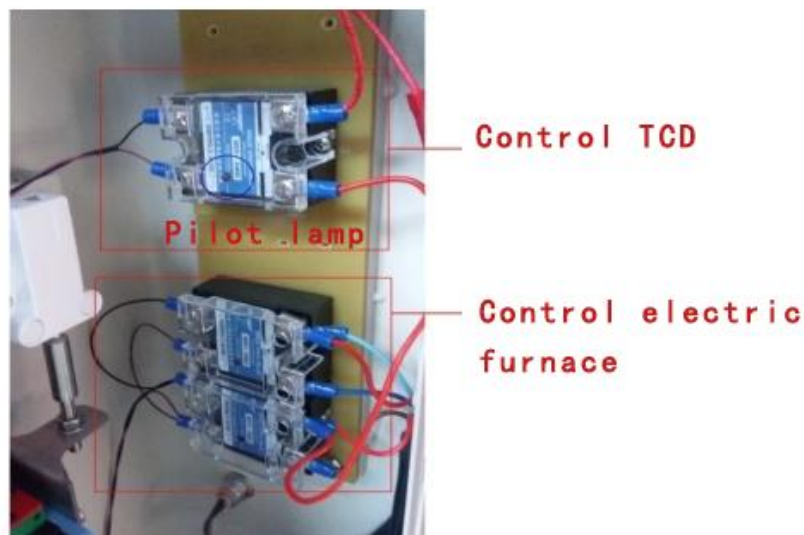


After power on, the green light remains on. When not in working mode, the blue light slowly flashes (in a "one exhale, one inhale" state); During normal operation, the blue light flashes frequently.

### (6) Relay malfunction



The motherboard indicator light is working normally, and the relay's working indicator light is not flashing.



## 2. Poor control of program heating rate

Instruments produced after June 2016 can be adjusted by adjusting the height of the programmed heating electric furnace (which can be appropriately lowered or raised by 0.3-0.5cm). If it cannot be improved, the electric furnace needs to be replaced

## 3. The heating curve jumps up and down in the high temperature range (blue line)

Thermocouples damaged or unable to operate at high temperatures (often occurring after 700 °C)

# 6、 TCD faults and troubleshooting methods

## 1. TCD constant temperature does not work

(1) Run the temperature control meter and check if the relay is working properly. If the indicator light does not flash, replace the relay. If the relay is working properly, it indicates that the heating device is damaged.

(2) The temperature control meter shows HHHH, and the temperature measurement PT



resistance is damaged

## **2. TCD working indicator light does not light up**

- (1) TCD switch damaged
- (2) Damaged power supply
- (3) Polarity switch not placed in the correct position

## **7、 Electromagnetic valve faults and troubleshooting methods**

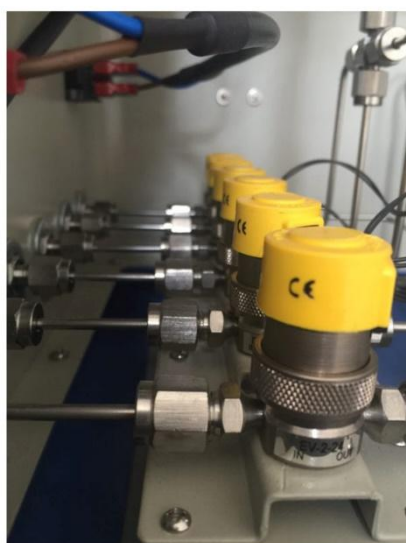
### **1. damage**

When the software is online normally, click the control button, and the solenoid valve does not have any "action" (no sound). You need to open the solenoid valve to confirm whether it is damaged or contaminated. For a two-way solenoid valve, there is no obvious damage or contamination after opening, and it needs to be installed before control to eliminate the problem of "membrane" sticking after a long time of use. If it still cannot be controlled, you need to change the control position of the solenoid valve on the motherboard to eliminate the motherboard fault; In addition to the above-mentioned issues, the two position three-way solenoid valve also has the problem of "gas leakage" and the inability to switch the gas path caused by the change of valve core position during disassembly and assembly. The reason for not switching the gas path is because the valve core position is not aligned with the relative suction surface, and the valve core needs to be "knocked" back to the sealing surface; The main reason for "gas leakage" is that the position of the valve core exceeds the sealing surface and is uneven. It needs to be returned to its original position by tightening the top joint; The most corroded and damaged solenoid valve is valve No. 8, whose fault first manifests as abnormal TCD signal line. Therefore, TCD problems should be first investigated for valve No. 8 fault

### **2. contaminated**



The solenoid valve is a detachable and cleanable "diaphragm" type solenoid valve. When disassembling and cleaning, attention should be paid to the installation position and direction of the diaphragm and gasket; After cleaning, please confirm that the air circuit is unobstructed. If necessary, it needs to be cleaned repeatedly to confirm the cleanliness of the air circuit.



## Chapter 10 Instrument Accessories

### 1、 Attachment Table

**Tip: The goods shall prevail**

Sort	Encode	Packing details	Specifications/models	Quantity
Main cargo	/	Chemical adsorption instrument host	PCA-1200	1
	031-0009-002	Program to heat up the electric furnace	TP-1200	1
	028-0010-001	Stove power cord	PW-1200	1
	/	PC software	/	1
Workstation	selectable	PC (win10 及以上, 500G)	/	1
Photothermal system	selectable	Photothermal furnace	/	1
	selectable	Xenon lamp	/	1
Supplies	901-0004-004	Quartz reactor	U-1200H	10
	905-0001-001	Metal funnels	L-240HJ	3
	901-0003-003	U-shaped cold trap tube glass	U-120	5
Spare parts	029-0003-001	316L pneumatic tubing	2m/root	5
	029-0004-001	Plastic hoses	10m/root	2
	001-0001-005	Viton seals	2.2*1.9	50



	001-0001-003	Viton seals	5*2.5	20
	001-0001-007	Viton seals	5*1.9	20
	001-0001-001	Viton seals	9*3	20
	011-0002-001	nut	M8*1	10
	005-0014-001Z	thermocouple	K1	2
	005-0004-001	flash drive	16G	1
	005-0013-001	Quartz wool	10g/pack	1
	013-0001-002	Tee joints	3mm	2
	005-0016-006Z	Cold trap cups	500ml	1
	005-0012-001	Loading hook	general	1
	028-0004-004	Power cord	general	1
	028-0001-006	USB cable	general	1
	005-0008-001	Power strips	general	1
file	/	Operator's manual	/	1
		Operate video	/	1

## 2、 Attachment Introduction

(1) Sealing accessories for gas pipelines, as shown in Figure 10.2.1; Exhaust pipe as shown in Figure 10.2.2



Figure 10.2.1



Figure 10.2.2





(2) Sampling and sampling accessories

1、Reactors: mainly divided into two types: low-temperature reactors and high-temperature reactors. When the experimental temperature exceeds 600 °C, a high-temperature reactor must be used. The outer wall of the high-temperature reactor is marked with the words "1200 °C". As shown in Figure 10.2.3



Figure 10.2.3

2、Reactor and sample loading accessories, as shown in Figure 10.2.4; Reactor installation accessories, as shown in Figure 10.2.5

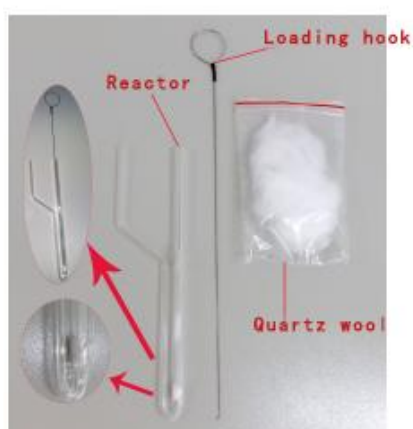


Figure 10.2.4



Figure 10.2.5

(3) Host and other accessories, as shown in Figure 10.2.6



Temperature programmed electric furnace

Cold trap

Figure 10.2.6

## Chapter 11 After sales Service Commitment

The service includes two parts: technical service commitment and product maintenance commitment.

The following is 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.

The content of technical services:

Biotech Electronics Technology Co., Ltd. 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 warranty service**

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.

### **(2) 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.

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.

### **(3) 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.

#### 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.

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