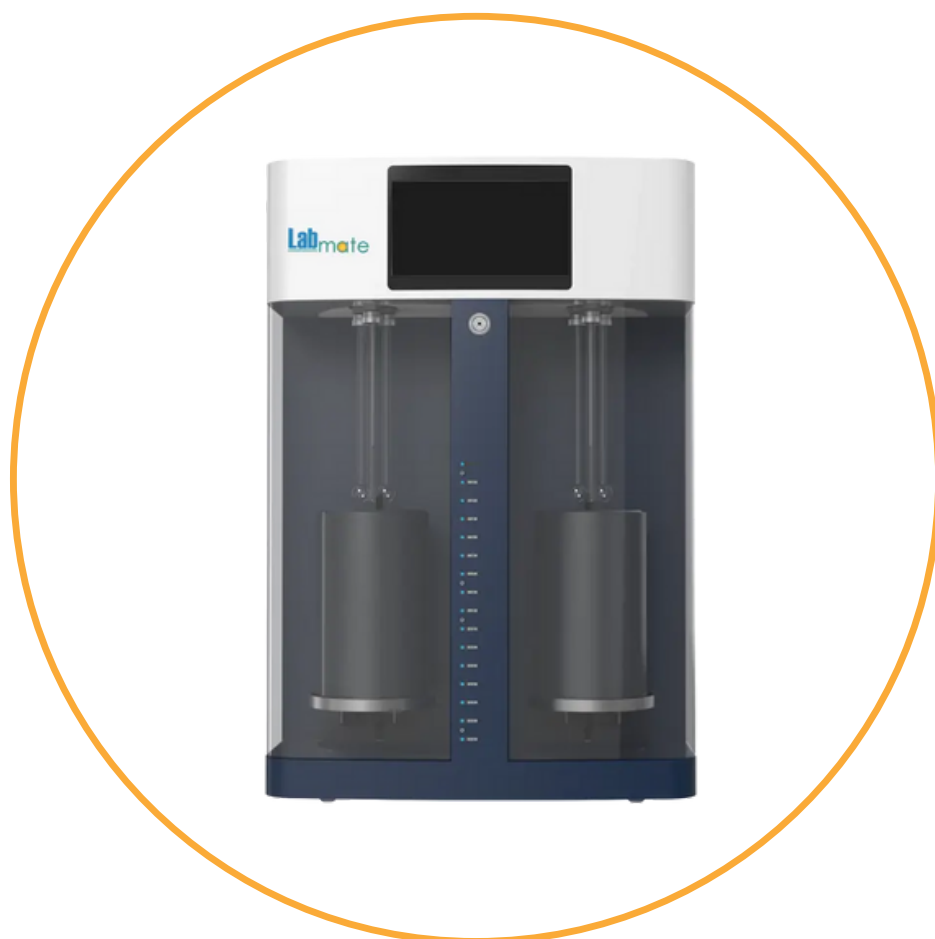




Automatic Surface Area Analyzer LMSAA-101

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1. Safety Measures

- 1) Make sure the power line is long enough to connect the analyzer to power. If not, use another longer power line or connect with a qualified power strip.
- 2) Check power, voltage, power line, and power strip carefully; make sure they are in good condition.
- 3) Must shut down power if needed to move the analyzer.
- 4) After the operation finishes, shut off the analyzer first, then the power line.
- 5) Must shut down power before maintenance.
- 6) The UPS system is better equipped if you have.
- 7) Better not to operate instruments in thunder weather.
- 8) An earth wire is a must.
- 9) Better to place a rubber blanket under the analyzer if the fund/environment permits.
- 10) Check the power line regularly to see if aging has occurred; if yes, use another new power line timely manner.
- 11) Better to keep a dry laboratory floor and test bench, no water on them.
- 12) Does not open analyzer cover without authorization, especially for non-professionals.

Notices

- 1) The analyzer should be placed in an environment that keeps a constant temperature, has no dust, and has no electromagnetic interference.
- 2) Should install empty sample tubes in analyzer ports if you plan not to use it for a long time. To avoid the dust from going into the analyzer manifolds.
- 3) Gas cylinders should be placed in a cool and dry place, should also avoid direct sunlight. Gas cylinders should not be beyond the analyzer too far, but better within 2 meters.
- 4) Better supply the stabilized voltage for the analyzer and keep the wire cable and data cable separated.
- 5) Better warm up the analyzer for 30 minutes before testing, because pressure transducers need a few minutes to get heat balance.

Attention

- 1) In the procedures of pretreatment and analysis, must ensure the software selected port must match the analyzer port where the installed sample tube is; otherwise, atmospheric air will be inhaled into and cause heavy damage.
- 2) Must fill in the filter foam in the mouth of sample tubes; otherwise, samples may be inhaled into the manifold.
- 3) Make sure the sample tubes' bottoms touched the heating mantle bottom during the pretreatment procedure.
- 4) Normally the oil is clear and transparent. If the oil darkens, it should be changed. The oil should be changed after the first 100 operating hours and then at least every 2,000 to 3,000 operating hours or after one year.
- 5) At high intake pressure and intake temperature and/or when pumping contaminated gases, the oil will have to be changed more frequently.

2. Introduction

Automatic Surface Area Analyzer LMSAA-101 is an analytical instrument designed to measure the specific surface area of materials. It has independent sample testing and processing systems to minimize contamination. Ball screw integrated lifting system, controlled by stepping motor, overcomes the shortcomings of ordinary screw type.

3. Features

- Stainless steel micro welding vacuum pipeline system minimizes dead volume
- High-precision silicon film pressure sensor to measure pressure accurately
- High integration and anti-interference pneumatic valve control system
- Transparent plastic safety door
- Degassing treatment enhances accuracy
- Durable 4L stainless steel Inner dewar bottle
- Liquid level control system maintains constant liquid nitrogen level
- Multi-Mode data analysis supports various analysis approaches

4. Specifications

Model No.	LMSAA-101
Pore size measurement range	2 nm to 500 nm
Measurement range	0.0005 m ² /g and above
Specific Surface Area Repeatability Error	≤ ± 1.0%.
Number of samples tested	4
Test modes	Single Nitrogen Test Mode and Nitrogen, Helium Standard Test Mode
Test Gas	High purity N ₂ gas
Vacuum Pump	Built in two stage vacuum pumps
Vacuum system	Stainless steel micro welding vacuum pipeline system
Vacuum leakage rate	1× 10 ⁻¹⁰ (Pa × m ³ /s)
Pressure Range	0 to 3 Bar (0 to 2250 Torr)
Pressure Accuracy	0.0015
Control system	Pneumatic valve control system
Communication	RS485 or RS232 communication
Software	Windows compatible
Test Functions	Static volumetric principle, adsorption/desorption isotherms measurement, BET method, Langmuir method, T-plot external surface area measurement, BJH total pore volume and pore size distribution analysis, DR and DA filling theory, true density measurement; powerful online data analysis system and specific surface area and pore size distribution test system
Dimensions (L × W × H)	746 × 600 × 900 mm
Net Weight	80 kg

5. Applications

Automatic Surface Area Analyzers are used for precise and efficient measurement of surface area in materials, finding applications in research, quality control, and manufacturing processes.

6. Instrument Introduction

Instrument Overview

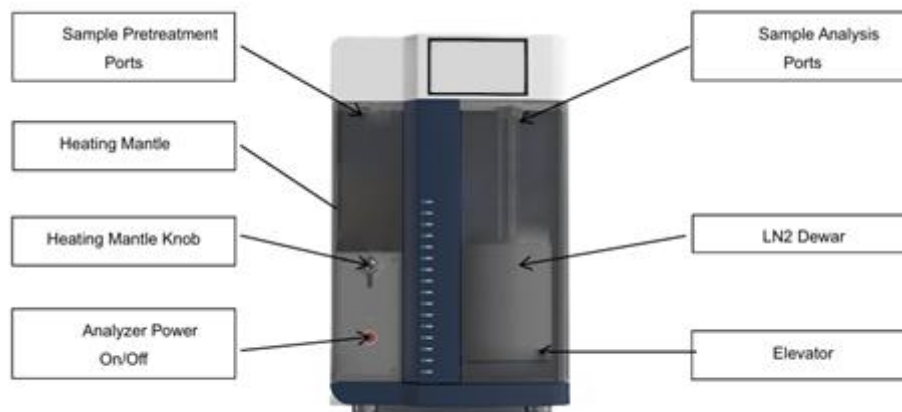


Figure-1

7. Software Installation

7.1 Introduction to Software Installation

The software has been installed in the built-in computer before the instrument is shipped. If you need to reinstall the software, follow the steps below:

- 1) Plug the USB into the USB port, double click  and it will pop up below the picture.

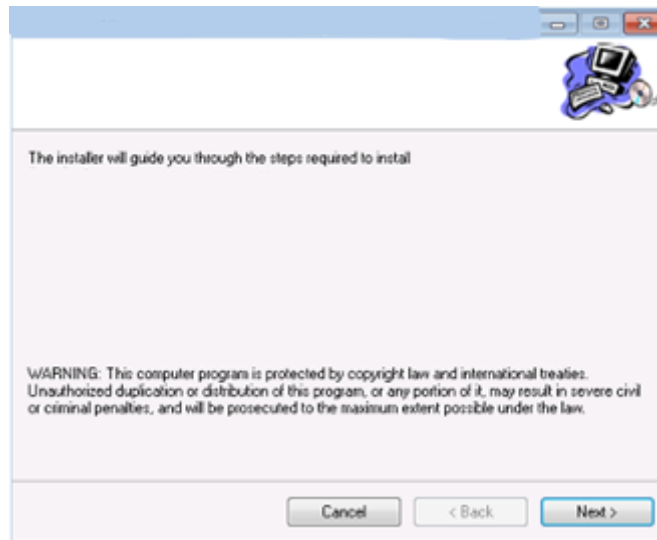


Figure-2

- 2) Click the "Next" button, select the installation folder, (Do not install to the C disk). If only one C disk, need to create a new folder, BUT do not install in the "Program Files" folder.

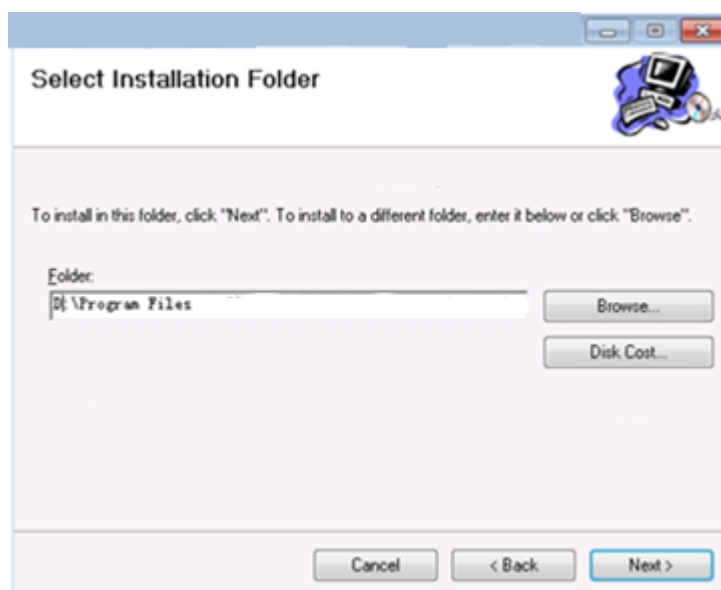


Figure-3

3) Then click the “Next” button to continue.

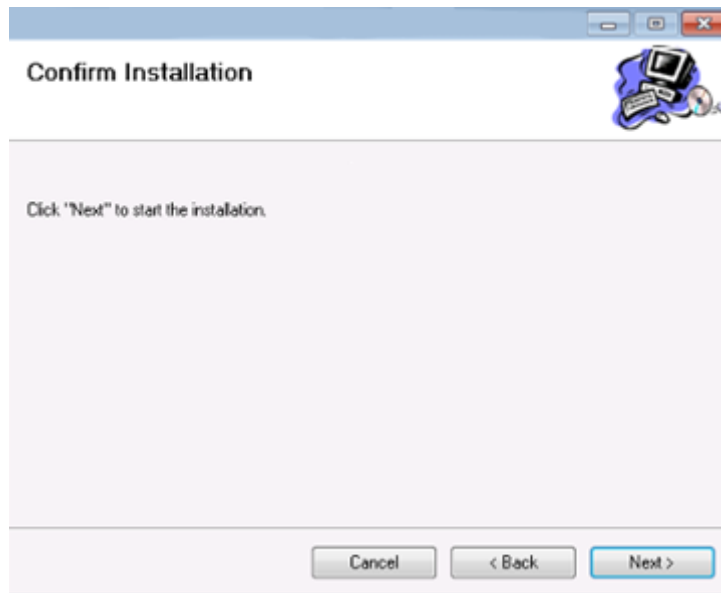


Figure-4

4) Click the “Next” button to continue and wait for the installation to complete.

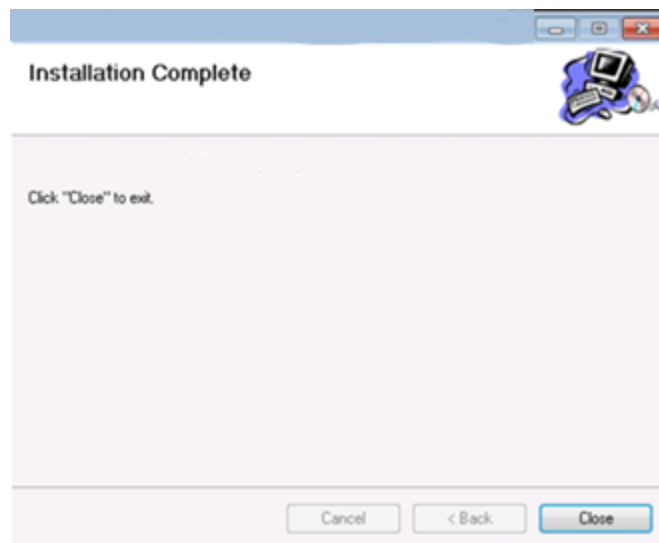


Figure-5

5) Click the “Close” button to complete the installation.

Attention: Copy the file from the USB and paste it into the \APP Instruments Software folder, where the software is installed. Every analyzer has one exclusive file. The analyzer cannot work if this file does not match.

7.2 Built-in Computer

- 1) The instrument has a built-in computer. The software has been installed. Switch on the instrument, the embedded computer will be turned up at the same time, then enter the boot screen:



Figure-6

- 2) Select the admin account and enter the password gylz-2020 to access the computer system, in which the software has been installed.

7.3 Accessory Installation

7.3.1 Gas Regulator Installation

- 1) **Cylinder valve:** Switch on/off the gas cylinder.
- 2) **Gas regulator:** Display gas pressure in the cylinder.
- 3) **Analyzer output:** Display output pressure for the analyzer.
- 4) **Adjustor:** Adjust the input pressure for the analyzer; “DEC” for decrease and “INC” for increase.
- 5) **Port to analyzer:** Connect the adjustor with the analyzer inlet port.

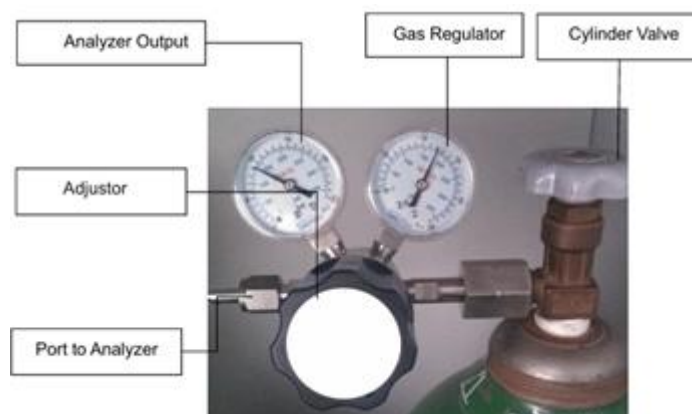


Figure-7

Notices:

- 1) Regulators are precise parts and should be handled slowly and carefully. Make sure the adjustor is in closed condition before switching on the cylinder valve.
- 2) Do not overtighten fittings; otherwise, you will crack the tubing and cause gas leakage. Wait regulator to reach a level off when it meets display lag.

Attention:

- 1) Appropriate two-stage gas regulators which have been leak-checked and specially cleaned, are required.
- 2) The pressure at the air outlet is indicated by a sub-pressure gauge, and the output pressure must keep at 0.15 MPa (0.5 MPa for the nitrogen cylinder when the pneumatic valve is used). Subject to actual conditions, adjust by engineers or under the guidance of engineers.
- 3) N₂ gas is a must for the analyser's running; even using other adsorbates such as Ar by the third gas inlet port, the N₂ is used as a driving source for the analyser's pneumatic valves.



Figure-8

7.3.2 Gas Installation

Copper pipe is used for connecting the instrument and the gas regulator. Accessories include a nut, a copper gasket, and an O-ring. Install in this order: O-ring large incline surface, small incline surface, copper gasket inclined surface, copper gasket plane, nut. Screw up the nut by hands firstly, then tighten by spanners.

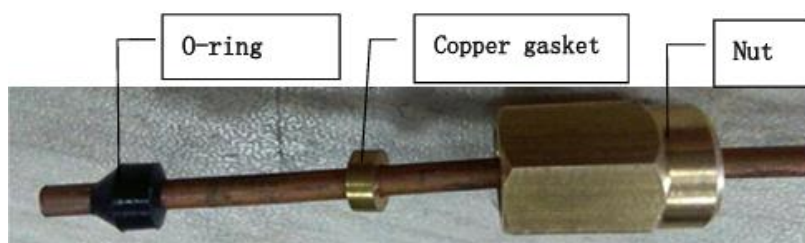


Figure-9

7.3.3 Vacuum Pump Installation

- 1) Find the vacuum pump from the accessory box and fill it with pump oil.
- 2) Open the analyzer's back cover, place the pump in the bottom layer where there should be a foam cushion for reducing the pump's vibration.
- 3) Connect the pump with the metal hose using by clamp.
- 4) Power on the pump by the bottom chassis power socket and always keep the pump being the "ON" position.

7.3.4 Sample Tubes Installation

- 1) Use the supplied funnel to fill samples into tubes and don't pour samples directly, in order to prevent samples from adhering to the tube's neck wall.
- 2) The volume of filled samples should not be beyond 3/4 of the bottom volume of the tube. Use a needle item to thrust samples if they are choked in the funnel.

- 3) The mass of the filled sample heavily depends on its surface area value. The higher it is, the less its mass. Generally, the ideal mass for the sample with $9.1 \text{ m}^2/\text{g}$ is about 1g. Check this rule to estimate your sample's mass.
- 4) Should use a balance with a ten-thousandth precision to cut down error



Figure-10

- 5) Do not let the sample adhered to the tube neck wall as it may not be soaked in liquid nitrogen bath.
- 6) Plug a glass rod into the sample tube after finished sample filling and weighing operations, to reduce void volume of the sample tube.
- 7) Put a sponge stuff at the end of glass rod plugged to prevent sample from running into the valve in case a sample suck-back occurs when evacuating.
- 8) Carefully screw the connecting nut together with the ferrule, to have them pressing the rubber O-ring tightly to achieve high airproof effect. Inset the funnel into sample tube and fill in the samples.
- 9) Following the same steps as installing sample tubes. Be sure the saturation pressure tube is installed properly on the P0 port.
- 10) Be aware that the sample tube is fragile and should be handled carefully.



Figure-11

8. Software Operations

8.1 Software Introduction

- 1) Click analyzer software logo from desktop.

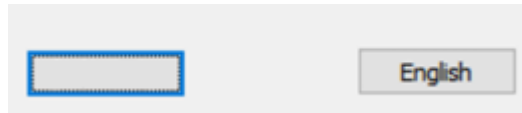


Figure-12

- 2) Select "English" language version, appear below screen.



Figure-13

- 3) Click on "new" in the upper left corner.

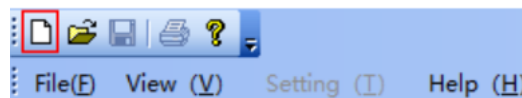


Figure-14

- 4) Select the view setting which appears in the picture below.

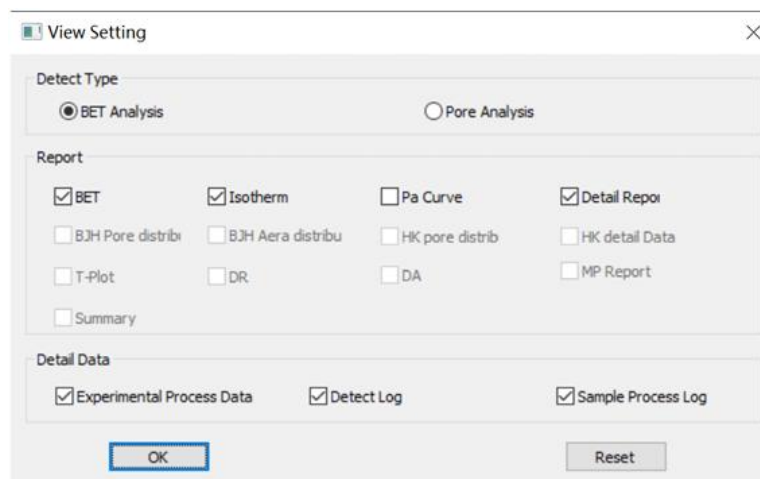


Figure-15

- 5) Software interface can be divided into below five zone. Zone I shows analysis data; zone II shows experimental control parameters; zone III shows experimental status; zone IV shows analysis log; zone V shows sample units. The Zone II is the key part which will be explained below.

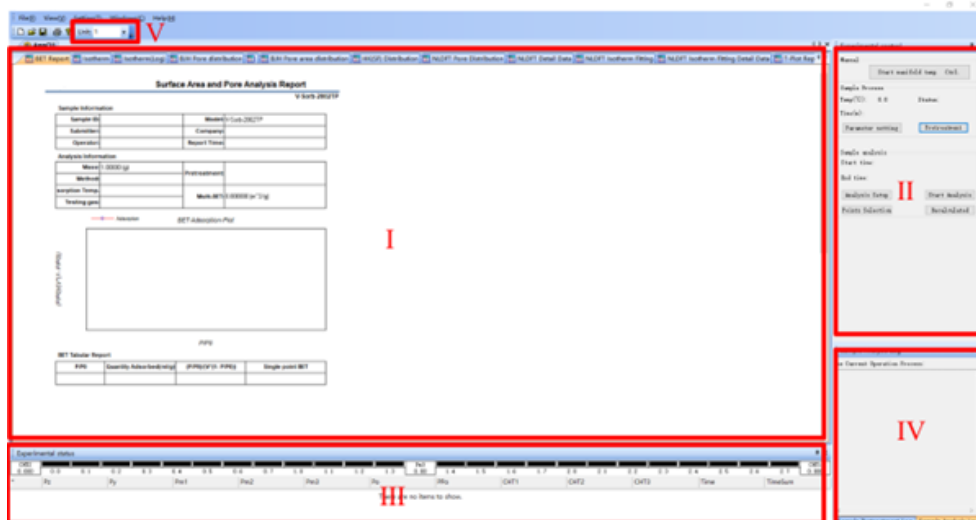


Figure-16

8.2 Sample Pretreatment

Pretreatment only available for single module instruments.

8.2.1 Introduction

Temperature: Shows the real time temperature during heating.

Status: Shows working status during pretreatment.

Time: Show samples pretreatment time.

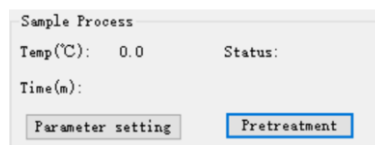


Figure-17

8.2.2 Parameters Setting

Click "Parameter setting", goes into parameter setting interface. It covers zone I: analysis selection; zone II: sample unit, zone III: pretreatment parameters.

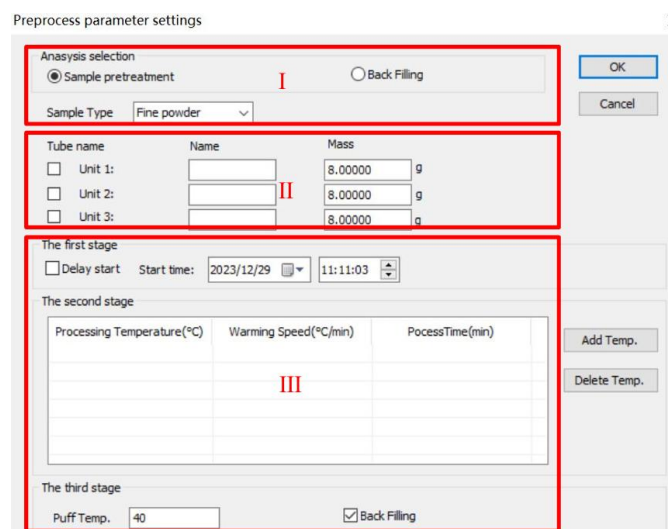


Figure-18

Zone I: including “sample degassing” and “gas backfill”

Sample degassing: Software will automatically finish vacuum evacuation, cooling, air charging, charging end steps if checked this function. When degassing finished, uninstall sample tube, weight its mass and install it to the analysis ports side.

If check the “sample degassing” but do not check zone III “Gas Backfill”: software will automatically finish the vacuum evacuation and cooling, then, stop degassing processes, it means samples are in the vacuum condition at this time. If want to uninstall sample tubes, need to check the “Gas Backfill” function in zone III, and samples will recover to atmospheric pressure condition.

The sample type can be selected as "fine powder" or "non-fine powder". For light samples, the fine powder type can be selected, thus effectively preventing pumping away during the vacuum and backfill inflation phases.

Zone II: After sample tube(s) installed, check the corresponding unit number(s).

Zone III: It can be divided into three stages.

The first stage: If a delayed start is selected, the start time of the preprocessing should be set according to the needs of the test.

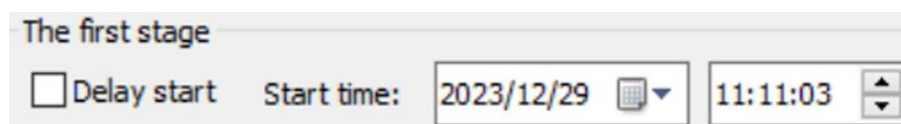


Figure-19

The second stage: Click “Add Temp.”, pops up “Processing Temperature Setting” dialog. Set the Processing Temperature, Warming Speed and Process time. the maximum processing temperature is 350°C.

Multi-stage temperature rise control can be set.

Click the “OK” button after all parameters were typed in. The temperature starts to rise, after reaching the set temperature and vacuuming is carried out until the treatment time is reached. then click “Start” to begin samples degassing/pretreatment.

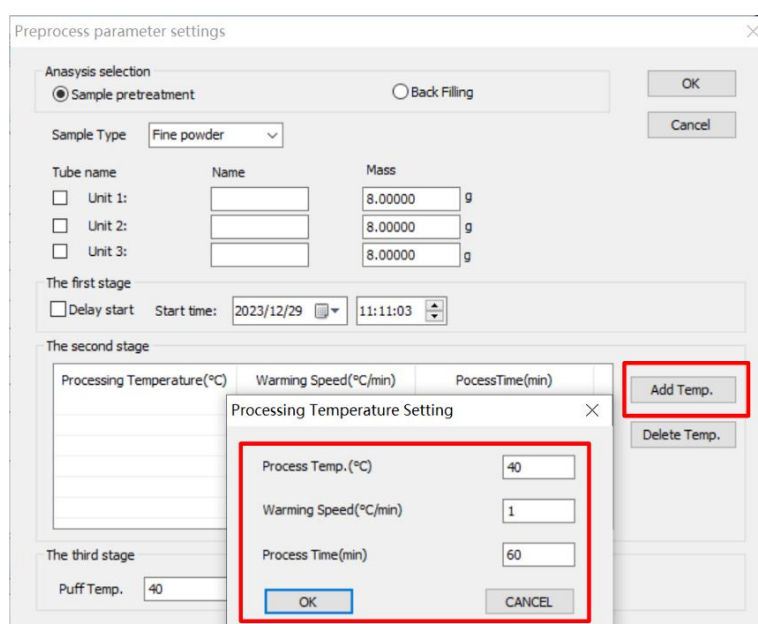


Figure-20

The third stage: It backfills the inflation. The sample tube returned to detachable mode for the next experiment. Software will automatically finish all set procedures, no manual operation needed after this.

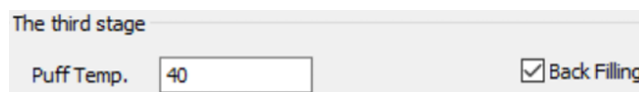


Figure-21

8.3 Sample Analysis

There will appear sample analysis begin and end time.

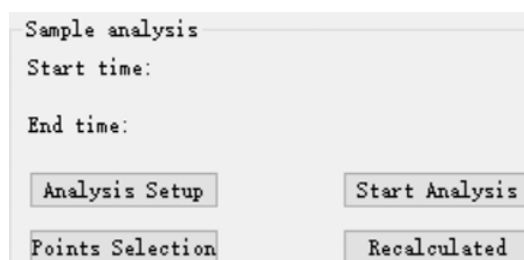


Figure-22

It will pop up analysis parameters setting window. If click “Analysis Setup” button, here you can type in analysis parameters. It can be divided into five zones. Zone I is “Analysis Function Options”, zone II is “Analysis Mode”, zone III is “Sample Parameters”, zone IV is “Air leakage rate”, zone V is “Analysis Parameters” and zone VI is “Analysis Information”.

NOTICE: Parameters vary for different models.

The 'Parameters Setting' window is divided into six zones:

- Zone I: Analysis selection**
 - ☒ BET Method
 - ☐ Warm Volume Calibration
 - ☐ Safe Descending
 - ☐ Pore Analysis
 - ☐ Cold Volume Calibration
 - ☐ Back Filling
 - ☐ Density Analysis
 - ☐ Saturation Po
- Zone II: Analysis Mode**
 - Gas type:
 - Molecular: 0.162 nm²
 - Density: 0.0015468
 - Sample Info: Sample Type:
- Zone III: Sample Parameters**

Unit No.	Warm Volume	Name	Mass	True Density	Cold Volume
☐ Unit 1	35.0000 ml		1.00000 g	2.0000 g/ml	10.0000 ml
☐ Unit 2	35.0000 ml		1.00000 g	2.0000 g/ml	10.0000 ml
☐ Unit 3	35.0000 ml		1.00000 g	2.0000 g/ml	10.0000 ml
- Zone IV: Air leakage rate(Pa·m³/s)**
 - Unit 1: Unit 2:
 - Unit 3: Cavity:
 - Manifold information: Manifold volume(ml) 21.825000
- Zone V: Analysis Parameters**
 - P/Po Point List:
 - Po Mode:
 - Gas Supply Mode:
 - Cryogen Level Control:
 - ☒ Run Adsorption
 - ☒ Auto Back fillings
 - ☒ BET Smart Point
 -
- Zone VI: Analysis Information**
 - Sample No.:
 - Name:
 - Submitter:
 - Degassing:
 - Operator:
 - Checker:
 - Company:
 - Adsorption:
 - Analysis:

Figure-23

1) Zone I: Analysis selection

BET method: To get BET data.

Pore analysis: To get adsorption isotherm and pore data.

Density Analysis: This function for ROUGHLY determining samples' true density values. Usually, operators need to do this function before running the single Nitrogen gas analysis mode.

Warm Volume calibration: This function for measuring the empty tube warm free volume at the room temperature. Operators need to do this measurement priorly when running "Density Analysis" or single Nitrogen gas analysis mode.

Cold Volume calibration: This function for measuring the empty tube's cold free volume of its fraction immersed in the cryogen liquid. Operators need to do this measurement priorly before running single Nitrogen gas analysis mode.

Saturation P0: This function for measuring the cryogen's saturation pressure. Normally be used when operators want to know how much your cryogen purity is.

Safe Descending: This function for assisting the cryogen flask elevator descend to bottom position safely. It is useful when the analyzer suffers from an unexpected power lose and the cryogen flask is still on the top position, or any other situations like this. In such emergency situations, operators must run this function before detaching sample tubes from analysis ports.

Back Filling: This function is used to backfill sample tubes to make the whole vacuum system return to normal atmosphere pressure. Operators must do it, in case analyser's analysis system is in vacuum state, before detaching sample tubes from analysis ports.

2) Zone II/III: Analysis Mode and Sample Parameters

Gas type: To choose the Adsorbate in analysing. Adsorbate parameter includes gas type, gas molecule cross section and density factor of selected adsorbate gas. Among the gas types, the adsorbate gases required for the test are selected, such as N₂, CO₂, Ar, Kr, H₂, etc.

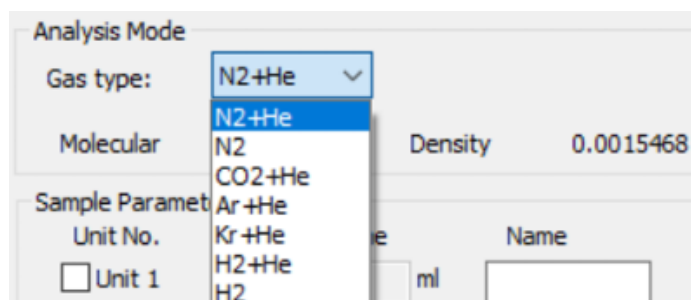


Figure-24

N₂+He, after selected the "N₂+He" analysis mode, many parameters such as the "Warm Volume", "Cold Volume" and "True Density" become gray, means these functions are inapplicable. When running this mode, the helium will be used to determine the warm free volume and cold free volume, and the nitrogen is used as adsorbate. This mode is recommended use for most analysis cases because it is easy and reliable.

The screenshot shows the 'Analysis Mode' section with 'Gas type' set to 'N2+He'. Below it, 'Molecular' is 0.162 nm² and 'Density' is 0.0015468. The 'Sample Info' section shows 'Sample Type' as 'Non Fine f'. The 'Sample Parameters' section has three units, each with 'Warm Volume' (35.0000 ml), 'Name', 'Mass' (1.00000 g), 'True Density' (2.0000 g/ml), and 'Cold Volume' (10.0000 ml) fields. The 'Warm Volume', 'True Density', and 'Cold Volume' fields are highlighted with red boxes.

Figure-25

N₂, after selected the “single N₂” mode, the “Warm Volume”, “True Density” and “Cold Volume” etc. all parameters are applicable, operators need to input them before or after running the “BET” or “Pore Analysis” function. Operators can determine the warm volume by the [Warm Volume Calibration](#) function and the cold volume by [Cold Volume Calibration](#) function by using an empty (or after finished experiment) sample tube priory. Operators need to determine samples’ true density by [Density Analysis](#) function, then, calculate samples’ true volume using their true density and mass. Operators must keep use the same empty tube to fill in samples and run analysis. Please be noted that the software will automatically subtract the occupied volume by sample (sample volume) from the warm volume or cold volume. This single nitrogen analysis mode is used when sample adsorbs the helium gas in case immersed in cryogen liquid, such as microporous samples: active carbon, molecular sieve etc., in this situation, the helium is not suitable to determine the warm free volume and cold free volume.

The screenshot shows the 'Analysis Mode' section with 'Gas type' set to 'N2'. Below it, 'Molecular' is 0.162 nm² and 'Density' is 0.0015468. The 'Sample Info' section shows 'Sample Type' as 'Non Fine f'. The 'Sample Parameters' section has three units, each with 'Warm Volume' (35.0000 ml), 'Name', 'Mass' (1.00000 g), 'True Density' (2.0000 g/ml), and 'Cold Volume' (10.0000 ml) fields. The 'Warm Volume', 'True Density', and 'Cold Volume' fields are highlighted with red boxes.

Figure-26

Besides, analysers also have “CO₂+He” analysis mode, and “Ar+He” mode. Operators can choose the proper one analysis mode as per your measurement request.

The screenshot shows the 'Gas type' dropdown menu with the following options: N2+He, N2, CO₂+He, Ar+He, Kr+He, H₂+He, and H₂. The 'N2+He' and 'CO₂+He' options are highlighted with red boxes.

Figure-27

Ar +He: The molecular cross-sectional area of argon is not a definite value but a range, it is necessary to select the right value according to the characteristics of the material. If this mode is selected, a pop-up box will appear prompting the entry of the argon molecular cross-sectional area.

Automatic Surface Area Analyzer LMSAA-101

H₂ /H₂+He: Test the material hydrogen storage performance at a target temperature.

Analysis Mode: Gas type: **H₂+He** (dropdown)

Sample Info: Sample Type: Non Fine f (dropdown)

Molecular: 0.21 nm² Density: 0.00128059

Adv. Params. situ degassing settir

Sample Parameters:

Unit No.	Warm Volume	Name	Mass	True Density	Cold Volume
☐ Unit 1	35.0000 ml		1.00000 g	2.0000 g/ml	10.0000 ml
☐ Unit 2	35.0000 ml		1.00000 g	2.0000 g/ml	10.0000 ml
☐ Unit 3	35.0000 ml		1.00000 g	2.0000 g/ml	10.0000 ml

Air leakage rate(Pa·m³/s)

Unit 1: 0 Unit 2: 0

Unit 3: 0 Cavity: 0

Manifold information: Manifold volume(ml) 21.825000

Analysis Parameters:

P/Po Point List: Default Points (dropdown)

Po Mode: Atmos. modification (dropdown)

Gas Supply Mode: Auto Control (dropdown)

Cryogen Level Control: Enabled (dropdown)

☒ Run Adsorptio

☒ Auto Back fillings

☒ BET Smart Point

Add Delete

Analysis Information:

Sample No.:

Name:

Submitter:

Degassing:

Operator:

Checker:

Company:

Adsorption:

Analysis:

Target Temp(°C): 0

Figure-28

Analysis Mode: Gas type: **H₂** (dropdown)

Sample Info: Sample Type: Non Fine f (dropdown)

Molecular: 0.21 nm² Density: 0.00128059

Adv. Params. situ degassing settir

Sample Parameters:

Unit No.	Warm Volume	Name	Mass	True Density	Cold Volume
☐ Unit 1	35.0000 ml		1.00000 g	2.0000 g/ml	10.0000 ml
☐ Unit 2	35.0000 ml		1.00000 g	2.0000 g/ml	10.0000 ml
☐ Unit 3	35.0000 ml		1.00000 g	2.0000 g/ml	10.0000 ml

Air leakage rate(Pa·m³/s)

Unit 1: 0 Unit 2: 0

Unit 3: 0 Cavity: 0

Manifold information: Manifold volume(ml) 21.825000

Analysis Parameters:

P/Po Point List: Default Points (dropdown)

Po Mode: Atmos. modification (dropdown)

Gas Supply Mode: Auto Control (dropdown)

Cryogen Level Control: Enabled (dropdown)

☒ Run Adsorptio

☒ Auto Back fillings

☒ BET Smart Point

Add Delete

Analysis Information:

Sample No.:

Name:

Submitter:

Degassing:

Operator:

Checker:

Company:

Adsorption:

Analysis:

Target Temp(°C): 0

Figure-29

NOTICE: Must connect an empty P0 tube on P0 port when you choose “actual determination” option at P0 mode menu.

Sample type: The sample type can be selected as "fine powder" or "non-fine powder". For light samples, the fine powder type can be selected, thus effectively preventing pumping away during the vacuum and backfill inflation phases.

3) Zone IV: Air leakage rate (Pa.m3/s)

The “Air leakage rate” is for amending errors caused by analyzer system slight leakage, environment temperature fluctuation etc. Usually, the software displays this value automatically, or you can try it by yourself. The “Unit 1” for port I, the “Unit 2” for port II, the “Unit 3” for port III and the “Cavity” for cavity.

Air leakage rate(Pa·m3/s)			
Unit 1:	0	Unit 2:	0
Unit 3:	0	Cavity:	0

Figure-30

4) Zone V: Analysis Parameters

P/P0 Point List: Two choices “Default Points” and “Manual Input” can be selected. Normally, we recommend choosing the “Default Points”. If operators want to run the “BET” or “Pore Analysis” with your preferred P/P0 points, please choose the “Manual Input” in the pull-down menu “P/Po Point List” window , then add your preferred P/P0 points.

Figure-31

P0 Mode: P0 value can be measured by four modes, “Atmos. modification” 、 “Previous value”, “Measure actually”, “Manual Input” mode. Different analysis mode selected different P0 Mode as default.

Figure-32

To determine the actual saturation P0 automatically, operators should choose the “Measure actually” from the pull-down menu “P0 mode”. Operators must connect an empty P0 tube on the P0 port when choosing “Measure actually” option.

Gas Supply Mode: There are two gas supply modes, one is by the “Auto Control” and another is by the “Fixed Quantity”. The “Auto Control” means the software will control the dosing amount each time according to samples’ adsorption amounts last several points intelligently. The “Fixed Quantity” means dosing fixed amount mol gas each time. The “Auto Control” option is recommended for most sample analysis.

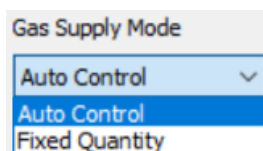


Figure-33

Cryogen level Control: The “Cryogen Level Control” used for controlling cryogen level, including two modes: “Enabled” and “Disable”. The “Enabled” option is recommended. Select it from the pull-down menu.

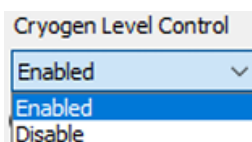


Figure-34

Run Adsorption Only: Check the “Run Adsorption Only” if operators only want to run adsorption procedure. Uncheck it to run both adsorption and desorption procedures.

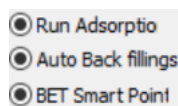


Figure-35

Auto Back Fillings: The “Auto Back Fillings” means analyzer will do back filling operation when analysis work is finished.

BET Smart Point: After checking the box, the software will automatically select points for BET fitting.

5) Zone V: Analysis Information

The “Analysis Information” for inputting analysis information for note purpose. The software will choose some of them to create the final analysis reports names. All our systems software has the store and remember function. Operators can use the same analysis information for the next time.

 A screenshot of a software interface titled "Analysis Information". It contains a form with several input fields: "Sample No.", "Name", "Submitter", "Degassing", "Operator", "Checker", "Company", "Adsorption", and "Analysis". Each field is represented by a rectangular text box.

Figure-36

8.4 Analysis Introduction

8.4.1 Determining Amount of Sample to Use

Clean, dry sample tubes are essential for accurate results. How much sample to use can be determined best by experiment. In general, a sample providing 20 square meters of total surface area is recommended for nitrogen analysis. Less than this may cause variability of results; considerably more than this extends unnecessarily the time required for analysis.

Smaller quantities are required for samples having high surface areas. These samples require careful weighing after degassing because a small error may represent a considerable percent of total weight. Proper weighing techniques are most important in this case. Use no less than 100 mg to reduce the effect of weighing errors. Care should be taken when loading powders; the accessory funnel is useful for this purpose. Large granules or chunks may be loaded with forceps.

NOTICE: Avoid touching the sample with your fingers because oils, moisture etc. may be transferred to the sample and can alter results or create degassing problems.

8.4.2 Determining the Mass of the Sample

Analysis results are expressed in units of surface area per gram of sample; therefore, the true mass of the sample must be known. Carefully weigh each sample tube set and sample as described below.

To eliminate weighing errors, recommend to use a 0.0001 precision balance. Write the Sample Tube number on the Sample Data Worksheet. Place a sample container (like beaker) onto the balance. Tare the balance and allow it to stabilize at zero (0). Place the sample tube set (filler rod, if used) on the sample container. Record the stabilized weight on the Sample Data Worksheet as Mass of empty sample tube.

NOTICE: Do not touch the sample or filler rod with bare hands while performing the following steps. Doing so could affect the accuracy of results.

Slowly add the sample to the sample tubes by a clean and dry funnel.



Figure-37

If some sample clings to the inside of the sample tube above the last 26 cm of the tube, use a pipe cleaner or lint-free wipe to remove it. Weigh the sample tube set containing the sample and record the weight on the Sample Data Worksheet as Mass of sample tube plus sample (Before Degas). Subtract the Mass of empty sample tube from the Mass of sample tube plus sample; record this value as the Mass of sample.

NOTICE: Care should be taken in choosing, conditioning, and filling sample tubes. Sample tubes for degas and analysis stations have a 1.3cm outside diameter (OD). Stepped ferrules, smaller O-rings, and filler rods are available for adapting degas and analysis ports. Filler rods help to ensure accuracy in samples with lower total surface areas by reducing the free-space volume. It is generally a good practice to use filler rods for samples having less than 100 square meters of total surface area. Filler rods are unnecessary for samples with total surface areas greater than 100 square meters. Filler rods can interfere with thermal transpiration correction and, therefore, should not be used when performing micropore analyses. Sample tubes and filler rods must be clean and dry before samples are added and weighed.

8.4.3 Degassing the Sample

Most solid materials absorb moisture and other contaminants when exposed to the atmosphere. The sample must be clean when an analysis is performed. The sample is heated and placed under vacuum to remove moisture and other contaminants. This process is referred to as degassing the sample. Degassing the sample is easy and fully automatic. In the procedures are as follows:

- 1) Install your sample tubes on degassing ports.
- 2) Screw up the heating mantle elevator slowly.
- 3) Place the heating mantle under the bulb of the sample tube, cover some heat insulation cotton on top of the mantle to ensure a sound degassing condition.
- 4) Run the software on computer, locate the “Sample Process” area and click the “Parameter setting” to fill in samples information.

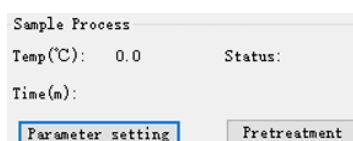


Figure-38

- 5) Select the “Sample Pretreatment” and “Sample Type”. The “fine powder” type can be selected for very light samples, thus effectively preventing the sample from being pumped away during the evacuation and backfilling phases. There are three stages.

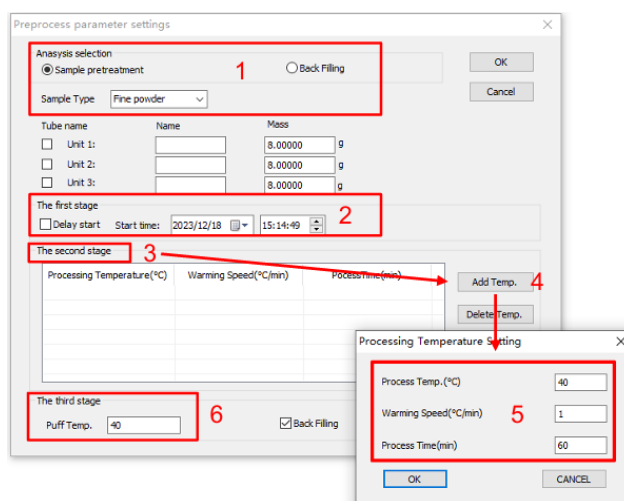


Figure-39

The first stage: If want to start the degassing from a fixed specific time, please fill in the “Delay Time” information. This feature is very important for operators who expect analyzer to work at the night time, then, operators can start to analyze the well degassed samples at their morning working time when they arrive labs.

The Second stage: Click “Add Temp.” Operators need to fill in the Process Temp. (°C), Warming Speed(°C/min), Process Time(min), after completing setting then click “OK”.

The third stage: The “Puff Temp.” means gas inlet temperature, suggest to keep them as the default value. Suggest to check the “Back Filling” function when sample degassing, otherwise, operators should do the back filling step after the degassing finished. Click “OK” after all information had filled in. Then, click the “Pretreatment” button to begin degassing operation and analyzer will finish the degassing procedure automatically.

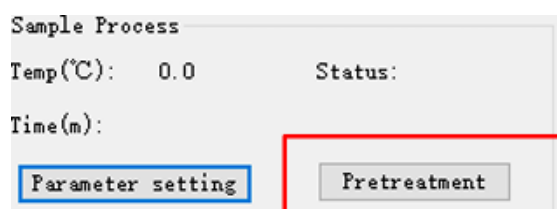


Figure-40

After degassing has completed, transfer the sample tube to the analysis port to start the analysis.

8.5 Data post-processing

8.5.1 P/P0 Data Points Selection

Click the “Points Selection” from software main interface; then, operators can see the Points Selection window.

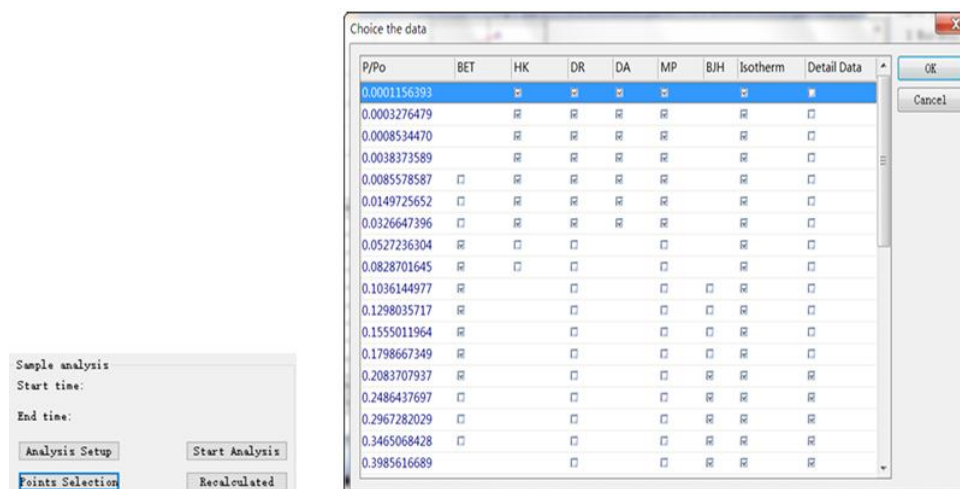


Figure-41

Operators can select appropriate P/P0 points set for different theory calculating models. When points set are added or reduced, need to click the “Recalculated” button on main software window interface to make them take effect.

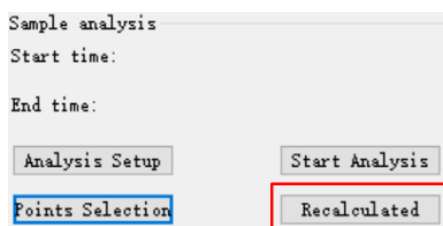


Figure-42

Click the “OK” button after P/P0 points are selected.

8.5.2 Analysis Results Review

1) Isotherm

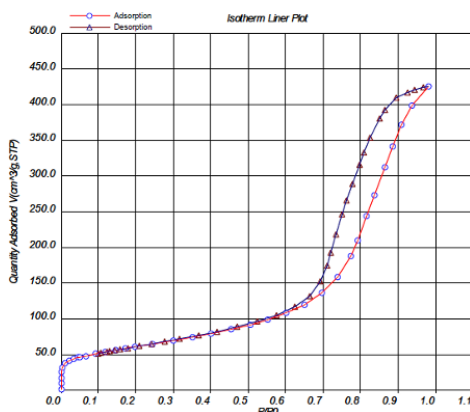
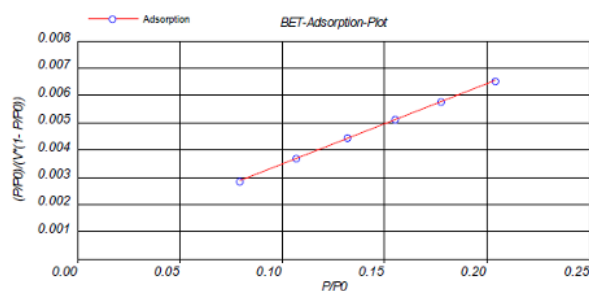


Figure-43

2) BET Report



BET Tabular Report

P/P0	Quantity Adsorbed(ml/g)	(P/P0)/(V*(1-P/P0))	Single point BET
0.204220978280	39.377032724975	0.006517257486	136.386652719577
0.177790290477	37.614464968106	0.005748711971	134.608934920853
0.155209208093	36.010058128540	0.005102047401	132.406526320567
0.131902729410	34.423011518071	0.004414042693	130.062965237400
0.106822138922	32.735440388141	0.003653466521	127.260180759644
0.079308683714	30.475975612315	0.002826500571	122.125980217516
Slope	Intercept	Vm(ml/g)	C Value
0.029546391950	0.000498725982	33.283277578275	60.243739128100
R	Multi-BET Area	Langmuir Area	Pressure Curve
0.999939342547	144.864671220544	200.325872133311	

Figure-44

3) BJH Adsorption Pore Distribution

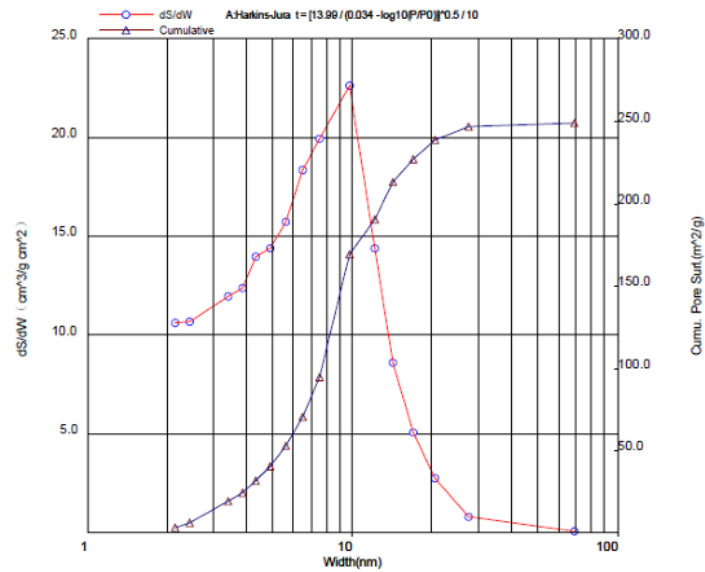
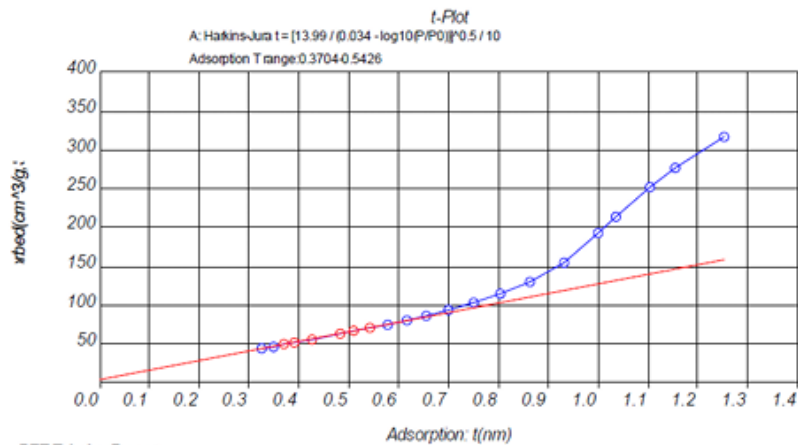


Figure-45

4) t-Plot Report



BET Tabular Report

P/P0	Adsorbed thickness(nm)	Quantity Adsorbed(ml/g)			
0.362097	0.542603	69.581730			
0.311984	0.509056	65.270228			
0.270234	0.481966	61.828144			
0.185646	0.427552	55.122063			
0.131913	0.391295	50.766543			
0.103405	0.370446	48.185185			
Slope	Intercept	C	Micro Vol.(ml/g)	Micro Area(m ² /g)	External Area(m ² /g)
123.892517	2.234150	0.999940	0.003456	9.320588	191.636945

Figure-46

8.6 Reports

- 1) **Print Reports:** Operators can print the contents of one or more sample data files, if had installed the printer. Click File-----Print, the containing file types are displayed. Select the desired files to print.
- 2) **Export Reports as Excel Format:** Operators can export the contents of one or more sample data files as Excel Format.
Click File-----Export EXCEL. Locate the folder that you expect to store the file.
- 3) **Export Report as PDF Format:** Operators can export the contents of one or more sample data files as PDF format. Click File-----Print, select the "Adobe PDF" from the draw-down list of printer name. Click OK to get the PDF format report.
NOTICE: The measurement analyzer must had installed the Adobe Acrobat software if you want to print the reports as PDF format. Click to download the Adobe Acrobat software.
- 4) **Export Report as ACI Format:** Operators can save and export the contents of one or more sample data files as ACI format, further, open the ACI format by TXT software. Click File-----Save As. Locate the store folder that you expect to store the file. Locate the file, right click your mouse and open as TXT format.

9. Maintenance

9.1 Cleaning the Analyzer

The analyzer should be cleaned every three months, or sooner if required. Clean the outside casing of the analyzer with a clean cloth dampened with isopropyl alcohol (IPA), a mild detergent in water, or a 3% hydrogen peroxide solution. Use only a mild detergent in water to clean the protecting doors. Use only a mild detergent in water to clean the shields. Do not use isopropyl alcohol. Isopropyl alcohol could damage the surface of the shield.

9.2 Lubricating the Elevator Screw

The Dewar elevator screw should be lubricated every six months, or sooner if required. Apply the supplied lubricating oil to the elevator screw, accessed from the rear of the instrument, as needed.

9.3 Checking the Analysis Port Dewar

The analysis Dewar should be cared after every operation. Ice and suspended frost particles may accumulate in the bottom of the analysis port Dewar. Particles or deposits exceeding 0.60 cm in depth may jam between the bottom of the sample tube and the bottom of the Dewar, causing the Dewar not to raise fully. Accumulated ice must be removed before placing the Dewar on the elevator. To ensure problems do not develop due to ice accumulation, check the Dewar after each use. Clean on a weekly basis as follows:

Step 1: Lift out the entire analysis port Dewar.

Step 2: Pour out liquid nitrogen into an appropriate storage cryogenic container.

Step 3: Rinse the Dewar with warm water to melt any ice accumulation which may remain in the Dewar, then dry thoroughly.

Step 4: Put upside down the Dewar for drying it.

9.4 Inspecting and Changing Vacuum Pump Fluid

The environmental temperature of the vacuum pump is 12 to 40°C. The fluid in the vacuum pump should be changed every six months or when the efficiency of the vacuum pump declines. The fluid can first be inspected to determine if a change is necessary.

Step 1: View the vacuum pump fluid through the oil level indicator window (in analyzer left side). Fluid in good condition is clean, clear or light in color, and transparent. If the color of the fluid is darkened to 4.0 value, the fluid should be changed.

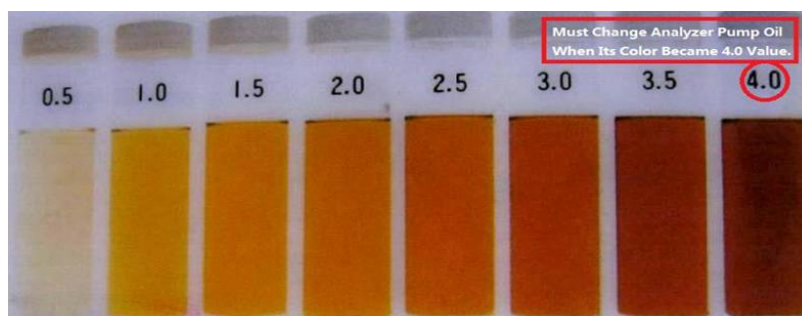


Figure-47

Step 2: Open analyzer back cover.

Step 3: Disconnect the vacuum pump power cord from its power source.

Step 4: Disconnect the vacuum pump hose and O-ring from the top of the vacuum pump.

Step 5: Grasp the handle on top of the vacuum pump and lift it out of the analyzer; place the vacuum pump on a worktable.

Step 6: Place a waste container under the drain fitting, remove the plug from the drain spout and allow the oil to drain into the waste container.

Step 7: Add fresh fluid to the oil-fill port until the level is midway between the two indicator lines.

NOTICE: Adding fluid above the midway position on the fluid level indicator may cause fluid to splash into the vacuum hoses and leak from the internal vacuum pump.

Step 8: Check the washer or O-ring used at the oil-filling port; replace if necessary.

Step 9: Place the vacuum pump back into the analyzer and reconnect the vacuum pump hose by clamp.

Step 10: Reconnect the vacuum pump power cord.

Step 11: Place the analyzer ON/OFF switch in the ON position. The pump must run a few hours to eliminate air and moisture from the fresh fluid and to produce efficient vacuum operations.

10. Troubleshooting

Most operational problems are caused by leaks (commonly around the sample tube O-ring at the analysis port), sample weighing errors, use of too much analysis bath fluid in the Dewar at the start of an analysis, or entry of incorrect system volume for analysis. Always check these first when expected analysis results are not obtained. Some common operational problems, which are not indicated on the video monitor screen, and their respective causes and solutions are provided in the following table.

Trouble	Reason	Solution
Analysis Dewar cannot be raised or lowered.	Elevator that moves Dewar stuck in up position, down position, or somewhere in between.	Check for possible obstruction to elevator movement.
		Restart the working computer after a long period continuous working.

11. Accessories

Optional Accessories

Test gas (Ar, Kr)