BELSORP

High Precision - Specific Surface Area / Pore Size Distribution Measuring Unit

Instruction Manual

Ver. 1. 3. 3

BELSORP-max

BELSORP series

【 Content 】

Cautions

1. BEL JAPAN, INC. reserves all rights to this instruction manual and to the software contained herein.
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3. All the information contained in this instruction manual and the software specifications are subject to change without notice.
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5. We are not liable for any effect resulted from using this instruction manual and the software contained herein.
6. Please store the system disk carefully.
7. We are not liable for any damages resulting from improper use of this product.
8. The warranty term for **BELSORP-max** shall be for one (1) year after delivery.
9. Although this manual has been prepared with every precaution to ensure accuracy, please contact our company if you find any questionable points, errors, omissions, etc. in this manual.
10. Product(s) (including parts, technical data or information thereto) described in this manual shall be subject to export control laws and regulations of Japan or the US. You need to obtain the approval from appropriate government (s9 when you export if such laws ant regulations require.

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For safety use

Thank you very much for selecting **BELSORP-max.**

This manual describes safety precautions, instrument installation, sample measurement, etc., of which users should be aware before using this product.

Read through this instruction manual before you attempt using **BELSORP-max.**

After you read this manual, please keep it so as to be available at any time.

Precautions

For safety use, various precautions are described throughout this instruction manual. Thoroughly understand the precautions first, and then read the text. Be sure to follow the precautions.

To prevent any accident, the precautions that require your special attention are highlighted with a frame as shown on the right.

Indication level of severity

Warning	**Hazardous or unsafe practice that could result in severe injury or dead.**
Caution	**Hazardous or unsafe practice that could result in minor injury, or equipment and/or material could be damaged.**

Warning symbol

Symbol	**Caution (attention) is required.**
Symbol	**Prohibited (never do) .**
Symbol	**Enforced (must do) .**

Warning

Turn off power and disconnect the power plug in the event of any smoke, abnormal smell, or noise.

- **Using with abnormal conditions may result in fire and/or electric shock. Please contact our company.**

Turn off power and disconnect the power plug in the event of spilling water in the instrument.

- **Using the instrument with water spilled may result in fire and/or electric shock. Please contact our company.**

Install the instrument in a flat place with less vibrations, and take appropriate measures to prevent from falling down.

Use single phase AC power indicated on the back of the instrument, and be sure to install a grounding conductor.

Do not damage the power cable.

- **It may result in electric shock and/or failure of the instrument.**

Do not remove the rear panel.

- **There are rotating parts including a fan, and electric-charged parts in the instrument; therefore, touching directly these parts may result in injury and/or electric shock.**

Caution

Fix the power cable firmly so that it is not disconnected.

- **A plug is liable to generate heat. It may cause fire.**
- **The cable own weight applies a tension to the connections; therefore, be sure to fix it firmly.**

Use the instrument in a place with less electric noises.

- **Electric noise may cause improper operation.**

Please request us internal cleaning, when dust is accumulated inside the instrument.

- **It may result in fire and/or failure of the instrument.**

Do not install the instrument in a place with high temperature and humidity.

- **Use the instrument in a place having a temperature of 10 to 35 °C, and humidity of 20 to 80 %.**

Specifications

Measurement method	Volmetric gas adsorption method＋AFSM^{TM}	
Adsorptive	N_2, Ar, Kr, H_2, CO, O_2, CH_4, and other non-corrosive gas Steam (A corrosion resistant option is required for adsorption measurement of corrosive gas such as NH_3 and amine, and organic vapor such as CH_3OH, C_6H_6, etc.)	
Number of samples to be measured	Standard mode ($P/P_0 = 10^{-8}$ to 0.997): 1 to 3 samples High precision mode ($P/P_0 = 10^{-8}$ to 0.997): 1 to 2 samples (according to AFSM^{TM})	
Specific surface area measurement range [1)]	0.01 $m^2 g^{-1}$ or more (N_2 / 77K) 0.0005 $m^2 g^{-1}$ or more (Kr / 77K)	
Pore size distribution (diameter)	0.35 to 500 nm	
Pressure sensor	133 kPa	5 units (Accuracy: ±0.25 % of F.S.)
	1.33 kPa	2 units (Accuracy: ±0.5 % of R.)（OP. + 1 unit）
	13.3 Pa	1 unit (Accuracy: ±0.15% of R.)（OP. + 1 unit）
Pressure resolution	1.6×10^{-6} Pa	
manifold temperature	40 °C (Option 50 °C)	
Dewar vessel	Volume: 2.6 L　　Holding time: 60 h	
Sample cell	About 1.8 cm^3 (Option: 5 cm^3)	
Exhaust system	Turbo molecular pump + Rotary pump : Attainable vacuum: 6.7×10^{-7} Pa or less (manufacturer's specification) (Option: Oil free exhaust system)	
Vacuum gauge	Cold cathode gauge (2×10^{-7} pa to 1 Pa)	
Measurement program	Pretreatment and adsorption/desorption isotherm measurement	
Analysis program (BELMaster^{TM})	・ Adsorption /desorption isotherm ・ Specific surface area by Langmuir method ・ Pore volume calculation by DA method ・ Micro-pore analysis by MP method, HK method, and SF method ・ Difference of adosorption method ・ Specific surface area by BET method ・ Meso-Pore analysis by DH method, CI method, BJH method ・ Micro-pore volume and micro-pore diamether by t-method, α_s method ・ Equivalence diffential adsorption heat analysis ・ Pore size distribution analysis by the NLDFT/GCMC (BELSim^{TM}) (Optional)	
Required PC environment	OS: Windows 2000, XP, Vista, 7　　CPU: Intel Processor Memory: 2 GB or more　　Hard disc: Free spaces of 1 GB or more USB port: USB 2.0	
Auxiliary equipment	Rotary pump displacement: 50 L min^{-1}　　Attainable vacuum: 6.7×10^{-2} Pa	

Utility	He, absorptive (N_2, Ar, etc.) : 1/8” Swage lock joint (0.1MPa (G)) Air for valve operation : 1/4” Plastics tube quick connect (0.5 to 0.6MPa (G)) Rotary pump connection port : OD ϕ11 mm Hose nipple
Dimension / weight	W565 x H850 x D580 mm, 84 kg (Excluding a vacuum pump and computer related equipment)
Power	Single phase, AC100 to 120 V or AC200 to 240 V /800 VA (max. 700VA for roughing vacuum pump)

1) The minimum measurable specific surface area depends on the sample density.

Optional equipment specifications

Temperature controller	Power	Single phase, AC100 to 240 V / 600 VA	
	Dimension / weight	W145 x H200 x xD300 mm, 4 kg	
450 °C heater	Number of ports	3	
	Temperature range	50 to 450 °C	
	Temperature stability	±0.2 °C	
	Overheat detection temperature	500 °C	
	Temperature control	PID control using a temperature controller in the temperature device controller	
	Dimension / weight	W280 x H240 x D270mm, 6.5 kg	
550 °C heater	Number of ports	3	
	Temperature range	50 to 550 °C	
	Temperature stability	±0.5 °C	
	Overheat detection temperature	600 °C	
	Temperature control	PID control using a temperature controller in the temperature device controller	
	Dimension / weight	W280 x H240 x D270 mm, 9 kg	
1100 °C electric furnace	Number of ports	1	
	Temperature range	50 to 1100 °C	
	Temperature stability	±0.5 °C	
	Overheat detection temperature	1150 °C	
	Temperature control	PID control using a temperature controller in the temperature device controller	
	Dimension / weight	W280 x H300 x D270 mm, 6 kg	
Water bath	Operating temperature range	-10 °C to 70 °C	
	Wetted material	SUS304, SUS410, silicon rubber	
	Dimension	W280 x H240 x D270 mm	
	Weight	Open system	6 kg
		Closed system	6 kg
	Volume	Open system	2.1 L
		Closed system	Internal bath: 0.7 L
			External bath: 1.4 L
	Joint	OD. ϕ13 mm hose nipple	

Gas selector	Number of ports	4
	Gas connection port	1/8” swage lock joint (0.5 to 1.5 bar)
⚠ In case of using corossive gas, use the specially designed port V2 or V3.		
Flow gas pretreatment line	Connected to the unit using a flow gas sample cell	
Corrosion resistant exhaust system	Rotary pump (Fonbrin oil type) : Displacement: 30 L min^{-1} Attainable vacuum: 200 Pa	
Oil free exhaust system	Diaphragm pump: Displacement : 15 L min^{-1} Attainable vacuum: 350 Pa	
	Corrosion resistant diaphragm pump: Displacement: 20 L min^{-1} Attainable vacuum: 200 Pa	

Before measuring samples

About BELSORP-max

The gas adsorption method is one of the important experimental means for characterizing fine particles or porous materials. However, operation of the conventional adsorbing device was complicated, and a skilled person was required to obtain the measurement data accurately. BELSORP-max, an automatic gas adsorption measuring unit for gas adsorption, vapor adsorption and chemisorption, is designed to measure accurately, quickly the properties of fine particles, such as the specific surface area, pore size distribution and metal dispersion, by easy operation using the gas adsorption method.

1. Various measurement items

Various items, such as the specific surface area, pore size distribution and metal dispersion, can be measured using a single measuring unit, by applying gas adsorption, vapor adsorption, and chemisorption.

2. A wide range of adsorption isotherm

The adsorption isotherm can be measured in the range from $P/P_0 = 10^{-8}$ to 0.997.

3. Vapor adsorption as standard unit

The vapor adsorption measurement is included as standard equipment.

4. Measurement system without refrigerant control (AFSM™)

The refrigerant level control is not required for the unique dead volume measurement system. This eliminates troubles related to the level control, and enables highly repeatable measurement.

5. Automatic measurement of the saturation vapor pressure

In the nitrogen adsorption measurement at liquid nitrogen temperature and the argon adsorption measurement at liquid argon temperature, the saturation vapor pressure is influenced largely from atmospheric pressures, and the measured data especially around the relative pressure of 1.0 have a large error. **BELSORP-max** eliminates the error caused from the saturation vapor pressure change, by measuring simultaneously the saturation vapor pressure of nitrogen and argon using the specially designed port (P0 port).

6. Automatic measurement of the adsorption/desorption isotherm and the metal dispersion

Various own-developed software applications offer high operability. Once the sample cell is set, the subsequent measuring processes are performed automatically. The measured data are analyzed using a variety of software applications, and then output to a display or a printer. Moreover, the instrument can be diagnosed by running the system check software application.

7. Multi-gas applicability

Nitrogen, argon, krypton, steam, and other adsorptive gas (except corrosive gas) can be measured for the respective adsorption isotherms. The optional gas selector enables seven different types of gas to be connected at a time.

8. Measuring up to 3 samples in parallel

Each measurement port is equipped with pressure sensors; and measuring up to 3 samples can be

performed in parallel.

9. Automatic measurement with 8 measuring units

A PC controls up to 8 units of BELSORP-max.

10. Compact and economy

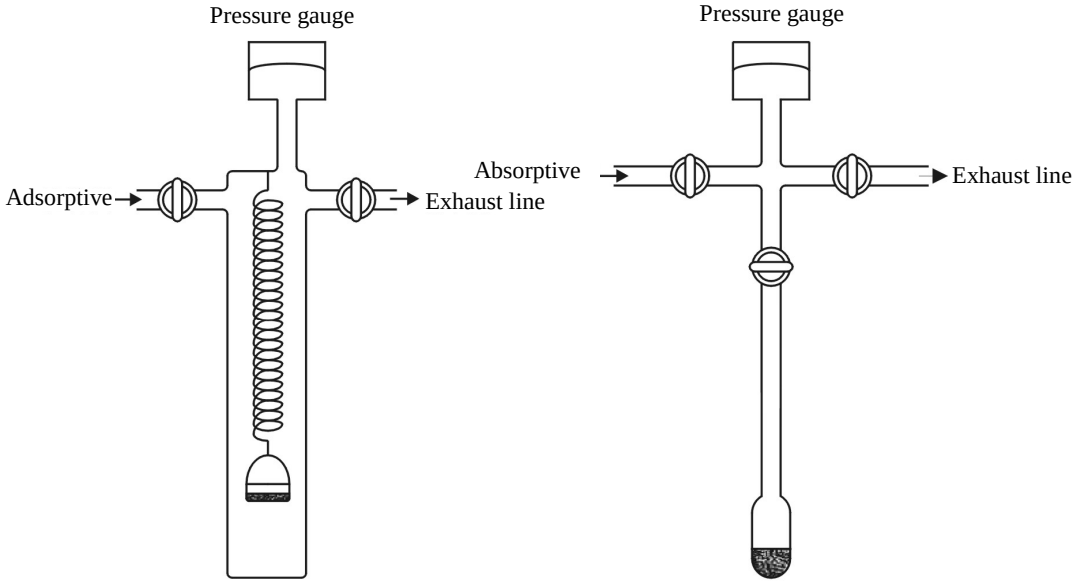
The streamlined design enabled a compact instrument, and offers at a low cost.

The general principle of gas adsorption method, and measurement system of BELSORP-max

ooo Volumetric and gravimetric gas adsorption method ooo

1. General Principle

Among various adsorption measurement methods, the gravimetric and the volumetric gas adsorption method are extensively used (Fig. 1).



Gravimetric gas adsorption method　　Volumetric gas adsorption method

Fig. 1

The gravimetric gas adsorption method is relatively simple in terms of the measurement principle and the experiment method; therefore, it has been extensively used as the adsorption isotherm measurement method. As to the gravimetric gas adsorption method, however, a skillful technique is required for a stable operation of a highly sensitive microbalance, and it is difficult to measure accurately a small weight change by adsorption. Furthermore, the gravimetric gas adosorption method in which the sample is hung down requires verifying whether the sample temperature has reached the adsorption temperature, and also whether it has reached the heat equilibrium since adsorption heat is generated. For the reasons mentioned above, the gravimetric gas adosorption requires a highly skilled technique for obtaining the adsorption data accurately.

As to the volumetaric gas adsorption, on the other hand, it reaches the heat equilibrium quickly before and after the adsorption process. With the volumetaric gas adsorption method, the measurement system volume is measured precisely to determine the adsorption. Then, the adsorption is calculated from the gas pressure change in the measurement system, using the equation of state for gas. Moreover, the sample handling is much easier than those by the gravimetric gas adsorption method.

2. Measurement system of BELSORP-max

BELSORP-max is a full automatic gas-adsorption measuring unit, which adopts the constant volume method for the measurement system. The reference volume buffer and each measurement port are equipped with pressure sensor, all of which enable higher accuracy measurement. Highly repeatable data can be obtained by easy operation.

ooo Pretreatment ooo

1. Principle

The gas adsorption measurement process includes; sample pretreatment, dead volume measurement, adsorption measurement, and then desorption measurement.

Solid surfaces are sensitively influenced by environment. Samples with a larger specific surface area are influenced more strongly. In the pretreatment, appropriate environments shall be provided according to the measurement intended, and it shall not change any properties on the sample surface.

The gas adsorption measurement determines the adsorption per 1g-sample. When the error in mass is as large as 1 %, the measurement accuracy is influenced by the error to the same extent. Therefore, it is required to accurately determine the sample weight from the blank sample cell weight and the sample cell weight after pretreatment.

In order to measure the adsorption isotherm accurately, it is important to perform the pretreatment with suitable conditions. The pretreatment shall be performed on such conditions as the gas and moisture adsorbed surface can be removed without denaturalizing the sample. The sample mass shall be obtained accurately. The pretreatment methods include the vacuum heating treatment, the flow gas heating treatment, etc.

2. Pretreatment with BELSORP-max

Pretreatment with **BELSORP-max** can be performed while vacuuming at room temperature. **The optional heater and electric furnace** enables the vacuum heating pretreatment, whereas the flow gas pretreatment line enables the flow gas pretreatment.

⚠ **When volatile vapor or gas are generated by heating samples, it may contaminate the instrument. Pre-treat or dry samples with BELPREP-vac II, BELPREP-flow II, or other pretreatment devices before the pretreatment with BELSORP-max.**

ooo Dead volume measurement ooo

1. General Principle

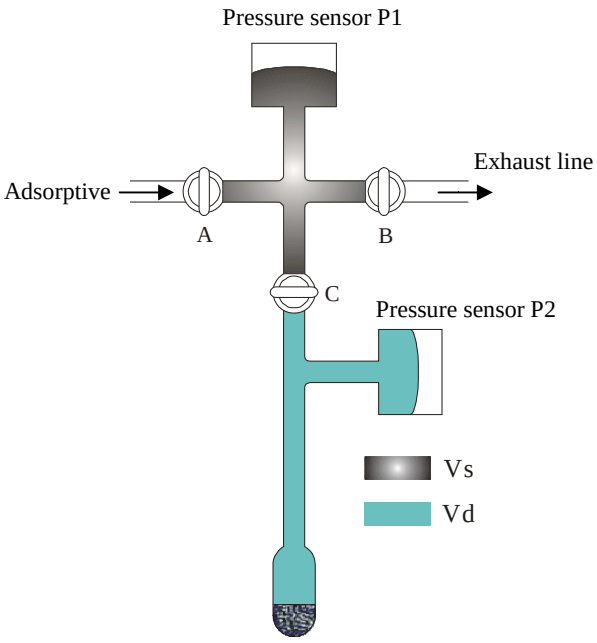


Fig. 2

The space volume in the sample cell is called " Dead Volume (*Vd*) " (Fig. 2).

When the sample cell and the amount of samples change, naturally the *Vd* value changes. Therefore, *Vd* has to be measured for every adsorption measurement. Here, *Vd* is determined as follows.

Install the sample cell to the adsorbing device after pretreatment, and then exhaust the measurement system. Keep the sample temperature at constant. Dose helium gas into the reference volume buffer *Vs*, and measure the pressure $P1_i$ *(1)* using the pressure sensor P1. The reference volume buffer *Vs* has been calibrated, and is specific to each instrument. Open the valve C between *Vd* and *Vs* to diffuse helium gas in the space of *Vd*, and then close the valve C several seconds later. Now, $P1_e$ *(1)* represents the pressure at the pressure sensor P1, while $P2_e$ *(1)* represents the pressure at the pressure sensor P2. With an assumption that the helium gas adsorption to the sample and/or the wall could be negligible, the first point dead volume *Vd (1)* can be determined using the following equation.

$$Vd(1) = \frac{\left(P1_i(1) - P1_e(1)\right) \times Vs}{P2_e(1)}$$

Then, dose additional helium gas, and measure the pressure $P1_i$ *(2)* with the pressure sensor. Diffuse helium gas into the space of *Vd* using the same procedure as for the first point. Now, $P1_e$ *(2)* represents the pressure at the pressure sensor P1, while $P2_e$ *(2)* represents the pressure at the pressure sensor P2. The second point dead volume *Vd (2)* can be determined as the following equation.

$$Vd(2) = \frac{\left(P1_i(2) - P1_e(2)\right) \times Vs + P2_e(1) \times Vd(1)}{P2_e(2)}$$

In the same manner, the *n*-th point dead volume can be expressed as follow.

$$Vd(n) = \frac{\left(P1_i(n) - P1_e(n)\right) \times Vs + P2_e(n-1) \times Vd(n-1)}{P2_e(n)}$$

2. Dead volume measurement system with BELSORP-max (AFSM™)

In the nitrogen adsorption measurement, the sample section is refrigerated with liquid nitrogen to keep the adsorption temperature in the sample section constant. The dead volume (*Vd*) changes as the liquid nitrogen level changes. Generally, the liquid nitrogen level is controlled as to be constant, and the dead volume is fixed.

BELSORP-max uses a new dead volume measurement system (Advanced Free Space Measurement) that does not require the liquid nitrogen level control, and accordingly it offers a compact design, low cost, and high operability. Liquid nitrogen is filled in the Dewar vessel (about 2.6 L) for cooling samples before measurement. The liquid nitrogen in the Dewar vessel vaporizes during the adsorption measurement of samples, and it decreases gradually (it retains for 60 hours or more). The dead volume in the sample cell changes gradually along with this level drop. **BELSORP-max** measures the changing dead volume as follows. A dead volume reference cell (blank sample cell: the same as the sample cell used for measurement) is soaked in liquid nitrogen together with the sample cell (Fig. 3). Prior to the adsorption measurement, the dead volume in the sample cell is measured, as well as those in the dead volume reference cell. Now, the liquid nitrogen level is at level 1 as shown in Fig. 3. $Vd_{(smp)}(1)$ represents the dead volume of the sample cell, while $Vd_{(ref)}(1)$ and $P_{(ref)}(1)$ represent the dead volume of the reference cell and the pressure, respectively. $P_{(ref)}(2)$ represents the pressure in the dead volume reference cell when the liquid nitrogen level drops to level 2. Then, the dead volume of the reference cell $Vd_{(ref)}(2)$ and the difference $\Delta Vd_{(ref)}$ at level 2 are expressed as follow.

$$Vd_{(ref)}(2) = Vd_{(ref)}(1) \times P_{(ref)}(1) / P_{(ref)}(2)$$

$$\Delta Vd_{(ref)} = Vd_{(ref)}(2) - Vd_{(ref)}(1)$$

Since the dead volume change in the sample cell is equal to those in the dead volume reference cell ($\Delta Vd_{(smp)} = \Delta Vd_{(ref)}$, within ± 0.2 % based on our data), the dead volume, $Vd_{(smp)}(2)$, of the sample cell at level 2 can be expressed as follow.

$$Vd_{(smp)}(2) = Vd_{(smp)}(1) + \Delta Vd_{(smp)}$$

$$= Vd_{(smp)}(1) + \Delta Vd_{(ref)}$$

In such a way, highly reliable data can be obtained by measuring the dead volume at each measuring point.

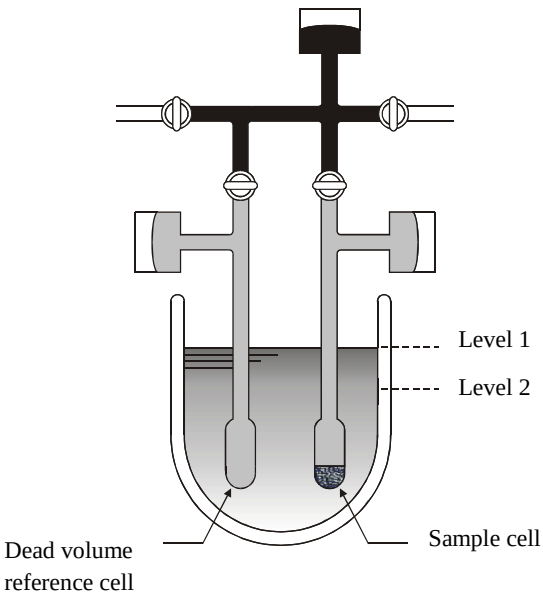


Fig. 3

ooo Adsorption measurement and desorption measurement ooo

1. The generally principle of adsorption measurement

The adsorptive gas pressure dosed to the sample cell from the reference volume buffer *Vs* decreases due to adsorption. In the volumetric method, the amount of adsorption is determined from this pressure difference before and after the adsorption.

Now, $P1_i(n)$ (kPa) represents the adsorptive pressure dosed to *Vs* through the valve-A, as shown in Fig.2 on page 15, at the *n*-th point adsorption measurement. Also, $P1_e(n)$ represents the pressure at the pressure sensor P1, and $P2_e(n)$ represents the pressure at the pressure sensor P2 while reaching the adsorption equilibrium by opening the valve C; the *n*-th point adsorption $V(n)$ (amount of adsorption per 1g-adsorbent being converted into the gas volume in a standard state) can be determined using the equation of state for ideal gas, as follow.

Firstly, the gas volume *V1* in *Vs* (cm^3 (standard state) / g^{-1}), which changed before and after the adsorption, is expressed as the following equation. *Ws* and *T* are the sample weight and the absolute temperature of *Vs*, respectively.

$$V1 = \frac{\left(P1_i(n) - P1_e(n)\right) \times Vs \times 273.15}{101.30 \cdot Ws \cdot T}$$

The gas volume *V2* in *Vd*, which changed before and after adsorption, can be determined from the (*n*-1)-th point equilibrium pressure $P2_e(n\text{-}1)$ in *Vd*, and the *n*-th point equilibrium pressure $P2_e(n)$ in *Vd*, as follow.

$$V2 = \frac{\{P2_e(n-1) - P2_e(n)\} \times Vd \times 273.15}{101.30 \cdot Ws \cdot T}$$

The sample's adsorption change ΔV in the *n*-th point's adsorption process is equal to the gas volume in *Vs* and *Vd* that changed before and after adsorption; therefore it is expressed with the following equation.

$$\Delta V = V1 + V2$$

Hence, the adsorption $V(n)$ at the adsorption equilibrium pressure $P2_e(n)$ is expressed as follow.

$$V(n) = V(n-1) + \Delta V$$

2. The general of principle desorption measurement

The desorption isotherm is measured as follows. Now, $P1_i(n)$ (kPa) represents the adsorptive pressure when *Vs* is depressurized through the valve B while closing the valve C, shown in Fig.2 on page 15, at the n-th point desorption measurement. Also, $P1_e(n)$ represents the pressure at the pressure sensor P1, and $P2_e(n)$ represents the pressure at the pressure sensor P2 while reaching the adsorption equilibrium by opening the valve C. Then, the *n*-th point's adsorption $V(n)$ can be determined using the same equation as for the adsorption process.

ooo Saturation vapor pressure measurement ooo

1. Principle

The nitrogen gas adsorption measurement at liquid nitrogen temperature is effective to determine the specific surface area and pore size distribution of samples. This measurement uses liquid nitrogen as refrigerant to cool samples. The liquid nitrogen boiling point changes as the atmospheric pressure changes, and accordingly the saturation vapor pressure of nitrogen changes. When a fixed value (e.g. atmospheric pressure = 101.3 kPa) is used for the saturation vapor pressure, highly repeatable data cannot be obtained especially in high relative pressure areas.

2. Saturation vapor pressure measurement system with BELSORP-max

BELSORP-max enables highly repeatable measurement in high relative pressure areas by measuring the saturation vapor pressure. The saturation vapor pressure value can be specified by the following two different modes.

1 Saturation vapor pressure measurement mode	This mode is selected for the nitrogen adsorption measurement at liquid nitrogen temperature, and the argon adsorption measurement at liquid argon temperature. The saturation vapor pressure is measured at each adsorption measurement point, by liquefying adsorptive gas at the P0 port, a specially designed port for the saturation vapor pressure measurement. Highly precise data can be obtained.
2 Input value mode	An input value is used to specify the saturation vapor pressure.

ooo Non-ideality correction of the adsorptive gas ooo

1. General principle

The relation between the gas pressure P, the molecular volume V_m, and the temperature T is expressed as:

$$PV_m = zRT$$

Where, z is referred to as compressibility factor, which is always 1 for the ideal gas. For the real gas, however, it deviates from the ideal gas to some extent. The compressibility factor z of nitrogen gas, for example, is 1.000 at pressure of 100 kPa or less, and temperature of 300 K, but it is 0.956 at pressure of 100 kPa, and temperature of 77 K. This means that the number of moles is about 4 % larger than those estimated for the equation of state of ideal gas when the nitrogen gas in a certain volume is refrigerated at liquid nitrogen temperature, and the pressure is around atmospheric pressure. Therefore, when calculating the adsorption by the volumetric method, it is necessary to correct the non-ideality to obtain a highly accurate result.

2. System of BELSORP-max

BELSORP-max corrects the non-ideality of nitrogen gas at liquid nitrogen temperature. When the dead volume Vd is divided into the real volume V_L that is put in liquid nitrogen and the real volume V_R at room temperature as shown in Fig. 4, the dead volume Vd is expressed as follow.

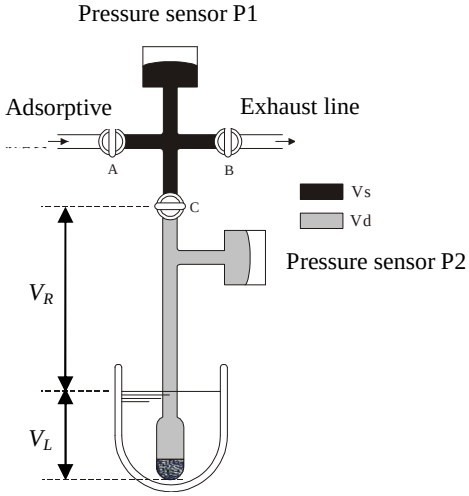


Fig. 4

$$Vd = V_R + V_L \frac{T_R}{77.35} \qquad (1)$$

Where, T_R represents room temperature. The correction of non-ideality at nitrogen adsorption / desorption measurement is applied to V_L. Using a compressibility factor z, the dead volume Vd' after correction is expressed as:

$$Vd' = V_R + V_L \frac{T_R}{77.35 \cdot z} \qquad (2)$$

Accordingly, the correction Δ added to the dead volume measured with helium is expressed as:

$$\Delta = Vd' - Vd = V_L \frac{T_R}{77.35}\left(\frac{1-z}{z}\right) \qquad (3)$$

Once V_L is determined, the dead volume after correction can be determined. Now, the dead volume at room temperature Vd_{RT} can be expressed:

$$Vd_{RT} = V_R + V_L \qquad (4)$$

Therefore, V_L can be determined by subtracting (4) from (1), as follow.

$$V_L = (Vd - Vd_{RT})\left(\frac{77.35}{T_R - 77.35}\right) \qquad (5)$$

The compressibility factor z is determined using the virial equation of state as follow.

$$z = 1 + \frac{B}{RT}P$$

Where, B represents the second virial coefficient, while P represents pressure.

ooo Correction of thermal transpiration ooo

1. General principle

When two vessels with different temperature are connected with a tube, the pressure in a vessel may differ from those in the other vessel. This effect is referred to as thermal transpiration. It may be significant when the pressure is 1 kPa or less, the tube diameter is several millimeters or less, and the temperature difference between the vessels is large.

2. System with BELSORP-max

BELSORP-max corrects the thermal transpiration using the following equation.

$$\frac{P_2}{p_1} = \frac{AX^2 + BX + C\sqrt{X} + \sqrt{(T_2/T_1)}}{AX^2 + BX + C\sqrt{X} + 1}$$

$$X = 7.50 \times 10^{-3} P_2 d$$

$$A = 1.4 \times 10^4 \exp(1.17 \times D \times 10 \times (T)^{-2}$$

$$B = 5.6 \exp(1.40 \times D \times 10) \times (T)^{-1}$$

$$C = ((1.10 \times 10 / D) - 14) \times (T)^{-0.5}$$

$$T = \frac{T_1 + T_2}{2}$$

P_1: Sample cell pressure (Pa)

P_2: Manifold pressure (Pa)

T_1: Sample cell temperature (K)

T_2: Manifold temperature (K)

d: Sample cell inner diameter (mm)

D: Molecular diameter (nm)

Nomenclature and function of individual components

ooo Main unit-front ooo

① Lifting switch

A manual switch to lift the Dewar vessel, water bath, heater, and electric furnace. It is usually controlled by a signal from a computer; however, it can be controlled manually where applicable.

▲: Lift-up button. Press this button for a short period to lift up. Hold depressing this button for 5 seconds to keep lifting up even after releasing it. It stops by pressing the stop button or when it lifts up to the upper limit position.

■: Stop button. It stops by pressing this button while lifting. It blinks when a protective function works due to overload while lifting. In such a case, press the **reset switch** in the temperature controller pocket ③.

▼: Lift-down button. Press this button for a short period to lift down. Hold depressing this button for 5 seconds to keep lifting down even after releasing it. It stops by pressing the stop button or when it lifts down to the lower limit position.

⚠ Do not press the lifting switch while measuring samples.
⚠ Do not put your hand or other materials on the temperature device while lifting.

② Power indicator

This indicates power switch to the instrument. It is interlocked with a power switch on the back of the instrument.

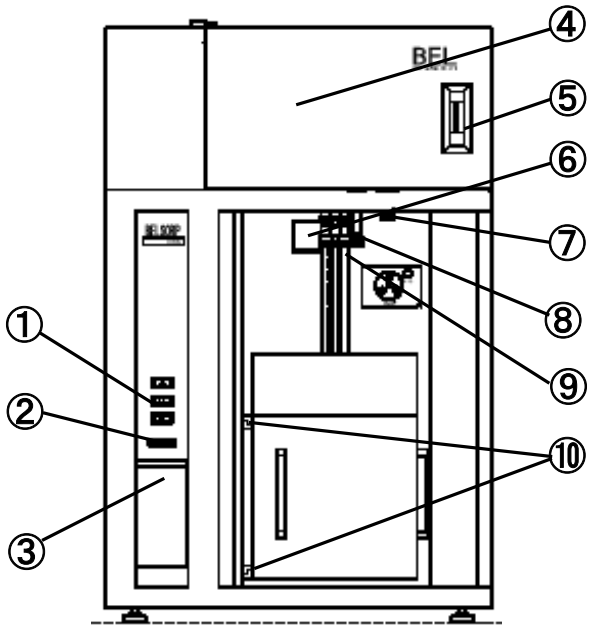
③ Temperature controller pocket

Pull the handle to open the pocket. The following devices are mounted in the pocket.

Temperature controller for the thermostatic chamber: A temperature controller used for the thermostatic chamber.

Reset button for the elevator: The lifting operation stops when the elevator is overloaded. In such a case, it is not recovered automatically even after removing the load. Press this reset switch to release the protection.

⚠ Do not change the temperature setting for the thermostatic air chamber temperature controller.
⚠ Pay careful attention to prevent your finger from being pinched while closing the pocket.



⑥ Needle valve adjustment screw storage shutter

The needle valve adjustment screw is located inside this shutter. Adjust the valve opening with this screw to control the gas flow. Viewing from the front of the main unit, the screw C (for a large flow rate) is on the left, while the screw F (for a small flow rate) is on the right.

⚠ Do not keep the shutter open, because the thermostatic chamber is connected to the shutter.

⑦ Adsorptive gas dosing port (ads. 2)

This is used as a gas connection port by mounting a liquid bottle and an accessory conversion connector.

⑧ Flow gas sample cell connection port

This port is for connecting the flow gas line only (when the optional “flow gas pretreatment line” is selected).

⑨ Measurement port and saturation vapor pressure measurement P0 port

A small tube in the middle is the saturation vapor pressure measurement P0 port, and the other 3 tubes are the measurement ports.

④ Head cover

This cover offers thermal insulation to the manifold temperature.

⑤ Flow meter

The flow rate at the flow gas pretreatment can be monitored (when the optional "flow gas pretreatment line" is selected).

⑩ Elevator hanger

Slide this to mount the temperature devices, such as the Dewar vessel, water bath, heater, electric furnace, etc. Be sure to slide it to the back limit, when mounting the device.

ooo Main unit-back side ooo

① Compressed air (gas) supply port

This is to operate the pneumatic valve for changing the internal flow circuit. Supply compressed air or inert gas of 0.5 to 0.6 MPa (gauge pressure).

② Helium gas supply port

Connect the helium gas line. Supply helium gas of 0.1 MPa (gauge pressure).
Connector: 1/8" swage lock

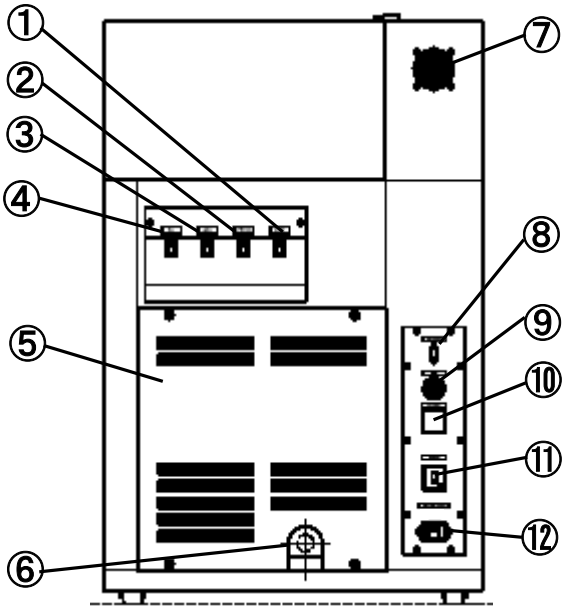
③ Adsorptive gas dosing port (Ads. 1)

Connect the adsorptive gas line. Supply adsorptive gas of 0.1 MPa (gauge pressure).
Connector: 1/8" swage lock

④ Vent connection port

This exhaust port is to discharge the used gas in the flow gas pretreatment. Connect a 1/8" tube, and install the other end to a well-ventilated place.
Connector: 1/8" swage lock

❗ It is dangerous to perform the flow gas pretreatment without the exhaust tube connected to the vent port, since the used gas is discharged. Be sure to install an exhaust line to the vent port when performing the flow gas pretreatment.



⑤ Back door latch lock

Remove this door when performing the turbo molecular pump maintenance. Turn the slot on the clip screw (at 4 locations each) to the vertical direction to remove the door. Turn the slot to the horizontal direction to secure the door.

⑥ Pump hose hole

Connect the roughing vacuum pump to the turbo molecular pump in the instrument with a hose by passing it through this hole.

⑨ USB communication connection port

USB male connector. This is to connect to the computer. Use a USB male (type A) and USB male (type B) cable to connect to the computer.

⑩V. P. AC

Connect the power cable to the rotary pump.

⚠ Be sure to connect the rotary pump power cable to this AC outlet.
⚠ Do not connect any equipment except the pump.

⑦ Cooling fan This fan cools inside the instrument. It is interlocked with a power switch on the back of the instrument.	**⑪POWER switch** Turns on / off the power to the main unit and roughing vacuum pomp.
⑧ Connection port of Temp. cntl. D-sub 9-pin, female connector. This is to connect to the optional temperature controller. Use a straight cable with D-sub 9 pin, male and D-sub 9 pin, female to connect to the temperature controller.	**⑫ Power inlet** Supply power to the main unit. Be sure to use the accessory IEC power cable.

ooo Main unit-left side ooo

① Solenoid valve access door

An access door for the solenoid valve to control pneumatic valve and the internal cooling fan.

② Gas supply port (Aux. 1 to 4)

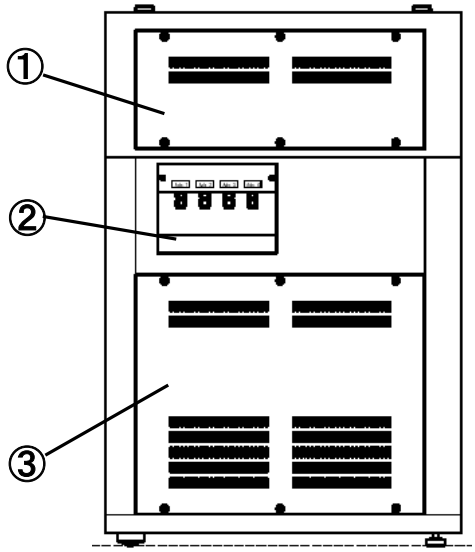
Connect the dosing gas line. Supply the dosing gas at pressure of 0.1 MPa (gauge pressure) (when the optional “gas selector” is selected).

⚠ In case of using corossive gas, use the specially designed port V2 or V3.

③ Control section access door

Electric control parts are located in the door.

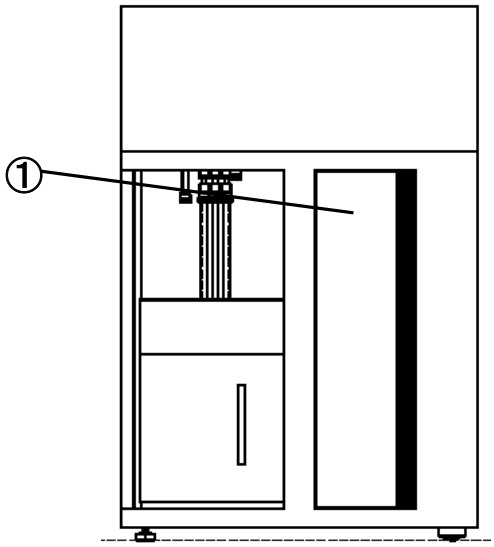
⚠ Some electric-charged parts are exposed inside the door. Except an engineer from our company, do not open this door. Otherwise, it may result in electric shock.



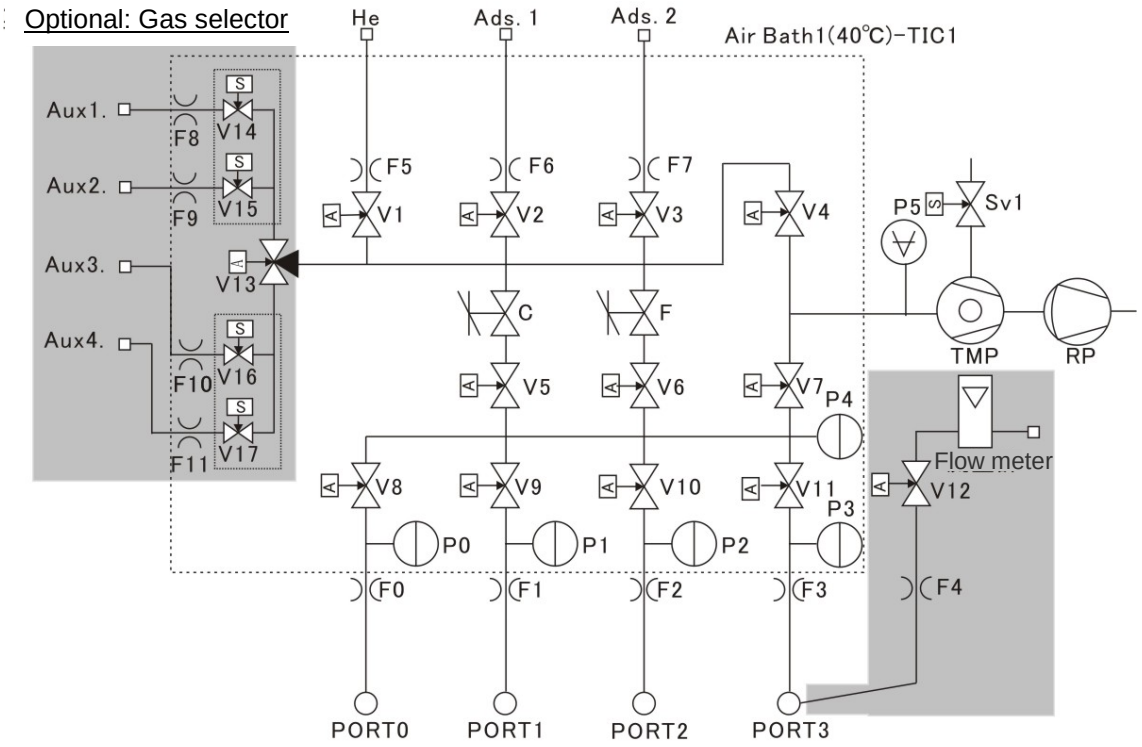
ooo Main unit-right side ooo

① Fan filter cleaning door

A filter is located in the door. This filter is for the cooling-fan of turbo molecular pump. Check periodically the filter and clean it up where applicable.



ooo Main unit-inside ooo



① V1 to V17 : Valve

② C, F : Flow rate adjustment needle valve

③ P0 to P4 : Pressure sensor

④ P5 : Vacuum gauge

⑤ TIC1 : Manifold temperature controller

⑥ Sv1 : Vent valve

⑦ TMP : Turbo molecular pump

① Valve (Standard unit: V1 to 11)

V1 to 11: pneumatic valves

V1 : To dose helium gas

V2 : To dose the Ads. 1 gas

V3 : To dose the Ads.2 gas or vapor

V4 : To exhaust the gas accumulator

V5 : To dose gas to the reference volume (a large flow rate line)

V6 : To dose gas to the reference volume (a small flow rate line)

V7 : To exhaust the reference volume

V8 : A valve between the P0 port and the reference volume

V9 : A valve between the measurement port 1 and the reference volume

V10 : A valve between the measurement port 2 and the reference volume

V11 : A valve between the measurement port 3 and the reference volume

Valve (optional)

Flow gas pretreatment line

A pneumatic valve

V12 : To be used in the flow gas pretreatment

Gas selector

V13: Pneumatic valves, V14 to V17: Solenoid valve

V13 : A valve between the gas selector and the gas accumulator

V14 : To dose the Aux. 1 gas

V15 : To dose the Aux. 2 gas

V16 : To dose the Aux. 3 gas

V17 : To dose the Aux. 4 gas

⚠ In case of using corossive gas use the specially designed port V2 or V3.

② Flow rate adjustment needle valve (C, F)

C : For a large flow rate (pressure) control

F : For a small flow rate (pressure) control

③ Pressure sensor (P0 to P4)

The following pressure gauges, with the indicated respective full scale, are installed to each port.

P0 : 133 kPa
To measure pressure at the saturation vapor pressure measurement port (P0 port)

P2 : 133 kPa
To measure pressure at the measurement port 2

P4 : 133 kPa, 1.33 kPa
To measure pressure in the reference volume

P1 : 133k Pa (Optional: 1.33 kPa, 0.0133kPa)
To measure pressure at the measurement port 1

P3 : 133 kPa, 1.33 kPa, 0.0133 kPa
To measure pressure at the measurement port 3

Accuracy: 133 kPa → ±0.25 % (full scale)
: 1.33 kPa → ±0.5 % (reading)
: 0.0133 kPa → ±0.15 % (reading)

④ Vacuum gauge (P5)

Pirani gauge + cold cathode type pressure sensor

Measurement range: 5 x 10^{-7} Pa to atmospheric pressure

⚠ Do not use the vacumue gauge at pressure of 0.1 Pa or more.

⑤ Manifold temperature controller (TIC1)

Turn on the power switch on the back of the main unit, then the thermostatic manifold temperature is controlled to be 40°C (Option 50 °C). The thermostatic manifold temperature is measured using a platinum temperature detector.

⑥ Vent valve (Sv1)

Turn off the power switch on the back of the main unit, then this valve opens to vent the exhaust line to the open air to prevent the pump oil from flowing back to the instrument when the rotary pump is in operation.

⚠ Be sure to turn off the power switch and vent the exhaust line to the open air, before you shutdown the pump. Otherwise, the pump oil may flow back to the main unit, which may cause failure.

⑦ Turbo molecular pump (TMP)

Attainable vacuum: 6.7 x 10^{-7} Pa or below (manufacturer's specification)

It makes a high vacuum in the system.

⚠ Do not shut down power to the main unit while the turbo molecule pump is in operation, or for 10 minutes after it stops. When power is removed during the operation, or immediately after the shutdown, the system pressure rises rapidly up to atmospheric pressure. It may apply overload to the turbo molecule pump, which may cause failure.

⚠ When accumulated operation time of the turbo molecule pump exceeds 20,000 hours, a maintenance work is prompted on the PC screen. Please contact our company if you find this message.
Accumulated operation time: 20,000 hours → Replace oil

ooo Measurement temperature device ooo

BELSORP-max has the measurement temperature devices as follow. Prepare the appropriate devices according to the desired pretreatment and measurement.

1. Dewar vessel

Dewar vessel

Volume: 2.6 L

Retention time: 60 hours

While sliding, install it to the **elevator hanger** ⑩ on the front of the main unit (P. 21).

⚠ The Dewar vessel is made of glass. Careful attention is required for the handling.
⚠ Do not drop the fitting into the Dewar vessel. It may cause a danger because the Dewar vessel glass is broken.
⚠ When removing the Dewar vessel with the sample cell still installed, be sure to move down the Dewar vessel to the bottom limit.
⚠ When installing to the main unit, slide it to the back limit until it is engaged with the fixing latch.
⚠ Do not use it as installed to different equipment (Our company shall not be liable for any accident or failure resulted from installing to different equipments.).

2. Water bath (optional)

1. General

The water bath temperature is maintained constant by circulating water or oil etc. using a circulating system (optional). While sliding, install it to the **elevator hanger** ⑩ on the front of the main unit (P. 21).

Two different types of water baths are available:

Open system: Temperature is controlled by circulating heat media between the circulating system and the water bath.

Closed system: The water bath has a dual-layered structure. The heat media temperature in the inner structure is controlled as to be constant by circulating heat media in the outer layer.

2. Specification

Temperature control method	Temperature is controlled using a temperature controller in the water bath.
Dimension and weight	W280 x H240 x D270 mm, 6 kg

3. Detailed description

① Detachable thermostatic bath set hole

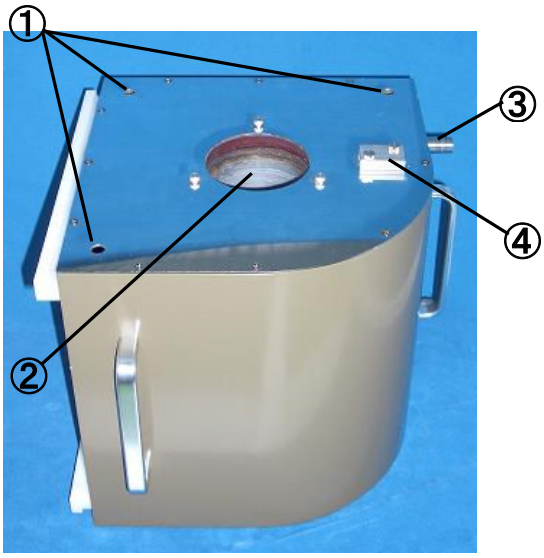
The accessory detachable thermostatic bath is set using these holes.

② Sample cell temperature control hole

When the water bath is lifted for installing to the BELSORP-max main unit, the sample cell enters to this hole.

③ Circulation hose connection port

Connect a hose from the circulation system (Port size: ϕ13 mm hose nipple).

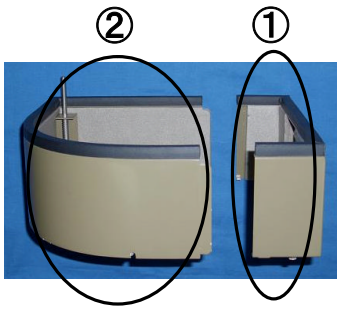


④ Thermometer fitting Attach a platinum temperature detector or a thermo couple to this fitting as required when measuring the water bath temperature or controlling temperature using an external sensor of the circulation system. Loosen the screws on the top of the electric furnace. Thermometer sandwichwed by the upper clamp, and tighten these two screws.	

4. Usage

1. Connect a hose from the circulation system to the circulation hose connection port ③ on the water bath.
2. Circulate the temperature-controlled heat media until the temperature is stabilized.
3. For the vapor adsorption measurement, install a liq. bottle to the adsorptive gas dosing port (Ads. 2) ⑨ on the front of the instrument (P. 21), and mount the accessory detachable thermostatic bath according to the following procedure.

How to mount the detachable thermostatic bath

 **Detachable thermostatic bath**		
Insert the part ① of the detachable thermostatic bath to the **detachable thermostatic bath set hole** ① on the top of the water bath.	Then, mount the part ② of the detachable thermostatic bath.	When it is complete, install the water bath to the instrument. When lifting the water bath, the detachable thermostatic bath opens the **needle valve adjustment screw storage shutter** ⑥ on the front of the main unit (P. 21), and the detachable thermostatic bath temperature is controlled.

⚠ When filling water to the bath, do not wet the instrument and the auxiliary equipments. Otherwise, it many cause rust on the instrument or short circuit of the internal electric equipment.
⚠ Connect firmly the hose from the circulation system.
⚠ When removing the Dewar vessel with the sample cell still installed, be sure to move down the Dewar vessel to the bottom limit.
⚠ When installing to the main unit, slide it to the back limit until it is engaged with the fixing latch.
⚠ When removing the detachable thermostatic bath with the sample cell still installed, be sure to prevent the sample cell from being broken.

3. Temperature controller

1. General

This is a control box to operate the 450 °C heater, 550 °C heater, and 1100 °C electric furnace for BELSORP-max. Connect a communication cable to the **Temp. cntl. computer port ⑧** on the back of the main unit (P. 22). Then the temperature setting can be adjusted from the controller and on the PC screen, and the pretreatment and measurement at high temperature can be controlled automatically.

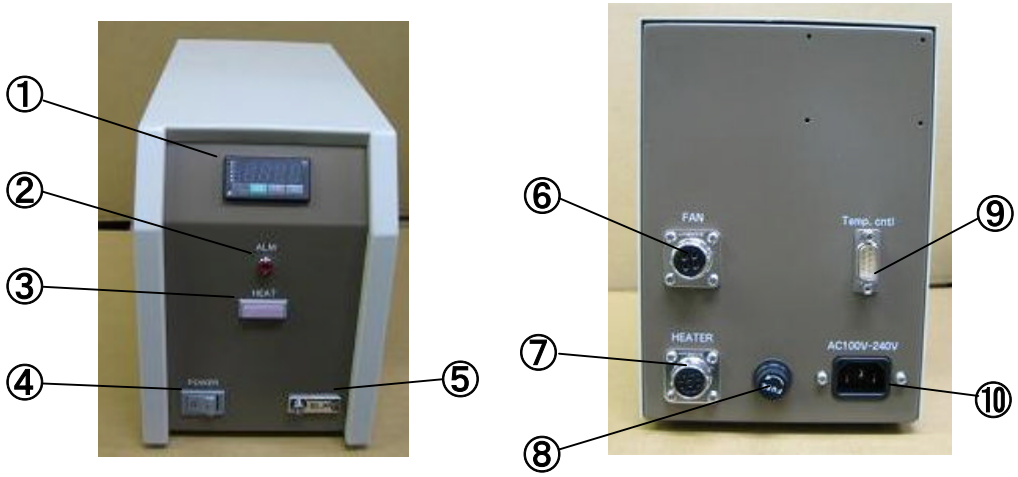
⚠ The heater and the peripheral equipment are hot while heating with the heater or immediately after the heating. Be careful to protect yourself from being burned.
⚠ The sample cell after heating is hot. Be careful to protect yourself from being burned.
⚠ Do not place any flammable material close to the heater while heating.
⚠ Be sure to install the "heat protection cover" on the heater, which is supplied with temperature controller, during heating and awhile after heating.
⚠ When heating up to high temperature using the 1100 °C electric furnace, be sure to use a quartz sample cell.

2. Specification

Temperature control method	PID control using a temperature controller
Dimension and weight	W145 × H200 × D300 mm, 4 kg
Power	AC100 V to 120 V or AC200 to 240 V, 600 VA (when using 550 °C heater)

⚠ Use single phase, AC power according to the specifications of the temperature measurement device.

3. Detailed description



① Temperature controller

This is used to display the current temperature and change the temperature setting. The temperature setting can be adjusted also from the PC screen.

In the case of overshoot is big, peform autotuning according to P. 34.

Default value of P : 5, Default value of I: 120, Default value of D : 20

② Overheat indicator

In case of overheat of the temperature controller, the overheat protection works to turn off heater automatically. In such a case, this LED lights up. The overheat protection works at the following temperature.

When using the 450 °C heater:	500 °C
When using the 550 °C heater:	600 °C
When using the 1100 °C electric furnace:	1150 °C

(When the overheat protection works, remove the cause and then supply power again to recover. In case the cause cannot be clarified, contact our company.)

③ Heater switch

This is a momentary type push button. Press this switch to start heating up to the temperature setting. This switch lights up while heating. Turning on/off heating can be controlled also from the PC screen.

④ Power switch

This is used to turn on/off the temperature controller.

⑤Power indicator

This indicates power supply to the temperature controller. It is interlocked with the power switch on the back of the temperature controller.

⑥ 4-pin female connector (for electric furnace rapid cooling fan)

Connect a 4-pin male connector from the 1100 °C electric furnace (It is not used except with the 1100 °C electric furnace.).

⑦ 10-pin female connector (for heater, electric furnace, thermocouple for temperature control, thermocouple for overheat prevention)

Connect the cable from the relevant temperature device.

⑧ Fuse

The fuse capacity is 10 A.

⚠ When a fuse blows out, remove the cause and replace the fuse. When recovering without removing the cause, it may result in short circuit.
⚠ Turn off the power switch before replacing a fuse.

⑨ Temp. cntl. connection port

D-sub 9-pin, male connector. Connect it to the optional temperature controller. Use a straight cable with D-sub 9-pin, male and D-sub 9-pin, female to connect to the main unit.

⑩ Power cable connection port

Connect a power cable of 110 V/220 V (Do not use wrong power supply.).

4. Usage

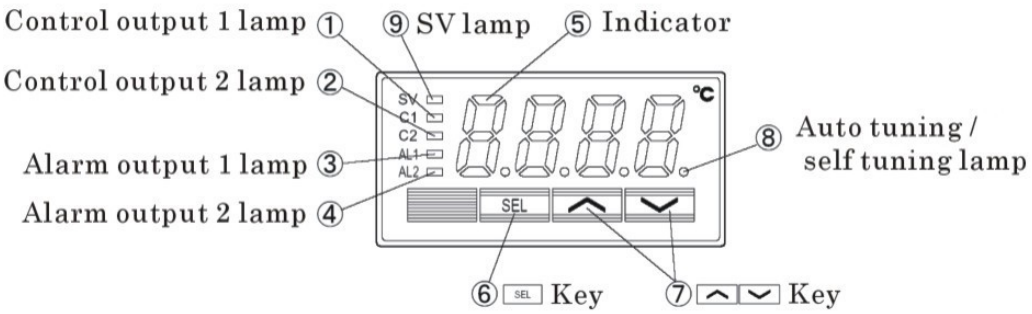
1. Verify that the **power switch ④** on the front of the temperature device controller is off, and connect power using the accessory power cable.

2. Connect a 10-pin male connector from the temperature device to the **10-pin, female connector ⑦** on the back of the temperature controller. When using the 1100 °C electric furnace, connect a 4-pin male connector to the **4-pin, female connector for the fan ⑥** on the back of a temperature controller.

3. Use the supplied straight cable with D-sub 9pin, male and D-sub 9-pin, female to connect to the **Temp. cntl. connection port** on the back of the temperature controller and the back of the main unit, respectively.

4. **Turn on the temperature controller after connecting as described above.** Then, the temperature setting can be performed on the PC. For the pretreatment, specify the parameters appropriately according to “ooo Pretreatment ooo, P. 122”. For the measurement, set the relevant parameters appropriately according to “ooo Setting the measurement parameter ooo, P. 128”.

Parts and function of Temperature controller



① **Control output 1 lamp**
It lights up when Control output 1 is ON.

② **Control output 2 lamp**
It lights up when Control output 1 is ON.

③ **Alarm output 1 lamp**
It lights up when Alarm output 1 is in operation. Also, it flashes on and off when ON delay is in operation.

④ **Alarm output 2 lamp**
It lights up when Alarm output 2 is in operation. Also, it flashes on and off when ON delay is in operation.

⑤ **Indicator**
PV (measured value) or SV (Set value) is displayed. However, parameter name or parameter value is displayed during parameter setting.

⑥ SEL **key**
It is used for switching between PV and SV, selection of parameter block, parameter selection and parameter confirmation.

⑦ **key**
It is used for changing SV, loading parameter and changing parameter.

⑧ **Auto tuning / self tuning lamp**
It flashes on and off during auto tuning or self tuning.

⑨ **SV lamp**
PV (measured value) is displayed normally (when it is not on). Pressing SEL key lights on SV and display SV (setting value). But it is off when displaying parameter or data.
It flashes on and off when the indicator displays PV(measured value) during standby.

1. How to set

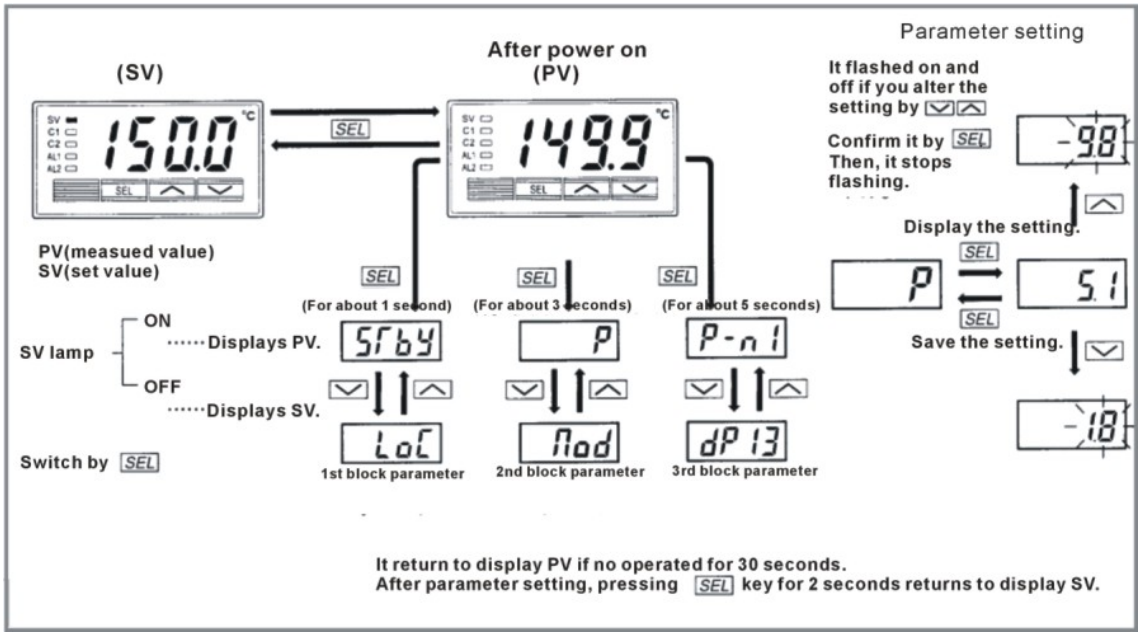
Change set point

- When current temperature is displayed, if SEL key is pressed SV lamp will lights up and set temperature will be displayed on the indicator. At this point, set temperature can be altered.
- Alter set point using ∧ and ∨.
- A new set point will be saved automatically approx. 3 seconds after changing, and the display will go back to indicate the current temperature.

⊘ Do not set the temperature over max. temperature of the each devices (450 °C heater : 450 °C, 500 °C heater : 500 °C, 1100 °C electric furnace : 1100 °C). It is very dangerous to unlock the protection and set a temperature over the max. temperature.

Parameter change

- Keep pressing SEL key for about 1 sec, 3 sec or 5 sec transfers to 1st, 2nd or 3rd block parameter relatively.
- Display desired parameter using ∧ and ∨.
- Pressing SEL displays set value.
- Alter set value using ∧ and ∨.
- Save a new set value by pressing SEL.



2. Parameters setting

Parameter display symbol	Parameter	Description	Setting range and default value	About set value
1st block parameter				
AT	Auto tuning	Used when P,I,D constant is set by auto tuning.	0: Stop 1: Standard AT Start 2: Low PV type AT start	Set to 1 or 2 when auto tuning is performed. It is automatically changed to 0 after auto tuning.
LoC	Key lock	Parameter setting: able to alter/ unable to alter	0,3: not locked (0: unlock) 1,4: not able to alter 2,5: able to alter only SV	Default value: 0
2nd block parameter				
P	Proportional zone	ON/OFF control Set to 0.0 for 2 position control	0.0 ~ 999.9%	Default value: 5
I	Integration time		0 ~ 3200 sec	Default value: 120
d	Differentiation time		0.0 ~ 999.9 sec	Default value: 20
3rd block parameter				
dSP1 ~ dSP9 dP10 ~ dP13	Parameter skip	Display/ not display setting	0 ~ 255	

3. Parameters

Auto tuning (setting range: 0/1/2)

- This is used for determining P, I, D parameters for temperature control automatically. Parameter is determined according to set point.
- If the power is switched OFF during auto tuning, auto tuning become of no use and P, I, D parameters does not change. If you want to restart set to 1 or 2 again.
- If you want to abort auto tuning, set to 0. Auto tuning will stop. P, I, D parameters does not change.
- Once P, I, D are set by auto tuning, it is no lot necessary to perform auto tuning ant more. The parameters are saved on the unit even if the power is OFF.
- If you set 1 or 2 auto tuning starts, and the value becomes 0 automatically after auto tuning is complete.
- The first decimal point of SV indicator flashes on and off during auto tuning.
- There are following types of auto tuning: Set code [1]: SV normal mode (ON/OFF control with SV as a standard), Set code [2]: Low PV mode (ON/OFF control with SV-10 %FS as a standard).
- [Note] since ON/OFF control is in operation during auto tuning overshoot against SV occurs. Please perform low PV mode auto tuning (Set code [2]) if you want to avoid overshoot.
- Auto tuning can be performed right after operation started, during control or during stable state.

4. Heater (450 °C Heater, 550 °C Heater)

1. General

This heater is installed to BELSORP-max, and used to control the sample section temperature. It enables the vacuum pretreatment and the adsorption measurement at high temperature.

2. Specification

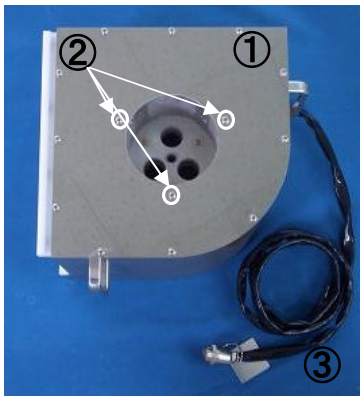
Specification	450 °C (Aluminum block)	550 °C (Brass block)
Number of ports	4 (including P0)	
Error between ports	±0.4 °C	±0.5 °C
Temperature range	50 to 450 °C	50 to 550 °C
Overheat detection temperature	500 °C	600 °C
Temperature stability	±0.2 °C	±0.5 °C
Dimension and weight	W280 x H300 x D270 mm, 6 kg	W280 x H300 x D270 mm, 9 kg
Power voltage	AC100 to120 V or AC200 to240V, 360 VA	AC100 to 120V or AC200 to 240 V, 600 VA

3. Detailed description

① Heater

4 holes for samples and saturation vapor pressure measurement are provided on the heater. When lifting up the heater for installing to BELSORP-max, the sample cell and the saturation vapor pressure cell are inserted in these holes. Align the sample cell with the hole by loosening the "heater alignment screw ②" before lifting up the heater.

⚠ Do not drop any material into the holes.



② Heater alignment screw

This is used to align the sample cell installed to the unit with the holes. Loosen the 3 screws to align in any direction with the holes. Alignment with the holes shall be made according to "Heater alignment" on the next page.

⚠ The sample cell or the saturation vapor pressure cell may be broken when lifting up the heater if misaligned with the heater holes. Be sure to align before use.

③ Heater control cable

Connect it to the temperature controller.

⚠ Prevent the cable from contacting heated parts while heating. If melted, it may cause short circuit.

4. Usage

1. Connect the **heater control cable** ③ to the **10-pin female connector** ⑤on the back of the temperature controller.

2. Slide and install it to the **elevator hanger** ⑩ on the front of the main unit.

⚠ When removing the heater with the sample cell still installed, be sure to move the heater down to the bottom limit.
⚠ When installing to the main unit, slide it to the back limit until it is engaged with the fixing latch.
⚠ Do not use it with other equipment.（Our company shall not be liable for any trouble.）
⚠ The heater and the peripheral equipment are hot while heating with the heater or immediately after the heating. Be careful to protect yourself from being burned.
⚠ The sample cell after heating is hot. Be careful to protect yourself from being burned.
⚠ Do not place any flammable material close to the heater while heating.
⚠ Be sure to install the “heat protection cover” on the heater, which is supplied with the temperature controller during heating and awhile after heating.

5. Heater alignment

Align the BELSORP-max sample cell with the heater holes as follows.

⚠ If misaligned, the sample cell may be broken when lifting up the heater. Be sure to align before use. Turn off the heater, and verify that it has been cooled down to room temperature before starting the alignment work.

1. Install a specially designed sample cell to BELSORP-max.
2. Loosen the **heater alignment screw** ②**.**
3. Lift up the heater step-by-step using the lifting button on the BELSORP-max main unit. Align the heater so that the sample cell and the P0 cell are inserted to the center of the holes. Once aligned, tighten the heater adjustment screw to fix the heater. (For use of the lifting button, refer to “Nomenclature and function of individual components, P. 21” in this manual.)
4. Lift down the heater, and tighten the **heater alignment screw** ② to fix the heater position.
5. Lift up the heater step-by-step, and verify again that the sample cell and the P0 cell are inserted to the holes. The alignment is now complete.

5. 1100°C electric furnace

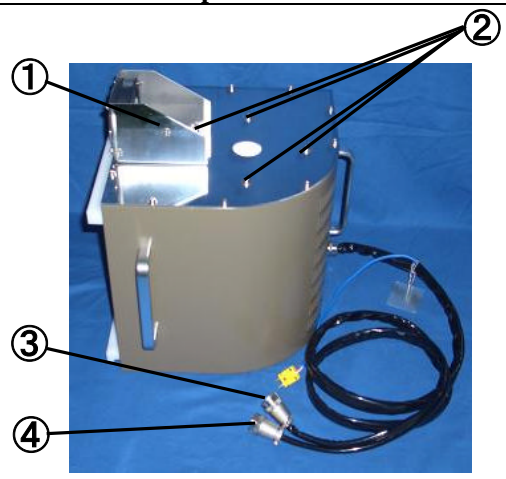
1. General

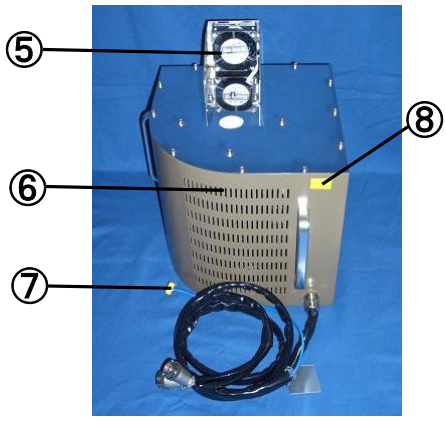
This heater is installed to BELSORP-max, and used to control the sample section temperature. This enables the vacuum pretreatment / flow gas pretreatment and the adsorption measurement at a high temperature up to 1100°C.

2. Specification

Number of ports	1
Temperature range	50 to 1100°C
Temperature stability	±0.5°C
Overheat detection temperature	1150°C
Dimension and weight	W280 x H300 x D270 mm, 6 kg
Power voltage	AC100V to 120V or AC200 to 240V, 330VA

3. Detailed description





① Electric furnace

The electric furnace has a hole to accommodate the sample cell. When lifting it up for installing to BELSORP-max, the sample cell installed to Port 3 is inserted to the hole. Align the sample cell with the hole by loosening the “heater alignment screw” before lifting it up.

⚠ When using the electric furnace, do not install the sample cell to Port 1 and 2 or a saturation vapor pressure cell.

② Electric furnace alignment screw

This is used to align the sample cell installed to the unit with the holes. Loosen the 4 screws to align in any direction with the holes.

⚠ The sample cell may be broken when lifting up the electric furnace if misaligned with the holes. Be sure to align before use.

③ Electric furnace control cable, ④ Rapid cooling fan control cable

Connect to the **10-pin, female connector** ⑦ and **4-pin, female connector** ⑥ (for rapid cooling fan) on the back of the temperature controller, respectively (P. 31).

⚠ Prevent the cable from contacting hot parts while heating. If melted, it may cause short circuit.

⑤ Upper fan

This fan is installed to the electric furnace for releasing heat from the heating port while heating. Connect the **electric furnace control cable** ③, and turn on the temperature controller. This starts automatic operation.

⑥ Rapid cooling fan

A fan is equipped inside. Connect the **electric furnace control cable** ③ and the **rapid cooling fan control cable** ④. When the electric furnace temperature exceeds 1150 °C, the system turns off the electric furnace and starts the rapid cooling fan. When setting FAN as ON on the software "Vacuum/flow gas sequential pretreatment" screen, this rapid cooling fan starts (For the above mentioned pretreatment, refer to P. 122).

⑦ Control thermometer connector (male)

For internal temperature control (temperature control using a thermometer in the electric furnace), connect this to the **internal temperature control thermometer connector (female)** ⑧ on the electric furnace. For external temperature control (temperature control using a thermometer that is inserted to the accessory flow gas sample cell thermometer sheath), connect this to the accessory external temperature control thermometer connector (female).

It is recommended to use the external temperature control for more accurate temperature control of the sample section. When using the internal temperature control, calibrate the internal temperature by measuring the sample section temperature using another thermometer.

⑧ Internal temperature control thermometer connector (female)

Connect the **control thermometer connector (male)** ⑦ when using the internal temperature control. Do not connect anything to this connector when using the external temperature control.

4. Usage

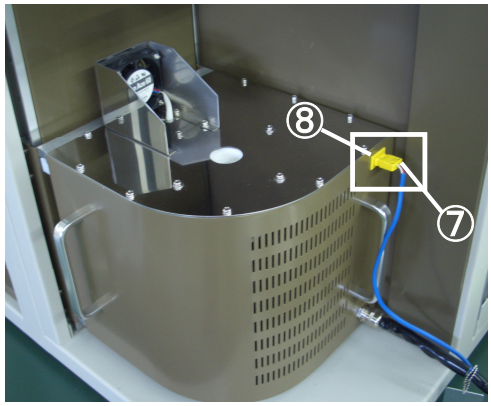
1. Connect the electric furnace control cable ③ and the rapid cooling fan control cable ④ to the 10-pin, female connector ⑦ and 4-pin female connector ⑥ on the back of the temperature controller (P. 31), respectively.
2. While sliding, install it to the **elevator hanger** ⑩ on the front of the main unit (P. 21).

⚠ When removing the electric furnace with the sample cell still installed, be sure to move the electric furnace down to the bottom limit.
⚠ When installing to the main unit, slide it to the back limit until it is engaged with the fixing latch.
⚠ Do not use it with other equipment (Our company shall not be liable for any trouble.).
⚠ The electric furnace and the peripheral equipment are hot while heating with the electric furnace or immediately after the heating. Be careful to protect yourself from being burned.
⚠ The sample cell after heating is hot. Be careful to protect yourself from being burned.
⚠ Do not place any flammable material close to the electric while heating.
⚠ Be sure to install the "heat protection cover" on the electric furnace while heating, which is supplied with the temperature controller.
⚠ Be sure to use quartz sample cell when heating up to high temperature with the electric furnace.

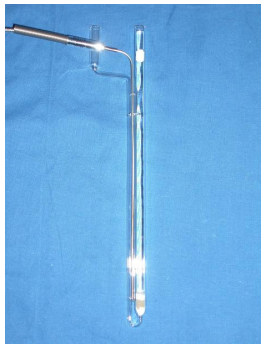
3. According to the control method used, connect the relevant connector as follows.

For internal temperature control

Connect the **control thermometer connector (male)** ⑦ to the **internal temperature control thermometer connector (female)** ⑧ as shown on the right.



For external temperature control

		
Connect the external temperature control thermometer connector (female), an accessory of the 1100 °C electric furnace, to the **control thermometer connector (male)** ⑦.	Insert the thermometer to the flow gas sample cell thermometer sheath (Bend the thermometer at an angle of 90 degrees as appropriate.).	Install the flow gas sample cell to the main unit. For installation, refer to "How to install the flow gas sample cell (optional), P.59".

5. Electric furnace alignment

Align the BELSORP-max sample cell or the flow gas sample cell with the electric furnace as follows.

⚠ If misaligned, the sample cell may be broken when lifting up the heater. Be sure to align before use. Turn off the heater power, and verify that it has been cooled down to room temperature before starting the alignment work.

1. Install the sample cell or a flow gas sample cell to the BELSORP-max, Port 3.
2. Loosen the electric furnace alignment screw on the top of the electric furnace.
3. Lift up the electric furnace step-by-step using the lifting button on the BELSORP-max main unit. Align the electric furnace so that the sample cell is inserted to the center of the holes. Once aligned, tighten the electric

furnace adjustment screw to fix the electric furnace (For use of the lifting button, refer to “Nomenclature and function of individual components, P.21” in this manual.).

4. Lift down the electric furnace, and tighten the electric furnace alignment screw to fix the electric furnace position.
5. Lift up the electric furnace step-by-step, and verify again that the sample cell or the flow gas sample cell is inserted to the holes. The alignment is now complete.

ooo Manual-switching gas selector (Option) ooo

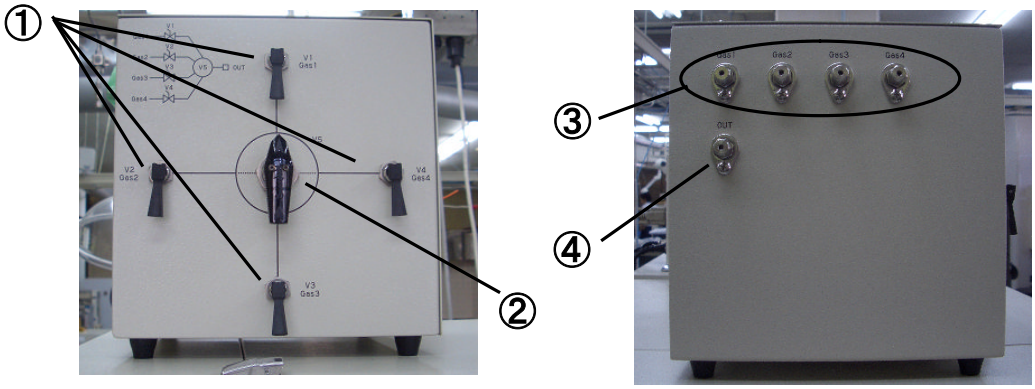
1. General

This instrument is an accessory to switch the gas introduced into BELSORP-max by manual operations of the valves.

2. Specification

Available gas	N_2, Ar, Kr, H_2, CO, O_2, CH_4, other non-corrosive gas
Material of joint	SUS, brass
Gas port	4 at inlet, 1 at outlet (joint: 1/8” SWG)
Pressure	0.1 MPa (G)
Dimension	W240×H240×D240 mm (except the projection of the valve)

3. Detailed description



①Switching valves (V1 to V4)

Gas supply to the tubes connected with Gas 1 to Gas 4 is started/stopped by these manual-switching valves.

②Gas switching valve (V5)

Gas supplied to BELSORP-max is selected by this rotary-type manual-switching valve.

③Gas inlets (Gas 1 to Gas 4)

Gas is supplied from here to the main unit.

④Gas outlet (OUT)

The gas supplied to the main unit is exhaust from this outlet.

4. Usage

1. How to connect the tube

①Close the switching valves on the front panel (V1 to V4). The valves are closed by bringing down the cocks.

②Connect the gas outlet (OUT) on the side panel with Aux. 1 to Aux. 4 and Ads. of BELSORP-max.

③Connect the gas to be used with the gas inlets (Gas 1 to Gas 4) on the side panel.

⚠ Adjust the secondary pressure of the gas cylinder to 0.1±0.01 MPa (1kg cm^{-2}).

2. Washing the gas line

①Open a switching valve (one of V1 to V4), and turn the gas switching valve (V5) to the line to be washed.

②Select a line, and wash the gas line as referring to “Installing and replacing gas cylinders, P. 83”.

3. How to switch gas

①Close the switching valve which has been used (one of V1 to V4).

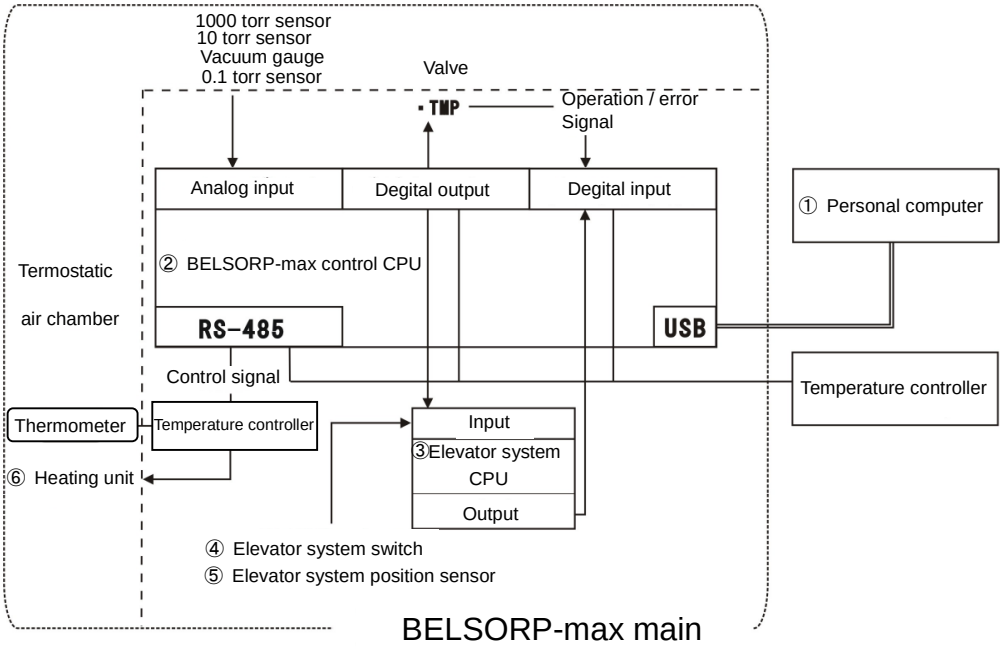
②As referring to “Installing and replacing gas cylinders, P. 83”, wash the gas line up to the switching valve (one of V1 to V4).

③Turn the gas switching valve (V5) to the line to be used.

④Open the switching valve of that line (one of V1 to V4), and wash the gas line as referring to “Installing and replacing gas cylinders, P. 83”.

⚠ To avoid gas from blending, be sure to wash the gas line before switching gas.

ooo System configuration ooo



① Personal computer

It use specialized measurement software for thefolowing controls. It communicates with the main unit via USB.

· Operating and closeing valve
· Reading pressure in the system
· Operating the vacuume gauge
· Operating the turbo molecular pomp
· Operating the elevator
· Reading temperature of the thermostatic air snd temperature device
· Operating the temperature controller

② BELSORP-max control CPU

This is a computer built in main unit. It recives signals from the personal computer to control various functions.

③ Elevetor system CPU

This is a computer bilut in main unit. It recives from the lifting switch on the front of main unit and the personal computer to control the elevator.

④ Elevator system switch

It consists of the [▲] (lift-up) switch and [▼] (lift-down) switch. For details, refer to “ooo Main unit-front ooo, P. 21”.

⑤ Elevator system position sensor

This detects that the elevator has moved down to the bottom limit.

⑥ Heating unit

This unit located in the thermostatic manifold temperature. It integrates a fan and heater, and controls automatically the thermostatic aie chamber temperature to be 40 ^{o}C (Option 50 ^{o}C), when the main unit switch is turned on.

Caution

Do not use a pump with a maximum operating current of more than 7A.

Do not turn off power to the pump with the main unit switch turned on. Otherwise, oil may flow back to the instrument, which may contaminate inside the unit.

The Dewar vessel or the temperature unit moves up and down during the sample measurement. Do not place your fingers or any materials on the Dewar vessel or the temperature unit. It may cause bodily injury and/or instrument failure.

Do not press the lifting switch during the sample measurement using refrigerant. When lifting the Dewar vessel out of necessity during the sample measurement, careful attention is required as follows.

When lifting down the Dewar vessel, it returns from the refrigerant temperature to room temperature. At this moment, the nitrogen or argon that is adsorbed or condensed to the sample section or at the saturation vapor pressure P0 port may evaporate, and generate higher pressure than atmospheric pressure. This may cause an error of the measurement data, but also result in damage to the instrument since it raises pressure in the glass tube, and detaches it from the instrument connection port over time. This measurement software stops forcedly the sample measurement and degasses the system automatically, when the measurement port pressure exceeds 130 kPa.

Prior to measurement, install the heat insulation cap after removing the ice or water adhering to it.

BELSORP-max installation

ooo BELSORP-max package items and other requisites ooo

1. BELSORP-max package items

Standard BELSORP-max includes the main unit and accessories as follows. Please verify that the complete accessories are provided. If you find any items missing or damaged, please contact our company.

Main unit or accessories		Quantity
-	BELSORP-max main unit	1
①	Ads. 2 conversion connector (Connect to the "adsorptive gas dosing port (Ads. 2) " on the front of the unit).	1
②	Power cable	1
③	Communication cable (USB cable)	1
④	Rotary pump*	1
⑤	NW16 flexible tube*	1
⑥	NW16 clamp*	2
⑦	NW16 centering*	2
⑧	Dewar vessel	1
⑨	Dewar vessel heat insulation lid	1
⑩	Sample cell heat insulation sleeve (to be used when using the Dewar vessel)	6
⑪	P0 cell heat insulation sleeve (to be used when using the Dewar vessel)	1
⑫	P0 cell o-ring Securing screw	1 2 1
⑬	P0 port cap	1
⑭	Sample cell weighing stand (to be used as the sample cell holder on a balance pan when measuring the sample weight)	
⑮	Sample cell stand	1
⑯	Reference sample (0.2 g) (non-porous carbon black A_{BET} = 49.17 ± 1.5 $m^2 g^{-1}$, P/ P_0 : 0.04-0.15)	1

*: Different depending on the pump specification


①Ads.2 conversion connector


②Power cable


③Communication cable


④Rotary pump

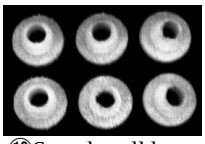

⑤NW16 flexible tube


⑥NW16 clamp


⑦NW16 centering

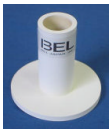

⑧Dewar vessel


⑨Dewar vessel heat insulation lid


⑩Sample cell heat insulation sleeve


⑬P0 port cap


⑫P0 cell (right)
o-ring (upper left)
Securing screw (lower left)

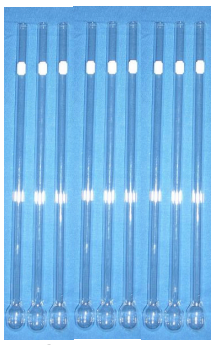

⑭Sample cell weighting stand


⑮Sample cell stand

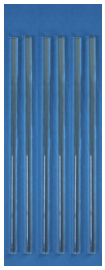

⑯Sample cell weighting stand

	Accessories	Quantity
⑰	Port plug	4
⑱	Sample cell (Pyrex) (Op. silica)	9
⑲	Glass rod (Pyrex) (Op. silica)	6
⑳	Sampling funnel	3
㉑	Liq. bottle (Pyrex)	1
㉒	Dewar vessel	1
㉓	FKM O-ring (Op. FFKM)	6
㉔	Sample scattering prevention filter	6
㉕	Sample cell cap	10
㉖	Instruction manual (this manual)	1
㉗	Analysis software manual	1
㉘	Instruction manual-Supplement (Measurement example)	1
㉙	Setup CD	1
㉚	Config CD (Instrument specific data is stored, including the reference volume. Keep this carefully.)	1
㉛	WIBU-key (for analysis software to recognize effective software)	1

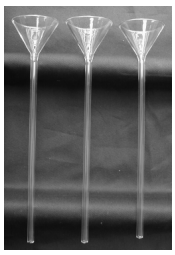
⑰Port plug



⑱Sample cell



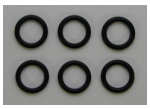
⑲Glass rod



⑳Sampling funnel

㉑Liq. bottle

㉒Dewar vessel



㉓P9 o-ring

㉔Sample scattering prevention filter (P-9)

㉕Sample cell cap (ϕ9)

㉖Instruction manual

㉗Analysis software manual

㉘Instruction manual-Supplement

㉙Setup CD

㉚Config CD

㉛WIBU-key

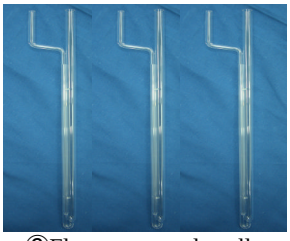
2. Optional equipment

The following options are available separately from the standard package.

Accessories	
①	Sample cell (silica)
②	Glass rod (silica)
③	Flow gas sample cell (Pyrex or silica)
④	Silica glass wool
⑤	Small volume sample cell*1
⑥	Glass rod for small volume sample cell*2
⑦	Large volume sample cell*1
⑧	Glass rod for large volume sample cell*2
⑨	Pellet sample cell*1
⑩	Glass rod for pellet sample cell*2
⑪	Quick seal*1
⑫	Glass rod for quick seal*2
⑬	NSD capusle*2
⑭	NSD capusel sample cell

*1 Standard glass rods cannot be used.

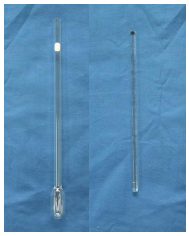
*2 Standard sample cell cannot be used.



③Flow gas sample cell

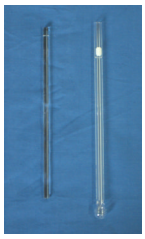
④Silica glass wool

⑤Small volume sample cell
⑥Glass rod for small volume sample cell



⑦Large volume sample cell
⑧Glass rod for large volume sample cell

⑨Pellet sample cell
⑩Glass rod for pellet sample cell



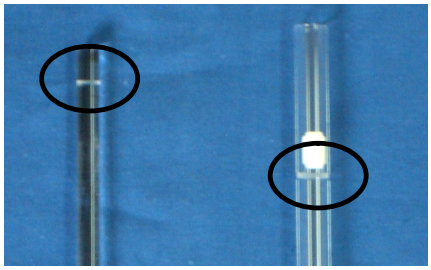
①Sample cell (silica)
②Glass rod (silica)

⑫Glass rod for quick seal

⑪Quick seal

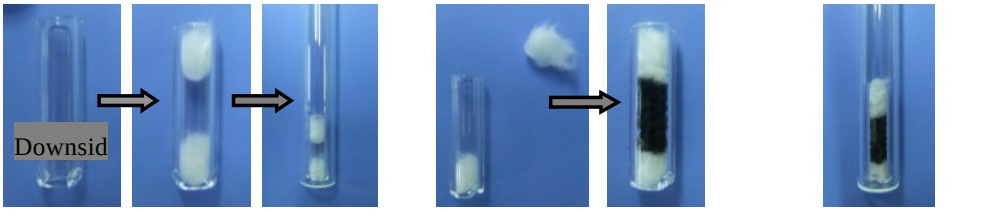
⑬NSD capusel
⑭NSD capusel sample cell

Silica glass devices are marked with a white line as shown below for identification.



■Usage of NSD capsule

1. Blank weight measurement → 2. Sample packing → 3. Pretreatment start



NSD capsule

A dedicated sample cell and a glass rod (Pay attention to direction.)

Take upper glass wool out.

Sample packing

Set it in the sample cell.

Measure the blank weight with silica wool inside. To pack the sample, stand the NSD capsule with a dent side down, place the sample on the silica wool in the NSD capsule as shown above, and insert upper silica wool that was taken out. Place the NSD capsule in which the sample is packed in the sample cell for NSD capsule, put a glass rod.

Water bath

Accessories	Quantity
Water bath	1
Detachable thermostatic bath	1

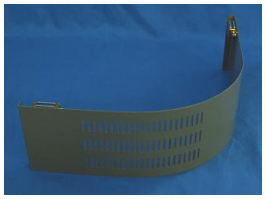
Temperature controller

Accessories	Quantity
Temperature controller	1
Power cable	1
Communication cable (D-sub 9 pin straight cable)	1
Heater protection cover (to be installed on the top of the heater when using the heater)	1

.

Temperature controller

Power cable

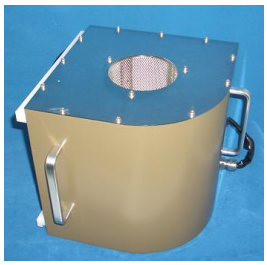


Heater protection cover

Communication cable

450 °C heater

Accessories	Quantity
450 °C heater	1



450 °C heater, 550 °C heater

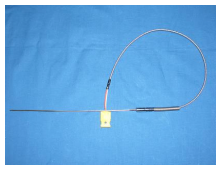
550 °C heater

Accessories	Quantity
550 °C heater	1

1100 °C electric furnace

Accessories	Quantity
1100 °C electric furnace	1
External temperature control thermometer	1

1100 °C electric furnace



External temperature control thermometer

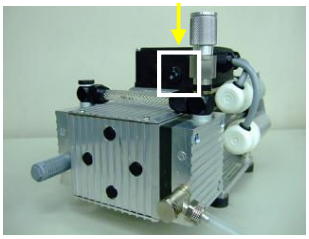
Diaphragm pump

Accessories	Quantity
Diaphragm pump	1

Diaphragm pump

The diaphragm pump is equipped with a voltage switch.

Switch voltage in accordance with the place of use before using the pump.



Corrosion resistant diaphragm pump

Accessories	Quantity
Corrosion resistant diaphragm pump	1
Flexible tube	1
Flexible tube connection clamp	2
Flexible tube connection centering	2

Corrosion resistant diaphragm pump

Flexible tube

Clamp

Centering

Corrosion resistant rotary pump

Accessories	Quantity
Corrosion resistant rotary pump	1
Flexible tube	1
Flexible tube connection clamp	2
Flexible tube connection centering	2

Corrosion resistant rotary pump

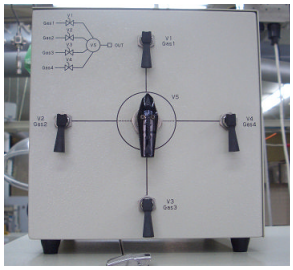
Flexible tube

Clamp

Centering

Corrosion resistant gas selector

Accesories	Quantity
Gas selector unit	1
Earthquake resistant stopper	1
Magnetic sheet	4



Corrosion resistant gas selector

3. Utility and other requisites

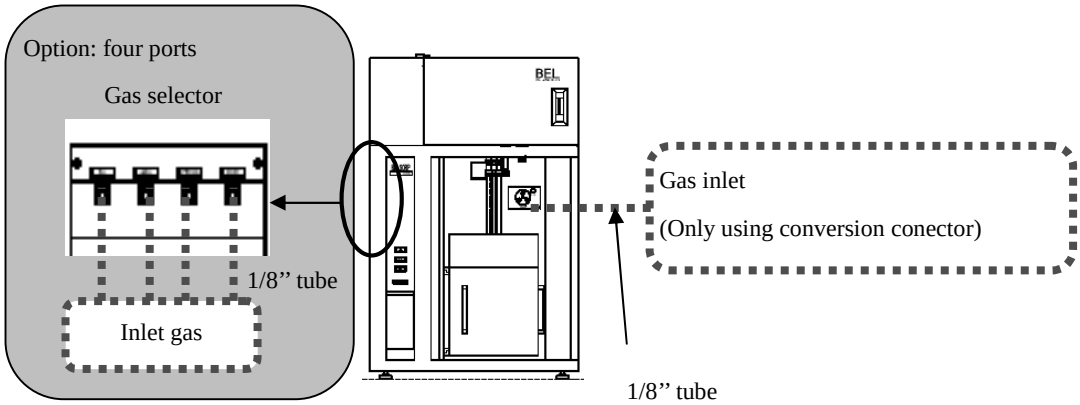
BELSORP-max requires the following utilities. Provide them appropriately according to the following "Requirements". Personal computers are also available from our company.

Requisites		Requirements
Personal computer		Refer to "System environment required, P. 75".
Dosing gas	To be provided by the customer	Purity : Use the products of purity 99.999 % or more Pressure : 0.1 MPa (gauge pressure) Connection : 1/8'' Swage lock
Helium gas		Purity : Use the products of 99.999 % or more Pressure : 0.1 MPa (gauge pressure) Connection : 1/8'' Swage lock
Air and gas to drive pneumatic valves		Pressure : 0.5 to 0.6 MPa (gauge pressure) Connection : 1/4'' plastics tube quick connect
Vent line		Connection : 1/8'' Swage lock

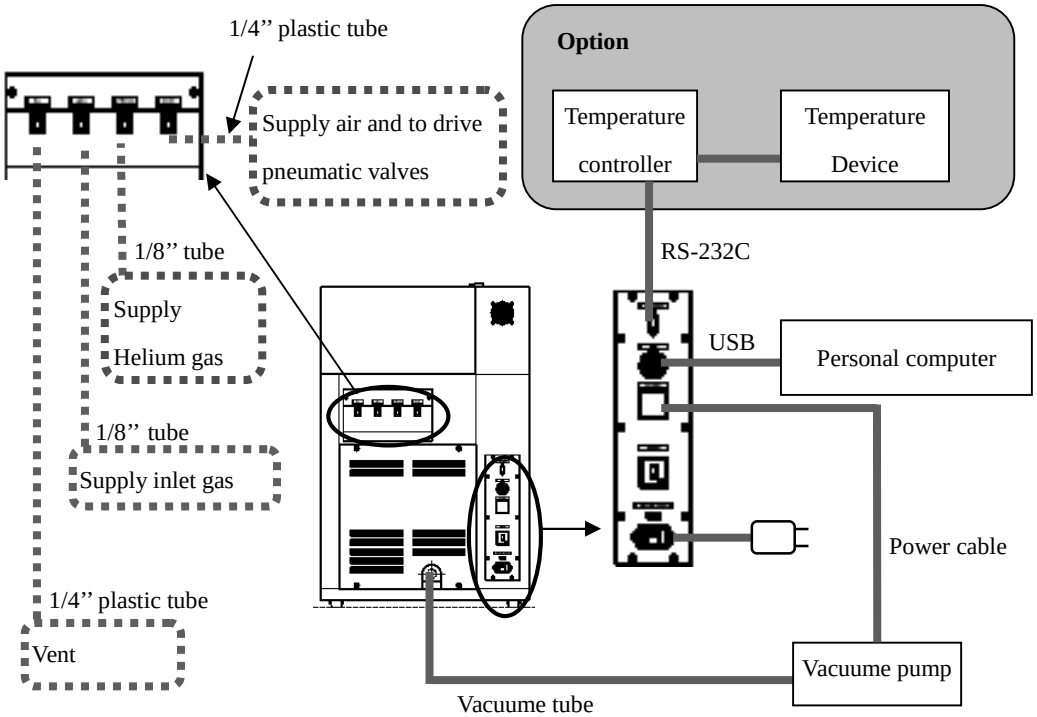
: Part packaged as an accessory of BELSORP-max or its options

: Part needing to be prepared separately

(Such parts are not included in the accessories of BELSORP-max and its options. You need to prepare them or request us to prepare them.)



Main unit-front



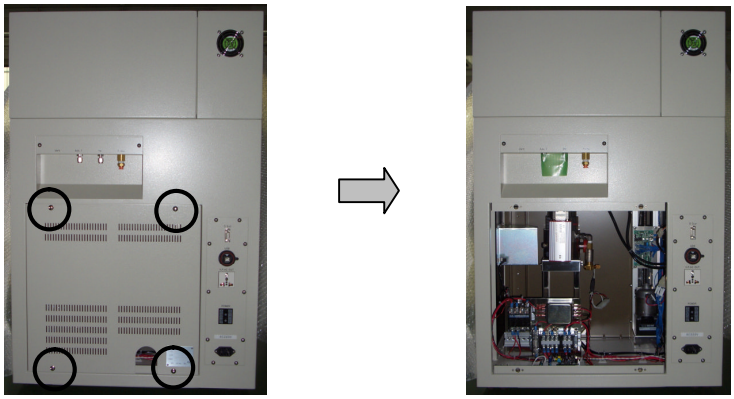
Main unit-back

ooo Installation procedure ooo

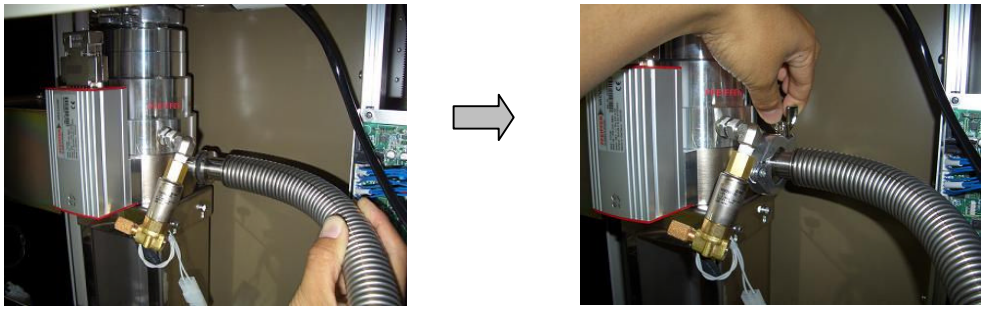
1. Connection with auxiliary equipment

1. Place the **BELSORP-max** main unit on a horizontal place, and verify that it seats stably.

2. Remove the access door on the back of the instrument. Turn the slot on the latch lock (at 4 locations) to the vertical direction using a flat head driver. The lock is then released. Pull out the door to remove.



3. Attach the flexible tube to the turbo molecular pump located deep on the back of the unit. Insert the accessory "flexible tube connection centering (NW16)" between the "flexible tube" and the turbo molecular pump connection port, and fix it using the "flexible tube connection clamp (NW16)". Lastly, firmly tighten the screw on the clamp.



4. Replace the access door in place, which was removed in **2.** In this step, lead the "flexible tube" out of the instrument through the hole on the access door.

5. Connect the other end of flexible tube to the rotary pump suction port.

⚠ Oil mist is discharged from the pump exhaust port. Process it appropriately.

⃠ Do not connect any pump with voltage except those specified on the nameplate on the back of the instrument.

6. Connect the rotary pump power plug to the AC outlet on the back of the main unit.

7. Install an exhaust line to the vent port.

The main unit connection port uses a 1/4” plastics tube quick connect only when the optional flow gas pretreatment line is selected.

> ❗ It is dangerous to perform flow gas pretreatment without an exhaust line attached to the vent port, since used gas is discharged. When performing the flow gas pretreatment, be sure to attach an exhaust line to the vent port.

8. Connect a gas line of the adsorptive gas (0.1 Mpa (gauge pressure)). The main unit connection port uses a 1/8’’ Swage lock.

9. Connect a gas line of helium gas (0.1 MPa (gauge pressure)). The main unit connection port uses a 1/8’’ Swage lock.

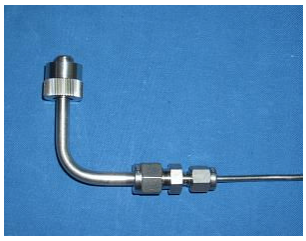
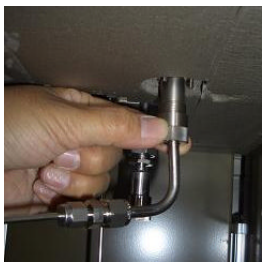
10. Supply air and gas (to drive pneumatic valves, 0.5 to 0.6 MPa). The main unit connection port uses a 1/4” plastics tube quickly connect.

11. Use a USB cable to connect the USB connector on the personal computer to the USB connector (type-B, female) on the main unit.

12. Connect the power source specified on the nameplate on the back of the main unit.

2. How to connect the gas line to the “adsorptive gas dosing port (Ads.2)”

1. When supplying gas from the Ads.2 port on the front of the main unit, connect the relevant gas line as follows.

		
Connect the gas line of adsorptive gas (0.1 MPa gauge pressure) to the “Ads. 2 conversio connector” provided.	Attach an o-ring to the “Ads. 2 conversion connector”, and insert it to the “adsorptive gas dosing port (ads. 2)” until it contacts a metal part on the back.	Tighten a nut on the “Ads. 2 conversion connector” by hand. Connection is now complete.

3. How to connect the gas line to the “gas selector (optional)”

Connect a gas line of the dosing gas (0.1 MPa (gauge pressure)). All ports use the 1/8’’ Swage lock.

4. How to install the sample cells and liq. bottles

- Use the same type of sample cells, dead volume reference cells, and glass rods.
- For sample measurement, insert a glass rod both to the sample cell and the dead volume reference cell. Slide down the glass rod slowly along the glass wall so that the glass tube is not broken.
- Before using sample cells, glass rods, and a liq. bottle ; wash and dry them sufficiently.
- Store them in a desiccator after washing.

⚠ The sample cells and the liq. bottles are made of glass. Careful attention is required for the handling.

Sample cell

Glass rod

P0 cell (right)
o-ring (upper left)
Securing screw
(lower left)

1. Insert a glass rod to both the sample cell and the dead volume reference cell (when measuring the dead volume).

2. Install the sample cell after removing a fitting and o-ring from it. When the sample cell is installed with the fitting still attached, it may deform the o-ring, and accordingly it may result in vacuum leakage. When removing the sample cell from the instrument, Viton o-ring may remain inside the connection port of the instrument. Verity that there is Viton o-ring remaining inside the connection port.

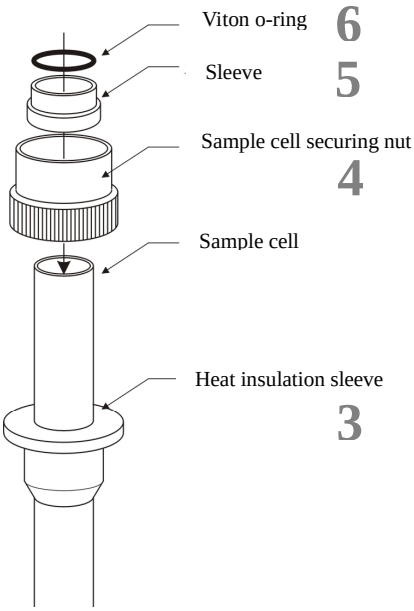
⚠ Do not drop any fitting into the Dewar vessel. It may be dangerous because the Dewar vessel glass will break.

3. Attach the heat insulation sleeve to the sample cell as shown on the right.

4. Attach the sample cell securing-nut. Do not use upside down.

⚠ Do not use a heat insulation sleeve when using a heater for pretreatment or measurement. Otherwise, it may cause a fire.

5. Attach the sleeve. Do not use upside down.

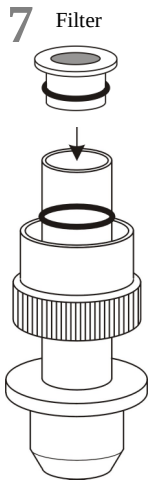


6. Attach Viton o-ring. The measurement section is sealed with this o-ring pressed on the sample cell and the measurement ports. When the o-ring is damaged, or any foreign material is pressed with the o-ring, air may flow in the measurement section. This may affect data accuracy. Be sure that no foreign material is contained. Replace the damaged o-ring, if any, with a new one.

7. Insert the filter from the top of the sample cell.

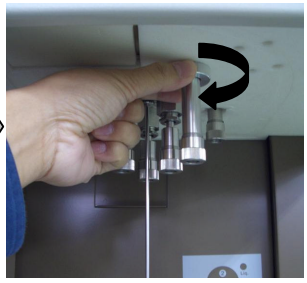
8. Hold the sample cell firmly, and insert it to the sample port until it contacts the metal wall in the fitting. Tighten the sample cell securing-nut by hand.

9. Slide the heat insulation tube halfway down the sample cell. The liq. bottle is installed in the same way as the sample cell, but no heat insulation sleeve (3.), filter (7.) are required in this case.



5. How to install the flow gas sample cell (optional)

1. There are 2 connection ports for installing the flow gas sample cell. Prior to installing the sample cell, align the connection port with the sample cell as follows.

		
Loosen a nut on the flow gas pretreatment line (optional) on the front of the main unit, so that the flow gas pretreatment line can be moved.	Place a flow gas sample cell from the bottom, and align the flow gas pretreatment line with it.	After aligning the flow gas pretreatment line, tighten the nut to fix the flow gas pretreatment line position. Alignment is now complete.

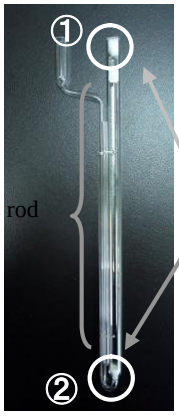
⚠ The flow gas sample cell is made of glass. Careful attention is required for the handling.

2. Attach the sample cell securing-nut, sleeve, and o-ring to the flow gas sample cell according to " How to install the sample cells and liq. bottles on P. 57".

6. Sampling procedure when using a flow gas sample cell

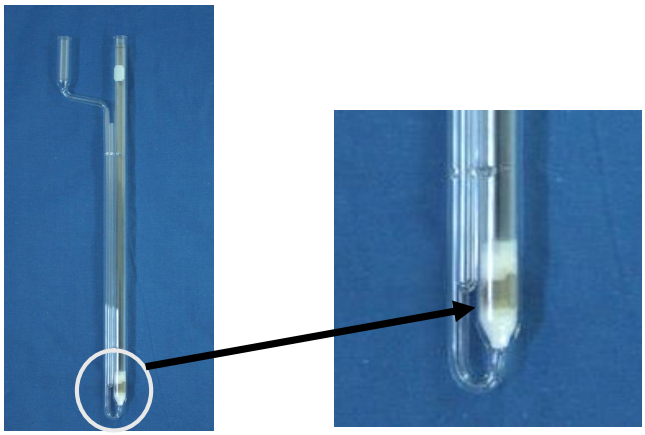
When using a flow gas sample cell, use the silica glass wool, an accessory of the flow gas pretreatment line (optional) to perform the following sampling procedure.

1. Prepare two appropriate sized balls of silica glass wool (standard: 0.01 to 0.02 g).
2. Insert them and a glass rod in the flow gas sample cell as shown in the photo on the right (Measure the blank weight at this time).



3. Remove the silica glass wool ① and a glass rod, and feed a sample on the silica glass wool ② using a funnel.

4. Replace the silica glass wool and a glass rod ① (Measure the sample weight at this time).

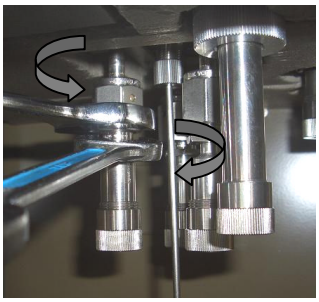
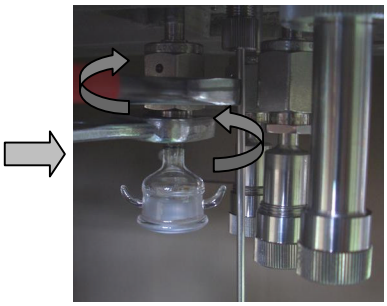
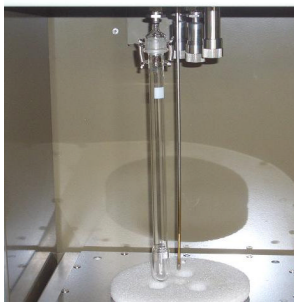


⚠ When using silica glass wool, careful attention is required for the handling as follows.

- When handling the glass wool, take appropriate measures for local exhausting because dust is easily scattered.
- Use a dust mask.
- Do not handle it with bare hands.
- When the wool adheres to your clothing, remove it completely.
- After handling, gargle and wash your hands sufficiently.

7. How to install the pellet sample cell (optional)

Install the pellet sample cell as follows.

		
Remove the port to be installed using two wrenchs, and remove the 4VCR gasket.	Place the 4VCR gasket on the 4VCR at the upper part of the pellet sample cell, and install it to the instrument.	Insert the glass rod for the pellet sample cell into the sample section of the pellet sample cell, install it to the instrument, and secure it with a spring. Installation is now complete.

8. How to install/remove the Dewar vessel, temperature measurement device

⚠ The Dewar vessel is made of glass. Careful attention is required for the handling.

⚠ When removing the Dewar vessel temperature device with the sample cell still installed, be sure to move down the Dewar vessel temperature device to the bottom limit.

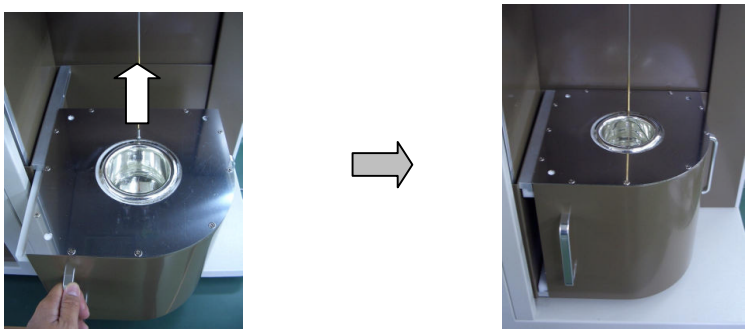
⚠ When installing to the main unit, slide it to the back limit until it is engaged with the fixing latch.

⚠ The heater and the peripheral equipment are hot while heating with the heater or immediately after the heating. Be careful to protect yourself from being burned.

⚠ The sample cell after heating is hot. Be careful to protect yourself from being burned.

⚠ Be sure to install the "heat protection cover" on the heater or the electric furnace while heating, which is supplied with the temperature controller.

1. Insert the slide rail on the side of the Dewar vessel temperature device to the "elevator hanger" on the front of the main unit, and slide it to the back.



2. Slide it to the back limit, until it is engaged with the fixing latch.
3. When removing the Dewar vessel temperature device from the main unit, move the Dewar vessel temperature device down to the bottom limit, and pull the handle.

9. Conversion of the flow meter value by gas type

The flow meter value indicated at the upper right of the instrument front panel varies depending on the gas type; therefore, convert it appropriately using the following equation.

(Example) When applying a H_2 (hydrogen) flow meter to He (helium), the actual flow rate should be smaller than the indication, as follows.

$$QHe = QH_2\sqrt{\frac{\rho H_2}{\rho He}} = QH_2\sqrt{\frac{2}{4}} \fallingdotseq 0.7QH_2$$

Q: Flow rate [$cm^3\ sec^{-1}$]

ρ: Density of liquid [$g\ cm^{-3}$]

10. Safety devices

BELSORP-max is equipped with the following safety devices. When any safety device is activated, it takes actions automatically as follows. Reset it as listed below after you remove the cause.

Safety device	Action	How to reset
Manifold temperature overheat detection	When the temperature in the manifold temperature on the top of the instrument exceeds 90 °C, it detects faults and turns off the instrument power.	When the temperature in the manifold temperature drops below 90°C, turn off the power switch on the back of the instrument, and then turn it on again to reset.
Temperature device overheat detection	When the temperature device (450 °C heater, 550 °C heater, 1100 °C electric furnace) exceeds the specified temperature (for details refer to the temperature controller on P. 32), the temperature controller detects faults and stops heating (in such a case, the "overheat indicator" on the front of the temperature controller lights up). When the measurement software is running, it exits automatically.	After cooling the temperature device, turn off the power switch on the front of the temperature controller, and then turn it on again to reset. Verify that the "overheat indicator" on the front of the temperature controller lights off.
Elevator overload detection	When the elevator load exceeds 50kg, it detects faults, and disables lifting operation (in such a case, the lifting switch ■ on the front of the instrument blinks red). When the measurement software is running, it exits automatically.	A reset switch for the elevator is located in the "temperature controller pocket" on the front of the instrument. After you remove the cause, press the reset switch to release any protective actions.
System faulty pressure detection (Only when the measurement software is started)	When any pressure gauge exceeds 130 kPa, it detects faults and triggers an emergency stop (exhaust process). Only in the adsorption/desorption measurement, it detects faults when P0 exceeds 110 kPa.	Leave it without any action until the software exhausting process is complete.
Turbo molecular pump overload detection (Only when the measurement software is started)	When air is sucked while the turbo molecular pump is in operation, it detects load and stops the TMP operation (in such a case, "TMP error" appears on the software screen).	Even when the TMP error is indicated, the measurement is not aborted. Stop the low-pressure measurement because vacuum is degraded. To stop measuring and reset the error, turn off the instrument power switch 10 minutes or more after the error detection, and turn it on again another 5 seconds later to reset.
Recovering from system error	In case measurement is stopped by some reason, urgent processing will begin at restart of the software.	When measurement is stopped, finish the software and restart it. Leave it as it is, since urgent processing will start automatically.

⚠ Do not shut off power to the main unit while the turbo molecule pump is in operation, or for 30 minutes after it stops. When power is removed during operation, or immediately after shutdown, the system pressure rises rapidly up to atmospheric pressure. It may apply overload to the turbo molecule pump, and accordingly it may cause failure.

ooo Startup and shutdown ooo

1. Startup of the BELSORP-max main unit

1. Turn on the power switch on the back of the main unit. The power indicator at the front of the main unit lights up.
2. Connect the rotary pump power cable to the AC outlet on the back of the instrument, and turn on the rotary pump.
3. When the instrument has not been used for a long time period, purge the instrument tubing according to “ooo Installing and replacing gas cylinders ooo, P. 90”.

⚠ Be sure to connect the rotary pump power cable to the “AC outlet”.

⚠ The Dewar vessel is made of glass. Careful attention is required for the handling.

⚠ It takes 5 hours for the pressure gauge used in the main unit to stabilize the pressure reading. Start measurement about 5 hours after power is supplied to the main unit.

2. Shutdown of the BELSORP-max main unit

In case using the instrument for the measurement, following turn-off procedure should be done more than 30 minutes after the measurement.

1. Turn off the power switch on the back of the main unit. The power indicator at the front of the main unit lights off.
2. Turn off the rotary pump immediately.
3. When the instrument will not be used for a month or more, close the gas main valve.

⚠ Caution

In case you turn off the pump first, turn off the instrument immediately.

- **When the instrument is still on after the rotary pump is turned off, oil may flow back into the instrument. This may contaminate the instrument with oil.**

Do not shut off power to the main unit while the turbo molecular pump is in operation, or for 30 minutes after it stops. When power is removed during operation, or immediately after the shutdown, the system pressure rises rapidly up to atmospheric pressure. It may apply overload to the turbo molecular pump, and accordingly it may cause failure.

- **Our company shall not be liable for any damage of the instrument caused by wrong operational procedures.**

Options

To meet various measurement requirements from customers, the following options are available with **BELSORP-max**. The software operations are common to all types of specifications; therefore, refer to ooo Basic software operation ooo on P. 83, and “Guidance support” on P. 90.

Model	Port No.	Pressure sensor in the sample section				Exhaust system
		1000 Torr	10 Torr	1 Torr	0.1 Torr	
Model 1 (0.1 Torr sensor)	1	○	△	−	△	TMP+RP (OP. DP) ＋WRG
	2	○	−	−	−	
	3	○	○	−	○	
Model 2 (1 Torr sensor)	1	○	−	△	−	TMP＋RP (OP. DP) ＋WRG
	2	○	−	−	−	
	3	○	−	○	−	
Model 3 (10 Torr sensor + TMP)	1	○	△	−	−	TMP＋RP (OP. DP) ＋WRG
	2	○	−	−	−	
	3	○	○	−	−	
Model 4 (10 Torr sensor)	1	○	△	−	−	RP＋ Pirani sensor
	2	○	−	−	−	
	3	○	○	−	−	

△ : selctable marker

※ For temperature device options, refer to P. 8.

※ For other optional equipment and oil-free exhaust system, refer to P. 9.

For details of the optional models, refer to the following pages.

ooo 1 Torr sensor specification ooo

This model is equipped with a 1 Torr sensor and a turbo molecular pump, and measures the adsorption isotherm in the range of $P/P_0 = 10^{-7}$ to 0.997. For other features, refer to "About BELSORP-max on P. 11".

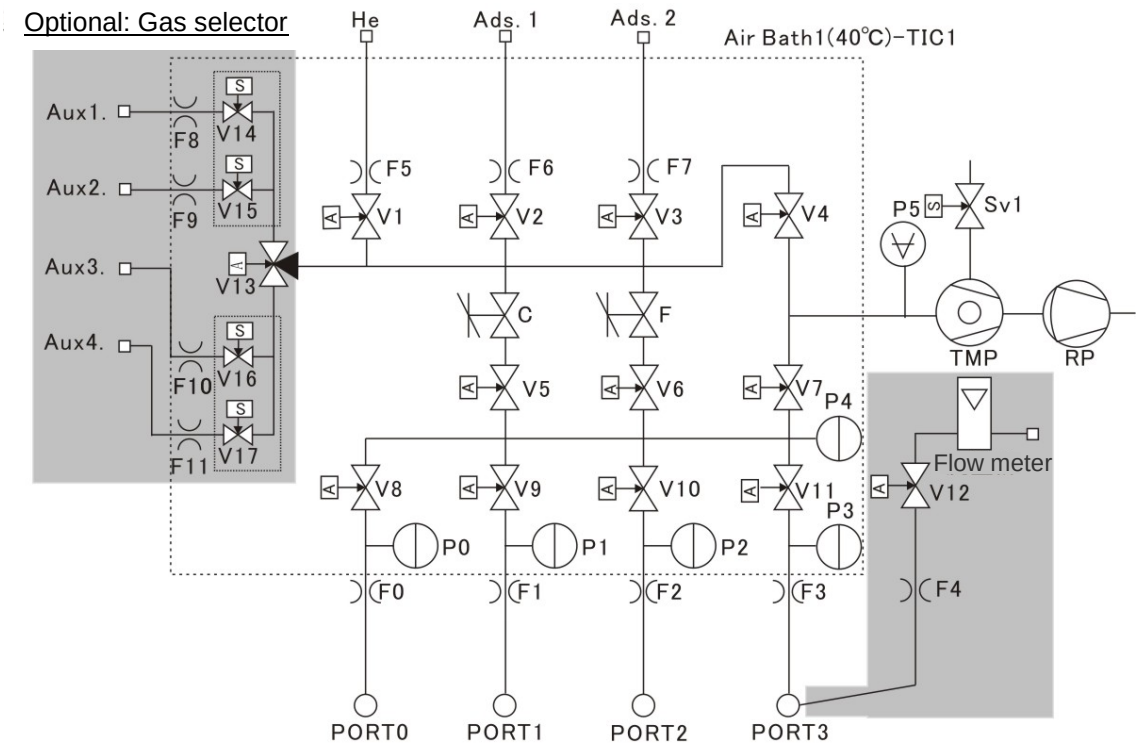
Model 2 (1 torr sensor) specification

Measurement method	Constant volume gas adsorption method+AFSM™	
Adsorptive	N_2, Ar, Kr, H_2, CO, O_2, CH_4, and other non-corrosive gas Steam (A corrosion resistant option is required for the adsorption measurement of corrosive gas such as NH_3 and amine, and organic vapor such as CH_3OH, C_6H_6, etc.)	
Number of samples to be measured	Standard mode ($P/P_0 = 10^{-7}$ to 0.997): 1 to 3 samples High precision mode ($P/P_0 = 10^{-7}$ to 0.997): 1 to 2 samples (according to AFSM™)	
Specific surface area measurement range [1)]	0.01 $m^2 g^{-1}$ or more (N_2 / 77K) 0.0005 $m^2 g^{-1}$ or more (Kr/77K)	
Pore size distribution (diameter)	0.35 to 500 nm	
Pressure sensor	133 kPa	5 units (Accuracy: ±0.25 % of F. S.)
	1.33 kPa	1 unit (Accuracy: ±0. 5 % of R.)
	133 Pa	1 unit (Accuracy: ±0.12 % of R) (OP. + 1 unit)
Pressure resolution	1.6×10^{-5} Pa	
Manifold temperature	40 °C (Option 50 °C)	
Dewar vessel	Volume: 2.6 L, Holding time: 60 h	
Sample cell	About 1.8 cm^3 (Optional: 5 cm^3)	
Exhaust system	Turbo molecular pump + Rotary pump : Attainable vacuum 6.7 x 10^{-7} Pa or less (manufacturer's specification) (Optional: Oil free exhaust system)	
Vacuum gauge	Cold cathode gauge (2 x 10^{-7} Pa to 1 Pa)	
Measurement software	Pretreatment and adsorption/desorption isotherm measurement	
Analysis program (BELMaster™)	・ Adsorption/desorption isotherm ・ Specific surface area by the Langmuir method ・ Pore volume calculation by the DA method ・ Micro-pore analysis by the MP method, HK method, and SF method ・ Diferece of adosorption method ・ Specific surface area by the BET method ・ Meso-pore size distribution by the DH, CI, BJH method ・ Micro-pore volume and micro-pore size analysis by the t-method and the α_S-method ・ Equivalent differential adsorption heat analysis ・ Pore size distribution analysis by the NLDFT/GCMC (BELSim™) (Optional)	

Required PC environment	OS: Windows 2000, XP, Vista, 7 CPU: Intel Processor Memory: 2 GB or more Hard disc: Free spaces of 1 GB or more USB port: USB 2.0
Auxiliary equipment	Rotary pump displacement: 50 L min^{-1} Attainable vacuum: 6.7 x 10^{-2} Pa
Utility	He, adsorptive (N_2, Ar, etc.) : 1/8" Swage lock joint (0.1MPa (gauge pressure)) Air for valve operation : 1/4" plastics tube quick connec(0.5 to 0.6MPa (gauge pressure)) Rotary pump connection port : NW16
Dimension / weight	W565 x H850 x D580 mm, 84 kg (Excluding a vacuum pump and computer related equipment)
Power	Single phase, AC100 to 240 V /800 VA (max. 700 VA for roughing vacuum pump)

1) The minimum specific surface area to be measured depends on the sample density.

Model 2 (1 Torr sensor) specification - main unit



①	V1 to V17	: Valve
②	C, F	: Flow rate adjustment needle valve
③	P0 to P4	: Pressure sensor
④	P5	: Vacuum gauge
⑤	TIC1	: Manifold temperature controller
⑥	Sv1	: Vent valve
⑦	TMP	: Turbo molecular pump

① Valve (Standard unit: V1 to 11)

V1 to 11: pneumatic valves

V1 : To dose helium gas

V2 : To dose the Ads. 1 gas

V3 : To dose the Ads. 2 gas or vapor

V4 : To exhaust the gas accumulator

V5 : To dose gas to the reference volume (large flow rate line)

V6 : To dose gas to the reference volume (small flow rate line)

V7 : To exhaust the reference volume

V8 : A valve between the P0 port and the reference volume

V9 : A valve between the measurement port 1 and the reference volume

V10 : A valve between the measurement port 2 and the reference volume

V11 : A valve between the measurement port 3 and the reference volume

Valve (optional)

Flow gas pretreatment line

A pneumatic valve

V12 : To be used in the flow gas pretreatment

Gas selector

V13: Pneumatic valve, V14 to V17: Solenoid valves

V13 : A valve between the gas selector and the gas accumulator

V14 : To dose the Aux. 1 gas

V15 : To dose the Aux. 2 gas

V16 : To dose the Aux. 3 gas

V17 : To dose the Aux. 4 gas

② Flow rate adjustment needle valve (C, F)

C : For large flow rate (pressure) control

F : For small flow rate (pressure) control

③ Pressure sensor (P0 to P4)

The following pressure gauges, with the indicated respective full scale, are installed to each port.

P0 : 133 kPa

To measure the pressure at the saturation vapor pressure measurement port (P0 port)

P1 : 133k Pa (Optional: 0.133kPa)

To measure the pressure at the measurement port 1

P2 : 133 kPa

To measure the pressure at the measurement port 2

P3 : 133 kPa, 0.133 kPa

To measure the pressure at the measurement port 3

P4 : 133 kPa, 1.33 kPa

To measure the pressure in the reference volume

Accuracy: 133 kPa → ±0.25 % (full scale)

: 1.33 kPa → ±0.5 % (reading)

: 0.133 kPa → ±0.12 % (reading)

④ Vacuum gauge (P5)

Cold cathode type pressure sensor

Measurement range: 2 x 10^{-7} Pa to 1 Pa

⚠ Do not use the vacuum gauge at pressure above 0.1 Pa.

⑤ Manifold temperature controller (TIC1)

Turn on the power switch on the back of the main unit, then the thermostatic manifold temperature is controlled to be 40°C (Option 50 °C). The manifold temperature is measured using a platinum temperature detector.

⑥ Vent valve (Sv1)

Turn off the circuit breaker on the back of the main unit, then this valve opens to vent the exhaust line to the open air to prevent the pump oil from flowing back to the instrument when the rotary pump is in operation.

⚠ Be sure to turn off the power switch and vent the exhaust line to the open air, before you shutdown the rotary pump. Otherwise, the pump oil may flow back to the main unit, and accordingly it may cause failure.

⑦ Turbo molecular pump (TMP)

Attainable vacuum: 6.7 x 10^{-7} Pa or below (manufacturer's specification)

It makes a high vacuum in the system.

⚠ Do not shut off power to the main unit while the turbo molecular pump is in operation, or for 30 minutes after it stops. When power is removed during the operation, or immediately after the shutdown, the system pressure rises rapidly up to atmospheric pressure. It may apply overload to the turbo molecular pump, and accordingly it may cause failure.

⚠ When accumulated operating time of the turbo molecular pump exceeds 20,000 hours, a maintenance work is prompted on the PC screen. Please contact our company if you find this message.

ooo 10 Torr sensor specification ooo

This model is equipped with a 10 Torr sensor and a 1000 Torr sensor, and performs the adsorption/desorption measurement in the range of $P/P_0 = 10^{-5}$ to 0.997. By installing an optional turbo molecular pump, it can perform the measurement at low pressure (Model 3 with a turbo molecular pump performs the measurement in the range of $P/P_0 = 10^{-6}$ to 0.997). For other features, refer to "About BELSORP-max on P. 11".

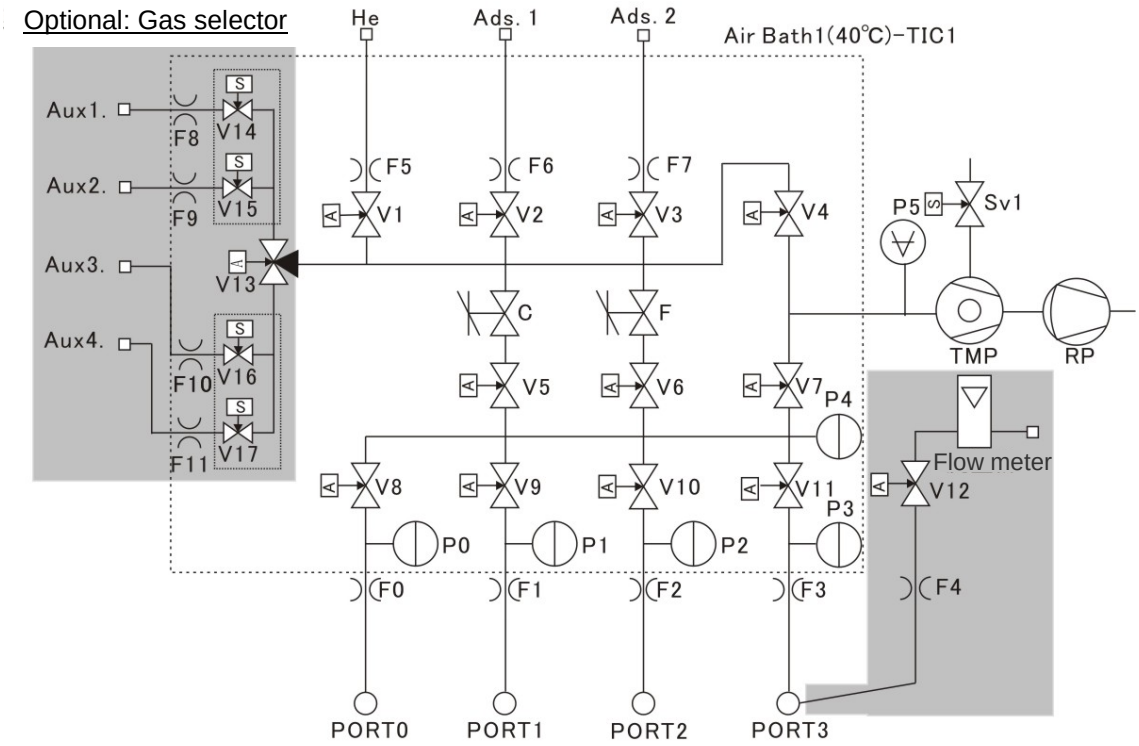
Model 3, 4 (10 Torr sensor) specification

Measurement method	Constant volume gas adsorption method+AFSM^{TM}	
Adsorptive	N_2, Ar, Kr, H_2, CO, O_2, CH_4, and other non-corrosive gas Steam (A corrosion resistant option is required for the adsorption measurement of corrosive gas such as NH_3 and amine, and organic vapor such as CH_3OH, C_6H_6, etc.)	
Number of samples to be measured	Standard mode ($P/P_0 = 10^{-5}$ to 0.997): 1 to 3 samples High precision mode ($P/P_0 = 10^{-5}$ to 0.997): 1 to 2 samples (according to AFSM^{TM}) In case of Model 3 + TMP: $P/P_0 = 10^{-6}$ to 0.997	
Specific surface area measurement range [1)]	0.01 $m^2 g^{-1}$ or more (N_2 / 77K) 0.0005 $m^2 g^{-1}$ or more (Kr/77K)	
Pore size distribution (diameter)	0.35 to 500 nm	
Pressure sensor	133 kPa	5 units (Accuracy: ±0.25 % of F. S.)
	1.33 kPa	1 unit (Accuracy: ±0. 5 % of R.)
Pressure resolution	1.6×10^{-5} Pa	
Manifold temperature	40 °C (Option 50 °C)	
Dewar vessel	Volume: 2.6 L, Holding time: 60 h	
Sample cell	About 1.8 cm^3 (Optional: 5 cm^3)	
Exhaust system	Rotary pump In case of Model 3 + TMP =: Attainable vacuum 6.7 x 10^{-7} Pa or less (manufacturer's specification) (Optional: Oil free exhaust system)	
Vacuum gauge	Pirani gauge (model3 Cold cathode gauge (2 x 10^{-7} Pa to atmospheric pressure)	
Measurement software	Pretreatment and adsorption/desorption isotherm measurement	

Analysis program (BELMaster™)	・ Adsorption/desorption isotherm ・ Specific surface area by the Langmuir method ・ Pore volume calculation by the DA method ・ Micro-pore analysis by the MP method, HK method, and SF method ・ Adsorption difference measurement ・ Specific surface area by the BET method ・ Meso-pore size distribution by the DH, CI, BJH method ・ Micro-pore volume and micro-pore size analysis by the t-method and the α_S-method ・ Equivalent differential adsorption heat analysis ・ Pore size distribution analysis by the NLDFT/GCMC (BELSim™) (Optional)
Required PC environment	OS: Windows 2000, XP, Vista, 7 CPU: Intel Processor Memory: 2 GB or more Hard disc: Free spaces of 1 GB or more USB port: USB 2.0
Auxiliary equipment	Rotary pump displacement: 50 L min^{-1} Attainable vacuum: 6.7 x 10^{-2} Pa
Utility	He, adsorptive (N_2, Ar, etc.) : 1/8" Swage lock joint (0.1MPa (gauge pressure)) Air for valve operation : 1/4" plastics tube quick connec(0.5 to 0.6MPa (gauge pressure)) Rotary pump connection port : NW16
Dimension / weight	W565 x H850 x D580 mm, 84 kg (Excluding a vacuum pump and computer related equipment)
Power	Single phase, AC100 to 240 V /800 VA (max. 700 VA for roughing vacuum pump)

1) The minimum specific surface area to be measured depends on the sample density

Model 3, 4 (10 Torr sensor) specification-unit



①	V1 to V17	: Valve
②	C, F	: Flow rate adjustment needle valve
③	P0 to P4	: Pressure sensor
④	P5	: Vacuum gauge
⑤	TIC1	: Manifold temperature controller
⑥	Sv1	: Vent valve
⑦	TMP	: Turbo molecular pump

① Valve (Standard unit: V1 to 11)

V1 to 11: pneumatic valves

V1 : To dose helium gas

V2 : To dose the Ads. 1 gas

V3 : To dose the Ads. 2 gas or vapor

V4 : To exhaust the gas accumulator

V5 : To dose gas to the reference volume (large flow rate line)

V6 : To dose gas to the reference volume (small flow rate line)

V7 : To exhaust the reference volume

V8 : A valve between the P0 port and the reference volume

V9 : A valve between the measurement port 1 and the reference volume

V10 : A valve between the measurement port 2 and the reference volume

V11 : A valve between the measurement port 3 and the reference volume

Valve (optional)

Flow gas pretreatment line

A pneumatic valve

V12 : To be used in the flow gas pretreatment

Gas selector

V13: Pneumatic valve, V14 to V17: Solenoid valves

V13 : A valve between the gas selector and the gas accumulator

V14 : To dose the Aux. 1 gas

V15 : To dose the Aux. 2 gas

V16 : To dose the Aux. 3 gas

V17 : To dose the Aux. 4 gas

② Flow rate adjustment needle valve (C, F)

C : For large flow rate (pressure) control

F : For small flow rate (pressure) control

③ Pressure sensor (P0 to P4)

The following pressure gauges, with the indicated respective full scale, are installed to each port.

P0 : 133 kPa

To measure the pressure at the saturation vapor pressure measurement port (P0 port)

P1 : 133k Pa (Optional: 1.33kPa)

To measure the pressure at the measurement port 1

P2 : 133 kPa

To measure the pressure at the measurement port 2

P3 : 133 kPa, 1.33 kPa

To measure the pressure at the measurement port 3

P4 : 133 kPa, 1.33 kPa

To measure the pressure in the reference volume

Accuracy: 133 kPa → ±0.25 % (full scale)

: 1.33 kPa → ±0.5 % (reading)

④ Vacuum gauge (P5)

Pirani gauge

Measurement range: 5 x 10^{-7} Pa to atmospheric pressure

(OP. cold cathode type pressure sensor Measurement range: 2 x 10^{-7} Pa to 1 Pa)

⚠ Do not use the vacuum gauge at pressure above 0.1 Pa.

⑤ Manifold temperature controller (TIC1)

Turn on the power switch on the back of the main unit, then the thermostatic manifold temperature is controlled to be 40°C (Option 50 °C). The manifold temperature is measured using a platinum temperature detector.

⑥ Vent valve (Sv1)

Turn off the circuit breaker on the back of the main unit, then this valve opens to vent the exhaust line to the open air to prevent the pump oil from flowing back to the instrument when the rotary pump is in operation.

⚠ Be sure to turn off the power switch and vent the exhaust line to the open air, before you shutdown the rotary pump. Otherwise, the pump oil may flow back to the main unit, and accordingly it may cause failure.

⑦ Turbo molecular pump (TMP)

Attainable vacuum: 6.7 x 10^{-7} Pa or below (manufacturer's specification)

It makes a high vacuum in the system.

⚠ Do not shut off power to the main unit while the turbo molecular pump is in operation, or for 30 minutes after it stops. When power is removed during the operation, or immediately after the shutdown, the system pressure rises rapidly up to atmospheric pressure. It may apply overload to the turbo molecular pump, and accordingly it may cause failure.

⚠ When accumulated operating time of the turbo molecular pump exceeds 20,000 hours, a maintenance work is prompted on the PC screen. Please contact our company if you find this message.

BELSORP-max measurement software

ooo System environment required ooo

This automatic gas adsorption measurement software is specialized for “BELSORP-max”.

It can be used in the following system environments. Standard BELSORP-max does not include a personal computer. You may use your personal computer.

<table>
<tr><th colspan="5">[Personal computer]</th></tr>
<tr><td></td><td colspan="4">Required system environment</td></tr>
<tr><td rowspan="2">Operating system</td><td>Microsoft Windows® 7 Home Basic or Home Premium or more</td><td>Microsoft Windows® Vista</td><td>Microsoft Windows® XP Home or Professional Edition</td><td>Microsoft Windows® 2000</td></tr>
<tr><td colspan="4">(Any computer that can run the English version of these OS)</td></tr>
<tr><td>CPU</td><td colspan="4">Intel processor</td></tr>
<tr><td>Memory</td><td colspan="2">2 GB or more</td><td>512 MB or more</td><td>256 MB or more</td></tr>
<tr><td>Display</td><td colspan="4">XGA (1024 × 768 dots) or more</td></tr>
<tr><td>Hard disk capacity</td><td colspan="4">10 GB or more space is required during operation.</td></tr>
<tr><td>USB port</td><td colspan="4">At least one USB1.1 / USB2.0 port</td></tr>
<tr><td>Disk drive</td><td colspan="4">CD-ROM drive
(For setup CD installation, and reading Config CD)</td></tr>
</table>

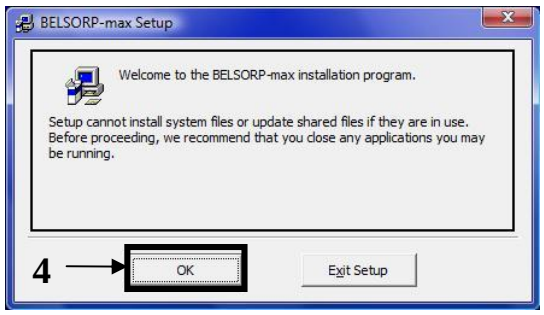
ooo Installing the software ooo

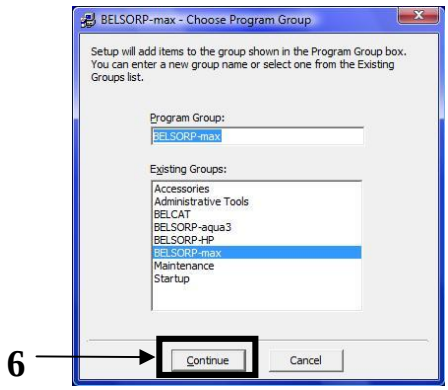
★ When measurement software has been already installed, delete the measurement software according to the "Uninstallation" in the next section before conducting the following installation procedure.

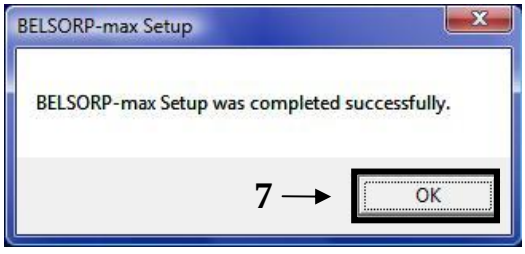
(For the basic operation of Windows, refer to the Windows instruction manual. The screens shown below are those in the Windows 7 mode.)

1. Installing the measurement software

1. Insert "Setup CD" to the CD-ROM drive.
2. Start "SETUP.EXE" in the "Measurement software ¥SETUP" folder stored in the "Setup CD".
3. After this is loaded, a screen appears as shown on the right.
4. Select the [OK] button.
5. Confirm the installation destination folder and then click the  button.
6. A screen appears as shown on the right.
   Select the [Continue] button to start setup.
7. When the setup is complete, a screen appears as shown below.
   Select the [OK] button.







2. Installing the USB driver

The main unit and the computer are connected using a USB cable. Install the USB driver according to the procedure described below.

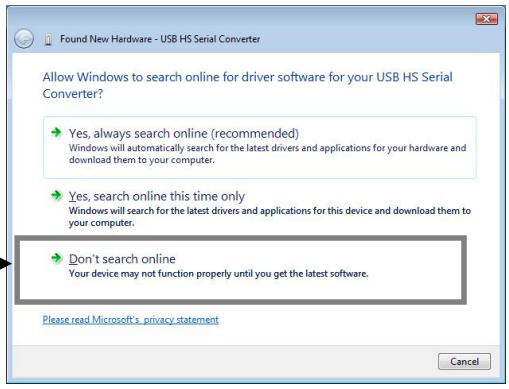
In the case of Windows® Vista

1. Turn on the main unit and the computer.

2. Connect the main unit to the computer using a USB cable.

3. A screen appears as shown on the right. Select "Locate and install driver software (recommended)".

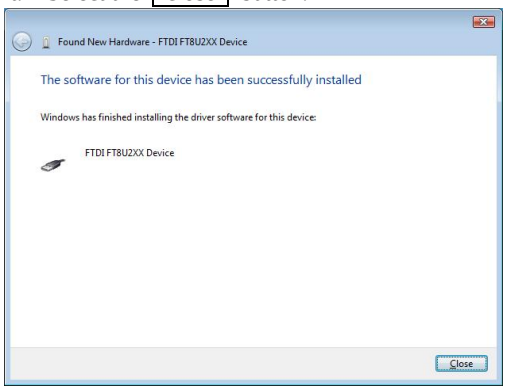
4. A screen appears as shown on the right. Select "Don't search online".



5. A screen appears as shown on the right. Insert "Setup CD" to the CD-ROM drive.

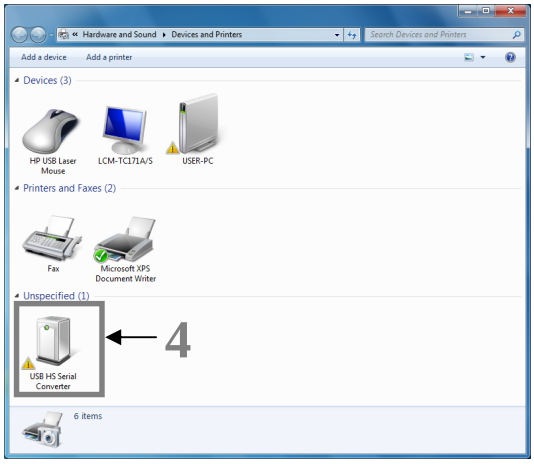
6. Select Next.

When the setup is complete, a screen appears as shown on the right. Select the close button.

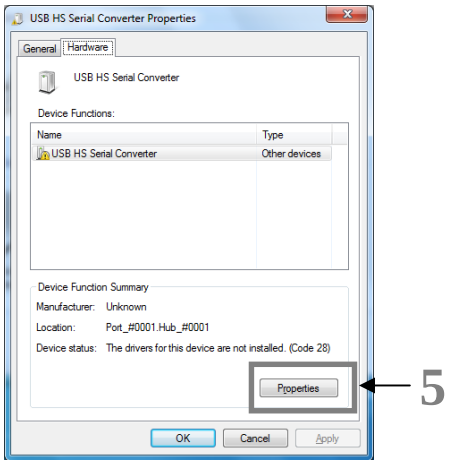


In the case of Windows® 7

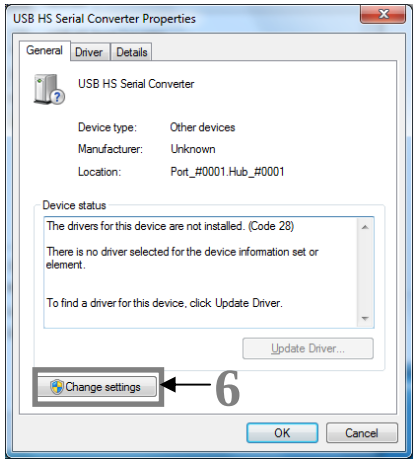
1. Turn on the main unit and the computer.
2. Connect the main unit to the computer using a USB cable.
3. Select Windows “Start” > “device and printa”
4. A screen appears as shown on the right. Righe click “USB HS Serial Converter”, and select “property”.



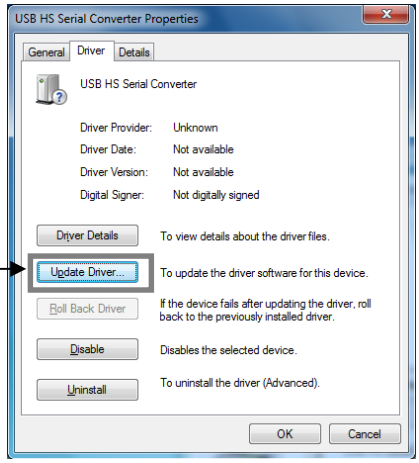
5. A screen appears as shown on the right. Select “property”.



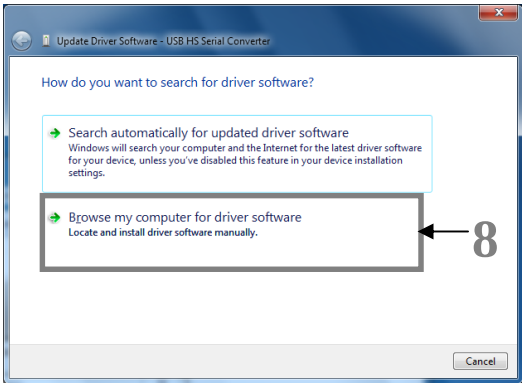
6. A screen appears as shown on the right. Select “change settings”.



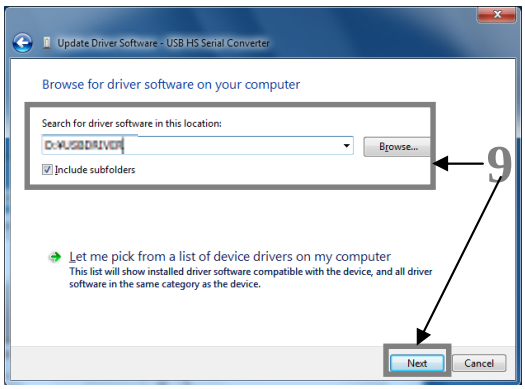
7. A screen appears as shown on the right. Select “updating driver”.



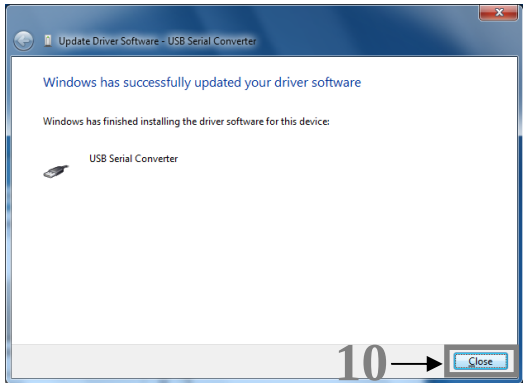
8. A screen appears as shown on the right. Select "Browse my computer for driver software".



9. A screen appears as shown on the right. Select "D: ¥USB DRIVER" in the "Setup CD", and select Next.



10. A screen appears as shown on the right. Select "close".



3. Copying the initial configuration file (Config. ini),(after Ver. 1.0.14)

1. The initial configuration file (Config. ini) contains various values specific to BELSORP-max, including the “reference volume”, etc. Copy these numeric data from the “Config CD” to the “SendDate” folder in the “BELmax” folder that has been already installed in section **1.** .

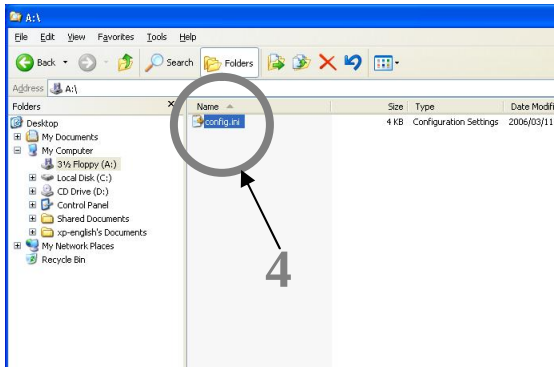
```
:**************************************************************************
:*                          Initial parameter file                        *
:**************************************************************************
:
:==========================================================================
:[Product name] BELSORP-max
:--------------------------------------------------------------------------
:[Last update] 2009/9/15
:==========================================================================
:
:------------------
:Instrument parameter
:------------------
        S/N             ="00001"        :Product serial No. [S/N]
        BELmax_type     =121            :Type 111, 121
                                        :     211, 212, 221, 222
                                        :     311, 321
:
        VS_0            =24.0000        :Standard volume (Vs[ml])
:
        FLOW_V12        =0              :Flow V12           (0=none, 1=yes)
        FLOW_V13_V17    =0              :Flow V13-17        (0=none, 1=yes)

        PUMP_KIND       =1              :0=RP,1=DF
:
:------------------
:RDA-016 Paramater
:------------------
        USB_SERIAL_NO   ="BELmax 00001"  :USB serial No. BELmax 00000
:
        AVE_COUNT       =40             :%

:------------------
```

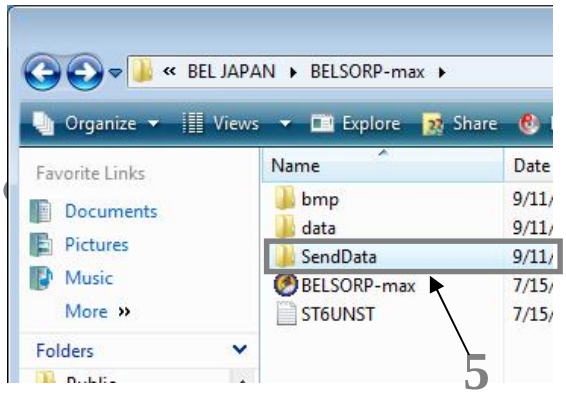
2. Insert the “Config CD” to the CD Drive (D:).

3. Select the Windows “Start” > “Program” > “Accessory” > “Explorer” to start “Explorer”.



4. Select the “Config. ini” file in the “CD Drive (D:)” folder, and Copy (C) it.

5. Paste (P) the “Config. ini” file to the “SendDate” folder in the folder that was created through “BELmax” or “**1.** Installing the measurement software – **5**”.



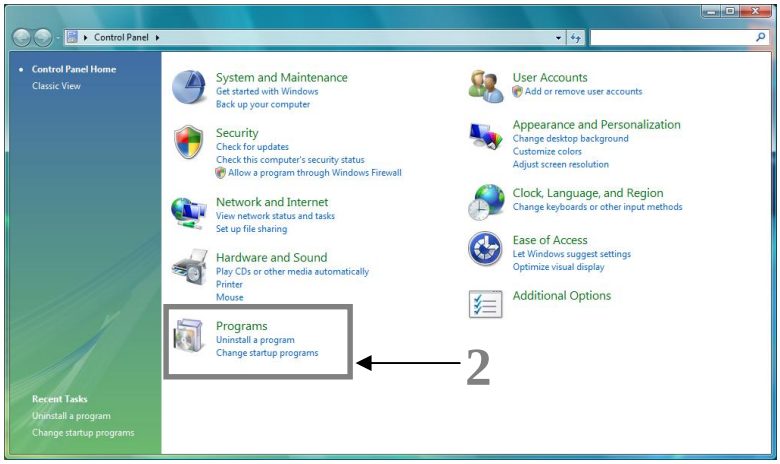
6. Exit “Explorer”. Now, the measurement software installation is complete.

ooo Uninstalling the software ooo

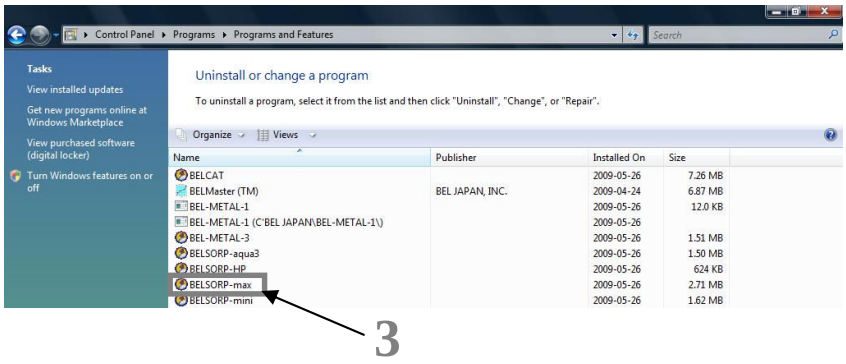
1. Uninstalling the measurement software

1. Select the Windows “Start” > “Control panel” to display the “Control panel” window.

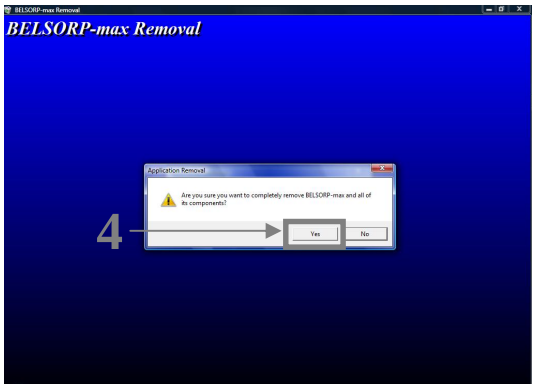
2. Start “Add or Remove Programs”.



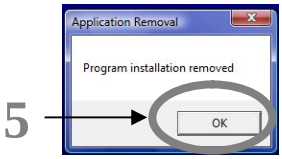
3. Select "BELmax" in the list.



4. Press the [Yes] button to start uninstallation.



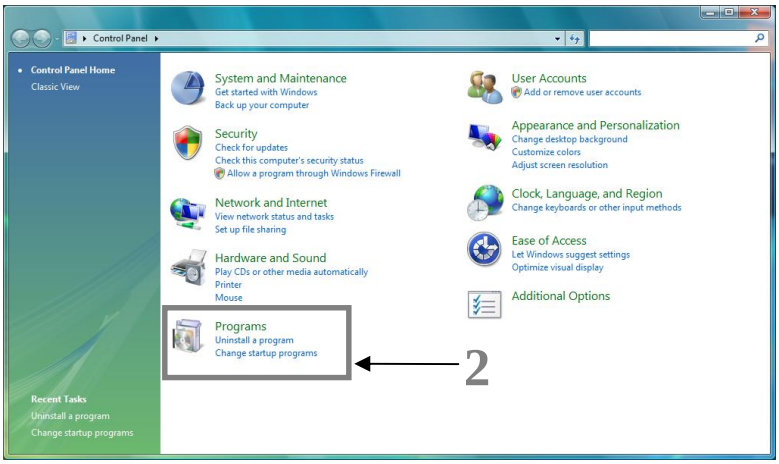
5. When the uninstallation is complete, a window appears as shown on the right. Press the [OK] button.



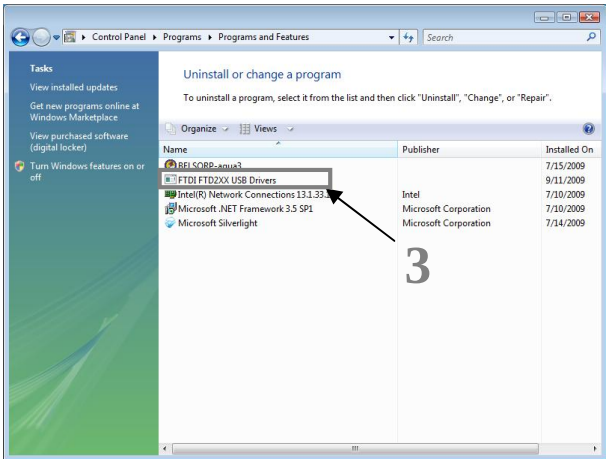
2. Uninstalling the USB driver

1. Select the Windows “Start” > “Control Panel” to display the “Control Panel” window.

2. Start “Add or Remove Programs”.

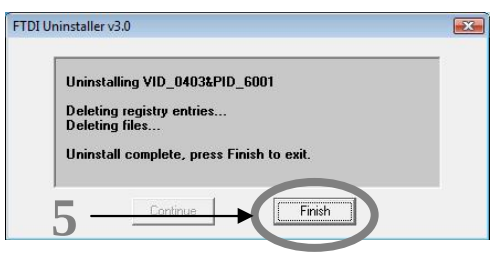


3. Select “FTDI FTD2XX USB Drivers” in the list of the applications installed, and select the “FTDI FTD2XX USB Drivers” button.



4. Press the Continue button to start uninstallation.

5. When the uninstallation is complete, a window appears as shown on the right. Press the Finish button. Now, uninstallation is complete.



ooo Basic software operation ooo

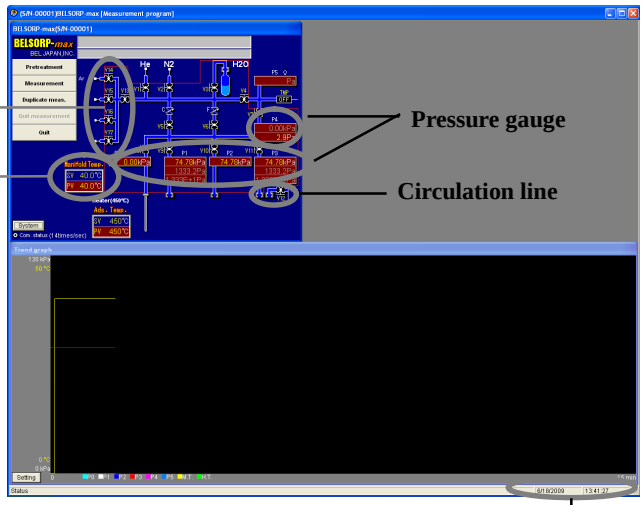
Starting the measurement software

1. Verify that the main unit and the computer are connected properly using a connection cable.
2. Check that power is supplied to the instrument.
3. Turn on the personal computer, and start “Windows”.
4. Select “Start” > “Program” menu > “BELmax” > “BELmax”.
5. The measurement software starts, and then the “Main” window appears. When the current status is displayed for the following items, startup is complete (The software screen shown below appears when you select the gas selector and the flow gas pretreatment line as options.).

- Current date and time
- Pressure gauge (P0-P4)
- Maniforld temperature (TIC1)

The following items are displayed when you select it as an option.

- Gas selector (V13 to V17)
- Circulation line (V12)



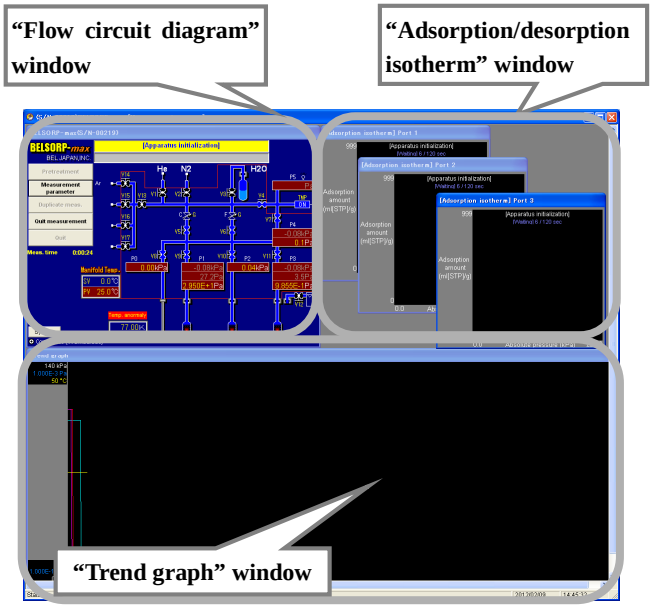
6. In the event of error in the communication line, including such cases that the main unit and the computer are not connected properly using a connection cable, etc. “Communication error” window is displayed. Check the power supply to the main unit, as well as connection of the power cable and the communication cable.

* If the valves (V1 to V17) still remain as opened manually when the software was used previously, all valves close when the software starts.

Operating the stop valves and specifying the temperature setting from the “Flow circuit diagram” window

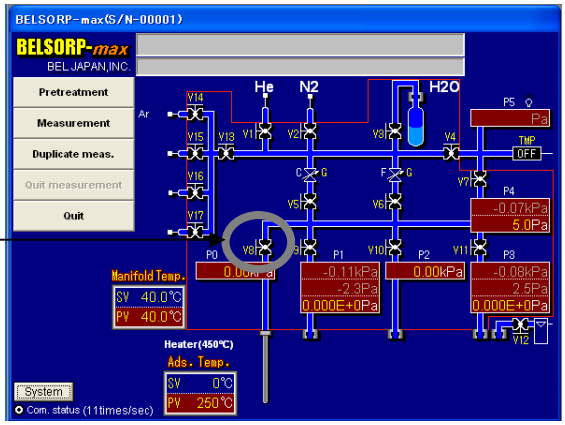
You can control the stop valves or change the temperature setting manually at any time; however, do not operate it during the automatic measurement.

The “Main” window of the measurement software (shown on the right) consists of the “Flow circuit diagram” window, the “Trend graph” window, and the “Adsorption/desorption isotherm” windows. The stop valves (V1 to V17) operation, vacuum gauges (P5), and turbo molecular pump (TMP) can be controlled from the “Flow circuit diagram” window, while the manifold temperature and adsorption temperature (when using a heater and electric furnace) can be specified from the same window.

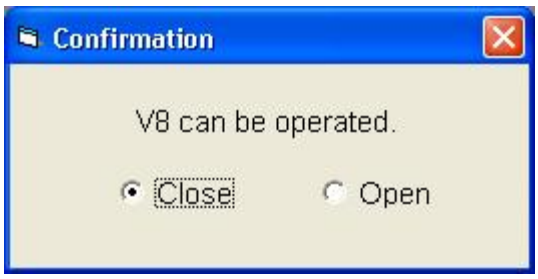


Operating the valves

1. On the “Flow circuit diagram” window, double-click the relevant mark of the valve (V1 to V17) to be operated.



2. Select “Close” or “Open” to operate the valve, and click the [X] button to close the “Confirmation” window.

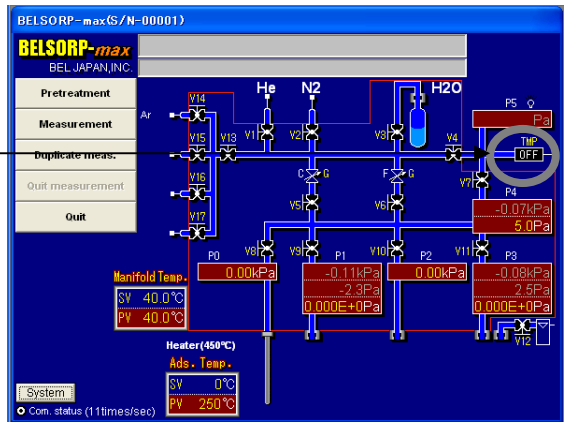


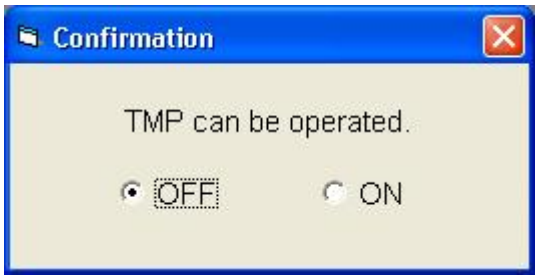
⚠ When you operate the valves manually, careful attention is required to prevent toxic/flammable gas from being discharged and to protect the turbo molecular pump from being overloaded.

Operating the turbo molecular pump

1. On the “Flow circuit diagram” window, double-click the ON or OFF mark of the TMP (turbo molecular pump).

2. Select OFF or ON, and use the × button to close the “TMP control” window. The TMP is controlled.

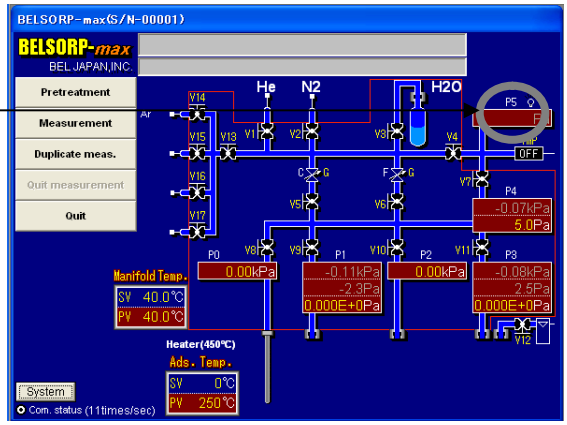




⚠ When you operate the turbo molecular pump manually, careful attention is required to prevent the turbo molecular pump from being overloaded.

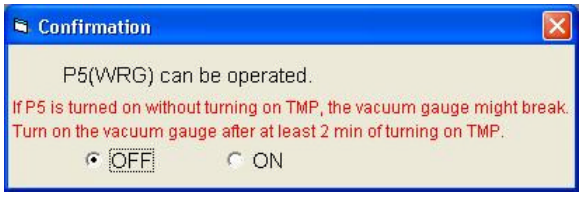
Operating the vacuum gauge P5 (WRG)

1. On the “Flow circuit diagram” window, double-click the lamp mark of the vacuum gauge P5 (WRG).



⚠ Do not turn on the WRG when the vacuum level is not sufficient. Otherwise, it may degrade filaments.

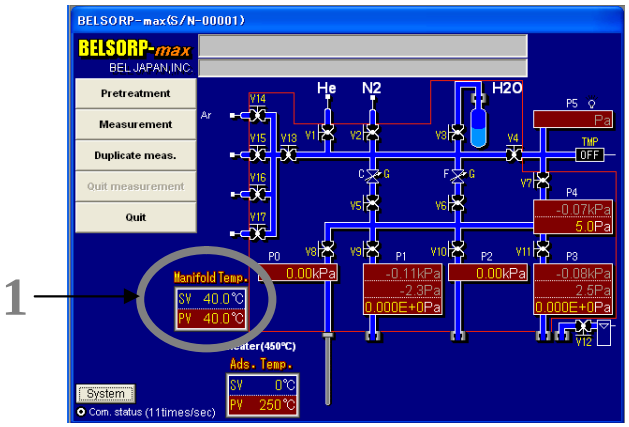
2. Select OFF or ON, and use the × button to close the “Confirmation” window. The vacuum gauge P5 (WRG) is controlled.



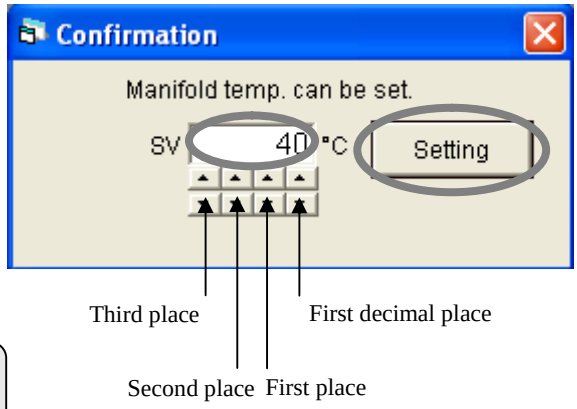
With 10 Torr (without TMP) specification, P5 is to be always displayed.

Operating the manifold temperature (Manifold Temp.)

1. On the “Flow circuit diagram” window, double-click the SV temperature indication of the manifold temperature (TIC1).



2. Enter the SV value (temperature setting), and select the Setting button. The SV value can be entered by using the ▲ (to increase), or ▼ (to decrease) button. Normally set it to 40 °C (Option 50 °C).



⚠ You may not specify any temperature above 40 °C(Option 50 °C).

Operating the measurement temperature (Heat Temp.)

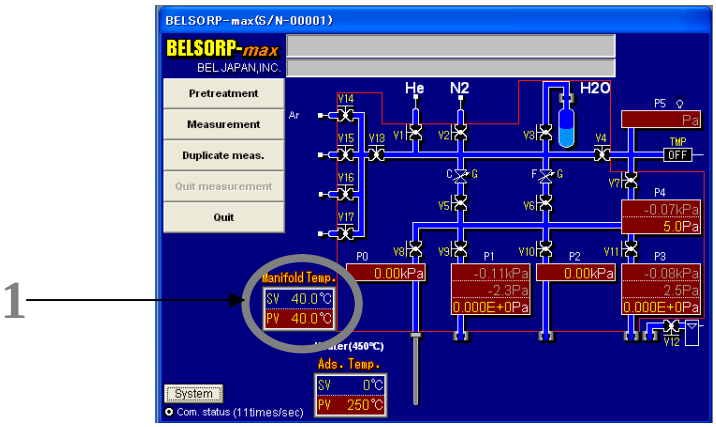
1. It can be operated when some temperature devices are connected, including a heater, electric furnace, and a temperature controller. On the “Flow circuit diagram” window, double-click the SV temperature indication of the adsorption temperature (TIC2). When any temperature device is not connected, the temperature indication is not displayed.

The temperature setting range is:

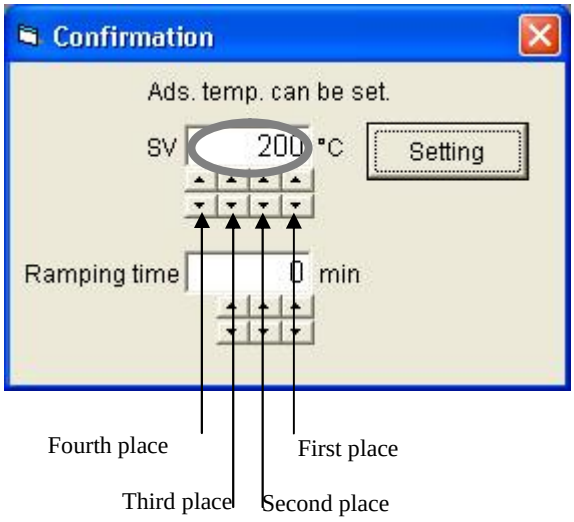
50 °C to 450 °C for the 450 °C heater,

50 °C to 550 °C for the 550 °C heater, and

50°C to 1100°C for the 1100 °C electric furnace.

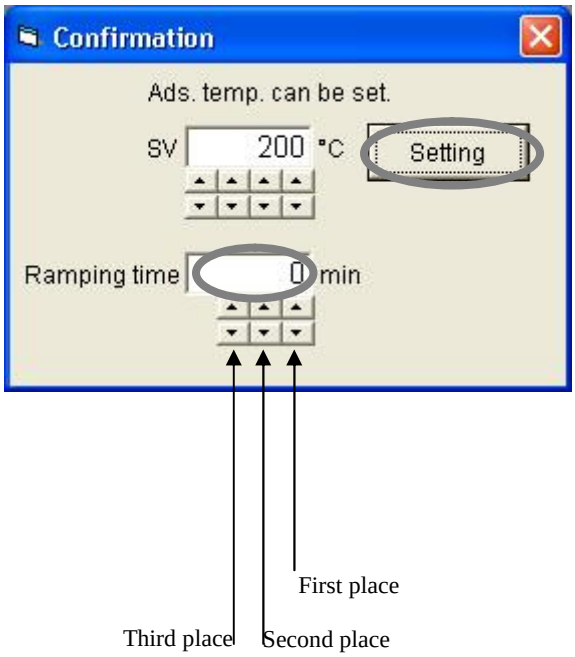


2. Enter the SV value (temperature setting). The SV value can be entered by using the ▲ (to increase), or ▼ (to decrease) button.



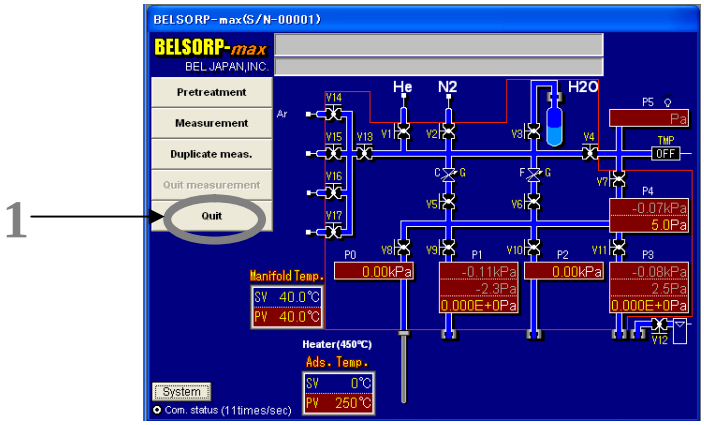
3. Enter the ramping time (minute). It can be entered by using the ▲ (to increase), or ▼ (to decrease) button. When the time setting is 1 minute or more, the temperature is controlled so that it changes from the initial PV value (current temperature) to the specified SV value in the time specified. In some cases, it cannot reach the target temperature in the time specified.

4. Select the Setting button. The adsorption temperature is controlled.

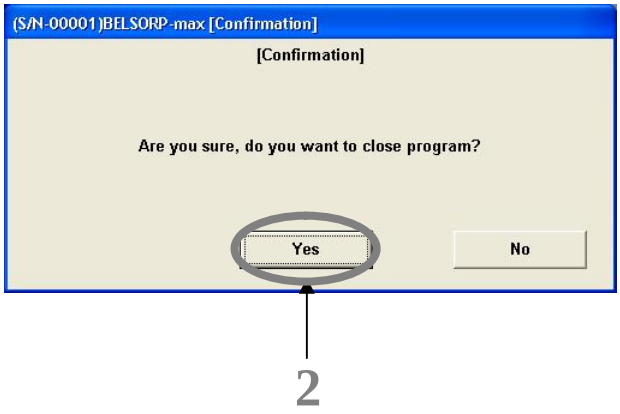


Exiting the measurement software

1. Select the Quit button, then the "Confirmation" window appears.



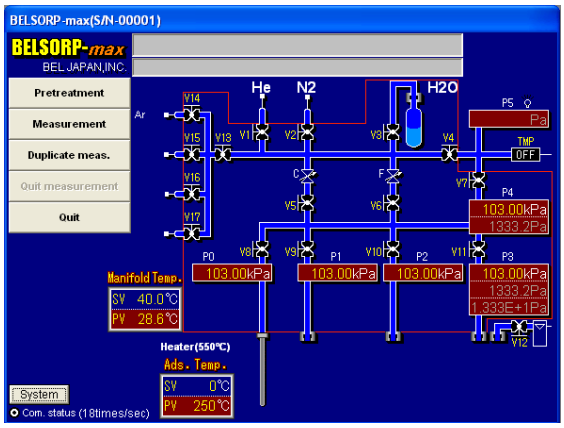
2. Select Yes to exit the measurement software. This software automatically closes all stop valves, and returns the instrument to the initial state.



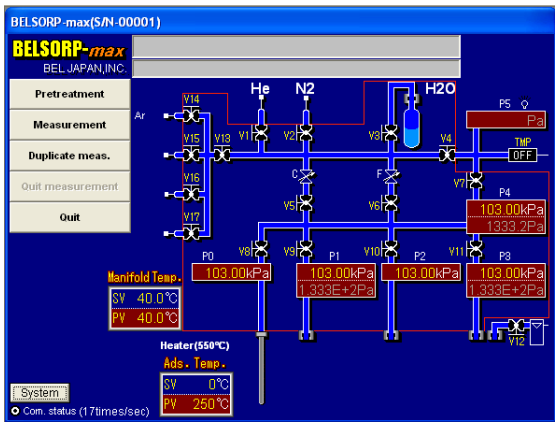
Software screen with optional specification

With 1 Torr, or 10 Torr sensor specification, the software screen appears as shown below (The basic software operation is the same as those with 0.1 Torr sensor specification).

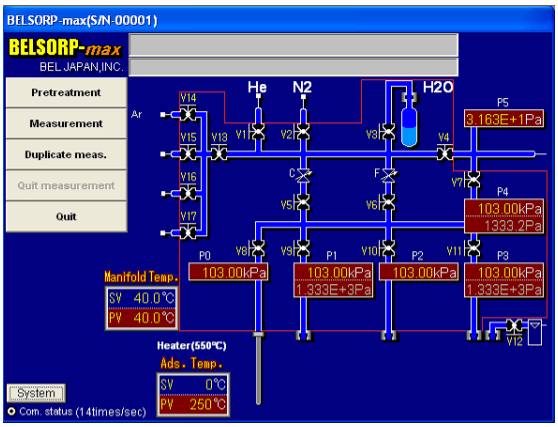
With 1 Torr specification



With 10 Torr specification (with TMP)



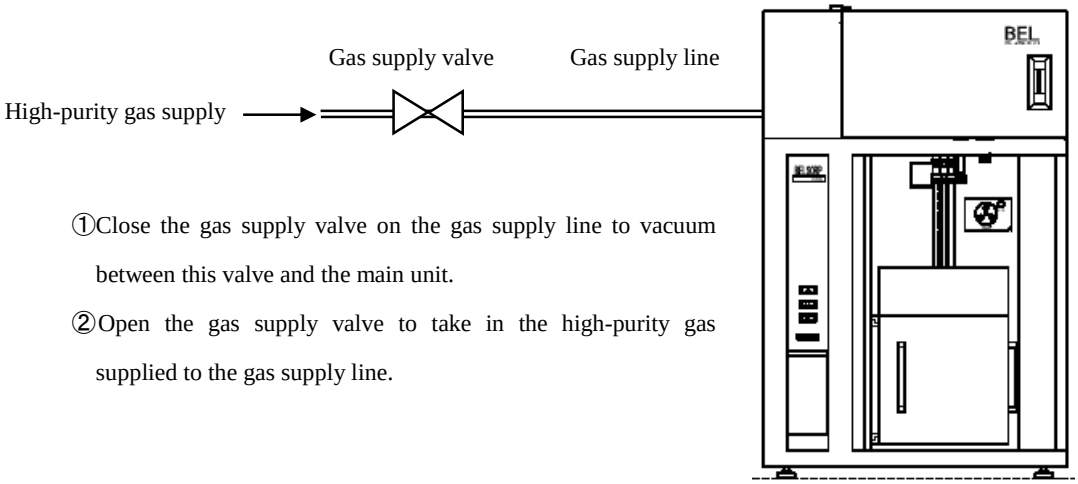
With 10 Torr specification (without TMP)



Guidance support

ooo Installing and replacing gas cylinders ooo

To measure adsorption/desorption correctly, a pure adsorptive gas and He gas are necessary. When a gas cylinder is replaced, air comes into the tube, so that gas purity in the gas supply line is degraded. Hence, it is necessary to wash the tube and fill in the gas supply line with a high-purity gas. The outline of tube washing is as follows:



①Close the gas supply valve on the gas supply line to vacuum between this valve and the main unit.

②Open the gas supply valve to take in the high-purity gas supplied to the gas supply line.

Fill in the gas supply line with the high-purity gas only by repeating the steps ① to ②.

BELSORP-max's measurement software is equipped with a guidance function to ensure easy operation when a gas pipe is attached for changing adsorptive gas or replacing the gas cylinder. The procedure to perform work by following this guidance is described from the next page. In this procedure, a gas cylinder is used as the gas source. Even when using other gas sources such as centralized line, wash the gas line in accordance with the following procedure. When you wash two or more different gas lines, be sure to perform it one by one.

The section of pipe washing by the software guidance function describes the case in which the regulat or mentioned on the next page is used. Change the operational method according to your regulator

Caution

Do not apply pressure more than 0.2 MPa (2 kgf cm^{-2}).

- **Otherwise, gas may flow back in the system, and accordingly the pressure sensor may be damaged.**

Do not use any corrosive gas (except for the corrosion resistant type).

Check the gas line for leakage, and verify that there is no leak.

Prevent any flammable gas or toxic gas from being discharged. Conduct abatement where applicable.

Make an appropriate arrangement for the pump exhaust port.

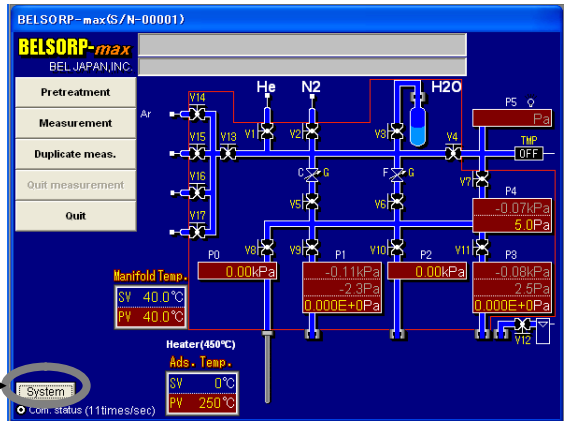
- **Particular attention is required in handling flammable gas, or toxic gas.**

Attention is required in using gas cylinders and compressed gas.

1. Connect the gas line between a gas cylinder and the **BELSORP-max** main unit. Check the line for leakage after it is connected.

2. Start the **BELSORP-max** measurement software. Close all valves when the software has been already started and valves are open.

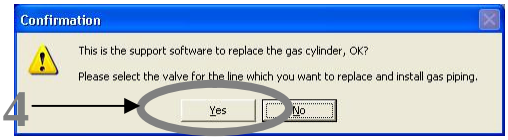
Press the System but ton at the lower left on the "Flow circuit diagram" window.



3. A window appears as shown on the right.

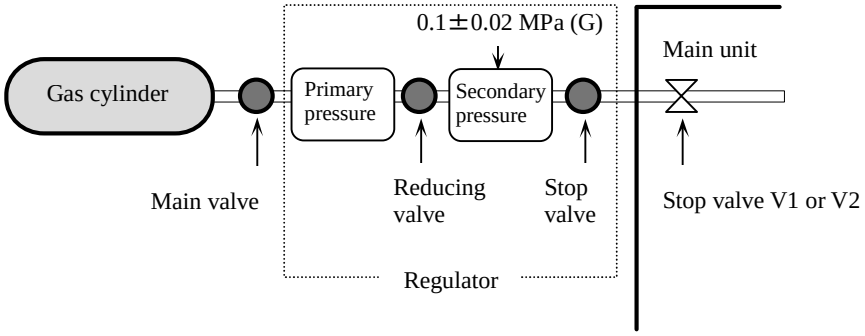
Select Exchanging gas cylinder.

4. When the "Confirmation" window is displayed, select Yes. In the next step **5** and later, follow the guidance message displayed on the "Confirmation" window.

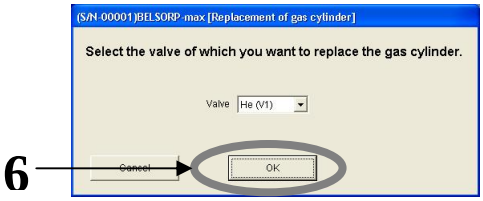


5. The terminology used in the "Confirmation" window is as follows.

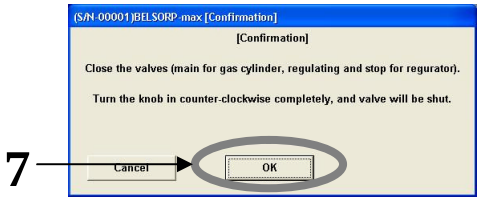
Main valve	A manual valve attached to a gas cylinder.
Reducing valve	A valve attached to a regulator for adjusting the secondary pressure.
Stop valve	A valve attached to a regulator. Gas is discharged when it opens.
Gas accumulator	A part of tubing in the main unit.



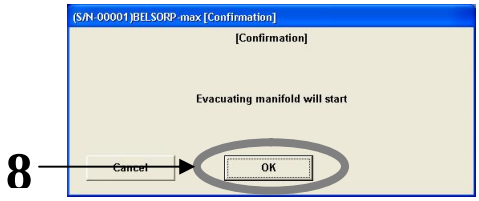
6. Select the valve of which gas cylinder you want to replace. After it is selected, press the OK button. For the adsorptive gas assignment to each valve, refer to the "Measurement parameter setting, Gas dosing valve assignment settings on P. 134".



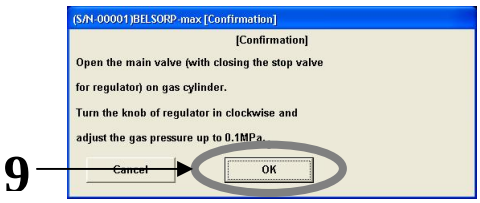
7. Close the gas cylinder main valve, the regulator stop -valve and the regulator reducing-valve; and then press the OK button.



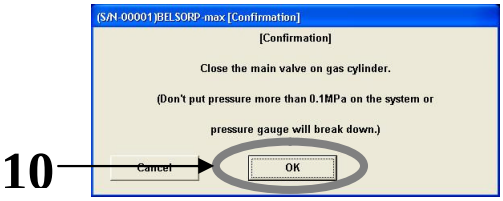
8. When a message is displayed as shown on the right, press the OK button. Firstly, air in the connection line is drawn into the system gas accumulator, and then discharged.



9. When a message is displayed a little while later as shown on the right, follow the message guidance, and then press the OK button to proceed to the next step.

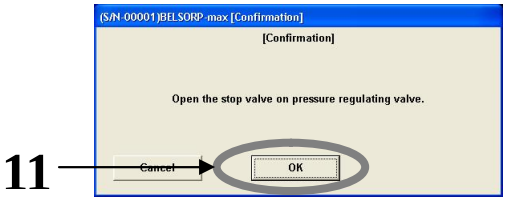


10. When a message is displayed as shown on the right, close the gas cylinder main valve, and then press the OK button to proceed to the next step.

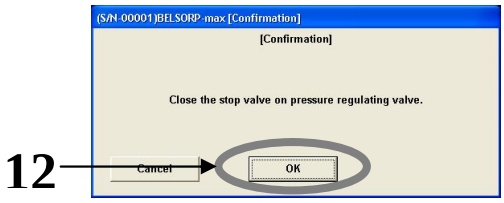


> Do not apply any pressure over 0.1 Mpa (G). The pressure sensor may be damaged.

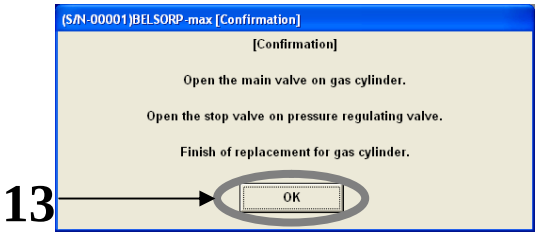
11. When a message is displayed as shown on the right, open the regulator stop valve, and then press the OK button to proceed to the next step. The helium gas accumulated in the regulator in steps **9.** and **10.** is released into the system, and then discharged (for washing inside the regulator).



12. When a message is displayed a little while later as shown on the right, close the regulator stop valve, and then press the OK button to proceed to the next step. Return to **9.,** and repeat the steps **9.** to **12.** two more times, i.e. a total of 3 times.



13. When a window appears as shown on the right, the gas cylinder replacement is complete. Finally, open the gas cylinder main valve and the regulator stop valve. Press the OK button to complete.



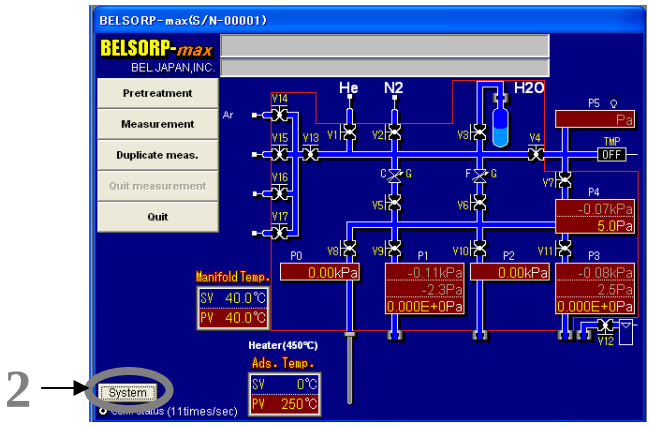
- Be sure to open the main valve and the stop valve at the end. Otherwise, any measurement cannot be performed with the valve closed.
- Verify that the regulator secondary pressure is 0.1 ± 0.02 MPa (G) after the replacement. In the automatic measurement and the “system check” described later, the regulator secondary pressure should be within the range mentioned above.

ooo System check ooo

Although individual devices are adjusted and inspected sufficiently before delivery, regulator needle valves for gas flow rate adjustment may be misadjusted during transportation. The “system check” function is used to check easily the regulator needle valve adjustment, the pressure sensor operation, and the valve operation. Any faults in the instrument may not only affect the measurement data accuracy, but also result in failure of the instrument. After the unit is installed or transferred, or when the unit has not been used for a long time period, be sure to perform this “system check”.

⚠ Do not install the sample cell to the measurement port, until it is prompted.

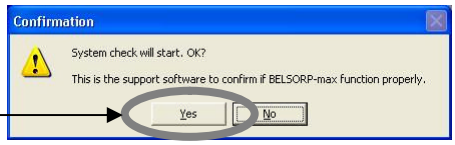
1. Start the BELSORP-max main unit and the measurement software.
2. Select the System button on the “Flow circuit diagram” window. The “System version information” window appears.



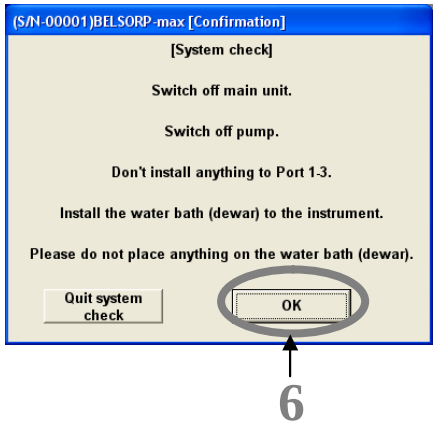
3. The system checks for the needle valve flow rate, valve operation, pressure sensor, Dewar vessel lifting operation, vacuum level, leakage, etc.. Remove the sample cells from Port 1 to 3 before you start the system check.

4. Select the System check… buttonon the “System version information” window.

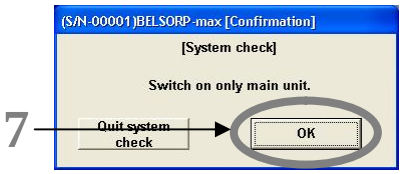
5. Select [Yes] to start the "System check".



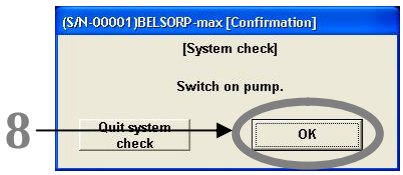
6. The system depressurizes the line between the main unit and the pump down to atmospheric pressure. When a window is displayed as shown on the right, turn off the main unit. Also, turn off the pump.



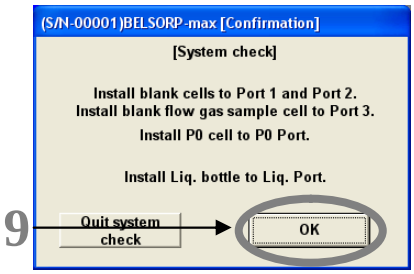
7. When a window is displayed as shown on the right, turn on the main unit only. Press the [OK] button.



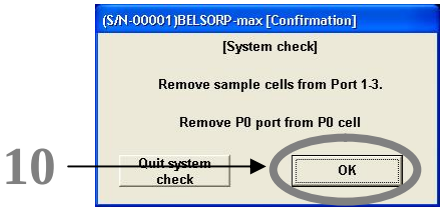
8. When a window is displayed as shown on the right, turn on the pump. Press the [OK] button.



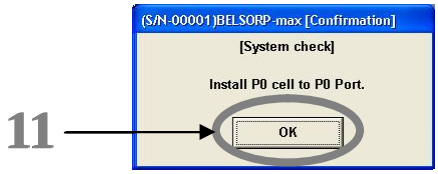
9. When a window is displayed as shown on the right, install blank sample cells to Port 1 to 2, a blank flow gas sample cell Port 3, a saturation vapor pressure reference cell to P0 port, and a liq. bottle to the adsorptive gas dosing port (ads.2), and then press the [OK] button. When the flow gas pretreatment line is equipped, install a blank flow gas sample cell to Port 3.



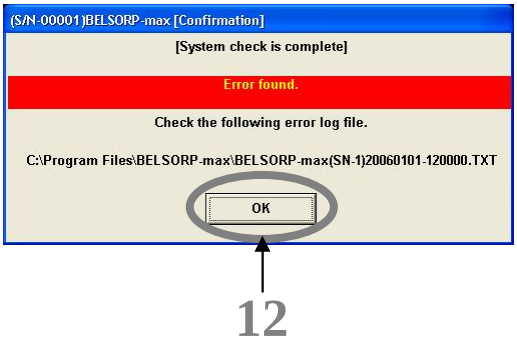
10. A window is displayed as shown on the right after the leak check. Remove the sample cells from Port 1 to 3, and the saturation vapor pressure reference cell from P0 port, and then press the [OK] button.



11. A window is displayed as shown on the right after you adjust the needle valves V5 and V6 flow rate. Install a saturation vapor pressure reference cell to P0 port, and press the OK button.



12. When the system check is not successfully complete, a window is displayed as shown on the right. Check the error log file. Press the OK to close the window.

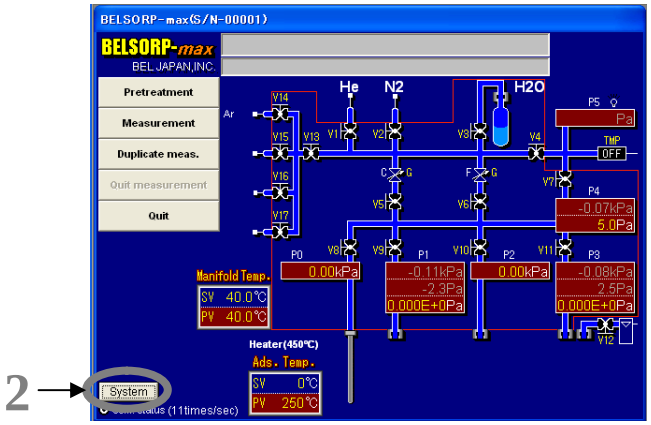


ooo Adjusting the needle valve ooo

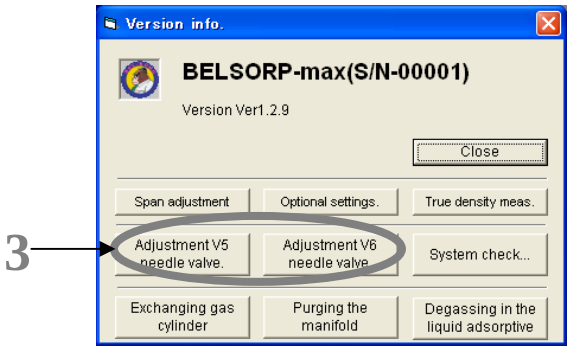
Adjust the needle valve to the appropriate flow rate.

	Function
Adjustment V5 needle valve	Check the flow rate through the needle valve V5 (C) for a large flow rate. Select this button to check the flow rate through the needle valve V5 (C). Adjust the needle valve V5 (C) so that it takes 20 to 30 seconds to dose helium gas up to 100 kPa. For the vapor measurement, adjust the needle valve V5 (C) so that it takes 7 to 10 seconds to dose helium gas up to 100 kPa.
Adjustment V6 needle valve	Check the flow rate through the needle valve V6 (F) for a small flow rate. Select this button to check the flow rate through the needle valve V6 (F). Adjust the needle valve V6 (F) so that it takes 20 to 30 seconds to dose helium gas up to 1 kPa. For the vapor measurement, adjust the needle valve V6 (F) so that it takes 80 to 100 seconds to dose helium gas up to 100 kPa.

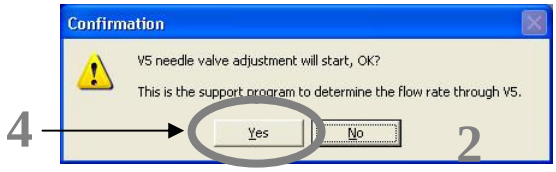
1. Start the **BELSORP-max** main unit and the measurement software.
2. Press the System button on the "Flow circuit diagram" window. The "System version information" window appears.



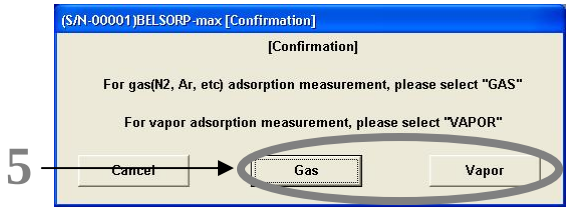
3. Select "Adjustment V5 needle valve" (or V6) on the "System version information" window.



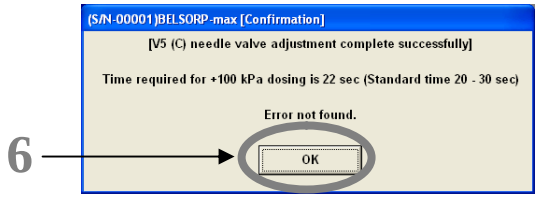
4. Select Yes.



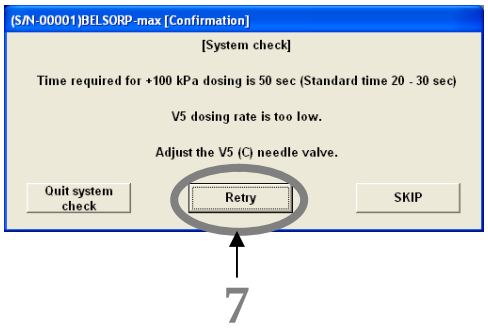
5. Select Gas for gas adsorption measurement, or select Vapor for vapor adsorption measurement. Then, start dosing helium gas to the reference volume buffer to determine the gas flow rate through the needle valve V5 (C) or V6 (F).



6. When the valve flow rate is appropriate, a window is displayed as shown on the right. Press the OK button.



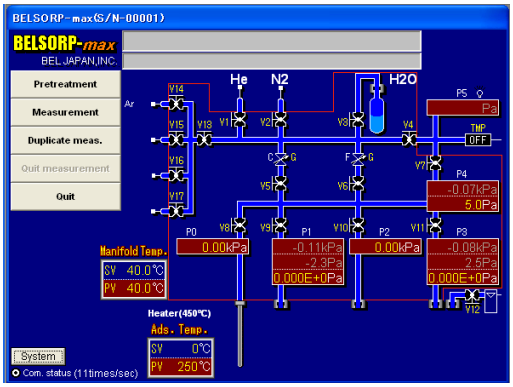
7. When the valve flow rate is not appropriate, a window is displayed as shown on the right. Select Retry to adjust it again.



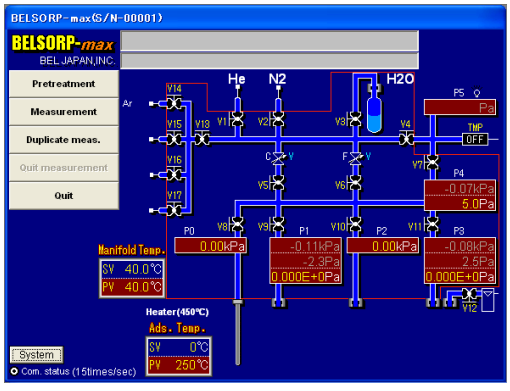
8. You can locate the “needle valve adjustment screw” in the back of the main unit measurement port. Adjust it by hand. Turn it counterclockwise to close the valve so that flow rate is decreased, or turn it clockwise to open the valve so that the flow rate is increased.

9. You can check adjustment of the needle valve is either gas (G) or vapor (V) in the flowchart.

【Setting gas (G)】



【Setting vapor (V)】



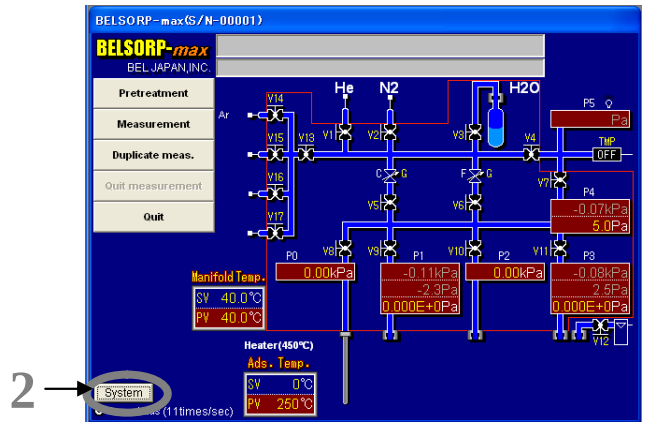
ooo Span adjustment ooo

The span adjustment is to prevent each pressure gauge from deviating, and to adjust it so that good balance is maintained. Perform this span adjustment when the instrument is installed, or the instrument has not been used for a long time period, or the pressure gauges deviate from each other. It is recommended to perform this span adjustment every time you perform the measurement at low pressure, or the low specific surface area measurement, the vapor measurement.

> ⚠ It is recommended to perform this span adjustment every time you perform the measurement at low pressure, or the low specific surface area measurement, the vapor measurement.

1. Start the BELSORP-max main unit and the measurement software.

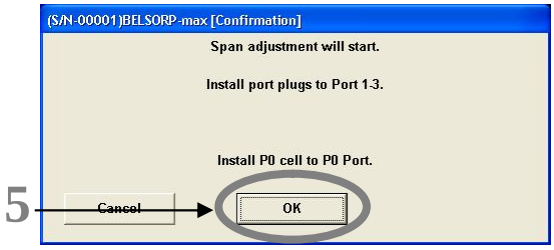
2. Press the System button on the "Flow circuit diagram" window. The "System version information" window appears.



3. Select the Span adjustment button.

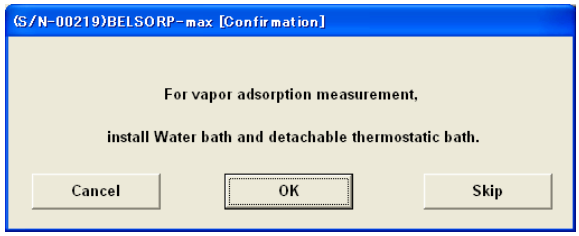
4. Select Yes to start the "span adjustment".

5. Install the port plugs to Port 1 to 3, install a saturation vapor pressure cell to P0 port, and then press the OK button. The span adjustment starts.

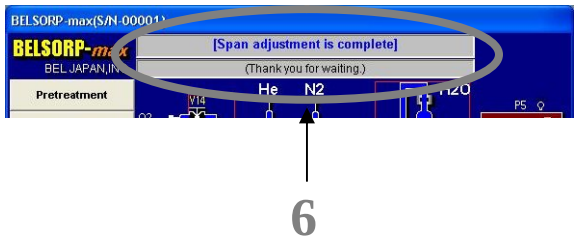


6. For vapor adsorption measurement, press the OK button.

For other adsorption measurement, choice Skip button.



7. When a message is indicated as shown on the right, the span adjustment is complete.



ooo Purging the tubing in the instrument ooo

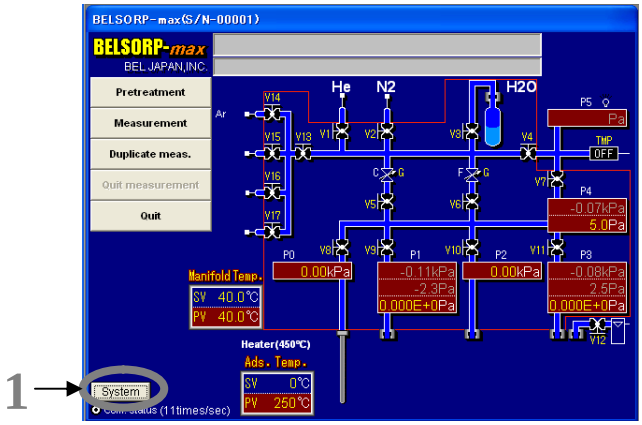
After the adsorptive is changed, the adsorptive previously used may still remain in the instrument tubing. This affects the subsequent measurement accuracy. When the instrument has not been used for a long time period such as more than two weeks, air and/or moisture may be contained in the instrument tubing. Additionally perform a purge each time a measurement is finished if a corrosive gas or a gas oxidizing in air (e.g. NO) is used. In such cases, you must purge the instrument tubing. With **BELSORP-max**, the measurement software has a guidance function to make this purging process easy. The following section describes the working procedure according to the guidance.

Caution

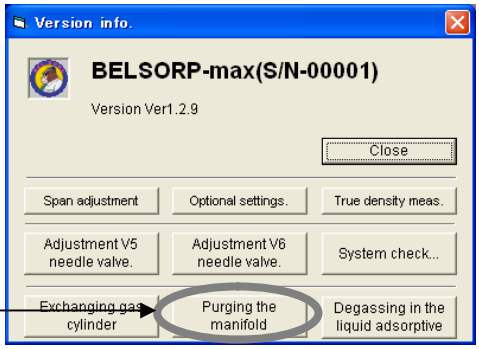
Install a helium gas cylinder before you attempt this work.

- **Use helium gas for purging.**

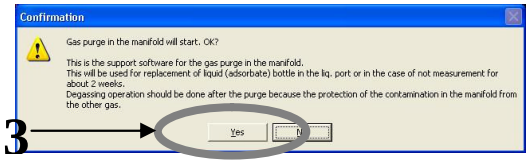
1. Start the **BELSORP-max** measurement software. Close all valves when the software has been already started and valves are open. Press the System button at the lower left on the "Flow circuit diagram" window.



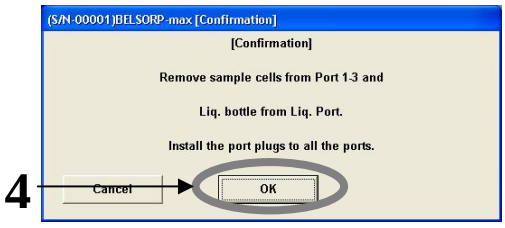
2. A window appears as shown on the right. Select Purging the manifold.



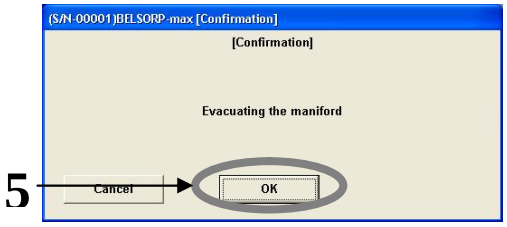
3. When the "Confirmation" window is displayed, select Yes. In the following step **4** and later, follow the guidance messages displayed on the "Confirmation" window.



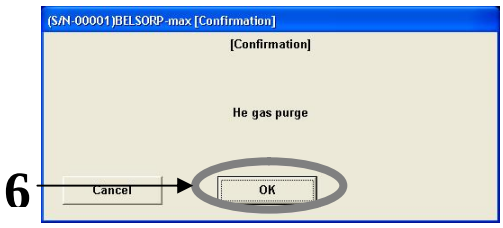
4. Remove the sample cell and the liq. bottle from the instrument, and install port plugs alternatively. When this is complete, press the OK button to proceed to the next step.



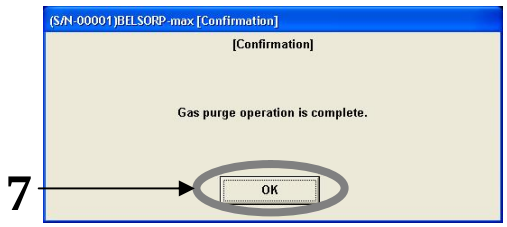
5. When a message is displayed as shown on the right, press the OK button to proceed to the next step. The whole instrument tubing is evacuated automatically for about 3 minutes.



6. A message will be displayed about 3 minutes later as shown on the right. Press the OK button to proceed to the next step. The main unit fills the whole instrument tubing with helium gas, and then discharges it. This purging process is repeated 3 times automatically.



7. When a window appears as shown on the right, the purging process is complete. Press the OK button.



ooo Degassing from adsorbate ooo

When you perform measurement using vapor gas as an adsorptive by installing a liq. bottle to the dosing gas port (ads. 2) (available only when the optional water bath is selected), you must remove the gas other than the adsorptive that is remaining in the liq. bottle and in the tubing between the valve V3 and the liq. bottle, and the gas dissolved in the adsorbate (liquid). When the instrument has not been used for a long time period such as more than a month, air and/or moisture may be contained in the liq. bottle or the tubing. In such cases, perform "degassing from adsorbate" according to the following procedure. With BELSORP-max, the measurement software has a guidance function to make degassing easy. The following section describes the working procedure according to the guidance.

Caution

Keep the adsorptive liquid level lower than 70 % of the liq. bottle.

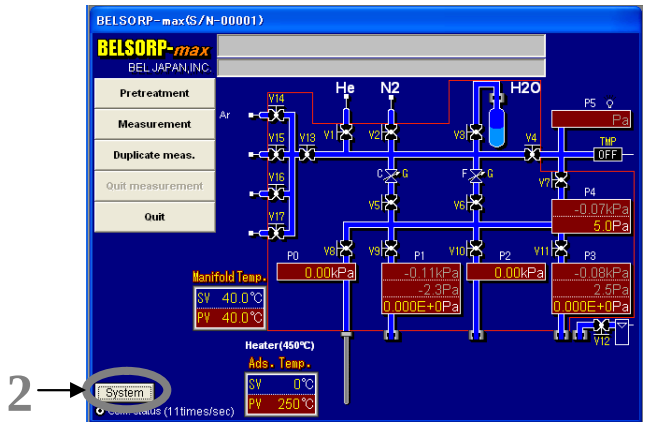
- **Otherwise, the liq. bottle may be broken during the degassing process.**

Careful attention is required in handling liquid nitrogen.

1. Wash the liq. bottle and dry it sufficiently.

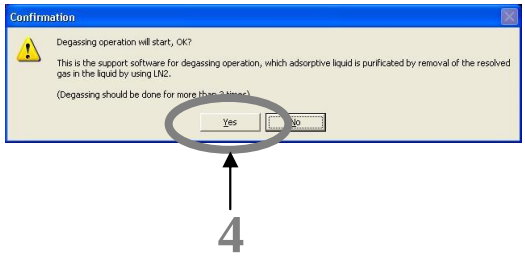
Be sure to wash the bottle with pure water, and then dry it sufficiently. Unclearn bottles may degrade the adsorptive purity. Accordingly, it may affect the measurement accuracy.

2. Start the **BELSORP-max** measurement software. Close all valves when the software has been already started and valves are open. Press the System button at the lower left on the "Flow circuit diagram" window.

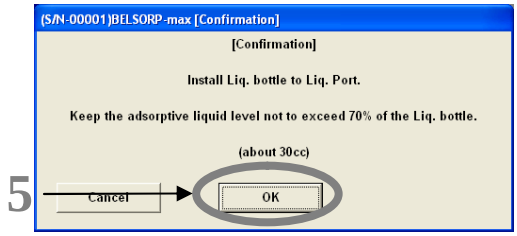


3. A window appears as shown on the right. Select "Degassing the liquid adsorptive".

4. When the "Confirmation" window is displayed, select Yes. In the following step **5.** and later, follow the guidance messages displayed on the "Confirmation" window.

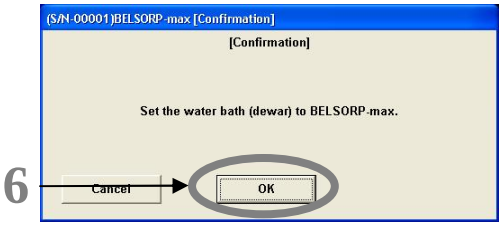


5. Install a liq. bottle according to "How to install sample cells and Liq. Bottles, P.57". When it is complete, press the OK button.



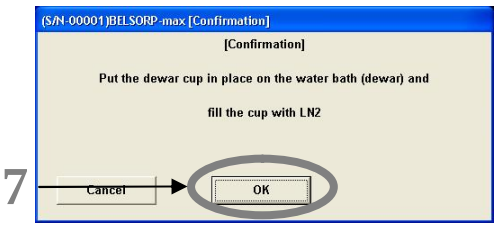
> Keep the adsorptive liquid level lower than 70% of the liq. bottle. When you perform the following degassing process with too much liquid in the bottle, the liq. bottle may be broken.

6. Install the water bath (Dewar vessel) to the instrument.



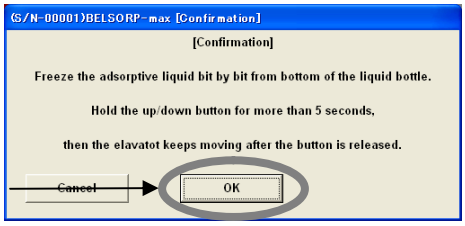
7. Fill fully the Dewar cup with liquid nitrogen. When it is complete, press the OK button.

> Careful attention is required in handling liquid nitrogen.



8. Install the Dewar cup to the top of the water bath (Dewar), and start freezing the adsorptive in the liq. bottle with liquid nitrogen. Press the lifting switch ▲ button on the front of the main unit to lift up the water bath (Dewar). If the button is depressed for more than 5 seconds, the water bath keeps moving up even the switch is released. Press the ■ button to stop it.

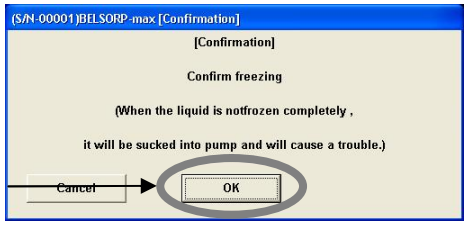
9



9. When the bottom of the liq. bottle comes into contact with the liquid nitrogen, the liquid (adsorptive) in the liq. bottle starts freezing gradually. Lift up the Water bath (Dewar) as the liquid nitrogen level drops, so that the bottom of the liq. bottle keeps contact with the liquid nitrogen. When the adsorptive in the liq. bottle has frozen completely, press the OK button.

10. Verify that the adsorptive in the liq. bottle has frozen completely, and then start vacuuming. Press the OK button. Vacuuming will start automatically (for about 5 minutes).

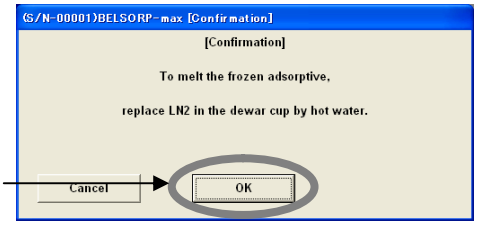
10



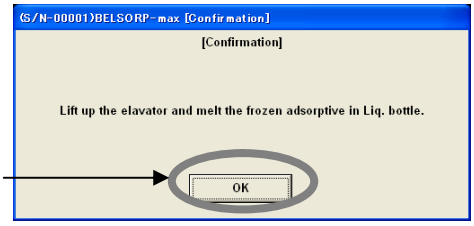
11. When vacuuming is complete, the water bath is lowered to the bottom limit.

12. Remove the liquid nitrogen in the Dewar vessel, and fill it with hot water. Install it to the top of the water bath. Press the OK button. Then a message appears as shown on the right. Lift up the water bath (Dewar) so that the frozen liq. bottle is soaked into hot water to melt the adsorptive in the bottle.

12

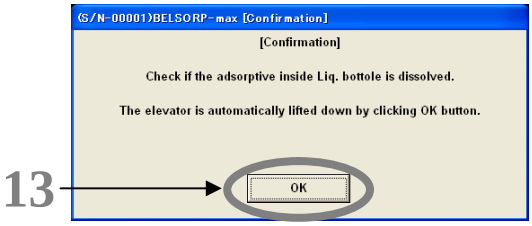


12

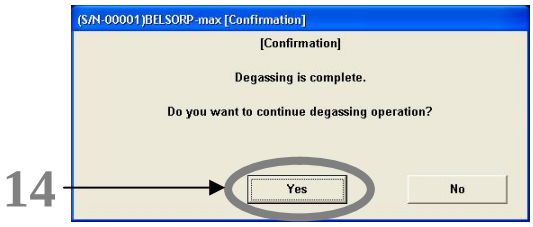


⚠ Careful attention is required in heating the liq. bottle. Heating (melting) partially a frozen liq. bottle may break it.

13. When degassing in step **12.** is complete, the "Confirmation" window appears as shown on the right. Lower the water bath (Dewar) and verify the adsorptive in the liq. bottle has melted completely. It may not have melted completely depending on the type and quantity of the adsorptive in the liq. bottle. If it is not melted completely, lift up the water bath (Dewar) and leave it for a while. It will melt completely. Then, press the OK button.



14. For obtaining accurate measurement data, repeat degassing steps **3.** to **13.** for a total of over 3 times. The degassing process is now complete.

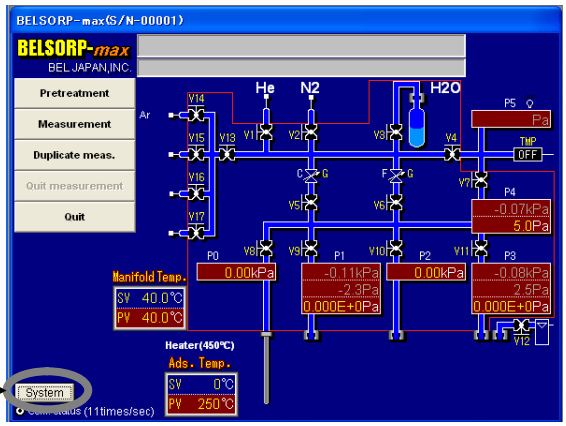


ooo Optional settings ooo

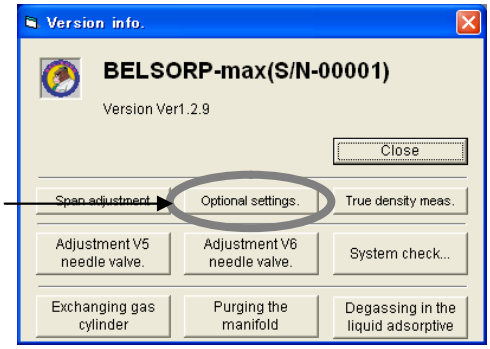
Optional settings can be specified, including the heater, electric furnace, water bath (optional), as well as standard Dewar vessel. Measurement at various adsorption temperature levels is available by using the optional heater, electric furnace, or water bath. Pretreatment (vacuum/flow gas pretreatment) is also available by using the optional heater, electric furnace, or water bath.

1. Start the BELSORP-max main unit and the measurement software.

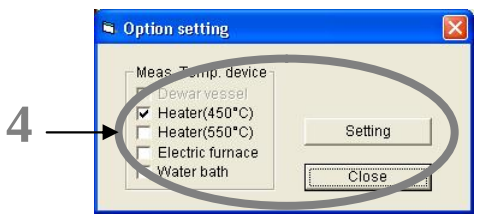
2. Press the System button on the "Flow circuit diagram" window. The "System version information" window appears.



3. Select the Optional settings button. The "Option setting" window appears.



4. Check Heater (450 °C), Heater (550 °C), Electric furnace, or Water bath to be used, and then select the Setting button. Dewar vessel shall always be checked. The heater or electric furnace can be controlled for the adsorption temperature by the software. Select the Close button to return.



ooo Measuring true density ooo

True density is obtained by quantity of solid / powder dividing effective volumetric capacity. In the case of the BEL SORP max, true density is calculated in the following way. At the first, the sample volumetric capacity is calculated by "sample weight after pre-treatment / (blank sample cell 1 −blank sample cell + sample 2)", each of volumetric capacity measured by He. BELSORP-max has a function which automatically covers various tasks from measuring the true density of a solid/powder to outputting data simply after measurement conditions are set and the sample is weighed. Measurement accuracy is 35 ± 0.007 cm^3 (= ± 0.02 %).

* Please measure enough sample weight to increase measurement accuracy.

1. General

1. Set measurement parameters, and start measuring true density.

2. Start blank Vd measurement.

To measure blank Vd:	To skip blank Vd measurement:
1) Install an empty sample cell to port 2. 2) Install the water bath and detachable thermostatic bath, raise water bath, and wait for 70 minutes until the temperature is stabilized. Then, measure blank Vd. ⚠ Set the water bath tempetrature at 40 °C in advance.	Entry values are used. Go to the pretreatment of the sample.

3. Start the pretreatment of the sample.

Start the pretreatment of the sample according to the preset pretreatment parameter. Weigh the sample, and then go to "Sample-available Vd measurement" (If skipping pretreatment, weigh the sample, and then go to "Sample-available Vd measurement".).

4. Start "Sample-available Vd measurement".

1) Install the pre-processed sample cell to port 2.

2) Install the circulating water bath and detachable thermostatic bath, raise water bath, and wait for 70 minutes until the temperature becomes stable. Then, perform "Sample-available Vd measurement".

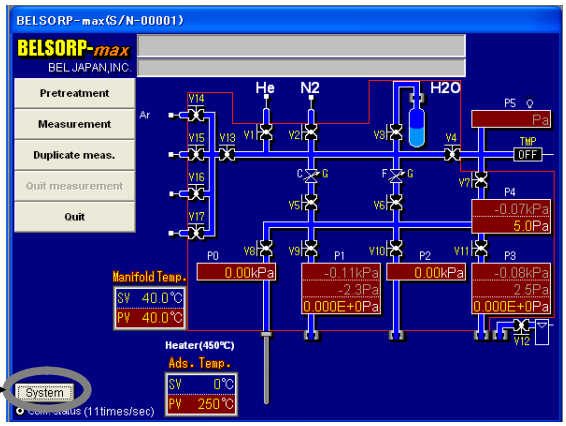
5. The data of true density measurement is saved in the specified file "…DENn.CSV".

2. Procedure of measurement

BELSORP-max's measurement software is equipped with a guidance function to ensure measurement of true density without problems. In this section, a procedure to measure true density in accordance with this guidance is described.

1. Start the BELSORP-max main unit and the measurement software.

2. Press the System button on the "Flow circuit diagram" window. The "System/Version Information" window appears.

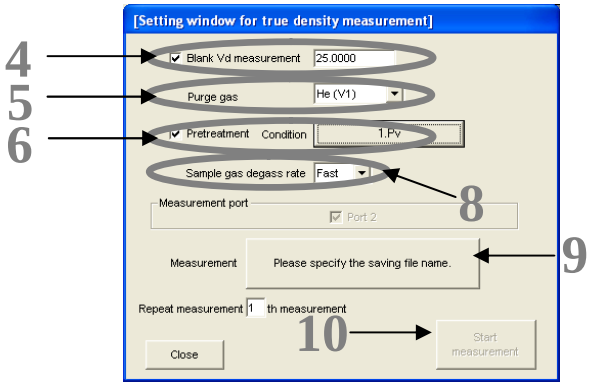


3. Select the true density measurement button. The "true density measurement parameter settings" window appears.

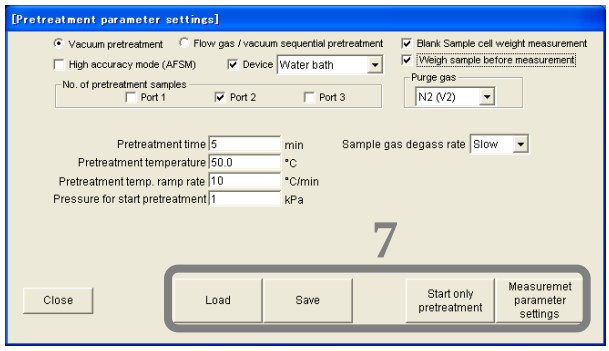
4. Check if performing "Blank Vd measurement". Fill in the measured value of the blank Vd measured in advance otherwise.

5. Select a gas to purge inside of the sample cell after the pretreatment.

6. To perform pretreatment by the main unit, check "Pretreatment" to set up parameters. By selecting the "Pretreatment parameters" button, the window appears as shown on the right. For the setup method of each item, refer to "Pretreatment on P. 111". If not checked, pretreatment is not performed. Pre-treat the sample by the optional pretreatment instrument (BELPREP-flow II/BELPREP-VAC II) as needed.

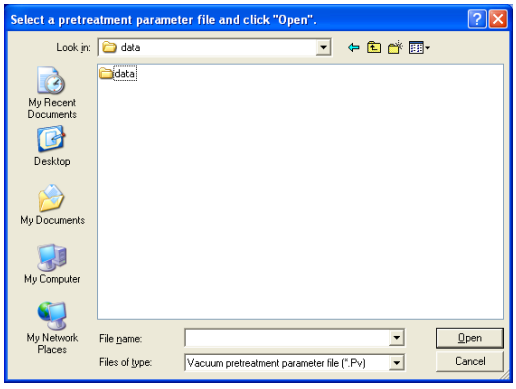


7. Load and save the “Pretreatment parameter setting” file.

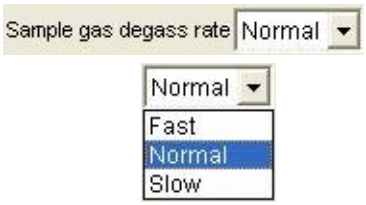


Load	Select the [Load] button to load the existing “Pretreatment parameter setting” file. However, it is necessary that the file has been saved by the [Save] button in advance. After the file has been loaded, select the [Overwrite] button to return to the screen of step 4.
Save	Select the [Save] button to save the existing “Pretreatment parameter setting” file, and return to the screen of step 4.
Overwrite	Select the [Overwrite] button to over write the “Pretreatment parameter settings” file, and return to the screen of step 4.
Delete	Select the [Delete] button to cancel the setting, and return to the screen of step 4. Set the pretreatment parameter again.

When you select the [Load] or [Save], the “Pretreatment parameter file” window appears. Specify the “location to load from” or the “location to save in”. Select or enter the “file name”, and then select the “Open” or “Save”. An extension of “.Pv” is automatically set to the vacuum pretreatment parameter file, whereas “.Pf” is set to the vacuum / flow gas sequential pretreatment parameter file.



8. Select the sample gas degassing rate using [▼] button.

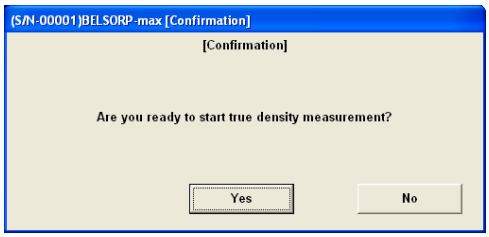


9. Specify a name of the file to save the true density measured data. Set a place to save, enter a file name, and select “Save”. “-Den” is automatically appended to the file name of the measured data, and the extension is “.CSV”.

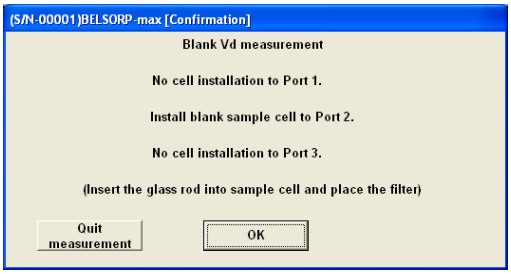
10. Select the [Start measurement] button to start measuring true density. After measurement has started, follow the window displayed.

Now, this section explains the flow when "Blank Vd measurement" and "Pretreatment" are checked.

11. Select the Start measurement button. Then the Confirmation window is displayed. By selecting Yes, true density measurement starts. Or select No to return to the "True density measurement parameter" window.

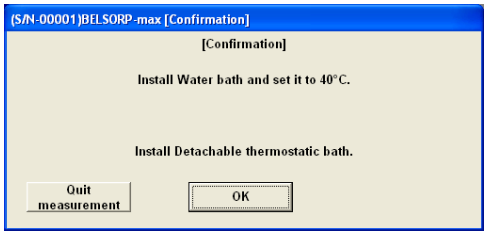


12. The window as shown on the right appears. Install the empty sample cell inserted the glass rod and attached the filter, and select the OK button. By clicking the Quit measurement button, measurement will be aborted, and return to the "True density measurement parameter" window.

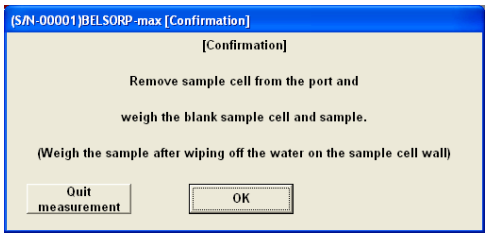


⚠ Only port 2 is used for real density measurement. Insert the glass rod into the sample cell, and attach the filter.

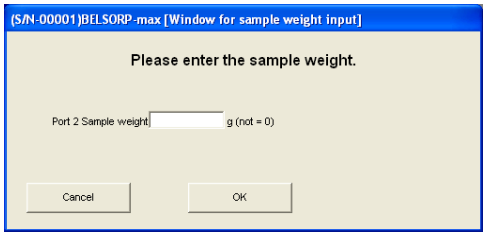
13. Install the water bath and detachable thermostatic bath, and select the the OK button. Raise the water bath, wait for 70 minutes until the temperature is stabilized, and measure blank Vd.



14. When blank Vd measurement has finished, the water bath is lowered, and the confirmation window as shown on the right appears. Weigh the empty sample cell, sample, and select the OK button. Pretreatment is performed continuously based on the pretreatment parameter which was set up at step 6. Follow the direction on the window.



15. After pretreatment, the confirmation window as shown on the right appears. Weigh the sample to enter the measured weight, and select the OK button.



16. Install the sample cell, and select the OK button. In the same manner as applied to step 13, install the water bath, wait for temperature to become stable, and start “Sample-available Vd measurement”.

17. True density measurement has finished when the message as shown on the right appears.

Measurement data

The data below are included in saved files.

Vdb / cm^3	Average value of “blank Vd”
Vds / cm^3	Average value of “sample-available Vd”
Sample weight / g	Mass entered at step **15**
Sample density / g cm^{-3}	True density = measurement result

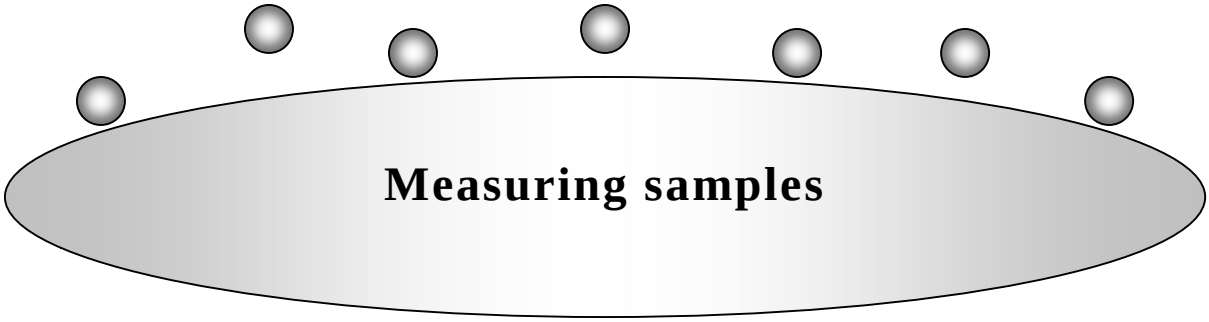
Blank (Vs+Vd)

Pressure	55kPa	60kPa	65kPa	70kPa	75kPa	80kPa	85kPa	90kPa	95kPa	100kPa	105kPa	110kPa
Pen-1	47.48773235	52.17401389	57.47903019	62.21737589	68.09753316	72.61931541	76.95979541	82.67575961	86.81645808	91.57879761	97.25142374	101.5880652
Pe2n-1	47.51298125	52.20247784	57.51183662	62.25657263	68.13766875	72.66048649	77.00519292	82.72450319	86.87111845	91.63316167	97.30688715	101.6486878
Ten-1	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15
Te2n-1	313.15	313.15	313.1691781	313.15	313.15	313.15	313.15	313.15	313.15	313.1705479	313.15	313.15
Pin	54.1169063	59.6847047	64.19357017	70.5352276	74.50459784	78.77555034	85.06354404	88.56623963	93.57895555	99.62817633	103.4152358	108.6791101
Pen	52.17401389	57.47903019	62.21737589	68.09753316	72.61931541	76.95979541	82.67575961	86.81645808	91.57879761	97.25142374	101.5880652	106.5757465
Pe2n	52.20247784	57.51183662	62.25657263	68.13766875	72.66048649	77.00519292	82.72450319	86.87111845	91.63316167	97.30688715	101.6486878	106.6397401
Tin	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15
Ten	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15
Te2n	313.15	313.1691781	313.15	313.15	313.15	313.15	313.15	313.15	313.1705479	313.15	313.15	313.15
Vdb	35.27679222	35.30689097	35.31885333	35.24407926	35.24809881	35.26781583	35.2916542	35.3169899	35.25960807	35.3083075	35.26026947	35.28564422

Sample (Vs+Vd)

Pressure	55kPa	60kPa	65kPa	70kPa	75kPa	80kPa	85kPa	90kPa	95kPa	100kPa	105kPa	110kPa
Pen-1	47.31250197	53.49853531	57.82670008	62.731024	67.18962473	72.90189393	77.33204788	81.94042761	87.61540735	92.00835602	96.50045897	101.5292148
Pe2n-1	47.34136762	53.52646583	57.85935918	62.76634255	67.22863302	72.94478541	77.37969828	81.99019659	87.66824113	92.06327101	96.55921958	101.5889738
Ten-1	313.15	313.16	313.15	313.15	313.15	313.17	313.15	313.15	313.15	313.15	313.15	313.15
Te2n-1	313.1697183	313.15	313.15	313.15	313.1791667	313.15	313.15	313.15	313.15	313.15	313.15	313.15
Pin	56.04532582	59.61786778	64.76181297	69.03822582	75.26028244	79.17982275	83.85958904	89.9791195	93.84606959	98.38622097	103.6285905	108.6235286
Pen	53.49853531	57.82670008	62.731024	67.18962473	72.90189393	77.33204788	81.94042761	87.61540735	92.00835602	96.50045897	101.5292148	106.5302456
Pe2n	53.52646583	57.85935918	62.76634255	67.22863302	72.94478541	77.37969828	81.99019659	87.66824113	92.06327101	96.55921958	101.5889738	106.5924504
Tin	313.1866197	313.15	313.15	313.15	313.1543478	313.15	313.15	313.15	313.15	313.15	313.15	313.15
Ten	313.16	313.15	313.15	313.15	313.17	313.15	313.15	313.15	313.15	313.15	313.15	313.15
Te2n	313.15	313.15	313.15	313.1791667	313.15	313.15	313.15	313.15	313.15	313.15	313.15	313.15
Vds	35.21774427	35.21741023	35.23219405	35.21394756	35.24905503	35.19487274	35.19935896	35.24027135	35.20424569	35.23590031	35.19131936	35.21213978

Vdb	35.28208365	cm3		Sample weight	0.50019	g
Vds	35.21737161	cm3		Sample density	7.730601317	g/cm3



Measuring samples

Measuring with BELSORP-max

ooo For accurate adsorption isotherm ooo

BELSORP-max is a high precision automatic gas-adsorption measuring unit that uses the volumetric method. By the volumetric method, the adsorption to the sample is determined from the adsorptive gas pressure difference using the state equation of gas. Anyone can obtain accurately and quickly, fine particle properties by easy operation, including the specific surface area and the pore size distribution. To obtain data accurately, your careful attention is required for the following points.

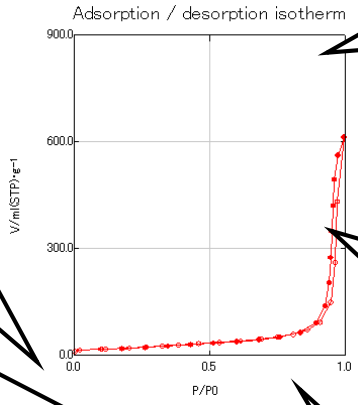
Appropriate amount of samples

The adsorption is determined from the pressure difference due to adsorption. Accurate data cannot be obtained from small amount of adsorption and small pressure difference. Conversely, too much amount of adsorption requires repeated dosing and exhaust, and measurement takes a long time. It is recommended that the **total surface area of the sample be 2 to 40**

No leakage from stop valves or outside

The measurement system keeps negative pressure during adsorption measurement. Accurate data cannot be obtained if there are any leakages at the sample cell connection port from outside. **When you install the sample cell, pay careful attention not to press any foreign materials onto the o-ring.** When the sample scattered in the measurement system adheres to the stop valve, it may result in leakage. **Be sure to use a filter during measurement.**

For accurate adsorption isotherm



Proper sample pretreatment

Correct data cannot be obtained when gas or moisture remains on the sample.

Perform sample pretreatment with the appropriate conditions. Measure the sample mass accurately after pretreatment.

Accurate pressure

The measurement system pressure is measured using a precision pressure sensor and a high resolution A/D converter.

Accurate measurement system volume

The reference volume *Vs* and the dead volume *Vd* are determined with accuracy within 0.1 %.

Appropriate judgment on equilibrium

Accurate isotherm cannot be obtained when the measurement time is too short to reach the adsorption equilibrium. Generally, non-porous samples reach the adsorption equilibrium in 10 minutes at medium or less relative pressure. However, such samples with molecular-sized pores may not reach the adsorption equilibrium even after an extended period. **Judge the equilibrium appropriately for each sample.**

Accurate measurement system temperature

The system reads the temperature at measurement points, and applies temperature correction.

ooo Outline of the measurement ooo

With BELSORP-max, an automatic gas adsorption measurement unit, measurements up to 3 samples can be performed in parallel.

		(AFSM mode)	(Standard mode)
		Measurement of 1 to 2 samples	**Measurement of 1 to 3 samples**
Dead volume change measurement		Yes	No
Measurement port	Port 1	Sample measurement	Sample measurement
	Port 2	Dead volume change measurement	Sample measurement
	Port 3	Sample measurement	Sample measurement
Saturation vapor pressure correction		For saturation vapor pressure, select either "Actual measurement at P0 port" or "Input" mode.	
Dead volume correction		Measurement at Port 2	Refer to the existing DVd file or no dead volume correction

DVd file (data file for dead volume correction)

When you control the measurement temperature using a refrigerant filled in the Dewar vessel, such as liquid nitrogen and liquid argon, the refrigerant level in the Dewar vessel tends to drop due to its evaporation. The dead volume (*Vd*) in the sample cell decreases as the refrigerant level drops; therefore, it is necessary to correct the dead volume change during measurement. When you perform measurement of the dead volume correction using a dead volume reference cell, the data of "ΔVd (ratio of dead volume change) to t (time)" can be saved in the DVd file (data file for dead volume correction). Specify the file name to which the data is saved at "setting the measurement parameter". When you do not perform measurement of the dead volume, the dead volume is corrected by referring to the data file for the dead volume correction that has been measured in advance. Specify the file name from which the data is loaded at "setting the measurement parameter".

ooo Outline of the measurement (low-pressure measurement) ooo

For low-pressure measurements, measurements up to 2 samples can be performed in parallel, using the low-pressure pressure gauge Port 1 (optional) and Port 3.

		(AFSM mode)	(Standard mode)
		Measurement of 1 to 2 samples	**Measurement of 1 to 2 samples**
Dead volume change measurement		Yes	No
Measurement port	Port 1	Sample measurement (optional)	Sample measurement (optional)
	Port 2	Dead volume change measurement	-
	Port 3	Sample measurement	Sample measurement
Saturation vapor pressure correction		For saturation vapor pressure, select either “Actual measurement at P0 port” or “Input” mode.	
Dead volume correction		Measurement at Port 2	Refer to the existing DVd file, or no dead volume correction.

Low-pressure measurement

For adsorption measurement at low pressure, it is necessary to evacuate the sample sufficiently. BELSORP-max performs sufficient evacuation at “Leak check”. A change in the lower pressure section must be detected using a pressure gauge during measurement; therefore, use the low-pressure specification ports (Port 3 and Port 1) for the measurement.

ooo Measurement sequence ooo

The saturation vapor pressure is measured at P0 port.		An input value is used as the saturation vapor pressure.	
With dead volume change measurement	**Without dead volume change measurement**	**With dead volume change measurement**	**Without dead volume change measurement**

1. Sample pretreatment

Perform the pretreatment under such conditions that the gas and moisture adsorbed on the surface can be removed without denaturalizing the sample. Measure the sample mass after pretreatment.

For the pretreatment with BELSORP-max, refer to "Pretreatment, P. 122".

Pretreatment can be performed independently by using optional pretreatment unit, BELPREP-flow II or BELPREP-vac II.

2. Starting the BELSORP-max main unit and the measurement software

Refer to "Starting the BELSORP-max main unit and the measurement software, P. 120".

3. Measurement parameter setting

Refer to "Setting the measurement parameter, P. 128".

4. Starting the automatic sample measurement (system initialization)

Press the Start measurement button on the "Measurement parameter settings" window to start measurement.

5. Preparing the Dewar vessel (or, optional heater, electric furnace, or water bath), and installing the sample cell

Prepare the Dewar vessel and install the sample cell when a window appears showing "【Procedure 1】… to the Dewar vessel (or, the heater, electric furnace or water bath)…, 【Procedure 2】… to Port 1….". Refer to "How to install sample cells …, P. 57". Press the OK button. Then the measurement system is evacuated, and the automatic measurement starts. The gas pressure change and the adsorption isotherm are displayed on the screen during the sample measurement. Refer to "Windows during measurements, P. 159".

6. Leak check

When "Leak check" is selected, the sample cell is evacuated repeatedly while checking the pressure in the sample section until it reaches appropriate condition for measurements.

7. Measurement of the dead volume in the sample cell (room temperature): "when the dead volume measurement before adsorption measurement is selected"

When "non-ideality correction" is selected, the dead volume in the sample cell at room temperature is measured using helium gas. After the measurement, about 100 kPa of helium is dosed to the sample cell.

8. Dead volume measurement at the saturation vapor pressure P0 port (room temperature)

The dead volume in the saturation vapor pressure P0 port (room temperature) is measured using nitrogen. After the measurement, about 100 kPa of nitrogen is dosed to the saturation vapor pressure P0 port.

9. Dead volume measurement in the dead volume reference cell (room temperature) The dead volume in the dead volume reference cell at room temperature is measured using helium gas. After the measurement, about 100 kPa of helium is dosed to the dead volume reference cell. When the adsorptive gas is nitrogen and the adsorptive temperature is 77 K to 78 K, or when the adsorptive gas is argon and the adsorptive temperature is 77 K to 78 K or 87 K to 88 K, the adsorptive gas is used for the measurement.		**9. Dead volume measurement in the dead volume reference cell (room temperature)** The dead volume in the dead volume reference cell at room temperature is measured using helium gas. After the measurement, about 100 kPa of helium is dosed to the dead volume reference cell. When the adsorptive gas is nitrogen and the adsorptive temperature is 77 K to 78K, or when the adsorptive gas is argon and the adsorptive temperature is 77 K to 78 K or 87 K to 88 K, the adsorptive gas is used for the measurement.	

10. Setting up the Dewar vessel (or, optional heater, electric furnace, or water bath)

The Dewar vessel moves up automatically, and the sample cell is soaked into liquid nitrogen or argon.

⚠ Do not place any foreign material on the Dewar vessel.
⚠ Liquid nitrogen may spill out sometimes. Careful attention is required.

11. Measurement of the dead volume in the sample cell (adsorption temperature): "when selecting the dead volume measurement before the adsorption measurement"

The dead volumes in each sample cell at adsorption temperatures are measured using helium gas. Refer to "Dead volume measurement, P. 15". After the measurement, helium in the sample cell is degassed.

12. Condensing adsorptive gas in the saturation vapor pressure P0 port When nitrogen or argon is used, the adsorptive gas may be condensed. Refer to "Saturation vapor pressure measurement, P. 18".	

13. Adsorption measurement

Dosing and adsorption are repeated until the equilibrium pressure reaches the target adsorption pressure. Refer to ”Adsorption measurement and desorption measurement, P. 17”. The data is saved in the relevant file specified for each measurement point. When all the target adsorption pressure values are cleared, it proceeds to the desorption measurement.

14. Desorption measurement

Depressurizing and desorption are repeated until the equilibrium pressure reaches the target desorption pressure. Refer to ”Adsorption measurement and desorption measurement, P. 17”. The data is saved in the relevant file specified for each measurement point. When all the target desorption pressure values are cleared, it proceeds to the next step.

15. Duplicate measurement

When you start “Duplicate measurement, P. 156”, it performs the degassing process (to evacuate the sample for the time specified), and then performs the desorption measurement as many times as specified.

16. After the measurement

When the dead volume measurement “after the adsorption/desorption measurement” is selected, it performs the following measurements.

Dead volume measurement in the sample cell (adsorption temperature)

Dead volume measurement in the sample cell (room temperature): when “non-ideality correction” is selected.

17. Post-treatment

The measurement system is degassed, and then the Dewar vessel moves down. It performs purging and enters the weight according to the settings.

18. Exiting the measurement software and shutdown of BELSORP-max

Refer to “Exiting the measurement software and shutdown of BELSORP-max, P. 166”.

⚠ **When the liquid nitrogen level drops below the sample section or the saturation vapor pressure P0 port, the adsorbed or condensed nitrogen or argon on them may evaporate and generate higher pressure than atmospheric pressure. This may not only cause an error of the measurement data, but also may result in damage to the instrument since it raises pressure in the glass tube, and over time the glass tube is detached from the instrument connection port. This measurement software aborts the sample measurement and degasses the system automatically, when the measurement port pressure exceeds 130 kPa, or the saturation vapor pressure measurement port pressure exceeds 110 kPa. Careful attention is required when you perform measurements for 60 hours or more (For low-pressure measurement, the software manages the time elapsed, and performs the specified post-treatment (post-dead volume measurement, etc.) forcedly, when the measurement lasts 60 hours or more.) .**

ooo Starting the BELSORP-max main unit and the measurement software ooo

1. Starting the BELSORP-max main unit

1. Turn on the power switch on the back of the unit. The power indicator on the front of the main unit lights on.
2. Turn on the rotary pump.
3. When the instrument has not been used for a long time period, refer to “Installing and replacing gas cylinders, P. 90”.

Caution

Turn on the instrument first, and then turn on the pump.

- **When power is supplied to the rotary pump from an external power supply, and the rotary pump is turned on with the instrument turned off, air may be sucked from a vent valve opened. This may result in overheat or failure of the pump.**

2. Starting the measurement software

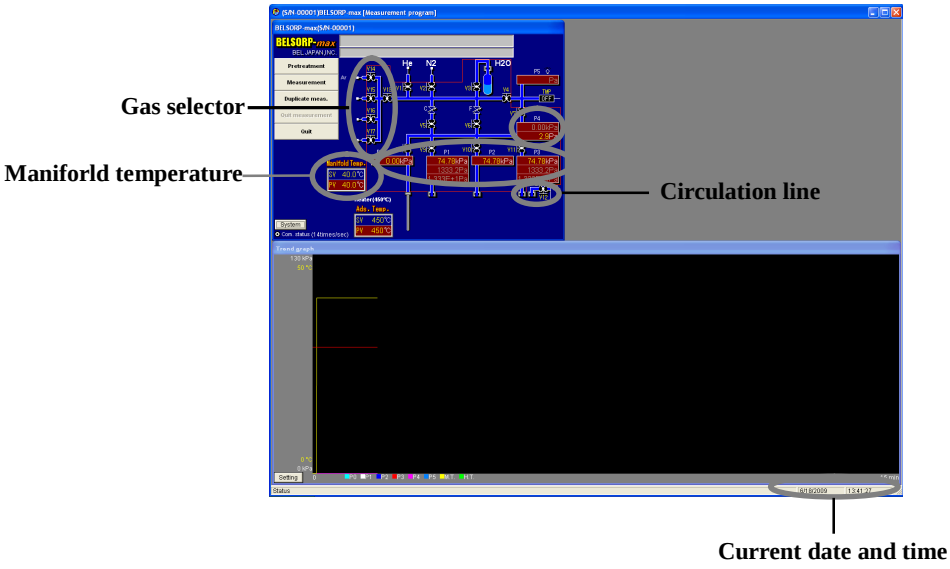
1. Verify that the main unit and the personal computer are connected properly using a connection cable.
2. Check that power is supplied to the instrument.
3. Turn on the personal computer, and start “Windows”.
4. Select “Start” > “Program” menu > “BELmax” > “BELmax”.

5. The measurement software starts, and the “Main” window appears. When the current status is displayed for the following items, startup is complete (The screens shown below are those in the Windows XP mode.).

・Current date and time

・Pressure gauge (P0 to P4)

・Manifold temperature (TIC1)



The following items are displayed when you select them as options.

・Gas selector (V13 to V17)

・Flow gas pretreatment line (V12)

6. In the event of error in the communication line, including such cases that the main unit and the personal computer are not connected properly using a connection cable, the “Communication error” window is displayed. Check the power supply to the main unit and the connection.

* If the valves (V1 to V10) still remain as opened manually when the software was used previously, all valves close when the software starts.

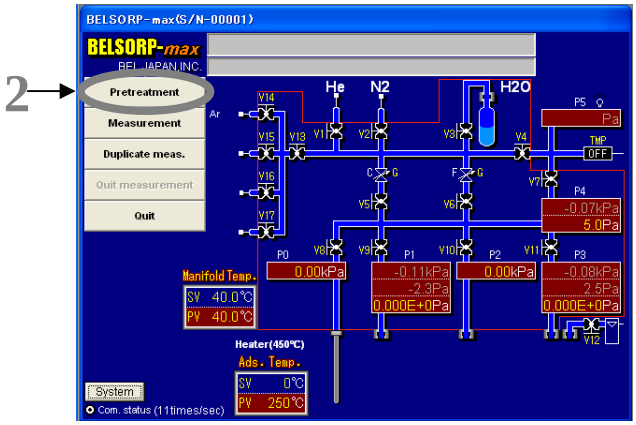
ooo Pretreatment ooo

Before you start measurements with BELSORP-max, perform weighing and pretreatment of the sample to be measured. [When you perform pretreatment of the sample with this instrument (the software requests you to select "weighing samples before the measurement" or "weighing samples after the measurement"), a message related to weighing samples appears. Follow the instructions in the message. When you perform pretreatment of the sample with an optional pretreatment device (BELPREP-flow II, BELPREP-VAC II), weigh samples independently.]

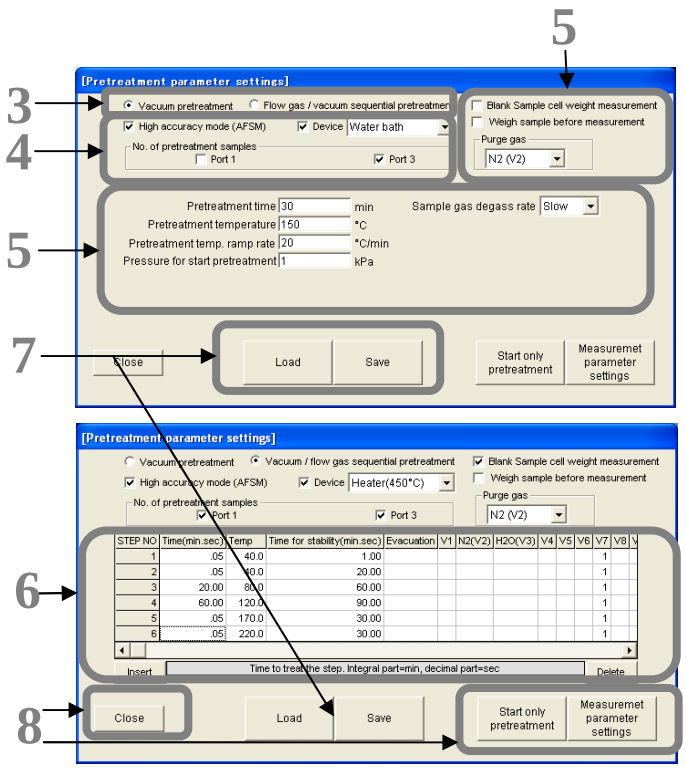
This manual describes the sample pretreatment with this instrument (at pretreatment temperature using the heater, electric furnace, or water bath).

1. Start the BELSORP-max main unit and the measurement software.

2. Select the Pretreatment button on the "Flow circuit diagram" window.



3. The Pretreatment parameter settings window appears. Select either Vacuum pretreatment or Vacuum/flow gas sequential pretreatment. The flow gas sequential pretreatment can be performed only when the flow gas pretreatment line is provided.

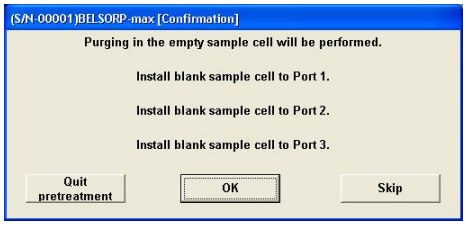


4. Specify the AFSM, device, and pretreatment samples.

AFSM	When you measure with a dead volume reference cell, check AFSM (Port 2 is used for the reference cell.).
Device	When you use an adsorption temperature device, check Device and select the device using ▼ button. Setting for the device shall be in accordance with "Optional settings, P. 107".
Pretreatment samples Port 1 to 3	For the sample cell to be used, check Port 1, Port 2, or Port 3. The port used for the dead volume change measurement is not displayed, and cannot be selected. Check at least one of them.

5. When the vacuum pretreatment is selected, specify the following items.

Pretreatment time	Enter the pretreatment time in minutes (contein none of rump time).
Pretreatment temperature	Enter the pretreatment temperature in °C. Specify the temperature from the personal computer when the device is the heater or electric furnace.
Pretretment temp. ramp. rate	Enter the pretreatment temp. ramp. rate in °C /min^{-1} . 450 ^{o}C / 550 ^{o}C heater: Setting pretreatment temp. ramp. rate 1-20 ^{o}C min^{-1} Electric furnance (- 1000 ^{o}C) : Setting pretreatment temp. ramp. rate 1-100 ^{o}C After heating enable to pressure attainment, first heated to "(Pretretment temperature) – (Pretretment temp.ramp. rate)", and then heated to "Pretretment temperature"
Heating enable to pressure	Enter the pressure to start heating in kPa. When the pressure drops below the specified level, the device moves up and it starts heating. Setting for the device shall be in accordance with "Optional settings, P. 107". ⚠ Please note that if there is a possibility of rapid heating to sccatter.
Sample gas degassing rate	Select the appropriate sample gas degassing rate using ▼ button. Select "Slow" to degas slowly to prevent the sample from scattering. Select "Normal" to degas stepwise to prevent the sample from scattering. Select "Fast" to degas quickly.
Weighing samples before the measurement	Check "Weigh sample before measurement" to weigh the sample after pretreatment. When checked, a window appears to prompt you to weigh after the pretreatment.

Blank sample cell weight measurement	Check this when you measure the blank sample cell weight. When you check it, a window appears to prompt you to install a blank sample cell. Once installed, it evacuates the sample cell in the gas purging process. At the evacuation, a window appears as shown on the right. When you mistakenly install the sample cell with a sample in it, select Quit pretreatment or Skip. Press OK to start evacuating the blank sample cell. 
Purging after the measurement	Check this to purge after the measurement. Select the purge gas using ▼ button. It purges when weighing the blank cell and the sample. ⚠ Warning: Do not purge with dangerous gas.

6. When selecting the flow gas / vacuum pretreatment, specify the following items.

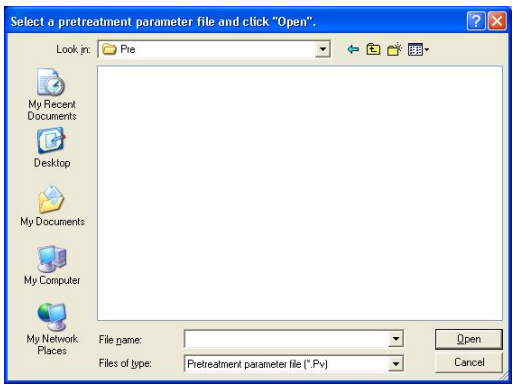
Time (min., sec.)	Specify the treatment time required for this step. The integer part represents the time in minutes and the decimal part represents the time in seconds. The sum of the minutes and seconds is the treatment time in this step.
Target temperature	Specify the temperature to complete this step (when you use the heater or electric furnace that can be controlled from the personal computer). When it does not reach the target temperature in the time specified, it waits until it reaches the target temperature ±2 °C.
Time for stability (min., sec.)	Specify the waiting time for stability after the treatment time (min., sec.) is over. The integer part represents the time in minutes and the decimal part represents the time in seconds. The sum of the minutes and seconds is the time for stability.
Evacuation	Specify the evacuation. Set to “0” not to evacuate, “1” to evacuate quickly, and “2” to evacuate slowly.
V1 to 17	Specify the valve open/closed. Set to “0” to close, and “1” to open. Only a single valve can be opened, out of V1 to V3, and V14 to V17. Otherwise, gases are mixed. After closed, the gas accumulator is exhausted for 30 seconds.
Device	Specify the temperature device switch. Set to “1” to turn on the switch, which enables to set the target temperature. Set to “2” to turn off the switch.
FAN	Specify the fan when the electric furnace is selected as the temperature device. Set to “1” to turn on the fan, which cools down the electric furnace rapidly. Set to “0” to turn off the fan.
UP	Specify to move up the elevator. Set to “1” to move it up. Set to “0” not to move it.
DN	Specify to move down the elevator. Set to “1” to move it down. Set to “0” not to move it.

WRG	Specify the indication of the vacuum gauge P5. Set to “1” to indicate. Set to “0” not to indicate.
TMP	Specify the turbo molecular pump. Set to “1” to start rotating. Set to “0” to stop rotating.
Insert button	This is used to insert a line to the line where a cursor is placed. The texts on the line where a cursor is placed are copied onto the line inserted. All the lines below the line where a cursor is placed are shifted a line down.
Delete button	This is used to delete a line where a cursor is placed. All the lines below the line deleted are shifted a line up.

7. Load and save the “pretreatment parameter settings” file, where applicable.

Load	Select the Load button to load the existing “pretreatment parameter setting” file. The file must have been saved using the Save button.
Save	Select the Save button to save the pretreatment parameter settings to the relevant file.

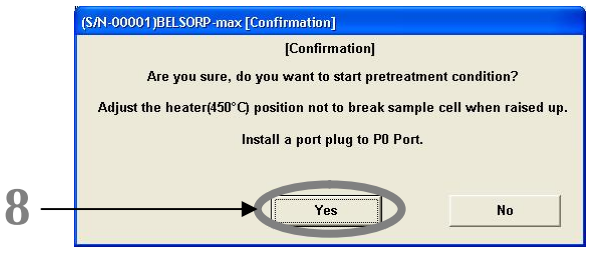
When selecting Load or Save, the “Pretreatment parameter file” window appears. Specify the “location to load from” or the “location to save in”. Select or enter the “File name”, and then select “Open” or “Save”. An extension of “.Pv” is automatically set to the vacuum pretreatment parameter file, whereas “.Pf” is set to the vacuum / flow gas sequential pretreatment parameter setting file. For the rules to enter a file name, refer to the Windows instruction manual.



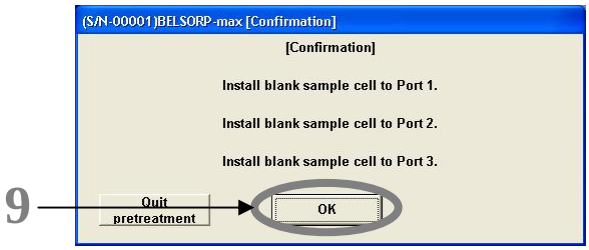
8. Select the Close button to cancel the settings and finishi the pretreatment parameter setting.

Select the Measurement parameter settings button. Then the “Measurement parameter settings” window appears. Parameter setting can be performed in sequence from the pretreatment to the measurement (refer to “Setting the measurement parameter, P. 128) .

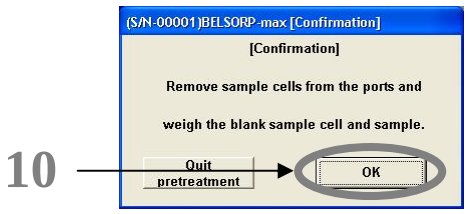
Select the Start only pretreatment button. Then the “Confirmation” window as shown on the right appears. Verify that the sample cell has been removed, and then select the Yes button to start the pretreatment. Select the No button to return to the “Pretreatment parameter setting” window. Once the pretreatment starts, follow the instructions on the window.



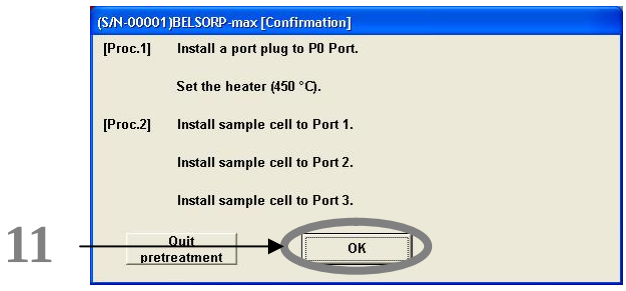
9. Install a blank sample cell to the relevant port, and press the OK button. Then, it evacuates and purges (This is when selecting “weigh samples before the measurement” or “weigh samples after the measurement”. Proceed to step 11, when not selected.).



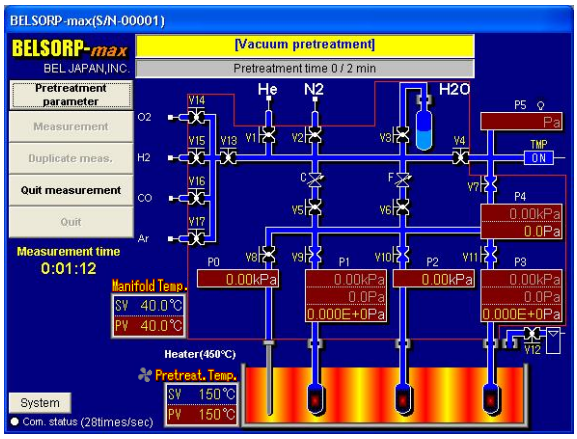
10. Remove the sample cell from the relevant port, weigh the blank cell and the sample, and then press the OK button.



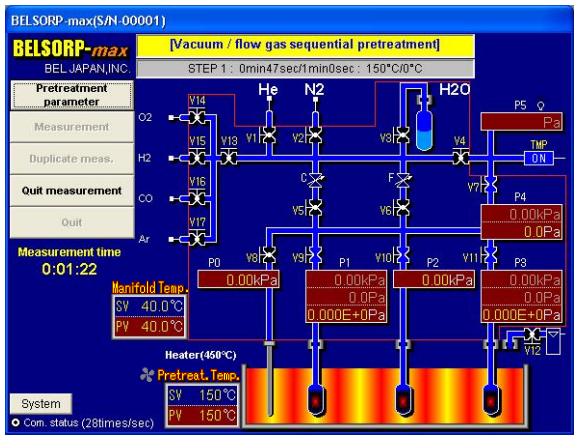
11. When using a device, install the relevant device. Specify the pretreatment temperature for the device that cannot be controlled from the personal computer. Install the sample cell that has been weighed to the relevant port, and press the OK button.



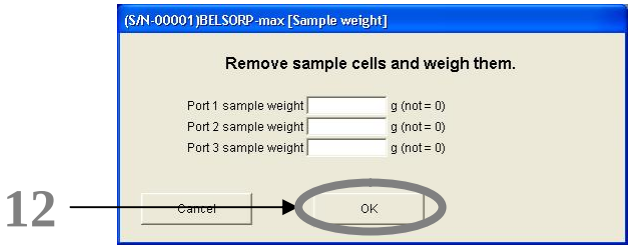
Vacuum pretreatment



Vacuum / flow gas sequential pretreatment



12. When “Weigh samples before the measurement”is checked, the system purges after the pretreatment, and then displays the “Sample weight” window. Weigh the sample, enter the measured weight, and press the OK button.



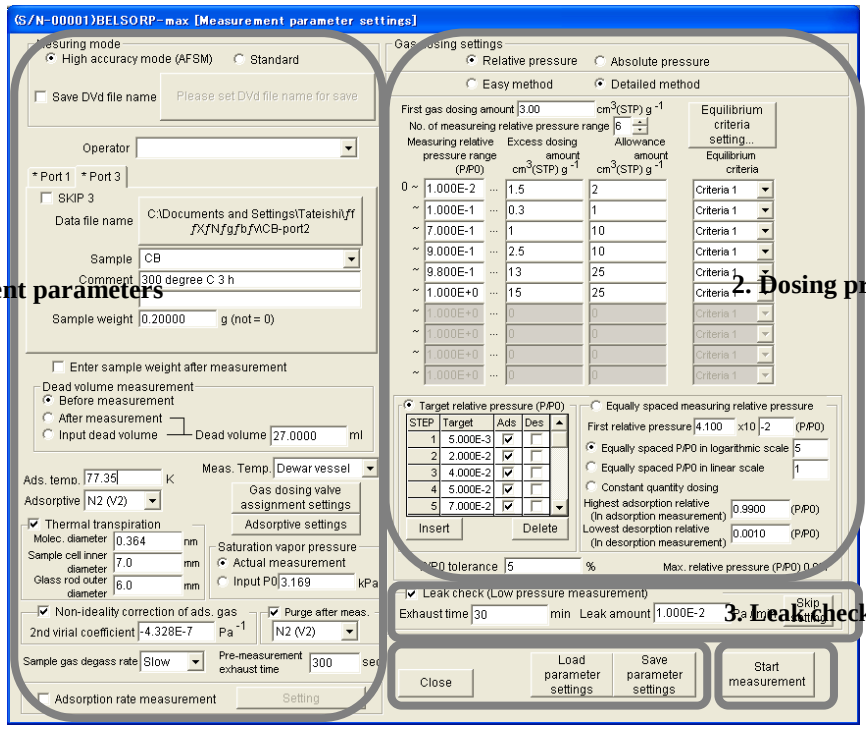
13. When Only start pretreatment button is selected in step **8.**, it finishis the sequence. When Measurement parameter settings button is selected, it proceeds to the measurement. Follow the instructions on the screen.

ooo Setting the measurement parameter ooo

1. Start the measurement software, and select the Measurement button on the "Main" window. It is not necessary to select the Measurement button, when you navigate from "Pretreatment, P. 122" or "Duplicate measurement, P. 156".

2. The "Measurement parameter settings" window appears as follows. Specify the conditions related to the adsorption measurement for the sample.

⚠ Remove the sample cell from the measurement port.

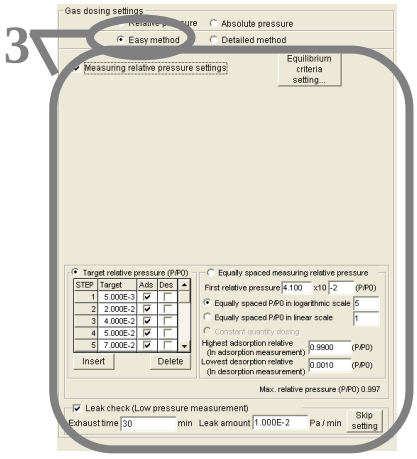


1. Measurement parameters **2. Dosing pressure settings**

3. Leak check parameter setting

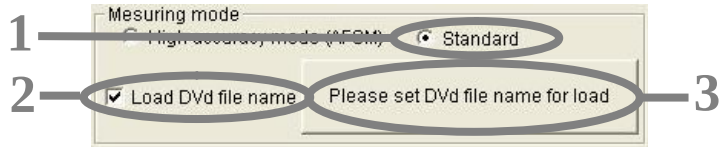
4. Load / Save **5. Start measurement**

3. When you select "Easy method" in "2 . Dosing pressure setting", the screen changes as shown on the right.



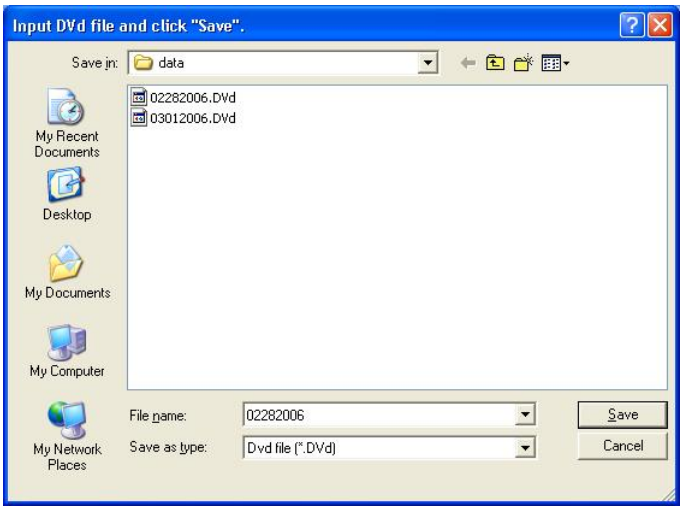
1. Measurement parameters

1. Select the measuring mode.

High accuracy (AFSM)	 Select this mode when you measure the dead volume using a dead volume reference cell. The dead volume changes as the liquid nitrogen level changes. The dead volume data that was measured using a dead volume reference cell can be saved in the relevant file (This mode is effective for the low-pressure measurement using liquid nitrogen; however, it is not necessary for the measurements using the water bath or heater.). These files offer the dead value reference data to the subsequent sample measurement. To save the measured Save DVd file name dead volume data, check and specify the "DVd file name for save".
Standard	 Select this mode when you do not measure the dead volume using a dead volume reference cell. The dead volume changes over time during sample measurement. Therefore, dead volume reference data is required when you do not measure the dead volume using a dead volume reference cell. To refer to the file for the measured dead volume data, check Load DVd file name and specify the dead volume reference data for the "DVd file name for load". At this time, select the dead volume data that was measured using a dead volume reference cell with the same conditions as those for the sample cell to be measured.

2. When [DVd file] is checked, select [Load] to refer to the file for the dead volume data, or select [Save] to save the dead volume data measured using a dead volume reference cell to the relevant file. Once [DVd file] is unchecked, [Load] or [Save] from/to the file is disabled.

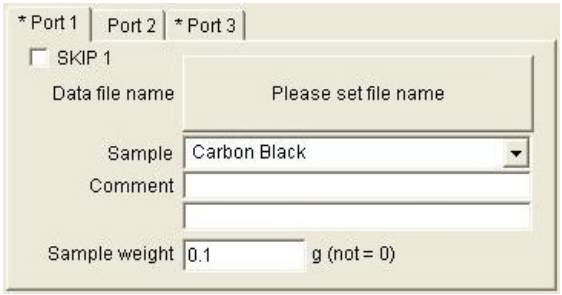
3. Select the Please set DVd file name for save button. Then the "Save DVd file" window appears.



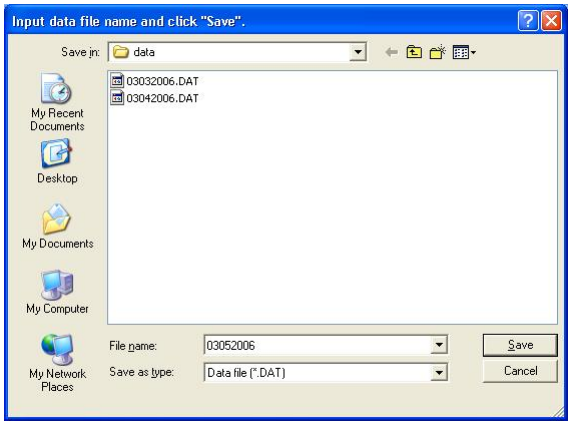
4. Specify the "location to load from" or the "location to save in". Select or enter the "File name", and then select "Save". An extension of ".DVd" is automatically set to the DVd file.
For the rules to enter a file name, refer to the Windows instruction manual.

5. Specify the operator name. 20 operator names can be registered at the maximum.

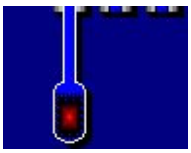
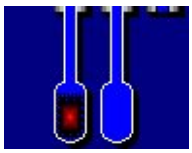
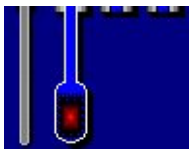
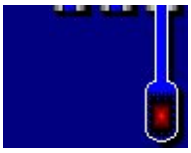
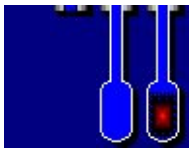
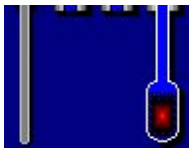
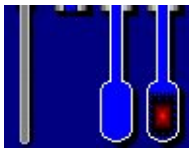
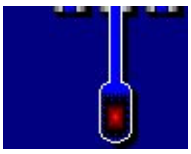
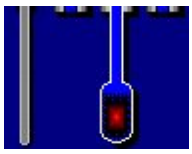
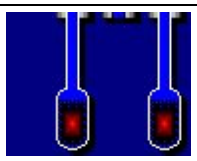
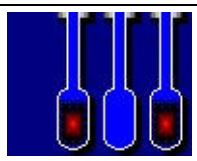
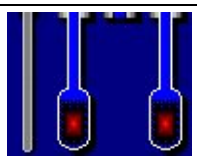
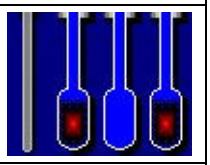
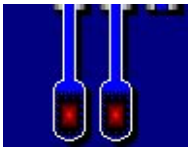
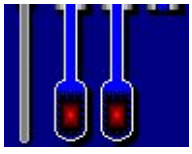
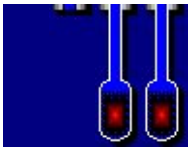
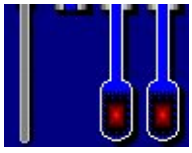
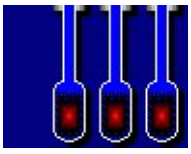
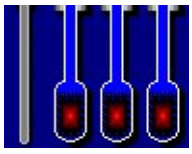
6. Specify the relevant items for each measurement port (Port 1 to Port 3). Port 2 is used (to measure the dead volume) when the AFSM measuring mode is selected; therefore, Port 2 is not displayed.
When "Leak check (low pressure measurement)" is checked, Port 2 is not used for the low-pressure measurement and it is not available for setting.

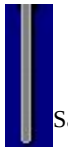


7. Specify the relevant items for the sample to be measured.

SKIP	Uncheck this when the relevant port is used for the measurement. When checked, the relevant port is not available. Items of “Data file name” or below are not displayed. The “ * ” mark is attached to the head of the port number to be used.
Measurement data file name	Select the [Please set file name] button.  The “Save file” window appears. Specify the [location to save in], enter the [file name], and select the [Save] button. An extension of “.DAT” is automatically set to the file. For the rules to enter a file name, refer to the Windows instruction manual.
Sample name	Enter the sample name. Press the [▼] button to display the sample name list entered previously, and select the sample name from them.
Operator name	Enter the operator name. Press the [▼] button to display the operator name list entered previously, and select the operator name from them.
Comment	Enter textual information related to the sample. 200 half-sized characters are accepted at the maximum.
Sample weight (g)	Enter the sample weight measured. When “Weigh sample after measurement” is cheeked, enter an approximate value.

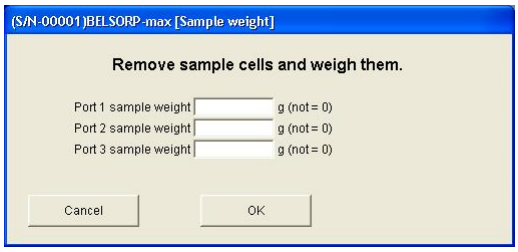
8. Each port is used as combined as follows.

Number of samples to be measured	Low pressure measurement	Port P0 1 2 3	Port P0 1 2 3	Port P0 1 2 3	Port P0 1 2 3
				Saturation vapor pressure measurement using P0 port	Saturation vapor pressure measurement using P0 port
			Dead volume measurement using Port 2		Dead volume measurement using Port 2
1 sample	○				
	○				
	×				
2 samples	○				
	×				
	×				
3 samples	×				

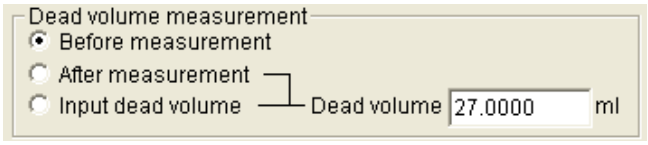
 Sample cell  Dead volume cell  Saturation vapor pressure P0 port

9. Check “Enter sample weight after measurement” where 

applicable.

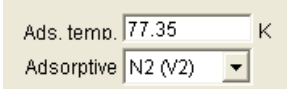
Enter sample weight after measurement	When checked, the “Enter sample weight after measurement” window appears after post-treatment purging after measurement. Enter the sample weight to the relevant port, and select the OK button. The system performs purging without exception; therefore, specify the purge gas used for post-treatment purging. 

10. Select “Before measurement”, “After measurement” or “Input dead volume” for the Dead volume measurement. When “After measurement” or “Input dead volume” is selected, enter the dead volume. During measurement, the adsorption is determined using the input dead volume.



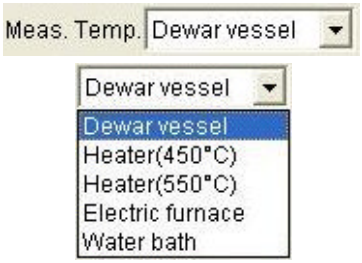
Before measurement	Select “Before measurement” to measure the sample dead volume before adsorption measurement.
After measurement	Select “After measurement” to measure the sample dead volume after adsorption measurement using the “dead volume” entered to the right of the above screen. Then, the adsorption is re-calculated. The nitrogen holding time in a fully filled Dewar vessel is 60 hours; therefore, the system automatically stops the measurement and measures the dead volume when the measurement time exceeds 60 hours, when you select “After measurement”.
Input dead volume	When “Input dead volume” is selected, the sample dead volume is not measured. Enter the dead volume.

11. Specify the adsorption conditions.

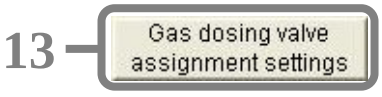


Adsorption temperature (K)	Enter the adsorption temperature in K (Kelvin). The heater or the electric furnace is controlled from the personal computer for the adsorption temperature. Dewar vessel: Enter liquid nitrogen “77.0” to “78.0”. Enter liquid argon “77.0” to “78.0”, or “87.0” to “88.0”. Heater (450 °C): Enter “323.15” to “723.15” (when using an optional device). Heater (550 °C): Enter “323.15” to “823.15” (when using an optional device). Electric furnace: Enter “323.15” to “1373.15” (when using an optional device). Circulation water bath: Enter “278.15” to “343.15” (when using an optional device).
Adsorptive name	Select N_2, Ar, etc. (specified in “Gas dosing valve assignment settings”) using the ▼ button.

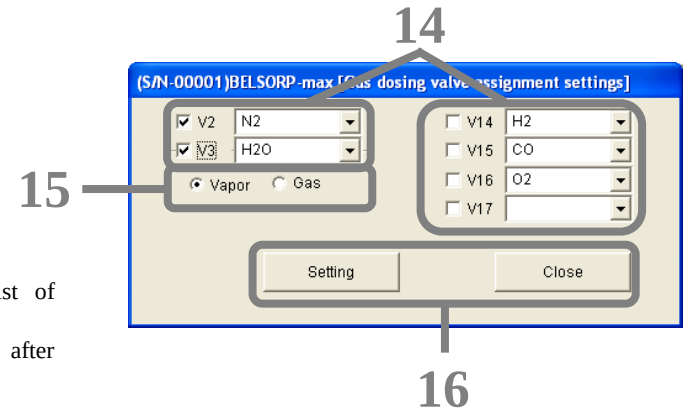
12. The measurement device can be selected from the devices checked in “Optional settings, P. 107”. When only “Dewar vessel” is checked in the optional setting, no measurement device is displayed. Select the appropriate device using the ▼ button.



13. To change the adsorptive name assigned to valve V2, V3, or V14 to V17, select the Gas dosing valve assignment settings button. The following “Gas dosing valve assignment settings” screen appears.



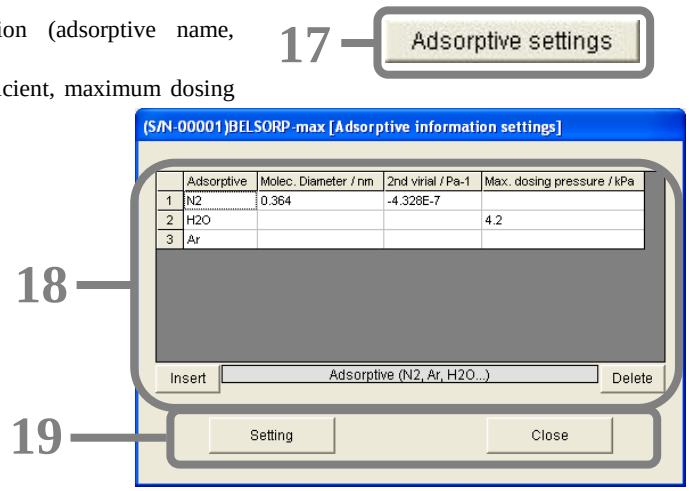
14. Enter directly the adsorptive name, or select it using the ▼ button (specified in “Adsorptive settings”). When checked, it can be selected from the list of “adsorptive name”, and “purge after measurement”.



15. Select Vapor when you connect Vapor to V3 and use it as adsorptive, otherwise select Gas.

16. Select the Setting button. Or, select the Close button to close the window without any setting. Return to the Measurement parameter settings window for additional setting.

17. To change the adsorptive information (adsorptive name, molecular diameter, second virial coefficient, maximum dosing pressure), select the Adsorptive settings button. The following "Adsorptive information settings" window appears. This information of the "molecular diameter", "second virial coefficient", and "maximum dosing pressure" is used as the measurement parameter when the "adsorptive name" is selected in step **11**.



In order to save the information of the adsorbate changed during measurement in JKN, select save in the measurement setting screen after finishing measurement.

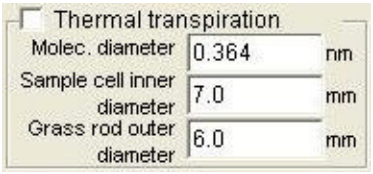
18. Specify the following items.

Adsorptive name	Enter the adsorptive name.
Molecular diameter (nm)	Average molecular diameter is used for the thermal transpiration correction. For nitrogen adsorption, enter "0.364".
Second virial coefficient (Pa^{-1})	For the "adsorptive gas non-ideality correction", enter the second virial coefficient at adsorption temperature when applicable. When not used, it may remain blank.
Maximum dosing pressure (kPa)	For the vapor adsorptive, the system temperature is 40 °C. Therefore, it cannot be dosed at pressure above the saturation vapor pressure at 40 °C. It is recommended to enter the saturation vapor pressure at 30 °C (4.2 kPa in the case of water) in consideration of non-uniform temperature in the manifold temperature. The vapor adsorption measurement cannot be performed since the "Start measurement" button does not change from gray to black without this numerical entry.
Insert button	This is used to insert a line to the line where a cursor is placed. Texts on the line where a cursor is placed are copied onto the line inserted. All the lines below the line where a cursor is placed are shifted a line down.
Delete button	This is used to delete a line where a cursor is placed. All the lines below the line deleted are shifted a line up.

For the molecular diameter and the second virial coefficient, refer to " P.178".

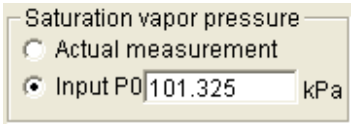
19. Select the [Setting] button. Or, select the [Close] button to close the window without any setting. Return to the [Measurement parameter settings] window for additional setting.

20. For the thermal transpiration correction, check the box and specify the following items.



Molecular diameter (nm)	Enter the average molecular diameter. For nitrogen molecule, enter “0.364”.
Sample cell inner diameter (mm)	The standard sample cell inner diameter is 7 mm. Change the setting when using the sample cell with different inner diameter.
Glass rod outer diameter (mm)	The standard glass rod outer diameter is 6 mm. Change the setting when using a glass rod with different outer diameter.

21. Select either the “Actual measurement” or “Input” mode for the saturation vapor pressure.



Actual measurement	When “Actual measurement” is selected, the value measured at the saturation vapor pressure P0 port is used as the saturation vapor pressure.
Input (kPa)	When “Input” is selected, change the “Saturation vapor pressure” where applicable.

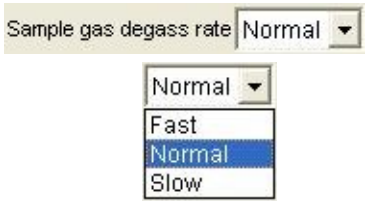
The setting range is limited as follows. ○: Available, ×: Not available

Setting range	Measurement device, adsorptive name, adsorption temperature			
	Dewar vessel			Heater, electric furnace, circulation water bath Except for the adsorptive name and adsorption temperature listed on the left
	N_2: 77K to 78K	Ar 77K to 78K	Ar 87K to 88K	
Actual measurement	○	×	○	×
Input	○	○	○	○

22. When you perform the adsorptive gas non-ideality correction, check the box and specify the second virial coefficient.

Second virial coefficient at the adsorption temperature	When you perform the adsorptive gas non-ideality correction, specify the second virial coefficient at adsorption temperature where applicable.

23. Select the sample gas degassing rate using ▼ button.



Slow	It degasses slowly to prevent the sample from scattering.
Normal	It degasses stepwise to prevent the sample from scattering.
Fast	It degasses quickly.

Generally, select “Normal”. When the sample scatters by selecting “Normal”, select “Slow”. Select “Fast” only when you measure the palletized or granular-shaped sample that does not scatter.

24. This is the exhaust time immediately before the first adsorption measurement point is measured. It is defaulted to 300 seconds. Set the exhaust time where applicable.

25. When you perform the purge after measurement, check this box.

Select the purge gas using ▼ button. When “Enter sample weight after measurement” is checked in step **9.**, the system performs purging without exception. Specify the purge gas.

⚠ Do not use dangerous gas for purging.

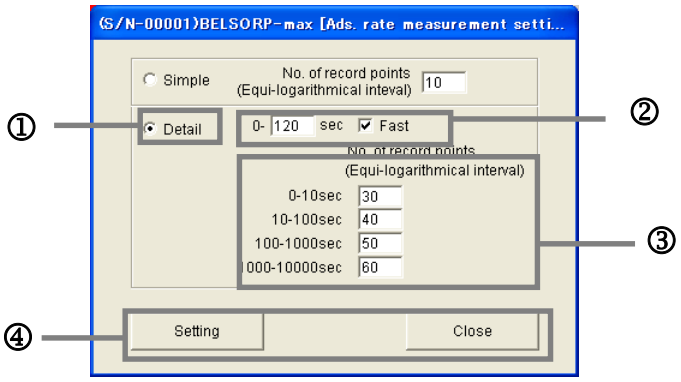
26. To conduct adsorption rate measurement[*1], check 

. (*1 adsorption rate measurement is an optional function.) The adsorption rate measurement is a single sample measurement in Port 3.

In the adsorption rate measurement, pressure change at each measurement point of adsorption side is saved. Further, since pressure change before equilibrium is reached is read with the pressure gauge at the Vs, it is saved as one adsorption point once gas is introduced even when target relative pressure is not reached.

Therefore, the number of measurement points may increase than usual due to inappropriate amount of excessive introduction. In the case of unknown samples, it is recommended to conduct adosorption rate measurement after doing a generally adsorption measurement to determine the appropriate amount of excessive introduction from the adsorption isotherm.

When Setting is selected, following window is displayed.



Select “Simple” in order to save data (pressure value) at certain equi-logarithmical intervals from start of measurement to equilibrium. The minimum data saving interval is the one point per second. Enter number of record points of adsorption time at equi-logarithmical intervals in the column of “No. of record points”. Larger input values result in increase of number of measurement points at each equi-logarithmical interval.

Select “Details” in order to specify data saving intervals each time. In the range of 0~120 seconds, selection of “Fast” enables data saving of about 5~8 points[*2] per second. (*2 Saving intervals depend upon communication intervals.)

[Method of setting “Details”]

①Select “Details”.

②Select “Fast” and enter the storage time (0-120 seconds can be entered).

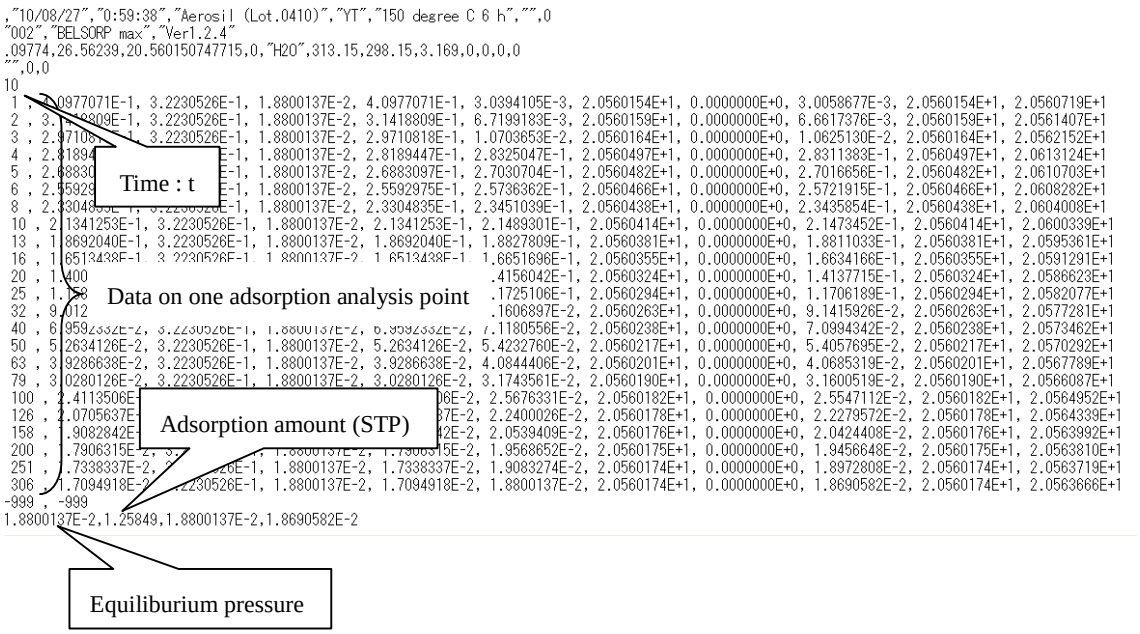
“Fast” is prioritized within the time for which “Fast” is selected. In the case of above setting, input values of “0-10sec” and “10-100 sec” are neglected up to 120 seconds.

When “Fast” is not selected, number of records at equi-logarithmical intervals input for each time is saved.

③Enter number of records at equi-logarithmical intervals in “10-100sec”, “100-1000sec” and “1000-10000sec”. For each time “0-500” points can be entered, and total number of saving is 2000 points.

④Select Setting after setting conditions. Select Close to return to the window for measurement condition setting.

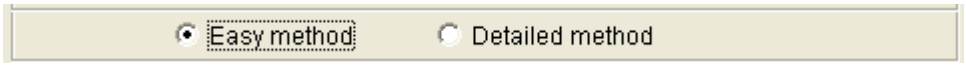
⑤Rate measurement data are saved in “….RAT” data. Conduct analysis using the rate measurement analysis software BEL-Dyna®, or analyze RAT file (CSV type file) graphically using the spreadsheet software.



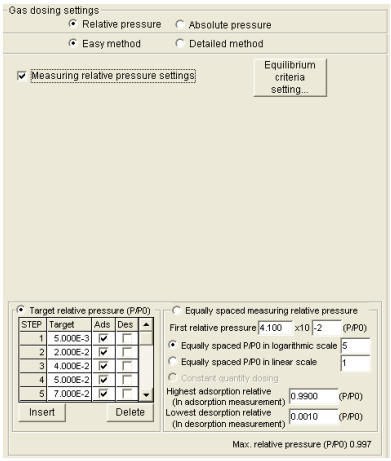
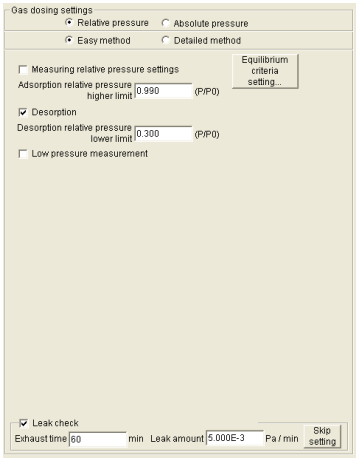
■ Setting the dosing pressure

1. Specify the adsorptive dosing pressure. Desired adsorption isotherm can be designed by specifying the dosing rate appropriately.

2. Specify the items related to the dosing pressure.

Relative / Absolute pressure indication	When the “Relative pressure” indication is selected, entries and indications are in P/P_0. When the “Absolute pressure” indication is selected, entries and indications are in kPa. 
Quick /Easy / Detailed method	When easy method is selected, setting can be accomplished easily without detailed condition setting of “target relative (absolute) pressure”, “excessive introduction amount” and “increase and decrease allowance of adsorption amount”. When detailed method is selected, setting of “target relative (absolute) pressure”, “excessive introduction amount” and “increase and decrease allowance of adsorption amount” becomes possible. Conducting detailed setting in accordance with necessary data enables to obtain data that better fit the purpose. 

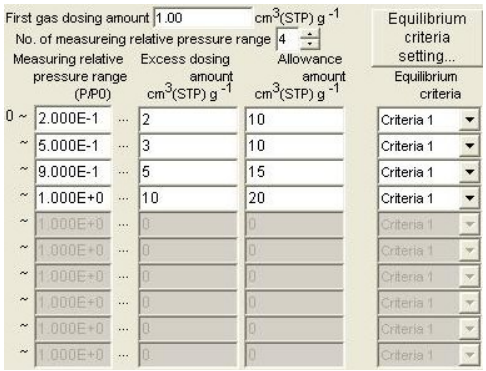
3. When “Easy method” is selected, specify the associated items as follows.

Measuring relative pressure settings	When this is checked, “Measuring relative pressure in adsorption measurement” and “Measuring relative pressure in desorption measurement” can be specified. When not checked, a predetermined value is used.	
Adsorption relative pressure higher limit (P/P_0)	Enter the adsorption higher limit in relative pressure P/P_0.	
Desorption measurement Desorption relative pressure lower limit (P/P_0)	When performing the desorption measurement, check the box. Enter the desorption lower limit in relative pressure P/P_0.	
Low pressure measurement	When “Low-pressure measurement” is checked, measurements from relative pressure of 1E-8 are carried out in N_2-77K measurements. In the case of absolute pressure, measurements from “Saturation vapor pressure x 1E-8” Pa are carried out.	

Check the low pressure measurement to perform micro-pore examination, analyze new analysis method (NLDFT/GCMC method). Untick the the low pressure measurement to perform steam adosorption measurement, analyze old classical analysis (t method, BJH method etc…).

4. When “Detailed method” is selected, specify the associated items as follows.

When the “Absolute pressure” indication is selected, assume the “relative pressure” as “absolute pressure”, and “P/P_0” as “kPa”.

Initial dosing rate $cm^3(STP)\cdot g^{-1}$	Specify the initial dosing rate (initial dosing rate for the 1st measurement point). 	
Number of measuring relative pressure range	Specify the number of “Measuring relative pressure range”.	
Measuring relative pressure range (P/P_0)	Specify the “measurement relative pressure range”.	
Excess dosing amount $cm^3(STP)\cdot g^{-1}$	In the adsorption/desorption process, the dosing pressure is determined so that it reaches the “target relative pressure” specified. With this calculation, however, dosing or exhausting gas may be repeated numerous times. For this reason, specify this excess dosing amount, which is added to or subtracted from the dosing amount calculated from the target relative pressure.	
Allowance amount $cm^3(STP)\cdot g^{-1}$	When the difference in adsorption between the previous and current measurement points exceeds this value, the current point is measured as an adsorption point, and plotted on a graph, even if the equilibrium pressure does not reach the target relative pressure.	
Equilibrium judgement	Select “Judgement 1 to 5” using ▼. Judgement is based on the following equilibrium judgement settings.	
Equilibrium judgement setting button	When this button is selected, the following “Equilibrium condition settings” window appears.	

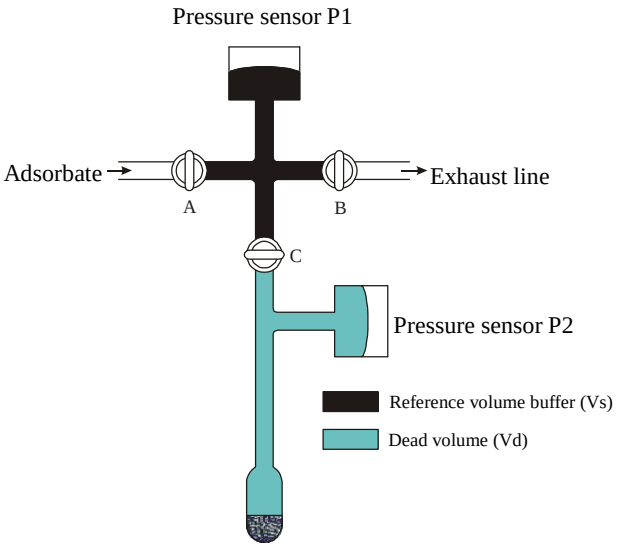
～ About excess dosing amount and allowable amount ～

1. Adsorption calculating procedure (Abridged edition)

Before describing the excess dosing amount and allowable amount, the following section describes the adsorption calculating procedure.

1. As shown in the figure on the right, the pressure sensor P1 and P2 is mounted to the "reference volume buffer: Vs" and "dead volume: Vd"
in the system, respectively.

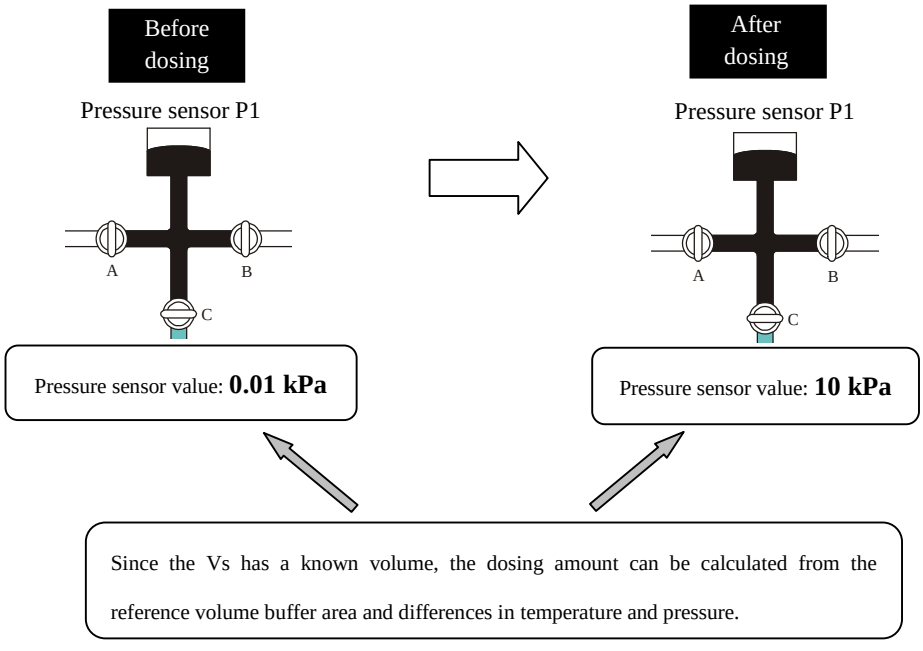
- Vs: This volume buffer has been measured at factory prior to shipment and is specific to the system.
- Vd: Since the volume of sample cell varies with each measurement, this volume must be measured using helium gas.



2. To perform the adsorption measurement, thoroughly exhaust gas from the space of Vs and Vd.

3. Dose the adsorption gas only in the space of Vs. The gas amount n_{Vs} dosed in the space of Vs can be calculated from pressure sensor values before and after dosing. (At this stage, the space of Vs is in a vacuum state.)

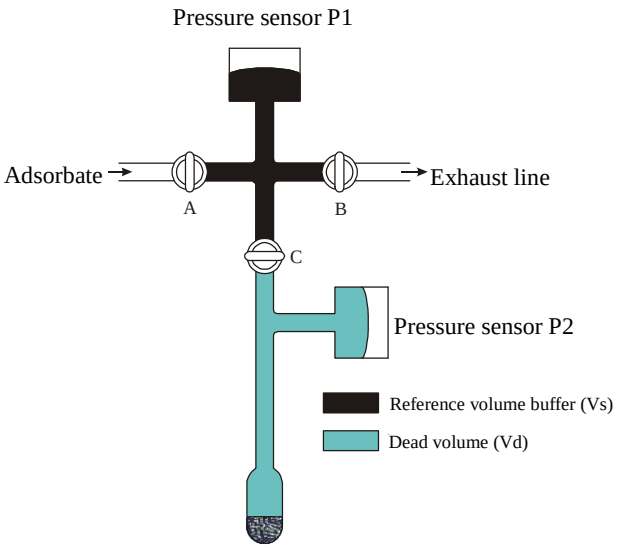
Example 1:



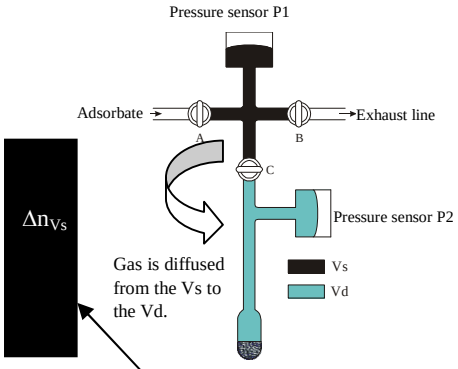
4. Open the valve C. Since the space of Vd is in a vacuum state, the suction gas in the Vs area flows into the space of Vd.

5. Close the valve C. Following the same procedure as that for Step 3, it can be measured how much the gas Δn_{Vs} has reduced.

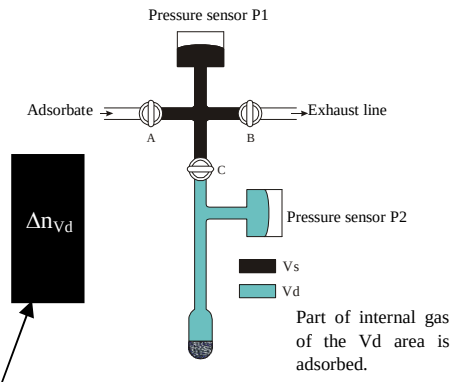
6. For the space of Vd, it can be calculated how much the gas amount Δn_{Vd} has entered according to the same procedure as that for the space of Vs.



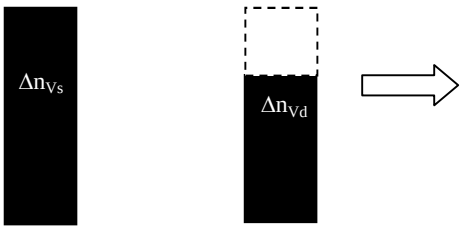
7. Normally, Δn_{Vs} calculated in Step 5 corresponds to Δn_{Vd} calculated in Stop 6. However, if adsorption takes place in the space of Vd, the pressure value will decrease, thus resulting in decreased Δn_{Vd}. The adsorption has been calculated according to differences between these Δn_{Vs} and Δn_{Vd}.



Gas amount Δn_{Vs} reduced by dosing gas in the Vd area



Gas amount Δn_{Vd} remaining after adsorption by dosing gas in the Vd area



It indicates that a difference in this gas amount adsorbs the sample cell.

Δn_{Vs}

Reduced gas amount in the reference volume buffer area

Δn_{Vd}

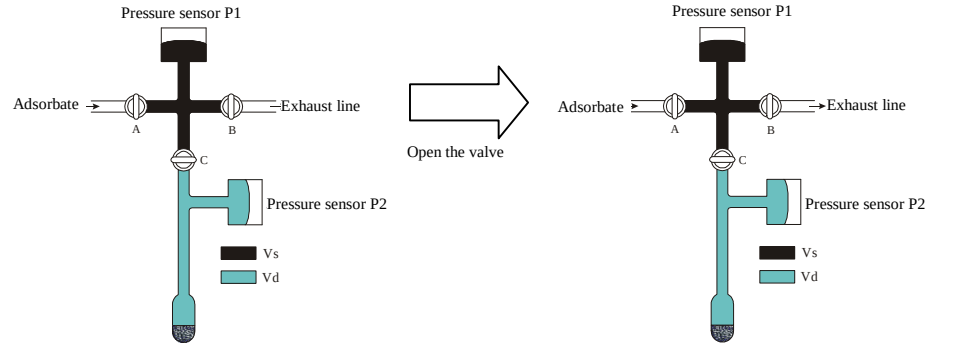
Increased gas amount in the sample cell area

2. Targeted relative pressure (Absolute pressure)

Perform the actual measurement as follows in order to automatically proceed with adsorption (desorption).

1. Determine the amount of gas to be dosed in the space of Vs first. Dose gas in the Vs area so that pressure in the space of Vd will come to a value set with the measurement parameter "Targeted relative pressure (Absolute pressure)".

Example 2: Suppose that the volume of the space of Vs is the same as that of the space of Vd and each space is in a vacuum state. In such case, consider a case where the parameter "Targeted relative pressure" is set to 10 kPa.



Dose a 20 kPa gas in the space of Vs.

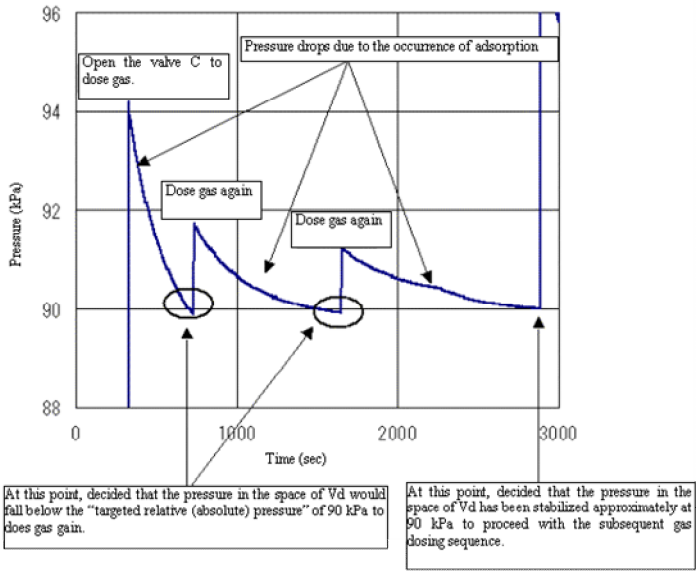
The pressure sensor P1 reads 10 kPa.

In this case, the amount of gas to be dosed in the space of Vs comes to 20 kPa.

2. The example shown above does not take into account a case where adsorption (a pressure drop) occurs in the space of Vd. When adsorption occurs, pressure in the space of Vd will drop to below the set "targeted relative (absolute) pressure". Consequently, the pressure is adjusted so that when the pressure in the space of Vd drops to below the set "targeted relative (absolute) pressure", the suction gas will be dosed again to raise the pressure to the "targeted relative (absolute) pressure". This adjustment is repeated until the pressure in the space of Vd reaches the "targeted relative (absolute) pressure".

Example 3: The following section shows pressure changes in the space of Vd at the time of adsorption measurement in the case where the parameter "Targeted relative (absolute) pressure" is set to 90 kPa.

∗Pressure stabilization for adsorption and desorption measurement can be set with the measurement parameter "Equilibrium".



3. Excess dosing amount

Example 3 shown above indicate that the system has dosed gas and waited for temperature stabilization three times to collect isothermal data at a single point, thus taking approximately 2,500 seconds to collect data at a single point. The excess dosing amount is a parameter used to increase the amount of gas dosed in the space of Vd and accelerate the measurement.

Gas dosed in the space of Vd when the "Excess dosing amount setting" parameter is set to "Disabled":

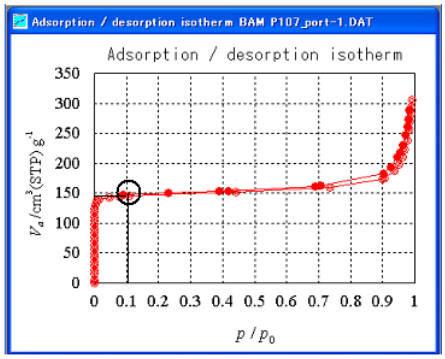
Dose gas only by the amount of gas dosed to set pressure in the space of Vd to the "targeted relative (absolute) pressure in the space of Vd.

Gas dosed in the space of Vd when the "Excess dosing amount setting" parameter is set to "Enabled":

Does gas by the amount of gas dosed to set pressure in the space of Vd to the "targeted relative (absolute) pressure + excess dosing amount in the space of Vd.

While gas was dosed three times in the case of the Example 3 shown above, using the excess dosing amount requires dosing of gas just one time to facilitate measurement.

Example 4: Suppose that you want to collect isothermal data at a point with P/P_0 of "0.1" for a sample that can obtain the isotherm shown below.



Set the "Targeted relative (absolute) pressure" parameter to "0.1" first. According to the isotherm, adsorption at relative pressure of 0.1 is approximately 150 cm^3 g^{-1}. Therefore, set the "Excess dosing amount" parameter to 150 cm^3 g^{-1}. Thus, data at the point having relative pressure of approximately 0.1can be collected in a shorter period of time without repeating dosing of gas as shown in Example 3.

Cautions:

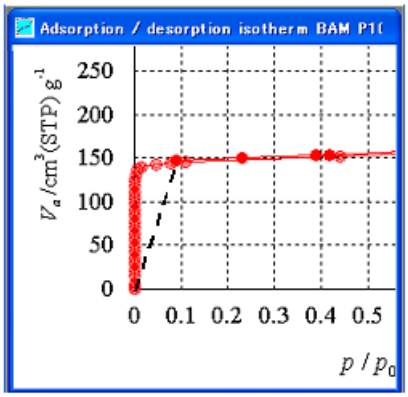
・With the example shown above, when the "Excess dosing amount" parameter is set to "250 cm^3 g^{-1}", even if the "Targeted relative (absolute) pressure" parameter is set to "0.1", a point with approximately 0.95 P/Po will be taken as the adsorption point. To avoid that, do not enter a value exceeding the adsorption at the "targeted relative (absolute) pressure" at which you want to collect the isothermal data in the "Excess dosing amount" parameter.

・The excess dosing amount is difficult to be set unless the adsorption of a sample, the shape of adsorption/desorption isotherm, and others can be forecast. For entirely unknown samples, make measurement using the "Abridged measurement procedure" and set measurement parameters as appropriate to make remeasurement.

4. Allowable amount

The allowable amount is a parameter used to set intervals to determine measurement points to the vertical axis of the adsorption isotherm ($cm^3\ g^{-1}$).

Example 5: Suppose that the first point of the "Targeted relative pressure" has been set to "0.1" and the "Allowable amount" parameter to "10 $cm^3\ g^{-1}$" for samples that can obtain isotherm shown on the right. Since the adsorption at the "targeted relative pressure" of approximately 0.1 is approximately 150 $cm^3\ g^{-1}$, 15 points (150/10=15) will be taken as the adsorption points before the "targeted relative pressure" reaches "0.1".

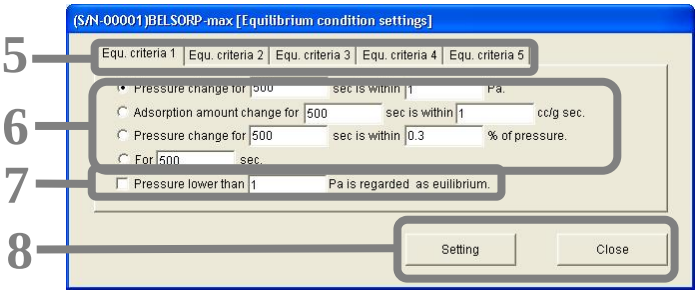


In contrast, in the case of the Type-I isotherm shown on the right, when the "Allowable amount" parameter is not set and the first point of the "Targeted relative pressure" parameter is set to "0.1", the isotherm becomes a straight line (black dotted line) up to "0.1", causing a deviation from the original isotherm.

Caution

・If the "Excess dosing amount" parameter is set to a value larger than that set with the "Allowable amount" parameter, isothermal points cannot be taken at intervals set with the "Allowable amount" parameter. As a guideline, enter a value equivalent to 50 to 80 % of the value set with the "Allowable amount" parameter as the "excess dosing amount".

5. When the Equilibrium judgement setting button is selected, the "Equilibrium condition settings" window appears as shown on the right. Select "Equ. criteria 1 to 5."

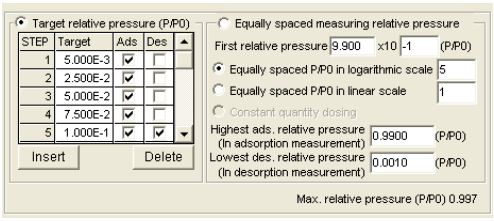


6. Select one of the conditions out of "Pressure change (Pa)", "Adsorption amount change (cc $g^{-1}\ s^{-1}$) ", "Pressure change (%)", or "without condition". Enter the equilibrium time (sec) and the change limit.

7. When the pressure below a certain level is regarded as equilibrium, enter the pressure and check in the box.

8. Select the [Setting] button. Or, select the [Close] button to close the window without setting any item. Return to the [Measurement parameter settings] window for additional setting.

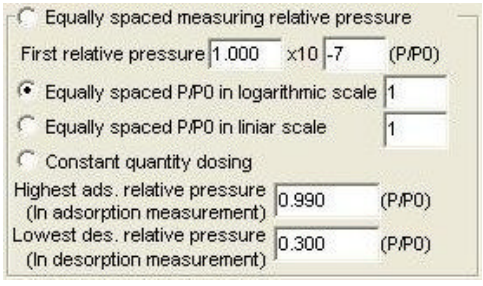
9. Select any one of “Target relative pressure” or “Equally spaced measuring relative pressure”, “Constant quantity dosing”.

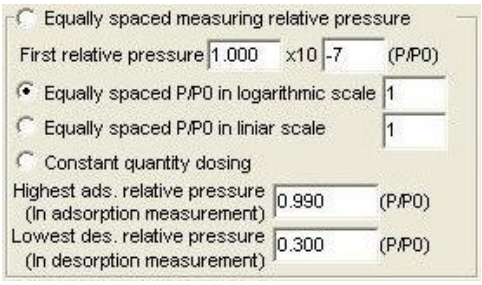


10. When “Target relative pressure” is selected, specify the associated items as follows.

Target relative pressure (P/P_0)	Specify the “target relative pressure” in the adsorption measurement.	
Adsorption	When checked, the target adsorption relative pressure on the line is effective.	
Desorption	When checked, the target desorption relative pressure on the line is effective.	
Insert	Press the [Insert] button to insert a line of step. It can be added up to 99 steps.	
Delete	Press the [Delete] button to delete a line of step. It is can be deleted to a single step.	

11. When “Equally spaced measuring relative pressure” is selected, specify the associated items as follows.

First relative pressure (P/P_0)	Enter the relative pressure to start adsorption. The adsorption points are equally spaced from the first relative pressure to the highest adsorption relative pressure.	

Equally spaced in logarithmic scale Equally spaced in linear scale Constant quantity dosing	Select one. For the equally spaced in logarithmic scale, and the equally spaced in linear scale, enter the number of adsorption/desorption points. With "equally spaced in logarithmic scale", the specified numbers of adsorption/desorption points are equally spaced in a logarithmic scale. With "equally spaced in linear scale", the specified numbers of adsorption/desorption points are equally spaced. In the "constant quantity dosing", the adsorption/desorption points reflect the first line of excess dosing amount. The lower limit of input value is "0.01".	
Highest adsorption relative pressure	Enter the highest adsorption relative pressure.	
Lowest adsorption relative pressure	Enter the lowest adsorption relative pressure. The desorption points are equally spaced from the lowest adsorption pressure to the highest adsorption relative pressure.	

12. Specify the equilibrium relative pressure allowable range.

It cannot be specified when the "Easy method" is selected.

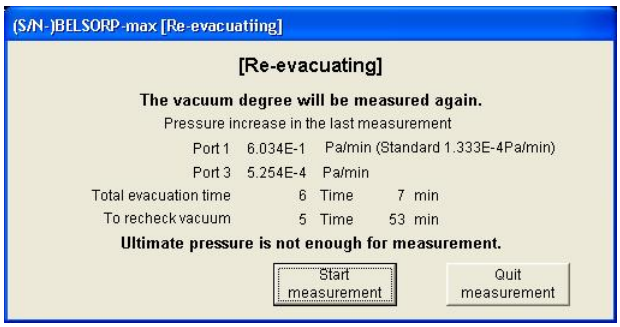
P/ P_0 tolerance (%)	Specify the allowable range to clear the target relative pressure. When the difference between the relative pressure during measurement and the target relative pressure is within the range, it clears the step and proceeds to the next step.

2. Setting the leak check

In order to check the vacuum and the amount of pressure increase in the sample portion before measurement,

check.

To measure adsorption isotherm from extremely low pressure (p/p_0 = 1E-8), be sure to do “Leak check”. In leak check, exhaust the sample cell for the specified “exhaust time”, close the valve of the sample portion, and check the amount of pressure increase. If the pressure increase is more than the specified “leak amount”, the window shown on the right is displaied, and repeats evacuation and leak check until it drops below the “leak amount”.



Setting of “exhaust time” and “Leak amount” can be changed even during leak check. Selection of Start measurement button forcedly starts measurement.

The leak check results are saved in the “VAC.CSV” file in “Send Data” folder of the “BELSORP-max” folder. Refer to the results to check the leak amount (Note that “VAC.CSV” is overwritten at every leak check).

If you check “Leak check”, “Measurement time” and “Elapsed time” are displayed on the measurement screen. They count the following times, respectively.

Measurement time·····It counts the measurement time that excludes leak check. Refer to it as an indication of time period after the Dewar vessel is filled with liquid nitrogen.

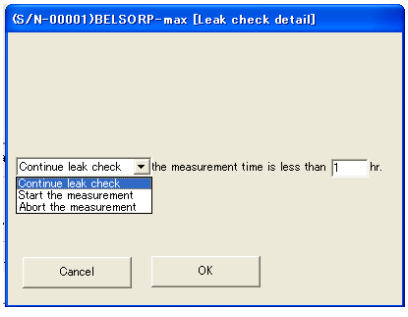
Elapsed time·····It counts the total measurement time that includes leak check.

Since measuring devices other than a dewar are set in the main unit before a leak check starts, each measuring device rises automatically if a leak check is passed successfully with a result of “Not larger than allowable pressure rise”.

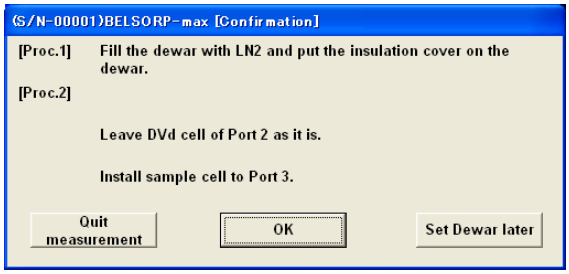
When a dewar is used and if it needs to be raised automatically after a certain time of a leak check, set the following conditions. (This function is only for dewars.)

1. Select “Leak check” on the measuring condition setting screen.

2. The window to the right appears when Detail is selected. If the leak check is not passed even when a specified measuring time (a time while the liquid nitrogen can be retained) has elapsed, select “Abort the measurement”, “Start the measurement”, or “Continue leak check”. (The selection can be changed even after the leak check starts.)

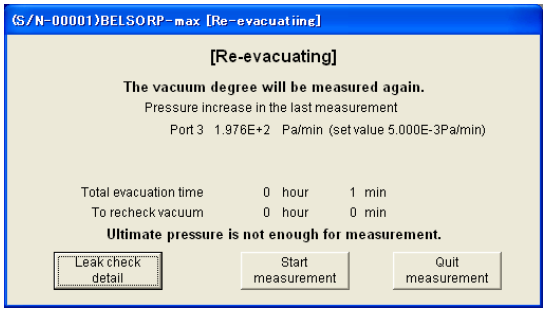


3. Set a dewar when the confirmation window to the right appears before a leak check after starting the measurement.



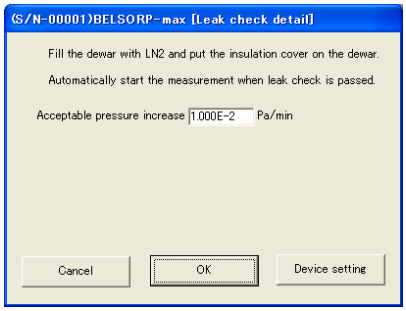
Select Set Dewar later if you set a dewar after a leak check is finished. In this case, a window appears to instruct you to set a dewar after a leak check is finished. Set a dewar following the instructions on the window.

After a leak check is started and if it is not passed with the setting value, the window below appears and the leak check is performed again.



[When a dewar is set later]

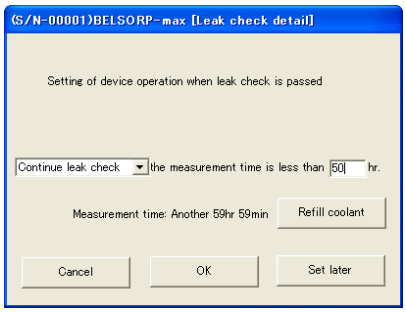
The screen to the right appears when Detail [Leak check detail] is selected.



Select Device setting if a device is set during the check. If it is selected, the screen is changed to the one shown at the right below.

[When a dewar is set from the beginning]

The screen to the right appears when Leak check detail is selected.



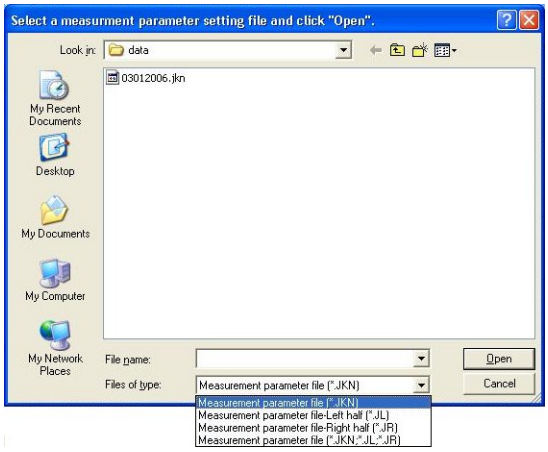
Select Refill coolant if coolant (liquid nitrogen) is replenished. The liquid nitrogen retention time (remaining time of measurement) is reset. Values, such as an allowable pressure rise value, can be changed during a leak check. A dewar having been set once can also be set again after a leak check (Select Set later).

3. Load / Save

1. Load or save the “Measurement parameter settings” file, where applicable.

Load	Select the Load button to load the existing “Pretreatment parameter setting” file. The file must have been saved using the Save button.
Save	Select the Save button to save the pretreatment parameter settings to the relevant file.

2. When you select Load or Save, the “Measurement parameter setting file” window appears. Specify the “location to load from” or the “location to save in”. Select or enter the “File name”, and then select “Open” or “Save”. An extension of “.JKN”, “.JL”, or “.JR” is automatically set to the measurement parameter setting file. For the rules to enter a file name, refer to the Windows instruction manual.



File types

Measurement parameter setting file (*.JKN):

to load or save the settings of **1. measurement parameter, 2. dosing pressure**

Measurement parameter setting file left half (*.JL):

to load or save the settings of **1. measurement parameter** on the left half

Measurement parameter setting file right half (*.JR):

to load or save the settings of **2. dosing pressure** on the right half

Limit of the measurement parameter entries

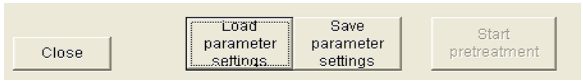
To prevent wrong measurement parameters from being entered, BELSORP-max limits parameter entry according to the instrument specification. When you attempt entering any parameter beyond the limitation, the value you entered is modified to the upper or lower limit value.

4. Starting the measurement

1. After specifying all the items, press the Start measurement button to start the measurement.

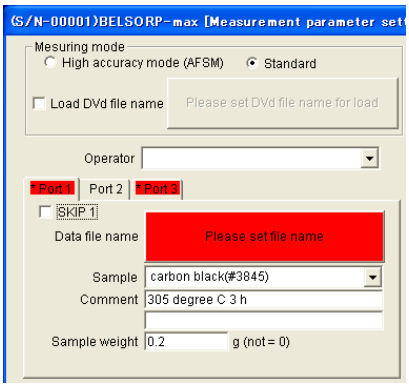
The measurement starts according to the measurement conditions predetermined in steps **1** to **3**.

When you navigate from “Pretreatment, P. 122”, select the Start pretreatment button to start the pretreatment.



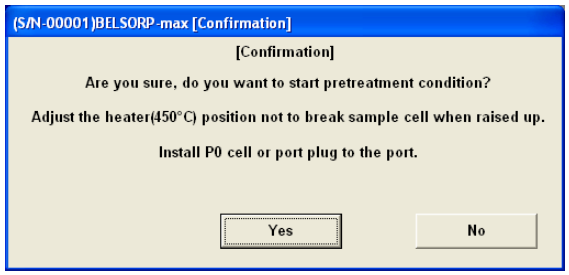
When the Start measurement button cannot be selected, there are some items remaining that must be specified (e.g. measurement data file name, etc.). Specify all the relevant items.

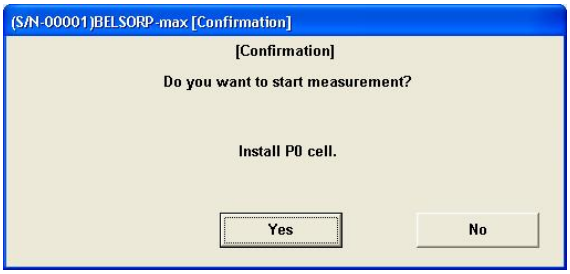
If Measurement start button cannot be selected, there are unset items. When main input items (Dvd saving file name, measurement data file name, adsorptive setting, saturation vapor pressure) are not input, items that are not input are displayed in red as shown in the window on the right. Set those items.



2. Press the Close button to cancel all the settings specified, and return to the “Main” window.

3. Select the Start measurement button. Then the “Confirmation” window (on the right) is displayed. Verify that the sample cell has been removed, and select Yes. Or, select No to return to the “Measurement parameter setting” window. After measurement is started, follow the instructions on the screen.

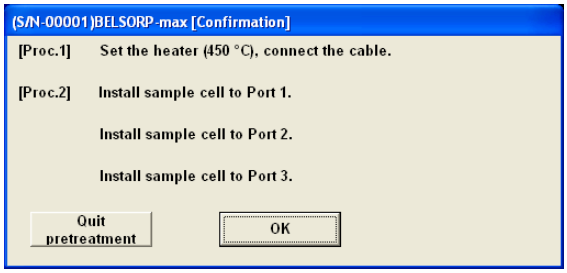




When Start measurement is selected, the “Confirmation” window (on the right) appears. Select Yes. Or, select No to return to the “Measurement parameter setting” window. After pretreatment is started, follow the instructions on the screen. You can perform continuously from pretreatment to measurement.

ooo Preparing the Dewar vessel and installing the sample cell ooo

On the “Measurement parameter setting” window, select the Start measurement button. Then, the system adjusts the pressure gauge. When the Confirmation window appears as shown on the right, prepare the Dewar vessel and install the sample cell, and then select the OK button (A similar message appears when you use the water bath, heater, or electric furnace.).



1. Preparing the Dewar vessel

⚠ The Dewar vessel is made of glass. Careful attention is required for the handling.
⚠ Do not drop the fitting into the Dewar vessel. It may be dangerous because the Dewar vessel glass may break.
⚠ Remove the ice or water adhered to the Dewar vessel and the heat insulation cap, before the sample measurement.
⚠ Do not turn on the Dewar vessel lifting-switch during the sample measurement.

1. Install the Dewar vessel to the main unit, according to “ How to install/remove the Dewar vessel, temperature measurement device P.61”.

2. Fill the Dewar vessel fully with liquid nitrogen. The Dewar vessel volume is about 2.6 L, and it retains for 60 hours.

3. After the Dewar vessel is filled with liquid nitrogen, install a heat insulation cap. Insert the Dewar vessel set pin to the set hole on the heat insulation cap.

2. Installing the sample cell

1. Install the sample cell, dead volume reference cell, and P0 cell to the measurement port according to the instruction on the "Confirmation" window.

2. Insert a glass rod to both the sample cell and the dead volume reference cell (when measuring the dead volume).

Sample cell and
Dead volume reference cell

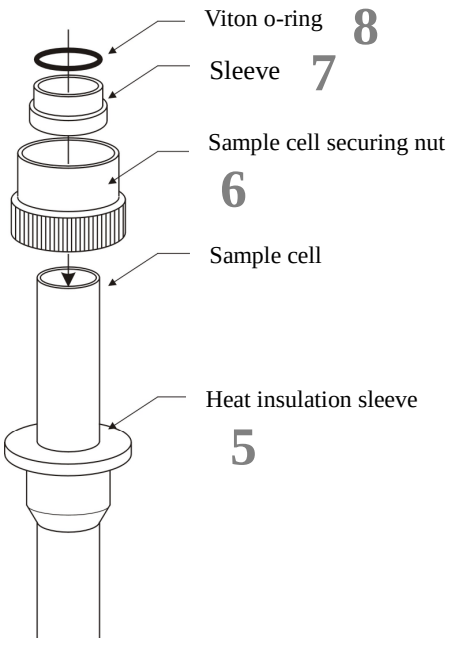
Glass rod

P0 cell (right)
o-ring (upper left)
Securing screw (lower left)

3. Install the sample cell after removing fitting and o-ring. When the sample cell is installed with the fitting still attached, it may deform the o-ring, and accordingly it may result in vacuum leakage. When you remove the sample cell from the instrument, the o-ring may remain inside the connection port. Verify that there is no o-ring inside the connection port.

4. Attach the fittings to both the sample cell and the dead volume reference cell, while paying attention not to use it upside down. Descriptions in parenthesis (< >) are for the P0 cells.

⚠ Do not drop the fitting into the Dewar vessel. It may be dangerous because the Dewar vessel glass may break.



5. Attach the heat insulation sleeve to the sample cell.

6. Attach the sample cell securing-nut <securing screw>.

7. Attach the sleeve.

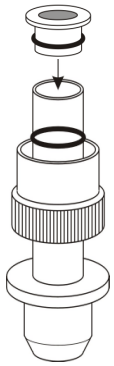
⚠ Do not use a heat insulation sleeve when using the BELSORP-max heater in the pretreatment or the measurement.

- It may result in a fire.

8. Attach a FKM (black) or FFKM (white) o-ring < o-ring >. The measurement section is sealed with this o-ring pressed on the sample cell and the measurement ports. Verify that the o-ring is not damaged, and no foreign material is pressed onto the o-ring. If air flows into the measurement section from outside, it may affect data accuracy. Replace the damaged o-ring, if any, with a new one.

9. Insert the filter (with a FKM (black) or FFKM (white) with o-ring) to the top of the sample cell.

9 Filter



10. Hold the sample cell firmly, and insert it to the sample port until it contacts the metal wall in the fitting. Tighten the sample cell securing-nut by hand.

11. Slide the heat insulation sleeve halfway down of the sample cell.

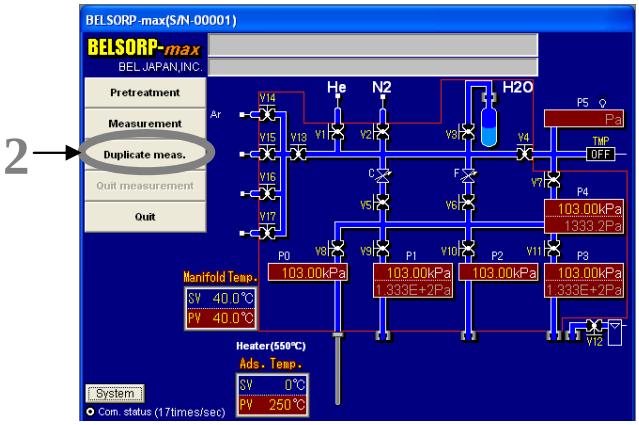
12. Set the sample cell in place, and press the OK button on the “Confirmation” window.

ooo Duplicate measurement ooo

In the duplicate measurement, continuous adsorption measurement (up to 10th) can be performed by selecting “Chemisorption measurement” or “Customized setting”. In chemisorption measurement, adsorption measurement is performed continuously with a fixed set of measurement parameters. In the customized setting, it is performed continuously with various measurement parameters.

1. Start the BELSORP-max main unit and the measurement software.

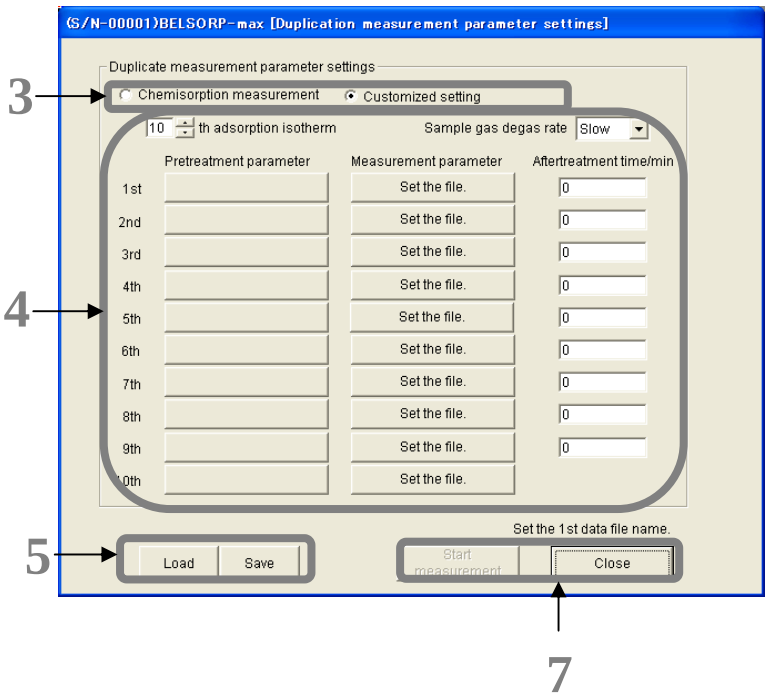
2. Select the Duplicate measurement button on the “Flow circuit diagram” window.



3. The “Duplication measurement parameter settings” window appears. Select “Chemisorption measurement” to duplicate measurement with a fixed set of measurement parameters, or select “Customized setting” to duplicate measurement while changing the measurement parameters, including the gas dosing port, adsorption temperature,leack check,dead vlume measurement, weight sample measurement etc.

When “Chemisorption measurement” is selected, specify a single set of pretreatment, measurement parameters since the measurement is duplicated with the fixed set of parameters.

When “Customized setting” is selected, specify the preatreatment, measurement parameter for every measurement of n-th adsorption. However, numebrs.of Sample cell, kind of sample cell, sample gas degas rate are fixed to these parameter selected 1st. Adsorptive becomes one choice gas either in steam.



* “Chemisorption measurement” is selected only the Dead volume measurement before measurement.

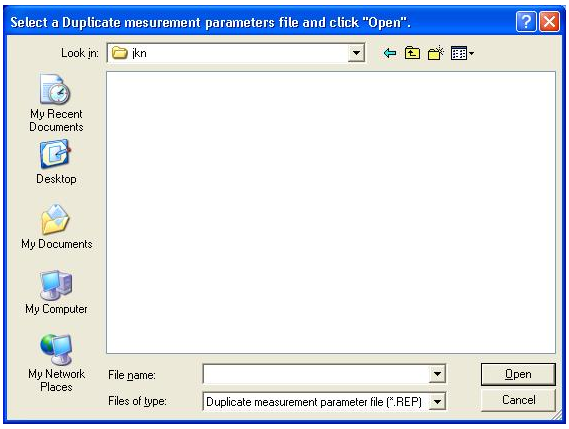
4. Set the following items.

n-th isotherm	Enter the degree of duplicated measurement, in a range of 1 to 10.
Pretreatment parameter	Specify the pretreatment parameter. Select the button on the line (degree) to specify. The “Pretreatment parameter settings” window appears. For setting, refer to “Pretreatment, P. 122”. -Button function on the “Pretreatment parameter settings” window-  Select the Load button to load the existing “Pretreatment parameter settings” file. Select the Save button to save the “Pretreatment parameter settings” file, return to the “Duplicate measurement settings” window, and associate the file with the duplicate measurement parameter. Select the Overwrite button to overwrite the “Pretreatment parameter settings” file, and return to the “Duplicate measurement settings” window to associate the file with the duplicate measurement parameter. Select the Delete button to cancel the settings, return to the “Duplicate measurement settings”, and disable the pretreatment on the line. Select the Close button to cancel the settings, and return to the “Duplicate measurement settings” window.
Measurement parameter	Specify the measurement parameter. Select the button on the line (degree) to set. The “Measurement parameter settings” window appears. For setting, refer to “Setting the measurement parameter, P. 128”. -Button function on the “Measurement parameter settings” window-  Select the Load parameter settings button to load the existing “Pretreatment parameter settings” file. Select the Save parameter settings button to save the “Pretreatment parameter settings” file, return to the “Duplicate measurement settings” window, and associate the file with the duplicate measurement parameter. Select the Overwrite button to overwrite the “Pretreatment parameter settings” file, and return to the “Duplicate measurement settings” window to associate the file with the duplicate measurement parameter. Select the Close button to cancel the settings, and return to the “Duplicate measurement parameter settings” window.
Sample gas degas rate	Select these sample gas degas trate of Sample cell.
After treatment time (min)	Specify the degassing time (minutes) after the adsorption/desorption measurement.

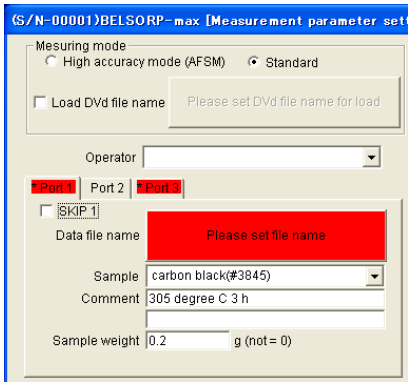
5. Load or save the “Duplicate measurement parameter settings” file, where applicable.

Load	Select the Load button to load the existing “Duplicate measurement parameter settings” file. The file must have been saved using the Save button.
Save	Select the Save button to save the duplicate measurement parameter settings to the relevant file.

6. When you select Load or Save, the “Duplicate measurement parameters file” window appears. Specify the “location to load from” or the “location to save in”. Select or enter the “File name”, and then select “Open” or “Save”. An extension of “.REP” is automatically set to the duplicate measurement parameter setting file. For the rules to enter a file name, refer to the Windows instruction manual.



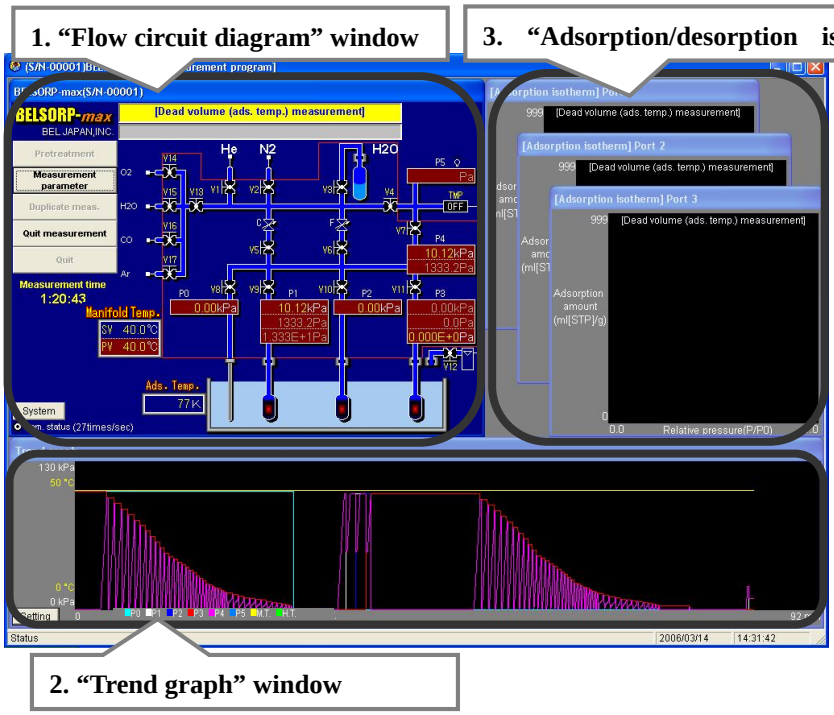
When all items are specified, use the Start measurement button to start measurement. Measurement is performed according to the measurement parameters specified in the above steps **1.** to **3.**



When the Start measurement button cannot be selected, there are some items remaining that must be specified (e.g. Dvd saving file name, measurement data file name, adsorptive setting, saturation vapor pressure, etc.). When main input items are not input, items that are not input are displayed in red as shown in the window on the right. Set those items. Specify all the relevant items. Use the Close button to cancel all the settings specified, and return to the “Main” window.

ooo “Main” window ooo

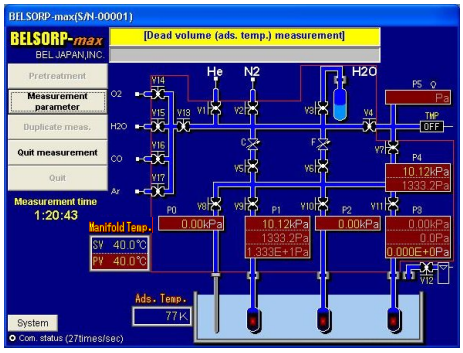
The “Main” window of the measurement software consists of the “Flow circuit diagram" window, the “Trend graph” window, and the “Adsorption/desorption isotherm" window. These windows provide information about the instrument operating status, pressure variations, measured data (adsorption isotherm), etc. in real time during the sample measurement. You can control the valve operation (V1 to V17) from the “Flow circuit diagram” window (For the stop valve operation from the “Flow circuit diagram” window, refer to P. 84.).



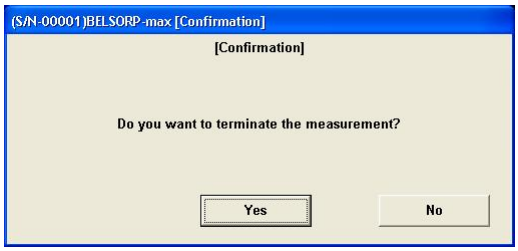
ooo “Flow circuit diagram” window ooo

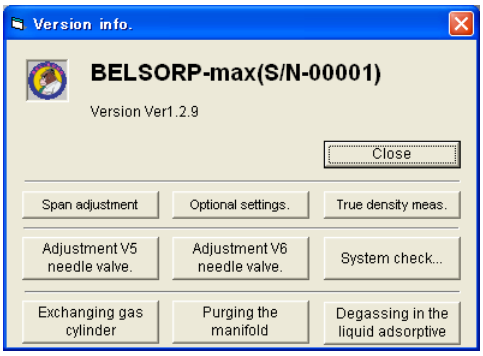
1. While measuring

The “Flow circuit diagram” window appears as shown on the right while measuring. The stop valve status, pressure, and temperature can be monitored. The buttons and their functions on the window are as follows (The stop valves can be operated manually even while measuring; however, do not do this because it may cause measurement error.).



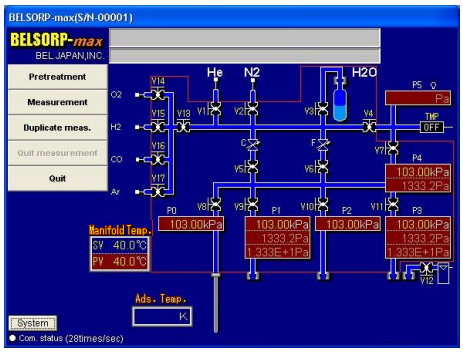
Measuring samples

Pretreatment	The pretreatment parameters can be viewed during sample pretreatment. Including “pretreatment time”, it can be changed during pretreatment. Select the Setting button to change the settings. Use the Close button to close the window.
Measurement parameter	The measurement parameters can be viewed during sample measurement. Including “dosing amount”, it can be changed during measurement. The setting range depends on the measuring status. Change the relevant items, and select the Setting button to update the settings. Use the Close button to close the window.
Duplicate measurement	The duplicate measurement parameters can be viewed during sample measurement. Including “after treatment time”, “pretreatment parameter”, and “measurement parameter”, it can be changed during measurement. Change the relevant items, and select the Setting button to update the settings. Use the Close button to close the window.
Quit measurement	Select Yes on the following “Confirmation” window to finishi the current measurement, and perform post-treatment. 

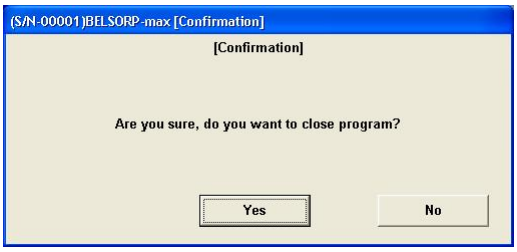
System	The software version information window appears. 

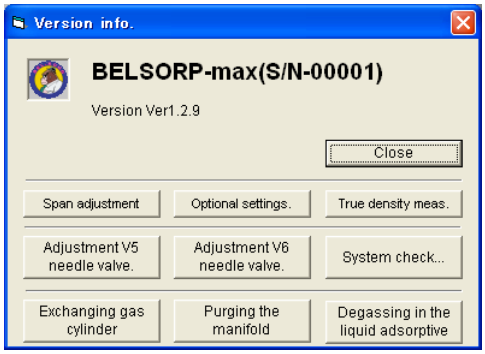
While not measuring

The “Flow circuit diagram” window appears as shown on the right while not measuring. You can control the stop valve operation. The pressure and temperature can be monitored. The buttons and their functions on the window are as follows.



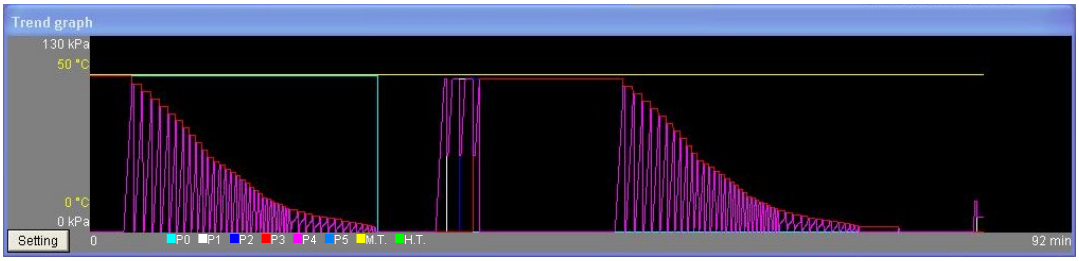
> ⚠ When you operate the valves manually, careful attention is required to prevent toxic, flammable gas from being discharged, and to prevent the turbo molecular pump from being overloaded.

Pretreatment	Select this button to pre-treat the sample. When the Pretreatment button is selected, the “Pretreatment parameter settings” window appears.
Measurement	Select this button to measure the sample. Select the Measurement button. Then the “Measurement parameter settings” window appears.
Duplicate measurement	Select this button to duplicate the measurements. When the Duplicate measurement button is selected, the “Duplicate measurement” window appears.
Quit	Select the Yes button on the following “Confirmation” window. Then, the instrument closes all valves, returns to the initial state, and then exits the software. 

System	The “System version information” window appears. For the span adjustment, refer to “Span adjustment, P. 99”. For the optional setting, refer to “Optional settings, P. 107”. For the needle valves V5 and V6 adjustment, refer to “Adjusting the needle valve, P. 97”. For the system check, refer to “System check, P. 94”. For the gas cylinder replacement, refer to “Installing and replacing gas cylinders, P.90”. For purging, refer to “Purging the tubing in the instrument, P. 101”. For degassing, refer to “Degassing adsorptive, P. 103”. 

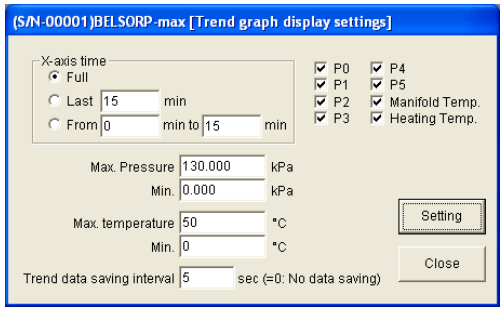
ooo “Trend graph” window ooo

Trend graphs are displayed for different pressures (P0 to P5) and temperatures (TIC1, Ads. Tmp). The pressure variations due to adsorption or desorption to/from the sample can be monitored during the sample measurement.



Buttons and their functions on the window are as follows.

Display setting	The trend graph scale can be changed. You can also set the interval to save the “TrdData.CSV” in the “BELSORP-max_SendData” folder in the “Trend data saving interval” field.



Colors used in the “trend graph” are as follows.

		Color
Temperature (M. T.)	Manifold temperature / °C	Yellow
Heating temperature (H. T.)	Temperature device / °C	Green
Pressure (P0)	Pressure at port 0 / kPa	Light blue
Pressure (P1)	Pressure at port 1 / kPa	White
Pressure (P2)	Pressure at port 2 / kPa	Red
Pressure (P3)	Pressure at port 3 / kPa	Blue
Pressure (P4)	Reference volume pressure / kPa	Purple
Pressure (P5)	Vacuum gauge / Pa (common logarithm)	Deep blue

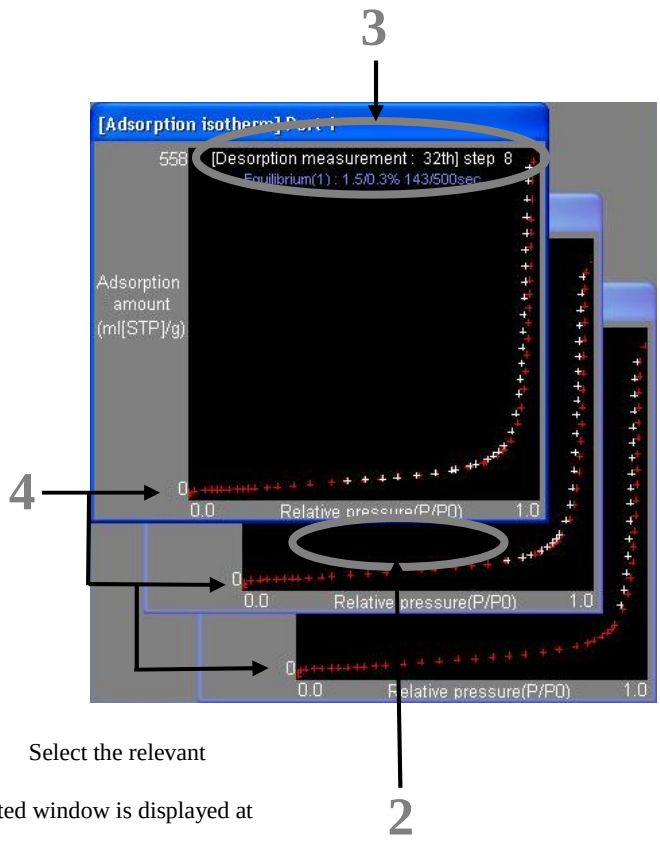
ooo “Adsorption/desorption isotherm” window ooo

1. The measured results are displayed on the “Adsorption/desorption isotherm” window during the “sample measurement”.

2. The measurement point is identified with the following colors.

- Adsorption measurement point: Red
- Desorption measurement point: White

3. The current measurement point is indicated in the message column. In the window shown on the right, it is indicated that the system is currently measuring the 32nd point in the desorption process. The step 8 corresponds to the step of “Target relative pressure” that was predetermined through the “measurement parameter Setting”.



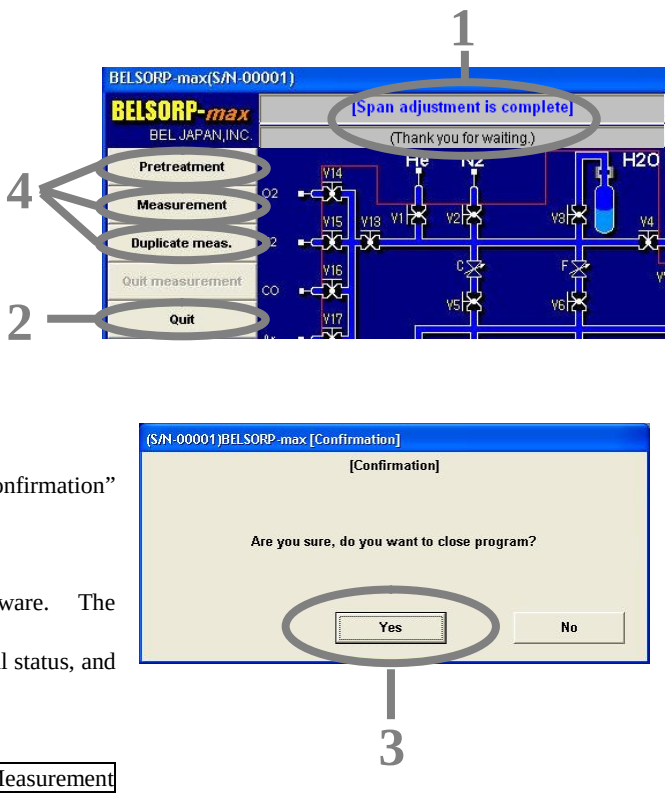
4. The results measured at Port 1 to 3 are separately displayed on the individual windows. Select the relevant window to view the measured data. The selected window is displayed at the front.

- The port 1 window is displayed when Port 1 is specified for the measurement parameter setting.
- The port 2 window is displayed when Port 2 is specified for the measurement parameter setting.
- The port 3 window is displayed when Port 3 is specified for the measurement parameter setting.

Operation to terminate the measurement

ooo Exiting the measurement software and shutdown of BELSORP-max ooo

1. Exiting the measurement software



1. When the “Sample measurement” is completed normally, the “Flow circuit diagram” window as shown right is displayed. For the “Pretreatment”, it displays a message of “Pretreatment complete”.
2. Select the Quit button, then the “Confirmation” window is displayed.
3. Select Yes to exit the measurement software. The instrument closes all valves, returns to the initial status, and then exits the software.
4. When you continue measuring, select the Measurement button. When you perform pretreatment, select the Pretreatment button. When you duplicate measurement, select the Duplicate meas button to start measuring, respectively.

2. Shutdown of BELSORP-max

In case using the instrument for the measurement, following turn-off procedure should be done more than 30 minutes after the measurement.

1. Turn off the main unit by pushing down the power switch on the back of the unit. The power indicator on the front of the main unit lights off.
2. Turn off the rotary pump immediately.
3. Close the main gas valve of a gas cylinder when the instrument will not be used for a month or more.

Caution

Do not turn off the pump with the instrument main unit turned on.

- **When the main unit remains turned on after the pump is turned off, oil may flow back and the instrument may be contaminated with the oil.**
- **We are not liable for any damages resulting from improper use of this product.**

Measurement data file

ooo Measurement data file configuration ooo

1. Measurement data

The “measurement data” of BELSORP-max is saved in a file according to each measurement step. The “measurement data” file can be opened and analyzed using the “BELSORP analysis software”, which is useful to control the measuring status.
The “measurement data” file is in the tab-deliminated text format. It can be opened using commercial spreadsheet software after measurement.

Do not open the “measurement data” file using any software except the BELSORP analysis software during measurement. Otherwise, it may prevent the measurement data from overwriting.

The “measurement data” file is saved with a name specified at measurement. An extension of “.DAT” is set to the file. An example of the “measurement data” file is shown in the following section **1.** .

When you select “After adsorption/desorption measurement” in the measurement parameter “dead volume measurement”, a file with extension of “-Z.DAT” is saved additionally (After measurement, the file corrected the dead volume with extension of “.DAT” is changed). This file contains the data before the dead volume correction.

2. Example of the measurement data file

```
==================
System property BELSORP-max Ver1.2.9
==================
"Instrument S/N:"     000002
"Vs/ml:"     31.364
==================
Measurement parameter
==================
"Adsorptive:"          H2O
"Meas. Temp./K:"     298.15
"Molecular cross-sectional area:"          0
"Molecular weight of adsorbate:"          0.00
"Equilibrium time/sec:"          0
"Measurement mode:"          2
==================
Sample information
==================
"Sample weight/g:"     0.09540
"Comment1:"          "Aerosil (Lot.0410)"
"Comment2:"          ""
"Comment3:"          "150 degree C 6 h"
"Comment4:"          "Leak amount : 0.006Pa/Min, Vacuum degree before measurement:5.469E-1Pa"
"as/m2 g-1:"          0.0000
"Molecular weight of adsorbent:"          0.00
==================
Time, dead volume
==================
"Date of measurement:"          12/01/11
"Time of measurement:"          19:23:48
"Slope of dead volume:"          5.6697E-7
"Intercept of dead volume:"          2.1205E+1
"Average dead volume:"          2.1236E+1
==================
Adsorption data
==================
"No."    "Pe/kPa"     "P0/kPa"     "Vd/ml"     "V/ml(STP)･g-1"
1          2.8403E-2    106.17       14.178      4.7988
2          6.8979E-2    106.67       14.153      10.387
3          1.1483       106.70       14.132      15.043
4          2.7673       106.46       14.122      15.935
5          5.3033       106.29       14.103      16.564
                  ⋮
0          0            0                0           0
==================
Desorption data
==================
"No."    "Pe/kPa"     "P0/kPa"     "Vd/ml"     "V/ml(STP)･g-1"
1          101.18       104.20       13.543      508.84
2          100.77       104.23       13.523      481.32
3          100.47       104.18       13.503      454.48
4          100.28       104.15       13.488      428.07
5          100.08       104.07       13.473      402.04
                  ⋮
0          0            0                0           0
```

No.: Adsorption point
Pe/kPa: Adsorption equilibrium pressure
P0/kPa: Saturation vapor pressure
Vd/ml: Dead volume change
Vd/ml(STP)*g^{-1}: Adsorption amount

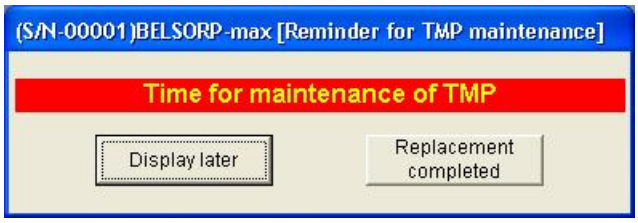
End of adsorption data

No.: Desorption point
Pe/kPa: Desorption equilibrium pressure
P0/kPa: Saturation vapor pressure
Vd/ml: Dead volume change
Vd/ml(STP)*g^{-1}: Desorption amount

End of desorption data

ooo Turbo molecular pump maintenance ooo

When the accumulated operating time of the built-in turbo molecular pump exceeds 20,000 hours, BELSORP-max prompts for maintenance on the PC screen as shown on the right. Once you select "Display later", the window will be displayed again later. Please contact our company if you find this message.



ooo Rotary pump (GHD-030) maintenance ooo

When you use the rotary pump, you must periodically replace its oil. This section describes two critical maintenance items for the rotary pump (GHD-030).

1. Replacing oil: Replace oil once every 3 months, or for every 6,000 hours operation.
2. Replacing the shaft part: Replace the pump shaft part once a year.

⚠ Failure to comply with the above periodical maintenance may result in failure of the pump, and accordingly it may cause failure of the instrument (due to backflow of oil resulted from the failure). Be sure to perform periodical maintenance as follows.

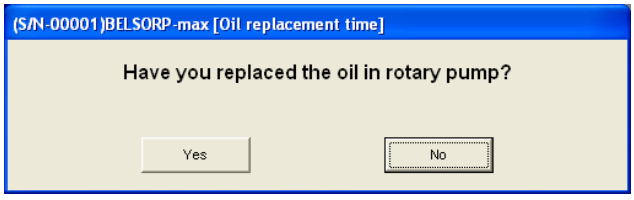
Message for oil replacement

1. Message below appears when the timing of oil replacement has come.

2. If selecting the Replace button, the message below will appear. Replace oil according to the operational procedure of "Oil replacement p. 154". After oil has been replaced, select Yes.

Select No, if oil has not been replaced. The message of step 1 will appear again.

In case of selecting the Display later button, the message above will appear again after the measurement software is started again or measurement has finished.



Replacing oil

Tools and other requisites: New oil, 6mm Allen wrench, a container for oil, towels

1. After disconnecting from the instrument hose, remove the black cap **1** as shown in the photo on the right.

> ⚠ Power is supplied to the pump through the main unit. To prevent oil from flowing back, be sure to turn off the instrument power before you disconnect the pump power cable.

2. Remove the screw **2** using a 6 mm Allen wrench. When you loosen the screw, oil may flow out. Receive the oil with a container for oil.

3. After discharging the oil completely, wipe off the oil around the drain port, and mount the screw that you removed in step **2**.

Fill new oil up to the circle marked position.

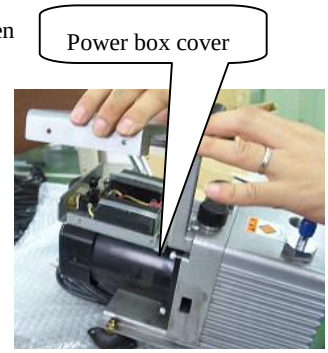
4. Fill new oil from the port **1**. Fill it up to the oil level gauge as shown in the photo on the right, which is located on the side of the pump.

5. Replace the cap **1**. Replacing oil is now complete.

> ⚠ Power is supplied to the pump through the main unit. To prevent oil from flowing back, be sure to turn off the instrument power before you disconnect the pump power cable.
>
> ⚠ When oil gets into your eye, wash it sufficiently with clean water, and obtain medical advice.
>
> ⚠ When oil contacts with your skin, wash the skin with soap and water.

3. Replacing the shaft part (spider)

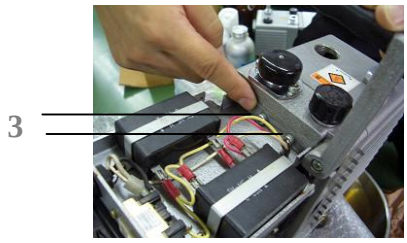
Tools and other requisites: Shaft part (spider), 4 mm, 5 mm and 6 mm Allen wrenches, Philips driver, a container for oil, towels



1. Discharge oil from the pump in the same way as described in **1. Replacing oil**, steps **1** and **2**.

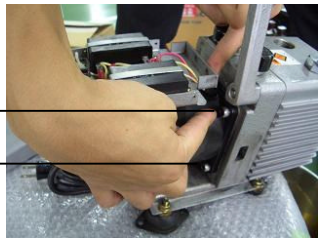
2. Remove the 4 mm screw using a Philips driver, and remove the power box cover.

3. Remove the screw **3,** using a 4mm Allen wrench.



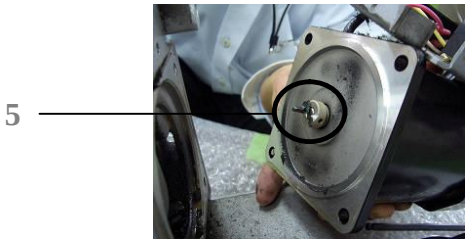
4. Remove the screw **4,** using a 5mm Allen wrench.

Once you remove the screw, the motor can be detached from the pump. Your careful attention is required.



Shaft part (spider)

5. Wipe off the dirt both on the motor and main unit, remove the shaft part (spider) at the position **5,** and then install a new spider.



6. After installing the spider, mount the pump, and then refill the pump with oil in the same way as described in **1. Replacing oil**, steps **4** and **5**. Now, replacing the spider is complete. After you refill the pump with oil, connect the pump to the main unit to check the evacuation operation.

⚠ Power is supplied to the pump through the main unit. To prevent oil from flowing back, be sure to turn off the instrument power before you disconnect the pump power cable.

⚠ When oil gets into your eye, wash it sufficiently with clean water, and obtain medical advice.

⚠ When oil contacts with your skin, wash the skin with soap and water.

ooo Daily maintenance ooo

· When power is supplied:

Verify that the power indicator lights on.

Verify that the rotary pump operates normally.

In addition, verify that the pump's exhausting noise quiets down within 1 to 2 minutes.

In the event of trouble, turn off instrument immediately, and disconnect the power cable from the wall outlet.

· Rotary pump:

Perform the appropriate maintenance to the rotary pump according to the instruction manual for the pump. In the event of any abnormal sound and/or smoke, turn off the instrument immediately, and disconnect the power cable from the wall outlet. Appropriately arrange the pump's exhaust port, i.e., connecting it to the exhaust duct, etc. .

· Power cable and communication cable:

Verify that the power cable and the communication cable are not degraded, and they are connected properly. In the event of trouble, turn off the instrument immediately, and take appropriate measures; i.e., replacing the cable, etc.

ooo Trouble shooting ooo

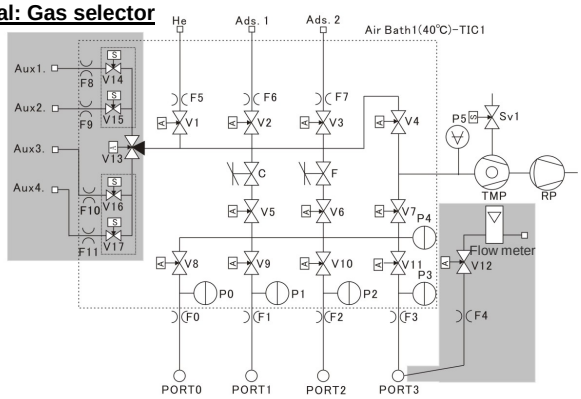
Symptoms	Check point
Power is not supplied.	・ Is the power cable connected correctly?
Insufficient vacuuming	・ Check the rotary pump operation (Is the switch on? Any trouble with oil?) . ・ Check for any loosening at the pump suction port, the connection port of the turbo molecular pump and the rotary pump. ・ When the system vacuums the sample port, check for any leakage in the sample cell, and/or any degradation of the o-ring.
The measurement system valve does not operate.	・ Is the cable connected firmly between the main unit and the computers?
The instrument stops due to communication error.	・ Is the cable connected firmly between the main unit and the computers?
Faults in adsorption amount	・ Check the purity of gas. ・ Verify that the measurement procedure is correct. ・ Measure the reference sample from Bel and verify the results.
The elevator abnormal noise	・ Grease may deteriorate. Please contact our company.
The elevator does not operate.	・ Verify that the elevator is not overloaded, and then press the elevator reset switch in the "Temperature controller pocket" on the front of the main unit.

ooo How to recover from abnormal stop ooo

If measurement is aborted after measurement is started and the Dewar vessel or the heater is lifted up for any reason (power failure, exiting software by mistake, communication failure, etc.), recover it according to the following procedure.

⚠ Moving down the Dewar vessel manually with gas charged in the sample section may cause rapid pressure increase because the sample section temperature returns to room temperature, and accordingly it may disconnect the sample cell. Be sure to comply with the following procedure.

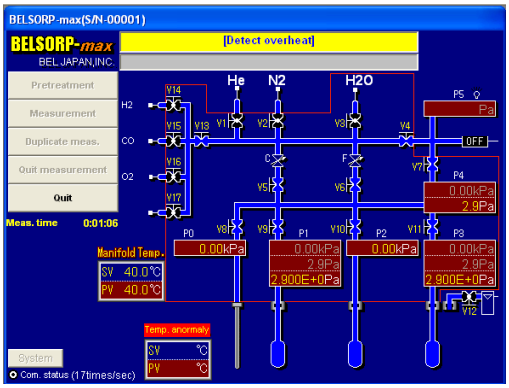
Optional: Gas selector



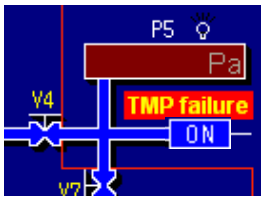
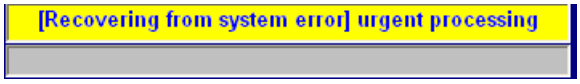
Flow diagram

The measurement stops with the Dewar vessel lifted (In case of two samples measurement).	① Exit the measurement software, and start it again. ② Open V4 to 8, and 10 (do not open V10 when the sample is installed to Port 2). ③ Close V4, 7 and 10. ④ Open V1, 5 and 6, and dose helium up to the pressure in the sample section while observing the pressure at P4. ⑤ After dosing, close all valves. ⑥ Open V9 and 11 (open also V10 when the sample is installed to Port 2). ⑦ Open V4 to 6 to exhaust slowly the sample section. ⑧ When the pressure at P4 drops below 3 kPa, move down the Dewar vessel manually. ⑨ Continue exhausting until the sample cell returns to room temperature. When it returns to room temperature, close all valves. ⑩ The procedure is now complete.
The measurement stops with the heater lifted.	① Exit the measurement software, and start it again. ② Turn off the heater switch of the temperature controller (refer to P.31). ③ Move down the heater manually. ④ Wait until the sample cell returns to room temperature. ⑤ The procedure is now complete.

ooo How to recover from error message ooo

Error message	How to reset
Temperature device overheat detection When the temperature device (450 °C heater, 550 °C heater, 1100 °C electric furnace) exceeds the specified temperature (for details refer to the temperature controller on P. 32), the temperature controller detects faults and stops heating (in such a case, the “overheat indicator” on the front of the temperature controller lights up). When the measurement software is running, it exits automatically. 	After cooling the temperature device, turn off the power switch on the front of the temperature controller, and then turn it on again to reset. Verify that the “overheat indicator” on the front of the temperature controller lights off.
Elevator overload detection When the elevator load exceeds 50 kg, it detects faults, and disables lifting operation (in such a case, the lifting switch ■ on the front of the instrument blinks red). When the measurement software is running, it exits automatically. 	A reset switch for the elevator is located in the “temperature controller pocket” on the front of the instrument. After you remove the cause, press the reset switch to release any protective actions.
System faulty pressure detection ***(Only when the measurement software is started)*** When any pressure gauge exceeds 130 kPa, it detects faults and triggers an emergency stop (exhaust process). Only in the adsorption/desorption measurement, it detects faults when P0 exceeds 110 kPa. 	Leave it without any action until the software exhausting process is complete.

Maintenance

Turbo molecular pump overload detection ***(Only when the measurement software is started)*** When air is sucked while the turbo molecular pump is in operation, it detects load and stops the TMP operation (in such a case, “TMP error” appears on the software screen). 	Even when the TMP error is indicated, the measurement is not aborted. Stop the low-pressure measurement because vacuum is degraded. To stop measuring and reset the error, turn off the instrument power switch 10 minutes or more after the error detection, and turn it on again another 5 seconds later to reset.
Recovering from system error In case measurement is stopped by some reason, urgent processing will begin at restart of the software. 	When measurement is stopped, finish the software and restart it. Leave it as it is, since urgent processing will start automatically.

ooo Consumables ooo

The following consumables are available with BELSORP-max. Please contact us for any inquiry.

model	Item and Specification
【Sample cell】	
010-20000-0-0	Pyrex sample cell (MAX.500 °C) 3/set
010-20001-0-0	Silica sample cell (1.8 ml) 1/set
010-20002-0-0	Pyrex small volume sample cell (MAX.500 °C, 0.5 ml, 200 mm) 1/set
010-20003-0-0	Silica small volume sample cell (0.5 ml, 200 mm) 1/set
010-20004-0-0	Pyrex large volume sample cell (MAX.500 °C, 0.5 ml, 200 mm) 3/set
010-20005-0-0	Silica large volume sample cell (5 ml, 200 mm) 1/set
010-20008-0-0	Pyrex flow gas sample cell (Max.500 °C,230 mm) 1/set
010-20009-0-0	Silica flow gas sample cell 1/set
010-22021-0-0	NSD capucel sample cell 1/set
010-22014-0-0	Pellet sample cell 1/set
【Others】	
010-21000-0-0	Glass rod (Pyrex sample cell, Pyrex flow gas sample cell: 010-20000-0-0,010-20008-0-0) 3/set (Pyrex)
010-21001-0-0	Glass rod (Silica sample cell, Silica flow gas sample cell: 010-20001-0-0,010-20009-0-0) (Silica)
010-21002-0-0	Glass rod for Pyrex large volume sample cell (010-20004-0-0) 3/set (Pyrex)
010-21003-0-0	Glass rod for silica large volume sample cell (010-20005-0-0) (Silica)
010-21006-0-0	Glass rod for Pyrex small volume sample cell (010-20002-0-0) 3/set (Pyrex)
010-21007-0-0	Glass rod for silica small volume sample cell (010-20003-0-0) (Silica)
010-21010-0-0	Glass rod for quick seal (010-20000-0-0) 3/set (Pyrex)
010-21011-0-0	Glass rod for quick seal (Pyrex large volume sample cell: 010-20004-0-0) 3/set (Pyrex)
010-21012-0-0	Glass rod for quick seal (Silica sample cell: 010-20001-0-0) (Silica)
010-21013-0-0	Glass rod for quick seal (Silica large volume sample cell: 010-20005-0-0) (Silica)
010-21008-0-0	Glass rod for pellet sample cel (010-22014-0-0)
010-22015-0-0	Pellet sample tube (Pellet sample cell: 010-22014-0-0)
900-00017-0-0	Sample cell heat insulation sleeve 3/set
010-22024-0-0	P0 Cell heat insulation sleeve 3/set
010-22003-0-0	Dewar vessel heat insulation lid

model	Item and Specification
【Others】	
010-22000-0-0	Sampling funnel 3/set (Pyrex)
010-22001-0-0	Liq. bottle 2/set
010-22002-0-0	P0 cell (SUS)
010-22022-0-0	P0 port cap (4 cm × 3φ)
900-00016-0-0	Sample cell cap 10/set
900-00011-0-0	Sample scattering prevention filter 6/set 20m (Viton)
900-00013-0-0	Sample scattering prevention filter 6/set 20m (Perflour)
900-20002-0-0	Sample scattering prevention filter (quick seal) 1/set (Viton)
900-20003-0-0	Sample scattering prevention filter (quick seal) 1/set (Perflour)
900-10005-0-0	Sample cell weighting stand
900-10004-0-0	Sample cell stand
900-00004-0-0	Perflour o-ring 6/set
900-00000-0-0	Viton o-ring for P0 cell (P-3) 12/set
910-00001-0-1	Corrosion resistant rotary pump (GHD-030(Fomblin oil))
910-00000-0-1	rotary pump (GHD-30)
010-10011-0-0	Dewar vessel
010-10022-0-5	Measurement site safety cover
910-20000-0-0	Standard oil for vacuum pump (1 L) 600 cc/unit (GHD-30)
SMR-100	Standard oil for vacuum pump (1 L) 600 cc/unit (Trivac, GLD-030, GLD-51)
Ultra Grade 19	Standard oil for vacuum pump (1 L) ・RV3
910-20002-0-0	Fomblin oil for vacuum pump (1 kg) (GHD-030(Fomblin oil))

Appendix

Adsorptive		Boiling point/K	Critical point/K	Temp. /K	Saturation vapor pressure/kPa*[1]	2nd virial coefficient/Pa^{-1}
Nitrogen	N_2	77.4	126.2	77.4	101.325*[3]	-4.264×10^{-7}
Argon	Ar	87.3	150.8	77.4	30.615*[6]	-4.587×10^{-7}
				87.3	101.325*[3]	-3.300×10^{-7}
				298.15	-	-6.093×10^{-7}
Methane	CH_4	111.6	190.4	298.15	24973*[4]	-1.689×10^{-7}
Ethane	C_2H_6	184.6	305.4	293.15	3553.0	-7.985×10^{-8}
				298.15	3916.2	-7.569×10^{-8}
				303.15	4301.9	-7.181×10^{-8}
Carbon dioxide	CO_2	194.7	304.1	194.7	101.325	-2.143×10^{-7}
				298.15	6444.8	-5.031×10^{-8}
Hydrogen	H_2	20.4	33.2	293.15	-	3.464×10^{-9}
				298.15	-	3.436×10^{-9}
				303.15	-	3.407×10^{-9}
Krypton	Kr	119.9	209.4	77.4	0.3310*[5]	-1.007×10^{-6}
Ammonia	NH_3	239.8	405.5	293.15	861.87*[2]	-1.217×10^{-7}
				298.15	1007.2*[2]	-1.132×10^{-7}
				303.15	1170.3*[2]	-1.056×10^{-7}
Oxygen	O_2	90.2	154.6	293.15	-	-6.845×10^{-9}
				298.15	-	-6.312×10^{-9}
				303.15	-	-5.814×10^{-9}
Nitrogen monoxide	NO	121.4	180	298.15		-3.319×10^{-9}
Carbon monoxide	CO	81.7	132.9	293.15	-	-3.530×10^{-9}
				298.15	-	-3.063×10^{-9}
n-butane	n-C_4H_{10}	272.7	425.2	293.15	208.23	-3.121×10^{-7}
				298.15	243.08	-2.950×10^{-7}
				303.15	283.14	-2.791×10^{-7}
iso-butane	iso-C_4H_{10}	261.4	408.2	293.15	300.51	-2.803×10^{-7}
				298.15	348.66	-2.652×10^{-7}
				303.15	284.18	-2.511×10^{-7}
Benzene	C_6H_6	353.2	562.2	298.15	12.778	-6.074×10^{-7}
Toluene	$C_6H_5CH_3$	383.8	591.8	298.15	3.822	-9.439×10^{-7}
Methanol	CH_3OH	337.7	512.6	298.15	17.050	4.290×10^{-7}
Ethanol	C_2H_5OH	351.4	513.9	298.15	7.958	5.429×10^{-7}
Cyclohexane	C_6H_{12}	353.8	553.5	298.15	13.100	-6.902×10^{-7}
				323.15	36.439	-5.199×10^{-7}
Carbon tetrachloride	CCl_4	349.9	556.4	298.15	15.323	-6.104×10^{-7}

ooo Physical Properties of Adsorptives ooo

*1 For the calculation of saturation vapor pressure, Antoine equation is used when 101.325 kPa or below and Wagner equation (partly Harlacher-Braun equation) is used when 101.325 kPa or above. The blank ones in the table are above the critical point.

*2 Harlacher-Braun equation is used.

*3 The saturation vapor pressure of nitrogen (77.4 K) and argon (87.3 K) is just the pressure at the boiling point. So for them, the 2nd virial coefficient at the atmospheric pressure is listed.

*4 Since carbon dioxide does not exist as a liquid state under the atmospheric pressure, the sublimation pressure is listed as the saturation vapor pressure.

*5 Krypton is solid at 77.4 K. The saturation vapor pressure is obtained from Clausius-Clapeyron equation as a super cooled liquid (ASTM D 4780-95).

*6 Argon is solid at 77.4 K. The saturation vapor pressure is obtained from Wagner equation as a super cooled liquid (Gasses & Liquids 4th Edition Robert C. Reid).

ooo Molecular diameter ooo

Adsorption		Molecular diameter(nm)*6
Nitrogen	N_2	0.364-0.37
Argon	Ar	0.355-0.36
Methane	CH_4	0.409
Hydrogen	H_2	0.268-0.269
Krypton	Kr	0.404-0.408
Oxygen	O_2	0.392-0.398

*6 T. Takaishi and Y. Sensui, 'Thermal Transpiration Effect of Hydrogen, Rare Gasses and Methane' Trans. Faraday Soc., **59**, 2511 (1963).

ooo Calculation of Saturation Vapor Pressure ooo

The saturation vapor pressure of pure substances is listed in various literatures. For instance, there are "Physical constant" edited by the society of chemical engineers, and "Physical constant of chemical engineering". In the table of physical properties of adsorptives, the saturation vapor pressure of common substances is listed. Also, Figure 1 shows the vapor pressure of water at various temperatures.

Figure 1: Saturation vapor pressure of water

θ/°C	**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
0	0.611	0.657	0.706	0.758	0.813	0.872	0.936	1.002	1.073	1.148
10	1.228	1.313	1.402	1.498	1.599	1.705	1.818	1.938	2.064	2.198
20	2.338	2.487	2.644	2.810	2.985	3.169	3.362	3.567	3.781	4.007
30	4.245	4.495	4.757	5.033	5.322	5.626	5.945	6.279	6.629	6.996
40	7.381	7.784	8.205	8.646	9.107	9.589	10.093	10.620	11.170	11.745
50	12.344	12.970	13.623	14.303	15.012	15.752	16.522	17.324	18.159	19.028
60	19.933	20.873	21.852	22.869	23.925	25.024	26.164	27.348	28.578	29.854
70	31.177	32.550	33.974	35.450	36.980	38.565	40.207	41.908	43.668	45.490
80	47.376	49.327	51.345	53.432	55.589	57.819	60.123	62.503	64.962	67.501
90	70.122	72.827	75.619	78.500	81.471	84.535	87.694	90.951	94.307	97.765

* Wargner equation is used (Gasses & Liquids 4th Edition Robert C. Reid).

There are many suggested equations to estimate the saturation vapor pressure. The representative equations are: Antoine equation, Harlcher-Braun equation, and Wagner equation.

Antoine equation

This is often used when the converted temperature ($T_r = T/Tc$ where Tc is the critical temperature) is less than 0.75 and the pressure is 0.1-200 kPa. At the temperature T, the saturation vapor pressure P_0 is expressed as the following with the constant A, B, and C:

$$\log P_0 = A - \frac{B}{C + T[\mathrm{K}]}$$

Wagner equation

McGarry compared 8 vapor pressure equations and found out Wagner equation is the best estimation of vapor pressure up to the critical point. At the temperature T, the saturation vapor pressure P_0 is expressed as the following with the constant A, B, C, and D:

$$\ln(P_0 / P_c) = (Ax + Bx^{1.5} + Cx^3 + Dx^6)/(1-x)$$

where

$$x = 1 - T/T_c$$

and T_c and P_c are the critical temperature and critical pressure respectively.

Harlcher-Braun equation

This equation estimates the vapor pressure well from the boiling point to the critical point. At the temperature T, the saturation vapor pressure P_0 is expressed as the following with the constant A, B, C, and D:

$$\ln P_0 = A + B/T[\mathrm{K}] + C\ln(T[\mathrm{K}]) + \frac{DP_0}{(T[\mathrm{K}])^2}$$

The constants are listed in the Chemistry handbook or other references.

There are many other equations suggested to estimate the saturation vapor pressure, but the estimation values do not necessarily coincide with the actual values.

Reference:

R.C. Reid, J.M.Prausnitz and B.E. Poling, "The Properties of Gases & Liquid", 4th ed. McGraw-Hill, Inc.

ooo Calculation of the 2nd virial coefficient ooo

In the volumetric method, the volume of adsorption is determined by using the ideal gas equation. The virial coefficient allows us to correct for the nonideality of real gases. By using the 3rd and 4th virial coefficient, it is possible to perform correction in the wide range of pressure, but here, the calculation of 2nd virial coefficient is discussed in detail. If you want to calculate the 3rd and 4th virial coefficient, please refer to other scientific sources since there should be plenty of information in them. Please enter B_p (Pa^{-1}) value to correct for the nonideality.

For single component gases, the 2nd virial coefficient is calculated by using the following equations:

$$\frac{BP_C}{RT_C} = B^{(0)} + \omega B^{(1)} + B^{(2)}$$

$$B^{(0)} = 0.083 - \frac{0.422}{T_r^{1.6}}$$

$$B^{(1)} = 0.139 - \frac{0.172}{T_r^{4.2}}$$

$$B^{(2)} = \frac{a}{T_r^6} - \frac{b}{T_r^8}$$

$$T_r = \frac{T}{T_C}$$

$B^{(2)}$ is the term for polar molecules. This is reported by Tsonopoulos. a, b cannot be determined accurately, so they are only classified into some values.

Class	Compound	a	b
1	Ketenes, aldehydes, nitrides, Ethers, NH_3, H_2S, HCN, esters	$-2.112 \times 10^{-4}\mu_r - 3.877 \times 10^{-21}\mu_r^8$	0
2	Mercaptans	0	0
3	Monoalkyhalides	$2.076 \times 10^{-11}\mu_r^4 - 7.048 \times 10^{-21}\mu_r^8$	
4	Alcohols	0.0878	0.04-0.06
5	Phenol	-0.0136	0

μ_r in the table is determined by the following equation:

$$\mu_r = \frac{10^5 \mu^2 P_C}{T_C^2}$$

P_C : Critical pressure / bar

T_C : Critical temperature / K

μ : Dipole moment /debyes

ώ :Acentric Factor (Asymmetry factor for molecules)

Reference:

R.C. Reid, J.M.Prausnitz and B.E. Poling, “The Properties of Gases & Liquid”, 4th ed. McGraw-Hill, Inc.

ooo Pretreatment ooo

Pretreatment is performed to remove water and other molecules adsorbed on the surface of adsorbent and inside pores (for the pretreatment of chemisorption, it might involve the chemical reactions at the surface, e.g. oxidation and reduction).

As the pretreatment method:

1.Heat the sample under the flow of inactive gas (nitrogen, helium, etc.)

2.Heat the sample while in vacuum

In case of method 1, the structure of pretreatment instrument can be simple, but the heating time needs to be longer than method 2. Also, when cooling down the sample to the room temperature under the flow of gas, the impurities in the gas might re-adsorb inside the pores if the sample has micropores. If it happens, it influences the measurement badly. Pay attention to the purity of the gas you use. In case of method 2, it is generally enough to vacuum with a rotary pump (1 Pa). If you need to measure the adsorption isotherm at 10^{-2} Pa or less to analyze the pore size distribution with DFT theory or simulation, you need to achieve the better vacuum by using a turbo-molecular pump.

The higher temperature pretreatment is performed, the more efficient it can remove impurities. However, it is important not to change the structure of sample by putting too much heat. Ideally, one can investigate the effect of temperature increase to the sample by using a thermogravimetric balance. If the temperature cannot be set high (e.g. pretreatment of polymer materials), the pretreatment time needs to be longer. Carbon material does not change its structure even at high temperature. In case of active carbon, water molecules cannot be desorbed from the pores unless the temperature is high. Generally, pretreatment is performed at about 573 K. It is the same for zeolite, but the temperature needs to be increased gradually to avoid the structure change of pores by the thermo-hydration reaction.

The table below lists the typical pretreatment temperature of common substances. Determine the pretreatment temperature after considering the physical properties and surface characteristics of the substance you actually measure.

	Pretreatment temp. /°C	Remarks
Active carbon	150-500	There is possibility that surface functional groups desorb at 300 °C or higher, but it does not affect the surface area and properties of pores so much.
Zeolite	300-500	If performing the measurement from extremely low pressure, pretreatment needs to be performed for 5 hours or longer under the high vacuum.
Polymer	r. t. -100	If the temperature cannot be increased, it takes longer time for pretreatment.
Alumina	100-150	At 150 °C or higher, the surface hydroxyl might be condensed or dehydrated.
Silica	100~150	At 200 °C or higher, the surface hydroxyl might be condensed or dehydrated.
Titania	100-500	By heating under the vacuum, it might produce reducing atmosphere and the surface area might be changed because of desorption of oxygen at the surface. It is desirable to perform pretreatment under the flow of high purity air or oxygen.
Calcium carbonate	200-400	It is thermally decomposed around 900 °C.
Cement	100-120	Because of the hydrate, dehydration reaction occurs during the high temperature pretreatment.