MICROTRAC MRB

PARTICLE CHARACTERIZATION


# BELSORP SERIES

## GAS & VAPOR SORPTION INSTRUMENTS

## CHARACTERIZATION OF POROUS MATERIALS

part of VERDER scientific

MICROTRAC MRB

# INDEX

MICROTRAC MRB

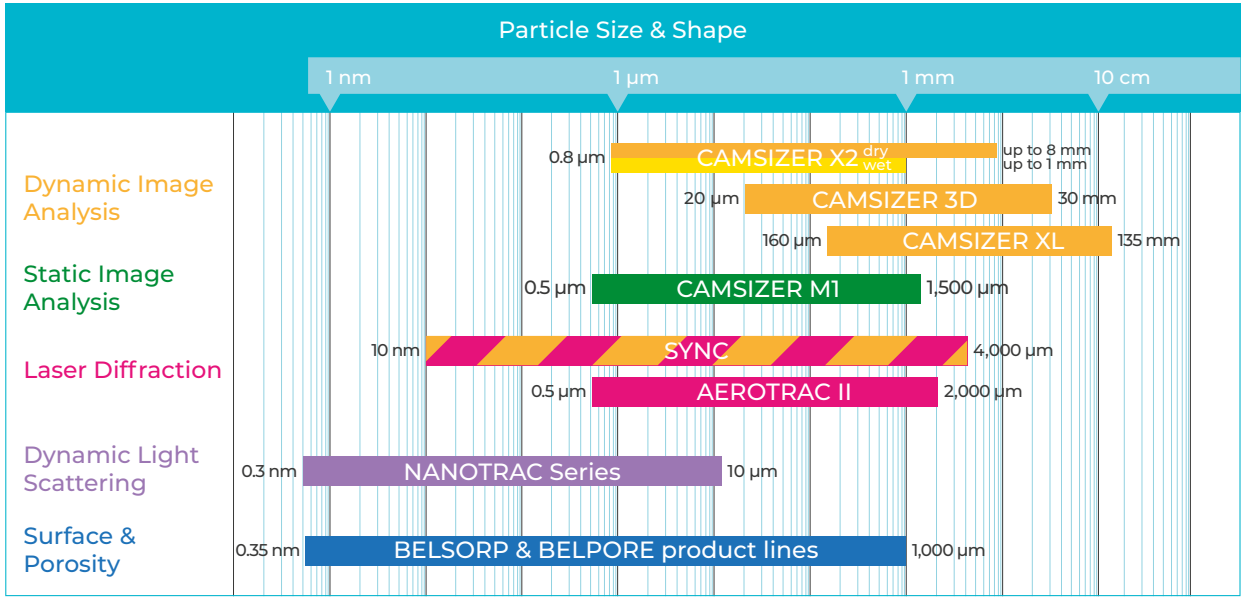
# PARTICLE CHARACTERIZATION AT ITS BEST


**Microtrac MRB** is your preferred partner for the comprehensive characterization of particulate systems. We provide our customers with advanced technologies to obtain consistently reliable results. Innovation and quality form the basis of our business.

As part of the Verder Scientific Group we provide worldwide support through a network of subsidiaries and distributors in every country.

MICROTRAC MRB

# THREE PILLARS OF EXCELLENCE




Microtrac MRB offers three product lines with centers of excellence on three continents.

**I Surface & Porosity**

Specific surface area (BET) and porosity of powders and solids are determined by gas adsorption or mercury intrusion method. The competence centers for these product lines are located in Osaka, Japan and Haan, Germany.

**I Scattered Light Analysis:**

Microtrac MRB is a leading supplier of both dynamic and static light scattering systems for particle size determination. The portfolio includes laser diffraction as well as dynamic light scattering instruments perfectly suited for the characterization of nano particles. The development and production site for this product line is located in Pennsylvania, USA.

**I Image Analysis:**

With the CAMSIZER series Microtrac MRB provides high-quality systems for the determination of particle size and particle shape based on both static and dynamic imaging. These instruments are developed and built in our production site in Haan, Germany.

MORE THAN 30 YEARS

# THE HISTORY OF THE BELSORP SERIES

## 1991

**BELSORP 28 SA**

Japanese 2nd generation automatic gas adsorption system

## 2001

**BELSORP MINI & MINI II**

3rd generation instrument, 1st model with Advanced Free Space Measurement (AFSM)

## 2006

**BELSORP MAX**

World's 1st model with 0.1 Torr pressure sensor for micropore investigation

## 2016

**BELSORP MAX II**

First model (4th generation) with Gas Dosing Optimization (GDO)

VERDER scientific

## 2019

**ACQUISITION**

MicrotracBEL, Microtrac Inc and Retsch Technology merge as part of Verder Sc.

## 2023

**BELSORP MAX X**

Release of high-end gas / vapor sorption analyzer with smallest footprint

## 1987

**BELSORP 28**

Japanese 1st generation automatic gas adsorption system for BET, PSD, etc.

## 1995

**BELSORP 18**

World's 1st vapor adsorption measurement using the volumertirc method

## 2003

**BELSORP AQUA 3**

High precision vapor sorption measurement of 3 samples simultaniously

## 2013

**BELSORP MR SERIES**

Gas adsorption measurement using the dynamic gas flow method

## 2018

**BELSORP MINI X**

World's smallest and most lightweight gas adsorption instrument

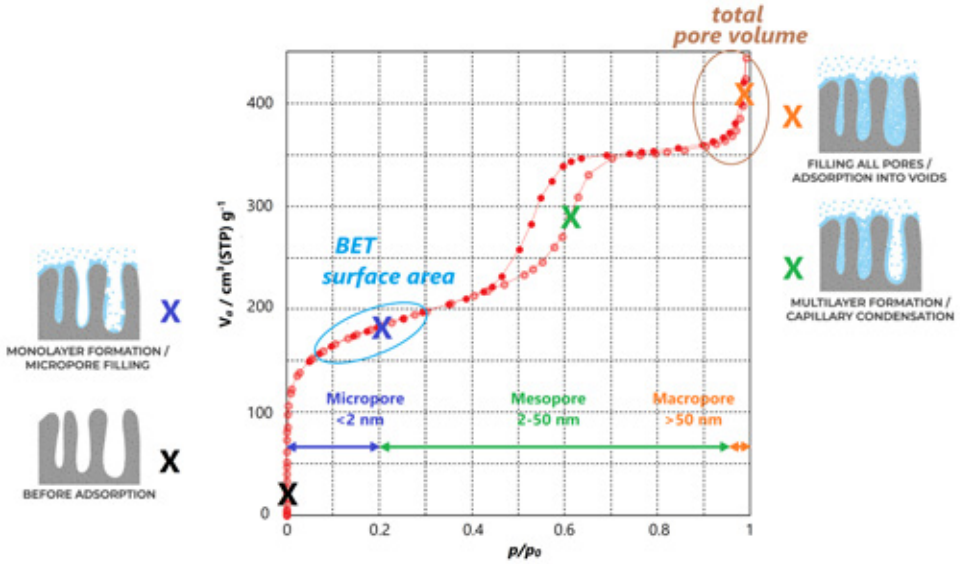
## 2020

**BELSORP MAX G**

Compact gas adsorption instrument for micropore analyses

BASIC ADSORPTION PRINCIPLES

# THE BASICS OF ADSORPTION & ADSORPTION ISOTHERM

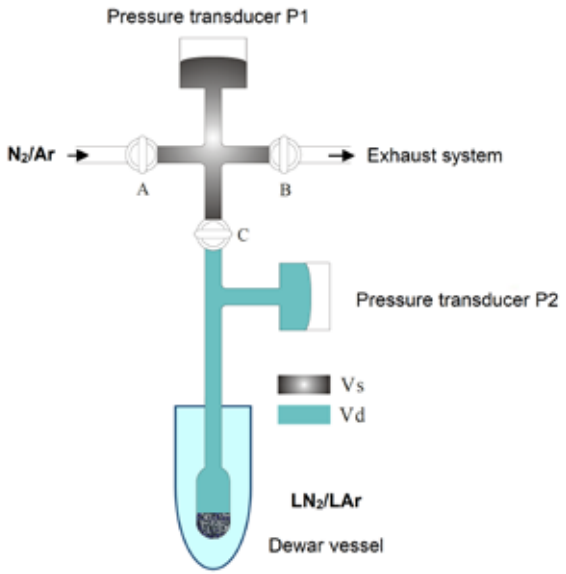



The adsorption isotherm is defined as the relationship between the adsorbed amount of an adsorbent and the equilibrium pressure of a gas or vapor at a constant temperature. The adsorbed amount is depicted on the vertical axis and related to the mass of the adsorbent, whereas the pressure is represented on the horizontal axis and usually represented as a relative pressure, namely the equilibrium pressure related to the saturated vapor pressure. The pressure thus ranges from “0 to 1”. The relative pressure of “0” describes the state before adsorption (i.e. after pretreatment), while “1” describes the state after all pores have been filled (saturated state). In general, by measuring adsorption isotherms such as $N_2$ at 77 K and Ar at 87 K, the specific surface area can be obtained from BET theory in the relative pressure range of 0.05 to 0.30. This range can be extended to values below 0.05 for microporous materials. The pore size distributions can also be calculated from the sorption isotherm, using different ranges of relative pressures depending on pore size and evaluation method. Typically, micropores (≤ 2 nm) are characterized at $p/p_0 \leq 0.20$, mesopores (2-50 nm) at $p/p_0 = 0.20 - 0.97$. Finally, macropores (≥ 50 nm) are evaluated from more than $p/p_0 = 0.97$. In recent years, we have been able to analyze the entire pore size range up to several 100 nm using statistical thermodynamics models (NLDFT & GCMC methods) in a single theory.

The figure above shows the nitrogen adsorption isotherm (77 K) of an SBA-15 ordered mesoporous silica. Significant increases in the amount of adsorption were observed at relative pressures of 0 - 0.05 and 0.40 - 0.70, indicating the presence of micro- and mesopores.

## BASIC ADSORPTION PRINCIPLES

# VOLUMETRIC (MANOMETRIC) METHOD



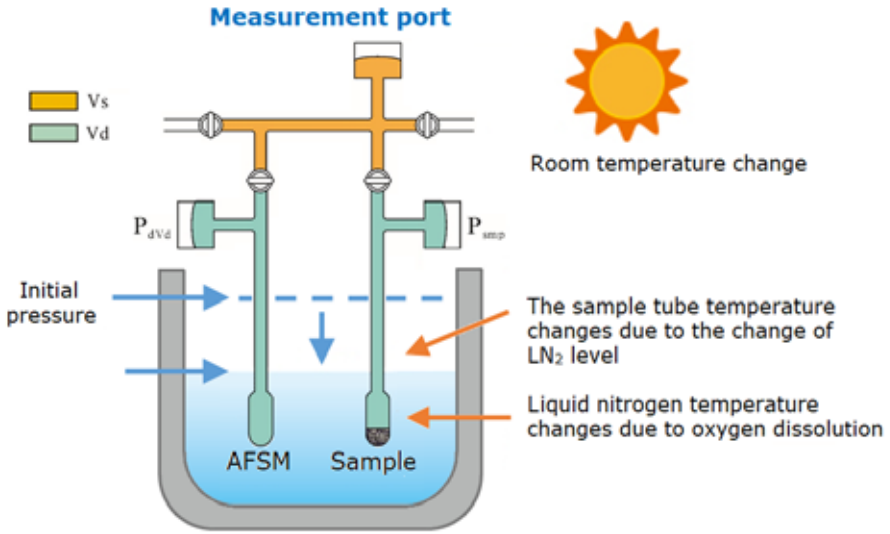

The accurate measurement of an adsorption isotherm is essential for determining the specific surface area, pore size distribution, pore volume, adsorption rate, and surface properties of various non-porous and porous materials. The principles of gas adsorption methods are divided into volumetric, gravimetric, pulse adsorption and dynamic methods. Instruments based on the volumetric method – the most common method for adsorption analysis – must be equipped with an adsorbate gas dosing function, pressure transducers (P1, P2), a vacuum pump and valves.

First, the sample is filled into the sample cell and pretreated at a suitable temperature (heat and vacuum). Then, the sample cell is transferred to the measurement port (if pretreated externally) and the system is evacuated. To keep the cryogenic temperature constant, a refrigerant such as liquid nitrogen or liquid argon is used and filled into a Dewar vessel.

In the volumetric system, the adsorbed amount is calculated from the pressure change before and after adsorption based on the non-ideal gas equation. A certain gas dosing quantity with pressure ($p_i$) is filled into the manifold with known volume ($V_S$: standard volume of the respective device). The valve C to the sample port is opened and the pressure ($p_e$) is measured after reaching equilibrium. From the pressure difference between $p_i$ and $p_e$ and the free space ($V_d$), the adsorbed volume can be calculated. The process described above is repeated at different pressures so that an adsorption isotherm is obtained. For each measurement point the actual free space has to be considered, which is accurately determined by our patented AFSM™ technology.

## BASIC ADSORPTION PRINCIPLES

# ADVANCED FREE SPACE MEASUREMENT METHOD: AFSM™




When measuring the adsorption isotherm (adsorbed amount), it is not only necessary to accurately measure the adsorption amount, but also to ensure fast and high reproducibility. The actual measurement of the smallest changes in free space $V_d$ due to refrigerant evaporation is especially important when the specific surface area is small. MICROTRAC MRB's patented AFSM™ (Advanced Free Space Measurement) method enables accurate and fast measurements even for materials with small surface areas – with the highest reproducibility worldwide.

The free space in the sample cell gradually changes with the level of the refrigerant. Typically, it is determined at the beginning or end of the measurement and an attempt is made to keep it constant throughout. In this conventional method, several factors affecting $V_d$ such as variations in the liquid refrigerant level, dissolution of $O_2$, changes in room temperature and ambient pressure during the measurement cannot be taken into account. Thus, the amount of adsorption will not be accurately evaluated. Our patented AFSM™ is a groundbreaking method for the constant measurement of free space $V_d$ during adsorption measurement. With AFSM™, an initial free space of both the sample cell and the reference cell is determined simultaneously. Since the change in free space in the sample and reference cells is the same, the free space change is continuously tracked across the reference cell. Therefore, AFSM™ allows the adsorbed volume to be calculated based on the measured free space at any point without the need to keep the liquid level of the refrigerant constant and also taking into account all the ambient changes.

BASIC ADSORPTION PRINCIPLES

# AFSM™ VERSION 2: NEW & EFFICIENT

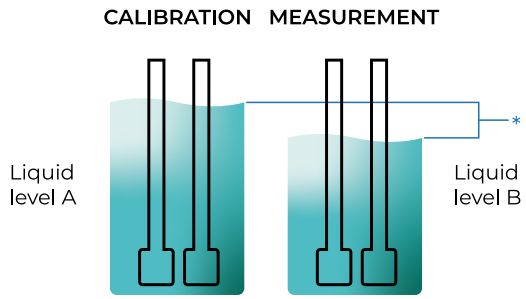
## Adsorption Definitions

Absolute Excess Net

**He-gas-free, short-time measurement**

Measurement techniques for determining free space often use calculated values of free space at both room temperature and measurement temperature of each sample tube (including the volume reduction filling rod and filter) and the true density of the sample.

With the new technique "AFSM™2", although the liquid level is not always the same during calibration and measurement (liquid levels A and B in the figure), the change in free space is the same for both conditions. This new method takes advantage of a highly reproducible AFSM and free space determination that eliminates the need for He gas. This makes it possible to obtain the highest repeatability in the world without the need for He gas.



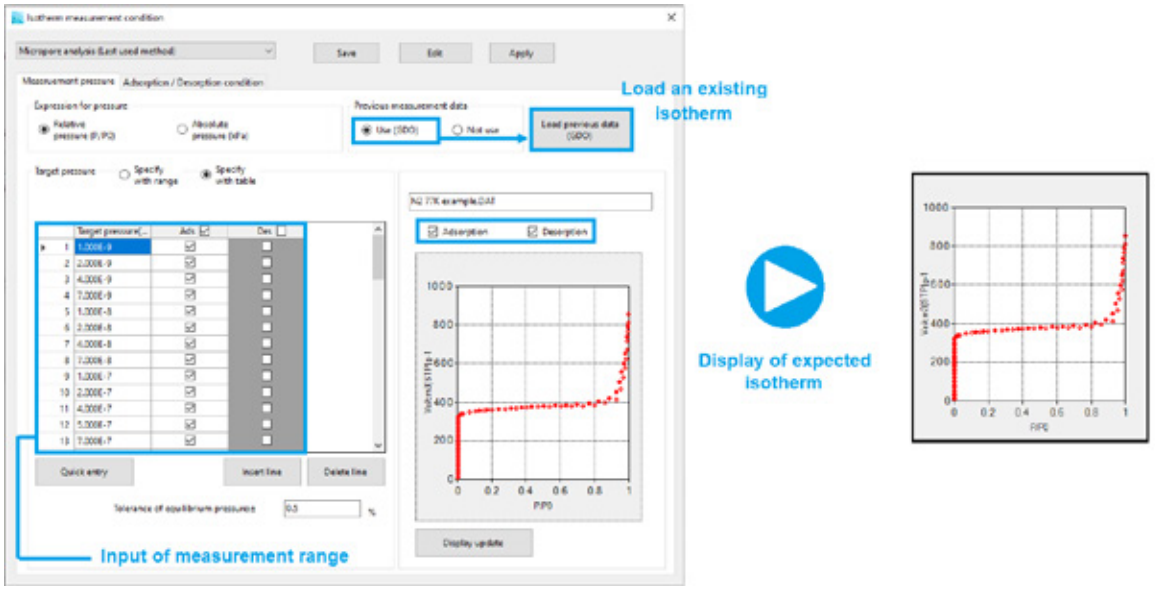

* The difference in free space is calculated by the AFSM technique.

### AFSM™2 Features

- World-class analysis method of adsorbed quantity with the same accuracy as conventional AFSM™
- He gas not required with AFSM™2
- Elimination of He adsorption and outgassing during measurements of microporous materials
- No effect on measurement accuracy of adsorbed amount due to temperature fluctuation (oxygen dissolution)
- Direct measurement of net adsorption
- Accurate evaluation of storage volumes

BASIC ADSORPTION PRINCIPLES

# GAS DOSING OPTIMIZATION (GDO)




### Gas Dosing Optimization

Gas Dosing Optimization (GDO) is an effective function that allows to measure with optimal conditions by using the previous adsorption isotherm data for the sample.

By utilizing GDO, the measurement isotherm can be configured easily by adding and deleting measurement points. This makes it easier for the user to automatically determine the amount of gas to be introduced – a previously cumbersome process, thus enabling short-term measurements.

### Feedback Valve Control for Gas Dosing

By detecting the gas dosing rate in conjunction with the installation environment (secondary pressure of supplied gas cylinders; He, $N_2$, etc.) before the measurement, it is now possible to reduce the measurement time through device-specific optimal valve control.

### Reduction of measurement time by GDO

| | Simple | GDO | Reduction |
|---|---|---|---|
| **Meso-porous** | 34 hrs | 19 hrs | **44%** |
| **Micro-porous** | 46 hrs | 20 hrs | **57%** |

## Summary of BELSORP Features

- **Precise measurement of the adsorption isotherm according to the volumetric method**
- **High reproducibility and repeatability with Advanced Free Space Measurement method (AFSM™)**
- **Short-time measurement with AFSM™2, no He-gas required**
- **Faster measurement through adsorbate gas dosing optimization function (GDO)**

## BELSORP MINI X

# SMALLEST & LIGHTEST IN THE WORLD

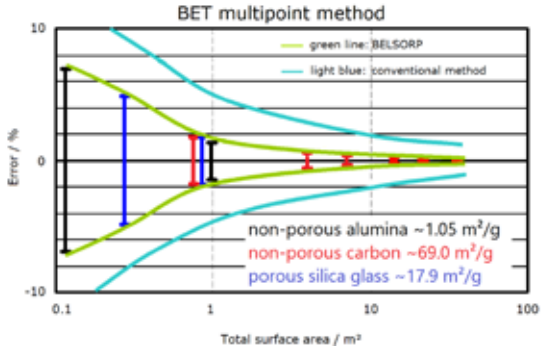
- 4 independent measurement ports and one dedicated port for saturated vapor pressure measurements
- Dedicated pressure transducers for each ports
- High-precision measurement with AFSM™
- Quick BET mode for high throughput
- Simultaneous control of up to 20 measurement ports via multi-device control (5 units)
- IoT: Process monitoring via e-mail notification system
- Gas adsorption isotherm & NET adsorption measurement through AFSM™2 without the need of He-gas
- Optional micropore analysis by molecular probe method
- Optional FDA 21 CFR Part 11 compliance


## BELSORP MINI X Features

- **The BELSORP MINI X is available as 3 or 4 port model**
- **Specific surface area range:**
  I 0.01 $m^2/g$ or more ($N_2$)
- **Pore size distribution range:**
  I 0.7 to 500 nm (opt. ~0.35 nm)
- **Three modes are available:**
  I High-precision mode for R&D
  I Quick BET mode for QC
  I Multi-sample mode and GDO for high throughput

Microtrac MRB's BELSORP MINI X shows outstanding features resulting into the world's highest repeatability with significantly reduced measurement time. The instrument is equipped with up to 4 sample measurement ports and new high-throughput functions including multi-device control. Equipped with dedicated pressure sensors on each sample measuring port and a dedicated port for saturated vapor pressure, it enables completely independent simultaneous measurements. In addition, the new measurement software improves user productivity by displaying the measurement progress, grasping the maintenance timing, and sending the measurement results via e-mail. Further, the new analysis software, BELMASTER (Ver. 7), enables the structural evaluation of a wider range of materials than ever before. The BELSORP MINI X allows measuring specific surface area, pore size distribution and total pore volume.

Further, all Microtrac MRB sorption instruments are equipped with a diagnosic tool for service matters. The System Check proofs the functionality of the main parts and the equipment status. The result will be saved as a report which summarizes leakage rates, the functionality of single parts, and more.

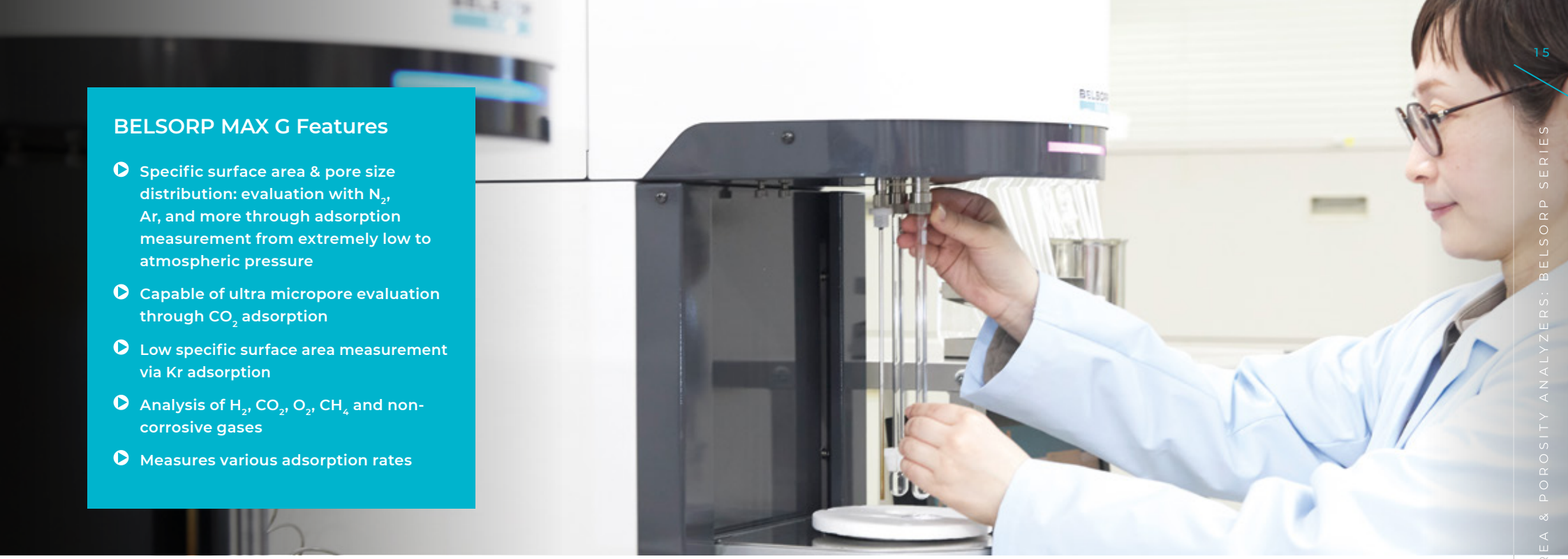



BELSORP high precision determination of BET surface areas

## BELSORP MAX G

# HIGH PRECISION GAS ADSORPTION ISOTHERM

- Highly reproducible BET specific surface area and pore size distribution evaluation from extremely low pressure
- Low BET specific surface area by Kr gas measurement at 77.4K
- Porosity from micro- to meso- and macropores by gas adsorption measurement of $N_2$, Ar, $CO_2$ and more
- High performance PSD analysis by GCMC & NLDFT with the BELMASTER (Ver. 7) software
- Actual and short-time evaluation for each adsorption point by Gas Dosing Optimization (GDO) function
- Gas and NET adsorption measurement via AFSM™2, without the need for He gas
- Optional vacuum gauge to monitor ultimate vacuum degree
- IoT: Process monitoring via e-mail notification system



## BELSORP MAX G Features

- Specific surface area & pore size distribution: evaluation with $N_2$, Ar, and more through adsorption measurement from extremely low to atmospheric pressure
- Capable of ultra micropore evaluation through $CO_2$ adsorption
- Low specific surface area measurement via Kr adsorption
- Analysis of $H_2$, $CO_2$, $O_2$, $CH_4$ and non-corrosive gases
- Measures various adsorption rates

BELSORP MAX G is a new range of powerful, compact and economical models in the BELSORP MAX series by Microtrac MRB. Its special feature is the measurement of gas adsorption isotherms starting from extremely low pressures for the evaluation of micro-, meso- and macroporous materials, as well as non-porous materials. This instrument is equipped with one measurement port, one dedicated port for saturated vapor pressure measurement and one port for free space measurement. Each port is equipped with a dedicated pressure sensor for high-precision measurements.

The BELSORP MAX G surface area & pore size distribution analyzer is capable of measuring various materials such as pellets, molded bodies, substrates, and finely dispersed samples using special-purpose sample tubes. Additionally, it is possible to mount a sample tube with an outer diameter of 9 mm or more on the measurement port. The BELSORP MAX G supports a wide range of adsorbates and measurement conditions.

Depending on our customers' needs, we are offering two models, namely the BELSORP MAX G LP (low pressure) and the BELSORP MAX G MP (medium pressure), which are both equipped with different pressure transducers:

| | BELSORP MAX G LP | BELSORP MAX G MP |
|---|---|---|
| **Port 1** | 133 kPa<br>1.33 kPa<br>13.3 kPa | 133 kPa<br>1.33 kPa<br>13.3 kPa |
| **Port 2** | 133 kPa | |
| **Saturation vapor pressure port** | 133 kPa | |
| **Turbomolecular pump** | ✓ | ✓ |

BELSORP MAX G models and their configurations

## BELSORP MAX X

# HIGHLY ACCURATE GAS & VAPOR ADSORPTION

- Smallest footprint: more compact design, lower weight
- Highly reproducible BET specific surface area and pore size distribution evaluation
- Highest throughput with simultaneous measurement of up to 4 samples
- Advanced Free Space Measurement: AFSM™ and AFSM2™ (Helium-free)
- Low specific surface area evaluation by Kr adsorption at 77.4 K
- Evaluation of hydrophilic and hydrophobic material
- Adsorption rate measurement for various gases and vapors
- Supports a wide range of gas / vapor adsorbates and measurement conditions
- Chemisorption option
- Measures various materials such as molded bodies, pellets, and fine powders

The BELSORP MAX X is a versatile instrument that measures specific surface area, pore size distribution, vapor adsorption, and chemisorption. The instrument allows for comprehensive surface characterization, such as BET surface area and micropore analysis, by measuring the adsorption isotherms from extremely low pressures, organic vapor sorption or hydrophilicity / hydrophobicity characterization through water vapor adsorption.

These capabilities are accomplished by the proprietary technical advantages of heated manifold blocks (50°C, opt. 80 °C) for a constant ambient temperature, heated air bath, and electropolished manifold lines to avoid surface wetting and corrosion. Furthermore, the BELSORP MAX X features pneumatic valves to minimize leakages or outgassing when working with high vacuum.

The BELSORP MAX X not only supports a wide range of gas and vapor adsorbates, but various measurement conditions as well. In addition, the most suitable conditions for each measurement are automatically set based on the user's adsorption isotherm data through Gas & Vapor Dosing Optimization (GDO).

## BELSORP MAX X Features

- **Specific surface area range:**
  - 0.01 $m^2/g$ or more ($N_2$)
  - 0.0005 $m^2/g$ or more (Kr)
- **Pore size distribution range:**
  - 0.35 to 500 nm
- **Highly accurate vapor adsorption measurement under strict temperature control**
- **Advanced GCMC / NLDFT method offers higher resolution & more precise PSD analysis**
- **IoT: Measurement status & results remotely via e-mail system**


BELSORP MAX X

# SPECIAL MODELS OF THE BELSORP MAX X SERIES

**BELSORP MAX X HT**

The BELSORP MAX X HV is a special model, enabling various types of vapor adsorption (water vapor, VOCs, and more) at higher temperatures than the regular version. The manifold block can be heated up to 80°C, enabling a wider application range under more realistic conditions. The instrument is used in application fields such as:

- Cement, concrete and building materials
- Heat transformation / air conditioning
- Electrode battery (LiB)
- GDL fuel cells

**BELSORP MAX X HP**

The BELSORP MAX X HP has been added as a custom solution to the BELSORP MAX X product line to enable gas adsorption, BET surface area, pore size distribution, vapor adsorption, and the evaluation of adsorption rates at high pressure up to 900 kPa. The instrument is used in application fields like:

- Efficient utilization of $CO_2$
- Energy storage ($CH_4$ / $CH_3C_6H_{11}$ / $H_2$)
- Heat pumps
- Air separation material used in PSA / TSA

## Features of the BELSORP MAX X Models

### BELSORP MAX X HT

- Manifold block heated up to 80°C
- Vapor adsorption isotherm evaluation up to 70°C and up to 0.95 of relative pressure
- High resolution isotherms of polar or non-polar organic vapors

### BELSORP MAX X HP

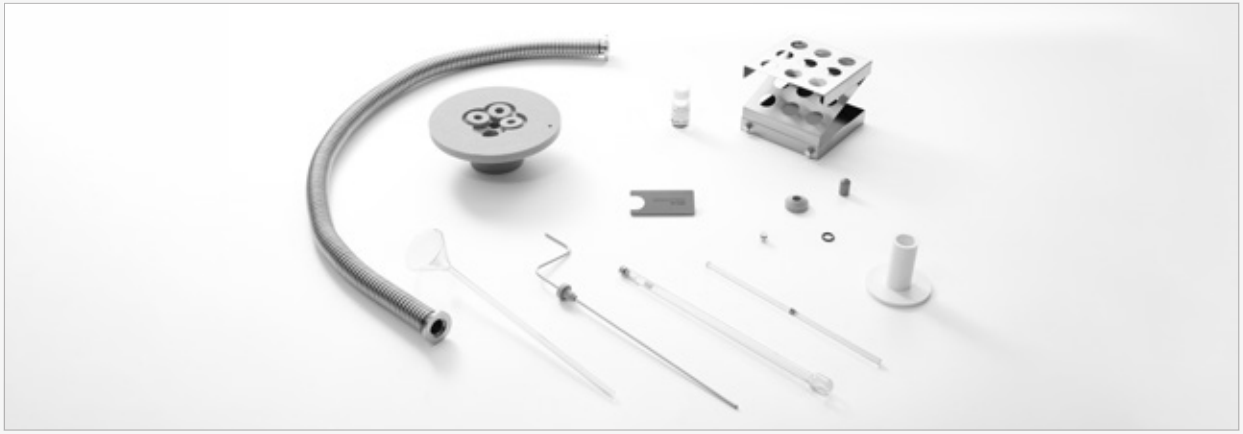
- Evaluation of adsorbed amounts of various gases up to 900 kPa
- Accurate adsorption quantity evaluation by automatic correction for non-ideality of various gases
- Pore sizes from ultramicropores to mesopores measured by CO2 (900 kPa at 298 K, GCMC)


| System | BELSORP MAX X | BELSORP MAX X HT | BELSORP MAX X HP |
|---|---|---|---|
| Measurement port | 4 ports maximum | 4 ports maximum | 3 ports maximum<br>1 port for high pressure |
| Measurement range (vapor adsorption) | $P/P_0$ = ~ 0.95 @ 40°C | $P/P_0$ = ~ 0.95 @ 70°C | $P/P_0$ = ~ 0.95 @ 40°C |
| Measurement range (high pressure adsorption) | - | - | 10 Pa ~ 900 kPa |
| Pressure transducer 1 MPa | - | - | 1 |
| Pressure transducer 133 kPa | 6 | 6 | 5 |
| Pressure transducer 1.33 kPa | 4 at maximum | 4 | 3 |
| Pressure transducer 13.3 Pa | 3 at maximum | - | 2 |
| Thermostatic chamber | 50°C | 80°C | 50°C |

04/2023 Subject to technical modifications and errors

BELSORP SERIES

# FURTHER OPTIONS & ACCESSORIES



### STANDARD CONSUMABLE GOODS

- Our standard consumables consist of sample cells, filler rods, filters, O-rings, caps and weighing platforms that are required for adsorption isotherm measurements. Further, NSD capsules, liquid bottles, various sizes of sample cells, quick seals, and much more are part of the consumable goods.

### HEATER & CONTROLLER

- Pretreatment of the sample from 50°C up to 550°C.

### WATER BATH

- Water bath for measurement temperature ranging from -10°C to 70°C. A refrigerated / heated circulator is required for usage.

### ACCESSORIES FOR VAPOR SORPTION

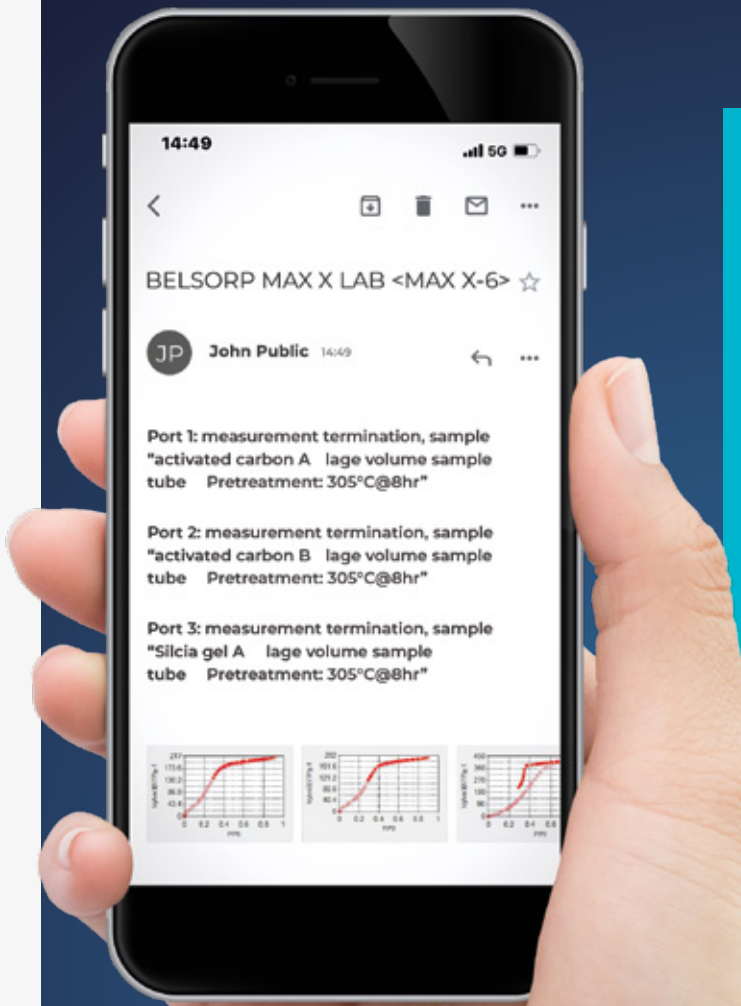
- Our accessories for vapor sorption include a detachable airbath, glas vessel for liquids, a reference sample for vapor sorption, and a Dewar for the degassing of liquids.

GAS SELECTORS

I Up to 12 gases (depending on the BELSORP model) can be mounted with external gas selectors to accommodate different types of adsorbates.

SAFETY COVER

I The safety cover for the BELSORP series increases the already high safety during measurements.




## IoT: The Internet of Things for our BELSORP product line

- **Measurement status and results remotely via e-mail notification system**
  - I sorption isotherms of all ports at a glance
  - I detailed sample information
  - I BELMASTER (Ver. 7) measurement files
  - I plotted graphs
- **Notifications can be sent to multiple recepients at once**
- **Labor productivity improvement**
- **Seamless measurement procedure**
- **Faster troubleshooting through automatic alerts**

## BELSORP SERIES

# BELCONTROL OPERATION SOFTWARE

The software has given the highest priority to simplify the operation and has been equipped with many functions to increase the labor productivity. Since the BELSORP instruments offer many features and possibilities, it gets more and more important to simplify the use. Our software will guide you step-by-step for the implementation of several procedures e.g. execution of measurements, replacement of gas cylinder, purging of the manifold and degassing of liquid adsorptive. This user-friendly feature is making the instrument accessible even for non-experienced users.

For the isotherm measurement conditions two possibilities are offered depending on the level of user-experience. Firstly, the 'automated setting' enables an easy operation by entering the sample information, selecting pretreatment conditions (skippable if externally done) and measurement points/ range. Therefore, it is ideal for measurement of unknown samples or unexperienced users. If a prior measurement with comparable sorption behavior is available, the GDO function can be used to reduce the measurement time. Secondly, the 'advanced setting' offers detailed configuration possibilities for control of dosing amounts and equilibrium criteria to optimize measurement conditions manually.

The e-mail notification automatically sends the measurement status and results as an e-mail. With this function easy and reliable monitoring will be given. Our instruments are equipped with a diagnostic service tool, the so-called System Check. It enables functionality proof of the main parts and the equipment status. The System Check result is saved as a report, summarizing the leakage rates, functionality of single parts.


I Control up to 5 units / 20 measurement ports with a single PC

## Software Features

- **Microtrac MRB's measurement operation software features a uniform user experience and can be used with BELSORP MINI X, MAX G, and MAX X**
- **The software offers automated and manual settings so that optimization can be made based on user experience**
- **Three sub modes are available:**
  - I High-precision mode for R&D
  - I Multi-sample mode for high throughput
  - I Quick BET mode for QC

### High Precision Mode

For high-precision measurements the amount of free space change in the sample section is simultaneously measured at the reference port (AFSM™). The other remaining ports are used for measuring the adsorption / desorption isotherms, while the saturated vapor pressure is constantly monitored with a dedicated port.

I Resolution: 0.01 $m^2$

I Reproducibility:

Total surface area 1.0 $m^2$ → ± 1.2%*

Total surface area 10 $m^2$ → ± 0.4%

### Multi-Sample Mode

This mode allows for measuring adsorption and desorption isotherms with up to four samples, while the saturation vapor pressure is constantly measured at the dedicated port. The free space change is automatically calculated from the prior saved free space file *(dvd)*.

I Resolution: 0.01 $m^2$

I Reproducibility:

Total surface area 10 $m^2$ → ± 0.5%

### Quick BET Mode

The quick BET mode can be used to maximize the sample throughput. In this mode it is possible to measure three BET adsorption points for four samples in approx. 15 minutes.

* The total surface area ($m^2$) is the product of both the specific surface area ($m^2/g$) and the sample mass.

## BELMASTER (VER. 7)

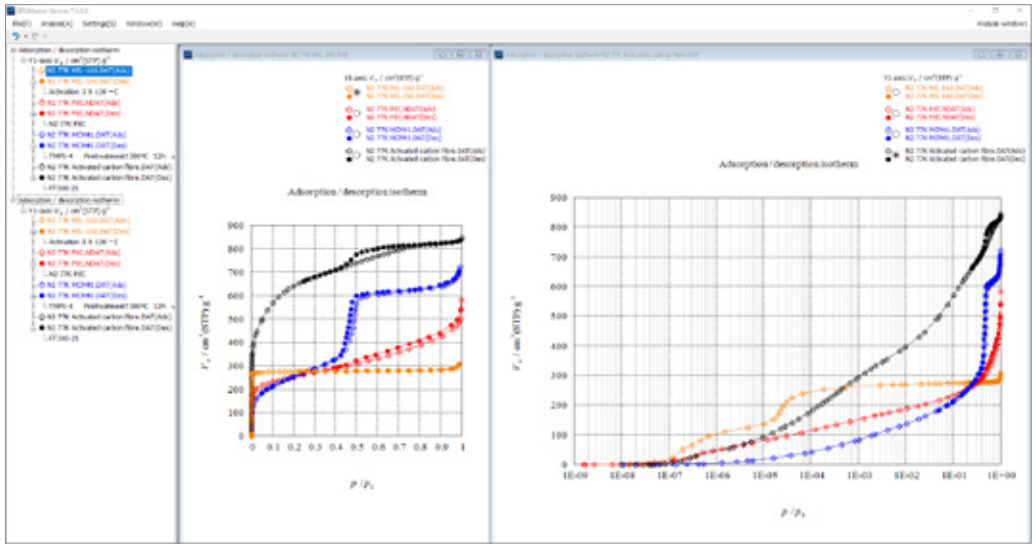
# POWERFUL & EFFICIENT SOFTWARE

- Analysis data and results can be saved by Drag & Drop (MS Excel format)
- Easy change of chart overwriting, X-Y axis scaling, unit conversion, point markers and color
- Analysis results window can be saved for further analysis after a computer restart
- Equipped with a routine analysis setting function, useful for performing the same analysis every time
- Customized data can be registered as standard reference isotherms in pore profile analyses, t-plot and αs
- Improved visibility for different analyses through individual color setting for custom data

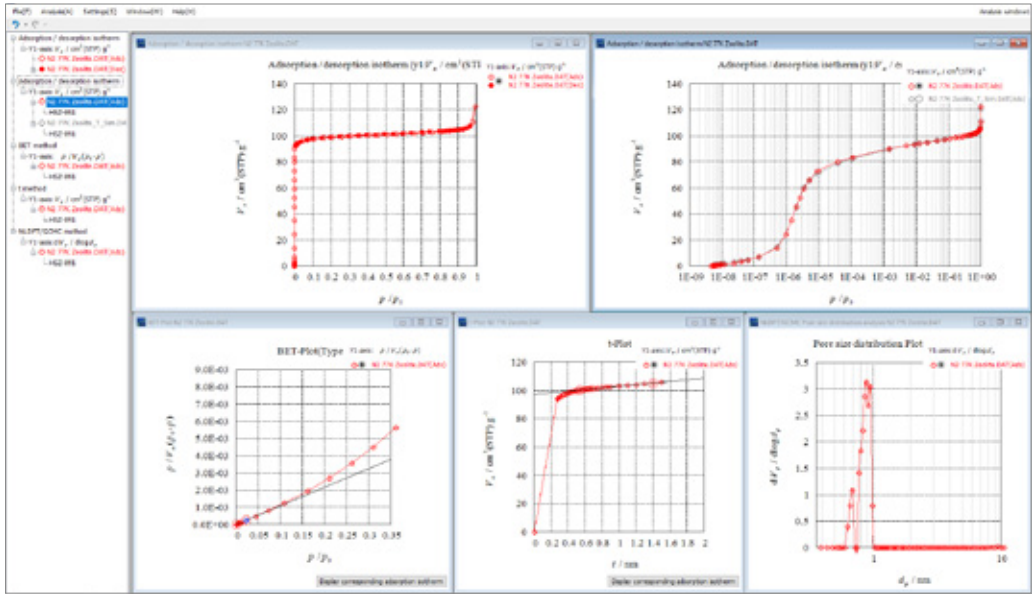
The evaluation software BELMASTER (Ver. 7) gives a wide range of basic and advanced analytical theories – developed over many years of experience – and offers the widest characterization of the samples.

- Adsorption-desorption isotherm / PCT curve
- BET Specific Surface Area, incl. ISO9277 / Rouquerol plot for Type I isotherms
- Langmuir & Freundlich specific surface area
- INNES, BJH DH & CI method (mesopores)
- HK, SF & CY method (micropore distribution, only for BELSORP MAX series)
- t-plot method (micro to mesopore analysis)
- αs plot method (micro to mesopore analysis)
- MP method (micropore distribution)
- Dubinin-Astakhov & Dubinin-Radushkevich method (micropore volume)
- Isosteric heat of adsorption (for MAX series)
- Differential adsorption isotherm
- Fractal dimension
- Molecular Probe Method (ultra micropore analysis)
- Adsorption rate analysis (option only available for MAX series)
- Metal dispersion
- BELSim™: NLDFT / GCMC (ISO15901-2) for micro-to-macropore distribution

- Isotherms starting from relative pressure of approx. $10^{-9}$

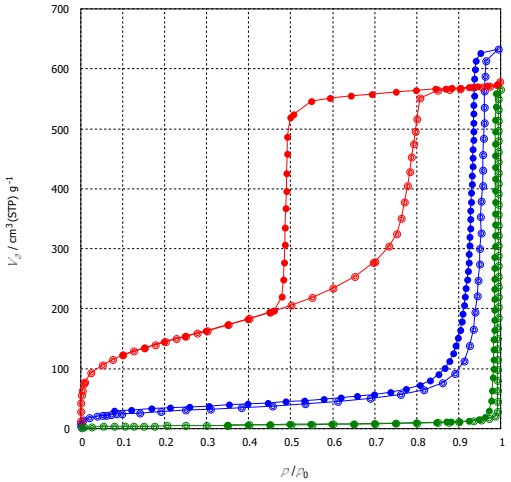


- Analysis results: Isotherm, BET (according to ISO 9277), t-plot and pore size distribution by GCMC



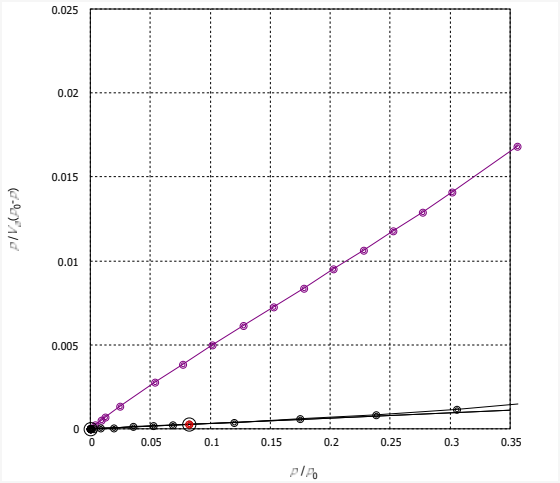
## MEASUREMENT RESULTS

# BELSORP MINI X



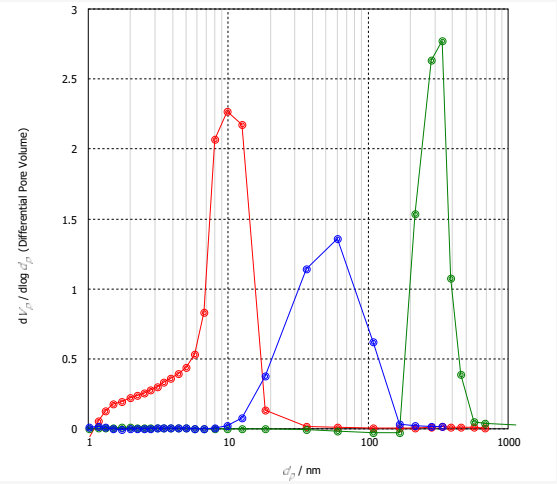

Nitrogen sorption isotherms of silica materials at 77.4 K

The adsorption isotherm is known as the relationship between the adsorbed amount on the adsorbent and the equilibrium pressure of a gas / vapour at constant temperature. The adsorbed amount is shown on the vertical axis and is usually related to the mass of the adsorbent, while the horizontal axis represents the relative pressure ($p/p_0$; p = equilibrium pressure and $p_0$ = saturation vapour pressure). In general, the sorption isotherm delivers information about the specific surface area, pore size distribution and pore volume.




BET plot: The specific surface area is usually determined by the BET method (named after Brunauer-Emmett-Teller) for physisorbed gases. The calculation is done according to ISO 9277

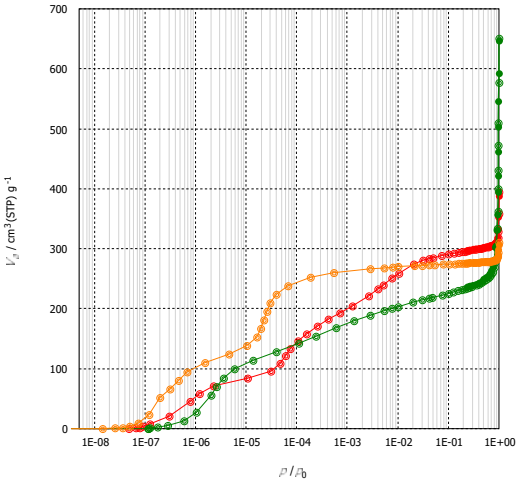
The classical pore size distributions (PSD) are the INNES method (slit shape) and BJH, DH, CI methods (cylinder shape), which evaluate mesopores based on the capillary condensation theory. HK (slit), SF (cylinder), and CY (cage) methods can also be used to evaluate micropores based on the adsorption potential theory. The DA method and DR method are also commonly used for pore volume evaluation as pore structure evaluation. The new PSD and capacity evaluation methods, NLDFT and GCMC, are described in detail on the next page and are specified in ISO 15901-2.



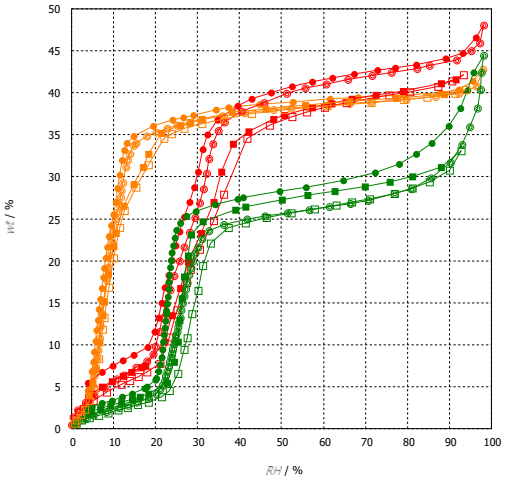

Classical BJH pore size distributions of silica materials based on nitrogen adsorption isotherms at 77.4 K

## MEASUREMENT RESULTS

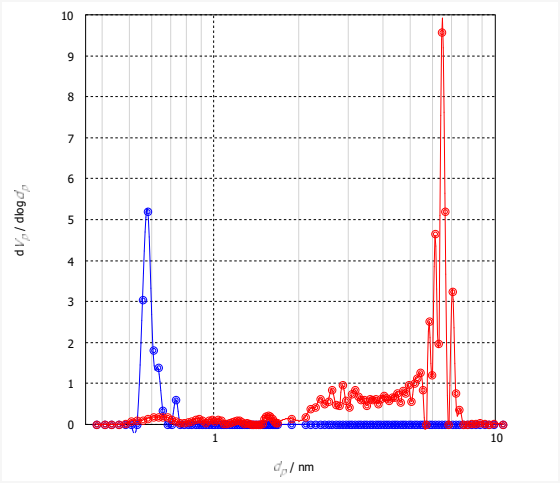
# BELSORP MAX G
# BELSORP MAX X




Nitrogen sorption measurements of the three metal-organic frameworks (MOFs): Aluminum-fumarate (green), UiO-66 (red) and MIL-160 (orange) at 77.4 K




Water sorption measurements of the three metal-organic frameworks (MOFs) at different temperatures: Aluminum-fumarate (green), UiO-66 (red) and MIL-160 (orange)




GCMC pore size distributions of SBA-16 (red) and MS-5A (blue) based on argon adsorption isotherms at 87.3 K

In recent years, attention has been focused on pore structure evaluation methods using computer simulations, such as the novel pore distribution analysis NLDFT (Non-localized Density Functional Theory) and GCMC (Grand Canonical Monte Carlo) method, which can measure micropores to meso- and macropores using a unified theory. Pore size distributions obtained from the same adsorption isotherm are different between classical and novel PSD analyses, and even in between novel methods, because the filling pressure obtained from each theory is different.

Microtrac MRB provides evaluation methods which cover a wide range of pore sizes and various adsorbates, such as $N_2$ (77.4 K), Ar (87.3 K), and $CO_2$ (298 K). It uses NLDFT / GCMC kernels of slit, cylinder, and cage pore models with carbon and metal oxide surface atoms, resulting in the most appropiate description of porous materials. Our BELMASTER software (Ver. 7) allows for the easy comparison between experimental and simulated isotherms, with the simulated isotherm serving as a basis for the PSD calculation. The similarity between them is an indicator for the correct PSD calculation.

## BELPREP VAC II & VAC III

# DEGASSER FOR VERSATILE SAMPLE PRETREATMENT

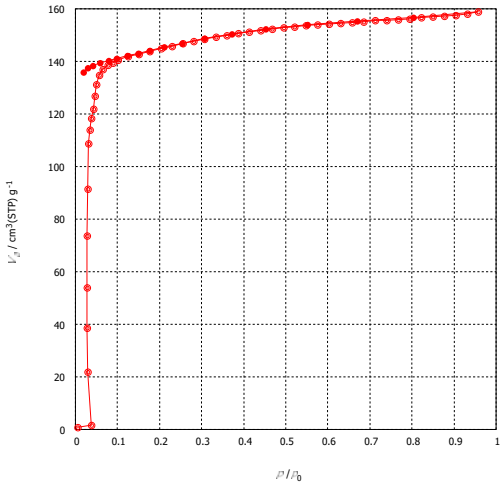
Accurate adsorption measurement requires material pretreatment. This can be done with an adsorption instrument's dedicated heater or externally with Microtrac MRB's BELPREP degassers. These independent heating pre-treatment instruments prepare the sample for analysis in a vacuum or inert gas stream. Using external pretreatment devices is often preferred to achieve a higher sample through-put, as pretreatment and measurement can be performed simultaneously. Depending on customer requirements, we offer two models: The BELPREP VAC II and BELPREP VAC III.

| Technical data | BELPREP VAC II | BELPREP VAC III |
|---|---|---|
| Flow / heat degassing | optional | optional |
| Vacuum / heat degassing | ✓ | ✓ |
| Pretreatment ports | 3 | 6 |
| Temperature range (maximum) | 430°C | 450°C |
| Temperature accuracy | ±5°C | ±5°C |
| Programmable temperature control function | ✓ 1 program, up to 8 pairs of ramp-soak | ✓ 8 programs, up to 32 segments each (ramps, soak, steps) |
| Automatic purge gas stop function | ✓ | - |
| Automatic vacuum pumping speed for dispersion prevention | ✓ | - |
| Dimensions (W x H x D) and weight | 321 x 158 x 363 mm, 15 kg | 400 x 317 x 383 mm, 15 kg |
| Power supply | AC 100-120 / 200-240 V (50 / 60 Hz) / 10 A | AC 100-120 / 200-240 V (50 / 60 Hz) / 12 A |

BELCRYO

# CRYOGENIC TEMPERATURE CONTROL UNIT

Microtrac MRB's BELCRYO enables the evaluation of material surface properties at cryogenic temperatures. This very reliable method supports the simultaneous measurement with optical devices (such as XRPD and SAXS), as well as the simultaneous measurement of gas adsorption behavior and structural changes. In fact, with the BELCRYO it is possible to measure the amount of adsorbed gas at the temperature of liquid oxygen (90.2 K), which was previously deemed a safety issue. The BELCRYO is also available for the evaluation of gas storage materials.



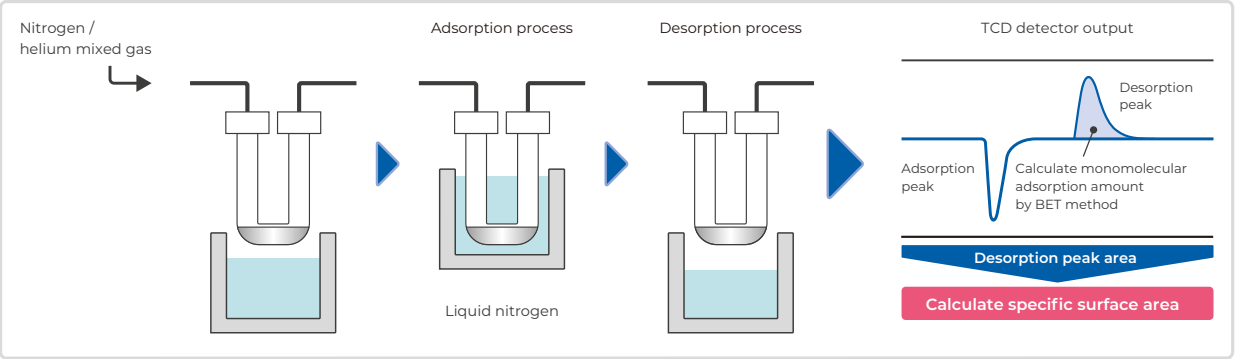

Exemplary oxygen sorption measurement of porous coordination polymer at 90.2 K

## BELCRYO Features

- Adjustable temperature control from cryogenic levels at 50 K to 473 K within 0.01 K
- Standard cell volume (1.8 $cm^3$) and small cell volume (0.5 $cm^3$) available
- Enables automatic measurement in combination with BELSORP MAX series
- Multiple sample units, up to 3 samples
- Support of high pressure analyses (0.9 MPa) with BELSORP MAX X HP
- $N_2$, $CO_2$, $O_2$, $H_2$, HCs, COs and other inactive gases

## BASIC ADSORPTION PRINCIPLES

# DYNAMIC GAS FLOW METHOD




In the dynamic gas flow method, a known concentration of an adsorptive gas with helium as a carrier gas is passing over a sample at a constant rate. Typically, 30% of nitrogen gas which is diluted with helium ($p/p_0$ = 0.30) is utilized. For the adsorption process a dewar with liquid nitrogen is moved up to cool the sample tube. Nitrogen gas is adsorbed by the sample. The concentration of nitrogen in the gas mixture decreases, resulting in a negative peak in the detector signal (TCD). When the sample is saturated, the detector signal returns to the baseline and the adsorption step is finished.

The liquid nitrogen Dewar is lowered and the nitrogen molecules start to desorb. As a result, the concentration of nitrogen in the gas mixture increases, resulting in a positive peak in the detector signal (TCD). When the desorption is finished, the detector signal returns to the baseline. By integration of this positive peak signal the adsorbed amount is precisely determined with high reproducibility. Based on the BET theory the specific surface area can be calculated using the adsorbed volume (at monolayer), and the cross-sectional area of the adsorptive gas.

For the calculation of the single-point BET surface area, only one measurement point (e.g. at $p/p_0$ = 0.30) is measured. It is then transformed into the linearized BET formula to obtain the slope ($V_m$ = 1/s) under the assumption the BET curve is passing the origin and intercept becomes zero. The BET surface area is calculated by inserting $V_m$ into the following equation:

$$S_{BET} = \frac{V_m \times N_A \times A_{cs}}{22414\ \text{ml mol}^{-1} \times W_s}$$

$N_A$ = $6.022 \times 10^{23}$ mol$^{-1}$
$A_{CS}$ ($N_2$) = 0.162 nm$^2$
$W_S$ = sample mass (g)

## BET SURFACE AREA ANALYZER

# BELSORP MR1

The BELSORP MR1 is a highly efficient, stand-alone device that allows simultaneous pretreatment and measurement of samples. The specific surface area of materials is determined using the BET single-point method. Due to the highly sensitive measurements with a thermal conductivity detector (TCD), thermometers & pressure gauges, the measurement result is achieved in about 15 minutes. The automatic Dewar movement, the calibration function and the operation via the touch panel makes the BELSORP MR1 extremely user-friendly, especially for inexperienced users. The results are exported as a text file, Excel spreadsheet or printed report (rich text).

**Highly efficient measurement**

- Simultaneous pre-treatment and measurement
- BET single-point measurement in approx. 15 mins (including calibration)

**Highly accurate measurement**

- Measurement range (~0.01 m2/g)
- High accuracy, sensitivity and reproducibility
- User-friendly touch panel
- Auto-Zero function equipped with a highly sensitive thermal conductivity detector
- Dedicated calibration valve enables simple and stable calibration measurements
- Automatic measurement of temperature and pressure for accurate calibration
- Easy handling thanks to an automatic Dewar elevator and a cooling fan
- Measurement results and trend data can be saved on a USB flash drive
- Compact design without external PC

BELSORP SERIES

# APPLICATIONS

The BELSORP MINI X is used in various application fields, including catalysts, all-solid-state batteries and other batteries, fibers, polymer materials, chemicals, pigments, cosmetics, magnetic powders, separation membranes, filters, toners, cement, ceramics, and semiconductors.

The BELSORP MAX series is used in a variety of fields as well. These include catalysts, carbon, zeolite, MOF / PCP, batteries, all-solid-state batteries, fibers, polymer materials, chemicals, pigments, cosmetics, magnetic powders, separating membranes, filters, toners, cement, ceramics, and semi-conductors.

The BELSORP MR1 is used in applications such as catalysts, fuel cells, batteries, fibers, polymer materials, chemicals, pigments, cosmetics, magnetic powders, separating membranes, filters, toners, cement, ceramics, and semi-conductor materials.

## TYPICAL FIELDS OF APPLICATION

Batteries · Catalysts · Zeolite · Ceramics · Carbon · Electronics · Fuel Cells · Toner · Cement · Medicine · Silica · MOFs / PCPs · Pigments · Cosmetics

BELSORP SERIES

# COMPARISON OF MEASUREMENT METHODS

| | BELSORP MINI X | BELSORP MAX G | BELSORP MAX X | BELSORP MR1 |
|---|---|---|---|---|
| Pore size distribution | ⊕ | ⊕ | ⊕ | - |
| Micropore | + | ⊕ | ⊕ | - |
| Mesopore | ⊕ | ⊕ | ⊕ | - |
| Macropore | ⊕ | ⊕ | ⊕ | - |
| Isotherm | ⊕ | ⊕ | ⊕ | - |
| Single point BET | ⊕ | ⊕ | ⊕ | ⊕ |
| Multi point BET | ⊕ | ⊕ | ⊕ | - |
| Vapor adsorption | - | - | ⊕ | - |
| Chemisorption | - | - | ⊕ | - |
| True density | ⊕ | ⊕ | ⊕ | - |

⊕ *suitable* + *suitable to a limited extent* - *not suitable*


BELSORP SERIES

# TECHNICAL SPECIFICATIONS

BELSORP MR 1

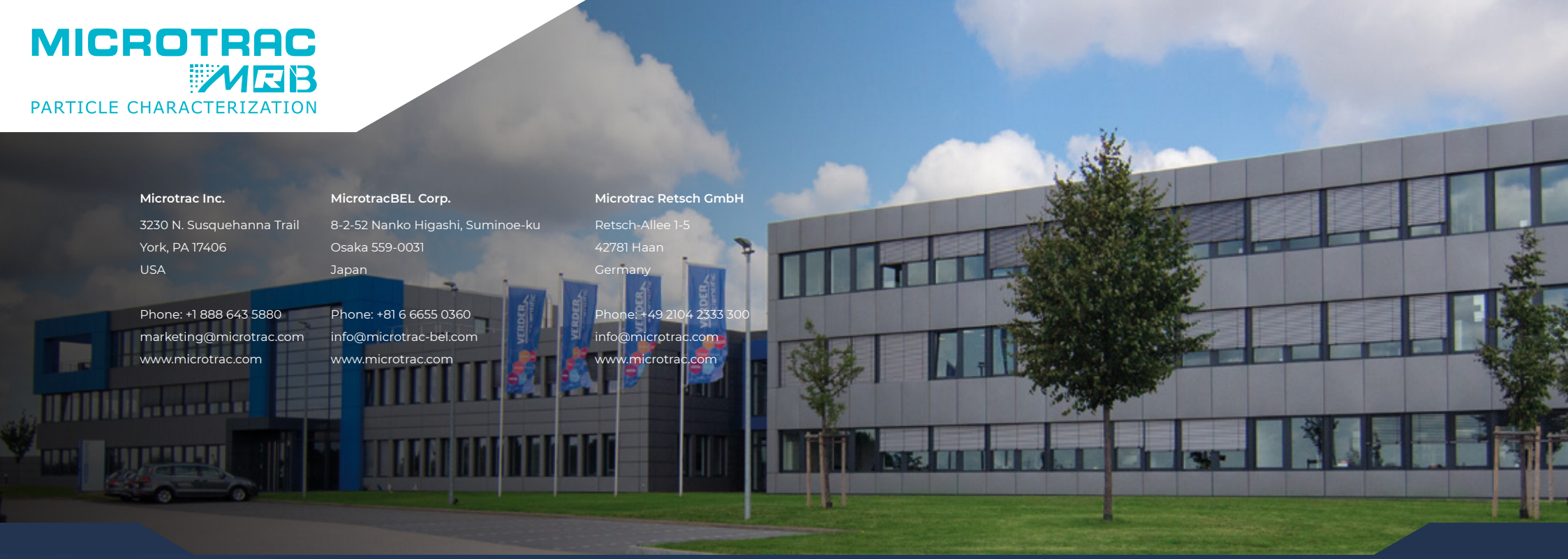
| System | BELSORP MR1 |
|---|---|
| Measurement principle | Dynamic flow gas method<br>(Single point BET method) |
| Detector | TCD |
| Adsorption gas | $N_2$/ Kr |
| Carrier gas | He |
| Number of measured samples | **1** |
| Pretreatment temperature | Up to 400°C |
| Measuring range | 0.01 $m^2/g$ and above |
| Reproducibility | within ±1.0% |
| Measurement time | Approx. 15 minutes<br>(including calibration, excluding pretreatment time) |
| Dimensions (W x H x D), weight | 350 x 553 x 368 mm, 30 kg |
| CE certificate | |

| System | | BELSORP MINI X | BELSORP MAX G | BELSORP MAX X |
|---|---|---|---|---|
| Measurement principle | | Volumetric method + AFSM™ (Advanced Free Space Measurement) | | |
| Adsorption gas | | $N_2$, Ar, Kr (MAX G only), $CO_2$, $H_2$, $CH_4$, butane, and various other non-corrosive gases | | $N_2$, Ar, Kr, $CO_2$, $H_2$, $O_2$, $CH_4$, $NH_3$, NO, CO, butane, and various other (non-)corrosive gases |
| Adsorption vapor | | - | | $H_2O$, MeOH, EtOH, $C_6H_6$, $CCl_4$, hexane, and various other (non-)corrosive vapors |
| Number of measurements (high accuracy mode) | | Max. 4 ports simultaneously (3) | Max. 1 port | Max. 4 ports simultaneously (3) |
| | Specific surface area | 0.01 $m^2/g$~ ($N_2$), 0.0005$m^2/g$~ (Kr) (depending on sample density) | | |
| | Pore size distribution (ø) | 0.7~500 nm [1] | 0.35~500 nm | |
| | Low pressure isotherm | $P/P_0 = 10^{-4}$ ~ ($N_2$ @ 77K, Ar @ 87 K) | $P/P_0 = 10^{-8}$ ~ ($N_2$ @ 77K, Ar @ 87K) | $P/P_0 \approx 10^{-9}$ ~ ($N_2$ @ 77K, Ar @ 87K) |
| | Vapor adsorption | - | - | $P/P_0$ = ~ 0.95 @ 40°C |
| Pressure transducer | 133 kPa (1000 Torr) | 6 | 3 | 6 |
| | 1.33 kPa (10 Torr) | - | 1 | 4 (max.) |
| | 0.0133 kPa (0.1 Torr) | - | 1 [2] | 3 (max.) |
| Thermostatic air oven | | - | - | 50°C |
| Gas ports | | 2 ports (5 ports max.) | 2 ports (5 ports max.) | 3 ports * (optional: 6, 9 or 12 ports max.) |
| CE certificate | | ✓ | ✓ | ✓ |

[1] 0.35 ~ 500 nm possible by molecular probe method [2] 0.0133 kPa (0.1 Torr) for LP model or 0.133 kPa (1 Torr) for MP model available * Corrosion-resitant

MICROTRAC MRB

PARTICLE CHARACTERIZATION

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VERDER scientific

VERDER SCIENTIFIC

# SCIENCE FOR SOLIDS

Verder Scientific is a business field belonging to the Verder Group and sets standards in the development, manufacture and sale of laboratory and analytics devices. Used in quality control, research and development for test-piece preparation and the analysis of solids.

For several decades our companies have supplied production plants and research institutes, laboratories for quality testing and analytics, all kinds of technical specialists and scientists with modern, reliable devices to solve the many and varied challenges they face.

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