MICROTRAC
a VERDER company

BET SPECIFIC SURFACE AREA & PORE SIZE ANALYZER

BELSORP MINI X

For simple, fast and highly accurate characterization of porous materials

Microtrac's BELSORP MINI X's outstanding features result in world class repeatability, with significantly reduced measurement time. The MINI X is equipped with up to 4 sample measurement ports and new high-throughput functions including multi-device control.

The BELSORP MINI X is meticulously designed to measure specific surface area, pore size distribution and pore volume with extreme accuracy. Equipped with dedicated pressure sensors on each sample measuring port and a dedicated port for saturated vapor pressure, it enables completely independent simultaneous measurements. The new measurement software improves user productivity by streamlining measurement procedures with in software step-by-step instructions, displaying the measurement progress and sending the measurement results via e-mail. Depending on your sample throughput, a 3-port and 4-port models are offered. Additionally, the new analysis software (BELMASTER^{TM}) enables structural evaluation of a wider range of materials than ever before.

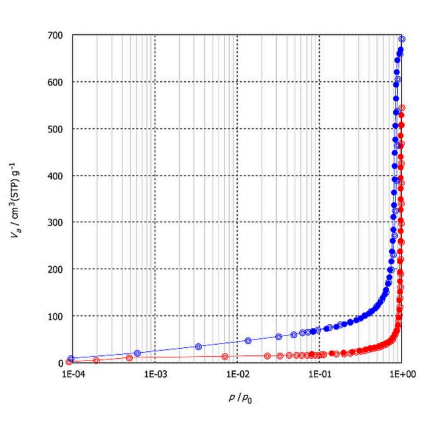
WORLD CLASS LEVELS OF REPRODUCIBILITY

- Up to four independent measurement ports and one dedicated port for saturated vapor pressure measurements
- Smallest & lightest in the world
- Unbeatable reproducibility by patented AFSM™
- Gas adsorption isotherm & NET adsorption measurement through AFSM™2 without the need of He-gas
- Multiple Measurement Methods: High-precision measurements, multi-sample measurement, and quick BET measurement modes to suit your purpose
- Dedicated pressure transducer for each port
- Simultaneous control of up to 20 measurement ports via multi-device control (5 units)
- Short measurement time with GDO
- IoT: Process monitoring via e-mail notification system
- Optional micropore analysis by molecular probe method
- Optional FDA 21 CFR Part 11 compliance

BET SPECIFIC SURFACE AREA & PORE SIZE ANALYZER BELSORP MINI X

KEY FEATURES

BET specific surface areas from 0.01 m^2/g ~ (N_2) and pore size distribution from 0.7 ~ 500 nm (option: 0.35 ~ 500 nm by molecular probe method).



Simultaneous Measurement of Multiple Samples

Up to 4 specimens can be measured simultaneously, enabling the measurement times for multiple samples to be shortened significantly.

Multiple Measurement Options

This instrument brings together highly accurate measurements with several high throughput modes (e.g. Quick BET mode, Gas Dosing Optimization). It can be used flexibly depending on customer needs, while integrated step-by-step instructions make it easy to use even for inexperienced users.

Ease of Use: Simple Setting of Measurement Software

The BELSORP MINI X is a fully automatic measurement system that enables the user to easily set the measurement conditions using Simple Mode. This mode allows for accurate measurements with minimal sample information (e.g. pre-treatment conditions and measuring range). This is particularly useful for unknown materials. Detailed measurement configurations can be set by experienced users in Professional mode. This system allows anyone to easily obtain accurate measurement results.

Gas Dosing Optimization (GDO)

Simple Mode includes the Gas Dosing Optimization (GDO) option, which automatically calculates the optimal gas dosing amount from previous measurement results. With this function, the measurement set points can be reliably obtained and the measurement times can be reduced significantly.

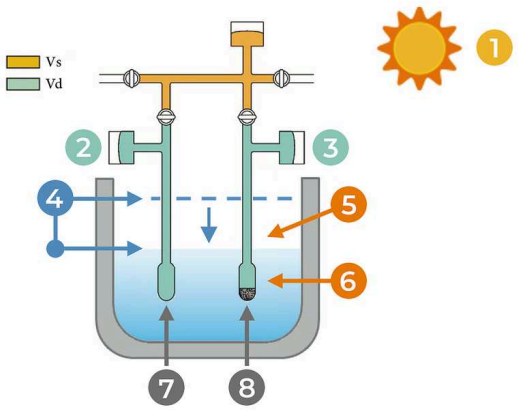
1. Load an existing isotherm | 2. Display of expected isotherm | 3. Input of measurement range | 4. Automatic optimization of conditions such as gas introduction volume |

Innovative Free Space Measurement for Highest Accuracy (AFSM™)

With AFSM™, the level control of liquid coolant (e. g. liquid nitrogen or argon) is not necessary anymore. Instead, we have adopted our groundbreaking, continuous free space measurement method, the patented Advanced Free Space Measurement (AFSM™). This method has achieved highest reproducibility by using a reference cell (empty sample cell; same type of sample cell used for measurement) to follow the second-to-second changes of free space. Our instrument determines an initial free space of the sample cell and reference cell. Since the change of the free space is equal in both cells (same environmental conditions), the free space change can be continuously followed by the pressure change of reference cell. Free space fluctuations caused by environmental factors can be considered:

- LN_2 level changes
- Atmospheric temperature / pressure changes
- Temperature changes of coolant due to oxygen dissolution

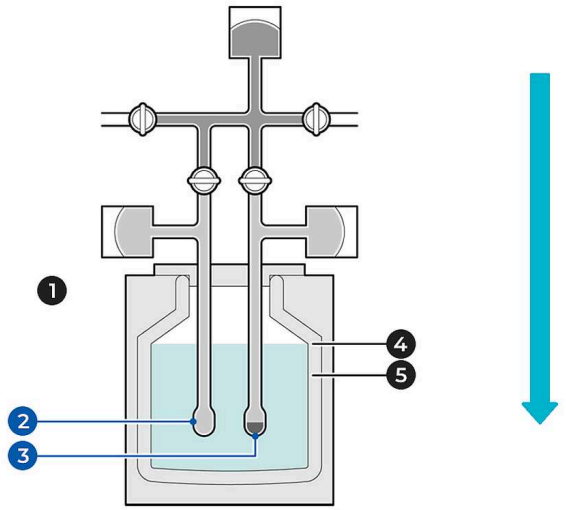
With this superior technique, environmental factors can be taken into account that were not considered before.
For more information, please use our knowledge base.



1. Room temeprature change | 2. P_{ref} | 3. P_{smp} | 4. Intial pressure | 5. The sample tube temperature changes due to the change of LN_2 level | 6. Liquid nitrogen temperature changes due to oxygen dissolution | 7. AFSM | 8. Sample

Adsorption Measurement Without Using Helium Gas (AFSM™2)

In a first step the free space measurement for a blank sample cell and a blank reference cell is performed with the adsorptive gas at the appropriate adsorption temperature. Then, the adsorption measurement can be achieved by utilizing both cells without using helium gas. In the same measurement condition, since the free space changes for both cells are same, the measurement results can be obtained in shorter measurement time. There is no need for time consuming operations such as keeping the liquid level of coolant constant between the blank measurement and adsorption measurement of the free space.



1. Reference cell for measuring fluctuations within free space continously | 2. Reference cell | 3. Sample cell | 4. Liquid level 1 | 5. Liquid level 2

AFSM (He-based method)

1. **Sample exhaust**
2. Free space (@room temperature) measurement with sample
3. Sample exhaust
4. **Reference tube, p_0 tube gas introduction**
5. Free space (@adsorption temperature) measurement with sample
6. Sample exhaust
7. **Adsorption measurement, free space change aquisition**
8. **Sample exhaust**
9. **Purge**

AFSM2 (He-free method): Step 1 --> 4 --> 7 --> 8 --> 9

Compact & Lightweight

Through complete optimization and rationalization of the materials, we succeeded in reducing the device size and weight.

Optional Pretreatment of Samples at Measurement Port

For an accurate adsorption measurement, a pretreatment of the samples is required. The pretreatment process (often called an activation process) is usually performed by applying vacuum under heat to remove adsorbed gas and/or water molecules from the surface of the material without affecting the sample structure (avoid denaturation). Microtrac offers two options for sample pretreatment. Firstly, it can be performed externally using BELPREP VAC II or VAC III, which is usually preferred to increase sample throughput. Alternatively, the activation process can be performed directly at the measurement port of the BELSORP MINI X using a heater (see accessories list). Using the heater accessory, a transfer from the external pretreatment device to the measuring port can be avoided, which is an important option for sensitive (e.g. hydrophilic) samples.

Data Analysis

The analysis software BELMASTER allows users to obtain a wide variety of analysis results through operations, such as displaying adsorption / desorption isotherms, evaluating specific surface areas using BET method etc., evaluating pore volumes (from ultra-micro- to meso- and macropores) by applying the t-plot or αS method. Further, conducting mesopore analysis using the DH and BJH methods, micropore analysis using the HK or SF methods, or optional GCMC / NLDFT analysis are possible. Even more analysis options are given in our BELMASTER analysis software.

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BELCONTROL: NEW OPERATION SOFTWARE

The versatility of BELSORP equipment is truly world leading. The numerous features and capabilities are complemented by BELCONTROL the intuitive and user-friendly operation software. It guides the user step-by-step through the analysis process. This includes the setup of analysis conditions, executing the measurements, when to fill and setup the liquid nitrogen or other bath, when to replace the gas cylinder, the degassing steps, and much more. The software is designed to make the instrument accessible and operable to everyone, including inexperienced users.

For inexperienced users or for measurements of unknown samples, BELCONTROL only requires basic sample information (name, mass, etc.), pre-treatment conditions (if not performed externally) and the measurement range.

Detailed control of the configuration and measurement settings is possible to optimize the measurement conditions (e.g. dosing settings, equilibrium criteria, leak test option, etc.). This allows the user to fully customize the sample analysis to his needs.

	BELCONTROL	
Quick BET	ja	Multi-point BET surface area in less than 20 minutes
Helium-Free Measurement	ja	AFSM^{TM} 2 enables He-free measurements with unmatched accuracy
Adsorption Kinetics	optioneel	Rate of adsorption measurements for diffusion analysis

MORE BELCONTROL FEATURES

- Overlaying adsorption / desorption isotherms and comparing the measured data between the various ports during measurement
- All pressures, temperatures, valve actuations, etc. are stored in trend data, allowing for immediate examination
- A system check function is available for diagnosing the instrument status
- E-mail notification automatically transmits measurement status and results
- Interactive program in Japanese or English ensures easy, reliable operation
- Extensive help functions, including step-by-step instructions during operation

BET SPECIFIC SURFACE AREA & PORE SIZE ANALYZER BELSORP MINI X

TOEBEHOREN EN OPTIES

Microtrac offers various accessories for its entire gas & vapor adsorption product line.

STANDARD CONSUMABLE GOODS

The standard consumables comprise sample cells, filler rods, filters, O-rings, caps and weighing platforms. Various sizes of sample cells, quick seals, and other optional consumables are also available.

WATER BATH

Easily swap the Dewar with a water bath for measurement temperatures ranging from -10°C to 70°C. A refrigerated / heated circulator is required.

HEATER

In-situ pretreatment of the sample from 50°C to 450°C. With this option, samples can be pretreated directly at the measurement port, without the need to transfer the sample cell before analysis. Particularly useful for sensitive samples.

GAS SELECTOR

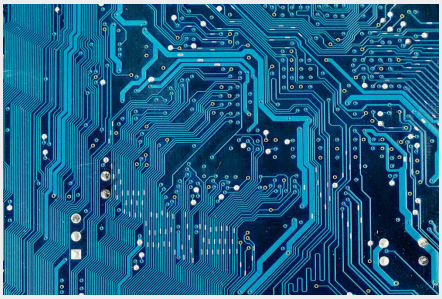
The gas selector enables the connection of up to 4 adsorptives simultaneously (1 x Helium + 4 x adsorptive).

BET SPECIFIC SURFACE AREA & PORE SIZE ANALYZER BELSORP MINI X

TYPISCHE TOEPASSINGEN

Microtrac's gas adsorption analyzers are used in a variety of fields. These include catalysts, batteries, fibers, polymer materials, zeolite, fuel cells, chemicals, pigments, cosmetics, MOF / PCP, magnetic powders, separation membranes, filters, toners, cement, ceramics, semi-conductors, and many more.

- batterij materialen
- Katalysatoren
- zeoliet
- keramiek
- koolstof



- elektronische componenten
- Brandstofcellen
- toner
- cement
- geneesmiddel / medicijnen

- Silicium
- MOF / PCP
- pigmenten
- cosmetica

... en meer!

The principles of volumetric adsorption measurement are explained in our knowledge base:

BET SPECIFIC SURFACE AREA & PORE SIZE ANALYZER BELSORP MINI X

TECHNISCHE GEGEVENS

Meetprincipe	Volumetric method + AFSM™
Adsorption gas	Nitrogen (N2), Argon (Ar), Methane (CH4), Ethane (C2H6), Carbon dioxide (CO2), Hydrogen (H2), n-Butane (n-C4H10), iso-Butane (iso-C4H10) and other non-corrosive gases
Gas ports	2 ports (5 ports max.)
Number of measurements (High Accuracy mode)	Max. 4 ports simultaneously (3 ports in High Accuracy mode)
Measurement range (specific surface)	0.01 m2/g and above (N2) (depending on sample density)
Pore size distribution (Diameter)	0.7 - 500 nm, Option: 0.35 nm by molecular probe method possible
Low pressure isotherm	p/p0 = 10^-4 (N2 @77K, Ar @87K)
Pressure transducer	133 kPa (1000 Torr) x 6 units
Vacuum gage / pump	Rotary pump
Sample tube	Standard tube, approx. 1.8 cm3 (optional: 5 cm3)
Dewar vessel	Volume: 2 l Holding time: 30 h
Pretreatment heater	50 - 450°C (4 ports)
Water bath	-10 - 70°C (4 ports)
Analysis software BELMaster™ 7	Adsorption isotherm, BET specific surface area type I (ISO9277)BET auto analysis, Langmuir specific surface area, BJH, DH, CI, INNES method
Analysis software BELMaster™ 7 cont.	t-plot, NLDFT / GCMC (OP BELSim™), MP method, Dubinin-Astakhov method, Molecular probe, as-plot
Afmetingen (B x H x D)	280 x 650 x 465 mm (excluding vacuum pump & PC)
Weight (main body)	38 kg (excluding vacuum pump & PC)
Utility - Gas	He, N2 (99.999% or higher purity), 0.1 ± 0.02 MPa, joint: 1/8" Swagelok Exhaust: Rotary pump exhaust port, ø 11 mm
Utility - Power	Single phase, AC 100~240 V (50 / 60 Hz) / 10A (incl. R.P.), 50 / 60 Hz
CE certified	Ja
Recommended Monitor	Full HD monitors

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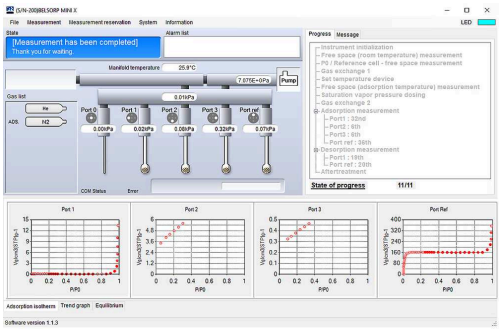
OPERATION SOFTWARE

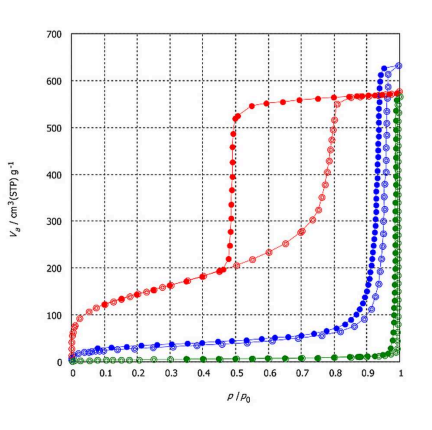
When developing the software, we focused on simplifying the operations and equipped it with many functions to increase labor productivity. Since the BELSORP instruments offer so many features, it is important to make those features easy to use. Our software will guide you step-by-step for the implementation of several procedures, i.e. execution of measurements, replacement of gas cylinders and purging of the manifold. The step-by-step instructions allows for users of all levels of experience to use the machinery safely and effectively. Depending on the level of user-experience two modes are offered, Simple Mode and Professional Mode. Simple Mode enables an easy operation by decreasing the customizability of the measurement – the only information you will need is the sample labeling, selecting pretreatment conditions (skippable if externally done) and measurement points. This low maintenance measurement is ideal for measuring unknown samples. If a prior measurement with comparable sorption behavior is available, the GDO function can be used to reduce the measurement time. Professional Mode offers the user a detailed configuration of the measurement, i.e. dosing settings, equilibrium criteria, leak-check option, and more.

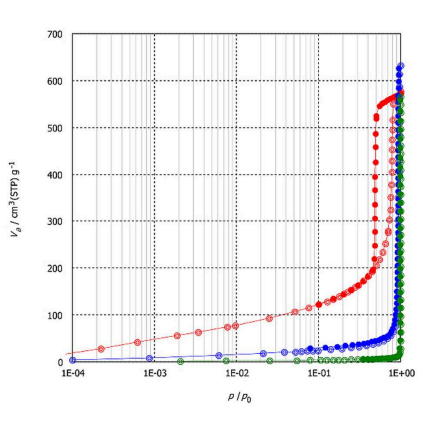


GAS ADSORPTION MEASUREMENTS OF POROUS & NON-POROUS MATERIALS: MOFS, ZEOLITES, CARBONS, AND MORE

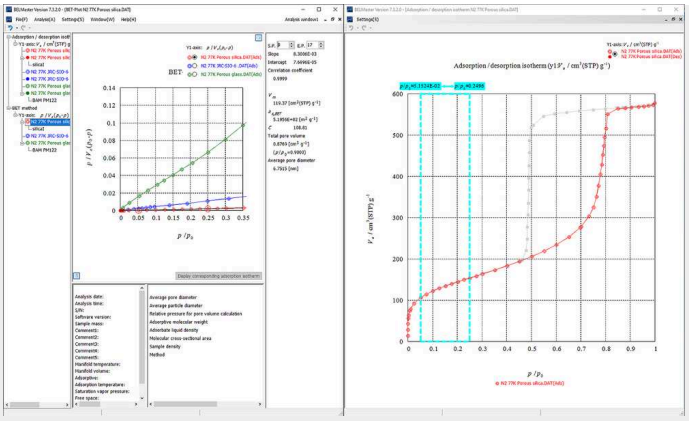
EVALUATION SOFTWARE BELMASTER

A sorption isotherm is defined as the relationship between the adsorbed amount onto an adsorbent and the equilibrium pressure of the adsorptive gas – commonly related to the saturation vapor pressure – at constant temperature. The gas sorption isotherm (e.g. nitrogen) delivers information about the specific surface area, pore size distribution and pore volume of measured material. In the following graph some exemplary sorption isotherms are shown.





The specific surface area (SSA) refers to the accessible surface area of the sample and is of great importance in adsorption, homogeneous and heterogeneous catalytic reactions. The specific surface area can be calculated according to ISO 9277 with the BET method (BET: Brunauer, Emmett and Teller) or Langmuir method. The following graph shows an exemplary evaluation of the specific surface area according to the BET method in our BELMASTER software:



Select the right pressure range (multi point BET) or a measurement point (single point BET) and the surface area will be calculated automatically. Further, our BELMASTER software also provides the BET surface area calculation according to ISO 9277 Annex C (also known as Rouquerol-plot), which is recommended for microporous materials.

The BELSORP MINI X's ability to measure low-surface area samples with high accuracy without the need of krypton gas (no additional pressure transducer or cryogenic temperature controller required), along with its "Quick BET Mode", which allows users to obtain BET specific surface areas (e.g. three-point-BET) of four samples in approx. 15 minutes, has cemented its place as a world class gas adsorption instrument. Pore size distributions from 0.7 up to 500 nm (optional 0.35 nm) are also achieved through the BELMASTER software from obtained isotherms.

HIGHEST ACCURACY AT LOWEST SURFACE AREAS

AFSM™

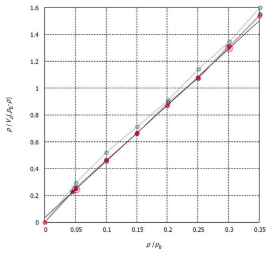
The following measurements are conducted using our patented, Advanced Free Space Measurement (AFSM™). This method allows for high reproducibility by using a reference cell to follow the changes in the free space volume. Our instrument measures the initial free space of the sample cell and reference cell. Since the change of the free space is equal in both cells (same environmental conditions), the change can be continuously followed by the pressure change of the reference cell.

In a product test, a certified reference material CRM-170 (nonporous alpha alumina; certified value of nitrogen BET specific surface area SSA: 1.05 ± 0.05 m^2/g) is used to check the accuracy of measurement in low total surface area (TSA) measurements. Although the TSA is reduced from approx. 2 m^2 to lower than 0.4 m^2, the determined BET specific surface areas SSA stay almost identical.

ADVANTAGES OF PATENTED AFSM TECHNIQUE

- LN_2 level maintenance is less important
- Improves reproducibility both in surface area and in pore volume
- Enables adsorption isotherm of low surface area material without using krypton gas (down to 0.1 m^2 in total surface area)

Sample	Adsorption	Total surface area in cell [m^2]	Sample amount [g]	SSA BET [m^2/g]*1	C constant
BCR-170_1	N_2	~2 m^2	~1.94	1.03	114
BCR-170_2	N_2	~1 m^2	~1.12	1.02	143
BCR-170_3	N_2	~0.7 m^2	~0.75	1.03	101
BCR-170_4	N_2	~0.4 m^2	~0.38	1.02	50



*Correlation coefficient 0.9995 or higher

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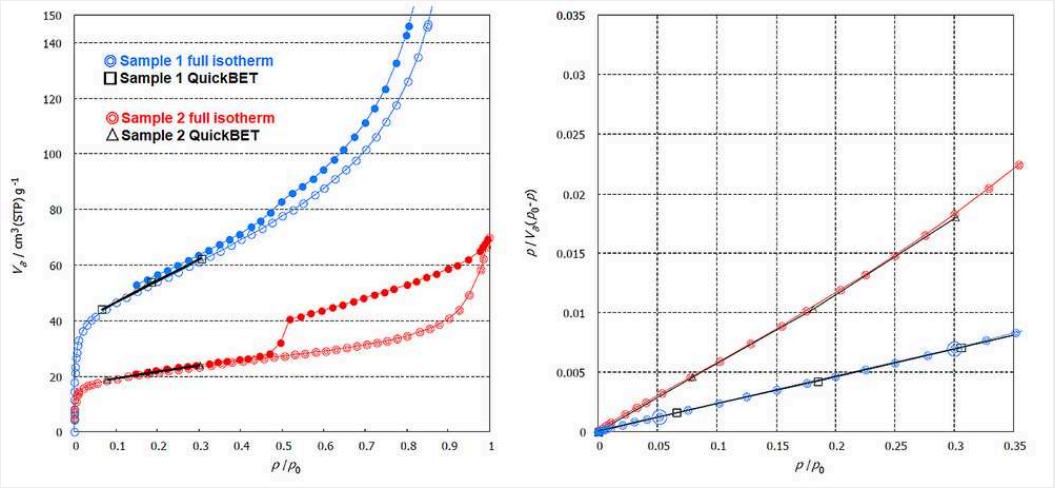
QUICK BET MODE

The Quick BET Mode can be used to maximize the sample throughput for BET specific surface area measurements. In this mode it is possible to measure three BET adsorption points of four samples in approx. 15 minutes (sample density required). Further, the Quick BET Mode offers two types of free space measurements: Firstly, the free space input option gives you the biggest opportunity to save time. Once the free space is determined for the sample cell, the free space measurement file (dvd file) can be reused for future quick BET measurements. Secondly, the free space can be determined with the actual measurement. A comparison of the obtained BET surface area from full-isotherm measurement (standard mode) and Quick BET Mode is given in the following graphs and table:



Sample	Adsorption	Multi-point BET SA [m^2/g]		Single-point BET SA [m^2/g]	
		Standard Mode	Quick BET	Standard Mode	Quick BET
1	N_2	189.9	190.5	188.1	190.3
2	N_2	75.7	77.4	74.7	75.7

*Multi-point BET in the p/p0 range of 0.05 - 0.30, single-point BET at p/p0 of 0.20

The measurement results obtained by multi-point BET in Standard Mode is assumed to be the most accurate result. The multi-point as well as single-point BET surface area measurements in Quick BET Mode are delivering fully comparable results compared to the standard mode, although the measurement time is significantly reduced to approx. 15 minutes for three samples (multi-point BET). This function is recommended for optimization of sample throughput e.g. in quality control.

BET SPECIFIC SURFACE AREA & PORE SIZE ANALYZER BELSORP MINI X

PRINCIPE

Analysis of Pore Size Distribution via NLDFT & GCMC Method

The classical pore size distributions (PSD) are calculated by 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, as well as the DR method, are also commonly used for pore volume evaluation and pore structure evaluation. The new PSD and capacity evaluation methods, NLDFT and GCMC (optional for BELSORP MINI X), are used for wide range analyses (from micro- to meso and macropores) and more accurate methods, specified in ISO15901-2.

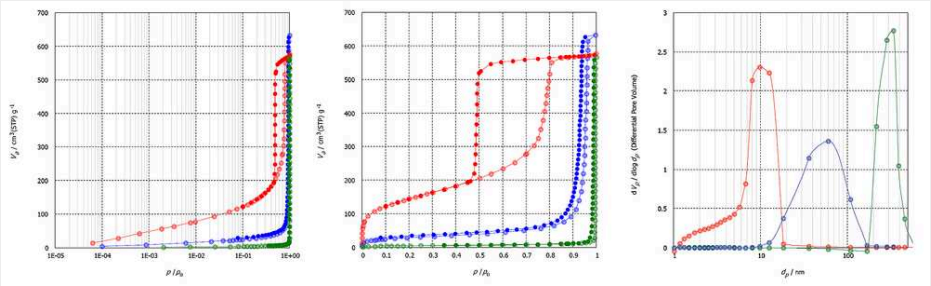
Theory	Interaction Surface and Gas	Adsorbate	Applicable Pore Size Range
BJH, CI, DH, INNES method	Kelvin equation (Surface tension and contact angle)	Bulk liquid density	> 2 nm Meso and macropore
HK, SF, CY method	Lennard-Jones potential (Interaction and repulsion force)	Bulk liquid density	0.4 - 2 nm Micropore
NLDFT, GCMC	Statistical thermodynamic model		0.35 - 500 nm Whole pore range

*[1] BELSORP MINI X is equipped with 1000 torr pressure transducers; isotherm starts from ~p/p_0 of $1x10^{-4}$
*[2] Option: 0.35 nm possible by molecular probe method

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 analyze 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 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 appropriate description of porous materials.

The BELMASTER software allows easy comparison between experimental and simulated isotherms, with the simulated isotherm serving as a basis for the PSD calculation.

In the following part an example for pore size distribution calculation by BJH method is given:



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