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.

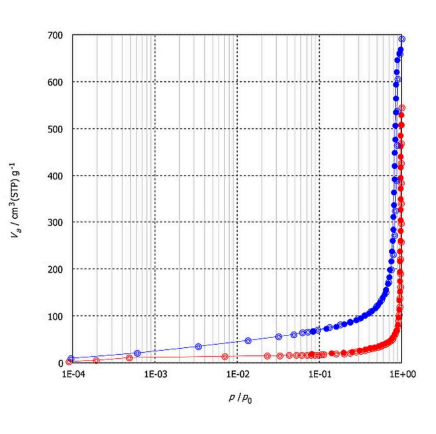
WORLD CLASS LEVELS OF REPRODUCIBILITY

- 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

КЛЮЧЕВАЯ ОСОБЕННОСТЬ

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.

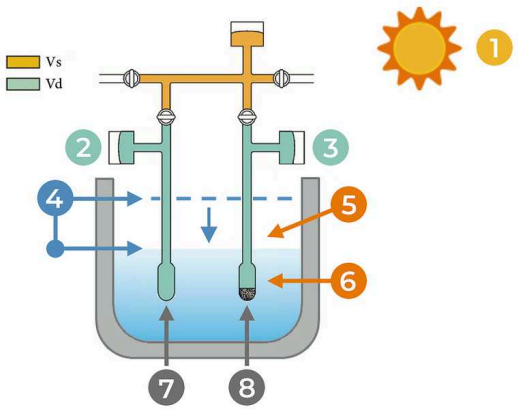
1. Load an existing isotherm | 2. Display of expected isotherm | 3. Input of measurement range | 4. Automatic optimization of conditions such as gas introduction volume |

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

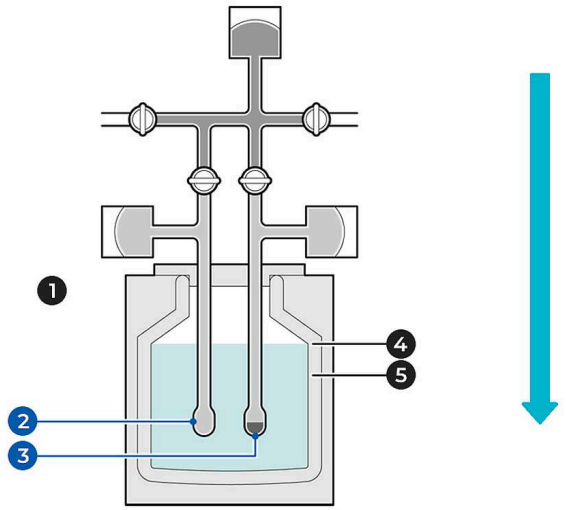
With this superior technique, environmental factors can be taken into account that were not considered before.
For more information, please use our knowledge base.



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^{TM}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	Да	Multi-point BET surface area in less than 20 minutes
Helium-Free Measurement	Да	AFSM™ 2 enables He-free measurements with unmatched accuracy
Adsorption Kinetics	опционально	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

АКСЕССУАРЫ И ОПЦИИ

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

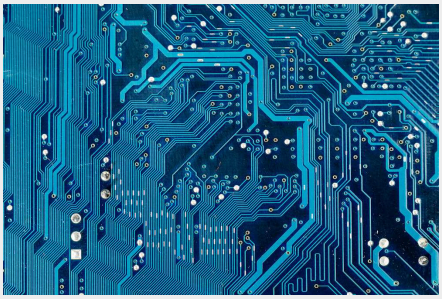
The gas selector enables the connection of up to 4 adsorptives simultaneously (1 x Helium + 4 x adsorptive).

BET SPECIFIC SURFACE AREA & PORE SIZE ANALYZER BELSORP MINI X

ТИПИЧНЫЕ ПРИМЕРЫ ПРИМЕНЕНИЯ

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.

- материалы аккумуляторов
- катализаторы
- цеолит
- керамика
- Углерод



- электронные компоненты
- Топливные элементы
- Тонер
- цемент
- медицина / фармацевтика

- Кварц
- MOF / PCP
- пигменты
- косметика

... и еще много чего!

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

BET SPECIFIC SURFACE AREA & PORE SIZE ANALYZER BELSORP MINI X

ХАРАКТЕРИСТИКИ

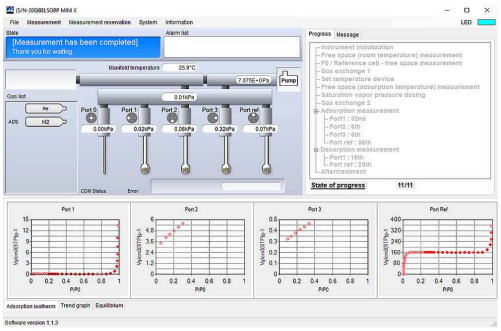
Принцип измерения	Объемный метод + AFSM ™
Адсорбция газа	Азот (N_2), Аргон (Ar), Метан (CH_4), Этан (C_2H_6), диоксид углерода (CO_2), водород (H_2), н-бутан (n-C_4H_{10}), изобутан (i-C_4H_{10}) и другие некоррозионные газы
Порты газа	2 порта (макс. 5 портов)
Количество измерений (Режим высокой точности)	Макс. 4 порта одновременно (3 порта в режиме высокой точности)
Диапазон измерений (удельная поверхность)	0,01 м2/г и выше (N2) (в зависимости от плотности образца)
Распределение пор по размерам (Диаметр)	0,7 - 500 нм, опция: возможно 0,35 нм методом молекулярного зонда
Изотерма низкого давления	p/p0 = 10^-4 (N2 @77K, Ar @87K)
Датчик давления	133 кПа (1000 Topp) x 6 единиц
Вакуумметр / насос	Роторный насос
Пробирка для образца	Стандартная трубка, приблизительно 1,8 см3 (опционально: 5 см3)
Сосуд Дьюара	Объем: 2 л Время выдержки: 30 ч
Подогреватель предварительной обработки	50 - 450°C (4 порта)
Водяная баня	10 - 70°C (4 порта)
Программное обеспечение для анализа BEL Master° 7	Изотермы адсорбции, BET удельной поверхности области тип I (в соответствии с ISO 9277) BET автоматический анализ, удельная поверхность Ленгмюра, BJH, DH, CI, INNES метод
Аналитическое программное обеспечение BELMaster ™ 7 cont.	t-участок, NLDFT / GCMC (OP BELSim™), MP метод, метод Дубинин-Астахова, молекулярный зонд, as-участок
Размеры (Ш x В x Г)	280 x 650 x 465 мм (без учета вакуумного насоса и ПК)
Вес (основной корпус)	38 кг (исключая вакуумный насос и ПК)
Используемый газ	He, N2 (99,999% или более высокая чистота), 0,1 ± 0,02 МПа, соединение: 1/8 " Быстросъемное/ / выпуск: выпускное отверстие роторного насоса, ø 11 мм
Используемая мощность	Однофазный, переменный ток 100~240 В (50/60 Гц) / 10A (ВКЛ. R. P.), 50/60 Гц

Отвечает нормам европейского сертификата безопасности	да
Рекомендуется Монитор	Мониторы с разрешением Full HD

BET SPECIFIC SURFACE AREA & PORE SIZE ANALYZER BELSORP MINI X

OPERATION SOFTWARE

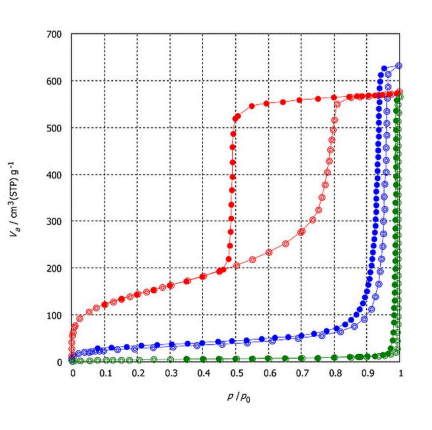
Программное обеспечение имеет наивысший приоритет для упрощения работы и оснащено множеством функций для повышения производительности труда. Поскольку приборы BELSORP предлагают множество функций и возможностей, становится все более и более важным упростить их использование. Наше программное обеспечение поможет Вам шаг за шагом выполнить несколько процедур, например, выполнить измерения, заменить газовый баллон, продуть коллектор и т.д. Эта удобная функция делает прибор доступным даже для неопытных пользователей. В зависимости от уровня пользовательского опыта предлагаются два режима, а именно Простой режим и Профессиональный режим. Простой режим обеспечивает простое управление путем ввода информации об образце, выбора условий предварительной обработки (можно пропустить, если выполняется извне) и диапазона измерений. Поэтому он идеально подходит для менее опытных пользователей и/или измерения неизвестных образцов. Если доступно предварительное измерение с сопоставимыми характеристиками сорбции, функция GDO (оптимальное дозирование газа) может быть использована для сокращения времени измерения. Кроме того, профессиональный режим предлагает подробные возможности настройки, например настройки дозирования, критерии равновесия, опция проверки на герметичность и т.д.

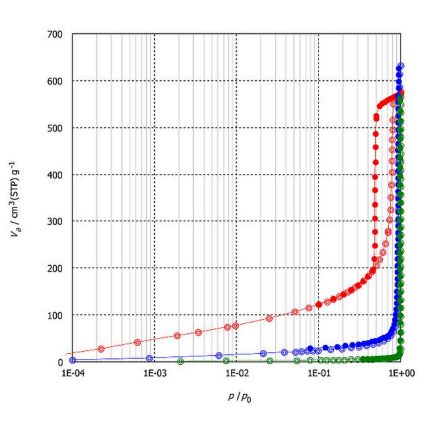


GAS ADSORPTION MEASUREMENTS OF POROUS & NON-POROUS MATERIALS: MOFS, ZEOLITES, CARBONS, AND MORE

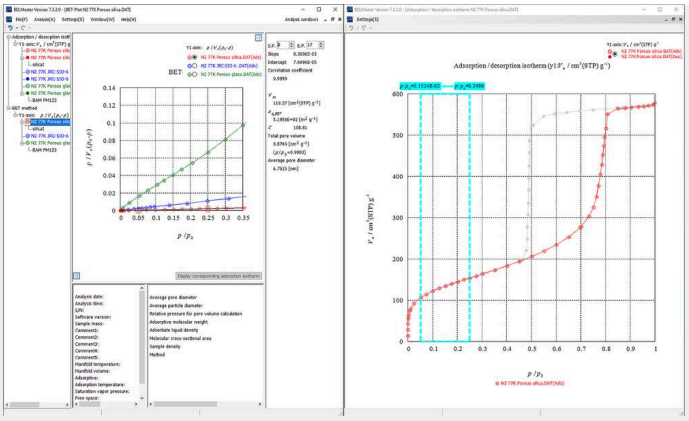
EVALUATION SOFTWARE BELMASTER

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.

ВЫСОЧАЙШАЯ ТОЧНОСТЬ ПРИ НАИМЕНЬШИХ ПЛОЩАДЯХ ПОВЕРХНОСТИ

AFSM™

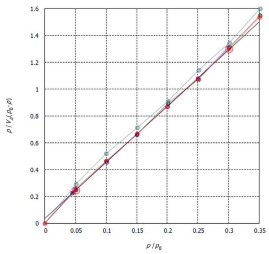
Следующие измерения проводятся с использованием нашего запатентованного непрерывного метода измерения свободного пространства, а именно Advanced Free Space Measurement (AFSM™). Этот метод достиг наивысшей воспроизводимости за счет использования эталонной ячейки для отслеживания своевременных изменений свободного пространства от секунды к секунде. Наш прибор определяет начальное свободное пространство ячейки образца и контрольной ячейки. Поскольку изменение свободного пространства одинаково в обеих ячейках (одинаковые условия окружающей среды), за изменением свободного пространства может непрерывно следовать изменение давления в контрольной ячейке.

Для проверки точности измерений при измерениях с низкой общей площадью поверхности (TSA) используется сертифицированный эталонный материал CRM-170 (непористый альфа-оксид алюминия; сертифицированное значение удельной площади поверхности азота BET SSA: 1,05 ± 0,05 м^2/г). Несмотря на то, что TSA снижается примерно с 2 м^2 до менее 0,4 м^2, определенные удельные площади поверхности BET SSA остаются практически идентичными.

ПРЕИМУЩЕСТВА ЗАПАТЕНТОВАННОЙ ТЕХНОЛОГИИ AFSM

- Не нужно поддерживать уровень LN_2
- Улучшает воспроизводимость как по площади поверхности, так и по объему пор
- Обеспечивает изотерму адсорбции материала с низкой площадью поверхности без использования газообразного криптона (общая площадь поверхности до 0,1 м^2)

Образец	добавки	Общая площадь поверхности в ячейке [м^2]	Количество образца [г]	SSA-BET [м^2/г]*1	C-постоянная
BCR-170_1	N_2	~2 m²	~1.94	1.03	114
BCR-170_2	N_2	~1 m²	~1.12	1.02	143
BCR-170_3	N_2	~0.7 m²	~0.75	1.03	101
BCR-170_4	N_2	~0.4 m²	~0.38	1.02	50



*Коэффициент корреляции 0,9995 или выше

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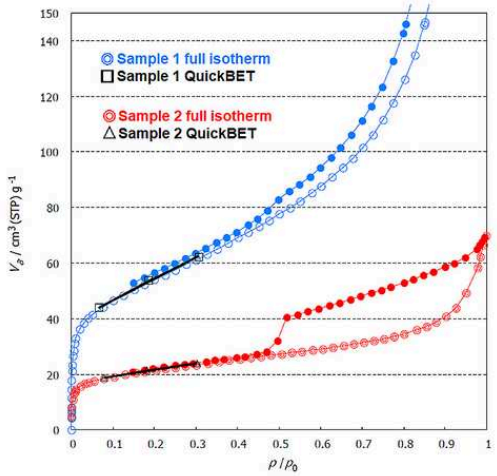
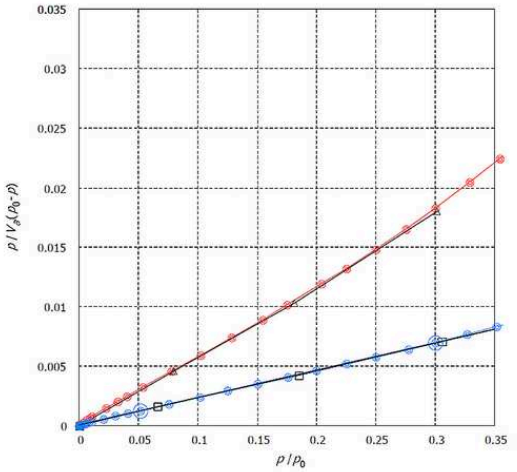
РЕЖИМ БЫСТРОГО BET

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:





Образец	добавки	Многоточечная BET-SA [м²/г]		Одноточечная BET-SA [м²/г]	
		Стандартный режим	Режим быстрого BET	Стандартный режим	Режим быстрого BET
1	N_2	189.9	190.5	188.1	190.3
2	N_2	75.7	77.4	74.7	75.7

*многоточечная BET в диапазоне p/p0 0,05 - 0,30, одноточечная BET при p/p0 0,20

Предполагается, что результаты измерений, полученные с помощью многоточечной BET в стандартном режиме, являются наиболее точным результатом. Многоточечные, а также одноточечные измерения площади поверхности BET в режиме быстрого BET дают полностью сопоставимые результаты по сравнению со стандартным режимом, хотя время измерения значительно сокращается до прибл. 15 минут на три пробы (многоточечная BET). Эта функция рекомендуется для оптимизации производительности отбора проб, например, при контроле качества.

BET SPECIFIC SURFACE AREA & PORE SIZE ANALYZER BELSORP MINI X

ПРИНЦИП РАБОТЫ

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.

Теория	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

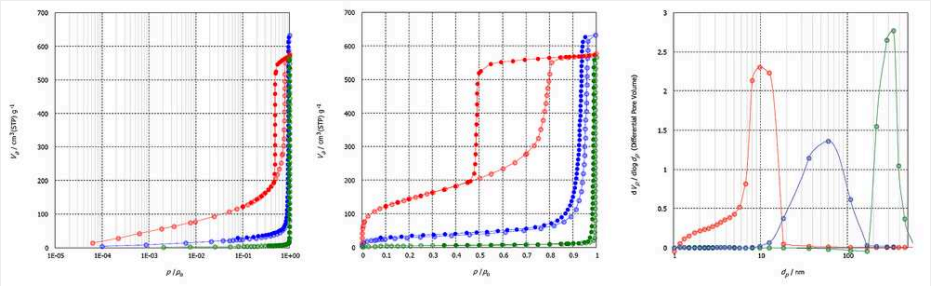
*[1] BELSORP MINI X оснащен датчиками давления 1000 торр; Изотерма начинается с ~p/p_0 of $1x10^{-4}$

*[2] Опция: 0.35 нм возможно с помощью метода молекулярного зонда

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:



www.microtrac.com/belsorp-mini-x