MICROTRAC
a VERDER company


BELCAT II

ADVANCED CATALYST CHARACTERIZATION

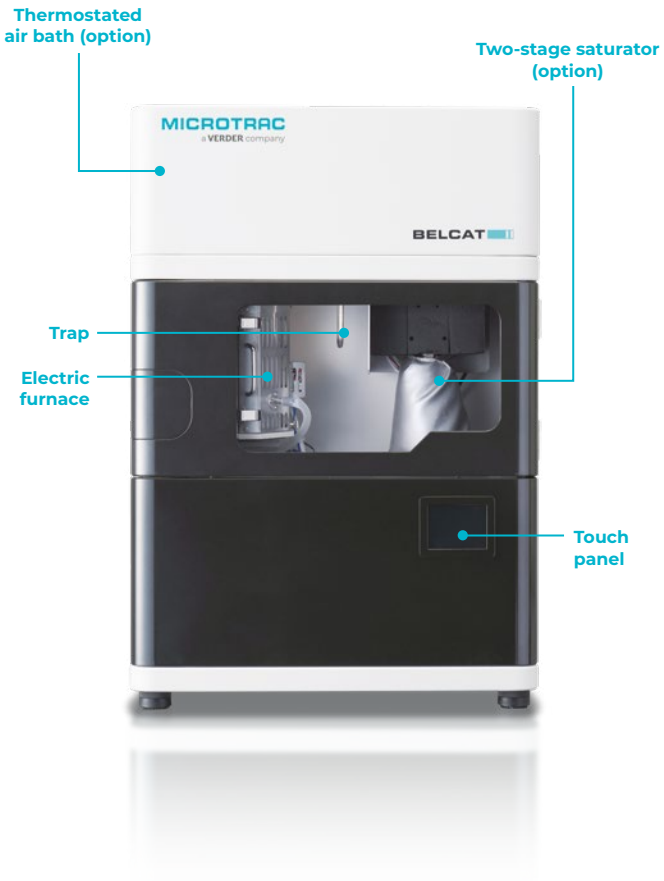
# TEMPERATURE-PROGRAMMED TECHNIQUES, PULSE CHEMISORPTION, SURFACE AREA, AND CATALYST EVALUATION

BELCAT II

# ALL-IN-ONE, FULLY-AUTOMATIC AND MULTI-PURPOSE ANALYZER

The reactivity of solid catalysts is generally influenced by the specific surface properties. Optimizing catalyst performance requires careful consideration of the nature and distribution of active sites. In addition to acidity and basicity, intrinsic properties such as redox behavior, electronic structure, and surface morphology are all critical factors that impact catalytic activity and selectivity. Metal dispersion rates, metal surface areas, and average particle size of supported precious metal catalysts are analyzed to enhance performance and reduce costs. Furthermore, specific surface areas (BET), adsorption kinetics, and saturated adsorption amounts are crucial parameters for the development of new adsorbent materials.

The BELCAT II serves as a catalyst analyzer capable of performing all these measurements in a single device. It is applicable to various other measurements and supports a wide range of customization, functioning as a comprehensive catalyst analysis tool.




## KEY FEATURES

- Dedicated instrument for chemisorption analysis with minimized dead volume
- State-of-the-art triple sample cell design
- Multi-purpose gas dosing design - connect once, access all required gases
- Versatile measurement condition settings
- Compact instrument design with small footprint
- Outstanding safety measures
- Modular design with upgrade capability

## I Compact Design

The BELCAT II features a compact housing with an attractive design, offering a small footprint and various functionalities.

## I Optimized Gas Flow Path

The gas flow path volume has been minimized by an integrated manifold valve block. The gas flow volume is controlled by high-precision mass flow controllers to enable stable and precise measurements.

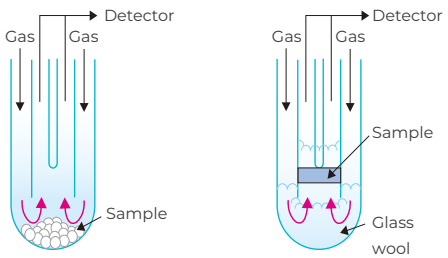
## I High-Resolution TCD Detector

A highly sensitive 4-element thermal conductivity detector (TCD) and an enhanced circuit board enable the measurement of a smoother spectrum.

## I Triple Sample Tube

The cylindrical triple sample tube facilitates efficient gas preheating at the outer peripheral section adjacent to the electric furnace before injection into the sample. For standard measurements, samples are easily exchanged by removing the outer tube. The cylindrical design of the tube enhances ease of handling and increases safety by being tension-free and less prone to breakage. This significantly improves operational efficiency.

**Quartz Triple Sample Tube**
Accurate, reliable and easy to handle.




**Pulse Chemisorption, TPD, BET measurement and more**

**Breakthrough curve**

## I Unique Gas Flow Design and Gas Mixing Function

While typical gas sorption systems are equipped with gas lines usable for single purpose only (gases are connected for pretreatment , analysis or pulse indepentely), the BELCAT II features ground breaking multi-purpose gas lines. This innovative and superior technology, enables each of the 8 multi-purpose gas lines to be used for pretreatment, analysis gas and pulse loop. There is no need to connect the same gas multiple times, making the instrument signifantly more compact.

Additionally, the integrated standard gas mixing section can be used to generate gas mixtures with any desired composition. It is useful for measurements such as temperature-programed reduction (TPR), or temperature programmed oxidation (TPO), without the use of conventional pre-mixed gas cylinders.

## I Vapor Dosing (Option)

The system includes a condenser that enables precise vapor dosing. The vapor adsorption amount is measured using pulse chemisorption techniques, facilitating the evaluation of catalytic reactions in humidified gas environments. It supports both continuous vapor flow and the measurement of vapor adsorption amounts with various pulsed steam flows. These capabilities are also applicable to catalytic reactions in humidified atmospheres.

## I Safety Measures

The electric furnace is fully enclosed, with the door securely locked during heating. Additionally, all heating parts feature overheat protection separate from the main control circuit. If the temperature exceeds the set value, heating and gas supply are simultaneously stopped, accompanied by an alarm. The system is equipped with an interlock connected to pressure sensors, a flow rate alarm, and an optional gas detector for enhanced safety.

# APPLICATIONS

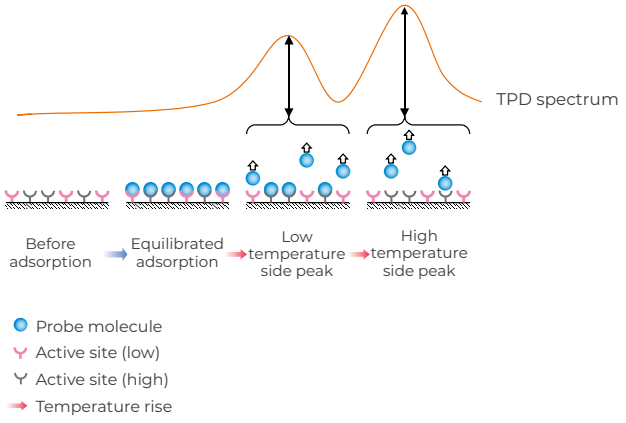
The BELCAT II can be used to analyze various types of materials such as catalysts, fuel cells, batteries, polymer materials, separation membranes, cement, ceramics, and much more.

CATALYSTS | CCUS/CCS | MOF/PCP | CARBON

FUEL CELLS | BATTERIES | PETROCHEMISTRY | GAS SEPARATION

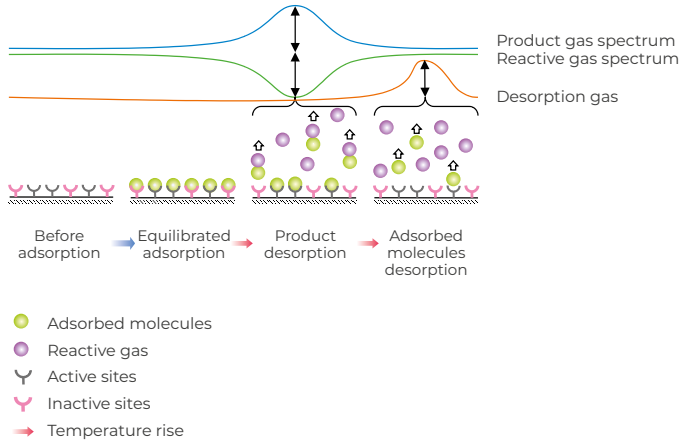
## I Temperature-Programmed Desorption Measurement (TPD)

This method is known for examining the chemical adsorption characteristics on solid surfaces and is generally represented by a spectrum with desorbed gas concentration as the Y-coordinate and temperature as the X-coordinate. By raising the sample temperature continuously and detecting the desorbed gas, desorption peak number, desorption temperature (activation energy for desorption) and desorption amount (number of adsorption points) can be obtained. Typically, $NH_3$-TPD is utilized to evaluate the acidic sites of solid acid catalysts, whereas $CO_2$-TPD is commonly employed to assess the basic sites of solid base catalysts.



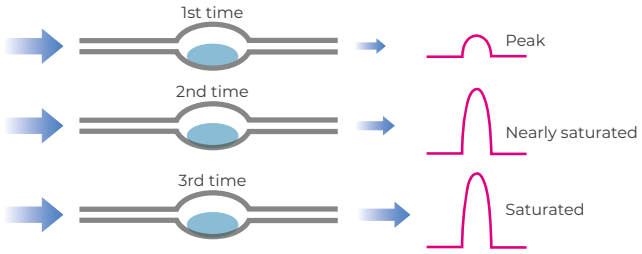

## I TPOxidation/TPReduction and TPReaction measurement

These techniques are used to analyze the surface reaction characteristics of solid catalysts, represented by plotting the consumption or production of reactants on the Y-axis and temperature on the X-axis. Temperature characteristics of catalytic reactions can be measured continuously and especially, each step can be individually observed. Possible methods are NOx reduction, CO oxidation reactions, and organic heating reactions by assessing the reduction temperature of samples under a hydrogen atmosphere.



## I Pulse measurement

The metal dispersion rate is a critical parameter in heterogeneous catalysis. It refers to the proportion of metal atoms that are exposed on the surface of a catalyst relative to the total number of metal atoms present (often expressed as a percentage). Metal dispersion can be calculated through pulse chemisorption using gases such as CO or $H_2$, which selectively chemisorb onto the metal surface. This is achieved by continuously pulsing a specified amount of gas into the sample until saturation is reached. Pulse measurement determines the chemisorbed quantity by taking the saturated peaks as a reference. The difference in peak areas between unsaturated and saturated state is giving the adsorbed amount. The metal dispersion rate is essential for assessing, optimizing, and understanding the performance and longevity of metal-based catalysts.



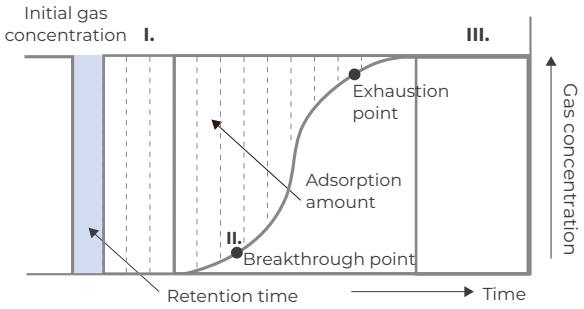

## I Adsorption breakthrough curve measurement

The breakthrough curve is a concept to describe the concentration of an adsorbate passing an adsorption column over time. It is an essential tool for understanding how an adsorbent material captures one or more components from a mixture of gases as it passes through the column.

I. Initial Phase: The adsorbent effectively captures the adsorptive and the concentration of the remaining adsorptive is low.

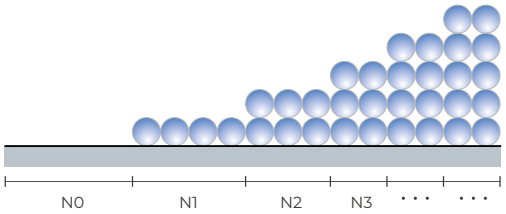
II. Breakthrough Point: The adsorptive concentration in the effluent starts to rise significantly, indicating that the adsorbent is becoming saturated and less effective at capturing the adsorbent.

III. Equilibrium Phase: The adsorptive concentration in the effluent gets equal to the influent concentration as the adsorbent becomes fully saturated.




## I BET specific surface area

The specific surface area, defined as the total surface area per unit dry mass of a solid, is an important parameter for the characterization of catalysts. It can be determined using the BET method measuring the amount of a gas (e.g. nitrogen) desorbed after the sample is cooled to liquid nitrogen temperature under a helium-diluted adsorptive gas flow and then returned to room temperature. This method is not only dispensable for solid catalysts, but also for various powdered samples, such as adsorbents.



# • SOFTWARE

The user-friendly software is equipped with numerous functions that increase the operator's productivity. Our advanced BELCAT II, developed over many years, allows easily to perform measurements achieving reliable and reproducible results through various sophisticated analysis tools. The analysis results are obtained as ASCII files and can be easily exported.

## I Simple operation measurement software

Dedicated tabs for predefined measurements allow an easy programming for standard analysis. The operational status, TCD charts, and temperature are displayed in real-time, enabling users to monitor the measurement process at a glance.

## I Capable of automatic zero-point adjustment

TCD's zero-point is automatically adjusted before measurement. The measurements are consistently performed at the same baseline, making data comparison straightforward. Continuous measurements with different carrier gases can be accurately performed.

## I Sequential measurement mode

The sequential measurement mode is an automated process in which measurements are taken one after another in a specific order. Each measurement occurs independently. This mode is used for a systematic characterization of catalysts without the need of interaction by the user, e.g. ammonia, TPD at different conditions, metal dispersion rate depending on temperature or cyclic stability analysis.

## I High reliability with automatic multi-point calibration

Multi-point calibration can be automatically performed after the TPD or TPR measurements by using the MFCs of the instrument to generate different gas concentrations.

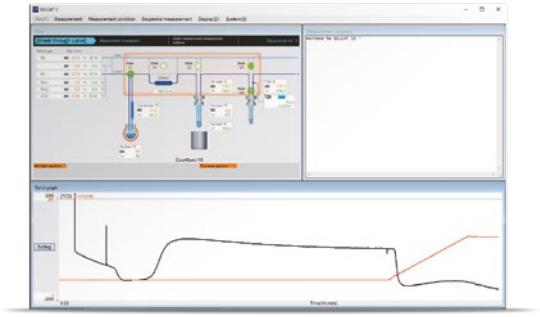
## I Waveform analysis software

The analysis data received by BELCAT II can easily be evaluated. The software calculates peak areas from spectra obtained in TPD and TPR measurements. A layer overlay function is provided for easy comparison of spectra. The waveform deconvolution function also enables peak separation. Further analysis tools are available for convenient data processing.

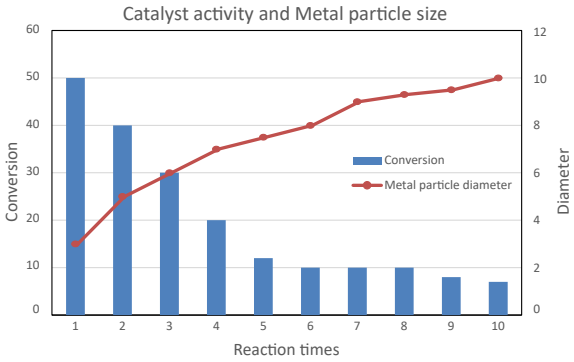
## I BELMASS link software (option)

BELCAT II measurement software can be linked with an online quadrupol mass spectrometer (QMS) gas analyzer, BELMASS II. The software performs an analysis in synchronization with the start and end of BELCAT II measurement while capturing the sample temperature. Additionally, the software supports external signal acquisition and timer control, so that it can be connected to other devices as well as used independently.

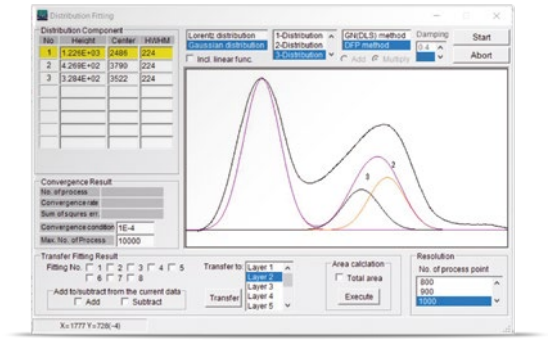
• **BELCAT II measurement view**



• **Result of sequential measurements**




• **Waveform deconvolution view**



• **BELMASS II measurement view**

## I Pulse chemisorption analysis function

A report of pulse measurement results is generated automatically for convenient data processing. Complex manual calculations such as adsorption amount or metal dispersion are not required. The recalculation function can be used to change the metal content or the stoichiometry factor after the measurement.

## I Data output examples (Pulse measurement)

### ❶ Label

Measurement date, file name, sample name, weight, etc.

### ❷ Supported metal species

Atomic weight, density, supported amount, stoichiometric factor of the metal, etc.

### ❸ Analysis result

Adsorption amount ($cm^3/g$) Metal dispersion rate (%)
Metal surface area (Sample, $m^2/g$) Metal surface area (Metal, $m^2/g$)
Average particle size (nm)

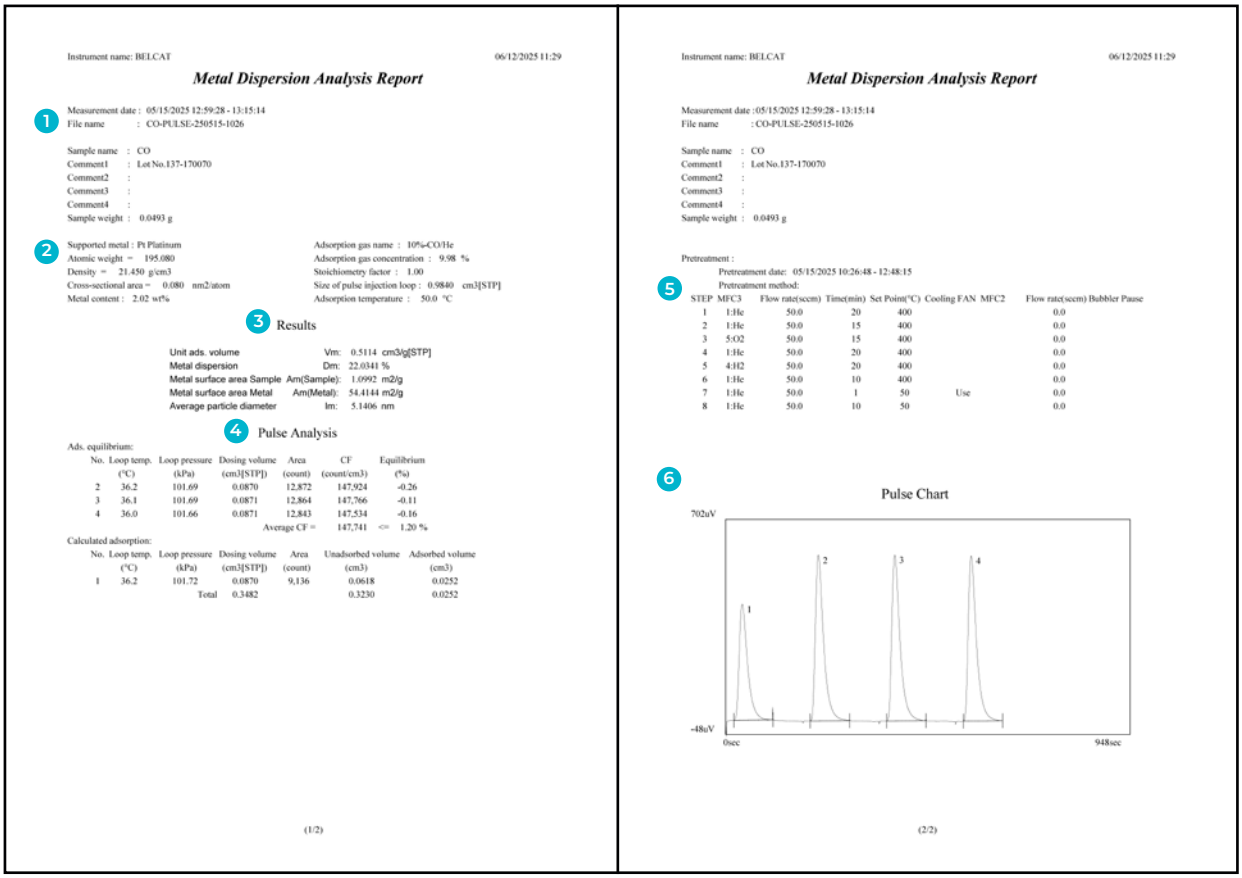
### ❹ Pulse details

Several peak parameters like dosed amount, peak area, equilibrium rate, etc.

### ❺ Pretreatment conditions

Pretreament gas species, temperature, time, etc.

### ❻ Pulse chart



Instrument name: BELCAT 06/12/2025 11:29

**Metal Dispersion Analysis Report**

❶
Measurement date : 05/15/2025 12:59:28 - 13:15:14
File name : CO-PULSE-250515-1026

Sample name : CO
Comment1 : Lot No.137-170070
Comment2 :
Comment3 :
Comment4 :
Sample weight : 0.0493 g

❷
Supported metal : Pt Platinum
Atomic weight = 195.080
Density = 21.450 g/cm3
Cross-sectional area = 0.080 nm2/atom
Metal content : 2.02 wt%
Adsorption gas name : 10%-CO/He
Adsorption gas concentration : 9.98 %
Stoichiometry factor : 1.00
Size of pulse injection loop : 0.9840 cm3[STP]
Adsorption temperature : 50.0 °C

❸ Results

| | | | |
|---|---|---|---|
| Unit ads. volume | | Vm: | 0.5114 cm3/g[STP] |
| Metal dispersion | | Dm: | 22.0341 % |
| Metal surface area Sample | Am(Sample): | | 1.0992 m2/g |
| Metal surface area Metal | Am(Metal): | | 54.4144 m2/g |
| Average particle diameter | | Im: | 5.1406 nm |

❹ Pulse Analysis

Ads. equilibrium:

| No. | Loop temp. (°C) | Loop pressure (kPa) | Dosing volume (cm3[STP]) | Area (count) | CF (count/cm3) | Equilibrium (%) |
|---|---|---|---|---|---|---|
| 2 | 36.2 | 101.69 | 0.0870 | 12,872 | 147,924 | -0.26 |
| 3 | 36.1 | 101.69 | 0.0871 | 12,864 | 147,766 | -0.11 |
| 4 | 36.0 | 101.66 | 0.0871 | 12,843 | 147,534 | -0.16 |
| | | | | Average CF = | 147,741 | <= 1.20 % |

Calculated adsorption:

| No. | Loop temp. (°C) | Loop pressure (kPa) | Dosing volume (cm3[STP]) | Area (count) | Unadsorbed volume (cm3) | Adsorbed volume (cm3) |
|---|---|---|---|---|---|---|
| 1 | 36.2 | 101.72 | 0.0870 | 9,136 | 0.0618 | 0.0252 |
| | | | Total | 0.3482 | 0.3230 | 0.0252 |

(1/2)

Instrument name: BELCAT 06/12/2025 11:29

**Metal Dispersion Analysis Report**

Measurement date : 05/15/2025 12:59:28 - 13:15:14
File name : CO-PULSE-250515-1026

Sample name : CO
Comment1 : Lot No.137-170070
Comment2 :
Comment3 :
Comment4 :
Sample weight : 0.0493 g

Pretreatment :
Pretreatment date: 05/15/2025 10:26:48 - 12:48:15
❺ Pretreatment method:

| STEP | MFC3 | Flow rate(sccm) | Time(min) | Set Point(°C) | Cooling FAN | MFC2 | Flow rate(sccm) | Bubbler Pause |
|---|---|---|---|---|---|---|---|---|
| 1 | 1:He | 50.0 | 20 | 400 | | | 0.0 | |
| 2 | 1:He | 50.0 | 15 | 400 | | | 0.0 | |
| 3 | 5:O2 | 50.0 | 15 | 400 | | | 0.0 | |
| 4 | 1:He | 50.0 | 20 | 400 | | | 0.0 | |
| 5 | 4:H2 | 50.0 | 20 | 400 | | | 0.0 | |
| 6 | 1:He | 50.0 | 10 | 400 | | | 0.0 | |
| 7 | 1:He | 50.0 | 1 | 50 | | Use | 0.0 | |
| 8 | 1:He | 50.0 | 10 | 50 | | | 0.0 | |

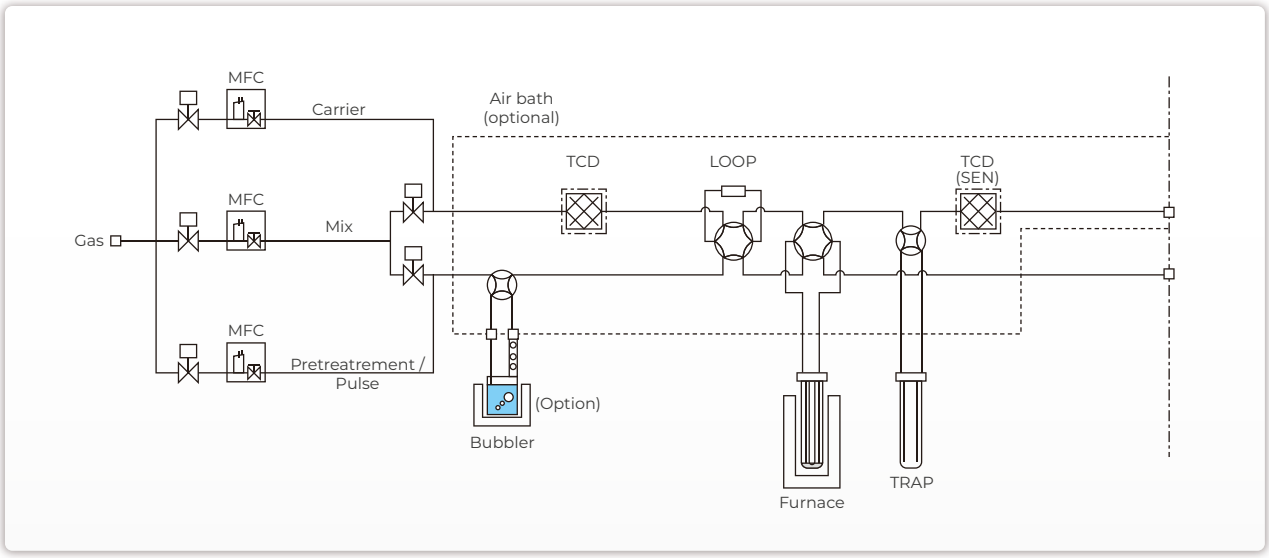
❻ Pulse Chart



(2/2)

# • FLOW PATH DIAGRAM

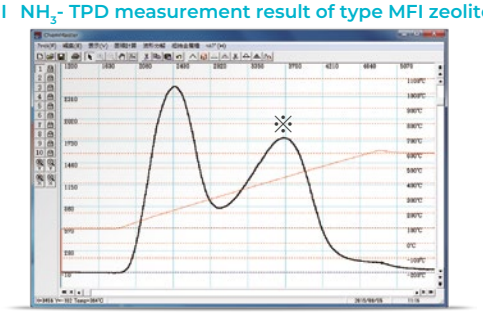
The gas injection system consists of a carrier line, a pretreatment/pulse gas line and a mix line. Gases in the mix line can be mixed with either the carrier line or the pre-treatment/pulse gas line.
Various applications are possible, such as mixing $H_2$ with Ar for TPR measurements or injection amount control by diluting the pulse gas and many more.
By implementing the pretreatment line, contamination of the detector due to degassing of the sample can be prevented. Stable data can be obtained over a long period of time.




* Simplified flow diagram

# • MEASUREMENT EXAMPLES

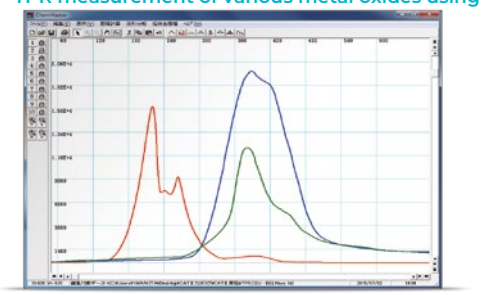
### I $NH_3$- TPD measurement result of type MFI zeolite



Desorption amount: 0.861 mmol/g
Peak temperature: 440 °C

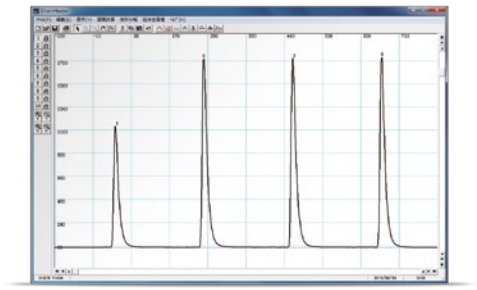
※ H peak value

### I TPR measurement of various metal oxides using $H_2$/Ar



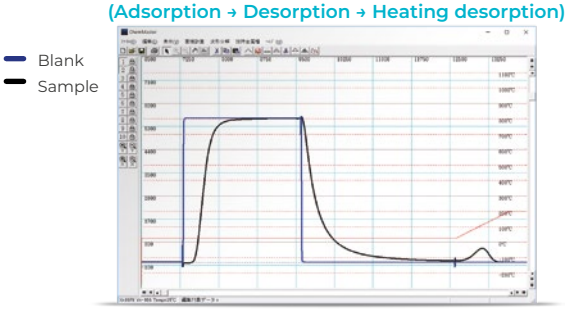
- $CuO+H_2 \rightarrow Cu+H_2O$
- $NiO+H_2 \rightarrow Ni+H_2O$
- $CoO+H_2 \rightarrow Co+H_2O$

### I $Pt/Al_2O_3$ metal dispersion rate by CO pulses



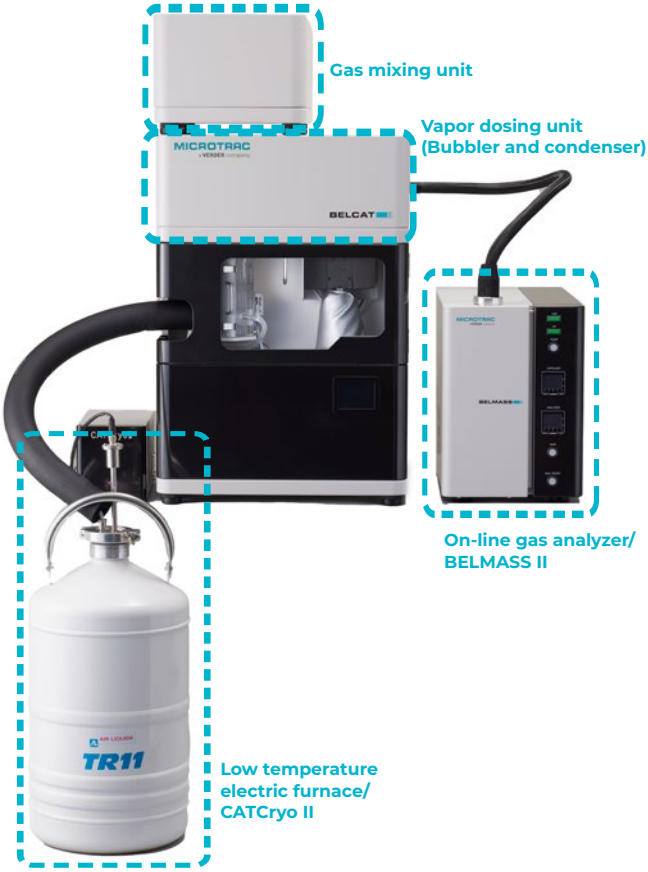
Desorption amount: 0.566 $cm^3/g$
Metal surface area: 1.22 $m^2/g$
Metal dispersion rate: 25.4%
Metal particle size: 4.7 nm

### I $CO_2$ adsorption breakthrough curve measurement (Adsorption → Desorption → Heating desorption)




Adsorption gas: 1% $CO_2$/He
Desorption amount: 30.6 $cm^3/g$
Adsorption amount: 33.8 $cm^3/g$
Heating desorption amount: 2.9 $cm^3/g$

# • OPTIONS




### I Vapor dosing unit

Consists of an air thermostat unit, bubbler, heater and condenser. The condenser eliminates excess vapor. Vapors can be injected accurately at stable concentrations.

### I External gas mixing unit

Allows mixing of three or more gases. One unit can install up to three lines. Corrosive gases are supported.

### I Low temperature electric furnace/CATCryo II

By applying the liquid nitrogen spray, the sample temperature can be continuously controlled from -120 °C. This can be utilized e.g. for the metal dispersion rate measurement for the ceria carrier and TPR measurement at room temperature. The cooling performance is improved with the optimal internal structure and the liquid nitrogen consumption is drastically reduced.

### I On-line gas analyzer/BELMASS II

Systemized quadrupole mass spectrometer, BELMASS II can be connected with BELCAT II.
Multiple components of gases unable to separate by TCD can be measured at high quantitative accuracy while linked with the BELCAT II software. It can also be utilized for the catalytic reaction analysis

### I AIRGUARD measurement system

Functional materials can react with moisture and oxygen in the air, resulting in changes of their structural properties or the generation of corrosive gases. To measure materials without exposing them to the air, MICROTRAC developed the new AIRGUARD measurement system.

### I Separation performance evaluation of mixed gas

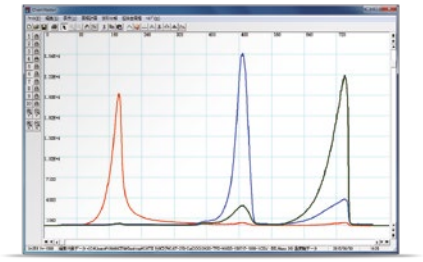
For DAC, CCUS, and CCS applications, the system enables breakthrough curve measurements with multiple gaseous components such as $CO_2$ and $H_2O$ that can be analyzed using dedicated highly sensitive detectors.

# • MEASUREMENT EXAMPLES

### I Thermal decomposition measurement of calciumoxalate monohydrate

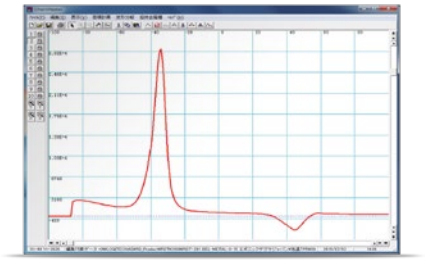
- *m/z*=18($H_2O$)
- *m/z*=28(CO)
- *m/z*=44($CO_2$)

Used option:
BELMASS II



### I Temperature-programmed reduction (TPR) of supported palladium catalyst from low temperature (-100 °C)

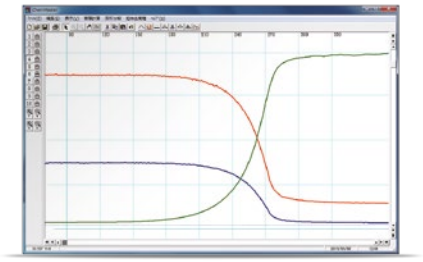
Used option:
CATCRYO II



### I Temperature-programmed oxidation reaction of carbon monoxide using precious metal catalyst

- m/z=28(CO)
- m/z=32($O_2$)
- m/z=44($CO_2$)

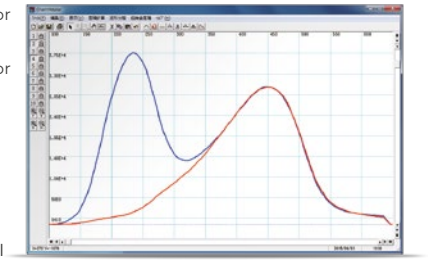
Used option:
MIX gas unit,
BELMASS II



### I Water vapor treatment for zeolite at $NH_3$-TPD(m/z=16)

- Without vapor treatment
- Without vapor treatment

Used option:
Vapor dosing unit, BELMASS II



# • ACCESSORIES

| Article | Description |
|---|---|
| 062-20000-0-0 | Sample cell holder |
| 062-20002-0-0 | Triple sample cell 3 (Outer), 3pcs |
| 062-20003-0-0 | Triple sample cell 2 (Inner), 3pcs |
| 062-20004-0-0 | Triple sample cell 1 (Thermocouple), 3pcs |
| 062-20005-0-0 | Sample cell (trap/outer for large volume inner cell), 1pcs |
| 062-20006-0-0 | Bubbling bottle |
| 062-20019-1-0 | Thermocouple (K-type) |
| 062-20013-1-0 | Filter (2μ), 3pcs |
| 900-00040-0-0 | O-ring (P-10A, FFKM), 3pcs |
| 900-00050-0-0 | O-ring (P-20, FFKM), 3pcs |
| 900-00052-0-0 | O-ring (P-4, FFKM), 3pcs |
| 900-30000-1-0 | Quartz glass wool (10g) |
| 990-00013-0-0 | Reference sample (2wt%Pt/Al2O3, 1g) |
| 990-00014-0-0 | Reference sample (MFI, 1g) |
| 062-20029-0-0 | AIRGUARD sample cell holder |

# • SPECIFICATIONS

## I Catalyst analyzer/BELCAT II

| | | |
|---|---|---|
| **Measurement principle** | | Dynamic flow method |
| **Detector** | | Semi-diffusion type 4-element thermal conductivity detector (TCD) |
| **Measurement/ pretreatment port** | | 1 |
| **Injection gas** | | He, Ar, $N_2$, $O_2$, $H_2$, CO, $CO_2$, $NH_3$, $N_2O$, NO, etc. |
| **Gas** | Carrier | 3 |
| | Pretreatment/Pulse/MIX | 8 (Corrosion-resistant × 2) |
| **Mass flow Controller** | Carrier | F.S. 100 sccm |
| | Pretreatment/Pulse/MIX | F.S. 100 sccm |
| | MIX | F.S. 30 sccm |
| **Electric furnace** | | Regular temperature: 1100 °C |
| | | Maximum temperature: 1200 °C |
| | | Quick cooling: 30min (400→50 °C) |
| | | CATCryo II (option) -120 °C supported |
| **Vapor injection (option)** | | $H_2O$, $CH_3OH$, $C_2H_5OH$, toluene, benzene, etc.. |
| **Utility** | Gas | Measurement gas: 0.1 MPa(G), Valve driving: 0.45-0.55 MPa(G) |
| | | Joint: 1/8" Swagelok connection |
| | Power | AC110V/220V, 1,300W |

## I On-line gas analyzer/BELMASS II

| | |
|---|---|
| **Mass range** | 1-200 amu |
| **Detector** | Faraday cup / SEM |
| **Min. detection limit** | < 1 ppm (depending on gas) |
| **Resolution** | M/ΔM ≥ 2M |
| **Scan speed** | Auto, 0.01, 0.03, 0.1, 0.3, 1, 3, 10 sec / amu |
| **Sniffer probe** | 1/16" capillary tube |
| **Max. temperature of heat hose** | 200 °C (SUS), 120 °C (PEEK) (optional) |
| **Gas consumption rate** | Approx. 0.6 cc/min (at 1 atm) |
| **Sample gas pressure** | Atmospheric pressure (50-150 kPa) |
| **Vent connection** | 1/4" one-touch connection |
| **Measurement channels** | Max. 16 CH |
| **Measurement software Quadvision2** | Selected ion monitor, Mass peak monitor |
| **Analysis software ChemMaster II** | Spectrum image display, Distribution fitting, Area calculation, Conversion to temperature axis, Metal dispersion calculation |
| **Other functions** | System check, Analog input, Conversion of the saved data into CSV |
| **Interface** | RS232C |
| **Analog input** | 1 CH (DC 0-10 V, mainly used as a temperature input) |
| **Dimensions, weight** | 280 (W) × 400 (H) × 600 (D) mm, 36 kg |

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