

Catalyst analyzer

# BELCAT II

Pulse, TPD, TPR, BET

Breakthrough curve, various catalytic reactions



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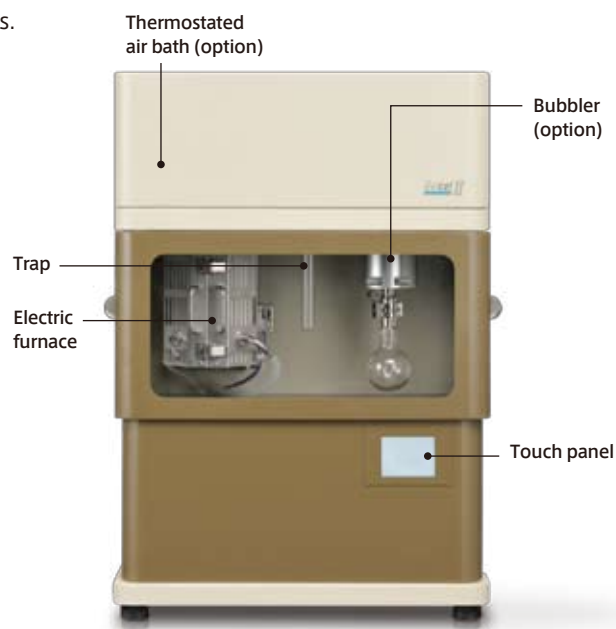
Specialists in Adsorption

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# All-in-one, fully-automatic and multi-purpose analyzer

## BELCAT II

The surface properties of solid catalyst is studied in various ways. The catalytic activity of solid acid catalyst is determined by its acidity/basicity. In the supported precious metal catalysts, the dispersion rate of the supported metal, metal surface area and particle size are strongly associated with cost and performance. Adsorption kinetics and the saturated adsorption amount are keys to develop new adsorbent material. The solid catalyst reaction generally happens on its surface and the specific surface area is an important factor. BELCAT II is a catalyst analyzer to perform these measurements in one. This can be applied to other various measurements and supports a wide range of customization. You can use it as a comprehensive catalyst analysis tool.



## Features

### 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 the high precision mass flow controller to enable stable measurements.

### High resolution TCD detector

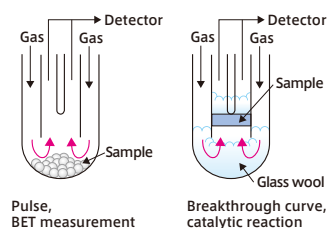
Highly sensitive 4-element thermal conductivity detector (TCD) and improved circuit board makes smoother and highly sensitive spectrum can be measured.

### Triple sample tube

The triple sample tube is adopted as proven in the thermal analysis. The gas preheated at the outer peripheral portion close to the electric furnace can be injected to the sample. For pulse chemisorption measurement, the sample can be exchanged by simply removing the outer tube. The handling is greatly improved.

#### Quartz triple sample tube

Accurate, reliable and easy handling.



### Compact design

Attractively designed compact cabinet having a small footprint with various features.

### Unique gas flow design and standardized gas mixing function

BELCAT II is equipped with 8 gas ports.

All the gases can be used for pretreatment/ pulse gas.

Without changing the gas connection, the gas ports can support various measurements.

Additionally, they can be used for the standard gas mixing function. It is useful for measurement such as temperature-programmed reduction (TPR), or temperature programmed oxidation (TPO). Without the use of conventional pre-mixed gas cylinders, measurements are available by preparing pure gas cylinders.

### Bubbler injector (option)

Equipped with a condenser, vapor dosing with the accurate concentration is available. The vapor adsorption amount can be measured by using pulse chemisorption measurement, and catalytic reaction in a humidified gas can be evaluated. Not only continuous vapor flow, but also the adsorption amount measurement of vapor with various pulsated steam is supported and it can also be used in the catalytic reaction in a humidified atmosphere.

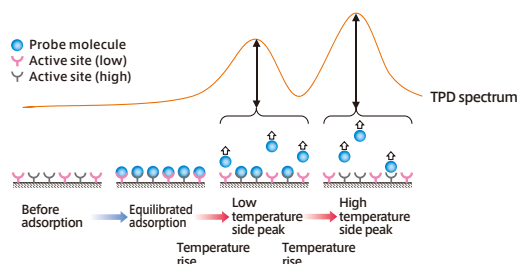
### Safety measures have been implemented to protect the researchers

Electric furnace is fully covered and the door is locked when the furnace is heated. In addition, all of the heating units are equipped with over heat protection function separately from the control circuit. Heating and gas are stopped together with an alarm when it exceeds the set value. The interlock is also equipped and connected to pressure, along with a flow rate alarm and gas detector (optional), for use with confidence.

# Applications

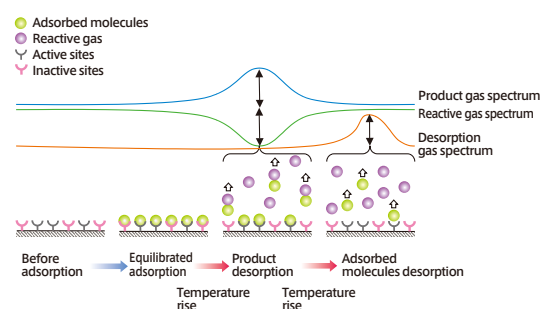
## Temperature-programmed desorption measurement (TPD)

Known as a method to examine the chemical adsorption characteristics on the solid surface and generally represented by the 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 (kind of adsorption points), desorption temperature (activation energy for desorption) and desorption amount (number of adsorption points) can be obtained.  $\text{NH}_3$ -TPD as acid nature evaluation for solid acid catalyst and  $\text{CO}_2$ -TPD as basic evaluation for solid base catalyst are widely utilized.



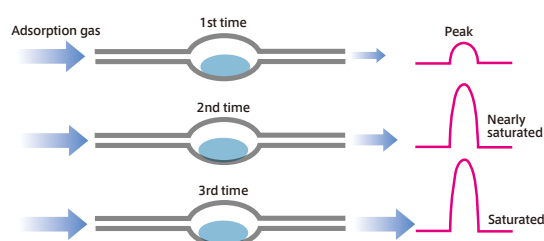
## TPO/TPReduction and TPReaction measurement

Known as a method to examine the reaction characteristics of solid surface and represented by the spectrum with consumption or production of reactant as Y-coordinate and temperature as X-coordinate. Temperature characteristics of catalytic reaction can be measured continuously and especially, each step of step-by-step reaction can be individually observed. Utilized for TPR measurement,  $\text{NO}_x$  reduction,  $\text{CO}$  oxidation reaction, organic heating reaction, etc. by measuring the reduction temperature of the sample under a hydrogen atmosphere.



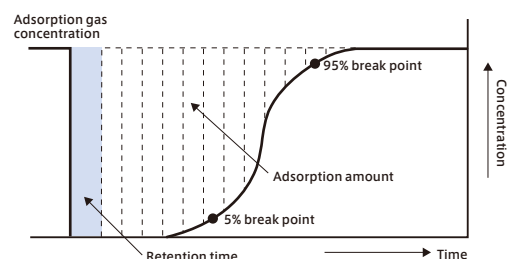
## Pulse measurement

Pulse measurement is to calculate the adsorption amount from the difference of the peak area between unsaturation and saturation by injecting a certain amount of gas to the sample continuously until saturation. Metallic dispersion can be calculated by the pulse measurement using such gas as  $\text{CO}$ ,  $\text{H}_2$ , etc. that selectively chemisorb on the metal surface. Other applications are reported on oxygen storage capacity (OSC) measurement using  $\text{O}_2$  pulse, metallic dispersion measurement for supported  $\text{Cu}$  catalyst using  $\text{N}_2\text{O}$ , etc.



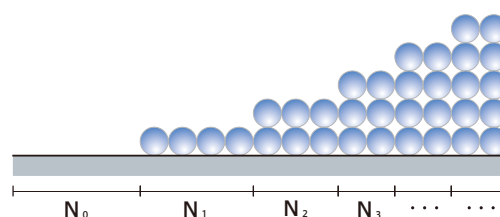
## Adsorption breakthrough curve measurement

The adsorption gas is introduced to the sample, and its concentration is sensed by the detector at the downstream of the sample. Initially, almost all the adsorption gas is adsorbed to the adsorbent and gas concentration detected at the downstream side is nearly zero. When the amount of adsorption to the sample gets close to saturation, the concentration of the gas leaking out to the downstream becomes high and it becomes the same as the injected gas concentration when it reaches saturation. Evaluation of the adsorbent is available by measuring the behavior and adsorption amount until it reaches saturation.



## BET specific surface area

This is surface area per unit weight of solid and in the case of solid catalyst, it largely influences catalytic activity as the contact area with the reactant. Specific surface area is calculated with the BET principle by measuring the nitrogen desorption amount when the sample is cooled down to the liquid nitrogen temperature under the helium-diluted nitrogen gas flow and then returned to room temperature. Not only for solid catalyst, this is a measurement method dispensable for various powder sample evaluations such as adsorbent.



# Software

## Simple operation measurement software

Setting up dedicated tabs for measurement items, fully automatic measurement is available only by inputting simple conditions. Measurement instruments operating status, TCD charts and temperature are displayed in real time and you can check the measurement status at a glance.

## Capable of automatic zero-point adjustment

TCD's zero-point is automatically adjusted before measurement. Measurement is always performed at the same baseline and data comparison is easy. Continuous measurements with different carrier gases can be accurately performed.

## High reliability with automatic multi-point calibration

Highly reliable multi-point calibration is automatically performed after the TPD or TPR measurements. Calculations are also automatic not requiring cumbersome manual calculations.

## Sequence measurement mode

Valve operation, gas flow amount and sample temperature, etc. can be freely programmed to control. Any measurement is available with its own measurement program and it can also be applied to complex catalyst reaction experiments.

## BELMass link software (option)

BELCAT II measurement software can be linked with online gas analyzer, BELMass. While capturing the sample temperature, an analysis linked with the measurement start and end of BELCAT II is performed. Also, it supports the external signal capturing and timer control and a single operation is also available while connected to other equipment.

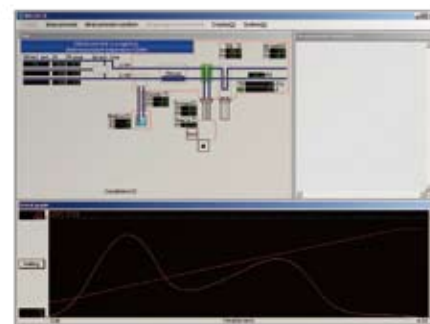
## Waveform analysis software

Peak area is calculated from the spectrum obtained from the TPD and TPR measurement. The layer over-writing function is equipped and the spectrum comparison is easy. Peak separation is available with the waveform decomposition function.

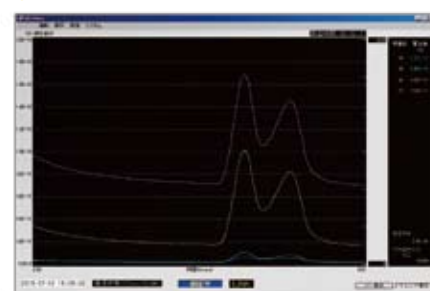
## Pulse analysis software

The measurement software makes complex calculations such as adsorption amount - and metal dispersion rate, etc. and creates the measurement results report is automatically created. Using the recalculation function, metal supported amount or the stoichiometry factor can be changed after the measurement.

### BELCATII measurement view



### BELMass measurement view



### Waveform decomposition view (TPD, TPR measurement)



## Data output examples (Pulse measurement)

### 1 Label

Measurement date, File name, Sample name, Weight, etc.

### 2 Supported metal species

Atomic weight, Density, Supported amount, Stoichiometric factor of the metal, etc.

### 3 Analysis result

Adsorption amount (cm<sup>3</sup>/g)      Metal dispersion rate (%)  
Metal surface area (m<sup>2</sup>/g)      Metal surface (m<sup>2</sup>/g)  
Average particle size (nm)

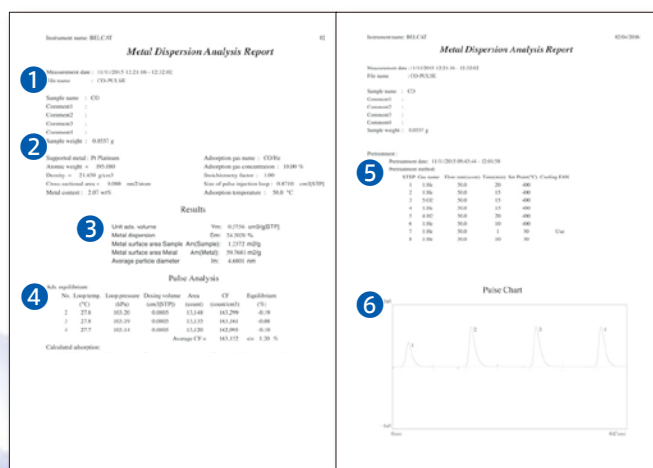
### 4 Pulse details

Dosed amount, Peak area, Equilibrium rate, etc. for each pulse peak

### 5 Pretreatment conditions

Pretreatment gas species, Temperature, Time, etc.

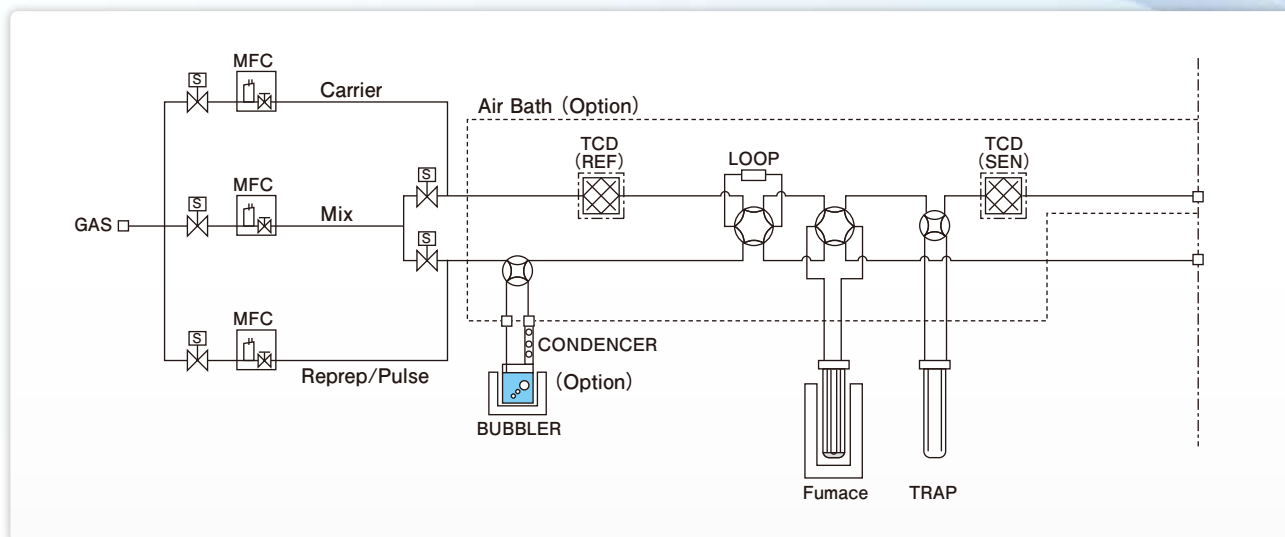
### 6 Pulse chart





## Flow pass figure

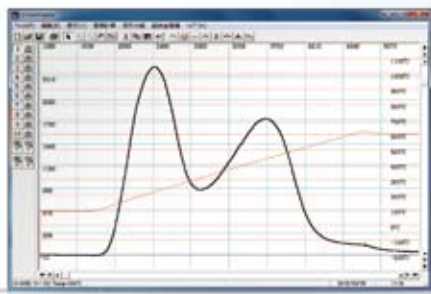
The gas injection system is equipped with the gas mixing line, in addition to the carrier line, pretreatment/pulse gas line. Gas in the mixing line can be mixed to any of the carrier lines and pretreatment/pulse gas line. Various applications are available such as TPR measurement with mixture of H<sub>2</sub> to Ar as a carrier, injection amount control with the diluted pulse gas, etc. Also, by implementing the pretreatment line, contamination of the detector by degassing from the sample can be prevented. Stable data retrieval is available over a long period of time.



※Partially simplified flow pass figure

## Measurement Examples

### NH<sub>3</sub>-TPD measurement result of type MFI zeolite

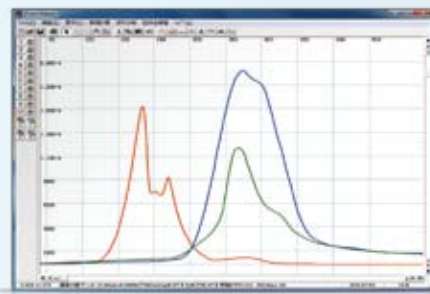


Desorption amount: 0.861mmol/g  
Peak temperature: 440°C

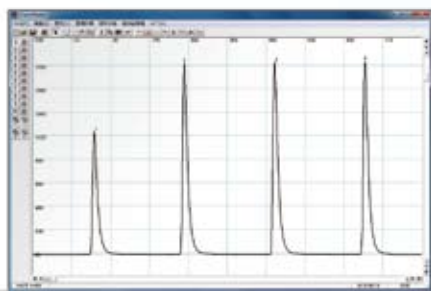
※H peak value

### TPR measurement of various metal oxides using H<sub>2</sub>/Ar

- CuO+H<sub>2</sub> → Cu+H<sub>2</sub>O
- NiO+H<sub>2</sub> → Ni+H<sub>2</sub>O
- CoO+H<sub>2</sub> → Co+H<sub>2</sub>O



### Pt/Al<sub>2</sub>O<sub>3</sub> metallic dispersion degree by CO peak

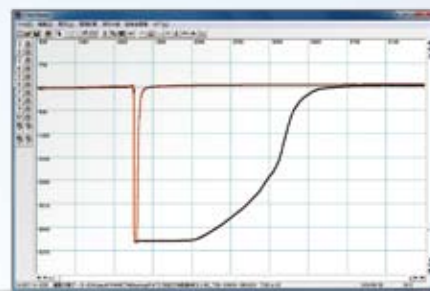


Adsorption amount: 0.566cm<sup>3</sup>/g  
Metal surface area: 1.22m<sup>2</sup>/g

Metal dispersion degree: 25.4%  
Metal particle size: 4.7nm

### NH<sub>3</sub> adsorption breakthrough curve measurement

- Black
- Sample



Adsorption amount: 2.6mmol/g  
5% break point: 714sec

95% break point: 1627sec

## Options

### Vapor dosing unit

Consisting of an air thermostat unit, bubbling, heater and condenser. The condenser eliminates excess vapor and vapor at an accurate and stable concentration can be injected.

### Gas mixing unit

Mixing 3 kinds or more gases. A unit can install up to 3 lines. The corrosive gas is supported.

### Low temperature electric furnace/ CATCryo II

By applying the liquid nitrogen spray, the sample temperature can be continuously controlled from  $-120^{\circ}\text{C}$ . This can be utilized for the metal dispersion rate measurement for the ceria carrier and TPR measurement under room temperature. The cooling performance is improved with the optimal internal structure and the liquid nitrogen consumption is drastically reduced.

### On-line gas analyzer/BELMass

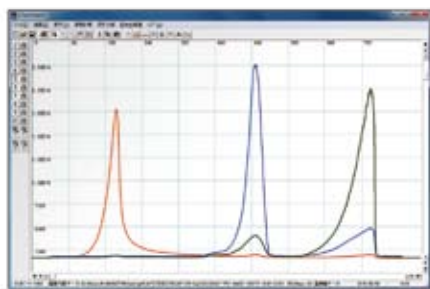
Systemized quadrupole mass spectrometer, BELMass can be connected with BELCAT II. Multiple components of gases unable to separate by TCD can be measured at high quantitiveness while linked with the BELCAT II software. It can also be utilized for the catalytic reaction analysis



## Measurement examples using the options

### Thermal decomposition measurement of calcium oxalate monohydrate

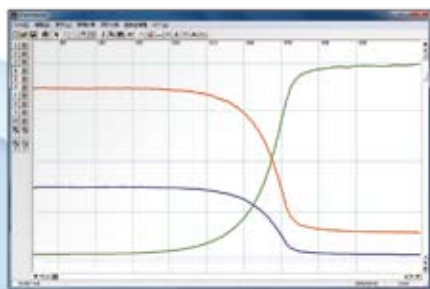
- $m/z=18(\text{H}_2\text{O})$
- $m/z=28(\text{CO})$
- $m/z=44(\text{CO}_2)$



Used option: BELMass

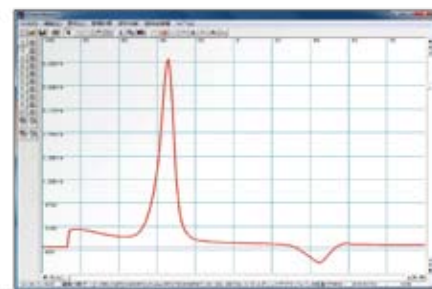
### Temperature - programmed oxidation reaction of carbon monoxide using precious metal catalyst

- $m/z=28(\text{CO})$
- $m/z=32(\text{O}_2)$
- $m/z=44(\text{CO}_2)$



Used option: MIX gas unit, BELMass

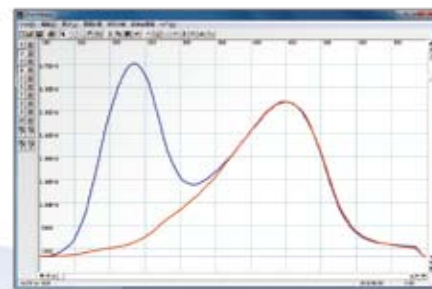
### Temperature-programmed reduction (TPR) of supported palladium catalyst from low temperature ( $-100^{\circ}\text{C}$ )



Used option: CATCryoII

### Water vapor -treatment for zeolite at $\text{NH}_3$ -TPD ( $m/z=16$ )

- Without vapor treatment
- With vapor treatment



Used option: Vapor dosing unit, BELMass

## Accessories



Model number	Description
062-20000-0-0	Sample cell holder
062-20001-0-0	Quartz triple sample tube set (outer tube, inner tube, casing tube)
062-20002-0-0	Quartz triple sample tube (outer tube)
062-20003-0-0	Quartz triple sample tube and trap tube (inner tube) 3 pcs/set
062-20004-0-0	Quartz triple sample tube (casing tube) 3 pcs/set
062-20005-0-0	Trap tube (outer tube)
062-20006-0-0	Bubbling bottle
062-20007-0-0	Clip for bubbling bottle
062-20012-0-0	Type K thermocouple
062-20013-0-0	Filter (6mm in diameter, 5μ) 3 pcs/set
900-00040-0-0	Perfluoro O-ring (6 pcs/set) <P10A> for inner tube
900-00050-0-0	Perfluoro O-ring (3 pcs/set) <P20> for Outer tube
900-00052-0-0	Perfluoro O-ring (6 pcs/set) <P4> for casing tube
900-20061-0-0	P21 - Perfluoro O-ring (6 pcs/set)
900-20062-0-0	P29 - Perfluoro O-ring (6 pcs/set)
900-20063-0-0	P9 - Perfluoro O-ring (6 pcs/set)
900-10006-0-0	Dewer Vessel
900-30000-1-0	Quartz glass wool (fine) 10g
060-20049-0-0	Reference sample for metallic dispersion measurement 2wt%Pt/Al <sub>2</sub> O <sub>3</sub>
060-20050-0-0	Reference sample for NH <sub>3</sub> -TPD measurement Type MFI zeolite
990-00004-0-0	Reference sample for specific surface area measurement Carbon black

## Specifications

### Catalyst analyzer/BELCAT II

Measurement principle		Normal pressure flow method
Detector		Semi-diffusion type 4-element thermal conductivity detector (TCD)
Measurement/pretreatment port		1
Injection gas		He, Ar, N <sub>2</sub> , O <sub>2</sub> , H <sub>2</sub> , CO, CO <sub>2</sub> , NH <sub>3</sub> , N <sub>2</sub> O, NO, etc...
Gas	Carrier	3
	Pretreatment/Pulse/MIX	8 (Corrosion-resistant × 2)
Mass flow Controller	Carrier	F.S.100sccm
	Pretreatment/Pulse	F.S.100sccm
	MIX	F.S.30sccm
Electric furnace		Regular temperature: 1,100°C,
		Maximum temperature: 1,200°C
		Quick cooling: 30min (400→50°C)
		CATCryoII (option) -120°C supported
Vapor injection (option)		H <sub>2</sub> O, CH <sub>3</sub> OH, C <sub>2</sub> H <sub>5</sub> OH, toluene, benzene, etc...
Dimensions, Weight		500(W) × 750(H) × 500(D)mm, 80kg
Utility	Gas	Measurement gas: 0.1MPa(G), Valve driving: 0.45~0.55MPa(G) Joint: 1/8" Swagelok connection
	Power	AC100V, 1,300W

### On-line gas analyzer/BELMass

Measurement principle	Quadrupole mass spectrometer
Detector	Faraday cup/Secondary electron multiplier
Measureable mass range	1~200a.m.u.
Measureable limit	<1ppm*
Gas inlet	1/16-inch capillary tube
Gas inlet temperature	Up to 150°C

※Limited depending on injection gas type

Gas suction volume	1cc/min or less
Measurement channel	Up to 16ch
Analog input	DC 0-10V
Communication interface	RS-232C
Dimensions, Weight	216(W) × 368(H) × 717(D)mm, 36kg
Power	AC100V, 600W



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