

ChemBET-3000

Chemisorption Analyzer for Metal Area,
TPR, TPO, TPD plus BET Surface Area



Quantachrome
INSTRUMENTS

QUANTACHROME

ChemBET™ -3000 Series Overview

CHEMBET 3000 TPR/TPD

Affordable catalyst characterization in a compact package.

Features five flow methods of analysis: three temperature programmed analyses (TPR, TPO and TPD), pulse titration and physisorption (BET surface area) for maximum resolution.

A specially designed high-temperature furnace, combined with a programmable controller, ensures linear heating ramps for reliable temperature programmed analysis (TPA) profiles and activation energy calculations.

The unique sample cell holder permits both in-situ monitoring of sample temperature and sampling of the gas (just downstream of the sample) into an optional mass-spectrometer. The reusable quartz sample cell is both easy to load and clean.

Furnace control plus temperature and detector signals are integrated into a single interface housing that also serves as a convenient monitor stand.

TPRWin (The ChemBET's Windows® compatible software) captures the signals for archiving and report generation.

Plumbed with stainless steel for maximum chemical compatibility, the ChemBET 3000 TPR/TPD is ideal for use with a wide range of gases e.g. ammonia for acid-site strength determination by TPD, oxygen for TPO characterization of diesel soot and exhaust catalysts, hydrogen and carbon monoxide for fuel cell catalysts, nitrous oxide (N₂O) titration for copper surface area determinations, etc.

The injection loop is a standard feature and allows for rapid titration of metal surfaces. Each ChemBET is supplied with a variety of loop sizes, which can be swapped out with a simple tool.

Multiple gas inputs and a minimum-volume selector valve ensure flexibility when switching between protocols for different catalysts. Pretreatment temperatures of up to 450°C are afforded by the compact quartz heating mantle and built-in set-point temperature controller. A diversion valve and cold trap (between sample cell and detector) are included to remove unwanted gaseous products.

This cost effective flow sorption analyzer hosts numerous features and a wealth of capability. Straightforward operation and accessibility of controls makes it an ideal choice for routine metal area/dispersion measurements in industry, universities and colleges.



Measurement Capabilities

TPR: Temperature Programmed Reduction

Many heterogeneous catalysts are used as the zero-valence metal, but start life as the oxide. An important factor in catalyst design and use is the ease of reduction of the metal oxide and TPR is a direct measure of that. A reducing gas mixture, say 2%-5% H₂ in N₂, flowing over the oxide will cause reduction at some point as the temperature is raised using a linear heating ramp. The signal caused by consumption of hydrogen represents the rate of reaction and goes through a maximum at a temperature that is characteristic of both the oxide and the heating rate.

Repeating the same analysis on a fresh sample at a different heating rate is the means by which activation energy for the process can be evaluated. Low loadings of metal oxides, especially surface oxides, generate little water and a successful analysis can be done without trapping it. Larger amounts of moisture generated by the reduction of bulk oxides can be trapped prior to reaching the detector to leave a clean signal based solely on the change in hydrogen concentration.

TPO: Temperature Programmed Oxidation

Carbons and carbides are amenable to evaluation by careful oxidation while being heated. A stream of diluted oxygen (e.g. 2-10% O₂ in He) directed over the sample during a linear heating ramp generates a signal due to the loss of O₂ from the gas stream. The products of oxidation, CO and CO₂, need not be trapped. The specially chosen filaments used in the ChemBET's standard TCD detector are resistant to oxidation and operate normally in the suggested gas mixtures.

Different forms of carbon such as amorphous, nanotube, filament and graphitic, oxidize at different temperatures due to varying availability of reactive carbon-carbon bonds. In this way, fullerenes, soots, cokes on catalysts, etc can be quickly characterized and differentiated. Oxidation catalysts, e.g. those incorporating chromium, cobalt, copper and manganese, and redox supports like ceria can also be characterized by TPO.

TPD: Temperature Programmed Desorption

Species previously adsorbed can be desorbed into a stream of pure carrier gas to generate a characteristic fingerprint. The most common application is ammonia TPD, by which one can evaluate relative acid site strength of, for example, zeolites. Basic sites can similarly be evaluated by TPD of carbon dioxide.

Some materials may be characterized by decomposition, or dissociation, of the bulk solid, not merely by desorption from the surface. Such examples include carbonates resulting from CO₂ removal studies, hydrides used as potential hydrogen storage materials, etc.

Pulse Titration: Quantitative Analysis

This technique is used to determine the following data:

- (i) strong chemisorption uptake, (ii) active metal area, (iii) metal dispersion,
- (iv) average nanocluster (crystallite) size.

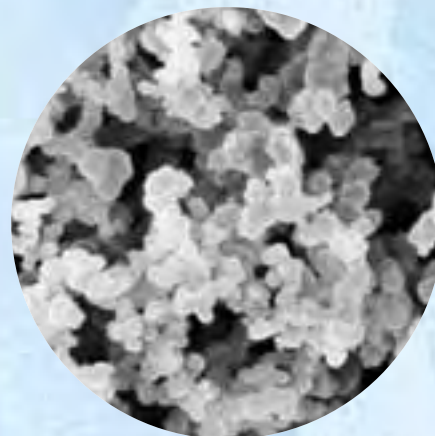
After suitable in-situ preparation, which may be combined with TPR/TPO, the sample is titrated with small, known volumes (pulses) of reactive gas. The detector senses the excess gas which does *not* react with the sample. The total volume of gas which *does* react with the sample is automatically determined by simple back calculation using TPRWin™ software.

B.E.T. Surface Area: Physisorption

The ChemBET 3000 can determine total (B.E.T.) surface area with remarkable sensitivity. By flowing various mixtures of nitrogen and helium over the sample cooled with liquid nitrogen, the surface area can be determined from 0.1 square meters upwards. Using mixtures of krypton and helium the limit of detection is extended down to 0.01 square meters. A single point B.E.T. result can be obtained in under ten minutes. TPRWin software records the signals automatically, computes the B.E.T. "C" constant, y-intercept, slope and correlation coefficient of the least-squares best-fit.



▲ Industrial Catalysts



▲ Carbons

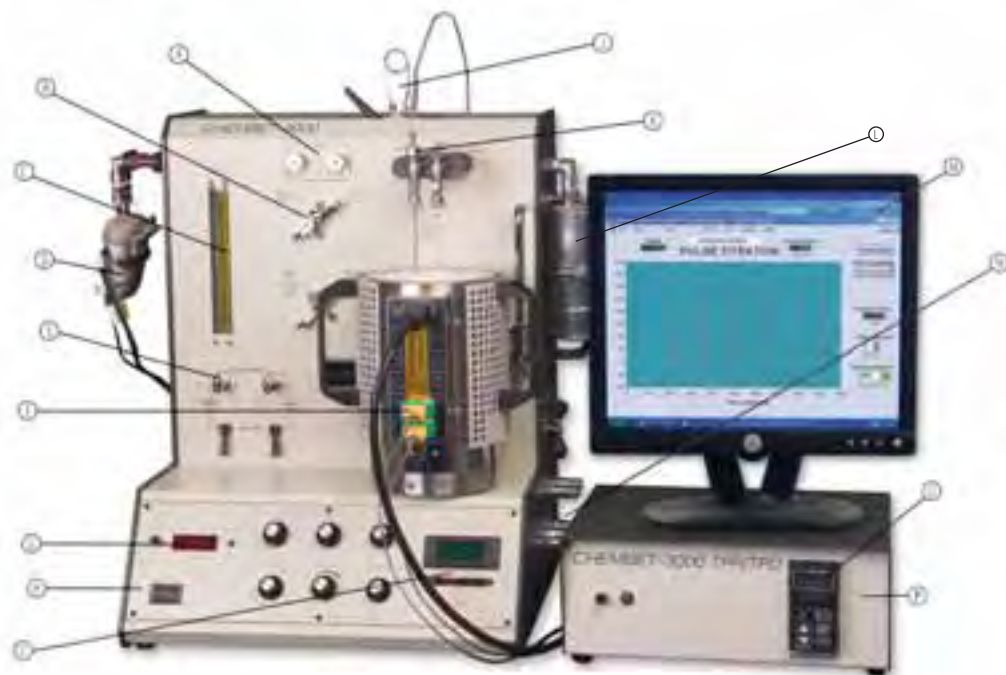


▲ Zeolites



▲ Supported Metals

Instrument and Software Features



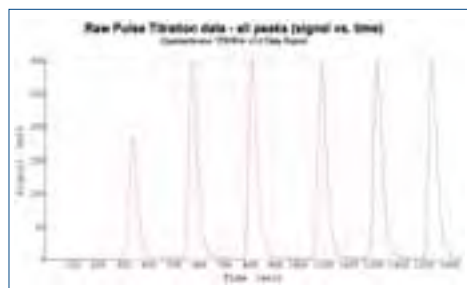
Conveniently placed displays and controls ensure ease of operation. Occupying less than 0.5m² of benchtop, including interface housing and monitor, the ChemBET 3000 TPR/TPD conserves valuable laboratory space. PC and monitor supplied separately.



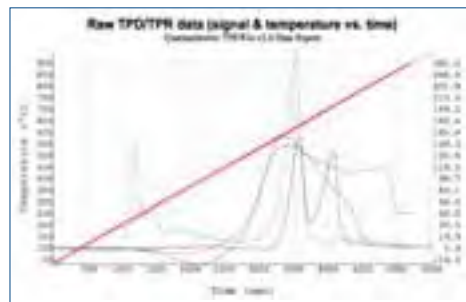
User-friendly software not only records and displays both detector signal and temperature in real time, but also offers a wealth of data reduction and report generation options. Once gas flow is established and, if appropriate, the heating ramp is started each analysis type is controlled by a few mouse-clicks. Graphs generated during an analysis rescale automatically for optimum readability.

TPA profiles can be presented as signal and temperature versus time, or signal versus temperature according to your preference. Multiple profiles can be overlaid for comparison of different samples for example or, when obtained at different heating rates on the same material, used in the Kissinger (Redhead) equation for calculation of activation energy according to first-order kinetics.

Metal area calculations can be setup to automatically select calibration peaks and the associated nanocluster (crystallite) size calculation features an adjustable particle shape factor. Monolayer capacity, metal area, nanocluster size and dispersion are conveniently



▲ Rapid titration and extreme sensitivity.



▲ TPA overlays for easy comparison.

reported together in a single table. All reports feature a detailed header which includes analysis and data reduction parameters, and which can be customized with the name of your company or institution.

Data and results can be copied and pasted into other Windows[®]-compatible programs for presentation and publishing, and complete reports can be archived in pdf and mdi formats using appropriate document software.

ChemBET-3000 Includes:



- A. Calibration Port
- B. Bypass Valves
- C. Flow Rate Meter
- D. Degassing/Prep Station
- E. Flow Rate Control
- F. 1100°C Furnace
- G. Peak Area Display
- H. Set-Point Controller
- I. Multi-Function Display
- J. Interchangeable Injection Loop
- K. In-Situ Sample Temperature Monitoring
- L. Vapor Trap
- M. TPRWin™ Software (PC and monitor supplied separately)
- N. Multiple Gas Inputs
- O. PID Furnace Controller
- P. Signal Conditioning

TPRWin™ Includes:

- On-screen prompts
- Real-time signal and temperature display
- Metals database
- Gases database
- Peak area integration
- Signal baseline adjustment post-acquisition
- Automatic calibration peak selection with manual override
- Monolayer capacity calculation
- Metal area calculation
- Metal dispersion calculation
- Nanocluster (crystallite) size calculation
- Activation energy calculation
- Single-point BET calculation
- Multi-point BET calculation
- Customizable, automatic naming of data files
- Customizable report font selections
- User-selectable plot colors
- Graph zoom and adjustable scaling
- Data import into popular external spreadsheet and plotting programs
- For Windows[®] XP

Instrument Specifications

Capability

| | |
|------------------------------|---|
| BET Surface Area | ✓ |
| Pulse Titration (metal area) | ✓ |
| TPR | ✓ |
| TPO | ✓ |
| TPD | ✓ |

Features

| | |
|--|--------|
| Injection Loop | ✓ |
| Calibration Port | ✓ |
| Quartz Glassware | ✓ |
| Self-sealing Sample-Cell Holders | ✓ |
| Stainless-Steel Plumbing | ✓ |
| Multiple-Gas Selector Valve | ✓ |
| Variable Gas Flow Rate Control | ✓ |
| Sample Cell Bypass | ✓ |
| In-Line Cold Trap with Bypass | ✓ |
| Supplementary Outgas/Preparation Station | ✓ |
| Mass Spec Connection Port | ✓ |
| User Adjustable Detector Sensitivity | ✓ |
| Multi-Function Display | ✓ |
| Built-In Peak Area Integrator | ✓ |
| High Temperature (450°C) Heating Mantle | ✓ |
| High Temperature (1100°C) Furnace | ✓ |
| PID Programmable Controller | ✓ |
| In-Situ Sample Thermocouple | ✓ |
| Data Acquisition Interface & Software | ✓ |
| Mass Flow Controller/ Gas Blender | option |

Physical

| | |
|-------------------|-------------------------------|
| ChemBET Weight: | 24 kg (excluding accessories) |
| ChemBET Height: | 61 cm (excluding loop) |
| ChemBET Width: | 47 cm (at benchtop level) |
| ChemBET Depth: | 27.5 cm (excluding fittings) |
| Interface Weight: | 3.25 kg |
| Interface Height: | 15 cm |
| Interface Width: | 36 cm |
| Interface Depth: | 27.5 cm (excluding fittings) |

Environmental

| | |
|----------------------|-------------------------|
| Ambient Temperature: | 15-40°C |
| Relative Humidity: | 20-90% (non-condensing) |

Hardware

| | |
|-------------------------------|---|
| Thermal Conductivity Detector | Dual-Filament Diffusion Type |
| TCD Filaments | Oxidation and Ammonia Resistant |
| Filament Current | Adjustable, Continuously Variable |
| Sensitivity, Dynamic Range: | 512 |
| Gas Input Ports | 5 |
| Loop Volumes Supplied | 50, 100, 250 μL (others available) |

Performance

| | |
|-----------------------|--|
| Volume Adsorbed | 0.001 to $>100\text{cm}^3$ |
| Specific Volume | 0.0001 cm^3/g |
| Total Surface Area | 0.1 to 280 m^2 |
| Specific Surface Area | 0.01 m^2/g to upper limit set only by weighing accuracy of smallest sample |
| Pore Volume | 0.0001 to 0.15 cm^3 |
| Accuracy, Volume | $\pm 1\%$ |
| Reproducibility | 0.5% |

Gases

| | |
|-------------------------|---|
| Compatibility: | $\text{H}_2, \text{O}_2, \text{CO}, \text{CO}_2, \text{NO}, \text{N}_2\text{O}, \text{NO}_2, \text{SO}_2, \text{NH}_3, \text{N}_2, \text{Ar}, \text{Kr}, \text{He}$ |
| Input Pressure (gauge): | 70-140 kPa (10-20 psig) |
| Gas Lines: | 5 x 1.5m 1/8" s.s. (supplied) |
| Vent: | hosebarb connector |

Heating

| | |
|--|------------------------|
| Mantle, Max temp | 450°C |
| Mantle Power | 125 W |
| Set-point Controller | 1 degree increments |
| Furnace, Max Temp | 1100°C |
| Furnace Power | 575 W |
| Controller Type | Full PID, programmable |
| Controller Tuning | Automatic |
| Program Steps | Ramp, Hold (soak) |
| Ramp Types | Rate, Time |
| Furnace Heating Rate, up to 500°C | 1 to 100 deg/min |
| Furnace Heating Rate, 500°C to 750°C | 1 to 50 deg/min |
| Furnace Heating Rate, 750°C to 1000°C | 1 to 30 deg/min |
| Furnace Heating Rate, 1000°C to 1100°C | 1 to 20 deg/min |

Electrical

| | |
|------------------------|---------------|
| Voltage: | 100 - 240 VAC |
| Frequency: | 50/60 Hz |
| Power (excl. furnace): | 70 VA |

Accessories

Reference Material CHRM7001

A typical catalyst sample, 1% Pt dispersed on 1/8" alumina pellets, is available for ChemBET qualification and ongoing performance verification. Each bottle of reference material comes with a detailed data sheet and instructions.

Gas Regulator Assembly

Proper ChemBET functioning is assured when high-quality gas regulators are used. Quantachrome supplies complete assemblies which include two-stage regulators with dual gauges, cylinder connector, isolation valve and 1/8" gas line connector. The regulators feature stainless steel, non-venting diaphragms and the appropriate CGA fitting for specific gases. Different assemblies are available for nitrogen and other inerts including helium, hydrogen, carbon monoxide, oxidizing gases etc.



Rotary Micro Riffler

Like most powder and porous materials characterization, chemisorption studies generally require sub-samples much smaller than the original samples. The Rotary Micro Riffler uses the most accurate way of splitting a powder sample into smaller fractions- spin riffling. The vibrating hopper features adjustable feed rate and the variable-speed collector uses standard or micro test tubes.



Mass Spectrometer

Your catalyst studies might require identification of multiple gaseous species that the standard ChemBET detector cannot differentiate. In this case, you will need a mass spectrometer complete with its own vacuum station and gas introduction port. Quantachrome can supply such a system, suited to your application needs, with your ChemBET. An optional thermocouple and interface are available for integrated mass and temperature data.

Gas Blender (Mass Flow Controller)

TPA and physisorption measurements require mixed gases, e.g. 5% H_2 in N_2 for TPR or 30% N_2 in He for BET surface area. While tanks of pre-mixed gases are generally readily available, if you want to use a number of different concentrations or want to quantitatively control the gas flow rate, Quantachrome offers this two-channel gas mixer. Simply dial in the required gas flow, up to 20 ml/min, into each of the two precision mass flow controllers. One channel comes ready calibrated for helium and hydrogen, the other for eight different gases including CO, N_2 and CO_2 .





Quantachrome Instruments' corporate headquarters in Boynton Beach, Florida.

Quantachrome®

Renowned innovator of ideas for today's porous materials community.

For more than 37 years, Quantachrome's scientists and engineers have revolutionized measurement techniques and designed instrumentation to enable the accurate, precise, and reliable characterization of powdered and porous materials:

- Adsorption/Desorption Isotherms
- Surface Area Measurement
- Pore Size Distribution
- Chemisorption Studies
- Water Sorption Behavior
- Mercury Porosimetry
- True Solid Density
- Tapped Density

Not only are Quantachrome products the instruments of choice in academia, but the technology conceived and developed by our expert staff is applied in

industrial laboratories worldwide, where research and engineering of new and improved porous materials is ongoing. Manufacturers also rely on porous materials characterization technology to more precisely specify bulk materials, to control quality, and to isolate the source of production problems with greater efficiency.



Quantachrome Instrument's Application Laboratory.

Quantachrome is also recognized as an excellent resource for authoritative analysis of your samples in our fully equipped, state-of-the-art powder characterization laboratory.

WORLDWIDE SALES AND SERVICE

| | |
|-----------------|----------------------|
| Argentina | Mexico |
| Australia | Middle East |
| Austria | Morocco |
| Bahrain | Netherlands |
| Belarus | New Zealand |
| Belgium | Norway |
| Brazil | Oman |
| Bulgaria | Pakistan |
| Canada | Peru |
| Central America | Philippines |
| Chile | Poland |
| China | Portugal |
| Colombia | Puerto Rico |
| Croatia | Romania |
| Cyprus | Russia |
| Czech Republic | Saudi Arabia |
| Denmark | Singapore |
| Egypt | Slovak Republic |
| Estonia | Slovenia |
| Finland | South Africa |
| France | South Korea |
| Germany | Spain |
| Greece | Sri Lanka |
| Hungary | Sweden |
| India | Switzerland |
| Indonesia | Taiwan |
| Ireland | Tanzania |
| Israel | Thailand |
| Italy | Turkey |
| Jamaica | Ukraine |
| Japan | United Arab Emirates |
| Jordan | United Kingdom |
| Kuwait | Uruguay |
| Latin America | Uzbekistan |
| Latvia | Venezuela |
| Lithuania | Vietnam |
| Malaysia | |

CORPORATE HEADQUARTERS

Quantachrome Instruments

1900 Corporate Drive
 Boynton Beach, FL 33426 USA
 Phone: +1 (561) 731-4999
 Fax: +1 (561) 732-9888
 E-mail: qc.sales@quantachrome.com

CHINA

Quantachrome Representative Office

Rm. 1701B, Everbright Building
 No. 6 Fuxing Men Wai Street
 Beijing 100045 China
 Phone: 010-68561530; 010-68560808-172
 Fax: 010-68561530; 010-68029962

EUROPE

Quantachrome UK Limited

Pale Lane Farm, Pale Lane
 Hartley Wintney
 Hook RG27 8BA, UK
 Phone: +44 (0) 1252819719
 Fax: +44 (0) 1252819901

EUROPE

Quantachrome GmbH

Rudolf-Diesel Str. 12
 85235 Odelzhausen, Germany
 Phone: +59 (0) 8134/93240
 Fax: +59 (0) 8134/932425



Serving Porous
 Materials and Powder
 Characterization
 Needs Since 1968



www.quantachrome.com

Trademarks and registered trademarks are the property of their respective owners.

07118