

ChemBET-TPR/TPD

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



Quantachrome
INSTRUMENTS

© 2009-2010 Quantachrome Corporation 0110 07118 REV B

QUANTACHROME

ChemBET-TPR/TPD Overview



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 flexibility.

A specially designed high-temperature furnace, combined with software control, 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.

TPRWin (the ChemBET's Windows®- compatible software) controls gas switching, temperature profiles and also captures the signals for archiving and report generation.

Plumbed with stainless steel for maximum chemical compatibility, the ChemBET 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 quickly and easily swapped out.

Multiple gas inputs with automatic valves ensure flexibility when switching between protocols for different catalysts. A diversion valve and cold trap (between sample cell and detector) are provided for removal of unwanted gaseous products when necessary.

This cost effective flow sorption analyzer hosts numerous features and a wealth of capability. Straightforward operation, PC interface 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% H_2 in N_2 , 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% O_2 in He) directed over the sample during a linear heating ramp generates a signal due to the loss of O_2 from the gas stream. The products of oxidation, CO and CO_2 , 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 CO_2 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.

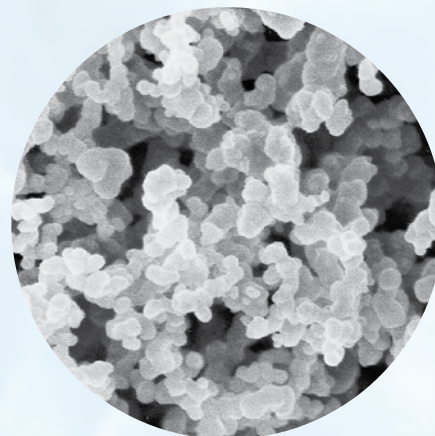
After suitable in-situ preparation, which may be combined with TPR/TPO, the sample is titrated with small, known volumes (pulses) of reactive gas. The detector senses the excess gas which does not react with the sample. The total volume of gas which does react with the sample is automatically determined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

B.E.T. Surface Area: Physisorption

The ChemBET 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 Specifications

Capability

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

Features

Injection Loop (manual)	✓
Calibration Port	✓
Quartz Glassware	✓
Self-sealing Sample Cell Holders	✓
Stainless-Steel Plumbing	✓
Multiple-Gas Selection (automatic)	✓
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	✓
High Temperature (450°C) Heating Mantle	✓
High Temperature (1100°C) Furnace	✓
PC Temperature Control	✓
In-Situ Sample Thermocouple	✓
Data Acquisition Interface & Software	✓
Mass Flow Controller / Gas Blender	option

Physical

Weight:	24 kg (excluding accessories)
Height:	61 cm (excluding loop)
Width:	47 cm (at benchtop level)
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 block
TCD Filament Material	Oxidation and ammonia resistant (W/Re)
Filament Type	Heated, constant current
Sensitivity, Dynamic Range:	512
Gas Input Ports	5
Loop Volumes Supplied	50, 100, 250 µL (others available)

Performance

Volume Adsorbed	0.001 to >100cm 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	± 1%
Reproducibility	0.5%

Gases

Compatibility:	H ₂ , O ₂ , CO, CO ₂ , NO, N ₂ O, NO ₂ , SO ₂ , NH ₃ , N ₂ , Ar, Kr, He
Input Pressure (gauge):	70-140 kPa (10-20 psig)
Gas Lines:	5 x 1.5m 1/8" s.s. (supplied)
Vents:	Hosebarb connector

Heating

Mantle, Max temp	450°C
Mantle Power	125 W
Furnace, Max Temp	1100°C
Furnace Power	575 W
Controller Type	PID via PC
Program Steps	Multiple 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, 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

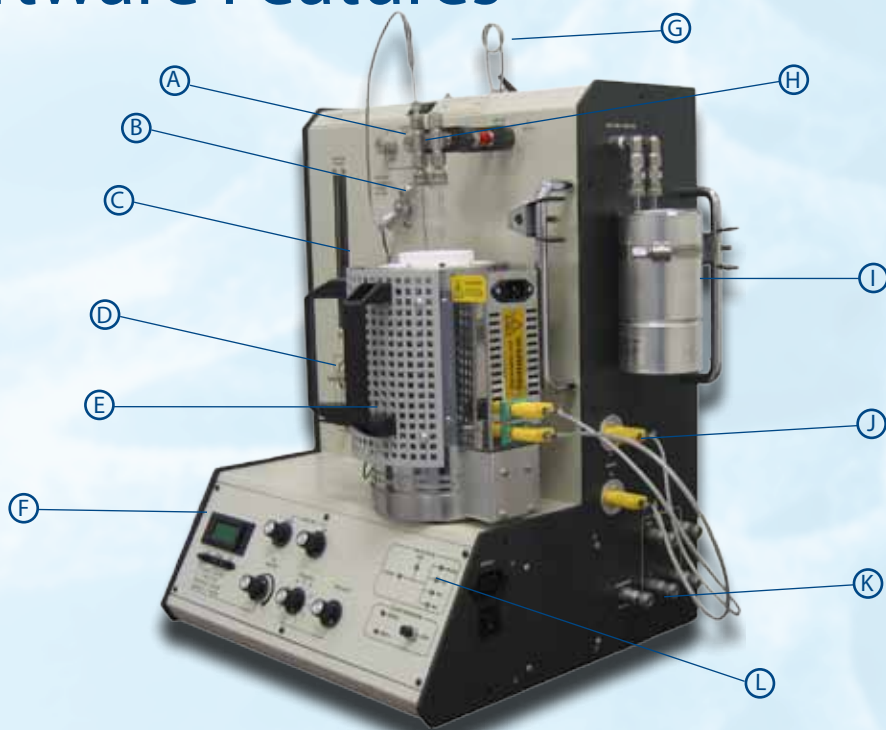
Your catalyst studies might require identification of multiple gaseous species that the standard ChemBET detector cannot differentiate. In this case, you will need a mass spectrometer complete with its own vacuum station and gas introduction port. Quantachrome can supply such a system, suited to your 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% H₂ in N₂ for TPR or 30% N₂ 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, N₂ and CO₂.



Instrument and Software Features

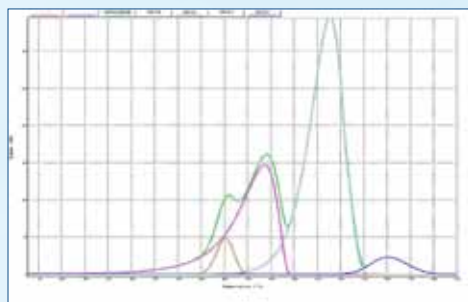


Conveniently placed displays and controls ensure ease of operation. Occupying less than 0.2m² of benchtop, the ChemBET TPR/TPD conserves valuable laboratory space. PC and monitor supplied separately.

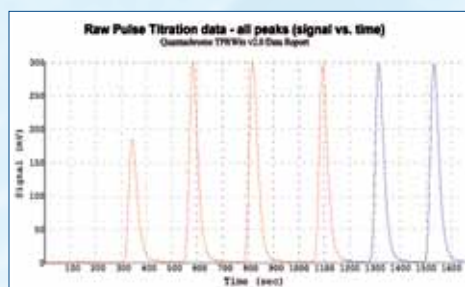
ChemBET-TPR/TPD Includes:



- A. Calibration Port _____
- B. Bypass Valves _____
- C. Flow Rate Meter _____
- D. Flow Rate Control _____
- E. 1100°C Furnace _____
- F. Multi-Function Display _____
- G. Interchangeable Injection Loop _____
- H. In-Situ Sample Temperature Monitoring _____
- I. Vapor Cold Trap _____
- J. Over-Temperature Safety _____
- K. Multiple Gas Inputs _____
- L. Automatic Gas Switching _____



▲ Deconvolution of overlapping TPA peaks.



▲ Rapid titration and extreme sensitivity.

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. 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. Overlapping peaks can be deconvoluted, with or without baseline subtraction.

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 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.

TPRWin™ Includes:

- On-screen prompts _____
- Real-time signal and temperature display _____
- Baseline subtraction (linear, non-linear) _____
- Deconvolution of overlapping peaks _____
- Peak area integration _____
- 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 _____
- Metals database _____
- Gases database _____
- 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, Vista _____



Quantachrome Instruments' corporate headquarters in Boynton Beach, Florida.

Quantachrome®

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

For over 40 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 is also recognized as an excellent resource for authoritative analysis of your samples in our fully equipped, state-of-the-art powder characterization laboratory.



Quantachrome Instruments Application Laboratory.

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
www.quantachrome.com

CHINA

Quantachrome Representative Office

M806. Jingbao Garden
183 Andingmenwai Street
Beijing 100011, China
Tel/Fax: +86 10 64400892
www.quantachrome.com.cn

EUROPE

Quantachrome UK Limited

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

EUROPE

Quantachrome GmbH & Co. KG

Rudolf-Diesel Str. 12
85235 Odelzhausen, Germany
Phone: +49 (0) 8134/93240
Fax: +49 (0) 8134/932425
www.quantachrome.de
www.quantachrome.nl



Quantachrome Instruments' quality management system is certified to be in accordance with ISO9001:2008.

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	

Quantachrome

INSTRUMENTS

Serving Porous
Materials and Powder
Characterization
Needs Since 1968



www.quantachrome.com

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