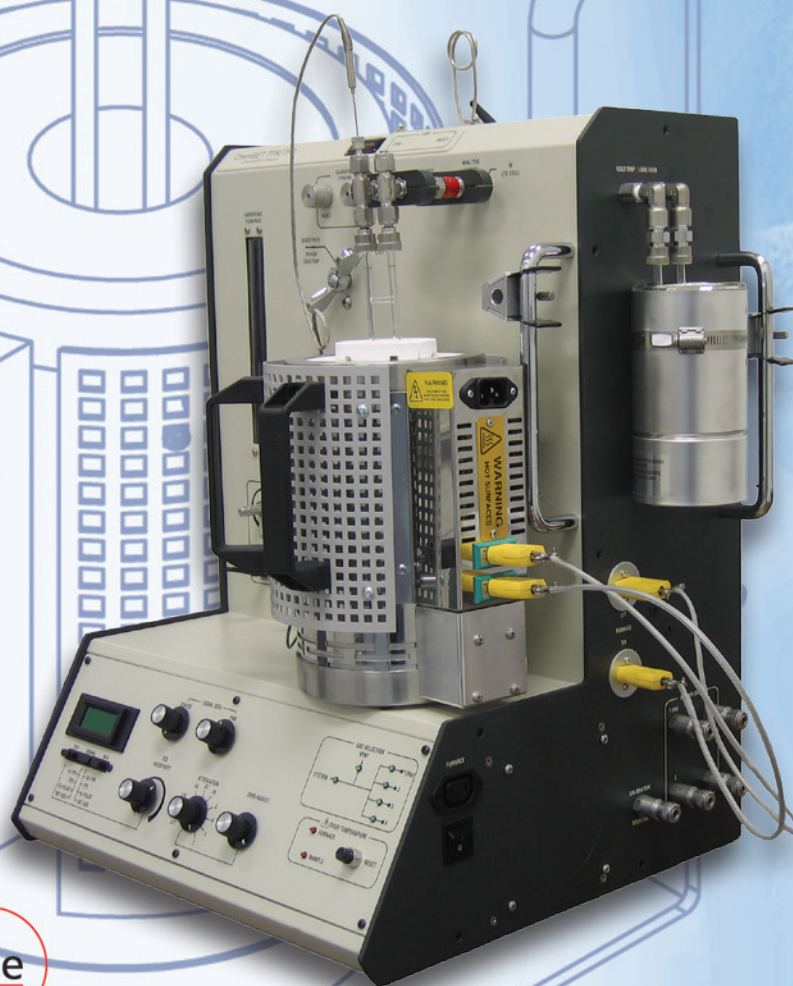


# ChemBET-TPR/TPD

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



**Quantachrome**  
INSTRUMENTS

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# 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 ( $\text{N}_2\text{O}$ ) 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.

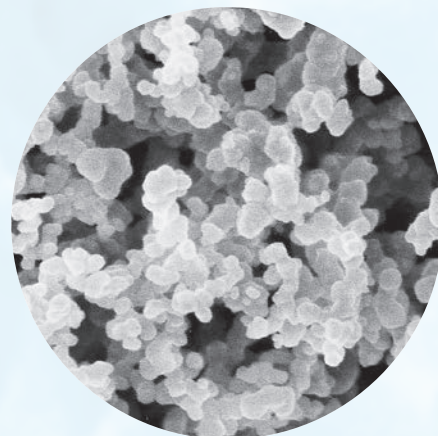
After suitable in-situ preparation, which may be combined with TPR/TPO, the sample is titrated with small, known volumes (pulses) of reactive gas. The detector senses the excess gas which does not react with the sample. The total volume of gas which does react with the sample is automatically determined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

## 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 <sup>3</sup>
Specific Volume	0.0001 cm <sup>3</sup> /g
Total Surface Area	0.1 to 280 m <sup>2</sup>
Specific Surface Area	0.01 m <sup>2</sup> /g to upper limit set only by weighing accuracy of smallest sample
Pore Volume	0.0001 to 0.15 cm <sup>3</sup>
Accuracy, Volume	± 1%
Reproducibility	0.5%

## Gases

Compatibility:	H <sub>2</sub> , O <sub>2</sub> , CO, CO <sub>2</sub> , NO, N <sub>2</sub> O, NO <sub>2</sub> , SO <sub>2</sub> , NH <sub>3</sub> , N <sub>2</sub> , 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.



## Sub-Ambient Option

Allows data to be collected from a starting temperature as low as -100°C. Includes jacketed sample cell, heat exchanger and dual thermocouple.

## 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.

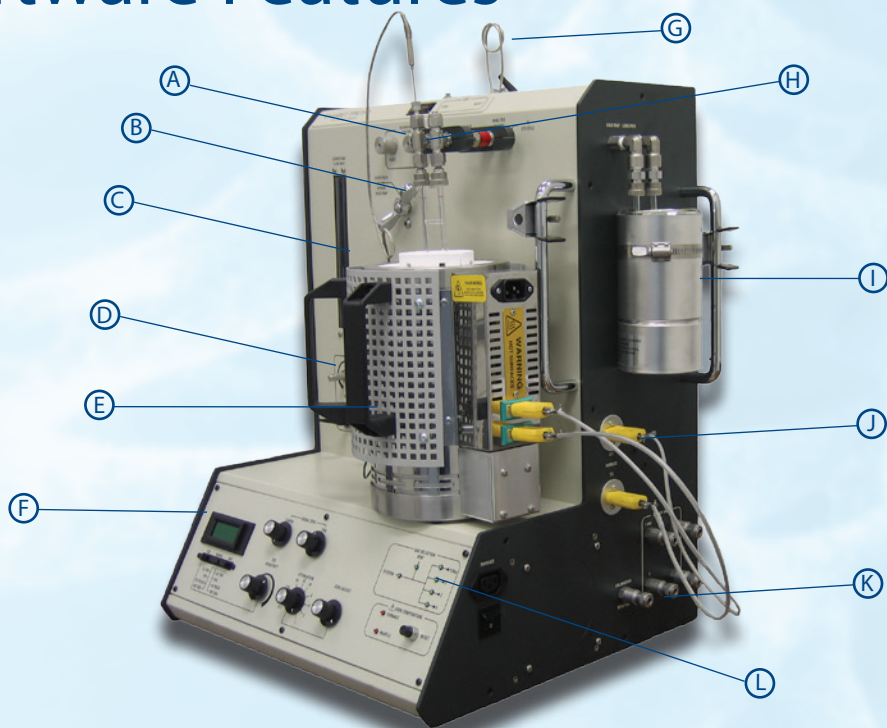
## Gas Blender (Mass Flow Controller)

TPA and physisorption measurements require mixed gases, e.g. 5% H<sub>2</sub> in N<sub>2</sub> for TPR or 30% N<sub>2</sub> 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<sub>2</sub> and CO<sub>2</sub>.

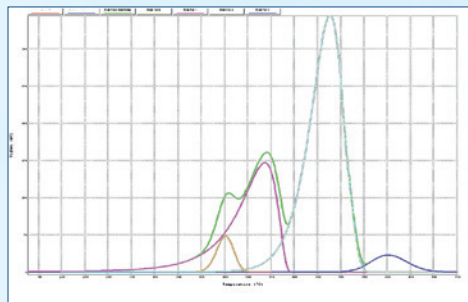




# Instrument and Software Features



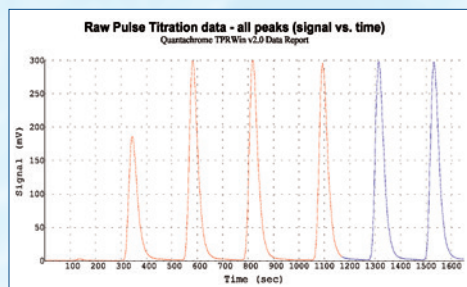
Conveniently placed displays and controls ensure ease of operation. Occupying less than 0.2m<sup>2</sup> of benchtop, the ChemBET TPR/TPD conserves valuable laboratory space. PC and monitor supplied separately.



▲ Deconvolution of overlapping TPA peaks.

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 overlayed 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.

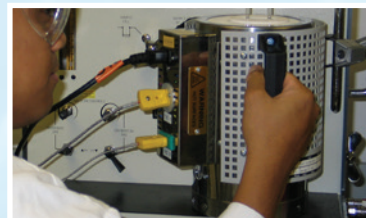


▲ Rapid titration and extreme sensitivity.

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.

## 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

## 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





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### 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](mailto:qc.sales@quantachrome.com)  
[www.quantachrome.com](http://www.quantachrome.com)

### CHINA

Quantachrome Representative Office

M806. Jingbao Garden  
183 Andingmenwai Street  
Beijing 100011, China  
Tel/Fax: +86 10 64400892  
[www.quantachrome-china.com](http://www.quantachrome-china.com)

### 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](http://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](http://www.quantachrome.de)

### JAPAN

Quantachrome Instruments Japan G.K.

KSP W311 Sakado 3-2-1, Takatsu-ku  
Kawasaki-shi, Kanagawa 213-0012  
Japan  
Phone: +81 44 829 1525  
Fax: +81 44 829 1527  
[www.quantachrome.co.jp](http://www.quantachrome.co.jp)



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