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INSTRUMENTS
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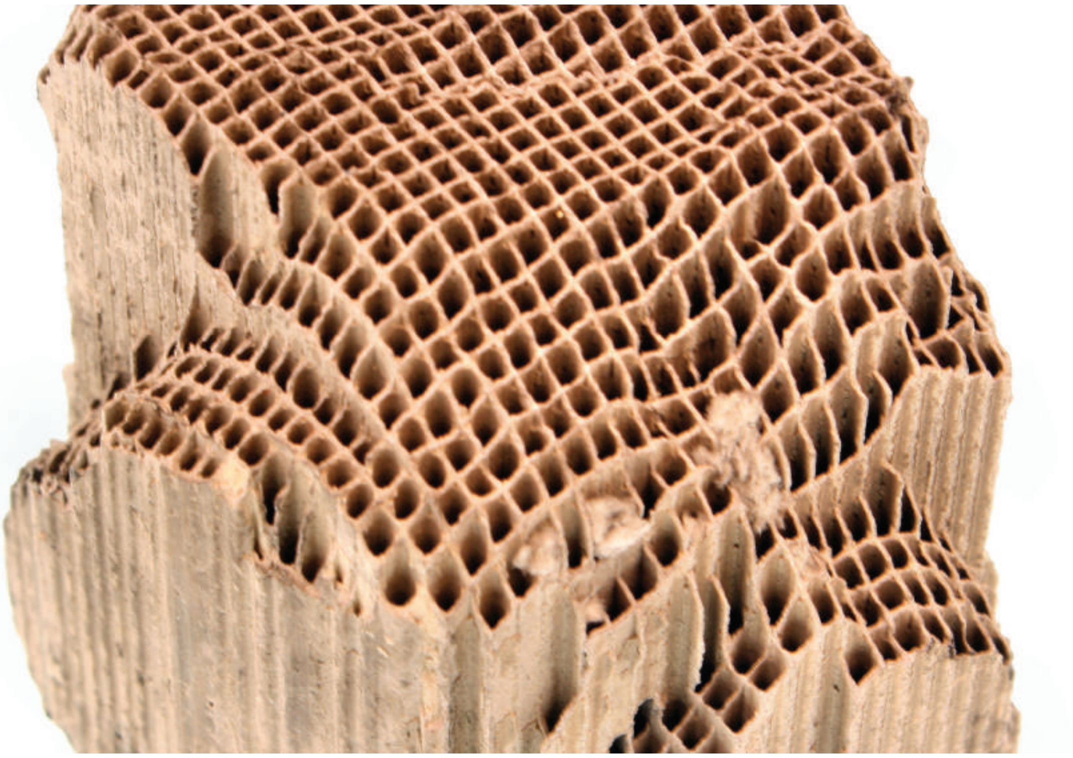
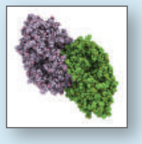
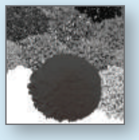
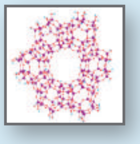
TPR/TPD
ChemBET Pulsar
Automatic Chemisorption Analyzer



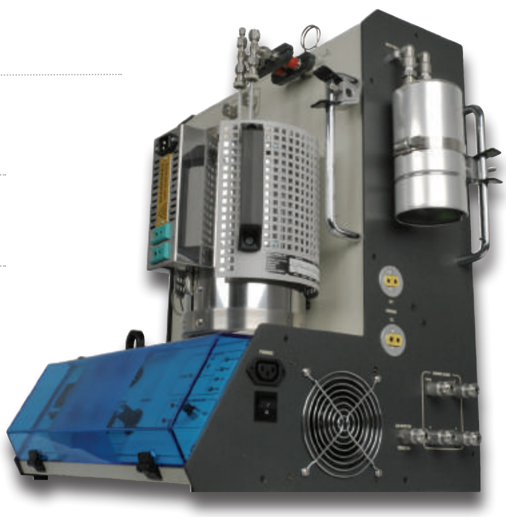
ChemBET Pulsar -TPR/TPD







- Crystallite Size
- Dispersion
- Metal Area
- Pulse Titration
- TPR / TPO / TPD



TPR/TPD ChemBET Pulsar

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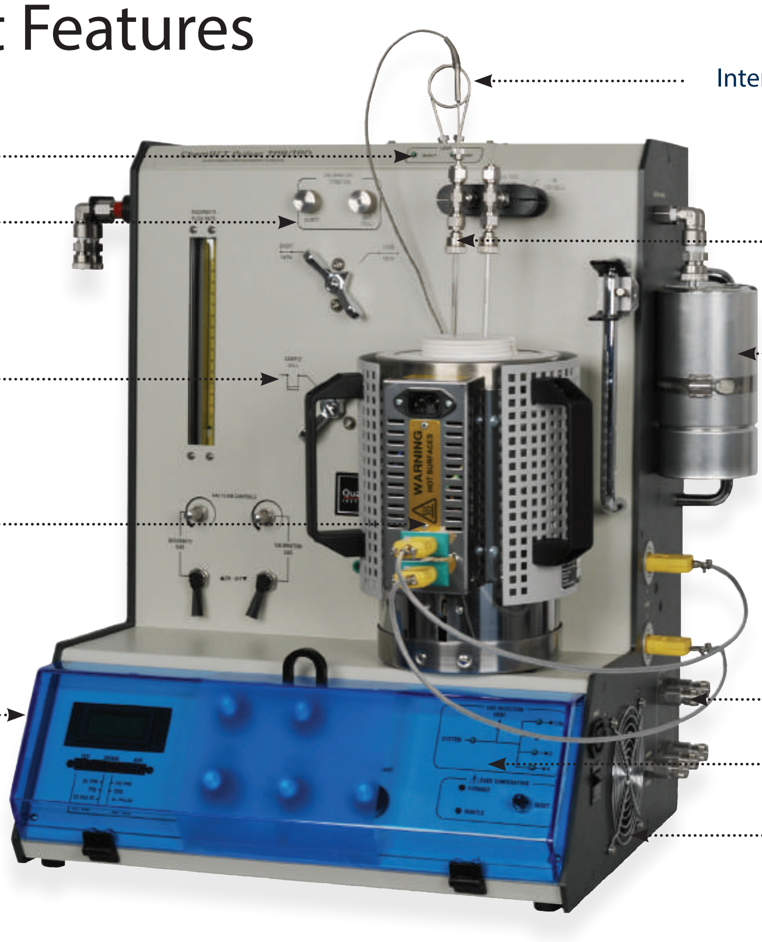
Instrument Features

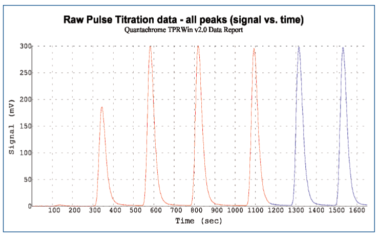


Automation

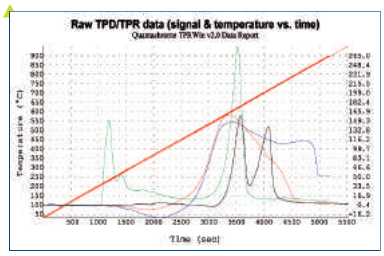
The ChemBET Pulsar TPR/TPD represents the very best in catalyst characterization using automated flow methods of analysis.

Fully automated analysis sequences are programmed using the TPRWin software. Titrations for metal area and dispersion determination use an automatic loop injector and automatic gas switching. Furnace temperature ramping provides for temperature programmed methods and sample preparation, both including rapid furnace cooling using forced air for higher throughput. The Pulsar uses a proven TCD detector both oxidation AND ammonia resistant, with stable current control for baseline stability and reproducible signals. Plumbed in stainless-steel for maximum chemical compatibility, the Pulsar is ideal for use with a wide range of gases. High-temperature quartz sample cells are standard, as is the in-cell thermocouple providing accurate sample temperature measurements.

An optional quadrapole mass spectrometer is available allowing for species differentiation during temperature programmed analyses.



Rapid titration & extreme sensitivity.



TPA overlays for easy comparison.

TPR/TPD

ChemBET Pulsar

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Specifications

Capability (Automatic)

Pulse Titration (metal area)
Temperature programmed Reduction (TPR)
Temperature programmed Desorption (TPD)
Temperature programmed Oxidation (TPO)
Temperature programmed Surface Reaction (TPSR)

Features

Automatic Injection Loop
Automatic Gas Switching between 4 ports 1
Automatic Forced Air Cooling of Furnace Calibration Port 2
Quartz Glassware
Self-sealing Sample Cell Holders
Stainless-Steel Plumbing
Variable Gas Flow Rate Control
Sample Cell Bypass
In-Line Cold Trap with Bypass
Supplementary Outgas/
Preparation Station
Mass Spec Connection Port
High Temperature (350°C) Heating Mantle
High Temperature (1100°C) Furnace
Cell Sample Thermocouple

Software Control

Programming of the following actions creates a customized multi-step "*macro*" which automatically controls the analysis:

Gas switching
Manifold purge
Start/stop signal acquisition
Temperature ramping (by rate)
Temperature ramping (by time)
Multiple heating/cooling profiles
Cooling fan on/off
Pulse injection

The following data are presented on screen in real time and automatically stored:

TCD signal
Sample temperature
Time

Utilities

Gas Compatibility:	H_2, O_2, CO, CO_2, N_2O, 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)
Voltage:	100 - 240 VAC
Frequency:	50/60 Hz
Power:	70 VA
Mantle, Max Temp:	350°C
Mantle Power:	125 W
Furnace, Max Temp:	1100°C
Furnace Power:	575 W

Hardware

Thermal Conductivity Detector:	Dual-Filament Diffusion Type
TCD Filaments:	Oxidation and Ammonia Resistant
Furnace Cooling:	Forced air (PC Controlled)
Gas Input Ports:	5
Loop Volumes Supplied:	50, 100, 250 µL (others available)

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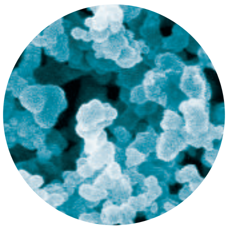
Measurement Capabilities & Applications

Industrial Catalysts
(eg. Hydrocracking, Hydrodesulfurization, Hydrodenitrogenation and Fischer-Tropsch)



Carbons, Fuel Cells, etc.

Zeolites
(eg. FCC, Isomerization)

Supported Metals

(Reforming, Partial Oxidation, Hydrogenation, Automotive Exhaust, etc.)

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Serving Porous Materials and Powder Characterization Needs Since 1968

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 Pulsar's 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 finger-print. 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 automatically 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 Pulsar 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.