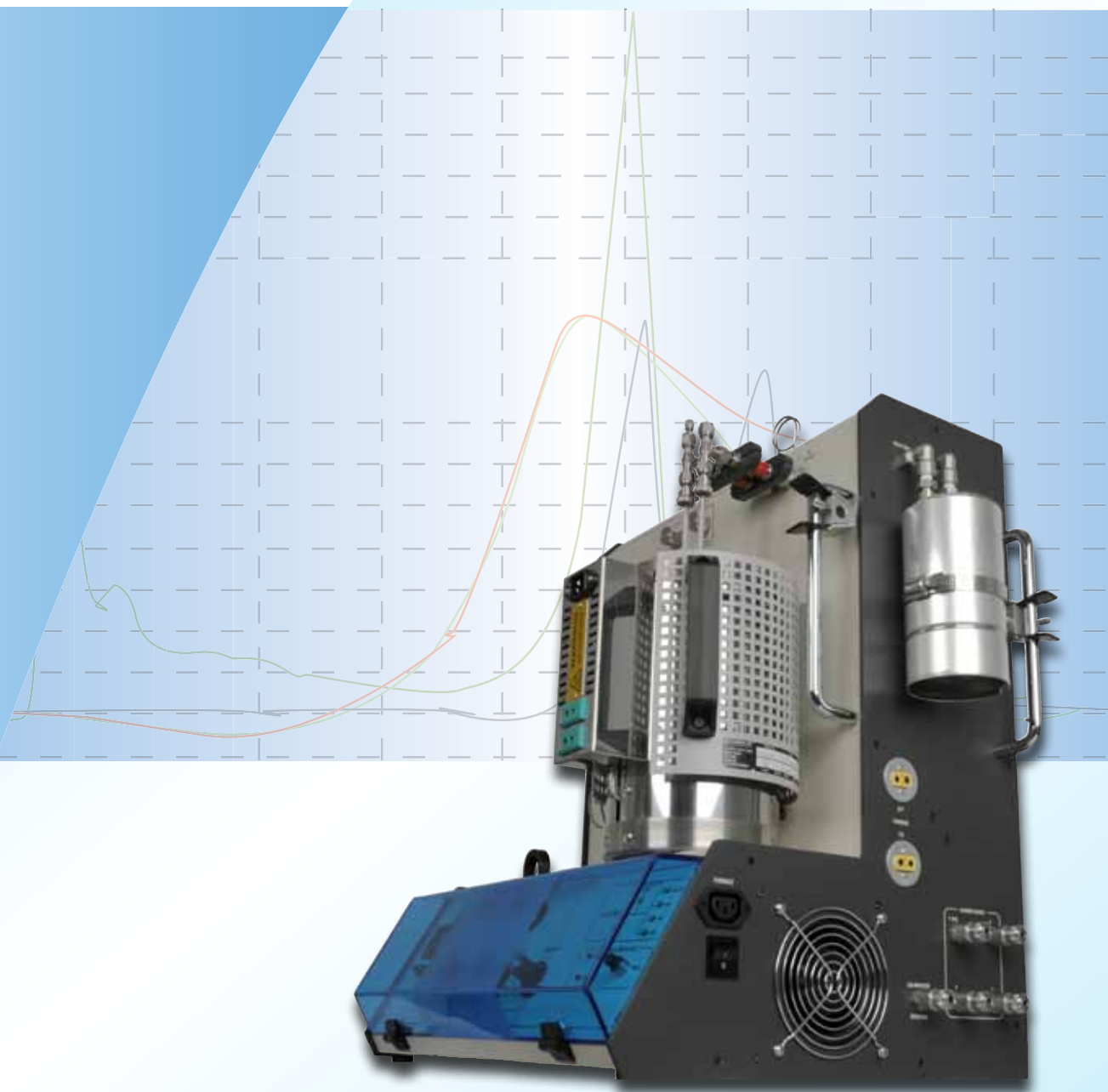


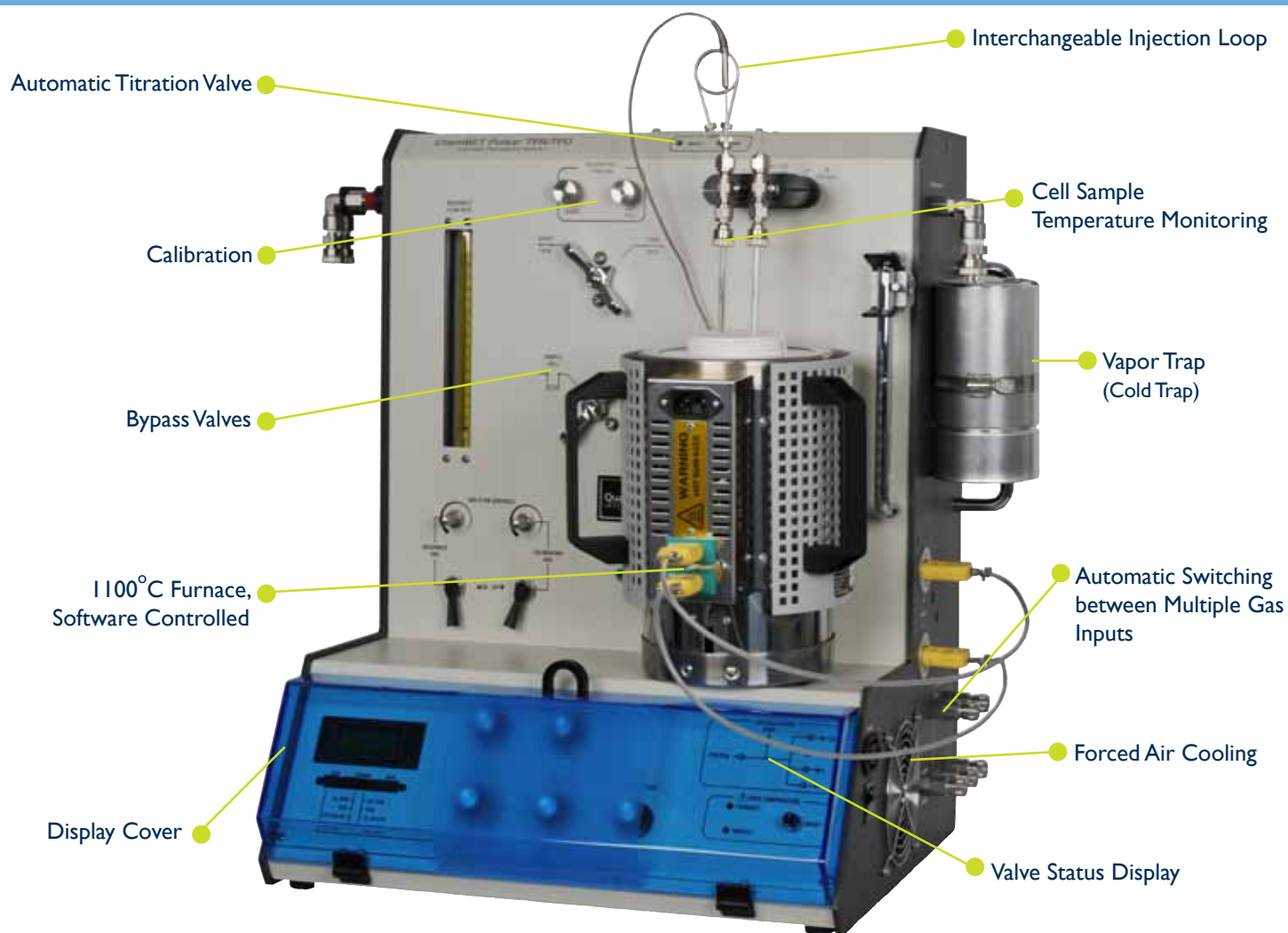


Automatic Chemisorption Analyzer

ChemBET PULSAR™ TPR/TPD



Instrument Features

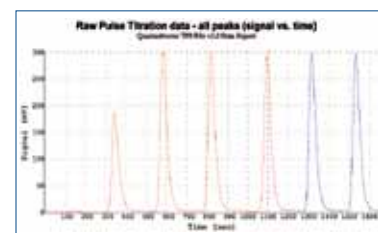


Automation

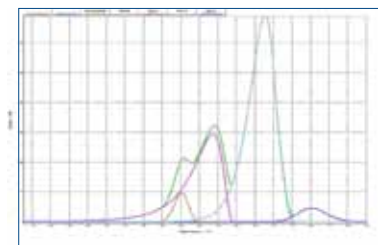
The ChemBET Pulsar TPR/TPD represents the very best in catalyst characterization using automated flow methods of analysis in a remarkably small package.

Fully automated analysis sequences are programmed using the powerful TPRWin PC software. Titrations for metal area and dispersion determination use a new 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 retains the 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.

Options include a Quadrupole Mass Spectrometer, and an external digital gas blender/mass flow controller.



▲ Rapid titration and extreme sensitivity.



▲ Deconvolution of overlapping TPA peaks.

Contact your local representative or call 800.989.2476 (in USA/Canada)

● 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 **2**
Calibration Port
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 (450°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

Graphical and tabular output features deconvolution of overlapping TPX peaks, quantitative peak area determination, and linear and non-linear adjustment of baseline.



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 μ L (others available)

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: 450°C
Mantle Power: 125 W
Furnace, Max Temp: 1100°C
Furnace Power: 575 W

Accessories

Gas Regulator Assembly

Proper Pulsar 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.



Gas Blender (Mass Flow Controller)

Temperature programmed 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 .



● Measurement Capabilities



▲ Industrial Catalysts

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 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

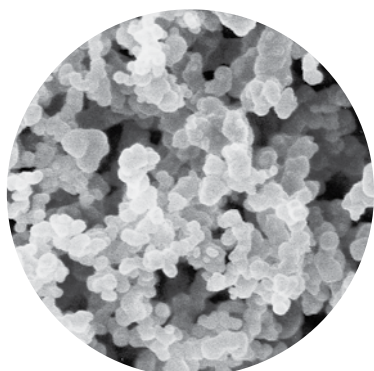
This technique is used to determine the following data:

- (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 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.



▲ Carbons



▲ Zeolites



▲ Supported Metals



Quantachrome Instruments Application Laboratory.

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

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- Pore Size Distribution
- Chemisorption Studies
- Water Sorption Behavior
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- Tapped Density

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07118-P Rev.C 0110