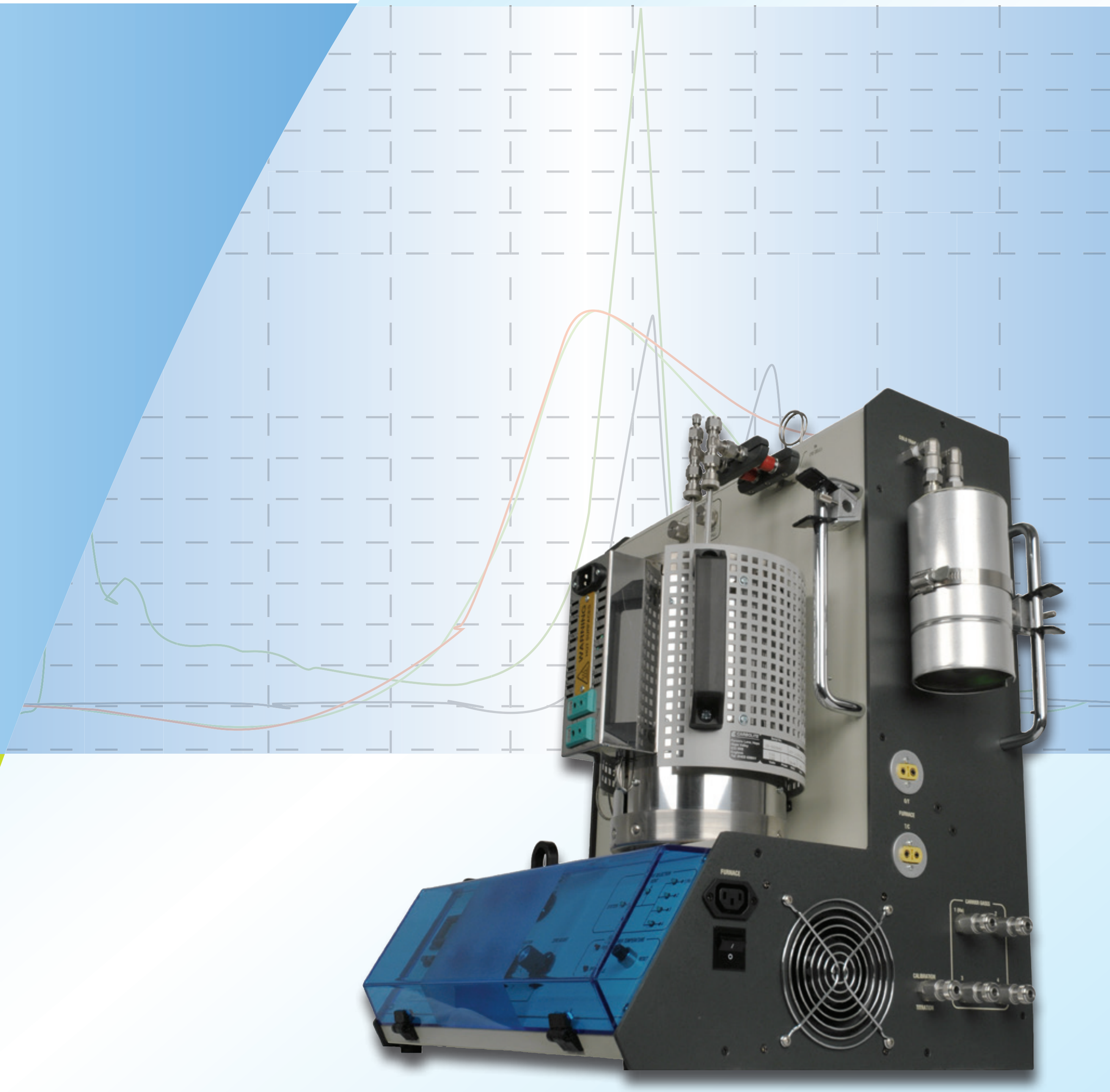
Quantachrome
CORPORATION

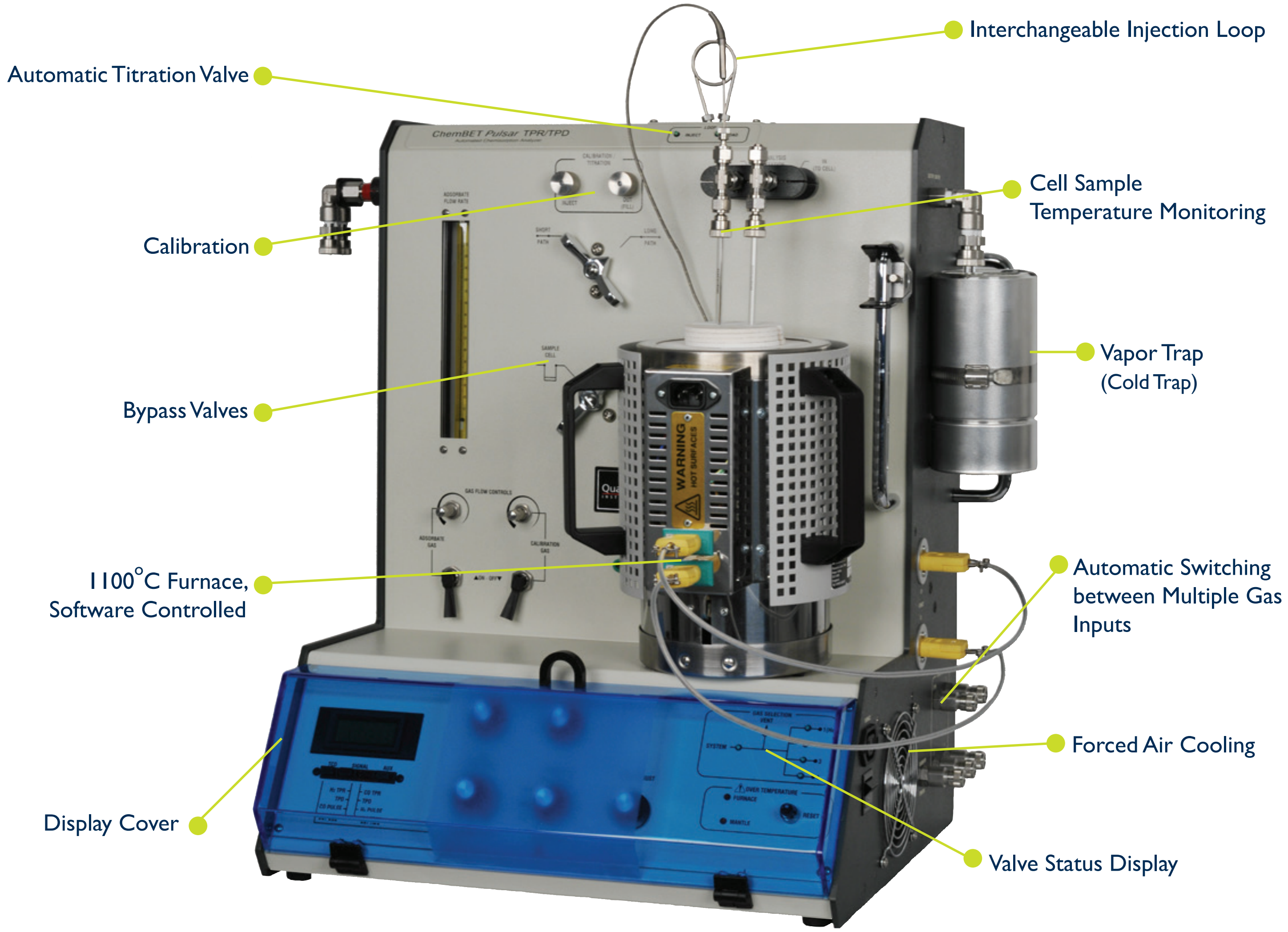
Automatic Chemisorption Analyzer

ChemBET PULSAR™ TPR/TPD



07118-P Rev.E 0313

Instrument Features

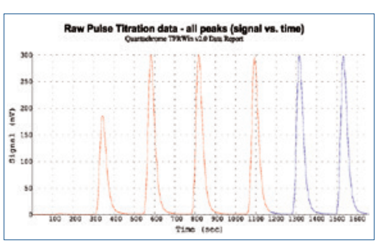


Automation

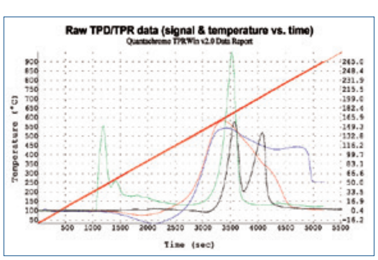
The ChemBET Pulsar TPR/TPD represents the very best in catalyst characterization using automated flow methods of analysis. New to the Quantachrome product line, the Pulsar builds on the reputation of the ChemBET - combining its affordability with the automation of the Autosorb-1C/TCD.

Fully automated analysis sequences are programmed using the new 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 ChemBET's 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, an external digital gas blender/mass flow controller, and a Sub-ambient TPX accessory.



▲ Rapid titration and extreme sensitivity.



▲ TPA overlays for easy comparison.

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

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)

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

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

Accessories

Gas Regulator Assembly

Proper Pulsar functioning is assured when high-quality gas regulators are used. Quantachrome supplies complete assemblies which include 2-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 N_2 (and other inerts including He), H_2, Co, 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.

Sub-Ambient TPX Option

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

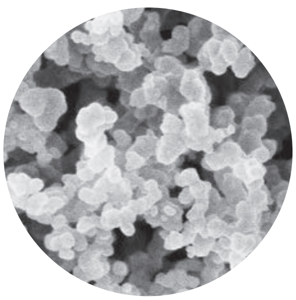
Measurement Capabilities & Applications

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

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.



Carbons, Fuel Cells, etc.

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.

Zeolites
(eg. FCC, Isomerization)

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.

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

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.

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

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Serving Porous Materials and Powder Characterization Needs Since 1968

Quantachrome®

For 45 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' quality management system is certified to be in accordance with ISO9001:2008

Quantachrome INSTRUMENTS

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