AutoChem™ 2950 HP

Chemisorption Analyzer

micromeritics®

The Science and Technology of Small Particles™

Catalyst Characterization in an Extended-Pressure Environment

AutoChem 2950 HP *Chemisorption Analyzer*

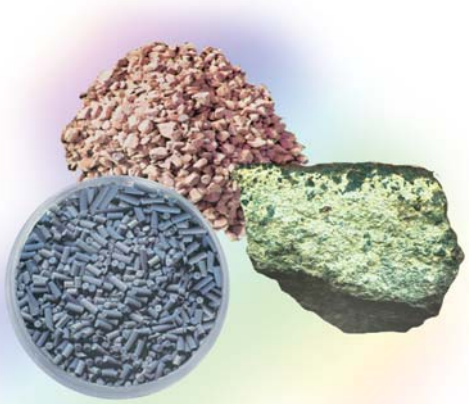
Superior Performance Under Extended Pressure

Micromeritics' AutoChem 2950 HP Chemisorption Analyzer is an automated high-pressure catalyst characterization system that is capable of preparing and analyzing samples at elevated pressures up to 1000 psia and at temperatures from -100 to 1100 °C. The instrument can perform a variety of experiments including pulse chemisorption, BET surface area, temperature-programmed reduction (TPR), desorption (TPD), oxidation (TPO), and reaction analyses. Equipped with many of the features of Micromeritics' AutoChem II 2920, the AutoChem HP is suitable for a variety of applications including fuel cell and hydrogen storage research. This microreactor, combined with a mass spectrometer, can also be used to determine product yields and catalytic activity under commercially viable conditions.

The use of a microreactor in the development process is a cost-effective alternative to pilot plants for many development projects. A microreactor is also an affordable option for small start-up companies that cannot afford to invest in a pilot plant to demonstrate their process.

Benchtop Microreactor Suitable for a Variety of Applications

- HPTPR (High-pressure TPR) to emulate a commercial activation of a supported metal catalyst
- Fuel cell applications that require reforming hydrocarbon feeds to hydrogen



- Hydrogen storage applications using mixed metal hydrides that change hydrogen capacity as temperature changes – the user can vary hydrogen pressure to determine storage capacity as a function of pressure
- Simple chemical reactions where the product mix changes as a function of pressure – the AutoChem 2950 HP functions as a microreactor at typical process temperatures and pressures, allowing the user to collect reaction data at commercial conditions

Standard AutoChem 2950 HP Features

- Allows preparation and analysis of sample at pressures up to 1000 psia with built-in safety checks
- Versatile analysis protocol permits complex sequencing and experimental design
- In situ experiments with virtually unlimited steps
- Records pressure as part of the experiment
- Capable of TPD, TPR, TPO, BET, and pulse chemisorption
- Three mass flow controllers and an electronically controlled pressure regulator provide precise gas control
- Twelve gas inlets – four each for preparation, carrier, and loop gases
- Stainless-steel sample tubes are capable of withstanding temperatures and pressures well above the system's limits
- An interactive reporting system that includes an extremely versatile graphic user interface allowing custom presentation of results

Typical AutoChem 2950 HP Applications

Catalysts
The active surface area and porous structure of catalysts have a great influence on production rates. Limiting the pore size allows only molecules of desired sizes to enter and leave; creating a selective catalyst that will produce primarily the desired product. Chemisorption experiments are valuable for the selection of catalysts for a particular purpose, qualification of catalyst vendors, and the testing of catalyst performance over time to establish when the catalyst should be reactivated or replaced.

Fuel Cells
Platinum-based catalysts including Pt/C, PtRu/C, and PtRuIr/C are often characterized by temperature-programmed reduction to determine the number of oxide phases and pulse chemisorption to calculate:
- Metal surface area
- Metal dispersion
- Average crystallite size

Partial Oxidation
Manganese, cobalt, bismuth, iron, copper, and silver catalysts used for the gas-phase oxidation of ammonia, methane, ethylene, and propylene are characterized using:
- Temperature-programmed oxidation
- Temperature-programmed desorption
- Heat of desorption of oxygen
- Heat of dissociation of oxygen

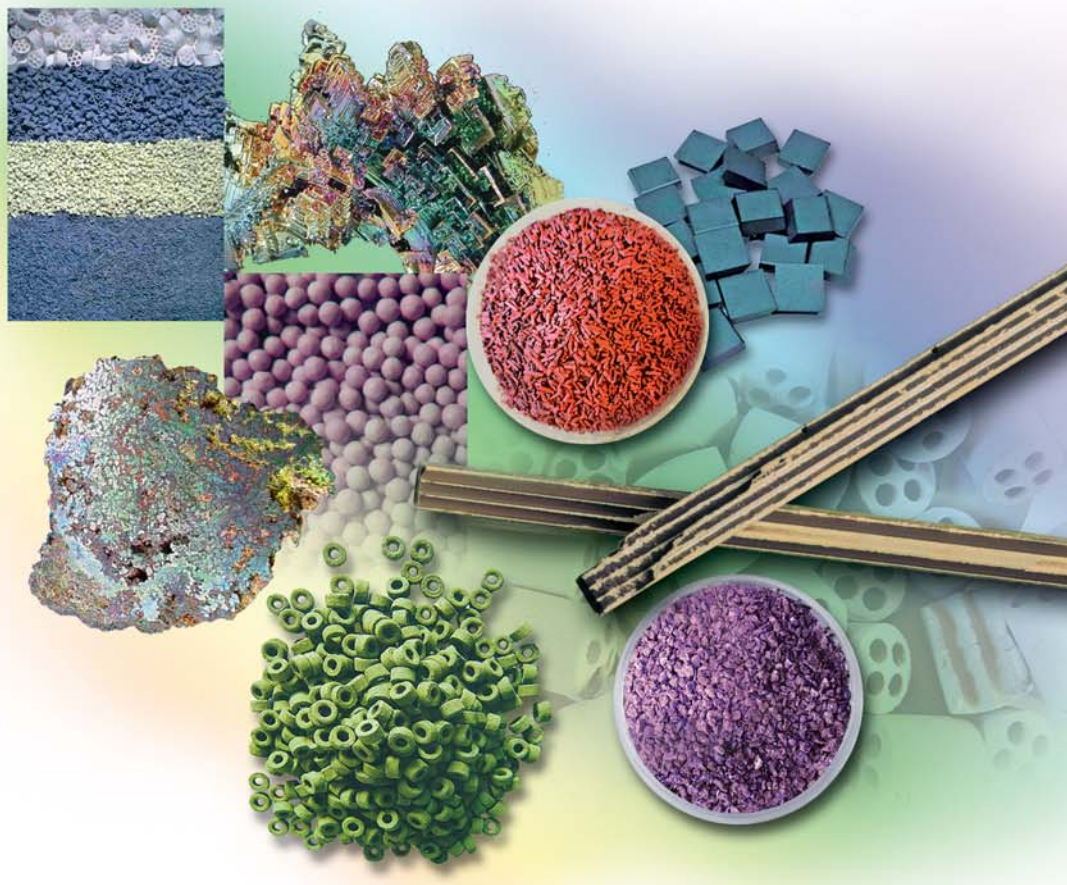
Catalytic Cracking
Acid catalysts such as zeolites are used to convert large hydrocarbons to gasoline and diesel fuel. The characterization of these materials includes:
- Ammonia chemisorption
- Temperature-programmed desorption of ammonia
- Temperature-programmed decomposition of alkyl amines
- Temperature-programmed desorption of aromatic amines

Catalytic Reforming
Catalysts containing platinum, rhenium, tin, etc. on silica, alumina, or silica-alumina are used for the production of hydrogen, aromatics, and olefins. These catalysts are commonly characterized to determine:
- Metal surface area
- Metal dispersion
- Average crystallite size

Isomerization
Catalysts such as small-pore zeolites (mordenite and ZSM-5) containing noble metals (typically platinum) are used to convert linear paraffins to branched paraffins. This increases the octane number and value for blending gasoline and improves the low temperature flow properties of oil. The characterization of these materials includes:
- Temperature-programmed reduction
- Pulse chemisorption

Hydrocracking, Hydrodesulfurization, and Hydrodenitrogenation
Hydrocracking catalysts typically composed of metal sulfides (nickel, tungsten, cobalt, and molybdenum) are used for processing feeds containing polycyclic aromatics that are unsuitable for typical catalytic cracking processes. Hydrodesulfurization and hydrodenitrogenation are used for removing sulfur and nitrogen respectively from petroleum feeds. The characterization of these materials includes:
- Temperature-programmed reduction
- Oxygen pulse chemisorption

Fischer-Tropsch Synthesis
Cobalt, iron, etc. based catalysts are used to convert syngas (carbon monoxide and hydrogen) to hydrocarbons larger than methane. These hydrocarbons are rich in hydrogen and do not contain sulfur or nitrogen. The characterization of these materials includes:
- Temperature-programmed desorption
- Pulse chemisorption



Hardware Versatility

AutoChem 2950 HP Hardware Advantages

The AutoChem HP features stainless-steel construction, fully automated flow control and pressure control, an embedded microprocessor with real-time control, and an intuitive graphical user interface for reactor control. Like the AutoChem 2920, the AutoChem 2950 HP features a temperature-controlled, stainless-steel flow path to provide a stable inert operating environment and reduce the potential for condensation in the flow path. The embedded microprocessor allows the AutoChem series to operate with real-time control; this provides enhanced stability by separating the AutoChem control from a PC or workstation.

- **Twelve gas inlets** (4 each for preparation, carrier, and loop) provide the capability to perform sequential experiments such as TPR/TPO cycle experiments.
- Equipped with **high-precision, independently calibrated mass flow controllers** providing extremely accurate, programmable gas control. The ability to control gas flow with accuracy assures a stable baseline and accurate determination of gas volumes.
- Analysis gas may be introduced to the carrier stream by a **precision automated loop**. A conveniently located septum is also provided through which analysis gas can be injected by means of a calibrated syringe.
- **Thermal conductivity detector (TCD)** is capable of detecting minute differences in the concentration of gases flowing into and out of the sample reactor. Its corrosion-resistant filaments are operated at constant temperature to prevent thermal runaway, which can destroy filaments in other systems.
- A **clamshell furnace** can heat the quartz sample reactor up to 1100 °C. The AutoChem 2950 HP permits any number of ramp rates and sequences to facilitate customized experiments. The included **KwikCool** feature permits cooling the furnace temperature rapidly down to near ambient, reducing analysis time and increasing throughput. With the **CryoCooler option**, analysis temperatures can be ramped over the range of -100 °C to 1100 °C.
- **Four internal temperature-controlled zones** can be heated independently of each other to as much as 150 °C. This prevents condensation in the flow path and allows studies to be performed with vapors.

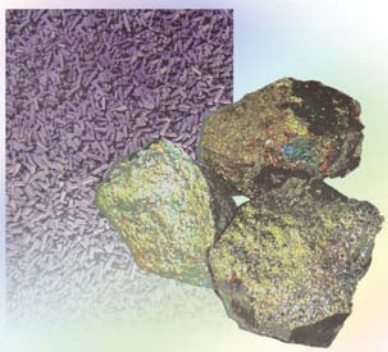
- The extremely **low volume of the internal plumbing** minimizes peak spreading and significantly enhances peak resolution. Furthermore, it reduces the time lag between sample reactions and the corresponding detector response.
- A **cold trap** can remove certain substances before the gas flow reaches the detector. The cold trap can also be used as a delay path for BET surface area experiments.
- **Injection loops** are provided for injecting carefully measured doses of gases for analyses such as pulse chemisorption. The AutoChem HP is shipped with a 1.0-cm^3 loop installed; 0.5- and 5.0-cm^3 loops are also included. If sample files are set up so that a loop is used for introducing gas into the analyzer, the instrument automatically doses the sample as specified in the sample file.
- **Stainless-steel sample tubes** are capable of safe operation up to 1500 psia

Standard 316L Stainless-Steel Reactor
Inquire about other options or metallurgy

An Example of Use

Consider a temperature-programmed reduction (TPR) in which a metal oxide is reacted with hydrogen to form a pure metal, in this case platinum. Argon, which has a very low thermal conductivity, is used as a carrier gas. It is blended in a fixed proportion with hydrogen, an analysis gas with a much higher thermal conductivity. Then the gas mixture flows through the analyzer, through the sample, and past the detector.

When the gas blend begins flowing over the sample, a baseline reading is established by the detector. This baseline is established at a low enough temperature so that no reduction of the sample is occurring. The proportion of gases flowing over the detector is the same as the proportion of gases entering the analyzer, because at the low temperature, there is no interaction.



The temperature is then gradually increased and when a critical temperature is reached, hydrogen atoms in the gas flow react with the sample, forming H_2O molecules which are removed from the gas stream using a cold trap. As a result, the amount of hydrogen in the argon/hydrogen gas blend decreases and the proportion between the two gases shifts in the direction of argon, as does the mixture's thermal conductivity.

Since argon has a lower thermal conductivity than hydrogen, the mixture's thermal conductivity consequently decreases. The flowing gas removes heat from the filament more slowly, requiring less electricity to maintain a constant filament temperature. The instrument records the electrical demands as it changes (this is called the detector signal). The detector signal is recorded continuously over a range of temperatures. The resulting signals may present either positive or negative peaks.

This example illustrates the fundamental concept upon which the analyzer operates. Of course, the various types of analyses the AutoChem can perform result in different types of traces. For example, a pulse chemisorption analysis results in a series of peaks that gradually increases in size as the sample is dosed with separate but equal increments of gas. Initially, the gas uptake by the sample results in smaller peaks. But when all the active sites are saturated, no more gas can be taken up and the peaks become equal.

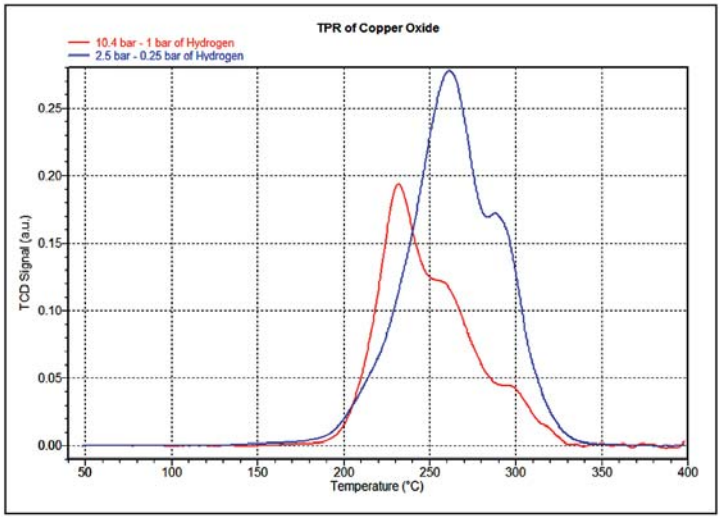
Software and Reporting Versatility

AutoChem 2950 HP Software Features

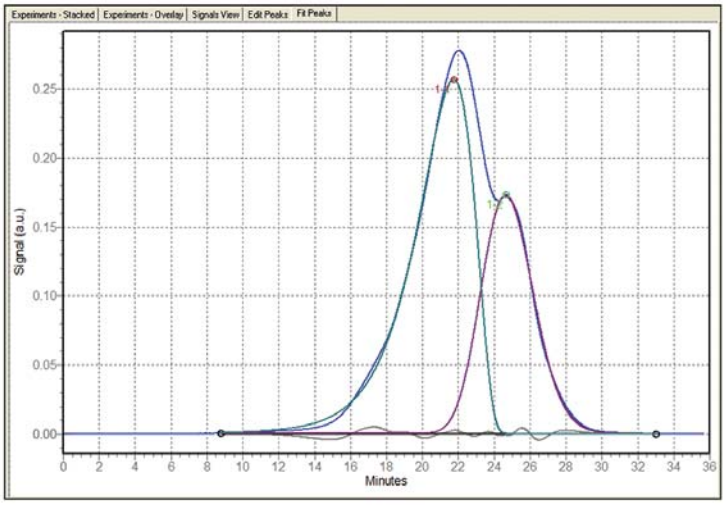
The AutoChem 2950 HP is controlled by powerful Windows®-based software. It provides all the convenient features you are accustomed to when using Windows-based programs. Point-and-click operations, pull-down menus, access to multiple printers and network drives, multitasking capability, and much more are featured in the AutoChem 2950 HP user interface. In addition, the familiar Windows format reduces the time required for training new operators, resulting in fast start-up and increased productivity.

Additional Capabilities

- An Instrument Schematic screen displays the instrument's current operating status, including the availability of analysis and pretreatment gases and vapors, direction of the gas flow, TCD reading, and allows the operator to assume manual control of the instrument if desired.
- Set up Analysis Protocol Sequencing from any number of preprogrammed experiments or create a customized sequence. The user can easily select the pretreatment and analysis task, and specify criteria such as temperature ramp rates, gas flow rates, and data measurement intervals in the desired sequence. Modifications may be made to the analysis protocol at any time, even during analysis.
- Fully integrated, interactive peak editor package enables the user to evaluate results quickly and easily, edit peaks, and produce reports that reflect specific needs. Adjusting peak boundaries is a matter of simply pointing and clicking. The peak editor can also be used to deconvolute overlapping peaks. The peak deconvolution is a simple-to-use option on the peak space editor that allows the user to maximize the information.

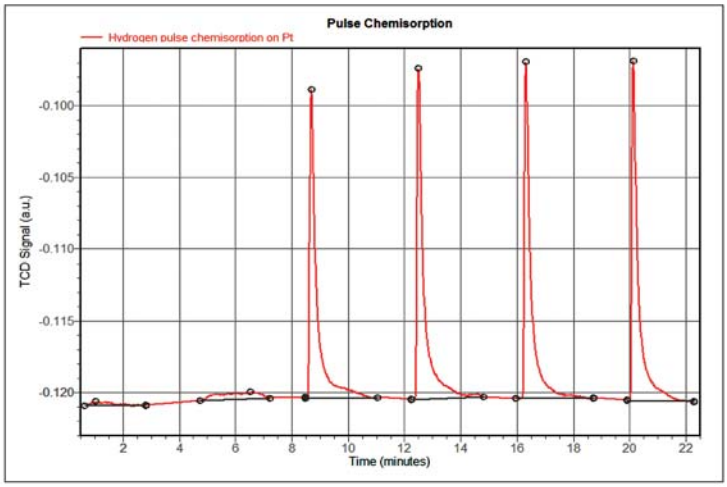


The AutoChem HP features user-programmable, back-pressure control. This allows the user to evaluate materials, user pressures, and temperatures that are similar to real-world operating conditions. The TPR of copper oxide demonstrates the change in reduction kinetics as the partial pressure of hydrogen is increased.

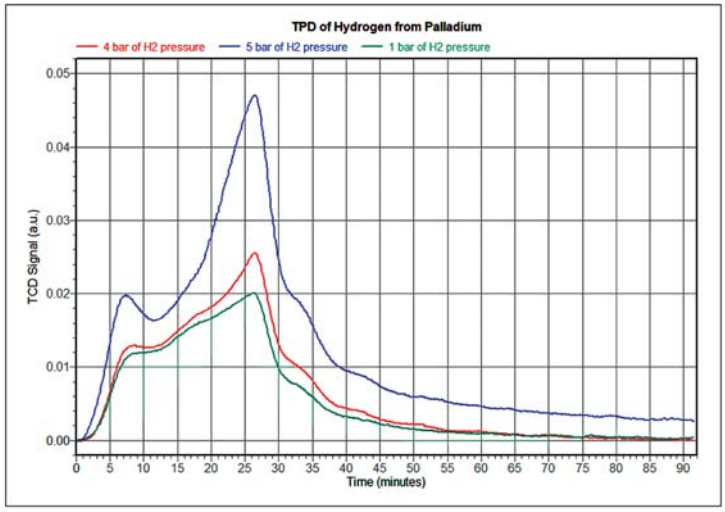


The AutoChem HP includes easy-to-use peak fitting. This allows the user to rapidly deconvolute TPR, TPD, and TPO data to obtain a better understanding of the material being tested.

- Numerous plots can be overlaid for easy comparison of different samples or for comparison of different data reduction techniques applied to the same sample. The operator can also plot two externally derived signals against time or sample temperature (e.g., mass spectrometer and gas chromatograph).
- Integration of mass spectrometer data files, which allows the manipulation of the TCD and mass spectrometer simultaneously.
- One computer can control two Micromeritics AutoChem HP analyzers making efficient use of valuable lab space.



Traditional methods like pulse chemisorption are a standard feature of the AutoChem 2950 HP. Pulse chemisorption calculations provide a rapid and accurate method for assessing the active monolayer of a catalyst.



Varying the hydrogen pressure allows additional hydrogen to be stored in palladium as can be demonstrated using the temperature-programmed desorption of hydrogen from palladium.

Experiment 1: H2 Pulse Chemisorption

Analysis Type: Pulse Chemisorption
Calibration: None
Measured Flow Rate: 50.13 cm³ STP/min
Signal Offset: 0.00000
Signal Inverted: No

Peak Table

Peak Number	Temperature at Maximum (°C)	Quantity Adsorbed (cm³/g STP)	Cumulative Quantity (cm³/g STP)
1	36.3	0.04583	0.04583
2	35.4	0.04138	0.08722
3	35.0	0.00147	0.08869
4	34.2	0.00000	0.08869
5	33.6	0.00095	0.08964
6	33.1	0.00000	0.08964

Pulse Chemisorption Analysis Summary

Element	Percent of Sample Mass (%)	Atomic Weight	Stoichiometry Factor	Atomic Cross-Sectional Area (nm²)	Density (g/cm³)
platinum	0.5000	195.090	2.000	0.0800	21.450

Active Loop Volume at 110.8 °C: 0.03595 cm³ STP
Cumulative Quantity: 0.08964 cm³/g STP
Metal Dispersion: 31.2077%
Metallic Surface Area: 0.3854 m²/g sample
Metallic Surface Area: 77.0779 m²/g metal
Active Particle Diameter: 3.6291 nm

The AutoChem HP features a full reporting system that includes calculations for active surface characterization. The pulse chemisorption report includes dispersion, active surface area, and crystallite size. Additional software features include a library of parameters for active metals and the capability to determine Arrhenius parameters based upon first-order kinetics.

To request a quote or additional product information, visit Micromeritics' web site at www.micromeritics.com or contact your local Micromeritics sales representative.

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