AutoChem™ II 2920

The Catalyst Characterization Laboratory

micromeritics®

AUTOCHEM II 2920

A Catalyst Characterization Laboratory in a Single Analytical Instrument

Micromeritics' AutoChem II 2920 Chemisorption Analyzer is a fully automated instrument capable of conducting a comprehensive array of highly precise chemical adsorption and temperature- programmed reaction studies. The instrument enables the researcher to obtain valuable information about the physical properties of catalysts, catalyst support, and a variety of other materials. Researchers can investigate active metal surface area, surface acidity, distribution and strength of active sites, BET surface area, and more. The AutoChem II performs pulse chemisorption, temperature-programmed reduction (TPR), desorption (TPD), oxidation (TPO), and reaction analyses. Multiple experiments can be run using the same sample.

Wide Variety of Features and Benefits

- Four internal temperature-controlled zones can be heated independently up to 150 °C. This prevents condensation in the flow path and allows studies to be performed with vapors.
- Low internal plumbing volume assures high resolution, fast detector response, and reduces error when calculating gas volumes.
- Highly sensitive linear thermal conductivity detector (TCD) assures the calibration volume remains constant over the full range of peak amplitudes so the area under the peak is directly proportional to the volume of gas reacted.
- Four high-precision mass flow controllers provide extremely accurate, program- mable gas control. This assures a stable baseline and accurate determination of gas volumes.
- Corrosion-resistant detector filaments are compatible with most destructive gases and reduce the likelihood of fila- ment oxidation.
- Clamshell furnace can heat the quartz sample reactor to 1100 °C. Any number of ramp rates and sequences facilitate customized experiments. The KwikCool feature cools the furnace temperature rapidly down to near ambient, reducing analysis time and increasing throughput.
- Four gas inlets each for the preparation, carrier, and loop gases permit four-gas sequential experiments, such as TPR/TPO cycles.
- Mass spectrometer port and software integration allows virtually simultaneous detection on both the thermal conductivity detector and mass spectrometer.
- Optional Vapor Generator permits analysis using vaporized liquids in an inert carrier stream.
- Optional CryoCooler enables the start of an analysis at subambient temperature.

Typical AutoChem II 2920 Applications

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.

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. 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 not suitable for typical catalytic cracking processes. 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. The characterization of these materials includes:

- Temperature-programmed desorption
- Pulse chemisorption

Innovative Design: AutoChem II 2920 Hardware Advantages

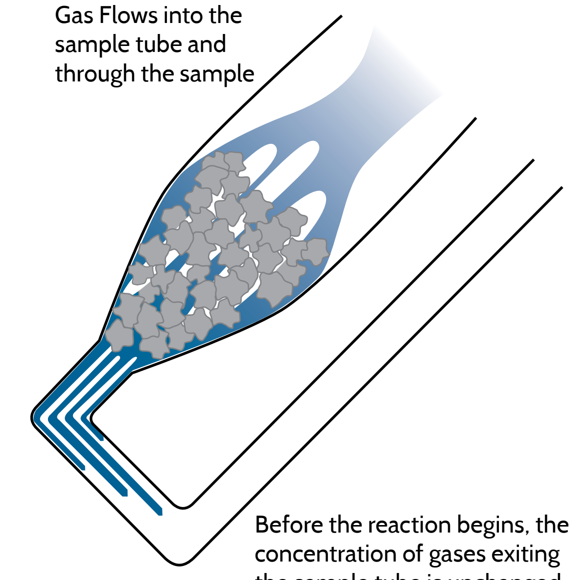
The AutoChem II features stainless-steel construction, fully automated flow and pressure control, an embedded microprocessor with real-time control, and an intuitive graphical user interface for reactor control. A temperature-controlled, stainless-steel flow path provides an inert and stable operating environment, and reduces the potential for condensation in the flow path.

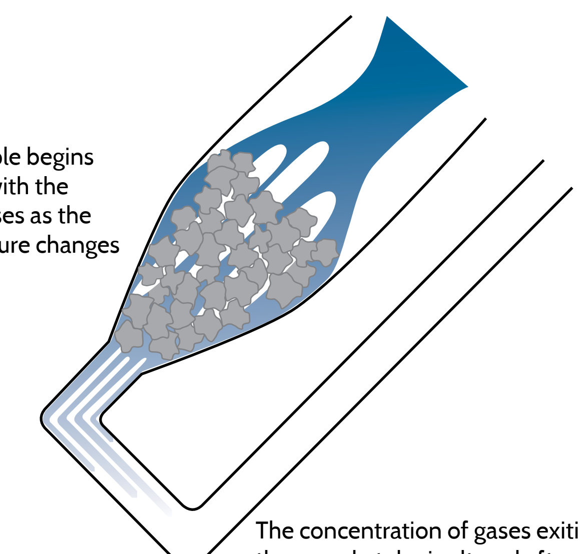
- Twelve gas inlets (4 each for prep, carrier, and analysis) provide the capability to perform sequential experiments such as TPR/ TPO cycle experiments.
- Equipped with four high-precision, independently calibrated mass flow controllers providing extremely accurate, programmable gas control. The ability 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 calibrated, 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 II 2920 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 the sample reactions and the corresponding detector response.

The AutoChem II Technique

During the TPR, a metal oxide reacts with hydrogen to form a pure metal. This reaction is referred to as a reduction; for example, TPR of a catalyst containing platinum. Argon, which has a very low relative thermal conductivity, is used as a component in the carrier gas. It is blended in a fixed proportion with hydrogen, the reducing gas with a much higher thermal conductivity. Then the gas mixture flows through the analyzer, through the sample, and past the detector. When the hydrogen and argon 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 occurs. The baseline level indicated by the detector is that of the thermal conductivity of the two gases in their fixed proportion. In other words, 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 with the sample.

The temperature is then raised and, when a critical temperature is reached, hydrogen atoms in the gas flow react with the sample, forming H_2O molecules. The H_2O molecules are removed from the gas stream using a cold trap. As a result, the amount of hydrogen in the argon/hydrogen gas blend inside the analyzer 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 demand as it changes (this is called the detector signal). The detector signal is recorded continuously over a range of temperatures. When these readings are graphed, the data form one or more peaks. Peaks can be positive or negative.

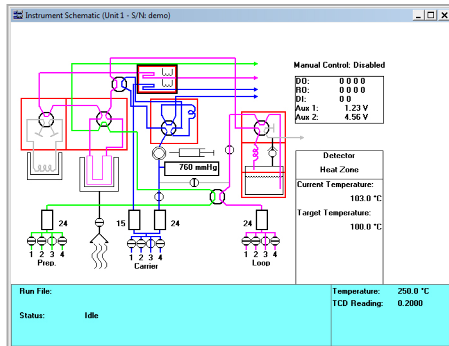


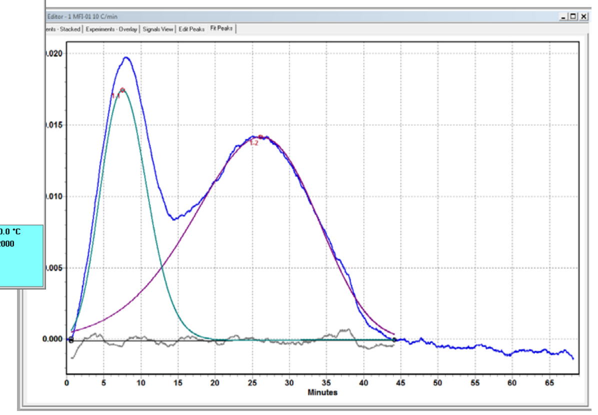


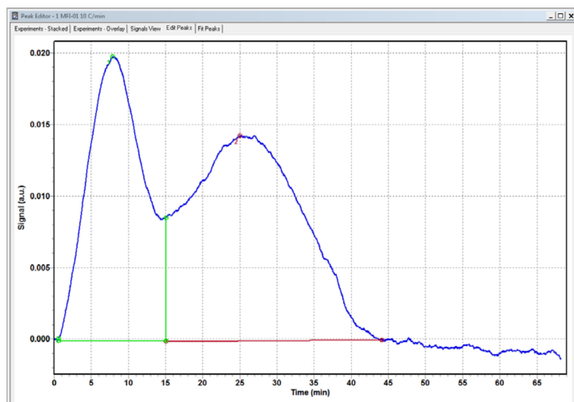
Software Features and Data Reduction/Reporting

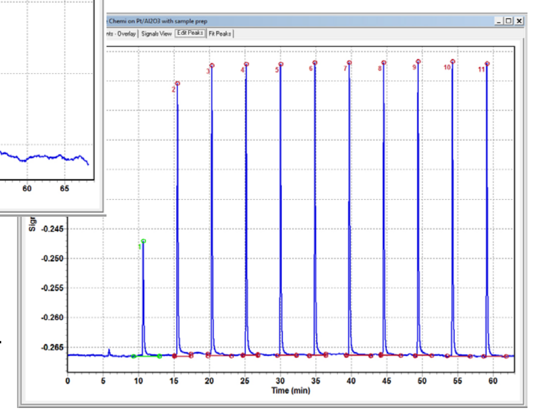
The easy-to-use AutoChem II software utilizes a Windows® interface that includes Wizards and applications to help plan, launch, and control the analysis. You can collect, organize, archive and reduce raw data, and store standardized sample information and analysis conditions for easy access during later applications.

- Set up analysis protocol sequencing from any number of preprogrammed experi- ments or create a customized sequence. The user can easily select the pretreat- ment and analysis task and specify criteria such as temperature ramp rates, gas flow rates, and data measurement intervals
- The 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, and TCD reading.
- Numerous plots can be overlaid for easy comparison of different samples or for comparison of different data reduction techniques





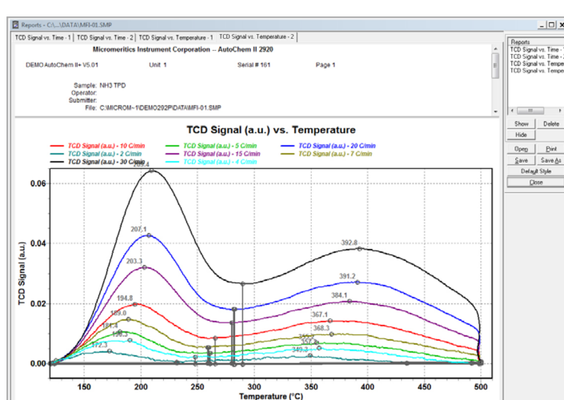


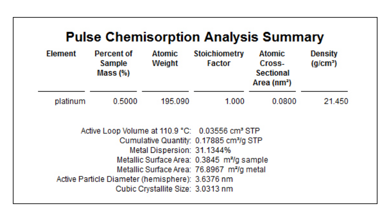


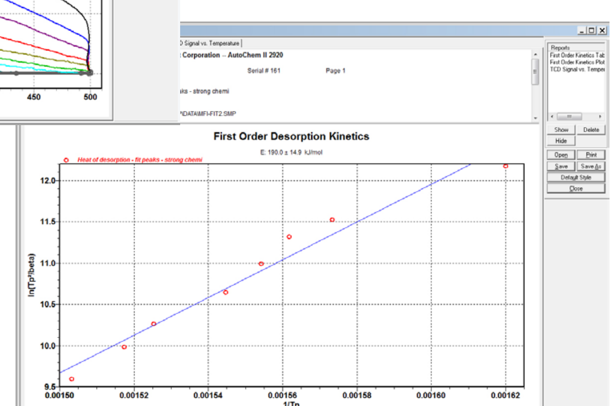
A full-featuer peak editor and integration package is included with the AutoChem II. This easy-to-use package provides a rapid method for evaluating common temperature-programmed and dynamic adsorption data.

Powerful Peak Editor

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 AutoChem II Also Includes

- Ten user-configurable graphical reports
- BET, Langmuir, and total pore volume
- Pulse chemisorption, % dispersion, metal surface area, and crystallite size
- First-order kinetics, heat of desorption, and activation energy
- Integration of mass spectrometer data fil

Specifications

Electrical	
Voltage	85 to 265 VAC
Frequency	50/60 Hz
Power	1100 VA, operating, max
Control Module: Minimum Requirements	
Processor	Pentium 333 MHz or equivalent
Operating Systems	64 bit, Windows® 7 Professional or higher operating system
Hard Disk Space	1-gigabyte hard drive
Graphics Card	800 x 600 super VGA
Temperature System	
Range	-100 °C to 1100 °C with Cryo-Cooler option Ambient to 1100 °C without Cryo-Cooler option
Selection	Digitally set, 1 °C increments
Ramp Rates	Up to 50°C per minute from 120 to 500 °C range Up to 30 °C per minute from 500 to 750 °C range Up to 10°C per minute from 750 to 1100 °C range
Options	
Vapor Generator, CryoCooler	
Gases	
Loop (Analysis)	H_2, CO, O_2, N2O, NH_3 vapors such as pyridine, water, etc.
Carrier	He, Ar, and other gases
Preparation	H_2, O_2, He, Ar, and others
Gas Flow Rate	
All Mass Flow Controllers (MFCs)	
Manual Control	0 to 100 mL/minute*
Automatic Analysis	10 to 75 mL/minute
Gas Delivery	
Inlet Ports	(12), 4 each for preparation gas, carrier gas and loop (analysis) gas
Temp Control	Internal gas lines and valves heated up to 150 °C
Sample Tube	
Type	Fused quartz Flow-Thru sample tubes, for use up to 1100 °C, accepts powders and pellets up to 9 mm in diameter
Physical	
Height	62 cm (24.5 in.)
Width	66 cm (26 in.)
Depth	58 cm (22.75 in.)
Weight	60 kg (130 lbs)
Environment	
Temperature	15°C to 35°C operating, 0°C to 50°C non-operating
Humidity	20 to 80% relative,non-condensing

**Rate for Hydrogen; other gases have a different range.*
**Due to continuous improvements, specifications are subject to change without notice.*

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To request a quote or additional product information, visit

micromeritics.com

Contact your local Micromeritics sales representative
or our Customer Service Department at

770-662-3636

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