ChemiSorb™ Series

Chemisorption Analyzers

micromeritics®

The Science and Technology of Small Particles™

Economical Chemisorption Analyses

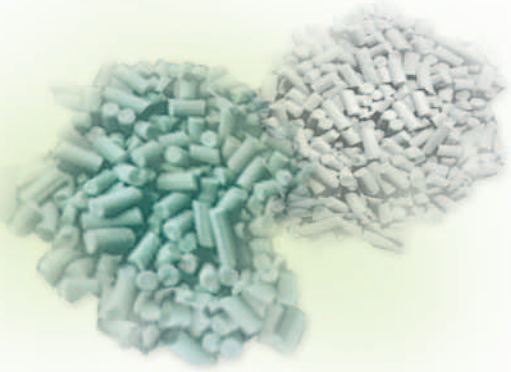
ChemiSorb Series Chemisorption Analyzers

The Versatility of the Chemisorption Technique for Catalyst Characterization

Knowledge of surface chemistry and surface structure is essential in the design, production, and application of a catalytic material. The Micromeritics ChemiSorb 2720 and 2750 quickly and accurately measure the quantity of adsorbed or desorbed molecules allowing determination of these characteristics. The ChemiSorb 2720 makes chemisorption and physisorption analyses affordable for applications in which owning an instrument had once been impractical. Furthermore, if analytical needs grow, the basic system can be upgraded to provide more capability. The ChemiSorb 2750 allows the same analyses as the ChemiSorb 2720 with the added advantages of higher precision, faster throughput, and more convenience in accommodating a variety of experiments.

Versatility in an Inexpensive Chemisorption System – The ChemiSorb 2720

- Dual ports, one for analysis and one for sample preparation.
- Built-in sample cooling fan, four carrier gas inlets, and one prep gas inlet.
- The basic instrument can measure percent dispersion, active metal area, crystallite size, and quantify acid and base sites using pulse chemisorption. Physisorption tests include BET and Langmuir surface area, and total pore volume.
- An optional access fitting allows the ChemiSorb to utilize a mass spectrometer or other external detector for identification of desorbed species or reaction products.



ChemiSorb 2720

ChemiSorb 2750

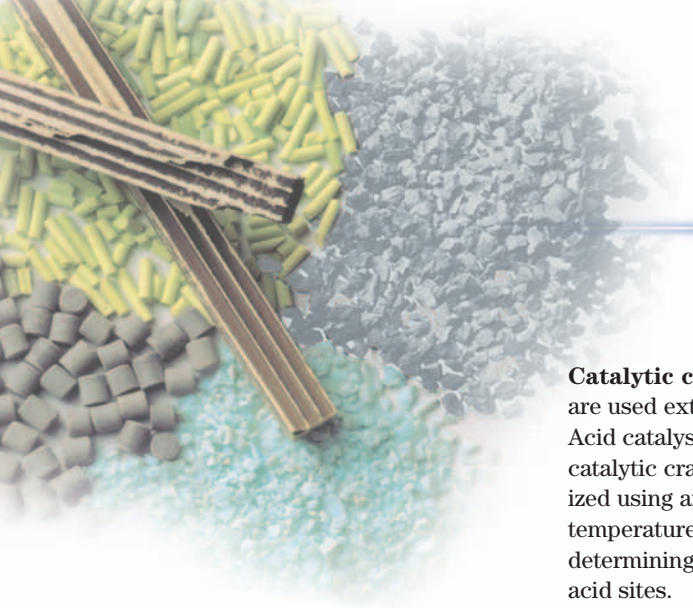
Higher Precision and Versatility – The ChemiSorb 2750

- Dual-function sample ports can be used as either a sample port or degas port. Once the sample has been degassed, reduced, or otherwise prepared, the port is switched to "test" mode for analysis. Possible contamination of a prepared sample by exposure to the atmosphere is eliminated.
- Higher precision, repeatability, and reproducibility are provided by the incorporation of an injector loop valve in addition to the injection septum. The loops are easily exchanged to provide different injection volumes. Electrically activated inlet valves allow the use of gases containing H_2, CO, O_2, N_2O, NH_3, liquid vapor sources, or other adsorptives.
- Three built-in prep gas inlets and four carrier gas inlets allow for a variety of experiments without having to disconnect, reconnect, and purge gas lines.

Added Capability – Optional ChemiSoft TPx System

- Optional ChemiSoft TPx System (temperature-programmed controller and software) expands the capabilities of the ChemiSorb 2720 and 2750 to include temperature-programmed reactions, data archiving, and advanced data reduction and reporting options.
- Expanded physisorption capability includes multipoint BET surface area.

TPx Controller and Furnace



Typical ChemiSorb Applications

Catalysts – The active surface area and pore structure of catalysts have great influence on reaction rates and yield of product. 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 a catalyst's 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 may be characterized by temperature-programmed reduction to determine the number of oxide phases or by pulse chemisorption to characterize the metal surface area, metal dispersion, and average crystallite size.

Partial oxidation – Manganese, cobalt, bismuth, iron, copper, and silver oxides are often used for the gas-phase oxidation of ammonia, methane, ethylene, propylene, etc. Temperature-programmed oxidation and temperature-programmed desorption may be used to measure the heat of desorption of oxygen from these catalysts and the heat of dissociation of oxygen from the metal oxide.

Catalytic cracking – Catalytic processes are used extensively for refining petroleum. Acid catalysts such as zeolites are used for catalytic cracking and are often characterized using ammonia chemisorption and temperature-programmed desorption for determining the number and strength of the acid sites.

Catalytic-reforming catalysts containing platinum, rhenium, tin on silica, alumina, or silica-alumina are used for the production of hydrogen, aromatics, and olefins. These catalysts are often characterized using pulse chemisorption techniques to determine the number of active sites, the percent metal dispersion, and 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 and thus increase the octane number and value for blending gasoline. Temperature-programmed reduction and pulse chemisorption are often combined to characterize these catalysts.

Hydrocracking, hydrodesulfurization, and hydrodenitrogenation catalysts are typically composed of metal sulfides (nickel, tungsten, cobalt, and molybdenum). Hydrocracking catalysts are used for processing feeds containing polycyclic aromatics that are unsuitable for typical catalytic cracking processes. The hydrocracking process is used for upgrading these low-value products to gasoline and diesel fuel. Hydrodesulfurization and hydrodenitrogenation are used for removing sulfur and nitrogen, respectively, from petroleum feeds. Both sulfur and nitrogen are catalytic poisons and also are the source of pollution (acid rain) if they are not removed from gasoline and diesel fuel. Temperature-programmed reduction and oxygen chemisorption are used to characterize the oxide phases and active surface area of these materials.

Fischer-Tropsch synthesis uses cobalt and iron-based catalysts to convert syngas (carbon monoxide and hydrogen) to hydrocarbons larger than methane. The Fischer-Tropsch processes are of great importance as they provide hydrocarbons that are rich in hydrogen and do not contain sulfur or nitrogen. These hydrocarbons are a potential liquid fuel that is easily transported and distributed, and can then be reformed to hydrogen to supply fuel cells. These catalysts are often characterized by pulse chemisorption and temperature-programmed desorption to determine the metal surface area and the average size of the metal crystallites.

Theory and Design

The Analytical Technique

The ChemiSorb 2720 and 2750 both utilize the dynamic (flowing gas) technique of analysis. The quantity of gas adsorbed from the gas stream by the sample is monitored by a downstream thermal conductivity detector (TCD). The temperature and pressure at which adsorption/desorption occurs is either known or monitored. The instruments can be used to study physical or chemical adsorption. Preparation usually is accomplished by flowing either an inert or chemically active gas over the sample. After preparation, another gas is selected for analysis. Prep and carrier gases typically used to allow both physical and chemical adsorption experiments are He, Ar, N_2, He/N_2 mixtures, H_2, and O_2, some serving as both prep and carrier.

Chemical Adsorption

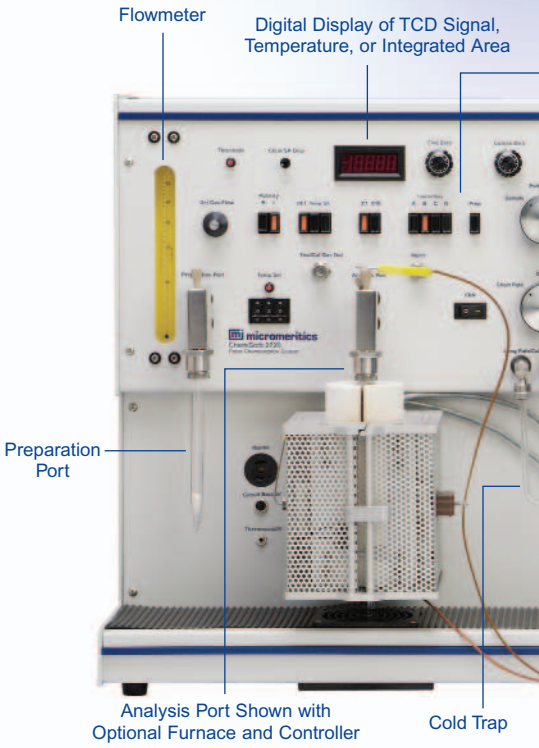
Any of a number of reactive gases such as anhydrous NH_3, CO_2, CO, H_2, N_2O, O_2, and H_2S can be used to react with the active surface. A series of injections of a known quantity of reactive gas is injected into an inert gas stream that passes through the bed of catalysts. Downstream from the reactor is a detector, which determines the quantity of reactive gas that is removed from each injection. Chemisorption tests ideally are made with the sample at a temperature such that only chemisorption occurs. The active surface of the sample is saturated when the detector indicates that the total quantity of subsequent injections passes through the sample bed without any loss. The sum of the injected quantity minus the quantity of gas that passed without adsorption equals the quantity adsorbed.

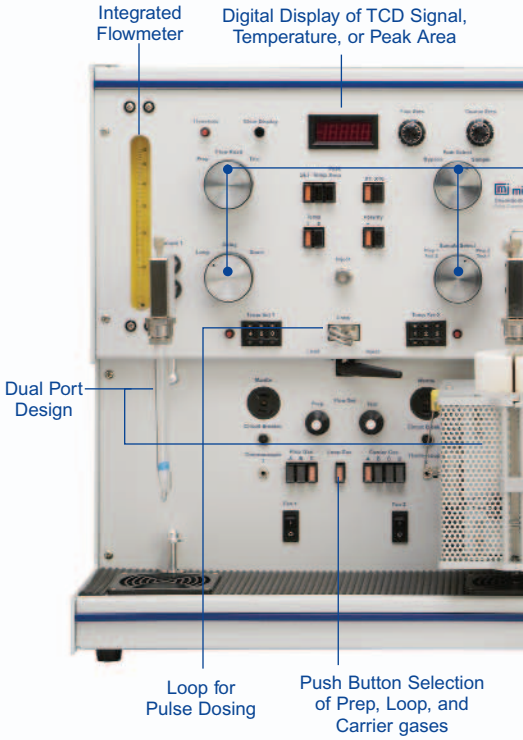
Unlike physical adsorption, the injected gas chemically adsorbs only on the active surface and not on the support. Thus, the number of gas molecules required to cover the active surface area, once determined, leads directly to the active surface area. Applying the stoichiometry factor for the gas/metal reaction yields the number of accessible atoms of active metal. Furthermore, using the total quantity of active metal per gram of catalyst material (determined from the manufacturing formula) leads to the determination of the percent dispersion of active metal. Using the information gathered plus the density of the metal, the size of the metal crystallite can be estimated if it is assumed that these particles have uniform geometry of known volume-to-area ratio.

Physical Adsorption

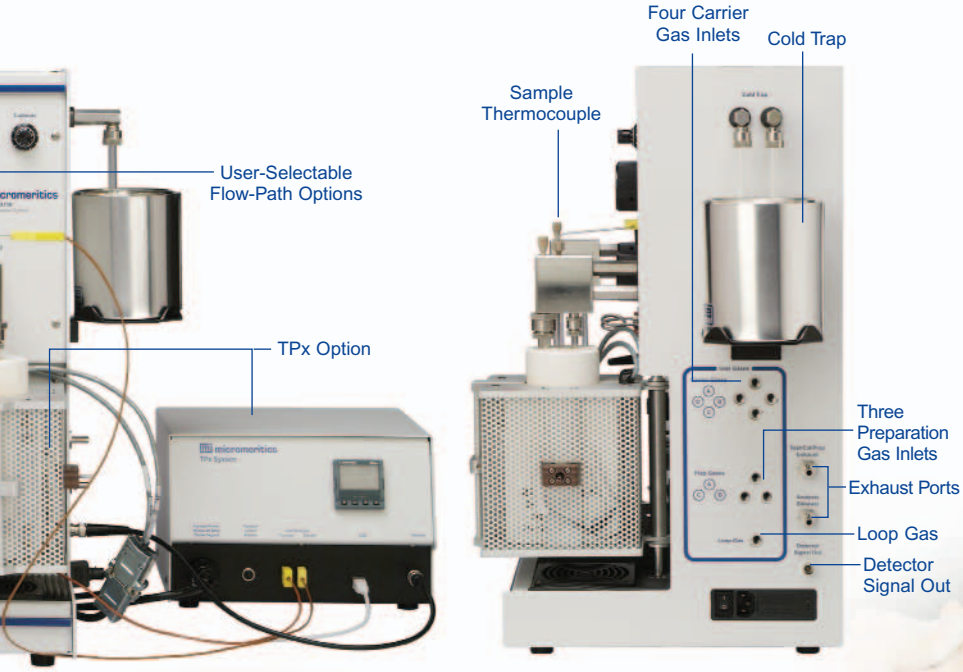
The surface area of granulated and powdered solids or porous materials is measured by determining the quantity of a gas required to form a monomolecular layer on a sample. Physical adsorption tests typically are performed at or near the boiling point of the adsorbate gas; N_2 being most common with a liquid N_2 bath being used to maintain the analysis temperature. Under these conditions, a nitrogen and helium mixture of 30 volume percent nitrogen achieves the partial pressure condition most favorable for the formation of a monolayer of adsorbed nitrogen at atmospheric pressure.

Under such specific conditions, the area covered by each gas molecule is known within relatively narrow limits. The area of the sample is thus calculable directly from the number of adsorbed molecules, which is derived from the gas quantity at the prescribed conditions, and the area occupied by each. Additionally, atmospheric pressure and ice water temperature may establish appropriate conditions for an n-butane and helium mixture. Other gases at other conditions are also usable.





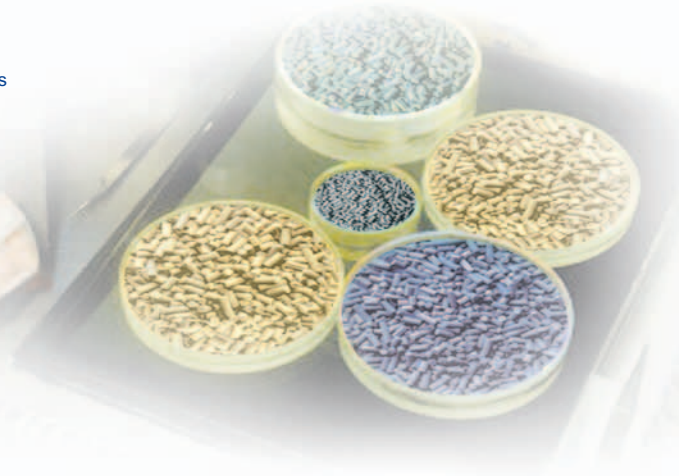
ChemiSorb 2720



ChemiSorb 2750

ChemiSorb Features	2720	2750
Analysis Ports	1	2†
Preparation Port	1	†
Injection Septum	Y	Y
Injection Loop		Y
Sample Reactor	Quartz	Quartz
Gas Inlets		
Carrier	4	4
Preparation	1	3
Loop		1
Temperature Control		
Integrated	2‡	2
Max Temperature	400 °C	400 °C
With TPx option	1100 °C	1100 °C
Fan-assisted Cooling	1	2
Standard Analyses		
Pulse Chemisorption	Y	Y
Physisorption	Y	Y
ChemiSoft TPx Analyses		
TPR	Y	Y
TPD	Y	Y
TPO	Y	Y
Pulse Chemisorption	Y	Y
Physisorption	Y	Y
Loop Calibration		Y
ChemiSoft TPx Reports		
% Metal Dispersion	Y	Y
Metal Surface Area	Y	Y
Average Crystallite Size	Y	Y
First Order Kinetics	Y	Y
Single-Point Surface Area	Y	Y
BET Multipoint Surface Area	Y	Y
Langmuir Surface Area	Y	Y
Total Pore Volume	Y	Y

†Dual function Analysis/Preparation
‡One dedicated controller for preparation port and one dedicated controller for the analysis port



Temperature-Programmed Chemisorption Option

ChemiSoft TPx System

When the optional programmable furnace system and accompanying **ChemiSoft TPx software** are added to the 2720 or the 2750, another category of chemical adsorption testing can be performed ——Temperature-programmed reactions reduction (TPR), oxidation (TPO), and desorption (TPD).

Temperature control is provided by a furnace that operates from ambient (20 °C) to 1100 °C, and is able to produce temperature ramps of up to 50 °C/min within the 20 to 500 °C range, 30 °C/min within the 500 to 750 °C range, and up to 10 °C/min in the 750 to 1100 °C range. The furnace controller can be programmed to provide multiple ramps and soak times.

Temperature-programmed Chemisorption

Temperature-programmed chemisorption provides information about adsorption strength when a catalyst is at working condition or at an elevated temperature. TPD analyses determine the number, type, and strength of active sites available on the surface of a catalyst from measurement of the amount of gas desorbed at various temperatures. During a TPR analysis, a metal oxide is reacted with hydrogen to form a pure metal. TPR determines the number of reducible species present in the catalyst and reveals the temperature at which reduction occurs. TPO examines the extent to which a catalyst can be reoxidized and measures the degree of reduction of certain oxides.

ChemiSoft TPx Software

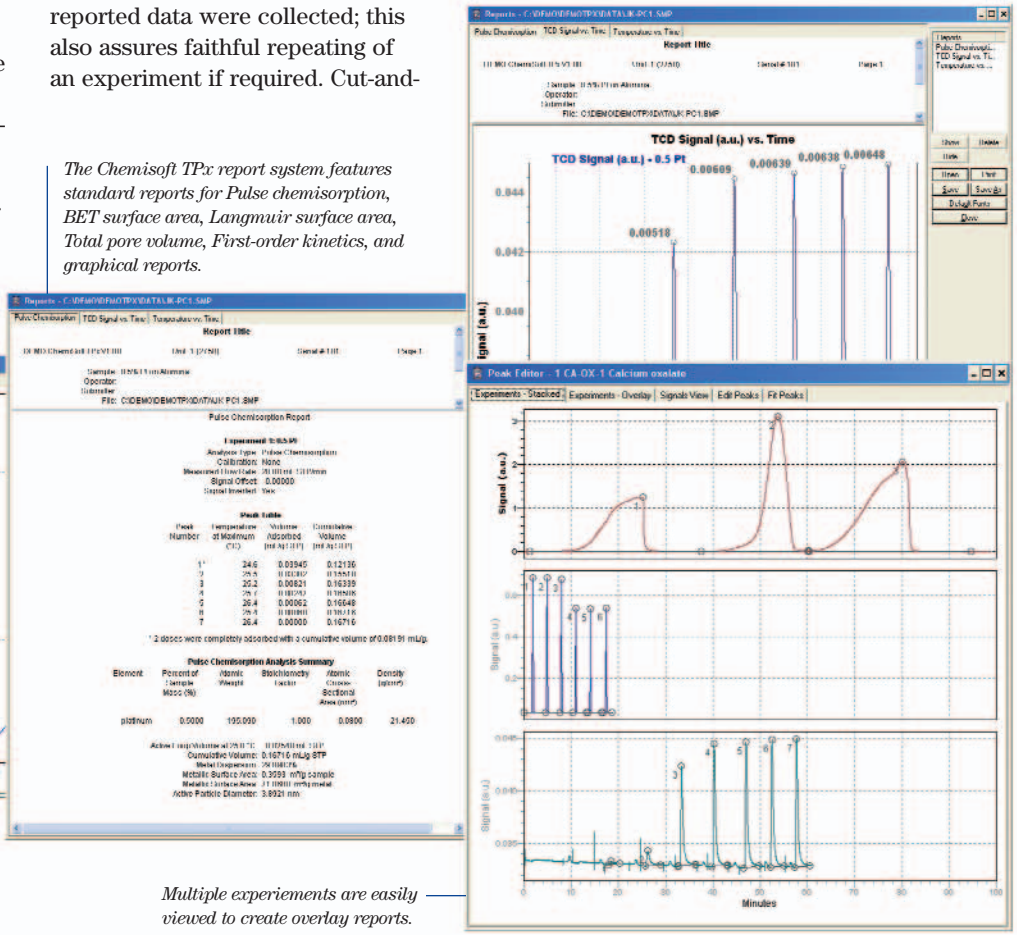
Included in the Temperature-Programmed Chemisorption option is Micromeritics' ChemiSoft software that can be used to simplify chemisorption and physisorption as well as temperature-programmed analyses. The software tracks and records time, monitors and records the analytical temperature and detector output, creates and organizes data files, reduces collected data, and produces a variety of user-defined reports. Advanced peak integration capabilities assure reliable results.

With ChemiSoft, you can create and store standard sets of analysis conditions for guiding frequently performed analyses. Analysis and prep conditions also are reported to provide a record of the environment under which the reported data were collected; this also assures faithful repeating of an experiment if required. Cut-and-paste and data export features simplify moving data to reports or incorporating chemisorption data with data from other analytical techniques.

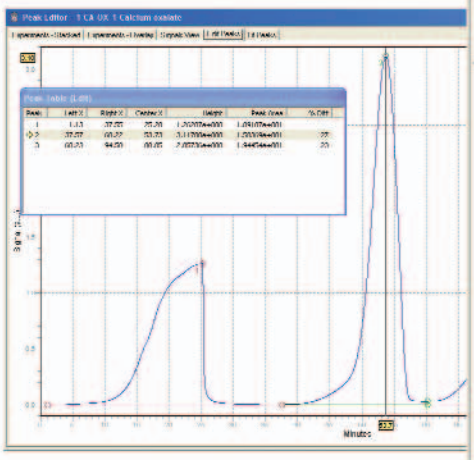
ChemiSoft TPx software can be set up to run independently of the instrument. This means that data files can be reviewed, calculation parameters changed, and reports generated on any computer anywhere, anytime.

For the novice operator, the software features prompts for each step in the analysis process, literally talking the user through an analysis sequence. When you are ready to move up to Micromeritics' more advanced AutoChem 2920 or ASAP 2020 Chemi, or to incorporate other Micromeritics products into your laboratory, your operators will find that the format of the operating software is the same from product to product, thus training time is minimized.

The graphical reports can be customized by the user to show company logos, user-selected fonts, and user-selected line colors.



The Chemisoft TPx report system features standard reports for Pulse chemisorption, BET surface area, Langmuir surface area, Total pore volume, First-order kinetics, and graphical reports.



The integrated Peak Editor is a valuable tool for rapidly integrating the TCD signal. Each peak is clearly marked to show the peak start, end, maximum, and baseline. A peak summary table is also provided.

Multiple experiements are easily viewed to create overlay reports.

Data Reduction and Reporting Versatility

The basic instrument without the Temperature-Programmed Chemisorption option provides two ways to obtain data:

- Front panel meter readout
- Chart recorder monitoring the analog output from the thermal conductivity detector

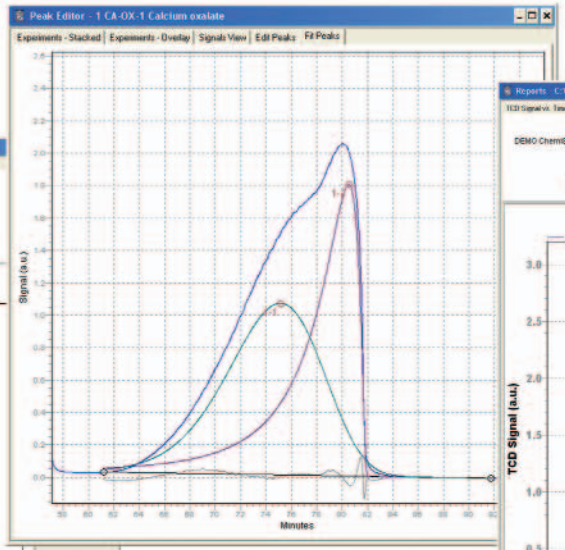
Data are reduced and reported automatically by the **ChemiSoft TPx software**, which is part of the Temperature-Programmed Chemisorption option and can be used with either ChemiSorb instrument. Reports include single- and multipoint BET surface area, percent dispersion, peak area volume, calculated stoichiometry factor, gram molecular weight, cross-sectional area, calculated metal density, active (metallic) surface area, active particle (crystallite) size, and activation energy (first order kinetics).

When used with the ChemiSorb 2750, additional reports on Injection loop calibration, Injection loop volume with temperature change, and calibration error are available.

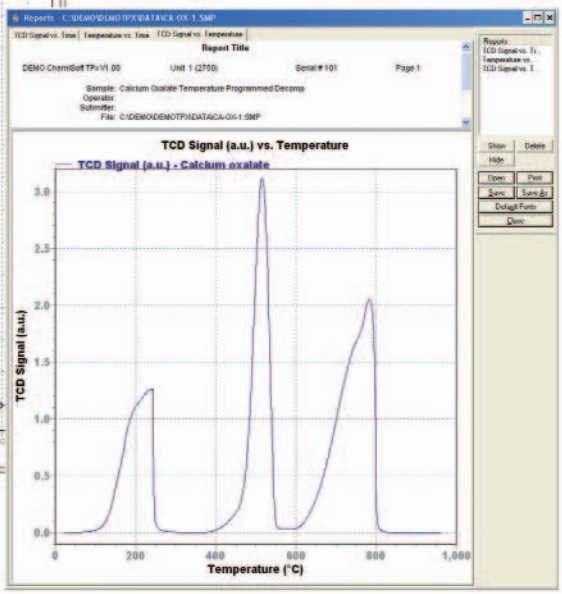
Other functions of ChemiSoft:

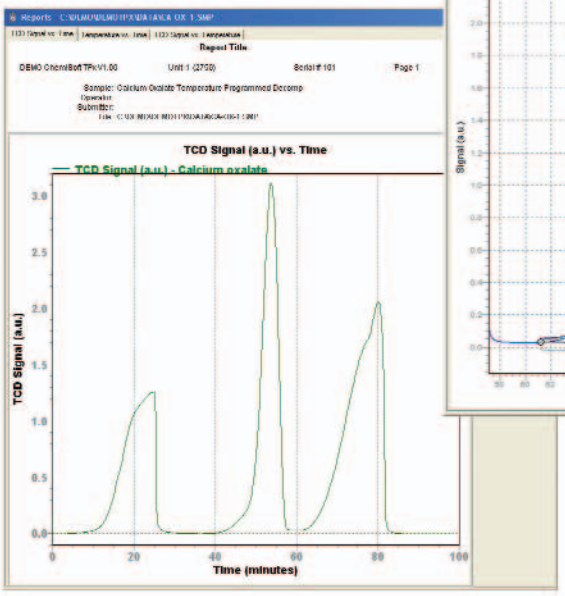
- Allows control of units, axis scales, and reporting range
- Prints report to screen, printer, or file (text only)
- Cut-and-paste capabilities
- Capture displayed plots as a series of x-y coordinates
- Capture tables from screen as ASCII text files
- Integrates detector signals both automatically and manually
- Displays and prints peak graphs and reports
- Establishes calibration curves for calculation of unknown sample concentrations
- Reprocess stored analysis data using different parameters
- Exports data in ASCII text format for use in other applications
- Allows for off-line data manipulation
- Provides the ability to monitor two instruments from one computer
- Monitors and records furnace temperature

Complex TCD signals can be deconvolved to provide valuable insight and demonstrate that the composite signal is the sum of two peaks.



The detector signal can be plotted versus temperature. This allows the user to easily compare temperature-programmed analyses for multiple samples.





The ChemiSoft TPx accessory allows the user to acquire the detector signal, the sample temperature, and the furnace temperature. These siganls can then be included graphically in reports.

To request a quote or additional product information, visit Micromeritics' web site at www.micromeritics.com, contact your local Micromeritics sales representative, or our Customer Service Department at (770) 662-3636.

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