3P INSTRUMENTS

Characterization of
particles · powders · pores

Altamira Series

- Automated Reactor Systems
- Temperature Programmed Reduction
- Temperature Programmed Oxidation
- Temperature Programmed Desorption
- Pulse Chemisorption
- Flow BET

Catalyst Characterization by Chemisorption

AMI Chemi Family Overview / Contents

Overview

The AMI Chemi family of instruments combines a variety of temperature-programmed techniques, including: temperature-programmed desorption (TPD), temperature-programmed oxidation (TPO), temperature-programmed reduction (TPR), and temperature-programmed reaction (TPRx). The AMI Chemi family of instruments can also perform pulse chemisorption, dynamic BET, and gas-phase iso-thermal reaction experiments. The AMI family includes high-pressure instruments with the AMI-300HP; sulfur resistant models with the AMI-300S, kinetic determining instrumentation with the AMI-300 SSITKA, and the new AMI-300IR for direct determination of adsorption/desorption surface process.
The leader in automated TPD/TPR/O/TPRx, and pulse chemisorption instruments since 1985, Altamira Instruments is the catalyst researcher's primary resource for automated catalyst test instruments and bench-scale reactor systems. The AMI series of instruments were the first fully automated chemisorption machines on the market.

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AMI Family of Catalyst Characterization Instruments

AMI-300 Functions:

- Temperature Programmed Reduction
- Temperature Programmed Oxidation
- Temperature Programmed Desorption
- Pulse Chemisorption
- Flow BET

AMI-300

- "flagship" of the AMI family
- single station instrument with stand-alone PC and LabVIEW-based control software
- basis upon which the rest of the AMI-technology and software is based

AMI-300IR

- uses heated IR transmission cell coupled with an FTIR
- delivers real time information as to what is actually occurring on the surface of a catalyst

AMI-300IP

- two-workstation version wherein one station conducts pretreatment steps, while the second station performs a characterization experiment
- both workstations can run either a pretreatment step or a characterization experiment

AMI-300Lite

- entry-level version, which sheds some features of the AMI-300 like the saturator and gas blending as a concession to pricing
- operates on an unattended basis and performs all TPR, TPO, TPD, and Pulse Chemisorption functions

AMI-300R

- reaction version of the AMI-300
- equipped with additional gas feeds and HPLC pump(s) with vaporizer(s) to facilitate reaction studies.
- features an auxiliary vent to route reactant gases directly to a secondary detection device (e.g., RGA, GC, or MS)

AMI-3300

- three-workstation version of AMI-300
- provides all of the featuresof the AMI-300 instrument and can run three independent experiments simultaneously
- controlled via one single PC

AMI-3300Lite

- three-workstation version of the AMI-300Lite
- provides all features of the AMI-300Lite and can run three independent experiments simultaneously
- controlled via one single PC

AMI-300R (HP)

- custom high-pressure version of the AMI-300R
- equipped to provide 100 bar (or more) of operating pressure during a reaction experiment

AMI-5300

- five-workstation version of the AMI-300, which can run five independent experiments simultaneously
- controlled via one or two PC's

AMI-5300Lite

- five-workstation version of the AMI-300Lite, which can run five independent experiments simultaneously
- controlled via one or two PC's

AMI-8300

- eight-workstation version of the AMI-300, which can run eight independent experiments simultaneously
- controlled via two PC's

AMI-8300Lite

- eight-workstation version of the AMI-300Lite, which can run eight independent experiments simultaneously
- controlled via two PC's

AMI-300 Chemisorption Analyzer

AMI-300

Perform dynamic temperature-programmed catalyst characterization experiments unattended with the latest generation fully automated chemisorption analyzer from Altamira Instruments. Determine metal dispersion, relative activity, adsorption strength, in one third the time of traditional volumetric methods. Analyze the off gas with the standard TCD or integrate a mass spectrometer or other detectors (FID, GC, FTIR) as options. Let us customize the instrument so you can, for example:

- Use it as an atmospheric reactor,
- carry out lengthy pre-treatments in a separate station,
- provide fast-switched feed compositions to mimic lean-rich burn scenarios,
- provide SSITKA capability.

Dynamic Chemisorption

The AMI-300 utilizes our proven technology for performing the following dynamic procedures:

- Temperature programmed desorption (TPD)
- Temperature programmed reduction/oxidation (TPR/O)
- Temperature programmed reaction (TPRx)
- Gas-phase isotherm reaction experiments (standard integrated gas-mixing)
- Pulse chemisorption
- Catalyst treatment
- Flow BET surface area
- Pulse calibration

Up to 99 procedures can be linked together consecutively to provide a complete characterization experiment. All experimental procedures can be designed and stored for easy retrieval.

Hardware and Operation

The AMI-300 is a fully automated catalyst characterization instrument wherein the LabVIEW-based software is used to switch gas streams, control gas flow rates, blend gases, control temperatures, control ramp rates, and to collect all the data needed to quantify the adsorption and desorption of gas molecules on the surface of a catalyst.

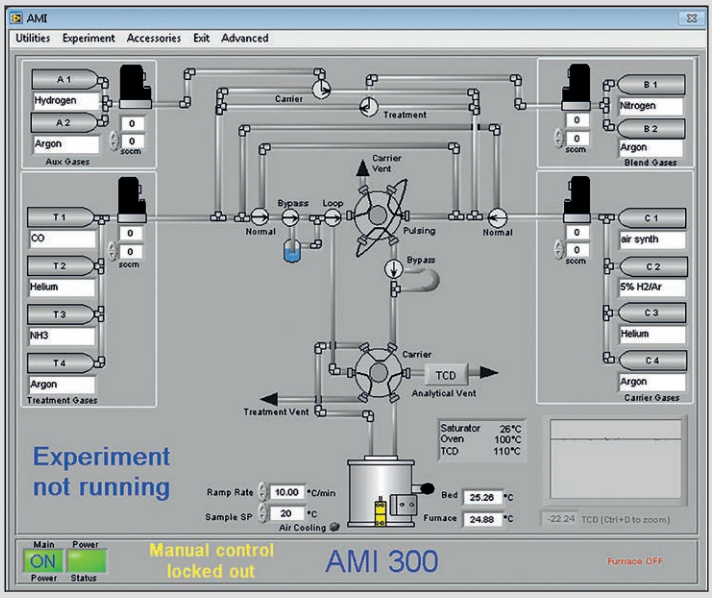


Figure 1 *Screenshot of the LabVIEW-based software*

Functions

- High-precision electronic mass flow controllers, which ensure a stable baseline with changing temperatures and also ensure quality of data
- Use of 1/16" stainless steel tubing to minimize dead volume
- A clamshell furnace with adjustable mounting to ease insertion and removal of samples
- Capability of heating catalyst samples to 1,200 °C with linear temperature ramps from 1 °C to 50 °C per minute. Higher and lower ramp rates are also available.
- A subambient option which provides the capability to operate linearly, between -130 °C and 1,200 °C.
- Automatic air-cooling of the furnace which is provided to speed the time between experiments
- A Universal temperature zone which controls the temperature of all valves and lines downstream of the reactor. The temperature of this zone is controlled by the software and serves to prevent both condensation and retention of the adsorbate in valves and lines upstream of the detector.
- A saturator is provided as an option to suffuse the treatment gas with liquid vapors. The temperature of the saturator is controlled by the software. In applications where condensation of liquid vapors in the line between the saturator and the universal temperature zone is a concern, a reflux heater is available to ensure that no condensation takes place.
- A highly linear thermal conductivity detector (TCD) is standard. The resolution of the TCD is operator defined, and is controlled by the software.
- A choice of TCD filaments is offered to maximize sensitivity and chemical compatibility of the instrument

Computer Control and Data Acquisition

The AMI-300 is fully automated and computer-controlled for ease of operation and reliability. It is designed so that experiments can be run on an unattended basis. This PC, supplied with a Windows based operating system, can be networked and is capable of managing other laboratory tasks in addition to operation of the instrument. The control and data acquisition functions are performed by LabVIEW applications software. Using this software, a complete experiment can be set-up in minutes, and it can be saved for future use or modification. The control portion of the software controls and regulates all valve positions, temperatures, flow rates, and detector parameters. The software also allows for the use of an alarm matrix to ensure the safety of the system. Data acquisition occurs at a selected rate for optimum performance. An "Overview" screen shows the status of the unit at a glance, providing information on the position of all valves, type of gas connected to each port, temperatures, and detector signal. Lines change color to illustrate the current flow path. In manual mode any valve can be switched just by a click of the mouse on its icon. Gas flow and temperature setpoints can be entered from this screen. The most sophisticated data handling package in the industry is included which allows the user to display and integrate signal peaks, calculate chemisorptive parameters, peak fit, and overlay data.

Detectors

The AMI-300 comes standard with a highly linear TCD. In addition to the TCD, AMI instruments have been delivered with a wide range of auxiliary detection devices. Choices of auxiliary detectors have included:

- Mass Spectrometer
- GC with FID/TCD/FPD
- Flame Ionization Detector
- FTIR

Any detector that provides an analog output or which can communicate through DDE, Serial ports, OPC, ActiveX, or other Windows protocols can be triggered by the AMI-300. The degree to which the auxiliary detection device is to be integrated with the instrument can be specified and incorporated into the control software.

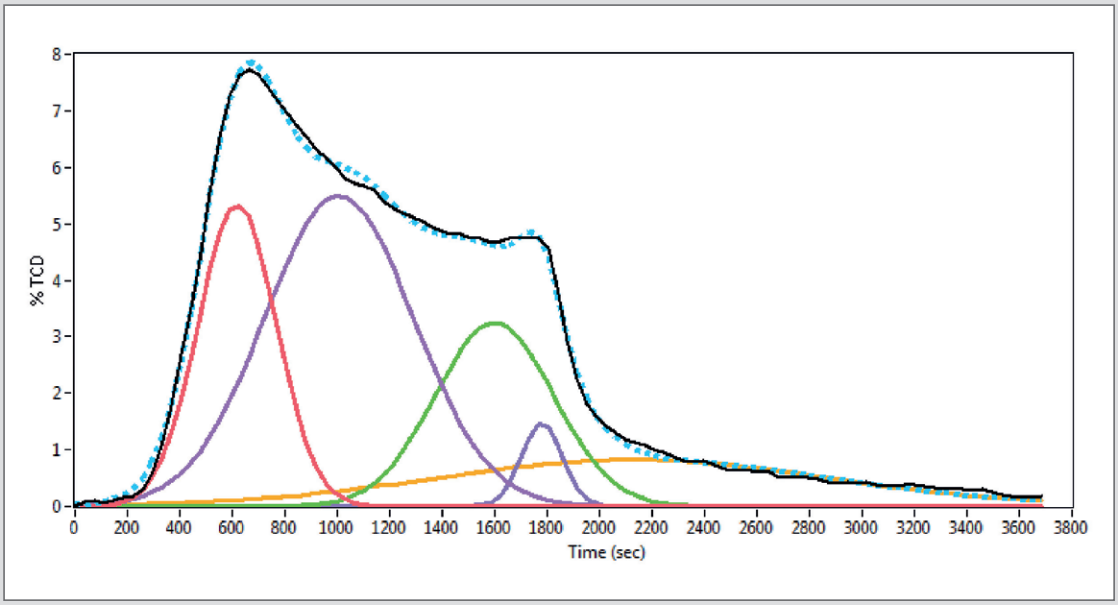


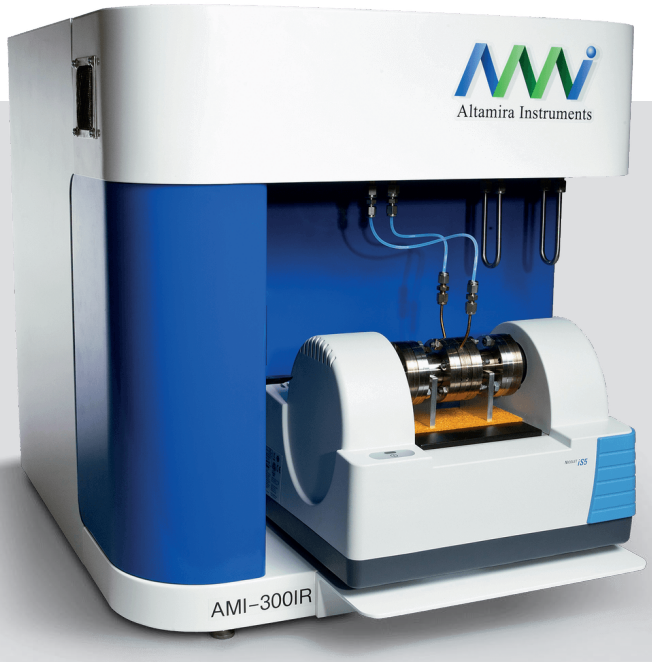
Figure 2
Deconvolution of hydrogen TPD from a 64 % Ni/SiO_2 catalyst (software screenshot). Different peaks correspond to different adsorbed species

Specifications	
Standard catalyst charge	0.1 – 1.0 grams (can be increased with custom sample tubes)
Temperature Range	-130 °C to +1,200 °C with subambient option
Ramp rate	1 – 50 °C/min
Operating Pressure	Atmospheric
Gas Inlets	4 carrier, 4 treatment, 2 blend (others available)
Gas Flow Rates	5 – 50 mL/min (others available)
Reactor Types	Quartz U-tubes, bubble tubes, monolith tubes (up to 1" diameter)
Primary Detectors	4 filament TCD with choice of material (W, Au/W)
Materials of construction	■ Flow path: 316 stainless steel ■ Seals: Viton, Buna-N, or Premium Seals (depending on customer chemical compatibility requirements)
Dimensions / Weight	64 cm (W) x 64 cm (H) x 64 cm (D) / ca. 55 kg
Power Supply	110 – 120 or 220 – 240 Volts, single phase, 50 or 60 Hz. ~20 amp service

Features

- **Seven (7) Standard Procedures** - Pulse chemisorption, TPR/TPO, TPD, TPRx, treatment, pulse calibration, and flow BET. Ability to link 99 procedures into one experiment.
- **Three Independent Gas Sources** - In addition to a carrier gas and a treatment gas, the AMI300 provides one additional independently controlled auxiliary gas inlet which can be blended with the carrier gas or treatment gas.
- **Ten (10) Gas Ports** - Gas ports located on the back of the instrument minimizes the time expended changing gases. Four ports each for treatment and carrier gases. Two ports are available for gas blending or auxiliary gases. Additional ports can be added.
- **Electronic Flow Controllers** - Unit uses high quality linear mass flow controllers to display and control gas flows. Flow rate span is 0 – 50 mL / min (standard). Other ranges are available upon request.
- **Interchangeable Valve Loops** - Provide an easy way to reach the appropriate level of sensitivity for different samples.
- **Liquid Vaporizer** - Unit can be equipped with a heated sparger-type saturator for easy introduction of volatile liquids.
- **High Temperature Furnace** - Temperature up to 1,200 °C; with sub-ambient cooling, the lower limit is -130 °C. Furnace can be linearly ramped from 1°C / min to 50°C/min.
- **Air Cooling** - Cools the furnace rapidly for quick sample turn around.
- **Sample Temperature** - Measured by a moveable thermocouple placed at the top of the sample bed.
- **Various Sample Holders** - Unit accommodates a variety of quartz U-tubes sized to accommodate various catalyst volumes and sizes: straight, bubble, preheat, monolith. Samples may be powders, pellets, extrudates or honeycomb cores.
- **Easy Sample Loading** - A movable furnace allows easy removing and loading of the sample holder.
- **Trap** - A trap that may be filled with a desiccant or used as a cold trap is provided downstream of the sample holder in order to remove condensables before the TCD.
- **Heated Null Station** - Ensures accurate calibration pulses without contact with the sample.
- **Injection Port** - A syringe injection port is provided for exact loop volume calibration.
- **Heated Lines** - All lines, valves, and parts of the liquid vaporizer are heated to prevent condensation.
- **Thermal Conductivity Detector** - Highly reliable 4-filament TCD used to quantify gas uptakes. Excellent linearity, accuracy, sensitivity, and stability. Several filament options are available.
- **Direct Mass Spectrometer Link** - MS data can be integrated in real-time with AMI-300 data using Direct Data Exchange (DDE).
- **Auxiliary Detectors** - Can accept any auxiliary detector providing a voltage analog output, such as a flame ionization detector (FID).
- **Low Internal Volume** - Low volume valves and 1/16" lines are used to reduce void volume and minimize peak spreading.
- **Materials of Construction** - Seals and materials are custom engineered to meet your specifications.
- **Safety Features** - Among others: independent over-temperature protectors for furnace, resealable pressure relief valves, check valves, circuit breakers, and fuses. System is designed to fail in safe mode.

AMI-300IR Chemisorption Analyzer with IR cell



IR Transmission Cells

As part of the development of this new product, Altamira has designed a variety of heated IR transmission cells which fit comfortably into commercial FTIR instruments and which in turn fit into the furnace cavity of a standard AMI-300.

A sample can be prepared in the form of thin, self-supported catalyst wafers by pressing approx. 100 mg of catalyst powder into a disk and then securing it in a proprietary catalyst holder within the IR cell. The IR beam can thus pass through the catalyst wafer. Fig. 3 and 4 show a schematic of the cell and a photograph of an actual cell, respectively.

The sample can then be subjected to all the standard AMI-300 procedures while monitoring the catalyst surface and the adsorbates via the IR spectrometer. At the same time, the effluent gas can be monitored using the instrument's standard TCD or an optional mass spectrometer.

In this manner one can gather information, in real time, as to what is actually occurring on the surface of a sample.

AMI-300IR

Chemisorption and thermal desorption techniques, such as TPD (temperature programmed desorption), have been widely used for the characterization of catalysts. These techniques rely solely on the observation of evolved gases from a catalyst surface as detected by a thermal conductivity detector (TCD) or, in some cases, a mass spectrometer. With these techniques it is possible to learn about the number and strengths of sites but not about the nature of the sites, the type of adsorption, or whether there exist multiple types of sites. In order to overcome this drawback, Altamira has developed the AMI-300IR which combines all the standard AMI techniques with real-time observation of the catalyst surface by Fourier Transform Infrared (FTIR) spectroscopy. This combination of techniques allows for the direct observation of the adsorbed species and thus expands the knowledge of the nature of the adsorption/desorption process.

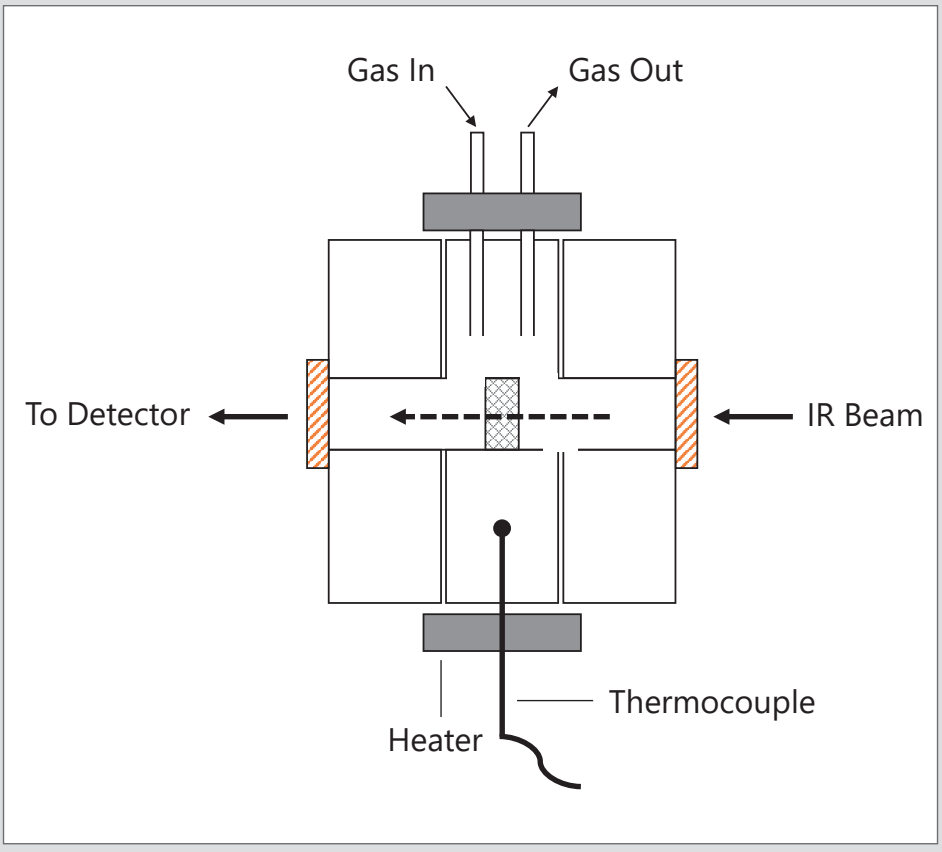


Figure 3 *Diagram of IR transmission cell*

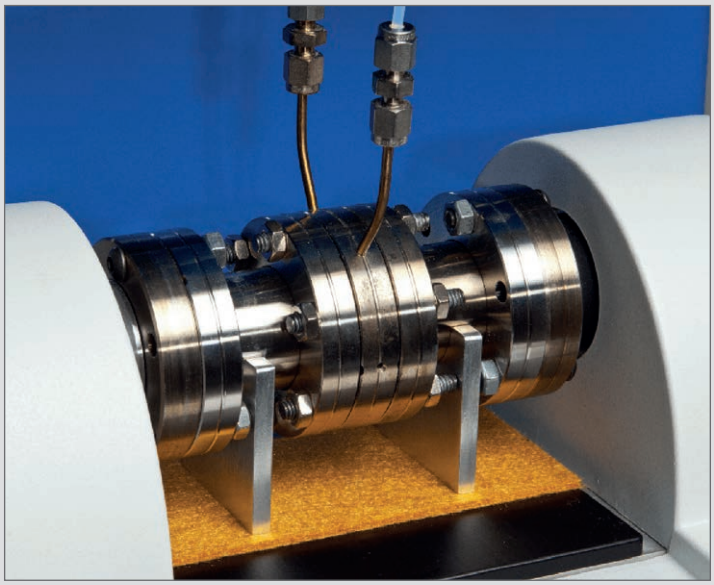


Figure 4 *IR transmission cell. Heaters and insulation have been removed for clarity*

Observation of CO adsorbed on a Pt Catalyst

An example of the kind of information that can be obtained with this technique is the mode of adsorption and desorption of CO on a platinum surface. A 1% Pt/Al_2O_3 catalyst was pressed into a wafer and mounted on an IR cell. The sample was reduced for several hours at 200 °C, cooled to room temperature, and then flushed with inert gas for an hour in order to remove the gas-phase and any loosely held CO. The resulting IR spectrum (background subtracted) showed a single sharp line at approximately 2,060 cm^{-1} corresponding to linearly adsorbed CO (see Fig. 5).

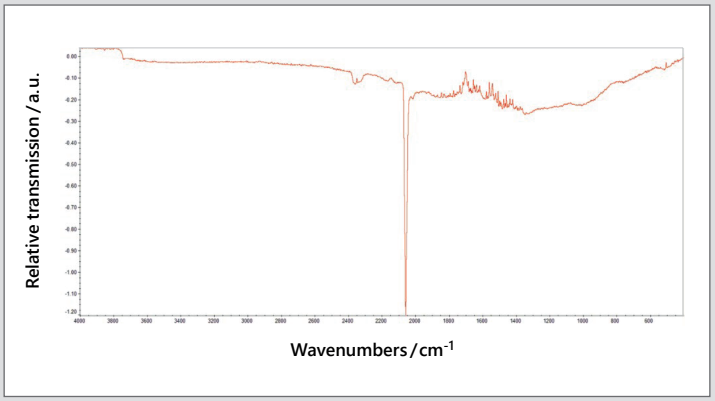


Figure 5 *IR spectrum of CO adsorbed on a 1% Pt/Al_2O_3.*

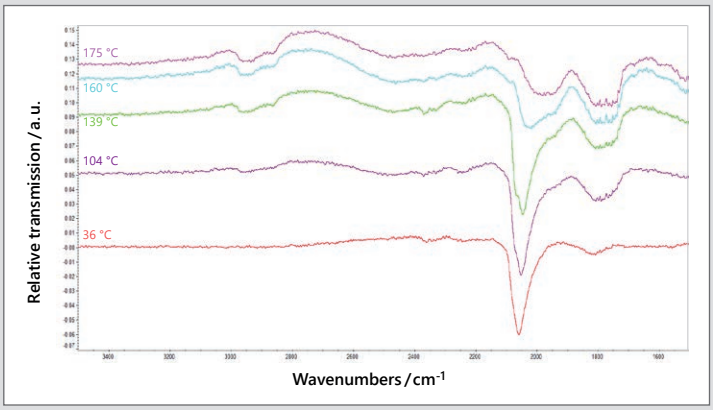


Figure 6 *CO signal as a function of temperature*

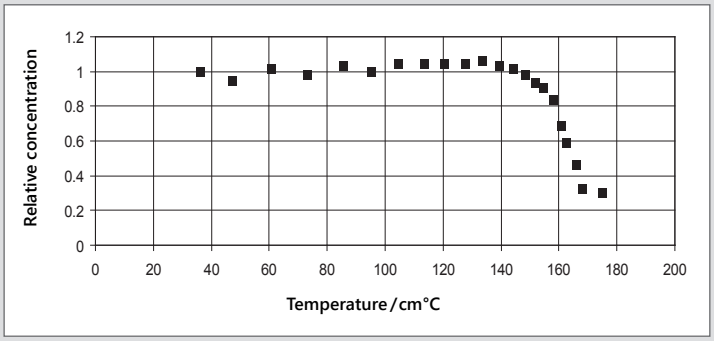


Figure 7 *Isobar of CO adsorbed on 1% Pt/Al_2O_3*

This sample was then heated and the CO band followed as a function of temperature (Fig. 6). According to Beer's Law, absorbance is proportional to concentration so from these measurements it is possible to construct an isobar and obtain a derived TPD from that. These are shown in Fig. 7 and 8, respectively.

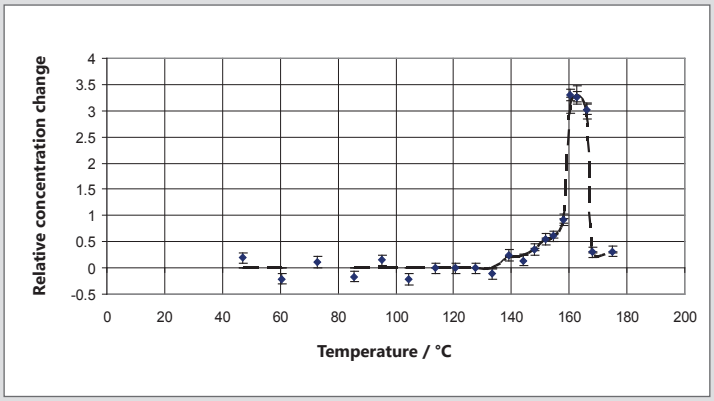


Figure 8 *Derived CO TPD.*

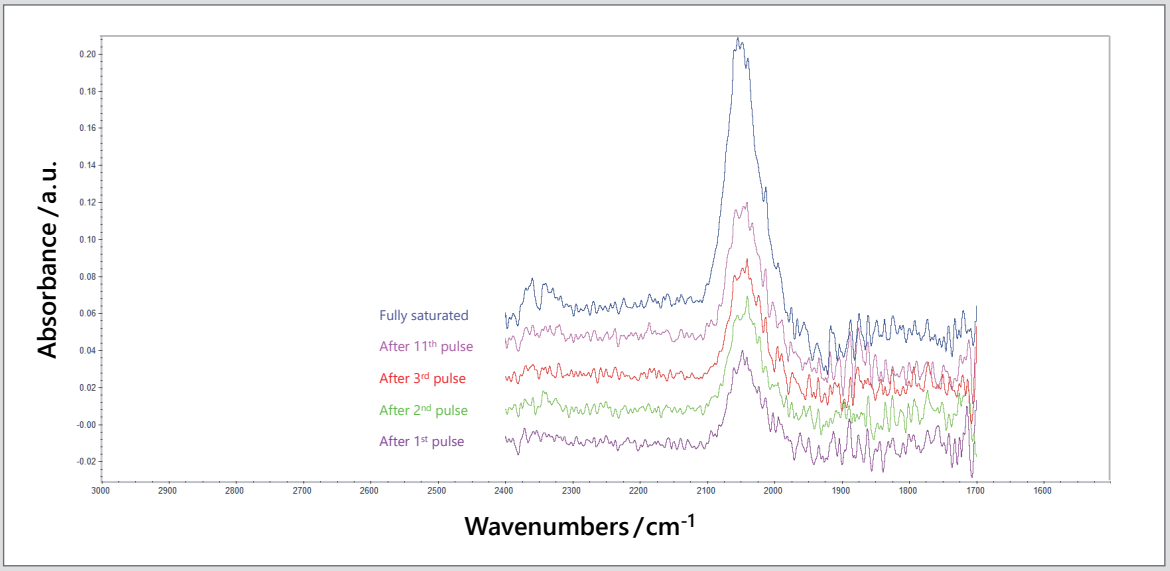


Figure 9
Pulse chemisorption of CO on Pt by FTIR

Pulse Chemisorption Detection by IR

IR detection can also be used during pulse chemisorption procedures to ascertain the mode(s) of adsorption at different coverages. Fig. 9 illustrates CO adsorption on platinum as the coverage increases. Even at low coverages all the CO is adsorbed in a single mode, linearly, and there is no evidence for "bridged" CO. These kinds of observations can only be achieved using IR detection since it involves looking at the actual catalyst surface and not at the evolved gases.

Differentiation between Brønsted and Lewis acid sites using Ammonia chemisorption

Ammonia can be used as a probe molecule to determine the magnitude and type of acid sites in a catalyst. Below, in Fig. 10, is an example of ammonia adsorbed on a silica-alumina material. Three broad bands were identified as belonging to the adsorbed ammonia, at approximately 1,760, 1,480, and 1,380 cm^{-1}. The band at 1,480 cm^{-1} can be ascribed to ammonia adsorbed on Brønsted acid sites, the others to ammonia adsorbed on Lewis sites (see for example, M. Niwa et al., J. Phys. Chem. B, 110 (2006) p. 264). By carrying out a temperature programmed experiments and following the absorbance of the three bands as a function of temperature, it is possible to measure the isobars for each type of adsorption and examine the strength of each particular adsorption. These isobars are shown in Fig. 11.

It can be seen from these data that the adsorption reflected in the 1380 cm^{-1} band is more strongly held than the other two perhaps indicating a stronger Lewis-type bond.

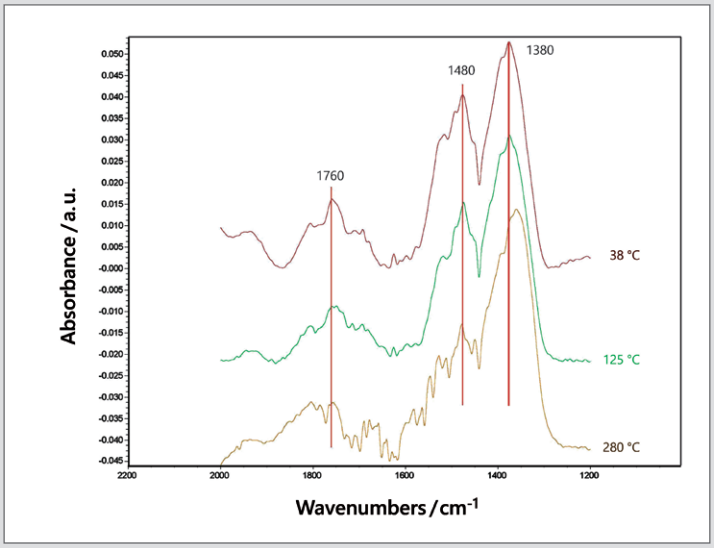


Figure 10 *Ammonia bands on silica-alumina shown at three different temperatures*

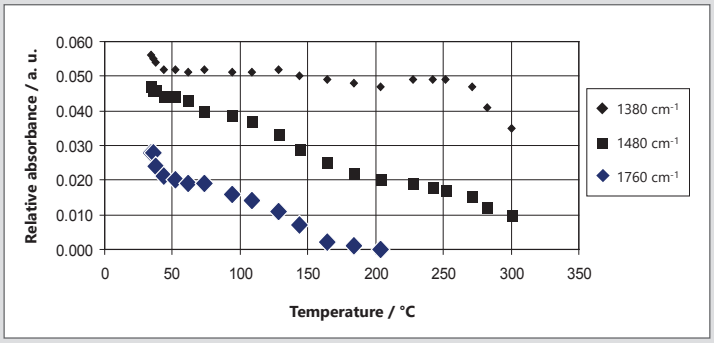


Figure 11 *Isobars for each of the three main ammonia bands on silica-alumina*

AMI-300RHP

The AMI-300HP represents an automated high pressure chemisorption analyzer. The AMI-300HP performs dynamic high-pressure temperature-programmed experiments designed to reach 100 bar (higher pressures available in custom instruments). When integrated with a pump, the AMI-300RHP provides the most sophisticated and automated characterization and micro-reactor instrument in the industry. The sophisticated design allows for the system to also double as a single-station Bench-top reactor.

Functions

The AMI-300HP can be designed with any number of pressure and temperature ranges in mind. The AMI-300HP utilizes our proven technology to perform:

- High pressure isothermal reaction studies
- High pressure chemisorption experiments
- Temperature programmed reduction / oxidation / desorption (TPR/O)
- Temperature programmed reaction experiments with vaporized liquids (TPRx)
- Pulse chemisorption
- Pulse calibration
- Catalyst treatment
- Dynamic BET (optional)

Up to 99 procedures can be linked together back-to-back to provide a complete characterization experiment. Routine experiments can be designed and stored for easy retrieval.

Hardware and Operation

- A clamshell furnace capable of 1,200 °C (reactor type limits max temperature) with ramp rates from 1 °C to 50 °C per minute.
- High-precision mass flow controllers (MFCs) to ensure TCD baseline stability
- A heat-traced stainless-steel flow-path to alleviate concerns of condensation and to provide an inert surface.
- A highly linear Thermal Conductivity Detector (TCD)

Options for:

- Dynamic BET
- Quartz Reactor
- Vacuum Activation
- Liquid delivery
- Inconel, Hastelloy, or other high temperature reactors
- Sub-ambient (-130 °C or lower) temperatures

Computer Control and Data Acquisition

The AMI-300HP is fully-automated for ease of use and reliability. The control portion of the software controls and regulates all valve positions, temperatures, flow rates, and detec-

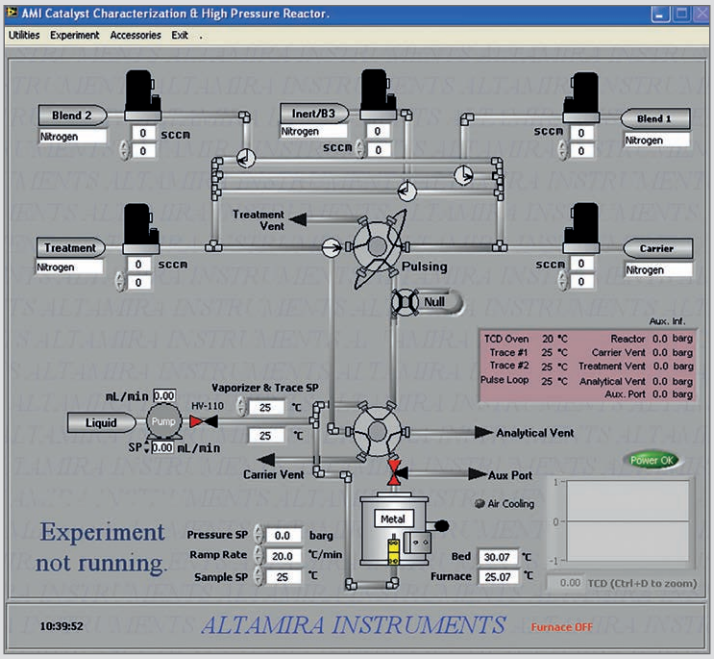


Figure 12 *Operating Screen - A complete overview of all experimental parameters*

tor parameters. Data acquisition occurs at a selected rate for optimum performance. The front panel screen shows the status of the unit at a glance. This screen provides information on the position of all valves, type of gas connected to each port, temperatures, and detector signal.

The data handling package allows the user to display and integrate signal peaks, calculate chemisorptive parameters, and overlay data.

Choice of Detectors

With the AMI-300HP, you can use the standard thermal conductivity detector or choose other detectors of your choice. We supply integrated mass spectrometers that can be used in addition to the TCD. In addition, we can incorporate any detector that provides an analog output or can communicate through Dynamic Data Exchange (DDE).

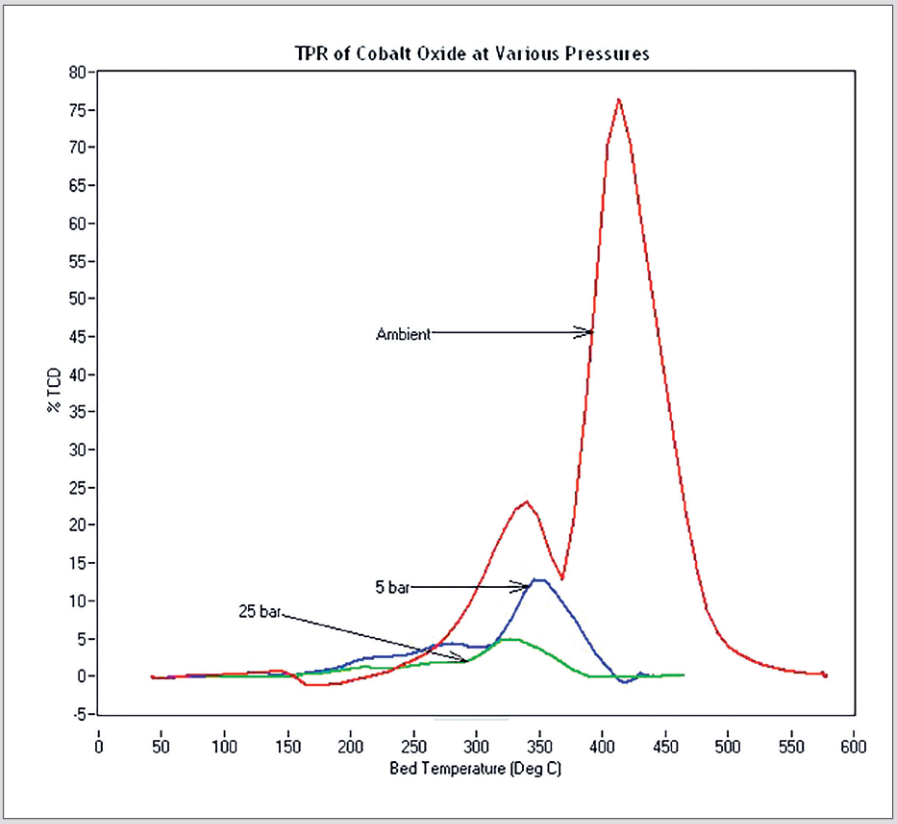
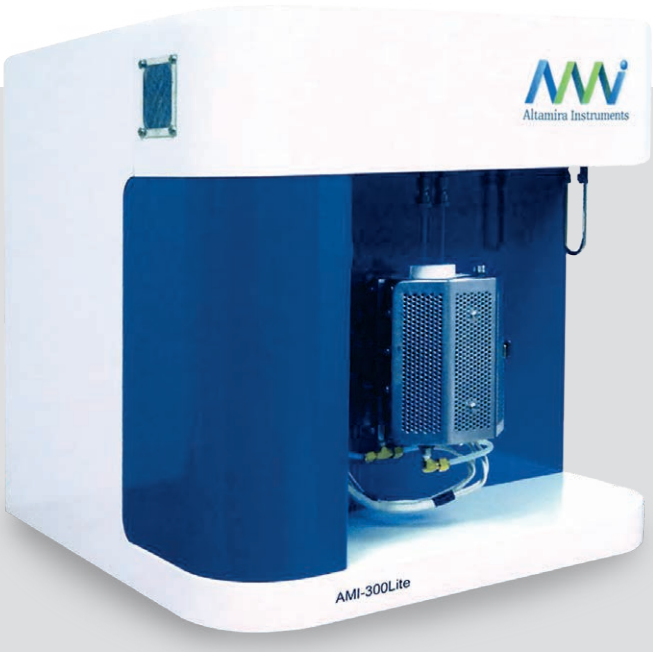


Figure 13 *TPR of 10% Co / 1% Re on Al_2O_3 at different pressures*

Specifications	
Catalyst Charge	0.1 – 1 g (1)
Temperature Range	-130 to 1,200 °C (2)
Ramp Rate	1 – 50 °C/min
Operating Pressure	100 bar (3)
Gases	4 MFCs for carrier, treatment, pulse, and TCD gases (4)
Gas Flow Rates	User defined
Reactor Types	SS 316 (5)
Detector	4 filament TCD with choice of material (W, Au/W)

(1) Custom reactors available for increased loading
(2) Standard temperature range is 25 °C – 650 °C; -130 °C – 1,200 °C requires options
(3) Higher pressure available in custom instruments
(4) Number of MFCs can change to increase capability or lessen cost
(5) Other reactor materials are available

AMI-300Lite Basic Chemisorption Analyzer for Routine Analyses



AMI-300Lite

Based on our successful AMI-300 instrument, the AMI-300Lite allows for rapid chemisorption studies in a compact, affordable package. The AMI-300Lite was designed specifically for the price-conscious customers with applications that are more routine or less demanding. As always, full automation and powerful data-handling software assure data accuracy and improve laboratory efficiency.

Features

The AMI-300Lite represents a new affordable standard in automated chemisorption analysis. Developed by and for catalyst researchers, it and its predecessors, the AMI-1/100/200, were first in the industry to offer a fully automated system capable of performing all the major dynamic techniques required for fully characterizing a catalyst. The AMI-300Lite utilizes proven technology for performing the following procedures:

- Temperature programmed desorption (TPD)
- Temperature programmed reduction/oxidation (TPR/O)
- Pulse chemisorption
- Catalyst treatment
- Pulse calibration
- Single point BET

Up to 99 procedures can be linked together back-to-back to provide a complete characterization experiment. Routine experiments can be designed and stored for easy retrieval.

Hardware and Operation

The AMI-300Lite is a fully automated, atmospheric pressure system. Through software control, it can switch gas streams, control gas flow rates, control temperature and temperature ramps, and collect desorption data in order to quantify the adsorption and desorption of gas molecules for a catalyst surface.

To improve the quality of data, the AMI-300Lite utilizes a high-precision electronic mass flow controller for both the carrier and treatment gases. This ensures constant treatment flow and a stable baseline with changing temperatures. Dead volume is minimized by using 1/16" plumbing in order to reduce peak spreading. The furnace can heat the sample to 1,000 °C (1,200 °C optional) with linear temperature ramps to 50 °C/min.

The AMI-300Lite is fitted with a highly linear thermal conductivity detector (TCD). A choice of filament types is offered to maximize the sensitivity for your particular analysis.

AMI-300Lite Basic Chemisorption Analyzer for Routine Analyses

Computer Control and Data Acquisition

The AMI-300Lite is fully-automated for ease of use and reliability. The control portion of the software controls and regulates all valve positions, temperatures, flow rates, and detector parameters. Data acquisition occurs at a selected rate for optimum performance. An "Overview" screen shows the status of the unit at a glance. This screen provides information on the position of all valves, type of gas connected to each port, temperatures, and detector signal.

Also included are industry leading safety features with auto abort available from software.

The data handling package allows the user to display and integrate signal peaks, calculate chemisorptive parameters, and overlay data.

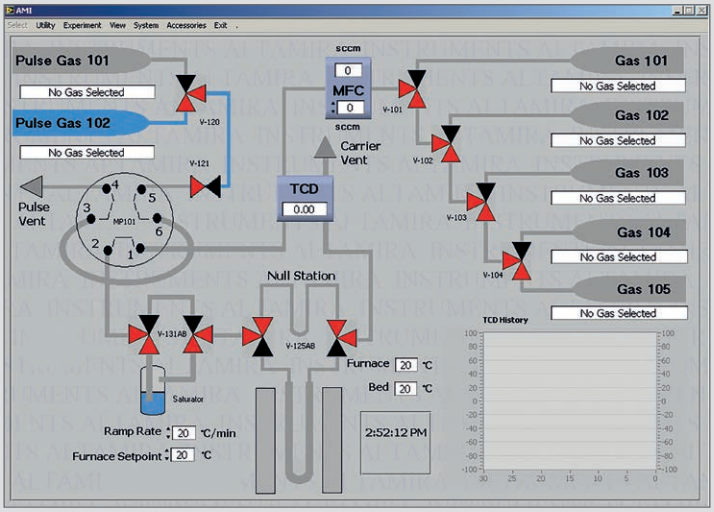


Figure 14 *Operating Screen - A complete overview of all experimental parameters*

Specifications	
Catalyst Charge	0.1 – 1 g
Temperature Range	25 – 1,000 °C (1,200 °C option)
Ramp Rate	1 – 50 °C / min
Operating Pressure	Atmospheric
Gases	5 carrier/treatment 2 pulse gases; including H_2, CH_4, C_2H_4, CO_2, CO, NO, O_2, N_2, SO_2, NH_3, N_2O, He, Ar
Gas Flow Rates	5 – 50 mL / min
Reactor Types	Quartz u-tubes
Detector	4 filament TCD with choice of material (W, Au/W)
Options	Extra MFC for blending; Sub-ambient operation (-130 °C); Sample pre-treatment station
Materials of Construction	Stainless steel; seals are either Buna-N, Viton or Kalrez
Dimensions	61 cm (W) x 64 cm (H) x 61 cm (D)

AMI-300EZ Most Economic Chemisorption Analyzer

AMI-300EZ

This instrument the AMI-EZ operates on an unattended basis and comes with our user-friendly LabVIEW designed software package for programming, monitoring, and data analysis. Also included with the system is a computer with the software loaded and various test methods ready to run. For sample analysis, a high quality, linear Thermal Conductivity Detector is included, with heating capabilities up to 200 °C to help prevent condensation and corrosion. Programming and monitoring are accomplished through the LabVIEW software.

The AMI-300EZ has the following functions:

- Temperature programmed desorption (TPD)
- Temperature programmed reduction/oxidation (TPR/O)
- Pulse chemisorption
- Catalyst treatment
- Pulse calibration
- Gas phase reaction studies

Hardware and Operation

A high-quality programmable furnace is standard with heating up to 500 °C with option for operation to 1,200 °C, including fast cool down capabilities. Instrument layout was designed with the service engineer in mind. All components are easily accessible and of high quality with self-diagnostic features available through the operating software. Internal tubing has minimal dead volume through the use of 1/16 inch tubing and an optimized flow path to ensure the least sample lag-time possible. To prevent interference from any moisture formed during reactions, an analytical trap is provided down-stream of the sample holder that can be filled with a desiccant or set up as a coldtrap. For sample calibration, a 500 µl loop is provided, or one of customer specification. If desired, a calibration run can be performed after your sample has been analyzed, automatically. This will assure fast and accurate calculations on all testing parameters from the LabVIEW analysis software.

Computer Control & Data Acquisition

The AMI-300EZ comes with a LabVIEW based software package that automates operation of the system. The operation portion controls and regulates all valve positions, temperatures and detector parameters. Up to 99 methods can be programmed and run-in sequence without the operators' presence. An overview screen shows the status of the unit and provides read outs and position of all valves, flow paths and temperatures. A separate data analysis package performs all calculations for the user, based upon the data from each experiment. Software operations include peak-integration, calculating chemisorptive parameters, peak fit and overlay data. A full report on each run can be printed out.

AMI-300EZ Most Economic Chemisorption Analyzer

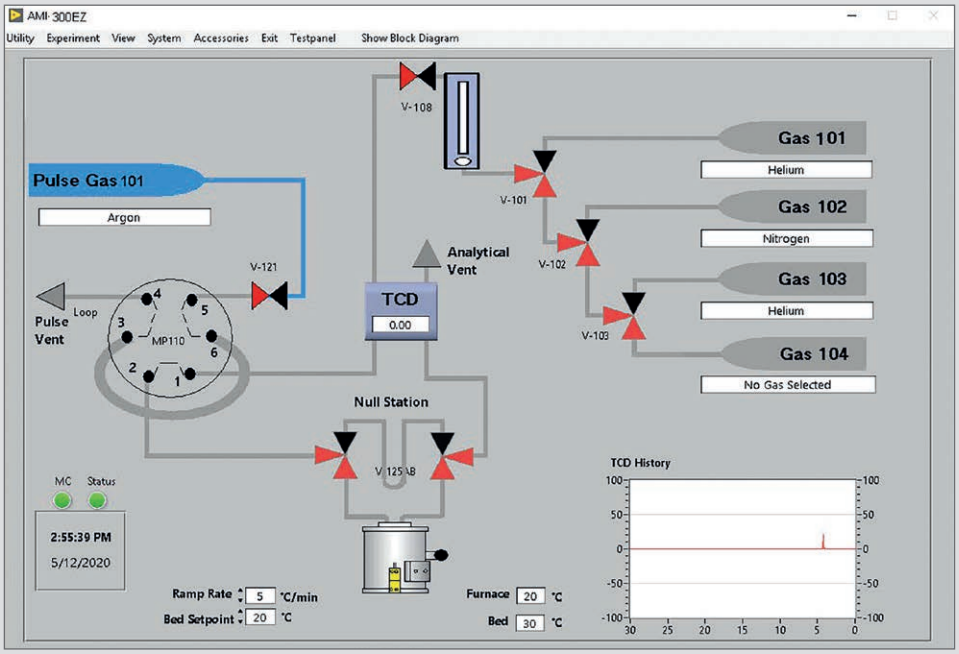


Figure 15 *Operating Screen - A complete overview of all experimental parameters*

Analysis Calculations Include

- Catalyst Uptake
- Crystallite Size
- Consumption
- Percent Dispersion
- Activation Energy of Desorption
- Percent Reduction/Oxidation
- Maximum Desorption Temperature

Specifications	
Catalyst Charge	0.1 – 1 g
Temperature Range	25 – 500 °C; 1,200 °C option
Ramp Rate	1 – 50 °C / min
Operating Pressure	Atmospheric
Gases	5 inputs standard; option for 7
Gas Flow Rates	15 – 150 mL / min Rotameter w / auto shut off
Reactor Types	Quartz u-tubes
Detector	4 filament TCD with choice of filament type (W, Au/W)
Materials of Construction	Stainless steel & Viton seals; option for Buna-N, or Kalrez
Dimensions / Weight	64 cm (W) x 64 cm (H) x 64 cm (D) / 55 kg

µBenchCAT

The µBenchCAT by Altamira Instruments represents a complete, bench-top reactor for catalytic studies. All the components required for either gas-phase or liquid-phase reaction studies are contained in a fully automated compact package. A variety of options makes the µBenchCAT suitable for a wide range of studies.

Hardware

Reactor Feed: Up to 6 gases and 2 liquids can be used in the standard configuration of the µBenchCAT. Each gas train incorporates a filter, electronic mass flow controller (MFC), check valve, and positive shut-off valve. The range and gas calibration of each MFC is specified by the customer. Liquids are delivered using high precision HPLC pumps.

Condenser: A tube-in-tube condenser is located outside the oven at the outlet of the reactor. A thermocouple is used to monitor the coolant return.

Gas/Liquid Separator: The gas/liquid separator is found downstream of the condenser. Two level switches (high and low) activate an automatic valve and serve to maintain level control.

Pressure Control: Exit pressure is measured with a separate pressure transducer and the pressure is controlled using an automatic tapered needle valve.

Product Sampling Valve: An optional product sampling valve can be used to route an effluent sample to an external analytical device.

Heated Oven: Most process components are housed inside an isothermal oven operated at up to 200 °C. The following components are found in the heated oven:

- Integral gas preheater and liquid preheater/vaporizer operating at up to 300 °C
- Feed mixer
- Reactor by-pass valves
- Reactor furnace with measuring and safety-switch thermocouples
- Reactor with internal thermocouple
- Pressure transducer isolator
- Lines leading to pressure transducer and pressure relief valve mounted outside the oven

Computer Control

The µBenchCAT is fully automated for ease of operation and reliability. It is designed so that it can be run unattended. The operator simply inputs process parameters as a series of steps and schedules a start time. Valve positions, flow rates, temperatures, pressures, and product sampling are controlled by the operating software. Data readback of the process are monitored at a rate specified by the user. The data are saved in a text-delimited format for easy transfer to other programs.

The control and data acquisition are performed using a LabVIEW application software specifically written for the µBenchCAT. Complete experiments can be written in minutes and saved for future use.

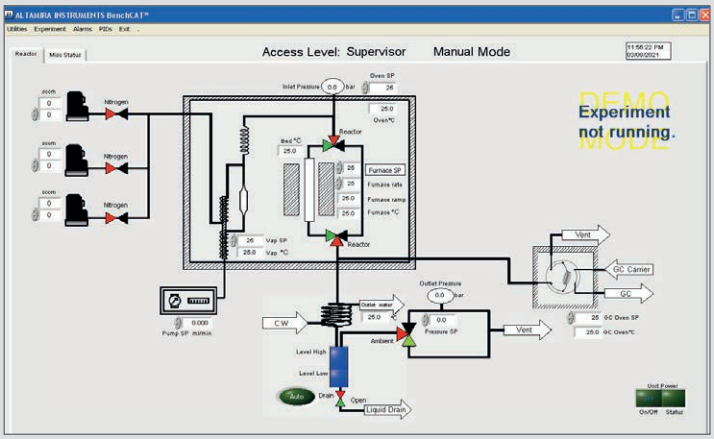


Figure 16 *Main Operation Screen of the µBenchCAT*

Safety

A number of safety features are built into every µBenchCAT. These include:

- Check valves in all gas and liquid feed lines to prevent backflow
- Software-coded alarms that monitor temperatures and pressure for possible excursions. These alarms are based on the equipment safety limitations, and are configured by Altamira Instruments
- User-defined alarm matrix where the user can specify lower and upper limits of process parameters and what actions to take in the event that these limits are reached
- Hardware over-temperature limit switch for the furnace
- Firmware-level alarms on all heaters
- Preset relief valves to prevent over-pressurization
- Power switch mounted on the front of the instrument to control power output to the instrument in case of an emergency
- Double fusing in all 220 VAC process equipment

System Features	
Maximum Operating Temperature	650 °C or 1,200 °C, depending on reactor material
Maximum Operating Pressure	Atmospheric, 30 bar, or 100 bar (higher pressures available)
Number of Gas Feeds	Up to 6
Number of Liquid Feeds	0, 1, or 2
Reactor Material	Stainless Steel, Quartz, or Incoloy
Wetted Materials Include	Stainless Steel, PEEK, Kalrez, Viton, Incoloy, and Quartz
Other Features	■ Isothermal oven housing most process components ■ Full automation of process using LabVIEW ■ Redundant safety thermocouples
Connection to External Detectors	The product effluent can be directed to an external detector, such as a gas chromatograph, via an optional heated or unheated sampling valve.

BenchCAT

The BenchCAT family of instruments are custom-designed microreactors, which are fully automated and designed for unattended operation. All BenchCATs are designed around: number of gas inlets, number of liquid pumps, flow rates, pressure and temperature maximums, reactor material, preference of control software, future upgrade possibilities, etc. BenchCAT microreactors can be designed to conduct iso-thermal or temperature-programmed experiments and analyze the effluent with a mass spectrometer, gas chromatograph, FTIR, or your choice of detector. Single or multi-station instruments are available. Microreactors built specifically for biofuel applications are also part of this line of instruments.

Applications

- Hydrocarbon hydrogenation
- Studies of gas-phase reactions, such as methanol to formaldehyde
- Ethylbenzene dehydrogenation
- Biofuel studies

BenchCAT 1000R HP – Studies of Hydrocarbon Hydrogenation

This unit consists of one fixed bed reactor for a gas phase hydrogenation reaction. The feed module can deliver three gases and one liquid. The reactor tube is stainless steel and can operate at temperatures up to 600 °C at 100 atmospheres. The reactor module uses a three-zone furnace and the flow can be operated in an up-flow or down-flow configuration. The reactor effluent is routed to a gas / liquid separator where analysis can occur. Safety features include a hydrocarbon detector integrated into the computer software prompting error messages, alarms, or automatic shutdown.

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