

ICCS – In-situ Catalyst Characterization System from Micromeritics



Above photo shows the ICCS with the Effi Microreactor from PID Eng. & Tech (a Micromeritics company)

The ICCS is a new instrument that complements the MicroActivity Effi and brings new capabilities to users. The ICCS adds two new capabilities – Temperature Programmed Analyses (TPx) and Pulse Chemisorption. These well-known and time-tested techniques may now be performed on a fresh catalyst and then performed on a used catalyst without the need to remove the material from the reactor. Users benefit from obtaining both temperature programmed analyses and pulse chemisorption data for the same aliquot of sample used for their reaction studies. Performing these analyses in situ virtually eliminates the possibility of contamination from atmospheric gases which may damage the active catalyst.

PRINCIPLE OF OPERATION

The typical operation of the ICCS begins with loading of the catalyst into the reactor. The catalyst may then be characterized by the temperature programmed methods; temperature programmed reduction is commonly used for supported

metal catalysts and temperature programmed desorption may be the best choice for acidic or basic catalysts. The temperature programmed analysis is often followed by pulse chemisorption to determine the number of active sites. This use of TPx and pulse titration provides a description of the fresh (unused) catalyst at reaction conditions (ex. Pressure).

After performing this initial characterization, a user may then proceed with their reaction studies on the exact same material which was previous characterized without the need to add any additional catalyst or transport the catalyst to a different device.

Upon deactivation, the used catalyst may then be analyzed in a similar manner to the fresh material employing temperature programmed analyses and pulse chemisorption at the same reaction pressure. This strategy provides a method for comparing the catalyst and the number of active sites before and after use. This allows the researcher to obtain this critical information without the need to remove the catalyst from the reactor.

PRODUCT DATA SHEET

ICCS – KEY FEATURES & BENEFITS

- *in situ* characterization under reaction conditions (temperature and pressure) of catalysts, catalyst support and a variety of other materials without sample needed to be exposed to the external environment.
- A wide variety of tests can be performed including pulse chemisorption, temperature – programmed reduction (TPR), desorption (TPD), and oxidation (TPO).
- Multiple characterization of the same catalyst may be performed using the same sample.
- A high precision Thermal Conductivity Detector (TCD) to monitor changes in the concentration of gases flowing into and out of the sample reactor.
- The ICCS provides fully automated flow and an additional pressure controller, real-time control.
- Access for the researcher comes via a touch screen with an intuitive graphical user interface for unit control to visualization of each variable.
- A temperature-controlled stainless-steel flow path provides an inert and stable operating environment and reduces potential for condensation in the flow path. Two internal temperature control zones may be operated independently by as much as 180 °C to prevent condensation in the flow path allowing studies to be performed.
- An internal cold trap featuring additional temperature control zone for condensable fluids (ex. water produced during reduction of oxides)
- The ultra-low volume flow path minimizes peak broadening and significantly enhances peak resolution.
- Corrosion-resistant detector filaments are compatible with most commonly utilized TPx and pulse chemisorption gases.
- Interactive peak editor package enables the researcher to conveniently convert data to information. Adjusting peak boundaries is a matter of simply pointing and clicking. This package includes peak deconvolution.

ANALYSIS CAPABILITIES

The production rates of one reaction are well influenced by the active surface and porous structure of the catalyst used on it.

- Metal surface area
- Metal dispersion
- Average active particle size
- Activity, selectivity and deactivation or poisoning
- Temperature-programmed oxidation (TPO)
- Temperature-programmed desorption (TPD)
- Temperature-programmed reduction
- Other applications – Pharmaceuticals (e.g. adsorption of water), building materials, etc.)

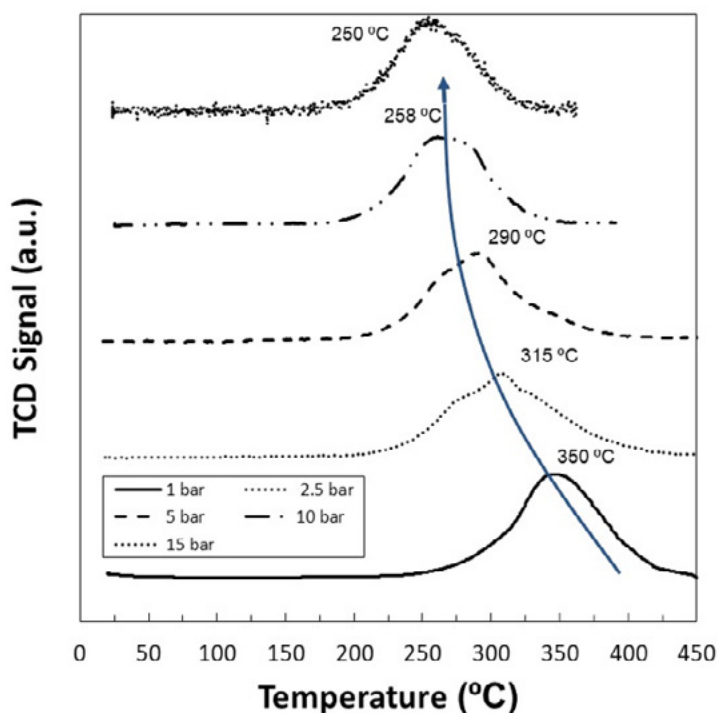


Figure 1: Example data illustrates the impact of increasing pressure upon reduction temperature

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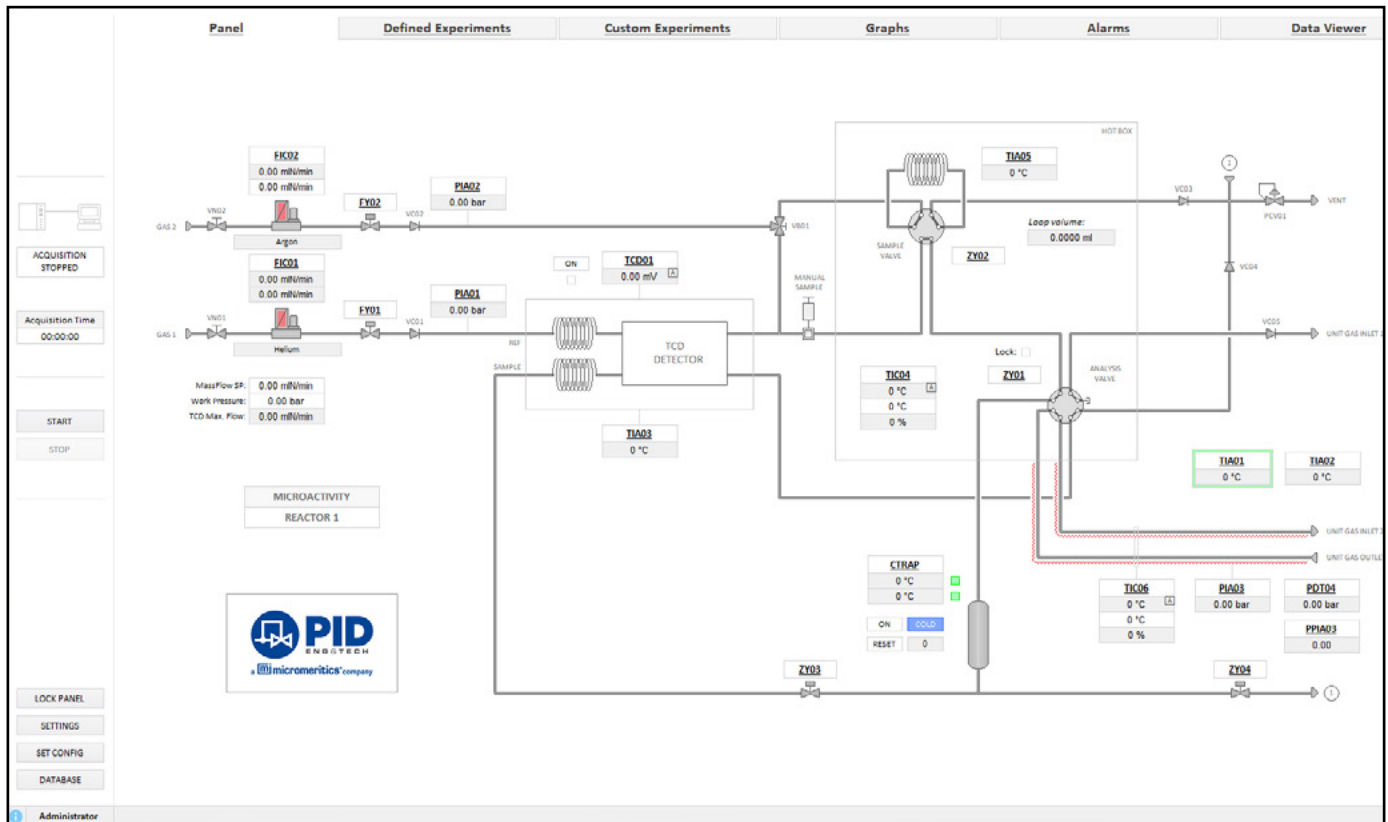


Figure 2: System Diagram

EUROPEAN DIRECTIVES CONFORMITY

PED – Directive 2014/68/UE

Pressure Equipment Directive (PED)

The plant meets Directive 2014/68/UE of the European Parliament on the approximation of the laws of the Member States concerning pressure equipment, and RD 709/2015, passing the applicable regulations relative to the design, manufacture and evaluation of the conformity of pressure equipment, based on said directive.

The equipment will be delivered with the mark according to current regulations.

EMC – Directive 2014/30/UE

Electromagnetic Compatibility Directive (EMC)

Test of EMC immunity according to Standard EN 61326
 Test of EMC emissions according to Standard EN 61326

LVD – Directive 2014/35/UE

Low Voltage Directive (LVD)

Test of electrical safety according to Standard EN 61010-1

ATEX – Directive 2014/34/UE

Equipment and protective systems intended for use in potentially explosive atmospheres (ATEX)

This equipment is excluded and should not be used in potentially explosive atmospheres

RoHS – Directive 2011/65/UE

Restriction of Hazardous Substances

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SPECIFICATIONS

ELECTRICAL

Voltage	240 VAC 10 A, single phase
Frequency	50 - 60 Hz
Power	10 A, single phase

CONTROL MODULE: MINIMUM REQUIREMENTS

Processor	Intel Core I3 or equivalent
Operating Systems	Windows 7/8/10 (32/64 bits)
RAM	4 GB
Hard Drive	500 GB

TEMPERATURE SYSTEM

Valve Box	Up to 180 °C
Heated Line	Up to 180 °C
Cold Trap	From - 18 °C up to 50 °C

OPTIONS

Loop Volume	0.5 cc
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GAS FLOW RATE

Mass Flow Controller 1	
Inlet pressure	30 bar
Flow range	Range 1: 0 - 150 ml/min; Range 2: 150 - 3000 ml/min

GAS DELIVERY

Requirements	Pressure of 30 bar and vent connections with 1/8" connection. Connectors to cylinders are not included, to be provided by the customer
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GAS CONTROL MASS FLOW CONTROLLER

Gas Control	Flow up to 200 cm ³ /min
Mass Flow Controller	± 1% of set-point

PHYSICAL

Height	445 mm
Width	545 mm
Length	500 mm (without computer)
Weight	40 kg (88.2 lbs.)

ENVIRONMENT

Temperature	10 - 35 °C operating
Humidity	10 - 60 % without condensation
Recommendation	Avoid direct sunlight, and direct cool or hot sources