Hiden CATLAB Systems

Microreactor for Catalysis Studies & Thermal Analysis

Hiden CATLAB

An Integrated Microreactor – Mass Spectrometer for Catalyst Characterisation and Evaluation

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Microreactor for Catalysis Studies & Thermal Analysis

Integrated Microreactor – Mass Spectrometer System

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Microreactor for Catalysis Studies & Thermal Analysis

CATLAB Microreactor

- Unique interchangeable catalyst cartridge system.
- 'In-Bed' thermocouple for accurate sample temperature measurement.
- Shut-off / bypass flow configuration to seal reactor and sampling port.
- Air thermostat and trace heating to minimise condensation on inlet and gas sampling connections for use with vapour species.
- Close coupled hot zone evolved species probe.

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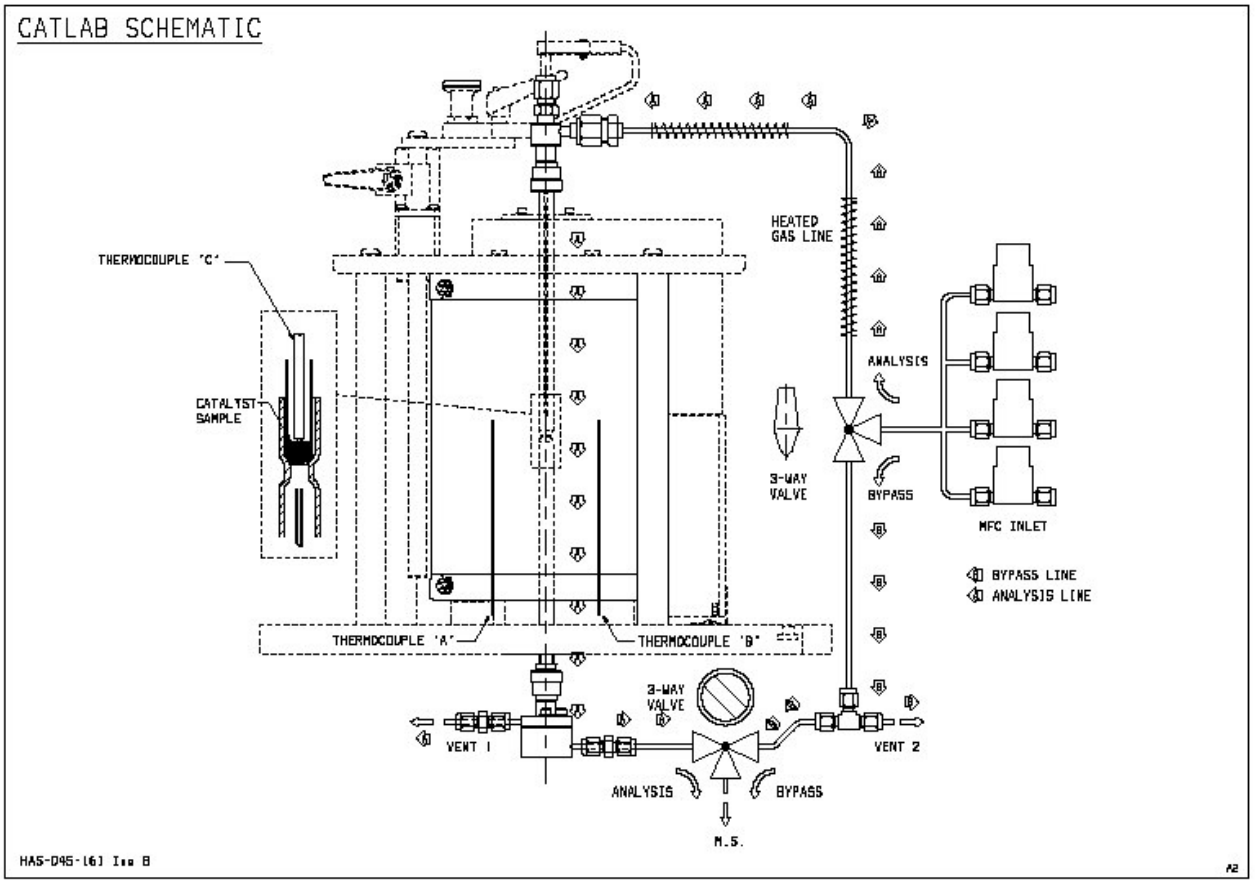
CATLAB Schematic



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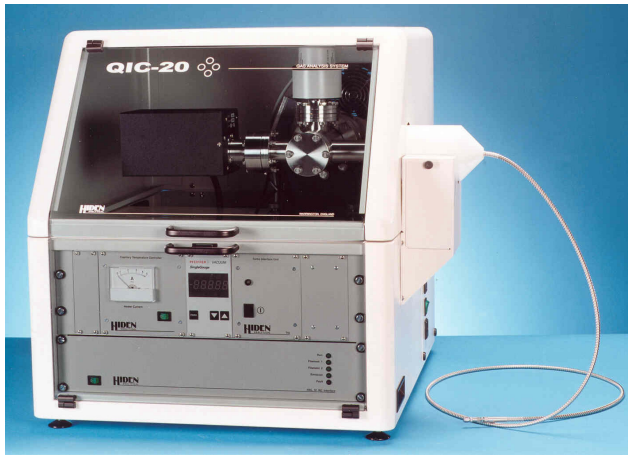
QIC Series Mass Spectrometer



- 200 AMU mass range, dual Faraday/Electron Multiplier detector, with a detection capability from 100% to 0.1ppm.
- Fast scan speeds 100 amu/sec for transient analysis.
- < 500 ms response time to changes in gas concentrations.
- Low dead volume, heated inlet for fast response to vapours.
- Soft ionisation for the analysis of complex organics.

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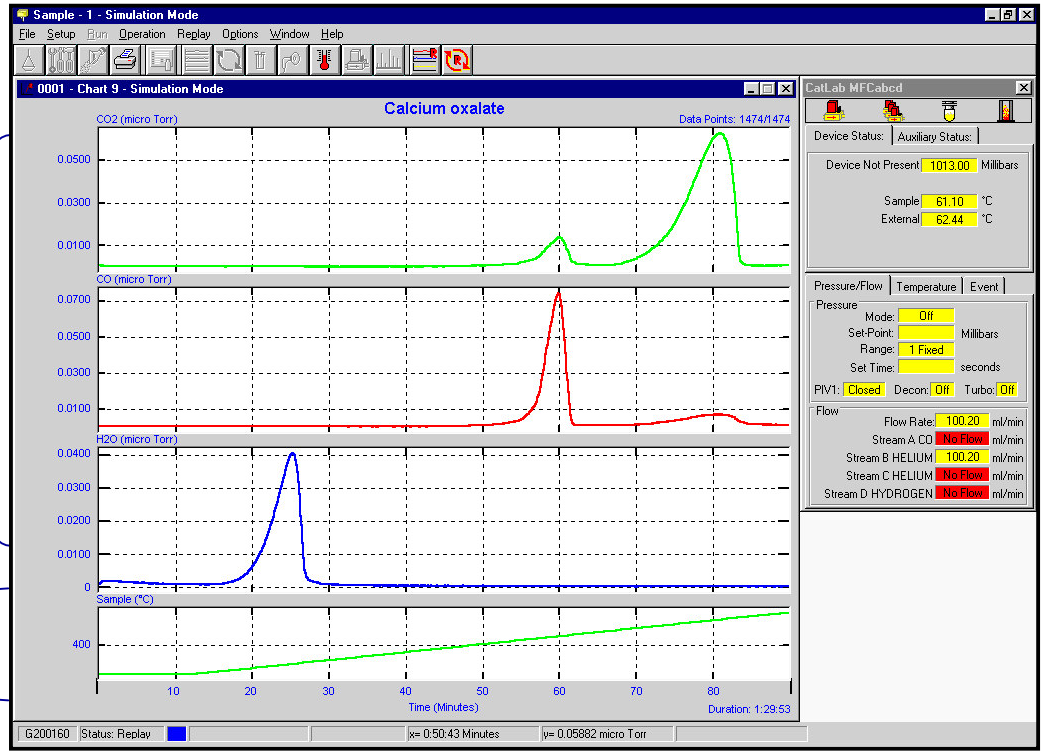
Integrated Software Package

Mass Spectrometer Display

Sample Temperature Display



Gas Flow Monitoring

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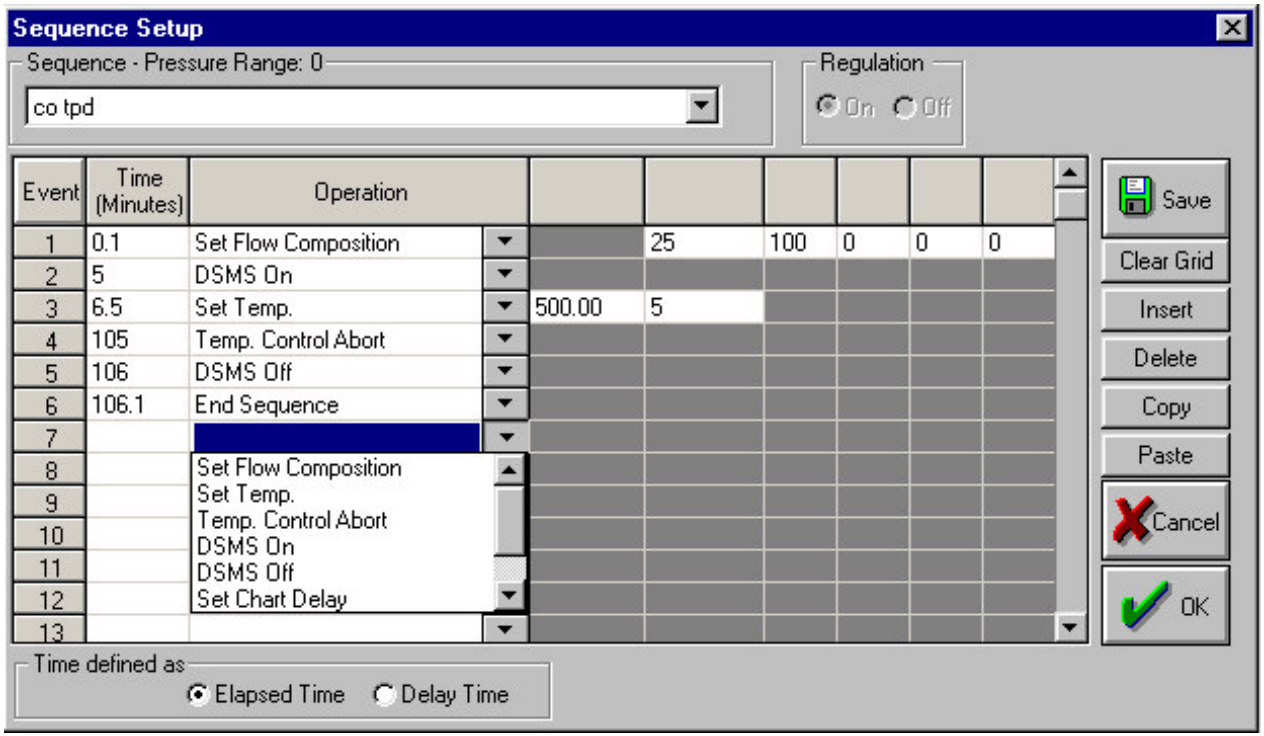
Automatic Experimental Control



- **Fully automated control of experimental procedures using user definable sequences**

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Catalyst Characterisation Using Temperature Programmed Techniques

- **Metal Surface Area / Coverages, (Adsorption/TPD).**
- **Binding Energy of Adsorbed Molecules, (TPD).**
- **Reaction Kinetics (combustion, oxidation, methanation), (TPRx).**
- **Determination of Surface Acidity, (NH_3 or Pyridine TPD).**
- **Reducability of Catalysts, (TPR).**
- **Reaction Mechanisms, (TPRx).**
- **Surface Carbon Deposit Characterisation, (TPO).**

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Detectors for Temperature Programmed Techniques[1]

	Gas Chromatograph (GC)	Thermal Conductivity Detector (TCD)	Quadrupole Mass Spectrometer (QMS)
Advantages	Can be used for more than one desorbing gas. Accurate quantification of evolved gases.	Continuous detection of product.	Continuous detection of selected components. Can detect multiple components simultaneously. Components can be quantified continuously Leaks & impurities easily detected
Disadvantages	Measurements cannot be carried out continuously. "Unknown" products cannot be identified by GC alone.	Can only be used to monitor one component present in carrier gas. Water must be removed before detection.	Cost (?)

1. A. Trovarelli, *6th Italian Chem. Soc. Seminar on Catalysis*, **2001**, 220

Temperature Programmed Desorption (TPD)

- **Identifies Strength, Number and Type of Active Site**
- **Kinetics of Adsorption / Desorption mechanisms**
- **Adsorption / Desorption Activation Energies**
- **Heats of Adsorption**
- **Order of Desorption**

  Zero order: desorption rate independent of coverage

  1st order: desorption rate ∝ instantaneous coverage

  2nd order: desorption rate ∝ (instantaneous coverage)^2

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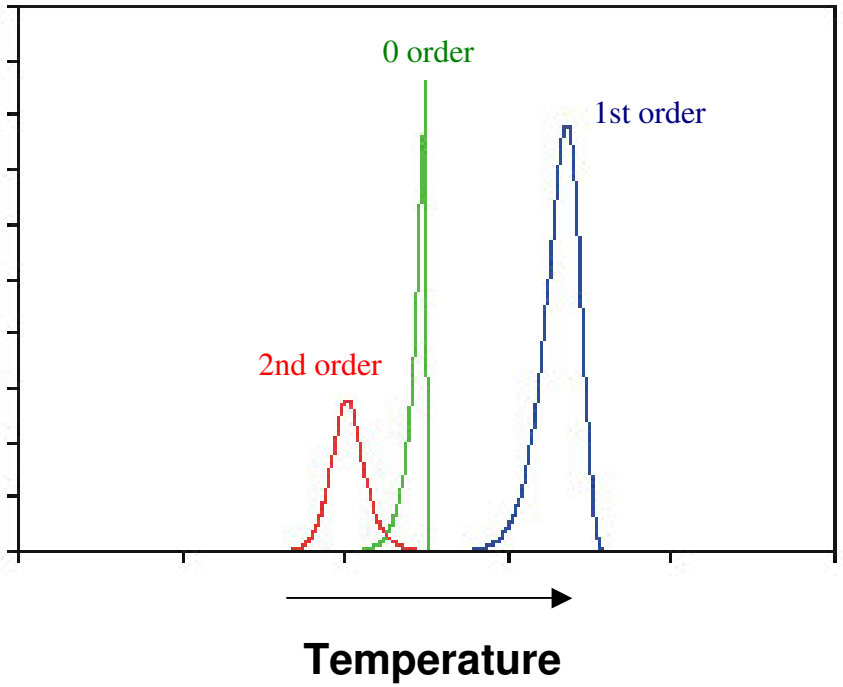
TPD Desorption Order



Zero Order Peaks – Display a leading edge and a sharp cut-off after the peak maxima.

First Order Peaks – Display a leading edge with more gradual decrease than zero order peaks, integration of the 2 peak halves shows a non-symmetrical aspect with an approximate 60:40 split.

Second Order Peaks - Peak is symmetrical about the desorption maximum.

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Temperature Programmed Reduction (TPR)

- **Determination of reducible species on catalyst surface**
- **Involves the reaction of H_2 with material to be reduced**
- **Reaction:**

$$S_{(solid)} + 1\%\ H_2/He \longrightarrow R_{(solid)} + H_2O$$

TPR can also provide:

- **Determination of formal oxidation state of supported metal catalysts or metal ions in zeolites and oxides**
- **Interaction of support with metal**
- **Quantification of spillover phenomena and formation of alloys**

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Temperature Programmed Oxidation (TPO)

- **Utilizes dilute oxygen to oxidise the catalyst surface or carbonaceous deposits.**
- **Study of oxidation processes**
- **Examination of the extent of surface oxidation**
- **Can act as a calcination to remove undesired contaminants which can affect the reactivity of the catalytically active phase.**

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Temperature Programmed Reaction (TPRx)

Can be performed under two conditions

1. **Co-adsorption of reactants before heating in an inert gas.**
2. **A mixture of gases in a carrier gas flow over the catalyst during the temperature ramp.**

Allows:

- **Evaluation of Kinetic parameters**
- **Optimum reaction temperature**
- **Reaction mechanism studies**
- **Deactivation studies**

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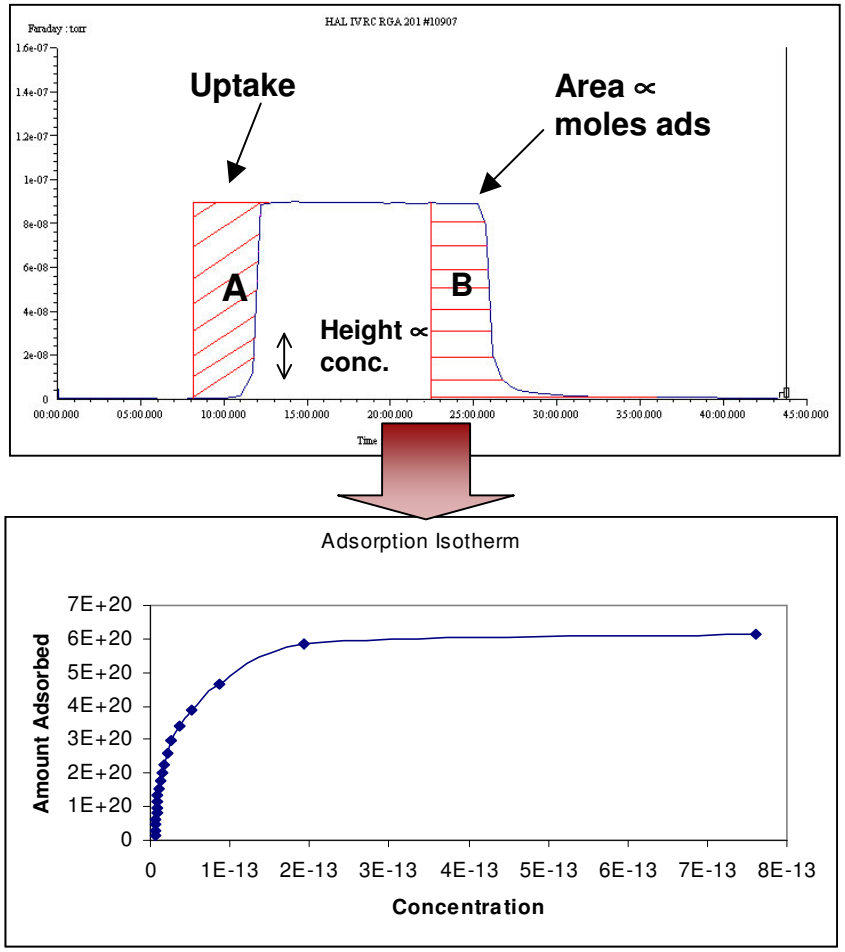
Adsorption Plots



Adsorption isotherms - determines the coverage of an adsorbate at an equilibrium pressure on a surface at constant temperature

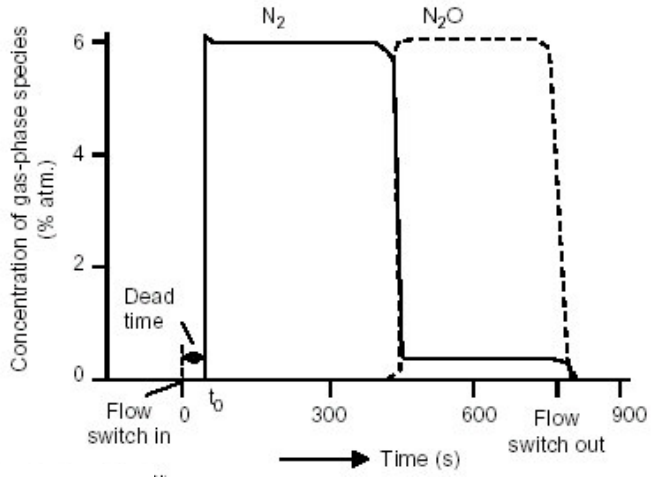
Stripwise Integration of Area B gives Adsorption Isotherm

***e.g.* CO adsorption on Pt/Al_2O_3**

Metal Cu Surface Area Using Frontal Chromatography

Quantification of metallic Cu surface area of reduced Cu-ZnO/Al_2O_3 catalyst by N_2O chemisorption:

$$2\,Cu_{(s)} + N_2O \longrightarrow Cu_2O_{(s)} + N_2$$



Summary

- **The Hiden CATLAB is the ideal choice of the researcher wishing to study catalysts using temperature programmed techniques.**
- **Also allows characterisation data such as metal surface area, adsorption isotherms and coverages to be determined.**
- **System is optimised for maximum mass spectrometer sensitivity.**
- **Integrated software means the mass spectrometer, furnace, gas supply etc. are controlled using only one software package.**

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