Hiden CATLAB Systems

Microreactor for Catalysis Studies & Thermal Analysis

HIDEN ANALYTICAL

vacuum analysis

surface science

gas analysis

plasma diagnostics

CATLAB overview

The Hiden **CATLAB** is a catalyst characterisation and microreactor system designed to make the analysis of catalysts rapid and simple.

CATLAB consists of **two modules:**

- **Module 1:** is the Microreactor including temperature and flow control.
- **Module 2:** is the Hiden Quadrupole Mass Spectrometer system, which can also be used as a stand-alone instrument.

The two modules are complimentary and have been designed to optimise system performance for continuous real time analysis of catalysts and evaluation of multiple reaction components simultaneously. Close-coupled connection means the mass spectrometer inlet is as close to the sample as possible. The result is maximum sensitivity and < 500 millisecond response time.

Accurate synchronisation of mass spectrometer signal with sample temperature is achieved via an integrated I/O subsystem.

Example Data

Typical CATLAB experiments include temperature programmed studies (TPD/R/O etc), reaction testing and pulse chemisorption. Some examples are shown below:

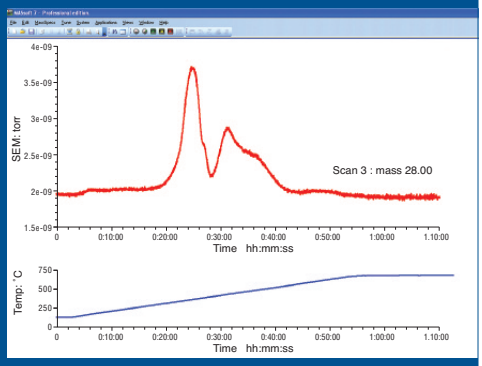


Fig. 1 **Temperature Programmed Desorption**

Temperature Programmed Desorption (TPD).

Figure 1 shows the results of a TPD experiment of CO from a 1% Pd/Al_2O_3 sample. TPD experiments are performed by linearly heating a predosed sample and monitoring the evolved gases.

Key Benefits:

- High sensitivity mass spectrometer. Detection limit 0.1 to 1 ppm subject to spectral interference.
- Close coupled MS for synchronous detection of desorbing gas and temperature measurement.
- MS data and temperature collected in one software package.

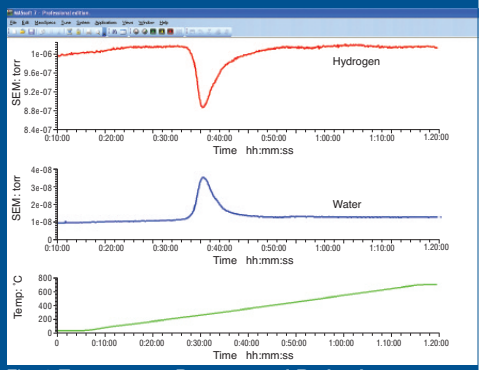


Fig. 2 **Temperature Programmed Reduction**

Temperature Programmed Reduction (TPR).

Figure 2 shows the results of a TPR experiment performed on a CuO sample. TPR experiments involve linear heating of the sample under a reducing atmosphere such as H_2.

Key Benefits:

- Independent foreline and bypass pumps provide optimum performance for applications that use light gases H_2/He etc.
- Excellent H_2 sensitivity – more than x2 sensitivity for H_2 compared with published standard RS factors.
- No need for removal of condensable gases before analysis.

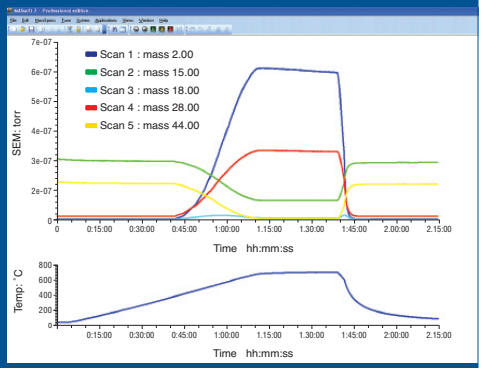


Fig. 3 **Temperature Programmed Reaction**

Temperature Programmed Reaction (TPRx).

The TPRx plot in **Figure 3** shows the results of the conversion of $CH_4 + CO_2 \rightarrow 2H_2 + 2CO$ over a Ni catalyst during a linear temperature ramp to 700ºC.

Key Benefits:

- Unlimited number of masses can be measured simultaneously.
- Heated inlet for sampling of condensable gases, e.g. H_2O vapour.
- Soft Ionisation mode for simplified spectra of complex molecules.

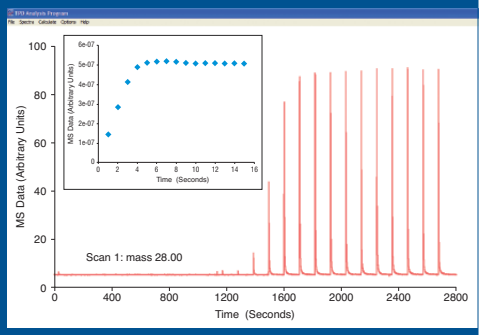


Fig. 4 **Pulse Chemisorption**

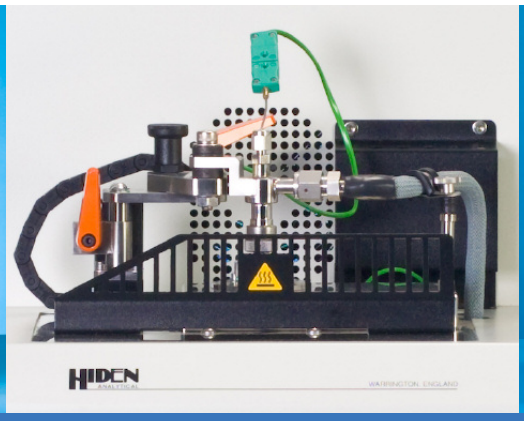
Pulse Chemisorption.

The pulse experiment shown in **Figure 4** was performed over a 5% Pd/Al_2O_3 catalyst. The sample was dosed with multiple pulses of CO until saturation was achieved.

Key Benefits:

- Fast data acquisition speeds > 500 measurements/s.
- Minimal internal volumes reduce peak spreading.
- < 500 ms response time to changes in gas concentrations.

CATLAB



CATLAB Technical Specifications

module 1

sample mass	typically 25 - 250mg
	up to 2.0g optional
pressure	up to 1 bar
temperature	ambient to 1000°C
accuracy	+/- 1°C
ramp rate	1 to 20°C/min
temperature sensor	type K thermocouple
mass flow controllers	4 streams 3-100ml/min standard
	up to 8 streams with user defined
	flow rates optional
minimum flow pressure	3 bar
port connection	1/8 " Swagelok
power requirement	100-240V AC, 50-60Hz, 1.0kVA

module 2

mass range	standard 200 amu. options 300 or 510 amu
ion source	direct inlet high pressure source
ion source control	all parameters adjustable in real time
detector	dual faraday cup / channeltron electron multiplier
detection limit	5×10^{-11} torr with faraday cup detector
	2×10^{-14} torr with channeltron detector
gas sensitivity	krypton (^{84}Kr) in air at 0.5 ppm with faraday detector
	xenon (^{129}Xe) in air at 25 ppb with channeltron detector
response speed	from sample to QMS, less than 500ms
analyser bakeout	250°C
quartz inlet capillary	typical inlet flow rate/gas consumption 20 atm ml/min
	low flow rate versions to 1 atm ml/min available
power requirement	100-240V AC, 50-60Hz, 1.5kVA

Further system options

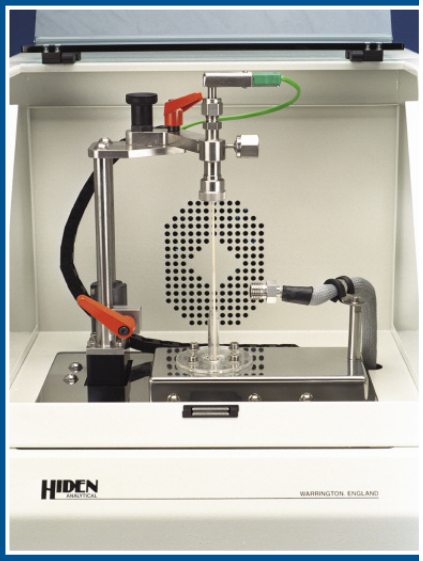
- Corrosion resistant upgrade for Modules 1 & 2.

- Vapour generator.

CATLAB technology

Advanced features make the Hiden **CATLAB** the instrument of choice. All system elements are designed and integrated to ensure the maximum reproducibility of results.

- A single integrated software package allowing manual or automated control over both the CATLAB microreactor and mass spectrometer parameters such as temperature ramp rates and set points, flows, mass detection.
- Quartz Catalyst Cartridge System for reproducible sample positioning.
- Low Thermal Mass Furnace for rapid linear response.
- Sample 'In-bed' Thermocouple for optimum temperature accuracy.
- Precision Mass Flow Controllers for accurate flow measurement.
- Zero Dead volume valves ensuring rapid, reproducible response.



- Pulse chemisorption option for uptake measurements, adsorption isotherms and catalyst dispersion.
- Fully programmable and automated analysis cycle.
- Data analysis software packages.

CATLAB characterisation

Catalyst Characterisation is performed using both Temperature Programmed (TPD, TPO, TPR, TPRx) and isothermal techniques. These techniques allow a whole range of parameters to be characterised with one system. Information obtained using these techniques include:

- Metal surface area
- Surface coverage
- Determination of strength / number of active sites
- Adsorption isotherms

CATLAB

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