HIDEN ANALYTICAL

CATLAB

Microreactor for Catalysis Studies & Thermal Analysis

Detailed product information / introduction

A combined bench-top microreactor and mass spectrometer system for rapid and reproducible catalyst characterisation and reaction studies.

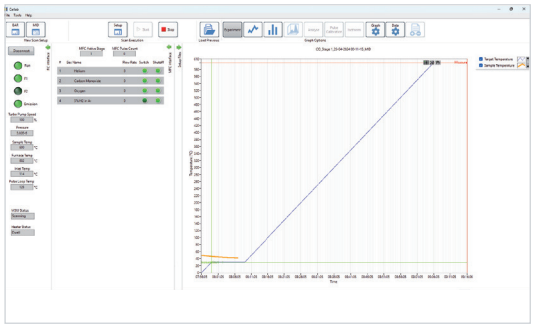
Applications:

- TPD/TPO/TPR/TPRx
- catalysis
- catalyst screening
- pulse chemisorption
- pulse calibration
- dispersion measurements
- adsorption isotherms
- reaction studies

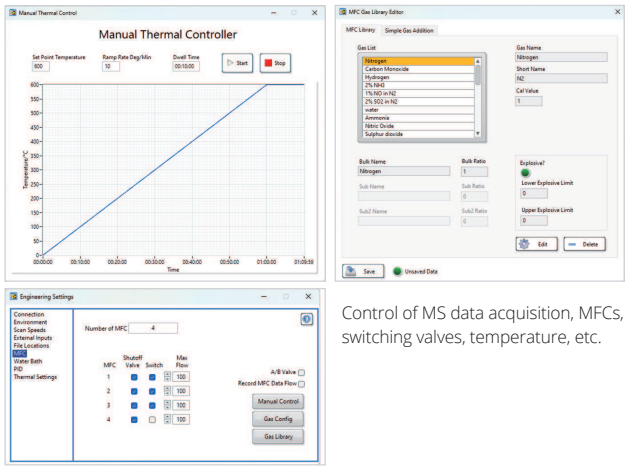
Key Features

- Fast response, low thermal mass furnace with integrated air-cooling
- 1-20°C/min heating rates up to 1000°C
- Hiden's unique interchangeable Catalyst Cartridge System
- Close-coupled hot-zone evolved species probe & direct QIC inlet for <500 ms response
- 'In-Bed' thermocouple for direct catalyst temperature measurement
- Automatic 4 (8 optional) gas stream manifold or combined gas/vapour manifold
- Independent Mass Flow control of each channel at 3-100 ml/min (other flow options available)
- Corrosion resistant manifold configurations also available
- Seamless on-line, real-time studies with data acquisition and PC analysis
- Air thermostat and trace heating to minimise condensation of vapour feeds/products
- Shut-off/bypass flow configuration to seal reactor and gas sampling port
- Vapour generator option for the introduction of vapours such as water, etc.

Software



CATLAB software



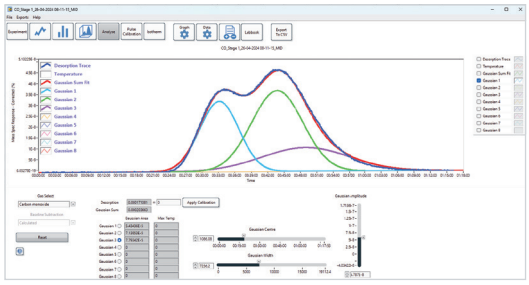
Control of MS data acquisition, MFCs, switching valves, temperature, etc.

CATLAB Software

A dedicated application specific software package for automated control of MS data acquisition fully integrated with all necessary devices including MFCs, furnace temperature, switching valves, pulse injection, etc.

Key Features – CATLAB Software

- Control of the experiment following a user defined temperature profile
- MS data, temperature and flow all collected in one software package
- Data analysis routines included in the software for determination of catalyst properties such as metal surface area, dispersion, pulse adsorption isotherms, etc.
- 10 peak spectral library
- Automatic subtraction of spectral overlaps
- Analysis of up to 16 gases



Peak integration/ deconvolution

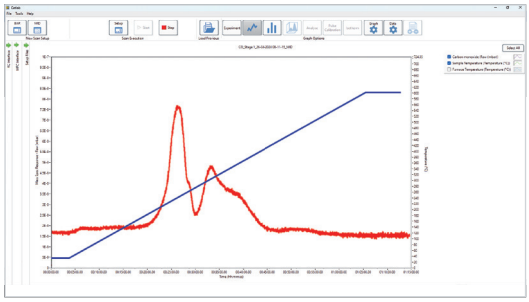


Fig. 1. Temperature Programmed Desorption

CATLAB Example Data

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

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 100% to 100 ppb 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.

Software (continued)

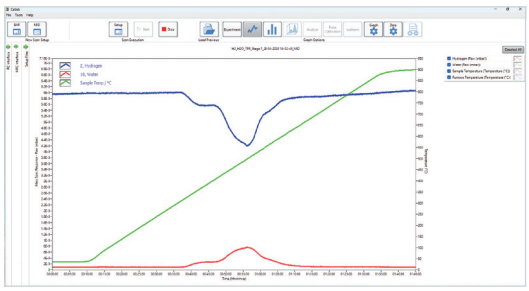


Fig. 2. Temperature Programmed Reduction

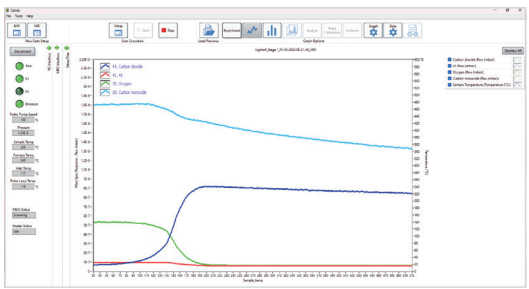


Fig. 3. Temperature Programmed Reaction

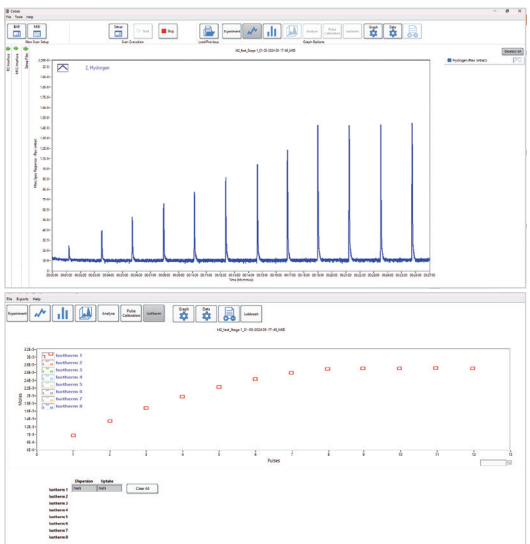


Fig. 4. Pulse Chemisorption

CATLAB Example Data (continued)

Temperature Programmed Reduction (TPR)

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

Key Benefits:

- High capacity dry pumping system provides 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.

Temperature Programmed Reaction (TPRx)

The TPRx plot in Figure 3 shows the results of the CO oxidation light-off test ($2CO + O_2 \rightarrow 2CO_2$) during a linear temperature ramp to 500°C.

Key Benefits:

- Simultaneous measurement of up to 16 gases/mass species.
- Heated inlet for sampling of condensable gases, e.g. H_2O vapour.
- Soft Ionisation mode for simplified spectra of complex molecules.

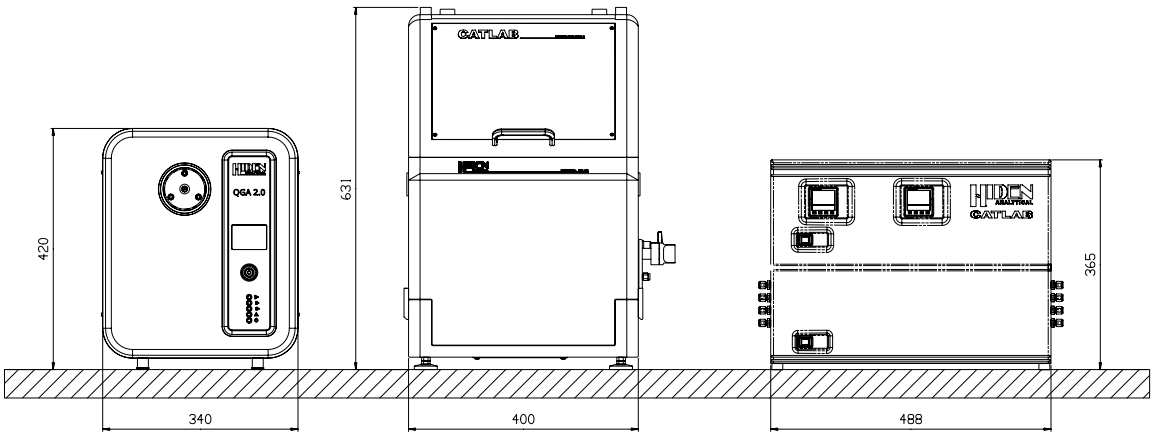
Pulse Chemisorption

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:

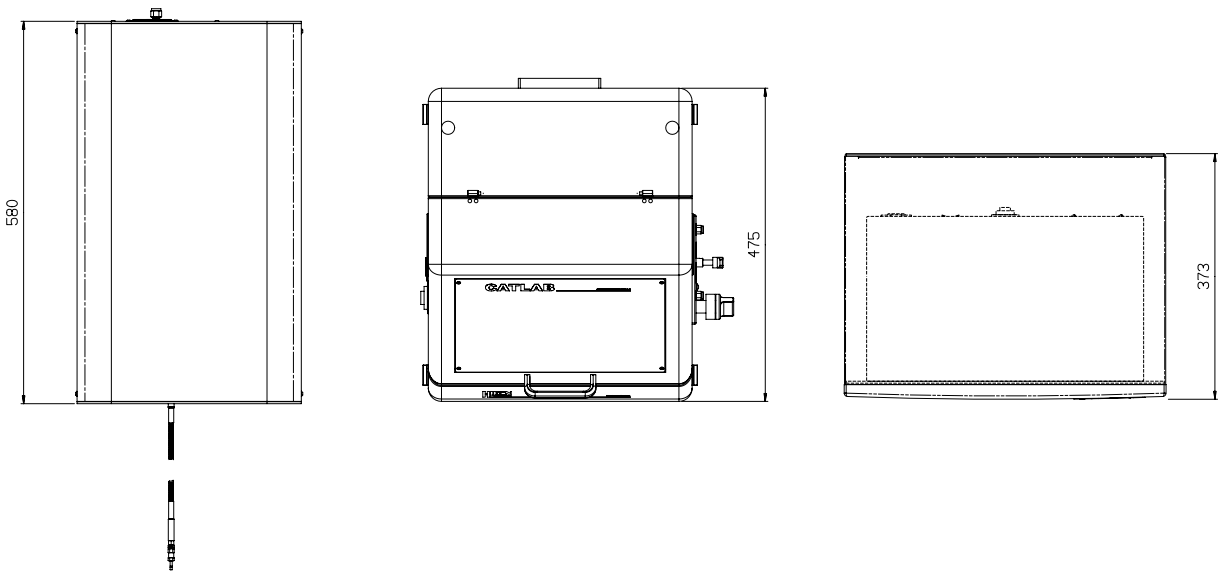
- Fast data acquisition speeds > 500 measurements/s.
- Minimal internal volumes reduce peak spreading.
- < 500 ms response time to changes in gas concentrations.

Technical Data



CATLAB MODULE 1	TECHNICAL SPECIFICATIONS
Sample mass:	Typically 25 - 250 mg (up to 2.0 g optional)
Reactor sizes (ID):	4, 7 or 12 mm
Pressure:	Up to 1 bar
Temperature:	Ambient to 1000ºC
Accuracy:	+/- 1ºC
Ramp Rate:	1 to 20ºC/min
Temperature sensor:	Type K thermocouple
Mass flow controllers:	4 streams 3-100 ml/min standard up to 8 streams with user defined flow rates optional
Minimum flow pressure:	3 bar
Port connection:	1/8¨ Swagelok
Software	CATLAB Software Windows compatible
Dimensions (L x W x H):	Microreactor: 631 x 400 x 475 mm Electronics Unit: 365 x 488 x 373 mm QGA 2.0: 580 x 340 x 420 mm
Weight:	Microreactor: 30 kg Electronics Unit: 27kg QGA 2.0: 29.5kg – 33.5kg, depending on configuration, Scroll pump 26kg
Power requirement:	100-240 V AC, 50-60 Hz, 1.0 kVA

Technical Data (continued)



CATLAB MODULE 2	TECHNICAL SPECIFICATIONS
Mass range, amu:	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 / Channeltron electron multiplier
Detection limit:	5×10^{-11} torr with Faraday 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 time:	300 ms
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:	100-240 V AC, 50-60 Hz, 1.5 kVA
	FURTHER SYSTEM OPTIONS
	Corrosion resistant upgrade for Modules 1 & 2 Vapour generator

System Configuration & Options

ITEM	DESCRIPTION	PARTCODE
SYSTEM	**CATLAB-PCS Module 1: The Microreactor System** A Quartz Microreactor with integrated low thermal mass, air-cooled radiant furnace and close-coupled QIC sampling. This item includes the CATLAB-PCS Windows® software and computer control of temperature (ramp/set-point) and flow. Low thermal mass furnace for linear ramp control and set-point regulation to 1000°C: Includes a high temperature quartz sample reactor for operation to 1 bar with high temperature sensors and interface.	303230
	CATLAB-PCS Module 2: The QGA 2.0 Mass Spectrometer A QGA 2.0 bench-top gas analysis system, including Hiden HAL 201 RC mass spectrometer with dual Faraday/Channeltron Electron Multiplier detector. Mass range 200 amu. Includes external scroll pump. QGA 2 & MASsoft Professional software packages included as standard. Includes standard QIC capillary inlet for operation up to 200°C	305110
OPTIONS & ACCESSORIES	Corrosion resistant upgrade	303250
	Corrosion resistant upgrade - additional feed stream	303255
	8 Flow Stream option. Additional 4 channel MFC flow control unit integrated to provide 8 stream gas selection.	303258
	Vapour feed system	303259
	Extended mass range. 300 amu mass range (in place of standard 200 amu mass range)	305113
SPARES KIT	**Mass spectrometer spares** QGA 2.0 Mass Spectrometer Spares kit including: ▸ Replacement capillary liner ▸ Replacement capillary leak orifice ▸ Twin filament	303148
	Microreactor spares	
	CATLAB module 1 maintenance kit. Includes replacement 'O' ring seals and tools	3101210
	CATLAB reactor tube. Precision quartz reactor tube assembly with integral gas sampling tube	3101012
	CATLAB sample tube kit. Includes 6 sample tubes, preparation tool, quartz wool and 'O' ring	3101016
	PCS – Additional valve sample loop	303260

Hiden**APPLICATIONS**

Hiden's quadrupole mass spectrometer systems address a broad application range in:

GAS ANALYSIS

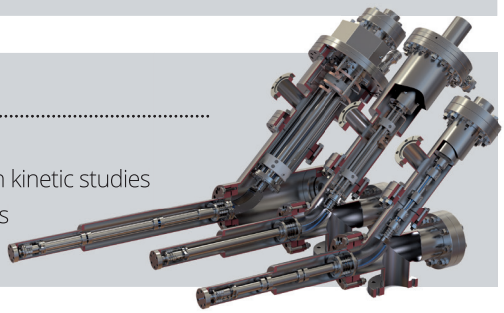
- dynamic measurement of reaction gas streams
- catalysis and thermal analysis
- molecular beam studies
- dissolved species probes
- fermentation, environmental and ecological studies

SURFACE ANALYSIS

- UHV TPD/TDS
- ToF qSIMS and SIMS analysers
- end point detection in ion beam etch
- elemental imaging – 3D mapping
- SIMS system with simultaneous dual polarity analysis

PLASMA DIAGNOSTICS

- plasma source characterisation
- etch and deposition process reaction kinetic studies
- analysis of neutral and radical species



VACUUM ANALYSIS

- partial pressure measurement and control of process gases
- reactive sputter process control
- vacuum diagnostics
- vacuum coating process monitoring

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