



Mass Spectrometers for Catalysis & Thermal Analysis Applications

MASS SPECTROMETERS for Catalysis & Thermal Analysis Applications

Hiden Analytical have been designing and developing the highest quality quadrupole mass spectrometer based gas analysis systems for over 30 years. We have built a reputation for delivering instruments with superior sensitivity, accuracy and reproducibility together with a first class global service and applications support network. From dedicated triple filter UHV TPD quadrupoles to fully integrated catalysis microreactor and mass spectrometer systems, Hiden have developed a range of analytical mass spectrometers that address the most advanced and demanding applications.



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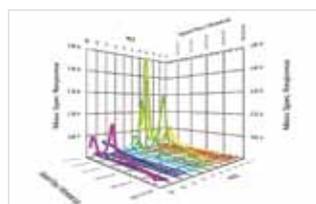
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CATLAB data display

CATLAB

Automated Microreactor/MS System

The Hiden CATLAB is a bench-top microreactor and combined mass spectrometer system for rapid and reproducible catalyst characterisation and reaction studies. The microreactor and mass spectrometer are delivered as a complete system from a single manufacturer, unique to the industry, ensuring optimum analysis via seamless hardware and software integration. The modular design further allows both instruments to operate as stand-alone components so that they may be interfaced with existing laboratory equipment such as TGAs (TG-MS) or Gas Chromatographs.

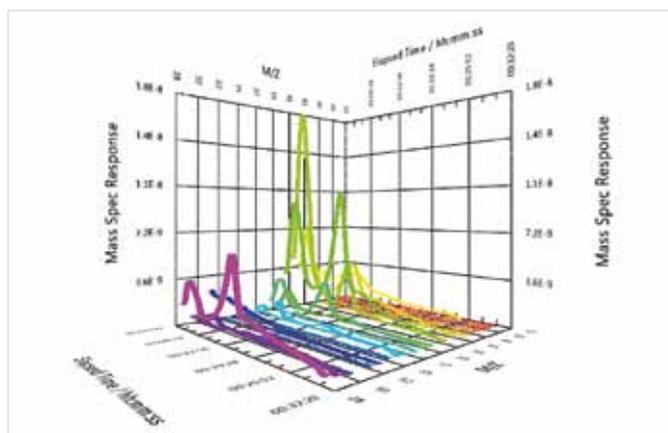
The Hiden CATLAB delivers a range of completely automated, dynamic, temperature programmed, pulse chemisorption and isothermal techniques. Template driven CATLAB software provides automatic control of gas composition and delivery, temperature ramp and set-point as well as full mass spectrometer parameter controls – a true first for the catalyst researcher. The standard system has 0-200 amu capability with options extending this to 1000 amu.

APPLICATIONS:

- ▶ temperature programmed desorption (TPD)
- ▶ temperature programmed reduction/oxidation (TPR/O)
- ▶ temperature programmed reaction (TPRx)
- ▶ pulse chemisorption
- ▶ pulse calibration
- ▶ dispersion measurements
- ▶ adsorption isotherms
- ▶ reaction studies



CATLAB microreactor module



CATLAB data display

CATLAB-FB

Horizontal Furnace/MS System for Catalyst Core Quantification



CATLAB-FB

The CATLAB-FB is a combined furnace and mass spectrometer system for the analysis of catalyst core specimens at temperatures up to 1000°C, with multi-stream gas/vapour flow control and mass spectral analysis of both feed and downstream gases.

The furnace houses a horizontal quartz reactor tube with in-bed thermocouple for optimum thermal precision, a standard internal diameter of 26 mm and a uniform central hot-zone of 150 mm. The furnace is programmable from 50°C through to 1000°C at a fastest ramp rate of 20°C/minute. The system has capacity for up to 12 mass flow controlled gas streams, selectable with flow rates from 0.1-10 L/minute and for operation with corrosive feed gases.

Options also includes a pulse chemisorption (PCS) mode for injection of sorbates to the reactor and a vapour feed system to enable vapour reaction characterisation in both PCS and continuous flow modes. All system elements are programmable from the integrated control program together with mass spectrometer data acquisition, data display and data interpretation.

There is also the possibility that a customer supplied furnace can be integrated into the CATLAB-FB set-up.

OPTIONS:

- ▶ temperature range up to 1000°C
- ▶ heated length: ranges from 130 to 1300 mm
- ▶ tube diameters: 20 to 170 mm
- ▶ horizontal or vertical configurations
- ▶ heated reactor bypass option
- ▶ the inclusion of up to 12 MFCs, corrosion resistant options available
- ▶ thermocouple inputs: options for 8 or 16

QIC SERIES

for Gas Analysis

BASELINE SYSTEM FEATURES INCLUDE:

- ▶ Sample pressure range 2 bar to 100 mbar
- ▶ Continuous sampling with inlet flow rate configurable down to 1 ml/min
- ▶ Low dead volume, heated inlet for fast response to vapours
- ▶ Species molecular weight range to 200 amu
- ▶ Fast data acquisition speeds > 500 readings/second in transient mode
- ▶ Fast 300 ms sampling response
- ▶ Time/intensity trend monitoring of multiple species
- ▶ APSI-MS soft ionisation mode for suppression of spectral fragmentation providing simplified analysis of complex mixtures
- ▶ Integration of external process data (temperature, weight, pressure)

SYSTEM OPTIONS INCLUDE:

- ▶ Species molecular weight range to 300, 510 or 1000 amu
- ▶ Special gas sampling interface options for high pressure, high temperature applications
- ▶ Multi-stream selectors - up to 80 stream versions available
- ▶ Application specific software:
 - **QGA for quantitative gas analysis** - included as standard with QGA systems
 - **EGAsoft for evolved gas analysis** - included as standard with HPR-20 QIC EGA systems



QGA - bench-top gas analyser



HPR-20 QIC R&D



HPR-20 QIC R&D^{PLUS}

HPR-20 QIC TMS

Transient MS for Fast Event Gas Analysis



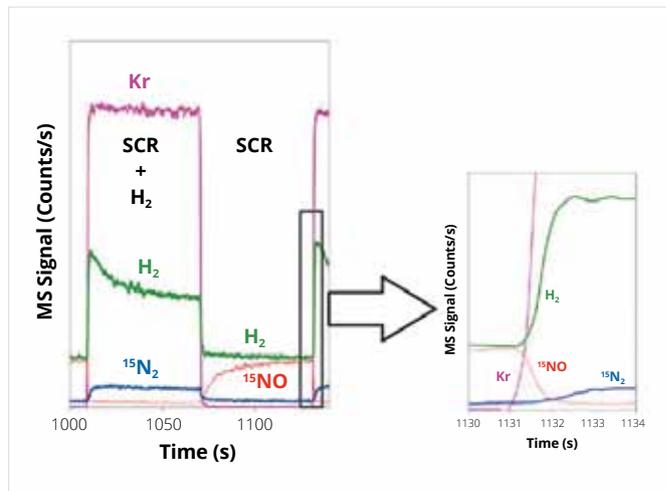
HPR-20 QIC TMS transient MS QIC inlet

The Hiden HPR-20 QIC TMS Transient MS is configured for fast event gas analysis of gases and vapours at pressures near atmosphere. Ideal for fast gas switching experiments, the MS features the Hiden QIC quartz-lined 0.9 m sampling interface. The inlet, operating at 200°C, provides response times of less than 150 ms to changes in gas composition with a 5 decade response time in < 100 ms.

The QIC inlet is coupled directly to Hiden's Pulse Ion Counting (PIC) digital MS which is capable of measurement speeds of up to 500 data points/s over the entire 7 decade dynamic range.



HPR-20 transient MS data - > 5 decades response in < 100 ms



Mass spectrometer response for $^{15}\text{N}_2$, ^{15}NO , H_2 and Kr when switching 0.72% H_2 in and out of a SCR (NO_x with octane) feed stream over a catalyst at 300 °C (J. P. Breen, R. Burch, C. Hardacre, C. J. Hill and C. Rioche, J. Catal., 2007, 246, 1-9).

FEATURES:

- ▶ 0.9 m fast response QIC capillary - 150 ms response time
- ▶ open ion source and optimised pumping configuration for fast response
- ▶ digital PIC detector - 7 decades continuous log scale
- ▶ detection of low ppm to high % levels in < 100 ms

TA-MS for Evolved Gas Analysis

The Hiden HPR-20 QIC EGA gas analysis system is configured for continuous analysis of evolved gases and vapours from thermogravimetric analysers (TGA). Custom designed interfaces are available for special requirements with alternative systems being offered for applications requiring direct sampling from advanced thermogravimetric analysers operating at higher pressures of up to 30 bar.

All of Hiden's QIC series gas analysers may additionally be equipped with fast response, low dead volume interfaces for the most popular TGA equipment. Each interface has been custom engineered in collaboration with TGA manufacturers (e.g. PerkinElmer, TA Instruments, Mettler Toledo, Setaram, Netzsch etc.) and includes, where necessary, robust clamping arrangements and in-line heated filter assembly between the outlet of the TGA and the MS capillary inlet.

EGAssoft is an application specific software package for use in TA-MS applications with features such as 3D bar scan view mode, export to specific file types for import to TGA manufacturers' software and NIST export for database searching of unknowns.



HPR-20 QIC EGA

FEATURES:

- ▶ minimum dead volume
- ▶ controllably heated sample inlet – no cold spots
- ▶ inert materials
- ▶ high performance gas handling for operation with low molecular weight gas components (H_2 , He) and for flow matching with the TGA



TGA-MS Interface

SpaciMS

Spatially Resolved Capillary Inlet MS

The Hiden Analytical spatially resolved capillary inlet MS (SpaciMS) is the first commercially available instrument of its kind. Recipient of a R&D 100 award, the SpaciMS inlet was originally conceived and developed by researchers at the Oak Ridge National Laboratory and Cummins, Inc. to study diesel catalysis¹ and has been further developed for a whole range of applications.

SpaciMS allows both radial and axial species determination and temperature profiles, with high spatial and temporal resolution and with negligible interference in flow or temperature. The 16 channel multi-inlet is coupled to Hiden's fast transient MS (HPR-20 QIC TMS) to provide automatic and rapid mapping of temperature and species distributions.

Up to 16 capillary sampling probes and thermocouples are arranged in an X-Y array. A Z-shift provides movement and accurate positioning of the array in the Z plane. In usual practice, the 16 capillary sampling probes are sequentially analysed by the MS. The Z-shift is then actuated to move the sampling probe array to the next incremental Z position and the analysis sequence is repeated. On completion the analytical data provides a spatial representation of temperature and sample gas composition of the volume enclosed within the X-Y array over the total incremental Z distance moved.

[1] Partridge, W.P. et al. 2000 *Journal of Fuels & Lubricants* **109** 2992-2999

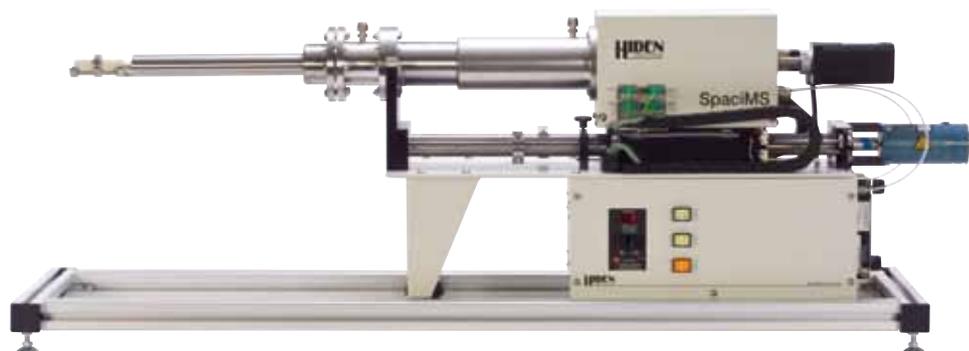


Intra-catalyst sampling of gas and temperature



SpaciMS sample holder

SpaciMS



APPLICATIONS:

- ▶ fuel cell studies
- ▶ diesel engine catalysts
- ▶ air exhaust mixing systems
- ▶ non-thermal plasma reactors

FEATURES:

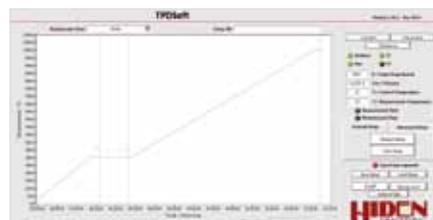
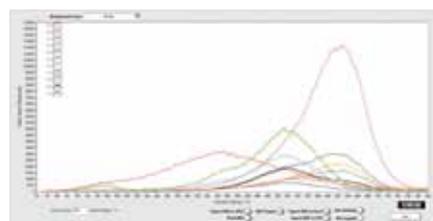
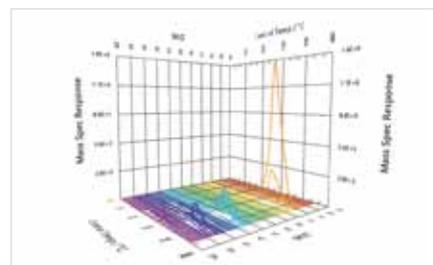
- ▶ quantifies intra-catalyst-channel species transients and distributions
- ▶ high temporal resolution
- ▶ minimally invasive

TPD WORKSTATION

A System for UHV Temperature Programmed Desorption (TPD/TDS) Studies

The Hiden TPD Workstation is a complete experimental system for analysis of thermal desorption products by UHV TPD/TDS. The TPD Workstation features a multiport UHV chamber with heated sample stage coupled to a high precision triple filter analyser with digital pulse ion counting detector for ultimate sensitivity and time resolution. The triple filter mass analyser is configured with a cooled shroud giving minimum outgassing with optimum sensitivity of the analyser to desorption products from the sample. A fast sample load lock with sample transfer mechanism is included to provide for rapid sample change. The unique sample transfer mechanism means only the sample is transferred from the load lock to the heater stage ensuring no sample holder outgassing during the TPD experiment.

Hiden's TPDsoft thermal analysis PC software included with the Workstation provides automatic control of sample temperature integrated with analyser control. TPD analysis routines (e.g. peak integration, deconvolution and background subtraction etc.) are also included in this package.



TPD Workstation



TPD Workstation with hot sample

TPDsoft for control and analysis of TPD experiments



FEATURES:

- ▶ Hiden 3F PIC mass spectrometer for fast data acquisition (> 500 data points per second)
- ▶ multiport UHV chamber for attachment of additional instrumentation (e.g. ellipsometry)
- ▶ sample transfer mechanism and load lock, including gate valve and viewport
- ▶ heated sample stage to 1000°C
- ▶ Z-drive for optimum sample/detector positioning
- ▶ bakeout jacket (200°C max)
- ▶ integrated software control of experimental protocols

UHV TPD

for Fast Event UHV Studies

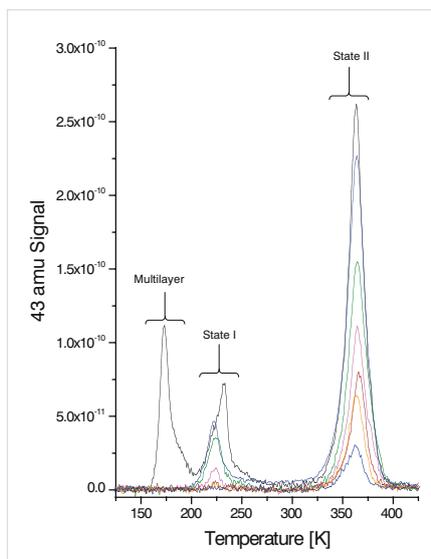


3F PIC

Hidden's 3F PIC Series Quadrupoles are high precision triple filter analysers with digital detectors for ultimate sensitivity and time resolution in fast event studies such as UHV TPD. 3F Series analysers are available with UHV compatible mass filter shrouds and low profile ion source for close positioning to the desorption surface.

Both analogue and digital inputs are provided for synchronous acquisition start and sample temperature data display alongside mass channel data.

Application specific software and external device program protocols (RS485, Modbus) enable the user to control temperature ramp profiles and collect data in the same program (TPDsoft) or to simply collect MS data and temperature in the same program (EGAssoft).



TPD Plot courtesy of M. Kadodwala (University of Glasgow, UK)



Range of Shrouds

FEATURES:

- ▶ low profile ion source
- ▶ fast data acquisition
- ▶ > 500 data points per second
- ▶ wide dynamic range
- ▶ 7 decade continuous log scale
- ▶ gating input for pulsed gas studies down to 100 ns gating resolution

HPR-60 MBMS

Molecular Beam Sampling Mass Spectrometer

The Hiden HPR-60 molecular beam mass spectrometer is a compact skimmer inlet MS for the analysis of reactive gas phase intermediates. Radicals are sampled via a multi-stage differentially pumped skimmer inlet and transferred to the MS ion source with minimal interaction with other species and without wall collisions. Customisable inlets allow connection to many different reactor systems, including atmospheric plasmas.

The skimmer system, combined with a Hiden triple filter precision mass spectrometer, offers a sampling system with ultra-fast response and high accuracy.

The combination of plasmas and catalysis under moderate temperatures is an emerging area. The introduction of a plasma to a catalytic system may have several effects including the change in the distribution or type of reactive species available for reaction. The integration of a Hiden HPR-60 molecular beam mass spectrometer system (MBMS) allows the analysis of both the gaseous species and the ions and radicals produced in the plasma.

APPLICATIONS INCLUDE:

- ▶ catalytic reactors
- ▶ reaction kinetics
- ▶ study of transients
- ▶ plasma chemistry

FEATURES:

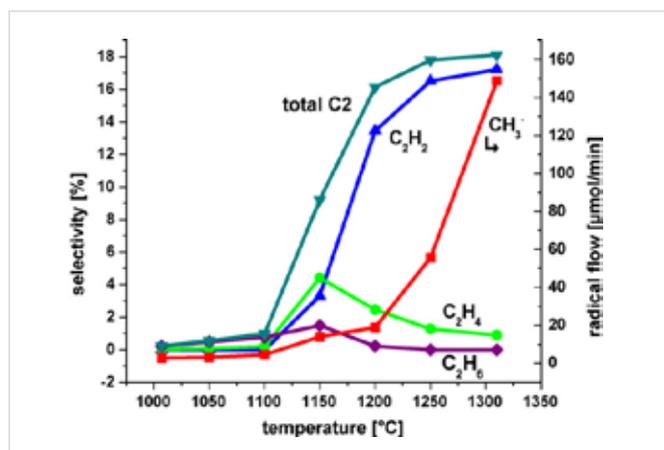
- ▶ molecular beam sampling at atmospheric pressure
- ▶ positive and negative ion analysis
- ▶ user replaceable skimmer cones (can be biased)
- ▶ electron attachment ionisation mode for the study of electro-negative radicals
- ▶ APSI-MS soft ionisation mode for radicals analysis
- ▶ mass range options: 300, 510 or 1000 amu
- ▶ energy range options: 100 eV or 1000 eV



Molecular beam inlet



HPR-60 MBMS



Selectivities of ethane, ethylene and acetylene and comparison of CH₃ radical flows depending on the temperature in the catalytic partial oxidation of methane. (For more information, see M. Geske et al., *Catalysis Today*, 2009, **142**, 61–69).

APPLICATION SOFTWARE

QGA

QGA Professional Edition software is an application specific software package for quantitative gas and vapour analysis providing real-time continuous analysis of up to 32 species with species concentration measured in the range 0.1 ppm to 100%. The software can be used in either single stream mode or multi-stream mode for use with multi-stream gas selection valves with up to 80 streams.

The software features easy to use calibration routines for both cracking pattern and Relative Sensitivity (RS) measurement. Analysis is performed using simple template setup routines and features automatic spectral removal algorithms and correction factor determination to output quantitative data. Integrated inputs from external devices such as CO analysers make the software versatile for a whole range of gas analysis applications.



QGA PROFESSIONAL main screen

FEATURES:

- ▶ quantitative gas analysis of up to 32 gases
- ▶ 10 peak spectral library with intelligent library scan feature
- ▶ component gas calibration with background correction
- ▶ automatic triggering of analysis from an external input
- ▶ read multiple inputs, temperature or pressure for example
- ▶ x-axis can display time or an external input, e.g. temperature

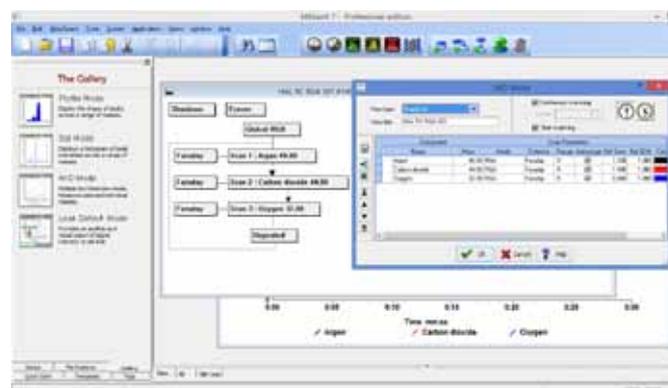
MASsoft

All Hiden instruments are supplied with MASsoft mass spectrometer control software. MASsoft Professional is a multilevel software package allowing both simple control of mass spectrometer parameters and complex manipulation of data and control of external devices.

Quick start tabs with user configurable single key start functions means novice users can start collecting data within seconds.

Scan templates allow fast set up of scans from previous similar experiments.

User selected alarm facilities (including status indication with message send and output drive capability) provide powerful control for process environments.



MASsoft PROFESSIONAL overview

FEATURES:

- ▶ mass spectrometer ionisation energy control for soft ionisation of complex mixtures
- ▶ export data to NIST MS database for analysis of unknowns
- ▶ export to external data analysis software, e.g. Excel, Origin
- ▶ control of external devices, e.g. MFCs, gas switching/sampling valves and furnace PID controllers
- ▶ output data as percentage or ppm files
- ▶ real-time subtraction of overlapping peaks

THERMAL ANALYSIS SOFTWARE

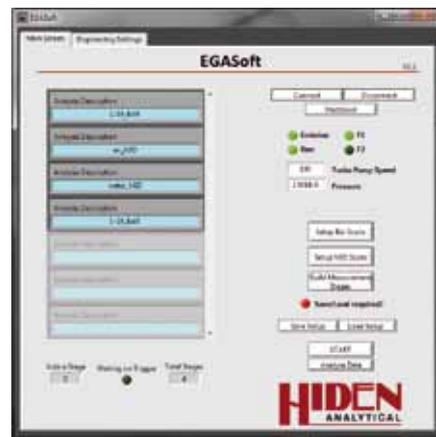
Application Specific Software for Data Acquisition and Control

A range of software packages are available for data acquisition and control of other devices for more integrated experimental procedures. Three software packages are available depending on the level of integration required.

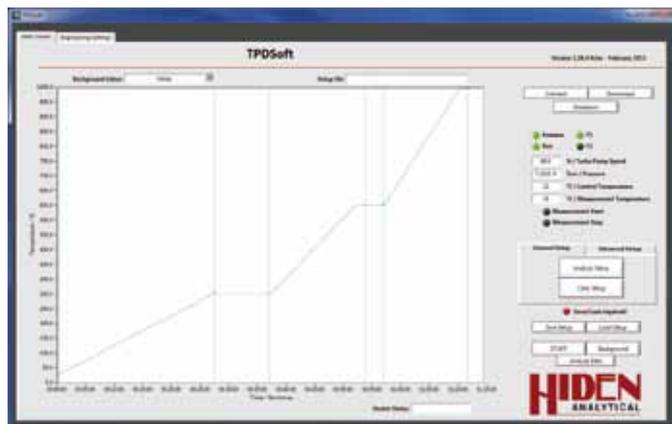
All feature a simple MS interface for Bar or MID scan setup, 3D bar scan data viewing for identification of desorption trends and peak integration/deconvolution functions.

EGAssoft

For use with thermal analysis equipment (TA-MS) e.g. TGA/DSC systems or Hiden Mass Spectrometers coupled to external reactor/furnace systems. The software features inputs for temperature signals and simple start/stop triggering from external devices where available. In addition automatic exports to TGA manufacturers' software packages are available. MS analyses can be configured in stages triggered by start/stop signals for use with TGA autosamplers.



EGAssoft main screen



TPDsoft software

TPDsoft

For use with the Hiden TPD workstation or for users of a Hiden MS in conjunction with a compatible Eurotherm controller (e.g. model 2416, RS485, Modbus Enabled) for heater/furnace control. A ready configured temperature control unit is also available from Hiden. The software controls a temperature profile on to which MS measurement stages can be added. MS measurement stages are triggered by temperature or time depending on where in the temperature profile the scan is placed. This allows complete control of a TPD experiment in one software package for easy synchronisation of MS data and temperature signal.

FEATURES:

- ▶ simple MS setup and control
- ▶ 3D bar scan view
- ▶ peak integration/deconvolution
- ▶ automatic overlap removal
- ▶ custom export formats for NIST, TA Instruments, Mettler Toledo, PerkinElmer, Setaram etc.
- ▶ external temperature inputs
- ▶ automatic MS trigger

THERMAL ANALYSIS SOFTWARE

Application Specific Software for Data Acquisition and Control

CATLAB software

For use with the Hiden CATLAB & CATLAB-FB systems. Controls MS data acquisition alongside other devices such as MFCs, furnace temperature, switching valves etc. The software can also be used with existing compatible furnace systems when supplied with the Hiden Gas Control unit and a suitable Eurotherm controller.

The software controls the experiment by following a temperature profile defined by the user. This profile is then used to trigger changes in gas composition, inject pulses of gas and start/stop MS analysis files. Different analysis files can be configured for different parts of the experiment ensuring the MS is always configured with the optimum settings for that part of the experiment.

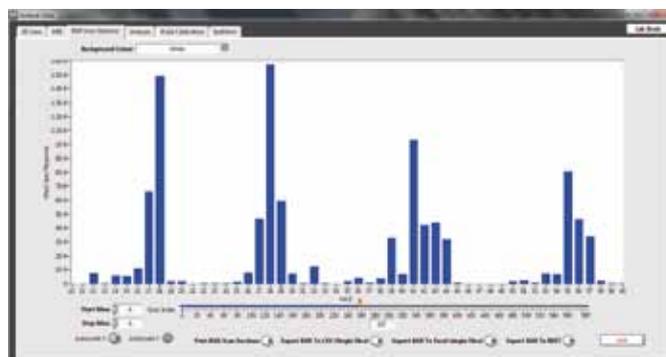
A number of data analysis routines are also included in the software for determination of catalyst properties such as metal surface area, dispersion and pulse adsorption isotherms etc.



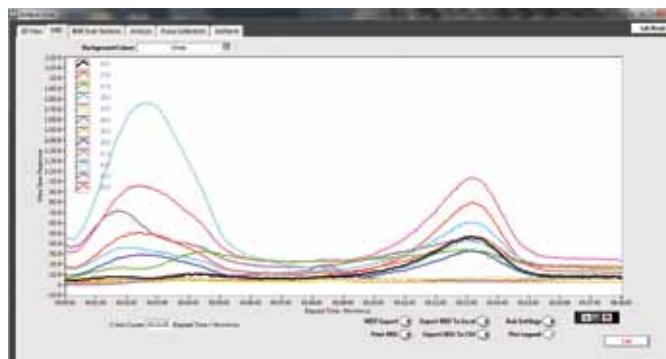
CATLAB software

APPLICATIONS:

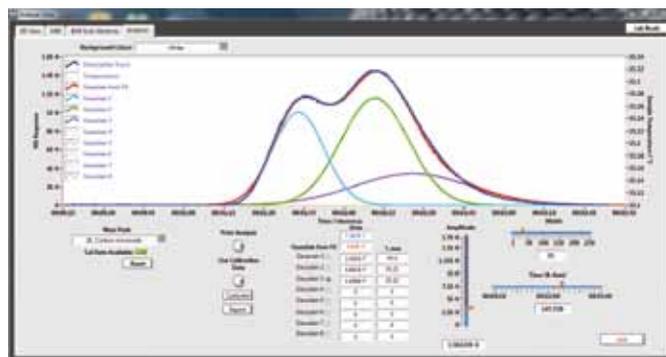
- ▶ TA-MS
- ▶ catalysis
- ▶ UHV TPD
- ▶ off-gas analysis
- ▶ TPD/R/O



2D bar scan mode



MID scan mode



Peak integration/deconvolution

Hidden **APPLICATIONS**

Hidden'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
- ▶ SIMS
- ▶ end point detection in ion beam etch
- ▶ elemental imaging – 3D mapping



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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