MASS SPECTROMETERS FOR

CATALYSIS & THERMAL ANALYSIS

HIDEN ANALYTICAL

MASS SPECTROMETERS

for Catalysis and Thermal 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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www.HidenAnalytical.com

CONTENTS

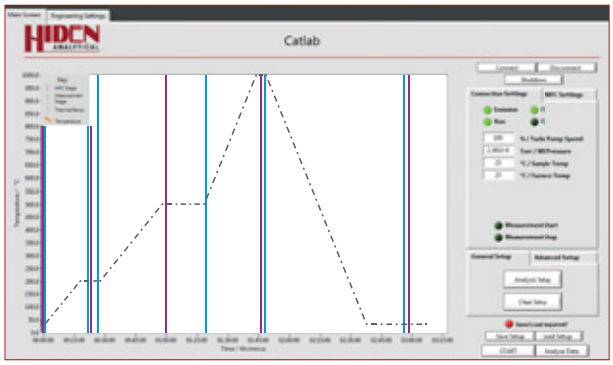
CATLAB

Automated Microreactor/MS System

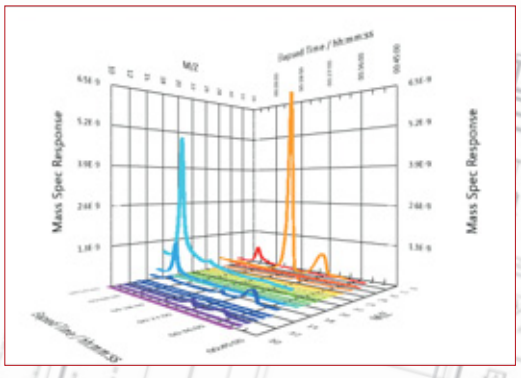
The Hiden CATLAB is a bench-top microreactor and combined mass spectrometer system for rapid and reproducible catalyst characterisation and reaction studies. The microreactor and mass spectrometer are delivered as a complete system from a single manufacturer, unique to the industry, ensuring optimum 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.

CATLAB microreactor module

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.



CATLAB software



CATLAB data display

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

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QIC SERIES

for Gas Analysis

QGA – bench-top gas analyser

MID trace

The Hiden QIC Series Gas Analysers are a range of realtime bench-top mass spectrometers for monitoring evolved gases and vapours across the pressure range 100 mbar to 2 bar. Mass range options are 200 amu, 300 amu, 510 amu and 1000 amu with an ultimate detection limit of 5 ppb.

They are offered with a wide range of interfaces for connection to thermal analysis equipment such as TGAs or tube furnaces and all components are designed to be user serviceable.

Each analyser is configured with Hiden's heated Quartz Inert Capillary (QIC) inlet for continuous sampling of permanent gases and vapours with a response time of less than 300 ms.

A choice of pumping options (independent or combined foreline and bypass pumping) for different applications allows the inlet flow rate to be chosen over the range 1 sccm to 20 sccm for matching to specific outlets and gas compositions. The high sample throughput and unique design virtually eliminates the incidence of blockages compared with other inlet types. The fast response and recovery of the QIC inlet to vapour species far exceeds conventional capillary inlets making these analysers ideal for TPD, TPR/O and TPRx studies as well as continuous monitoring of reaction processes.

FEATURES:

quantitative data output

simultaneous detection of more than 100 species from internal library

soft ionisation mode for reducing fragmentation

analogue inputs for logging external signals, e.g. temperature and pressure

multiple I/Os for data synchronisation with external events

OPTIONS:

control of external devices e.g. MFCs, temperature controllers

heated inlet particulate filter

multiport sampling valve

HPR-20 QIC R&D^{Plus}

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SPACI-MS

Spatially Resolved Capillary Inlet MS

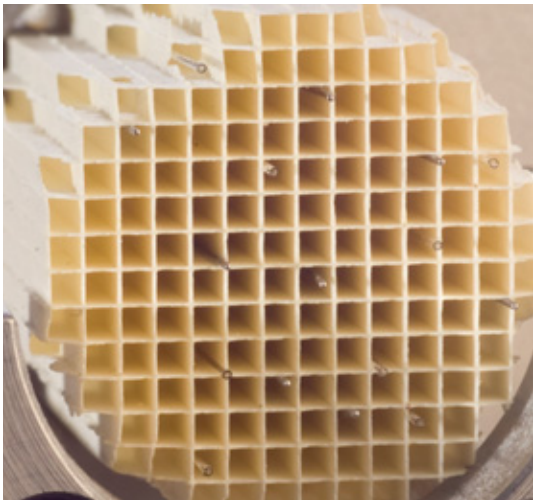
The Hiden Analytical spatially resolved capillary inlet MS (Spaci-MS) is the first commercially available instrument of its kind. Recipient of a R&D 100 award, the Spaci-MS inlet was originally conceived and developed by researchers at the Oak Ridge National Laboratory and Cummins, Inc. to study diesel catalysis(1) and has been further developed for a whole range of applications.

Spaci-MS 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.

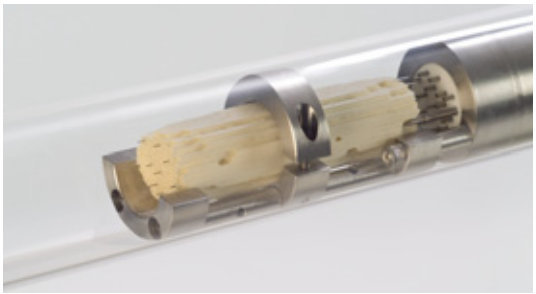
Up to 16 capillary sampling probes and thermocouples are arranged in an X-Y array. A Z-shift provides movement and accurate positioning of the array in the Z plane. In usual practice, the 16 capillary sampling probes are sequentially analysed by the MS. The Z-shift is then actuated to move the sampling 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.

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

Spaci-MS



Intra-catalyst sampling of gas and temperature



Spaci-MS sample holder

FEATURES:

quantifies intra-catalyst-channel species transients and distributions

high temporal resolution

minimally invasive

APPLICATIONS:

fuel cell studies

diesel engine catalysts

air exhaust mixing systems

non-thermal plasma reactors

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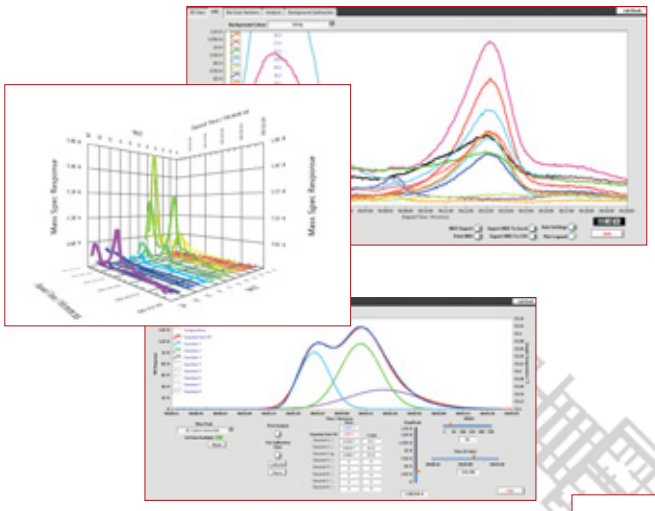
TPD WORKSTATION

Complete UHV TPD System

TPD Workstation viewport

The Hiden TPD Workstation is a complete experimental system for UHV temperature programmed desorption (TPD) studies. 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.

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.



TPDsoft for control and analysis of TPD experiments

FEATURES:

Hiden 3F PIC mass spectrometer for fast data acquisition (> 500 data points per sec)

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

TPD Workstation

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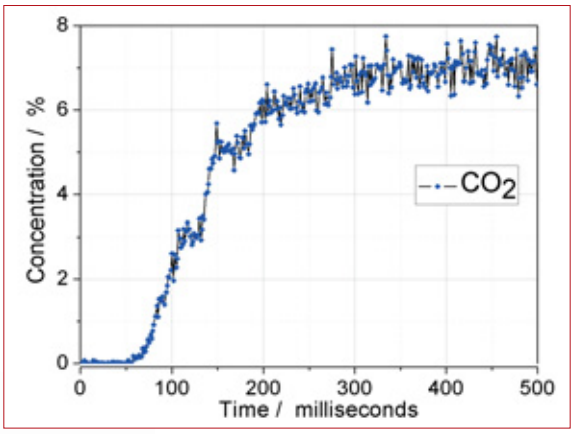
HPR-20 QIC TMS

Transient MS for Fast Event Gas Analysis

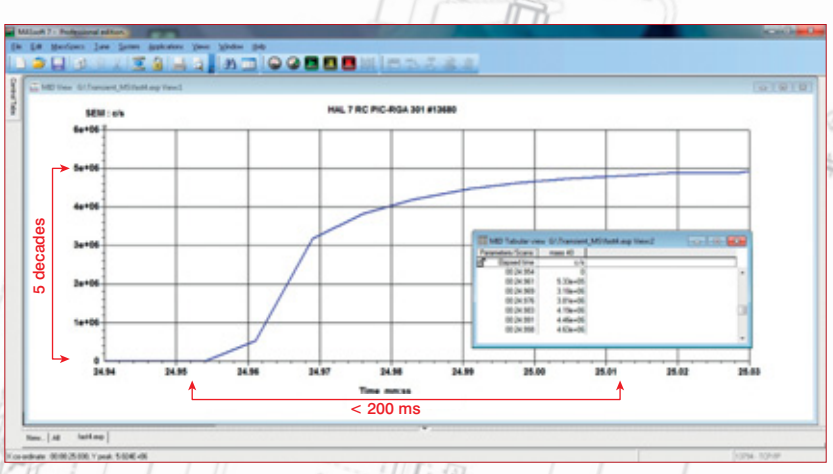
The Hiden HPR-20 QIC TMS (Transient Mass Spectrometer) is designed specifically for fast transient gas analysis at near atmospheric pressure making it ideal for analysis techniques such as SSITKA (Steady State Isotopic Transient Kinetic Analysis) where a very fast response is desired. The TMS features a 0.9 m QIC quartz-lined sampling interface and optimised pumping configuration. 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 < 200 ms. The inlet is coupled directly to Hiden's 3F PIC (pulse ion counting) digital MS which is capable of measurement speeds of over 500 data points/sec over the entire 7 decade dynamic range.

Data acquisition can be triggered and synchronised by the gas pulse signal via the on-board and comprehensive range of I/O facilities of the MS.

HPR-20 QIC TMS transient MS QIC inlet



CO_2 breath analysis, 700 data points per second



HPR-20 transient MS data – > 5 decades response in < 200 ms

FEATURES:

fast response inlet < 150 ms

fast data acquisition, > 500 data points/sec

wide dynamic range, 7 decade continuous log scale

5 decade response in < 200 ms

synchronised data acquisition via TTL inputs

TA-MS

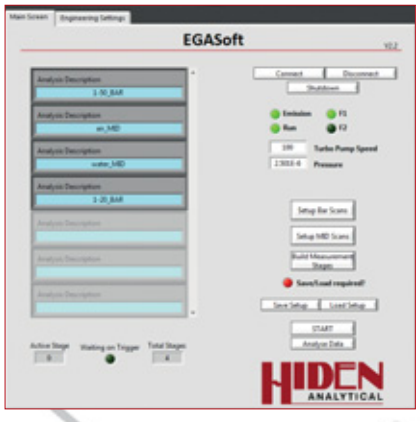
for Evolved Gas Analysis

HPR-20 QIC EGA

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, 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.

EGAsoft 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.



EGAsoft

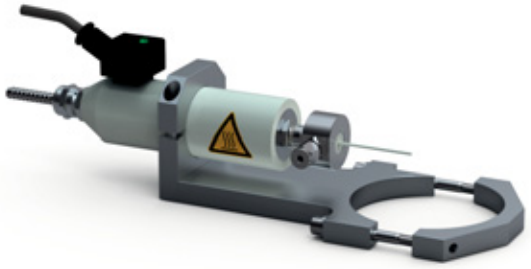
FEATURES:

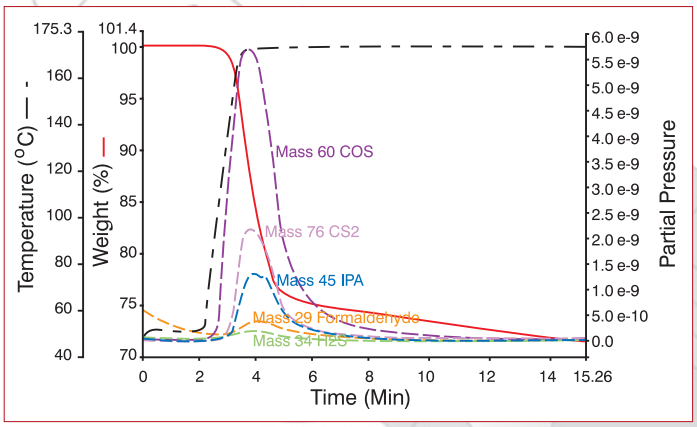
minimum dead volume

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 plot

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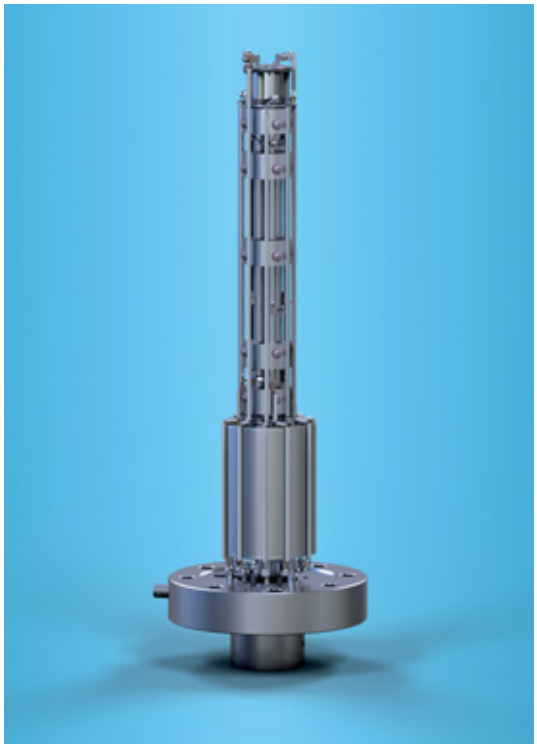
UHV TPD

for Fast Event UHV Studies

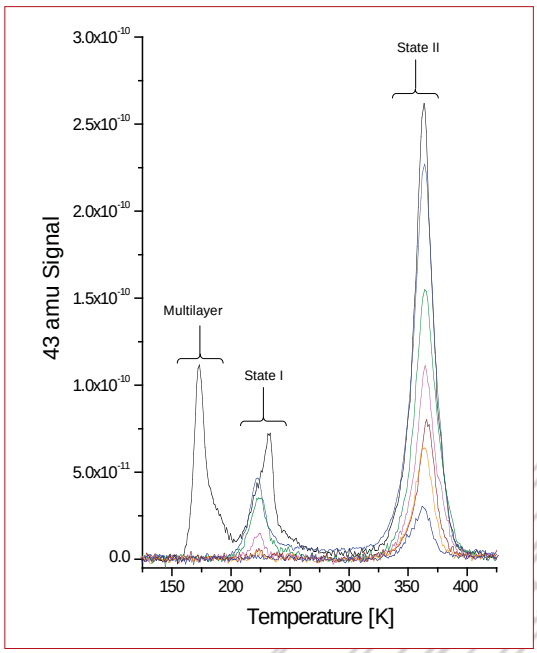
Hiden's 3F PIC Series Quadrupoles are high precision triple filter analysers with digital detectors for ultimate sensitivity and time resolution in fast event studies such as UHV TPD. 3F Series analysers are available with UHV compatible mass filter shrouds and low profile ion source for close positioning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



3F PIC



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

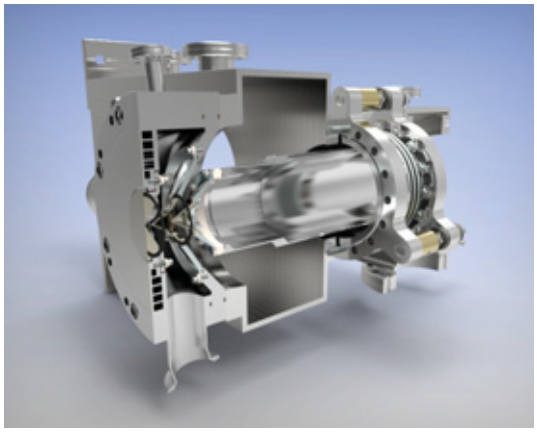
wide dynamic range

7 decade continuous log scale

gating input for pulsed gas studies down to 100 ns gating resolution

HPR-60 MBMS

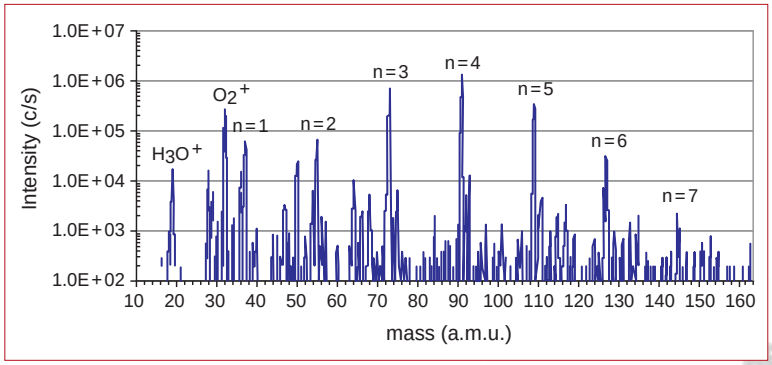
Molecular Beam Sampling Mass Spectrometer



Molecular beam inlet

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 multistage 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.

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



Hydrated cluster ions from atmospheric dielectric barrier discharge

FEATURES:

molecular beam sampling at atmospheric pressure

+ve and -ve ion analysis

user replaceable skimmer cones

mass range options: 300, 510 or 1000 amu

APPLICATIONS INCLUDE:

catalytic reactors

reaction kinetics

study of transients

plasma chemistry

HPR-60 MBMS

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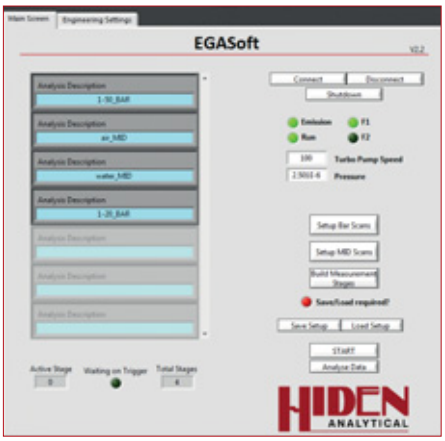
THERMAL ANALYSIS SOFTWARE

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.



EGAsoft main screen

EGAsoft

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.

TPDsoft software

TPDsoft

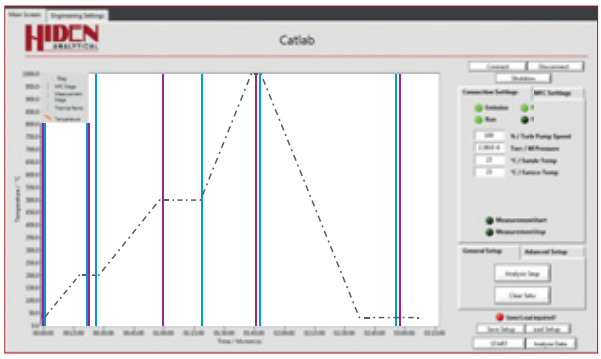
For use with the Hiden TPD workstation or for users of a Hiden MS in conjunction with a compatible Eurotherm controller (e.g. model 2416, RS485, Modbus Enabled) for heater/furnace control. A ready configured temperature control unit is also available from Hiden. The software controls a temperature profile 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:

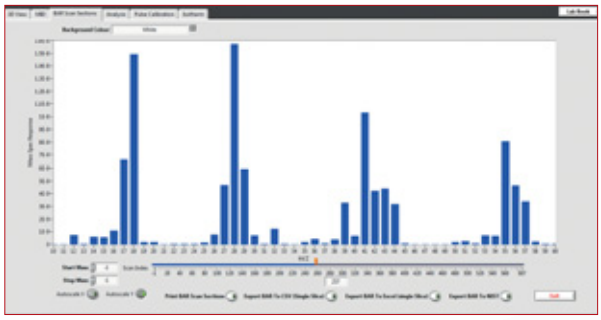
- **simple MS setup and control**
- **3D bar scan view**
- **peak integration/deconvolution**
- **automatic overlap removal**
- **custom export formats for NIST, TA Instruments, PerkinElmer, Setaram etc.**
- **external temperature inputs**
- **automatic MS trigger**

THERMAL ANALYSIS SOFTWARE

Application Specific Software for Data Acquisition and Control



CATLAB software



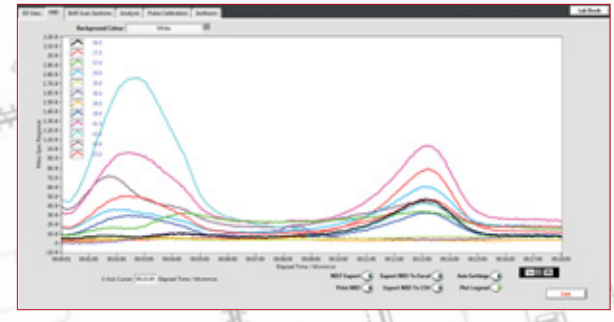
2D bar scan mode

CATLAB Software

For use with the Hiden Microreactor system. 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 included in the software for determination of catalyst properties such as metal surface area, dispersion and pulse adsorption isotherms etc.



MID scan mode

Peak integration/deconvolution

APPLICATIONS:

TA-MS

catalysis

UHV TPD

off gas analysis

TPD/R/O

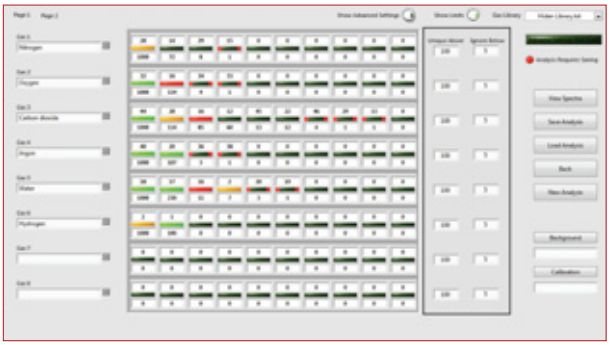
QGA PROFESSIONAL

for Quantitative Gas and Vapour Analysis

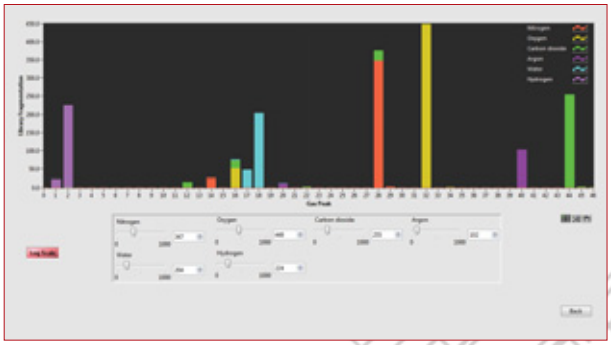
QGA Professional Edition software is an application specific software package for quantitative gas and vapour analysis providing realtime continuous analysis of up to 16 species with species concentration measured in the range 0.1 ppm to 100 %. The software can be used in either single stream mode or 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 overlap 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



Automatic mass spectral analysis setup



Spectra view

FEATURES:

quantitative gas analysis of up to 16 gases

10 peak spectral library with intelligent library scan feature

component gas calibration with background correction

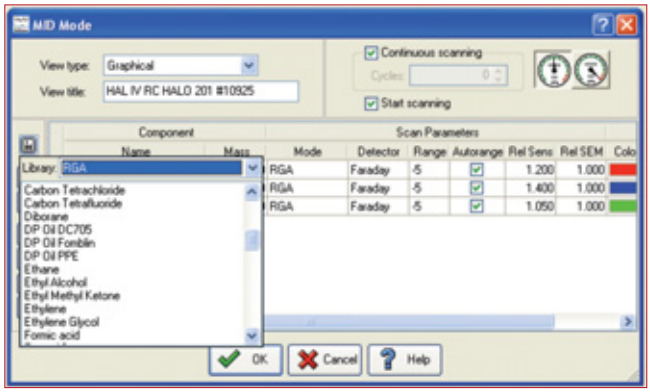
automatic triggering of analysis from an external input

read multiple inputs, temperature or pressure for example

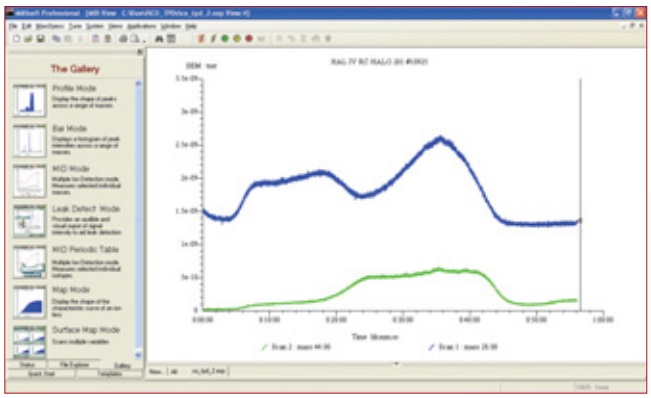
x-axis can display time or an external input, e.g. temperature

MASsoft PROFESSIONAL

Control Software



Trend analysis (MID) setup

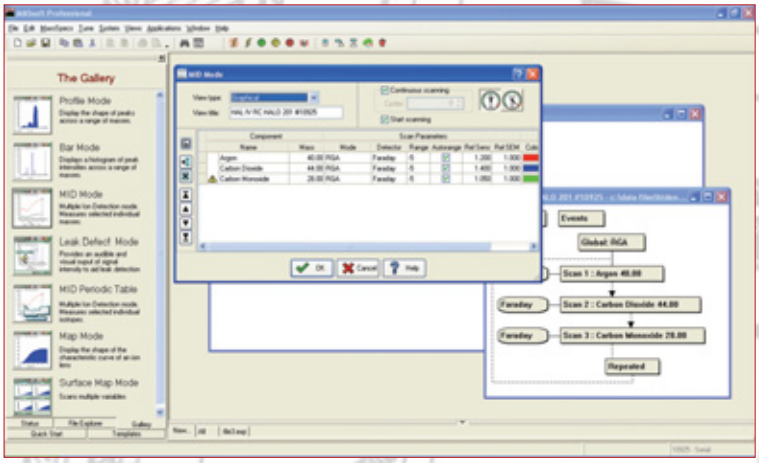


Scan gallery

All Hiden instruments are supplied with MASsoft Professional 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.



MASsoft Professional - overview

FEATURES:

full control of the mass spectrometer ion source enables soft ionisation of complex organics, and appearance potential mass spectrometry for simplification of complex spectra

export data to NIST mass spectral database for comprehensive analysis of unknowns

export to external data analysis software, e.g. Excel, Origin

control of external devices, e.g. mass flow controllers, gas switching/sampling valves and furnace temperature controllers

output data as percentage or ppm files

realtime subtraction of overlapping peaks for quantitative measurements

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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 Science

UHV TPD

SIMS

end point detection in ion beam etch

elemental imaging – surface mapping

Plasma Diagnostics

plasma source characterisation

etch and deposition process reaction kinetic studies

analysis of neutral and radical species

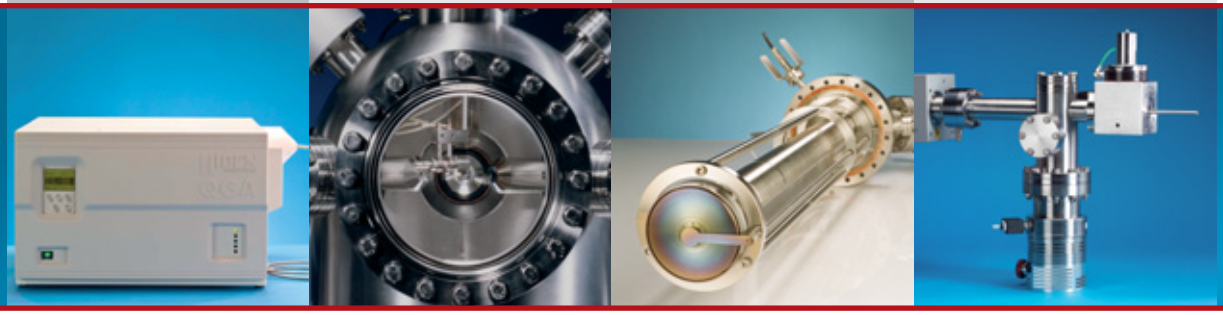
Vacuum Analysis

partial pressure measurement and control of process gases

reactive sputter process control

vacuum diagnostics

vacuum coating process monitoring



quadrupoles for advanced science

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