HIDEN
ANALYTICAL

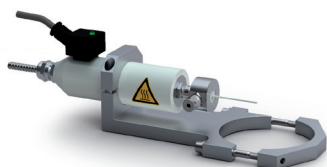# Mass Spectrometers
for Catalysis &
Thermal Analysis
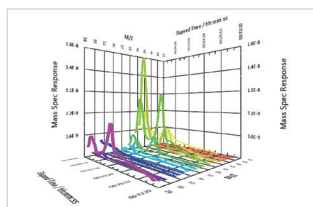
MASS SPECTROMETERS
for Catalysis & Thermal Analysis

Hiden Analytical have been designing and developing the highest quality quadrupole mass spectrometer based gas analysis systems for over 40 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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TGA-MS Interface

CATLAB data display

TDSLab SERIES

HPR-20 OEMS

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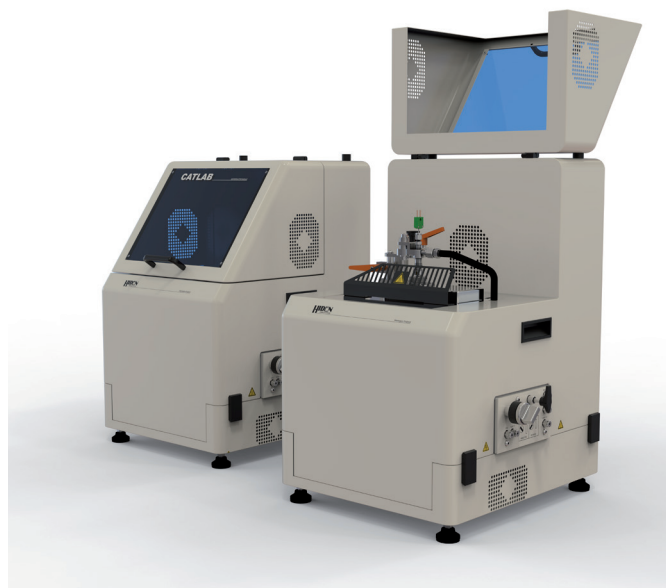
The Hiden CATLAB delivers a range of completely automated, dynamic, temperature programmed, pulse chemisorption and isothermal techniques. Template driven CATLAB software provides automatic control of gas composition and delivery, temperature ramp and set-point as well as full mass spectrometer parameter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

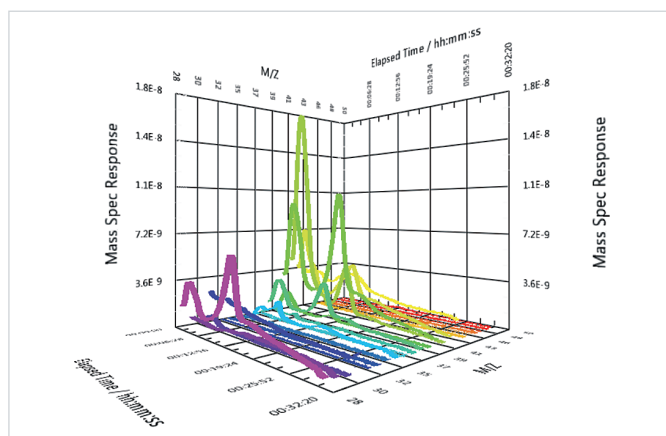**CATLAB** microreactor module

TECHNIQUES:

▶ **temperature programmed desorption (TPD)**

▶ **temperature programmed reduction/oxidation (TPR/O)**

▶ **temperature programmed reaction (TPRx)**

▶ **pulse chemisorption**

▶ **pulse calibration**

▶ **dispersion measurements**

▶ **adsorption isotherms**

▶ **reaction studies**

▶ **catalyst screening**

CATLAB data display

FEATURES:

▶ 1000°C furnace

▶ 4 MFC gas mixing unit (8 Optional)

▶ 3 reactor sizes

▶ trace heating

▶ integrated switching and pulse injection valves

▶ integrated software to control MS and reactor parameters

▶ optimised MS sampling

SYSTEMS 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)
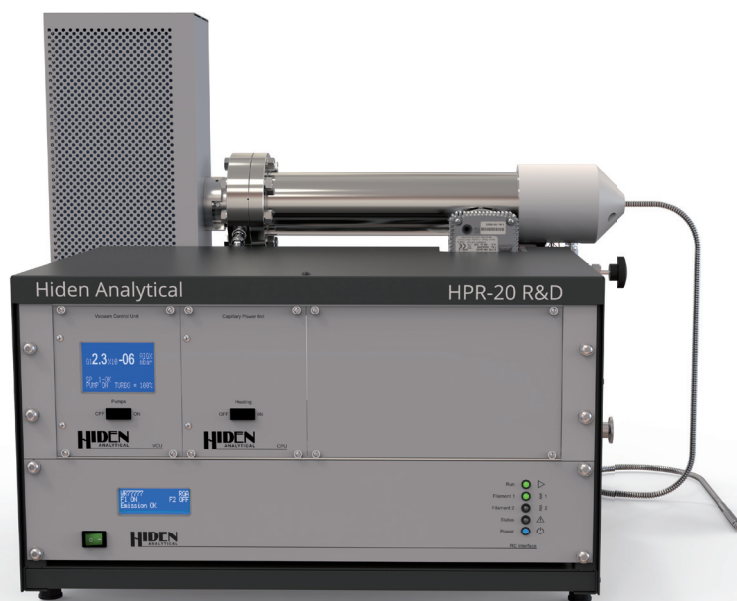
HPR-20 R&D - specialist gas analysis system for advanced research

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 2.0 for quantitative gas analysis** - included as standard with QGA systems

- **EGAsoft for evolved gas analysis** - included as standard with HPR-20 EGA systems

QGA 2.0
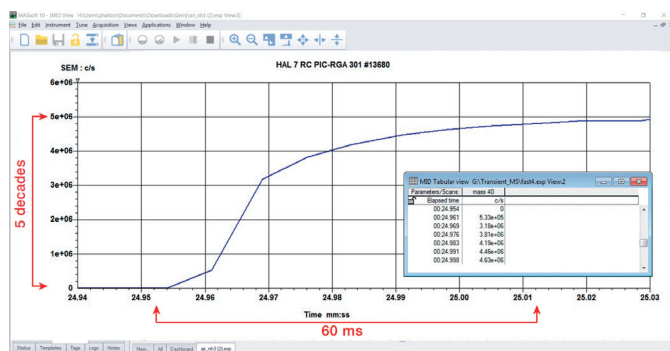
HPR-20 TMS
Transient MS for Fast Event Gas Analysis


HPR-20 TMS transient MS QIC inlet

The Hiden HPR-20 TMS Transient MS is configured for fast event 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.

Suited to fast data acquisition in the measurement of gas and vapour compositions in the sub ppm to low % concentration range.


HPR-20 transient MS data - > 5 decades response in < 100 ms
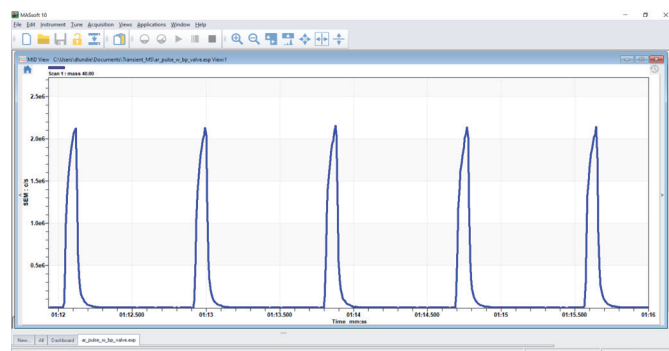
Analysis of fast gas pulsing experiments

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 dynamic range
▸ detection of low ppm to high % levels in < 100 ms
▸ fast scan speeds, 500 amu/s for transient analysis
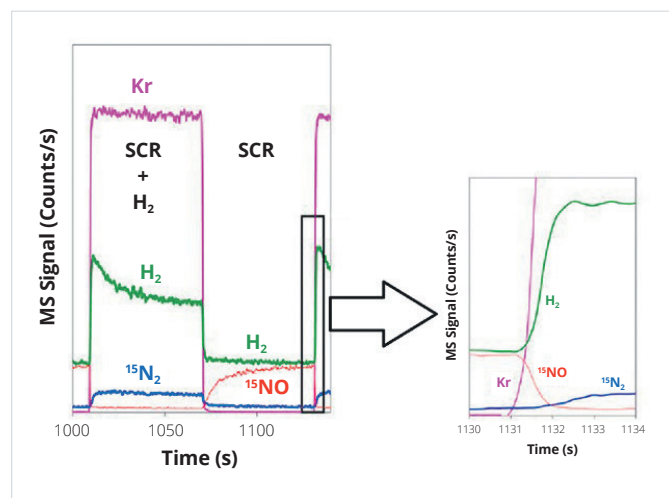
Mass spectrometer response for $^{15}N_2$, ^{15}NO, H_2 and Kr when switching 0.72% H_2 in and out of a SCR (NO$_x$ with octane) feed stream over a catalyst at 300 ºC (J. P. Breen, R. Burch, C. Hardacre, C. J. Hill and C. Rioche, J. Catal., 2007, 246, 1-9).
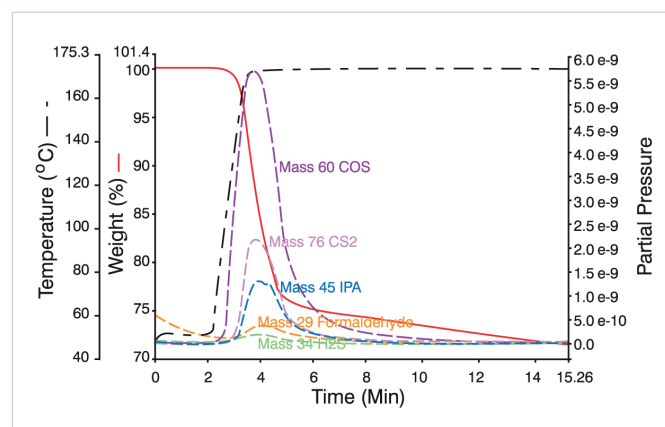
HPR-20 EGA
for Evolved Gas Analysis in TGA-MS

HPR-20 EGA

The Hiden HPR-20 EGA gas analysis system is configured for continuous analysis of evolved gases and vapours from thermogravimetric analysers (TGA). Interface systems are available for most TGA instruments. The TGA interface includes re-entrant furnace sampling, providing close coupling to the TGA furnace region for optimized evolved gas/vapour analysis.

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 to 30 bar.
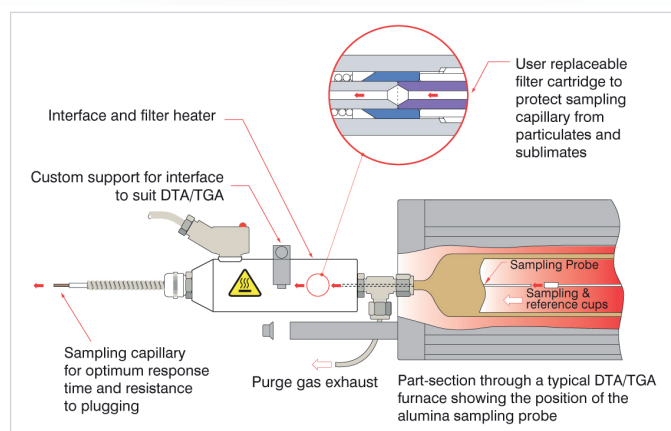

Typical **TGA** inlet

TGA-MS plot

Each interface has been custom engineered in collaboration with TGA manufacturers and includes, where necessary, robust clamping arrangements and an 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.

FEATURES:

▸ enhanced pumping for light gases

▸ heated inlet for non-tailing response to desorbed gases and vapours

▸ control of ionisation energy parameters for simplification of cracking patterns

▸ custom, low dead volume interface to specific manufacturers' TGA systems

▸ optimised data acquisition using EGAsoft

▸ mass range option 200 or 300 amu
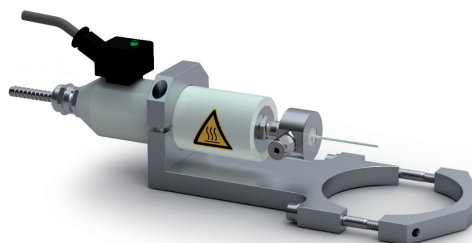
SpaciMS
Spatially Resolved Capillary Inlet MS

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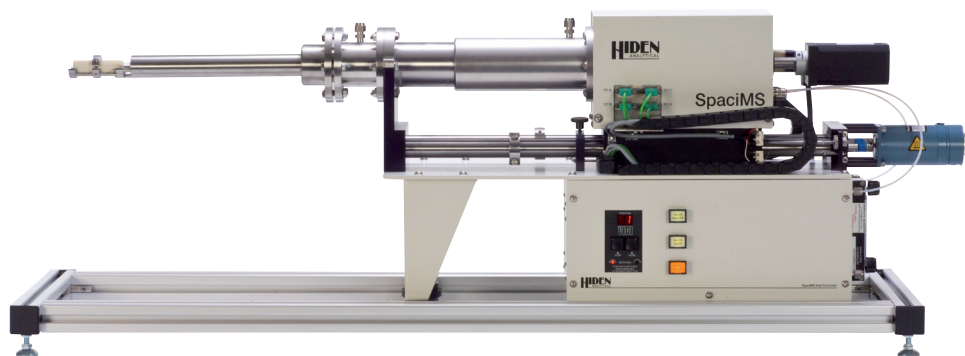
[1] *Partridge, W.P. et al.* 2000 *Journal of Fuels & Lubricants* **109** 2992-2999

Intra-catalyst sampling of gas and temperature

SpaciMS sample holder

SpaciMS

FEATURES:

▸ quantifies intra-catalyst-channel species transients and distributions

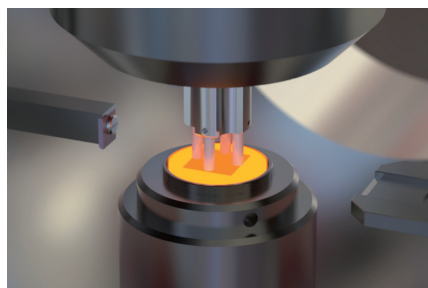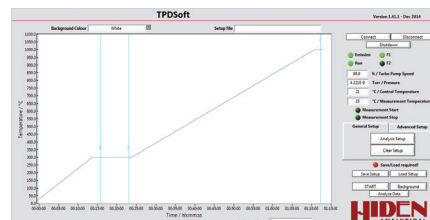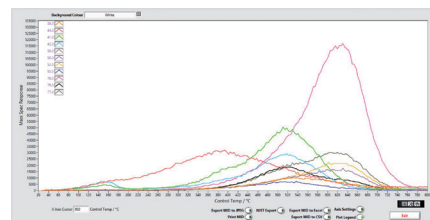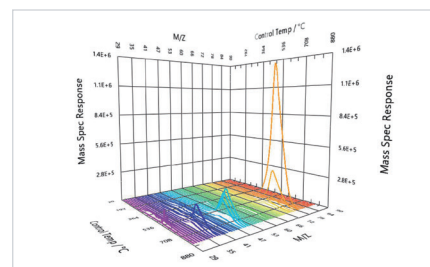▸ high temporal resolution

▸ minimally invasive

TDSLab SERIES
TDSLab Series for Advanced Materials Analysis

The TDSLab Series, including the TDSLab-6, TDSLab-9, and TDSLab-20, represents the next generation in thermal desorption spectrometry, replacing the highly regarded TDSLab. These advanced systems are designed to meet the rigorous demands of modern scientific research and industrial applications, providing unparalleled precision, reliability, and versatility. The TDSLab Series enhances and expands upon the capabilities of the original TDSLab, offering improved precision, higher mass resolution, and advanced user-friendly features to support a wider range of scientific applications.

TPDsoft for control and analysis of TPD experiments

TDSLab Series

TDSLab Series with hot sample

TDSLab-6

FEATURES:

▸ 1000°C sample stage with PID control module

▸ high precision triple filter quadrupole mass spectrometer

▸ multiport UHV chamber

▸ water cooled heater shroud

▸ linear sample transfer mechanism and loadlock

▸ Z-drive for optimal sample/detector positioning

▸ mass filter shroud

▸ TPDsoft control and analysis software

▸ bakeout jacket (150°C max)

UHV TPD
for Fast Event UHV Studies

Range of Shrouds

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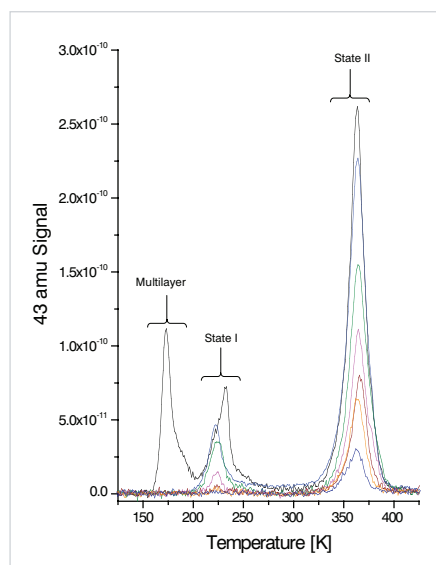
TPD Plot courtesy of M. Kadodwala (University of Glasgow, UK)

3F PIC

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-40 DEMS

for Dissolved Gas Analysis and Off-gas Analysis in Electrochemistry

The Hiden HPR-40 DEMS is a bench mounted or mobile cart mounted module for the analysis of dissolved species in electrochemistry. The system is modular and adaptable. The system includes two differential electrochemical mass spectrometry 'DEMS' cell inlets, designed for material/catalysis studies (cell type A), and electrochemical reaction studies (cell type B).

For applications where Online Electrochemical MS (OEMS) from an existing cell or reactor is required, a range of standard inlet options is available offering both evolved off-gas and dissolved species analysis solutions. The HPR-40 DEMS system has a mass range of 200 amu (300 amu option) and sub ppm detection levels.
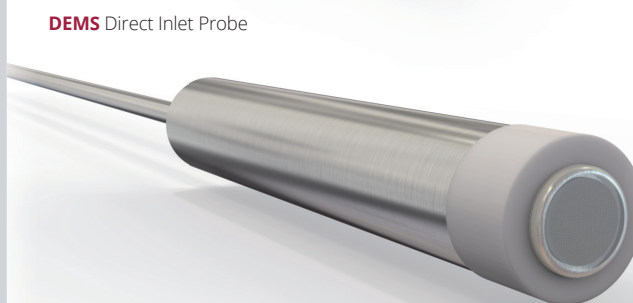
HPR-40 DEMS system
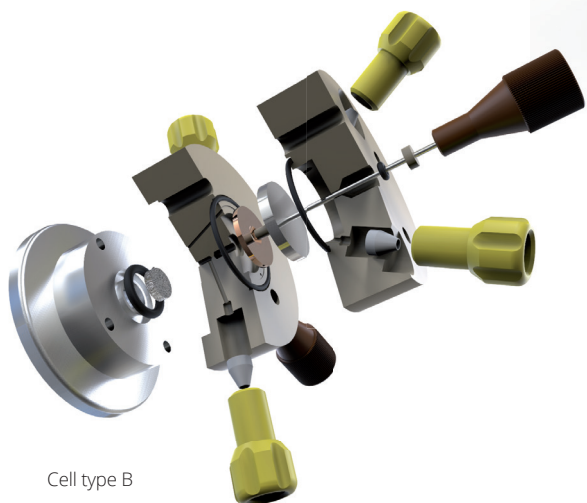
Cell type B

Cell type A

DEMS Direct Inlet Probe

FEATURES:

▸ compact benchtop mass spectrometer system

▸ mass scanning, and time/intensity trend monitoring of multiple species

▸ modular, user configurable system including DEMS cell

▸ fast response (< 1 second), nano-porous electrolyte/MS interface

▸ DEMS off-gas analysis capillary sampling option with micro-flow inlet

▸ mass range: 200 amu is standard. 300 amu option

HPR-20 OEMS

for Continuous Analysis of Evolved Gases and Vapours in Electrochemistry

The Hiden HPR-20 OEMS is a gas analysis system configured for continuous analysis of evolved gases and vapours for analysis of evolved gases in electrochemistry as a function of the applied potential on the real-time scale. The ultralow flow real time sampling capillary allows for connection for head space sampling and/or connection to electrochemical cells, El-Cell for example.

The standard system with 200 amu mass range has a detection capability to less than 100 parts-per-billion. The optional 3F series 300 amu system with triple-stage mass filter offers extended detection levels to 5 parts-per-billion together with enhanced contamination resistance and is recommended for applications requiring optimum abundance sensitivity and/or analyses of corrosive or condensable species.

HPR-20 OEMS system

Ultra-Low Flow (ULF) QIC Heated Capillary Inlet

FEATURES:

▸ response time as fast as 300 ms

▸ detection from 100 ppb – 100%

▸ fast data acquisition: up to 1000 measurements per second

▸ flow rate – 12 l/min to 16ml/min

▸ APSI-MS soft ionisation mode

▸ mass range: 0-300 amu

▸ broad range of sampling accessories

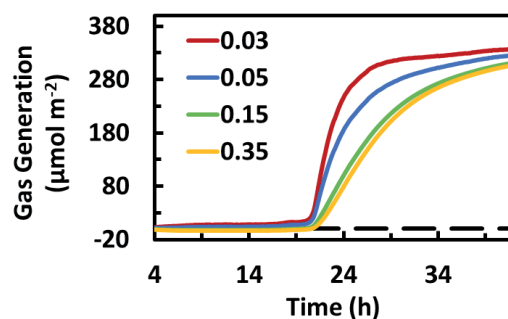▸ custom interfaces for diverse OEMS applications
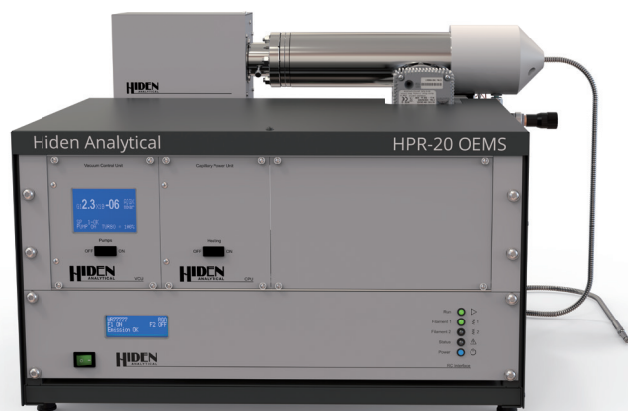
Figure 1. In-situ OEMS data plotted with gas generation from closed-cell measurements of a lithium | LNO half-cell.
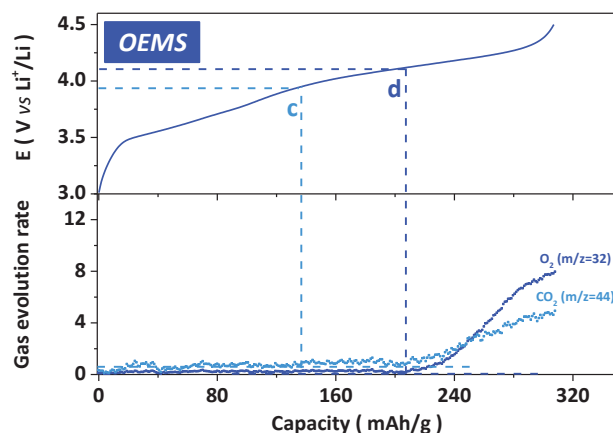

Figure 2. Potential (top panel) and gas evolution rate (bottom panel) as a function of capacity for $Li_2Ru_{0.75}Ti_{0.25}O_3$ measured by OEMS.

*Reference: Journal of The Electrochemical Society (2018) 165, A3326*

APPLICATION SOFTWARE

QGA 2

QGA 2 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.
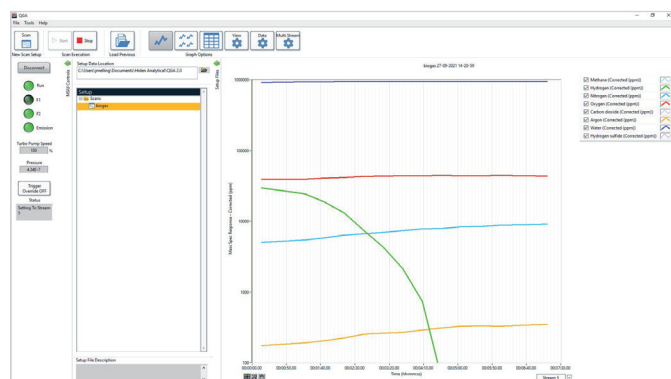
QGA 2 home screen

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.
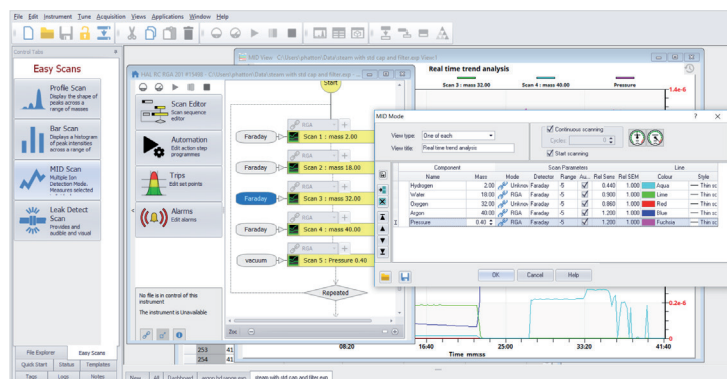
MASsoft PROFESSIONAL overview

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.

EGAsoft

For use with thermal analysis equipment (TA-MS) e.g. TGA 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.
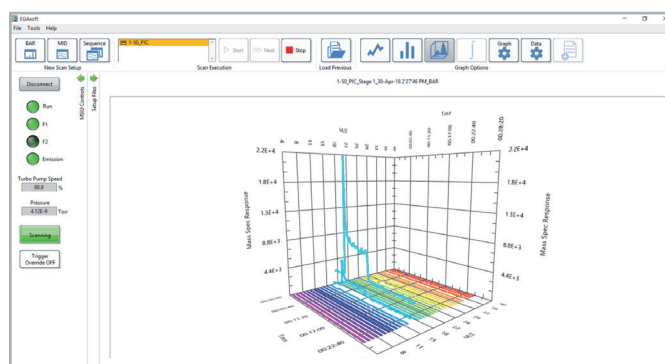
EGAsoft 3D bar mode

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.
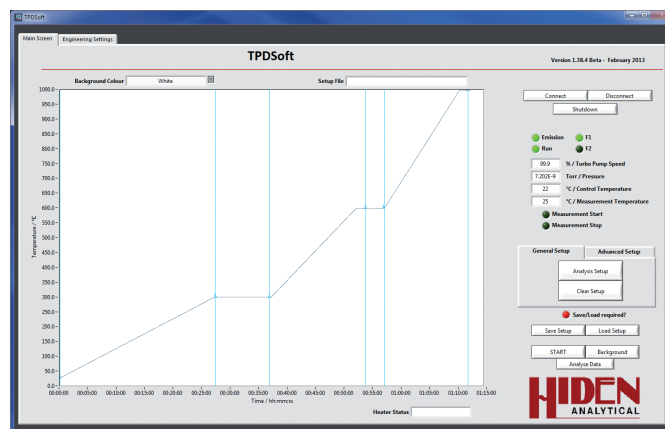
TPDsoft software

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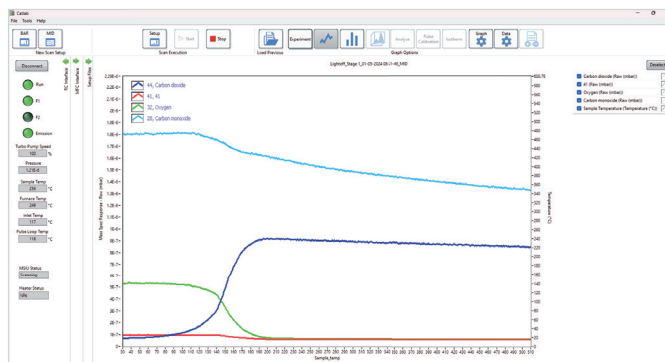
A number of data analysis routines are also included in the software for determination of catalyst properties such as metal surface area, dispersion and pulse adsorption isotherms etc.
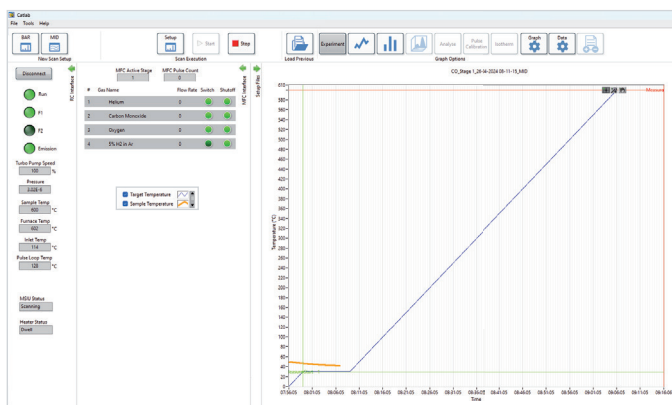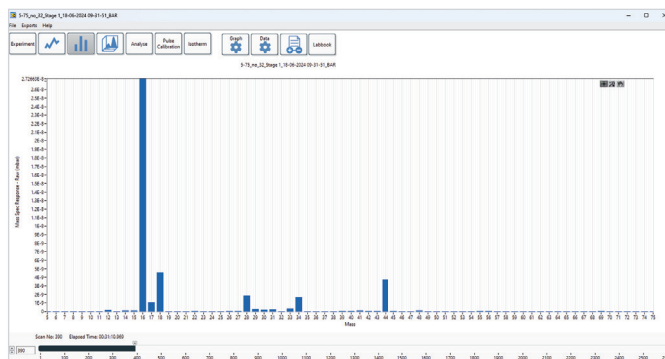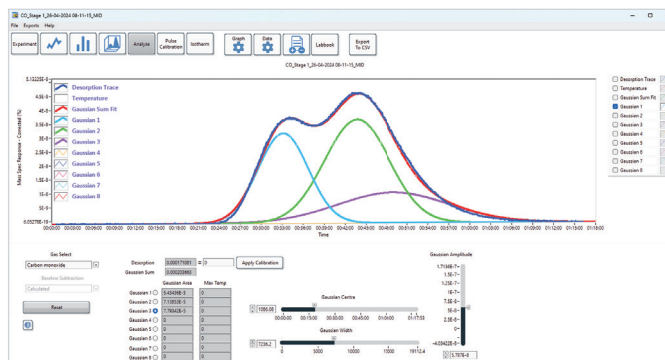

2D bar scan mode

MID scan mode

Peak integration/deconvolution

CATLAB software
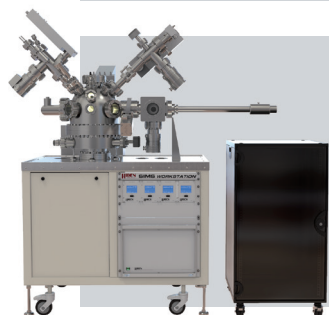
Hiden**APPLICATIONS**

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

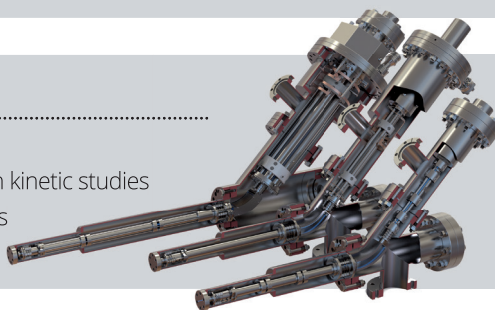## SURFACE ANALYSIS

▶ 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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