



Operando Studies

HPR-20 for *Operando* Experiments

Overview.

One of the more recent developments in catalyst characterization has been the increased use of *operando* techniques.

The *operando* methodology combines the evaluation of the active catalyst structure (spectroscopy) and catalytic activity/selectivity (gas analysis) in a single experiment.

The study of reaction mechanisms under real working conditions gives unique insight into the processes involved in the reaction *i.e.* reaction intermediates. Understanding of reaction mechanisms is important for the improvement of current catalysts or the rational design of new ones. *Operando* methodologies are an excellent tool for assessing the structure-reactivity relationships at a molecular scale since catalyst structure and catalytic performance are determined simultaneously

Operando techniques combine spectroscopic characterisation of catalysts under realistic reaction conditions using techniques such as FTIR (e.g. DRIFTS [1]), UV-Vis and Raman spectroscopies or x-ray based techniques such as XAFS [2] with simultaneous real-time online analysis of reaction products e.g. Mass Spectrometry. For online analysis of products Hiden produce the HPR-20 gas analysis system.

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Figure 1: HPR-20 Gas Analysis System with Selected Sampling Options

HPR-20 Gas Analyser

The Hiden HPR-20 is a compact, bench top gas analysis system (Figure 1) which features Hiden's heated Q/C (Quartz Inert Capillary) inlet with controlled by-pass for rapid and continuous sampling in the process pressure range 100 mbar to 2 bar absolute. The unique Q/C inlet design allows for a range of custom adaptors, flow and temperature matched, to be interfaced with other devices for combined spectroscopic evaluation and product analysis.

Sensitivity

The standard HPR-20 system has a 200 amu mass spectrometer with dual faraday/electron multiplier detectors giving detection limits in the ppm range. The system can also be configured with optional Triple Filter 3F mass spectrometer for enhanced sensitivity (ppb) and increased mass range (300,500).

Response Time

The QIC capillary is a 1.8m long quartz lined capillary heated to over 160 °C. The response time of this inlet is <300 ms from the end of the capillary to detection by the MS. Response times can be decreased by using the optional 1m capillary giving possible response times of <150 ms.

Acquisition Speed

Acquisition speed of >500 data points per second can be achieved with the HPR-20. The system can also be configured with the Hiden PIC detector of very fast data acquisition with 7 decades continuous log scale measurement. This, coupled to very fast response times, is important for experiments such as Steady State Isotopic Transient Kinetic Analysis (SSITKA) measurements [3] where fast switches of gas species occurs.

Interface

The design of the interface between the mass spectrometer and any other equipment is important in order to minimise dead volume which would add significantly to the response time. The HPR-20 is configured with a dual rotary pumping system for independent pumping of the sample bypass and MS chamber. This pumping configuration allows a high throughput of gas, (16 sccm) including light gases such as He and H₂ meaning any dead volume effects are minimised. In addition to the fast sampling speed the inlet can be configured with a range of custom designed inlets to further reduce dead volume giving optimum sampling from the external device.

Device Integration

Multiple I/Os can be configured on the HPR-20 for control and integration of external signals and devices such as control/measurement of temperature, MFC's and switching valve control. I/Os include:

5 configurable I/O TTL lines, 2 analog inputs, 2 trip relay outputs.

3 RS485 (multi protocol) to interface with a wide range of external devices, MFCs for example.

8 digital inputs compatible with 5V and 24V logic levels.

4, 8 or 16 channel analog outputs, 0-10V, 14bit (optional).

Software

All Hiden systems are supplied with MASsoft Pro control/data acquisition software. This software gives easy control of all the mass spectrometer parameters. Data output can be configured to give partial pressure, ppm or percentage outputs.

In addition to MASsoft, the system can be supplied with the Labview based QGA software. This easy to use package features auto-calibration and spectral overlap removal algorithms to give quantitative outputs in either ppm or percentage units.

Conclusion

The HPR-20 gas analysis system is ideal for use as the product analyser in *operando* studies. The unique adaptability of the sampling interfaces coupled with multiple control I/Os makes this the most flexible mass spectrometer system on the market.

References

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