Process Raman Systems
Established Expertise

Profile

The HORIBA Jobin Yvon Raman Division is the world leader in Raman spectroscopy, designing and manufacturing state-of the-art dispersive Raman spectrometer systems for over four decades. HORIBA Jobin Yvon has been at the forefront of Raman spectroscopy since its infancy. Introducing ground breaking innovations such as the first Raman microscope, the first holographic diffraction gratings, and the world’s first remote Raman sampling probe.

The Raman Division consists of the combined expertise of the Jobin Yvon, Dilor and SPEX brand names and is proud of having more than 3000 Raman systems installed worldwide.

Our Raman process systems are available as part of a complete range of HORIBA process solutions. HORIBA is a favoured supplier of environmental, analytical and process monitoring solutions throughout the world.

Introduction

Raman spectroscopy has been established in semiconductor research, polymer characterisation, pharmaceutical development and in general analytical chemistry for many years.

The technique offers a non-destructive, non-contact method of analysis. The sample is illuminated with a laser and the scattered light is collected. It is this scattered light that contains the highly specific spectral fingerprint which enables chemical identification, phase, and concentration analyses to be obtained.

In recent years developments in spectrograph design, gratings, lasers and sampling probes have successfully moved Raman out of the research and analytical laboratory and into industrial and process monitoring applications.

Modern Raman spectroscopy is ideally suited to real-time reaction monitoring and to the characterization of industrial compounds. Raman analysers have become an important part of the PAT (process analytical technologies) initiative, yielding greater understanding and control of industrial processes.
Advantages of Raman Monitoring

- Optical fibre coupling up to 100’s of meters in length can be used for remote analyses.
- Suitable for harsh environments, such as high pressure and temperature.
- Multiplexing advantage with multiple probes from a single Raman base unit.
- Non-contact measurements minimise the risk of sample contamination.
- No interference from water or CO₂ vapour. Aqueous solutions are easily analysed (compared to FTIR).
- Little or no sample preparation or sample extraction is required.
- Chemical and molecular information is rich with high specificity compared to NIR.
- The standard spectral range covers from 100 cm⁻¹ to 4000 cm⁻¹ making the technique ideal for both organic and inorganic species.
- Raman bands are usually sharper and interference weaker than in the Mid-IR and NIR, making data analysis far easier.
- Inexpensive glass or quartz sample holders are ideal in many lab situations and no consumables are required (compared to GC, MS, ICP).

Applications:
- Solutions / reaction mixes
- Emulsions
- Slurries and suspensions
- Headspace in vials/reactors
- Raw and bulk material feeds
- Vessel cleaning effluents

Sampling locations:
- Micro-reactors for development work
- Window/port sampling in reactors and UHV chambers
- Re-circulating flow loops
- Raw and bulk material hoppers and supply feeds
- Reactor drain and product storage
PCl₃ is a base ingredient used in the synthesis of numerous inflammable products and agro-chemicals. The naturally favoured reaction product of phosphorous and chlorine is PCl₃. However, the process can follow several other reaction paths depending upon the ratio and the concentrations of the starting materials. One such process can occur if phosphorous is introduced too quickly, allowing pyrophoric phosphorous to pool on the bottom of the reactor (reaction 2). This can cause a catastrophic uncontrollable state. Likewise, the reaction path can favour the formation of highly toxic PCl₅ should the chlorine content become excessive (reaction 3).

The remote non-contact nature of Raman spectroscopy provides safe and effective monitoring and control of this important industrial process.
**Complete Solutions**

*Industrial Solutions from the Experts*

HORIBA Jobin Yvon has major applications laboratories in Europe, USA and Japan and can provide the most complete level of service from provisional proof of principle measurements, to method evaluation work and finally to full process installations. The full range of Raman equipment including lab based systems, research instruments and process analysers enables HORIBA Jobin Yvon to assist its global customers in the interaction between the process engineer and the laboratory analyst.

With Raman process installations in industries such as chemical production, petrochemicals, polymers and pharmaceuticals, HORIBA Jobin Yvon has the expertise necessary to fully explore any Raman monitoring application and the HORIBA Jobin Yvon software team can provide custom solutions for data analysis and hardware operation.

In short, with the technical, applications, and service support structure of HORIBA Jobin Yvon, we can provide complete solutions for the industrial customer and are the partner of choice in implementing Raman technology in industry.

- Spectrograph / spectrometer hardware
- Housings and enclosures
- Probe design and integration
- Fibre-microscope for QC/Lab sampling
- Software and model development
- Interfacing
- Global service support
- Off-line sampling

4-20 mA output to a DCS remote control station and CSV file log output
Raman Process Analysers

The RPA-HE and RPA-AX RAMAN PROCESS ANALYSERS have been developed to provide advanced and robust performance with maximum reliability for process control applications. The compact, rugged construction with no internal moving parts is perfectly adapted to demanding industrial environments. The analysers have been designed to function with low maintenance, minimal downtime and little operator training.

Innovative and rugged design based on concave holographic grating or axial configurations provide unsurpassed sensitivity and total spatial coverage over the common Raman range from 150 – 3200 cm\(^{-1}\). High spectral resolution versions are also available for more demanding applications, without the drop in efficiency or spectral artifacts found in split or stitched spectral data acquisition (typically found in echelle or split grating designs).

The RPA-AX spectrograph has a unique optical imaging capability, which is ideal for remote, in-situ multiplexing. It has the capability to multiplex up to 64 separate channels of information on the CCD detector – outperforming conventional types of spectrometer designs limited to only 4 tracks of information. Thus, it will aid in lowering the cost of analysis and can, for instance, accommodate increased numbers of fail-safe or reference tracks.

Unique optical multiplexing capabilities of the RPA-AX. Multiplexing of numerous sampling probes can provide advantages in process control and the capital cost of equipment.

<table>
<thead>
<tr>
<th>RPA-HE analyser</th>
<th>RPA-AX</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fixed spectrograph design, rugged and compact construction</td>
<td>Fixed spectrograph design, rugged and compact construction</td>
</tr>
<tr>
<td>532, 633, 785 nm versions (optimised for specific laser)</td>
<td>532, 633, 785, 830 nm versions (optimised for specific laser)</td>
</tr>
<tr>
<td>Full range ~150-3200 cm(^{-1})</td>
<td>Full range ~ 150-3200 cm(^{-1})</td>
</tr>
<tr>
<td>4 track multiplexing (maximum)</td>
<td>High resolution versions available on request</td>
</tr>
<tr>
<td>TE air cooled CCD detector 1024x256 pixel. Specialised high sensitivity CCD Chip for process control</td>
<td>TE air cooled CCD detector 1024x256 pixel. Specialised high sensitivity CCD Chip for process control</td>
</tr>
<tr>
<td>Fibre link connection, 19&quot; rack mounted</td>
<td>Fibre link connection, 19&quot; rack mounted</td>
</tr>
<tr>
<td>Environmental housings NEMA</td>
<td>Environmental housings NEMA</td>
</tr>
</tbody>
</table>
**InduRAM - Versatile process development system**

The InduRAM system is designed for the maximum flexibility required in feasibility testing, process evaluations and general remote monitoring requirements. It is an ideal development tool offering both flexibility and performance.

<table>
<thead>
<tr>
<th>InduRAM</th>
<th>Key Features</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lasers</td>
<td>488, 514, 532, 633, 785, 830 nm versions (dual laser option)</td>
</tr>
<tr>
<td>Technology</td>
<td>Scanning spectrometer – Full range ~100-3600 cm(^{-1})</td>
</tr>
<tr>
<td></td>
<td>High resolution version 1 cm(^{-1}) /pixel</td>
</tr>
<tr>
<td>Sampling</td>
<td>Up to 8 track multiplexing</td>
</tr>
<tr>
<td>Mounting</td>
<td>Fibre link connection, 19” rack mounted, integrated lasers</td>
</tr>
</tbody>
</table>

**Lasers**

Highly stable solid state 785 nm and 532 nm laser sources are standard in most HORIBA Jobin Yvon process systems. The lasers are optimized for fibre-linking and can provide access to typically 100-500 mW of laser energy - thereby maximising the speed of acquisition and multiplexing capabilities. These solid state lasers are robust and have good long term stability, often superior to older laser technologies. They are well established in demanding process applications. Alternative laser sources can also be supplied, and whatever the application HORIBA Jobin Yvon can provide the best solution:

- 532 nm lasers for improved sensitivity or resonance enhancement
- 633 nm lasers for cost effective solutions
- 785 nm lasers for sample fluorescence suppression

**Software**

The IndSPEC module of the software is standard with all process monitoring systems. It offers data acquisition and analysis routines for method evaluation, multi-probe acquisition (depending upon hardware) and component time plot functions to enable a reaction to be followed in real time. Data can be exported into various different formats so that proprietary or in-house data handling software may also be used.

4-20 mA output voltages through the computer interface can enable full integration within process control loops and a distributed control system (DCS).

Autocalibration for external factors such as laser power, temperature, and process ageing can be accomplished through both hardware and software solutions depending upon each specific parameter.
The final component of the Process Raman Analyser is the sampling probe. The purpose of the Raman probe is to efficiently deliver the laser beam to the sample material, and to collect and filter the returning Raman signal. All SuperHead series of Raman probes utilise a single fibre for delivering the laser excitation and another for the signal collection. High efficiency filter designs incorporated within the probe heads offer high signal transmission and effective Raman signal separation. Standard high throughput telecommunication optical fibres (50-200 micron diameters) are used throughout, reducing costs and increasing performance. Low OH fibres and ceramic core cladding are available for increased NIR transmission and maximum thermal stability.

### Probe Features & Specifications:

- 514, 532, 633, 785 nm standard laser wavelengths.
- Holographic notch or dielectric (long life) rejection filter options.
- Immersion optics - 1/4" to 1.4" outer diameter, short or extended focus.
- Hastelloy or 316 SS casings.
- Flange mount options.
- Sapphire or quartz window options.
- Options for up to 400°C and 3000 psi.
- One in / one out fibre design, SMA or FC connectors.
- Optical fibre length of 1m to several 100 m.
- Toughened or armoured cabling and protective enclosures.
- 180° back scattering measurement.
- Non-electrical laser safety options.
- Anti-fouling options.
- Patented Reference Calibration Device (RCD) for data calibration.
- ATEX certified products.

---

**HORIBA Process Technology**

HORIBA JOBIN YVON is part of the HORIBA GROUP – one of the world’s major suppliers of environmental and process monitoring equipment. The combined HORIBA Group companies are leaders in automotive exhaust analysers, ambient air monitoring, continuous emission monitoring for stack gases, process gas and liquid stream monitoring, chemical analysers and water pollution measurement systems.