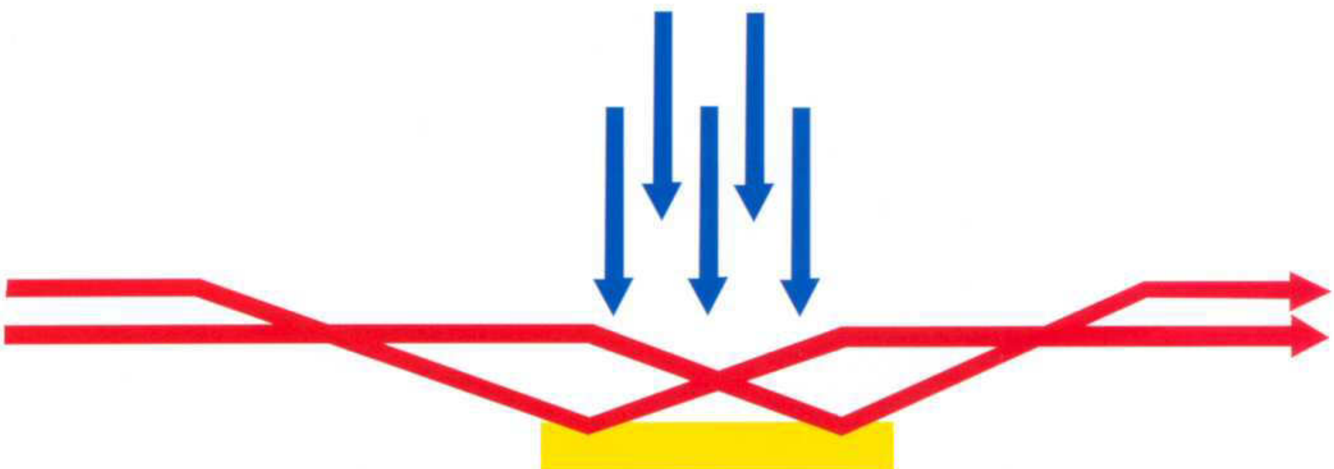
Applications in ***Spectroscopy***

Advancing Polymer Spectroscopy



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Laboratory Equipment

Advancing Polymer Spectroscopy

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The development of new instrumentation can be initiated by a number of different factors. In the case of the #S, the factors involved were our long standing activity in polymer and bio-polymer research through products like the #20[1,2,3], the #2000-A[4,5,6], versions of the #RA4000-EXP technology[7,8] and a request from an academic institution to provide them with new facilities for their polymer research program. They wanted to compare two polymer thin film samples in the same experiment where variable temperature would be one parameter in a controlled environment. They wished to use infrared spectroscopy for the sample analysis utilizing the optical techniques of grazing angle incidence and transmission. Multiple scans over long time periods were required to be taken alternately from each of the two samples and the scanning sequence had to be programmable and so that they could vary this depending on the experiment. These were novel requirements at the time and none of our existing products could be adapted to fulfill this group of functions. However, the experience we had with the #2000-A system would be the basis for the new #S. The #2000-A is optimized for a number of features, in particular to give an exceptional maximum upper temperature capability (950 C) and this aspect determined many of its special design features. Different requirements could now be specified for the new product, the #S, to meet the emerging requirements of polymer research.

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Design Concept

The new #S would be optimized for a reduced upper temperature appropriate to polymers, so 500 C was taken as a sensible upper temperature requirement. This in turn allowed us greater freedom in designing the #S and in particular allowed us to build maximum experimental flexibility into the product concept. Other specifications which would go into the design would be the use of single piece 316 grade stainless steel with no welds or brazes to ensure a broad level of general chemical inertness and inherent quality of construction. So the central chamber and all other parts would be machined from solid blocks of stainless steel, including the formation of sealing surfaces, window installations, gas ports, etc. All window ports were to be fully and easily demountable to allow a free choice of window materials. A pressure specification from Ultra High Vacuum (UHV) to a pressure of 1000 psi was adopted. Like the #2000-A, the specification is for all electrical wiring connections to be external to the chamber. External surfaces of the chamber would be temperature regulated to remain near ambient.

Figure 1.
#S Experimental Chamber showing four of the eight spectroscopy window ports in the single piece chamber body. The installed top plate is formatted for gas inlet/outlet and sensor introduction.

Dual Sample Spectroscopy

The dual sample handling capability is illustrated in Figure 1 which shows the core #S experimental chamber. There are four optical window ports on each side of the chamber. These are paired so that two adjacent ports on one side are optimized for transmission and the next pair of ports on the same side are optimized for grazing angle (Large Angle Reflectance Infrared, LARI). The entire chamber mounts on a computer controlled motorized table and in this way each of the two samples can be alternately shifted into the beam of the FTIR spectrometer. Different types of sample holding probe can be installed from either end of the chamber. In addition to presenting the samples to the optical beam in a specific optical mode, these sample probes carry controlled heating and/or cooling for the sample. Optical components can be introduced from the other end of the chamber to provide a choice of optical modes. The top plate of the chamber is removable and this can be formatted to be experiment specific, for example, providing an Ultra High Vacuum port or gas inlets/outlets or sensor introduction. The external surfaces are temperature regulated by fluid circulation. The base of the chamber features a keyed mounting system so that its orientation is optical mode specific.

Sample Irradiation

Having designed the first version of the #S, we were soon asked for more versions to meet the requirements of other polymer experimenters. A relatively straightforward requirement was to introduce simulated sunlight via an optic fiber during a variable temperature experiment using the LARI optical mode for infrared based polymer structural studies. This was readily achieved by formatting the top plate to accept a suitable optic fiber and provide a gas tight seal.

Raman Spectroscopy

The next request posed a more fundamental problem when we were asked to provide a variable temperature Raman spectroscopy cell. The problem that had to be overcome was how to introduce a relatively

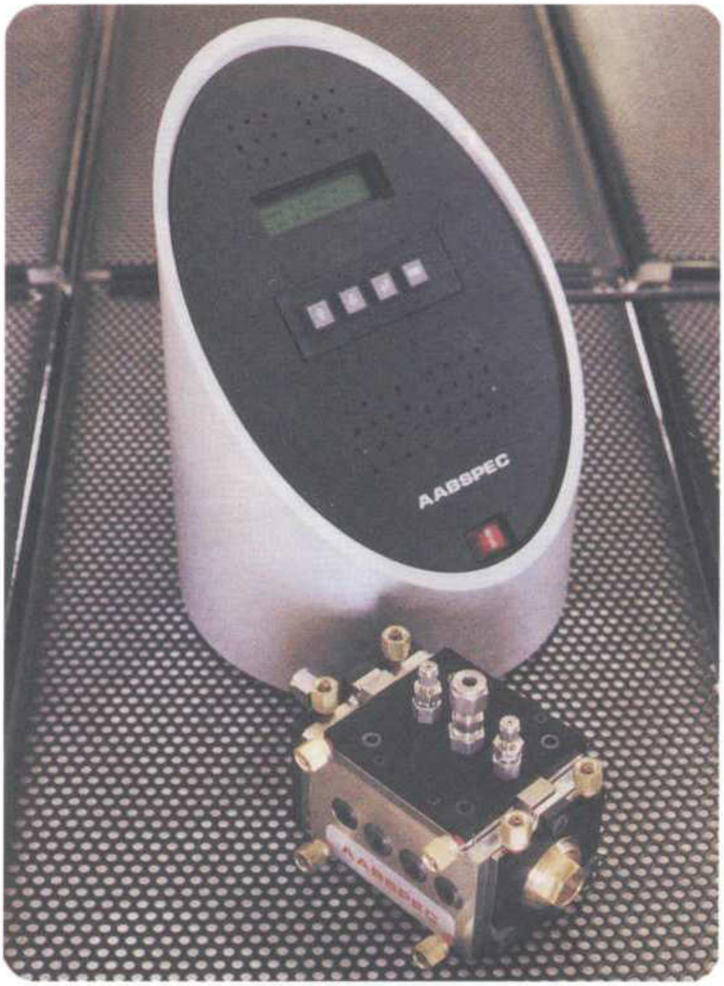

Figure 2. *#S Experimental Chamber fitted with specially designed Raman End Window for variable temperature Raman Spectroscopy under controlled environment conditions. The #STP-6, also shown here, provides highly flexible temperature programming.*

large experimental chamber into the sample compartment of popular Raman spectrometers in combination with the relatively short focal length of the collecting optics and the requirement to maximize the collection of the relatively weak Raman signal. We solved this with a new development[9] which provided a special window design to collect the back-scattered Raman signal and present it to the spectrometer collection optics. The result was the first substantial experimental chamber capable of providing optimized variable temperature Raman spectroscopy in controlled sample environmental conditions. The Raman Spectroscopy format is shown in Figure 2 along with the #STP-6 software based temperature programmer.

Raman Microscopy

Raman microscopy was the next version to emerge. Again, to provide variable temperature in a controlled environment and utilize standard short focal length Raman microscopes imposed difficulties for the design task. Short focal length microscope objectives are important in collecting the maximum Raman signal. A new version of the removable top plate of the #S chamber was designed to incorporate a large viewing window. This allowed short focal length microscope objectives travel in the horizontal plane across the entire region of the sample surface that is located below the viewing window. The Raman Microscopy format is shown in Figure 3.

Further Applications

In addition to the features already outlined, the #S has a number of further applications in such areas as pharmaceuticals. The extremely flexible design enables the #S to be configured to meet a very wide range of experimental conditions and to perform across the entire optical spectrum. In the standard #S versions that are currently available, the temperature range can be from as low as -150 C and up to 500 C. This capability is combined with a pressure range from UHV to 1000 psi. Primarily designed and optimized for polymer research, the flexibility of the #S design gives it a wide range of potential applications for polymer research, pharmaceuticals and many other fields of research.

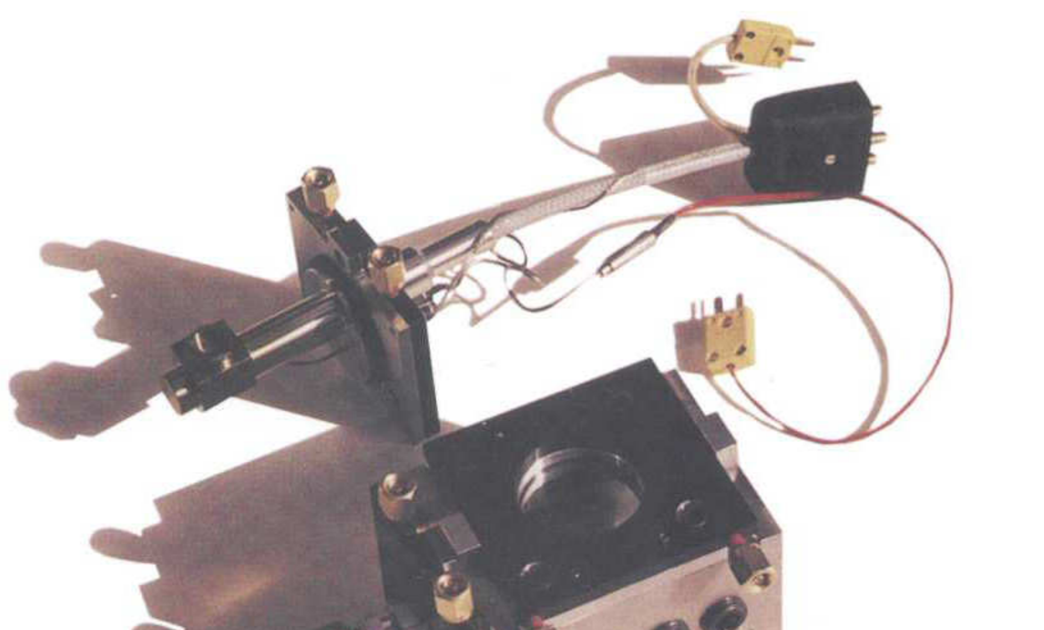

Figure 3. *#S Experimental Chamber fitted with Raman Microscopy top window optimized for short focal length microscope objectives and shown with the sample probe. The large window allows the entire sample surface to be examined under varying experimental conditions.*

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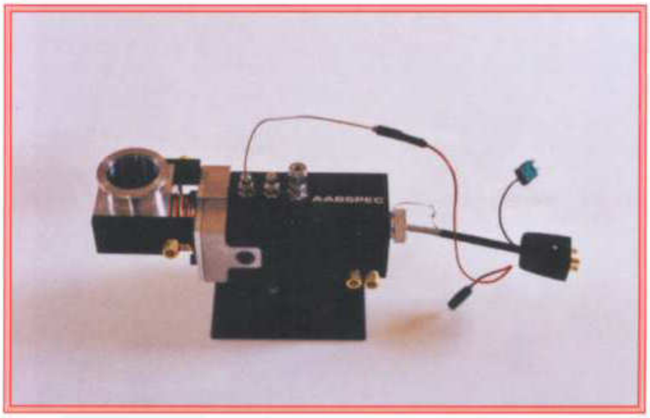
Related Products:

The #S is a highly versatile product, one of our many products, some of which are outlined here. For further product information, see our website www.aabspec.com or e-mail val@aabspec.com for personal attention to your requirements.

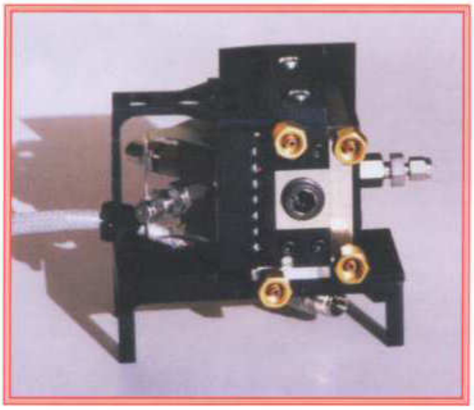
#2000-A

The Aabspec #2000-A is similar to the #S, but gives you a wider temperature and pressure range, so it is ideal for catalysts and other applications requiring very high temperatures. Capable of handling static liquids, powders, solids, thin films, etc., the #2000-A spans the temperature range from -170degC to 950degC and the pressure range from ultra-high vacuum to 2000psi. Optical modes include transmission, specular reflectance, large angle reflectance, Raman.



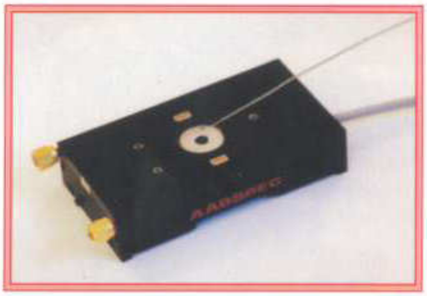
#RA4000-EXP

The Aabspec #RA4000-EXP incorporates unique advanced technology to meet the most demanding conditions in the spectroscopy of fluids. This ultra-low volume flow-through cell operates from ambient to 450degC and to pressures of 4000psi. Variable optical path is optimized to traverse the entire sample, generating transmission-like spectra with easy spectral subtraction. Ideal for process simulation and process monitoring under high temperature + pressure conditions. Full range spectral access FTIR or NIR plus optional Raman.



#M

The Aabspec #M, a compact Microscope Hot Stage offering flexible sample handling. External surfaces are water cooled. The versatile sample holder unit comes with sample temperature read-out. The #M can be controlled by the #STP-6 temperature programmer providing capability for advanced thermal experiments.



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