

wavelength and disperse the Raman-scattered light. These HOEs use Kaiser's proprietary Volume Phase Technology (VPT). The first commercial product using VPT were notch filters used to block the laser line in Raman and fluorescence spectroscopies. The SuperNotch-Plus filter in the HoloProbe provides in excess of 10^6 attenuation of the laser line, yet allows Raman spectra to be collected within 50 cm^{-1} of the laser line for strong Raman scatterers.

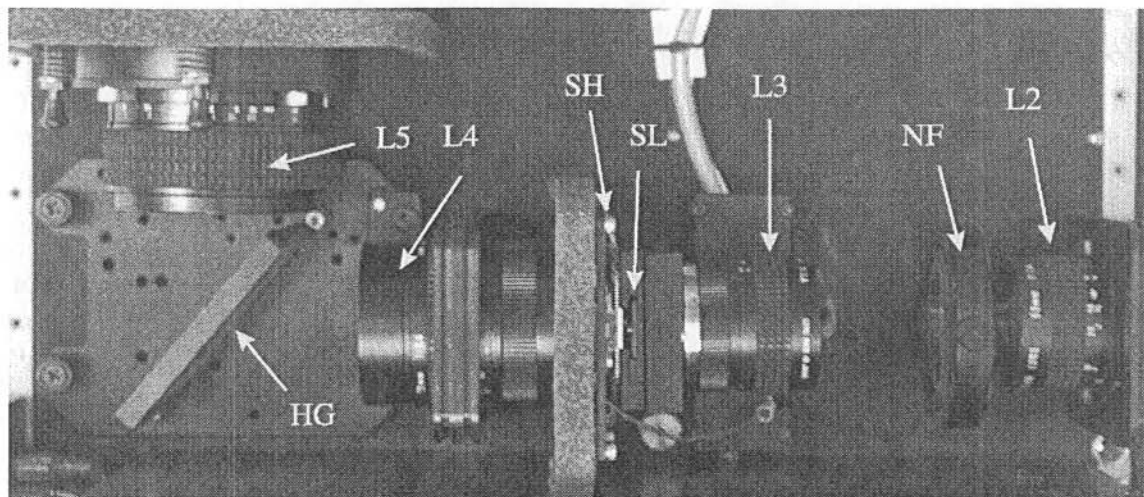
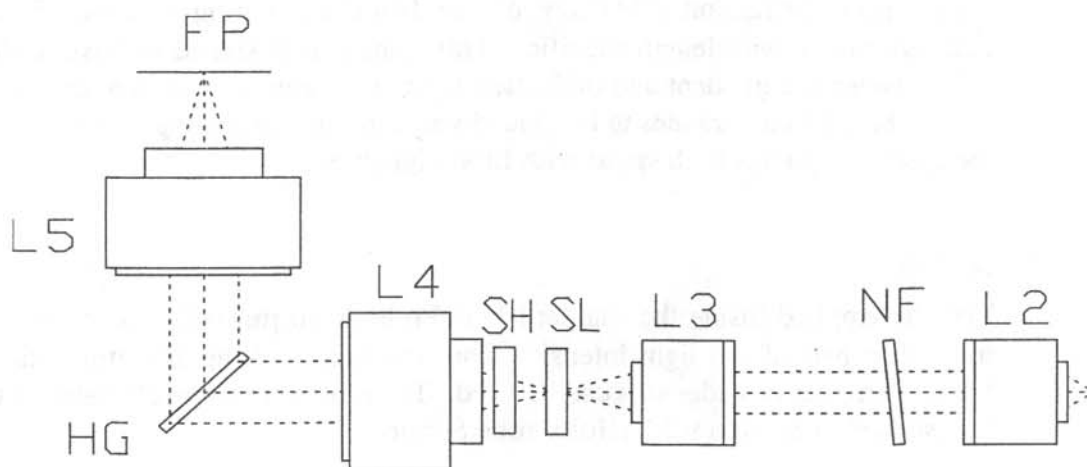


Figure 7.6 Spectrograph stage of the HoloProbe base unit. L2, pre-filter collimating lens; NF, holographic notch filter; SH, shutter; L3, pre-filter focusing lens; SL, spectrograph entrance slit; L4, spectrograph collimating lens; HG, HoloPlex grating; L5, spectrograph focusing lens; FP, spectrograph focal plane for CCD detector.

7.4.3 HoloPlex Grating

The HoloPlex transmission grating disperses and divides the collected Raman light into a low wavenumber shift stripe and a high wavenumber shift stripe on the CCD detector and then stacks the two stripes on top of each other. In this way the HoloPlex grating nearly doubles the spectral coverage of the HoloProbe without reducing its spectral resolution (the two stripes have some spectral overlap, which reduces the spectral coverage below double). The typical peak diffraction efficiency of the HoloPlex grating is about 65%, although this is wavelength-specific. This grating is designed to have a 90° fold between the incident and diffracted light at the central wavelength. This allows the f/1.4 camera lens to be placed very close to the grating. As a result, the spectrograph has f/1.8 speed with little vignetting.

7.4.4 Slit

The slit supplied inside the standard HoloProbe is 50 μm wide. It transmits more than half of the light intensity from the image of the 100 μm optical fiber. Narrower or wider slits can be used. The procedure for exchanging slits is described in Section 9.3.2 (HoloProbe Service).

7.5 Fiber Optic Probe

In order to take Raman spectra remotely over more than a few meters of optical fiber, it is necessary to filter the light at the end of the fiber, both before and after it strikes the sample. As the laser light travels through the fiber towards the sample, a large amount of Raman signal and sometimes fluorescence is generated in and efficiently collected by the fiber. It is thus necessary to separate the laser light from the background before illuminating the sample so that the fiber background does not overwhelm the Raman signal. Similarly, it is necessary to filter out the laser light that is either reflected back into the collection lens or Rayleigh (elastically) scattered by the sample before it travels down the collection fiber, or else it could generate a significant amount of Raman and fluorescence background.

7.5.1 General Description

Light Path. The fiber-optic probe head, pictured in Figure 7.7, performs both of these optical filtering functions. Laser light exits the excitation fiber.

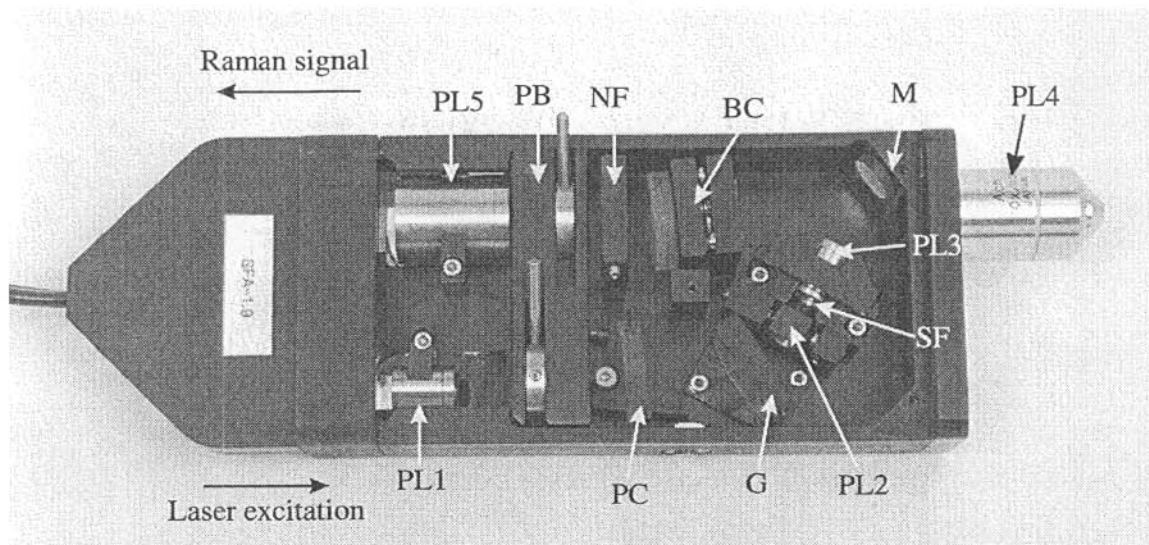
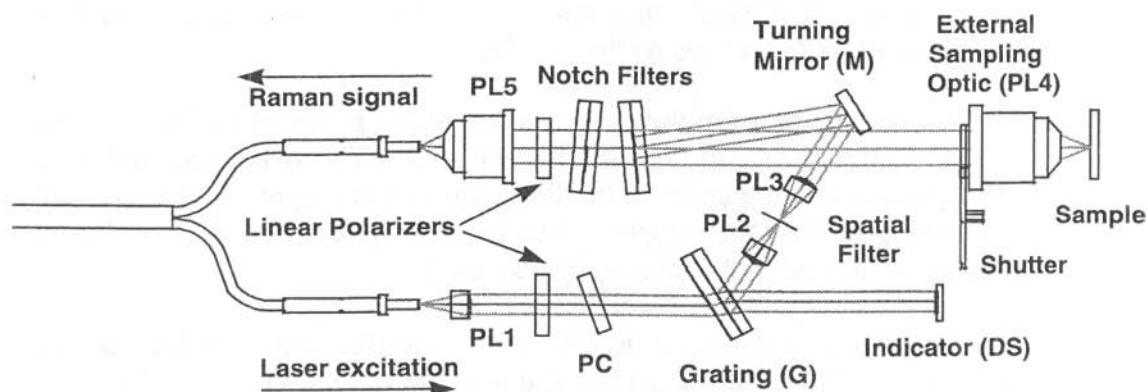


Figure 7.7 Optical configuration of fiber-optic probe head, showing lenses one through five (PL1-PL5), laser power control adjustment (PC), grating (G), diffuse screen indicator (DS), probe head shutter, spatial filter (SF), turning mirror (M), beam combiner (BC), notch filter (NF), and the block containing the linear polarizers (PB).

The majority of the laser light and some of the fiber's Raman and fluorescence background are diffracted by the grating, G, while a small fraction of laser light is undiffracted and passes through the grating. This undiffracted light illuminates a diffusing screen, DS, visible from the outside of the housing, thereby serving as an indicator showing when the probe head is in use. A dispersed image of the excitation fiber is formed at the spatial filter, SF, and only the laser wavelength is passed. The laser light is then folded into an

objective lens, PL4, by a turning mirror, M, and a notch filter acting as a beam combiner, BC, and focused on the sample.

Light backscattered from the sample is collimated by the objective lens, PL4, and passes through a pair of notch filters, BC and NF, providing several orders of magnitude of attenuation of the Rayleigh-scattered light. The inelastically scattered light from the sample is then focused by lens PL5 into a collection fiber and delivered to the imaging spectrograph.

The fiber-optic probe head uses no electricity that could be hazardous in explosive environments. It is packaged in a compact housing 8" x 3" x 1.5".

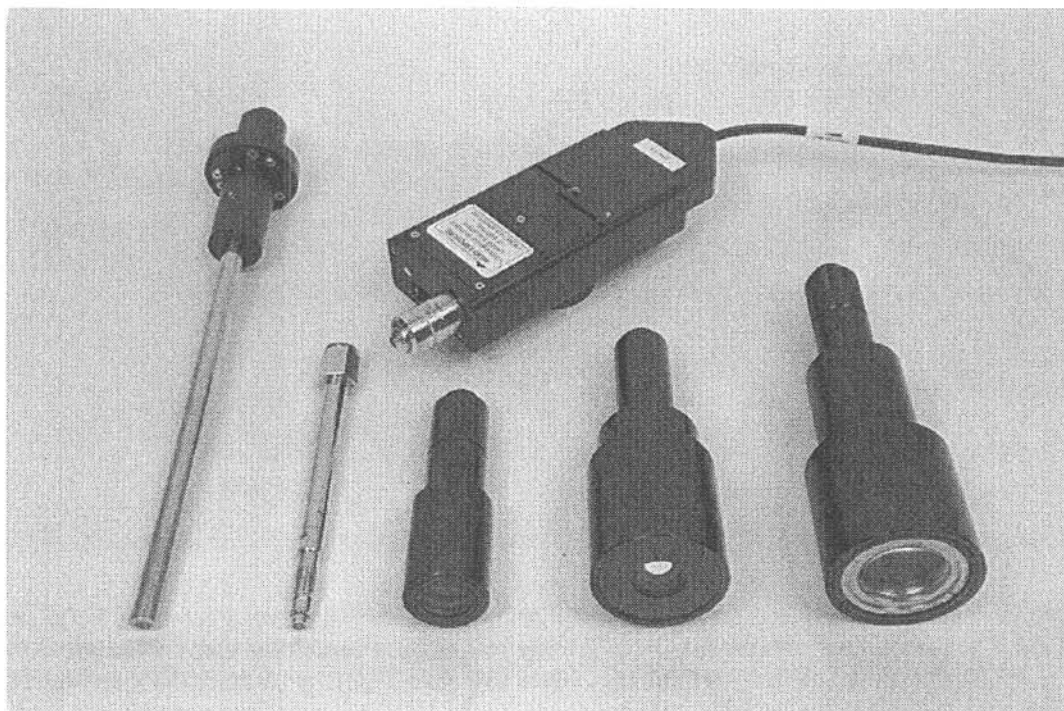


Figure 7.8 Probe head (with a 10X objective) and optional lens attachments: immersion optic f/2, Dynisco® threaded extrusion optic, non-contact optic f/2 with 2.5" working distance, non-contact optic f/1.8 with 1.3" working distance, and non-contact optic f/10 with 17" working distance.

7.5.2 Probe Head Accessory Optics

The probe head is shown in Figure 7.5 and Figure 7.8 with a microscope objective, providing a 0.1" working distance. Other lenses are available to provide working distances up to 17". Additionally, a probe extension with a small lens at the end of several inches of collimated space is available. This probe configuration is ideal for looking through windows into process streams. The probe head can be mounted using one or two of the 1/4-20 tapped holes on the bottom of the probe head cover. Over-tightening the screw in the center 1/4-20 hole can cause probe misalignment.

7.5.3 Benefits of the Probe Head Design

The fiber optic probe head images the laser excitation light from a 50 μm optical fiber onto the sample and images the sample emission onto a 100 μm optical fiber. These two individual fibers connect the fiber optic probe head to the HoloProbe base unit. The use of one collection fiber, rather than many collection fibers, significantly reduces the cost of long (>100 meters) fiber optic cables without reducing the amount of light collected from the sample. It also makes better use of the CCD detector area. Since only about 100 μm of slit height is illuminated, several probe heads can be monitored by a single HoloProbe base unit.

Confocal Aperture. The input end of the collection fiber also acts as a confocal aperture. Out-of-focus light is therefore discriminated against. Confocal operation enhances the HoloProbe's ability to measure Raman spectra of packaged samples without interference from the packaging material.

Silica Raman Removal. Laser light traveling through a silica optical fiber generates silica Raman emission. If this emission were to reach the spectrograph, it could obscure the Raman spectrum of the sample. This problem is especially severe when long lengths of optical fiber are used. The fiber optic probe head used by the HoloProbe removes the silica Raman light from the laser light after the light exits the excitation fiber and before it reaches the sample. The fiber optic probe head also removes laser light from the sample emission before it reaches the collection fiber. As a result, silica Raman is not a problem for the HoloProbe, even when very long optical fibers are used.

Collection Lens Flexibility. The collection lens mount on the fiber optic probe head is threaded for standard microscope objective lenses. A wide range of commercially available lenses can therefore replace the collection

lens (PL4 in Figure 7.7) in order to meet the requirements of different applications. Standard lenses for the fiber optic probe head available from Kaiser Optical Systems, Inc. have working distances ranging from less than 1 mm up to 20 inches. Custom lenses can also be supplied. (See Figure 7.8) Contact Kaiser Optical Systems, Inc. for more information.

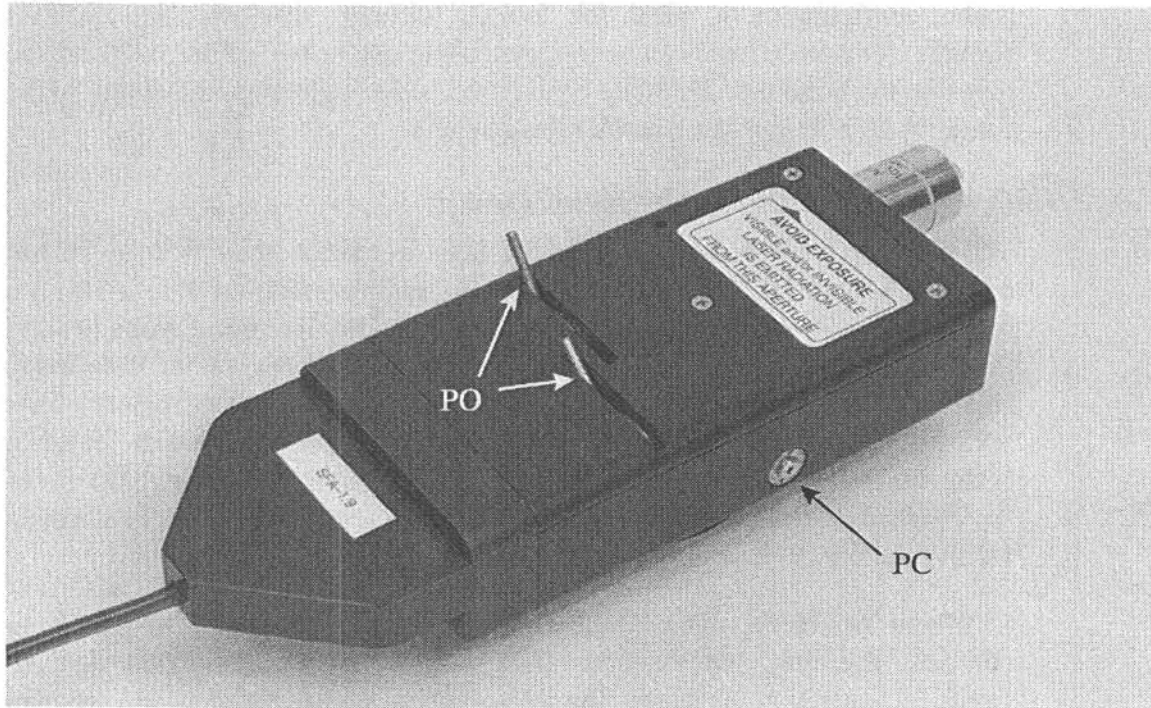


Figure 7.9 Dual polarization adjustments (PO), and laser power control adjustment (PC).

Adjustable Power Control. The laser power intensity output from the probe head can be adjusted by inserting a 5/32" allen wrench into the hole labeled PC in Figure 7.9 and turning slightly. This changes the angle of the flexure mount holding the holographic filter, which in turn changes the laser wavelength optical density.

7.5.4 Dual Polarizer Probe Head

Polarization Selector. The probe head is equipped with dual rotation polarizers to allow the user to control both the excitation and collection polarization vectors. Rotating both levers to the right—clockwise—(as seen looking down on a probe head pointing away from the user) orients the excitation laser light polarization vector parallel to the surface of the probe.