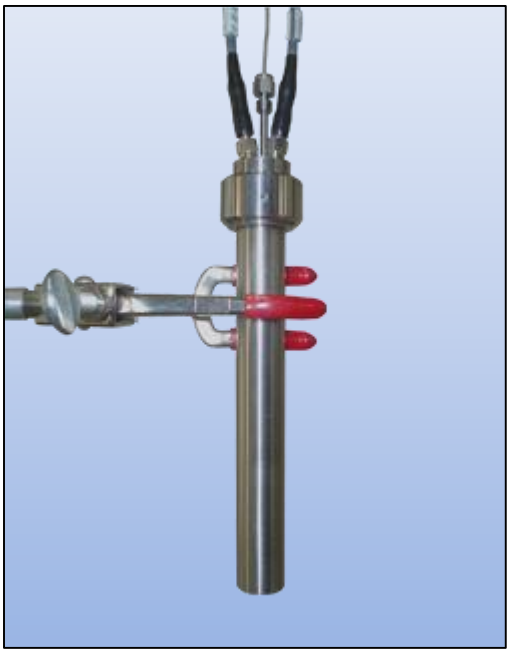
Diamond Probe

The Diamond Probe is a fiber optic ATR accessory designed for analyzing a wide range of samples in the mid-infrared. Sample types include liquids, pastes, slurries, powders, and solids. Samples can be located up to 1.5 meters from the spectrometer. The single reflection 45° diamond ATR element is Harrick's crowned hemisphere, mechanically sealed in 316 stainless steel. The Probe has a diameter of 19 mm and an immersion depth of 125 mm. It is separately purgeable. An MCT detector-equipped FTIR spectrometer is required. The Probe is especially useful in monitoring liquid reactions. Temperatures up to 140 °C and pressures up to 0.55 MPa can be tolerated. Applications include fermentation, crystallization, and polymerization. The Diamond Probe requires that a fiber optic coupler, such as the Harrick FiberMate2™, be installed in the spectrometer. Also, the Probe and FiberMate2™ must be optically connected via two SMA terminated fiber optic cables such as those offered by Harrick.



APPLICATIONS

- Analysis of the widest variety of samples including liquids, pastes, slurries, powders, and solids.
- Analysis of organics and inorganics.
- Analysis of hard solids.
- Reaction and drying process monitoring.
- Quantitative and qualitative analysis.

FEATURES

- Single-reflection 45° diamond ATR sampling outside the FTIR spectrometer.
- Wavelength range: 6500 to 600 cm^{-1} (6500 to 1700 cm^{-1} with chalcogenide fibers; 2000 to 600 cm^{-1} with silver halide fibers).
- Distance from spectrometer: 1.5 m.
- Construction: 316 stainless steel.
- Diameter: 19 mm.
- Immersion depth: 125 mm.
- Purge capability.
- Designed for use with MCT detectors.
- SMA 905 compatible.
- Operable from room temperature to 140 °C.
- Operable from ambient pressure to 0.55 MPa (80 psi).
- Use with Harrick FiberMate2™ fiber optic coupler and fiber optic cable sets.
- Optional Diamond Probe Heater and temperature controller available for taking background spectra at elevated temperatures.

INCLUDES

- Diamond ATR probe.

ORDERING INFORMATION

	CATALOG NO
Diamond Probe	DMP-PRB
Near/Mid- IR Fiber Set (CIR fibers, 6500 to 1700 cm^{-1}.)	OMN-F-CIR
Mid-IR Fiber Set (PIR fibers, 2000 cm^{-1} to 600 cm^{-1})	OMN-F-PIR
FiberMate2™	FM2-XXX

OPTIONS

Diamond Probe Heater (requires temperature controller)	DMP-PHT-3
Replacement Heater Assembly (heaters with connector)	DMP-HTR
Replacement Heater	HTRS-16
Temperature Controller, 110V input, 24V output. Includes USB adapter.	ATK-024-3
Temperature Controller, 220/240V input, 24V output (CE marked). Includes USB adapter.	ATK-024-4

Harrick Scientific Products, Inc.
141 Tompkins Ave, 2nd Floor, Pleasantville NY 10570
Ph: 800-248-3847 or 914-747-7202, FAX: 914-747-7209, web site: www.harricksci.com, e-mail: info@harricksci.com

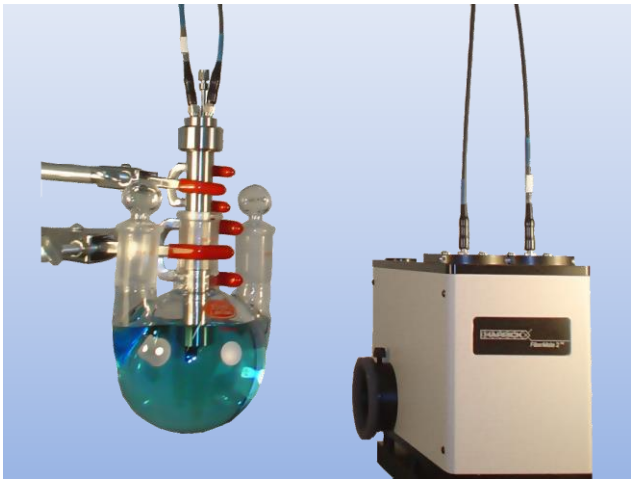


Figure 1. Diamond Probe installed on the FiberMate2™.

The Diamond Probe is a fiber optic ATR accessory designed for analyzing liquids, pastes, slurries, powders, and solids in the mid-infrared region of the spectrum. Samples can be located up to 1.5 meters from the spectrometer. The single reflection 45° diamond ATR element is Harrick Scientific's patented Monolithic™ diamond crowned hemisphere, mechanically sealed in the 316 stainless steel body of the probe. The strength and scratch resistance of the diamond, as well as its crowned geometry, allow its use on hard solids. The corrosion resistance of the stainless steel and diamond allow use with a wide range of chemicals. The Probe has a diameter of 19 mm and an immersion depth of 125 mm. It is separately purgeable. An MCT detector-equipped FTIR spectrometer is required.

The Diamond Probe is especially useful in monitoring liquid reactions. Temperatures up to 140 °C and pressures up to 0.55 MPa (80 psi) can be tolerated. The flexible nature of fiber optics allows complete versatility in setting up such applications, which now can be constructed outside the FTIR sample compartment. Since the Diamond Probe allows in situ analysis, there is no need for elaborate pumping schemes and the problems they engender. The Probe is in the reaction providing real-time tracking of reactants, intermediates, and products. Example applications include fermentation, crystallization, and polymerization. Since your existing FTIR is employed, there is no need to purchase an additional, special purpose spectrometer for reaction studies. And, when not being used for such work, your FTIR is free for general use.

The Diamond Probe requires that a fiber optic interface, such as the Harrick FiberMate2™, be installed in the FTIR spectrometer. Also, the Diamond Probe and FiberMate2™ must be optically connected via two SMA 905 terminated fiber optic cables. Such cables are available from Harrick. Two sets of two cables each are offered. Each set is suitable for a different wavelength region. The chalcogenide set is used in the 6500 to 1700 cm^{-1} region. The silver halide set is used in the 2000 to 600 cm^{-1} region. Note that the diamond material itself has crystal lattice bands which limit the signal-to-noise in the 2300 to 1850 cm^{-1} region. An optional Diamond Probe Heater and compatible temperature controller are available for taking background spectra at elevated temperatures.

Several examples are shown below using the Diamond Probe with the FiberMate2™ (see Figure 1). For these experiments, the FTIR spectrometer, equipped with an MCTA detector, was set for 256 scans at 8 cm^{-1} resolution.

Distinguishing different plastic types has become a major interest for recycling and disposal. Mid-infrared spectra can be used to fingerprint plastic samples to obtain unambiguous identifications. The isolation of nylon, a polyamide, is of particular interest, since hydrogen cyanide is formed when this plastic is incinerated. Figures 2 and 3 show the spectra of nylon 6 and white Delrin®, a polyacetal. Although both of these materials are similar in appearance, the spectra are clearly unique. The peaks at 1635 and 1536 cm^{-1}, characteristic of amides, are present only in the nylon spectrum. The bands at 1087 and 893 cm^{-1} are present only in the Delrin® spectrum.

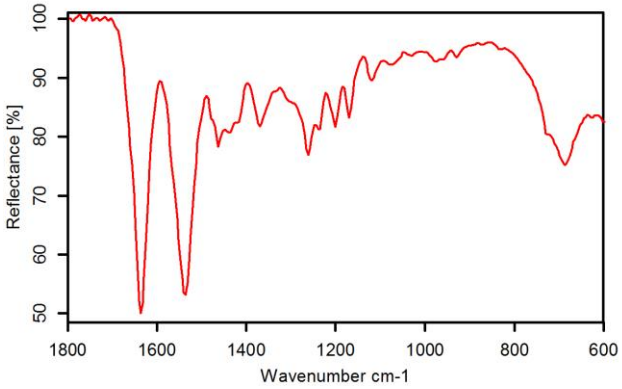


Figure 2. Nylon 6 Spectrum.

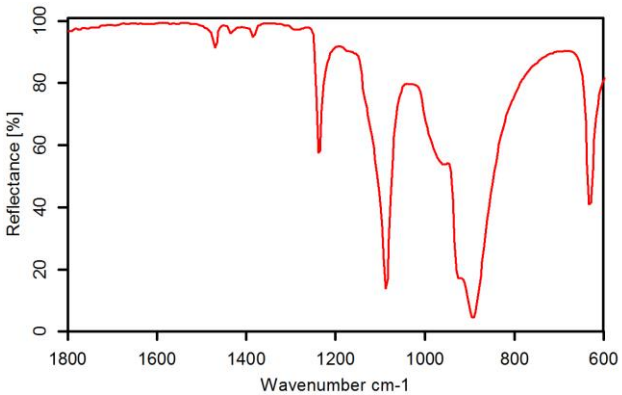


Figure 3. Delrin™ Spectrum.

Incoming raw material inspection is a standard practice in many industries. The Diamond Probe can be used to quickly verify material identification without any sample preparation. Figure 4 shows the two characteristic peaks at 1205 and 1150 cm^{-1} of powdered PTFE, used as a molding compound and as a lubricant. Figure 5 reveals the distinctive spectrum of

anhydrous D-lactose, used by the pharmaceutical industry in tablet formulations.

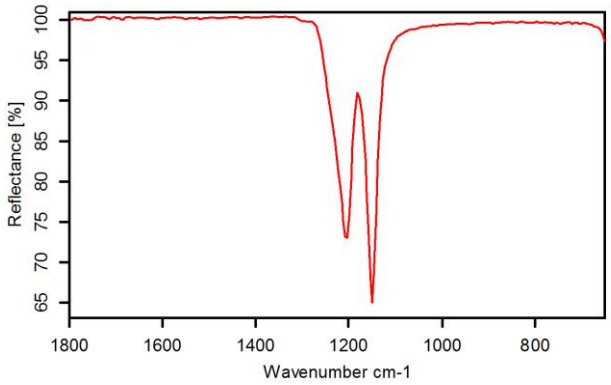


Figure 4. PTFE Spectrum.

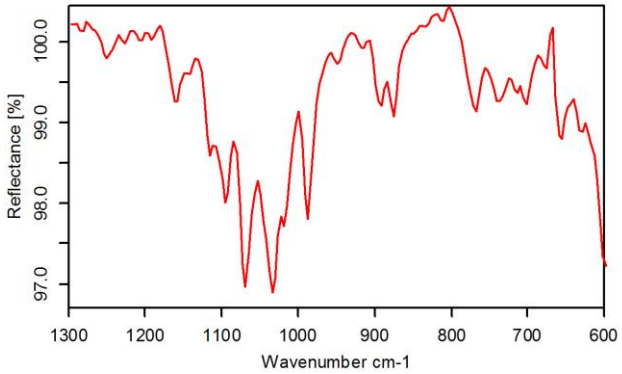


Figure 5. Lactose Spectrum.

The absorbance peaks of many liquids decrease in intensity and shift wavelength maxima as the temperature increases. Especially in quantitative analysis, it is important to understand the thermal behavior of spectral features of a given chemical species. The decrease in intensity and the shift in wavelength will be different for different peaks, even within the same compound. Figure 6 shows the spectra of canola oil at room temperature and 150 °C.

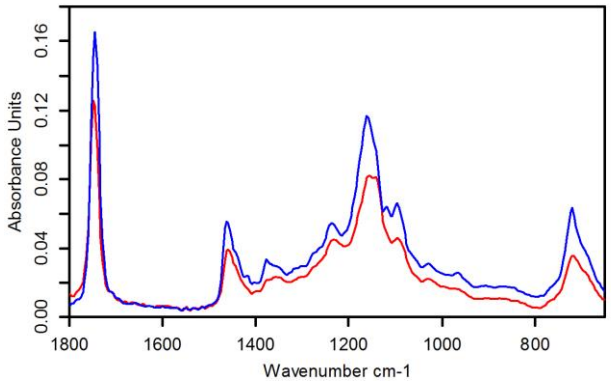


Figure 6. Canola Oil Spectra at Room Temperature (upper) and 150 °C (lower)

Reaction monitoring in liquids is a common application. Less common is the monitoring of reactions taking place in pastes and slurries. In one experiment, the curing of an RTV silicone was monitored for a period of 60 minutes, at 15 minute intervals. The overlapping spectra for 15 to 60 minute intervals indicate that the material was cured within the first 15 minutes of exposure to air. In that time, the peak at 787 cm^{-1} grew and the peaks at 950, 1225, 1372, and 1741 cm^{-1} disappeared.

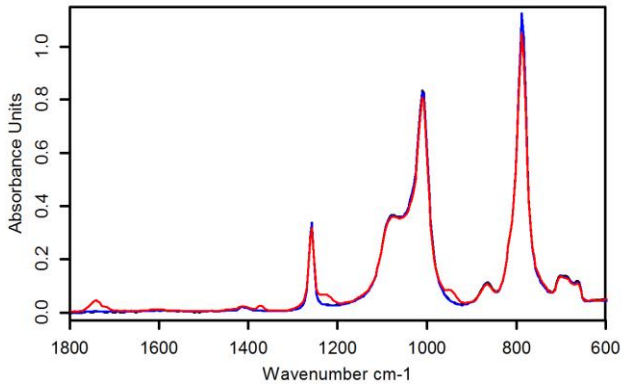


Figure 7. RTV Silcone Cure at 0, 15, and 60 Minutes.