Analysis of Materials in the Far-IR Spectral Region by ATR/FTIR

The far infrared (far-IR) can be an important spectral region for material analysis. Applications include studies of lattice vibrations in crystals, anti-ferromagnetic resonance and energy gap measurements in superconductors.[1] Conventional methods for sample analysis for the far-IR spectral region include the preparation of a film, a mull or a pellet using far-IR transparent materials. This process is, of course, tedious and adds the possibility of contamination during sample preparation.

The use of attenuated total reflectance (ATR) techniques has increased dramatically in recent years due to the desire to reduce sampling time. By using ATR the sample can be simply placed into contact with the ATR crystal, pressure applied in the case of solid materials and its spectrum is measured. In other words, sample preparation is generally not required for ATR analysis. Solids, liquids, pastes, films and gels are easily measured by ATR. In the far-IR spectral region, ATR provides an additional benefit – the increase in band absorbance due to the wavelength dependence upon depth of penetration.[2] For example, the depth of penetration of the IR beam in ATR is 5 times greater at 200 cm^{-1} (50 microns) than it is at 1000 cm^{-1} (10 microns). Therefore, the relatively weak IR band absorbance we expect in the far-IR spectral region is “magnified” by analysis using ATR.

Analysis in the far-IR spectral region requires use of specialized FTIR optics – a minimum of compatible beamsplitter and detector are required. Depending upon the chosen combination of FTIR optics, one may observe spectral features beyond our traditional mid-IR spectral range cutoff at 400 cm^{-1} to below 50 cm^{-1} (200 microns). For the ATR accessory, diamond is an ideal crystal for the far-IR spectral region due to its transparency to 2 cm^{-1} (5 mm).[3]

Figure 1. GladiATR accessory with diamond crystal plate and high pressure clamp.

Experimental Conditions

A PIKE Technologies GladiATR (Fig. 1) equipped with diamond crystal plate was used for the sample analysis.[4] The GladiATR utilizes all reflective optics and when fitted with the diamond crystal plate, its available spectral range spans the mid and far-IR spectral regions. About a milligram of sample was placed over the diamond crystal and force was applied to the sample by rotation of the pressure clamp to its click-stop release.

The FTIR spectrometer was equipped with a far-IR beamsplitter and polyethylene windowed DTGS detector. The standard mid-IR source was used. Spectra were collected at 4 cm^{-1} spectral resolution utilizing a 10 minute data collection time. The FTIR spectrometer was purged with dry nitrogen gas.

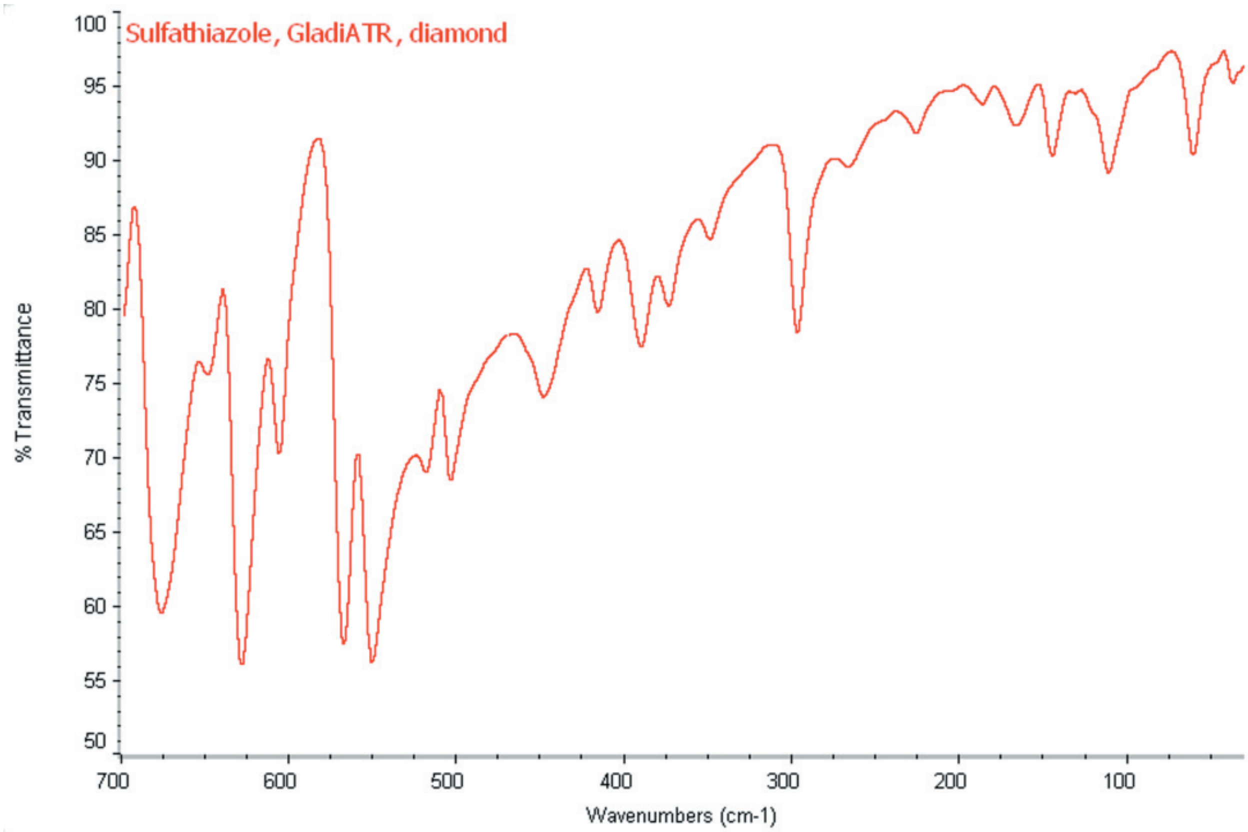

Figure 2. Sulfathiazole powder measured by ATR/FTIR using the GladiATR accessory with diamond crystal plate.

Results and Discussion

Figure 2 shows a spectrum of sulfathiazole powder measured on the GladiATR accessory in the far-IR spectral region from 700 to 30 cm^{-1}. High signal-to-noise ratio (SNR) spectral data are shown for this sample and these optical parameters using the GladiATR accessory with diamond ATR crystal.

Summary

High quality far-IR spectral data are easily produced using the GladiATR accessory with diamond crystal plate when this accessory is used in combination with an FTIR equipped with far-IR optics.

References:

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3. Optical Materials for Infrared Spectroscopy, D. Warren Vidrine, Handbook of Vibrational Spectroscopy, Vol. 1, Chalmers, Griffiths, Eds., Wiley & Sons, 2002.
4. GladiATR product data sheet, PIKE Technologies, 2007. www.piketech.com