

Capabilities and Performance of the iD7 ATR Accessory

Key Words

iD7 ATR Accessory, All-reflective Optics, Attenuated Total Reflectance (ATR), Diamond Crystal, FT-IR, Ge Crystal, ZnSe Crystal

Background

The Thermo Scientific™ iD7 ATR is a single-bounce attenuated total reflectance (ATR) device that accommodates interchangeable diamond, zinc selenide (ZnSe) and germanium (Ge) crystal plates. It optimizes the material characterization capabilities of the Thermo Scientific™ Nicolet™ iS™5 FT-IR spectrometer when analyzing solid or liquid samples (Figure 1). This technical note describes features and performance characteristics of the iD7 ATR demonstrating how FT-IR analysis can be quickly and easily achieved.

The Nicolet iS5 FT-IR is a high performance, compact spectrometer commonly used in academic teaching and industrial QC labs. Its low cost and small footprint make it ideal for organizations with limited budgets and space that still require high-quality FT-IR data for routine analysis. Utilizing Thermo Scientific™ OMNIC™ software, users can collect, process, and analyze spectra; create automated standard operating procedures; and generate reports with ease. The iD7 ATR, with its monolithic diamond crystal and all-reflective optics, provides high quality spectra across the full spectral range of the Nicolet iS5 spectrometer in a robust and easy-to-use device.



Figure 1:
Nicolet iS5 FT-IR Spectrometer with iD7 ATR accessory

ATR Technique

Attenuated Total Reflection (ATR) is the most popular sampling technique for FT-IR analysis. Developed in the 1980's, ATR has supplanted transmission analysis for many applications due to its ease of use and minimal sample preparation. To work properly, the crystal must have an index of refraction greater than that of the sample. In addition, the angle of incidence of the infrared light must be small enough so it reflects off the interface between the sample and crystal rather than transmitting into the sample (Figure 2). The depth of penetration into the sample is on the order of a few micrometers. Therefore, it is critical for samples to be in intimate contact with the ATR crystal. For solid samples, this requires pressing the sample into the crystal using a pressure device.

Common crystals used in ATR include ZnSe, Ge and diamond. ZnSe is a good and relatively inexpensive crystal that can be used down to 550 cm^{-1} , but it is somewhat soft and tends to scratch. Ge is much harder so is more robust than ZnSe and is useful to about 650 cm^{-1} . Its high index of refraction limits the sample depth of penetration which can be advantageous for highly absorbing materials, such as carbon-filled polymers. Diamond is the most highly desired crystal because it is both chemically and physically robust. It has a wide spectral range, down to 200 cm^{-1} or less, depending on the range of the instrument in which it is used. Historically, diamond's cost has limited its use. A popular alternative has been the use of thin diamond films laminated over ZnSe or other crystals to take advantage of the ruggedness of diamond while minimizing the expense. Disadvantages to this approach include limiting the spectral range because of the ZnSe crystal, reducing the strength due to the very thin diamond, and the potential for delamination of the diamond film from the ZnSe base layer. Reductions in the cost of synthetic diamonds coupled with the unique design of the iD7 ATR offer the advantages of laminated diamonds (lower cost, higher energy throughput) while avoiding the disadvantages of many all-reflective diamond accessories (higher cost, prominent diamond spectral features). These points will be discussed in more detail in this note.

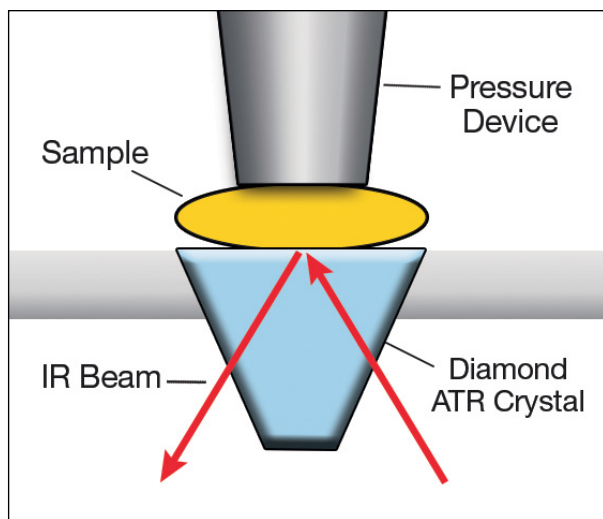


Figure 2: ATR technique: The incoming light is focused through the crystal onto the sample/crystal interface. Some light is absorbed by the sample. Unabsorbed light is reflected out of the ATR crystal to the detector.

Features of the iD7 ATR Accessory

The iD7 ATR is well-suited as a general single-bounce accessory for the Nicolet iS5 spectrometer. Table 1 lists some of the features and benefits that make the iD7 ATR an attractive choice.

Features	Benefits
All reflective optics	Full spectral range, more spectral information
Solid diamond crystal	Superior chemical and physical resilience
Interchangeable crystals	Analyze virtually any sample
Smart Chip technology	Automatic accessory set up and recognition

Table 1: Features and Benefits of the iD7 ATR accessory

A key part of the unique design of the iD7 ATR is all-reflective optics. These highly reflective surfaces minimize the energy loss associated with passing the beam through windows or focusing optics. The iD7 ATR is compatible with multiple crystal types, allowing flexibility in choosing the right crystal for a particular application. Crystal plates are available separately and are easily exchangeable. Of particular value is the monolithic diamond crystal that is extremely robust and provides the benefits of diamond without the need for a ZnSe focusing lens. The crystal design virtually eliminates diamond residual bands from sample spectra which are typically prevalent in many monolithic diamond accessories. Two types of diamond plates are available – Anti-reflectance (AR) coated and Extended Range. The AR-coated diamond provides superior energy throughput, far above other monolithic diamond accessories available on the market. The Extended Range diamond provides a wider spectral range, yet still offers very good energy throughput due to the iD7 ATR optical design. The slip-clutch pressure tower clicks audibly when the maximum pressure is applied, ensuring consistent results for solid samples. Two pressure tips and a volatiles cover are included with the accessory (Figure 3). The standard flat steel anvil is articulated to conform to uneven surfaces of solid samples. The pellet tip has a cavity to prevent powders or polymer pellets from slipping from under the tower during application of pressure. The volatiles cover reduces the evaporation of volatile liquids. Because the iD7 ATR is an integrated Nicolet iS5 accessory, it utilizes the same Smart Chip technology found in other iD accessories. This allows the instrument to recognize the accessory and automatically load the appropriate experiment parameters, eliminating the need to redefine them when swapping from one accessory to the next.



Figure 3: Pressure tower tips and volatiles cover

Performance of Diamond iD7 ATR

The monolithic diamond iD7 ATR represents an advancement in performance over previously available ATR accessories. It offers advantages compared to other all-diamond ATR accessories with higher energy throughput and a price comparable to laminated diamond accessories. Energy throughput is determined by taking a background measurement without the accessory in place, then placing the accessory in the instrument and collecting a sample spectrum. Throughput is the ratio of energy transmitted with the accessory in place compared to the open beam path. The iD7 Diamond ATR shows exceptionally good throughput for an all-diamond accessory, even in the region of the strongly absorbing diamond lattice bands between 2200–2000 cm^{-1} (Figure 4). The AR-coated diamond has significantly higher throughput compared to typical monolithic diamond ATR accessories. Even the extended range diamond still has relatively high throughput owing to the optical design of the accessory.

The all-reflective optics and the lack of ZnSe focusing lenses allows the use of the full spectral range of the Nicolet iS5 FT-IR spectrometer. Figure 5 shows the low-end cutoff of the iD7 ATR diamond crystals demonstrating that analysis is possible to 400 cm^{-1} or below. By contrast, Figure 5 shows a diamond/ZnSe laminate with spectral cutoff around 520 cm^{-1} .

Summary

The new iD7 ATR is a high-quality accessory enabling the full capabilities of the Nicolet iS5 spectrometer. The ATR crystal options allow flexibility to provide the solution needed for a large range of applications. ZnSe is a good all-around crystal for softer materials, while Ge is available for harder materials that may be highly absorbing. The monolithic diamond in either AR-coated for higher energy throughput or extended range versions are the best choice for extremely hard or caustic materials or where the complete spectral range of the instrument is needed. The all-reflective optics allow the highest throughput possible without spectral range losses associated with using focusing lenses or elements. These features combine to provide rapid data collection of high signal-to-noise spectra with the most complete mid-infrared spectral information.

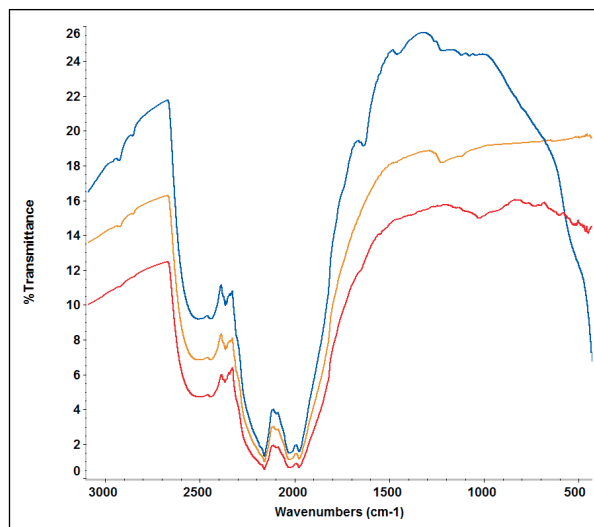


Figure 4: Example of typical throughput for iD7 ATR monolithic diamond AR-coated crystal (blue trace), extended range diamond crystal (gold trace), and another monolithic diamond accessory (red trace).

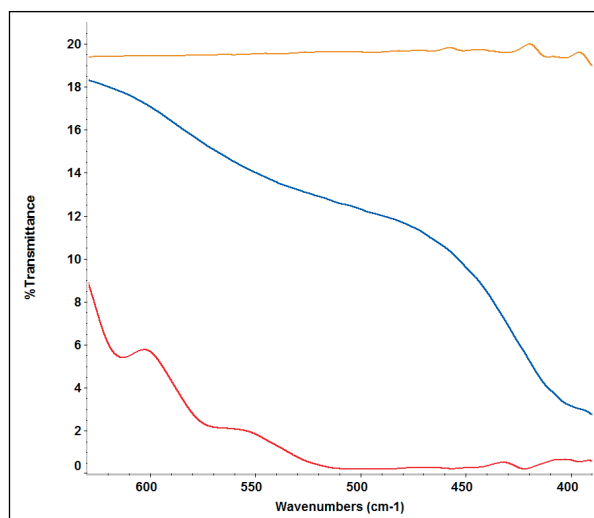


Figure 5: Comparison of the low wavenumber %T energy cutoff for the iD7 ATR extended range diamond (gold trace), AR-coated diamond (blue trace) and another laminate diamond accessory (red trace).

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