

Advantages of Fourier-Transform Near-Infrared Spectroscopy

Key Words

- Connes Advantage
- Dispersive NIR
- Fellgett Advantage
- FT-NIR
- NIR

Introduction

A near-infrared spectrum ($12000 - 4000 \text{ cm}^{-1}$) is composed of combination and overtone bands that are related to absorption frequencies in the mid-infrared region. These combination and overtone bands correspond to the frequencies of vibrations between the bonds of the atoms making up the material. Because each different material is a unique combination of atoms, no two compounds produce the exact same near-infrared spectrum. Therefore, near-infrared spectroscopy can result in a positive identification (qualitative analysis) of each different material. In addition, the size of the peaks in the spectrum is a direct indication of the amount of material present. With modern software algorithms and statistical treatments, NIR spectroscopy is an excellent tool for quantitative analysis, offering a practical alternative to time-consuming wet-chemical methods and liquid chromatographic techniques. NIR has become a versatile technique with no sample preparation, decreased costs and analysis time, and the ability to sample through glass and packaging materials.

Fourier transform near-infrared (FT-NIR) spectroscopy was developed in order to overcome the limitations encountered with dispersive NIR instrumentation. Using a prism or moving grating, the dispersive instrument separates the individual frequencies emitted from the near-infrared source. A detector then measures the amount of energy at each frequency that has passed through the sample. To reduce the analysis time per sample, a method was needed to measure all of the infrared frequencies simultaneously, rather than individually. The FT-NIR spectrometer produces a unique type of signal called an interferogram that has all of the infrared frequencies “encoded” into it. The signal can be measured very quickly, with the collection of multiple scans per second. This increased speed of measurement is known as the Fellgett Advantage. Via a well-known mathematical technique called Fourier transformation, the interferogram is transformed or “decoded” into a spectrum of frequency versus intensity.

Analysis Process

The origin of the NIR energy is a halogen light source. The energy beam enters the interferometer where the “spectral encoding” takes place. The resulting interferogram signal exits the interferometer and is transmitted through or reflected off the surface of the sample. This is where specific frequencies of energy, which are uniquely characteristic of the sample, are absorbed. For final measurement, the beam passes to the detector. The measured signal is digitized and sent to the computer where the Fourier transformation takes place. The final near-infrared spectrum is then presented to the user for interpretation and further manipulation. Figure 1 shows the typical layout of an FT-NIR spectrometer.

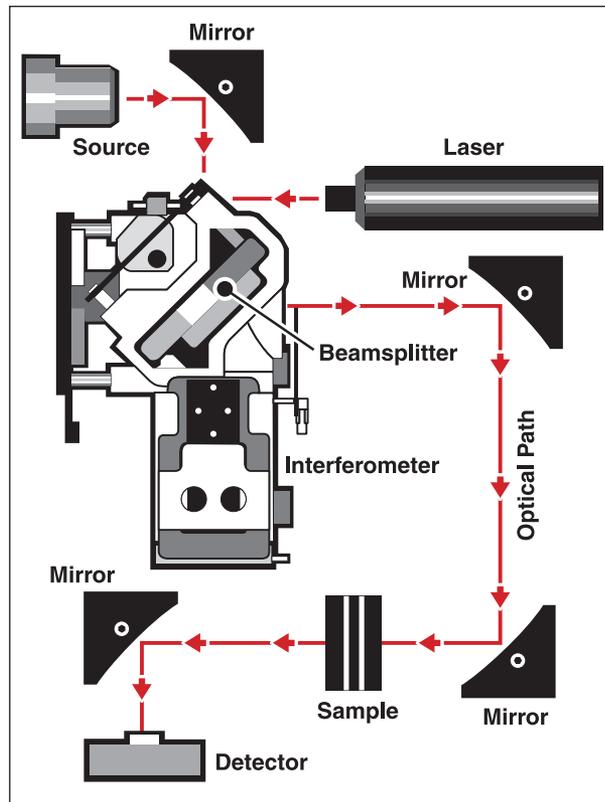


Figure 1: Illustration of spectrometer layout

An internal reference laser is used to control the interferometer and provides internal wavelength calibration. When the laser, a monochromatic light source, is directed into the interferometer, a modulated signal is created that is used to track the speed and position of the moving mirror. All of the spectral frequencies are referenced to this very precise HeNe standard laser frequency.

Advantages of FT-NIR

FT-NIR spectroscopy provides several clear advantages over classical dispersive techniques. These advantages are most important when analyzing NIR spectra of chemicals, polymers, and pharmaceuticals.

FT-NIR spectrometers are mechanically simpler than traditional dispersive instruments because the moving mirror in the interferometer is the only continuously moving part in the instrument. Thus, there is very little possibility of mechanical breakdown. In most dispersive instruments, gratings and filters must move in order to generate a spectrum. The benefit of mechanical simplicity is a more reliable, robust scanning mechanism that translates into a more reliable analyzer.

Dispersive NIR instruments rely on a prism or grating to separate (resolve) the near-infrared frequencies. The best grating can at best separate frequencies 50 cm^{-1} apart. However, most pharmaceutical, chemical, and polymer samples have spectral information that resolves at 8 cm^{-1} . Important spectral information for these types of samples cannot be measured on dispersive instruments so they employ a slit mechanism to achieve higher resolution. Because the slit limits the amount of beam that is measured, a substantial energy loss is incurred, making it difficult or impractical to measure samples at higher resolutions. Because the stroke length of the moving mirror determines resolution on an FT-NIR system, there is no degradation of optical throughput as is caused by the slits in dispersive instruments. With no degradation in performance, high resolution spectra can be quickly and easily measured by an FT-NIR system. This advantage is clearly shown in the spectrum of the NIST SRM 1920a standard reference material (Figure 2). With more spectral information, less

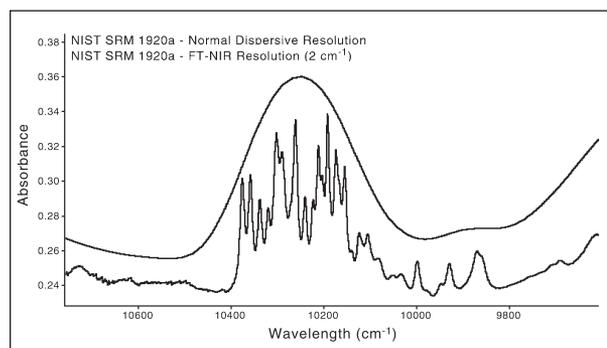


Figure 2: Comparison of NIST SRM 1920a spectra generated on an FT-NIR and dispersive instrument

reliance is needed on sophisticated chemometric algorithms, which translates into fewer standards being required to develop methods.

The use of an internal reference laser by FT-NIR instruments is referred to as Connes Advantage. The benefit of internal calibration is accuracy and precision of better than 0.1 cm^{-1} . Shifts in peak positions from scan to scan create artifacts in the data (Figure 3). Dispersive instruments utilize a mechanically complex prism or grating which cause errors in peak positions and inaccuracies from scan to scan. With the inherent inaccuracy problems of dispersive instruments, reference materials must be employed for calibration. The external calibration has to be repetitively performed, adding difficulty and operator error to measurements.

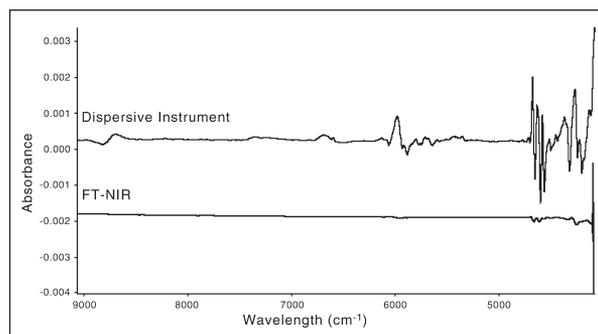


Figure 3: Subtraction of repeated scans of a polystyrene standard confirms that the presence of spectral artifacts is greater in dispersive instruments

With negligible FT-NIR instrumental artifacts due to wavelength inaccuracies, fewer standards are required, providing better results than with dispersive instrumentation. In addition, without wavelength error, large commercial digital libraries can be employed to quickly identify materials.

Conclusion

These advantages make FT-NIR measurements extremely accurate and reproducible. Thus, it is a very reliable technique for the positive identification and quantitation of any sample. This makes FT-NIR an invaluable tool for quality control and quality assurance applications.

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