

Infrared Spectroscopy



A Combined Mid-IR/ Near-IR Spectrometer

Introduction

When analysts are faced with the decision of selecting either mid-IR (MIR) or near-IR (NIR) spectroscopy for materials characterization, the decision is usually based on the analytical, sampling, and/or operational needs.

For example, when the greatest analytical specificity is required or certain inorganic materials need to be measured, the choice favors MIR; whereas the need to keep an unstable or dangerous material inside a sealed glass container during measurement might suggest NIR as the preferred choice. In practice, MIR is available in most analytical laboratories, but the use of NIR is growing faster, especially in the quality control environment. For most materials, the analytical requirement is satisfied by both mid-IR and near-IR.

Those laboratories handling a wide range of samples often choose mid-IR 'because it is there', often passing over the benefits of near-IR because in today's budget-conscious times it may be difficult to justify both the mid-IR and near-IR instruments. Ideally, in order to select the most fit-for-purpose method for the widest range of materials, it is highly desirable to have both techniques at one's disposal.

This situation has not been helped by the very different development histories of mid-IR and near-IR. With routine near-IR entering the marketplace much later than IR, the instrument vendors usually focused on one 'solution' or another; consequently, users were sometimes provided less than impartial advice on the most appropriate choice of spectral region. This also resulted in an inevitable split in the user community into MIR and NIR groups. Even today, some laboratory functions are segregated into mid-IR and near-IR sections with astonishingly little communication between them. In addition, the highly useful long wavelength near-IR region (ca 1.5-2.5 microns) is now readily accessible with modern, high performance FTIR spectrometers with performance levels that satisfy many application needs. This presents a difficult predicament for the new user: Faced with a choice of mid-IR or near-IR purchase, a high performance, wide range FT-IR might be a safer choice than a near-IR system. But would it be the optimum choice?

This sharp division of mid-IR and near-IR use in the laboratory is largely an artificial one created by vendors and users rather than a genuine need to best fulfil the application requirements. Thus, it would surely be better if it were not necessary to choose between the use of a mid-IR or near-IR spectrometer.

Combining recent MIR and NIR developments

The concept of a multi-range FT-IR spectrometer is not new. In the past, instruments have provided for multiple sources, beamsplitters and detectors, enabling access to the various spectral regions on a single bench. However, these instruments did not provide a high level NIR performance, and were largely adopted by more advanced users in R&D laboratories requiring sampling flexibility and customizable experimental setups. However, switching between ranges required a level of care in system reconfiguration and equilibration which was not consistent with routine use. In short, the instruments were too temperamental.



Figure 1: The PerkinElmer Frontier combined mid/near-IR spectrometer and sampling accessories.

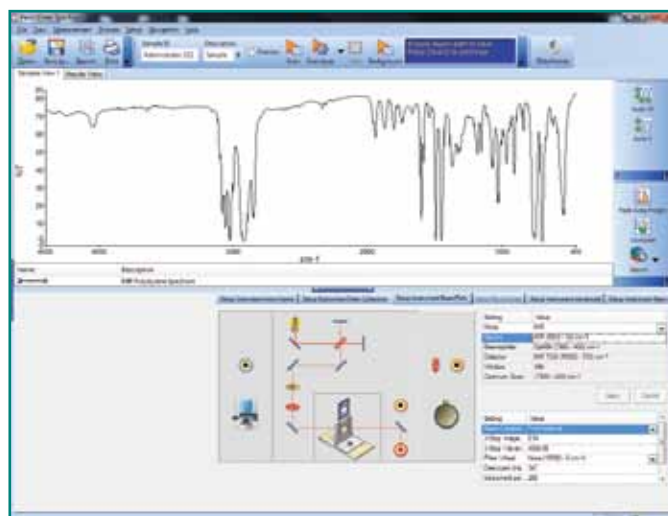


Figure 2: Automatic component selection and system optimization under software control.

In the meantime, there have been a number of significant developments in instrument, sampling, and software aimed specifically at the more routine user. Improvements in instrument stability and standardization include high sensitivity short wavelength transmission sampling and QA/QC software. The problem was that these developments were generally associated with dedicated mid- or near-IR instruments and not available to the earlier multi-range instruments.

PerkinElmer® has developed a combined mid-IR/near-IR spectrometer which brings the latest developments in mid-IR and near-IR together (see Figure 1). This system incorporates sophisticated automated range switchover and fully exploits the best in both mid-IR and near-IR QA/QC sampling and imaging developments. The result is a highly versatile, yet fully optimized FT-IR and NIR system on a single bench with a full suite of sampling and software, including 21 CFR11 compliance.

The Frontier MIR/NIR system provides high specification IR and NIR measurement, optimized sampling, imaging, and routine QA/QC software in a single instrument. The major design features include:

- Proven high performance interferometer (based on >8000 units manufactured) incorporating the latest design enhancements. No sampling or performance specification has been compromised in developing the combined instrument.
- Optimized FT-NIR configuration – again with no compromise in design compared with a dedicated FT-IR.
- Automated range switching and system optimization requiring no user adjustment (see Figure 2).
- Full suite of smart IR and NIR sampling options representing the best of IR and NIR sampling. For example, universal ATR with diamond crystal for IR, combined transmission/reflection short wavelength NIR sampler for automated tablet and power analysis, and combined IR/NIR imaging.

Multiple techniques can be sited permanently, with motorized beam switching for changeover. For example, NIR tablet analysis by transmission for content uniformity can be combined with NIR imaging for coarse (>50 microns) spatial distribution studies in tablets, plus mid-IR ATR imaging for fine (<5 microns) particle studies and mid-IR ATR macro sampling for packaging characterization. These four techniques can reside side by side in a single instrument with rapid switchover by using automatic system optimization routines (Figure 3).



Figure 3: Frontier MIR/NIR with a Spotlight 400 mid-IR/near-IR imaging system.

Benefits of a combined system

For a busy laboratory that tests a wide range of materials, the Frontier MIR/NIR offers the freedom to select the most appropriate (i.e., fit-for-purpose) combination of range and sampling for the application. In methods development laboratories, use of common software and analysis platforms helps in the comparison of the relative performance of IR and NIR methods. Once a technique is chosen for, say, a manufacturing plant, a single frequency range version of the Frontier may be used with dedicated sampling resulting in excellent reproducibility between the methods development and production instruments. This is facilitated by advanced standardization of wavelength and lineshapes of both instruments using high resolution gas phase spectra. The system offers a wider range of applications than single range systems.

In laboratories handling many samples per day, the ability to rapidly switch range and be always 'ready for the next sample' will provide significant productivity improvements over time. Other benefits of using a common software platform for both IR and NIR ranges include faster learning and lower training needs. This clearly results in a lower overall cost of ownership compared with separate instruments from different vendors – not only in terms of initial investment but also in afterservice support and maintenance.

So much for the general benefits of choosing a combined instrument, but what kind of laboratories would benefit from this? Typical examples include the following:

Analytical and QC laboratories measuring a wide range of solids and liquids

Many powdered solids are granular or even waxy with particle sizes from a few microns to millimeters. NIR diffuse reflection is often the ideal technique to use. However, some solvents (e.g., homologous series) are homogeneous and require a high level of discriminatory power. While it is possible to use special reflectors to obtain a spectrum of a clear liquid with a diffuse reflectance accessory, this arrangement is far from optimal. The NIR range used and the ordinate errors introduced by such a sampling configuration will limit the reliability of the method. Mid-IR UATR, or even direct transmission measurement in mid- or near-IR, is likely to offer superior analytical results. A combined system with both NIR diffuse reflectance and UATR installed simultaneously, with selectable methods that automatically switch beampaths, will provide a superior solution.

Another common situation is in pharmaceuticals raw materials testing where NIR libraries are developed for most of the raw materials coming into the plant. However, there are common ingredients, such as anhydrous silica, which do not provide a useful NIR spectrum. On the other hand, the mid-IR spectrum of this ingredient is highly distinctive and reproducible. In these circumstances, the combination of NIR diffuse reflectance with mid-IR UATR is more appropriate than NIR alone.

Samples with specific handling requirements

Sodium acetate is produced in industrial quantities and has a very wide range of uses in industries such as textiles, foods, and cosmetics. Here, IR is widely used for materials testing. The material is a white, hygroscopic powder and attempting to measure the anhydrous form by mid-IR UATR is problematic as the spectrum changes in time with the exposure to moist air (see Figure 4).

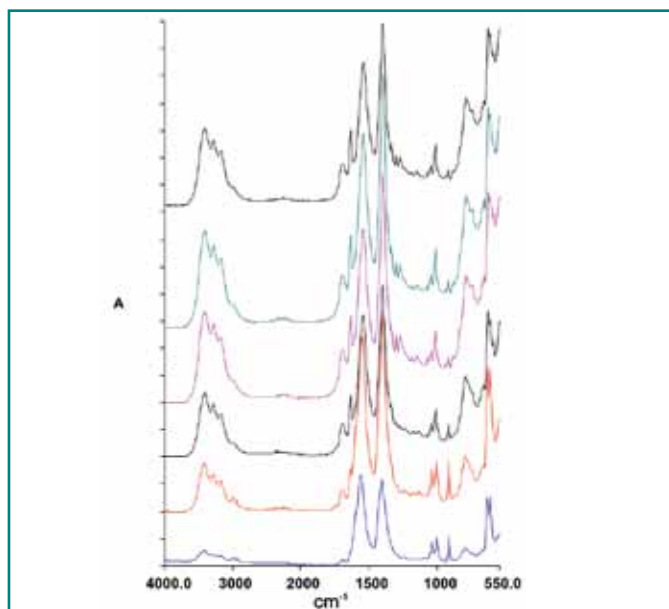


Figure 4: IR ATR spectra of sodium acetate after exposure to air.

For this and similarly behaving materials, it makes more sense to measure the powder in a sealed vial or jar, non-invasively using NIR diffuse reflection. Again, where commonly a standard IR is used for bulk chemicals and in the textiles industries for most materials, a combination of both techniques is more useful. In addition to the considerations for unstable materials, this argument also applies to materials requiring special handling from a health and safety perspective.

Combining MIR and NIR imaging techniques

NIR chemical imaging is becoming widely recognized in the pharmaceutical industry as an invaluable tool for studying the distribution of certain ingredients in solids dosage forms. However, the measurement is a diffuse reflection technique which limits the spatial resolution and level of detail that can be seen. If the domain sizes for the ingredients are greater than 50 microns, for example, current NIR imaging systems generally work well, and offer sampling and speed advantages over Raman or standard MIR imaging. It is considered the preferred technique for imaging whole tablet areas in the shortest time. However, we know that many ingredients are introduced into the process with particle sizes less than 10 microns. If particle aggregation does not occur during the processing steps, it is unlikely NIR will observe these particle distributions in the final dosage form. This is where mid-IR ATR imaging comes in. The nature of the ATR technique is such that incident radiation only penetrates the sample to a depth of ca. 1-2 microns (10 to 100 times less than with NIR diffuse reflectance) and the incident beam travels through a high refractive index medium, in contact with the sample before entering the sample. These two factors contribute to a huge improvement in spatial resolution and image clarity as compared with NIR. Single particles of ca. 5 microns have been readily observed using ATR imaging. For troubleshooting products, where ingredient domain sizes can range from less than 10 microns to many hundreds of microns, the combinations of mid-IR ATR and near-IR imaging are truly complementary (see Figure 5).

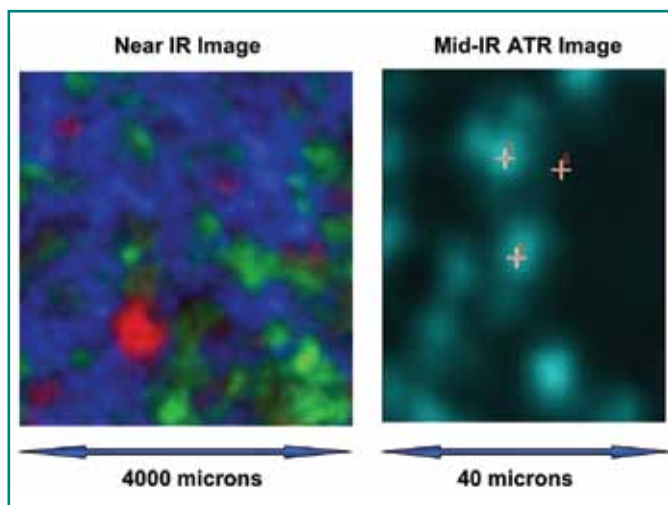


Figure 5: Near-IR and mid-IR ATR images of pharmaceutical tablets. The ATR technique can readily reveal single particles of less than 10 microns. The NIR technique images larger areas and whole tablets more efficiently.

Conclusion

Since equipment utilization concerns influence instrument investment decisions to an ever greater degree (also due to the performance enhancements of FT-NIR), it now makes sense to combine high performance mid-IR and near-IR into a single instrument. The most successful applications tend to be those that use the most appropriate selection of spectral range, sampling, and analysis. Almost any laboratory handling a diversity of samples will find use of both techniques to be advantageous. Given the freedom to use either technique, the analyst no longer needs to choose between a mid-IR and near-IR spectrometer, but can direct his efforts into finding the most fit-for-purpose method for a particular sample. This will result in fewer failed methods and more productive instrument use.