APPLICATION NOTE

FT-NIR Spectroscopy

Solid Materials Checking Using the Near-IR Reflectance Accessory (NIRA)

Versatility of Sampling

The NIR Reflectance Accessory (NIRA) for the PerkinElmer Frontier™ System is designed for quick, easy sampling without compromising spectral quality. Sampling is simple as materials are measured directly in containers placed on the rugged sample platform. In the case of tablets, tablet blister packs, and transdermal patches, samples are placed directly on the sampling window where they are illuminated from below. Samples can also be measured in a range of containers, from conventional vials, plastic bags or bottles, to colored glass bottles. The original containers which the samples are transported to the instrument are more convenient, reproducible, and carry less risk of cross-contamination than using fiber-optic dipping probes.

To illustrate the versatility of NIRA sampling, a few examples and their resultant spectra are shown in this note.

PerkinElmer®
For the Better

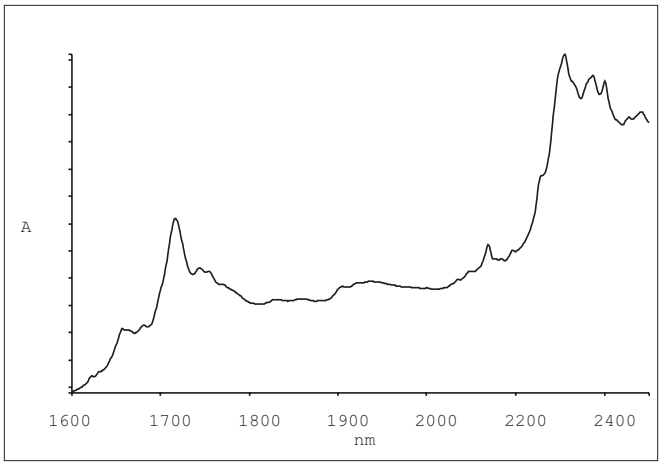

Figure 1. NIR spectrum of aspirin tablet in pack. It is unnecessary to remove a tablet from a blister pack before obtaining the spectrum using the NIRA. Simply place the sample you want, in its blister packing, directly on the sampling window.

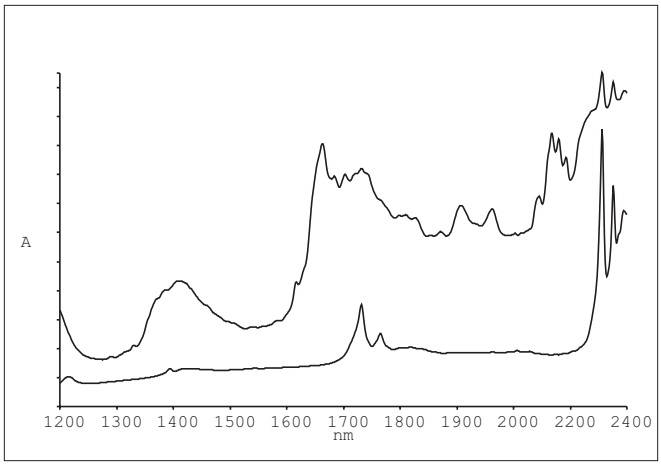

Figure 3. NIR spectra of a polyethylene bag (lower) and polybutlylene terephthalate (PBT) in a polyethylene bag (upper). This PBT sample was received in a polyethylene bag, and scanned intact.

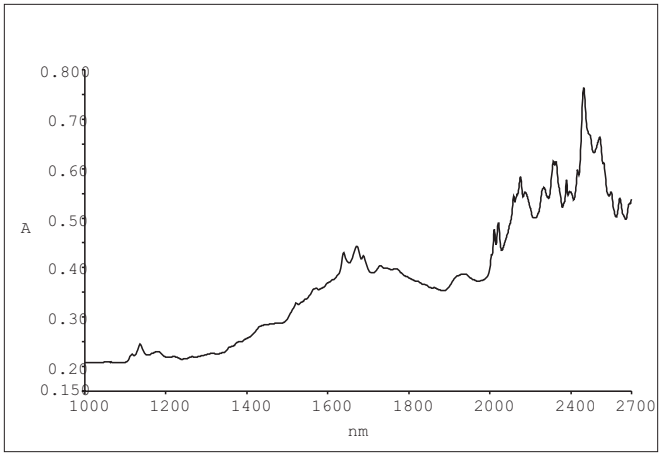

Figure 2. NIR spectrum of paracetamol tablet. The spectrum of a paracetamol tablet was obtained by simply placing the tablet on top of the sampling window.

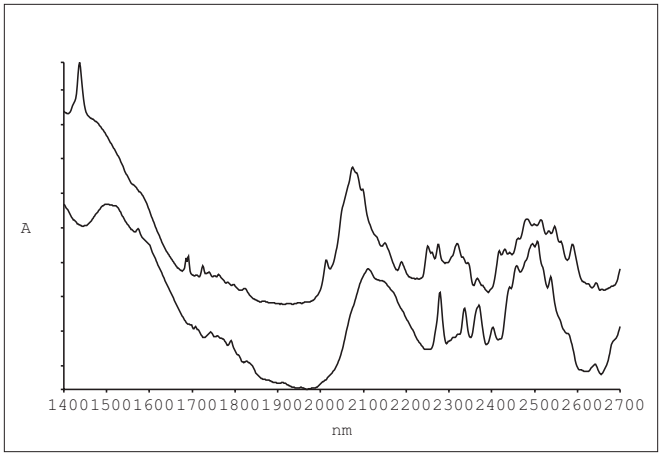

Figure 4. NIR spectra of sucrose (upper) and dextrose (lower) scanned in amber colored glass vials.

Advantages of the NIRA Accessory

By eliminating the need for any special sample preparation or accessory cleaning, the NIRA ensures more consistent measurement conditions. The large illumination area allows for the analysis of samples with varying (large) particle size and inhomogeneity is improved. A large sample area is measured, therefore decreasing the influence of inhomogeneities and providing a more representative sampling, for example polymer flakes and pellets.

For more granular and inhomogenous materials such as grains, an optical spinning sample cup is available to ensure the most representative sampling.

Reliability and Reproducibility

The NIRA accessory eliminates the major problems associated with using fiber-optic probes for materials checking. Hand-held fiber optic probes are inherently unstable. The NIRA, however, offers a robust sampling platform. The automated self referencing feature also eliminates the need to collect separate background spectra.

Test data for replicate spectra of calcium ascorbate from a commercially available fiber optic probe system and the NIRA are shown in Figures 5 and 6. The results from the NIRA show much less variability than those of the probe system.

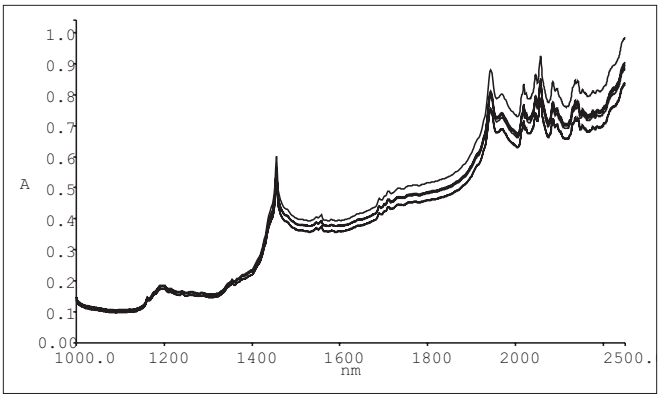

Figure 5. NIR spectra of calcium ascorbate in a glass bottle recorded using the NIRA.

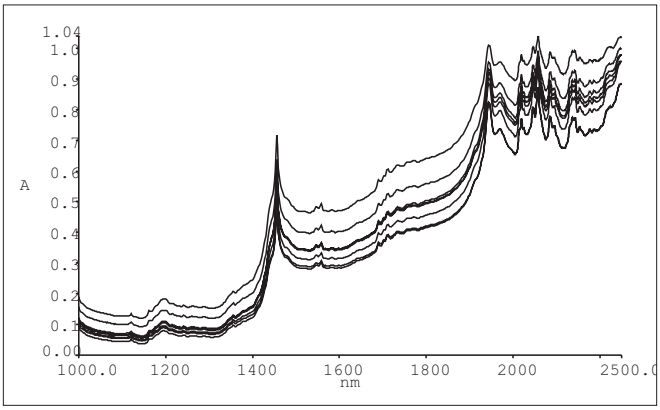

Figure 6. NIR spectra of calcium ascorbate recorded using a fiber optic solids probe.

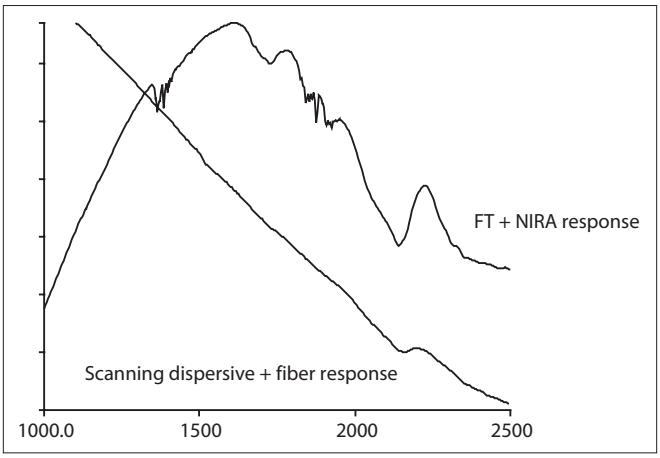

Figure 7. Typical response of the NIRA compared with a typical scanning dispersive instrument with a fiber probe.

Spectral Response

Traditionally, NIR sampling has been confined to fiber optic and dispersive systems with spectra measuring between ca. 10000 – 2200 nm (10000 – 4545 cm^{-1}). These systems typically provide the highest response in the short wavelength region.

The response from the NIRA is shown in Figure 7 along with that of a typical dispersive system. Sample spectra are shown in Figures 8, 9 and 10 with the high quality long wavelength information gained when using the NIRA.

One advantage to this response allows for sampling to occur in original colored glass bottles which typically show spectra with little useful information in the shorter range. By collecting data in the longer wavelength region where the glass is transparent, sample identity can often be verified without removal from the original container. The NIRA is optimized for operation in the longer wavelength range in order to maximize the information content available.

Resolution

The NIRA delivers new levels of information in NIR spectra due to the higher resolution available. In addition to improving spectral interpretation, this offers improved accuracy for qualitative analysis, and assists in transferring multivariate calibrations between instruments.

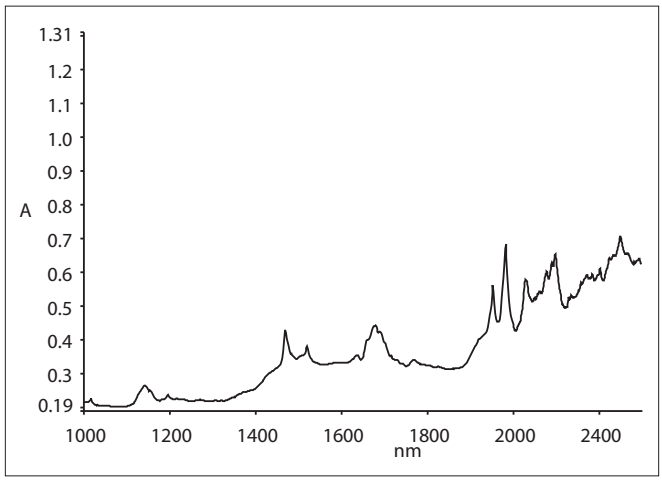

Figure 8. Tegretol® tablet.

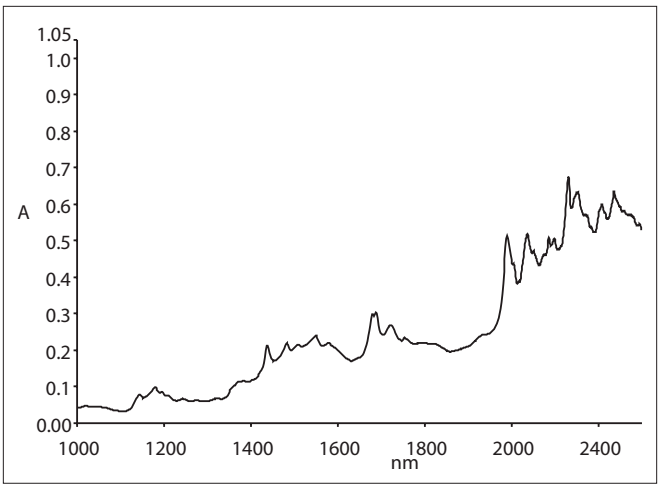

Figure 9. Procainamide tablet.

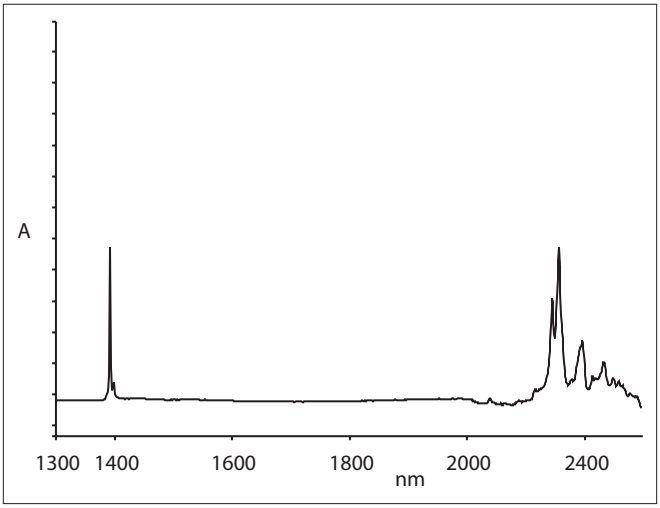

Figure 10. Talc.

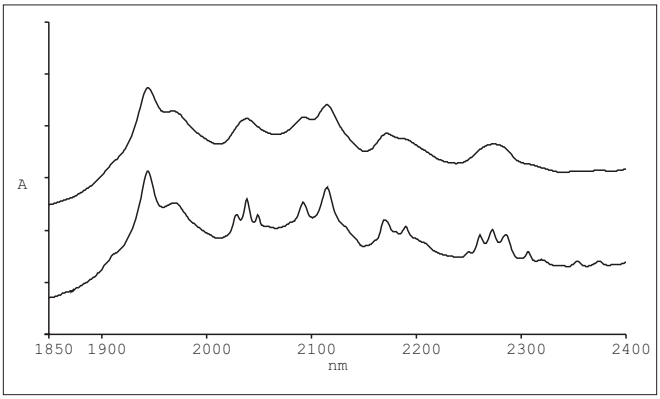

Figure 11. NIR spectra of calcium ascorbate at typical dispersive NIR instrument resolution 3 nm (upper) and FT resolution of 0.4 nm (lower).

Conclusion

The NIRA offers simple but effective sampling solutions for materials testing without compromising spectral quality or time. The combination of optimized range and resolution reveals information which can potentially improve qualitative and quantitative applications. This extra level of spectral detail should provide new opportunities for analyses in terms of both easier sampling and improved spectral discrimination and quantitation.

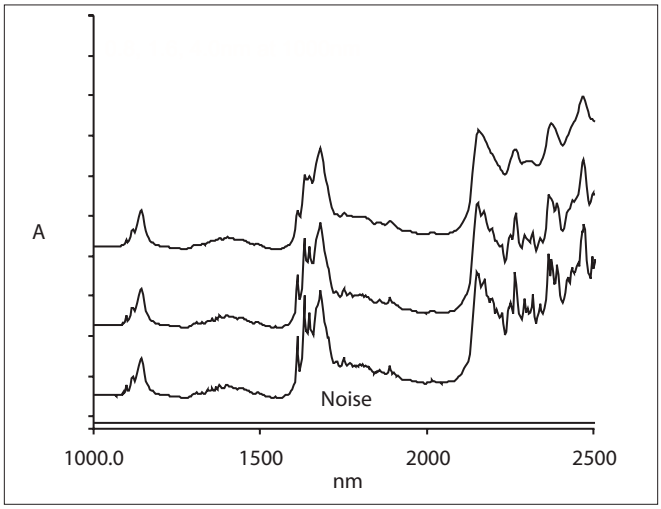

Figure 12. NIR spectra of clotrimazole at 4, 1.6, 0.8 nm resolution (upper to lower). The increased spectral information gained at higher resolution is real as the noise profile is shown on the same scale.

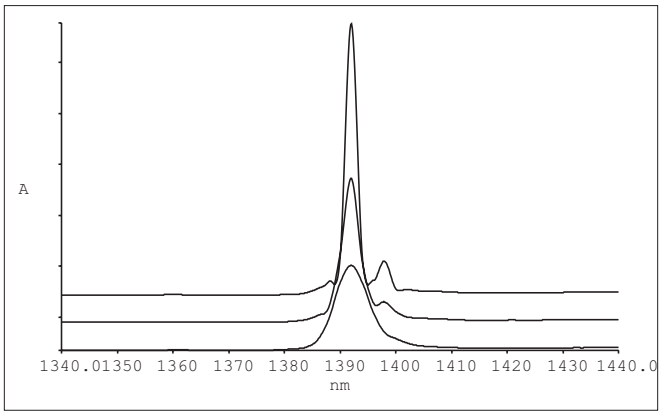

Figure 13. NIR spectra of talc at 0.4 nm, 1.6 nm and 3.2 nm resolution (upper to lower).

PerkinElmer, Inc.
940 Winter Street
Waltham, MA 02451 USA
P: (800) 762-4000 or
(+1) 203-925-4602
www.perkinelmer.com

PerkinElmer®

For a complete listing of our global offices, visit www.perkinelmer.com/ContactUs

005662B_01