



FT-NIR Spectroscopy

Key Features:

- High-performance NIR reflectance measurements
- Easy and convenient sampling for a wide range of sample forms
- Sample cup spinners to improve sampling of inhomogeneous materials
- Software interface allowing simple workflow development for qualitative and quantitative analysis

Near-Infrared Reflectance Sampling Accessories for the Frontier and Spectrum Two N FT-NIR Spectrometers

Introduction

Near-infrared (NIR) measurements of solids and liquids have never been easier and more reliable than they are now with the improved near-infrared reflectance accessories

for PerkinElmer's range of FT-NIR spectrometers, including the Spectrum Two N™.

NIR spectroscopy is used extensively in a wide range of industries, such as fine chemicals, pharmaceuticals, food, agriculture, and polymers. It has the advantage of being able to perform qualitative and quantitative measurements on a variety of forms of samples without the requirement for sample preparation. Fast and easy measurements can be performed on a variety of sample types, such as powders, pellets, continuous solids, gels and slurries, and liquids over a large range of viscosities, all measured using NIR reflectance. NIR can be used throughout the manufacturing process from raw material testing all the way through to final product testing. Plus, with PerkinElmer's FT-NIR solutions, you can leverage IQ/OQ protocols and Enhanced Security™ (ES) software control of the instrument and sampling accessory ensure the performance and validity of data within highly regulated environments, facilitating 21 CFR Part 11 compliance in the pharmaceutical industry.



Figure 1. The NIRA II on the Frontier FT-NIR.



Figure 2. The NIR Reflectance Module on the Spectrum Two N FT-NIR.

High-Performance NIR Reflectance Measurements

Diffuse reflectance measurements in the mid-infrared spectral region lead to extremely high absorbances and spectral distortions. The greatly reduced absorptions in the NIR spectral region prevent these problems and are perfectly matched to performing measurements directly on samples without any sample preparation or dilution.

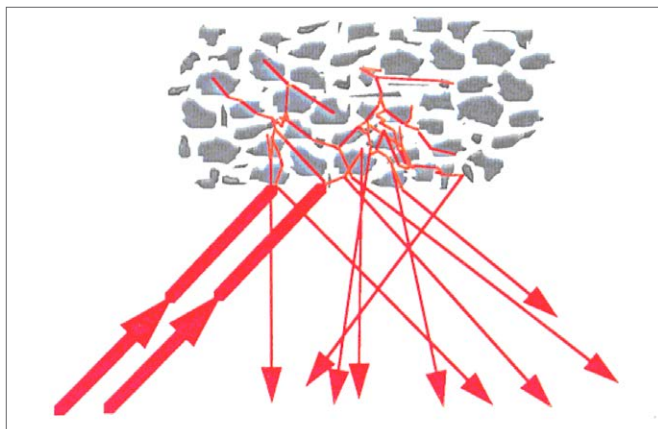


Figure 3. Schematic representing diffuse reflectance of scattering materials.

All of the components within the sampling accessories are optimized to give the highest performance possible. Gold-coated optics are used to maximize the energy throughput and a high sensitivity InGaAs detector gives excellent performance over the entire NIR spectral range. The accessory is sealed to protect it from environmental and contamination problems. Within the sealed environment is a high-stability internal check standard used for reference collection, preventing the contamination that can occur with external standard materials.

The accessory optics are designed to provide tight control of the optical geometry, generating excellent spectral uniformity across the beam and a high collection efficiency leading to excellent consistency, repeatability, and sensitivity of measurements. High-quality NIR spectra can be collected within a few seconds. The optical arrangement also greatly reduces stray light from sample containers, such as vials or Petri dishes, ensuring the measured spectrum contains only material-specific information. Figures 4 and 5 demonstrate the high performance of the NIR reflectance accessory.

The combination of all of these design optimizations results in a sampling module that facilitates easy transferability of methods from one instrument to another, a key requirement for method deployment on multiple instruments that can be at different sites in different parts of the world.

Ease and Convenience

Working with the plug and play NIR sampling accessories is simple. The accessory is automatically recognized when inserted in the instrument and the software will setup the scan parameters to the required settings (Figure 6).

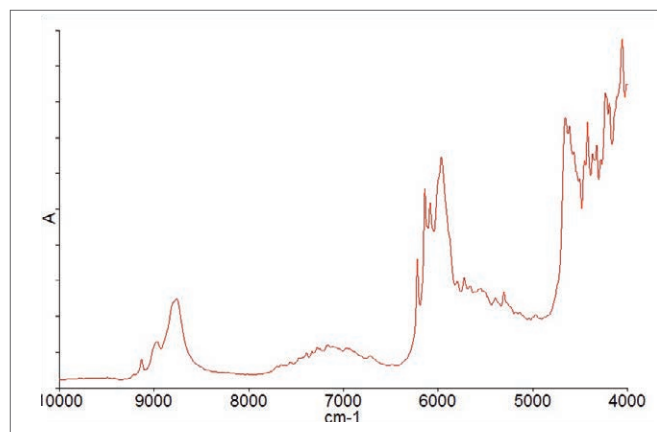


Figure 4. Spectrum of clotrimazole powder in a glass vial collected in five seconds.

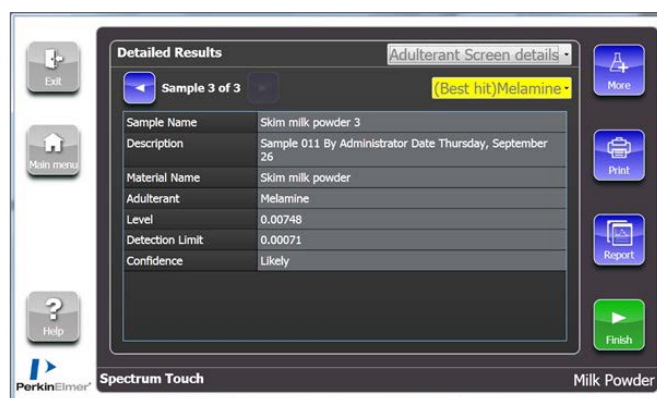


Figure 5. Adulterant Screen result showing the detection of low level (0.7%) of melamine in milk powder. The estimated detection limit is 0.07%.

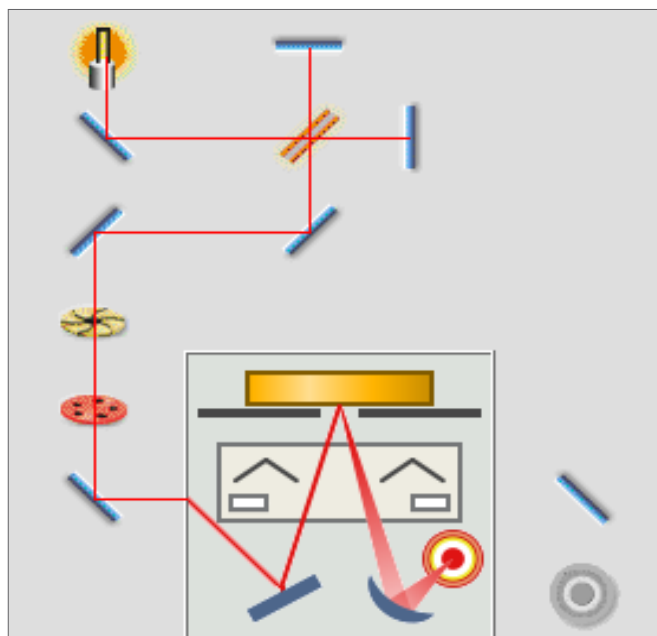


Figure 6. Instrument beampath diagram showing the NIR Reflectance Module on the Spectrum Two N.

If you are going to use the sample spinner, you simply need to place the spinner assembly on top of the accessory and the magnets will position it onto the kinematic location bearings and hold it in place. There are no cables to plug in, since the spinner gets electrical power via a wireless WIFI transmitter.



Figure 7. Removal of the sample cup spinner.

NIR sampling can be performed using low-cost disposable media such as glass vials and Petri dishes without any further sample preparation. Samples can be measured through blister packs and polythene bags too. Many types of samples, for example tablets or plastics, can be measured directly on top of the accessory sample window. For cleaning convenience, the accessory has a flat, easy-clean top surface. In the case of a major spillage, the accessory edges overlap the main instrument preventing internal contamination, and the spinner or other sub-accessories can be simply lifted off for cleaning (Figure 7).

Sampling Versatility

A wide range of physical forms of materials can be measured on the accessory ranging from solids (powders, pellets, tablets, plastics) through to gels and liquids. A range of sampling adaptors are available to work on the accessory to optimize the sampling for the different sample types. The sample spinner is designed to be used in cases where a large amount of the sample is available and the sample suffers from inhomogeneity. As the sample spectrum is being measured, the spinner will be rotating such that different parts of the sample in a Petri dish are being scanned and the inhomogeneities averaged out. This is particularly important for natural materials, such as herbs and spices, where significant variation is observed within the sample. To allow for different sample sizes, there are two versions of the sample spinner for 60 mm and 100 mm Petri dishes.

Powder samples can be conveniently measured in flat-bottomed glass vials of a wide range of diameters. To ensure repeatable positioning and sample measurement, a kinematically mounted variable aperture vial holder can be used. The variable iris of the vial holder will center the vial in the middle of the accessory window and will also close down around the vial to eliminate any possibility of stray light.



Figure 8. 60 mm and 100 mm sample spinners.



Figure 9. Vial-positioning device for NIR Reflectance Module.



Figure 10. Sampling containers for NIR reflectance measurements.

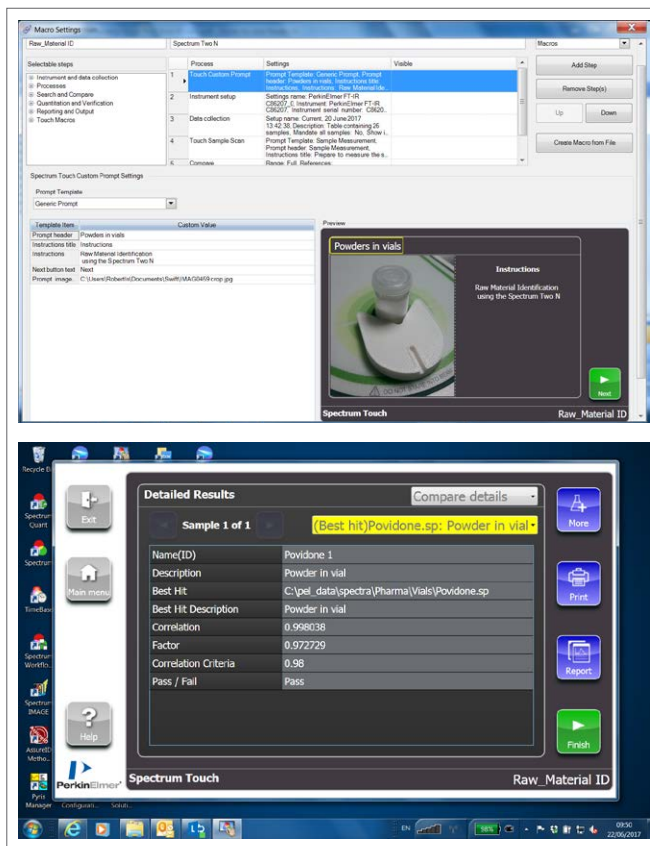
Gels and liquid samples can be measured on the accessory using a technique called "transflectance". In a normal transparent liquid, all of the incident radiation to the sample will simply pass through the sample and very little will be reflected back to the detector. By placing an inert reflective surface (the transflectance adaptor) in the liquid, the incident radiation will be reflected back through the sample to the built-in accessory detector. The effective pathlength is the distance through the sample and then back through the sample. The height of the transflectance adaptor is fixed giving a constant pathlength for all measurements. This allows for fast and simple measurements of liquids on the accessory.



Figure 11. Transreflectance adaptor for the NIR reflectance accessories.

Simple Workflow Development

Direct control of the accessory can be achieved in the Spectrum 10 software for setup and data collection. These setup and data collection steps can be incorporated into a "workflow" within the Spectrum Touch™ (or Spectrum Touch ES version) environment that can incorporate data analysis functions, such as Spectrum Quant, Adulterant Screen, Search and MultiSearch, Verify and Compare, to provide a total analysis of the sample by following simple on-screen instructions.



Figures 12a and 12b. Example screens from the Spectrum Touch software.

Ordering Information

	Description	Part Number
Frontier	Near-Infrared Reflectance Accessory (NIRA II) NIR diffuse reflectance accessory for solids, liquids and powders using PerkinElmer's Frontier FT-NIR spectrometer. It can be used for a wide variety of sample types in different container sizes. The accessory comprises innovative collection optics (patent pending) designed for robust, accurate operation, offering increased performance with dramatically improved inter-unit variability.	L1280450
	Near-Infrared Reflectance Module NIR diffuse reflection accessory for solids, liquids, and powders using PerkinElmer's Spectrum Two N FT-NIR spectrometer. It can be used for a wide variety of sample types in different container sizes. The accessory comprises innovative collection optics (patent pending) designed for robust, accurate operation, offering increased performance with dramatically improved inter-unit variability.	L1390010
Wireless Spinner Options	60 mm Sample Spinner The 60 mm spinner provides representative sampling of non-homogenous samples. The accessory is easy to clean with detachable covers, a flat base, and a wireless contact-free power connection, making it easy to use and clean (patent pending). The 60 mm spinner is suitable for holding sample containers up to 55 mm OD.	L1280410
	100 mm Sample Spinner The 100 mm spinner provides representative sampling of non-homogenous samples. The accessory is easy to clean with detachable covers, a flat base, and a wireless contact-free power connection (patent pending). The 100 mm spinner is suitable for holding sample containers up to 95 mm OD.	L1280417

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