

FT-IR and FT-NIR Spectroscopy

**Key Features:**

- Accurate temperature control of samples
- Range of inserts to accommodate disposable vials, cuvettes, or transmission cells
- Integrated unit with direct temperature control from Spectrum software
- Operates within the Mid-IR or Near-IR spectral ranges

Heatable Transmission Module for FT-IR and FT-NIR

Introduction

Infrared spectral measurements are generally performed in the instrument sample compartment at ambient temperature. For the vast majority of samples, this is a perfectly acceptable approach even if the ambient temperature fluctuates. However, there are some samples where a small change in the sample temperature can cause undesired small spectral changes, such as wavelength shifts. Hence, accurate temperature control of the sample is required. In addition, some studies are performed to observe the effect of elevated temperatures on the sample. And in some cases, a higher sample temperature is required to facilitate easier sampling of the material, such as melting of materials that are solid at ambient but melt at slightly higher temperatures, to allow liquid transmission measurements. Temperature effects can be quite marked for many samples, particularly where strong H-bonding is present. This can severely compromise quantitative work especially where elevated temperatures are required to make the samples liquid.

The Heatable Transmission Module (HTM) for the Spectrum Two™ platform (including both the Spectrum Two FT-IR and Spectrum Two N™ FT-NIR) allows for easy elevated temperature measurements on samples in the Mid-IR or Near-IR regions of the spectrum, the module accepting fixed pathlength sealed cells, cuvettes, or disposable vials. The accessory is designed for use with the instrument detector and compatible with both Mid-IR and Near-IR models giving detector choice to suit the application for a range of sample types for high to low transmitting samples.



Figure 1. Disposable vial sampling for NIR measurements on the HTM.

Widest Range of Window Material and Pathlength Options

The cell requirements for Mid-IR and NIR measurements are significantly different, not only in the optimum optical materials, but also the sample pathlength. Long sample pathlengths are required in the NIR region since the absorptions are generally 10-20 times weaker than in the Mid-IR region. Short sample pathlengths are required in the Mid-IR to avoid saturation of the spectral peaks. The HTM allows for interchangeable cell cartridges to enable the swap-in of different cells optimized to the specific application being run.

For NIR measurements, the HTM can be used with disposable glass vials of 5, 8, and 12 mm outer diameter and with a range of rectangular cuvettes made from NIR Quartz SUPRASIL® 300, Quartz SUPRASIL®, and Special Optical Glass, with pathlengths of 1, 2, 5, and 10 mm.

For Mid-IR measurements, the HTM can be used with a demountable-style cell with window materials of CaF₂, BaF₂, KBr, or ZnSe at pathlengths of 50, 100, 200, 500, and 1000 microns. The window selection will depend on the materials being analyzed and the lower limit of the spectral range required. It should be noted that these optical materials will also transmit light up into the NIR region of the spectrum.

Ease and Convenience

NIR sampling for the HTM can be achieved using low-cost disposable vials of different pathlengths. Since sample pathlengths in NIR measurements can be in the range of mm, it makes the

sampling procedure significantly easier and can be achieved by pipetting the sample into the vial. It also makes sampling of viscous materials, such as liquid polymers or edible oils, significantly easier. Using disposable vials is a simpler option than trying to clean out short pathlength cuvettes between samples. However, fixed pathlength cuvettes should be utilized when the most accurate quantitative measurements are required. Each vial/cuvette/cell has its own mounting block that can be swapped for a different type in seconds.



Figure 2. Disposable sample vials for NIR transmission measurements.



Figure 3. Range of cuvette and disposable vial inserts.

When temperature control is required for the experiment, the sample vial should be inserted and left for a suitable time for the sample temperature to equilibrate. To reduce the equilibration time for multiple samples, when using 5 or 8 mm vials, there are two additional slots in the sample holder allowing the sample and two extra samples to be equilibrating. If multiple samples require equilibration, an external electrical heating block is available.

Automatic accessory recognition in the Spectrum 10 software will detect when the HTM is inserted and will setup the scan parameters for that sampling module. The temperature of the module is also controlled within the software including scan delays to allow the sample temperature time to equilibrate.

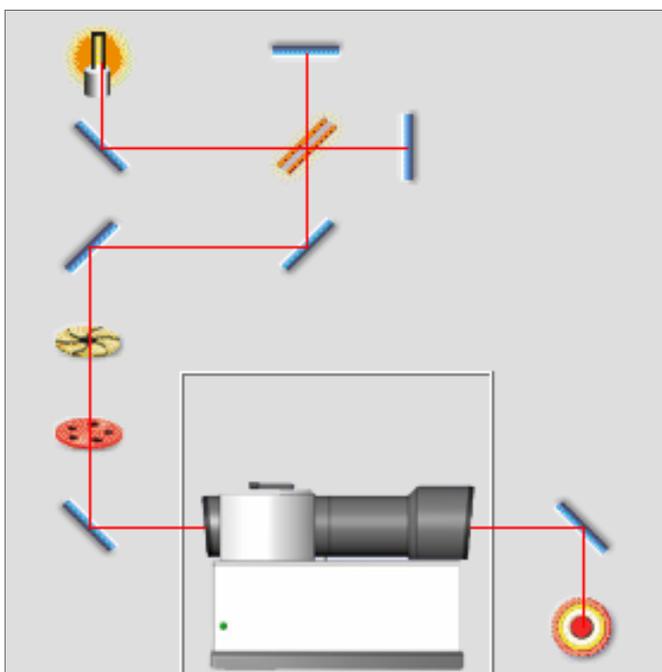


Figure 4. Spectrum 10 instrument beampath with HTM.

High Performance from the HTM

The HTM is designed to give optimum performance for a wide range of liquid samples. Long pathlengths can be used when trying to measure low concentration components or small differences in sample sets, while shorter pathlengths can be used for measuring components at higher concentrations. Whichever cell or vial type is used, the optical performance is maximized. The different vial holders have built-in beam apertures to eliminate the possibility of stray light passing around the outside of the vials. The apertures also ensure consistent beam path for the background and sample measurement, avoiding wavelength shifts that would occur with mismatched apertures. The cell holders are offset at a small angle to prevent back reflections and to minimize channeling effects within the cells.

The HTM can be operated up to a set temperature of 80 °C in 0.1 °C increments with an accuracy of better than 1 °C, giving repeatable temperatures for repeatable measurements.

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 Enhanced Security™ Spectrum Touch ES version) environment that can incorporate data analysis functions, such as Spectrum QUANT, Adulterant Screen, Search and MultiSearch, as well as Verify and Compare, to provide a total analysis of the sample by following simple on-screen instructions.



Figure 5. Example screens from the Spectrum Touch software.

Elevated Temperature Operation

The HTM is particularly suited to the measurement of edible oils, especially palm oil, which can be solid at low ambient temperatures. The samples need to be heated to a temperature above ambient to convert them to the liquid phase and a much more transparent state. Quant methods can be developed for measuring some of the critical quality parameters of these edible oils, such as solid fat content (SFC).

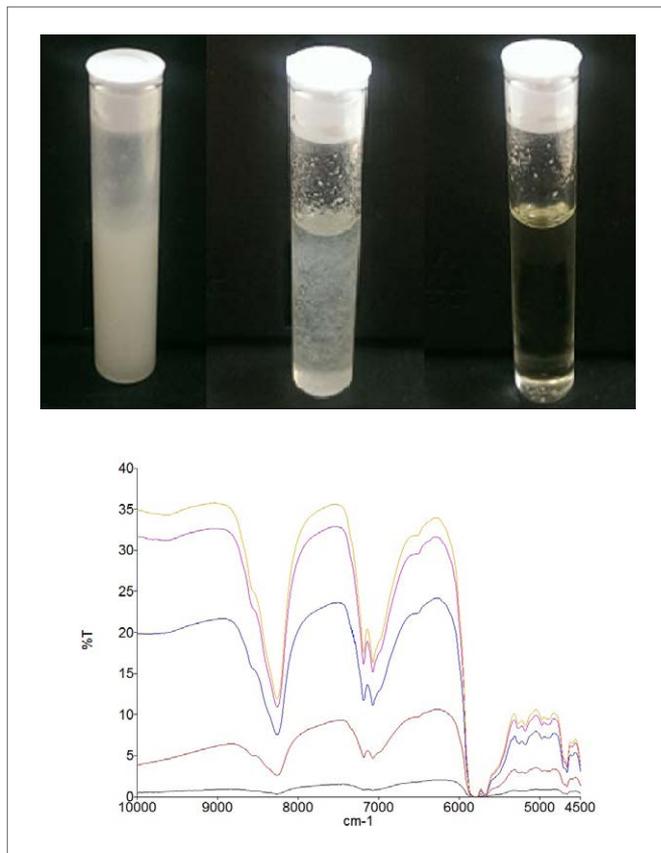


Figure 6. a. (left to right) Palm oil sample at room temperature, 35 and 50 °C; b. Palm oil spectra from room temperature (lowest intensity) up to 50 °C (highest intensity).

Accurate Temperature Operation

In the polymer industry, the HTM can be applied to the quantitative NIR measurement of the hydroxyl number of polymers, e.g. polyols. The quantitative accuracy requires excellent control of the stability and accuracy of the sample temperature, since spectral peaks will shift if there is temperature variation.

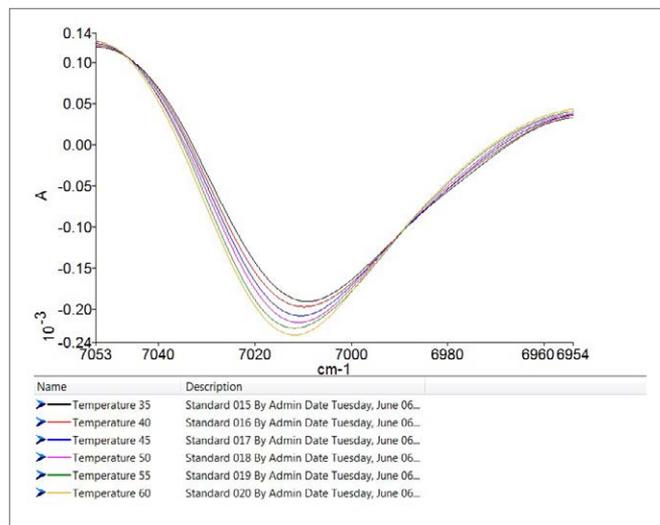


Figure 7. 2nd derivative spectra of a polyol at different temperatures showing spectral shifts.

Ordering Information

Description	Part Number
Spectrum Two N FT-NIR Heatable Transmission Module (HTM) Uses Spectrum Two N built-in detector. Includes 8 mm vial holder insert. <i>Note: Choose additional vial or cuvette holder options from below if required</i>	L1390029
Additional HTM Accessories	
5 mm Vial Holder Insert	L1390136
8 mm Vial Holder Insert <i>Note: one is included with the module</i>	L1390137
12 mm Vial Holder Insert	L1390138
1 mm Cuvette Holder Insert	L1390147
5 mm Cuvette Holder Insert	L1390148
10 mm Cuvette Holder Insert	L1390149
5 mm Vials with Caps	L1390187
8 mm Vials with Caps	L1390188
12 mm Vials with Caps	L1390189