HEATABLE TRANSMISSION MODULE (HTM)

FOR USE WITH SPECTRUM TWO FT-IR AND SPECTRUM TWO N FT-NIR SPECTROMETERS

User's Guide

PerkinElmer®

Release History

Part Number	Release	Publication Date
L1050302	A	September 2017

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Contents

Warnings and Safety Information

General Safety

The Heatable Transmission Module (HTM) is intended for use with PerkinElmer Spectrum Two FT-IR and Spectrum Two N FT-NIR spectrometers.

These instruments have been designed and tested in accordance with PerkinElmer specifications, and in accordance with the safety requirements of the International Electrotechnical Commission (IEC). The Accessory conforms to IEC 61010-1 ("Safety Requirements for electrical equipment for measurement, control and laboratory use") as it applies to IEC Class 1 (earthed) appliances and therefore meets the requirements of EC low voltage directive 2006/95/EC.

Radiation Emitted by the Instrument NIR Source

The NIR source in the PerkinElmer Spectrum Two N FT-NIR spectrometer uses a tungsten halogen bulb that emits ultraviolet, visible and infrared radiation. The majority of this energy is in the infrared region. Do not stare into the beam produced by this bulb.

Measurements of the infrared radiation emitted from the spectrometer's sample window show that exposure limits recommended by the American Conference of Governmental Industrial Hygienists (ACGIH) and the International Commission on Non-Ionizing Radiation Protection (ICNIRP) will not be exceeded during normal operation.

WARNING

Do not look directly into the beam emitted from the sample window. Prolonged exposure (>10 seconds) may cause permanent eye damage.

AVERTISSEMENT

Ne regardez pas directement le faisceau émis dans la fenêtre d'échantillonnage. Une exposition prolongée (> 10 secondes) peut causer des lésions oculaires permanentes.

For further safety and warning information, refer to the appropriate User's Guide for your instrument. These are distributed, as .pdf files, on the **Spectrum Two Manuals CD** (L1050242).

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 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 temperature but melt at slightly higher temperatures, to allow for 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 The Heatable Transmission Module (HTM)

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

The infrared beam from the instrument enters the HTM through the port on the left-hand side of the accessory and is directed through the HTM to the sample window in the Spectrum Two N.

Sample Options

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).

A range of cuvettes and disposable vial inserts (see Figure 2) are available from PerkinElmer. See *Additional Parts* on page 16 for more information.

Figure 2 Cuvette and disposable vial inserts available from PerkinElmer

Packing List

The following items are provided with the HTM:

Part Number	Description	Quantity
L1390137	8mm Vial Holder Insert	1
L1390188	8mm Vials and Caps	1
L9002298	Power Supply	1

Power Supply

The power supply connects directly to the HTM accessory and is required to operate the HTM. This power supply is standard for all Spectrum Two N accessories.

Installing the Module

The HTM can be simply installed into the sample compartment of the spectrometer, after first removing any other sampling accessory fitted.

To remove the current accessory and install the HTM:

1. Pull the baseplate of the current sample accessory towards you, and slide the accessory out of the sample area (see Figure 3).

   Store the accessory in a safe place for future use.

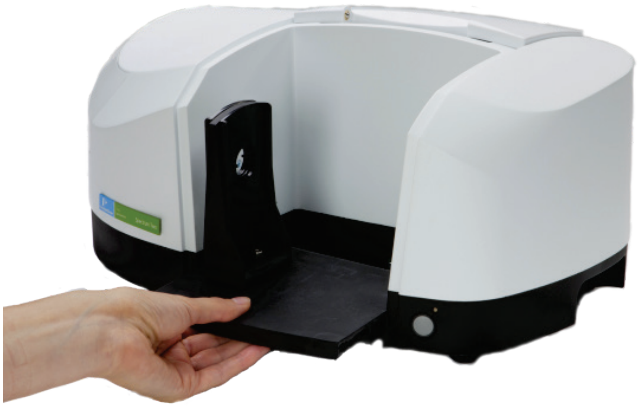


Figure 3 Removing the standard baseplate

2. Plug in the power supply to the connector located on the bottom of the HTM.

3. Route the cable under the first strain relief then snap it into the white strain relief then position cable out the front of the module.

   Ensure the power supply is connected to power and ensure power cord is safely routed to prevent accidental pulling.

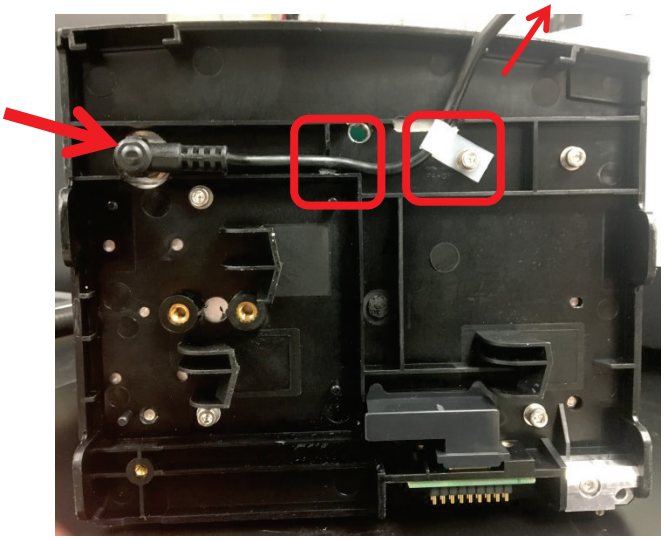


Figure 4 The power cable connected to HTM

4. Position the HTM in front of the spectrometer sample area, rest the back of it on the ledge in the sample area, and slide it into position.

5. Push it firmly back to ensure that the multi-way connector on the rear of the accessory engages properly with the spectrometer connector. There should not be any significant gaps between the HTM and the Spectrum Two N.

   When correctly installed, a seal around the inside of the HTM cover protects the optics in the sample area from contamination if samples are spilt.

Figure 5 HTM installed in a Spectrum Two N FT-NIR Spectrometer

NOTE: If you have Spectrum software and do not have an instrument installed, see the *Spectrum Administrator's Guide* (L1050095) on the *IR & Raman Manuals CD* (L1050002) for details of the Instrument Install Wizard.

Nitrogen Purge

The HTM can be purged with dry Nitrogen in order to remove ambient water vapor and carbon dioxide.

To connect the Nitrogen Purge, refer to the Spectrum Two User's Guide.

The best performance of the Nitrogen purge is achieved when the sample compartment lid is attached and lowered down.

Module Detection by Spectrum 10 Software

NOTE: The HTM will only be detected and operate correctly when used with Spectrum 10 software version 10.6.0 or later. If you are using a spectrometer purchased previously, you may also need to update the firmware. See *Appendix A: Upgrading a Spectrometer to Control the HTM* for further details.

Automatic module 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.

When the Spectrum 10 software detects the presence of the module, the HTM icon  is added to the toolbar. The **Setup Instrument Basic** (see Figure 6) and **Setup Instrument BeamPath** (see Figure 7) tabs are updated to show that the HTM is in position.

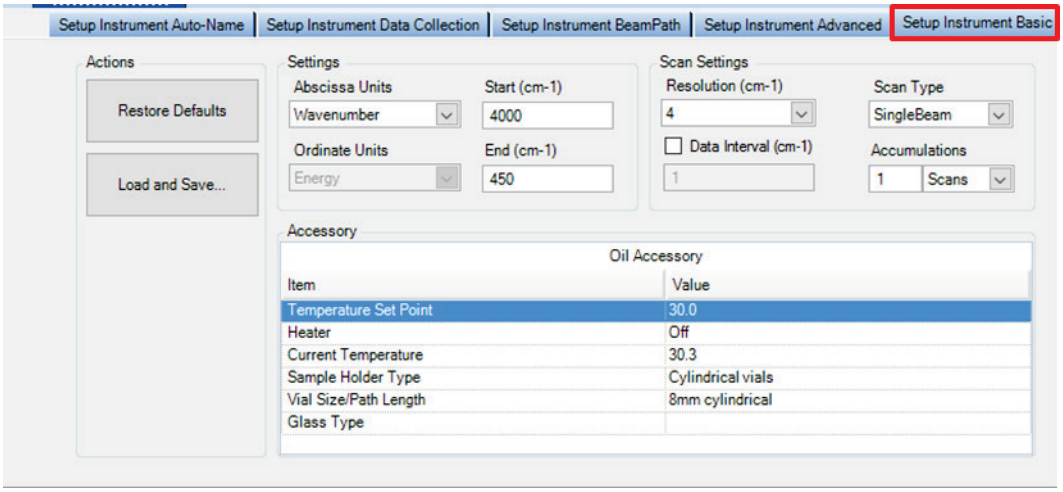


Figure 6 Setup Instrument Basic tab with HTM

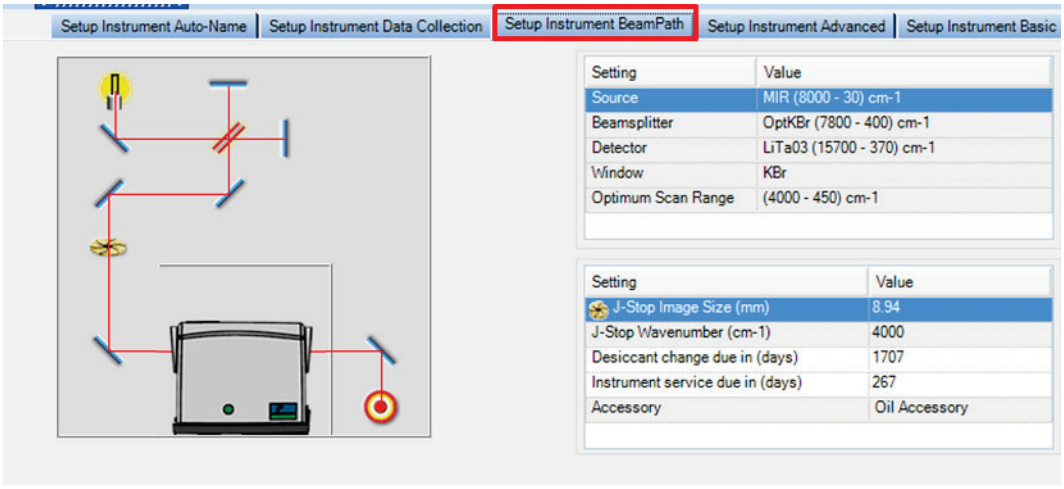


Figure 7 Setup Instrument BeamPath tab with HTM

Cuvettes and Vial Inserts

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

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.

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 from PerkinElmer.

Using the HTM Cuvettes and Vial Inserts

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.

Preparing your Sample

Little to no sample preparation is necessary when using the HTM. The HTM is particularly suited for the measurements of samples that are solid at ambient temperature but when heated above ambient are converted to the liquid phase and a much more transparent state. To prepare your sample simply fill either a disposable vial or cuvette with the sample of interest and place in corresponding HTM insert. If heating the sample, be sure to allow the sample to come to temperature and fully equilibrate prior to collecting spectra.

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.

IMPORTANT:	When handling hot oil vials take the proper safety precautions by wearing safety glasses and gloves. Use tweezers to remove the hot vials from the HTM.

CAUTION	*When working with elevated temperatures the cell holders, vials and cuvettes are HOT.*
ATTENTION	*Lorsque vous travaillez avec des températures élevées, les supports de cellules, les flacons et les cuvettes sont CHAUDS.*

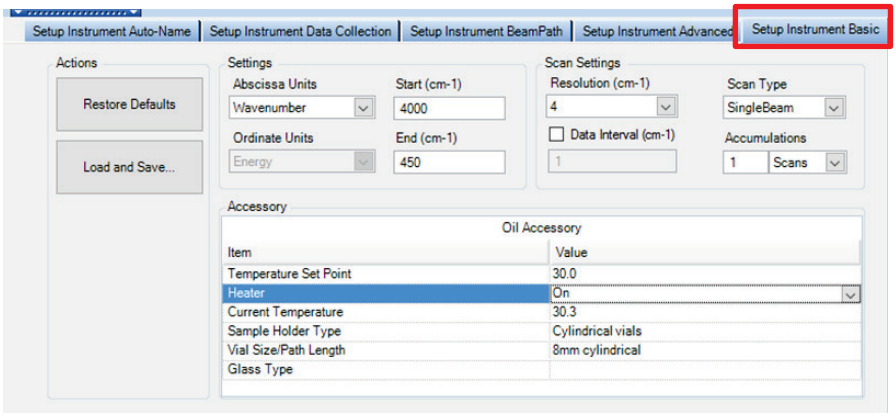
Performing a Scan in Spectrum 10

1. Select **Instrument** from the **Setup** menu.

   The Setup Instrument tabs are displayed.

2. Select the **Setup Instrument Basic** tab.

3. Set the **Temperature Set Point** for the HTM and set the heater to **On.**



   LED status on the front of the HTM:
   - Red = Unit is heating to set temperature
   - Blue = Unit is cooling to set temperature
   - Green = Unit is at set temperature
   - No light = User has heater turned off

4. Select the **Sample Holder Type**, **Size**, and **Glass Type**.

5. Check the scan and instrument parameters on the **Instrument Settings** toolbar and, if required, enter a new, unique **Sample ID** and **Description** for your sample.

   When your accessory is installed in the instrument, Spectrum 10 will default to the instrument settings that were last used to perform a successful scan with that accessory.

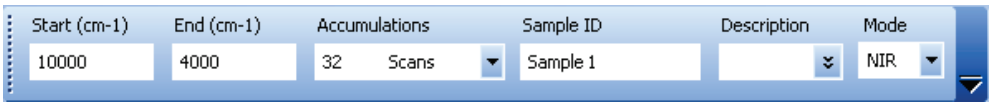


Figure 8 The Instrument Settings toolbar

NOTE: The **Sample ID** and **Description** are automatically supplied by the AutoName function. See *AutoName* in the *Setup and Administration* book in the Spectrum on-screen Help for more information.

6. Click  on the **Measurement** toolbar to collect a spectrum.

For most applications, we recommend that the default instrument settings are used. However, if you want to change the settings (for example, to collect a **Background**), see *Manual Scanning*.

Manual Scanning

1. Select **Instrument** from the Setup menu.

   The Setup Instrument tabs are displayed.

2. Select the **Setup Instrument Basic** tab.

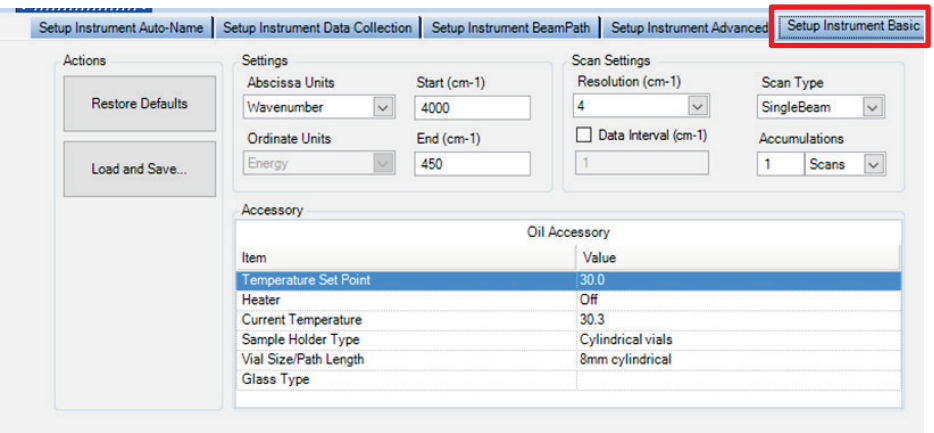


Figure 9 Setup Instrument Basic tab with Sample as the Scan Type

3. Select the **Scan Type** for your spectrum.

   The recommended setting for this option is **Sample**.
   The other options available are: Background, Interferogram, and SingleBeam. See the Spectrum on-screen Help for more information on these settings.

NOTE: You can change the abscissa units on the Setup Instrument Basic tab. The options are wavenumbers, nanometers and microns. The **Start** and **End** values will automatically update.

4. If Spectrum 10 detects that a background spectrum is required, the Scan icon will display a flag at the bottom right-hand corner  . Click the icon and a background scan will be collected before the sample spectrum.

   If at any time you wish to collect a background scan but do not wish to save it, click  .

   If you wish to collect a background and save the spectrum, select **Background** as the **Scan Type** on the Setup Instrument Basic tab (see Figure 9) and then click  .

NOTE: The Spectrum Help file describes how to format, process and report your results. To open the Help file, select **Contents** from the Help menu.

Maintenance

Cleaning the HTM

The outer casing of the HTM is designed to be easily cleaned to prevent spurious peaks occurring in the spectrum due to cross-contamination.

- Use a damp cloth to wipe off any spilt sample material.

  For water-insoluble residue, use a mild solvent such as ethanol or propanol (iso-propyl alcohol, IPA).

CAUTION	*Do not use acetone to clean the HTM.*
ATTENTION	*Ne pas utiliser d'acétone pour nettoyer le HTM.*

CAUTION	*Do not submerge the HTM or place it under running water when cleaning it.*
ATTENTION	*Ne pas submerger le HTM ou le placer sous l'eau courante lors du nettoyage.*

Cleaning the Drain Hole

After extended usage with solids that when heated turn to a liquid, spills over time my clog the drain hole.

CAUTION	*Before beginning, make sure the HTM is cool to the touch.*
ATTENTION	*Avant de commencer, assurez-vous que la HTM est froide au toucher.*

To clean the drain hole:

1. Remove the sample holder from the HTM and locate the drain hole.

   The hole goes through the HTM and exits on the bottom.

2. Using a pipe cleaner push it through the hole until it is completely unclogged.

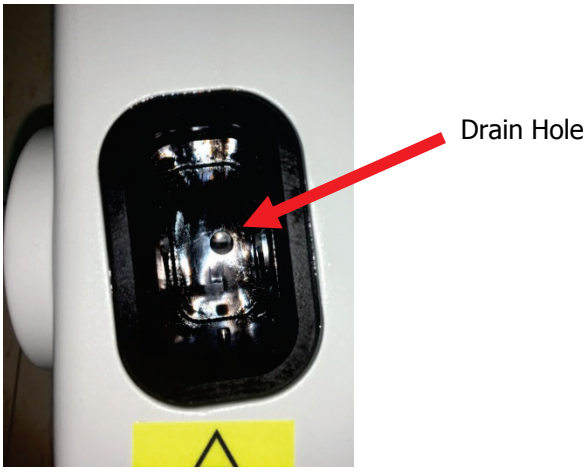


Figure 10 Drain Hole Location

3. Use a damp cloth to clean the areas and dry thoroughly.

Additional Parts

The following optional parts are available for use with the HTM.

Part Number	Description
L1390136	5 mm Vial Holder Insert
L1390137	8 mm Vial Holder Insert *Note: one is included with the module*
L1390138	12 mm Vial Holder Insert
L1390147	1 mm Cuvette Holder Insert
L1390148	5 mm Cuvette Holder Insert
L1390149	10 mm Cuvette Holder Insert
L1390187	5 mm Vials with Caps
L1390188	8 mm Vials with Caps
L1390189	12 mm Vials with Caps

Appendix A: Upgrading a Spectrometer to Control the HTM

If you have acquired a HTM accessory to be used with a previously purchased Spectrum Two spectrometer, you may need to upgrade the firmware in your spectrometer to provide the capability to control the accessory. This can be confirmed with technical support (IRTechSupport@PERKINELMER.com)

Before upgrading the firmware, you must install Spectrum software version 10.6.0 (or above) on your PC, and use it to connect to your spectrometer.

Follow the procedure below to upgrade the firmware:

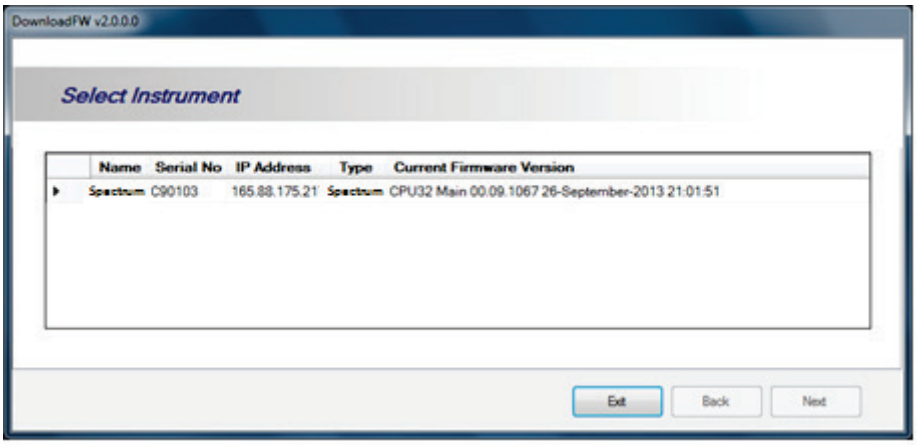
1. Ensure that Spectrum software or any other software controlling the spectrometer is closed.

2. Using your browser, navigate to:

   C:\Program Files (x86)\PerkinElmer\ServiceIR

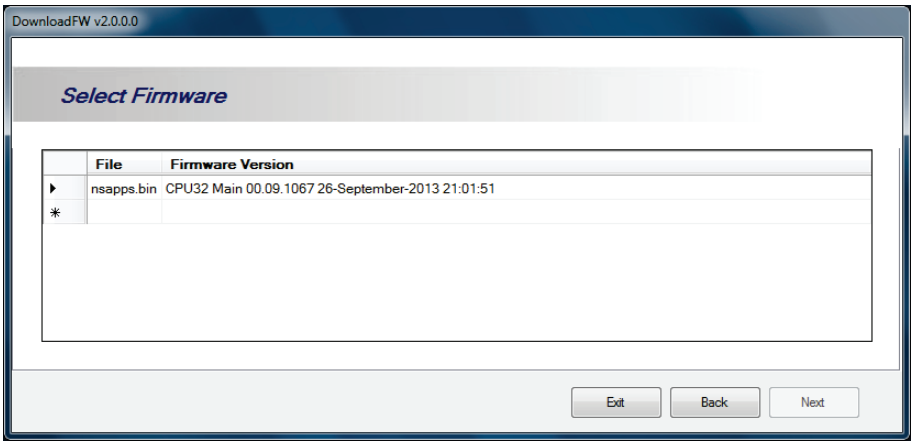
3. Double-click the **Download Firmware** shortcut file.

   Communications with the spectrometers installed on your system are established and the firmware download wizard is initiated.



4. Select the instrument you want to upgrade from the list, and then click **Next**.

   The **Select Firmware** screen is displayed.



5. Select the firmware file to use for upgrading the instrument, and then click **Next**.

Normally the default file displayed, **scrfpgapps_v4_xs.bin**, will be the latest version. If you want to use a different version, click the asterisk (*) and browse to the firmware file location on your PC.

6. Check the download details displayed on the Prepare for Download screen, and if they are correct, click **Next**.

   The firmware download is initiated.

7. After the firmware download is complete, check for any errors on the Complete screen, and then click **Exit** to close the wizard.

8. Switch off the spectrometer using the power switch at the rear of the instrument, wait 10 seconds and then switch the instrument back on.

   After the instrument has initialized, it will be capable of controlling the HTM accessory.

9. Proceed with the installation of the HTM accessory as described on page 9.