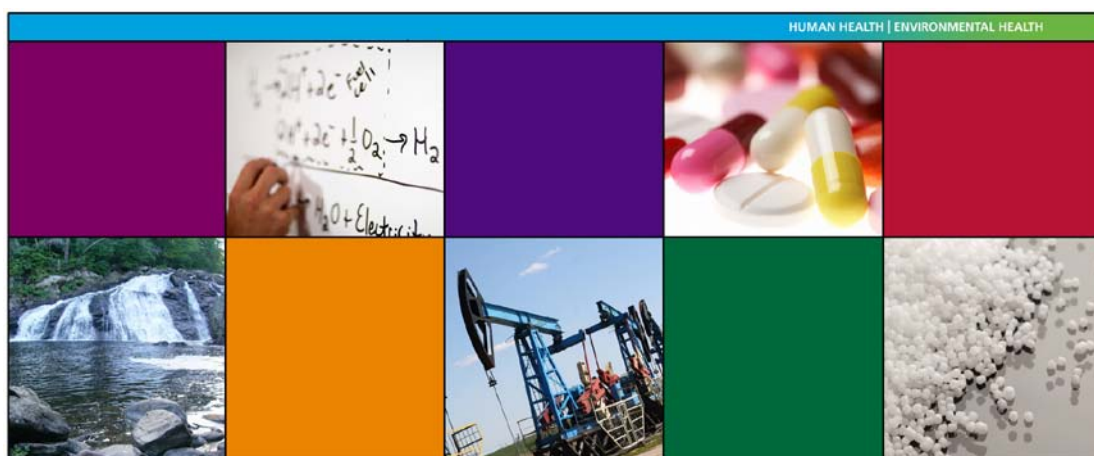


HATR SAMPLING ACCESSORY



User's Guide



Release History

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Conventions Used in this Manual

Normal text is used to provide information and instructions.

Bold text refers to text that is displayed on the screen.

UPPERCASE text, for example ENTER or ALT, refers to keys on the PC keyboard. '+' is used to show that you have to press two keys at the same time, for example, ALT+F.

All eight digit numbers are PerkinElmer part numbers unless stated otherwise.

The term 'instrument' refers to a Frontier IR System, Spectrum 400 Series or Spectrum 100 Series spectrometer, and any sampling accessory fitted.

Notes, Cautions and Warnings

Three terms, in the following standard formats, are also used to highlight special circumstances and warnings.

NOTE: A note indicates additional, significant information that is provided with some procedures.

CAUTION

*We use the term CAUTION to inform you about situations that could result in **serious damage to the instrument** or other equipment. Details about these circumstances are in a box like this one.*



WARNING

*We use the term WARNING to inform you about situations that could result in **personal injury to yourself or other persons**. Details about these circumstances are in a box like this one.*

Warnings and Safety Information

Before handling ZnSe (zinc selenide), Ge (germanium) or AMTIR-1 crystals, please ensure that you have read the appropriate Material Safety Data Sheets (MSDS).

You can search for up-to-date copies of safety data sheets on materials used in PerkinElmer products that are known to have safety issues from the Technical Resources section of the PerkinElmer website. The MSDS information is available in a range of languages, and includes data items required in specific national, supra-national and state jurisdictions.

To obtain a safety data sheet for a particular compound, follow the steps described below.

NOTE: To read MSDS .pdf files you will need Adobe Reader 5.0 or later. An installation of this software is available on the *Software Utilities CD*.

1. Launch your web browser and navigate to the PerkinElmer web site:
www.perkinelmer.com
If you are not redirected automatically you may have to select the home page appropriate to your location.
2. Search for the term MSDS using the search box located at the top of the home page. The **Search for Material Safety Data Sheets (MSDS)** page is displayed.
3. Enter the key words for the compound, in the **Product name** box, and then click **Go**. A full list of all MSDS documents that refer to the compound is displayed.
4. Select the MSDS document you want to view.

ZnSe (zinc selenide) crystals



WARNING

During routine use of your HATR Sampling Accessory the ZnSe crystal presents no hazard, but:

DO wear protective gloves when handling the crystal.

DO NOT use acids to wash the crystal because they react to emit H_2Se , which is very toxic and irritating.

DO NOT allow the crystal to come into contact with oxidizers.

The crystal is highly toxic by ingestion.

Cleaning ZnSe crystals

Avoid contact of the crystal with oxidizers and acids. ZnSe can be cleaned in pure dry acetone or methanol using a soft, lint-free cloth. Dry in a current of warm air so that there is no possibility of condensation forming on the crystal. Other suitable solvents are petroleum ether and hexane. It may also be cleaned in some commercial laboratory detergents, but they must be neutral. Alkaline solutions will slightly etch the surface, and acids will severely attack the material. A final rinse in distilled water and then drying in a current of warm air is recommended.

Ge (germanium) crystals



WARNING

During routine use of your HATR Sampling Accessory the Ge crystal presents no hazard, but:

***DO** wear protective gloves when handling the crystal.*

May be harmful if ingested in quantity, and may irritate or cause physical damage to eyes.

***DO NOT** use acids to wash the crystal.*

Ge can react violently with oxidizers, and can ignite in contact with chlorine and bromine.

Cleaning Ge crystals

Clean the crystal using an organic solvent; do not use acids or oxidizers.

AMTIR-1 crystals



WARNING

During routine use of your HATR Sampling Accessory the AMTIR-1 crystal presents no hazard, but:

***DO** wear protective gloves when handling the crystal.*

***DO NOT** use strong acids, strong bases or strong oxidizers with the AMTIR-1 crystal.*

Cleaning AMTIR-1 Crystals

Use an organic solvent to clean the crystal.

Introduction



Figure 1 The HATR Sampling Accessory

What is the HATR used for?

The HATR (Horizontal Attenuated Total Reflectance) Sampling Accessory is an internal reflection accessory used with Frontier IR System, Spectrum 100 Series or Spectrum 400 Series spectrometers for simplifying the analysis of solids, powders, pastes, gels and liquids. The technique is non-destructive.

As the beam does not penetrate too far into the sample, this technique is ideal for analyzing strong infrared absorbing solutions, such as emulsions or aqueous solutions. The technique can also prove useful in measuring homogenous solid samples, solid surfaces and coatings on solid samples.

How it works

The technique involves placing a sample on top of a crystal with a high refractive index. An infrared beam from the instrument is passed into the accessory and up into the crystal. It is then reflected internally along the crystal, and back towards the detector that is housed within the instrument. Each time the beam is reflected within the crystal, it penetrates into the sample by a few microns. Figure 2 illustrates this process.

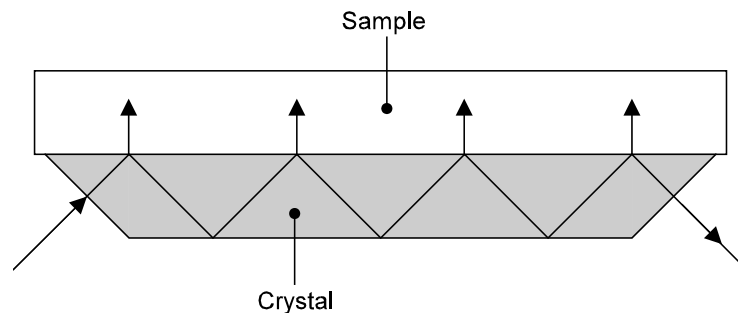


Figure 2 Principle of HATR operation

Choosing the top plate

The HATR can be used to analyze powders, pastes, gels and liquids using a trough top plate, where the sample is poured into the trough. Alternatively, a flat plate may be used for homogenous solid samples, solid surfaces, or coatings on solid samples. Force may be applied to ensure good contact between the sample and the crystal. The flat plate can also be used to analyze gels and pastes, by spreading them on the surface of the plate.

The HATR can be supplied with top plates fitted with zinc selenide (ZnSe), germanium (Ge) or AMTIR-1 crystals. Consult your PerkinElmer Sales Representative for further information about available top plates.

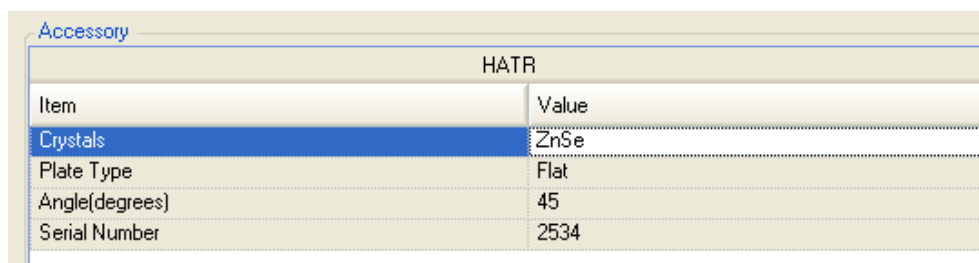
Choosing the correct crystal

Zinc selenide (ZnSe) crystals are most commonly used as they have a wide range (650–17000 cm^{-1}) and a refractive index of 2.4. However, zinc selenide is incompatible with many acids and bases. Therefore AMTIR-1 is often used as its properties are very similar, but it is more resistant to acids.

Germanium (Ge) crystals have a high refractive index of 4.0 and are used for highly absorbing samples such as carbon-filled polymers and rubbers, and samples with a high water content. These crystals are also resistant to strong acids and bases.

NOTE: Derivative shaped bands can be observed in the spectrum if the refractive index of the sample is too close to the refractive index of the crystal. For this reason, alternative crystal types may be required for specific sample types. Consult your PerkinElmer Sales Representative for advice.

Details of the top plate crystal are displayed in Spectrum software, and this information is stored in the status information for each spectrum collected.



The screenshot shows a software window titled 'Accessory' with a sub-tab 'HATR'. It contains a table with two columns: 'Item' and 'Value'. The 'Crystals' row is highlighted in blue.

Item	Value
Crystals	ZnSe
Plate Type	Flat
Angle(degrees)	45
Serial Number	2534

Figure 3 Top plate details displayed on the Setup Instrument Basic tab in Spectrum software

Please ensure that you have read the safety information starting on page 4 of this User's Guide for your crystal type before continuing.

Choosing the angle of the crystal face

The number of internal reflections and depth of penetration of the beam can be decreased and absorbance bands in the spectrum weakened by using, for instance, a 60° crystal rather than a 45° crystal.

Installing the Accessory

Fitting the Accessory

The HATR Sampling Accessory can be simply installed into the sample compartment of the spectrometer, after first removing any other sampling accessory that is currently fitted.

To remove the current accessory and then install the HATR:

1. Raise the sample cover to the vertical position, press the release clip and lift the cover upwards, clear of the instrument.
Store it in a safe place for future re-use.
2. Reach in under the base of the current accessory and pull the release handle towards you to release the accessory.
3. Now simply slide the accessory out of the sample area (Figure 4).
Store it in a safe place for future re-use.



Figure 4 Removing the basic sample slide

- Slide in the HATR Sampling Accessory, rest the back of it on the ledge in the sample area and slide it into position. Push it firmly home to ensure that the connector on the rear of the HATR Sampling Accessory mates properly with the spectrometer connector.



Figure 5 Installing the HATR accessory

- Place the required top plate (trough or flat plate type) on the HATR accessory, with the arrows pointing towards the back (Figure 6).



Figure 6 HATR accessory top plate with arrows circled

The HATR Sampling Accessory is now ready for use.




Figure 7 HATR installed in a Frontier IR System

NOTE: When the HATR is installed, a connection is made between the accessory and the purge system used in the instrument. To purge the accessory, use the left-hand (Sample) purge connector on the rear of the instrument. The right-hand purge connector purges the instrument.

Accessory Detection by Spectrum Software

NOTE: If you do not have an instrument installed, see the Administrator's Guide for your software for details of the Instrument Install Wizard.

The Spectrum software detects the presence of the accessory, and the HATR icon  is added to the toolbar. The Setup Instrument Basic (Figure 8) and Setup Instrument BeamPath (Figure 9) tabs are updated to show that the HATR Accessory is in position.

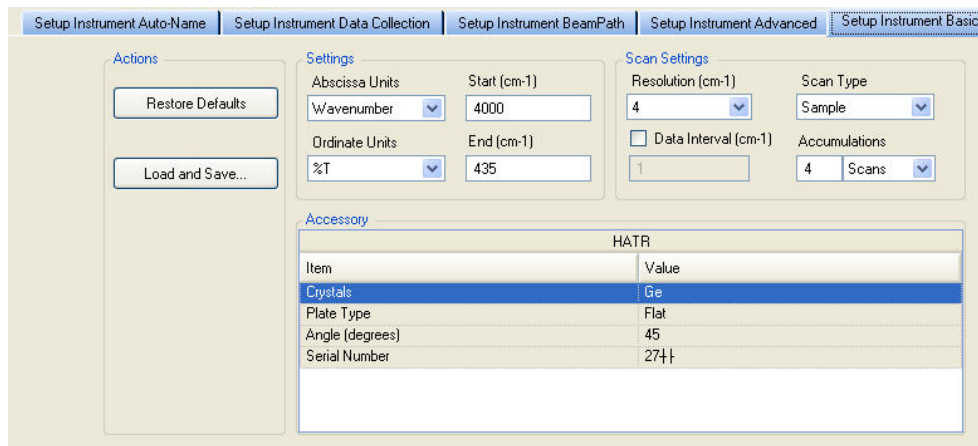


Figure 8 Setup Instrument Basic tab with HATR

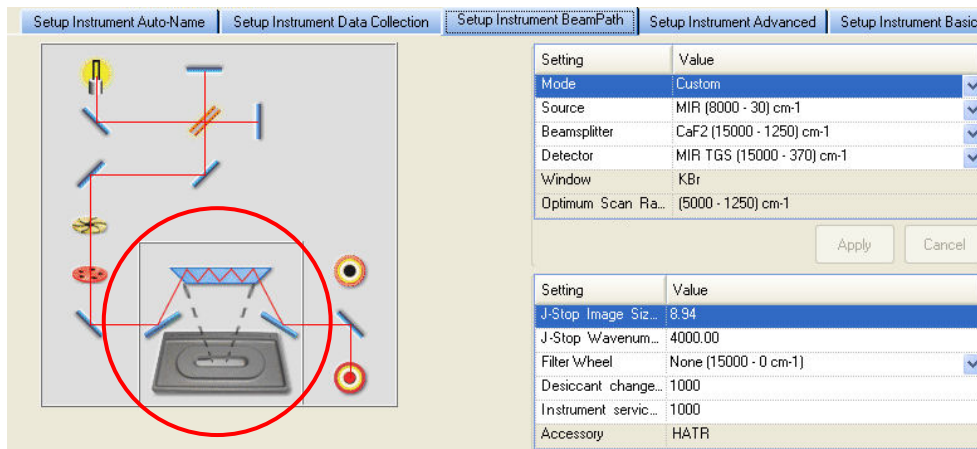


Figure 9 Setup Instrument BeamPath tab with HATR icon circled

Contamination Check

The surface of the crystal must be clean before a measurement is taken. The contamination check can be used to ensure this. If the crystal is not properly cleaned, you may observe negative bands in the spectra.

Cleaning the crystal

Once data has been collected, clean the crystal using a cotton bud or cotton wool moistened with water or an organic solvent. Take care not to scratch the surface of the crystal. Ensure the crystal is completely dry before re-use.

Top plates can be cleaned at the same time as the crystal. However, we recommend that you remove the top plate from the accessory before cleaning, as a safeguard against spillage.

Do not leave the top of the accessory uncovered for long periods of time. Always place the dust cover over the accessory when the top plate is removed.

Contamination Ready Check

<p>NOTE: For information about setting up Ready Checks see the Spectrum on-screen Help. The following description assumes that Ready Checks are already set up and enabled.</p>
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To perform a Ready Check:

1. From the Measurement menu, select the Instrument Checks sub-menu and then **Contamination** from the Ready Checks available.
The Ready Checks dialog is displayed.
2. Make sure that you have removed your sample and cleaned the top plate, and then click **Scan**.
A new background spectrum is collected, compared to the reference background spectrum and the result of the test is displayed.
3. If required, click the link that enables you to see a print preview of the Instrument Ready Checks Report.

Applying Pressure

Good contact between the sample and the surface of the crystal is important to prevent loss of beam penetration. To aid this, a pressure arm can be used to apply controlled force to the sample to make better contact with the crystal. The pressure arm is used in conjunction with either a volatiles cover (part number L1205436) or a powder press (part number L1201944), as described in Table 1.

Table 1 Use of volatiles cover and powder press

Item	Flat top plate	Trough top plate
Volatiles cover	Used to apply pressure to polymer films or laminates.	Used to prevent evaporation (without use of pressure arm).
Powder press	Not applicable.	Used to apply pressure to ensure good contact when analyzing powders.

Using the Accessory with Spectrum Software

The following pages outline the procedure for obtaining a sample spectrum using a Frontier instrument fitted with an HATR and running Spectrum software.

For further information on how to use Spectrum software, see the on-screen help provided.

1. Enter the required scan and instrument parameters in the Instrument Settings toolbar. When your accessory is installed in the instrument, Spectrum will default to the instrument settings last used to perform a successful scan with that accessory.

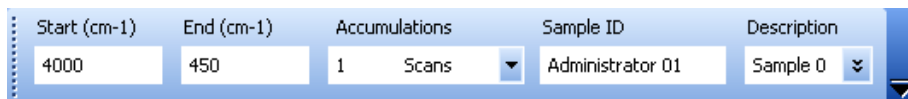



Figure 10 The Instrument Settings toolbar

If you want to set instrument parameters that are not displayed in the Instrument Settings toolbar, select **Instrument** from the Setup menu. The Setup Instrument pages are then displayed in the Dialog Pane at the bottom of the workspace.

Figure 8 on page 11 shows the Setup Instrument Basic tab.

2. If you need to collect a background spectrum, ensure that the top plate is clean and

then click  on the Measurement toolbar.

Spectrum automatically alerts you when you need a new background, and can be configured to request a new background at set intervals (on the Setup Instrument Data Collection tab).

NOTE: If you want to collect a background that will be added to the Sample View and can be saved separately, then select Background as the **Scan type** on the Setup Instrument Basic tab.

3. Place your sample on the top plate.
4. If appropriate, place a powder press or volatiles cover over the sample.

5. If you are using a powder press swing the pressure arm round from the docked position until it is above the center of the cover as shown in Figure 11.



Figure 11 Pressure arm in operating position, over a powder press

CAUTION

The pressure arm must NOT be used without a volatiles cover or powder press, as this will result in damage to the top plate.

When using a flat top plate and a solid sample, the surface of the sample must be flat. You must not try to correct for uneven surfaces by applying extra force as you may damage the crystal. Samples with uneven surfaces should be ground to a powder and analyzed using a trough top plate.

6. Screw down the pressure pin to apply force to the sample.

CAUTION

Excessive force can crack and permanently damage the crystal in the top plate or damage the pressure arm. Be careful not to apply too much force.

7. Enter a suitable **Sample ID** and **Description** in the toolbar, and then click  to enter Preview mode.

The Live tab is displayed.

NOTE: If you have deselected the **Preview** checkbox on the toolbar, you should select the **Monitor** option from the Measurement menu in order to enter Preview mode.

- Screw down the pressure pin to apply the minimum force to the sample required to give a good spectrum, as shown on the Live tab.

If an adequate spectrum, with a transmission level of between 40 and 80%, cannot be achieved, the pressure should be released and a new sample prepared with an increased or decreased amount of material.

The **Force Gauge** area of the dialog will display the force being applied. If the indicator bar turns red, then so much force is being applied that the pressure arm and/or the crystal is in danger of being damaged.

NOTE: The Force Gauge area is not displayed when the HATR is used in conjunction with a Spectrum 65 spectrometer.

NOTE: If you want to compare various samples, or use quantitative techniques, ensure you use the same amount of force each time.

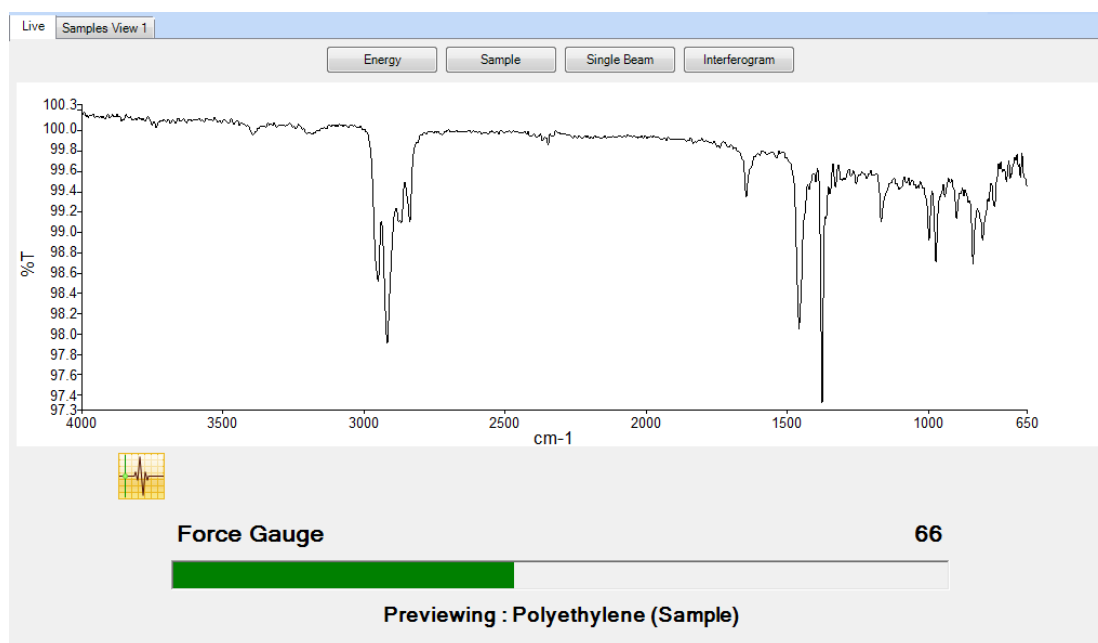



Figure 12 Force Gauge indication in Spectrum software

You are now ready to collect data.

- Click  to obtain the sample spectrum.

The **History** settings of your sample will contain information about the HATR accessory type.

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

