SPECTRUM TWO HATR

User's Guide

PerkinElmer®

Release History

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Contents

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 the Spectrum Two FT-IR 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), KRS5 (thallium bromo-iodide) 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.

KRS5 (Thallium bromo-iodide) Crystals

WARNING

During routine use of your HATR, the KRS5 crystal presents no hazard, but:

***DO** wear protective gloves when handling the crystal. **DO NOT** allow the crystal to come into contact with oxidizers.*

*We recommend that you **DO NOT** wash the crystal with water.*

The crystal is extremely toxic by ingestion, and is harmful in contact with skin. May evolve toxic fumes in a fire.

Cleaning KRS5 Crystals

Clean the crystal using an organic solvent; do not use an oxidizer.

Introduction

The Horizontal Attenuated Total Reflectance (HATR) Accessory is an internal reflection accessory used for simplifying the analysis of solids, powders, pastes, gels and liquids (Figure 1). The technique is non-destructive.

Figure 1 The Horizontal Attenuated Total Reflectance (HATR) Accessory

As the beam does not penetrate 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 spectrometer. Each time the beam is reflected within the crystal, it penetrates into the sample by a few microns. Figure 2 illustrates this process.

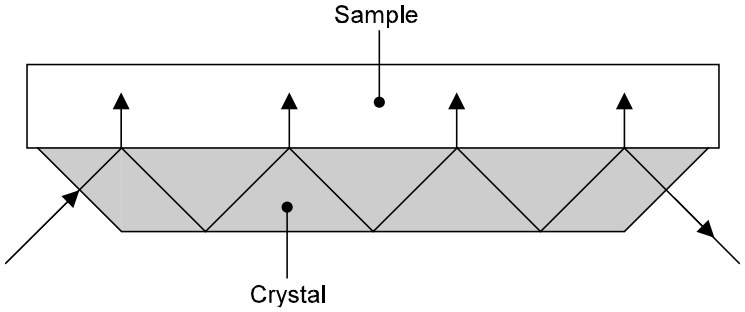

Figure 2 Principle of HATR operation

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 is supplied with a top plate fitted with a ZnSe crystal. Top plates fitted with germanium (Ge), KRS5 or AMTIR-1 crystals (Table 1) are available.

Table 1 HATR top plates

	Sample trough	Flat sampling plate
ZnSe	L1360266	L1360267
Ge	L1360268	L1360269
AMTIR-1	L1360270	L1360271
KRS5	L1360272	L1360273

Choosing the correct crystal

For most applications involving organic materials, ZnSe is the most useful HATR crystal material. It combines a relatively wide transmission range (17 000–650 cm^{-1}), a high refractive index (2.4) and hardness, and is insoluble in distilled water. ZnSe is, however, incompatible with many acids and bases.

AMTIR-1 has a similar hardness and refractive index to ZnSe, so absorption bands will be of similar intensity to within approximately 10%. The transmission range is slightly narrower than ZnSe (11 000–700 cm^{-1}). AMTIR-1 is more resistant than ZnSe to acids (to approximately 6 N), and is therefore more appropriate for use with some acidic solutions. We recommend that you DO NOT use AMTIR-1 with strong acids, strong bases and strong oxidizers.

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 Customer Care Representative for advice.

You must verify that the sampling crystal is not soluble or otherwise damaged by the sample. A summary of the physical properties of commonly used crystal materials is provided in Table 2.

NOTE: We recommend that you remove the sample plate from the HATR when you clean the crystal.

Table 2 Properties of crystal materials

	ZnSe	Ge	KRS5	AMTIR-1
ATR range (cm^{-1})	17 000–650	5500–600	14 000–400	11 000–700
Refractive index at 1000 cm^{-1}	2.4	4.0	2.37	2.50
Density (g/cm^3)	5.27	5.32	7.37	4.4
Max. useful temp. in air (°C)	300	270	200	300
Hardness (Knoop #)	150	1150	40.2	170
Cleaning agents	acetone, water	toluene, water	MEK	organic solvents
Solvents that attack material	acids, strong alkalis	hot H_2SO_4, aqua regia	complexing agents	strong acids, strong bases, strong oxidizers
Remarks	hard, easily cracked	hard and brittle, high reflection loss	deforms under pressure, toxic, does not cleave	hard and brittle

NOTE: The ATR crystal is bonded to the sample plate with an adhesive, we therefore recommend that you do not leave the crystal in contact with solvents, particularly chlorinated solvents, for long periods.

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

Installing the Accessory

CAUTION	*The mirrors in the HATR are aluminum-coated and, although they are durable, their surfaces are relatively soft and difficult to clean without scratching. Therefore, take care to avoid dropping materials on the mirrors when you are handling the unit and placing samples on the sampling surface.*

The HATR is pre-mounted on the Spectrum Two baseplate for quick installation into the sample compartment of the spectrometer.

To remove the current accessory and then install the HATR:

1. Open the sample cover of the Spectrum Two, if fitted.

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

   Store it in a safe place for future re-use.

Figure 3 Removing the sampling accessory

3. Start Spectrum software and connect to the instrument.

4. Select **Monitor** from the Measurement menu in Spectrum software and record the energy throughput of the open beam.

   When you have finished, click **Halt.**

5. If you are using the flat sampling plate or analyzing powder samples using the trough sampling plate, ensure that the pressure clamp is fitted before you install and align the HATR.

   If required, follow the instructions to install the pressure clamp in *Installing the Pressure Clamp* on page 17.

6. Slide in the HATR accessory baseplate (Figure 4). Push it in firmly to ensure that the connector on the rear of the baseplate connects properly with the spectrometer connector.

Figure 4 Installing the HATR accessory

7. Place the required top plate (trough or flat plate type) on the HATR accessory.

Although the HATR is pre-mounted on the baseplate, we recommended that you maximize the energy throughput of the system as described in *Aligning the HATR* on page 14 after installing the accessory, or after changing between top plates with crystals of very different refractive index.

Accessory detection by Spectrum software

When the accessory is installed in the sample compartment, Spectrum software detects it, and the Setup Instrument Basic (Figure 5) and Setup Instrument BeamPath (Figure 6) tabs are updated to show that the HATR Accessory is in position. The Accessory toolbar displays the HATR icon  .

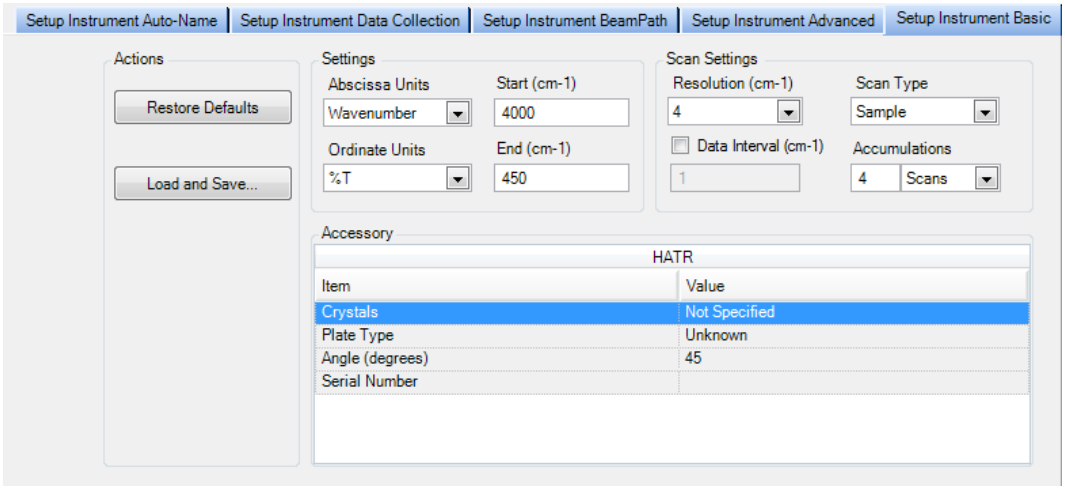

Figure 5 Setup Instrument Basic tab with HATR

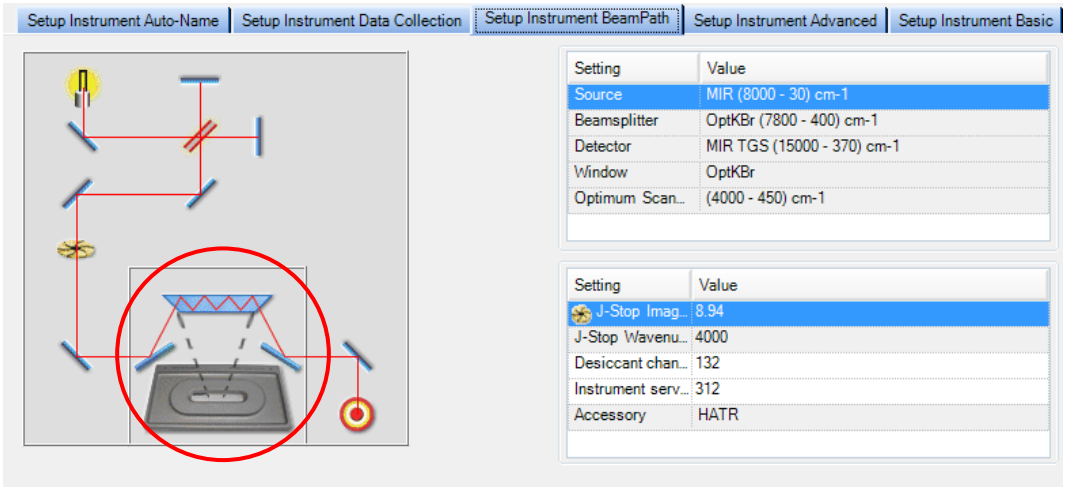

Figure 6 Setup Instrument BeamPath tab with HATR icon circled

Aligning the HATR

Make sure that the sample trough or the flat sampling plate and, if applicable, the pressure clamp are properly mounted on the base assembly before aligning the HATR.

1. In Spectrum software, select **Monitor** from the Measurement menu.
   The Live tab is displayed.

2. Record the energy throughput of the HATR.

3. Compare this value with the value for the energy throughput of the open beam (recorded at the beginning of the installation procedure – Step 4 on page 11).
   If you did not record the value, remove the HATR from the spectrometer and then measure the value.
   If you do not observe an energy throughput, or if you are aligning your accessory for the first time, continue at Step 4.
   If you do observe an energy throughput, and your accessory has been aligned previously, continue at Step 15.

4. Click **Halt** to exit the Live tab.

5. Insert the alignment tool in position on the baseplate (Figure 7).
   Press the alignment tool mount firmly to locate the two pins on the base of the alignment tool into the holes on the baseplate.

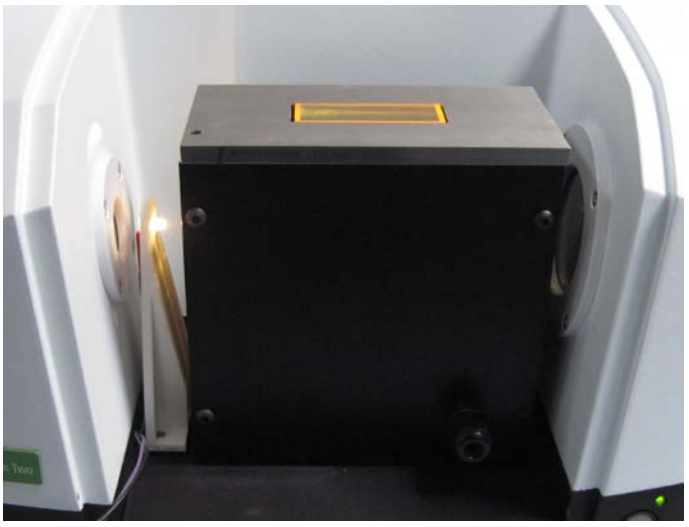

Figure 7 Alignment tool in position on the HATR baseplate

6. Connect the 15-way connector on the cable of the alignment tool into the EXT DETECTOR port on the rear of the spectrometer (Figure 8).
   The white LED on the alignment tool should be lit.

Figure 8 Alignment tool connected to EXT DETECTOR port

7. Make sure that mirrors 1 and 4 (Figure 9) are against their alignment pins, using the wrench supplied.

   The top plate of the accessory can be removed to confirm this. Figure 9 shows the mirrors and the corresponding adjustment nuts on the rear of the HATR are shown.

   Mirror 1 should be rotated fully clockwise and mirror 4 should be rotated fully counterclockwise, looking from the front of the sample compartment.

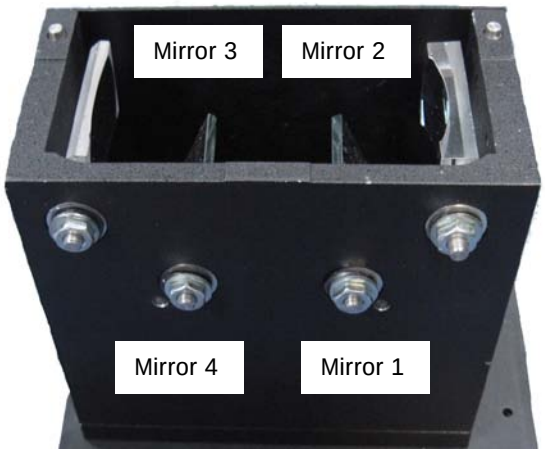

Figure 9 HATR with top plate removed (viewed from rear to show the adjustment nuts)

Figure 10 shows the optical path of the accessory, viewed from the rear as in Figure 9.

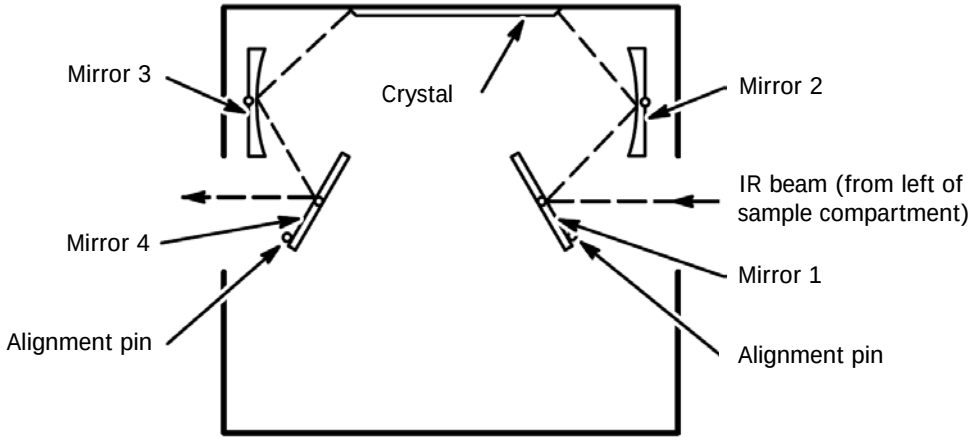

Figure 10 Optical path of the accessory

NOTE: Take care when adjusting mirrors 2 and 3. They are very sensitive to rotational adjustment.

8. Place the top plate on the accessory.

9. Look at the crystal from above, and gently adjust mirror 2 until the crystal is illuminated as brightly as possible.

10. Place a card between the exit port of the HATR and the window on the right of the sample compartment.

11. Adjust mirror 3 until the movable part of the beam is centered on the exit window.

12. Remove the card from the HATR.

13. Remove the white-light alignment tool from the baseplate.

14. Select **Monitor** from the Measurement menu.

    The Live tab is displayed.

15. Gently adjust mirror 2, using a wrench, until you obtain an energy maximum.

16. Repeat for mirror 3.

NOTE: You may need to adjust mirror 2 and mirror 3 several times to find the optimum position.

17. Record the maximum throughput energy and then click **Halt**.

18. Compare the maximum energy with that recorded at the beginning of the installation procedure (Step 4 on page 11).

    If the energy throughput of the HATR is 15–25% of the energy throughput of the open beam, no further adjustments are needed.

    If the energy throughput of the HATR is lower than 15% of the energy throughput of the open beam, repeat the alignment procedure.

Purging the HATR

With the Spectrum Two spectrometer, you can purge the HATR rather than the sample compartment. In this way the purge is maintained when you change sample (provided that you do not remove the top plate) and the purge volume is less.

Use polyethylene tubing with a 6 mm external diameter and a 4 mm internal diameter to carry the gas to the purge inlet on the HATR.

1. Loosen the knurled nut on the HATR (Figure 11).

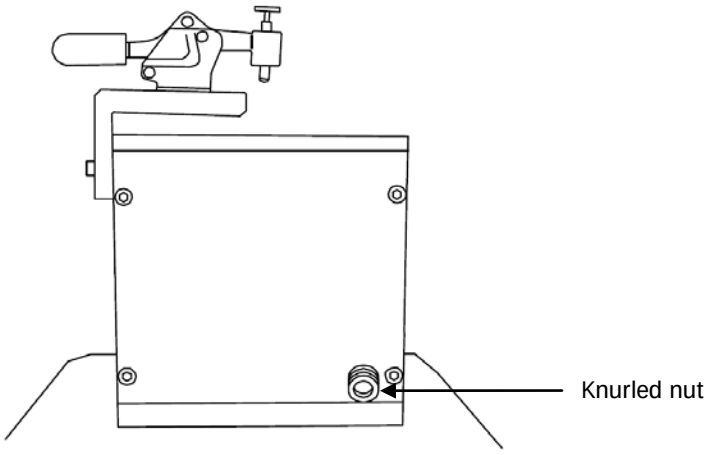

Figure 11 The purge connection on the HATR

2. Push the tube through the nut until it rests against the interior connector.
3. Tighten the nut to clamp the tube in place.
4. Purge the HATR using a flow rate of approximately 1 or 2 l/min.
5. Monitor the baseline until H_2O/CO_2 vapor is reduced or negligible.
   You should turn off the atmospheric correction in Spectrum software, by deselecting the **CO2/H2O** check box on the Setup Instrument Advanced tab.

Installing the Pressure Clamp

CAUTION	*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 used on a trough top plate.*

It is important to have good contact between the sample and the surface of the crystal to prevent loss of beam penetration. To aid this, an optional pressure clamp can be used to apply controlled force to the sample to make better contact with the crystal.

The pressure clamp enables you to improve the contact between a film sample and the sample plate. When analyzing powder samples, it can also be used to apply pressure to powder in the sample trough. The pressure clamp is used in conjunction with either a volatiles cover (L1205436) or a powder spacer (L1362431), as described in Table 3.

Table 3 Use of volatiles cover and powder spacer

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 clamp).
Powder spacer	Not applicable.	Used to apply pressure to ensure good contact when analyzing powders.

Your accessory may have been supplied with the pressure clamp installed. If, however, you need to fit the pressure clamp, refer to the following procedure.

1. Attach the clamp to the body of the HATR using the two screws provided (Figure 12).

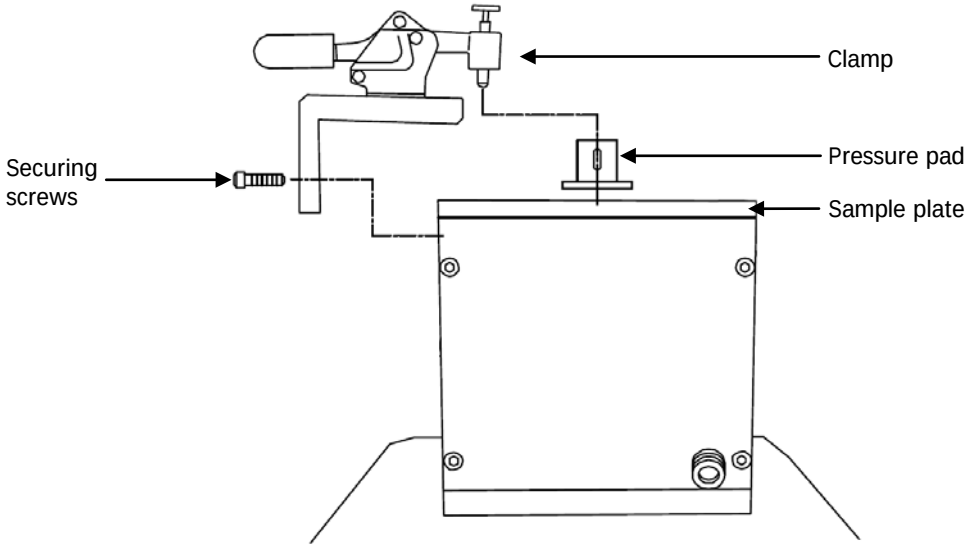

Figure 12 Installing the pressure clamp

2. Place the pressure pad on the sample plate, in the V-shaped slot in the clamp, so that the graduated scale on the pressure plate faces toward you.

3. Push the lever of the clamp into position so that pressure is applied to the sample plate.

Adjusting the pressure

The clamp is supplied pre-set to apply approximately 50 newtons of pressure to a flat crystal assembly (45° ZnSe). This is a satisfactory setting for most applications.

1. Read the scale on the pressure pad and use Figure 13 to calculate the approximate pressure being applied.

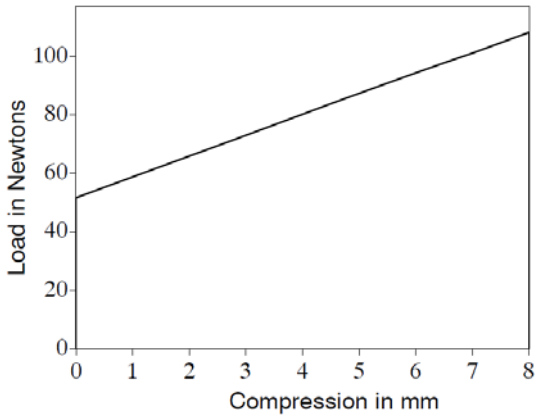

Figure 13 Pressure as a function of the spring height of the clamp

2. To adjust the pressure, turn the screw on the top of the clamp.

CAUTION	*Excessive pressures can crack and permanently damage the ATR crystal. Do not use the pressure clamp to crush the sample, and do not apply pressure to the crystal for extended periods of time.*

Using the Accessory with Spectrum Software

CAUTION	*Excessive pressures can crack and permanently damage the ATR crystal. Do not use the pressure clamp to crush the sample, and do not apply pressure to the crystal for extended periods of time.*

The Measurement bar (Figure 14) displayed by default at the top of the workspace includes the tools you need to collect a spectrum from a sample. You can also select these commands from the Measurement menu.

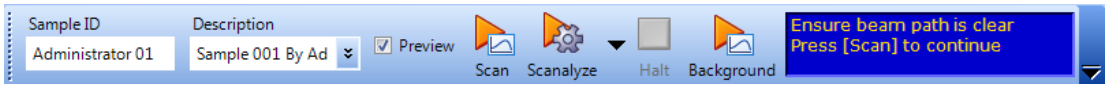

Figure 14 Spectrum Two Measurement bar

To perform a scan:

1. Ensure that the required scan and instrument parameters have been set.

   When you first fit the accessory, the software automatically sets default scan parameters to values that are appropriate to the HATR.

   If you want to set any of the instrument parameters that are not included on the toolbars, select **Instrument** from the Setup menu to display the Setup Instrument tabs.

2. If you want, enter a unique **Sample ID** and **Description** for the sample on the Measurement bar.

3. Ensure that the top plate is clean and then click  to collect a background spectrum.

4. Place your sample on the top plate and, if appropriate, place a powder spacer or volatiles cover over the sample.

   Refer to *Sample Preparation* on page 22 for more details of how to prepare your sample for analysis.

5. If **Preview** is selected on the Measurement bar, click  to view the spectrum.

NOTE: If **Preview** is not selected, you can preview the spectrum by selecting **Monitor** from the Measurement menu.

The Live tab is displayed. The spectrum is displayed, but no data is recorded.

6. If you are using the sample plate and pressure clamp, or collecting a spectrum from a powder using the sample trough and powder spacer, adjust the pressure of the clamp to find the optimum pressure for your sample.

   Slowly turn the screw on the clamp to increase the pressure while observing the intensity of the peaks in the sample spectrum. Continue to adjust the pressure until the peaks do not change any further.

   If the intensity of the peaks does not improve (transmission greater than 90%), you may need to place more sample on the top plate, or prepare the sample again as there is poor contact.

7. Click  again to collect the spectrum.

   By default, during scanning the sample data is displayed on the Live tab in the Viewing Area.

   The completed spectrum is displayed on the Graph tab, and added to the current Samples View in the Data Explorer.

   If, for any reason, you want to stop scanning your sample, click  .

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

8. Clean the sample off the crystal, using a cotton swab moistened with an appropriate solvent (refer to Table 2 on page 10).

NOTE: You can use the ATR correction to enable you to compare spectra collected using the HATR with transmission spectra. See the Spectrum on-screen Help system for more information.

Sample Preparation

To maximize the performance of your system, you must use the sample plate and crystal best suited to your application; for information on crystals see Table 2 on page 10.

For information on collecting spectra using Spectrum software, refer to *Using the Accessory with Spectrum Software* on page 20.

Analyzing Liquids, Pastes and Gels

When analyzing liquids, pastes and gels, we recommend that you use the sample trough.

1. Collect a background spectrum with the empty, clean sample trough.
2. Place your sample material into the sample trough.

   Make sure the sample covers the entire crystal surface.

   If the sample is volatile, place the volatiles cover (L1205436) on top of the sample trough. This prevents vapor entering the beam path and contributing to the collected spectrum.
3. Collect the sample spectrum, ratioing against the background spectrum.
4. Clean the sample off the crystal, using a cotton swab moistened with an appropriate solvent (see Table 2 on page 10).

Analyzing Powders

When analyzing powders, we recommend that you use the sample trough and apply pressure to the powder using the pressure clamp.

1. Make sure that the pressure clamp is properly installed

   If required, refer to *Installing the Pressure Clamp* on page 18.
2. Collect a background spectrum with the empty, clean sample trough.
3. Place the powder sample in the sample trough to a depth of approximately 1 mm, then level the powder with a spatula.

   Make sure the sample covers the entire crystal surface.
4. Place the powder spacer (L1362431) on top of the powder, and then place the pressure pad on the powder spacer (Figure 15).

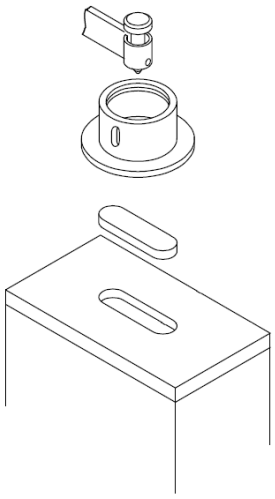

Figure 15 Using the trough plate and pressure clamp for analyzing powders

5. Place the pressure pad in the V-shaped slot in the clamp, so that the graduated scale on the pressure plate faces toward you.

6. Apply pressure to the sample with the clamp.

CAUTION	*Excessive pressures can crack and permanently damage the ATR crystal. Do not use the pressure clamp to crush the sample, and do not apply pressure to the crystal for extended periods of time.*

7. Collect the sample spectrum, ratioing against the background spectrum.

8. Clean the sample off the crystal, using a cotton swab moistened with an appropriate solvent (see Table 2 on page 10).

Analyzing Films

When analyzing films, we recommend that you use the flat sampling plate.

1. Make sure that the pressure clamp is properly installed.
   If required, refer to *Installing the Pressure Clamp* on page 18.

2. Collect a background spectrum with the empty, clean sample plate.

3. Place the sample on the sample plate.
   Make sure that the sample covers the entire crystal surface.

4. Apply pressure to the sample with the clamp.

CAUTION	*Excessive pressures can crack and permanently damage the ATR crystal. Do not use the pressure clamp to crush the sample, and do not apply pressure to the crystal for extended periods of time.*

5. Collect the sample spectrum, ratioing against the background spectrum.

6. Clean the crystal, using a cotton swab moistened with an appropriate solvent (see Table 2 on page 10).

Contamination Check

ATR is a surface measurement technique. Therefore, it is important that the surface of the crystal is clean before a measurement is taken. To ensure that the crystal is clean, use the Contamination Check. If the crystal is not properly cleaned, you may observe negative bands in the spectra.

Cleaning the crystal

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

Contamination Ready Check

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.

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.