HATR SAMPLING ACCESSORY

User's Guide

PerkinElmer®
For the Better

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 either the Spectrum 100 FT-IR or Spectrum 100N FT-NIR 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

ZnSe (Zinc Selenide) Crystals

WARNING

During routine use of your HATR, 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.

Hazard Class	6.1
UN Number	2811
CAS Number	1315-09-9

Physical Data

Description	Yellow
Melting point	1525 °C
Boiling point	Not applicable
Specific gravity	Not applicable
Solubility in water	Immiscible or insoluble
Vapor pressure	Not applicable
Vapor density	Not applicable

Fire and Explosion Hazard

May evolve toxic fumes in a fire	
Description	Yellow
Explosive limits	Not applicable
Auto-ignition temperature	Not applicable
Firefighting measures	Use extinguisher suitable for surrounding fire

Health Hazard

In contact with gastric juices, very toxic and irritating H_2Se is evolved. Chronic effects: can cause dermatitis and digestive disturbances. Garlic odor of breath is a common symptom. May cause pallor, nervousness and depression	
Toxicity data	TLV (soluble compounds) 0.2 mg/m^3 as for selenium compounds; LD_{50} unknown
Carcinogenicity	No evidence of carcinogenic properties
Mutagenicity / Teratogenicity	No evidence of mutagenic or teratogenic effects
Exposure limits OES, mg / m^3	0.2 Se (Long-term, 8 hour TWA)

First Aid

Affected area		**Emergency First Aid**
Eyes	Harmful	Irrigate thoroughly with water for at least 10 minutes; OBTAIN MEDICAL ATTENTION if discomfort persists
Lungs	Irritates	Remove from exposure, rest and keep warm; OBTAIN MEDICAL ATTENTION in severe cases
Skin	Irritates	Wash thoroughly with soap and water; OBTAIN MEDICAL ATTENTION if discomfort persists
Mouth		Wash out mouth thoroughly with water; OBTAIN MEDICAL ATTENTION

Reactive Hazards

Stability	Stable
Reaction with water	May emit hydrogen selenide (H_2Se)
Decomposition products	Highly toxic fumes. Decomposition results from extreme heat temperatures greater than 400 °C especially in oxidizing atmosphere
Other known hazards	Reacts with acids to give highly toxic hydrogen selenide (H_2Se)
Avoid contact with:	Water (no)
	Acids (yes)
	Bases (no)
	Oxidizers (yes)
	Combustibles (no)

Spillage disposal

Wear appropriate protective clothing	
Small spillage	Wear gloves and goggles. Spread sand over the spillage and carefully sweep up. Arrange for removal by a disposal company. Clean site of spillage thoroughly using a neutral solution of water and detergent

Storage and Handling

Special requirements	Wear protective gloves when handling the crystal

Ge (Germanium) Crystals

WARNING

During routine use of your HATR, 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.

Hazard Class	NR
UN Number	-
CAS Number	7440-56-4

Physical Data

Description	Silvery brittle metal
Melting point	936 °C
Boiling point	2700 °C
Specific gravity	5.35
Solubility in water	Immiscible or insoluble
Vapor pressure	Not applicable
Vapor density	Not applicable

Fire and Explosion Hazard

Not applicable	
Flash point	Not applicable
Explosive limits	Not applicable
Auto-ignition temperature	Not applicable
Firefighting measures	Not applicable

Health Hazard

May be harmful if ingested in quantity. May irritate or cause physical damage to eyes	
Toxicity data	No data
Carcinogenicity	No evidence of carcinogenic properties
Mutagenicity / Teratogenicity	No evidence of mutagenic or teratogenic effects
Exposure limits OES, mg / m^3	(Long-term, 8 hour TWA)

First Aid

Affected area	**Emergency First Aid**
Eyes	Irrigate eyes thoroughly with water. If discomfort persists, obtain medical attention
Lungs	Remove from exposure.
Skin	Wash off thoroughly with soap and water.
Mouth	Wash out mouth thoroughly with water. In severe cases, obtain medical attention

Reactive Hazards

Stability	Stable
Reaction with water	None
Other known hazards	Can react violently with oxidizing agents. Can ignite in contact with chlorine and bromine
Avoid contact with:	Water (no)
	Acids (yes)
	Bases (no)
	Oxidizers (yes)
	Combustibles (no)

Spillage disposal

Wear appropriate protective clothing. Contain the spillage, and clean up and transfer the spilled material to a separate container for disposal. Clean the site of the spillage thoroughly with water and detergent. Arrange for removal by a disposal company.

Storage and Handling

Special requirements	Wear protective gloves when handling the crystal

AMTIR-1

WARNING

During routine use of your HATR, 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.

Hazard Class	Non-hazardous
UN Number	-
CAS Number	77518068-3

Physical Data

Description	Dark-colored glassy metallic solid
Melting point	Glass transition temperature 362 °C; maximum application temperature 300 °C
Boiling point	Not applicable
Specific gravity	4.4
Solubility in water	Insoluble
Vapor pressure	Not applicable
Vapor density	Not applicable

Fire and Explosion Hazard

May evolve toxic fumes in a fire	
Flash point	Not applicable
Explosive limits	Not applicable
Auto-ignition temperature	Not applicable
Firefighting measures	Use an extinguisher suitable for the surrounding fire

Health Hazard

TLV (Threshold Limit Value)	A TLV has not been established for AMTIR_1. The recommended exposure limits for individual components are: **Arsenic compounds:** 0.01 mg per cubic meter of air determined as a TWA (Time-Weighted Average) exposure for up to 8 hours. (Source: OSHA 1910.1000, Subpart Z, Table Z-1, revised.) **Selenium compounds:** 0.2 mg per cubic meter of air determined as a TWA exposure of up to 8 hours. (Source: OSHA 1910.1000, Subpart Z, Table Z-1.) **Germanium compounds:** A TLV has not been adopted for germanium and its compounds (except for germanium tetrahydride)
Possible effects of exposure	No adverse health effects should occur from exposure to AMTIR-1 material. Under extreme conditions, individual components of AMTIR-1 material may cause non-specific symptoms such as nausea, vomiting, diarrhea, hot flushes and progressive anxiety. However, separation of the individual components of AMTIR-1 is not expected to occur

First Aid

Affected area

Emergency First Aid

Eyes	Flush with flowing water for 15 minutes after contact with dust or fumes
Lungs	If ill-effects develop, remove from exposure, and rest and keep warm; obtain medical attention
Skin	Flush with plenty of water after contact with dust or fumes
Mouth	Wash out mouth thoroughly with water. If AMTIR-1 is swallowed, induce vomiting and obtain medical attention

Reactive Hazards

Stability	Stable
Reaction with water	None
Other known hazards	No data

Avoid contact with:	Water (no)	
	Acids (yes)	Do not exceed a concentration of 6 N; AMTIR-1 has been tested and found to be compatible with 6 N solutions of sulphuric, nitric, hydrochloric and hydrofluoric acids
	Bases (yes)	Do not exceed a concentration of 6 N; AMTIR-1 has been tested and found to be compatible with a 6 N solution of sodium hydroxide. Note that a highly-concentrated solution of sodium hydroxide – syrupy consistency – dissolved AMTIR-1
	Oxidizers (yes)	Do not use strong oxidizers
	Combustibles (no)	

Spillage disposal

Wear appropriate protective clothing. Contain the spillage, and clean up and transfer the spilled material to a separate container for recovery or disposal. Note that the material has value and can be reclaimed. Dispose of non-reclaimable material in accordance with appropriate local, state or federal regulations. Arrange for removal by a disposal company. Clean the site of the spillage thoroughly with water and detergent.

Storage and Handling

Special requirements	Wear protective gloves when handling the crystal

Introduction

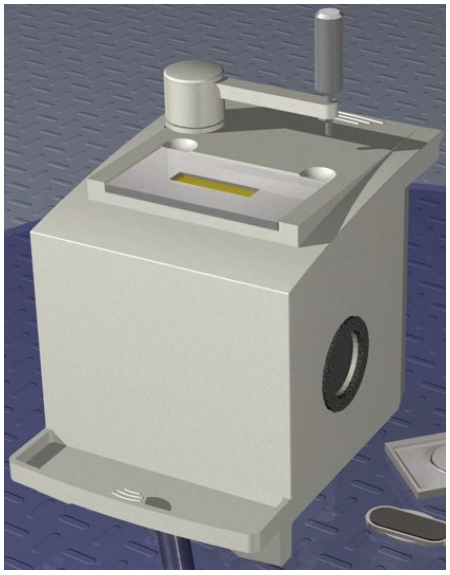

Figure 1 The Horizontal Attenuated Total Reflectance (HATR) Sampling Accessory

What is the HATR used for?

The HATR is an internal reflection accessory used with the Spectrum 100 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 Spectrum 100 or Spectrum 400. Each time the beam is reflected within the crystal, it penetrates into the sample by a few microns. Figure 2 illustrates this process.

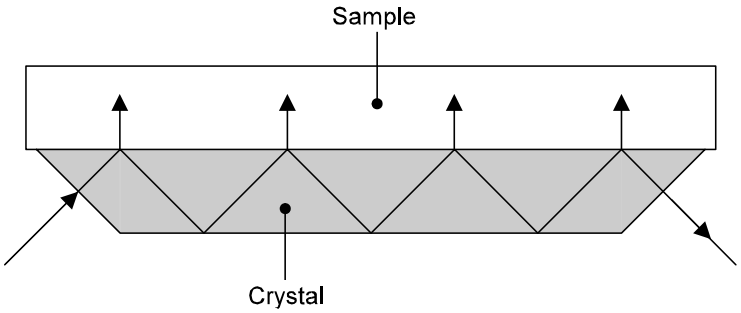

Figure 2 Principle of HATR operation

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 the Spectrum or the Spectrum Express software, and this information is stored in the status information for each spectrum collected.

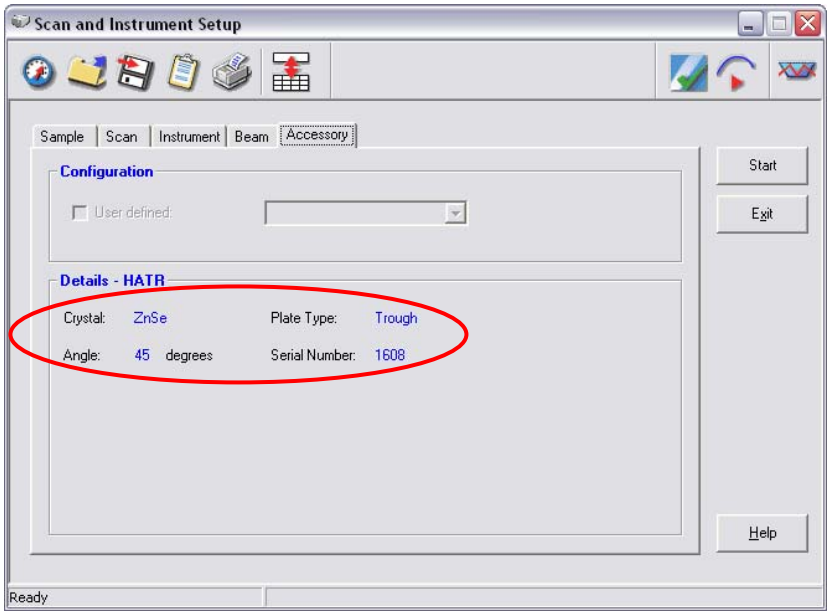

Figure 3 Top plate details displayed in Spectrum software

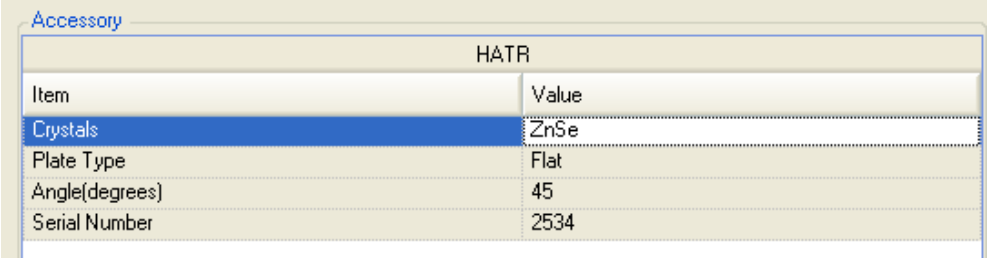

Figure 4 Top plate details displayed on the Setup Instrument Basic tab in the Spectrum Express software

Please read the safety information (starting on page 4 of this User's Guide) for the different crystal types 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 Spectrum 100 or Spectrum 400.
   Store it in a safe place for future re-use.

2. Reach in under the base of the current accessory and pull the blue release handle towards you to release the accessory.

3. Now simply slide the accessory out of the sample area (Figure 5).
   Store it in a safe place for future re-use.

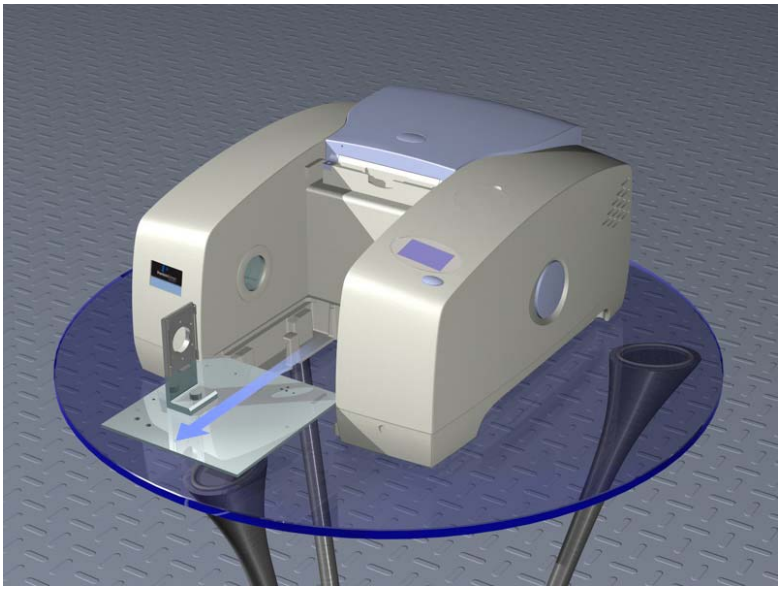

Figure 5 Removing the basic sample slide

4. 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.

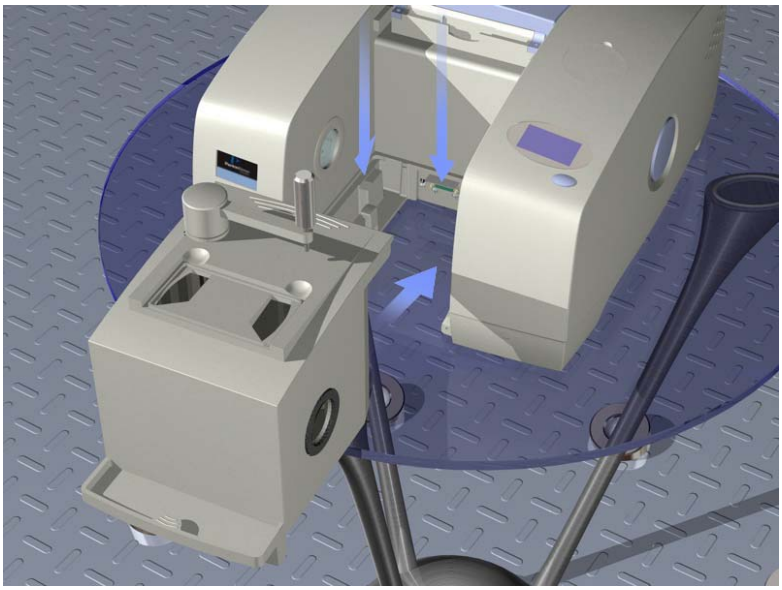

Figure 6 Installing the HATR accessory

5. Place the required top plate (trough or flat plate type) on the HATR accessory, with the arrows pointing towards the back (Figure 7).

   The HATR Sampling Accessory is now ready for use.

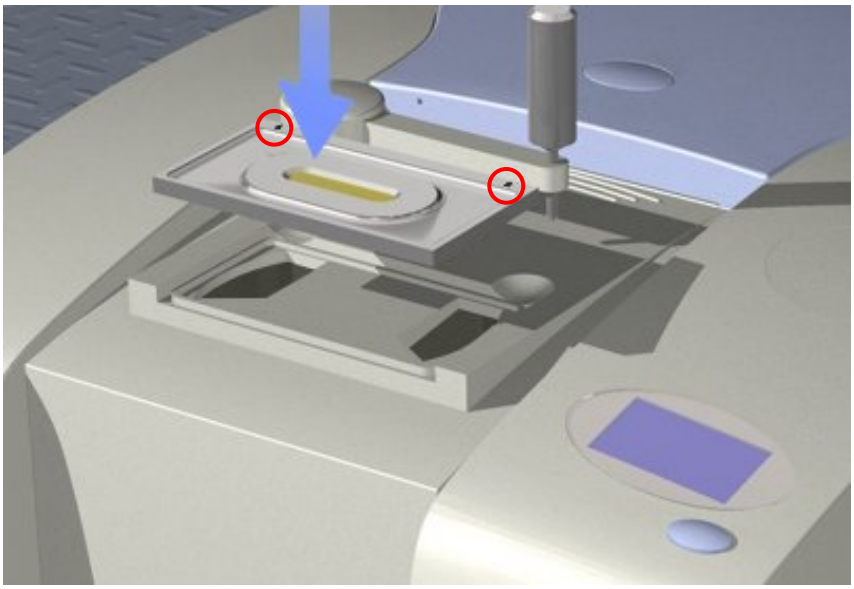

Figure 7 HATR accessory top plate with arrows circled

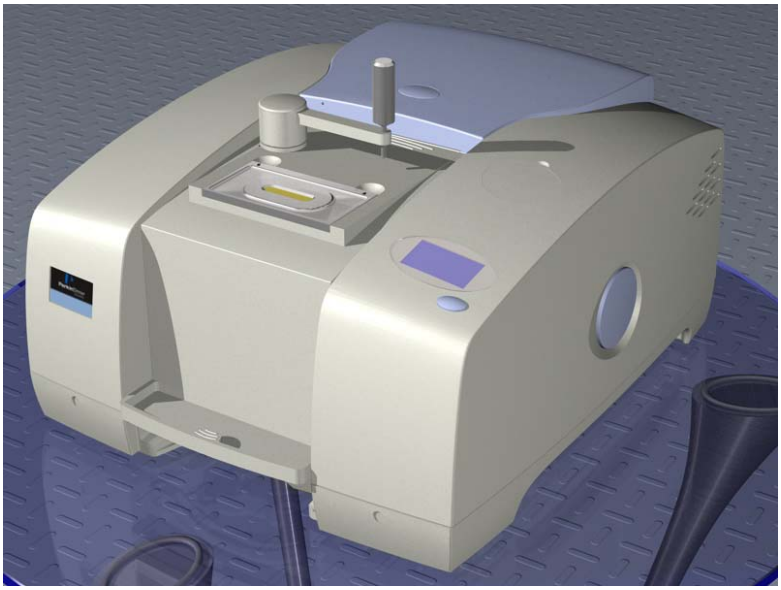

Figure 8 HATR installed in the spectrometer

The Instrument Install Wizard

If you have Spectrum software and do not have an instrument installed you will need to run the Instrument Install Wizard. The Instrument Install Wizard also enables you to test your accessory (against, for example, any shipping damage) and makes sure that any data necessary for its operation is present on your system. For a description of the Instrument Install Wizard, see your spectrometer user's guide, which shipped as a pdf on the *Spectrum Manuals CD* (L1050002).

NOTE: If you want to test an accessory after it has been installed, there is no need to re-run these accessory installation tests. Validate your instrument by running System Suitability checks instead. See the Spectrum on-screen Help for more information.

If you have Spectrum Express software and do not have an instrument installed, see the *Spectrum Express Installation Leaflet* (L1050054) that shipped with your software for details of the Instrument Install Wizard. The accessory will be detected by the Spectrum Express software when fitted in your spectrometer.

Accessory Detection by Spectrum Software

When the HATR Sampling Accessory is installed, the software detects the presence of the accessory and updates to show that you now have the HATR Sampling Accessory in position.

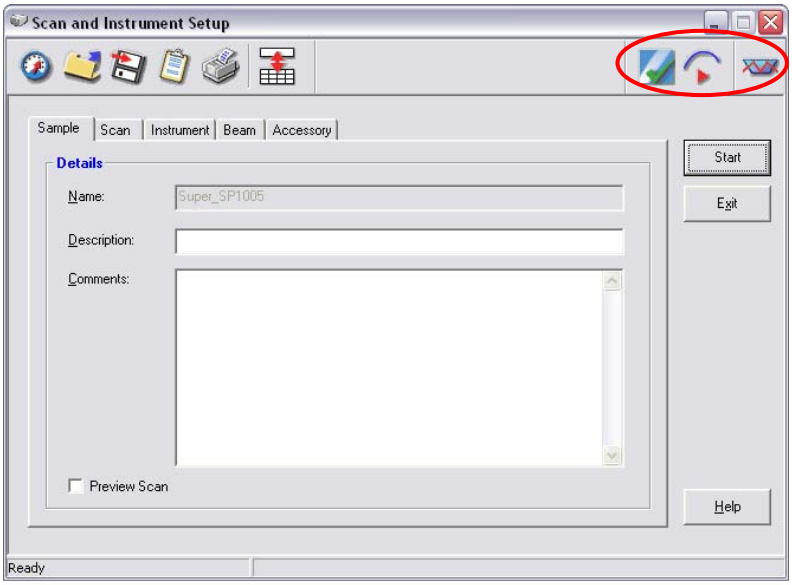

Figure 9 Scan and Instrument Setup dialog with HATR icons circled

Accessory Detection by Spectrum Express Software

When the HATR Sampling Accessory is installed, the software detects the presence of the accessory and updates the Setup Instrument BeamPath tab to show that you now have the HATR Sampling Accessory in position.

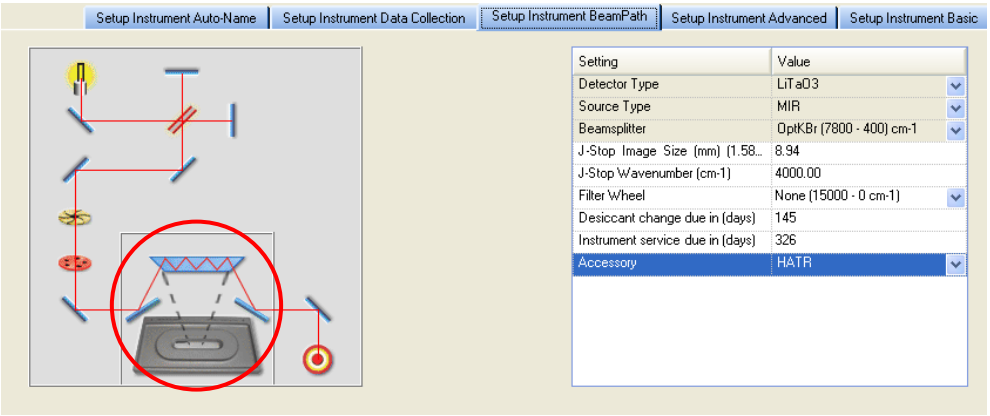

Figure 10 Setup Instrument BeamPath tab with HATR icon circled

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 Check in Spectrum software

NOTE: For information about setting up System Suitability checks see the Spectrum on-screen Help. The following description assumes that the Contamination System Suitability check is already set up and enabled.

1. On the Scan and Instrument Setup dialog, click the Contamination Check tool  (Figure 11).

   This check enables you to ensure that the crystal is clean before you place your sample on the crystal or perform a background spectrum. The software will report whether the test has been passed or failed.

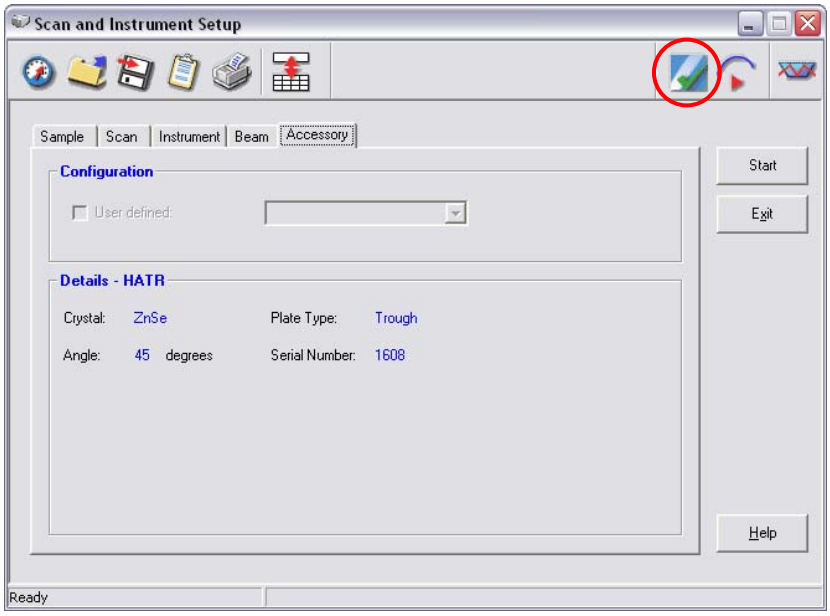

Figure 11 Scan and Instrument Setup dialog – Contamination check tool

2. If the test has failed, clean the crystal as described on page 22, and then select **Repeat** to re-run the check.

   If you click on **Report**, a report on the contamination check is displayed.

Contamination Ready Check in Spectrum Express software

NOTE: For information about setting up Ready Checks see the Spectrum Express 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.

Applying Pressure

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.*

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

1. If pressure is going to be applied, place the appropriate pressure plate over the sample.

2. Swing the pressure arm round from the docked position until it is above the center of the pressure plate as shown in Figure 12.

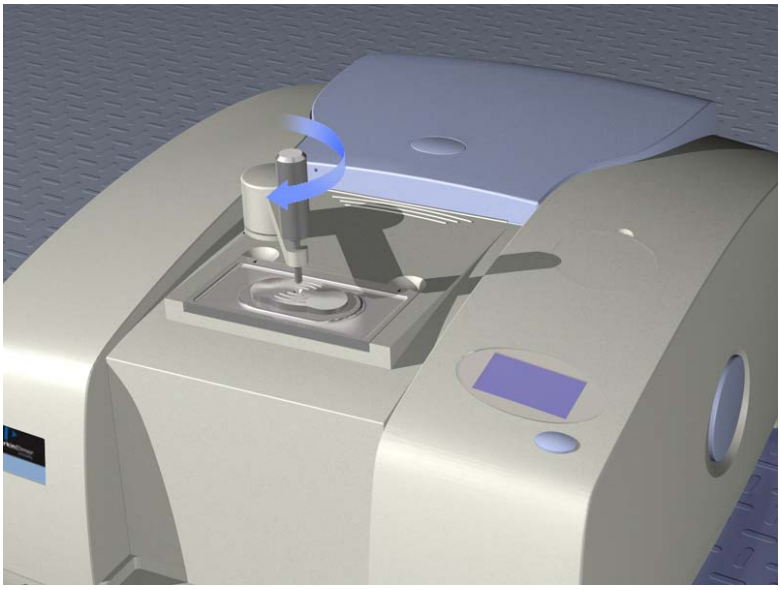

Figure 12 Pressure arm in operating position

3. 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.*

4. Switch to **Monitor** by clicking  on the Scan and Instrument Setup dialog (Spectrum software).

   The Display tab is displayed.

   OR

   Switch to **Monitor** by clicking  on the Measurement toolbar (Spectrum Express software).

   The Live tab is displayed.

5. Referring to the readout in the **Force Gauge**, adjust the force applied to an appropriate level.

   The **Force Gauge** displays 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.

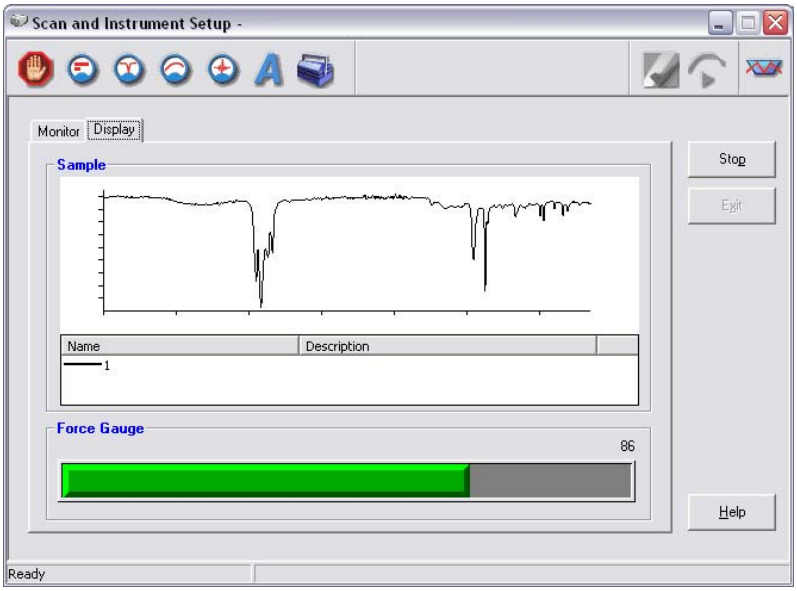

Figure 13 Force Gauge indication in Spectrum software

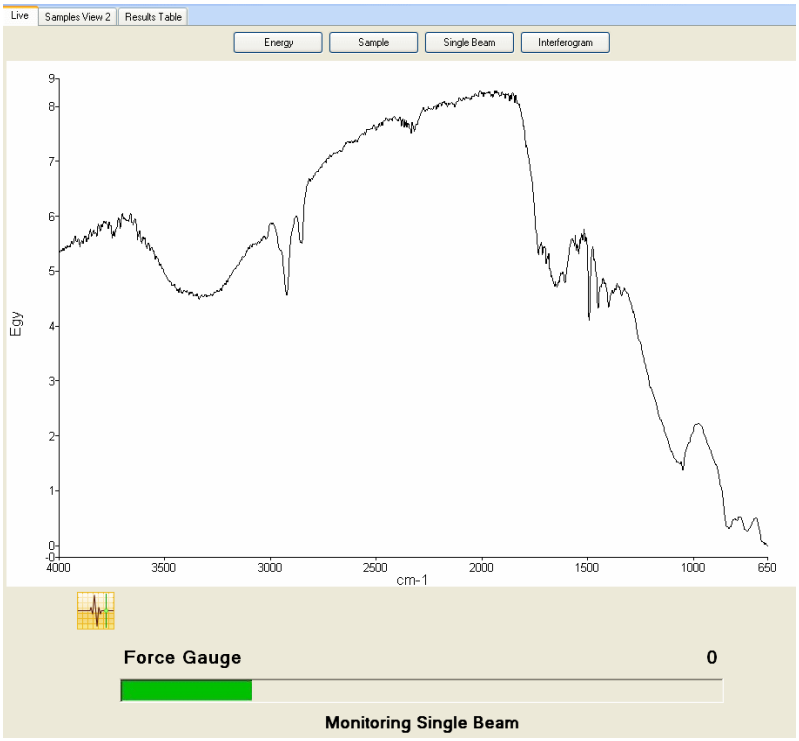

Figure 14 Force Gauge indication in Spectrum Express software

You are now ready to collect data.

Using the Accessory with Spectrum Software

1. Select **Scan** on the Instrument menu.
   The Scan and Instrument Setup dialog is displayed.

2. Enter sample details, scan parameters and instrument settings.
   The information shown will depend upon your user level.

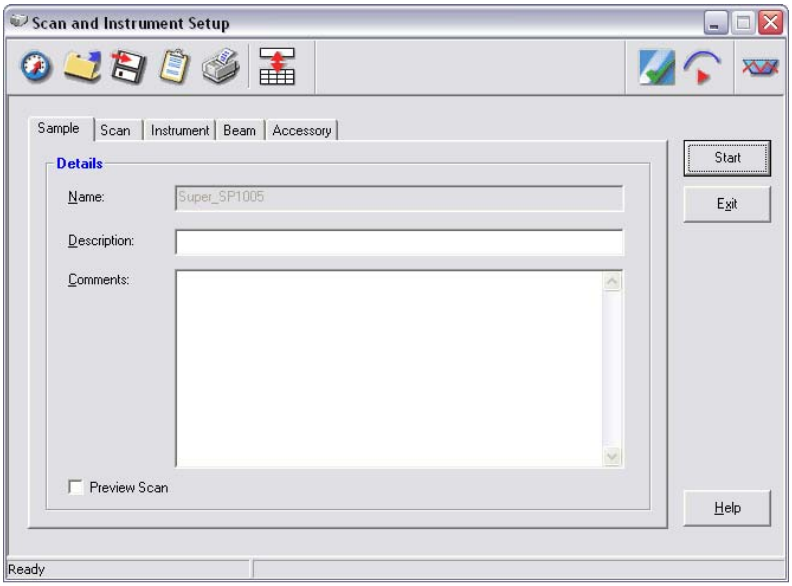

Figure 15 Scan and Instrument Setup dialog – Sample tab

3. Select the **Accessory** tab.
   Details of the current top plate, crystal and angle are shown, and a choice of pre-determined configurations.

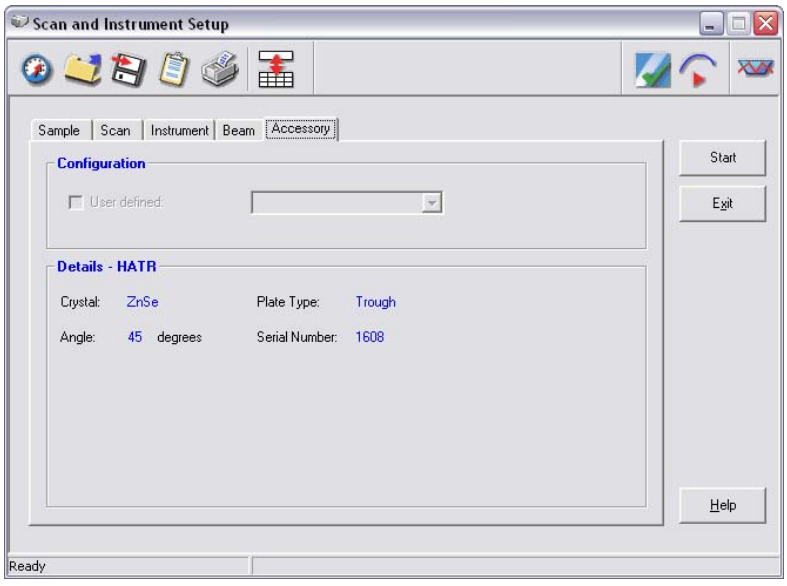

Figure 16 Scan and Instrument Setup dialog – Accessory tab

4. Click **Start** to begin collecting data.

   The Display tab is displayed as the scans are collected and then the spectrum appears in the graph window in the main application.

Although Spectrum automatically alerts you when you need a new background, and can be configured to request a new background at set intervals (using Data Collection Setup), if you want to collect a background, use the **Scan type** option on the Scan tab or click  .

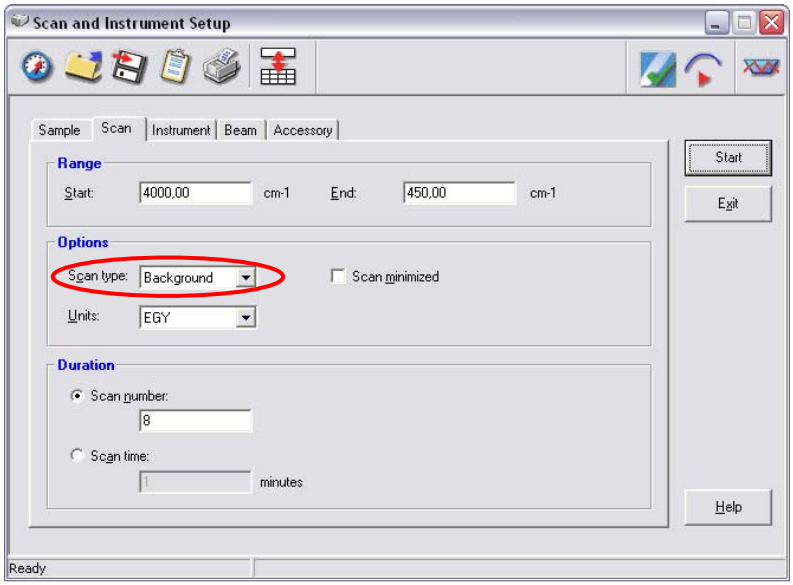

Figure 14 Scan tab with Background selection circled

Using the Accessory with Spectrum Express Software

1. Enter the required scan and instrument parameters in the Instrument Settings toolbar.

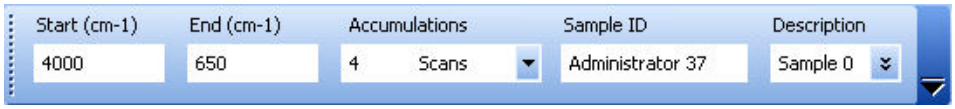

Figure 17 The Instrument Settings toolbar

If you want to set instrument parameters that are not displayed in the Scan toolbars, use the Setup Instrument tabs in the Dialog Pane at the bottom of the workspace.

To open the Dialog Pane, select **Instrument** from the Setup menu.

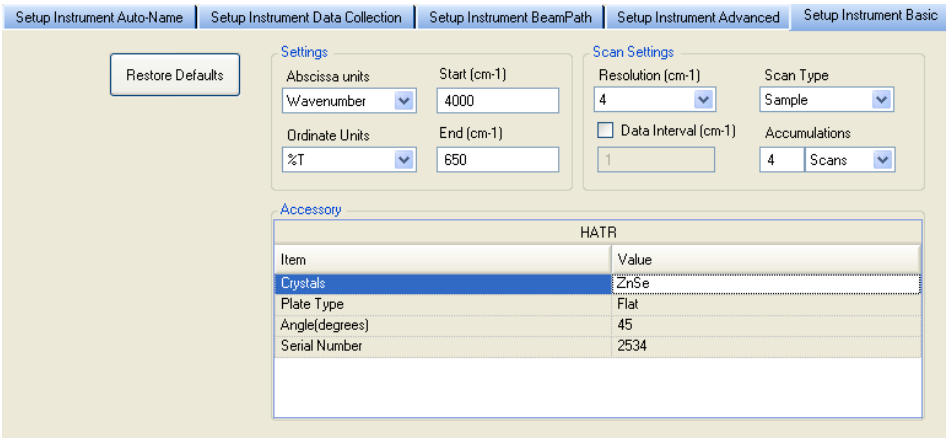

Figure 18 Setup Instrument Basic tab showing the HATR top plate details

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

2. If you need to collect a background spectrum, ensure that the top plate is clean and then click  on the Measurement toolbar to collect a background spectrum.

   Spectrum Express 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).

   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, and then click  on the Measurement toolbar to collect a spectrum.

   The **History** settings of your sample will contain information about the top plate.

NOTE: The Spectrum Express on-screen Help describes how to format, process and report your results. To view the Help, select **Contents** from the Help menu.