

UNIVERSAL ATR 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), KRS5 (thallium bromo-iodide) or Ge (germanium) 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 Universal ATR 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.

KRS5 (thallium bromo-iodide) crystals



WARNING

During routine use of your Universal ATR Sampling Accessory, the KRS5 crystal presents no hazard, but:

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

***DO NOT** allow the crystal to come into contact with oxidizing agents, acids or strong bases.*

The crystal is extremely toxic by ingestion, with a cumulative effect.

Cleaning KRS5 crystals

Avoid contact of the crystal with oxidizers, acids, and strong bases. KRS5 can be cleaned in pure dry acetone using a soft, lint-free cloth and drying 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. A final rinse in distilled water and drying in a current of warm air is recommended.

Ge (germanium) crystals



WARNING

During routine use of your Universal ATR 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.

Introduction

The Universal Attenuated Total Reflectance Accessory (UATR) is an internal reflection accessory, used with a Frontier IR System, Spectrum 400 Series or Spectrum 100 Series spectrometer, 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.



Figure 1 The Universal ATR Sampling Accessory with Pressure Arm

How the UATR 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 in the crystal, and back towards the detector which is housed in the instrument. When the beam is reflected within the crystal, it penetrates into the sample by a few microns. Figure 2 illustrates this process.

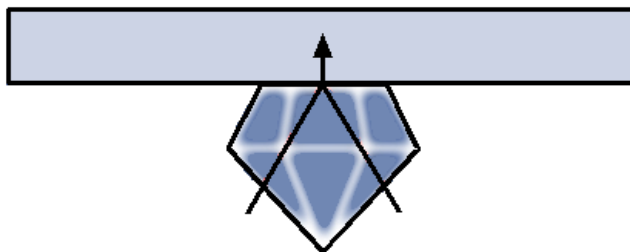


Figure 2 Principle of UATR operation

Choosing the top plate

The UATR can be used to analyze powders 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. A flat plate can also be used to analyze pastes and gels, by spreading them over the surface of the crystal.

Optional sample positioning plates are available, to ensure that the sample is positioned correctly over the crystal. Force may be applied to ensure good contact.

The standard UATR top plates have composite Zinc Selenide (ZnSe) and diamond crystals, Germanium, or composite KRS5 and diamond crystals. Consult your PerkinElmer representative for further information about the available top plates.

Choosing the correct crystal

The crystal most commonly-used is a composite of Zinc Selenide (ZnSe) and diamond. The diamond is cost-effective, is not easily scratched, is resistant to strong acids and bases, and can withstand high pressures.

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 representative for advice.

When you change top plates, the crystal details are displayed in the software and this information is stored in the status information for each spectrum collected.

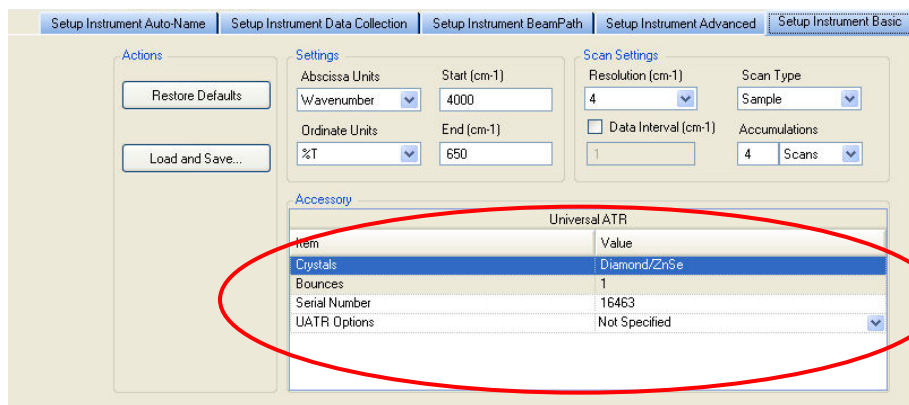


Figure 3 Top plate details displayed in Spectrum software

Choosing the correct crystal configuration

Standard UATR top plates are available in three configurations:

- Single bounce – A flat plate where the infrared beam contacts the sample at only one point. The effective pathlength is 2 μm .
- Triple bounce – A flat plate where the infrared beam contacts the sample at three points. The effective pathlength is 6 μm .
- Nine bounce – A trough plate for liquids and powders. The effective pathlength is 18 μm .

NOTE: The effective pathlength changes with wavelength.

Installing the UATR

Fitting the accessory

The UATR fits into the sample compartment of a Frontier IR System, Spectrum 400 Series or Spectrum 100 Series spectrometer, after first removing any other sampling accessory that is currently fitted.

To remove the current accessory and install the UATR:

1. Raise the sample cover to the vertical position, press the release clip and lift the cover upwards, clear of the spectrometer.
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

4. Rest the back of the UATR 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 accessory mates properly with the instrument connector.



Figure 5 Installing the accessory

5. Place the required top plate on the UATR.
If required, you can fix the top plate in position using two small screws located in its front and right hand edges. Refer to *Appendix 2: Top-plate Shipping and Anti-Theft Screws* on page 18.




Figure 6 UATR installed in a Frontier IR System

The UATR is now ready for use.

NOTE: When the UATR 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 UATR icon  is added to the toolbar.

The Setup Instrument Basic (Figure 7) and Setup Instrument BeamPath (Figure 8) tabs are updated to show that the UATR Accessory is in position.

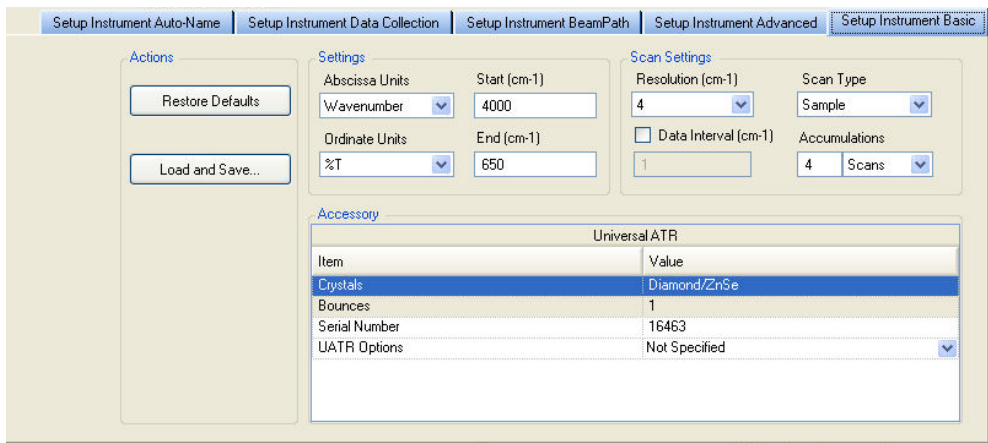


Figure 7 Setup Instrument Basic tab with UATR

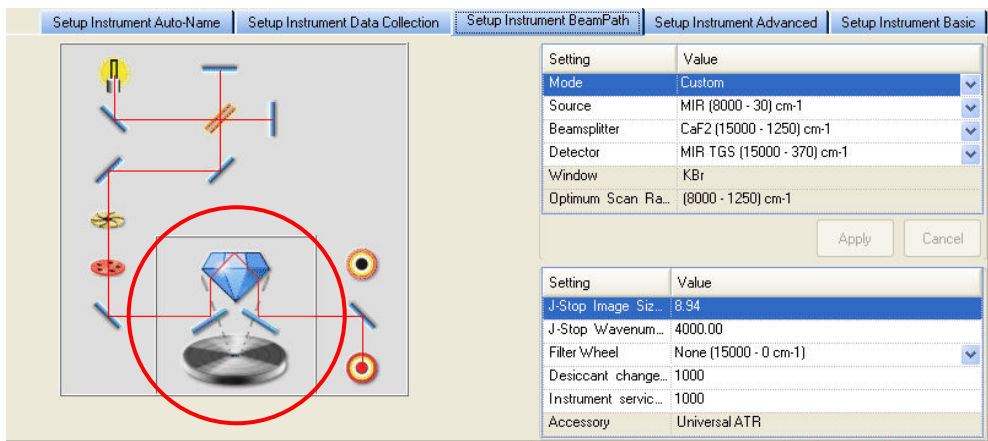


Figure 8 Setup Instrument BeamPath tab with UATR icon circled

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 negative bands in the spectra may be observed.

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. Take care not to spill solvent inside the accessory.

Do not leave the top of the accessory uncovered for long periods of time.

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 crystal, 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

NOTE: The Liquid UATR supplied with the Biodiesel IR FAME Analyzer does not have a pressure arm.

CAUTION

The pressure arm should not be used in conjunction with a nine bounce UATR top plate, as this will result in damage to the crystal.

Good contact between the sample and the surface of the crystal is important to prevent loss of beam penetration. To aid this good contact, the UATR optional pressure arm can be used, in conjunction with a "shoe" designed to apply a controlled force to the sample and make better contact with the crystal.

The following shoes are available:

- 1.5 mm diameter flat end (part number L1202049).
- 6 mm diameter flat end (part number L1202050).
- Cone shoe (part number L1201980), for use with powders.

Using the Accessory with Spectrum Software

The following pages outline the procedure for obtaining a sample spectrum using a Frontier instrument fitted with a UATR 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.

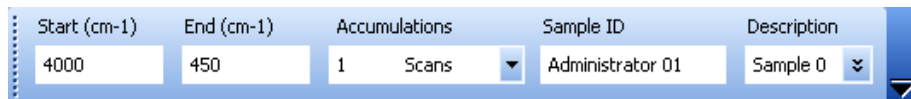



Figure 9 The Instrument Settings toolbar

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 7 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 toolbar, to collect a background spectrum.

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. Push the pressure arm back, until it is clear of the top plate.
4. Slide the appropriate shoe over the end of the pin until it clips into place.

CAUTION

The pressure arm should not be used without a shoe, as this may result in damage to the crystal.

5. Place your sample in the center of the UATR top plate, over the crystal.
6. Swing the pressure arm across, so that the end is positioned directly above the sample and the crystal.

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.

8. 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 UATR 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.

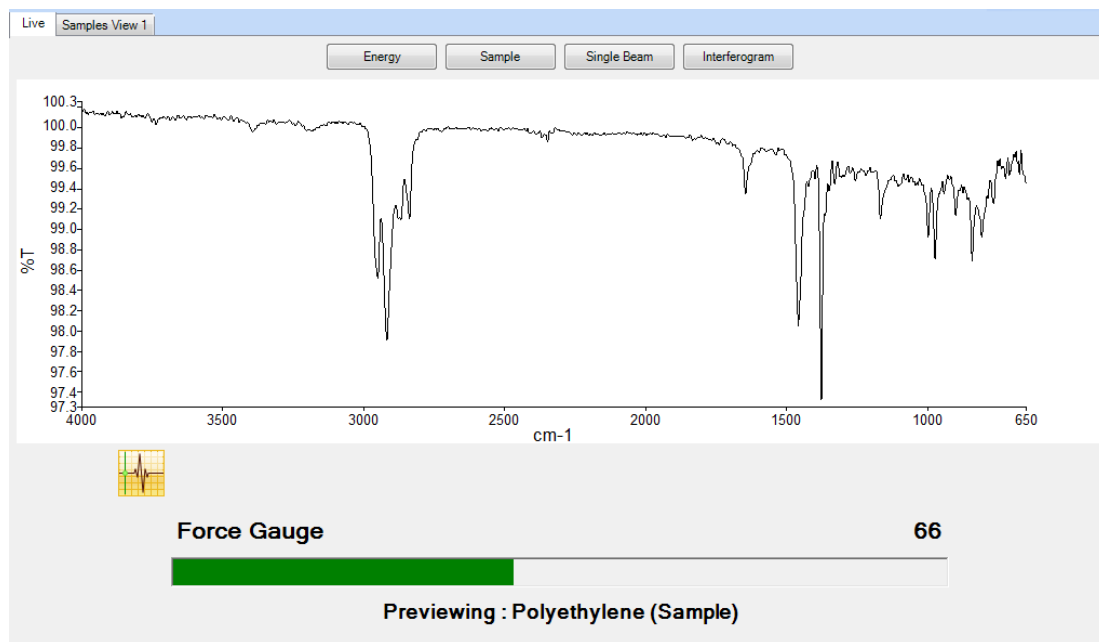



Figure 10 Force Gauge indication in Spectrum software

You are now ready to collect data.

NOTE: An over-pressure device is fitted to the pressure arm that significantly reduces the risk of crystal breakage. This clutch-slip mechanism should never operate in day-to-day use, as the pressure required for this to happen is greater than should be necessary to achieve optimum transmission of between 40 and 80%.

The Force Gauge indicator bar, if displayed, will turn red before the over-pressure device comes into effect.

9. Click  to obtain the sample spectrum.
The **History** settings of your sample will contain information about the UATR 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.

Appendix 1: The Over-Pressure Device and Pressure Pin

NOTE: The Liquid UATR supplied with the Biodiesel IR FAME Analyzer does not have a pressure arm.

In the unlikely event that the pressure pin is completely retracted, the pressure pin cannot be screwed down because the over-pressure device slips. To release the pressure pin and reset the over-pressure device:

1. Lever the cap off the over-pressure device knob, exposing three screws and a small protruding pin.

CAUTION

DO NOT remove or adjust the screws, which are factory set.



Figure 11 Pressure pin

2. Press in the pin, turning the knob slowly clockwise.
After about 10 degrees rotation the pin drops down flush, locking the over-pressure device.
3. Continue to carefully turn the knob clockwise, sufficiently to release the pressure pin.
4. Reset the over-pressure device by turning the knob slightly anti-clockwise.
The locking pin pops up and protrudes as before.
5. Replace the cap.
The pressure pin and over-pressure device function normally.

Appendix 2: Top-plate Shipping and Anti-Theft Screws

The top plate is lightly held by a pair of captive screws in its rim, approximately 120 degrees apart.

These screws keep the top plate in place when the instrument or accessory is moved or shipped, and help avoid the top plate from being casually lifted from the instrument or stolen.

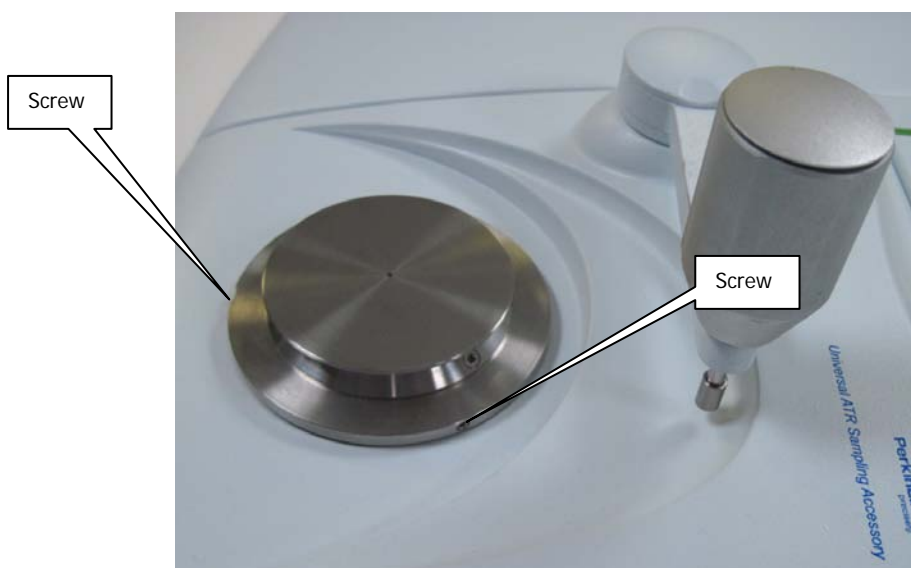


Figure 12 Top-plate movement and anti-theft screws

These screws have no effect on the kinematics of the top plate, or on sample contact.

- To hold the top plate in place, it is sufficient for the screw heads to be approximately flush with the rim. Do not fully tighten the screws; use the hexagonal key supplied, with care.
- If your work requires you to change the top plate frequently, you may leave these shipping screws loose.