

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 Spectrum Two 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.

Introduction

The Universal Attenuated Total Reflectance Accessory (UATR) is an internal reflection accessory, used with a Spectrum Two Series spectrometer, for simplifying the analysis of solids, powders, pastes, gels and liquids. The technique is non-destructive.

As the beam does not penetrate deeply 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

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.

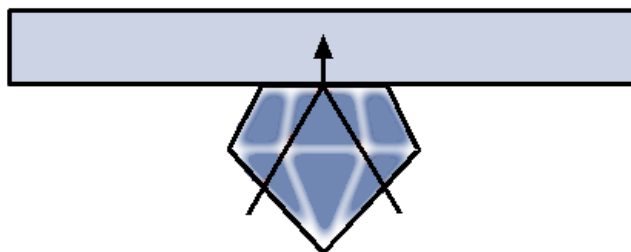


Figure 2 Principle of UATR operation

The UATR can be used to analyze homogenous solid samples, solid surfaces, or coatings on solid samples. Force may be applied to ensure good contact.

The Spectrum Two UATR uses a diamond crystal. The diamond crystal is hard, is not easily scratched, is resistant to strong acids and bases, and can withstand high pressures. The diamond UATR has an effective scanning range that matches the full range of the instrument, although sensitivity is somewhat reduced in the approximate range 1900–2700 cm^{-1} .

Installing the UATR

Fitting the accessory

The UATR fits into the sample compartment of the spectrometer, replacing the sample plate.

NOTE: Before you begin you should, if necessary, loosen the pressure arm and release the transit block that secures the UATR pressure arm during transport.

You may also need to remove the covers on the left and right sides of the accessory, and the top-plate dust cover.

Store all these in a safe place for future use, if required.

To remove the sample plate and install the UATR:

1. Raise the sample cover of the spectrometer to the vertical position and lift it upwards, clear of the instrument.
Store it in a safe place for future re-use.
2. Slide the sample plate out of the sample area (Figure 3).
Store it in a safe place for future re-use.

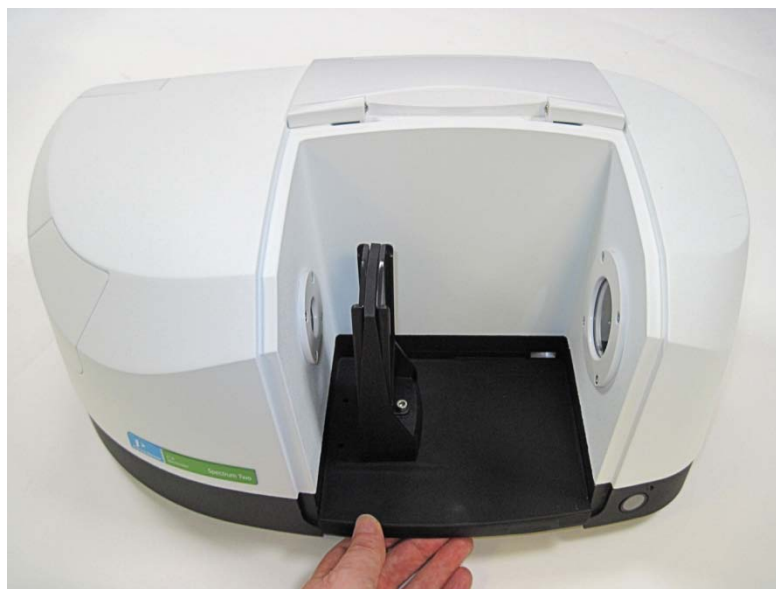


Figure 3 Removing the basic sample plate

3. Pivot the UATR on the ledge at the front of the sample area (Figure 4a), then tip it backwards (Figure 4b).

NOTE: If the accessory is pivoted too far forward on the sample area ledge it will not move correctly into position.



(a)



(b)

Figure 4 Installing the accessory

4. Push the UATR home firmly, ensuring that the top of the accessory mates properly with the lid of the instrument.

The UATR is now ready for use.



Figure 5 UATR installed in a Spectrum Two

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, connect to the white plastic purge connector on the underside of the instrument, at the right.

Accessory detection by Spectrum software

The Spectrum software detects the presence of the accessory automatically, and the Setup Instrument Basic (Figure 6) and Setup Instrument BeamPath (Figure 7) tabs are updated to show details of the UATR accessory.

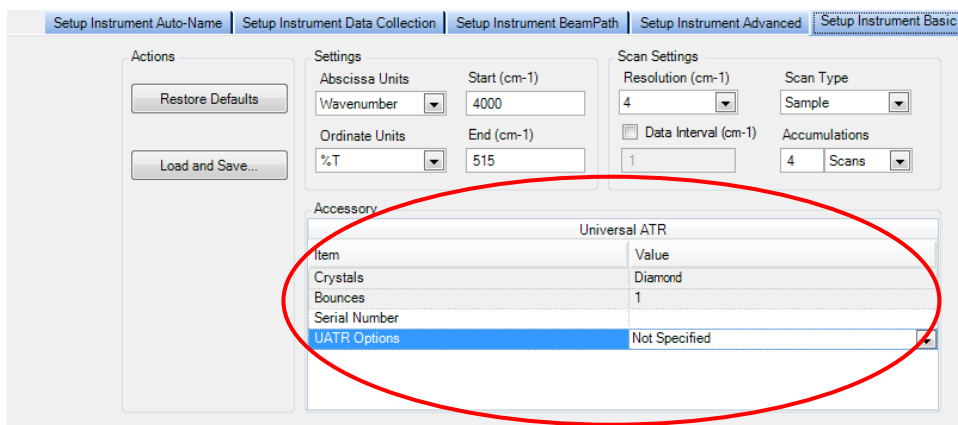


Figure 6 Setup Instrument Basic tab with UATR details circled

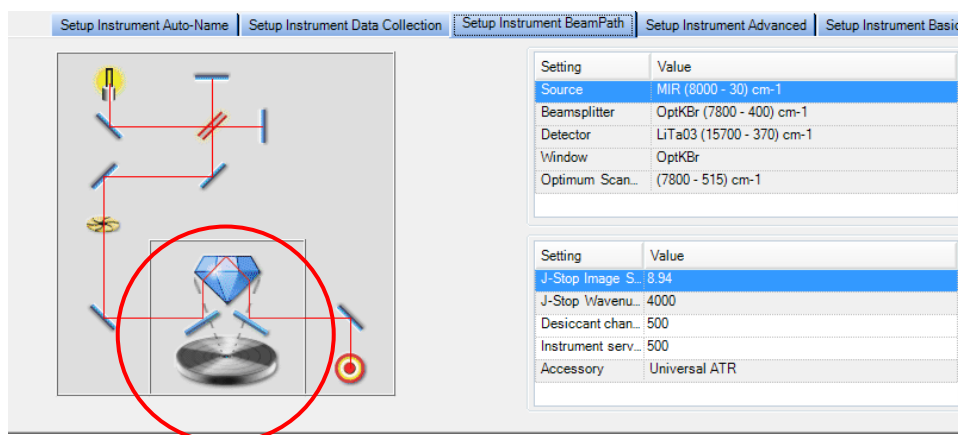


Figure 7 Setup Instrument BeamPath tab with UATR icon circled

Taking a Reference Background spectrum

Once you have installed your UATR, and before you use it for the first time, you should consider taking a background spectrum which will act as a reference background for use with those Ready Checks that need one for comparison purposes; including the *Contamination Check* described on page 10.

NOTE: For information about taking a background spectrum, see the Spectrum on-screen Help.

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, the crystal should be cleaned.

The surface of the crystal can be cleaned with a cotton-bud moistened with pure water or a pure organic solvent. The liquid should not be allowed to evaporate, but should be removed with a clean, dry cotton-bud.

Take care not to scratch the surface of the crystal. Ensure the crystal is completely dry before re-use.

A PVC "apron" (L1601856) is provided, that protects the top and front of the accessory when cleaning with solvent.

Performing a 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 Contamination 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 any sample from the UATR and cleaned the crystal, and then click **Scan**.
A new background spectrum is collected, compared to the reference background spectrum (a spectrum taken when the crystal is known to be totally clean), and the result of the test displayed.
3. If required, click the link that enables you to see a print preview of the Instrument Ready Checks Report.

Performing Ready Checks that use Polystyrene

The Ready Checks available in the Spectrum software can be used to demonstrate that various aspects of the performance of your complete system, including your UATR, are fit-for-purpose.

Some of these Ready Checks, including Abscissa Check and Ordinate Check, use a polystyrene sample as reference.

A polystyrene sample that can be used on the top-plate of your UATR is provided for use when carrying out these Ready Checks.

NOTE: For information on Ready Checks, see the Spectrum on-screen Help.
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Handling Samples

Care should be taken with all samples to ensure that spillages do not occur and that the sample does not come into contact with any part of the accessory other than the top-plate.

A PVC “apron” (L1601856) is provided, that protects the top and front of the accessory.

Solid samples

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 pressure arm can be used, in conjunction with a “shoe”, to apply a controlled force to the sample and make better contact with the crystal.

The following shoes are available:

- Large (L1601743) – for low sample pressures
- Medium (L1601742) – for higher sample pressures

Liquid samples

Viscous liquids can be placed directly onto the surface of the UATR crystal.

Samples of volatile and non-viscous liquids should be placed in a “volatiles cup” (L1601746).

Obtaining a Sample Spectrum with Spectrum Software

The following pages outline the procedure for obtaining a sample spectra using a Spectrum Two instrument fitted with a UATR and running Spectrum software.

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

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

Obtaining a spectrum of a solid sample, using the pressure arm

1. To view or change any of the instrument parameters, select **Instrument** from the Setup menu.

The Setup Instrument pages are displayed in the Dialog Pane at the bottom of the workspace. Figure 6 on page 9 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 shaft until it clips into place.
5. Place your sample in the center of the top-plate, over the crystal.
6. Swing the pressure arm across, so that the end is positioned directly above the sample and the crystal.



Figure 8 Moving the pressure arm into its operating position (large shoe)

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 arm 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 too much force is being applied; the pressure arm and/or the crystal is in danger of being damaged.

NOTE: If you want to compare various samples, or use quantitative techniques, ensure you use the same amount of force each time. You should also ensure that you have the same type of shoe fitted.

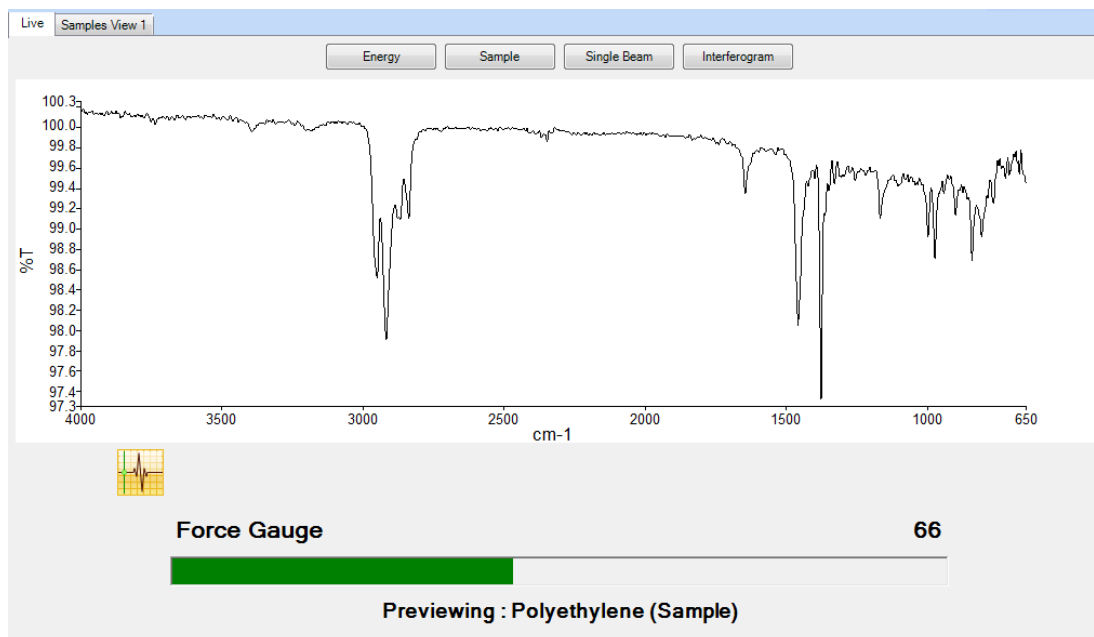


Figure 9 Force Gauge indication in Spectrum software

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.

Obtaining a spectrum of a liquid sample

1. To view or change any of the instrument parameters, select **Instrument** from the Setup menu.

The Setup Instrument pages are displayed in the Dialog Pane at the bottom of the workspace. Figure 6 on page 9 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. If your sample is volatile or non-viscous, continue the procedure below; otherwise, place a drop of your sample on top of the crystal and go straight to step 11.
4. Push the pressure arm back, until it is clear of the top-plate.
5. Slide the medium size shoe over the end of the shaft until it clips into place.
6. Place an empty volatiles cup in the center of the UATR top-plate, over the crystal.
7. Swing the pressure arm across, so that the shoe is positioned directly above the cup.



Figure 10 Using the pressure arm with a medium shoe to position the volatiles cup

8. Screw down the pressure arm gently, until the shoe just meets the cup.
This ensures that the center of the cup is positioned exactly over the crystal.
9. Loosen the pressure arm, so that it is clear of the cup, and then swing it back, until it is clear of the top-plate.

10. Place a drop of your liquid sample in the cup.

If your sample is particularly volatile you can bring the pressure arm back into position and screw it down gently, so that the shoe acts as a cover.



Figure 11 Using the pressure arm and medium shoe to cover the volatiles cup

NOTE: The volatiles cup is not intended to be used as a pressure shoe for solid samples.

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

The Live tab is displayed, showing a preview of your sample spectrum.

You are now ready to collect data.

12. 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.

CAUTION

Clean the volatiles cup immediately after use. Do not place a used volatiles cup back a storage peg, on the top of the accessory, until it has been cleaned.

Appendix: Replacing the Top-Plate

In the event that the top-plate becomes damaged, it can be replaced with one of the same type. Contact your PerkinElmer representative for details of part numbers.

1. Remove the accessory from your Spectrum Two instrument.
2. Position the transit block supplied with your UATR on top of the crystal.
3. Swing the pressure arm across and screw it down, securing the transit block firmly in place.

This provides an even pressure over the surface of the top-plate.

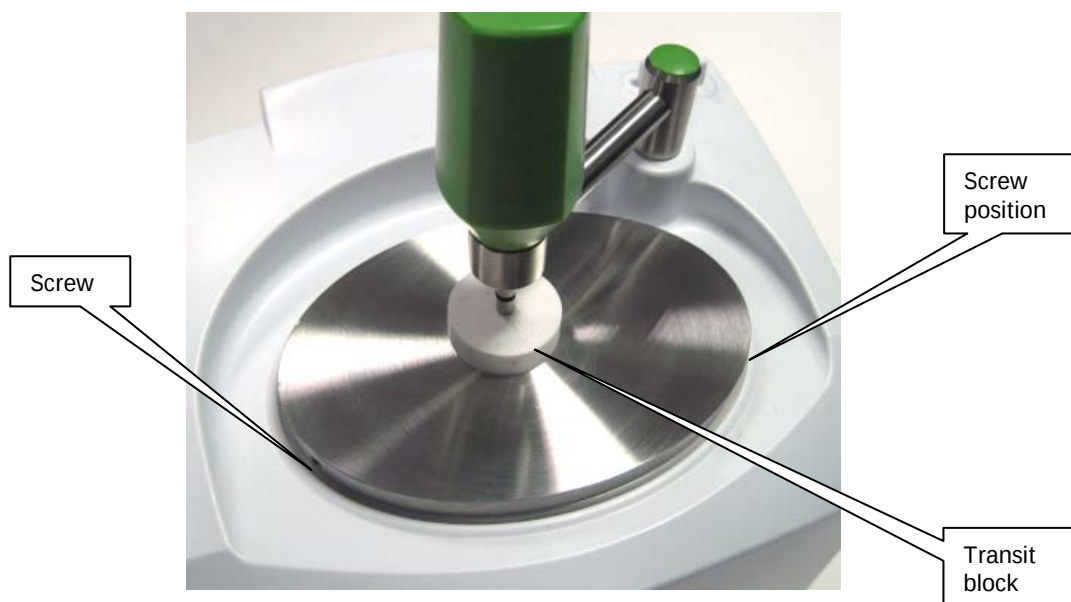


Figure 12 Removing the top-plate

4. Use a ball-ended hexagonal wrench to remove the two fixing screws from the rim of the top-plate.
5. Loosen and swing back the pressure arm.
6. Remove the transit block and then lift the top-plate off the body of the accessory.

7. Insert the two fixing screws into the rim of the new top-plate, so that they are just captured.



Figure 13 Fixing screws captured in top-plate

8. Place the new top-plate on the body of the accessory.
Ensure that it is oriented correctly, so that the indent on the underside accommodates the gold mirror that protrudes from the body of the accessory.

CAUTION

If you put the top-plate on in the wrong orientation you may cause damage to the gold mirror

The top-plate should fit easily into position on top of the two support pillars and on the O-ring inside the rim. Ensure that the O-ring is seated correctly and is not twisted or deformed.



Figure 14 UATR with top-plate removed

9. Secure the transit block on the top-plate, with the pressure arm, and then tighten the two fixing screws so that they are flush with the rim of the top-plate.
10. Remove the transit block and store it for future use.