

Agilent Cary 630 FTIR Spectrometer

User's Guide



Notices

Manual Part Number

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Agilent Technologies, Australia (M) Pty
Ltd
679 Springvale Road
Mulgrave, Victoria, 3170
Australia
www.agilent.com

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Safety Notices

CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A **WARNING** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a **WARNING** notice until the indicated conditions are fully understood and met.

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The Agilent Cary 630 FTIR is a small, mid-infrared spectrometer designed to provide the capabilities of much larger, traditional FTIR spectrometers, but without the complexity, maintenance requirements or cost. The main optical unit dimensions are only 16 x 22 x 13 centimeters (6.3 x 8.7 x 5.1 in) and it weighs only 2.9 kilograms (6.3 lb). The Cary 630 FTIR provides versatility for use in a traditional analytical chemistry laboratory. The system is intended for indoor use and is not designed for water-resistance or other extreme outdoor conditions.

WARNING**Fire Hazard**

The Cary 630 FTIR systems are NOT intrinsically safe. Use the system only in atmospheres that have been tested for flammable materials. If the equipment is used in a manner not specified in this guide, the protection provided by the equipment may be impaired.

The Cary 630 FTIR uses technology known as Fourier transform infrared (FTIR) spectroscopy, which is the current state-of-the-art technique for molecular compound identification and quantification. FTIR uses an infrared light source to pass through the sample and onto a detector, which precisely measures the amount of light absorbed by the sample. This absorbance creates a unique spectral fingerprint that is used to identify the molecular structure of the sample and determine the exact quantity of a particular compound in a mixture. At the heart of the Cary 630 FTIR spectrometer is a unique Michelson interferometer. This proprietary design is the key to making FTIR technology compact, lightweight and rugged.

The Cary 630 FTIR is available in two configurations depending on the frequency range necessary for your application. Please see the Cary 630 FTIR specifications for details.

Introduction

In addition, a number of different sampling accessories are available to suit various sample types and sensitivities. The accessories accommodate the analysis of a range of liquids, powders, gases, pastes and gels. They include:

- **Diamond ATR:** For the chemical analysis of liquids, powders, pastes and gels. The attenuated total reflectance (ATR) system is available as a single-reflection (most common) ATR.
- **ZnSe ATR:** For the chemical analysis of liquids, gels and soft solids within a pH range of 5-9. The attenuated total reflectance (ATR) system is available as a single-reflection ATR (most common) and a multi-bounce ATR (Liquids only)
- **Transmission:** A classic infrared sampling interface, allowing measurement of solids, liquids and gases. A 50 mm slide-mount gas cell is also available from Agilent.
- **TumblIR:** Single pathlength transmission for rapid chemical analysis of liquids under ambient conditions.
- **DialPath:** For rapid chemical analysis of liquids under ambient conditions, with three set pathlengths that allow you to choose the sensitivity necessary for your analysis.
- **Diffuse Reflectance:** For quantitative and qualitative measurement of powdered and solid samples. Provides more sensitive quantitative analysis than ATR, while still being easy to use.
- **Germanium ATR:** For measuring samples that are too highly absorbing for diamond ATRs or for samples with high amounts of carbon black.
- **10 degree Specular Reflectance:** For measuring specular reflectance of solid samples at near normal.
- **45 degree Specular Reflectance:** For measuring specular reflectance of solid samples at angle. This accessory is placed into the transmission compartment.

The Cary 630 FTIR is computer-controlled using Agilent MicroLab PC software. MicroLab PC is intuitive, simple to use and requires no specialized technical training. With a click of the mouse, the system provides valuable information about the identity and amount of chemical substances present in a material.

Information symbols

The following is a list of symbols that appear in conjunction with warnings on the spectrometer. The hazard they describe is also shown.

A triangular symbol indicates a warning. The meanings of the symbols that may appear alongside warnings in the documentation or on the instrument itself are as follows:



The following symbol may be used on warning labels attached to the instrument. When you see this symbol, refer to the relevant operation or service manual for the correct procedure referred to by that warning label.



The following symbols appear on the instrument for your information.

| | |
|-------|-----------------|
| I | Mains power on |
| 0 | Mains power off |
| — — — | Direct current |

Introduction

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Getting Started

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Unpacking

To unpack your spectrometer system:

- 1 After receiving the Agilent Cary 630 FTIR delivery, do not immediately open the shipping container. Instead, place the shipping case in a room-temperature environment and allow several hours for the contents of the container to reach the room ambient temperature. This is to avoid unnecessary condensation on the components prior to the initial setup and installation process.

Your Cary 630 FTIR has been packed to ensure that it arrives at your facility ready to go to work. Custom foam-in-place inserts protect the instrument from damage during travel. Whenever your instrument is shipped via common carrier, it needs to be re-packed in the original shipping container. Using the original shipping container, your Cary 630 can be removed and re-packed in its container in a matter of seconds, while maintaining instrument security. Shipping the Cary 630 FTIR in packaging other than the original shipping container will void the warranty.

- 2 The shipping container should contain the standard items listed below as well as any optional items ordered. Inspect the enclosure closely to make sure all items have been removed from the shipment packaging. Also ensure that all the items in the packing list below have arrived undamaged and are in good working condition. Contact Agilent immediately if any items are found to be missing or damaged in shipment.

Getting Started

NOTE

Keep all original packing material for storing, shipping and transporting the system in the future.

NOTE

Shipping the Cary 630 FTIR in packaging other than the original shipping container will void the warranty.

- 3 Remove the Cary 630 FTIR system from its shipping case and place it on its side on a flat, stable surface. The instrument must be kept away from hot surfaces and any sources of electromagnetic interference.

Packing list

- One Agilent Cary 630 FTIR spectrometer system
- One Agilent Cary 630 FTIR spectrometer User's Guide
- One Agilent MicroLab Software USB flash drive (UFD)
- One Agilent Cary 630 FTIR AC power cable
- One Agilent Cary 630 FTIR external power supply
- One Agilent Cary 630 FTIR USB cable
- One shipping container

Options

Although the Cary 630 FTIR system comes standard as shown above, there are a number of options available, including:

- Laptop/Desktop computer
- Interchangeable Accessories as ordered
- Spare USB cable
- Spare Power Cable/Supply

Installation

The Cary 630 FTIR is designed to be completely customer-installable. Install your Cary 630 according to the guidelines below. For a quick reference guide refer to the installation card.

Components

Attaching the sampling accessory or interface is necessary in order to ensure proper working order of the Cary 630 FTIR. To install any of your accessories:

- 1 Press the release lever outward.
- 2 The accessory is meant to slide into the recessed or dovetailed area. This is done by matching the dovetail to the front plate and sliding in a downward fashion (see Figure 1).
- 3 The accessory is in place when the release lever snaps back into place.

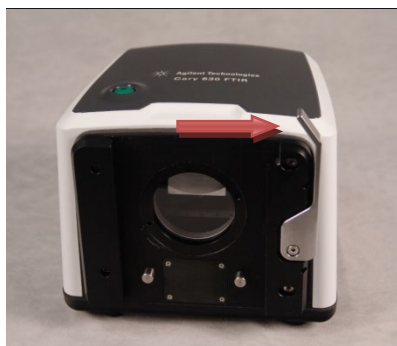


Figure 1. Press the release lever of the Cary 630 Engine outwards before sliding the accessory into place.

Connecting power

To connect power to the spectrometer system:

- 1 Insert the provided power connection from power supply into the power connector located on the lower left side at the rear of the Cary 630 FTIR system (see Figure 2).



Figure 2. Attaching the power cord.

- 2 Thread the blue cap onto the power connector by rotating it clockwise until finger-tight. To avoid damage to the plastic connector, be careful not to over-tighten.
- 3 The appropriate power supply cabling will be provided with the system. Connect the opposite end of the power cable to the AC power outlet.
- 4 Connect the Mini USB communication cable to the USB connector on the back right of the instrument.
Thread the blue cap onto the USB connector by rotating it clockwise until finger-tight. To avoid damage to the plastic connector, be careful not to over-tighten.

CAUTION

To prevent any power startup damage or problems, connect the power cord to the spectrometer system first and then to the power outlet.

CAUTION

To avoid damage to the system due to power surges or a faulty power source, always use a UL approved power surge protection strip between the power outlet source and the system power cord.

CAUTION

All cords must be kept away from high traffic areas. The system could sustain damage to the apparatus or adapters if excessive strain to the power cord connections occurs.

Position the equipment for easy access to the disconnecting switch.

Power cord selection

All power supplies for the Agilent (product, accessories) should be IEEE 519-2022 standard compliant single-phase AC. 3 wire systems (active, neutral, ground; or two active and ground).

CAUTION

If necessary, replace the power cord only with a cord equivalent to the one specified below.

The following power cords can be used:

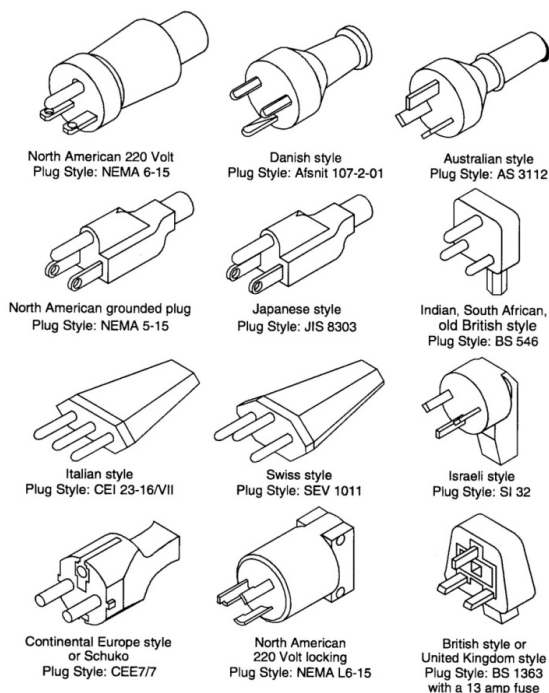


Figure 3. Suitable power cords.

Environment

CAUTION

Your Cary 630 FTIR must be kept away from hot surfaces and sources of electromagnetic interference.

MicroLab PC software

Your Cary 630 FTIR is supplied with Agilent MicroLab PC software. For instructions on installing the software, see the Agilent MicroLab PC Software User's Guide. The software manual is located on the Software installation CD. It provides more in depth information about logging on, managing users, editing methods and reviewing data.

Turning On the Instrument

To turn on the instrument:

- 1 Press the power button of the Cary 630 FTIR system and hold for two (2) seconds. Initially, the light will flash between red and green while the firmware is being loaded. This process should take less than 15 seconds. When the LED light illuminates solid green, the instrument is now ready to use.
- 2 The spectrometer must have a warmup period of 5 minutes before reliable analysis may begin.

When finished using the instrument, press the green power button again to turn off the system. The LED light will turn red.

Power switch LED status

The power button contains a two-colored LED. The color displayed on the LED can indicate the state of the system. The table below lists the colors of the LED and the status of the instrument.

Table 1 Power switch LED status

| Status | LED-color | Action (% duty cycle) |
|-----------------|---------------------|--|
| System OFF | No LED illumination | N/A |
| System OFF | Red | 100% |
| System startup | Red / green | Green 0.5 s on / red 0.5 s on |
| System ON | Green | 100% |
| Firmware update | Red / green | Green blinks rapidly twice then red blinks rapidly twice |

Turning Off

When you have finished using the spectrometer, press the green power button to turn off the system. The LED light will turn red.

NOTE

To help keep moisture out of the Cary 630 FTIR, it is recommended to leave the spectrometer on, even when not in use.

CAUTION

The power button is a momentary switch, to avoid accidental power off. In order to turn the system on or off, hold down the button for two (2) seconds.

Performance Verification

Your Cary 630 FTIR has been thoroughly tested at the factory, so no alignment steps are required. However, it is recommended to run the performance test initially to ensure the spectrometer is running properly. To run and interpret the performance test, refer to the Agilent MicroLab PC Software User's Guide. The performance test measures the energy level of the spectrometer (based on the interferogram voltage or height). A successful test will be indicated by a green circle at the top of the software screen. You are then ready to analyze a sample. A yellow or red circle indicates the spectrometer is operating outside of the factory-defined parameters. These parameters are listed in Table 1 as well as on the Advanced Features software page.

NOTE

If the circle at the top of the software screen is yellow or red, contact Agilent technical support for assistance.

Like any measurement device, it is important to verify that the Cary 630 FTIR is performing properly before using the system to make critical measurements. The Cary 630 provides both diagnostic values and performance validation tests to demonstrate the system's performance.

The diagnostic values provide an easy to understand, quick assessment of the spectrometer's function. If the spectrometer is not functioning properly, one of the diagnostic values will be out of specification.

Performance validation tests are longer, more involved tests that measure how well the spectrometer is working. Validation tests check the spectrometer's sensitivity (performance), stability and frequency precision (laser calibration).

Each industry has different requirements for instrument verification. In general, the diagnostic values should be checked on a daily or weekly basis. Typically, the spectrometer will not collect data if the diagnostic values are grossly out of specification, but it is good practice to verify that the spectrometer is working properly.

Performance validation should be run on a quarterly, bi-annual or annual basis. Highly-regulated industries may require the performance validation to be run monthly, depending on the use of the instrument.

Diagnostic values

The following values can be viewed on the Diagnostics page in the MicroLab PC software. In each case, the Optimal value indicates that the Cary 630 FTIR system is running at its intended performance level. The Marginal value indicates that the spectrometer is functioning, but at a lower performance level. The Critical value indicates that the system is not working correctly, in which case you should contact Agilent technical support for assistance.

NOTE

When using the Diffuse Reflectance sampling accessory, the appropriate reflectance disk must be in place to make these measurements. The diffuse gold reference disk should be used with the Diffuse Reflectance sampling accessory. This disk is permanently mounted into Position 1 of the sampling tray.

Table 2 Diagnostic values

| Value | Optimal (green) | Marginal (yellow) | Critical (red) | Comment |
|-----------------------|-----------------|----------------------|-----------------------|--|
| Energy (center-burst) | 28,000-15,000 | >28,000 or <10000 | > 30,000 or < 3000 | Indicates the overall alignment and proper gain adjustment for the system. |
| Source | 1.9 A | >2.2 A or <1.6 A | >2.4 A or <1.4 A | Indicates a problem with the infrared source control voltage, or that the source has burnt out. Both voltage and amperage are displayed; however, amperage provides a sufficient diagnostic. |
| Laser signal | 15,000-3,000 | >15,000 or <3,000 | >17,000 or < 2,000 | Can verify gross alignment errors even if the reflectance cap is not in place. |
| Detector temperature | 38–42 | <38 or >42 | <36 or >44 | Indicates a problem with the cooling circuitry or an ambient temperature over the specified range. |
| CPU temperature | 10–75 | >75 | >80 | Indicates the ambient temperature over the specified range. |

Validation

The MicroLab PC software has three tests appropriate for performance validation of the Cary 630 FTIR. Performance validation verifies the key aspects of the instrument's ability to measure good data. If the performance validation tests are within specification, the spectrometer should provide data that works well with methods developed for the Cary 630. As with any instrument, however, results can always be verified by running a known sample with the sample-specific method.

Getting Started

The validation tests are accessed from the Advanced Features, System Check page of the MicroLab PC software. The Cary 630 FTIR system should be warmed up for at least 120 minutes before conducting any of these tests.

Performance (signal-to-noise) test

This test measures the signal-to-noise level at two regions of the infrared spectrum: $2,500\text{ cm}^{-1}$ and $1,000\text{ cm}^{-1}$. Both the background and sample are measured at 4 cm^{-1} resolution with a one minute collection time. This test takes two minutes per test. You can specify a number of tests to be conducted. For diffuse reflectance measurements, the appropriate sample position must be in place with the diffuse gold mirror. No sample is needed when using the Diamond ATR sampling accessory. At least 10 tests should be collected in order to obtain an accurate picture of the performance.

Stability test

This test measures the short-term stability at two regions of the spectrum: $3,000\text{ cm}^{-1}$ and $1,000\text{ cm}^{-1}$. Stability is a measurement of the baseline differences observed over the selected time period. The test measures a background at the beginning, then one sample every minute for the duration of the test as specified in the 'Number of minutes' field in the software. For diffuse reflectance measurements, the appropriate sample position must be in place with the diffuse gold mirror. No sample is needed when using the Diamond ATR sampling accessory. The test results are expressed as %Transmittance (difference versus 100%) of maximum deviation during the Stability test.

Laser Frequency Calibration test

The Laser Frequency Calibration test measures the frequency (X-axis) accuracy. The test is conducted by measuring a spectrum of a polystyrene film. Absorbance frequencies from this spectrum are compared to frequencies set by NIST SRM 1921 polystyrene film. The software allows administrative-level users to use the test results to set the laser calibration. Laser calibrations should only be set after consultation with an Agilent technical support engineer. For this test, a background is measured first. After the background collection, a spectrum of polystyrene film is measured, as instructed by the software. For the Diamond ATR sampling accessory, no sample is required for a background measurement, but the polystyrene test sample must be pressed firmly against the diamond sample interface during the sample collection using the sample press attachment. Neither the multi-bounce ATR, nor the diffuse reflectance accessory measurements should be used for the laser frequency calibration test.

3 Analyzing samples

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The Agilent Cary 630 FTIR spectrometer requires a sampling accessory in order to present the sample to the infrared beam. You will have selected one or more sampling accessories when you ordered your Cary 630 FTIR. Additional sampling accessories can be purchased at a later date if your sampling needs change.

The basic steps for analyzing a sample with all sampling accessories are:

- 1 Clean the sample interface.
- 2 Collect a background spectrum.
- 3 Collect a sample spectrum.

NOTE

To ensure accuracy of the measurement, it is recommended that the system is configured in the software to collect a background spectrum before every sample is analyzed. This provides a baseline profile of the system conditions with no sample loaded on the instrument. By collecting a background automatically before each sample measurement, negative effects of changes in the environment can be avoided.

This section covers specific instructions for how to analyze a sample with each sampling accessory, as well as how to interchange accessories.

Interchanging Sampling Accessories

To switch between different sampling accessories:

- 1 Before attempting to change the sampling accessory, ensure that the Cary 630 FTIR system is on a solid surface with the accessory facing towards you.
- 2 To remove an installed sampling accessory, push the retaining lever located on the right side of the instrument to the side. While pushing the lever to the side, slide the accessory upwards to remove it from the main Cary 630 FTIR unit.



Figure 4. Interchanging sample accessories.

CAUTION

When removed, the opening in the mounting surface of the sampling accessory is exposed to the ambient environment, and delicate optical components are contained inside. Use caution when handling to ensure the accessory stays clean and nothing comes in contact with the optical components.

- 3 Slide the new sampling accessory into the recessed areas of the main unit. The lever will click back into place when the accessory is properly seated.

Notice the two stainless steel dowel pins protruding out of the Cary 630 FTIR unit. This is to ensure proper placement and alignment of the accessories.

- 4 Store the removed sampling accessory in a clean, protected area, such as in a plastic bag inside foam-lined shipping container.
- 5 The MicroLab PC Software will recognize the new sampling accessory and will display the appropriate accessory picture in the software.
 - The recognition of the accessory takes a few moments for the RFID reader to recognize the accessory and change the diagnostic values to represent the appropriate sampling device and throughput level. Wait for the software

Analyzing samples

- to return you to the main screen where the image will change to the new accessory.
 - Once this image changes, note the status indicator in the software to determine when the instrument is ready to use.
 - The user should also change to an appropriate method for the new accessory and the software will prompt for action.
- 6 To ensure everything is correctly working, review the Diagnostics values section on Page 19.

Diamond ATR Sampling Accessory

The Diamond attenuated total reflectance (ATR) sampling accessory takes advantage of the physical properties of light when encountering two materials with differences in index of refraction. When coming in contact with a sample having a different index of refraction, the infrared light creates an evanescent wave, which provides a very small and specific depth of penetration into the sample before reflecting back into the detector of the spectrometer. This small and consistent pathlength means no sample preparation is required to get good measurement results from a variety of samples. The key to obtaining good results with the Diamond ATR accessory is to make good contact between the sample and ATR crystal. The ATR technique can be used for analyzing liquids, pastes, powders and even some solid samples.

The Diamond ATR accessory uses a type IIa diamond crystal as the interface between the sample and the infrared energy. Advantages of a diamond crystal are extreme hardness and chemical resistivity, plus it can accept samples with a pH range from 1 to 14. Therefore, hard or abrasive samples and even strong acids can be safely analyzed. The diamond in the Agilent ATR accessory is the world's most durable substance and has been designed for maximum infrared signal.

The Diamond ATR is a single-reflection ATR. It is most suitable for higher absorbing samples such as rubber, polymers, paints and fibers. Powder and solid samples are also best measured with the single-reflection ATR, by using the sample press device, which applies high pressure to powder and solid samples to ensure good contact with the diamond crystal. The single-reflection ATR is also a good choice when the amount of available sample is limited. The single-reflection diamond has a 1 mm diameter sampling surface with 200 μm active area and provides approximately 2 μm depth of penetration for infrared energy at $1,700\text{ cm}^{-1}$. The single-reflection ATR protrudes slightly above the metal mounting plate.

Analyzing samples

In the sample loading and cleaning mode, the sample press should be in the highest position, so the sample press tip is well above the diamond sampling window (see Figure 5). It is in this position that the sample mounting area can be easily accessed for loading the sample into place and for cleaning the sampling surfaces before the next sample is analyzed.

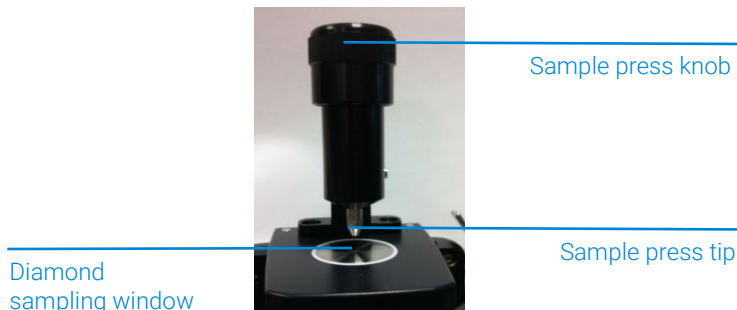


Figure 5. Diamond ATR accessory sample press in highest position.

In the sample analysis mode, the sample press is lowered such that the tip is in contact with a solid sample. In this position, contact is made between the sample and the infrared energy emitting from the diamond window.

NOTE

If the sample is a liquid or paste, the sample press does not need to be used at all.

CAUTION

When using the swivel press mechanism, please ensure that the press and tip are in the upmost position before turning the press out of or into position.

When in the sample analysis mode, the ATR sample pathlength is fixed based on the number of reflections in the ATR. The alignment of the accessory is also pre-set at the factory, and so there is no optical or mechanical adjustment required.

Analyzing samples

Cleaning

To clean the Diamond ATR accessory in preparation for analysis:

- 1 Open the sample press by rotating the top knob counterclockwise until the top of the press travel is reached (see Figure 5).
- 2 Clean the sample press tip using an extra-low lint, non-abrasive wipe and a suitable solvent, such as acetone, methanol, ethanol or isopropyl alcohol.
- 3 Clean the diamond sampling window.

CAUTION

Do not break the spectrometer seal and attempt to clean interior surfaces. Breaking the seal will void the warranty.

Collecting a background spectrum

For instructions on collecting a background using the software, refer to the Agilent MicroLab PC Software User's Guide.

CAUTION

To ensure an accurate background spectrum is collected, perform a visual inspection of the diamond sampling window, looking for any haze or film present from measuring the previous sample. If a film is observed, repeat the cleaning procedure on Page 25 until the sampling window is clear of any residue.

Collecting a sample spectrum

To load a sample and collect a spectrum using the Diamond ATR accessory:

- 1 Turn the sample press knob in a counterclockwise direction to open the sample press so that the tip is elevated slightly from the diamond sampling window (see Figure 5).
- 2 Place a small amount of material to be measured on the diamond crystal. The crystal is the clear, circular shaped material held in place by the surrounding metal disk.
- 3 Ensure that the sample covers the entire surface area of the diamond crystal.

In the case of a volatile sample, larger amounts of sample can be applied without concern of leakage or damage to the instrument. However, using the smallest amount of sample possible will ease in the cleaning process.

Analyzing samples

CAUTION

The Diamond ATR accessory windows are made from type IIa synthetic diamond, which is extremely chemically-resistant. However, it is still possible the diamond crystal or metal mounting plate can be damaged by extreme samples. Only measure samples with a pH between 1 and 14. Do not leave extremely acidic samples on the metal mounting plate for an extended time.

CAUTION

Although diamond is a very hard material, the ATR window is relatively thin (0.5 mm or less) and can crack under extreme pressure conditions. Ensure that the sample makes contact with the entire surface area of the diamond and not just a point within the diamond. Avoid using the sample press on samples that may be sharp or pointed.

If the sample is a liquid or paste, the sample press does not need to be used at all. In this case, you are now ready to proceed with the analysis.

If the sample is a powder or solid, the sample press must be in contact with the sample. To make contact, rotate the sample press knob in a clockwise direction until it reaches the detent (click-in) position. The sample press arm uses a slip clutch mechanism, whereby the maximum applied pressure cannot be surpassed — once maximum pressure has been reached, the slip clutch mechanism will click and not allow any extra pressure to be exerted on the sample.

- 4 For instructions on collecting a sample spectrum using the software, refer to the Agilent MicroLab PC Software User's Guide.
- 5 After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided on Page 25. It is important to make sure both the diamond sampling window and the sample press tip are free of any residue from the previous sample.

For instructions on reviewing results and handling sample data, refer to the Agilent MicroLab PC Software User's Guide.

ZnSe ATR Sampling Accessory

The ZnSe attenuated total reflectance (ATR) sampling accessory takes advantage of the physical properties of light when encountering two materials with differences in index of refraction. When coming in contact with a sample having a different index of refraction, the infrared light creates an evanescent wave, which provides a very small and specific depth of penetration into the sample before reflecting back into the detector of the spectrometer. This small and consistent pathlength means no sample preparation is required to get good measurement results from a variety of samples. The key to obtaining good results with the ZnSe ATR accessory is to make good contact between the sample and ATR crystal. The ZnSe ATR technique should be used primarily for analyzing liquids, gels and some soft solids.

The ZnSe ATR accessory uses a Zinc Selenide crystal as the interface between the sample and the infrared energy. Advantages of a ZnSe crystal are its high throughput without the diamond band interference seen with the DATR. It can accept samples with a pH range from 5 to 9. Hard or abrasive samples as well as strong acids should not be analyzed using the ZnSe ATR.

The ZnSe ATR is a single-reflection ATR. It is typically used as an all-purpose ATR for measuring liquids, powders, gels and soft solids. Powder and solid samples are best measured with the single-reflection ATR, by using the sample press device, which applies relatively low pressure to powder and solid samples to ensure good contact with the ZnSe crystal. The single-reflection ATR is also a good choice when the amount of available sample is limited. The single-reflection ZnSe ATR, similar to the diamond ATR, has a 1 mm diameter sampling surface with 200 μm active area and provides approximately 2 μm depth of penetration for infrared energy at $1,700\text{ cm}^{-1}$. The single-reflection ATR protrudes slightly above the metal mounting plate to ensure good contact and allow the user to clean the crystal effectively.

In the sample loading and cleaning mode, the sample press should be in the highest position, so the sample press tip is well above the ZnSe sampling window (see Figure 6). It is in this position that the sample mounting area can be easily accessed for loading the sample into place and for cleaning the sampling surfaces before the next sample is analyzed.

Analyzing samples

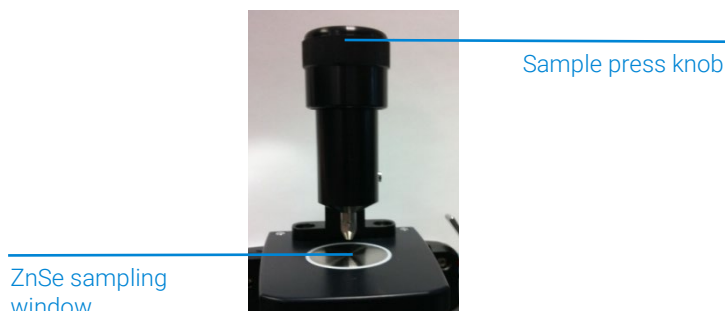


Figure 6. ZnSe ATR accessory sample press in highest position.

In the sample analysis mode, the sample press is lowered such that the tip is in contact with a solid sample. In this position, contact is made between the sample and the infrared energy emitting from the ZnSe window.

NOTE

If the sample is a liquid or paste, the sample press does not need to be used at all.

CAUTION

When using the swivel press mechanism, please ensure that the press and tip are in the upmost position before turning the press out of or into position.

When in the sample analysis mode, the ATR sample pathlength is fixed based on the number of reflections in the ATR. The alignment of the accessory is also pre-set at the factory, and so there is no optical or mechanical adjustment required.

Cleaning

To clean the ZnSe ATR accessory in preparation for analysis:

- 1 Open the sample press by rotating the top knob counterclockwise until the top of the press travel is reached (see Figure 6).
- 2 Clean the sample press tip using an extra-low lint, non-abrasive wipe and a suitable solvent, such as acetone, methanol, ethanol or isopropyl alcohol.
- 3 Clean the sampling window.

CAUTION

Do not break the spectrometer seal and attempt to clean interior surfaces. Breaking the seal will void the warranty.

Analyzing samples

Collecting a background spectrum

For instructions on collecting a background using the software, refer to the Agilent MicroLab PC Software User's Guide.

CAUTION

To ensure an accurate background spectrum is collected, perform a visual inspection of the sampling window, looking for any haze or film present from measuring the previous sample. If a film is observed, repeat the cleaning procedure on Page 25 until the sampling window is clear of any residue.

Collecting a sample spectrum

To load a sample and collect a spectrum using the ZnSe ATR accessory:

- 1 Turn the sample press knob in a counterclockwise direction to open the sample press so that the tip is elevated slightly from the ZnSe sampling window (see Figure 6).
- 2 Place a small amount of material to be measured on the crystal. The crystal is the clear, circular shaped material held in place by the surrounding metal disk.
- 3 Ensure that the sample covers the entire surface area of the ZnSe crystal.

In the case of a volatile sample, larger amounts of sample can be applied without concern of leakage or damage to the instrument. However, using the smallest amount of sample possible will ease in the cleaning process.

CAUTION

Although ZnSe is fairly resistant to most chemicals, it is still possible that the ZnSe crystal or metal mounting plate can be damaged by extreme samples. Only measure samples with a pH between 5 and 9. Do not leave extremely acidic samples on the metal mounting plate for an extended time.

CAUTION

Hard or abrasive material can scratch or crack the ZnSe Crystal and cause deterioration in the ZnSe ATRs performance.

CAUTION

The ZnSe crystal is naturally a hard and brittle material. The ATR window is relatively thin (0.5 mm or less) and can crack under excessive pressure conditions. Ensure that the sample makes contact with the entire surface area of the ZnSe and not just a point within the ZnSe. Avoid using the sample press on samples that may be sharp or pointed.

If the sample is a liquid or paste, the sample press does not need to be used at all. In this case, you are now ready to proceed with the analysis.

If the sample is a powder or solid, the sample press must be in contact with the sample. To make contact, rotate the sample press knob in a clockwise direction until it reaches the detent (click-in) position. The sample press arm uses a slip clutch mechanism, whereby the maximum applied pressure cannot be surpassed — once maximum pressure has been reached, the slip clutch mechanism will click and not allow any extra pressure to be exerted on the sample.

- 4 For instructions on collecting a sample spectrum using the software, refer to the Agilent MicroLab PC Software User's Guide.
- 5 After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided on Page 25. It is important to make sure both the ZnSe sampling window and the sample press tip are free of any residue from the previous sample.

For instructions on reviewing results and handling sample data, refer to the Agilent MicroLab PC Software User's Guide.

Germanium ATR Sampling Accessory

The Agilent Germanium attenuated total reflectance (Ge ATR) sampling accessory for the Cary 630 FTIR takes advantage of the physical properties of light when encountering two materials with differences in index of refraction. When coming in contact with a sample having a different index of refraction, the infrared light creates an evanescent wave, which provides a very small and specific depth of penetration into the sample before reflecting back into the detector of the spectrometer. This small and consistent pathlength means no sample preparation is required to get good measurement results from a variety of samples. The Ge ATR, while similar in design to the Agilent diamond ATR accessory, offers different characteristics due to the use of a Ge crystal. Shallower depth of penetration is achieved with a Ge crystal, resulting in a shorter pathlength. This is important when measuring samples containing carbon black, such as tires, rubbers and o-rings, as the characterization and identification of these highly absorbing samples is made simpler with shorter pathlengths. The key to obtaining good results with the Ge ATR accessory is to make good contact between the sample and ATR crystal. The ATR technique can be used for analyzing liquids, pastes, powders and solid samples.

The Ge ATR accessory uses a single bounce 45° crystal as the interface between the sample and the infrared energy and is a single-reflection ATR. It is most suitable for higher absorbing samples such as tires, rubbers and o-rings, or any sample containing carbon black. Powder and solid samples are also best measured with the single-reflection ATR, by using the sample press device, which applies high pressure to powder and solid samples to ensure good contact with the Ge crystal. The single-reflection ATR is also a good choice when the amount of available sample is limited. The single-reflection Ge has a 1 mm diameter sampling surface and provides the following effective pathlengths:

- 0.15 μm at 4,000 cm^{-1}
- 0.36 μm at 1,700 cm^{-1}
- 1.02 μm at 600 cm^{-1}

In the sample loading and cleaning mode, the sample press should be in the highest position, so the sample press tip is well above the Ge sampling window (see Figure 7). It is in this position that the sample mounting area can be easily accessed for loading the sample into place and for cleaning the sampling surfaces before the next sample is analyzed.

Analyzing samples



Figure 7. Ge ATR accessory sample press.

In the sample analysis mode, the sample press is lowered such that the tip is in contact with a solid sample. In this position, contact is made between the sample and the infrared energy emitting from the Ge window.

NOTE

If the sample is a liquid or paste, the sample press does not need to be used at all.

CAUTION

When using the swivel press mechanism, please ensure that the press and tip are in the upmost position before turning the press out of or into position.

When in the sample analysis mode, the ATR sample pathlength is fixed, based on the number of reflections in the ATR. The alignment of the accessory is also pre-set at the factory, so there is no optical or mechanical adjustment required.

Cleaning

To clear the Ge ATR accessory in preparation for analysis:

- 1 Open the sample press by rotating the top knob counterclockwise until the top of the press travel is reached (see Figure 7).
- 2 Clean the sample press tip using an extra-low lint, non-abrasive wipe and a suitable solvent, such as acetone, methanol, ethanol or isopropyl alcohol.
- 3 Clean the Ge sampling window.

CAUTION

Do not break the spectrometer seal and attempt to clean interior surfaces. Breaking the seal will void the warranty.

Collecting a background spectrum

For instructions on collecting a background using the software, refer to the Agilent MicroLab PC Software User's Guide.

CAUTION

To ensure an accurate background spectrum is collected, perform a visual inspection of the Ge sampling window, looking for any haze or film present from measuring the previous sample. If a film is observed, repeat the cleaning procedure until the sampling window is clear of any residue.

Collecting a sample spectrum

To load a sample and collect a spectrum using the Ge ATR accessory:

- 1 Turn the sample press knob in a counterclockwise direction to open the sample press so that the tip is elevated slightly from the Ge sampling window (see Figure 7).
- 2 Place a small amount of the material to be measured on the Ge crystal. The crystal is the clear, circular shaped material held in place by the surrounding metal disk.
- 3 Ensure that the sample covers the entire surface area of the Ge crystal.

In the case of a volatile sample, larger amounts of sample can be applied without concern of leakage or damage to the instrument. However, using the smallest amount of sample possible will ease in the cleaning process.

CAUTION

The Ge ATR accessory windows or metal mounting plate can be damaged by extreme samples. Only measure samples with a pH between 1 and 14. Do not leave extremely acidic or basic samples on the metal mounting plate for an extended time.

CAUTION

Although Ge is a hard material, the ATR window can crack under extreme pressure conditions. Ensure that the sample makes contact with the entire surface area of the crystal and not just a point within the crystal. Avoid using the sample press on samples that may be sharp or pointed.

Analyzing samples

If the sample is a liquid or paste, the sample press does not need to be used at all. In this case, you are now ready to proceed with the analysis.

If the sample is a powder or solid, the sample press must be in contact with the sample. To make contact, rotate the sample press knob in a clockwise direction until the pressure arm no longer goes down. A spring mechanism keeps pressure on the sample; take care not to over tighten the clamp. Once the pressure arm stops moving, apply no more than $\frac{1}{4}$ turn of the sample knob.

- 4** For instructions on collecting a sample spectrum using the software, refer to the Agilent MicroLab PC Software User's Guide.
- 5** After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided on Page 32. It is important to make sure both the Ge sampling window and the sample press tip are free of any residue from the previous sample.

For instructions on reviewing results and handling sample data, refer to the Agilent MicroLab PC Software User's Guide.

5 Bounce Zinc Selenide ATR Sampling Accessory

The 5 bounce zinc selenide attenuated total reflectance (5 bounce ZnSe ATR) sampling accessory takes advantage of the physical properties of light when encountering two materials with differences in index of refraction. When coming in contact with a sample having a different index of refraction, the infrared light creates an evanescent wave, which provides a very small and specific depth of penetration into the sample before reflecting back into the detector of the spectrometer. The light is reflected multiple times to lengthen the effective pathlength, giving lower detection levels than a single bounce ATR.

Although easy to use, single bounce ATR does have the limitation of low sensitivity compared to other FTIR techniques. Many methods now require multi-bounce ATRs because of the greater sensitivity they provide. The 5 bounce ZnSe ATR improves levels of detection while still providing the ease-of-use of a single bounce ATR. In addition, the accessory complies with the ASTM D-7371 Biodiesel method, making it ideal for this application.

When in the sample analysis mode, the ATR sample pathlength is fixed based on the number of reflections in the ATR. The alignment of the accessory is also pre-set at the factory, and so there is no optical or mechanical adjustment required.

Analyzing samples

Cleaning

Clean the 5 bounce ZnSe sampling window area using an extra-low lint, non-abrasive wipe or cotton swab and a suitable solvent, such as acetone, methanol, ethanol or isopropyl alcohol.



Figure 8. Cleaning the 5 bounce ZnSe ATR sampling window.

CAUTION

Do not break the spectrometer seal and attempt to clean interior surfaces. Breaking the seal will void the warranty.

Collecting a background spectrum

For instructions on collecting a background using the software, refer to the Agilent MicroLab PC Software User's Guide.

CAUTION

To ensure an accurate background spectrum is collected, perform a visual inspection of the 5 bounce ZnSe sampling window, looking for any haze or film present from measuring the previous sample. If a film is observed, repeat the cleaning procedure on Page 35 until the sampling window is clear of any residue.

Analyzing samples

Collecting a sample spectrum

To load a sample and collect a spectrum using the 5 bounce ZnSe ATR accessory:

- 1 Place a small amount of the liquid to be measured on the ZnSe crystal. The crystal is the clear, circular shaped material held in place by the surrounding metal disk.
- 2 Ensure that the sample covers the entire surface area of the ZnSe crystal.

In the case of a volatile sample, larger amounts of sample can be applied without concern of leakage or damage to the instrument. However, using the smallest amount of sample possible will ease in the cleaning process.

CAUTION

The 5 bounce ZnSe ATR accessory windows are made from ZnSe, which is chemically-resistant. However, it is still possible the crystal or metal mounting plate can be damaged by extreme samples. Only measure samples with a pH between 4 and 9. Do not measure samples or use cleaners that fall outside this pH range.

-
- 3 For instructions on collecting a sample spectrum using the software, refer to the Agilent MicroLab PC Software User's Guide.
 - 4 After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided on Page 35. It is important to make sure the 5 bounce ZnSe sampling window is free of any residue from the previous sample.

For instructions on reviewing results and handling sample data, refer to the Agilent MicroLab PC Software User's Guide.

Transmission Sampling Accessory

The standard Transmission sampling accessory (see Figure 9) has a card slide mount holder, which is compatible with industry standard sized holders. Such holders include those for KBr pellets, liquid cells and salt plates.

Analyzing samples

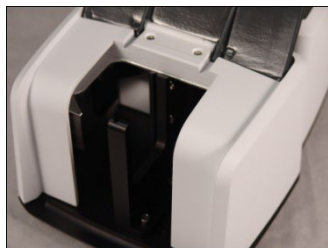


Figure 9. Transmission accessory showing sample compartment.

A recessed area in the front panel allows for tubing access to liquid cells.

Transmission analysis is typically used for the identification of unknown materials. Salt plates are most commonly used as the sample support for unknown liquid materials, whereas KBr pellets are used to dilute and support solid materials into the infrared beam.

Transmission analysis can also be used for quantitative analysis using sealed cells or demountable cells with constant pathlengths. The Transmission accessory for the Cary 630 FTIR is specifically designed to accept industry standard sized cells. These can be obtained directly from Agilent or from your preferred accessory vendor.

The Agilent MicroLab PC Software enables you to perform simple linear or Beer's law calculations based on peak heights, areas, or ratios. Modeling via PLS programs is also available and can be easily integrated into the software. For information on how to do this, refer to the Agilent MicroLab PC Software User's Guide.

Cleaning

To clean the Transmission accessory in preparation for analysis:

- 1** Ensure that the compartment is free of any debris or other matter that may cause interference with your sampling.
- 2** Follow the manufacturer's recommendations for cleaning of any transmission accessory that is used for measurements, such as liquid cells, KBr pellet holders and salt plates.

NOTE

Agilent is not responsible for the improper care of accessories used with the Transmission accessory.

CAUTION

Do not attempt to clean the mirrors within the Transmission accessory compartment. Solvents and/or canned air may cause irreversible spectral interferences, as well as damage the front surface mirrors. This can have a detrimental effect on the throughput and performance of the system. Doing so will void the warranty.

Collecting a background spectrum

To collect a background spectrum using the Transmission accessory:

- 1 Place the material used for background measurements in the Transmission accessory sample compartment. This can be an empty sample compartment or it can be an empty cell, depending on your standard operating procedure.
- 2 Collect a background spectrum. For instructions on collecting a background using the software, refer to the Agilent MicroLab PC Software User's Guide.

Collecting a sample spectrum

To collect a sample spectrum using the Transmission accessory:

- 1 Place your sample in the sample holder

CAUTION

Ensure that the sample is correctly placed in the holder and that it is properly aligned for the infrared beam to pass through the sample. Use the positioning pin at the bottom of the holder to establish the correct distance from the bottom of the sample compartment.

- 2 Collect a sample spectrum. For instructions on collecting a sample spectrum using the software, refer to the Agilent MicroLab PC Software User's Guide.

For instructions on reviewing results and handling sample data, refer to the Agilent MicroLab PC Software User's Guide.

10 and 45 Degree Specular Reflectance Sampling Accessories

The Agilent 10 and 45 degree specular reflectance accessories (SRA) for the Cary 630 FTIR allow for near-normal (<10 degrees) or at-angle (>10 degrees) specular reflectance measurements. Specular reflectance measurements are made on samples that, in general, are designed to reflect light from a flat surface. Examples include mirrors, glass, windows and thin film coatings placed upon highly reflective substrates. The intended end use of the product typically dictates whether a sample is measured at near-normal and/or at-angle. Use the 10 degree SRA for near-normal specular reflectance measurements and the 45 degree SRA to measure specular reflectance at-angle and for the measurement of relatively thin films on reflective substrates.



Figure 10. 10 degree (left) and 45 degree (right) SRAs.

The 10 degree SRA slides in and out of the Cary 630 FTIR in seconds with no alignment required. See Page 22 for installation instructions.

The 45 degree SRA easily slides into the transmission accessory.

To install the 45 degree SRA into the Cary 630 FTIR:

- 1 Undo the two screws holding the standard transmission holder and then replace the holder with the one provided with the 45 degree SRA. Screw in the two screws to secure it to the transmission accessory. This modified holder has a shorter supporting arm on the front to allow greater access to the top of the accessory for larger samples.
- 2 Slide the transmission accessory onto the Cary 630 FTIR.
- 3 Slide the 45 degree SRA into the transmission accessory.

Analyzing samples

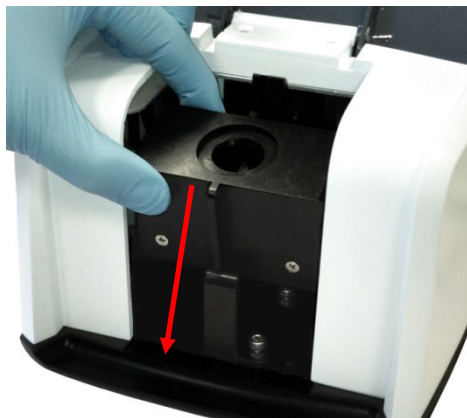


Figure 11. Installing the 45 degree SRA into the Cary 630 FTIR transmission accessory.

The Agilent MicroLab PC Software enables you to perform simple linear or Beer's law calculations based on peak heights, areas, or ratios. Modeling via PLS programs is also available and can be easily integrated into the software. For information on how to do this, refer to the Agilent MicroLab PC Software User's Guide.

Cleaning

To clean the 10 degree or 45 degree SRA in preparation for analysis:

- For the 10 degree SRA, ensure that the sampling window is free of any debris or other matter that may cause interference with your sample. Do not measure powdered samples or samples with coatings that can fall into the accessory and contaminate the mirrors inside the accessory.
- For the 45 degree SRA, ensure that the transmission accessory and sampling window are free of any debris or other matter that may cause interference with your sampling.

CAUTION

Do not attempt to clean the mirrors within the Transmission accessory compartment. Solvents and/or canned air may cause irreversible spectral interferences, as well as damage the front surface mirrors. This can have a detrimental effect on the throughput and performance of the system. Doing so will void the warranty

Analyzing samples

Collecting a background spectrum

To collect a background spectrum using the Transmission accessory:

- 1 Place the material used for background measurements on the 10 degree or 45 degree SRA accessory sampling window. This is commonly the gold coated mirror supplied with the accessory.
- 2 Collect a background spectrum. For instructions on collecting a background using the software, refer to the Agilent MicroLab PC Software User's Guide.

Collecting a sample spectrum

To collect a sample spectrum using the Specular Reflectance accessory:

- 1 Place your sample on the 10 degree or 45 degree SRA sampling window.



Figure 12. Sample introduction for the 10 degree and 45 degree SRA accessory.

- 2 Collect a sample spectrum. For instructions on collecting a sample spectrum using the software, refer to the Agilent MicroLab PC Software User's Guide.
- 3 For instructions on reviewing results and handling sample data, refer to the Agilent MicroLab PC Software User's Guide.

TumblIR Sampling Accessory

The dedicated TumblIR sampling accessory is a proprietary liquid transmission sampling accessory designed and manufactured exclusively by Agilent to optimize the analysis of many liquid, paste and gel samples. Compared to traditional liquid cells for general analytical laboratories, the TumblIR makes it much easier to prepare, load, analyze and clean the sample.

The TumblIR operates in two modes: sample loading/cleaning, and sample analysis.

In the sample loading and cleaning mode, the TumblIR is rotated so the sampling window is facing up. In this position, the sample mounting area can be easily accessed for loading the sample into place and for cleaning the sampling surfaces before the next sample is analyzed.

In the sample analysis mode, the TumblIR is rotated such that the accessory optical window is facing down towards the sample mounting area. In this position, infrared energy passes through the sample making measurement possible. Be sure to rotate the TumblIR arm completely around until it reaches the detent and clicks into position. This detent keeps the accessory stationary during sample analysis.

When in sample analysis mode, the TumblIR pathlength is 100 microns, an optimal pathlength for mid-infrared analysis of many liquid, paste and gel samples. The alignment of the accessory is pre-set at the factory, so there is no adjustment required.

Cleaning

To clean the TumblIR sampling windows in preparation for analysis:

- 1 Rotate the TumblIR arm so the optical sensor is pointing upward.
- 2 Clean the top window using soft cotton cloth dipped in acetone.
- 3 Clean the bottom window.

NOTE

Use only soft cotton cloth, such as cotton swabs or a suitable substitute to clean the optical sensor and sampling area.

Analyzing samples

CAUTION

The windows are made of an infrared-transmitting material, ZnSe. ZnSe is a relatively durable material, but can be easily scratched or damaged if too much pressure is applied during cleaning, or if an abrasive material is used. Materials such as cotton swabs dipped in acetone are recommended for cleaning.

CAUTION

ZnSe is relatively chemically-resistant to materials with a pH range between 4 and 9, but there are some materials, such as strong acids or heavily basic materials, that can harm ZnSe. Avoid having materials in contact with the ZnSe window that are outside of this recommended pH range.

CAUTION

Do not break the spectrometer seal and attempt to clean interior surfaces. Breaking the seal will void the warranty.

Collecting a background spectrum

For instructions on collecting a background using the software, refer to the Agilent MicroLab PC Software User's Guide.

CAUTION

To ensure an accurate background spectrum is collected, perform a visual inspection of the ZnSe window surfaces, looking for any haze or film present on the windows from measuring the previous sample. If a film is observed, repeat the cleaning procedure on Page 42 until the window surfaces are clear of any residue.

Collecting a sample spectrum

To load a liquid sample and collect a spectrum using the TumbllR accessory:

- 1 Open the TumbllR by rotating the arm counterclockwise.
- 2 Place a small amount of material on the bottom window located on the TumbllR base plate. The sample window is the 2 mm diameter yellow material held in place by the surrounding metal disk.
- 3 Ensure that the sample covers the entire surface area of the bottom window.

In the case of a volatile sample, such as with fuels analysis, larger amounts of sample can be applied without concern of leakage or damage to the instrument; however, using the smallest amount of sample possible will ease in the cleaning process.

Although it is safe to run a variety of liquid samples, including, aqueous solutions or even thick pastes such as grease, the TumbllR is not to be used with any solid or powder samples, such as tablets. Using the TumbllR with solid samples will damage the ZnSe windows or modify the pre-set transmission pathlength.

CAUTION

The top and bottom windows are made from ZnSe. ZnSe can be damaged by samples with a pH below 4 and above 9. Only measure samples with a pH between 4 and 9.

CAUTION

ZnSe windows can be easily scratched by hard or abrasive samples. Avoid use of samples that may scratch the surface of the windows.

Optimum transmission pathlength is one important consideration when using the TumbllR for a range of liquid sample types. Because the TumbllR pathlength is pre-aligned and fixed at 100 microns, some sample types, such as high infrared absorbing materials, may not give optimum results.

- 4 Close the TumbllR by rotating the arm clockwise until it clicks into place.
- 5 For instructions on collecting a sample spectrum using the software, refer to the Agilent MicroLab PC Software User's Guide.
- 6 After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided on Page 42. It is important to make sure both the top and bottom windows are free of any residue from the previous sample.

Analyzing samples

For instructions on reviewing results and handling sample data, refer to the Agilent MicroLab PC Software User's Guide.

DialPath Sampling Accessory

The DialPath sampling accessory is based on the same technology as the TumbIIR sampling accessory. The DialPath uses the TumbIIR's unique liquid transmission sampling accessory designed and manufactured exclusively by Agilent to optimize the analysis of many liquid, paste and gel samples. Compared to traditional liquid cells for general analytical laboratories, the DialPath makes it much easier to prepare, load, analyze and clean the sample, while providing the flexibility of choosing from three factory-set pathlengths.

The DialPath operates in two modes: sample loading/cleaning and sample analysis.

In the sample loading and cleaning mode, the DialPath is rotated so the optical window is facing up (see Figure 13). In this position, the sample mounting area can be easily accessed for loading the sample into place and for cleaning the sampling surfaces before the next sample is analyzed.

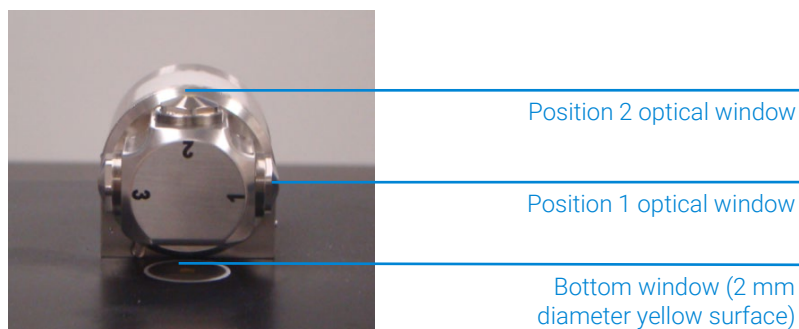


Figure 13. DialPath with optical window facing up.

In sample analysis mode, the DialPath is rotated such that the optical window is facing down towards the sample mounting area. In this position, infrared energy passes through the sample, making measurement possible. The DialPath arm must be completely rotated around until it reaches the detent and clicks into position. This detent keeps the accessory stationary during sample measurement.

Analyzing samples

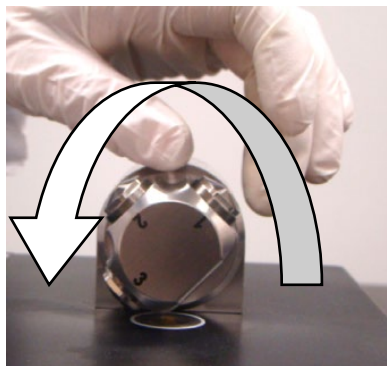


Figure 14. Rotating the DialPath.

When in the sample analysis mode, the DialPath pathlength is set to a predetermined factory-set value in microns. The alignment of the accessory is also pre-set at the factory and so there is no adjustment required.

Cleaning

To clean the DialPath sampling windows in preparation for analysis:

- 1** Rotate the DialPath so that optical window Position 2 is pointing upward (see Figure 13).
- 2** Clean the window of the position last used for analysis, using a cotton swab dipped in acetone (see Figure 15, which shows cleaning Position 3).
- 3** Clean the bottom sample mounting area (see Figure 15).
- 4** If using Position 2, clean either Position 3 or 1, depending on the direction the DialPath was rotated.

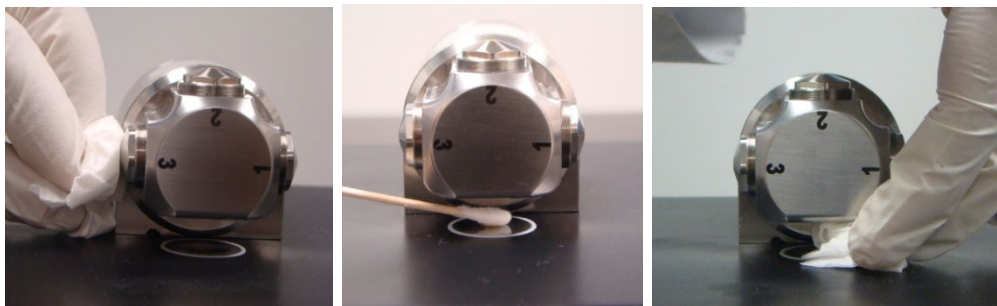


Figure 15. Cleaning the DialPath Position 3 window (left) and cleaning the sample mounting area.

Analyzing samples

NOTE

Use only soft cotton cloth, such as cotton swabs or a suitable substitute to clean the optical windows and sample mounting area.

CAUTION

The windows are made of an infrared-transmitting material, ZnSe. ZnSe is a relatively durable material, but can be easily scratched or damaged if too much pressure is applied during cleaning or if an abrasive material is used. Materials such as cotton swabs dipped in acetone are recommended for cleaning.

CAUTION

ZnSe is relatively chemically-resistant to materials with a pH range between 4 and 9, but there are some materials, such as strong acids or heavily basic materials, that can harm ZnSe. Avoid having materials in contact with the ZnSe window that are outside of this recommended pH range.

CAUTION

Do not break the spectrometer seal and attempt to clean interior surfaces. Breaking the seal will void the warranty.

Collecting a background spectrum

For instructions on collecting a background using the software, refer to the MicroLab PC Software User's Guide.

NOTE

The background measurement should be taken at the same pathlength that the sample will be measured.

CAUTION

To ensure an accurate background spectrum is collected, perform a visual inspection of the ZnSe window surfaces looking for any haze or film present on the windows from measuring the previous sample. If a film is observed, repeat the cleaning procedure on Page 46 until the window surfaces are clear of any residue.

Analyzing samples

Collecting a sample spectrum

To load a liquid sample and collect a spectrum using the DialPath accessory:

- 1 Open the DialPath by rotating the arm (see Figure 14).
- 2 Place a small amount of sample material on the bottom sample mounting window (see Figure 16). The sample mounting window is the 2 mm diameter yellow material held in place by the surrounding metal disk.

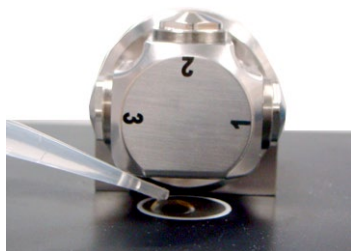


Figure 16. Placing sample on the DialPath accessory sample mounting window.

- 3 Ensure that the sample covers the entire surface area of the sample mounting window.

In the case of a volatile sample, such as with fuels analysis, larger amounts of sample can be applied without concern of leakage or damage to the instrument; however, using the smallest amount of sample possible will ease in the cleaning process.

Although it is safe to run a variety of liquid samples, including aqueous solutions or even thick pastes such as grease, the DialPath is not to be used with any solid or powder samples, such as tablets. Using the DialPath with solid samples will damage the ZnSe windows or modify the pre-set transmission pathlength.

CAUTION

The top and bottom windows are made from ZnSe. ZnSe can be damaged by samples with a pH below 4 and above 9. Only measure samples with a pH between 4 and 9.

- 4 Close the DialPath by rotating the arm clockwise until it clicks into place.
- 5 For instructions on collecting a sample spectrum using the software, refer to the Agilent MicroLab PC Software User's Guide.

Analyzing samples

- 6 After completing the sample measurement, immediately clean the sample from the DialPath using the instructions provided previously. It is important to make sure both the top and bottom windows are free of any residue from the previous sample.

For instructions on reviewing results and handling sample data, refer to the Agilent MicroLab PC Software User's Guide.

Diffuse Reflectance Sampling Accessory

The Diffuse Reflectance sampling accessory provides quantitative and qualitative analysis of powdered or solid samples. This technique provides more sensitivity in a quantitative analysis than ATR, while still being easy to use.

Powdered samples are commonly mixed in KBr powder at a level of approximately 3 to 5%. However, the loading of the sample in KBr can be adjusted to obtain the required sensitivity. The Diffuse Reflectance accessory is optimized for use with the Cary 630 FTIR optical system, providing the greatest sensitivity available for quick, accurate measurements.

The Diffuse Reflectance accessory uses a four-position sample holder (see Figure 17), which slides into a slot on the right side of the accessory. The first position of the holder contains a diffuse reflectance gold mirror, which is used for background collection. The other three positions are used for filling with KBr/sample mixtures. Detents in the holder allow you to gauge which position is being measured.



Figure 17. Diffuse Reflectance sample holder.

Analyzing samples



Figure 18. Diffuse Reflectance accessory attached to Cary 630 FTIR. The sample holder pushes into the slot on the lower right of the accessory.

Cleaning

To clean the Diffuse Reflectance accessory sample holder in preparation for analysis:

- 1 Remove the sample holder from the accessory and dispose of the KBr/sample mixture accordingly.
- 2 Remove all residual dust with a swab or appropriate device.

CAUTION

Do not scratch the diffuse gold reflectance mirror.

CAUTION

Do not contaminate the gold mirror with water, as it will hold moisture and be evident in your spectral response.

CAUTION

Do not break the spectrometer seal and attempt to clean interior surfaces. Breaking the seal will void the warranty.

Collecting a background spectrum

To collect a background spectrum using the Diffuse Reflectance accessory:

NOTE

Since the sample holder contains three sample positions, it may be convenient to place KBr sample mixtures into Positions 2 to 4, and measure the background, immediately followed by each sample.

- 1 Insert the sample holder into the Diffuse Reflectance accessory.
- 2 Collect a background spectrum using the diffuse gold mirror. For software instructions for collecting a background, refer to the MicroLab PC Software User's Guide.

NOTE

On occasion, it may be necessary to use the KBr in pure form as the background material. This is especially true if the KBr contains any moisture.

CAUTION

To ensure an accurate background spectrum is collected, perform a visual inspection of the diffuse gold mirror surface looking for any haze, film or major scratches present on the mirror from use or cleaning. If any of these are observed, a new reference mirror may be needed.

Collecting a background spectrum

To measure a solid material using the Diffuse Reflectance accessory:

- 1 Sample preparation is key to achieving acceptable diffuse reflectance spectra. Mix the sample with KBr at a 3 to 5% concentration level. This is typically done with a mortar and pestle to achieve a homogenous sample.
- 2 Transfer the samples to the appropriate holder position. Do not contaminate other positions.
- 3 Completely fill the position and then use a spatula or flat sided tool to create a flat surface and remove any overfill.
- 4 Slide the holder into the Diffuse Reflectance accessory, and move the holder to the desired sample position.
- 5 Collect a sample spectrum using an appropriate method. For software instructions on collecting a sample spectrum, refer to the Agilent MicroLab PC Software User's Guide.

Analyzing samples

- 6 After completing the sample measurement, immediately clean the sample from the holder using the instructions on Page 50. It is important to make sure that all sample positions are free of any residue from the previous sample.

For instructions on reviewing results and handling sample data, refer to the Agilent MicroLab PC Software User's Guide.

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Maintenance

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Cleaning

When the outside of your Agilent Cary 630 FTIR system needs cleaning, disconnect the power cord and any other connections.

All cleaning should be done with a soft cloth. If necessary, this cloth can be dampened with water, ethanol, or a mild detergent. Do not use abrasive cleaning agents. Avoid use of chlorinated and/or aggressive solvents.

Any spills should be wiped up immediately.

Do not immerse the Cary 630 FTIR in water.

Replacing the Desiccant

The desiccant in the Cary 630 FTIR is a disposable cartridge (see Figure 19), which can be replaced by the user when required.



Figure 19. Old desiccant cartridge (left) and new desiccant cartridge (right).

Maintenance

There are two humidity indicators— one at the back of the spectrometer (see Figure 20), and one on the bottom where the desiccant cartridge is accessed (see Figure 21). There are three levels on the back humidity indicator: 10, 20 and 30%. The level is determined by viewing the indicator and determining which section is a 'lavender' (rather than pink or blue) color. New instruments will come with an updated desiccant cartridge (see Figure 22), which has just the color indicator instead of the % and color indicator. Changing the desiccant is recommended once the humidity level has reached 30% or the color indicator is pink.



Figure 20. Humidity indicator on rear of Cary 630 FTIR (left), and close-up (right).

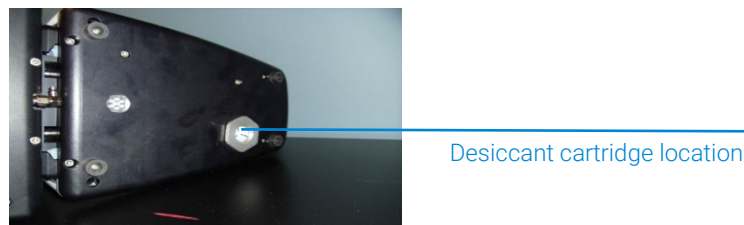


Figure 21. Desiccant cartridge location on bottom of Cary 630 FTIR.

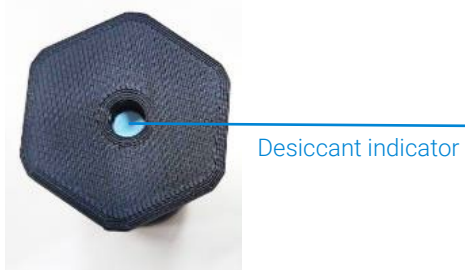


Figure 22. New desiccant cartridge design.

To replace the desiccant:

- 1 Remove the desiccant from the sealed bag.
- 2 Turn off the spectrometer.
- 3 Turn the spectrometer onto its side.
- 4 Use a wrench to remove the spent desiccant cartridge.

Maintenance

- 5 Replace the desiccant cartridge with a new one. Firmly tighten by hand, but do not over-tighten.
- 6 Dispose of the old cartridge appropriately.

NOTE

After the main unit has been exposed and opened to the environment, allow the system to sufficiently warm up and equilibrate prior to use. This will depend on how long the system was open, and can take 30 minutes to 2 hours.

Replacing Windows

Each Cary 630 FTIR spectrometer and sampling accessory has a window that seals the system shut (except for the Transmission sampling accessory). The exit window of the main unit is dependent on the version of the system (KBr or ZnSe), as well as the type of ATR sampling accessory. All other sampling accessories use the ZnSe window, due to the optical components of the accessories. The windows on the sampling accessories and main unit are the same shape and size.



Figure 23. Optical windows.

It may be necessary, sometime within the lifespan of your Cary 630 FTIR to change the window. This could happen for a variety of reasons.

To replace the window:

- 1 Remove the sampling accessory, as outlined on Page 22.
- 3 The window is kept in place by a locking ring device. Position the locking ring extraction tool supplied with the replacement window so that the pins are in the corresponding recessed areas. Turn the tool counterclockwise to remove the locking ring.
- 3 Remove the window.

CAUTION

Handle windows only by the edge. Best practice is to wear disposable gloves when installing windows, in order to prevent inadvertent fingerprints on the window surface.

- 4 Ensure that the o-ring is still intact, and remove any debris from the window placement area.
- 5 Position the new window. The locking ring has a one degree angle to it. So once the ring is in place, turn it a quarter turn counterclockwise and then begin to tighten by turning clockwise. The ring is seated when it is slightly recessed from the front plate. This ensures that the accessories will gently slide into place. Do not over tighten.

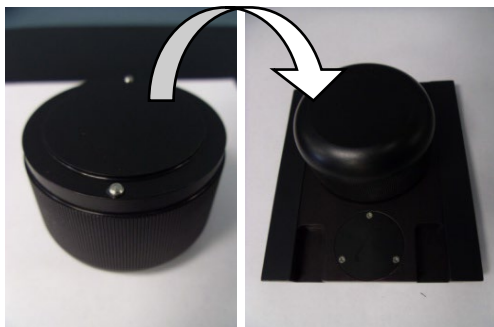


Figure 24. Window replacement tool and replacing locking ring.

Replacing the Source

The typical lifetime of the infrared source is three years. So it may need to be replaced on occasion. You should contact your local service representative if you are not sure whether or not the source should be replaced.

NOTE

To perform this procedure, you will need:

- 3/32 ball driver
- Phillips screw driver

Maintenance

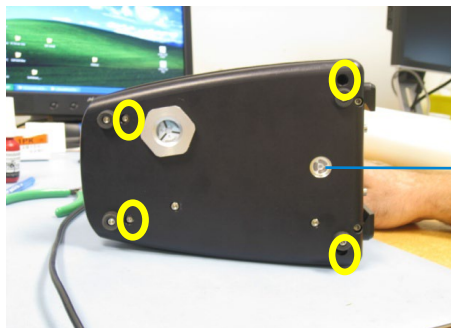
Before starting this procedure, turn off your Cary 630 FTIR. Unplug the system from the main power source and from the USB connection to the computer. Also, remove any sampling accessory from the Cary 630 FTIR so you are left with just the main unit.

CAUTION

Removing the sampling accessory will expose the ZnSe or KBr optical windows. Avoid touching or placing any object near the optical windows.

To replace the source:

- 1 Tilt the spectrometer on its side to expose the bottom plate (see Figure 25). There are two visible screws; and two deep, recessed screws securing the instrument. Using the 3/32 ball driver, remove all four screws.



Desiccant cartridge

Figure 25. Bottom of Cary 630 FTIR, showing screws to remove (circled).

NOTE

All four screws are captive and will not completely disengage. Be careful not to mistake the screws that hold the foot placements as correct screws for removing the base plate.

- 2 Return the instrument to its upright position, and remove the cover.

Maintenance

- 3 Disengage the power harness from the instrument. The power harness is identifiable by its 4-pin connector and the blue, black and red wires (see Figure 26).

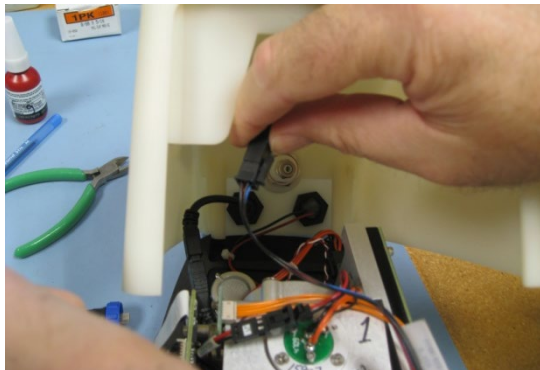


Figure 26. Disengaging the power harness.

- 4 Disconnect the USB cable. The USB connector is the black connector (see Figure 27).

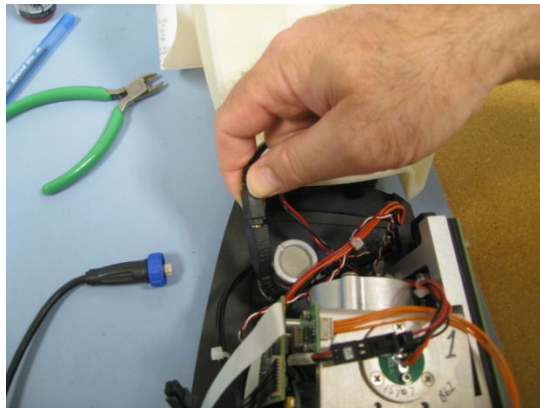


Figure 27. Disconnecting the USB cable.

Maintenance

- 5 Disconnect the laser/source harness from the power board. The laser/source harness is identifiable by its 6-pin connector and orange, black and red wire combination (see Figure 28).

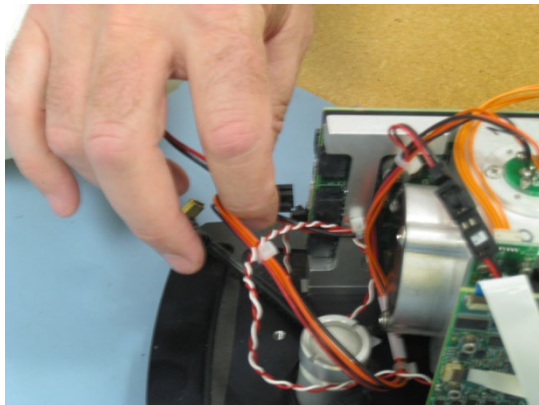


Figure 28. Disconnecting the laser/source power harness.

- 6 Identify the source assembly. The source is located on the top of the interferometer frame. It is secured by three Philips head screws (see Figure 29). Using the appropriate Philips head screwdriver, remove all three screws.

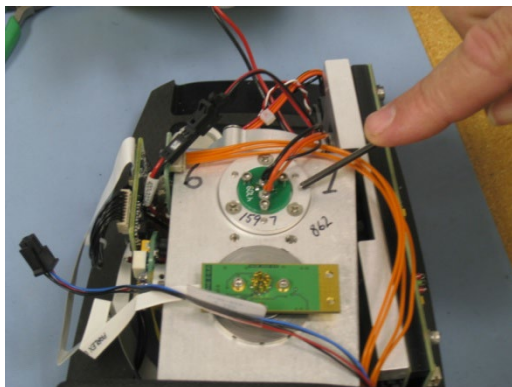


Figure 29. Source assembly in interferometer frame.

- 7 Remove the source assembly from the frame (see Figure 30). Set aside the source and replace it with the new source assembly.

NOTE

For alignment purposes, the opening on the source assembly goes towards the front of the instrument (where the sampling accessory is normally located).

- 8 Attach with the three Philips head screws that were removed previously.

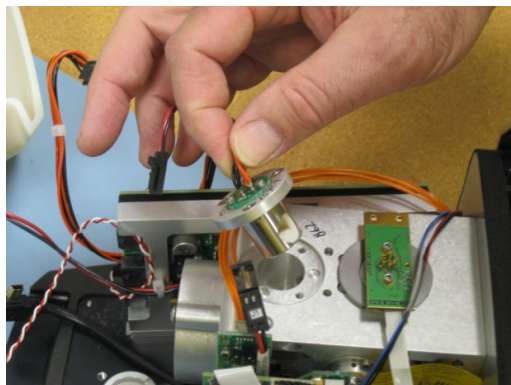


Figure 30. Removing the source assembly.

NOTE

The hole placement pattern has been designed to prevent improper placement of the source.

- 9 Reconnect the laser/source power harness to the proper connector as outlined in the removal process. Then connect the USB cable and the power harness (also outlined previously). Once all of the cables have been reconnected, replace the cover to its original position. Set the instrument on its side and tighten the four base plate retaining screws. Remember, these did not fully disengage and should already be in place.
- 10 Replace your sampling accessory. Reconnect the power and USB external cords, and proceed as normal. You will want to check the energy values in the Diagnostics screen so that the value is correct. You may need to adjust the gain in order to achieve the appropriate amount of signal through the instrument (refer to the Diagnostics values section on Page 19 for proper energy values).

WARNING



Hazard

User servicing of parts and components beyond the procedures outlined in this manual may pose a safety risk to you, damage your instrument, and invalidate the instrument warranty. Perform only those procedures described in this manual. For all other technical problems, contact your authorized Agilent representative.

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Spare parts and Consumables

Table 3 Spare parts

| Part number | Description |
|---------------|--|
| G8043-67483 | Replacement flat tip for ATR |
| G8043-67484 | Replacement pellet tip for ATR |
| G8043-67405 | Polystyrene film for ATR/Transmission sampling accessory |
| G8043-67406 | Polystyrene film for Tumbler/DialPath sampling accessory |
| PIKE-162-5450 | NIST traceable polystyrene from PIKE approximately 38 μm (for IQOQ) |
| 925-0128 | NIST traceable polystyrene from ICL approximately 35 μm (for IQOQ) |
| G8043-67400 | Infrared source with purge fitting |
| G8043-67401 | Desiccant with purge fitting |
| G8043-67402 | KBr window with removal tool |
| G8043-67403 | ZnSe window with removal tool |
| G8043-67003 | Cary FTIR Swivel Diamond ATR Press |
| G8043-67004 | Cary FTIR Swivel Ge-ZnSe Press |
| G8043-67012 | Purge kit for engine and Transmission sampling accessory |
| 430-0135 | 15VDC 2.66A Power Supply |
| 430-0018 | FTIR USB Cable |

For service contracts, repairs and refurbishment services, contact Agilent.

Spare parts and Consumables

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6

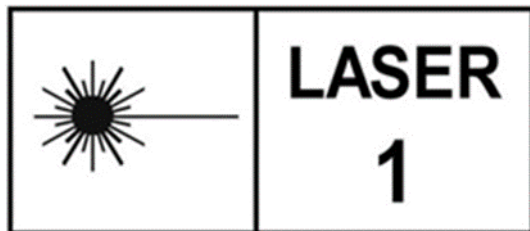
Specifications

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Technical

- **Interferometer geometry:** High-throughput Michelson interferometer with fixed and moving flat mirrors
- **Standard beamsplitter:** KBr or ZnSe
- **Maximum spectral resolution:** 2 cm^{-1}
- **Laser:** Low-powered solid state
- **Infrared source:** Wire-wound element
- **Sampling accessories:** Diamond ATR, Transmission, TumbIIR, DialPath, Diffuse Reflectance, Ge ATR, ZnSe ATR, 10 degree and 45 degrees Specular Reflectance
- **Detector:** 1.3 mm diameter, thermoelectrically-cooled dTGS
- **Width:** 21 cm (8.25 in)
- **Depth:** 10.2 cm (4.00 in)
- **Height:** 17.1 cm (6.75 in)
- **Weight:** 2.9 kg (6.5 lb)
- **Power supply input:** 100–240 V AC, 0.93 A, 50–60 Hz
- Power supply output: 15 V DC, 2.66 A
- Cary 630 requires a power supply of 15 V DC, 17 W

Safety information



The Cary 630 FTIR system contains a low-powered solid state laser required for operation but in no operation or maintenance mode will the operator be exposed to radiation levels that exceed those that define a Class 1 laser product.

Class 1 Laser

Complies with 21 CFR 1040.10 and 1040.11 except for conformance with IEC 60825-1 Ed. 3., as described in Laser Notice No. 56, dated May 8, 2019.

Environmental Conditions

- Temperature (during use): 0–40 °C; -32–104 °F
- Temperature (for storage): -30–60 °C; -22–140 °F
- Humidity non-condensing (during use): ZnSe: Up to 80%, KBr: 20-50%
- Humidity non-condensing (for storage): ZnSe: Up to 40%, KBr: Up to 40%
- Altitude: Up to 2000m
- Pollution Degree 2
- Overvoltage Category II

In This Guide

The guide describes the following:

- Introduction
- Getting Started
- Analyzing Samples
- Maintenance
- Spare parts and Consumables
- Specifications



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Agilent Technologies Australia [M] Pty Ltd
679 Springvale Road
Mulgrave, VIC 3170, Australia

