

Agilent 4500 Series FTIR

Operation Manual



Agilent Technologies

Notices

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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

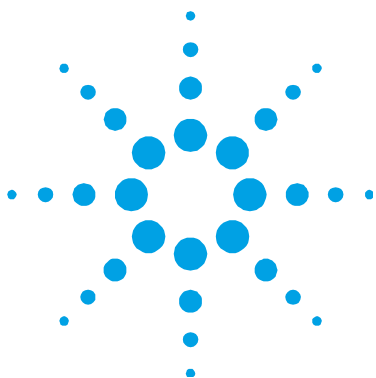
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Contents

1. Introduction	5
2. Getting Started	7
Unpacking	7
Agilent 4500t FTIR packing list	8
Agilent 4500a FTIR packing list	8
Agilent 4500 DialPath FTIR packing list	9
Connecting power	10
Power cord selection	12
Turning on the instrument	13
Power switch LED status	13
Starting the software	14
Performance verification	14
Diagnostic values	15
Performance validation	17
3. Analyzing Samples	19
Lube samples with the Agilent 4500t FTIR	19
Cleaning the TumbIIR accessory	21
Collecting a background spectrum	22
Measuring a sample	22
Agilent 4500t FTIR with a TumbIIR	24
Cleaning the accessory	25
Collecting a background spectrum	26
Measuring a liquid sample	27

Contents

Agilent 4500a FTIR with an ATR	28
Cleaning the accessory	32
Collecting a background spectrum	32
Collecting a sample spectrum	33
Agilent 4500 DialPath FTIR	35
Cleaning the DialPath accessory	37
Collecting a background spectrum	39
Measuring a liquid sample	39
4. Spare Parts	43
5. Specifications and Safety Information	45
Specifications	45
Safety Information	46
Environmental conditions	46
Electromagnetic compatibility	46
EN55011/CISPR11	46
ICES/NMB-001	47



1. Introduction

The Agilent 4500 Series FTIR instruments are small sized, mid-infrared region spectrometer platforms specifically designed for portable, onsite chemical analysis outside the laboratory environment. The systems are small (21.6 x 29.2 x 19.1 cm; 8.5 x 11.5 x 7.5 in), lightweight (6.8 kg; 15 lb) and packaged in a weather-resistant enclosure suitable for outdoor use. They come standard with internal battery power for operation of up to 4 hours or can be operated with 110/220 volt AC power for extended use.

WARNING**Fire Hazard**

4500 Series 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 manual, the protection provided by the equipment may be impaired.

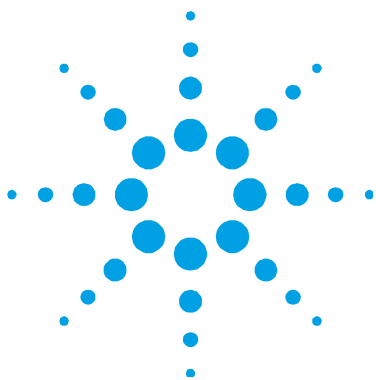
The 4500 Series FTIR instruments use technology known as Fourier transform infrared (FTIR) spectroscopy, which is the current state-of-the-art technique for molecular compound identification and quantification. FTIR uses an infrared (IR) light source to pass through the sample and onto a detector, which 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.

The heart of the Agilent FTIR spectrometer system is a patented Michelson interferometer design mounted on shock-dampening mechanisms to protect the components from shock and vibration in the field. This proprietary design is the key to successfully making FTIR technology compact, lightweight, rugged and field-portable.

The 4500 FTIR system is available in a number of different sampling configurations to accommodate the analysis of a range of liquids, powders, pastes and gels. The optimal use of the various configurations of the 4500 FTIR system are:

- **Agilent 4500t FTIR Tumbler System:** For the rapid chemical analysis of liquids under ambient conditions. The Agilent 4500t FTIR is a Tumbler accessory-based instrument specifically designed for onsite oil analysis. This system is an ideal tool for oil analysis of diesel generators, wind farms, off-shore applications or marine applications.
- **Agilent 4500a FTIR ATR System:** For the chemical analysis of liquids, powders, pastes and gels. The attenuated total reflectance (ATR) system is available in single reflection (most common) or three reflection versions.
- **The Agilent 4500 DialPath FTIR** is for the rapid chemical analysis of liquids under ambient conditions that require multiple pathlengths.

All systems are equipped with a software user interface that is intuitive, simple to use and requires no specialized technical training. With the touch of a button, the system provides valuable information about the identity and amount of chemical substances present in a material.



2. Getting Started

Unpacking	7
Connecting power	10
Turning on the instrument	13
Starting the software	14
Performance verification	14

Unpacking

To unpack your spectrometer system:

- 1 After receiving the Agilent 4500 Series FTIR delivery, do not immediately open the shipping container. Instead, place the shipment 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.
- 2 The shipping enclosure should contain the items listed below. 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 in shipment or damaged.

NOTE

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

-
- 3 Remove the 4500 Series FTIR system from its shipping case and place it on a flat, stable surface. The instrument must be kept away from hot surfaces and any sources of electromagnetic interference.

Locate the PC keyboard and mouse for ergonomically correct access.

Agilent 4500t FTIR packing list

- One Agilent 4500t FTIR mid-IR spectrometer system, including TumbIR sampling accessory
- One Agilent 4500 Series FTIR Operation Manual
- One power cord
- One installation disk
- One shipping container

Options

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

- Laptop computer
- Surfactant kit
- Extra cords

Agilent 4500a FTIR packing list

- One Agilent 4500a FTIR mid-IR spectrometer system, including appropriate sampling accessory
- One Agilent 4500 Series FTIR Operation Manual
- One power cord
- One installation disk
- One shipping container

Options

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

- Laptop computer
- Single-bounce ATR (includes sample press)
- Multi-bounce ATR
- Extra cords

Agilent 4500 DialPath FTIR packing list

- One Agilent 4500 DialPath FTIR mid-IR spectrometer system, including DialPath accessory
- One Agilent 4500 Series FTIR Operation Manual
- One power cord
- One installation disk
- One shipping container

Options

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

- Laptop computer
- Extra cords

Connecting power

To connect power to the spectrometer system:

- 1 Before connecting the power cord to the power source, remove the connector cap and then insert the power cord female connector provided into the male power plug located on the left side of the spectrometer system.
- 2 Connect the other end of the power cord to the power source. The appropriate power supply for your location will be provided with the system. When the system is not in field use, keep it plugged in to the local AC power to ensure a fully-charged battery pack (see Figure 1).

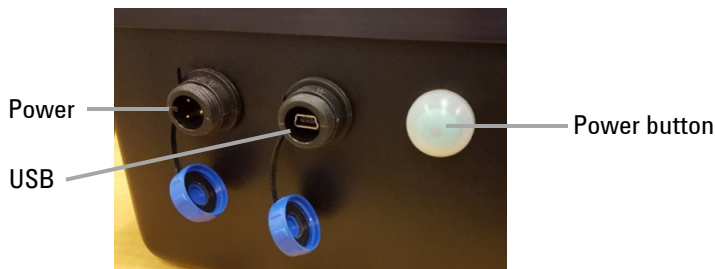


Figure 1. Attaching the power cord

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.

Once AC power is connected, the power button located next to the power and USB connectors on the left side of the 4500 FTIR, will appear a solid red color. This means the battery pack is being charged, but the instrument is off.

NOTE

The 4500t FTIR contains a Molicel lithium-ion rechargeable battery pack. Agilent have fully charged the 4500t system prior to shipment, but the system should also be plugged into a power outlet when not in use. The 4500t battery life under continuous use is approximately 3.5 hours. It is recommended to allow the battery pack to be re-charged at initial setup prior to turning on the power to the instrument.

Molicel lithium-ion rechargeable battery pack specifications:

- Capacity (nominal): 6600 mAh
- Nominal voltage: 11.1 V
- Energy: 73.2 Wh
- Size: 149.5 x 89.3 x 20 mm
(5.8 x 3.5 x 0.8 in)
- Weight: 480 g (1.1 lb)

Battery operating specifications:

- Operating voltage: 12.6 to 9.0 V
- Charge voltage: 12.6 V
- Cutoff voltage: 7.5 V
- Temperature range
 - Discharge: -20 to 60 °C (-4 to 140 °F)
 - Charge: 0 to 45 °C (32 to 113 °F)
- Maximum discharge current: 3.5 A
(continuous)
- Maximum charge current: 3.5 A

Power cord selection

The following power plugs can be used:

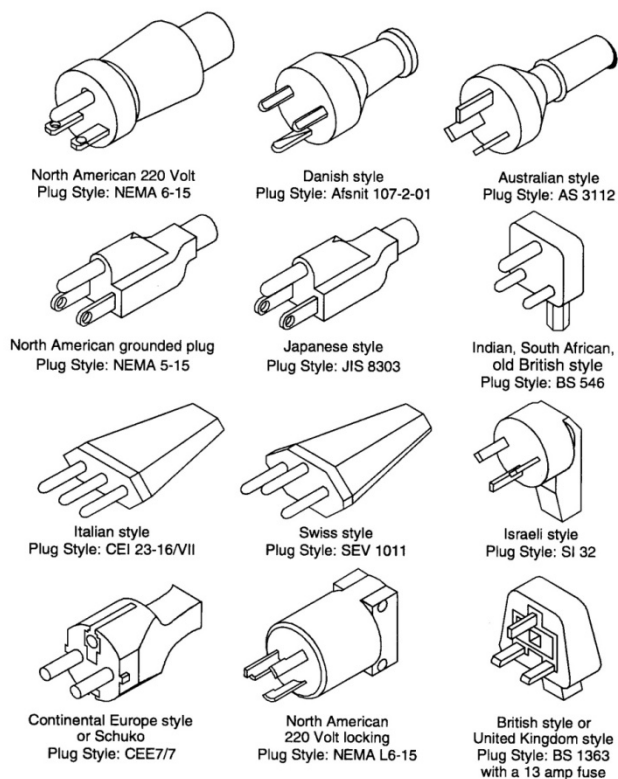


Figure 2. Suitable power cords

When replacing the mains power cord, use only a mains power cord with a rating equivalent to the one supplied with the instrument.

Turning on the instrument

To turn on the instrument:

- 1 Press the power button of the 4500 Series FTIR system and hold for two (2) seconds. Initially the light will blink between red and green while the firmware is being loaded. This process should take less than 30 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 then 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%
Low battery	Green	50% (0.5 s on / 0.5 s off)
Critically low battery	Red	50% (0.5 s on / 0.5 s off)
Dead battery	No LED illumination	N/A
Firmware update	Red/green	Green blinks rapidly twice then red blinks rapidly twice

CAUTION

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

Starting the software

For detailed instructions on initial software logon and user management, refer to the MicroLab Software Operation Manual.

For additional instructions on adding a user to the system and other initial software setup procedures, refer to the MicroLab Software Operation Manual.

Performance verification

The 4500 Series FTIR spectrometer systems have 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 instrument is running properly. To run and interpret the performance test, refer to the MicroLab Software Operation Manual. The performance test measures the energy level of the instrument (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 now ready to analyze a sample. A yellow or red circle indicates the instrument is operating outside of the factory-defined parameters. These parameters are listed 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 4500 Series FTIR instrument is performing properly before using the system to make critical measurements. The instrument 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 instrument's function. If the instrument is not functioning properly, one of the diagnostic values will be out of specification. Typically, the instrument will not allow data collection if the diagnostic values are out of specification. Performance validation tests are longer, more involved tests that measure how well the instrument is working. The tests listed under 'Performance validation' check the instrument'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. The instrument will not collect data if the diagnostic values are grossly out of specification, but it is good practice to verify that the instrument is working properly. Performance validation should be run on a quarterly or bi-annual basis. Highly-regulated industries may require the performance validation to be run monthly, depending on the use of the instrument. The performance validation verifies the key aspects to the instrument's ability to measure good data. If the performance validation tests are within specification, the instrument should provide data that works well with methods developed for the 4500 Series FTIR spectrometer. As with any instrument, however, results can always be verified by running a known sample with the sample specific method.

Diagnostic values

The following values can also be viewed on the Diagnostics page in the MicroLab PC software. These values can be used to determine if the instrument is working properly. They can be used on a daily basis to determine that the instrument is operating as intended. In each case, the Optimal value indicates that the system is running at its intended performance level. The Marginal value indicates that the instrument is still functioning, but at a lower performance level. The Critical value indicates that the system is not working correctly. Contact Agilent technical support for assistance if there is a problem with your instrument.

Table 2. Diagnostic values

Value	Optimal (green)	Marginal (yellow)	Critical (red)	Comment
Energy (centerburst)	26,000–21,000	>30,000 or <15,000	>31,000 or <10,000	Indicates the overall alignment and proper gain adjustment for the system.
Battery life	AC or >30 min	30–5 min	<5 min	
Source	1.9 A	>2.2 or <1.6	>2.5 or <1.0	Indicates a problem with the 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	35–44	<35 or >44	<30 or >48	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.

Performance validation

The MicroLab PC software has three tests appropriate for performance validation of the instrument. All of these tests can be accessed from the Advanced Features, System Check page of the MicroLab PC software. The 4500 Series FTIR system should be warmed up for at least 30 minutes before conducting any of these tests. These tests can be run on a quarterly, bi-annual or annual basis to determine that the instrument is performing within its specification.

Performance (signal-to-noise) test

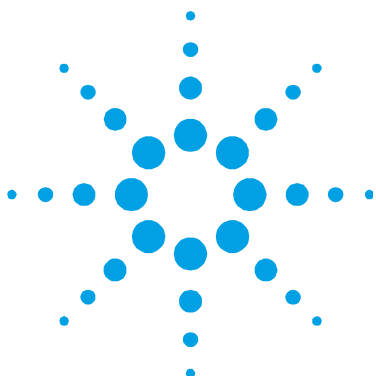
This test measures the signal-to-noise level at two regions of the IR spectrum: 2500 cm^{-1} and 1000 cm^{-1} . The signal-to-noise is defined as the reciprocal of the root mean squared (RMS) noise in the defined region for a blank sample measured with a blank background. Both the background and sample are measured at 4 cm^{-1} resolution with a one minute collection time. This test takes 2 minutes per test. The user can specify a number of tests to be conducted. 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: 3000 cm^{-1} and 1000 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. 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. The polystyrene test sample must be pressed firmly against the diamond sample interface during the sample collection.



3. Analyzing Samples

Lube samples with the Agilent 4500t FTIR	19
Agilent 4500t FTIR with a TumbIIR	24
Agilent 4500a FTIR with an ATR	28
Agilent 4500 DialPath FTIR	35

Lubrication samples with the Agilent 4500t FTIR

The Agilent 4500t FTIR instrument is provided with a dedicated oil and lubrication sampling interface called the TumbIIR. The TumbIIR is a proprietary liquid transmission sampling accessory designed and manufactured exclusively by Agilent to optimize the analysis of lubrication samples. When comparing the TumbIIR to traditional liquid cells for general analytical laboratories, the TumbIIR makes it much easier to prepare, load, analyze and clean the sample.

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

In the sample loading and cleaning mode, the TumbIIR is rotated so the accessory window is facing up (see Figure 3). 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.

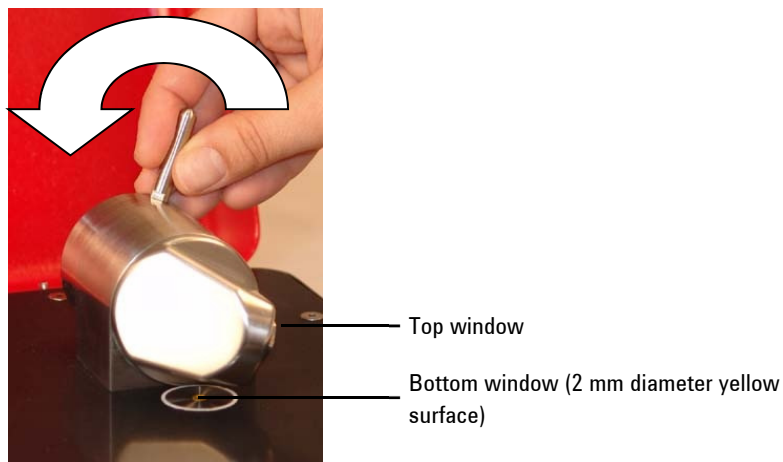


Figure 3. TumbIIR with accessory window facing up

In the sample analysis mode, the TumbIIR is rotated such that the accessory optical window is facing down towards the 4500t FTIR sample mounting area. In this position, infrared (IR) energy passes through the sample making measurement possible. Be sure to rotate the TumbIIR arm completely around until it reaches the detent and snaps into position. This detent keeps the accessory stationary during sample measurement (see Figure 4).

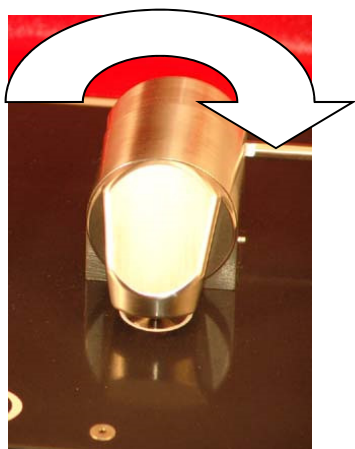


Figure 4. TumbIIR with accessory window facing down

When in the sample analysis mode, the TumbIR pathlength is 100 micrometers, an optimal pathlength for mid-IR analysis of lubrication samples. The alignment of the accessory is pre-set at the factory and so there is no adjustment required.

The steps involved with analyzing a lubrication sample with the 4500t FTIR are:

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

Cleaning the TumbIR accessory

To clean the TumbIR accessory:

- 1 Rotate the TumbIR arm so the optical sensor is pointing upward.
- 2 Clean the top window first, then clean the bottom mounting window, indicated in Figure 3.

NOTE

Only use soft cotton cloth, such as found on cotton swabs or a suitable substitute to clean the optical sensor and sampling area.

CAUTION

The windows are made of an IR-transmitting material called zinc selenide (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, such as Kimwipes. 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

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.

For detailed software instructions for collecting a background, refer to the MicroLab Software Operation Manual.

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 procedures above until the window surfaces are clear of any residue.

Measuring a sample

To apply a lubrication sample to the system:

- 1 Open the sampling device (TumblIR) by rotating the arm counterclockwise (see Figure 3).
- 2 The bottom window should be visible at this point. Place a small amount of material on the sample window located on the TumblIR base plate. The bottom 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 (see Figure 5).



Figure 5. Applying a sample to the bottom window on the Tumbler

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 lubrication samples, including aqueous solutions or even thick pastes such as grease, the Tumbler is not to be used with any solid samples, such as hard graphite lubricant. Use of the Tumbler with solid samples will damage the ZnSe windows.

CAUTION

The sample mounting window and accessory window 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 5 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.

CAUTION

Do not open your system and attempt to clean interior surfaces. Opening your system will void the warranty.

- 4 Close the device by rotating the arm clockwise until it clicks into place (see Figure 4).
- 5 Click the **NEXT** button on the software screen to proceed with the analysis as shown below.
- 6 For detailed software instructions for measuring a sample, refer to the MicroLab Software Operation Manual.
- 7 After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided above. It is important to make sure both the sample mounting window and the accessory window are free of any residue from the previous sample.
- 8 For detailed software instructions on reviewing results and handling sample data, refer to the MicroLab Software Operation Manual.

Agilent 4500t FTIR with a TumbIIR

The Agilent 4500t FTIR instrument can be provided with a dedicated sampling interface called the TumbIIR accessory. The TumbIIR is a proprietary liquid transmission sampling accessory designed and manufactured exclusively by Agilent to optimize the analysis of many liquid, paste or gel samples. When comparing the TumbIIR to traditional liquid cells for general analytical laboratories, the TumbIIR makes it much easier to prepare, load, analyze and clean the sample.

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

In the sample loading and cleaning mode, the TumbIIR is rotated so the accessory window is facing up (see Figure 3). 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.

In the sample analysis mode, the TumbIIR is rotated such that the accessory optical window is facing down towards the 4500t FTIR sample mounting area. In this position, infrared (IR) energy passes through the sample making measurement possible. Be sure to rotate the TumbIIR arm completely around until it reaches the detent and snaps into position. This detent keeps the accessory stationary during sample measurement (see Figure 4).

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

The steps involved with analyzing a sample on the 4500t FTIR system with a TumbIIR are:

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

Cleaning the accessory

To clean the TumbIIR accessory:

- 1 Rotate the TumbIIR arm so the optical sensor is pointing upward.
- 2 Clean the top window first, then clean the bottom window, indicated in Figure 3.

NOTE

Only use soft cotton cloth, such as found on cotton swabs or a suitable substitute to clean the optical sensor and sampling area.

CAUTION

The windows are made of an IR-transmitting material called zinc selenide (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, such as Kimwipes. 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

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.

For detailed software instructions for collecting a background, refer to the MicroLab Software Operation Manual.

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 above until the window surfaces are clear of any residue.

Measuring a liquid sample

To apply a liquid sample to the system:

- 1 Open the sampling device (TumblIR) by rotating the arm counterclockwise (see Figure 3).
- 2 The bottom window should be visible at this point. Place a small amount of material on the bottom window located on the TumblIR 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 (see Figure 5).

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 TumblIR is not to be used with any solid or powder samples, such as tablets. Use of the TumblIR 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 5 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.

CAUTION

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

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

- 4 Close the device by rotating the arm clockwise until it clicks into place (see Figure 4).
- 5 Click the **NEXT** button on the software screen to proceed with the analysis.
- 6 For detailed software instructions for measuring a sample, refer to the MicroLab Software Operation Manual.
- 7 After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided above. It is important to make sure both the top and bottom windows are free of any residue from the previous sample.
- 8 For detailed software instructions on reviewing results and handling sample data, refer to the MicroLab Software Operation Manual.

Agilent 4500a FTIR with an ATR

The Agilent 4500a FTIR instrument can be provided with a dedicated sampling interface called the Attenuated Total Reflectance (ATR) accessory. The ATR 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 (IR) 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 4500a FTIR system. This small and consistent pathlength provides the advantage of no sample preparation required to get good measurement results on a variety of samples. The key to obtaining good results with an ATR accessory is making good contact between the sample and ATR crystal of the sampling device. The ATR technique can be used for analysis of liquids, pastes, powders and even some solid samples.

All Agilent ATR accessories use a type IIa diamond crystal as the interface between the sample and the IR energy. The diamond provides the advantage of extreme hardness as well as chemical resistivity. Diamond can accept samples with a pH range from 1 to 14, which means hard or abrasive samples and even strong acids can be safely analyzed. Agilent offers two choices of ATR sampling accessories: a single reflection or triple reflection DuraDisk system. The diamond is the world's most durable substance and the ZnSe substrate is used for maximum IR signal.

The single reflection ATR is most suitable for higher absorbing samples such as rubber, polymers, paints and fibers. Powder and solid samples are best measured on the single reflection ATR as well due to the sample press device that can apply high pressure to powder and solid samples to ensure good contact with the diamond sampling surface. 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 micron depth of penetration for IR energy at 1700 cm^{-1} . The single reflection ATR protrudes slightly above the metal mounting plate.

The triple reflection ATR is most suitable for liquid samples with lower IR absorption properties. The triple reflection ATR has a 2 mm diameter sampling surface with 200 μm active area and provides approximately 6 micron effective penetration depth for IR energy at 1700 cm^{-1} . The triple reflection ATR is mounted flush with the metal mounting plate and does not operate with a sample press device.

CAUTION

Although diamond is a very hard material, the ATR window is relatively thin 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 the use of the sample press on samples that may be sharp or pointed.

To open the sample press:

- 1 Pull the bottom spring loaded locking pin away from the sample press body.
- 2 Pull the sample press up as far as it can go.

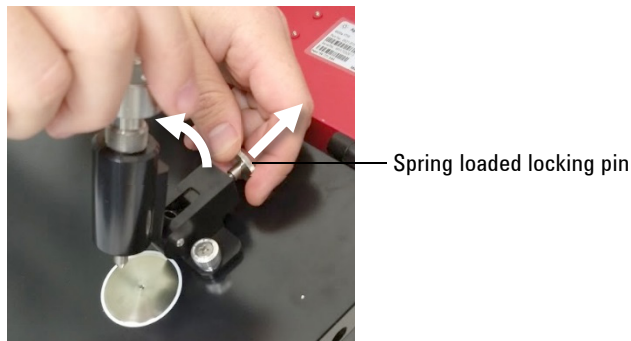


Figure 6. Positioning the sample press

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 window sampling surface (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.

NOTE

Only the single reflection ATR version has a sample press device.

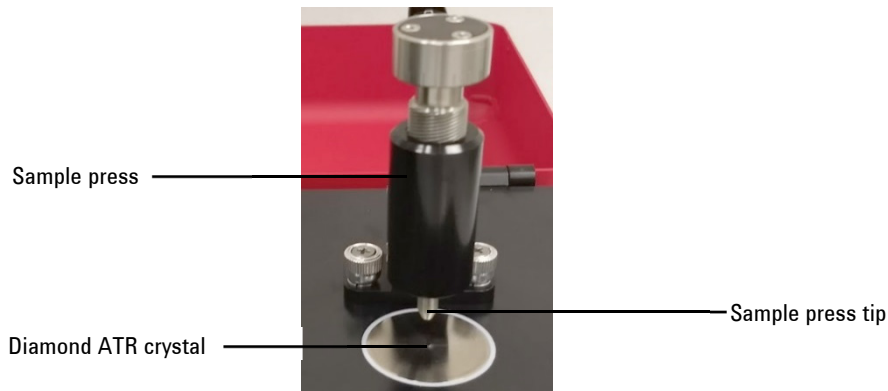


Figure 7. Sample press in highest position providing easy access for sample loading and cleaning

In the sample analysis mode, the ATR sample press is lowered such that the sample press tip is in contact with the sample. In this position, contact is made between the sample and the IR energy emitting from the diamond ATR window (see Figure 8).

NOTE

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. Only liquid or paste samples should be used with the triple reflection ATR accessory.



Figure 8. Sample press lowered for analysis

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.

The steps involved with analyzing a sample with the 4500a FTIR and an ATR are:

- 1 Clean the ATR sample mounting window.
- 2 Collect a background spectrum.
- 3 Collect a sample spectrum.

Cleaning the accessory

To clean the ATR accessory:

- 1 Open the ATR sample press arm by lifting back until the sample press tip reaches the top of its travel.
- 2 Clean the sample press tip first, then clean the sample mounting window, indicated in Figure 7.

NOTE

Kimwipes or a suitable substitute (such as cotton swabs) should be used to clean the sensor and sampling area. Clean the sample interface and press with a suitable solvent such as acetone, methanol, ethanol or isopropyl alcohol.

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 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.

For detailed software instructions for collecting a background, refer to the MicroLab Software Operation Manual.

CAUTION

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

Collecting a sample spectrum

To load an ATR sample into the system:

- 1 Open the sample press by twisting the knurled nut until the sample press tip is elevated slightly from the diamond ATR surface (see Figure 9).



Figure 9. Opening the sample press

- 2 The diamond sampling window should be visible at this point. Place a small amount of material to be measured on the diamond ATR 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.

CAUTION

The sample mounting window and accessory window are made from type IIa synthetic diamond, which is extremely chemically-resistant. However, it is still possible the diamond 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 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 the use of the sample press on samples that may be sharp or pointed.

CAUTION

Do not open your system and attempt to clean interior surfaces. Opening your system will void the warranty.

- 4 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.

NOTE

Only liquid or paste samples should be used with the triple reflection ATR accessory.

If the sample is a powder or solid, the sample press must be in contact with the sample. To make contact, twist the knurled nut until it reaches the detent (snap-in) position. The spring-loaded sample press tip is set to apply 15 pounds of force assuming a 0.03 in sample thickness.

- 5 Click the **NEXT** button on the software screen to proceed with the analysis.

- 6 For detailed software instructions for measuring a sample, refer to the MicroLab Software Operation Manual.
- 7 After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided above. It is important to make sure both the sample mounting window and the sample press tip (for single reflection only) are free of any residue from the previous sample.
- 8 For detailed software instructions on reviewing results and handling sample data, refer to the MicroLab Software Operation Manual.

Agilent 4500 DialPath FTIR

The Agilent 4500 DialPath FTIR instrument is provided with a dedicated sampling interface (the DialPath), which is based on the same technology as the Tumbler accessory. The DialPath uses the Tumbler's patented liquid transmission sampling accessory, designed and manufactured exclusively by Agilent, to optimize the analysis of many liquid, paste or gel samples. When compared to traditional liquid cells for general analytical laboratories, the DialPath makes it much easier to prepare, load, analyze and clean the sample, while giving the user 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 accessory window is facing up (see Figure 10). It is in this position that the sample mounting area can be easily accessed for loading the sample into place and cleaning the sampling surfaces before the next sample is analyzed.

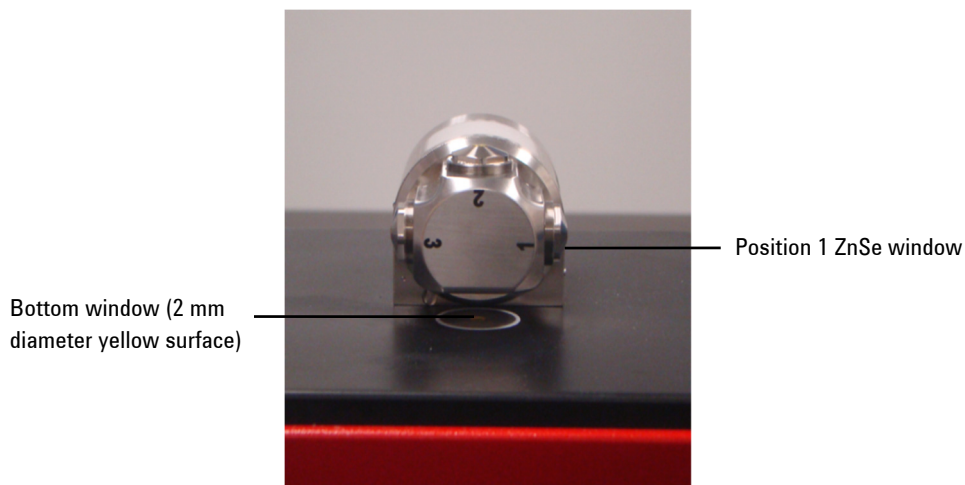


Figure 10. DialPath with accessory window facing up

In sample analysis mode, the DialPath is rotated such that the accessory optical window is facing down towards the 4500 DialPath FTIR sample mounting area. In this position, infrared (IR) energy passes through the sample, making measurement possible. Make sure that you rotate the DialPath arm completely around until it reaches the detent and snaps or clicks into position. This detent keeps the accessory stationary during sample measurement (see Figure 11).

NOTE

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

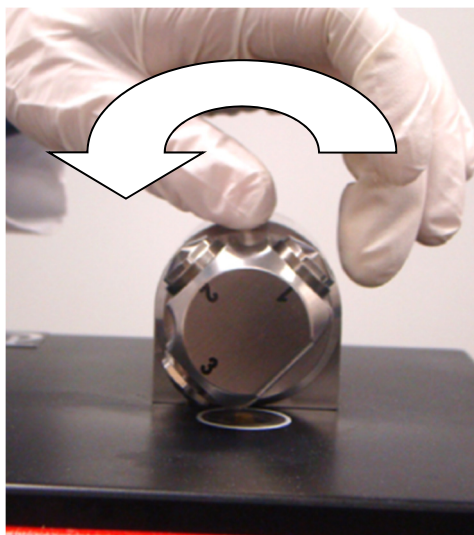


Figure 11. Rotating the DialPath so that the accessory optical window is facing down

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

The steps involved with analyzing a sample with the 4500 DialPath FTIR are:

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

Cleaning the DialPath accessory

Rotate the DialPath so that optical sensor #2 is pointing upward. Clean the window of the position used first, then clean the bottom window, indicated in Figure 10. If using Position 2, you will find it necessary to clean either Position 3 or 1, depending on the direction the DialPath was rotated in.

NOTE

Only use soft cotton cloth, such as that found on cotton swabs, or a suitable substitute to clean the optical sensor and sampling area.

CAUTION

The windows are made of an IR-transmitting material called zinc selenide (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 such as Kimwipes is used. Materials such as cotton swabs dipped in acetone are recommended for cleaning (see Figure 12).

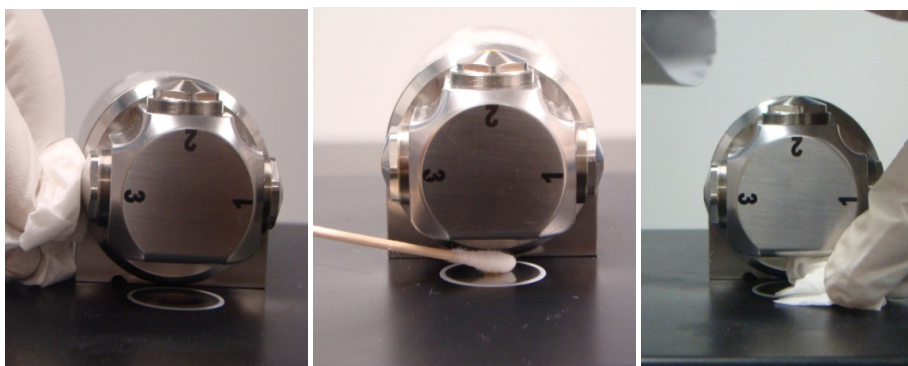


Figure 12. Cleaning the Position 3 window and sample substrate

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 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

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.

For detailed software instructions for collecting a background, refer to the MicroLab Software Operation Manual.

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 above until the window surfaces are clear of any residue.

Measuring a liquid sample

To apply a liquid sample to the system:

- 1 Open the sampling device (DialPath) by rotating the arm (see Figure 10).
- 2 The bottom window should be visible. Place a small amount of material on the bottom window located on the DialPath base plate. The sample window is the 2 mm diameter yellow material held in place by the surrounding metal disk.
- 3 Ensure the sample covers the entire surface area of the bottom window (see Figure 13).

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 possible amount of sample will ease in the cleaning process.



Figure 13. Applying a sample to the DialPath bottom window

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. Use of 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.

CAUTION

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

CAUTION

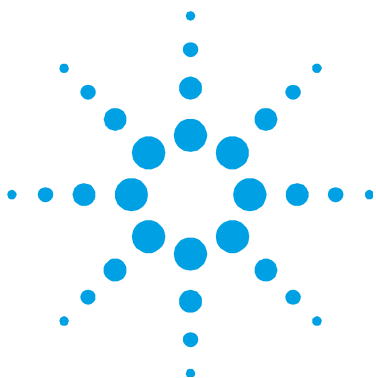
Do not break the spectrometer seal and attempt to clean interior surfaces. Breaking the seal will void the warranty.

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

- 4** Close the device by rotating the arm clockwise until it clicks into place (see Figure 11).
- 5** Click the **NEXT** button on the software screen to proceed with the analysis.
- 6** For detailed software instructions for measuring a sample, refer to the MicroLab Software Operation Manual.
- 7** After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided above. It is important to make sure both the top and bottom windows are free of any residue from the previous sample as well as adjacent windows to minimize any cross-contamination issues.
- 8** For detailed software instructions on reviewing results and handling sample data, refer to MicroLab Software Operation Manual.

For additional instructions on editing of methods and reviewing data using the MicroLab PC Software, refer to the MicroLab Software Operation Manual.

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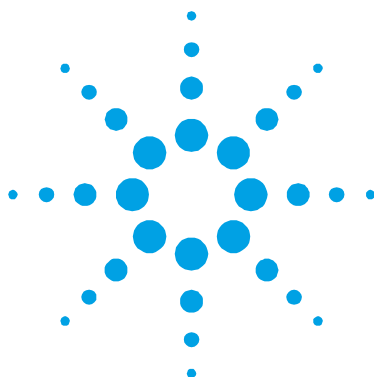
4. Spare Parts

Part number	Description
430 - 0001	Agilent 4500 Series FTIR power supply
430 - 0018	Agilent 4500 Series FTIR USB cable

For sampling accessories, service contracts, repair and refurbishment services, contact Agilent.

Spare Parts

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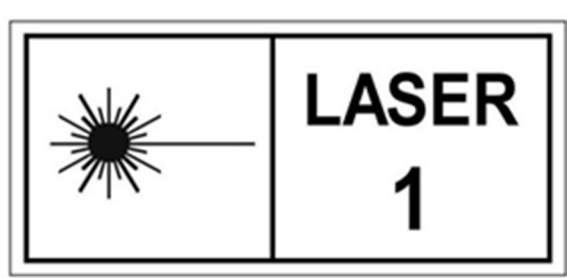
5. Specifications and Safety Information

Specifications	45
Safety Information	46
Environmental conditions	46
Electromagnetic compatibility	46

Specifications

- **Interferometer geometry:** High-throughput Michelson interferometer with fixed and moving flat mirrors
- **Standard beamsplitter:** Zinc selenide
- **Maximum spectral resolution:** 4 cm^{-1}
- **Laser:** Low-powered solid state
- **Infrared source:** Wire-wound element
- **Spectral range:** $4000\text{ to }650\text{ cm}^{-1}$
- **Detector:** 1.3 mm diameter, thermoelectrically-cooled dTGS
- **Power supply:** 100 to 240 V AC, 3 A, 50 to 60 Hz

Safety Information



Agilent 4500 Series FTIR systems contain 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 Product

Complies with 21 CFR 1040.10 and 1040.11 except for deviations pursuant to Laser Notice No. 50, dated June 24, 2007 CDRH Accession No. 0321242 IEC 60825-1:2014.

Environmental conditions

- **Operating Temperature:** 0 to 50 °C; 32 to 122 °F (during use)
- **Storage Temperature:** Minus 30 to 60 °C; -22 to 140 °F (storage)
- **Humidity:** Up to 95%, non-condensing
- **Altitude:** Up to 2000 m

Electromagnetic compatibility

EN55011/CISPR11

Group 1 ISM equipment: group 1 contains all ISM equipment in which there is intentionally generated and/or used conductively coupled radio- frequency energy which is necessary for the internal functioning of the equipment itself.

Class A equipment is equipment suitable for use in all establishments other than domestic and those directly connected to a low voltage power supply network which supplies buildings used for domestic purposes.

This device complies with the requirements of CISPR11, Group 1, Class A as radiation professional equipment. Therefore, there may be potential difficulties in ensuring electromagnetic compatibility in other environments, due to conducted as well as radiated disturbances.

Operation is subject to the following two conditions:

- 1 This device may not cause harmful interference.
- 2 This device must accept any interference received, including interference that may cause undesired operation.

If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

- 1 Relocate the radio or antenna.
- 2 Move the device away from the radio or television.
- 3 Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
- 4 Make sure that all peripheral devices are also certified.
- 5 Make sure that appropriate cables are used to connect the device to peripheral equipment
- 6 Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.

Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

ICES/NMB-001

This ISM device complies with Canadian ICES- 001.

Cet appareil ISM est conforme à la norme NMB-001 du Canada.

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In This Guide

The guide describes the following:

- Introduction
- Getting Started
- Analyzing Samples
- Spare Parts
- Specifications and Safety Information

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