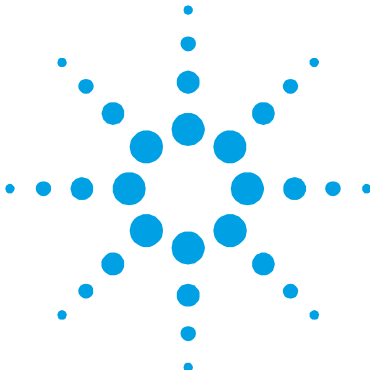

Agilent 5500 Series FTIR

Operation Manual

Agilent Technologies

Notices

Manual Part Number

0020-401

Edition

Fourth edition, March 2012

Printed in USA

Agilent Technologies, Inc.

Errata Statement

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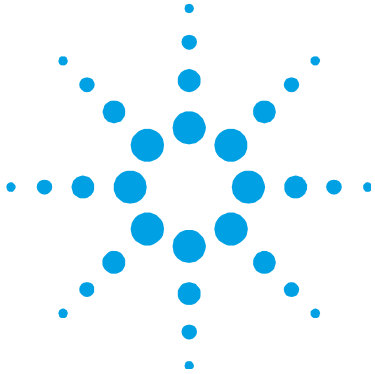
CAUTION

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.

Contents



1. Introduction

The Agilent 5500 Series FTIR instruments are small sized, mid-infrared region spectrometer platforms specifically designed to provide the capabilities of much larger, traditional FTIR spectrometers, but without the complexity, maintenance requirements or cost. The instrument dimensions are only 20.3 x 20.3 x 11.4 centimeters (8.0 x 8.0 x 4.5 in) high and it is very lightweight (3.6 kg; 8 lb). Although the hardware platforms are in many ways the same, the Agilent 5500t FTIR system is designed specifically for onsite oil analysis, and the Agilent 5500a FTIR system is designed for sample analysis in challenging, multi-user environments. Both platforms provide versatility for use in a traditional analytical chemistry laboratory, a temporary field laboratory or even in the field. However, it is primarily intended for indoor use and is not designed for water-resistance or other extreme outdoor conditions.

WARNING

Fire Hazard

5500 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 5500 Series FTIR systems 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, rugged Michelson interferometer, field-proven for onsite and mobile use. This proprietary design is the key to successfully making FTIR technology compact, lightweight, rugged and field-portable.

The 5500 Series FTIR instruments are controlled using an external computer running a Microsoft® Windows® XP operating system. The computer must have at least a Pentium® IV 3 GHz processor with 1 GB of memory and at least 40 GB hard disk capacity. The laptop computer is offered as an option, which provides the flexibility of supplying it yourself or purchasing from Agilent.

The 5500a FTIR system is available in a number of different sampling configurations to accommodate the analysis of a range of liquids, powders, gases, pastes and gels. The optimal use of the various configurations of the 5500a FTIR system are:

- **Agilent 5500a FTIR Gas Cell System**: For the chemical analysis of gas samples under ambient conditions.
- **Agilent 5500a FTIR TumblIR System**: For the rapid chemical analysis of liquids under ambient conditions.
- **Agilent 5500a 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), three reflection and nine reflection versions. The nine reflection version has an optional flowcell configuration.

The 5500t FTIR system is a TumblIR liquid cell 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.

Both 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.

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2. Getting Started

Unpacking

To unpack your spectrometer system:

1. After receiving the Agilent 5500 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 5500 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.

Agilent 5500t FTIR packing list

- One Agilent 5500t FTIR mid-IR spectrometer system, including TumblIR sampling accessory
- One Agilent 5500 Series FTIR Operation Manual
- One Agilent MicroLab Software Operation Manual
- One power cord
- One USB cable
- One installation disk (needed as backup copy only)
- One shipping container

Options

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

- Dell laptop computer
- Surfactant kit
- Extra cords

Agilent 5500a FTIR packing list

- One Agilent 5500a FTIR mid-IR spectrometer system, including appropriate sampling accessory
- One Agilent 5500 Series FTIR Operation Manual
- One Agilent MicroLab Software Operation Manual
- One power cord
- One USB cable
- One installation disk (for backup use only)
- One shipping container

Options

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

- Dell laptop computer
- Single bounce ATR (includes sample press)
- 3 or 9 bounce ATR
- Gas cell option (15 and 50 mm versions)
- Extra cords

To convert your 5500a FTIR system to a different accessory, contact Agilent.

Connecting power

To connect power to the spectrometer system:

1 Insert the provided power cord into the power connector located on the lower left side at the rear of the 5500 Series FTIR system (see Figure 1).

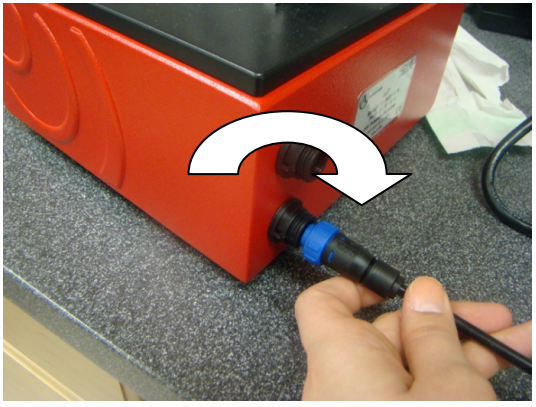

Figure 1. Attaching the power cord

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.

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.

Power cord selection

The following power cords can be used:

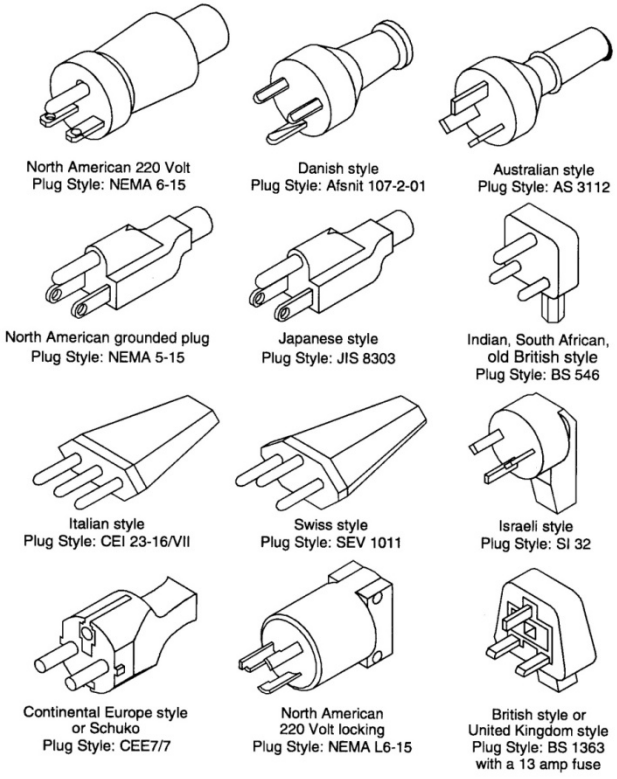

Figure 2. Suitable power cords

Connecting the computer

To connect the instrument to the computer:

1 Insert the provided USB cable into the USB connector located on the upper left side at the rear of the 5500 Series FTIR system (see Figure 3).

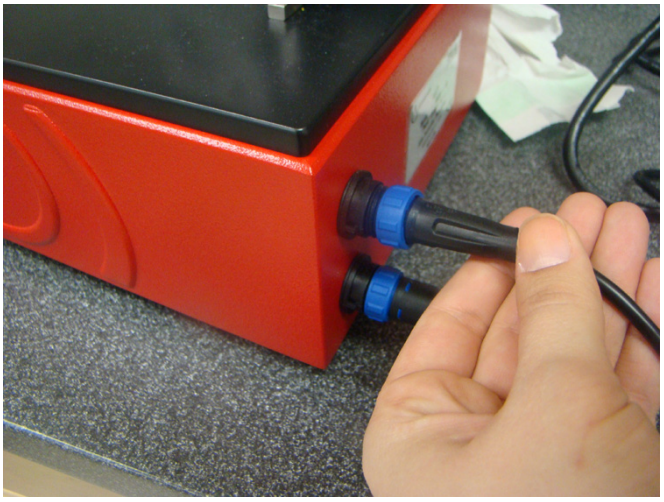

Figure 3. Attaching the USB cable

2 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.

3 Connect the opposite end of the USB cable to the computer that will run MicroLab PC software.

NOTE The 5500t FTIR must be directly connected to the computer USB slot and NOT through a USB hub or adapter.

Turning on the instrument

Press the green power button located on the front of the 5500 Series FTIR system and hold for two seconds. The LED light will illuminate green when the system is activated.

Press the power button to turn off the 5500 Series FTIR system — the light will turn red. During a firmware update, the light will turn red and blink.

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.

Power switch LED

The power button contains a two-colored LED. The color displayed on the LED can indicate the state of the system. Table 1 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

Starting the software

To start the software:

1. Ensure that the system is active, then double-click the **MicroLab PC** icon.
2. The system must have a warmup period of 5 minutes, before analysis may begin.

Logging on to MicroLab PC for the first time

The 5500 Series FTIR systems are supplied with the Agilent MicroLab PC software, which has already been installed on your external laptop computer.

In addition, the system is provided with a shortcut to the MicroLab PC software in the Start menu.

Upon initial power up of the spectrometer system, the MicroLab PC software application splash screen briefly appears, showing the version, copyright and trademark information. The software will launch and display a logon screen.

When the logon screen appears, the user name will be 'Admin' – when logging onto the system for the first time, the software automatically initiates in an Administrator mode. The default password for initial logon to MicroLab PC software is 'admin'.

NOTE The user name and password are case-sensitive.

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, plus editing methods and reviewing data using MicroLab PC software, refer to the MicroLab Software Operation Manual.

Performance verification

The 5500 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 5500 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 5500 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 from 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)	29,000–21,000	>29,000 or <21,000	> 31,000 or < 18,000	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	12,000–4,000	>12,000 or <4,000	>15,000 or < 3,000	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 5500 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. For 45 degree external reflectance and grazing angle reflectance measurements, the appropriate reflective reference cap should be in place for the entire test. No reference cap is needed when using the ATR sample interface. 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. When using the 45 degree external reflectance and grazing angle reflectance interfaces, the polystyrene sample should be placed between the reference cap and the sample interface. For the ATR sample interface, the reference cap is not required for a background, but the polystyrene test sample must be pressed firmly against the diamond sample interface during the sample collection.

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3. Analyzing Samples

Lube samples with the Agilent 5500t FTIR

The Agilent 5500t FTIR instrument is provided with a dedicated oil and lubrication sampling interface called the TumblIR. The TumblIR is a proprietary liquid transmission sampling accessory designed and manufactured exclusively by Agilent to optimize the analysis of lubrication samples. When comparing the TumblIR 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 accessory window is facing up (see Figure 4). 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.

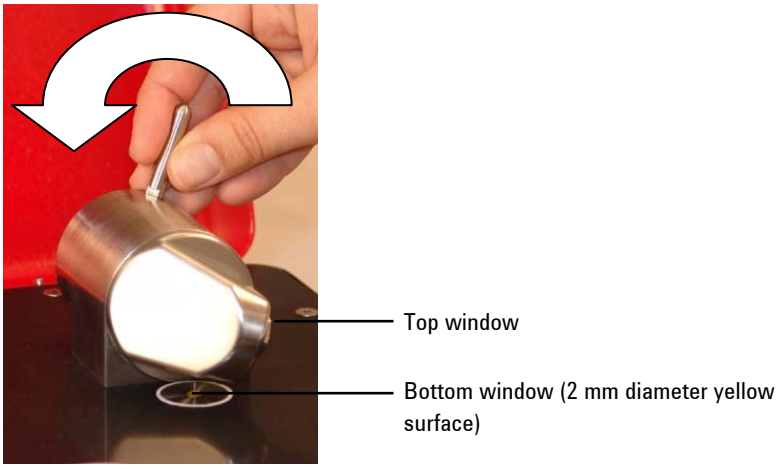

Figure 4. TumblIR with accessory window facing up

In the sample analysis mode, the TumblIR is rotated such that the accessory optical window is facing down towards the 5500t FTIR sample mounting area. In this position, infrared (IR) energy passes through the sample making measurement possible. Be sure to rotate the TumblIR arm completely around until it reaches the detent and snaps into position. This detent keeps the accessory stationary during sample measurement (see Figure 5).

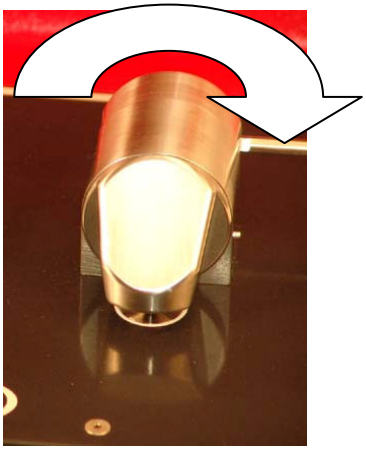

Figure 5. TumblIR with accessory window facing down

When in the sample analysis mode, the TumblIR 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 lube sample with the 5500t FTIR are:

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

Cleaning the TumblIR accessory

To clean the TumblIR accessory:

1. Rotate the TumblIR arm so the optical sensor is pointing upward.
2. Clean the top window first, then clean the bottom mounting window, indicated in Figure 4.

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 lube sample to the system:

1. Open the sampling device (TumblIR) by rotating the arm counterclockwise (see Figure 4).
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 6).

Figure 6. Applying a sample to the bottom window on the TumblIR

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 TumblIR is not to be used with any solid samples, such as hard graphite lubricant. Use of the TumblIR 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 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 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 5).

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 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.

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

Agilent 5500a FTIR with a TumblIR

The Agilent 5500a FTIR instrument can be provided with a dedicated sampling interface called the TumblIR accessory. The TumblIR 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 TumblIR 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 accessory window is facing up (see Figure 4). 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 TumblIR is rotated such that the accessory optical window is facing down towards the 5500a FTIR sample mounting area. In this position, infrared (IR) energy passes through the sample making measurement possible. Be sure to rotate the TumblIR arm completely around until it reaches the detent and snaps into position. This detent keeps the accessory stationary during sample measurement (see Figure 5).

When in the sample analysis mode, the TumblIR 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 5500a FTIR system with a TumblIR are:

1 Clean the sample windows.

2 Collect a background spectrum.

3 Collect a sample spectrum.

Cleaning the accessory

To clean the TumblIR accessory:

1 Rotate the TumblIR arm so the optical sensor is pointing upward.

2 Clean the top window first, then clean the bottom window, indicated in Figure 4.

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 4).
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 6).

   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 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 TumblIR for a range of liquid sample types. Because the TumblIR 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 5).

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.

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

Agilent 5500a FTIR with an ATR

The Agilent 5500a 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 5500a 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 three choices of ATR sampling accessories: a single reflection, triple reflection and nine reflection DuraDisk system. All Agilent ATR disks use a patented diamond and zinc selenide (ZnSe) composite design. 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.

The nine reflection ATR is most suitable for the most demanding liquid samples with the lowest IR absorption properties. The nine reflection ATR has the largest sampling surface (6 mm diameter) with 2 mm active area and provides approximately 12 microns of effective penetration depth for IR energy at 1700 cm^{-1}. The nine reflection ATR is slightly recessed into the mounting plate and does not operate with a sample press device. It is, however, designed to accommodate an optional flow cell attachment.

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 the use of the sample press on samples that may be sharp or pointed.

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.

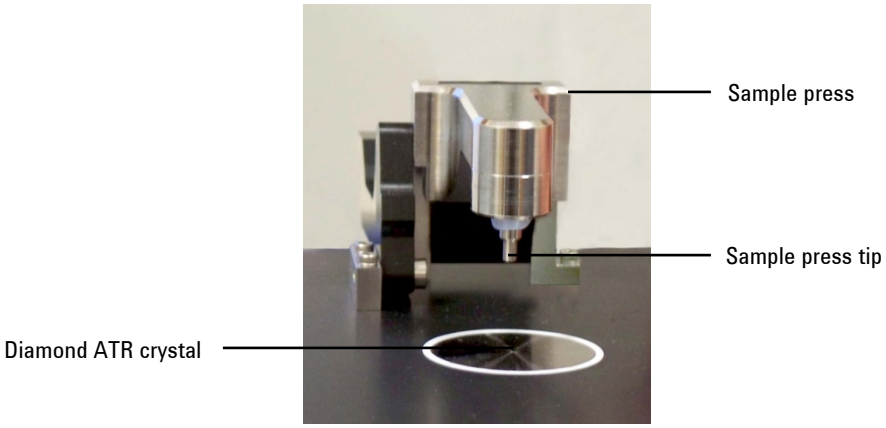

Figure 7. Sample press in highest position providing easy access for sample loading and cleaning

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 and nine 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 5500a 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 moving the arm back as far as it can go, so that 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 and nine reflection ATR accessory.

If the sample is a powder or solid, the sample press must be in contact with the sample. To make contact, pull the sample press arm forward 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 inch 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.

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

Nine reflection ATR flowcell attachment

The Agilent nine reflection ATR accessory is provided with a flowcell attachment (see Figure 10). The flowcell is not required for operation of the nine reflection ATR, however, it can be useful in certain applications. The most common use of this attachment is for measuring volatile samples. The flowcell assembly when used in combination with the Luer Lock caps, completely isolates the sample from the ambient environment.

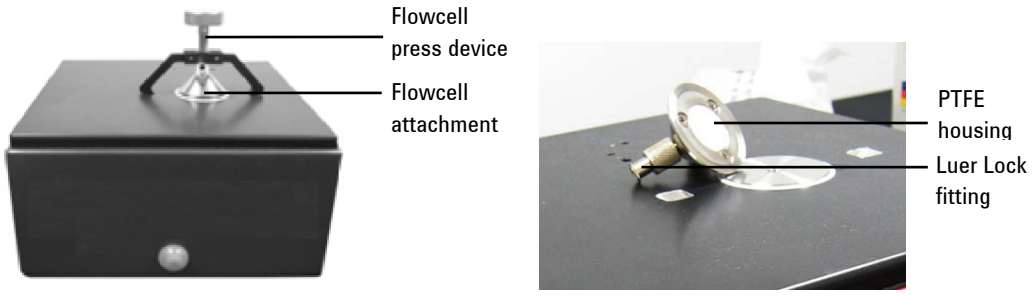

Figure 10. Nine reflection ATR flowcell attachment

In addition, it can be used to reduce sample handling time by flowing large volumes or multiple samples through the ATR using a low-pressure pumping system, such as a peristaltic pump (provided by the user). The flowcell assembly consists of the flowcell press device, main housing, Luer Lock fittings (2) and Luer Lock caps (2). The flowcell outside housing and fittings are made from 316 stainless steel and the inner housing in contact with the sample is PTFE, both of which have very high chemical resistivity (see Figure 10). This means any liquid sample you would consider using with the ATR can also be used with the flowcell. Instructions for both uses of the flowcell are provided below.

Volatile samples

To use the flowcell to measure a volatile sample:

1. Follow the instructions on Page 36 on ‘Cleaning the accessory’ and ‘Collecting a background spectrum’.
2. Quickly load a small amount of sample onto the nine reflection diamond ATR surface (the clear, circular shaped material held in place by the surrounding metal disk). Ensure that the sample covers the entire surface area of the diamond crystal; however, using the smallest amount of sample possible will ease in the cleaning process.
3. Once the sample is loaded onto the ATR, immediately place the flowcell housing on top of the ATR, aligning the ATR cavity with the circular PTFE protrusion on the bottom of the inner housing.

4 Rotate the thumbscrew of the flowcell press device counterclockwise to provide clearance for attachment to the housing. Attach the flowcell press device to the 5500a FTIR baseplate and flowcell housing. Insert the two fingers at the bottom of the arms of the press into the slots of the 5500a FTIR baseplate. With one hand, align the tip of the screw to the center of the housing counterbore. With the other hand, tighten the screw clockwise until the screw tip engages with the flowcell housing (see Figure 11). Continue further until the press device is finger-tight (typically no more than a half turn is required once intial contact is made with the housing). Be careful not to over-tighten, or damage could occur to the press assembly.

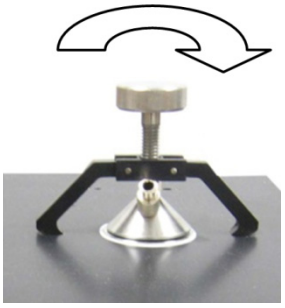

Figure 11. Securing the flowcell press device

You are now ready to proceed with the analysis.

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

Large sample volumes

To use the flowcell to reduce the sample handling time when measuring a large volume of sample:

1. Follow the instructions on Page 36 on ‘Cleaning the accessory’ and ‘Collecting a background spectrum’.
2. The flowcell should be installed first, and then the sample is loaded into the flowcell using Luer Lock syringes (provided by the user). To install the flowcell, rotate the thumbscrew of the flowcell press device counterclockwise to provide clearance for attachment to the housing. Attach the flowcell press device to the 5500a FTIR baseplate and flowcell housing. Insert the two fingers at the bottom of the arms of the press into the slots of the 5500a FTIR baseplate. With one hand, align the tip of the screw to the center of the housing counterbore. With the other hand, tighten the screw clockwise until the screw tip engages with the flowcell housing (see Figure 11). Continue further until the press device is finger-tight (typically no more than a half turn is required). Be careful not to over-tighten, or damage could occur to the press assembly.
3. It is recommended, but not required, that two (2) Luer Lock syringes are used to load the sample into the flowcell. One syringe is filled with the sample and the second syringe is empty and is used to draw the sample into the flowcell creating a vacuum suction condition. This procedure reduces the internal pressure in the flowcell and minimizes the risk of leakage of the sample. Be sure to inject enough sample to ensure it reaches and covers the entire surface area of the diamond crystal.

   You are now ready to proceed with the analysis.

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

4 After completing the sample measurement, immediately clean the sample from the flowcell attachment by using a solvent injected into the flowcell using syringes. Again, it can be helpful to have two syringes and to agitate the solvent back and forth within the flowcell for the best cleaning process.

5 Once the flowcell is clean, repeat Steps 3 and 4 until all of the sample has been analyzed.

Multiple samples

To use the flowcell to reduce the sample handling time when measuring multiple samples:

1 Follow the instructions on Page 36 on 'Cleaning the accessory' and 'Collecting a background spectrum'.

2 The flowcell should be installed first, and then the sample is loaded into the flowcell using a low-pressure pumping system (provided by the user). To install the flowcell, rotate the thumbscrew of the flowcell press device counterclockwise to provide clearance for attachment to the housing. Attach the flowcell press device to the 5500a FTIR baseplate and flowcell housing. Insert the two fingers at the bottom of the arms of the press into the slots of the 5500a FTIR baseplate. With one hand, align the tip of the screw to the center of the housing counterbore. With the other hand, tighten the screw clockwise until the screw tip engages with the flowcell housing (see Figure 11). Continue further until the press device is finger-tight (typically no more than a half turn is required). Be careful not to over-tighten, or damage could occur to the press assembly.

3 Once the flowcell is installed, you are ready to pump the sample into the flowcell. It is recommended, but not required, that a peristaltic-style pumping system is used. Whatever system is used, it should not exceed 10 psi pressure on the flowcell to ensure proper operation and to avoid possible leakage from the flowcell. Be sure to pump in enough sample to ensure it reaches and covers the entire surface area of the diamond crystal. It is also recommended that the measurement be done in a stop flow situation. Measuring the sample in a dynamic condition can create measurement errors due to the possible presence of air pockets and other anomalies during the pumping process.

You are now ready to proceed with the analysis.

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

4 After completing the sample measurement, immediately clean the sample from the flowcell attachment by using a solvent injected into the flowcell using syringes or by automatically pumping a solvent into the flowcell. Again, it can be helpful to agitate the solvent back and forth within the flowcell for the best cleaning process.

5 Once the flowcell is clean, repeat Steps 3 and 4 until all of the samples have been analyzed.

Agilent 5500a FTIR with a gas cell

The Agilent 5500a FTIR instrument can be provided with a dedicated gas cell sampling interface for analyzing gases. The gas cell is a proprietary Agilent product designed for ease of operation and maintenance. The cell is supplied with zinc selenide (ZnSe) optical windows, which allow gases with water vapor to be analyzed without danger of damage to the gas cell or its windows.

Agilent offers two choices of gas cell sampling accessories: a 15 cm pathlength gas cell and a 50 cm pathlength gas cell. These cells are customer-interchangeable and can be purchased together or separately. Contact Agilent for details.

Initial setup

The 5500a FTIR system comes with the gas cell accessory already installed on the spectrometer.

To prepare the gas cell for analyzing a gas sample:

1 Remove the gas cell housing and inspect the gas cell sampling interface for cleanliness.

2 Using a pressurized hose, clean the sampling interface (see Figure 12). The immediate area should be free of all debris and dust.

Figure 12. Cleaning the sample interface

3 Reattach the gas cell using the arrows and lock indicators. Rotate the gas cell clockwise until the cell is locked in place (see Figure 13).

Figure 13. Attaching the gas cell

4 Thread the purge gas hose through the 1/8 inch Swagelock inlet fitting and tighten it onto the gas cell inlet (shown in Figure 14 on the right side of gas cell). It is recommended to connect any purge or sample gas to the inlet connector so that the gas flow is from the bottom of the cell moving upwards. Do not hook up the inlet gas line backwards.

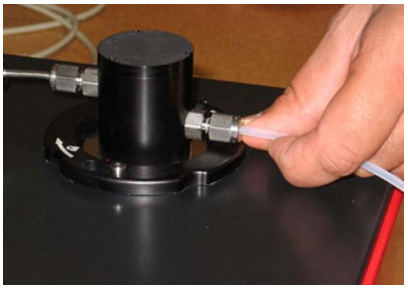

Figure 14. Attaching the purge gas hose to the gas cell inlet

5 Purge the cell using dry air or nitrogen gas. Collect a background spectrum. The gas cell is now ready for use with a real sample. For detailed software instructions on collecting a background, refer to the MicroLab Software Operation Manual.

   It is recommended to purge the gas cell with an appropriate background reference gas for taking the background spectrum.

6 To analyze a gas sample, connect the sample supply line to the gas cell inlet. It is recommended that a valving system is used for the inlet and the exhaust outlet to control and stop the flow during measurement. Having a valving system to switch between background and sample gas will avoid the requirement of frequently connecting the hoses to the gas cell inlet. Gas inlet and outlet plumbing is the responsibility of the customer.

7 When starting the flow of the sample gas into the gas cell, immediately open any outlet exhaust valving to aid in the flow of the sample into the cell. Ensure sufficient time has passed to completely fill the gas cell volume with the sample gas.

8 Once the sample gas has filled the cell, it is recommended that you stop the continuous flow of the gas before starting the data collection measurement (stop flow mode).

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

10 For detailed software instructions for measuring a sample, refer to the MicroLab Software Operation Manual.

For detailed software instructions on reviewing results and handling sample data, refer to the MicroLab Software Operation Manual.

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

CAUTION If analyzing gas samples that are highly volatile, take appropriate safety precautions. Safely delivering the gas to the gas cell and exhausting the gas from the gas cell are the responsibility of the user.

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.

WARNING

Hazard

The 5500a FTIR gas cell systems are designed and manufactured to withstand a MAXIMUM pressure of 50 psi. Do not supply reference or sample gas pressures above this level. Failure to strictly follow this procedure can result in injury to the user and damage to the equipment.

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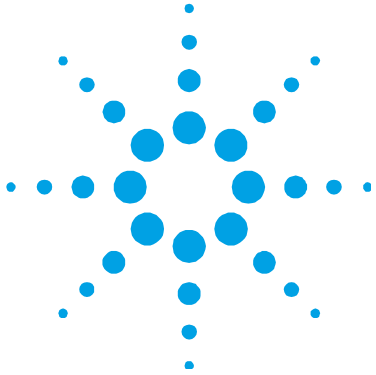

4. Spare Parts

Part number	Description
430 - 0001	Agilent 5500 Series FTIR power supply
430 - 0018	Agilent 5500 Series FTIR USB cable
0020 - 900	Surfactant kit

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

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5. 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
- **Source:** Wire-wound element
- **Spectral range:** 4000 to 650 cm^{-1}
- **Detector:** 1.3 mm diameter, thermoelectrically-cooled dTGS
- **Power supply:** 100/120/240 V AC, 3 A, 50 to 60 Hz
- **Computer:** External Dell laptop Pentium IV 3 GHz processor, Microsoft Windows XP operating system
- **Operating temperature range:** Minus 10 to 50 °C (14 to 122 °F)

WARNING

Laser Hazard

Agilent 5500 Series FTIR systems contain a low-powered solid state laser required for operation. The laser emits radiation and can cause injury to the eye. Do not stare into the beam.

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

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