Agilent

Agilent 4300 Handheld FTIR

Operation Manual

Notices

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Instrument Manufacturing

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Safety Notices

CAUTION

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

WARNING

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

Contents

1 Introduction

The Agilent 4300 Handheld FTIR instrument is a portable, handheld, battery-operated analyzer designed to measure a variety of solid and liquid samples in locations difficult or impossible to analyze in a laboratory. It can be used for quantitative or qualitative analysis of materials.

The system is small, lightweight (2 kg; 4 lb) and packaged in a weather-resistant enclosure suitable for outdoor use. It comes standard with internal battery power for operation up to 4 hours or can be operated with 110/220 volt AC power for extended use.

The instrument comes with a DTGS detector capable of covering the spectral range of 4500 to 650 cm^{-1}. It can be safely used in operating temperatures ranging from 0 to 50° C (32° to 122° F) and in humidity environments as high as 95% for extended periods of time.

NOTE The Agilent 4300 Handheld FTIR with MCT detector, which covers the spectral range from 5000 to 1050 cm^{-1}, and operates from 0 to 40° C (32 to 104 ° F), is no longer available.

WARNING **Fire Hazard**

4300 Handheld 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.

CAUTION **Although the 4300 Handheld FTIR system is designed to withstand significant shock and vibration found in a field environment, use caution when handling and avoid dropping the system when it is out of the shipping case. Dropping the system may result in equipment failure.**

The 4300 Handheld FTIR uses technology known as Fourier transform infrared (FTIR) spectroscopy, which is the current state-of-the-art technique for molecular compound identification and quantification. FTIR uses an infrared (IR) light source to pass through the sample 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.

In addition to the capability to measure samples onsite that in the past were typically measured in the lab, this system is a non-destructive tool (NDT) that allows the analysis to be performed without damaging the sample.

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 4300 Handheld FTIR system can be configured for a number of different sampling configurations to accommodate the analysis of a range of liquids, solids, powders, pastes and gels. The various sampling interfaces are designed to be easily interchangeable, even in the field, and are automatically recognized by the system. Information on the optimal use of the different sampling configurations of the 4300 Handheld system is listed in the following categories.

- Diffuse reflectance sample interface: The diffuse reflectance sampling capability allows the 4300 Handheld FTIR to handle a variety of important applications. Testing has determined good results on a wide variety of samples including artwork, soils, rocks and minerals, composites, rough plastics, fabrics and corrosion on metal surfaces. In general, if the sample reflects little light, the diffuse reflectance interface will be the sampling method of choice. In many cases, the diffuse reflectance provides the easiest to use sample interface for the 4300 Handheld FTIR.
- External reflectance sample interface: The specular reflectance interface allows the analysis of films and coatings on reflective metal surfaces such as aluminum or steel. The angle of incidence is 45 degrees. The infrared energy passes through the film, reflects off the steel, passes back through the film and is collected by the specular reflectance interface. In addition, it can be used for the analysis of smooth, opaque samples where infrared light reflects off the surface.

- Grazing angle sample interface: The grazing angle specular reflectance interface is similar in concept to the specular reflectance interface. It differs in the angle of incidence. The grazing angle interface has an angle of incidence of 82 degrees making it ideal for the analysis of very thin (sub-micron) films. The increased angle of incidence causes more interaction of the infrared energy within the thin sample and has the secondary benefit of increasing the pathlength of the sample. It is ideal for looking at trace contamination on reflective metal surfaces such as cleaning validation studies.
- Diamond ATR sample interface: The ATR interface is ideal for the analysis of solids, liquids, pastes and gels. The interface is comprised of a diamond window, which makes it impervious to corrosion and scratching. The sample is brought into contact with the diamond window. ATR is a surface technique and only the top 2-3 microns are analyzed.
- Ge ATR sample interface: The ATR interface is ideal for the analysis of highly absorbing solids and liquids. The interface is comprised of a germanium crystal. The surface of the sample is analyzed by bringing it into contact with the germanium crystal. Only the top 0.5 to 2 micrometers is measured, making this technique ideal for strongly absorbing samples such as carbon filled elastomers and rubbers.

The 4300 Handheld FTIR is equipped with an integrated CPU as standard and can be interfaced to the optional laptop computer for development of new methods and for detailed viewing and manipulating the spectra. The 4300 Handheld software user interface is intuitive, simple to use and requires no specialized technical training. The spectral display is represented with easy to read status bars and report formats, which increases the ease of use on the smaller handheld PC display. 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 4300 Handheld FTIR delivery, do not immediately open the shipping container. Instead, place the shipping case in a room-temperature environment and allow several hours for the contents of the container to reach the room ambient temperature. This is to avoid unnecessary condensation on the components prior to the initial setup and installation process.
   Your 4300 Handheld FTIR has been packed to ensure that it arrives at your facility ready to go to work. Custom foam inserts protect the instrument from damage during travel. Whenever your instrument is shipped via common carrier, it needs to be re-packed in the shipping case. Using this case, your 4300 Handheld can be removed and re-packed in the case in a matter of seconds, while maintaining instrument security.
2. Confirm that the shipping enclosure contains the standard items listed below, as well as any optional items ordered. Inspect the enclosure closely to make sure all items have been removed from the shipment packaging. Also ensure that all the items in the packing list below have arrived undamaged and are in good working condition. Contact Agilent immediately if any items are found to be missing or damaged in shipment.

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

3 Remove the 4300 Handheld FTIR system from its shipping case and place it on its side on a flat, stable surface.

CAUTION **Keep the instrument away from hot surfaces and any sources of electromagnetic interference.**

If the optional instrument stand was ordered, place the instrument in the stand with the appropriate stage.

Packing list

- One Agilent 4300 Handheld FTIR spectrometer system
- One Retro-Reflector Reference Interface
- One Agilent 4300 Handheld FTIR Operation Manual
- One Agilent 4300 Handheld FTIR AC power cable
- One Agilent 4300 Handheld FTIR external power supply
- One Agilent 4300 Handheld FTIR USB cable
- One Agilent 4300 Handheld FTIR wrist strap
- One Agilent 4300 Handheld FTIR stylus
- One polystyrene reference sample
- Four Li-ion batteries for field use of the 4300 Handheld FTIR
- One battery charger for Li-ion batteries
- One MicroLab Software UFD
- One shipping container

Options

Although the 4300 Handheld FTIR system comes standard as shown above, there are a number of options available, including:

- Laptop computer
- Agilent 4300 Handheld FTIR instrument stand with appropriate stages where necessary
- Diffuse reflectance sample interface
- External reflectance sample interface
- Grazing angle sample interface

- Diamond ATR sample interface
- Ge ATR sample interface

Components

The 4300 Handheld FTIR is designed to measure samples in a handheld configuration. Sample measurement can be started using the handle-mounted trigger or from the integrated CPU touch screen.

Figure 1 Agilent 4300 Handheld FTIR

Connecting power

The 4300 Handheld FTIR can be operated remotely for up to 4 hours using the two 10.8 volt lithium-ion batteries, or indefinitely using the AC power cable when AC power is available. The 4300 Handheld uses a single connector for attaching to AC power. The connector is at the back of the battery compartment.

Battery

The 4300 Handheld FTIR has a door on the front of the handle. The door on the front gives access to the system's two batteries. The 4300 Handheld should arrive with four batteries in the shipping case. All batteries should be fully charged from the factory and ready for use. There is an LCD display on the front of the battery indicating the level of charge. Five bars indicate a full charge.

You can open the front of the battery compartment and swap out a battery while the system is in use. This is known as a hot swap of the battery. This allows you to continually use the system without having to shut the system down, or re-initialize the system after changing a battery. The four batteries allow for approximately 8 hours of usage. Additional spare batteries can be purchased if a longer time is required for field use.

In addition, a battery charger is shipped with the system to allow the user to charge batteries when not in the 4300 Handheld FTIR.

NOTE Batteries can only be charged in the appropriate battery charger. They do not charge in the 4300 Handheld FTIR system.

WARNING **Electrical Shock Hazard**
Avoid shock hazard. Never allow liquid to run into the 4300 Handheld FTIR battery compartment.

To install the battery:

1 Pull directly on the battery compartment door. The door opens to expose the two batteries within (see Figure 2).

Figure 2 Battery compartment door

2 Slide the battery into the compartment with the tab of the battery facing downward and towards the front side of the 4300 Handheld FTIR.

3 Slide the battery towards the back of the compartment until you feel the battery engage. There will be a slight resistance when the battery begins to engage.

4 Continue to slide another eighth of an inch until completely engaged. If the battery is not fully engaged, the system will not operate properly.

5 Insert or replace the second battery as necessary by following the above procedure.

6 Close the battery compartment door.

Depending on temperature and age of the battery, the system should operate for a period of up to four hours on battery power.

To remove the battery from the compartment, open the battery compartment door, then pull on the black plastic tab to slide the battery out of the 4300 Handheld FTIR housing.

Do not store the battery in the 4300 Handheld FTIR unless it is connected to the main power source for operation. Like a laptop PC battery, the 4300 Handheld battery will slowly discharge when left plugged into the system. For use with batteries that have built in protection. For use with Agilent specified spare batteries only.

AC power

The 4300 Handheld FTIR is provided with an AC power cord and external power supply 15 V DC converter. This can be used to operate the 4300 Handheld over extended periods of time. The 4300 Handheld FTIR uses a single connector for line power and a separate USB connector for communication to an external computer. Both connectors are included with the 4300 Handheld FTIR.

To connect AC power to the Agilent 4300 Handheld FTIR:

1 Before connecting the AC power cable to the power source, lift the lower cover to access the power connection (see Figure 3). Next, insert the power supply cable with the blue female connector into the 4300 Handheld FTIR power connector. Thread the blue cap onto the connector by rotating clockwise until finger-tight. To avoid damage to the plastic connector, do not over-tighten. The appropriate power supply cabling is provided with the system.

Figure 3 Power connections

CAUTION **Use only the included 4300 Handheld FTIR power supply. Use of other power supplies may damage the 4300 Handheld FTIR. If necessary, replace the power cord only with a cord equivalent to the one provided with this product. See Chapter 5 for ordering information.**

2 Locate the AC power cord and connect it to the 15 V DC converter box. Then 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.

NOTE When the system is not in field use, place the batteries in the included battery charger to ensure fully charged batteries for field deployment.

CAUTION **Connect the power cord to the spectrometer system first and then to the power outlet and computer to prevent any power startup damage or problems.**

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, depress the power button located on the back of the unit under the integrated CPU screen of 4300 Handheld FTIR (see Figure 4). The light to the left of the button will begin to blink once the unit is turned on.

This will also initiate the software on the CPU.

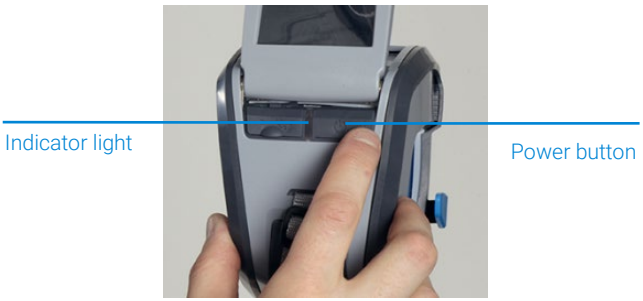

Figure 4 Power button

NOTE The 4300 Handheld FTIR contains two lithium-ion rechargeable battery packs. Agilent has fully charged the 4300 Handheld system prior to shipment, but the batteries should also be placed in the charger when the system is not in use. The 4300 Handheld battery life under continuous use is approximately 4 hours. Allow the battery pack to be recharged at initial setup prior to turning on the power to the instrument.

Power cord selection

The following power cords can be used:

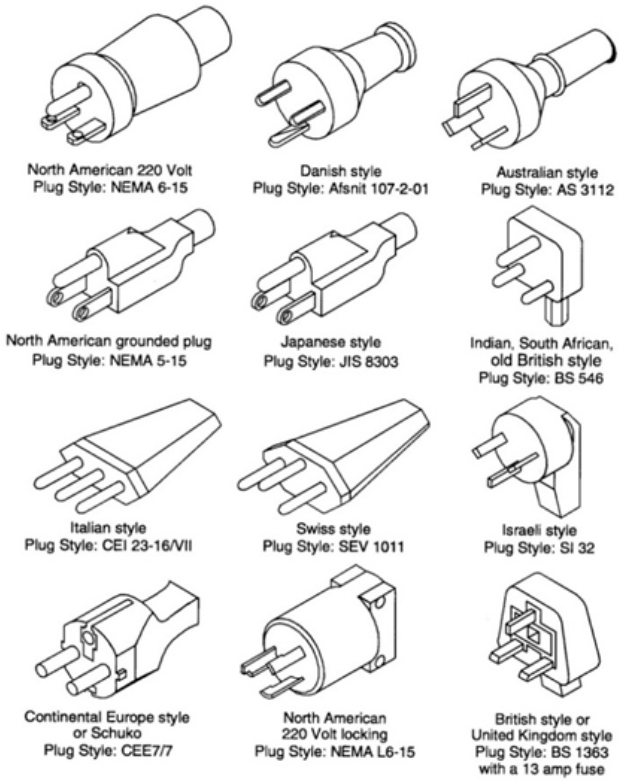

Figure 5 Suitable power cords

Changing the desiccant

The 4300 Handheld FTIR comes with a removable desiccant cartridge.

The cartridge can be removed, and the desiccant replaced with a new desiccant or recharged desiccant. Refer to Section 5 to purchase new desiccant cartridge.

To replace the desiccant:

1 Access the desiccant by opening the back access panel on the 4300 Handheld FTIR. This is located below the power button and USB connector.

2 Remove the desiccant cartridge by moving both locking pins downward as shown in Figure 6 below.

3 The cartridge can then be placed into a lab oven to recharge the silica gel. The recommended temperature is approximately 120°C. Drying time may vary dependent on the amount of moisture. The desiccant should be heated until it returns to the original dark blue color.

4 Once the desiccant is recharged, place the desiccant into the instrument and rotate the locking pins upwards to secure the desiccant cartridge in place.

Figure 6 Desiccant replacement

Turning on the instrument

To turn on the instrument:

1 Press the power button of the 4300 Handheld FTIR system and hold for 2 seconds. Initially, the light will turn orange while the firmware is being loaded. This process should take less than 15 seconds. When the LED light illuminates solid green, the instrument is ready to use.

2 Allow the spectrometer to warm up for a period of 5 minutes for reliable analysis.

When you have finished using the instrument, press and hold the power button for 5 seconds to turn off the system. The LED light will then extinguish.

CAUTION **The power button is a momentary switch to avoid accidental power off. To turn the system on or off, hold the button down for 2 to 5 seconds.**

Starting the software

The 4300 Handheld FTIR systems are supplied with the Agilent MicroLab Mobile software, which has already been installed on your integrated CPU computer. The software will start automatically when the system is turned on. The software will also automatically shut down when the 4300 Handheld FTIR is turned off.

The integrated CPU runs off of the 4300 Handheld FTIR's battery packs, or external power when the system is plugged into an AC power supply. There is no separate power supply for the integrated CPU.

The integrated CPU screen is mounted on a hinge for viewing position optimization. It is recommended that the screen is not rotated beyond the vertical position (approximately 90° from the 'home' position).

CAUTION **The integrated CPU screen rotated beyond the vertical position will result in damage to the flat flex cable connected to the screen.**

Logging on to MicroLab Mobile for the first time

To start the software:

Turn on the 4300 Handheld FTIR by pressing the power button firmly for 2 seconds. See Figure 4 to locate the power button. The system will initialize and start the MicroLab Mobile software.

Upon initial launch of the system, the MicroLab Mobile software automatically loads and a MicroLab application splash screen appears briefly showing software version, copyright and trademark information.

The software will launch and display a logon screen.

NOTE An admin account must be the first account setup on the system. This must be done prior to entering the software for the first time. 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.

Communication to external PC

The USB port for communication to an external PC is located under a cover next to the power button. Insert the provided USB cable into the USB port on the instrument and connect the other end to a PC.

Synchronization and method transfer can be performed using MicroLab Lite on the PC. Analysis can also be performed by the MicroLab PC software on the computer. Refer to the MicroLab Software Operation Manual for more details.

CAUTION

The USB cable must be inserted at a slight upwards angle into the instrument USB connector as shown in Figure 7. Caution needs to be taken when inserting and removing the cable to prevent damage to the USB port.

Always place the instrument on its side when the USB cable is connected.

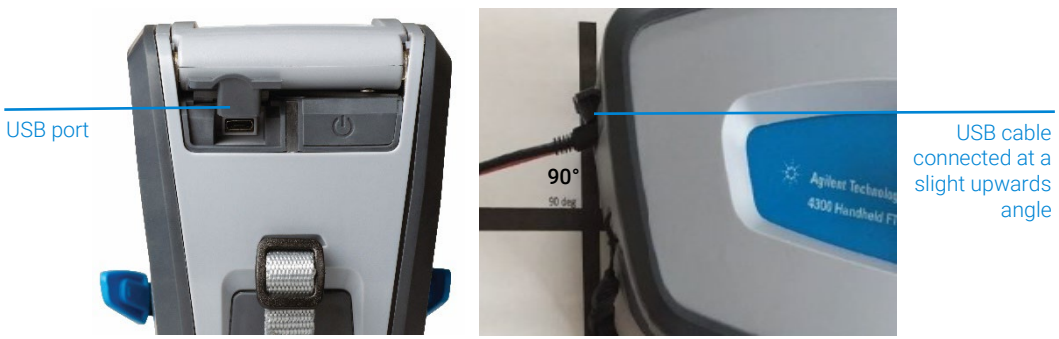

Figure 7 USB port (Left) & USB cable connected to instrument (Right)

Performance verification

The 4300 Handheld FTIR 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 below.

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 4300 Handheld FTIR is performing properly before using the system to make critical measurements. The 4300 Handheld FTIR 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. This should be checked on a daily or weekly basis.
- 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, frequency precision (laser calibration), and spectral resolution. The performance validation verifies the key aspects to the instrument's ability to measure good data. Performance validation should be run on a quarterly or bi-annual basis.

Diagnostic values

The following values can be viewed on the Diagnostics page in the MicroLab Mobile or MicroLab PC software under Advanced Features. 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.

- 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 1 Diagnostic values

Value	Optimal (green)	Marginal (yellow)	Critical (red)	Comment
Energy (centerburst)	29,000 - 15,000	>29,000 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	5 - 30 min	<5 min	
Source	1.9 A	>2.2 A or <1.6 A	>2.5 A or <1.0 A	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 reference cap is not in place.
Detector temperature	DTGS 35 - 44	<35 or >44	<30 or >48	Indicates a problem with the cooling circuitry or an ambient temperature over the specified range.
	MCT -51 to -49	<-51 or >-49	<-53 or >-47	The MCT detector is thermo-electrically cooled to approximately -50° C.
CPU temperature	10 - 75	>75	>80	Indicates the ambient temperature over the specified range.

Performance validation

The MicroLab software have four tests appropriate for performance validation of the 4300 Handheld FTIR. All of these tests can be accessed from the Advanced Features > System Check page on the MicroLab PC and MicroLab Mobile software.

The 4300 Handheld 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.

NOTE All performance tests should be run with the appropriate Retro-Reflector Sampling interface provided with the 4300 Handheld FTIR.

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.

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 Check

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 1921b 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 directed by the software by placing the designated film in the retro-reflector.

Spectral Resolution Check

The spectral resolution is tested in transmission measurement mode. This test involves calculating the absorbance difference between the maximum and adjacent minimum for a pair of slightly unresolved peaks, in separate regions of the measured polystyrene film spectrum.

3 Analyzing Samples

The Agilent 4300 Handheld FTIR instrument is provided with at least one sample interface as specified in your order. This section covers instructions on how to analyze a sample with the sampling interfaces offered on the 4300 Handheld FTIR, as well as how they can be interchanged if more than one sampling interface are ordered. Sample interface are shipped separately from the instrument.

External Reflectance technique

The 4300 Handheld FTIR instrument can be provided with a type of reflectance technique called the external reflectance. It is categorized into specular reflection for smooth surfaces, or diffuse reflections for rough surfaces.

The following sampling interfaces are offered with the 4300 Handheld FTIR:

- The 45° external (specular) reflectance sampling interface,
- The grazing angle (specular) sampling interface, and
- The diffuse reflectance sampling interface.

When coming in contact with a sample, the infrared (IR) light passes through the top surface of the sample before being reflected back out of the sample and into the detector of the 4300 Handheld 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 external reflectance is making sure the tip of the sampling interface makes stable contact with the sample surface and does not move during the measurement process. The reflectance technique can be used for thickness measurement, chemical degradation of composites and analysis of anodization of metals.

Table 2 Optimal uses of external reflectance sampling interfaces

Sampling Interface	Optimal Uses
External reflectance	• Suitable for analysis of smooth, opaque samples that reflect infrared light • Suitable for analysis of thin films and coatings greater than 1 micron in thickness on reflective metal surfaces
Grazing angle	• Suitable for analysis of ultra-thin films less than 1 micron in thickness • Suitable for analysis of very low-level contamination (nanometer thickness level)
Diffuse reflectance	• Suitable for analysis of samples that reflect little light

Reference caps

External reflectance measurements require the use of a reference sample for background measurement prior to analyzing samples. The reference sample is incorporated into the 4300 Handheld FTIR by means of a disk mounted into a cap that fits onto the tip of the reflectance sample interface.

The 4300 Handheld FTIR system offers four reference caps; each reference cap serves a different purpose depending on the type of sampling interface and sample being measured.

- Coarse Gold Reference Cap (with matte finish) for standard diffuse samples or 45° external reflectance measurements when the sample is semi reflective.
- Coarse Silver Diffuse Reference Cap with diffuse reflectance sample interface for highly scattering sample surfaces (e.g. soils, rocks, minerals, powders, tablets, coal, textiles, wood samples etc.).
- Gold Mirror Reference Cap is solely for grazing angle measurements with mid- to very reflective sample surfaces.
- SS (Stainless Steel) Mirror Reference Cap for specular measurements with the 45° external reflectance sample interface where the sample examined is moderately to highly reflective.

NOTE The reference cap being used should be as close to the sample's reflectivity as possible.

Attach reference caps

How to attach the reference caps

1 Each reference cap has a center-release buckle strap that can be attached onto a sample interface (see Figure 8). Ensure that the buckle strap is secured onto the fastener on the sample interface before usage.

CAUTION **Always attach the reference cap strap to the sample interface when in use.**

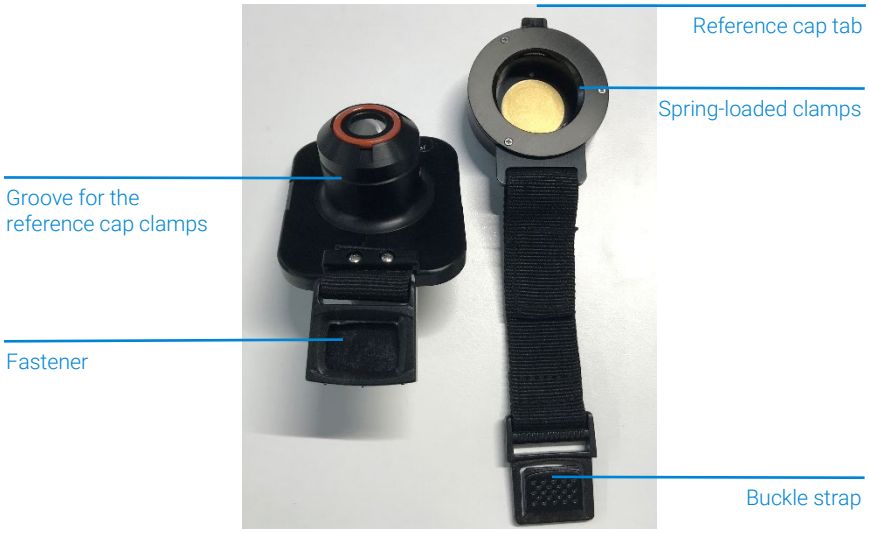

Figure 8 Sample interface (left) and reference cap (right)

2 Depress the tab on the reference cap to push the spring-loaded clamps to the side.

3 Place the cap over the sample interface tip and release the tab when the reference cap covers the sample interface. Ensure that the clamps sit on the sample interface groove and the reference cap is secure in position.

4 To remove the reference cap, depress the tab and remove the reference cap.

External reflectance analysis

The steps involved in performing an external reflectance analysis are:

1 Clean the sample interface tip.

2 Collect a background spectrum using the reference cap.

3 Collect a sample spectrum.

Cleaning the sample interface

1 Before using the 4300 Handheld FTIR external reflectance system on a new sample, it is important to ensure that the surface of the sampling interface is clean.

2 Clean the sample interface and sampling area using lint free cleaning wipes or suitable substitute (such as cotton swabs), moistened with suitable solvent such as acetone, methanol, ethanol or isopropyl alcohol.

3 Visually inspect the sample interface after cleaning to ensure there is no sample or cleaning residue present.

CAUTION **Do not pour or spray solvent directly onto the sample interface, or solvent may leak onto the optical components inside the sample interface housing.**

Do not touch the optical components inside the sample interface.

NOTE The diffuse reflectance and external reflectance sample interfaces both have O-rings installed to cushion the contact with the sample. The O-rings can be removed for cleaning or replaced if required. Visually inspect the sample interface tip to ensure it is clean to avoid possible contamination of the sample surface. See Section 4 for more details on how to remove O-rings for cleaning.

CAUTION **Do not open the spectrometer casing and attempt to clean interior surfaces. Opening the case 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 using an appropriate reference cap. By collecting a background automatically before each sample measurement, negative effects of changes in the environment can be avoided.

To collect a background spectrum:

1 First, choose the appropriate method and Activate it.

2 From the software HOME screen, tap Start to begin the measurement.

3 The software will collect a background automatically if the selected method is set up as recommended. Install the appropriate reference cap tightly against the tip of the sample interface when prompted.

NOTE Enable background collection before every sample by selecting the 'Collect new Background on every Sample' checkbox in the Method's 'Type' page using MicroLab Lite or MicroLab PC.

Each sampling interface comes with a default reference cap which can be removed and replaced by another reference cap. Refer to page 24 for more details.

4 Once the background has finished collecting, remove the reference cap and proceed to collect a sample spectrum.

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

Collecting a sample spectrum

To collect a sample spectrum using the external reflectance sample interface:

1 Remove the reference cap once the background has finished collecting.

CAUTION **Always use the provided wrist strap to secure the 4300 Handheld FTIR to the user and avoid damage due to dropping the system.**

2 Place the sample interface in contact with the sample as prompted by the software.

NOTE
- Ensure that the sample makes solid contact with the entire surface area of the sample interface tip, and is not held at an angle against the sample.
- Ensure that no movement occurs between the sample and the sample interface during the measurement.
- It is also possible to use a material like the sample to obtain more accurate results if the provided reference caps are inadequate.

3 The software will display a bar on MicroLab Mobile showing the intensity (absorbance) of the sample measurement. This screen is used to ensure that the sample has been correctly placed against the sampling interface.

For 4300 Handheld FTIR applications using reflectance, the value should be between 0.1 and 2 AU. Use the real-time data viewer in the software to ensure good contact between the sample and the sample interface is obtained.

4 Tap the Next button on the software screen to proceed with the analysis.

NOTE Sampling Continuity Check is recommended when using the 4300 Handheld FTIR. The function checks that the sample is kept in focus during the measurement. It can be enabled in the Method's 'Type' page using MicroLab Lite/PC.

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

NOTE Tapping the Home button will stop the measurement and return you to the Home screen.

5 After completing the sample measurement, immediately clean the sample from the sample interface.

NOTE Clean the surface of the sample interface with lint free cleaning wipes, or suitable substitute (such as cotton swabs) and an appropriate solvent such as methanol, ethanol or acetone.

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

Internal Reflectance technique (ATR)

The 4300 Handheld FTIR instrument can be provided with a dedicated sampling interface called the attenuated total reflectance (ATR). 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 that provides a very small and specific depth of penetration into the sample before reflecting back into the detector of the 4300 Handheld 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 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.

The Agilent 4300 Handheld FTIR is offered with two types of ATR crystal:

- Diamond, and
- Germanium

Table 3 Internal reflectance sampling interfaces

Sampling interface	Optimal Uses
Diamond ATR	• Ideal for analysis of solids, liquids, pastes and gel • Only the top 2-3 microns are analyzed
Germanium ATR	• Ideal for analysis of highly absorbing solids and liquids, e.g. carbon filled elastomers and rubbers • Only the top 0.5 to 2 micrometers is measured

See Appendix A for more details on the Diamond ATR crystals.

ATR analysis

The steps involved in performing an ATR analysis are:

1 Clean the ATR sample mounting window.

2 Collect a background spectrum.

3 Collect a sample spectrum.

Cleaning the sample interface

1 Before using the 4300 Handheld FTIR ATR system on a new sample, it is important to ensure the sampling interface mounting window is clean.

2 Clean the mounting window using lint free cleaning wipes, or a suitable substitute (such as cotton swabs), with a suitable solvent such as acetone, methanol, ethanol or isopropyl alcohol.

3 Visually inspect the mounting window after cleaning to make sure there is no sample or cleaning residue present.

CAUTION **Do not open the spectrometer casing and attempt to clean interior surfaces. Opening the case 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.

To collect a background spectrum:

1 From the software, choose a method and **Activate** it.

2 From the HOME screen, tap **Start** to begin the measurement.
The software will collect a background automatically if the method selected was set up as recommended.

NOTE The ATR should not be in contact with the sample or any substance other than the air in the atmosphere during the background measurement.

NOTE Enable background collection before every sample by selecting the 'Collect new Background on every Sample' checkbox in the Method's 'Type' page using MicroLab Lite or MicroLab PC.

3 Once the background has finished collecting, the software will instruct you to collect a sample spectrum.

For detailed software instructions on 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 ATR sample mounting surface, look for any contamination present on the ATR crystal from the previous sample. If residue is observed, repeat the cleaning procedure until the ATR mounting window is clear of any residue.**

Collecting a sample spectrum

To collect a sample spectrum using the ATR sample interface:

1 Once the background has finished collecting, the software will instruct you to put the ATR sample interface in contact with the sample.
Place a small amount of sample to be measured against the ATR crystal. The crystal is the clear, circular shaped material held in place by the surrounding metal disk at the surface of the sample interface.

NOTE

- For liquid samples, simply immerse the tip of the sample interface in the sample. Small samples can be measured by placing a drop of sample on the center of the ATR interface.
- Solid samples require establishing good contact between the ATR mounting window and the sample. Press the ATR sample interface firmly into the sample. Tilt the instrument to ensure that the sample interface surface is flat to the sample.
- 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 possible amount of sample will ease in the cleaning process.

In all cases, ensure that the sample covers the entire surface area of the ATR crystal.

If the sample is a powder or solid, the optional ATR sample press may be needed to ensure good contact with the sample. For detailed instructions on using the sample press, refer to the 4300 Handheld FTIR Stand Operation Manual.

2 Use the real-time data viewer in the software to ensure good contact between the sample and the ATR crystal.

3 Tap the NEXT button on the software screen to proceed with the analysis.

NOTE

Sampling Continuity Check is recommended when using the 4300 Handheld FTIR. The function checks that the sample is kept in focus during the measurement. It can be enabled in the Method's Type page using MicroLab Lite/PC.

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

NOTE

Tapping the Home button will stop the measurement and return you to the Home screen.

4 After completing the sample measurement, immediately clean the sample from the ATR sample mounting surface. It is important to make sure the sample mounting window is free of any residue from the previous sample.

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

CAUTION **The diamond ATR crystal is 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 of 1 to 14. Do not leave extremely acidic samples on the metal mounting plate for an extended time.**

Germanium is not resistant to scratching or pressure. Great care should be taken when using the Ge ATR to not scratch or break the Ge crystal.

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.**

Interchanging sampling accessories

The 4300 Handheld FTIR system is configured to accept a number of different sampling interfaces to accommodate the analysis of a range of samples. These sampling interfaces can easily be changed in the lab or in the field by even the novice user.

The 4300 Handheld FTIR is designed with a lock in place mechanism to ensure ruggedness and proper placement of the sampling interfaces.

Each sampling interface has an RFID chip on its guiding pin. The guiding pin allows for a one-way only installation. The RFID chip also allows the user to set the gain based on the sampling interface and save that information on the RFID chip. This information is then read by the firmware when a new sampling interface is installed. There are also two positioning pins on the 4300 Handheld FTIR instrument that guides the sample interface into position. The Retro-Reflector Reference interface is used in Figure 9.

NOTE Only interchange the sample interface when the instrument is turned off or when the Home screen appears. Do not change the sample interface during an analysis.

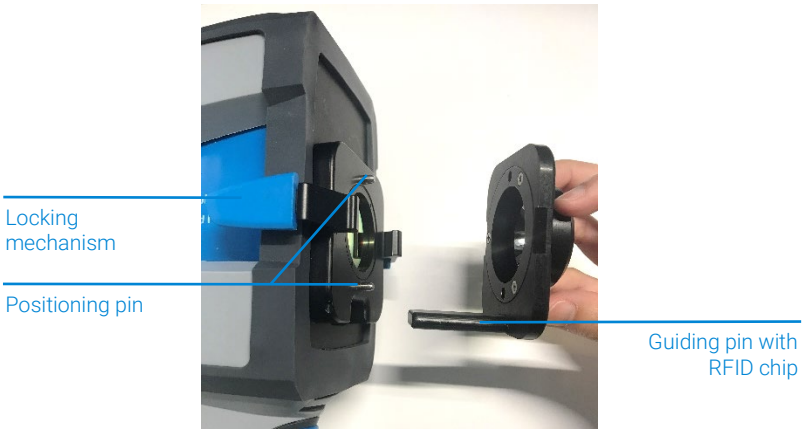

Figure 9 Interchanging 4300 Handheld FTIR Sampling Accessory

To switch between different sampling interfaces:

1 To remove a sample interface, press the locking mechanism on both sides to release. The sampling interface can then be removed while holding the locking arms down (see Figure 10).

Figure 10 Removing the sampling interface

2 Once removed, store the sampling interface in a clean and protected area, such as in a plastic bag inside the 4300 Handheld FTIR foam-lined shipping container.

CAUTION **When removed, the opening in the mounting surface of the sampling interface is exposed to the ambient environment. Use caution when handling to ensure the sample interface stays clean and nothing comes in contact with the delicate optical components.**

3 To install a sampling interface, simply insert the guiding pin into position and press the sampling interface against the locking mechanism. A slight clicking sound can be heard when it is secured in place by the locking mechanism.

4 Ensure that an appropriate gain is set for the sampling interface before using it. For external reflectance techniques, ensure that the proper reference cap is being used (see page 24).

4 Maintenance and Updates

WARNING

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

WARNING

Hazard
The 4300 Handheld FTIR was designed to operate in remote locations. However, analyzing certain materials and some maintenance procedures may be hazardous if the following guidelines are not practiced.

Cleaning

When the outside of the Agilent 4300 Handheld FTIR system needs cleaning, disconnect the power cord and any other connections and ensure the battery compartment is closed.

Use a damp, soft cloth and a mild detergent to wipe down the outside of the unit. Do not completely immerse the 4300 Handheld FTIR in water. Although the main optical housing is sealed, water can penetrate the handle and trigger components when completely immersed.

Clean the dust off the surfaces of the optics inside of the sample interface with clean, dry, oil-free compressed air or dry nitrogen if required.

Charge batteries

To recharge the batteries:

NOTE Batteries can only be charged in the appropriate battery charger. Batteries do not charge in the 4300 Handheld FTIR system.

1. Ensure the 4300 Handheld FTIR instrument is powered off.
2. Open the battery compartment door and remove the batteries out of the 4300 Handheld FTIR housing.
3. Use the provided battery charger to charge the batteries. Align the battery slots with the charger fins while inserting the battery into the charger.
   The charger can hold up to two batteries.
4. There are two LED indicator on the charger that represent the status of each battery.
   - Blinking green indicates that the battery is charging.
   - Solid green indicates that the battery is fully charged.
   - Solid red indicates that an error has occurred.
5. Once the batteries are fully charged, insert the batteries into the instrument for use. Refer to page 12 for instructions on inserting the batteries.

O-Ring replacement

To remove the O-ring:

The O-ring on the sample interfaces need replacing when there is visible damage or suspected contamination. Remove the sample interface from the 4300 Handheld FTIR.

- For PTFE encapsulated silicone O-ring replacements, refer to the instructions (P/N: G8184-90000) provided with the sample interface on how to remove and replace the O-ring.
- For EPDM O-ring replacements, remove the O-ring with your fingernails and wear a pair of disposable gloves to replace the O-rings with a new set.

Refer to Section 5 for the O-ring replacement kit spare part numbers.

Recharge desiccant

The desiccant cartridge is located in the back of the instrument. Refer to page 16 for detailed instructions on how to remove and recharge the desiccant.

MicroLab Mobile software update

To update the 4300 Handheld FTIR software:

1 Insert the UFD containing the software into a computer.

2 Restart the 4300 Handheld FTIR instrument.

3 On startup, exit the MicroLab Mobile software on the device to enter "Pass Through" mode, and connect to the computer using the USB connection.

- To exit MicroLab Mobile, select Logoff on the Home screen, click Exit and click OK when the Exit Confirmation dialog box appears.
- It is recommended to use AC power to power the instrument to avoid accidental shut down during the upgrade process.

4 Locate the MicroLab Mobile folder for the 4300 Handheld FTIR in the UFD.

- e.g. The folder name will be "V5.x.xxxx_4300" where x represents a number

5 The executable file will be displayed within the folder. Right-click on the file and select Run as Administrator option.

- e.g. The file name is "V5.x.xxxx_4300_NB.exe" where x represents a number

6 This will start the 4300 Series Software Update dialog box. Select the appropriate settings and click Next.

7 Follow the onscreen instructions.

8 When the update has been successfully completed, a pop-up box will appear. Click OK.

NOTE During the update, the LED will flash and change color.

9 The instrument will take several minutes to reboot and load the new software. When the instrument has completed the re-configuration, the instrument screen will display a message to prompt synchronization.
Refer to MicroLab Software Operation Manual for more details on synchronization.

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5 Spare Parts

Table 4 Spare parts

Part number	Description
G8180-67560	Coarse Gold Reference Cap (Matte finish)
G8180-67562	SS Mirror Reference Cap
G8180-67563	Coarse Silver Diff Reference Cap
G8180-67564	Gold Mirror Reference Cap
G8180-67300	4300 HH FTIR Polystyrene Ref Sample
G8180-67147	4300 HH FTIR Replacement Battery
G8180-67000	4300 HH FTIR Battery Charger
G8180-67135	4300 HH FTIR 15V DC Power Supply
G8180-67610	4300 HH FTIR MCT Retro Reflector
G8181-67611	4300 HH FTIR DTGS Retro Reflector
X3202-60106	O-Ring Removal Tool
G8184-67000	4300 O-Ring Replacement Kit (PTFE encapsulated silicone O-ring)
G8184-67001	O-Ring Replacement Kit (017 EPDM O-ring for external reflectance sample interface)
G8184-67002	O-Ring Replacement Kit (013 EPDM O-ring for diffuse reflectance sample interface)
G8180-67514	4300 HH FTIR Desiccant Cartridge (includes silica gel desiccant)
430-0164	USB cable 2 meter A to Micro B
G8180-68001	4300 HH FTIR D-ATR
G8180-68002	4300 HH FTIR Ge-ATR
G8180-68003	4300 HH FTIR Ext Ref
G8180-68004	4300 HH FTIR Diff Ref
G8180-68005	4300 HH FTIR GA Ref
G8180-20029	4300 Handheld FTIR wrist strap
250-0113	4300 Handheld FTIR stylus

For sampling accessories, upgrades, service contracts, repair and refurbishment services, contact Agilent or go to the Agilent website at www.agilent.com.

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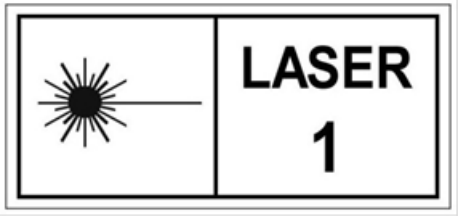
6 Specification

Technical

- **Interferometer geometry:** High-throughput Michelson interferometer with fixed and moving flat mirrors
- **Standard beamsplitter:** Zinc selenide
- **Maximum spectral resolution:** 4 cm^{-1} (both DTGS and MCT detectors)
- **Laser:** Low-powered solid state
- **Source:** Wire-wound element
- **DTGS Spectral range:** 4500 to 650 cm^{-1}
- **MCT Spectral range:** 5000 to 1100 cm^{-1}
- **Sample interface:** Diamond ATR, Germanium ATR, External Reflectance - 45°, Grazing Angle and Diffuse Reflectance
- **Detector:** 1.3 mm diameter DTGS; 1.0 mm diameter MCT
- **Width:** 10.16 cm (7.5 in)
- **Depth:** 19.05 cm (7.5 in)
- **Height:** 34.5 cm (13.6 in)
- **Weight:** 1.88 kg (4.15 lbs) without batteries; 2.22 kg (4.9 lbs) with batteries
- **Power supply:** 100 to 240 V AC, 3 A, 50 to 60 Hz
- IEC/EN 60529: IP54

Safety Information



Agilent 4300 Handheld 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 conformance with IEC 60825-1 Ed. 3., as described in Laser Notice No. 56, dated May 8, 2019.

Environmental conditions

- DTGS Operating Temperature: 0 to 50 °C; 32 to 122 °F (during use)
- MCT Operating Temperature: 0 to 40 °C; 32 to 104 °F (during use)
- Storage Temperature: -25 to 75 °C; -13 to 167 °F
- Humidity: Up to 95%, non-condensing
- Altitude: Up to 2000 m

Integrated CPU

- Processor: 520 MHz Intel® PXA270
- Memory: 64 MB low power RAM
- Hard drive: 128 MB NAND flash

Information symbols

The following symbols may appear on the instrument for your information:

The equipment may be used in conditions with dust and moisture.

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Appendix A: Diamond ATR Crystal

The Agilent Diamond ATR uses a type IIa synthetic 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.

The 4300 Handheld FTIR ATR sampling interface is a single reflection system. It uses diamond because it is the world's most durable substance and gives the 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 optional instrument stand and 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 is 2 mm in diameter with a 200 µm active area and provides an approximate 2 micron depth of penetration for IR energy at 1700 cm^{-1}. The single reflection ATR element protrudes slightly above the probe's metal mounting plate.

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Appendix B: Access the MAC Address

The MAC address is required in order to register a library (e.g., ST Japan).

Access MAC address of the 4300 Handheld FTIR instrument:

1. Ensure a sample interface is attached to the instrument.
2. Login to the MicroLab Mobile software as the administrator.
3. From the home page, click on the More Options  button at the bottom of the screen.
4. Select Advanced Features.
5. Select Diagnostics from the Advanced Features page.
6. On the Diagnostics page, select the More Options  button at the bottom of the screen.
7. Two options will appear: Adjust Gain and Mac Address. Select Mac Address.
8. The Mac Address for the unit will be displayed.
9. Click on the Home button to return to the home page.

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Appendix C: Transferring Library Files with TSFileTransfer Program

MicroLab Lite's Synchronize function (Advanced Features > Synchronize) allows for results, system log, reports and methods transfer between the 4300 Handheld FTIR instrument and the computer. Refer to the MicroLab Software Operation Manual for more information.

NOTE This procedure is not applicable in 21 CFR Part 11 environment.

CAUTION **Performing any procedure other than the one listed in this manual can result in the loss of data.**

For library files transfer, a program called TSFileTransfer.exe is utilized to transfer library files to and from the instrument. The program is installed during the installation of MicroLab software (revision B.05.0 or higher).

Transferring files from the computer to the instrument:

1 Turn on the 4300 Handheld FTIR and exit MicroLab Mobile to enter 'Pass Through' mode.
Refer to the MicroLab Software Operation Manual for more information.

2 Connect the instrument to the computer via the USB cable.

CAUTION **The USB cable must be inserted at a slight upwards angle into the instrument USB connector as shown in Figure 7. Caution needs to be taken when inserting and removing the cable to prevent damage to the USB port.**

Always place the instrument on its side when the USB cable is connected.

3 Navigate to C:\Program Files (x86)\Agilent\MicroLab PC folder on the computer.

4 Locate TSFileTransfer.exe and double-click to launch the program.

5 Click on the green Connect button. The button will change to a red Disconnect button when the software is loaded and communicating with the instrument.

6 Click on the Download Files tab to download files to the instrument.

7 Click on the  button next to the 'Source files(s) on PC' field.

8 An Open window will appear. Select the source files you want to transfer to the instrument.

- Library files are located in C:\Users\Public\Documents\Agilent\MicroLab\Libraries folder.

NOTE Each library consists of three files : *.a2l, *.a2x, and *.lib. All three files must be transferred together for the library to be readable.
Use CTRL or SHIFT on your keyboard to select more than one file.

9 Click on the Open button to confirm file selections.

10 Click on the  button next to the 'Destination file or directory on SOM' field to select destination folder on the instrument.

11 The 'CEFileTree' window will appear. Locate YAFFS_Part1 folder on the left pane and expand the folder.

12 Locate the Libraries subfolder.

NOTE Different file types are stored in different folders. Storing the library files in any other folder will prevent the instrument from accessing them.

13 Click Ok to confirm destination directory.

14 Click the green Download button on the 'TopScan File Transfer' window. All selected files will be transferred to the corresponding folder on the 4300 Handheld FTIR instrument.

Transferring files from the instrument to the computer:

1 Repeat Steps 1 - 5 above.

2 Click on the Upload Files tab to upload files from the instrument.

3 Click on the  button next to the 'Source files(s) on SOM' field.

4 The 'CEFileTree' window will appear. Locate YAFFS_Part1 folder on the left pane and expand the folder.

5 Locate the Libraries subfolder.

6 Select the library files you want to transfer.

NOTE Each library consists of three files : *.a2l, *.a2x, and *.lib. All three files must be transferred together for the library to be readable.
Use CTRL or SHIFT on your keyboard to select more than one file.

7 Click Ok to confirm file selections.

8 Click on the  button next to the 'Destination file or folder on PC' field.

9 The 'Browse For Folder' window will appear. Navigate to C:\Users\Public\Documents\Agilent\MicroLab\Libraries folder.

10 Click OK to close the window.

11 Click the green Upload button on the 'TopScan File Transfer' window. All selected files will be transferred to the MicroLab PC folder on the computer.

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In This Book

The manual describes the following:

- Introduction
- Getting Started
- Analyzing Samples
- Maintenance and Updates
- Spare Parts
- Specifications

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