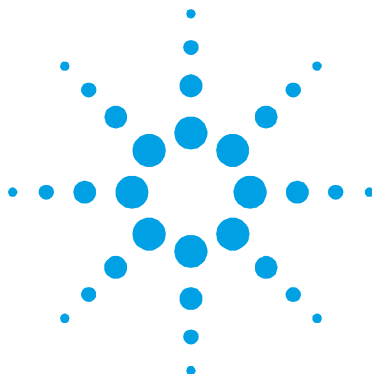




PM-IRRAS Accessory

Hardware Manual



Agilent Technologies

Notices

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Safety Notices

CAUTION

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

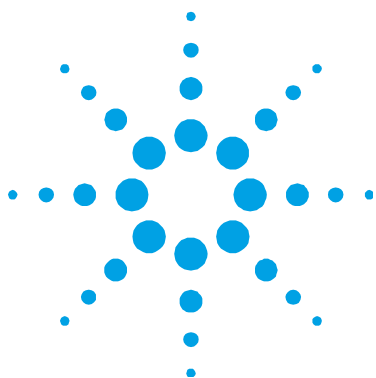
WARNING

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

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1. Safety Practices and Hazards

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The Polarization Modulation Infrared Reflection Absorption Spectroscopy (PM-IRRAS) accessory has been carefully designed to be used as an accessory for Agilent FTIR spectrometers. When used properly it provides an accurate, fast, flexible and safe analytical system.

If the equipment is not used in a manner specified by the manufacturer, the protection provided by the equipment may be impaired.

Information on safety practices appear throughout the documentation (both printed and online) provided with your spectrometer. Before using the PM-IRRAS accessory, you must thoroughly read these safety practices.

Observe all relevant safety practices at all times.

Cryogenic cooling

Detectors used with your PM-IRRAS accessory are cryogenically cooled. The liquid nitrogen used in this process is extremely cold and can cause damage to the human body. Use appropriate protective equipment when working with liquid nitrogen.

Electrical hazards

The Agilent PM-IRRAS accessory contains electrical circuits, devices, and components operating at dangerous voltages. Contact with these circuits, devices and components can cause death, serious injury, or painful electrical shock.

Good grounding/earthing is essential to avoid a potentially serious electric shock hazard. Ensure that there is an integral ground connection between the metal surfaces of the PM-IRRAS accessory and the 3 pin earth-grounded receptacle.

NOTE

The above model is Equipment Class I

Application of the wrong supply voltage can create a fire hazard and a potentially serious shock hazard, and could seriously damage the PM-IRRAS accessory.

Replace blown fuses with fuses of the size and rating as stipulated in the text adjacent to the fuse holder or in the manuals where listed.

Do NOT use power cords with faulty or frayed insulation.

Laser safety

The Agilent PM-IRRAS accessory is used with Agilent FTIR spectrometers, which incorporate a helium-neon (He-Ne) laser operating in the visible region at 632.8 nanometers. The spectrometer is a Class 2 laser product, powerful enough to warrant caution in its use. Agilent FTIR spectrometers comply with FDA and CE standards for light-emitting products.

An attenuated portion of the laser beam passes into and through the spectrometer sample compartment and can be directed from there into the accessory. Although not powerful enough to harm your skin should your hand intercept it, the laser light could cause retinal (eye) damage during prolonged direct viewing. This is not possible given the normal optical layout of the spectrometer. However, if a highly reflective surface such as a mirror is allowed to intercept the beam, the beam could be redirected out of the sample compartment resulting in on-axis or direct viewing. Care must be taken to avoid this.

Refer to the spectrometer hardware manual for more information about the laser.

Other precautions

Use of the PM-IRRAS accessory may involve materials, solvents and solutions which are flammable, corrosive, toxic or otherwise hazardous. Careless, improper, or unskilled use of such materials, solvents and solutions can create explosion hazards, fire hazards, toxicity and other hazards which can result in death, serious personal injury, and damage to equipment and property.

ALWAYS ensure that laboratory safety practices governing the use, handling and disposal of such materials are strictly observed. These safety practices should include the wearing of appropriate safety clothing and safety glasses.

Warnings and cautions

Carefully read all warnings and cautions and observe them at all times.

A Warning message is used in the text when failure to observe instructions or precautions could result in death or injury. Warnings have the following format:

WARNING**Hazard Type**

Nature of the hazard, information on how to avoid the hazard, and possible consequences if you don't.

The triangular symbols that appear in conjunction with warnings are outlined in the next section.

A Caution message is used when failure to observe instructions could result in damage to equipment (Agilent-supplied and/or other associated equipment). Cautions have the following format:

CAUTION

Caution information appears here.

Information symbols

The following triangular symbols appear in conjunction with warnings on the spectrometer and associated documentation. The hazard they depict is shown below each symbol:



Broken glass



Corrosive liquid



Electrical shock



Extreme cold



Eye hazard



Fire hazard



Heavy weight
(danger to feet)



Heavy weight
(danger to hands)



Hot surface



Laser hazard



Moving parts



Noxious gas



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

The following symbols also appear on the instrument or in the documentation:



Mains power on.



Mains power off.



Fuse.



Single phase alternating current.



When attached to the rear of the instrument, indicates that the product complies with the requirements of one or more EU directives.



When attached to the rear of the product, indicates that the product has been certified (evaluated) to CSA 61010.1 and UL 61010-1.

CE compliance

The Agilent Polarization Modulation Infrared Reflection Absorption Spectroscopy (PM-IRRAS) accessory has been designed to comply with the requirements of the Electromagnetic Compatibility (EMC) Directive and the Low Voltage (electrical safety) Directive (commonly referred to as the LVD) of the European Union. Agilent has confirmed that each product complies with the relevant directives by testing a prototype against the prescribed EN (European Norm) standards.

Proof that a product complies with the directives is indicated by:

- The CE marking appearing on the rear of the product.
- The documentation package that accompanies the product, containing a copy of the Declaration of Conformity. This declaration is the legal declaration by Agilent that the product complies with the directives and also shows the EN standards to which the product was tested to demonstrate compliance.

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:

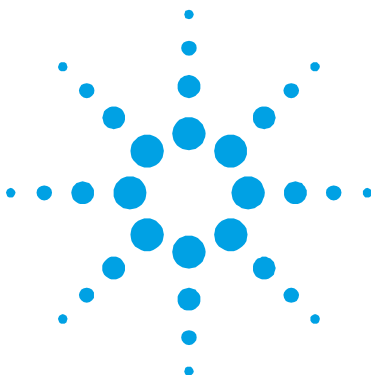
- 1 Relocate the radio or antenna.
- 2 Move the device away from the radio or television.
- 3 Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
- 4 Make sure that all peripheral devices are also certified.

- 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.

ICES/NMB-001

This ISM device complies with Canadian ICES- 001.

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



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Specifications

Environmental

The Agilent Polarization Modulation Infrared Reflection Absorption Spectroscopy (PM-IRRAS) accessory is designed for indoor use. It is suitable for the following categories:

- Installation category II
- Pollution degree 2
- Equipment Class I

These conditions are required for proper operation of your PM-IRRAS accessory:

- Temperature: 20 °C to 26 °C (68 °F to 80 °F)
- Temperature gradient: 1 °C/hr (1.8 °F/hr) maximum
- Relative humidity: 20% to 50% non-condensing

- Altitude: 3000 m (10,000 ft) maximum
- Free from corrosive and flammable fumes
- Free from strong electromagnetic fields

NOTE

Performance degradation may result from the exposure of the spectrometer to strong radio frequency energy. If degradation is experienced, reorient or relocate the spectrometer or the radio frequency source.

- Free from vibrations

Power

- PEM controller: 60 VA
- 100 to 240 VAC, 60 W, 50 or 60 Hz. An auto-ranging power supply is used. No reconfiguration is required for voltage changes.
- Dedicated, 10-A circuit with grounded receptacle.

Purge gas

A source of clean, dry air (dried to dew point of -70°C) or liquid nitrogen boil-off to be used as a purge gas. Where compressed nitrogen is used, it must be dry, oil-free and uncontaminated, with purity of 99.996% or better.

A flow rate of 13 L/min ($30\text{ ft}^3/\text{hr}$) maximum is required to assure a non-condensing environment.

The gas supply must be equipped with fittings to accept 6 mm outer diameter (OD) tubing.

Tubing should be clean and free of any dust and debris. Do not use tubing treated with talcum powder.

Liquid nitrogen

WARNING**Extreme Cold Hazard**

Liquid nitrogen is very cold and can cause damage to the human body. Use appropriate protective equipment when handling liquid nitrogen.

The detector in the PM-IRRAS accessory will require a supply of liquid nitrogen to cool it. It takes about 500 milliliters (16 ounces) of liquid nitrogen for the initial fill to bring the Dewar to an equilibrium temperature. It will take about 20 minutes to reach equilibrium. Then add an additional 200 milliliters (7 ounces) of liquid nitrogen to top off the Dewar.

Weights and dimensions

- Width: 67.5 cm (27 in)
- Depth: 61 cm (24 in)
- Height: 27.5 cm (11 in)
- Weight: 45 kg (100 lb)

Space

A flat and level surface is required. It must be rigid enough to support the weight of the spectrometer and the PM-IRRAS accessory and any additional accessories (if applicable) without warping or sagging. The loaded flatness tolerance is 0.4 millimeter per 300 millimeters of length (1/64 inch per foot of length).

A minimum of 30 centimeters (12 inches) of free space is required behind the spectrometer and PM-IRRAS accessory.

An overhead space of 75 centimeters (30 inches) is required.

Installation requirements

Before receiving your Agilent FTIR spectrometer and PM-IRRAS accessory, you will have been given a copy of the FTIR spectrometer Site Preparation Guide, which describes the environmental and operating requirements of the spectrometer system. You must prepare your laboratory according to these instructions before the system can be installed. You should keep the pre-installation manual for future reference. If you have misplaced your copy, you can obtain a replacement from your local Agilent office.

Training

The software provided with your Agilent FTIR spectrometer includes an extensive Help applicable for the spectrometer and the PM-IRRAS accessory. If you require additional instruction or training, contact your local Agilent office.

Documentation

This hardware manual provides basic instructions for operating and maintaining your PM-IRRAS accessory.

In addition to this manual you may receive other hardware and software manuals depending on the type of configuration purchased.

Further information and instructions about software, analysis methodology, operating procedures and various accessories is provided in the Help and electronic manuals saved onto your computer hard drive during software installation for your Agilent FTIR spectrometer.



3. Hardware

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Overview

The Agilent Polarization Modulation Infrared Reflection Absorption Spectroscopy (PM-IRRAS) accessory is designed to be used as an accessory for Agilent FTIR spectrometers. The PM-IRRAS accessory is used for grazing-angle reflectance spectroscopy.

The PM-IRRAS accessory includes:

- External Experiment Module (EEM) (including Plexiglas cover).
- Base plate with optics mounts.
- Base plate with one liquid nitrogen-cooled detector.
- Photoelastic Modulator (PEM).
- PEM controller (complete with user manual).
- This hardware manual.

NOTE

The PEM and the PEM controller are manufactured by Hinds Instruments, Inc. Details concerning operation and specifications of these devices are not provided in this manual. Should you require information about these devices please contact Hinds Instruments, Inc. directly.

The PM-IRRAS accessory is attached to the right side of an Agilent 600 Series FTIR spectrometer. It is installed and aligned by your Agilent customer service representative.

Beam path

The infrared beam exits the spectrometer and is directed by two flat mirrors (A and C in Figure 1) to the parabolic mirror (D) for focusing.

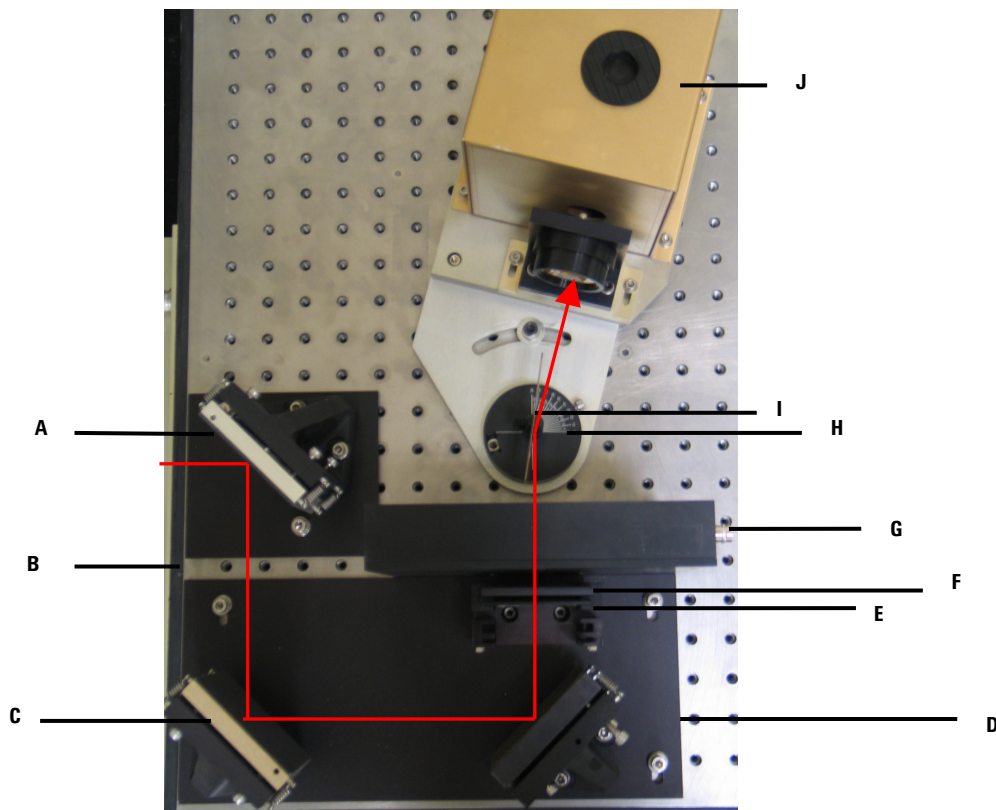


Figure 1. Agilent PM-IRRAS accessory beam path with cover removed for clarity

- | | | |
|----------------------------|---------------------------|----------------|
| A) Flat mirror | B) Beam from spectrometer | C) Flat mirror |
| D) Parabolic mirror | E) UDR filter | F) Polarizer |
| G) Photo-elastic modulator | H) Angle Indicator | I) Sample |
| J) Detector | | |

Filters

The focused beam may pass through three optical elements before passing through the Photoelastic Modulator (PEM). As shown in figure 2 these elements are:

- Infrared polarizer (A in Figure 2): with the plane of polarization set parallel to the plane of incidence at the sample.
- UDR filter (B in Figure 2) location for UDR4 or UDR8 filter: The UDR filters are optical lowpass filters which transmit below $3,950\text{ cm}^{-1}$ (UDR4 filter) or $1,975\text{ cm}^{-1}$ (UDR8 filter), and are used with the UDR=4 or UDR=8 parameter setting, respectively, of Resolutions Pro software.
- Quartz window (optional, C in Figure 2): acting as a high pass optical filter transmitting above $2,000\text{ cm}^{-1}$. When used with the UDR4 filter (see below), allows data collection at UDR=8 for the bandpass between $3,950$ and $2,000\text{ cm}^{-1}$. To display data in the required range click 'Transforms' > 'Fourier Transform' > 'Advanced' and use the 'Included Frequency' parameter.

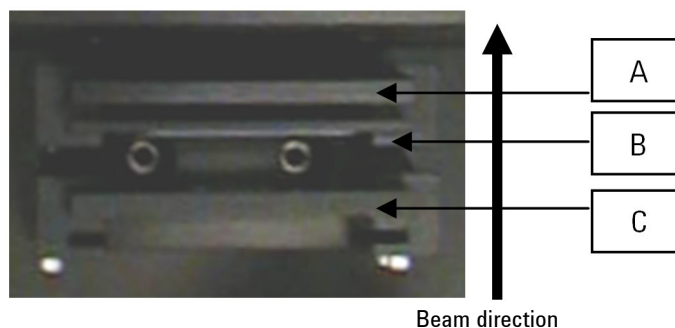


Figure 2. Infrared polarizer plane of polarization
A) Polarizer B) UDR filter C) Quartz window

The filters and polarizers must be placed in the order as shown in Figure 2.

Orientation of sample and detector

Mount the sample (maximum sample size 75 mm) in the optical clamp so that the clamp grips the edge of the sample. The detector should be offset from 90° by twice the sample angle. The beam exits the PEM along the 90° line. Typically, the sample should be set along the 80° line (10° offset) and the detector set at 70° (20° offset). The detector position may be optimized by maximizing the magnitude of the interferogram centerburst in rapid scan setup mode while making small adjustments of the detector angle. The ZnSe lens assembly has been optimized on installation. Do not adjust the lens relative to the detector.

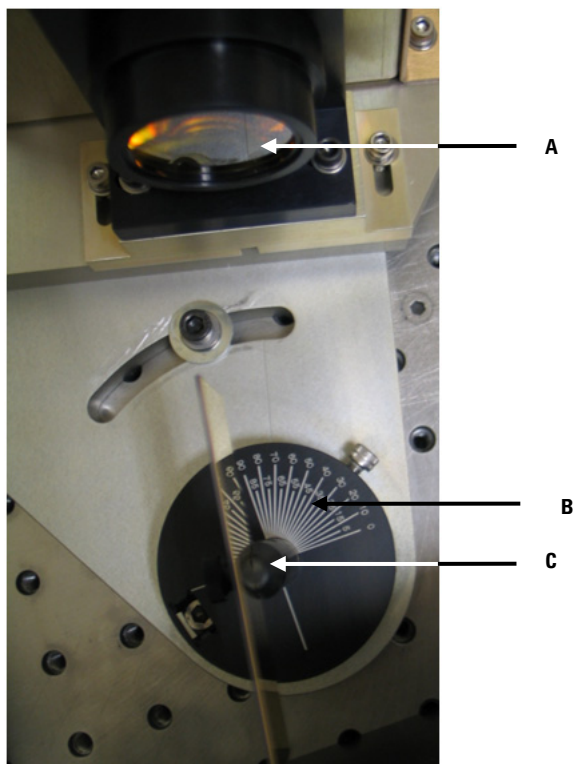
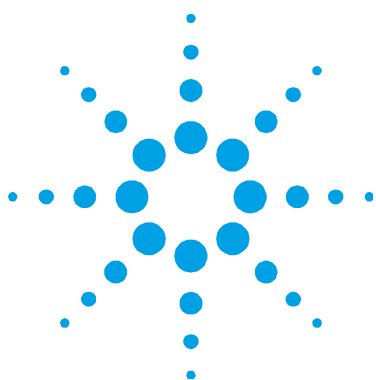


Figure 3. Sample and detector orientation

A) Detector

B) Angle indicator

C) Sample



4. Basic Operation

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Introduction

A schematic diagram of a PM-IRRAS experiment is shown in Figure 4:

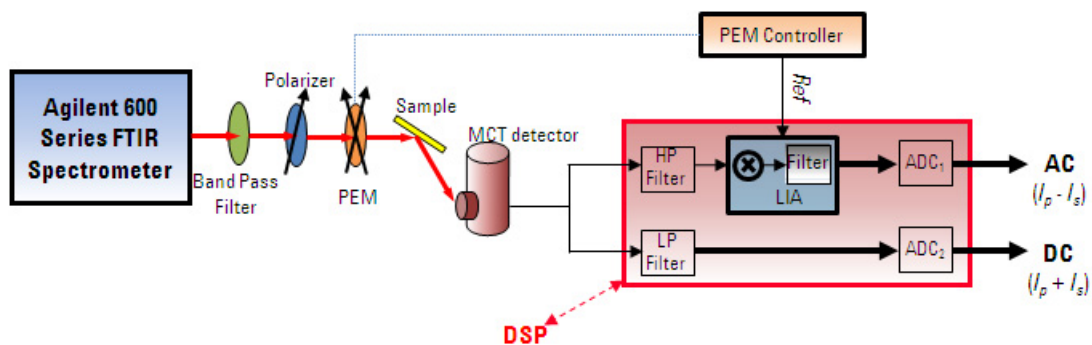


Figure 4. Schematic diagram of an IRRAS experiment

Agilent provides two modes of collection for PM-IRRAS measurements, that is, rapid scan or step scan. In rapid scan mode, broadband infrared radiation from a ceramic source is modulated by rapid scanning (typically at 12.5 kHz) of the interferometer in the spectrometer. The modulated radiation is filtered with a low pass optical filter transmitting radiation below $3,950\text{ cm}^{-1}$ or $1,975\text{ cm}^{-1}$. The purpose of these low pass filters is to enable higher throughput at frequencies of interest. After filtering, the IR radiation is polarized by a wire grid polarizer. The polarized IR beam is then modulated between 's' and 'p' polarization at 74 kHz by the ZnSe PEM before reflecting off the sample at a grazing angle of incidence; typically, 80° to 85° from normal. Most commonly, the sample is a self-assembled monolayer film on a metallic substrate, such as gold. The IR light is subsequently focused onto a narrow band MCT detector with on-axis refractive optics.

After the detector and preamplifier, the signal is sent along two parallel paths as displayed in Figure 4. In the top path, shown in Figure 4, the signal is high pass filtered and then demodulated for polarization modulation by a Lock-in Amplifier (LIA). The output of the LIA provides the dichroic difference spectrum between 's' and 'p' polarized light, often called the AC spectrum. At large angles of incidence, absorption of 'p' polarized light by molecules adsorbed on a metallic surface is enhanced. In contrast, absorption of 's' polarized light is virtually zero. Therefore, the dichroic difference spectrum is only sensitive to the thin film on the substrate, allowing the surface signal to be digitized with high dynamic range.

In the lower path above, the detector is low-pass filtered to eliminate the high frequency PEM component, providing the single beam (DC) spectrum of the substrate. The final step of data processing is to ratio the AC to the DC spectrum to obtain the surface spectrum with high sensitivity and free from atmospheric contamination.

In step scan mode, the IR radiation is phase modulated by the interferometer, typically at phase modulation frequencies of 400 or 800 Hz at an amplitude of 1.0 or 2.0 λ HeNe, and stepping at 0.5–2.5 Hz. The modulated radiation follows the same optical path as in the rapid scan measurement described above. In step scan mode the optical low pass filters have dual purpose. In addition to enabling higher throughput at frequencies of interest, a major advantage is a reduction in collection time. Although the optical arrangement is identical between step and rapid scan, there is a significant difference in the way that the detector signal is processed. In step scan mode, unique digital signal processing (DSP) software within Resolutions Pro is used to replace the function of the LIA, the electronic filters and the second A/D converter.

Data collection

Data can be collected either in rapid scan mode or in step scan mode, which uses DSP(3) software. See the DSP(3) section in the Help.

Preparing the PEM-IRRAS

Power

The PEM controller should be plugged into an AC (alternating current) outlet. For power requirements refer to the PEM-100 User Manual.

Power for the detector is supplied from the FTIR spectrometer.

Detector cooling

Fill the detector Dewar with liquid nitrogen for each detector to be used, as described on Page 15.

WARNING**Extreme Cold Hazard**

Liquid nitrogen is very cold and can cause damage to the human body. Use appropriate protective equipment when handling liquid nitrogen.

Sample purge

Turn on the purge, if desired. The purge fitting is in the back of the PM-IRRAS accessory and accepts 6 millimeter OD tubing. Set the purge rate to 13 liters per minute.

Scanning**To set up and scan:**

- 1 Install the UDR4 filter and the Polarizer ('0 deg' should be at the top).
- 2 Install the IR Reflective Test Slide in the sample holder on the rotary stage. The coated side of the slide should be facing to the right in the beam path. The coated side will be the side facing you with the etched corner in the upper right corner. Set the angle of the slide at 90 degrees.
- 3 Optical setup:
 - a PEM in beam path (TURNED OFF)
 - b Polarizer in holder
 - c UDR4 filter
 - d IR Reflective Test Slide at 90 degrees
 - e Detector at 70–75 degrees
- 4 In the 'Method Editor' in Resolutions Pro, set the following instrument parameters:
 - a Common Settings:
 - Resolution = 8 cm⁻¹

- b Collect:**
 - Speed = 25 KHz
 - UDR = 2
 - Filter = 6.4 KHz
 - Sensitivity = 1
- c Spectrometer Configuration:**
 - IR Source = Rear: Mid-IR
 - Beam Path = Right
 - Detector = Ext. 1
 - Aperture = 2 cm^{-1}
 - Attenuator = 50%
- 5 Click **Signal Monitor** and rotate the test slide to optimize the interferogram signal.**
- 6 Select **Single Beam** while in ‘Signal Monitor’ and check that the slide is correctly positioned. Figure 5 shows a spectrum of the coated side and Figure 6 shows a spectrum of the glass side.**

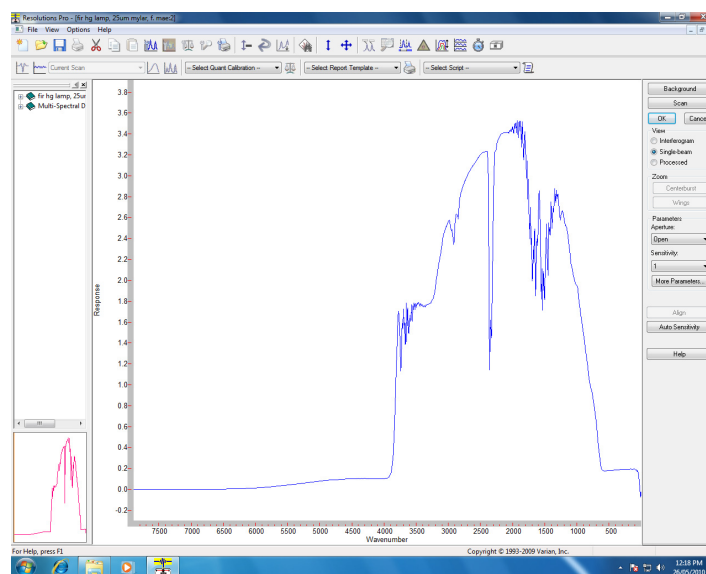


Figure 5. Single beam spectrum of the coated side of the test slide

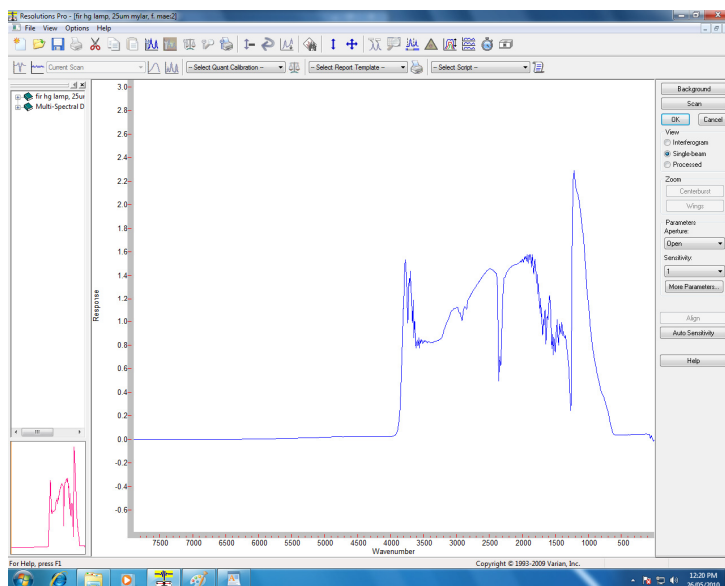


Figure 6. Spectrum of the glass side of the test slide

- 7** Set up the PEM controller as follows: (refer to the PEM-100 controller manual for details)
 - a** $\lambda = 7500 \text{ nm}$
 - b** Retardation = 0.5λ
 - c** Frequency = $2F$

NOTE

The PEM frequency may take several minutes to stabilize after it has been turned on or settings have been changed (for example, retardation amplitude or wavelength). Allow the PEM to stabilize before starting data collection.

- 8** To perform PM-IRRAS measurements in step scan mode proceed to the next section. To perform PM-IRRAS measurements in rapid scan mode skip forward to Page 32.

Data Collection in Step Scan Mode

- 1** Go to **Collect > Step Scan > Step-Scan**. Select the following parameters:
 - a** Electronics:
 - Speed = 800 Hz
 - UDR = 2
 - Filter = NONE
 - b** Optics:
 - Source = Mid-IR
 - Beam Path = Right
 - Detector = Ext. 1
 - Aperture = 2 cm⁻¹
 - Attenuator = 50%
- 2** Click **Setup**.
- 3** Click **Align**.
- 4** Click **Find Centerburst**.
- 5** Go to **Collect > Step Scan > PM-IRRAS DSP(3)** and set the parameters as:
 - a** PM-IRRAS DSP(3)
 - Linear Dichroism (Signal at 74 KHz)
 - Sample Modulation Frequency = 50 Hz
 - Modulation Amplitude = 1 λ
 - Delay After Step (ms) = 55
 - PEM Frequency in Hz = Set to equal the 2F frequency displayed on PEM controller (may need to be calculated)
 - Save Raw Data, Starting Step = 75
 - Save Raw Data, Total Step Number = 1

b Electronics:

- Speed = Unavailable
- UDR = 4
- Filter = Unavailable
- Resolution = 8 cm^{-1}
- Sensitivity = 1
- Scans to Co-add = 1

c Optics:

- Source = Mid-IR
- Beam Path = Right
- Detector = Ext. 1
- Aperture = 2 cm^{-1}
- Attenuator = 50%

d Advanced:

- Interferogram Symmetry = Asymmetric

6 The switches on the DSP3 Filter Box should be set as follows:

- Filter = IN
- Frequency = 74 KHz

7 Now do a Setup from PM-IRRAS DSP(3) window. Click the **DSP Calibrate** button. When the calibration is complete, click **OK**.

8 Click **Scan**.

9 When the scan is complete, look at Spectrum 1 and 5. Both should be strong interferograms. If either interferogram is weak or only noise, then the actual PEM frequency must be calculated.

10 To calculate the PEM frequency, select Spectrum 11. Go to **Transforms > Fourier Transforms**. A 'Compute' window will open. On the **General** tab, select **Apodization Function = Triangular**, **Zero Filling Factor = AUTO**. On the **Advanced** tab select **Use Standard Processing**. Click **OK**.

- 11** In the resulting spectrum, there should be a strong peak at approximately 0.41 as shown in Figure 7. Multiply the peak location by 28571. Subtract this product from 85714. This result is the actual frequency of the PEM. Enter this value for the 'PEM Frequency in Hz' parameter and then click Scan again. This value may be different than the value on the PEM controller display. Note the difference between the calculated value and the displayed value. This difference will be constant.

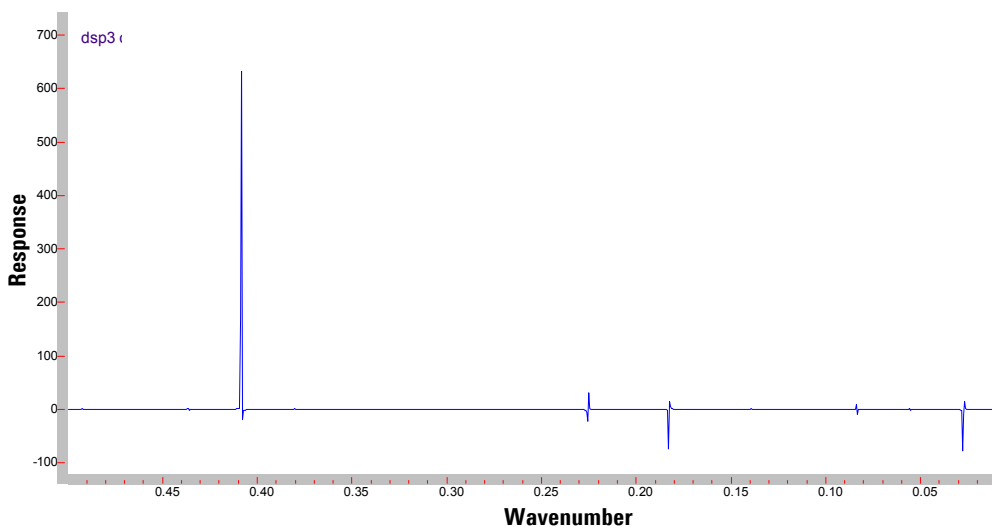


Figure 7. Spectrum of time based raw data for step number 75Error! No bookmark name given.

- 12** When the scan is complete, select Spectrum 1 and Spectrum 5. Go to **Transforms > Fourier Transforms**. Set Apodization Function = TRIANGULAR and set Zero Function = TRIANGULAR and set Zero Filling factor = 4 and then click OK to compute the interferograms. Spectrum 5 should look similar to Figure 8.

Basic Operation

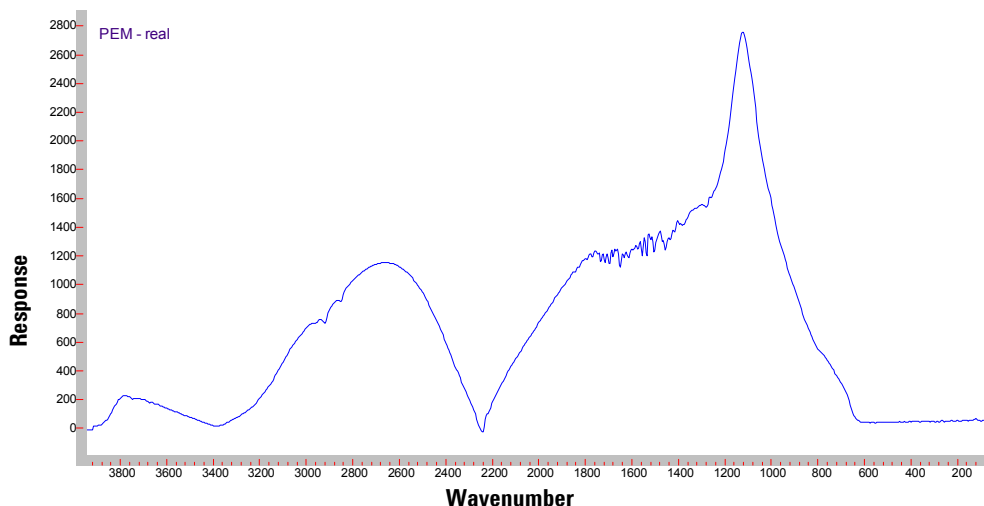


Figure 8. AC spectrum ($I_p - I_s$)

- 13 Select Spectrum 5 and go to **Transforms > Spectral Calculator > Paired Spectra**. A 'Spectral Arithmetic: Paired Spectra Operations' dialog box will be displayed. Select **{/} Divide by Reference**. Select **Use Current** and then select **1. Phase Modulation - Real** from the list of spectra in the bottom right corner. Click **Replace**.
- 14 Zoom in on Spectrum 5 between approximately 1500 and 900 cm^{-1} . The spectrum should look similar to Figure 9 with a large broad peak around $\sim 1110 \text{ cm}^{-1}$ and a weaker peak around $\sim 1265 \text{ cm}^{-1}$.

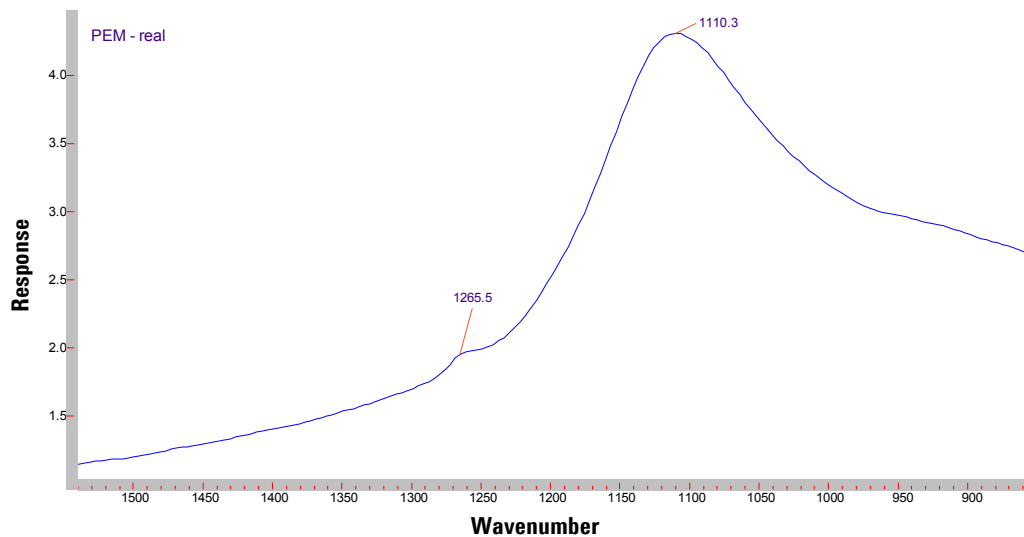


Figure 9. Ratio of AC spectrum ($I_p - I_s$) to DC spectrum ($I_p + I_s$)

Data Collection in Rapid Scan Mode

- 1 Connect the hardware as shown in the schematic.

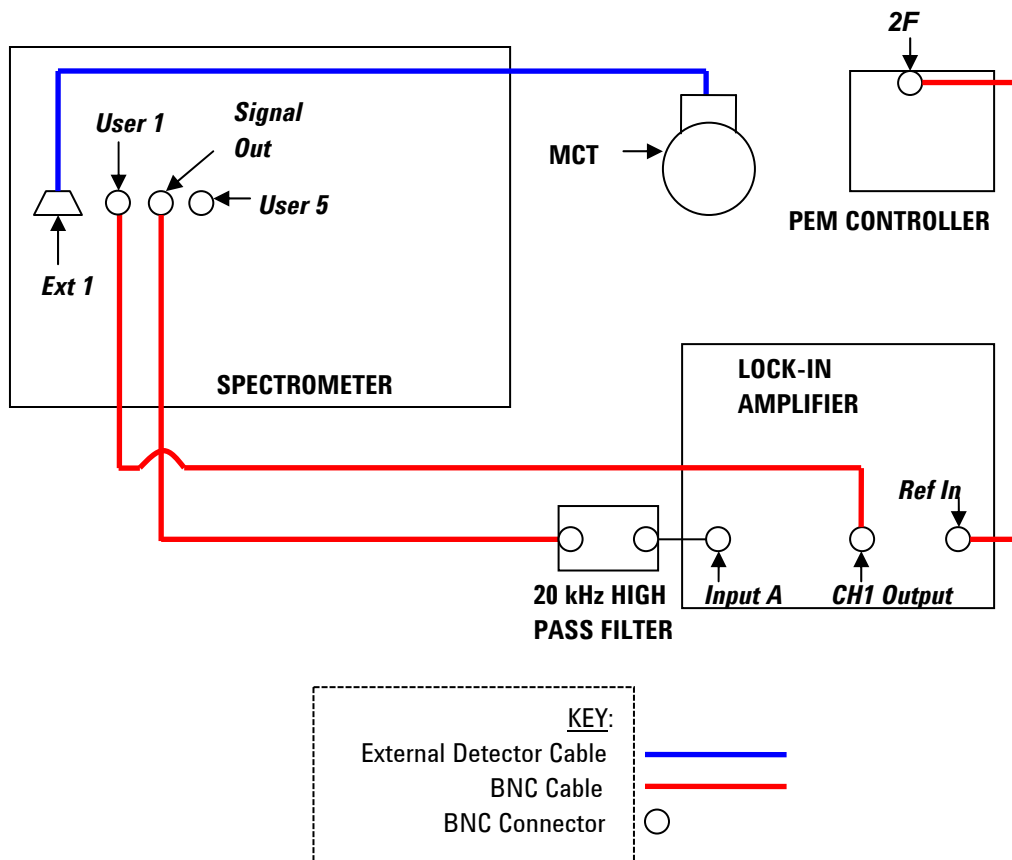


Figure 10. Schematic of cabling for rapid scan PM-IRRAS measurement

- 2 Go to **Collect > Method Editor...** Select the following parameters:
 - a Common Settings:
 - Sample Scans = 64
 - Resolution = 8 cm⁻¹

b Collect:

- Speed = 12.5 KHz
- UDR = 2
- Filter = 3.2 KHz
- Sensitivity = 1
- Interferogram Symmetry = Asymmetric
- Signal = User 1
- Dual A/D Collect = checked

c Spectrometer Configuration:

- IR Source = Rear: Mid-IR
- Beam Path = Right
- Detector = Ext. 1
- Aperture = 2 cm⁻¹
- Attenuator = 50%

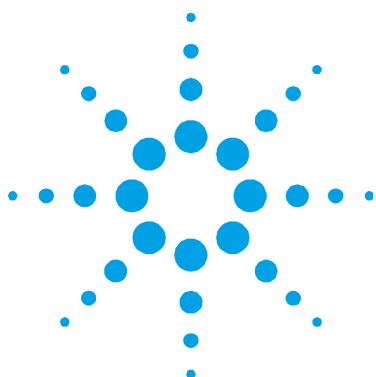
3 SR830 Lock-in Amplifier**a Time constant = 30 µs****b Slope/Oct = 18 dB****c Sensitivity = 1 x 1 V (variable)****d Reserve = Normal****e Signal Input**

- Input connector = A
- Coupling = AC
- Ground = Ground

f Channel 1

- Display = X
- Ratio = off
- Expand = off
- Output = X
- Press **CH1 OFFSET Auto** before each measurement

- g** Reference = Freq
 - h** Trig = Pos Edge
 - i** Press AUTO Phase before each measurement
- 4** Click **Signal Monitor**
 - 5** Press and hold **CH1 OFFSET AUTO** on the lock-in amplifier to remove any DC offset from the interferogram.
 - 6** Adjust the sensitivity on the lock-in amplifier to achieve an interferogram signal of 1–9 V.
 - 7** Unplug the BNC cable from User 1 and plug it back into User 5.
 - 8** On the Collect page of Method Editor make Signal = **AUTO**.
 - 9** Click **Scan**.
 - 10** At the end of the collect two spectra called Signal Channel and Reference Channel will be added to the spreadsheet. The Signal Channel corresponds to the AC spectrum ($I_p - I_s$) from the lock-in amplifier. The Reference Channel corresponds to the DC spectrum ($I_p + I_s$).
 - 11** Select the Signal Channel spectrum in the spreadsheet and go to **Transforms > Spectral Calculator > Paired Spectra**. A 'Spectral Arithmetic: Paired Spectra Operations' dialog box will be displayed. Select **{/} Divide by Reference**. Select **Use Current** and then select the Reference Channel spectrum from the list of spectra in the bottom right corner. Click **Add**.
 - 12** The resultant spectrum should look similar to that shown in Figure 9 on Page 31.



5. Maintenance

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This chapter includes maintenance procedures for the Polarization Modulation Infrared Reflection Absorption Spectroscopy (PM-IRRAS) Accessory that may be carried out by an operator. Any maintenance procedures not specifically mentioned in this chapter or in the Help should be carried out only by an Agilent-trained, Agilent-qualified or Agilent-authorized customer service representative.

WARNING



Electrical Shock Hazard

This accessory contains electrical circuits, devices, and components operating at dangerous voltages. Contact with these circuits, devices and components can cause death, serious injury, or painful electrical shock. Always follow the procedures described by Agilent.

Cleaning

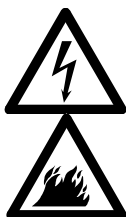
Any spills on the PM-IRRAS should be immediately wiped up.

The **exterior** surfaces of the PM-IRRAS should be kept clean. All cleaning should be done with a soft cloth. If necessary, this cloth can be dampened with water or a mild detergent. Do not use organic solvents or abrasive cleaning agents.

Fuses

The only user-accessible fuse in the PM-IRRAS is the mains inlet fuse for the PEM controller. Refer to the PEM-100 User Manual for details. Always turn off the power and remove the power cord before replacing fuses. Fuses should be replaced only with the same type and rated fuses as specified on the rear of the controller.

WARNING



Shock and Fire Hazards

This accessory contains electrical circuits, devices, and components operating at dangerous voltages. Contact with these circuits, devices and components can cause death, serious injury, or painful electrical shock. To prevent reduced safety protection or unwanted fusing, ALWAYS ensure that fuses are only replaced with fuses of the correct type and rating.

To check a fuse:

- 1 Disconnect the instrument from the mains power supply.
- 2 Remove the fuse holder from the IEC mains power inlet connector with a small flat blade screwdriver.
- 3 Check that the fuse is the correct type and is not damaged. If necessary, replace the fuse.
- 4 Fit the fuse holder back into the IEC mains power inlet connector.
- 5 Reconnect the instrument to the mains power supply.

NOTE

If a fuse repeatedly blows, it may indicate other problems with the accessory. A service call may be required.

Spare parts

For information about spare parts and their part numbers, refer to the Agilent website, www.agilent.com

In This Guide

The guide describes the following:

- Safety Practices and Hazards
- Introduction
- Hardware
- Basic Operation
- Maintenance

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