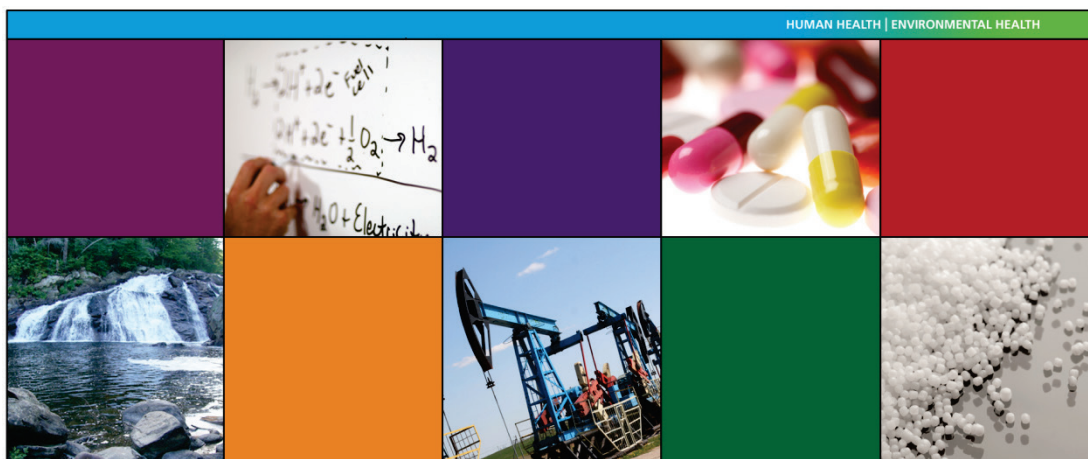


# Spotlight 150



## User's Guide

## Release History

| Part Number | Release | Publication Date |
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# Contents

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|   |           |
|---|-----------|
| <b>Introduction.....</b>  | <b>5</b>  |
| About this Manual .....   | 6         |
| Conventions Used in this Manual .....                               | 7         |
| Notes, Cautions and Warnings.....                                   | 7         |
| Definitions.....  | 10        |
| Related Documents.....  | 10        |
| Requirements for using the Spotlight 150.....                       | 10        |
| Connecting the PC to the Local Area Network.....                    | 10        |
| <b>Warnings and Safety Information .....</b>                        | <b>11</b> |
| The Spotlight 150.....  | 12        |
| Summary .....   | 13        |
| General Operating Conditions.....                                   | 14        |
| Electrical Safety .....   | 15        |
| Location and Ventilation .....                                      | 16        |
| Warning Labels .....  | 17        |
| Microscope Safety Labels .....                                      | 17        |
| Warning Signs on the Microscope .....                               | 18        |
| Lifting the Spotlight 150 .....                                     | 19        |
| EMC Compliance .....  | 20        |
| System Requirements.....  | 21        |
| Electrical Requirements.....  | 21        |
| Safety Specifications.....  | 22        |
| Microscope.....   | 22        |
| <b>Overview of the Spotlight 150 .....</b>                          | <b>23</b> |
| A Guided Tour of the Spotlight 150 .....                            | 24        |
| Connections.....  | 25        |
| Operation .....   | 26        |
| The Optical System .....  | 27        |
| Visible Light Optics .....  | 27        |
| Infrared Optics .....   | 30        |
| <b>Getting Ready to Use the Spotlight 150 .....</b>                 | <b>33</b> |
| Before Using the Spotlight 150.....                                 | 34        |
| Cooling the MCT Detector .....                                      | 35        |
| Setting up the Spotlight 150 .....                                  | 38        |
| Focusing the Microscope .....                                       | 39        |
| Fitting an Attenuator to the Spectrometer .....                     | 41        |
| Setting Scan Parameters .....                                       | 42        |
| <b>Preparing Samples .....</b>                                      | <b>45</b> |
| Preparing Samples .....   | 46        |
| Tools for Sample Preparation.....                                   | 47        |
| Tools Provided with the Spotlight 150 .....                         | 47        |
| Other Useful Tools.....   | 48        |
| Specialized Accessories .....                                       | 49        |
| Items to Have Available .....                                       | 49        |
| Common Window Materials.....  | 50        |
| Techniques for Preparing Samples for Transmission Measurements..... | 51        |
| Flattening Solids .....   | 51        |
| Slicing Samples from Solids .....                                   | 52        |
| Polymers.....   | 53        |
| Particles.....  | 55        |
| Fibers .....  | 56        |
| Fibrous Solids.....   | 56        |
| Coatings on Substrates .....  | 57        |
| Liquids.....  | 57        |
| <b>Techniques for Collecting Spectra .....</b>                      | <b>59</b> |
| Techniques for Collecting Spectra.....                              | 60        |

|  |            |
|--|------------|
| Using the Compression Cell .....                                   | 60         |
| Collecting a Spectrum from a Thick Sample .....                    | 61         |
| Collecting a Spectrum in an Inert Atmosphere .....                 | 62         |
| Viewing a Sample with the Visible Polarizer Accessory.....         | 64         |
| The Theory of Light Polarization.....                              | 64         |
| Applications .....   | 65         |
| Equipment.....   | 66         |
| Collecting IR Spectra Using the Infrared Polarizer Accessory ..... | 68         |
| Using the Polarizer .....  | 69         |
| Collecting Spectra With the ATR Objective .....                    | 70         |
| ATR Objective Specification .....                                  | 72         |
| Fitting the ATR Crystal Holder to the Microscope.....              | 73         |
| Measuring the Energy in Reflectance Mode .....                     | 75         |
| Adjusting the Height of the Crystal .....                          | 76         |
| Centering the Crystal.....   | 77         |
| Checking the Infrared Alignment.....                               | 78         |
| Removing the ATR Crystal Holder .....                              | 79         |
| Fitting a Crystal Assembly to the ATR Objective .....              | 80         |
| Manual ATR Objective .....   | 80         |
| Automated ATR Objective.....                                       | 80         |
| Cleaning the ATR Objective Crystal .....                           | 82         |
| Auto ATR Cleaning Procedure.....                                   | 83         |
| Materials Needed .....   | 83         |
| Cleaning Procedure .....   | 83         |
| Collecting Spectra with the ATR Crystal.....                       | 88         |
| Manual ATR Objective .....   | 88         |
| Automated ATR Objective.....                                       | 89         |
| Reflectance FT-IR Microspectroscopy .....                          | 91         |
| Diffuse Reflectance .....  | 91         |
| Specular Reflectance .....   | 92         |
| Reflection-Absorption .....  | 93         |
| <b>Maintenance.....</b>  | <b>94</b>  |
| Maintenance.....   | 95         |
| Inspecting the Microscope .....                                    | 95         |
| Protecting the Microscope.....                                     | 96         |
| Cleaning .....   | 96         |
| Cleaning the Cover.....  | 96         |
| Replacing the Microscope Fuse .....                                | 97         |
| Service.....   | 98         |
| Accessories .....  | 99         |
| Sample Preparation Tools.....                                      | 99         |
| Electrical Connections .....                                       | 100        |
| Fitting the Plug .....   | 100        |
| Connecting the Microscope to the Electrical Supply .....           | 101        |
| Connecting the Microscope to the Spectrometer.....                 | 101        |
| <b>Appendices.....</b>   | <b>102</b> |
| Appendix 1: Decontamination and Cleaning.....                      | 103        |
| Appendix 2: WEEE Instructions for PerkinElmer Products .....       | 104        |
| Index.....   | 105        |

# **Introduction**

## ***About this Manual***

---

This introduction gives you information about this manual, to enable you to use it effectively when learning to operate the Spotlight 150.

Before you start using your Spotlight 150 we recommend that you:

- Read the *Warnings and Safety Information* starting on page 11;
- Read the rest of this *Introduction* and become aware of conventions and requirements;
- Read *Overview of the Spotlight 150* starting on page 23;
- Read *Getting Ready to Use the Spotlight 150* starting on page 33.

Four chapters in this User's Guide contain information on using and maintaining your Spotlight 150:

- *Getting Ready to Use the Spotlight 150* gives you information on how to set up your Spotlight 150 at the beginning of the day's work, to make sure that it is working properly.
- *Preparing Samples* describes techniques for preparing many types of microscopic samples. It includes descriptions of the sample preparation tools provided with, or available for, the Spotlight 150.
- *Techniques for Collecting Spectra* describes how you can use accessories and different techniques to collect spectra.
- *Maintenance* contains maintenance information. It gives you information on how to care for the Spotlight 150 and about the performance checks that we recommend that you perform routinely.

## ***Conventions Used in this Manual***

---

Normal text is used to provide information and instructions. *Italic* text is used to provide commentary. **Bold** text refers to text that is displayed on the screen.

UPPERCASE text, for example ENTER or ALT, refers to keys on the PC keyboard. "+" is used to show that you have to press two keys at the same time, for example, ALT+F.

All eight digit numbers are PerkinElmer part numbers unless stated otherwise.

"Spectrometer" refers to the Frontier IR Systems, Spectrum 400 Series, Spectrum 100 Series, Spectrum 65 or Spectrum One spectrometer supplied with your Spotlight 150.

### ***Notes, Cautions and Warnings***

Three terms, in the following standard formats, are also used to highlight special circumstances and warnings.

|   |
|---|
| <p><b>NOTE:</b> A note indicates additional, significant information that is provided with some procedures.</p> |
|---|

|                  |   |
|------------------|---|
| <b>CAUTION</b>   | <p>We use the term <b>CAUTION</b> to inform you about situations that could result in <b>serious damage to the instrument</b> or other equipment. Details about these circumstances are in a box like this one.</p>   |
| <p><b>D</b></p>  | <p><b>Caution (Achtung)</b><br/>Bedeutet, daß die genannte Anleitung genau befolgt werden muß, um einen <b>Geräteschaden</b> zu vermeiden.</p>  |
| <p><b>DK</b></p> | <p><b>Caution (Bemærk)</b><br/>Dette betyder, at den nævnte vejledning skal overholdes nøje for at undgå en <b>beskadigelse af apparatet</b>.</p>   |
| <p><b>E</b></p>  | <p><b>Caution (Advertencia)</b><br/>Utilizamos el término <b>CAUTION (ADVERTENCIA)</b> para advertir sobre situaciones que pueden provocar <b>averías graves en este equipo</b> o en otros. En los recuadros como éste se proporciona información sobre este tipo de circunstancias.</p>              |
| <p><b>F</b></p>  | <p><b>Caution (Attention)</b><br/>Nous utilisons le terme <b>CAUTION (ATTENTION)</b> pour signaler les situations susceptibles de provoquer de <b>graves détériorations de l'instrument</b> ou d'autre matériel. Les détails sur ces circonstances figurent dans un encadré semblable à celui-ci.</p> |
| <p><b>I</b></p>  | <p><b>Caution (Attenzione)</b><br/>Con il termine <b>CAUTION (ATTENZIONE)</b> vengono segnalate situazioni che potrebbero arrecare <b>gravi danni allo strumento</b> o ad altra apparecchiatura. Troverete informazioni su tali circostanze in un riquadro come questo.</p>                           |
| <p><b>NL</b></p> | <p><b>Caution (Opgelet)</b><br/>Betekent dat de genoemde handleiding nauwkeurig moet worden opgevolgd, om <b>beschadiging van het instrument</b> te voorkomen.</p>  |
| <p><b>P</b></p>  | <p><b>Caution (Atenção)</b><br/>Significa que a instrução referida tem de ser respeitada para evitar a <b>danificação do aparelho</b>.</p>  |



**WARNING**

We use the term **WARNING** to inform you about situations that could result in **personal injury** to yourself or other persons. Details about these circumstances are in a box like this one.

**D****Warning (Warnung)**

Bedeutet, daß es bei Nichtbeachten der genannten Anweisung zu einer **Verletzung** des Benutzers kommen kann.

**DK****Warning (Advarsel)**

Betyder, at brugeren kan blive **kvæstet**, hvis anvisningen ikke overholdes.

**E****Warning (Peligro)**

Utilizamos el término **WARNING (PELIGRO)** para informarle sobre situaciones que pueden provocar **daños personales** a usted o a otras personas. En los recuadros como éste se proporciona información sobre este tipo de circunstancias.

**F****Warning (Danger)**

Nous utilisons la formule **WARNING (DANGER)** pour avertir des situations pouvant occasionner des **dommages corporels** à l'utilisateur ou à d'autres personnes. Les détails sur ces circonstances sont données dans un encadré semblable à celui-ci.

**I****Warning (Pericolo)**

Con il termine **WARNING (PERICOLO)** vengono segnalate situazioni che potrebbero provocare **incidenti alle persone**. Troverete informazioni su tali circostanze in un riquadro come questo.

**NL****Warning (Waarschuwing)**

Betekent dat, wanneer de genoemde aanwijzing niet in acht wordt genomen, dit kan leiden tot **verwondingen** van de gebruiker.

**P****Warning (Aviso)**

Significa que a não observância da instrução referida poderá causar um **ferimento** ao usuário.

## **Definitions**

**OPERATOR:** Person operating the equipment for its intended purpose.

**RESPONSIBLE BODY:** Individual or group responsible for the use and maintenance of the equipment and for ensuring that the **OPERATORS** are adequately trained.

## **Related Documents**

### **Manuals for Spectrometer**

Information on using your spectrometer can be found in the manuals that were supplied with it. The *IR & Raman Manuals CD* (L1050002) includes instrument manuals for the Frontier IR Systems, Spectrum 400 Series and Spectrum 100 Series spectrometers. The *Spectrum Two Manuals CD* (L1050242) contains the instrument manual for the Spectrum Two spectrometer.

### **Spectrum Software**

Spectrum software has an on-screen Help system, which you can access by selecting **Contents** from the Help menu, by using the F1 key, or by clicking the **Help** button in a dialog. The Help information assumes that you are familiar with the hardware components of the Spotlight 150 microscope contained in this guide.

## **Requirements for using the Spotlight 150**

We assume that the Spotlight 150 has been properly set up and aligned. This installation must be performed by a PerkinElmer Service Representative.

Ensure that your PC meets the minimum requirements for hardware and software set out in the *PC Requirements* section of the *Spectrum Administrator's Guide* (L1050095).

## **Connecting the PC to the Local Area Network**

The PC provided is connected point-to-point to the Spotlight 150. If you wish to connect the PC to your local area network to enable transfer of data, we recommend that you contact your local PerkinElmer Service Representative.

# **Warnings and Safety** **Information**

## ***The Spotlight 150***

---

The Spotlight 150 consists of a microscope, FT-IR spectrometer and a PC.



**Figure 1 Spotlight 150 – Microscope and Frontier IR System**



**Figure 2 Spotlight 150 – Microscope and Spectrum Two System**

## Summary

---

The PerkinElmer Spotlight 150 has been designed to comply with a wide variety of international standards governing the safety of laboratory equipment. In routine use, the Spotlight 150 poses virtually no risk to you. If you take some simple, common-sense precautions, you can make sure that you maintain the continued safe operation of the Spotlight 150.

- DO make sure that all parts of the Spotlight 150 are properly connected to the electrical supply; in particular, make sure that the ground (earth) wires are securely connected.
- DO disconnect the electrical power supply before opening the main cover of the microscope and spectrometer.
- DO keep the microscope dry. Avoid spilling liquid into the microscope and spectrometer. Clean all external spills immediately. If anything that is spilled enters the main body of the microscope or spectrometer, switch off the power and call a PerkinElmer Service Representative.
- If your Spotlight 150 is fitted with an MCT (mercury cadmium telluride) detector, DO wear safety glasses and protective gloves when you are filling the detector dewar in the microscope with liquid nitrogen. Slowly pour the liquid nitrogen into the dewar. Stand back from the detector during filling because liquid nitrogen may be expelled from the dewar flask. Use only liquid nitrogen.
- DO NOT use a flammable gas to purge the spectrometer or microscope. The spectrometer contains a hot lamp, and fire or explosion may result. Use only clean, dry, oil-free nitrogen or air to purge the instrument.
- DO read the more detailed information on safety in the following pages.



*Removal of covers and fixtures not specified in this manual may result in hazardous radiation exposure.*

*Use of controls or adjustments or performance of procedures other than those specified herein may result in hazardous radiation exposure.*

## ***General Operating Conditions***

---

For information about the spectrometer, refer to the User's Guide supplied with the instrument. The *IR & Raman Manuals CD* (L1050002) includes instrument manuals for the Frontier IR Systems, Spectrum 400 Series and Spectrum 100 Series spectrometers.

The microscope has been designed and tested in accordance with PerkinElmer specifications and in accordance with the safety requirements of the International Electrotechnical Commission (IEC). The microscope conforms to IEC publication 61010–1 ("Safety requirements for electrical equipment for measurement, control, and laboratory use") as it applies to IEC Class 1 (earthed) appliances and therefore meets the requirements of EC low voltage directive 2006/95/EC.

Only use the microscope indoors and under the following conditions:

Temperature        15 °C to 35 °C  
Relative humidity   80% maximum (non-condensing)

If possible, avoid any adjustment, maintenance and repair of the opened, operating instrument. If any adjustment, maintenance and repair of the opened instrument is necessary, this must be done by a skilled person who is aware of the hazard involved.

Whenever it is likely that the microscope is unsafe, make it inoperative. The microscope may be unsafe if it:

- Shows visible damage;
- Fails to perform the intended measurement;
- Has been subjected to prolonged storage in unfavorable conditions;
- Has been subjected to severe transport stresses.

The microscope has been designed to be safe under the following conditions:

- Indoor use;
- Altitude up to 2000 m;
- Ambient temperatures of 5 °C to 40 °C;
- A maximum ambient relative humidity of 80% for temperatures up to 31 °C decreasing linearly to 50% relative humidity at 40 °C;
- Mains fluctuations not exceeding  $\pm 10\%$  of the nominal voltage. For example, 230 V  $\pm$  10%.



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

## ***Electrical Safety***

---

Connect the microscope to a power supply line that includes a switch or other adequate means of disconnection from the electricity supply.

Plug the microscope into electricity-supply sockets that are provided with a protective ground (earth) connection only. The microscope must be earthed.

If fuses need replacing, use only those with the required current rating and of the specified type. Do not use makeshift fuses and do not short-circuit fuse holders.

When the microscope is connected to its electricity supply, terminals may be live. Removing covers other than those which can be removed by hand is likely to expose live parts.

**NOTE:** There are no user-serviceable parts in the microscope.

Capacitors inside the microscope may still be charged even if the microscope has been disconnected from all voltage sources.

Disconnect the microscope from all voltage sources before opening for any adjustment, replacement, maintenance or repair. Any adjustment, replacement, maintenance or repair must be performed by a PerkinElmer Service Representative.

The microscope must only be connected to equipment meeting the requirements of IEC 61010-1 (Safety requirements for electrical equipment for measurement, control and laboratory use – general requirements) or IEC 60950 (Safety of information technology equipment).



**WARNING**

*Any interruption of the protective ground (earth) conductor inside or outside the microscope or disconnection of the protective ground (earth) terminal can make the microscope dangerous.*

## ***Location and Ventilation***

---

The Spotlight 150 is installed by a PerkinElmer Service Representative, who will be able to advise on the positioning of the system. To allow for adequate cooling, the system should not be sited near to room heating equipment, for example, central-heating radiators. There should be a minimum gap of at least 15 cm (6 inches) from the top and side surfaces of the microscope to permit adequate cooling.



**WARNING**

*Make sure that the switch at the electrical supply inlet at the rear of the microscope is not obstructed.*



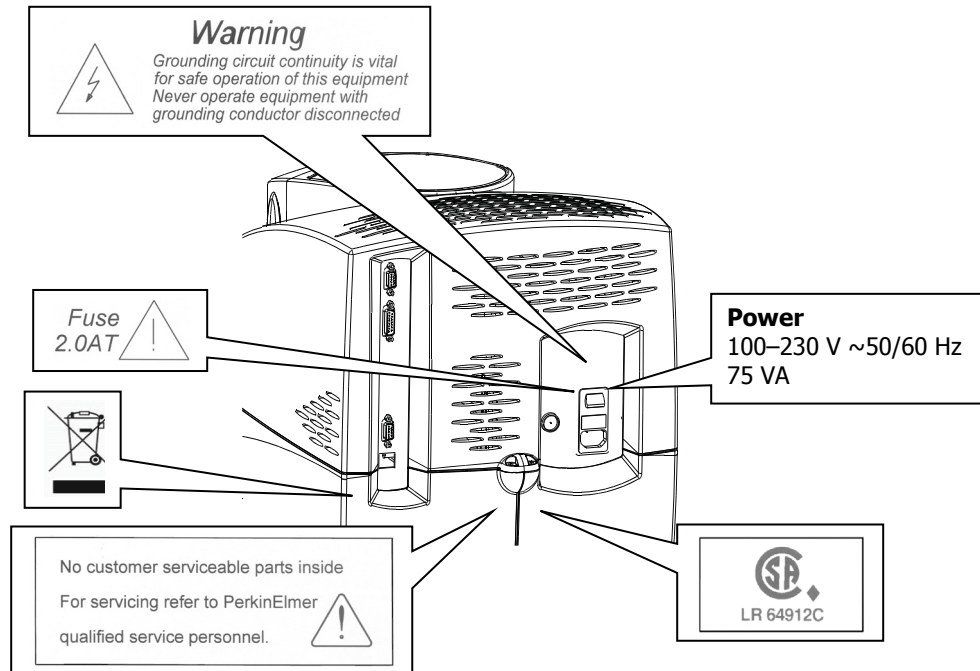
## Warning Labels



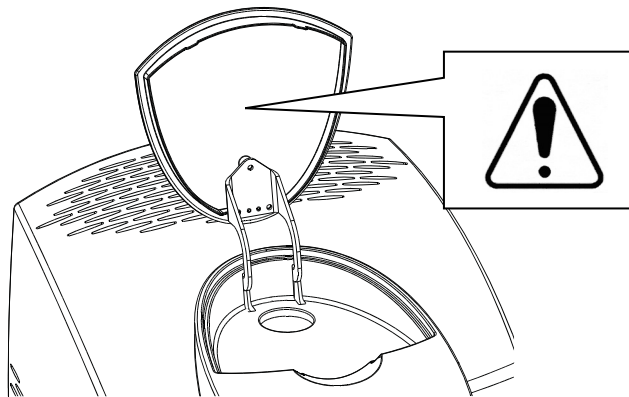
*When this label is attached to an instrument it means "Caution, risk of danger". Refer to the manual to find out the nature of the potential hazard and any actions which have to be taken.*

## Microscope Safety Labels

The following safety labels are fixed to the microscope.



**Figure 3 Rear of Microscope**



**Figure 4 Inside Dewar lid – MCT detector versions only**

**NOTE:** See *Cooling the MCT Detector* on page 35 for information on how to fill the Dewar.

## **Warning Signs on the Microscope**



*Caution, risk of electric shock.*



*Caution, risk of danger.*

*Refer to accompanying documents to find out the nature of the potential hazard and any actions which have to be taken.*

## ***Lifting the Spotlight 150***

---



**WARNING**

*The microscope and spectrometer are heavy precision instruments, so two people are required for safe handling.*

*Consult the local codes of practice issued by safety advisors before attempting to lift these instruments.*

*Take care not to injure yourself or others, or to drop the instruments.*

The microscope weighs approximately 32 kg.

- Lift the microscope only by the base.
- Do not attempt to lift it by the stage, cassegrain assembly or other attachments.

The spectrometer weighs approximately 34 kg unpacked and has a lifting recess on either side.

Do not move the Spotlight 150 after it has been installed without consulting your local PerkinElmer Service Representative.

## ***EMC Compliance***

---

### ***EC directive***

The Spotlight 150 has been designed and tested to meet the requirements of the EMC Directive 2004/108/EC.

The Spotlight 150 complies with the EMC standard EN61326, (EMC standard for electrical equipment for measurement, control and laboratory use) and EN55011 (ISM) class A (rf emissions).

### ***FCC rules and regulations***

This product is classified as a digital device used exclusively as industrial, commercial, or medical test equipment. It is exempt from the technical standards specified in Part 15 of the FCC Rules and Regulations, based on Section 15.103(c).

## ***System Requirements***

---

Give attention to the following points before installing the Spotlight 150.

### ***Electrical Requirements***

The power consumption of the microscope does not exceed 75 VA.

The nominal power consumption of the spectrometer is 120 VA (65 VA for the Spectrum Two).

The line supply must be within 10% of the nominal voltage. For example, 230 V  $\pm$  10%.

If possible, do not connect any parts of the Spotlight 150 to circuits that have heavy duty equipment, such as large motors, connected.

If possible, do not use photocopiers, discharge lamps, radio transmitters, and other equipment with large or frequent transient loads, on the same supply circuit.

### ***Microscope***

The microscope can operate on electricity supplies of 50 or 60 Hz and in a voltage range of 100 to 240 V. The primary fuse (2 A T, 250 V, part number 04970839) for the microscope is in the drawer on the mains inlet connector.

|  |
|--|
| <b>NOTE:</b> No voltage selection is required. |
|--|

### ***Site Requirements***

A minimum bench space of 170 × 75 cm (68 × 30 inches) is required to accommodate the microscope, spectrometer, PC and ancillaries.

To get the best performance from your Spotlight 150:

- Place the Spotlight 150 in an environment that is relatively dust-free.
- Make sure that the bench top is free from vibrations or mechanical shocks, and is flat and level.
- Do not place the Spotlight 150 near to room heating equipment, for example central heating radiators.

Leave a gap of at least 15 cm (6 inches) between any vertical obstacle and the sides of the microscope to permit an adequate flow of cooling air.

## ***Safety Specifications***

---

### ***Microscope***

|              |   |
|--------------|---|
| Power supply | 100–230 V $\pm$ 10%, 50–60 Hz $\pm$ 10% |
| Primary fuse | 2.0 A T (time-lag), 250 V               |
| Weight       | 32 kg (without the sample stage)        |

# **Overview of the** **Spotlight 150**

## ***A Guided Tour of the Spotlight 150***

---

The Spotlight 150 enables you to collect IR spectra from extremely small samples. The PerkinElmer cassegrain collection optics give high-performance infrared microspectroscopy. The microscope includes a camera and viewing system that magnifies the visible-light image of the sample so that you can see and position a point of interest. The image of your sample is displayed on the Setup Microscope tab in Spectrum software. Spectrum enables you to control the operation of your Spotlight 150, and collect IR spectra from the sample.

The microscope includes the following features:

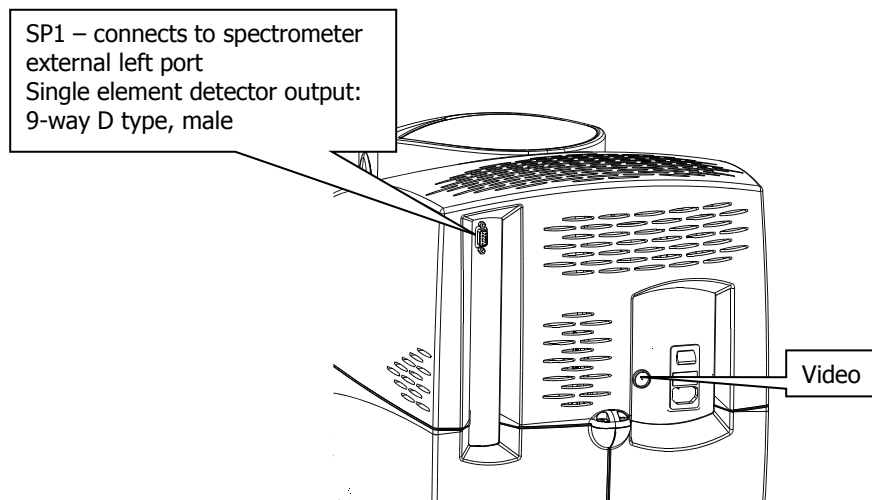
- A high-performance cassegrain mirror system; this has a wide collection angle (high numerical aperture) and is highly efficient in collecting infrared radiation for microspectroscopy.
- Spectra can be collected in either reflectance or transmittance sampling modes.
- Internal coaxial LED illumination, with variable intensity.
- An automatic infrared aperture that closes to the size and rotation selected when you select a scan or monitor command.
- Spectrum software controls the operation of the microscope: for example, the illumination of the sample, adjusting the correction, and changing to reflectance or transmittance. Some versions of the microscope also feature LED lighting of the stage area which is controlled from the software.
- An optional micro-ATR objective for data collection from optically thick and non-reflective samples. Manual and automatic ATR objectives are available.
- Data collection (including saving the visible image) and post-run processing for IR spectra.
- The same cassegrain is used to send both infrared radiation and visible light to the remote aperture. The microscope continuously views and monitors infrared concurrently; there is no beam path switching between visible and infrared. Spectrum software allows you to view the visible image and the IR spectrum simultaneously.
- The lower cassegrain can be removed. This provides space for thick samples to be studied in reflectance mode.
- An InGaAs (indium gallium arsenide), a DTGS (deuterated triglycine sulfate), or an MCT (mercury cadmium telluride) MIR detector is fitted.



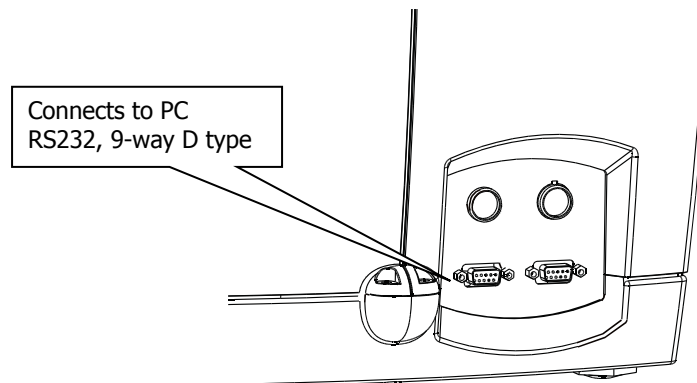
## Connections

---

Figure 5 and Figure 6 show the connections on the microscope.



**Figure 5 Connections (top rear of microscope)**



**Figure 6 Connections (bottom rear of microscope)**

## Operation

---

The table below lists the modes of operation that should be used for particular samples.

|                    |  |
|--------------------|--|
| Transmittance      | <p>Single areas of thin solids, fibers and films. The optimal aperture range depends upon the detector type, as follows:</p> <p><b>MCT detector:</b> Between 10 <math>\mu\text{m}</math> and 100 <math>\mu\text{m}</math>.</p> <p><b>DTGS detector:</b> Between 50 <math>\mu\text{m}</math> and 200 <math>\mu\text{m}</math>.</p> <p><b>InGaAs detector:</b> Between 10 <math>\mu\text{m}</math> and 200 <math>\mu\text{m}</math>.</p> |
| Reflectance        | <p>Single areas of coatings and thick solids. The optimal aperture range depends upon the detector type, as follows:</p> <p><b>MCT detector:</b> Between 10 <math>\mu\text{m}</math> and 100 <math>\mu\text{m}</math>.</p> <p><b>DTGS detector:</b> Between 50 <math>\mu\text{m}</math> and 200 <math>\mu\text{m}</math>.</p> <p><b>InGaAs detector:</b> Between 10 <math>\mu\text{m}</math> and 200 <math>\mu\text{m}</math>.</p>     |
| With ATR objective | Thick, non-reflecting samples.   |

**NOTE:** In general, the larger the aperture setting, the better the spectral quality. Sample sizes may be larger than the maximum aperture sizes given in the table above.

## ***The Optical System***

---

The microscope has optics for infrared microspectroscopy and visible light. A video camera enables you to view the sample. The two systems intersect at the aperture. When a sample on the sample stage is in focus, its conjugate image is focused at the remote aperture.

Spotlight 150 enables you to select between transmittance and reflectance operation. This section describes what happens within the optical system when the system changes from visible light to infrared mode in transmittance and reflectance operation.

Dichroic mirrors form part of the optical system. The dichroic mirrors used in the microscope reflect infrared and transmit visible light.

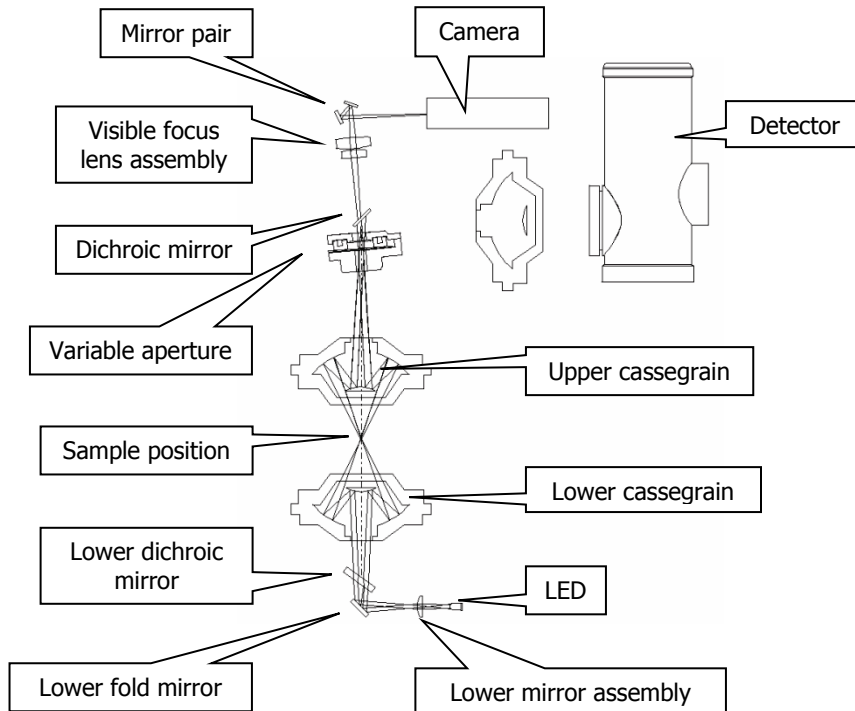
### ***Visible Light Optics***

When you view a sample with the video camera, you are really looking at the conjugate image of the sample, located at the remote aperture (Figure 7).

## Visible light optics in transmittance

When the microscope is in viewing mode in transmittance (Figure 7):

- The lower fold mirror, beneath the cassegrains, receives light from the LED source and directs it up through the lower dichroic mirror onto the lower cassegrain.
- The lower cassegrain condenses the beam to an appropriate size for a microscopic sample and focuses it at the sample position.
- The upper cassegrain collects light from the sample and sends it upward through the aperture and the upper dichroic mirror.



**Figure 7 Path of the visible beam for viewing a sample in transmittance**

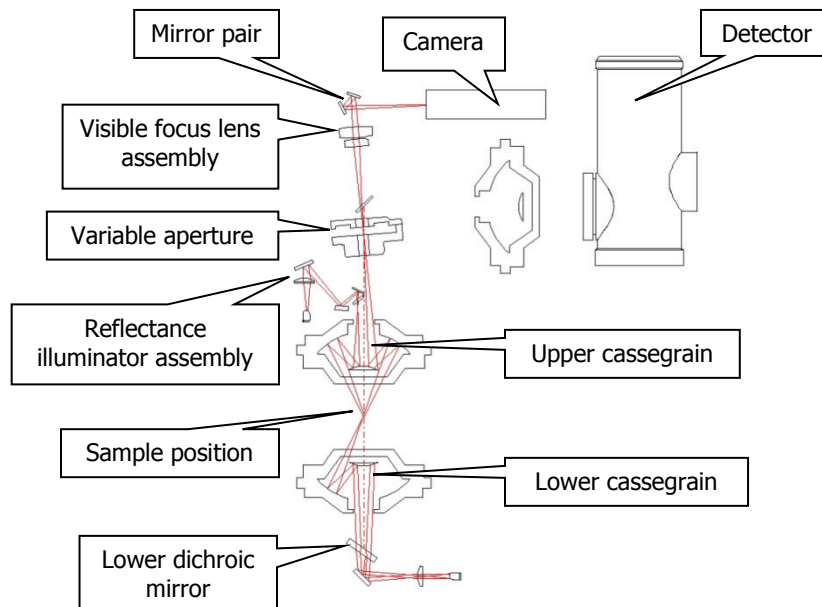
**NOTE:** Figure 7, above, illustrates a Spotlight 150 with an MCT detector. Other detector types may look different, but the optical arrangement is the same in all cases.

## Visible light optics in reflectance

When the microscope is in viewing mode in reflectance (Figure 8):

- The reflectance illuminator assembly directs light from the upper LED down through one side of the cassegrain onto the sample.
- The upper cassegrain collects the reflected light from the sample and sends it upward through the aperture.

The lower “transmission” illuminator is also active in reflectance mode, giving simultaneous illumination from above and below.



**Figure 8 Path of the visible beam for viewing a sample in reflectance**

**NOTE:** Figure 8, above, illustrates a Spotlight 150 with an MCT detector. Other detector types may look different, but the optical arrangement is the same in all cases.

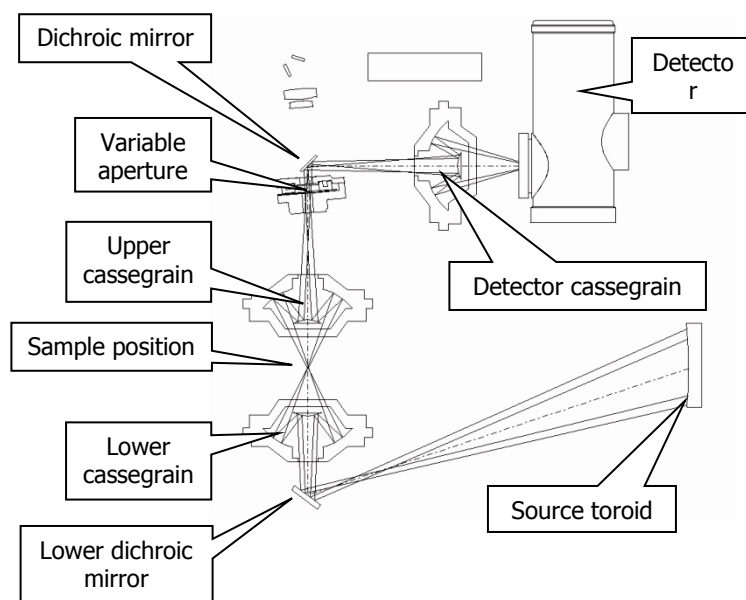
## Infrared Optics

The upper cassegrain is used for both visible light and infrared radiation. For this reason, when you adjust the sample position so that the visible image of the sample is in focus, the sample is also correctly positioned for collecting infrared spectra. Similarly, when you adjust the aperture so that the required area of the sample is isolated visually, you have also isolated the area of the sample from which the IR spectrum is to be collected.

### IR optics in transmittance

IR optics in transmittance differs from when viewing a sample in transmittance as follows (Figure 9):

- Instead of receiving light from the illuminator, light from the spectrometer is reflected off the toroid onto the lower dichroic mirror which sends it through the lower cassegrain.
- The upper dichroic mirror reflects the beam onto the detector cassegrain.
- The detector cassegrain focuses the beam on to the detector.



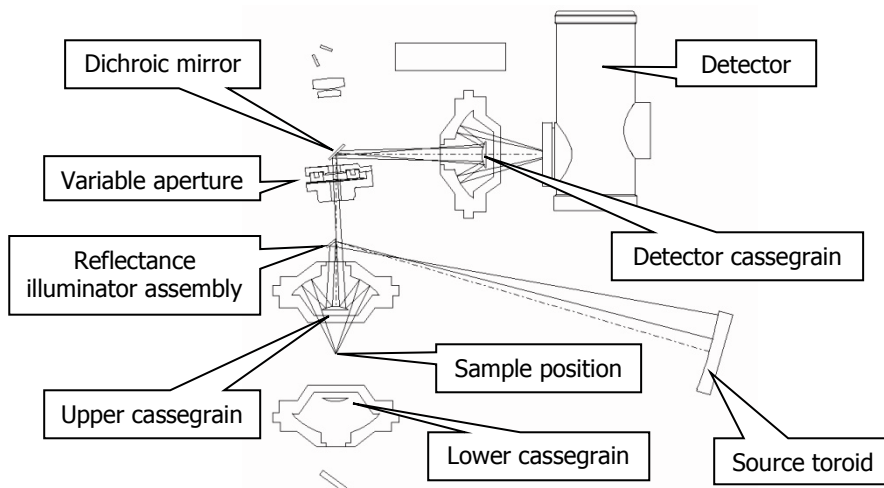
**Figure 9 Path of the infrared beam for collecting an image in transmittance**

**NOTE:** Figure 9, above, illustrates a Spotlight 150 with an MCT detector. Other detector types may look different, but the optical arrangement is the same in all cases.

## IR optics in reflectance

IR optics in reflectance differs from viewing in reflectance as follows (Figure 10):

- The toroid moves to send the beam to the Reflectance illuminator assembly dichroic mirror, which sends it through the upper cassegrain.
- The beam is reflected off the sample and back through the other side of the cassegrain, toward the remote aperture.
- The detector cassegrain focuses the beam on to the detector.



**Figure 10 Path of the infrared beam for collecting IR spectra in reflectance**

**NOTE:** Figure 10, above, illustrates a Spotlight 150 with an MCT detector. Other detector types may look different, but the optical arrangement is the same in all cases.





**Getting Ready to Use the**  
**Spotlight 150**

## ***Before Using the Spotlight 150***

---

Before you use the Spotlight 150 you must:

- Make sure the spectrometer is switched on;
- Switch the microscope on;
- Cool the detector (MCT detector versions only);
- Set up the microscope;
- Focus the microscope.

We recommend that you use these procedures at the beginning of the day's work, or any time the Spotlight 150 has not been in use or has been used by others.

## Cooling the MCT Detector

---

If the microscope is fitted with an MCT (Mercury Cadmium Telluride) detector, the detector must be cooled to  $-196^{\circ}\text{C}$  before you collect spectra. It is mounted in a dewar that can be filled with liquid nitrogen. As you fill the dewar, the temperature of the detector drops, and the preamp supplies power to the detector. Use the following procedure to cool the MCT detector.

**NOTE:** If your Spotlight 150 is fitted with a DTGS or InGaAs detector, cooling is not required.



**WARNING**

*The extremely low temperature of liquid nitrogen can burn skin and eyes. Avoid exposure by wearing heavy gloves and safety goggles whenever you work with it.*



**WARNING**

*When liquid nitrogen warms to room temperature, nitrogen gas vaporizes so rapidly that resulting pressures can send a funnel or detector cap suddenly and forcefully shooting upward from the top of the microscope.*



**WARNING**

*Be sure to wait the specified time when filling the funnel and before replacing the detector cap. This enables the bubbling nitrogen to settle down and the pressure to dissipate. In addition to wearing safety goggles at all times, stand back from the microscope after each time you fill the funnel.*

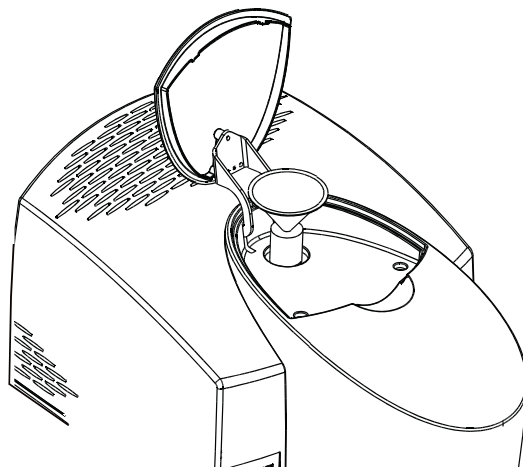


**WARNING**

*Do not site the instrument in a poorly ventilated area.*

*Oxygen depletion in an enclosed space does not trigger a gasping reflex, and errors of judgment, confusion, or unconsciousness can occur in seconds and without warning.*

1. Open the flap covering the dewar.
2. Remove the dewar cap.
3. Place the small funnel supplied with the microscope in the opening in the detector dewar (Figure 11).



**Figure 11** The dewar opening with the funnel inserted

**CAUTION**

*Stand where you can see the inside of the funnel as you pour the nitrogen in, but without positioning your head over the funnel itself. Pour slowly, so that neither the funnel nor the dewar overflows. If liquid nitrogen runs down the outside of the dewar, it can damage the optics of the microscope.*

4. Carefully fill the funnel with liquid nitrogen. Stand back and let the funnel empty completely.  
The liquid nitrogen bubbles rapidly as it drains into the dewar. This first amount of liquid nitrogen vaporizes completely as it cools the dewar.
5. Add another one and a half funnels of liquid nitrogen. Stand back and wait two minutes.  
This nitrogen also vaporizes as the dewar continues to cool. The two-minute wait enables the bubbling to settle down and the pressure of the vaporizing nitrogen to dissipate.
6. Continue to pour liquid nitrogen into the funnel, adding a little more each time the funnel empties.  
The funnel takes longer to empty as the dewar fills. This happens after two to three more funnels of liquid nitrogen.  
Now the dewar has cooled, the liquid nitrogen does not vaporize, but instead fills the dewar.

7. Remove the funnel and wait two minutes.

The liquid nitrogen settles down and bubbling slows.

8. When the nitrogen stops bubbling, refit the detector cap.

The filled dewar cools the MCT to the correct operating temperature for several hours. After that, the dewar begins to return to room temperature, and the preamp switches off power to the MCT.

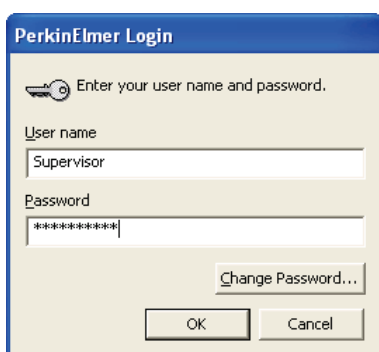
|  |
|--|
| <p><b>NOTE:</b> If the level of coolant is low, a message prompting you to refill the dewar will be displayed in the Status bar at the bottom of the Spectrum workspace.</p> |
|--|

## Setting up the Spotlight 150

---

To set up your Spotlight 150:

1. Make sure the spectrometer is switched on.  
It can take the spectrometer up to two hours to equilibrate after being switched off overnight.
2. Switch on the microscope at the rear switch.  
The blue LED light at the front comes on.
3. At your PC, from the Start menu select **Programs**; the **PerkinElmer Applications** group; the **Spectrum** sub-group and then the **Spectrum** application.  
The Spectrum start-up splash-screen is displayed, followed by a dialog that may require your login details:



4. If required, enter your **User name** and **Password**, and then click **OK**.  
An instrument selection dialog is displayed.
5. Select the **Instrument** you want to use.

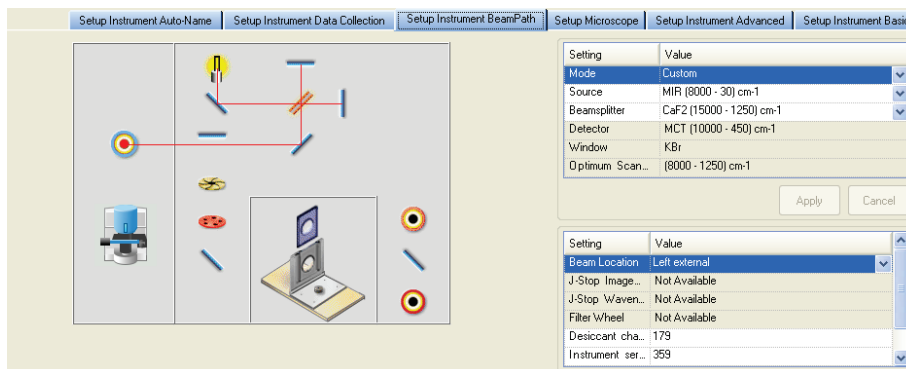
OR

If you want to work with data that has been collected previously, without connecting to an instrument, select to work offline.

**NOTE:** Working offline can free a networked instrument for use by another user.

6. Click **OK**.  
Spectrum starts.
7. If you are connected to an FT-IR instrument, select **Instrument** from the Setup menu.  
The Setup Instrument tabs are displayed.
8. Select the Setup Instrument BeamPath tab.

9. Select the **Beam Location** that directs the beam path to the Spotlight 150.



**Figure 12** Beam path directed to the Spotlight 150

10. Select the Setup Microscope tab.  
You are prompted to start the microscope initialization and aperture calibration procedure.
11. Make sure that the lower cassegrain is fitted, that if the ATR objective is fitted it is retracted, that the stage is centered and that there is no sample on the stage, and then click **OK**.  
The microscope initializes and runs the aperture calibration procedure.

**NOTE:** If you do not want to initialize your instrument at this time, you can click **Cancel**. You will not be able to use any of the microscope controls on the Setup Microscope tab until you have initialized the microscope. To initialize the microscope later, click **Initialize** on the Setup Microscope tab.

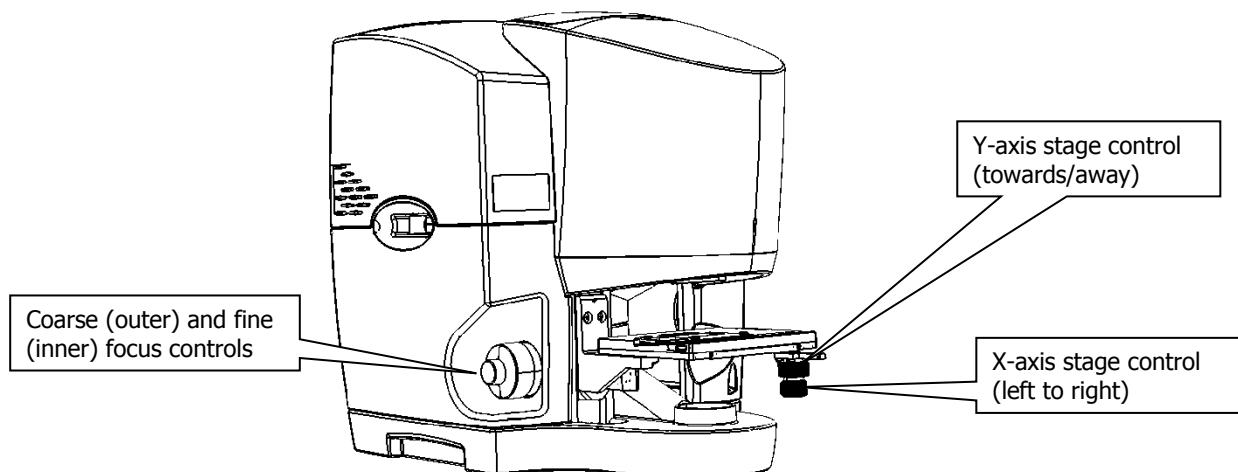
## ***Focusing the Microscope***

### ***Microscope sample positioning and focus controls***

The focus of the microscope is changed by moving the sample stage up or down. While focusing, you can view the image on the Setup Microscope tab.

Turn the coarse or fine focus controls (Figure 13) clockwise to move the stage up and counter-clockwise to move the stage down.

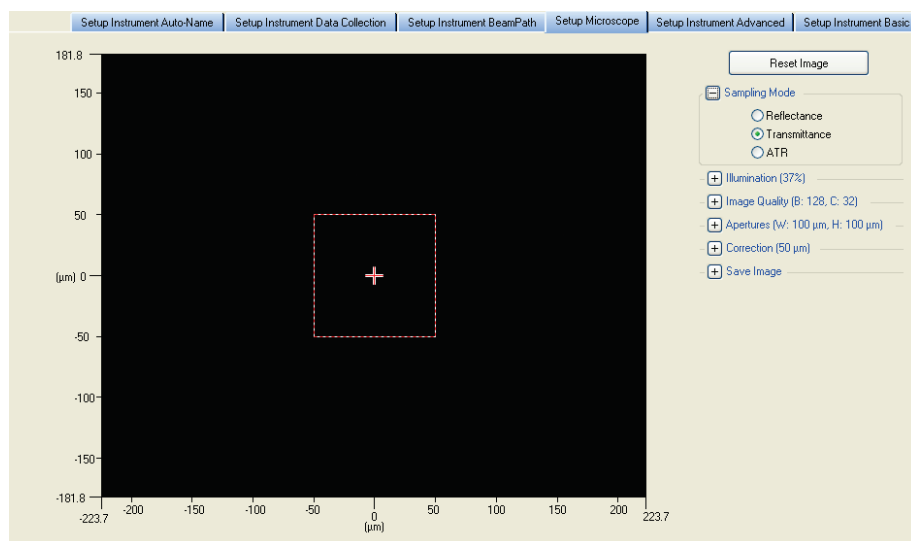
The default position for the lower cassegrain gives optimal illumination for thin samples. For optimal performance it may be necessary to refocus the lower cassegrain to compensate for the sample's refractive index.



**Figure 13 Focus and stage position controls on the Spotlight 150 Microscope**

### ***Focusing the image***

1. Place your sample on the sample stage.
2. Select the Setup Microscope tab.





**Figure 14 Setup Microscope tab showing the Sampling Mode group expanded**

3. Position your sample using the stage controls on the microscope so that you can see the live image of the object of interest.
4. Focus on the sample using the microscope focus controls.  
You may also need to change the illumination of the sample, or the correction, when focussing the image.



**NOTE:** If your Spotlight 150 is supplied with a Frontier or Spectrum 400 FT-IR/FT-NIR dual-range spectrometer and this has been setup to work in the near infrared, you may find that there is a red tint to the video image. To remove this, fit a 1%T attenuator to the external beam window of the spectrometer. See *Fitting an Attenuator to the Spectrometer* on page 41 for details.

This attenuator should be removed during data acquisition.

5. If you want to examine the sample in reflectance mode, select **Reflectance** in the Sampling Mode section.  
Transmittance is selected by default.
6. If you are examining the sample in transmittance mode, then you may need to adjust the position of the lower cassegrain so that the field of view is evenly illuminated:  
Adjust the **Correction** by clicking on the up (  ) or down (  ) buttons.  
This is especially useful if the sample is in a compression cell or held between windows of high refractive index.
7. Adjust the **Illumination** to give good illumination of your sample.

### ***Fitting an Attenuator to the Spectrometer***

If your Spotlight 150 is supplied with a Frontier or Spectrum 400 FT-IR/FT-NIR dual-range spectrometer or if your Spotlight 150 has an InGaAs detector, you will be provided with an Attenuator kit (part number L1160560) containing 1%T, 4%T, 6%T, 14%T and 32%T attenuators. The attenuators attach to the external beam window of the spectrometer magnetically.

When examining samples in the visible range on systems fitted with an MCT detector, use of the 1%T attenuator is recommended if the image appears with a red tint. However, the attenuator should be removed during data acquisition.

The use of an attenuator is recommended if a detector overload error message is displayed in the Status Bar when monitoring or collecting data. This attenuator should be kept in place during data acquisition. For example, a detector overload error message may be displayed when using your Spotlight 150 with a dual-range spectrometer in the near infrared with a large aperture size. To resolve this, fit a 32%T attenuator to the external beam window of the spectrometer.



**Figure 15 KBr window with 14%T attenuator**

## Setting Scan Parameters

The Scan toolbars at the top of the workspace include the tools you need to collect a spectrum from a sample.



**Figure 16 Scan toolbars**

By default, sensible values for the scan and instrument parameters are entered in the Instrument Settings toolbar. The values applied depend on your instrument and accessory. To amend any value, select the parameter and then enter your new value, or select a value from the drop-down list.

8. Set the **Start** and **End** points of the scan range (by default in wavenumbers, but the abscissa units can also be set to nanometers or microns on the Setup Instrument Basic tab).

For mid-IR analysis it is normal to set the upper wavenumber limit to  $4000\text{ cm}^{-1}$  and the lower limit to the low wavenumber specification of the detector, as shown in Table 1.

**Table 1 Low wavenumber specifications of Spotlight 150 detectors**

| Detector type | Low wavenumber limit ( $\text{cm}^{-1}$ ) |
|---------------|---|
| MCT mid-band  | 580                                       |
| MCT wide-band | 450                                       |
| DTGS          | 400                                       |

Your PerkinElmer Service Representative can advise you of the lower limit to the low wavenumber, at the time of installation, or it will be recorded on the Service Installation test spectra.

For near-IR measurements, on a system fitted with an InGaAs detector, the upper wavenumber value is typically set to between  $15800$  and  $8000\text{ cm}^{-1}$  and the lower limit to  $4000\text{ cm}^{-1}$ .

**NOTE:** To work in the near-IR range of the spectrum the Spotlight 150 must be attached to a dedicated near-IR spectrometer or a dual-range spectrometer which has been set up to operate in the near-IR range.

9. Set the **Accumulations** required, either as a number of scans, or as a length of time. There are no firm rules about these selections since the number of scans needed to generate acceptable spectra will depend on the nature of the sample and the requirements of the application. It is probably better to "over-scan"; 50 scans taken at  $8\text{ cm}^{-1}$  resolution using an MCT or InGaAs detector (20 scans for a DTGS detector) is relatively quick.
10. Enter a unique **Sample ID** and **Description** for the sample.
11. If a background scan is required, the **Scan** button includes a small background flag. Clear the instrument beam path, or insert a suitable background material, and then click




to collect a background spectrum.

The background spectrum is displayed briefly, and then the Viewing Area is prepared for data collection from your sample.

A new background scan will be required if you change the aperture dimensions or the sampling mode. However, you can modify the correction and illumination without requiring a new background. We recommended that you run a background scan before every sample.

If you are sampling in transmittance, you may simply chose to move the stage to an area away from the sample and focus on the surface of the substrate using the microscope focus controls. If you are sampling in reflectance, you may want to use an appropriate reference, such as the gold mirror supplied with your Spotlight 150.

See the Spectrum on-screen help for more information.

12. Click  to begin scanning your sample.
- By default, during scanning the sample data is displayed on the Live tab in the Viewing Area.
- The completed spectrum is displayed on the Graph tab, and added to the current Samples View in the Data Explorer.

**NOTE:** If a detector overload error is reported in the Status Bar when monitoring or collecting data, then fit an attenuator to the external beam window of the spectrometer. See *Fitting an Attenuator to the Spectrometer*, on page 41, for details.

This attenuator should be kept in place during data acquisition.

13. If, for any reason, you want to stop scanning your sample, click .


### ***Optimizing Transmission measurements***

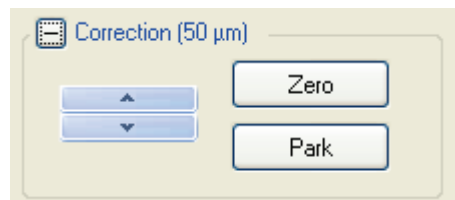
In most cases samples for transmission measurements are placed flat on top of an IR-transmitting window such as NaCl, KBr, BaF<sub>2</sub>, ZnSe or diamond. In some cases, such as flattened fibers, the sample is simply secured across an open aperture mounted in the standard sample holder.

These samples should be prepared such that they are thin, ideally 10 to 25 microns, but certainly thinner than 50 microns (see section on sample preparation) and also they should sit as flat as possible on the window.

In transmission measurements, the IR beam passes through the window supporting the sample and this will alter the beam characteristics (focus, etc.). This variation will be different for different window materials depending on their refractive indices. To optimize the IR energy throughput through the window (and sample) the lower cassegrain is moved up or down using the **Correction** facility on the Setup Microscope tab to compensate for this refractive index variation.

1. Place the thin, flat, sample onto a window (or across an aperture) and focus on the sample using the microscope controls.
2. Set the apertures to the appropriate size for sample area.
3. Move the sample away from the centre of the viewing screen, and focus on the top surface of the window, ensuring that the window is clean.

4. Select **Monitor** function from the Measurement menu or click  to monitor the energy throughput.
5. Adjust the lower cassegrain for maximum energy using the up and down arrows of the **Correction** function.  
Clicking **Zero** will move the cassegrain to the optimum position for an open beam (suitable for very thin samples).



**Figure 17 Correction control**

6. Once the maximum energy through the window (or aperture) has been achieved, press **Halt** on the Measurement toolbar.
7. Collect a background spectrum through this point on the window (or aperture) using the correct aperture size as selected previously.
8. Return the stage to the selected sample position, adjust the correction, and then record the sample spectrum.

**NOTE:** If a detector overload error is reported in the Status Bar when monitoring or collecting data, then fit an attenuator to the external beam window of the spectrometer. See *Fitting an Attenuator to the Spectrometer*, on page 41, for details.

This attenuator should be kept in place during data acquisition.

If the same window is used repeatedly for analysis, height correction of the lower cassegrain need only be done once.

## **Preparing Samples**

## ***Preparing Samples***

---

The Spotlight 150 enables you to examine live image of the sample on the Setup Microscope tab and to choose the area where you want to collect images or spectra. To make sure that you collect good quality spectra, it is important that you prepare samples properly. Sample preparation is needed when the sample is too thick for transmission work, or the area of interest is on the inside (for example in a laminated sample).

- If you are going to collect transmission spectra, the sample should ideally be thin enough (approximately 5 to 20  $\mu\text{m}$ ) to give good detail and undistorted absorption bands.
- The area of the sample must be large enough to give an adequate signal; otherwise, the scan time must be increased.

Preparing a sample, therefore, often involves flattening it; this both thins it and increases its area. The sample can be flattened by rolling, squeezing, or pressing.

This chapter tells you how to prepare samples for spectroscopy with the Spotlight 150. It includes:

- A list of useful tools;
- A list of window materials commonly used for mounting samples;
- Descriptions of special techniques used to prepare particular types of samples.

## ***Tools for Sample Preparation***

---

This section lists the tools you need for preparing samples:

- Tools provided with the Spotlight 150;
- Tools in the microsampling toolkit;
- Materials to have available;
- Specialized accessories you may want to purchase.

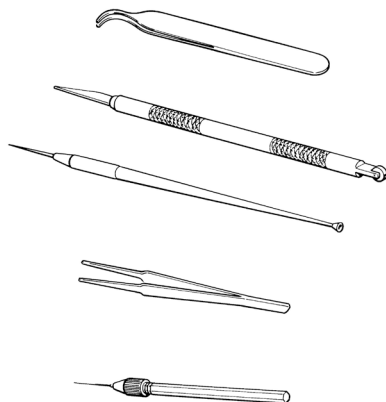
### ***Tools Provided with the Spotlight 150***

The following items for use in sampling are provided in the Sampling Accessories Kit that is supplied:

| <b>Item</b>                | <b>Use</b>   |
|----------------------------|--|
| Holder for 13 mm disks     | Supporting 13 mm disks on the sample stage   |
| Slides, glass (box)        | Supporting samples for sample preparation  |
| Rotating 13 mm disk holder | Supporting samples and allows rotation of the sample   |
| Support for large samples  | Clips on to the sample stage; supporting bulky samples so that the stage clip does not interfere with them |
| Gold mirror assembly       | Reflection measurements  |
| KBr windows (2)            | Supporting samples   |

The following tools are provided in the Microsampling toolkit.

| <b>Tool</b>                            | <b>Use</b>   |
|--|--|
| Steel tweezers                         | Picking up extremely small objects                     |
| Roller knife                           | Cutting (knife end) and flattening (roller end)        |
| Steel probe                            | Pulling samples apart, separating fibers               |
| Forceps, 4½ inch, Cd plated            | Picking up small objects                               |
| Tungsten alloy needle                  | Transferring particles                                 |
| Pin vise                               | Holds needles for sharpening or for flattening samples |
| Interchangeable handle for micro tools | Handle for tungsten needle or steel probe              |



**Figure 18 Some of the Tools in the Microsampling Toolkit**

### ***Other Useful Tools***

Depending on the type of samples that you usually work with, it may be helpful to have some of the following tools:

| <b>Tool</b>   | <b>Part Number</b> |
|---|--------------------|
| Microprobe with right angle bend  | N9302606           |
| Forceps, round tips   | N9302607           |
| Forceps, narrow needle points   | N9302608           |
| Windows: All 13 mm diameter   |                    |
| BaF <sub>2</sub> (1 mm thick)   | N9302611           |
| BaF <sub>2</sub> (2 mm thick)   | N9302612           |
| ZnSe (1 mm thick)   | N9302613           |
| NaCl (2 mm thick)   | N9302614           |
| KBr (2 mm thick)  | N9302615           |
| Wide-tipped forceps, hooked   | 09908138           |
| Wide-tipped forceps, flat   | 09908400           |
| 1.5 mm microdisk; fits in 13 mm disk holder to support very small samples | 01861043           |



## ***Specialized Accessories***

The following accessories are extremely useful in preparing certain types of samples (as described in *Techniques for Preparing Samples*, starting on page 51):

- Miniature Diamond Anvil Cell (part number N9302618);
- Fiber Optic Illuminator (part number N9302602);
- Microtome.

## ***Items to Have Available***

In addition to the items provided with the Spotlight 150, we recommend that you have the following available:

- Tape with adhesive on both sides ("double-sided tape") for holding long or large samples on the sample stage;
- Single-edge razor blades for cutting samples.

## ***Common Window Materials***

---

Both liquid and solid samples are often mounted on salt windows. Very thin windows, 1 to 2 mm thick, give the best spectra. The following materials are commonly used in windows:

- KBr: Potassium bromide is inexpensive, and it transmits infrared radiation to below  $400\text{ cm}^{-1}$ . The major disadvantage of this material is that it is hygroscopic, so that the windows fog easily.
- BaF<sub>2</sub>: Barium fluoride is not hygroscopic. Its transmittance cut-off is  $750\text{ cm}^{-1}$ . It can break or crack easily.
- NaCl: Sodium chloride transmits infrared down to  $600\text{ cm}^{-1}$ . Otherwise, its properties are similar to KBr.
- ZnSe: Zinc selenide is not hygroscopic. Its transmittance cut-off is  $650\text{ cm}^{-1}$ . ZnSe is more durable than the other windows but is yellow, so that the live image on the Setup Instrument Microscope tab appears yellow.

## ***Techniques for Preparing Samples for Transmission Measurements***

---

This section describes some useful techniques for preparing various types of samples.

### ***Flattening Solids***

Flattening samples by pressing or squeezing often enables you to make thick samples thin enough to give good infrared spectra. As the samples are usually quite small, only moderate force is necessary.

#### ***Rolling with the roller knife***

The roller end of the roller knife provided in the microsampling tool is one of the simplest and most effective devices for flattening samples. It is especially useful for flattening fibers or particles.

You can treat different types of sample in different ways:

- If the sample is soft, you can roll it on a small salt window.
- If the sample is hard, you can roll it on a hard surface, such as glass or metal. A flat, black cap from a jar makes a good surface for rolling a light-colored sample.
- If you roll the sample on a small, flat piece of metal you can view it and collect spectra in reflectance mode. Samples rolled on windows transparent to infrared can be examined in transmittance.
- If you flatten fibers on a glass microscope slide, they can then be peeled off and mounted either on a window or over the aperture for the microscope slide.

#### ***Squeezing with a pellet press***

You can squeeze samples between the polished anvils of a KBr pellet press without KBr. To collect the spectra use one of the following methods:

- Peel the flattened sample off the anvil with a probe or knife and place it on a sample mount. Collect the transmittance spectrum.
- Leave the sample on the polished anvil and collect the reflection spectrum. Use a clean area of the anvil as the reference.

#### ***Using a diamond anvil cell***

See *Diamond anvil cell* on page 53 for information on flattening samples with a miniature diamond anvil cell.

### ***Compressing between infrared transmitting windows***

Pressing two windows together, with the sample between them, compresses the sample. This also provides optical contact between the windows and the sample, reducing surface scattering.

Windows made of NaCl or KBr are relatively soft. If your sample is hard, or if it is wet, use BaF<sub>2</sub> or ZnSe.

### ***Pressing with the heel of a probe***

Press on small samples with the flat end of the probe handle. Even moderate pressure usually produces considerable thinning.

### ***Pressing with a needle***

Pressing with the point of a needle or probe applies a high force per unit area, because the area of contact is small.

Rolling a hard sample with the side of a sewing needle held in a pin vise presses it into a flake.

## ***Slicing Samples from Solids***

### ***Cutting a wedge of sample***

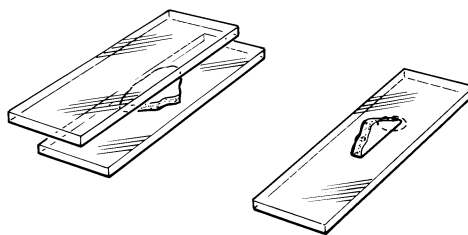
Cutting a wedge-shaped piece from its edge enables you to produce a thin sample while destroying very little of the original. This technique can be used with laminates, plastics, films, paint chips and paper.

To cut a wedge-shaped piece from a relatively thick sample:

- Hold the sample in tweezers as you slice a thin wedge from it with a razor blade. Taper the wedge to as thin a slice as possible.

To cut a wedge-shaped piece from a relatively thin sample:

1. Place the sample between two offset glass slides. Allow a triangular portion of the sample to protrude as shown on the left in Figure 19.



**Figure 19 Cutting a wedge-shaped sample**

2. Run a razor blade or the roller knife along the edge of the upper slide.  
The triangular piece of the sample is sliced off, giving a wedge-shaped sample.
3. To mount the sample, rotate it so that it is positioned as shown on the right in Figure 19.
4. Position it under the microscope so that the infrared beam goes through the thin end of the wedge (circled in Figure 19).

### **Microtoming**

A microtome enables you to slice a sample into thin cross-sections, 0.5 to 20  $\mu\text{m}$  thick. It is commonly used to prepare samples for light microscopy; the same range of thicknesses is also appropriate for infrared microspectroscopy.

If you are trying to identify the individual components of a laminate, microtomed samples give the best results.

Samples are often embedded in a supporting medium before they are microtomed. If you must use an embedding material, choose it carefully so that it does not alter the sample by reacting with it, dissolving it, or contaminating it. Some commonly used materials are:

- Paraffin wax: This is the preferred medium for infrared spectroscopy. It produces few spectral interferences, and it can usually be easily removed from the sample with warm xylene.
- $\beta$ -pinene wax: This material is similar to paraffin.
- Plastic embedding materials: These can be used depending on the size and porosity of the sample.
- Acrylic and epoxy resins: Although these are commonly used in light microscopy, they are not recommended for infrared, because they are hard to remove and can cause spectral interferences.

### **Polymers**

Pressing or squeezing enables you to reduce the pathlength of polymer samples such as paint chips, thick films, elastomers, or fibers.

#### **Diamond anvil cell**

The miniature diamond anvil cell, shown in Figure 20, enables you to press polymers (or other compressible samples). It enables you to both thin the sample and collect its spectrum in the same device; this is an advantage when you have limited material available. It is small enough to be easy to manipulate, and fits in the recessed retainer in the support for large samples. By collecting a background spectrum of an empty area of the cell, you can completely compensate for the absorption bands of the diamonds.

To thin a sample in the miniature diamond anvil cell:

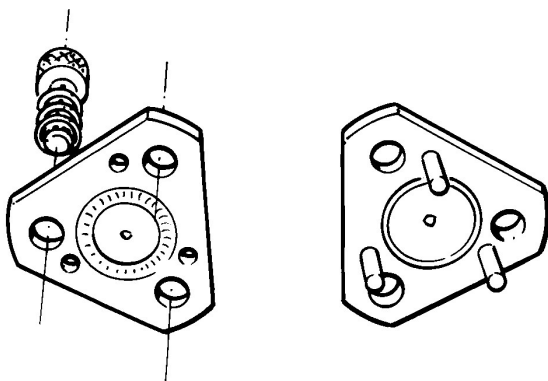
1. Loosen and remove the three screws that hold the cell together.
2. Lift off the top half of the cell and set it aside.
3. Place the sample on the bottom half of the cell. (The sample must be small.)
4. Put the top half back on the cell, lining up the red lines on the top and bottom halves. Do not tighten the screws yet; applying uneven shear forces may damage the diamonds.

**CAUTION**

*One diamond can damage another.*

5. Press down on the cell with your thumb to thin the sample.
6. Replace the three screws and tighten them finger tight.

**NOTE:** If the spectrum collected with the diamond anvil cell shows interference fringes, place some KBr in the cell and collect a background spectrum through it.



**Figure 20** The miniature diamond anvil cell

***Pressing elastomers between windows***

If your sample is elastic and you are compressing it between windows, you must apply pressure continuously. Use the following procedure:

1. Press on the windows with a probe, flattening the sample.
2. While maintaining the pressure, apply small amounts of quick-setting nitrocellulose cement to the edges of the salt plates.  
When the cement is dry, the sample remains compressed.

The compression cell (see *Using the Compression Cell* on page 60) enables you to compress this kind of sample.

## **Filled polymers**

When a polymer contains a high concentration of fillers, and you want to analyze the polymer, you have to prepare a sample for analysis that is free of filler.

Often you can obtain a suitable sample by cutting a thin wedge of the material with a sharp blade. If the filler is not uniformly dispersed, you can find clear regions of polymer for analysis.

You can use pyrolysis to remove the fillers. As you heat the sample, the polymer volatilizes, and the fillers are reduced to ash. The sample can be pyrolyzed in the following ways:

Using a disposable pipette:

1. Place the polymer in a disposable pipette and seal the large end.
2. When this end cools, tap the polymer into it, then heat the sample gently.  
The pyrolyzed polymer condenses on the walls of the pipette. The filler is left behind as ash.
3. Score and break the pipette between the ash and the pyrolysate.
4. Add a drop of solvent to the pyrolysate to wash it on to a salt plate.

If the amount of sample is small, use a capillary tube instead of a disposable pipette.

Using a microbrush to pyrolyze micro amounts:

1. Seal the end that is away from the brush fibers and tap the sample particle into this end then heat it gently with a microtorch.
2. After pyrolysis, break off and discard the end of the tube that contains the ash.
3. While holding the fibers of the brush against the salt plate, add a drop of solvent to the broken end.
4. Allow the solvent containing the pyrolysate to flow into the fibers.  
When it evaporates, the pyrolysate remains on the salt plate for analysis.

## **Particles**

### **Crushing**

Crushing enables you to thin samples such as large particles that cannot be sliced. This can be done in various ways:

- If the sample is small, crush it with the roller end of the roller knife.
- If the sample is larger, use a pestle and mortar.

### ***Separating by aperturing***

Powders and other particulate solids may contain several different components. Instead of separating them, use the infrared aperture to isolate the component you want to sample:

1. Spread the sample out with a probe so that you can visually distinguish the components.
2. Looking at the Monitor Visible window, find a particle of the component you want to sample.
3. Center this particle in the field of view.
4. Adjust the infrared aperture until only the particle that is of interest is visible.

You can easily pick up extremely small particles and transfer them with a very fine-pointed tungsten needle. Scoring the surface of the salt plate with the needle makes a simple map to help you positively identify the particles under the microscope.

### ***Transferring with a tungsten needle***

When necessary, sharpen the tungsten needle.

### ***Nujol or fluorolube mulls***

Suspending fine particles of a solid sample in nujol or fluorolube reduces or eliminates the surface reflections that can distort absorption measurements. These oils also reduce the amount of radiation lost to reflection or scattering.

If the film is thin enough, you can correct the spectrum for the presence of the oil by subtracting a spectrum of the pure liquid. It is difficult, however, to obtain the correct thickness for a good subtraction.

### ***Fibers***

You can roll fibers to flatten them (see *Flattening Solids* on page 51), or they can be pressed in a diamond anvil cell (see *Polymers* on page 53).

### ***Fibrous Solids***

If a fibrous sample, such as paper, is too thick, tear it and examine the torn edges. The edges contain single fibers and thin clumps of fibers.



## Coatings on Substrates

If the sample is coated on a substrate, the method for collecting its spectrum depends on the nature of the substrate:

- If the substrate is reflective, you can analyze the sample in reflectance.
- If the substrate is opaque, scrape off a sample of the coating; use the roller knife to scrape a small piece on to a KBr or BaF<sub>2</sub> disk.
- Coatings on reflective or opaque substrates can be measured using the micro-ATR objective. See *Collecting Spectra With the ATR Objective* on page 70 for details.

## Liquids

### Solutions of samples

Although liquids are seldom analyzed with the Spotlight 150, sometimes the sample of interest is in solution.

1. Transfer the solution on to a salt plate.
2. Allow the solvent to evaporate, leaving the sample on the plate.

### Micropipettes

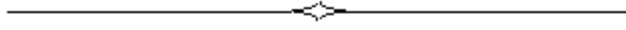
You can use a micropipette to apply liquid to the surface of a salt plate, or to the edge of the junction between two salt plates. In the latter case, the liquid flows between the plates by capillary action.

### Preventing liquids from spreading

If the amount of liquid being transferred to the salt plate is very small, restrict it to a small area of the plate. There are several ways to do this:

- Use a microbrush to transfer solutions. The bristles of the microbrush hold the liquid in a small region of the salt plate until the solvent evaporates.  
Repeatedly jab a small area of the salt plate with a tungsten probe. Leave the resulting small salt particles in the well that is produced.  
The capillary spaces between the salt particles retain the liquid and minimize spreading.
- Place the salt plate on a small metal washer that is being gently heated.

Because there is more heat at the outside of the salt plate than near the center (over the hole in the washer), the droplet of liquid is forced toward the center.



# **Techniques for Collecting** **Spectra**

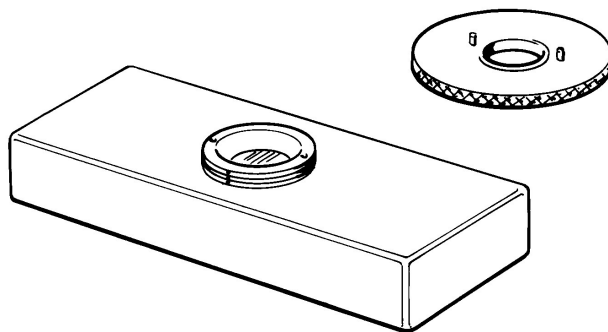
## ***Techniques for Collecting Spectra***

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This chapter describes how accessories and collection techniques enable you to collect spectra from different types of sample.

### ***Using the Compression Cell***

The optional compression cell (part number N1870185, Figure 21) enables you to flatten soft materials. It also enables you to hold specimens flat and in optical contact with salt windows. The cell consists of an aluminum block, machined to accept salt windows, with window retainers and a special wrench to apply pressure across the windows. The sample is held between the two windows. The compression cell fits into the sample slide holder on the stage of the microscope. Windows with thicknesses equal to 1 mm and 2 mm, and outer diameter equal to 13 mm can be used with the cell. Two KBr windows (2 mm thickness) are included with the system. The cell can apply pressure without rotating the windows, and therefore avoids scratching them.



**Figure 21 The compression cell**

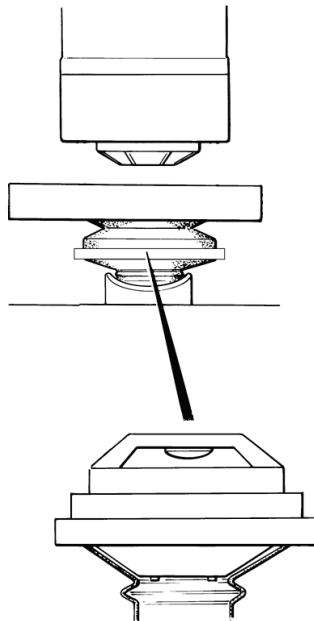
Although samples can be thinned using the compression cell, it does not replace the miniature diamond anvil cell as a sample-thinning device. The primary application of the compression cell is for keeping specimens flat over the entire visual field of view.

## Collecting a Spectrum from a Thick Sample

If your sample is very thick, you will have to lower the stage to focus the sample. For very thick samples you may have to remove the lower cassegrain assembly; then only the reflectance method can be used to view the sample and collect spectra.

### To remove the lower cassegrain and lower the stage

1. Remove any sample from the stage.
2. Click **Zero** in the Correction section of the Setup Microscope tab.  
The lower cassegrain moves to the position where the infrared beam is focused if no sample is on the stage.
3. Move the sample stage to its highest possible position.
4. Release the locking lever located on the right side on the back of the lower cassegrain assembly (Figure 22).



**Figure 22 The lower cassegrain**

5. Gently slide the cassegrain assembly forward and out of the dovetail connector.

**NOTE:** To make withdrawal easier, pull the locking lever gently.

6. Click **Park** in the Correction section of the Setup Microscope tab.  
The lower cassegrain bracket moves down. Park is the lowest position to which the lower cassegrain bracket can move when the lower cassegrain is fitted.  
The button text now updates to **Lower Park**.
7. Click **Lower Park** again.  
The lower cassegrain bracket moves to its lowest position.
8. Lower the stage.

### **To refit the lower cassegrain after use**

1. Raise the stage to its highest position and ensure that the stage is centered.  
Before moving the stage up from the Park or Lower park positions you must ensure that the stage is centered. The calibrated rules marked on the stage can help with this. The X-axis should be positioned at about 115 mm and the Y-axis should be positioned at about 10 mm.
2. Choose **Zero** in the Correction section of the Setup Microscope tab.  
The lower cassegrain moves to the position where the infrared beam is focused if no sample is on the stage.
3. Make sure that the cassegrain is correctly seated.
4. Place the gaiter over the cassegrain if the purge facility is required.
5. Slide the cassegrain assembly back into the dovetail connector, as far as it goes.

**NOTE:** To make replacement easier, pull the locking lever gently.

6. Tighten the locking lever.

### **Collecting a Spectrum in an Inert Atmosphere**

The purge system enables you to purge the spaces around the sample and the infrared beam (typically with nitrogen or dry air), to provide an inert atmosphere.

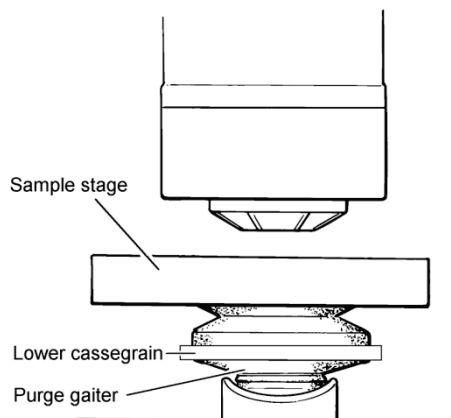


**WARNING**

*Do not use flammable gases for purging.*

### **Parts of the purging system**

Some parts of the purging system are shown in Figure 23.



**Figure 23 Parts of the purge system**

The purge system consists of:

- The gas inlet connector on the metal plate at the rear of the microscope and the connector at the rear of the spectrometer;
- The purge coupling molding connecting the spectrometer to the microscope;
- The lower purge gaiter under the lower cassegrain.

When all of these parts are in place, the gas entering through the inlet displaces air from the path of the infrared beam.

### ***Purging the system***

1. Make sure that all parts of the purge system, as listed above, are in place.
2. Set up the microscope.  
See *Getting Ready to Use the Spotlight 150* starting on page 33.
3. Place the sample in position.
4. Purge the microscope and spectrometer for 15 to 20 minutes at a rate of  $10 \text{ l min}^{-1}$ .
5. Collect the background spectrum and the spectrum of the sample.

### ***Changing the sample***

If you have changed a sample, any air that has entered the system must be flushed out.

## Viewing a Sample with the Visible Polarizer Accessory

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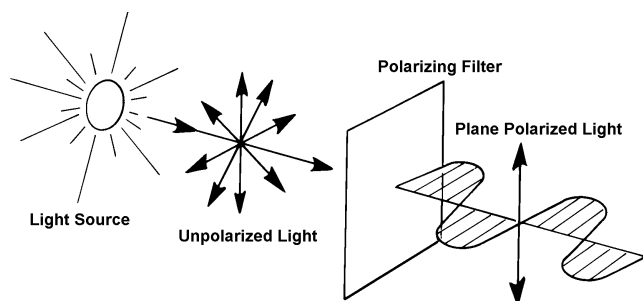
Polarized visible light can enable you to identify areas or structures that differ chemically and to solve problems commonly found in infrared microspectroscopy applications.

### The Theory of Light Polarization

Ordinary light and infrared radiation consists of waves vibrating in all possible planes perpendicular to the direction of propagation. This is represented in the left side of Figure 24.

Conventionally the plane of the light is taken to be the plane of the continuously varying electric vector.

If the light passes through a *polarizer*, the polarizer allows the passage of only those waves that have their plane of vibration in one particular direction. The light that emerges is said to be *polarized*, and is represented on the right side of Figure 24.



**Figure 24 Representation of unpolarized light (left) and polarized light (right)**

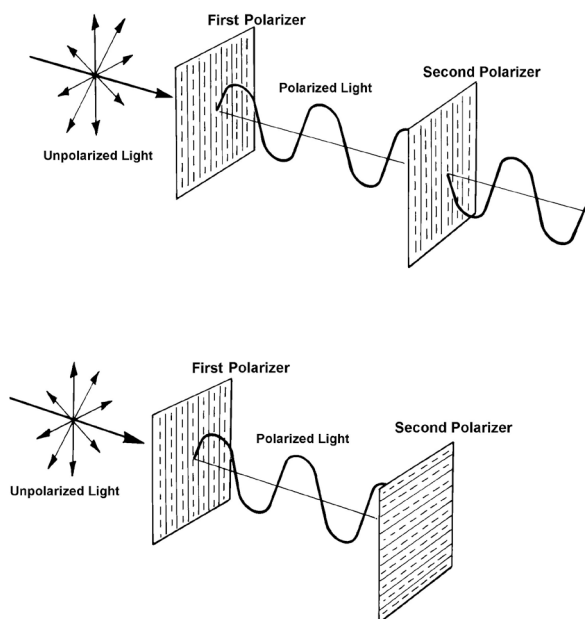
Because all components of the wave in the plane of polarization are transmitted, the ideal polarizer allows 50% of the light through.

If a second polarizer is placed in the path of the polarized light two things may result:

- If the second polarizer is placed in the same direction as the first (as at the top of Figure 25), the polarized light can pass straight through.
- However, if the second is placed at a right angle to the first, a situation which is referred to as crossed polarizers, the passage of the polarized light is blocked, that is, extinction occurs (as at the bottom of Figure 25).



The second occurs because the light transmitted by the first polarizer oscillates in exactly the plane that is blocked by the second polarizer.



**Figure 25 Polarizers parallel (top) and polarizers crossed (bottom)**

Some materials are *anisotropic* (or *birefringent*): their refractive index depends on their orientation. These materials can alter the polarization of light passing through them; this is dependent on the wavelength of the transmitted light.

When you look at an anisotropic sample using polarized light, the change in polarization caused by the sample means that some light leaks through the second polarizer. Because the change in polarization is dependent on the wavelength, the color of the light emerging changes with the distance traveled through the sample and the amount of birefringence encountered.

## Applications

Differences in the birefringence of an object or area may be an indication of chemical disparity. This can be useful in visibly separating or identifying an object or area of interest before collecting an infrared spectrum. Some examples are given below.

### Laminates

Many polymer structures consist of different layers of material and adhesives of varying thicknesses bonded together to meet physical requirements. If you view a cross section of the structure using polarized light, you can identify the individual layers and set the apertures to collect a separate infrared spectrum from each layer. This is useful for identifying the materials used to create the structure.

### ***Inclusions in polymer films***

Although they may appear identical when using non-polarized light, the film and inclusion present in a polymer film may exhibit different degrees of birefringence when using polarized light. If this is so, you can visibly identify the inclusion and collect an infrared spectrum to determine its composition.

### ***Rocks minerals and crystals***

Most crystals are characteristically birefringent and thus are ideally suited to this technique. Viewing a mixture consisting of crystals, such as an artificial sweetener, pharmaceutical powder, or an illicit substance, enables you to visibly separate the components by their relative size, shape and birefringence.

### ***Fibers***

Polarization may enable you to identify and separate fibers in a clump or to view a bicomponent fiber. Most fibers in their natural state are optically thick and their cylindrical shape can cause lensing effects. For these reasons, fibers are usually flattened in preparation for infrared microspectroscopy. This flattening affects the birefringence of the structure and may degrade the usefulness of this technique.

### ***Biological substances***

Birefringence can occur in some biological substances. You can collect infrared spectra of thin sections of these materials. In some cases, polarized light can reveal structures or chemical disparities in these structures, and infrared spectra can be collected of the different regions of interest.

## ***Equipment***

The equipment for visible polarization studies consists of two parts: the polarizer and the analyzer (Figure 26).

The polarizer polarizes the incoming beam from the illuminator and the analyzer contains a polarizing element that you can rotate to any orientation. It is placed in front of the camera.

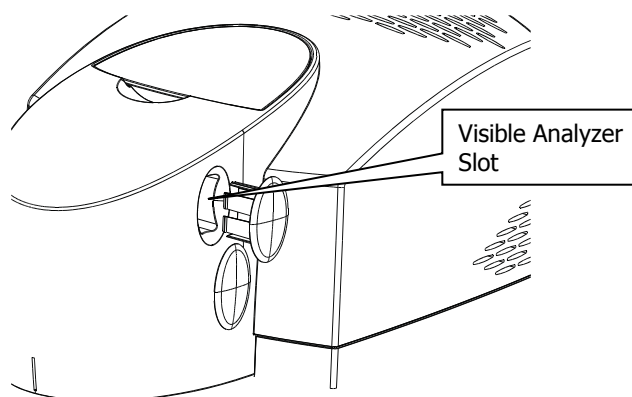
The polarizers for both transmittance and reflectance are built into the Spotlight 150, and are automatically switched into the beam when the analyzer is inserted.



**Figure 26 The analyzer**

### **Operating the Analyzer**

1. Insert the analyzer into the slot in the right side of the front cover (Figure 27). Push the analyzer in with the wheel facing towards you. It has two positions:
  - The first position allows the full beam to pass through.
  - When the analyzer is inserted fully, the polarizing element is in the beam.



**Figure 27 Analyzer position**

2. When the polarizing element is in the beam, rotate the wheel while viewing the sample image on the Setup microscope tab.

## Collecting IR Spectra Using the Infrared Polarizer Accessory

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An absorption band in the infrared range occurs when a vibration is accompanied by a change in dipole moment. The electric vector of the incident radiation must have a component in the direction of the dipole moment change.

In polarization spectroscopy, the absorption bands of greatest interest are those in which the direction of dipole moment change is related to a bond direction, for example, the nitrile stretching vibration. If, in a particular sample, all the bonds of a particular type are aligned in a specific direction, the strength of the absorption depends on the polarization of the incident radiation, that is, whether the electric vector is parallel to or perpendicular to the bond direction.

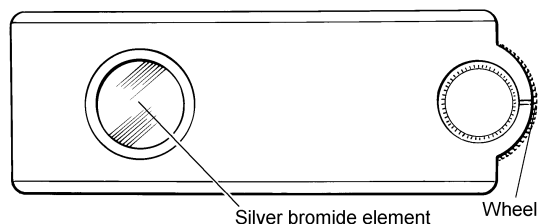
For example, stretching an acrylic fiber aligns the molecules with the general direction of the polymer chains parallel to the fiber axis, and the nitrile groups tend to be oriented perpendicular to the axis. If the spectrum is collected with the infrared radiation polarized perpendicular to the axis, the nitrile absorption peak is much stronger than if the spectrum is collected with radiation polarized parallel to the axis. The ratio of the two intensities (called the *dichroic ratio*) is a measure of the extent of alignment of the nitrile groups and thus of the polymer chains.

The Spotlight 150 enables you to collect polarization spectra of very small samples. These include:

- Single filaments (typically  $14 \times 70 \mu\text{m}$ );
- Films;
- Single crystals;
- Liquid crystals.

### Equipment

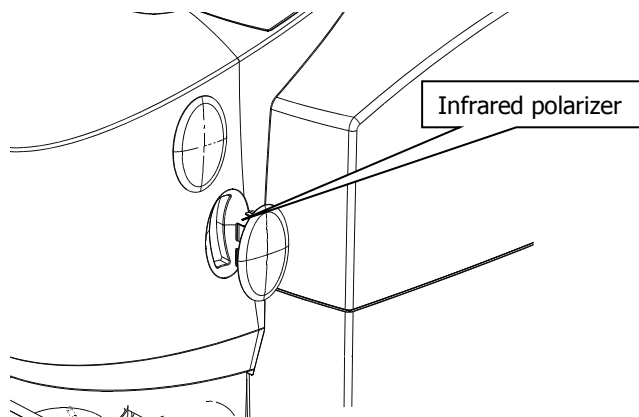
The optional polarizer has a rotatable silver bromide element in an aluminum mount (Figure 28).



**Figure 28 The infrared polarizer**

## Using the Polarizer

1. Remove the snap-in cover that masks the aperture for the infrared polarizer (Figure 29).
2. Slide the analyzer into the vertical slot on the sample holder that can be seen through this aperture.  
The flat side of the analyzer must be towards the rear of the microscope, and the wheel facing outwards.



**Figure 29 Infrared polarizer position**

3. Push the analyzer into the slot until it stops.
4. Turn the wheel to orient the polarizing element.

**CAUTION**

*The polarizer element is extremely fragile. Do not touch it with anything. It cannot be washed, dusted, or blown upon by air. If damaged, it cannot be repaired. When it is not in use, protect it in the case supplied.*

**NOTE:** Both the scribed line and the uneven coloration are usual, and do not affect the performance of the element.

## ***Collecting Spectra With the ATR Objective***

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The ATR (attenuated total reflectance) technique enables the collection of spectra from materials that are too opaque for transmission measurements, and too strongly absorbing for good reflectance measurements. Spectra can be collected with little sample preparation.

The ATR objective uses a crystal made from a material that transmits infrared radiation, and has a high refractive index. An infrared beam enters the crystal and is internally reflected within the crystal, creating an evanescent wave. At each reflection inside the crystal, the wave continues beyond the crystal surface into a sample that is held in close contact. The penetration depth depends on the refractive indices of the crystal and the sample. For a germanium crystal, the penetration depth for most samples is less than 1  $\mu\text{m}$ . The penetration depth also varies with the wavenumber of the infrared radiation:

$$d_p = \frac{\lambda}{2\pi n_1 (\sin^2 \theta - n_{21}^2)^{1/2}}$$

Where:

$n_1$  is the refractive index of the ATR crystal (for germanium 4.0)

$\lambda$  is the wavelength of the radiation

$\theta$  is the angle of incidence of the beam

$n_{21}$  is the ratio of the refractive indices of the sample and the ATR crystal.

### ***ATR objective***

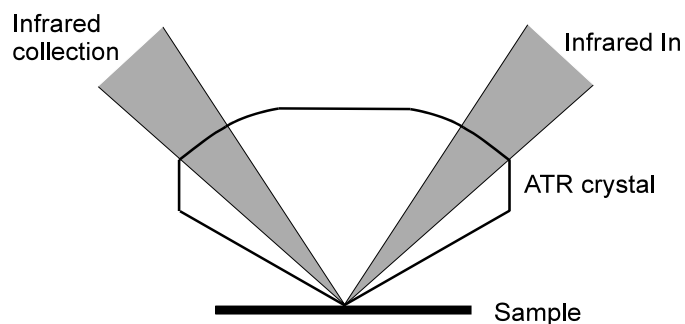
The ATR objective enables the microscopic examination of samples to locate the exact area of interest. The ATR crystal is moved vertically out of the beam path to enable you to view the sample, and place the area of interest in the center of the field of view. The crystal is then placed onto the sample under positive pressure to collect a spectrum.

The crystal has a small contact area formed by the flattened point of a cone; this ensures an approximately 100  $\mu\text{m}$  diameter contact area.

Two versions of the ATR objective are available. The **manual ATR objective** requires you to raise and lower the crystal using a toggle bar on the ATR assembly. The **automated ATR objective** controls the crystal automatically.

**Optical path**

Infrared radiation is directed into the crystal from the front half of the upper cassegrain, and is focused at the sample position. It is reflected once within the crystal, then the totally internally reflected beam is collected by the rear portion of the upper cassegrain, which focuses it on the remote aperture. The radiation is directed onto the detector in the microscope.



**Figure 30 Infrared radiation optical path**

## ***ATR Objective Specification***

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The ATR cassegrain is fitted to the microscope and the ATR crystal holder is supplied separately.

### ***ATR crystal materials***

Germanium (Ge), Silicon (Si), and Diamond Coated Germanium

### ***Range of measurement***

Ge:  $5500\text{ cm}^{-1}$  to  $600\text{ cm}^{-1}$

Ge/Di:  $5500\text{ cm}^{-1}$  to  $600\text{ cm}^{-1}$

Si:  $7800\text{ cm}^{-1}$  to  $800\text{ cm}^{-1}$

### ***Area of contact with sample***

Nominally  $100\text{ }\mu\text{m}$  diameter flat surface. Single internal reflection from surface.

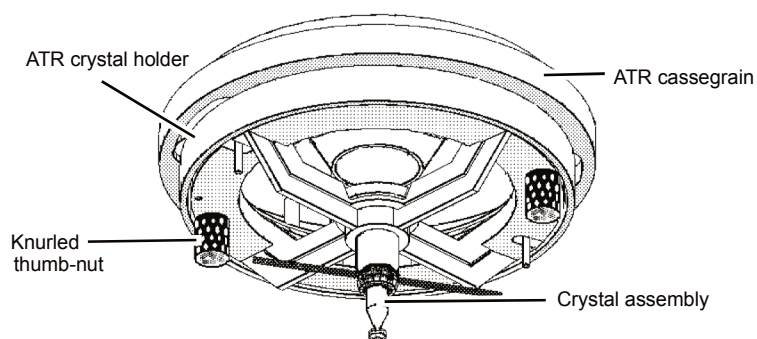
### ***Maintenance***

The ATR assembly may be removed (see *Removing the ATR Crystal Holder* on page 79), cleaned (see *Cleaning the ATR Objective Crystal* on page 82), and replaced and aligned by the user (see *Fitting the ATR Crystal Holder to the Microscope* on page 73).

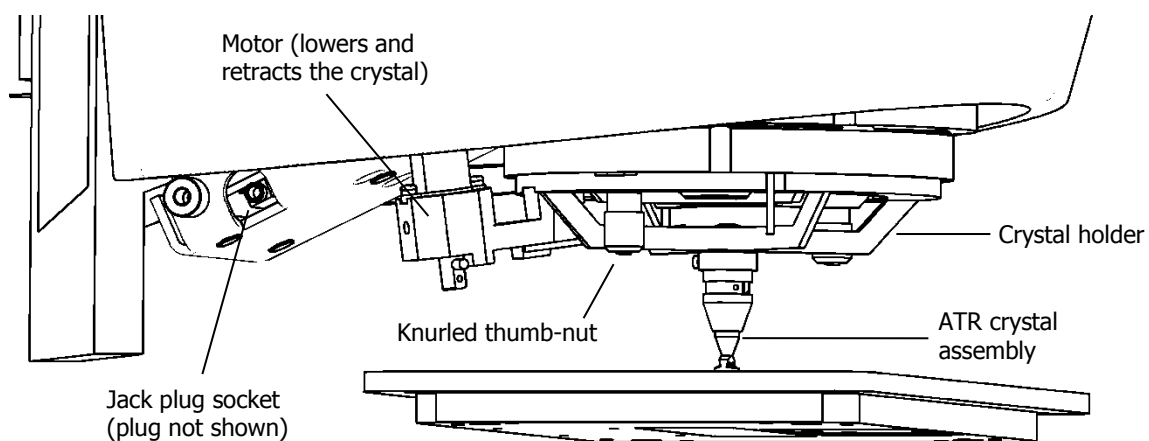


## **Fitting the ATR Crystal Holder to the Microscope**

1. Switch off the microscope at the electricity supply.
2. Stop any laser radiation from entering the microscope by switching the internal beam of the spectrometer to the internal sample compartment.  
For further information on how to do this, see the Spectrum software on-screen Help.
3. Hold the crystal assembly under the upper cassegrain.  
Be careful not to change the alignment of the two adjusting levers.
4. Fit the two adjustment thumb-nuts (see Figure 31).  
The assembly is aligned if the adjusting levers have not been moved.
5. If fitting the automated ATR objective, insert the jack plug into the socket at the rear of the cassegrain (Figure 32).  
Lower the stage if necessary to improve the access to the socket.
6. Direct the infrared beam to your microscope.
7. Switch on the microscope.



**Figure 31 The ATR crystal holder and ATR cassegrain**

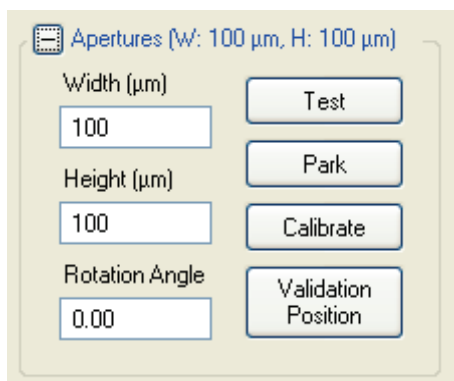


**Figure 32 The Automated ATR Crystal Holder and Cassegrain**



## ***Measuring the Energy in Reflectance Mode***

The following procedure allows you to determine the maximum energy reaching the detector when the ATR crystal is retracted. The value obtained here can be used to check that the crystal is aligned correctly; see *Checking the Infrared Alignment* on page 78 for details.

1. Make sure that your Spectrometer is switched on, and the source has warmed up.
2. Make sure that the microscope and PC are switched on and that Spectrum is running.
3. Ensure that the IR beam is directed through the microscope on the **Setup Instrument BeamPath** tab.
4. Place the slide holding the reference mirror (L1860409, supplied with the microscope) on the sample stage.
5. Make sure that the microscope is in reflectance mode by selecting **Reflectance** in the Sampling Mode section of the Setup Microscope tab.
6. Look at the live image and focus the beam on the surface of the mirror.  
There are often dust particles or scratches on the surface that you can use to focus on.
7. Enter **100** in the **Width** and **Height** text boxes in the Apertures section of the Setup Microscope tab.  
The aperture size is set.



**Figure 33 Aperture controls**

8. Click  on the Measurement bar and select to monitor the energy reaching the detector.  
The Live tab displays the energy reaching the detector.
9. Record the energy measured.
10. Click  .

## Adjusting the Height of the Crystal

### CAUTION

*To prevent serious damage to the crystal, do not move the Sample Stage while the ATR crystal is in the lower, working position and touching or near to the sample.*

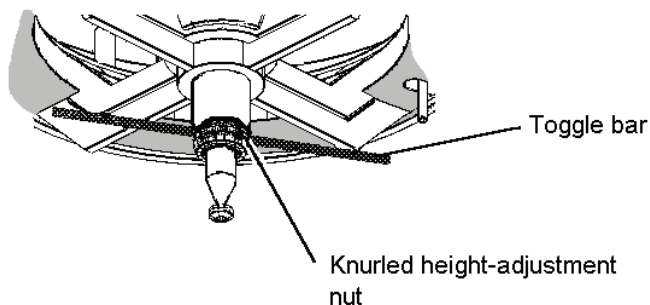
1. Lower the manual ATR crystal onto the reference mirror by turning the bar clockwise and slowly lowering the toggle bar.

You must always support both ends of the toggle bar when lowering or raising the crystal.

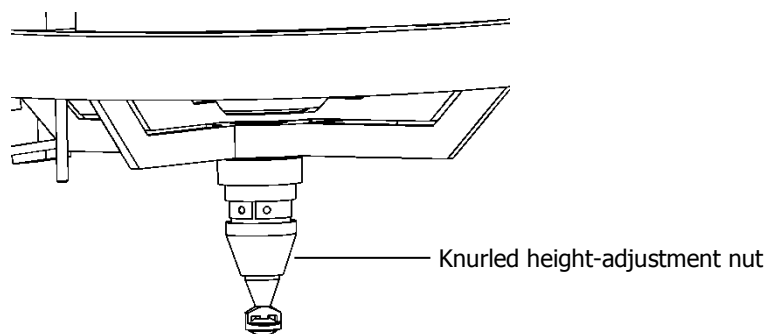
OR

Click the  button to lower the automated ATR crystal onto the reference mirror.

2. Using the knurled height-adjustment nut, adjust the height of the crystal so that it just rests on the mirror under positive pressure (Figure 34).



**Figure 34 Adjusting the Crystal Height (Manual ATR Objective)**



**Figure 35 Adjusting the Crystal Height (Automated ATR Objective)**

## Centering the Crystal

1. Retract the manual ATR crystal using the toggle bar, by lifting the bar then twisting counterclockwise.

OR

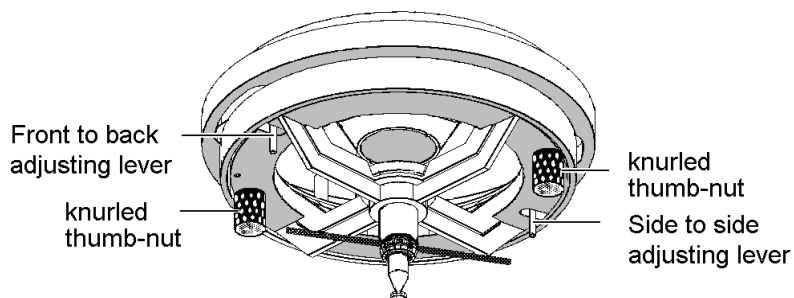
Click the  button to retract the automated ATR crystal.

2. Place a piece of black PVC electrical tape on a microscope slide, and put the slide on the microscope stage.
3. Move the stage so that the tape is directly below the crystal, and focus the beam on the tape.
4. Lower the crystal carefully onto the tape.  
If the crystal height is adjusted correctly, it will just touch the tape and make a shallow impression in it.
5. Retract the crystal.  
If there is no impression on the tape, the crystal height should be lowered slightly by turning the knurled height-adjustment nut clockwise a quarter of a turn.  
If there is a deep impression on the tape, which does not disappear within a few seconds, the crystal height should be raised slightly by turning the knurled height-adjustment nut clockwise a quarter of a turn.
6. If appropriate, repeat steps 4 and 5 until the crystal just touches the tape.
7. Once the crystal height has been corrected, look at the impression of the crystal on the tape. Decide whether the impression is in the center of the field of view.  
If the impression is in the center of the field of view, the assembly is aligned.

### If the impression is not in the center of the field of view:

1. Loosen the two knurled thumb-nuts under the ATR crystal holder half a turn.

**NOTE:** The centering adjustments are the same for both the manual and automated ATR objectives.



**Figure 36 Centering the ATR crystal**


2. Move the ATR crystal holder using the adjusting levers until it is centered.  
One lever moves the assembly backwards and forwards and the other from side to side.

3. Tighten the knurled thumb-nuts.
4. Move the microscope slide to a fresh area of tape.
5. Lower the crystal onto the tape.
6. Retract the crystal.
7. Look through the microscope at the impression of the crystal on the tape. Decide whether the impression is in the center of the field of view.
8. If necessary, repeat steps 1 to 7 until the ATR objective is aligned.

## ***Checking the Infrared Alignment***

The following procedure allows you to determine the energy reaching the detector when the ATR crystal is in place. If the crystal is aligned correctly, the energy level measured should be at least 20% of the maximum energy measured in reflectance mode; see *Measuring the Energy in Reflectance Mode* on page 75 for details.

1. Place the slide holding the reference mirror (supplied with the microscope) on the Sample Stage.
2. Look at the video image on the Setup Microscope tab and focus the microscope on the surface of the mirror.  
There are usually dust particles or scratches on the surface that you can use to focus on.
3. Lower the crystal onto the reference mirror.
4. Enter **100** in the **Width** and **Height** text boxes in the Apertures section of the Setup Microscope tab.

5. Click  on the Measurement bar and monitor the energy reaching the detector.  
The energy level measured should be at least 20 of that measured in reflectance mode; see *Measuring the Energy in Reflectance Mode* on page 75 for details.

6. Click  .

## ***Removing the ATR Crystal Holder***

---

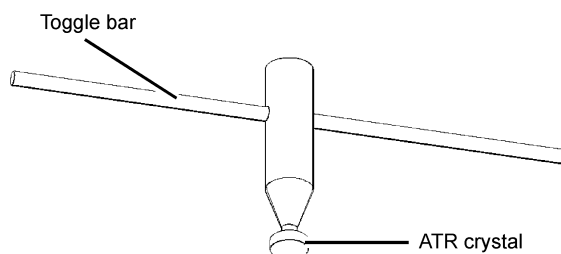
To use the microscope for conventional microspectroscopy, you need only retract the ATR crystal from its working position. If, however you need a very large working distance, for example if the sample is recessed, the ATR crystal holder can be removed.

1. Switch off the microscope at the electricity supply.
2. Stop any laser radiation from entering the microscope by switching the internal beam of the spectrometer to the internal sample compartment.  
See the Spectrum on-screen Help for further information.
3. Make sure that the ATR crystal is in the raised position.
4. Screw the plastic protective cover in place, over the crystal.
5. For the automated ATR objective, remove the jack plug from its socket.
6. While holding the crystal assembly, unscrew the two adjustment thumb-nuts completely. Allow the assembly to drop vertically from the cassegrain. Be careful not to disturb the two adjusting levers.  
Any movement of the adjusting levers will change the alignment of the assembly when it is refitted.
7. Store the assembly carefully for future use.  
When refitted, the assembly will still be aligned, provided that the adjusting levers have not been moved.
8. Direct the infrared beam back to the microscope.

## **Fitting a Crystal Assembly to the ATR Objective**

### **Manual ATR Objective**

1. Remove the ATR crystal holder, as described on page 79.
2. Place the crystal holder on a bench, with the crystal upwards.
3. While holding the crystal assembly in place, unscrew the knurled height-adjusting nut.  
The crystal assembly is under spring pressure. If you do not hold the assembly in place, the spring may be lost.
4. Remove the crystal assembly.  
Leave the spring in the ATR crystal holder.
5. Place the new crystal assembly into the ATR crystal holder and refit the height-adjusting nut.  
Take care not to damage the crystal.
6. Refit the ATR crystal holder to the microscope as described on page 73.
7. Align and adjust the ATR crystal holder, as described on page 77.

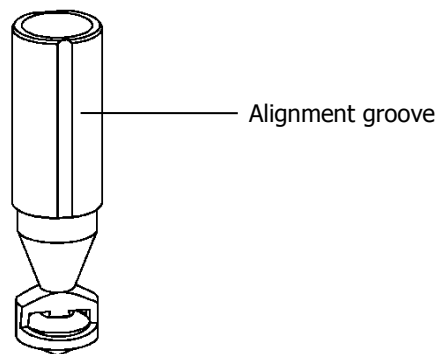


**Figure 37 The Manual ATR Crystal Assembly**

### **Automated ATR Objective**

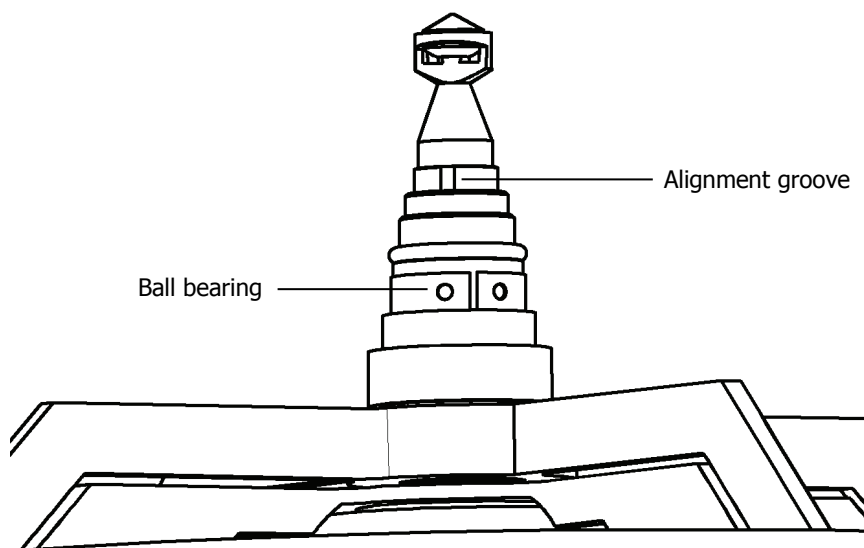
1. Remove the ATR crystal holder, as described on page 79.
2. Place the crystal holder on a bench, with the crystal pointing upwards and the motor pointing away from you.
3. Unscrew the knurled height-adjustment nut.
4. Pull the crystal assembly out of the holder.
5. Hold the new crystal assembly with the thin alignment groove facing you (Figure 38).





**Figure 38 The Automated ATR Crystal Assembly**

6. Slide the crystal assembly into the holder.  
You will feel it click into place as it comes into contact with a magnet inside the holder.
7. Rotate the crystal assembly until the alignment groove is in line with the sprung-loaded ball bearing in the wall of the holder (Figure 39).  
You will feel the ball bearing slide into the alignment groove and lock the crystal assembly in position.



**Figure 39 Crystal Assembly aligned in holder**

8. Refit the height-adjustment nut.  
Take care not to damage the crystal.
9. Refit the ATR crystal holder to the microscope, as described on page 73.
10. Align and adjust the ATR crystal holder, as described on page 77.

## ***Cleaning the ATR Objective Crystal***

---

Because the ATR objective crystal is in contact with the sample under test, it may become dirty during use. A dirty crystal may give spectra of the contaminant rather than the sample under test.

Great care must be taken when cleaning the crystal. It should be cleaned using the minimum of mechanical pressure, with a soft brush or lens tissue. Isopropanol or n-hexane may be used to clean the crystal.

**NOTE:** Do not use acetone or xylene to clean the crystal.

1. Pour a small volume of a solvent into a shallow dish.
2. Lower the sample stage, to allow the dish to be placed on the stage under the crystal.
3. Raise the stage, so that the tip of the crystal is just immersed in the solvent.

**NOTE:** Do not immerse the whole of the ATR crystal in the solvent.

4. Leave the crystal in the solvent for 5 minutes.  
We recommend that you do not leave the crystal in the solvent for longer periods.
5. If any material remains on the crystal, rub the crystal gently with a soft brush or lens tissue.

## ***Auto ATR Cleaning Procedure***

---

If you should see intermittent failures to raise or lower the crystal while running, it is recommended you clean your Auto ATR Accessory to keep it in good working order. Regardless of performance we recommend you clean the accessory at least once every 10,000 cycles or per the schedule below:

- Heavy User (25,000 cycles per year) – Every 4-5 months
- Medium User (15,000 cycles per year) – Every 8 months
- Light User (5,000 cycles per year) – Every 12 Months

### ***Materials Needed***



Methanol in a squirt bottle

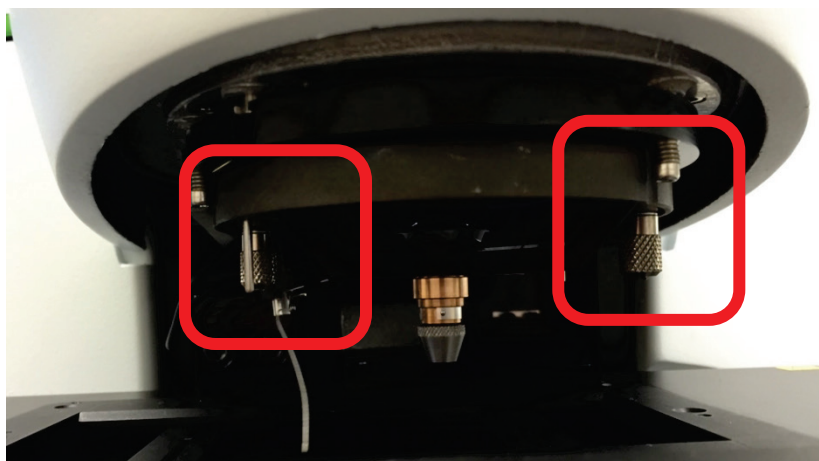


Small Foam Head Swab  
(Recommend [VWR P/N 89022-992](#))

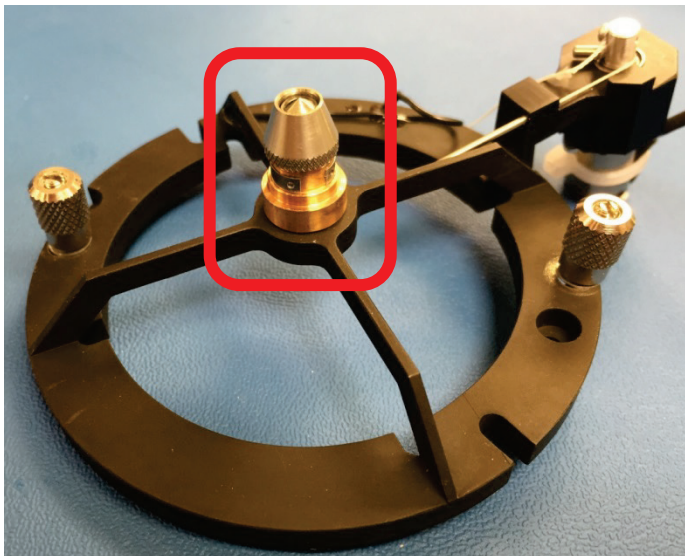
- Kimwipes or similar low lint cleaning wipes

### ***Cleaning Procedure***

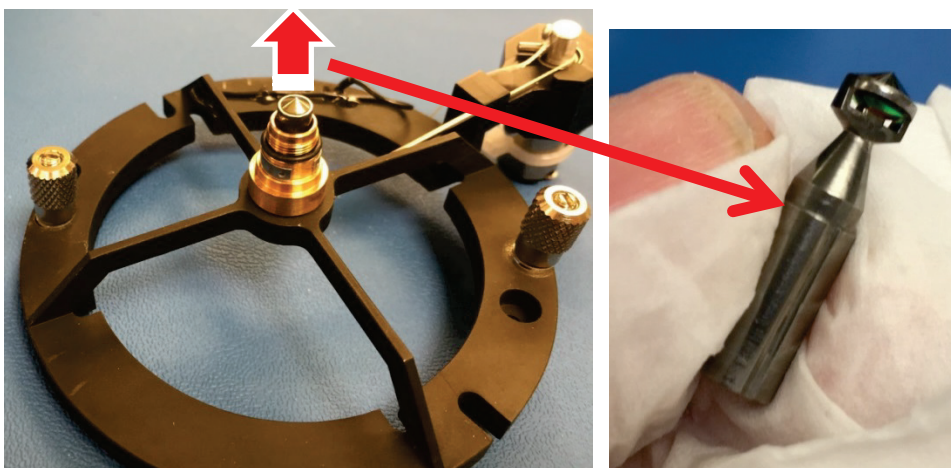
1. If not already done, use Spectrum 10 to raise the crystal into the assembly.
2. Disconnect the motor wire from the left side of the transceiver board.
3. Lower the sample stage, loosen the thumb screws, and carefully pull Auto ATR accessory from Spotlight microscope to remove it from the sample area.



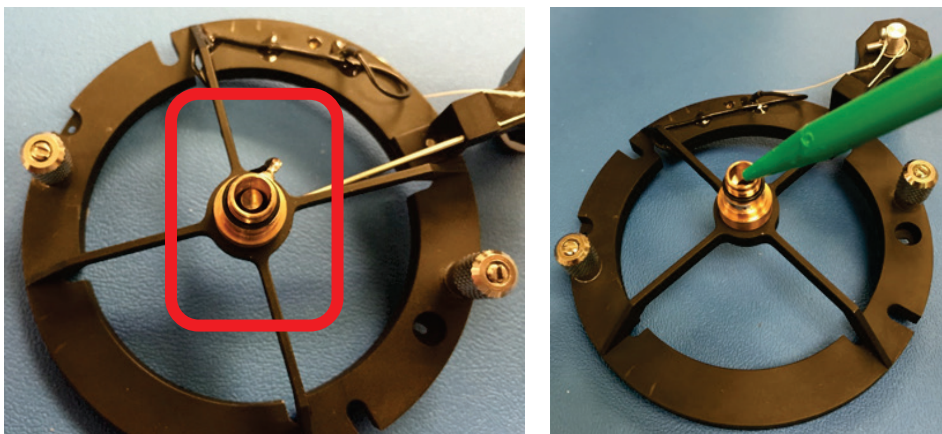
4. Lay the assembly, crystal up, on a clean work surface. Remove the coned knurled collar to expose the crystal holder.



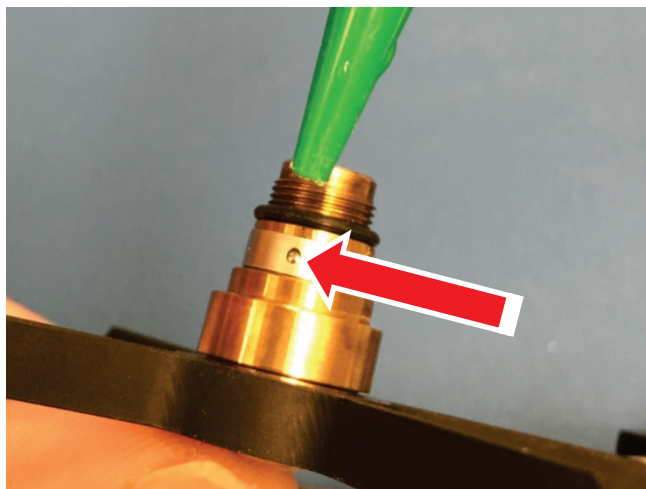
5. Carefully grab the crystal holder and remove it from the ATR assembly. Set the crystal holder aside and be careful not to damage the crystal.



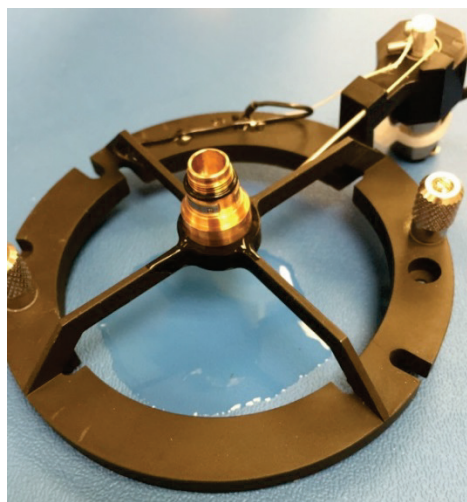
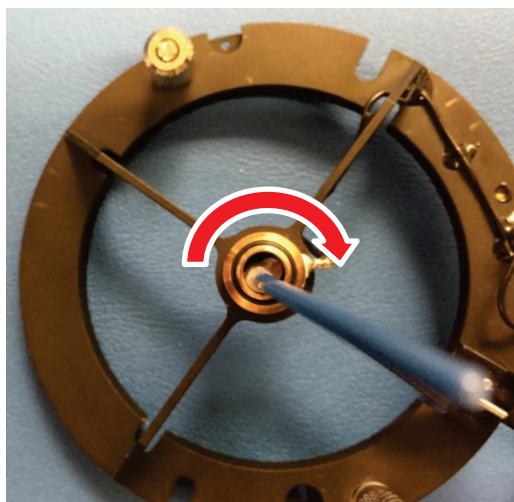
6. Look down into the center of the crystal bearing, notice the compression spring and the plunger retracted and off to one side. Spray methanol into the bore and allow it to drain from the assembly.



7. Spray methanol in hole above alignment bearing to flush out any free particles.



8. Insert a methanol moistened swab into the larger gap between the bearing and the retracted plunger and work it all the way around the bore of the bearing.

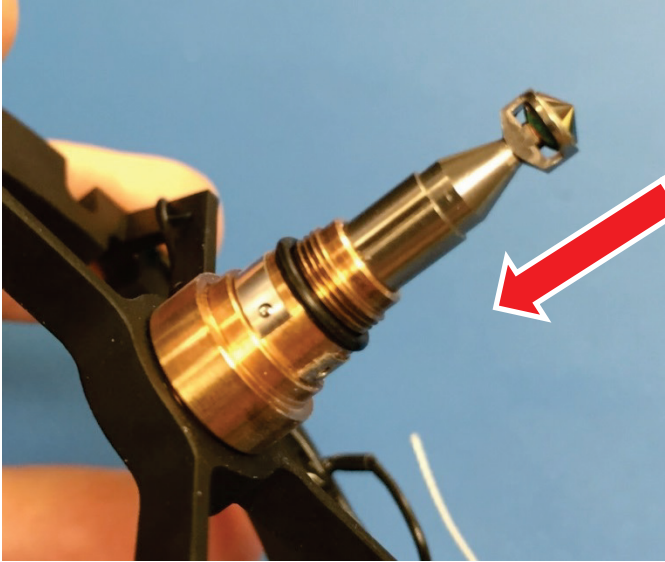


9. Use a methanol moistened Kimwipe to clean the outside bearing surface, alignment groove and contact flat opposite the groove on the crystal holder.  
It may be necessary to use a non-marring implement to clean any debris on the sliding surfaces (I use a finger nail). Take care not to damage the crystal. This is a good time to clean the crystal itself, refer to the User Guide for suggestions.





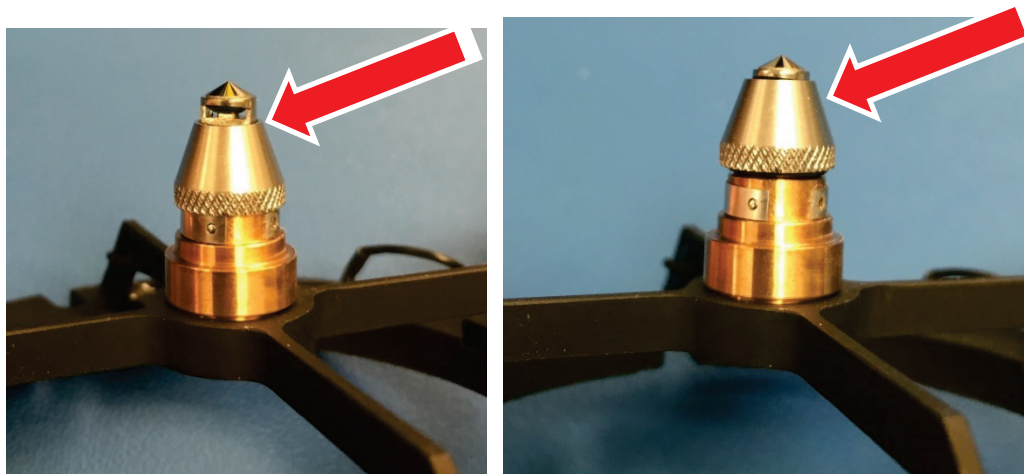
10. Once all of the methanol has evaporated, it's time to reassemble the system. Insert the crystal holder into the bore of the bearing being careful to line up the groove with the ball in the crystal holder.
11. Gently press down on the crystal holder until it stops. You may need to twist the holder a small amount from side to side to seat it on the retracted plunger. **Do not force it**, you may damage the stop contact.



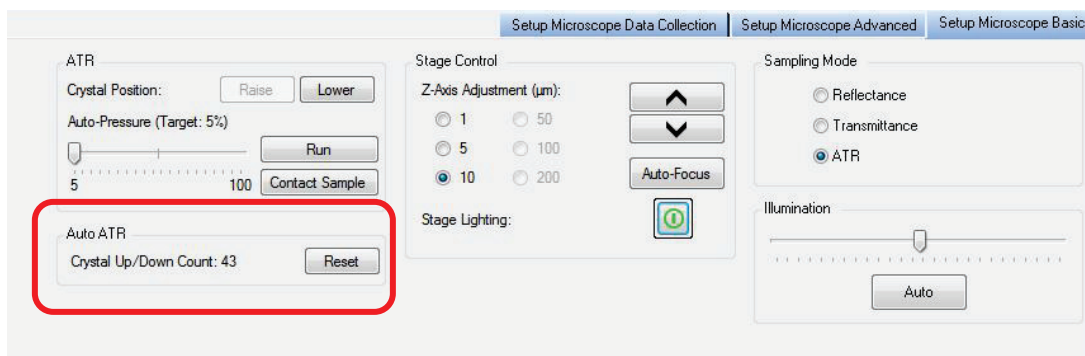
12. Using another swab, clean any debris from the knurled stop collar. Notice the black spots in the image below.



13. Screw the knurled collar on all the way then back off one and a half (1.5) full revolutions. This will set the lower limit of travel for the crystal.



14. Reinstall the Auto ATR accessory in your Spotlight system and perform the crystal alignment and focusing procedure to maximize IR energy.
15. Reset the counter in the Spectrum software on the **Setup Microscope Basic** tab, click the **Reset** button to reset the **Auto ATR Crystal Up/Down** counter.



## ***Collecting Spectra with the ATR Crystal***

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### ***Manual ATR Objective***

#### ***Collecting a Background Spectrum***

##### **CAUTION**

*To prevent serious damage to the crystal, do not move the sample stage while the ATR crystal is in the lower, working position and touching, or near to the sample.*

To collect a background spectrum in air:

1. Select **ATR** in the Sampling Mode section of the Setup Microscope tab.
2. Select the **Scan Settings** you want in the Setup Instrument Basic tab.
3. Set the infrared aperture to a suitable size and rotation on the Setup Microscope tab.  
For information on how to do this see the Spectrum on-screen Help.  
The contact area of the ATR objective is approximately 100  $\mu\text{m}$  in diameter. We therefore recommend that you use a maximum aperture size of 100  $\times$  100  $\mu\text{m}$ .
4. Select **Background** from the Measurement menu.  
The software will prompt you to lower the stage to prevent a collision.
5. Lower the stage and click **OK**.  
The software will prompt you to lower the ATR crystal.
6. Use the toggle bar to lower the crystal when prompted and click **OK**.  
The spectrometer starts to scan and the spectrum is displayed as it is collected.
7. Retract the ATR crystal when the scan is completed.

#### ***Collecting a Sample Spectrum***

Once you have collected a background spectrum, follow the procedure below to collect a spectrum of your sample.

1. Make sure that the ATR crystal is retracted.
2. Place the sample slide on the stage.
3. Move the sample to the center of the field of view.  
Even though the microscope may not be focused on the sample, you can see a change in the light intensity as the sample moves into the center of the field of view.
4. Look at the image on the Setup Microscope tab and focus on the sample using the microscope focus controls, then move the area of interest into the center of the field of view.



5. Select **Monitor** from the Measurement menu.  
The Live tab is displayed.

**NOTE:** Before you collect the sample spectrum, you should monitor the spectrum to make sure that there is good contact between the crystal and the sample.

6. Use the toggle bar to lower the ATR crystal to its working position.
7. Raise the stage carefully until the sample touches the crystal.

**CAUTION**

*To prevent accidental damage to the ATR crystal, always bring the sample slowly up to the crystal, rather than lowering the crystal directly on to the sample.*

8. Select **Sample**.  
The spectrum at the current aperture position is displayed.
9. Make sure that the spectrum monitored is satisfactory.  
If your sample is soft or there is poor contact, you may need to increase the contact of the crystal with the sample, by raising the stage slightly.
10. When the spectrum is satisfactory, click **Scan**.  
The software will again prompt you to lower the crystal and raise the stage but these messages can be cleared by clicking **OK**. The spectrometer starts to scan and the spectrum is displayed as it is collected.

**CAUTION**

*Always retract the ATR crystal immediately after data collection has been completed, in order to avoid accidental damage.*

## ***Automated ATR Objective***

### ***Collecting a Background Spectrum***

To collect a background spectrum in air:

1. Select **ATR** in the Sampling Mode section of the Setup Microscope tab.
2. Select the **Scan Settings** you want in the Setup Instrument Basic tab.
3. Set the infrared aperture to a suitable size and rotation on the Setup Microscope tab.  
For information on how to do this see the Spectrum on-screen Help.  
The contact area of the ATR objective is approximately 100 µm in diameter. We therefore recommend that you use a maximum aperture size of 100 × 100 µm.
4. Select **Background** from the Measurement menu.  
The software will prompt you to lower the stage to prevent a collision when the ATR crystal is lowered.

5. Lower the stage and click **OK**.

The automated ATR crystal will be lowered automatically. The spectrometer starts to scan and the background spectrum is displayed as it is collected.

### **Collecting a Sample Spectrum**

Once you have collected a background spectrum, follow the procedure below to collect a spectrum of your sample.

1. Make sure that the ATR crystal is retracted.
2. Place the sample slide on the stage.
3. Move the sample to the center of the field of view.  
Even though the microscope may not be focused on the sample, you can see a change in the light intensity as the sample moves into the center of the field of view.
4. Look at the image on the Setup Microscope tab and focus on the sample using the microscope focus controls, then move the area of interest into the center of the field of view.
5. Select **Monitor** from the Measurement menu.  
The Live tab is displayed. The software will prompt you to lower the stage to prevent a collision when the ATR crystal is lowered.
6. Lower the stage and click **OK**.  
The automated ATR crystal will be lowered automatically.

**NOTE:** Before you collect the sample spectrum, you should monitor the spectrum to make sure that there is good contact between the crystal and the sample.

7. Raise the stage carefully until the sample touches the crystal.

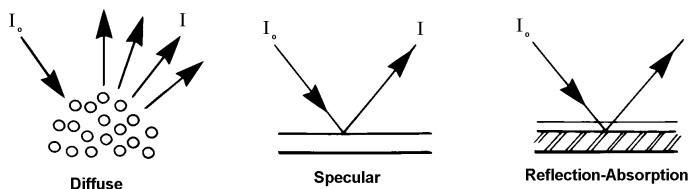
#### **CAUTION**

*To prevent accidental damage to the ATR crystal, always bring the sample slowly up to the crystal, rather than lowering the crystal directly on to the sample.*

8. Select **Sample**.  
The spectrum at the current aperture position is displayed.
9. Make sure that the spectrum monitored is satisfactory.  
If your sample is soft or there is poor contact, you may need to increase the contact of the crystal with the sample, by raising the stage slightly.
10. When the spectrum is satisfactory, click **Scan**.  
The software may prompt you to raise the stage to make contact with the sample. Click **OK** to proceed. The spectrometer starts to scan and the spectrum is displayed as it is collected.

## Reflectance FT-IR Microspectroscopy

The Spotlight 150 enables you to collect images and spectra from samples that display any of the three types of reflectance: diffuse reflectance, specular reflectance, or reflection-absorption.



**Figure 40 Three types of reflectance**

Figure 40 shows how the incident radiation ( $I_0$ ) is reflected in each type of reflectance. It is not uncommon for two or more of these processes to occur simultaneously, depending on the structure of the sample.

### Diffuse Reflectance

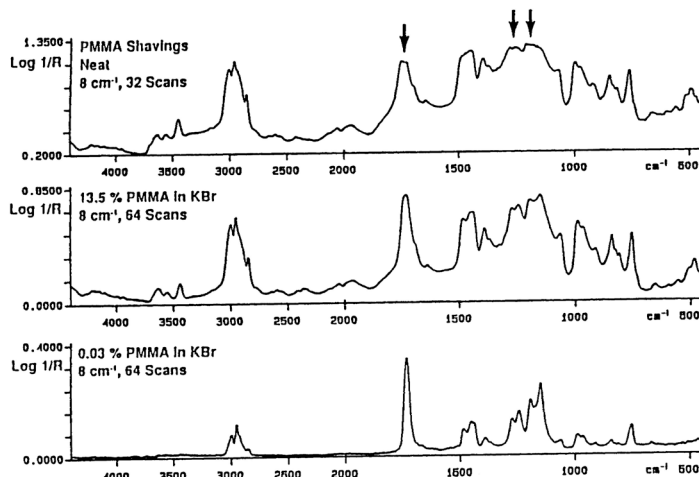
In diffuse reflectance, the incident radiation is reflected in all directions from the surface of the sample. This type of reflectance is seen in samples with matt surfaces, such as paper and powders. The broad collection angle of the microscope enables it to capture a large proportion of the diffusely reflected radiation and send it to the detector.

A problem often encountered when using the diffuse reflectance technique is that there is a large specular component in the reflected radiation. Figure 41 shows three diffuse reflectance spectra of polymethyl methacrylate (PMMA) shavings. In the top spectrum, the shavings were neat. In the next two spectra, the PMMA was diluted with successively larger amounts of KBr.

In the neat sample, the presence of interfering specular reflectance introduces non linearities to the spectral data. For example, the relative intensities of the strong C=O and C-O stretching absorptions are not as expected (arrows on the top spectrum).

As the sample is diluted in a non-absorbing matrix (KBr), specular reflectance is minimized. The bottom spectrum more closely matches an absorption spectrum.

**NOTE:** Sample cups for obtaining diffuse reflectance spectra of powders are available from PerkinElmer: regular cup (part number 01862760), micro cup (part number 01862761).

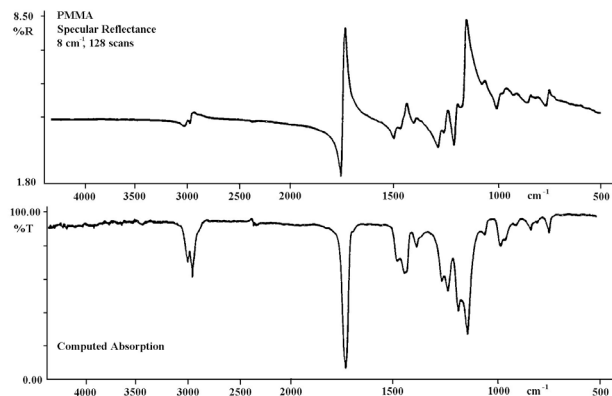


**Figure 41** Diffuse reflectance spectra of PMMA

## Specular Reflectance

Specular reflectance is reflection in one direction (Figure 40). This is the type of reflection that occurs from a smooth, polished surface.

Absorption information cannot be obtained directly from a specular reflectance spectrum of a dielectric material, because the reflectance spectrum is governed by dispersion in the refractive index. However, you can use the Kramers–Kronig integration to calculate the absorbance spectrum from the specular reflectance spectrum. This integration is performed by the Kramers–Kronig process in Spectrum.

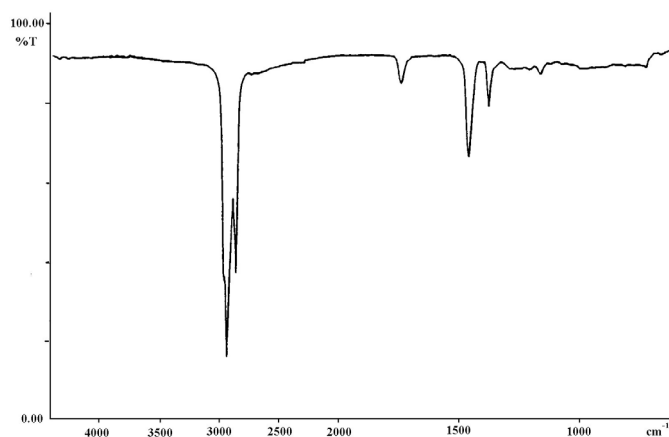


**Figure 42** Specular reflectance spectrum of a PMMA plate and the absorption spectrum calculated from it with the Kramers–Kronig process in Spectrum

The greatest limitation to this method is the presence of an interfering diffuse reflection signal. This occurs when the sample surface is not perfectly smooth. The Kramers–Kronig integration is not appropriate for spectra from such samples.

### ***Reflection-Absorption***

Reflection-absorption occurs when the incident radiation passes through a thin, absorbent film that is on a reflective surface (typically a metal) and is then reflected back through the film (see Figure 40). The absorbance spectrum of the film can be collected directly. Figure 43 shows the spectrum of residual oil on an electronic contact. The sample diameter was 100  $\mu\text{m}$ .



**Figure 43 Reflection-absorption spectrum of residual oil on an electronic contact**

# **Maintenance**

## Maintenance

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*Switch off the mains voltage and remove the mains cord before cleaning.*

A Responsible Body must perform routine maintenance on your microscope to make sure that it is safe and performing well. This involves:

- Inspecting the microscope;
- Protecting the microscope;
- Cleaning the optics;
- Cleaning the microscope cover;
- Renewing the fuse;
- Service.

This chapter contains a list of the available spare parts and optional equipment for the microscope.

If you need to replace a part, use only PerkinElmer approved spare parts.

### CAUTION

*The covers of the microscope must only be removed by a PerkinElmer Service Representative.*

## Inspecting the Microscope

At least once a year, or whenever the microscope has been subjected to adverse environmental conditions, visually inspect the housing to make sure that no covers are loose or distorted.

At the same time, make sure that all required labels are firmly in place; see *Warning Labels* on page 17.

## ***Protecting the Microscope***

The most important rule in caring for the microscope is to keep it as free from dust and dirt as possible. Dust, fingerprints, and smears on the optics reduce the quality of the images it produces.

Whenever the microscope is not in use, cover it with a plastic cover.

### **CAUTION**

*Ensure the power is switched off when the microscope is covered.*

## ***Cleaning***

The microscope was aligned, cleaned, and sealed at the factory. Do not attempt to take it apart.

### **CAUTION**

*Avoid the excessive use of solvents. Flowing solvents dissolve the cement on cemented optics; dissolved cement can damage mirror surfaces.*

## ***Cleaning the Cover***

### **CAUTION**

*Ensure the power is switched off and the supply lead is disconnected before cleaning the cover.*

You can clean the outside of the microscope using a damp cloth. Mild detergent may be used, if necessary. Always perform a patch test on an inconspicuous area, before you clean the entire instrument.

Avoid spilling liquid into the microscope. Clean all external spills immediately. If anything that is spilled enters the main body of any part of the system, switch off the power and call a PerkinElmer Service Representative.



## ***Replacing the Microscope Fuse***

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The microscope is not voltage sensitive and will operate at voltages between 100–240 V and at 50–60 Hz.

1. Switch off the microscope and disconnect it from the power supply.
2. Insert a screwdriver into the slot at the side of the fuse drawer; pull out and flip to one side over the mains inlet.  
The fuse may now be removed.
3. Fit the replacement fuse into the fuse drawer.  
You require a 2.0 A time-lag, 250 V fuse (part number 04970839).
4. Refit the fuse drawer.

## ***Service***

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All optical and mechanical equipment requires periodic servicing to keep it performing properly and to compensate for wear. We recommend that the Spotlight 150 is cleaned, examined, and adjusted periodically by a PerkinElmer Service Representative.

**NOTE:** If you experience unexpected problems with the microscope, contact your PerkinElmer office or representative immediately.

## Accessories

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Accessories can be ordered directly from PerkinElmer at <https://shop.perkinelmer.com/default.aspx>

| Description                                     | Part Number |
|---|-------------|
| Spare fuse for microscope (2 A time-lag, 250 V) | 04970839    |
| Visible Polarizer Kit                           | L1860294    |
| Sampling Accessory Kit                          | L1860250    |
| IR Polarizer Assembly                           | L1860408    |
| Manual ATR Accessory                            | L1860334    |
| Auto ATR Accessory                              | L1862046    |
| Holder for 13 mm Sampling Disks                 | L1860409    |
| Compression Cell                                | N1870185    |
| Rotatable Sample Disk Holder                    | N1873039    |
| Holder for Reflection Transmission Measurements | N1873124    |
| BaF <sub>2</sub> Window (1 mm thick)            | N9302611    |
| BaF <sub>2</sub> Window (2 mm thick)            | N9302612    |
| ZnSe Window                                     | N9302613    |
| NaCl Window                                     | N9302614    |
| KBr Window                                      | N9302615    |
| Miniature Diamond Anvil Cell                    | N9302618    |
| Spare Germanium Crystal (Manual)                | L1860268    |
| Spare Silicon Crystal (Manual)                  | L1860269    |
| Spare Diamond Coated Germanium Crystal (Manual) | L1862054    |
| Spare Germanium Crystal (Auto)                  | L1862044    |
| Spare Silicon Crystal (Auto)                    | L1862051    |
| Spare Diamond Coated Germanium Crystal (Auto)   | L1862047    |

## Sample Preparation Tools

These are described in *Tools for Sample Preparation* on page 47.

## Electrical Connections

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### Fitting the Plug

The power cable for the electrical supply plugs into the back of the microscope. It has a molded connector at one end. If it is necessary to fit a plug on the power cable, use the wire color code below:

| Plug Pin       | Wire Color<br>(100/110/120 V) | Wire Color<br>(220/230/240 V) |
|----------------|-------------------------------|-------------------------------|
| Ground (Earth) | Green or Green/Yellow         | Green/Yellow                  |
| Line           | Black                         | Brown                         |
| Neutral        | White                         | Blue                          |

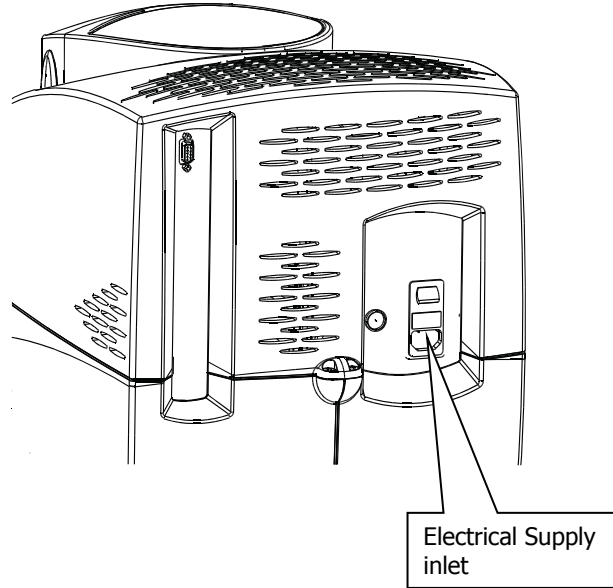


*To ensure safe and satisfactory operation of the instrument, it is essential that the green or green/yellow ground (earth) wire of the power cord is connected to a ground that complies with the regulations of the local electricity supply authority (or equivalent body); ground circuit continuity is essential for safe operation of the equipment.*

## Connecting the Microscope to the Electrical Supply


The microscope operates on an electrical supply with a frequency of 50 or 60 Hz and at voltages in the range 100 to 240 V without adjustment.

Fit the molded connector of the power cable into the inlet at the rear of the microscope.



**Figure 44** The location of the electrical supply inlet

## Connecting the Microscope to the Spectrometer

The cable from the microscope to the spectrometer comes from the preamp inside the microscope, to a connector marked  on the rear of the spectrometer.

The connections from the pre-amp in the microscope to the spectrometer are described below:

| PI 1 (PCB) | D-type Connector | Line                | Power Requirement |
|------------|------------------|---------------------|-------------------|
| 1.         | 11               | Output Signal       |                   |
| 2.         | 10               | Output Signal (0 V) |                   |
| 3.         | 9                | 0 V                 |                   |
| 4.         | 12               | 0 V                 |                   |
| 5.         | 13               | +12 V               | 250 mA            |
| 6.         | 14               | −12 V               | 200 mA            |

# **Appendices**

## ***Appendix 1: Decontamination and Cleaning***

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Before using any cleaning or decontamination methods except those specified by PerkinElmer, users should check with PerkinElmer that the proposed method will not damage the equipment.

### ***Decontamination***

**Customers wishing to return instrumentation and/or associated materials to PerkinElmer for repair, maintenance, warranty or trade-in purposes are advised that all returned goods must be certified as clean and free from contamination.**

The customer's responsible body is required to follow the "Equipment Decontamination Procedure" and complete the "Certificate of Decontamination". These documents are available on the PerkinElmer public website:

<http://las.perkinelmer.com/OneSource/decontamination.htm>

Alternatively, if you do not have access to the internet contact Customer Care:

|                              |                   |                   |
|------------------------------|-------------------|-------------------|
| <b>Customer Care USA:</b>    | 1-800-762-4000    | (inside the USA)  |
| (8:30 a.m. – 7 p.m. EST)     | (+1) 203-925-4602 | (outside the USA) |
| <b>Customer Care Canada:</b> | 800-561-4646      |                   |
| <b>Customer Care EU:</b>     | 0800 40 858       | (Brussels)        |
|                              | 0800 90 66 42     | (Monza)           |

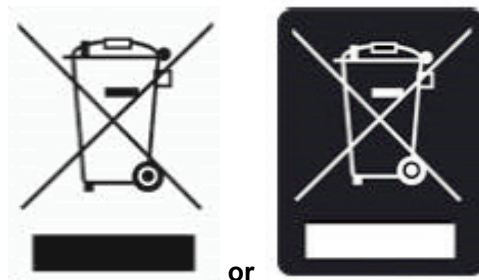
If you are located outside of these regions, please call your local PerkinElmer sales office for more information.

### ***Cleaning the Instrument***

Exterior surfaces may be cleaned with a soft cloth, dampened with a mild detergent and water solution. Do **not** use abrasive cleaners or solvents.

## **Appendix 2: WEEE Instructions for PerkinElmer Products**

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A label with a crossed-out wheeled bin symbol and a rectangular bar indicates that the product is covered by the Waste Electrical and Electronic Equipment (WEEE) Directive and is not to be disposed of as unsorted municipal waste. Any products marked with this symbol must be collected separately, and in accordance with the regulatory guidelines in your area.

The objectives of this program are to preserve, protect and improve the quality of the environment, protect human health, and utilize natural resources prudently and rationally. Specific treatment of WEEE is indispensable in order to avoid the dispersion of pollutants into the recycled material or waste stream. Such treatment is the most effective means of protecting the customer's environment.

The requirements for waste collection, reuse, recycling, and recovery programs are set by the regulatory authority in your location. Contact your local responsible person (such as your laboratory manager) or authorized representative for information regarding applicable disposal regulations.

See the PerkinElmer web address below for information specific to PerkinElmer products, and contact details for the Customer care department in your region.

<http://las.perkinelmer.com/OneSource/Environmental-directives.htm>

Products from other manufacturers may also form a part of your PerkinElmer system. These other manufacturers are directly responsible for the collection and processing of their own waste products under the terms of the WEEE Directive. Please contact these manufacturers directly before discarding any of their products. Consult the PerkinElmer web address (above) for manufacturer's names and web sites.



# Index

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## A

|                                |            |
|--------------------------------|------------|
| Accessories                    |            |
| compression cell .....         | 60         |
| diamond anvil cell .....       | 49, 51, 53 |
| illuminator, fiber optic ..... | 49         |
| list .....                     | 99         |
| microtome .....                | 49, 53     |
| pellet press .....             | 51         |
| ATR                            |            |
| collecting spectra .....       | 88         |
| measuring energy .....         | 75         |
| ATR crystal                    |            |
| adjusting height .....         | 76         |
| centering .....                | 77         |
| cleaning .....                 | 82         |
| IR alignment .....             | 78         |
| ATR crystal holder             |            |
| fitting .....                  | 73         |
| removing .....                 | 79         |
| ATR objective                  |            |
| crystal assembly .....         | 80         |
| description .....              | 70         |
| optical path .....             | 71         |
| specification .....            | 72         |
| Attenuators .....              | 41, 42     |
| Auto ATR crystal               |            |
| cleaning .....                 | 83         |

## C

|                                      |         |
|--------------------------------------|---------|
| Cassegrain .....                     | 24      |
| removal and refitting of lower ..... | 61      |
| Cleaning                             |         |
| ATR crystal .....                    | 82      |
| Auto ATR crystal .....               | 83      |
| microscope cover .....               | 96      |
| optical system .....                 | 96      |
| Cleaning the instrument .....        | 103     |
| Compliance                           |         |
| EC directive .....                   | 20      |
| EMC .....                            | 20      |
| FCC .....                            | 20      |
| Connections                          |         |
| microscope .....                     | 25, 101 |
| power .....                          | 100     |
| Conventions .....                    | 7       |
| Cooling                              |         |
| MCT detector .....                   | 35      |
| Counter, reset .....                 | 87      |

## D

|                   |    |
|-------------------|----|
| Definitions ..... | 10 |
|-------------------|----|

## F

|                  |        |
|------------------|--------|
| Fibers .....     | 66     |
| Fuse             |        |
| microscope ..... | 21, 97 |

## I

|                        |        |
|------------------------|--------|
| Illuminator .....      | 24     |
| Image                  |        |
| collecting .....       | 30     |
| focusing .....         | 40, 41 |
| in reflectance .....   | 31     |
| in transmittance ..... | 30     |
| IR polarizer .....     | 68     |
| Inert atmosphere ..... | 62     |
| Inspection             |        |
| microscope .....       | 95     |
| IR polarizer           |        |
| equipment .....        | 68     |
| operation .....        | 69     |

## L

|                        |    |
|------------------------|----|
| Liquids                |    |
| small quantities ..... | 57 |
| Location .....         | 16 |

## M

|                                   |     |
|-----------------------------------|-----|
| Maintenance .....                 | 95  |
| inspection .....                  | 95  |
| microscope fuse .....             | 97  |
| protection .....                  | 96  |
| servicing .....                   | 98  |
| MCT detector                      |     |
| cooling .....                     | 35  |
| Microscope                        |     |
| electrical connections .....      | 101 |
| fuse .....                        | 97  |
| inspecting .....                  | 95  |
| protecting .....                  | 96  |
| Microtome .....                   | 53  |
| acrylic resins .....              | 53  |
| beta-pinene wax .....             | 53  |
| epoxy resins .....                | 53  |
| paraffin wax .....                | 53  |
| plastic embedding materials ..... | 53  |
| Mulls                             |     |
| fluorolube .....                  | 56  |
| nujol .....                       | 56  |

## O

|                            |    |
|----------------------------|----|
| Operating conditions ..... | 14 |
| Optical system .....       | 27 |
| cleaning .....             | 96 |

**P**

|                    |     |
|--------------------|-----|
| Polarization       |     |
| theory .....       | 64  |
| Power cables ..... | 100 |
| Protecting         |     |
| microscope .....   | 96  |
| Purge system ..... | 62  |
| Pyrolysis .....    | 55  |

**R**

|                             |    |
|-----------------------------|----|
| Reflectance                 |    |
| description .....           | 91 |
| diffuse .....               | 91 |
| reflection-absorption ..... | 93 |
| specular .....              | 92 |
| Related documents .....     | 10 |
| Requirements                |    |
| electrical .....            | 21 |
| site .....                  | 21 |
| system .....                | 21 |

**S**

|                               |    |
|-------------------------------|----|
| Safety                        |    |
| electrical .....              | 15 |
| labels .....                  | 17 |
| specifications .....          | 22 |
| Sample                        |    |
| accessories .....             | 49 |
| area .....                    | 46 |
| flattening .....              | 46 |
| other useful tools .....      | 48 |
| preparation .....             | 46 |
| preparation tools .....       | 47 |
| thickness .....               | 46 |
| viewing .....                 | 27 |
| Sample preparation            |    |
| coatings .....                | 57 |
| elastomers .....              | 54 |
| fibers .....                  | 56 |
| fibrous solids .....          | 56 |
| filled polymers .....         | 55 |
| liquids .....                 | 57 |
| micro amounts .....           | 55 |
| particles .....               | 55 |
| polymers .....                | 53 |
| techniques .....              | 51 |
| Service .....                 | 98 |
| Setting scan parameters ..... | 42 |
| Setting up .....              | 38 |
| Specification                 |    |
| ATR objective .....           | 72 |
| Spectra                       |    |
| collecting .....              | 60 |
| Spotlight 150                 |    |

|                       |    |
|-----------------------|----|
| before using .....    | 34 |
| features .....        | 24 |
| focusing .....        | 39 |
| lifting .....         | 19 |
| operation .....       | 26 |
| scan parameters ..... | 42 |
| setting up .....      | 38 |

**T**

|                                      |        |
|--------------------------------------|--------|
| Techniques                           |        |
| compressing .....                    | 51     |
| crushing .....                       | 55     |
| diamond anvil cell, using .....      | 53     |
| flattening solids .....              | 51     |
| for biological substances .....      | 66     |
| for fibers .....                     | 66     |
| for laminates .....                  | 65     |
| for minerals & crystals .....        | 66     |
| for polymer films .....              | 66     |
| for soft materials .....             | 60     |
| for thick samples .....              | 61     |
| microbrush, using .....              | 55     |
| microtoming .....                    | 53     |
| mulls, using .....                   | 56     |
| needle, pressing sample with .....   | 52     |
| needle, transferring with .....      | 56     |
| probe, compressing sample with ..... | 52     |
| purge system .....                   | 62     |
| pyrolysis .....                      | 55     |
| salt windows, using .....            | 50     |
| sample preparation .....             | 51     |
| separating by aperturing .....       | 56     |
| slicing from solids .....            | 52     |
| wedge, using .....                   | 52     |
| windows, compressing between .....   | 52     |
| Tools                                |        |
| disk holder .....                    | 47     |
| forceps .....                        | 47     |
| forceps, needle point .....          | 48     |
| forceps, round tip .....             | 48     |
| forceps, wide tip flat .....         | 48     |
| forceps, wide tip hooked .....       | 48     |
| gold mirror .....                    | 47     |
| microbrush .....                     | 57     |
| microdisk .....                      | 48     |
| micropipette .....                   | 57     |
| microprobe .....                     | 48     |
| needle, tungsten alloy .....         | 47     |
| other useful .....                   | 48     |
| pin vise .....                       | 47     |
| probe, steel .....                   | 47     |
| provided .....                       | 47     |
| roller knife .....                   | 47, 51 |
| rotating disk holder .....           | 47     |
| slides .....                         | 47     |

|                                 |    |
|---------------------------------|----|
| support, large samples .....    | 47 |
| tungsten probe .....            | 57 |
| tweezers .....                  | 47 |
| window, salt .....              | 48 |
| Transmission measurements ..... | 43 |

## **V**

|                             |    |
|-----------------------------|----|
| Ventilation .....           | 16 |
| Viewing                     |    |
| in reflectance .....        | 29 |
| in transmittance .....      | 28 |
| sample .....                | 27 |
| Visible polarizer           |    |
| biological substances ..... | 66 |
| equipment .....             | 66 |

|                           |    |
|---------------------------|----|
| fibers .....              | 66 |
| laminate structures ..... | 65 |
| minerals & crystals ..... | 66 |
| operation .....           | 67 |
| polymer films .....       | 66 |
| theory .....              | 64 |

## **W**

|                           |     |
|---------------------------|-----|
| Warning labels .....      | 17  |
| microscope .....          | 18  |
| WEEE Directive .....      | 104 |
| Windows                   |     |
| compressing between ..... | 52  |
| types .....               | 50  |
| Windows Start Menu .....  | 38  |