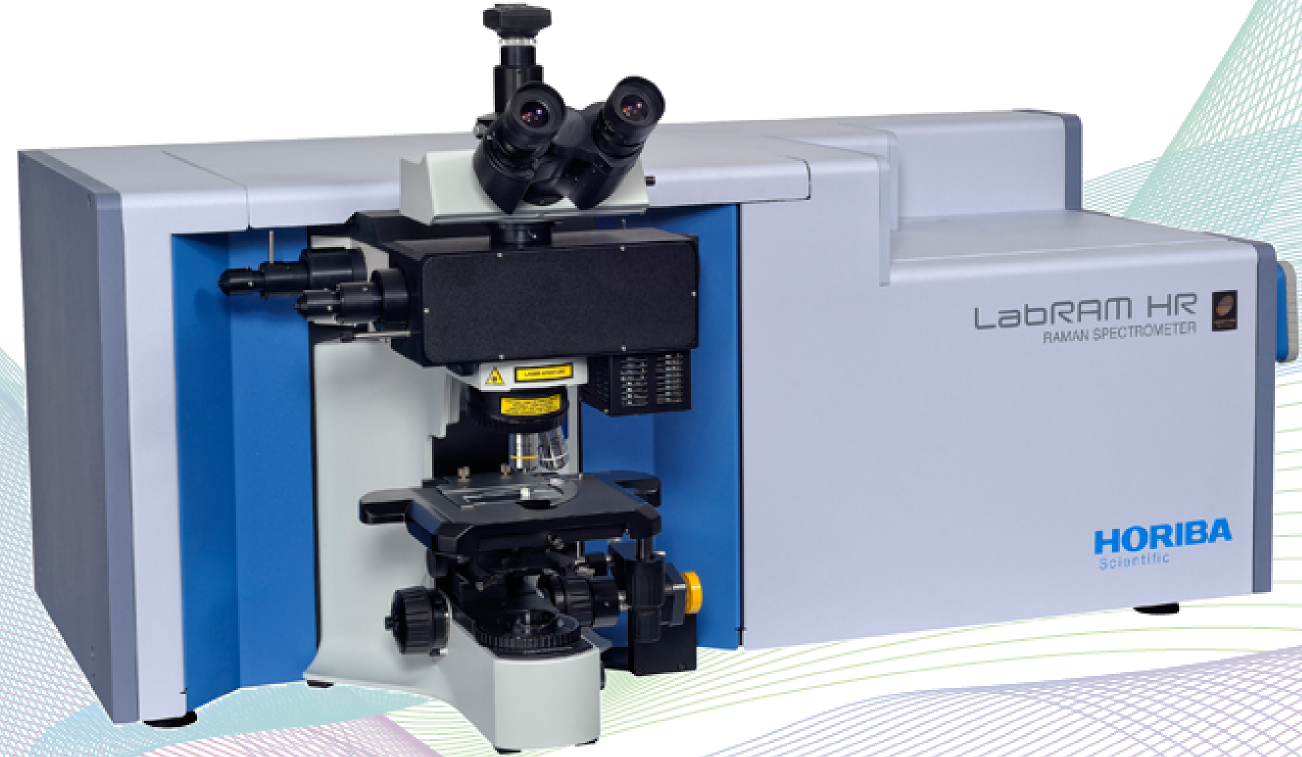
LabRAM HR Evolution

User Manual



Part Number: 31 087 164

Highly compressed PDF version without video tutorials

COPYRIGHT

NOTICE TO THE USER

This manual should not be construed as any representation or warranty with respect to the unit named herein. Occasionally, changes or variations exist in the unit that are not reflected in the manual. Generally, should such changes or variations exist and affect the product significantly, a release note would accompany the manual. In such a case, be sure to read the release note before using the product.
Trademarks: LabRAM, LabSpec are registered trademark of HORIBA, Windows XP/VISTA/7 are registered trademarks of Microsoft Corporation.

NP/LabRAM HR Evolution.fm - 06/26/2013

HORIBA Jobin Yvon S.A.S. **Headquarters**	16-18, rue du Canal 91165 LONGJUMEAU CEDEX (France) Tel: +33 (0)1 69 74 72 00 Fax: +33 (0)1 69 09 07 21 Internet site: http://www.horiba.com
HORIBA Jobin Yvon S.A.S. **Molecular & Microanalysis Division**	231, rue de Lille 59650 - Villeneuve d'Ascq (France) Tel: +33 (0)1 69 74 72 00 Fax: +33 (0)3 20 59 18 08 Email: raman@horiba.com
HORIBA Instruments Inc. **(Edison office)**	3880 Park Avenue, EDISON - New Jersey 08820 (USA) Tel: +1 (732) 494 86 60 - Fax: +1 (732) 549 51 25
HORIBA Jobin Yvon GmbH	Neuhofstrasse 9 - D-64625 BENSHEIM (Germany) Tel: +49 6251 84 75-0 - Fax: +49 6251 84 75-20
HORIBA UK Ltd	2 Dalston Gardens - STANMORE Middlesex HA7 1BQ (Great Britain) Tel: +44 (20) 8204 8142 - Fax: +44 (20) 8204 6142
HORIBA Jobin Yvon Srl	Via Cesare Pavese 35/AB - 20090 OPERA - Milano (Italy) Tel: +39 (2) 57 60 30 50 - Fax +39 (2) 57 60 08 76
HORIBA Trading (Shanghai) Co., Ltd. **(Beijing office)**	Room 1801, Capital Tower - No.6 Jia Jianguomenwai Avenue Chaoyang District - Beijing 100022 (China) Tel: +86 (0) 10 8567 9966 - Fax: +86 (0) 10 8567 9066
HORIBA Ltd **(Tokyo office)**	Kanda-awaji nichome Building 2-6 Kanda-awajicho, Chiyoda-ku, TOKYO, 101-0063 (Japan) Tel: +81 (0) 3 6206 4721
HORIBA Korea Ltd	Dong yang B/D 8F, 112-6, Sokong-Dong, Choong-Gu 100-070 SEOUL (Korea) Tel: +82 2 753 7911 - Fax: +82 2 756 4972

Thank you for choosing HORIBA Scientific

HORIBA Scientific is a world leader in Raman spectroscopy, designing and manufacturing state of the art dispersive Raman spectrometer systems for over four decades.

We have been at the forefront of Raman spectroscopy since its infancy, introducing ground breaking innovations such as the first Raman microscope and the world's first commercial remote Raman sampling probe.

HORIBA Scientific's Raman Division today comprises the combined expertise of the HORIBA Jobin Yvon, Dilor and SPEX companies and is proud of having more than 3000 Raman systems installed worldwide. Well equipped applications labs and full technical support enable HORIBA Scientific to provide services right through from the earliest proof of principle investigations. With a broad range of technologies available, HORIBA Scientific can uniquely provide solutions for analytical, research and industrial Raman analyses.

If you have any questions regarding the installation or the maintenance of your system, please contact one of our representatives:

HORIBA Jobin Yvon SAS

Molecular & Microanalysis Department

231, rue de Lille
59650 Villeneuve d'Ascq
France
raman@horiba.com
Tel: +33 (0)1 69 74 72 00
Fax: +33 (0) 3 20 59 18 08
or visit our website: www.horiba.com/raman to find the local HORIBA Scientific office nearest to you.

Customer support: raman-service-sci.fr@horiba.com
Tel: +33(0)1 69 74 72 02

06/26/2013

Warranty

HORIBA Scientific warranties each instrument for one year from the date on which the equipment leaves the plant. The warranty is limited to repair of operating or manufacturing defects which come to light in that period. It shall apply in said period only if the equipment has been used properly. It will determine inter alia in event of faulty maintenance or use, or of repairs by the Buyer or a third party. The Seller's warranty does not cover «consumable parts» (such as filaments, zeolite joints, electron multipliers, electronic components, membranes, diaphragms, crucibles, glass, etc.).
In the case of components covered by the manufacturer's warranty, such as computers, the Seller shall cooperate with the Buyer in enforcement of the manufacturer's warranty, but the Seller assumes no obligation for such components.

The warranty is limited to replacement of parts found by the Seller to be defective. To be able to claim the benefit of said warranty, the Buyer will have to advise the Seller by return-receipted registered mail within 28 days of discovery of the defects which come to light. The contractual warranty is limited, at the Seller's option, to reconditioning or replacement of parts found by it to be defective, expenses of dismantling, reinstallation and technicians' travel excepted. All warranty work shall be done by the Seller or a contractor of its choosing according to its instructions. All shutdown-related expenses shall in any event be incumbent on the Buyer.

The warranty does not cover the following:

- **Any malfunction attributable to improper operation**
- **Any malfunction attributable to repair or modification by any party not authorized by HORIBA Scientific**
- **Any malfunction attributable to the use in an improper environment**
- **Any malfunction attributable to violation of the instructions in this manual**
- **Any malfunction attributable to operations in the manner not specified in this manual**
- **Any malfunction attributable to natural disasters, or accidents or mishaps not involving HORIBA Scientific**
- **Any deterioration in appearance attributable to corrosion, rust, and so on.**
- **Consumables and replacement of consumables**
- **Products of other companies**

THE SELLER MAKES NO OTHER TACIT OR IMPLIED WARRANTY. IT SHALL HAVE NO LIABILITY FOR INJURY TO THIRD PARTIES, DIRECT OR INDIRECT, COMMERCIAL OR FINANCIAL INJURY, CONSEQUENTIAL DAMAGES, OR FOR ANY OTHER REASON.

Obligations

The products are for professional application, and the Seller's acceptance of an order is with the understanding that only qualified individuals, trained and familiar with users' procedures, will handle them. If Buyer fails to observe this obligation, Buyer agrees to hold the Seller harmless from any liability resulting from the operation of the products.

LabRAM HR Evolution Presentation

The Labram HR evolution is a fully integrated confocal raman microscope instrument offering full automation and high versatility. Its spectrograph combines the high luminosity of a single stage spectrometer and the high spectral resolution because of its long focal length (800mm). More over the achromatic design of the spectrograph offers potentially the access to an extended wavelength range from 200nm to 2000nm (depending on the instrument configuration). The labram HR evolution is ideally suited to both micro and Macro measurements and offers advanced confocal imaging capabilities in 2D and 3D. The true confocal microscope enables the most detailed images and analysis to be obtained with speed and confidence. The labram evolution utilizes HORIBA scientific's Labspec 6 spectroscopy suite. Labspec6 provides complete instrument control and data processing, assuming fast & reliable results.

Contents

General Safety Instructions

IMPORTANT: the General Safety Instructions must be printed and kept near to the system workstation.

S1 General instructions

The LabRAM HR Evolution system is a CLASS 3B laser product. Optional enclosure accessory is available for using the system in CLASS 1 laser product.

Regulations

CLASS 3B laser product IEC60825-1 (2008)

CLASS IIIb (CDRH)

CLASS 1 laser product with optional enclosure accessory

EMC: 2004/108/EC, standard EN 61326-1 (2006), Class A product, basic requirements

LVD: 2006/95/EC, standard EN 61010-1 (2010)

Other regulations: FDA: 21 CFR1040.10

The procedures for using this system are classified as follows:

- **Service**

«Service» means any adjustment or repair performed by HORIBA Service Team or highly trained and skilled HORIBA representatives.

- **Operation**

«Operation» means all performance described in the User Manual. CLASS 3B laser light is only emitted from the objective lens during the actual execution.

- Laser products of Class 3B are products that can only be used under control of laser safety officer.
- Assign a laser safety officer and ensure safety in accordance with the instruction given by the said laser safety officer.
- In "IEC 60825-1, Safety of laser products – Part 1: Equipment classification, requirements and user's guide", it requires safety preventive measures for users of laser products.
- Before using, read the Directive 2006/25/CE carefully and then, use the products after enforcing adequate measures described in the document.

- Unless the safety measures indicated in IEC 60825-1 are provided, our company cannot insure safety of the product since it may cause exposure of dangerous laser emission.

WARNING: The LabRAM HR Evolution System must not be operated without prior reading of this document.

The user manual contains important information on how to operate the LabRAM HR Evolution System correctly, safely and most efficiently. Observing these instructions will enable safe operation of the equipment and will help to avoid accidental damage, to reduce repair costs and to increase the instrument lifetime and reliability.

HORIBA Scientific equipment is perfectly safe as long as it has been properly installed and is operated according to the instructions which are given in this instruction manual.

The installation of the equipment is to be strictly carried out by properly trained personnel who are designated by HORIBA Scientific and should not be attempted by the end user.

This manual must always be available whenever operating the equipment.

Any person working with the LabRAM HR Evolution system, whether it is an engineer or an operator, must be aware of the statements enclosed within this document and apply its contents. Tasks requiring familiarity with the manual include; routine operation (including setting up), sample loading, and instrument troubleshooting.

WARNING: Never make any modifications, additions or conversions to the equipment (especially those which might affect safety) without the supplier approval. This also applies to the installation and adjustment of safety devices and accessories.

In the event of safety relevant modifications or changes in the behavior of the LabRAM HR Evolution during operation, stop the equipment, and namely the laser source, immediately and report the malfunctioning to HORIBA Scientific or your authorized local representatives.

S2 Warnings

- **Performance of any procedures not specified by the manufacturer may result a hazardous radiation exposure.**
- **Only the accessories which meet the manufacturer's specifications shall be used.**
- **In case of filters adaptation, it could be necessary to open the upper door for internal access. This upper door is secured by a laser off safety switch when the door is opened.**
- **There are no user service parts for the LabRAM HR Evolution System. The System, including peripherals, must never be opened with the exception of parts mentioned in this manual. Service must only be performed by designated members of HORIBA Scientific Service Team or by authorized local representatives.**

S3 Laser Radiation Safety

- **In its basic configuration the LabRAM HR Evolution is a class 3B Laser Product.**
- **A class 1 enclosure accessory is available for the microscope and provides a greater level of laser isolation should this be required. It is important that the operators understand which version of the system they will be operating and familiarise themselves with the appropriate safety precautions.**
- **Laser sources used with the LabRAM HR Evolution constitute a hazard to personnel during periods of operating and servicing.**
- **Lasers are high intensity light sources producing visible or invisible light at specific wavelengths. This concentrated energy in a narrow laser beam may cause damage to biological tissues, especially to eyes.**
- **The LabRAM HR Evolution System can be ordered with one internal and/or several external laser sources. In such a case, It is the customer's responsibility to ensure that additional external laser units comply all local laws, regulations and include appropriate safety protection. Each additional laser source must include additional cover(s) preventing access to laser radiation. These lasers must comply the class 3B Laser Product and installed according to the IEC 60825-1 (2008) regulations.**

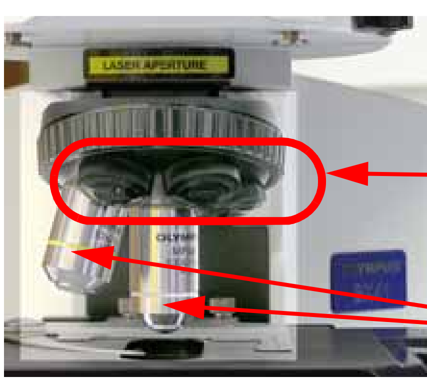
CAUTION:

- **Class 3B laser system: Laser beams and reflected beams could be dangerous. Diffused reflected beams in most cases do not present any problem.**
- **In class 3B operation, the laser beam remains only accessible for the functional use below the microscope and within the sample area. The end-user must ensure that it is operated in a safe condition, with all the class 3B laser safety requirements.**
- **The various parts of the LabRAM HR Evolution giving access to the laser source have been secured by the means of a main unit enclosure. In the optional class 1 version, safety housings enclose the microscope.**
- **The LabRAM HR Evolution System itself can include a 17mW/HeNe/633nm laser source. On request other external laser sources can be ordered but are not integrated inside the LabRAM HR Evolution System. The end-user must comply the legal and safety requirements during the use of the lasers.**
- **The various parts of the LabRAM HR Evolution giving access to the laser beam have been secured by the means of enclosures and tubes from the laser source input to the microscope. These tubes are firmly tightened and prevent any exposure from the operator to any laser radiation. Never remove these protective parts.**

The following precautions must be observed

- Refer to your laser source manual for specific safety requirements.
- In case of modification of the laser source, the LabRAM HR Evolution labelling may be adapted to conform the laser source class.
- Warning signs indicating the area in which the laser is enclosed should be clearly displayed.
- Local and national regulations governing the safe use of lasers should be adhered to at all times.
- Personnel must never look directly into the laser beam and should wear protective eye wear at all times if protective covers are removed while the laser is switched on.
- All personnel in the vicinity of the laser should wear protective eyewear, if protective covers are removed while the laser is switched on. Only qualified and trained personnel should be permitted to operate the laser.
- Never attempt to output the laser beam outside the system by inserting a mirror or a similar object (gold ring) in the light path. Otherwise, the laser beam may enter your eyes, which is extremely hazardous.
- Do not place or remove a sample during the laser exposure.
- Place the sample horizontally on the stage. If the sample inclines, the laser beam may reflect around the microscope, which is extremely hazardous.
- Do not turn the microscope turret during the laser exposure, some out-of-path reflections could exit from the turret.
- Ensure that the laser is properly ventilated using a suitable exhaust. Do not connect the exhaust to breathing air systems (i.e. air conditioning or ventilation systems).
- The extracting fan located at the rear of the laser must not be blocked at any time.
- Viewing laser beam with certain optical instruments (eye loupes, magnifiers, binoculars or telescopes) is strictly forbidden.
- The laser warning labels affixed to the system according to the safety regulations (see warning chapter) must not be removed.
- The laser can only be switched on with the key switch. This prevents inadvertent or unauthorized starting of the laser. It cannot be operated with the key in the OFF position and the key cannot be removed in ON position.
- If mishandling of the instrument or of the safety devices results nevertheless in direct eye exposure to the laser beam, the exposed operator should consult a doctor or a competent eye testing institution.
- Caution: The laser exit is through the microscope objective. Before the System use, check that all objectives or caps are mounted on the microscope as shown below.

Example: two objectives are mounted on a 5-location turret



Do not turn the turret during the laser exposure! and never remove an objective or a cap!

Three caps are mounted in the unused objectives on the microscope turret. These block the laser from exiting from these positions.

Mounted objectives

06/26/2013

S4 Laser safety key and emission indicator

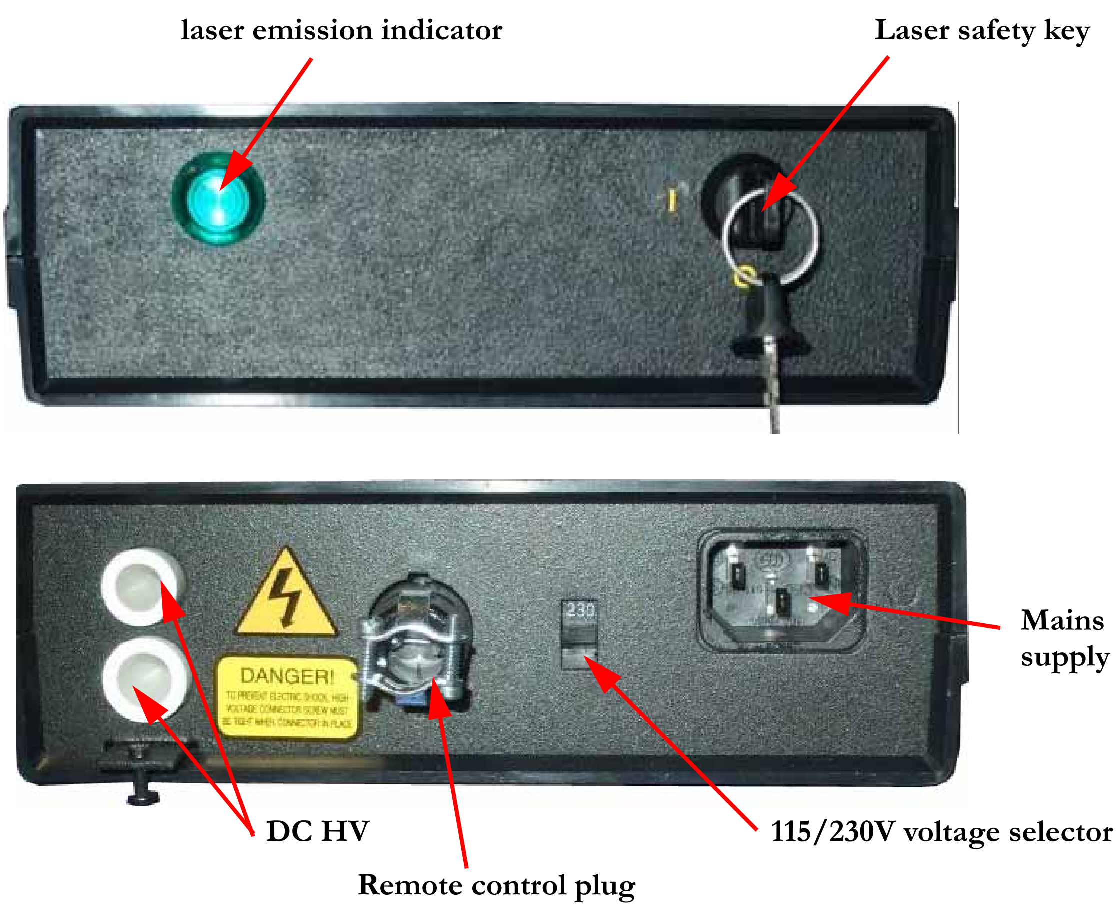
Each laser system has its own safety devices composed of a laser safety key with an emission indicator. A laser can only be switched ON with the laser safety key located on the laser source. When the key is turned on, a laser emission indicator shows that the laser source is ON. The key prevents inadvertent or unauthorized starting of the laser. Each external laser source has its own safety laser key. Follow the instructions describes

S5 Laser safety key for internal laser

Figure below shows the power supply box for internal laser HeNe (optional).

The safety laser key of the internal laser (HeNe/633 nm) is located on the external Power Supply Unit. This key belongs to the laboratory responsible person or to the authorized person using the LabRAM HR Evolution is strictly personal. Remove the control key when the laser or system is not in use.

External Power Supply for internal laser (option)

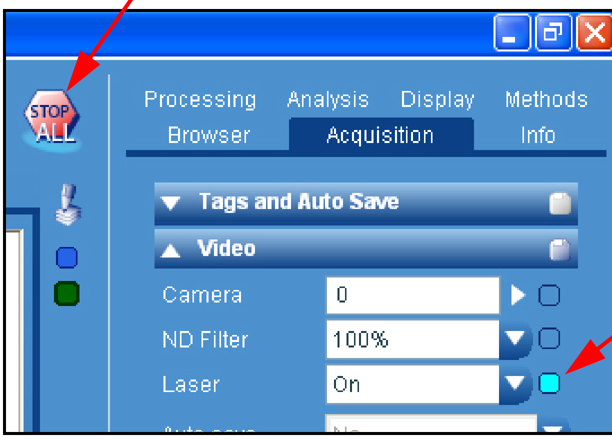


- «LASER OFF» or «0»: In this position, the LabRAM HR Evolution is unusable,
- «LASER ON» or «I»: In this position, the system is usable and all personnel in the vicinity of the laser must wear protective eyewear adapted to the laser wavelength(s) and power(s).

S6 Laser emergency stop

The laser beam is emitted only if an acquisition (Real Time Display or Acquisition measurement) has been launched. During this period, the laser emission can be stopped from the labSpec6 software by pressing the  button. The laser emission can also be deactivated by unticking the box located near to the laser parameter from the **Video** section in the **Acquisition** control panel.

Laser emergency stop button



de/activation of the laser

S7 List of safety labels

The following safety labels are affixed to the system and/or to the optional parts according to the different options installed. Not all the labels will be present where a class 1 safety enclosure has been included.

Labels 1 to 5 indicate a hazardous situation where there is a risk of serious injury due to possible laser radiation.

Label 1

The LabRAM HR Evolution System operates with laser source(s) emitting visible and/or invisible continuous laser radiation typically below 500 mW. The class of the laser product is 3B or 1 (with optional protection housing).

The laser class depends on the end user laser source and must be placed on the laser source. This label will also be placed near the «identification label» (see Figure 1-7, “Labels location for LabRAM HR Evolution with laser bench”, page 18).

λ = 325-1064nm P ≤ 500mW
VISIBLE AND/OR INVISIBLE LASER RADIATION
AVOID EXPOSURE TO BEAM
CLASS 3B LASER PRODUCT

For CLASS 3B System

DANGER! AVOID EXPOSURE OF THE LASER BEAM TO THE EYES

CLASS 1 LASER PRODUCT

For CLASS 1 System

CLASS 3B LASER PRODUCT
AVOID EXPOSURE TO THE BEAM
IEC60825-1 (08/2001) – 21 CFR 1040.10
30 mW Maximum at 632.8 nm

This label is stick on the internal laser source

Figure 1-1 Safety label 1

Label 2

Figure 1-2 Safety label 2

DANGER! DURING OPERATION, THE LASER BEAM IS EMITTED IN THE LABRAM HR EVOLUTION SYSTEM

Location: On the main cover of the spectrometer and the microscope

Label 3

LASER APERTURE

Figure 1-3 Safety label 3

A laser beam is emitted through that aperture.

DANGER! AVOID EXPOSURE TO THE BEAM.

Label 4

The LabRAM HR Evolution System has panels which are fixed and secured by screws. These panels must not be opened or removed, except when applying specific instructions described in the manual.

06/26/2013

CAUTION - CLASS 3B VISIBLE AND/OR INVISIBLE LASER RADIATION WHEN OPEN

AVOID EXPOSURE TO THE BEAM

Figure 1-4 Safety label 4

DANGER! the laser product class may increase if covers or casings are removed. Dangerous visible and/or invisible laser beams may become accessible and pose a possible risk of exposure.

Label 5

CAUTION

CLASS 3B VISIBLE AND/OR INVISIBLE LASER RADIATION WHEN OPEN AND INTERLOCKS DEFEATED

AVOID EXPOSURE TO THE BEAM

Figure 1-5 Safety label 5

DANGER! This label is located on the cover of the Class1 LabRAM HR Evolution. The laser product class may increase if covers or casings are removed. Dangerous visible and/or invisible laser beams may become accessible and pose a possible risk of exposure.

This label is also affixed on the safety laser unlock device delivered with the system and only for Service use.

Label 6

CAUTION - Class 3B VISIBLE AND/OR INVISIBLE LASER RADIATION

SWITCH OFF THE LASER SOURCE BEFORE CHANGING THE OBJECTIVE

Figure 1-6 Safety label 5

DANGER ! Microscope objectives are designed to be removed or switched over during normal operation and are not protected by any safety interlocks. SWITCH off the laser before changing or replacing microscope objectives.

NOTICE: if one (or several) objectives are not mounted on the microscope turret, the empty positions must be blocked with a suitable cap. Replacement or additional caps can be purchased from HORIBA Scientific if required.

Label 7

This symbol indicates that the device on which it is located can move and hence caution should be taken to avoid any potential injury.

Always remove your fingers from the table when motors are in operation.

Location: on the motorized XY Stage

06/26/2013

Label 8

Identifies any terminal which is intended for connection to an external conductor for protection from electric shock in case of a fault, or the terminal of a protective earth (ground) electrode.

Located inside the spectrometer and the main power supply.

Label 9

Some devices like CCD cameras need liquid nitrogen for cooling

You must be careful. Dangers include:

* **Nitrogen can spatter (possibly in eyes) while being poured.**
* **Wear goggles whenever pouring or dumping nitrogen. Nitrogen can spatter into the eyes, and potentially blinding pieces of frozen things can fly around when we drop it.**
* **Use a glove and/or tongs to handle any object going into or out of nitrogen and to carry the nitrogen dewar.**

Label 10

Recycling

This symbol located on a device shows that this DEVICE HAS BEEN PLANNED FOR ECOLOGICAL DESTRUCTION AT THE END OF ITS LIFE.

S8 Labels location

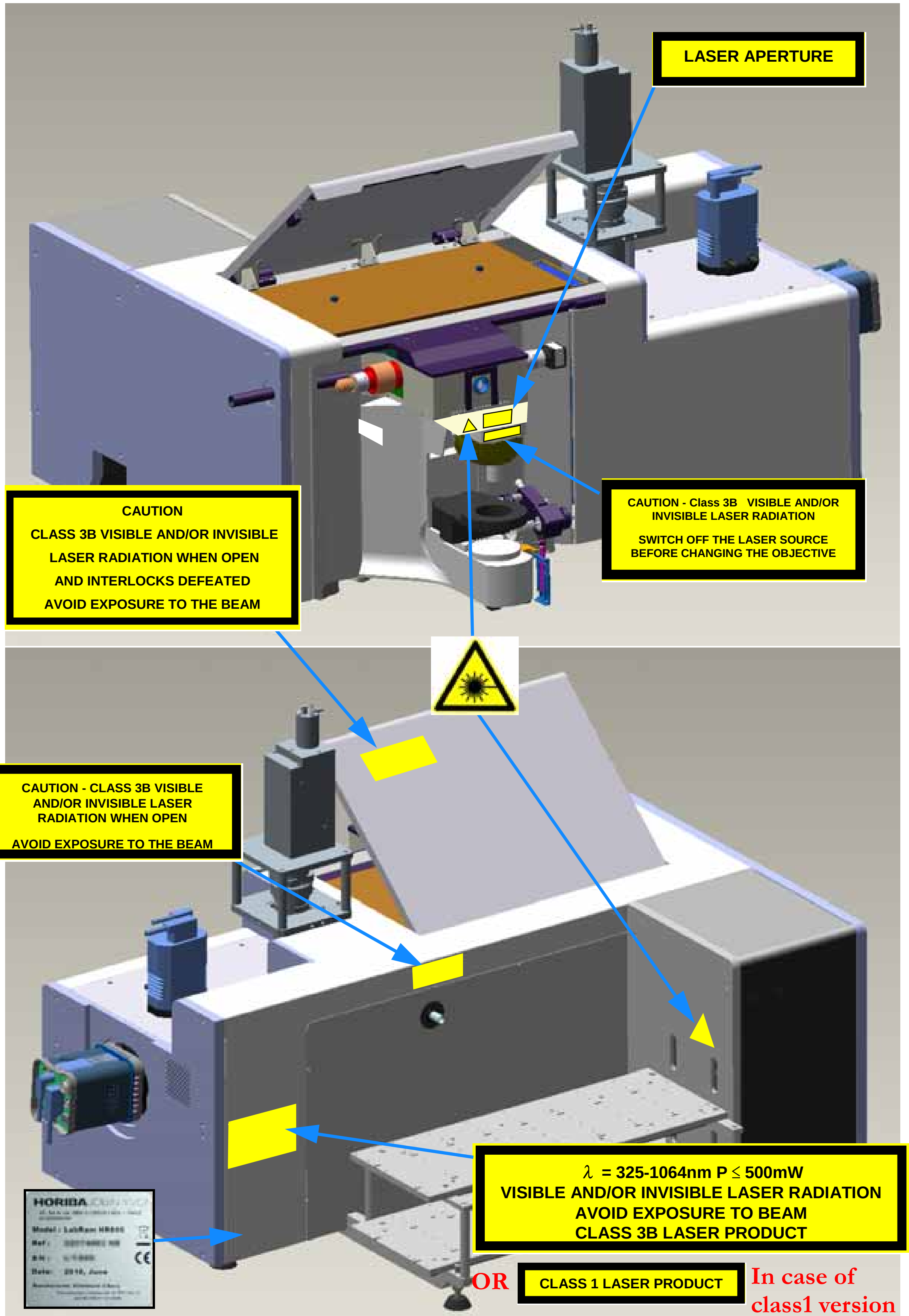


Figure 1-7 Labels location for LabRAM HR Evolution with laser bench

06/26/2013

Figure 1-8 Labels location for Class 1 (laser product) LabRAM HR Evolution with optional enclosure accessory

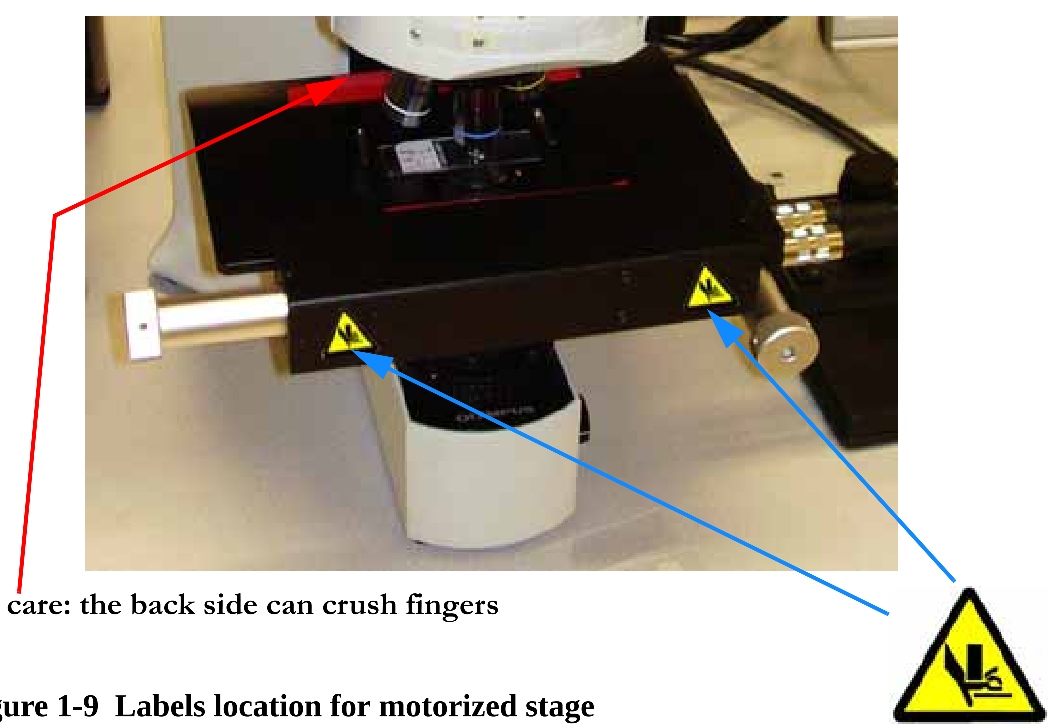


Figure 1-9 Labels location for motorized stage

06/26/2013

CHAPTER 1

LabRAM HR Evolution

S1 Summary

This User Manual contains the facilities design and equipment installation requirements for the LabRAM HR Evolution System.

S2 Purpose

The purpose of the document is to enable the facilities manager at the customer site, and HORIBA Scientific representatives and service engineers, to prepare for the installation of the LabRAM HR Evolution System and to determine the necessary requirements for the environment in which the LabRAM HR Evolution System is to be installed. It also provides guidelines and directions for the correct use and operation of the LabRAM HR Evolution system.

S3 Intended Readers

This document is intended for customers, HORIBA Scientific representatives, sales and service engineers.

1.1 System Installation

1.1.1 Packaging

The LabRAM HR Evolution System is delivered in packaging designed for protection during shipment. However, if the packaging is damaged, or it appears after unpacking that the instrument presents external or functional defects, please fill out the necessary forms with the shipper for declaration to the manufacturer (the guarantee is then limited to 3 days).

Moreover, there is a “tilt watch” indicator apposed onto the shipment boxes to ensure that the instrument has been kept upright during transit. If mishandling is revealed, you should leave the instrument in its original container and packaging and contact the manufacturer.

Before signing receipt of delivery, the packaging should be inspected and if the «tilt watch» indicator has been activated or damage is observed then clearly record this upon the delivery receipt. Your local HORIBA Scientific Service office should then be contacted.

It is recommended that the original packaging materials be saved for possible storage or transport of the instrument. Should it be necessary to send the instrument back to the factory, contact HORIBA Scientific or your local representative for prior approval. Pack the instrument in its original packaging if undamaged. Otherwise contact your local representative for new packaging material. Once the «return» slip has been received, send the instrument to the address that has been specified by your local representative.

1.1.2 Installation

The LabRAM HR Evolution System and associated software must be installed and started by HORIBA Scientific or an approved representative.

1.1.3 Operating Defects

After you have carefully read this manual, you suspect that the system has an operating defect, please contact HORIBA Scientific or your local representative for prior return approval. If the instrument is returned to the factory, the fault should be explained as clearly as possible.

1.1.4 Composition of the equipment

The LabRAM HR Evolution System is composed of the following:

- Basic System,
- Optional Sub-units,
- Accessories.

1.1.5 Requirements

Usually a «requirements list» is sent with the quotation. This chapter details these requirements.

The amount of floor space required for your LabRAM HR Evolution System can be quickly and accurately determined by using the **Figure 1-1** and the chapter **“Overall Dimensions” on page 37**.

Keep the following points in mind when selecting the system location and performing the final site layout:

- Position the LabRAM HR Evolution on a suitable solid table.
- Provide at least 0.6 meter (24 in.) of clearance at the front and rear of the system cabinet/table to allow space for servicing. HORIBA Scientific recommends 1 meter (39 in) of space for servicing.
- Make sure there is adequate workspace around the system so that operating personnel can move freely, without affecting system operation.

Figure below shows a layout example of LabRAM HR Evolution laboratory.

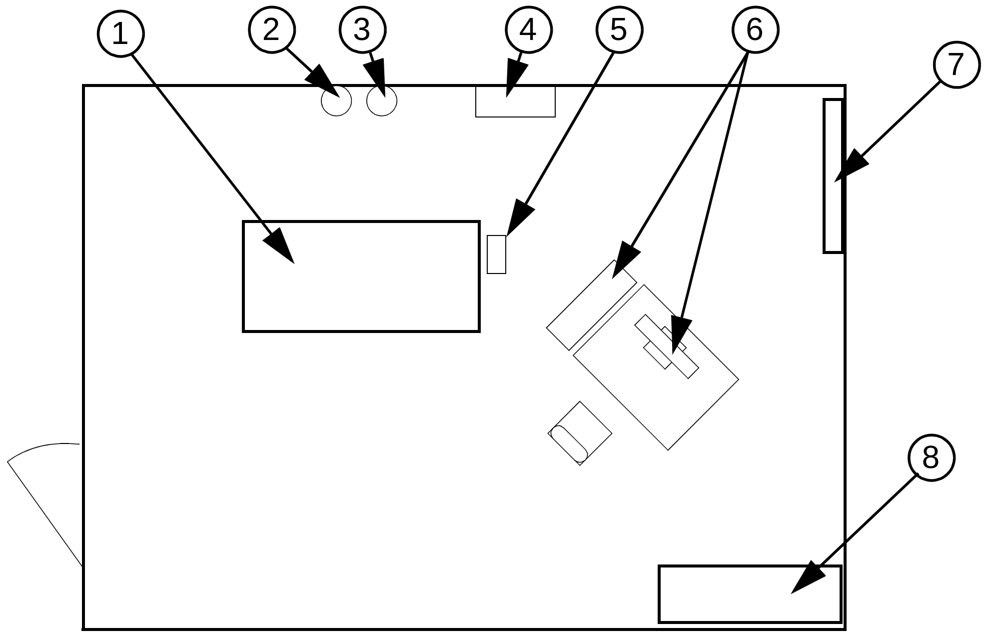


Figure 1-1 Laboratory layout example

1 LabRAM HR Evolution unit

2 Water supply (for Monochannel/Multichannel Detectors, laser sources..)

3 Liquid Nitrogen supply (CCD with Dewar)

4 Line power distribution box + ground cable

5 Power supply box

6 Computer and monitor

7 Window air conditioner

8 Storage (for software + accessories)

1.1.5.1 Air conditioning

To obtain the best level of performance of the LabRAM HR Evolution system, it is recommended for the laboratory temperature to be stabilized as much as possible. Air conditioning will provide the highest level of stability when maintained within the range 20°C to 25°c with variations of ± 1 °C maximum around the nominal value.

If you intend to install air-conditioning, please make sure that it does not cause sudden variations in temperature due to blowing fans.

The humidity level of the room must be lower than 80% till 31°C and linearly decline till 50% at 40°C.

Recommendations:

- It is recommended that the system is installed in a dark room. When the laser safety enclosure is not fitted, room lights can produce artefacts in the Raman spectra for long integration time.
- Make sure that the instrument is not directly exposed to strong airflows, sunlight or any source of heat which produce significant temperature gradients.
- We advise that air-conditioning vents should be away from the instrument so that they gently diffuse the conditioned airflow, rather than directly exposing the instrument to the cooled conditioned air.
- The size of the air-conditioner must be adequate with respect to the volume of the room and the heat output of the instrument and its laser sources.

1.1.5.2 Vibrations

Be sure that the chosen lab room is enough isolated from the building vibrations or external vibration sources (road, flights). Verify also that the ventilation fans or air conditioning devices do not induce vibrations.

1.1.5.3 Liquid Nitrogen (LN_2) Supply

The Liquid Nitrogen is required for CCD detectors which includes a Dewar recipient mounted on the CCD chip. Liquid Nitrogen is filled inside the Dewar and thus the CCD detector can be cooled down to -196 °C (-320 °F).

Liquid nitrogen can cause cryogenic burns if the substance itself, or surfaces which are or have been in contact with the substance (e.g. metal transfer hoses), come into contact with the skin. Local pain may be felt as the skin cools, though intense pain can occur when cold burns thaw and, if the area affected is large enough, the person may go into shock.

You must be careful. Dangers include:

* Nitrogen can spatter (possibly in eyes) while being poured.

* Wear goggles whenever pouring or dumping nitrogen. Nitrogen can spatter into the eyes, and potentially blinding pieces of frozen things can fly around when we drop it.

* Use a glove and/or tongs to handle any object going into or out of nitrogen and to carry the nitrogen dewar.

1.2 Presentation

There are three basic configurations of LabRAM HR Evolution Systems, on which specific options and accessories can be added on request. This chapter describes these versions.

1.2.1 LabRAM HR Evolution Visible (440-1100 nm)

The LabRAM HR Evolution VISIBLE is a fully integrated confocal Raman microscope instrument. It is automated and compact. The single stage spectrometer is an 800 mm focal length Czerny-Turner type spectrograph equipped with mirrors (reflective based on optics). Optical paths commutations are motorized and controlled by computer (lasers, detectors exits, etc...). The VISIBLE version covers the 440-1100 nm wavelength range.

Integrated laser (option) Raman instrument for confocal and punctual analysis including a confocal microscope, transfer and filtering optics, an achromatic spectrograph equipped with two gratings, a multichannel detector and a computerization package.

Many options and accessories are available to permit micro and macro measurements, mapping, temperature measurements or remote analysis. It can be coupled to AFM instruments.

Various laser sources can be mounted on the Instrument. Confocal and punctual analysis including a microscope, transfer and filtering optics, an achromatic spectrograph equipped with two gratings, a multichannel detector and a computerization package are included in the system. However, no laser source is included in the basic configuration. A HeNe/633nm/17W laser source can be included inside the LabRAM HR Evolution housing. Laser list is detailed in the chapter "Lasers Options" on page 60.

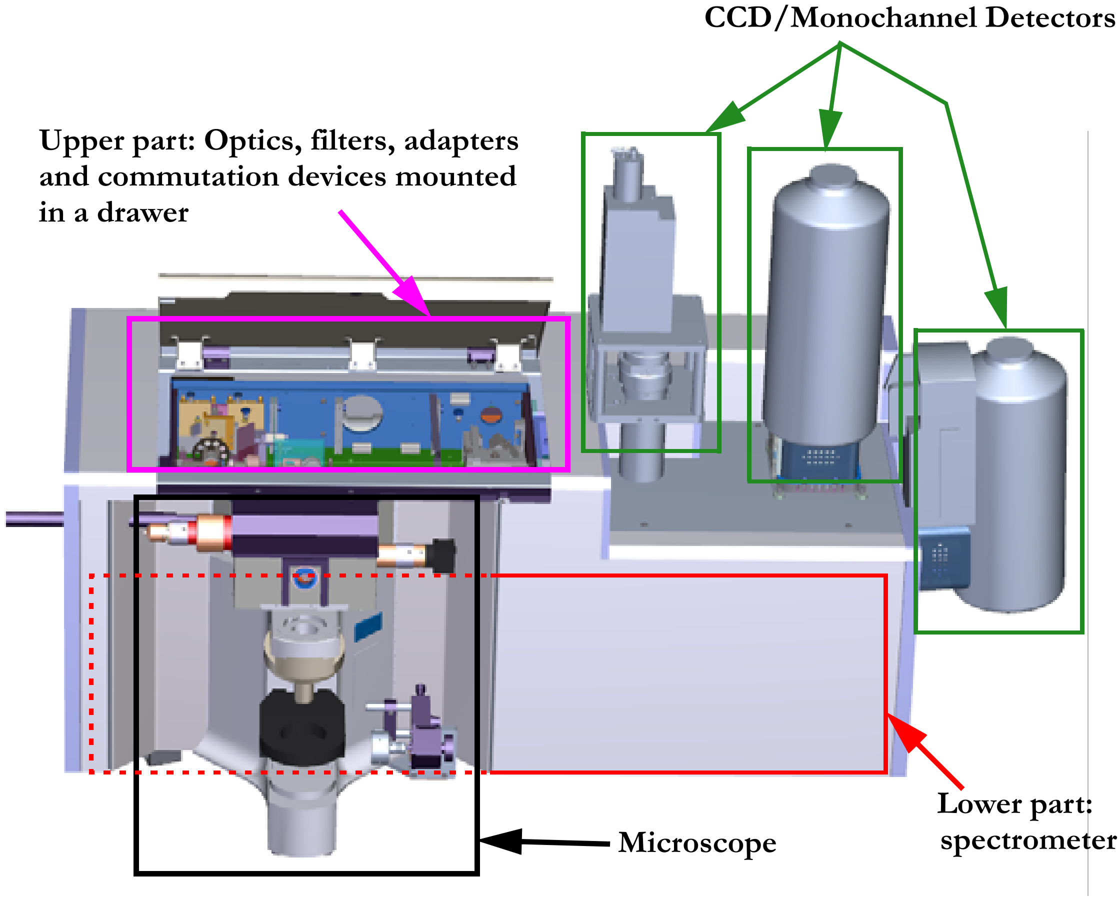


Figure 1-2 LabRAM HR Evolution: Internal and External Main Optical Parts

1.2.1.1 Entrance optics assembly

Includes:

- A motorized commutation between internal and external lasers. Up to 4 laser sources can be mounted on the optional fully automated lasers bench,
- A filter wheel with 9-position neutral density filters (100%, 50%, 25%, 10%, 5%, 3% ,1%, 0,1%, 0,01%) controlled by software, for decreasing laser power on the sample,
- An interference filter holder,
- A spatial filter to purify the laser beam (for visible system only),
- An adjustable kinematic rejection filter (Notch or Edge) mount allowing fine tuning of the filter operation angle for low frequency cut-off adjustment and easy and quick exchange of excitation wavelength. Automation in option.
- A set of one rejection filter and one interferential filter for each wavelength is necessary.

1.2.1.2 High stability BX41 confocal microscope

Supplied with:

- An XY manual mechanical stage,
- A white light illumination system for Koehler illumination by reflection with variable light intensity,
- An internal white light illuminator by transmission supplied with an Abbe condenser,
- A revolving turret equipped with the following «standard» plan achromatic objectives: 10X, 50X and 100X,
- A high definition USB color camera for simultaneously visualizing the sample under white light illumination and the laser spot, using the LabSpec software,
- Automated switching between visualization mode and Raman measurements mode (controlled by software.

1.2.1.3 Confocal coupling optics between the microscope and the spectrograph

Includes:

- A continuously adjustable confocal pinhole from several µm to 1200 µm driven by software to define with accuracy the size of the analyzed volume.
- Coupling optics to focus the Raman beam on the entrance slit of the spectrograph (lens system) equipped with a shutter.
- free locations to insert polarization components.

1.2.1.4 A 800 mm focal length achromatic flat field monochromator

Includes:

- Two 76x76 mm gratings, 1800 gr/mm and 600 gr/mm (other gratings: see Appendix 1), mounted on a motorized turret driven by software, to vary both spectral resolution and spectral coverage in one shot. Both gratings can be quickly and easily interchanged without realignment.
- A scanning mechanism using a high precision sine bar.
- A motorized entrance slit equipped with a shutter. All motorized elements are controlled by software.
- A laser diode device allowing the control of the optical alignment from detector to sample

1.2.1.5 Detector

A multichannel air cooled (-70°C) CCD detector:

- 1024 x 256 pixels – Open Electrode front illuminated chip,
- MPP selected,
- Spectral Range: 200 – 1050 nm,

- Pixel size : 26 x 26 microns,
- Chip size : 26.6 x 6.7 mm,
- Quantum efficiency (QE) > 30 % between 500 and 800nm,
- Typical read out noise: 4 ē rms,
- Dark noise < 0.002 ē/pixel/sec,
- Other detectors. See Appendix 1 chapter.

1.2.1.6 Computer and software

- LabSpec6 Software used under Windows OS, supplied with two computer licences, permitting the control of the instrument, data acquisition, data manipulation, processing and storage options. This software also includes macro programming capabilities using visual basic scripts and a multivariate analysis.
- A computer with the latest configuration, supplied with a flat screen (printer not included)

1.2.1.7 Main Technical Specifications

- Spectral Range: 440 nm – 1100 nm,
- Spectral dispersion: 0.35 cm-1/pixel at 633 nm with 1800 gr/mm grating,
- Spectral resolution: 0.35 cm-1 (typical Full Width at Half Maximum) at 837nm with 1800gr/mm grating,
- Spatial Resolution: Truly confocal microscope, offering submicron lateral resolution and axial confocal performance better than 2 microns.

1.2.1.8 Raman performance validation

- A quality certification
- Tools kit including sample references, Neon lamp, laser interlock
- User and Q/C manuals including performance tests made for each instrument

1.2.2 LabRAM HR Evolution VIS-NIR (400-2200 nm)

The LabRAM HR Evolution VIS-NIR (**N**ear **I**nfra **R**ed) has the same characteristics than the Basic LabRAM HR Evolution VIS with the exception of the following elements:

- Includes high throughput achromatic coupling optics optimized to work from Visible to NIR with a pre-aligned 2 positions confocal coupling module with silver coated mirrors for NIR and lenses for visible Raman measurements. This device is fully automated.

The LabRAM HR Evolution VIS (Visible) can not be upgraded to LabRAM HR Evolution VIS-NIR.

1.2.3 LabRAM HR Evolution UV-VIS-NIR (220-2200 nm)

The LabRAM HR Evolution UV-VIS-NIR (**U**ltra **V**iolet-**VIS**ible-**N**ear **I**nfra **R**ed) has the same characteristics than the Basic LabRAM HR Evolution VIS with the exception of the following elements:

- Includes high throughput achromatic coupling optics optimized to work from UV to NIR with a pre-aligned 2 positions confocal coupling module with aluminium coated mirrors for UV and lenses for visible Raman measurements. This device is fully automated.

1.3 Description

The LabRAM HR Evolution is an integrated Raman system. The microscope is coupled confocally to a 800mm focal length spectrograph equipped with two switchable gratings.

Figure 1-1 and figure 1-3 shows the main optical parts of a standard LabRAM HR Evolution when the cover is removed.

1.3.1 Optical path description

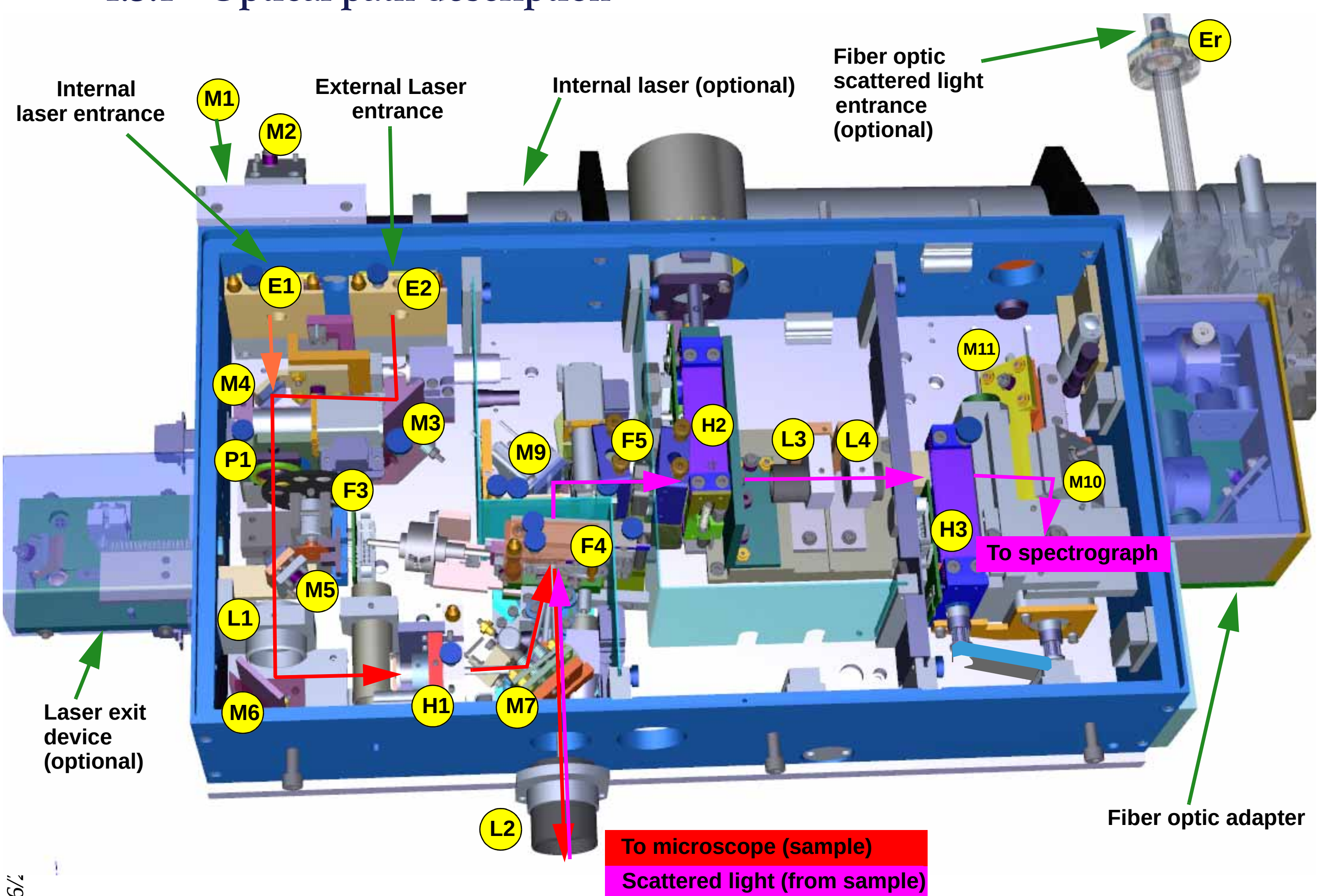


Figure 1-3 LabRAM HR Evolution Optical path - VISIBLE

The internal HeNe 17 mW laser (optional) is mounted on the back of the instrument: it supplies a vertically polarized 633 nm excitation wavelength. Up to 2 laser entrances can be used: E1 for internal laser and E2 for external laser(s). Using an external bench with commutation devices, it becomes possible to use many other lasers. If an external laser is used, the beam is introduced to the main path via the E2 input and the removable mirrors M3 and M4.

The internal laser beam is directed by the two mirrors M1 and M2 (M1 is located below the M2) in order to go through the polarizer P1 and the laser intensity absorption filter F3. This 6-filter wheel, driven by the software, is dedicated to absorb laser intensity: there are 9 different positions ND neutral filters with the optical densities 100%, 50%, 25%, 10%, 5%, 3%, 1%, 0.1% and 0.01%. Then, the L1 lens focuses the laser beam on the pinhole H1. The resulting laser image is focused by the lens L3 via the mirror M7 on the F4 notch filter with an appropriate angle in order to be completely reflected towards the sample. This filter is used to purify the plasma lines of the laser. An appropriate notch filter is necessary for every exciting line of a laser.

The pinhole H1 is conjugated with the spot on the sample and is used as a reference for alignment. It is replaced with a field lens when working in laser scanning image mode.

The lens L2 produces a parallel beam that is focused on the sample by the infinite-optics microscope objective.

The Raman collected by the microscope objective in back scattering configuration follows the same way back. The Raman beam passes through the notch filter. L2 reforms the image of the laser spot on the sample onto the confocal hole H2. An additional polarizer filter or a second filter (for higher suppression of the exciting line) can be inserted on the F5 position.

- For Visible version, the lenses L3 and L4 are placed on the path and image the confocal hole H2 onto the entrance slit H3 and adapt the aperture of the beam to the spectrometer. This LabRAM version uses silver coated mirrors.
- For Visible/NIR version, the lenses L3 and L4 are placed on the path when the Visible range is used. The 2-position motorized module switches automatically to the NIR range when required. In this case three mirrors M12, M13 and M14 are mounted and pre-aligned for optimal NIR transmission (see figure 1-3). This LabRAM version uses silver coated mirrors.
- For Visible/NIR/UV version, a first set of L3 and L4 lenses are mounted on a pre-aligned 2-position motorized module for a Visible/NIR use. The second optical set is composed of three Al (Aluminium) coated mirror for UV transmission (see figure 1-3).

The shutter is placed just behind the entrance slit H3. The mirror M10 sends the Raman scattering to the spectrograph.

Raman scattering can also be entered via a fiber optic probe (fiber optic coming from the «Super Head» remote device). The fiber optic entrance Er is also located on the back side of the instrument. An optical coupling composed of a mirror M11, sends the scattered signal to the spectrograph entrance. In this case, the M10 mirror must be turned down via a motorized commutation. Using the «Super Head», the laser is coupled to another fiber optic going to the probe using the 10x objective of the microscope.

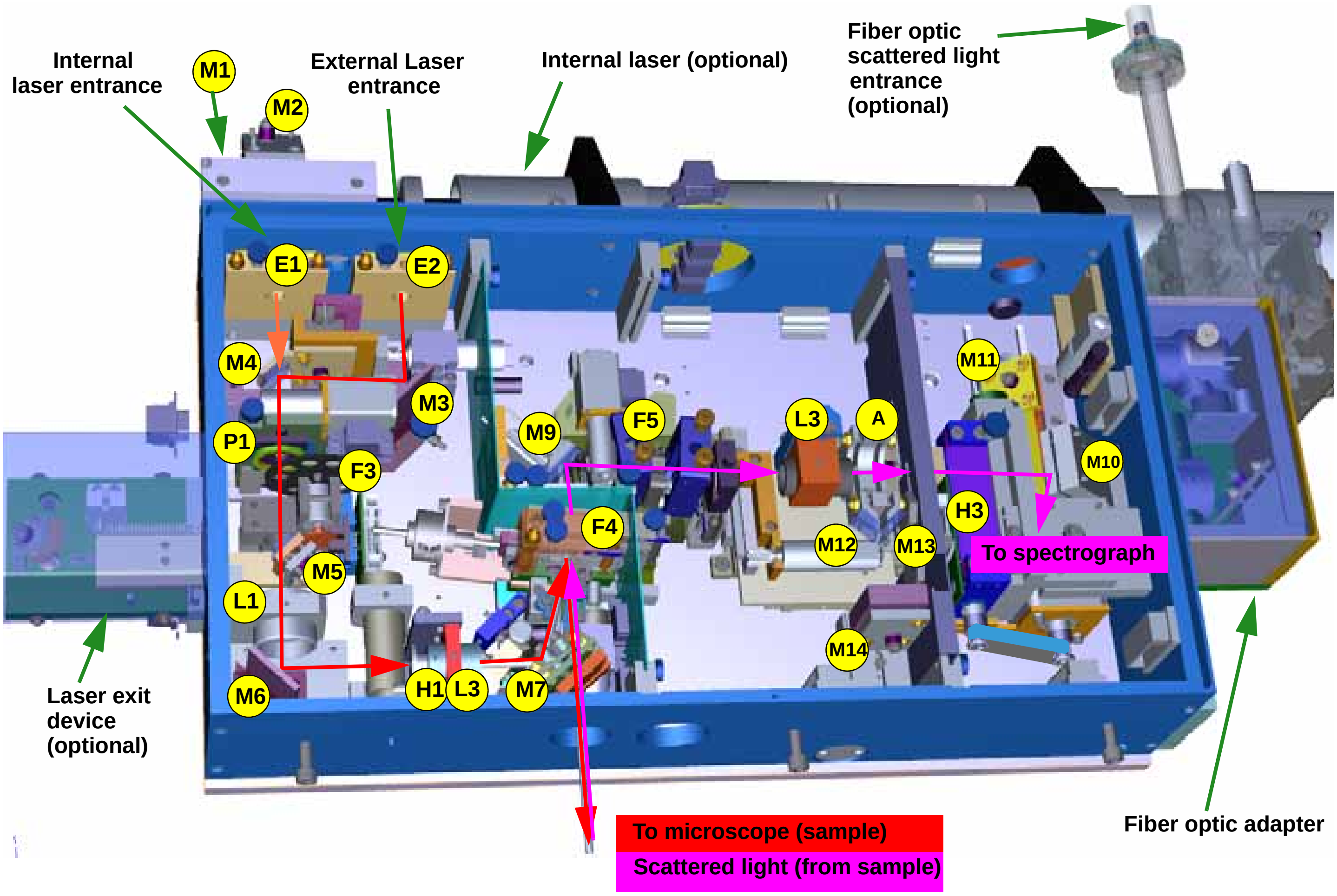


Figure 1-4 LabRAM HR Evolution Optical path - VISIBLE/UV and VISIBLE/NIR

1.3.2 Spectrograph optical path

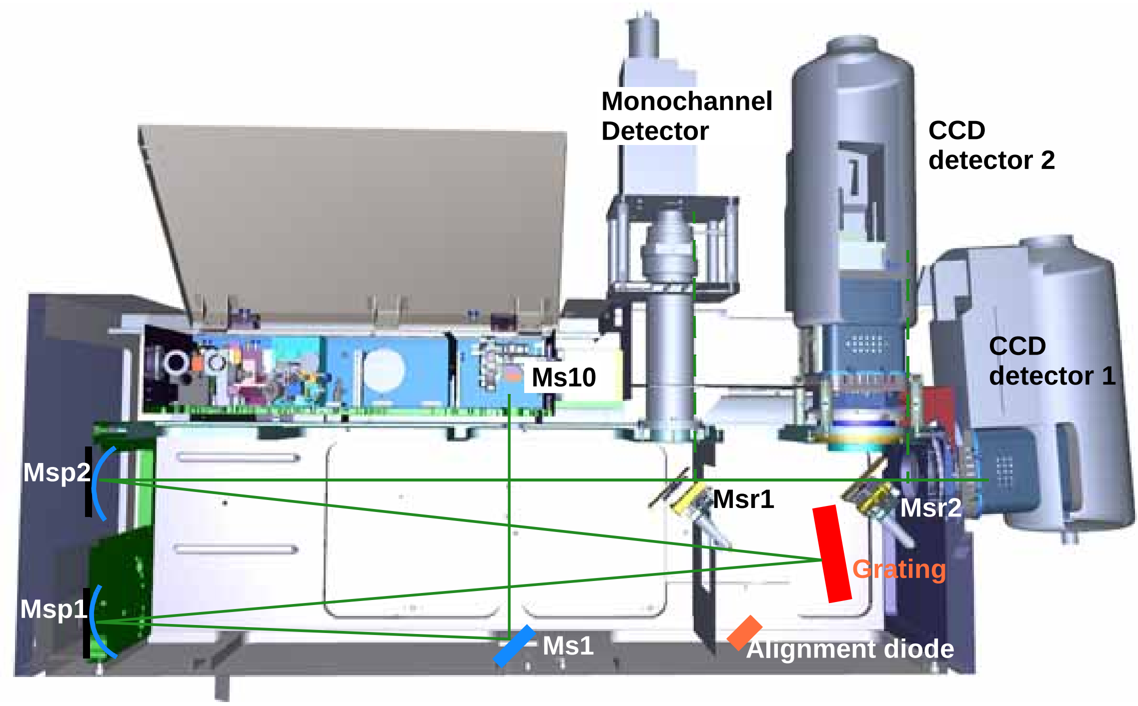


Figure 1-5 Spectrograph optic path

The LabRAM HR Evolution spectrograph is a symmetric Czerny Turner which is optimized for flat field and for minimum optical aberrations.

The diverging beam coming from the slits is reflected by the mirror M10 and then by Ms1 and becomes parallel after the spherical mirror Msp1 (800 mm focal length). The mirror Msp1 reflects the parallel beam onto the grating. The diffracted first order is collected by the spherical mirror Msp2 (800 mm focal length) and focused onto the CCD detector 1, or CCD Detector 2 or Monochannel Detector via the retractable mirrors Msr1 and Msr2.

Gratings are mounted back to back on kinematic interchangeable holders. Many different gratings are available: see Appendix 1 chapter, "Other Gratings" on page 62.

1.3.2.1 Internal alignment Laser diode

A low power diode is integrated to the LabRAM HR Evolution spectrograph. The emitted beam travels along a reverse path through the entrance slit and then towards the entrance optics to the microscope.

This laser diode beam is used for alignment of the whole coupling optics from the spectrograph to the sample. This device is for Service Team only.

1.3.3 Instrument Control

The LabRAM HR Evolution instrument is controlled from the LabSpec6 Software and the main actions are motorized.

1.3.3.1 Integrated controller

The hardware electronic controller is fully integrated inside the Instrument. The power supply of the system is provided from an external power supply box.

1.3.4 White light illumination

A white light source is necessary to illuminate the sample when a microscope is used. This illumination can be done «by transmission» or «by reflection», depending on your sample.

1.3.5 Options

They are listed in the chapter “Appendix 1: Options and Accessories” on page 48). Imaging options are detailed in the chapter “Appendix 2: Imaging Options” on page 48.

1.4 Interconnection

1.4.1 Interconnection (without options)

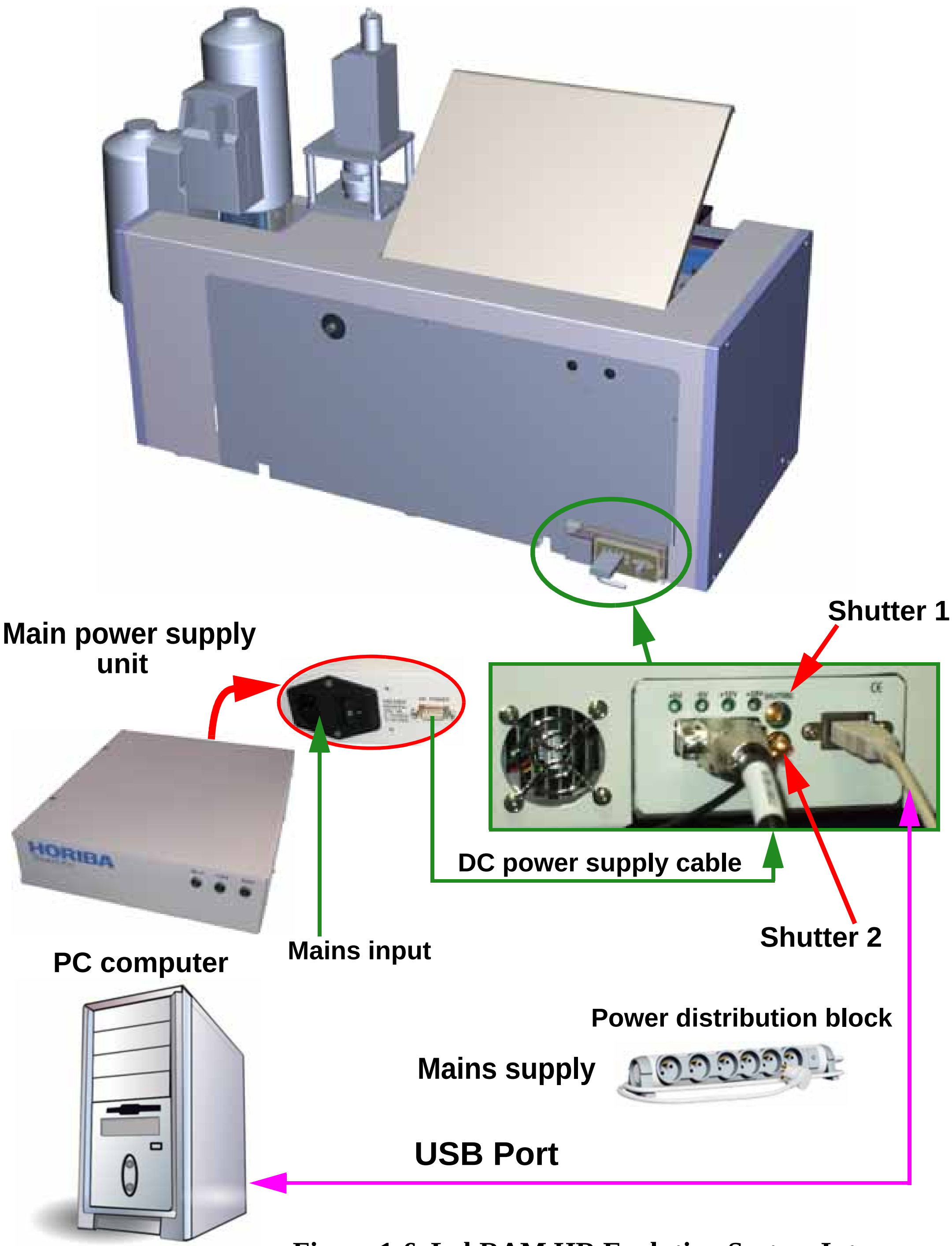


Figure 1-6 LabRAM HR Evolution System Interconnection

06/26/2013

1.5 Specifications

1.5.1 Regulations

- **CE compliant:**
  EMC: 2004/108/EC, standard EN 61326-1 (2006), Class A product, basic requirements
  LVD: 2006/95/EC, standard EN 61010-1 (2010)
  IEC 60825-1 (2008)
  Other regulations: FDA: 21 CFR1040.10

1.5.2 Optical

1.5.2.1 Dispersion

The spectral dispersion of the LabRAM HR Evolution at 585 nm, with the 1800 gr/mm, is typically of 0.35 cm-1/pixel. The spectral resolution will then depend on the entrance slit aperture and CCD pixel size.

1.5.2.2 Axial resolution

The spatial resolution obtained along the Z axis at 514 nm and derived from the FWHM of the intensity profile of the Silicon line at 520,7 cm^{-1} is < or = to 2µm, measured on a LabRAM HR Evolution VIS, with a 100X objective (n.a. = 0.9) and a confocal hole set at 100µm.

1.5.2.3 Repeatability

Under normal conditions of temperature stability (± 1° C), the repeatability is <1cm^{-1} measured on a silicon sample at 633 nm with a 1800 gr/mm grating during one hour (about 50 measurements).

1.5.2.4 External Lasers

The acceptable characteristics for a laser coupled to the LabRAM HR Evolution are:

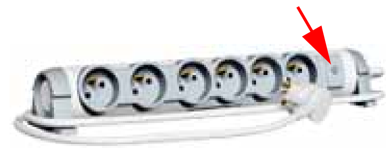
- Beam diameter <2 mm
- Wavelengths range 200-1100nm
- Beam divergence <1 mrad
- Maximum laser power acceptable 500 mW (visible range) for class 3B.

1.5.3 Electrical

The power supply for the LabRAM HR Evolution is provided by an external power supply box.

System connection/disconnection

The LabRAM HR Evolution System must be connected to the mains electrical power supply via the power distribution block (delivered). It is designed to be left powered up during usual daily usage. The LabRAM HR Evolution System includes several independent power supplies which must be all connected to this power distribution block.



Power distribution block part number: Legrand, 050083

Standard: NF C 61-314-2003, NF C 61-314-2008, VDE

1.5.3.1 Mains supply

The LabRAM HR Evolution System is a permanently connected instrument which includes several power supplies, connected to the power distribution block.

- Main power supply box

- Additional options (Autofocus, CCD Camera etc.)

A full electrical specification is detailed in the table below.

LabRAM HR Evolution	
Electrical detail	**Data**
Installation Category	II
Safety Class	1
Pollution degree	2
LabRAM HR Evolution Mains supply: Voltage variation Phase Frequency Frequency variation Power	100-240 VAC -10%, +6% Single 50/60 Hz ± 2% 150 VA
2 Fuses are located in the mains connector on the back panel of the main power supply box.	2 x TT1A 220V

06/26/2013

Internal Laser source (selectable from power supply back panel) see page 13. Voltage variation Phase Frequency Frequency variation Power	115/230 volts ± 10% Single 50/60 Hz ± 2% 50W
White lamp (reflection/transmission): must be chosen with the order according to the laboratory mains voltage.	120 V, 50/60 Hz, 150W 230 V, 50/60 Hz, 150W
CCD Camera Synapse Voltage variation Phase Frequency Frequency variation Current	100-240 Volts -10%, +6% Single 50/60 Hz ± 2% 1A max.
DuoScan Device Voltage variation Phase Frequency Frequency variation Current	100-240 Volts -10%, +6% Single 50/60 Hz ± 2% 100 VA
Desktop PC computer Voltage variation Phase Frequency Frequency variation Current	100-240 Volts -10%, +6% Single 50/60 Hz ± 2% 400 VA
Power with the following accessories: LabRAM HR Evolution White light illumination CCD camera Duoscan and Pifoc Motorized XY stage PC computer	1.1 kW

1.5.3.2 Grounding (Earthing)

The following grounding/earthing conditions must be satisfied.

- Use a good earthing conductor in accordance to class 1 equipment and the local safety regulations
- The ground of the spectrograph must always be connected to the LabRAM HR Evolution electronics

1.5.3.3Mains isolation

The Unit is connected to the mains supply via a standard plug. This plug can be removed for safe maintenance of the instrument.

A cut off switch must be accessible in case of emergency.

1.5.4 Overall Dimensions

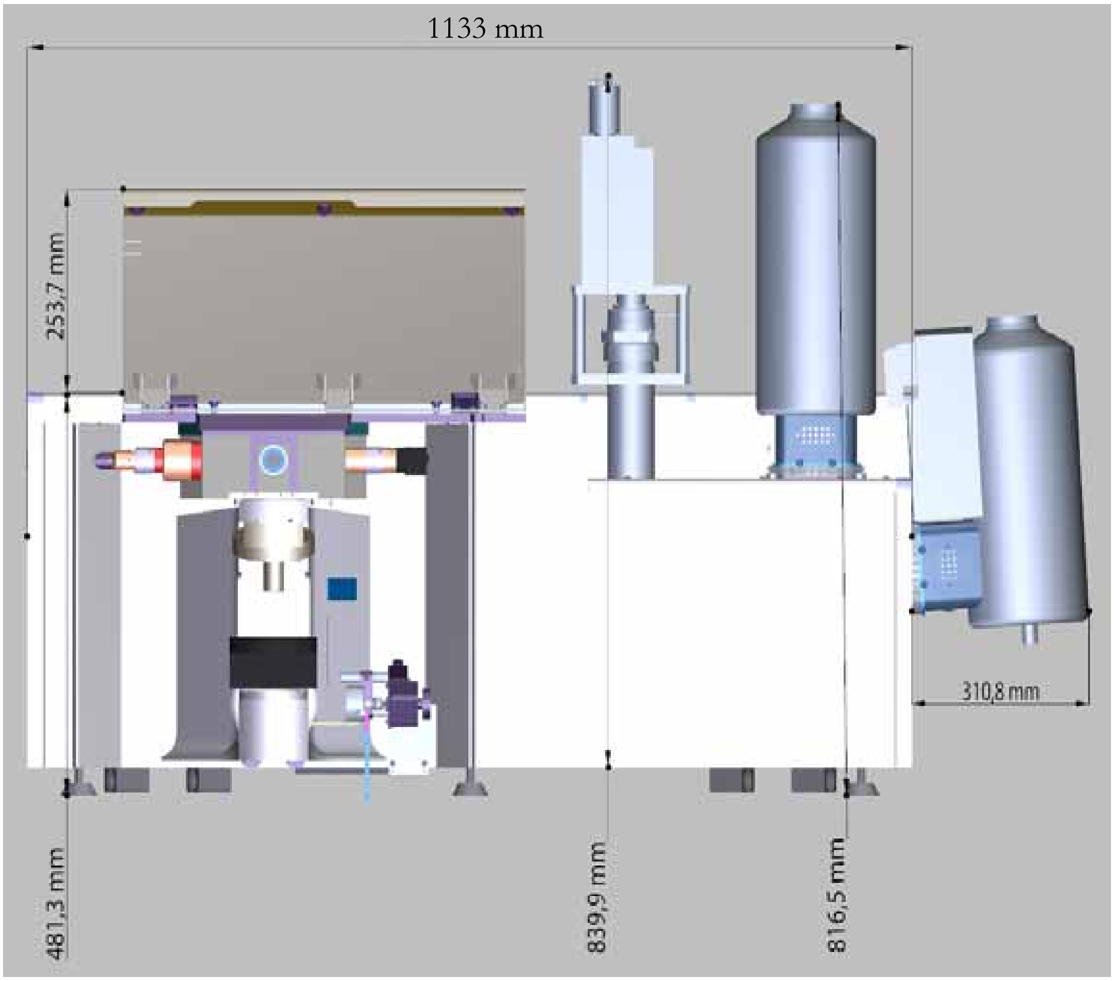
Figures below show various dimensions according to the options.

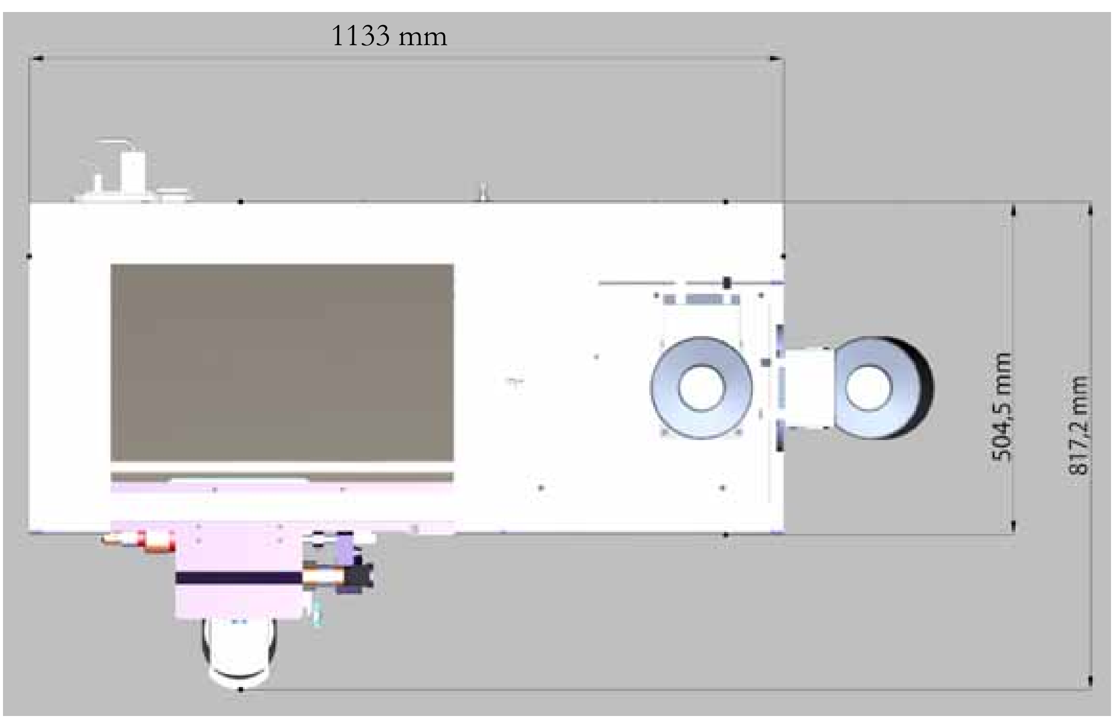
Overall dimensions of the LabRAM HR Evolution alone with microscope: 1133 (W) x 473 (H) x 817 (D)

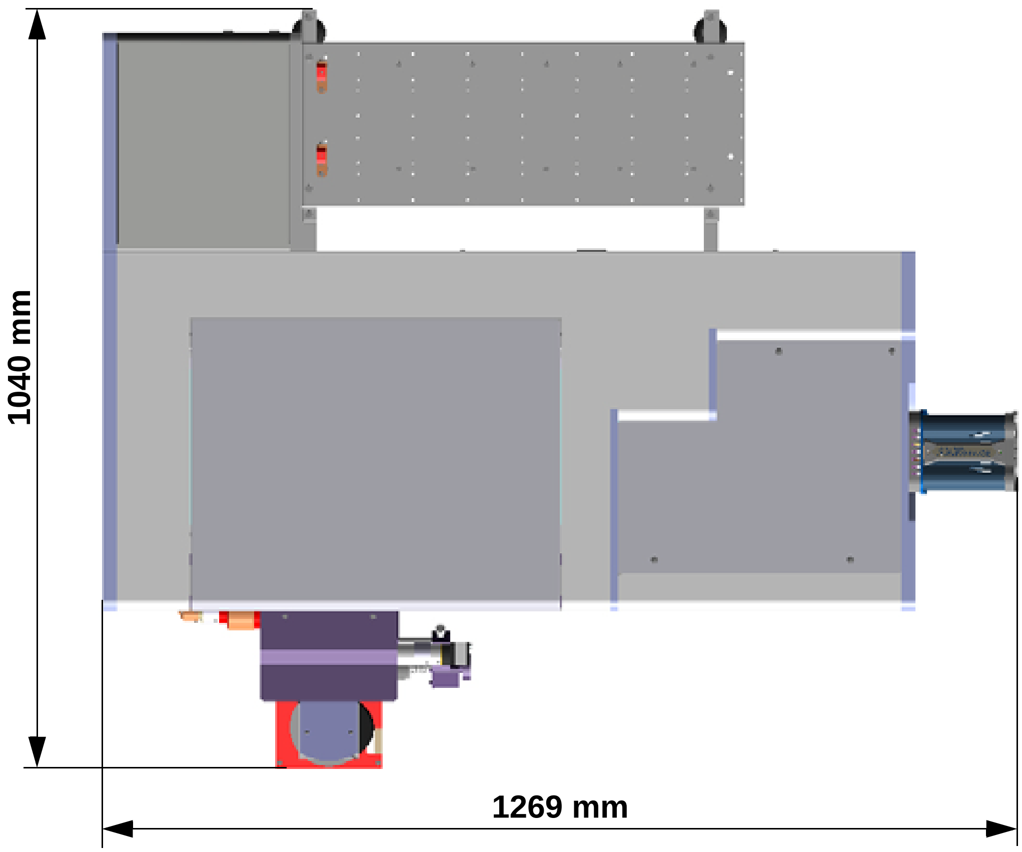
Overall dimensions with axial CCD (with Dewar) and opened upper door:
1277 (W) x 735 (H) x 817 (D)

Overall dimensions with axial CCD and laser bench:
1269 (W) x 473 (H) x 1040 (D)

Weight: 95 kg (without CCD)


1133 mm
253,7 mm
310,8 mm
481,3 mm
839,9 mm
816,5 mm


1133 mm
504,5 mm
817,2 mm



1.5.5 Environmental conditions

Environmental conditions	Conditions	Remarks
Indoor use		
Operating temperature (guaranteed results)	between +18°C and +25°C in a ± 1°C range	air conditioning with air regulation is required
Operating temperature Safe use (results not garanteed)	≤ +40°C > +10°C	No condensation
Relative Humidity	Up to 80% till 31°C and linear decline till 50% at 40°C	No condensation
Altitude	Max 2000 meters	

The room in which the instrument is installed must be properly ventilated.

1.5.6 Storage

If this unit has to be stored before it is installed, it must be kept in a store room where the storage conditions are listed in the following table:

Storage	Conditions	Remarks
Temperature	+5 to +40°C	
Humidity	Up to 80% till 31°C and linear decline till 50% at 40°C	No condensation
Packing requirements	Special packing	Must stay closed during storage to prevent moisture and dust entering the unit

1.5.7 Cleaning Instructions

The outside part of the unit should be cleaned once a year to mainly get rid of dust. Use a lint free cloth with 90° alcohol. Do not use any other chemical products.

No cleaning should be undertaken inside the optical parts of the instrument, and namely the optical drawer and spectrograph.

User is responsible for waste disposal after analysis.

CHAPTER 2

Getting Started

2.1 Preparation

After the LabRAM HR Evolution System has been installed and started up by HORIBA Scientific or an approved representative, the user should follow certain rules and recommendations, listed below.

2.1.1 Safety

Read carefully the Safety instructions described in the chapter "General safety Instructions" on page 6.

2.1.2 Hardware Foresights

The CCD detectors must not be continuously exposed to bright light, especially in the ultraviolet wavelengths. This exposure will increase dark current. This damage can occur independent of power applied to the device.

Most of the electronic components used in the detector system are highly electrostatic sensitive. Full ESD (ElectroStatic Discharge) handling procedures of the components of the system are essential. Failure to comply will damage the system.

Improper use or grounding of the LabRAM HR Evolution System could damage the system and/or present the user with a potentially lethal hazard.

Do not connect or disconnect the cables while the System is on. The resulting power surges may damage the Units.

- **Reliability**

Electronic and electric circuits are subject to the following constraints whenever the unit is turned on or off:

- a brief transient period subjecting certain components to a severe operating mode,
- a slow transient period (several hours) during which the electric and electronic circuits will progressively reach a stable operating mode, from both electronic and thermal viewpoints.

It is strongly advised to leave the unit turned on, with the exception of the laser(s).

- **Reproducibility**
  - Extreme temperature variations within a short period of time can influence measurement results.
  - For example, measurement reproducibility will be optimal if a temperature regulation of 1 °C is observed.
- **Powered on the system**

The first time the LabRAM HR Evolution System is connected to a power supply, or after a long shutdown period, the need for an electronic and thermal stabilization period should be taken into account in order to obtain good results.

For example, the following minimum periods should be taken into account:

- Mechanical and optical part of the LabRAM HR Evolution unit: approximately 24 hours in a climate-controlled, regulated environment,
- Electronic boards: approximately 30 min.

2.2 Using LabRAM HR Evolution

This chapter describes how to analyze a silicon sample with a basic LabRAM HR Evolution System. We will consider as «basic» the following system:

- **LabRAM HR Evolution instrument (VIS, VIS/NIR, VIS/UV/NIR),**
- **Internal laser and/or up to 4 external lasers,**
- **Manual XYZ sample stage,**
- **CCD Detector.**

2.2.1 Preliminary

The LabRAM HR Evolution System has been installed and implemented by HORIBA Scientific/or an authorized representative.

The system must be permanently powered on to insure optimal mechanical and electrical stabilization.

It is strongly advised to leave the unit turned on, with the exception of the light sources and laser(s).

2.2.2 Procedure

1	**1** Check to ensure that the LabRAM HR Evolution Unit and its sub-assemblies are correctly connected to the power, **2** Some sub-assemblies need additional attention for proper operation, do not forget to read the manuals delivered with these equipments. E.g. CCD with Dewar must be filled with liquid Nitrogen, some lasers are cooled with water circulation etc... **3** Power ON the computer and the screen. **4** Switch the laser source ON: please refer to the Manual delivered with the unit.
2	Run the LabSpec6 software.
3	**How to select a laser** From LabSpec6, select the **Acquisition** Panel, then choose the **Instrument setup** section  **Select the laser wavelength: the instrument will automatically commute to the selected line**

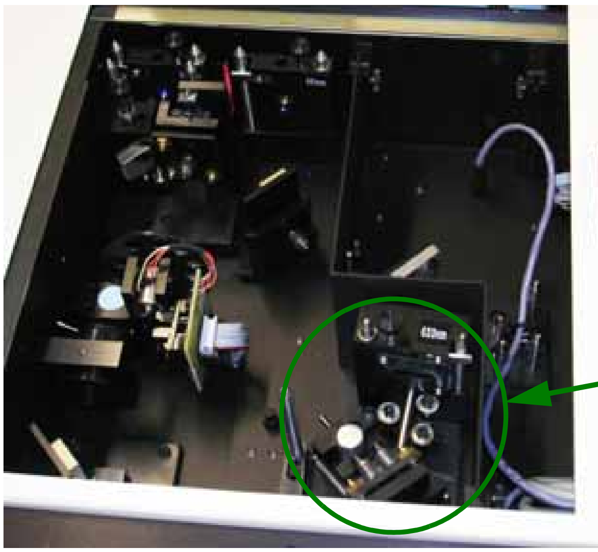
06/26/2013

4

Insert the appropriate rayleigh filter according to the laser wavelength

These filters edge or notch are clearly identified by the laser wavelength inscribed on the holder. See detailed procedure with films in the “Appendix 5 Instrument setup when lasers are selected” on page 63.

See additional information on “Appendix 6: Edge Filters” on page 66



rayleigh filter

Spacer

Example of Rayleigh filters mounted on holders

Figure 2-1 Rayleigh filter location

5

Insert the appropriate spacer

According to your Rayleigh filter, an angle fine tune is necessary obtain a good compromise between the scattered light band-pass and the laser wavelength rejection. This fine tune is obtain by inserting a spacer. Each LabRAM HR Evolution is delivered with a spacers set. Each spacer has a specific diameter which bias the Rayleigh filter angle.

To know the spacer to use, please refer to the LIRS page in the QC book of your system.

Spacer Diameter (mm)	4	5	6	7	8	9	10	11
Angle of the rayleigh filter (degrees)	9.84	8.69	7.58	6.59	5.69	4.86	4.05	3.3

06/26/2013

6

Open the spectrograph entrance Confocal Hole

The confocal hole modifies the spacial resolution on the sample and the spectral resolution of the spectrograph. For our Silicon sample, we don't need spacial resolution, thus fully open the confocal hole.

Initialize the motors position

Enter the wished value of the confocal hole aperture

Move the motor to the entered value

7

Sample analyzing

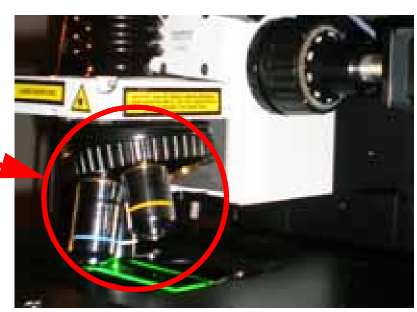
1 Place a Silicon sample on the stage.

2 Light ON the illumination light source

Light source exit to instrument

ON/OFF switch + light dimmer

3 Turn the objectives turret to the lower magnification objective.



06/26/2013

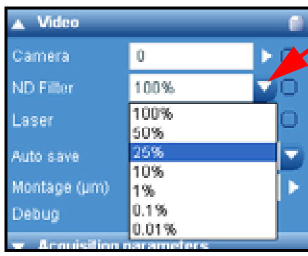
7

4 From the LabSpec6 software, select the camera icon  to launch the sample visualization window.

5 Select the required objective for the measurement and fine tune the focus if necessary. Start from a lower magnification and after a good focussing on the sample increase step by step to the required magnification and each time verify the focus.

6 Once done, click on the **Stop** icon  to stop the sample visualization.

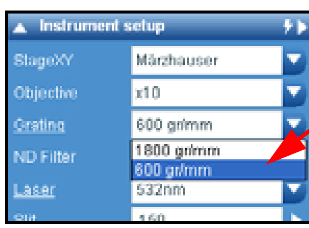
7 Choose the appropriate density filter: from LabSpec6, select the Acquisition Panel then choose the Video section. Click on the drop down menu of the ND filter parameter.



Select one of the 9 neutral density filters: 100%, 50%, 25%, 10%, 5%, 3%,1%, 0,1%, 0,01%

8 Verify the following parameters in the LabSpec6 software: grating, spectral range, objective. See details in the LabSpec6 User Manual, part number 31 089 151.

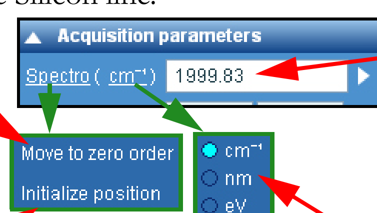
- Two gratings are mounted on a motorized turret. Select the grating you want to use.



Click here to select one of the available spectrograph gratings

- To look at a specific range of the Raman spectrum enter 520.07 cm^{-1} to set the central position to the Silicon line.

Move the spectrograph to the zero order position (0 nm)



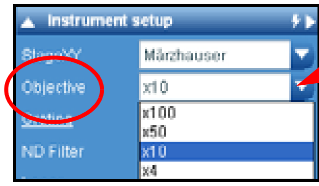
Enter here the spectrograph position value: 520.7 cm^{-1}

Initialize the Spectrograph. Move to the zero order position and then move to the position value previously set

Select the cm^{-1} working unit

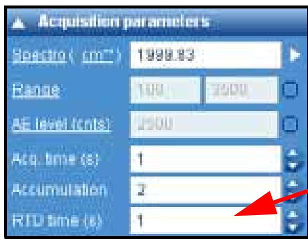
06/26/2013

9 Select the objective you have chosen in the paragraph 4.

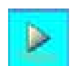


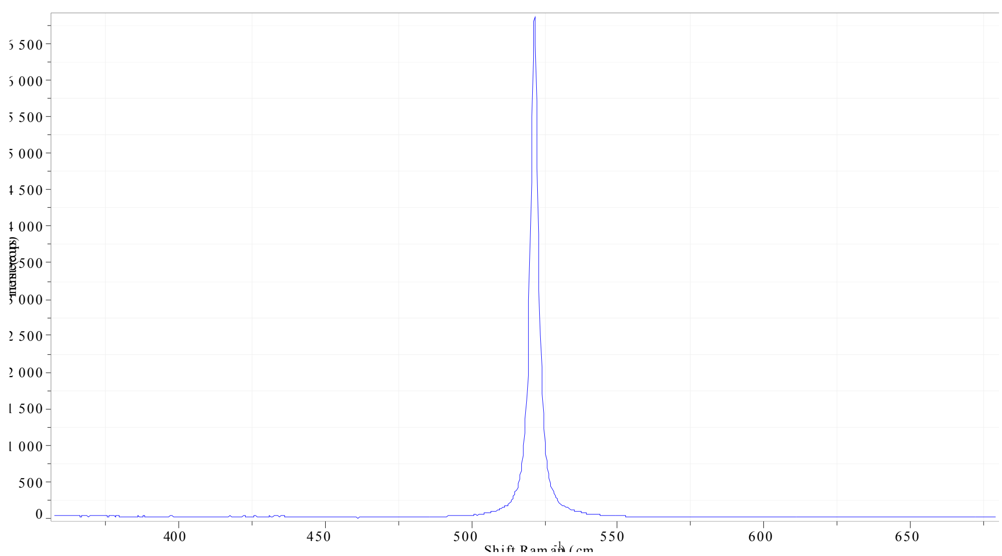
Click here and select one of the objectives available in your configuration

10 From the **Acquisition Panel > Acquisition Parameters**, enter 1(second) in the RTD time (s) parameter (see figure below). .



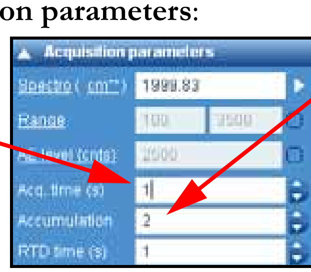
Enter an RTD Acquisition Time of 1s

11 Click on the RTD icon  to launch a continuous Real Time Display measurements. The acquired spectrum must shown the Silicon spectra line on 520.7 cm^{-1}. Click on  to stop the RTD measurements0.

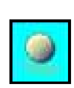


12 Before launching an acquisition, enter the following parameters located in the Acquisition **Panel > Acquisition parameters**:

Enter the integration time: 1s is a good starting value



Enter the number of accumulation. Acquiring multiple accumulations of data and averaging them results in improved spectrum quality

Once done, click on the Acquisition icon  to launch a single acquisition.

CHAPTER 3

Appendix 2: Imaging Options

Raman imaging is a powerful technique for generating detailed images based on acquired Raman spectra of a sample. A complete spectrum is acquired at each and every pixel of the image, and then interrogated to generate false color images based on material composition and structure.

To perform Raman imaging, several methods can be used based on options described in this chapter.

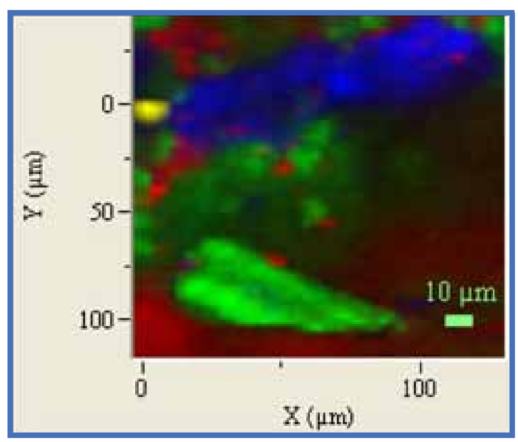
4.1 Motorized stage

4.1.1 XY motorized stage

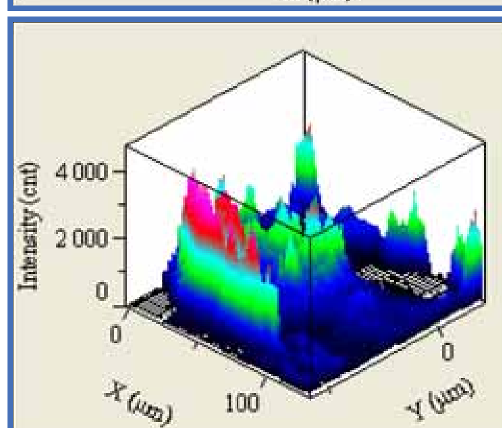
Depending on your LabRAM HR Evolution configuration and especially the microscope type, a motorized XY stage can be added.

The motorized stage is driven by the LabSpec6 software via an external controller. Once the sample has been focused, the acquisition surface mapping can be built. A XY Raman image of a sample can be delivered. Please refer to the LabSpec6 User Manual, P/N: 31 089 151. .

Example of 2D Raman image presentation



Example of pseudo 3D presentation: representation of 2D Raman image, where all three axes (intensity plus two spatial) are displayed.

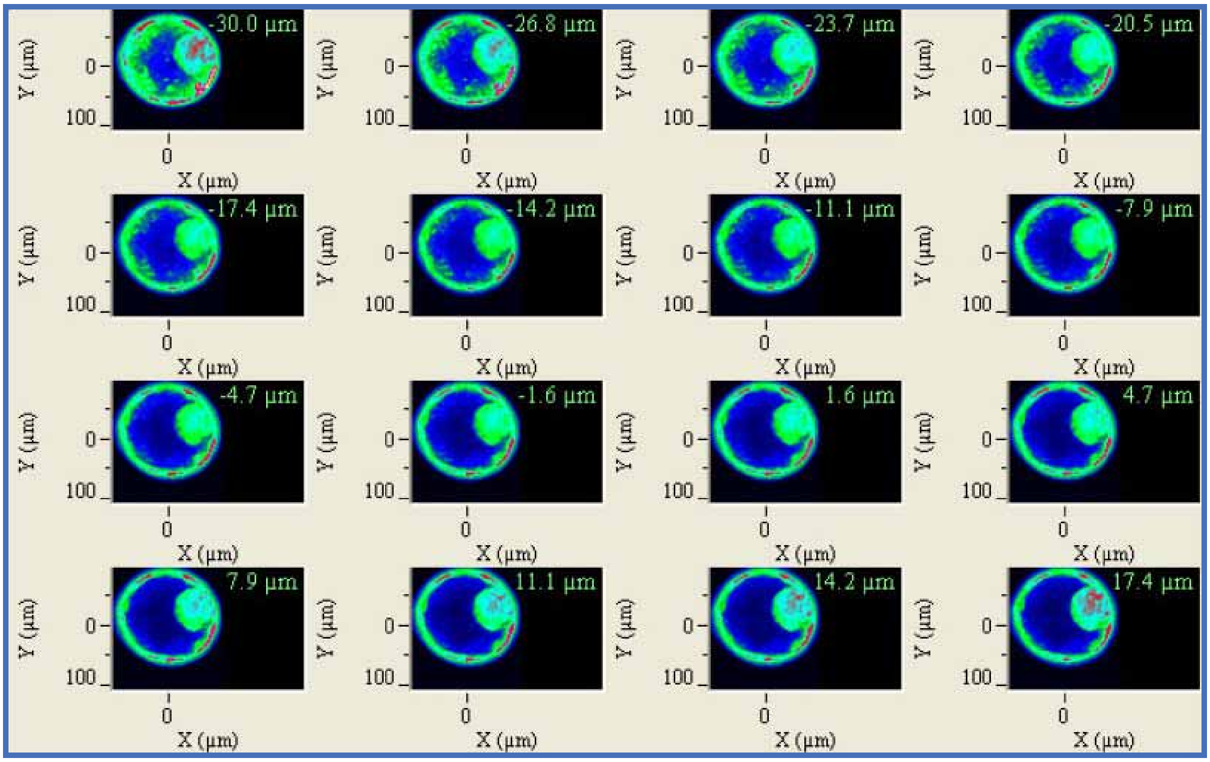


06/26/2013

4.1.2 XYZ motorized stage

The Z motorized stage adds new capabilities for a Raman 3D imaging. In conjunction with a small confocal hole, the depth dimension can be added to the XY mapping. The system then delivers a complete 3D Raman image.

The Z motorized functionality can be used to focus the sample with the Autofocus option or can be also used in combination with the confocal hole to perform a Z (depth) mapping on a sample. Please refer to LabSpec6 software manual, P/N 31 089 151..



Example of Raman images acquired between -30 to 17.4 µm Z depth, with a 3.2 µm step.

4.1.3 Confocal principle

The LabRAM HR Evolution includes an integrated confocal microRaman system. «Confocal » optics means that the sample is illuminated with a diffraction limited spot and that the illuminating spot is imaged on an ideally point-like detector. Practically, the point-like detector is realized with an adjustable pinhole called « **confocal hole** » in front of the real detector (hole placed at the spectrograph entrance).

06/26/2013

The advantage of confocal sampling is a considerable reduction of the depth of focus and thus an increased Z discrimination.

For example, this means that a confocal microscope allows separating the signal from each layer of a laminar/multilayer sample, or isolating the signal from an inclusion against the signal coming from the surrounding medium.

Confocality performances can be measured in different ways. One possible experiment, widely acknowledged, lies in the measure of the intensity of the silicon line when a flat silicon surface is scanned through the focus of a microscope objective.

Silicon is chosen because of its strong Raman signal and weak penetration depth. Thus a silicon flat surface models an infinite plane of infinitesimal thickness.

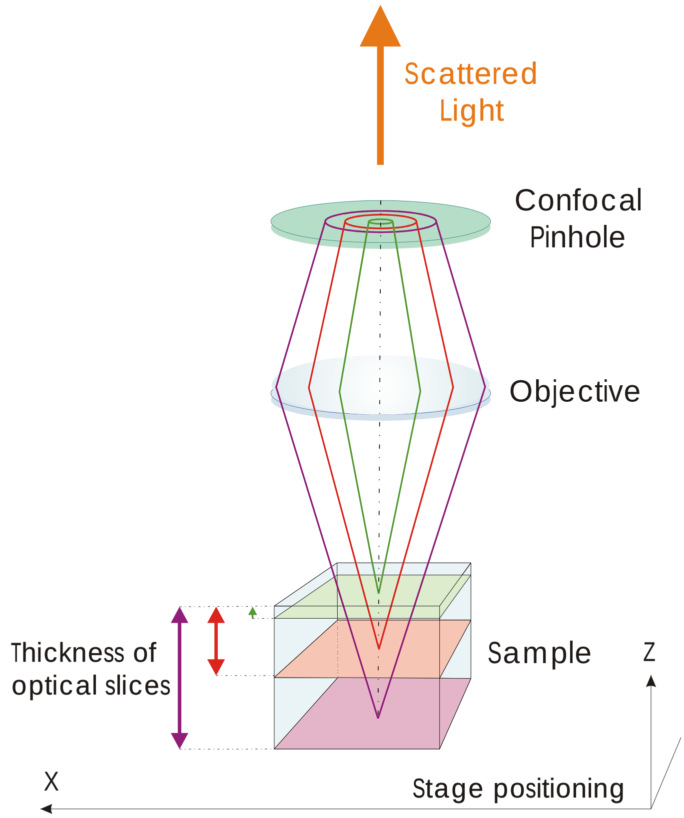


Figure 2-1 Confocal principle

When you scan a silicon surface along the optical axis, the intensity becomes very low when you are slightly out of focus (because the image of sample is out of focus on the sharp confocal hole) and reach a maximum when the silicon surface is on the focus of the laser spot (because in this case all the signal can propagate through the confocal hole).

The **FWHM** (Full Width at Half Maximum) of the curve Intensity vs. Defocus is taken as a measure of the « **confocality** ». The confocality changes in function of the aperture of the confocal hole. The smaller the confocal hole, the faster is the drop of the Raman signal as you go out from focus.

The figure 2-1 shows how a confocal pinhole determines the optical slice thickness of a sample. Thus, if the sample is located on a motorized XY stage it becomes easy to make an analysis surface inside a sample using the mapping feature. Using a Z motorized Sample Stage, this measure can be completely automated. A 3-D Raman image can be then obtained.

06/26/2013

1.2 DuoScan Mode

The DuoScan™ module consists in two moving mirrors which are located above the sample/objective lens. Each mirror moves in orthogonal direction so the laser beam rasters over an area (a macropixel) horizontally, vertically, and diagonally. The mirrors of the double scanner deflect the laser beam to cover an area without moving the sample. Thus DuoScanTM enables continuous fast scanning over a surface. This is the **Scanning mode**.

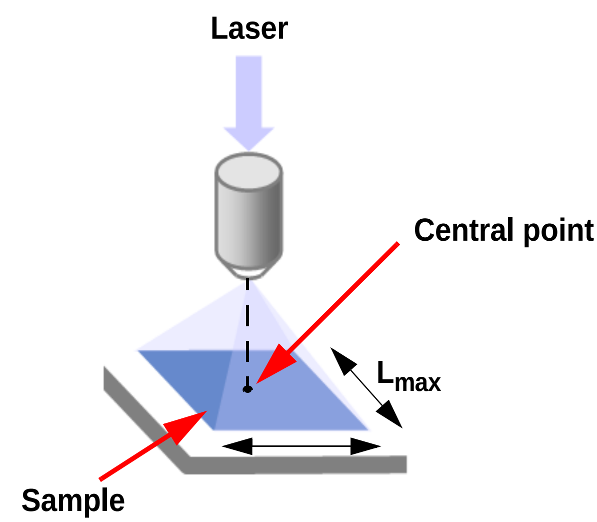


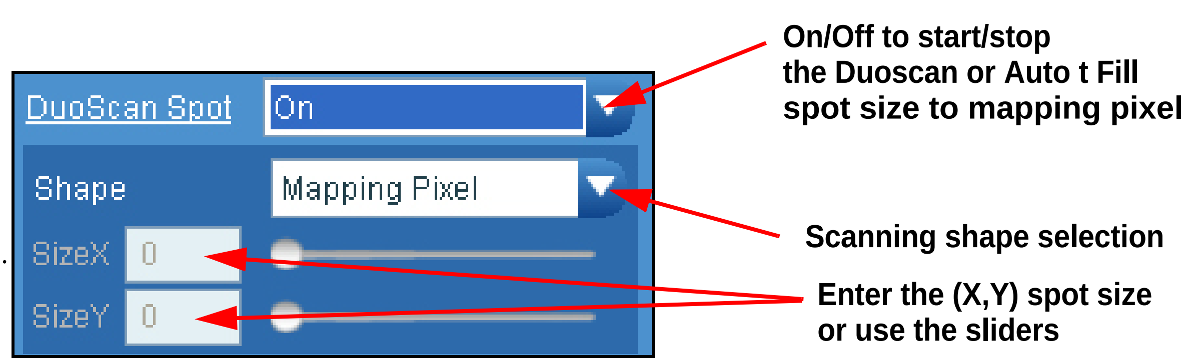
Figure 4-2 DuoScan™ principle

In addition, the mirrors included in the DuoScan™ deflect the laser beam with very high precision and discrete steps. This enables mapping of the sample with very small step size. This is the **Stepper mode**.In addition, the mirrors included in the DuoScan™ deflect the laser beam with very high precision and discrete steps. This enables mapping of the sample with very small step size. This is the **Stepper mode**.

1.2.1 Using DuoScan™ with LabSpec6 software

1.2.1.1 Scanning modes

When DuoScan™ is installed, acquisition can be started using the LabSpec6 software. From the Acquisition Main Panel, select the [Acquisition Parameters] section. Click on the **DuoScan spot** text to open the additional Duoscan setup menu (see the figure below). Enter the Size X and Y in µm. From the "DuoScan spot" drop down box, select **On** to start the mirrors scan, Auto to «**Fill spot size to mapping pixel**» or **Off** to stop the scanning.



On/Off duoscan button: This button start or stop the scanning device. It can be used for settings.

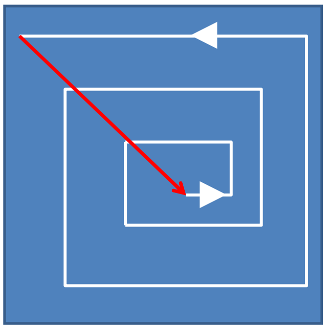
06/26/2013

Auto: Fit spot size to mapping pixel: if the **Auto** choice is selected, the scan area is set according to the pixel size specified in the *Acquisition > Map* section dialog window. Set the "X/Y" and "From/To" to define the area. See LabSpec6 User Manual for details.

Make a measurement, to obtain an average spectrum from the scanning region. When the measurement stops, DuoScan mirrors will automatically stop scanning.

When a new measurement is started, DuoScan mirrors will automatically start scanning.

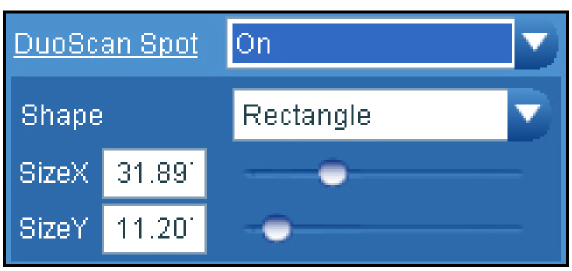
The scanning area is shown below:



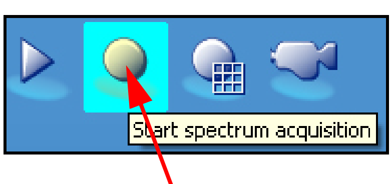
NOTICE: if SWIFT™ ultra-fast Raman mapping is activated the "Width" value will be set to 0, because SWIFT™'s continuous scanning in the X direction does not require DuoScan™ scanning in the X (width) direction.

Starting acquisition

- After you have selected a scanning mode.



- Launch a spectrum acquisition.



Start spectrum acquisition

- Once done, stop the DuoScan mirrors by selection **Off** from the **DuoScan Spot**.

1.2.2 Guideline to select the most efficient mapping mode

This table is given as a guide to the user for choosing the most suitable mapping mode. The dimension of the area and the mapping mode can obviously be chosen according to a specific application.

	Area < 10x10µm²	**10x10µm²< Area< 100x100µm²**	**Area > 100x100µm²**
	High accuracy mapping	High to Medium Accuracy mapping	Large area mapping
Steps	Down to 0.05µm	Down to 0.1µm	Over 1µm
Mode	DuoScan™ Stepper mode	XY stage	DuoScan™ Scanning mode +XY stage
Application	Submicron to micron scale objects	Micron to tens of microns objects	Tens of microns objects and above

1.3 XY, XYZ piezo mapping stage

The XY and XYZ mapping stage offer the same functionality as the motorized stage with the exception of the travel range which is 100x100 µm or 200x200x200 µm with a minimum step size of 1nm.

Thus, for small samples, a better overall precision can be reached.

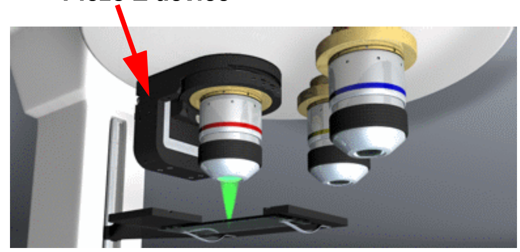
The Z adjustment can be used for focusing a sample with the Autofocus option or can be used to perform a Z (depth) mapping with the confocal hole.

To use this stage, please refer to the LabSpec6 User Manual.

1.4 Piezo Z device

The Piezo Z families is a small device screwed on the microscope objective revolver and compatible with all the objectives. A piezo-electric device insures a Z motion along 80µm or 300µm height with a precision of 1nm. Two versions are available: 80µm or 300µm height motion. The device is delivered with a servo control loop electronic.

Piezo Z device



As for the other Z motorized devices, the Piezo Z can be used to focus a sample with the Autofocus option or can be used in combination with the confocal hole to perform a Z (depth) mapping on a sample.

06/26/2013

To use this device, please refer to the LabSpec6 User Manual.

1.5 Autofocus option

The Piezo Z device or motorized Z stage is mainly aimed at two applications: Z-profiling and autofocus operation especially for mapping samples with rough surfaces. The Autofocus system is constituted of two parts: the Z motorization (Piezo Z device or Z stage stepping motor) and the light detection unit.

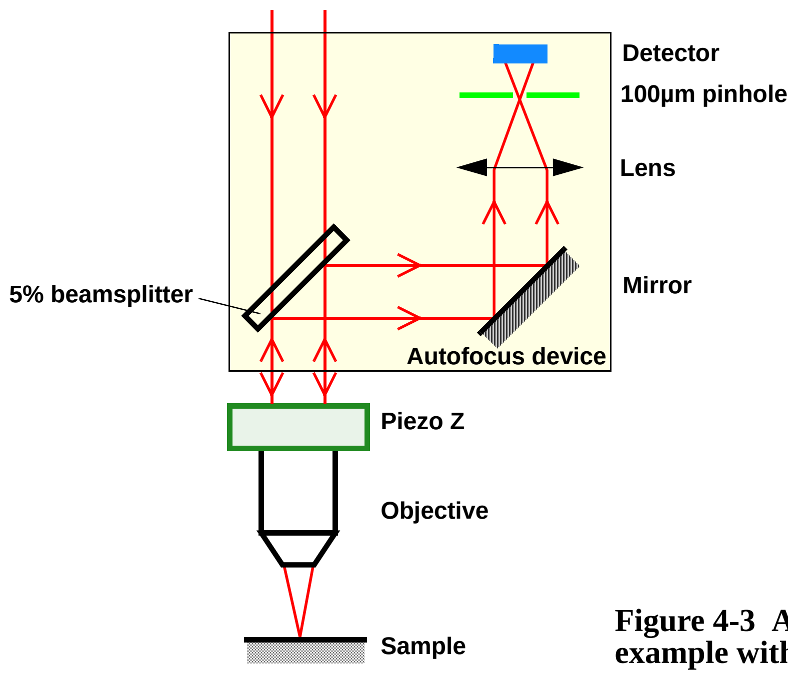


Figure 4-3 Autofocus diagram example with a Piezo Z device

On the optical path, a fixed beamsplitter splits 5% of the backscattered laser light towards the focusing lens on a 100μm pinhole. The detector collects the light intensity and leads the Z adjustment for a maximum intensity.

The global system accuracy is characterized by the positioning error Δz. This induces an error ΔI for the detection of the maximum intensity, corresponding to the focusing on the surface.

06/26/2013

CHAPTER 4

Appendix 3: Grating Replacement

This chapter explains how to change a grating inside the LabRAM HR Evolution instrument.

For a better understanding of the procedure, the films below show how to perform this action.

1

Film 7-1 Selecting the grating from LabSpec6

2

Film 7-2 Changing a grating

06/26/2013

HORIBA Scientific

Unit: LabRAM HR Evolution Sub-unit: Grating	UTILISATION SHEET	
	US01	Page 1/4
Subject: Grating replacement		Personnel: 1 Time: 10 mn
Tools needed: 1x 3mm male hex wrench Grating removing tool (delivered with LabRAM HR Evolution)		

The following procedure explains how to change a grating inside the Instrument.

Warning: do not touch or remove the parts which are not detailed in this procedure.

Preliminary: Shut down all the laser sources before changing the grating.

Caution: Never touch the grating surface!!

1

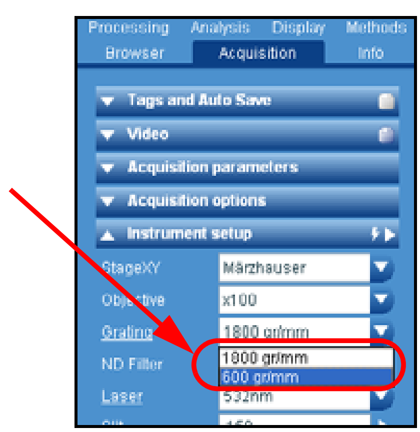
PRELIMINARY

IMPORTANT: The complete calibration without compromise has been factory made on the 1800 grooves/mm grating. The second grating has been ordered on request. If a new grating must be mounted inside the instrument, choose this second grating slot and do not replace the 1800 gr/mm grating.

At this step, if not already done, power on the system with the exception of the laser sources.

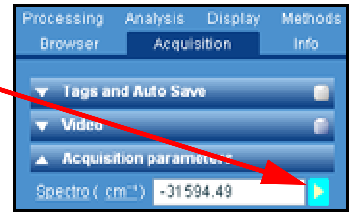
Using the LabSpec6 software, select the grating you wish to replace then activate the change. Move the grating to zero order: this action place the grating to replace ready to be removed. When the access trap will be opened, verify that the right grating label is face to you.

Grating selection

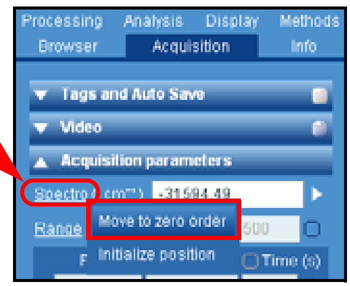


06/26/2013

Then activate the grating change



Click on the <spectro> parameter then select <Move to zero order>

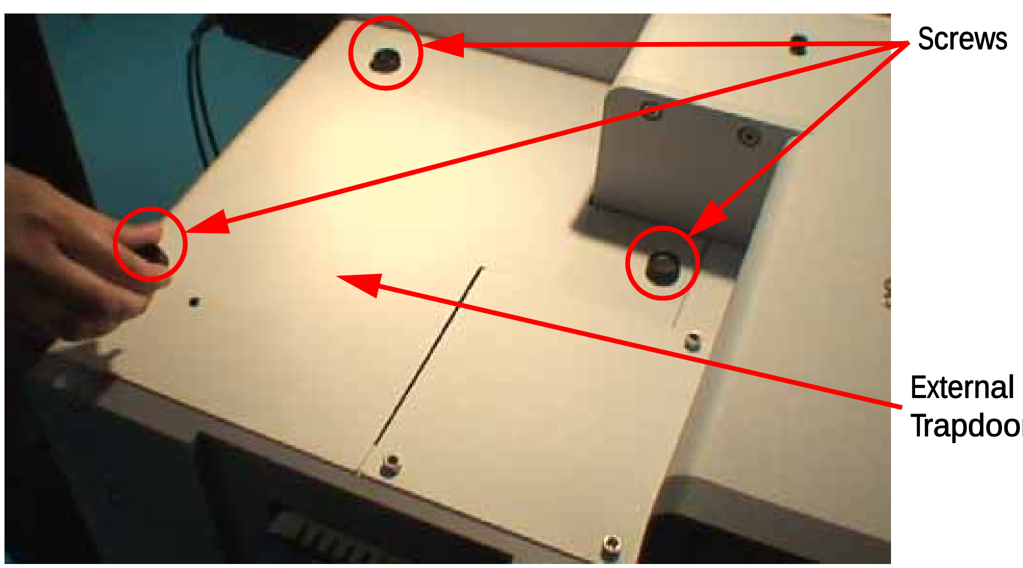


2

Access to the gratings:

There are 2 trapdoors to remove before to reach the gratings. The first trapdoor is located on the upper right hand side of the LabRAM HR Evolution Instrument.

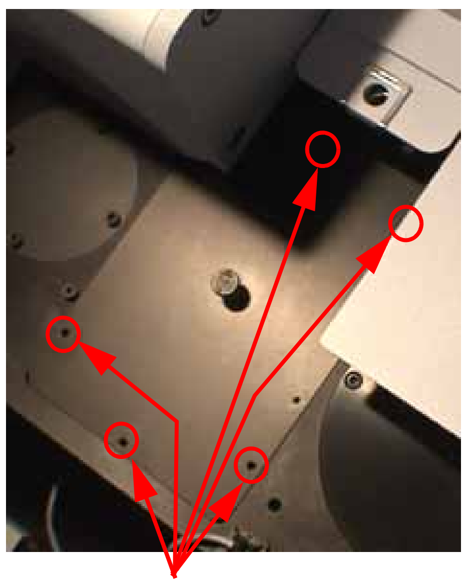
Using your fingers, unscrew the 3 screws which fix the trapdoor, then pull on the trapdoor to remove it.



06/26/2013

3

Once the external trapdoor has been removed, another trapdoor allows the direct access to the gratings. Figure below shows the trapdoor with the locking screws location. Using your fingers, remove the 5 screws, then pull out the trapdoor using the button located on the center.



location of the 5 screws securing the trapdoor

Grating

4

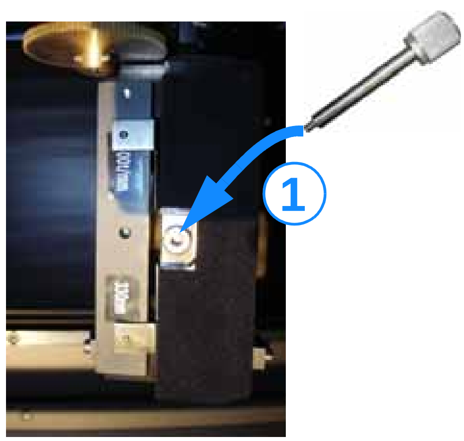
Each LabRAM HR Evolution is delivered with the «grating rod» tool shown on the figure below. This tool is required to remove the grating.

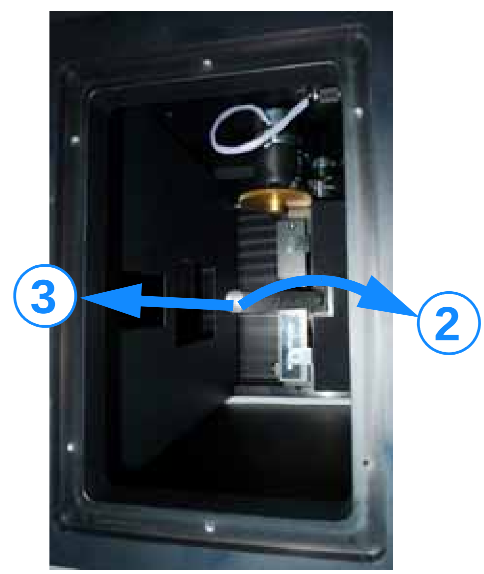
06/26/2013

5

Removing the sole plate

1. Looking to the grating, there are two holes located next to each other. Insert and screw the rod into the right hole,
2. Bend the rod to the right. This action free the locking snug;
3. While bending, pull out the sole plate.





06/26/2013

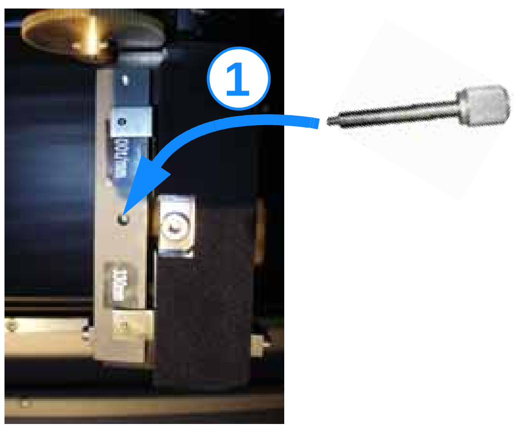
6

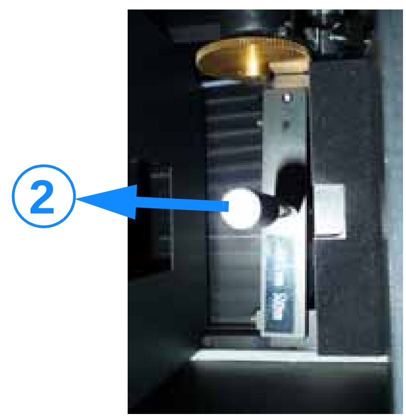
Removing the grating

1 Insert and screw the rod into the left hole, which is located on the grating frame.

2 Pull out the rod with the grating.

3 Insert immediately the grating inside the special dust-proof box and secure the grating with screws.





5

If another grating must be place, proceed in the reverse order of dismounting. There is no particular difficulty.

06/26/2013

CHAPTER 5

Appendix 4: Adding polarization accessories

Polarized Raman uses polarized laser excitation and a polarization analyzer that can be used to acquire spectra, either parallel or perpendicular to the excitation laser. The resulting spectral information gives an insight into molecular orientation and vibrational symmetry. In essence, it allows the user to obtain valuable information relating to molecular shape, for example in synthetic chemistry or polymorph analysis, and is most often used to understand the orientation of molecules in organized environments such as crystal lattices, liquid crystals and polymer samples.

A large choice of polarization accessories are available: see "Polarization Accessories for Raman Measurements" on page 55.

The films below show how to insert polarization accessories inside LabRAM HR Instruments.

1

Film 7-1 Accessing to the optical drawer

06/26/2013

2

Film 7-2 Adding a laser polarizer or an analyzer

CHAPTER 6

Appendix 5 Instrument setup when lasers are selected

The LabRAM HR Evolution has two laser entrances with an automated commutation. The entrance interferential filters are located on each laser path. These filters are located inside the lasers bench commutation box or in the optical drawer if the system has an internal laser. Each one is adapted to the used laser wavelength. Many external lasers can be mounted on the system. The selection of these lasers is made from LabSpec6 software and everything is done automatically with the exception of the Rayleigh filter which must be fit and set according to the selected laser wavelength.

This chapter details what are the elements to change and how to change them using figures and films (pdf version).

What I have to setup when I select a new laser wavelength?

- Rayleigh (Edge or Notch filter) set at the laser wavelength,
- Spacer for optimization of the Rayleigh (Edge or Notch) filter.

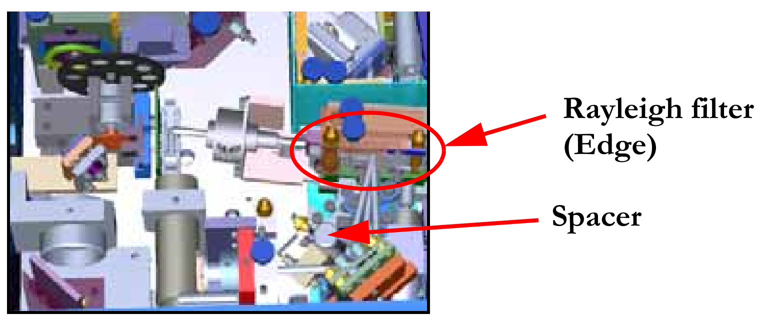


Figure 7-1 optical elements location

Please follow the two steps shown on the following films:

06/26/2013

1

Film 7-1 Accessing to the optical drawer

2

Film 7-2 Optical elements to change if another external laser is selected

How to choose the spacer size?

“Appendix 6: Edge Filters” on page 66 explains how the angular deviation of an edge filter acts on the rejection of the exciting line. The Edge filter operates in the same manner excepting that it is only a pass high filter and not a band-pass (notch filter).

The angular deviation is set by the spacer. LabRAM HR Evolution is delivered with a set of spacers. To know the spacer to use, please refer to the LIRS page in the QC book of your system.

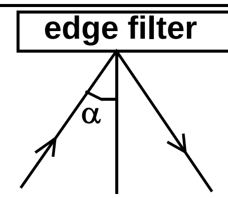
06/26/2013

Spacer Diameter (mm)	4	5	6	7	8	9	10	11
Angle of the rayleigh filter (degrees)	9.84	8.69	7.58	6.59	5.69	4.86	4.05	3.3

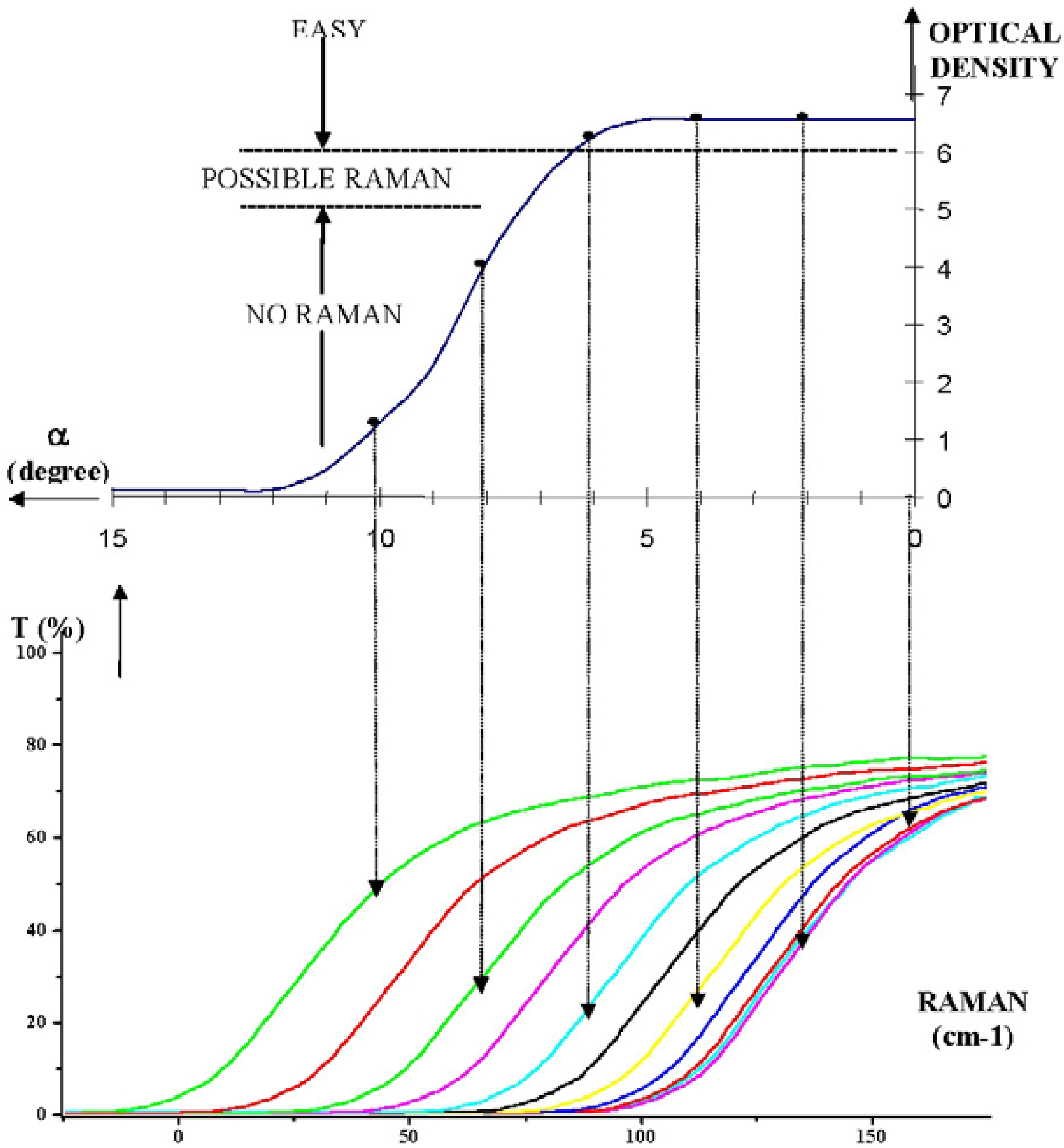
CHAPTER 7

Appendix 6: Edge Filters

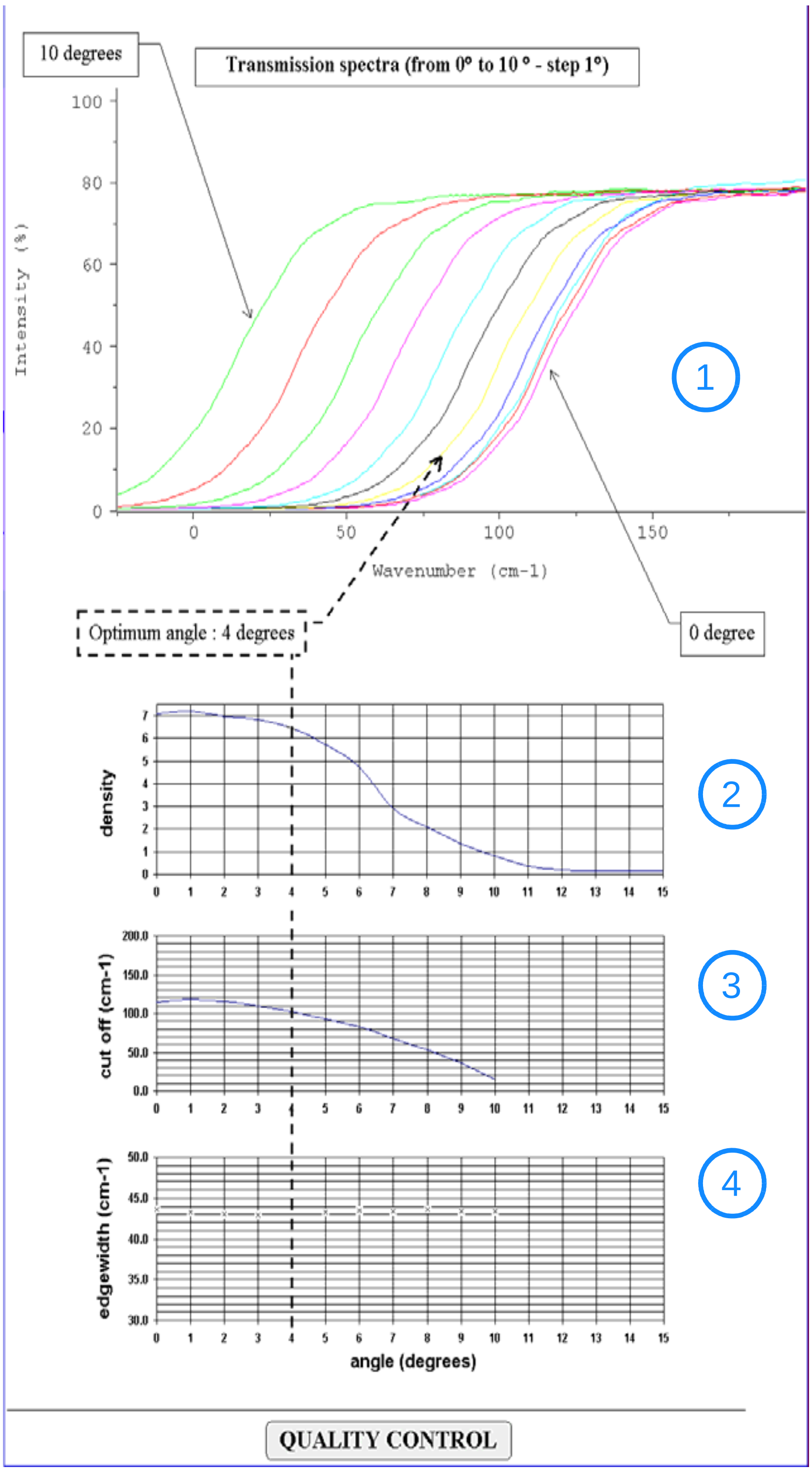
A edge filter is used in the LabRAM HR Evolution for the rejection of the exciting line. It is specific for one laser wavelength. When you change the excitation wavelength you must change also the edge filter.



The filtering of the exciting line depends on the α angle



Characterization example of a edge filter



1 Filter transmission versus α angle

2 Optical density versus α angle

3 Cut-off position versus α angle

4 Spectral edge width versus α angle

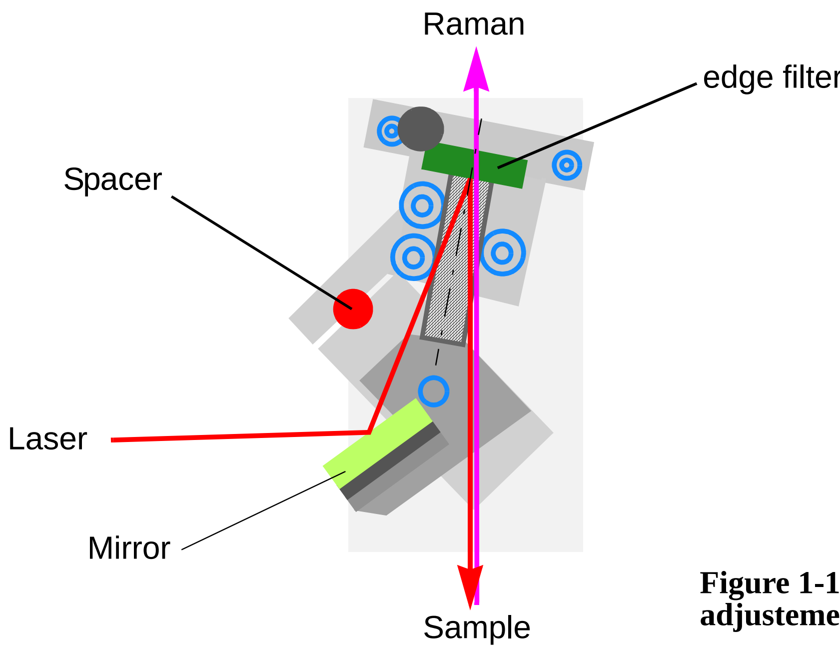


Figure 1-1 edge filter adjustement

Spacer Number	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Spacer Diameter (mm)	4	5	6	7	8	9	10	11
Angle of the edge (degrees)	9.84	8.69	7.58	6.59	5.69	4.86	4.05	3.3

The edge filter cut-off shape can be verified with the microscope transmission white lamp with a x10 objective. If the spectrograph is positioned on the exciting line you can record a spectrum.

Index