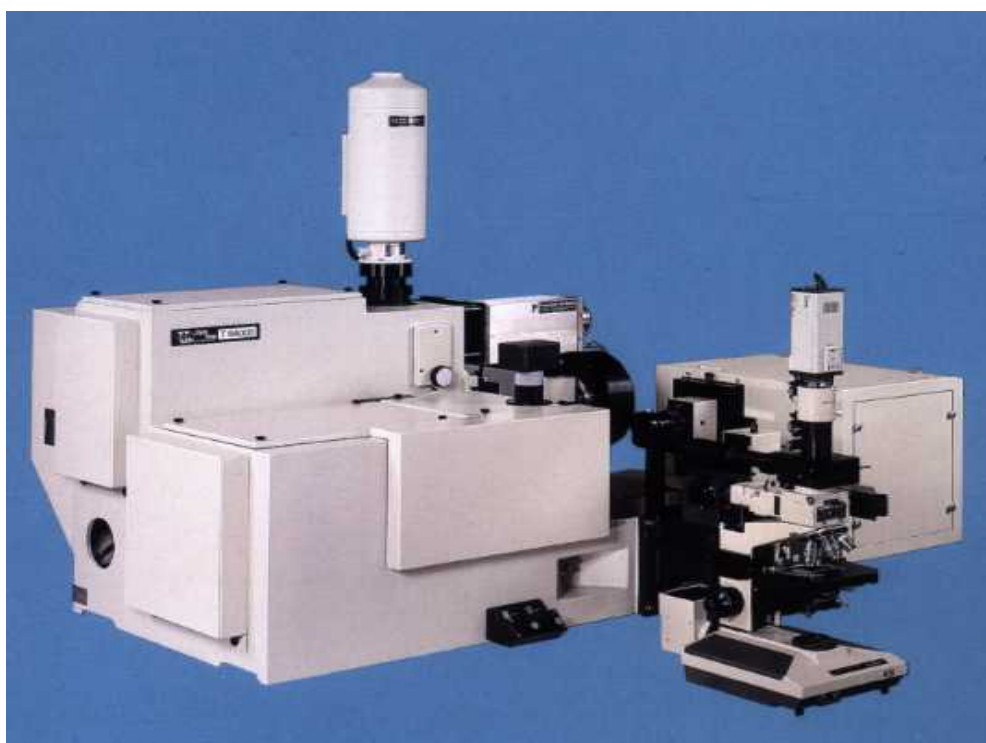


# T64000

## User's Training Course



## User's Training Course

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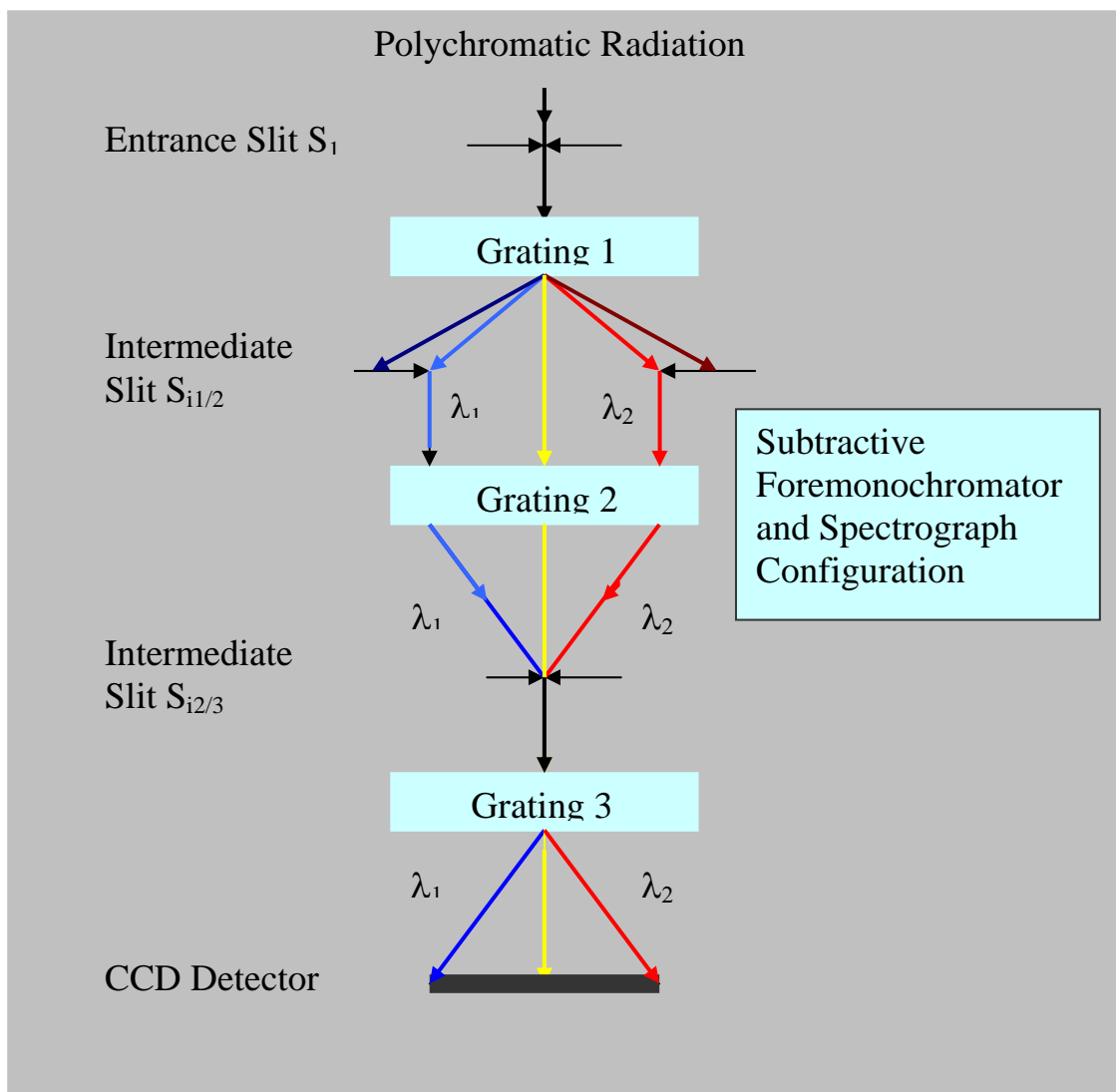
# 1. Optical configurations

## 1.1. Basic configuration : double subtractive + spectrograph

### 1.1.1. Optical diagram ( Figure 1)

### 1.1.2. Function of the double subtractive foremonochromator

The double subtractive foremonochromator is used as a tunable filter in the spectral range defined by the scanning mechanism and the gratings. A polychromatic radiation enters the first monochromator through the entrance slit  $S_1$  and is dispersed by the grating  $G_1$ . The exit slit of the first monochromator (entrance slit of the 2<sup>nd</sup> monochromator)  $S_{i1/2}$  selects a bandpass between  $\lambda_1$  and  $\lambda_2$ . The grating  $G_2$  in the 2<sup>nd</sup> monochromator recombines all the dispersed radiations on the exit slit  $S_{i2/3}$  giving again a polychromatic radiation, but limited to only the spectral range between  $\lambda_1$  and  $\lambda_2$  (see following diagram).



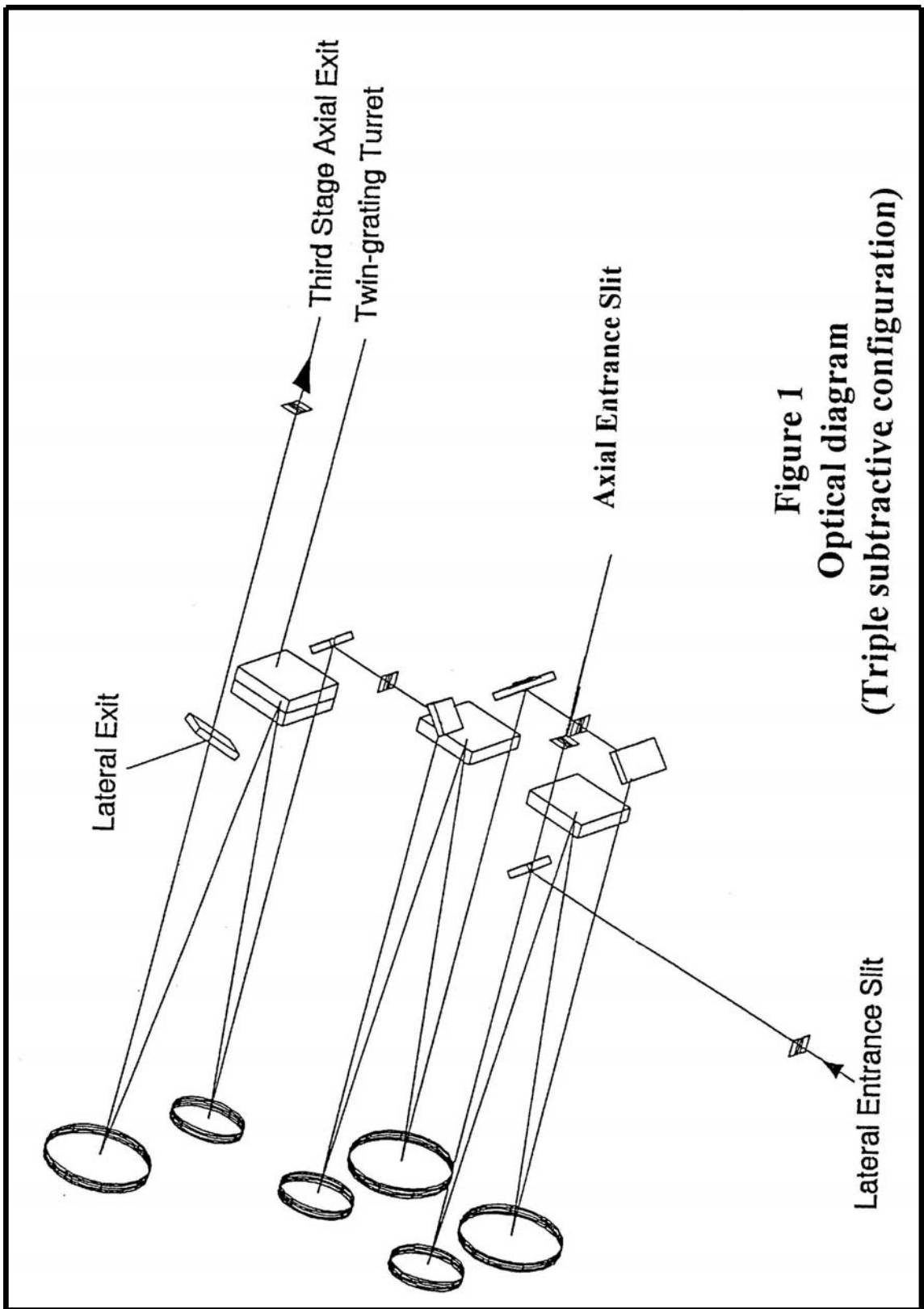


Figure 1  
Optical diagram  
(Triple subtractive configuration)

Remarks for the best use of this configuration :

1. The width of the slit  $S_{i2/3}$  must be equal to the width of the entrance slit  $S_1$  to have the foremonochromator and the spectrograph correctly coupled.
2. The width of the slit  $S_{i1/2}$  must be equal to the length of the multichannel detector for its full coverage when all the gratings have the same grooves density and the exit focal length of the foremonochromator and the spectrograph are the same. If the grooves density of the grating in the spectrograph stage is lower,  $S_{i1/2}$  must be wider than the length of the multichannel detector.

**1.1.3. Function of the spectrograph**

The spectrograph is used as a disperser.

The polychromatic radiation selected by the foremonochromator between  $\lambda_1$  and  $\lambda_2$  is dispersed by the grating  $G_3$  of the spectrograph. The spectrum is acquired with a multichannel detector which is mounted in the plane of the exit image or with a monochannel detector through an exit slit.

**1.1.4. General specifications**

1.1.4.1. Scanning mechanism

The T64000 system is equipped with two scanning mechanisms, one for the foremonochromator and another one for the spectrograph. Only the sine bar concept is used for the T64000 system. The advantage of this concept will be explained in the next paragraph (1.1.4.2.).

1.1.4.2. Spectral range

Basically, the T64000 is equipped with 1800gr/mm gratings defining a mechanical range of 0-950nm. With a cosecant bar mechanism, the spectral range would have been reduced to 300-900nm. That means that with a sine bar mechanism, we can work on the much larger range with dispersions available. This is not possible with a cosecant bar mechanism.

Example

Grating	1800gr/mm	600gr/mm	300gr/mm
Spectral range with a sine bar	0-950nm	0-2850nm	0-5700nm
Spectral range with a cosecant bar	300-900nm	900-2700nm	1800-5400nm



This table indicates clearly that when using an ionized Argon laser at excitation and a sine bar, we can use any type of grating and this is not the case with a cosecant bar. The reason for which we would need to change the dispersion will be analyzed in the next paragraph (1.1.4.3.). Naturally, the response or efficiency of the system depends on the efficiency of the gratings and of the detectors. Typical efficiency curves of the gratings 1800gr/mm and 600gr/mm are represented on the Figures 2 & 3

1.1.4.3. Interchangeability of the gratings

To complete the advantage of the sine bar mechanism, the T64000 system is designed to give the possibility to easily interchange the gratings in each stage. (See Annexes)

Advantages

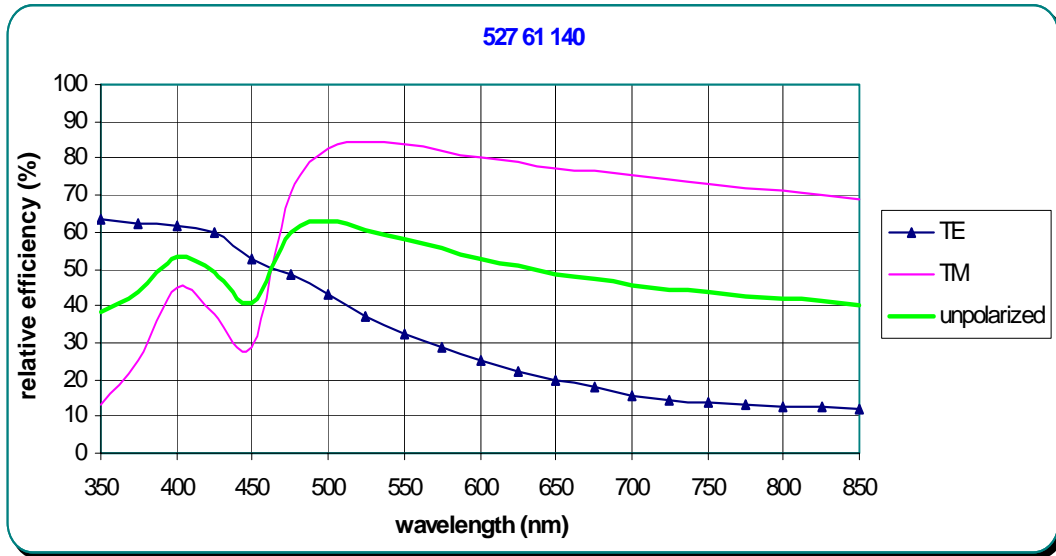
1. Large spectral range selection : from UV to IR
2. Changes the dispersion at a given wavelength
  - 2.1. Increases the detectivity of the system :  
 With a 1800gr/mm grating, a spectral line is seen by a number N of pixels. With a 600gr/mm grating at 600nm, because of the coverage on the CCD, the line is practically seen by a number N/3 of pixels. That means each pixel receives about 3 times more light, then the intensity of the line and the detectivity of the system is increased.
  - 2.2. Changes the spectral coverage on the CCD detector  
Example: with a CCD detector 25mm wide

Grating	1800gr/mm	600gr/mm
Dispersion at 600nm	0.64nm/mm or 17.68cm-1/mm	2.45nm/mm or 68.07cm-1/mm
Coverage on 25mm	About 16nm or About 440cm-1	About 61.25nm About 1700cm-1

- 2.3. Suppresses the artifacts introduced by gluing too many windows to have a complete spectrum.

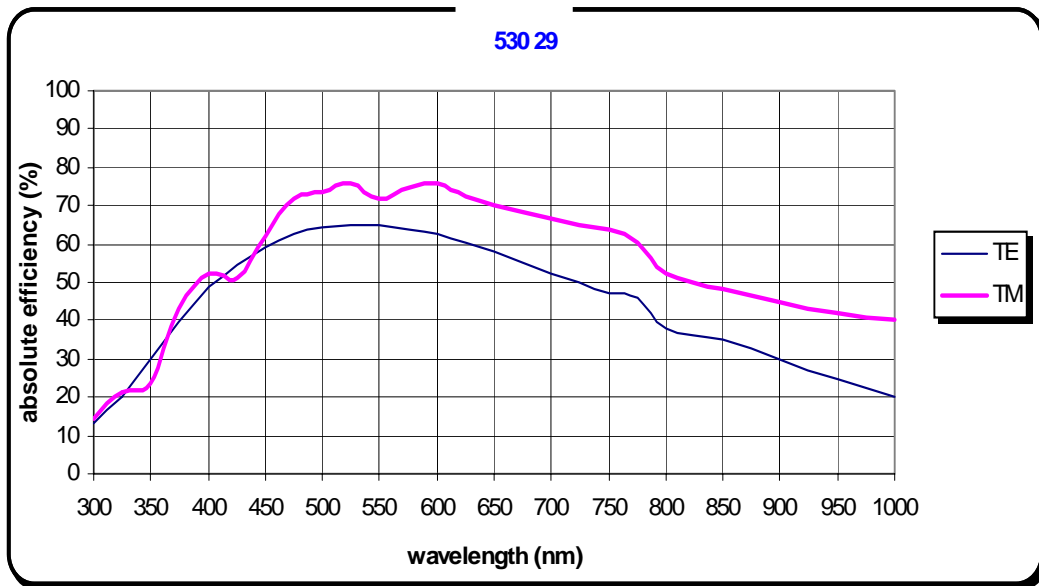
Disadvantages

The resolution is affected by using lower grooves density gratings.



Grating 1800gr/mm

Figure 2



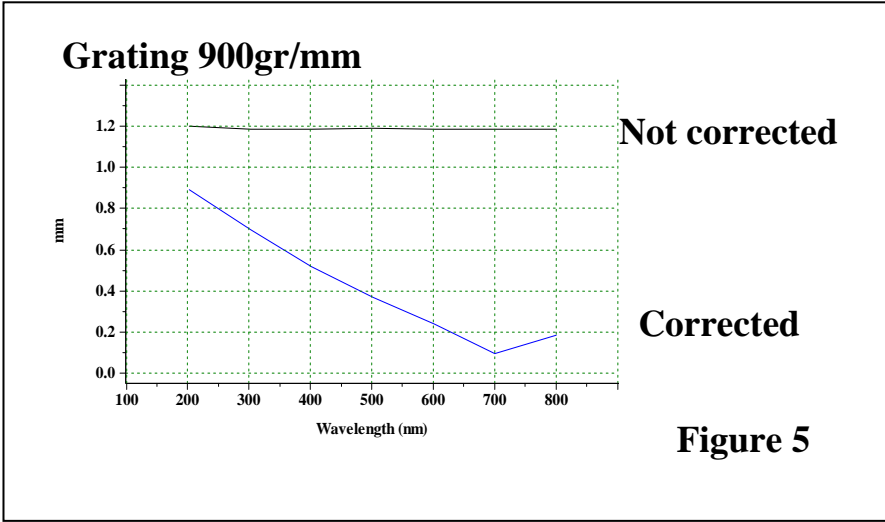
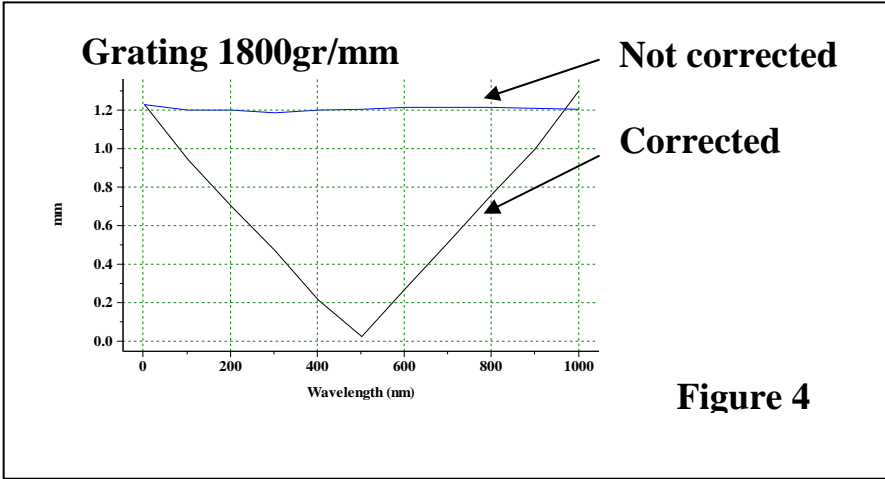
Grating 600gr/mm

Figure 3

1.1.4.4. Astigmatism corrected gratings

Astigmatism is characterized by the fact that a point at the entrance slit of a monochromator is imaged on a line at the exit slit. The geometry of the T64000 with its specific gratings corrects this effect and the image of a point remains a point at the exit slit. This correction presents many advantages combining the particular use of a CCD with the slit height limiter for stray light reduction and Raman imaging. Figures 4 & 5 show astigmatic curves of two types of gratings, 1800gr/mm blazed at 500nm and 900gr/mm blazed at 700nm.

**ASTIGMATISM / ONE STAGE  
CORRECTED AND NOT CORRECTED GRATING**



1.1.4.5. Height limiter on all the slits

The stray light is practically proportional to the surface of the slit. When working with the microscope or using the macro sample chamber to analyze solid samples giving small circular images on the entrance slit, the height limiter on the slits will be set at a small value for a good stray light rejection (See Figure 6)

1.1.4.6. Stray light rejection

The stray light rejection  $10^{-14}$ , cannot be directly measured on the T64000 system. This verification can be indirectly performed by acquiring Raman spectra of L-cystine powder or Silicon crystal, showing the possibility to scan very close the exciting line. (See Figures 7 & 8)

For the experimental conditions, see paragraphs concerning training with the macro sample chamber and with the microscope (2.3 and 3.3)

1.1.4.7. Resolution

Slit width and the type of detector, which is used, determine the resolution of the system in the basic configuration.

With a CCD detector

The resolution is better than 2 pixels. Any laser line or spectral line of different low-pressure spectral lamps can be used for the test. The entrance and the intermediate slits,  $S_1$  and  $S_{i2/3}$  must be equal or smaller than the size of the pixel. The intermediate slit  $S_{i1/2}$  is widely opened for the largest coverage on the CCD.

With a PMT detector

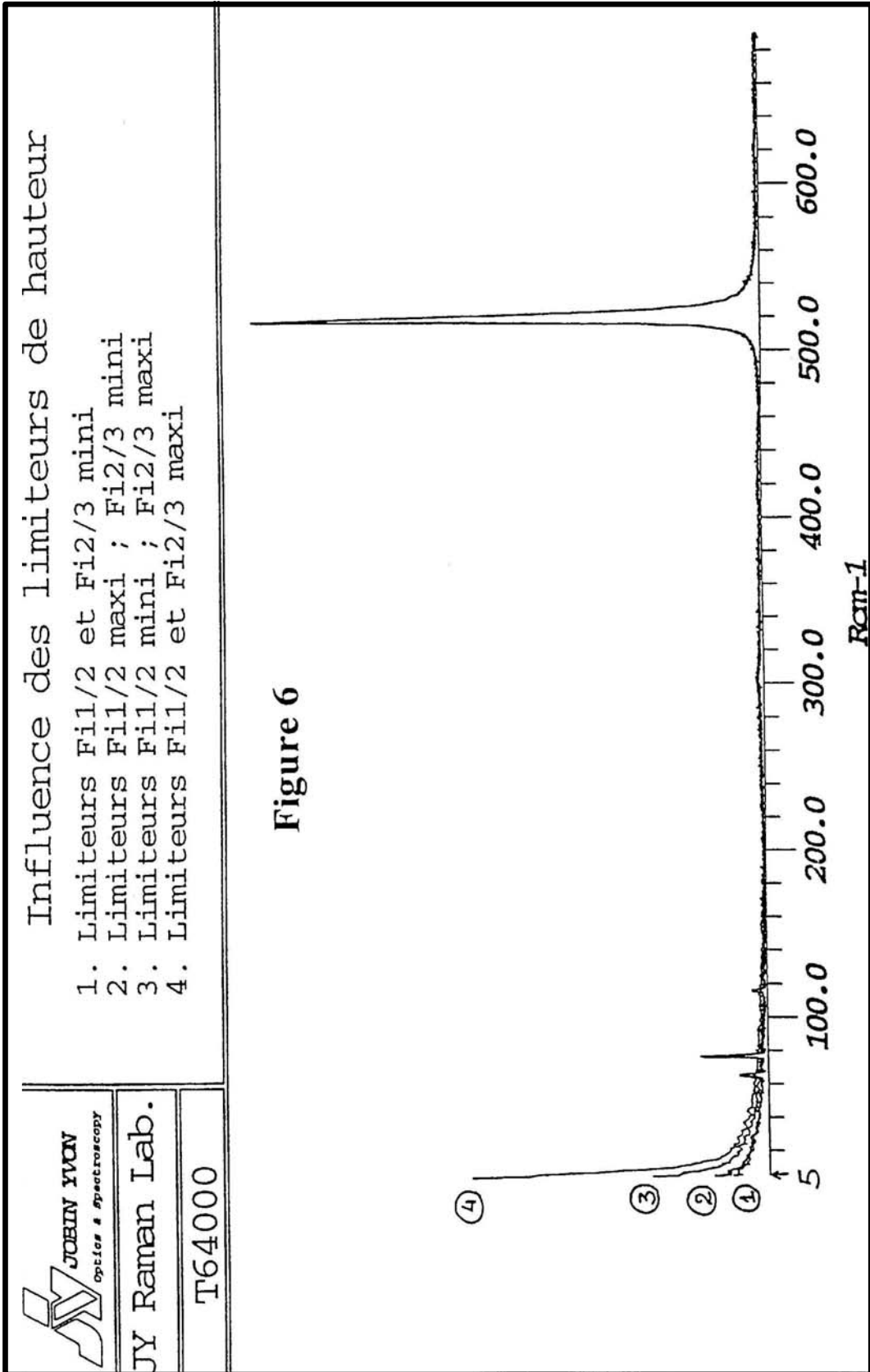
With the PMT detector, the highest resolution can be obtained using a narrow slit width. The resolution is better than 0.018nm with a 15 or 20  $\mu\text{m}$  slit width. Acquiring a  $\text{CCl}_4$  Raman spectrum showing the good separation of different isotopic lines can be the resolution test. (See Figures 9 & 10)

1.1.4.8. Accuracy

In the range between 450nm and 850nm, the wavenumber accuracy is  $\pm 1\text{cm}^{-1}$ .

1.1.4.9. Repeatability

Under normal conditions of temperature stability ( $\pm 1^\circ\text{C}$ ), the repeatability is 1 pixel with a CCD detector and  $0.1\text{cm}^{-1}$  with a PMT detector.



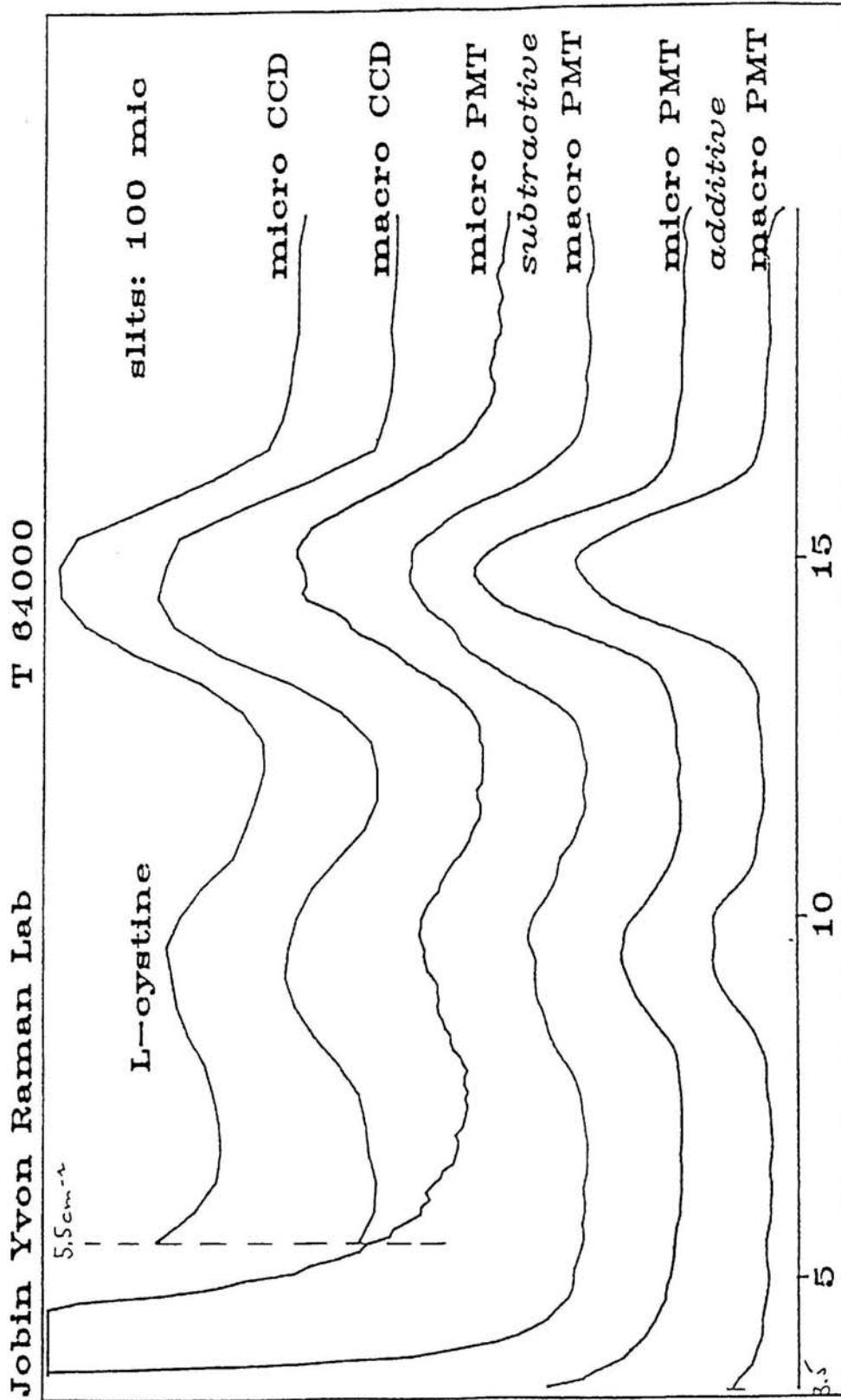


Figure 7

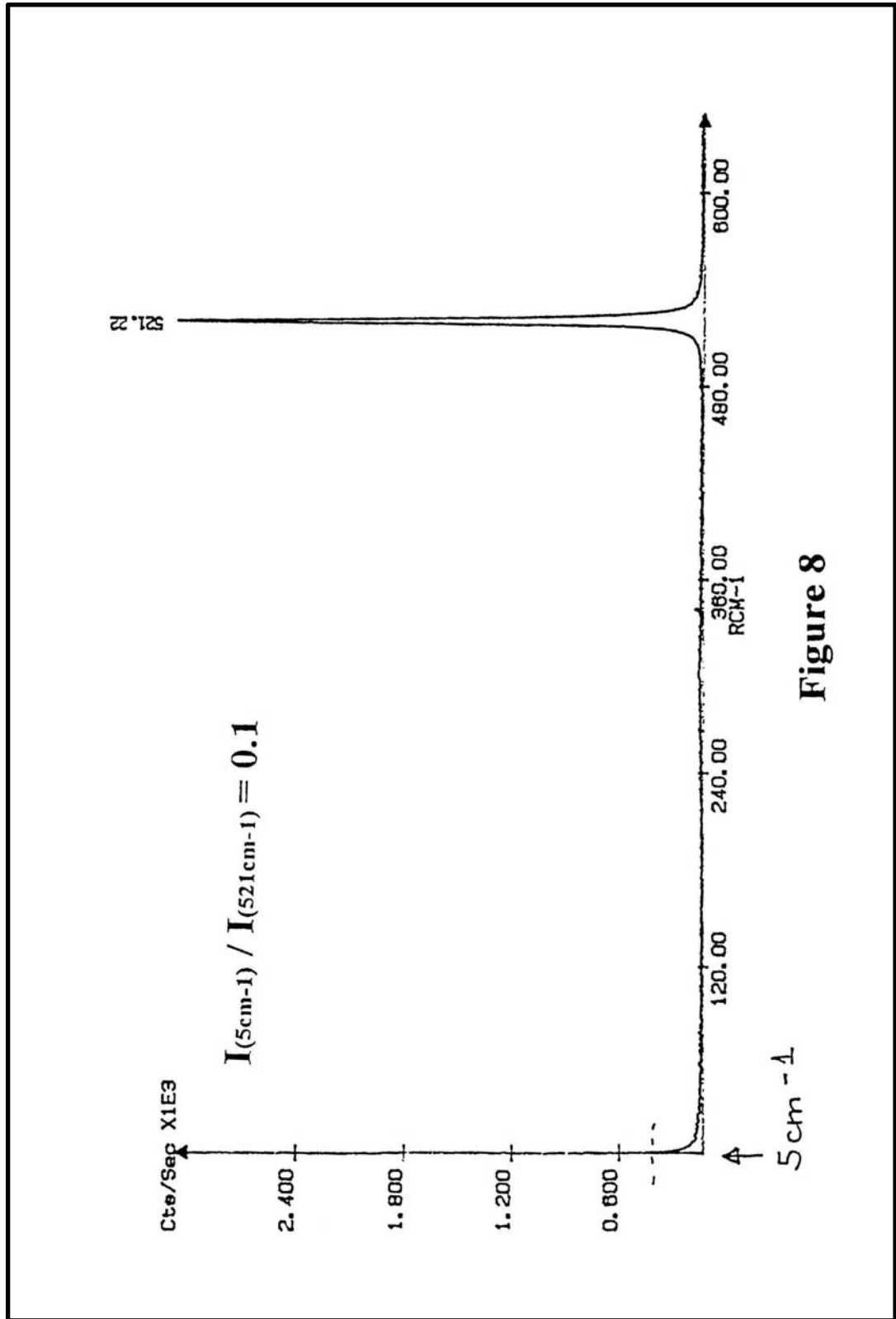


Figure 8

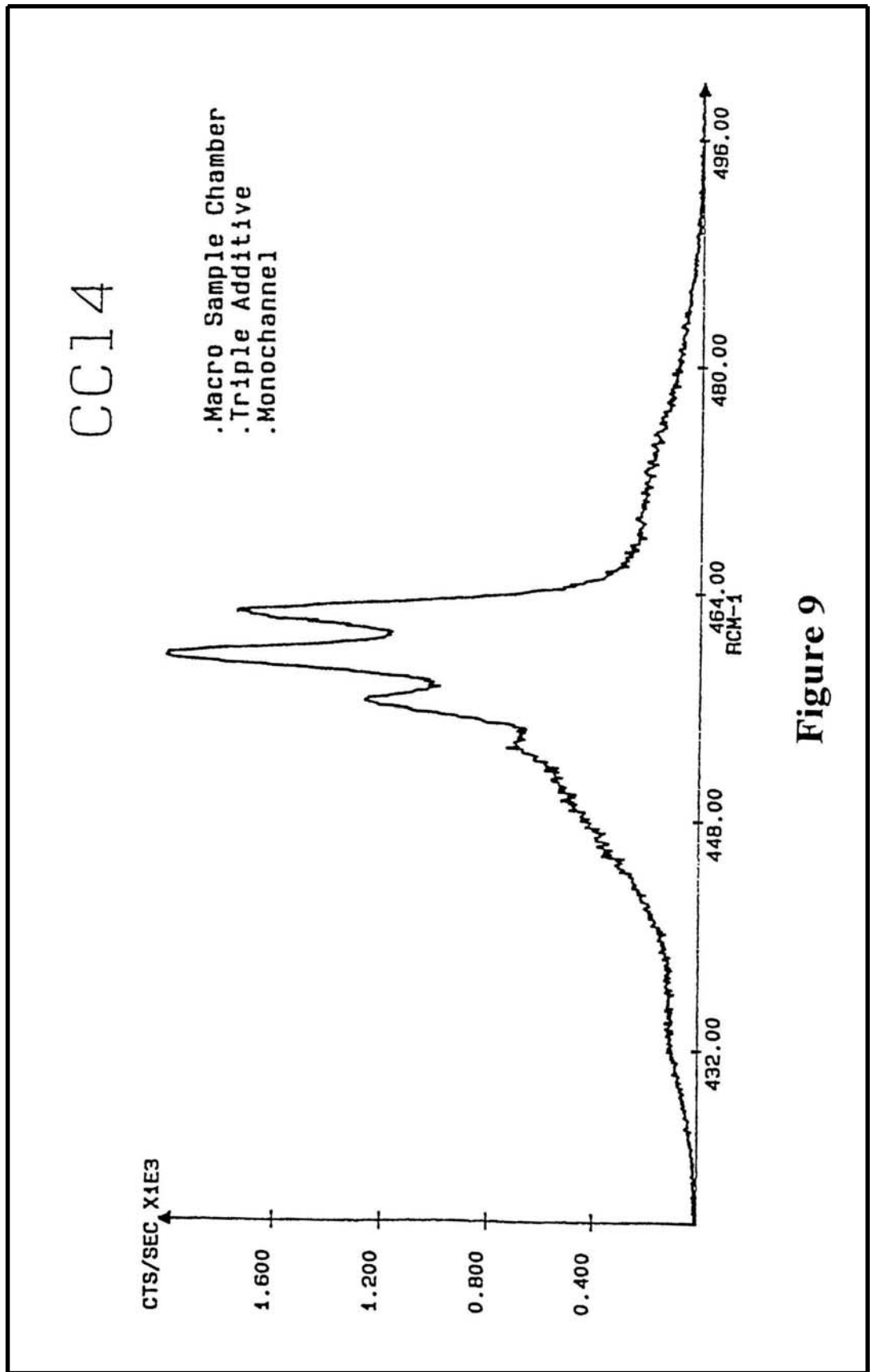


Figure 9



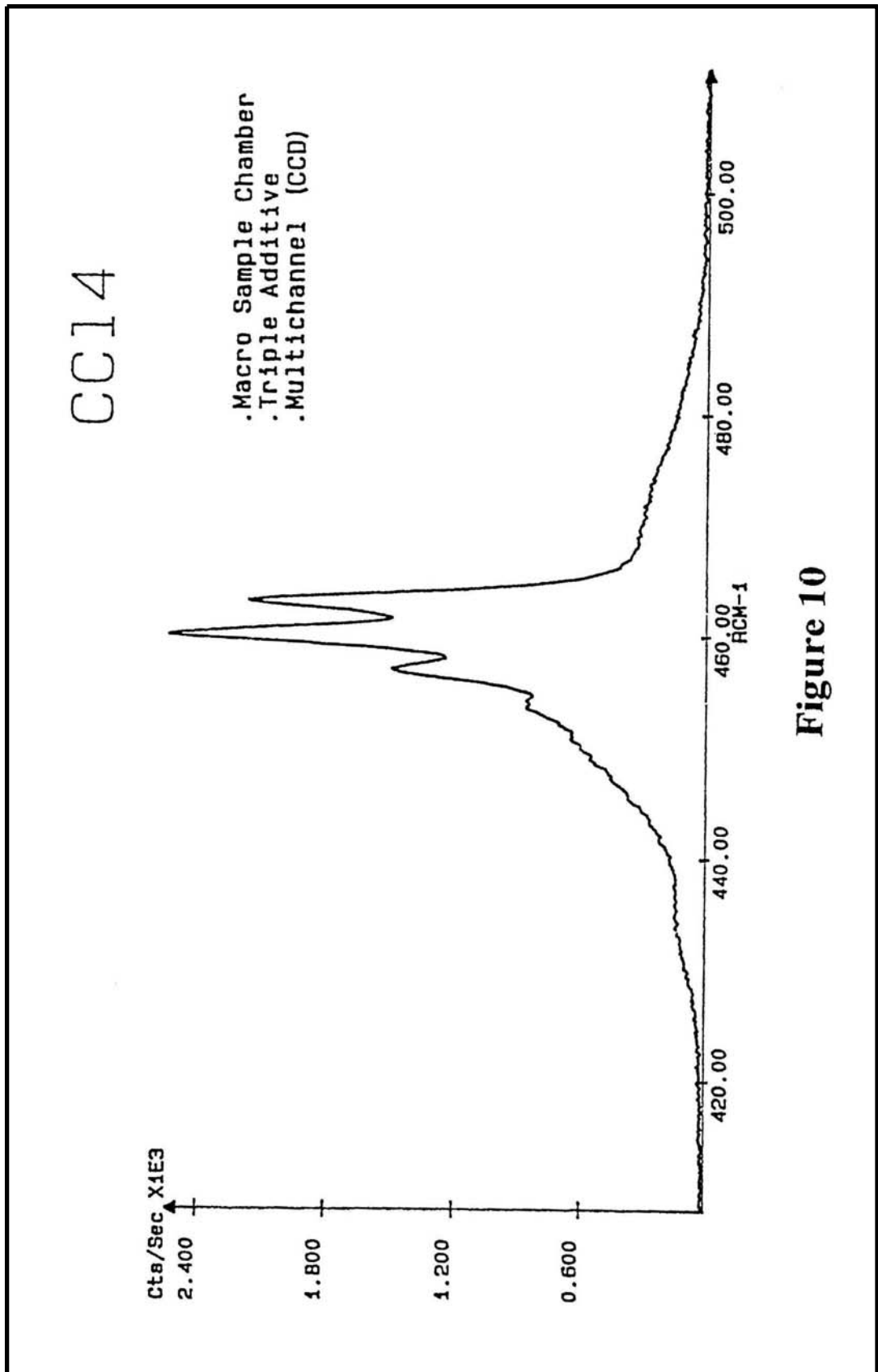


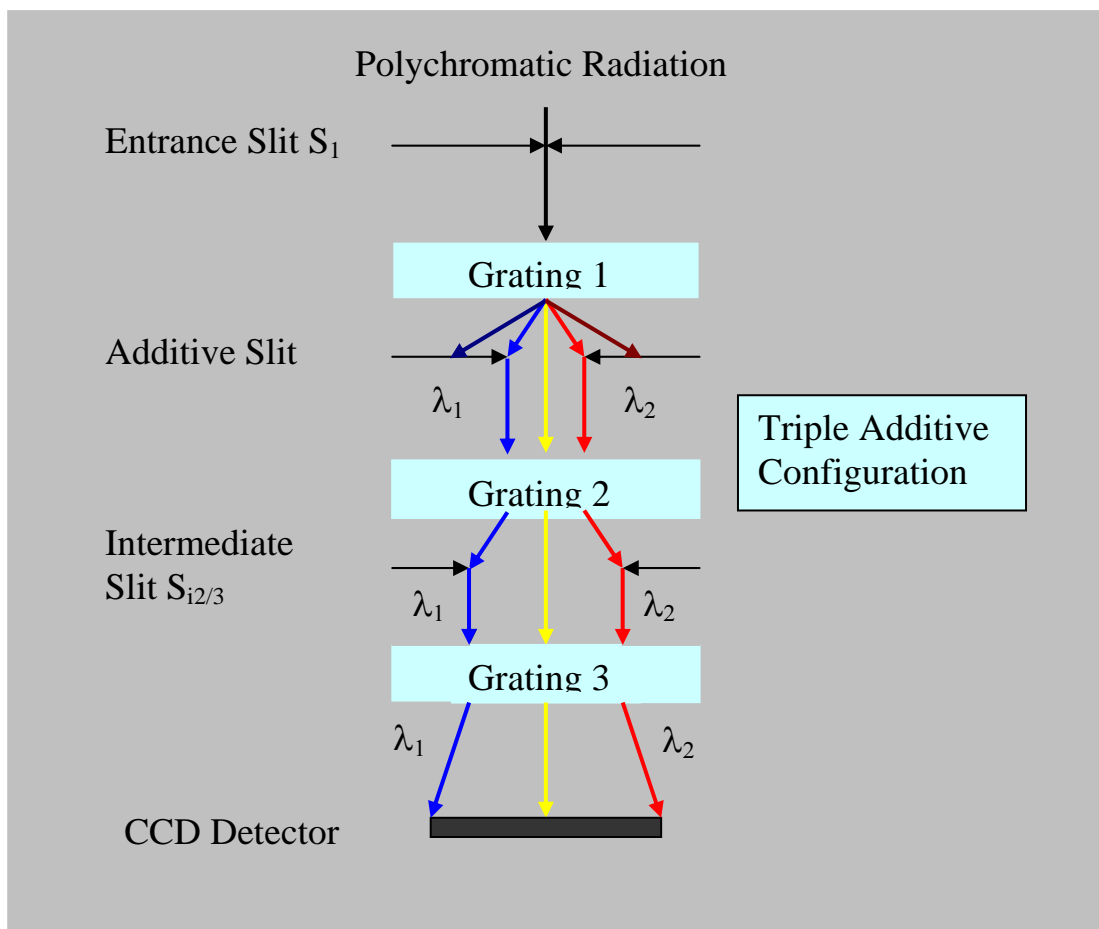
Figure 10

**1.2. Triple additive configuration: basic configuration+additive option**

**1.2.1. Optical diagram (Figure 11)**

**1.2.2. Function of the triple additive spectrograph or spectrometer**

The triple additive configuration permits to obtain a very high resolution. In the triple subtractive configuration, only the 3<sup>rd</sup> stage is used as a disperser. In the triple additive configuration, each stage contributes to disperse light. The triple additive dispersion is equivalent to that of a spectrograph of 1.92m focal length while for the triple subtractive, it is equivalent to only a focal length of 0.64m. (See following diagram)



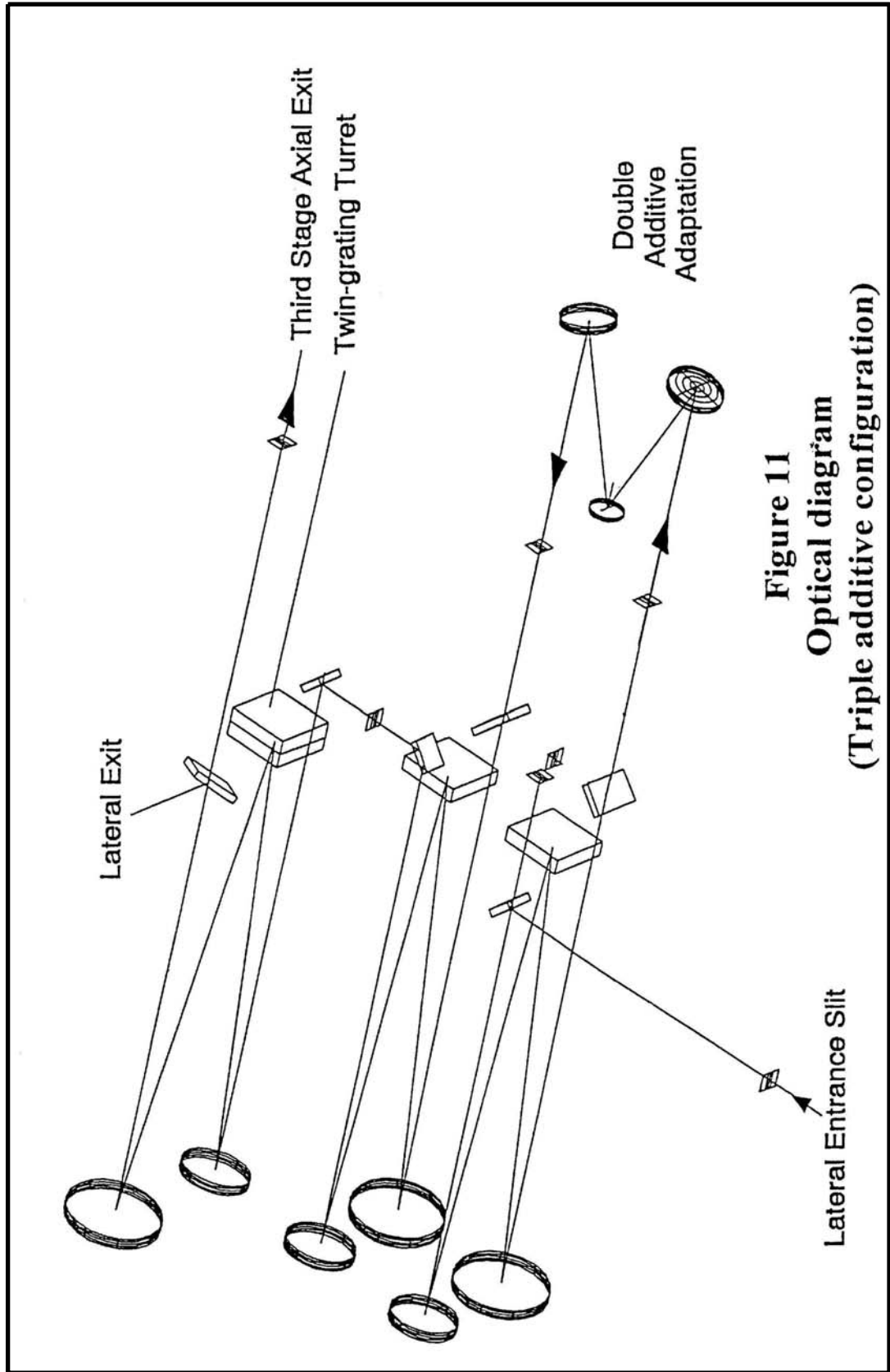


Figure 11  
Optical diagram  
(Triple additive configuration)

**1.2.3. General specifications**

1.2.3.1. Resolution

With a CCD detector, the resolution with narrow slits is always better than 2 pixels, while with PMT detector, it will be better than 0.2cm-1.

1.2.3.2. Other specifications

Concerning the other specifications like spectral range accuracy, repeatability, stray light rejection, ... , it is the same than those of the basic configuration.

**1.3. Practical limits of use with the triple system**

**1.3.1. With multichannel detectors IPDA or CCD**

When a multichannel detector is used in the triple additive configuration, only the entrance slit  $S_1$  is narrow, defining the resolution and all the other intermediate slits should be widely opened for the largest coverage on the CCD. Under these conditions, no filtering function exists in the system to reject the stray light coming from the exciting line and the analysis in the low frequency range is practically impossible.

It is necessary to remember that a monochromator has two functions: to disperse and to image. That means that the images of all the slits in the triple additive configuration are superimposed on the multichannel detector and located in the low frequency range. Because of the high level of Rayleigh scattered light in these slits, their images show then a high level of stray light and will not permit to record a spectrum closer than the largest bandpass defined by the slits. Then, a low frequency Raman spectrum can only be acquired by reducing the intermediate slit width but then the coverage on the CCD detector will also be proportionally reduced.

Remark:

For acquiring a large spectral coverage on the CCD detector down to the low frequency range, we recommend to use a Super Notch or Super Notch Plus filter, placed on the scattered beam to attenuate the Laser line.

Another important point is to limit the number of spectral windows to be glued. We recommend, at maximum, 4 or 5 windows so that we are sure that the background of the spectrum or a broad fluorescent band is real without any distortion due to the gluing operation.

**1.3.2. With monochannel detectors PMT or any other**

Generally, with the T64000, the monochannel detectors are useful only in four cases:

- A. Acquisition of a spectrum with a high resolution in the very low frequency range
- B. Acquisition of a spectrum in the range where the efficiency of the multichannel detectors drops down. For example, photoluminescence analysis in the NIR or IR range.
- C. Acquisition a large range spectrum. In this case, the entrance and the exit slits are narrow and the other intermediate slits must be a little more wider to eliminate the possible small wavelength difference between the two scanning mechanisms.
- D. Acquisition a very weak signal over a very intense background which would saturate the A/D converter of a multichannel detector, thanks to the much higher dynamic range of a monochannel detector over that of a multichannel detector.

**1.4. Single option with an holographic notch filter**

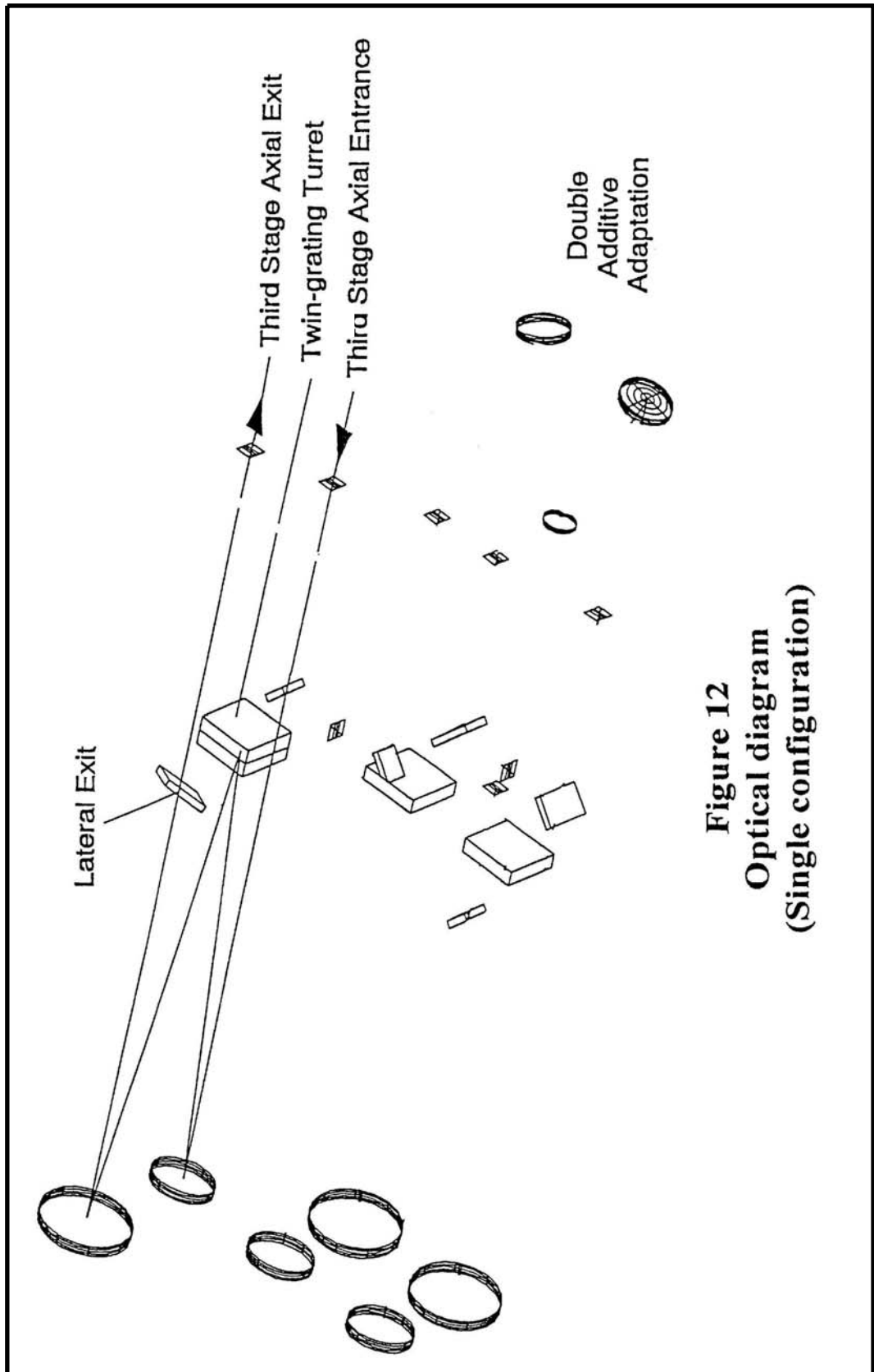
**1.4.1. Optical diagram (Figure 12)**

**1.4.2. Function of the single spectrograph or spectrometer**

The T64000 system offers a very interesting single spectrograph / spectrometer option for the analysis of innumerable samples in a short time and with a high detectivity. When the low frequency range is not required, the single spectrograph option with its high throughput, about 3 times higher than that of the triple system, equipped with an holographic notch filter must be used, specially when the signal is weak. If the spectrograph is equipped with a 2-gratings turret, the detectivity can again be increased by changing from the high dispersing grating to the low dispersing one. (See paragraph 1.1.4.3.)

**1.4.3. Holographic notch filter**

For a good use of the single spectrograph option an holographic notch filter is necessary for filtering the exciting line. Such a filter is not as good as the subtractive foremonochromator but when the low frequency range is not required, the filter is sufficient. A notch filter is specific for one wavelength. Changing the excitation wavelength requires changing the notch filter. Many types of filters exist, notch, notch plus, super notch, super notch plus. The difference between them concerns the laser attenuation and the spectral bandwidth. The table in the Figure 13 summarizes their specifications.



**Figure 12**  
**Optical diagram**  
**(Single configuration)**

## Holographic Notch Filters for Laser Spectroscopy Applications

Specifications	Notch	Notch-Plus	SuperNotch	SuperNotch-Plus
<b>Laser attenuation</b> Optical Density	> 4.0	> 6.0	> 4.0	> 6.0
<b>Spectral Bandwidth</b> .Wavenumbers between O.D.0.3 or 50% points .Bandwidth between O.D.0.3 or 50% points for 532nm filter	< 700cm-1 < 20nm	< 700cm-1 < 20nm	< 350cm-1 < 10nm	< 350cm-1 < 10nm
<b>Spectral Edgewidth</b> .Wavenumbers between O.D.0.3 and O.D.4 points .Edgewidth between O.D.0.3 and O.D.4 points for 532nm filter	< 300cm-1 < 9nm	< 300cm-1 < 9nm	< 150cm-1 < 4nm	< 150cm-1 < 4nm
<b>%Transmission outside notch</b> 475nm to 1500nm Drops off in UV range	> 80%	> 80%	> 80%	> 80%
<b>Wavelength range</b>	350nm to 1064nm	350 to 1064nm	488nm to 1064nm	488nm to 1064nm

**Figure 13**

## 1.5. Maintenance

### 1.5.1. Calibration and coupling

Now and then, the system must be verified, recalibrated and recoupled or when the current switches off. Also, the recalibration of the system is necessary for the good running of the instrument. For this operation, see Annexes.

### 1.5.2. Temperature effect

As seen before, a good repeatability of the system depends strongly on the temperature stability. An important variation of temperature will affect the results of the analysis, because in this case, the system can be decoupled. It is absolutely necessary that the laboratory is equipped with an air conditioner to reduce this temperature variation.



## 2. Macro sample chamber

### 2.1. General characteristics

#### 2.1.1. Optical diagram (Figure 14)

#### 2.1.2. Variable incidence angle

Until now, most of the Raman macro sample chambers offered only the right angle scattering and backscattering analysis. The new macro sample chamber of the T64000 was designed to offer more possibilities with its variable incidence angle to analyze any kind of sample with the best stray light rejection, stray light coming usually from the direct reflection of the laser beam into the monochromator. Naturally, the right angle scattering and the back scattering are the two limit positions offered by this attachment.

#### 2.1.3. Interchangeable optics

The new design allows to interchange all the optics present in this attachment, laser focusing and collecting optics responding to all the requirements of the experiment for obtaining the best results. How can we do that? Between the sample and the entrance slit, instead of a fixed objective normally used in a macro chamber to collect the scattered light, we have employed a two lens afocal system. The first lens L9 near the entrance slit, is fixed and its focal length corresponds to the distance L9 - Slit (260mm). The second L8, near the sample is interchangeable and its focal length corresponds to the distance L8 - Sample. Depending on the dimensions of the samples or of the accessories, a focusing or collecting lens, with a short or a long focal length can be selected for optimizing the results of the experiment.

Theoretically, the diameter  $d$  of the laser beam at the focus point is given by the formula:

$$d = 4\lambda F_f / \pi D$$

where  $\lambda$  is the excitation wavelength

$F_f$  is the focal length of the focusing lens

and  $D$  is the diameter of the incident laser beam

From this formula, we can see that a focusing lens with a short focal length gives a smaller laser spot at its focus point.

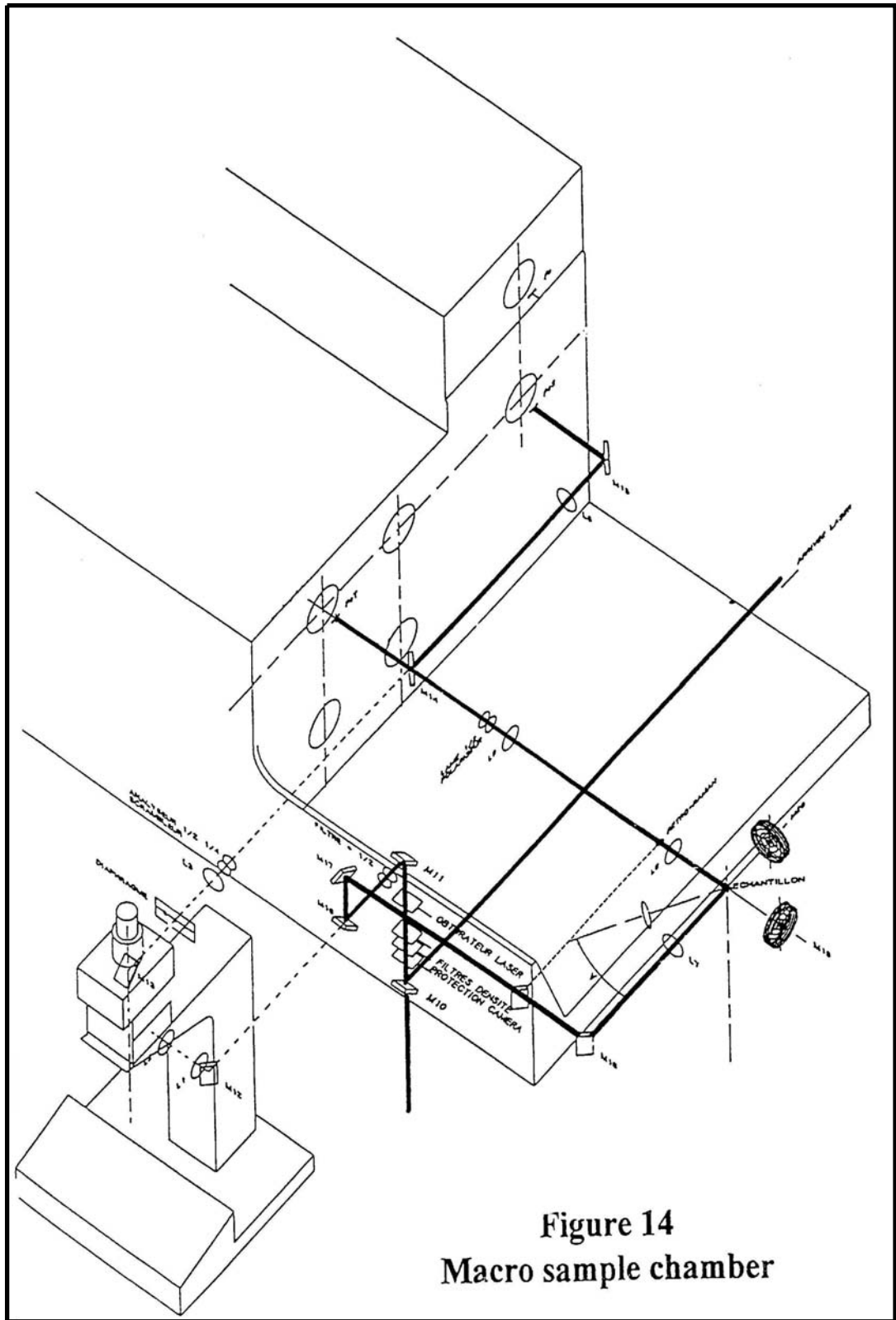


Figure 14  
Macro sample chamber

For solid samples which depth penetrations are short, like semi-conductors or black compounds, using a focusing lens with a short focal length improves the spatial resolution.

For liquid or gas samples, a focussing lens with a long depth focus, increasing the sample volume to be analyzed, is more appropriate.

The choice of the collecting lens depends strongly on the focusing lens and on the spectral resolution which required.

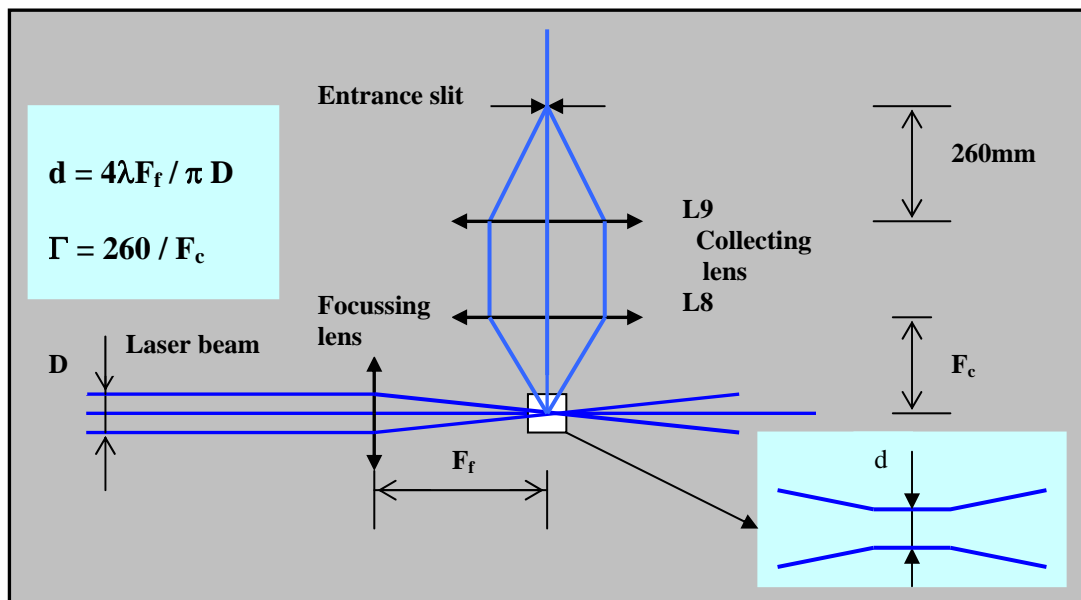
The magnification of the collecting optics is defined by:

$$\Gamma = \text{Distance(L9 - Slit)} / \text{Distance(L8 - Sample)}$$

$$= 260 / F_c \text{ (mm) where } F_c \text{ is the focal length of the lens L8}$$

A collecting lens with a short focal length has a higher collection solid angle and then collects a higher flux. But as the magnification will be higher with a short focal length than with a long focal length, it will be necessary to make a choice between:

- Either making the whole flux collected enter the monochromator by opening wider the entrance slit of the spectrometer which consequence will be that the spectral resolution is decreased.
- Or maintaining the spectral resolution, keeping the entrance slit narrow but reducing the flux entering the monochromator and then the signal to be detected.

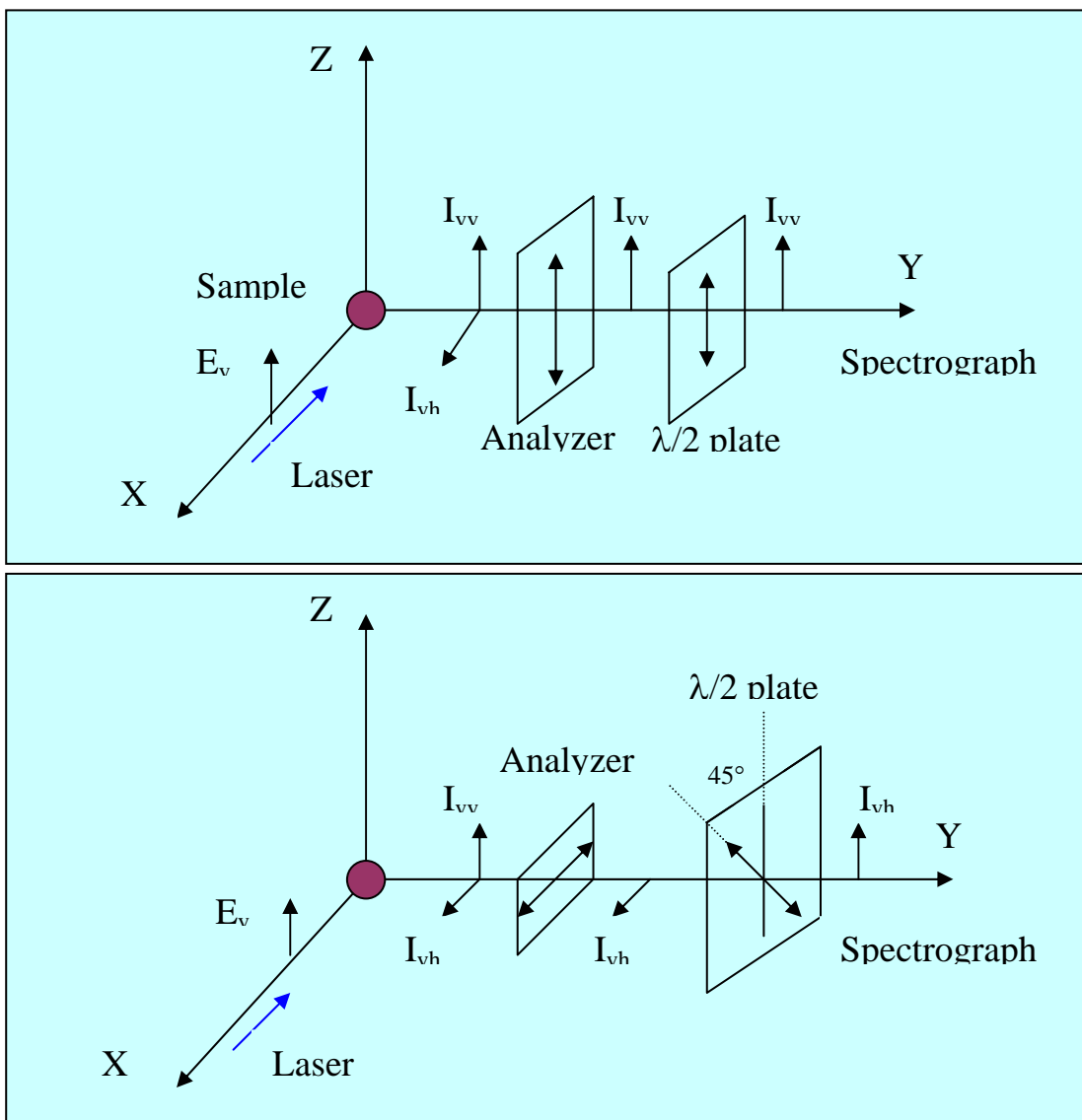


Recommendations for a good use of interchangeable optics

- For solid samples:  $F_f = 30\text{mm}$  and  $F_c = 30\text{mm}$
- For liquid or gas samples:  $F_f = 100\text{mm}$  and  $F_c = 80\text{mm}$
- For samples which are placed in big accessories, cryostat, furnace,...  
 $F_f = 150\text{mm}$  or  $200\text{mm}$  (depending on the distance to the sample) and  
 $F_c = 160\text{mm}$

**2.1.4. Polarization**

All polarization measurements are available with the macro sample chamber. On the laser beam, a half wave plate permits to change the polarization direction. On the scattered beam, an analyzer permit to select one component and a half wave plate or a quarter wave plate corrects the polarization effect introduced by the gratings on the efficiency. (See Figures 2 & 3)



A half wave plate rotates a linear polarization and a quarter wave plate or scrambler transforms a linear polarization to an elliptic or circular polarization. As shown in the Figures 2 & 3, the maximum of the efficiency of the grating is obtained when the linear polarization is perpendicular to the grooves of the grating for all the wavelengths higher than 460nm. For this reason, we recommend mostly to use a half wave plate for selecting this polarization direction. For all the wavelengths lower than 460nm, the polarization parallel to the grooves of the grating must be selected.

## **2.2. Maintenance**

### **2.2.1. Realignment of the macro sample chamber**

Sometimes, you will observe that the laser beam is not correctly aligned in the macro sample chamber. Before realigning it, let verify that the laser beam from the laser head to the mirror, which reflects it to the sample chamber, is in place. If it is, realign the macro sample chamber following the procedure indicated in the Annexes. If it is not, certainly the laser has been moved. A complete realignment is necessary from the laser to the sample chamber

### **2.2.2. Cleaning optical components**

For the good working of the system, you must check regularly the cleanness of different optical parts, specially that of the interferential filter, the half wave plate, the focussing and collecting optics, the analyzer ... These components can be cleaned with alcohol and soft cotton.

## **2.3. Training**

### **2.3.1. Acquisition of spectra of liquid or gas samples**

- Influence of slit width, slit height
- Comparison of results using different optics
- Polarization measurements
- Optimal acquisition parameters
- CCD use

### **2.3.2. Acquisition of spectra of solid samples**

- Influence of slit width, slit height
- Comparison of results using different optics
- Polarization measurements
- Optimal acquisition parameters
- CCD use

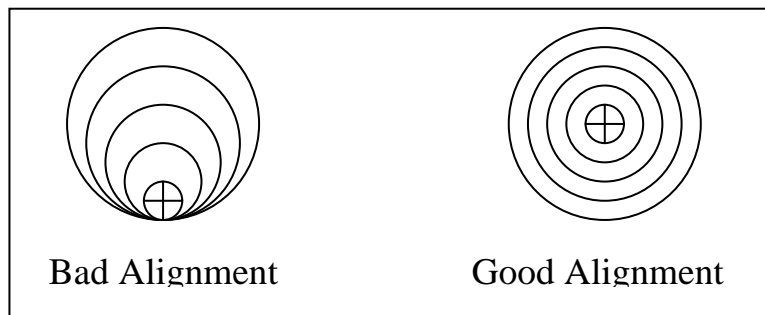
### 3. Confocal microscope

#### 3.1. General characteristics

##### 3.1.1. Optical diagram (Figure 15)

##### 3.1.2. Laser illumination

As shown on the optical diagram, the laser can be installed at the backside or at the bottom of the instrument. During the installation, the laser is aligned by a Jobin Yvon engineer to provide a good illumination on the sample. The laser spot is located on the TV monitor screen by a landmark. The image of the spot stays circular and concentric around the landmark when it is out of focus. (See following diagram)



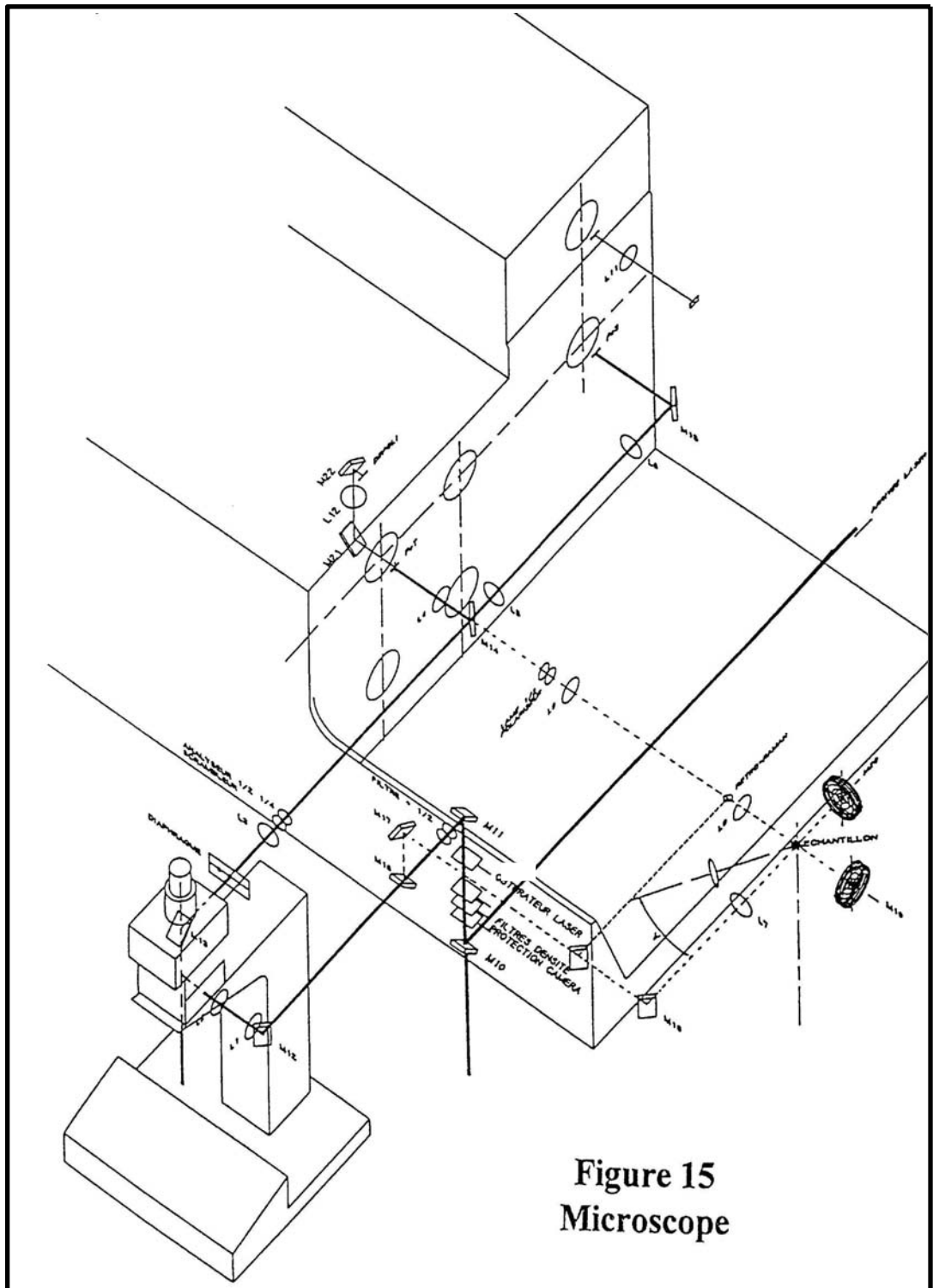
This illumination must be checked every time you change the laser line or if some parts between the laser and the T64000 are unstable.

If the laser illumination on the sample is not perfect, you can realign it, adjusting the two mirrors before the beamsplitter (for example  $M_{11}$  and  $M_{12}$ )

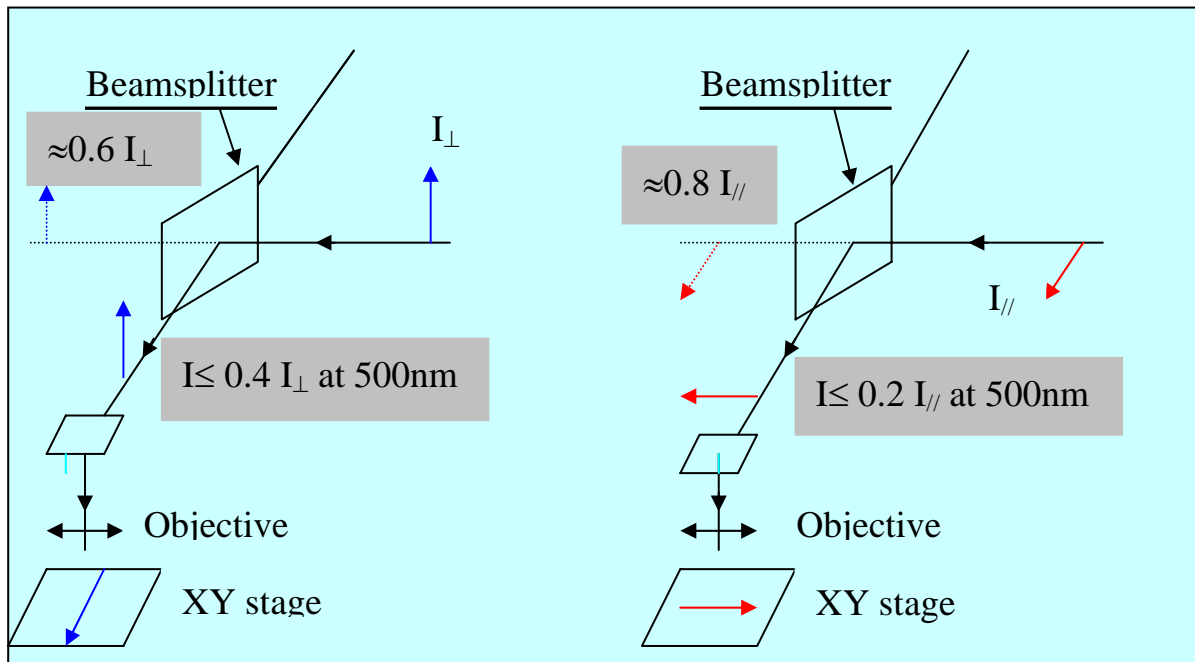
##### 3.1.3. Polarization

All polarization measurements are available with the microscope. On the laser beam, a half wave plate permits to change the polarization direction. On the scattered beam, an analyzer permits to select one component and a half wave plate or a quarter wave plate corrects the polarization effect introduced by the grating on the efficiency. Inside the microscope, there is a beamsplitter with a special coating which transmission and reflection factors depend on the polarization (see Figure 16). Instead of changing the polarization of the incident laser beam, we recommend to turn the sample by  $90^\circ$  to be sure that the laser power remains the same for the measurement. For the exact depolarization ratio determination, some correction is necessary because of the transmission dependence of the polarization.

The following diagrams explain this difference.

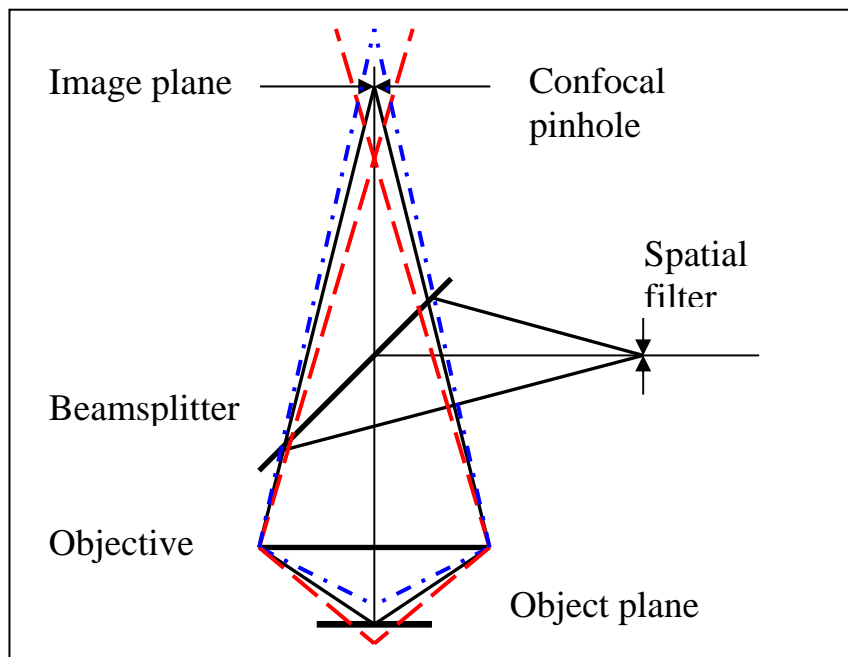


**Figure 15**  
**Microscope**

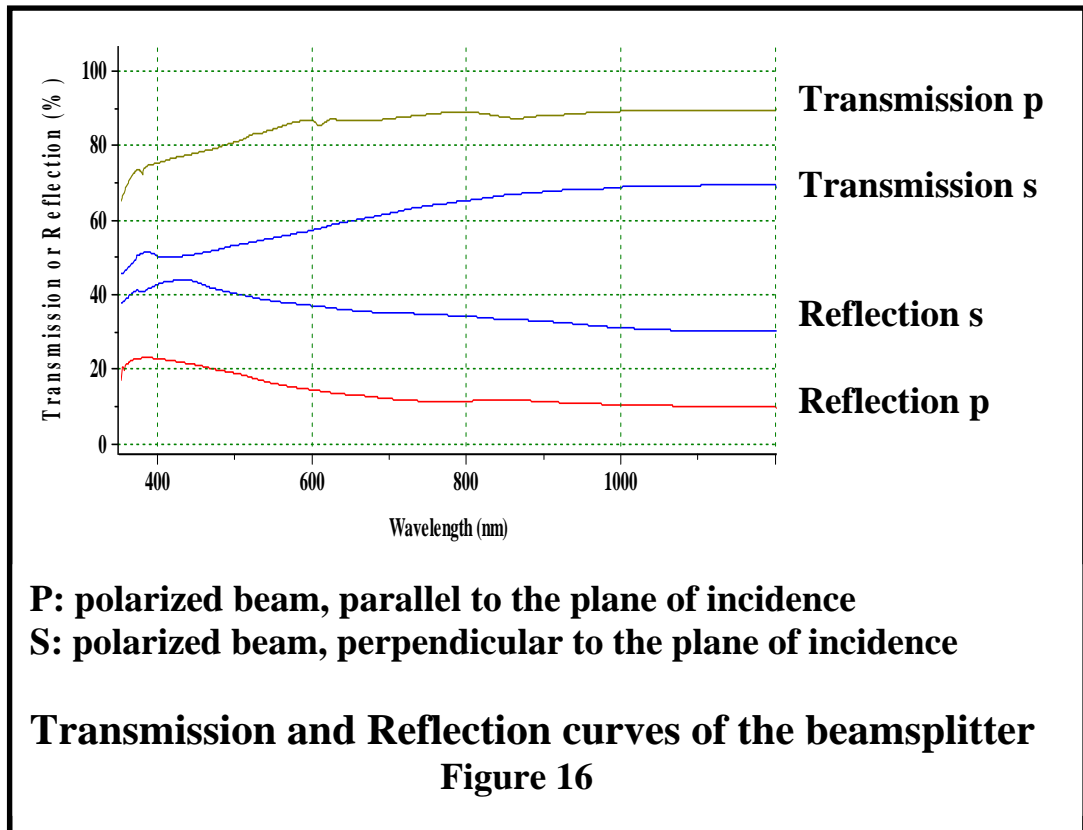


**3.1.4. Confocal advantages**

A confocal microscope increases the spatial, lateral and depth resolutions to the diffraction limits. The confocal attachment consists in two spatial filters. The first, placed on the incident laser beam, purifies the laser beam providing a perfect gaussian distribution of the laser. The second, placed on the image plane of the microscope, limits the analyzed surface of the sample and its depth focus. The two spatial filters and the focus plane of the microscope objective are perfectly conjugated with a correct aperture adaptation.







Many applications require this confocal property for obtaining significant results such as:

- Determination of the properties of multilayers or monolayers versus depth into the material.
- Multilayer foils
- Strained polymers (PE fiber)
- Local stresses or degradations
- Elimination of a matrix Raman scattering or fluorescence

### 3.1.5. Video camera protection

The sample can be illuminated simultaneously with white light and the laser. Because of the strong luminosity of the laser line, which can create saturation or blooming of the camera, and prevents the observation of the sample image, we are using a protection device. It consists in three neutral density filters (O.D. 4, 1.3, 0.7) mounted on the path of the laser beam to reduce this luminosity and provide an observation of the sample illuminated with white light, with the location of the laser spot.

The O.D. 4 filter is automatically in position when the light is transferred towards the camera. The two other filters, O.D. 1.3 and O.D. 0.7 can be controlled manually by switches.

## 3.2. Maintenance

### 3.2.1. Realignment

If the laser spot is not centered on the landmark and its image is not circular and concentric on it when it is out of focus, you must realign the laser beam. The realignment can be done by adjusting two mirrors before the beamsplitter and only these two mirrors (for example mirrors  $M_{11}$  and  $M_{12}$ ), if the misalignment is not very important. If the misalignment is total, a complete realignment from the laser head to the center of the mirror  $M_{11}$  is necessary and also from this mirror to the sample.

### 3.2.2. Cleaning optical components

For the good working of the system, you must check regularly the cleanness of different optical components, specially that of the interferential filter the haft wave plate, the objectives of the microscope, the analyzer ... These parts can be cleaned with alcohol and soft cotton.

## 3.3. Training

### 3.3.1. Acquisition of spectra of solid samples

- Influence of slit width, slit height
- Influence of different objective magnifications
- Polarization measurements
- Influence of confocal pinhole size
- Optimal acquisition parameters
- CCD use

### 3.3.2. Acquisition of spectra of liquid samples

In the case the instrument is not equipped with a macro sample chamber, liquid samples can be analyzed with the microscope. Use for this operation a low magnification objective to take benefit of the long depth focus. Naturally, the confocal pinhole diameter must be the widest possible.

## 4. Detectors

### 4.1. Multichannel detectors

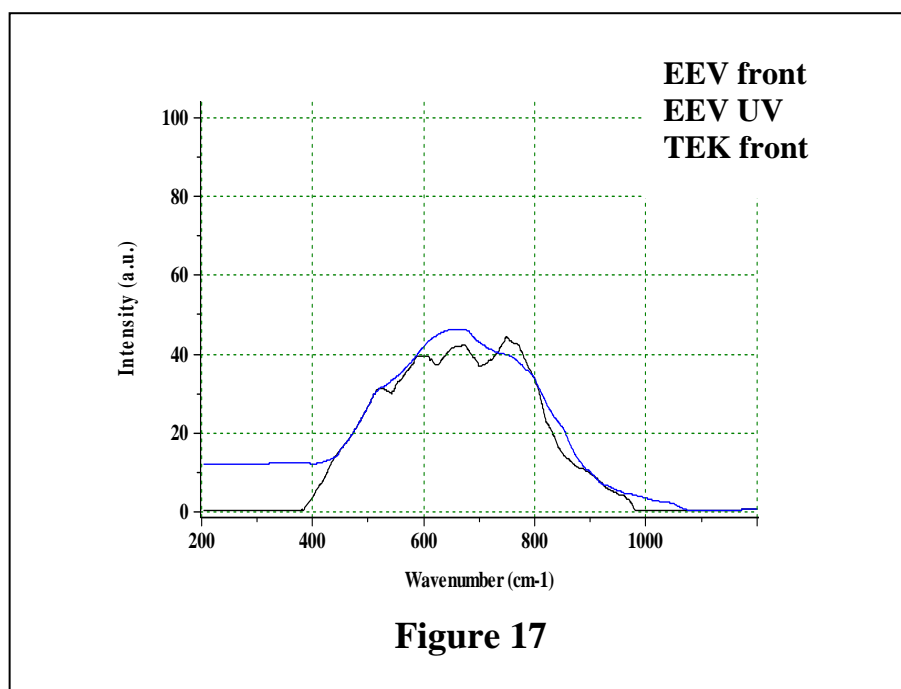
#### 4.1.1. CCD (Charge Coupled Device)

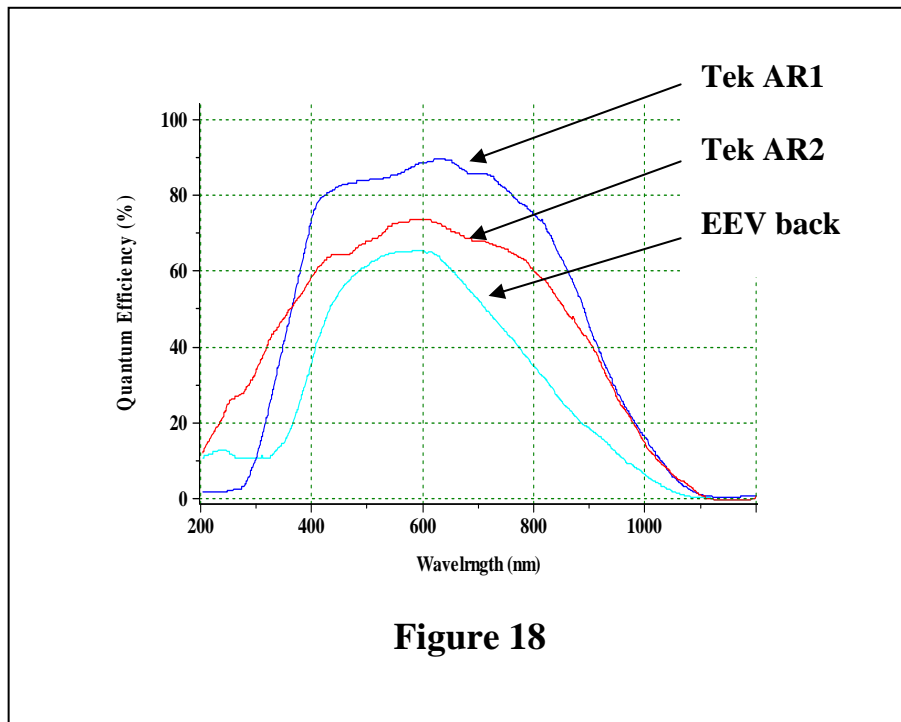
Various models of CCD detectors are available and manufactured by Jobin Yvon S.A. with difference in the number and the size of the pixels. For example :

- 1024 x 256 pixels pixel size : 26 $\mu$ m x 26 $\mu$ m
- 2000 x 800 pixels pixel size : 15 $\mu$ m x 15 $\mu$ m

They can be UV coated to enhance the efficiency in the spectral range 200nm to 450nm. Back illuminated CCD detectors provide again a higher quantum efficiency compared to the standard front illuminated CCD detectors. However, due to their "etalonning" effect in the NIR range, we do not recommend BI chips for measurements above 750nm. (See Figures 17 & 18) For the T64000 system, LN<sub>2</sub> CCD detectors, cooled by liquid nitrogen down to 140K, are selected to offer the highest performance to the system. A 3l Dewar allows to hold the low temperature at least during 3 days.

Other TE CCD models, air or water cooled by Peltier effect down to about 215K are also available for other instruments, together with Open Electrode and Deep Depleted chips. (Go to specific CCD leaflets for more information on these products)





The adjustment of the CCD concerning the focus and the orientation of the chip parallel to the entrance slit is very important for the good running of the system. See in Annexes "Procedure for the readjustment of the CCD detector".

Remarks on the origins of noises

Generally, we can consider there are three types of noises:

- photon shot noise
- thermal noise
- readout noise

The photon noise is proportional to the square root of the signal intensity.

The thermal noise depends on the level of the dark current. With LN<sub>2</sub> CCD, the dark current is extremely low (less than 1 electron/ pixel/ hour)

So this noise is practically negligible with LN<sub>2</sub> CCDs.

The readout noise is quite the same for different electronics (between 6 to 10 electrons/ pixel). This readout noise is generally the most important noise with the LN<sub>2</sub> CCD detectors.

Because the dark current is very low and the readout noise is high, we are always recommending, for spectrum acquisition, to select at first a long integration time without saturating the A/D converter but not a short integration time with a great number of accumulations. The accumulation is only used for improving the signal to noise ratio when samples are

fluorescent or present a high background that is limiting the integration time.

**4.1.2. IPDA (Intensified Photo Diode Array)**

Since the introduction of the CCD detectors, IPDA, first generation of multichannel detectors are practically no longer used in Raman spectroscopy because of their high dark current. However, gatable IPDA detectors are still used for some specific applications, like time resolved spectroscopy, fast acquisition...

Note that ICCD (Intensified CCD) detectors are now available.

**4.2. Monochannel detectors**

**4.2.1. PMT (PhotoMultiplier Tube)**

As explained previously, the use of a PMT detector on the T64000 should be particularly limited to only a few applications. (See paragraph 1.3.2.)

**4.2.2. Infrared detectors**

Any kind of infrared detectors can be adapted and mounted on the axial exit of the third spectrograph/spectrometer stage for recording spectra like luminescence ones. Evidently, the gratings have to be changed and selected for their highest efficiency in the range of analysis.

**4.3. Training**

**4.3.1. Use of CCD detector**

(Cf. paragraphs 2.3. and 3.3.)

**4.3.2. Use of PMT detector**

- Low frequency range with triple additive configuration
- Acquisition of a large range spectrum. For example, white light spectrum from 400nm to 900nm.
- Special instrumental conditions.

## **5. Electronic hardware & Software**

### **5.1. Electronic hardware**

#### **5.1.1. Spectralink**

The spectralink includes many electronics cards according to the configuration of the system.

- PS card, general power supply card.
- MDR cards (2 to 4) for the control of the motors of the 2 monochromators and of the 2 intermediate slits.
- INT card, interface card
- ACQ card for the acquisition of the signal with monochannel detectors.
- HV card for the monochannel detectors.
- PIO-COM card,

#### **5.1.2. Computer**

Because of the rapid evolution of computer manufacturing, we will not indicate the minimum configuration required today for working with the new software Labspec.

#### **5.1.3. CCD equipment**

The multichannel detection system is delivered with a CCD head, installed on the instrument, a controller, one or two CCD shutters, a setup disk, and cables to interconnect CCD head, controller and shutters.

NB : Previously, CCD detectors equipped with a CCD2000 controller were using a RISC board installed in the computer.

#### **5.1.4. PIO-COM & COM cards**

These cards permit to control all the different motorized commutations, like:

- Subtractive / Additive
- Triple / Single
- Motorized 2-grating turret
- Multichannel / Monochannel
- Macro / Micro / Triple / Single commutation wheel
- Axial / Lateral entrance of the foremonochromator
- Axial / Lateral entrance of the spectrograph

## **5.2. Software**

### **5.2.1. General concept**

The new LabSpec software is a completely integrated package of programs and special routines for the instrumental control and for the acquisition and treatment of spectroscopic data and images. This software allows the user to create, store, and modify at any time acquisition routines.

The LabSpec software was written to work with Windows 95, with Windows 98 or with Windows NT.

### **5.2.2. Instrumental control and Acquisitions**

The software controls all the functions of the T64000 system, including the move of the monochromators, of the commutations, and of the intermediate slits, the detectors with safety check and optimization of parameters, the XY motorized stage of the microscope, the confocal line scanning, ...

Different ways of spectra acquisition are possible, with spectrum adjustment mode or spectrum accumulation mode with multi-windows with or without automatic gluing. Video images and Raman or fluorescence images can be acquired.

#### **Spectrum acquisition**

The LabSpec software not only provides easy-to-use single spectrum adjustment and acquisition functions including multiwindows recording , but also allows time-dependent spectrum acquisition. Moreover it has the ability to control samples under process.

#### **Multiwindows automatic recording.**

Automatic recording and joining of spectral windows for full Raman recording.

Automatic recording of selected spectral windows.

Automatic adjustment of spectral intensity.

#### **Sample scanning.**

Single spectrum acquisition can be extended to image spectrum recording using a motorized stage controlled by the LabSpec software. This option opens a wide field of opportunities for sample surface analysis. A high resolution (0.1µm) XY stage is used as scanning device.

### 5.2.3. *Treatment*

#### Image mapping.

Different tools are available for image treatments. Three cursor pairs are used simultaneously to define spectrum bands to be displayed as Raman images. The basic image displays the integrated intensity between a pair of cursors on each scanned position to obtain a 2D image map of the selected line. Using the different pairs of cursors, three specific lines can be mapped at a time so that the presence of each compound can easily be determined on the surface.

#### Model fitting.

Another way to determine the composition of a sample consists in using mathematical models. A model spectrum of each compound can be extracted from the spectral image of the sample when each compound is found alone in the image map. If not, a single spectrum of the compound (recorded separately) can be used as the model. Using the mathematical model option, an image map is built for each model, computing the percentage of the compound presence in the mapped area.

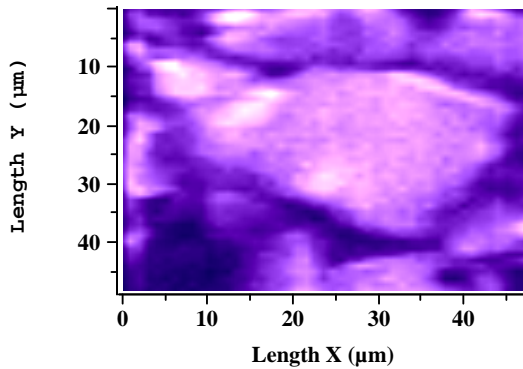
#### Peak fitting.

For some samples, it is helpful to fit a mathematical curve to the spectrum line. To do so, the LabSpec software provides a powerful peak fitting tool box. It allows the selection of numerous options for the curve, such as the type to be fitted (Gaussian, Lorentzian...), the computing time, etc. Then a wide range of peak characteristics can be displayed such as the peak shift, band amplitude, full width at half maximum, ratio between gaussian and lorentzian components, line surface. These functions are, for example, very useful for strained sample analysis, DLC samples...

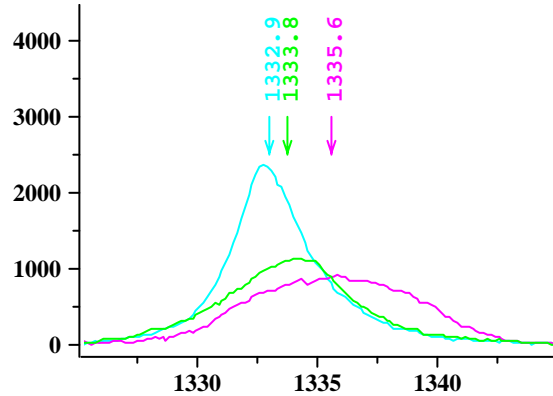


**Example with the study of the stress mapping in a microcrystal of diamond :**

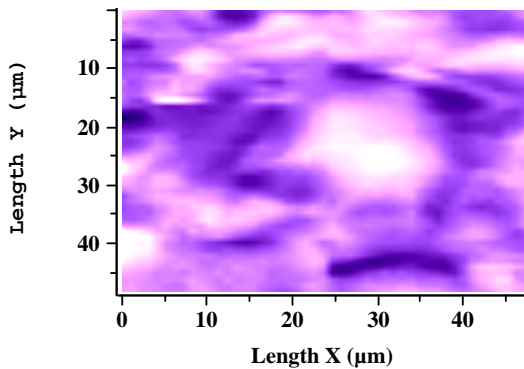
The very precise measurement of the band position and of the width of the diamond line informs on the stress to which is submitted the crystal.



*TV image*



*Particular spectra of the spectral image*

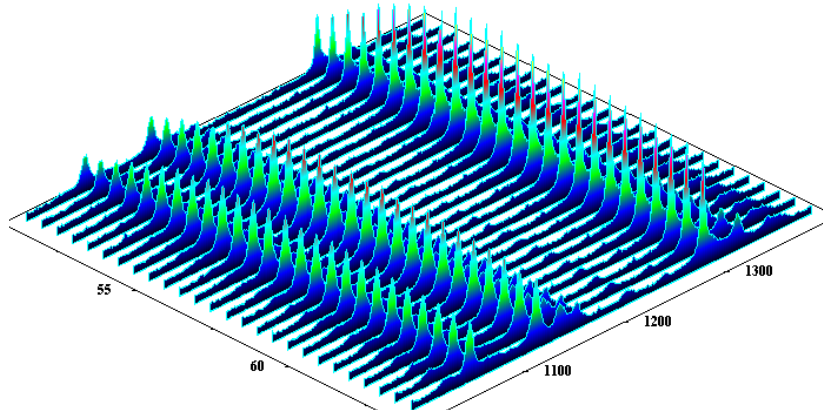


*Mapping of the position of the band. White color corresponds to the 1336 cm<sup>-1</sup> position, the purple one to the 1332 cm<sup>-1</sup> position*

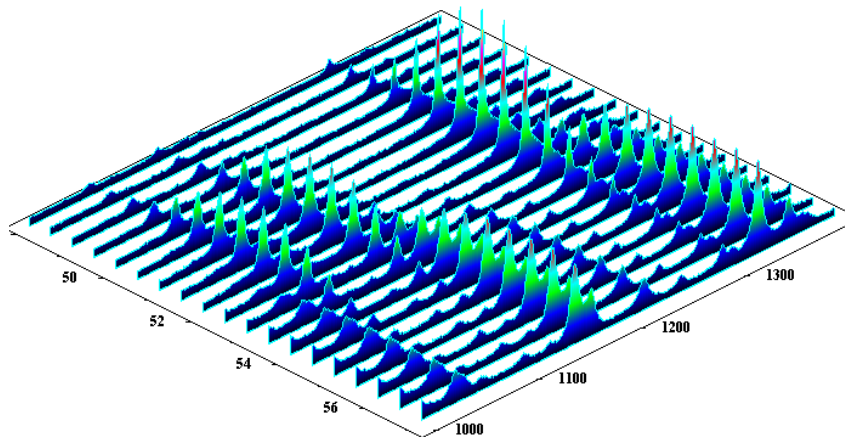
LabSpec also gives access to other treatments options such as:

- Mathematical operations.
- Baseline correction
- Smoothing
- Integral calculation
- Fourier filtration
- Mathematical correction

3D representation of spectra.



An easy way to monitor spectral shifts and shape bands.



All the spectra can be transferred in the other Windows programmes like Words... this is very useful for writing reports or articles.

This software is delivered with a user manual but a « help on line » is also available.

Datafiles can be stored in different formats, .tvf, .spc, .txt,...

## **6. Options & Accessories**

### **6.1. For the foremonochromator**

#### **6.1.1. Additional entrance and exit slits**

Basically, the axial entrance slit on the fore monochromator is reserved for the standard equipments of the T64000, like macro sample chamber and microscope. A 2<sup>nd</sup> entrance, lateral, is proposed for special equipments, for example, an optical fiber accessory or a source, ...

An additional axial entrance slit on the spectrograph is necessary for the use of the single option. (See paragraph 1.4.)

The double foremonochromator can be equipped with an axial exit slit for using a PMT in double additive configuration or for fluorescence analysis. Finally, an axial exit slit on the spectrograph permits to use any kind of monochannel detector. (See paragraph 4.2.)

#### **6.1.2. Viewer**

With the microscope, incident and scattered optical axis are already fixed. The operator has just to place a sample under the objective of the microscope and brings it to focus with the help of the TV camera and then makes an acquisition.

With the macro sample chamber, the adjustment of the optics and of the sample are more complicated. For that reason, on option, a viewer is proposed to facilitate this operation. The viewer, placed behind the entrance slit, intercepts the scattered light, reflects and focuses it on a screen or a TV camera, assuring a correct global adjustment. A final and perfect adjustment must be done with a signal in order to obtain the maximum of intensity.

This accessory is available under two versions:

- Viewer supplied with a viewing screen
- Viewer supplied with a C mount adapted to most color or B/W video cameras. Included in this adaptation, are three neutral density filters of O.D. 4, 1.3, and 0.7 for attenuating the laser beam.

#### **6.1.3. Additional gratings**

Jobin Yvon S.A manufactures a large range of gratings with different grooves densities, and different blaze wavelengths. Depending on the application and the spectral range of work, we can recommend the best solutions. Please contact us for a right proposal.

#### **6.1.4. 2 Gratings-Turret in the 3<sup>rd</sup> stage**

A 2 gratings-turret in the 3<sup>rd</sup> stage is advantageous for several reasons:

- It offers the possibility to change rapidly the dispersion to get a large spectral coverage and to increase the detectivity when using the single configuration. (See paragraph 1.4.)
- It permits to do Raman and Photoluminescence measurements on the same sample, changing from high to low dispersing gratings.

#### 6.1.5. Motorized commutations

All the commutations, which are inside the monochromator, are motorized assuring a perfect repeatability. These commutations are the following:

- Subtractive / Additive  
Change from double subtractive to double additive configuration by moving away two mirrors. (See Optical Diagrams in Figures 1 & 11)
- Multichannel / Monochannel  
Change the exit direction in the spectrograph by moving away a mirror. (See Figure 1)
- Triple / Single  
Change the entrance direction in the spectrograph by moving away a mirror. (See Figure 12)
- Axial / Lateral entrance slit on the foremonochromator  
Change the entrance direction in the foremonochromator by moving away a mirror.

#### 6.1.6. Motorized or manual commutations

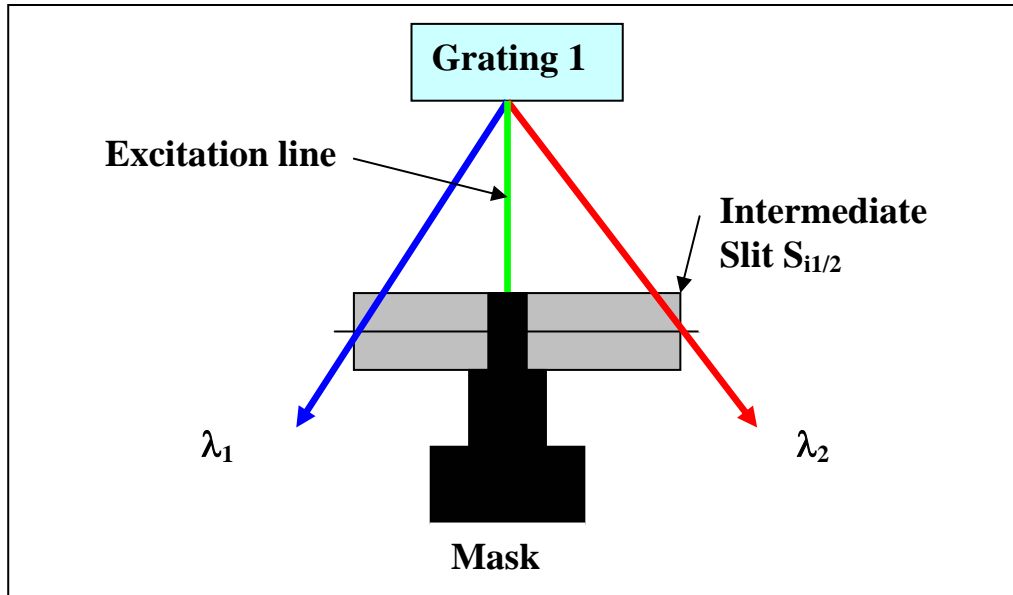
All the commutations which are outside the monochromators are manual but can be motorized on option.

- Macro / Micro  
This commutation permits to send the incident laser beam to the macro sample chamber or to the microscope if the instrument is equipped with the two sample attachments.
- 4-Positions wheel  
This commutations permits to work under all the different configurations: Macro / Triple, Macro / Single, Micro / Triple, Micro / Single, if the instrument is equipped with the two sample attachments and the single option.

#### 6.1.7. Stokes & AntiStokes measurement option

Simultaneous Stokes and AntiStokes measurements are possible on the T64000 with an accessory mounted on the intermediate slit  $S_{i1/2}$ . Naturally this function can be performed only in triple subtractive configuration. This accessory, composed of several masks with different sizes to stop the excitation line, offers the possibility to acquire simultaneously a Stokes

and Antistokes spectrum close to about  $25\text{cm}^{-1}$  from the Rayleigh line. Figures 19, 20 & 21 show Stokes and Antistokes spectra of  $\text{ZrO}_2$ ,  $\text{Bi}_2\text{O}_3$  and Air. The following diagram shows the principle of the device.



### 6.1.8. Special coating

Basically, all the optical components of the T64000 system are Al-coated giving a quite flat response over a large spectral range, at least from 200nm to more than  $1\mu\text{m}$ . Other coatings, Ag, Au, ... , are available on request. (See Figure 22)

## 6.2. For macro sample chamber

### 6.2.1. Focusing and collecting optics

As explained in the paragraph 2.1.3, all the optics, laser focusing and collecting lens in the macro chamber, are interchangeable. The sample compartment is delivered, in standard, with a glass focusing optics  $F=100\text{mm}$  and a glass collecting optics  $F=80\text{mm}$ .

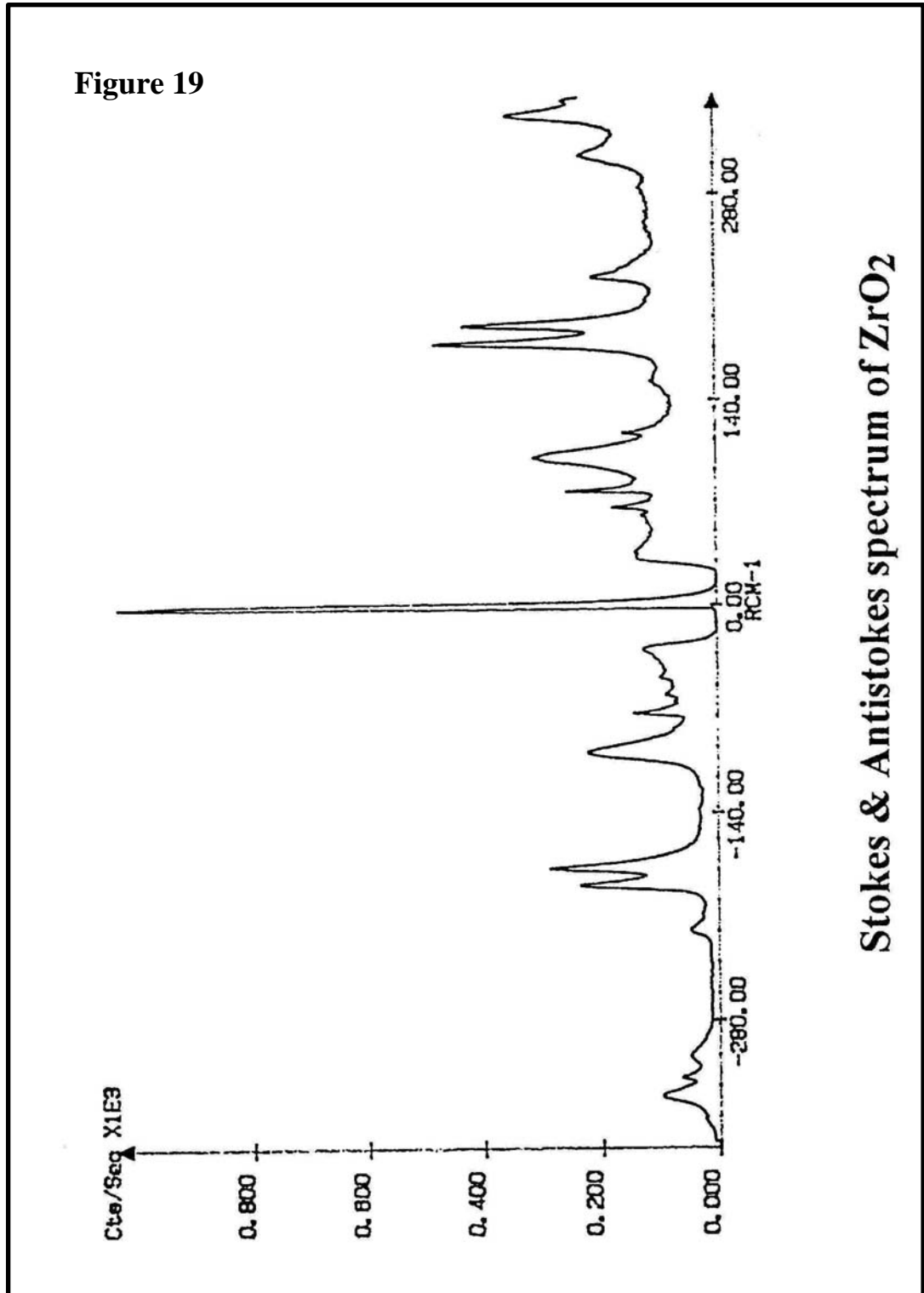
Others optics are available:

- Glass focusing optics:  $F=200\text{mm}$ ,  $F=150\text{mm}$ , or  $F=30\text{mm}$
- Glass collecting optics:  $F=160\text{mm}$ , or  $F=30\text{mm}$
- Quartz focusing and collecting optics for working in the UV range.

### 6.2.2. Cryostats

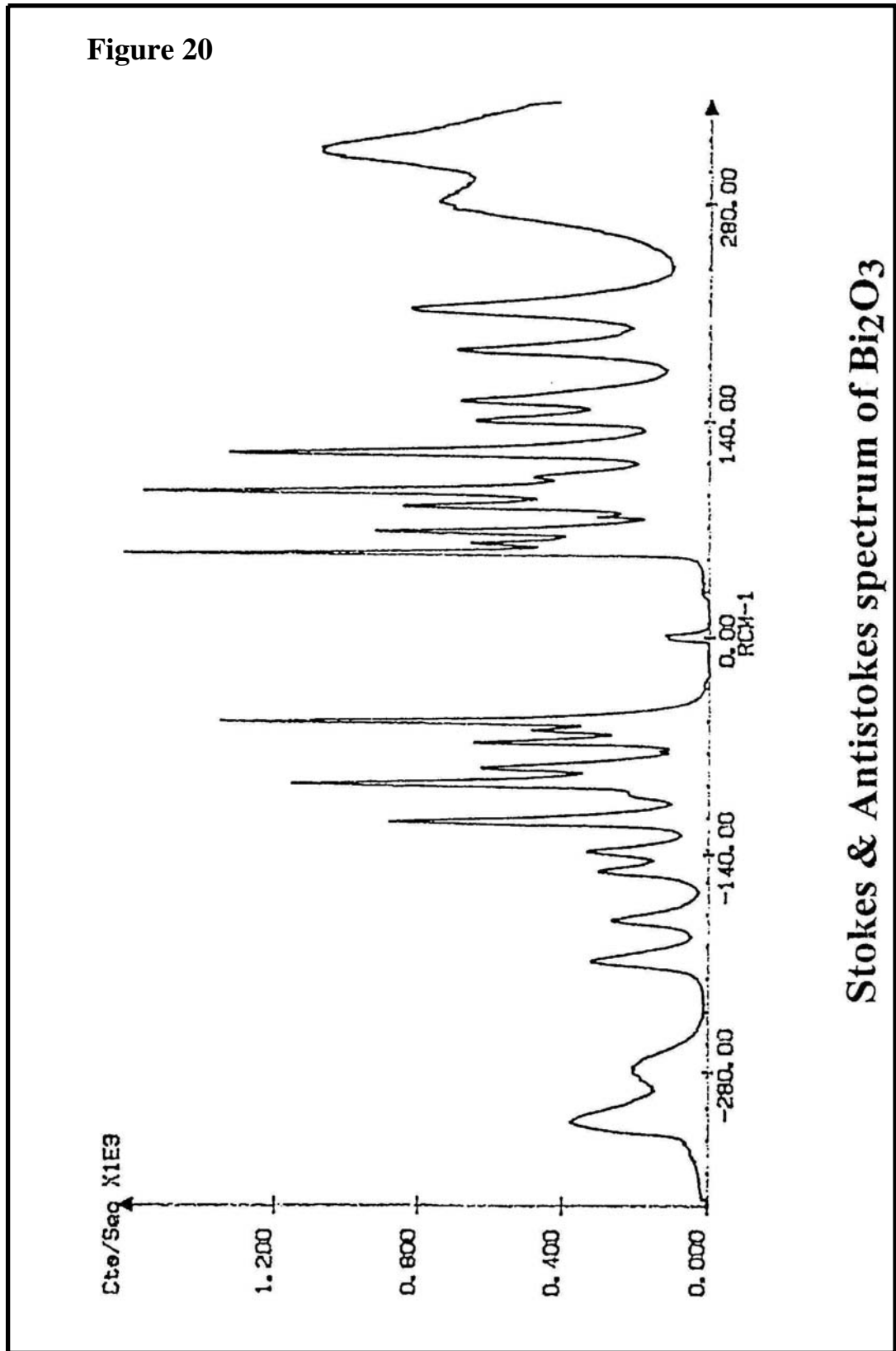
For low temperature measurements, two types of cryostats, Helium and Liquid Nitrogen are proposed. They are directly adaptable and mountable in the macro chamber without any modification.

Figure 19

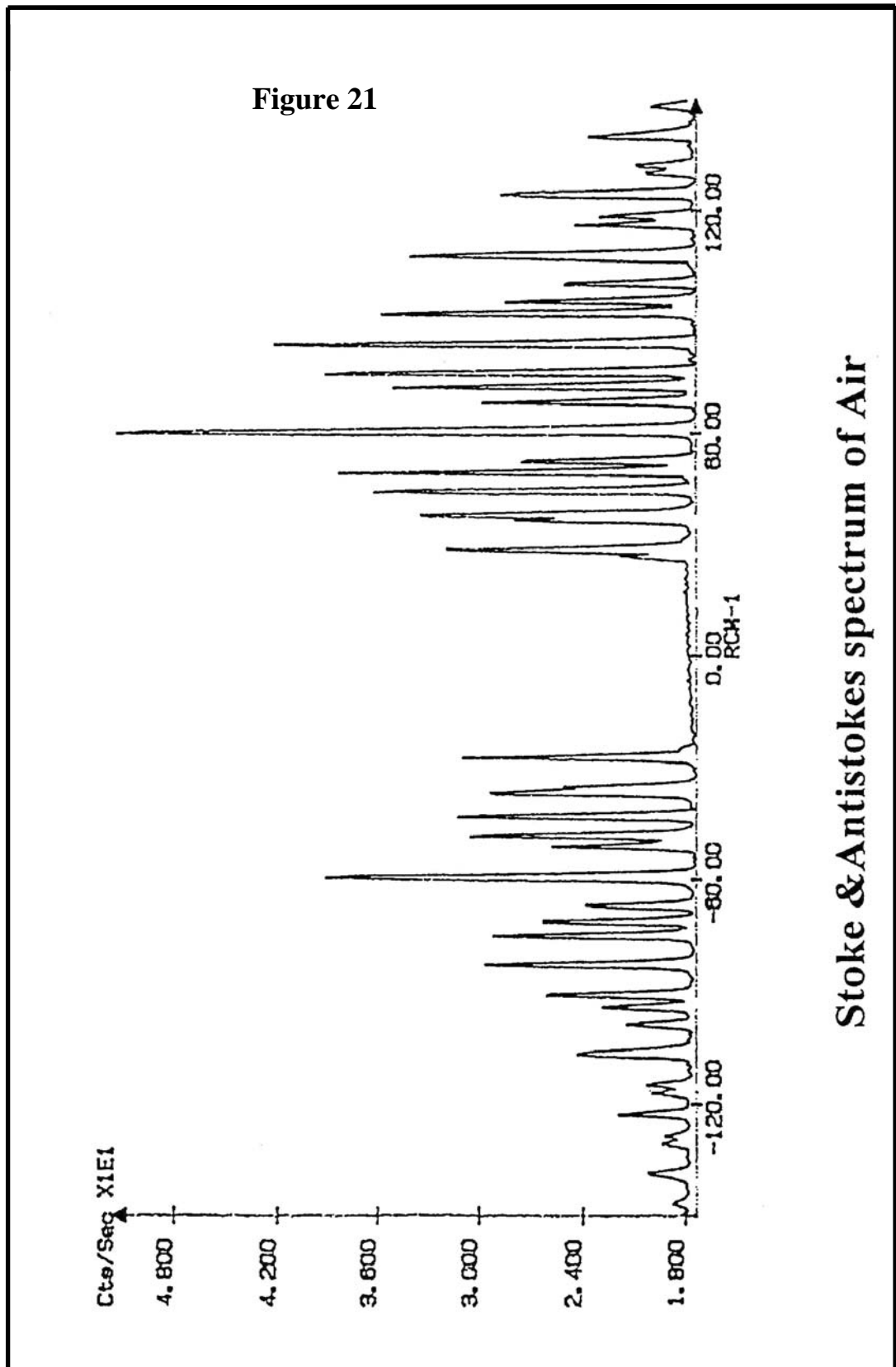


Stokes & Antistokes spectrum of ZrO<sub>2</sub>

Figure 20



Stokes & Antistokes spectrum of Bi<sub>2</sub>O<sub>3</sub>





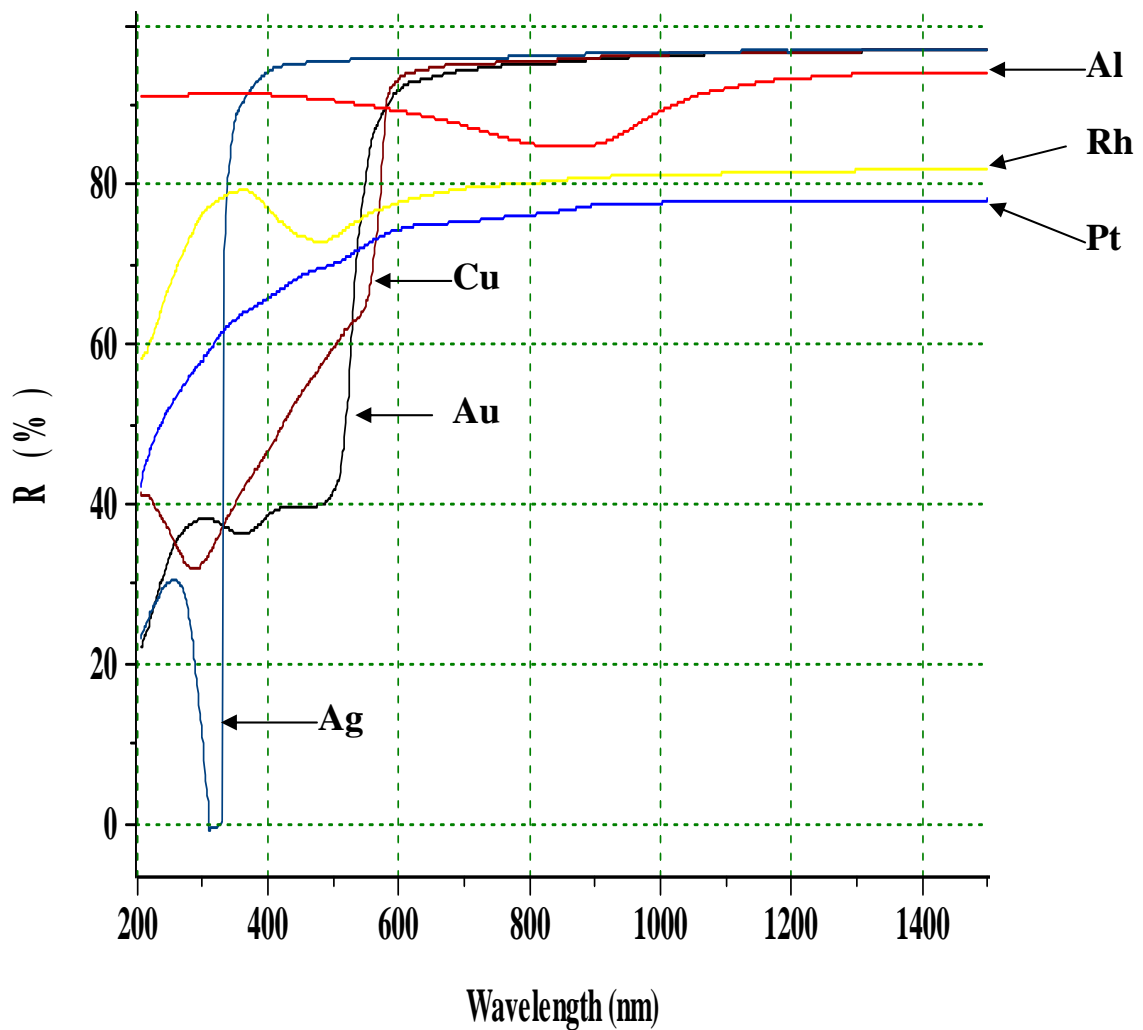


Figure 22

**Reflectance at normal incidence of some freshly evaporated mirror coatings**

**6.2.3. Furnace**

This accessory allows analyzing samples at high temperature, up to 400°C.

**6.2.4. Gas cell**

The cell, with two windows perpendicular to the laser beam, is equipped with two taps for filling gas. Windows under Brewster angle can be supplied on request.

**6.2.5. Spinning cell**

For many fragile samples, to avoid heating the sample and then burning it by the laser, the spinning cell accessory is necessary. It can be used with both solid or liquid samples. The rotation speed of the cell is variable and controlled by a small power supply.

**6.2.6. UV optics**

The material of the standard optics is glass which do not transmit in the UV range. Other quartz optics can be provided for the UV range.

**6.3. For microscope**

**6.3.1. Special objectives**

The BX40 microscope is delivered, in standard, with three plano-achromatic objectives 10X, 50X and 100X, optically adapted to the aperture of the instrument. For special applications, when a long working distance objective is needed, we propose:

- Long working distance plano-achromatic objective (10.6mm)  
50X / 0.50
- Long working distance plano-achromatic objective (3.4mm)  
100X / 0.80

Evidently, other objectives with lower magnification can be used but without a correct aperture adaptation which might introduce some vignetting effect.

Special IR objectives are necessary for working in the near infrared or in the infrared range.

**6.3.2. XY motorized stage for imaging and mapping and video board**

Two accessories are necessary for all the imaging operation.

- Video board: for storing a TV video image and for selecting the area of the sample, which dimensions and the number of analyzed points are defined by the user.
- XY motorized stage: fully controlled and calibrated for each objective by the software, it is used for moving, with extremely high precision, different points, defined on the video image, to be analyzed on to the laser spot. The results of the acquisition can provide a multitude of Raman images at various wavenumbers. Spectral information on each

point is stored and then data treatment is fully possible before reconstructing the image.

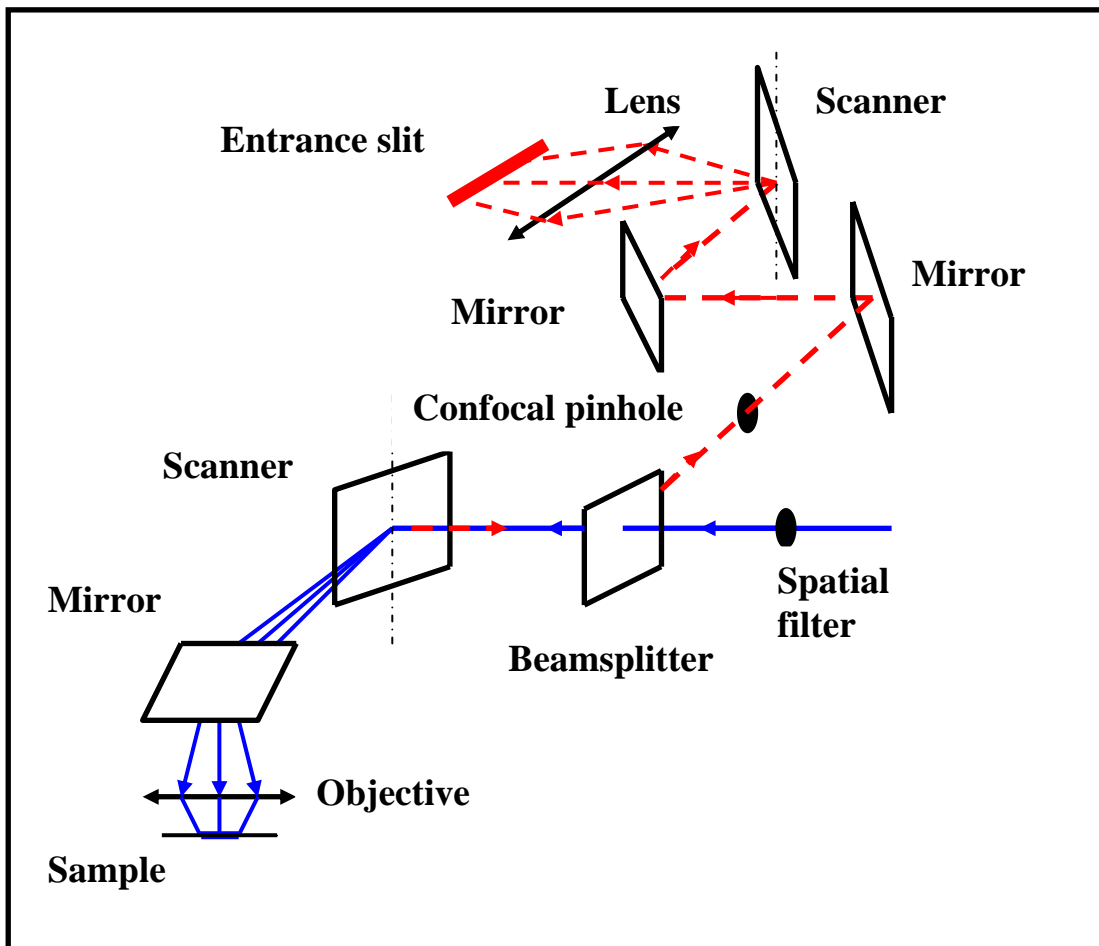
**6.3.3. Rotating stage**

For polarization measurements or for the observation of the sample with polarized white light, this accessory which helps defining the exact orientation of the sample is really useful.

**6.3.4. Heating and cooling stages**

Different heating and cooling stages exist now on the market and can be directly or easily adapted on the microscope. Generally, a long working distance objective 50X is necessary with these accessories. Some stages can be cooled down to liquid nitrogen or to helium temperature.

**6.3.5. Confocal line scanning**



This new device is being developed and will be introduced in year 2000.

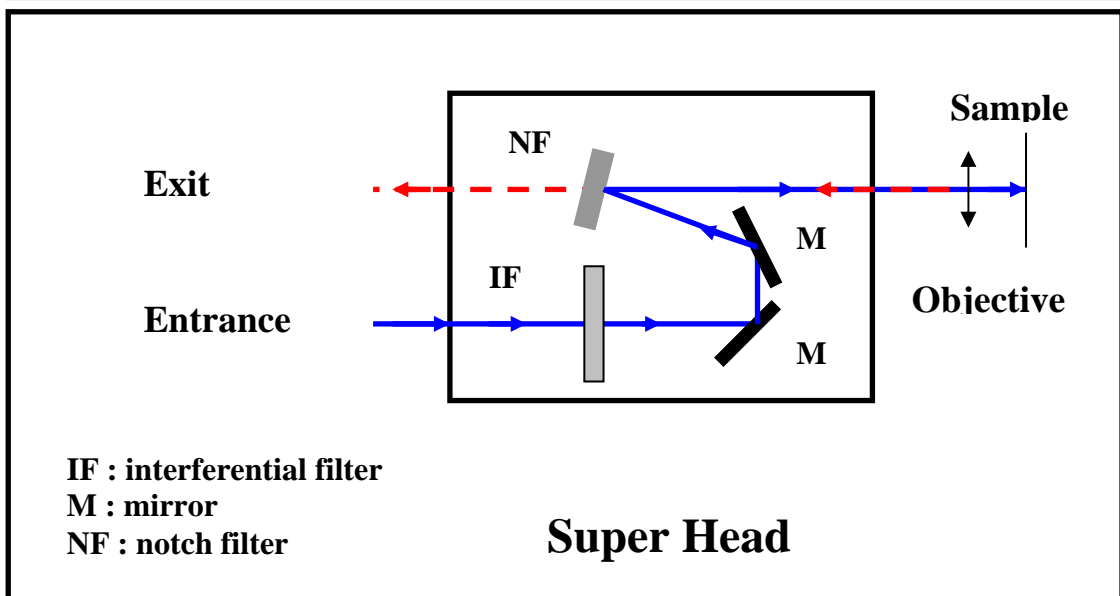
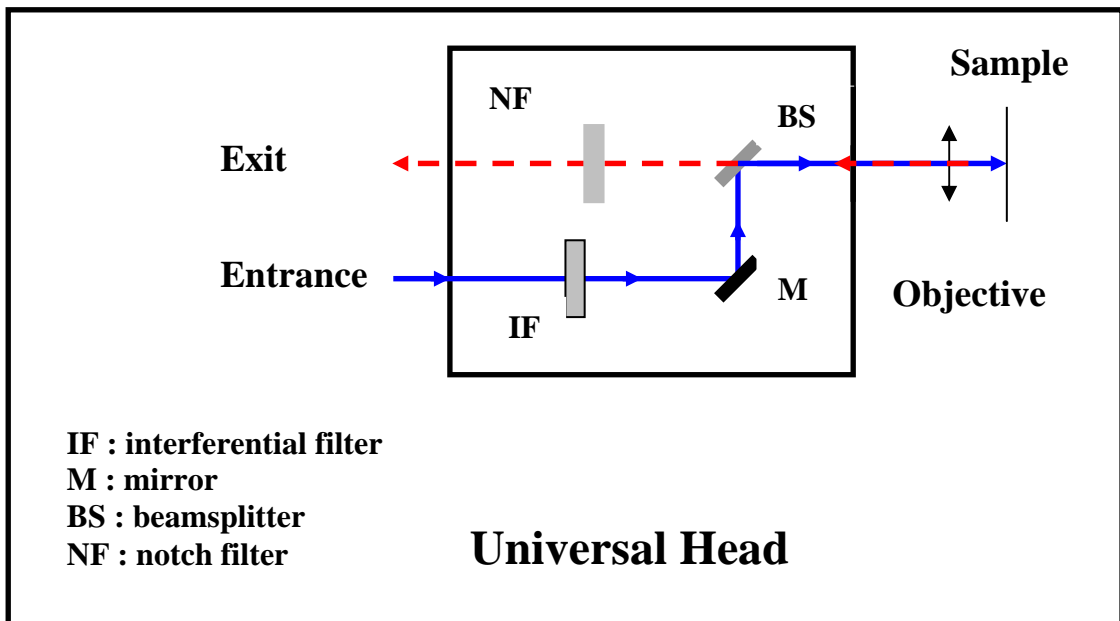
**6.3.6. UV microscope**

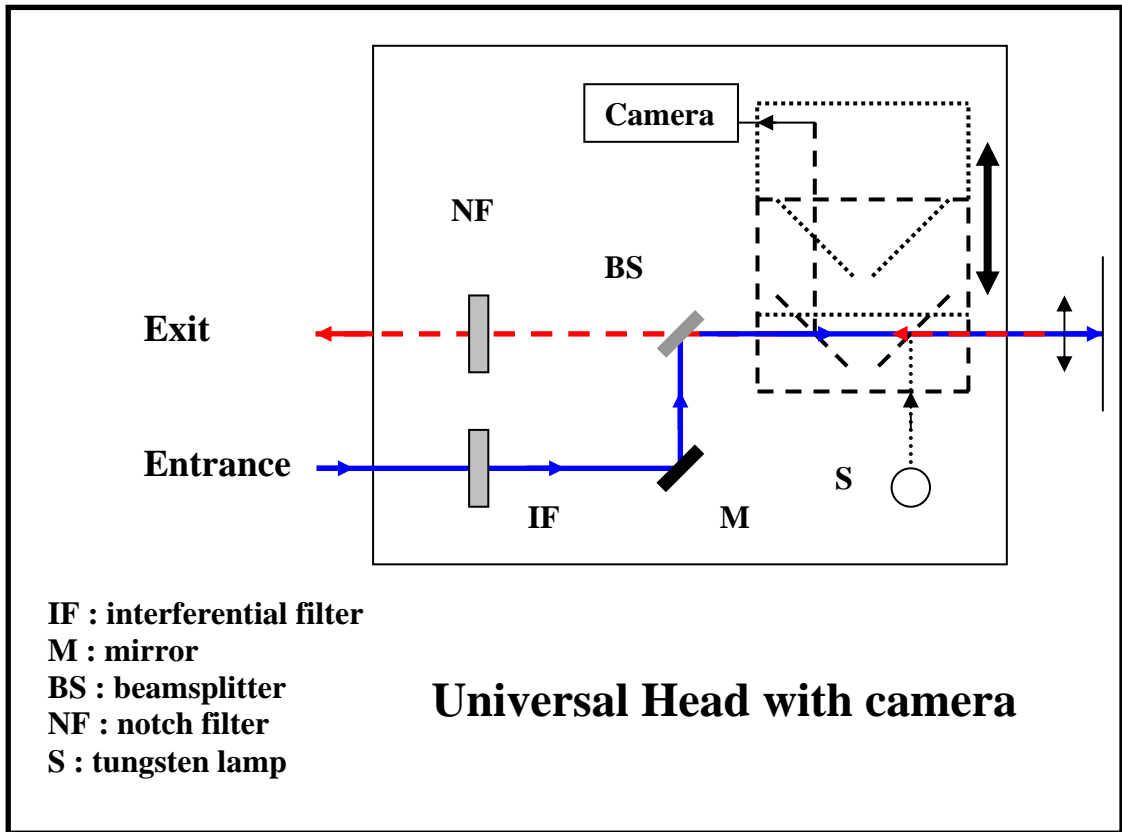
A microscope which will work in the UV range will also be introduced in year 2000.

6.4. Other accessories

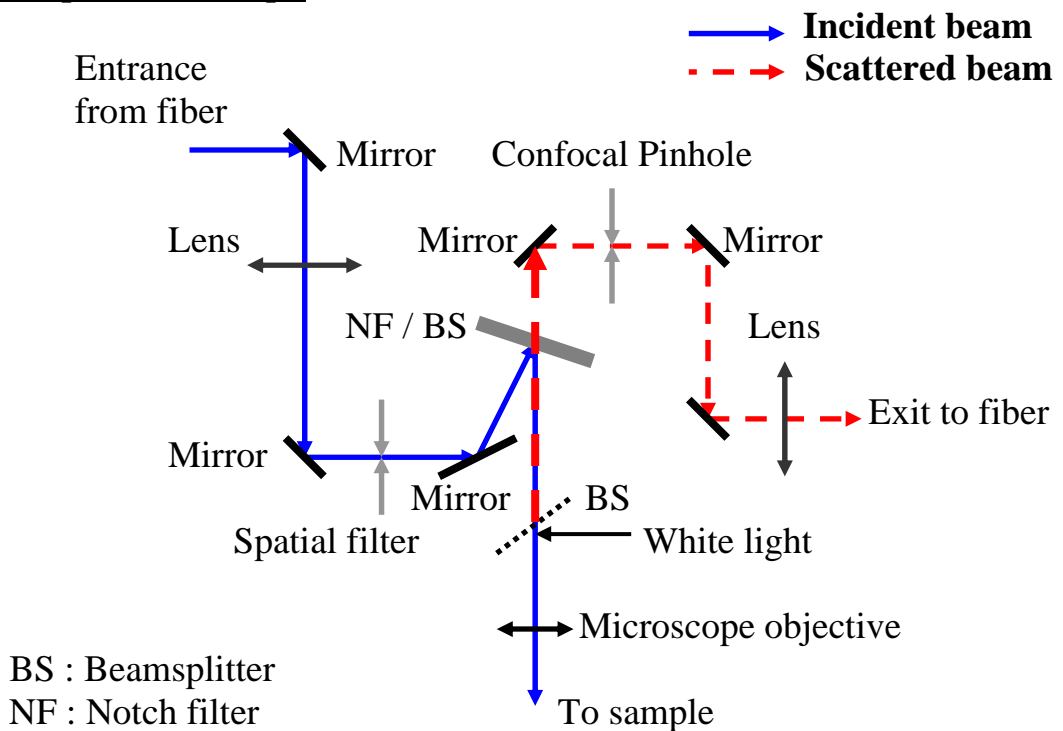
6.4.1. Optical fiber probes

Exciting a sample and collecting scattered light by mean of optical fibers is possible with our probes. They include in particular an interferential filter to eliminate all the plasma emission lines from the laser and the Raman emission from the fiber, a lens or a microscope objective to focus the laser beam on the sample and to collect the scattered light, and a Notch filter for attenuating the excitation line and then reducing the Raman emission of the collecting fiber.





**6.4.2. Fiber optic microscope**



## Annexes

**Annex 1a**

## Procedure for T64000 calibration and coupling with SpectraMax software

When following this procedure, use an emission line of a spectral lamp (for example, the 546.07nm line of Hg) or a Raman line (for example, the 521cm<sup>-1</sup> phonon line of silicon) or any other Raman line which frequency is perfectly known.

For the convention of components notation, refer to the attached optical schematic.

In the procedure that follows, we will use the silicon line at 521cm<sup>-1</sup> For calibration.

Four instrument configurations are covered:

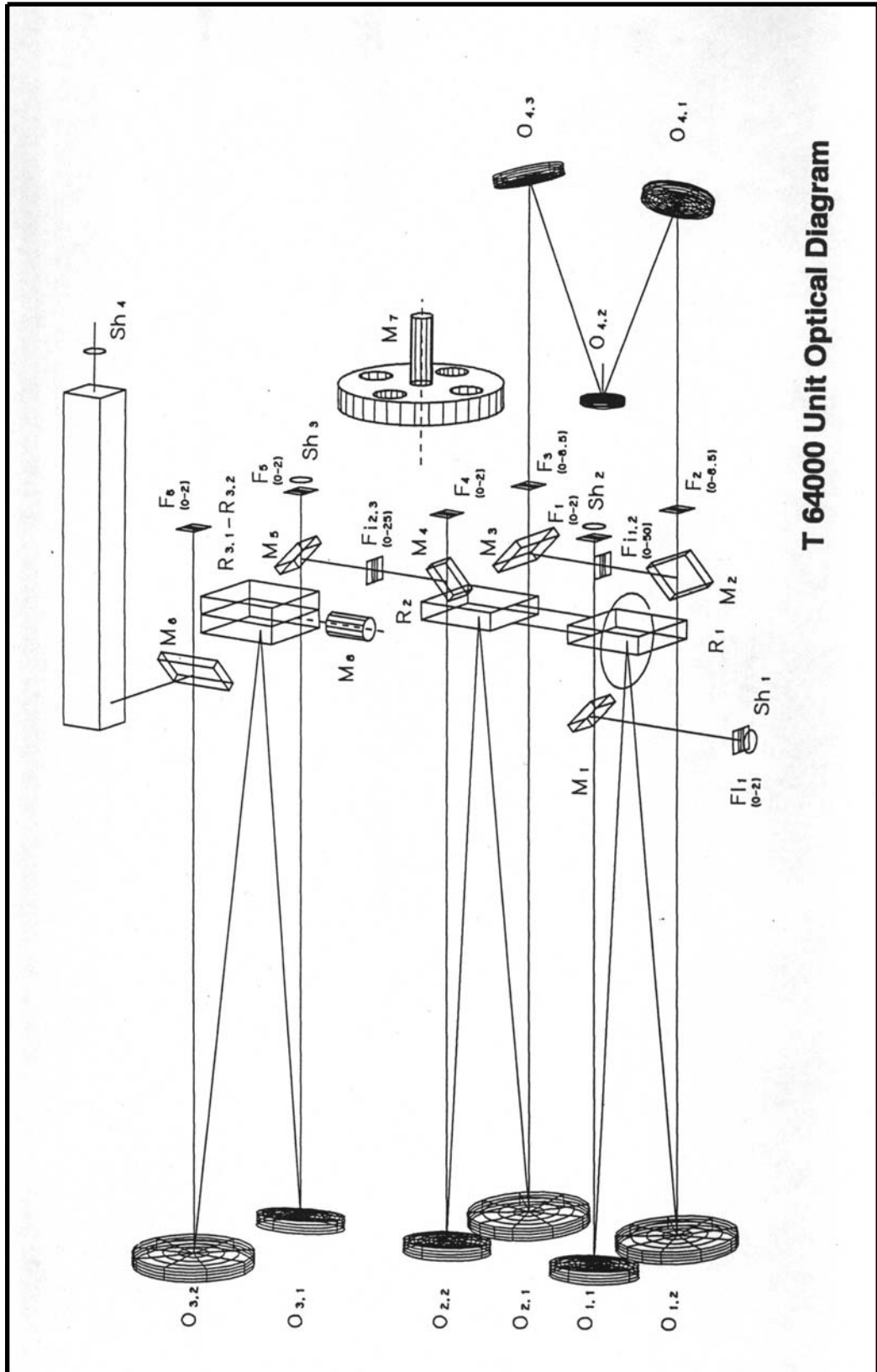
- Configuration 1: basic T64000 (double subtractive + spectrograph) with the additive and single spectrograph options
- Configuration 2: basic T64000 with the additive option
- Configuration 3: basic T64000 with single spectrograph option
- Configuration 4: basic T64000

When making adjustments, the following instructions will explain how to tilt one of the mirrors indicated on the schematic. All mirrors are mounted on a 3-point holder with 3-adjustment screws. In general, one uses **A** to move the line vertically, and **B** to move it horizontally. In no case **C** should be touched.

### Important remark:

**During the installation, the coupling of the system over the whole spectral range has been done and adjusted by our service engineer. For the good working of the system over this large spectral range, we strongly recommend you to do not touch any of the adjustment screws of the collimators in each monochromator (O<sub>1,1</sub> ; O<sub>1,2</sub> ; O<sub>2,1</sub> ; O<sub>2,2</sub> ; O<sub>3,1</sub> ; O<sub>3,2</sub>)**





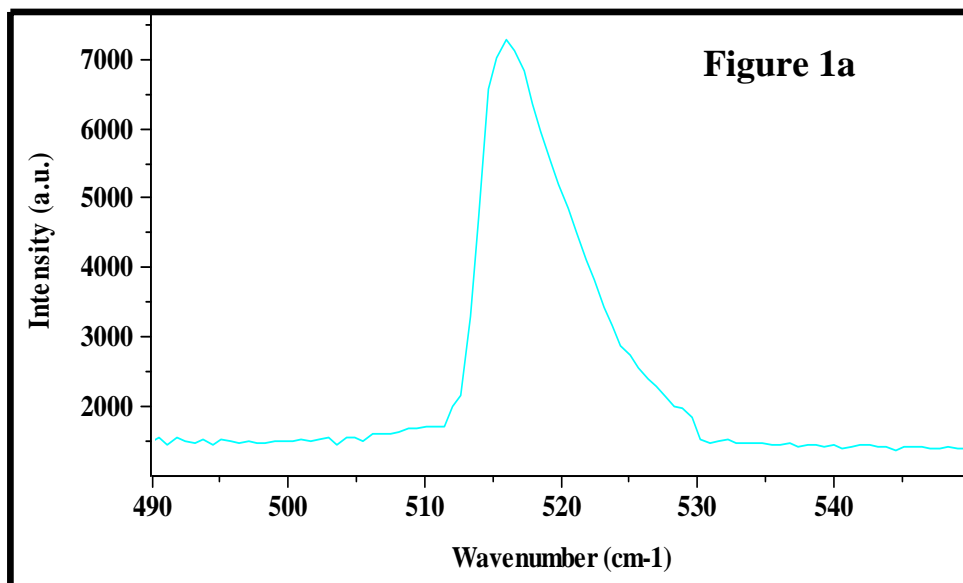
T 64000 Unit Optical Diagram

Configuration 1

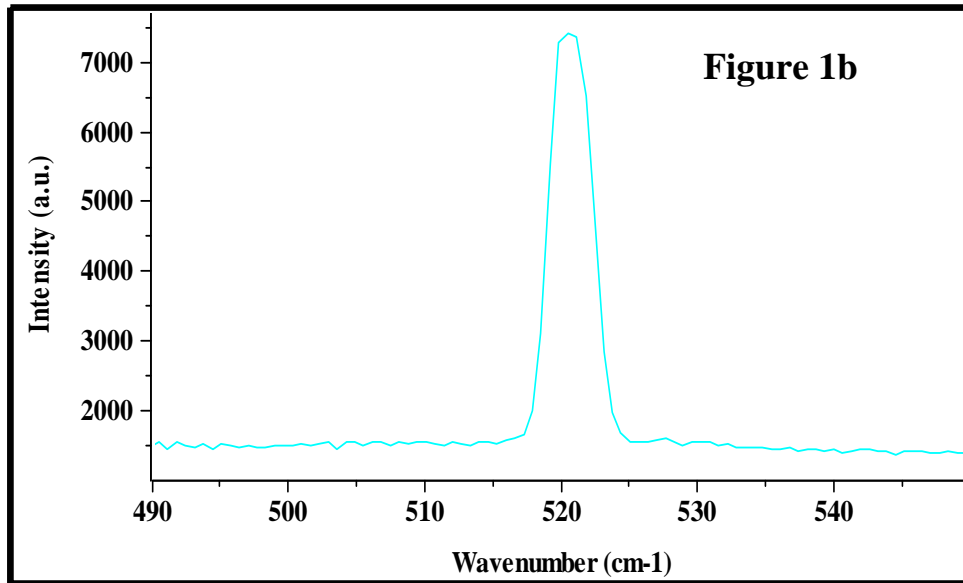
**Basic T64000 (Double Subtractive + Spectrograph)  
with the Additive and Single Spectrograph options**

**A. Calibration of the Double Additive  
Foremonochromator**

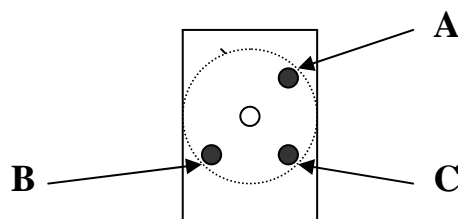
1. First set the instrument in Triple Subtractive mode, using System Status.  
In the program MOVE AND CALIBRATE, choose the central position,  $521\text{cm}^{-1}$ . (In Subtractive mode, both monochromators will be positioned at this frequency whereas in Additive mode, the foremonochromator will be displaced relatively to the spectrograph.)
2. Go into the program SYSTEM STATUS and select the TRIPLE ADDITIVE mode.  
Set  $F_1 = 100\mu\text{m}$   
 $F_2 = 500\mu\text{m}$   
 $F_3 = F_{i2/3} = 2\text{mm}$
3. Go into the RTD (Real Time Display) program and select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is asymmetric, it is not well centered on slit  $F_2$ . (Refer to Figure 1a)



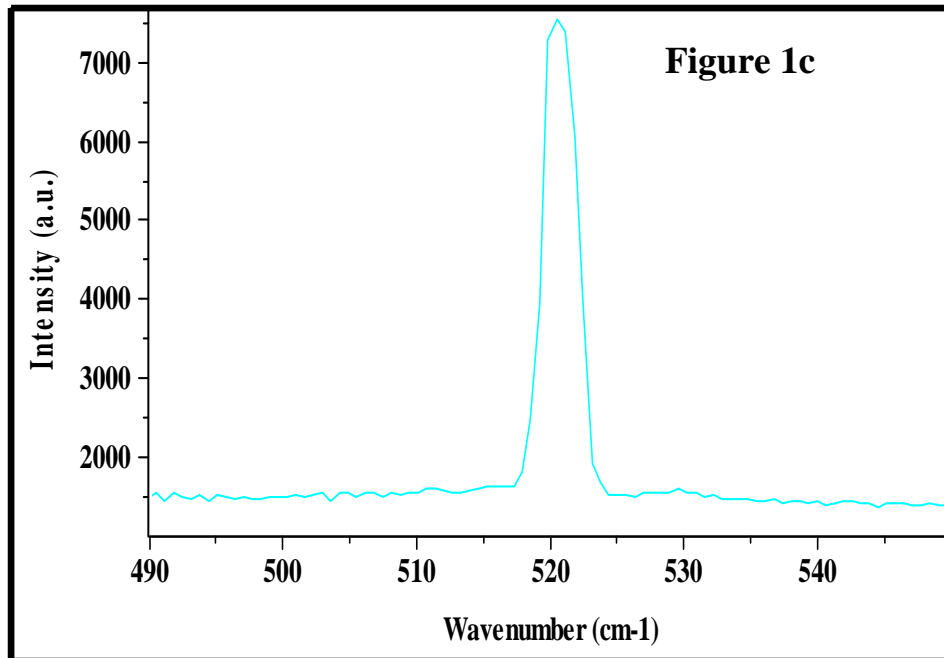
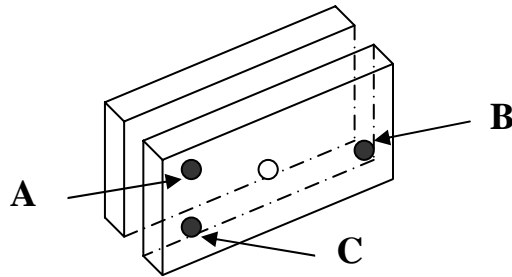
4. Progressively close  $F_2$  down  $100\mu\text{m}$ .  
Adjust the central wavenumber of the foremonochromator, (by moving it and **not by adjusting the collimator  $O_{1,1}$  or  $O_{1,2}$** ) in order to center the line on the slit  $F_2$ . When the line is correctly centered on the slit, one can see on the spectrum the symmetry of the silicon line. (Refer to Figure 1b)



5. Progressively close  $F_3$  down  $100\mu\text{m}$ .  
The line should keep the same profile and the same intensity as above. If not, adjust the mirror  $O_{4,2}$  by carefully turning the screw labeled A in the hereunder drawing in order to obtain the maximum signal. Reduce the height limiter of slit  $F_3$ . If there is a loss of signal when the height limiter of  $F_3$  is larger than the ones of  $F_2$  or  $F_1$ , carefully turn screw B in order to recover the maximum of the signal.



6. Progressively close  $F_{i2/3}$  down  $100\mu\text{m}$   
 The line should be well resolved while maintaining the intensity. If not, adjust the mirror M4 by carefully turning screw A in order to obtain the maximum signal. Reduce the height limiter of  $F_{i2/3}$  and, if necessary, turn screw B to maximize the signal.(Refer Figure 1c)



7. Exit the RTD program.  
 Go to the program MOVE & CALIBRATE and calibrate the foremonochromator at  $521\text{cm}^{-1}$

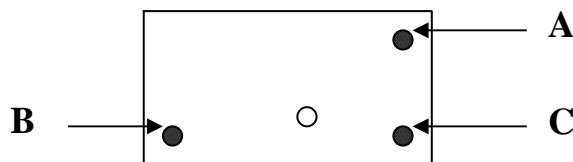
## **B. Calibration of the Spectrograph**

1. Go to the SYSTEM STATUS screen and select the Single Monochromator option. Turn the 4-position wheel in front of the entrance slit to direct light from the microscope or macro chamber, whichever is being used, onto slit  $F_5$  of the spectrograph.

2. Check that the light is entering the slit. Go into the RTD program and select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is not perfectly centered on the CCD detector, scan slowly the spectrograph and center the silicon line on the central pixel of the detector. Exit the RTD program.
3. Go to the program MOVE AND CALIBRATE and calibrate the spectrograph at  $521\text{cm}^{-1}$ .

### C. Coupling the Double Additive Foremonochromator and the Spectrograph.

Go into the RTD program and select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is not perfectly centered on the detector, adjust the mirror M5 by carefully turning the screw A in order to center the line at  $521\text{cm}^{-1}$  or on the central pixel of the detector. To access the adjustment screw of this mirror M5, it is necessary to remove the cover protecting the scanning mechanism of the T64000, that is at the back of the apparatus.

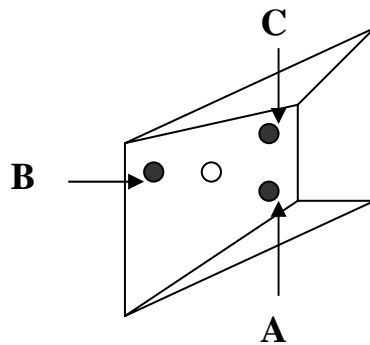


### D. Calibration of the Double Subtractive Foremonochromator

The monochromators being already calibrated in Triple Additive mode, none of the 2 grating drives should be moved during the Triple Subtractive calibration.

1. Go to the SYSTEM STATUS program and select the Triple Subtractive mode.  
 Set  $F_1 = 100\mu\text{m}$   
 $F_{11/2} = 500\mu\text{m}$   
 $F_{i2/3} = 2\text{mm}$

2. Go into RTD program and select the integration time and the X,Y expansion that enables a clear display of the silicon line.
3. Progressively close  $F_{i1/2}$  down  $100\mu\text{m}$ .  
 If there is an asymmetry or a loss in signal, (refer to Figure 1a) adjust the mirror M2 by carefully turning the screw A to maximize the signal. Reduce the height of slit  $F_{i1/2}$  while turning screw B on mirror M2 (if necessary) in order to keep the maximum of signal.  
 These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.



## **E. Coupling the Double Subtractive Foremonochromator and the Spectrograph**

Progressively close  $F_{i2/3}$  down  $100\mu\text{m}$ .  
 Gradually reduce the slit width and the height limiter to their minima while adjusting the mirror M3 to center the  $521\text{cm}^{-1}$  line on the slit and obtain the maximum signal. To center the  $521\text{cm}^{-1}$  line between the slit jaws and obtain the maximum signal, turn screw A. If there is a loss of intensity when closing the height limiter, turn screw B. Refer to Figure 1d showing the superposition of the lines in Triple Subtractive and Triple Additive modes.

These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.

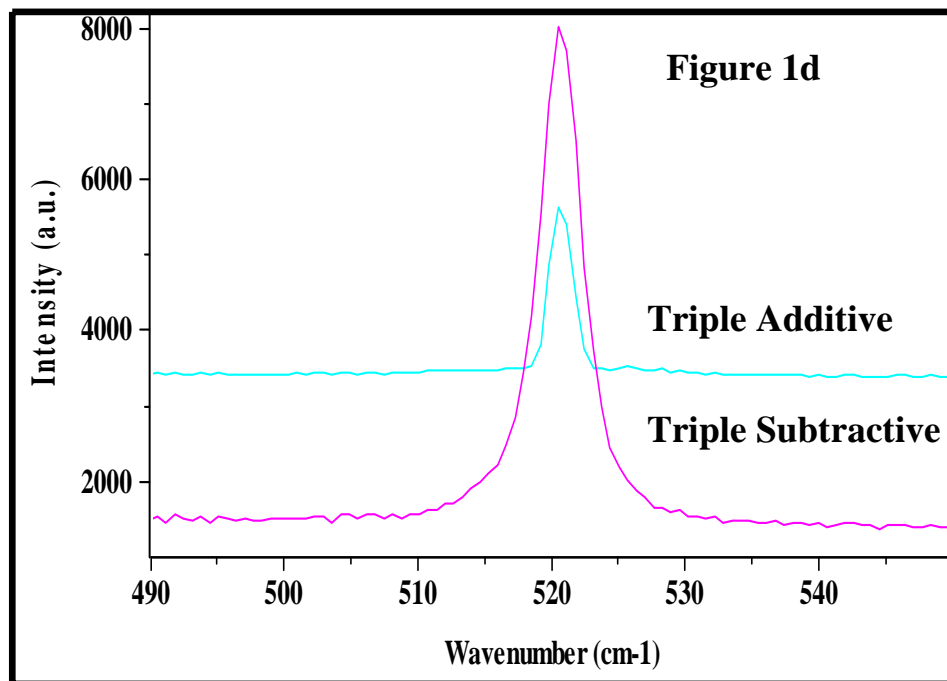
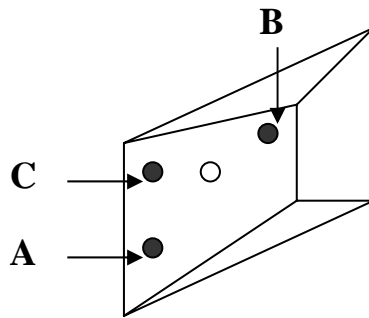


Figure 2 shows that, after this procedure, there is a perfect calibration of the two modes Triple Additive and Triple Subtractive, in the normal conditions of use; that is to say, with the intermediate slits wide enough to achieve full spectral coverage on the multichannel detector.

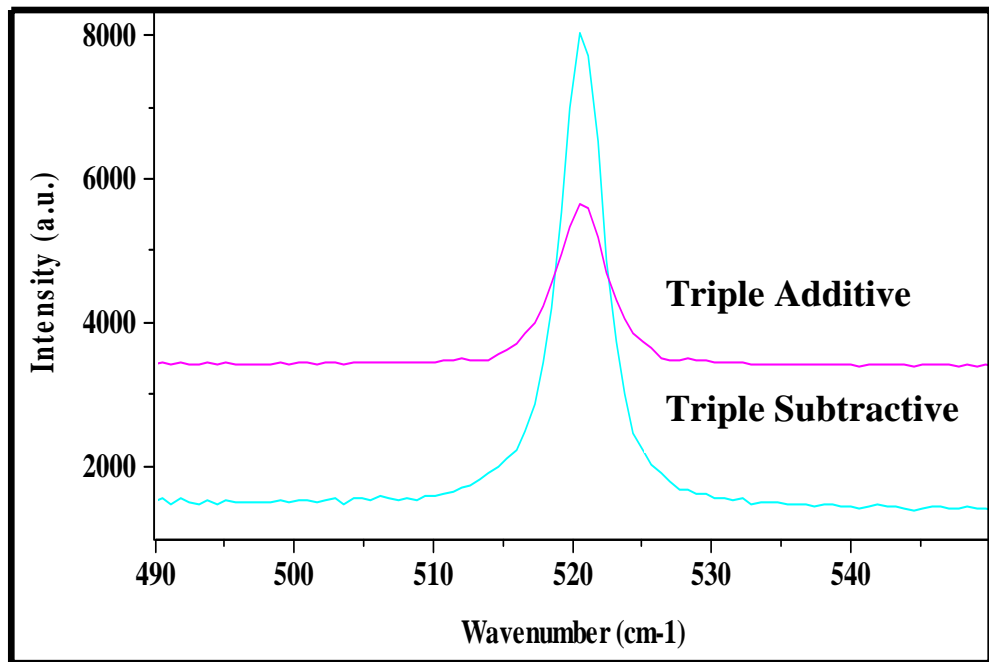
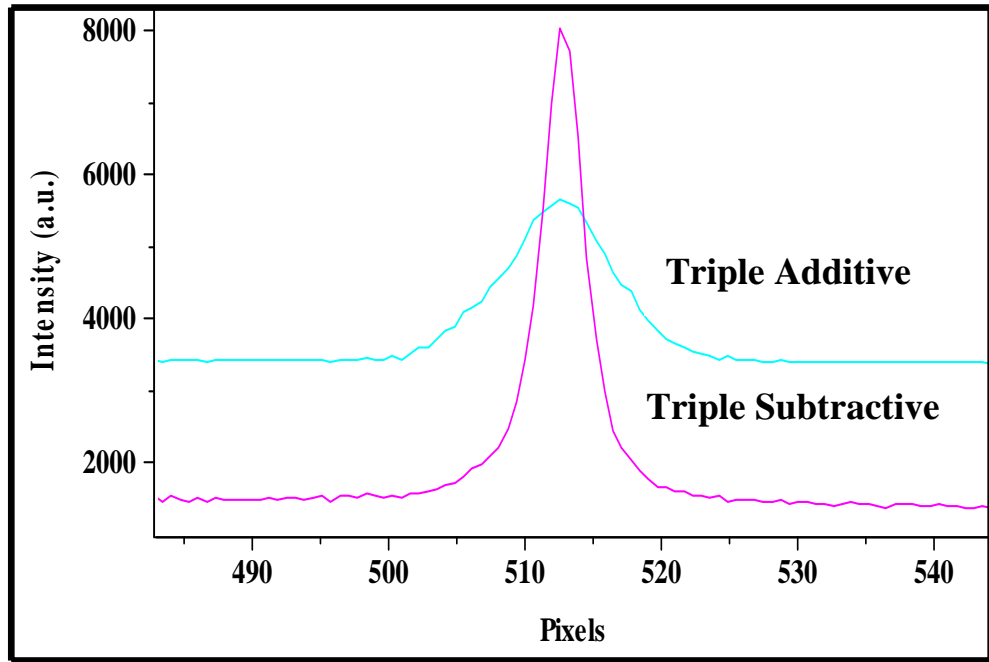


Figure 2



## Configuration 2

### Basic T64000 with the Additive option

#### A. Calibration of the Double Additive Foremonochromator

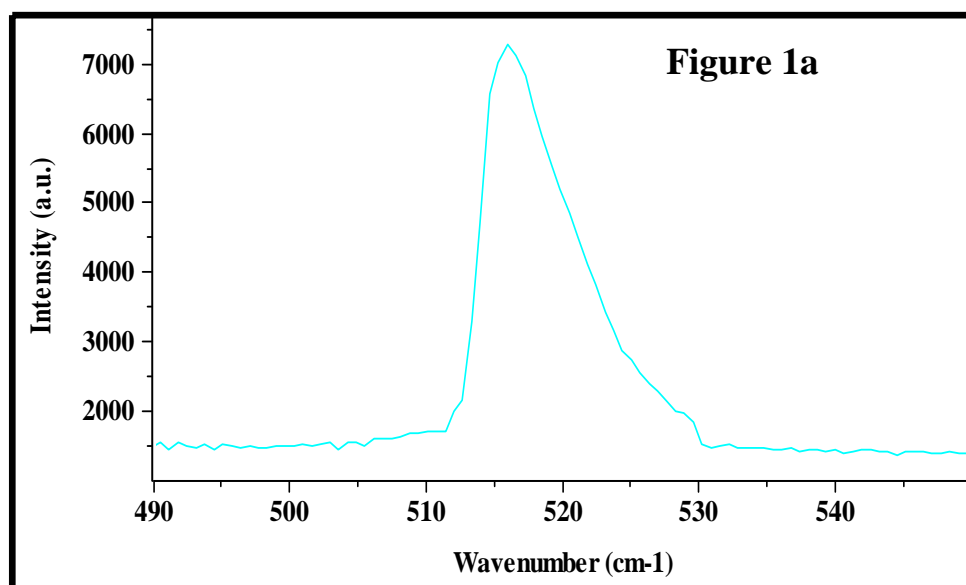
1. Initially, set the system in Triple Subtractive.

In the program MOVE AND CALIBRATE, choose the central position,  $521\text{cm}^{-1}$ . (In Subtractive mode, both monochromators will be positioned at this frequency whereas in Additive mode, the foremonochromator will be displaced relatively to the spectrograph.)

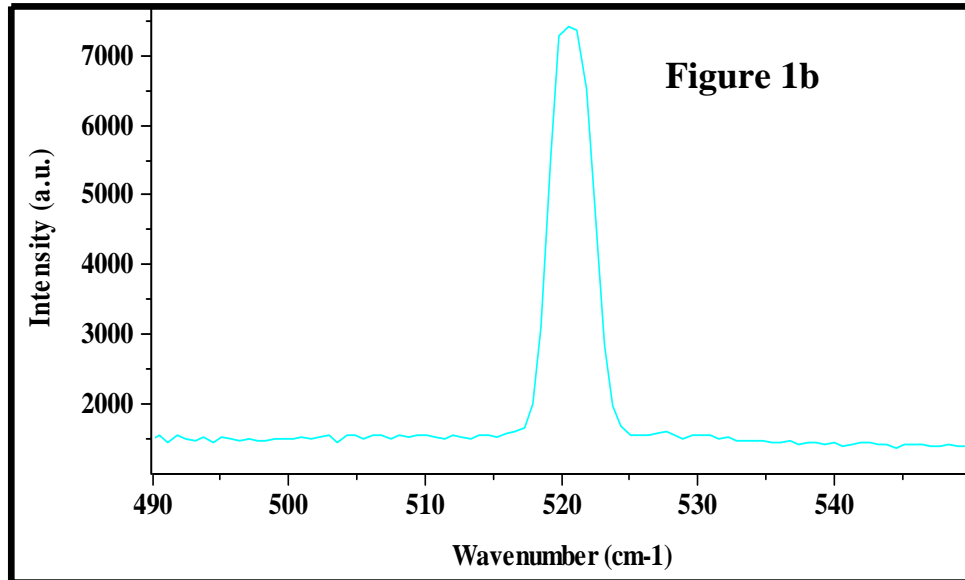
2. Go into the program SYSTEM STATUS and select the TRIPLE ADDITIVE mode.

Set  $F_1 = 100\mu\text{m}$   
 $F_2 = 500\mu\text{m}$   
 $F_3 = F_{i2/3} = 2\text{mm}$

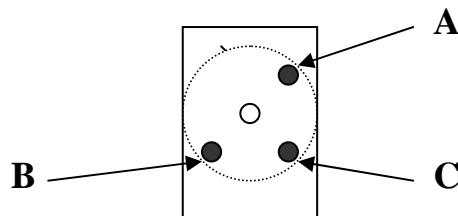
3. Go into the RTD (Real Time Display) program and select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is asymmetric, it is not well centered on slit  $F_2$ . (Refer to Figure 1a)



- Progressively close  $F_2$  down  $100\mu\text{m}$ .  
Adjust the central wavenumber of the foremonochromator, (by moving it and **not by adjusting the collimator  $O_{1,1}$  or  $O_{1,2}$** ) in order to center the line on the slit  $F_2$ . One can see on the spectrum the symmetry of the silicon line. (Refer to Figure 1b)

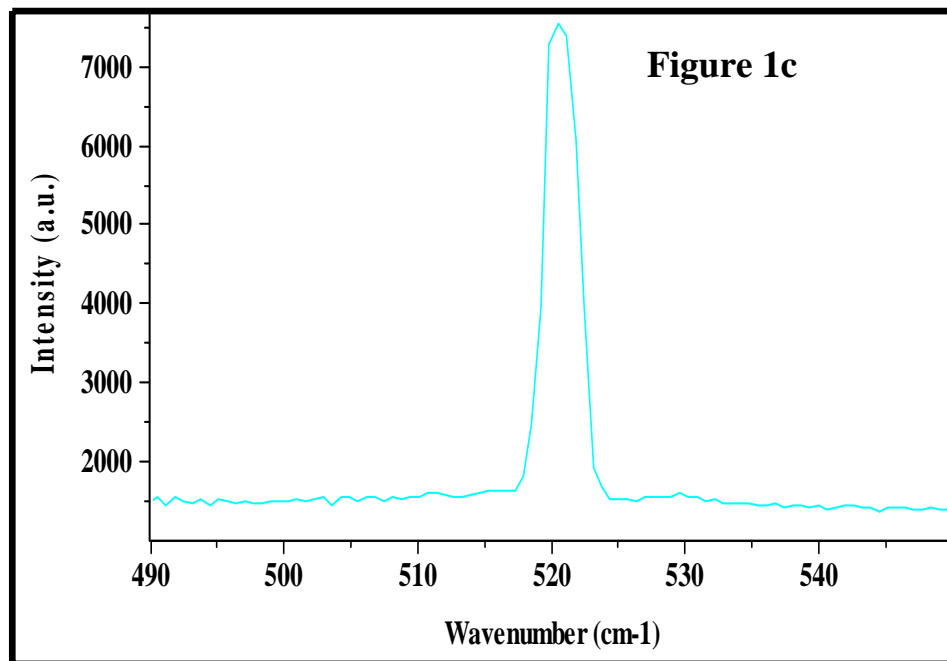
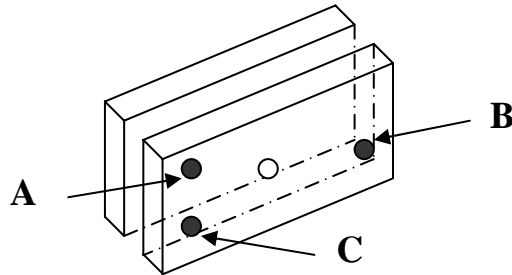


- Progressively close  $F_3$  down  $100\mu\text{m}$ .  
The line should keep the same profile and the same intensity as above. If not, adjust the mirror  $O_{4,2}$  by carefully turning the screw labeled A in the hereunder drawing in order to obtain the maximum signal. Reduce the height limiter of slit  $F_3$ . If there is a loss of signal when the height limiter of  $F_3$  is larger than that of  $F_2$  or  $F_1$ , carefully turn screw B in order to recover the maximum of the signal.



- Progressively close  $F_{12/3}$  down  $100\mu\text{m}$

The line should be well resolved while maintaining the intensity. If not, adjust the mirror M4 by carefully turning screw A in order to obtain the maximum signal. Reduce the height limiter of F<sub>12/3</sub> and, if necessary, turn screw B to maximize the signal.(Refer Figure 1c)



7. Exit the RTD program.  
Go to the program MOVE & CALIBRATE and calibrate the foremonochromator at 521cm<sup>-1</sup>

## **B. Calibration of the Spectrograph**

1. If the line is not perfectly centered at 521cm<sup>-1</sup>, scan the

Spectrograph in order to move the line to the center of the detector.  
(the line centered on the central pixel)

2. Go into the program MOVE AND CALIBRATE and calibrate the spectrograph at  $521\text{cm}^{-1}$ .

## C. Calibration of the Double Subtractive Foremonochromator

The monochromators being already calibrated in Triple Additive mode, none of the grating drive should be moved during the Triple Subtractive calibration.

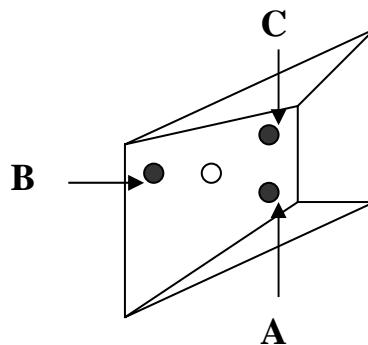
1. Go to the SYSTEM STATUS program and select the Triple Subtractive mode.

Set  $F_1 = 100\mu\text{m}$   
 $F_{i1/2} = 500\mu\text{m}$   
 $F_{i2/3} = 2\text{mm}$

2. Go into RTD program and select the integration time and the X,Y expansion that enables a clear display of the silicon line.
3. Progressively close  $F_{i1/2}$  down  $100\mu\text{m}$ .

If there is an asymmetry or a loss in signal, (refer Figure 1a) adjust the mirror M2 by carefully turning the screw A to maximize the signal. Reduce the height of slit  $F_{i1/2}$  while turning screw B on mirror M2 (if necessary) in order to maintain the maximum of signal.

These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.



## D. Coupling the Double Subtractive foremonochromator and the Spectrograph

Progressively close  $F_{12/3}$  down  $100\mu\text{m}$ .  
 Gradually reduce the slit width and the height limiter to their minima while adjusting the mirror M3 to center the  $521\text{cm}^{-1}$  line on the slit and obtain the maximum signal. To center the  $521\text{cm}^{-1}$  line between the slit jaws and obtain the maximum signal, turn screw A. If there is a loss of intensity when closing the height limiter, turn screw B. Refer to Figure 1d showing the superposition of the lines in Triple Subtractive and Triple Additive modes. These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.

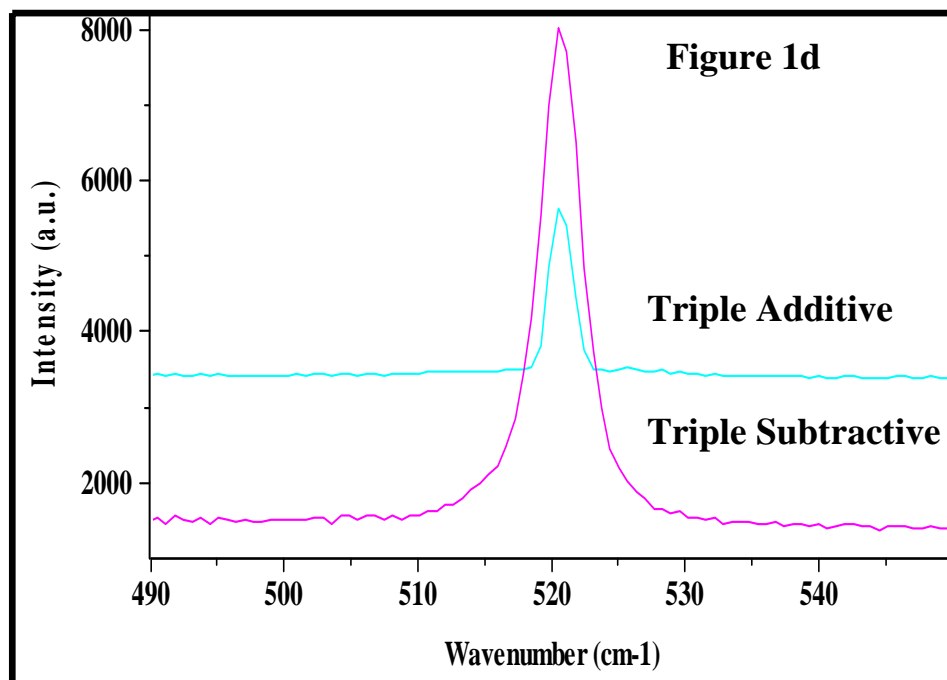
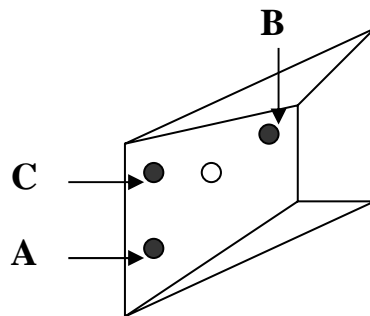
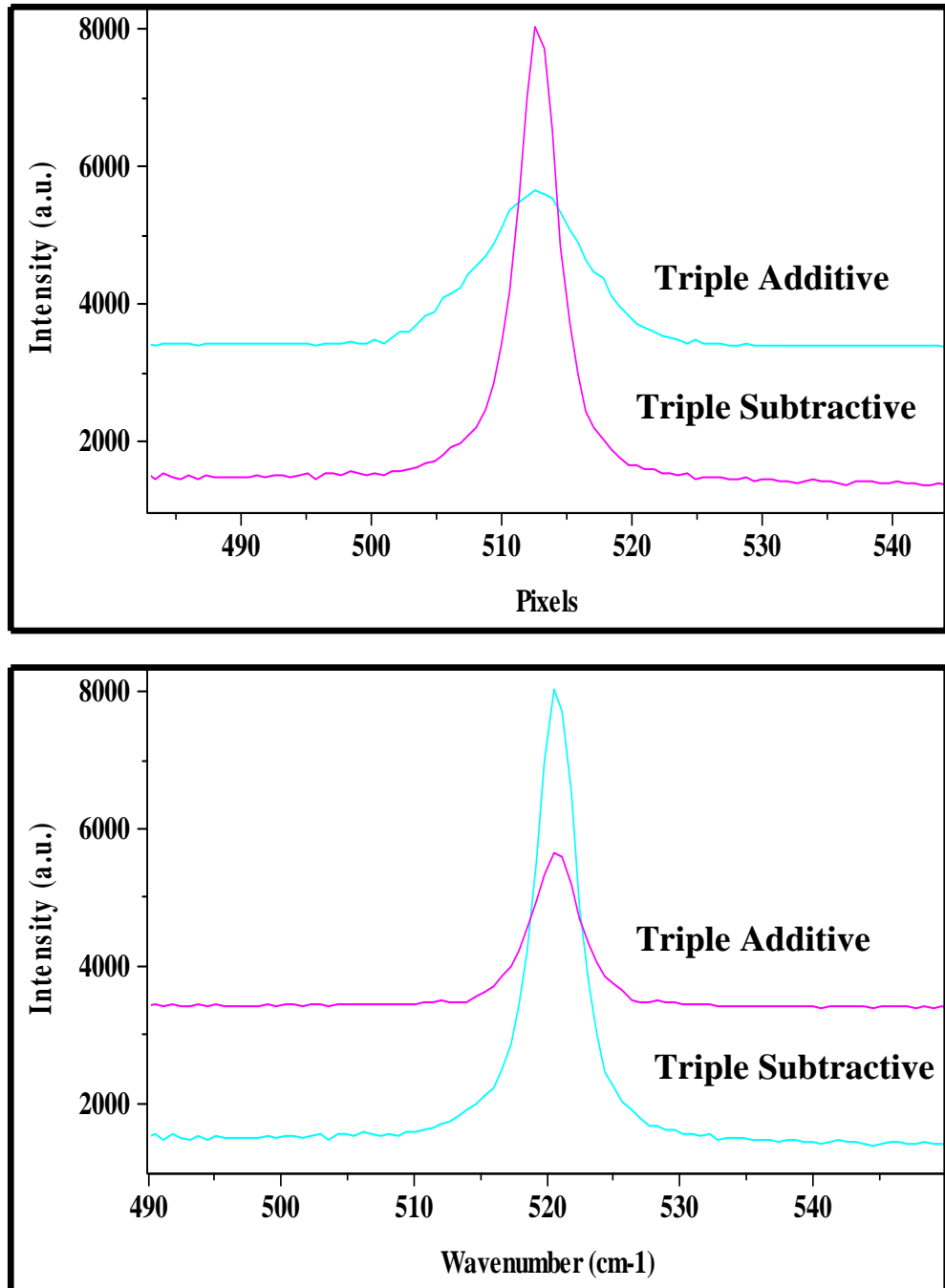


Figure 2 shows that, after this procedure, there is a perfect calibration of the two modes Triple Additive and Triple Subtractive, in the normal conditions of use; that is to say, with the intermediate slits wide enough to achieve full spectral coverage on the multichannel detector.



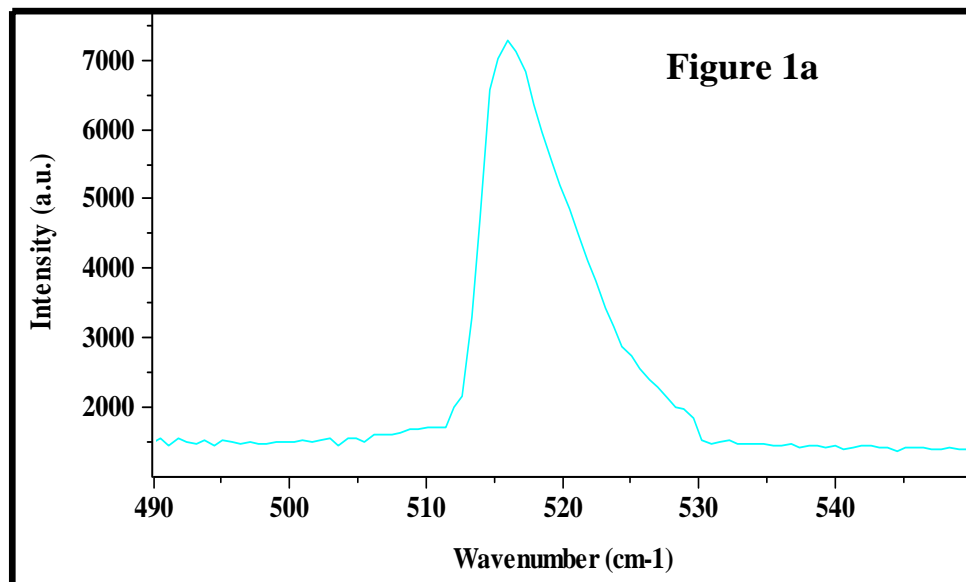
**Figure 2**

## Configuration 3

### Basic T64000 with the Single Spectrograph option

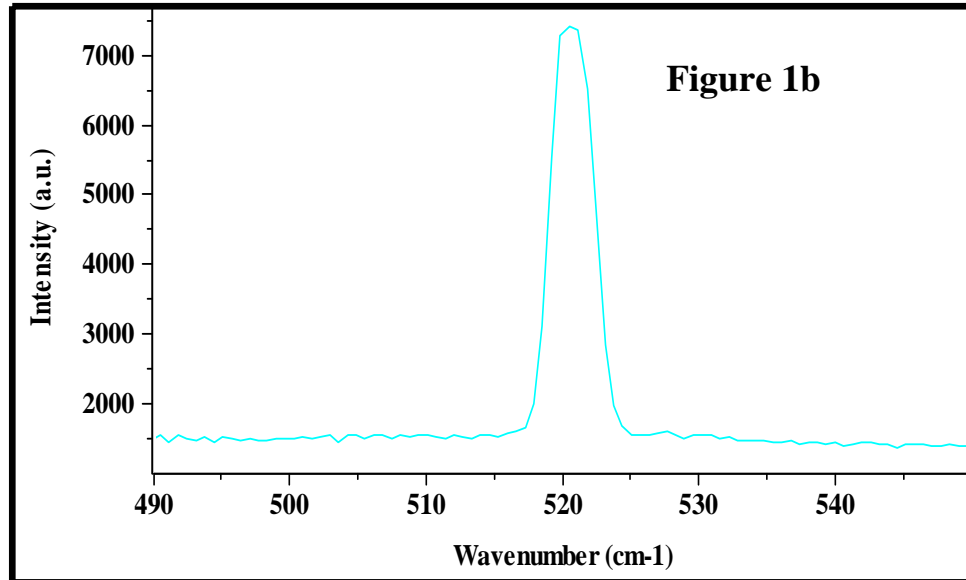
#### A. Calibration of the Double Subtractive Foremonochromator

1. In the program MOVE AND CALIBRATE, choose the central position  $521\text{cm}^{-1}$ . Both monochromators will be positioned at this frequency.
2. Set  $F_1 = 100\mu\text{m}$   
 $F_{i1/2} = 500\mu\text{m}$   
 $F_{i2/3} = 2\text{mm}$   
 Go into the RTD program and select the integration time and the X, Y expansion that enables a clear display of the silicon line.
3. Progressively close the slit  $F_{i1/2}$  down  $100\mu\text{m}$ .  
 If this line is asymmetric, it is not well centered on slit  $F_{i1/2}$ .  
 (Refer to Figure 1a).



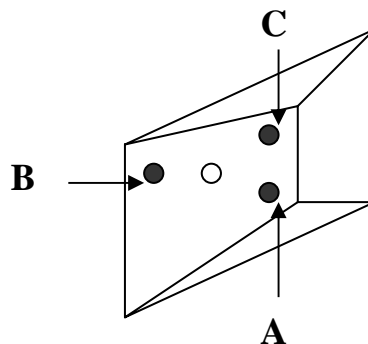
Adjust the central wavenumber of the foremonochromator, (by moving it and **not by adjusting the collimator  $O_{1,1}$  or  $O_{1,2}$** ) in

order to center the line on the slit  $F_{i1/2}$ . When it is centered, one can see on the spectrum the symmetry of the silicon line. (Refer to Figure 1b).



Reduce the height limiter of slit  $F_{i1/2}$ . If there is a loss of signal when the height limiter of  $F_{i1/2}$  is larger than that of  $F_1$ , carefully turn screw B on the mirror M2 in order to recover the maximum of the signal.

These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.

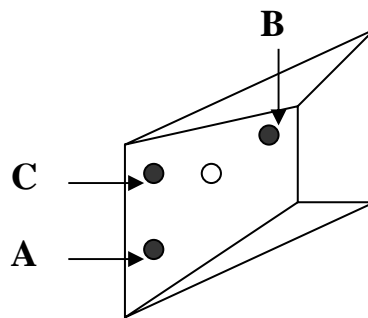


4. Progressively close  $F_{i2/3}$  down 100 $\mu$ m.



Gradually reduce the slit width and the height limiter to their minima while adjusting the mirror M3 to center the  $521\text{cm}^{-1}$  line on the slit and obtain the maximum signal. To center the  $521\text{cm}^{-1}$  line between the slit jaws and obtain the maximum signal, turn screw A. If there is a loss of intensity when closing the height limiter, turn screw B.

These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.



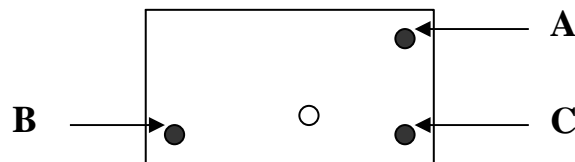
5. Exit the RTD program  
Go to the program MOVE & CALIBRATE and calibrate the foremonochromator at  $521\text{cm}^{-1}$

## **B. Calibration of the Spectrograph**

1. Enter the SYSTEM STATUS screen, then select the Single option. Rotate the 4-position wheel in order to direct the light onto the axial entrance slit  $F_5$  of the Spectrograph.
2. Check that the light is entering the slit. Go into the RTD program and select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is not perfectly centered on the CCD detector, move slowly the spectrograph and center the silicon line on the central pixel of the detector. Exit the RTD program.
3. Go to the program MOVE AND CALIBRATE and calibrate the spectrograph at  $521\text{cm}^{-1}$ .

### C. Coupling the Double Subtractive Foremonochromator and the Spectrograph

Go into the RTD program and select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is not perfectly centered on the detector, adjust the mirror M5 by carefully turning the screw A in order to center the line at  $521\text{cm}^{-1}$  or on the central pixel of the detector. To access the adjustment screw of this mirror M5, it is necessary to remove the cover protecting the scanning mechanism of the T64000, that is at the back of the apparatus.

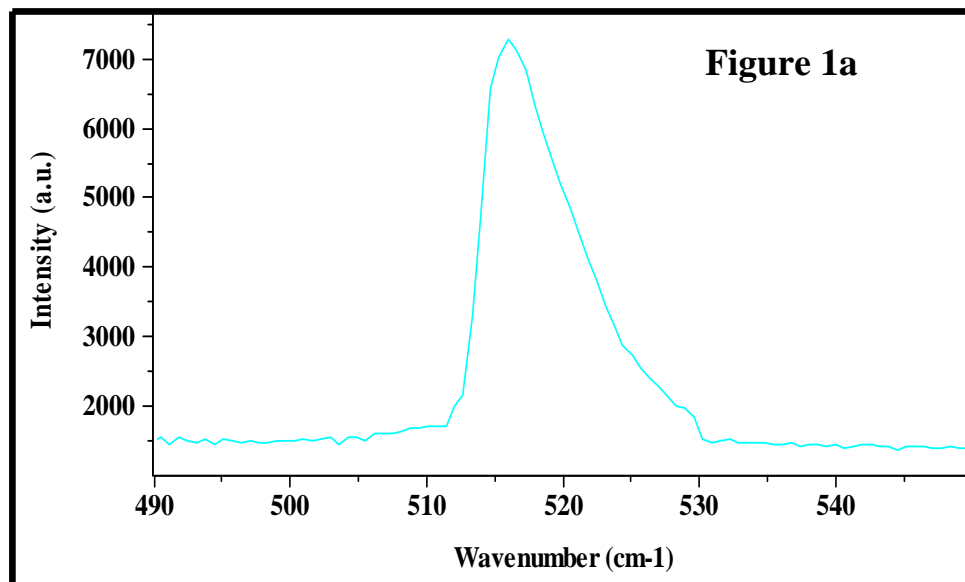


## Configuration 4

### Basic T64000 (Double Subtractive and Spectrograph)

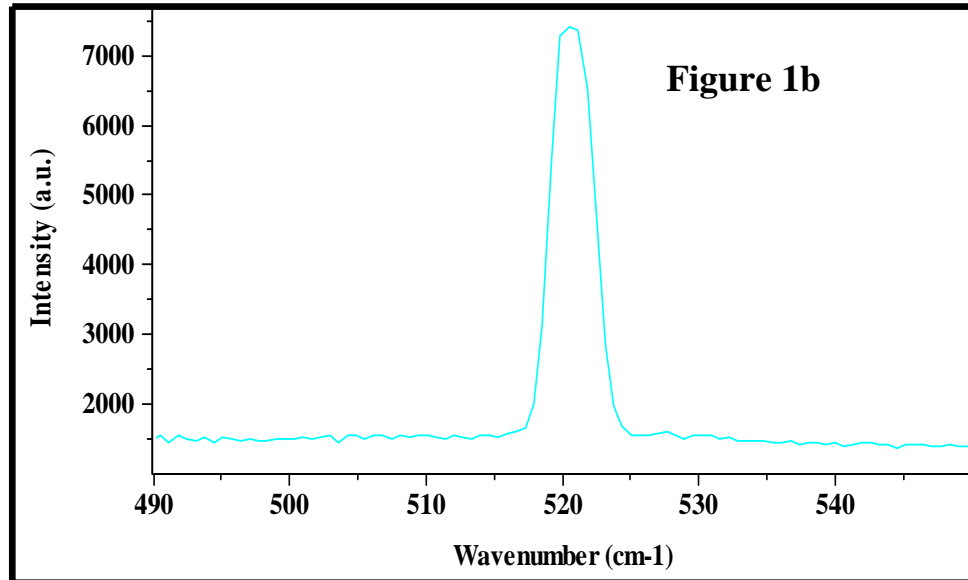
#### A. Calibration of the Double Subtractive Foremonochromator

1. In the program MOVE AND CALIBRATE, choose the central position  $521\text{cm}^{-1}$ . Both monochromators will be positioned at this frequency.
2. Set  $F_1 = 100\mu\text{m}$   
 $F_{i1/2} = 500\mu\text{m}$   
 $F_{i2/3} = 2\text{mm}$   
 Go into the RTD program and select the integration time and the X, Y expansion that enables a clear display of the silicon line.
3. Progressively close the slit  $F_{i1/2}$  down  $100\mu\text{m}$ .  
 If this line is asymmetric, it is not well centered on slit  $F_{i1/2}$ .  
 (Refer to Figure 1a).



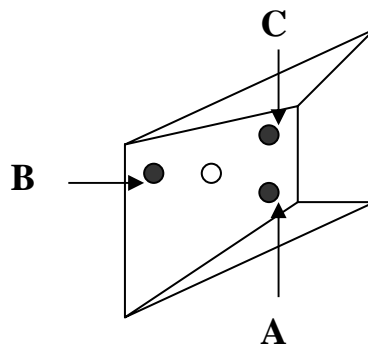
Adjust the central wavenumber of the foremonochromator, (by moving it and **not by adjusting the collimator  $O_{1,1}$  or  $O_{1,2}$** ) in

order to center the line on the slit  $F_{i1/2}$ . When the line is correctly centered, one can see on the spectrum the symmetry of the silicon line. (Refer to Figure 1b).



Reduce the height limiter of slit  $F_{i1/2}$ . If there is a loss of signal when the height limiter of  $F_{i1/2}$  is larger than that of  $F_1$ , carefully turn screw B on the mirror M2 in order to recover the maximum of the signal.

These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.

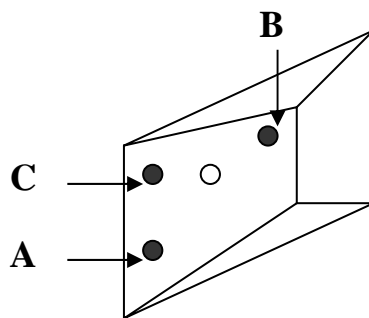


4. Progressively close  $F_{i2/3}$  down  $100\mu\text{m}$ .  
Gradually reduce the slit width and the height limiter to their

minima while adjusting the mirror M3 to center the  $521\text{cm}^{-1}$  line on the slit and obtain the maximum signal. To center the  $521\text{cm}^{-1}$  line between the slit jaws and obtain the maximum signal, turn screw A. If there is a loss of intensity when closing the height limiter, turn screw B.

These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.

5. Exit the RTD program  
Go to the program MOVE & CALIBRATE and calibrate the foremonochromator at  $521\text{cm}^{-1}$



## B. Calibration of the Spectrograph

1. If this line is not perfectly centered on the CCD detector, move slowly the spectrograph and center the silicon line on the central pixel of the detector. Exit the RTD program.
2. Go to the program MOVE AND CALIBRATE and calibrate the spectrograph at  $521\text{cm}^{-1}$ .

**Annex 1b**

## Procedure for T64000 calibration and coupling with Labspec software

When following this procedure, use an emission line of a spectral lamp (for example, the 546.07nm line of Hg) or a Raman line (for example, the  $521\text{cm}^{-1}$  phonon line of silicon) or any other Raman line whose frequency is perfectly known.

For the convention of components notation, refer to the attached optical schematic.

In the procedure that follows, we will use the silicon line at  $521\text{cm}^{-1}$  for calibration.

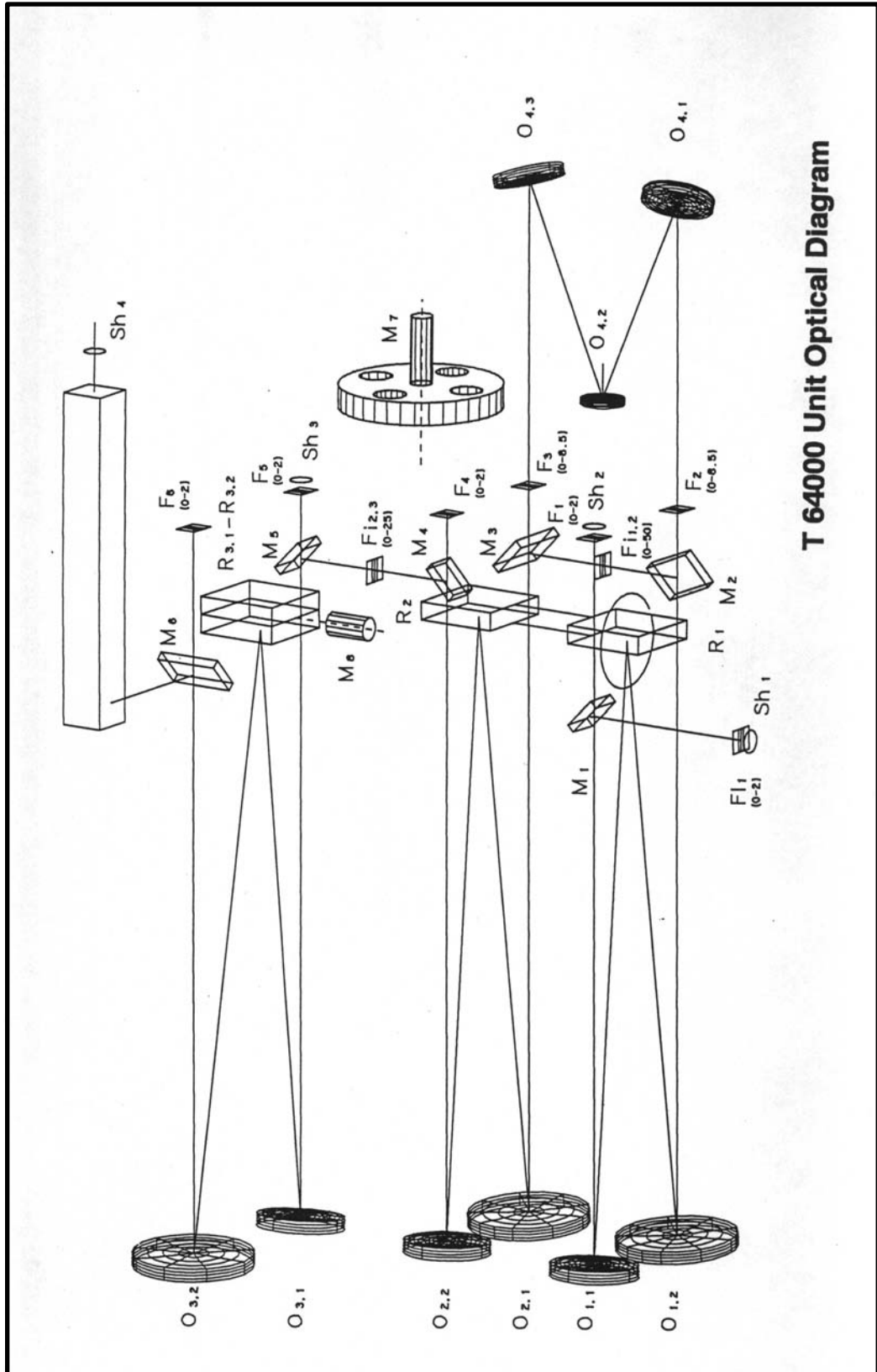
Four instrument configurations are covered:

- Configuration 1: basic T64000 (double subtractive + spectrograph) with the additive and single spectrograph options
- Configuration 2: basic T64000 with the additive option
- Configuration 3: basic T64000 with single spectrograph option
- Configuration 4: basic T64000

When making adjustments, the following instructions will explain how to tilt one of the mirrors indicated on the schematic. All mirrors are mounted on a 3-point holder with 3-adjustment screws. In general, one uses **A** to move the line vertically, and **B** to move it horizontally. In no case **C** should be touched.

### Important remark:

**During the installation, the coupling of the system over the whole spectral range has been done and adjusted by our service engineer. For the good working of the system over this large spectral range, we strongly recommend you do not touch any of the adjustment screws of the collimators in each monochromator ( $O_{1,1}$  ;  $O_{1,2}$  ;  $O_{2,1}$  ;  $O_{2,2}$  ;  $O_{3,1}$  ;  $O_{3,2}$ )**



T 64000 Unit Optical Diagram



## Configuration 1

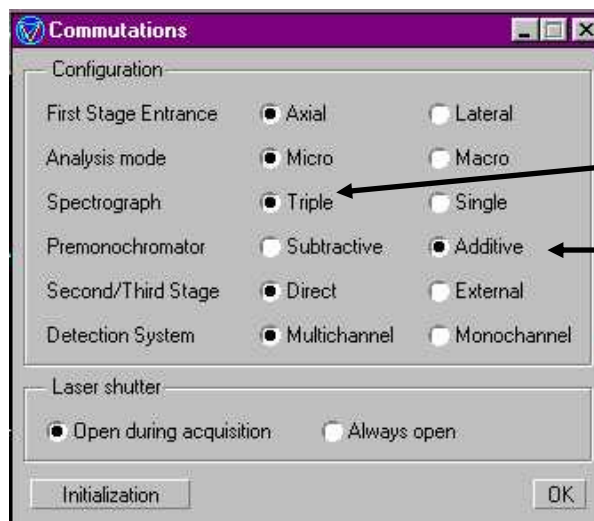
### Basic T64000 (Double Subtractive + Spectrograph) with the Additive and Single Spectrograph options

#### A. Calibration of the Double Additive Foremonochromator

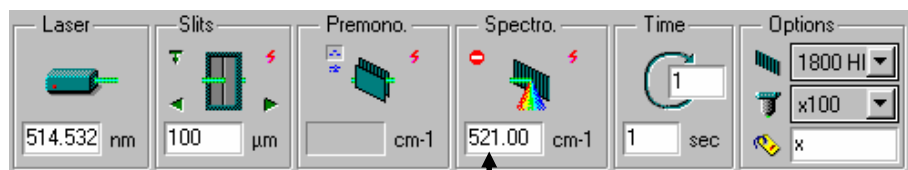
1. Click on the Commutations icon



A new display appears on the screen, select the option "Premonochromator Additive" and "Spectrograph Triple".



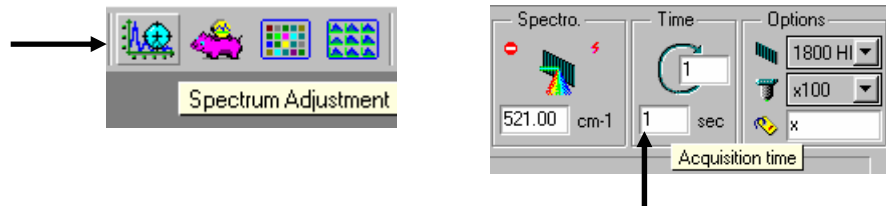
In this configuration, only the position (in wavelength or in wavenumber) of the Spectrograph is indicated, which is the common position of both Spectrograph and Foremonochromator.



2. Move the Spectrograph to the  $521\text{cm}^{-1}$  position.

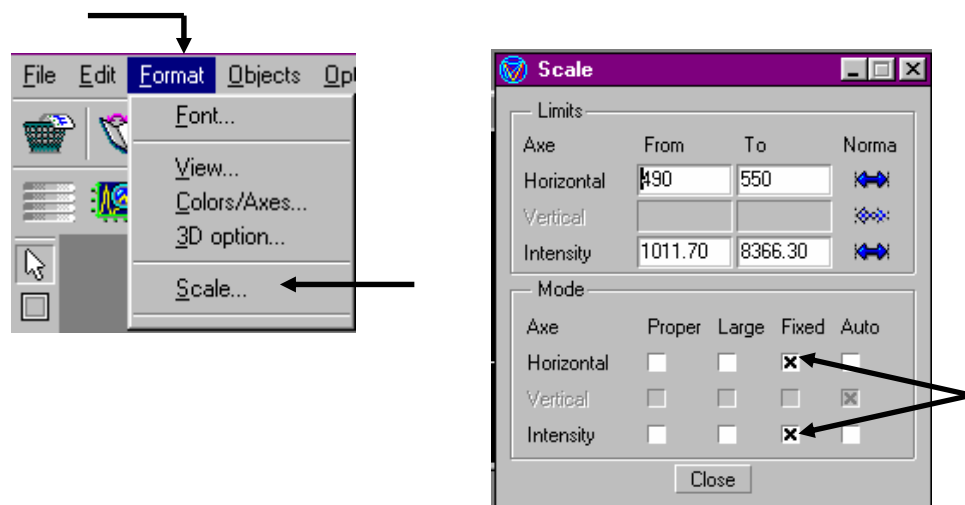
- Set  $F_1 = 100\mu\text{m}$
- $F_2 = 500\mu\text{m}$
- $F_3 = F_{i2/3} = 2\text{mm}$

Click on the Spectrum Adjustment icon to start continuous acquisition.

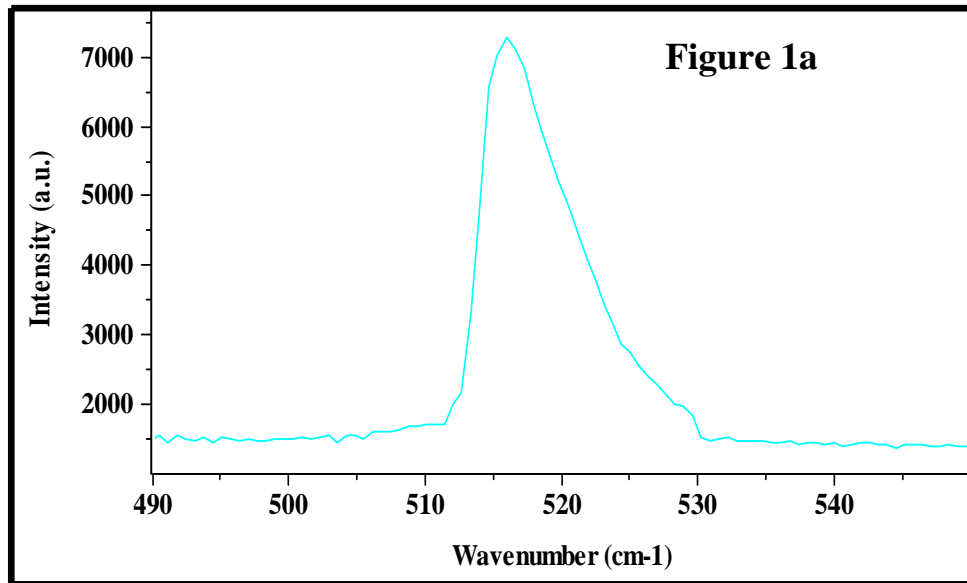


Select the acquisition time and the X, Y expansion that enables a clear display of the silicon line.

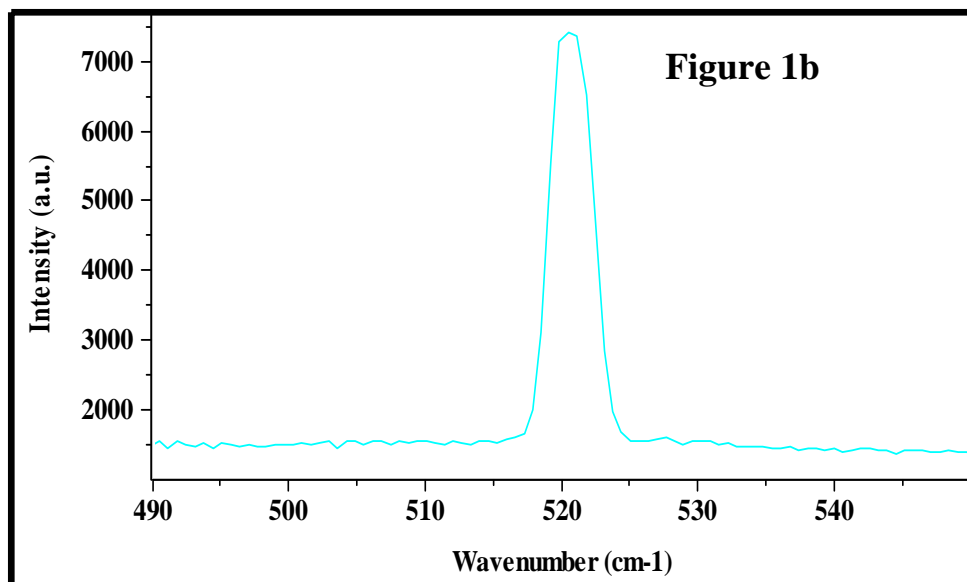
**To keep at any time the fixed scale during acquisition, select "Format" then "Scale". Validate the "Fixed mode" on the next display.**



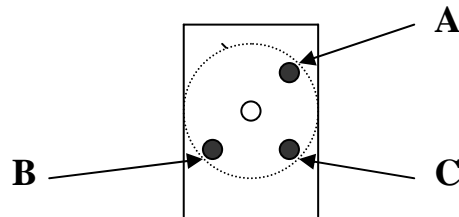
If the silicon line is asymmetric, it is not well centered on slit  $F_2$ . (Refer to Figure 1a)



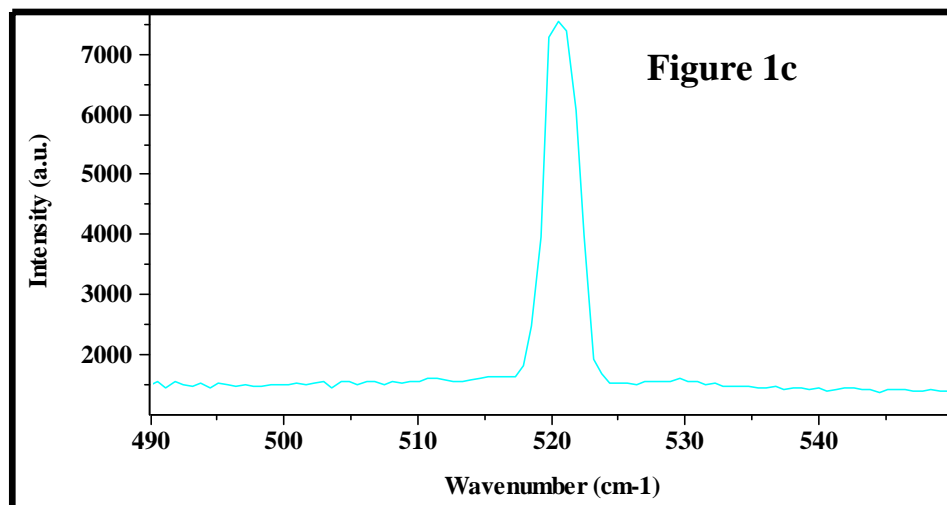
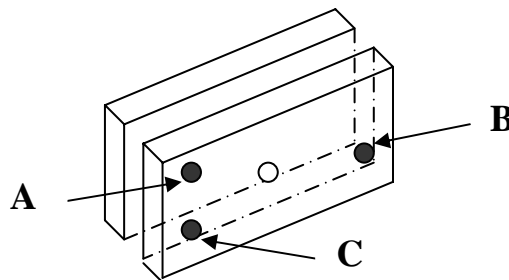
3. Progressively close  $F_2$  down  $100\mu\text{m}$ .  
Adjust the central wavenumber of the foremonochromator, (by moving it and **not by adjusting the collimator  $O_{1,1}$  or  $O_{1,2}$** ) in order to center the line on the slit  $F_2$ . When the line is correctly centered on the slit, one can see on the spectrum the symmetry of the silicon line. (Refer to Figure 1b)



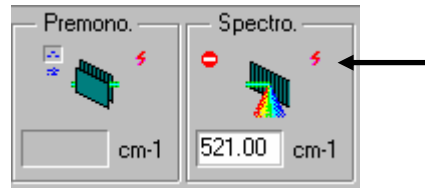
4. Progressively close  $F_3$  down  $100\mu\text{m}$ .  
 The line should keep the same profile and the same intensity as above. If not, adjust the mirror  $O_{4,2}$  by carefully turning the screw labeled A in the hereunder drawing in order to obtain the maximum signal. Reduce the height limiter of slit  $F_3$ . If there is a loss of signal when the height limiter of  $F_3$  is larger than that of  $F_2$  or  $F_1$ , carefully turn screw B in order to recover the maximum of the signal.



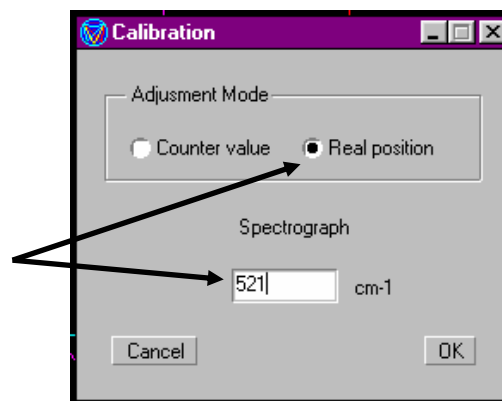
5. Progressively close  $F_{12/3}$  down  $100\mu\text{m}$   
 The line should be well resolved while maintaining the intensity. If not, adjust the mirror M4 by carefully turning screw A in order to obtain the maximum signal. Reduce the height limiter of  $F_{12/3}$  and, if necessary, turn screw B to maximize the signal.(Refer Figure 1c)



6. Calibration of the Double Additive foremonochromator  
Click on the Calibration icon in the Spectro box.



Enter the real value 521cm<sup>-1</sup> for Spectrograph position.



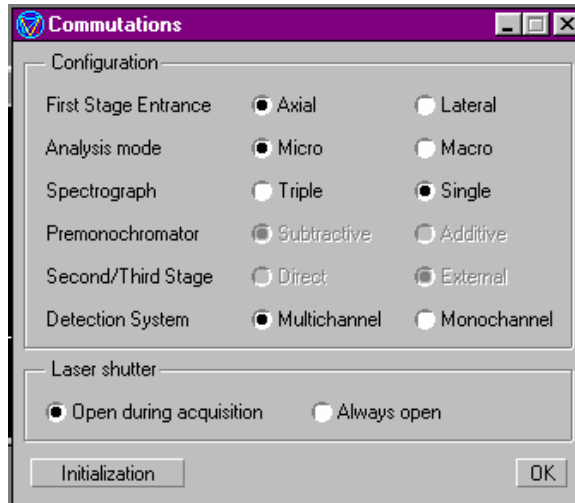
Remember that in the Triple Additive configuration, the Foremonochromator and the Spectrograph have the common position.

## **B. Calibration of the Spectrograph.**

1. Click on the Commutations icon



then select the option "Spectrograph Single"



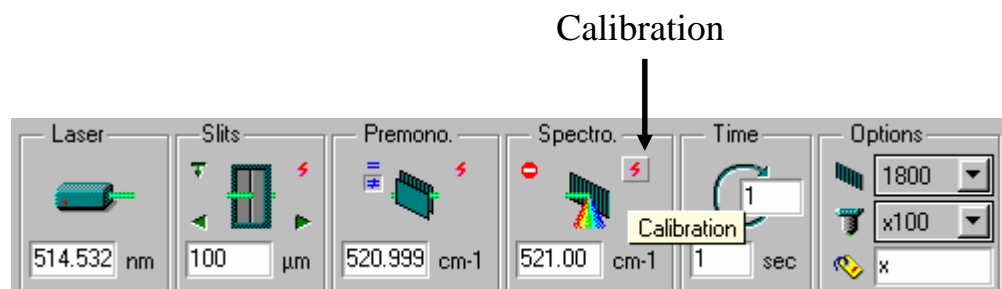
Turn the 4-position wheel in front of the entrance slit to direct light from the microscope or macro chamber, whichever is being used, onto slit F<sub>5</sub> of the spectrograph.

2. Check that the light is entering the slit.  
Click on the CCD Calibration icon to display the silicon spectrum.

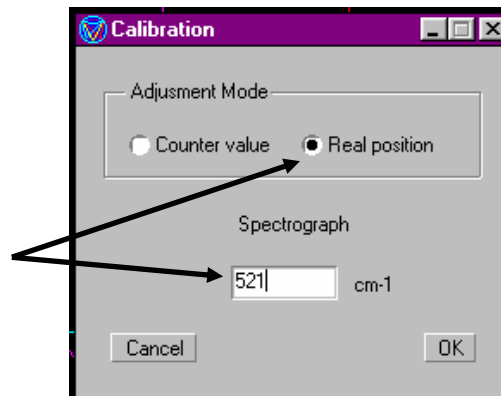


Select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is not perfectly centered on the CCD detector, move slowly the spectrograph and center the silicon line on the central pixel of the detector.  
Stop the CCD Calibration.

3. Calibrate the Spectrograph  
Click on the Calibration icon in the Spectro box.



Enter the real value  $521\text{cm}^{-1}$  for Spectrograph position.



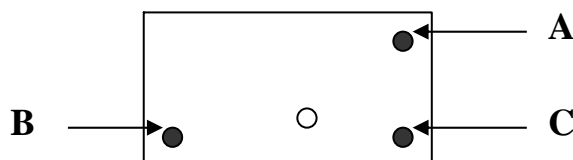
- Return to the Commutations icon and select the option "Spectrograph Triple" and "Premonochromator Additive"

### C. Coupling the Double Additive Foremonochromator and the Spectrograph.

Click on the CCD Calibration icon to display the silicon spectrum.



Select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is not perfectly centered on the CCD detector, adjust the mirror M5 by carefully turning the screw A in order to center the line on the central pixel of the detector. To access the adjustment screw of this mirror M5, it is necessary to remove the cover protecting the scanning mechanism of the T64000, that is at the back of the apparatus.



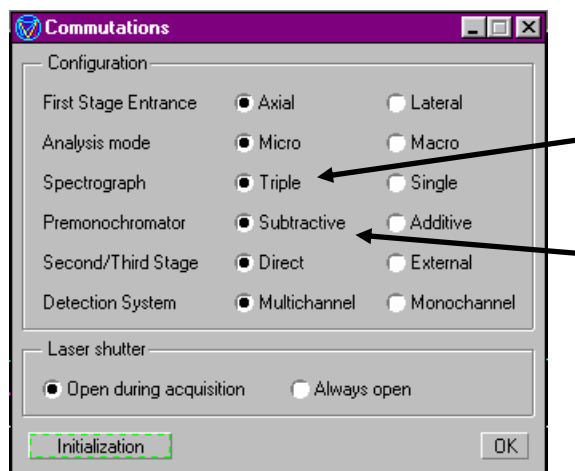
## D. Calibration of the Double Subtractive Foremonochromator

The monochromators being already calibrated in Triple Additive mode, none of the 2 the grating drives should be moved during the Triple Subtractive calibration.

1. Click on the Commutations icon

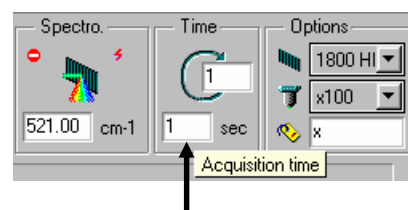


On the next display, select the option "Premonochromator Subtractive" and "Spectrograph Triple".



Set  $F_1 = 100\mu\text{m}$   
 $F_{i1/2} = 500\mu\text{m}$   
 $F_{i2/3} = 2\text{mm}$

2. Click on the Spectrum Adjustment icon to start continuous acquisition.



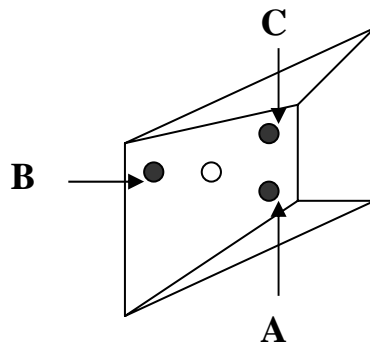


Select the acquisition time and the X, Y expansion that enables a clear display of the silicon line.

3. Progressively close  $F_{i1/2}$  down  $100\mu\text{m}$ .

If there is an asymmetry or a loss in signal, (refer Figure 1a) adjust the mirror M2 by carefully turning the screw A to maximize the signal. Reduce the height of slit  $F_{i1/2}$  while turning screw B on mirror M2 (if necessary) in order to keep the maximum of signal.

These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.



## E. Coupling the Double Subtractive Foremonochromator and the Spectrograph.

Progressively close  $F_{i2/3}$  down  $100\mu\text{m}$ .

Gradually reduce the slit width and the height limiter to their minima while adjusting the mirror M3 to center the  $521\text{cm}^{-1}$  line on the slit and obtain the maximum signal. To center the  $521\text{cm}^{-1}$  line between the slit jaws and obtain the maximum signal, turn screw A. If there is a loss of intensity when closing the height limiter, turn screw B. Refer to Figure 1d showing the superposition of the lines in Triple Subtractive and Triple Additive modes.

These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.

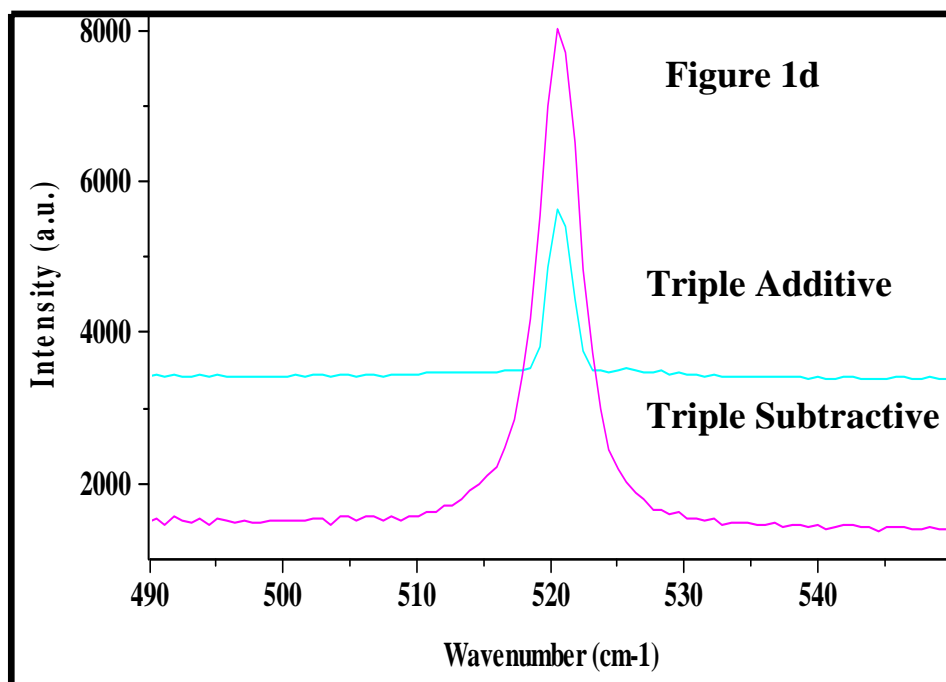
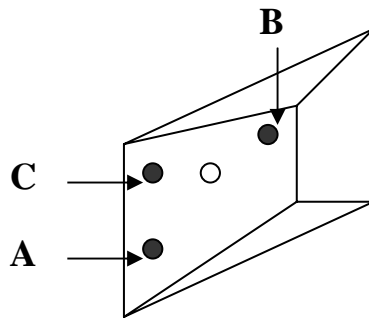
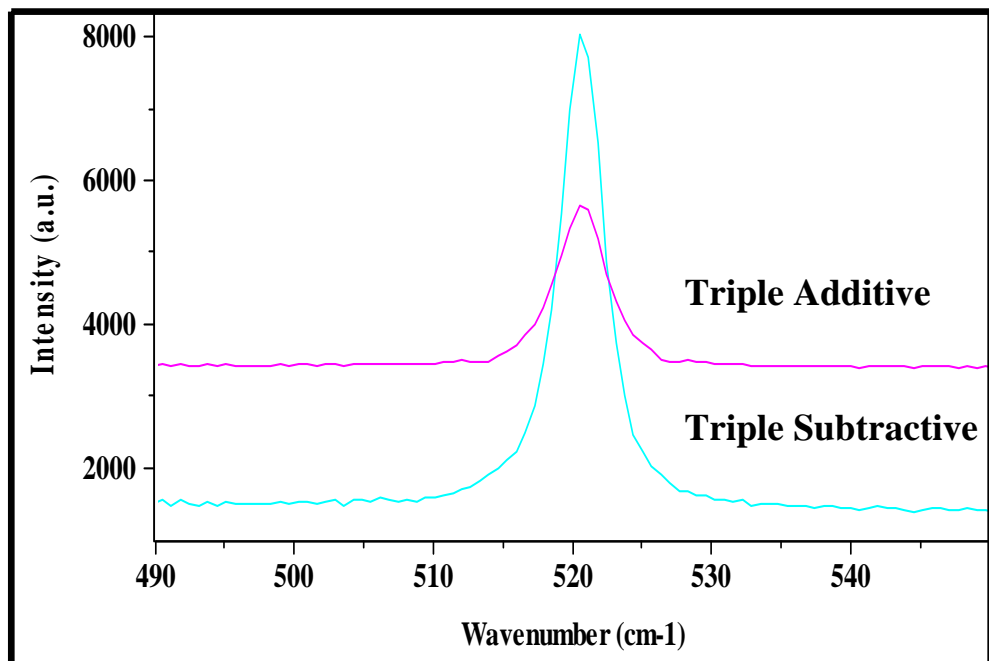
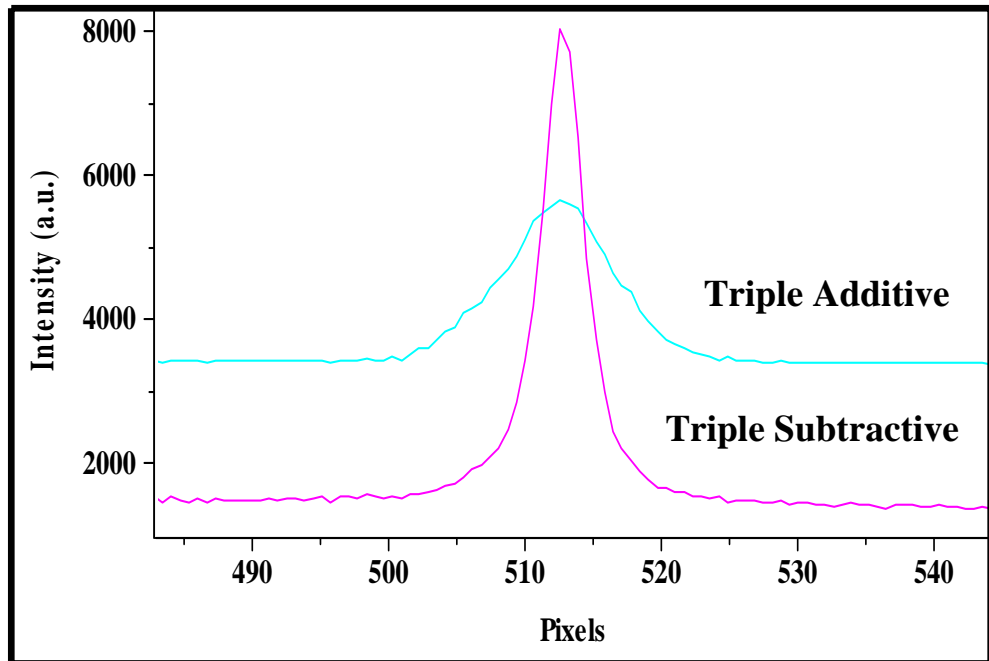


Figure 2 shows that, after this procedure, there is a perfect calibration of the two modes Triple Additive and Triple Subtractive, in the normal conditions of use; that is to say, with the intermediate slits wide enough to achieve full spectral coverage on the multichannel detector.



**Figure 2**

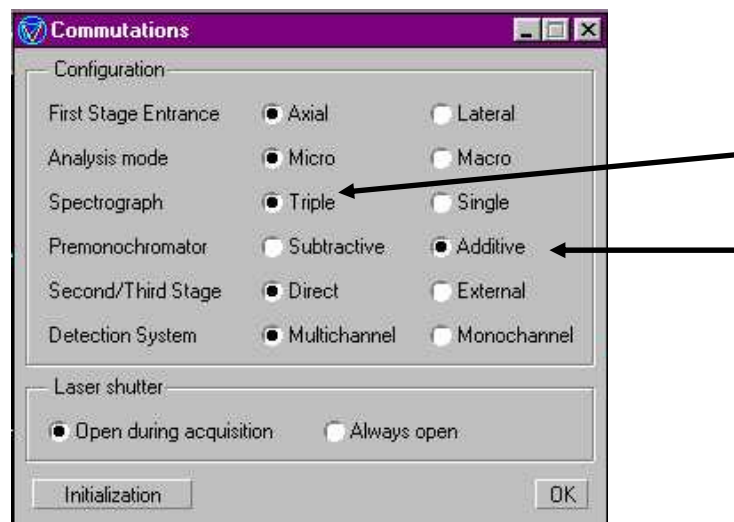
## Configuration 2 Basic T64000 with the Additive option

### A. Calibration of the Double Additive Foremonochromator

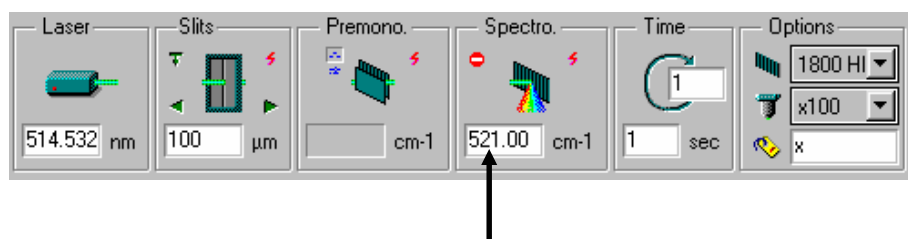
1. Click on the Commutations icon



On the next display, select the option "Premonochromator additive" and "Spectrograph Triple".



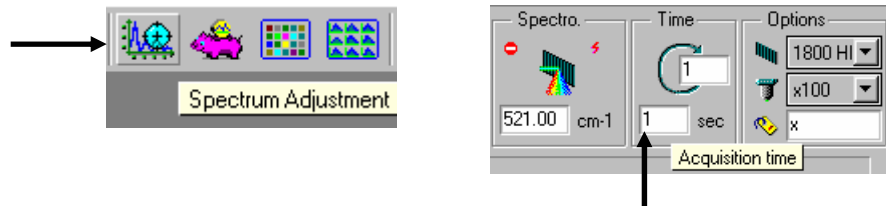
In this configuration, only the position (in wavelength or in wavenumber) of the Spectrograph is indicated, which is the common position of both Spectrograph and Foremonochromator.



2. Move the Spectrograph to the  $521\text{cm}^{-1}$  position.

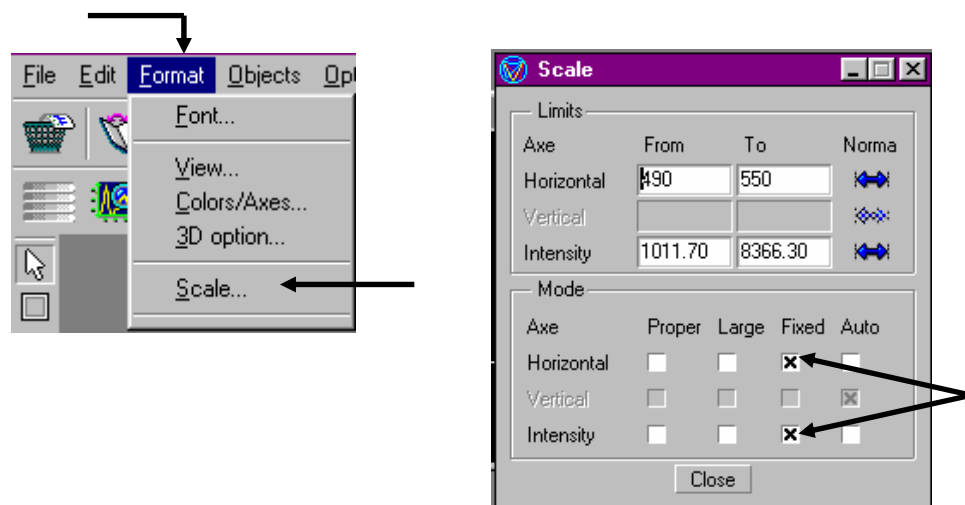
- Set  $F_1 = 100\mu\text{m}$
- $F_2 = 500\mu\text{m}$
- $F_3 = F_{i2/3} = 2\text{mm}$

Click on the Spectrum Adjustment icon to start continuous acquisition.

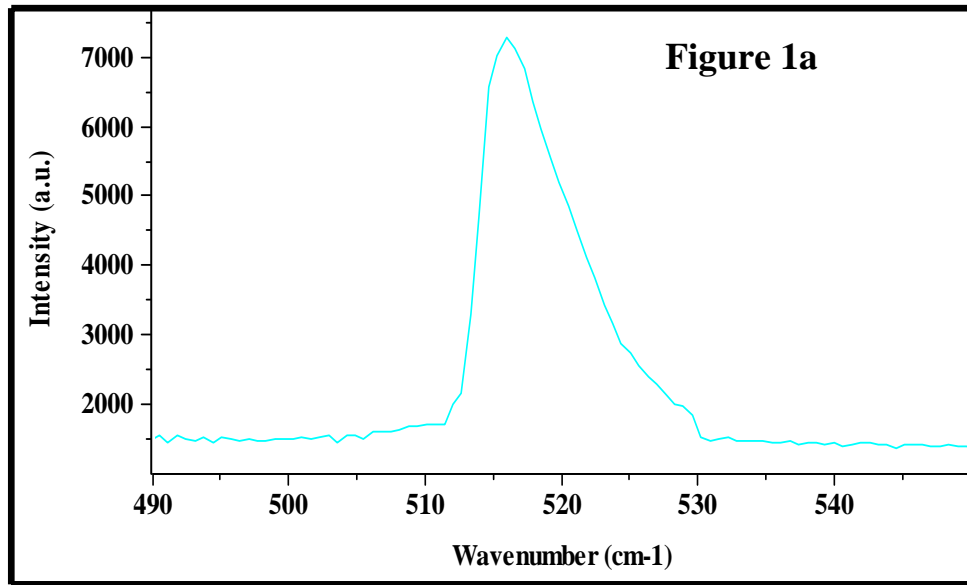


Select the acquisition time and the X, Y expansion that enables a clear display of the silicon line.

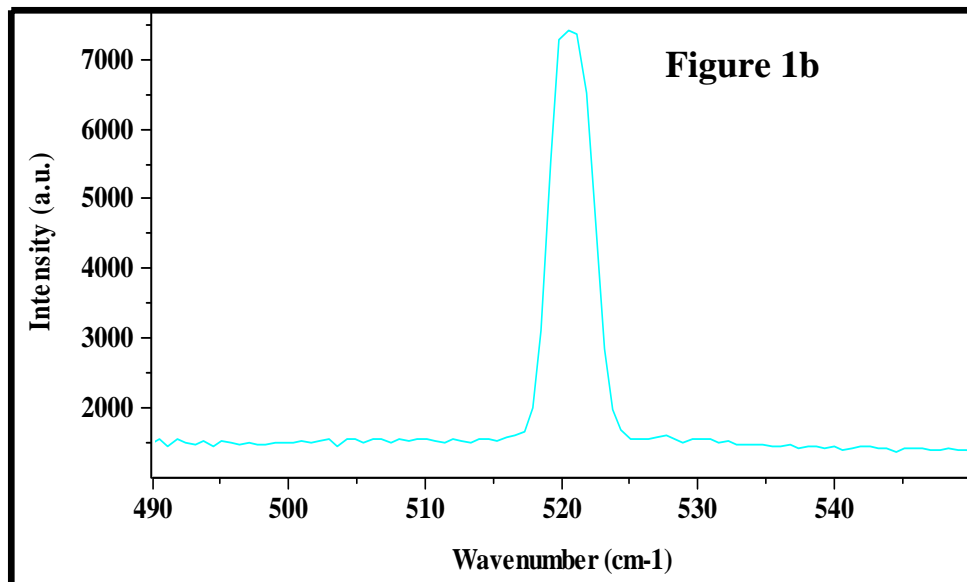
**To keep at any time the fixed scale during acquisition, select "Format" then "Scale". Validate the "Fixed mode" on the next display.**



If the silicon line is asymmetric, it is not well centered on slit  $F_2$ . (Refer to Figure 1a)

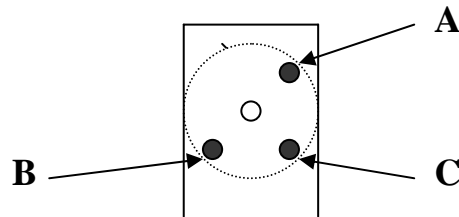


3. Progressively close  $F_2$  down  $100\mu\text{m}$ .  
 Adjust the central wavenumber of the foremonochromator, (by moving it and **not by adjusting the collimator  $O_{1,1}$  or  $O_{1,2}$** ) in order to center the line on the slit  $F_2$ . When the line is correctly centered on the slit, one can see on the spectrum the symmetry of the silicon line. (Refer to Figure 1b)



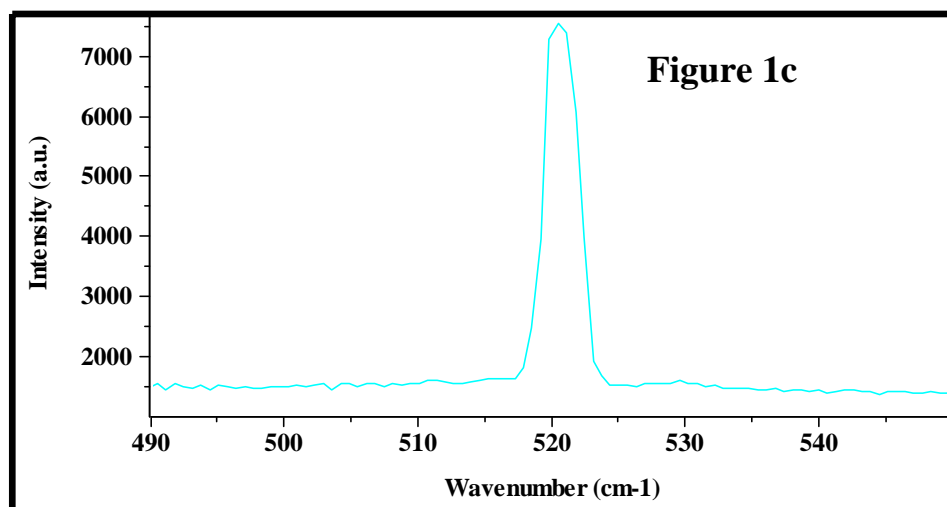
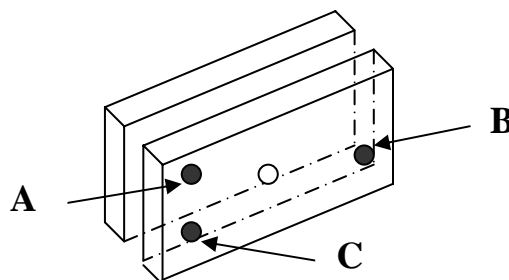
4. Progressively close  $F_3$  down  $100\mu\text{m}$ .

The line should maintain the same profile and the same intensity as above. If not, adjust the mirror  $O_{4,2}$  by carefully turning the screw labeled A in the hereunder drawing in order to obtain the maximum signal. Reduce the height limiter of slit  $F_3$ . If there is a loss of signal when the height limiter of  $F_3$  is larger than that of  $F_2$  or  $F_1$ , carefully turn screw B in order to recover the maximum of the signal.

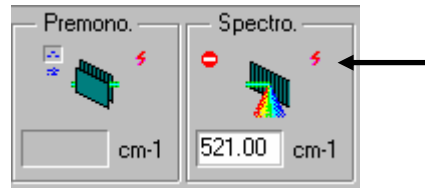


5. Progressively close  $F_{12/3}$  down  $100\mu\text{m}$

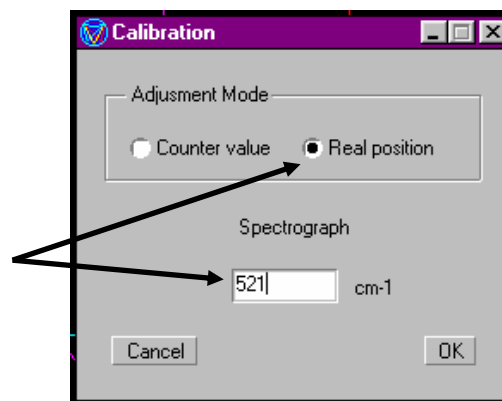
The line should be well resolved while maintaining the intensity. If not, adjust the mirror M4 by carefully turning screw A in order to obtain the maximum signal. Reduce the height limiter of  $F_{12/3}$  and, if necessary, turn screw B to maximize the signal.(Refer Figure 1c)



6. Calibration of the Double Additive foremonochromator  
Click on the Calibration icon in the Spectro box.



Enter the real value 521cm<sup>-1</sup> for Spectrograph position.



Remember that in the Triple Additive configuration, the Foremonochromator and the Spectrograph have the common position.

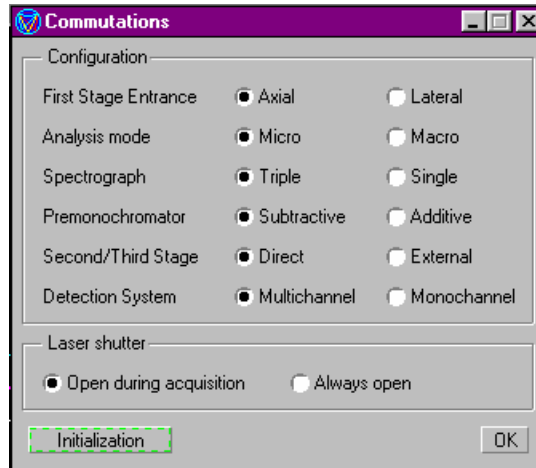
## B. Calibration of the Spectrograph.

1. Click on the Commutations icon



then select the option "Spectrograph Triple" and "Premonochromator Subtractive"





2. Click on the CCD Calibration icon to display the silicon spectrum.



Select the "Single moving" option.

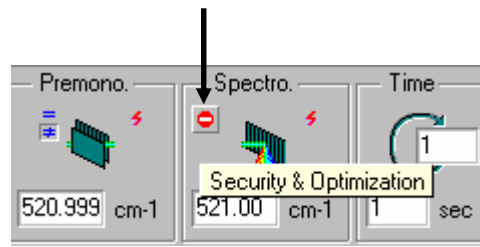


Select the integration time and the X, Y expansion that enables the clear display of the silicon line. If this line is not perfectly centered on the CCD detector, move slowly the spectrograph and center the silicon line on the central pixel of the detector.

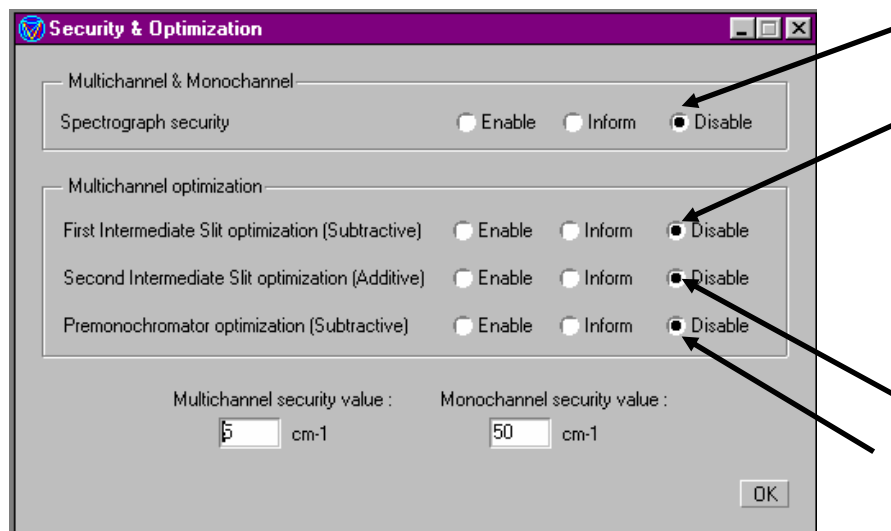
**Remark:**

To scan only the Spectrograph, the Foremonochromator sitting at the same position, it is necessary to disable all the functions "Security & Optimization".

To disable the Security & Optimization functions, click on the "Security & Optimization" icon.

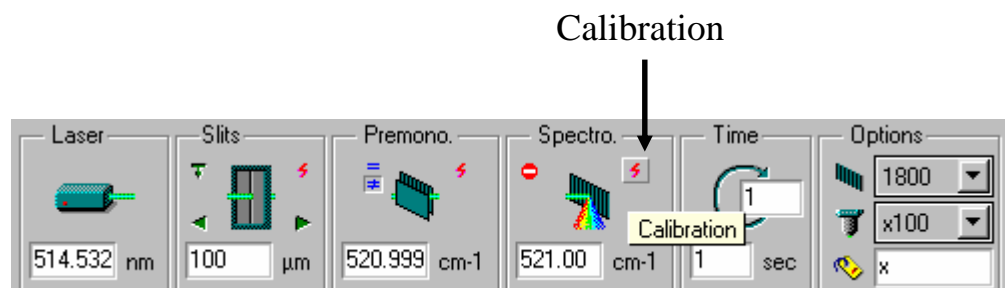


On the next display, select the option "Disable".

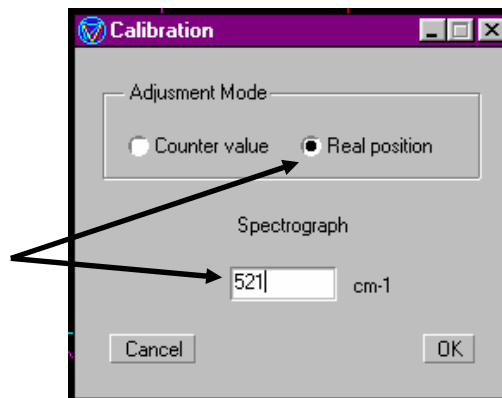


When the silicon line is perfectly centered on the central pixel of the CCD detector, stop the CCD Calibration.

3. Calibrate the Spectrograph.  
Click on the Calibration icon in the Spectro box.



Enter the real value 521cm<sup>-1</sup> for Spectrograph position.



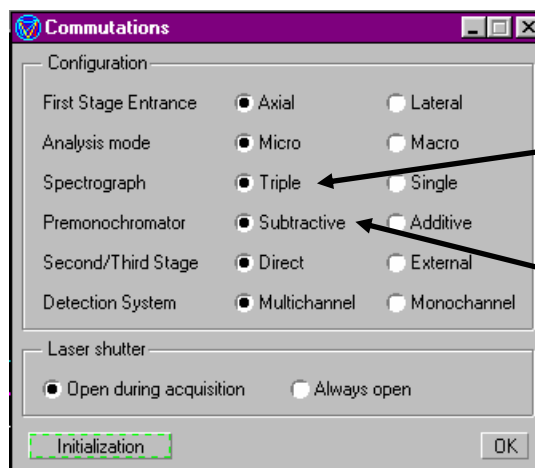
### C. Calibration of the Double Subtractive Foremonochromator

The monochromators being already calibrated in Triple Additive mode, none of the 2 the grating drives should be moved during the Triple Subtractive calibration.

1. Click on the Commutations icon

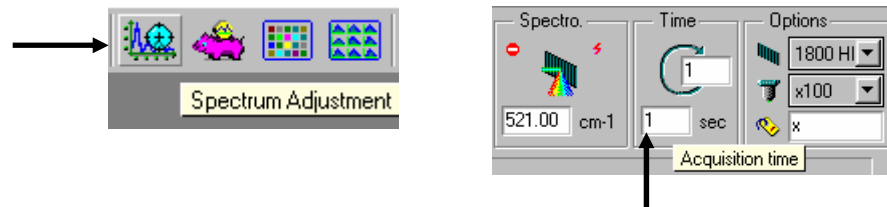


On the next display, select the option "Premonochromator Subtractive" and "Spectrograph Triple".



Set  $F_1 = 100\mu\text{m}$   
 $F_{i1/2} = 500\mu\text{m}$   
 $F_{i2/3} = 2\text{mm}$

- Click on the Spectrum Adjustment icon to start continuous acquisition.

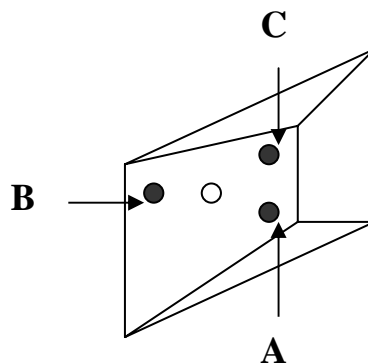


Select the acquisition time and the X, Y expansion that enables a clear display of the silicon line.

- Progressively close  $F_{i1/2}$  down  $100\mu\text{m}$ .

If there is an asymmetry or a loss in signal, (refer Figure 1a) adjust the mirror M2 by carefully turning the screw A to maximize the signal. Reduce the height of slit  $F_{i1/2}$  while turning screw B on mirror M2 (if necessary) in order to maintain the maximum of signal.

These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.



## D. Coupling the Double Subtractive Foremonochromator and the Spectrograph.

Progressively close  $F_{i2/3}$  down  $100\mu\text{m}$ .  
 Gradually reduce the slit width and the height limiter to their minima while adjusting the mirror M3 to center the  $521\text{cm}^{-1}$  line on the slit and obtain the maximum signal. To center the  $521\text{cm}^{-1}$  line between the slit jaws and obtain the maximum signal, turn screw A. If there is a loss of intensity when closing the height limiter, turn screw B. Refer to Figure 1d showing the superposition of the lines in Triple Subtractive and Triple Additive modes. These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.

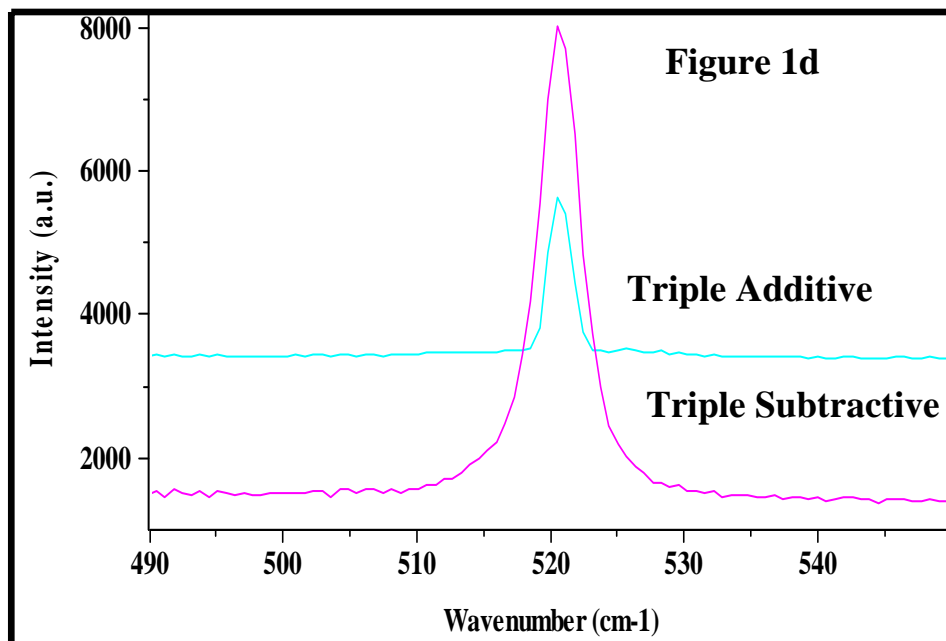
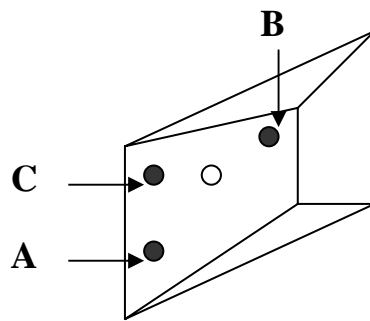
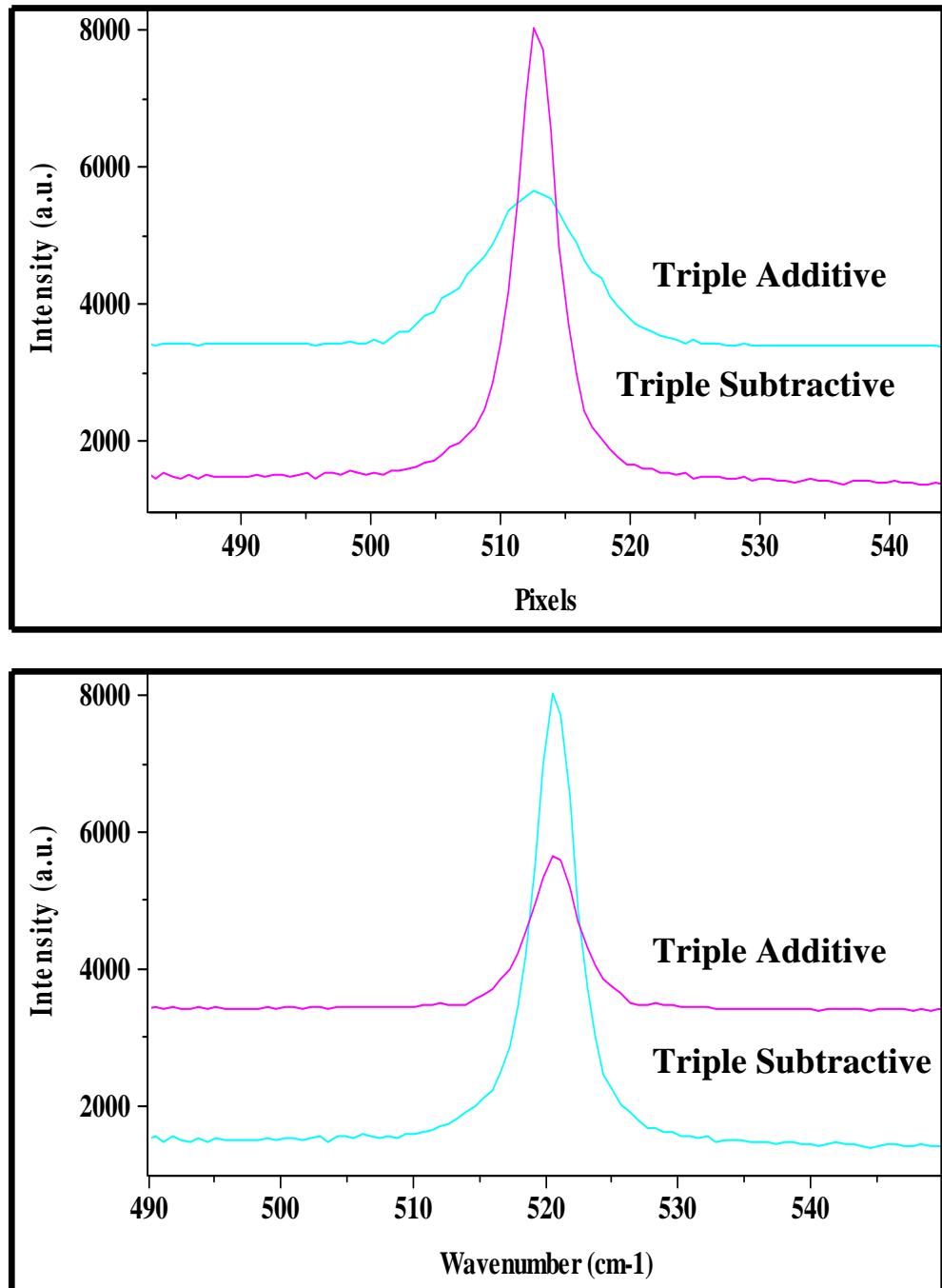


Figure 2 shows that, after this procedure, there is a perfect calibration of the two modes Triple Additive and Triple Subtractive, in the normal conditions of use; that is to say, with the intermediate slits wide enough to achieve full spectral coverage on the multichannel detector.



**Figure 2**

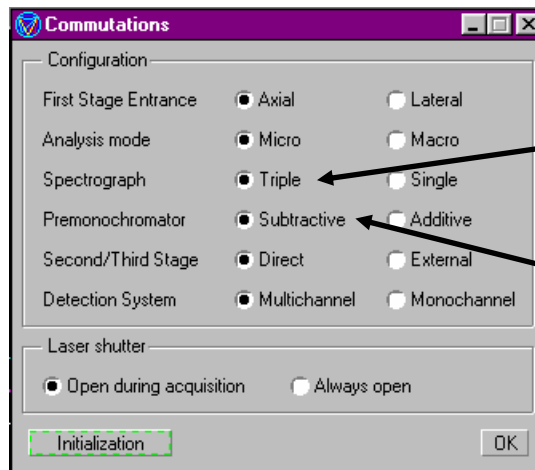
### Configuration 3 Basic T64000 with the Single Spectrograph option

#### A. Calibration of the Double Subtractive Foremonochromator

1. Click on the Commutations icon

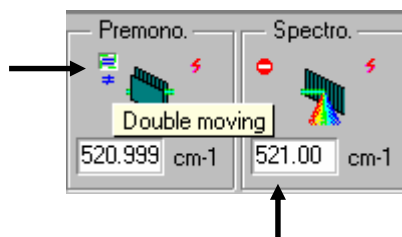


A new display appears on the screen, select the option "Premonochromator Subtractive" and "Spectrograph Triple".

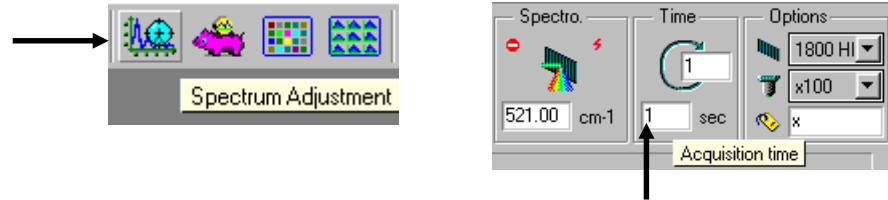


Set  $F_1 = 100\mu\text{m}$   
 $F_{i1/2} = 500\mu\text{m}$   
 $F_{i2/3} = 2\text{mm}$

2. Select the option "Double moving" and move the Spectrograph to the  $521\text{cm}^{-1}$  position.

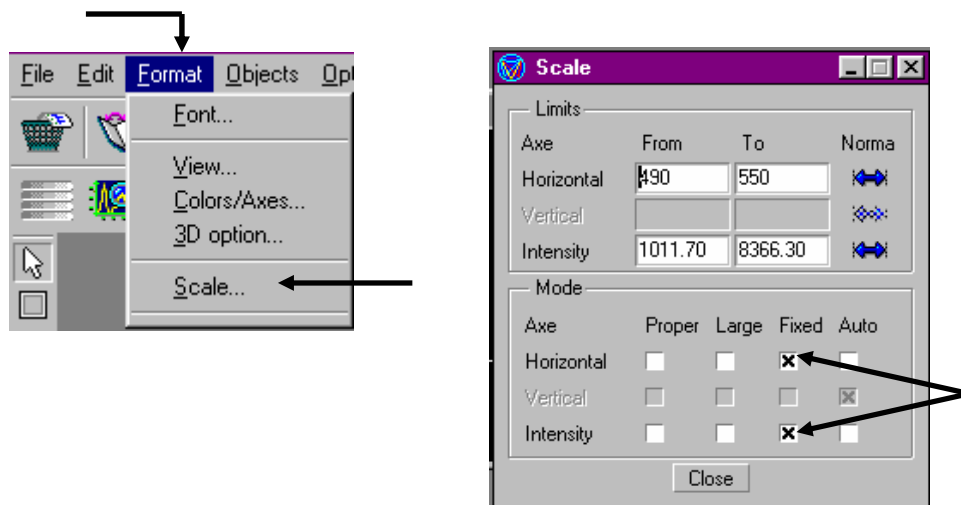


- Click on the Spectrum Adjustment icon to start continuous acquisition.



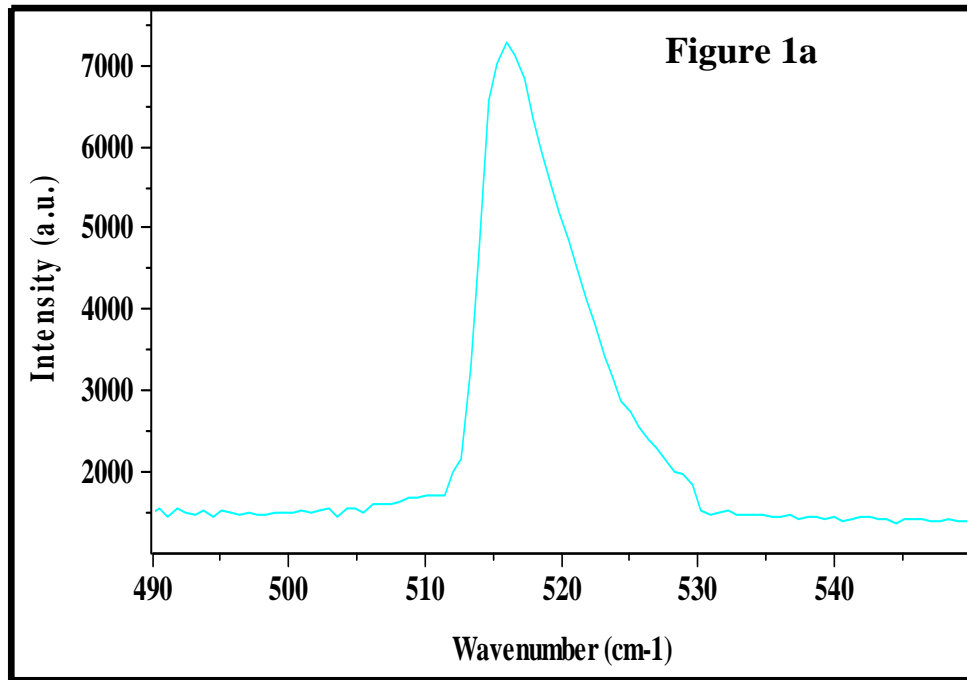
Select the acquisition time and the X, Y expansion that enables a clear display of the silicon line.

**To keep at anytime the fixed scale during acquisition, select "Format" then "Scale". Validate the "Fixed mode" on the next display.**



- Progressively close  $F_{i1/2}$  down  $100\mu\text{m}$ .  
If this line is asymmetric, it is not well centered on slit  $F_{i1/2}$ .  
(Refer to Figure 1a).



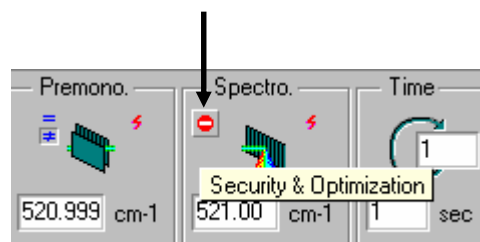


Adjust the central wavenumber of the foremonochromator, (by moving it and **not by adjusting the collimator O<sub>1,1</sub> or O<sub>1,2</sub>**) in order to center the line on the slit F<sub>11/2</sub>.

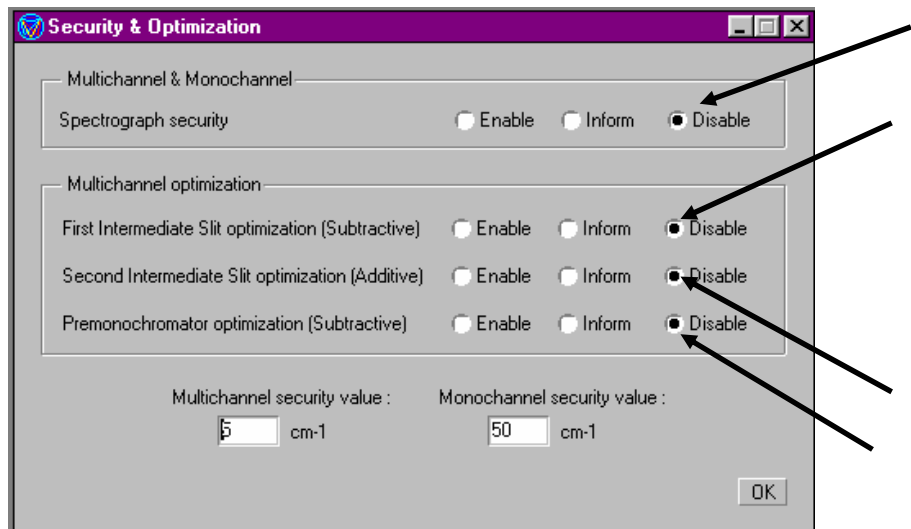
**Remark:**

To scan only the Foremonochromator, the Spectrograph sitting at the same position, it is necessary to disable all the functions "Security & Optimization".

To disable the Security & Optimization functions, click on the "Security & Optimization" icon.



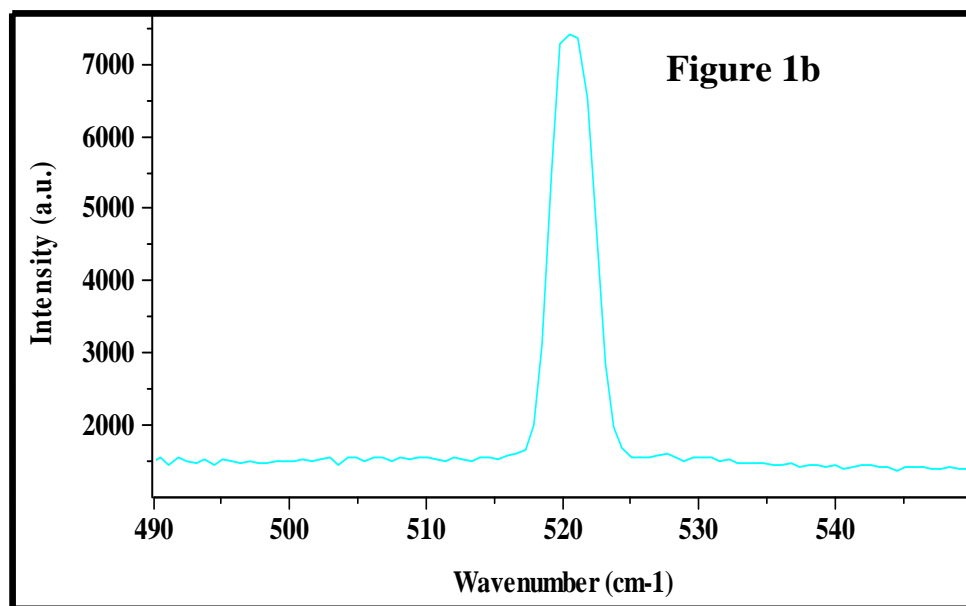
On the next display, select the option "Disable".



Select the "Single moving" option for only moving the foremonochromator.

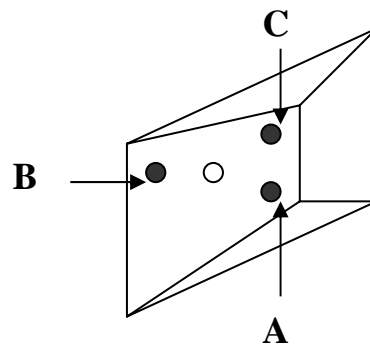


When the line is correctly centered on the slit  $F_{i1/2}$ , one can see on the spectrum the symmetry of the silicon line. (Refer to Figure 1b).



Reduce the height limiter of slit  $F_{i1/2}$ . If there is a loss of signal when the height limiter of  $F_{i1/2}$  is larger than that of  $F_1$ , carefully turn screw B on the mirror M2 in order to recover the maximum of the signal.

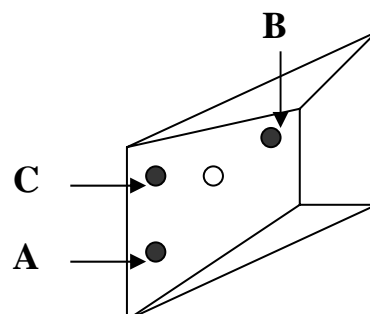
These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.



5. Progressively close  $F_{i2/3}$  down  $100\mu\text{m}$ .

Gradually reduce the slit width and the height limiter to their minima while adjusting the mirror M3 to center the  $521\text{cm}^{-1}$  line on the slit and obtain the maximum signal. To center the  $521\text{cm}^{-1}$  line between the slit jaws and obtain the maximum signal, turn screw A. If there is a loss of intensity when closing the height limiter turn screw B.

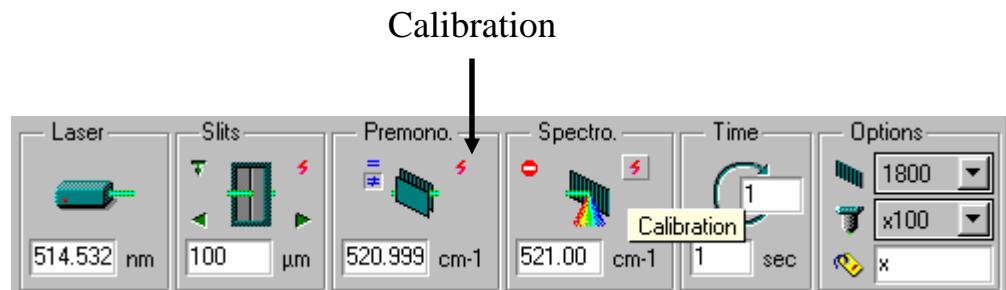
These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.



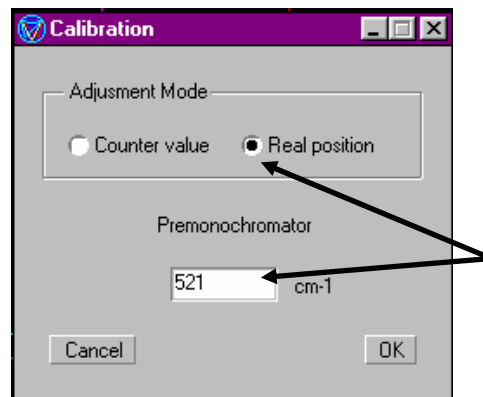
Stop the Spectrum Adjustment.

6. Calibrate the Foremonochromator.

Click on the Calibration icon in the Premono box.



Enter the real value 521cm<sup>-1</sup> for Premonochromator position.

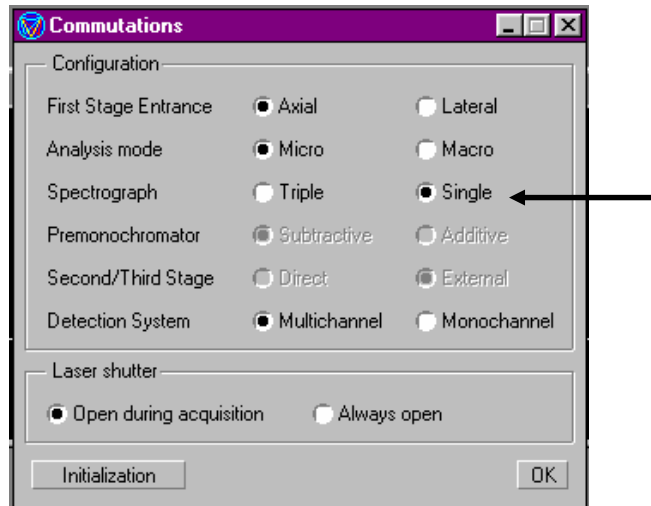


**B. Calibration of the Spectrograph.**

1. Click on the Commutations icon



then select the option "Spectrograph Single"



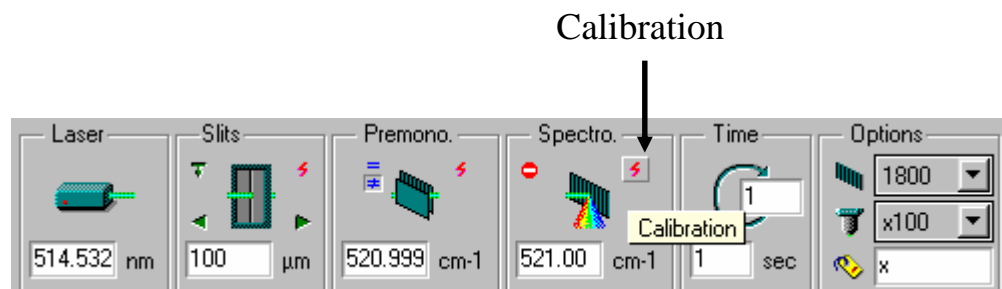
Turn the 4-position wheel in front of the entrance slit to direct light from the microscope or macro chamber, whichever is being used, onto slit F<sub>5</sub> of the spectrograph.

2. Check that the light is entering the slit. Click on the CCD Calibration icon to display the silicon spectrum.

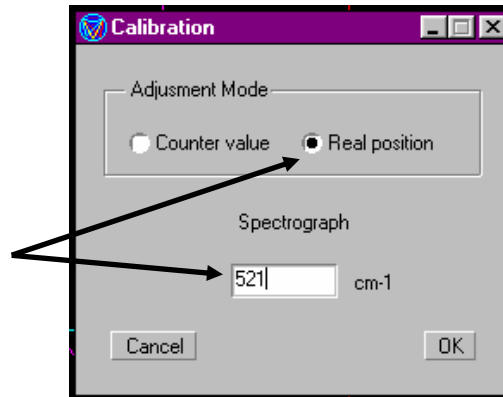


Select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is not perfectly centered on the CCD detector, move slowly the spectrograph and center the silicon line on the central pixel of the detector. Stop the CCD Calibration.

3. Calibrate the Spectrograph.  
Click on the Calibration icon in the Spectro box.



Enter the real value  $521\text{cm}^{-1}$  for Spectrograph position.

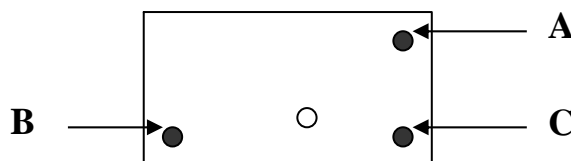


### C. Coupling the Double Subtractive Foremonochromator and the Spectrograph.

Click on the CCD Calibration icon to display the silicon spectrum.



Select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is not perfectly centered on the CCD detector, adjust the mirror M5 by carefully turning the screw A in order to center the line on the central pixel of the detector. To access the adjustment screw of this mirror M5, it is necessary to remove the cover protecting the scanning mechanism of the T64000, that is at the back of the apparatus.



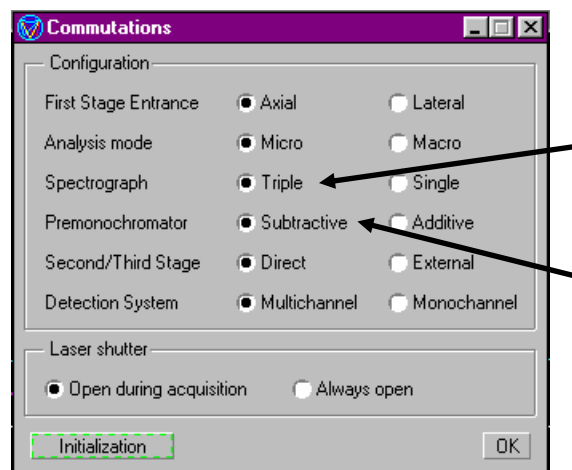
## Configuration 4 Basic T64000 (Double Subtractive and Spectrograph)

### A. Calibration of the Double Subtractive Foremonochromator

1. Click on the Commutations icon

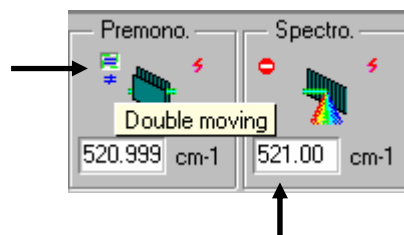


A new display appears on the screen, select the option "Premonochromator Subtractive" and "Spectrograph Triple".

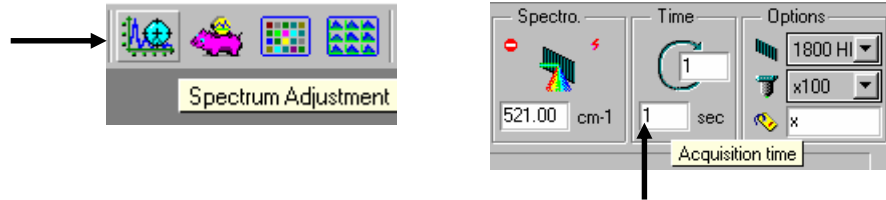


Set  $F_1 = 100\mu\text{m}$   
 $F_{i1/2} = 500\mu\text{m}$   
 $F_{i2/3} = 2\text{mm}$

2. Select the option "Double moving" and move the Spectrograph to the  $521\text{cm}^{-1}$ .

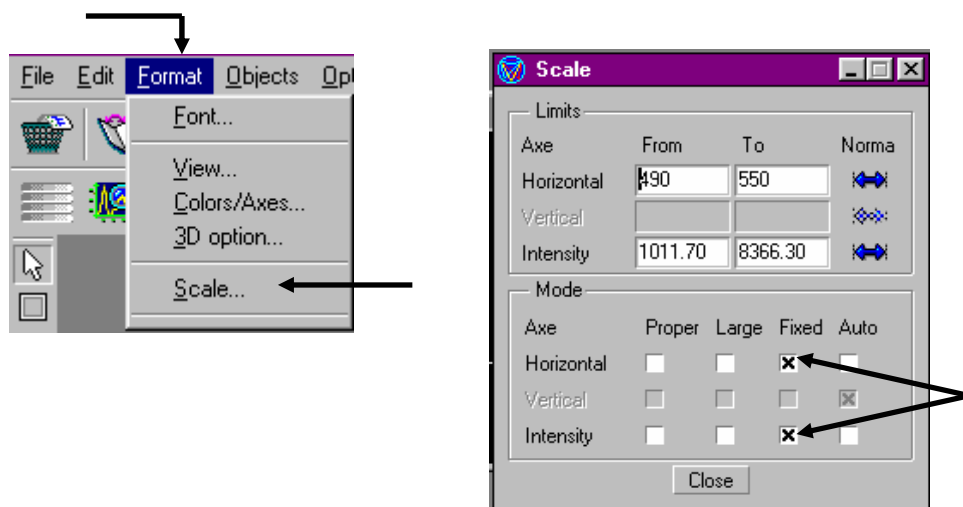


- Click on the Spectrum Adjustment icon to start continuous acquisition.



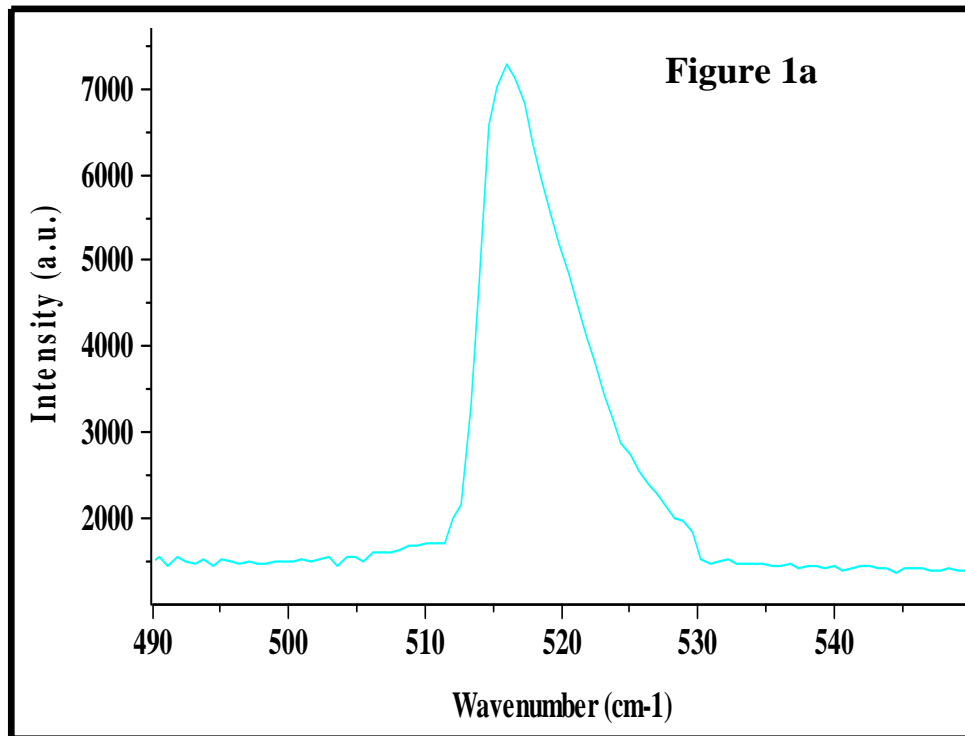
Select the acquisition time and the X, Y expansion that enables a clear display of the silicon line.

**To keep at anytime the fixed scale during acquisition, select "Format" then "Scale". Validate the "Fixed mode" on the next display.**

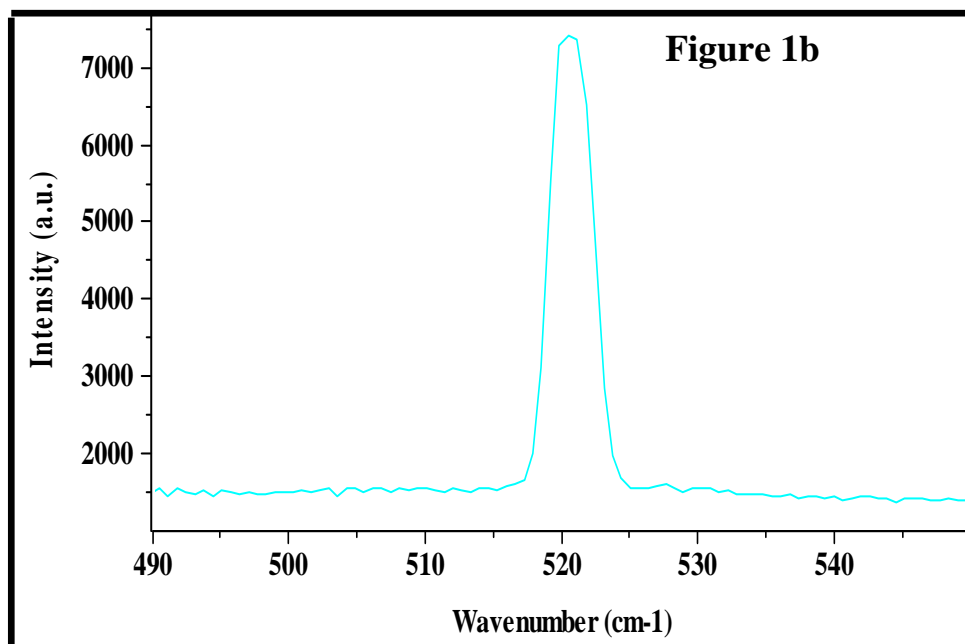


- Progressively close  $F_{i1/2}$  down  $100\mu\text{m}$ .  
If this line is asymmetric, it is not well centered on slit  $F_{i1/2}$ .  
(Refer to Figure 1a).



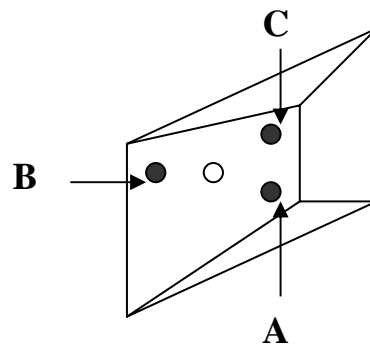


Adjust the central wavenumber of the foremonochromator, (by moving it and **not by adjusting the collimator  $O_{1,1}$  or  $O_{1,2}$** ) in order to center the line on the slit  $F_{i1/2}$ . When the line is correctly centered on the slit  $F_{i1/2}$ , one can see on the spectrum the symmetry of the silicon line. (Refer to Figure 1b).



Reduce the height limiter of slit  $F_{i1/2}$ . If there is a loss of signal when the height limiter of  $F_{i1/2}$  is larger than that of  $F_1$ , carefully turn screw B on the mirror M2 in order to recover the maximum of the signal.

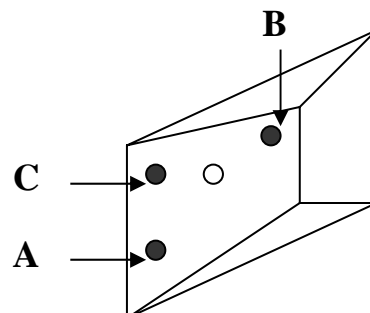
These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.



5. Progressively close  $F_{i2/3}$  down  $100\mu\text{m}$ .

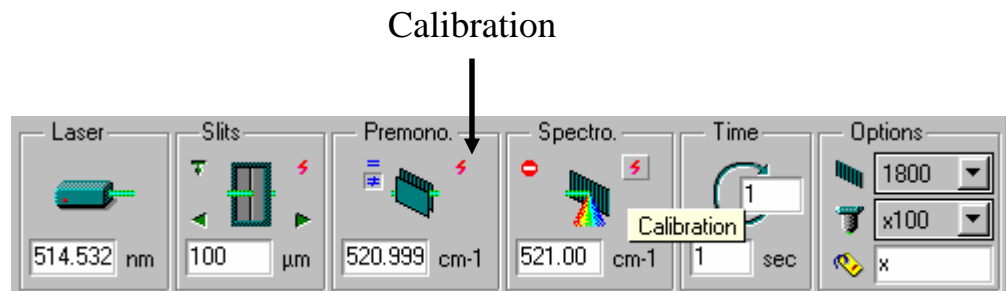
Gradually reduce the slit width and the height limiter to their minima while adjusting the mirror M3 to center the  $521\text{cm}^{-1}$  line on the slit and obtain the maximum signal. To center the  $521\text{cm}^{-1}$  line between the slit jaws and obtain the maximum signal, turn screw A. If there is a loss of intensity when closing the height limiter, turn screw B.

These screws, A, B and C are mounted on the casting and are visible from outside without removing any covers. Refer to the following figure.

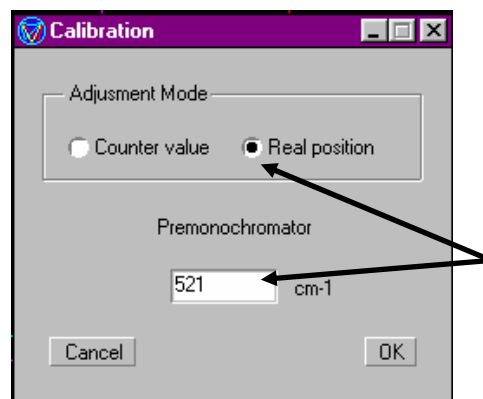


Stop the Spectrum Adjustment.

6. . Calibrate the Foremonochromator.  
Click on the Calibration icon in the Premono box.

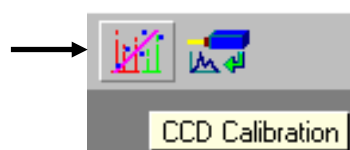


Enter the real value 521cm<sup>-1</sup> for Premonochromator position.

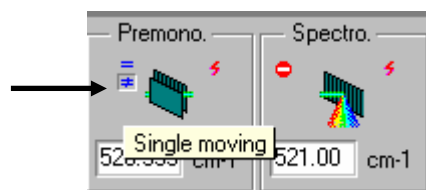


## B. Calibration of the Spectrograph

1. Click on the CCD Calibration icon to display the silicon spectrum.



Select the "Single moving" option.

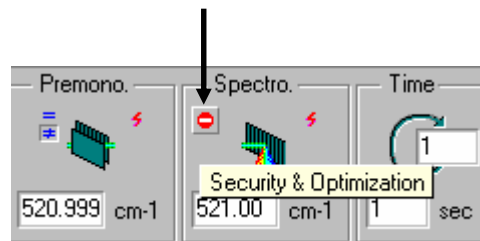


Select the integration time and the X, Y expansion that enables a clear display of the silicon line. If this line is not perfectly centered on the CCD detector, move slowly the spectrograph and center the silicon line on the central pixel of the detector.

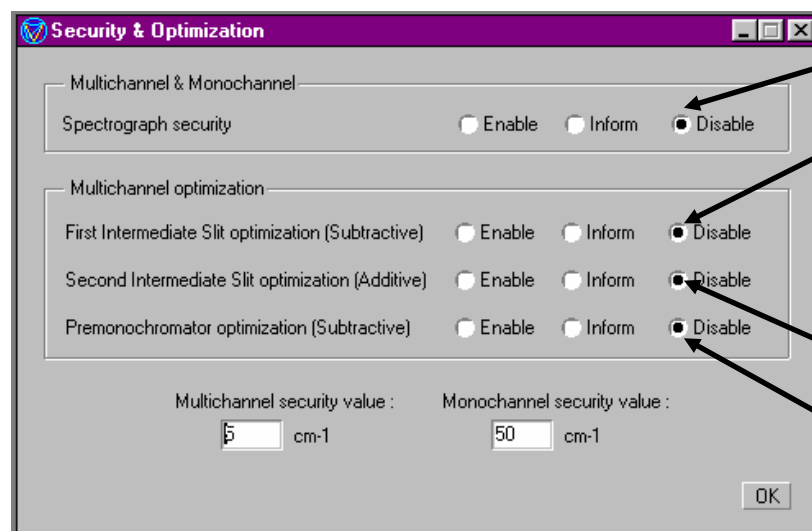
**Remark:**

To scan only the Spectrograph, the Foremonochromator sitting at the same position, it is necessary to disable all the functions "Security & Optimization".

To disable the Security & Optimization functions, click on the "Security & Optimization" icon.

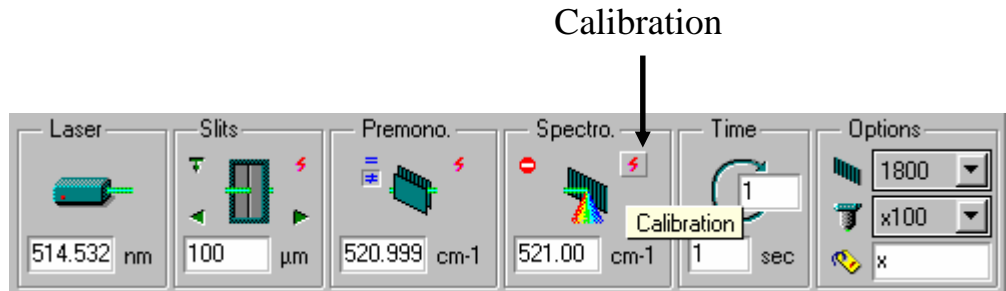


On the next display, select the option "Disable".

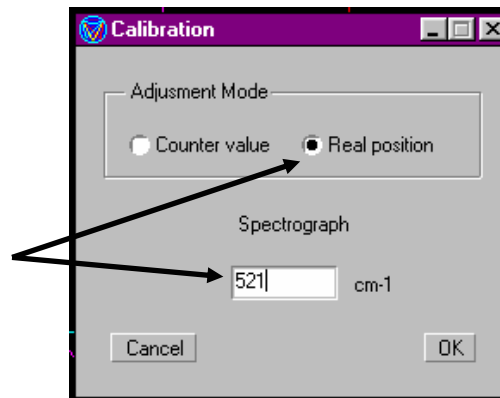


When the silicon line is perfectly centered on the central pixel of the CCD detector, stop the CCD Calibration.

2. Calibrate the Spectrograph.  
Click on the Calibration icon in Spectro box.



Enter the real value 521cm<sup>-1</sup> for Spectrograph position.



**Annex 2**

## Procedure for the realignment of the macro sample chamber

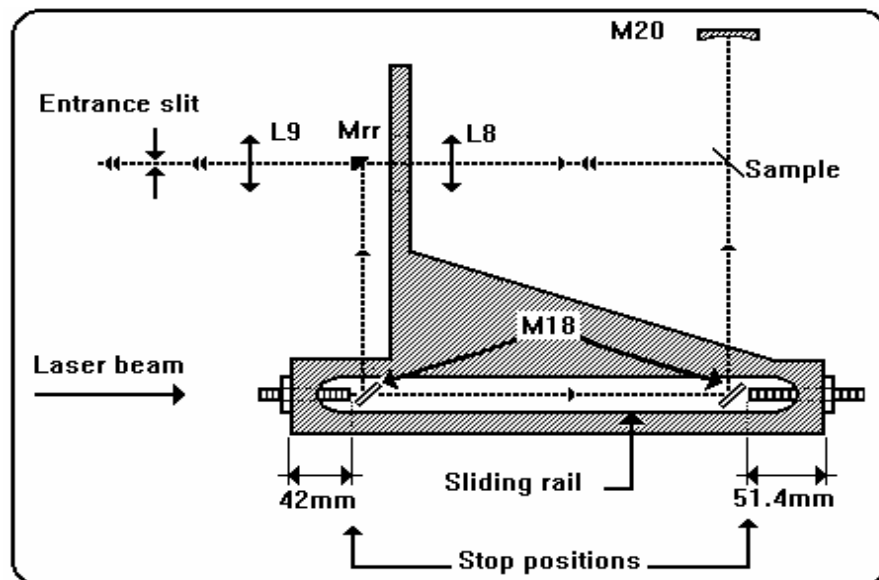
The T64000 system has been installed and implemented by an JY service engineer. The following procedure can be performed only if the laser beam has been centered from the laser entrance through the M<sub>10</sub> and M<sub>11</sub> mirrors (see Figure 1).

A. The first adjustments consist in aligning the incident optical axis of the laser and in centering it considering the entrance axis of the spectrometer defined by the centres of the entrance slit and of the collimator of the first stage.

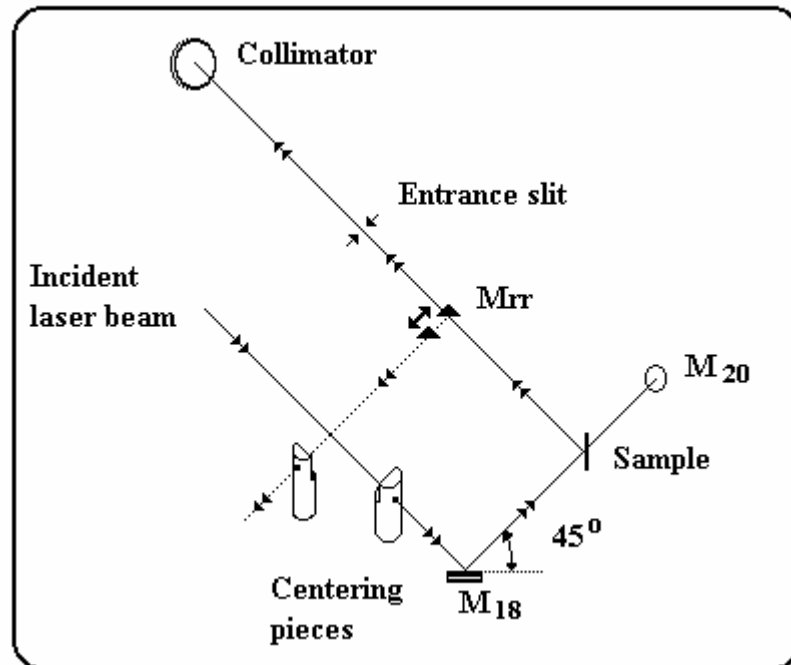
1. Remove the M<sub>18</sub> mirror from the sliding rail
2. Insert the two centering pieces on each end of the mirror sliding rail.  
Center the laser beam through the two centering pieces using the M<sub>16</sub> and M<sub>17</sub> mirrors.

### Remark

Verify, at first, that the left and right stop positions on the sliding rail are correctly positioned according to the following diagram.



3. Insert the  $M_{18}$  mirror at  $45^\circ$  on the right end side of the sliding rail.
4. Place the centering piece on the  $M_{20}$  mirror and adjust the laser beam on this centering piece with the  $M_{18}$  mirror.
5. Place a mirror (or a reflecting sample) on the sample location.
6. Remove the  $L_8$  and  $L_9$  lenses.
7. Place a centering piece on the entrance collimator of the first stage.
8. The laser beam must be centered on the entrance slit and on the collimator centering piece. If not, adjust the mirror (or the reflecting sample) on the sample location. The slit height must be at the minimum. The beam is perfectly centered if reducing the slitwidth the image on the collimator appears with perfect vertical diffracted spots.



9. Place and center the backscattering mirror  $M_{rr}$  on the laser beam. The reflected laser beam must be superposed to the direct beam. To control this last step, place a centering piece at  $90^\circ$  on the left end side of the sliding rail, the beam passing through the centering piece. If not, adjust the backscattering mirror  $M_{rr}$  (see Figure 2)
10. Remove the backscattering mirror and insert the  $L_9$  lens.
11. Adjust this lens to perfectly center the laser beam on the entrance slit and the collimator. The following procedure will improve the adjustment as the laser beam focusing is difficult to appreciate:



- a. Slowly close the slitwidth to display the diffraction spot on the collimator.
  - b. Move the L<sub>9</sub> lens to improve the beam focusing and reduce the diffraction spot. When it is possible, that means that the laser spot diameter is smaller on the entrance slit and then better focused. If it is not possible to reduce more the dimensions of the diffraction spot, the best focusing point is reached (the depth focus is intentionally long).
12. Insert and adjust the L<sub>8</sub> lens to keep the same beam centering.

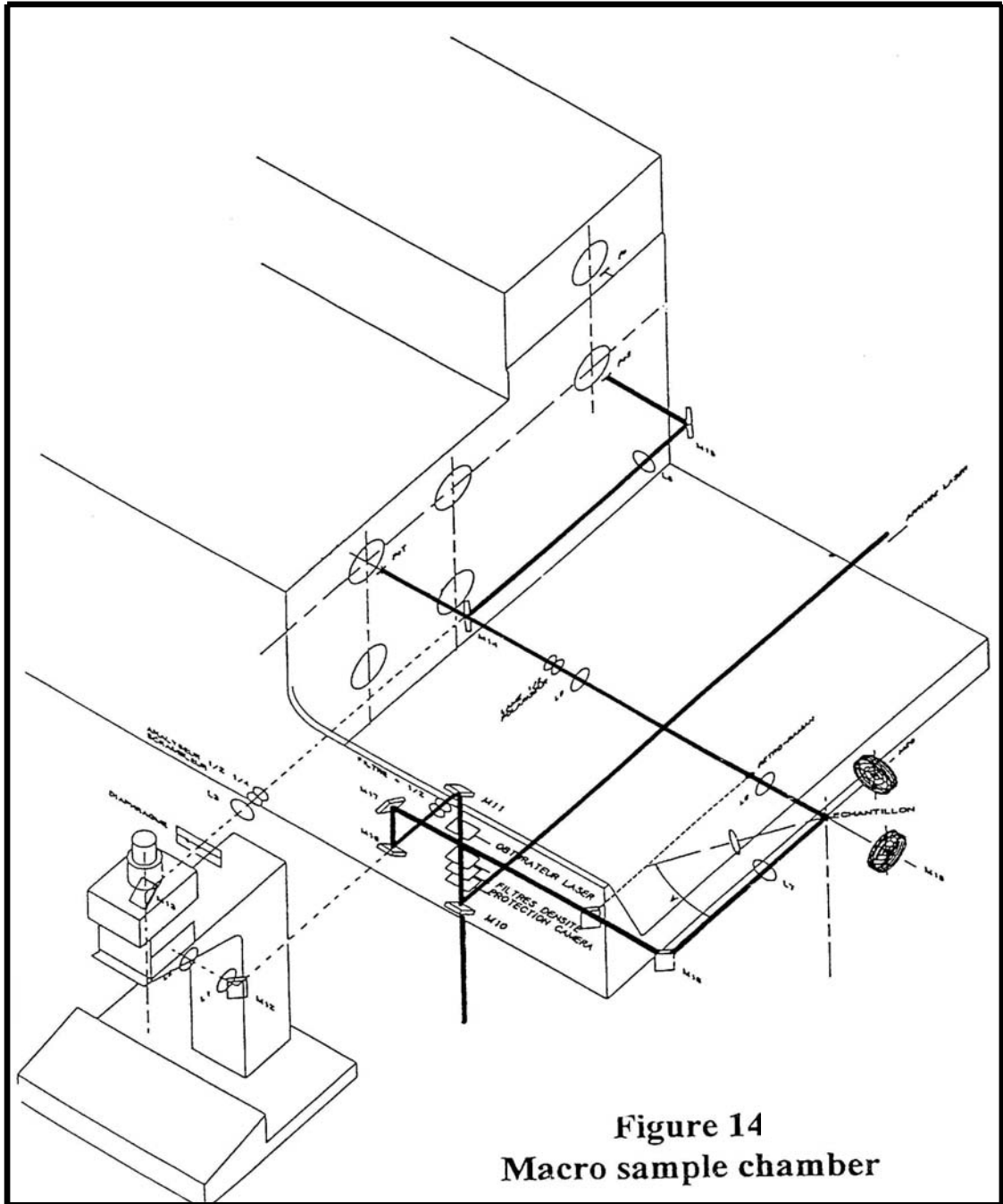
B. The final adjustments must be performed with all the focusing and collecting optics.

B.1. Right angle scattering

1. Remove the mirror (or the reflecting sample) on the sample location.
2. Install the laser focusing lens and verify that the beam is centered on the centering piece on the M<sub>20</sub> mirror.
3. Place back the mirror on the sample location. Readjust to center the beam on the collimator through the entrance slit.
4. Move vertically and horizontally the L<sub>8</sub> lens to enhance the beam centering
5. Move the L<sub>8</sub> lens to focus the beam on the entrance slit and to have the brightest image on the collimator.

B.2. Backscattering

1. Move the M<sub>18</sub> mirror to the left end side of the sliding rail. Set the backscattering mirror on. The beam must be reflected on the centre of the mirror back to the sample.
2. Move the L<sub>8</sub> lens to improve the adjustment.



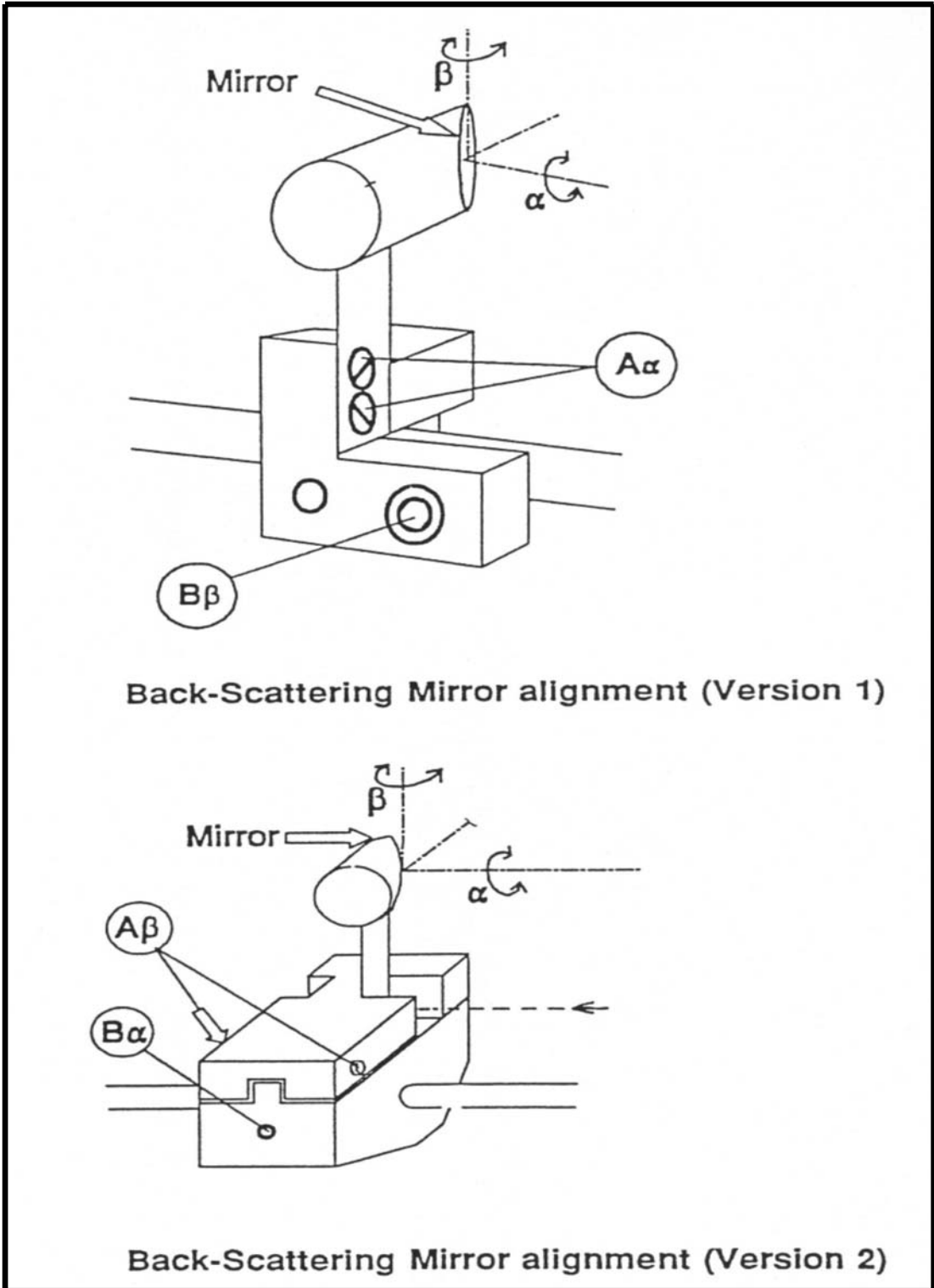


Figure 2

**Annex 3**

## **Procedure for the readjustment of the CCD detector**

The readjustment consists in three operations:

1. focusing the CCD, i.e. placing the CCD chip perfectly in the plane of the exit image.
2. centering the image of the spectrum on the chip.
3. orientating the chip parallel to the image of the spectrum.

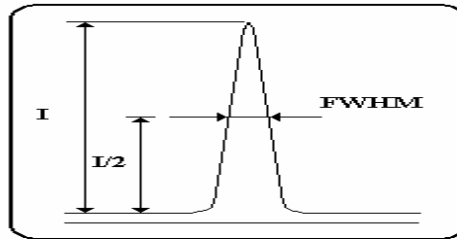
### **Operating modes**

1. If the instrument is equipped with the single spectrograph option, select this configuration for the adjustment.
  - . Move the spectrograph to the position 546.07nm.
  - . Illuminate the axial entrance slit of the spectrograph with a low pressure Hg lamp.
  - . Open the entrance slitwidth  $S_5$  to 10 $\mu$ m or 15 $\mu$ m and the slit height at minimum
2. If the instrument is not equipped with the single spectrograph option, select the triple subtractive configuration for the adjustment.
  - . Move the monochromators to the position 546.07nm.
  - . Illuminate the axial entrance slit of the double foremonochromator with a low pressure Hg lamp.
  - . Open the entrance slitwidth of the foremonochromator  $S_1$  and the intermediate slitwidth between the 2<sup>nd</sup> and 3<sup>rd</sup> stages  $S_{i2/3}$  to 10 $\mu$ m or 15 $\mu$ m and their slit height at minimum.
  - . The intermediate slitwidth between the 1<sup>st</sup> and 2<sup>nd</sup> stages is wide opened for the full coverage on the CCD.

#### **1. Adjust the focus**

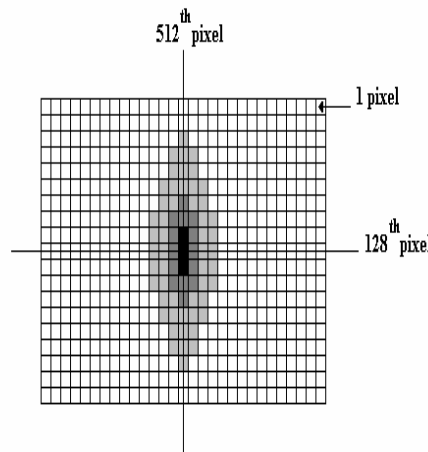
In the RTD ( real time display ) program, acquire continually spectra. Select an integration time of 0.1s. In the case of saturation of the CCD, use a neutral density filter to attenuate the Hg line intensity. Select in the RTD setup the X-axis display in pixels. Expand the spectrum around the 546.07nm spectral line. Turn the black ring (A in Figure 1) of the mechanical adaptation between the spectrograph and the CCD in order to increase the intensity of the line and to obtain it sharper and sharper.

As a final result, the FWHM (Full Width at Half Maximum) of the Hg line must be less than 2 pixels.



**2. Adjust the centre**

In the RTD program, change from Acquisition of spectra to Acquisition of CCD image. Select a longer integration time, between 0.5s to 1s. Acquire a CCD image. Suppose the size of the CCD chip is 1024x256 pixels, the image of the Hg line must be centered around the 512<sup>th</sup> pixel on the width and around the 128<sup>th</sup> pixel on the height.



If the image of the 546.07nm line is not centered on the CCD matrix:

**2.1. Adjust along the height**

To center the image on the 128<sup>th</sup> pixel, turn the screw (B in Figure 1) located on the rear side of the T64000, and verify every time the centre with the help of the CCD image.

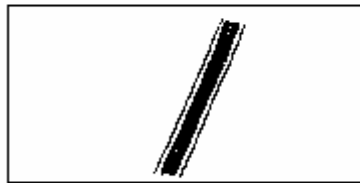
**2.2. Adjust along the width**

Return to the acquisition of spectra. Acquire continually spectra, and do expansion around the 546.07nm line. If the line is not centered on the 512<sup>th</sup> pixel ( $\pm 1$  pixel), unscrew the two screws (C in Figure 1), and turn the whole mirror holder (D in Figure 1) in

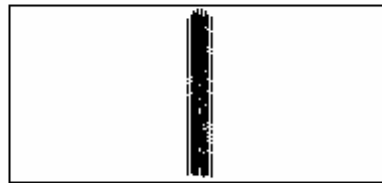
order to center the Hg line on the 512<sup>th</sup> pixel ( $\pm 1$  pixel).

### **3. Adjust the orientation**

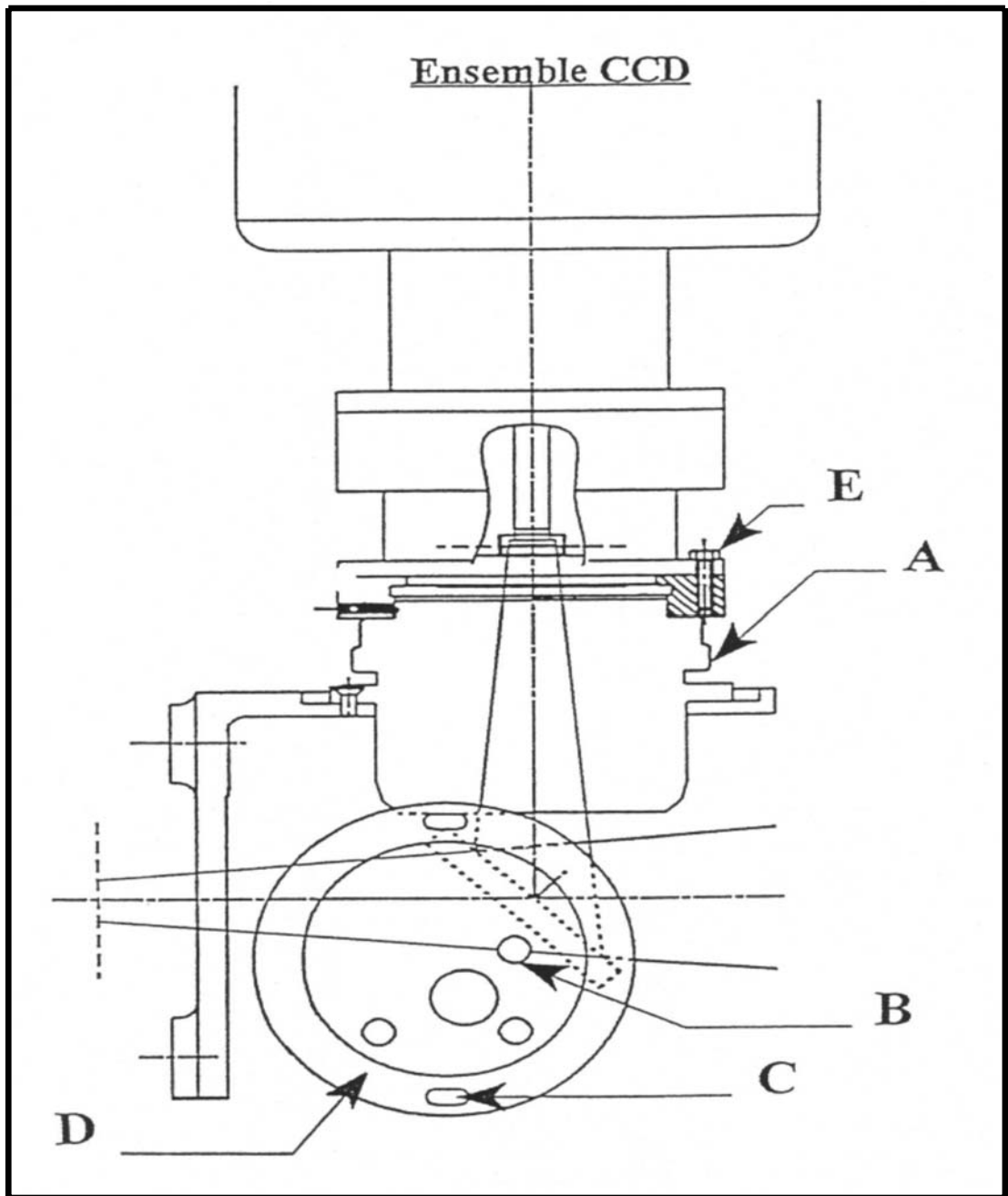
Open the slit S<sub>5</sub> or the slits S<sub>1</sub> and S<sub>2/3</sub> to 50 $\mu$ m and all the slit heights at maximum. Acquire a CCD image and verify that the image of the Hg line is perfectly parallel to the vertical axis of the CCD matrix. If it is not, unscrew the three screws (E in Figure 1), and turn very slightly the CCD detector and verify with the help of the CCD image that the image of the Hg line is perfectly parallel to the vertical axis of the CCD.



**Bad orientation**



**Good orientation**



**Figure 1**