

Report

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# **Appendix A:**

## Manual instruction for TriVista 557 Raman system







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## A. MANUAL INSTRUCTION FOR TriVista 577 RAMAN SYSTEM

## **Software Settings**

#### **1** Introduction

The S&I software was written in order to obtain an optimised access to all three stages of the TriVista. It is programmed in "Visual Basic" and runs in co-operation with Princeton Instruments' WinSpec software package, which is designed to operate a multitude of CCD, ICCD and InGaAs detectors and allows access to exclusive detector functions. S&I software controls spectrometer functions while WinSpec is used as a DLL and provides data acquisition and setup functions for multi-channel detectors. Since these features are accessed by S&I, WinSpec must always be active when the S&I software is running. WinSpec is not required when data is acquired by a singlechannel detector such as a PMT, but the data will be stored in the WinSpec "SPE" format.

Because WinSpec is used as a DLL, only some descriptions of the main functions of WinSpec are included in this manual. For more information, review the WinSpec manual.

Apart from these two software packages there exist three more:

- QED software for running and control of the optical camera 0
- Stage Control software for the control of XYZ motorized Stage, 0
- o Linkam software for the control of heating and cooling in Linkam microchamber.

#### 2. General Hardware Settings

The first screen you will see after starting the S&I-software is the Measuring window. Because you will be entering, changing, or verifying the TriVista hardware settings, you must click on the Hardware button at the lower left of the window (see Figure 1).

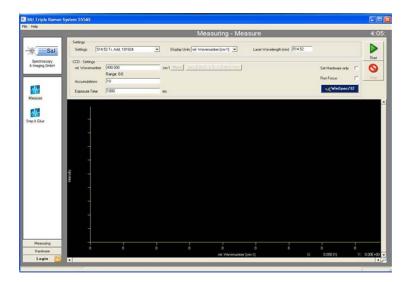


Figure 1. Window Displayed at Startup of the S&I Software



The Hardware window contains the System Settings table (automatically accessed by the program), displays descriptions of the currently selected measuring mode, and shows the current hardware configurations of the three stages (Fig.2).

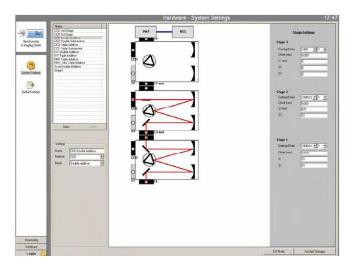


Figure 2. System settings within the Hardware window

Any of the currently defined modes can be selected and used to acquire data, but changes to hardware settings are password-protected. For details you can see original TriVista System Manuel.

## 3. Generating Different Configurations

Use the "System Settings" button (lower left) to switch to the main Hardware window. As shown in Figure 3, you will find a window that is divided into three panels:

- Main setup properties on the left side, •
- Illustration of the optical path in the center, and
- Individual stage settings on the right side •

He.	Hardware - System Settings	1
Konserversen Sectores Sectores System Setting System Setting		Steen Softwar Step 3 Galego Steel 100 20 20 20 Different 100 100 20 20 Different 100 20 Different 10
Glad College	55 S mot	Stage 2     300 mm       Other into     50.00       State     10       State     10
- Selings Naise (200 Brid Hage Method, (201 Brid A/T) Mode: (201 Brid A/T)		34442 1 Guang Data 100 ≥ 2 Officienty 5100 51 2 52 111
Neuron		
Kadware		Tet Moder Annual Charles

Figure 3. System Settings window





Every configured setup and every change within a setup has to be stored by using the "Accept Changes" button (right, bottom). To activate the stored settings, click on "Set Mode" or start data acquisition in the "Measuring" window.

## 3.1 TriVista Configurations

Each configuration of the system that you have stored can be recalled later by clicking on its name. Figure 4 shows of a list of such configurations. The setups differ from each other in the following ways:

- Mode (Figure 5), 0
- Method for detection (CCD, PMT), and 0
- Choice of gratings. 0

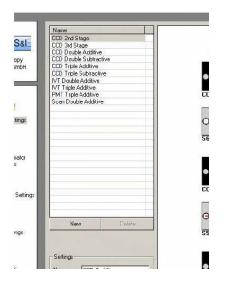


Figure 4. List of TriVista Configurations

– Settings –	
Name	CCD Double Additive
Method	CCD
Mode	Double additive
	Triple additive Triple subtractive
	Double additive Double subtractive
	Spectrograph only Stage 2 only Stage 1 only

Figure 5. Mode Selection (depending on 7 different ways of use of TriVista spectrometer)

## 3.2 Setting Main Properties

The first step to generate a new device configuration is to set the main properties. Therefore, you click the "New" button and:

- Enter a configuration name,
- o Select a detection method, and
- Select a mode for the spectrometer system. 0

To correctly configure the chosen "Mode", refer to the illustration of the optical path in the middle panel of the "System Settings" window.

For details related to the settings of main properties you can see Paragraph 5.4.2. in original TriVista System Manuel.



## 3.3 Stage Settings

"Stage Settings" is the last part of a system setup. The Stage Settings panel is at the right side of the "System Settings" window, as shown in Figure 54. For each stage it is possible to choose:

- Which grating in which order shall be used, 0
- The width of each slit, and 0
- The offset of the stage (see below). 0

## Gratings and Order

By selecting a grating you also define the dispersion. The higher the groove density of the grating, the higher the dispersion and the lower the CCD spectral coverage. Probable values for groove density of the gratings depend on the settings for the gratings that have been installed and set up in the "Monochromator Settings" window. It is also possible to define the order to be used for each grating.

2	Stage-Settings
-Stage 3	
Grating/Orde	1800 • 1 •
Offset (nm)	0.000
S7 mot	200
SB	9
SB	12
Stage 2	
Grating/Orde	
Offset (nm)	0.000
S3 mot	11000
S5	D
-Stage 1	
Grating/Orde	900 • 1 •
-	, _, _, _,
Offset (nm)	0.000
S1	10
\$2	10
Set Mode	Accept Changes

Figure 6. Stage Settings

#### Tips:

1. Subtractive mode is only possible if the groove density of the first two stages is the same. If it differs, the dispersion can not be inverted correctly.

2. To illuminate the whole **CCD** chip, the groove density of the last stage should be twice of the groove density in the first two stages in mode "Triple Subtractive".

#### 3.4 Slits

Slit names and status (manual, motorized, or disabled) are determined by the entries made in the "Monochromator Settings" window. The default names of the slits are S 1 - 2 in Stage 1, S3 - 6 in Stage 2 and S7 -9 in Stage 3. The active slits are represented in the "System Settings" window by the "Slit" fields (below the "Offsets" field in each stage). The function of the field depends on



whether the slit is manual (for example, an entrance slit) or motorized (for example, an intermediate slit):

- If the slit is manual, the field allows you to enter the current slit-width for a manual slit as a • comment or reminder of the slit-width it has been set to.
- If the slit is motorized, entering a slit width sends a command to actually change the slit-• width. The range of width values for a motorized slit is 10 - 12000 pm.

<u>Sta</u>	ge-Settings
-Stage 3	
Grating/Order	1800 🔽 1 💌
Offset (nm)	0.000
S7 mot.	200
S6	9
S8	12

Figure 7. Slit Settings in the 3<sup>rd</sup> Stage

TIPS:		
· · · ·	t at a low level, all slits that are not used when only one stage is used) should be	- · · ·
5	depends on the slit-width of the entrance diate slit works as an entrance slit.	ce slit. In "Triple Subtractive" mode,
• When using a PM' opened up to 150 p	$\Gamma$ , the intermediate slits should be narro om.	w for stray light rejection but can be
For standard applie	cations with a CCD camera we recomm	end to use the following slit-widths:
•		
Mode	Intermediate Slit 1 [µm]	Intermediate Slit 1 [µm]
Double Additive	12000	0
Double Subtractive	12000	0
Triple Additive	6000	12000
Triple Subtractive	12000	As Entrance
	Table 1. Mode vs. Intermediate Sli	t Width

## 3.5 Offsets

"Offset (nm)" fields in "Stage-Settings" are used for setting of offset values for the optimisation of accordance between stages and given application request.

<u>Sta</u>	ge-Settings
Stage 3	
Grating/Order	1800 🔽 1 💌
Offset (nm)	0.000
S7 mot.	200
S6	9
S8	12

Figure 8. Offset (nm) Setings



**CAUTION** 

The adjusted offset-position is only valid for the current grating and current wavelength! Note that the values in the offset-table in the "Monochromator Settings" window are set for the whole spectral range of the grating and not for a specific wavelength. Matching stages is explained in "5.6. Matching the stages".

Additional settings that are shared by all generated configurations are defined in the "Global Settings'' window and described in the Paragraph 5.4.6. in original TriVista System Manual.

## **B. INSTRUCTIONS FOR MEASUREMENTS**

## **1. Beginning with measurements**

When all the settings are performed and there is nothing left to be changed one can begin with measurements. After running S&I software from Measuring Window on the right screen, run programme WinSpec. WinSpec window will appear on the left screen (see Figure 8).

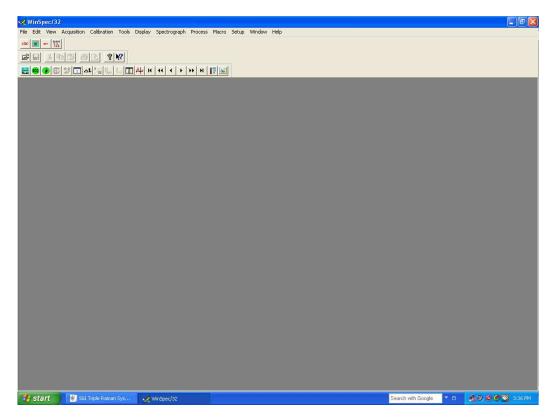


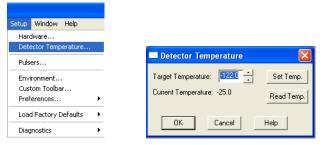
Figure 9. WinSpec window

## **1.1.** Cooling of the detector

Prior to measurements check the temperature of CCD detector cooled with liquid nitrogen whose working temperature is -122 °C. Open the falling menu Setup In the WinSpec window and click on Detector Temperature (Figure 9, left). A new window will appear (Figure 9, right) where the



present temperature of the CCD detector can be read. If the read temperature is higher than the working temperature (122°C) liquid nitrogen can be poured into the detector reservoir.



#### Figure 10. Setup menu (left) and a window with detector parameters (right)

Nitrogen is poured from the liquid nitrogen reservoir (Figure 10, left) using yellow tap on the left. The use of protection gloves is necessary. Nitrogen is first poured to the small metal reservoir and then from this reservoir to the detector with the use of filler cap.



Figure 11. Large reservoir for liquid nitrogen (left) and protection gloves (right).

#### 1.2. Turning the laser on

If the detector is cooled down to its working temperature the laser can be turned on. A combined Argon-Krypton  $(Ar^+/Kr^+)$  laser from the next-door Micro Raman laboratory (along with Jobin Yvon T64000) is used. Laser beam is directed by beam splitter (transmitting 50% of the output laser power in both directions) through the hole in the wall to the laboratory with TriVista system, and then is led with the series of mirrors to the TriVista microchamber entrance.

#### 1.2.1. Turning the laser cooling on

**Tips:** LASER PURE 20 cooling system is used for cooling of the magnet in the laboratory with TriVista spectrometer, which is why special attention should be paid to prevent shut down of the cooling system which could cause the damage to the magnet.

- 1. Turn on the tap on the water pipe if it is not already opened;
- 2. Turn on **POWER** button on **cooling system box** (LASER PURE 20) if it is NOT turned on;

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- 3. Turn on the taps that two black pipes are connected to (i.e turn them to horizontal positions);
- **4.** During an experiment the pressure and temperature of the distilled water in the closed system should be controlled at the appropriate meter Pressure on this meter MUST NOT get higher than 2.6 bars because it can damage pipes in the mere laser. Pressure on this meter MUST NOT get beneath 2 bars because the laser overheats in that case and shuts down automatically.



Temperature on the meter is usually around 22 °C.

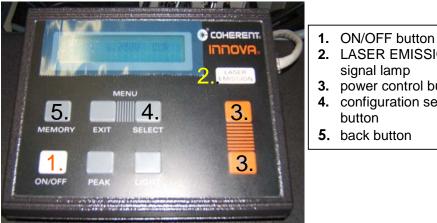
## 1.2.2. Turning on the laser feeding

- 1. Turn the transformer on (Figure 12, left) by turning it to the position 1;
- 2. Turn on the laser feeding box (Figure 12, right) (which lies under the table where the T64000 Raman system is placed) turning the key on the box to ON position after which two lamps should turn on (green light).



Figure 13. Transformer (left) and *laser feeding box* (right).

1.2.3. Turning the laser beam on



2. LASER EMISSION signal lamp 3. power control buttons

4. configuration settings

5. back button

Figure 14. Laser control unit.

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When the laser feeding on the control box screen appears the following text: LASER OFF **CUR 0.0** 

- 1. Press the ON/OFF button (1.) on the control box;
- 2. Laser needs 60-70 seconds to start lasing, if the emission doesn't start after this period nor after possible announced delay (~20s), TURN THE LASER OFF BY PRESSING THE ON/OFF BUTTON AND TURNING BACK THE KEY ON THE FEEDING **BOX, THEN ASK FOR HELP !**
- 3. When the laser starts lasing the signal lamp LASER EMISSION (2.) turns on and the following line appears:

10.0 A 0.000 W CUR;

4. Using the upper and the lower power control button (3.) laser output power can be changed.

#### 1.2.4. The choice of laser line wavelength

- 1. Changing of the laser line wavelength can be performed by rotation of two screws at the back of the laser box. These screws should be rotated until the maxima laser power is acquired (this can be read on the laser control box display) for the chosen laser line. The upper screw serves for approximate adjustment and the lower one for fine adjustment.
- 2. In order to set the wavelength value of the chosen laser line press SELECT (4) button, find the option



Figure 15. Laser backside

LASER LINE by pressing button (3), then confirm your choice with button (4). Pressing the button (3) find the appropriate wavelength value, confirm it with button 4. Use button (5) to return to the menu.

λ [nm]	p [W]
457,9	0,15
465,8	0,04
472,7	0,05
476,5	0,30
488,0	0,70
514,5	0,80
530,9	0,20
568,2	0,5
647,1	0,35

Table 2.  $Ar^+/Kr^+$  laser line wavelengths and appropriate maximum output laser powers for the corresponding wavelengths

WARNING!! IONIC LASER MIGHT BURN THE CLOTHES, FINGERS AND <u>CAUSE</u> <u>PERMANENT EYE DAMAGE</u>. DO NOT PUT SHINY OBJECTS IN THE LASER BEAM PATH. BEWARE WITH MIRRORS ADJUSTMENT. PERFORM BEAM FOCUSING AT SMALL LASER POWES LASERA. **BE CAUTIOUSI!** 

## 1.2.5 Turning the laser off

Laser can be turned off by performing series of operation in the inversed order from the order they were performed in when laser was turned on:

- 1. Decrease laser power to minimum.
- 2. Press ON/OFF button.
- 3. WAIT FOR TEN MINUTES FOR LASER TO COOL DOWN.
- 4. Turn the feeding box off.
- 5. Turn the transformer off.
- 6. Close the taps connected to black pipes from laser cooling system.
- 7. DON'T TURN OFF THE COOLING SYSTEM IF IT IS USED FOR COOLING OF THE MAGNET.

#### **1.3.** Adjusting the sample position

#### 1.3.1. Microscope

Olympus BX 51 (Fig.16) microscope is a part of micro-Raman spectrometer. This microscope operates using the confocal microscopy principle. Laser beam is led by the set of mirrors to the micro-chamber entrance (Fig.17). In the microchamber the beam is led to the semi-transmitting mirror where the part of the beam is reflected and led to the objective which focuses it on the sample. The sample is placed on the horizontal surface beneath the objective (Fig. 16). Back-scattered radiation is focused by objective on its way back to the semi-transmitting mirror where it is transmitted and led by a set of mirrors to the entrance of the TriVista first Stage.

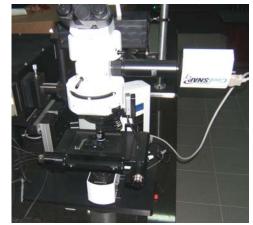


Figure 16. Microscope Olympus BX 51

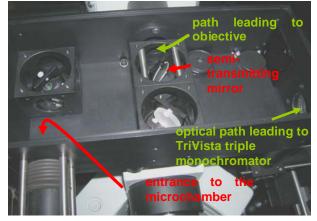


Figure 17. Microchamber and its elements

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Sl. 18 Linkam heating/cooling stage

Linkam heating/cooling stage is used for Raman measurements at the temperatures below or above room temperatures (Fig.18). The sample is placed in the stage chamber, beneath the optical window. Objectives with longer focal length are used in such a case.

## **1.3.2. XYZ Microscope Stage**

A sample may be moved in x, y or z directions either manually or using XYZ motorized Stage. Sample is moved in x and y direction with the help of metal screws on the sample holder edges. One big and one small black screw on the right side of the microscope are used for moving the sample along with sample holder in z direction (Fig.19). The big screw serves for movements with a large step and the small one is used for finer position adjustments.

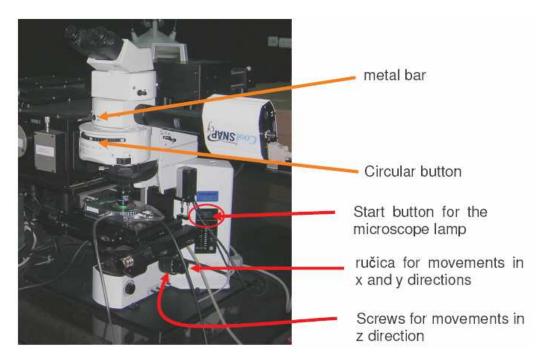


Figure 19. Microscope elements

Sample positioning in xy plane is performed with motorised XYZ Stage, either by joystick or by setting of the appropriate values in the programme Stage Control. Pressing the button on the top of the joystick enables quicker moving of the Stage. The finest z position adjustments are performed with a small black screw on the right side of the joystick holder (Fig. 20).

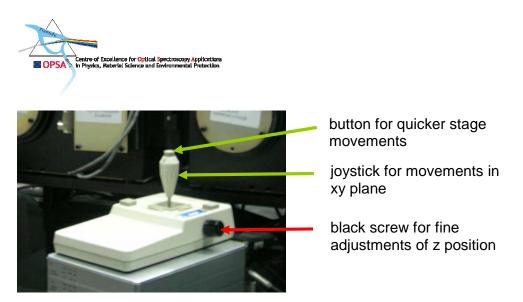


Figure 20. Joystick of the motorised XYZ Stage

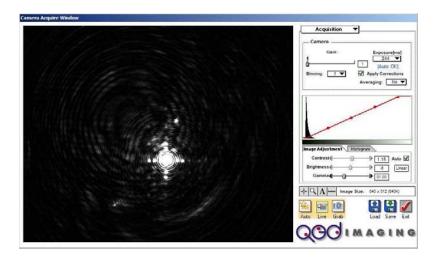
Motorised XYZ Stage can be also used for scanning of the sample along x, y and z axes. Raman spectra can be taken from different spots, with a predetermined spatial step using the programme Stage Control. Details of the procedure are described in Stage Control Manual.

Programme Stage Control has an Autofocus option, which enables the system to find the focus position itself (that is the position characterised by highest Raman signal intensity) in the predetermined z-axis range. Starting positions and used step are set in the programme Stage Control. Details of the procedure are described in Stage Control Manual.

#### 1.4. Focusing of laser beam on a sample

After a sample is placed on the sample holder microscope lamp (Fig. 19) is turned on. Circular button with positions numbered from 1 to 6 (Fig. 19) should be turned to position 1 (only in this position light of the lamp reaches the sample). Metal bar above the wheel is pushed in (activation of the camera) (Fig. 19).

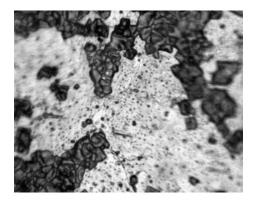
Programme QED Capture is run by clicking on its icon on the right screen and its active window appears on the left screen. In the lower right corner of the screen there is a LIVE button, clicking it shows camera's picture of the laser spot on the sample (Fig. 21a).



1.1. Figure 21a. Laser spot on the sample recorded by camera



A sample is positioned below the objective in the abovementioned way. One of two objectives with magnifications 20x or 100x is chosen. Moving the sample holder in z direction the focus position is reached which results in the appearance of the picture of the sample surface on the screen (Fig. 21b) if the laser power is decreased enough.



1.2. Figure 21b. Sample surface recorded by camera connected to the microscope

TIPS: Due to high intensity of laser radiation compared to the intensity of microscope lamp laser spot on the sample is shown on the screen when the camera is on. It is therefore necessary to decrease laser power, put some obstacle on the laser beam path, in order to see the picture of the sample surface with the camera.

After focusing of the beam is performed circular button is turned to position 6, the metal bar is pulled out (Fig.19) and one can begin with collection of the Raman spectra.

## **2 Data Acquisition**

To perform an acquisition, click on the "Measuring" button to open the Measuring window (Figure 22).

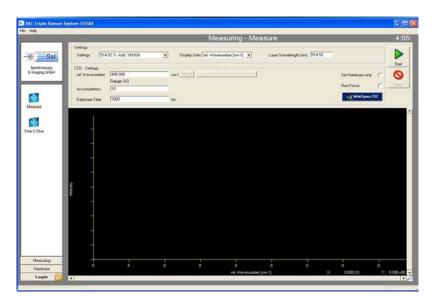


Figure 22. Measuring window

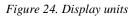


## 2.1 Global Settings and Commands

At first you need to choose one of the configurations that have been generated in "System Settings". The list of the configurations will appear by clicking the arrow at the first combo box in the "Settings" region.

Settings	CCD Triple Additive	<ul> <li>Dis</li> </ul>		
	CCD Triple Additive			
CD - Settings	CCD Triple Subtractive IVT Double Additive		_	
Wavelength	IVT Triple Additive PMT Triple Additive	nm M	Display Units	Wavelength [nm]
	PMT_Test Triple Additive			Wavelength [nm]
Accumulation	Scan Double Additive	-		abs. Wavenumber [cm-1]
	[Stage1			rel. Wavenumber [cm-1] Energy (eV)

Figure 23. Selection of a Setup



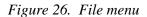
Independent from the chosen configuration it is possible to define the way the units of the spectra are displayed at the bottom of the measuring window. As Figure 60 shows it is possible to choose between:

- Wavelength [nm],
- Absolute wavenumber  $[cm^{-1}]$ ,
- Relative wavenumber [cm<sup>-1</sup>] and
- Energy [eV] ٠

If the selected units are either "Relative Wavenumbers" or "Energy", you also have to enter the used laser wavelength at the third field within "Settings" so the software can perform a correct calculation of the dispersion with reference to the stimulation energy or wavelength.



Figure 25. Start and Stop buttons



To start a measurement, click on the "Start" button. While the measurement is in progress, you can interrupt the measurement by clicking on the "Stop" button. The system will finish the actual data point of the measurement and stop. When you click the "Stop Button", the software will ask you if you really want to Stop or to Continue. If you stop a measurement, you will not lose the data that you have already taken.



To **save** an acquired spectrum, click "File" and "Save File" as shown in Figure 62. Measurements with a CCD camera can also be stored through "WinSpec". To **open** a stored spectrum, use "Get File".

**Caution:** For spectra that are measured with a CCD camera "Get File" is only able to transfer measuring data from WinSpec to the S&I software. Such spectra have to be opened by WinSpec first.

The File menu also allows you to store the measuring settings for each specific setup in "System Settings". To save the settings for a setup, click "Save Mode". "Clear Mode" stores the settings of the last measurement that is done in the current mode.

## 2.2 Data Format and illustration

A spectrum is always stored as an "SPE" file. This type of file contains a header (with information about the measurement properties) and the data. To view the properties, switch to WinSpec, select the "File" menu, and then select "File Information" as shown in Figure 27.

File Edit View Acquisition	Calibration	File Information:C:\Program Files\Roper Scientific\WinSpec32\DATA\3rd Stage 🗙
Open Close	Ctrl+0	General Hardware Experiment ROIs Calibration Processes Spectrograph
Save Save As Save All File Information	Ctrl+S	File Created By: WinSpec/32 Software Version: 2.5.19 Mar2005 File Header Version: 2.2
Print Print Preview Print Setup	Ctrl+P	Comments (5 Lines): [Edit Comments]
1 600_313nm 3rd order.SPE 2 hg_Oct19_04_48.SPE 3 hg_Oct19_04_48.SPE 4 spec_add18all.SPE 5 spec_add6612all.SPE 6 spec_add6618all.SPE		Send Data To Print Window
Exit		Close Send All Data To Print Window Help

Figure 27. Item "File Information" in WinSpec

Figure 28. File Information dialog (in WinSpec)

Then a popup dialog box will appear as Figure 28. Details related to this dialog box can be found in the paragraph 5.5.2 in the original TriVista System Manual.

An acquired spectrum can be zoomed by using the magnifying glass (Figure 29.) at the lower right corner of the Measuring window. Changes of the zoom-factor never cause loss of data.

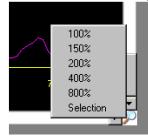


Figure 29. Zooming



## 2.3 CCD Detector and WinSpec

If "CCD" is chosen as measuring method in the setup, "CCD-Settings" will appear at the top of the panel.

CCD - Settings Wavelength	546.000	nm Move <<< < > >> >>>	Set Hardware only
Accumulations	Range: 545.891 - 546.109		Run Focus 🗖
Exposure-Time	50	ms	WinSpec/32

Figure 30. Settings for CCD Measurements

The name of the top field changes depending on the selected "Display Units" (chosen in the "Settings" panel). This is the field where you enter the center wavelength for your measurement. Below this field, you will see the calculated spectral range that will illuminate the CCD (this dispersion is calculated by the software from the focal length, inclusion angle, and detector pixel width entered in the "Monochromator Settings window). When you start the experiment, you will get a spectrum on the CCD whose resolution depends on the properties of your setup in the "System Settings" window. In the "Accumulations" field, you enter the number of exposures for the measurement of one spectrum. In the "Exposure Time" field you enter the illumination period for each exposure. These two values can also be defined through Winspec.

To move the system to a new center wavelength only, without taking a spectrum with the CCD, activate the "Set Hardware Only" checkbox. If you activate the "Run Focus" checkbox, clicking on the "Start" button will cause the system to take spectra in "Loop Mode" until you click on the "Stop" button. The "Run Focus" function should be used for alignment of the system or the sample. If "Run Focus" is not activate, the system takes only one spectrum with the given exposure time and the number of accumulations. During the acquisition, the spectra are also displayed in the WinSpec software. To monitor the acquisition, click on the "WinSpec/3ZM button and you will be in WinSpec.

TIPS: If WinSpec isn't running click on "WinSpec/32" icon before pressing "Start" button. Click on "Start" to begin with data acquisition.

Data acquisition with CCD or Array detectors is controlled and done trough WinSpec. For that reason, the general properties of the detector and the acquisition mode have to be defined there. For detailed procedure of detector properties definition you can refer to the Paragraph 5.5.4 in the original TriVista System Manuel.

## 2.4. Measurement Parameters in WinSpec

Nearly all parameters for the data acquisition with multi-channel detectors can be set in menu "Experiment Setup" dialog box (menu item "Acquisition", then "Experiment Setup"). Only "Exposure Time" and "Accumulations" are controlled through the S&I software (beginning of this chapter).

Centre of E OPSA to Physics, f	collence fo <mark>r Opt</mark> laterial Science a	ical <mark>Spec</mark> tre Ind Environr	scopy <mark>A</mark> pplicat nental Protecti
xperiment Setup		×	
ADC Timing Proce Main Data File R01 Set Exposure Time			
CCD Readout	<u> </u>		
Use Full Chip C Use     Readout Dimensions: >>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>	1997	et	
Accumulations			
	Cancel	Help	

Figure 31. Experiment setup in WinSpec

To understand the use of the "ADC", "Timing", "Process", "Save&Load" and "Data Corrections" tab pages, refer to the WinSpec manual. All parameters in WinSpec need to be set according to your experimental requirements. To take a picture with the detector, choose "Use Full Chip" at "CCD Readout". When measuring spectra, select "Use Region of Interest".

	es Save/Load
ain Data File ROI Setup	Data Corrections
Data File Name	
Name:	
Auto Increment File Name	
Enable Conent.Volue:	
and an and a second second	r
Overwrite/Append	Data Type
Overwrite  Existing Files	Contraction of the second
	FLOAT *
Confirm before overwriting	IFLOAT •
Auto-save and prompts:	
Automatically save file after ea	sch run 💌
Use a new window for each	hiun

The "Data File" (Figure 32.) parameters are also described in the WinSpec manual. For data acquisition through the S&I software "Data Type" must be set to "FLOAT"! All other data types are not supported.

TIPS: To save time during measurements, you may want to deactivate the checkbox "Confirm before overwriting" (in the "Overwrite and Append" section).

Figure 32. Format of Measurement Data

Setting up an "ROI" (Region Of Interest - Figure 33) can be used to optimize the spectral data that you will acquire.

- For maximum intensity the full height of the CCD may be "binned" (gathered) into one super pixel.
- To minimize the effect of dark charge and keep the background signal as small as possible, you can specify that only the illuminated area of the CCD be read out. CCD systems with less efficient cooling may have a relatively large dark charge, especially with increasing exposure time.
- To read out the whole chip, click on the "Full" button.

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kperin	nent Setup
ADC Main	
	ew Pattern: 1 🔄 Number Stored: 0 Imaging Mode 📀 Spectroscopy Mode
	'avelength Slit Start 1 ∓ Start 1 ∓
	End 384 - Height 576 -
Мо	ise Full Clear Clear All STORE
	Cancel Changes 🛛 🗂 Use Software Binning
Acquir	e Focus OK Cancel Help

Figure 33. Region of Interest for CCD chip

Never forget to use button "OK" after setting the Chip parameters.

## 2.5 Conversion of the collected spectra to ASCII format

Collected spectra can be converted to a text file, which is convenient for use in other programmes for results analysis (i.e. Microcal Origin).

The conversion of the files containing spectra is done in WinSpec programme. Open Tools menu and choose Convert To ASCII option (Figure 34a).



Figure 34.(a) Convert to ASCII option

A window will be opened (Fig. 34(b)) with help of which a spectrum maybe converted to the required format.

Fig. 34(b) shows a choice of parameters used to convert a spectrum to two columns, divided by Space, first contains values of Raman shift in rel. cm<sup>-1</sup> (Pixel), and the second one corresponding signal intensities (Intensity).

**Tip**: More spectra may be converted in the same time.

.SPE To ASCII Conv	ersion						
Choose Files	Choose	Output Directory	Done				
Get Active Window	C:\Doc	uments and Settin	Help				
3 files in list							
File Name		2.111 B	17. 0. 11				
C:\Documents and Se C:\Documents and Se C:\Documents and Se	ettings\Tri	Vista\My Document:	s\Testiranje\				
<			>				
Frame No.: 1 to File Extension : txt Delimiter C Tab C Semice © Space C Comma		Convert To ASCI Output Order Pixel , Intensity C Intensity , Pixel					
X-Axis Unit rel. cm-1	·	<ul> <li>New Line Charac</li> <li>Carriage Retuine</li> <li>Line Feed</li> </ul>					
Output File Options One File Per Fram One File For All Fr	-		· ·				
Pixel Format C Preserve CCD X/ C Convert CCD X/Y							

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## 2.6 Collecting Raman spectra in wider range – Step & Glue mode

If there is a need for Raman spectra to be collected in wider range from the one determined by chosen combination of gratings in three Stages of TriVista system, Step&Glue mode entailed in S&I programme should be used.

While running S&I software, press "Step & Glue" icon at the left side of Measuring window (Figure 22). Measuring – Step & Glue window (shown on the right side of the Figure 34) will appear on the right screen. On the left screen WinSpec/32 window should remain active.

Input of the following parameters of the measurement should be made into the fields on the right screen:

- Units (rel.  $cm^{-1}$ , nm, ...),
- Range (From, To) in the set units, 0
- Overlap between the subsequent regions spectra are taken in expressed in percents, 0
- Accumulations, 0
- Exposure time. Ο

When the mentioned parameters are chosen, one can start the measurements by pressing Start button in the upper right corner of the right screen. On the left screen, in WinSpec window, parts of the collected spectrum that is being taken at the given moment will be shown.

After the performed measurement spectrum should be saved in regular way.

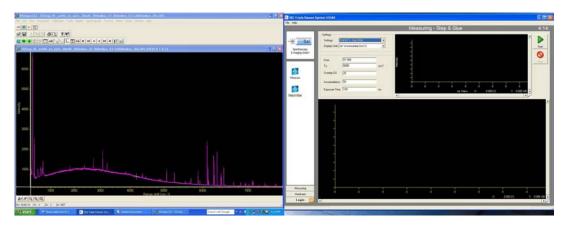


Figure 34. Windows shown during measurements in "Step&Glue" mode

## **3** Matching the Stages

Within the S&I software, there are two different locations that you need to access when matching the different stages: the general "Offset Table" in "Monochromator Settings" and the "Stage Settings" for a specific measurement mode. The matches are stored as "Offsets", which means an additional movement of the turret to the correct position.

The Offset Table in the "Monochromator Settings" Window is described in detail in Paragraph 5.6.1 of the original TriVista System Manuel.



## 3.1 Stage Settings within the "System Settings" window

"Stage Settings" in "System Settings" window are accessible by every user. The offsets in "Stage Settings" are specific values for only one specific configuration and only a specific wavelength range. If you change a grating, the position of one of the diverter mirrors, or change the wavelength, the values are no longer appropriate and you have to enter the new values for the new setup.

## 3.2 Matching - through "Advanced Slit Commands"

To execute the matching, it is necessary to start a measurement in "Run Focus" mode. This operation is only possible while a running measurement. The locating of the offsets occurs through "Advanced Slit Commands" on the "File" menu (Figure 34).

File	Help
G	et File
S	ave File
S	ave Mode
С	lear Mode
Ρ	references
A	dvanced Slit-Commands
D	ebug-Mode
В	eenden

Figure 34. Item "Advanced Slit Commands"

	(Move)	<<<	<<	<	>	>>	$\rightarrow$
	Move	<<<	<<	<	>	>>	>>>
			Accept Change				
nm	Move	<<<	<<	<	>	>>	>>>
nm	Move	<<<	<<	<	>	>>	>>>
nm	Move	<<<	<<	<	>	>>	>>>
	nm	nm Move nm Move	nm Move <<< nm Move <<<	Move         <<< <<           nm         Move         <<< <<	Move         <<< << <<           Acc           nm         Move           Move         <<< <<	Move         <<<         <         >           Accept (	Move         <<<         <         >>>           Accept Chang

Figure 35. Menu "Advanced Slit Commands"

In this dialog box, you have access to all motorized slits and to all turrets and can move all of them separately. You have two ways to enter or change a slit width or an offset position:

- 1. Enter absolute values in the input fields and execute the command by clicking on the "Move" button.
- 2. Use the arrow buttons to the right of the "Move" button. The effect of the buttons on the current value is:

<I>: Changes the value +/-10 pm for the slit-width and +/-0.01 nm for the wavelength << I >>: Changes the value +/- 100 pm for the slit-width and +/- 0.1 nm for the wavelength



<<< I >>>: Changes the value +/-1000 pm for the slit-width and +/-1 nm for the wavelength

If "Display Units" is set to "abs. wavenumbers" or "rel. wavenumbers", the region "Stage Offset" will change to relative wavenumbers (unit : cm).

Matching should be carried out in the following order:

1. Positioning Stage 1

a. The entrance slit should be opened to 10-20  $\mu$ m. All other slits that pertain to the current configuration should be completely opened.

b. Then, the first intermediate slit should be as narrow as possible to irradiate the second stage.

c. Next, Stage 1 has to be moved to the position where maximum intensity is displayed at the PMT or the CCD chip.

d. Step b and Step c should be repeated until only one position of Stage 1 leads to maximum intensity.

2. Positioning Stage 2

a. The positioning of Stage 2 occurs in the same way as in Stage 1 but with closing the second intermediate slit instead of the first one.

- b. The grating in Stage 1 must not be moved while matching Stage 2.
- 3. Positioning stage 3
  - a. If the positioning of Stage 3 occurs with a PMT at the exit slit, the exit slit

should be as narrow as possible to irradiate the PMT.

b. The intermediate slits can be reopened to measuring conditions. When using a CCD you must move Stage 3 until the peak of the wavelength corresponds to the central wavelength that is set in CCD-Settings in the "Measuring" menu.

c. The gratings in Stage 1 and Stage 2 must not be moved while matching Stage 3.