User Manual for operating the Synchrotron-based Fourier Transform Infrared (SR-FTIR) spectroscopy and microscopy beamline MIRAS

Prepared By: MIRAS Team

2024

1. Contents

2. Define experimental parameters

(with your local contact)

		Check box
1. Measurement mode	Microscope: Transmission, Reflection, ATR,…or Spectrometer	
2. Spectral resolution	Typically 4 cm^{-1}	
1. Aperture size	Typically 10 x 10 μm^2	
2. Integration time / number of scans	Typically 1 minute or 256 scans	
3. Spatial resolution (Step size)	Typically 5 μm^2	
4. Number of measurement points	Be aware of the max. measurement duration of the detector and the end of your shift.	

3. LOCAL CONTACT

You have a local contact (LC) allocated, for any problem or doubt contact him/her.

LC office Telephone numbers

Ibraheem	4085
Tanja	4042
Martin	4518

The local contact's working hours are from 9:30h-18h during the beamtime and could be on call between 19-22h.

You could call the floor coordinator after 22h only in case of emergency and until the availability of the LC at 9:30. The floor coordinator's telephone number is **5401**.

If you have problems with the **Front-End** you can call the control room (4331) 24h a day.

4. Prepare the beamline

4.1 Detector cooling

Fill the Single Element Detector (MCT) with liquid nitrogen (N2(l)), for your protection it is obligatory to use goggles and gloves (Figure 1b).

Fill the canister from the Dewar outside the hutch (Figure 1a). Turning the handle clockwise, the liquid N2 will flow. Make sure you close it properly after usage.

Figure 1a: N2 Dewar outside the hutch.

Figure 1b: Goggles, gloves and canister.

Place the funnel inside the MCT detector then add slowly the N_2 as shown in the figure below (Figure 2).

Figure 2: Detector filling with liquid nitrogen.

It is recommended when filling the detector to wait 5 minutes after filling it with 2 full funnels of N_2. In this way, the detector walls will be cooled down and will be easier to fill it completely.

When you start to see the liquid N2 pouring out of the detector neck, this means that the detector is filled.

If the detector was warm, wait for 15-20 minutes before starting any measurement in order to stabilize it.

Normally the N_2 liquid filling lasts for around 16h, therefore, it is recommended to refill the detector every 12-14 hours. It is recommended to make the refill between measurements and not during a running experiment.

4.2 Front-End

If you are planning to use the Synchrotron light, make sure that the Front end is open. That is indicated on the webpage and the status monitors:

https://www.cells.es/static/Files/Computing/Controls/Reports/msGUI.html

By default, the Front End should be open automatically. If not, please call:

- Your Local Contact
- Control room (4331)

5. Prepare the Software

5.1 Start OPUS

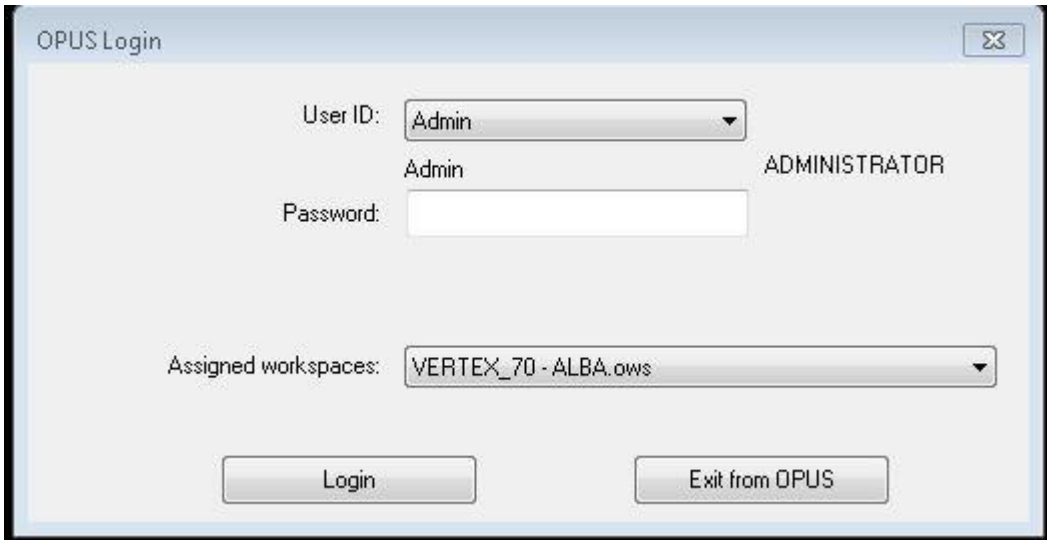
The FTIR measurements are controlled by the PC next to the microscope. (pcbl0106)

- Log in using your username and password provided to you by your Local Contact.

  Your username is the "u" letter plus the proposal number, i.e: ***u2024xxxxxx****.*
  Your password is the one you have chosen on the first day of beamtime. If you don't remember the password, you can ask your local contact to reset it for you.

- Start OPUS

  Password: No password is needed
  Assigned workspace: ***Vertex_70 - default.ows***
  Press login or Enter



6. Start the experiment

The purpose of the video wizard is to facilitate setting up and performing video-based mapping measurements. In the following each step of the Wizard will be explained. A more detailed description can be found in the OPUS help topics.

6.1 Starting the Video Guided Measurement

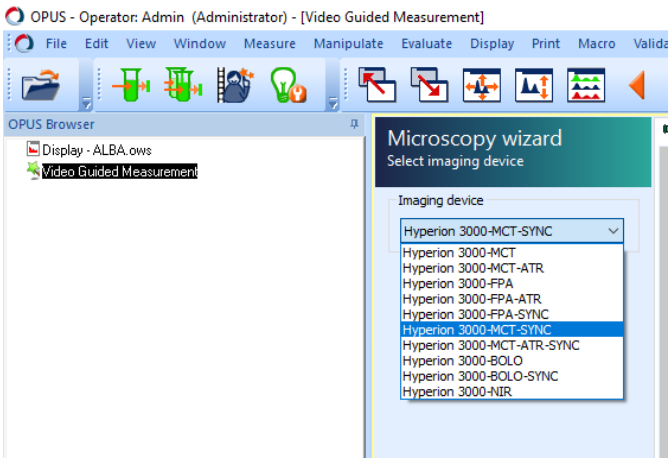
To start the **Video Guided Measurement** tool:

- either select in the Measure menu the **Video Guided Measurement** (wizard for video-guided measurements) tool.
- or click in the OPUS toolbar on the icon  .
- Thereupon, the window opens.

6.2 Selecting the device

On the first wizard page, you select the device configuration in question. Select the appropriate measurement parameters for your experiment:

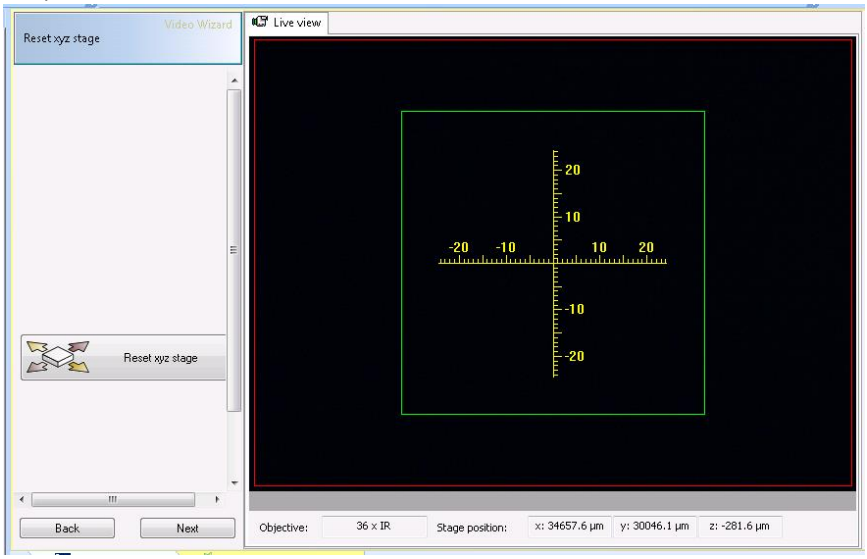
- For synchrotron light source with MCT detector: “Hyperion 3000-MCT-SYNC”
- For internal light source (Globar) with MCT detector: “Hyperion 3000-MCT”
- For internal light source (Globar) with FPA detector: “Hyperion 3000-FPA”
- For the ATR (coupled with the internal source) set to this mode. “Hyperion 3000-MCT-ATR” etc.



- After having selected the correct device, click on the Next button.

6.3 Resetting the motorized stage

- Stage reset means that the stage moves to its home position (x-position = 0 and y-position = 0). Note that the motorized stage is resettable only in the x- and y-direction, but not in the z-direction.



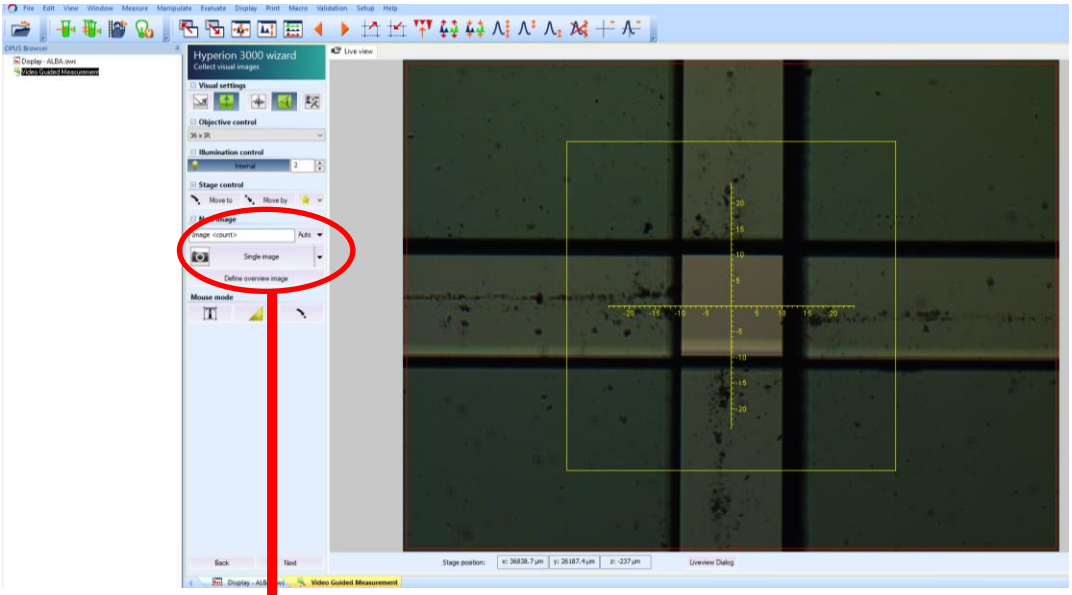
- **Make sure that the stage can move to its home position without hindrance. If required, remove the object(s) from the microscope stage and/or lower the condenser and/or move the stage slightly upwards.**
- Wait until the stage reset process is completed. As soon as the stage has reached its home position, the **Video Guided Measurement** switches automatically to the next page *Collect visual images*.

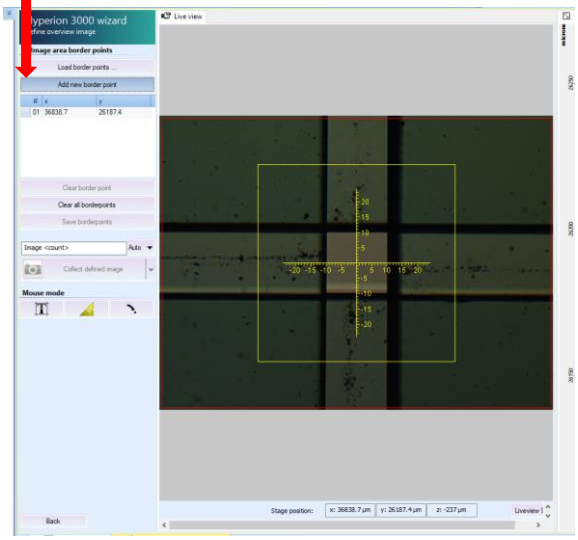
6.4 Setting the microscope for sample viewing

Next step allows you to select the preferred mode of operation (Transmission or reflection) and to visualize the sample and to take images. You can record a single image or larger maps. Before you can begin to examine the sample under the microscope, you have to:

- select the viewing mode (transmittance or reflectance) adequate to the sample you intend to analyse.
- set the illumination
- select an objective
- move the microscope stage in x- and/or y-direction until the sample is in the field of view
- focus on the sample

- Afterwards move the microscope stage in x- and/or y-direction until the sample area is in the field of view which is of interest to you.

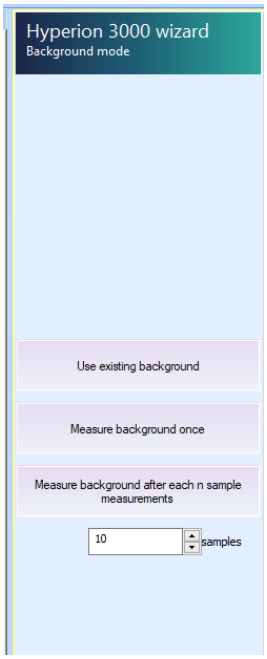




- When you find the sample area which you intend to analyse by means of FT-IR microspectroscopy take a snapshot of it. In this case, OPUS takes a single snapshot of sample video image currently shown in the live view.
- For larger maps. Click “Image of larger area…”. You need to select the border points for a bigger area. The minimum would be two points. Once you have selected the border points click on Collect Defined Image.
- For more details please go to *Help/Help Topics/Search* …. „Collect visual images”

6.5 Defining Background Mode

Define when and how often a background is to be measured. Depending on the microscope configuration, the following options are available on the wizard page *Background Mode*:



Use existing background: An existing background spectrum is used, provided that it is valid. In this case, the video wizard skips automatically the wizard pages for preparing the background measurement. So you can continue right away with defining the measurement positions for the spectroscopic sample measurement.

Measure background once: One background spectrum is measured before the sample mapping measurement.

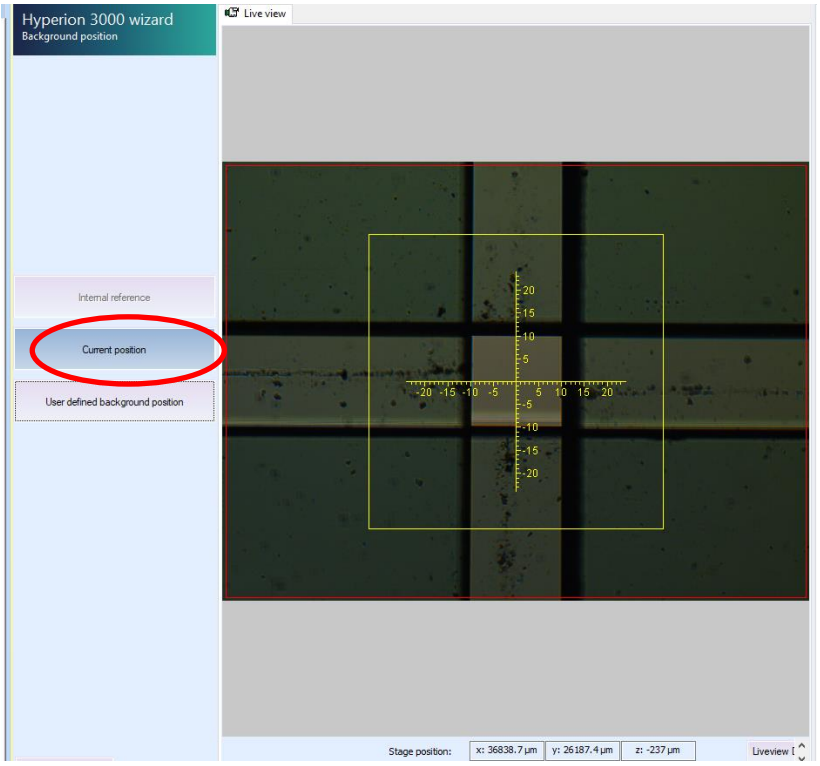
Measure background after every xth sample measurement: When you select this option, you can define a certain number of sample measurements after which a new background spectrum is to be measured automatically. This means, while the mapping measurement is running a new background spectrum is measured automatically after every xth sample measurement.

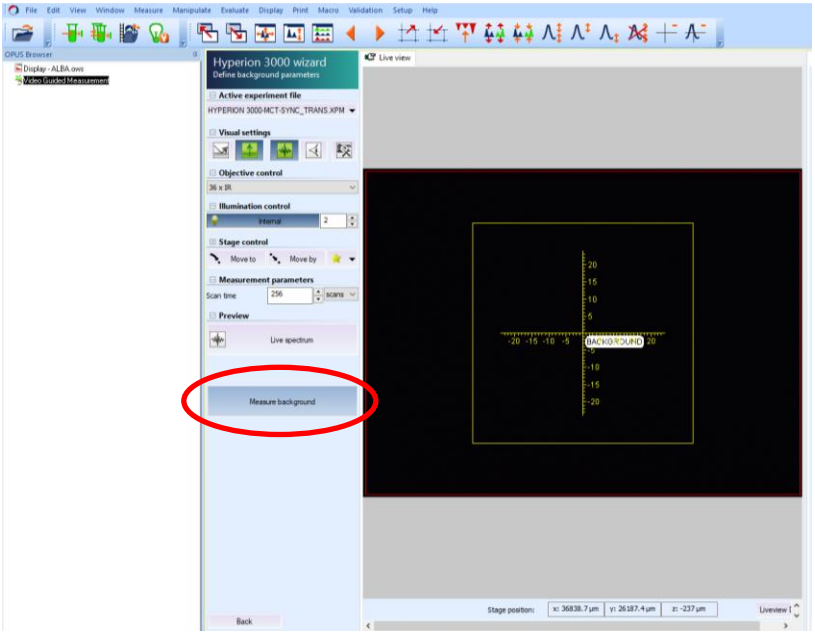
Depending on your experiment, select measure the background once or, if you are planning to do long measurements (3-6 hours or more), click measure background every X measurements. It is recommended to measure a background every 10-15 minutes to have it updated continuously along the experiment, so you can calculate this depending on the number of scans.

6.6 Defining the stage position for background measurement

Select the area where you want to measure the background using the joystick and click **current position**.

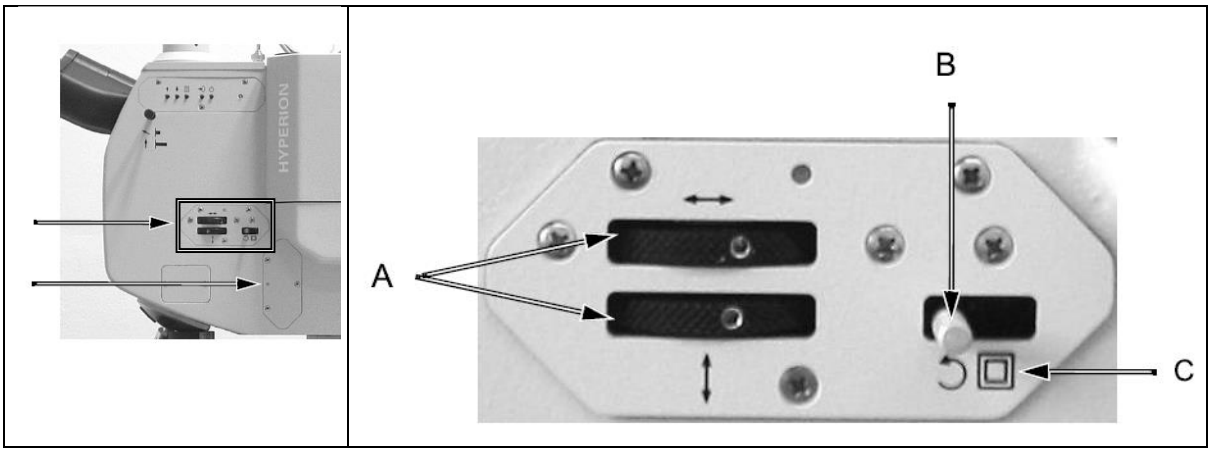
Select the background-position in a region empty of your sample.





6.7 Set apertures

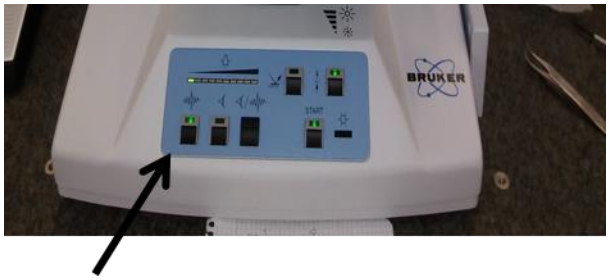
Before measure the background put the apertures using the wheels on the right part of the microscope.



6.8 Only for transmission mode: Align the condenser

This should be done every time you change experimental condition (sample, substrate, aperture, source, etc…)

Check the condenser position: Put the lower apertures of the microscope and focus on it by using the VIS mode of the microscope. To change to the visible mode, press the vis- button on the microscope terminal (second button from the left):

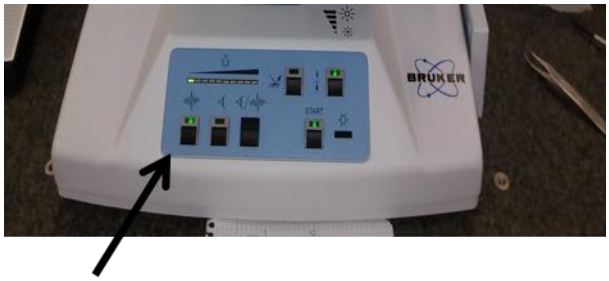


Use the bottom row of wheels of the condenser console to align the lower aperture in (X, Y and Z), in order to see the edges in focus. Now the condenser is aligned for the visible light.

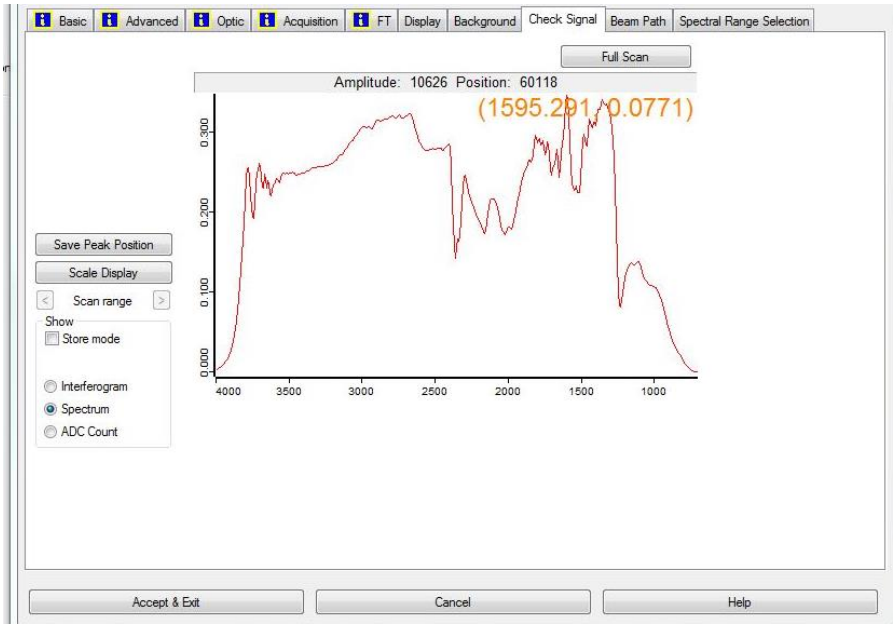
Now the aperture has to be removed again by opining fully the two wheels, unless measurements in confocal geometry is planned, please discuss it with your LC.

6.9 Synchrotron beam alignment

In order to align for the IR light, click on the Infrared Mode on the Microscope (left button).



Click check signal on the tab bar  and go to **check signal** tab. The intensity of the single beam spectrum (proportional to the power of the radiation reaching the detector) is given by the Amplitude when “Interferogram” or “Spectrum” is shown.

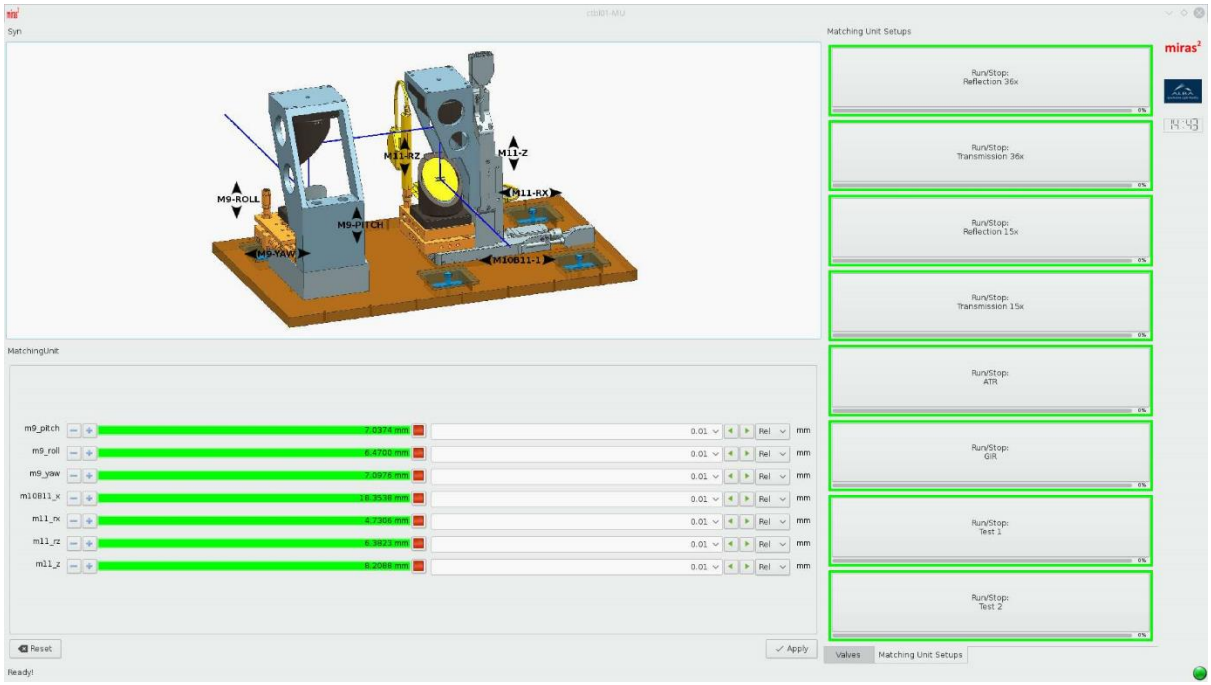


Tick spectrum to visualize the intensity and the Single beam spectrum shape. By aligning the condenser (only in transmission mode) and the mirrors of the matching box we aim to get the maximum amplitude and stability of the beam. (Note, if you are measuring using the internal source of radiation, then the matching unit is not needed for the alignment).

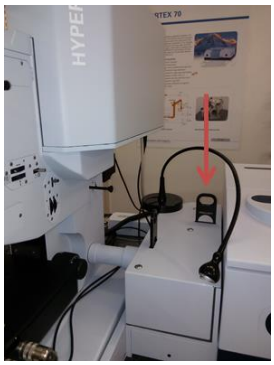
The matching box mirrors can help on improving the stability and the Amplitude of the beam. Move slightly the matching box mirrors by using the GUI at PC1 of the beamline to get maximum signal and stability. (It is recommended to move the mirrors in consequence

pattern, starting with the first mirror (M10B11_X) and ending with (m11_z) then repeat the cycle until you are satisfied with the spectral quality.

If this GUI is not appearing on the computer screen type “motors” in the Sardana interface.



Make sure that the maximum amplitude of the beam does not increase over 26.000 (Amplitude) as could generate artefacts in the spectra. If this is the case, use the ATTENUATOR and ask your local contact for help.

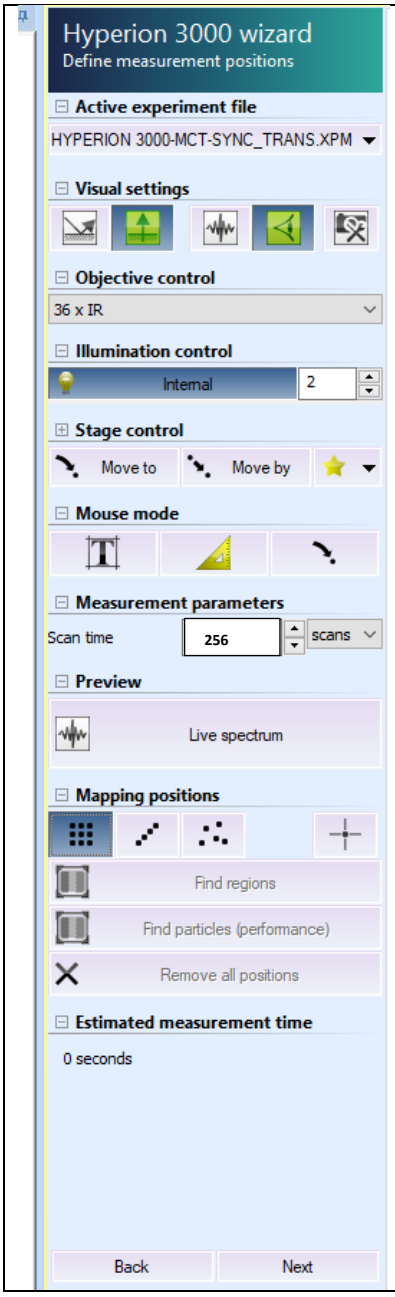


This image is showing the location of the attenuator between the microscope and spectrometer with the attenuator set.

6.10 Measure the background

Once the beam is aligned you can measure the background by clicking measure background.

Make sure the number of scans and the resolution are set correctly.



Mode	Depending on the kind of sample you intend to analyse, select the adequate measurement mode: **ATR, Reflectance or Transmission**.
Resolution	Define the spectral resolution. ***Typically we use 4cm^{-1}***. It is advisable not to specify a resolution higher than necessary because the higher the resolution is the longer the measurement will take.
Scan time	Specify the scan time (i.e. time for measuring one background spectrum) either in number of scans or in minutes. ***Typically, we use 256 scans.***
Objective	Select the objective with which you work.
Stage z-position control	This field can be ignored.
Measure background	When you click on this button, OPUS starts the background measurement. While the background measurement is running, a dialog shows the progress of the measurement. As soon as the background measurement is finished, the video wizard opens automatically the next page Define Measurement Positions.

Estimation of measurement time at a resolution of $4cm^{-1}$:

Number of scans	Time per measurment point
512	2min
256	1min
128	0.5in

6.11 Define Measurement Positions

There are 3 options of measurement – ***Random points, line or map***.

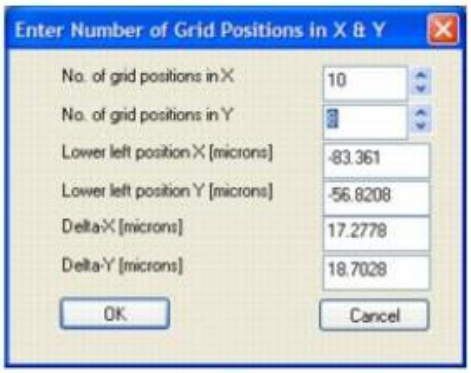
For defining the measurement positions for a mapping measurement, the following options are available:

-  Click on this icon to define randomly arranged measurement positions. (Single points map). Double-click, in case you want to add many points.

-  Click on this icon to define linearly arranged measurement positions. (Line map)

-  Click on this icon to define rectangular arranged measurement positions. (Raster scan map)

After you have selected the area you want to measure, you have to define the number of points; press “TAB” and the step size will be calculated. Alternatively, you can define the step size (Delta-X and Delta-Y) and the number of points will be calculated. You can also define the position of the first measurement point, which is in the lower left position of the measurement grid.



In many cases, you can increase the spatial resolution by overlapping measurement areas. For example, if you are using an aperture of 10 x 10 µm^2, you should use Delta-X and -Y smaller than 10 µm, ideally 5 µm, but this would depend on the experiment requirements.

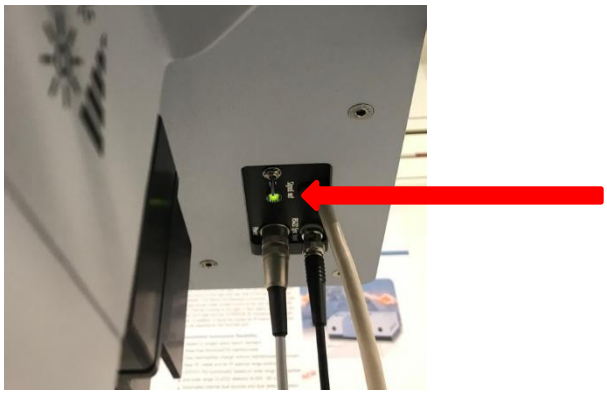
-  Click on to delete all measurement positions you have defined so far.

When you are happy with the selection click next.

6.12 Measurements by using the FPA detector

Fill the Focal Plane Array Detector (FPA) with liquid nitrogen (N2(l)), for your protection it is obligatory to use goggles and gloves (Figure 1b).

Before start cooling the FPA detector it is mandatory that detector is switched off and green light is off.



Fill the canister from the same Dewar outside the hutch (Figure 1a).

Place the funnel inside the FPA detector then add slowly the N_2 as shown in the figure below.

6.13 Starting the tool for the FPA measurements

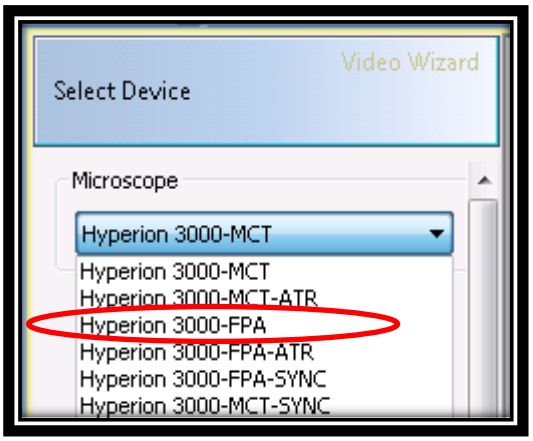
To start the **Video Guided Measurement**:

- either select in the Measure menu the **Video Guided Measurement** (wizard for video-guided measurements) tool.
- or click in the OPUS toolbar on the icon  .
- Thereupon, the window opens.

6.14 Selecting the FPA detector

On the first wizard page, you select the device configuration in question. Select the appropriate measurement parameters for your experiment:

- For internal light source (Globar) with FPA detector: “Hyperion 3000-FPA”



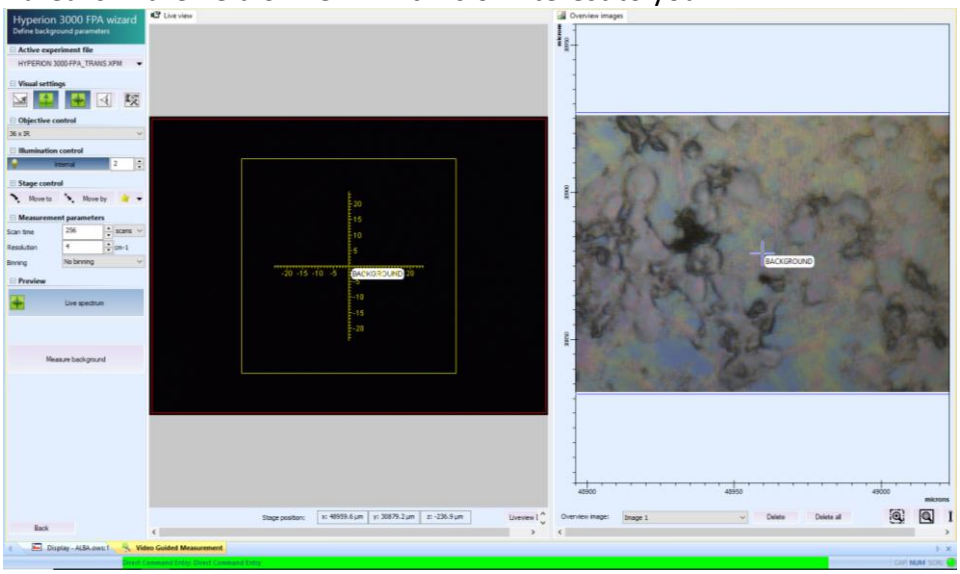
- After having selected the correct device, click on the Next button.

Setting the microscope for sample viewing

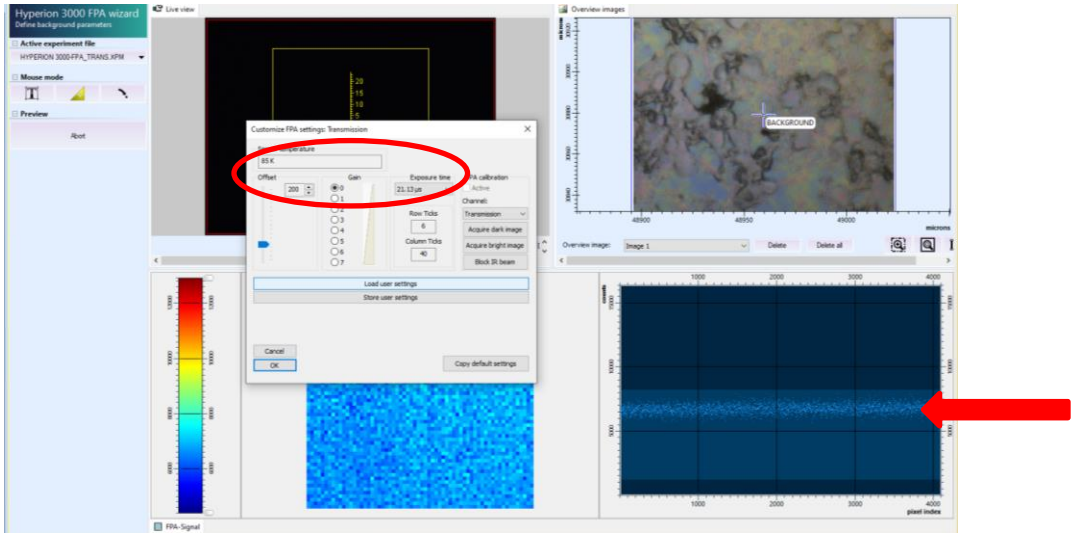
Next step allows you to select the preferred mode of operation and to visualize the sample and to take images (please see the section 6.14). Afterwards proceed with:

- select the viewing mode (transmittance or reflectance) adequate to the sample you intend to analyse.
- set the illumination

- select an objective
- move the microscope stage in x- and/or y-direction until the sample is in the field of view
- focus on the sample
- Afterwards move the microscope stage in x- and/or y-direction until the sample area is in the field of view which is of interest to you.

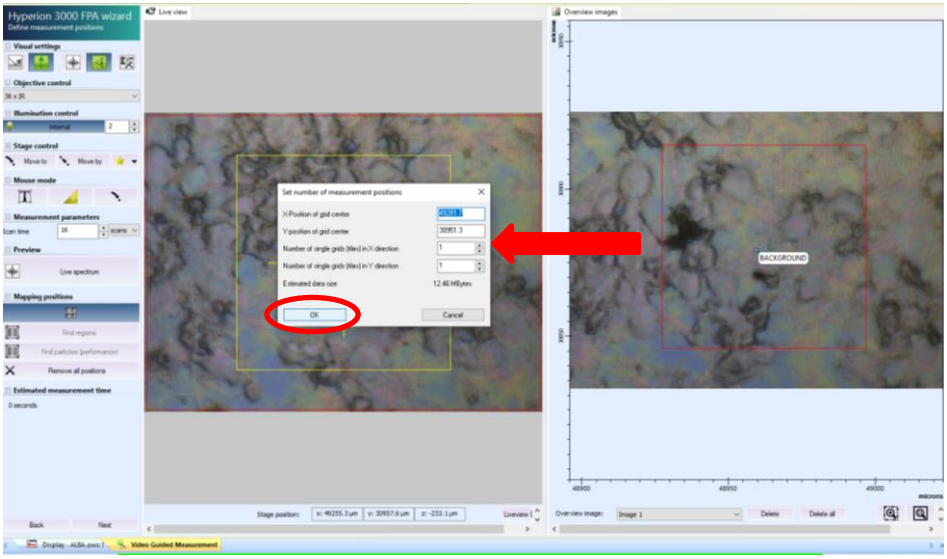


- When you select the sample area which you intend to analyse by means of FT-IR microspectroscopy take a snapshot of it. In this case, OPUS takes a single snapshot of sample video image currently shown in the live view.
- For larger maps. Click “Image of larger area…”. You need to select the border points for a bigger area. The minimum would be two points. Once you have selected the border points click on Collect Defined Image.
- Set the background as shown on the next image:
  - Click IR icon and the customize FPA:
  - Set Gain at 0



Click “Experts only” and set the next option “exposure time” and adjust the time in ms to bring a light blue line in the window and on the next image. Set OK and

- Set the Scan time and resolution



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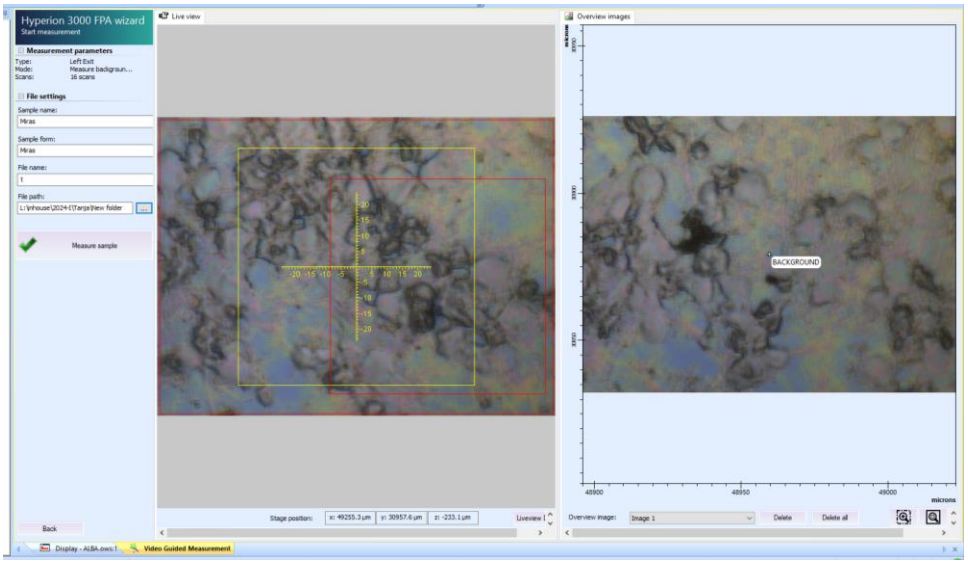
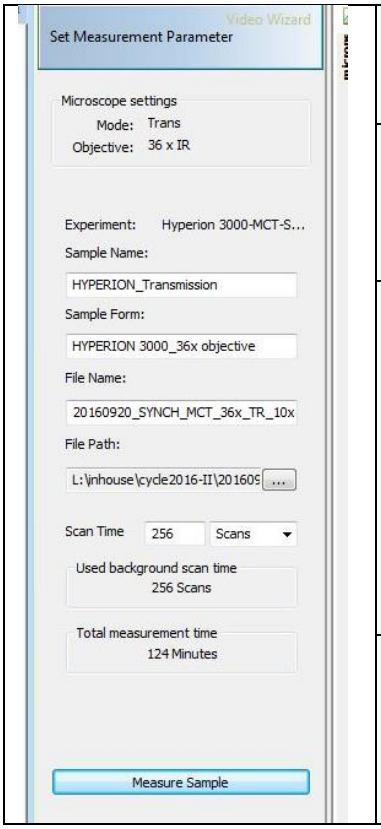
- **Set the area you want to scan and continue with the experiment**



6.15 Set Measurement Parameters and Start

You can change the name of the sample and the file and select the path to save the data.



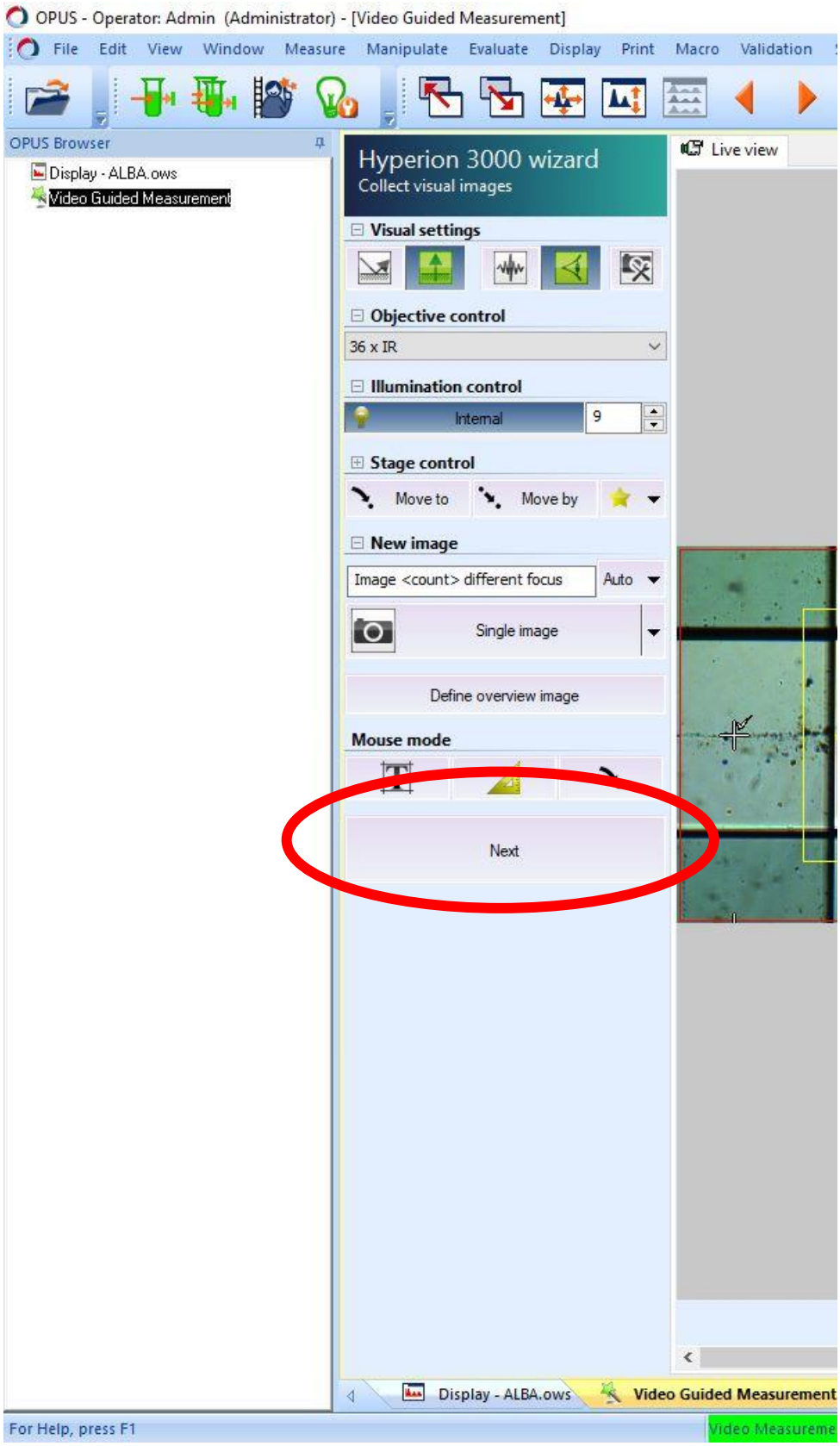
Sample name and File Name	Typically, we use the same name for sample and file. As you wish.
File Path	Make sure to safe your data in the **Z drive**. All data saved in ***Z*** are automatically backuped and you can access them remotely from anywhere.
Scan time	Time/Scans for one measurement point. Typically use the same value as set for the background measurement. Scanning time using the FPA detector is longer than what is normally estimated with the MCT detector. Also consider extra time in case the background measurements are repeated.
Total measurement time	…this is not always accurate. Better to calculate it yourself.

After checking the name, the path and the number of scans you can press start measurement.

6.16 Important!!! Finishing after the measurement.

After the measurement has been done, you have the possibility to take again a visible image of the sample. This might be useful in case you expect that the sample changed “visibly” during measurement. (at room conditions normally, the samples do not change, this is recommended if you are working with extreme conditions, like high pressure or temperature)

Finally, once the data is collected click **Next** to finish. The data is only saved after this selection.


OPUS - Operator: Admin (Administrator) - [Video Guided Measurement]
File Edit View Window Measure Manipulate Evaluate Display Print Macro Validation
OPUS Browser
Display - ALBA.ows
Video Guided Measurement
Hyperion 3000 wizard
Collect visual images
Visual settings
Objective control
36 x IR
Illumination control
Internal
9
Stage control
Move to
Move by
New image
Image <count> different focus
Auto
Single image
Define overview image
Mouse mode
Next
Live view
Display - ALBA.ows
Video Guided Measurement
For Help, press F1

7. Troubleshooting and frequently asked questions

7.1 To whom could I direct questions/comments/problems?

Questions and comments are always welcome! Please contact:

1. Your local contact.
2. Any MIRAS group member.
   https://www.cells.es/en/beamlines/bl01-miras/staff
3. The User office

   https://www.cells.es/en/users/contact

7.2 I don't remember my username or password.

Your username is the "u" letter plus the proposal ID, i.e: u2024xxxxxx.

An initial password is typically provided to you the first time you visit MIRAS for an experiment, with the recommendation to set your own password as well. If you don't remember the password or need to change it, you can directly put a new password from the User Office: https://useroffice.cells.es/ Just log in with your individual account (Auto-XXX), then go to Account Management on the left-side menu, search for your proposal number 2024xxxxxx and then you can set the new password directly.