EDINBURGH INSTRUMENTS

XS1 X-Ray Sample Chamber

User Guide


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XS1

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XS1 User Guide

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1.Introduction

The XS1 X-Ray Sample Chamber is an accessory for use with Edinburgh Instruments spectrometers which enables the user to perform x-ray excited photoluminescence measurements. By using a liquid light guide to route the emission to the sample chamber of the spectrometer, the user can perform time resolved and steady state measurements depending on the setup and hardware in the XS1 chamber and on their spectrometer.

The XS1 Sample Chamber offers a means to examine and characterise samples of different media with a series of simple exchange sample holders.

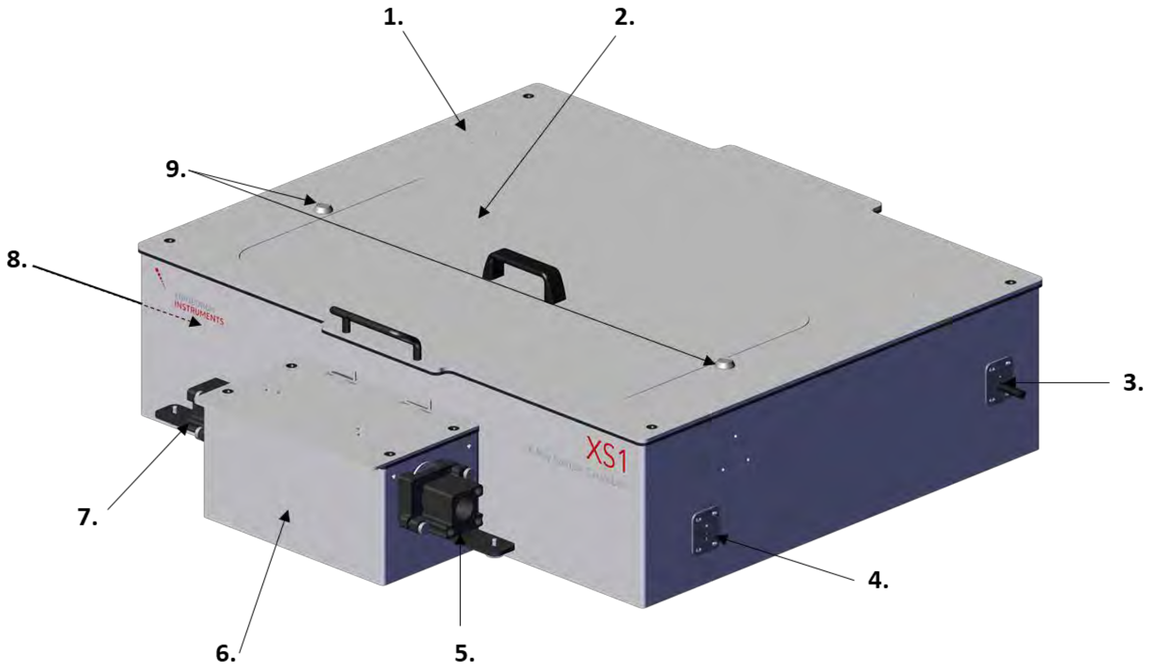
The modularity of the FLS1000 is carried through the XS1, with the option to configure their system with up to two x-ray sources which allow the user to perform the same measurements on samples using both x-ray and photoluminescence excitation. By expanding the excitation range of their Edinburgh Instruments spectrophotometer into the x-ray regime, the user can tap into a whole new realm of photoluminescence.

2. System Overview

2.1. Layout of System

The XS1 Chamber is laid out as detailed in Figures 1 (external) and 2 (internal) below.

A.



B.

Figure 1: A: External view of an XS1 sample chamber, complete with the laser steering box (for pulsed systems). **B.** A steady-state only XS1.

Item	Name	Description
1	Service lid	Hinged lid allowing access to the whole chamber
2	Inner lid	Routine access lid for sample exchange and source exchange
3	Feedthroughs for pulsed source cable	Cable clamps for the power cables and interface cables required for the pulsed source
4	Feedthroughs for pulsed source cable	Cable clamps for the power cables and interface cables required for the cw x-ray source
5	Laser mount for driving pulsed x-ray source	Laser mount to hold a diode laser or LED source to trigger the pulsed x-ray source. The mount is kinematic, allowing 3 points of adjustment to optimise input signal
6	Laser steering box (pulsed systems only)	This add-on steers the laser driving the pulsed source and adds an additional input for a diffuse reflectance measurement. The module includes steering mirrors, conditioning lens and mechanical shutters for the laser sources. For cw-only systems, a blank fascia panel is fitted
7	Diffuse excitation laser mount	A secondary laser mount to excite samples optically when loaded into the XS1
8	Main enclosure box	The outer housing is made from 5mm steel, to maximise absorption of the x-rays generated by the sources.
9	Key locks	These prevent the chamber from being opened accidentally when in use and form part of the interlock circuit

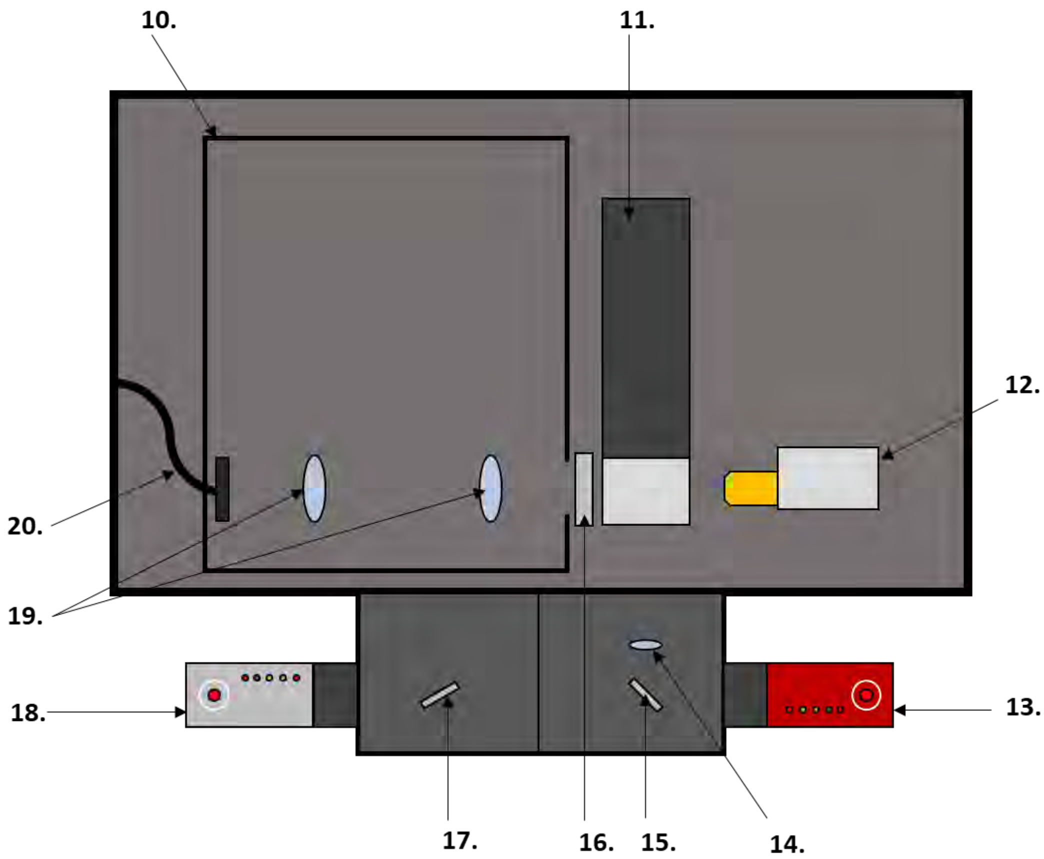


Figure 2: Simplified schematic of a full steady state and time resolved capable XS1

Item	Name	Description
10	Inner box	Secondary box to increase the shielding of the sample chamber
11	Hamamatsu N5084 source	Laser diode-driven x-ray source which produces a pulse of x-ray emission mirroring that of the source i.e. in both pulse duration and repetition rate
12	MoxTek MagPro source	Steady state x-ray source with a 12 W output power (60 kV supply voltage)
13	HPL laser	Pump source for the N5084 tube. Running at repetition rates of up to 40 MHz, this laser diode has two pulse mode settings – short pulses similar to the EPL lasers (<150ps) and a long pulse for high power applications (< 1ns). A VPL can be used for MCS measurements, allowing excitation pulses up to 1 ms in duration.
14	Beam conditioning lens	Optical element placed to optimise the illumination of the photocathode of the N5084
15	Steering mirror	Steers the laser beam through the lens to optimise the intensity on the photocathode
16	Sample holder	Drop-in sample holders for different media – see section on sample holders
17	Steering mirror	Second steering mirror to allow diffuse reflectance measurements
18	Secondary laser diode source	This position allows the laser to be used for diffuse reflectance
19	Collection optics	UV fused silica lenses give broad transmission range, and two lens assemblies are used to maximise the collection efficiency from the sample
20	Light guide (on translation stage)	5mm core liquid light guides are used to maximise the acceptance angle and minimise signal loss on coupling. Different ranges are available optimised for different spectral regions (see section)

2.2. Safety Features

The XS1 comes with integrated safety features designed to ensure that the accessory can be operated safely by all users. These features are described in detail in the subsections below.

Please note the following points:

1. Edinburgh Instruments fits and tests these safety features to the XS1. It is the user's responsibility to ensure that all connections remain in place.
2. The sources operate in such a way that if the interlock is broken during operation, their HV will shut off and must be manually restarted. The source cannot be turned on until the circuit is complete again.
3. These mechanisms should never be tampered with or bypassed.

X-ray laboratory safety is the user's responsibility – all laboratories should be examined by an appointed Radiation Protection Advisor to ensure compliance with the locally applicable ionising radiations regulations. Edinburgh Instruments will not be responsible for ensuring an x-ray laboratory is compliant to applicable ionising radiation regulations.

2.2.1. XS1 Chamber Construction

The chamber is constructed in such a way to ensure that there is at least 5mm steel around the whole enclosure. In addition to this, all feedthroughs for cables and optics are clamped to minimise leakage. The chamber is tested using a Geiger counter to ensure that there are no weak points around the wall joints, lids and feedthrough positions.

2.2.2. Interlock Circuits

The interlock circuit is routed as shown in Figure 3 below.

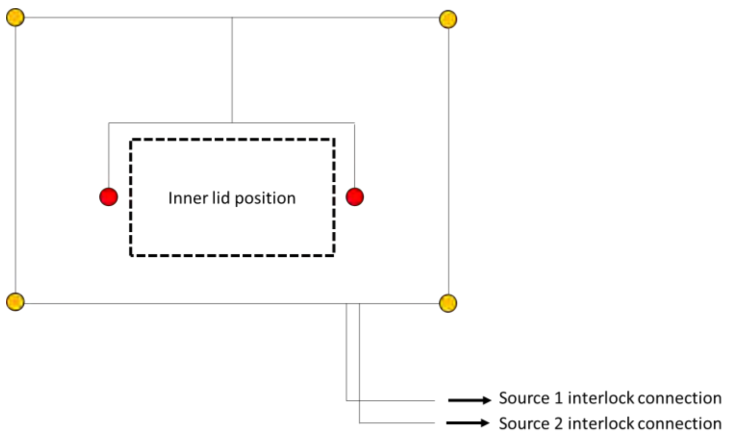


Figure 3: Interlock circuit in the XS1 showing the different break points

Figure 3 shows two different break points – the red indicates the locking keys which are used for routine use and access. When both locks are engaged, the lid cannot be removed and the interlock circuit is complete, meaning that the x-ray sources may be turned on and used. If one of these keys is not engaged, the source cannot be turned on.

The yellow positions indicate the location of the four screws on the hinged service access lid. These screws must be fully turned into their locators for the circuit to be complete. Failure to do so will result in the source not turning on.

2.2.3. Key lock mechanism

The key-locks are fitted with a failsafe pin which prevents the key from being turned when the inner lid is not in place, therefore preventing a user from accidentally completing the interlock circuit and allowing the source to turn on. The mechanism is shown in Figures 4 and 5 below.

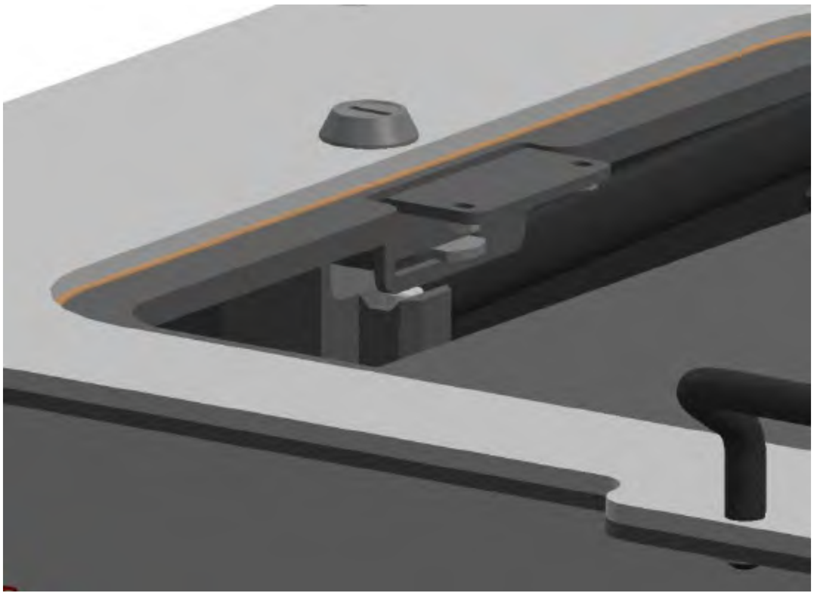


Figure 4: Safety pin mechanism, showing the arrangement when the top lid is in place (hidden here for clarity)

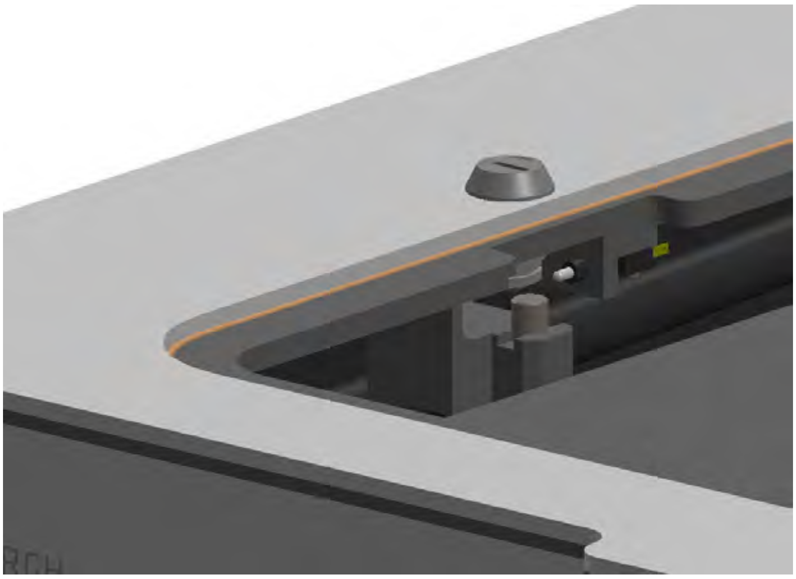


Figure 5: Safety pin mechanism, showing the arrangement when the top lid removed, with the pin up

2.3. Electrical Connectivity

As shown in Figure 1, there are feedthroughs in place to allow the electrical connections to the sample chamber. There are two such ports, one for the CW source and one for the pulsed option.

The scheme is shown below in Figure 6

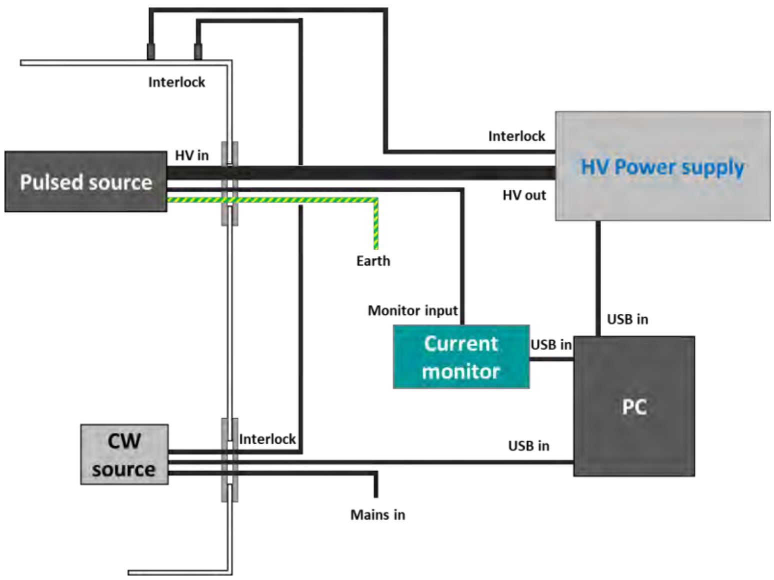


Figure 6: Electrical connectivity for both sources

2.4. Chamber Access

The XS1 offers two means of access, these being the routine access lid and service lid.

2.4.1. Routine access lid

Normal access should be done via the routine access lid. The following procedure should be followed:

1. Check that the source(s) are not emitting and power off sources if required (see section 4)
2. Ensure that all laser sources are switched off
3. Insert key in one lock and turn to open position
4. Remove key and insert into second lock. Turn to unlock.
5. Lift off lid.

The following actions require use of only the routine access lid:

- Sample exchange
- Source exchange
- Tuning optics for the straight-through path

2.4.2. Service lid access

For more complicated requirements, it may be necessary to open up the service lid. This large lid is gas-strut assisted to aid users in opening and closing the chamber.

To open the service lid, follow the procedure below:

1. Check that the source(s) are not emitting and power off sources if required (see section 4)
2. Ensure that all laser sources are switched off
3. Insert key in one lock and turn to open position
4. Remove key and insert into second lock. Turn to unlock.
5. Lift off routine access lid.
6. Remove the 4 screws at the corners of the service lid
7. Lift the lid up using the handle – when the lid has reached an angle of approx. 30 degrees, the gas struts will engage and assist the user with the lifting of the lid

Service lid access will be required for tasks such as:

- Changing the LLG for one with a different transmission range
- Installation of a new source for the first time (contact EI to ensure that the source to be integrated is compatible with the XS1) and removal of a source

- Removal of a source from the XS1

3. Options for the XS1

3.1. Source options

The XS1 has the option to have either pulsed, CW or both pulsed and CW sources integrated. The sources are described below. The key specifications of the sources are listed in Appendix 1.

3.1.1. CW source option – MokTek MagPro

We offer the MoxTek MagPro 12 W cw x-ray source as the solution for steady-state only requirements. With x-ray energies up to 70keV depending on the model, the power supply is compact and easy to integrate within the XS1. The power can be set by fine-tuning the current and voltage levels in the source software (see Figure 15).

The target material choice is at the user's discretion and should be chosen based on any specific needs of the samples under investigation.

3.1.2. Pulsed source option – N5084

We offer the Hamamatsu N5084 x-ray tube as the pulsed option. This source can be run in TCSPC mode (when combined with a HPL laser diode), or MCS mode with the appropriate HPL or VPL. The N5084 can also be run in a quasi-cw state by running the N5084 with high rep rate HPLs.

The N5084 works by generating an x-ray pulse with the same temporal profile and repetition rate as the input pulse from the optical source. So long as the current remains at a safe level on the x-ray target, this will allow the user to use pulses from the fs to ms range, depending on the source involved, giving access to a huge range of lifetimes.

This source requires the laser steering box to be included, as shown in Figure 1.

3.2. Sample holder options

There are five options for sample holders to be used within the XS1. These sample holders allow solids, powders and liquids to be investigated.

The sample holders for the XS1 have been designed to drop into the cradle inside the chamber. The groove in the cradle mates with an external ridge on the sample holders themselves, making for a fast, secure and repeatable means of removing and replacing different samples and holders. The cradle also features two thumbscrews to aid the user to position the sample as close as possible to the x-ray source.

The mechanism is shown in Figure 7 below:

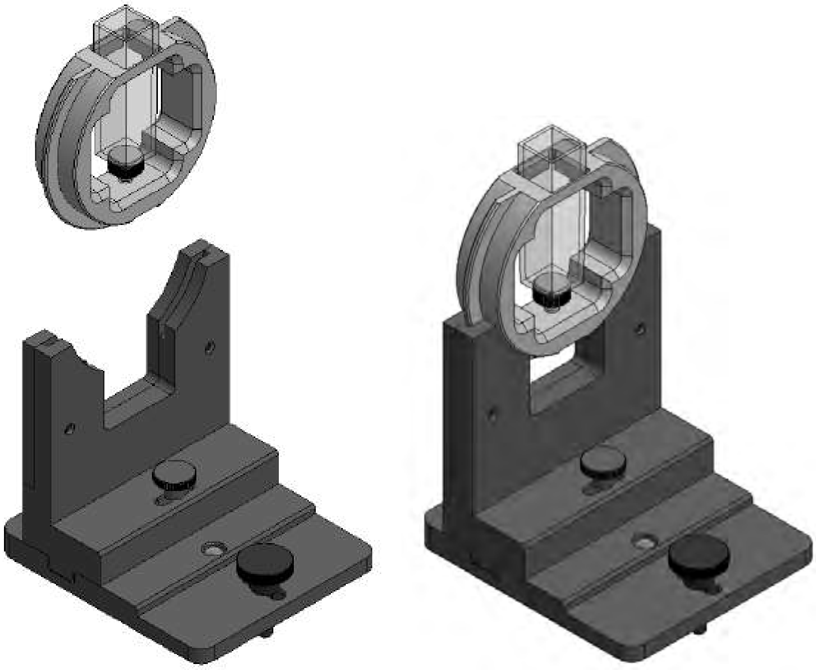


Figure 7: Drop-in sample holder cradle, showing a standard cuvette holder being fitted.

3.2.1. Powder cell

The powder cell option is illustrated in Figure 8. This sample holder has two UV fused silica windows, and to load the sample in the user removes the 3 x M3 pozi-drive countersunk screws (visible in black in Figure 8), presses in the powder, and re-seals the powder cell.

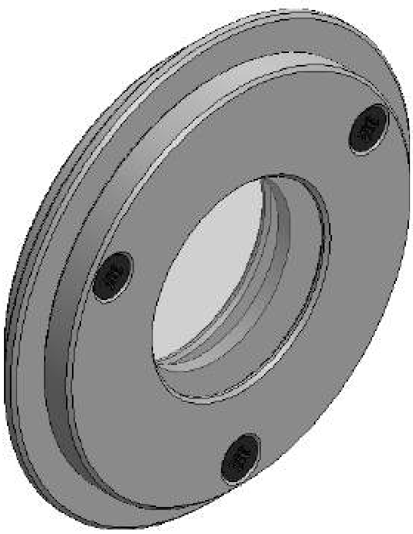


Figure 8: Powder cell sample holder for the XS1

3.2.2. Solid sample holder

The solid sample holder allows the user to clamp solids with a minimum dimension of 10mm to samples up to 25mm square. The two adjustor thumbscrews (shown in Figure 9 below) allow the user to position the sample in the centre of the holder before loading into the cradle in the XS1.

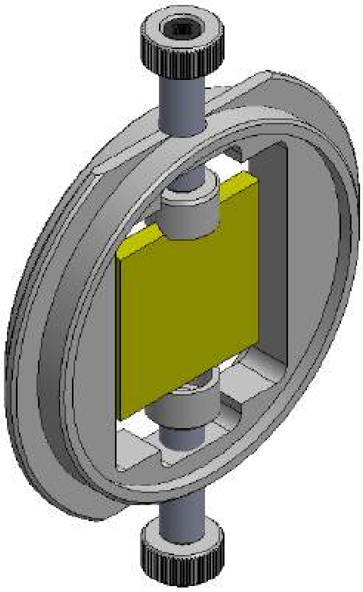


Figure 9: Solid sample holder for the XS1

3.2.3. Single crystal/small volume sample holder

This option allows the user to measure single crystals or low powder volumes by loading material into a 100mm EPR tube. The screw in the bottom of the sample holder (Figure 10) can be adjusted to centre the sample in the holder for maximum excitation efficiency, and locked using a 7mm open spanner.

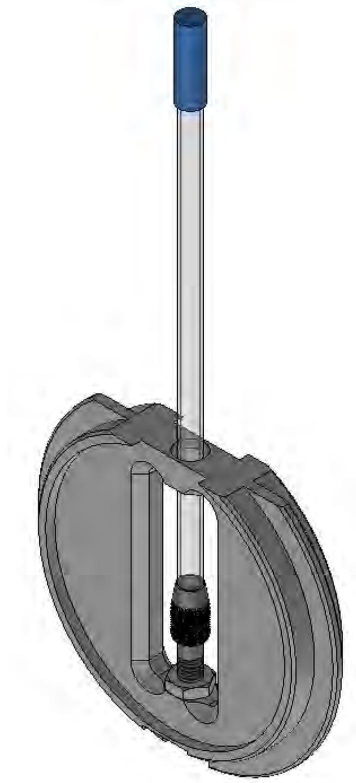


Figure 10: Small volume sample holder for the XS1

3.2.4. Flat cell cuvette sample holder

For small volumes of liquid, this sample holder holds a 1mm path length standard width cuvette. This sample holder, shown in Figure 11, is adjustable in height with a thumbscrew.

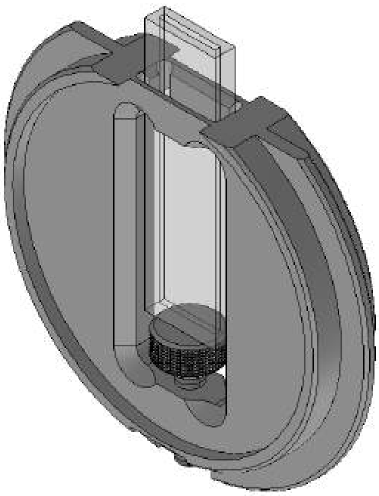


Figure 11: Flat cell cuvette sample holder for the XS1

3.2.5. Cuvette sample holder

This sample holder allows standard volume cuvettes to be fitted in the XS1. The standard 12.7 x 12.7 footprint allows full volume and micro-volume cuvettes to be installed depending on the user's preference and the sample under investigation.

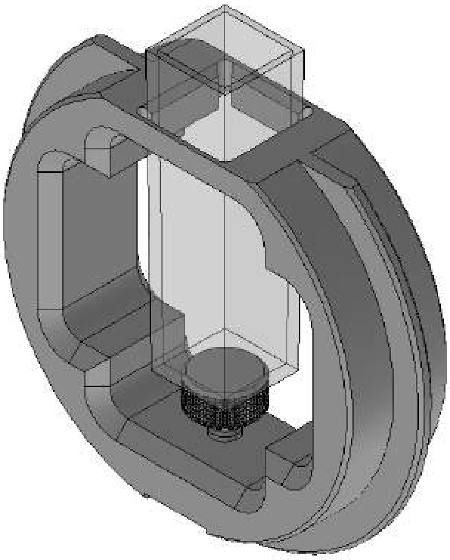


Figure 12: Cuvette sample holder for the XS1

In addition to this, we can offer a cradle to adapt to a standard front-face sample holder which allows the user to install each of these sample holders into an FLS1000 sample chamber to perform standard photoluminescence measurements.

3.3. Light guide options

We offer several light guide options available, allowing the user to measure from 220nm to 2000nm depending on the choice of light guide. Speak to your sales representative to ensure that you have the best option for your sample measurements.

Coverage	Range (nm)
DUV-Vis	220-600
UV-Vis	300-650
Vis	340-800
Vis-NIR	420-2000

4. Connecting the XS1 Chamber to Edinburgh Instruments Spectrofluorometers

4.1. Optical connection to Edinburgh Instruments spectrometers

The XS1 is a remote accessory, requiring an optical link to be made to a spectrophotometer by a liquid light guide (Figure 13). As such, the user must have not only the XS1 and liquid light guide supplied, but also the appropriate liquid light guide launcher for their spectrophotometer (Figure 14).

The accessory is designed to take full advantage of the flexibility and modularity of the FLS1000, though can be used with an FS5 (contact EI Sales to discuss this).

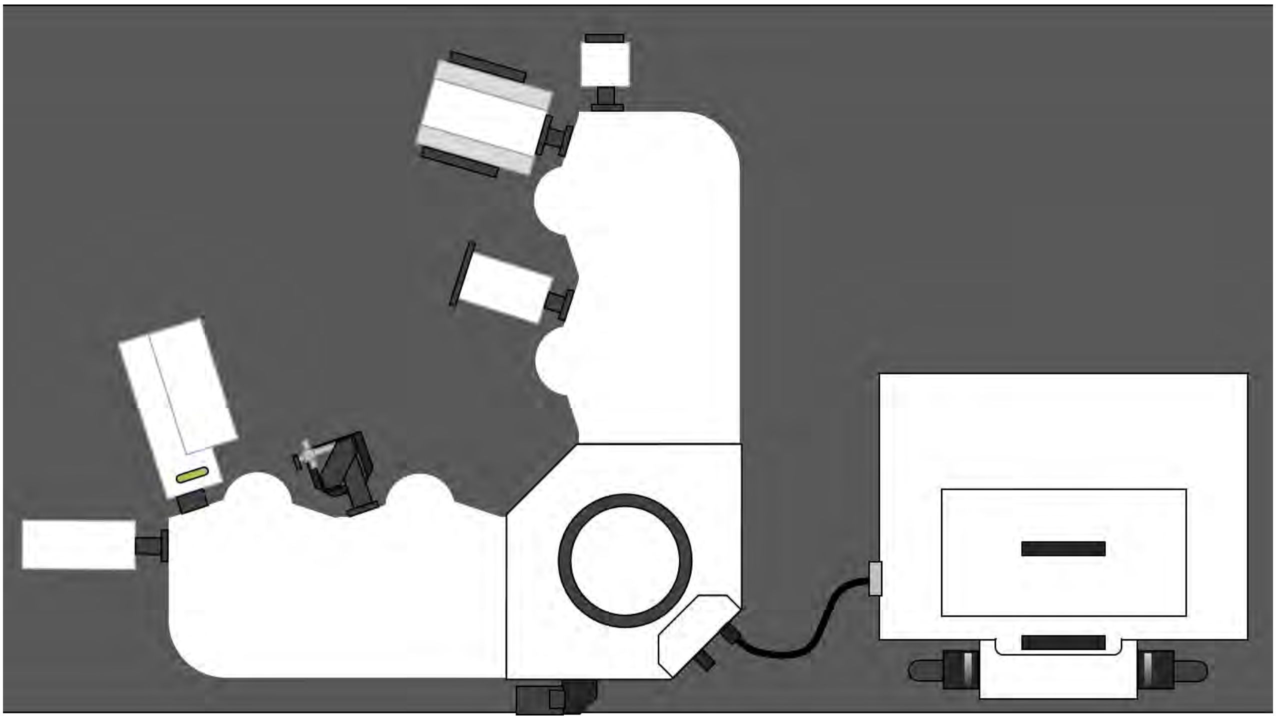


Figure 13: Connection to FLS1000

Figure 14: FLS1000 liquid light guide launcher (L) and SC-50R fibre launch cassette for the FS5 (R)

Upon installation of an XS1 Sample Chamber, a light guide will be fitted into the XS1 and fed through to the FLS1000. The process of installing or changing a light guide to one of a different transmission range is covered in Procedure 6.1.2.

For use with older Edinburgh Instruments spectrometers, please get in touch with an EI sales representative to confirm compatibility and specifications.

5. Measurements using the XS1 Sample Chamber

The spectrometer configuration and choice of x-ray source dictate the measurements possible. The table below illustrates what is and is not possible with each source and the spectrometer specification. This section will run through the steps required to perform each measurement type in the Fluoracle environment.

Spectrometer Configuration	Steady state	MCS	TCSPC
FLS1000-S FS5 CORE	✓	×	×
FLS1000-SM FS5 MCS	✓	✓	×
FLS1000-STM FS5 TCSPC MCS	✓	✓	✓

If you're unsure about the exact specification of your spectrometer, please get in touch with EI Sales to discuss your system with your local representative.

On top of this, the specific arrangement of the XS1 also shapes the measurement options

XS1 Configuration	Steady state	MCS	TCSPC
MagPro only	✓	×	×
N5084 only	✓*	✓	✓
MagPro and N5084	✓	✓	✓

The (*) in the table above indicates that although spectral measurements are possible with a N5084 only arrangement, this is performed using a quasi-CW situation, i.e. the HPL is set up to run in high power mode and at the maximum rep rate with an intensity on the x-ray tube of ≤10 μA.

Before running any measurements with the XS1 chamber, ensure that the XS1 is configured correctly (source position, sample holder choice, LLG fitted) and that the manual shutters are both closed.

Since the XS1 is connected to the FLS1000 by a light guide, the transmission curve of the light guide will need to be taken into account to give accurate spectral measurements. As a result, a separate emission light path should be set up for each light guide used with the XS1.

This will be taken into account at the point of installation for new systems including an XS1 accessory, and for upgrades a correction file will created either on-site or in advance.

5.1. Steady State Measurements – MagPro CW source

To perform a steady state measurement using the MagPro source from a system-off state, follow the procedure outlined below:

1. Power on the spectrometer and launch Fluoracle.
2. When the spectrometer has fully initialised:
    a. Select the appropriate light paths on the signal rate window (Figure 15), i.e. MagPro light path as the excitation source, and the XS1 as the emission path with the correct emission properties (grating, detector and light guide).
    b. Set the emission wavelength to the peak emission of your sample (if this is known) and the bandwidth to 0.5 nm.

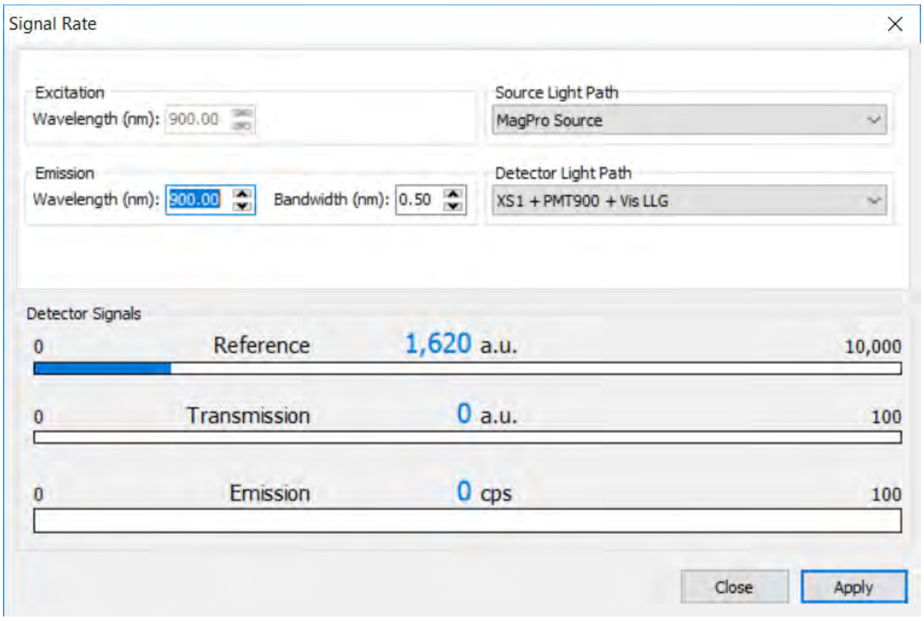


Figure 15: Signal rate screen, MagPro source for CW measurements.

3. Launch the MagPro control software (Figure 16 – supplied with the source, the installation engineer will have left a shortcut on the desktop of the spectrometer computer).
4. Choose the parameters required (note that the max voltage and current can be set independently to give a maximum power of 12 W). When you've set the desired parameters, click 'X-Ray Enable'. The button will turn red, indicating the source is live.

Figure 16: MagPro control software showing it after initialisation (left) and when the source has been started (right)

5. Return to Fluoracle. Adjust the bandwidth until you see an acceptable count rate. For unknown samples leave the bandwidth at 0.5 nm.
6. Perform an emission scan over the desired range, and upon completion review the monochromator settings used (i.e. scan range, resolution, bandwidth and integration time) and adjust as required. Details of scan setup are covered in the spectrometer manual.
7. Remove the sample and either fit a new sample before returning to step 3, or power off the spectrometer.
8. When all measurements are completed, power off the MagPro (by Clicking the red 'X-ray disable' button).

5.2. Steady State Measurements – N5084 Pulsed Source

To perform a steady state measurement using the N5084 source from a system-off state, follow the procedure outlined below:

1. Power on the spectrometer and launch Fluoracle.
2. When the spectrometer has fully initialised:
    a. Select the appropriate light paths on the signal rate window (Figure 17), i.e. TCSPC diode light path as the excitation source, and the XS1 as the emission path with the correct emission properties (grating, detector and light guide).
    b. Set the emission wavelength to the peak emission of your sample (if this is known) and the bandwidth to 2 nm.

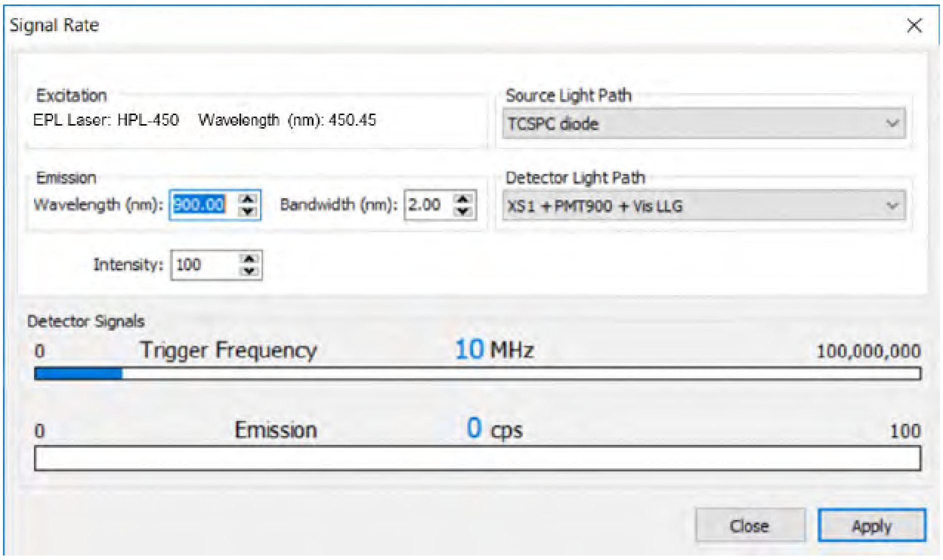


Figure 17: Signal rate screen for a TCSPC source configured for quasi CW measurements.

3. Launch the XMaster control software (Figure 18). The COM ports will have been set up during installation, so when ready to use select 'HV On'. The system will ramp up with a warning message displayed on the window, and the source on beacon will light up.

Figure 18: XMaster control software

4. Turn the ND wheel on the laser mount for the HPL to the lowest setting, select the maximum rep rate on the unit (40 MHz) and turn the laser diode on high power mode by pressing and holding the power on button for 3 seconds before releasing. The emission LED will flash, indicating that the module is running on high power mode.

Note: If the laser source has been changed for your measurement, you will need to tune the path of the beam. See Procedure 6.1.4 for details on this process.

5. Open the laser shutter and gradually reduce the attenuation of the ND wheel until the reading under 'Current meter' in the XMaster software displays close to 10 μA. If the current exceeds 10 μA then the HV will turn off – this is a safety mechanism to prevent damage to the N5084.
6. Return to Fluoracle and the signal rate screen. Adjust the bandwidth until you see an acceptable count rate.
7. Perform an emission scan over the desired range. Upon scan completion review the monochromator settings used and adjust as required. Details of scan setup are covered in the spectrometer manual.
8. Remove the sample and either fit a new sample before returning to step 3, or power off the spectrometer.
9. When all measurements are completed, power off the laser/LED source first, close the shutter on the XS1 and then power off the N5084 (by Clicking the 'HV Off' button).

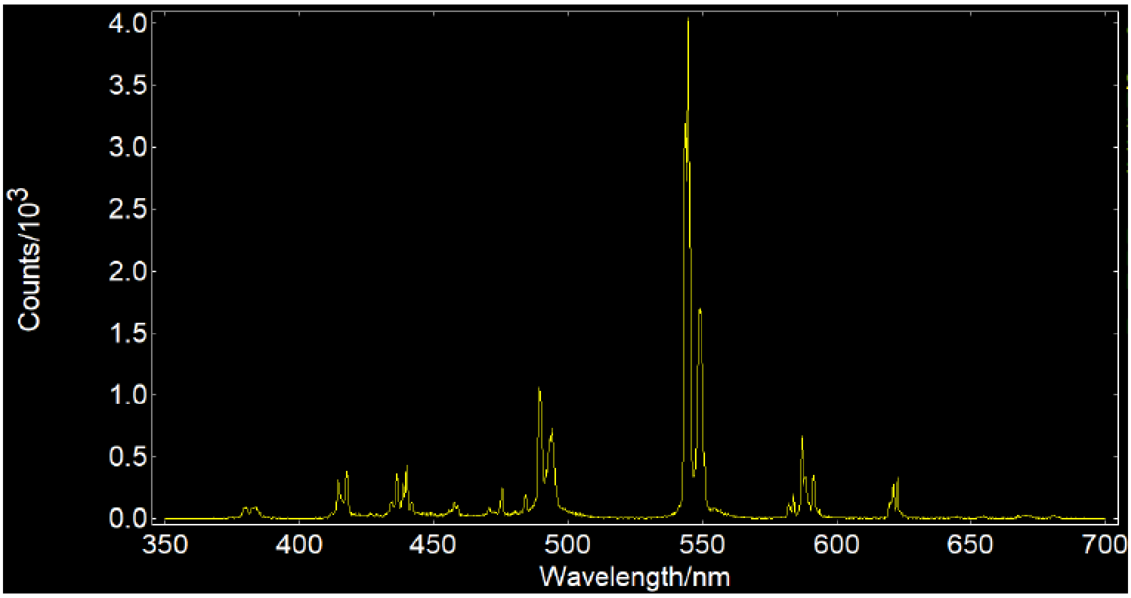


Figure 19: Example of a steady state measurement taken using an XS1 and using the N5084 (in quasi cw mode) as the source.

5.3. MCS Measurements – N5084 Pulsed Source

To perform a Multi-channel Scaling (MCS) measurement using the N5084 source from a system-off state, follow the procedure outlined below:

1. Power on the spectrometer and launch Fluoracle.
2. When the spectrometer has fully initialised:
    a. Select the appropriate light paths on the signal rate window (Figure 20), i.e. MCS diode light path as the excitation source, and the XS1 as the emission path with the correct emission properties (grating, detector and light guide).

b. Set the repetition rate for the source in Setup → EPL MCS Setup, then set the emission wavelength to the peak emission of your sample (if this is known) and the bandwidth to 0.5 nm.

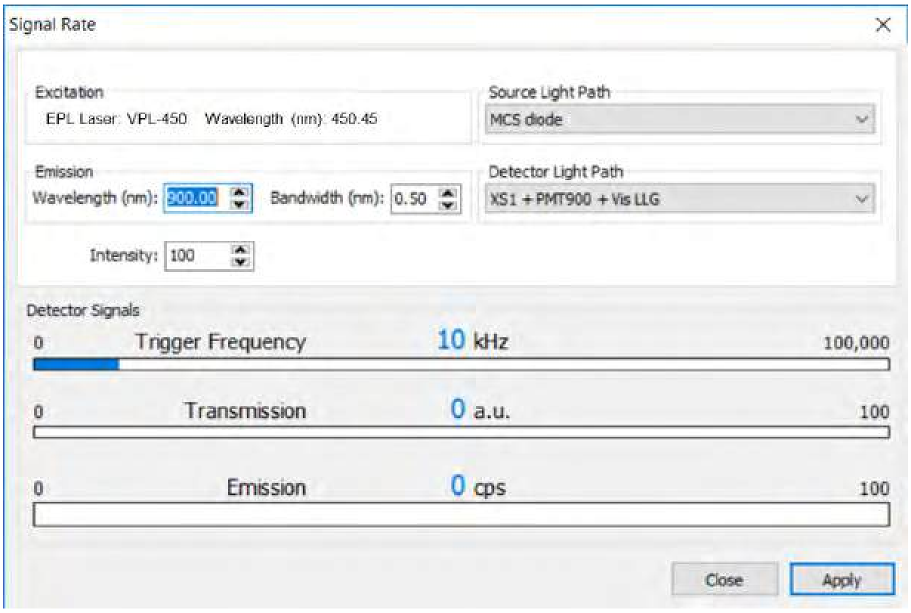


Figure 20: Signal rate screen, MCS measurements.

3. Launch the XMaster control software. The COM ports will have been set up during installation, so when ready to use select 'HV On'. The system will ramp up with a warning message displayed on the window, and the source on beacon will light up.
4. Set up the laser diode:
   a. Turn the ND wheel on the laser mount for the laser diode to the minimum value.
   b. If using a VPL450, select the desired pulse duration and turn on using the red power button on the back.
   c. If using a HPL450 turn the dial to triggered mode and turn the laser diode on high power mode by pressing and holding the power on button for 3 seconds before releasing. The emission LED on the HPL450 will flash, indicating that the module is running on high power mode.

Note: If the laser source has been changed for your measurement, you will need to tune the path of the beam. See Procedure 6.1.4 for details on this process.

5. Open the laser shutter and gradually reduce the attenuation of the ND wheel until the reading under 'Current meter' in the XMaster software displays close to 10 µA. If the current exceeds 10 µA then the HV will turn off – this is a safety mechanism to prevent damage to the N5084.
6. Return to Fluoracle and adjust the bandwidth until you see an acceptable count rate. For unknown samples simply leave the bandwidth set to 0.5 nm.

7. Set the scan parameters for the MCS measurement as per the spectrometer manual and run the measurement. Upon scan completion review the settings used and adjust as required.
8. When the measurement(s) are completed, power off the laser/LED source first, close the shutter and then power off the N5084 (by Clicking the 'HV Off' button). Remove the sample and either fit a new sample before returning to step 3, or power off the spectrometer.

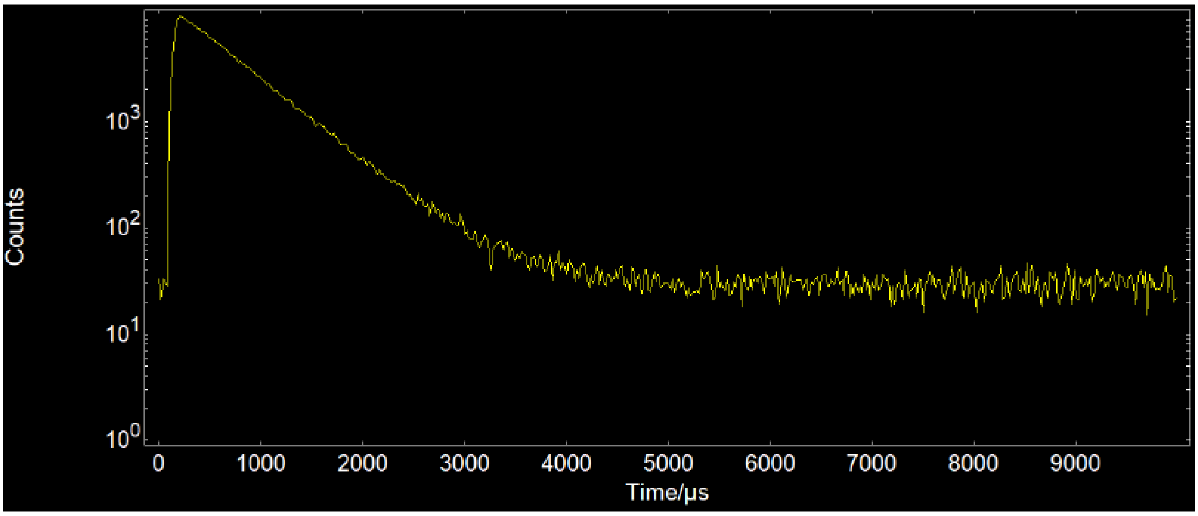


Figure 21: Example MCS measurement taken using the XS1 chamber with the N5084 as the source (pumped in this case by a VPLED400 with an optical pulse duration set to 100 µs, driven at a frequency of 1 kHz).

5.4. TCSPC Measurements – N5084 Pulsed Source

To perform a Time Correlated Single Photon Counting (TCSPC) measurement using the N5084 source from a system-off state, follow the procedure outlined below:

1. Check the arrangement of the XS1 i.e. light guide installed, laser diode installed (HPL450) sample holder, source positioning. Set it up for your desired measurement and place your sample in the cradle. Ensure that both shutters are closed on the laser box.
2. Power on the spectrometer and launch Fluoracle.
3. When the spectrometer has fully initialised:
   a. Select the appropriate light paths on the signal rate window (Figure 22), i.e. TCSPC diode light path as the excitation source, and the XS1 as the emission path with the correct emission properties (grating, detector and light guide).
   b. Set the emission bandwidth to 5 nm. If the optical properties of the sample are known, set the wavelength of the emission arm to the peak of your sample.

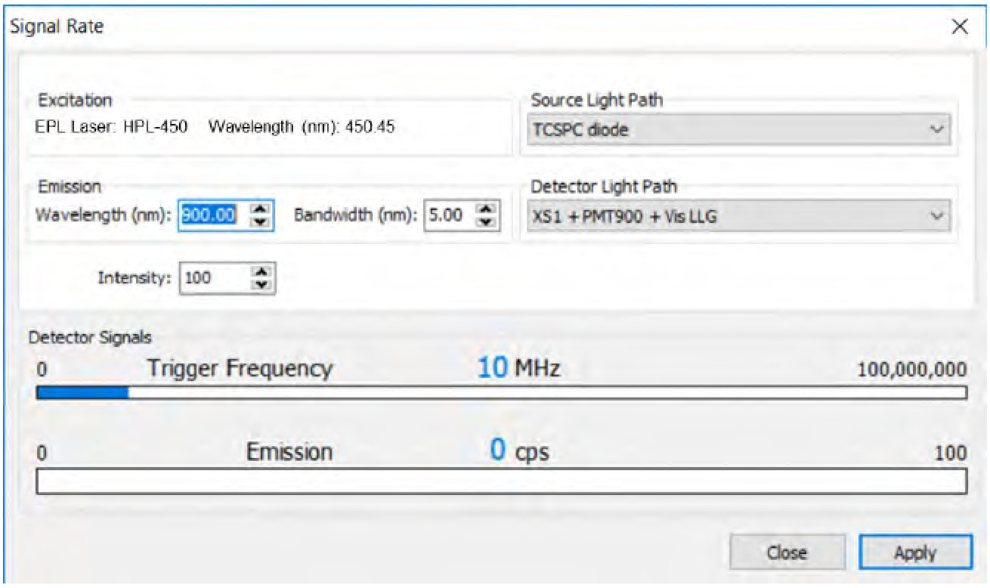


Figure 22: Signal rate screen, TCSPC measurements.

4. Launch the XMaster control software. The COM ports will have been set up during installation, so when ready to use select 'HV On'. The system will ramp up with a warning message displayed on the window, and the source on beacon will light up.
5. Turn the ND wheel on the laser mount for the HPL to the lowest setting, select the maximum rep rate on the unit (40 MHz) and turn the laser diode on high power mode by pressing and holding the power on button for 3 seconds before releasing. The emission LED will flash, indicating that the module is running on high power mode.

Note: If the laser source has been changed for your measurement, you will need to tune the path of the beam. See Procedure 6.1.4 for details on this process.

6. Open the laser shutter and gradually reduce the attenuation of the ND wheel until the reading under 'Current meter' in the XMaster software displays close to 10 µA. If the current exceeds 10 µA then the HV will turn off – this is a safety mechanism to prevent damage to the N5084.
7. Set the scan parameters for the TCSPC measurement as per the spectrometer manual and run the measurement. Upon scan completion review the settings used and adjust as required.
8. When the measurement(s) are completed, power off the laser/LED source first, close the shutter and then power off the N5084 (by Clicking the 'HV Off' button). Remove the sample and either fit a new sample before returning to step 3, or power off the spectrometer.

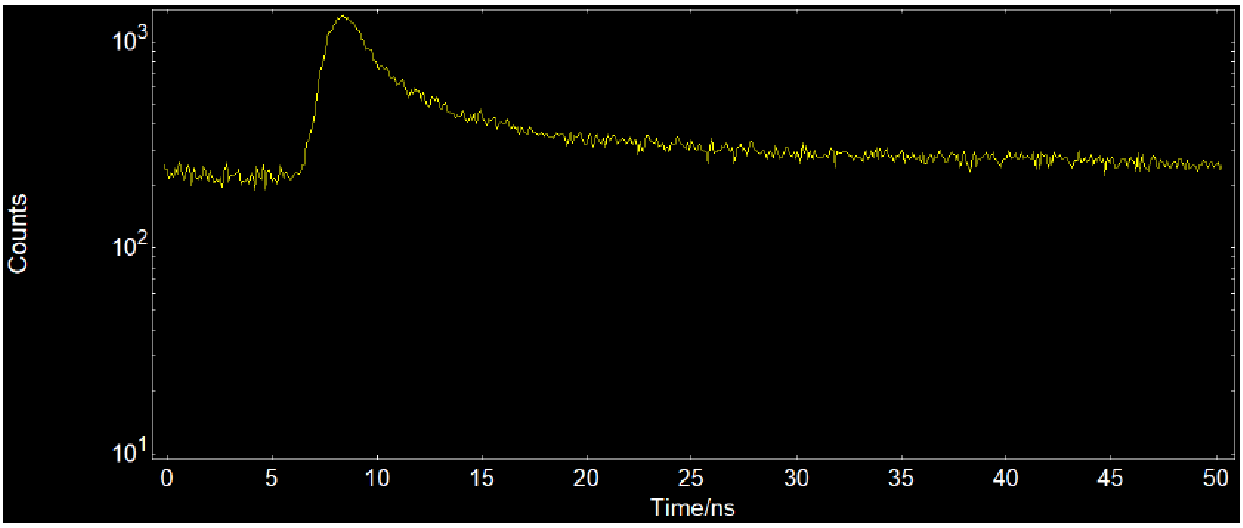


Figure 23: Example TCSPC measurement taken using the XS1 chamber with the N5084 as the source (pumped in this case by a HPL450 in high power mode).

5.5. TRES Measurements – N5084

To perform a Time Resolved Emission Scan (TRES) measurement using the N5084 source from a system-off state, follow the procedure outlined below:

1. Check the arrangement of the XS1 i.e. light guide installed, laser diode installed (HPL450, VPLED400 or VPL450) sample holder, source positioning. Set it up for your desired measurement and place your sample in the cradle. Ensure that the both shutters are closed on the laser box.
2. Power on the spectrometer and launch Fluoracle.
3. When the spectrometer has fully initialised:
    a. Select the appropriate light paths on the signal rate window, i.e. MCS diode light path for MCS TRES (Figure 20) or TCSPC diode for TCSPC TRES (Figure 22), and the XS1 as the emission path with the correct emission properties (grating, detector and light guide).
    b. Set the repetition rate for the source in Setup → EPL MCS Setup if appropriate.
4. Launch the XMaster control software. The COM ports will have been set up during installation, so when ready to use select 'HV On'. The system will ramp up with a warning message displayed on the window, and the source on beacon will light up.
5. Reduce the emission bandwidth to 0.5 nm (for MCS TRES) or 5 nm (TCSPC TRES). If the optical properties of the sample are known, set the wavelength of the emission arm to the peak of your sample and increase the bandwidth until you see an acceptable count rate. For unknown samples leave the bandwidth set to the starting value.
6. Set up the laser diode:
    a. Turn the ND wheel on the laser mount for the laser diode to the minimum value.
    b. If using a VPL450, select the desired pulse duration and turn on using the red power button on the back.

c. If using a HPL450 turn the dial to triggered mode and turn the laser diode on high power mode by pressing and holding the power on button for 3 seconds before releasing. The emission LED on the HPL450 will flash, indicating that the module is running on high power mode.

Note: If the laser source has been changed for your measurement, you will need to tune the path of the beam. See Procedure 6.1.4 for details on this process.

7. Open the laser shutter and gradually reduce the attenuation of the ND wheel until the reading under 'Current meter' in the XMaster software displays close to 10 μA. If the current exceeds 10 μA then the HV will turn off – this is a safety mechanism to prevent damage to the N5084.
8. Set the scan parameters for the TRES measurement as per the spectrometer manual and run the measurement. Upon scan completion review the settings used and adjust as required.
9. When the measurements are completed, power off the laser/LED source first, close the shutter and then power off the N5084 (by Clicking the 'HV Off' button). Remove the sample and either fit a new sample before returning to step 3, or power off the spectrometer.

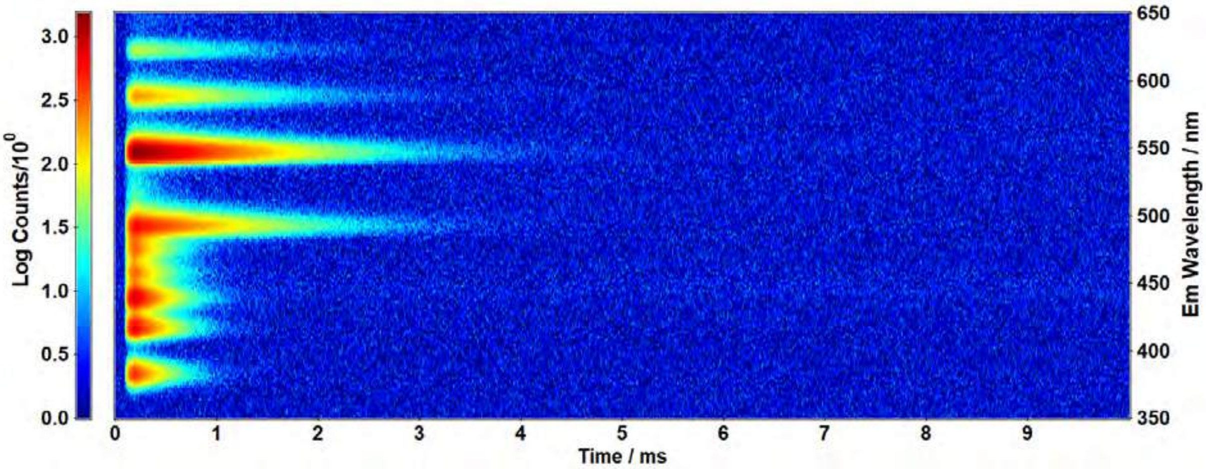


Figure 24: Example TRES map taken using the N5084 tube, driven by a VPLED400.

6. Operator and Troubleshooting Procedures

This section covers two categories of procedures: operator procedures, namely the steps one should go through when doing some sort of configuration change to the XS1 and troubleshooting procedures in case of some issue in trying to perform a measurement, for example.

6.1. Operator Procedures

6.1.1. Changing the engaged source

The process for switching between the pulsed and continuous sources is as below:

1. Ensure that all sources are powered off, and if the spectrometer is running that the emission detectors are protected by opening the sample chamber lid – this closes the shutter at the entrance to the monochromator.
2. Remove the six screws shown in Figure 25 (you will need a 3mm Allen key to do this).

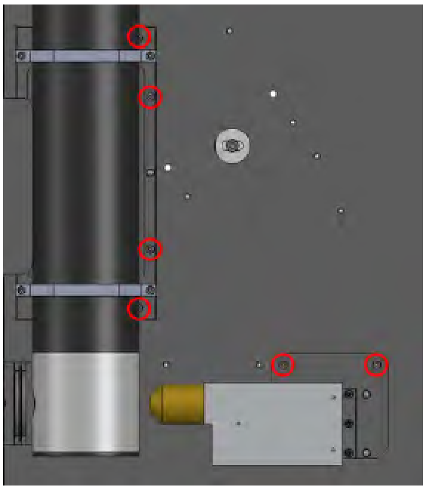


Figure 25: Location of all mounting screws for both x-ray sources.

3. Temporarily move the MagPro assembly to the bottom right corner of the XS1 (as indicated in Figure 26). This is required to allow the N5084 to move to the 'out of line' position.

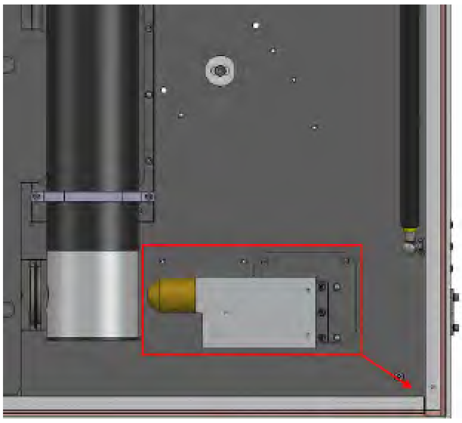


Figure 26: Direction to move MagPro to during source exchange.

4. Move the N5084 to its out of beam position, fitting the dowels into the holes. Secure using 4 x M4 screws.
5. Move the MagPro into the forward position, locating the dowels on the holder into their holes. Secure using 2 x M4 screws (Figure 27).

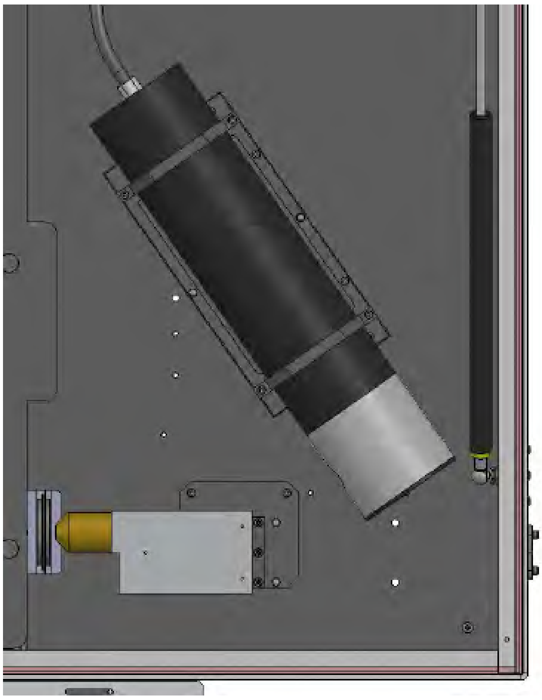


Figure 27: Sources swapped to have MagPro as excitation source.

The process can be run in reverse to exchange from MagPro to N5084.

6.1.2. Changing the Liquid Light Guide

In order to change LLG to one with a different transmissive range, one should follow the process below:

1. Ensure that all sources are powered off, and if the spectrometer is running that the emission detectors are protected by removing the sample chamber lid – this closes the shutter at the entrance to the monochromator.
2. Remove the inner lid and place safely to the side.
3. Remove the four screws on the service access lid corners (2.5 mm allen key) and take care to remove any loose bezels. Lift the lid, allowing the gas struts to hold it safely in place.
4. Remove the four thumbscrews and take off the inner lid (Figure 28):

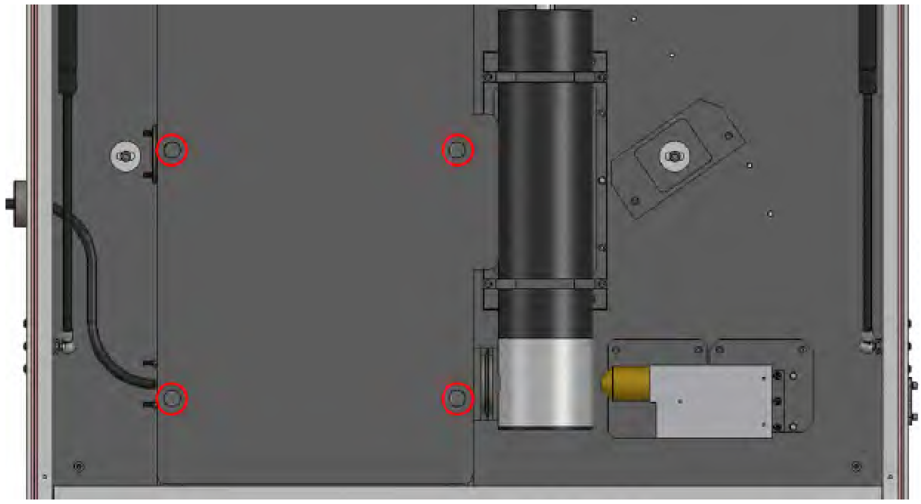


Figure 28: Location of thumbscrews holding inner chamber cover.

5. Remove the three pozi-drive countersunk screws on the feedthrough port and the grub screw retaining the light guide in the xy translator (1.5mm allen key) (Figure 29):

Figure 29: Positions of screws on the feedthrough (L) and retaining screw on the LLG mount (R).

6. Feed the light guide out through the inner chamber and out through the outer wall, removing the metal collar (above, with the three screws highlighted) and rubber grommet.
7. Loosen the screw on the LLG holder in the sample chamber (2mm allen key for FLS1000, 1.5mm for FS5, shown in Figure 30):

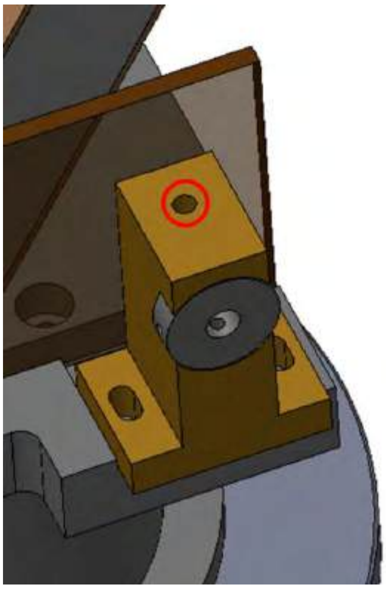


Figure 30: Positions of retaining screws on the FLS1000 LLG mount (L) and on the FS5 LLG mount (R).

8. Remove the LLG split clamp from the grommet on the sample chamber wall and remove the LLG end from inside the sample chamber. At this stage the old LLG has been fully extracted from the setup.

Note: if fitting the vis-NIR LLG, the yellow taped end of the fibre must be fitted at the XS1 end as this has a UV filter fitted to protect the core of the LLG.

9. Feed the new LLG through the sample chamber grommet, and secure in place using the screw highlighted above. Re-fit the split clamps around the LLG as it passes through the grommet.
10. Feed the other end of the light guide through the port plate (see image 1 between points 5 and 6). After this, run the LLG ferrule through the outer wall, then through into the inner chamber.
11. Secure the ferrule in place using the grub screw (image 2 between points 5 and 6).
12. Fit the rubber grommet around the LLG at the feedthrough point (out of the outer wall) and re-fit the plate using the 3 countersunk screws.
13. Refit the inner chamber lid, close the hinged service lid and screw down using the 4x M4 countersunk screws.

6.1.3. Fitting the fibre launch assembly (FLS1000)

In order to change between the fibre launch sample holder assembly and other accessories, one should follow the process below:

1. Remove the pan lid and laminated sliding hatch (highlighted below in Figure 31):

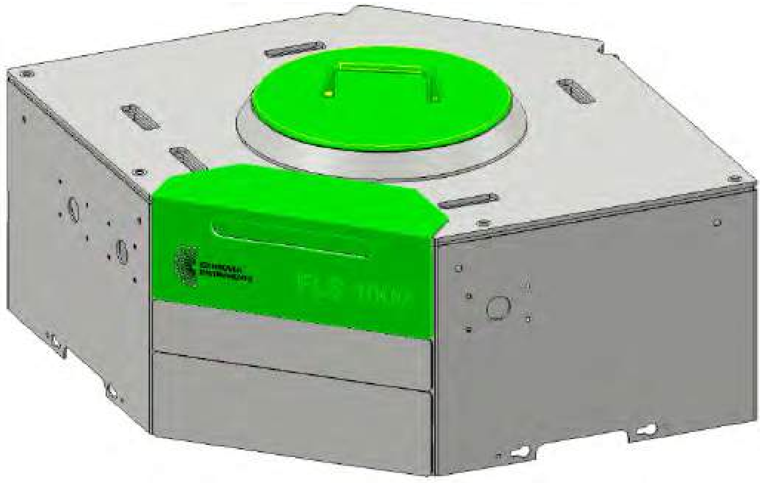


Figure 31: Pan lid and sliding hatch, FLS1000.

2. Remove the two nuts highlighted and the fascia panel (Figure 32):

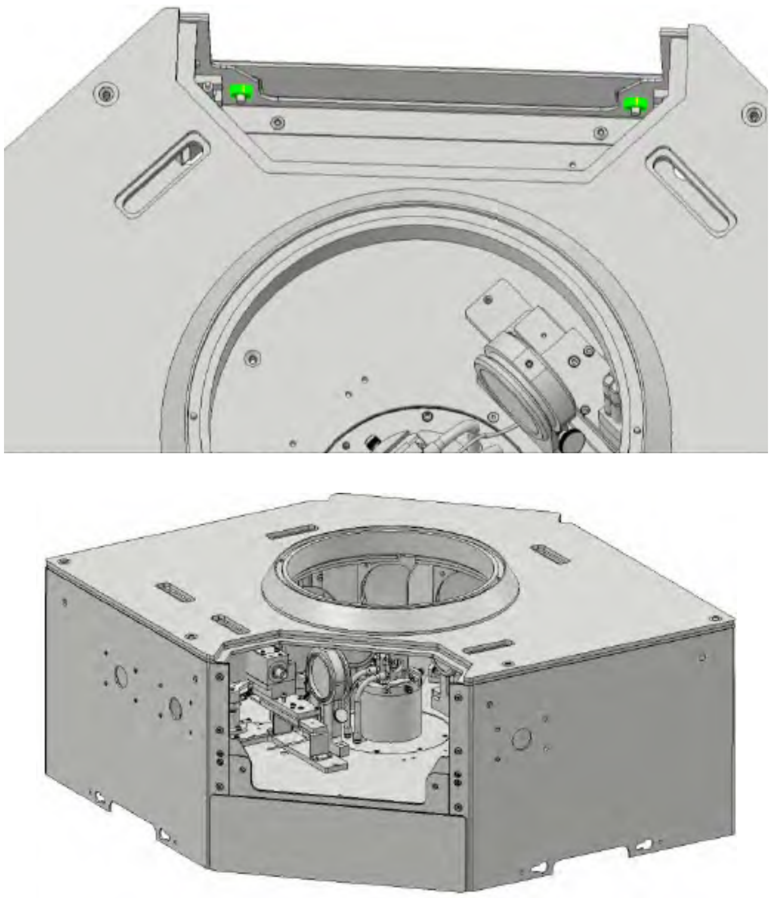


Figure 32: Fascia panel thumbscrews (top), and sample chamber after fascia removal (bottom).

3. Remove the sample holder from the chamber and fit the fibre launch accessory. Be sure to line the accessory up as illustrated in Figure 33:

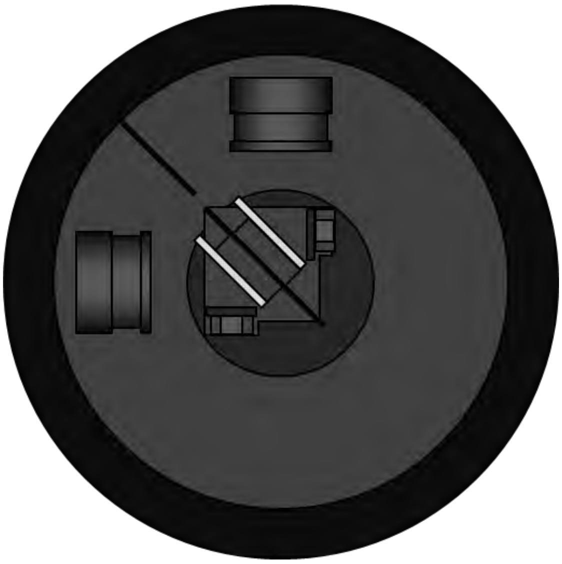


Figure 33: Top-down view of sample holder position, showing LLG launcher installed in correct geometry.

4. Fit the fibre launcher fascia panel and secure using the thumbscrew nuts. Replace the sliding hatch and pan lid.

6.1.4. Tuning the pump diode alignment

When the pump laser diode has been changed (between the HPL and VPL options), it is necessary to fine-tune the optical alignment of the beam to maximise the efficiency of photoelectron generation. The procedure below outlines the process to do this.

1. Check the arrangement of the XS1 i.e, that the required laser diode has been fitted (HPL450, VPLED400 or VPL450). Ensure that the both shutters are closed on the laser box.
2. Power on the spectrometer and launch Fluoracle.
3. When the spectrometer has fully initialised:
    a. Select the appropriate light paths on the signal rate window, i.e. MCS diode light path for MCS TRES or TCSPC diode for TCSPC TRES, and the XS1 as the emission path with the correct emission properties (grating, detector and light guide).
    b. Set the repetition rate for the source in Setup → EPL MCS Setup if appropriate.
4. Launch the XMaster control software and turn on the HV supply to the N5084 tube.
5. Open the laser shutter and gradually reduce the attenuation of the ND wheel until the reading under 'Current meter' in the XMaster software displays close to 4 μA.
6. First adjust the three thumbscrews on the laser mount (Figure 34) to maximise the current displayed on the Current Meter. You will have to cycle around all screws at least twice to ensure that the signal

level generated is maximised. If the signal gets above 8 μA, attenuate the beam using the ND wheel to reduce back to 4 μA.

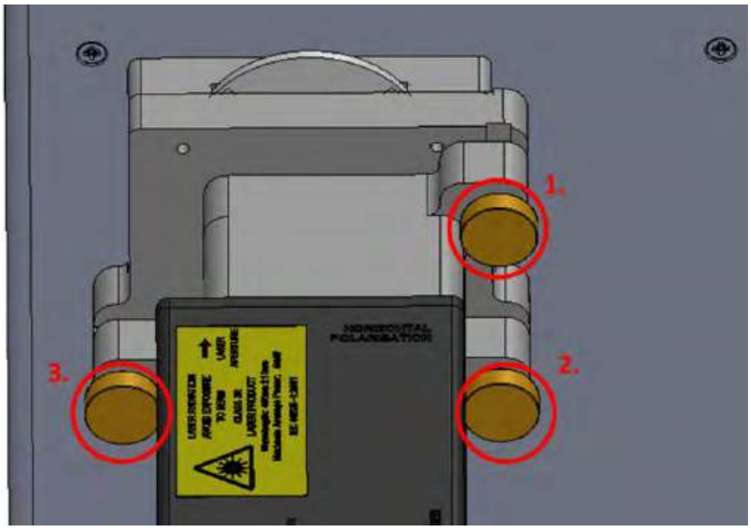


Figure 34: Thumbscrews on laser diode mount to tune beam alignment.

7. Using a 2mm allen key, fine tune the internal mirror through the laser steering box lid (Figure 35):

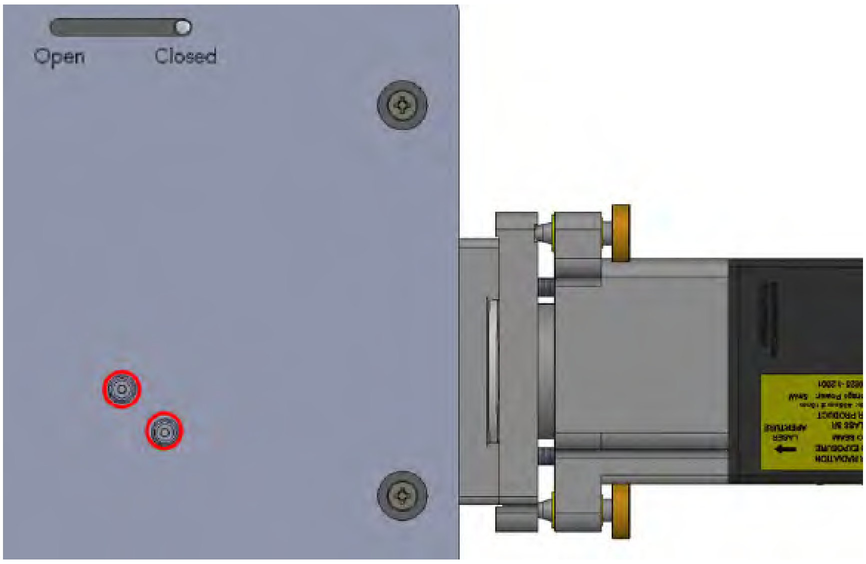


Figure 35: Location of mirror adjustment holes for final beam alignment.

8. Once the current on the Current meter has peaked, reduce the pump diode beam attenuation using the ND wheel to increase the current back to 10 μA ready for measurements.

6.2. Troubleshooting Procedures

This section outlines a series of steps the user should take for some issues which could arise when using an XS1.

6.2.1. No emission signal

1. First stage is to perform a CW measurement, either a quasi or full CW scan, depending on the XS1 options. If both options are available, always perform CW scans with the MagPro source.
2. Check that the correct source is forward in the excitation position in the XS1.
3. Check that the source is active, e.g. no interlock points are broken, that the shutter for the pump laser is open (for the N5084) etc.
4. Ensure that the sample chamber of the FLS is fully closed (pan lid, sliding hatch).
5. Check that the pump laser diode has been properly connected to the FLS1000.
6. Check your detector and light path choice in the FLS –detectors have a limited detection range, so be sure to match the LLG and detector to the arrangement you require.
7. Do a quick survey scan (across the full range of the LLG, with a step size equal to the emission bandwidth and minimum integration time (0.1s). This will allow you to ensure you're looking at the correct wavelength.
8. Use a reference scintillator sample to ensure that the system performs as it should (e.g. gadolinium oxysulphide)
9. If none of these steps work, contact Edinburgh Instruments Service Department

6.2.2. Low max current on N5084

The first stage is to check that the shutter is open and that the ND wheel is turned to let as much light as possible through. If this is the case, follow the steps below:

1. Reduce the transmission of the ND until < 2μA is displayed on the digital multimeter
2. Follow Procedure 6.1.4. to ensure that the pump beam is properly aligned.
3. Try a different laser diode (if the HPL is not working as a pump source, try the VPL and vice versa)
4. If the second diode works, then there is an issue with the first and it ought to be investigated. Check the output power under the conditions specified in the test record for the diode to see if it conforms. If not, arrange for a return of the faulty diode through EI service
5. If the second diode doesn't work, contact EI Service reporting a fault with the N5084 tube

Note: the beam pointing between different laser diodes is not always the same, so adjustments will be required when changing between VPL(ED) and HPL pump sources.

6.2.3. Low throughput from the XS1

Should the emission collection optics be compromised somehow (whether this be by user adjustment for some additional measurement or by accidentally moving one of the optical components) then the path can be re-optimised by the following procedure. Note that the chamber will have markings indicating a starting alignment position to reset the system to in the event of it being compromised.

1. Power on the spectrometer and launch Fluoracle (if it's not already running).
2. If the Current meter reading is low, follow Procedure 6.2.2. to eliminate poor excitation throughput as the cause.
3. Open the sample chamber lid to close the shutter to the emission monochromator – this is to protect the detector.
4. Ensure the x-ray sources are both powered off, remove the access lid and open the XS1 service lid (to do this, follow the Procedure 6.1.2. as far as Step 4).
5. Fit the laser diode source (HPL is preferable, though the VPL can be used if necessary) onto the diffuse reflectance laser port as illustrated below.
6. Sit a piece of white card upright in the sample holder cradle.
7. Ensuring the shutter is closed and ND turned to the minimum transmission position, remove the interlock cable from the laser diode and turn the source on (this will put the source into eye-safe alignment mode).
8. Tune the laser mount and steering mirror if required to ensure that the laser spot hits the centre of the sample holder position on the piece of card.
9. Do a visual check of the positions of the lens assemblies and xy translator holding the LLG. If they look to be collinear, then no loosening should be required. If not, loosen the hex nut (5mm allen key) on the magnetic base(s) of the optical assembly/assemblies out of line and move until they line up with the sample holder cradle. This is illustrated in Figure 36 below:

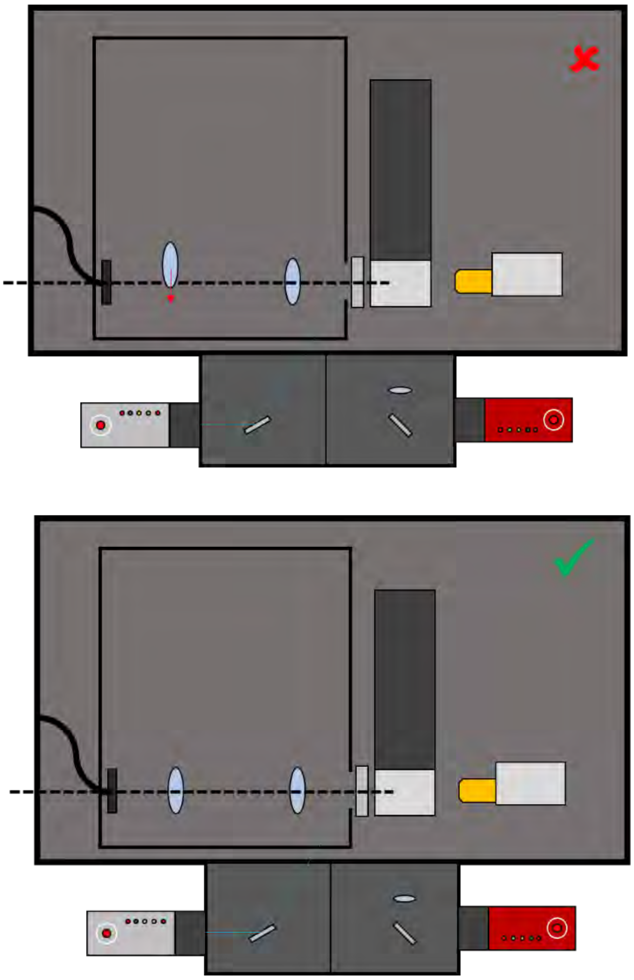


Figure 36: Illustration of the colliniear emission collection optics, showing incorrect (top) and correct (bottom) arrangements.

10. The first lens assembly (closest to the sample) should be positioned so that it is as close as possible to the sample. If this is not the case, make this adjustment.

The rest of the alignment should be performed in a dark laboratory to see the light spot and to protect the detector.

11. Locate the image of the laser spot after the second lens assembly to determine what adjustment is needed.
12. Move the lens position (sliding the base along the chamber baseplate) until the image of the laser spot is in line with the entrance to the LLG. Secure all magnetic bases loosened at this stage with the 5mm hex key on the side.
13. Once the image is centred on the position of the LLG check the vertical alignment. If the alignment is off, loosen the post holder on the lens assembly closest to the LLG (5mm allen key required) and change the height to centre it on the entrance to the LLG.

14. In Fluoracle, set the bandwidth to 0.2 nm and the emission wavelength to that of the laser diode. Close the lid, making sure that the count rate is at a level to not damage the PMT (for this process you're looking for <300,000 cps). If the count rate is too low, increase the bandwidth carefully.
15. Using the x-y translators, maximise the signal on the detector. Take care to monitor the count rate and decrease the bandwidth if it gets too high.
16. Once complete, open the spectrometer sample chamber lid. At this point the lights can be turned back on.
17. Re-fit the inner lid, close the service lid and set up your measurement with the sample as detailed previously.

6.2.4. Poor alignment of LLG output into monochromator (FLS1000)

If the output from the LLG is not properly aligned, then you will see a lower than expected signal level on your detector. If the output signal from the XS1 is still extremely low and you have been through Procedures 6.2.2. and 6.2.3., then you can check the optical alignment into the FLS1000 emission monchromator by following the steps outlined below:

1. Following on from Procedure 6.2.3., set the emission bandwidth to 0.1 nm, set the emission wavelength to the laser peak wavelength and close the sample chamber lid. The count rate should be of the order of a few hundred thousand counts.
2. Increase the laser attenuation by means of the ND wheel on the XS1 laser mount to reduce this count rate to approx. 10,000 cps.
3. Remove the lid and loosen the two set screws circled below in Figure 37 – these are the locking screws for the mirror mount. Note you need only loosen the screws for the emission steering mirror:

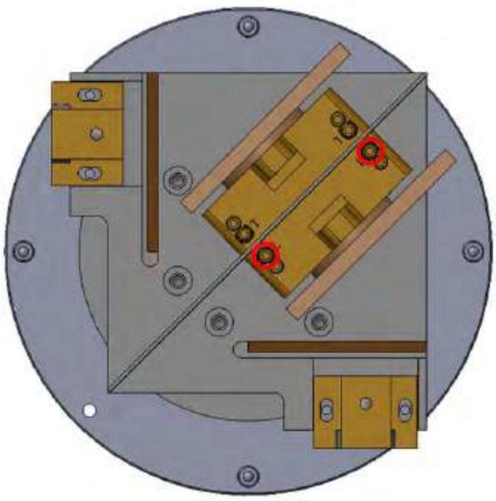


Figure 37: Location of locking setscrews on FLS1000 LLG launcher.

4. Starting with the screw circled in green in Figure 38, rotate the screw one full turn and replace the pan lid to check the effect on the signal rate. It is worth waiting for ten seconds to get an idea of the effect the change has made to the signal level:
    a. If the signal rate increases or stays the same, continue to adjust this screw in the same direction, replacing the pan lid after each adjustment to check the effect on the signal level
    b. If the signal rate decreases, rotate the screw in the opposite direction

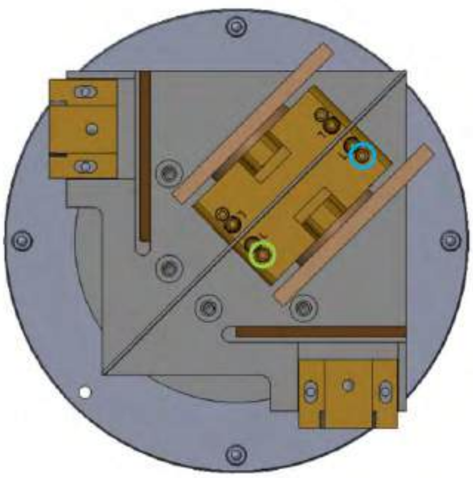


Figure 38: Location of adjustment setscrews on FLS1000 LLG launcher.

5. Once you have maximised the signal using the first screw, move the screw highlighted in blue and perform the same adjustments:
    a. If the signal rate increases or stays the same, continue to adjust this screw in the same direction as above
    b. If the signal rate decreases, rotate the screw in the opposite direction
6. Once the signal has been maximised with the second screw, repeat steps 4 and 5 to ensure that you have maximised the signal into the monochromator.
7. When the peak signal into the monochromator has been reached, tighten the locking set screws.

6.2.5. Poor alignment of LLG output into monochromator (FS5)

For users of an FS5 with an XS1, then the following procedure applies to align the light into the emission monochromator:

1. Following on from Procedure 6.2.3., set the emission bandwidth to 0.1 nm, set the emission wavelength to the laser peak wavelength and close the sample chamber lid. The count rate should be of the order of a few hundred thousand counts.
2. Increase the laser attenuation by means of the ND wheel on the XS1 laser mount to reduce this count rate to approx. 10,000 cps.

3. Open the SC-50R lid and starting with the thumbscrew circled in green in Figure 39 rotate the screw one full turn. Close the lid to check the effect on the signal rate. It is worth waiting for ten seconds to get an idea of the effect the change has made to the signal level:
    a. If the signal rate increases or stays the same, continue to adjust this screw in the same direction, replacing the pan lid after each adjustment to check the effect on the signal level
    b. If the signal rate decreases, rotate the screw in the opposite direction

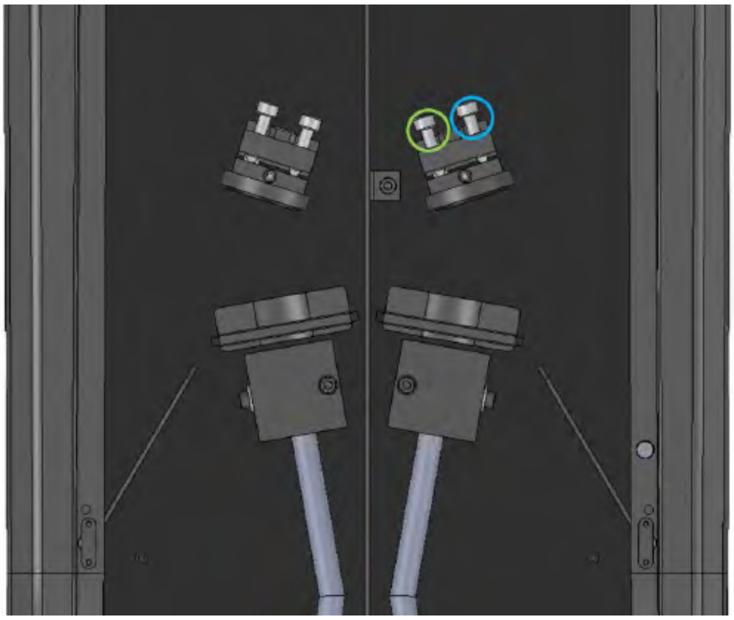


Figure 39: Location of adjustment setscrews on FS5 LLG launcher (SC-50R).

4. Once you have maximised the signal using the first thumbscrew, move the thumbscrew highlighted in blue and perform the same adjustments:
    a. If the signal rate increases or stays the same, continue to adjust this screw in the same direction as above
    b. If the signal rate decreases, rotate the screw in the opposite direction
5. Once the signal has been maximised with the second thumbscrew, repeat steps 4 and 5 to ensure that you have maximised the signal into the monochromator.

For users of the FLS1000 and FS5 who move between different fibre types on the same launcher, note that these two procedures may be required each time you change the fibre mounts.

6.2.6. Residual laser pump light in emission channel

Should you encounter emission light from the pump diode source (450 nm from laser sources, 400 nm from the VPLED), then first check if the laser baffle is fitted in the sample chamber (Figure 40):

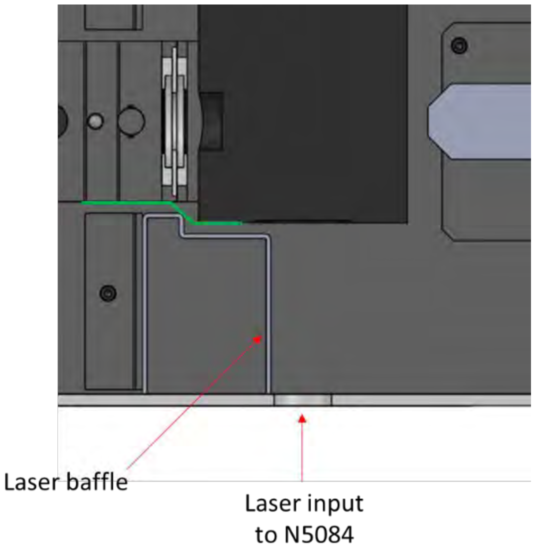


Figure 40: Position of laser baffle in XS1 chamber.

If the baffle is fitted correctly then the following steps can be tried:

1. Try fitting an excitation filter in the sample chamber in either the fibre launcher sample holder assembly or in the drop-in hanger before the emission monochromator.
2. If the laser line interferes with the x-ray fluorescence of the sample making filtering an unsuitable option, try fitting additional blocking material along the line illustrated in green (e.g. blackout card). Alternatively, contact EI Service for advice on additional optical shielding to isolate the pump light from the collection optics.

Appendices

Appendix I: Technical details about sources

Selected details about the sources used are listed below. If more details are required, please get in touch with EI Sales.

N5084 Tube

Parameter	Value	Unit
Target material	W	
HV Potential	40	kV
Recommended Beam Current	10	µA
Max Beam Current	50	µA
Min pulse duration	100*	ps
Radiant sensitivity (QE)	10	% (at 400nm)

MagPro Source

Parameter	Value	Unit
Target materials	W, Rh, Ag, Cr, Cu,	
HV Potential	4 - 60	kV
Beam Current	5 - 1000	µA
Max Power	12	W
Typical spot size	400	µm
Cooling	Forced air	
Operating temp	-10 - +65	°C

Appendix II: Tools required for general use and maintenance

Allen keys:

- 1mm
- 1.27mm
- 2mm
- 2.5mm
- 3mm
- 4mm
- 5mm

Spanner:

- 7mm open/ultra-compact adjustable

Screwdrivers:

- PZ0 pozi-drive
- PZ1 pozi-drive

Appendix III: Table of Figures

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