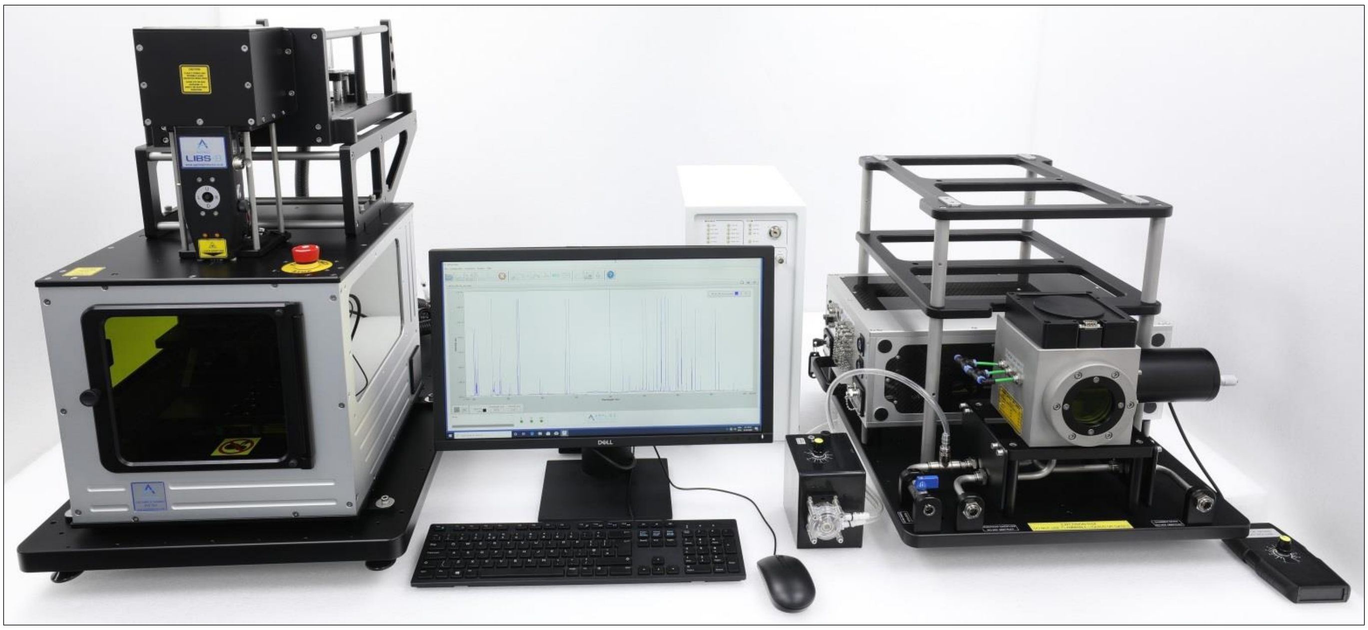
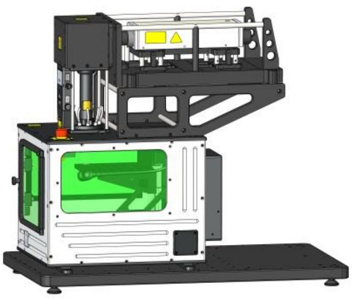
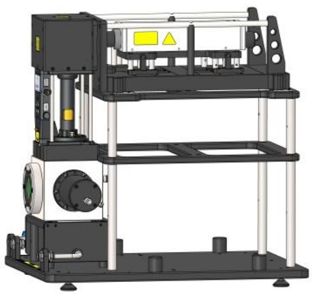
LIBS-6™ & LIBS-8™

Modular
Laser-Induced Breakdown Spectroscopy (LIBS)
System







User's Manual for Example Configuration 3

June 2022

APPLIED PHOTONICS

LASER SPECTROSCOPY SOLUTIONS

Applied Photonics Ltd
Unit 8 Carleton Business Park, Skipton, North Yorkshire BD23 2DE, United Kingdom
Tel +44 (0) 1756 708900 Fax +44 (0) 1756 708909 Web: www.appliedphotonics.co.uk

© 1998 - 2022 Applied Photonics Ltd

Contents

Appendices

© 1998 - 2022 Applied Photonics Ltd

1 Introduction

The modular Laser-Induced Breakdown Spectroscopy (LIBS) system described in this User's Manual is designed to be a highly versatile product for use by research scientists, application engineers and OEMs. Essentially a "LIBS system building block", the LIBS-6 and LIBS-8 modules may be used together with a variety of Q-switched Nd:YAG lasers and optical spectrometers to form a customised, modular LIBS system tailored to suit the requirements of a specific application or experimental research project. The LIBS modules remove the need for the research scientist or application engineer to design and construct their own laser focussing optics and plasma light collection optics by combining these features into a compact, integrated and easy to use device. An optional imaging kit may be added to the LIBS module, increasing further its functionality and versatility.

The modular LIBS system described in this User's Manual comprises the following main components: a LIBS-8 module fitted with an optional imaging camera (IMG-HR), an AP-Nano adaptor platform, a Litron Nano SG 150-10 laser, a SpectroModule-8 multi-channel LIBS spectrometer, an XYZ-750 modular sample chamber, an SC-LQ2 liquids sample chamber and a computer running LIBSoft software. In some sections of this User's Manual, the following components and devices are also described: LIBS-6 module, SpectroModule-6 six-channel LIBS spectrometer, Litron Nano LG 300-10 laser, an SC-2L modular sample chamber and a dual-camera imaging system (IMG-HR-2).

This User's Manual provides the necessary information needed to configure the LIBS equipment and how to operate the equipment safely. It is assumed that the user is familiar with the safety issues arising from the use of high-power lasers and will have preferably been trained in the safe use of high-power (Class 4) laser products.

2 Safety

2.1 Laser radiation

The modular LIBS system utilises a high-power Q-switched Nd:YAG laser (Class 4) and so it is imperative the equipment is operated only by suitably trained and experienced persons who are fully aware of the hazards inherent to this type of high-power laser equipment. It is imperative also that, prior to using the equipment, an appropriate risk assessment is conducted in such a way as to take account of the proposed use of the equipment, the environment in which the equipment is to be operated, and how its use may affect people who are not directly involved with the use of the equipment.

Example of a laser warning product label

The modular LIBS system is designed to meet the laser safety requirements of the relevant European standards (BS EN 60825) and USA standards (ANSI Z136.1). Although the modular LIBS system is supplied with a sample chamber which provides adequate containment of the laser radiation to Class 1 Accessible Emission Limits, it is possible for the user to operate the LIBS equipment without the sample chamber (i.e. operation in "open beam" mode as may be required for certain types of experiment). Accordingly, it is necessary to consider the modular LIBS system to be a **Class 4 Laser Product** and so, by definition, the equipment poses a risk of personal injury (eye, skin injury) <u>and</u> poses a fire risk. As with all Class 4 laser products, appropriate safety precautions must be taken as identified via a suitable risk assessment conducted by the user in consultation with a suitably qualified and experienced Laser Safety Officer.

The most significant hazard relating to exposure of personnel to the laser radiation is eye injury since direct or scattered laser radiation produced by the equipment can cause serious and permanent injury to the eyes including blindness - such injury may be instantaneous. Precautions must be taken to avoid exposure of personnel to hazardous levels of laser radiation. Such precautions may include the setting up of a temporary or permanent laser controlled area (e.g. a laser laboratory). Other measures may also be necessary, as determined by appropriate and thorough assessment of the risks (i.e. a risk assessment) conducted by the personnel responsible for the safe use of the laser equipment. Consult the User's Manual supplied with the laser for further guidance on the safe use of the laser.

The door of the sample chamber is equipped with a dual electrical interlock switch which is designed to prevent activation of the laser unless the door is fully closed. The door interlock switch is electrically connected to the "Interlock In/Out" port on the LIBS module via the 9-pin connector located directly above the laser aperture. The "Interlock In/Out" port connects with the SpectroModule-8 (using the supplied lead) which in turn is connected to the interlock circuits of the laser power supply. The laser safety interface between the SpectroModule-8 and the laser power supply depends on the make and model of laser being used. Throughout this User's Manual, it is assumed that a Litron NANO SG 150-10 laser and associated power supply is used, together with an XYZ-750 3-axis computer-controlled sample chamber.

Removal of the sample chamber from the LIBS-8 module, or disconnection of the Interlock In/Out lead, will also activate the interlock (i.e. prevent activation of the laser). The SpectroModule-8 multi-channel LIBS spectrometer is equipped with an Interlock Override facility (a keyswitch) which allows the interlock to be disabled so that the LIBS system may be used without a sample chamber (i.e. "open beam" mode). In view of this, the modular LIBS system **must be categorised as a Class 4 laser product** since, by design, the product may be used in such a way that the laser beam is not contained (i.e. "open beam" mode of operation)**.** If, however, the sample chamber is correctly fitted to the LIBS-8 module and the Interlock Override keyswitch is switched off and the key removed, then the laser radiation is adequately contained to Class 1 Accessible Emission Limits by the design of the hardware.

IMPORTANT

- READ and UNDERSTAND both this User's Manual and the instructions provided by the manufacturer of the laser before operating the LIBS equipment.
- ENSURE that an appropriate risk assessment has been conducted to establish whether or not the laser safety windows fitted to the modular sample chambers provide adequate protection against exposure to laser radiation **for the specific laser you intend to use with the LIBS equipment**. For advice on this matter, consult your Laser Safety Officer and/or Applied Photonics Ltd.

- ONLY suitably qualified and authorised persons should activate the Interlock Override keyswitch. The key should be removed from the keyswitch and held by the Laser Safety Officer when this feature is not required.
- ALWAYS use appropriate laser safety protective eyewear when operating the LIBS equipment in “open-beam” configuration – you should seek advice from your Laser Safety Officer on this matter.
- ALWAYS switch the laser off when not in use <u>and</u> remove the key from the keyswitch of the laser power supply to prevent unauthorised activation.
- ALWAYS thoroughly inspect the LIBS equipment for damage prior to use. Particular attention should be given to the laser safety windows and the laser safety interlock system, which should be tested each time the equipment is used.
- NEVER allow unauthorised and/or untrained persons operate the LIBS equipment.
- NEVER operate the LIBS equipment in areas where explosive gas mixtures may be present.
- NEVER operate the LIBS equipment with any access cover, panel, or protective window removed.
- NEVER place inside the sample chamber flammable liquids or any other material which may give rise to flammable / explosive gas mixtures. Activation of the laser under these conditions could result in an explosion leading to severe personal injury and/or fire hazard. Remember that the laser-induced plasma is a source of ignition.
- NEVER point the LIBS laser beam at a person (even with laser switched off), especially towards the eyes, even if the person is wearing laser safety eyewear. **The laser should be considered “active” unless the laser power supply is fully deactivated.**
- NOTE that the transparent acrylic nozzle aperture of the LIBS-6 or LIBS-8 module does not provide any protection to the user against exposure to direct or scattered laser radiation. The function of the nozzle aperture is described later in this User’s Manual.

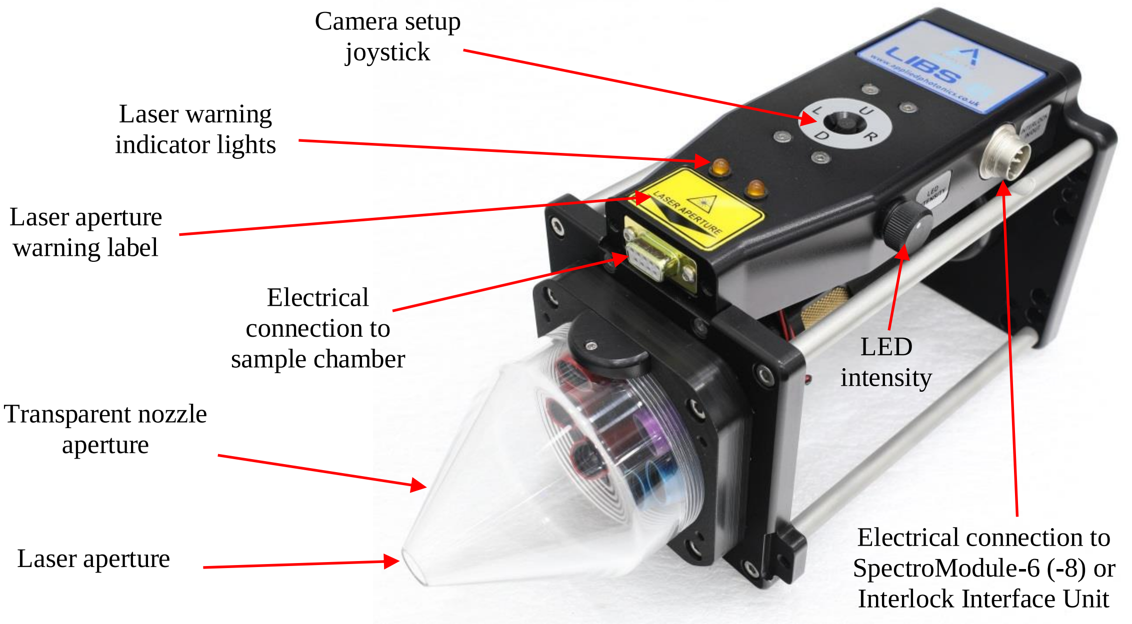


Image of LIBS-6 module showing location of laser warning indicator lights, laser aperture and warning label

2.2 Note on the laser safety window material used in the modular sample chambers

The laser safety window material fitted to the sample chamber is rated as follows:

Laser Wavelength (nm)	USA Standard ANSI Z 136.1 – 2014	UK & European Standard BS EN 207:2017
	Optical Density	Protection Level R (Q-switched laser)
1064	OD 6+	L6
355	OD 5+	L5
266	OD 5+	L5

R L6 at 1064 nm indicates a protection level of maximum spectral transmittance of 10^{-6} at 1064 nm for a pulsed laser of pulse length 10^{-9} – 10^{-7} seconds (i.e. a Q-switched laser).

The laser safety windows used in Applied Photonics Ltd's range of modular sample chambers provide adequate protection against scattered laser light of wavelength 1064 nm, 355 nm and 266 nm such that Class 1 Accessible Emission Limits are met if the sample chamber is used correctly and laser pulse energies are not excessively high (i.e. typically less than 300 mJ with a 5 - 10 ns pulse length). Note that the protective windows are NOT suitable for use with a 532 nm laser. Given that the LIBS-6 and LIBS-8 modules may be used with a variety of lasers, it is the responsibility of the user to conduct a risk assessment to establish whether or not the laser safety windows provide adequate protection for the particular laser being used. If in any doubt, you should consult your Laser Safety Officer and/or Applied Photonics Ltd for advice on this matter.

Warning – the Applied Photonics Ltd modular sample chambers are designed specifically for use with our LIBS-6 / LIBS-8 integrated LIBS modules and our LIBSCAN range of products and should not be used with any other laser device or product. If in doubt, seek advice from the manufacturer, Applied Photonics Ltd.

2.3 Electrical

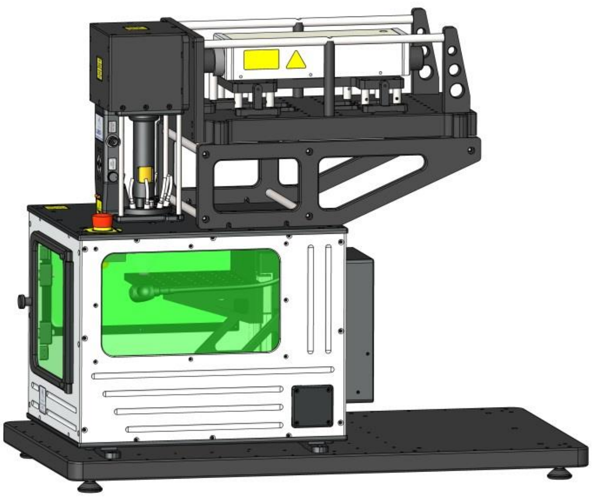
The LIBS-6 and LIBS-8 integrated LIBS modules, the SpectroModule-6 / SpectroModule-8 multi-channel LIBS spectrometer and the XYZ-750 modular sample chamber contain electrical circuits operating at 12 VDC at a maximum current of 5 Amps. Accordingly, they pose no electric shock risk. The laser head and associated power supply, however, contain electrical circuits operating at potentially lethal voltage and current levels. Consult the manufacturer's User Manual supplied with the laser for further guidance on the safe use and routine maintenance of the laser.

3 General description

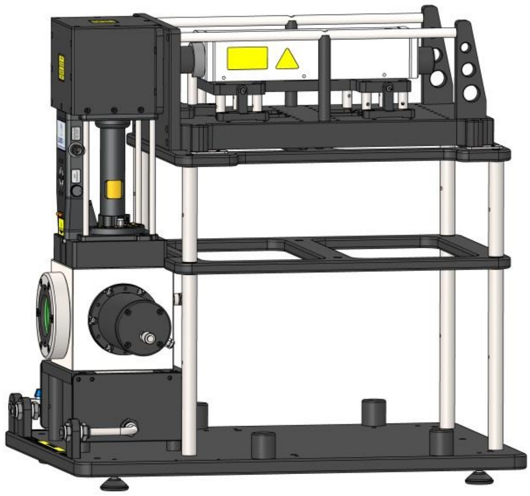
3.1 Overview

This configuration of our modular LIBS instrument consists of the following main components:

• LIBS-8 module (1064 nm)	Qty 1
• Right-angle mirror module (1064 nm)	Qty 1
• Adaptor Platform (AP-Nano)	Qty 1
• Litron Nano SG 150-10 laser (1064 nm)	Qty 1
• SpectroModule-8 multi-channel LIBS spectrometer	Qty 1
• XYZ-750 computer-controlled sample chamber	Qty 1
• SC-LQ2 liquids sample chamber	Qty 1
• Imaging kit IMG-HR (single camera)	Qty 1
• LIBSoft software	Qty 1



LIBS-8 module, AP-Nano Adaptor Platform with right-angle mirror module, Litron Nano SG 150-10 laser, XYZ-750 computer-controlled sample chamber

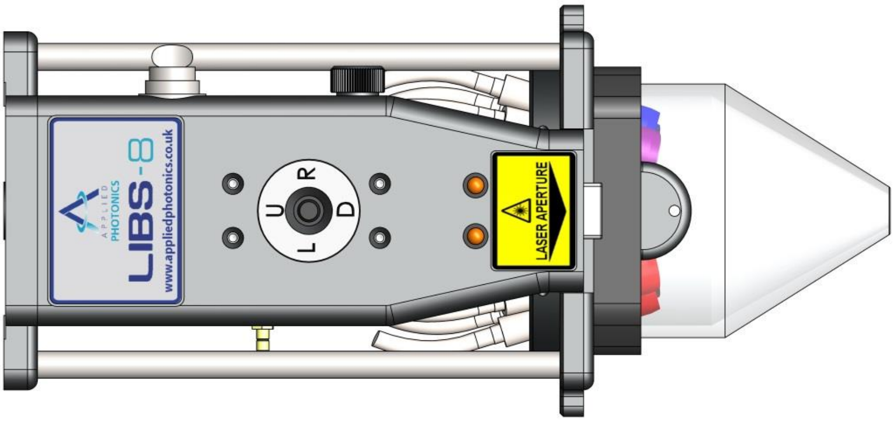


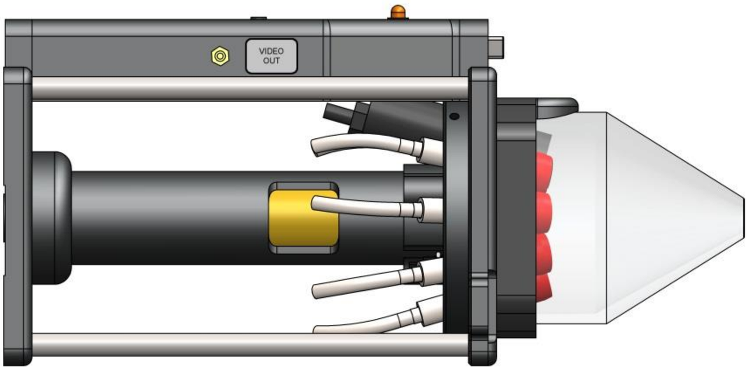
LIBS-8 module, AP-Nano Adaptor Platform with right-angle mirror module, Litron Nano SG 150-10 laser, SC-LQ2 liquids sample chamber

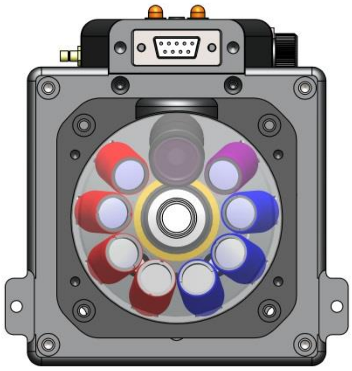
Litron LPU 350 laser power supply

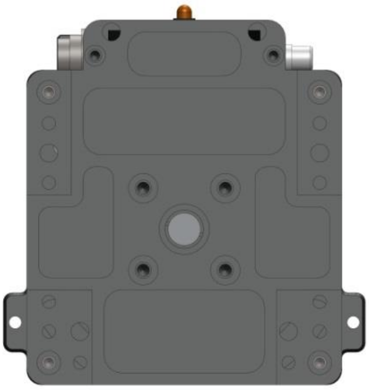
SpectroModule-8 multi-channel LIBS spectrometer

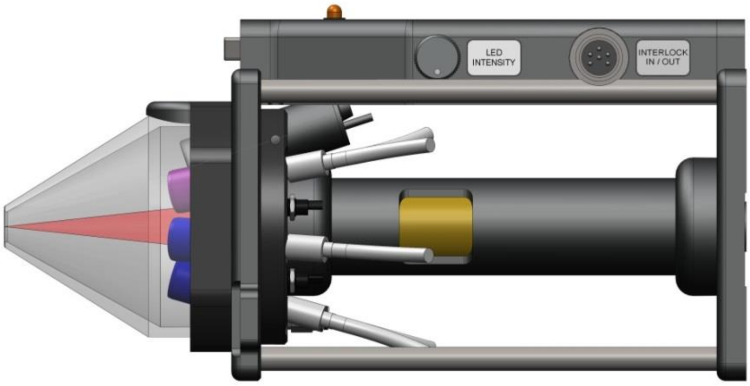
The main components of the LIBS-6 and LIBS-8 integrated LIBS module are illustrated in the following figures. The LIBS-8 module is essentially identical to the LIBS-6 module except that it has eight plasma light collection channels rather than six.

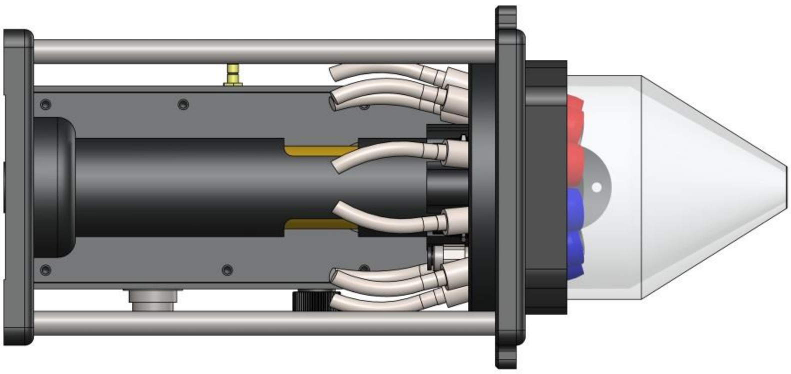


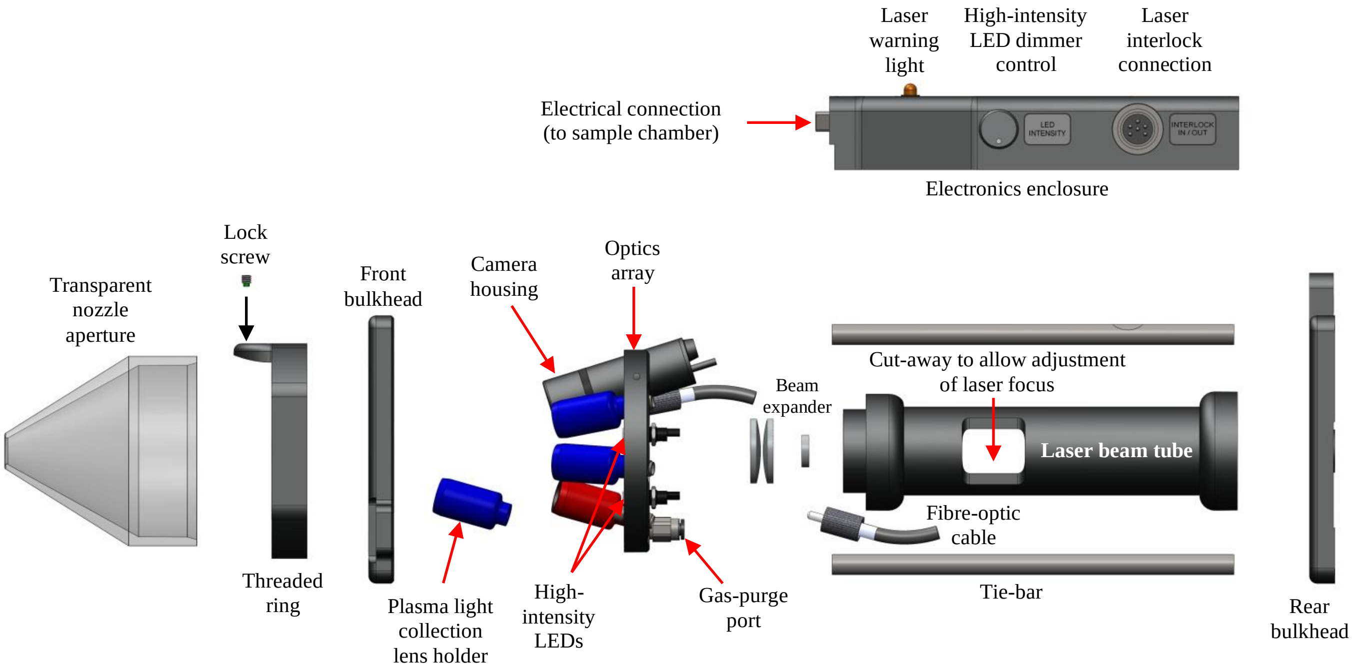






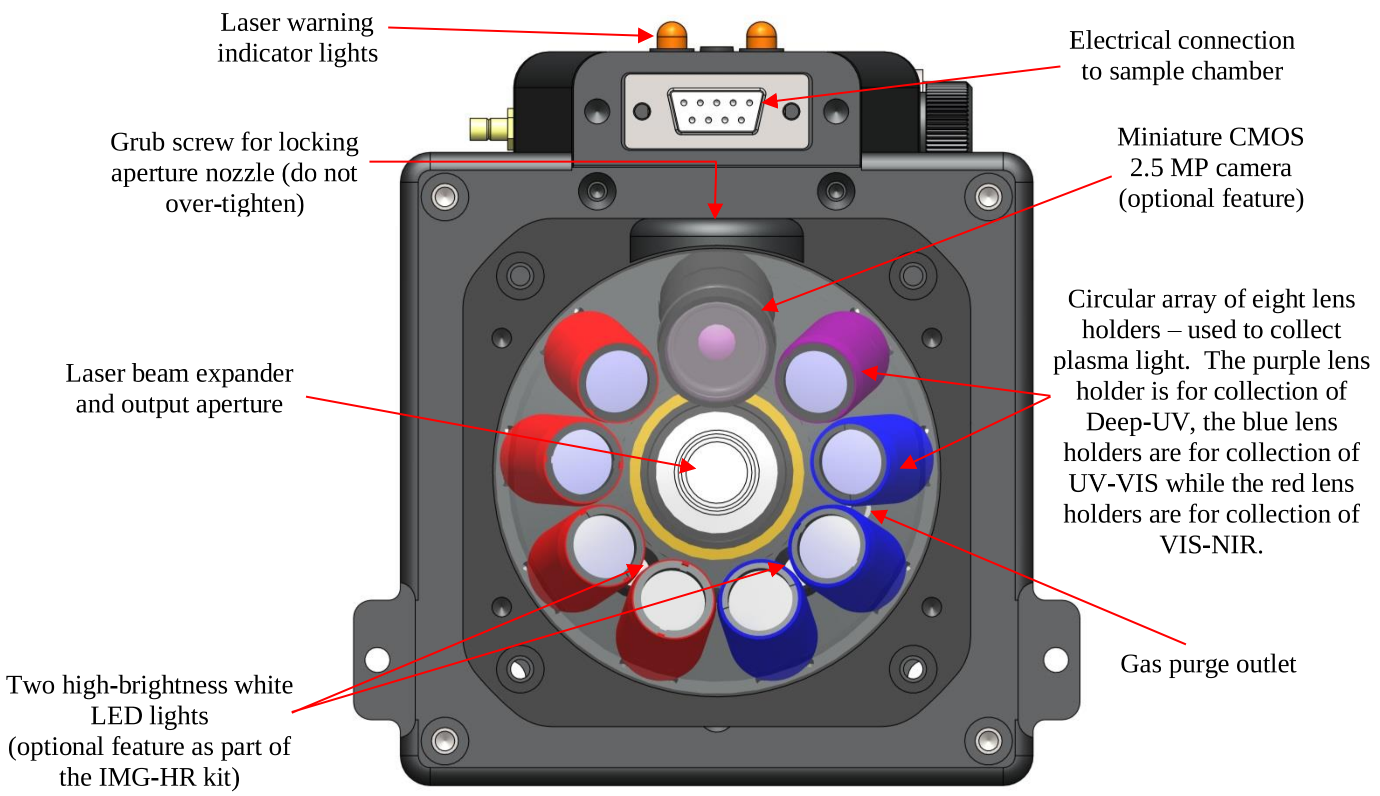




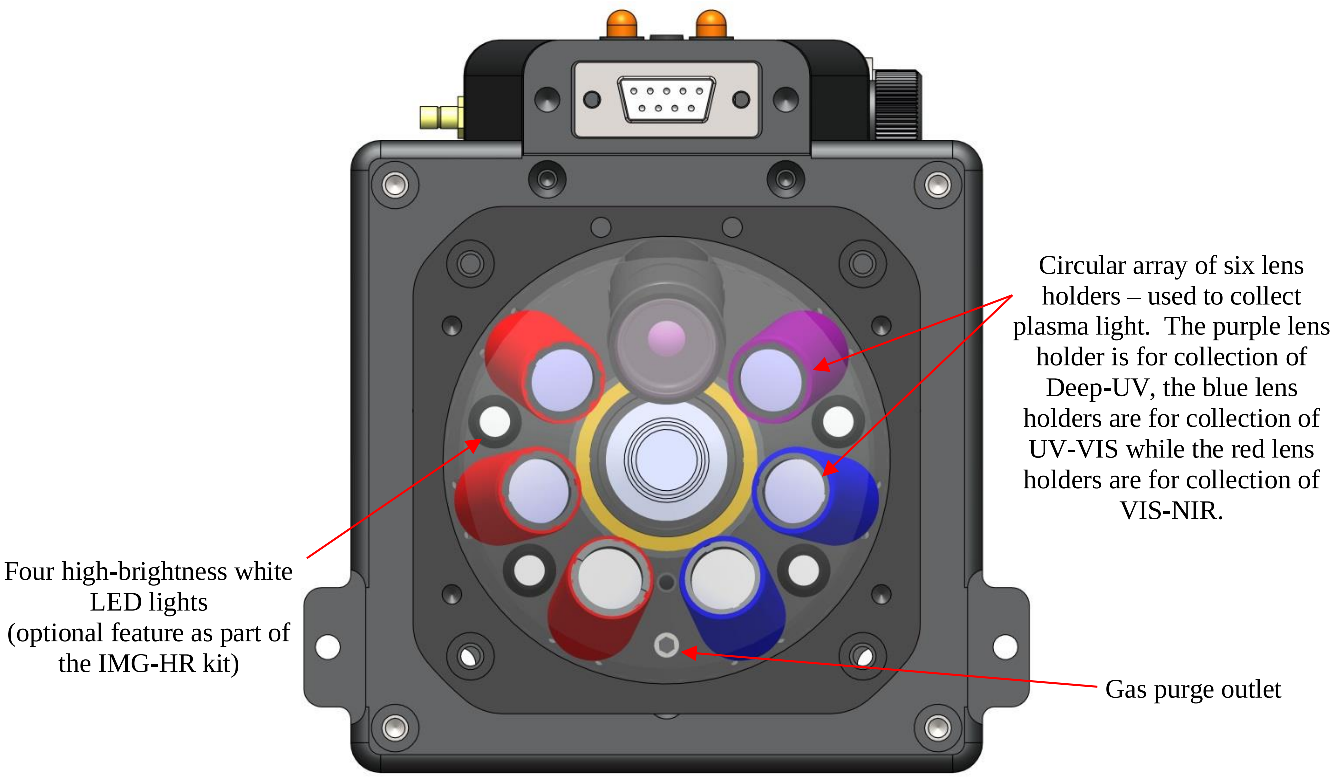


The LIBS modules include the following features:

- Plasma light collection lens array (six or eight channels) having a relatively large depth-of-field to allow efficient light collection even when the plasma position varies by approximately ±3 mm along the optic axis of laser beam.
- 3-lens laser beam expander with adjustable focus (adjustment of focal plane made by rotating the brass beam expander lens holder via the cut-way in the laser beam tube – adjustment range of approx. 14 mm).
- Adjustable nozzle aperture which provides a convenient means of setting the distance to the sample surface (adjustment range of approx. 14 mm). The nozzle aperture may be removed if this feature is not required.
- Gas-purge port (accepts 4 mm flexible nylon tube) which may be used to feed inert gas (e.g. argon, helium, nitrogen) to the sample surface. To ensure purge gas is directed to the sample, it is necessary to fit the nozzle aperture.
- Optional miniature CMOS colour video camera with array of dimmable high-brightness white LEDs for illuminating sample surface.
- Laser safety interlock with keyswitch operated override facility (via the SpectroModule-6 or SpectroModule-8 multi-channel LIBS spectrometer or an Interlock Interface Unit if an alternative spectrometer is used).
- Compatible with the Applied Photonics Ltd (APL) range of modular sample chambers.



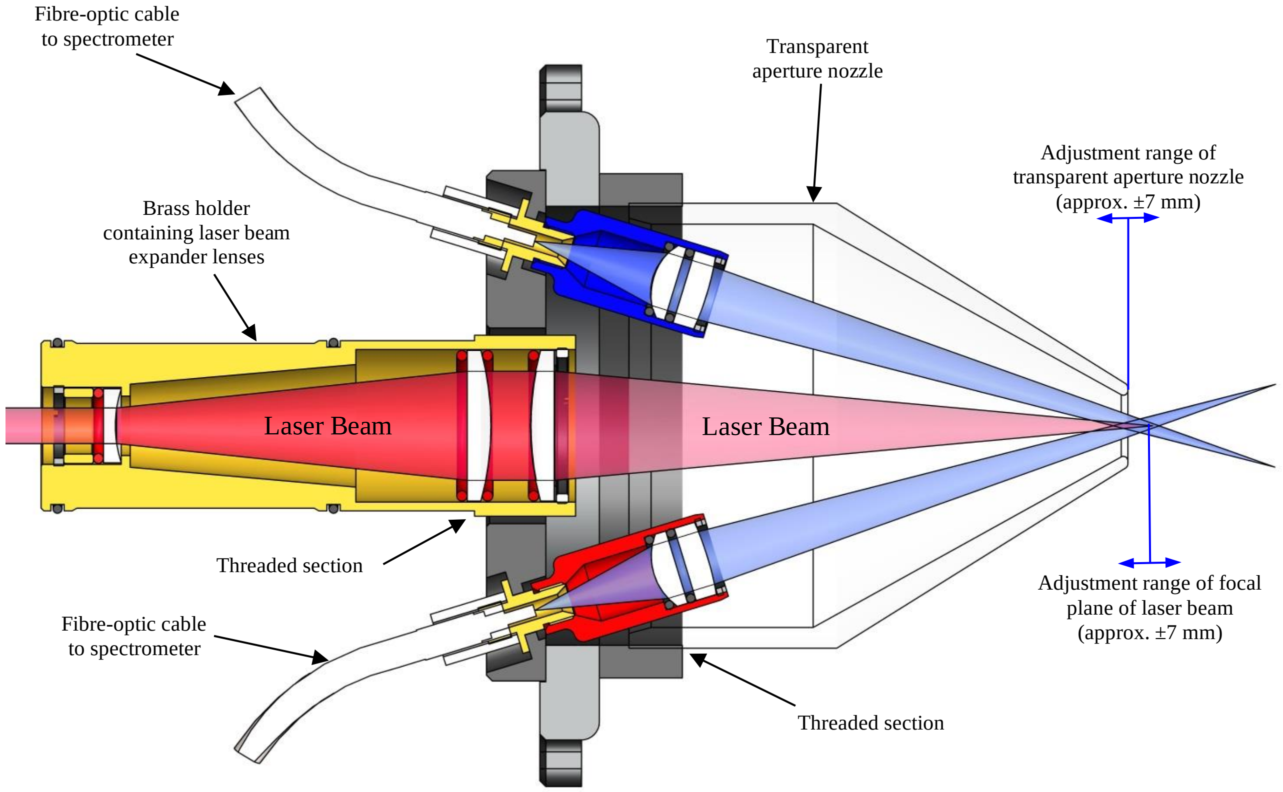
Front view of LIBS-8 module showing plasma light collection lens array, CMOS 2.5 MP camera, LED lights, laser aperture and gas purge outlet



Front view of LIBS-6 module showing plasma light collection lens array, CMOS 2.5 MP camera, LED lights, laser aperture and gas purge outlet

3.2 Laser beam expander and plasma light collection optics

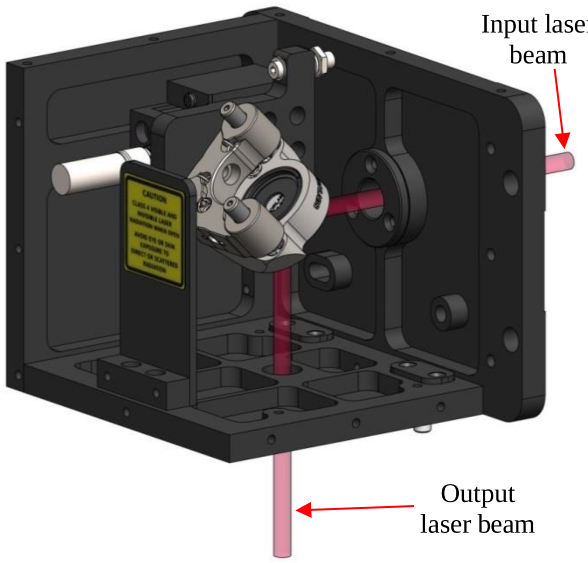
The optical configuration used in the LIBS-6 and LIBS-8 modules is illustrated schematically in the following diagram. The laser beam expander consists of three lenses and is used to provide a tightly focussed laser beam at nominally 100 mm from the aperture of the beam expander. The optical design is specific to the make and model of laser which the LIBS-6 or LIBS-8 module is to be used with – this is specified at the time of ordering of the LIBS module. The brass tubular piece which houses the beam expander lenses is threaded (1 mm pitch thread) so that rotating it causes the focal plane of the laser beam to move. The design allows for approximately ±7 mm of adjustment, as illustrated in the following diagrams. The plasma light collection optics are angled at approximately 17 degrees and are designed to collect light from the region in space defined in the following diagram. The transparent nozzle aperture is threaded so that rotation causes it to move along the optic axis of the laser beam – range of travel is approximately ±7 mm. The nozzle aperture may be removed from the body of the LIBS-6 or LIBS-8 module by unscrewing further or by removing the threaded black acrylic piece it screws into by unscrewing the four M4 cap head screws. The main purpose of the transparent nozzle aperture is to provide a convenient means of setting the distance to the sample surface. The nozzle aperture also provides containment of the purge gas, if used, to ensure the sample surface and laser-induced plasma region are effectively purged of atmospheric air. Physical protection for the laser beam optics and plasma light collection optics (and imaging camera if fitted) is also provided by the nozzle aperture.



3.3 Right-angle Mirror Module

The right-angle Mirror Module is fitted between the Adaptor Platform and the LIBS-6 / LIBS-8 module and is required if one of APL's "top entry" modular sample chambers (e.g. XYZ-750, SC-2L, SC-2XL etc) is used with the LIBS equipment.

Mirror Module (1064 nm)



Internal view of Mirror Module showing laser beam path

The Mirror Module provides precise alignment of the laser beam for the LIBS-6 / LIBS-8 module which is attached to the output side of the mirror module. Optical alignment is carried out at APL during final assembly of the LIBS equipment and so there should be no need for the user to adjust the internal mirror assembly. The Mirror Module, Adaptor Platform and LIBS-6 / LIBS-8 module each include dowel pin fittings so that precise optical alignment is maintained on removing and refitting these components. If you suspect that the optical alignment of the laser beam has been compromised, contact APL for advice before attempting to realign the optics.

3.4 Attaching the LIBS-6 / LIBS-8 module to a laser

3.4.1 Adaptor Platforms

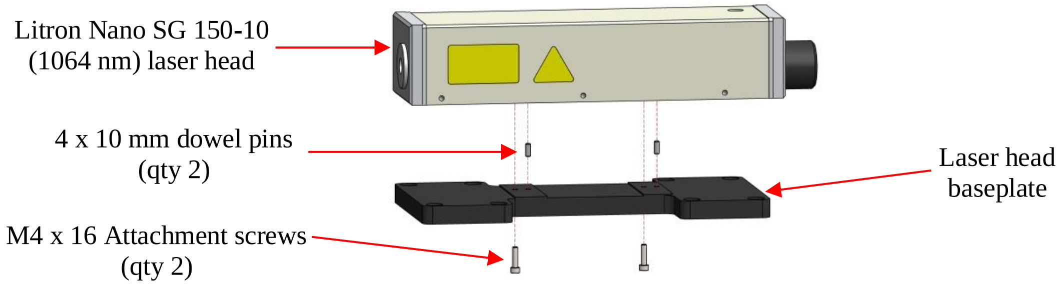
The LIBS-6 / LIBS-8 modules are suitable for use with virtually any commercially-available Q-switched Nd:YAG laser which has operating characteristics appropriate for LIBS. In order to attach either the LIBS-6 or LIBS-8 module to a laser head, it is necessary to use an appropriate adaptor platform. At the time of writing of this User's Manual, Applied Photonics Ltd supplies adaptor platforms for the following lasers:

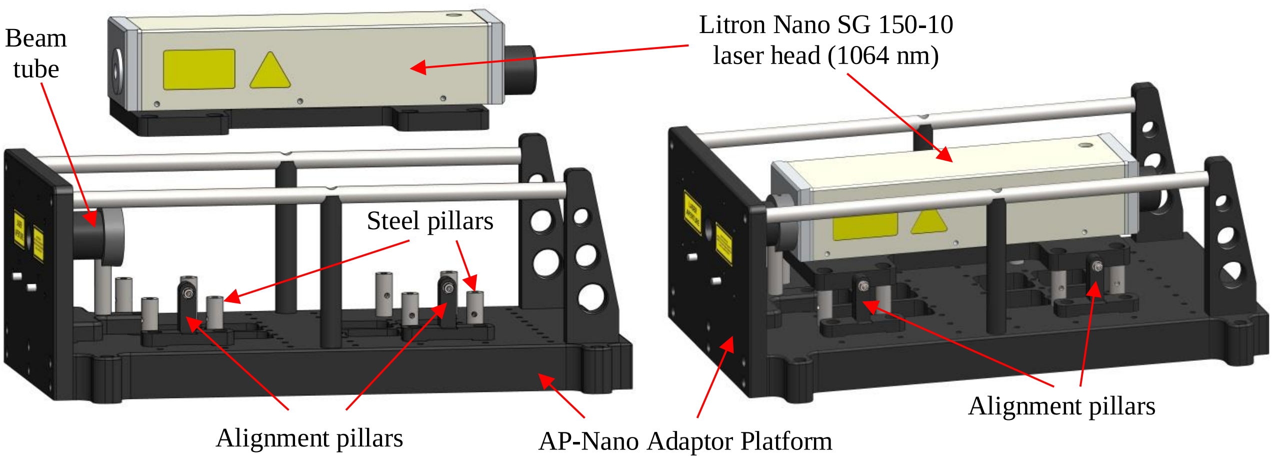
Laser Product	Adaptor Platform
Quantel Brilliant and Brilliant B	AP-Brilliant
Quantel (Big Sky) Ultra CFR range of lasers	AP-Ultra
Quantel (Big Sky) CFR 200 range of lasers	AP-CFR200
Quantel (Big Sky) CFR 400 range of lasers	AP-CFR400
Quantel QSmart 450 and 850	AP-QSmart
Innolas SpitLite	AP-SpitLite
Ekspla	AP-Ekspla
Litron Nano	AP-Nano
Litron Nanolite	AP-Nanolite
Litron Bernoulli (double-pulse, 1064 nm & 266 nm)	AP-Bernoulli

Should your laser not be listed above, contact Applied Photonics Ltd to enquire about availability of a suitable adaptor platform giving details of the make and model of your laser.

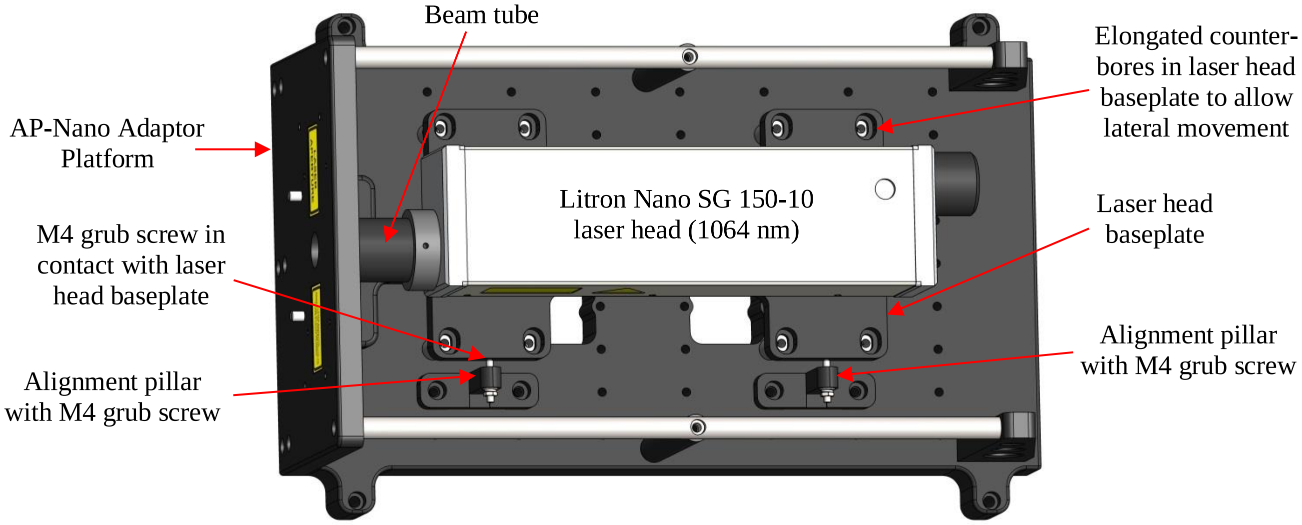
3.4.2 Litron Nano SG 150-10 (1064 nm) laser

The Litron Nano SG 150-10 laser head fits to the Adaptor Platform (AP-Nano) as shown in the following illustrations. The Litron NANO SG 150-10 laser head is attached to the eight steel pillars on the adaptor platform using eight M6x16 cap head screws. Two alignment pillars (fitted by Applied Photonics Ltd during final assembly) are used to provide a means for correctly aligning the laser head with respect to the adaptor platform. These alignment pillars should not be removed or adjusted by the user as they provide critical optical alignment of the laser beam for the laser head supplied with the equipment.

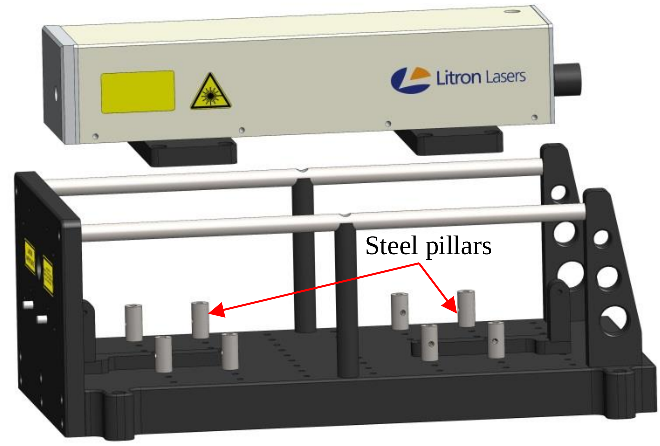




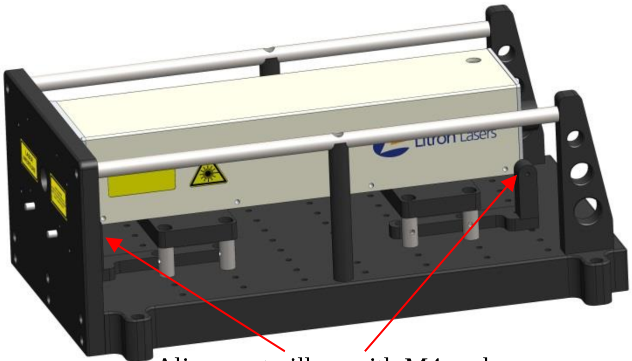
The laser head baseplate should be in contact with the M4 grub screws located in each alignment pillar to achieve correct optical alignment of the laser beam. This is achieved by sliding the laser head up against the two alignment pillars before fully tightening the six fixing screws used to attach the laser head baseplate (with attached laser head) to the adaptor platform.



3.4.3 Litron Nano LG 300-10 (1064 nm) laser

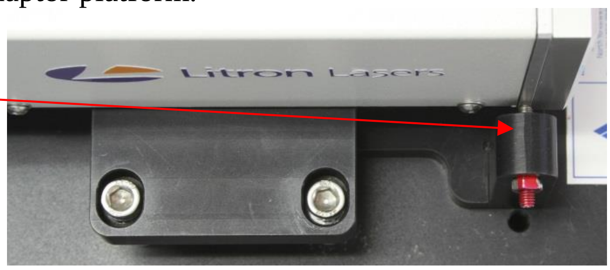


The Litron Nano LG 300-10 laser head is attached to the eight steel pillars on the adaptor platform using eight M6x16 cap head screws. Two alignment pillars (fitted by Applied Photonics Ltd during final assembly) are used to provide a means for correctly aligning the laser head with respect to the adaptor platform. These alignment pillars should not be removed or adjusted by the user as they provide critical optical alignment of the laser beam for the laser head supplied with the equipment.



The metal body of the laser head should be in contact with the M4 grub screws located in each alignment pillar to achieve correct optical alignment of the laser beam. This is achieved by sliding the laser head up against the two alignment pillars before fully tightening the eight M6 x 16 fixing screws used to attach the laser head to the adaptor platform.

Close-up view of the front alignment pillar



Close-up view of the rear alignment pillar

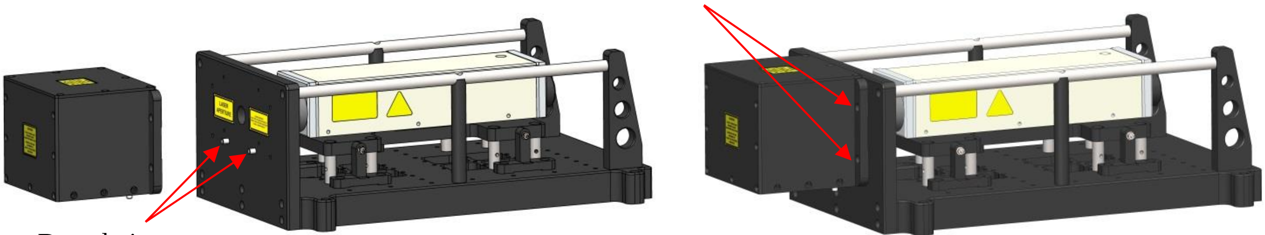
The laser head should not be removed from the adaptor platform unless absolutely necessary (e.g. should the laser head need to be returned to the manufacturer for repair etc).

Note: Do not remove or adjust the alignment pillars / M4 grub screws without first contacting Applied Photonics Ltd for advice.

3.4.4 Configuration options

The LIBS-6 / LIBS-8 modules may be attached to the Adaptor Platform in various ways, as shown in the following CAD illustrations.

M5 x 16 fixing screws
(qty 2 each side)

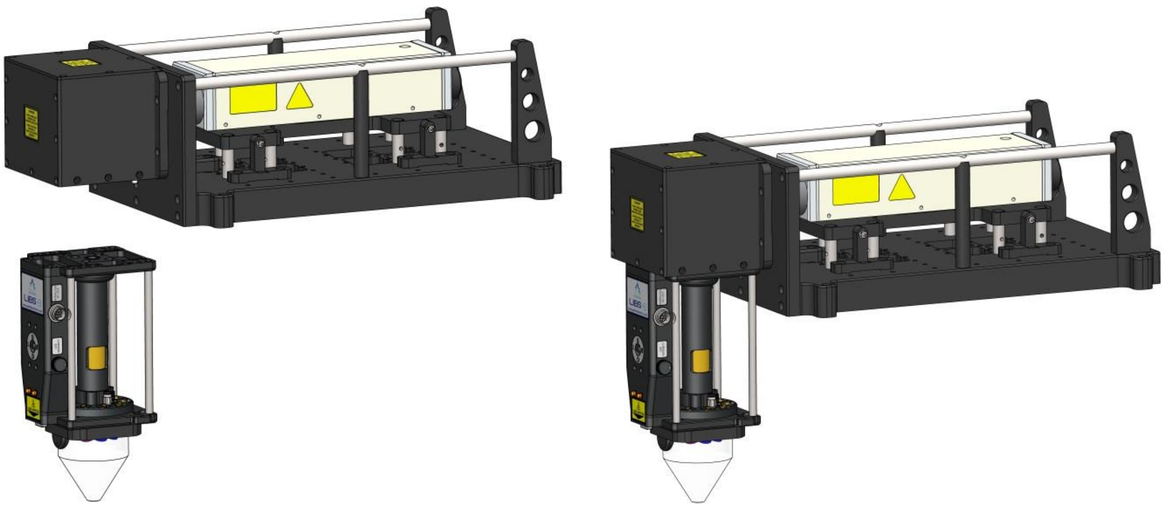


Dowel pins
(qty 2)

Mirror Module attachment to the Adaptor Platform

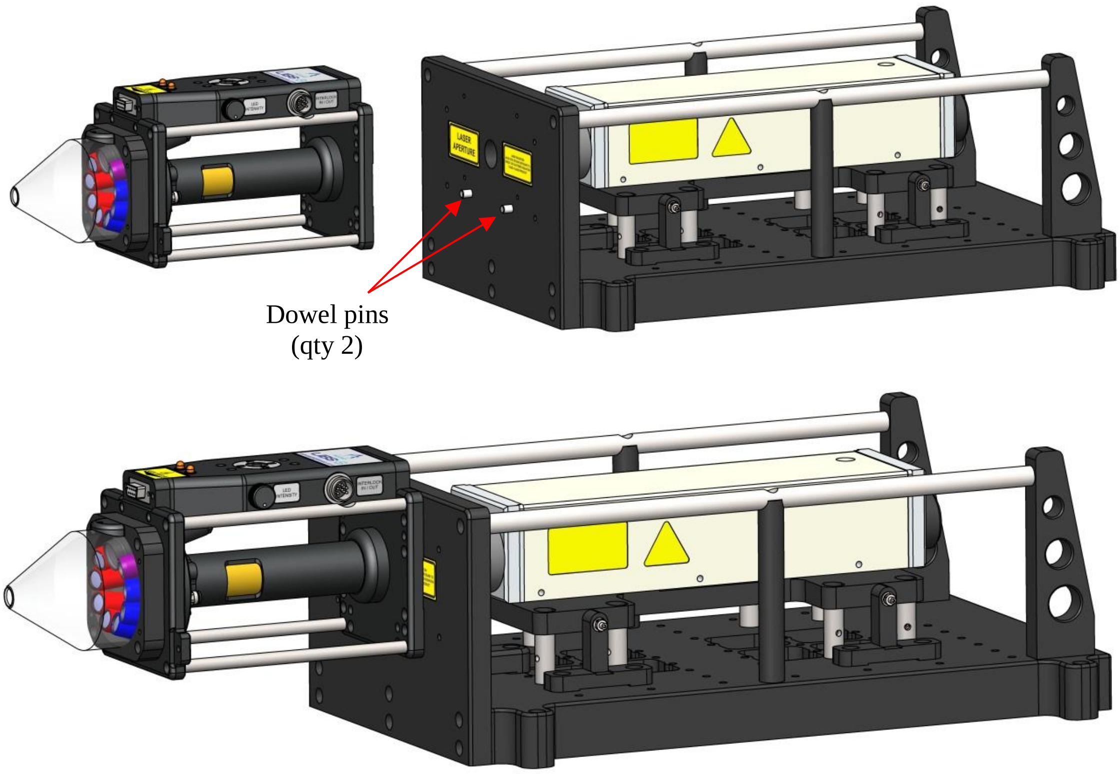
© 1998 - 2022 Applied Photonics Ltd

The following configuration is used with our “top entry” sample chambers such as our SC-2L and XYZ-750 products. This configuration may also be used in situations where a sample chamber is not required but the laser beam is required to interact with the sample vertically from above.



Mirror Module and LIBS-6 / LIBS-8 module attachment to the Adaptor Platform

The following configuration is used with our “side entry” sample chambers such as our SC-1 and SC-2 products. This configuration may also be used in situations where a sample chamber is not required but the laser beam is required to interact with the sample horizontally.



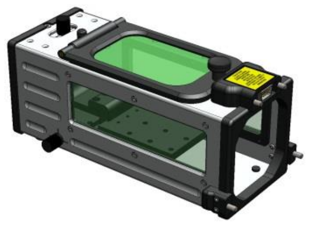
LIBS-6 / LIBS-8 module attachment to the Adaptor Platform without use of a Mirror Module

Dowel pins in the mating surfaces of the Adaptor Platform and modules are used to maintain correct optical alignment allowing the user to reconfigure the hardware without undertaking a full optical realignment of the laser beam optics.

3.5 Modular sample chambers

3.5.1 Overview

A range of modular sample chambers is available for use with the LIBS-6 / LIBS-8 modules (see table below). These sample chambers are also suitable for use with our LIBSCAN range of products. The XYZ-750 and SC-2L sample chambers are described in more detail in the remainder of this User's Manual.

			
SC-1	SC-2C	SC-2M	SC-2L
Single axis translation stage, 20 mm travel per stage (manual control). Approx. overall dimensions: 110 x 120 x 210 mm, approx. 2.0 kg	2-axis translation stage, 20 mm travel per stage (manual control). Approx. overall dimensions: 110 x 120 x 260 mm, approx. 2.5 kg	2-axis translation stage, 20 mm travel per stage (manual control) Fume extract port. Internal LED light. Approx. overall dimensions: 160 x 160 x 260 mm, approx. 4.0 kg	3-axis translation stage, 50 mm travel per stage (manual control). Fume extract port. Internal LED light. Approx. overall dimensions: 280 x 250 x 330 mm, 14 kg
			
SC-2XL	XYZ-750	XYZ-2500	SC-LQ2
3-axis translation stage, 50 mm travel per stage (manual control). Fume extract port. Internal LED light. Approx. overall dimensions: 330 x 360 x 430 mm, approx. 18 kg	3-axis translation stage, 75 mm travel per stage (computer-controlled). Fume extract port. Internal LED light. Approx. overall dimensions: 330 x 360 x 480 mm, approx. 21 kg	3-axis translation stage, X & Y travel = 250 mm, Z travel = 100 mm (computer-controlled). Fume extract port. Internal LED light. Approx. overall dimensions: 470 x 770 x 730 mm, approx. 56 kg	Modular sample chamber designed to analyse liquids. Further details can be found in APL Tech Note #6. Approx. 19 kg

Current range of modular sample chambers manufactured by Applied Photonics Ltd

WARNING

The above range of modular sample chambers is designed specifically for use with the LIBS-6 / LIBS-8 integrated LIBS modules or the LIBSCAN range of modular LIBS systems. The specifications for the laser safety windows are as follows:

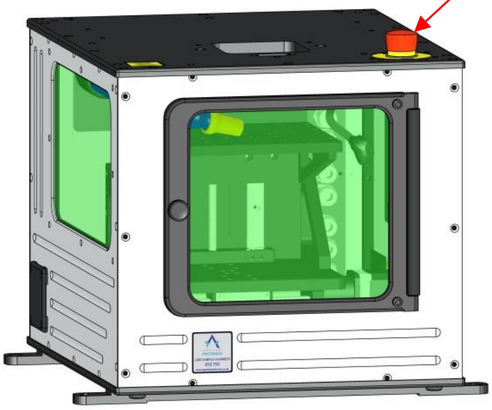
Laser Wavelength (nm)	USA Standard ANSI Z 136.1 – 2014	UK & European Standard BS EN 207:2017
	Optical Density	**Protection Level R (Q-switched laser)**
1064	OD 6+	L6
355	OD 5+	L5
266	OD 5+	L5

R L6 at 1064 nm indicates a protection level of maximum spectral transmittance of 10^{-6} at 1064 nm for a pulsed laser of pulse length 10^{-9} – 10^{-7} seconds (i.e. a Q-switched laser).

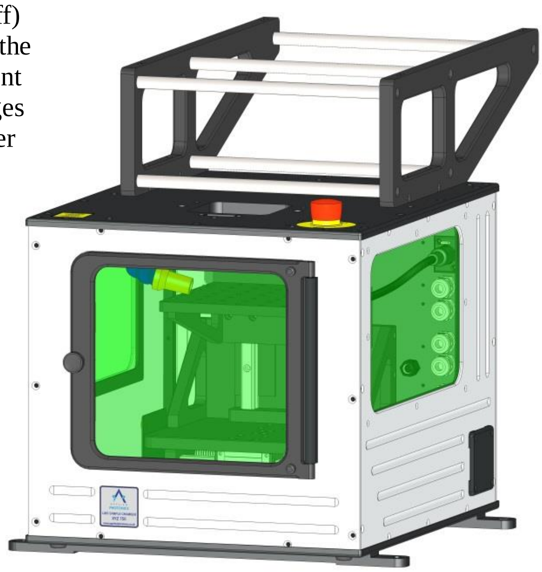
Since the LIBS-6 / LIBS-8 integrated LIBS modules may be used with various laser devices, it is the responsibility of the user to establish whether the protection offered by the laser safety windows is adequate for the laser being used. If in doubt, seek advice from a suitably qualified Laser Safety Officer or contact Applied Photonics Ltd before operating the laser equipment.

3.5.2 XYZ-750 computer-controlled sample chamber

The XYZ-750 computer-controlled sample chamber is one of APL's range of modular sample chambers designed for use with APL's modular LIBS systems. The XYZ-750 requires LIBSoft software to operate as it has no manual control of the three stages.



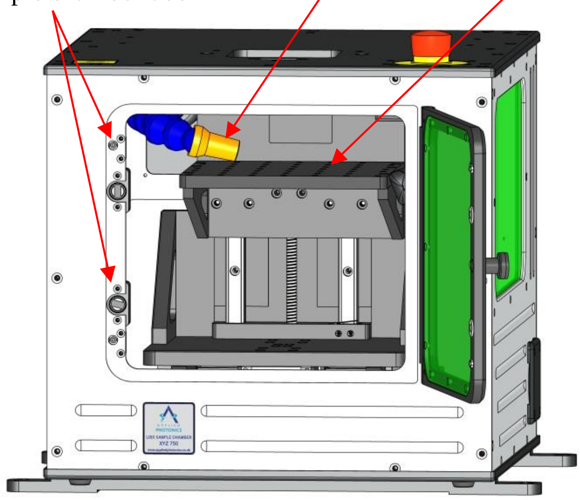
XYZ-750 computer-controlled, modular sample chamber



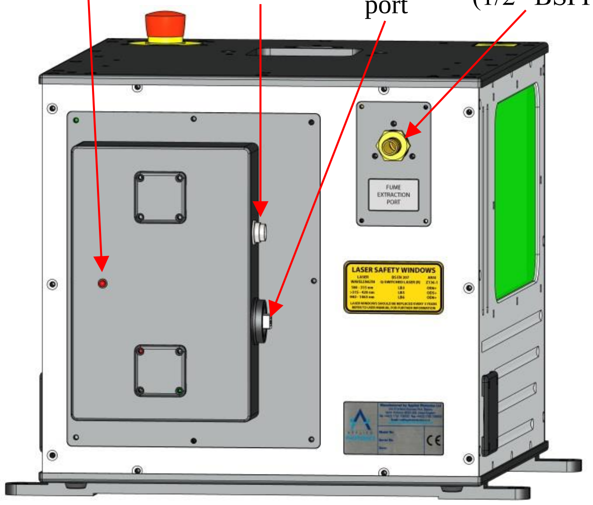
XYZ-750 with triangular support brackets fitted (for laser head Adaptor Platform)

The XYZ-750 has the following features:

- 3-axis motorised and computer-controlled translation stages each with 75 mm travel
- Precision ball-screw design which reduces rotational friction and virtually eliminates mechanical backlash
- Heavy-duty linear rail bearings (two rails per stage)
- Translation stage linear resolution of 1 µm
- Sample table breadboard with array of M6 tapped holes on 25 mm centres (5 kg load rating)
- Hinged door with magnetic catch and dual redundant electrical safety interlock
- Laser protective windows on two sides and hinged door
- Fume extraction port with internal flexible tube (external fume extraction system not included)
- Internal lighting
- External dimensions of approx. 330 x 360 x 430 mm. Weight approx. 21 kg



XYZ-750 front view

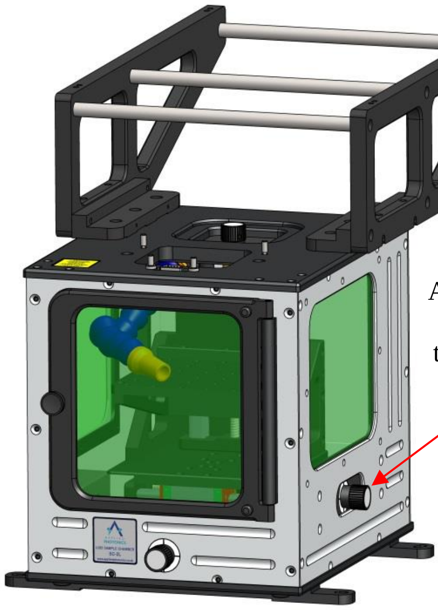


XYZ-750 rear view

3.5.3 SC-2L modular sample chamber

The SC-2L manually-operated sample chamber is one of APL's range of modular sample chambers designed for use with APL's modular LIBS systems.

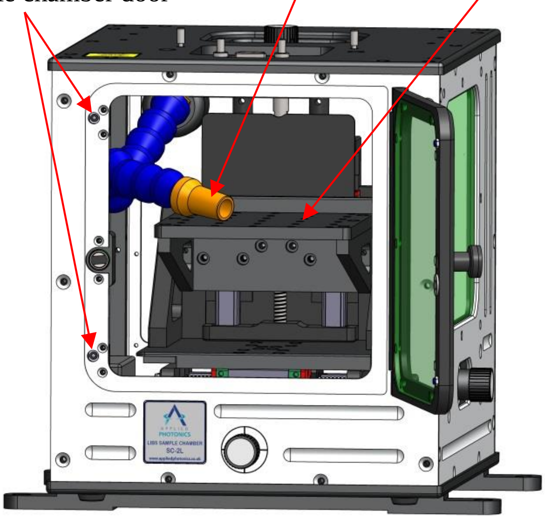
SC-2L modular sample chamber



SC-2L with triangular support brackets fitted (for laser head Adaptor Platform)

The SC-2L has the following features:

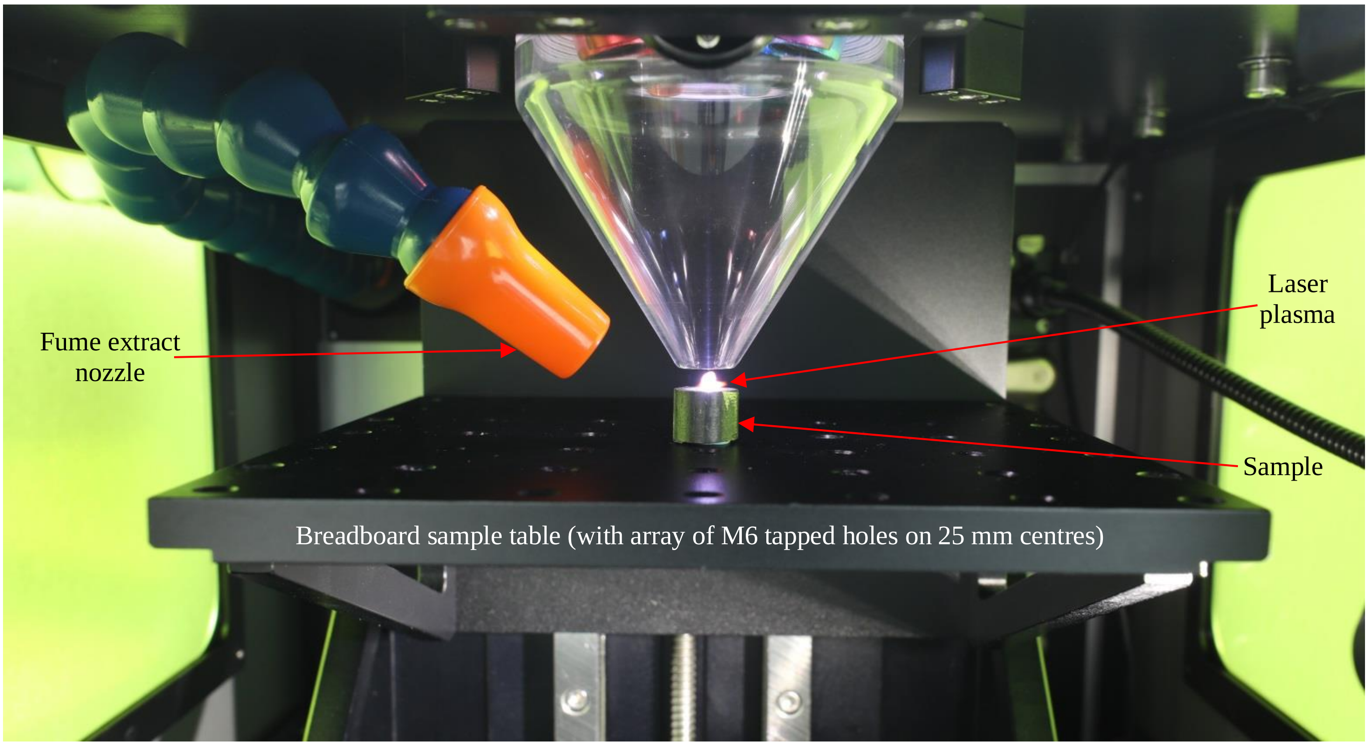
- 3-axis manually-operated translation stages each with 50 mm travel
- Precision ball-screw design which reduces rotational friction and virtually eliminates mechanical backlash
- Heavy-duty linear rail bearings (two rails per stage)
- Translation stage linear resolution of 2.5 mm per revolution (125 µm per division on adjuster knob)
- Sample table breadboard with array of M6 tapped holes on 25 mm centres (5 kg load rating)
- Hinged door with magnetic catch and dual redundant electrical safety interlock
- Laser protective windows on two sides and hinged door
- Fume extraction port with internal flexible tube (external fume extraction system not included)
- Internal lighting
- External dimensions of approx. 260 x 260 x 320 mm. Weight approx. 14 kg



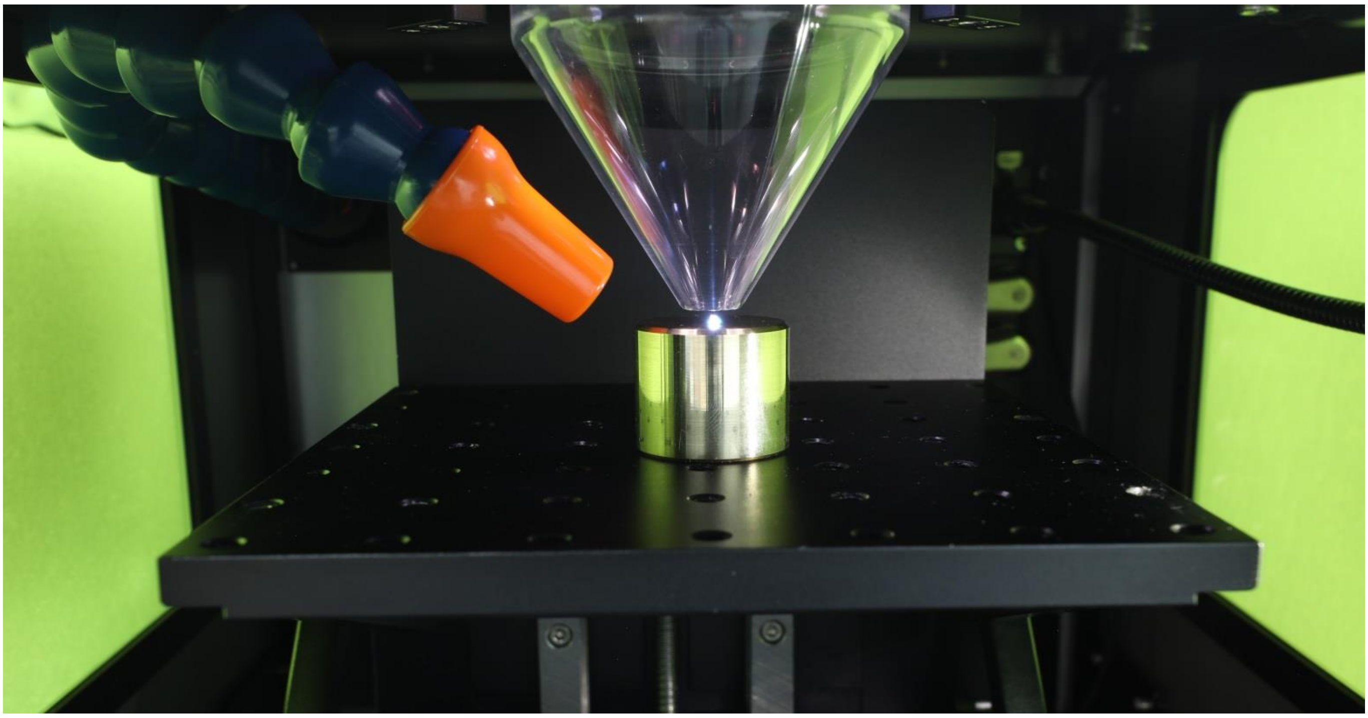
SC-2L front view

SC-2L rear view

© 1998 - 2022 Applied Photonics Ltd



Close-up view of inside of XYZ-750 sample chamber with LIBS-6 module fitted
and showing a laser-induced plasma on a metallic sample (high-purity gadolinium)



Close-up view of inside of XYZ-750 sample chamber with LIBS-6 module fitted
and showing a laser-induced plasma on a metallic sample (CZ121 brass)

3.6 SpectroModule-6 and SpectroModule-8 multi-channel LIBS spectrometers

The SpectroModule-6 and SpectroModule-8 multi-channel LIBS spectrometers are illustrated in the following diagrams.

SpectroModule-6 six-channel LIBS spectrometer

SpectroModule-8 eight-channel LIBS spectrometer

SpectroModule-6 is fitted with up to six optical spectrometers whereas SpectroModule-8 is fitted with up to eight optical spectrometers. The spectrometer specifications are as follows:

SpectroModule-6 **Six-channel LIBS spectrometer**	**SpectroModule-8** **Eight-channel LIBS spectrometer**
Spectrometer channel 1 (Spec-1) Approx. 190 – 261 nm, 3600 grating, FWHM = approx. 0.05 nm **Spectrometer channel 2 (Spec-2)** Approx. 260 – 320 nm, 3600 grating, FWHM = approx. 0.05 nm **Spectrometer channel 3 (Spec-3)** Approx. 319 – 421 nm, 2400 grating, FWHM = approx. 0.07 nm Order sorting filter: OSF-305 **Spectrometer channel 4 (Spec-4)** Approx. 420 – 503 nm, 2400 grating, FWHM = approx. 0.07 nm Order sorting filter: OSF-395 **Spectrometer channel 5 (Spec-5)** Approx. 502 – 721 nm, 1200 grating, FWHM = approx. 0.14 nm Order sorting filter: OSF-475 **Spectrometer channel 6 (Spec-6)** Approx. 720 – 910 nm, 1200 grating, FWHM = approx. 0.14 nm Order sorting filter: OSF-600	**Spectrometer channel 1 (Spec-1)** Approx. 182 – 254 nm, 3600 grating, FWHM = approx. 0.04 nm **Spectrometer channel 2 (Spec-2)** Approx. 252 – 312 nm, 3600 grating, FWHM = approx. 0.04 nm **Spectrometer channel 3 (Spec-3)** Approx. 311 - 361 nm, 3600 grating, FWHM = approx. 0.05 nm Order sorting filter: OSF-305 **Spectrometer channel 4 (Spec-4)** Approx. 360 – 454 nm, 2400 grating, FWHM = approx. 0.07 nm Order sorting filter: OSF-305 **Spectrometer channel 5 (Spec-5)** Approx. 453 – 530 nm, 1200 grating, FWHM = approx. 0.09 nm Order sorting filter: OSF-395 **Spectrometer channel 6 (Spec-6)** Approx. 529 – 648 nm, 1200 grating, FWHM = approx. 0.09 nm Order sorting filter: OSF-515 **Spectrometer channel 7 (Spec-7)** Approx. 647 – 853 nm, 1200 grating, FWHM = approx. 0.14 nm Order sorting filter: OSF-600 **Spectrometer channel 8 (Spec-8)** Approx. 852 – 1016 nm, 1200 grating, FWHM = approx. 0.14 nm. Order sorting filter: OSF-600

Spectrometer Specifications

The detector is a linear CMOS array (HAM-4096CL). Each spectrometer has a single linear CMOS array of 4096 pixels.

Spectrometers may be externally triggered (by Q-switch TTL pulse from laser power supply)

Trigger jitter is 21 ns (typical)

Integration time is 9 µs – 40 s

Minimum delay time (with respect to laser Q-switch pulse) is 0.9 microseconds

Slit width 10 microns

Spectrometer sensitivity (quoted by the manufacturer Avantes) is 218,000 counts/µW per ms integration time

Signal/Noise is 335:1

16 BIT, 6 MHz A/D converter

Stray light is 0.19% to 1.0% depending on grating

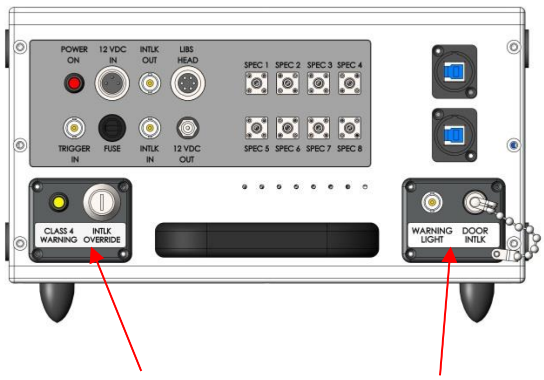
Each spectrometer channel is fibre-optically connected to the corresponding plasma light collection optic in the LIBS-6 or LIBS-8 module, as described later in section 3.7. In addition to the optical spectrometers, SpectroModule-6 and SpectroModule-8 are equipped with control electronics for the laser safety interlock system and so electrically interface with the modular sample chamber via the LIBS-6 / LIBS-8 module and also the laser power supply using the special cables supplied with the LIBS equipment. The electrical connections between the SpectroModule-6 / SpectroModule-8 and the other components of the LIBS system are illustrated in section 3.8 of this User's Manual

The laser safety interlock system is designed to inhibit laser beam emission unless the sample chamber door is closed. It will also inhibit laser beam emission if the sample chamber is disconnected from the LIBS-6 / LIBS-8 module or if the above-mentioned special cables are not connected. The laser safety interlock system is designed to "fail safe", as required by international standards on the safety of laser products. Accordingly, the LIBS equipment meets the requirements of a Class 1 laser product where under normal circumstances it is not necessary for the user to use laser safety eyewear. It is, however, necessary for the user to periodically test the correct functioning of the laser safety interlock circuit, ideally each time the equipment is used. The procedure for this test is described later but involves opening the sample chamber door while monitoring the status of the laser in order to check that the laser safety interlock system is working correctly. In the event of it not working properly, the LIBS equipment should be immediately taken out of service (remove the key from the laser keyswitch on the laser power supply) until the fault has been rectified.

The user has the option of using the LIBS equipment without a sample chamber for special experiments where it is not practical to use a sample chamber. In these "open beam" applications, the LIBS equipment must be treated as a Class 4 laser product and all relevant safety measures observed. During "open beam" use it is necessary to override the laser safety interlock system and this is achieved using the Interlock Override keyswitch located on the front panel of the SpectroModule-6 or SpectroModule-8. To override the interlock, the keyswitch should be turned "on" by turning clock-wise as indicated in the image below. The orange / yellow LED indicator to the left of the keyswitch (labelled Class 4 Warning) will flash continuously when the keyswitch is activated. When the keyswitch is set to "off", the LED will be extinguished indicating that the laser safety interlock is operating. The key should be removed from the Interlock Override keyswitch to prevent unauthorised activation of the interlock override. Consult your Laser Safety Officer regarding where this key should be stored.

WARNING
When the Interlock Override keyswitch is activated, the LIBS system becomes a Class 4 Laser Product, hence appropriate safety procedures and precautions MUST be adopted when operating the LIBS system in this mode. The key should be removed when this feature is not required and held by a responsible person (e.g. your Laser Safety Officer).

The two BNC connectors on the right side of the control panel are for optional connection to a laboratory door interlock switch and/or to an external warning light. Note that the "Warning Light" BNC connector provides 12 VDC (1 Amp max., centre pin +ve) whenever the laser is active. Note also that the "Door Intlk" BNC connector needs to be electrically "shorted" in order to satisfy the laser safety interlock circuit – this is achieved by fitting the blanking cap to the "Door Intlk" BNC connector when it is not connected to a laboratory door interlock switch.

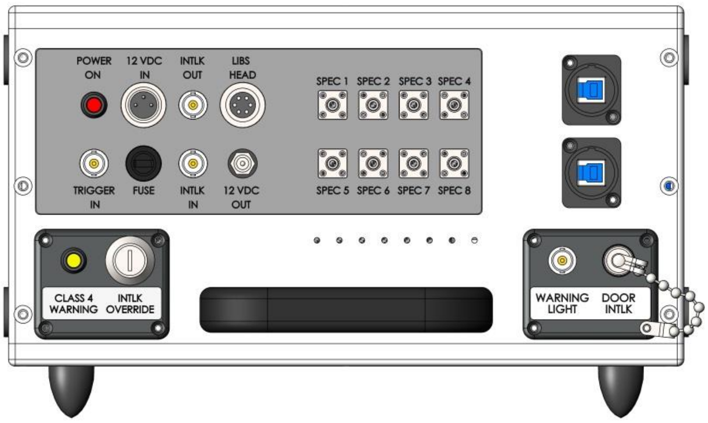


Interlock Override keyswitch and warning lamp (Class 4 warning lamp will flash when the keyswitch is activated)

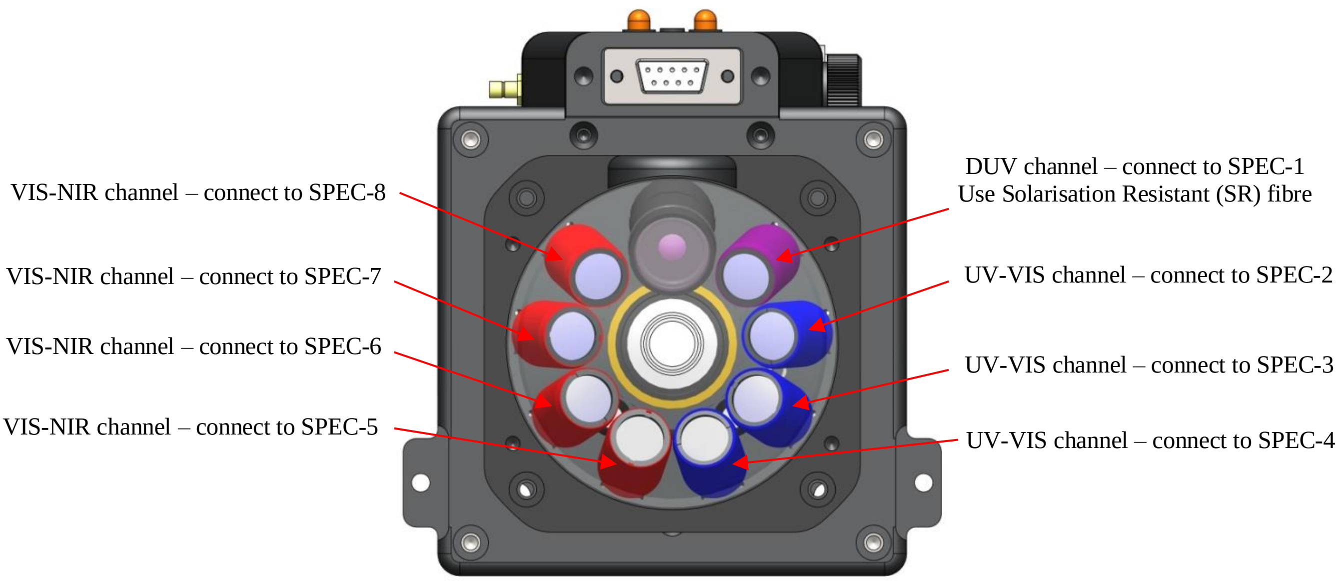
External warning light and door interlock connections (optional)

3.7 Fibre-optic connections

The LIBS-6 / LIBS-8 module is optically connected to the SpectroModule-6 / SpectroModule-8 spectrometer using six or eight optical fibres. One of the optical fibres is marked “SR” (solarisation resistant) and this fibre should be used for the Deep UV (DUV) channel (i.e. SPEC-1 on the spectrometer front panel and the purple lens holder at the 1 o’Clock position as viewed from the front of the LIBS-6 / LIBS-8 module). Proceeding in a clockwise manner, the remaining plasma light lens holders should be connected to the SPEC-2 – SPEC-8 channels on the spectrometer using the fibre-optic cables. It is important to connect the plasma light collection optic to the relevant spectrometer channel on SpectroModule-6 / SpectroModule-8 and so the collection optics in the LIBS-6 / LIBS-8 module are colour coded accordingly. This is described in the following section.

SpectroModule-8 multi-channel LIBS spectrometer	Plasma light collection optics in LIBS-8 module
	**SPEC 1 connects to PURPLE lens holder** **SPEC 2 connects to BLUE lens holder** **SPEC 3 connects to BLUE lens holder** **SPEC 4 connects to BLUE lens holder** **SPEC 5 connects to RED lens holder** **SPEC 6 connects to RED lens holder** **SPEC 7 connects to RED lens holder** **SPEC 8 connects to RED lens holder**

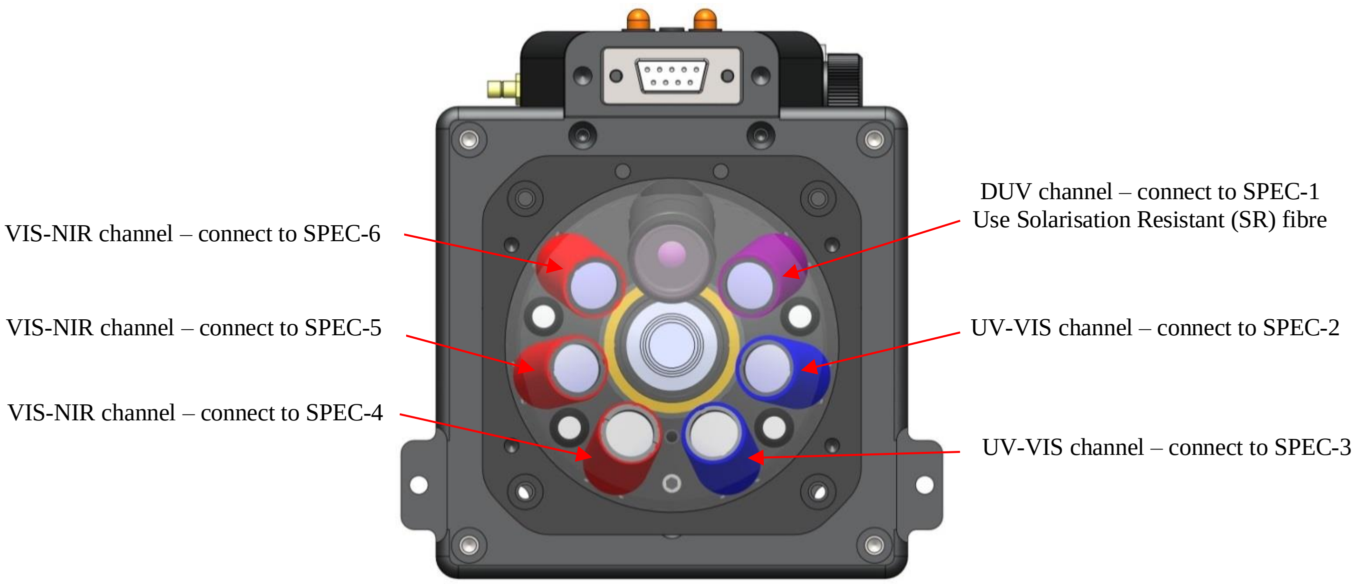
Fibre-optic connections between SpectroModule-8 and LIBS-8 module



Schematic diagram illustrating fibre-optic connectivity between LIBS-8 module and SpectroModule-8 spectrometer

SpectroModule-6 multi-channel LIBS spectrometer	Plasma light collection optics in LIBS-6 module
	**SPEC 1 connects to PURPLE lens holder** **SPEC 2 connects to BLUE lens holder** **SPEC 3 connects to BLUE lens holder** **SPEC 4 connects to RED lens holder** **SPEC 5 connects to RED lens holder** **SPEC 6 connects to RED lens holder**

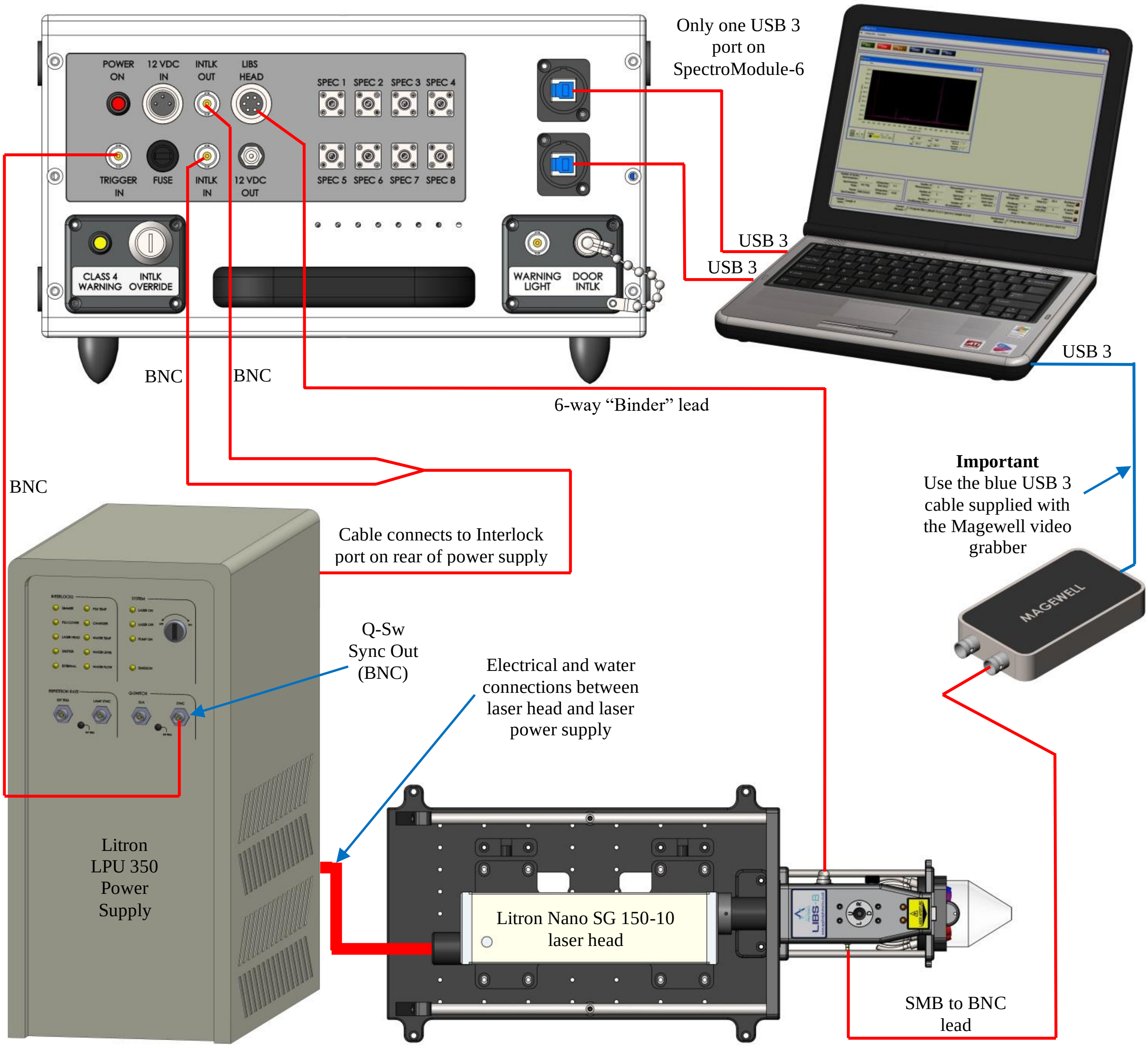
Fibre-optic connections between SpectroModule-6 and LIBS-6 module



Schematic diagram illustrating fibre-optic connectivity between LIBS-6 module and SpectroModule-6 spectrometer

© 1998 - 2022 Applied Photonics Ltd

3.8 Electrical connections



Schematic diagram illustrating the electrical connections between the various components of the LIBS system

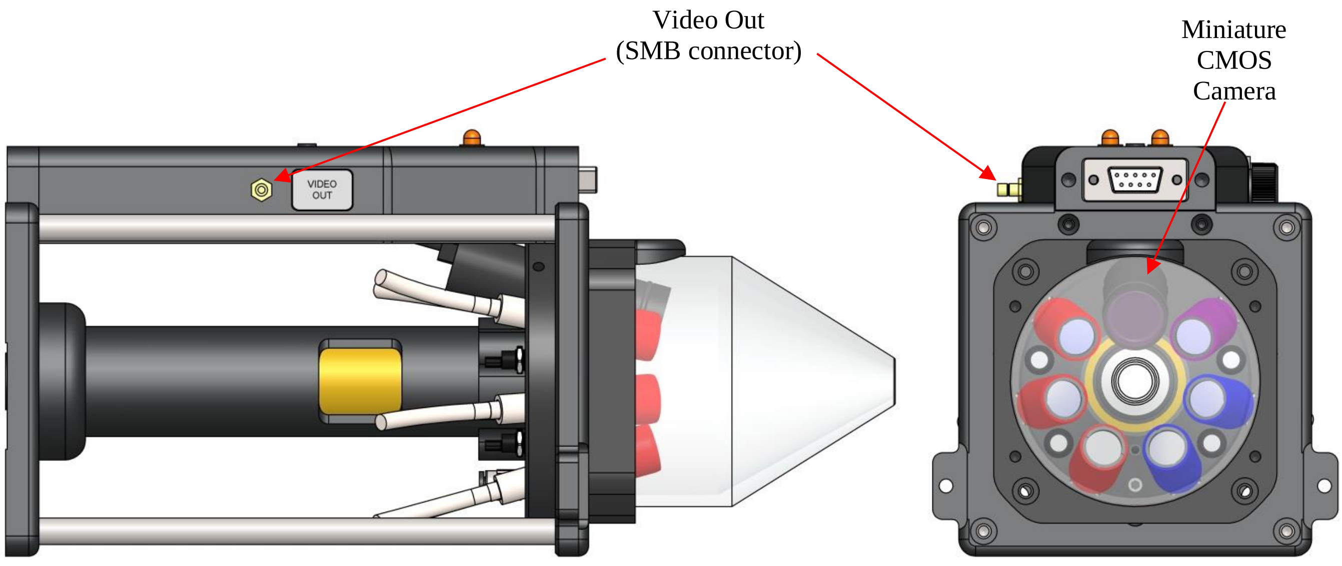
3.9 Imaging camera and associated components

This configuration of modular LIBS instrument is equipped with our IMG-HR imaging kit, which uses a single miniature CMOS camera for viewing the sample. The camera is integrated with the LIBS-8 module and so views the sample from above.

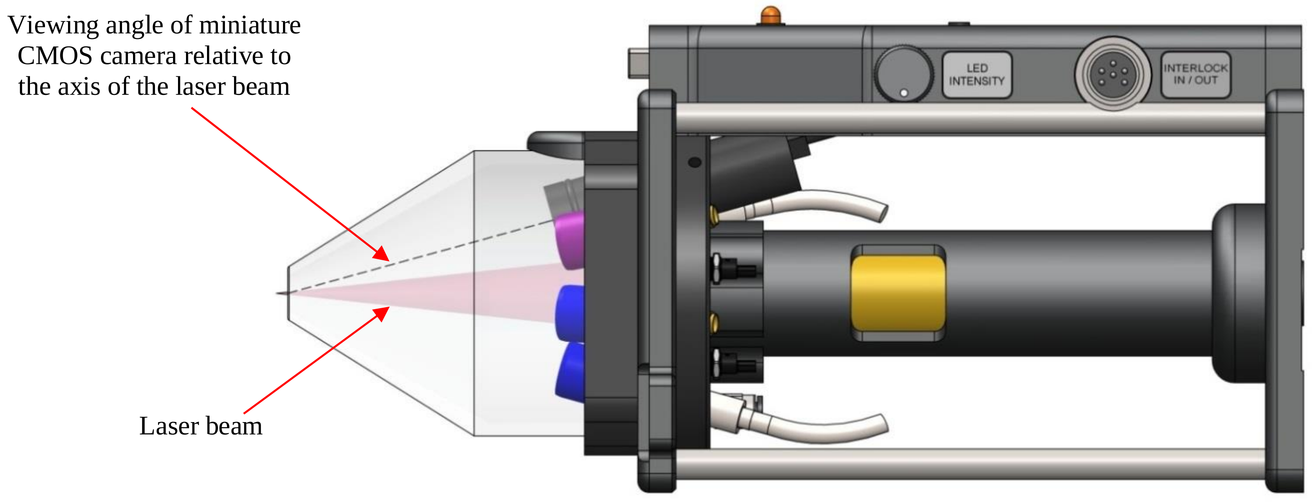
Camera specifications:

- 1/3-inch CMOS colour sensor 2.5 MP (1956 x 1266)
- Adjustable focus (approx. 20 mm to infinity – factory set during testing of LIBS system)
- Lens: f = 12 mm, f/1.8
- 12 VDC @ <1 A

Miniature CMOS camera integrated with the LIBS-6 module (optional extra)



CAD images of LIBS-6 module showing location of the "Video Out" SMB connector and the CMOS camera

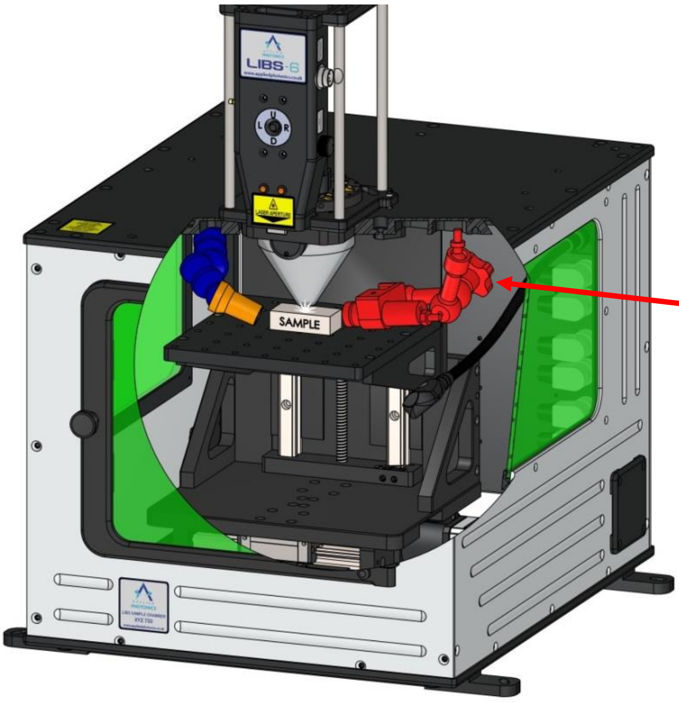


CAD image of LIBS-6 module showing viewing angle of integrated miniature CMOS camera (IMG-HR imaging kit)

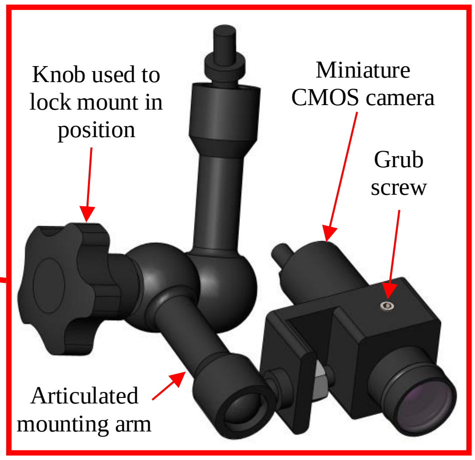
The video images are fed to the system computer via the USB frame grabber / capture device (Magewell) supplied with the imaging kit. There is one Magewell frame grabber / capture device for each camera being used. Note that these devices must be connected to USB 3 ports on the system computer using the proprietary USB cable (coloured blue) supplied with these devices.

Second miniature CMOS camera integrated with the XYZ-750 sample chamber (optional extra)

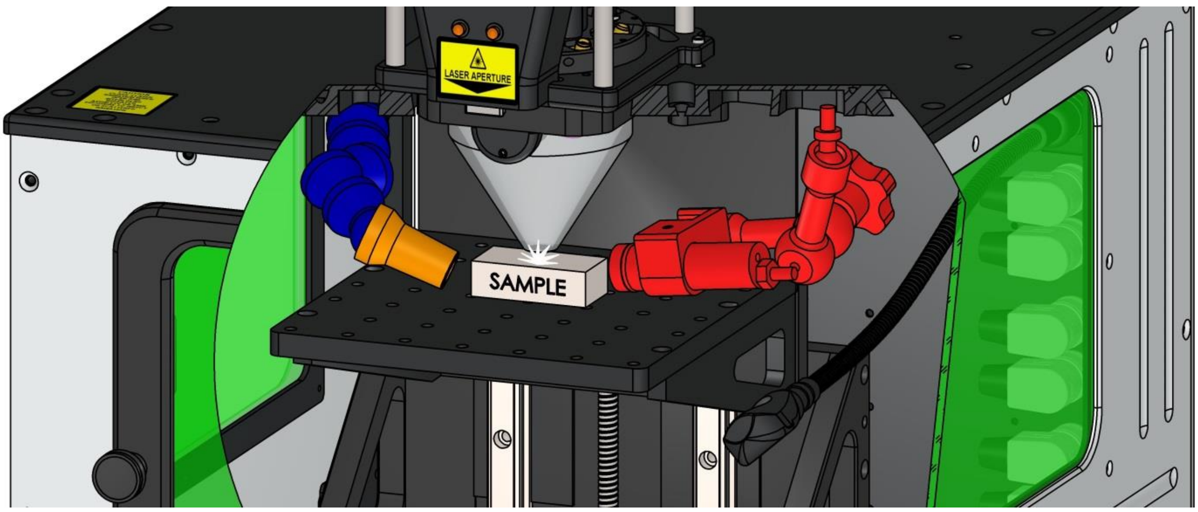
The second camera, which is supplied as part of our IMG-HR-2 dual camera imaging kit, is fitted inside the sample chamber as shown in the following diagrams. This camera provides a side view of the sample in the vicinity of the laser-induced plasma. The associated cable exits the sample chamber via a cable gland located in the rear panel of the sample chamber. This camera is equipped with an articulated mounting arm which allows the camera to be positioned manually at the desired position and then locked in place. In the diagram below, the camera and articulated mounting arm are highlighted in red. Note that the camera focus has been set during final testing of the LIBS equipment so it is necessary to position the camera at a distance from the object being viewed which results in a clearly focussed image. It may be necessary to adjust the position of the camera relative to the articulated arm mount by slackening the grub screw (see below) and sliding the camera towards or away from the object to help in this regard.



CAD view of XYZ-750 sample chamber showing second camera and articulated mounting arm (highlighted in red)



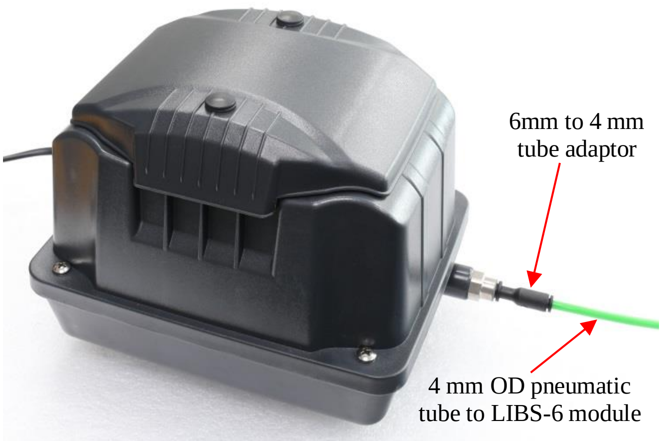
Detailed view of second camera with articulated mounting arm. The knob on the left is used to lock the knuckle joints of the articulated mounting arm when the desired position for the camera is found by manually positioning the camera. The camera may be repositioned relative to the articulated mount by slackening the grub screw which is used to secure it to the mount.



Close-up view showing second camera and articulated mounting arm (highlighted in red) relative to the sample position

3.10 Gas purge feature and air pump

The LIBS-6 and LIBS-8 modules are equipped with a gas purge feature which allows air, nitrogen or preferably argon gas to be supplied via the acrylic nozzle aperture to the area of the sample where the laser beam is directed. Argon is often used as a purge gas with LIBS since it increases significantly the intensity of the recorded spectra, especially in the UV region (see Test Report for example spectra which show this). Although it is not necessary to use gas purge when conducting LIBS analysis of a material, it is recommended that at least air is used as a purge gas so as to help to keep the optics free of ejected material from the laser plasma. In view of this, we provide with most of our bench top LIBS instruments a simple 20 litres per minute (lpm) air pump of the type used for aquariums to bubble air through the water. This is illustrated below. The pneumatic tube (coloured green) is connected to the "push fit" connector located on the optics array of the LIBS-6 / LIBS-8 module as shown below in the image on the right. This air pump may be left switched on for the entire time the LIBS equipment is in use.



230 VAC diaphragm air pump with 4 mm outside diameter (OD) pneumatic tube fitted

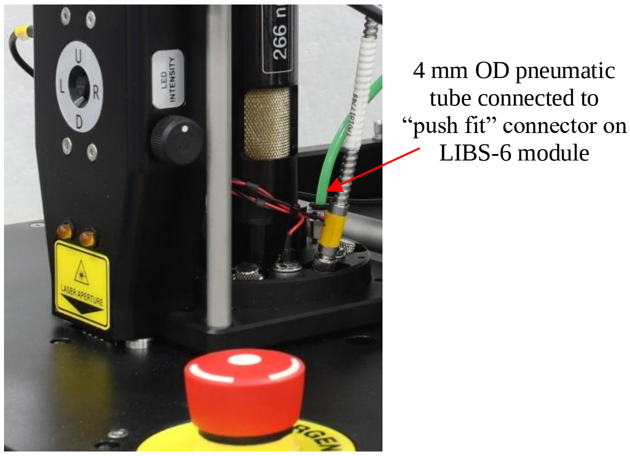
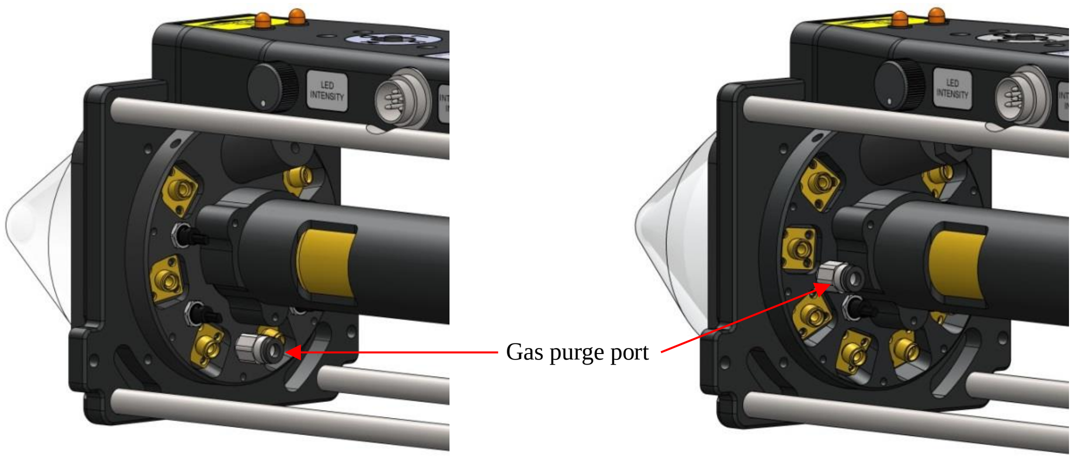


Image of LIBS-6 module showing pneumatic tube connected to "push fit" connector



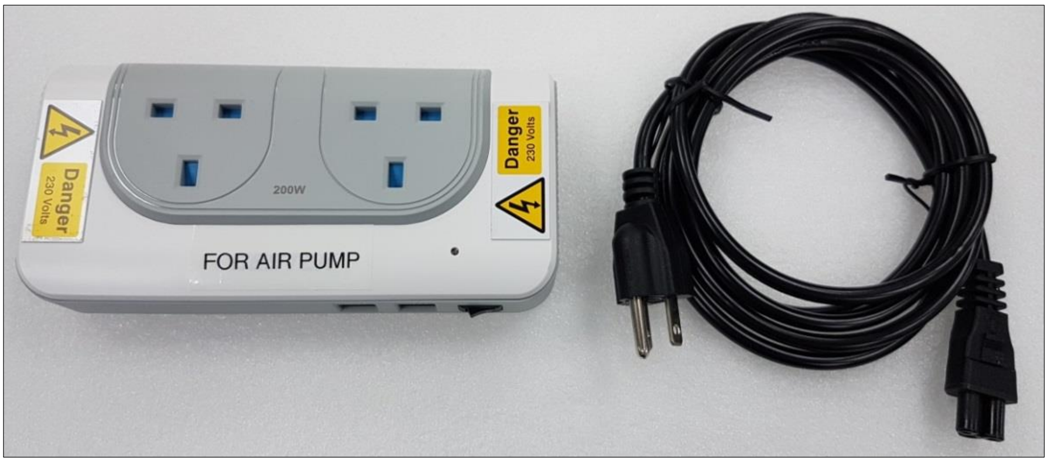
Location of the gas purge port (4 mm tube "push fit" connector) on the LIBS-6 (left) and LIBS-8 (right) modules

The above air pump is not used when other gases are used, such as nitrogen or argon. When using a gas other than air, alternative means of controlling the pressure and flow rate of the purge gas must be used such as a suitable regulator and flow meter (not supplied with the LIBS equipment). A flow rate of typically 5 to 10 lpm is usually sufficient. The gas pressure must be limited to not more than about 5 pounds per square inch (psi), although longer lengths of pneumatic hose may require higher pressure but never more than about 10 psi.

WARNING
To avoid an explosion risk, never use flammable gases as purge gas

© 1998 - 2022 Applied Photonics Ltd

We have included with the air pump a step-up transformer so that it can be run from a 120 VAC mains supply in the USA. The step-up transformer is short-circuit / over-current protected which makes it safe against accidental overloading but we recommend it is used only for the air pump. The air pump is equipped with a British 3-pin plug which is compatible with the step-up transformer.



120 to 230 VAC (200 Watts) step-up transformer for powering the air pump from a 120 VAC 50-60 Hz supply

3.11 LIBSoft software

APL's LIBSoft software has been designed specifically for APL's range of modular LIBS systems. LIBSoft can control the various hardware components that can form part of our modular LIBS systems including the laser, the spectrograph, the delay generator, the sample chamber translation stages and the imaging kit (single or dual camera options). LIBSoft also controls data acquisition offering a number of features specifically developed for LIBS. LIBSoft has some data processing features including emission line identification and tools for constructing calibration plots for quantitative analysis. LIBSoft is continually being developed further to include more features and APL offers free software upgrades to its customers for a period of two years. A detailed User's Manual for LIBSoft is supplied with the LIBS equipment.

3.12 System computer specifications

The minimum computer specifications for running LIBSoft with the modular LIBS system described in this User's Manual (i.e. incorporating a SpectroModule-6 or SpectroModule-8 multi-channel LIBS spectrometer) are as follows:

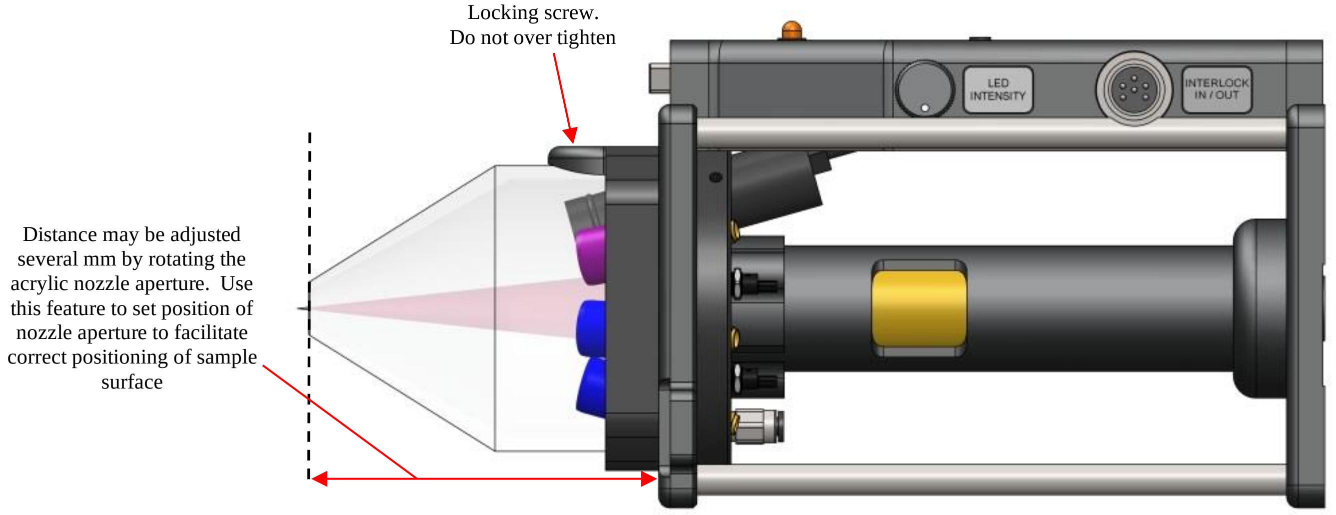
- Microsoft Windows 7, 8.1 or 10
- Preferably an i5/i7 processor or equivalent
- At least 4 GB of memory
- Screen resolution of at least 1024 x 768
- At least 3 spare USB3 ports (i.e. not including ports used for mouse, keyboard etc)
- At least one serial port (RS 232)

Please note the following:

- The SpectroModule-6 requires one USB3 port
- The SpectroModule-8 requires two USB3 ports
- The IMG-HR single camera imaging kit requires one USB3 port
- The IMG-HR-2 dual camera imaging kit requires two USB3 ports
- One serial port is required for the laser (we do not recommend using a USB-to-Serial converter)

3.13 Setting the position of the acrylic nozzle aperture

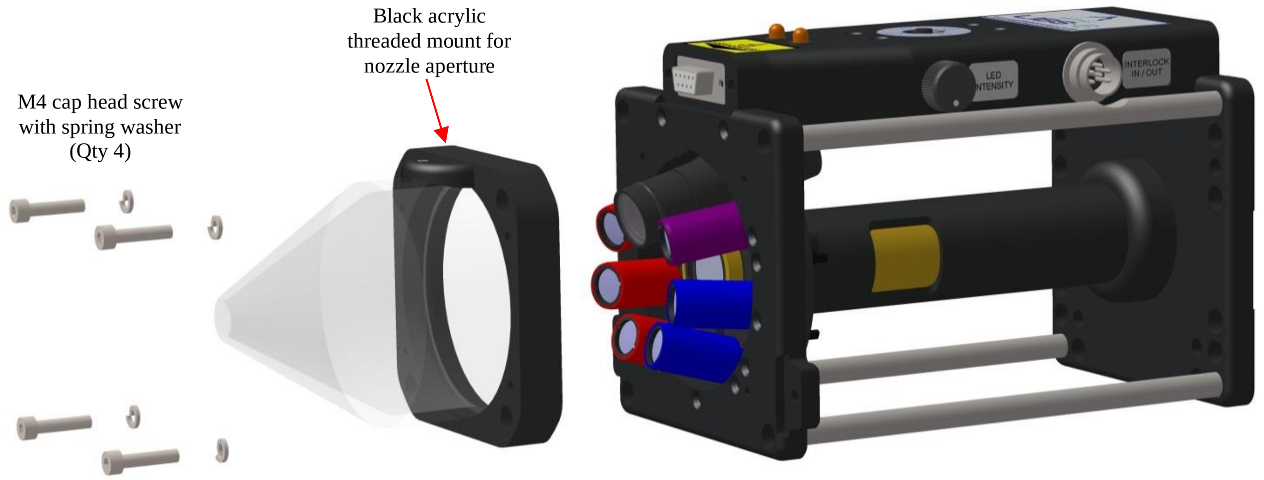
The primary purpose of the transparent acrylic nozzle aperture is to provide a convenient means of setting the sample position with respect to the laser beam focus. It also provides a means of directing the purge gas, if used, to where it is needed, i.e. the region where the laser-induced plasma is formed. The use of the acrylic aperture nozzle is optional and so it may be removed if required although an alternative method of setting the sample position will be required. The acrylic nozzle aperture is threaded so rotating it will move the position of the aperture along the axis of the laser beam. During final factory testing of the LIBS equipment, the acrylic nozzle aperture was set at a near optimum position for the solid samples used for testing the LIBS equipment (see the Test Report supplied with the equipment).



CAD diagram of a LIBS-6 module illustrating adjustment of the acrylic nozzle aperture

The user may choose to set the acrylic nozzle aperture such that it is several mm from the sample and then use a distance piece (e.g. a flat section of plastic of appropriate thickness) to set the correct position for the sample.

To avoid losing the position of the nozzle aperture by unscrewing it from its mount, it is recommended that the nozzle aperture complete with its mount (black acrylic threaded component) is removed from the LIBS-6 module by unscrewing the four M4 cap head screws used to secure the mount to the LIBS-6 module, as illustrated in the following diagram.

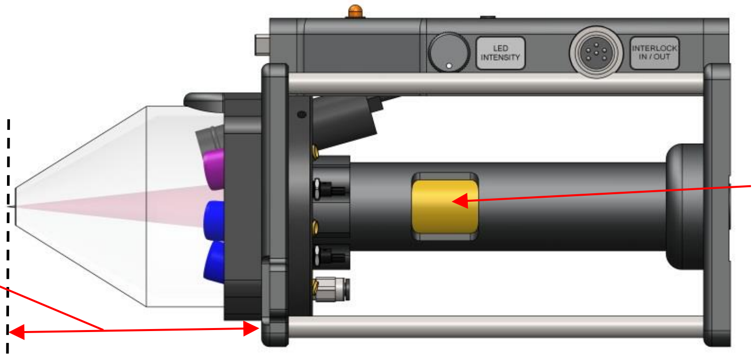


CAD diagram of a LIBS-6 module illustrating removal of the acrylic aperture nozzle complete with its threaded mount

© 1998 - 2022 Applied Photonics Ltd

3.14 Adjusting and setting the laser beam focus

The following diagram illustrates the method of adjusting the position of the focal plane of the laser beam. During factory testing, the LIBS-6 module was set such that the sample is assumed to be in contact with the acrylic nozzle aperture (or within 1 mm of contact) and the focal plane of the laser beam was set to be 2 to 3 mm beyond the plane of the nozzle aperture. This is illustrated clearly in the image below which shows a laser-induced plasma in air.



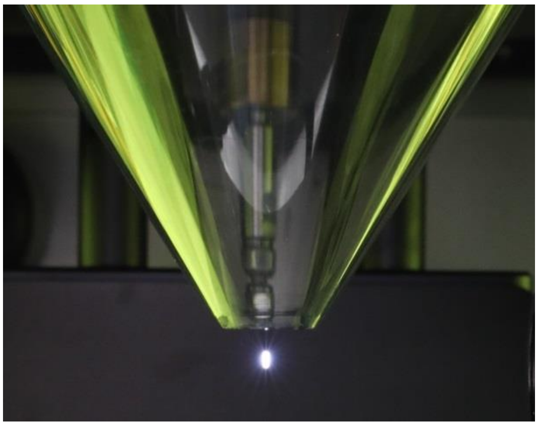
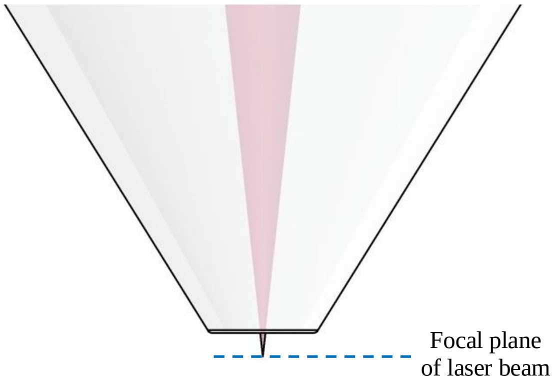
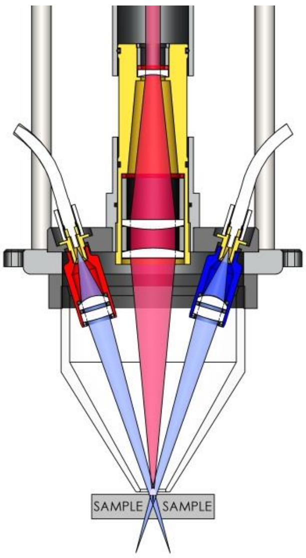


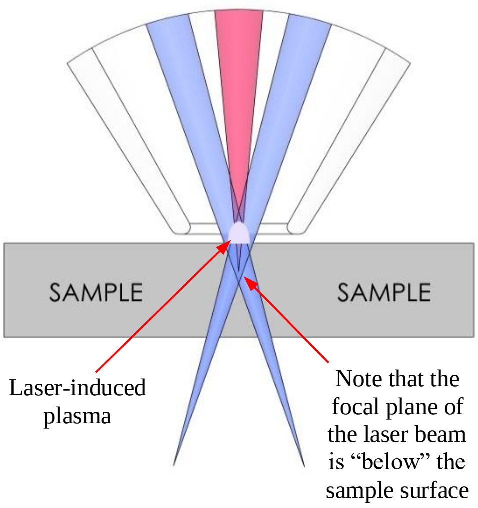
Image showing laser-induced plasma in air located a few mm below the acrylic nozzle aperture



CAD view showing position of focal plane of laser beam relative to the acrylic nozzle aperture



CAD illustration of field of view of the plasma light collection optics



Close-up view of field of view of collection optics in the vicinity of the laser-induced plasma

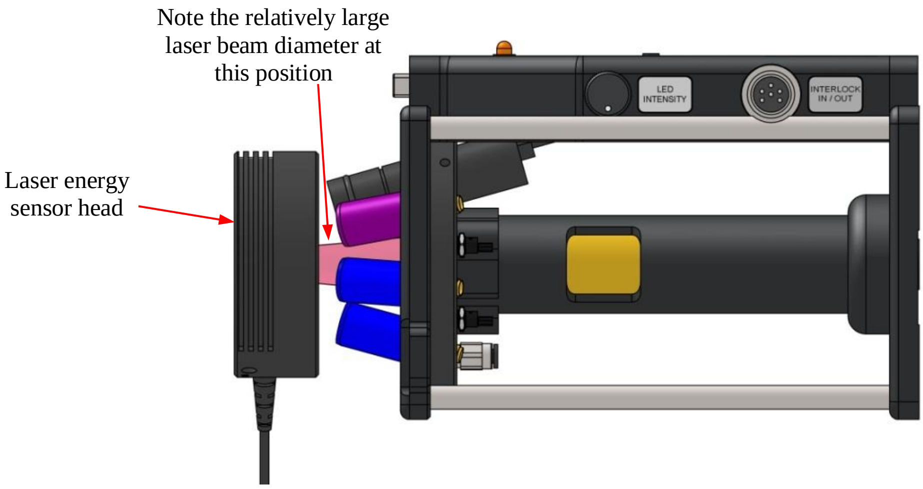
The collection optics array has a fixed field of view and so it is necessary that the laser-induced plasma is located within the convergence point of the six collection optics. This is illustrated in the diagrams on the left. Note that the focal plane of the laser beam is "below" the surface of the sample by approximately 1 to 2 mm – this is normal and what we recommend when conducting LIBS analysis of solid samples. The optimum laser focussing conditions will depend on the nature of the experiment - contact Applied Photonics Ltd for further advice.

3.15 Using an energy / power meter to measure the laser pulse energy

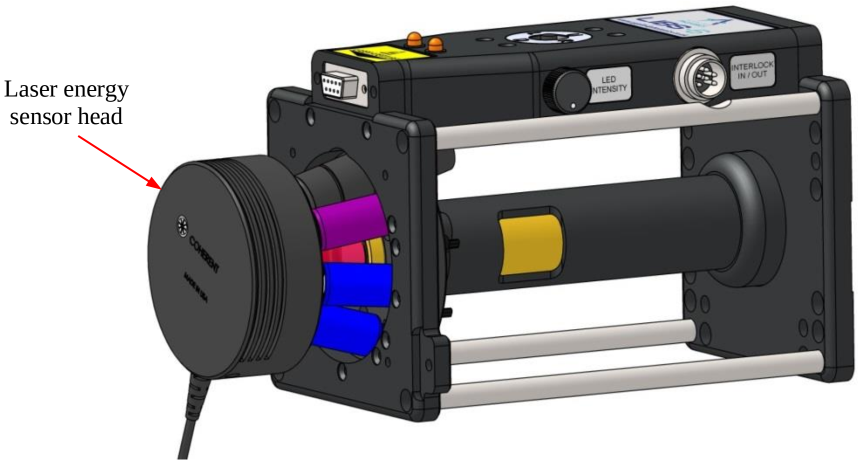
We recommend the Coherent LabMax TOP laser energy meter with J-50MB-YAG sensor head. To avoid damaging the sensor head of the energy meter through exposure to excessively high laser beam power densities, it is important that the following procedure is followed.

Remove the acrylic aperture nozzle from the LIBS-6 / LIBS-8 module. To avoid losing the position of the nozzle aperture by unscrewing it from its mount, it is recommended that the nozzle aperture complete with its mount (black acrylic threaded component) is removed from the LIBS-6 / LIBS-8 module by unscrewing the four M4 cap head screws used to secure the mount to the LIBS-6 / LIBS-8 module. The sensor head of the energy meter should then be positioned centrally with respect to the laser beam axis and close the collection optics as shown in the following diagram. The laser beam diameter will be larger at this position and so the power density on the sensor head will be relatively low.

Important: Under no circumstances should the sensor head be positioned further away where the laser beam diameter is much smaller as the sensor head could be permanently damaged due to excessively high laser beam power density.



Laser energy meter sensor head correctly positioned with respect to the LIBS-6 / LIBS-8 module



Laser energy meter sensor head correctly positioned with respect to the LIBS-6 / LIBS-8 module

Never Do This!

3.16 Important note on back-reflections of laser radiation

Please note that when using high-power Q-switched lasers of the type used with LIBS equipment, it is very easy to cause serious damage to the optical components of the LIBS equipment, including the laser, by inadvertently creating a situation where a significant amount of laser radiation is reflected back into the LIBS-6 module and/or laser head. Persons familiar with the use of these types of laser will appreciate that merely placing a glass window in the path of the laser beam can result in up to 4% of the laser radiation **from each optical surface** to be reflected back towards the laser source. If the laser beam is being focussed to a small spot (as in the case of a LIBS instrument), the back-reflected laser light will also be focussed to a small spot and if this happens to coincide with an optical element such as a lens, output coupler or laser rod, significant and permanent damage to that optical element can occur. Accordingly, it is very bad practice (and potentially a very expensive mistake on the part of the user) to carelessly place objects in the beam path of a Q-switched laser. Clearly with LIBS it is necessary to place an object in the laser beam path so certain precautions must be observed, as follows.

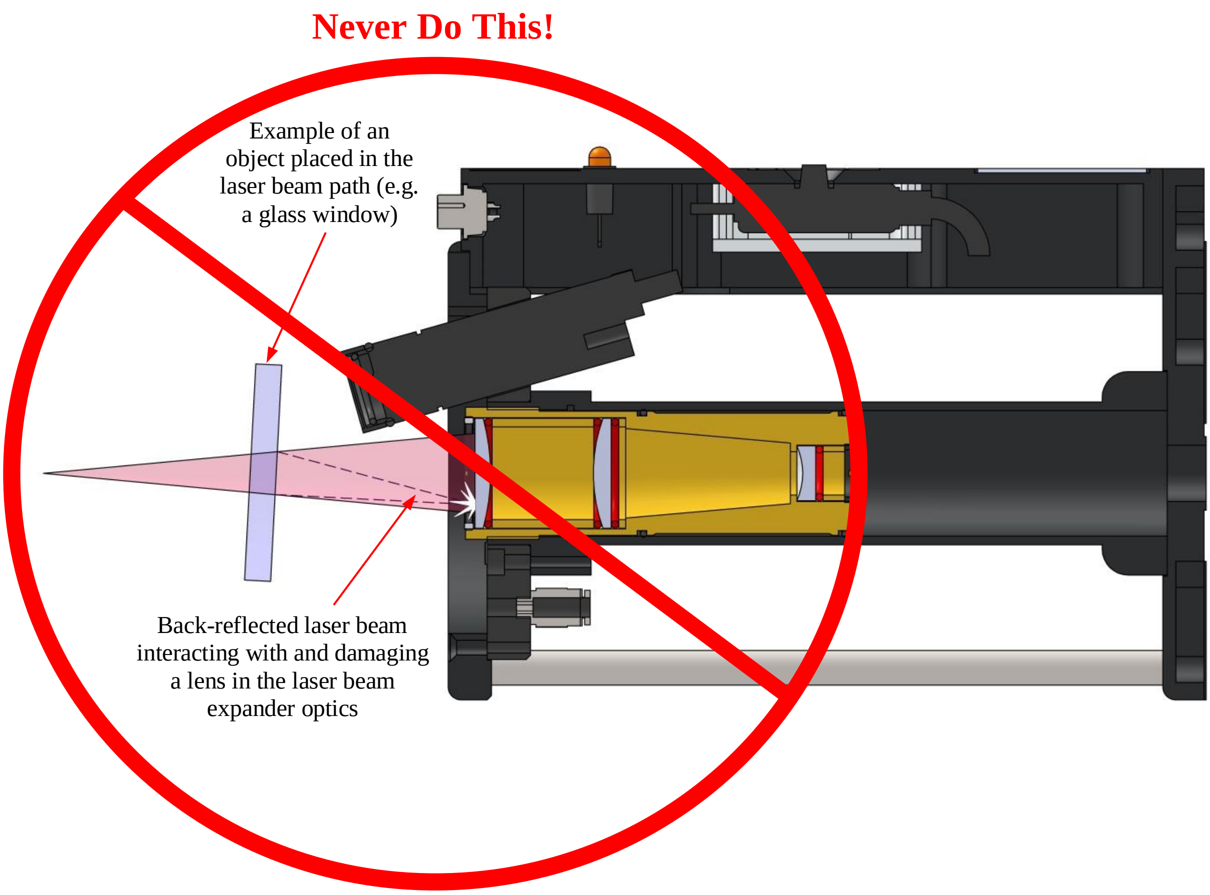
One way of considering this issue is as follows. If a laser plasma is being formed, either on the sample or object you have placed in the laser beam path or in the air / gas in front of the sample / object, then back-reflections are highly unlikely to be a problem since most of the laser energy will be absorbed by the plasma. It is when a laser plasma is not formed that you have to really worry about back-reflections. If you have placed an object in the laser beam path in such a way that a laser plasma is not formed on that object, then you should carefully consider what is happening to the laser energy which is impinging on the surface of the object since all materials will reflect at least some of the laser radiation.

Unless the user is particularly familiar with the use of Q-switched Nd:YAG lasers and is fully aware of the risks of damage to optics due to back-reflections of laser radiation, we strongly recommend that the LIBS-6 / LIBS-8 module is **used only with the nozzle aperture fitted**. The presence of the nozzle aperture prevents the user from placing an object too close to the laser beam expander optics in the LIBS-6 / LIBS-8 module, so that the object can only be located close to the focal plane of the laser beam, under which conditions a plasma will be formed unless the laser pulse energy is set to a very low value, or beyond the focal plane, where the laser beam will be diverging. Our advice to the user is that they should carefully consider the issue of back-reflections before placing any object in the laser beam path. Under

© 1998 - 2022 Applied Photonics Ltd

most situations, placing an object at or beyond the focal plane of the laser beam will not result in back-reflections which are of sufficient intensity to cause damage. There are, however, always scenarios where damaging back-reflections can still occur, such as placing a concave mirror in the laser beam path beyond the focal plane of the laser beam! If you have any doubts or concerns about your proposed application potentially causing damaging back-reflections, contact Applied Photonics Ltd before using your LIBS equipment. It is the responsibility of the user to minimise the risk of damage to optics caused by back-reflections of laser radiation. Please note that the manufacturer's warranty does not cover damage caused by back-reflections of laser radiation.

The following diagram illustrates back-reflected laser radiation from an object placed in the laser beam path. This object could be a piece of steel, a glass window, or virtually any type of material since most materials reflect at least some of the laser beam. The case of the glass window is a good example as some experimenters may wish to conduct LIBS analysis of a solid sample (or gas sample) inside a glass vessel or through a window of a vacuum chamber. Although most of the laser beam energy will be transmitted by the glass window, approximately 4% of the laser beam will be reflected from each surface (outer and inner) which is more than enough to cause permanent damage to an optical element on which the reflected laser beam impinges. It is possible to conduct LIBS analysis through a window but great care needs to be exercised to avoid potentially damaging back reflections. Contact Applied Photonics Ltd should you require any advice on this matter.



CAD sectional view of LIBS-6 / LIBS-8 module showing back-reflected laser beam from object placed in the beam path

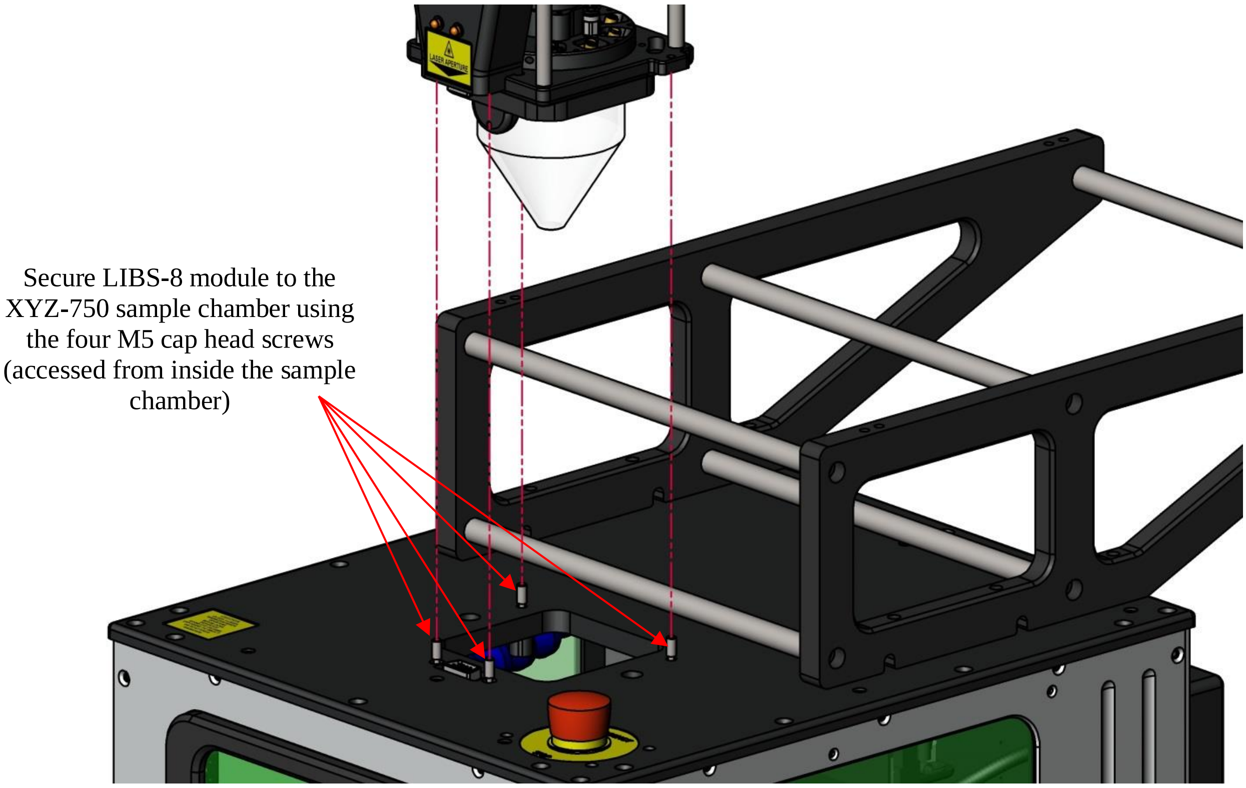
4 Assembly of the LIBS equipment

4.1 Introduction

It will be necessary for two people to be available to conduct some aspects of the assembly work, in particular manually lifting the Adaptor Platform with attached laser head and fitting it to the sample chamber support brackets and the Mirror Module. The following steps are relevant to assembling the equipment where the XYZ-750 sample chamber is used. The assembly and use of the SC-LQ2 liquids sample chamber is described later in this User's Manual.

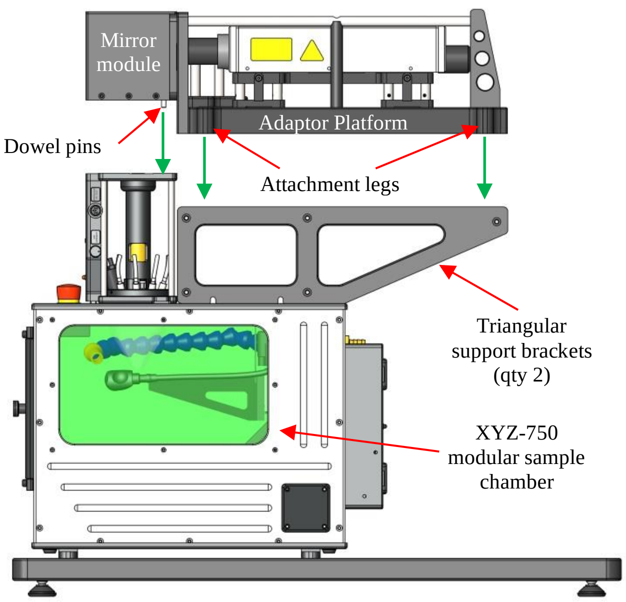
4.2 Step-by-Step guide to configuring the LIBS equipment

Step 1 Fit the LIBS-8 module to the XYZ-750 sample chamber as illustrated in the following diagram:



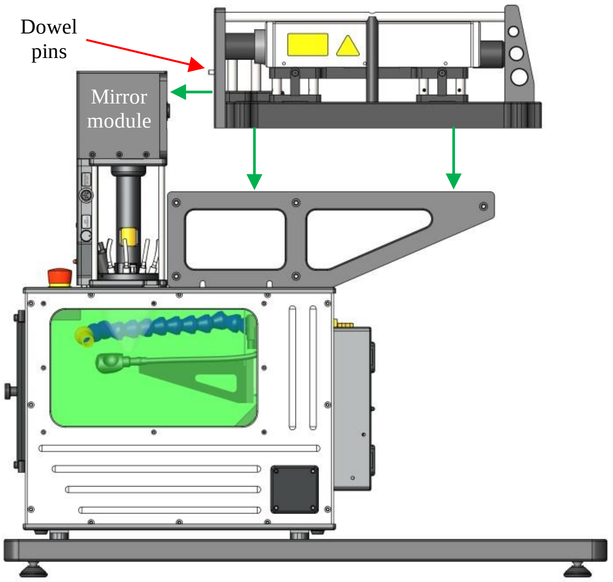
The LIBS-8 and LIBS-6 modules are designed to fit to the top of the SC-2L / XYZ-750 sample chambers using four captive M5 screws as illustrated in the figure above. Before fitting the LIBS module to the sample chamber, ensure that the sample table is positioned sufficiently low so as not to make contact with the aperture nozzle of the LIBS module. Be careful to fit the LIBS module squarely to the sample chamber so as not to cause damage to the electrical connectors.

Step 2 This step will require two people. Take the adaptor platform with attached laser head and mirror module and carefully offer it up (in the direction of the green arrows) to the LIBS-8 module and triangular support brackets of the XYZ-750 sample chamber as illustrated in the following diagram:

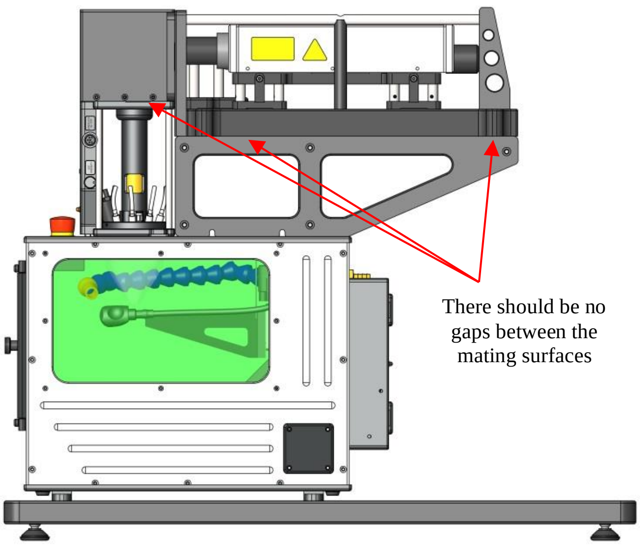


Carefully align the two dowel pins (located in the lower bulkhead of the mirror module) with the holes in the upper bulkhead of the LIBS-8 module. Once the dowel pins are correctly located, the whole assembly should then be lowered further until the four legs of the adaptor platform come into contact with the triangular brackets. Use the supplied four M6x20 cap head screws to secure the adaptor platform to the triangular brackets.

Note that there are four M4x16 cap head screws supplied with the equipment that may be used to secure the upper bulkhead of the LIBS-8 module to the mirror module – although it is not necessary to do this for this particular configuration since the dowel pins provide an adequately secure fixing on their own.



Alternatively, the Mirror Module may be first fitted to the LIBS-6 / LIBS-8 module (as shown in the illustration on the left) and the Adaptor Platform with attached laser head may be lowered onto the triangular support brackets and then slid towards the Mirror Module so that the dowel pins engage with the holes in the Mirror Module.



Before fully tightening the four M6x20 cap head screws, check that the legs of the adaptor platform are in contact with the triangular brackets and that the mirror module is in contact with the upper bulkhead of the LIBS-8 module (i.e. there should not be any gaps between the mating surfaces).

The laser head is fitted to the laser head baseplate (see section 3.4.2) prior to shipping from Applied Photonics Ltd. Please note that the laser head should not be removed from the laser head baseplate without first seeking advice from Applied Photonics Ltd since the laser head is critically aligned (refer to Section 3.4 for further information on how to remove and refit the laser head (with attached laser head baseplate) from/to the adaptor platform).

Step 3 Connect the LIBS-6 / LIBS-8 module to the SpectroModule-6 / SpectroModule-8 using the supplied lead (terminated with 6-way circular "Binder" connectors).

Step 4 Connect the coolant pipes and electrical cables (supplied with the laser) between the laser head and the laser power supply. Refer to the laser manufacturer's instructions.

Step 5 Using the supplied BNC cables, connect the INTLK IN connector on the SpectroModule-8 to the interlock connector on the rear panel of the Litron laser power supply.

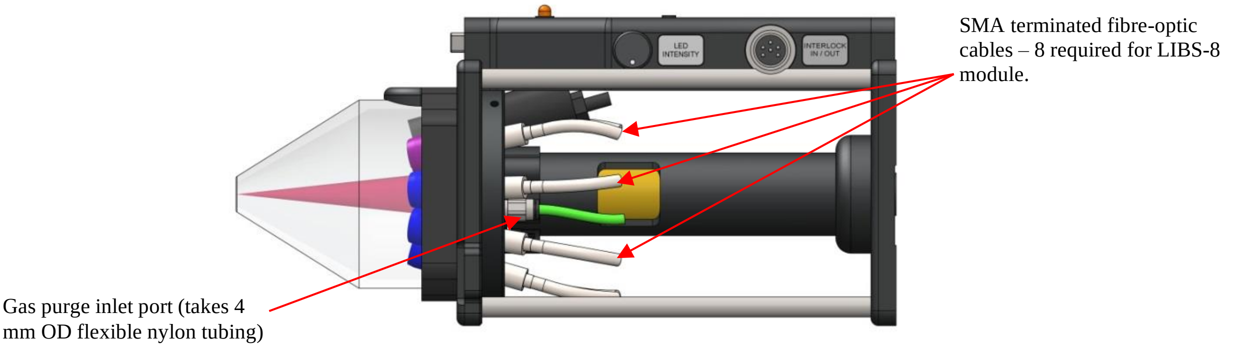
Step 6 Connect the "Trigger In" port on the SpectroModule-6 / SpectroModule-8 to the "Q-Switch Sync OUT" port on the front of the laser power supply using the supplied BNC lead.

Step 7 Connect the USB frame grabber (Magewell) device to the system computer using the USB leads supplied with the frame grabber (coloured blue) and connect the frame grabber to the Video Out port on the LIBS-6 / LIBS-8 module using the supplied SMB-to-BNC lead.

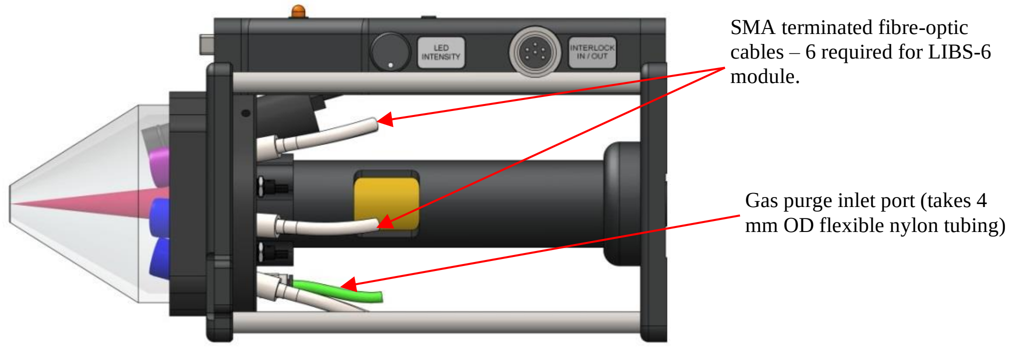
Step 8 Connect the fibre optic cables between the LIBS-6 / LIBS-8 module and the SpectroModule-6 / SpectroModule-8 spectrometer, following the instructions provided in section 3.7

Step 9 Connect the supplied serial cable between the laser power supply and the system computer.

Step 10 If the gas-purge feature is to be used, connect a suitable inert gas supply (Argon, Helium, Nitrogen, Air) to the "Inert Gas Supply" port using 4 mm OD flexible tubing (a length of this type of tubing is supplied with the LIBS module – coloured green). **WARNING - the gas supply MUST be externally regulated to restrict pressure to less than approx. 5 psi (2 to 3 psi should be adequate) and to control the flow-rate. DO NOT USE FLAMMABLE GASES!**



LIBS-8 module showing fibre-optic and pneumatic tube connections



LIBS-6 module showing fibre-optic and pneumatic tube connections

5 Operating procedure

5.1 Introduction

This operating procedure assumes that you are using an Applied Photonics Ltd SpectroModule-6 (or SpectroModule-8) LIBS spectrometer as part of your LIBS equipment. If you are using an alternative spectrometer, you may need to use an additional device to interface the LIBS-6 / LIBS-8 module with your spectrometer and the laser power supply. If in any doubt, you should contact us for advice prior to setting up your LIBS equipment as incorrect or incomplete electrical connections between the LIBS-6 / LIBS-8 module and the laser power supply will render the laser safety interlock functions ineffective. For operation and maintenance of the laser, refer to the relevant manufacturer's User's Manual that is supplied with the laser system.

5.2 Step-by-Step guide to operating the LIBS equipment

Step 1 Follow the laser manufacturer's instructions supplied with the laser for correct procedure for adding coolant (deionised or distilled) water to the laser power supply. Distilled water is preferable to deionised water.

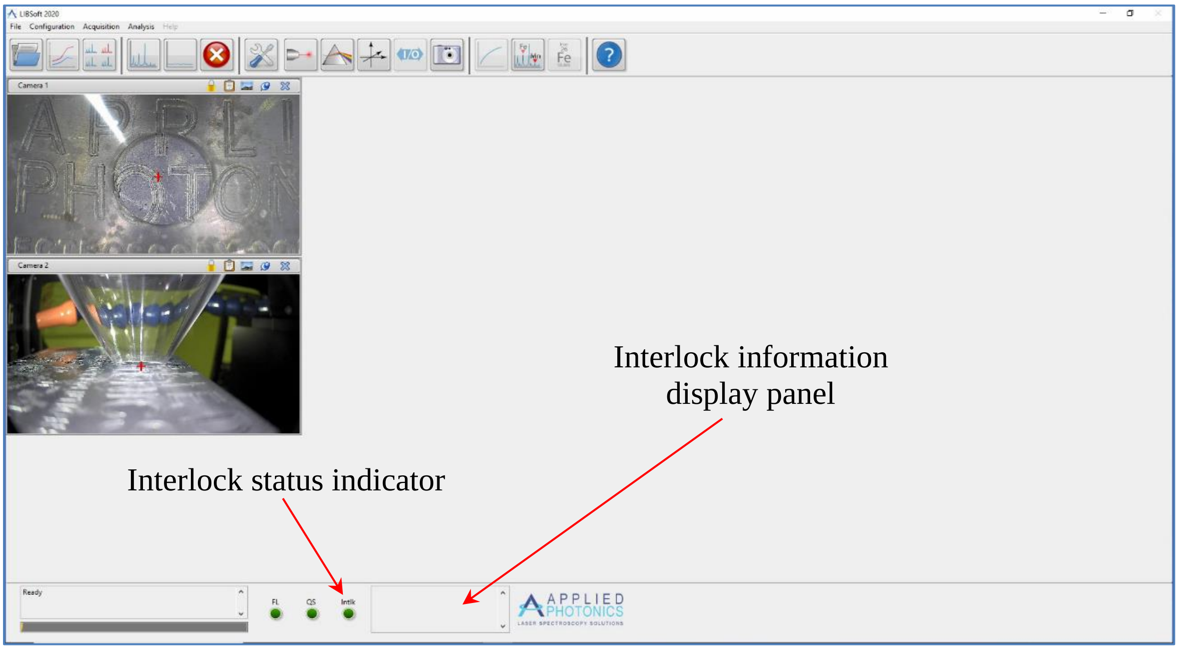
Step 2 Power up the laser by connecting to mains electrical supply and switching on the key switch (refer to laser manufacturer's instructions).

Step 3 Follow the instructions provided in the LIBSoft User's Manual to install LIBSoft on the system computer. It is important that the software installations instructions are followed exactly as stated in the manual.

Step 4 Connect the SpectroModule-6 / SpectroModule-8 to the system computer using the supplied USB lead (two USB leads are used with the SpectroModule-8).

Step 5 Power up the SpectroModule-6 / SpectroModule-8 using the supplied 12 VDC plug-in power supply.

Step 6 Follow the instructions provided in the Software User's Manual to launch the application and display the main operating screen (see screenshot below).

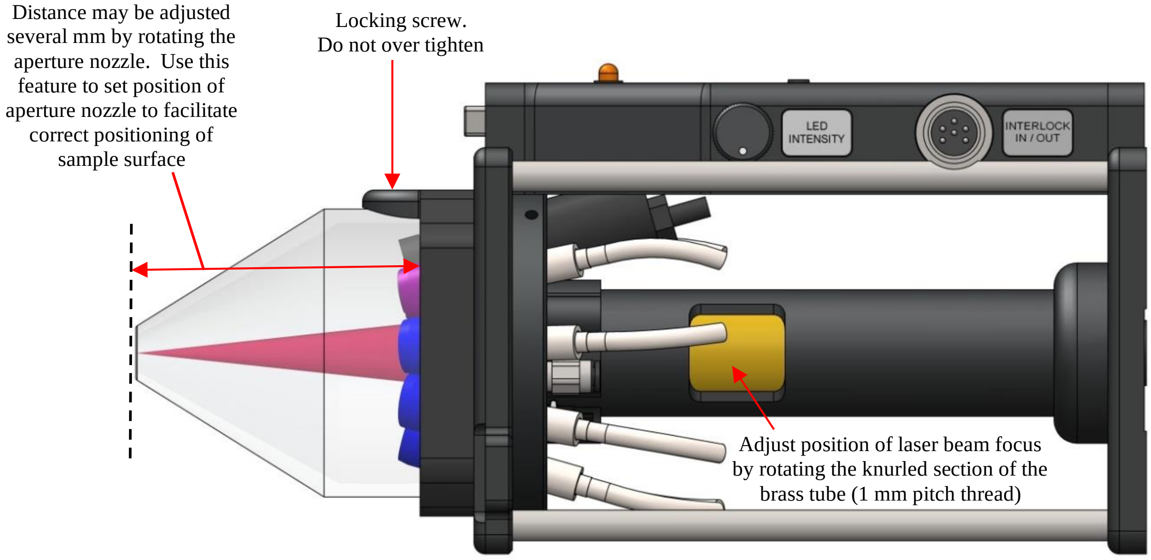


When one of the interlocks connected to the laser is activated the interlock status indicator changes from green to red and the source of the interlock is displayed in the adjacent information panel. For example, if the door of the sample chamber which is connected to the laser interlocks is opened, the interlock status indicator will be red and the interlock information panel will display ALARM = [REMOTE INTERLOCK].

Step 7 Ensure that the sample chamber door is fully closed. Via the LIBSoft software, activate the laser flashlamp (but NOT the Q-Switch). The flashlamps will commence flashing but no laser beam will be emitted. Now check for correct operation of the safety interlock by observing the interlock status light on the computer screen (described above) and also the laser warning light on the LIBS-6 / LIBS-8 module - opening the door of the sample chamber should deactivate the laser flashlamps, the LIBSoft interlock status light should revert to bright red (indicating an interlock is not satisfied) and the laser warning light on the LIBS-6 module should turn off. If the interlock is found not to be operating correctly, refer to the *Fault Finding* section of this User's Manual. Note that if the LIBS-6 / LIBS-8 module is removed from the sample chamber, the interlock circuit is designed to de-activate the laser (see section 7 on operating the LIBS equipment in "open-beam" configuration). **Warning – Do not operate the equipment if the safety interlock is not functioning correctly**.

NB When the activated interlock is cleared, e.g., the sample chamber door is closed, the laser automatically performs a short reset procedure. This is indicated in the status panel at the bottom of the main screen and the flashlamps cannot be restarted or an acquisition initiated until this procedure is complete.

Step 8 Place a sample of material (e.g. a metal block) at the focal plane of the Nd:YAG laser beam inside the sample chamber. Close the door of the sample chamber. Using the laser controls on the LIBSoft main screen, activate a single pulse of the laser beam by i) first activating the flashlamp and ii) then activating the Q-Switch. If the sample material is located at or near to the focal plane of the laser beam, a laser-induced plasma will be produced on the surface of the sample. It may be necessary to adjust the position of the aperture nozzle to obtain correct positioning of the sample surface (refer to section 3.13 of this User's Manual).



CAD view of LIBS-8 module illustrating method for adjusting position of nozzle aperture and laser beam focus

Step 9 If using LIBSoft software to control the spectrometers, follow the instructions provided with in the LIBSoft software User's Manual to correctly configure the spectrometers and the laser for acquiring data.

Step 10 Adjustment of the laser output energy is achieved by adjusting the Flashlamp-to-Q-Switch (FL-QS) delay time. When running the laser in manual mode, the FL-QS delay time may be adjusted via the Litron LUCi controller (refer to laser manufacturer's instructions). When running the laser via LIBSoft, the laser energy may be adjusted in a similar fashion but by entering an appropriate FL-QS delay time via LIBSoft rather than the Litron LUCi controller. If the laser energy has been calibrated (i.e. output energy vs FL-QS delay), it is possible to enter this calibration into LIBSoft so that the laser energy is displayed in mJ rather than as FL-QS delay in microseconds. Refer to the LIBSoft User's manual and/or the application "Help" function available in LIBSoft for guidance on this.

Step 11 After successfully testing the LIBS-6 / LIBS-8 module and associated equipment, it is now ready for use. Measurement conditions such as Nd:YAG laser pulse energy and position of sample surface with respect to laser beam focal plane will need to be adjusted to suit the requirements of the application.

6. Shut-down procedure

Step 1 Using the LIBSoft software, turn the laser flashlamps off.

Step 2 Shut down LIBSoft software and then switch off laser power supply (using the keyswitch on the laser power supply) and isolate from mains electrical supply.

Step 3 Remove the keys from the laser power supply keyswitch to prevent unauthorised activation of the laser.

Step 4 Switch off the SpectroModule-6 / SpectroModule-8 by isolating the 12 Volt DC plug-in power adapter from mains electrical supply and disconnect from the SpectroModule-8 spectrometer.

Step 5 Disconnect purge gas supply if connected.

7. Operating the LIBS equipment in "open beam" configuration

Warning Class 4 Laser Product.
Only suitably trained and experienced persons under the supervision of a duly appointed Laser Safety Officer should operate the LIBS equipment in "open beam" configuration.

The LIBS equipment may be used without the sample chamber if required (e.g. when analysing a sample which is too large to fit inside the sample chamber). Under these conditions, the laser beam is not contained (i.e. the equipment is a Class 4 laser product) and hence additional safety precautions must be observed including the use of appropriate laser protective eyewear and operating the laser equipment in a suitable controlled environment (e.g. a laser laboratory). **Important - it is the responsibility of the user to conduct an appropriate risk assessment prior to using the LIBS modules in open-beam mode**. If the sample chamber is removed, it will be necessary to activate the Interlock Override keyswitch located on the control panel of the SpectroModule-6 or SpectroModule-8 spectrometer (refer to sections 2.1 and 3.6 for further information on the Interlock Override feature). The LED warning light (labelled "Class 4 Warning") will commence flashing. Consult your Laser Safety Officer before operating the LIBS equipment in this mode.

8. Maintenance and inspection

The LIBS equipment should be periodically inspected for signs of damage or wear and tear. Of particular importance are the safety features including the laser safety windows and the laser safety interlock mechanisms and associated electrical circuits. It is strongly recommended that the interlock circuit is tested prior to each use of the equipment. If any damage to the laser safety windows is observed or suspected, or the sample chamber door interlock switch is not functioning correctly, the equipment should be temporarily removed from service until the fault is rectified. For maintaining and inspecting the laser, the documentation supplied with the laser should be consulted. If in any doubt, contact the manufacturer, Applied Photonics Ltd (www.appliedphotonics.co.uk), for further advice on maintenance and inspection of their LIBS products.

9. Shipping and storage

The LIBS equipment should be kept in a clean, dry environment which is free from extremes of temperature. The equipment contains sensitive optical and electro-optical components and so should be protected from excessive vibration or shock. During transport, the equipment should be packed in such a way as to prevent damage from shock or vibration and protected from ingress of dust. For shipping or storage of the laser, refer to the laser manufacturer's instructions for the correct procedure.
Note: Permanent damage to the laser can occur if it is subjected to temperatures of below 4 Celsius while it contains coolant water (due to freezing of the coolant). The coolant water MUST be completely drained from the laser head and the power supply if there is any possibility of the laser being subjected to temperatures below 4 Celsius (e.g. during shipping). Refer to the User's Manual for the laser for guidance on how to drain the coolant water.

10. Trouble-shooting / fault finding

Laser does not activate:

1. Are the safety interlock connections made correctly as described in section 3.8?
2. Is the sample chamber door fully closed? If a sample chamber is not fitted to the LIBS module and the equipment is required to be operated in "open-beam" (i.e. Class 4 Laser Product) mode, then the Interlock Override keyswitch on the SpectroModule-8 will need to be activated.
3. If the safety interlock appears to be working correctly but the laser still does not activate, refer to the operating and fault-finding instructions provided with the laser.

Laser-induced plasma appears to be adequately intense although a poor signal is observed on some or all of the spectrometer channels:

1. If using LIBSoft software, are the data acquisition settings on the software set up correctly?
2. Is the composition of the sample such that emission lines are not expected to be seen on some or all of the spectrometer channels? If yes, then use an alternative sample which is known to have numerous emission lines (e.g. zinc, tin, or an iron-containing material such as steel) to check for correct operation of the LIBS system.
3. Is the sample positioned correctly relative to the laser beam focus? It may be necessary to adjust the position of the aperture nozzle and/or the position of the focal plane of the laser beam.
4. Is the laser pulse energy too low? Increase if necessary.

Recorded spectra show some emission lines which are saturating the detector (i.e. a "flat top" appearance to the emission line):

1. Reduce the pulse energy of the laser using the controls on the front panel of the laser power supply (manual mode of operation) or via the LIBSoft software (software control mode of operation).

2. Increase spot size of laser beam on sample by adjusting the position of the sample relative to the focal plane of the laser beam.
3. Increase the integration delay setting using the relevant menu in LIBSoft.

Recorded spectra show some emission lines suffering from "self-reversal" (i.e. a "dip" in the centre of the emission line):

1. Reduce the pulse energy of the laser.
2. Increase spot size of laser beam on sample by adjusting the position of the sample surface relative to the focal plane of the laser beam (it is usual to set focus to be approximately 1 to 3 millimetres "into" the sample surface)
3. Increase the integration delay setting using the relevant menu in LIBSoft.

Air-breakdown is observed in the path of the laser beam just in front of the sample surface:

1. The position of the sample and or the focal plane of the laser beam is set incorrectly (ie. the focal plane is located in front of the sample) – adjust position of sample so that the focal plane of the laser beam is coincident, or preferably just "into", the sample surface.
2. On irradiation by the laser beam, the sample is creating considerable quantities of particulates in the path of the laser beam. Try cleaning the surface of the sample if loose material (eg oxide, surface contamination etc) is present. Reducing the laser pulse repetition rate and/or pulse energy and/or increasing laser beam spot size on the surface of the sample may also help. Gas purge using an inert gas such as argon will also help.

Appendix A1

SC-LQ2 liquids sample chamber with a Litron Nano SG 150-10 laser

WARNING

Never allow flammable liquids or gases to enter or accumulate inside the SC-LQ2 liquids sample chamber. Doing so would present a serious risk of explosion as the laser plasma is a source of ignition.

IMPORTANT

ALWAYS use an inert gas purge (air, nitrogen, argon, etc) when operating the liquids sample chamber, otherwise, vapour will accumulate inside the sample chamber which could lead to liquid droplets forming on the fused silica window which would compromise the optical transmission of both the laser beam and the plasma light.

1. Principle of operation

Problems of splashing of liquids during LIBS analysis are well-documented in the literature. The design of our SC-LQ2 liquids sample chamber avoids laser interaction with bulk liquid by presenting the liquid to the focussed laser beam as a thin surface film on a solid substrate. Our design uses a rotating wheel made of nickel plated 316 stainless steel (although other materials are available) since both materials are highly corrosion resistant and nickel also has a relatively high laser ablation threshold. This means that it is easier to generate a LIBS plasma on the nickel surface without significant ablation of the nickel, thereby minimising the contribution to the LIBS spectra of nickel. The rotating wheel is partly immersed in the liquid sample being analysed and slowly rotates (user adjustable rpm) so that the surface is continually wetted by the liquid so that each laser pulse is presented with fresh sample. Gas purge (air, nitrogen, argon) is used to spread the liquid thinly on the surface of the rotating wheel so that droplets cannot form – if they do there is an increased chance of splashing and contamination by liquid droplets of the inner surface of the fused silica window of the sample chamber. The rotating wheel has a 45 degree angled surface so that its position relative to the focal plane of the laser beam may be adjusted simply by moving the wheel in the horizontal plane to the left or to the right (as viewed from the front of the sample chamber). Moving the wheel to the right (increasing the reading on the micrometer) has the effect of lowering the surface of the wheel relative to the focal plane of the laser beam, whereas moving the wheel to the left (decreasing the reading on the micrometer) has the effect of raising the wheel surface relative to the focal plane of the laser. So horizontal movement of the wheel is effectively the same as moving the wheel surface (or solid sample in the case of a normal LIBS experiment) up and down relative to the vertical position of the laser focus. The operating principle of our SC-LQ2 liquids sample chamber has been shown to be quite effective but it does take some time and patience to optimise the experimental conditions, these being:

- Relative position of the rotating wheel
- Relative position of the laser focus position (focal plane of the laser beam)
- Laser pulse energy
- Rotational speed of the rotating wheel
- Laser pulse repetition rate
- Gas purge flow rate

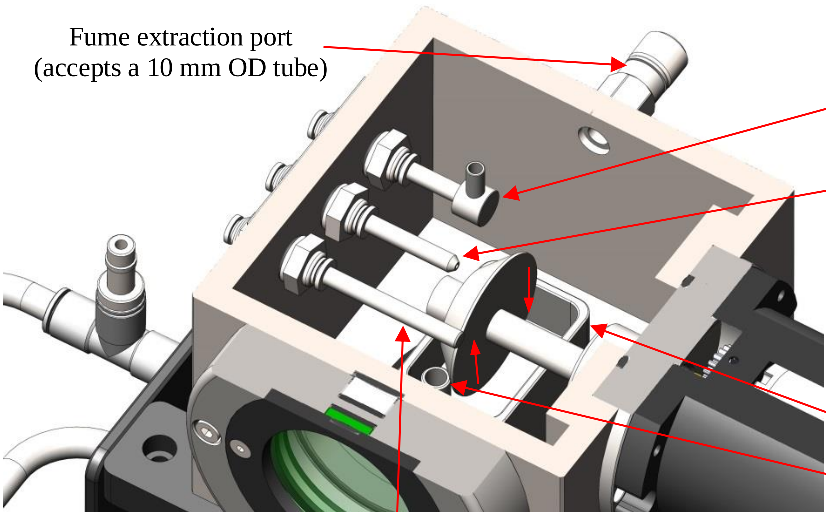
The SC-LQ2 does take some time to master but good analytical performance is possible as is shown in the example calibration discussed later in this appendix.

© 1998 - 2022 Applied Photonics Ltd

Note that the SC-LQ2 liquids sample chamber is designed for use with a 1064 nm pulsed laser. We have not yet investigated the use of other laser wavelengths (532 nm, 355 nm, 266 nm) although we do not expect any significant improvement in analytical performance when using these laser wavelengths.

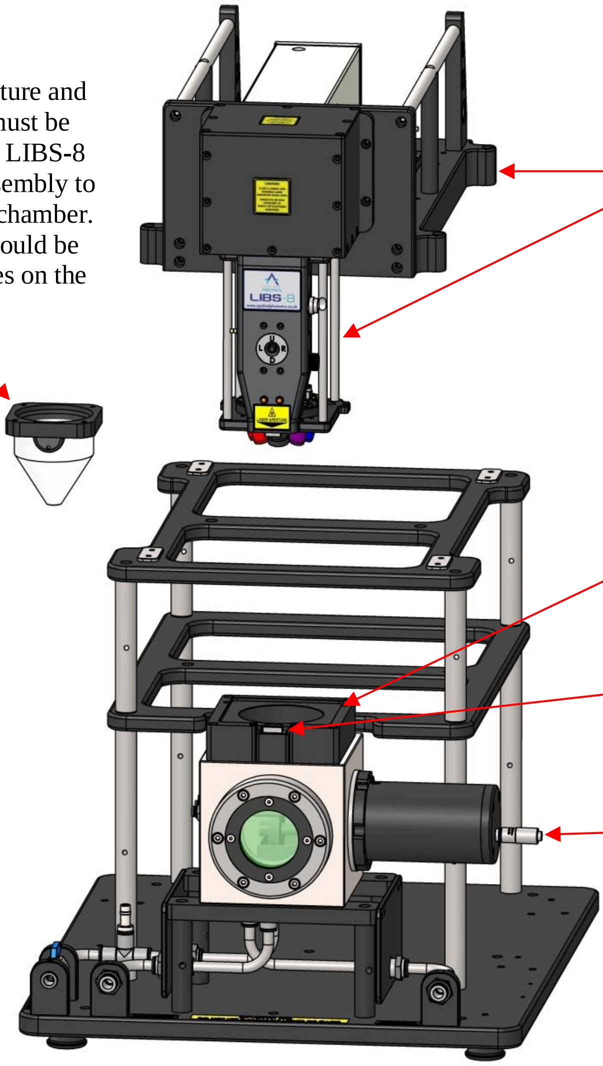
There are three gas purge ports on the left hand side of the SC-LQ2 liquids sample chamber, as shown in the following image.

The following CAD cut-away view shows the three gas purge tubes inside the sample chamber together with a description of their function.

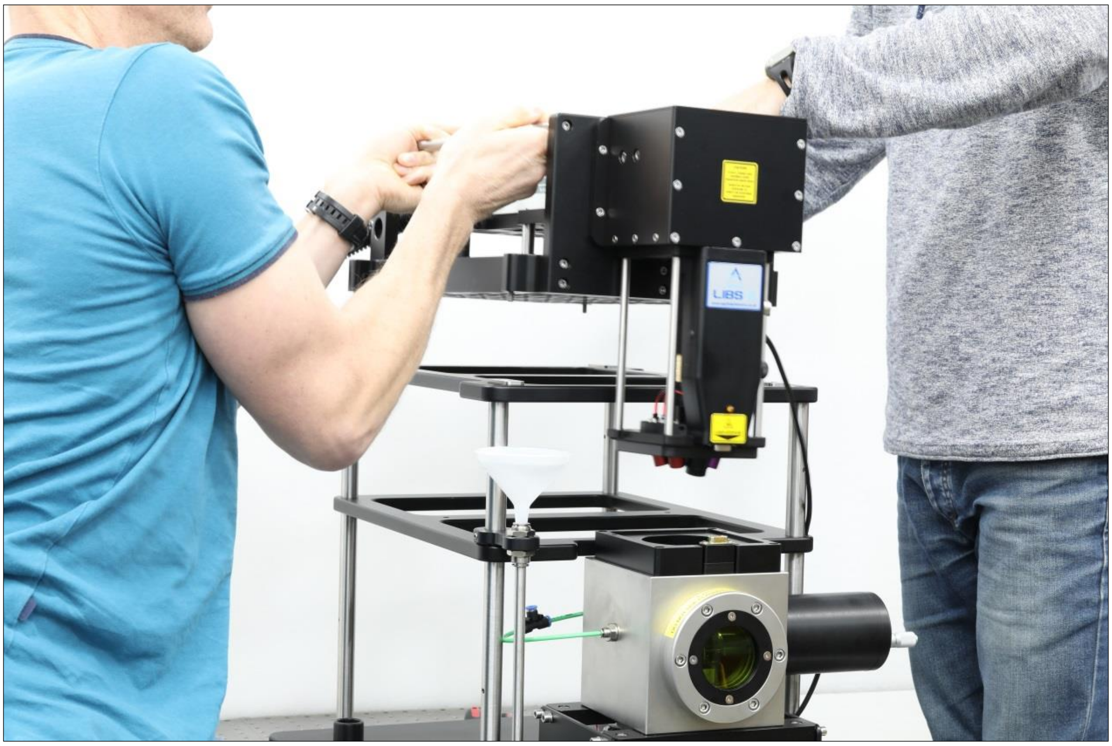


2. Setting up the SC-LQ2 liquids sample chamber

The laser head, adaptor platform, mirror module and LIBS-6 / LIBS-8 module assembly are fitted to the SC-LQ2 liquids sample chamber, as illustrated in the following schematic diagram. Note that the transparent nozzle aperture and associated threaded ring must first be removed from the LIBS-6 / LIBS-8 module (four M4 cap head screws).



Four M6 cap head screws are used to attach the laser head adaptor platform to the support frame. The LIBS-6 / LIBS-8 module fits inside the recess of the black square section on top of the liquids sample chamber (take care to correctly align the 9-pin D-plug electrical connectors). Note that there are no fasteners to attach the LIBS-6 / LIBS-8 module to the black square section fitted to the top of the liquids sample chamber, as it is designed to interface via the electrical connector and recess only.

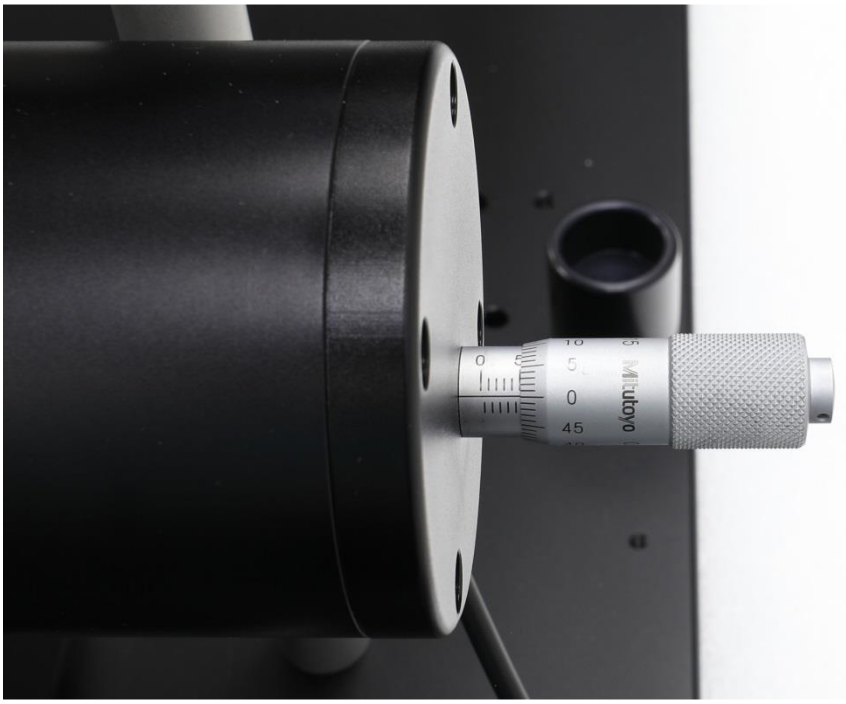


Two people are required to fit the laser adaptor platform / LIBS-6 module assembly to the support frame and liquids sample chamber. Care should be taken to correctly align the 9-pin D-type electrical connectors so as not to damage the pins.

3. **Setting the laser beam focus position**

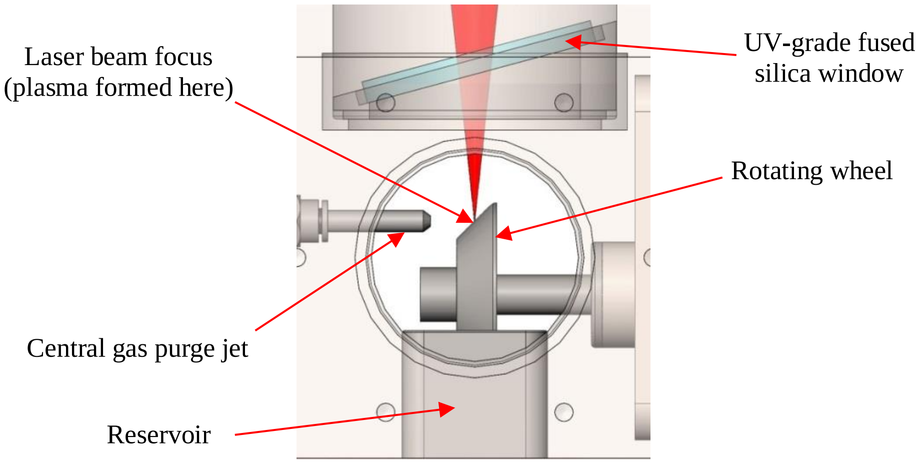
If the LIBS system has been previously set up for solids analysis, e.g. using one of our modular sample chambers such as SC-2L or XYZ-750 sample chambers, then the position of the laser beam focus may need to be adjusted (by rotating the brass beam expander tube in the LIBS-6 / LIBS-8 module). On initial use of the SC-LQ2 liquids sample chamber, minor adjustments of the laser beam focus position may be necessary to optimise the quality of the recorded LIBS spectra. We strongly recommend that you take a note of the adjustments you may make, so that the laser beam focus can be reset to its factory set position if required.

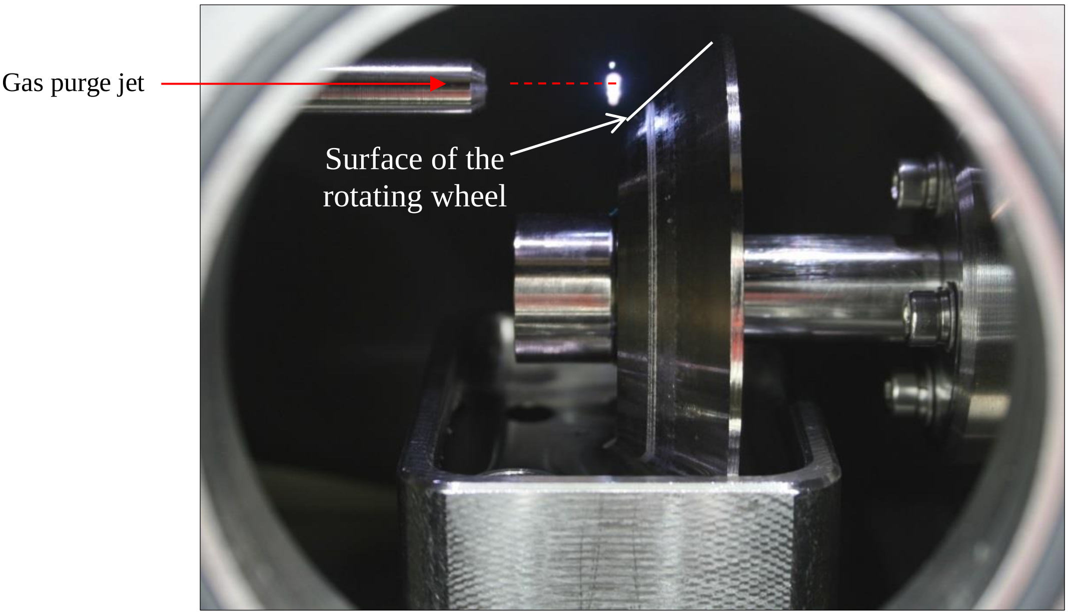
It may also be necessary to adjust the position of the rotating wheel in the sample chamber using the micrometer located on the right-hand side of the geared motor housing. The micrometer position, however, was set at the optimum position during factory testing of the equipment so we recommend that it is adjusted only if absolutely necessary. The factory setting for the micrometer is 5.00, as shown in the following image.

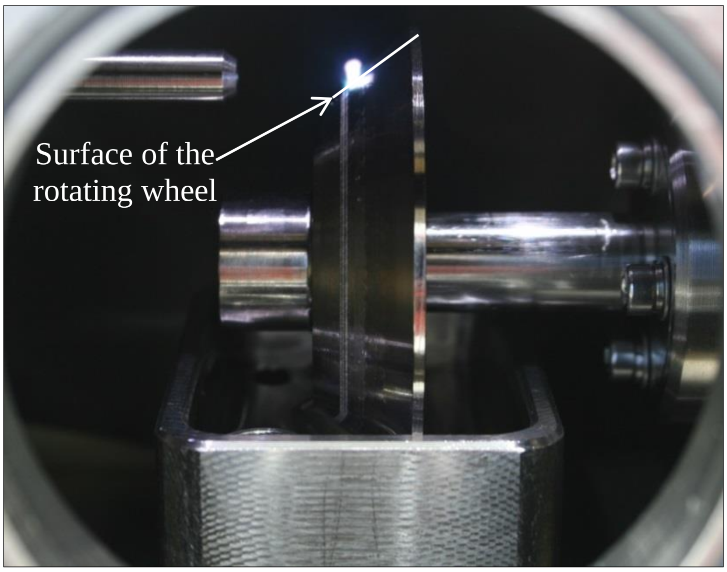


One easy way to check that the laser beam focus position is close to where it needs to be, is to move the rotating wheel to the right (by turning the micrometer to increase the reading) and fire the laser to produce a plasma in the air / gas, as shown in the image below. The position of the plasma should be in line with the central axis of the gas purge nozzle (see red dotted line in image below). When you are satisfied that the laser beam focus position is correct, the rotating wheel may be moved to the left so that the plasma is formed on the surface of the wheel, as shown in the second image (following page). It will be necessary to make further (but small) adjustments to the position of the rotating wheel, to optimise the quality of the recorded spectra. It is unlikely that the optimum setting for the micrometer will be more than 1 mm either side of the factory setting of 5.00 mm. When optimised, the recorded spectra should have strong lines of the elements of interest in the liquid being analysed, but have relatively weak lines from the nickel plating of the wheel. If the application is such that the liquid being analysed contains nickel, and it is this element that needs to be detected, it will be necessary to use an alternative wheel – contact Applied Photonics Ltd for advice on this.

© 1998 - 2022 Applied Photonics Ltd


Laser beam focus
(plasma formed here)
UV-grade fused
silica window
Rotating wheel
Central gas purge jet
Reservoir


Gas purge jet
Surface of the
rotating wheel

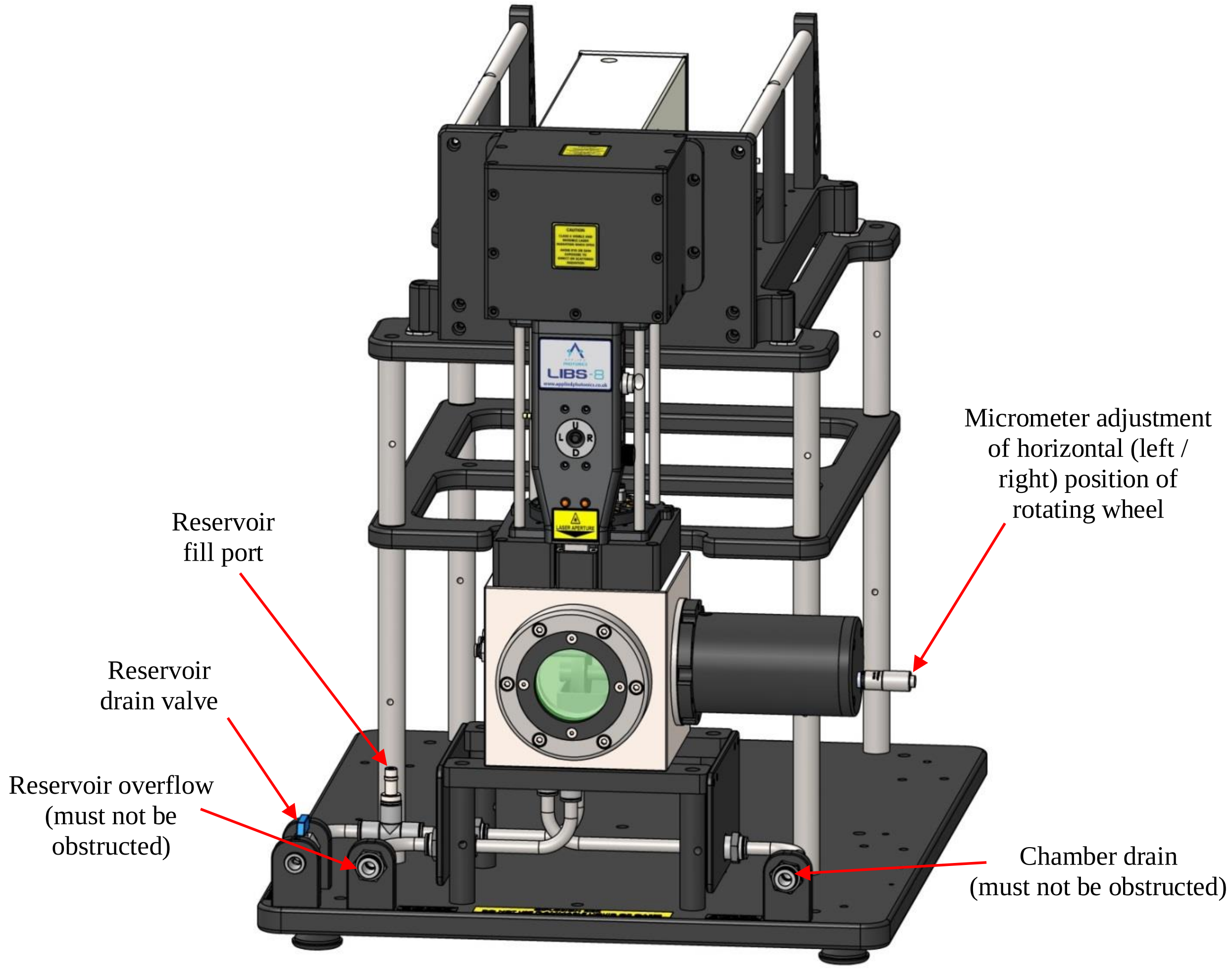

Surface of the
rotating wheel

4. How to fill/drain the liquids reservoir inside the SC-LQ2 sample chamber

The SC-LQ2 liquids sample chamber may be operated with either a fixed volume of liquid, or as a "flow through" system where the liquid sample is continuously pumped into the sample chamber and drains via the Reservoir Overflow port. In the following section, the liquids sample chamber is being used in the "fixed volume of liquid" mode.

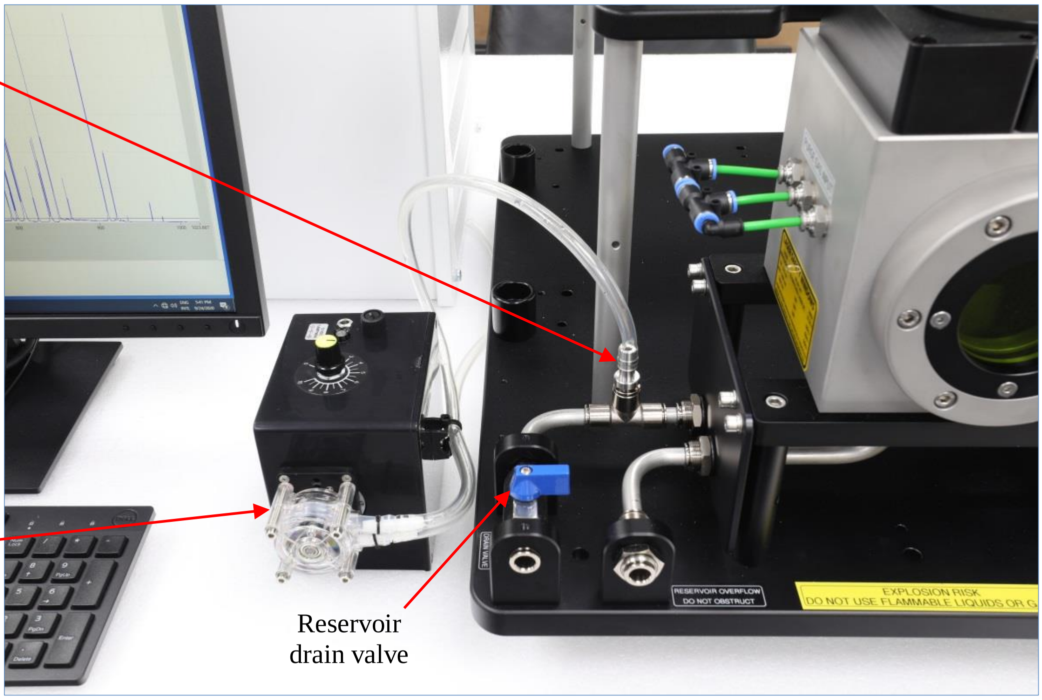
At the bottom of the SC-LQ2 there are three pipe connections. One pipe is used to fill/drain the liquids reservoir with the liquid for analysis. In order to fill the reservoir, the liquid is pumped into the Reservoir Fill Port using the supplied peristaltic pump, as illustrated in the following schematic diagram and image. The connectors used in the three ports are of the "push-fit" type and suitable for 10 mm outside diameter tubing.

The reservoir inside the liquids sample chamber is designed to ensure that the rotating wheel is continually wetted with liquid as the wheel rotates. The reservoir holds approx. 35 ml of liquid, but a larger volume of liquid will be required since the feed pipe etc will also need to be filled with liquid. The reservoir has an overflow which sets the level of the liquid and prevents overfilling of the reservoir. Liquid sample is slowly pumped into the Reservoir Fill port until the liquid starts to flow out of the Reservoir Overflow port. The reservoir is now filled to the required level. Should liquid be pumped into the sample chamber too quickly, it is possible that the reservoir may overspill into the void inside the liquids sample chamber and starts to escape via the Chamber Drain port. This in itself is not a problem but indicates that too much liquid has entered the sample chamber. The level of liquid in the reservoir will return to the correct level after the excess liquid has drained away and so your experiments may continue.

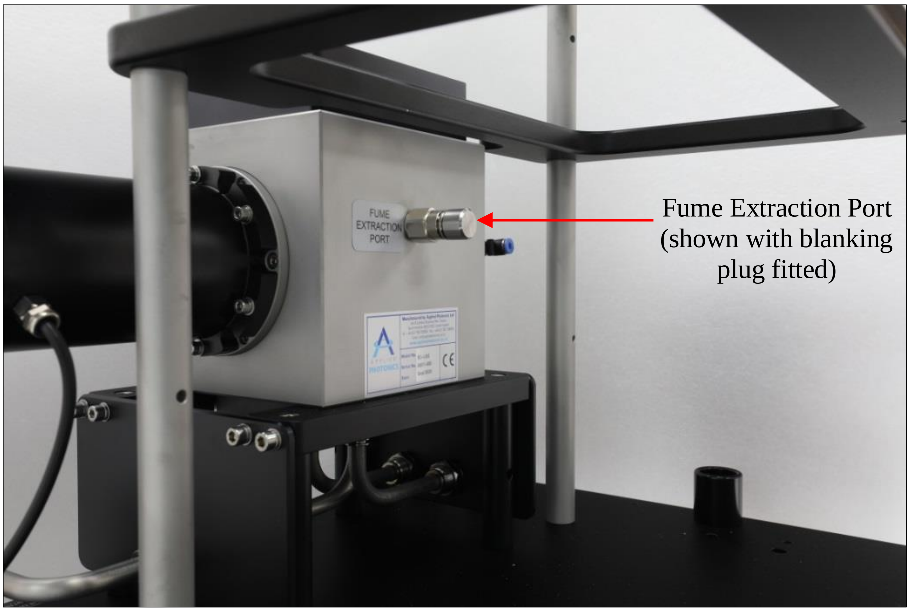


IMPORTANT

Neither the Reservoir Overflow port, or the Chamber Drain port should be obstructed at any time. They must be maintained in an open state so that both liquid <u>and</u> gas may escape from the liquids sample chamber. This means that any tubing connected to these ports must terminate in air, and not be placed below the surface of a liquid (e.g. do not place the tube end in a container such that it will end up below the level of liquid in the container as this will inhibit the free escape of gases from the liquids sample chamber).



The reservoir drain valve should be closed prior to admitting liquid sample via the peristaltic pump. It is important that the Reservoir Overflow port and the Chamber Drain port are not obstructed in any way. If this cannot be assured, then the Fume Extraction Port located on the rear face of the sample chamber (see image below) should be connected to a suitable fume extraction device. The Fume Extraction Port is equipped with a push-fit pneumatic connector suitable for a 10 mm outside diameter tube.



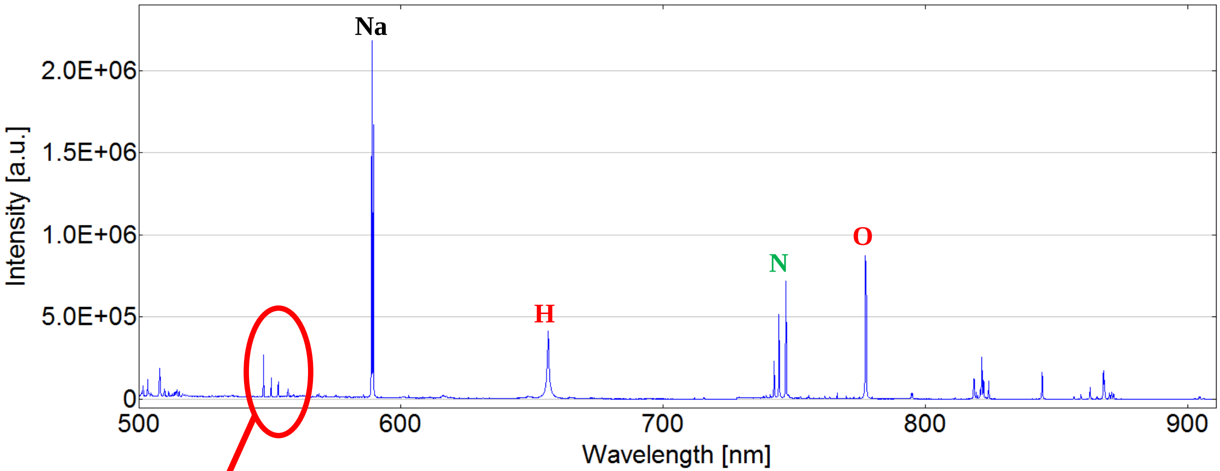
An example experiment – calibration curve for Mo in an aqueous solution of Na_2MoO_4

In order to demonstrate the capabilities of the SC-LQ2 liquids sample chamber, the LIBS system was tested using prepared aqueous solutions of varying concentrations of Na_2MoO_4 (0 ppm: distilled water, 1000, 5000 and 10000 ppm by mass of Mo). The reservoir inside the sample chamber was filled with each solution for it to be analysed. Between each solution analysis, the reservoir was rinsed with distilled water by filling and draining at least three times.

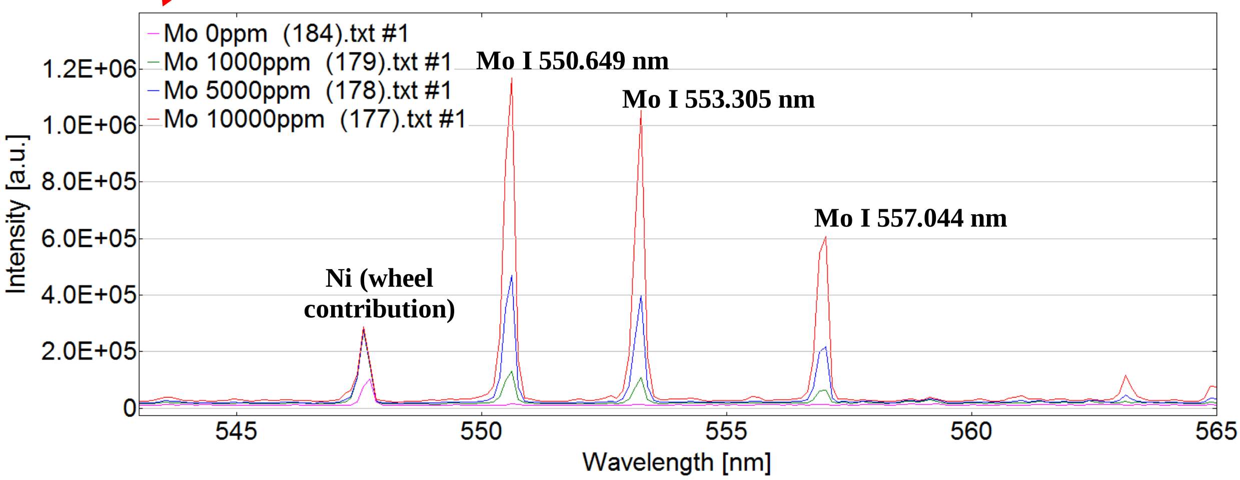
The spectra were acquired using the following experimental conditions:

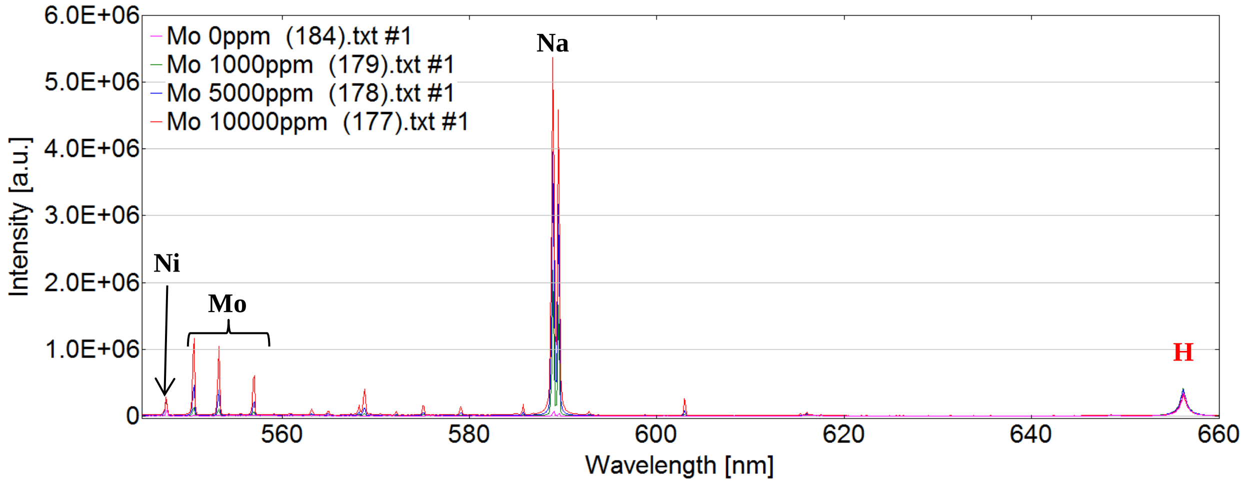
- 100 accumulation shots
- 10 measurements per sample
- 30 µs integration time
- 0.9 µs integration delay (minimum value) unless otherwise stated
- Laser energy at sample = approx. 100 mJ
- 10 Hz laser repetition rate
- Wheel speed approx. 6 rpm
- Air purge gas

LIBS spectrum of Na_2MoO_4 (1,000 ppm Mo) in distilled water (spectral range: 500 -915 nm, purge gas: Air)

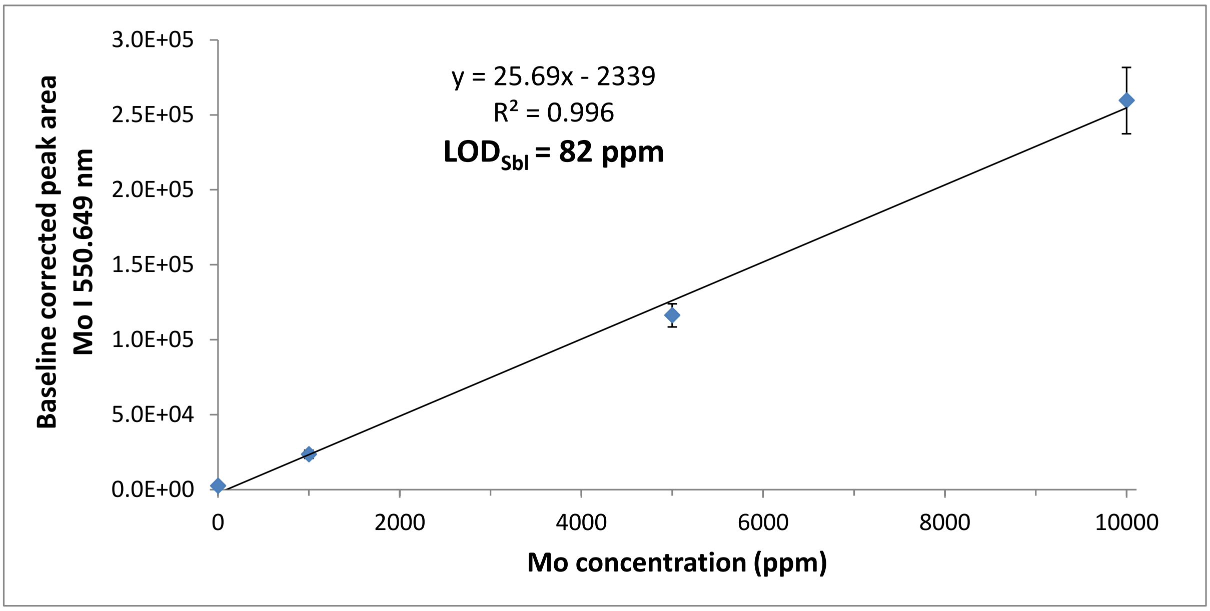


Mo detection in samples with different Na_2MoO_4 concentration
(pink: 0 ppm, green: 1,000 ppm, blue: 5,000 ppm and red: 10,000 ppm Mo)





Calibration curve of Mo in aqueous solution of Na_2MoO_4



Mo calibration curve data (10 replicate measurements per sample)

Mo concentration by mass (ppm)	Mean Mo baseline-corrected peak area Mo I 550.650 nm (arbitrary units)	SD	RSD (%)
10,000	2.60 E+05	2.2 E+04	8.5
5,000	1.16 E+05	7.8 E+03	6.7
1,000	2.34 E+04	2.7 E+03	11
0	2.51 E+03	7.0 E+02	28

The limit of detection (LOD) is defined as LOD = 3 s_{bl}/m, where s_{bl} is the standard deviation of the blank sample and m is the slope of the graph, the LOD was calculated to be 82 ppm.

© 1998 - 2022 Applied Photonics Ltd