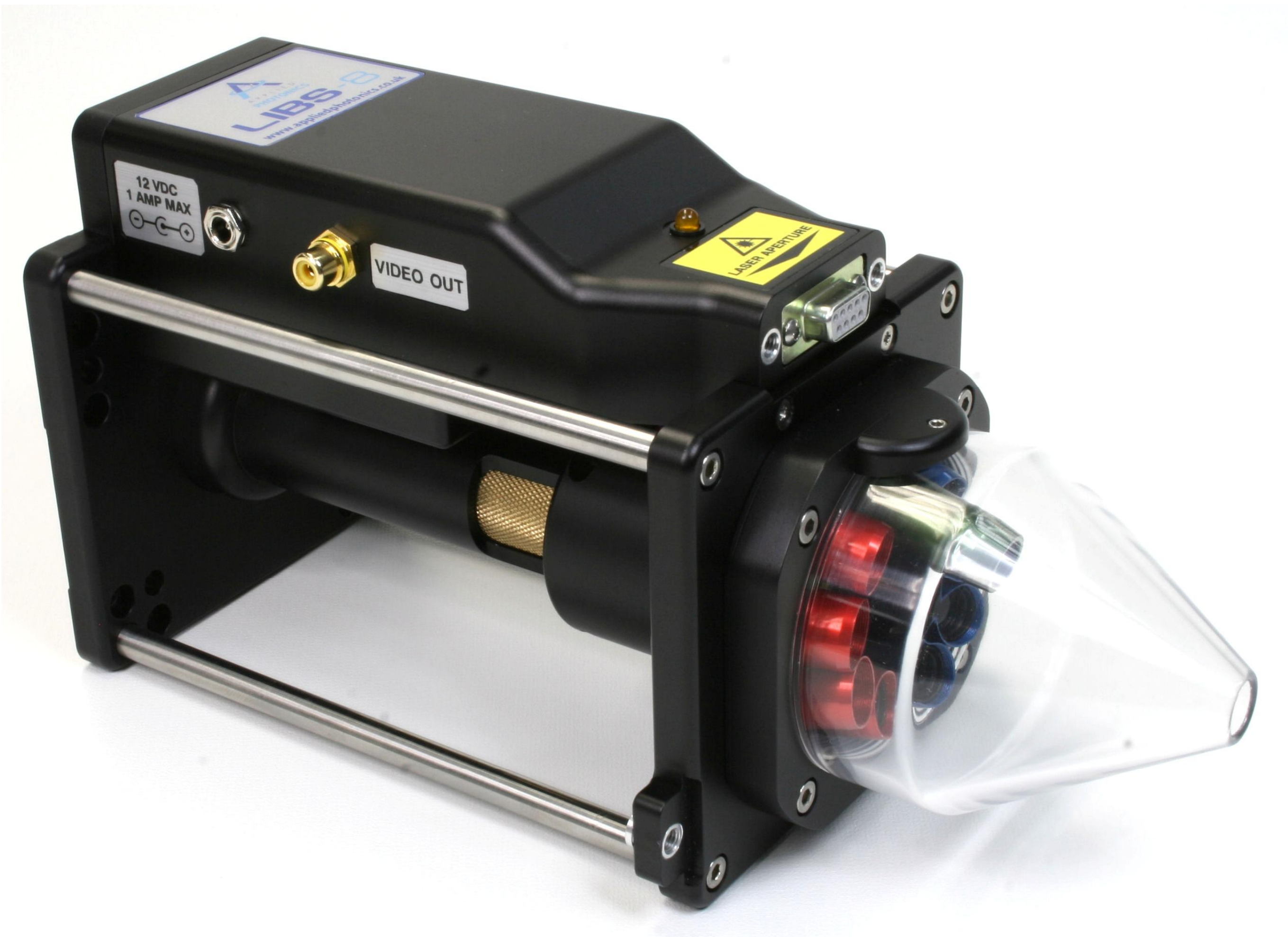
LIBS-6™ & LIBS-8™

Integrated LIBS Modules



User's Manual

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APPLIED
PHOTONICS

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1 Introduction

The LIBS-6 and LIBS-8 integrated LIBS modules are designed to be a highly versatile, adaptable and upgradeable product. 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 LIBS system tailored to suit the requirements of your specific application or experimental research project. The LIBS modules remove the need to design and construct your own laser focussing optics and plasma light collection optics by combining these features into a compact, integrated and easy to use device.

This User’s Manual provides the necessary information needed to configure the integrated LIBS modules and how to use them safely. It is assumed that the user will be familiar with the safety issues arising from the use of high-power lasers and will have preferably been trained in the safe use of laser products.

Examples of typical configurations of the LIBS modules, including illustrations of how they may be used with various lasers and sample chambers, are given in Appendix A2.

2 Safety

2.1 Laser radiation

The LIBS-6 and LIBS-8 integrated LIBS modules are designed for use with 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 LIBS-6 and LIBS-8 integrated LIBS modules are designed to meet the laser safety requirements of the relevant European standards (BS EN 60825) and USA standards (ANSI Z136.1 – 2007). Although these products may be 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 modules without the sample chamber (ie. operation in “open beam” mode as may be required for certain types of experiment). Accordingly when fitted to an appropriate laser, it is necessary to consider the LIBS modules to be **Class 4 Laser Products** and so, by definition, the equipment poses a risk of personal injury (eye, skin injury) and 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 (eg. a laser laboratory). Other measures may also be necessary, as determined by appropriate and thorough assessment of the risks (ie. a risk assessment) conducted by the personnel responsible for the safe use of the laser equipment. Consult the 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 laser power supply via the “Interlock Override Unit” using the supplied lead. Note that the “Interlock Override Unit” is specific to the laser being used – contact Applied Photonics Ltd for further information on this. Removal of the sample chamber from the LIBS module, or disconnection of the Interlock In/Out lead, will also activate the interlock (ie. prevent activation of the laser). The key-switch operated Interlock Override Unit (see section 3.5 of this User’s Manual) allows the safety interlock feature to be disabled. It is necessary to use this override feature when operating the LIBS module without a sample chamber (ie. operating in “open beam” mode). In view of this, the LIBS modules **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 (ie. “open beam” mode of operation)**.** If, however, the sample chamber is correctly fitted to the LIBS module and the Interlock Override Unit 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 integrated LIBS module (LIBS-6 or LIBS-8) and associated laser 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.
- NEVER allow unauthorised and/or untrained persons operate the LIBS module and associated laser equipment.
- Only suitably qualified and authorised persons should activate the Interlock Override Unit (IOU). The key should be removed from the IOU and held by the Laser Safety Officer when this feature is not required.
- ALWAYS use appropriate laser safety protective eyewear when operating the LIBS modules and associated laser 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 and remove the key from the keyswitch of the laser power supply to prevent unauthorised activation.

- NEVER operate the LIBS module and associated laser equipment in areas where explosive gas mixtures may be present.
- NEVER operate the LIBS module and associated laser equipment with any access cover 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.
- NOTE that neither the opaque black Delrin nozzle aperture nor the transparent acrylic nozzle aperture 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.
- ALWAYS thoroughly inspect the LIBS module and associated laser equipment for damage prior to use. Particular attention should be given to the laser safety windows and the electrical safety interlock fitted to the door of the sample chamber.
- NEVER point the LIBS module and associated laser equipment 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 deactivated <u>and</u> the safety shutter fitted to the laser head is switched to the CLOSED position**.

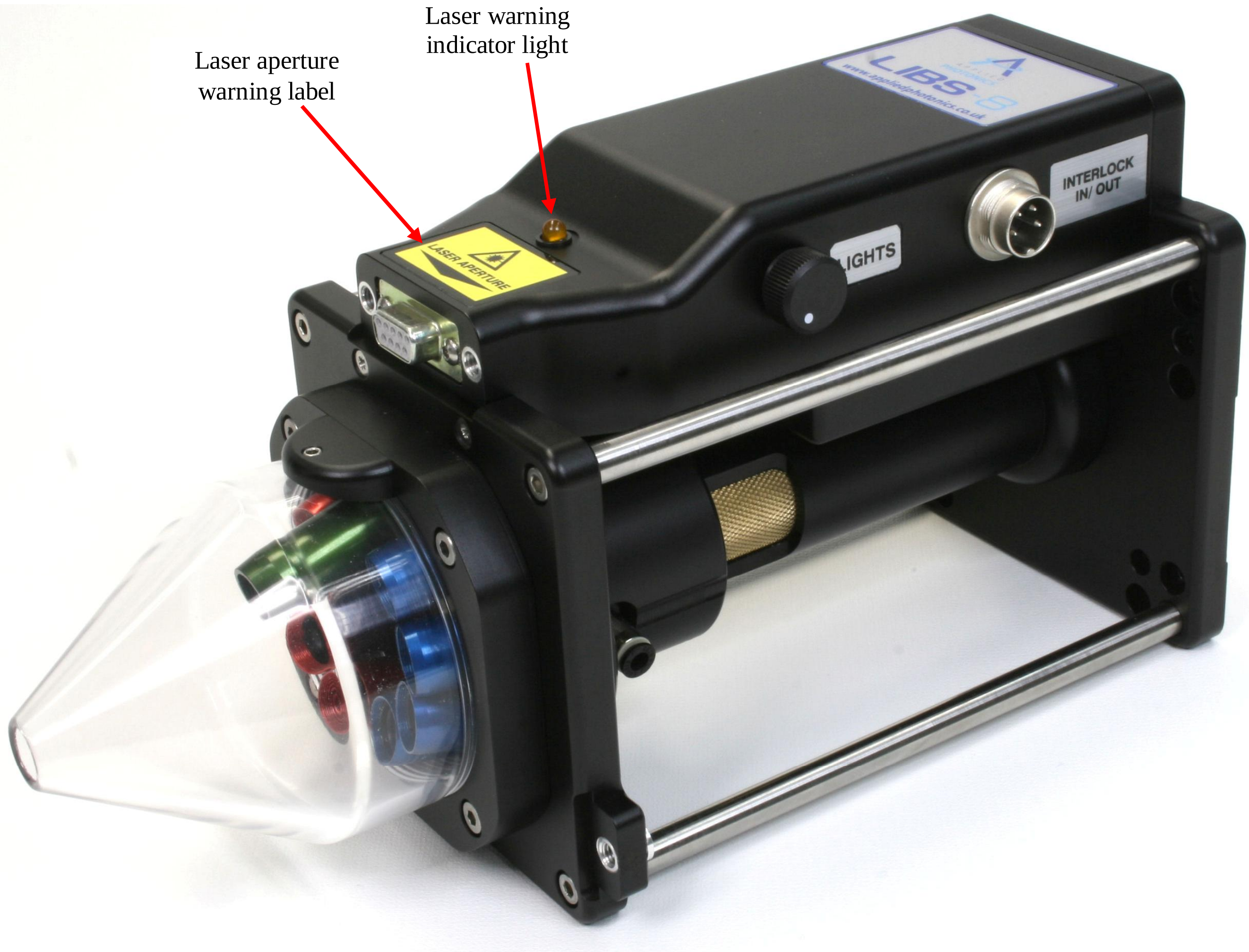


Image of LIBS-8 module showing location of warning indicator light and laser aperture warning label

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 – 2000**	**UK & European Standard BS EN 207:1999**
	Optical Density	**Protection Level R (Q-switched laser)**
1064	OD 6	L6
355	OD 5	L5
266	OD 3	L3

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 (ie. 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 (ie. typically less than 250 mJ with a 5 - 10 ns pulse length). 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.

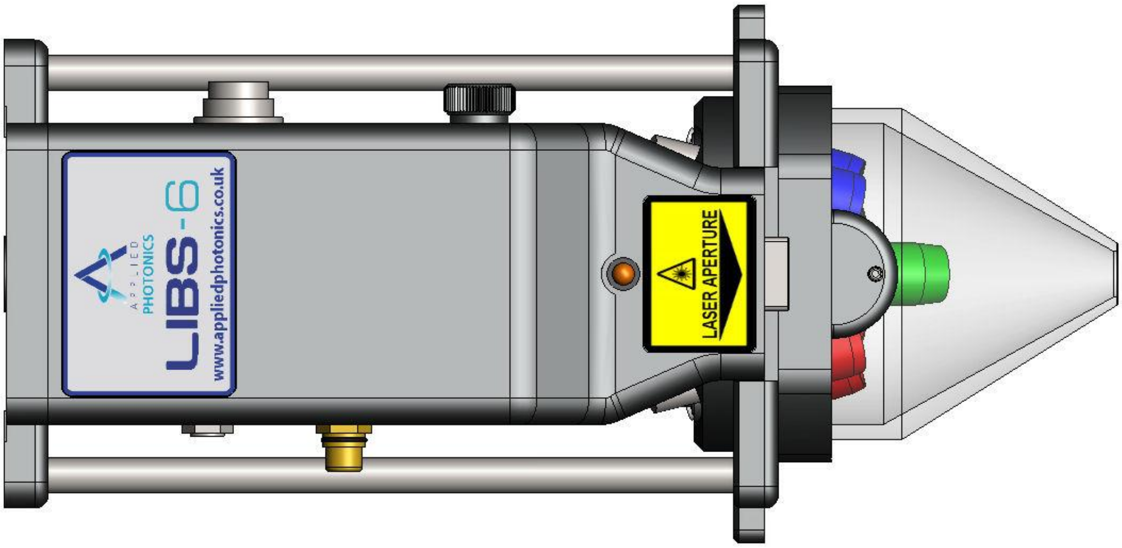
2.3 Electrical

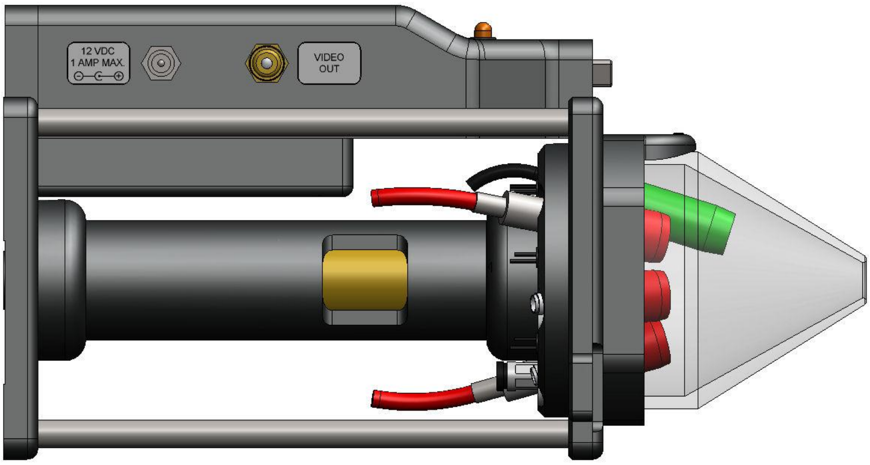
The LIBS-6 and LIBS-8 integrated LIBS modules contain electrical circuits operating at 12 VDC at a maximum current of 1 Amp. Accordingly, they pose no electric shock risk. The laser head and associated power supply, however, contain electrical circuits operating at potentially lethal voltage and current levels. Consult the manufacturer's User Manual supplied with the laser for further guidance on the safe use of the laser.

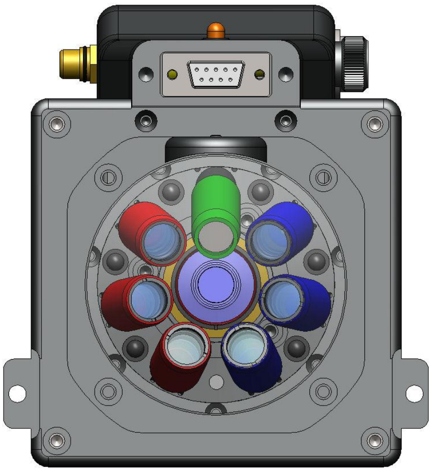
3 General description

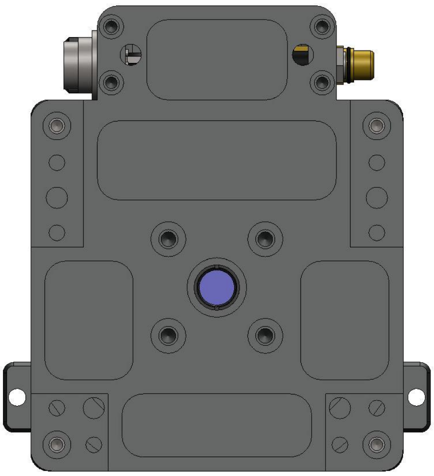
3.1 Overview

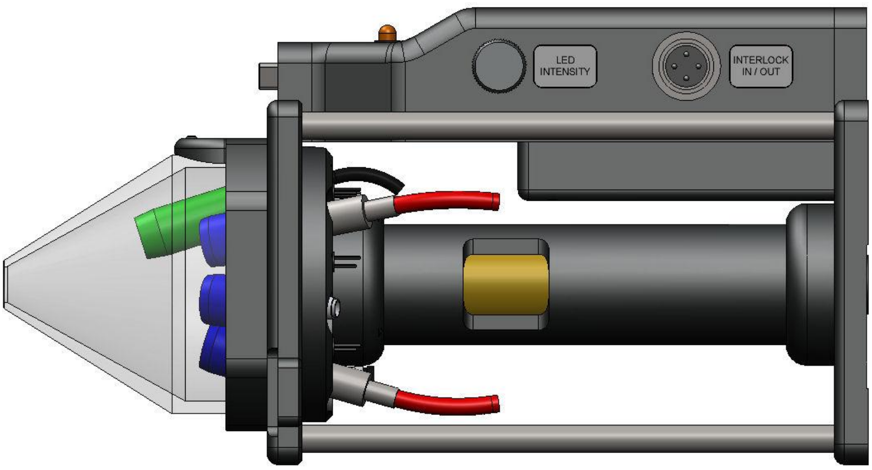
The main components of the LIBS-6 integrated LIBS module are illustrated in the following figures. The LIBS-8 module is essentially identical to the LIBS-6 module except that it has eight plasma light collection channels rather than six.

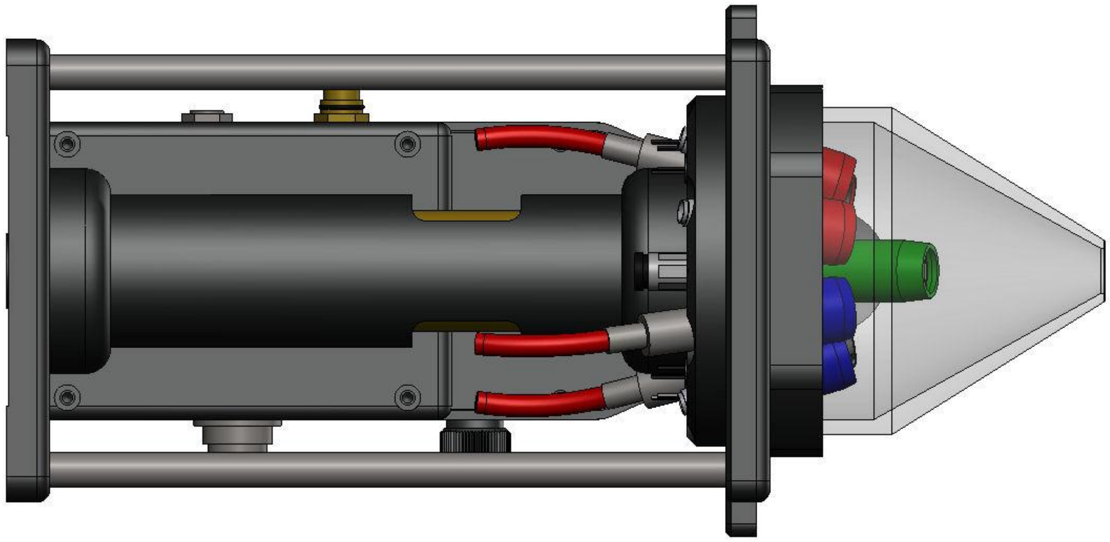


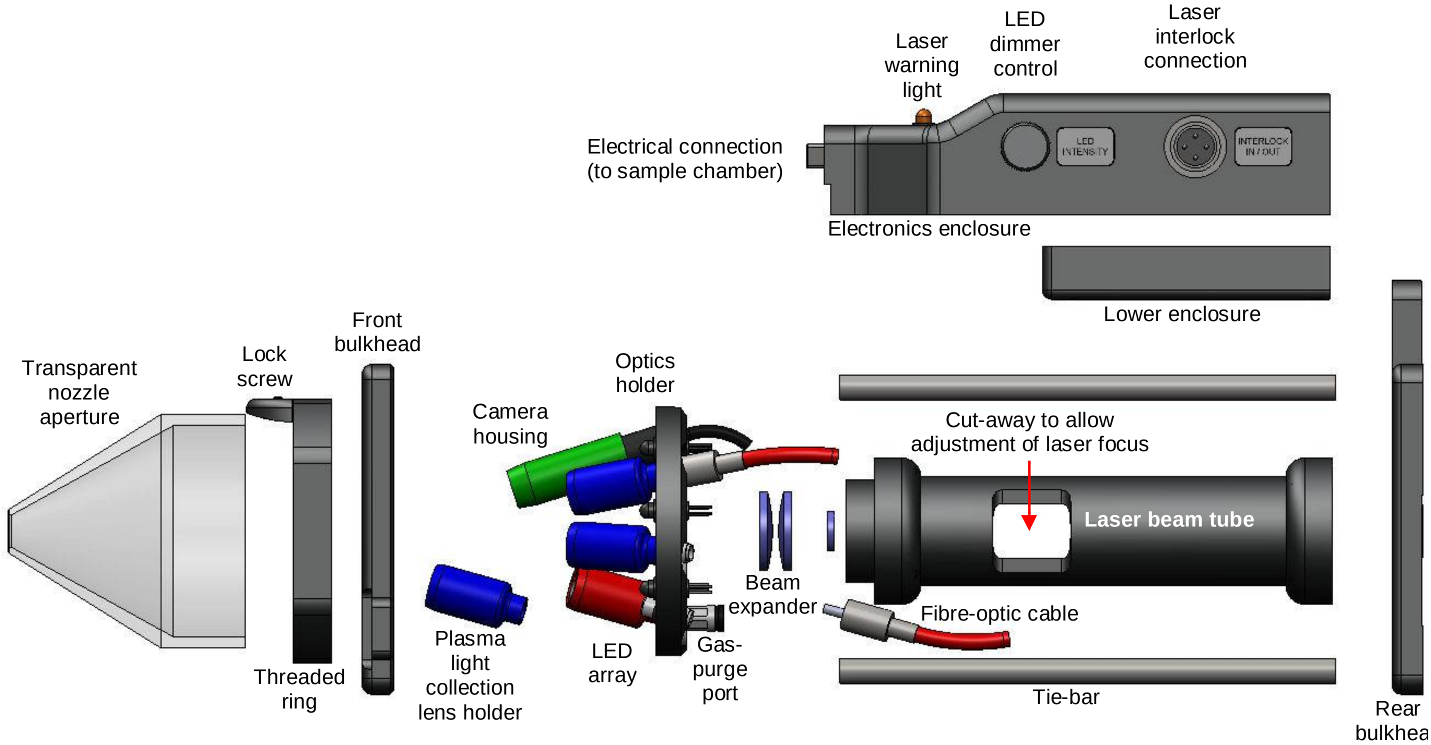






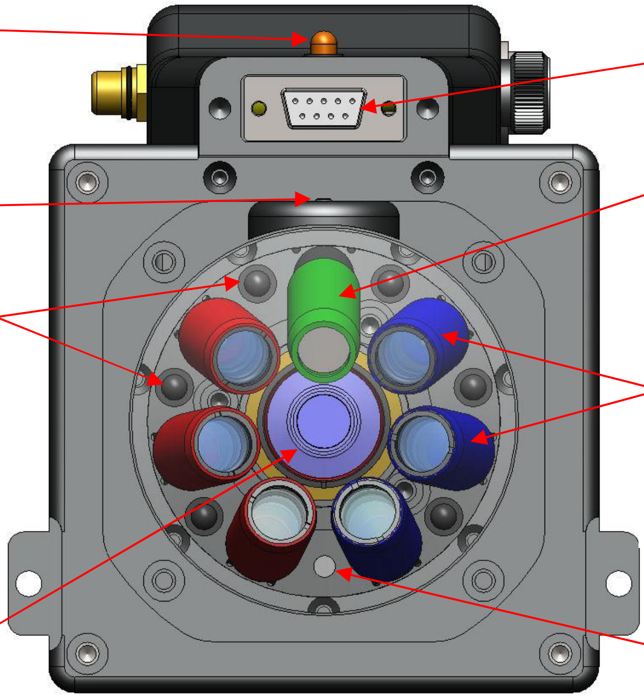






The LIBS modules provide the following features:

- Plasma light collection lens array having a relatively large depth-of-field to allow efficient light collection even when plasma position varies by ±5 mm along 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. 15 mm).
- Adjustable nozzle aperture which provides a convenient means of setting the distance to the sample surface (adjustment range of approx. 15 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 (eg. 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 CCD 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 Interlock Override Unit – not illustrated in the above figures).
- Compatible with the Applied Photonics Ltd range of modular sample chambers.

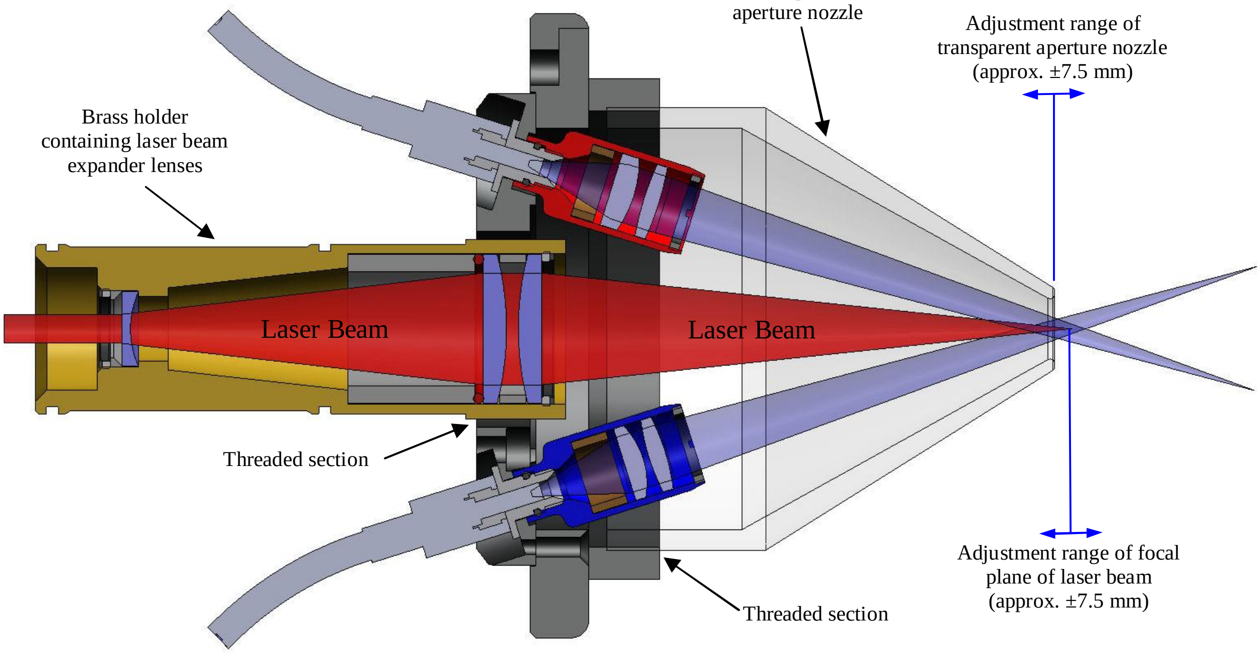


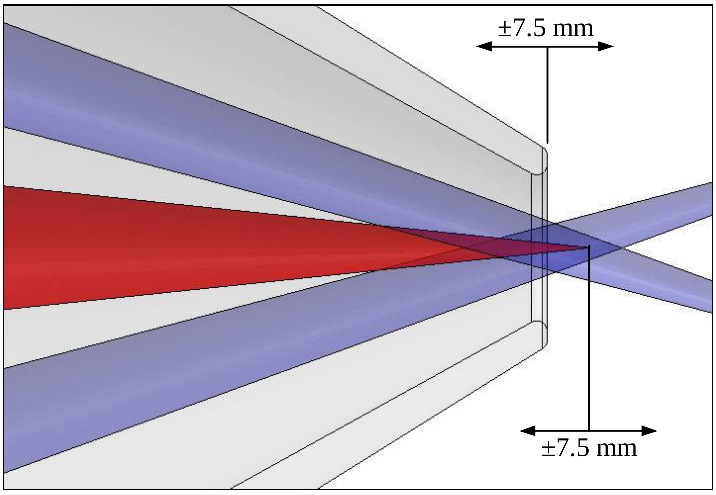
Front view of LIBS-6 module showing plasma light collection lens array, CCD camera, LED lights, laser aperture and gas purge outlet

The above diagrams illustrate the LIBS-6 module. Throughout the remainder of this User's Manual, for the purposes of simplicity, only the LIBS-8 module together with a Quantel Big Sky Ultra laser and either the SC-1 or the SC-2L modular sample chambers are described.

3.2 Laser beam expander and plasma light collection optics

The optical configuration used in the integrated LIBS 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 90 mm from the aperture of the beam expander (80 mm for the LIBS-6 beam expander). The optical design is specific to the make and model of laser which the LIBS 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 so that rotating it causes the focal plane of the laser beam to move. The design allows for approximately ±7.5 mm of adjustment, as illustrated in the following diagrams. The plasma light collection optics are angled at approx. 15.7 degrees (17.4 degrees for the LIBS-6 module) and are designed to collect light from the region in space defined in the following diagram. The transparent aperture nozzle is threaded so that rotation causes it to move along the optic axis of the laser beam – range of travel is approximately ±7.5 mm (it may be removed from the body of the LIBS module by unscrewing further). The main purpose of the transparent aperture nozzle is to provide a convenient means of setting the distance to the sample surface. The nozzle aperture also provides containment of the purge gas to ensure the sample surface and laser-induced plasma region are effectively purged of atmospheric air, and provides physical protection for the laser and plasma light collection optics (and imaging camera if fitted).





Close-up view of laser beam focus and field-of-view of plasma light collection optics

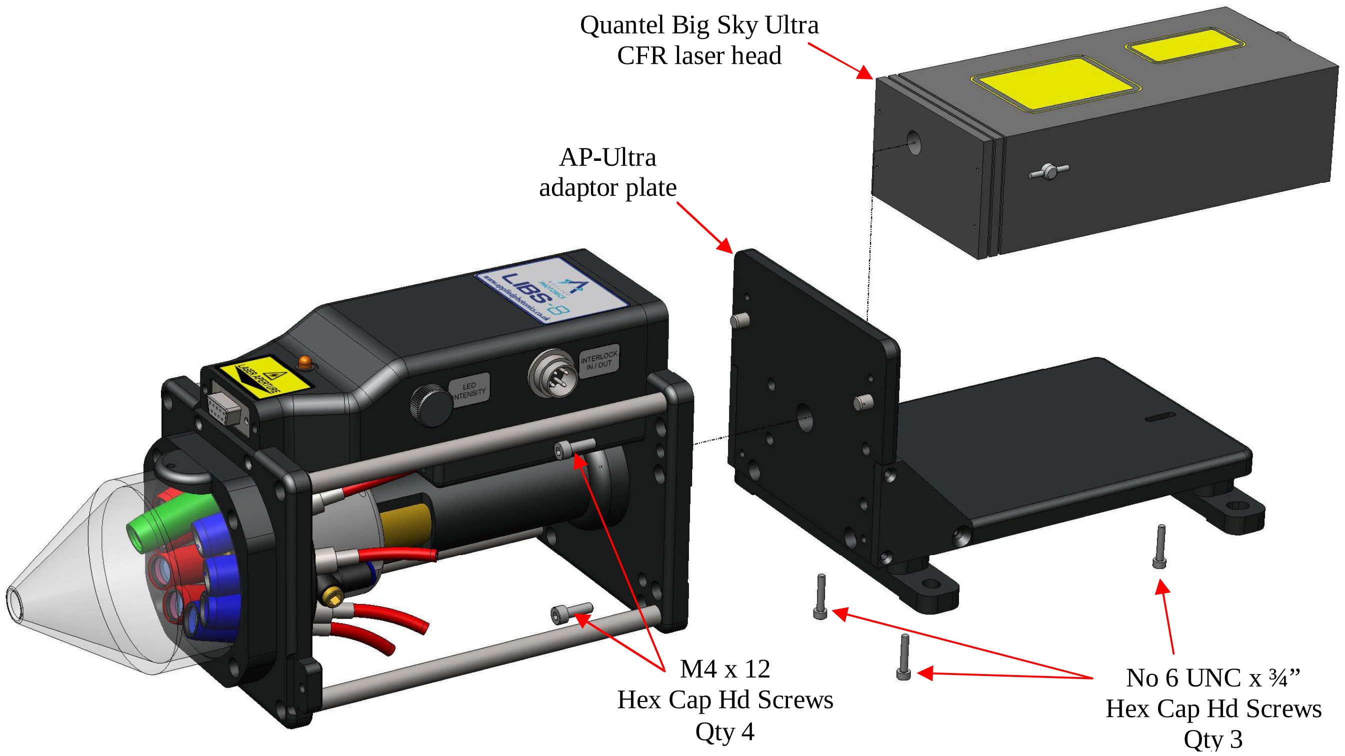
3.3 Attaching the LIBS module to a laser

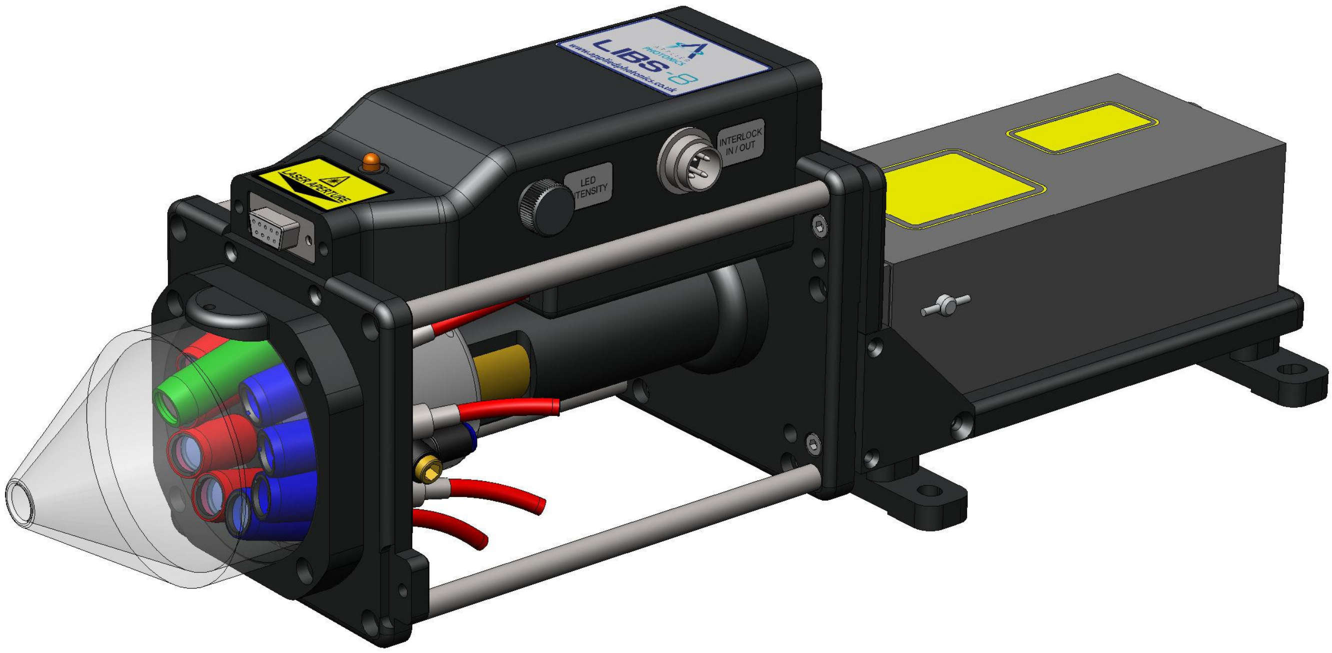
The LIBS modules are suitable for use with virtually any commercially-available Q-switched Nd:YAG laser which has operating characteristics appropriate for LIBS. In order to attach either the LIBS-6 or LIBS-8 module to a laser head, it is necessary to use an appropriate adaptor plate. At the time of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

Laser Product	Adaptor Plate
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

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

The method of assembly of the adaptor plate and laser head is illustrated in the following figures.

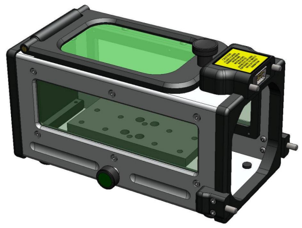
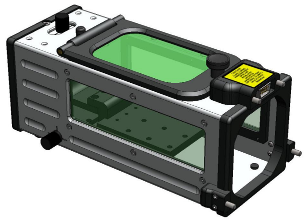
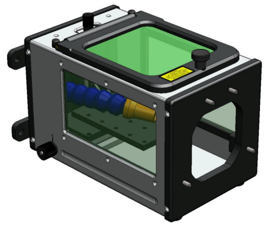
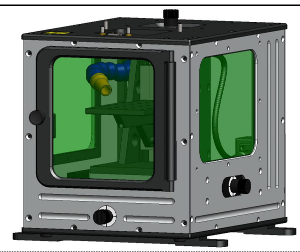
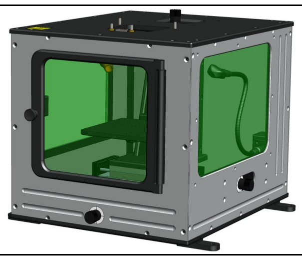
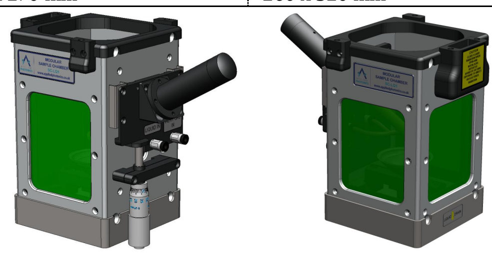




LIBS-8 module fitted to Quantel Big Sky Ultra laser head using AP-Ultra adaptor plate

3.4 Modular sample chambers

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. Two types (SC-2C and SC-2L) 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 200 mm	2-axis translation stage, 20 mm travel per stage (manual control). Approx. overall dimensions: 110 x 120 x 250 mm	2-axis translation stage, 20 mm travel per stage (manual control) Fume extract port. Internal LED light. Approx. overall dimensions: 170 x 170 x 270 mm	3-axis translation stage, 50 mm travel per stage (manual control). Fume extract port. Internal LED light. Approx. overall dimensions: 260 x 260 x 320 mm
			
SC-2XL	SC-3L	SC-LQ1	
3-axis translation stage, 50 mm travel per stage (manual control). Fume extract port. Internal LED light. Approx. overall dimensions: 475 x 370 x 450 mm	3-axis translation stage, 50 mm travel per stage (XY computer-controlled). Fume extract port. Internal LED light. Approx. overall dimensions: To Be Advised.	Modular sample chamber designed to analyse liquids. Requires inert gas and flow of liquid to be connected to chamber via two ports designed to accept 4 mm flexible tubing. Liquid drain port in base of chamber. Micrometer control of position of liquid surface relative to focal plane of laser beam. Approx. overall dimensions: 225 x 245 x 200 mm	

Current range of modular sample chambers manufactured by Applied Photonics Ltd

WARNING

The above range of modular sample chambers are 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 – 2000**	**UK & European Standard BS EN 207:1999**
	Optical Density	**Protection Level R (Q-switched laser)**
1064	OD 6	L6
355	OD 5	L5
266	OD 3	L3

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 (ie. a Q-switched laser).

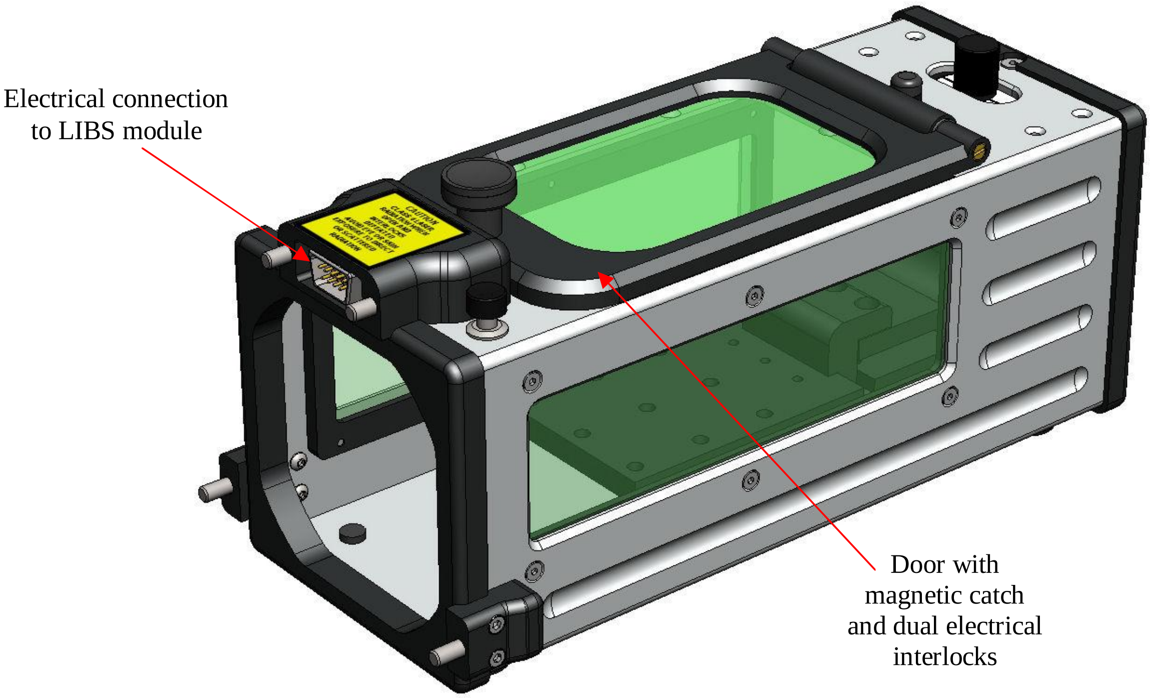
Since the LIBS-6 / LIBS-8 integrated LIBS modules may be used with various laser devices, it is the responsibility of the user to establish whether the protection offered by the laser safety windows is adequate for the laser being used. If in doubt, seek advice from a suitably qualified Laser Safety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

3.4.1 SC-2C modular sample chamber

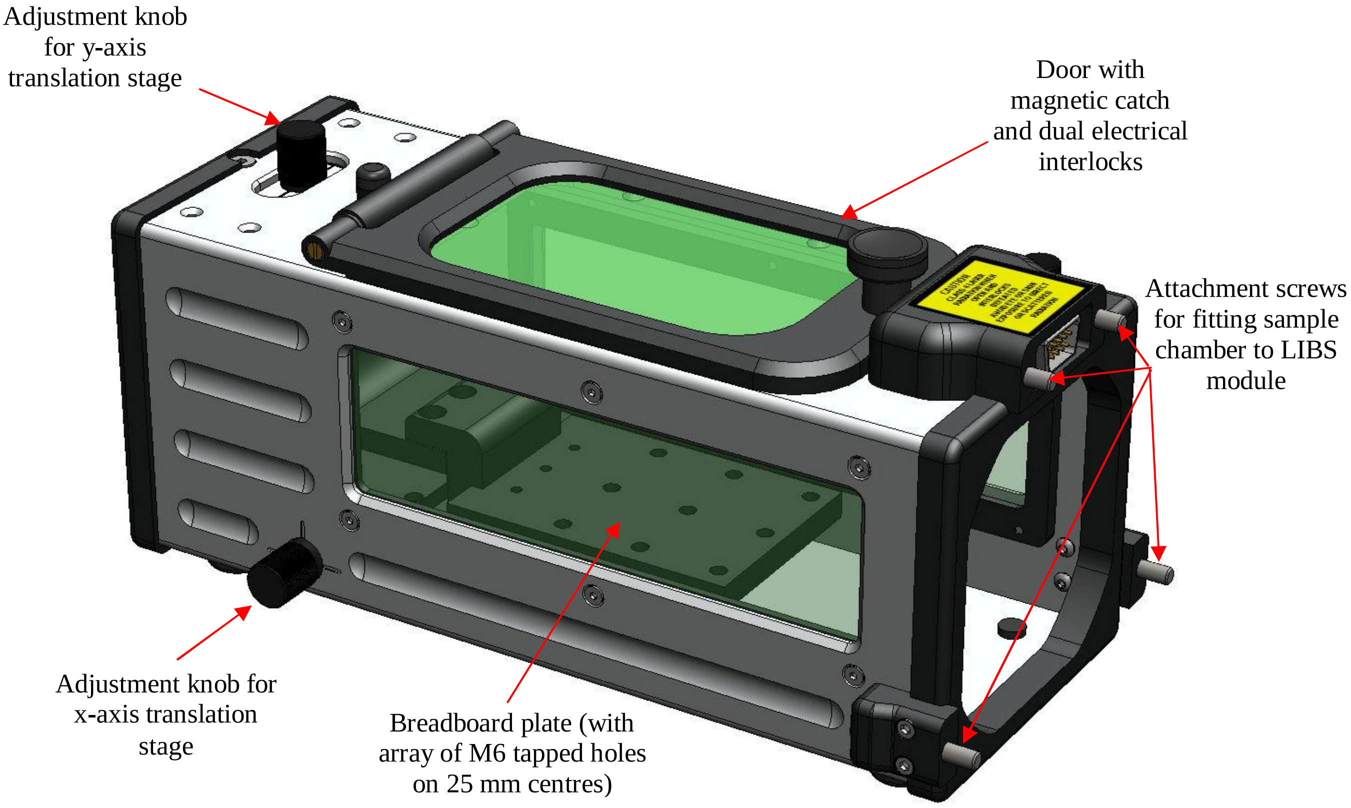
A general view of the SC-2C modular sample chamber is given in the following figure. The sample chamber is equipped with a breadboard plate which is attached to a manual two-axis translation stage (approx. 20 mm travel per stage). The breadboard plate has an array of M6 tapped (blind) holes on 25 mm centres and which may be used to facilitate the attachment of a sample holder etc. Movement of the breadboard is achieved by adjustment of the knobs on the side and top of the sample chamber, as illustrated below.

View of SC-2C modular sample chamber

The sample chamber is equipped with a number of features as illustrated in the following figures.

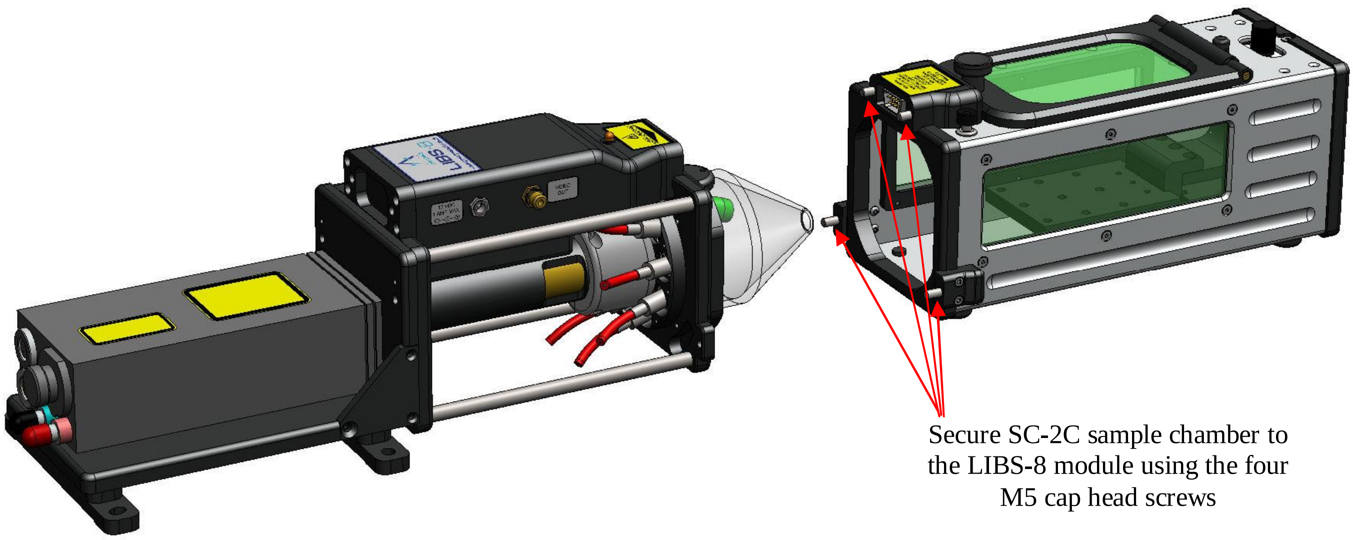


CAD view of SC-2C modular sample chamber

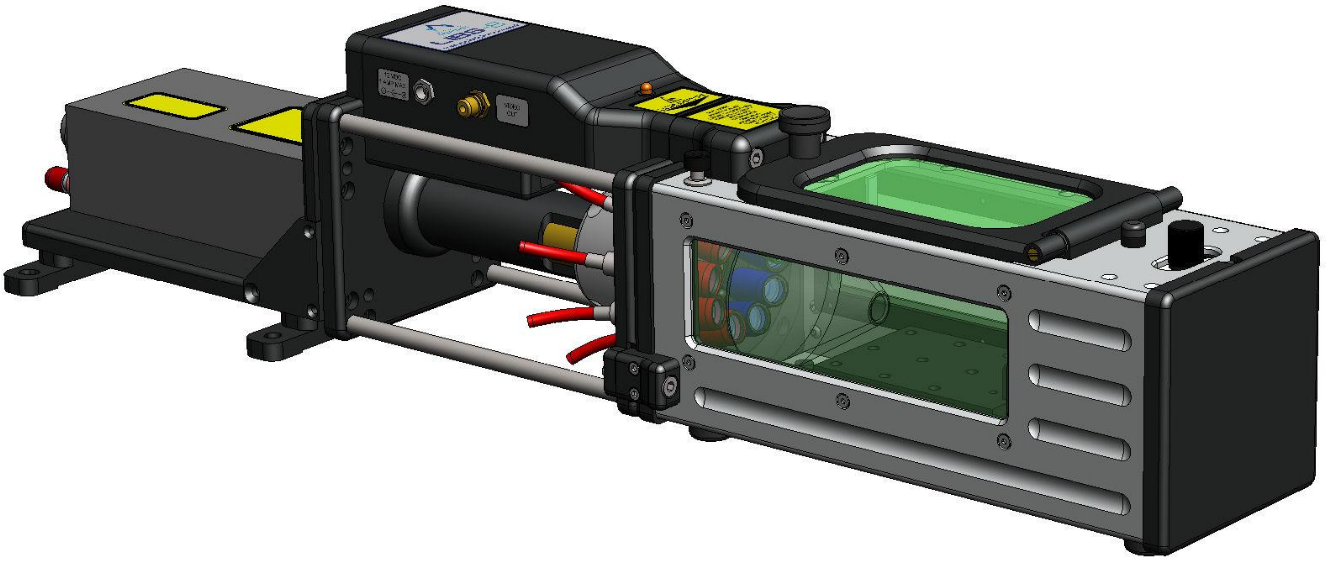


CAD view of SC-2C modular sample chamber

The SC-2C modular sample chamber is designed to fit to the LIBS module using four M5 screws as illustrated in the following figures. Before fitting the sample chamber to the LIBS module, ensure that the breadboard plate is positioned sufficiently low so as not to make contact with the aperture nozzle of the LIBS module (use the y-axis adjustment knob to lower the breadboard plate). Be careful to fit the sample chamber squarely to the LIBS module so as not to cause damage to the electrical connectors.



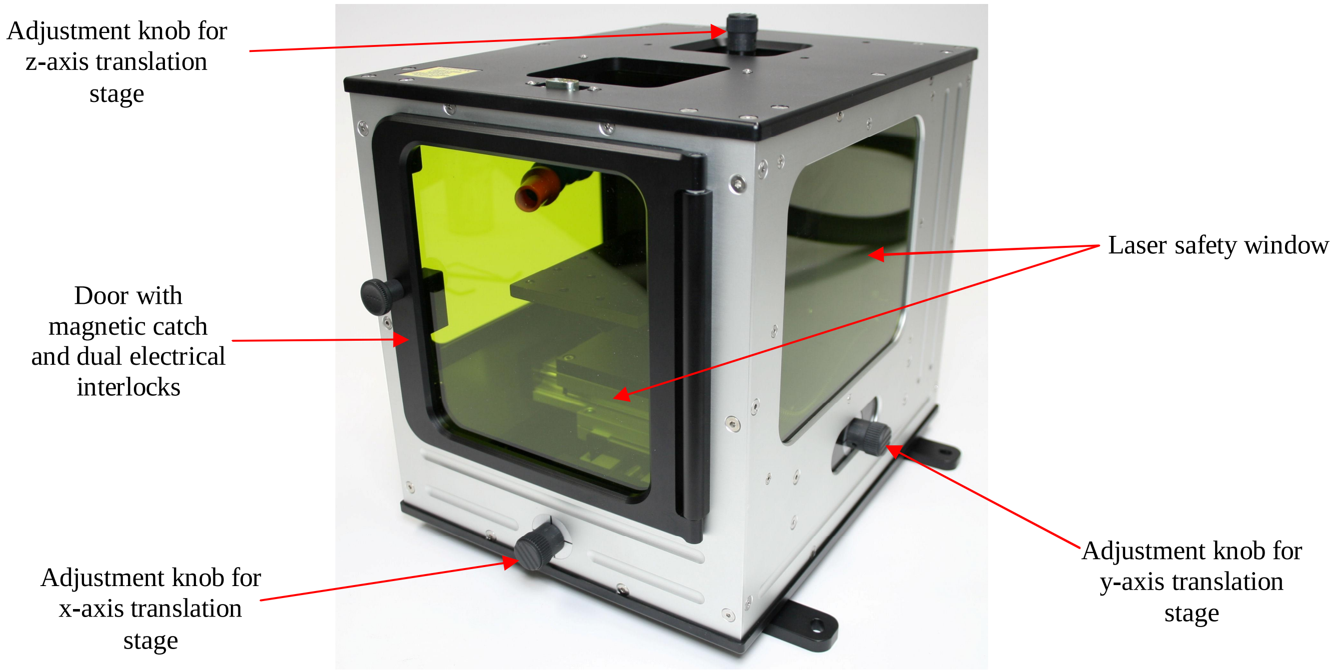
CAD views of SC-2C modular sample chamber illustrating method of attachment to LIBS-8 module



CAD views of SC-2C modular sample chamber attached to LIBS-8 module

3.4.2 SC-2L modular sample chamber

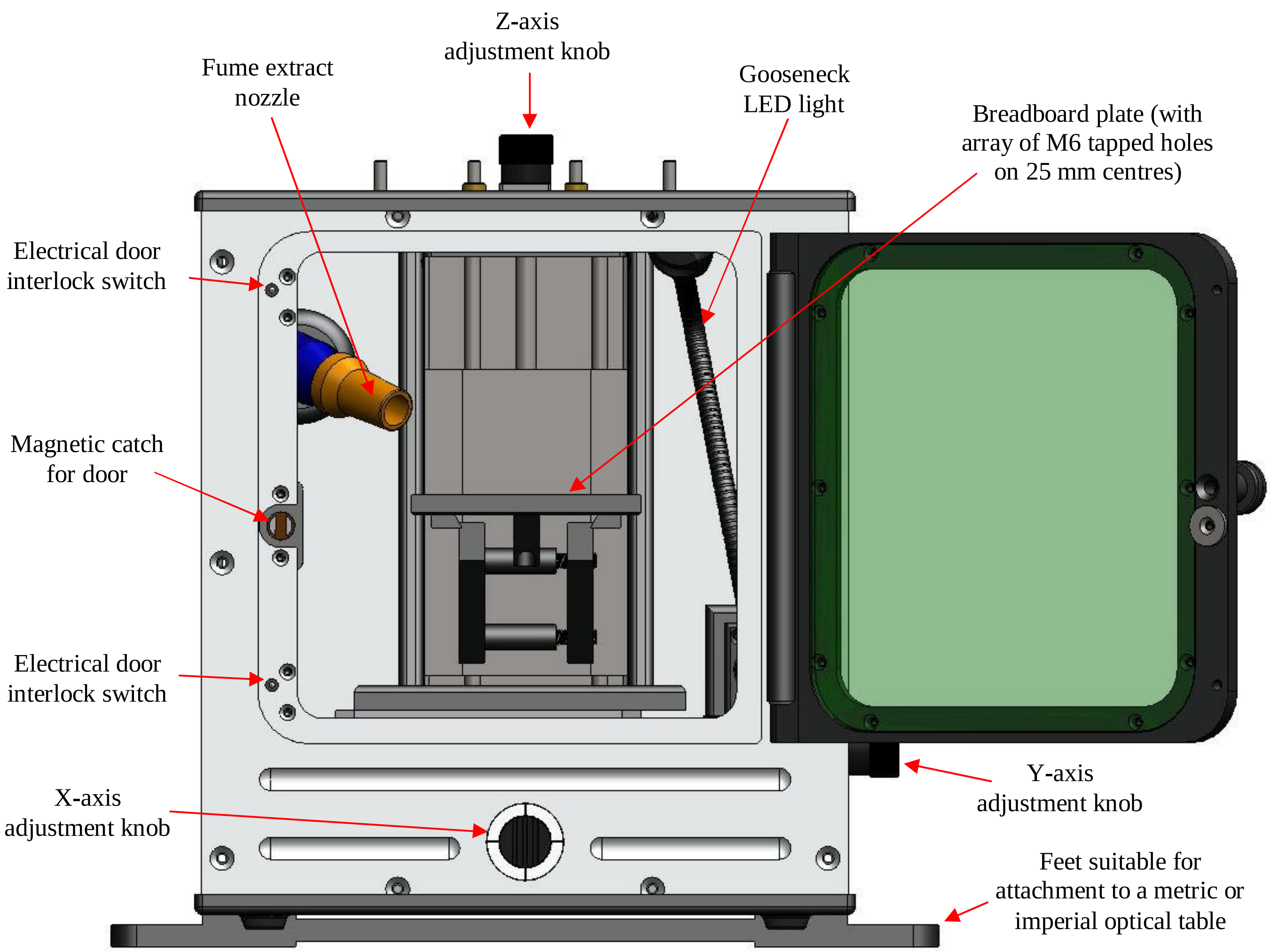
A general view of the SC-2L modular sample chamber is given in the following figure. The sample chamber is equipped with a breadboard plate which is attached to a manual three-axis translation stage (50 mm travel per stage). The breadboard plate has an array of M6 tapped (blind) holes on 25 mm centres and which may be used to facilitate the attachment of a sample holder etc. Movement of the breadboard is achieved by adjustment of the knobs on the sides and top of the sample chamber, as illustrated below.



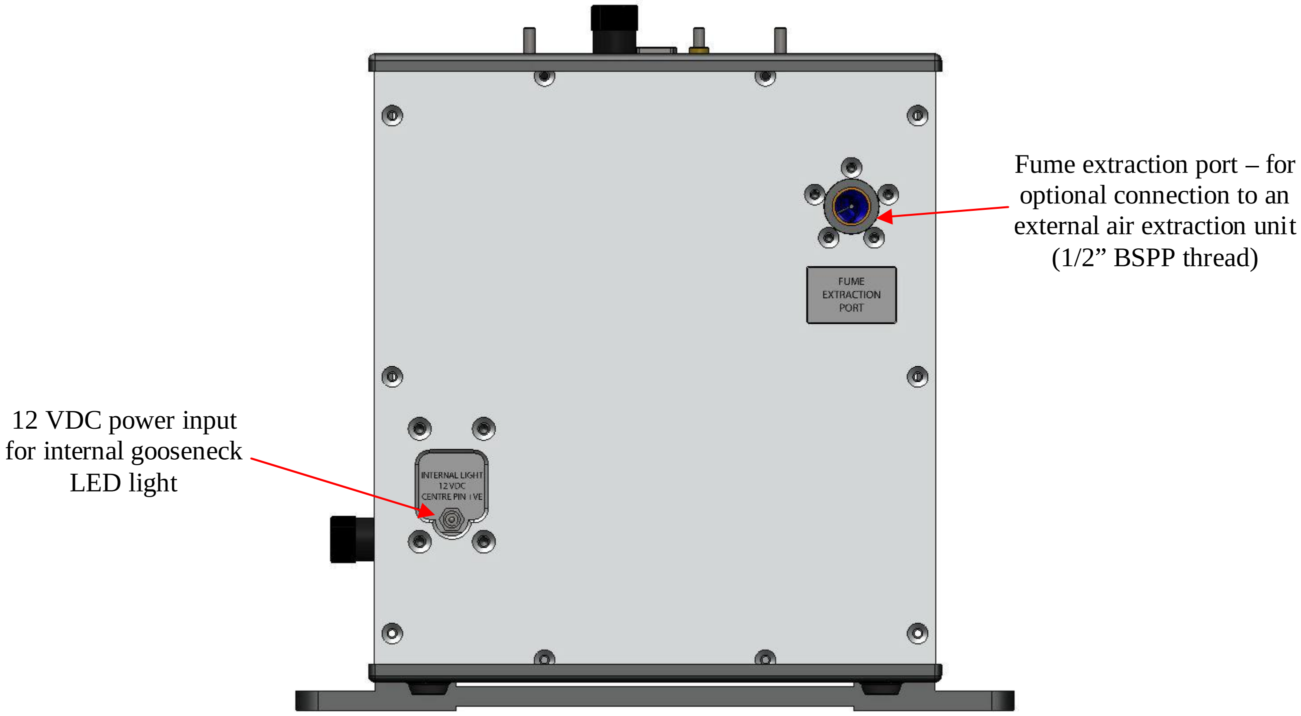
View of SC-2L modular sample chamber

The sample chamber is equipped with a number of features as illustrated in the following figures.

CAD views of SC-2L modular sample chamber

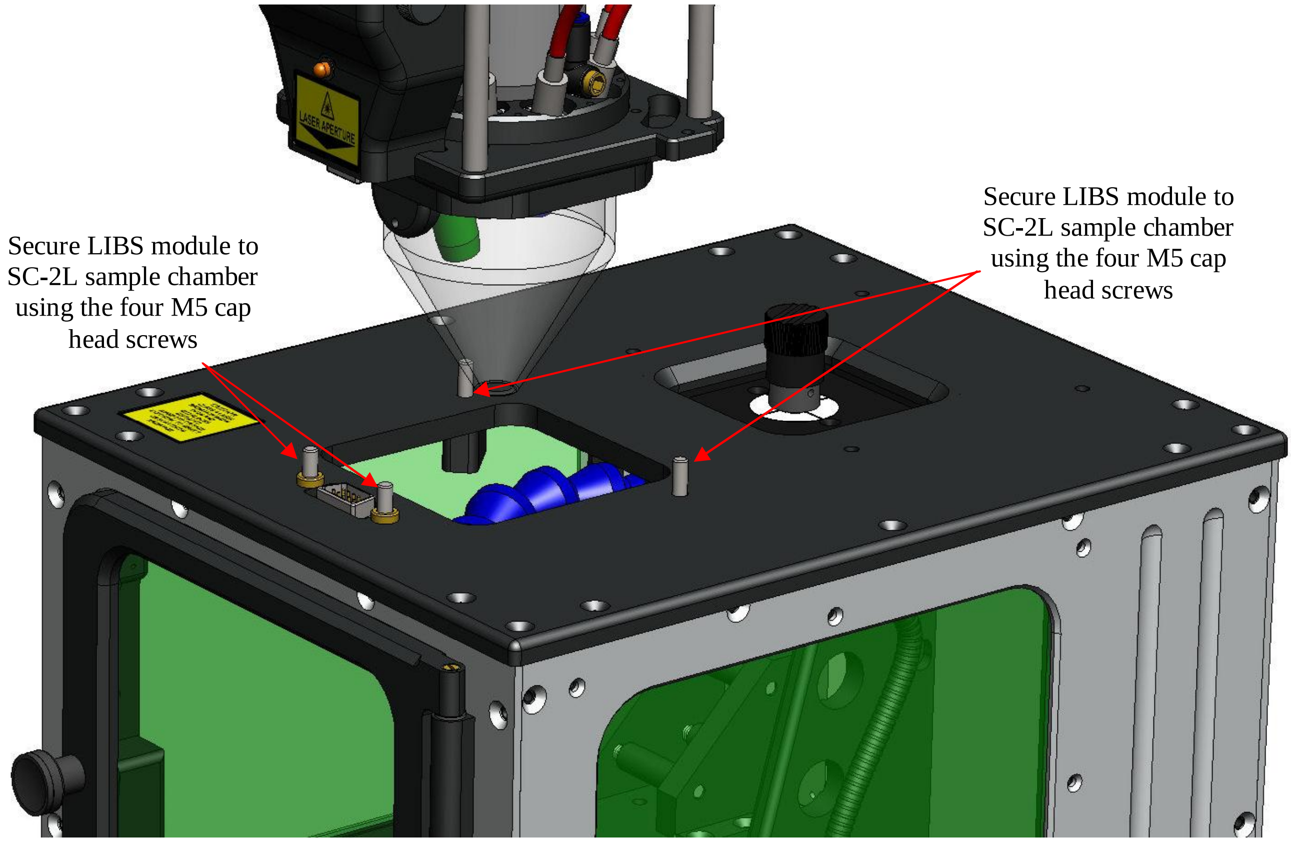


CAD view of SC-2L modular sample chamber

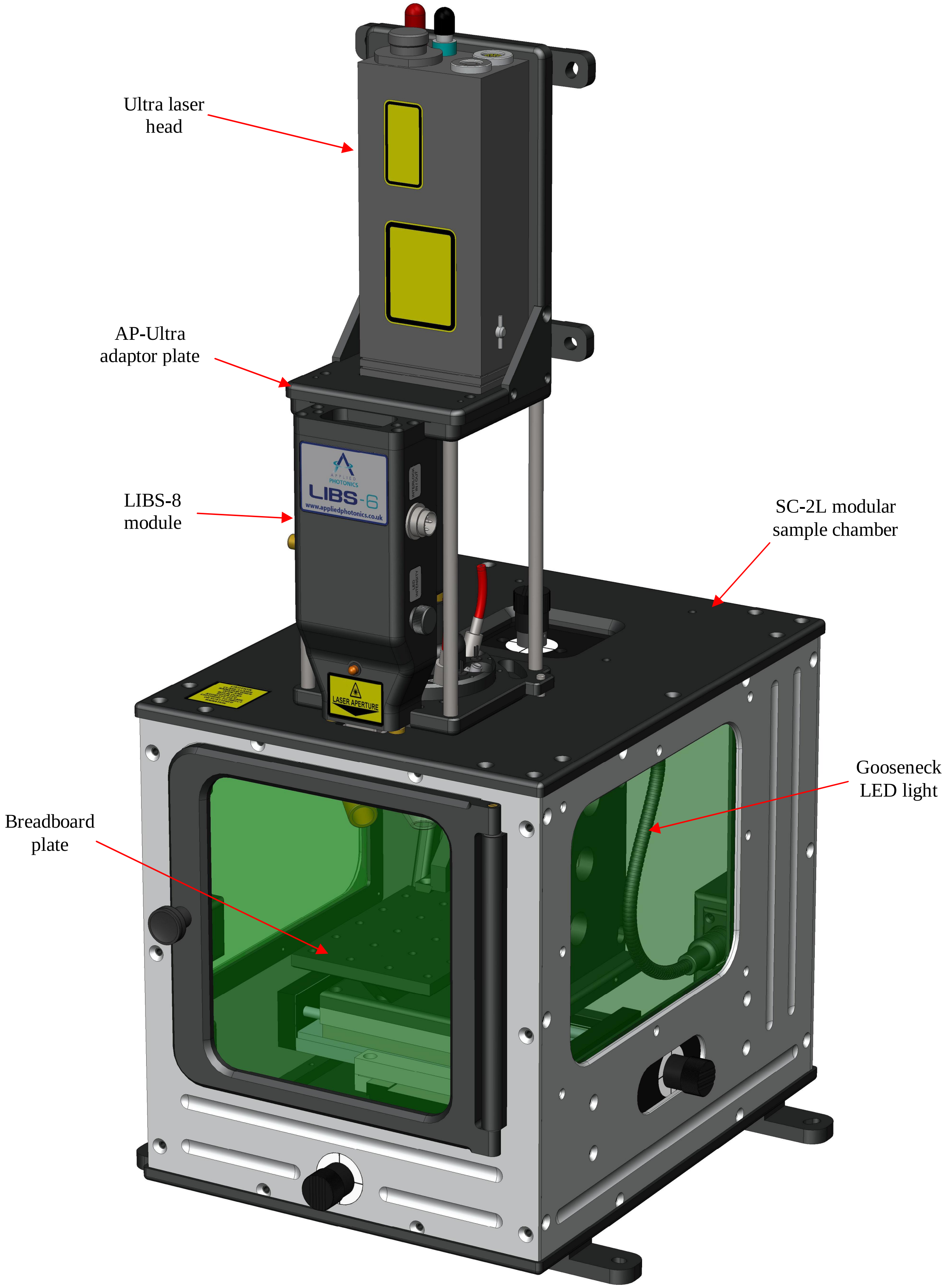


CAD view of rear panel of SC-2L modular sample chamber

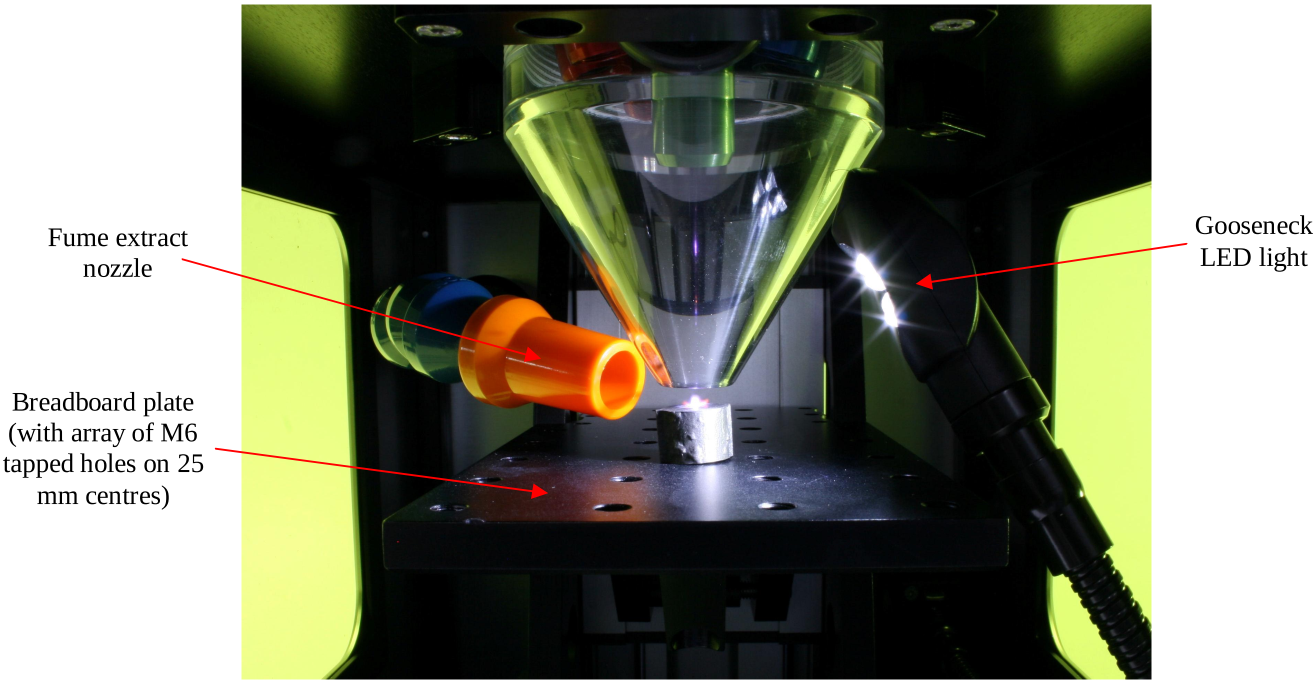
The LIBS-8 and LIBS-6 modules are designed to fit to the top of the sample chamber using four M5 screws as illustrated in the following figures. Before fitting the LIBS module to the sample chamber, ensure that the breadboard plate is positioned sufficiently low so as not to make contact with the aperture 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.



CAD view of SC-2L modular sample chamber illustrating method of attachment of LIBS-8 module



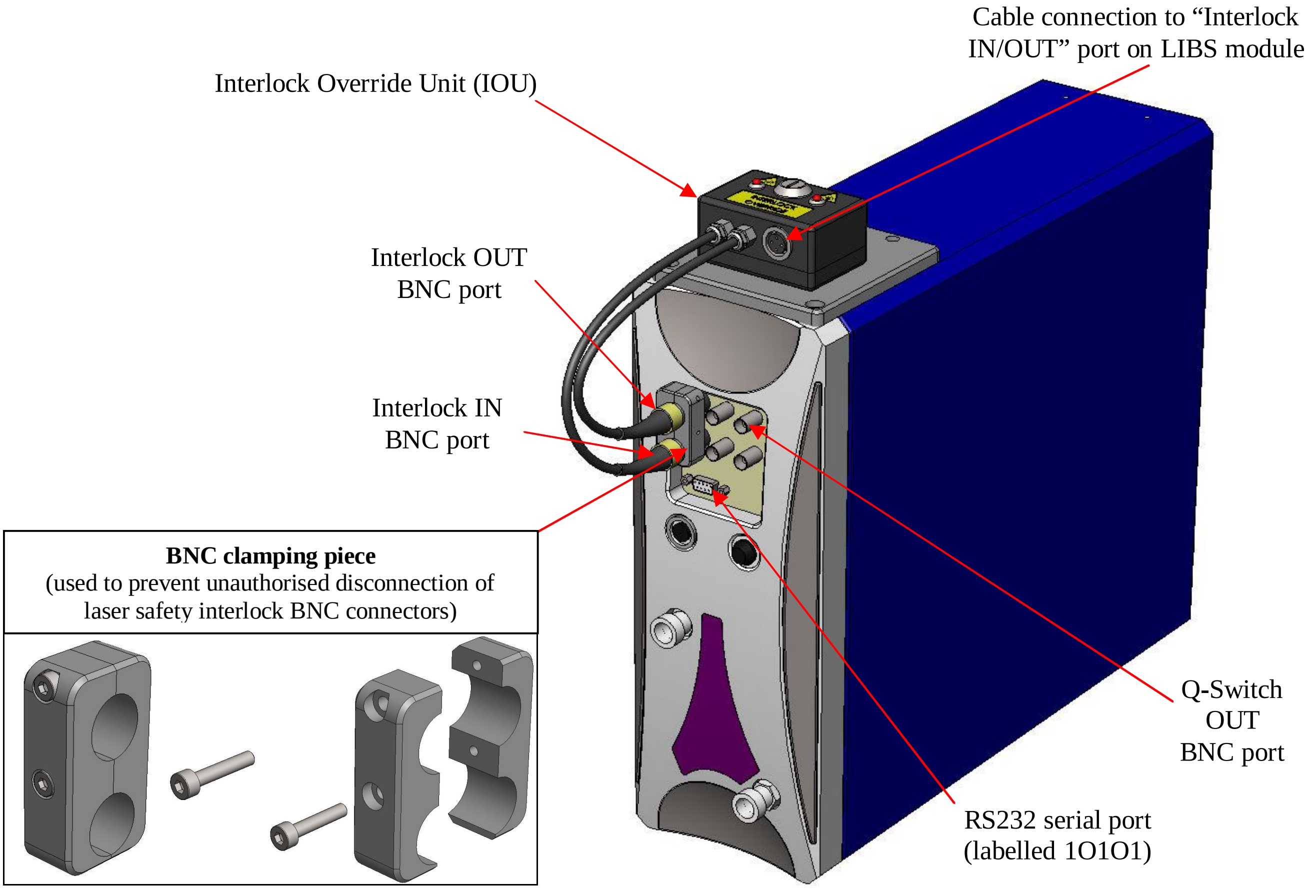
CAD view of LIBS-8 module with Ultra laser fitted to SC-2L modular sample chamber



Close-up view of inside of SC-2L sample chamber with LIBS module fitted
and showing a laser-induced plasma on a metallic target

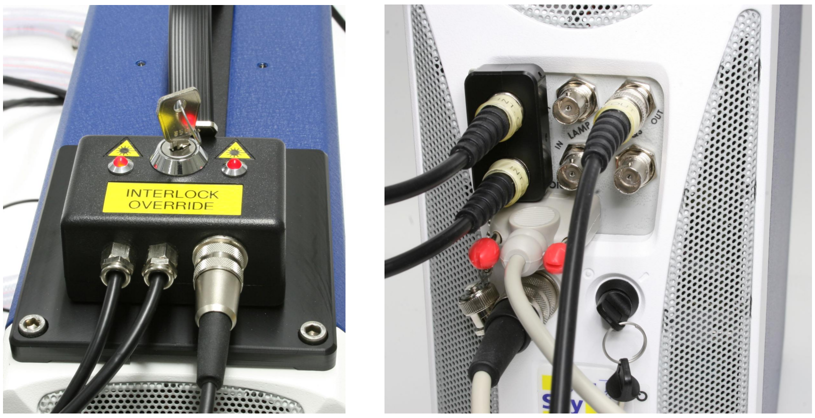
3.5 Quantel Ultra ICE 450 laser power supply and Interlock Override Unit

See the Quantel's instructions for a complete description of the ICE 450 laser power supply and cooling group unit. On initial unpacking of the product, the Interlock Override Unit (IOU) will need to be fitted to the ICE 450 laser power supply as illustrated in the following diagram.



Schematic diagram illustrating Interlock Override Unit and electrical connections to ICE 450 laser power supply

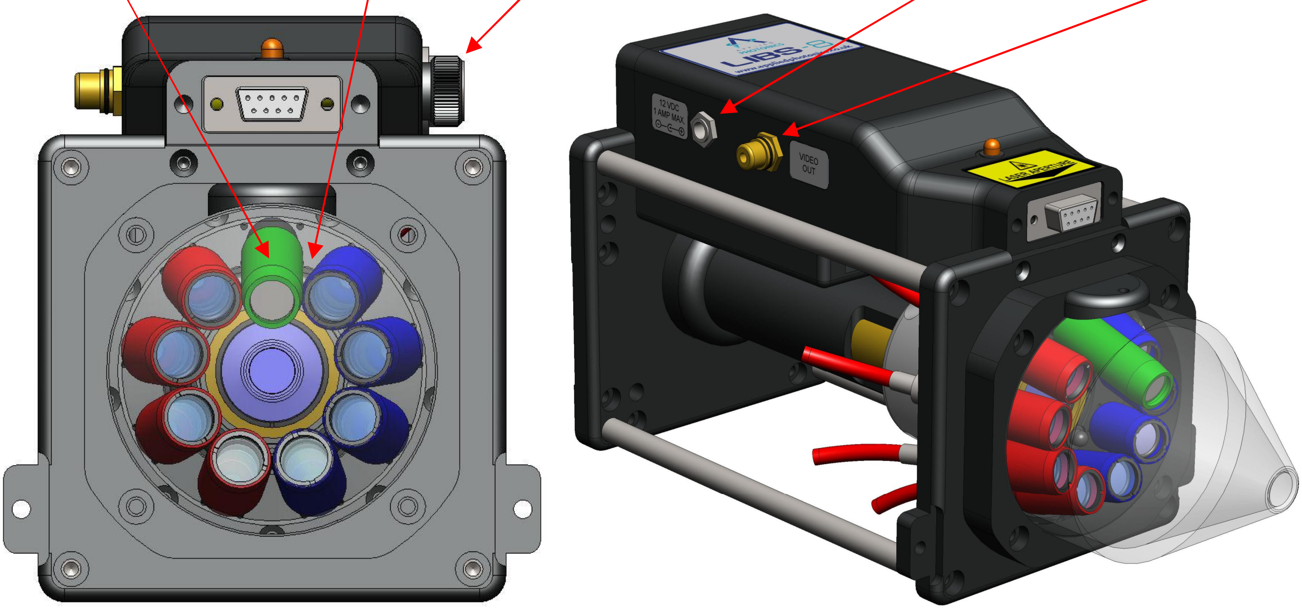
The Interlock Override Unit is used to override the laser safety interlock when the LIBS-6 or LIBS-8 module is required to be used in "open beam" configuration (ie. without a sample chamber). To override the interlock, the keyswitch should be turned "on" by turning clock-wise as indicated in the image below. The two red LEDs will flash continuously when the keyswitch is activated <u>and</u> the laser is in "active" mode. When the keyswitch is set to "off", the red LEDs will be extinguished indicating that the laser safety interlock is operating. The key should be removed from the Interlock Override Unit to prevent unauthorised activation of the interlock override.



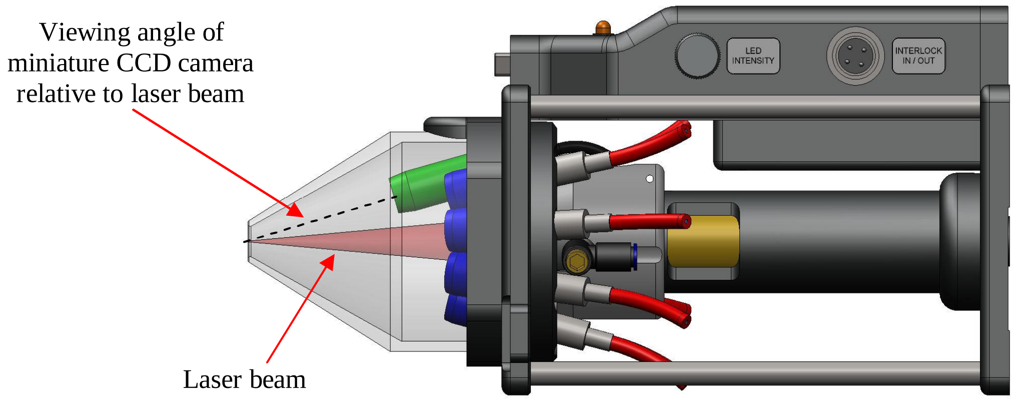
Images illustrating Interlock Override Unit and electrical connections to ICE 450 laser power supply

3.6 Imaging camera and associated components

The imaging camera is offered as an optional feature for both the LIBS-6 and the LIBS-8 modules (product code IMG-1). As illustrated below, a miniature colour CCD camera is located within the green tubular holder at the 12 o'clock position adjacent to the array of plasma light collection optics. The tubular holder is fitted with an optical filter to protect the CCD camera from damage due to possible high levels of stray laser light (laser wavelength must be specified when placing an order). The camera requires a 12 VDC supply which is derived from the power in port located on the side of the LIBS module. The video signal is obtained via connection to the "Video Out" phono connector.



CAD images of LIBS-8 module showing location of CCD camera and associated electrical connections



CAD image of LIBS-8 module showing viewing angle of miniature CCD camera

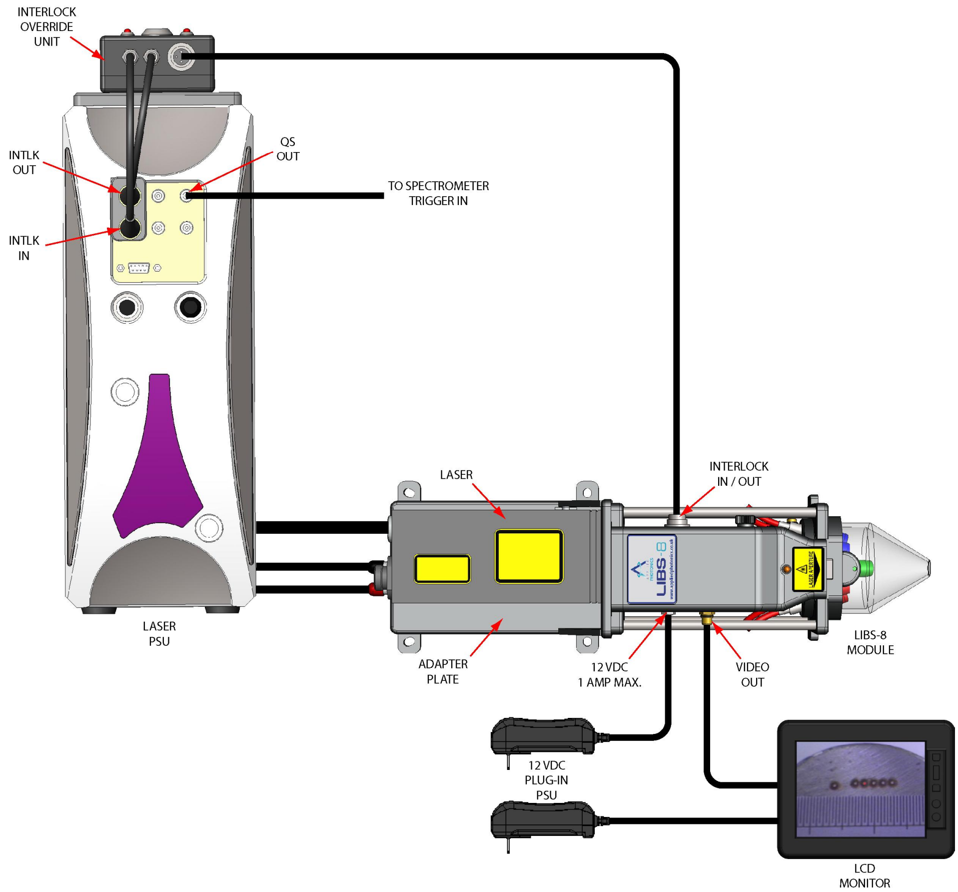
The specifications for the miniature CCD camera are:

1/7-inch CCD colour sensor (340,000 pixels)	Field angle: approx. 51º horizontal, 42º vertical	
Micro f = 2.1 mm, f/2.9 lens	Adjustable focus (approx. 20 mm to infinity)	12 VDC (110 mA)

An LCD colour monitor is supplied with the imaging camera kit and is connected as illustrated in the schematic diagram on the following page (section 3.7). Alternatively, the video images may be fed to a computer via the USB frame grabber also supplied with the imaging kit. A future version of APL's LIBSoft software will provide an imaging feature (available summer 2010).

3.7 Low-voltage electrical connections

The following schematic diagram illustrates how the LIBS module is electrically connected to the Interlock Override Unit (mounted on top of the Quantel Ultra laser ICE 450 power supply), the 12 VDC plug-in power supply and the LCD monitor (which forms part of the optional imaging camera kit).



Schematic diagram illustrating electrical connections between LIBS module and associated components

3.8 Assembly of system based on LIBS-8, Ultra laser and SC-2L sample chamber

Follow Quantel’s instructions for filling the Ultra’s ICE 450 laser power supply with cooling water and for connecting the water and electrical supplies to the laser head. Using the schematic diagram on the preceding page (section 3.7) make all electrical connections using the supplied leads (interconnecting leads supplied with the LIBS-8 modular LIBS system are labelled to facilitate final assembly). The following image illustrates how the system should look after final assembly (fibre-optic cables, LCD monitor, laser power supply remote box not shown).

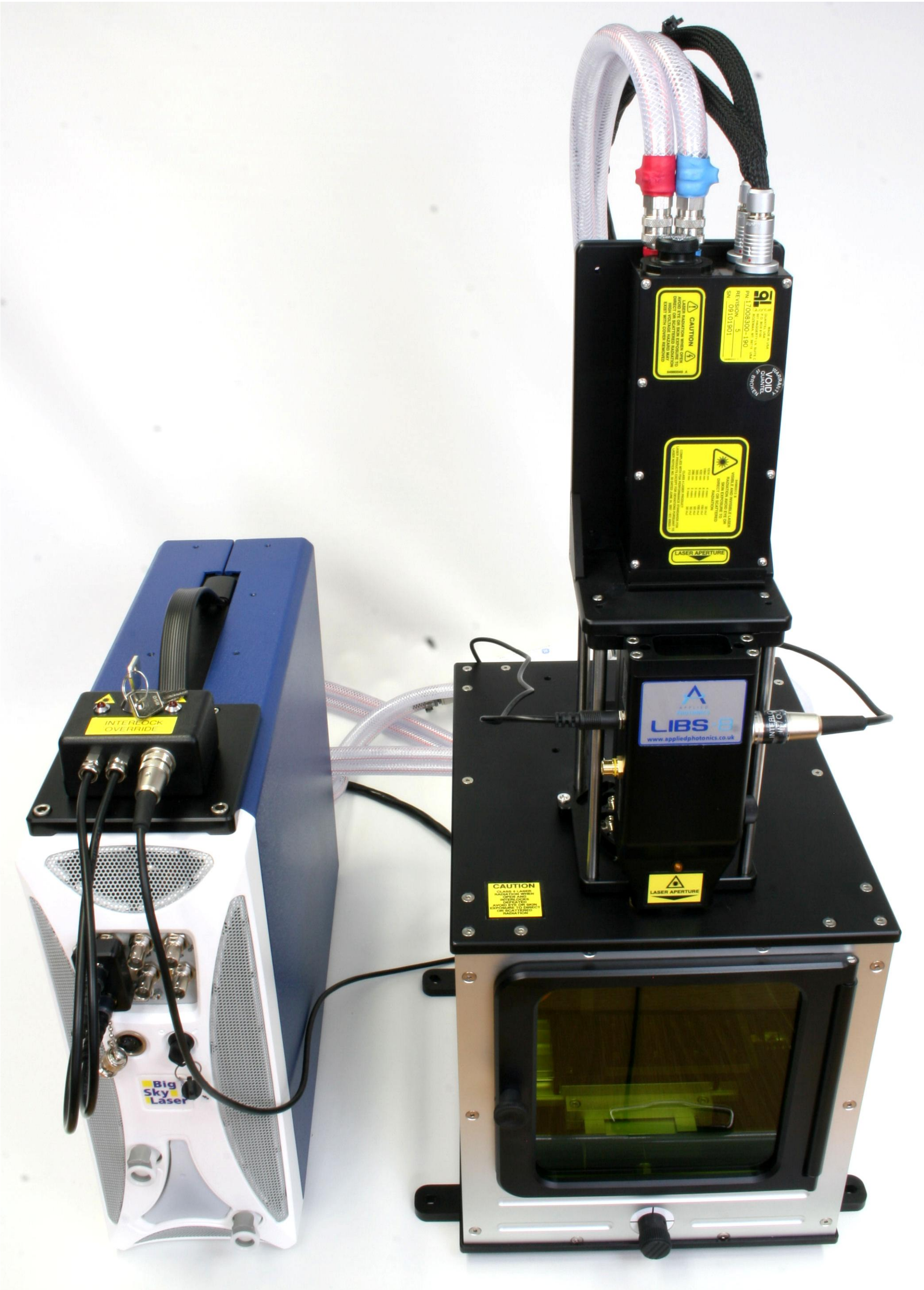


Image of LIBS-8, Ultra laser and SC-2L sample chamber after final assembly

4 Operating procedure

Step 1 The LIBS module and associated components should first be checked for obvious signs of damage, loose fixings, etc prior to use. If any of the components of the instrument are found to be of suspect condition, take remedial action before assembling and using the instrument. Of particular importance are the safety critical components such as the laser safety windows and electrical lid interlock of the sample chamber. Seek advice from the manufacturer if necessary. Do not operate the equipment with any of the covers removed.

Step 2 Prior to connecting the laser power supply, sample chamber etc, the LIBS module should first be placed on a suitable surface such as a laboratory bench or optical table. It is highly desirable to secure the LIBS module in some way (eg. by utilizing the feet attached to the base of the LIBS module which are designed to suit a metric or imperial optical table) to prevent accidental dropping of the module resulting in possible damage to the sensitive components inside.

Step 3 Fit the laser head to the LIBS module using the appropriate adaptor plate and attach assembly to the LIBS module (as illustrated earlier in this User’s Manual).

Step 4 If the sample chamber is to be used, fit the LIBS module to the sample chamber using the four M5 screws (supplied with the unit) as illustrated in the previous figures.

Step 5 Connect the 12 Volt DC (1.0 Amp) plug-in mains power adaptor to the power in port located on the side of the LIBS module.

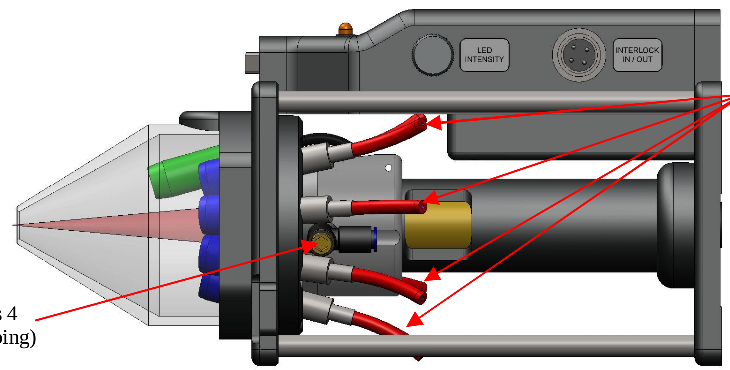
Step 6 Connect the coolant pipes and electrical cables (supplied with the laser) between the laser head and the ICE 450 laser power supply. Refer to the laser manufacturer’s instructions.

Step 7 Using the supplied interlock cable, connect the “Interlock IN/OUT” port on the LIBS module to the Interlock Override Unit (located on the top front of the ICE 450 laser power supply).

Step 8 Connect the “Trigger In” port on your spectrometer system to the “Q-Switch Out” port on the front of the laser power supply (requires a BNC-to-BNC lead).

Step 9 Connect fibre optic cables (not supplied with LIBS module) between LIBS module and your spectrometer system.

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 5 psi (2 to 3 psi should be adequate) and to control the flow-rate. DO NOT USE FLAMMABLE GASES!**



Step 11 Follow instructions supplied with laser for correct procedure for adding coolant (deionised) water to the laser power supply.

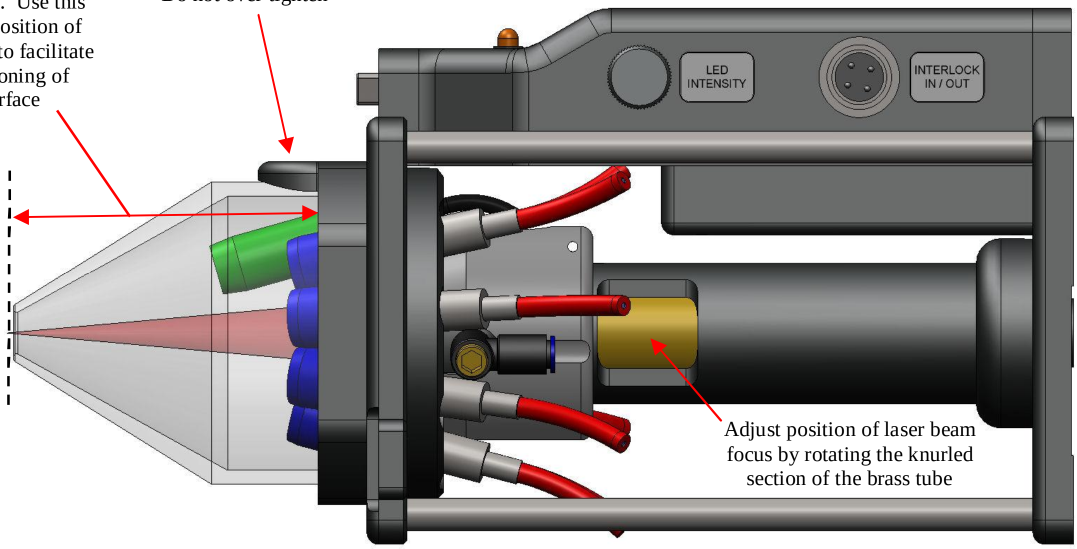
Step 12 Open laser safety shutter (refer to laser manufacturer's instructions).

Step 13 Activate laser by switching on key switch (refer to laser manufacturer's instructions).

Step 14 With the laser switched on (coolant water flowing, but laser flashlamp not yet activated), check for correct operation of the safety interlock by observing the "Interlock" light on the front panel of the laser power supply controller (see following figure) and opening / closing the door of the sample chamber (with LIBS module fitted and all interlock cables correctly fitted). If the interlock is working correctly, the "Interlock" light should flash when the sample chamber door is open and be constantly illuminated when the door is closed. 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 module is removed from the sample chamber, the interlock circuit is designed to de-activate the laser (see section 6 on *Operating instrument in "open-beam" configuration*). **Warning – do not operate the equipment if the safety interlock is not functioning correctly**.

Step 15 Place a sample of material (eg. 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 controls on the Remote Box of the ICE 450 laser power supply (see following figure and refer to laser manufacturer's instructions), the laser beam may now be fired 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 3.2 of this User's Manual).

View of front panel of Ultra laser power supply controller
(referred to as Remote Box in Quantel's documentation)



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

Step 16 If using LIBSoft software to control the laser, connect the personal computer to the serial port on the front panel of the ICE 450 laser power supply using the supplied Serial-to-Serial lead. If the personal computer is not equipped with a serial port, it will be necessary to use a Serial-to-USB converter (note that this device usually requires the installation of a software driver on the personal computer – refer to instructions written on the CD:ROM supplied with the Serial-to-USB converter lead).

Step 17 If using LIBSoft software to control the spectrometers, follow the instructions provided with the LIBSoft software to correctly configure the spectrometers and the laser for acquiring data. Note that the laser Remote Box should not be used while LIBSoft is running as this will result in the laser reverting to manual control. To resume LIBSoft software control of the laser, it will be necessary to switch off the laser and close LIBSoft, then switch on the laser followed by re-starting LIBSoft.

Step 18 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 Remote Box (refer to laser manufacturer's instructions). For the Ultra 100 mJ laser, the factory setting is typically around 140 microseconds for maximum laser output energy, although this will vary from laser to laser. Increasing the FL-QS delay will reduce the laser output energy. For the Ultra 100 mJ laser, the user will only be able to adjust the FL-QS delay setting from the minimum factory setting (typically 140 microseconds) up to 500 microseconds, although the laser output energy will likely be approaching zero with a FL-QS delay setting of approx. 400 microsconds. 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 Remote Box.

Step 19 After successfully testing the LIBS 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 experiment.

5. Shut-down procedure

Step 1 Shut down LIBSoft software (if being used) and then switch off laser power supply and isolate from mains electrical supply.

Step 2 Close the laser safety shutter on the laser head (refer to laser manufacturer's instructions).

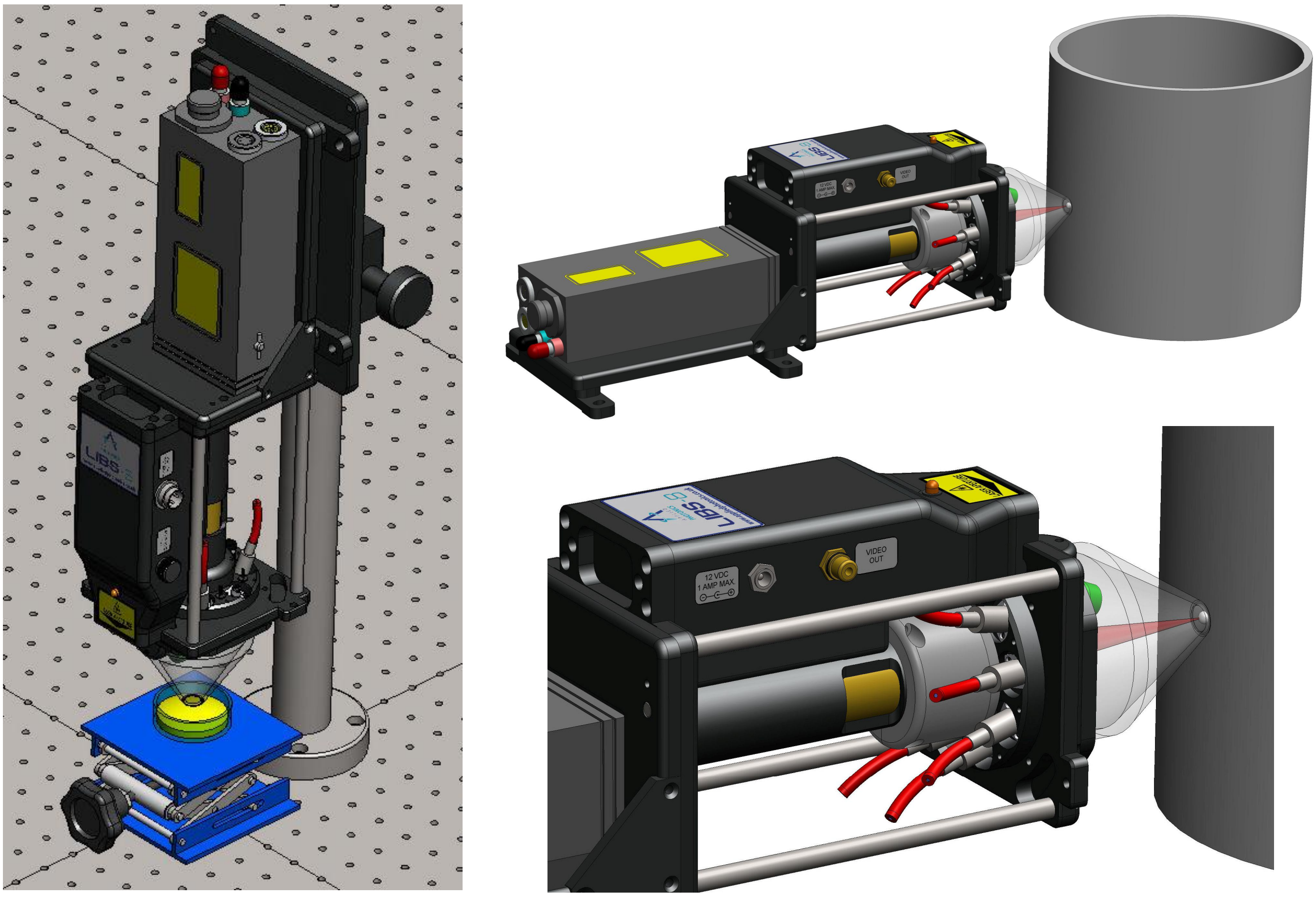
Step 3 Isolate 12 Volt DC plug-in power adaptor from mains electrical supply and disconnect from the LIBS module.

Step 4 Disconnect gas supply if connected.

6. Operating LIBS-6 and LIBS-8 modules 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-6 or LIBS-8 modules in "open beam" configuration.

The LIBS modules may be used without the sample chamber if required. Under these conditions, the laser beam is not contained (ie. 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 (eg. 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 not fitted to the LIBS module, it will be necessary to activate the Interlock Override Unit by turning the key switch to the "ON" position (ie. turn clockwise). The two LED warning lights will commence flashing (assuming the laser is in an active state). Consult your Laser Safety Officer before operating the LIBS modules in this mode.



Examples of a LIBS-8 module fitted to a Quantel Ultra CFR laser and being used in "open beam" mode

7. Maintenance and inspection

The LIBS-6 and LIBS-8 modules and associated equipment including the adaptor plate, safety interlock components, and the sample chamber (if used) 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. 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, for further advice on maintenance and inspection of their LIBS products.

8. Shipping and storage

The LIBS-6 and LIBS-8 modules should be kept in a clean, dry environment which is free from extremes of temperature. The module contains sensitive optical and electro-optical components and so should be protected from excessive vibration or shock. During transport, the module and associated components 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.

9. Trouble-shooting / fault finding

Laser does not activate:

1. Is the laser shutter set to the OPEN position?
2. Is the safety interlock connected correctly?
3. Is the "Interlock" indicator light on laser power supply controller flashing? If yes, then check that sample chamber door is fully closed. If a sample chamber is not fitted to the LIBS module and the equipment is required to be operated in "open-beam" (ie. Class 4 Laser Product) mode, then the Interlock Override Unit will need to be activated.
4. 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 has numerous emission lines (eg. 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 ("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.

Recorded spectra show some emission lines suffering from “self-reversal” (ie. 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)

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

1. The focal plane of the laser beam is set incorrectly (ie. it is set to be 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 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

Quick guide to acquiring spectra using LIBSoft V1.0

1. Start the LIBS system

- Ensure that all cables are connected correctly (refer to schematic diagram on page 21)
- Apply 12 VDC power to the LIBS module
- Power-up the laser
- Power-up the system computer
- Start the LIBSoft application (if being used)
- Check that the LIBSoft recognises the laser and, if software control of the spectrometer(s) is required, all of the spectrometers are recognised. Note that the current version of LIBSoft only supports Avantes spectrometers – contact Applied Photonics Ltd for further information on this.

2. Configure the laser

- Select ***Configuration>Configure laser*** from the menu or click on the blue ***LASER*** shortcut button to open the laser configuration window
- Set the Q-switch ***Delay (us)*** parameter to the required value
  This parameter adjusts the laser energy – the lower the value the higher the energy
  NB The default value when the laser is first powered-up is the minimum value of Q-switch delay for the specific laser in use, i.e., the maximum energy
- Set the Q-switch ***Rep rate (Hz)*** parameter to the required value
  20 Hz is the default value.
  Occasionally errors may be generated at high rep rates due to time taken to acquire data from the spectrometers following each Q-switch trigger pulse and the acquisition may then time out. If this proves problematic reduce the rep rate to the next lower value.
- Click the ***OK*** button to save the laser parameter settings and close the laser configuration window

3. Configure the spectrometers

- Select ***Configuration>Configure spectrometers*** from the menu or click on the blue ***SPECT*** shortcut button to open the spectrometer configuration window
- Set ***Start wavelength*** and ***Stop wavelength*** parameters for each spectrometer so that there is no wavelength overlap between spectrometers
  For example, if the *Stop wavelength* of one spectrometer is 353.1 nm the *Start wavelength* of the neighbouring spectrometer should be set to 353.2 nm
  Once set, these values are stored by the system, but can be changed at a later date if required
- Select which spectrometer is to be used as the ***Master***
  This is the spectrometer to which the Q-switch trigger from the laser is connected
  Once set, this value is stored by the system, but can be changed at a later date if required
- Ensure that each of the required spectrometers is ***Activated***
  If activated, the ***Status*** of the spectrometer will be identified as ***IN_USE_BY_APPLICATION***
- Set the ***Integration Time (ms)*** parameter to the required value
  Recommended value = 1.1 ms
- Set the ***Integration Delay (us)*** parameter to the required value
  Recommended value = 1.27 us
- Ensure that the ***Mode*** parameter is set to ***Ext Trig***
- Click the ***OK*** button to save the spectrometer parameter settings and close the spectrometer configuration window

4. Configure the measurement parameters

§ Select ***Configuration>Configure measurement*** from the menu or click on the blue ***MEAS*** shortcut button to open the measurement configuration window

§ Set the ***Number of Measurements*** parameter to the required value

§ Set the ***Number of Spectra*** parameter to the required value

§ Set the ***Number of Accumulations*** parameter to the required value

§ Set the ***Number of Conditioning Shots*** parameter to the required value

NB A measurement is defined as the acquisition of data from one target location on a sample. One measurement consists of the specified *Number of Spectra* each of which comprises the specified *Number of Accumulations* at that same target location. The specified *Number of Conditioning Shots* are fired prior to the acquisition of the first spectrum of a measurement in order to condition the sample surface. For a *Number of Measurements* > 1 the sample must be moved between measurements.

§ Set the ***Sample name*** parameter to the required value

§ Set the ***Sample filepath*** parameter to the required value

§ Set the ***Background name*** parameter to the required value
Only required if background correction is enabled

§ Set the ***Background filepath*** parameter to the required value
Only required if background correction is enabled

§ Enable/disable the ***Auto-increment filename*** function as required
If enabled, a numeric extension starting at *Current value* and incrementing by 1 for each subsequent measurement will be added to the *Sample name* to form the filename under which measurement data will be saved

§ Enable/disable the ***Auto-save spectra*** function as required
If enabled, the acquired spectra will be displayed and automatically saved
If disabled, the acquired spectra will be displayed and the user will be given the opportunity to save or discard the data

§ Enable/disable the ***Background correction*** function as required
It is suggested that this function is enabled
If enabled, the background spectrum specified by *Background name* and *Background filepath* is subtracted from each acquired spectrum
If enabled, it is important to ensure that a background spectrum has been acquired using the ***same*** spectrometer and measurement parameters as to be used for the measurements before any measurements are made

§ Click the ***OK*** button to save the measurement parameter settings and close the measurement configuration window

5. Acquire background spectrum

NB This is only required if the Background correction function has been enabled and a suitable background spectrum does not already exist

§ Select ***Acquisition>Acquire background*** from the menu or click on the orange ***B/G*** shortcut button
A spectrum will be acquired and displayed using the Master spectrometer's internal trigger to initiate the acquisition and a file dialogue box will be displayed allowing the user to save the spectrum

§ Save this background spectrum
NB The spectrum can be saved using the default filename (the *Background name* set on the measurement configuration screen) or a new name. If a new name is used the *Background name* on the measurement configuration screen will be updated accordingly and this file will be used to correct subsequent measurement data

6. Acquire measurement spectra

- Position the sample in front of the laser aperture
- Select ***Acquisition>Acquire spectra*** from the menu or click on the green ***ACQ*** shortcut button

  The laser flashlamps will be started if not already on and the laser Q-switch will fire in accordance with the specified laser and measurement parameters

  The acquired spectrum will be displayed and, if the *Auto-save spectra* function is enabled, saved.

  On completion of the measurement acquisition the laser Q-switch will be stopped, but the flashlamps will remain on

  If more than one measurement has been specified, the user will be prompted to move the sample to the next target location

  The spectrum can be viewed in more detail by maximising the spectrum display window and/or using the zoom and cursor function buttons

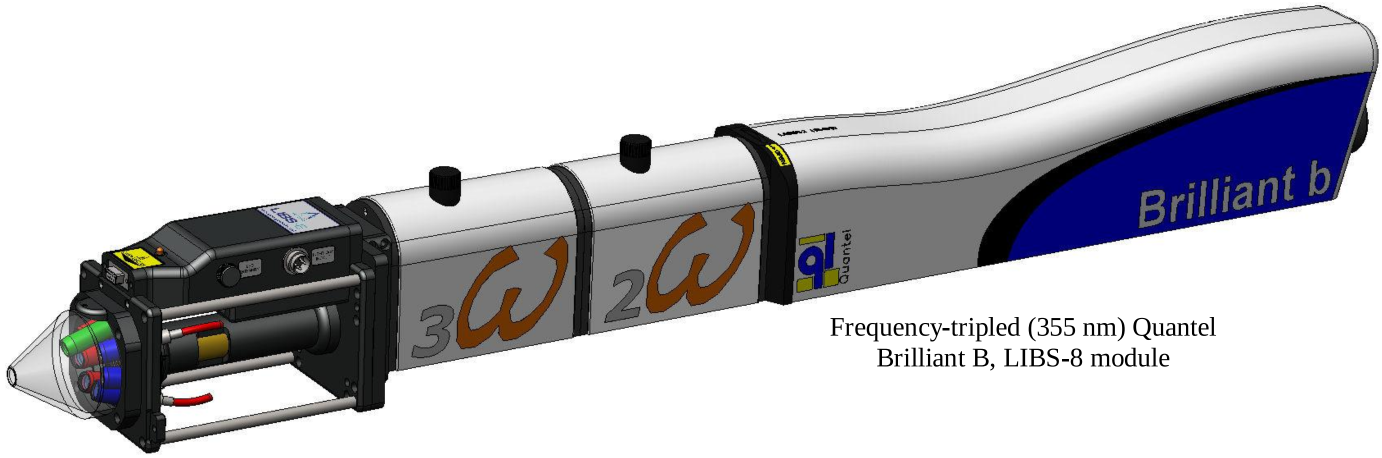
  If the *Number of spectra* > 1, each spectrum in the measurement can be viewed individually by adjusting the *Spectrum Number* control on the spectrum display window

  If the *Auto-save spectra* function is disabled, the displayed spectrum data can be saved by selecting the *Save* option from the menu on the spectrum display window

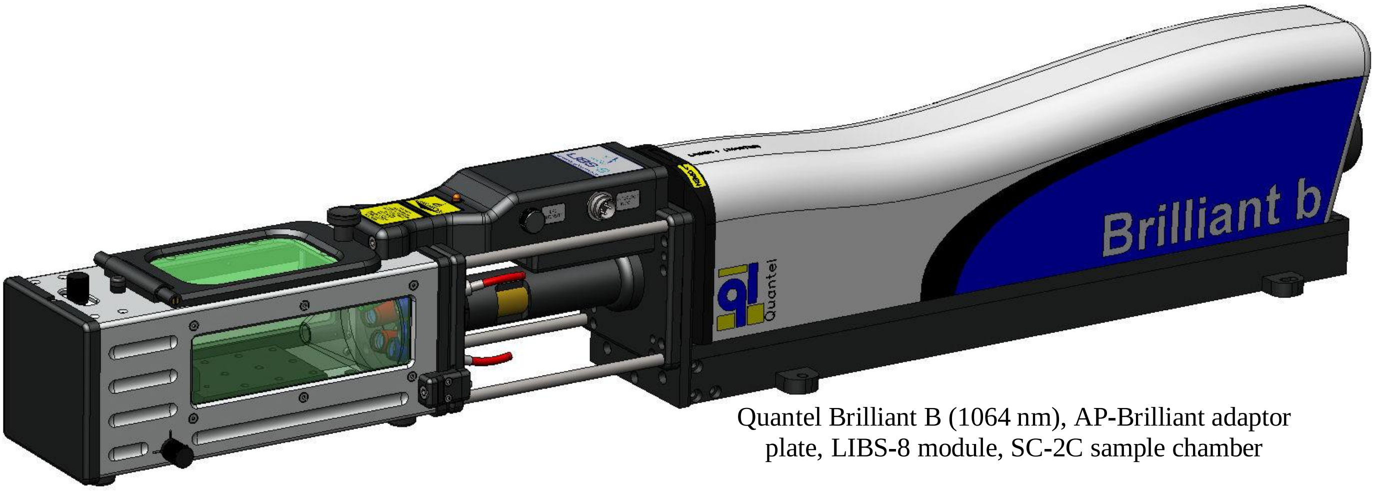
  To stop a measurement while the system is acquiring click on the red *STOP* shortcut button

Appendix A2

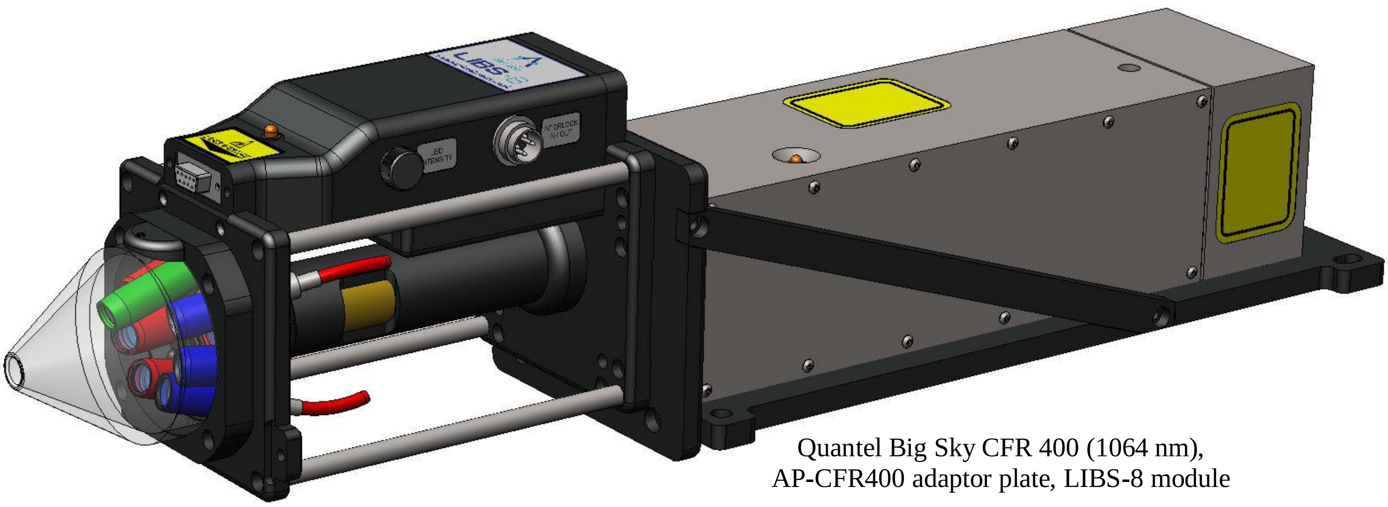
Example configurations of LIBS-6 and LIBS-8 modules



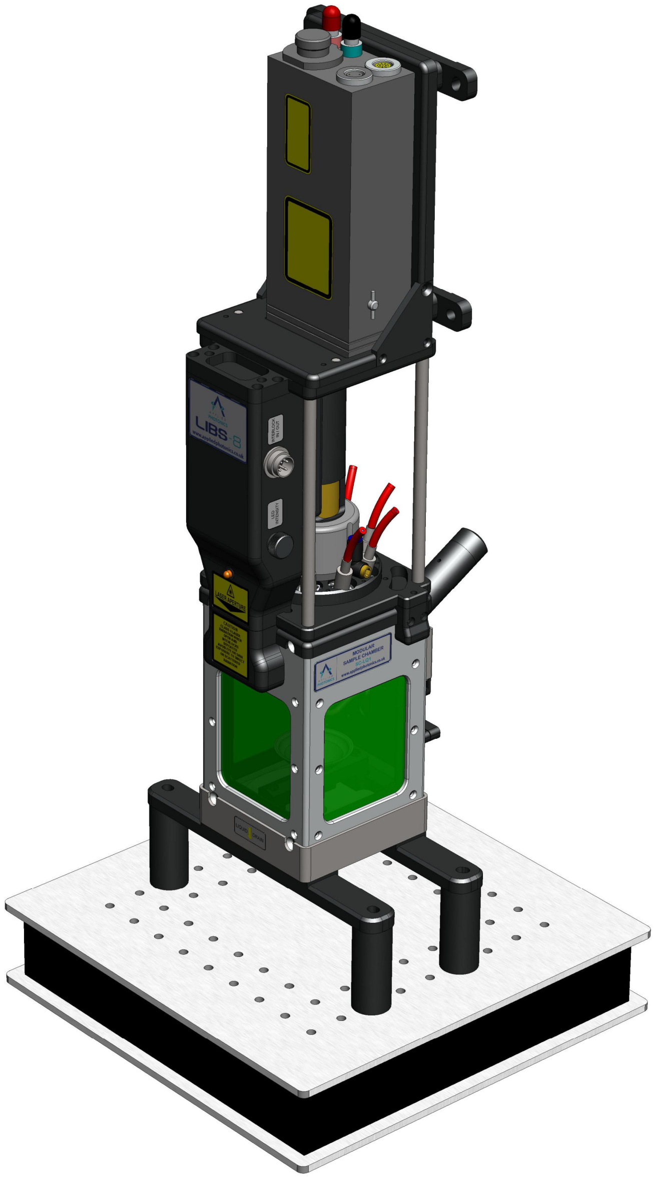
Frequency-tripled (355 nm) Quantel
Brilliant B, LIBS-8 module

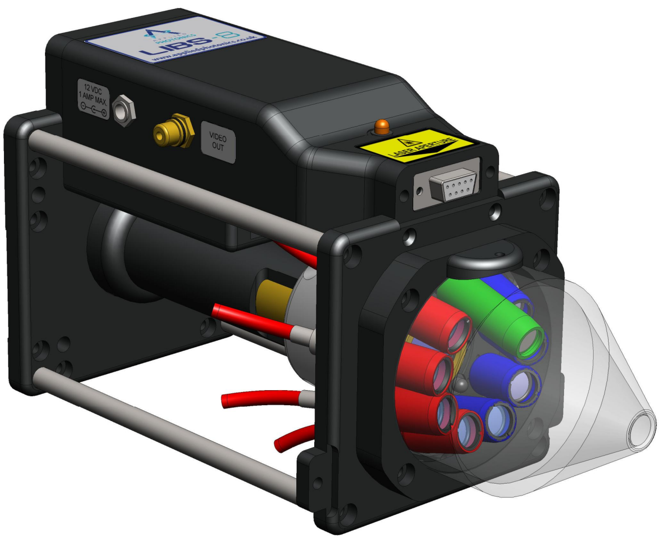


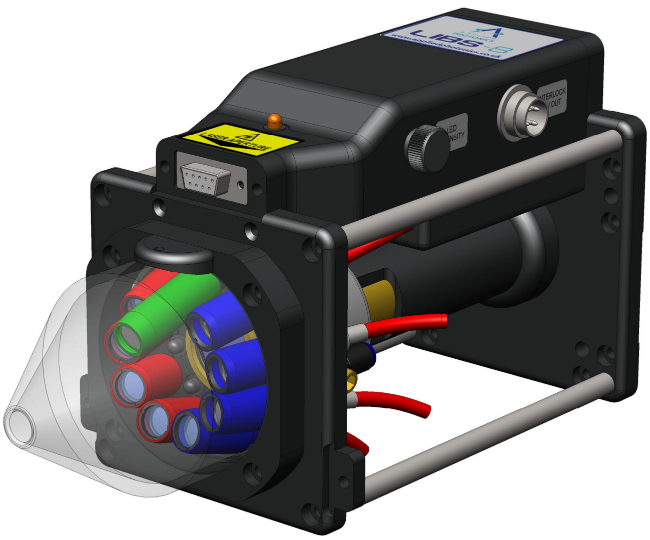
Quantel Brilliant B (1064 nm), AP-Brilliant adaptor
plate, LIBS-8 module, SC-2C sample chamber



Quantel Big Sky CFR 400 (1064 nm),
AP-CFR400 adaptor plate, LIBS-8 module







Appendix A3

Certificate of Conformity

Applied Photonics Limited
Unit 8 Carleton Business Park
Skipton North Yorkshire
BD23 2DE United Kingdom

EC
Declaration of Conformity

Applied Photonics Ltd declares that the product listed below has been designed and manufactured in compliance with the relevant standards as follows:

Product name: **LIBS-6 and LIBS-8 Integrated LIBS Modules**

Model Number: **LIBS-6 / LIBS-8**

Laser product safety
This device conforms with the principal objectives of safety of laser products by application of the following standards: **PD IEC TR 60825-14:2004 and BS EN 207:1999**

Electrical Safety
This device conforms with the principal safety objectives of the **European Directive 73/23/EEC**, as implemented by the Electrical Equipment (Safety) Regulations 1994, by application of the following standard: **BS EN 61010-1:2001.**

Electro-Magnetic Compatibility
This device conforms with the principal objectives of the European Directive (89/336/EEC) as amended by 91/31/EEC and 93/68/EEC, as implemented by The EMC Regulations (SI 1992 No. 2372 and amendment SI 1994 No. 3080), by application of the following standard: **BS EN 61326-1:2006**

Year of affixation of the CE Marking: 2010

Signed:

Name: Andrew I. Whitehouse

Title: Managing Director

Place: Applied Photonics Ltd, Unit 8 Carleton Business Park, Skipton, North Yorkshire BD23 2DE, United Kingdom

Date: April 2010