Xcalibur, Gemini

[Instrument version from September 2013 onwards]
X-ray Diffractometer Systems

User Manual

Version 2.4, October 2014

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For Assistance and Support

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CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

Safety Summary

General Safety Precautions

The following general safety precautions must be observed during all phases of operation of this instrument. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the instrument.

Agilent Technologies Inc. assumes no liability for the customer's failure to comply with these requirements.

Before operation, review the instrument and manual for safety markings and instructions. You must follow these to ensure safe operation and to maintain the instrument in safe condition.

General

This product is a Safety Class 1 instrument (provided with a protective earth terminal). The protective features of this product may be impaired if it is used in a manner not specified in the operation instructions.

Environment Conditions

This instrument is intended for indoor use in an installation category II, pollution degree 2 environment.

Refer to the specifications tables for the ac mains voltage requirements and ambient operating temperature range.

Before Applying Power

Verify that all safety precautions are taken. The power cable inlet of the instrument serves as a device to disconnect from the mains in case of hazard. The instrument must be positioned so that the operator can easily access the power cable inlet. When the instrument is rack mounted the rack must be provided with an easily accessible mains switch.

Ground the Instrument

To minimize shock hazard, the instrument chassis and cover must be connected to an electrical protective earth ground. The instrument must be connected to the ac power mains through a grounded power cable, with the ground wire firmly connected to an electrical ground (safety ground) at the power outlet. Any interruption of the protective (grounding) conductor or disconnection of the protective earth terminal will cause a potential shock hazard that could result in personal injury.

Do Not Operate in an Explosive Atmosphere

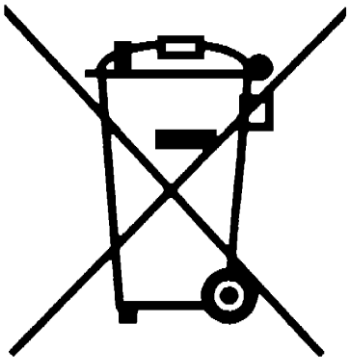
Do not operate the instrument in the presence of flammable gases or fumes.

Do Not Remove the Instrument Cover

Operating personnel must not remove instrument covers, except for the particular cases described in this manual. Component replacement and internal adjustments must be made only by qualified personnel.

Instruments that appear damaged or defective should be made inoperative and secured against unintended operation until they can be repaired by qualified service personnel.

Environmental Information



This product complies with the WEEE Directive (2002/96/EC) marketing requirements. The affixed label indicates that you must not discard this electrical/electronic product in domestic household waste.

Do not dispose in domestic household waste.

To return unwanted products, contact your local Agilent office, or see **www.agilent.com/environment/product/** *for more information.*

Important Information

This user manual applies to the Xcalibur and Gemini systems manufactured in Poland by Agilent Technologies Singapore International (ATSI) from September 2013 onwards (for previous models please refer to the appropriate other user manual).

Product:	Xcalibur (single X-ray source) or
	Gemini (dual X-ray source)
Electrical Ratings:	1/N AC 200-240V 50/60Hz 4000W
	Max 15A (33A peak): X-Ray Generator
	Max 5A: remaining circuits of the instrument

Before attempting to operate the system, PLEASE READ THE INSTRUCTIONS.

This product should only be used by persons legally permitted to do so.

If the equipment is used in a manner not specified in the User Manual, the protection provided by the equipment may be impaired.

Important Health and Safety Notice

When returning components for service or repair it is essential that the item is shipped together with a signed declaration that the product has not been exposed to any hazardous contamination or that appropriate decontamination procedures have been carried out so that the product is safe to handle.

Care has been taken to ensure the information in this manual is accurate and at an appropriate level. Please inform Agilent Technologies if you have any suggestions for corrections or improvements to this manual.

Service and support is available for technical and operational issues as indicated below.

- **Web** : www.agilent.com/chem/contactus
- **E-mail**: XRDSupport@agilent.com
- **Phone**: +44 (0) 1865 291600 between 8 a.m. and 4.30 p.m. (UK time), Monday to Friday
- **Fax**: +44 (0) 1865 291601

Original instructions in English language.

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1. Health and Safety Information

1.1 General

In normal operation the system is designed to operate safely. All users of Xcalibur/Gemini should be aware of potential hazards which exist in and around equipment of this type and the ways of avoiding possible injury and equipment damage which may result from inappropriate ways of working. A description of such potential hazards and how to avoid them is given in this section.

This manual adopts the following convention:

Indicates warning or caution. If you see this symbol on a product you must refer to the manuals for specific Warning or Caution information to avoid personal injury or damage to the product.

Warning symbols on the equipment are:

Protective conductor terminal

Earth (ground) terminal

CAUTION
Risk of electric shock

CAUTION
Refer to accompanying documents

WARNING
Radiation Hazard. This instrument produces X-rays when energised

(CANADA) WARNING
Radiation Hazard. This instrument produces X-rays when energised

CAUTION
Risk of finger squeeze between moving parts

See original manufacturers' manuals for further safety data on third party equipment supplied with the system. A list of these is given in this manual.

WARNING

Do not take risks. You have a responsibility to ensure the safe condition and safe operation of equipment.

WARNING

The equipment should only be operated and maintained by authorised operators of the system. An authorised operator is a person who has undergone specialist radiation training and has been trained in the use of Xcalibur/Gemini by Agilent Technologies personnel.

1.2 Electrical Safety

In normal use the user is protected from the dangers associated with the voltage, current and power levels used by the equipment.

1.2.1. Potential Electrical Hazards

The following list is not intended as a complete guide to all the electrical hazards on the system, but serves to illustrate the range of potential hazards that exist:

- electric shock
- electric burn
- fire of electrical origin
- electric arcing

1.2.2. Recommended Precautions

WARNING

All of the electrical equipment supplied as part of the system should be provided with a protective ground. Do not remove protective grounds as this may give rise to an electrical safety hazard. It is vitally important that the system is properly grounded at all times.

Follow local and national electrical regulations and procedures.

Do not defeat interlocks, remove connectors, disconnect equipment, open safety covers, dismantle or modify equipment unless you are qualified and authorised to do so and you are fully conversant with its operation and potential hazards, or have total assurance through your local electrical permit to work system that the equipment has been made safe.

Ensure that the mains supply is fused at an appropriate rating, or fitted with a circuit breaker, and that it can be isolated locally via a clearly labelled, clearly visible and easily accessible isolating switch. Isolate the supply before carrying out any maintenance work.

Do not touch any unshielded wires or connectors while mains power is supplied to the system.
Do not allow water or any other foreign objects to come into contact with the equipment's electrical components.

WARNING

Mains voltages are present in the system. High voltages are used by the X-ray tube and power supply.
Only personnel qualified to work with high voltages and currents are permited to perform service or maintenance work on such equipment.

1.3 Mechanical Handling Safety

WARNING

Lifting points are provided for safe handling of components and safe handling practice must be observed to comply with local regulations.
Check that lifting points are used only for the job intended.
The system itself and some components are heavy and require careful handling.
Use safe lifting procedures for heavy items to prevent possible strain injury.

1.4 Safe Mechanical Practice

In normal use personnel are not required to undertake mechanical work. However, servicing or repair may necessitate access to any part of the system. Only personnel who have been trained by Agilent Technologies to carry out service work on this equipment are permitted to service the equipment.

Water connections should be made and tested in accordance with any local and national safety regulations.

1.5 Mechanical stress

The protection cabinet housing protects personnel from exposure to various risks (including electrical, mechanical, thermal and electro-magnetic radiation). Care should be taken to avoid mechanical damage of the cabinet housing. The metal panels of the housing shield the device against an impact energy of up to 5J, whereas the transparent window in the front door is resistant to an impact energy up to 1J (IK06 rating). If damage is found on the enclosure panels or window then stop using the instrument immediately and inform Agilent Technologies.

IK06

WARNING

Protect the window against mechanical damage. Do not use the instrument if the window is broken.

1.6 Moving Parts

There are a number of moving parts in the system which are powered by electric motors.

WARNING

Injury could result if clothing or body parts become caught in moving mechanisms. Keep clothing, hands and body parts away from moving mechanisms.

WARNING

Between the moving parts of the goniometer there are a number of places at which a potential finger squeeze or shearing hazard exists. The warning triangle shown to the left is visible on the goniometer close to these places. Keep hands away from the goniometer when parts are moving.

When the protection cabinet is open the goniometer moving parts are disabled. The Xcalibur/Gemini system contains two **MOTION ENABLE** switches in front of the goniometer, inside the protection cabinet. During some operations, such as mounting a sample, the goniometer must be moved between defined positions while the protection cabinet is open. In order to do this **BOTH** Motion Enable switches must be pressed and held simultaneously and this ensures that both hands of the operator are kept away from the potential finger squeeze or shear locations.

In the event that the goniometer moving parts become blocked by an obstruction, the movement will be automatically stopped and a small reverse movement made so that the blockage can be safely released. The control software will report a movement error and will wait for user intervention. The procedure to follow to restart the instrument is shown in the troubleshooting section of this user manual.

1.7 X-ray Radiation

WARNING

This equipment contains an X-ray tube. Ensure that safe working practices relating to radiation are employed. Follow any local, national or international rules and guidelines.

Intentional or reckless misuse of the X-ray generator or its safety devices including safety interlocks and cabinet shielding can result in serious injury or even death.

During operation, there is an acceptable level of X-ray radiation as based on the recommendations on risk published by the International Commission of Radiological Protection (ICRP) and endorsed by the National Radiological Protection Board (NRPB) in the UK. For use in the UK, the Ionising Radiations' Regulations 1999 should be adhered to. For countries outside the UK the appropriate laws apply such as registration and inspection.

Customers should be aware of their duty of safety to their employees and visitors and should consult national authorities to determine local operational requirements.

WARNING

To prevent injury to personnel and possible damage to the equipment, please note the following guidelines:

1. **Only authorised personnel who have received appropriate instruction and are aware of the laboratory rules that govern the use of this type of system should operate the system.**
2. **Never dismount the beam stop when the system is operational.**
3. **Do not operate the system without the collimator, unless performing the beam alignment procedure.**
4. **Use appropriate X-ray detection equipment to perform regular radiation checks as per any laboratory rules**
5. **Use only genuine firmware X-ray tubes, X-ray generators, goniometer heads and collimators, as recommended by Agilent Technologies. Use of other products may compromise the performance of the shielding and safety system, and may invalidate your warranty.**

1.8 Extreme Temperatures

WARNING

Systems fitted with the low temperature option use liquid nitrogen and/or liquid helium as a coolant. Liquid nitrogen and liquid helium are cryogenic liquids and can cause cold burns. Wear gloves when handling cryogenic liquids and use eye protection. Refer to the information supplied with the equipment for more information.

1.9 Vacuum

WARNING

When handling and using X-ray tubes and the CCD detector, particular care should be taken to avoid injury caused by possible implosion of the vacuum tube. Wear eye protection.

The CCD detector front panel is made from beryllium and can be easily damaged by a strong force by hand or a gentle force by a sharp instrument. Such damage can result in vacuum implosion of the CCD detector. There is danger of exposure to potentially toxic beryllium material. In case of such an event take action to close the protection cabinet doors and panels and obtain specialist assistance to clean up the beryllium.

1.10 High Pressures

WARNING

Know the law about high pressure gas cylinders and follow it. High pressure cylinders are often used to store gases (typically at pressures up to 200 bar). Most countries have laws about using them.

- **Chain cylinders to a fixed object or keep them in specially designed trolleys**
- **Only use approved and tested high pressure fittings**

1.11 Hazardous or Toxic Materials

Beryllium and beryllium oxide are toxic materials. Follow appropriate handling, shipping, use, storage and disposal procedures and regulations. Refer to BrushWellman Material Safety Data Sheet No. M10 for further information.

Beryllium is contained in the front panel of the CCD detector and in the X-ray tubes. Care should be taken not to touch the beryllium panels/windows on the detector and tubes.

WARNING

If Beryllium is exposed to fire, it may oxidise to highly toxic beryllium oxide powder. Do not attempt to clear up the remains of any fire, but contact the relevant local agency stating that there is an incident involving possible beryllium or beryllium oxide contamination.

1.12 Maintenance

The manufacturer will not be held responsible for the safety, reliability or performance of the equipment unless assembly operations, extensions, re-adjustments, modifications and repairs are carried out only by persons authorised by the manufacturer. Interchangeable parts which are subject to deterioration during operation must be serviced or interchanged during the intervals given.

2. Introduction

2.1 Scope

This manual applies to the Xcalibur/Gemini system designed and manufactured by Agilent Technologies.

2.2 How To Use This Manual

This manual is aimed at operators and maintenance personnel of the Xcalibur/Gemini system. Operators of the system should be computer literate, familiar with X-ray diffraction techniques, have had training in the use of the Xcalibur or Gemini system by Agilent Technologies staff, and have had training about radiation safety.

This manual is intended to provide operators with a practical guide to the system and its operation. This is intended to familiarise the user with how the system works and provide a better understanding of the system operation.

All personnel who are likely to operate the system or who are likely to come into contact with any of the system components should read the **HEALTH AND SAFETY INFORMATION** section of the manual. This provides basic information aimed at highlighting the safety hazards associated with the equipment.

More detailed information and instructions for component parts of the system are given in the third party manuals supplied with the system, which are listed in this manual. These manuals should also be read and understood before operating the system.

The purpose of this manual is to:

- explain how to operate the equipment
- explain how to interface to the equipment
- list performance characteristics of the equipment
- describe how the equipment operates
- assist with simple fault finding and maintenance

2.3 System Overview

Xcalibur systems are single crystal diffractometers that use the property of X-ray diffraction to determine the crystal structure of materials. They are intended for use with single crystals of chemical substances (inorganic, organic or organo-metallic), mineralogical and biological samples. Xcalibur systems may also be used in the analysis of powder samples. Intended samples should have a maximum unit cell dimension of 500 Angstrom for macromolecular PX systems.

Xcalibur systems may be used with crystal conditioning devices. Specifically, low and high temperature attachments and high pressure cells. Some minor modifications may be required by Agilent Technologies to enable use of these devices.

When the Xcalibur system is equipped with two X-ray sources it is named Gemini.

3. Specifications

3.1 Environmental Requirements

It is essential that the climate of the laboratory is controlled to ensure that the CCD detector is not damaged. Typically air-conditioning should be installed to maintain the temperature and humidity within the ranges listed below. The Relative Humidity is particularly important as the CCD and its cooling pipes can reach ~17°C, condensation should not be allowed to collect on the CCD at any time.

Xcalibur/Gemini with KMW3000 (i.e. external water cooled) dissipates ~6000 BTU/hr heat, not including any optional cryogenic devices.

Xcalibur/Gemini with TF3500 (i.e. air cooled) dissipates ~16000 BTU/hr heat, not including any optional cryogenic devices.

Air temperature in the room during operation.	18 – 25 °C
Stability of ambient temperature during operation	± 1 °C
Storage temperature	>10°C <40°C
Relative humidity	20 - 80 % non – condensing
Location	Clean, dust free environment >2m from air conditioning or heating units
Floor strength	Able to bear system weight of 500 kg on a footprint 80x80cm

3.2 Services

3.2.1. Electrical Supply

Total power requirements	200-240V~ Max 15A (33A peak): X-Ray Generator Max 5A: remaining circuits of the instrument
Number of outlets required	4 single-phase outlets 16A/200-240V (32A/100-120V) for diffractometer system (comprising goniometer, interface, detector, etc) computer, monitor and external water chiller (such as KMW3000C) 1 single phase outlet 33A/200-240V for HV Generator 4 single-phase outlets (for temperature attachments)
Voltage fluctuation	< ± 10 % (with line voltage regulator fitted if necessary)
Location of outlets	On wall behind system
Protection	Circuit breakers to be fitted to all outlets

3.2.2. External Water Cooling (when used with KMW3000)

Min flow rate	1.8 l/min at 12 °C
Pressure	3-5 bar gauge
Return line pressure	1-2 bar less than supply line pressure (**Take care to check this on closed loop water systems**)
Temperature stability	± 5 °C
Temperature range	5 – 20 °C

3.3 Performance Data

3.3.1. X-ray Tube

Maximum radiation dose due to scattering or leakage at 10cm distance from any outside surface: < 1µSv/hr

Typical Operating Conditions

Tube	**Voltage (kV) setting**	**Current (mA) setting**	**Resulting power (W)**
Mo	55	40	2200
Cu	50	40	2000

Maximum Operating Conditions

Tube	Voltage (kV) setting	Current (mA) setting	Resulting power (W)
Mo	55	54	3000
Cu	50	44	2200

3.3.2. CCD Detector

3.3.2.1. General

CCD chip	Truesense Imaging KAF4320-E
Scintillator material	Gadox
Peltier cooling	-40°C (three stage cooler)
Temperature stability	± 0.05°C(micro-processor PID)
Analogue-to-digital resolution	True 18 bit
System noise (so-called read noise)	<12 e- RMS
Dark current	<0.05 e-/pix.s
Communication	Gigabit Ethernet
Readout time (EosS2, AtlasS2, TitanS2)	0.22s (4 x 4 binning) 0.38s (2 x 2 binning) 0.75s (1 x 1 binning)
Readout time (Eos, Atlas, Titan)	0.28s (4 x 4 binning) 0.46s (2 x 2 binning) 1.59s (1 x 1 binning)

3.3.2.2. Eos, EosS2 detectors

Active area	95mm diagonal
Weight	10kg
Pixel size on scintillator	31µm
Fibre optic reduction	1.3; low distortion reduction taper

3.3.2.3. Atlas, AtlasS2 detectors

Active area	135mm diameter
Weight	16kg
Pixel size on scintillator	48µm
Fibre optic reduction	2.0:1; low distortion reduction taper

3.3.2.4. Titan, TitanS2 detectors

Active area	165 mm diameter
Weight	20kg
Pixel size on scintillator	60 µm
Fibre optic reduction	2.5:1; low distortion reduction taper

3.3.3. PC CCD Interface

Communication	Gigabit Ethernet
Drivers	Win 7
Typical host computer	Pentium IV class PC >2.8 GHz ≥1.0 Gb RAM 20" colour display

3.3.4. Four-circle Kappa Geometry Goniometer

Type	Four–circle Kappa geometry X-ray goniometer
Sphere of omega, kappa, phi coincidence	10 µm
Maximum load Phi axis	2 kg
Resolution	0.00125 deg for Omega and Theta 0.0025 deg for Kappa 0.005 deg for Phi
Scanning speed range	0.005 to 3.0 deg/sec
CCD detector angular range	-115 to 157 deg (depending on the instrument configuration)
CCD detector to sample distance	40 to 150 mm (depending on the detector)

Shutter response time	3 ms

3.4 Electrical Data

	X-ray Generator	**Goniometer Interface**	**CCD Detector**
Power connection	1/N AC 230 V ± 10% 50/60 Hz	1/N AC 200-240V 50/60 Hz	1/N AC 200-240V 50/60 Hz
Maximum power consumption	3000W	250 W	125 W
Maximum mains current	19A	1.2 A	1.2 A
Main fuse	32A	6.3 A	3.15 A
Ground terminal	None	2.5 mm^2 Cu	2.5 mm^2 Cu

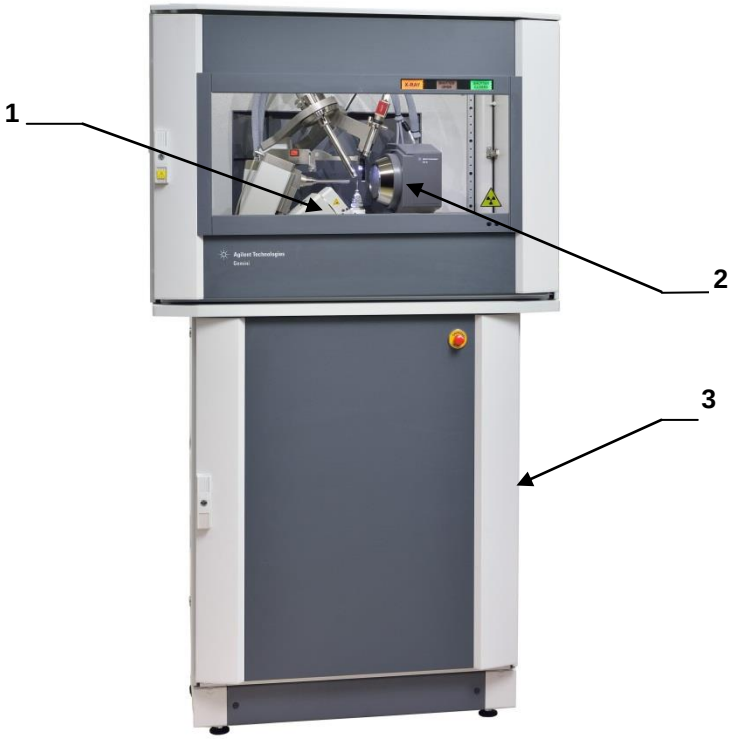
	CCD Water chiller	**KMW3000 Water chiller**
Power connection	1/N AC 200-240V 50/60 Hz	1/N AC 200-240V 50/60 Hz
Maximum power consumption	300W	900W
Maximum mains current	1.5A (230V)	4A (230V)
Main fuse	T 2.5A/230V	T 6.3A/230V
Ground terminal	2.5 mm^2 Cu	2.5 mm^2 Cu

4. Technical Description

4.1 Overview of Xcalibur/Gemini

The Xcalibur/Gemini system consists of:

1. A kappa geometry, 4-circle diffractometer
2. A CCD area detector
3. An instrument cabinet with electronics rack
4. System software installed on PC workstation



Note:

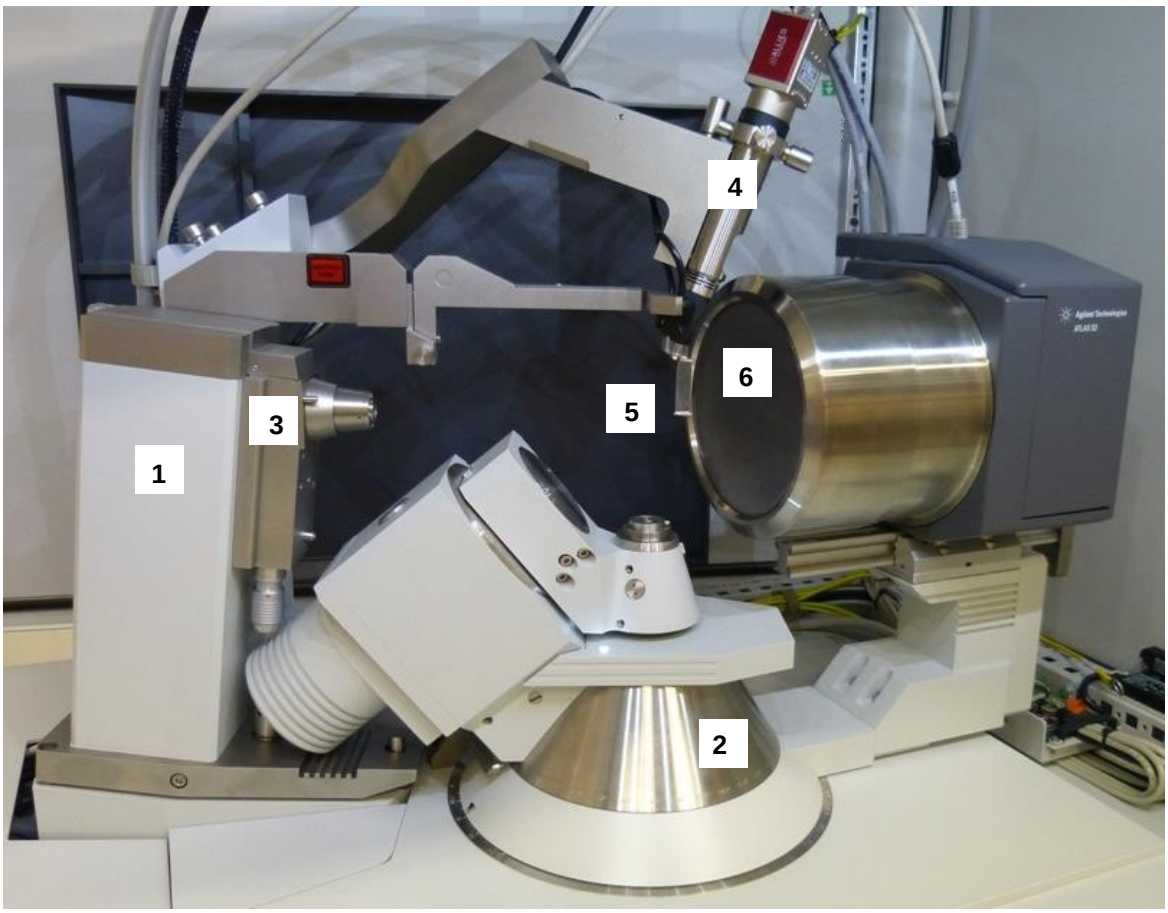
The numbers on this diagram refer to the numbered list above

Figure 1 Components of a typical Xcalibur/Gemini system

Figure 2 Computer and monitor

The diffractometer and CCD area detector are mounted inside a cabinet. The cabinet experiment area is mounted on top of the electronics rack. The water cooler for the CCD detector is positioned in the electronics rack. The water cooler for the X-ray source is positioned on the floor adjacent to the rack. The system is supplied with a control PC, monitor, keyboard and mouse.

The diffractometer consists of one or two X-ray tubes, a 4-axis Kappa goniometer (omega, kappa, phi and theta axis) for sample orientation with a detector arm, which has a universal mount capable of supporting any Agilent Technologies CCD area detector. The CCD area detector is used to measure the X-quanta diffracted from the sample.



Key

1. X-ray tube
2. 4-circle Kappa goniometer
3. X-ray Shutter
4. Video microscope
5. Beamstop
6. Beryllium Window

Figure 3 View of a typical diffractometer

The X-rays are generated by a long fine focus sealed tube, which is mounted on the goniometer and powered by the high voltage generator. The X-ray optics consist of a high speed shutter located next to the tube shield, graphite monochromator and a collimator for refining the X-ray beam.

The sample can be viewed with the video microscope, which is attached to the stand doming the instrument. The image is displayed on the computer monitor.

The CCD area detector works according to the following principle: The X-rays enter the detector through a Beryllium window to the vacuum-sealed detector unit. A scintillation screen transforms the X-ray photons to light, which is conducted via a fibre optic reduction taper towards the scientific grade CCD chip. The CCD signal

is digitised to 18-bit resolution by a correlated double sampling circuit with analog-to-digital converter located in the detector head. The data transfer is via a gigabit Ethernet link to the PC workstation. The control program stores the data for further data analysis to the hard disk.

The five goniometer axes are driven by microprocessor-controlled stepping motors with 12,800 microsteps per revolution.

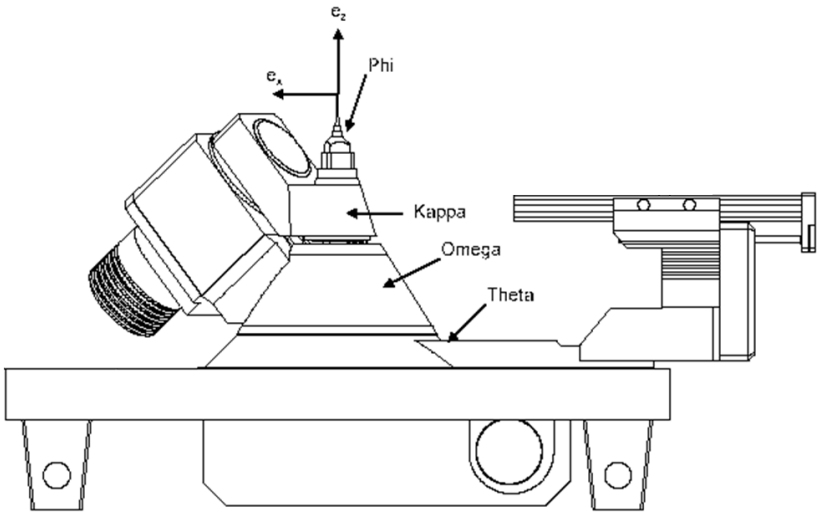


Figure 4 Goniometer phi, kappa, omega and theta axes (shown on a typical diffractometer)

The sample is aligned using a CCD video microscope. The sample picture is displayed on the control PC. A high brightness LED lighting system provides brilliant, high contrast illumination of the sample at all goniometer positions.

4.2 Mo / Cu X-ray Source

This source unit contains a long fine focus Mo or Cu X-ray sealed tube, dual shutter, pre-aligned graphite monochromator, exchangeable collimator and X-ray beamstop. The sources are positioned with manual adjustment screws. The sources require water cooling by a water cooler such as the KMW3000C from Agilent Technologies, which is described in a separate manual.

The X-ray tube is connected to the HV generator unit. The range of voltage is 10-60kV. The supplied high voltage cable should be connected between the X-ray tube and the HV Generator.

The X-ray source contains both a fast shutter and a safety shutter. The safety shutter is for safety purposes only and cannot react fast enough to make time-controlled exposures of a CCD detector. Fast exposures are created using the fast shutter. The source also contains an over-temperature safety switch.

4.3 X-ray Generator

The X-ray generator is located in the electronics rack in the instrument cabinet.

The generator power trips out if the interlock circuit opens, which happens if X-ray tube overheats, the cooling water flow is insufficient, the cabinet warning lamp fails or the emergency stop button is pressed. Warning lights on the front panel indicate the operational status of the generator.

The PC controls and communicates with the X-ray Generator using a RS232 COM port connection.

4.4 Water Cooler for CCD detector

The Water Cooler (model name SBC 70) is located in the electronics rack in the instrument cabinet. It is used for cooling only the CCD detector.

The chiller operates only in water-to-water mode with a water reservoir (see Figure 5) on the left-hand side of the electronic rack. This reservoir is used to fill the internal circuit for the CCD detector.

To fill the reservoir in the rack use 1:10 solution of ethylene glycol in distilled water (pre-mixed) to prevent the liquid and circuits from algae growth. 3L of liquid should be used to fill the reservoir and an additional 3L of liquid should be needed to fill the pipes. The filling port is on the top of the reservoir in the rack. The left hand side panel of the electronics rack (at the bottom of the system) will need to be removed in order to access the reservoir. This is done by using the same key as used on the protection cabinet doors, to rotate a lock at the top and bottom of the panel.

The Cooler needs an external water supply (tap water, integrated building water circuit or additional water chiller) to cool the device. The heat from the internal circuit is delivered to the external water circuit. The external circuit is connected in series with the X-ray tube water cooling circuit and the external water supply is connected to supply and return fittings at the rear of the system.

4.5 Electronics Rack

4.5.1. Water distribution panel

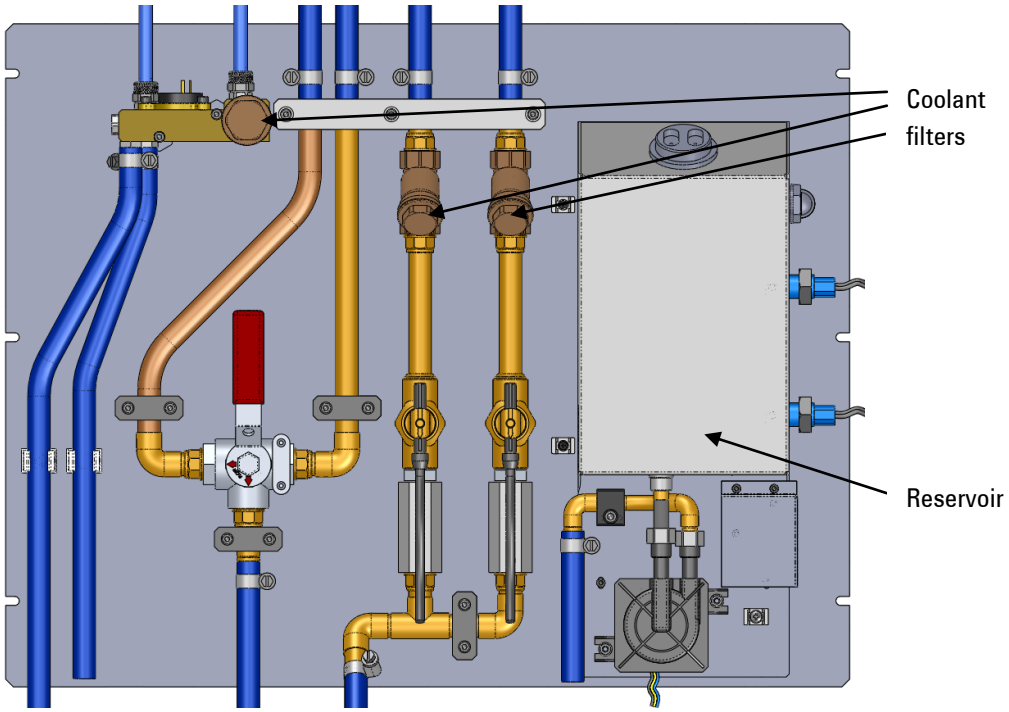


Figure 5 Water distribution panel and reservoir (Gemini version)

The water distribution panel is mounted in the left side of the rack. This panel contains in-line water filters for the internal water circuits (through CCD detector and X-ray sources). A reservoir is mounted on this panel which fills the internal circuit (separated from external cooling water).

4.5.2. IO Device for safety interlocks

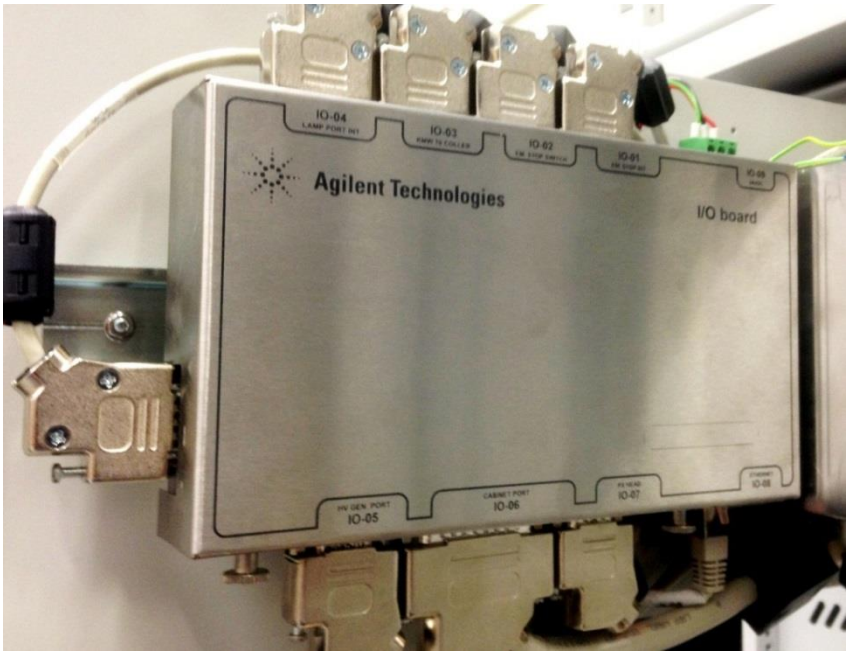


Figure 6 Safety IO Device

Safety interlock wiring is organised through a PCB (named IO Device) in the system electrical enclosure.

After an error state the IO Device has to be reset to clear the error. This must be done in order to close the interlock circuit to allow the X-ray generator to operate. The IO Device window (shown in the figure below) is opened by clicking on the IO icon near the top right of the Crysalis^{Pro} software main window. The user must click on the **Reset** button to clear the error. The cabinet lights can also be remotely controlled from this window.

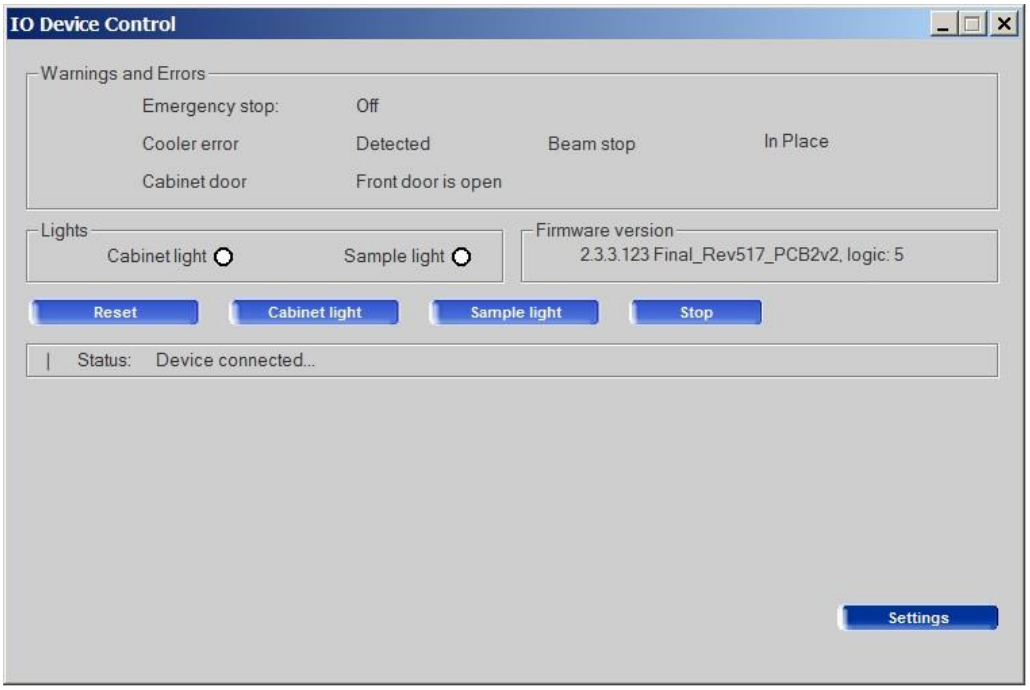


Figure 7 IO Device Control window

4.5.3. Ethernet switch

The Ethernet switch inside the DC8 system Interface is used for connections between the system PC and Ethernet enabled devices such as the CCD water cooler and the IO Box. This allows the PC to remotely control and communicate with devices via Ethernet even when isolated from an external network.

4.6 Software

The programs controlling the measurement procedures of the system are WIN32 applications, which run under Windows XP or Windows 7. The data acquisition and reduction are run concurrently with the program CrysAlis^{Pro}.

4.7 Low Temperature Option

If a cryogenic cooler is fitted to the diffractometer system, then the correct adapter must be used to mount the cooler on the stand for optional equipment. Further details can be obtained from Agilent Technologies.

4.8 Safety Features

X-rays are generated and projected in a totally enclosed cabinet constructed of steel and lead equivalent glass.

Xcalibur/Gemini has magnetic switches mounted on the front door and rear, left and right side panels of the protective enclosure. During operation the X-ray shutters will not open unless the doors are closed. If the doors are then opened whilst the X-ray shutters are open the X-ray shutters are both immediately caused to close.

Access to the interior of the cabinet via the front door is required when changing samples. When the door is opened during sample change and alignment, the X-ray generator remains on for optimum performance, but the safety shutter is automatically closed. Whilst the cabinet is open only the phi axis of the goniometer may be rotated by software. The other axes may be commanded to move by the software but motion will only physically happen if the two Motion Enable button in the cabinet are simultaneously pressed.

Indicator lights mounted on the outside of the enclosure show when the X-ray generator power is on (orange light) and whether the X-ray shutter is open (red light) or closed (green). If these lights are defective then the X-ray generator will not operate (in the case of the orange light) and the X-ray shutters will not open in the case of the red and green shutter lights.

The door of the cabinet should remain locked when the system is unattended to prevent unauthorised access to the system.

5. Handling, Installation, Storage and Transit Information

5.1 Reception and Handling

5.1.1. Delivery

Carry out the following steps on delivery of the Xcalibur/Gemini system:

1. When the system arrives, check that there is no visible damage, with the delivery driver present. If damage has occurred contact the carrier and Agilent Technologies **immediately**.

WARNING

The packing crates are heavy and could cause serious injury and damage to the equipment if not handled correctly. Use suitable lifting equipment and procedures. Only lift the packing cases from the bottom.

CAUTION

Do not remove the equipment from the packing crates until they have been moved to their designated installation site. The equipment has been carefully packed to protect the equipment from damage in transit. Removal of the packing equipment could make the equipment vulnerable to damage during transit.

2. Always lift packing cases from the bottom using suitable lifting equipment (refer to list of component weights in the following section.
3. Move packing cases into the designated installation site.
4. Contact Agilent Technologies to notify them that the equipment is awaiting installation by an Agilent authorised service provider.

5.1.2. Unpacking

1. Retain all packing material until installation of the system is completed.

2. Ensure that special tools are stored safely for use during maintenance periods.

5.1.3. Mechanical Handling

5.1.3.1 Weights, Dimensions and Lifting Points

Description	NET Weight kg	Dimensions (width x height x depth) cm	Centre of gravity	Lifting points
Kappa goniometer	106	43 x 64 x 47	Offset from centre of unit towards side of X-ray tube mount	At four corners (DO NOT lift from below)
X-ray generator	40	48 x 22 x 69	Centre of unit	From the sides and below
Protection cabinet	120	100 x 72 x 84	Centre of unit	By hand from bottom four corners
Electronics rack	140	80 x 104 x 80	Centre of unit	By hand from top or bottom four corners
CCD water cooler	20	48 x 13 x 48	Centre of unit	At the four corners and from below
OPTIONAL ITEMS:				
KMW3000C	56	38 x 54 x 96	Centre of unit	At the four corners and from below
Helijet	10	30 x 25 x 10 (head only)	Centre of component parts	From below with transfer tube removed
Cryojet5	15	15 x 35 x 15 (head only)	Centre of component parts	From below or using handles whilst supporting transfer tube.
MercuryITC	5	44 x 10 x 30	Centre of unit	By hand from below
GFC1	12	45 x 26 x 38	Centre of unit	Front panel handles

5.1.3.2 Boxed Weights and Dimensions

Box (No.)	Item	Weight (kgs)	Length (cm)	Width (cm)	Height (cm)
1	Goniometer	160	95	60	90
2	Electronics rack	185	90	90	134
3	Protection cabinet	220	115	100	100
4	Chiller / Accessories	195	122	80	95
5	X-ray source(s) / Generator	195	122	80	95
6	CCD detector	145	120	95	117
7	LN2 Dewar	120	70	70	155
8	Cryojet	145	70	80	190
9	Helijet	90	60	95	55
10	KMW3000C Chiller	105	47	79	100

The weights and dimension above are an estimate and should only act as an indication of the lifting requirements when the system is delivered. All boxes are fitted with the facility to use forks to unload. There is 15 cm clearance from floor to the base of each box.

It is recommended that a fork lift truck is available to unload the delivery vehicle with a pallet truck to move the packing cases into the systems final location.

5.2 Installation and Setting to Work

5.2.1. Preparation of Site and Services

5.2.1.1. Environmental Requirements

It is the customer's responsibility to ensure that all local building and safety regulations are met.

Ensure that the environmental conditions of the installation site conform to the requirements stated in the SPECIFICATIONS section of this manual.

5.2.1.2. System Layout

Adequate space is required around the system for servicing. The minimum clearance from the walls and a suggested system layout are shown in Figure 25.

When the low temperature option is fitted an extra 100 cm space on the left-hand side of the system is required. When the system is in the Gemini configuration (two sources) then this 100cm space is also required on the

left-hand side of the system to allow the side panel to be removed to carry out the wavelength change procedure.

Unpacked, the largest subassembly will fit through a door aperture of 85 cm. Check the door aperture to ensure the system can be assembled in its designated area.

5.2.1.3. Electrical Services

One single phase 32 A outlet (for X-ray generator) and two single phase 16 A outlets are required. Additionally four single-phase outlets are required for optional accessory items.

Use only the power cables supplied.

Do not connect the electrical power supply circuit to any other devices. Limit the electrical noise in the system by attaching the earth cable exclusively to an external earth terminal. The instrument is equipped with a terminal to attach an external protective grounding cable, which should have a cross-section of not less than 4mm^2.The resistance between the ground connection and the instrument terminal should be not greater than 0.1 ohm. This grounding connection should be made in a secure manner to avoid accidental disconnection.

Fit a line voltage regulator if the power supply voltage fluctuates more than ±10%.

Locate the mains outlet on the wall behind the system. The mains outlet should be of the circuit breaker type. (Outlet and connecting plugs are not supplied). The mains plug should be readily accessible by the operator when the equipment has been installed.

In areas where the mains power supply is unreliable an 'uninterruptible power supply' (UPS) is recommended. The UPS should have specifications of 10 kVA with 3-phase input and single phase output. For example the following unit from APC will run Xcalibur/Gemini from a 400V 3-phase input for about 100 minutes:

APC Smart-UPS VT 10kVA 400V w/3 Batt Mod Exp to 4, Start-Up 5X8, Int Maint Bypass, Parallel Capable + (1)SYBT4 Battery Unit

Description	**Voltage V**	**Frequency Hz**	**Maximum nominal mains current A**
X-ray generator	230 ±10%	50/60	19
Water cooler	100 – 240	50/60	2.5
CCD detector	90-130 / 180-260	50/60	2.5
Goniometer Interface, video monitor, cabinet lamps, sample illumination	90-130 / 180-260	50/60	2.5
Computer and peripherals	100 – 240	50/60	2.5
Cryojet controller (option)	100 / 240	50/60	2
KMW3000C water cooler	230 ±10%	50/60	1.5 (4A at startup)

5.2.1.4. Water Supply

A cooling system is required to dissipate the heat produced by the X-ray tube. A closed circuit cooling system is supplied to minimise the effects of particles, low pressure and water temperature fluctuation on the performance of the system from local tap water supply. This cooling system could be the Agilent Technologies KMW3000C (water-to-water chiller) or a third party water-to-air chiller (for example TF3500). These chillers are closed circuit cooling systems suitable for this purpose.

The system is supplied with hoses that have a 10mm inside diameter. In the case of KMW3000 water-to-water cooling the distance between the water supply and the chiller is not limited but the supply must deliver 3 – 5 bar gauge pressure with a minimum 1.8 litres/min flow (if the water supply temperature is 12 degC).

The water supply should have a wall mounted shut off valve.

Care should be taken on closed loop water supplies that the return line pressure is not so high that water flow is blocked. The difference between supply and return line pressures must be 1-2 bar.

5.2.1.5. Low Temperature Option

A suitable high vacuum pump, ideally 70 Litres/sec turbo is required to periodically evacuate the low temperature device.

To demonstrate the operation of the device, e.g.Cryojet, 100 litres of liquid nitrogen are required.

The customer should supply a suitable rack for the device controller.

5.2.1.6. CCD Camera Pumping

A suitable high vacuum pump, ideally 70 Litres/sec turbo-molecular pump, is required to periodically evacuate the CCD camera.

5.2.1.7. Helijet Option

The customer should provide a minimum of 50 litres of liquid helium and a minimum of 1 full helium gas cylinder of at least 99.99% grade helium gas in order for the operation of the Helijet to be demonstrated.

5.2.2. Setting to Work

5.2.2.1. Equipment Required

Table/ shelving for low temperature device controller and a table / pedestal stand for the computer, monitor, keyboard and mouse.

5.2.2.2. Personnel Required for Installation

5 persons for lifting of heavy components

5.2.1. Installation Procedures

Agilent Technologies personnel perform the installation. The duration of the installation is typically 3 working days, with an additional 1 day for the low temperature option. This is followed by 2 days training from an Agilent Technologies applications engineer.

5.3 Storage

Before installation commences, or when the system is not being used for extended periods, store the diffractometer in accordance with the environmental conditions for temperature and humidity stated in the SPECIFICATIONS section of this manual.

Always store the Xcalibur or Gemini instrument in a secure room.

6. Operation

Xcalibur/Gemini is a computer-controlled system. All functions are controlled from the computer terminal. Power is switched on and off via manual switches located on the Xcalibur or Gemini.

WARNING

Local rules and regulations may apply to the use of the diffractometer. If these exist, refer to these local rules before operating the system.

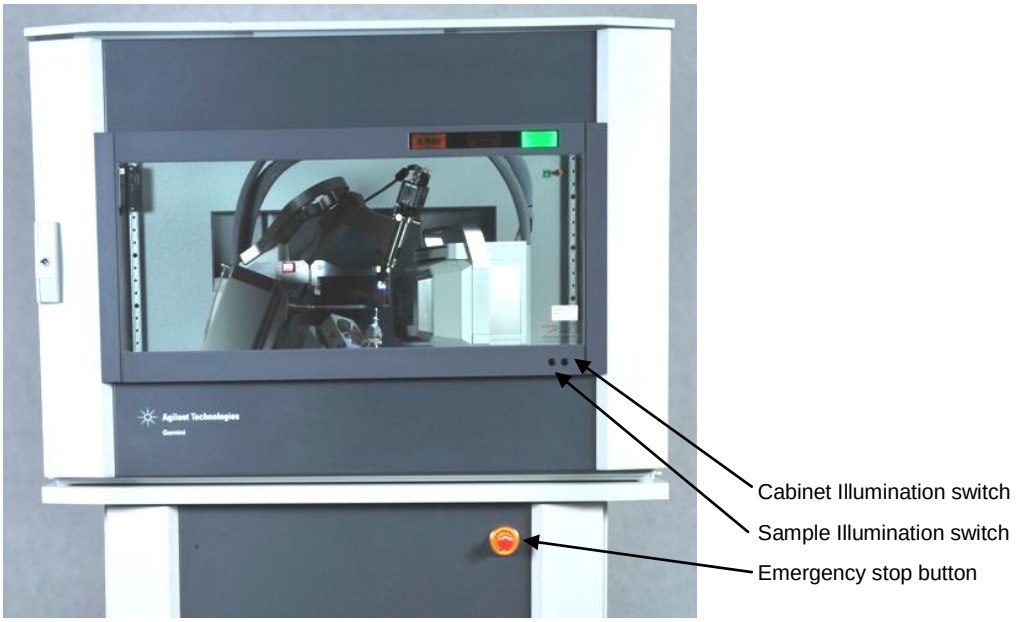
6.1 Controls and Indicators

6.1.1. List of controls

Control	Type	Location	Effect
Emergency Stop	Red button	Top right corner of front door of electronics rack	Shutdown X-ray generator power and stop goniometer
Motion Enable	Two metallic push buttons	Inside the protection cabinet, in front of the goniometer	Pushing and holding both buttons simultaneously enables goniometer movement while the protection cabinet is open
Cabinet Illumination	Small black button	Front of protection cabinet	Illuminate inside of enclosure
Sample illumination	Small black button	Front of protection cabinet	Illuminate crystal sample

6.1.2. Power

Control	Type	Location	Effect
Electrical Rack	Red switch	Top panel of rack; Inside front door of electronics rack	"Master" power on/off to whole rack, **except X-ray generator**
Diffractometer goniometer interface	Red switch	Interface front panel, left; Inside front door of electronics rack	Power on / off to goniometer
X-ray Generator	Black switch	Rear panel; inside rear door of electronics rack	Power on / off to X-ray generator
Water cooler	Red switch	Water cooler front panel, left; Inside front door of electronics rack	Power on/off to water cooler
DC power supply	Switch	Inside rear door of electronics rack, underneath power supply	Power on / off to CCD detector



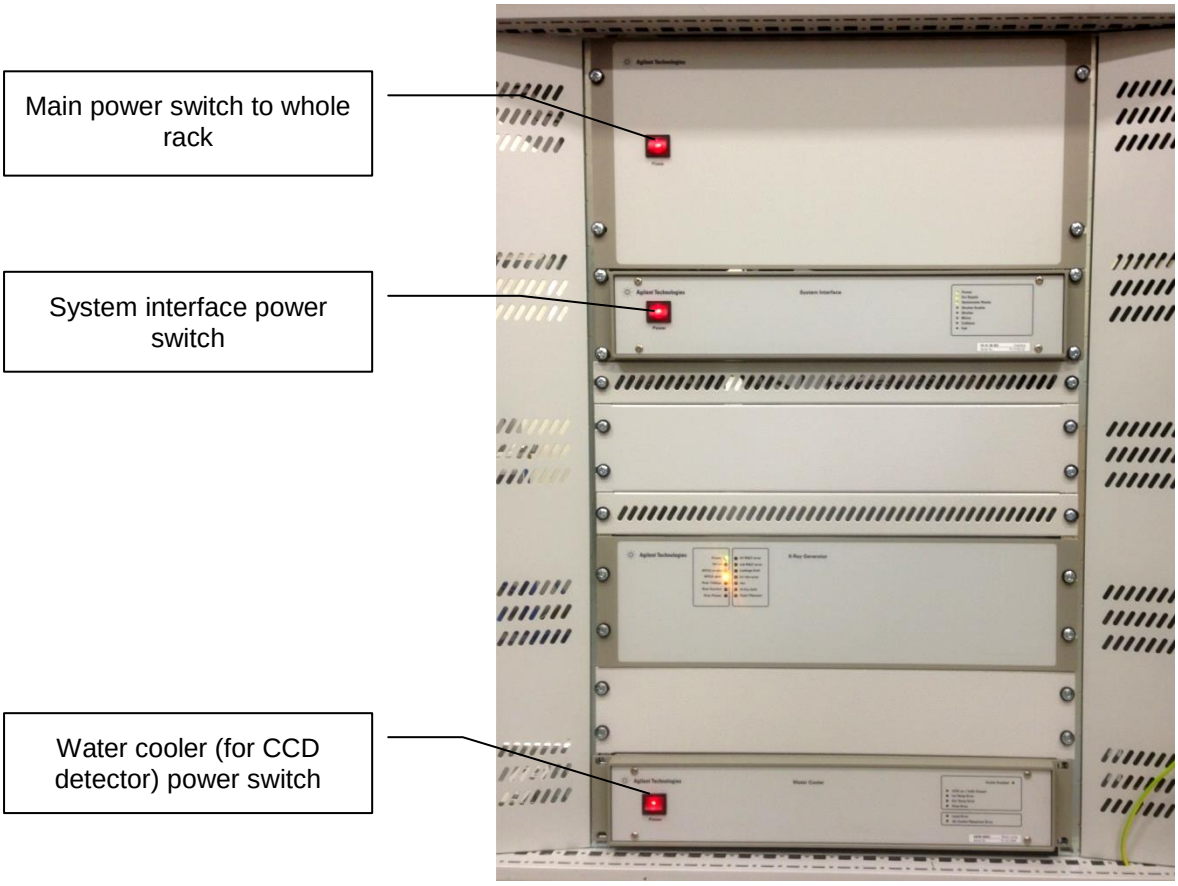


Figure 8 Location of switches

6.1.3. List of indicators

Indicator	Location	Meaning
Orange light (X-ray)	Outside, top right of the protective enclosure	X-ray power is on
Red light (Shutter open)	Outside, top right of the protective enclosure	X-ray shutter open
Green Light (Shutter closed)	Outside, top right of the protective enclosure	X-ray shutter closed
Red light (Shutter open)	Inside, on beamstop mounting arm	X-ray shutter open
Orange LEDs	Generator front panel, right; Inside front door of electronics rack	High voltage is ON. Interlock is closed thus enabling shutter operation.
Red LEDs	Generator front panel, right; Inside front door of electronics rack	Interlock is open thus disabling shutter operation. Error status as indicated.
Green LEDs	Water cooler front panel, right; Inside front door of electronics rack	Water cooler circuit (for CCD detector) is operational
Orange LEDs	Water cooler front panel, right; Inside front door of electronics rack	Water cooler circuit (for CCD detector) has closed interlock for CCD operation to be activated
Red LEDs	Water cooler front panel, right; Inside front door of electronics rack	Water cooler circuit (for CCD detector) has an error as indicated

6.2 X-ray Source

Figure 9 Two X-ray sources mounted on Gemini diffractometer

6.2.1. X-ray generation

This unit generates a focussed beam of X-rays during normal operation. The long fine focus X-ray tube generates X-rays when supplied with high voltage and filament current from the HV generator unit. The dual shutter blocks X-ray emission from the source when either one of the two shutters is closed. The shutters are positioned between the X-ray tube and the monochromator. X-rays shine onto the monochromator only when both shutters are opened.

WARNING

The X-ray tube is contained within a shielded housing. The shielding should NEVER be removed whilst the high voltage generator is active as this would cause human exposure to damaging X-rays. The tube shield housing should only be removed by Agilent authorised service provider.

The long fine focus X-ray tube is a consumable item which in time will need to be replaced. This should only be carried out by a fully trained operative and the procedure is described later in this manual.

6.2.2. Water cooling

Generation of X-rays causes the production of heat and thus the X-ray source requires water cooling. On the underside of the source unit there are two "quick connectors" for the input and output of cooling water. The direction of water flow is important and is indicated with labels. The water cooler unit should be used to supply

cooling water to the X-ray source. The HV generator unit is interlocked to the cooling unit and thus high voltage cannot be applied until cooling water is flowing normally. Do not switch off the water supply while the X-ray tube is operational since this will cause it to shut down quickly and this will shorten the lifetime of the tube. The tube should first be ramped down slowly to zero power before stopping the water flow.

6.2.3. Temperature Sensor

The X-ray source contains a high temperature cut-off switch. This switch is connected to the HV generator unit interlock circuit. If the temperature of the tube exceeds the limit then the HV generator will shut down the high voltage in order to allow the tube to cool down. Such overheating may be caused by failure of cooling water supply or by a fault inside the X-ray tube. Do not turn on the tube again until the fault has been discovered and corrected.

6.2.4. Safety shutter operation

The safety shutter will only open if the safety interlocks are closed. This shutter is automatically controlled and will close if the interlock opens.

If the beam stop is open then an intermittent alarm will sound.

The fast shutter also cannot be opened if the interlock is open. If the protection cabinet is opened while the two shutters are opened then the interlock circuit is opened and causes the two shutters to close automatically.

6.2.5. Beamstop rotation operation

The beamstop may be rotated between ‘down’ and ‘up’ positions. The ‘down’ position is for normal operation and covers the primary beam (or two beams in the case of a dual source SuperNova) to prevent them from reaching the CCD detector. The ‘up’ position moves the beamstop 90 degrees towards the rear of the instrument and thus opens access around the crystal position to aid crystal mounting, especially when mounting in liquid nitrogen (underneath Cryojet cooling) is required.

The beamstop must be moved back to the ‘down’ position before making an X-ray exposure. The beamstop contains an interlock switch which will result in a beeping noise sounding when the beamstop is in the ‘up’ position and will also prevent the X-ray shutter from opening.

The software collision model of the instrument assumes the beamstop is in the ‘down’ position. Therefore do not move the goniometer axes while the beamstop is in the ‘up’ position since a collision might occur. When moving the beamstop between positions take care not to strike the CCD detector beryllium window with the end of the beamstop. If this happens then the beamstop may become misaligned and in the worst case the beryllium window may be damaged.

The beamstop handle is rotated by hand, gripping it in the position shown in the figure below. Pulling the beamstop handle forwards and downwards will rotate the beamstop from the ‘down’ to the ‘up’ position. The opposite movement will return it to the ‘down’ position. The beamstop will be felt to click into position at both the ‘up’ and ‘down’ positions.

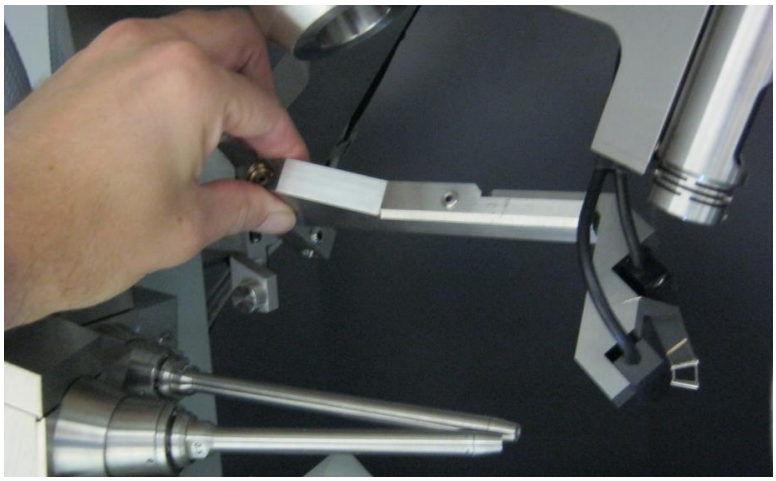


Figure 10 Rotating the beamstop position

6.3 System Start-Up

Safety devices in the diffractometer protect against damage to the system and the operator during start up. The following initial switch on procedure should be followed.

1. Plug in all mains power cords to their wall mounted sockets (system, accessories).
2. Ensure that the external circuit water valves are turned on.
3. Open all water valves on the KMW3000C water cooler (if installed).
4. Turn on the KMW3000C or TF3500 water cooler using the key switch/button on the front.
5. Open the front door of the electrical rack and switch on the top red switch.
6. Turn on the CCD Water Cooler (red switch on front panel).
7. During the start-up phase all error status LEDs will be illuminated. When they are turned off the unit is operating normally.
8. Only when start-up has completed successfully the CCD detector will be supplied with power.
9. Allow 60 minutes for the CCD detector to cool the Peltier and chip to -40°C.

CAUTION

The CCD detector requires 60 minutes cool down time. Allow another 6 hours cool down time for all internal parts to equalise in temperature and give optimum detector performance.

10. Turn on the interface using the red switch located on the left side of the interface front panel and wait 10 seconds. Ensure 2 green LEDs are illuminated on the right of the interface.
11. Confirm the PC has powered up automatically. If not then manually restart it using the front panel button.
12. Turn on computer monitor.
13. Turn on the X-ray HV generator (black switch on rear panel).
14. Launch the CrysAlis^{Pro} program. The goniometer should auto-initialise providing a system ready dialogue in the CrysAlis history window (if command prompt window is opened).

6.4 Switching the wavelength (Gemini only)

CAUTION

The wavelength switching procedure is a manual process which involves the exchange of the high voltage cables. In order to minimise wear and tear on the high voltage cables it is advised that this process should be performed not more frequently than monthly.

The Gemini system contains two X-ray sources (one Molybdenum and the other Copper wavelength). The procedure below describes how the User should switch the operating wavelength. The User should be appropriately trained to carry out this procedure.

1. The CrysAlis^{Pro} software must be running and in normal operation.
2. On the CrysAlis^{Pro} software click on the button to switch wavelength Mo/Cu.
3. The software will then open the window shown in the figure below, which guides the User through the following steps. Completing each step successfully will activate the next step.

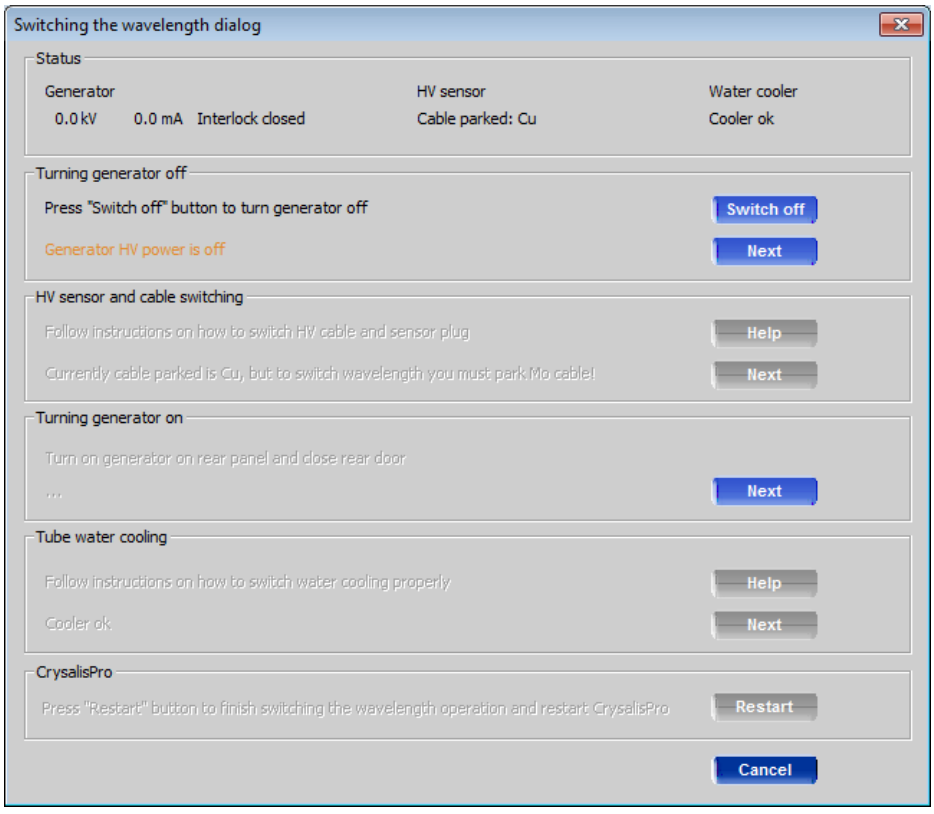


Figure 11 Software dialog for wavelength change (Gemini only)

4. Click the “switch off” button and the software will then automatically ramp down the X-ray generator to zero settings and then turn off HV. Click first “Next” button.
5. Open the rear door of the electronics rack (this will cause the HV generator interlock to open and X-rays cannot be generated).

6. Turn off the HV generator using the switch on the rear panel.

Figure 12 Rear of electronics rack showing HV cables

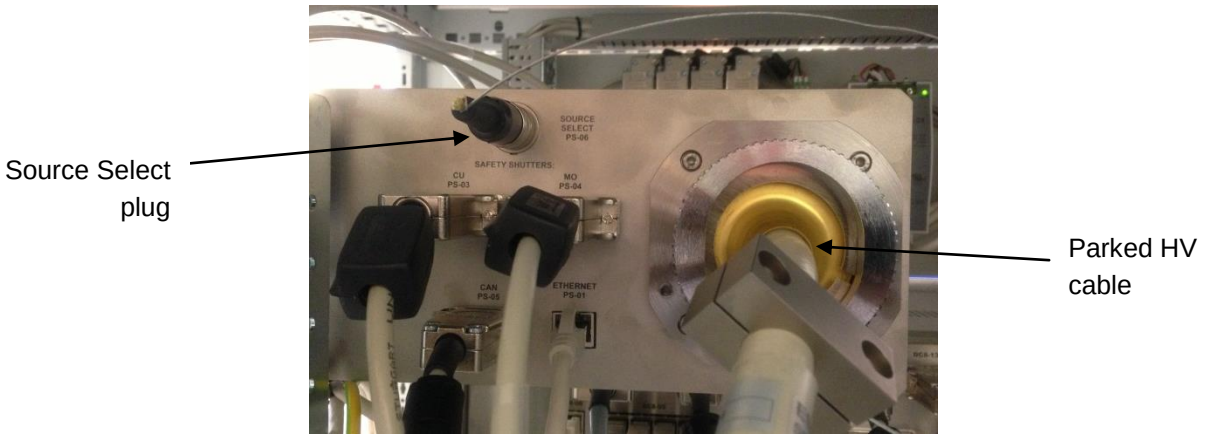


Figure 13 HV cable parking position with Source Select plug (Gemini only)

7. Unscrew the HV cable from the back of the generator.
8. Locate the second 'parked' HV cable above the system interface in the rear of the electronics rack.
9. Remove the HV cable from its parking position and also remove the indicator plug from the *Source Select PS-06* socket (see **Figure** 13).
10. Move that HV cable from the parking position down to the HV generator and screw it in.
11. Take the unused HV cable to the parking position and screw it in.
12. **Be sure to attach the indicator plug** for that cable into the *Source Select PS-06* socket next to the HV cable 'parking' position. Click second "Next" button.
13. Turn on the HV generator using the switch on the rear panel. Click third "Next" button.

14. Close the rear door of the electronics rack (this will cause the HV generator interlock to close).
15. Open the left side panel of the electronics rack by turning the locks at the top and bottom using the same key used to open the protection cabinet doors. Hold the side panel in the middle of the left and right edges and carefully lift it forwards (it is supported by two hooks inside the top edge) and then lower it to the floor. Stand the panel in a safe place where it will not fall over.

WARNING

The side panel is heavy and should be held in the middle of the left and right edges to avoid the risk of finger trapping underneath the panel. Take care to lower the panel gently to the floor without trapping hands or feet underneath.

16. Turn the manual valve (see **Figure** 14 **Water switching valve**) on the water system by 90 degrees to the position for the selected wavelength. **NOTE: the selected water valve position and the selected HV cable in the generator must be for the same wavelength.**

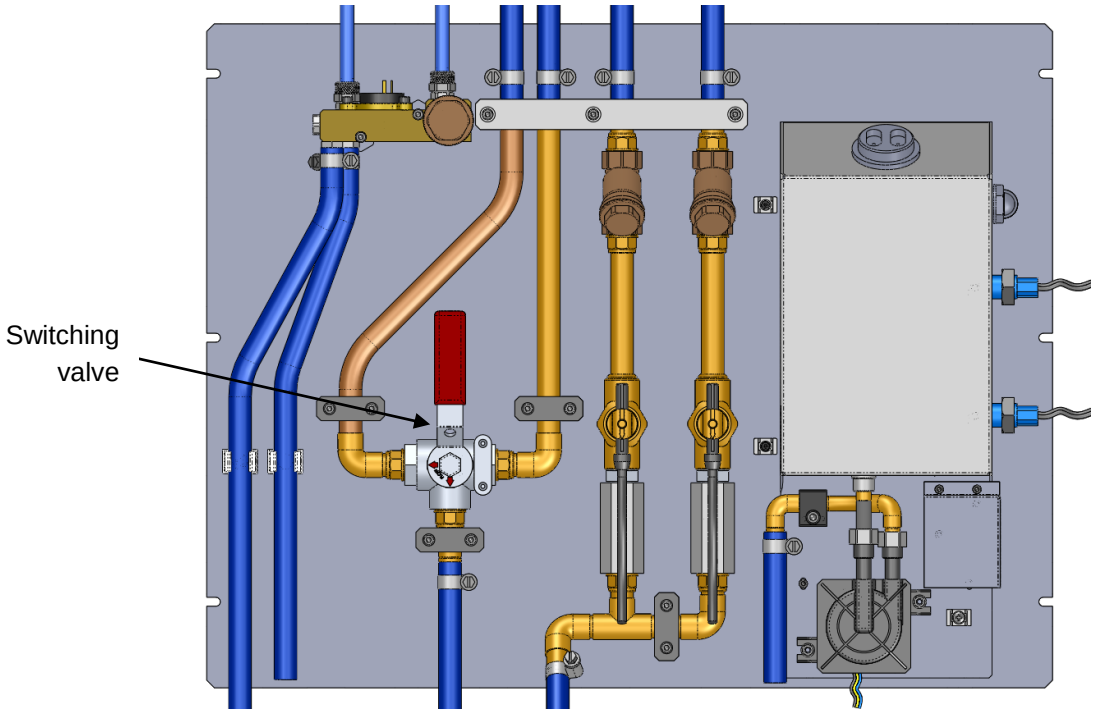


Figure 14 Water switching valve (Gemini only) in Mo position

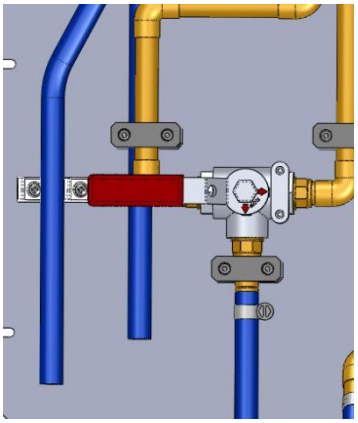


Figure 15 Water switching valve (Gemini only) in Cu position

17. Hold the side panel in the middle of the left and right edges and carefully lift it and tip the top edge towards the rack to engage it onto two hooks inside the top edge. Lower the side panel to be vertical and check it is hanging symmetrically between the front and rear sides of the rack. Lock the panel into place at the top and bottom using the cabinet key.
18. Click fourth “Next” button.
19. Click the “Restart” button to restart the software.
20. The system can then be used in normal operation through the CrysAlis^{Pro} software.

6.5 System Standby and Normal Shutdown Procedure

Depending on the period of diffractometer inactivity, follow one of the 3 procedures below.

6.5.1 Shutdown Procedure (Diffractometer not expected to be used for 2-3 days)

- Ramp down the X-ray generator to zero settings and then turn off HV using Crysalis^{Pro} software (improves lifetime of the x-ray tube).
- Park the goniometer in its home position simply by exiting the CrysAlis^{Pro} program.
- Turn off the cabinet & sample illumination using the front panel mounted switches.
- Warm up/turn off sample cooling device (following manufacturers guidelines) – where applicable.
- Turn off computer Monitor.
- Do NOT turn off the KMW3000C or TF3500 water chiller since this provides cooled water to the CCD cooler and it is advisable to keep this running.

6.5.2 Shutdown Procedure (Diffractometer not expected to be used for 1-3 weeks)

1. Follow the Shutdown Procedure (Diffractometer not expected to be used for 2-3 days).
2. Turn off x-ray generator (by using the black switch on the rear panel).
3. Turn off the diffractometer interface (red illuminated switch located on the left side of the front face).
4. Do NOT turn off the KMW3000C or TF3500 water chiller since this provides cooled water to the CCD cooler and it is advisable to keep this running

6.5.3 Shutdown Procedure (Diffractometer not expected to be used for more than 3 weeks)

1. Follow the Shutdown Procedure (diffractometer not expected to be used for 1-3 weeks).
2. Turn off the CCD Water Cooler, which in turn shuts down the CCD detector. (It is advisable to leave this item running unless absolutely necessary. Cooling the CCD detector from room temperature will be the time limiting factor when re-starting the diffractometer).
3. Turn off the KMW3000C or TF3500 water chiller using the key switch/button on the front panel.
4. Turn off system power using red switch on top panel inside front door of electronics rack
5. Turn off mains water supply.
6. Unplug system power cords – if required.

6.6 Emergency Shutdown

The emergency shutdown procedure should be used:

- If there is a fire or any other emergency requiring the evacuation of personnel from the area

6.6.1. Emergency Shutdown Procedure

WARNING

The emergency stop button will only shut down HV from the X-ray generator. Other equipment on the Xcalibur/Gemini system will still be powered on.

- Press red emergency stop button located on the front right of the Xcalibur/Gemini.
- Switch off all power at the mains electrical supply (including separately connected water chillers).
- Turn off mains water supply.

7. Maintenance Schedules

7.1 Introduction

Various maintenance tasks must be performed to ensure that the diffractometer system continues to operate safely and reliably. These tasks are detailed in the maintenance schedules given below.

WARNING

1. Maintenance tasks must only be carried out by authorised operators who have undergone specialist radiation training. Refer to local rules for further details.

2. Failure to perform scheduled maintenance tasks properly and at the correct intervals can affect the safety and performance of this system.

3. Before performing any maintenance task ensure that you have read and understood the HEALTH AND SAFETY INFORMATION at the beginning of this manual and any local rules governing the use of the diffractometer system

Planned maintenance that can be performed by the user is limited to replacing consumable items, alignment procedures, pump-down procedures, and checking radiation levels and safety features. The user's authorised service representative should carry out other tasks. If in any doubt about the performance of the diffractometer, contact Agilent Technologies.

NOTE

X-ray radiation leakage check should be performed by trained authorised personnel as often as required by local rules.

7.2 Weekly Maintenance Schedule

Tools and Materials: None

Action	Personnel	Estimated task duration	Estimated elapsed time
1. Check door safety interlocks	Authorised Operator	2 minutes	2 minutes

7.3 Monthly Maintenance Schedule

Tools and Materials: none

Action	Personnel	Estimated task duration	Estimated elapsed time
1. Check Emergency Stop	Authorised Operator	10 minutes	10 minutes

7.4 Six Monthly Maintenance Schedule

This maintenance schedule should also be completed after adjustment of beam, collimator, CCD detector position etc.

Tools:
Ylid test crystal - ($C_{10}H_{11}SO_2$)

Set of Allen keys

Action	Personnel	Estimated task duration	Estimated elapsed time
1. Refining machine parameter file	Authorised Operator	30 mins	4 – 5 hours
2. Check alignment of video microscope	Authorised Operator	30 mins	30 mins
3. Check alignment low temperature attachment (if fitted)	Authorised Operator	30 mins	30 mins
4. Cleaning water filters	Authorised Operator	30 mins	30 mins
5. Refilling water reservoir	Authorised Operator	30 mins	30 mins

7.5 Yearly Maintenance Schedule

Tools and Materials:
Special 'T' tool (carried by Agilent Technologies service engineer)
Phillips screw driver
Rotary vacuum pump capable of obtaining a pressure of $1x10^{-1}$ mbar
Vacuum tubing and adaptor

Action	Personnel	Estimated task duration	Estimated elapsed time
1. CCD detector – pump out vacuum	Agilent Engineer	1 hour	16 hours

7.6 10,000 Hours Maintenance Schedule

Please contact Agilent Technologies to perform these maintenance tasks.

Action	Personnel	Estimated task duration	Estimated elapsed time
1. Changing the X-ray tube	Agilent Engineer	1 - 2 hours	1 - 2 hours
2. Alignment of X-ray optics	Agilent Engineer	2 – 3 hours	2 – 3 hours

8. Maintenance Instructions

WARNING

1. Read and understand the Health and Safety Information section of this manual before performing any maintenance procedures.

2. Follow any local, national or international rules and guidelines that apply to this equipment when performing maintenance tasks.

3. Maintenance tasks must only be performed by authorised operators of the diffactometer.

8.1 Refining the Machine Parameter File

Task Elapsed Time: 4 – 5 hours

When: After adjustment of beam, collimator, CCD detector position etc. Otherwise twice a year.

Tools: Ylid test crystal - ($C_{10}H_{11}SO_2$)

Procedure:

See operating sections 'Refining of Machine Parameter File' and 'Service Calibration Experiments' in software manual. Please note that the test crystal must be precisely centred on the goniometer before starting this refinement/calibration task.

8.2 Aligning the Video Microscope

(Including Sample Centring and Sphere of Confusion Check)

Task Time: <30 minutes

When: When it becomes impossible to centre a sample correctly on the video microscope screen, although this can also indicate the much more serious (and unlikely) problem of the Goniometer being out of alignment.

Tools: Pre-mounted 0.3 mm steel ball, adjustment key for goniometer head

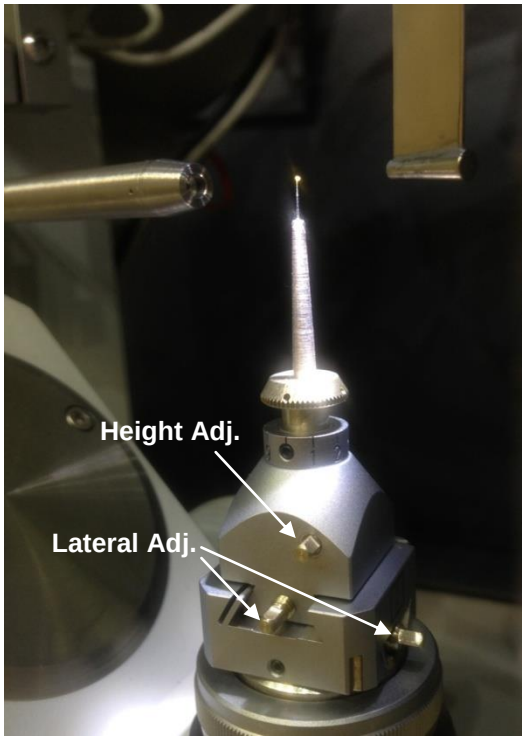


Figure 16 Goniometer Head Adjustments

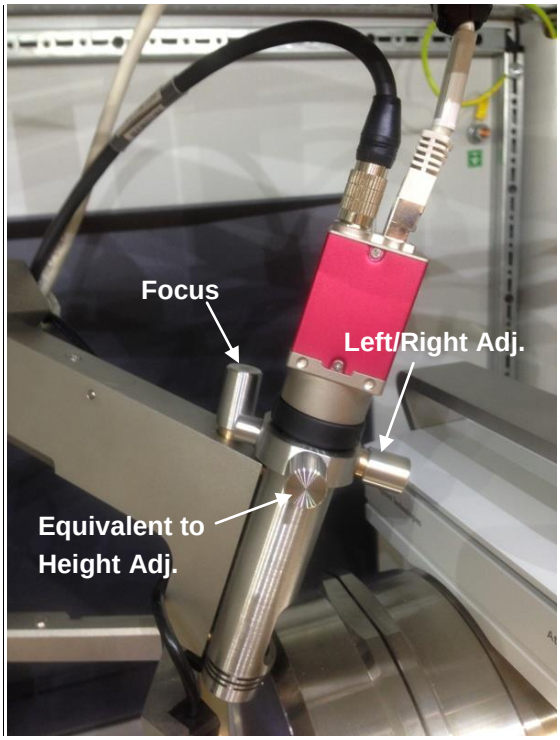


Figure 17 Video Microscope Adjustments

When first assembled the Goniometer sample centre and the video microscope focal point should be close to each other, however the field of view is approximately 1.2mm x 0.8mm and its depth of field is similarly limited. Adjustments need to be made so that the sample centre and microscope focal point coincide exactly. Both the Goniometer head and the video microscope have adjustments to move their centres in three planes. To aid this procedure a pre-mounted 0.3mm steel ball is supplied in the accessories box.

The three adjustments for the microscope can be seen in the picture above. The first is to adjust the focal length of the unit, the next has the equivalent adjustment to the height of the sample on the Goniometer (when sample is in 'Lower' or 'Upper' position). Finally there is the left / right adjustment.

To centre both the video screen and check the Sphere of confusion, use the following procedure:-

Procedure

1. Press 'F12' on the computer keyboard to start the alignment procedure (a new window opens)

2. Mount the 0.3mm steel ball onto the Goniometer.
3. Press the 'Lower' then '0' buttons and wait for the Goniometer to stop moving (note that if the cabinet is open the Motion Enable buttons will need to be simultaneously pressed).
4. Find the steel ball on the video screen using the Goniometer Head adjustments.
5. Press '180' and watch the steel ball as it is rotated, it will probably move off screen. Use the adjustments on both the Head and Microscope equally to move the ball back to the centre, press '0'. The ball should end up close to the centre of the screen, adjust again to make it central, repeat as necessary until the movement of the ball is minimised.
6. Press '90' and move the ball to the centre using the Head only.
7. Press '270' and watch the ball as it rotates. If the microscope is correctly centred left to right it will be possible to centre the ball using only the Head adjustments, if not small adjustments of the microscope may be required, repeat until the ball appears to spin on the spot for all four angle positions.
8. Press '0', then 'Upper', watching the ball as it moves. (note that if the cabinet is open the Motion Enable buttons will need to be simultaneously pressed).
9. Use the height adjustments on both the Head and the microscope equally to re-centre the ball, send back to 'Lower' and repeat until there is no movement of the ball as the Goniometer moves between the two positions.
10. Confirm that the left-right position is ok for 'Upper' as well as 'Lower'.
11. Adjust the focus on the microscope to bring the ball into sharp focus. Once this process is complete the microscope should not need adjusting again, unless it is knocked or extra weight is added to, or removed from the support arm, for example, a low temperature environment is added or removed. All sample centring should involve only the adjustments on the Head.

NOTES

An indication that the microscope is not properly centred is if the sample cannot be centred to the middle of the video screen, then small adjustments of the microscope may be needed during the sample centring process. The focus may also need adjusting occasionally if the Crystal size varies widely from one experiment to another.

For a correctly centred sample, and video microscope, the variation in position of the sample (Sphere of Confusion) as it rotates to any of the positions on the remote control (except home) should be one small division on the display (10µm). In the unlikely event that correct centring cannot be achieved contact Agilent Technologies for advice.

When fully centred in the Upper and Lower positions the Goniometer should be sent back to 'Home' once this is complete the ball (or Crystal) will appear to be displaced towards the left of the video screen. This is an optical illusion caused by the angle of the microscope with respect to the Crystal at 'Home' and can be ignored.

8.3 Checking the Door Safety Interlocks

Task Time: 2 minutes

When: Once a week

Tools: None

Procedure

WARNING

Ensure the interlocks are not defeated. If interlock defeated there is the risk of exposure to X-ray radiation.

- With the diffractometer in normal operation and at normal X-ray generator settings, with the shutter open, open the front door of enclosure cabinet. The X-ray shutter should immediately close.
- Close the enclosure door. From the software open the shutter again.
- Open the left side panel of the cabinet. The X-ray shutter should immediately close.
- Close the side panel. From the software open the shutter again.
- Open the right side panel of the cabinet. The X-ray shutter should immediately close.
- Close the side panel. From the software open the shutter again.
- Open the back panel of the cabinet. X-ray shutter should immediately close.
- Close the back panel.
- Record the date, persons testing and sign off the outcome.

8.4 Checking the Emergency stop

Task Time: 10 minutes

When: Once a month

Tools: None

Procedure

1. With the diffractometer running at minimum settings (for example 10 kV, 0.05 mA), press red Emergency Stop button.
2. The x-ray generator should switch off immediately, release the emergency stop button, restart the x-ray generator. Return to required settings.
3. The Crysalis software will indicate that the emergency stop was activated: switch off the interface, restart the interface and then type command **gon reinit** in order to restart the system.
4. Record details of the test in the local radiation log, stipulating the date, person testing, outcome and signature.

8.5 Checking the X-ray Radiation Levels

Task Time: 20 minutes

When: As required by local rules

Tools: Radiation meter

Procedure

1. With the X-ray generator on (at normal settings) and the shutter closed, use the radiation meter to sweep the area around the X-ray tube housing, fast shutter and collimator for any radiation leak (inside the enclosure).
2. Close the protective enclosure doors and open the X-ray shutter (command **sh o** in CrysAlis^{Pro})
3. Sweep the outside of the enclosure using the radiation meter, paying particular attention to the plane of the X-ray tube and the door seals.
4. Record details of the test in the local radiation log, stipulating the date, person testing, outcome and signature

8.6 Pumping Out Vacuum of CCD detector

Task Elapsed Time: 16 hours

When: Once a year

Tools: Special 'T' tool (carried by Agilent Technologies service engineer)
1 set of Allen keys
Rotary vacuum pump capable of obtaining a pressure of $1x10^{-1}$ mbar, or if available turbo-molecular pump.
Vacuum tubing and adaptor

Procedure

1. Switch off the CCD Water Cooler.
2. Wait for 12 hours for the detector to warm up.
3. Unscrew cover from pump-out port (see below). For the Eos detector this is underneath the collision protection cover which is removed by loosening four screws (as shown in below). On the S2 detector range the pump-out port is accessed from the rear end of the detector by removing a circular cover (as shown in Figure 20 below) by inserting the appropriate hexagon key and rotating anticlockwise until the cover becomes loose and drops out.
4. Screw into place the 'T' tool (the S2 detector range requires a longer version of this tool, which is inserted into the opening on the rear side).
5. Attach vacuum pump to the side arm of 'T' tool using a suitable adaptor and vacuum tubing.

CAUTION

Only use new, clean tubing. Apply vacuum before removing the CCD detector vacuum seal to prevent oil etc. diffusing into the detector and causing damage.

6. Switch on vacuum pump and leave for 5 minutes.
7. Depress 'T' tool plunger and screw into place. This screws into the o-ring sealed metal stopper that secures the detector vacuum.
8. Withdraw plunger to remove stopper. Secure plunger in place.
9. Leave under vacuum for 16 hours.
10. Depress 'T' tool plunger to replace stopper and unscrew plunger

CAUTION

Do not turn off the vacuum pump until the CCD detector has been sealed using the relevant stopper.

11. Switch off vacuum pump and disconnect pipes.
12. Unscrew 'T' tool and replace cover on pump-out port (or collision cover in the case of Eos, leaving 1mm gap to allow the cover to move). On the S2 detector range replace the circular cover on the rear side and lock it into place by inserting the appropriate hexagon key and rotating clockwise until tight.
13. Switch on the CCD Water Cooler.

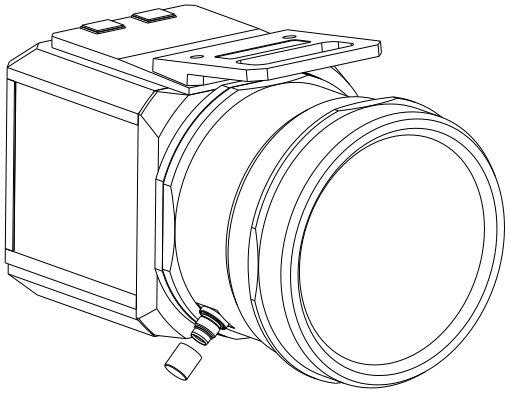


Figure 18 View of the pump out port of the Titan CCD detector

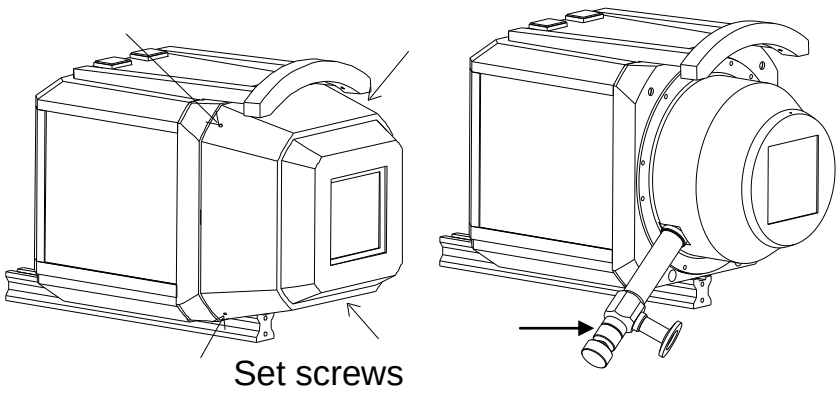


Figure 19 View of the pump out port of the Eos CCD detector

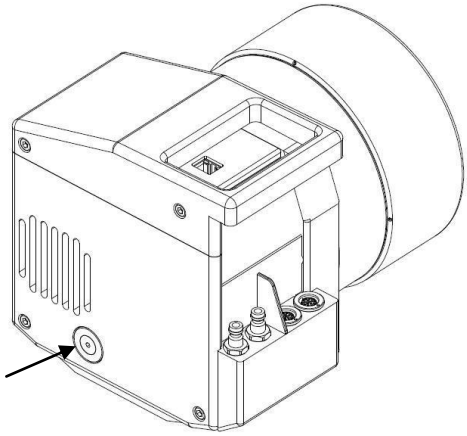


Figure 20 View of the pump out port cover on the AtlasS2 detector

8.7 Aligning Low Temperature Attachment

Task Time: 30 mins

When: When the low temperature device's cold stream is observed to be misaligned from the crystal sample which is mounted and centred on the goniometer.

Tools: 1 set of Allen keys, Helijet alignment tip, goniometer head with metal pointer

Procedure

The following instructions show how to align Helijet on the Xcalibur or SuperNova diffractometers. A similar procedure will be used for aligning other low temperature attachments. Each of them will require a different compatible support ring (to be attached to the support arm which is universal for all attachments). The support rings for low temperature attachments from most major producers can be purchased from Agilent Technologies.

For low temperature attachments, such as Oxford Instrument's Cryojet and Oxford Cryosystem's Cryostream the compatible support ring also allows the attachment to be translated up and down (towards or away from the crystal) and held in place with a side-mounted locking screw. Before proceeding with the alignment procedure ensure that the attachment has been translated to the bottom position, closest to the crystal.

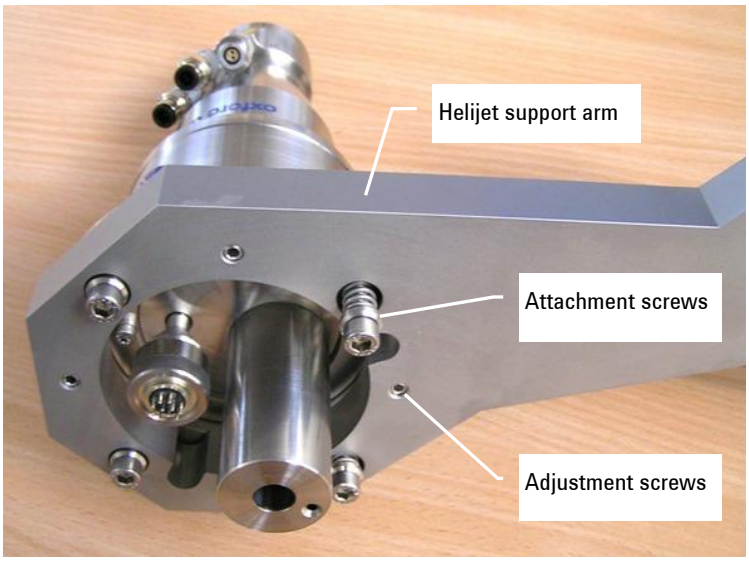


Figure 21 Helijet adapter plate screws

1. Retract the adjustment screws in the support arm so that the support ring is clamped against the underside of the support arm. The attachment screws might require tightening to provide sufficient clamping force through the springs.

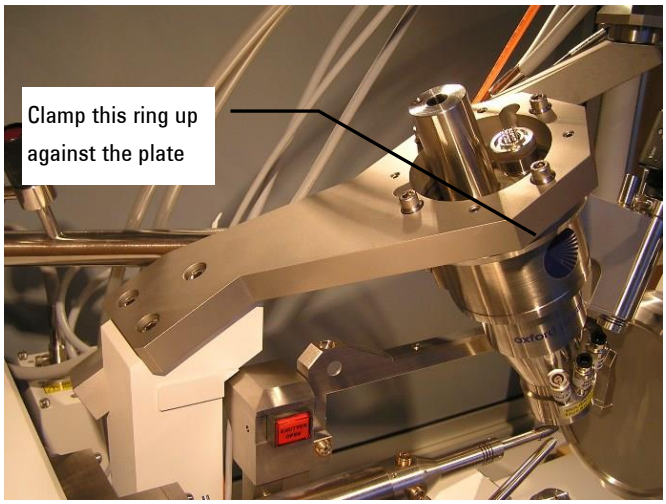


Figure 22 Starting position for Helijet alignment

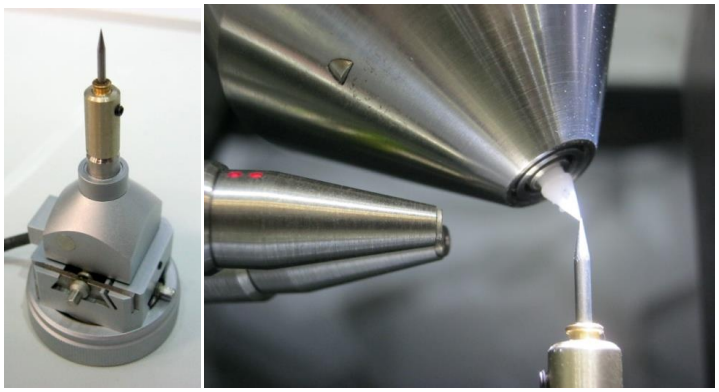


Figure 23 Helijet alignment tip

2. Place the alignment tip in the end of the Helijet nozzle.
3. Fit a metal pointer to the XYZ head (as shown in the photo above) and place on the goniometer.
4. Align optically the end of the metal pointer (using the XYZ screws on goniometer head) with the centre of the goniometer using the F12 command from the Crysalis^{Pro} software.
5. Adjust the Helijet alignment screws until the alignment tip nearly comes in contact with the end of the metal pointer. By screwing in all of the alignment screws equally the Helijet will move down towards the pointer (and vice-versa). By screwing in only one of the alignment screws the Helijet will move sideways relative to the pointer.
6. Ensure all four of the adjustment screws are touching the support ring and are not loose in space above the ring.
7. Ensure all four attachments screws are tight so that the Helijet remains clamped in the aligned position.
8. Remove the goniometer head.
9. Remove the alignment tip.

8.8 Cleaning Water Filters

8.8.1 System water filters

The system includes water filters mounted inside the left side of the electronics rack. These are visible on Figure 24. The CCD coolant filter is present on all versions of the system. Xcalibur instruments, which have one X-ray source, will have one X-ray source coolant filter and Gemini instruments will have two filters, one for each source.

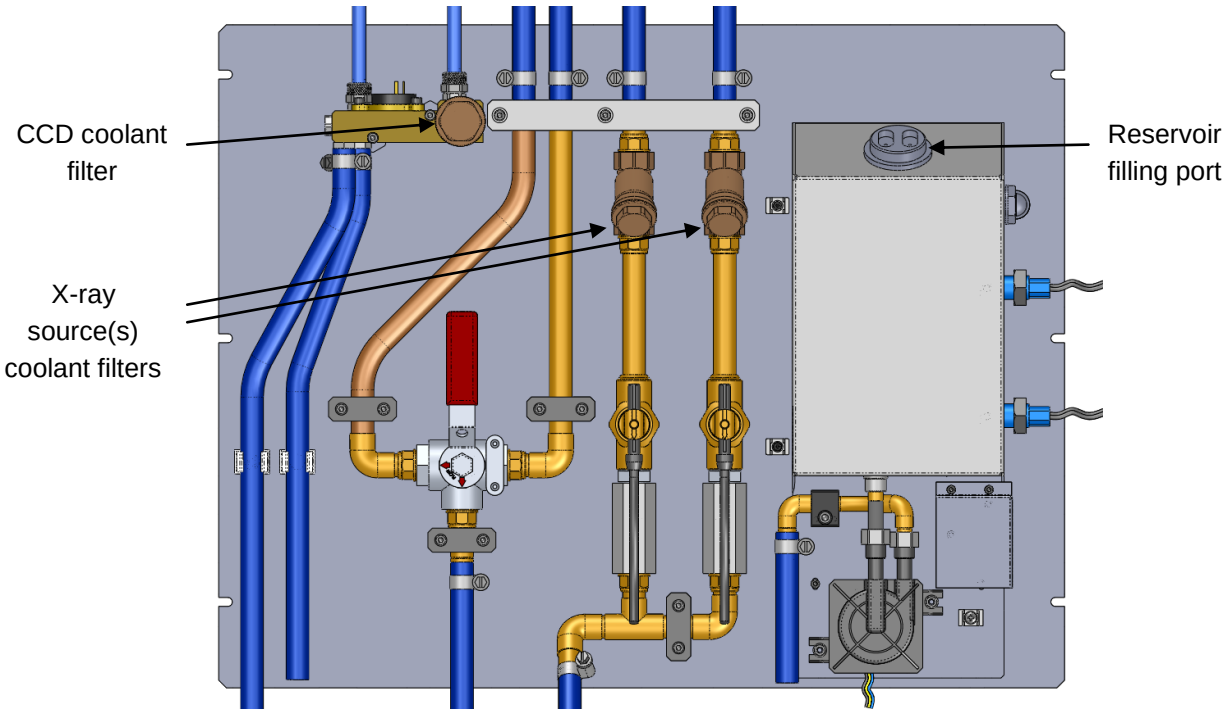


Figure 24 Water filters and reservoir

In order to clean the **CCD coolant filter** the CCD detector will need to be turned off for a period of time while the filter is opened. This will cause the CCD to warm up and therefore this procedure should not be carried out frequently, but only on a regular preventive maintenance visit or in the case of a coolant flow error on the CCD detector circuit.

Procedure to clean the CCD coolant filter is as follows:

1. Open the front door of the electronics rack
2. Locate the CCD water cooler (bottom module in the rack) and turn it off using the front panel mounted power switch
3. The CCD detector will be turned off automatically by the previous step
4. Open the left side panel of the electronics rack by turning the locks at the top and bottom using the same key used to open the protection cabinet doors. Hold the side panel in the middle of the left and right edges and carefully lift it forwards (it is supported by two hooks inside the top edge) and then lower it to the floor. Stand the panel in a safe place where it will not fall over.

WARNING

The side panel is heavy and should be held in the middle of the left and right edges to avoid the risk of finger trapping underneath the panel. Take care to lower the panel gently to the floor without trapping hands or feet underneath.

5. Locate the CCD coolant filter towards the top left of the panel which will be visible inside.
6. Using a cloth or container to catch any spilled liquid, unscrew the filter's cap (it is pointing horizontally towards the user)
7. Remove the metal filter sieve and clean it under running water and use a brush if necessary
8. Replace the filter sieve and the cap, ensuring it is fitted correctly and the o-ring is in place
9. Turn on the CCD water cooler using the front panel mounted power switch
10. Wait for the water cooler to start and after about one minute the CCD detector should re-start automatically.
11. Check the filter cap is secure and not leaking liquid. Repair if necessary
12. Hold the side panel in the middle of the left and right edges and carefully lift it and tip the top edge towards the rack to engage it onto two hooks inside the top edge. Lower the side panel to be vertical and check it is hanging symmetrically between the front and rear sides of the rack. Lock the panel into place at the top and bottom using the cabinet key.
13. Close the front door of the electrical rack.
14. Close and re-open the CrysAlisPro software to ensure re-connection to the CCD detector

In order to clean the **X-ray source coolant filter** the KMW3000 or TF3500 water cooler will need to be turned off which will remove cooling water from the external side of the CCD water cooler. After a short period of time this will result in a temperature error and the CCD water cooler will shut down and hence also turn off the CCD detector. Thus the same statement applies as above: this will cause the CCD to warm up and therefore this procedure should not be carried out frequently, but only on a regular preventive maintenance visit or in the case of a coolant flow error on the X-ray source circuit.

Procedure to clean the X-ray source coolant filter(s) is as follows:

1. Use CrysAlisPro software to turn off the X-ray source power. Wait for the active source power to ramp down to zero.
2. Wait for 20 minutes for the X-ray source to cool down
3. Turn off the KMW3000 or TF3500 water cooler using the front mounted key switch or button (the CCD water cooler will now start to warm up and result in an error after about 1 minute)
4. Open the left side panel of the electronics rack by turning the locks at the top and bottom using the same key used to open the protection cabinet doors. Hold the side panel in the middle of the left and right edges and carefully lift it forwards (it is supported by two hooks inside the top edge) and then lower it to the floor. Stand the panel in a safe place where it will not fall over.

WARNING

The side panel is heavy and should be held in the middle of the left and right edges to avoid the risk of finger trapping underneath the panel. Take care to lower the panel gently to the floor without trapping hands or feet underneath.

5. Locate the X-ray source coolant filter(s) towards the top of the panel which will be visible inside.
6. Using a cloth or container to catch any spilled liquid, unscrew the filter's cap (it is pointing diagonally down towards the user)
7. Remove the metal filter sieve and clean it under running water and use a brush if necessary
8. Replace the filter sieve and the cap, ensuring it is fitted correctly and the o-ring is in place
9. For Gemini instruments repeat for the second X-ray source filter
10. Turn on the KMW3000 or TF3500 water cooler using the front mounted key switch or button.
11. Open the front door of the electronics rack and locate the CCD water cooler at the bottom
12. Only in the case of an error light on the CCD water cooler: turn the cooler off using the front panel mounted power switch, wait 5 seconds and turn on again. After about one minute the CCD detector should restart.
13. Check the filter caps are secure and not leaking liquid. Repair if necessary
14. Hold the side panel in the middle of the left and right edges and carefully lift it and tip the top edge towards the rack to engage it onto two hooks inside the top edge. Lower the side panel to be vertical and check it is hanging symmetrically between the front and rear sides of the rack. Lock the panel into place at the top and bottom using the cabinet key.
15. Close the front door of the electrical rack
16. Close and re-open the CrysAlisPro software to ensure re-connection to the CCD detector
17. Ramp up the active X-ray source to the required power

8.8.2 KMW3000 water chiller filters

Please refer to the separate user manual for KMW3000 for information on the type of water filters and the procedure for replacing them.

8.9 Refilling water reservoir

The system includes a small water reservoir mounted inside the left side of the electronics rack. This is visible on Figure 24. The software may report to the user a water level warning, and if no action is taken, later a water level error which will shut down the water cooler for the CCD detector. The front panel of the water chiller in the electrical rack will also show this warning with a flashing LED. In case of this warning or error the user is required to refill the reservoir. The procedure is as follows:

1. Open the left side panel of the electronics rack by turning the locks at the top and bottom using the same key used to open the protection cabinet doors. Hold the side panel in the middle of the left and right edges and carefully lift it forwards (it is supported by two hooks inside the top edge) and then lower it to the floor. Stand the panel in a safe place where it will not fall over.

WARNING

The side panel is heavy and should be held in the middle of the left and right edges to avoid the risk of finger trapping underneath the panel. Take care to lower the panel gently to the floor without trapping hands or feet underneath.

2. Locate the reservoir tank which is towards the front side of the rack.
3. Open the filling port on the top of the reservoir.
4. Pre-mix a solution of 10% ethylene glycol – 90% distilled water.
5. Pour the solution into the filling port until the level is visible just under the filling port edge.
6. Close the filling port.
7. Hold the side panel in the middle of the left and right edges and carefully lift it and tip the top edge towards the rack to engage it onto two hooks inside the top edge. Lower the side panel to be vertical and check it is hanging symmetrically between the front and rear sides of the rack. Lock the panel into place at the top and bottom using the cabinet key.
8. If the water chiller of the CCD detector had shut down due to the level error, then open the front door of the electronics rack, switch off the water chiller using the front panel illuminated switch, wait 5s, and switch on again.
9. The system should then restart automatically. Observe that the level error LED is not illuminated on the front of the water chiller.
10. Close the front door of the electronics rack.

9. Trouble Shooting

Symptom	Fault	Solution
X-ray shutter will not open	1. Protective cabinet doors or side panels are not closed properly	Close doors and side panels properly
	2. X-ray generator signal light defective	Check light connections and bulb. Replace bulb
	3. Beamstop is in 'up' position	Move beamstop to 'down' position to block x-ray beam
	5. Shutter-open and /or shutter closed lights defective	Check light connections and bulbs (including the one on the beamstop mounting arm). Replace bulbs
	6. Protective cabinet doors were opened whilst the shutter was open and safety circuit was activated	In CrysAlis^{Pro} re-start experiment
Intermittent beeping sound from the system	1. Beamstop is in 'up' position	Move beamstop to 'down' position to block x-ray beam
Generator will not generate X-rays	1. Interlock is open	1. Check rear door of electronics rack is closed 2. Confirm that cooling water is supplied 3. Confirm that there is no temperature error caused by overheating the X-ray tube 4. Confirm that Emergency stop has not been pressed
	2. No power to generator	Switch on at rear panel
	3. Emergency stop has been pressed and not released	1. Twist emergency stop button clockwise to release 2. Reset generator using switch on rear panel

Symptom	Fault	Solution
	4. No cooling water through X-ray source	1. Turn on the KMW3000C or TF3500 Water Cooler 2. Confirm all water hoses are connected properly and valves are open 3. Confirm no error states on Water Cooler front panel.
	5. Cabinet X-ray warning lamp is disconnected or not functioning.	Confirm cable is connected to warning lamp (inside protection cabinet on top frame of front right door), then contact Agilent Technologies support.
Problem reading kV and mA from generator	Cable(s) not connected properly.	Check cables connected to rear of generator and serial cable connected to PC.

Symptom	Fault	Solution
Goniometer lost angles positions from memory	1. Power loss to goniometer	1. Restore power to Xcalibur/Gemini, restart the PC and CrysAlis^{Pro} program. 2. Switch off the system interface; wait 5 seconds and then switch on. Wait 15 seconds for interface to initialise. 3. In CrysAlis^{Pro} open command prompt and type **KM4 MGCUTIL** and press 'enter'. 4. Click on 'parameters' and the two 'Edit deg' buttons. Set both to 1.0000 and click on 'slow'. Click 'enter' 5. Select offending angle from the radio boxes. Click on "Set angles" and 'Start'. Input actual position of each axis, as read from the mechanical goniometer. Pay special attention to '+' or '-' 6. Select "Rsync all" and click 'Start'. Note that at the end of the Rsnync operation the "Rsync" flags should read 1. This is located on the text line below the parameter button. 7. Select "Hsync all" and click 'Start'. Note that at the end of the Hsnync operation the "Hsync" flags should read 1. This is located on the text line below the parameter button. Click on 'OK' 8. Type "gon reinit" in the command bar and press "enter". This will reinitialise all drives and leave the goniometer in its home position ready for operation.

Symptom	Fault	Solution
	2. Emergency stop button pressed	Release the emergency stop button by twisting clockwise and follow the instructions on PC screen: Power off interface; wait 5 seconds and then power on. Wait 15 seconds for interface to initialise. Type "gon reinit" in CrysAlis^{Pro} and press "enter". This will reinitialise all drives and return the goniometer to the position it was at when the emergency stop button was pressed
Goniometer movement error	Goniometer stopped after collision or blockage prevents movement	Remove the blockage from the goniometer and follow the instruction on the PC screen. Power off interface; wait 5 seconds and then power on. Type "gon reinit" in CrysAlis^{Pro} and press "enter". This will reinitialise all drives and return the goniometer to the position it was at when the movement error occurred. If the error persists then close and reopen the CrysAlisPro software. When the software initialises it will test the goniometer slits definitions and if a fault is found it will ask the user to re-run the slit definition. Follow the instructions on the PC screen.
Strange artefacts on CCD images (lines, blocks, chequer board)	1. Errors occurring on Ethernet communication between detector and PC.	1. Reset the CCD detector by switching off the Water Cooler power on the front panel, waiting 10s, and restarting. 2. Close and restart Crysalis^{Pro} and open command prompt to confirm that communication has been re-established with the CCD.

Symptom	Fault	Solution
Blank images due to X-ray shutter not opening	1. Protection cabinet doors are not closed properly.	Ensure all doors/panels are closed and secured. If the safety circuit is closed then CrysAlis^{Pro} will show this on the IO device window (click IO icon to display this).
	2. Shutter open/closed lamps are defective.	To check if LED arrays are working; turn off the interface, wait 5 seconds and turn back on the interface. For a short period all shutter indicators should be illuminated (e.g open/closed on the exterior of the cabinet and the Shutter Open (red) lamp inside the cabinet on the beamstop mounting) If the LED arrays do not function as indicated contact Agilent Technologies XRD sevice.
	3. X-ray generator is not turned on or is not generating X-rays.	1. Ensure generator is turned on and that the software indicates that X-ray power settings are normal. 2. Ensure that the orange X-ray LED array is illuminated (see section above for fault finding the X-ray generator system).
	4. Shutter signal cable	Ensure that the shutter cable (Fischer connection) is secured to the optics/shutter housing.
	5. Beamstop	Ensure beamstop is rotated into data collection position (i.e. vertical)
	6. Emergency stop has been pressed.	Ensure emergency stop has not been pressed (generator will also not function). If yes, twist to release and turn off/on the interface.
Goniometer will not move	Goniometer will not move when the cabinet is open	Press and hold the two Motion Enable buttons simultaneously to allow the goniometer to move.

Water Cooler:

Symptom	Fault	Solution

Unit cannot be turned on from the front panel	Mains power is not connected	Connect mains power at the rear panel.
Level error LED lights after switching on module	Reservoir water level is too low	Fill the Reservoir with coolant (pre-mixed solution of 90% distilled water and 10% of glycol).
Flow error LED lights after switching on module	Some air bubbles remain in the water circuit	Turn off and on the device. If needed, repeat the procedure a few times to get rid of the air in hoses and pipes.
Flow error LED flashes during operation	Liquid cannot flow through the internal circuit (through CCD detector)	Check if the hoses are properly connected and valves are open. Check if the filter is not blocked and clean it if necessary.
Reservoir error LEDs lights	Reservoir cable is not connected	Turn off the Water Cooler power supply at the front panel. Connect Reservoir cable to the unit. Turn on the unit.
Int/Ext Temp Error LED lights	Liquid in the circuit is too hot	Check that the KMW3000C or TF3500 is operating without error. Check water valves are open and that water can flow in series through the X-ray tube and the SBC Water Cooler in the electronics rack. Reduce the temperature of water in the external circuit (by changing the thermostat setting on the KMW3000C or TF3500).

10. Spares

10.1 Fuses

Device	Location	Value	Package	Designators
Power distribution	Electronics rack (right side)	10 A / 250 V fast	5x20 mm	F1
Chiller	Back of the chiller	3.15 A / 250 V fast		F1

10.2 Bulbs

Device	Location	Description
Protection cabinet X-ray and shutter indicators	On cabinet front door	M-LIGHT LED modules (yellow, green, red)
Shutter open indicator	On beamstop base	MultiLED T5, 5 RED 12 V / 25 mA (Swisstac)
Protection cabinet illumination	Inside (top) protection cabinet	MEGA X1R WHITE LED
Sample illumination	On beamstop arm	LED WHITE 10MM 6DEG

11. Disposal Instructions

11.1 X-ray Tube and CCD Detector

The X-ray tube and CCD detectors have beryllium windows. Dispose of Beryllium in accordance with local government regulations.

11.2 Third Party Equipment

Refer to third party manuals for information about disposing of third party equipment.

12. Additional Information

12.1 Third Party Information

Information marked optional is only supplied when that option is fitted to the system.

Title	Supplier	
Material Safety Data Sheet M10 Beryllium	Brushwellman	
Cryojet User Manual	Oxford Instruments	optional
Cryostream User Manual	Oxford Cryosystems	optional
Helijet User Manual	Agilent Technologies	optional

12.2 Drawings

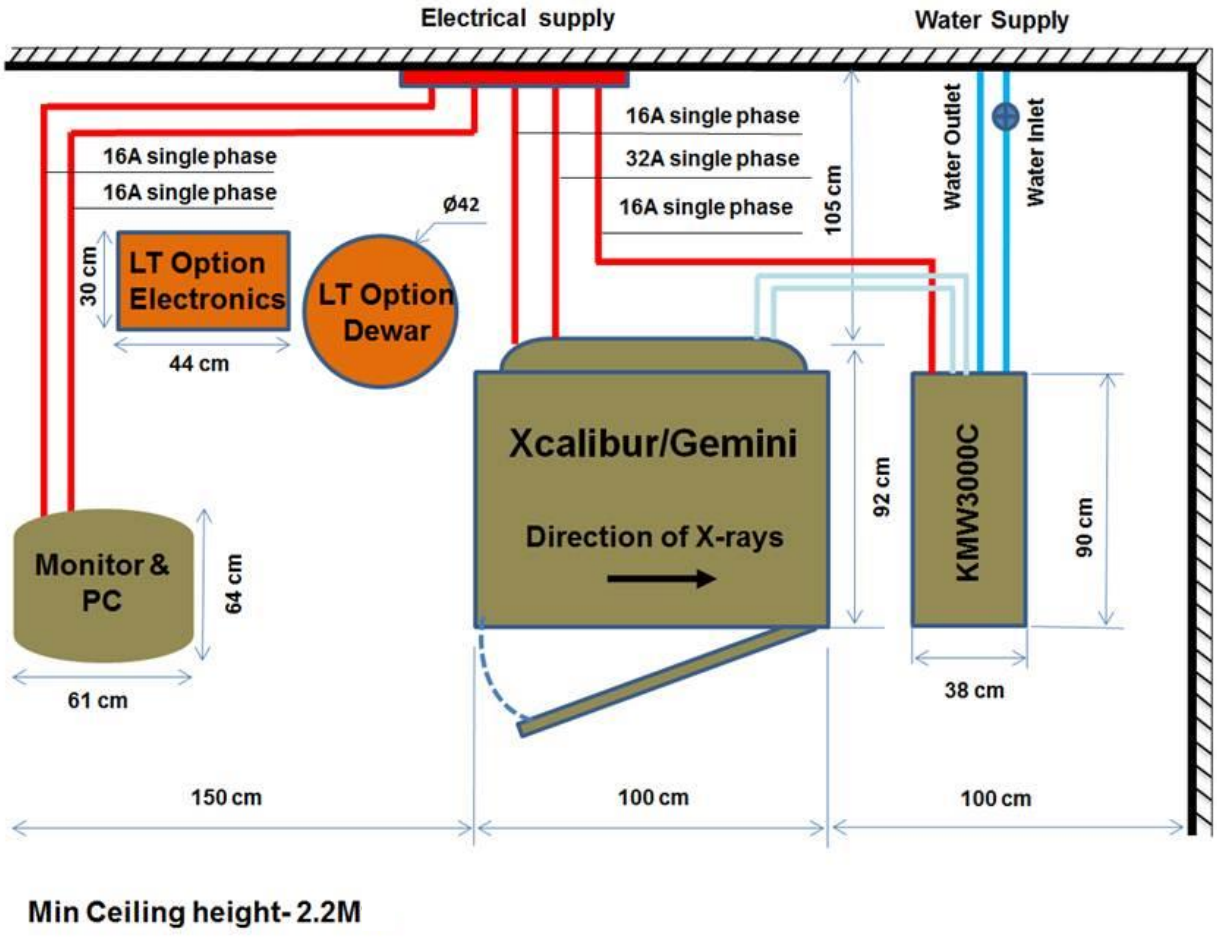
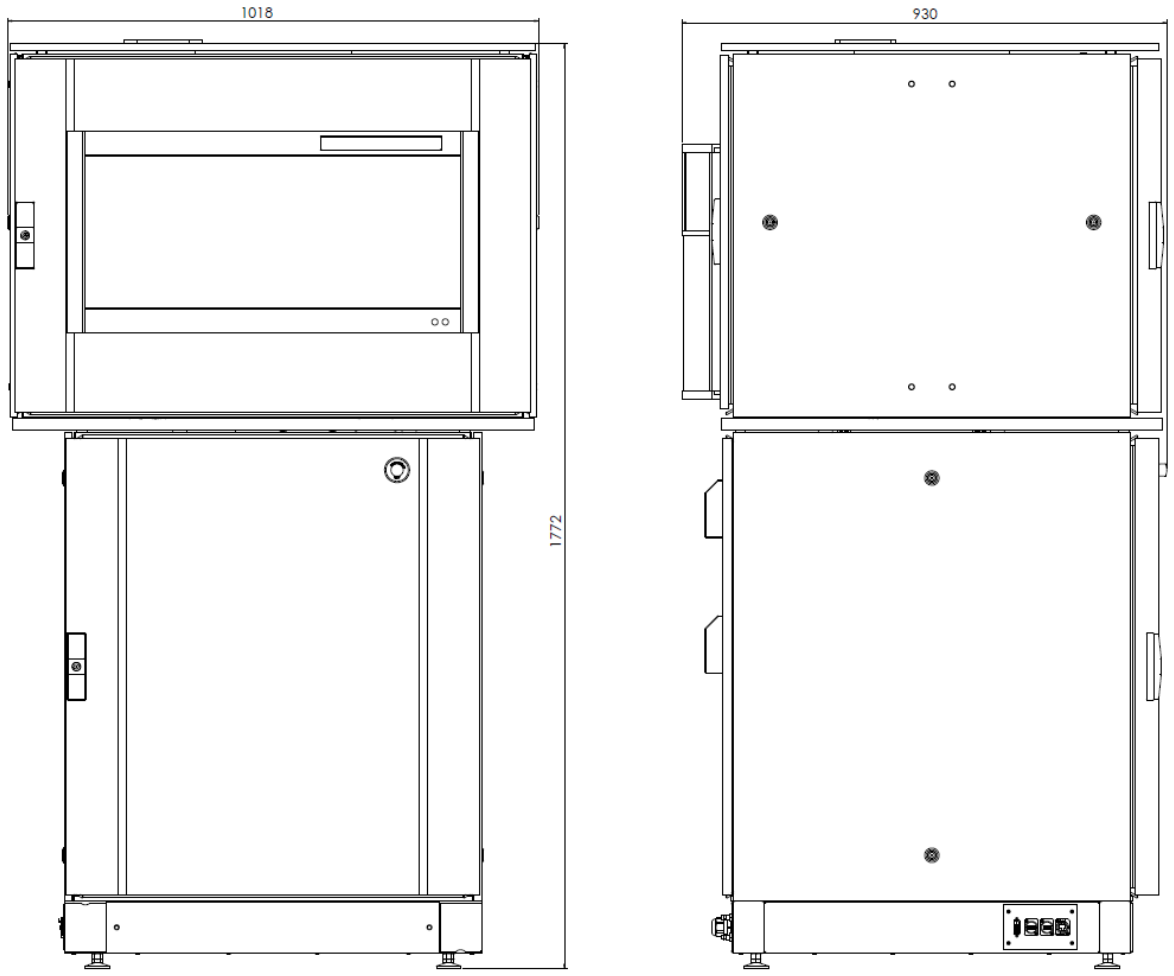


Figure 25 Xcalibur/Gemini Suggested Layout

Note the figure above shows the suggested location for the computer, monitor and keyboard based upon the cable lengths supplied and the need to access the left side panel of the instrument to change the wavelength in the Gemini configuration. The customer should provide a suitable table or pedestal for the User to work in front of the monitor.



Dimensions in mm

Figure 26 Xcalibur/Gemini System Dimensions

Appendices

Appendix 1 X-ray Tubes Wavelengths

Anode	$K\alpha_1$	$K\alpha_2$	$K\beta_1$	$K\alpha_1/K\alpha_2$
Cr	2.28976(2)	2.293663(6)	2,08492(2)	1.00170
Co	1.78901(1)	1.79290(1)	1.62083(2)	1.00217
Cu	1.540598(2)	1.544426(2)	1.39225(1)	1.00248
Mo	0.7093187(4)	0.713609(6)	0.632305(9)	1.00605
Ag	0.5594214(6)	0.563812(4)	0.497081(4)	1.00785
W	0.180199(2)	0.185080(2)	0.158986(3)	1.02709

Appendix 2 Standard Crystal Parameters

Compound name: Ylid (2-Dimethylsulfuranylidene-1,3-indandione)

Compound formula: ($C_{10}H_{10}O_2S$)

Cell constants: a= 5.947 Å $\alpha=\beta=\gamma=90°$

b= 9.026 Å

c= 18.399 Å

Appendix 3 Temperature Scales Conversion

To convert Fahrenheit to Celsius:	subtract 32 from F then multiply by 5/9	C=5/9(F-32)
To convert Celsius to Fahrenheit:	multiply by 9/5 then add 32	F=(9/5C)+32
To convert Celsius to Kelvin:	add 273.15	K=C+273.15
To convert Fahrenheit to Kelvin	first convert F to C then add 273.15	

Appendix 4 Maintenance Records

The attached record sheets are provided as examples of the type of records of maintenance checks that should be completed for Xcalibur or Gemini. These records are to show only that maintenance has been completed as

directed in this manual. Other maintenance records should be kept as required by any local, national or international regulations.

Weekly Maintenance Record Sheet				
Week No.	**Date of maintenance**	**Name of person performing test**	**Signature**	**Comments**
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				

Weekly Maintenance Record Sheet				
Week No.	**Date of maintenance**	**Name of person performing test**	**Signature**	**Comments**
23				
24				
25				
26				
27				
28				
29				
30				
31				
32				
33				
34				
35				
36				
37				
38				
39				
40				
41				
42				
43				
44				
45				
46				

Weekly Maintenance Record Sheet				
Week No.	**Date of maintenance**	**Name of person performing test**	**Signature**	**Comments**
47				
48				
49				
50				
51				
52				

Monthly Maintenance Record Sheet						
Month	**Date of maintenance**	**Name of person performing test**	**Signature**	**Emergency Stop Check OK? Y/N**	**X-ray radiation check OK? Y/N**	**Comments**

Six Monthly Maintenance Record Sheet			
Date of maintenance	**Name of person performing test**	**Signature**	**Comments**

Yearly Maintenance Record Sheet					
Date of maintenance	**Name of person performing test**	**Signature**	**Machine parameter file refined? Y/N**	**CCD detector vacuum pumped? Y/N**	**Comments**

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