D8 VENTURE

- User Manual

Part Number: DOC-M86-EXX188 V2
Publication Date: 28 September 2016

Innovation with Integrity

SC-XRD

This document covers the D8 VENTURE system.

References to this document should be shown as DOC-M86-EXX188 D8 VENTURE User Manual.

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Printed in the U.S.A.

Revision History

Revision	Date	Changes
0	18 January 2012	Initial release.
1	02 March 2012	Revised and expanded.
2	28 September 2016	Revised and expanded.

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1 About This User Manual

This User Manual is designed as a quick and easy guide to starting the D8 VENTURE diffractometer and collecting data on a crystal standard using the APEX3 or PROTEUM3 software. It is intended for those unacquainted with these Bruker products, and for those interested in getting an introductory experience with them.

1.1 Referenced Documentation

The following Table contains a list of documentation referenced by this document. It is necessary to have this additional documentation available as you work with this document. In the document part numbers, a variable revision number is given immediately after the part number. Always use the most current revisions available.

All of the documents shown may be found at Bruker's comprehensive support site www.brukersupport.com, or on the Online Documentation CD-ROM that accompanies the shipment.

Table 1.1 — Referenced documentation

Part Number	Title
<none as of this publication>	

1.2 Terms and Conventions

1.2.1 Typographical Conventions

Table 1.2 shows typographical conventions used to help quickly locate and identify information in this document.

Table 1.2 — Typographical conventions

Convention	Usage
boldface	Software user interface controls (such as icons, menu items, and buttons) to be selected as part of the current procedure.
italics	New terms and words requiring emphasis.
`monospace`	Information read from or entered into a field or command prompt.
>	Navigation through a hierarchical menu. For example, "Choose **Start > Programs > Bruker AXS > APEX3**" describes navigating Windows' menus from **Start** to **Programs** to **Bruker AXS** to **APEX3**.
[square brackets]	Keyboard input.

1.2.2 Equivalent Terms

1.2.2.1 Greek and Roman Text

This document uses scientific terminology that may be rendered in Greek text. However, this document follows a convention of using Roman text to the greatest extent possible.

Table 1.3 — Greek and Roman Text

Greek	Roman
θ	theta
ω	omega
φ	phi
χ	chi
κ	kappa

1.2.2.2 Frame/Image

In 2-dimensional X-ray diffraction, the terms *frame* and *image* refer to the same 2-dimensional view of the diffraction pattern.

1.2.3 Notices: Danger, Caution, and Note

This document contains notices that must be observed to ensure personal safety, as well as to protect the product and connected equipment. These notices are highlighted as follows according to the level of danger.

DANGER

The word “DANGER” alerts you to an immediate or potential hazard that can result in death, severe personal injury, or substantial property damage.

CAUTION

The word “CAUTION” alerts you to a potential practice or condition that could result in minor personal injury or damage to the product or property.

NOTE: The word “NOTE” in bold capital letters draws your attention to particularly important information on the product or handling of the product, or to a particular part of the product documentation.

1.3 Safety Considerations

1.3.1 Qualified Personnel

This product may only be set up and operated in conjunction with this document. Only personnel authorized by Bruker are allowed to perform installation or service on this product or its components. All repairs, adjustments, and alignments performed on any components of this product must be carried out strictly in accordance with the established safety practices and standards of the country in which the product is installed.

1.3.2 Correct Usage of this Product

This product and its components may only be used for the applications specified in its User Manual or Specification Sheet, and only in conjunction with devices or components from other manufacturers that have been approved or recommended by Bruker.

CAUTION

This product can only function correctly and safely if it is transported, stored, set up, and installed correctly, and if it is operated and maintained as recommended by Bruker. If this product is used in a manner not specified by Bruker, the product's safety features may be impaired.

1.3.3 Beryllium Safety

DANGER

This product's X-ray window contains beryllium, a toxic metal. Fumes or dust from beryllium and its compounds can be hazardous if inhaled!

Corrosion of the beryllium may occur during use.

Beryllium must not be cut, machined, or handled under any circumstances.

Disposal of beryllium must comply with all applicable national, state, and local regulations.

1.3.4 X-ray Safety

DANGER

X-ray equipment produces potentially harmful radiation and can be dangerous to anyone in the equipment's vicinity unless safety precautions are completely understood and implemented. All persons designated to operate or perform maintenance on this instrument must be fully trained on the nature of radiation, X-ray generating equipment, and radiation safety. All users of the X-ray equipment may be required by local regulations to accurately monitor their exposure to X-rays by proper use of X-ray dosimeters.

For safety issues related to operation and maintenance of your particular X-ray generator, diffractometer, and shield enclosure, please refer to the manufacturer's operation manuals or to your Radiation Safety Officer. The user is responsible for compliance with local safety regulations.

1.4 Service and Technical Support

You are invited to contact Bruker whenever there are problems or questions related to the system. Before you contact Bruker, please:

- Have the system's serial number available;
- Determine the system's software version (if you suspect a software problem);
- Record any error messages that appear; and
- Determine steps and conditions that recreate the problem (if possible).

CAUTION

Failure to refer instrument servicing to qualified Bruker personnel may result in injury or property damage!

1.4.1 North American Service Center Contact Information

Table 1.4 — Bruker AXS North American Service Center contact information

Bruker AXS North American Service Center	
Address:	Bruker AXS Inc. Customer Support 5465 East Cheryl Parkway Madison, WI 53711-5373 USA
Toll-free telephone:	1 (800) 234-XRAY [9729]
Direct line:	1 (608) 276-3000
Fax:	1 (608) 276-9162
E-mail:	customer.service@bruker.com
Web:	www.bruker.com

1.4.2 Outside North America

Outside North America, contact your local Bruker Service Center (see www.bruker-axs.com for details, or email customer.service@bruker.com).

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2 Getting Started

2.1 The D8 VENTURE Diffractometer

Figure 2.1 — D8 VENTURE system

The Bruker AXS D8 VENTURE X-ray diffractometer is optimally designed for use in all X-ray diffraction applications in chemical and biological crystallography. All D8 VENTURE goniometers are equipped with stepper motors with optical encoders to ensure extremely precise angular values. The APEX3 or PROTEUM3 software, in tandem with the programs BIS Server and Measurement Server (which support the diffractometer hardware), provides all functionality for crystal structure data collection and analysis.

The software allows remote instrument control via intranet or internet for convenient operation. The D8 VENTURE diffractometer offers full security protection according to the safety regulations in force.

The example data collection for which instructions are provided involves the use of a standard crystal (i.e., sulfonium “ylid” or hen egg-white lysozyme).

NOTE: Before starting the instrument and performing a measurement, the proper instrument installation and alignment should have been carried out by a Bruker Service Engineer.

To learn more about working with APEX3/PROTEUM3 and DIFFRAC.SUITE (which contains the Measurement Server software), refer to the User Manuals for these products.

2.1.1 Software Relationships

Bruker D8 QUEST and D8 VENTURE instruments are delivered with two software suites:

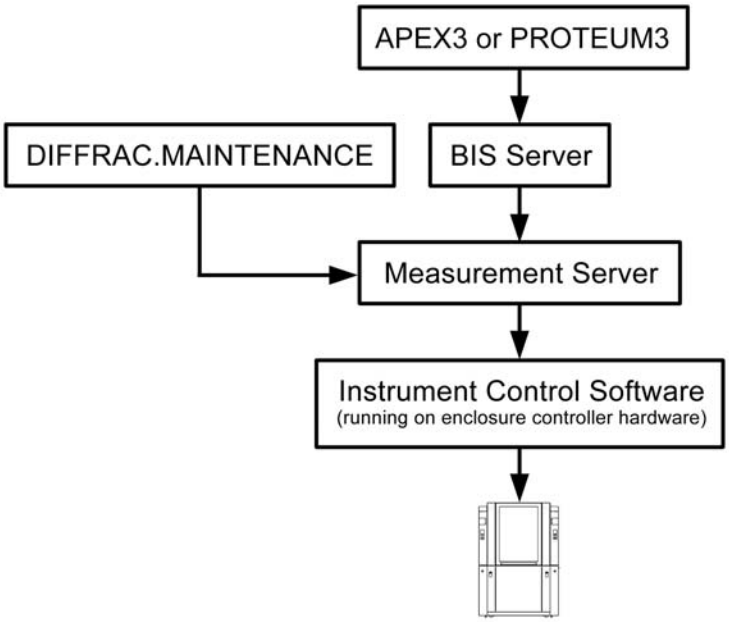
- The Bruker Suite, which includes APEX3 or PROTEUM3 and is used to perform most measurement- and analysis-related tasks.
- DIFFRAC.SUITE, which facilitates communication between Bruker Suite programs and the instrument.

Communication with the instrument takes place via a program called the *Measurement Server*, which is part of DIFFRAC.SUITE. The Measurement Server is started via a Desktop icon

, and information about the Measurement Server is shown in the Windows Taskbar's hidden icons.

Additionally, the DIFFRAC.MAINTENANCE program communicates with the Measurement Server to allow hardware control (e.g., error and alarm resolution, alignment, etc.).

Figure 2.2 — Software relationships



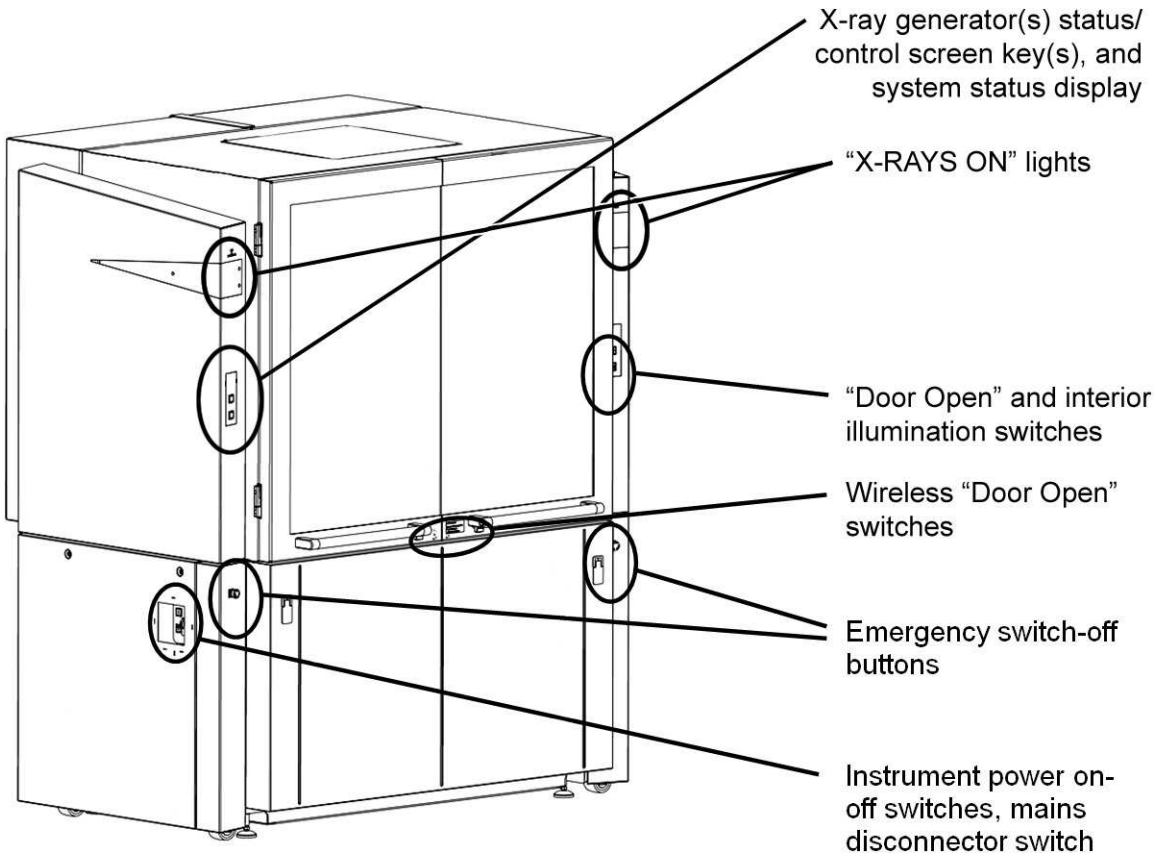
Before working with APEX3 or PROTEUM3, you must use Measurement Server to select the instrument you want to work with, establish a connection between it and the Measurement Server, and then enable the Measurement Server to get control of the instrument. This is done via the Measurement Server's "Select Instruments" and "Status Window" windows.

Once the Measurement Server is connected to the instrument, you can start BIS Server, which interprets commands sent by APEX3 or PROTEUM3. Then, in APEX3 or PROTEUM3, you can connect to BIS Server.

2.2 Startup and Shutdown

2.2.1 Start Up the Instrument

Figure 2.3 — System status and control buttons



1. Turn the mains disconnection key switch (located on the instrument’s lower left-hand side) from “0” to “I”.

   The power supply to the instrument and its electrical components is switched on.

2. Make sure that your detector’s power supply is switched on (otherwise, the Instrument Control Software cannot connect to it on startup).

3. Press the power-up button (i.e., the green button inscribed with the symbol “I”) on the instrument’s lower left-hand side.

   If an error or malfunction occurs, a symbol indicating the error will appear on the generator screen key instead of the [I] icon. Errors can be corrected using the DIFFRAC.MAINTENANCE program’s “TOOLS” plug-in.

   The generator screen key on the enclosure’s left-hand side shows white, and the system status screen key directly below it blinks white. This blinking stops after a few seconds (indicating that the system is ready), and an “I” symbol appears on the generator screen key.

4. If the system uses cooling water for the X-ray source, check for water leaks.

5. If no error has been detected (or after detected errors have been repaired), the [I] icon appears on the generator screen key(s). Press the screen key(s) to activate the X-ray generator(s).

   For a TURBO X-RAY SOURCE, the generator screen key will display status icons for vacuum pump-down and anode acceleration.

   The generator screen key turns yellow and starts blinking, indicating that the cathode is heating up. After a few seconds, the blinking stops and an X-ray symbol appears, indicating that X-ray generation has started. In this phase, the generator voltage is ramped up to a value that has been set in the hardware configuration. When this value is reached, the X-ray symbol's colors become inverted (), indicating that the generator is ready for measuring operations.

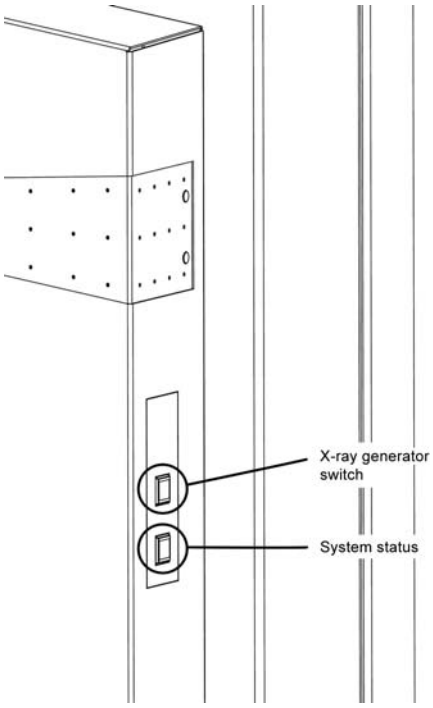
The system is now ready.

2.2.1.1 Screen Keys: Status Display and High-Voltage Operation

The system has two screen keys on the enclosure's left-hand side (systems with two X-ray sources have three screen keys: one for each of the generators, and one for the system status).

The upper screen key(s) is (are) used for high-voltage control. The lower screen key is used for system status information, and it has no function when pressed.

Figure 2.4 — Screen keys: status display and high-voltage operation (single X-ray source shown)



Upper Screen Key (X-ray Generator Status and Control)

Table 2.1 — Upper screen key states and descriptions

State	Description	Meaning
	White blank display	Shown during the instrument's startup sequence.
	Switch-on symbol	Shown when the X-ray generator is switched off and is ready to be switched on. Press once to start the X-ray generator.
	Confirm filament type	For TURBO X-RAY SOURCE, prompts you to confirm the filament type in DIFFRAC.MAINTENANCE.
	Vacuum pump-down	For TURBO X-RAY SOURCE, indicates that the vacuum is pumping down.
	Vacuum OK	For TURBO X-RAY SOURCE, indicates that the vacuum is at an acceptable level for X-ray generation.
	Anode accelerating	For TURBO X-RAY SOURCE, indicates that the anode is spinning up to its required speed for X-ray generation.
	Waiting for jet stability	The ICS is waiting until the METALJET's jet stability is confirmed; only low power (i.e., 20 W) is available.
	Heating on	Blinks yellow when the generator's tube heating is enabled and no X-rays are being generated. Constant yellow when the switch-off circuit has been closed but X-rays are not being generated. Press once to switch off the generator.
	X-rays on, generator is busy	Shown when the X-rays are switched on and the generator is ramping its power (yellow background). Press once to switch off the generator.
	X-rays on, generator ready	Shown when the generator has reached its set values (black background). Press once to switch off the generator.
	X-ray safety circuit error	The X-ray generator will be switched off if a safety error occurs. For details, examine the safety board in DIFFRAC.MAINTENANCE's "TOOLS" plug-in. If the error is fixed, the symbol will disappear and the switch-on symbol will appear (if no subsequent error is pending).
	White blinking	Safety warning is present. See DIFFRAC.MAINTENANCE's "TOOLS" plug-in for detailed information. This is not an error, but X-ray generation is not possible if this symbol is visible.
	Generator alarm	Either the generator or one of its connected components (tube, safety circuit, and water cooling system) has an error. See DIFFRAC.MAINTENANCE's "TOOLS" plug-in for detailed information. If the error is fixed, this symbol will disappear and the "switch-on" symbol will appear (if no subsequent error is pending).

State	Description	Meaning
	Generator error	Generator error. See DIFFRAC.MAINTENANCE's "TOOLS" plug-in for detailed information. Either the generator is in an undefined state, or the tube was not detected successfully.
	Cooling system alarm	The system has switched off the generator due to an error in the cooling system. For details, examine the cooling unit in DIFFRAC.MAINTENANCE's "TOOLS" plug-in. If the error is fixed, this symbol will disappear and the "switch-on" symbol will appear (if no subsequent error is pending).
	Conditioning enabled	When this symbol blinks blue, X-ray source conditioning is enabled. To abort conditioning, press the button once; the generator will switch off. You can then proceed with operation in normal mode. It is not recommended to abort tube conditioning, because this process helps to establish a longer X-ray source lifetime.
	Jet starting	The METALJET's jet is starting.
	Vacuum progress	If blinking, the METALJET's vacuum is pumping down. If steady, the vacuum is stable.
	Generator stopping	The generator is stopping.

Lower Screen Key (System Status)

Table 2.2 — Lower screen key states and descriptions

State	Description	Meaning
	White blinking blank display	The instrument is booting.
	White blank display	The instrument has booted and is ready for operation.
	Green blank display	The instrument is controlled by a client (i.e., Measurement Server)
	Measurement running	The symbol flashes blue if a measurement is in progress.
	Door open	The door is open. A measurement is only possible with closed doors.
	Sample changer error	The built-in sample changer has an error and needs a user interaction before proceeding (see the TOOLS module for a detailed description of the pending error).
	Detector error	The detector has an error.
	Drive collision	At least two drives are collided. For information on resolving the collision, see DIFFRAC.MAINTENANCE's "TOOLS" plug-in.
	Drive error	At least one drive has an error. For information on resolving the error, see DIFFRAC.MAINTENANCE's "TOOLS" plug-in.

NOTE: Pressing the lower screen key five times will display the system's IP address.

NOTE: Typically, pending system alarms or warnings (i.e., red display elements) will also appear as messages on the computer screen. DIFFRAC.TOOLS can be used to get a complete diagnosis of any pending alarms or warnings. DIFFRAC.TOOLS is also useful in resolving the alarms or warnings.

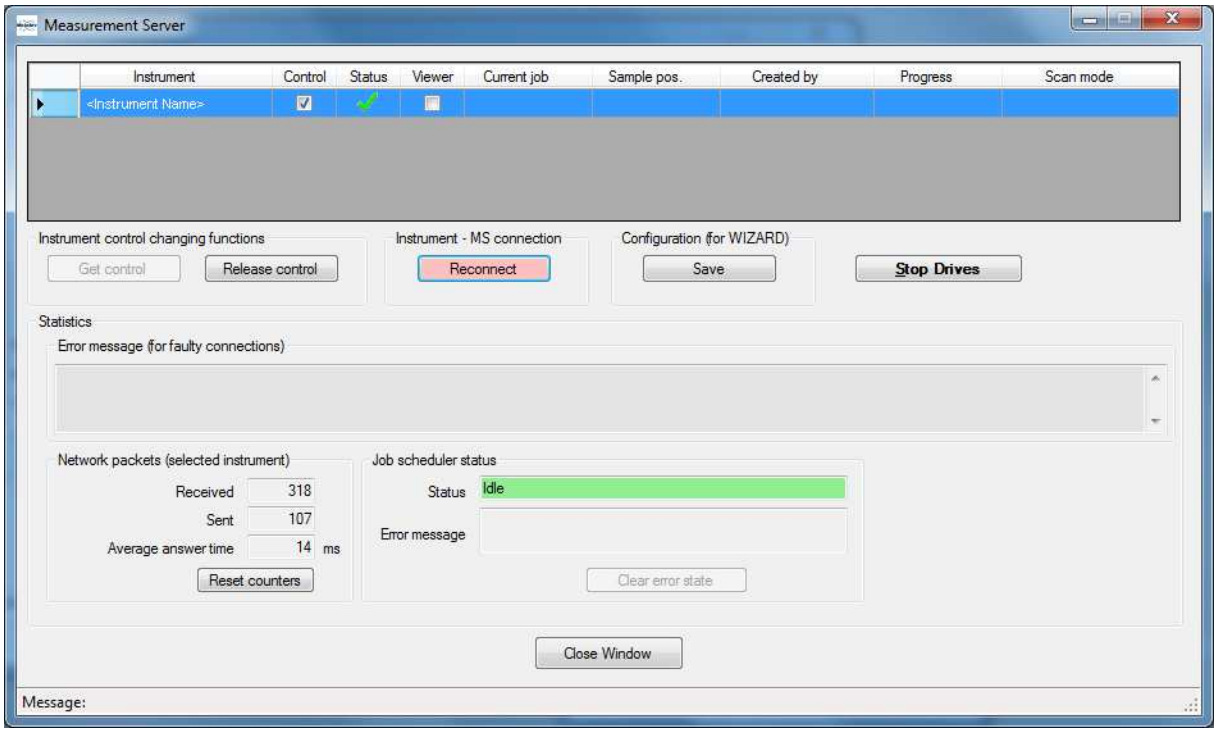
2.2.1.2 Start the Measurement Server

On the computer controlling the instrument (i.e., the single computer or the Server in a dual-computer configuration), double-click the Measurement Server icon

.

The Measurement Server starts.

Figure 2.5 — Measurement Server at startup

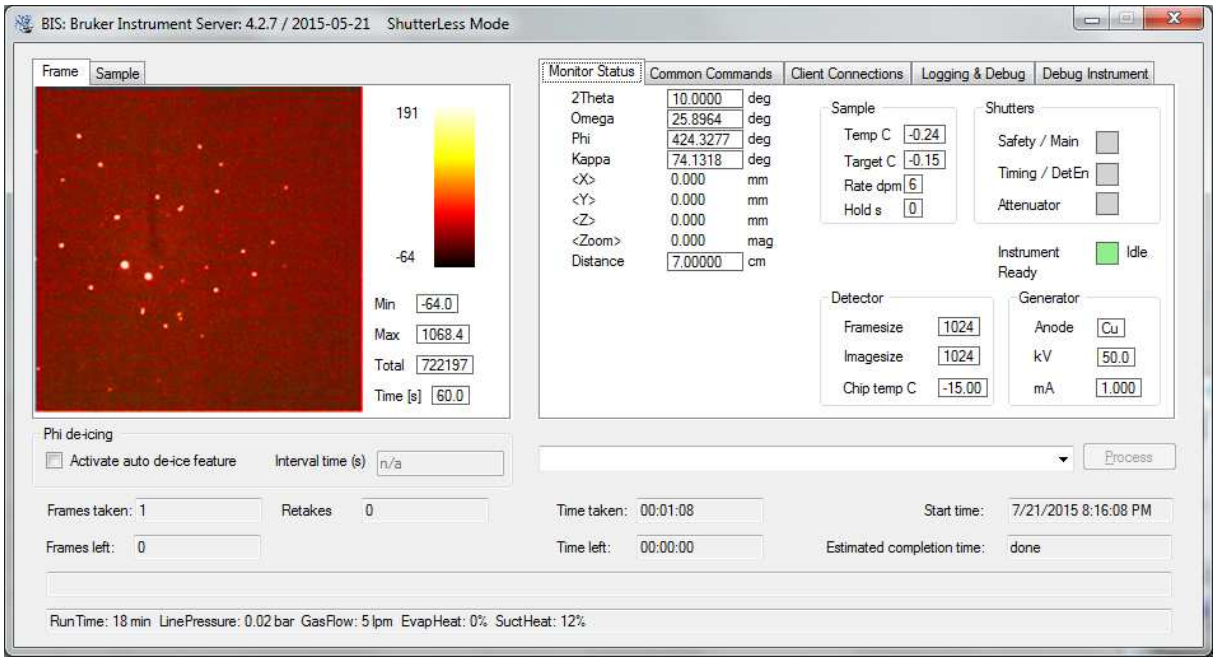


2.2.1.3 Start BIS Server

1. On the computer controlling the instrument (i.e., the single computer or the Server in a dual-computer configuration), double-click the **BIS Server** icon .

   BIS Server starts.

Figure 2.6 — Example of BIS Server window



2. Wait for BIS Server to completely initialize before connecting any clients.

   When BIS Server is completely initialized, the green status bar will disappear.

2.2.1.4 Start APEX3 or PROTEUM3

Depending on your installed software suite, start the APEX3 or PROTEUM3 application:

Start APEX3

Double-click the APEX3 icon on the Desktop (or choose **Start > All Programs > Bruker AXS > APEX3** if the license is properly installed).

APEX3's main window opens.

Start PROTEUM3

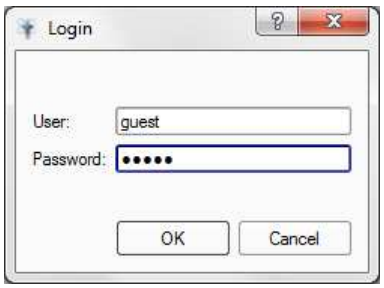
Double-click the PROTEUM3 icon on the Desktop (or choose **Start > All Programs > Bruker AXS > PROTEUM3** if the license is properly installed).

PROTEUM3's main window opens.

2.2.1.5 Log In

At the “Login” dialog, enter your user name and password (as shipped by Bruker, the default account is `guest` with password `guest`). Click **OK**.

Figure 2.7 — “Login” dialog



NOTE: If your setup does not require individual login accounts, you can configure the software to automatically log into a specified account by setting the shortcut’s target to e.g., “`C:\bn\src\scripts\apex.bat user:guest password:guest`”.

2.2.1.6 Connect to BIS Server (or BIS Classic)

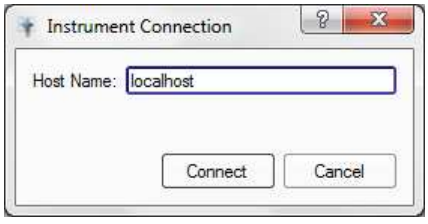
NOTE: This step is optional; plug-ins will connect when needed.

1. To use the software in online mode, choose **Instrument > Connect...**.

   The “Instrument Connection” dialog appears.

2. Enter the name (or IP address) of the computer running BIS Server or BIS Classic (the dialog shows the default host; if these are running on the same computer as APEX3, the host name is `localhost`). Click **Connect**.

Figure 2.8 — “Instrument Connection” dialog



2.2.1.7 Mount the Goniometer Head

1. Open the enclosure door(s).
2. Carefully remove the goniometer head containing the crystal from its case.

CAUTION

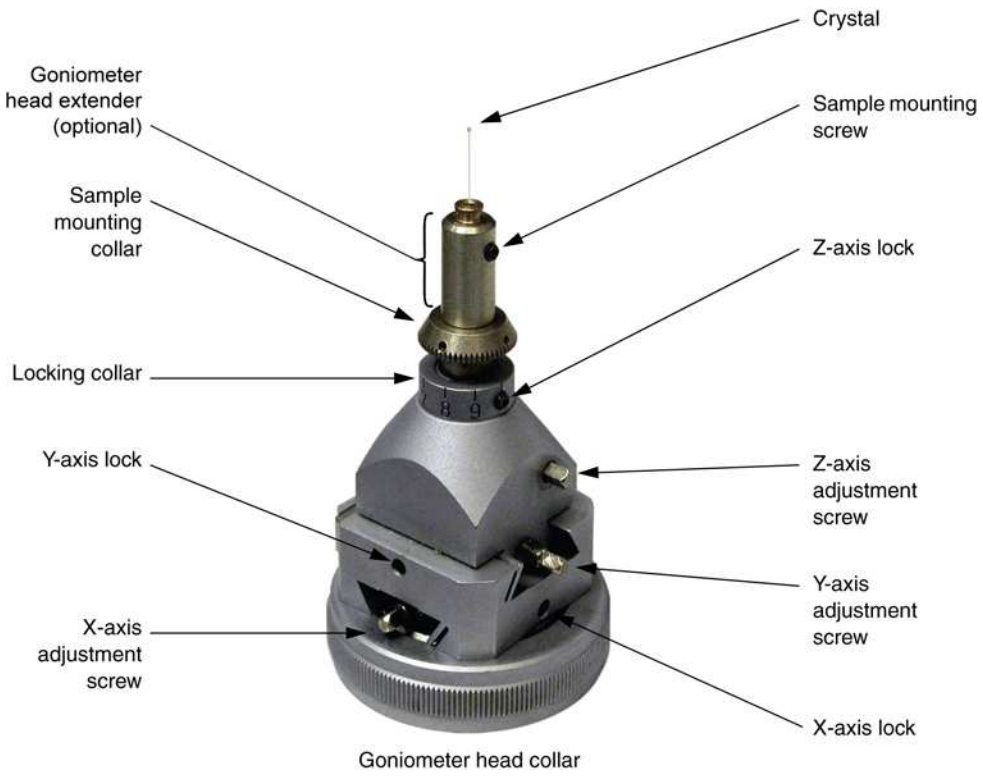
Use extreme care when handling the goniometer head to prevent damage to the crystal on the end of the small glass fiber.

3. Place the goniometer head onto its base on the phi drive. Line up the slot on the bottom of the goniometer head with the pin on the mounting base (Figure 2.9).

Figure 2.9 — Goniometer head bottom

4. Screw the goniometer head collar to the base so that the head does not move. Do not overtighten it (finger-tighten only).

Figure 2.10 —Huber goniometer head



2.2.1.8 Perform Measurements with the Instrument

Once the instrument is started and connected to the computer, you are ready to perform measurements and analyze data with APEX3 or PROTEUM3 (see the appropriate User Manual for your program).

2.2.2 Shut Down the Instrument

1. Press the generator screen key to ramp down the generator.

   The screen key's color-inverted X-ray symbol reverts to its original state as the generator voltage is reduced until X-ray generation ceases. At this point, the screen key reverts to white and the I symbol reappears. You can now switch off the instrument.

2. Press the power-down button, which is the button inscribed with "STAND BY" in the niche on the lower left-hand side of the instrument.

CAUTION

Do not press the power-down button while X-rays are being generated by the high-voltage generator, as indicated by the X-ray symbol on the generator screen key. This can shorten the service lives of the X-ray tube and the high-voltage generator.

   The instrument is powered down (i.e., switched into standby mode). The X-ray generator and all drives stop immediately.

3. Turn the mains disconnection switch (i.e., the key on the lower left-hand side of the instrument, in a counterclockwise direction from "|" to "0".

   The power supply to the instrument and all its electrical components is shut off. Having shut down the instrument in this way, you can remove the connector from the mains socket if necessary.

2.3 General Physical Principles

2.3.1 Production of X-rays

X-rays are photons that are emitted when electrons strike a metal target and interact with the electrons orbiting the metal atoms' nuclei.

In X-ray *sealed tubes*, electrons are:

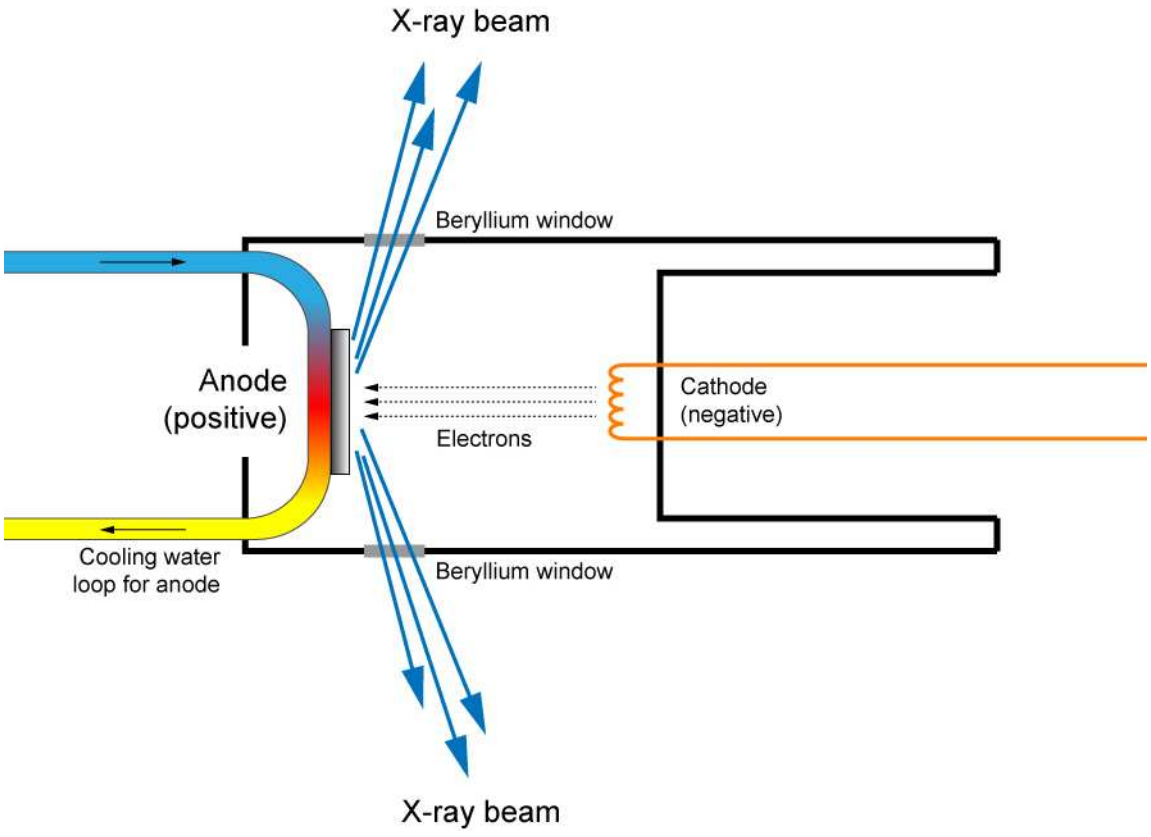
1. Emitted by a heated cathode (consisting of a tungsten filament);
2. Accelerated by a high voltage (typically 20 - 60 kV); and
3. Made to strike a metal anode (e.g., Cu, Mo, or Ag) that produces a characteristic X-ray wavelength.

The electromagnetic waves (in the λ range 0.1 - 2.0 Å) are emitted in all directions.

Much of the energy supplied to the tube is converted to heat, while less than 1% of it is converted into X-rays. A cooling system (using either water or air) prevents the tube from overheating.

X-ray tubes usually have a thin beryllium window through which the X-rays pass to the outside. The rest of the tube is surrounded by a heavy X-ray-absorbing material (e.g., lead) that prevents the X-rays emitted in other directions from penetrating to the outside.

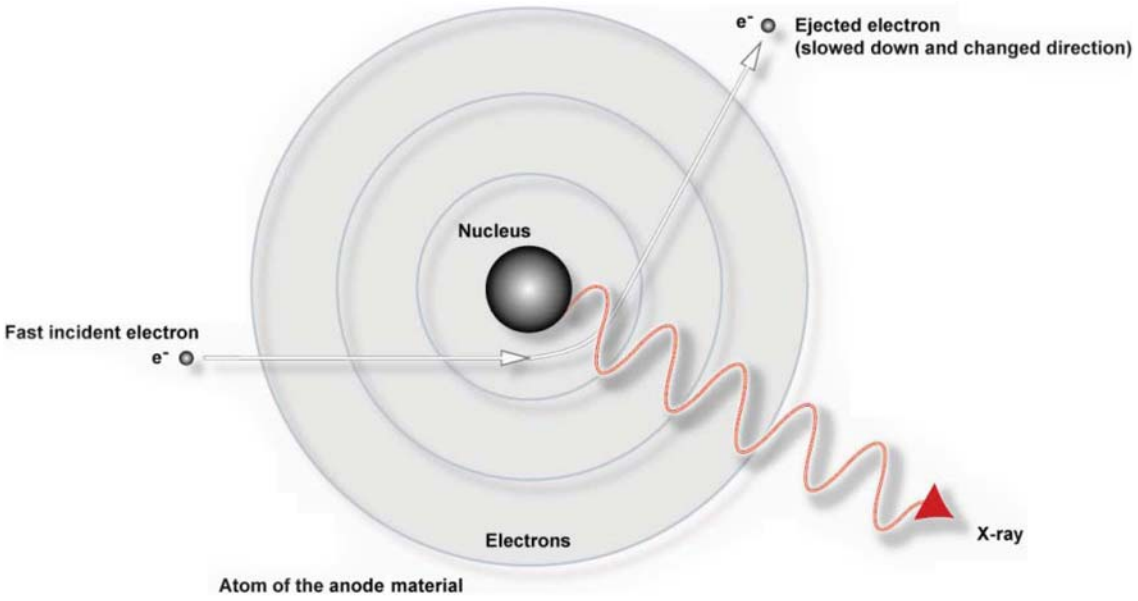
Figure 2.11 — X-ray sealed tube schematic diagram



2.3.2 Continuous Spectrum

When accelerated electrons collide with atoms in the metal target, they can be stopped on impact (in which case all their energy is converted into radiant energy), or they can be decelerated in steps (in which case a part of their total energy is released in the form of X-rays of greater wavelengths at each step until it is expended). The incident electron enters the electric field between the electrons and the nucleus of an atom and is deflected and decelerated by it at the same time. This process is repeated when the deflected electron enters the electric fields of other atoms.

Figure 2.12 —Production of continuous (white) radiation



The waves of X-rays generated in this way are called “continuous” radiation because they form a continuous spectrum of wavelengths.

2.3.3 Characteristic Spectrum

High-energy electrons can also interact with the atoms of the metal target to produce another type of radiation called “characteristic” radiation because the wavelengths of the photons emitted are characteristic of the anode metal.

In this case, the incident electron collides with an electron of the first, innermost electron shell of an atom and knocks it out of its orbit. To restore stability to the energy state of the atom, the ejected electron is replaced by an electron from an outer shell. Because the energy levels of shells increases with their distance from the nucleus of the atom, the transition of a replacement electron from an outer to an inner shell is accompanied by a loss in energy, which is released in the form of X-rays.

Figure 2.13 —Production of characteristic radiation (simplified representation)

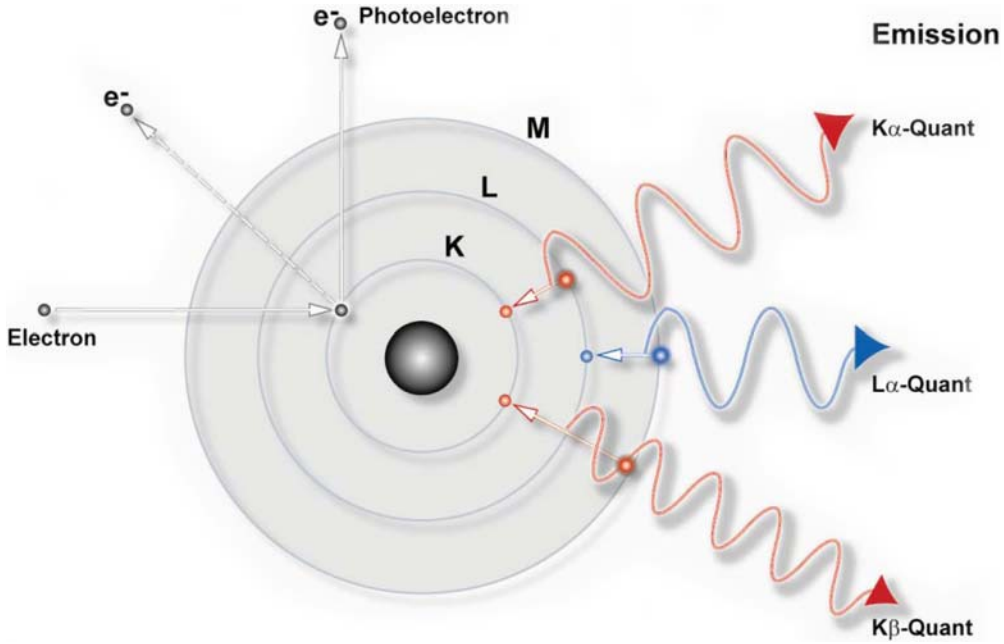
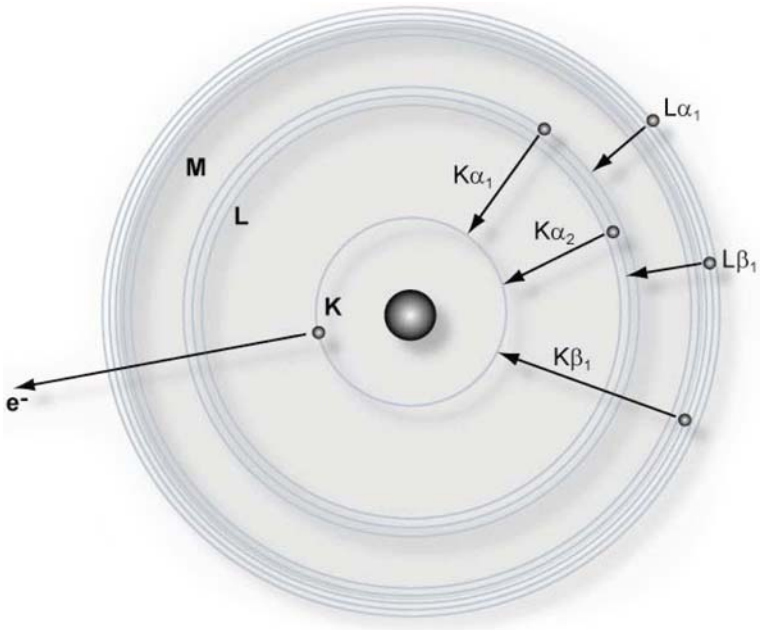


Figure 2.13 is a simplified representation of the process by which characteristic radiation is produced through the transition of electrons at the atomic level. In this diagram the conventional notation for the electron shells and the photons produced by the transition of their respective electrons has been adopted. Kα radiation is produced by an electron transfer from shell L to shell K, Kβ by a transfer from shell M to shell K, and Lα by a transfer from M to L. Note that the photons have different characteristic wavelengths.

In fact, on closer examination of the atom and its electron shells, it can be seen that the shells are divided into subshells as shown in Figure 2.14 below. For this reason, Kα radiation can be divided into $K\alpha_1$ and $K\alpha_2$ radiation, Kβ radiation into $K\beta_1$ and $K\beta_2$ radiation, and so on. The difference between the wavelengths of photons produced by the transfer of electrons of different subshells within the same shell is very small.

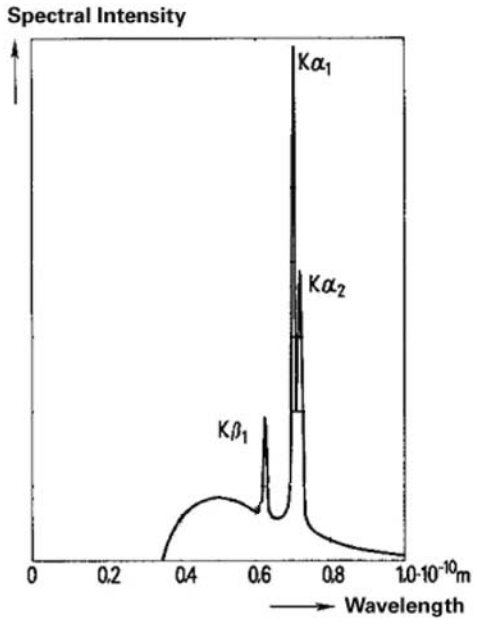
Figure 2.14 —Subshells and their transferred electrons



In Figure 2.15 below, the wavelength distribution of both continuous and characteristic radiation is shown as a function of intensity or the number of pulse counts per second. The characteristic peaks $K\alpha_1$, $K\alpha_2$, and $K\beta_1$ are clearly visible above the continuous spectrum.

The point on the wavelength scale at which the continuous spectrum begins is known as the short wavelength limit (SWL) and corresponds to the radiation produced by the electrons that give up all their energy on the initial impact. Thus for a particular supply voltage, these electrons give rise to photons of maximum energy (i.e., minimum wavelength). With an increase in supply voltage, the SWL as well as the point of maximum intensity of the continuous spectrum moves down the wavelength scale. In other words, an increase in operating potential produces a shift of the continuous spectrum in the direction of lower wavelengths and higher energy. On the other hand, the characteristic spectrum, because it is characteristic of the anode metal, merely undergoes an increase in intensity (i.e., pulse count rate).

Figure 2.15 —Spectrum of an X-ray tube



2.3.4 X-ray Diffraction

X-ray diffraction is a process in which X-rays impinging on a crystalline material are reflected by the atoms, which are arranged in ordered arrays, when certain geometrical conditions are met. The resulting X-ray diffraction pattern is characteristic of the material and can be used to identify it and elucidate the geometry of the unit cells or of its crystal structure.

Diffraction results from the phenomenon known as *scattering*. Periodically-changing electric fields of the incoming waves of X-rays excite the electrons of the atoms to periodic vibrations; these electrons then become the source of secondary X-rays with the same wavelength as the incoming X-rays (i.e., coherent scattering).

In a crystalline material, secondary radiation emanates from families of atom-containing planes in the three-dimensional crystal lattice structure. This radiation is usually weak and absorbed by the material. However, under certain conditions the waves of secondary X-rays have sufficient intensity to escape absorption and be measured by a detector. This occurs when the waves are in phase and thus reinforce one another (constructive interference). Waves constructively interfere when the incident X-ray beam strikes the surface of the crystal at a definite angle, called the Bragg angle, which is related to the distance between the planes and the wavelength of the X-rays. When the Bragg equation (Section 2.3.4.1) is thus fulfilled, diffraction occurs.

2.3.4.1 The Bragg Equation

The secondary X-ray waves emanating from a family of atom-containing planes in the crystal lattice structure are in phase and constructively interfere with one another when the Bragg condition is met. This is expressed in the equation

$N\lambda = 2d \sin \theta$

where N is an integer multiple called the *order of reflection*, λ is the wavelength of the X-ray beam, d is the distance between the planes, and θ is the angle of deflection.

The Bragg equation is derived in the following way:

Figure 2.16 —Fulfillment of the Bragg equation

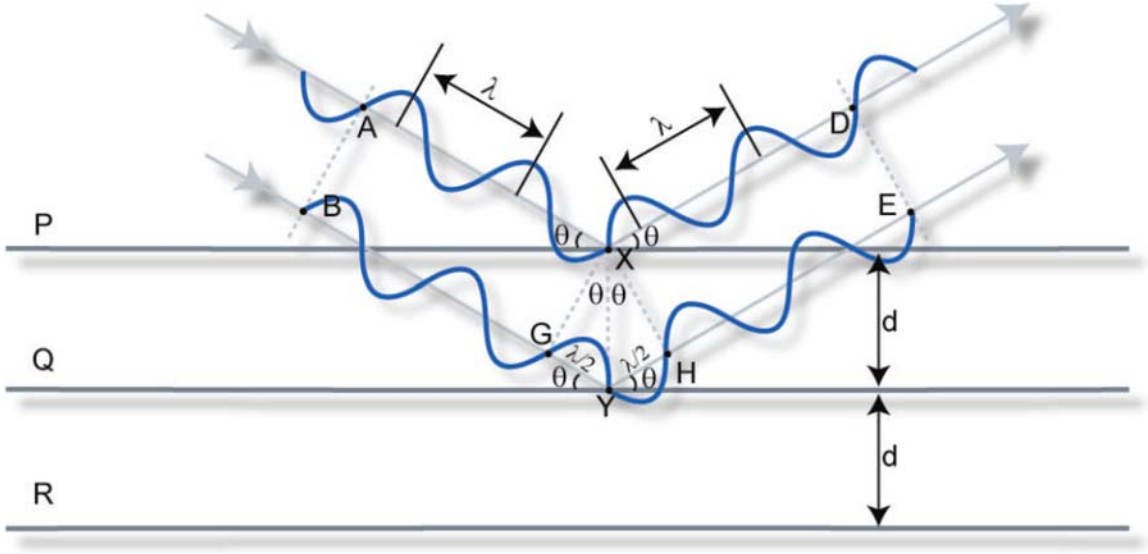


Figure 2.16 shows a monochromatic beam of X-rays incident on the surface of a crystal at an angle θ. P, Q and R represent a family of planes separated by the distance d. Plane P reflects AX in XD. Similarly, plane Q reflects BY in YE at the same angle θ. Although the beam penetrates many more planes, we need only to consider the top two.

Since Q is lower than P, the beam path BYE is longer than AXD by the amount GY + YH. This is called the *path difference*.

Since angle AXG = θ + angle PXG = 90°, then angle PXG = 90° - θ

Since angle PXY = PXG + GXY = 90°, then angle GXY = θ

Similarly, one can show that angle YXH = θ

From the triangle GXY, sin θ = GY/d, therefore GY = d sin θ

From the triangle YXH, sin θ = YH/d, therefore YH = d sin θ

Therefore, the path difference (GY + YH) = 2d sin θ

Now the two reflected rays will constructively interfere with one another, that is, they will be in synch, when the path difference is equal to the wavelength (λ) or a multiple of it. Thus $N\lambda = 2d \sin \theta$.

Once the Bragg angle θ is determined, this can be used, together with the known wavelength, to calculate the distance d and thus the geometry of the unit cells of the lattice.

2.4 Introduction to Single-Crystal X-ray Diffraction

Single-crystal X-ray diffraction, commonly referred to as X-ray crystallography, is an analytical technique in which X-ray methods are employed to determine with certainty the actual arrangement of atoms within a crystalline specimen. The science of X-ray crystallography originated in 1912 with the discovery by Laue that crystals diffract X-rays. Since that time, single-crystal X-ray diffraction has developed into the most powerful method known for obtaining the atomic arrangement in the solid state.

X-ray crystallographic structure determination can be applied to a wide range of sizes of structures, from very small molecules and simple salts, to complex minerals, synthetically-prepared inorganic and organometallic complexes, natural products, and biological macromolecules such as proteins and even viruses.

The precise knowledge of the molecular geometry is becoming increasingly important in nearly all fields of chemical and biological research. The three-dimensional atomic coordinates obtained from crystallographic studies, available in comprehensive crystallographic databases such as the Protein Data Bank and the Cambridge Structural Database, are often the starting point for most molecular modeling, drug design and molecular orbital calculations. In fact, many of most significant advances in structural chemistry and structural biology are based upon results obtained from X-ray crystallographic analyses.

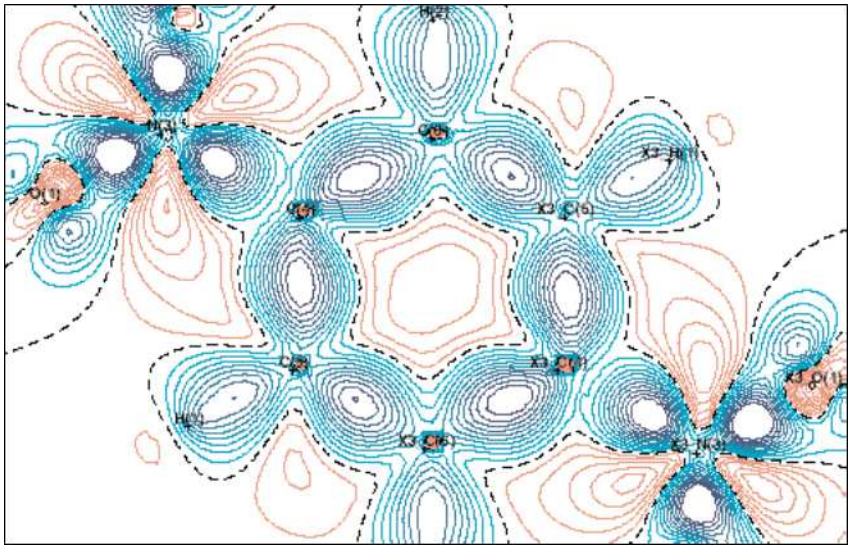
The results of X-ray crystallographic analyses are complementary to other commonly used solid-state techniques, such as X-ray powder diffraction, solid-state NMR, EPR, FT-IR and Raman spectroscopy, and neutron diffraction. Chemists also routinely use such techniques as nuclear magnetic resonance, infrared and ultraviolet spectroscopy, mass spectrometry, X-ray fluorescence, and elemental analysis for the identification and characterization of compounds prepared and isolated in their laboratories. The experimental data obtained from these techniques may, after suitable analysis and interpretation, yield important information concerning the composition and structure of the compound. However, such information is often incomplete, fragmentary and ambiguous. There are many classes of chemical compounds such as natural products, organometallic complexes, inorganic salts, metal cluster systems, organic and inorganic reaction products for which the structure cannot be deduced even with all of the other methods combined. X-ray crystallography is uniquely capable of unambiguously determining the complete three-dimensional molecular structures (including the absolute stereochemistry) of chemical substances. Modern X-ray crystallographic data is often of sufficiently good quality to permit routine location and refinement of solvent molecules and hydrogen atoms.

A significant reason for the increased use of X-ray crystallographic methods is due to the remarkable improvements in instrument performance achieved through the introduction of two-dimensional detectors, especially CCD and CMOS systems. The improvements in X-ray crystallographic instrumentation, along with associated advances in computer hardware and software have also resulted in enormous progress in efficiency and productivity. Crystal screening and identification tasks may now be performed in a matter of minutes, while analytical crystal structures may be completed in less than one hour and publication-quality structures are typically done in 3 to 13 hours, depending upon the size of the specimen. Crystallographic calculations that once required overnight execution on a mainframe computer are now easily carried out on a notebook computer. A single instrument may now be used to carry to screen and identify many thousands of samples and to determine several hundred structures per year, dramatically reducing the cost per structure.

X-ray crystallography, once regarded as an expensive and time-consuming technique used only by specialists, continues to gain new users in all branches of chemistry and biochemistry. Modern commercial instruments feature easy-to-use graphical user interfaces and automated crystallographic routines that allow routine crystal structure analyses to be carried out quickly and easily by users with only minimal training in X-ray crystallography. The high throughput and low operating costs of modern single-crystal instruments—and their ease of use—now make them suitable for use in synthetic laboratories as routine analytical tools.

The improvements in speed of data collection and data quality offered by new instrument technology have also extended the use of X-ray crystallography to a further level. Charge-density studies, which were previously limited to very small molecules due to long data collection times (often 3 to 6 months on conventional instruments), may now be carried out in less than one week on much larger molecules. At this level, it becomes possible not only to determine with very high positional accuracy the three-dimensional positions of atoms in molecules (and hence their bond lengths and angles), but also a measure of bond strength, atomic charges, dipole moments and the electrostatic potential. All of these properties are extremely important in predicting the chemical behavior of molecules.

Figure 2.17 —Electron density difference map for p-dinitrobenzene

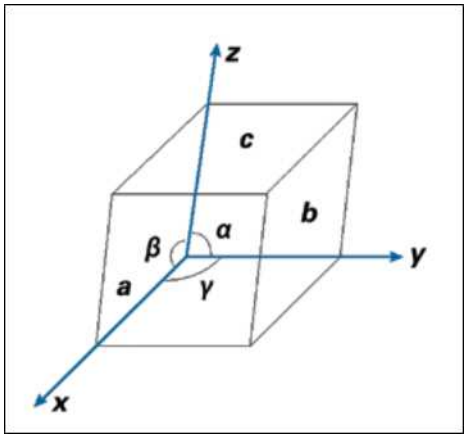


The range of materials which may be studied by X-ray crystallography has recently been extended by the availability of higher-flux X-ray sources (e.g., microfocus sources, rotating anode generators, X-ray mirror optics, synchrotron beam lines) and more sensitive X-ray detectors (e.g., CCD and CMOS detectors), by the use of high-speed computers with large amounts of mass storage, and the development of new algorithms for solving and refining large and problematic structures (e.g., twinned specimens, incommensurate structures). These advances—coupled with progress in the related fields of crystal growth, cryocrystallography, and crystal mounting techniques—now permit X-ray structure determinations to be carried out on very small specimens (minimum dimensions of a few microns), on materials with very large unit cells (maximum axis length 400Å) and on materials which are liquids at room temperature or which undergo solid-state phase changes.

2.4.1 Fundamentals

A perfect crystalline solid (i.e., a single crystal) is made up of a large number of identical molecules which are arranged in a precisely-regular way, repeated in all directions to give a highly-ordered structure. The basic building block (or motif) in a crystal is the *unit cell*. A crystal is made up of large numbers of identical unit cells arranged in a three-dimensional crystal lattice. Each crystalline substance has a unique set of lattice constants (a, b, c, α, β, γ) that define the size and shape of the unit cell (Figure 2.18).

Figure 2.18 —Unit cell nomenclature: the reference axes x, y, z are right-handed. The length of the unit cell edge parallel to each reference axis is a, b, c, respectively. The inter-axial angles are α, β, γ respectively.



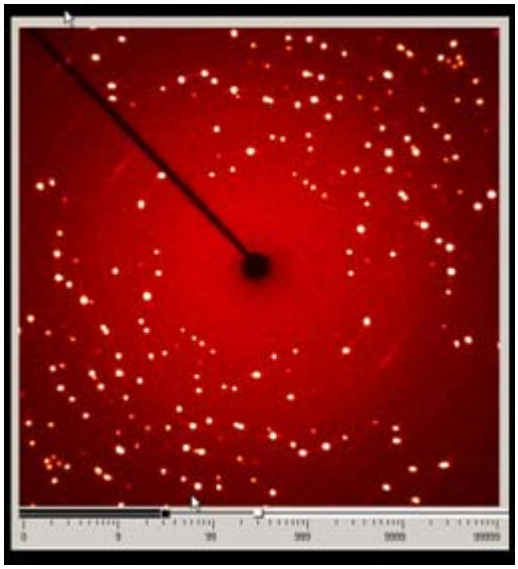
Based upon the lattice constants and the symmetry that the unit cells possess, the substance may be classified into one of seven *crystal systems*:

- triclinic
- monoclinic
- orthorhombic
- tetragonal
- cubic
- trigonal
- hexagonal

Finally, each substance may be further classified as belonging to one of 230 three-dimensional *space groups*. The lattice constants, crystal system, and space group are important physical constants for crystalline substances and may be used in conjunction with other physical measurements (e.g., density, conductivity, hardness) to explain a solid-state material's properties.

When a beam of parallel monochromatic X-rays (of approximately 1Å wavelength) strikes a single crystal, the crystal acts as a three-dimensional diffraction grating and produces an X-ray diffraction pattern (Figure 2.19).

Figure 2.19 —An example of an X-ray diffraction pattern produced by a randomly-oriented single-crystal specimen, rotated in the X-ray beam.

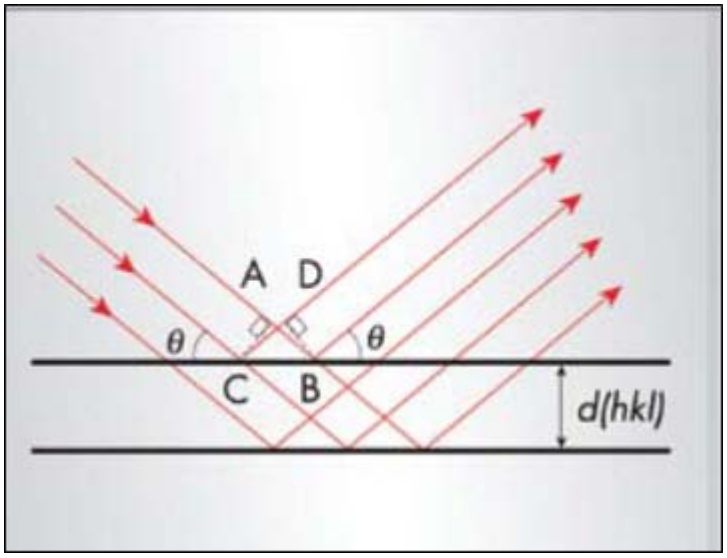


This diffraction consists of a three-dimensional array of reflections that satisfy the conditions of *Bragg's law*:

$n\lambda = 2d \sin\theta$

where n is a small integer giving the order of diffraction, λ is the wavelength of the incident X-rays, d is the distance between a set of parallel lattice planes, and θ is the angle between the incident X-ray beam and the atomic lattice plane in the crystal dimensions of the unit cell in the crystal.

Figure 2.20 —Diagrammatic representation of Bragg's Law showing the diffraction angle θ and the interplanar spacing d(hkl). Two rays reflected from the same plane do not suffer any relative phase change or path difference (i.e., AB = CD)



Each reflection may be assigned a set of indices (hkl) that indicate its location in the diffraction pattern or in *reciprocal space*. The diffraction pattern in reciprocal space has a Fourier transform relationship to the electron density in the unit cell in real space.

Experimentally, the unit cell parameters for a crystalline specimen may be determined from an analysis of the spatial arrangement of the reflections in its X-ray diffraction pattern. On modern instruments, the measurement of reflection positions and the subsequent calculation of unit cell parameters is an automatic process which is carried out in a few minutes as part of the specimen screening process. There are now several commercial databases (e.g., CSD) that contain unit cell, space group, chemical composition, and bibliographic data on hundreds of thousands of compounds for which X-ray structure determinations have been carried out. Experimental unit cell data obtained in the crystal screening process and chemical composition information for the sample being studied may be used to search the databases. If an exact match is found, the compound has been identified and further single-crystal analysis may be unnecessary.

The analysis of the positional information for reflections, often called *geometric data collection*, may be used to provide direct information concerning the size and shape of the cell. In combination with density and elemental analysis measurements, it may also yield information concerning the cell contents; however, it does not reveal the actual locations of the atoms within the unit cell.

The determination of the arrangement of atoms in the unit cell requires a very detailed analysis of the relative intensities of all unique reflections in the diffraction pattern. The precise measurement of the reflections' relative intensities is termed *intensity data collection*. The collection of accurate intensity data requires a highly-stable X-ray source, a precise mechanical goniometer for sample positioning, and a very efficient counting system. An intensity data set consists of several thousand reflections indexed by h, k and l for which an integrated intensity I(hkl) has been accurately measured.

The X-ray diffraction pattern consists of the superposition of scattered waves of varying amplitude and phase. Each diffraction maximum or reflection has associated with it a *structure factor* F(hkl) measured relative to the scattering by a single electron. The structure factor may be represented as a complex vector:

$$\mathbf{F(hkl) = A(hkl) + iB(hkl)}$$

where **A(hkl)** and **B(hkl)** are the real and imaginary components of **F(hkl)** (Figure 2.21).

The magnitude or length of the vector **|F(hkl)|** may then be represented as

$$\mathbf{|F(hkl)|} = \{[\mathbf{A(hkl) + iB(hkl)}] \times [\mathbf{A(hkl) - iB(hkl)}]\}^{1/2}$$

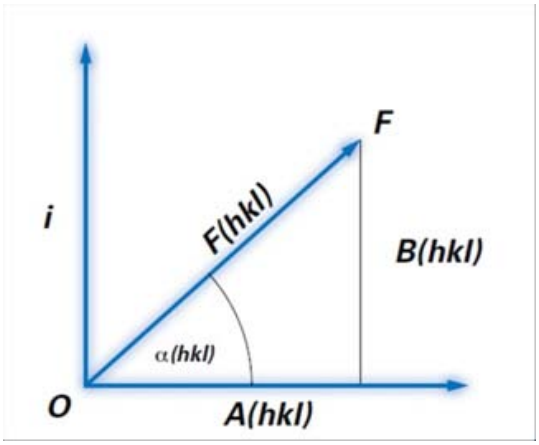
$$= [\mathbf{A(hkl)}^2 + \mathbf{B(hkl)}^2]^{1/2}$$

Alternatively, **F(hkl)** may be expressed as an exponential quantity:

$$\mathbf{F(hkl)} = \mathbf{|F(hkl)|} \exp[i\boldsymbol{\alpha}\mathbf{(hkl)}]$$

where **|F(hkl)|** is the amplitude of the scattered wave and **α(hkl)** is its phase angle.

Figure 2.21 —Structure factor **F(hkl)** plotted on an Argand diagram. **α(hkl)** is the phase angle, and the amplitude is represented by OF.



From Figure 2.21, it may be seen that

A(hkl) = **|F(hkl)|** cos **α(hkl)**

B(hkl) = **|F(hkl)|** sin **α(hkl)**

And, therefore

tan **α(hkl)** = **|B(hkl)|** / **|A(hkl)|**

|F(hkl)| may be calculated directly from the measured intensity **I(hkl)** for a reflection, since

I(hkl) = K**|F(hkl)|**^{2}

where K is a constant. However, the phase angle **α(hkl)** cannot be measured experimentally and must therefore be obtained indirectly through a variety of numerical techniques.

The central problem in the solution of a crystal structure is the assignment of phase angles to each reflection in the data set. The solution of the phase problem is considerably simplified for crystals that possess crystallographic centers of symmetry, since—to a first approximation—the imaginary components **B(hkl)** are zero for centrosymmetric space groups and the phase angles are therefore restricted to values of 0° or 180°. A structure is considered solved when a set of phase angles has been found that allows the atoms to be located and the experimental diffraction pattern to be matched to the calculated diffraction pattern.

Since the electron density in a crystal varies continuously and periodically in three-dimensional space, the electron density ρ(xyz) at a point with fractional coordinates x, y, z in a unit cell of volume V may be expressed as a three-dimensional Fourier series:

$$\rho(\mathrm{xyz}) = V\sum_{h}\sum_{k}\sum_{l}|F(hkl)|\cos[2\pi(hx+ky+lz)\cdot\alpha(hkl)]$$

If both the amplitude **|F(hkl)|** and the phase **α(hkl)** of each reflection are known, the electron density within the unit cell of the crystal can be calculated directly. On the other hand, if the positions of the atoms in the unit cell are known, both the structure factor and the phase for each reflection may be calculated from the structure factor equation:

$$F(hkl) = \sum_{i} f_j \exp[2\pi(hx_i + ky_i + lz_i)]$$

where **fj** is the atomic scattering factor for the atom j and xj, yj, zj are its fractional coordinates. In an actual structure determination, both forms of the Fourier transform equations are utilized to arrive at a model structure from which the observed diffraction pattern can be reproduced.

2.4.2 Instrumentation

The basic hardware components of typical automated single-crystal X-ray diffractometer system include:

- An X-ray source, consisting of:
  - a high-stability X-ray generator;
  - a copper, molybdenum, or silver target X-ray tube;
  - a tube shield with associated shutters, attenuators and safety interlocks;
  - a monochromator or X-ray mirror system; and
  - an incident-beam collimator;
- A three- or four-circle goniometer system that allows the specimen to be precisely oriented in any position while remaining in the X-ray beam;
- A video camera for aligning the specimen and indexing crystal faces;
- A two-dimensional X-ray detector system (typically CMOS-based);
- A low-temperature attachment for cooling the specimen during data collection;
- A microprocessor-based interface module that receives commands from the host computer and carries out all real-time instrument control functions to drive goniometer motors, monitor the detector system, open and close the shutter, and monitor collision sensors and safety interlocks; and
- A host computer with a large hard disk mass storage device, a video monitor and keyboard, and diffractometer control programs to control the data collection strategy and to send commands to the microprocessor.

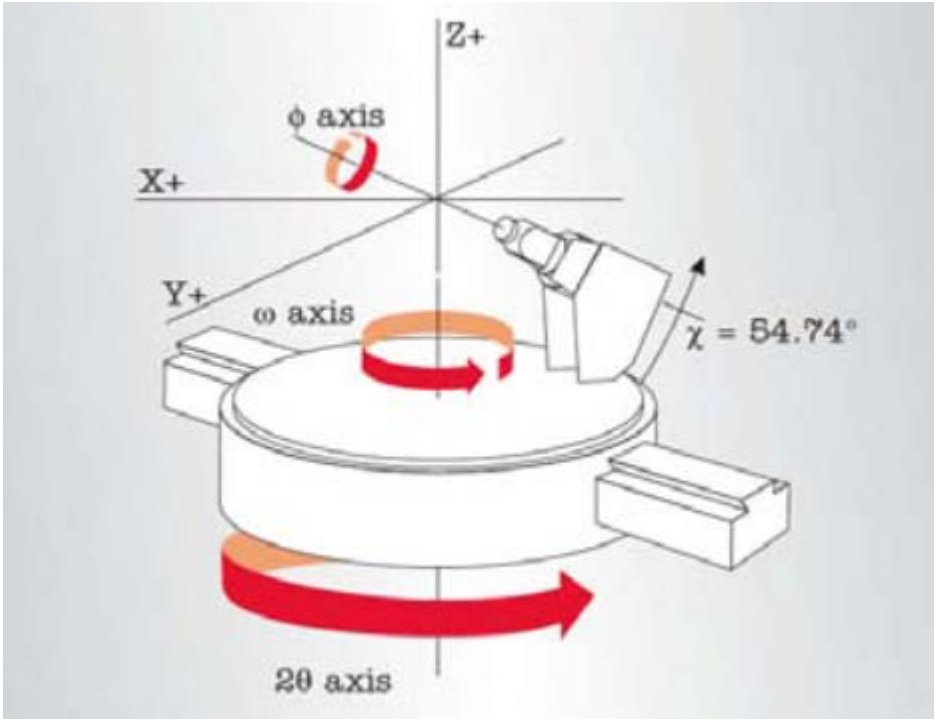
Structure determination may be carried out on the computer used for data collection, or on a second computer connected to the diffractometer system. A large variety of hardware configurations is available, depending upon the requirements of the individual laboratory.

Figure 2.22 —D8 VENTURE CMOS-based single-crystal X-ray diffractometer system

2.4.2.1 Goniometer

The most critical mechanical component in an X-ray diffractometer system is the goniometer assembly, which must be capable of keeping the specimen centered in the incident X-ray beam while at the same time changing its orientation in order to collect many thousands of frames of data in reciprocal space. The most commonly used type of goniometer is illustrated in Figure 2.23.

Figure 2.23 —Diagram of axes for a three-circle goniometer (chi is fixed at approximately 54.7°)



2.4.2.2 Detector

The most important and most expensive component of any modern single-crystal diffractometer system is its detector system.

CMOS

CMOS detector technologies have overtaken CCDs in high-end applications. The PHOTON II CMOS detector employs a large sensor of 100 × 140 mm, without the need for a fiber-optic taper to increase the detection size. This improves the X-ray efficiency and increases the total number of X-ray reflections collected per image.

Figure 2.24 —PHOTON II CMOS detector

2.4.3 Experimental Procedure

The experimental procedure typically consists of the following steps:

1. The first step in a crystal structure analysis is concerned with the selection and mounting of a suitable specimen. Ideally, a crystal whose structure is to be determined must be a single crystal of 0.1 mm to 0.5 mm size, not cracked and not *twinned*. Crystals in the ten-micron range are not uncommon with higher-intensity microfocus sources. The techniques required to obtain such crystals vary considerably depending upon the types of compounds to be analyzed. Stable crystals of typical organic, organometallic, or coordination complexes can usually be grown by slow recrystallization from common solvents. Other types of compounds may require the use of sublimation, zone refinement, solvent-diffusion, low-temperature and/or inert-atmosphere techniques in order to isolate suitable specimens.

   Once a suitable specimen has been selected, it is glued or otherwise securely attached to a goniometer head (i.e., the sample holder) in an arbitrary orientation.

2. The goniometer head is placed on the base of the goniometer assembly, and the goniometer head's orthogonal X, Y, and Z translations are adjusted until the specimen is optically aligned to the center of the incident X-ray beam (as viewed by a video camera or microscope).

3. A preliminary rotational image is collected for up to one minute to screen the specimen for analysis and to select suitable parameter values for subsequent steps.

4. The unit cell is determined:

   4.1. A preliminary set of frames is measured using an automatic routine. For example, three sets of frames are collected in different parts of reciprocal space.

   4.2. These frames are processed to locate spots on individual frames and to then determine the centers of reflections.

   4.3. An auto-indexing routine selects the appropriate reduced primitive unit cell and calculates the corresponding orientation matrix and lattice constants.

   4.4. This preliminary unit cell is then refined using a nonlinear least squares algorithm and converted automatically to the appropriate crystal system and Bravais lattice. This new cell is refined by the non-linear least-squares algorithm to yield an accurate orientation matrix which may be used to index crystal faces and to carry out integration calculations after intensity data collection.

5. After the geometric data collection steps have been completed and an accurate orientation matrix has been calculated, intensity data collection is carried out.

   Typically, a set of scans is calculated to acquire a dataset covering the unique data and sufficient redundancy for accurate scaling. Data is usually collected using a narrow-frame scan method in which several sets of frames (i.e., *runs*) are collected by scanning in 0.3° to 0.5° increments in the ω and/or φ angle, while keeping all other instrument angles constant. There are options to limit data to a unique set of reflections, thereby reducing data collection times for high-symmetry crystal systems. A complete data set may require anywhere from a few hours to overnight depending upon the size of the specimen and its diffracting power. Typical exposure times of 10 to 30 seconds per frame for a hemisphere of data require from 6 to 13 hours of total data collection time.

6. When the complete set of frames has been collected for a given specimen, the entire data set must be processed to obtain accurate integrated intensities for individual reflections. This process includes corrections for instrumental factors, polarization effects, X-ray absorption and possibly crystal decomposition. The integration process reduces the raw frame data—which requires from 500 to 2000 megabytes of disk storage—to a small set of individual integrated intensities for individual reflections. The final unit cell constants are calculated from the centroids of up to 10000 reflections selected from the entire data set and typically have relative errors of less than 1/10,000.

7. Once the structure amplitudes are known, the phase problem must be solved to find a self-consistent set of phases that can be combined with the structure factor amplitudes to obtain the electron density and thereby determine the structure of the crystal.

   A number of crystallographic techniques exist for obtaining the phases of diffracted waves; the most widely-used approaches to the phase problem involve the use of either vector methods based on $\mathbf{|F(hkl)|^2}$ or direct or statistical methods. Typically, the solution to the structure yields only a partial or approximate model, which must be improved by successive applications of Fourier-transform methods before the complete structure has been determined.

8. After the entire molecular structure has been determined, the approximate positions of the atoms are refined by nonlinear least-squares techniques to give the best fit between the calculated and observed intensity data for the specimen. The refinement process yields very accurate values for atomic positions from which bond lengths, bond angles, and other structural parameters may be calculated.

9. Finally, upon completion of the X-ray diffraction analysis, the structure of the molecule or crystal lattice must be displayed or plotted (Figure 2.25 and Figure 2.26).

Figure 2.25 —Thermal ellipsoid plot of the final structure of an organic compound ($C_{18}H_{18}O_9$)

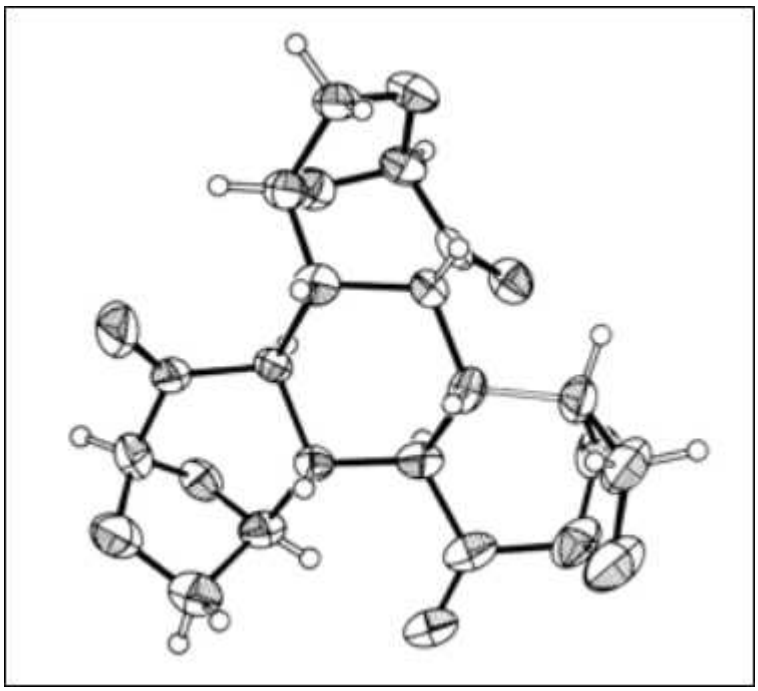
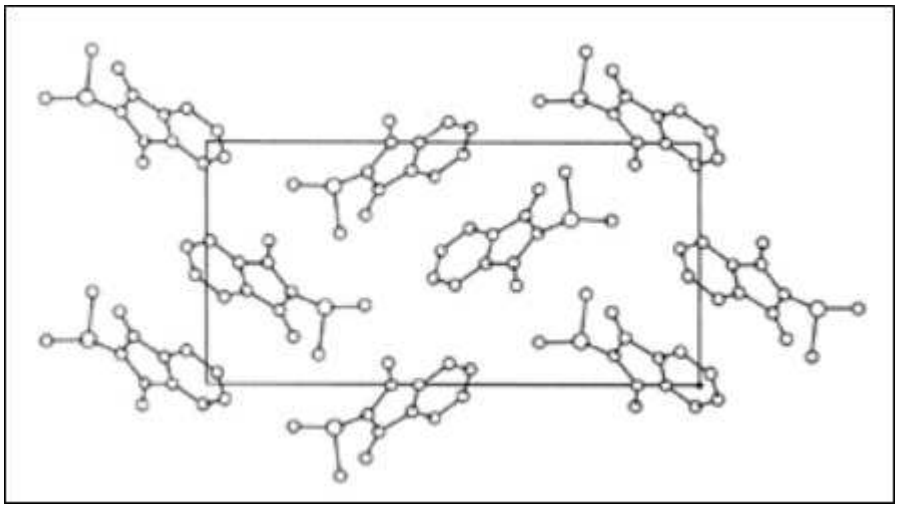


Figure 2.26 —Unit-cell diagram showing the arrangement of molecules within the cell

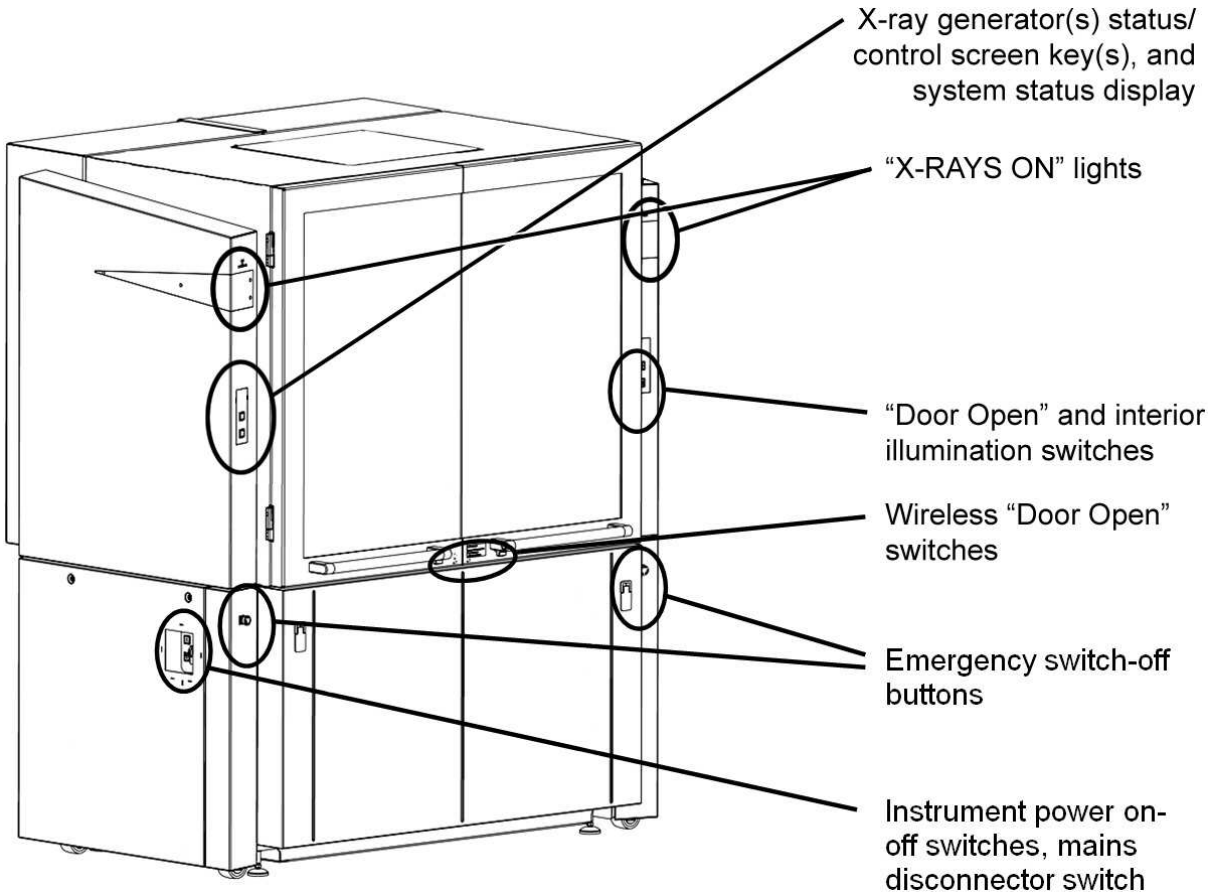


3 Locations and Functions of Basic Components

3.1 Enclosure Exterior

The diffractometer's controls are located on the front and left-hand sides of the instrument.

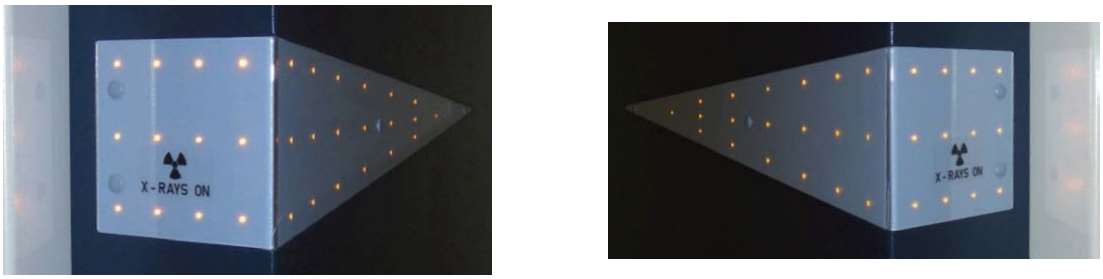
Figure 3.1 — Diffractometer external housing



3.1.1 Enclosure “X-RAYS ON” Lights

The illuminated orange “X-RAYS ON” warning lights on the enclosure’s right- and left-hand sides indicate that the high-voltage generator is turned on and the X-ray source is operating.

Figure 3.2 — Enclosure “X-RAYS ON” lights



The warning lights’ functionality is continually monitored by the X-ray safety control unit.

3.1.2 System Power Control

The system-wide power control functions are located on the lower part of the instrument: two emergency switch-off buttons on the left- and right-hand sides, and a power control panel on the left-hand side.

Figure 3.3 — System power control (left side)

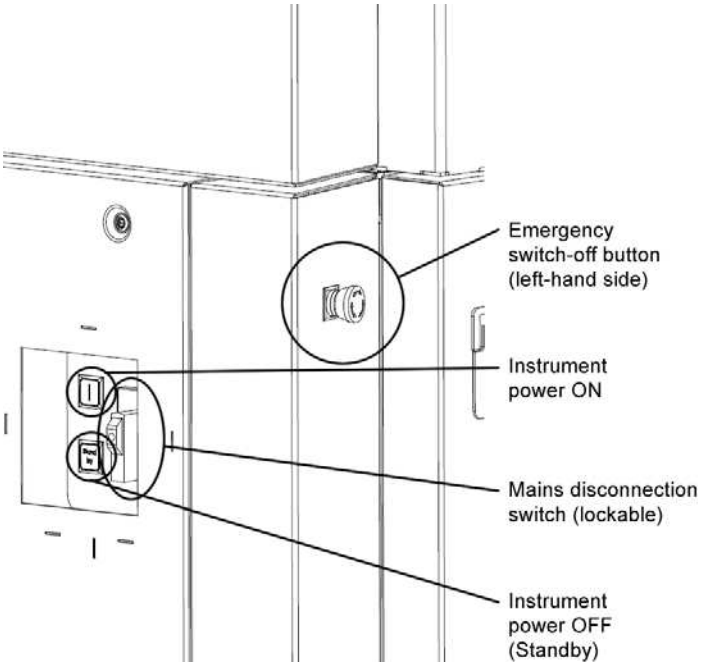


Table 3.1 — System power control switches and functions

Switch	Function
Emergency switch-off button	If pressed, switches off the control electronics, the X-ray generator, and all components connected to AC outlets X102 (located on the mains distribution unit). The X-ray source is turned off, and all moving drives stop instantly. Components connected to AC outlet X101 are not switched off. The emergency switch-off button should be used in emergency situations only, and not for normal system shutdown!
Instrument power ON	Green switch with an “|”. Powers up the instrument. This button switches on the control electronics, the X-ray generator, and all components connected to the AC outlets X102 (located on the mains distribution unit). After the boot procedure, the instrument is ready for operation. As soon as the system is ready, the generator symbol changes to a white “|” if no error is pending (otherwise, the error will be displayed) and the system display will change to blank white if no error is pending (otherwise, the error will be displayed). The X-ray generator can then be activated by pressing the X-ray generator key on the left-hand side.
Mains disconnection switch	This switch completely disconnects all power and disables all electrical components that are connected to the instrument’s main power distribution. This switch can be locked with a safety lock to prevent unauthorized switch-on of the instrument. Set this switch to “0” before unplugging the main power connector.
Instrument power OFF	Red switch with a “Standby” marking. Powers down the instrument and X-ray generator, and stops all drives immediately. Special components such as vacuum pumps (if installed) might remain powered on if they are connected to X101. Components connected to X102 are switched off.

NOTE: Do not press the power OFF button as long as the X-ray generator is generating X-rays (i.e., as long as the “X-RAYS ON” lights are illuminated). This could significantly reduce the lifetime of the X-ray source. Before switching off the system, it is recommended to first reduce the high voltage and current to standby values using the control software (e.g., BIS Server). Then, turn off the high voltage by switching off the X-ray generator. The “X-RAYS ON” lights will turn off. After that, the instrument can be switched off by pressing the Instrument power OFF button.

3.1.3 Enclosure Doors, Locks, and Opening Mechanism

3.1.3.1 “Door Open” Button on Side Panel

The door is unlocked by pressing the “Door Open” button, either on the side panel or on one of the door handles.

The front door is locked by means of a locking bolt. To unlock the door, you must press a “Door Open” button. Then, you can open the door outwards. If you press the “Door Open” button when the X-ray tube window is open, the X-ray shutter will close automatically. The X-ray shutter can be re-opened after the front door has been closed. The door can also be unlocked using DIFFRAC.MAINTENANCE’s “TOOLS” plug-in.

3.1.3.2 Wireless “Door Open” Buttons

Inside the door handles, toward the center of the instrument, there are two wireless “Door Open” buttons. Pushing these buttons will unlock the doors.

3.1.3.3 Slide/Swing Mechanism

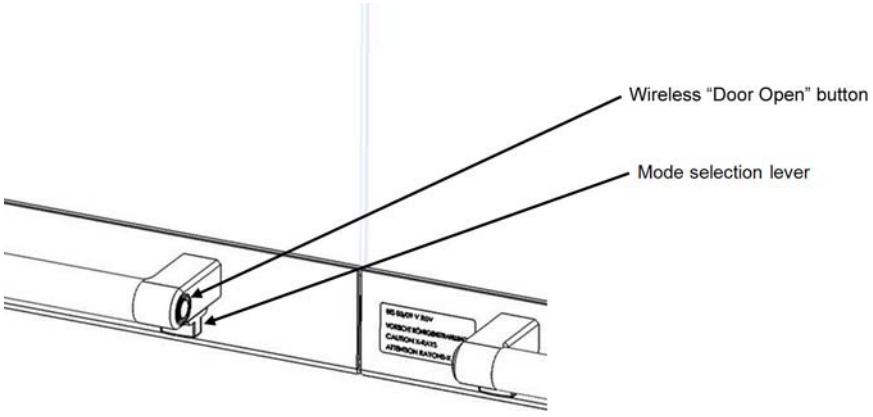
In standard operation mode, both doors can be slid open. If necessary (e.g., to bring large equipment into the enclosure), operation of the doors can be switched to *swing mode*.

In order to switch between the modes, use the lever under the door handle. With locked doors, push the lever to enable swing mode and pull the lever to enable slide mode.

When in swing mode, unlock the door by using one of the “Door Open” buttons. Slide the doors open until a mechanical stop is reached, and then swing it open.

When in slide mode, unlock the door by using one of the “Door Open” buttons. Slide the doors open until a mechanical stop is reached.

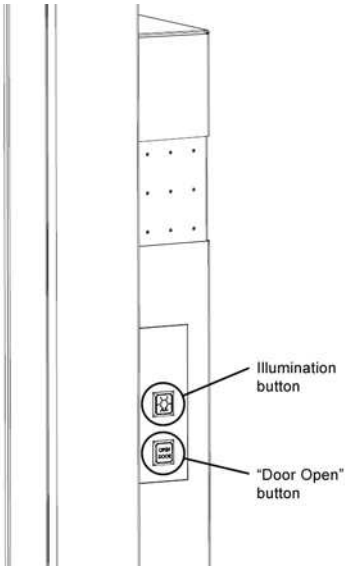
Figure 3.4 — Door mode selection lever



3.1.4 Enclosure Control Buttons

On the enclosure's right-hand side, two buttons directly control the illumination and the door lock mechanism.

Figure 3.5 — Enclosure control buttons



3.1.4.1 Illumination Button

This button switches the enclosure's internal illumination on and off (this also works if the instrument is powered down to standby mode). During instrument startup, the illumination is switched off. After startup, the illumination's brightness is determined by the door's open-or-closed status. By default, the brightness is reduced when the door is closed and increased if the door is open (this may, however, be adjusted in DIFFRAC.MAINTENANCE's "CONFIGURATION" plug-in). If the instrument is powered down, only on and off is possible using this switch; no brightness control is possible.

3.1.4.2 "Door Open" Button

Under normal operational conditions, the door handles are locked by a mechanical shutter. To open the front door, an "Door Open" button must be pressed. If the "Door Open" button is activated while the tube window is open, the X-ray shutter will close automatically. After closing the front doors, the X-ray shutter can be opened again. Alternatively, the Instrument Control Software's "Enclosure" function allows you to unlock the door via software.

3.1.4.3 System Status and Control Buttons

The instrument's status and control buttons are located on the instrument's left-hand front side.

If the system has two X-ray sources, the left-hand side has labeled screen keys for each of the sources.

CAUTION

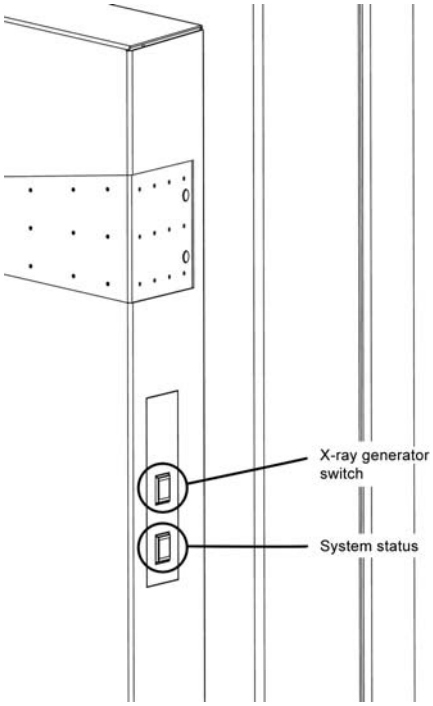
If you shut off the power using the "Power OFF" button, the AC outlets X101 located on the mains distribution panel will still have power! X101 can only be switched off by the mains disconnection switch, or by the user-installed external power switch near the instrument.

3.1.4.4 Screen Keys: Status Display and High-Voltage Operation

The system has two screen keys on the enclosure's left-hand side (systems with two X-ray sources have three screen keys: one for each of the generators, and one for the system status).

The upper screen key(s) is (are) used for high-voltage control. The lower screen key is used for system status information, and it has no function when pressed.

Figure 3.6 — Screen keys: status display and high-voltage operation (single X-ray source shown)



Upper Screen Key (X-ray Generator Status and Control)

Table 3.2 — Upper screen key states and descriptions

State	Description	Meaning
	White blank display	Shown during the instrument's startup sequence.
	Switch-on symbol	Shown when the X-ray generator is switched off and is ready to be switched on. Press once to start the X-ray generator.
	Confirm filament type	For TURBO X-RAY SOURCE, prompts you to confirm the filament type in DIFFRAC.MAINTENANCE.
	Vacuum pump-down	For TURBO X-RAY SOURCE, indicates that the vacuum is pumping down.
	Vacuum OK	For TURBO X-RAY SOURCE, indicates that the vacuum is at an acceptable level for X-ray generation.
	Anode accelerating	For TURBO X-RAY SOURCE, indicates that the anode is spinning up to its required speed for X-ray generation.
	Waiting for jet stability	The ICS is waiting until the METALJET's jet stability is confirmed; only low power (i.e., 20 W) is available.
	Heating on	Blinks yellow when the generator's tube heating is enabled and no X-rays are being generated. Constant yellow when the switch-off circuit has been closed but X-rays are not being generated. Press once to switch off the generator.
	X-rays on, generator is busy	Shown when the X-rays are switched on and the generator is ramping its power (yellow background). Press once to switch off the generator.
	X-rays on, generator ready	Shown when the generator has reached its set values (black background). Press once to switch off the generator.
	X-ray safety circuit error	The X-ray generator will be switched off if a safety error occurs. For details, examine the safety board in DIFFRAC.MAINTENANCE's "TOOLS" plug-in. If the error is fixed, the symbol will disappear and the switch-on symbol will appear (if no subsequent error is pending).
	White blinking	Safety warning is present. See DIFFRAC.MAINTENANCE's "TOOLS" plug-in for detailed information. This is not an error, but X-ray generation is not possible if this symbol is visible.
	Generator alarm	Either the generator or one of its connected components (tube, safety circuit, and water cooling system) has an error. See DIFFRAC.MAINTENANCE's "TOOLS" plug-in for detailed information. If the error is fixed, this symbol will disappear and the "switch-on" symbol will appear (if no subsequent error is pending).

State	Description	Meaning
	Generator error	Generator error. See DIFFRAC.MAINTENANCE's "TOOLS" plug-in for detailed information. Either the generator is in an undefined state, or the tube was not detected successfully.
	Cooling system alarm	The system has switched off the generator due to an error in the cooling system. For details, examine the cooling unit in DIFFRAC.MAINTENANCE's "TOOLS" plug-in. If the error is fixed, this symbol will disappear and the "switch-on" symbol will appear (if no subsequent error is pending).
	Conditioning enabled	When this symbol blinks blue, X-ray source conditioning is enabled. To abort conditioning, press the button once; the generator will switch off. You can then proceed with operation in normal mode. It is not recommended to abort tube conditioning, because this process helps to establish a longer X-ray source lifetime.
	Jet starting	The METALJET's jet is starting.
	Vacuum progress	If blinking, the METALJET's vacuum is pumping down. If steady, the vacuum is stable.
	Generator stopping	The generator is stopping.

Lower Screen Key (System Status)

Table 3.3 — Lower screen key states and descriptions

State	Description	Meaning
	White blinking blank display	The instrument is booting.
	White blank display	The instrument has booted and is ready for operation.
	Green blank display	The instrument is controlled by a client (i.e., Measurement Server)
	Measurement running	The symbol flashes blue if a measurement is in progress.
	Door open	The door is open. A measurement is only possible with closed doors.
	Sample changer error	The built-in sample changer has an error and needs a user interaction before proceeding (see the TOOLS module for a detailed description of the pending error).
	Detector error	The detector has an error.
	Drive collision	At least two drives are collided. For information on resolving the collision, see DIFFRAC.MAINTENANCE's "TOOLS" plug-in.
	Drive error	At least one drive has an error. For information on resolving the error, see DIFFRAC.MAINTENANCE's "TOOLS" plug-in.

NOTE: Pressing the lower screen key five times will display the system's IP address.

NOTE: Typically, pending system alarms or warnings (i.e., red display elements) will also appear as messages on the computer screen. DIFFRAC.TOOLS can be used to get a complete diagnosis of any pending alarms or warnings. DIFFRAC.TOOLS is also useful in resolving the alarms or warnings.

3.2 The Experiment Area

3.2.1 Automatic Component Recognition

Through the use of identifiers located on the components themselves, it is possible for the DIFFRAC.SUITE software to recognize the presence and position of the most commonly-used components. The word “component” refers to the tubes, optics, and detectors that are equipped with an identifier. The identifier is an embedded “OpCo” chip.

Automatic component recognition is possible within the framework defined in the CONFIGURATION plug-in, as described in the DIFFRAC.SUITE User Manual. In CONFIGURATION, the possible component types, positions and properties are specified. After CONFIGURATION is properly set up, the DAVINCI plug-in then provides an overview of the components currently mounted on the arms and at the center of the goniometer of the connected instrument, as well as their properties. Refer to the DIFFRAC.SUITE User Manual for detailed information regarding CONFIGURATION and DAVINCI.

3.2.2 X-ray Sources: Configurations and Combinations

The system is available in either single- or dual-source configurations:

- A single-source configuration may feature either a point-focus Siemens sealed tube (powered by a Kristalloflex 430 generator), or an Incoatec IμS microfocus source.
- A dual-source configuration will feature either:
  - A Siemens point-focus sealed-tube source (powered by a Kristalloflex 430 generator), paired with an Incoatec IμS microfocus source; or
  - Two Incoatec IμS sources.

Single-crystal X-ray diffraction applications typically use Cu radiation or Mo radiation. Other radiation types are sometimes used.

3.2.2.1 The Siemens Sealed X-ray Tube

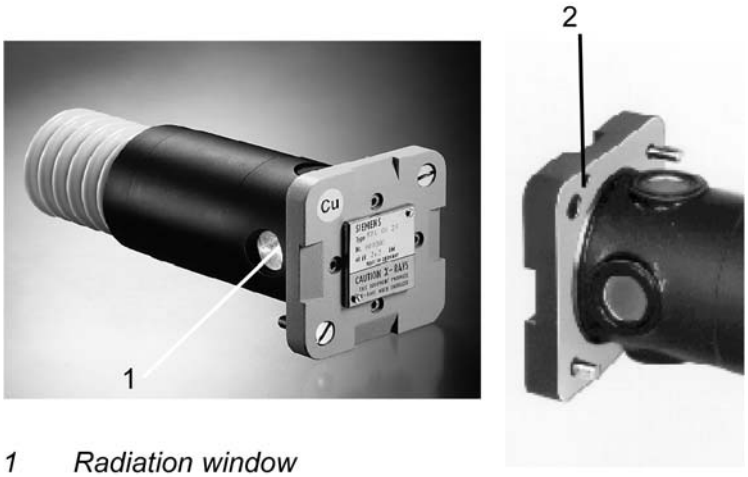
Sealed X-ray tubes have two beryllium windows from which the X-rays can escape in a single direction. One of the beryllium windows lies parallel to the long side of the luminous spot on the anode, which can be regarded in a good approximation as rectangular. This window is used for *line focus* operation. The other beryllium window lies parallel to the short side of the luminous spot and is used for *spot focus* operation. The exact size of the focus in the particular operating mode is dependent on the type of tube used and is specified in the X-ray tube's technical documents.

Within the framework of the type approval for the diffractometer, all anode materials whose atomic numbers are less than 42 (i.e.,molybdenum and below) can be used. Anode materials with a higher atomic number can also be used in the diffractometer; however, these require approval over and above that accorded by the type approval.

For line focus and spot focus operation, there are different cooling plates. These differ in that the cooling circuits are set at an angle of 90° to each other.

A KF...4KE vacuum-tight X-ray diffraction tube is available with Cr, Fe, Co, Cu, Mo, Ag or W anode. The optical focus can be modified by changing the emission angle. A normal emission angle of 5° reduces the projection of the focus to 1/10 of its length.

Figure 3.7 — KF...4KE X-ray diffraction tube (ceramic tube type)



1 Radiation window
2 Bore hole for positioning the tube in the tube stand

3.2.2.2 The Incoatec IμS Source

The IμS is a microfocus-source-based beam delivery system that is equipped with Montel-type multilayer optics. Usually, the delivery includes a high-voltage generator, the X-ray source, the optics, and a housing for the alignment of the optics. Depending on the model, it may include a mechanical stand, a vacuum pump and vacuum gauge, an evacuable flight tube, additional equipment for the alignment, etc.

The IμS source consists of two main components:

- **Generator** (model IG-250 for D8 Series II instruments, model IG-380 for D8 QUEST and D8 VENTURE), consisting of a high-voltage power supply, a safety board, and a vacuum monitor; and
- The IμS X-ray tube assembly consisting of:
  - The tube in its tube housing;
  - The optics housing; and
  - The optics mounting bracket.

Figure 3.8 — Basic IμS system components (IμS 3.0 with IG-380 generator pictured)

1	Generator
2	Tube
3	Tube housing
4	Optics housing and bracket (behind shroud)

Depending on your system, it may also include:

- A mechanical stand or alignment stage (DOF);
- A vacuum pump;
- An evacuable flight tube; and/or
- Accessories such as additional equipment for alignment.

3.2.3 Goniometer: FIXED-CHI or KAPPA

The system has two goniometer options:

- FIXED-CHI (with a fixed chi stage); or
- KAPPA (with a kappa axis).

3.2.3.1 The FIXED-CHI Goniometer

Figure 3.9 — FIXED-CHI goniometer (with DX drive)

The FIXED-CHI 3-circle goniometer incorporates a fixed-chi stage with a chi angle of approximately 54.7° and a phi drive with 360° rotation, which is so compact that it swings under the incident beam collimator, allowing free rotation in omega. This design allows you to collect complete data sets for any crystal symmetry by using a combination of phi and omega scans. The open geometry leaves the sample easily accessible for low-temperature devices and high-pressure cells.

The horizontally-oriented D8 goniometer base has 2theta, omega, phi, and optional DX drives, tracks for the X-ray source and detector, and an additional mounting ring for accessories such as optional low-temperature attachments.

The D8 design mounts the incident beam components—source(s), X-ray optics, and beam collimators—on a precision track. This ensures reproducible positioning, making exchange or addition of new optics very easy.

Stepper motors with optical encoders ensure extremely-precise angular values for the most demanding applications, while maintaining superb alignment for many years. Fast goniometer positioning enhances productivity.

3.2.3.2 The KAPPA Goniometer

Figure 3.10 —KAPPA goniometer

The KAPPA goniometer is Bruker's latest 4-circle goniometer which combines the gold-standard high-precision D8 base with the most versatile kappa stage available.

With a sphere of confusion of only 7 microns, the mechanics of the KAPPA goniometer are much more precise than those found in any other 3- or 4-circle goniometer design. This ensures that, as they rotate, even the tiniest microcrystals will remain at the instrument center and be fully bathed in small X-ray beams. The compact and short primary beam path ensures the highest flux from any X-ray source. The kappa geometry allows for extremely high-resolution and high-redundancy work (e.g., charge density studies, SAD data, or data collection with copper radiation to determine the absolute configuration of light-atom compounds).

The D8 base makes it easy to add new X-ray optics and mount accessories (e.g., low-temperature devices) directly on the base ring.

Software-controlled goniometer axes omega, phi, and kappa offer full sample positioning freedom and provide easy access to the sample for low-temperature and other conditioning devices. The sample can be positioned to take photographs about any axis for immediate verification of crystallographic symmetry. Automatic homing and extremely high slew and scan speeds distinguish this precision kappa goniometer from all others on the market.

The KAPPA goniometer also incorporates a software-controlled detector track, for optimum sample-to-detector distances at a mouse click. This not only allows faster and more efficient data collection at short distances, but also provides automatic withdrawal to prevent overlap with twinned crystals or supercell problems.

The goniometer’s short beam path guarantees the highest intensity.

For more information about the KAPPA goniometer, refer to DOC-M86-EXX105 KAPPA Goniometer User Manual.

3.2.4 The PHOTON II CMOS Detector

The PHOTON detector is Bruker's newest solid-state X-ray area detector. It uses CMOS (Complementary Metal Oxide Semiconductor) technology for unprecedented speed and dynamic range. The PHOTON communicates over USB and performs all corrections and data processing using its on-board electronics.

The detector has a very large (140 cm^2) active area. It can collect a data frame of 768 × 1024 pixels with a pixel size of 135 µm on a side.

The CMOS sensor's continuous-readout capability makes the detector able to operate in *shutterless mode*, which removes nearly all additional time introduced by opening and closing an experimental shutter and repositioning the goniometer between exposures. Shutterless mode also greatly reduces the systematic error introduced by goniometer repositioning between exposures.

3.2.5 Labyrinths

On the floor of the enclosure, to the left and right of the goniometer, are located two labyrinths whose purpose is to provide a safe and convenient passage for the various cables and hoses entering and leaving the enclosure. These include water hoses (if applicable) and electrical lines supplying current to the motors and the X-ray tube. The labyrinths are designed to prevent radiation from escaping to the outside of the instrument during instrument operation. If you use any additional equipment in the enclosure that is not included in the delivery, **use the labyrinth on the right-hand side** of the instrument to route the lines to the equipment.

- In the D8 VENTURE enclosure, both labyrinths (left and right) can be slid into and out of position.

3.3 Underneath the Experiment Area

3.3.1 Inside the Enclosure

Table 3.4 — Location of components inside the enclosure

Location	Components
Front	Power distribution board, X-ray generator(s), safety board (s)Additionally, if applicable: K430 generator internal water cooler
Rear	Ventilators, HV, LAN cable routingAdditionally, if applicable: K430 generator F1 circuit breaker, water inlet and outlet lines
Left	Mains distribution board with mains filter, terminals, circuit breakers, and fuses
Right	Enclosure controller, universal I/O board, indexer boards, dual-source controller board
Inside the enclosure	Goniometer, X-ray source(s), detector, labyrinths

3.3.1.1 Lower Front Side

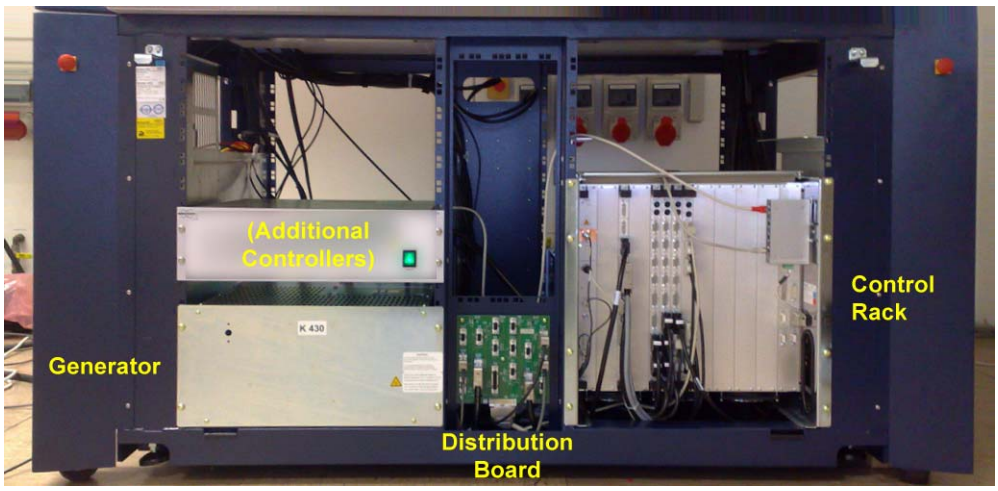
The following components are located on the instrument's lower front side:

- Distribution board
- Generator
- Control rack

The distribution board is located in the center of the lower front side.

The right-hand side contains the control rack and, optionally, additional slide-in units.

Figure 3.11 —Lower front side, underneath the experiment area



The control rack accommodates the detector board, the universal I/O board, and the indexer boards. Each indexer board can be either two-axis or four-axis, meaning control of either two or four motors, respectively. Attached to the control rack at the top right is the Ethernet switch, which is connected by four lines to the control PC, the detector controller, the control rack, and the network (LAN).

Figure 3.12 —Control rack

In the frame accommodating the control rack are four extra sets of slots which can be used to accommodate additional components.

3.3.1.2 Lower Rear Side

The following components are located on the instrument's lower rear side:

- Optional water chiller for K430 generator;
- Safety board;
- Water inlet and outlet lines
- Ventilators

The F1 generator switch is located at the rear of the K430 generator. This switch is actuated in case of a malfunction, and interrupts the flow of high-voltage current to the generator. It can be reset manually.

Openings are provided in the rear panel for two water lines (inlet and outlet) and a power supply line for the generator. Two additional holes on the upper left and upper right side of the rear panel are provided for additional routings (e.g., for vacuum hoses).

Two ventilators are housed in the rear panel, and are connected to the instrument by a power line.

3.3.1.3 Lower Right-Hand Side

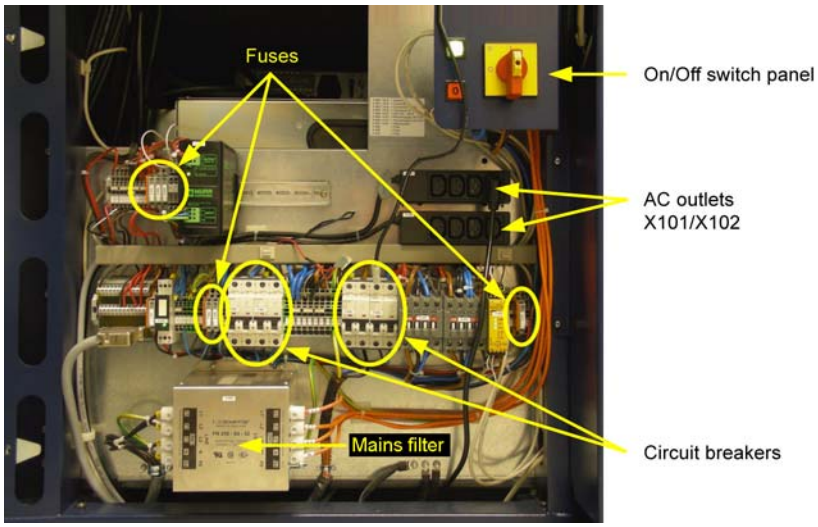
The K430 generator's optional water chiller can be accessed from the right-hand side of the instrument. This might be necessary when filling the chiller's water tank.

3.3.1.4 Lower Left-Hand Side

The following components are located on the instrument's lower left-hand side:

- Mains distribution board with the mains filter
- Terminals
- Circuit breakers
- Fuses

Figure 3.13 —Lower left-hand side of instrument



The mains filter is located at the bottom left. Running across the center of the distribution board is a row of terminals, circuit breakers, and fuses. Some fuses are also located above this row at the top left. The circuit breakers are automatically triggered in the event of an overload or short circuit, and they can be manually reset to resume normal operation. The fuses are accommodated in fuse holders that are identified by a number prefixed by "F," (e.g., "F604", "F611" and "F606").

To change a fuse, flip the fuse holder upwards, open its cover, and replace the fuse with the spare fuse stored to the left of it in the holder. Then, close the cover and flip the fuse holder back into place.

The fuses can be located with the help of the diagram attached to the mains panel.

There are two AC outlet strips (with three sockets each) inside the mains distribution unit (behind the enclosure's left-hand side panel, to the right and above the row of terminals, circuit breakers, and fuses). These outlets (labeled X101 and X102) are mainly used internally for powering the control electronics and cabinet fans.

Depending on the electrical state of the instrument, these AC outlet strips can be supplied or not supplied with power:

- The mains disconnection switch can turn off power for both X101 and X102.
- The Power Off (Standby) and Emergency Switch Off buttons can turn off power for X102, but **X101 remains powered if the Power Off (Standby) or Emergency Switch Off buttons are pressed.**

4 Common Tasks

4.1 Reset the X-ray Safety System

Before resetting the safety system, make sure that no screen keys are flashing red (i.e., press the screen key a second time to change it to yellow).

1. In DIFFRAC.MAINTENANCE's "TOOLS" plug-in, navigate to the safety board(s). Choose **Utilities > Reset Safetyboard**.

   The "Reset Safetyboard" dialog appears, prompting you for a password.

2. Enter the password (set to `password` by default), and click **Send**.

The safety system is reset.

4.2 Removing the Panels

4.2.1 Remove the Front Panel

1. Pull up the two black latches at the top of the front panel, and turn them to the right or left.

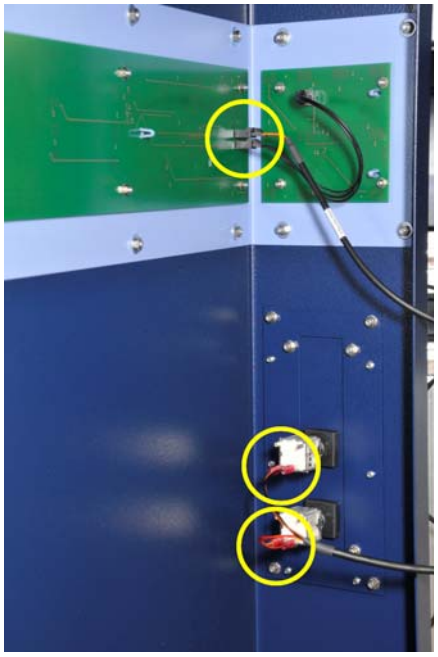
   The panel can then be swung open.

2. If necessary, detach the panel completely by lifting it out of the suspension holes at the foot of the enclosure housing.

4.2.2 Remove the Upper Right-Hand Side Panel

1. Raise the panel up, so that it comes free from its hooks (this can be done by one person).
2. While the panel is raised, disconnect the cables from their dedicated positions.

Figure 4.1 — Right-hand side panel cable connections



3. Lift the panel away from the enclosure.
4. Set the panel aside.

4.2.3 Remove the Lower Right-Hand Side Panel

1. Unscrew the right-hand side panel's cam lock screws (which are fitted with a spring) at the top of the panel.

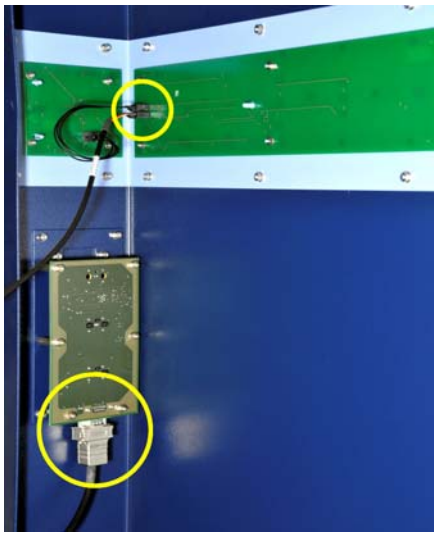
   The panel can then be swung open.

2. If necessary, detach the panel completely by lifting it out of the suspension holes at the foot of the enclosure housing.

4.2.4 Remove the Upper Left-Hand Side Panel

1. Raise the upper left-hand side panel, so that it comes free from its hooks (this can be done by one person).
2. While the panel is raised, remove the cables from their dedicated connections.

Figure 4.2 — Left-hand side panel cable connections



3. List the panel away from the enclosure.
4. Set the panel aside.

4.2.5 Remove the Lower Left-Hand Side Panel

1. Unscrew the left-hand side panel's cam lock screws (which are fitted with a spring) at the top of the panel.

   The panel can then be swung open.
2. If necessary, detach the panel completely by lifting it out of the suspension holes at the foot of the enclosure housing.

4.2.6 Remove the Lower Rear Panel

NOTE: The lower rear panel can only be removed after the left-hand side panel has been removed.

1. Unscrew the rear panel's screws.

   The panel can then be swung open.
2. If necessary, detach the panel completely by lifting it out of the suspension holes at the foot of the enclosure housing.

4.3 Cleaning the System

When cleaning the enclosure interior, do not use any liquids or aggressive cleaning agents. The glass doors, however, can be cleaned with any standard household-type window cleaner.

CAUTION

Before cleaning, completely turn off the system power including the high-voltage generator, control electronics, and all accessory components! Failure to do so may result in injury or component damage.

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