

Instrument Introduction

Instrument Components

All single-crystal, X-ray diffraction instruments include 4 major components, the radiation source, the radiation optics, the goniometer, and the detector. The *radiation source* emits X-ray radiation used in the experiment. The *optics*, that are usually located between the radiation source and the sample, shape and enhance the radiation impinging on the sample and permit only a narrow range of energies of radiation to hit the sample. The *goniometer* is simply a device for moving the sample and the detector into a variety of positions. The reciprocal lattice of the sample follows the movements of the crystal. Diffraction events occur whenever points on the reciprocal lattice of the crystal are moved into the Ewald sphere. The diffraction events are measured with the *detector*.

The instrument in the Chemical Crystallography Laboratory has a sealed-tube radiation source, optics made up of two curved mirrors, an Kappa-geometry goniometer, and a Photon II CMOS area detector. The instrument also includes an Oxford Cryosystems Cryostream 800 low-temperature device to cool the sample.

X Rays are generated whenever a beam with enough energy strikes an object. In laboratory settings, the initial beam is made when energetic electrons hit a block of metal (the anode). Two types of X rays are emitted. Radiation with a continuous band of energies (having a definite maximum energy) called Bremsstrahlung (or braking radiation) is emitted when the electrons pass near to the nucleus of the metal atoms. X Rays with a discrete set of energies are also emitted when the original electron beam knocks an electron out of a low energy orbital of the metal and an electron from a higher energy shell falls in energy to fill up the vacated hole giving off a quantum of radiation (an X-ray photon). The energy of this discrete quantum is the difference in energy between the two orbitals in the metal and is *characteristic* of the type of metal.

The optics of a single-crystal instrument serves the two functions of *collimating* the incident beam and *monochromating* the beam. The incident beam is made nearly monochromatic by bouncing the X-ray beam off of two mirrors. Associated with the optics are two shutters to block the incident radiation.

The D8 goniometer moves the sample using a combination of ω (vertical rotation axis, parallel with the rotation axis of the detector) and ϕ (spindle axis of the goniometer head) motions. The angle of the detector arm (sometimes called the 2θ or the swing arm) to the incident beam can also be adjusted by the goniometer. The crystal is centered in the X-ray beam and the movements of all circles of the instrument by making fine adjustments to the goniometer head while viewing the sample with a video camera. A low temperature device blows a stream of cold (typically 100 K) nitrogen gas onto the sample.



Instrument Safety

The X rays produced in this lab are penetrating, ionizing radiation that can cause tissue damage. The most dangerous part of the instrument is the incident beam. In this instrument the incident beam is exposed from the end of the collimator, through the sample, to the beam stop, a distance of about 3 centimeters. The incident beam is about 0.1 mm in diameter. If no part of your body ever enters this beam path, then there is little possibility of acute bodily harm from the instrument. Also, whenever possible, keep the outer door of the instrument closed to keep even scattered radiation from coming into the room.

Exposure to the incident beam poses significant health problems that would produce health issues within hours. The scattered radiation could cause stochastic effects, or long-term effects such as cancer, genetic defects, or eye injury. Both acute and stochastic health issues can be mitigated by following the safety rules outlined below.

The Oklahoma Department of Environmental Quality oversees the activities of this laboratory. The DEQ follows the rules promulgated by the U.S. Nuclear Regulatory Commission. The DEQ encourages users to reduce any exposure to radiation by requiring that 3 general rules should be followed when working around radiation sources. These rules are called *ALARA*, As Low As Reasonably Achievable.

1. Reduce the time you are exposed to the radiation source.
2. Increase the distance between yourself and the radiation source.
3. Increase shielding between yourself and the radiation source.

In our lab these rules are achieved primarily by the design of the instrument. The closed protective enclosure of the instrument blocks all incident and scattered radiation. When the enclosure is opened, the X-ray safety shutter must close (a part of the fail-safe mechanism of the instrument). If any of the warning lamps that indicate when X rays are being generated should burn out, then the generator will turn off (another part of the fail-safe mechanism). *ALARA* goals are also achieved when the user keeps all parts of their body out of the incident beam path, and when the user keeps the enclosure door closed except when aligning their crystal.

A second potential hazard of the instrument is the electrical power of the instrument. The X-ray “generator” is a highly-regulated, DC power supply that typically operates at 50 kV and 1 mA. There are exposed wires at dangerous voltages in the lower cabinet of the instrument. If you drop anything under the instrument, let the lab director or some qualified electronics engineer retrieve the object for you.

Another potential hazard is the liquid nitrogen used to cool samples. The lab director will usually handle all transfers of liquid nitrogen.

All events regarding safety should be reported to the lab director (home: 248-8911, cell: 420-3800) and to the OU Radiation Safety Office (271-6121). If you feel there is a safety issue in the lab involving the radiation source, please report that issue to the OU Radiation Safety Office, and if you wish, to the Oklahoma Department of Environmental Quality. Contact

information to the DEQ is available on the notices posted near the safety shower. Before overriding any interlocks on the instrument, you *must* get approval to do so from both the CCL lab director and from the OU Radiation Safety Office.

If you are pregnant, or believe you may be pregnant, and you chose to not come into the CCL due to safety concerns for your unborn child, then arrangements can be made to have your data collected for you.

A first-aid kit, SDSs, and operating procedures are available on the shelf around the corner from the safety shower. The chemical spill kit is in the metal cabinet that is closer to the safety shower. General Laboratory safety information is at the [OU Laboratory Manual](#) page. Safety Data Sheets may be obtained at the [OU SDS](#) web site. Information about radiation safety in the X-ray laboratory is at the [radiation safety](#) page. Special procedures to be used in the Chemical Crystallography Laboratory are at the Laboratory Notes section of the [Notes & Manuals](#) page.