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 Bruker instrument in the Chemical Crystallography Laboratory has a sealed-tube radiation source, optics made up of both a graphite monochromator and a monocapillary collimator, an Eulerian goniometer with fixed χ arm, and an APEX ccd (charge-coupled device) area detector. Most of the time the instrument uses Mo $K\alpha$ radiation. The instrument also includes an Oxford Cryosystems Cryostream 700 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 from energetic electrons and the object that the electrons hit is a block of metal. Two types of X rays are emitted. A continuous band of radiation called Bremsstralung (breaking 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 these discrete lines is the difference in energy between the two orbitals in the metal, and is *characteristic* of the type of metal.

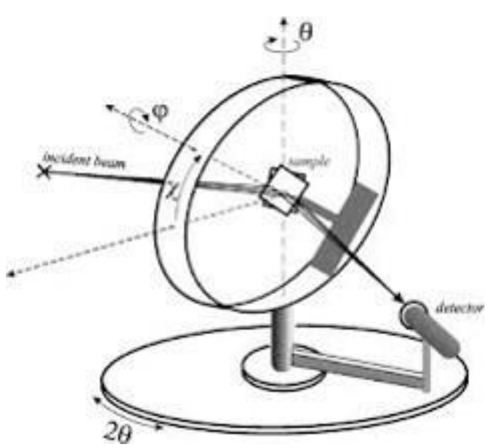
The optics of a single-crystal instrument serves the two functions of *collimating* the incident beam and *monochromatizing* the beam. The incident beam is made monochromatic by diffracting the X-ray beam off a block of another crystal. In our lab that crystal is a section of graphite cut on the (001) face. The beam is collimated using a tube that is narrow at both ends. The intensity of the incident beam is increased by reflecting the *divergent* incident beam back onto itself using many flat surfaces inside of the mono-capillary collimator.

Associated with the optics are two shutters to block the incident radiation. The first shutter is a safety shutter that is opened before collecting a set of frames and closed after collecting the set of frames. If the safety enclosure is accidently opened while data are being collected, the safety shutter will automatically close. A high-speed, rotary shutter is also in the beam path. The rotary shutter is used to set the time of the exposures of the detector.

The D8 goniometer moves the sample using a combination of ϕ (spindle axis of the goniometer head) and ω (parallel with the axis of the detector) motions. The χ arm is fixed at the angle

54.7° (the angle of one face of a cube to the body diagonal). The angle of the detector arm (sometimes called the 2θ or the swing arm) to the incident beam can also 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.

Detector  Source



Eulerian goniometer. The ω and θ circles are rotate around the same axis, with ω rotating the sample and θ rotating the detector. On our instrument χ is fixed. The φ circle is the spindle axis of the goniometer head.

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 is located to the beam stop, a distance of about 3 centimeters. This beam is about 0.5 mm in diameter. As long as no part of your body ever enters this beam path, there is little possibility of acute bodily harm. Also whenever possible, keep the outer doors 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 a matter of hours. The scattered radiation could cause stochastic effects, or long term effects such as cancer or genetic defects. Both acute and stochastic health issues can be mitigated by following the safety rules outlined below.

Both the Oklahoma Department of Environmental Quality and the U. S. Nuclear Regulatory Commission oversee the activities of this lab. Both organizations encourage users to reduce any harmful effects of exposure by requiring that 3 simple rules should be followed at all times. These rules are called *ALARA*, *A*s *L*ow *A*s *R*easonably *A*chievable.

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 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 doors 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. There are exposed wires at these or other dangerous voltages in the lower cabinet of the instrument. Thus, 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: 701-8725, cell: 420-3800) and to the OU Radiation Safety Office (271-6121). If you feel there is a safety issue in the lab, 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 closer to the safety shower.