X-ray Instruments

Martin Lutz

Bijvoet Center for Biomolecular Research, Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands. m.lutz@uu.nl

Introduction

An X-ray single crystal diffractometer principally consists of three parts: X-ray generator, goniostat, and detector.

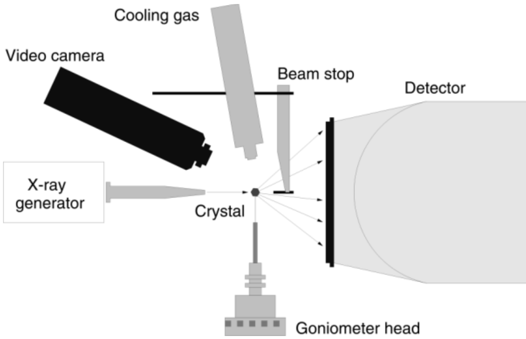

Figure 1. Setup of a single crystal diffractometer

X-ray source

For laboratory X-ray sources one can choose between sealed tube, microsource, rotating anode, and liquid anode. All these sources provide X-rays which are not monochromatic. To monochromatize the radiation, one can use filters, monochromators and/or focusing optics. Still, the monochromatic radiation will contain the two wavelengths of $K\alpha_1$ and $K\alpha_2$. Depending on the setup there can also be a significant contribution from the white Bremsstrahlung.

Figure 2. X-ray spectrum of the Mo diffractometer at Utrecht University

X-rays from monochromators or focusing optics are polarized. In general, X-rays from laboratory sources are divergent. Beam collimators or focusing mirrors are used to limit the beam size and divergence.

Synchrotron X-ray sources are known for their high intensity. They have a broad spectrum from which a specific wavelength can be chosen with a suitable monochromator. Usually, X-rays from synchrotrons are very parallel. They can have a small source size and they are polarized.

Goniostat

The goniostat is used to bring the crystal into diffracting position. During the common rotation/oscillation experiment, the goniostat is also used to rotate the crystal while the diffracted intensity is recorded. It is highly recommended that for the rotation experiment, the axis is oriented perpendicular to the primary beam. The precision of the goniostat motors can differ between manufacturers. It can also differ between the crystal positioning and the rotation experiment.

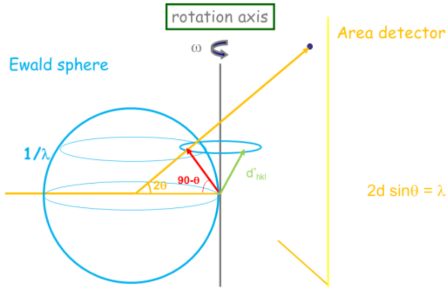

Figure 3. Diffraction geometry of the rotation method

The easiest setup for a goniostat has a single rotation axis. A single rotation axis gives no difficulties with collisions. The setup is thus very suitable also for large detectors. Crystal mounting robots and open-flow cooling equipment can easily be installed. Associated with the single axis experiment is a blind region which is wavelength dependent. The blind region can be overcome with the use of a mini-kappa goniometer head.

More flexible is a four-circle diffractometer. By moving the different motors a blind region is avoided. As additional advantage, the same reflection can be measured redundantly in different geometrical settings. The orientation of the crystal along certain crystal axes can be easily accomplished. Goniostats with an Eulerian cradle have the advantage of high mechanical stability. They can be operated with heavy equipment such as high-pressure cells or closed low-temperature units. Goniostats with Kappa geometry have the upper half-sphere available for equipment as for example an open-flow low-temperature unit. The mechanical collision map in four-circle diffractometers can be rather complicated.

A special case of the four-circle diffractometer has a fixed χ angle of 45°. Here, only three axes can be moved.

Detector

Historically the oldest detection method is the photographic film. It is a 2D detection technique but involves a lot of work for the chemical development of films in the dark room and the estimation of intensities.

In the 1960's the film methods were slowly superseded with automatic diffractometers using scintillation counters. These were point detectors and they could directly convert the X-ray photons into detectable electrons. Usually, these diffractometers had a fixed detector distance.

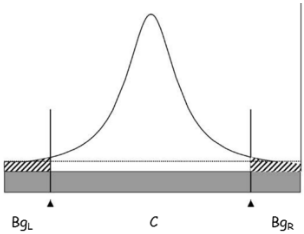

Figure 4. Background-Peak-Background method for the determination of I and σ.

The area detector returned to crystallography in the 1980's with multiwire detectors. In 1986 the first image plate detector was introduced. After the exposure, the image plate is read-out by scanning with a laser and afterwards reset with a flashlight. The plate is then ready for a new exposure. In the 1990's the CCD detector for Mo radiation appeared on the market. After the exposure, the chip can be read out electronically. This gives a speed advantage with respect to the image plate. The most recent detector technology is based on CMOS chips which have a very fast read-out time and allow the so-called "shutter-less measurements".

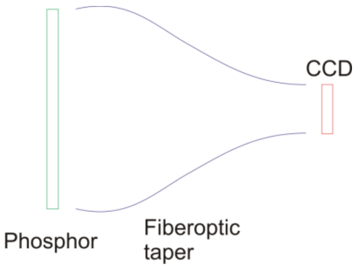

Figure 5. Schematic drawing of a CCD detector.

Detector quality is mainly determined by pixel size, presence/absence of a fiberoptic taper, dynamic range, sensitivity, and noise level. Shortcomings of the detector can sometimes be compensated by a higher redundancy. The detector speed can thus contribute to the data quality.
With modern detectors it is possible to acquire a large amount of data in a short period of time. A corresponding infrastructure for computing and data storage is necessary.

References:

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http://www.xtal.iqfr.csic.es/Cristalografia/parte_06-en.html