RECENT ADVANCES IN FLAT FILM SINGLE CRYSTAL CAMERA DESIGN for structure studies of large unit cell biological and protein crystals

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The study of crystal structure has become vital to molecular biologists and to scientists working in the field of protein chemistry. The scientists working in this field found wide use of single crystal four-circle computer-controlled diffractometer for measuring reflections from a single crystal all over the Ewald sphere for complete evaluation of crystal structure. The speed and accuracy of the four-circle diffractometer was preferred to photographic methods like Weissenberg and Buerger precession cameras.

Neverthless, photographic techniques, by virtue of their being inexpensive and other inherent advantages, never lost their importance. Apart from simplicity, the intensities once recorded on photographic film are permanent and because of comparatively shorter exposure time, the biological crystal material is less susceptible to damage by the exposure of X-rays. Owing to such reasons, the Weissenberg cameras and precession cameras are still widely used in crystallographic laboratories.

As the Molecular biologists and chemists succeeded in developing large sized crystals, crystal analysis in many laboratories became the central problem. The hitherto commonly available single crystal cameras were not entirely suitable for large molecule crystallographers because of limited dispersion, distorted spot recording, layer-screen method of data collection and much less usable information area on the photographic film in one exposure. With the advent of computer-controlled programmable Microdensitometer, it became possible to evaluate reciprocal lattice reflections on the film which do not lie on the straight line as the position of the diffraction spots could be computed and as such screenless layer line recording with flat film cassette in normal beam rotation geometry became a desired method.

Dr. U.M. Arndt and his group in the famous centre of large molecule crystallography — the Medical Research Council at Cambridge, developed a new photographic data recording system known as Arndt-Wonnacott oscillating camera. This device dispenses with the layer-line screening and operates with flat film in normal beam rotation geometry. Screenless rotation method has its main application, and probably is the only method, in biological crystal structure studies in which reflections from crystals with spacing greater than 2A° to nearly 200A° are recorded by screenless rotation method.

In the screenless method, the crystal movement should be very small [illegible] the super-imposition of reflections and in consequence the reflection in one exposure will be partially recorded as the reciprocal lattice points have not completely passed the EWALD sphere or sphere of reflection due to very small rotation of the crystal. With the increase in unit cell size the movement should be still smaller as the quantum of reciprocal lattice points is governed by the mosaic spread of the crystal and its size, increasing with the size of the crystal (as given by formula 19.74 x volume/ $\sqrt{3}$). Therefore, high standard of efficiency is achieved when it is possible to sum the parts of spots recorded on two successive exposures. Unlike Precession methods, in rotation method the reciprocal lattice points pass through the surface of Ewald sphere and hence it is not required to apply the Lorentz correction factor.

The Arndt-Wonnacoot oscillation camera is a high precision camera. The crystal can be correctly aligned accurately in the X-ray beam with the help of a microscope mounted on the rotation-oscillation device. The horizontal spindle of the rotation oscillation device is driven by a 500 step per revolution stepping motor. One step of motor thus gives a motion of .002° of the crystal. The backlash is not more than one step of the motor, that is, 002°. The backlash is further reduced by the overshoot during reverse movement and then returning to the desired position. For very long exposures, the crystal is oscillated through a range 10 x and 100x during each exposure.

The camera turntable accepts 8 flat film cassettes brought successively in the normal beam by a programmer. The automatic film changing guarantee long time data collection without further attention. The film cassette is perpendicular to the incident beam better than two minutes of the arc both in the horizontal and vertical planes. To achieve desired dispersion, the film to crystal distance can be adjusted from 50 to 120 mm. The shutter automatically closes the X-ray window during cassette change or crystal shaft rotation. The crystal shaft rotation per exposure can be set between .05° and 4.95° and speed of shaft rotation can be adjusted within 40 x 10^4 sec./degree to 5 sec/degree in 13 steps.

Facilities are also provided for stepping the crystal back by 0, 4, 8, 20, 40 or 100 per cent of the chosen scan before starting next exposure to permit overlap of angular ranges. The scan angle is indicated by a digital display with an accuracy of .001°. For manual operation e.g. during crystal can be rotated with speeds up to 180°/minute while the angular indicator is following this movement. Provision is also made for the mounting of Rasmussen monochromator.

The A-W oscillation camera is a useful facility now available to biological chemists and fills a void between the more expensive single crystal diffractometers and hitherto available conventional single crystal cameras. Very many reciprocal latttice reflections can be recorded on flat films automatically without requiring any further attention once the camera is set. The camera can be easily mounted on Enraf generator tube shield and also on other make generator tube shields with the help of suitable adaptors available.