14-Diffraction Physics and Optics

High accuracy is not critical for strong reflections when determining crystal structures with X-ray structure factors. The least squares residuals for refinements of the atomic coordinates are dominated by high angle reflections which, on average, are less intense. When measuring vibration tensors it is sufficient to achieve comparable relative precision for low and high angle structure factors. Different criteria apply to high precision diffraction imaging of the deformation density $\Delta p$. Small single crystals are used increasingly for those studies, to ensure that no extinction correction is far from unity, ensuring that the reliability of the extinction corrections is not limited by the uncertain validity of the underlying theory.

If the high order reflections are measured for very small crystals using a conventional X-ray tube source, the precision of $\Delta p$ images is often limited by counting statistics. Poisson statistics errors can be reduced dramatically by using synchrotron radiation. The smoothness of the $\Delta p$ maps for recent synchrotron radiation experiments indicates that precision is no longer limited by counting errors.

The reproducibility of synchrotron radiation $\Delta p$ maps does not yet approach estimates based on statistical errors alone. The accuracy of most $\Delta p$ images is limited by residual systematic error for the strong low order reflections. It is the absolute error in the structure factors, and not their relative precision, which limits the precision of diffraction images of the deformation density. The accuracy desired for the strong reflections imposes serious demands on how well the absorption, extinction and dead-time corrections are to be evaluated.

Precise absorption corrections require correctly indexed faces, measured with a precision which, for small crystals, taxes the power of optical microscopes. Care is required when evaluating absorption corrections by the analytical formula (N. W. Alcock, Acta Cryst., 1974, B30, 639-644) to retain precision near points where the basic expression is indeterminate. The extinction correction formula of Zachariasen (Acta Cryst., 1967, A23, 558-564) implies tighter constraints on structure factor magnitudes which are not necessarily satisfied in practical cases. Dead-time corrections applied automatically by the circuitry are not necessarily precise enough for high precision studies.

Methods for attaining and checking the precision of absorption, extinction and dead-time corrections for standard diffraction experiments on small crystals will be described.