s6.m22.o3 **Precise Absolute-Structure Determination in Light-Atom Crystals.** Simon Parsons[a] and Howard Flack[b], [a]*The University of Edinburgh, Scotland and* [b]*Université de Geneve, Switzerland. E-mail: S.Parsons@ed.ac.uk*

**Keywords: Absolute Structure; Absolute Configuration; Refinement**

Absolute-structure determination (*e.g.* for absolute-configuration determination from enantiopure crystals) may be ambiguous, particularly in the case of 'organic' or 'light-atom' structures. The cause of the ambiguity lies in the weakness of the resonant scattering (anomalous dispersion) producing the intensity differences between Friedel opposites that may be analysed to reveal information on absolute structure. The magnitude of these differences depends on the wavelength of the X-rays used to collect the diffraction data and the atomic composition of the crystal. Specifically, it is the imaginary contribution (*f*") to the scattering factor which must be significant if the absolute structure is to be determined with acceptable precision.

In an absolute-structure determination one absolute structure is refined competitively against the alternative. The result is expressed by the Flack (1983) parameter $x(u)$ which for absolute-configuration determination can be interpreted as the mole fraction of the alternative enantiomer in the crystal. The physical range of $x$ is 0 to 1 and even if the bulk material is known to be enantiopure the standard uncertainty ($u$) should be less than 0.08 before any firm conclusions regarding the absolute structure can be drawn. This criterion has proved to be extremely demanding for light-atom structures.

It is possible to record the intensities of Friedel opposites in a way that systematic errors such as absorption and extinction are identical for the two measurements. One procedure involves measuring the intensity $I(\mathbf{h})$ of one reflection at setting angles 2θ, ω, χ and ϕ, and the intensity of its Friedel opposite $I(-\mathbf{h})$ at -2θ, -ω, χ and ϕ. These measurements can be combined in the form:

$$D_{obs}(\mathbf{h}) = \frac{I(\mathbf{h}) - I(-\mathbf{h})}{I(\mathbf{h}) + I(-\mathbf{h})} \cong (1-2x)\frac{|F(\mathbf{h})|^2 - |F(-\mathbf{h})|^2}{|F(\mathbf{h})|^2 + |F(-\mathbf{h})|^2}$$

and the quantities $D_{obs}(\mathbf{h})$ which are thus free from systematic errors are used as observations (restraints) in a least-squares refinement along with the conventional intensity data and including all the usual parameters.

Symmetry-equivalent measurements of $I(\mathbf{h})$ and $I(-\mathbf{h})$ were carried out for six inversion-sensitive reflections of L-alanine and the mean values of $D_{obs}(\mathbf{h})$ were applied as restraints. Whereas the unrestrained absolute-structure refinement converged to $x$ = -0.1(2), application of restraints yielded $x$ = -0.03(7), an improvement in precision by a factor of three. This determination has strong inversion-distinguishing power, and enables a positive statement to be made about the absolute configuration of the molecule studied.

Flack, H.D. (1983). *Acta Cryst.* A**39**, 876-881.

s6.m22.o4 **Image Plate and CCD Detectors for Light-Atom Absolute Structure Determination: Comparison and Contrast.** Joseph D. Ferrara, Lee M. Daniels, Ron Benson, Hiromi Ota[b], Katsuanari Sasaki[b], Maksymilian Chruszcz[c], [a]*Rigaku/MSC, Inc., 9009 New Trails Drive, The Woodlands, TX 77381,* [b]*Rigaku Corporation, 3-9-12 Matsubara-cho, Akishima-shi TOKYO 196-8666 JAPAN,* [c]*University of Virginia, Department of Molecular Physiology & Biological Physics, 1300 Jefferson Park Avenue, Charlottesville, VA 22908, USA. E-mail: jdf@rigakumsc.com*

**Keywords: Absolute Structure; Detectors; Image Plate**

CCD detectors offer advantages such as very fast readout and high sensitivity when collecting X-ray diffraction data. But when using Cu (or other long-wavelength) radiation one is limited by the solid angle intercepted by the CCD phosphor, and therefore multiple scans at low and then high 2θ angles are required in order to obtain a complete data set and to insure adequate redundancy in the data. The use of a curved image plate may be advantageous in this situation, since both negative and positive 2θ reflections can be collected simultaneously while very high resolution data are included at the same time. But even when data are collected very carefully using a curved imaging plate, is it possible to unequivocally determine absolute structure when no atoms heavier than oxygen are present? Comparisons of detector performance with such crystals will be presented and critically analyzed.