02-Methods for Structure Determination and Analysis, Computing and Graphics

Xtal3.2 includes 30 programs especially for macromolecular calculations. Additional applications are under development. Current macromolecular programs include merging and scaling of multiple data sets from heavy-atom derivatives; MIR phasing; geometrically constrained refinement (PROLSQ); energy minimization restrained refinement (CEDAR); density modification; maximum entropy phasing; and forward and reverse fast-Fourier transform calculations. All programs are symmetry general. Soft interfaces to other packages such as FRODO, XENGEN, SCHAKAL, MOGLI, SHELX and Mathematica are also incorporated. §

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PS-02.08.20 SOFTWARE SYSTEM FOR MICROCRYSTALLOGRAPHY WITH WHITE SR LAUE METHOD, By K.Hagiya* (1),T.Takase(2), Y.Shimizugawa(3), K.Ohsumi(4), M.Miyamoto(2) and M.Ohmasa(1)
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For structual studies of very small crystals with the sizes of submicrometer range, the Laue method combined with synchrotron radiation(SR) has been proved to be a powerfull tool(K.Ohsumi et al.,1991,J.Appl.Cryst.,24, 340-348). A software system to deal with the Laue pattern recorded on an imaging plate(IP:Fuji Co.Ltd; J.Miyahara et al.,Nucl.Instrum.Methods,1986,A246, 572-578) has been developed and successfully applied to some inorganic materials(K.Ohsumi et al.,1992,Rev.Sci. Instrum.,63(1),1181-1184).
The software systems for the Laue method have been developed and applied to many cases of protein crystals (for example; J.R.Helliwell et al.,1989,J.Appl.Crysta., 22,483-497),but it is unsuitable to inorganic materials in the sense of precise determination of crystal structures .
The features of the present software is that the intensities of Laue spots which are superposition of several Bragg reflections are used as observed values in the course of least-squres refinement of the structure, and an evaluation of the result obtained by this refinement is made.
The procedure of data processing and structure refinement are shown below.

Data processing
1.determination of indices of Laue reflections
based on interplane angles.
2.simulation of the Laue pattern
to confirm indices assigned.
3.refinement of crystal orientation
based on coordinates(x,y) of Laue spots on IP.
with parameters of camera length, origin of IP, inclination of IP and axial ratio of the sample.
4.integration of intensities
with determination of background level.

Refinement of a structure
1.data correction for absorption and extinction
if necessary.
2.refinement of the structure
based on intensities of Laue spots
by minimizing R

$$R = \frac{\sum_h |I_O(h) - k\cdot I_C(h)|^2}{\sum_h |I_O(h)|^2} \qquad \sigma^2(p_i) = M_{ii}^{-1}\{\frac{\sum_{n-h} W\Delta^2}{m-n}\}$$

considering such factors as, structural parameters, polarization of incident white SR, spectrum of incident white SR, and quantum efficiency of IP with respect to the wavelength.
3.evaluation of the whole process of the refinement
based on the comparison of observed and calculated structure factors,

$$r = \frac{\sum ||F_O(nh)| - |F_C(nh)||}{\sum |F_O(nh)|} \qquad |F_O(nh)| = \{I_C^B(nh)\times\frac{I_O(h)}{I_C(h)}\}^{1/2}$$

and symmetry related reflections

$$R_{eq} = \frac{\sum_{i\neq j} ||F_O(h)_i| - |F_O(h)_j||^2}{\sum_i |F_O(h)_i|^2}$$

PS-02.08.21 SYMMETRY TESTING DURING LEAST-SQUARES REFINEMENT. By H.D. Flack, Laboratoire de Cristallographie, University of Geneva, 24 quai Ernest-Ansermet, CH-1211 Genève 4, Switzerland.

One is aware from the work of R.E. Marsh and others of the fairly frequent occurrence of the publication of structures in a space group of lower symmetry than that really necessary for the diffraction data used in the structure refinement. Symmetry testing programs such as Le Page's *MISSYM* enable potential cases of incorrect symmetry to be detected from the assumed space group, the cell dimenisons and the refined atomic coordinates. However a robust methodology for quantifying the deviations (and their statistical significance) of a structure in one space group from that in another are clearly lacking. The poster will present the first steps in the development of such a suitable technique and some practical tests of its application. The basis of the technique is to split the electron density of the model into a fully symmetric part and an 'anti-symmetric' component premultiplied by a global population parameter. The 'anti-symmetric' component expresses the deviations of the low space-group symmetry structure from the best expression of the diffraction data that can be obtained in the high-symmetry space group. Such a decomposition respects Marsh's criterion that the atomic electron density representation in the low- and high- symmetry space groups should be identical in order to avoid an implicit 'anharmonicity' being interpreted as an explicit 'non-centrosymmetricity'. The correct treatment of any potential twinning is another essential element of the method. Test examples are drawn from the literature *e.g.* 1,8 octanediamine dihydrobromide, $C_8H_{20}N_2.2HBr$ [Brisson, J. and Brisse, F. (1984) *Acta Cryst.* C**40**, 1405-1407]. It will be shown that the use of a realistic atomic model is essential to the treatment of such cases. The question of the quantification for non-centrosymmetric space groups of their polarity and/or enantiomorphism is also being considered.

PS-02.08.22 A SYSTEMATIC STUDY OF COORDINATE PRECISION IN X-RAY STRUCTURE ANALYSIS: INDICATORS OF STRUCTURAL PRECISION FOR USE WITH THE CAMBRIDGE STRUCTURAL DATABASE. Jason C.Cole* and Judith A.K.Howard, Department of Chemistry, University of Durham, South Road, Durham DH1 3LE, UK and Frank H. Allen, Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK.

Experimental and structural features affecting the precision of the atomic coordinates of any atom A, as determined by X-ray analysis, were studied by Cruickshank [Acta Cryst. (1960), 13, 774-777]. He showed that $\bar{\sigma}$(A)

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