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. §

Stewart's contribution to this work has been supported under contract N00014-92-J-1556 of the Office of Naval Research.

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)

was inversely proportional to $\bar{s}$ (the mean reciprocal radius for the observed reflections) and to $\sqrt{p}$ (where p is the difference between the number of observed reflections, Nr, and the number of refined parameters, Np). Further, $\bar{\sigma}(A)$ is shown to be proportional to the R-factor and to $\sqrt{ZA}$, where ZA is the number of atoms of A needed to give a scattering power at s which is equal to the scattering power of all atoms in the asymmetric unit.

The possibility of estimating $\bar{\sigma}(A)$ using some equation similar in form to that of Cruickshank is of interest in screening entries in the Cambridge Structural Database (CSD) prior to their inclusion in data analysis projects. The esd's of individual coordinates have only been included in the CSD for structures published since 1985. The only precision indicators available for ALL entries are the R-factor and a flag (AS) which categorizes the mean esd of a C-C bond length into four numerical bands: 0.001-0.005 (AS = 1), 0.006- 0.010 (AS=2), 0.011-0.030 (AS=3), and 0.031 and above (AS=4). Not only are the upper bands too broad, but AS is also unavailable for some 16% of CSD entries. Preliminary attempts to estimate $\bar{\sigma}(C\text{-}C)$, and hence $\bar{\sigma}(C)$, were described briefly by Allen & Doyle [Acta Cryst.(1987), A43, C291] but the number of entries having coordinate esd's was then too small (ca. 4000). We now extend the study to encompass the ca. 25,000 entries having esd's and with R < 0.080 that are currently available.

We have used the methods of correlation, simple linear regression and multiple linear regression to study the relationship between $\bar{\sigma}(C\text{-}C)$ (the dependent variable) and a variety of 'independent' variables which are available in each CSD entry or which can be calculated from the stored information. These include such items as: R-factor, Zmax (the maximum atomic number in the structure), ZA (as defined by Cruickshank), T (the temperature of data collection), Na (the number of atoms in the asymmetric unit), etc. We find that only the R-factor and some function of the atomic numbers (Z) are sufficiently independent for use in regression procedures.

For a dataset of 25959 entries with R < 0.08 and $\bar{\sigma}(C\text{-}C)$ in the range 0.001 to 0.040, we find that an expression of the form:

$$\bar{\sigma}(C\text{-}C) = k\,R\,ZA$$

will estimate 75.5% of $\bar{\sigma}(C\text{-}C)$ values within 50% of their reported values (79% within + or - 0.005A of their reported values in absolute terms). Results obtained with regression expressions of the form

$$\bar{\sigma}(C\text{-}C) = a + k\,R\,ZA \text{ or } \bar{\sigma}(C\text{-}C) = a + bR + cZA$$

are marginally less effective estimators.

For 687 of these entries, we have added values of Nr/Np (no. of observed reflections/no. of refined parameters) directly from the literature (these values are not available in the CSD). Here again the simplest regression, now $\bar{\sigma}(C\text{-}C) = k\,R\,ZA\,/\,(Nr/Np)$ to follow Cruickshank's general philosophy, provides the best estimation, with 84.4% of the $\bar{\sigma}(C\text{-}C)$ being estimated within + or - 50% of their reported values (86% within 0.005A of the reported values in absolute terms).

A variety of graphical and numerical statistics concerning structural precision will be presented as general background to the correlation and regression experiments.

PS-02.08.23 SOFTWARE FOR X-RAY AND NEUTRON DIFFRACTION EXPERIMENT. By V.E.Anisimov,N.B.Bolotina, L.F.Malakhova.* Institute of Crystallography of Russian Academy of Sciences, Moscow, Russia.

New software was developed for certain 4-circle X-ray and neutron diffractometers controlled by a PC computer. The software is based on measurement techniques developed in our and other structural labs, experience of the diffraction measurements and capabilities of the PC computer. Some procedures of diffraction intensity measurements for perfect high symmetrical crystals, for incommensurate modulated phases, for twins, for samples with restricted angle settings (eg. in diamond cell), for intensity distribution measurements in given part of reciprocal space are involved.

The software contains an effective procedure of automated unit cell determination, measurements in the general geometry of angle setting (azimutal rotation)(Bolotina N.B. Kristallografiya, 1989, 34, 4,993, Bolotina N.B.,Chernaya T.S.,Golubev A.M. Kristallografiya, 1990, 35, 2, 303) and correct calculation of optimum measurements time. Comprehensive calculation and results of the measurements are collected in the expanding table which is suitable for subsequent analysis and treatment of experimental information. The software is written in FORTRAN and MSDOS system.

PS-02.08.24

RUNNING XRS-82 RIETVELD SYSTEM IN PC LEVEL MICRO COMPUTER UNDER MS-DOS OPERATING SYSTEM By Yihua XU. Department of Chemistry, Jilin University, Changchun 130023, China.

The XRS-82 Rietveld system is a useful package of programs for structural parameters refinement of powder diffraction data with whole pattern. The system had been written by Prof. Ch. Baerlocher and about fifty authors had great contributions (Ch. Baerlocher. The XRS-82 X-ray Rietvels System. Institut fuer Kristallographie, ETH. Zurich, Switzerland). Many structures, especially for zeolites, had been refined by the system and good results obtained. Unfortunately it seems there are some limits to use the system; 1. The system needs a large memory of the computer. So only a few research workers, who have a big or supper-computer, can use the system. 2. The system needs a set of step scan diffractional data collected on a diffractometer. So the computer should be connected with a diffractometer or else the scanning data should be transmited into the computer. It is not easy to do it for a big or super computer. 3. It is too expensive for running the system in large or a super computer. 4. Some programs for display and drawing must be modified in different computers, that is time-consuming. As it is well known, the micro personal computers, such as IBM PC/XT, AT 286, 386 or 486 and their compatible computers used in many research groups for many years, particularlye in China. Some X-ray diffractometer have beed supplied micro computer to control and collect data. So it is very convenient to use the XRS-82 system by more researchers if the system can be used in a micro computer. In order to run the system on a micro personal computer, we try to rewrite the system. Although it is difficult to load big programs into a small computer. In this paper we report the rewriting of the system on the Micro-Computer with MS-DOS V3. 3 operating system. The XRS-82 system has 12 main programs: STEPCO, PEAK, SPRING, DATRDON, LOAD, CRYLSP, FOUR, BONDLA, PROPT, RELIST, PEKP and WRITU. All of these programs had been written in FORTRAN IV, and in super computer they would be loaded together and