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

considering such factors as, structural parameters, polarization 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,

\[ R = \sum |F_{o} - F_{c}| \sum |F_{o}| \]

and symmetry-related reflections

\[ R_{int} = \sum \frac{|F_{o} - F_{c}|}{\sqrt{2} \sum |F_{o}|} \]

PS-02.08.20 SOFTWARE SYSTEM FOR MICROCRYSTALLOGRAPHY

using multiple programs especially for macromolecular calculations. Additional applications are under development. Current macromolecular programs include merging and scaling of multiple data sets from every atom derivative; MMJ phasing; geometrically constrained refinement; energy minimization restrained refinement (CEDAR); density modification; maximum entropy phasing; and forward and reverse Fast Fourier transform calculations. All programs use symmetry general. Soft interfaces to other packages such as FRODO, XENGEN, SCIRAX, MOGLI, SHELXL and Mathematica are also incorporated. Stewart's contribution to this work has been supported under contract N00014-92-J-1550 of the Office of Naval Research.

PS-02.08.21 SYMMETRY TESTING DURING LEAST-SQUARES REFINEMENT

One is aware from the work of R.E. Marsh and others of the importance of using the structure of space group of lower symmetry than that really necessary for the diffraction data used in the structure refinement. Symmetry testing programs such as LEAP's MDDK enable potential cases of incorrect symmetry to be detected from the assumed space group, the cell dimensions 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 is clearly lacking. The paper 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 multiplied by a global population parameter. The 'anti-symmetric' component expresses the deviations of the low symmetry group 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 any implicit 'anomalous scattering' being interpreted as an explicit 'non-centrosymmetry'. 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 octanodiamine dihydrobromide, CgHgN2·2HBr [Brisson, J and Brisse, F (1984) Acta Cryst. C40, 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 of non-centrosymmetric space groups of their polarity and enantiomorphism is also being considered.

was inversely proportional to \( s \) (the mean reciprocal radius for the observed reflections) and to \( s + f \) (where \( f \) is the difference between the number of observed reflections, \( N_r \), and the number of refined parameters, \( N_p \)). Further, \( \Delta(\Lambda) \) is shown to be proportional to the R-factor and to \( C_{\text{ZA}} \), where \( Z_A \) is the number of atoms of A present to give a scattering power at \( s \) equal to the scattering power of all atoms in the asymmetric unit.

The possibility of estimating \( \Delta(\Lambda) \) using some equation similar 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 sets 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 \( A(5) \) which categorizes the mean end of a C-C bond length into four numerical bands: 0.001-0.005 \( A(5) - 1 \), 0.006-0.010 \( A(5) - 2 \), 0.011-0.020 \( A(5) - 3 \), and 0.021 and above \( A(5) - 4 \). Not all the upper bands are broad, but \( A(5) \) is also unavailable for some 19% of CSD entries. Preliminary attempts to estimate \( \Delta(\Lambda) \), and hence \( G(\Lambda) \), were described briefly by Allen & Dowty (Acta Crystallogr. 1987), A43, C291 but the number of entries having coordinate cts was too small (ca. 4000). We now extend the study to encompass the ca. 25,000 entries having cts and with \( R < 0.01 \) that are currently available.

We have used the methods of correlation, simple linear regression and multiple-linear regression to study the relationship between the observed dependent variables and a variety of independent variables which are available in each CSD entry or which can be calculated from the stored data. These include such items as: \( R_{\text{factor}} \), \( Z_{\text{max}} \) (the maximum atomic number in the structure), \( C_{\text{ZA}} \) (as defined by Cruickshank), \( T \) (the temperature of data collection), \( N_{\text{A}} \) (the number of atoms in the asymmetric unit), etc. We find that only the R-factor and some function of the atomic numbers \( Z_A \) are sufficiently independent for use in regression procedures.

For a dataset of 25,000 entries with \( R < 0.01 \) and \( G(\Lambda) \) in the range 0.61 to 0.40, we find that an expression of the form

\[
\Delta(\Lambda) = k R C_{\text{ZA}}
\]

will estimate 75.5% of \( G(\Lambda) \) values within 50% of their reported values (72% within \( + \) or \( -0.005 \)A of their reported values in absolute terms). Results obtained with regression expressions of the form

\[
\Delta(\Lambda) = a + k R C_{\text{ZA}} C_{\text{Z}}
\]

are marginally less effective estimators.

For 660 of these entries, we have added values of \( s N_r / N_p \) (no of observed reflections/no. of refined parameters) directly from the literature (these values are not available in the CSD). Here again the simplest estimation, \( \Delta(\Lambda) = k R Z_A / s N_r / N_p \), follows Cruickshank's general philosophy, provides the best estimation. With 46.4% of the \( \Delta(\Lambda) \) being estimated within \( + \) or \( -0.5 \)A 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-02.02.24**

**RUNNING XRS-82 RIETVELD SYSTEM IN PC LEVEL MICRO COMPUTER UNDER MS-DOS OPERATING SYSTEM**

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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. Baelen and about fifty authors had great contributions (Ch. Baelen. The XRS-82 X-ray Rietveld System, Institut fur Kristalllographie, 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 super-computer, can use the system. 2. The system needs the 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 transmitted 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, particularly in China. Some X-ray diffractometer have bred 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 on 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, DIFTRON, LOAD, CRYLSY, FOUR, BONDIA, PROPT, RELIST, PEK and WRITL. All of these programs had been written in FORTRAN IV, and in super computer they would be loaded together and