# Short Communications

## A Series of Crystallographic Computer Programs for CD-3300

T. DAHL, F. GRAM, P. GROTH, B. KLEWE
and CHR. RØMMING

*Department of Chemistry, University of Oslo,
Oslo 3, Norway*

The crystallographic program set at the University of Oslo is written in Mass Storage FORTRAN language for CD-3300 and operates under the MASTER system. This implies that only a core storage of 32 K 24 bits words is available (floating point number occupying two words).

Programs and data are stored on disks. Two different disk files contain the structural data.

File No. 1 contains (as integers): $h$, $k$, $l$, $F$, $\sigma(F)$, $\sin^2\theta/\lambda^2$, IUNOBS, $\chi$ or E, $\phi$ or P, where IUNOBS marks unobserved reflections, $\chi$ and $\phi$ are diffractometer angles, E normalized structure factors, and P their phases.

File No. 2 consists of seven records:
Record 1: cell parameters etc.
  » 2: data for phase determination
  » 3: scale factors *etc.*
  » 4: atomic form factor tables stored as $f(\sin\theta/\lambda)^{-\frac{1}{2}}$
  » 5: equivalent position matrices
  » 6 and 7: atomic parameters (programs changing these parameters put the result on record 7 while starting parameters are stored on record 6).

*Data collection and reduction.* (a) *Film data.* Corrections for Lorentz and polarisation effects as well as cross layer scaling are carried out for Weissenberg and precession diagrams. Film scaling within each layer and layer scaling by structure factor calculation are optional.

(b) *Diffractometer data.* Refinement of cell parameters from diffractometer setting measurements and diffractometer setting data are obtained from programs written by A. T. Christensen and H. Hope.[1] Corrections for Lorentz and polarisation effects are carried out. Use of crystal monochromator and uncertainties in attenuator constants as well as in reflection scaling are taken into account.

Relevant results from these programs are stored on file No. 1.

*Structure determination.* The structure factors are statistically[2] put on absolute scale (scale factor $K$) and the overall temperature factor, $B$, together with the corresponding unitary (or normalized) structure factors are calculated. Optionally may $K$ and $B$ be input parameters. The Harker-Kasper inequalities:[3]

$$(U_h \pm U_{h'})^2 \le (1 \pm U_{h+h'})(1 \pm U_{h-h'})$$

are applied systematically[4] with the resulting sign relationships, the U-distribution and all Sayre-interactions among the 40 largest unitary structure factors as output.

On the basis of this output one may apply Sayre's equation[5] systematically. Maximum eight symbols[6] may be introduced and the sign indications together with the corresponding probabilities[7] are printed.

An alternative way of obtaining a basic set of signs is the Cochran-Douglas method[8] combined with "zero-check".[9] The program permutes all combinations of maximum 15 signs and prints the corresponding $\chi$- and $\psi_0$-values for the most probable sets.

The basic set may be extended by means of Sayre's equation.[7] The probabilities[9] as well as the number of interactions are used as criteria in an iterative process. The accepted signs are placed on file No. 1 for Fourier or E-map calculation.

Trial structures may be tested by programs based on the minimum residual method.[10]

Translations of all (or a part of) the atoms through desired areas of the cell,

calculating the $R$-value for each step, are also possible.

*Structure refinement.* The structure factor routine written by Gantzel *et al.*[11] is applied. The Fourier programs handle all space groups. Maps are printed or placed on file for peak scanning (peaks being stored on file No. 2). The Patterson programs have options for sharpening and origin peak removal. It is possible to weight[12] or omit reflections in Fourier syntheses based on structure factor calculations.

In the least squares refinement programs the multiplicity factors may be variables and anomalous scattering taken into account. A weight analysis is carried out. The maximum numbers of parameters that can be refined simultaneously range from 121 to 127.

The block diagonal version (modified by J. Solbakk) may handle 250 parameters.

Matrix generation and inversion routines are optimized in COMPASS language by O. Aurmo.

*Absorption and extinction corrections.* A program written by Larson *et al.*,[13] utilizing a numerical integration method,[14] has been modified. The crystal shape and dimensions are characterized by Cartesian coordinates of three points in each of maximum 25 surface planes. Weissenberg or diffractometer data (with $\omega = 0$) may be corrected.

Corrections for secondary extinction effects are based on a formula given by Zachariassen[15] and the procedure is essentially as described by Åsbrink and Werner.[16] * Data collected with monochromized radiation may be handled.

*Miscellaneous programs.* Bond distances and angles, dihedral angles, *etc.* are calculated together with corresponding standard deviations from the variance-covariance matrix stored by the least squares program. Corrections in interatomic distances due to riding or independent motion[17] are carried out. The programs are partially based on ORFFE.[18] Scan of interatomic distances within specified limits (with corresponding angles) may be performed.

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* It should be noted that the introduction of the normalizing factor $1/A^{*\prime}(0)$ in the correction term of Zachariassen's formula, presupposes that the crystal has a spherical shape, and is not correct in the case of Åsbrink and Werner's non-spherical crystal.

Calculations of least squares planes, hydrogen atom positions, cell dimensions from $2\theta$-measurements, Dirichlet's reduced cell,[19] and analysis of atomic anisotropic thermal vibrations may be carried out.

The rigid-body motion analysis program of Trueblood and Shoemaker[20] (TLS) is adapted to the program system, and positional parameters corrected for libration are stored on file No. 2.

Programs for editing and updating of the data files and storing of structural data on magnetic tapes are available.

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