

16.2-8 DIFFRACTOMETER SOFTWARE (CAD4F).

by J.L. de Boer^x and A.J.M. Duisenberg^{xx}

x) Laboratory of Inorganic Chemistry, Materials Sciences Center, University of Groningen,
 xx) Dept. of Structure Chemistry, University of Utrecht, The Netherlands.

The CAD4F/OS8 (FORTRAN IV) software is updated and now includes as new facilities:
 ANIVREC: analyzing and handling of high anisotropic mosaicity. Also suited for split and most types of twinned crystals. (1)
 BATCH: diffractometer control input is read from a batch input file, enabling the user to compose a complete, automatic, job.
 CONE SEARCH: looks for a particular reflection, lying on the surface of a cone, whose axis is given by the position of one known reflection.
 (LOW) TEMPERATURE CONTROL: computer controlled, accurate temperature setting (range: 85-305K, stability: $\pm 0.2^\circ$). Temperature is read and stored with each intensity measurement. Data collection possible at various pre-chosen temperatures by using BATCH. (4)
 MULREF: avoids multiple reflections by analyzing an azimuthal range for each reflection
 OTFLOT: omega/theta plot of a reflection. Facilitates the choice of scan parameters (2,3)
 Q-MODE: data collection on non commensurate structures, using a fourth, broken, index.
 SET4: setting angles derived from four settings of each listed reflection, leading to cell dimensions freed largely from mis-alignment errors.
 1) A.J.M. Duisenberg, Acta Cryst. (1983).A39, 211-216
 2) CAD4 Manual, ENRAF-NONIUS, Delft (1977). ChXIII, p30
 3) A. McL. Mathieson, Acta Cryst. (1982).A38, 378-387
 4) J.L. de Boer, K.D. Bronsema and G.A. Wiegers. This conference.

16.2-9 CELL PARAMETER REFINEMENTS OF SMALL CRYSTALS USING A SINGLE CRYSTAL SPECTROMETER. By V.I. Lisoivan, S.A. Gromilov, Institute of Inorganic Chemistry, Siberian Branch of the Academy of Sciences of the USSR.

The methods of a samples selection and initial cell parameter determination of single crystals with unknown structures have already been described (G.B. Bokij, M.A. Porai-Koshits, X-ray Anal. vol.1 (1964)). These parameters may be refined using the single crystal spectrometer (V.I. Lisoivan, R.R. Dikovskaia, Prib. Tekn. Eksp. (1969) 4, 162). The sample must be so mounted that its axis would pass along the spectrometer axis. Two linear and one angular parameter may be refined when the crystallographic axis directions of the sample are determined. Otherwise, it is necessary to find some reflections, to plot a reciprocal lattice net and to choose basal planes. The polar coordinates for the origin point in plotting the reciprocal lattice are $d^* = 0$, $\rho = 1/d$. For the next points d^* angle is determined by: $2(180 - d^*) = \varphi_{n1} + \varphi_{n2} - \varphi_{o1} - \varphi_{o2}$, where φ_{n1} and φ_{n2} are the angular positions of n plane for reflections on both sides of zero, and φ_{o1} , φ_{o2} are those of the origin point. The remaining lattice parameters are refined, when the crystal axis is set in the equatorial plane of the spectrometer. The reflection angle measurements with the 5" accuracy can be carried out by the method described by R.L. Barns (Adv. X-ray Anal. (1972) 15, 330). The method is illustrated by the example of the inner complex compound bis-[4-(2-oxistyryl)methyl-2,2,5,5-tetramethyl-3-imidazolin-1-oxil] cobalt(II): $a=12,8943(8)\text{\AA}$, $b=19,336(1)\text{\AA}$, $c=12,8236(7)\text{\AA}$, $\beta=110,458(6)^\circ$ (without refraction correction).

16.2-10 A NEW TWO-CRYSTAL MONOCHROMATOR FOR SYNCHROTRON X-RAYS WITH FAST AND PRECISE SCAN IN THE RANGE $50^\circ \leq \theta_{\text{BRAGG}} \leq 85^\circ$. By U. Bonse, Th. Risse, R. Nußhardt, U. Dretzler, G. Ernst, S. Krasnicki, Universität Dortmund, Institut für Physik, Postfach 500500, D-4600 Dortmund 50, Fed. Rep. of Germany.

For making best use of the unique properties of synchrotron X-rays in crystallography it is essential to have at hand a monochromator system fulfilling the following requirements:
 1. High energy resolution $E/\Delta E$ at good transmission, with the option to decrease $E/\Delta E$ at the benefit of transmission. 2. Wide tuning range of typically $5\text{keV} \leq E \leq 100\text{keV}$. 3. Possibility of either reject or choose a single or a given set of harmonics. 4. Mechanical/thermal stability. 5. On-line computer operation with flexibility to change mode of operation according to varying user requirements. 6. Fixed position of incident and monochromatized beam.

We have built a corresponding device in which two crystals C_1 and C_2 are translated in opposite direction by a pair of coupled lead screws combined with precision sleds. Throughout the motion parallelism of C_1 and C_2 is maintained by means of a pair of parallel guides operating at right angles with respect to each other similar to the device of Golovchenko, Leversque and Cowan (Rev. Sci. Instr. 52, 509 (1981)). The accessible range for the Bragg angle θ_B is $50^\circ \leq \theta_B \leq 85^\circ$. The resolution for the translation is $0.5 \mu\text{m}$ per step and encoder unit. This ensures even in the low Bragg angle range an energy increment $\Delta E \leq 0.1\text{eV}$. Fine alignment of the crystals with respect to parallelism is achieved by combined stepping motor piezo-electric drives at crystals C_1 and C_2 . This fine control also allows the rejection/selection of harmonics by means of suitable misalignment with respect to exact parallel orientation.

The instrument is operable through a micro-computer. First results obtained at DORIS/HASYLAB will be presented.

Fig. 1: Beam paths for three different Bragg angles θ_B

$$\begin{aligned} C_1 C_2: \theta_B &= 5^\circ \\ C'_1 C'_2: \theta_B &= 45^\circ \\ C''_1 C''_2: \theta_B &= 85^\circ \end{aligned}$$

