C R Y S O M

An Interactive Program for Operating

Rich.Seifert

Single-Crystal Four-Circle Diffractometers

Version: 2.80

March, 1996

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INTRODUCTION

The program CRYSOM assumes the existence of a full-circle Eulerian cradle, but it can also be used with non-full-circle ones. Since the principles of such four-circles goniometers are well known and have been described in detail elsewhere[1], we shall discuss them here briefly.

The goniometer allows four independent rotations: three for the crystal (ω, χ, φ) and one for the radiation detector (2θ). The full circle of the goniometer can rotate about a vertical axis passing through the goniometer center (ω rotation). The goniometer head can rotate inside of the full circle (χ rotation) and around itself (around the principal axis of the goniometer head, φ rotation). By rotating the ω, χ and φ axes, any reciprocal point of the crystal can be brought onto the surface of Ewald sphere at the equatorial plane of the goniometer. The detector can rotate about an axis that coincides with the ω axis (2θ rotation) to receive the Bragg reflexion.

The crystal setting angles (ω, χ, φ) are normally calculated in the so called bisecting geometry (the plane of the χ circle bisects the angle between the incident and the diffracted beams). In reciprocal space each lattice plane is represented by a point at a distance λ/d from the origin of the reciprocal space in a direction perpendicular to that plane, determining the so called reciprocal-lattice vector $\boldsymbol{d}^*$. The reciprocal points representing all possible lattice planes form a three-dimensional lattice, called reciprocal lattice. In this way, for each reciprocal point there is a reciprocal-lattice vector of the type:

$$\boldsymbol{d}^* = h\boldsymbol{a}^* + k\boldsymbol{b}^* + l\boldsymbol{c}^*$$

where h,k,l are integers, known as the Miller indices of the lattice plane, and $\boldsymbol{a}^*$, $\boldsymbol{b}^*$, $\boldsymbol{c}^*$ are the base vectors of the reciprocal lattice, related to the base vectors of the real

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[1] *Arndt, U.W. and Willis, B.T.M.*, Single crystal diffractometry, University Press, Cambridge 1966.

lattice by:

$$a.a^* = b.b^* = c.c^* = \lambda$$
$$a.b^* = b.c^* = c.a^* = a^*.b = b^*.c = c^*.a = 0$$

To describe the orientation of the crystal in the goniometer, we use an orthogonal coordinate system attached to the goniometer. If all angles are zero, the x-axis points, in the equatorial plane, in the same sense as the incident beam, the z-axis is vertical (pointing upwards) and the y-axis completes a right-handed system. Using this coordinate system, the three base vectors of the reciprocal lattice can be written in terms of their components along these axes, and so we can build a matrix *OM* which is known as the orientation matrix:

$$OM = \begin{pmatrix} a_x^* & b_x^* & c_x^* \\ a_y^* & b_y^* & c_y^* \\ a_z^* & b_z^* & c_z^* \end{pmatrix}$$

The orientation matrix can easily be used to calculate the coordinates of any reciprocal point (the components of any reciprocal vector d^*):

$$\begin{pmatrix} x \\ y \\ z \end{pmatrix} = OM . \begin{pmatrix} h \\ k \\ l \end{pmatrix}$$

The x, y, z coordinates can be related to the goniometer angles for the bisecting setting through the following equations:

$$d^* = \sqrt{x^2 + y^2 + z^2} = 2.\sin\theta$$
$$x = d^*.\cos\chi.\sin\phi$$
$$y = d^*.\cos\chi.\cos\phi$$
$$z = -d^*.\sin\chi$$

From these relations we can find χ and φ, but we choose the solution where $-90 \le \chi \le 90$.

At this point we shall briefly describe the procedure to be carried out before the diffracted intensities can be measured. The goniometer head (with the crystal mounted on it) is mounted on the goniometer. The crystal is then centered by hand, using the translations of the goniometer head, to bring it into the goniometer center, i.e. it is moved into a position such that when any axis (χ and φ) is rotated no further crystal displacement is observed. With such an arbitrary orientation of the crystal in the goniometer center we can then operate to search for several reflexions (normally 25) occurring in a small angular range. The observed angles where these reflexions occur can then be converted into points of the reciprocal space and thus we can look for the three shortest vectors (not lying in a plane) which will define the base vectors of the reciprocal lattice. Once the reciprocal lattice is known and the scan conditions have been selected, the goniometer can then automatically be positioned for collecting the intensity of every reciprocal point.

Two special features of CRYSOM are represented by the possibilities of:

- Using two manual slits instead of two automatic ones (normal case). This option is defined in the so called hardware file.

- Doing surface diffraction. This option can be switched on or off using just a software command. See also the Appendix.

- Assuming non-integer hkl reciprocal points for quasi-crystals cases (normally from the so called File 3).

THE PROGRAM

The CRYSOM program is written in a full interactive mode. For this purpose, once the program is loaded into memory, one can use several commands which carry out the work to be done, covering from the selection of general conditions, the peak-hunting procedure (to obtain the orientation matrix) up to the data collection process (measurement of the diffracted intensities). CRYSOM shows the "Ready..." status meaning that it can accept any external operator command.

The existing commands are listed below and are presented in groups according to their purpose. There is also a HELP command which informs about the meaning of the other ones.

```
HELP

FILES   DUMP1  PRON    PROFF   WAVE    POWER   SIGMA   ATTEN   LLIM
NREF    SURPAR QUASI   DATI    DATO    DATCOL  DATCON

ALIGN   LZER   MODZER

VIEW    SEARCH DETCEL  INDEX   REDCEL MODOM    TWIN    LATREF
PKLIS   PKINC  PKSKP   PKRES   PKREM
LOM     LDCEL  LRCEL   LRMT

CENFAC CENMOD CENPK   CENTAB

GOANG   GOHKL  HSLIT   VSLIT   SHUTO   SHUTC  XCOM    SCAN    PLOT
LANG

EXIT

Input-EOF: Press  CTRL  Z   and  press Return
Interrupt: Press  CTRL  A   and  wait
```

Some of these commands need additional operator input, which is automatically asked when it is needed. The input expected from the operator is presented in terms of clear questions, normally accompanied with default values for the answers, shown between brackets. The meaning of these individual commands is explained somewhere in this manual.

GETTING STARTED

The program is loaded into memory by typing CRYSOM from the MS-DOS prompt and normally on the XDATA directory. At the moment of loading it asks for a job file having an extension of type X1A. Just type again CRYSOM and press the Return key. This file (CRYSOM.X1A) is just a void file which is used as an interface to the general Rich.Seifert way of programming.

The following paragraphs explain some operations to be done by the operator at program start.

USING THE ATTACHED PRINTER

CRYSOM produces an amount of information as the result of every command being introduced. This information can also be directed to an attached printer. This option can be activated at program start, answering "yes" to the corresponding question, or at any time when the program shows the "Ready..." status, using the command PRON (PRinting ON). The command PROFF (PRinting OFF) switches off this output option.

FILES ASSIGNED TO THE SESSION

Every time that CRYSOM is loaded into memory, three files are prepared. The first one (File 1), a binary file, will contain the information related to the actual experiment: tube parameters (wavelength, power), general conditions for peak recognition and centering, setting angles and indices of the reflexions used to calculate the orientation matrix, cell parameters and conditions to be used to collect the intensities. The second file (File 2) is an ASCII one prepared to contain the output of the data collection process (hkl indices, intensities and other measurement data). The third file (File 3), also ASCII, is dedicated to contain a list of

input *hkl* indices, only to be used if the reflexions to be collected are not to be automatically generated by the program; this file can also contain non-integer *hkl* indices for quasi-crystals, in which case the QUASI instruction should previously be used to select this feature. The names and extensions of these three files can be selected by the operator and normally will be selected to remember its contents. Thus, if the file names are selected as the compound identification (for instance TEST), File 1 can be selected as TEST.TAB to remember that it is dedicated to contain the TABle of parameters to be used in the experiment, File 2 can be selected as TEST.DAT to remember that it will contain the output of the DATa collection process and File 3 can be chosen as TEST.HKL (it may contain the *hkl* indices of the reflexions to be collected). In this way, if one has to interrupt a working session and restart it again, all the information of the old session will be automatically loaded if the appropiated files are selected at the program start. The files assigned to the session can be changed at any time just using the command FILES.

CHECKING SOME GENERAL CONDITIONS

Before starting the work, the value of some general parameters should be checked.

If the wavelength shown does not correspond to the actual X-ray tube, use the WAVE command to update it.

Check that the tube parameters listed (kV and mA) are appropiated for your tube and, in case, correct them by using the POWER command.

Check that the "Peak centering factor" listed is 1.00 degrees (this is an appropiated value for almost any crystal) and, if necessary, correct it with the CENFAC (Centering FACtor) command.

The peak centering mode should normally be 1; change it, if necessary, using CENMOD.

Check also that the "Sigma criterion" to recognize peaks shows a value of 15.00 (this is also a reasonable value to start with) and, if necessary, use the SIGMA command to update it.

The output line showing the Plotting conditions should contain the following values: Plot: YES, Hardcopy: NO, Max. cps: 10000 (or a

value not very high); use the PLOT command if necessary to correct these values.

The information listed for the attenuator filter should be the correct one (normally: "Attenuator 1 to be set if the intensity is greater than 30000 cps"); use the ATTEN command if necessary.

If the working version of the program is not using two variable slits, two other parameters should be checked at this time. These are the values of the slits (HSLIT and VSLIT) being used and to be changed manually. The program, at start, assumes two values for the slits which are printed out. If these values do not correspond to the actual values of the slits, the operator should react accordingly, that is, changing the slits manually to the values listed or, on the contrary, giving to the program the slit values that are actually set (commands HSLIT and VSLIT).

The switch which selects the conditions for doing surface diffraction is also printed out. Check it and react accordingly using the general command SURPAR (SURface diffraction PARameters).

Finally, some message related to quasi-crystals can also show that this feature has been selected. If you do not agree with it, just use the QUASI instruction to change it. Thus, at this time the output generated by the program will be of the type shown below:

```
Printing is ON
----------------------------------------------------------------
Program: CRYSOM  Version: 2.80
Rich.Seifert u. Co., Ahrensburg   (Germany)
Crystallography Dept., CSIC, Madrid (Spain)
----------------------------------------------------------------
Zero corrections set in \xdal\software.x1a file ...
    Z-2theta    Z-omega     Z-chi     Z-vertical
     -.0150      .0136      .6261         .0598
----------------------------------------------------------------
Setting up the files for the session

File 1 is TEST.TAB
File 2 is TEST.DAT
File 3 is TEST.HKL
Wavelength is  1.5418   (Ka1=  1.54060   Ka2=  1.54553)
Power (tube parameters):  40 kV      20 mA
              Standby :   5 kV       5 mA
Peak centering factor    1.00
Peak centering mode      1
Sigma criterion   15.00
Plot: YES    Hardcopy: NO    Max. cps:  10000.
Attenuator 1 to be set if intensity .gt. 30000. cps
The system is using variable slits
No surface diffraction wanted
Non-quasi-crystal assumed
```

CRYSTAL MOUNTING AND CENTERING

Before the crystal can be analyzed with the X-ray beam, the crystal must be properly mounted and centered on the goniometer. For this purpose the command VIEW sets the appropriate angular values to observe the crystal through the attached microscope (the goniometer may be operated with a mounted microscope if it is located at its rear position, preventing crashes with the χ circle) and shows a menu which can be used to the following steps. The crystal, mounted on the goniometer head is to be centered by hand, using the translations of the goniometer head, to bring it into the goniometer center, i.e. it is moved into a position such that when any axis (χ and φ) is rotated no further crystal displacement is observed.

Normally, this operation is done first by rotating the φ angle at 0 and 90 degrees and correcting, at both positions, the crystal translations perpendicular to the viewing direction and lying on the horizontal plane. Then, the crystal height (translation of the crystal up and down the goniometer head) should be corrected, setting previously the χ angle at 90 and -90 degrees. After this, the χ angle should be driven to the 0 position and the φ rotation should be used to correct carefully again the crystal translations lying on the horizontal plane (at φ = 0, 90, 180, 270 degrees). When these operations are done, just press the END key, put the microscope at the rear position and answer the asked questions. The program will show the "Ready..." status.

AUTOMATIC DETERMINATION OF THE CRYSTAL ORIENTATION

Before an automatic data collection process can be started, it is necessary to know the lattice constants and the orientation of the crystal (orientation matrix, OM). CRYSOM can determine this information from the unknown crystal in any arbitrary orientation. The program performs the necessary operations within a rather short time, so that this procedure should be used even if the lattice constants are known.

The first operation to be done is to use the SEARCH command to find out several reflexions (up to 25) within a preselected angular range. The region to be searched is determined by giving an angular range for θ, χ and φ. The region is searched along arcs of constant θ and χ, with alternately increasing and decreasing values of φ. The magnitudes of the steps in θ and χ depend on the detector apertures, which are automatically (or manually) used. Normally the default values shown for the parameters to be used are good enough to begin with an unknown crystal. This searching process stops when the complete angular range has been investigated, or when a preset number of peaks has been found, or when the operator interrupts the process by pressing CTRL A on the keyboard.

The output of the searching process gives information like the following one:

> SEARCH

Conditions for searching peaks:

```
Number of peaks to be found  25
Theta angle between   10.00 and   40.00
Constant defining steps in Theta   1.00
Chi   angle between  -60.00 and   60.00
Constant defining steps in Chi     1.00
Phi   angle between     .00 and  120.00
Step width for Phi     1.00
```

Peak	Theta	Chi	Phi	Ipeak	Fwhm
1	9.2947	-50.9447	16.2742	32285	.24
2	9.6910	-49.8377	57.1063	16075	.22
NC	9.4062	-49.2315	89.5433		
= 2 R	9.6898	-49.8187	57.1364	15678	.22
3	10.8902	-44.2111	9.6123	10981	.19
= 3	10.8978	-44.1943	9.6079	11133	.18
			...		
			...		
25	12.6523	14.3223	103.5906	8393	.23

The *Peak* column contains the peak number and some other additional information. NC means "no centering" and is displayed when for any reason the centering process at the shown angles was not possible. The = sign means that the corresponding angles of the actual peak are identified as very similar to the angles of a previous peak in the table having the same peak number. If no other information is shown, the "equal" (=) peak is added to the table, meaning that it has an intensity greater than the previous peak with the same peak number. If, on the contrary, the = sign is accompanied by a "R" (rejected) code, it means that the actual peak is rejected because it has lower intensity than a previous peak with the same peak number. The columns headed by *Theta, Chi* and *Phi* show the optimized angular values where the reflexions have been centered. *Ipeak* represents the peak intensities, in counts per second, measured on the top of the reflexions with a rectangular slits window of 1x1 degrees (or with the fixed slits being actually used). Finally, *Fwhm* shows the so called "full width at half maximum" of the peaks.

Once the SEARCH command has finished, one can use the angular information contained in the peak table to build a reciprocal lattice and therefore calculate the cell constants. For this purpose, one can use the command DETCEL (DETermine CELL). Some little additional information required (maximum cell length to be accepted and the maximum angle to consider two reciprocal vectors as parallel) is expected, although the default values are appropriate for almost any crystal. The first step of DETCEL is to convert the angular values, contained in the peak table, into orthogonal coordinates (*x, y, z*) of the reciprocal points according to the expressions given above. Each pair of points

forms a vector. DETCEL finds the shortest vector (=**a***), then the shortest one not parallel to **a*** (=**b***), and finally the shortest vector not in the plane of **a*** and **b*** (=**c***). The remaining vectors which are found parallel to **a***, **b*** or **c***, with equal moduli to them (or multiple moduli), are also averaged to a better determination of the base vectors. These three vectors describe always a primitive cell and determine the preliminary orientation matrix OM as described above. The OM matrix is printed out together with the so called Reciprocal Metric Tensor (RMT) defined as the product of OM$_t$.OM (where OM$_t$ = OM transposed), the reciprocal cell constants, the direct cell constants and the cell volume (shown as a negative value when the chosen cell vectors form a left-handed system of axes).

The lattice constants are calculated as follows:

$$a = \lambda\sqrt{(RMT^{-1})_{11}} \quad \cos\alpha = \frac{\lambda^2(RMT^{-1})_{23}}{bc}$$

$$b = \lambda\sqrt{(RMT^{-1})_{22}} \quad \cos\beta = \frac{\lambda^2(RMT^{-1})_{13}}{ac}$$

$$c = \lambda\sqrt{(RMT^{-1})_{33}} \quad \cos\gamma = \frac{\lambda^2(RMT^{-1})_{12}}{ab}$$

$$RMT^{-1} = OM^{-1} \cdot OM_t^{-1} \quad OM^{-1} = \begin{pmatrix} a_x & a_y & a_z \\ b_x & b_y & b_z \\ c_x & c_y & c_z \end{pmatrix}$$

The DETCEL process continues and, after a least squares indexing of the peaks, a more accurate OM matrix (and therefore more accurate cell parameters) is obtained. The Miller indices *hkl* of the reflexions are calculated as:

$$\begin{pmatrix} h \\ k \\ l \end{pmatrix} = OM^{-1} \begin{pmatrix} x \\ y \\ z \end{pmatrix}$$

If the crystal is not twinned or cracked and OM is sufficiently accurate, the calculated *hkl* indices should be integers. A deviation of ± 0.125 is accepted and the indices are rounded to

integers; otherwise they are printed as real numbers. New OM and RMT matrices are calculated using least-squares refinement with the reflexions having integer indices, and more accurate cell dimensions are printed. If not all indices are integers, once DETCEL is completed ("Ready..." status), the operator may reject the peaks showing fractional *hkl* indices by using the PKSKP (PeaK SKiP) or PKREM (PeaK REMove) commands and call DETCEL again. Finally, DETCEL proposes a cell reduction based on the Delaunay procedure. The cell reduction procedure can also be used as an individual command (REDCEL).

An example of DETCEL output is shown below:

```
> DETCEL

Determination of the orientation matrix

Maximum cell length to be accepted          : 1000. A
Maximum angle to accept vectors as parallel:    5. deg

Peak        x        y        z       Ipeak
  1      .0570    .1954    .2508     32285
  2      .1823    .1179    .2573     16075
  3      .0452    .2673    .2636     11133
                  ...
                  ...
 25      .4126   -.0997   -.1084      8393

Number of contributing peaks    25

Shortest vector defining a* between peaks    1    3
Contributing vector of length  2 times       1   17
Contributing vector of length  1 times      13   14
                ...
                ...
Shortest vector defining b* between peaks    6    9
Contributing vector of length  3 times       0    5
Contributing vector of length  2 times       1   15
                ...
                ...
Shortest vector defining c* between peaks    3    4
Contributing vector of length  2 times       1    8
Contributing vector of length  2 times       2    7
                ...
                ...
Peak number    11  not used
Peak number    12  not used
```

```
Orientation matrix OM  (as lambda times a* b* c* column vectors)

        .011306      .118890     -.038353
       -.076102      .003576     -.044083
       -.017129      .029978      .158550


Reciprocal metric tensor RMT (as OMt*OM  with OMt= OM transposed)

        .006213      .000559      .000205
        .000559      .015046      .000036
        .000205      .000036      .028552


Reciprocal cell constants and angle cosines

         .0511         .0796        .1096
       89.9018       89.1167      86.6881
        .001714       .015415      .057771


Direct cell constants, angle cosines and volume

       19.5957       12.5904       9.1255
       90.0473       90.8791      93.3108
       -.000825      -.015342     -.057752
     2247.4000


Indexing and least-squares refinement

Peak    h    k    l     Theta        Chi        Phi      Ipeak     Fwhm
  1    -3    1    1     9.2947    -50.9447    16.2742     32285      .24
  2    -2    2    1     9.6910    -49.8377    57.1063     16075      .22
  3    -4    1    1    10.8902    -44.2111     9.6123     10981      .19
                              ...
                              ...
 25     2    3   -1    12.6523     14.3223   103.5906      8393      .23


Orientation matrix OM  (as lambda times a* b* c* column vectors)

        .008670      .119392     -.038375
       -.077579      .003459     -.044275
       -.019475      .029970      .158813


Reciprocal metric tensor RMT (as OMt*OM  with OMt= OM transposed)

        .006473      .000183      .000009
        .000183      .015165      .000025
        .000009      .000025      .028655


Reciprocal cell constants and angle cosines

         .0522         .0799        .1098
       89.9318       89.9612      88.9408
        .001190       .000677      .018485
```

Direct cell constants, angle cosines and volume

```
     19.1669     12.5224      9.1082
     90.0675     90.0376     91.0591
    -.001177    -.000656    -.018484
   2185.7210
```

Cell reduction by Delaunay method

Direct cell constants (Latt.type P)   Niggli-matrix

```
     19.1669     12.5224      9.1082     367.3687  156.8096  82.9514
     90.0675     90.0376     91.0591       -.1343    -.1145  -4.4312
```

Proposal   1   Delaunay case 22   Lattice: Monoclinic   P

```
     12.5224      9.1082     19.1669    Transf.-matrix
     90.0376     91.0591     90.0675    .00    1.00     .00
                                        .00     .00    1.00
                                       1.00     .00     .00
```

MODIFYING THE CRYSTAL ORIENTATION

It can sometimes occur that the crystal used is not a single one, so that DETCEL does not produce an acceptable result, i.e. many indices are non-integers, or even no OM matrix is calculated. This also produces that the list of vectors contributing to the determination of the basic ones ($\mathbf{a}^*$, $\mathbf{b}^*$, $\mathbf{c}^*$) is very short (no superdetermination occurs). In this case, the operator has a lot of possibilities to play with. One can observe that, for instance, one of the reciprocal axes is determined by one pair of reciprocal points only (one vector). In this case, one can skip (PKSKP) or remove (PKREM) one of the reciprocal points (peaks) contributing to that vector and restart DETCEL again, until reasonable results are obtained. For specially important cases, where no other crystal is available, the command TWIN can be very helpful to detect the individual peaks of a two-crystals twin.

In the normal cases, when the crystal is single, DETCEL will produce a proper OM matrix. However, if the primitive cell calculated automatically by DETCEL does not correspond to the maximum crystal symmetry or to the standard crystal orientation, what normally is shown by the cell reduction results, one can transform the crystal orientation (the OM matrix) to get the correct one, using the transformation matrix shown at the right side of the corresponding output of the cell reduction proposal (see above). This operation can be performed using the MODOM command (MODify OM), which can also be used to set new values of the OM matrix (not to transform OM). This is shown below, as an example, transforming the crystal orientation given in the output of DETCEL (see above). Once the OM matrix is modified, a least-square indexing procedure is started automatically, giving rise to a new OM matrix, cell constants, etc.

```
> MODOM

Previous OM matrix was transformed by:

         .000000   1.000000    .000000
         .000000    .000000   1.000000
        1.000000    .000000    .000000

Orientation matrix OM  (as lambda times a* b* c* column vectors)

         .119392   -.038375    .008670
         .003459   -.044275   -.077579
         .029970    .158813   -.019475

Reciprocal metric tensor RMT (as OMt*OM  with OMt= OM transposed)

         .015165    .000025    .000183
         .000025    .028655    .000009
         .000183    .000009    .006473

Reciprocal cell constants and angle cosines

          .0799      .1098      .0522
        89.9612    88.9408    89.9318
        .000677    .018485    .001190

Direct cell constants, angle cosines and volume

        12.5224     9.1082     19.1669
        90.0376    91.0591     90.0675
       -.000656   -.018484    -.001177
       2185.7210
```

Indexing and least-squares refinement

Peak	h	k	l	Theta	Chi	Phi	Ipeak	Fwhm
1	1	1	-3	9.2947	-50.9447	16.2742	32285	.24
2	2	1	-2	9.6910	-49.8377	57.1063	16075	.22
3	1	1	-4	10.8902	-44.2111	9.6123	10981	.19
				...				
				...				
25	3	-1	2	12.6523	14.3223	103.5906	8393	.23

Orientation matrix OM (as lambda times a* b* c* column vectors)

```
         .119389   -.038383    .008641
         .003460   -.044272   -.077568
         .029969    .158803   -.019484
```

Reciprocal metric tensor RMT (as OMt*OM with OMt= OM transposed)

```
         .015164    .000024    .000179
         .000024    .028652    .000008
         .000179    .000008    .006471
```

```
Reciprocal cell constants and angle cosines

       .0799        .1098        .0522
      89.9654      88.9626      89.9353
       .000605      .018107      .001130

Direct cell constants, angle cosines and volume

      12.5226       9.1086       19.1695
      90.0335      91.0375      90.0641
      -.000584     -.018106     -.001119
    2186.1810
------------------------------------------------------------
```

GETTING A MORE ACCURATE CRYSTAL ORIENTATION

The accuracy obtained for the crystal orientation, and therefore for the lattice constants in the examples given above, is a consequence of a unique process of peak centering, the one carried out at SEARCH time. This means that we can still spend a little more time to improve the setting angles of the peaks contained in the table and used to calculate the OM matrix. Moreover, one can make some modifications in the table of peaks, removing (PKREM) some of them (probably because several ones have low indices, or they are to weak in intensity, or systematically one of the indices is very low, or too high, etc.). If this is so, one can include new peaks in the table by using the PKINC command (PeaK INClude), using several input modes (indices, symmetry operators, angles, coordinates). In both cases (no modifications done in the table of peaks, or new reflexion settings introduced, and therefore loaded with zero intensities) one should give a new oportunity to the goniometer to get a better set of angles for the table peaks. This is done using the CENTAB (CENter TABle) command, which automatically recenters every peak of the table and produces a new least-squares refinement of both OM matrix and lattice constants, as shown below:

```
> CENTAB
```

Peak	Theta	Chi	Phi	Ipeak	Fwhm
1	9.1306	−51.0844	16.0073	100838	.23
2	9.6952	−49.8707	57.1350	16065	.22
3	11.0022	−44.3128	9.7773	26600	.21
		...			
		...			
25	12.6470	14.3264	103.5731	8439	.23

Indexing and least-squares refinement

Peak	h	k	l	Theta	Chi	Phi	Ipeak	Fwhm
1	1	1	−3	9.1306	−51.0844	16.0073	100838	.23
2	2	1	−2	9.6952	−49.8707	57.1350	16065	.22
3	1	1	−4	11.0022	−44.3128	9.7773	26600	.21
				...				
				...				
25	3	−1	2	12.6470	14.3264	103.5731	8439	.23

```
Orientation matrix OM  (as lambda times a* b* c* column vectors)

        .118859   -.038467    .008473
        .003653   -.044261   -.077431
        .029853    .158608   -.019559

Reciprocal metric tensor RMT (as OMt*OM  with OMt= OM transposed)

        .015032    .000001    .000140
        .000001    .028595   -.000001
        .000140   -.000001    .006450

Reciprocal cell constants and angle cosines

         .0795      .1097      .0521
       90.0043    88.1835    89.9973
      -.000075    .014250    .000048

Direct cell constants, angle cosines and volume

       12.5766     9.1176     19.1998
       89.9957    90.8165     90.0028
        .000076   -.014250   -.000049
      2201.3970
------------------------------------------------------------
```

It is obvious that after the CENTAB procedure, the accuracy of the monoclinic cell, shown as an example, is improved.

ACCURATE DETERMINATION OF BRAGG ANGLES FOR LATTICE REFINEMENT

The procedure described above (CENTAB) is based on a recentering of the setting angles of the reflexions of the table to improve the crystal orientation, that is, to obtain an OM matrix which will predict satisfactorily the setting angles of the remaining reflexions. Therefore, the least squares refinement included in that process is always done considering a triclinic cell, that is with no symmetry restrictions on the lattice parameters.

However, in order to obtain the most accurate lattice constants, a least squares refinement including symmetry constrictions is normally used, minimizing some function including the Bragg angles of the reflexions. Just for this purpose, CRYSOM includes a special routine called LATREF which produces very accurate Bragg angles of the reflexions lying along several lines of the reciprocal space. Moreover, LATREF optimizes the reflexion angles at both positive and negative 2θ values, producing a list of indices and Bragg angles corrected for any possible 2θ zero shift. The input expected by LATREF is up to 25 lattice rows (given as default) to be scanned, so that every reflexion lying on these rows is centered. Optionally an output file is written containing a list of *hkl* indices and Bragg angles (see the example given below):

```
> LATREF
Theta angles determination for lattice refinement
  25 lattice rows to be scanned
              1  0  0      0  1  0     0  0  1
              1  1  0      1  0  1     0  1  1
             -1  1  0      1 -1  0    -1  0  1
              1  0 -1      0 -1  1     0  1 -1
              2  1  0      1  2  0     1  0  2
              1  1  1     -1  1  1     1 -1  1
              2  2  1      1  2  2     1 -2  2
             -2  2  1      1 -2 -2     2  1  2
             -2  1  2
        in the Theta range:  2.00  and  35.00
Output data to be written on file: LATREF.DAT

     h  k  l  Theta1   Theta2  <Theta>   <Chi>  <Phi> <Fwhm> <Ipeak>
     1  0  0  3.4971  -3.5091   3.5031 10.4456 11.2346 .21    6628
     2  0  0  7.0285  -7.0301   7.0293 10.4544 11.2358 .21    1746
     3  0  0 10.6065 -10.6105  10.6085 10.4540 11.2350 .20    1251
     4  0  0 14.1949 -14.1999  14.1974 10.4600 11.2308 .23   25450
     5  0  0 17.8545 -17.8601  17.8573 10.4598 11.2299 .21    1047
     6  0  0 21.5890 -21.5902  21.5896 10.4633 11.2305 .23    2919
NC   7  0  0
NC   8  0  0
NC   9  0  0

NC   0  1  0
     0  2  0  9.7340  -9.7388   9.7364 39.7472 66.2740 .24    6086
NC   0  3  0
     0  4  0 19.7819 -19.7902  19.7861 39.7534 66.2799 .24    2434
NC   0  5  0
                          ...
                          ...
HKL and THETA data are written on file: LATREF.DAT
-----------------------------------------------------------------
```

The output of LATREF can also be very useful to detect the existence of possible systematic absences in terms of the NC codes of the non-centered reflexions (a 2_1 axis in **b** direction in the example).

CHOOSING THE CONDITIONS FOR THE DATA COLLECTION

The data collection is the process carried out automatically to measure the intensities of all points of the reciprocal lattice which fulfill some conditions chosen by the operator (angular range of the Bragg angle and limits of the Miller indices). The results of these measurements will be saved on an output file (File 2).

Before a start can be done on this procedure, the values for some implied parameters must be selected. A print out of all these parameters can be obtained using the command DATO (DATa Output). See the example shown below. The general command DATI (DATa Input) allows the use of several subcommands (also including a HELP subcommand) to set the values for these parameters.

```
> DATO

TEXT = Test crystal. Small data collection ...

ZONE = 2Theta range:       2.00    10.00
        Limits for H:     -1        1
        Limits for K:      0        0
        Limits for L:      0        2
        Start    HKL:     -1        0        0
        Steps in HKL:      1        1        1

MODE = Scan mode : C
        Scan axis : M
        Scan width:   3.00 + .00 * tan(theta)
        Scan speed:    .50 (deg./s)

SLITS =  2.00 +  .00 * tan(theta)    for Hslit
         2.00 +  .00 * tan(theta)    for Vslit

MAXN  = Maximum number of scans:    1

RELUNC= Range for relative uncertainty:  5   100%

SYSABS= Conditions for systematic absences:
        H=0    L=0    KN2
        If these conditions are fulfilled, reflexions are skipped

SYMREF=  1 symmetry related reflexions to be measured
           -1  0  0    0 -1  0    0  0 -1
```

```
OUTMOD= N

REFER =  2 reference reflexions selected
           to be measured every  3 reflexions
           max. centroid variation   .0500 deg.
           max. intensity variation     50 %
              4    0    0      -4    0    0
```

The following parameters can only be modified using
the general command SURPAR ...
No surface diffraction wanted

The following condition can only be modified using
the general command QUASI ...
Non-quasi-crystal assumed

The subcommand TEXT is used to input any alfanumeric information
(crystal identification, etc.) to be included on the output file.
ZONE is a subcommand[2] which allows the selection of the range in
the Bragg angle limiting the reciprocal space containing the
reflexions to be measured, as well as other parameters related
with the zone of reciprocal space to be explored. The following
parameters are expected: maximum and minimum Bragg angle, lower
and upper limits for the indices, start values and increments to
be used for generating the remaining indices. Alternatively, the
individual *hkl* indices of the reflexions to be measured can be
given through a file (File 3) containing a free-format record for
each *hkl* triplet.

The MODE subcommand[3] is used to choose the scan mode, scan axis,
scan width and motor speed. If a $\omega/2\theta$ scan is wanted, the M
keyword for the scan axis should normally be selected, meaning
that for the upper angle reflexions a non-bisecting geometry will
be allowed. The scan width, which always refers to the 2θ width

---

[2] There is a useful general command (NREF) which can previously be
used to estimate the number of reflexions expected in an angular
range.

[3] There is a general SCAN command which can previously be used to
analyze the intensity profiles and therefore to select the most
appropiated conditions for the MODE subcommand (scan width, slits,
speed, etc.).

when 2θ is involved, is determined by two parameters in the form: *a + b tanθ*. Thus, making *b=0* a fixed scan width will be used. For a variable scan width (not normally needed) an estimated *b* parameter of 0.3 is good enough for Cu and Mo radiations.

The detector slits (fixed or variable) are selected by the subcommand SLITS. Two slits are used. The so called Hslit (Horizontal slit) is the slit which however can be opened and closed in the vertical sense. Therefore, the Vslit (Vertical slit) is the slit whose aperture can be variated in the horizontal sense. Both slits can be used as fixed (normal case) or with variable apertures (if available) during the data collection. Their apertures is determined by an expression similar to the one explained for the scan width. Two fixed slits of about 2 degrees give good results to collect intensities, but other values can be used if one previously uses the general SCAN command to select them for the sample being used.

The strategy of measurement is based on the following. Every reflexion is scanned with the selected conditions, the counts added to the previous scans and its relative uncertainty in percent, RU=100.σ(I)/I, is calculated. This value (RU) is then compared with two percentage limits (minimum and maximum RELative UNCertainty, see the RELUNC parameters). If RU is greater than the maximum RELUNC value, no additional scan is done, the reflexion is coded as very weak (flag=2) and the goniometer is positioned to measure a new reflexion. If RU lies between both limits, the scan is repeated until the RU value is less than the minimum RELUNC parameter (reflexion is coded as normal and gets a flag=0), or until a preselected maximum number of scans is reached (the reflexion is coded with flag=1 if RU is still between the limits). The maximum number of scans allowed is determined by the value selected with the MAXN subcommand.

The lattice points generated (reflexion indices) are skipped (or measured) if the indices fulfil (or not fulfil) the conditions selected by means of the SYSABS subcommand (SYStematic ABSences). This subcommand expects up to 6 lines of text containing keywords

which contain conditions to be checked. Each line may contain up to 3 keywords (separated by blanks) which will be considered as related each other with logical "AND" operators. Keywords given on separate lines will be related each other with logical "OR". The reflexion indices are checked against all the keywords given, so that the reflexion can alternatively be flagged according to the fact whether it fulfils, or not, all the keywords (combined with their respective logical operators). A total of 45 keywords are possible:

H=0	K=0	L=0	H=K	H=-K	K=L	H=L	K=-L
H=-L	H<0	K<0	L<0	H>K	H>L	K>L	K>H
L>H	L>K						

AH>AK AH>AL   AK>AL   AK>AH   AL>AH   AL>AK (^{4} see below)

HN2	HN3	HN4	KN2	KN3	KN4	LN2	LN3
LN4	LN6	HKN2	KLN2	LHN2	HKN4	KLN4	HLN4
2HLN4	HKLN2	-HKLN3	H-KLN3	HK-LN3		(^{5} see below)	

Thus, for example, selecting the following keywords given in 2 lines:

                   H=0 L=0 KN2
                   HLN2 K=0

will produce that reflexions of type:

                   h=0   and   l=0   and   k≠2n   or
                   h+l≠2n and   k=0
that is:
                   0k0 with k≠2n   or
                   h0l with h+l≠2n
will be skipped.

However, a new parameter (also considered in SYSABS) can decide just the contrary of the normal case, that is, if the reflexion

---

^{4} The letter A means 'absolute value of'. Thus, for example AH>AK means $|h| > |k|$.

^{5} The meaning of these conditions is better understood with some examples. In HKN2: HK means h+k, N means ≠, 2 means 2n; that is, h+k≠2n. In H-KLN3: H-KL means h-k+l, N means ≠, 3 means 3n; that is, h-k+l≠3n.

indices fulfil all the conditions, the reflexion will be measured. This can be useful just to ensure that the expected systematic absences are really absent.

The subcommand SYMREF is used to generate the indices of any symmetry related reflexions. Thus, after measuring a reflexion, its *hkl* indices can be used to generate (and therefore measure) up to 48 symmetry related reflexions. To generate the symmetry related indices, a symmetry operator matrix is used. Thus, for instance, to generate the indices *H=-h+k, K=k+l, L=-l* (not necessarily making any crystallographic sense), the corresponding operator will be the one shown below:

$$\begin{pmatrix} H \\ K \\ L \end{pmatrix} = \begin{pmatrix} -1 & 1 & 0 \\ 0 & 1 & 1 \\ 0 & 0 & -1 \end{pmatrix} \begin{pmatrix} h \\ k \\ l \end{pmatrix}$$

The output file written during the data collection process will contain the neto intensity and standard deviation of every measured reflexion. However, there is also the possibility to save some additional records for each reflexion, containing the intensity profile and/or the angles where the reflexion was positioned. This output alternative can be selected with the OUTMOD subcommand. Normally, the output mode N (neto) will be used.

The stability of the X-ray source, the crystal orientation and the possible crystal decomposition (intensity decay) can be controlled using several reference reflexions which are measured periodically. The subcommand REFER is used to select the indices of these references and the parameters implied in this control measurement. One of these parameters determines the maximum variation allowed for the centroid of the reference reflexions respect to the centroids measured at the start of data collection. If the variation is greater than the given parameter, the CENTAB command is automatically called, the references are remeasured and if their centroids still differ from the ones determined at the beginning, a warning message is produced and the new centroids are

taken as new reference values. Another parameter being input to REFER, determines the maximum variation allowed for the intensities of the references respect to the reference intensities at the start of data collection; if the variation is greater than the given value, the data collection is stopped.

Finally, the DATO output shows some other parameters like surface diffraction conditions or quasi-crystal assumed. However, these parameters cannot be modified with any DATI subcommand. For this purpose, the general commands SURPAR and/or QUASI must be used.

Once all parameters have been selected (using the described DATI subcommands), the SAVE subcommand must be used to save them and to return to the level of general commands, that is, to exit from the DATI level.

COLLECTING THE INTENSITY DATA

Once the operator has selected all the parameters to be used (DATI), the final step of the single-crystal work is just to collect automatically the intensity of every reflexion. There are two commands which start the data collection process: DATCOL and DATCON, the second one applying when an interrupted data collection is to be continued, appending the data to an old existing output file. Before DATCOL or DATCON is called, the operator can use the DATO command to print out all the parameters that configure the measurement process. An example of the output of the DATCOL or DATCON commands is shown below for a very short measurement.

```
> DATCOL
Plot: YES      Hardcopy: NO       Max. cps.: 10000.
Power (tube parameters):  40 kV    20 mA
                  Standby:   5 kV     5 mA
Standby values to be set at the end of the process

File 2 = TEST.DAT     has been previously erased

F=0, no attenuator filter
U=0, relative uncertainty .le. 5
U=1, relative uncertainty .gt. 5  and  .lt. 100
U=2, relative uncertainty .gt. 100
     (negative U value means negative Ineto)
A= abs(B1-B2)/sigma(B1-B2), B=background
     (this is an estimation of the background asymmetry)
N= number of maxima detected

    H   K   L    Ineto    Sigma  F   Fwhm  U  A  N  Psi   Record
R   4   0   0   254470.    496.  0    .88  0  1  1   .0      1
R  -4   0   0   253456.    510.  0    .88  0  8  1   .0      2
   -1   0   0    60870.    483.  0    .88  0  2  1   .0      3
    1   0   0    62430.    499.  0    .89  0  2  1   .0      4
   -1   0   1     1205.    543.  0  99.99  1  2  0   .0      5
    1   0  -1      998.    556.  0  99.99  1  2  0   .0      6
                           ...
                           ...
R   4   0   0   253256.    498.  0    .88  0  1  1   .0     13
R  -4   0   0   252965.    510.  0    .88  0  8  1   .0     14

Normal reflexions ...      6
Reference records ...      8
Total records     ...     14
------------------------------------------------------------------
```

The output produced by DATCOL or DATCON contains the following information. After the columns showing the *hkl* indices of the reflexions (including the R mark if it is a reference), the net intensity (*Ineto*) followed by its standard deviation (*Sigma*) are listed. The *F* code shows if the attenuator filter was used and is shown as a negative number when the intensity (in cps) is still greater than the maximum allowed for a linear answer of the detector (see ATTEN), even after a decrease made in the tube current, that is, when the reflexion was too strong to be measured properly. The *Fwhm* column shows the so called "full width at half maximum" of the reflexion profile. *U* is the code assigned according to the relative uncertainty of the measurement. The column headed by *A* is an estimation of the asymmetry of the background at both sides of the profile; an anomalous high value indicates that the scan width is to short, or that the crystal is not very well oriented. The value of *N*, normally 0 or 1, can be useful to detect possible crystal twinning (if N>1). *Psi* is the minimum value of the azimuth angle ψ used to collect the intensity, not equal to zero when the reflexion was positioned in a non-bisecant geometry to prevent goniometer collisions or shadowing at high Bragg angles and to fulfil the conditions (if any) selected for doing surface diffraction. Any reflexion within the expected limits being not accessible is printed with the flag NA (not accessible). Finally, the record number is shown on the last column.

DATCOL and DATCON also produce an output on File 2. The amount of information dumped on this file depends on the selection made previously with the OUTMOD subcommand (see also DATO and DATI). Three record types are possible on File 2 (see the Appendix).

DESCRIPTION OF COMMANDS AND SUBCOMMANDS

In addition to the main commands described above in terms of the
single steps that normally configure the operator work dedicated
to center a crystal, select the measurement parameters and collect
the intensity data, CRYSOM offers several other single commands
which will be briefly discussed here, together with the ones
presented before, so that some redundant information has to be
expected therein. There is a general on-line HELP command. The
commands are presented here in the order that they appear on the
output of the HELP command, that is, grouped in terms of their
purpose. These commands may be used when the program shows the
"Ready..." status. There are, however, some other instructions
(Input-EOF and Interrupt), also discussed below, which can be used
without the "Ready..." status.

FILES

Selection of the files assigned to the work session. Three files
are prepared. The first one (File 1), a binary file, will contain
all information related to the actual experiment: tube parameters
(wavelength, power), general conditions for peak recognition and
centering, setting angles and indices of the reflexions used to
calculate the orientation matrix, cell parameters and conditions
to be used to collect the intensities. The second file (File 2) is
an ASCII one prepared to contain the output of the data collection
process (*hkl* indices and intensities). The third file (File 3),
also ASCII, is dedicated to contain a list of input *hkl* indices,
only to be used if the reflexions to be collected are not to be
automatically generated by the program. The names and extensions
of these three files can be selected by the operator and normally
should be selected to remember its contents. Thus, if the file
names are selected as the compound identification (for instance
TEST), the File 1 can be selected as TEST.TAB to remember that it
is dedicated to contain the TABle of parameters to be used in the
experiment, the File 2 can be selected as TEST.DAT to remember
that it will contain the output of the DATa collection process and
File 3 can be chosen as TEST.HKL (it may contain the *hkl* indices
of the reflexions to be collected). In this way, if one has to
interrupt a working session and restart it again, all the
information of the old session will automatically be loaded if the
appropriate files are selected. This command is automatically
called at program start.

DUMP1

This command produces a dump (print out) of the parameters saved on File 1 (see FILES).

PRON

The attached printer can be activated with this command (PRinter ON).

PROFF

The attached printer can be switched off with this command (PRinter OFF).

WAVE

To change or just print out the wavelength of the X-ray tube.

POWER

To change the operating parameters of the X-ray tube (kV and mA) and to select the so called standby parameters, that is, kV and mA to be set at the end of the data collection process. The standby parameters (normally 5 kV and 5 mA) are very useful when the data collection ends during the week end or during the night.

SIGMA

This command inputs the sigma level to detect peaks above the background. It is used during the process of searching peaks needed to orient the crystal. See also SEARCH. A recommended value of 15 gives good results.

ATTEN

In order to avoid a non-linear answer of the detector at very high intensities, an attenuator filter is used during the data collection process. Two attenuator filters are mounted, for two different radiations. Two parameters can be selected using the ATTEN command: the most appropriate attenuator for the radiation used and the maximum intensity allowed to be measured without the attenuator. This intensity level should normally be estimated for every counter. An approximate value is about 50000 cps.

LLIM

This command produces a listing of the lower and upper limits of the goniometer angles, including the slits (if variable). The hardware limits are set into the general hardware file used by Seifert. However, some of these limits (those for 2θ, ω, χ, φ) are variable in terms of the so called zero corrections for the angular values, i.e., optical zeroes of the shafts, calculated as offsets of the hardware limits and set in the so called software file. See also ALIGN, LZER, MODZER.

NREF

This command calculates the number of lattice points (reflexions) expected within a selected angular range of the Bragg angle. The number of reflexions calculated corresponds to the whole Ewald's sphere. See also LLIM.

SURPAR

SURPAR is a general command which can be used to switch on or off the conditions for doing surface diffraction. If surface diffraction is wanted, the following SURface PARameters must be given: a) the χ and φ angles of the crystal surface selected, that is, the angles setting the normal of the surface onto the +y axis of the goniometer, and b) the angular range allowed for the angles α' and β' made between the normal of the surface and both, the incident (α') and reflected (β') beams. See also the Appendix.

QUASI

QUASI is a general command which can be used to switch on or off the condition for assuming quasi-crystal or non-quasi-crystal. This is of special interest if the *hkl* indices to be measured are included if the so called File 3, in which case they may be non-integer values. It also implies different output format for the reflexion records (see also the Appendix).

DATI

This is the general command (DATa Input) used to input the parameters governing the data collection process. After typing this command, the program is controlled by other subcommands (HELP, TEXT, ZONE, MODE, etc., explained below). The SAVE subcommand, exits DATI and returns control to the general command level. See also DATO.

DATI - TEXT

TEXT is used to input any alphanumeric information (crystal identification, etc.) to be also included in the output file produced by data collection (File 2).

DATI - ZONE

ZONE is a subcommand which allows the selection of the range in the Bragg angles limiting the reciprocal space containing the reflexions to be measured, as well as other parameters related with the zone of reciprocal space to be explored. The following parameters are expected: maximum and minimum Bragg angle, lower and upper limits for the indices, start values and increments to be used for generating the remaining indices. Alternatively, the individual *hkl* indices of the reflexions to be measured can be given through a file (see File 3 in FILES) containing a free-format record for each *hkl* triplet. See also NREF.

DATI - MODE

The MODE subcommand is used to select the scan mode, scan axis, scan width and motor speed (minimum of 0.01 deg/s). If a $\omega/2\theta$ scan is wanted, the M keyword for the scan axis should normally be selected, meaning that for the upper angle reflexions a non-bisecting geometry will be allowed. The scan width, which always refers to 2θ-width when 2θ is involved, is determined by two parameters in the form: *a + b tanθ*; thus, making *b=0* a fixed scan width will be used. For a variable scan width (not normally needed) an estimated *b* parameter of 0.3 is good enough for Cu and Mo radiations. A good way to determine the correct scan width is to use the general command SCAN on several reflexions, analyzing the results and choosing the proper parameters. The scan speed (in deg/s) refers to the 2θ axis when a $\omega/2\theta$ scan is selected. In this case ($\omega/2\theta$ scan), if the speed being input does not correspond to an appropriate speed for the coupled axis ω (1/2 of the main axis speed), the program will modify it slightly to ensure a 2:1 speed ratio.

DATI - SLITS

The detector slits (fixed or variable) are selected by the subcommand SLITS. The so called Hslit (Horizontal slit) is the slit which however can be opened and closed in the vertical sense. Therefore, the Vslit (Vertical slit) is the slit whose aperture can be variated in the horizontal sense. Both slits can be used as fixed (normal case) or with variable apertures (if available) during the data collection. Their apertures is determined by an expression similar to the one explained for the scan width (see DATI - MODE). Normally, two fixed slits of about 2 degrees give good results to collect intensities, but other values can be used if one previously uses the general SCAN command to select more accurate values of these parameters for the sample being used.

DATI - MAXN

MAXN selects the maximum number of scans allowed on a reflexion to get a minimum relative uncertainty of the intensity being measured. The strategy of measurement is as follows. Every reflexion is scanned with the selected conditions, the counts added to the previous scans and its relative uncertainty in percent, RU=100.σ(I)/I, is calculated. This value (RU) is then compared with two percentage limits (minimum and maximum RELative UNCertainty, see the DATI-RELUNC subcommand). If RU is greater than the maximum RELUNC parameter, no additional scan is done, the reflexion is coded as very weak (flag=2) and the goniometer is positioned to measure a new reflexion. If RU lies between both limits, the scan is repeated until the RU value is less than the minimum RELUNC parameter (reflexion is coded as normal, getting a flag=0), or until a preselected maximum number of scans is reached (the reflexion is coded with flag=1 if RU is still between the limits).

DATI - RELUNC

Two parameters are expected as input to RELUNC (RELative UNCertainty), the minimum and maximum relative uncertainty parameters used in the measurement strategy. See DATI-MAXN.

DATI - SYSABS

The lattice points generated (reflexion indices) are skipped (or measured) if the indices fulfil (or not fulfil) the conditions selected by means of the SYSABS subcommand (SYStematic ABSences). This subcommand expects up to 6 lines of text containing keywords which mean conditions to be checked. Each line may contain up to 3 keywords (separated by blanks) which will be considered as related each other with logical "AND" operators. Keywords given on separate lines will be related each other with logical "OR". The reflexion indices are checked against all the keywords so that the reflexion can alternatively be flagged according to the fact whether it fulfils, or not, all the keywords (combined with their respective logical operators). A total of 45 keywords are possible:

```
H=0     K=0     L=0     H=K     H=-K    K=L     H=L     K=-L
H=-L    H<0     K<0     L<0     H>K     H>L     K>L     K>H
L>H     L>K
```

```
AH>AK   AH>AL   AK>AL   AK>AH   AL>AH   AL>AK   (⁶ see below)

HN2     HN3     HN4     KN2     KN3     KN4     LN2     LN3
LN4     LN6     HKN2    KLN2    LHN2    HKN4    KLN4    HLN4
2HLN4   HKLN2   -HKLN3  H-KLN3  HK-LN3                 (⁷ see below)
```

Thus, selecting the following keywords given in 2 lines:

```
            H=0  L=0  KN2
            HLN2 K=0
```

reflexions of type:

```
            h=0     and   l=0     and   k≠2n    or
            h+l≠2n  and     k=0
```

that is:

```
            0k0  with  k≠2n     or
            h0l  with  h+l≠2n
```

will be skipped.

However, a new parameter (also considered in SYSABS) can decide just the contrary of the normal case, that is, if the reflexion indices fulfill all the conditions, the reflexion will be measured (this can be useful just to ensure that the expected systematic absences are really absent).

---

[6] The letter A means 'absolute value of'. Thus, for example AH>AK means $|h|>|k|$.

[7] The meaning of these conditions is better understood with some examples. In HKN2: HK means h+k, N means ≠, 2 means 2n; that is, h+k≠2n. In H-KLN3: H-KL means h-k+l, N means ≠, 3 means 3n; that is, h-k+l≠3n.

DATI - SYMREF

The subcommand SYMREF is used to generate the indices of any symmetry related reflexions. Thus, after measuring a reflexion, its *hkl* indices can be used to generate (and therefore measure) up to 48 symmetry related reflexions. To generate the symmetry related indices, a symmetry operator is used. Thus, for instance, to generate the indices *H=-h+k, K=k+l, L=-l* (not necessarily making any crystallographic sense), the corresponding operator will be:

$$\begin{pmatrix} H \\ K \\ L \end{pmatrix} = \begin{pmatrix} -1 & 1 & 0 \\ 0 & 1 & 1 \\ 0 & 0 & -1 \end{pmatrix} \begin{pmatrix} h \\ k \\ l \end{pmatrix}$$

DATI - OUTMOD

The output file (see File 2 in FILES) written during the data collection process will contain the net intensity (N) and standard deviation of every measured reflexion. However, there is also the possibility to save some additional records for each reflexion, containing the intensity profile and/or the angular information. This output alternative can be selected with the OUTMOD subcommand. Normally, the output mode N (net) will be used. Several record types are possible on File 2 (see the Appendix).

DATI - REFER

The stability of the X-ray source, the crystal orientation and the possible crystal decomposition (intensity decay) can be controlled using several reference reflexions which are remeasured periodically. The subcommand REFER is used to select the indices of these references and the parameters implied in this control measurement. One of these parameters determines the maximum variation allowed for the centroid of the reference reflexions respect to the centroids measured at the start of data collection. If the variation is greater than the given parameter, the crystal is reoriented, the references are remeasured and if their centroids still differ from the ones determined at the beginning, a warning message is produced and the new centroids are taken as new reference values. Another parameter being used by REFER, determines the maximum variation allowed for the intensities of the references respect to the reference intensities at the start of data collection; if the variation is greater than the given value, the data collection is stopped.

DATI - SAVE

When all DATI parameters have been selected, the SAVE subcommand
exits the DATI level and returns to the general command level. See
also DATO.

DATO

This (DATa Output) command is used to produce a print out of all
parameters selected at DATI time. See DATI.

DATCOL and DATCON

Once the operator has selected all the parameters to be used (see
DATI), the final step of the single-crystal work is just to
collect automatically the intensity of every reflexion. DATCOL
starts the data collection process. DATCON applies when an
interrupted data collection is to be continued, appending the data
to an old existing output file. Before DATCOL or DATCON is called,
the operator can use the DATO command to print out all the
parameters that configure the measurement process. The output
produced by DATCOL or DATCON contains the following information:
indices of the reflexions (reference reflexions are marked with an
R code and any reflexion within the expected limits being not
accesible is printed with the flag NA), the neto intensity
followed by its standard deviation, the attenuator code, the so
called "full width at half maximum" of the reflexion profile, a
code assigned to the relative uncertainty of the measurement, an
estimation of the asymmetry of the background at both sides of the
profile, the number of peaks detected on the profile, the
azimuthal angle ψ and a correlative record number.

DATCOL and DATCON also produce an output on File 2, depending on
the selection made by the DATI-OUTMOD subcommand. Thus, three
record types are possible on File 2 (see the Appendix).

MODZER

The zero reference points of the circles are set in the corresponding hardware file of the goniometer. These points are normally assigned at the time of the mechanical alignment and can differ up to one degree from the actual zero points (optical zeroes) defined by the X-ray beam. In order to calculate the real optical angle settings with respect to the X-ray beam, zero corrections should be introduced. If these corrections are large, there are two possibilities to introduce them. One is just to modify the corresponding hardware file, producing new mechanical reference points in that file. Alternatively, however, there is the possibility to maintain the mechanical reference points existing in the hardware file and use some corrections (software corrections) to that values which can be written into an assigned software file. For this second purpose, the command MODZER (MODify ZEROes) can be used. MODZER expects four zero corrections ($\Delta 2\theta$, $\Delta\omega$, $\Delta\chi$, ΔV), where V is the sum of the vertical upwards deviations of the X-ray source and the center of the slits system. The four values are given in degrees and are added (with their signs) to the zero corrections previously existing in the software file. See also ALIGN and LZER. When CRYSOM starts, the zero corrections existing in the software file are read and added to the zero reference points existing in the hardware file, producing correct angle settings.

LZER

LZER is the instruction used to print out the zero corrections existing in the software file. At program start, these corrections are automatically added to the zero reference points existing in the hardware file of the goniometer, thus producing correct angle settings. See also MODZER and ALIGN.

ALIGN

See before the MODZER command. The instruction MODZER is only used for large initial zero corrections. However, for fine adjustments the command ALIGN is much more useful. To use the ALIGN command, the goniometer must previously be set at a not too weak reflexion in the symmetrical setting at a χ angle near ±45 degrees. For this purpose it is not necessary to have a fully oriented crystal, but only one reflexion (roughly centered). For instance, one can mount a good reflecting crystal and use the SEARCH command to find out just one reflexion in the region χ near ±45. Once this reflexion is centered (after SEARCH), one can finally use the ALIGN command. The procedure of ALIGN is to center this reflexion in three

equivalent settings [(2θ, ω, χ, φ), (−2θ, −ω, χ±180, φ), (−2θ, −ω, −χ, φ±180)] and use these optimized setting angles to calculate the adjustments (zero corrections: Δ2θ, Δω, Δχ, ΔV) defined above (see MODZER). As indicated in MODZER, these corrections are added to the values previously existing in the software file. The instruction ALIGN must be repeated until the resulting adjustments (Δ2θ, Δω, Δχ, ΔV) are in the order of 0.01 degrees.

VIEW

The goniometer may be operated with a mounted microscope if it is located at its rear position, preventing crashes with the χ circle. VIEW is the command which can be used to set the appropriate angular values in order to observe the crystal through the microscope. The crystal, mounted on the goniometer head is to be centered by hand, using the translations of the goniometer head, to bring it into the goniometer center, i.e. it is moved into a position such that when any axis (χ and φ) is rotated no further crystal displacement is observed. Normally, this operation is done first by rotating the φ angle at 0 and 90 degrees and correcting, at both positions, the crystal translations perpendicular to the viewing direction and lying on the horizontal plane. Then, the crystal height (translation of the crystal up and down the goniometer head) should be corrected setting previously the χ angle at 90 and −90 degrees. After this, the χ angle should be driven to the 0 position and the φ rotation should be used to carefully correct the crystal translations lying on the horizontal plane (at φ = 0, 90, 180, 270 degrees). When these operations are done, just press the END key, put the microscope at the rear position and answer the asked questions. The program will show the "Ready..." status.

SEARCH

Before automatic data collection can be started, it is necesary to know the lattice constants and the orientation of the crystal (orientation matrix, OM). CRYSOM can determine this information from the unknown crystal in any arbitrary orientation. The program performs the necessary operations within a rather short time, so that this procedure should be used even if the lattice constants are known. The first operation to be done is to use the SEARCH command to find out several reflexions (up to 25) within a preselected angular range. The region to be searched is determined by giving an angular range for θ, χ and φ. The region is searched along arcs of constant θ and χ, with alternately increasing and decreasing values of φ. The magnitudes of the steps in θ and χ depend on the detector apertures, which are automatically (or manually) used. Normally the default values shown for the parameters to be used are good enough to begin with an unknown

crystal. This searching process stops when the complete angular range has been investigated, or when a pre-set number of peaks has been found, or when the operator interrupts the process by pressing CTRL A on the keyboard. The output of the SEARCH command can be used to determine the crystal orientation and hence the lattice constants. See also DETCEL and CENTAB.

DETCEL

Once the SEARCH command has finished, one can use the angular information contained in the peak table to build a reciprocal lattice and therefore calculate the cell constants. For this purpose, one can use the command DETCEL (DETermine CELL). Some little additional information required (maximum cell length to be accepted and the maximum angle to consider two reciprocal vectors as parallel) is requested with default values appropriate for almost any crystal. The first step of DETCEL is to convert the angular values, contained in the peak table, into orthogonal coordinates (*x, y, z*) of the reciprocal points according to the expressions shown in the Introduction. Each pair of points forms a vector. DETCEL finds the shortest vector (=$\mathbf{a}^*$), then the shortest one not parallel to $\mathbf{a}^*$ (=$\mathbf{b}^*$), and finally the shortest vector not in the plane of $\mathbf{a}^*$ and $\mathbf{b}^*$ (=$\mathbf{c}^*$). The remaining vectors which are found parallel to $\mathbf{a}^*$, $\mathbf{b}^*$ or $\mathbf{c}^*$ with equal moduli to them (or multiple moduli) are also averaged to a better determination of the base vectors. These three vectors describe always a primitive cell and determine the preliminary orientation matrix OM. The OM matrix is printed out together with the so called Reciprocal Metric Tensor (RMT) defined as the product of $OM_t.OM$ (where OM_t = OM transposed), the reciprocal cell constants, the direct cell constants and the cell volume (shown as a negative value when the chosen cell vectors form a left-handed system of axes). If the crystal is not twinned or cracked and OM is sufficiently accurate, the calculated *hkl* indices should be integers. A deviation of ± 0.125 is accepted and the indices are rounded to integers; otherwise they are printed as real numbers. New OM and RMT matrices are calculated using least-squares refinement with the reflexions having integer indices, and more accurate cell dimensions are printed. If not all indices are integers, once DETCEL is completed ("Ready..." status), the operator may reject the ones with fractional *hkl* indices using the PKSKP (PeaK SKiP) or PKREM (PeaK REMove) commands and calling DETCEL again. Finally, DETCEL proposes a cell reduction based on the Delaunay procedure. The cell reduction procedure can also be used as an individual command (see REDCEL).

INDEX

This is the command which carries out the least squares refinement to obtain the orientation matrix and the indexing of the reflexions included in the orientation table. INDEX is automatically called by other commands (DETCEL, MODOM, CENTAB).

REDCEL

This is the command used to perform a cell reduction using the Delaunay procedure. REDCEL is also automatically called by DETCEL. See also MODOM.

MODOM

In the case of a single crystal, DETCEL will produce a proper orientation matrix (OM). However, if the primitive cell calculated automatically by DETCEL does not correspond to the maximum crystal symmetry, or to the standard crystal orientation (what normally is shown by the cell reduction results) one can transform the crystal orientation (the OM matrix) to get the correct one, using the transformation matrix shown in the corresponding output of the cell reduction proposal (see DETCEL and REDCEL). This operation can be performed using the MODOM command (MODify OM), which can also be used to set new values for the OM matrix (not to transform). Once the OM matrix is modified, a least-square indexing procedure is started automatically, giving rise to a new output of OM matrix, cell constants, etc. See also LOM.

TWIN

In the case that DETCEL does not produce a proper orientation matrix (OM) because of the existence of a two single crystals in the sample, TWIN is a useful command which can report valuable information to detect the individual components of the sample.

LATREF

The procedure used by CRYSOM to improve the crystal orientation (see CENTAB) is based on a recentering of the setting angles of the reflexions in the orientation table. This procedure gives rise to an OM matrix which will predict satisfactorily the setting angles of the remaining reflexions. Therefore, the least squares refinement included in that process is always made considering a triclinic cell, that is, with no symmetry restrictions on the lattice parameters. However, in order to obtain the most accurate lattice constants (for an external use), a least squares refinement including symmetry constrictions is normally used, minimizing some function of the Bragg angles of the reflexions. Just for this purpose, CRYSOM includes a special routine called LATREF which produces very accurate Bragg angles of reflexions lying along several lines of the reciprocal space. Moreover, LATREF adjust the reflexions at both positive and negative 2θ angles, producing a list of Bragg angles corrected for any possible 2θ zero shift. The input expected by LATREF is up to 25 lattice rows (given as default) to be scanned. Optionally an output file is written containing a list of *hkl* indices and Bragg angles. The output of LATREF can also be very useful to detect the existence of possible systematic absences in terms of the NC codes of the non-centered reflexions.

PKLIS

This is the command used to list the information contained in the table of peaks used to orient the crystal. The output can be given in terms of setting angles or coordinates.

PKINC

This command INCludes PeaKs in the table of reflexions used to orient the crystal. See also PKREM. Several input modes are possible, indices, symmetry operators, angles and coordinates. The reflexions included in the table by PKINC are loaded with zero intensity, because they have not been centered. Therefore one should give a new opportunity to the goniometer to get a better set of angles for the peaks included in the table. This is done using the CENTAB command, which automatically recenters every peak of the table and produces a new least-squares refinement of both OM matrix and lattice constants.

PKSKP

In the case of a single crystal, DETCEL will produce a proper orientation matrix (OM). However, if for instance, the sample is no single (and the table of peaks shows peaks of the several components), DETCEL can produce non integer values for the reflexion indices, some of the vectors determining the cell can be poorly determined, or even no OM matrix will be produced. In these cases, some of the peaks being involved in the determination of the cell axes should be excluded and a new DETCEL started. This exclusion of peaks can be done removing (deleting) the selected peaks using the PKREM command. However if the operator wants just to exclude them (without deleting), PKSKP skips the selected peaks for the DETCEL procedure. Skipped peaks, also excluded from the indexing (INDEX) procedure, can be restored later on by the command PKRES.

PKRES

Several peaks of the table of reflexions being used to orient the crystal can be skipped for some operations (see PKSKP). The skipped peaks can be restored by using the PKRES command. See also DETCEL.

PKREM

If for any reason all or several peaks of the table of reflexions, being used to orient the crystal, are to be removed (deleted), PKREM performs this operation. See also PKINC, PKSKP, PKRES and DETCEL.

LOM

This is the command used to List (print out) the Orientation Matrix (OM). Some commands call automatically LOM. See also LRMT and MODOM.

LDCEL

This is the command used to List (print out) the Direct CELL. Some commands call automatically LDCEL. See also LRCEL.

LRCEL

This is the command used to List (print out) the Reciprocal CELL. Some commands call automatically LRCEL. See also LDCEL.

LRMT

This is the command used to List (print out) the Reciprocal Metric Tensor (RMT), defined as the product of $OM_t.OM$ (where OM_t = OM transposed). Some commands call automatically LRMT. See also LOM.

CENFAC

The peak centering process (optimization of peak angle settings) uses a parameter defining the factor which multiplies the scan amplitude used to center the peaks. This parameter can be modified using CENFAC (CENtering FACtor). A value of 1 is normally used, but for the case of big crystal plates, some value around 1.2 or 1.3 can give better results.

CENMOD

The peak centering process (optimization of peak angle settings using CENPK or CENTAB) can be done in two different ways. The normal procedure implies the following scans: φ, 2θ, χ, $2\theta/\omega$, which correspond to the value of 1 for the CENMOD parameter. However, in special cases, two additional scans (2θ and χ) can be forced after the first φ scan using the value 0 for CENMOD.

CENPK

This is the command to be used when a single PeaK should be
CENtered. This command assumes that the setting angles are roughly
on a peak. If the centering procedure does not find any peak, a
warning message of No Centering (NC) is printed. The strategy
being used to center peaks includes the following angle
optimizations: φ, 2θ, χ and a final ω/2θ (see also CENMOD). Each
angle optimization implies the determination of the angle
centroid, several corrections using the zero corrections (see
ALIGN) and the use of the proper automatic slits (if available)
for the given angle. The centering process produces a print out of
the optimized angles and the reflexion intensity measured on the
reflexion top with slits of 1x1 degrees (or with the fixed slits
being used). See also CENTAB.

CENTAB

The accuracy obtained for the crystal orientation, and therefore
for the lattice constants, after the peak searching process, is a
consequence of a unique process of peak centering, the one carried
out at SEARCH time. This means that one can still spend a little
more time to improve the setting angles of the peaks contained in
the table used to calculate the OM matrix. Moreover, one can make
some modifications in the table of peaks, removing (PKREM) some of
them (probably because several ones have low indices, or they are
too weak in intensity, or systematically one of the indices is
very low, or too high, etc.). If this is so, one can include new
peaks in the table by using the PKINC command (PeaK INClude),
using several input modes (indices, symmetry operators, angles,
coordinates). In both cases (no modifications done in the table of
peaks, or new reflexion settings introduced, and therefore loaded
with zero intensities) one should give a new opportunity to the
goniometer to get a better set of angles for the peaks included in
the table. This is done using the CENTAB (CENter TABle) command,
which automatically recenters every peak of the table and produces
a new least-squares refinement of both OM matrix and lattice
constants. See also CENPK and the centering strategy explained
there.

GOANG

This is the command to be used when selected angular values (2θ,
ω, χ, φ) are to be set. See also XCOM and LLIM.

GOHKL

This is the command to be used when a selected reflexion (given by its *hkl* indices and the azimuth angle ψ) is to be positioned. The corresponding setting angles (2θ, ω, χ, φ) are previously calculated and then positioned. The orientation matrix (OM) must be known. This command can also accept non-integer *hkl* indices.

HSLIT and **VSLIT**

Two detector slits are available. The so called HSLIT (horizontal slit) is the slit which however can be opened and closed in the vertical sense. The VSLIT (vertical slit) is the slit whose aperture can be variated in the horizontal sense. The commands HSLIT and VSLIT are used to set the selected apertures of these slits. See also XCOM and LLIM.

SHUTO

This command is used to Open the SHUTter. See also XCOM.

SHUTC

This command is used to Close the SHUTter. See also XCOM.

XCOM

This is the general communication command of the Seifert goniometers. It can also be used from the MS-DOS level. XCOM shows the actual values of some parameters, offers the possibility of moving the goniometer angles, including the detector slits (if variable ones are available), selecting the power parameters of the tube, opening and closing the shutter, starting the timer to get an intensity value on a peak, etc. See also GOANG, HSLIT, VSLIT, SHUTO, SHUTC and LANG. To exit XCOM just press the END key.

SCAN

Assuming that the goniometer angles are set on a reflexion peak, SCAN is the command used to analyse the peak profile. Some input parameters are expected: scan axis, scan mode, scan width, step width, speed or time and slits to be used. The output contains some valuable information as angular values at peak maximum and peak centroid, attenuator filter used, maximum and minimum intensity detected, background parameters, total and net intensities, standard deviation of the intensity, full width at half maximum and the number of maxima detected on the profile. A hardcopy of the profile is also possible (see PLOT). SCAN is the command to be used to make the analysis of the scan conditions for data collection (DATCOL or DATCON). See also DATI.

The SCAN command can also be used to make azimuth scans (ψ scans), meaning that the reflexion is scanned in angular positions corresponding to the different ψ values.

PLOT

The scans made during the peak searching process (SEARCH), peak centering (CENPK, CENTAB), scan analysis (SCAN), calculation of the angular zero corrections (ALIGN), determination of accurate Bragg angles for external lattice refinement (LATREF), and during the data collection process (DATCOL/DATCON), can be plotted in real time on the computer screen. The PLOT command allows this plotting capability, selects the maximum intensity of the plots and allows the possibility of producing a hardcopy on the printer. The conditions selected with the PLOT command will remain active until new conditions are selected.

LANG

The actual setting angles of the goniometer can be obtained using the LANG command (List ANGgles). See also XCOM.

EXIT

This is the program stop command. EXIT returns the control to the computer MS-DOS operating system.

Input-EOF (CTRL Z)

This is not a command, but a software possibility to produce the
so called input end-of-file, i.e. a soft mode of interrupting any
alphanumeric input to the program (during any command or
subcommand), returning the control to the "Ready..." status of the
program. The way to produce this Input-EOF is to press the CTRL
(control) key and (maintaining it so) press the Z key, release
both keys and press the Enter (Return) key.

Interrupt (CTRL A)

This is not a command in the sense described above for other
instructions. This interrupt is used to abort any program
operation which implies a scan (DATCOL, DATCON, ALIGN, SEARCH,
LATREF, CENPK, CENTAB and SCAN). It must be always used during a
scan process. The way to produce this interrupt is to press the
CTRL (control) key and (maintaining it so) press the A key.
Release both keys and wait until the actual scan is performed and
the program returns to the "Ready..." status.

LIST OF COMMANDS AND SUBCOMMANDS

These are the available general commands as they appear when using
the HELP command:

```
HELP

FILES   DUMP1   PRON    PROFF   WAVE    POWER   SIGMA   ATTEN   LLIM
NREF    SURPAR  QUASI   DATI    DATO    DATCOL  DATCON

ALIGN   LZER    MODZER

VIEW    SEARCH  DETCEL  INDEX   REDCEL  MODOM   TWIN    LATREF
PKLIS   PKINC   PKSKP   PKRES   PKREM
LOM     LDCEL   LRCEL   LRMT

CENFAC  CENMOD  CENPK   CENTAB

GOANG   GOHKL   HSLIT   VSLIT   SHUTO   SHUTC   XCOM    SCAN    PLOT
LANG

EXIT

Input-EOF:  Press  CTRL  Z   and  press Return
Interrupt:  Press  CTRL  A   and  wait
```

and these are the DATI-subcommands used to select the parameters
for DATCOL/DATCON:

```
HELP

TEXT    ZONE    MODE    SLITS   MAXN    RELUNC  SYSABS  SYMREF

OUTMOD  REFER   SAVE
```

APPENDIX

ON SURFACE DIFFRACTION

By activating the SURPAR command, CRYSOM allows the conditions for producing surface diffraction, that is, it collects the intensity data provided the crystal is positioned in such a way that the incident and reflected beams make a preselected angle with a given crystal surface. For this purpose, the angles χ and φ setting the normal of the crystal surface onto the +y axis of the goniometer, must be known. Then the operator must only decide the angular range allowed for the angles α' and β', in such a way that the angles formed between the outwards normal of the surface and both, the incident beam and the reflected beam are $\pi-\alpha'$ and β', respectively.

These conditions are obtained for each allowed reflexion if the scattering vector makes an angle (γ') with the outwards normal of the crystal surface in the range 0 to 90 degrees, that is, to ensure that the reflected beam does not pass through the bulky crystal. If this condition is fulfilled, the azimuth angle ψ is varied until α' and β' are in the allowed range, provided that the angular limits of the goniometer shafts are not reached and that the detector does not pass through a shadow zone.

RECORD TYPES ON FILE 2

The program produces three types of records on File 2 (see
DATCOL/DATCON), depending on the selection made by the DATI-OUTMOD
subcommand (see DATI and DATO).

Record type 1
The record of type 1, identical to the record produced on the
screen and on the printer, contains the following information:

1 mark h k l Ineto Sigma F Fwhm U A N ψ Record

corresponding to the following formats:

For non-quasi-crystals (see also the QUASI command)
(1x,'1',a1,6x,3i4,f14.0,f7.0, i3,f8.2,2x,i2,2x,i2,2x,i1,f6.0,i9)

For quasi-crystals (see also the QUASI command)
(1x,'1',a1, 3f8.3,f8.0,f7.0, i3,f8.2,2x,i2,2x,i2,2x,i1,f6.0,i9)

whit the following meaning:

1   record type (not printed out on the printer)
mark   blank or Q for normal reflexions (Q=quasi-crystal)
             R for reference reflexions
h k lthe reflexion indices (real numbers for quasi-crystals)
Inetothe net intensity
Sigmathe standard deviation of Ineto
F   the filter code (negative means TOO STRONG)
Fwhm   the full width at half maximum of the peak
U   the precision code of the measurement
A   the peak asymmetry code
N   the number of peaks detected
ψ   the azimuth angle
Record   the sequential record number

Record type 2

The record of type 2 is reserved for the peak profile:

2 mark numstp (buffer(j),j=1,numstp)

corresponding to the following format:

(1x,'2',a1,i4,9f8.0,/,(7x,9f8.0))

where:

2   is the record type
mark   blank or Q for normal reflexions (Q=quasi-crystal)
             R for reference reflexions
numstp   is the number of steps of the profile
buffer   contains the scan profile in cps

<u>Record type 3</u>

The record of type 3 is reserved for the angular information:

3 mark 2θ ω χ φ α' β' γ' ψ₀

corresponding to the following format:

(1x,'3',a1,f8.2,2x,3f8.2,2x,4f8.2)

where:

3	record type
mark	blank or Q for normal reflexions (Q=quasi-crystal)
	R for reference reflexions
2θ, ω, χ, φ	angles where the reflexion was positioned
α', β', γ', ψ₀	angles for surface diffraction, if activated (see SURPAR).

RECORD TYPES ON FILE 3

File 3 is an ASCII file dedicated to contain the *hkl* indices of the reflexions to be measured when the reciprocal points are not to be generated automatically by the program.

The structure of this file is always the same. It must have a first record, left justified, containing the string *File 3*, followed by free-format records with the indices (not necessarily integers) of the reciprocal points to be measured.
See the example shown below:

<u>Record 1</u>:
File 3

<u>Record 2 and following ones</u> (as many as necessary):
```
   3.256    -12.034    3
 4         6          -8
   5.4      -5         7.6667
```

The values of the indices are always read as real numbers, but their further interpretation will depend on the condition selected by the QUASI instruction. If the QUASI command selects a *non-quasy-crystal* condition, the *hkl* indices read from the file will be truncated to integers. On the contrary, if the QUASI instruction is used to select a *quasi-crystal* condition, the truncation of the indices will not take place.