Beginners guide to CRYSTALS

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1 Getting Started

This mini-guide is not meant to be a tutorial in solving and refining crystal structures, it is meant to assist users who know some basic crystallography, but want to be able to use CRYSTALS. If you want to learn about refinement in general there are a number of good sources:

- *The Control of Difficult Refinements*, D. Watkin, *Acta Cryst.* 1994, **50**(4) 747–749.
- *Structure refinement: some background theory and practical strategies*, D. Watkin, *J. Appl. Cryst.* 2008, **41**(3) 491–522.
- *Practical suggestions for better crystal structures*, P. Müller, *Cryst. Rev.* 2009, **15**(1) 57–83.
- *Crystal Structure Analysis Principles and Practice*, Chapters 12 & 13, Oxford University Press, Second Edition, Ed. Clegg.

1.1 Lists and the DSC file

In CRYSTALS refinement parameters and directives are held in lists, which are stored in the CRYSTALS "disc" file.[1] This file is a binary file (so don't try to open it with a text editor or print it) and has the extension dsc. The only way to interrogate the file is with the program CRYSTALS. Although there are more than thirty LISTS in the DSC file, it is generally only necessary to be familiar with very few of them. A full list is given in Section 3, but the most important are:

- LIST 5 - Atomic coordinates, A.D.P.s, etc.
- LIST 6 - Reflection data
- LIST 12 - Refinement directives including constraints
- LIST 16 - Restraints

1.2 Importing Data

There are several ways to start CRYSTALS. It is advisable to start with a folder containing your data, usually in the form of an import.cif or .hkl/ins files. From here you can start CRYSTALS from the icon

[1] Files in Microsoft Windows consist of two parts: a name and an extension. The extension tells the computer what type of file it is and what software to use to open it. Unfortunately, the default is to hide file extensions, and this can lead to lots of confusion, particularly when files have the same name or can be opened by more than one program. The default can be changed globally via the "Tools" menu in any open folder window (click the "View" Tab and unchecking the "Hide extensions for known file types" box). This is highly recommended.

on the desktop or in the start-up menu and browse for the working directory. However, the easiest way to begin is to navigate to the folder containing the data and right click on the folder icon on the top left of the task-bar and choose "Open Crystals here". If a .dsc file does not exist, this will create a new .dsc file in the folder you are currently viewing and open it.

The CRYSTALS "Guide" will try to help you to decide what to do at each stage. If The Guide doesn't start automatically when you open CRYSTALS, click on the brown CRYSTALS icon under the file menu. From here, what it tries to do depends on the files that are in the directory, but CRYSTALS will suggest you import the data (import.cif or .hkl/ins files); click "OK" and follow the advice. If the data are from a Nonius instrument and are contained in a file called import.cif, CRYSTALS will suggest you run the subroutine "KCCDIN" to import the data. This will ask a series of questions, including the Laue class, the space group, Z, the crystal size and colour, and the temperature of the data collection. Where files are in standard SHELX format (.hkl/ins), they will contain data as F-squared with sigmas (i.e use the defaults). On importing the data, it is advisable to merge symmetry equivalents and Friedel pairs unless the structure is in a non-centrosymmetric space group (as CRYSTALS advises) and use the recommended settings to filter reflections. If the data are in the form of .hkl/ins files CRYSTALS will try to determine the space group if it is included on the TITL card, if not, you will need to specify it yourself.

Once the data have been imported, CRYSTALS will then suggest you do an initial analysis of the data and will plot a series of graphs for inspection. Details of what information can be obtained from these is available under the analyse menu (Analyse > Help > Evaluate). When complete, click close. These graphs can be viewed at any time and are accessible via "Tabbed initial analyses" which can be found on the Analyse menu.

1.3 Structure Solution

If there are no atoms present, CRYSTALS will then suggest structure solution using direct methods with SIR92. Other solution software options are available through The Guide by choosing different options from the menu. Each solution package has a number of different options, but to begin with, try the defaults suggested by CRYSTALS. On closing the solution software, CRYSTALS will ask if you want to use the data (atomic positions). If you choose to import a solution, CRYSTALS will give you an opportunity to alter the atom types and and renumber the atoms ("Automatic" will number adjacent atoms sequentially as far as possible, while "Manual" will let you use an independent numbering scheme). Once you know what the material is, it is always advisable to number the atoms in a sensible, extensible manner before begining any refinement, because any restraints or constraints will refer to the atoms by name. For the same reason, the order of the atoms in the list is important as it is often quickest and/or easiest to refer to atoms in groups.

2 Refinement

2.1 The Guide

The Guide will lead you through a "routine" refinement, with standard options including refinement of positions, isotropic temperature factors and anisotropic temperature factors. If the refinement is well behaved, CRYSTALS will suggest what to do at each step and will finish by creating a CIF. An example of this type of structure can be found in the Demos (CYCLO). However, where there is disorder, or other problems it may be necessary to intervene.

2.2 LIST 5 - Refinable Parameters

LIST 5 contains all the atom parameters (positions, occupancies and temperature factors). Since all these data are stored in the binary data file, they cannot be manually edited. Many of these details can be changed using the context sensitive menu on the atom window and a file containing them can be opened and will be automatically read back into CRYSTALS on closing (Menu - Structure > Edit coordinates). However, in many cases, a better way of interrogating LIST 5 is to use the #EDIT command. This can be used to change the order atoms appear in the list, as well as altering specific values. Examples are given below (note, the "!" should not be typed and the text following only indicates what the command does).

```
\EDIT
AFTER N(1)
MOVE C(2) C(2) O(4)   ! Moves selected atoms after N(1)
END

\EDIT
CHANGE CL(5,OCC) 0.5  ! Changes the occupancy of CL(5) to 0.5
END

\EDIT
CHANGE CL(5,OCC) UNTIL CL(8) 0.5  ! Changes the atoms between CL(5) and CL(8)
END

\EDIT
CHANGE ENANTIO 0      ! Resets the Flack parameter to 0
END
```

For a full list of commands available within #EDIT, see the manual. All refined parameters can be seen using:

```
\PARAMETERS
END
```

2.3 LIST 12 - Constraints and Directives

LIST 12 contains the refinement directives including what parameters to refine and what constraints to be applied to them. Like LIST 5, the data cannot be manually edited directly, so the best way to edit them is via a text file. This file can be created through the GUI using the menus (Refinement > Edit directives and constraints) or by clicking the pair of interlinked rings (on the icon bar below the menu bar. This file contains a number of comment lines (beginning with # or a \ followed by a space) and the commands:

```
#LIST     12
BLOCK SCALE X'S, U[iso]   ! Refine the scale, all positions and isotropic ADPs
END

#LIST     12
BLOCK SCALE X'S, U[iso]   ! Refine the scale, all positions and isotropic ADPs
CONT C(10,OCC) C(11,OCC)  ! Refine the occupancy of atoms C(10) and C(11)
EQUIV C(10,OCC) C(11,OCC) ! Apply the same shift to atoms C(10) and C(11)
WEIGHT -1 C(11,OCC)       ! Apply opposite shifts to C(10,OCC) and C(11,OCC)
END
```

More details are given in Section 2.6.

2.4 LIST 16 - Restraints

LIST 16 contains details of restraints. Like LIST 12, the data in LIST 16 cannot be manually edited directly and is best edited via a text file. This file can similarly be created through the GUI using the menus (Refinement > Edit restraints) or by clicking the spring icon (on the bar below the menus). Like the file for LIST 12, this file contains some comment lines followed by commands:

```
#LIST     16
NO                    ! Indicates there are no restraints
END

#LIST     16
DIST 1.54 0.02 = C(1) to C(2)  ! Restrain C(1) to C(2) to 1.54(2) Ang.
END
```

More details are given in Section 2.6.

2.5 Hydrogen Addition

Hydrogen addition and refinement can be done in several ways, but the default as used by The Guide is "Add hydrogens + Fourier". This routine adds hydrogen atoms geometrically which it shows in white while it simultaneously displays peaks from the difference map in pink. The user can then delete any erroneously positioned hydrogen atoms and reassign Q-peaks as hydrogen if necessary. When OK is clicked, CRYSTALS will delete any remaining Q-peaks, apply a sensible numbering scheme to the hydrogen atoms and refine the hydrogen atoms against the data with additional restraints to maintain sensible geometries and A.D.P.s. Also, CRYSTALS will not try to add hydrogen atoms to nitrogen or oxygen atoms as this is particularly error-prone. In the case of these atoms, if a peak is visible in the difference map it can simply be identified as a hydrogen and CRYSTALS will do the rest. If the data are not adequate, it may be necessary to add the proton geometrically which is easiest to do by right-clicking the atom and choosing "Add hydrogens to *atom*". This hydrogen can then be refined as above either by repeating the "Add hydrogens + Fourier" or by using the "Refine hydrogen" option available from The Guide, or under Structure > Refine hydrogen (though the user may want to consider the validity of refining hydrogens that weren't visible in the difference map).

If using The Guide, these new hydrogen positions will then be used as relative positions for "RIDE" constraints which CRYSTALS will automatically add to LIST 12. If the refinement is more complex and The Guide cannot be used, either the RIDE cards or the additional LIST 12 commands must be added manually.

2.6 Example

This example shows how to deal with disorder in a tosylate group where S(21) is bonded to O(22), O(23) and O(24). The oxygen displacement ellipsoids were prolate, indicating the presence of disorder. Classically, this type of disorder is best modeled with two components. In CRYSTALS, atoms are best split using the right-click menu which in this case gives atoms O(220), O(221), O(230), O(231), O(240) and O(241). The original atoms, O(22), O(23) and O(24), can be deleted easily by selecting the ellipsoids and using "delete group" from the right-click menu.

Setting "part" numbers and changing atom names can most easily be done through the GUI under the "structure" menu or right-click "edit atom", so oxygen atoms O(220), O(230) & O(240), were

assigned to part 1 and O(221), O(231) & O(241) were assigned to part 2. Setting the U_{iso} values for the six oxygen atoms to the same value and sorting the atoms is best done via the command line and can be done in a number of ways, for example:

```
#EDIT
RESET U[ISO] 0.07 O(220) O(230) O(240) O(221) O(231) O(241)
AFTER O(220)
MOVE O(230) O(240) O(221) O(231) O(241)
END
```

LIST 12 (refinement directives)[2] for the initial partially isotropic refinement:

```
#LIST 12
BLOCK SCALE X'S PD(1,U'S) UNTIL C(31) ! Refine A.D.P.s for the other atoms
CONTINUE O(220,U[iso]) UNTIL O(241)   ! See footnote for information about
CONTINUE O(220,OCC) UNTIL O(241)      ! why these lines are not necessary
EQUIVALENT O(220,U[iso]) UNTIL O(241)
EQUIVALENT O(220,OCC) UNTIL O(241)
WEIGHT -1 O(221,OCC) UNTIL O(241)
END
```

Making all atoms anisotropic can be done as follows:

```
#LIST 12
BLOCK SCALE X'S U'S
CONTINUE O(220,OCC) UNTIL O(241)      ! Redundant; see footnote for info
EQUIVALENT O(220,OCC) UNTIL O(241)
WEIGHT -1 O(221,OCC) UNTIL O(241)
END
```

Note that the order that the atoms appear in LIST 5 (the model parameters) is critical here, because of the use of the "UNTIL" card. However, like many things in CRYSTALS, there is more than one way of doing this and if RESIDUE and PART numbers have been assigned,[3] this LIST 12 can be rewritten:

```
#LIST 12
BLOCK SCALE X'S U'S
```

[2]Note that all commands can be reduced to shorter strings, but are given here in full for clarity. For example, CONTINUE can be reduced to CONT. In addition, if a parameter is named explicitly in the constraints in LIST 12, it is not necessary to include it in the BLOCK command, though including them causes no harm.

[3]In CRYSTALS, if a structure contains two or more discrete molecular fragments, these are called RESIDUES. Atoms can be allocated to a RESIDUE, and the RESIDUE number be used to identify all those atoms e.g. RESIDUE(2). A RESIDUE can be divided (by the user) into PARTS. A PART is usually a bit of a RESIDUE of special interest - e.g. a disordered fragment. A PART has an identifier corresponding to 2 components: ASSEMBLIES and GROUPS. Different disordered regions are assigned to different ASSEMBLIES. Atoms in each part of a disordered region are assigned to a GROUP:

PART_ID = 1000*ASSEMBLY_ID + GROUP_ID

PARTS can be used to identify groups of atoms:

```
#EDIT
RESET OCC 0.5 PART(1001) PART(1002)
END
```

```
EQUIV PART(1001,OCC) PART(1002,OCC)
WEIGHT -1 PART(1002,OCC)
END
```

Note that these should be used with caution where there are hydrogen atoms in the structure because, for example, RESIDUE(1,U'S) directs CRYSTALS to refine anisotropic displacement parameters for ALL atoms included in RESIDUE 1.

Distance restraints may also be required:

```
#LIST 16
DISTANCE 1.47,0.01 = S(21) TO O(220) S(21) TO O(230) S(21) TO O(240)
CONTINUE S(21) TO O(221) S(21) TO O(231) S(21) TO O(241)
DISTANCE 2.45,0.01 = O(220) TO O(230) O(230) TO O(240) O(240) TO O(220)
CONTINUE O(221) TO O(231) O(231) TO O(241) O(241) TO O(221)
END
```

But it may be better to restrain all the distances to the same value:

```
#LIST 16
DISTANCE 0,0.01 = MEAN S(21) TO O(220) S(21) TO O(230) S(21) TO O(240)
CONTINUE S(21) TO O(221) S(21) TO O(231) S(21) TO O(241)
DISTANCE 0,0.01 = MEAN O(220) TO O(230) O(230) TO O(240) O(240) TO O(220)
CONTINUE O(221) TO O(231) O(231) TO O(241) O(241) TO O(221)
END
```

An alternative to the same distance restraints for the O$\cdots$O distances are angle restraints:

```
ANGLE 0,0.01=MEAN O(220) TO S(21) TO O(230) O(230) TO S(21) TO O(240)
CONTINUE O(240) TO S(21) TO O(220) O(221) TO S(21) TO O(231)
CONTINUE O(231) TO S(21) TO O(241) O(241) TO S(21) TO O(221)
```

Vibrational restraints and thermal similarity restraints can also be applied to groups of atoms. Not only can they be applied within a group but also between two or more grou ps:

```
DELU     S(21) O(220) O(230) O(240) AND S(21) O(221) O(231) O(241)
SIMU     S(21) O(220) O(230) O(240) AND S(21) O(221) O(231) O(241)
```

2.7 SHELX Commands

Below is a list of SHELX commands with a summary of how to access similar functions in CRYSTALS. For more detailed information see the CRYSTALS manual.

OMIT

There are two uses for the OMIT card in SHELX.

- OMIT can be used to specify reflections to be omitted from the refinement which can done via the GUI in CRYSTALS (Menu - Refinement > Filter reflections > OMIT tab) or by right clicking on disagreeable reflections on the Fo/Fc plot (Menu - Analyse > Fo vs Fc Graph).

- OMIT *atomnames* can be used to indicate dummy atoms that are retained in the atom list, but ignored in the structure factor calculation (sometimes used to give a clearer view of, say, disordered solvent). In CRYSTALS the easiest way to do this is to set the occupancy of the relevant atoms to zero and remove them from the refinement (in LIST 12). However, rather than actually editing LIST 12, it may be easier to simply recalculate structure factors (without refinement). This can be done through the GUI (Menu - Refinement > Recalculate phases).

SHEL & STIR

Resolution limits and merging instructions are most easily set through the GUI under the refinement menu (Refinement > Filter reflections). Note that the units are different to those used by SHELX.

TWIN & BASF

Twin matrices are stored in LIST 25, but can be easily input through the GUI. This includes both known twin-laws, or matrices determined using ROTAX (Analyse > ROTAX analysis/twins). The twin scale factors are stored in LIST 5 and can be easily seen using #PARAMETERS.

EXTI

The extinction parameter (EXTPARAM) is stored in LIST 5 and can be changed using #EDIT and viewed using #PARAMETERS. In order to refine an extinction parameter, it must be included in the BLOC card in LIST 12.

```
#LIST     12
BLOCK SCALE X'S, U'S EXTPARAM
END
```

MERG

Merging reflections is generally done when reflection data are originally input into CRYSTALS. If changes need to be made, it is usually best to reimport the data. However, functionality is available with #MERGE and details are available in the manual.

ANIS

Because the parameters refined by CRYSTALS are dictated by the contents of LIST 12, changing from an isotropic to an anisotropic refinement is done by substituting U[iso] with U'S for the relevant atoms.

AFIX & HFIX

Although there is no direct CRYSTALS equivalent, RIDE perfoms a similar function.

EXYZ & EADP

There are two ways of applying the same shift to parameters. EQUIVALENT and RIDE. EQUIVALENT applies a single shift to all the parameters specified, while RIDE will ensure that all *corresponding parameters* will receive the same shift. Thus:

```
EQUIVALENT C(1,X'S) C(2,X'S)
```

will apply the same shift to all the x, y and z parameters for C(1) and C(2), whereas,

```
RIDE C(1,X'S) C(2,X'S)
```

will apply the same shift to the x parameter of C(1) and the x parameter of C(2), but a different shift to the y parameters and the z parameters. The same rationale can be applied to A.D.P.s (U'S).

EQIV

EQIV and EQUIV have very different meanings in CRYSTALS and SHELX. In CRYSTALS, EQUIV (or EQUIVALENT) applies the same shift to two parameters whereas the SHELX EQIV card is used to indicate symmetry. In CRYSTALS, atoms are specified by $TYPE(SERIAL,S,L,Tx,Ty,Tz)$, where *TYPE* is the element; *SERIAL* is a number (1-9999), and *S*, *L*, *Tx*, *Ty* & *Tz* specify the symmetry relationship.

CONN, BIND & FREE

Controlling the connectivity table (LIST 40) is best done through the GUI. Adding bonds between two atoms is easiest by selecting the two atoms and using the "right-click" menu to alter the Bonding. Like LIST 5, LIST 12, LIST 16, it is also possible to edit them via a text file, which can be opened from the menu Structure > Add and remove bonds.

MOLE, RESI & PART

In both CRYSTALS and SHELX, it is possible to label molecules and parts of molecules so that they can be referred to when generating restraints, constraints and refinement directives. In CRYSTALS, different molecules are referred to with *RESIDUE* numbers which can be assigned automatically using the Structure menu. Parts of molecules can also be assigned *PART* numbers. A PART is usually a bit of a RESIDUE of special interest - e.g. a disordered fragment. A PART has an identifier corresponding to 2 components, ASSEMBLIES and GROUPS. Different disordered regions can be assigned to different ASSEMBLIES and atoms in each part of a disordered region assigned to a GROUP:

PART_ID = 1000*ASSEMBLY_ID + GROUP_ID

PARTS can then be used to identify groups of atoms:

```
#EDIT
RESET OCC 0.5 PART(1001) PART(1002)
END
```

DFIX, DANG & SADI

All restraints are stored in LIST 16. Distances can be restrained to a common value using the command "DISTANCE":

```
DISTANCE 1.47,0.01 = S(21) TO O(220) S(21) TO O(230) S(21) TO O(240)
```

where 1.47 is the distance in Å and 0.01 is the esd on the value. The distances are given sequentially of the form "*atom1* to *atom1*". Same distance restraints (the equivalent to SADI) can also issued with the DISTANCE command by replacing the distance:

```
DISTANCE 0,0.01 = MEAN S(21) TO O(220) S(21) TO O(230) S(21) TO O(240)
```

Angles can be similarly restrained:

```
ANGLE 0,0.01=MEAN O(220) TO S(21) TO O(230) O(230) TO S(21) TO O(240)
```

FLAT

Planarity restraints are stored in LIST 16 and the command "PLANAR" followed by an e.s.d. and a list of atoms can be used to restrain atoms to a plane.

```
PLANAR 0.01 C(11) C(12) C(13) C(14) C(15) C(16)
```

SAME

In Crystals, "SAME" can be added to LIST 16 to restrain similar fragments to the same geometry. Groups of atoms must be listed in the same order:

```
SAME N(11) C(12) C(13) O(14) C(15) AND N(21) C(22) C(23) O(24) C(25)
```

DELU & SIMU

"SIMU" and "DELU" are very useful for dealing with disorder and low resolution data. The "VIBRATIONS" card in LIST 16 can be can be used to set up "rigid bond restraints", i.e. restrain the vibration along the bond to similar values for adjacent atoms. In Crystals, these can be set up automatically for groups of atoms using "DELU". "DELU" works in a similar way to "SAME" and will not only restrain adjacent values in one set of atoms, but also apply restraints between sets.

```
DELU N(11) C(12) C(13) O(14) C(15) AND N(21) C(22) C(23) O(24) C(25)
```

The "U(IJ)'S" card can be used to restrain pairs of atoms to have the same U_{ij} components and "SIMU" can be used (in the same way as "DELU" and "SAME") to restrain U_{ij} values for groups of adjacent atoms.

ISOR

There is no direct Crystals equivalent.

SUMP & FVAR

Values can be restrained with equations in LIST 16, using the "RESTRAIN" card. This can be used to explicitly write an equation of restraint and Crystals will automatically calculate the value of the restraint and evaluate partial derivatives for each refinable parameter.

BLOC

Block refinement can be carried out by adding a second "BLOCK" card to LIST 12 and partitioning the parameters as required.

CGLS

There is no direct Crystals equivalent. However, because it is easier to alter the parameters being refined, refinement times can be significantly reduced by editing the "BLOCK" card in LIST 12 so that it only contains parameters of interest.

DAMP

The best way to control a refinement in Crystals is using shift-limiting restraints (Refinement > Add shift limiting restraints).

WGHT

The best way to modify weights is through The Guide (Optimise weights) or the GUI (Refinement > Choose weights) where Crystals will suggest parameters for several different weighting schemes.

BOND & CONF

By default, Crystals includes a complete, unique set of bonds and angles when a CIF is created. If the user also wishes to include torsion angles, these have to be appended to the file by the user. The following will add the C(1)–C(2)–C(3)–C(4) torsion to the end of the publish.cif.

```
#TORSION
ATOM C(1) C(2) C(3) C(4)
PUBLICATION PRINT=CIF
END
```

Substituting the ATOM card with:

```
ATOM C(1) C(2) C(3) C(4) C(5) C(6)
```

will additionally include C(1)–C(2)–C(3)–C(5) and C(1)–C(2)–C(3)–C(6) to the CIF.

RTAB, HTAB & MPLA

Crystals will automatically calculate hydrogen bond distances and angles and include them in the CIF. However, if additional geometric calculations are required, the "DISTANCES" routine will need to be used. Because Crystals stores the full variance co-variance matrix and is an active program, it is not necessary to carry out refinement to obtain distances with correct errors unless the parameter list (LIST 5) has been changed. For example, the following typed at the command prompt will give all the distance to atoms 1-3Å from O(11).

```
#DISTANCE
OUTPUT MONITOR=ALL
SELECT RANGE = LIMITS
LIMITS DMIN=1.00 DMAX=3.00
E.S.D.S COMPUTE=YES CELL=YES
PIVOT O(11)
END
```

More complex geometric calculations can be carried out using the "GEOMETRY" routine. For example, the following can be used to calculate the angle between two planes:

```
#GEOMETRY
ATOMS N(11) C(12) C(13) O(14) C(15)
PLANE
ATOMS N(21) C(22) C(23) O(24) C(25)
PLANE
ANGLE 1 AND 2
END
```

The "DISTANCES" and "GEOMETRY" routines contain an enormous amount of functionality which cannot be described fully here. Full details are available in the manual.

ACTA

CIFs are best written through the GUI either using the "Publish" option in The Guide, or using the menu, Results > Output CIF file. "ThetaFull", the value for which the programme calculates the completeness, is calculated automatically, however, it can be altered using the GUI, Results > Edif CIF goodies.

SIZE & TEMP

Crystal size and other data collection details can be altered using the GUI (Results > Edit CIF goodies).

WPDB

PDB and other format files can easily be created using the GUI (Results > Output other tables).

FMAP, GRID & PLAN

Peak searching in CRYSTALS is best done through the GUI (Fourier > Difference) and parameters can be set using the dialogue box. In general, the asymmetric unit is used for all space groups and peak searching activity.

It is also possible to view the 3D electron density by selecting atoms and using "Slant Fourier map" on the right-click menu in the model window. When dealing with disordered solvent, it is sometimes very useful visualise the actual electron density. The best way to do this is to set the occupancy of atoms in the disordered region to zero, calculate structure factors (Refinement > Recalculate Phases, or check the box in the Slant Fourier dialogue window), and then use the zero occupancy atoms to define a plane for the Slant difference Fourier.

3 List of LISTS

1. Cell parameters
2. Unit cell symmetry
3. Atomic scattering factors

4. Weighting schemes
5. Model parameters
6. Reflection data
7. Reflection data not used for refinement
8.
9.
10. Peak coordinates from Fourier
11. Least squares matrix
12. Refinement directives (inc. constraints)
13. Crystal and data collection data
14. Fourier directives
15.
16. General restraints
17. Special restraints
18.
19.
20. Transformation matrices from #GEOM*
21.
22. Refinement directive in internal format*
23. Structure factor control list
24. Least squares shift list*
25. Twin component operators*
26. Constraints in internal format*
27. Diffractometer scales
28. Reflection condition/filter list
29. Contents of asymmetric unit and element properties
30. General information
31. Cell parameter e.s.d.'s
32.
33. Internal - refinement control*
34.
35.

36. Tracking independencies of parameters*
37.
38.
39.
40. Bond forming/breaking directives
41. Bonds between atoms*

* indicates that the list cannot be directly input by the user.