

# CRYSTALS and SHELX

CRYSTALS and SHELX share much of a common functionality, but their syntax and method of use are quite different.

Listed below is a list of SHELX commands and their approximate equivalents in CRYSTALS compiled by Dr Amber Thompson.

In use, SHELX reads in a file of data and instructions mixed together, executes this file and outputs a new file of data and instructions, plus other files containing other information.

CRYSTALS is more like MS WORD in that it accumulates input from the user, and the stored data can be operated on by issuing instructions.

The accumulated data is kept in a database called `crfilev2.dsc`, which should NEVER be accessed by any other program than CRYSTALS. When CRYSTALS accesses this file, it opens a graphics window showing the current state of the structure, and provides a menu of most of the tools necessary for a normal refinement. These are arranged left to right more or less in the normal order of use.

See the item Beginners Guide under the Help tab.

## 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 be done via the GUI in CRYSTALS (Menu - Refinement > Filter reflections > OMIT tab) or by right clicking on disagreeable reflections on the  $F_o/F_c$  plot (Menu - Analyse >  $F_o$  vs  $F_c$  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 performs 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 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 to 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.