Chapter 2.1

PRELIM

Preparation of initial model file

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The programs under this menu item should normally be run at the beginning of a structural determination. The option AUTO-START causes an automatic execution and the programs may be run individually at any time during the structural analysis.

- The program E-STATISTICS carries out a Wilson plot, calculates the normalised structure factors (E's) and the statistics of the distributions of these E-values. This sometimes provide a valuable clue as to the presence of a centre of symmetry. NOTE however that the presence of heavy atoms, especially on special positions, and/or extended planar ring systems in light atoms structures, may give rise to misleading or even incorrect indications of the centricity. A graphical display of the Wilson plot and E-distributions is given. RMS deviations of the experimental curve (for all data) from those calculated for NONCENTRIC, CENTRIC and HYPERCENTRIC structures is calculated. A Figure of Merit (FOM) for the CENTRIC and ACENTRIC cases is then calculated, based on these percentage deviations. A FOM less than 0.1 is usually conclusive.

- The program ASSIGN SPACEGROUP first of all compares equivalent reflections in the .HKL file under all possible Laue symmetries. This provides a valuable check that the supposed Laue symmetry is indeed correct. The program then prints a full listing of potential systematic absences, and attempts to assign a space group. If E-STATISTICS has been previously run in the same session, then information about the centricity is passed to the program. A combined Figure of Merit (CFOM) is then calculated, based on the available information. The lower the value of CFOM, the more likely that the assignment is correct. A value below 10.0 indicates a satisfactory fit, while one below 1.0 indicates the chosen space group is highly likely.

- the program INITIALISE FILES will prepare the initial model file name.INS for a SHELXS run, and also write the file STRUCT.CIF. This latter file contains basic information about the crystal eg size, colour, crystal system, space group etc, which is not stored explicitly anywhere else. For instance the structure solution programs SIR92 and DIRDIF will read this file to obtain information about the unit cell and space group. The STRUCT.CIF file may of course be later modified, for example if the originally assigned space group was incorrect.

- The option AUTO-START will run the programs E-STATISTICS, ASSIGN SPACEGROUP and INITIALISE FILES in automatic sequence, with slightly different defaults and less output than when run individually. It will attempt to assign a unique space group, and will optionally run a default session of direct methods using SHELXS and open CAMERON to view the resultant .RES file.