Chapter 6.1.7.8

SHELXL-97

Fouriers & Peak-search

George W. Sheldrick
Dept. of Structural Chemistry
Tammannstraße 4
37077 Göttingen, Germany
email: gsheldr@shelx.uni-ac.gwdg.de

FMAP code[2] axis[#] nl[53]
The unique unit of the cell for performing the Fourier calculation is set up automatically unless specified by the user using FMAP and GRID; the value of axis must be non-zero to suppress the automatic selection. The program chooses a 53 x 53 x nl or 103 x 103 x nl grid depending on the resolution of the data. axis is1, 2 or 3 to define the direction perpendicular to the layers. Dispersion corrections are applied (so that the resulting electron density is real) and Friedel opposites are merged after the least-squares refinement and analysis of variance but before calculating the Fourier synthesis. This will improve the map (and bring the maximum and minimum residual density closer to zero) compared with SHELX-76. In addition, since usually all the data are employed, reflections with $\sigma(F)$ relatively large compared with F_c are weighted down. This should be better than the use of an arbitrary cutoff on $F_o/\sigma(F)$. The rms fluctuation of the map relative to the mean density is also calculated; in the case of a difference map this gives an estimate of the 'noise level' and so may be used to decide whether individual peaks are significant. Usually FMAP 2 is employed to find missing atoms, but if a significant part of the structure is missing, FMAP 5 or 6 may be better. ACTA requires FMAP 2 so that the difference density is on an absolute scale.

If code is made negative, both positive and negative peaks are included in the list, sorted on the absolute value of the peak height. This is intended to be useful for neutron diffraction data.

code = 2: Difference electron density synthesis with coefficients (F_o-F_c) and phases ϕ (calc).

code = 3: Electron density synthesis with coefficients F_o and phases ϕ (calc).

code = 4: Electron density synthesis with coefficients ($2F_o$-F_c) and phases ϕ (calc). $F(000)$ is included in the Fourier summations for code = 3 and 4.

code = 5: Sim-weighted ($2mF_o$-F_c) Fourier (Giacovazzo, 1992).

code = 6: Sim-weighted ($2mF_o$-F_c) Fourier with coefficients sharpened by multiplying with $\sqrt{E/F}$.

GRID sl[#] sa[#] sd[#] dl[#] da[#] dd[#]
Fourier grid, when not set automatically. Starting points and increments multiplied by 100. s means starting value, d increment, l is the direction perpendicular to the layers, a is across the paper from left to right, and d is down the paper from top to bottom. Note that the grid is 53 x 53 x nl points, i.e. twice as large as in SHELX-76, and that sl and dl need not be integral. The 103 x 103 x nl grid is only available when it is set automatically by the program (see above).

PLAN npeaks[20] d1[#] d2[#]

If npeaks is positive a Fourier peak list is printed and written to the *.res* file; if it is negative molecule assembly and line printer plots are also performed. Distances involving peaks which are less than r1+r2+d1 (the covalent radii r are defined via SFAC; 1 and 2 refer to the two atoms concerned) are printed and used to define 'molecules' for the line printer plots. Distances involving atoms and/or peaks which are less than r1+r2+|d2| are considered to be 'non-bonded interactions'; however distances in which both atoms are hydrogen or at least one is carbon (recognised by SFAC label 'C') are ignored. These non-bonded interactions are ignored when defining molecules, but the corresponding atoms and distances are included in the line printer output. Thus an atom or peak may appear in more than one map, or more than once on the same map. A table of the appropriate coordinates and symmetry transformations appears at the end of each molecule.

Negative d2 includes hydrogen atoms in the line printer plots, otherwise they are left out (but included in the distance tables). For the purposes of the PLAN instruction, a hydrogen atom is one with a radius of less than 0.4 Å. Peaks are assigned the radius of SFAC type 1, which is usually set to carbon. Peaks appear on the printout as numbers, but in the .res file they are given names beginning with 'Q' and followed by the same numbers. Peak heights are also written to the .res file (after the sof and dummy U values) in electrons $Å^{-3}$. See also MOLE for forcing molecules (and their environments) to be printed separately.

A default npeaks of +20 is set by FMAP; to obtain line printer plots, an explicit PLAN instruction with negative npeaks is required. If npeaks is positive the nearest unique atoms to each peak are tabulated, together with the corresponding distances. A table of shortest distances between peaks is also produced. For macromolecules and for users of the Siemens' SHELXTL system npeaks will almost always be positive! If npeaks is positive d1 and d2 have a different meaning. The default of d1 is then -1 and causes the full peaklist to appear in the .res file. If it is positive (say 2.3) then the full peaklist is still printed in the *.lst* file, but only suitable candidates for (full occupancy) water molecules appear in the *.res* file (with SFAC 4 and U set to 0.75). The water molecules must be less than 4 Å from an atom which begins with 'O', 'N' or 'W', and may not be nearer than d2 (default 3.0) from any atom which does not begin with 'O', 'N', 'W' or 'H', and may not be nearer than d1 to any 'O', 'N' or 'W' atom or to other potential waters which have larger peak heights. This facility is intended for extending the water structure of proteins in connection with BUMP and SWAT. To include the waters in the next refinement job, their names need to be changed and they need to be moved to before the HKLF instruction at the end of the atom list in the new *.ins* file. This can be performed automatically using SHELXPRO. It is recommended that the last water be called 'LAST' on the ISOR and CONN instructions so that its name does not need to be updated each job.

The heights and positions of the highest (difference) electron density maximum and the deepest minimum are output irrespective of the PLAN parameters.

MOLE n

Forces the following atoms, and atoms or peaks that are bonded to them, into molecule n of the PLAN output. n may not be greater than 99. n = 99 has a special meaning: the 'lineprinter plot' is suppressed for the following atoms, but the table of distances is still printed. This is sometimes useful for saving paper.