

3.4. INDEXING

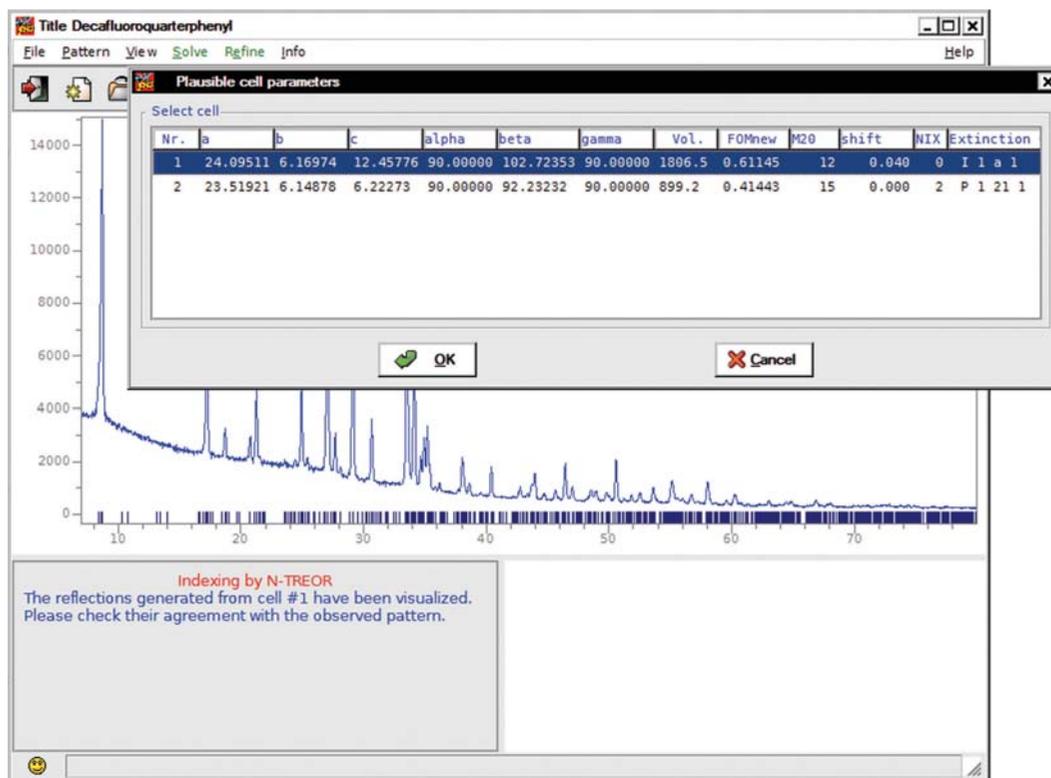


Figure 3.4.1

The list of possible cells for the decafluoroquarterphenyl structure automatically found using *N-TREOR09*.

The 2θ values of the first 25 peaks, in the range 5–25°, were determined by *WinPLOTR* and supplied to *DICVOL06*. The first 20 peaks were used for searching for the solution. No plausible cell was found when assuming that no impurity was present and exploring all the systems (from cubic to triclinic). *DICVOL06* was also unsuccessful when the non-default strategies of extended search and data correction for zero-point error were considered (by setting some flags to 1 in the input file). If it was supposed that two impurity lines might be present among the peaks (by setting the flag corresponding to the maximum number of accepted impurity/spurious lines to 2), *DICVOL06* was able to find the following monoclinic cell: $a = 8.7480$ (36), $b = 8.6313$ (32), $c = 7.6077$ (26) Å, $\beta = 97.201$ (33)°, with two unindexed lines, $M_{18} = 41.5$, $F_{18} = 125(0.0041, 35)$. The refinement of the cell by considering all the 25 lines gave $a = 7.6087$ (26), $b = 8.6295$ (30), $c = 8.7459$ (34) Å, $\beta = 97.201$ (34)°, which is very similar to the published one; 23 indexed lines, $M_{20} = 30.1$, $F_{20} = 102.6(0.0048, 41)$. The presence of the two impurity lines has been ascribed by the authors to a small amount of hexagonal plastic phase.

Example 2

Cu(II)–Schiff base complex (Banerjee *et al.*, 2002). Published information: $\text{Cu}(\text{C}_{15}\text{H}_{12}\text{NO}_2)_2$, triclinic, $a = 11.928$ (4), $b = 12.210$ (5), $c = 9.330$ (5) Å, $\alpha = 102.54$ (4), $\beta = 111.16$ (5), $\gamma = 86.16$ (4)°, $P\bar{1}$, experimental range 6–100° 2θ , $\lambda = 1.54056$ Å, RES = 1.22 Å, high-quality X-ray laboratory data, indexed by *DICVOL91*. The 2θ values of the first 30 peaks, in the range 6–25°, were determined by *WinPLOTR* and supplied to *DICVOL06*. The first 20 peaks were used for searching for the solution. If it was assumed that no impurity was present, no plausible cell was found down to the monoclinic system. When the triclinic system was explored, *DICVOL06* suggested only one plausible solution: $a = 12.2157$ (73), $b = 12.2031$ (77), $c = 9.3071$ (41) Å, $\alpha = 65.798$ (46), $\beta = 102.572$ (59), $\gamma =$

95.711 (61)°, with no unindexed lines, $M_{20} = 27.0$, $F_{20} = 77.0(0.010, 26)$. The refinement of the cell considering all the 30 lines gave $a = 12.2125$ (65), $b = 12.1989$ (61), $c = 9.3016$ (32) Å, $\alpha = 65.826$ (33), $\beta = 102.569$ (40), $\gamma = 97.755$ (44)°, no unindexed lines, $M_{20} = 27.9$, $F_{20} = 72.8(0.0106, 26)$. For this, the corresponding conventional cell is $a = 11.93313$ (61), $b = 12.2125$ (65), $c = 9.3016$ (32) Å, $\alpha = 102.569$ (40), $\beta = 111.152$ (33), $\gamma = 86.151$ (44)°, similar to the published one.

3.4.4.6.2. Indexing using *N-TREOR09*

Two examples of powder diffraction pattern indexing by using *N-TREOR09*, as implemented in the *EXPO* program, will be described. To activate the procedure some specific instructions must be given to *EXPO* via the input file or the graphical interface. As a first step, the peak-search procedure is automatically performed on the experimental powder pattern and the list of corresponding d values are supplied to *N-TREOR09*. During the indexing process a correction for zero-point error is automatically carried out (positive and negative shifts are taken into account). Both the examples below were successfully indexed by a default run of *EXPO*.

Example 3

Decafluoroquarterphenyl (Smrčok *et al.*, 2001). Published information: $\text{C}_{24}\text{H}_8\text{F}_{10}$, monoclinic, $a = 24.0519$ (9), $b = 6.1529$ (3), $c = 12.4207$ (5) Å, $\beta = 102.755$ (2)°, $I2/a$, experimental range 7–80° 2θ , $\lambda = 1.79$ Å, RES = 1.39 Å, medium-quality X-ray laboratory data. The first 43 peaks (in the range 7–67°) with intensities greater than a default threshold were selected (an intensity-based criterion is automatically adopted). The first 25 lines were used to find a possible cell that was then refined by considering all the 43 peaks. At the end of the automatic indexing procedure, *N-TREOR09* suggested two possible cells ranked according to WRIP20 [equation (3.4.5)], as shown in Fig. 3.4.1 (WRIP20 is denoted as