MS44-O4 Structure solution of nanocrystalline organic compounds from non-indexed powder data using cross-correlation functions

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Nanocrystalline organic compounds frequently show powder diffractograms with only 10 to 30, generally very broad reflections, which resist a reliable indexing. In such cases the crystal structure can, nevertheless, be solved by a direct fit of crystal structural models to the powder pattern using cross-correlation functions [1]. The cross-correlation function allows a fit of a simulated powder pattern to an experimental one, even if the lattice parameters considerably deviate and the calculated peaks do not overlap with the experimental ones. In the past, cross-correlation functions were used, e.g., for NMR spectra [2] or for assessment of the similarity between powder diagrams of different structures [3]. We use the cross-correlation functions for structure solution by global optimisation and developed a corresponding program called FIDEL (FIt with DEviating Lattice parameters) [1]. The procedure resembles the real-space methods used for structure solution from indexed powder data. The crystal structure is described by lattice parameters, space group, molecular geometry, molecular position and spatial orientation. Starting from random values, the lattice parameters, the molecular position and orientation are varied. In each step the powder diffractogram is calculated and compared wih the experimental one, and the structure is optimised until a good fit is achieved. In contrast to the real-space methods, lattice parameters are not given beforehand, but are optimised simultaneously with all other variables, starting from random values. This global optimisation is performed in all statistically frequent space groups. Subsequently the best structures are subjected to a Rietveld refinement with TOPAS, using a self-written automated refinement procedure. The best structure is finally post-refined by a user-controlled Rietveld refinement using TOPAS. The whole procedure is demonstrated on nanocrystalline samples of 2,9-dichloroquinacridone (R2=R9=Cl, R4=R11=H) and 4,11-difluoroquinacridone (R4=R11=F, R2=R9=H).

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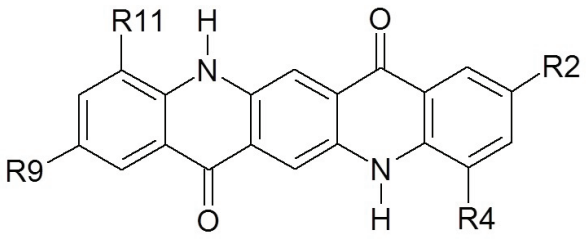

Figure 1.

Keywords: cross-correlation functions, structure determination from powder data, powder diffraction, organic compounds, nanocrystalline compounds