Poster

Stacking-disordered structure of Pigment Orange 13 (C32 H24 Cl2 N8 O2) from electron diffraction

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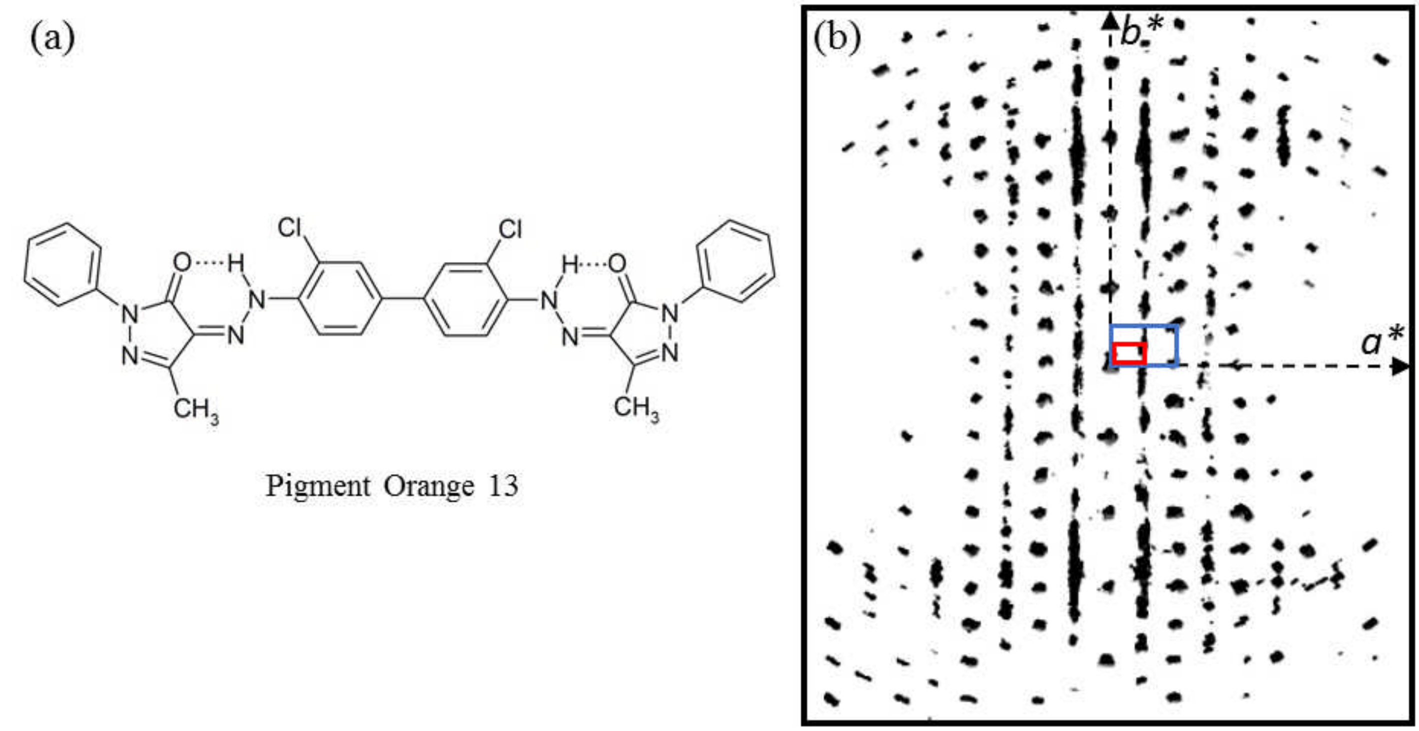
The α-phase of Pigment Orange 13 is a microcrystalline powder. No large crystals suitable for single-crystal X-ray diffraction could be grown. The X-ray powder diffractogram consists of only a few peaks, which could not be indexed reliably. Therefore, single-crystal 3D electron diffraction (3D ED) data were collected 1.



The electron diffraction space consists of sharp Bragg reflections and strong diffuse streaks (Fig. 1), associated with severe stacking disorder. The crystal structure could not be ab initio determined from electron diffraction data due to the disorder and the crystal deterioration in the electron beam. The structure was solved manually by analysis of the diffuse scattering and by exploring similari-ties with the previously determined crystal structure of the β-phase of the same compound. The structure consists of layers parallel to (010). In neighbouring layers, the molecules are rotated by about 90°. Subsequent layers are laterally shifted by either 1/4 or 3/4 unit cells in a-direction, leading to a stacking disorder. Consideration of only the Bragg peaks leads to the average structure in *Pbcn*, $Z = 2$, $Z' = 1/4$. The intensity maxima within the diffuse streaks point to a preferred local structure. We found that a 4-layer stacking with a zigzag arrangement (ABA'B) in the space group *C* 1 1 21/*g*, $Z = 8$, $Z' = 1$ best describes the features observed within the diffuse scatter-ing. This preferred local structure was validated by lattice-energy minimizations with DFT-D, and by fit to the experimental powder data. The stacking disorder is caused by the pseudosymmetry [2] of the layers: The shape of the layer has a higher symmetry than the layer itself; hence, a subsequent layer can be positioned in two different ways with almost the same packing energy, thus leading to the observed stacking disorder [1].

[1] Gorelik, T. E., Bekö, S. L., Teteruk, J., Heyse, W., Schmidt, M. U. (2023). Acta Cryst. B79, 122-137.

[2] Hörnig, A., Kölle, U., Schmidt, M. U., Herberich, G. E., Englert, U. (1993). Acta Chim. Hung. 130, 405-414.