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Powder structure analysis of oxotitanium phthalocyanine as charge generation materials

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Oxotitanium phthalocyanine (TiOPc) has been developed as a high sensitive charge generation material for organic photoconductors. It is known that TiOPc has four polymorphs (form I, II, C and Y) and the photosensitivity as charge generation materials depends on the crystal structures (sensitivity: form Y > I >> II >> C). The crystal structures of more stable polymorphs (form I and II) have been reported [1]. TiOPc form Y that has highest photosensitivity in the four polymorphs have never grown in large enough crystals for single crystal analysis. The crystal structure of form Y was determined by X-ray powder structure analysis using synchrotron radiation(Photon Factory BL4B2). TiOPc form Y crystal includes water solvents and MEM analysis using RIETAN-FP and VESTA[2] reveals the site of Oxygen atom of H_2O in the crystal. This result suggests that TiOPc form Y crystal has hydrogen bonds between the oxygen atoms of water solvents and Ti atoms of TiOPc.

[1] W. Hiller et al., Z.Krist., 159, 173 (1982)
[2] F. Izumi and K. Momma, Proc. XX Conf. Appl. Cryst., Solid State Phenom., 130, 15 (2007)

Keywords: powder structure determination, polymorphs, organic photoconductors

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Single crystal analysis of magnetically prepared pseudo-single crystal of sucrose

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The single crystal analysis with the x-ray diffraction is best suited for determining the three-dimensional structure of new compounds. However, preparing a single crystal that is large enough for the x-ray analysis is getting more and more difficult recently because the crystal growth is difficult for many compounds with complicated structures demanded in recent advanced materials sciences. Instead of the method of single crystal analysis, the ab-initio or direct space method is now available for the powder diffraction analyses and a number of crystal structures have been determined using this method. Unfortunately, the use of this method is limited to specialists who can correctly determine the cell constants and structure only from the two-dimensional information Recently, a magnetic technique of 3-dimensional alignment of powder crystallites has been developed. This technique enables to convert a powder to a pseudo-single crystal. In the present work, we demonstrate a single crystal analysis of a pseudo-single crystal of sucrose. We were able to index and solve the structure from this pseudo-single crystal using only a conventional method of single crystal analysis. The presentation includes the process of measurement, the solution of structure and the comparison of experimental data with simulation.

Keywords: pseudo-single crystal, magnetic technique, *ab-initio* structure determination

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Crystal structure model assembly program using Monte Carlo simulation

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A crystal structure model assembly program (SMAP) which designs starting models of Rietveld refinement was developed. This program requires XRD data, cell constants, space group, chemical formula and Z-number. On the basis of atomic positions selected by the Monte Carlo simulation, this program calculates the R-factor of the model from theoretical and observed XRD data. Hundreds of models of low-R-factor are selected and stored. Then, they gradually converge to a true structure. Using this program, the crystal structure of sibirskite ($CaHBO_3$) was solved and confirmed by the Rietveld refinement. The mineral sibirskite is monoclinic with space group $P2_1/a$. The calcium atom is surrounded by six oxygen atoms in an octahedral coordination, and a symmetrical pair of edge-shared CaO_6 octahedra forming a double chain elongates the crystallographic c axis. The BO_3 triangles are linked to three CaO_6 double chains to form the sibirskite structure. Sibirskite is isostructural with nahcolite ($NaHCO_3$), which consists of NaO_6 double chains and CO_3 triangles.

Keywords: Monte Carlo treatment, X-ray powder diffraction software, Rietveld method

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Application of the S-FFT phasing method to the solution of inorganic structures from powder data

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Since its discovery, the direct methods origin-free modulus sum function S [Rius (1993) Acta Cryst. A49, 406-409] has been used for solving some relatively complex inorganic crystal structures from powder X-ray diffraction data. This function was implemented in program XLENS [Rius (1999) Powder Diffraction 14, 267-273]. In this program phase values were estimated by maximising S with a modified tangent formula applied in sequential form. However, further progress in the powder diffraction field was hampered by the complexity of combining the tangent formula refinement (that makes explicit use of triple-phase sums) with the introduction of constraints in real space necessary to counterbalance the information loss produced by peak overlap. Recently, a considerably simpler and completely general phasing procedure (S-FFT) has been developed that maximises S by means of the FFT algorithm so that the triple-phase sums are considered implicitly [Rius, Crespi, Torrelles (2007) Acta Cryst A63, 131-134]. This algorithm has been adapted now to powder diffraction data and implemented in a new XLENS Windows version that can be retrieved from the toolbar of the FULLPROF-suite of programs (http://www.ill.fr/sites/fullprof/php/downloads.html) (Rodriguez-Carvajal, 2008). The effectiveness of the program