

at the atomic coordinates. In this contribution, we will demonstrate the use of dispersion-corrected DFT to reproduce molecular crystal structures in silico with unparalleled accuracy [2]. When the unit cell can be assumed to be reliable enough to be kept fixed, the computer resources required to perform an energy minimisation of all atomic coordinates are small enough for routine application, namely in the order of a couple of days on a single CPU. Because all atoms are treated independently and without reference to any user-supplied parameters or experimental data, the outcome of the dispersion-corrected DFT optimisation provides us an unbiased and independent validation criterion for the correctness of the crystal structure. Several examples will be shown.

[1] *Acta Cryst.* B63, 926.

[2] *J. Phys. Chem. B* 109, 15531.

Keywords: *ab-initio* powder structure determination, Rietveld refinement, density functional theory

P02.11.29

Acta Cryst. (2008). A64, C209

A comparison of co-crystal structure solutions through powder and single crystal techniques

Saul H Lapidus¹, Peter Stephens¹, Mike Zaworotko², Kapildev Arora², Tanise Shattock²

¹SUNY Stony Brook, Physics, SUNY Stony Brook, Stony Brook, NY, 11794, USA, ²Department of Chemistry, University of South Florida, Tampa, FL 33620, E-mail: slapidus@gmail.com

In powder diffraction, much information is lost by the collapse of the 3D space onto a 1D axis. This loss of information makes determination of the structure much more difficult. There is also some worry that this loss of information will lead to incorrect structure determination. However with single crystal techniques, the difficulty is in the preparation and growth of the single crystal. In some cases even, it is not possible to grow a single crystal. We have independently determined structures from powder and single crystal diffraction data of seven organic co crystals. The powder solutions were determined and refined with no knowledge of the single crystal solutions. We will compare the solutions given by these two techniques. This work was partially supported by Transform Pharma. Powder diffraction was performed at the National Synchrotron Light Source. The National Synchrotron Light Source at Brookhaven National Laboratory in New York, is a national user research facility funded by the U.S. Department of Energy's Office of Basic Energy Science

Keywords: powder structure determination, single-crystal structure determination, cocrystals

P02.11.30

Acta Cryst. (2008). A64, C209

The co-crystallisation and thermal behaviour of oxamic acid, nicotinamide and isonicotinamide

Adam J Cowell, Maryjane Tremayne

University of Birmingham, School of Chemistry, School of Chemistry, University of Birmingham, Edgbaston, Birmingham, West Midlands, B15 2TT, UK, E-mail: axc143@bham.ac.uk

In this poster, we will present our studies on a small group of molecular materials, while addressing three areas of small molecular crystallography that are of significant academic and

industrial interest: i) The systems under consideration contain both strong hydrogen bond acceptor and donor groups, making them potentially useful building units for co-crystal design and the study of supramolecular aggregation. The formation of molecular adducts of oxamic acid with both nicotinamide (vitamin B3) and isonicotinamide will be presented and the relative crystallisation and structural properties of these adducts discussed and compared to other results obtained for these isomeric amides. ii) In order to facilitate the full structural characterisation of these adducts, structure determination from X-Ray powder diffraction was required. By application of our direct space differential evolution algorithm we were able to solve the structures of these relatively complex structures from conventional laboratory data. Few molecular materials of this type have been determined from XRPD to date, but the ability to solve such structures has clear application to the emerging field of pharmaceutical co-crystals. iii) The thermal behaviour from 10 to 295K of both individual components and molecular adducts will also be presented. Such a study can be used as a valuable source of information in which to study the strength and directionality of intermolecular forces. We aim to invoke the reverse process, in which the directionality and strength of the intermolecular forces can be used to predict the thermal behaviour of a crystalline system.

References

1. McMahon, J. A., Bis, J. A., Vishweshwar, P., et al. *Z Kristallogr.* 220, 340-350

Keywords: direct space structure determination, molecular co-crystals, powder diffraction

P02.11.31

Acta Cryst. (2008). A64, C209-210

The third structure determination by powder diffractometry round robin (SDPDRR-3)

Lachlan M Cranswick¹, Armel Le Bail²

¹National Research Council Canada, Canadian Neutron Beam Centre, Building 459, Chalk River Laboratories, Chalk River, Ontario, K0J 1J0, Canada, ²Universite du Maine, Laboratoire des Oxydes et Fluorures, CNRS UMR 6010, Av. O. Messiaen, 72085 Le Mans, France., E-mail: lachlan.cranswick@nrc.gc.ca

The maturity of a scientific technique is considered established when the method is performed successfully "on demand". While small molecule single crystal structure solution is often described as routine (most structures solve easily and quickly "on demand"), structure solution from powder diffraction (SDPD) is generally not. In consequence of some literature reporting SDPD as "routine" (e.g., [1]), a first structure solution from powder diffractometry round robin was organized in 1998 [2], and a second in 2002 [3]. Both these round robins indicated that SDPD was still difficult, the ratio (submitted correct result)/(data download) being close to 2%. With the chairman of the IUCr Commission on Powder Diffraction (<http://www.iucr-cpd.org/>) indicating it is time for a new SDPD round robin [4], the competition was announced on the 1st of February this year with a deadline at the end of April 2008, providing three months to perform two SDPDs. Powder Diffraction datasets for an organic phase, and an inorganic phases were downloadable from a webpage (<http://sdpd.univ-lemans.fr/SDPDRR3/>) with cell parameters and probable chemical formula provided. At the time of writing this abstract (half round), there were 123 data downloads and 3 successful returns. Routine appears unattained yet in spite of 200 or so SDPDs currently published per year vs ~40,000 total structures published each year solved by non-powder methods.

[1] David, W. I. F., Shankland, K., and Shankland, N., (1998). *Chem.*

Commun., 931-932.

[2] Le Bail, A. & Cranswick, L. M. D. (2001). IUCr CPD Newsletter 25, 7-8.

[3] Le Bail, A. & Cranswick, L. M. D. (2003). IUCr CPD Newsletter 29, 31-33.

[4] David, W. I. F. (2007). IUCr CPD Newsletter 35, 2.

Keywords: powder diffraction, structure determination, round robin

P02.11.32

Acta Cryst. (2008). A64, C210

Magnetic alignment to convert powder crystallites into a pseudo-single crystal

Tsune-hisa Kimura, Fumiko Kimura

Kyoto University, Graduate School of Agriculture, tkimura@kais.kyoto-u.ac.jp, Kyoto, Kyoto, 606-8502, Japan, E-mail : tkimura@kais.kyoto-u.ac.jp

Magnetic alignment of feeble magnetic crystals, including most of organic and inorganic materials, has been well known for long time, and the biaxial alignment has been utilized in materials science, for example, to improve superconducting quality.[1] This technique of biaxial alignment is also useful for the diffraction study. There are two types of diffraction methods, that is, single crystal method and powder method. Using the magnetic technique, we can offer the third method (pseudo-single crystal method) that enables to obtain single crystal diffractions from a powder sample.[2,3] Biaxial alignment of powder crystallites is achieved using a dynamic magnetic field, and the obtained sample (pseudo-single crystal) exhibits the diffraction pattern equivalent to the corresponding real single crystal. The advantages of this method are (1) no large crystal is needed so that it is useful for the analysis of nano- and micro-crystallites, (2) the sensitivity is greatly enhanced compared to the powder method because the diffraction points randomly dispersed for the powder pattern are condensed, and (3) it helps to resolve overlapping peaks encountered in the two-dimensional powder diffraction analysis.

References

[1] PCT/NZ96/00108

[2] T. Kimura, M. Yoshino, Langmuir 21, 4805-4808 (2005).

[3] T. Kimura, F. Kimura, M. Yoshino, Langmuir 22, 3464-3466 (2006).

Keywords: pseudo-single crystal, magnetic alignment, X-ray diffraction

P02.11.33

Acta Cryst. (2008). A64, C210

3D alignment of LiCoPO₄ microrods by modulated magnetic fields for X-ray single crystal analysis

Fumiko Kimura^{1,2}, Chengkang Chang^{2,3}, Masataka Maeyama⁴, Katsunari Sasaki⁴, Tsune-hisa Kimura¹

¹Graduate School of Agriculture, Kyoto University, Division of Forestry and Biomaterials Science, Kitashirakawa Oiwakecho, Sakyo-ku, Kyoto, Kyoto, 606-8502, Japan, ²Tsukuba Magnet Laboratory, National Institute for Materials Science, 3-13 Sakura, Tsukuba, Ibaraki 305-0003, Japan, ³School of Materials Science and Engineering, Shanghai Jiaotong University, 800 Dongchuan Road, Shanghai 200240, P. R. China, ⁴Rigaku Corporation, 3-9-12 Matsubara-Cho Akishima, Tokyo 196-8666, Japan, E-mail : fkimura@kais.kyoto-u.ac.jp

Crystallographic analysis of particles of nano to micrometer sizes has drawn increasing attention. However, the analysis is limited to powder analysis because of the size of the particles to be examined. If the single crystal analysis is allowed on a powder sample, the information obtained will greatly increased. In this present work, we demonstrate a magnetic technique that enables to convert a powder to a pseudo-single crystal and the resultant XRD pattern. We demonstrated (1) that the biaxial crystals including the orthorhombic, monoclinic, and triclinic systems are aligned 3-dimensionally if a modulated rotating magnetic field is applied to the suspension of these crystals. We have succeeded in alteration of an L-alanine powder to a pseudo-single crystal using a frequency-modulated elliptic magnetic field (2). In the present study, a LiCoPO₄ powder was prepared by a modified hydrothermal method. The powder has square pole shape with ca. 2x2x20 mm³. The magnetic alignment of the powder sample was carried out using two different types of modulated rotating magnetic field: amplitude-modulated and frequency-modulated magnetic fields. The powder suspended in a photo-curable resin precursor was subjected to the modulated rotating magnetic fields, and the alignment was fixed by photo-polymerization of the precursor to obtain a pseudo-single crystal. The obtained samples exhibited almost the same X-ray diffraction pattern that was comparable to the pattern of its equivalent single crystal, enabling the structure analysis of this compound.

(1) T. Kimura, M. Yoshino, Langmuir 21, 4805-4808 (2005).

(2) T. Kimura, F. Kimura, M. Yoshino, Langmuir 22, 3464-3466 (2006).

Keywords: pseudo-single crystal, modulated rotating magnetic field, LiCoPO₄ microrod

P02.11.34

Acta Cryst. (2008). A64, C210-211

Preparation of pseudo-single crystal of sucrose from powder by magnetic alignment

Wataru Oshima, Fumiko Kimura, Tsune-hisa Kimura

Kyoto University, Forest and Biomaterials Science, oshima@wataru.mbox.media.kyoto-u.ac.jp, Kyoto City, Kyoto, 606-8502, Japan, E-mail : oshima@wataru.mbox.media.kyoto-u.ac.jp

Although the single-crystal X-ray analysis is a powerful means for the structure determination of crystals, a large single crystal is required for a successful analysis. In some cases, however, it is difficult to grow a large single crystal; only a powder sample is available. Powder analysis is also useful, but information obtained is limited. If the individual crystallites of a powder sample were all aligned three-dimensionally in a same manner, they would work as a pseudo-single crystal, giving X-ray diffraction comparable to the corresponding real single crystal. In fact, this is possible using magnetic alignment.(1,2) In this work, we report the preparation of a pseudo-single crystal of sucrose from its powder sample and discuss about the X-ray diffraction obtained from it. Large crystals of sucrose were pulverized to obtain a powder containing fine crystallites of 20 to 75 micrometer sizes. The crystallites were suspended in a UV-curable resin precursor and subjected to a modulated rotating magnetic field of 8 T, and then the achieved alignment was fixed by UV light irradiation. The obtained sample (a pseudo-single crystal) was subjected to the X-ray measurement. The obtained XRD pattern indicated that there are two types of crystal alignment in a magnetically aligned sample. This double alignment is regarded as a twin crystal. This observation is peculiar to the monoclinic system as expected by theoretical consideration.

(1) T. Kimura, M. Yoshino, Langmuir 21, 4805-4808 (2005).

(2) T. Kimura, F. Kimura, M. Yoshino, Langmuir 22, 3464-3466