

therefore extremely fast, enabling thousands of comparisons to the carried out per second on a standard PC.

[1] Morris R.J., Najmanovich R.J., Kahraman A., Thornton J.M., *Bioinformatics*, 2005, *in press*.

**Keywords:** bioinformatics, function prediction, active-site recognition

#### P.02.11.1

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##### Powder Structure Resolution of 1,7-Dioxaspiro[4.4]nonane

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Different attempts to crystallise compound 1,7-dioxaspiro[4.4]nonane, led to very small and irregular crystals which were not good enough to be analysed by the single-crystal X-ray diffraction technique. Moreover, the data collected with powder diffraction technique, was very poor to work with conventional direct methods for the structure solution.

The powder pattern was indexed using the program suite Crysfire. We have modeled different configurations in agreement with the other experimental analyses in order to test them with the powder diffraction data. We have located the different modeled solutions into the refined unit cell with the F.O.X. program. The Rietveld method was used for the refinement of the positions of non H atoms using the Bruker AXS Topas program.

Based on the results of the above mentioned method, it is possible to conclude that the technique of structural resolution by powder diffraction data is sensitive to changes of the atomic positions, or on the nature of atoms of the modeled molecule, and that this technique has allowed the confirmation of the structure of mentioned compound as it was suggested by means of spectroscopic techniques.

**Keywords:** powder structure resolution, organic structure determination, 1,7-dioxaspiro[4.4]nonanes

#### P.02.11.2

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##### The Structure Determination of Single-component Molecular-metal

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The single-component molecular metals, Ni(tmdt)<sub>2</sub> [1] and related materials, have attracted much interests due to their characteristic properties. Many other related materials have been produced by changing the metal atom and extended-TTF ligands. The crystal structure determination of newly produced materials is often very difficult due to only small amount of powder sample being available.

In this study, we determined 5 crystal structures of Ni(tmdt)<sub>2</sub> related materials, which are Ni(dt)<sub>2</sub>, Pd(dt)<sub>2</sub>, Au(tmdt)<sub>2</sub>, Pd(tmdt)<sub>2</sub>, and Pd(dmdt)<sub>2</sub>, by Genetic Algorithm combined with MEM/Rietveld method using synchrotron radiation X-ray powder diffraction data measured at SPring-8, BL02B2. The reliability factors, R<sub>wp</sub> and R<sub>I</sub>, of Rietveld refinements are in the range of 2~4% and 3~7%, respectively. It was found that the molecular stacking is different by the length of extended-TTF ligands. It is found that the positional relation of neighboring layers is closely related to the conductivity of materials.

[1] Tanaka H., et al., *Science*, 2001, **291**, 285-287.

**Keywords:** single-component molecular-metal, SR powder diffraction, genetic algorithm

#### P.02.11.3

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##### Ab-initio Structure Determination of C<sub>18</sub>H<sub>19</sub>N<sub>4</sub>S from Powder X-ray Diffraction

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The crystal structure of 1-(N-allylthiocarbamoyl)-3-(4-methylphenyl)-5-(pyrrol-2-yl)-2-pyrazoline, C<sub>18</sub>H<sub>19</sub>N<sub>4</sub>S, has been solved by the method of simulated annealing from synchrotron x-ray powder diffraction data. Pyrazolines are known to display various biological functions, such as fungicidal, antibacterial activities, pharmacological properties such as anti-inflammatory agents and industrial applications. The powder sample was sealed in 1mm capillary and diffraction data was collected with curved imaging plate method using 12KeV x-rays at the BL01C2 beamline in National Synchrotron Radiation Research Center (NSRRC). The structure was determined while following these procedures: (1) determination of the unit cell parameters, (2) determination of the space group, (3) extraction process by Pawley method, (4) structure solution by simulated annealing using DASH (David et al., 1998) and (5) Rietveld refinement by GSAS (Larson & Von Dreele, 1990) programs. The title compound crystallizes in triclinic system with space group,  $P\bar{1}$ , unit cell parameters of  $a = 12.603(14)$ ,  $b = 9.094(8)$ ,  $c = 8.494(8)\text{\AA}$ ,  $\alpha = 70.85(8)^\circ$ ,  $\beta = 105.26(8)^\circ$ ,  $\gamma = 109.10(7)^\circ$ , Volume =  $855.7\text{\AA}^3$  and  $Z=2$ . The final reliability factors of Rietveld refinement are  $R_{wp} = 0.039$   $R_p = 0.029$   $R_B = 0.118$  and  $S = 1.041$ .

**Keywords:** ab-initio structure determination, drug design, synchro powder diffraction

#### P.02.11.4

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##### Structure Determination from Powder Data of two Sub-peptides of Leu-enkephalin

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The crystal structures of two tripeptides, sub-peptides of leu-enkephalin which belongs to the opiate family of neuropeptides, have been solved from high resolution powder diffraction data using synchrotron radiation. Glycine-phenylalanine-leucine, C<sub>13</sub>N<sub>3</sub>O<sub>4</sub>H<sub>21</sub>, is monoclinic, space group  $P2_1$ , with  $a = 20.0024(8)\text{\AA}$ ,  $b = 4.8738(1)\text{\AA}$ ,  $c = 10.2778(2)\text{\AA}$ ,  $\beta = 103.940(1)^\circ$ ,  $Z = 2$ , at room temperature. Glycine-glycine-phenylalanine, C<sub>17</sub>N<sub>3</sub>O<sub>4</sub>H<sub>24</sub>·2H<sub>2</sub>O, recrystallised from water is orthorhombic, space group  $P2_12_12_1$ , with  $a = 30.3902(2)\text{\AA}$ ,  $b = 10.25972(8)\text{\AA}$ ,  $c = 4.83972(4)\text{\AA}$ ,  $Z = 4$ . The structures were solved via global optimization, programs TOPAS and FOX, and the use of maximum entropy maps.

**Keywords:** powder crystallography, peptides, synchrotron X-ray diffraction

#### P.02.11.5

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##### Crystal Structures of 8-Styrylxanthine Analogs from Powder Diffraction Data

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Adenosine modulates several physiological functions acting via specific G-protein-coupled receptor subtypes identified as A1, A2a, A2b and A3. Since the discovery that xanthines are the most important class of potent and selective antagonist at adenosine receptors (AR), the interest in this class of compounds has significantly increased. A novel classes of A2aAR antagonists [1] were investigated by means of X-ray powder diffractometry and the crystal structures of some analogs of 8-styrylxanthines: C<sub>14</sub>H<sub>13</sub>N<sub>6</sub>O<sub>2</sub>Cl, C<sub>14</sub>H<sub>14</sub>N<sub>6</sub>O<sub>2</sub> (azo-analogs) and C<sub>15</sub>H<sub>14</sub>N<sub>5</sub>O<sub>2</sub>Br (imine-

anlog) were solved from laboratory diffractometer data. The results can be useful for the development of more potent and selective A2aAR antagonists which are important for the treatment of Morbus Parkinson. Complexity of the investigated compounds solved by global optimization technique [2] ranges from 22 to 46 atoms in an asymmetric unit. Powder diffraction data were recorded both using Bragg-Brentano and DSH geometry (to reduce texture). It was astonishing that in the case of very strong texture one can encounter problems even in the indexing procedure. In such cases crystal structure solution process from powder data verifies the indexing and space group determination results.

[1] Muller C.E., Sauer R., Geis U., Frobenius W., Talik P., Pawłowski M., *Arch. Pharm. Pharm. Med. Chem.*, 1997, **330**, 181-189. [2] Favre-Nicolin V., Cerny R., *J. Appl. Cryst.*, 2002, **35**, 734.

**Keywords:** powder structure determination, pharmaceuticals, preferred orientation

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#### Preparation and Crystal Structure of Tetraperoxo Complexes of Molybdenum (VI) and Vanadium (V)

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Molybdenum and vanadium peroxo complexes have been widely investigated due to their potent catalytic properties in various reactions important for industry and environment. Peroxovanadium compounds have potent biochemical effects (effective insulin mimics, antitumor activity in mice).

In the poster we will present the crystal structures of potassium tetraperoxomolybdate (VI)  $K_2[Mo(O_2)_4]$ , caesium tetraperoxomolybdate (VI)  $Cs_2[Mo(O_2)_4]$  and lithium tetraperoxovanadate (V)  $Li_3[V(O_2)_4]$ .

The lattice parameters and space group were determined using PROSZKI package [1]. Structure models were built by using the global optimisation method (FOX program [2]) and refined by the Rietveld method (DBWS [3] and XRS-82 [4] programs).

[1] Łasocha W., Lewiński K., *J. Appl. Crystallogr.*, 1994, **27**, 437. [2] Favre-Nicolin V., Cerny R., *J. Appl. Cryst.*, 2002, **35**, 734. [3] Young R. A., Sakthivel A., Moss T. S., Paiva-Santos C. O., *J. Appl. Crystallogr.*, 1995, **28**, 366. [4] Baerlocher Ch., Hepp A., McCusker L. B., *XRS -82*, The X-ray Rietveld System of Crystallographic Programs for Powder Data, Zurich, 1982.

**Keywords:** peroxocomplexes, powder diffraction, Rietveld refinement

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#### Space Group Determination by EXPO2005

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The preliminary step to solve crystal structure is the determination of the space group. In case of powder diffraction data the peak overlap makes difficult the recognition of systematically absent reflections and therefore the extinction symbol identification. We have developed a new approach [1] based on the statistical analysis of the normalized intensities extracted by Le Bail method from the diffraction pattern. In order to improve the results new algorithms have been carried out regarding:

- the removal of impurity peaks;
- the background level determination;
- the variance associated to integrated intensity estimated;
- the selection of reflections relevant for the extinction group recognition;
- the graphic interface improvement

The new approach has been implemented in EXPO2005, the

evolution of EXPO2004 [2], and has been successfully tested using a large set of experimental data.

[1] Altomare A., Caliendo R., Camalli M., Cuocci C., da Silva I., Giacobuzzo C., Moliterni A.G.G., Spagna R., *J. Appl. Cryst.*, 2004, **37**, 957-966. [2] Altomare A., Caliendo R., Camalli M., Cuocci C., Giacobuzzo C., Moliterni A.G.G., Rizzi R., *J. Appl. Cryst.*, 2004, **37**, 1025-1028.

**Keywords:** space groups, powder diffraction, computing in crystallography

#### P.02.12.3

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#### Structures in Mo-Ru-Si System by Powder and Anomal X-ray Diffraction

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MoSi<sub>2</sub> attracted extensive interest during the last years because of its great potential for new high temperature structural materials. In order to improve MoSi<sub>2</sub> performance against "pest" phenomenon and glass corrosion at 1350°C additions of ruthenium metals have been made. This original way led us to investigate the Mo-Ru-Si ternary system that revealed the existence of five new phases noted  $\alpha$  (Mo<sub>15</sub>Ru<sub>35</sub>Si<sub>50</sub>),  $\beta$  (Mo<sub>26</sub>Ru<sub>47</sub>Si<sub>27</sub>),  $\gamma$  (Mo<sub>41</sub>Ru<sub>41</sub>Si<sub>18</sub>),  $\delta$  (Mo<sub>15</sub>Ru<sub>50</sub>Si<sub>35</sub>), and  $\sigma$  (Mo<sub>60</sub>Ru<sub>30</sub>Si<sub>30</sub>). The phases  $\alpha$  and  $\sigma$  are isotypes of the FeSi and FeCr structures respectively. The structures of the  $\beta$  and  $\gamma$  phases were determined ab-initio from synchrotron X-ray powder diffraction (XRPD) data. All of these phases are partially disorder with statistical occupancy of sites by the various metallic atoms. In order to increase the contrast between Mo and Ru and to obtain a crystallographic composition, X-Ray powder patterns were performed at the Mo-K threshold (20 keV) so as to use the anomalous effect. Therefore, multi refinement pattern using two wave lengths (one near the Mo Threshold, the other out) has been performed. Subsequently, an assessment of the site occupancy rates allowed to specify the distribution of the Mo ( $Z = 42$ ) and Ru ( $Z = 44$ ) atoms. Extension of the stability domain of  $\beta$  phase Mo<sub>4+x</sub>Ru<sub>9-x</sub>Si<sub>5</sub> ( $0 \leq x \leq 1$ ) is understood on the basis of the XRPD results and electronic properties calculation (KKR method) as a function of the composition [1].

[1] Littner A., François M., Tobola J., Elkaim E., Malaman B., Vilasi M., *Intermetallics*, 2005, in press.

**Keywords:** high melting systems, ab-initio powder structure determination, anomalous diffraction

#### P.02.12.4

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#### Algorithms for Solving Crystal Structure using Texture

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Texture is generally considered a nightmare for crystal structure solving or refinement. Wessels et al. [1] have demonstrated as texture can be used successfully to help the extraction of reliable structure factors from powder data to employ single crystal like structure solution methods.

The methodology has been extended a little further and some new algorithms will be presented to simplify the procedure in a unique improved step. Using an approach derived from Rietveld Texture Analysis [2] the simultaneous texture determination and structure factors extraction can be done. This was possible by introducing a new texture algorithm (EWIMV) and a texture aware extraction algorithm to combine the two procedures. By this method it is possible to extract structure factors from really highly overlapped pattern and use only one single experiment. An alternative method uses instead a Maximum Entropy Electron Map fitting approach to apply some constraints to the structure factors extraction.

The algorithms were implemented in the software Maud [3] along with other ab-initio structure solution routines for peak finding and indexing (through evolutionary algorithms), space group sorting and refinement constraints (energy computation, fragments etc.). Some