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.

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Keywords: powder structure determination, pharmaceuticals, prefferd 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:

a) the removal of impurity peaks;
b) the background level determination;
c) the variance associated to integrated intensity estimated;
d) the selection of reflections relevant for the extinction group recognition;
e) 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.

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Keywords: space groups, powder diffraction, computing in crystallography

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

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$MoSi_2$ attracted extensive interest during the last years because of its great potential for new high temperature structural materials. In order to improve $MoSi_2$ 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 α ($Mo_{15}Ru_{35}Si_{50}$), β($Mo_{26}Ru_{47}Si_{27}$), γ($Mo_{41}Ru_{41}Si_{18}$), δ($Mo_{15}Ru_{50}Si_{35}$), and σ($Mo_{60}Ru_{30}Si_{30}$). The phases α and σ are isotypes of the FeSi and FeCr structures respectively. The structures of the β and γ 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 anomal 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 β phase $Mo_{4+x}Ru_{9-x}Si_5$ ($0 \le x \le 1$) 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

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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 examples of application of the methodology will be presented as well.

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Keywords: structure determination, texture, Rietveld method

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New Tetragonal Phase in Al-Fe-U System

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Ternary aluminides in the Al-rich region of U-Fe-Al system have received considerable attention due to their interesting magnetic properties. Most of the structural studies of these materials were concentrated on UFe_xAl_{12-x} ($4\leq x\leq 6$) compounds of $ThMn_{12}$ type described by the *I4/mmm* space group [1]. Recently it was reported about another type of a ternary intermetallic phase with a composition UFe_2Al_{10} that could be formed in the U-Fe-Al system. Its structure was determined as orthorhombic, belonging to the space group *Cmcm* [2]. In the present work we report our results on structural characterization of a new ternary aluminide observed in Al-rich corner of U-Fe-Al system. Its approximate composition is Al-4.2at%Fe-8.5at%U suggesting the provisional stoichiometry of U_2FeAl_{20}.

Using transmission electron microscopy and electron microdiffraction technique [3] the structure of the new phase was established as tetragonal with the unit cell parameters a=12.41Å and c=10.30Å. The space group describing the structure is $I\bar{4}2m$. The atomic model of the structure was developed using direct methods applied to the data taken from X-ray powder diffraction. The reliability factors characterizing the Rietveld refinement are: Rp=12.9%, Rwp= 15.5%, Rbragg=7.07% and Rf=3.7%.

[1] Suski W., *Handbook of the Phys. and Chem. of Rare Earths*, 1996, **22**. [2] Meshi L., Zenou V.Y., Ezersky V., Munitz A., Talianker M., *J. Alloys and Compounds*, 2002, **347**, 178. [3] Mornirolli J.P., Steeds J.W., *Ultramicroscopy*, 1992, **45**, 219.

Keywords: structure determination, intermetallics, TEM

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Magnetic Structure of $BaFe_{12}O_{19}$ Determined by Resonant X-ray Magnetic Scattering

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Resonant X-ray magnetic scattering (RXMS) has attracted much interest as a useful tool to determine the magnetic structures associated with specific electronic states such as $3d$-$4p$ interactions. The resonant enhancement in the Bragg intensity between charge and magnetic scatterings [1] makes it possible for a tiny single-crystal to give a complete determination of the crystal structure and spin arrangement. M-type $BaFe_{12}O_{19}$ has been examined in this study, because there are five independent Fe sites in a hexagonal-ferrite structure, which are tetrahedral $4f_1$, bipyramidal $2b$, and octahedral $2a$, $4f_2$ and $12k$ sites.

RXMS experiments were performed at the Fe *K* absorption edge at BL-3A, Photon Factory. Diffraction profiles for more than 30 reflections of a single crystal of 0.07 mm in diameter were measured with right- and left-circularly polarized X-rays, which were produced passing through a diamond (001) phase retarder. The magnetic anomalous scattering factors were estimated in the structure-refinement procedure. The observed asymmetry ratios were in agreement with those made for the most appropriate spin-orientation.

[1] Namikawa K., Ando M., Nakajima, T., Kawata H., *J. Phys. Soc. Jpn.*, 1985, **54**, 4099.

Keywords: magnetic structure determination, resonant scattering, ferrites

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Neutron Diffraction Analysis of Photoisomerization of δ-Cyanobutyl Cobaloxime

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δ-Cyanobutyl groups in cobaloxime complexes were known to be isomerized to γ-, β-, α-cyanobutyl groups successively on exposure to visible light with retention of single crystal form. However, in case of the photoisomerization of δ-cyanobutyl cobaloxime with bulky diphenylborone in the neighborhood of alkyl group, Ph_2B-cobaloxime, only α-form was observed by X-rays as photoproduct and FT-IR experiments showed that β-form was produced with decreasing α-form and gradually increased, *i.e.*, δ→α→β reaction path not the "slide type" reaction δ→γ→β→α previously observed. This study was carried out to elucidate the detailed reaction mechanism by tracking the transfer of replaced two deuterium atoms. We prepared a large single crystal of δ-cyanobutyl-*2,2-d_2* Ph_2B-cobaloxime complex and carried out a single crystal neutron diffraction measurement after 2 days irradiation with BIX-3 diffractometer. The final R factor was 16.36 %. The result showed that a half of the δ-form was isomerized to α-form and one of the two deuterium atoms bonded to α position of δ-cyanobutyl group was transferred to δ position, *i.e.*, from Co–$CH_2CH_2CH_2C\mathbf{D}_2CN$ to Co–$C\mathbf{D}(CN)CH_2CH_2CH_2\mathbf{D}$. This result indicated the cyanobutyl group turned upside down after homolytic cleavage of Co-C bond and directly isomerized from δ-form to α-form.

Keywords: neutron diffraction, hydrogen transfer, crystalline state reactions

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Ab-intio Structure Determination of a Metal Complex from Laboratory X-ray Powder Data

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The crystal structure of Praseodymium tartrate, $Pr(C_4H_4O_6)_2.H_2O$, has been determined *ab initio* from laboratory X-ray powder diffraction data. Diffraction data were collected using monochromatized Cu Kα1 radiation (λ=1.5406Å) in the 2θ range 7.00-86.26° with a step width 0.02° and Bruker D8 Advance X-ray powder diffractometer. Indexing of the pattern was carried out using the program *NTREOR*. The solution with highest figure of merit [M_{20}=47.0, F_{20}=100.0] in the orthorhombic system having a=21.98(4), b=7.59(3), c=5.96(3) Å, agreed with the possible solutions obtained from the *DICVOL91* and *TREOR90* programs. Fitting the pattern with different possible space groups and a pseudo-Voigt function for the peak shape, the integrated intensities of 498 reflections were extracted based on the best fitted space group $P2_12_12_1$ using the program *TOPAS*. The position of the heavy atom was found from the Patterson map calculations. Attempts to build the structure with successive Fourier method were not successful. Finally the direct space approach using the program *FOX* with different input options led to a model structure containing one Praseodymium, two tartrate ions and one water molecule in the asymmetric unit. Rietveld refinement of the model structure with restraints using the program *EXPO2004* converged to Rp = 0.088, Rwp = 0.113, Re = 0.074 and GoF = 1.543, respectively. The final structure reveals that the Praseodymium atom is coordinated by nine O atoms of the ligands lying in the range 2.30 to 2.95 Å.

Keywords: ab-initio powder structure determination, direct space method, Rietveld refinement