FA4-MS35-P04

X-ray diffraction studies of intercalated

dichalcogenides at different temperatures. <u>SK Imran</u> <u>Ali</u>^a, Sander van Smaalen^a, Stefan Zoerb^b, Bernd Harbrecht^b, ^aLaboratory of Crystallography, University of Bayreuth, Germany, ^bDepartment of Chemistry, Philipps University, Marburg, Germany E-mail: <u>sk.imran@uni-bayreuth.de</u>

The crystal structures of intercalated transition metal dichalcogenides of the type 12R can be characterized by trigonal symmetry as determined by the structure from single crystal X-ray diffraction data [1]. Metal atoms of the MS_2 partial structure (M = Nb or Ta) have trigonal prismatic coordination. Cu atoms and additional M atoms may be incorporated on octahedral and tetrahedral sites in the Van der Waals gaps between layers MS_2 . Here we present the structural studies of selected intercalated compounds at different temperatures between 80K and 400K using single-crystal X-ray diffraction data. The interest in these compounds is the phase transitions related to ordering of the intercalated atoms and charge-density wave transitions at different temperatures.

[1] Harbrecht B., Kreiner G., Z. anorg. Allg. Chem., 572 (1989) 47-54

Keywords: X-ray diffraction, Intercalated transition metal dichalcogenides compound, Trigonal prism

FA4-MS35-P05

Chemical mixing and Raman hard mode spectroscopy in ferroelastic lead phosphate-arsenate: local symmetry splitting and multiscaling behavior. <u>Ulrich Bismayer</u>^a, Ekhard K.H. Salje^b, Tobias Beirau^a, Boriana Mihailova^a, Thomas Malcherek^a, ^aUniversität Hamburg, Germany, ^bUniversity of Cambridge, UK E-mail: ubis@mineralogie.uni-hamburg.de

The phase transition in ferroelastic Pb₃(PO₄)₂-Pb₃(AsO₄)₂ mixed crystals shows multiscaling behaviour with two relevant length and timescales. One length scale is macroscopic and shows uniform, weakly first-order phase transformations between a rhombohedral paraphase $(R\overline{3}m)$ and a monoclinic ferroelastic modification (C2/c). The second length scale is on the level of tetrahedral complexes which show monoclinic distortions at temperatures well above the point. transition macroscopic For example, in $Pb_3(P_{0,43}As_{0,57}O_4)_2$ the AsO₄ polyhedra show static deformation up to ca. 60 K above the deformation of the PO₄ tetrahedra. The two timescales are either short compared with the time of observation, namely the dynamic reorientation of the PO₄ tetrahedral distortion, or very long (optical birefringence, X-ray diffraction). The long timescale refers then to the quasi-static distortion of the AsO₄ tetrahedra which persists at $T > T_{C}$. These distortions appear to be uncorrelated or only weakly correlated and their random field leads to an order/disorder contribution of the ferroelastic phase transition which remains, on a phonon timescale, essentially displacive in character.

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Keywords: Hard Mode Raman spectroscopy, multiscaling

FA4-MS35-P06

Two polymorphs of DAEP anhydrate (C₁₄**H**₁₆**N**₂**O**₄) **from powder diffraction data.** Jürgen Brüning^a, Edith Alig^a, Jacco van de Streek^b, and Martin U. Schmidt^a ^a Goethe-University, Institute of Inorganic and Analytical Chemistry, Max-von-Laue-Str. 7, D-60438 Frankfurt, Germany, ^bAvant-garde Materials Simulation, Merzhauserstr. 177, D-79100 Freiburg, Germany

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N,N'-1,4-phenylene-bis(3-oxobutanamide) (1) is an industrial intermediate which is used as a coupling component in the synthesis of yellow azo pigments [1].



Two anhydrous forms could be obtained as powders by drying the dihydrate form either at 120°C (a phase) or over phosphorous pentoxide at room temperature (β phase). Single crystals of the anhydrate forms could not be obtained. The crystal structures were determined from laboratory X-ray powder diffraction data using DASH [2] and TOPAS [3]. The α phase crystallises in $P2_1/c$, Z = 2 with lattice parameters a =16.9749(5) Å, b = 4.8648(1) Å, c = 8.5372(2) Å, $\square =$ 93.396(2)°, with molecules on inversion centres [4]. The β phase crystallises in *P*-1 with a = 13.1696(4) Å, b = 7.9807(2)Å, c = 6.7519(2) Å, $\square = 105.037(1)^\circ$, $\square = 94.087(2)^\circ$, $\square =$ 96.075(1)°, Z = 2, with two symmetrically independent molecules, both on inversion centres. Thermal analyses and temperature-dependent X-ray diffraction experiments showed that the β form converts into the α form at 110°C. Both crystal structures were validated by dispersion-corrected DFT calculations using the program GRACE [5].

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Keywords: polymorphism, dispersion-corrected DFT calculations, structure determination from powder data

FA4-MS35-P07

Polymorphism of Pentadecanedioic Acid. <u>T. Calvet</u>^a, F. J. Novegil-Anleo^{a,*}, M. Font-Bardia^a, M.A.Cuevas-Diarte^a, Philippe Négrier^b and Denise Mondieig^b ^a Dep. Cristal·lografia, Mineralogia i Dipòsits Minerals, Facultat de Geologia Universitat de Barcelona (Spain) ^bCPMOH, Alliages Moléculaires et Stockage d'Énergie