

## FA4-MS35-P04

**X-ray diffraction studies of intercalated dichalcogenides at different temperatures.** SK Imran Ali<sup>a</sup>, Sander van Smaalen<sup>a</sup>, Stefan Zoerb<sup>b</sup>, Bernd Harbrecht<sup>b</sup>, <sup>a</sup>Laboratory of Crystallography, University of Bayreuth, Germany, <sup>b</sup>Department of Chemistry, Philipps University, Marburg, Germany  
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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<sub>2</sub> 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<sub>2</sub>. 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.** Ulrich Bismayer<sup>a</sup>, Ekhard K.H. Salje<sup>b</sup>, Tobias Beirau<sup>a</sup>, Boriana Mihailova<sup>a</sup>, Thomas Malcherek<sup>a</sup>, <sup>a</sup>Universität Hamburg, Germany, <sup>b</sup>University of Cambridge, UK  
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The phase transition in ferroelastic Pb<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>-Pb<sub>3</sub>(AsO<sub>4</sub>)<sub>2</sub> 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 $\bar{3}m$ ) 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 macroscopic transition point. For example, in Pb<sub>3</sub>(P<sub>0.43</sub>As<sub>0.57</sub>O<sub>4</sub>)<sub>2</sub> the AsO<sub>4</sub> polyhedra show static deformation up to ca. 60 K above the deformation of the PO<sub>4</sub> tetrahedra. The two timescales are either short compared with the time of observation, namely the dynamic reorientation of the PO<sub>4</sub> tetrahedral distortion, or very long (optical birefringence, X-ray diffraction). The long timescale refers then to the quasi-static distortion of the AsO<sub>4</sub> tetrahedra which persists at T > T<sub>c</sub>. 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.

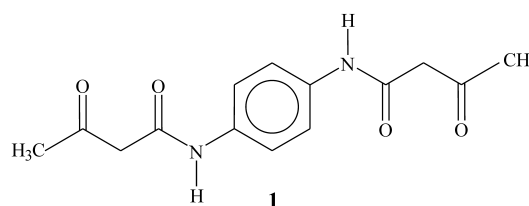
[1] Salje E.K.H., Beirau T., Mihailova B., Malcherek T., Bismayer U., *J. Phys.: Condens. Matter* 22 (2010) 045403.

**Keywords:** Hard Mode Raman spectroscopy, multiscaling

## FA4-MS35-P06

**Two polymorphs of DAEP anhydrate (C<sub>14</sub>H<sub>16</sub>N<sub>2</sub>O<sub>4</sub>) from powder diffraction data.** Jürgen Brüning<sup>a</sup>, Edith Alig<sup>a</sup>, Jacco van de Streek<sup>b</sup>, and Martin U. Schmidt<sup>a</sup>  
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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 ( $\alpha$  phase) or over phosphorous pentoxide at room temperature ( $\beta$  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  $\alpha$  phase crystallises in *P2<sub>1</sub>/c*, *Z* = 2 with lattice parameters *a* = 16.9749(5) Å, *b* = 4.8648(1) Å, *c* = 8.5372(2) Å,  $\angle$  = 93.396(2)°, with molecules on inversion centres [4]. The  $\beta$  phase crystallises in *P-1* with *a* = 13.1696(4) Å, *b* = 7.9807(2) Å, *c* = 6.7519(2) Å,  $\angle$  = 105.037(1)°,  $\angle$  = 94.087(2)°,  $\angle$  = 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  $\beta$  form converts into the  $\alpha$  form at 110°C. Both crystal structures were validated by dispersion-corrected DFT calculations using the program *GRACE* [5].

[1] Herbst, W., Hunger, K., *Industrial Organic Pigments*, 3rd ed., Wiley-VCH, Weinheim, 2004. [2] David, W.I.F., Shankland, K., Van de Streek, J., Pidcock, E., Motherwell, W.D.S., Cole, J.C., *J. Appl. Cryst.*, 2006, 39, 910-915. [3] A. A. Coelho, *TOPAS Academic*, version 4.1, 2007. [4] Brüning, J., Alig, E., Bats, J.W., Van de Streek, J., Schmidt, M.U., *Z. Kristallogr.*, 2009, 224, 593-597. [5] Neumann, M.A., Perrin, M.-A., *J. Phys. Chem.*, 2005, B109, 15531-15541.

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

## FA4-MS35-P07

**Polymorphism of Pentadecanedioic Acid.** T. Calvet<sup>a</sup>, F. J. Novegil-Anleo<sup>a,\*</sup>, M. Font-Bardia<sup>a</sup>, M.A. Cuevas-Diarte<sup>a</sup>, Philippe Négrier<sup>b</sup> and Denise Mondieig<sup>b</sup>  
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