X-ray diffraction studies of intercalated dichalcogenides at different temperatures. SK Imran Ali\textsuperscript{a}, Sander van Smaalen\textsuperscript{b}, Stefan Zoerb\textsuperscript{b}, Bernd Harbrecht\textsuperscript{a}, \textsuperscript{a}Laboratory of Crystallography, University of Bayreuth, Germany, \textsuperscript{b}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\textsubscript{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\textsubscript{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.


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

Chemical mixing and Raman hard mode spectroscopy in ferroelastic lead phosphate-arsenate: local symmetry splitting and multiscaling behavior. Ulrich Bismayer\textsuperscript{a}, Ekhard K.H. Salje\textsuperscript{b}, Tobias Beirau\textsuperscript{a}, Boriana Mihailova\textsuperscript{a}, Thomas Malcherek\textsuperscript{c}, \textsuperscript{a}Universität Hamburg, Germany, \textsuperscript{b}University of Cambridge, UK

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The phase transition in ferroelastic Pb\textsubscript{3}(PO\textsubscript{4})\textsubscript{2}–Pb\textsubscript{3}(AsO\textsubscript{4})\textsubscript{2} mixed crystals shows multiscaling behaviour with two relevant length and timescales. One length scale is macroscopic and shows uniform, weakly first-order phase transitions between a rhombohedral paraphase (R\textsuperscript{3}m) and a monoclinic ferroelastic modification (C\textsuperscript{2}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\textsubscript{3}(P\textsubscript{0.43}As\textsubscript{0.57}O\textsubscript{4})\textsubscript{2} the AsO\textsubscript{4} polyhedra show static deformation up to ca. 60 K above the deformation of the PO\textsubscript{4} tetrahedra. The two timescales are either short compared with the time of observation, namely the dynamic reorientation of tetrahedra, or very long (optical birefringence, X-ray diffraction). The long timescale refers to the quasi-static distortion of the AsO\textsubscript{4} tetrahedra which remains, on a phonon timescale, essentially displacive in character.


Keywords: Hard Mode Raman spectroscopy, multiscaling

Two polymorphs of DAEP anhydrate (C\textsubscript{14}H\textsubscript{16}N\textsubscript{2}O\textsubscript{4}) from powder diffraction data. Jürgen Brüning\textsuperscript{a}, Edith Alig\textsuperscript{b}, Jacco van de Streek\textsuperscript{c}, and Martin U. Schmidt\textsuperscript{d}

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Two anhydrous forms could be obtained as powders by drying the dihydrate form either at 120°C (α-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 P\textsubscript{2}1/c, Z = 2 with lattice parameters a = 16.9749(5) Å, b = 4.8648(1) Å, c = 8.5372(2) Å, α = 93.396(2)°, with molecules on inversion centres [4]. The β-phase crystallises in P\textsubscript{1}-1 with a = 13.1696(4) Å, b = 7.9807(2) Å, c = 6.7519(2) Å, α = 105.037(1)°, β = 94.087(2)°, γ = 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].


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

Polymorphism of Pentadecanedioic Acid. T. Calvet\textsuperscript{a}, F. J. Novegil-Anleoa, M. Font-Bardia\textsuperscript{a}, M.A.Cuevas-Diarte\textsuperscript{b}, Philippe Négrier\textsuperscript{a} and Denise Mondieig\textsuperscript{b}

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

Keywords: Hard Mode Raman spectroscopy, multiscaling

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Keywords: polymorphism, dispersion-corrected DFT calculations, structure determination from powder data
Dicarboxylic acids, HOOC-(CH2)n-COOH, are interesting compounds from the structural point of view [1]. A very long one-dimensional chain can be obtained through hydrogen bond formation at both ends of the molecule. These compounds configure layered structures in the solid state, as well as most of the polymorphic phases of long-chain compounds, such as n-alkanes, n-alcohols, and n-fatty acids. Dicarboxylic acids exhibit interesting thermal properties and a high enthalpy of melting that make them potential candidates for their use in the field of thermal protection and energy storage.

Dimorphism was prevalent in odd carbon members of dicarboxylic acids while one form is only present for even members: α form P21/c with Z=4 and β form C2/c with Z=4.

High-quality x-ray powder diffraction analyses of pentadecanedioic acid (C15H28O4) have been carried as a function of temperature. A new form, called γ has been observed just before melting; γ form C2/c with Z=4 at 383K. The packing arrangement of the carboxyl groups at both end of the chain is different in the three forms. From the structural point of view for γ form the distance interlayer is intermediate between α and β distances. Crystal structure of the pentadecanedioic acid is presented.

The carboxyl’s group in α form shows two different angles with middle plain of the chain. The packing arrangement of α and γ forms are radically different even if the molecular conformation are cis in both cases.

A reversible transition from β form to γ form was observed before melting. Slurry experiments indicate that β is the most stable form at room temperature.

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<th>36th European Crystallographic Meeting, ECM 26, Darmstadt, 2010</th>
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**Keywords:** organic semiconductors, crystal packing, polymorphism

**FA4-MS35-P08**

**Polymorphism as an Obstacle for the Applicability of Methoxy-substituted Distyrylpyrazines. Alain Collas**, Izabela Bagrowska, Krzysztof Aleksandrzak, Frank Blockhuys, Department of Chemistry, University of Antwerp, Universiteitsplein 1, B-2610 Wilrijk, Belgium, Faculty of Chemistry, Nicolaus Copernicus University, Gagarina 7, PL-87100 Torun, Poland

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**Distyrylpyrazines (DSPs) are a promising class of organic semiconductors because of their superior electronic properties, such as a highly efficient emission of light and a high electron affinity [1]. Since the supramolecular structure is of great importance when rationalizing and tuning these interesting properties for the materials' use in devices such as organic LEDs, five methoxy-substituted distyrylpyrazines were synthesized and their solid-state structures were investigated; (E,E)-2,5-bis[2-(4-methoxyphenyl)ethenyl]pyrazine has already been subjected to a synchrotron study by Scaccianoce et al. [2]. For (E,E)-2,5-bis[2-(3,4-dimethoxyphenyl)ethenyl]-pyrazine, (E,E)-2,5-bis[2-(3,4,5-methoxyphenyl)ethenyl]pyrazine and (E,E)-2,5-bis[2-(2,4,6-methoxyphenyl)ethenyl]pyr-