

**P.22.01.1***Acta Cryst.* (2005). **A61**, C475**Structure of LaSrCuO<sub>3.5</sub>**Joke Hadermann<sup>a</sup>, N. Creon<sup>b</sup>, O. Perez<sup>b</sup>, C. Michel<sup>b</sup>, M. Hervieu<sup>b</sup>,  
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The oxygen deficient La<sub>1.07</sub>Sr<sub>0.93</sub>Cu<sub>0.92</sub>O<sub>3.52</sub> has been synthesized and the structure has been determined using a combination of transmission electron microscopy, X-ray diffraction and neutron diffraction.

X-ray diffraction showed a basic K<sub>2</sub>NiF<sub>4</sub> type structure of  $a=3.7583(3)$  Å and  $c=13.005(3)$  Å in space group  $I4/mmm$ . However some reflections remained unexplained. Therefore electron diffraction was performed which showed the presence of an incommensurate modulation and allowed the determination of the superspacegroup and modulation vectors as  $I4/mmm(\alpha\alpha 0, \alpha-\alpha 0)00mg$ ,  $q_1=0.22a^*+0.22b^*$  and  $q_2=0.22a^*-0.22b^*$ . Following these observations neutron diffraction was used to determine the origin of these modulations.

The five-dimensional model obtained using the super space formalism shows oxygen-vacancy ordering, implying three different environments present for the copper in the structure: octahedral, pyramidal and square plane coordination. Significant displacements in the (a,b) plane occur for all atoms, no displacement is observed along the c-axis.

High resolution electron microscopy images support the model.

**Keywords:** incommensurate structures, copper oxides, structural determination

**P.22.01.2***Acta Cryst.* (2005). **A61**, C475**HMT-Resorcinol: An Example of Modulated Structure in Substituted HMT Adducts**M. Soraya Rodriguez, Gervais Chapuis, *Laboratoire de cristallographie, École Polytechnique Fédérale de Lausanne, Switzerland.* E-mail: soraya.rodriguez@epfl.ch

Hexamethylenetetramine (HMT) has a tendency to create modulated structures when forming adducts with hydroxyl moieties. Many examples have been reported recently [1], [2], [3], [4], [5]. We report here a model of the incommensurately modulated structure of HMT-Resorcinol solved at room temperature, in the superspacegroup  $Xmcm(0b0)s0s$  (where the X centering corresponds  $h+k+m=2n$ ). The model consists mainly in an occupation modulation of the Resorcinol, which can adopt two different positions related by a mirror plane. This compound undergoes a lock-in phase transition of second order at about 278K yielding a commensurate structure. X-ray diffraction data have been collected at 120K, with LT cell parameters related to the room temperature ones by ( $a_{LT}=a_{RT}$ ,  $b_{LT}=2b_{RT}$ ,  $c_{LT}=4c_{RT}$ ). A comparison of the supercell model and a commensurate model derived from the superspace formalism will be presented.

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**Keywords:** superspace refinement, incommensurate structures, modulated structure

**P.22.01.3***Acta Cryst.* (2005). **A61**, C475**The Way of Crystal of [cpMe<sub>4</sub>SiMe<sub>2</sub>N(t-Bu)]TiCl<sub>2</sub> from Disorder thru Modulation to Twinning**Ivana Cisařová<sup>a</sup>, Václav Petřiček<sup>b</sup>, Karel Mach<sup>c</sup>, <sup>a</sup>Department of Inorganic Chemistry, Charles University in Prague, Czech Republic. <sup>b</sup>Institute of Physics, Prague. <sup>c</sup>J. Heyrovský Institute of Physical Chemistry, Prague. E-mail: cisarova@natur.cuni.cz

Constrained geometry catalysts based on *ansa*-{(tert-butylamido-κN)dimethyl(η<sup>5</sup>-2,3,4,5-tetramethylcyclopentadienyl)silane} dichlorotitanium (IV) (**1**) allowed for a large scale production of new polymers and copolymers of specific properties [1] The structure of compound **1**

was investigated by X-ray diffraction a [2,3], however the geometrical parameters were not published in full.

From our reinvestigation [4] follows that the *tert*-butyl moiety is disordered in two positions over mirror operation of Pnma space group at room temperature. The new measurement of **1** at low temperatures revealed two phase transitions by reordering t-Bu moiety. The results of first one can be described in supercell **4a,b,c** (**1b**), and space group Pna2<sub>1</sub>, the second one (**1c**) as **3a,b,c** and P2<sub>1</sub>/n11 pseudomorphedically twinned into the lattice symmetry mmm. Whole pathway can be unified by applying the modulation concept, superspace group Pnma(a00)0s0 with two q-vectors (1/4,0,0) and (1/3,0,0) for **1b** and **1c**, respectively, affording unique opportunity to test various models (commensurate versus incommensurate etc.).

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**Keywords:** modulated crystal structures, phase transitions, catalyst structure

**P.22.01.4***Acta Cryst.* (2005). **A61**, C475**Structure of the Pseudodecagonal Al-Co-Ni Approximant PD4**Peter Oleynikov<sup>a</sup>, L. Demchenko<sup>a</sup>, S. Hovmöller<sup>a</sup>, X.D. Zou<sup>a</sup>, M. Döblinger<sup>b</sup>, B. Grushko<sup>c</sup>, <sup>a</sup>Structural Chemistry, Stockholm University, Stockholm, Sweden. <sup>b</sup>University of Oxford, Parks Road, Oxford OX1 3PH, U.K. <sup>c</sup>IFF, Forschungszentrum Ju'lich GmbH, Ju'lich, Germany. Department of Materials. E-mail: oleyniko@struc.su.se

The similarity of crystalline approximants to the quasicrystals in terms of their compositions, densities and diffraction patterns, makes them very important for determining the structures of quasicrystals. Several crystal structures of these approximants were determined [1–2].

Several periodic pseudodecagonal (PD) structures were found in the Al-Ni-Co system. They are named PD1, PD2 etc. and exhibit diffraction patterns with almost perfect tenfold symmetry and quasiperiodic reflection arrangements [3].

X-ray diffraction data (0.85 Å resolution) from a single crystal of PD4-phase was collected using Oxford XcaliburTM 3 diffractometer. The unit cell parameters for this structure are  $a = 101.302(9)$  Å,  $b = 32.102(2)$  Å,  $c = 4.1803(4)$  Å.

The phase problem for the approximant PD4 was solved by direct methods in the non-centrosymmetric space group  $Bbm2$ . The refinement of the structure using the deduced model is in progress.

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**Keywords:** quasicrystal crystallography, direct methods, structure simulation

**P.22.02.1***Acta Cryst.* (2005). **A61**, C475-C476**Diffuse Scattering from Composite Crystals Containing Stacking Faults**Mitsuko Onoda<sup>a</sup>, Yuzuru Miyazaki<sup>b</sup>, Tsuyosi Kajitani<sup>b</sup>, Yoshito Gotoh<sup>c</sup>, <sup>a</sup>AML, NIMS, Tsukuba, Japan. <sup>b</sup>Dept. of Applied Physics, Tohoku University, Sendai, Japan. <sup>c</sup>AIST, Tsukuba, Japan. E-mail: onoda.mitsuko@nims.go.jp

The compound (Ca<sub>2</sub>CoO<sub>3</sub>)<sub>0.62</sub>CoO<sub>2</sub>, a potential candidate for a thermoelectric material, has been revealed to be a misfit-layered compound, which consists of two interpenetrating monoclinic subsystems, CoO<sub>2</sub>-part of CdI<sub>2</sub>-type sandwiches and Ca<sub>2</sub>CoO<sub>3</sub>-part of ordered three-atom-thick NaCl-type blocks [1]. Sometimes reflections with specific indices showed rather large line-widths in powder diffraction patterns of the compound. They suggested occurrence of stacking disorder in one subsystem or in two subsystems. In some powder patterns of misfit-layered sulfides such as (PbS)<sub>1.12</sub>VS<sub>2</sub>, composed of VS<sub>2</sub> sandwiches and two-atom-thick NaCl-type PbS