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**Keywords:** chirality, chiral, borate

## MS87.P16

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### Recent developments in the SuperNova dual source micro-focus diffractometer.

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The introduction of the **SuperNova** in late 2008 being the first dual wavelength micro-focus diffractometer gave a tremendous boost to the capabilities and throughput for in-house crystallographic research. The combination of the high intensity Cu and Mo micro-focus sources (50W) co-mounted along with a 135mm Atlas CCD detector allows for true flexibility on a per-experiment basis.

Continued research and development in core technologies used in the **SuperNova** means that key aspects of the instrument have been updated and improved since its initial launch. Here we would like to highlight the overall benefits of these evolutionary changes and refinements in both hardware and software by presenting a number of absolute configuration determination studies. The examples presented include a broad spectrum of materials with greatly varying anomalous signal, including the first, purely hydrocarbon based molecule's experimental chirality determination using Bayesian methods.

**Keywords:** absolute configuration determination

## MS87.P17

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### A three-dimensional framework structure of vanadium and 1,2,4,5-benzenetetracarboxylate

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Blue block crystals were obtained from the hydrothermal synthesis in aqueous solution of vanadium pentoxide, titanium tetraisopropoxide and 1,2,4,5-benzenetetracarboxylic acid in the molar ratio 1:2:2 at 150°C for 4 days, and structurally analyzed by single crystal X-ray diffraction. The interest in solving this structure is the difficulty in defining the correct space group. The unit cell parameters are consistent with a tetragonal unit cell, yet the systematic absence data cannot unambiguously identify an appropriate space group.

Assuming the structure to be orthorhombic with *a* and *b* approximately equivalent provides for the space group  $P2_12_12_1$  with  $a = 11.1302(6)$ ,  $b = 11.1307(6)$ ,  $c = 13.4114(7)$  Å,  $Z = 4$  and  $V = 1660.457(15)$  Å<sup>3</sup>. To solve the structure, both racemic and pseudo merohedral twin law were applied; the Flack parameter for the current refinement is 0.20(8), which indicates inversion twinning. However, the best model obtained so far refines to give  $R_1 = 0.1286$ , and further investigation is needed. The structure contains two crystallographically-independent vanadium atoms in octahedral geometry coordinated by four carboxylate-oxygen atoms from fully deprotonated 1,2,4,5-benzenecarboxylate ligands in the equatorial positions and two aqua ligands occupying the axial positions generate the three dimensional framework structure.

**Keywords:** pseudo merohedral twin, vanadium carboxylate, framework structure

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### Synthesis and structural characterization of two new isoindol derivatives

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Isoindol derivatives are versatile intermediates for the synthesis of a variety of bioactive compounds, and possess a broad spectrum of biological activity such as insecticidal, antibacterial, antibiotic, antitumor, antifungal, psychotherapeutic, antihypertensive, HIV and other activities. In this study, two new derivatives of Isoindol, N-(1,3-dioxo-1,3,3a,4,7,7a-hexahydro-2H-4,7-methanoisoindol-2-yl)-2,2-dimethylpropanamide and 4,8-Methano-3-(2,5-dimethoxyphenyl)-6-(methylamino)4,4a,8,exo-8a-tetrahydro-exo-3aH isoxazolo [5,4-f]isoindole-5,7(6H,7aH)-endo-dione, have been synthesized and characterized by elemental analysis, FT-IR, UV-Vis and 1H NMR spectroscopy. The crystal structure of this compounds has been determined by single crystal diffraction method. The first compound (C<sub>14</sub>H<sub>18</sub>N<sub>2</sub>O<sub>3</sub>), which is an oxalamide derivative, crystallized in the orthorhombic system with space group Pca21,  $a = 24.038$  (2) Å,  $b = 9.3387$  (7) Å,  $c = 12.3004$  (9) Å,  $Z = 8$ . The final R value is 0.0066 for 5227 measured reflections. The compound has two molecules in the asymmetric unit, is a chiral compound with 8 stereogenic center. This compound occurs non-centrosymmetric dimeric structure with N-H...O type H-bond. The crystal structure of the compound is stabilized by intermolecular N – H...O type and intramolecular N – H...O, C – H...O type H-bonds, also C – O...pi weak interactions[1,2]. The second compound (C<sub>19</sub>H<sub>20</sub>N<sub>2</sub>O<sub>5</sub>) is an isoxazoline derivative, crystallized in the monoclinic system with space group P21/n,  $a = 9.7183$  (1)Å,  $b = 13.1735$  (2) Å,  $c = 13.2880$  (2)Å,  $\beta = 94.058$  (1)°,  $Z = 4$ . The final R value is 0.0036 for 3404 reflections. The molecule adopts Z-configuration. The structure is a chiral compound with 4 stereogenic center. Molecule occurs the fusion of the isoxazoline ring and dicarboximide onto norbornane unit. The crystal structure is stabilized by C – H...O type intra-molecular, C – H...N and C – H...O type inter-molecular interactions. In the crystal structure intermolecular N – H...N and C – H...O hydrogen bonds link the molecules to form an infinite one dimensional polymeric chain along b-axis. Crystallographic results are consistent with the literature [3], [4].

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**Keywords:** crystal , derivative, spectroscopy

## MS88.P01

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### Modulated *post - simple cubic* structures in compressed P and Ca: electronic origin

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Recent high-pressure x-ray diffraction studies revealed unusual complex structures in phosphorus and calcium that follow the simple cubic structure at pressure above 1 Mbar [1], [2]. These *post - simple cubic* (*post-sc*) phases P-IV and Ca-IV have close structural relation to the simple cubic via orthorhombic or tetragonal distortion of the basic cell and formation of the superlattice in one direction.

For the phase P-IV the basic cell is base-centered orthorhombic, *oC2*, with a incommensurate modulation defined by a wave vector 0.267. We consider a commensurate approximant with a 11-fold supercell along the c-axis and a modulation wave vector equal 3/11. The phase Ca-IV, tP8, has a tetragonally distorted cubic cell with a commensurate 4-fold supercell along the c-axis. P-IV and Ca-IV have some common structural features in the formation of the *post-sc* phases that implies some common physical reasons for such complexity. We consider configurations of Brillouin zones and the Fermi sphere within a nearly-free-electron model in order to analyze the importance of these configurations for the crystal structure energy [3] containing two main contributions: electrostatic (Ewald) and electronic (band structure) energies. The latter can be lowered due to a formation of a Brillouin zone plane and an opening of an energy gap at this plane.

Under pressure, the band structure energy part becomes more important leading to a formation of complex low-symmetry structures [4], [5]. The stability of the *post-sc* phases in P and Ca is attributed to the lowering of the electronic band structure energy due to Brillouin zone – Fermi surface interactions.

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**Keywords:** modulated structure, crystal stability, band structure

## MS88.P02

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### Incommensurate electronic motifs in tetramethylpyrene polyiodide crystals

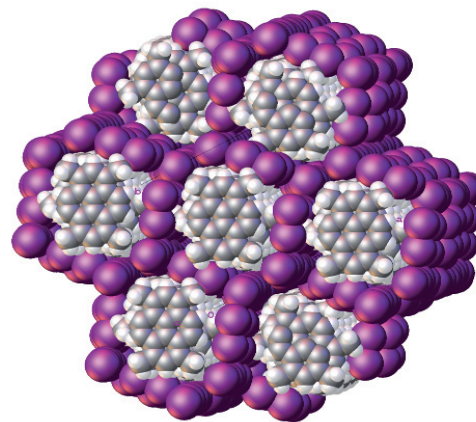
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Co-crystallization of neutral tetramethylpyrene (TMP) and elementary iodine yields black needles with metallic shine. The needles produce discrete X-ray diffraction maxima in two dimensions

and practically unresolvable quasi-continuous diffraction in third dimension.

After several attempts, a bunch of crystals was isolated that provided discrete diffraction in third dimension but with a very long identity period of  $c = 86.9 \text{ \AA}$ . The experimental data were collected using Cu radiation and the structure was successively solved to  $R=6.0 \%$ .

The structure consists of oxidized TMP molecules forming cation-radical stacks along z direction. The stacks are completely separated from each other being wrapped in rippled two-dimensional anionic polyiodide sheets.



The longest period of the unit cell corresponds to 26 molecular units of TMP with an average separation of 3.34 Å. In the embodying polyiodide substructure, the same period corresponds to 8 tri-iodide ions and 2 di-iodine molecules alternatively forming linear chains along the TMP stacks (these chains are cross-linked by extra di-iodine molecules).

The complex stoichiometry of the structure, corresponding to +0.615 charge per TMP unit and requiring di-iodine “spacers” to commensurate cationic and anionic substructures, exemplify it as a “degenerated” composite crystal.

It is important to point out that TMP cannot be oxidized by elementary iodine in solution. Therefore, the formation of the ion-radical salt is entirely due to the free energy of electronic delocalization in the crystals.

**Keywords:** incommensurate\_structures, composite\_crystals, ion-radical\_salts

## MS88.P03

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### Incommensurate BiMO<sub>3</sub> Perovskites: Bi<sub>2</sub>Mn<sub>2/3</sub>M<sub>2/3</sub>Ni<sub>2/3</sub>O<sub>6</sub> and Bi<sub>2</sub>M'M'O<sub>6</sub>

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Materials in which dielectric and magnetic properties are coupled are of interest for multiple state memory and information storage applications, and fundamentally in terms of the mechanisms for coupling these properties. Single phase materials which display these properties are important as detailed studies of structural response are possible to permit the identification of the underlying mechanisms for this behaviour. In our search for ferromagnetic ferroelectrics based on Bi<sup>3+</sup>, we have isolated new phases many of which are incommensurately modulated.