

single crystal X-ray diffraction and by DFT calculations. The X-ray crystal structure of $C_{10}H_{12}S_3$ was studied both at 295 K and at 100 K. The Space group is $C2/m$ at 295 K, which is transformed to $P2_1/n$ at 100 K. The mirror symmetry perpendicular to 2-fold axis disappears at low temperature. Such reduction of symmetry elements was also found in a previous study on the 2,5-dimethyl-6a-thiathiophene[1]. The bond lengths of two S-S bonds are crystallographically the same [2.3341(8)Å] at 295K, but are significantly different [2.3274(5) and 2.3393(5) Å] at low temperature. The experimental electron density is produced according to multipole model. The theoretical electron density is calculated by DFT calculation, where the basis set of 6-31G** is used for all the atoms but an additional diffuse function is added for S atom. Results on the electron density distribution will be presented in terms of deformation density, Laplacian maps and the topological properties. Sulfur K-edge X-ray absorption spectroscopy (XAS) is also undertaken to further our knowledge on the electronic configuration of S atom.

[1] Wang Y., Wu S. Y., Cheng A. C., *Acta Cryst.* 1990, **B46**, 850.

Keywords: charge density, XAS, DFT calculations

P.14.07.13

Acta Cryst. (2005). A61, C428

An Examination of All the Inter-ion Interactions in $(CH_3)_2N(H)CH_2CH_2N(H)(CH_3)_2$ (SCN)₂

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N,N,N',N' -tetramethylethylenediammonium $(CH_3)_2N(H)CH_2CH_2N(H)(CH_3)_2^+$ forms a di-thiocyanate hydrogen bonded salt in space group $P-1$ with $Z = 1$. X-ray data were collected on a Saturn 70 with Mo- K_α radiation to $2\theta(\max) = 105^\circ$. The multipole refinement was performed via XD [1] and all the topological interactions were then investigated. Of the 8 unique C-H hydrogen atoms, all but one forms significant interactions to the thiocyanate anion. These interactions constitute 4 (C-H...S), 2(C-H...C), 2(C-H... $\pi(C\equiv N)$), and 2(C-H...N) with two bifurcated C-H interactions. All 10 interactions satisfy all the eight of Koch & Popelier's criteria [2] for a weak interaction, though one interaction of a bifurcated pair is only marginally satisfactory. The N-H...N classical hydrogen bond is found to have weakened in the crystal when compared with the theoretically calculated values for an isolated ion pair.

[1] Koritsanszky T. S., Howard S., Macchi P., Gatti, C., Farrugia L. J., Mallinson P. R., Volkov A., Su Z., Richter T., Hansen N. K., *XD (version 4.10, July): A computer program package for multipole refinement and analysis of electron densities from diffraction data*, 2003. [2] a) Koch U., Popelier P. L. A., *J. Phys. Chem.* 1995, **99**, 9747; b) Popelier P., *Atoms in Molecules*, Prentice Hall, UK, 2000, 151.

Keywords: multipole refinement, hydrogen bonding, topological properties of charge distribution

P.14.07.14

Acta Cryst. (2005). A61, C428

Rb_4C_{60} Equation of States and Electronic Density Study by Compton Scattering

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In the family of compounds A_nC_{60} ($A=K, Rb$ and Cs ; $n=1,3,4$ and 6), Rb_4C_{60} exhibits an unusual behavior: it is a non-magnetic insulator at ambient pressure, whereas expected as a conductor, even a superconductor (cf. Rb_3C_{60}), by theory [1]. Furthermore Rb_4C_{60} undergo an insulator to metal transition under pressure around 0.8 GPa, as observed by NMR study of Rb_4C_{60} [2].

We have performed combined Compton scattering (ID15B) and diffraction experiments at ESRF (ID-30), at pressures below and

above the insulator-metal transition. Our diffraction experiment exhibits, for the first time, an abrupt jump in compressibility between 0.5 GPa and 0.8 GPa. We attributed this jump to a structural phase transition preserving the initial tetragonal symmetry [3]. By *ab-initio* calculations we reproduced the experimental phase transition and studied the pressure dependence of internal coordinates. Going further, the comparison with *ab-initio* LDA calculations allowed us to quantitatively evaluate contributions due to contraction of the unit cell as well as an unexpected contraction of the C_{60} molecule itself, evidenced by Compton measurements. In fact, this C_{60} molecule contraction leads to a major effect on electronic density of Rb_4C_{60} compound [5].

[1] Erwin S.C., *Buckminsterfullerenes*, ed. W. E. Billups M. A. Ciufolini, VCH, New York, 1993, 217. [2] Kerkoud R. *et al.*, *J. Phys. Solids*, 1996, **57**, 143. [3] Sabouri-Dodaran A.A. *et al.*, *Phys. Rev.*, 2004, **B70**, 174114.

Keywords: structural change, Compton scattering, high-pressure X-ray diffraction

P.14.07.15

Acta Cryst. (2005). A61, C428

An Examination of All the Inter-ion Interactions in Tetraphenylphosphonium Squarate

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The tetraphenylphosphonium squarate salt crystallises with a hydrogen bonded dimeric squarate anion in space group $P2_1/n$ with $Z = 4$. X-ray data were collected on a Saturn 70 with Mo- K_α radiation to $2\theta(\max) = 76^\circ$ at $-153^\circ C$. The multipole refinement was performed with XD [1] and all the topological interactions were then investigated. Currently of the 20 unique C-H hydrogen atoms, all but two form significant interactions either to the squarate ion (C-H...O interactions) or among the phenyl groups C-H...H-C, or C-H... π (Ph). The two remaining 'non-interacting' (C)H atoms are adjacent *ortho* hydrogen atoms, that look as though there ought to be an intramolecular H...H interaction though this has not yet been fully characterized. The remaining interactions satisfy all the eight of Koch & Popelier's criteria [2] for a weak interaction. The O-H...O classical hydrogen bond of the squarate is found to have weakened in the crystal when compared with the theoretically calculated values for an isolated dimeric anion

[1] Koritsanszky T. S., Howard S., Macchi P., Gatti, C., Farrugia L.J., Mallinson P.R., Volkov A., Su Z., Richter T., Hansen N. K., *XD (version 4.10, July): A computer program package for multipole refinement and analysis of electron densities from diffraction data*, 2003. [2] a) Koch U., Popelier P. L. A., *J. Phys. Chem.*, 1995, **99**, 9747; b) Popelier, P., *Atoms in Molecules*, Prentice Hall, UK, 2000, 151.

Keywords: multipole refinement, hydrogen bonding, topological properties of charge distribution

P.14.07.16

Acta Cryst. (2005). A61, C428-C429

X-ray Charge Density of a New Magnetic Metal Organic Framework, $Mn_3(C_8O_4H_4)_3(C_5H_{11}ON)_2$

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A new magnetic metal organic framework material has been synthesized, $Mn_3(C_8O_4H_4)_3(C_5H_{11}ON)_2$.¹ Magnetic susceptibility measurements from 2 to 400 K reveal anti-ferromagnetic ordering at ~4 K and a total magnetic moment of 6.0 μ_B . The magnetic phase transition is confirmed by heat capacity data (2 - 300 K). The crystal structure is studied by conventional single crystal X-ray diffraction data at 300, 275, 250, 225, 200, 175, 150, 125 and 100 K, and synchrotron data at 20 K. The electron distributions around the two unique Mn centers are different, and both have substantial anisotropy. Orbital population analysis reveals large electron donation (1.7 e) to each Mn atom and the maximum possible number of un-paired electrons is 3.2 for both