

B61, 46-57.

Keywords: hydrogen bonding, cocrystals, intermolecular packing

## P06.10.52

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### Crystal structure of (4*Z*)-2-phenyl-4-(3,4,5-trimethoxybenzylidene)-1,3-oxazol-5(4*H*)-one

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1,3-oxazole is a very useful intermediate synthetic organic material for the synthesis of imidazole that possess a wide spectrum of biological activities such as herbicides, antibacterial, antifungal, etc. In addition, the oxazole itself is also found to possess antibacterial and antifungal activities. In view of this, the crystal structure of title compound has been determined. The yellow colored, block shaped crystals of size  $0.3 \times 0.3 \times 0.2 \text{ mm}^3$  were grown by slow evaporation from the benzene solution. The compound crystallizes in triclinic system having space group *P*-1. The crystal data are:  $a = 7.3732(8) \text{ \AA}$ ,  $b = 15.7823(17) \text{ \AA}$ ,  $c = 8.1325(8) \text{ \AA}$ ,  $\alpha = 62.085(7)^\circ$ ,  $\beta = 82.506(7)^\circ$ ,  $\gamma = 91.954(6)^\circ$ ,  $Z = 2$ ,  $\mu = 0.100 \text{ mm}^{-1}$ ,  $D_x = 1.367 \text{ Mg-m}^{-3}$ . Intensity data on BRUKER AXS KAPPA APEX2 CCD diffractometer are collected in  $\omega$  and  $\phi$  scan with  $\theta$  ranging from  $2.53$  to  $25.0^\circ$  and 2885 unique reflections are recorded. The structure is solved by SIR92 program and refined by SHELXL-97 program to final R-value of 0.0597 for 2114 reflections with  $I > 2\sigma(I)$ . In the structure, all the three rings are planar within themselves, and the benzene ring and benzylidene moiety making a dihedral angle of  $11.23(15)$  and  $3.19(14)^\circ$  respectively. An intermolecular C-H...O interaction forms a dimer that exhibit  $R^2_2(14)$  graph-set motif. In addition crystal structure is stabilized by C-H... $\pi$  and  $\pi$ - $\pi$  stacking interactions.

Keywords: single-crystal X-ray diffraction, organic crystals, hydrogen bonding

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### Two proton transfer compounds from benzene-1,2,4,5-tetracarboxylic acid and 1,10-phenanthroline

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Recently, there has been considerable interest in proton transfer systems and their structures. Two proton transfer compounds (phenH)(btcH<sub>3</sub>) · H<sub>2</sub>O, **1**, and (phenH)<sub>2</sub>(btcH<sub>2</sub>) · btcH<sub>4</sub>, **2**, were obtained by the reaction of benzene-1,2,4,5-tetracarboxylic acid (btcH<sub>4</sub>) with 1,10-phenanthroline (phen) in 1:1 and 1:2 molar ratio, respectively. The characterizations were performed using IR, <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy and single-crystal X-ray diffraction. The compound **1** is crystallized in triclinic system and *P*1<sup>-</sup> space group with following

cell parameters:  $a = 7.8529(5) \text{ \AA}$ ,  $b = 9.8333(6) \text{ \AA}$ ,  $c = 12.2847(7) \text{ \AA}$ ,  $\alpha = 94.5910(10)^\circ$ ,  $\beta = 91.3670(10)^\circ$ ,  $\gamma = 94.7300(10)^\circ$ ,  $Z = 2$ . The final *R* value of **1** is 0.0354 for 3627 total reflection. The compound **2** is crystallized in the space group *P*2<sub>1</sub>/*c* of the monoclinic system, and contains two molecules per unit cell. The structure has been refined to a final value for the crystallographic *R* factor of 0.0364 based on 4881 reflections. The unit cell parameters are:  $a = 11.9154(6) \text{ \AA}$ ,  $b = 13.5560(6) \text{ \AA}$ ,  $c = 12.2030(6) \text{ \AA}$  and  $\beta = 110.4880(10)^\circ$ . The ranges of the D-H...A angles and the H...A and D...A distances indicate the presence of strong hydrogen bonding in both compounds. Hydrogen bonds with D...A distances ranging from 2.4331(15) to 3.4766(19) Å, are observed in the crystal structure of **1** whereas, the range of D...A distances in **2** is between 2.4472(13) to 3.4287(18) Å. In both structures, **1** and **2**, anionic and cationic units have been arranged in the lattice in a parallel manner connect the various components into self-associated supramolecular structures and providing considerable  $\pi$ - $\pi$  stacking between (phenH)<sup>+</sup> rings. The centroid distances between the planes are 3.4779(9) and 3.8707(8) Å, respectively.

Keywords: crystal structures, hydrogen bonding, supramolecular assemblies

## P06.10.54

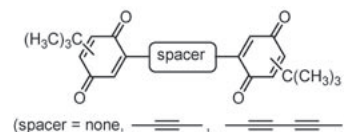
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### X-ray structures of quinone dimers linked either directly or through acetylene spacers

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X-ray structures of two directly-linked quinone dimers have been investigated. Although the positions of tert-butyl substituents differed, their X-ray structures were significantly close. The carbonyl oxygen atoms underwent van der Waals contact in an intramolecular manner, and quinone moieties were distorted into the boat shape in common. As MO calculations indicated the quinone moiety became flexible when quinones were directly linked, the origins of the boat forms should be attributed to the intrinsic flexibility of the molecular skeleton as well as the packing demand. In contrast, quinone moieties of quinone dimers bearing acetylene and diacetylene spacers have been found to be planar. This may be arisen from no intramolecular contact of oxygen atoms by introduction of the acetylene spacers. Flexibility of the molecular skeleton of these molecules will also be discussed.



Keywords: inter- and intramolecular interactions, packing, isomers

## P06.10.55

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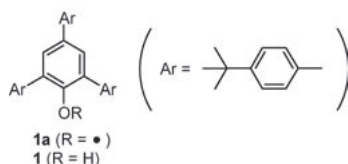
### Homogeneous and heterogeneous mixed crystals composed of phenoxy radical and phenol

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While 2,4,6-tris(4-tert-butylphenyl)phenoxy radical (1a) itself gave an amorphous solid (A), in the presence of 2,4,6-tris(4-tert-butylphenyl)phenol (1) it yielded mixed crystals B (1a:1 = 60:40) and C (1a:1 = 30:70) from solution. Unlike C, B was apparently heterogeneous: it had purple inside and colorless outside, indicating that 1a was significantly concentrated at inside and virtually absent at outside. The thermal behavior of B and C was not identical but close to that of A and 1, respectively. Single-crystal X-ray diffraction study revealed crystal structures of B, C, and 1 were almost identical. In spite of the higher 1a content, C-O bond distances in B were found to be essentially longer than those of C and a little shorter than those of 1. These observations likely indicate that B was composed of three layers: amorphous 1a (inside), mixed crystal of 1a and 1 (boundary) and 1 (outside).



Keywords: mixed crystals, thermal properties, isomorphous replacement

## P06.10.56

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### Crystal structure of connatin, a strong radical scavenger from *Lyophyllum connatum*

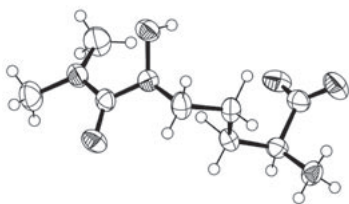
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In our continuing search for free radical scavengers from mushroom, we have isolated ergothioneine,  $\beta$ -hydroxyergothioneine, N-hydroxy-N',N'-dimethylurea, and connatin (N-hydroxy-N',N'-dimethylcitrulline) from the mushroom *Lyophyllum connatum*. We determined the crystal structure of  $\beta$ -hydroxyergothioneine [1], and report here the crystal structure of connatin (Figure 1). All the compounds displayed the ability to scavenge free radicals, based on a 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging assay. We found that ergothioneine and  $\beta$ -hydroxyergothioneine scavenged the radical in very fast manner (reaction almost completed less than 10 min in a UV-cell), therefore they can be classified as fast-reacting scavengers. On the other hand, N-hydroxy-N',N'-dimethylurea and connatin were slow reacting scavengers (for completion of the reaction required 2h). Furthermore, we also isolated N,N-dimethylcitrulline which is considered to be a plausible precursor of connatin. Crystallization of N,N-dimethylcitrulline is under progress.

[1] C. Kimura et al., *Biosci. Biotechnol. Biochem.* 69, 37(2005).

Keywords: connatin, radical scavenger, *Lyophyllum connatum*



## P07.01.01

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### Metal complexes of di(thiophenyl)ethenes: Synthesis, structure and bonding

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Di(thiophenyl)ethene ligands are of interest as prototypes for 'molecular switches' in future molecular electronic applications [1,2]. In this contribution we describe the synthesis of metal complexes of a di(thiophenyl)ethene ligand in the 'open' form, photolysis of which reversibly gives the 'closed' form (see Figure). Both open and closed forms have been characterised using single crystal X-ray diffraction, in addition to density functional theory and a variety of spectroscopic techniques.

Keywords: molecular electronics, photochemistry, thiophenes

## P07.01.02

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### Recent results from a long-term academic collaboration

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Several industrial and academic collaborations are underway at Newcastle Crystallography. Among the latter, the most fruitful has so far been the one with the group of Prof. R. E. Mulvey at the University of Strathclyde. About 70 crystal structures of air-sensitive, organometallic compounds were determined in Newcastle within the last 14 months, as part of this collaboration, with a good turnout of high-impact publications. The highlights from the published work, presented in this poster, show the systematic contribution of the structural investigation, not only as the ultimate means towards the identification of expected and unexpected products, but also as an effective tool for underpinning reaction pathways and intermediates.

Keywords: crystallography, metallorganic complexation, structural investigations

## P07.01.03

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### Structural study of an iron oxalate and a copper rubeanate layer on an ultra-smooth sapphire c-face

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The conductivity by proton in bulk such as a copper rubeanate complex has been studied [1]. Such complexes which consist of thin layers can be useful for a part of a fuel cell. Our purpose in this study