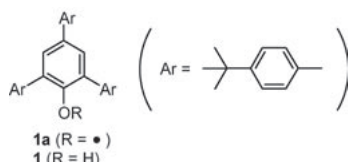


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While 2,4,6-tris(4-tert-butylphenyl)phenoxy radical (1a) itself gave an amorphous solid (A), in the presence of 2,4,6-tris(4-tert-butylphenyl)phenol (1) it yielded mixed crystals B (1a:1 = 60:40) and C (1a:1 = 30:70) from solution. Unlike C, B was 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

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

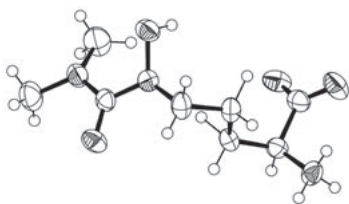
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In our continuing search for free radical scavengers from mushroom, we have isolated ergothioneine, β -hydroxyergothioneine, N-hydroxy-N',N'-dimethylurea, and connatin (N-hydroxy-N',N'-dimethylcitrulline) from the mushroom *Lyophyllum connatum*. We determined the crystal structure of β -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 β -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*



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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

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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

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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