## MS18-P04

### High-pressure neutron measurements of the highly-polymorphic 'ROY'

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Where neutron-powder measurements are needed to discriminate between similar-*Z* elements, identify magnetic behaviour, or simply locate hydrogen atoms, large sample volumes are generally required for a successful measurement. This might appear problematic for high-pressure experiments where small sample volumes are necessary to access higher pressures. However, the large-volume Paris-Edinburgh press on the high-pressure PEARL beamline at ISIS, UK, allows us to make such measurements—striking a balance between sample quantity and accessible pressure.[1]

5-methyl-2-[(2-nitrophenyl)amino]-3-thiophenecarbonitrile is one of the most polymorphic organic materials known in the Cambridge Structural Database. It is more colloquially known as 'ROY', owing to the red, orange, and yellow polymorphs that it forms, all occurring under ambient conditions.[2] In this presentation, a recent compression study on ROY and its behaviour up to 9.3 GPa will be reported.[3] We show that the quality of PEARL data obtained, and careful DFT-assisted guidance of the refinement procedure, produce usable structural models from a weakly-scattering, hydrogenated sample. We discuss its structural response in the context of lattice- and intermolecular-energy calculations.

References:

[1] Bull, C. L. et al. (2016), High Pressure Res., 36(4), 493-511.
[2] Yu, L., (2010), Acc. Chem. Res., 43, 1257-1266.
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**Keywords: pressure, neutron, polymorphism**

## MS18-P05

### A crystallographic study of L-threonine to extremely high pressures

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Our work focuses on pushing the boundaries of high-pressure crystallography on soft molecular systems. We have successfully determined crystal structures of the essential amino acid, L-threonine ($C_4H_9NO_3$) to 22 GPa.

L-threonine is zwitterionic in the solid-state, comprising of an ammonium cation and a carboxylate anion. Each molecule is connected by four H-bonds, resulting in a $P\ 2_12_12_1$ (Z=4) crystal structure with zig-zag chains formed along the long crystallographic axis, linked by carboxylate and hydroxyl interactions. Adjacent chains are interconnected by off-axis H-bonds between nitrile and carboxylate groups to form pleated layers. Single-crystal X-ray diffraction experiments were performed with synchrotron radiation in series using the diamond anvil cell and neon pressure transmitting medium. A reversible pressure induced phase transition going from the $P\ 2_12_12_1$ (Z'=1) crystal structure to $P\ 2_1$ (Z'=2) was observed at 18 GPa. All H-bonds shorten resulting in conformational changes of the carboxylate, hydroxyl and methyl moieties. Structure refinements were validated against periodic-DFT (CASTEP) optimisations and prior works.[1] Crystal lattice energies and the most energetically significant H-bond interactions for all structures were examined by the PIXEL method.[2]

References:

[1] R. O. Holanda, J. A. Lima, P. T. C. Freire, F. E. A. Melo, J. Mendes Filho and A. Polian, Journal of Molecular Structure 2015, 1092, 160-165.
[2] A. Gavezzotti in Bonding in Organic Molecules and Condensed Phases. The Role of Repulsions, (Ed. J. J. Novoa), The Royal Society of Chemistry, UK, 2017.

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