

**MS42-P1** **Structure solution and refinement of "Small" molecules** Yael Diskin-Posner *Unit of Chemical Research Support, Kimmelman Building, The Weizmann Institute of Science, Rehovot 76100, Israel.*  
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Single crystal X-ray analysis of small molecule today is not considered as challenging but a routine. However, it is very hard to publish a new compound without its crystal structure. Usually most of the small crystal structures contain about 20-50 atoms and they are relatively easy to solve and refine, when the crystals are big enough and ordered. Rarely, there are structures that contain a couple of hundred and more atoms in the asymmetric unit cell and make data collection, solving the structure and refinement extremely difficult. Furthermore during validation these structures fall under the restricting criteria of small molecules. Two different examples will be given.

A new lanthanide-organic framework (LOF) based on a long (21.2Å) and rigid bid-Gd complex synthon (complex 1) and carbonate anion spacers was discovered. Auxiliary Na<sup>+</sup> ions and water molecules are positioned around the carbonate ions and connect between the networks. Overall, in this new LOF the synthons are packed in criss-cross fashion and tethered by carbonate spacers, creating a crystal lattice perforated by 10.6 × 15.6Å and 18.6 × 15.6Å wide-open channels.

Copper(I) complexes with perfluorinated tails such as (Cu<sup>I</sup>L<sup>2</sup>AcN)-BF<sub>4</sub> where L<sup>2</sup>=6,6''-bis(perfluorohexyl)-2,2':6',2''-terpyridine and acetonitrile were synthesized and its structure was elucidated. It turned out that (Cu<sup>I</sup>L<sup>2</sup>AcN)-BF<sub>4</sub> crystallized in a low symmetry space group *P*<sub>1</sub>, with a very large unit cell that required synchrotron radiation to locate the side chains conformations.

**MS42-P2** **Copper(I) Complexes of Bipyridine and Terpyridine with Fluorous Tails and the Formation of Crystalline Materials with Fluorous Layers.** Yael Diskin-Posner,<sup>a</sup> Vedichi Madhu,<sup>b</sup> and Ronny Neumann<sup>b</sup>  
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Copper(I) complexes with perfluorinated tails such as (Cu<sup>I</sup>L<sup>2</sup>AcN)BF<sub>4</sub> and (Cu<sup>I</sup>L<sup>3</sup>AcN)-BF<sub>4</sub> where L<sup>2</sup>=6,6''-bis(perfluorohexyl)-2,2':6',2''-terpyridine and L<sup>3</sup>=6,6''-bis(2,2,3,3,4,4,5,5,6,6,7,7,7-tridecafluoroheptyl)-2,2'-bipyridine and L<sup>3</sup>=6,6''-bis(2,2,3,3,4,4,5,5,6,6,7,7,7-tridecafluoroheptyl)-2,2'-bipyridine and AcN = acetonitrile were synthesized their rather unique structures were elucidated. (Cu<sup>I</sup>L<sup>2</sup>AcN)BF<sub>4</sub> crystallizes in a low symmetry space group *P*<sub>1</sub>, with a very large unit cell that required synchrotron radiation to locate all atoms. The molecular structure of (Cu<sup>I</sup>L<sup>2</sup>AcN)BF<sub>4</sub> has an asymmetric tetrahedrally disordered square-planar coordination sphere around Cu<sup>I</sup>. The crystal packing of (Cu<sup>I</sup>L<sup>2</sup>AcN)BF<sub>4</sub> has a layered structure with clear segregation between the terpyridine moieties and the fluoros chains. The terpyridine units are arranged in a "back to back" fashion and tilted 20-25° relative to the interlayer plane. The overall layered structure is staggered with offsets of approximately 4L between the layers.

The structure of (Cu<sup>I</sup>L<sup>3</sup>AcN)BF<sub>4</sub> features a Y-shaped trigonal planar coordination sphere around copper(I). The crystal structure of (Cu<sup>I</sup>L<sup>3</sup>AcN)BF<sub>4</sub> shows a "herring bone" packing arrangement with an approximately 96° angle in the CubipyAcN units, which looks like an accordion fluoros layer.

**Keywords: copper coordination compound; layered structures; Fluorous compounds**