FA4-MS33-T04

The construction of open frameworks based on methanetriacetic acid: new objects with an old ligand. Catalina Ruiz-Pérez,[a] Laura Cañadillas-Delgado,[a] Tomás Martín,[b] Oscar Fabelo,[a] Jorge Pasán,[a] Mariadel Déniz,[a] Pau Díaz-Gallifa,[a] Carla Martínez,[a] Wilson Rodríguez,[a] Rafael Pino,[a] Francesc Lloret,[c] Miguel Julve[c]. [a]*Lab. de Rayos X y Materiales Moleculares (MATMOL), Universidad de La Laguna, Spain.* [b]*Instituto de Productos Naturales y Agrobiología (C.S.I.C.), Spain.* [c]*ICMol, Universitat de València, Spain.*
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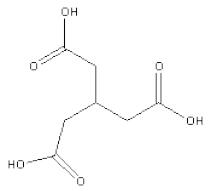

Methanetriacetic acid (H_3mta)

The preparation, X-ray crystallography and study of the properties (optical, magnetic, porosity,…) of the first examples of methanetriacetate (mta)-containing lanthanide(III) complexes of formulae $[Ln(mta)(H_2O)_3]_n \cdot 4nH_2O$ (**1**) $[Ln(mta)(H_2O)_3]_n \cdot 2nH_2O$ (**2**) and $[Ln_2(mta)_2(H_2O)_2]_n \cdot 2nH_2O$ (**3**) have been done. This tripodal ligand promotes the formation of interesting and rare 6^3 networks: a two-dimensional 6^3-**hcb** in **1**, a two-dimensional (6,3)-connected binodal $(4^3)(4^6 6^6 8^3)$-**kgd** net in **2** and a (6,6)-connected binodal **nia**-net in **3**, where the Ln(III) ions and the mta ligands act as octahedral and as trigonal prismatic nodes, respectively [1]. Concerning the magnetic properties, a relationship between the nature of the magnetic coupling and the type of bridge that links the Ln(III) centers has been proposed.

[1] Cañadillas-Delgado, L., Martín, T., Fabelo, O., Pasán, J., Delgado, F. S., Lloret, F., Julve, M., Ruiz-Pérez, C. *Chem. Eur. J.* 2010, 4037.

Keywords: crystal engineering, topology, properties of crystals

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Flurbiprofen: structure and properties of (hydroxylated) t-butylammonium salts. C. H. Schwalbe[a], M. Ramirez[a], B. R. Conway[a], C. J. Bache[a], S. J. Coles[b], P. Timmins[c]. [a]*School of Life & Health Sciences, Aston University, Birmingham B4 7ET, UK.* [b]*School of Chemistry, University of Southampton, Southampton SO17 1BJ, UK*; [c]*Bristol-Myers Squibb, Reeds Lane, Moreton CH46 1QW, UK.*
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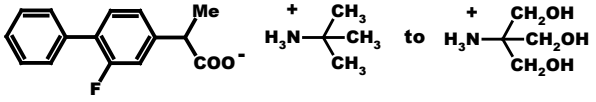
We have successively replaced CH_3 groups with CH_2OH in the t-butylammonium salt of the anti-inflammatory drug flurbiprofen (FTbut). Additional hydrogen bonding by these OH groups should affect both solubility and compactability.



In FTbut donation of hydrogen bonds from $^+NH_3$ to OCO^- forms the ladders built out of $R_4^3(10)$ rings that are common in amine carboxylates [1]. These remain unchanged after substitution with one OH group (FAmp): the OH group lacks a credible hydrogen bond acceptor and is disordered. However, a second OH group (FAmp2) changes the pattern fundamentally. Simple HNH...OCO...HNH...OCO hydrogen bonds still form a charge-assisted C(6) infinite chain traversing the entire crystal, but in addition cations deploy one NH and one OH hydrogen atom in hydrogen bonds to one anion forming a ring $[R_2^2(9)]$. The other OH group links intermolecularly to the first one while NH finds OCO^- $[R_3^2(9)]$. The third amino H atom pairs with OH as acceptor in the $R_2^2(10)$ centrosymmetric dimer. The Tris salt (FTris) exists in two polymorphs. Form II is well ordered with similar hydrogen bonds to those in FAmp2. In particular, the C(6) chain persists. Crystals of FTris form I have Z' = 2 in a triclinic cell; independent ions are related by pseudosymmetry and show disorder. Each carboxylate O atom accepts only one hydrogen bond, and the C(6) chain has disappeared. Each N atom is within 2.79-2.91 Å of four oxygen atoms but can only donate three hydrogen bonds. The twist angle in the biphenyl moiety of F is is 55° and 61° here but 44-46° in the other structures. These factors should imply increased energy for FTrisI, but it melts at a higher temperature than FTrisII. There is little difference in solubility between the two polymorphs. Despite a reasonable slip plane [2], FTrisI displays poor mechanical properties, producing weak compacts with troublesome elastic recovery. FTrisII has a slightly wider slip plane and forms strong tablets with shiny faces and excellent mechanical properties. We thank Drs. S. Callear, W. Clegg, R. Harrington, P. Horton, L. Russo and R. Stephenson for data collection, and we are grateful for use of beamline I19 at the Diamond synchrotron to collect data on FAmp and FTrisI.

[1] Lemmerer, A; Bourne, S. A.; Fernandes, M. A. *CrystEngComm*, 2008, 10, 1605-1612. [2] Sun, C.; Grant, D. J. *Pharm. Res.*, 2001, 18, 274-280.

Keywords: flurbiprofen salts, hydrogen-bond patterns, compaction of powders