

Poster Sessions

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Crystal structure of two new carbacylamidophosphates

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In recent years, carbacylamidophosphates with the C(O)NHP(O) skeleton have attracted much attention due to their properties as prodrugs [1,2], ureas inhibitors [3], and efficient ligands in coordination chemistry either as O-donor or O,O'-donor ligand [4,5]. In earlier works we considered the structural properties of some of these molecules with formula C₆H₅CONHPO(R')₂. To further investigate this area, here we have synthesized two new compounds with general formula C₆H₁₁C(O)NHP(O)R₂, R= 2-ClC₆H₄CH₂NH (**1**) and C₆H₁₁NH (**2**) and present their X-ray crystal structures.

Colorless crystals of **1** and **2** were obtained from a concentrated chloroform solution at room temperature. The compounds **1** and **2** crystallize in the triclinic and Orthorhombic systems with space groups *P*-1 and *Pmm*2₁ respectively. In the structure of compounds **1** and **2**, as in the most carbacylamidophosphates [6], the carbonyl and phosphoryl groups are in anti-positions to each other, and the bond lengths (1.226(2) and 1.4797(13) in **1**; 1.219(5) and 1.478(3) Å in **2** respectively) fall within the norms for these linkages. The dihedral angles between the PO double bond and the N-C amidic bond are -166.04(15) and 180° in **1** and **2** respectively. The phosphorus atoms have a slightly distorted tetrahedral configuration with angles in the range of 101.18(10)–119.75(8)° in **1** and 102.50(16)–116.25(10)° in **2**. As expected, the P–N_{amide} bond length (N_{amide}: the nitrogen atom of P(O)N(H)C(O) moiety) is longer than the P–N_{amine} distances (N_{amine}: the nitrogen atom of P(O)NHR moiety) and the C–N_{amide} bond is shorter than the C–N_{amine} bond lengths (1.374(2) Å for C–N_{amide} versus 1.453(2) and 1.469(2) Å for C–N_{amine} bonds in **1**). All of the P–N bonds in **1** and **2** are shorter than the typical P–N single bond length (1.77 Å) [7]. This is likely due to the electrostatic effects which overlap with P–N σ bond.

The two molecules in the unit cell of **1** are coupled into centrosymmetric dimers by two H-bonds between the phosphoryl oxygen atoms and the hydrogen atoms of the amidic groups forming an eight-membered cycle. The observed hydrogen-bonding pattern is of the DA = AD type. The dimers are linked into 1D chains along the *a* axis by means of another centrosymmetric hydrogen bonds of the DA = AD type involving the oxygen atoms of carbonyl groups and hydrogen atoms of 2-chlorobenzylamine groups (N2–H2 and N3–H3).

The O(1) oxygen atom in **2** functions as bifurcated acceptor and is involved in intermolecular hydrogen bonds with NH groups of cyclohexylamines. As a result of this type of hydrogen bonding and N(1)–H(1N)...O(2) interactions, 1D chains along the *b* axis are obtained.

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X-ray fluorescence imaging of ancient artifacts

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Synchrotron-based X-ray fluorescence imaging (XRFI) is a powerful tool for examining archeological artifacts. Many artifacts have been damaged by weathering and other processes, often to the point where letters or figures have become difficult or impossible to see by visual or topographic inspection. XRFI allows imaging of elemental concentrations, and can detect minute remaining traces of elements arising from paints, pigments and tool wear associated with the original pattern. Since X-rays penetrate a significant distance beneath the surface, XRFI can see through overlayers of paint and debris and can detect elements that have soaked or diffused into stone substrates.

We have imaged a large number of ancient Greek and Roman inscriptions on stone [1] and a variety of Mayan artifacts. Element maps have revealed significant new information, including letters outlined by traces of iron from tools and lead from pigments, and painted designs which were previously invisible due to overpainted layers. They have also proved useful in establishing inscription provenance [2]. Opportunities and challenges in applying XRFI to ancient artifacts will be discussed.

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Opacity in mosaic glass: crystals make technological history

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Opaque glass represents the first record of glass production at the end of the second millennium AD in Mesopotamian region. The opacity of glass, in most of the cases, is given by the presence of crystalline phases dispersed in the glass matrix. The crystal phases used as opacifiers changed across the centuries and different compounds were used to obtain the opaque effect and the desired nuances. Antimony-based opacifiers (lead antimonate yellow and calcium antimonate white)