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The Structure and Spectroscopy of α -Hydroxyphosphonates

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THE STRUCTURE AND SPECTROSCOPY OF α -HYDROXYPHOSPHONATES

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α -Hydroxyphosphonates are important intermediates in the preparation of various organophosphorus compounds. Methods for their preparation have been described.¹ We now report new aspects of their structure and spectroscopy. In the solid state, O,O-dimethyl α -hydroxy- α -phenyl- β -chloroethylphosphonate has been shown by X-ray diffraction studies to consist of hydrogen-bonded dimers with a hydrogen-bond length between the phosphoryl oxygen and hydroxyl hydrogen atom of 1.74 Å. The P=O bond is relatively short (1.463).

The ¹H and ¹³C nmr spectroscopy of these compounds is complicated by the presence of a chiral carbon atom in the α -position, resulting in magnetic non-equivalence of the alkoxy groups, and of the -CH₂ protons if present. Magnetic non-equivalence persists in the α -methoxy analogue, in which intramolecular or intermolecular hydrogen bonding cannot occur.

Whereas α -hydroxyphosphonates give only weak molecular ions in electron-impact mass spectrometry (due to thermal decomposition), MH⁺ ions are observed in the positive ion fast-atom bombardment mass spectra obtained using a glycerol matrix. Fragmentation of the MH⁺ ion occurs by pathways analogous to that occurring in thermal decomposition, which yields dialkyl phosphite and halogenoketone.¹ Either of these components separate as the neutral fragment, with the observed ion being the protonated form of the other.

1. Gy.Keglevich, I.Petneházy, L.Tőke and H.R.Hudson: *Phosphorus Sulfur*, 29, 341 (1987) and cited references.