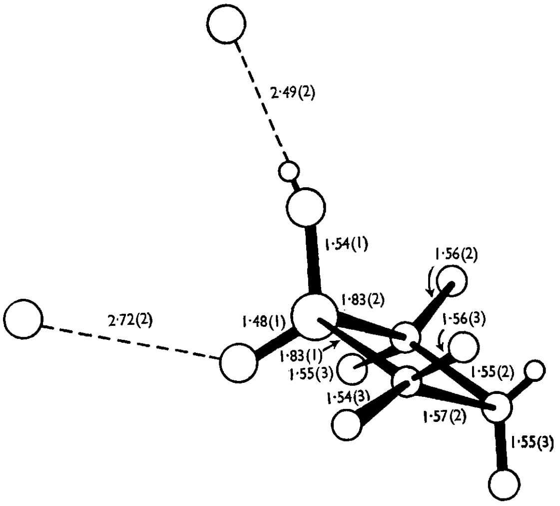
Crystal and Molecular Structure of 2,3,4-Trimethylpentane-2,4-phosphinic Acid, $PO_2C_8H_{17},H_2O$

By DUANE D. SWANK and CHARLES N. CAUGHLAN*

(*Department of Chemistry, Montana State University, Bozeman, Montana* 59715)

STRUCTURAL studies of cyclic organic phosphorus compounds are of importance in understanding the bonding at the phosphorus atom and the involvement of 3*d*-orbitals in these bonds.[1] Thus, the structural studies of several phosphinic acids have been undertaken; this is a preliminary report on one of these. Crystals of 2,3,4-trimethylpentane-2,4-phosphinic acid were furnished by Dr. Paul Haake, Wesleyan University, Connecticut.



FIGURE

The crystals are monoclinic with lattice dimensions $a = 13{\cdot}251$, $b = 6{\cdot}511$, $c = 26{\cdot}387$ Å, and $\beta = 101{\cdot}455°$. The space group is $C2/c$ with eight $PO_2C_8H_{17},H_2O$ molecules in the unit cell. The crystals were mounted in Lindemann capillaries since they tend to lose water of hydration when left in the air. Intensity data using Zr filtered Mo-radiation were collected on a General Electric XRD-5 diffractometer equipped with a single-crystal orienter and a scintillation counter. Intensities were measured using θ–2θ scan techniques, approximately 1000 independent reflections being above statistical fluctuations of the background.

The structure was determined using a combination of the symbolic-sign method and Patterson synthesis and refined using Ahmed's block-diagonal least-squares program[2] to $R = 7{\cdot}8\%$, with positional and individual anisotropic vibrational parameters. The structure with pertinent bond distances is seen in the Figure. The phosphorus bond angles are distorted from tetrahedral, the ring angles being C–P–C [82·5(5)], P–C–C [86·0(6), 85·7(6)] and C–C–C [101·0°(9)], and the others range from 106 to 121°. The phosphoryl oxygen is hydrogen-bonded to a water molecule with an O · · · O distance of 2·72(2) and the hydroxy-group is hydrogen-bonded to the screw-related water with an O · · · O distance of 2·49(2). This is an unusually short hydrogen bond.

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[1] R. F. Hudson, "Structure and Mechanism in Organo-phosphorus Chemistry", Academic Press, New York and London, 1965.

[2] F. R. Ahmed, Division of Pure Physics, National Research Council, Ottawa, Canada, 1966. Other programs used were from the M.S.U. crystallographic library.