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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1381). Services for accessing these data are described at the back of the journal.

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Ethylenebis(phosphonic acid)

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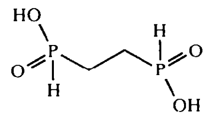
Abstract

The as yet structurally uncharacterized title compound, $C_2H_8O_4P_2$, was isolated as a by-product of the synthesis of 1,2-bis(dimethylphosphino)ethane (dmpe) from 1,2-bis(dichlorophosphino)ethane. In the crystal structure, which is characterized by strong intermolecular O—H···Osp^2 hydrogen bonds, only the R^*,S^* diastereomer is found. The molecule is located on a center of inversion intersecting the central C—C bond, thereby preserving the molecular C_i symmetry.

Comment

Phosphonic acid is known to form strong hydrogen bonds which result in enhanced viscosity with increasing concentration. In molecular crystals, hydrogen bonds are among the most important structure-determining interactions. The structure of methylenebis(phosphonic acid) (King *et al.*, 1996) is characterized by intermolecular hydrogen-bonded molecules, as is the structure of ethylenebis(phosphonic acid), (I), reported here.



(I)

In the monoclinic unit cell, the central C—C bond of ethylenebis(phosphonic acid) is located at a center of inversion and, as a result, only one-half of a symmetrically independent molecule needs to be discussed. The geometry at the P atom is very similar to that found in methylenebis(phosphonic acid), which has two independent P atoms in the asymmetric unit. Due to the presence of electron-withdrawing O atoms at the P atom, the P—C bond is shortened to 1.784 (2) Å. All H atoms have been located. An H atom is located at only one of the O atoms, and the mesomeric structure proposed for the liquid state is not present in the solid. The intermolecular distance between O atoms involved in hydrogen bonding is 2.511 (3) Å [O2—H2 0.94 (4), H2···O1^{ii} 1.58 (4) Å and O2—H2···O1^{ii} 171 (4)°; symmetry code: (ii) $\frac{1}{2}+x$, $\frac{3}{2}-y$, $z-\frac{1}{2}$]. This affords a co-operative chain of ···O=P—OH··· hydrogen bonds linking both ends

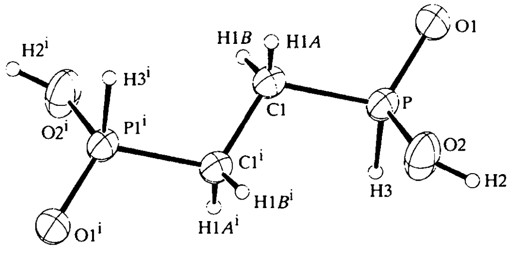


Fig. 1. View of the title compound with displacement ellipsoids at the 50% probability level. H atoms are shown as circles of an arbitrary radius. [Symmetry code: (i) $-x$, $1-y$, $1-z$.]

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of the molecule into two-dimensional layers parallel to (101). These layers have an H3$\cdots$O2^{iii} separation of 2.72 (3) Å, with a P—H3$\cdots$O2^{iii} angle of 112.5 (16)° [symmetry code: (iii) $x-1$, y, z]. A comparison with 259 P—H$\cdots$O intermolecular contacts between 2.2 and 3.5 Å contained in the Cambridge Structural Database Database (Allen & Kennard, 1993) reveals that this contact is on the borderline of the excluded region (Jeffrey & Saenger, 1991).

Experimental

Ethylenebis(phosphonic acid) was formed in small quantities during a synthesis of the chelating phosphane 1,2-bis(dimethylphosphino)ethane (Burt *et al.*, 1979) by hydrolysis of the precursor 1,2-bis(dichlorophosphino)ethane due to the presence of adventitious water. While the R^*,S^* diastereomer has crystallized here, there is no evidence that this diastereomer was formed preferentially over the R^*,R^* diastereomer, which can easily interconvert *via* the tautomeric equilibrium with 1,2-bis(dihydroxyphosphino)ethane. Crystals of the title compound were obtained from a hexane/pentane solution.

Crystal data

$C_2H_8O_4P_2$
$M_r = 158.02$
Monoclinic
$P2_1/n$
$a = 4.8912$ (6) Å
$b = 11.6238$ (12) Å
$c = 5.6581$ (3) Å
$\beta = 91.279$ (6)°
$V = 321.61$ (6) Å^3
$Z = 2$
$D_x = 1.632$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13.5$–29.9°
$\mu = 0.608$ mm^{-1}
$T = 293$ K
Tabular prism
0.53 × 0.42 × 0.25 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (Enraf–Nonius, 1995)
$T_{min} = 0.804$, $T_{max} = 0.859$
936 measured reflections
936 independent reflections

863 reflections with $I > 2\sigma(I)$
$\theta_{max} = 29.95°$
$h = -6 \rightarrow 6$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 7$
3 standard reflections frequency: 30 min intensity decay: 17%

Refinement

Refinement on F^2
$R(F) = 0.038$
$wR(F^2) = 0.118$
$S = 1.286$
936 reflections
53 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.2082P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.286$ e Å^{-3}
$\Delta\rho_{min} = -0.539$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	−0.0184 (5)	0.56349 (17)	0.5285 (4)	0.0323 (4)
O1	−0.0304 (4)	0.77739 (13)	0.3258 (3)	0.0398 (4)
O2	0.3284 (4)	0.62853 (17)	0.2035 (4)	0.0454 (5)
P1	0.03203 (11)	0.65438 (4)	0.27913 (9)	0.0279 (2)

Table 2. *Selected geometric parameters (Å, °)*

C1—C1^{i}	1.522 (4)	O2—P1	1.5501 (18)
C1—P1	1.784 (2)	O2—H2	0.94 (4)
O1—P1	1.4871 (16)	P1—H3	1.36 (3)
C1^{i}—C1—P1	112.82 (18)	O2—P1—C1	104.50 (11)
P1—O2—H2	121 (3)	O1—P1—H3	113.0 (12)
O1—P1—O2	115.63 (11)	O2—P1—H3	105.4 (12)
O1—P1—C1	113.35 (9)	C1—P1—H3	103.9 (13)

Symmetry code: (i) $-x, 1-y, 1-z$.

H atoms were located from difference Fourier synthesis and were refined isotropically.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1995). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *DATAP* (Coppens *et al.*, 1965). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*II (Johnson, 1976).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1229). Services for accessing these data are described at the back of the journal.

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