Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethane-1,2-diylbis(methylphosphinic acid)

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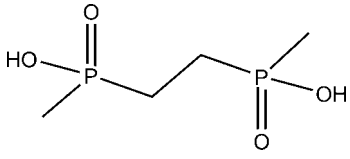
Received 4 December 2007; accepted 21 December 2007

Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.079; data-to-parameter ratio = 17.7.

In the title compound, $C_4H_{12}O_4P_2$, there are two crystallographically independent half-molecules in the asymmetric unit, both molecules lying on centres of symmetry. Each molecule is connected on both sides to neighbouring molecules *via* strong O—H···O hydrogen bonds. The –POOH groups accept and donate one hydrogen bond in interactions with the neighbouring –POOH group of the adjacent molecule, to give one-dimensional chains along [10$\bar{1}$]. As each phosphinic acid group donates and accepts one hydrogen bond, the connection between the molecules is best described by a ring motif which can be classified by the Etter symbol $R_2^2(8)$.

Related literature

For related literature, see: Bruckmann *et al.* (1999); Etter *et al.* (1990); Sicken *et al.* (2000).



Experimental

Crystal data

$C_4H_{12}O_4P_2$
M_r = 186.08
Monoclinic, $P2_1/c$
a = 6.7761 (18) Å
b = 18.703 (8) Å
c = 6.8401 (15) Å
β = 102.09 (3)°
V = 847.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 290 (2) K
0.40 × 0.35 × 0.30 mm

Data collection

Nicolet/Siemens P2$_1$/P3 four-circle diffractometer
Absorption correction: none
4847 measured reflections
2466 independent reflections
1879 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
3 standard reflections every 100 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.079
S = 1.01
2466 reflections
139 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O2	0.95 (4)	1.55 (4)	2.504 (2)	178 (3)
O1—H1···O3	0.94 (4)	1.56 (4)	2.499 (2)	179 (4)

Data collection: *R3m/V Software* (Siemens, 1989); cell refinement: *R3m/V Software*; data reduction: *R3m/V Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

The authors thank L. Langner for technical support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2077).

References

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supporting information

Acta Cryst. (2008). E**64**, o400 [doi:10.1107/S1600536807068122]

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S1. Comment

The title compound, [HOO(CH_3)P-(CH_2)$_2$-P(CH_3)OOH, crystallizes in the monoclinic centrosymmetric space group $P2_1/c$ with two crystallographically independent molecules in the asymmetric unit both of them lying on a centre of symmetry. The molecules are connected on both sides to the next molecules *via* strong O—H···H hydrogen bonds. The bond lengths and angles in the two crystallographic independent molecules are identical within the ranges of their standard uncertainties. As each posphinic acid group donates and accepts one hydrogen bond the motif of this connection between the molecules is best described by an eight-membered ring (Fig.1) which can be classified by the Etter symbol $R^2_2(8)$ (Etter *et al.*, 1990). A motif which is well known for acetic acid and its derivatives. Each –POOH group accepts and donates one hydrogen bond to the neighbouring –POOH groups of the next molecules to give a one-dimensional chains along [10–1]. This was surprising to us, as the very similar ethane-1,2-diylbis(phosphinic acid) forms a two-dimensional hydrogen bonded network (Bruckmann *et al.*, 1999).

S2. Experimental

The title compound is generally available by methods described in the literature (Sicken *et al.*, 2000). Recrystallization of the raw material from ethanolic solution at room temperature gave block shaped, colourless crystals.

S3. Refinement

After refinement of all non-hydrogen atoms using anisotropic displacement parameters, all H atom positions were obtained from successive difference Fourier synthesis. Atom coordinates as well as individual U_{iso} values are refined freely for each hydrogen atom.

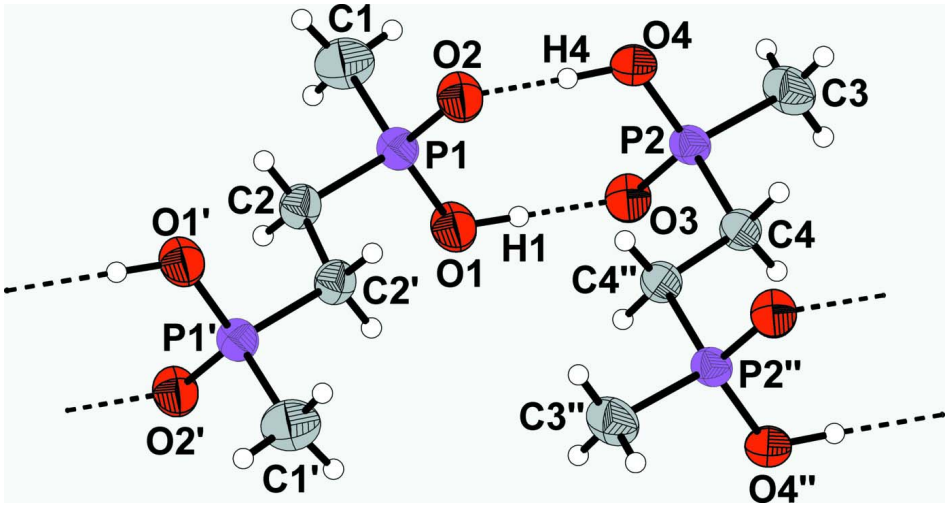

Figure 1

Part of the hydrogen bonded chain of the title structure. (displacement ellipsoids at the 50% probability level, H-atoms drawn with arbitrary radius, ′ = -*x*, 1 - *y*, 2 - *z*; ″ = 1 - *x*, 1 - *y*, 1 - *z*)

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Crystal data

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Hall symbol: -P 2ybc
a = 6.7761 (18) Å
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c = 6.8401 (15) Å
β = 102.09 (3)°
V = 847.7 (5) Å^3
Z = 4

$F(000)$ = 392
D_x = 1.458 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 37 reflections
θ = 5.1–14.3°
μ = 0.47 mm^{-1}
T = 290 K
Block, colourless
0.40 × 0.35 × 0.30 mm

Data collection

Nicolet/Siemens P21/P3-four-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
4847 measured reflections
2466 independent reflections
1879 reflections with $I > 2\sigma(I)$

R_{int} = 0.023
θ_{max} = 30.0°, θ_{min} = 2.2°
h = 0→9
k = −26→26
l = −9→9
3 standard reflections every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.079
S = 1.01
2466 reflections
139 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.025P)^2 + 0.41P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.10537 (7)	0.60187 (2)	0.89929 (7)	0.03542 (12)
O1	0.0298 (2)	0.59213 (9)	0.6716 (2)	0.0477 (3)
H1	0.114 (6)	0.606 (2)	0.586 (6)	0.145 (15)*
O2	0.32968 (19)	0.59006 (8)	0.96702 (19)	0.0455 (3)
C1	0.0385 (5)	0.68801 (12)	0.9733 (4)	0.0563 (6)
H11	0.067 (4)	0.6933 (14)	1.116 (4)	0.070 (8)*
H12	0.106 (5)	0.7222 (17)	0.917 (5)	0.094 (10)*
H13	−0.088 (4)	0.6940 (15)	0.925 (4)	0.075 (9)*
C2	−0.0220 (3)	0.53869 (9)	1.0246 (3)	0.0371 (4)
H21	0.017 (3)	0.5445 (12)	1.163 (3)	0.047 (6)*
H22	−0.157 (3)	0.5475 (12)	0.984 (3)	0.051 (6)*
P2	0.48253 (7)	0.61724 (2)	0.51341 (7)	0.03302 (11)
O3	0.25753 (19)	0.62778 (7)	0.44442 (18)	0.0412 (3)
O4	0.5560 (2)	0.62903 (7)	0.7409 (2)	0.0421 (3)
H4	0.470 (5)	0.6132 (18)	0.826 (5)	0.117 (12)*
C3	0.6086 (4)	0.67852 (12)	0.3826 (4)	0.0501 (5)
H31	0.575 (4)	0.7228 (16)	0.410 (4)	0.078 (8)*
H32	0.565 (4)	0.6729 (14)	0.247 (4)	0.065 (8)*
H33	0.738 (4)	0.6751 (15)	0.426 (4)	0.079 (9)*
C4	0.5508 (3)	0.52908 (9)	0.4481 (3)	0.0343 (3)
H41	0.693 (3)	0.5257 (11)	0.494 (3)	0.042 (5)*
H42	0.513 (3)	0.5270 (11)	0.311 (3)	0.044 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0352 (2)	0.0375 (2)	0.0329 (2)	−0.00097 (17)	0.00569 (17)	0.00385 (18)
O1	0.0359 (7)	0.0712 (10)	0.0338 (7)	−0.0062 (6)	0.0024 (5)	0.0038 (6)
O2	0.0350 (7)	0.0632 (9)	0.0364 (7)	−0.0004 (6)	0.0030 (5)	0.0047 (6)
C1	0.0677 (16)	0.0365 (10)	0.0624 (16)	0.0033 (10)	0.0087 (12)	0.0018 (10)
C2	0.0403 (9)	0.0362 (9)	0.0361 (9)	0.0017 (7)	0.0108 (7)	0.0015 (7)
P2	0.0359 (2)	0.02862 (19)	0.0346 (2)	−0.00337 (16)	0.00759 (17)	0.00217 (17)
O3	0.0379 (7)	0.0472 (7)	0.0371 (7)	0.0038 (5)	0.0047 (5)	0.0049 (5)

O4	0.0374 (7)	0.0493 (8)	0.0378 (7)	−0.0064 (6)	0.0037 (5)	−0.0068 (6)
C3	0.0576 (14)	0.0369 (10)	0.0563 (13)	−0.0105 (9)	0.0133 (11)	0.0095 (9)
C4	0.0380 (9)	0.0299 (8)	0.0357 (9)	−0.0019 (7)	0.0095 (7)	0.0018 (7)

Geometric parameters (Å, º)

P1—O2	1.5096 (14)	P2—O3	1.5115 (14)
P1—O1	1.5452 (15)	P2—O4	1.5468 (14)
P1—C1	1.776 (2)	P2—C3	1.779 (2)
P1—C2	1.7850 (19)	P2—C4	1.7942 (19)
O1—H1	0.94 (4)	O4—H4	0.95 (4)
C1—H11	0.96 (3)	C3—H31	0.89 (3)
C1—H12	0.92 (3)	C3—H32	0.92 (3)
C1—H13	0.86 (3)	C3—H33	0.87 (3)
C2—C2^{i}	1.529 (3)	C4—C4^{ii}	1.539 (3)
C2—H21	0.93 (2)	C4—H41	0.95 (2)
C2—H22	0.91 (2)	C4—H42	0.92 (2)
O2—P1—O1	113.08 (8)	O3—P2—O4	112.74 (8)
O2—P1—C1	110.24 (12)	O3—P2—C3	108.55 (11)
O1—P1—C1	110.11 (12)	O4—P2—C3	109.08 (11)
O2—P1—C2	108.23 (9)	O3—P2—C4	109.76 (8)
O1—P1—C2	108.33 (9)	O4—P2—C4	109.63 (8)
C1—P1—C2	106.60 (11)	C3—P2—C4	106.90 (11)
P1—O1—H1	119 (2)	P2—O4—H4	118 (2)
P1—C1—H11	112.0 (16)	P2—C3—H31	108.9 (18)
P1—C1—H12	109.5 (19)	P2—C3—H32	110.5 (16)
H11—C1—H12	110 (2)	H31—C3—H32	106 (2)
P1—C1—H13	107.7 (19)	P2—C3—H33	109.7 (19)
H11—C1—H13	110 (2)	H31—C3—H33	106 (3)
H12—C1—H13	107 (3)	H32—C3—H33	115 (2)
C2^{i}—C2—P1	112.62 (17)	C4^{ii}—C4—P2	111.78 (16)
C2^{i}—C2—H21	107.8 (14)	C4^{ii}—C4—H41	108.8 (12)
P1—C2—H21	110.3 (13)	P2—C4—H41	106.0 (12)
C2^{i}—C2—H22	109.4 (15)	C4^{ii}—C4—H42	112.7 (13)
P1—C2—H22	106.6 (14)	P2—C4—H42	105.1 (13)
H21—C2—H22	110.1 (18)	H41—C4—H42	112.3 (17)
O2—P1—C2—C2^{i}	−61.3 (2)	O3—P2—C4—C4^{ii}	−62.1 (2)
O1—P1—C2—C2^{i}	61.7 (2)	O4—P2—C4—C4^{ii}	62.3 (2)
C1—P1—C2—C2^{i}	−179.8 (2)	C3—P2—C4—C4^{ii}	−179.64 (19)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+2; (ii) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O2	0.95 (4)	1.55 (4)	2.504 (2)	178 (3)
O1—H1···O3	0.94 (4)	1.56 (4)	2.499 (2)	179 (4)