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Diethyl hydroxy(4-methoxyphenyl)-methylphosphonate

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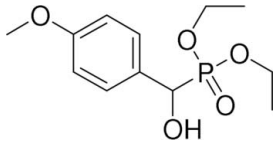
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.005 Å; R factor = 0.064; wR factor = 0.159; data-to-parameter ratio = 17.7.

The title compound, $C_{12}H_{19}O_5P$, was obtained by the reaction of 4-methoxybenzaldehyde and diethyl phosphonate. Intermolecular O—H···O hydrogen bonds between the phosphoryl O atom and the hydroxy group result in the formation of an infinite chain connecting the molecules along the b axis.

Related literature

For related literature, see: Babak & Rahman (2001); Chen *et al.* (1995); Martine *et al.* (1995); Smaardijk *et al.* (1985); Stowasser *et al.* (1992).



Experimental

Crystal data

$C_{12}H_{19}O_5P$
M_r = 274.24
Monoclinic, $P2_1/n$
a = 10.454 (4) Å
b = 7.745 (3) Å
c = 18.021 (7) Å
β = 105.228 (7)°
V = 1407.8 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 273 (2) K
0.30 × 0.20 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.941, T_{max} = 0.964
7612 measured reflections
2891 independent reflections
1818 reflections with $I > 2\sigma(I)$
R_{int} = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.159
S = 1.06
2891 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1B···O2^{i}	0.82	1.87	2.687 (3)	173

Symmetry code: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2226).

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supplementary materials

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Diethyl hydroxy(4-methoxyphenyl)methylphosphonate

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Comment

In recent years \a-hydroxyphosphonic acids esters have attracted much attention due to their wide biological activity (Stowasser *et al.*, 1992) and pharmaceutical interest (Chen *et al.*, 1995). They are useful reagents for the synthesis of enol ethers and \a-ketophosphonates (Babak *et al.*, 2001). Bond lengths and angles in the title compound, (I), are in agreement with the values reported for related compounds (Smaardijk *et al.*, 1985). Intermolecular O—H···O hydrogen bond between the phosphoryl O atom and the hydroxy group results in the formation of an infinite chain connecting the molecules along the *b* axis (Table 1 and Fig. 3).

Experimental

To a solution of 4-methoxybenzaldehyde (5 mmol, 0.68 g) and diethyl phosphonate (5 mmol, 0.69 g) in tetrahydrofuran (5 ml) at 0 \%C was added aqueous ammonia (25%, 1.6 ml). The mixture was left to stand at ambient temperature for 2 h, during which time a precipitate separated. The precipitate was filtered off and washed rapidly with cold diethyl ether (Martine *et al.*, 1995). Single crystals were obtained by crystallization of a dichloromethane/ petroleum ether (*v*/*v* = 1/4) solution (Fig. 1). CHN analysis, calculated for C~12~H~19Õ~5~P: C, 52.55%; H, 6.98%; Found: C, 52.41%; H, 6.96%.

Refinement

All H atoms were placed in geometrically idealized positions and treated as riding on their parent atoms, with C—H = 0.93 (aromatic), 0.96 (CH_3), 0.97 (CH_2) or 0.98 (CH), O—H = 0.82 \%A and U_{iso}(H) = 1.2U_{eq} (aromatic C, CH and CH_2) or 1.5U_{eq} (methyl C and O).

Figures

Fig. 1. Reaction scheme.

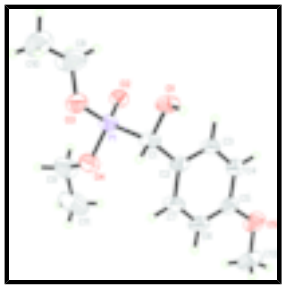

Fig. 2. Molecular view of I with the atom-numbering scheme. Displacement ellipsoids are drawn atthe 30% probability level. H atoms are represented as small spheres of arbitrary radii.

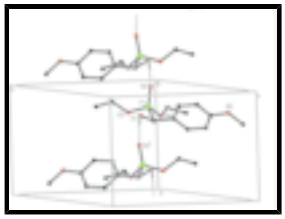

Fig. 3. Packing diagram of title compound showing the O—H···O interactions as dashed lines. The H atoms not involved in hydrogen bonding have been omitted. [Symmetry code: (i) −*x* + 1/2, *y* + 1/2, −*z* + 1/2)]

diethyl hydroxy(4-methoxyphenyl)methylphosphonate

Crystal data

$C_{12}H_{19}O_5P$	F_{000} = 584
M_r = 274.24	D_x = 1.294 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1884 reflections
a = 10.454 (4) Å	θ = 2.6–25.1º
b = 7.745 (3) Å	μ = 0.21 mm^{-1}
c = 18.021 (7) Å	*T* = 273 (2) K
β = 105.228 (7)º	Block, colourless
V = 1407.8 (9) Å^3	0.30 × 0.20 × 0.18 mm
Z = 4	

Data collection

Bruker APEX area-detector diffractometer	2891 independent reflections
Radiation source: fine-focus sealed tube	1818 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.068
T = 273(2) K	θ_{max} = 26.5º
φ and ω scan	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	*h* = −12→13
T_{min} = 0.941, T_{max} = 0.964	*k* = −9→9
7612 measured reflections	*l* = −18→22

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.065	H-atom parameters constrained
$wR(F^2)$ = 0.159	$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.210P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max} < 0.001$
2891 reflections	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
163 parameters	$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.14301 (7)	0.22551 (10)	0.12858 (4)	0.0579 (3)
C1	0.1690 (2)	0.3440 (4)	0.21623 (17)	0.0585 (8)
H1A	0.1783	0.4665	0.2051	0.070*
O1	0.29092 (16)	0.2847 (3)	0.26315 (12)	0.0686 (6)
H1B	0.3220	0.3574	0.2960	0.103*
C2	0.0569 (2)	0.3249 (3)	0.25236 (16)	0.0538 (7)
O2	0.12622 (17)	0.0406 (2)	0.13491 (10)	0.0610 (5)
O3	0.2581 (2)	0.2753 (3)	0.09399 (13)	0.0794 (7)
C3	0.0574 (3)	0.2032 (4)	0.30721 (17)	0.0631 (8)
H3A	0.1283	0.1268	0.3217	0.076*
O4	0.0233 (2)	0.3229 (3)	0.07794 (13)	0.0766 (6)
C4	−0.0443 (3)	0.1923 (4)	0.34103 (18)	0.0711 (9)
H4A	−0.0413	0.1103	0.3792	0.085*
O5	−0.2459 (2)	0.2769 (3)	0.35739 (14)	0.0913 (8)
C5	−0.1500 (3)	0.2997 (4)	0.31971 (18)	0.0643 (8)
C6	−0.1545 (3)	0.4176 (5)	0.2642 (2)	0.0738 (9)
H6A	−0.2276	0.4898	0.2483	0.089*
C7	−0.0514 (3)	0.4306 (4)	0.23163 (19)	0.0683 (8)
H7A	−0.0547	0.5139	0.1940	0.082*
C8	0.3671 (5)	0.1724 (7)	0.0963 (4)	0.154 (2)
H8A	0.4157	0.1605	0.1499	0.185*
H8B	0.3342	0.0585	0.0785	0.185*
C9	−0.0663 (4)	0.2564 (5)	0.0107 (2)	0.1039 (13)
H9A	−0.0628	0.1312	0.0115	0.125*
H9B	−0.0407	0.2960	−0.0344	0.125*
C10	0.4525 (5)	0.2167 (7)	0.0581 (3)	0.143 (2)
H10B	0.5230	0.1335	0.0672	0.215*
H10C	0.4883	0.3284	0.0750	0.215*
H10D	0.4089	0.2206	0.0042	0.215*
C11	−0.1979 (4)	0.3120 (7)	0.0065 (3)	0.142 (2)
H11A	−0.2576	0.2658	−0.0390	0.214*

H11B	−0.2016	0.4359	0.0048	0.214*
H11C	−0.2233	0.2719	0.0510	0.214*
C12	−0.3571 (3)	0.3881 (6)	0.3378 (3)	0.1105 (14)
H12A	−0.4172	0.3592	0.3681	0.166*
H12B	−0.4012	0.3751	0.2842	0.166*
H12C	−0.3283	0.5055	0.3479	0.166*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0565 (5)	0.0514 (5)	0.0661 (5)	0.0027 (4)	0.0168 (4)	0.0044 (4)
C1	0.0453 (15)	0.0469 (17)	0.081 (2)	−0.0007 (13)	0.0119 (14)	−0.0014 (14)
O1	0.0438 (11)	0.0669 (14)	0.0886 (15)	0.0018 (9)	0.0057 (10)	−0.0180 (11)
C2	0.0452 (15)	0.0466 (17)	0.0645 (18)	0.0029 (12)	0.0056 (13)	−0.0098 (13)
O2	0.0687 (12)	0.0461 (12)	0.0671 (13)	−0.0026 (9)	0.0157 (10)	−0.0039 (9)
O3	0.0787 (14)	0.0681 (15)	0.1036 (18)	0.0080 (12)	0.0456 (13)	0.0170 (12)
C3	0.0542 (17)	0.067 (2)	0.0642 (19)	0.0183 (15)	0.0087 (15)	−0.0023 (15)
O4	0.0772 (14)	0.0683 (16)	0.0764 (15)	0.0111 (11)	0.0065 (12)	0.0024 (11)
C4	0.0676 (19)	0.078 (2)	0.070 (2)	0.0145 (17)	0.0220 (17)	0.0075 (16)
O5	0.0689 (14)	0.116 (2)	0.0976 (18)	0.0117 (14)	0.0382 (13)	−0.0108 (14)
C5	0.0498 (17)	0.077 (2)	0.066 (2)	0.0040 (16)	0.0162 (15)	−0.0154 (17)
C6	0.0543 (17)	0.075 (2)	0.090 (2)	0.0214 (16)	0.0157 (17)	−0.0023 (19)
C7	0.0574 (17)	0.057 (2)	0.090 (2)	0.0149 (15)	0.0186 (16)	0.0101 (16)
C8	0.111 (3)	0.140 (5)	0.249 (7)	0.039 (3)	0.114 (4)	0.073 (4)
C9	0.109 (3)	0.098 (3)	0.086 (3)	0.014 (2)	−0.009 (2)	−0.010 (2)
C10	0.126 (4)	0.155 (5)	0.172 (5)	0.025 (4)	0.082 (4)	0.017 (4)
C11	0.089 (3)	0.140 (5)	0.163 (5)	0.002 (3)	−0.030 (3)	0.025 (4)
C12	0.067 (2)	0.129 (4)	0.147 (4)	0.020 (2)	0.049 (2)	−0.025 (3)

Geometric parameters (Å, °)

P1—O2	1.451 (2)	C6—C7	1.359 (4)
P1—O4	1.539 (2)	C6—H6A	0.9300
P1—O3	1.543 (2)	C7—H7A	0.9300
P1—C1	1.785 (3)	C8—C10	1.308 (5)
C1—O1	1.408 (3)	C8—H8A	0.9700
C1—C2	1.490 (4)	C8—H8B	0.9700
C1—H1A	0.9800	C9—C11	1.425 (5)
O1—H1B	0.8200	C9—H9A	0.9700
C2—C3	1.364 (4)	C9—H9B	0.9700
C2—C7	1.367 (4)	C10—H10B	0.9600
O3—C8	1.382 (4)	C10—H10C	0.9600
C3—C4	1.360 (4)	C10—H10D	0.9600
C3—H3A	0.9300	C11—H11A	0.9600
O4—C9	1.420 (4)	C11—H11B	0.9600
C4—C5	1.356 (4)	C11—H11C	0.9600
C4—H4A	0.9300	C12—H12A	0.9600
O5—C5	1.362 (3)	C12—H12B	0.9600
O5—C12	1.415 (4)	C12—H12C	0.9600

C5—C6	1.346 (5)		
O2—P1—O4	115.73 (12)	C6—C7—H7A	119.0
O2—P1—O3	113.70 (12)	C2—C7—H7A	119.0
O4—P1—O3	103.71 (13)	C10—C8—O3	120.3 (4)
O2—P1—C1	115.56 (13)	C10—C8—H8A	107.3
O4—P1—C1	100.60 (13)	O3—C8—H8A	107.3
O3—P1—C1	105.98 (13)	C10—C8—H8B	107.3
O1—C1—C2	113.4 (2)	O3—C8—H8B	107.3
O1—C1—P1	105.49 (18)	H8A—C8—H8B	106.9
C2—C1—P1	112.39 (18)	O4—C9—C11	110.3 (3)
O1—C1—H1A	108.5	O4—C9—H9A	109.6
C2—C1—H1A	108.5	C11—C9—H9A	109.6
P1—C1—H1A	108.5	O4—C9—H9B	109.6
C1—O1—H1B	109.5	C11—C9—H9B	109.6
C3—C2—C7	117.2 (3)	H9A—C9—H9B	108.1
C3—C2—C1	122.1 (2)	C8—C10—H10B	109.5
C7—C2—C1	120.7 (3)	C8—C10—H10C	109.5
C8—O3—P1	124.6 (2)	H10B—C10—H10C	109.5
C4—C3—C2	120.8 (3)	C8—C10—H10D	109.5
C4—C3—H3A	119.6	H10B—C10—H10D	109.5
C2—C3—H3A	119.6	H10C—C10—H10D	109.5
C9—O4—P1	125.4 (2)	C9—C11—H11A	109.5
C5—C4—C3	120.6 (3)	C9—C11—H11B	109.5
C5—C4—H4A	119.7	H11A—C11—H11B	109.5
C3—C4—H4A	119.7	C9—C11—H11C	109.5
C5—O5—C12	117.4 (3)	H11A—C11—H11C	109.5
C6—C5—C4	119.6 (3)	H11B—C11—H11C	109.5
C6—C5—O5	124.9 (3)	O5—C12—H12A	109.5
C4—C5—O5	115.5 (3)	O5—C12—H12B	109.5
C5—C6—C7	119.6 (3)	H12A—C12—H12B	109.5
C5—C6—H6A	120.2	O5—C12—H12C	109.5
C7—C6—H6A	120.2	H12A—C12—H12C	109.5
C6—C7—C2	122.1 (3)	H12B—C12—H12C	109.5
O2—P1—C1—O1	64.4 (2)	O2—P1—O4—C9	−33.9 (3)
O4—P1—C1—O1	−170.20 (17)	O3—P1—O4—C9	91.3 (3)
O3—P1—C1—O1	−62.5 (2)	C1—P1—O4—C9	−159.2 (3)
O2—P1—C1—C2	−59.6 (2)	C2—C3—C4—C5	1.5 (5)
O4—P1—C1—C2	65.8 (2)	C3—C4—C5—C6	0.4 (5)
O3—P1—C1—C2	173.50 (19)	C3—C4—C5—O5	179.9 (3)
O1—C1—C2—C3	−25.5 (4)	C12—O5—C5—C6	−1.5 (5)
P1—C1—C2—C3	94.0 (3)	C12—O5—C5—C4	179.0 (3)
O1—C1—C2—C7	154.2 (3)	C4—C5—C6—C7	−1.7 (5)
P1—C1—C2—C7	−86.3 (3)	O5—C5—C6—C7	178.8 (3)
O2—P1—O3—C8	−24.8 (4)	C5—C6—C7—C2	1.2 (5)
O4—P1—O3—C8	−151.3 (4)	C3—C2—C7—C6	0.6 (4)
C1—P1—O3—C8	103.2 (4)	C1—C2—C7—C6	−179.2 (3)
C7—C2—C3—C4	−1.9 (4)	P1—O3—C8—C10	172.4 (4)
C1—C2—C3—C4	177.8 (3)	P1—O4—C9—C11	142.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1B···O2^{i}	0.82	1.87	2.687 (3)	173

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

Fig. 1

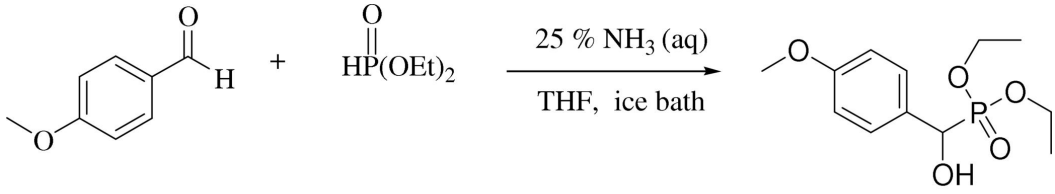

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Fig. 2

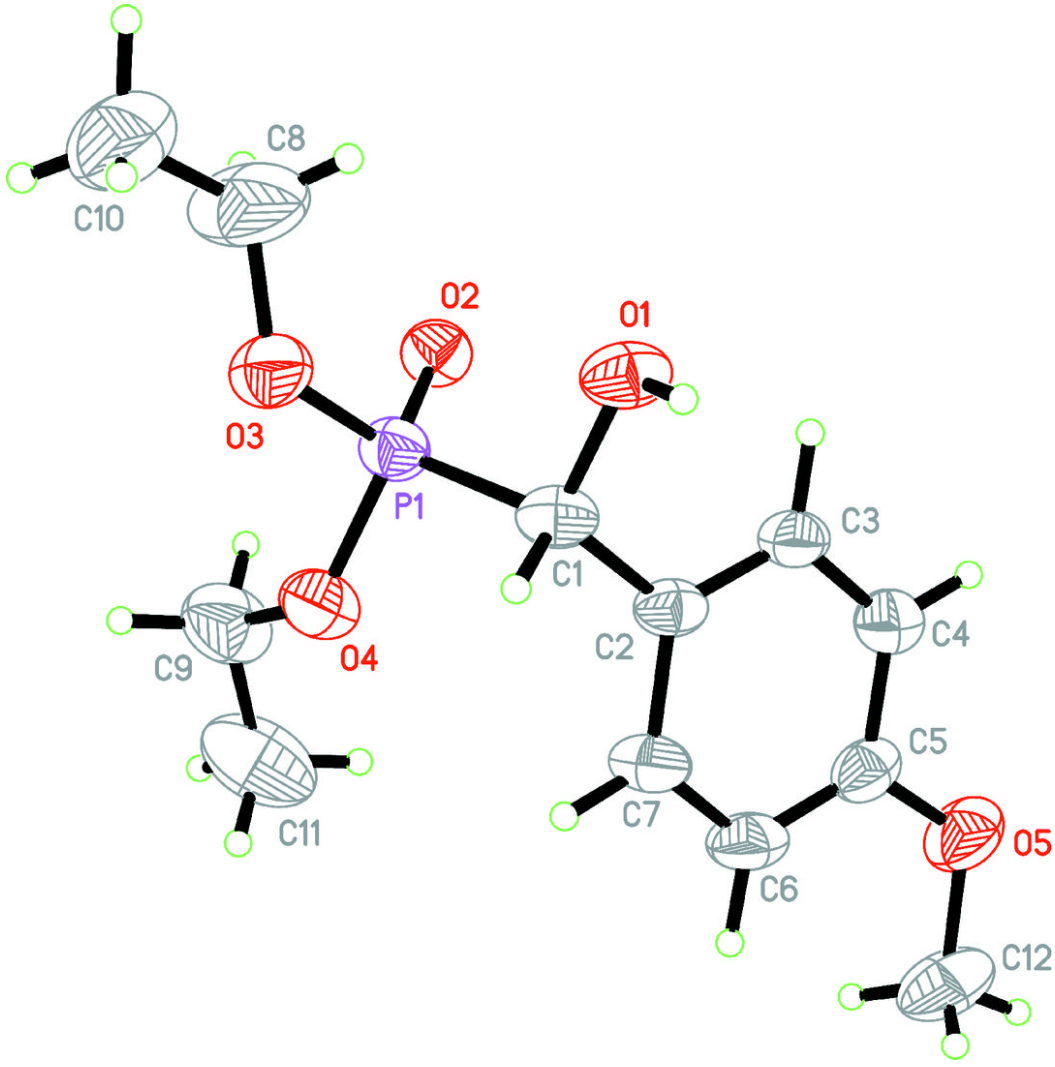

Fig. 3