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2-[(4-Chlorophenyl)(hydroxy)methyl]-5,5-dimethyl-1,3,2-dioxaphosphinan-2-one

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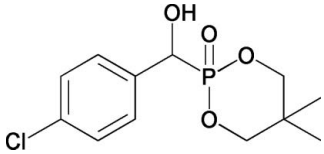
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.062; wR factor = 0.154; data-to-parameter ratio = 20.9.

In the title compound, $C_{12}H_{16}ClO_4P$, the phosphonate ring adopts a chair conformation. In the crystal, intermolecular O—H···O hydrogen bonds link the molecules into chains propagating along the b axis.

Related literature

For the synthesis of hydroxyphosphonates, see: Zhou *et al.* (2008). For the synthesis and biological activity of hydroxyphosphonate derivatives, see: Peng *et al.* (2007); Liu *et al.* (2006). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{12}H_{16}ClO_4P$
M_r = 290.67
Monoclinic, $P2_1/c$
a = 12.8965 (11) Å
b = 9.4449 (8) Å
c = 11.6425 (10) Å
β = 98.630 (1)°
V = 1402.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.39 mm^{-1}
T = 298 K
0.23 × 0.16 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
10123 measured reflections
3463 independent reflections
3141 reflections with $I > 2\sigma(I)$
R_{int} = 0.071

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.154
S = 1.15
3463 reflections
166 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O3^{i}	0.82	1.89	2.705 (3)	172

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2424).

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supporting information

Acta Cryst. (2011). E**67**, o926 [doi:10.1107/S1600536811008944]

2-[(4-Chlorophenyl)(hydroxy)methyl]-5,5-dimethyl-1,3,2-dioxaphosphinan-2-one

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S1. Comment

Acyclic alpha-hydroxyphosphonates and cyclic alpha-hydroxyphosphonates can be used as very convenient intermediates. They are also an attractive class of biologically active compounds (Peng *et al.*, 2007; Liu *et al.*, 2006). In our research work aimed at searching for novel agrochemicals, we have attempted to synthesize hydroxyphosphonate derivatives using literature procedures. Here we report the crystal structure of the title compound (Fig. 1). The bond lengths (Allen *et al.*, 1987) and angles show normal values. The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds (Table 1, Fig. 2).

S2. Experimental

The title compound was prepared according to literature procedures (Zhou *et al.* 2008). 4-Chlorobenzaldehyde (10 mmol) was added to a solution of 5,5-dimethyl-1,3,2-dioxaphosphinane (10 mmol) in triethylamine (10 mmol). The mixture was stirred at room temperature for 20 h. Pure title compound was afforded by column chromatography on silica gel (acetone/petroleum ether 1:2). Recrystallization from ethyl acetate over a period of one week gave colourless crystals of the title compound.

S3. Refinement

C-bound H atoms and the O-bound H atom were geometrically positioned (C—H 0.93–0.97 Å, O—H = 0.82 Å) and refined as riding, with U_{iso}(H) =kU_{eq}(C, O), where k = 1.5 for methyl H and OH and 1.2 for other H atoms.

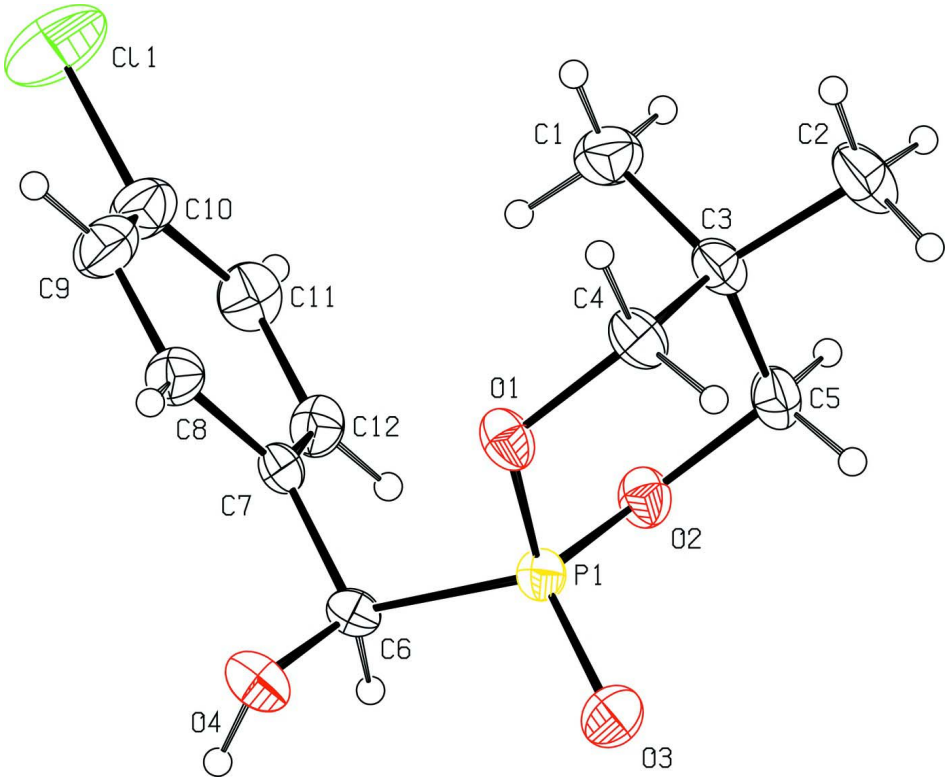

Figure 1

Molecular structure of the title compound, with displacement parameters drawn at the 50% probability level. Hydrogen atoms are shown as spheres of arbitrary radius.

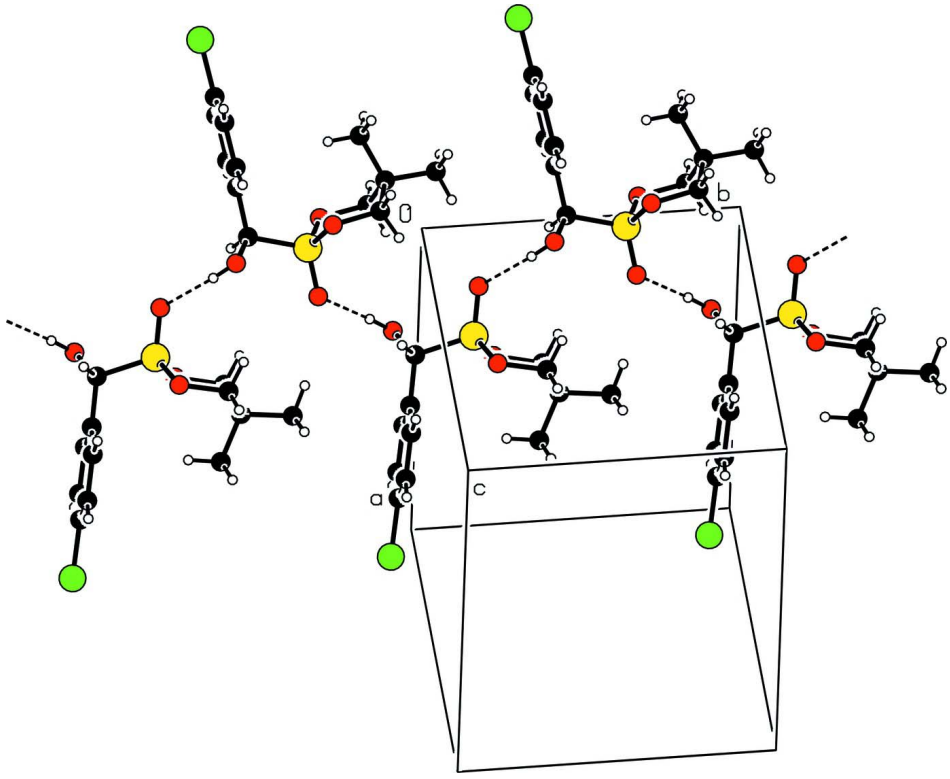

Figure 2

Part of the crystal packing, showing the intermolecular hydrogen bonds as dashed lines.

2-[(4-Chlorophenyl)(hydroxy)methyl]-5,5-dimethyl-1,3,2-dioxaphosphinan-2-one

Crystal data

$C_{12}H_{16}ClO_4P$
$M_r = 290.67$
Monoclinic, $P2_1/c$
$a = 12.8965$ (11) Å
$b = 9.4449$ (8) Å
$c = 11.6425$ (10) Å
$\beta = 98.630$ (1)°
$V = 1402.1$ (2) Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.377$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4935 reflections
$\theta = 2.7$–$28.2°$
$\mu = 0.39$ mm^{-1}
$T = 298$ K
Block, colourless
0.23 × 0.16 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
10123 measured reflections
3463 independent reflections
3141 reflections with $I > 2\sigma(I)$
$R_{int} = 0.071$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.7°$
$h = -13 \rightarrow 17$
$k = -9 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.154$
$S = 1.15$
3463 reflections
166 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.7742P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ 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 $F^2 > \sigma(F^2)$ 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}
C1	0.2944 (2)	0.3005 (3)	0.5354 (2)	0.0543 (6)
H1A	0.2923	0.3256	0.6150	0.081*
H1B	0.3596	0.3312	0.5135	0.081*
H1C	0.2883	0.1996	0.5267	0.081*
C2	0.2096 (3)	0.5330 (3)	0.4720 (3)	0.0635 (8)
H2A	0.1547	0.5765	0.4188	0.095*
H2B	0.2764	0.5659	0.4561	0.095*
H2C	0.2014	0.5577	0.5501	0.095*
C3	0.2035 (2)	0.3720 (2)	0.4576 (2)	0.0407 (5)
C4	0.2059 (2)	0.3377 (2)	0.3303 (2)	0.0430 (5)
H4A	0.1519	0.3917	0.2823	0.052*
H4B	0.2733	0.3655	0.3099	0.052*
C5	0.09896 (19)	0.3251 (2)	0.4897 (2)	0.0429 (5)
H5A	0.0971	0.3465	0.5708	0.052*
H5B	0.0428	0.3773	0.4434	0.052*
C6	0.12155 (17)	−0.0698 (2)	0.36460 (19)	0.0359 (4)
H6	0.0632	−0.1171	0.3939	0.043*
C7	0.21964 (16)	−0.09498 (19)	0.44979 (18)	0.0330 (4)
C8	0.31652 (19)	−0.1076 (3)	0.4118 (2)	0.0432 (5)
H8	0.3212	−0.0978	0.3333	0.052*
C9	0.4058 (2)	−0.1346 (3)	0.4902 (3)	0.0550 (6)
H9	0.4705	−0.1435	0.4647	0.066*
C10	0.3983 (2)	−0.1482 (3)	0.6055 (3)	0.0554 (7)
C11	0.3035 (2)	−0.1347 (3)	0.6457 (2)	0.0509 (6)
H11	0.2996	−0.1430	0.7245	0.061*

C12	0.21483 (19)	−0.1088 (2)	0.5673 (2)	0.0422 (5)
H12	0.1505	−0.1004	0.5936	0.051*
Cl1	0.51070 (8)	−0.18286 (16)	0.70446 (9)	0.1062 (4)
O1	0.18909 (13)	0.18663 (16)	0.30702 (13)	0.0403 (4)
O2	0.08252 (12)	0.17262 (17)	0.46986 (13)	0.0402 (4)
O3	−0.00798 (14)	0.1404 (2)	0.26116 (16)	0.0537 (5)
O4	0.12788 (16)	−0.12340 (19)	0.25263 (15)	0.0526 (5)
H4	0.0926	−0.1957	0.2418	0.079*
P1	0.08858 (4)	0.11632 (6)	0.34372 (5)	0.03362 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0489 (14)	0.0561 (16)	0.0541 (14)	−0.0019 (12)	−0.0045 (11)	0.0008 (12)
C2	0.088 (2)	0.0332 (12)	0.0674 (17)	−0.0063 (13)	0.0042 (16)	−0.0104 (12)
C3	0.0505 (13)	0.0286 (10)	0.0416 (11)	−0.0025 (9)	0.0024 (10)	−0.0031 (8)
C4	0.0560 (14)	0.0298 (10)	0.0439 (12)	−0.0069 (10)	0.0099 (10)	0.0024 (9)
C5	0.0511 (13)	0.0373 (11)	0.0411 (11)	0.0065 (10)	0.0093 (10)	−0.0078 (9)
C6	0.0345 (10)	0.0291 (9)	0.0438 (11)	−0.0071 (8)	0.0048 (8)	−0.0022 (8)
C7	0.0356 (10)	0.0188 (8)	0.0442 (11)	−0.0014 (7)	0.0045 (8)	0.0004 (7)
C8	0.0402 (12)	0.0439 (12)	0.0463 (12)	−0.0019 (10)	0.0096 (10)	0.0011 (10)
C9	0.0366 (12)	0.0619 (17)	0.0668 (16)	0.0031 (11)	0.0087 (11)	0.0032 (13)
C10	0.0442 (14)	0.0571 (16)	0.0606 (16)	0.0052 (12)	−0.0060 (11)	0.0079 (13)
C11	0.0615 (16)	0.0483 (14)	0.0421 (12)	0.0045 (12)	0.0049 (11)	0.0084 (10)
C12	0.0419 (12)	0.0384 (12)	0.0476 (12)	0.0039 (9)	0.0114 (10)	0.0043 (9)
Cl1	0.0629 (6)	0.1541 (11)	0.0912 (7)	0.0175 (6)	−0.0221 (5)	0.0266 (7)
O1	0.0520 (9)	0.0310 (8)	0.0412 (8)	−0.0050 (7)	0.0175 (7)	−0.0022 (6)
O2	0.0435 (9)	0.0373 (8)	0.0423 (8)	0.0009 (7)	0.0150 (7)	−0.0016 (6)
O3	0.0485 (10)	0.0528 (10)	0.0546 (10)	0.0105 (8)	−0.0089 (8)	0.0022 (8)
O4	0.0622 (12)	0.0444 (10)	0.0503 (10)	−0.0086 (8)	0.0054 (8)	−0.0130 (8)
P1	0.0342 (3)	0.0304 (3)	0.0358 (3)	0.0021 (2)	0.0039 (2)	0.0004 (2)

Geometric parameters (Å, º)

C1—C3	1.528 (3)	C6—P1	1.816 (2)
C1—H1A	0.9600	C6—H6	0.9800
C1—H1B	0.9600	C7—C12	1.385 (3)
C1—H1C	0.9600	C7—C8	1.392 (3)
C2—C3	1.530 (3)	C8—C9	1.382 (3)
C2—H2A	0.9600	C8—H8	0.9300
C2—H2B	0.9600	C9—C10	1.367 (4)
C2—H2C	0.9600	C9—H9	0.9300
C3—C5	1.518 (3)	C10—C11	1.379 (4)
C3—C4	1.522 (3)	C10—Cl1	1.742 (3)
C4—O1	1.463 (3)	C11—C12	1.373 (3)
C4—H4A	0.9700	C11—H11	0.9300
C4—H4B	0.9700	C12—H12	0.9300
C5—O2	1.469 (3)	O1—P1	1.5716 (16)

C5—H5A	0.9700	O2—P1	1.5748 (16)
C5—H5B	0.9700	O3—P1	1.4721 (18)
C6—O4	1.412 (3)	O4—H4	0.8200
C6—C7	1.505 (3)		

C3—C1—H1A	109.5	C7—C6—P1	113.47 (14)
C3—C1—H1B	109.5	O4—C6—H6	108.2
H1A—C1—H1B	109.5	C7—C6—H6	108.2
C3—C1—H1C	109.5	P1—C6—H6	108.2
H1A—C1—H1C	109.5	C12—C7—C8	118.8 (2)
H1B—C1—H1C	109.5	C12—C7—C6	120.54 (19)
C3—C2—H2A	109.5	C8—C7—C6	120.7 (2)
C3—C2—H2B	109.5	C9—C8—C7	120.4 (2)
H2A—C2—H2B	109.5	C9—C8—H8	119.8
C3—C2—H2C	109.5	C7—C8—H8	119.8
H2A—C2—H2C	109.5	C10—C9—C8	119.4 (2)
H2B—C2—H2C	109.5	C10—C9—H9	120.3
C5—C3—C4	109.08 (19)	C8—C9—H9	120.3
C5—C3—C1	110.8 (2)	C9—C10—C11	121.5 (2)
C4—C3—C1	110.9 (2)	C9—C10—Cl1	119.5 (2)
C5—C3—C2	107.2 (2)	C11—C10—Cl1	119.0 (2)
C4—C3—C2	108.0 (2)	C12—C11—C10	118.9 (2)
C1—C3—C2	110.6 (2)	C12—C11—H11	120.6
O1—C4—C3	111.34 (17)	C10—C11—H11	120.6
O1—C4—H4A	109.4	C11—C12—C7	121.1 (2)
C3—C4—H4A	109.4	C11—C12—H12	119.4
O1—C4—H4B	109.4	C7—C12—H12	119.4
C3—C4—H4B	109.4	C4—O1—P1	117.87 (14)
H4A—C4—H4B	108.0	C5—O2—P1	116.83 (14)
O2—C5—C3	111.10 (17)	C6—O4—H4	109.5
O2—C5—H5A	109.4	O3—P1—O1	114.12 (11)
C3—C5—H5A	109.4	O3—P1—O2	113.65 (10)
O2—C5—H5B	109.4	O1—P1—O2	105.62 (9)
C3—C5—H5B	109.4	O3—P1—C6	113.27 (10)
H5A—C5—H5B	108.0	O1—P1—C6	105.02 (9)
O4—C6—C7	113.10 (18)	O2—P1—C6	104.20 (9)
O4—C6—P1	105.57 (15)		

C5—C3—C4—O1	−57.7 (3)	C10—C11—C12—C7	−0.6 (4)
C1—C3—C4—O1	64.7 (3)	C8—C7—C12—C11	−0.1 (3)
C2—C3—C4—O1	−173.9 (2)	C6—C7—C12—C11	178.7 (2)
C4—C3—C5—O2	58.9 (2)	C3—C4—O1—P1	54.0 (2)
C1—C3—C5—O2	−63.5 (2)	C3—C5—O2—P1	−56.2 (2)
C2—C3—C5—O2	175.7 (2)	C4—O1—P1—O3	80.51 (18)
O4—C6—C7—C12	−151.46 (19)	C4—O1—P1—O2	−45.05 (18)
P1—C6—C7—C12	88.3 (2)	C4—O1—P1—C6	−154.86 (16)
O4—C6—C7—C8	27.3 (3)	C5—O2—P1—O3	−79.99 (18)
P1—C6—C7—C8	−92.9 (2)	C5—O2—P1—O1	45.86 (17)

C12—C7—C8—C9	0.5 (3)	C5—O2—P1—C6	156.25 (16)
C6—C7—C8—C9	−178.2 (2)	O4—C6—P1—O3	56.16 (18)
C7—C8—C9—C10	−0.3 (4)	C7—C6—P1—O3	−179.45 (15)
C8—C9—C10—C11	−0.4 (5)	O4—C6—P1—O1	−69.01 (15)
C8—C9—C10—Cl1	179.8 (2)	C7—C6—P1—O1	55.38 (17)
C9—C10—C11—C12	0.8 (4)	O4—C6—P1—O2	−179.83 (14)
Cl1—C10—C11—C12	−179.3 (2)	C7—C6—P1—O2	−55.44 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3^{i}	0.82	1.89	2.705 (3)	172

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