organic compounds

Bis(propan-2-yl) [(2S,3S)-2-hydroxy-3-nitrobutan-2-yl]phosphonate

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In the title compound, $C_{10}H_{22}NO_6P$, a staggered conformation is found when the molecule is viewed down the central P—C bond, with the oxo and hydroxy groups *gauche* to each other. The crystal structure features supramolecular chains of helical topology propagating along the *b* axis, mediated by O—H···O hydrogen bonds.

Related literature

For background to the enantioselective nitroaldol reaction of α-ketophosphonates and nitromethane and for the synthesis, see: Mandal *et al.* (2007).
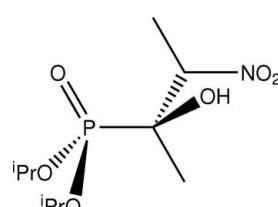
Experimental

Crystal data

$C_{10}H_{22}NO_6P$
$M_r = 283.26$
Orthorhombic, $P2_12_12_1$
$a = 7.8620$ (16) Å
$b = 11.369$ (2) Å
$c = 16.920$ (3) Å
$V = 1512.4$ (5) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.20$ mm^{-1}
$T = 173$ K
$0.32 \times 0.10 \times 0.05$ mm

Data collection

Rigaku AFC12/SATURN724 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.884$, $T_{max} = 1$
13441 measured reflections
3072 independent reflections
3020 reflections with $I > 2\sigma(I)$
$R_{int} = 0.089$
Standard reflections: 0

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.105$
$S = 1.07$
3072 reflections
166 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Absolute structure: Flack (1983), 1272 Friedel pairs
Flack parameter: 0.05 (11)

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4o···O1[i]	0.84	1.90	2.7289 (19)	172

Symmetry code: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2009).

References

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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supporting information

Bis(propan-2-yl) [(2*S*,3*S*)-2-hydroxy-3-nitrobutan-2-yl]phosphonate

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S1. Comment

The title compound, (I), was investigated as a part of previous studies on the enantioselective nitroaldol reaction of *α*-ketophosphonates and nitromethane for the synthesis of optically active *α*-hydroxy-*β*-nitrophosphonates (Mandal *et al.*, 2007). The crystal structure analysis of (I), Fig. 1, shows a staggered conformation when the molecule is viewed down the P–C7 axis in which the oxo and hydroxy groups are *gauche* to each other. The presence of O–H···O hydrogen bonding formed between the hydroxy-O4—H and O═P atoms leads to the formation of supramolecular chains along the *b* axis, Fig. 2 and Table 1.

S2. Experimental

The title compound was prepared as described in the literature (Mandal *et al.*, 2007).

S3. Refinement

The C-bound H atoms were geometrically placed (C—H = 0.98–1.00 Å) and refined as riding with $U_{iso}(H) = 1.2$–$1.5U_{eq}(C)$. The methyl H-atoms were rotated to fit the electron density. The O–H H atom was located from a difference map and refined with O–H = 0.840±0.001 Å, and with $U_{iso}(H) = 1.5U_{eq}(O)$.
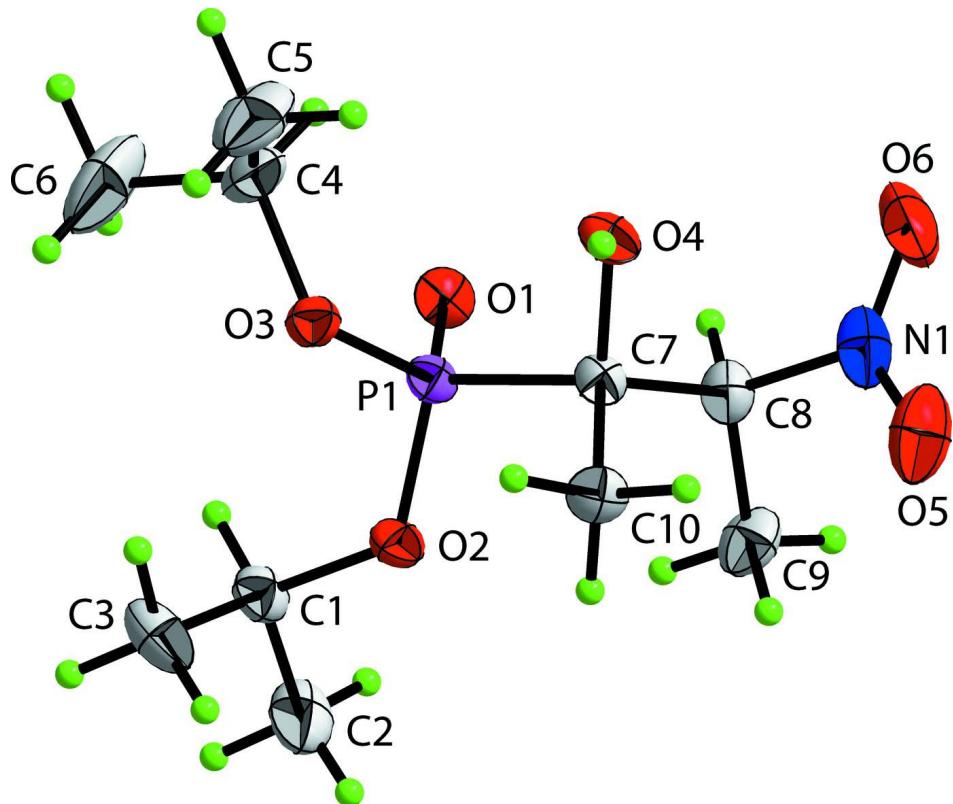
Figure 1

Molecular structure of (I), showing displacement ellipsoids at the 35% probability level.
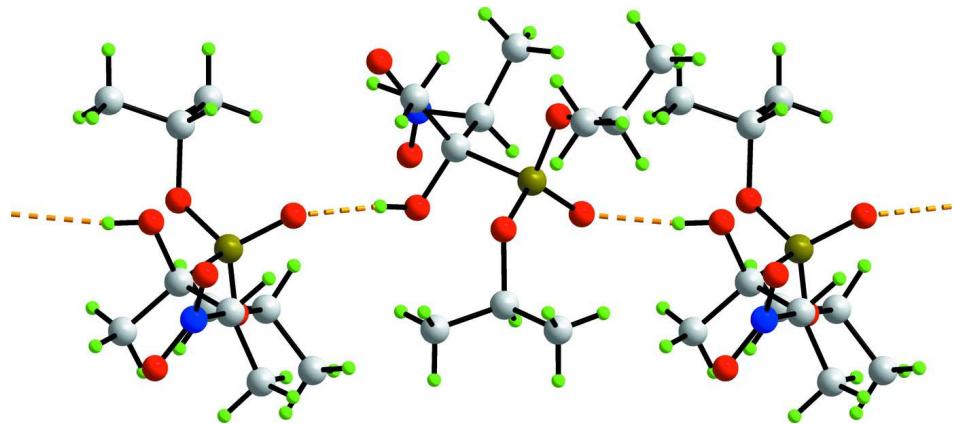
Figure 2

Supramolecular chain along the *b* axis in (I) mediated by O–H⋯O (orange dashed lines) hydrogen bonding. Colour scheme: P, olive; O, red; N, blue; C, grey; and H, green.

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Crystal data

$C_{10}H_{22}NO_6P$	Hall symbol: P 2ac 2ab
$M_r = 283.26$	$a = 7.8620$ (16) Å
Orthorhombic, $P2_12_12_1$	$b = 11.369$ (2) Å

$c = 16.920$ (3) Å
$V = 1512.4$ (5) Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.244$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2308 reflections
$\theta = 4.0$–30.1°
$\mu = 0.20$ mm^{-1}
$T = 173$ K
Block, pale-yellow
$0.32 \times 0.10 \times 0.05$ mm

Data collection

Rigaku AFC12K/SATURN724
   diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan
   (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.884$, $T_{max} = 1$

13441 measured reflections
3072 independent reflections
3020 reflections with $I > 2\sigma(I)$
$R_{int} = 0.089$
$\theta_{max} = 26.5°$, $\theta_{min} = 4.0°$
$h = -9 \rightarrow 8$
$k = -13 \rightarrow 14$
$l = -21 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.105$
$S = 1.07$
3072 reflections
166 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.2822P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Absolute structure: Flack (1983), 1272 Friedel
   pairs
Absolute structure parameter: 0.05 (11)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
P1	0.63065 (6)	0.29509 (4)	0.74005 (3)	0.03236 (14)
O1	0.51586 (18)	0.19464 (11)	0.75316 (9)	0.0416 (3)
O2	0.80539 (18)	0.26553 (13)	0.70112 (9)	0.0415 (3)
O3	0.67933 (17)	0.36408 (13)	0.81677 (8)	0.0383 (3)
O4	0.41385 (17)	0.46364 (12)	0.71762 (9)	0.0400 (3)
H4O	0.4448	0.5337	0.7241	0.060*
O5	0.3878 (4)	0.49342 (19)	0.51776 (13)	0.0848 (7)
O6	0.1751 (3)	0.4227 (2)	0.58457 (14)	0.0861 (7)
N1	0.3262 (3)	0.4256 (2)	0.56573 (14)	0.0608 (6)

C1	0.9313 (3)	0.18894 (18)	0.74033 (13)	0.0438 (5)
H1	0.8736	0.1412	0.7821	0.053*
C2	0.9985 (4)	0.1089 (3)	0.67718 (17)	0.0706 (8)
H2A	0.9051	0.0617	0.6556	0.106*
H2B	1.0849	0.0567	0.6998	0.106*
H2C	1.0495	0.1560	0.6348	0.106*
C3	1.0653 (3)	0.2668 (2)	0.7782 (2)	0.0656 (8)
H3A	1.0121	0.3162	0.8187	0.098*
H3B	1.1173	0.3168	0.7377	0.098*
H3C	1.1530	0.2175	0.8026	0.098*
C4	0.5800 (3)	0.3614 (2)	0.89018 (13)	0.0551 (6)
H4	0.4659	0.3254	0.8803	0.066*
C5	0.5601 (6)	0.4848 (3)	0.91703 (18)	0.0879 (11)
H5A	0.4970	0.5295	0.8771	0.132*
H5B	0.6726	0.5202	0.9246	0.132*
H5C	0.4976	0.4862	0.9671	0.132*
C6	0.6764 (7)	0.2898 (3)	0.94900 (18)	0.1056 (15)
H6A	0.6874	0.2089	0.9296	0.158*
H6B	0.6152	0.2896	0.9995	0.158*
H6C	0.7897	0.3238	0.9565	0.158*
C7	0.5409 (2)	0.40550 (16)	0.67209 (11)	0.0336 (4)
C8	0.4436 (3)	0.33911 (18)	0.60636 (12)	0.0419 (5)
H8	0.3722	0.2770	0.6318	0.050*
C9	0.5556 (4)	0.2804 (2)	0.54440 (13)	0.0558 (6)
H9A	0.4838	0.2406	0.5053	0.084*
H9B	0.6299	0.2227	0.5700	0.084*
H9C	0.6251	0.3401	0.5179	0.084*
C10	0.6767 (3)	0.49079 (18)	0.64308 (14)	0.0430 (5)
H10A	0.7312	0.5287	0.6885	0.065*
H10B	0.6239	0.5509	0.6095	0.065*
H10C	0.7623	0.4477	0.6125	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0303 (2)	0.0284 (2)	0.0384 (2)	0.00159 (18)	0.00078 (17)	0.00062 (19)
O1	0.0432 (7)	0.0284 (6)	0.0532 (8)	−0.0023 (5)	−0.0002 (6)	0.0044 (7)
O2	0.0357 (7)	0.0454 (8)	0.0435 (7)	0.0140 (6)	0.0051 (6)	0.0031 (6)
O3	0.0365 (7)	0.0426 (7)	0.0359 (7)	−0.0048 (6)	0.0024 (5)	−0.0017 (6)
O4	0.0342 (7)	0.0316 (7)	0.0541 (8)	0.0031 (6)	0.0021 (6)	−0.0080 (6)
O5	0.1205 (19)	0.0631 (12)	0.0708 (13)	0.0063 (14)	−0.0288 (14)	0.0171 (10)
O6	0.0593 (13)	0.1078 (17)	0.0912 (16)	0.0212 (13)	−0.0342 (12)	−0.0093 (14)
N1	0.0736 (16)	0.0513 (12)	0.0576 (12)	0.0103 (11)	−0.0261 (11)	−0.0073 (11)
C1	0.0408 (10)	0.0399 (11)	0.0507 (11)	0.0124 (8)	−0.0022 (9)	0.0007 (10)
C2	0.0768 (19)	0.0698 (17)	0.0650 (16)	0.0410 (16)	−0.0121 (14)	−0.0150 (14)
C3	0.0423 (12)	0.0584 (15)	0.096 (2)	0.0113 (11)	−0.0130 (13)	−0.0103 (14)
C4	0.0576 (14)	0.0725 (16)	0.0351 (10)	−0.0157 (12)	0.0104 (9)	−0.0010 (10)
C5	0.128 (3)	0.085 (2)	0.0507 (15)	0.038 (2)	0.0216 (17)	−0.0070 (15)

C6	0.189 (5)	0.081 (2)	0.0471 (15)	0.029 (3)	0.004 (2)	0.0180 (16)
C7	0.0344 (9)	0.0283 (8)	0.0383 (9)	0.0022 (7)	−0.0010 (8)	−0.0021 (8)
C8	0.0498 (12)	0.0332 (9)	0.0428 (10)	0.0031 (9)	−0.0088 (9)	−0.0032 (9)
C9	0.0772 (17)	0.0502 (13)	0.0399 (11)	0.0049 (13)	0.0000 (11)	−0.0097 (10)
C10	0.0451 (11)	0.0334 (10)	0.0506 (12)	−0.0024 (9)	0.0042 (9)	0.0032 (9)

Geometric parameters (Å, º)

P1—O1	1.4723 (14)	C4—C5	1.483 (4)
P1—O2	1.5602 (14)	C4—C6	1.492 (4)
P1—O3	1.5643 (15)	C4—H4	1.0000
P1—C7	1.8428 (19)	C5—H5A	0.9800
O2—C1	1.476 (2)	C5—H5B	0.9800
O3—C4	1.467 (2)	C5—H5C	0.9800
O4—C7	1.424 (2)	C6—H6A	0.9800
O4—H4O	0.8400	C6—H6B	0.9800
O5—N1	1.219 (3)	C6—H6C	0.9800
O6—N1	1.230 (4)	C7—C10	1.524 (3)
N1—C8	1.514 (3)	C7—C8	1.546 (3)
C1—C2	1.500 (3)	C8—C9	1.523 (3)
C1—C3	1.517 (3)	C8—H8	1.0000
C1—H1	1.0000	C9—H9A	0.9800
C2—H2A	0.9800	C9—H9B	0.9800
C2—H2B	0.9800	C9—H9C	0.9800
C2—H2C	0.9800	C10—H10A	0.9800
C3—H3A	0.9800	C10—H10B	0.9800
C3—H3B	0.9800	C10—H10C	0.9800
C3—H3C	0.9800		
O1—P1—O2	115.86 (9)	C4—C5—H5A	109.5
O1—P1—O3	114.44 (9)	C4—C5—H5B	109.5
O2—P1—O3	104.06 (8)	H5A—C5—H5B	109.5
O1—P1—C7	112.80 (9)	C4—C5—H5C	109.5
O2—P1—C7	102.75 (8)	H5A—C5—H5C	109.5
O3—P1—C7	105.67 (8)	H5B—C5—H5C	109.5
C1—O2—P1	121.86 (13)	C4—C6—H6A	109.5
C4—O3—P1	124.17 (14)	C4—C6—H6B	109.5
C7—O4—H4O	108.0	H6A—C6—H6B	109.5
O5—N1—O6	124.9 (3)	C4—C6—H6C	109.5
O5—N1—C8	118.1 (2)	H6A—C6—H6C	109.5
O6—N1—C8	117.0 (2)	H6B—C6—H6C	109.5
O2—C1—C2	105.91 (18)	O4—C7—C10	111.74 (15)
O2—C1—C3	108.14 (17)	O4—C7—C8	105.60 (16)
C2—C1—C3	114.2 (2)	C10—C7—C8	115.18 (18)
O2—C1—H1	109.5	O4—C7—P1	104.31 (12)
C2—C1—H1	109.5	C10—C7—P1	111.47 (14)
C3—C1—H1	109.5	C8—C7—P1	107.80 (13)
C1—C2—H2A	109.5	N1—C8—C9	108.94 (19)

C1—C2—H2B	109.5	N1—C8—C7	108.11 (16)
H2A—C2—H2B	109.5	C9—C8—C7	115.0 (2)
C1—C2—H2C	109.5	N1—C8—H8	108.2
H2A—C2—H2C	109.5	C9—C8—H8	108.2
H2B—C2—H2C	109.5	C7—C8—H8	108.2
C1—C3—H3A	109.5	C8—C9—H9A	109.5
C1—C3—H3B	109.5	C8—C9—H9B	109.5
H3A—C3—H3B	109.5	H9A—C9—H9B	109.5
C1—C3—H3C	109.5	C8—C9—H9C	109.5
H3A—C3—H3C	109.5	H9A—C9—H9C	109.5
H3B—C3—H3C	109.5	H9B—C9—H9C	109.5
O3—C4—C5	107.2 (2)	C7—C10—H10A	109.5
O3—C4—C6	107.8 (2)	C7—C10—H10B	109.5
C5—C4—C6	111.4 (3)	H10A—C10—H10B	109.5
O3—C4—H4	110.1	C7—C10—H10C	109.5
C5—C4—H4	110.1	H10A—C10—H10C	109.5
C6—C4—H4	110.1	H10B—C10—H10C	109.5
O1—P1—O2—C1	−63.20 (17)	O3—P1—C7—C10	68.93 (15)
O3—P1—O2—C1	63.34 (17)	O1—P1—C7—C8	−37.99 (17)
C7—P1—O2—C1	173.35 (15)	O2—P1—C7—C8	87.48 (15)
O1—P1—O3—C4	−21.1 (2)	O3—P1—C7—C8	−163.73 (13)
O2—P1—O3—C4	−148.58 (17)	O5—N1—C8—C9	−47.5 (3)
C7—P1—O3—C4	103.58 (19)	O6—N1—C8—C9	133.2 (2)
P1—O2—C1—C2	136.98 (19)	O5—N1—C8—C7	78.1 (3)
P1—O2—C1—C3	−100.2 (2)	O6—N1—C8—C7	−101.1 (3)
P1—O3—C4—C5	−132.8 (2)	O4—C7—C8—N1	51.7 (2)
P1—O3—C4—C6	107.1 (3)	C10—C7—C8—N1	−72.1 (2)
O1—P1—C7—O4	73.94 (14)	P1—C7—C8—N1	162.77 (16)
O2—P1—C7—O4	−160.59 (12)	O4—C7—C8—C9	173.70 (17)
O3—P1—C7—O4	−51.80 (13)	C10—C7—C8—C9	49.9 (2)
O1—P1—C7—C10	−165.33 (14)	P1—C7—C8—C9	−75.2 (2)
O2—P1—C7—C10	−39.85 (16)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4o···O1[i]	0.84	1.90	2.7289 (19)	172

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+3/2.