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Ethyl [(benzylazaniumyl)(2-hydroxyphenyl)methyl]phosphonate

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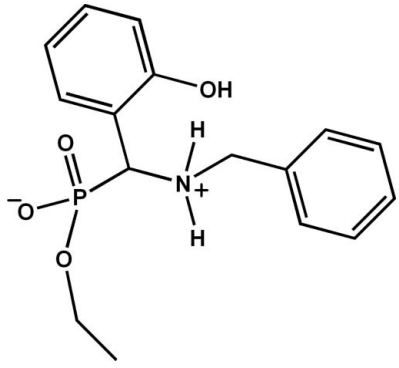
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.049; wR factor = 0.121; data-to-parameter ratio = 14.9.

The title compound, $C_{16}H_{20}NO_4P$, crystallizes as a zwitterion. In the molecule, the two aromatic rings form a dihedral angle of 55.2 (1)°. In the crystal, intermolecular N—H···O and O—H···O hydrogen bonds link the molecules into columns propagating in [010].

Related literature

For related structures, see: Zhang *et al.* (2005, 2007).



Experimental

Crystal data

$C_{16}H_{20}NO_4P$
M_r = 321.30
Monoclinic, $C2/c$
a = 28.069 (3) Å
b = 6.0927 (7) Å
c = 22.333 (3) Å
β = 124.464 (2)°
V = 3149.0 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 296 K
0.28 × 0.25 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.955, T_{max} = 0.969
8453 measured reflections
3104 independent reflections
1918 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.121
S = 1.00
3104 reflections
209 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 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
N1—H1*NA*···O1^{i}	0.97 (3)	1.77 (3)	2.738 (3)	179 (2)
N1—H1*NB*···O4	0.86 (3)	2.50 (3)	2.982 (3)	116 (2)
N1—H1*NB*···O2^{ii}	0.86 (3)	2.08 (3)	2.915 (3)	164 (2)
O4—H4*A*···O1	0.82	1.94	2.738 (3)	164
O4—H4*A*···O2^{ii}	0.82	2.58	2.925 (3)	107

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*, *PLATON* (Spek, 2009) and *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5124).

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supporting information

Acta Cryst. (2011). E**67**, o2006 [doi:10.1107/S1600536811026444]

Ethyl [(benzylazaniumyl)(2-hydroxyphenyl)methyl]phosphonate

Xiangdong Zhang, Rui Zhang, Chunhua Ge and Xiaoyan Zhang

S1. Comment

As a continuation of our structural study of (2-hydroxyphenyl)methylphosphonate derivatives (Zhang *et al.*, 2005; 2007), we present here the crystal structure of the title compound, (I), which crystallizes as a zwitterion.

In the title molecule (Fig. 1), two aromatic rings form a dihedral angle of 55.2 (1)°. In the crystal structure, intermolecular N—H···O and O—H···O hydrogen bonds (Table 1) link molecules into columns propagated in [010].

S2. Experimental

The title compound was synthesized following the reported method (Zhang *et al.*, 2005). Diethyl phosphonate (0.02 mol) was dissolved in 80 ml of ethanol. The solution was added dropwise to a mixture of salicylaldehyde (0.02 mol) and benzylamine (0.02 mol) in 30 ml e thanol which was refluxed for 4 h. The resulting solution was refluxed until solid appeared. The product was filtered and washed with ethanol. Yield 43%.

S3. Refinement

H atoms attached to C atoms were positioned geometrically and refined using a riding model, with Csp^3—H = 0.96–0.98 Å or Csp^2—H = 0.93 Å and with U_{iso}(H) = 1.2U_{eq}(C). H atom attached to O atom was O—H = 0.82 Å and with U_{iso}(H)=1.5U_{eq}(O). H atoms attached to N atom were located by difference Fourier synthesis with the range 0.86–0.97 Å and refined isotropically.

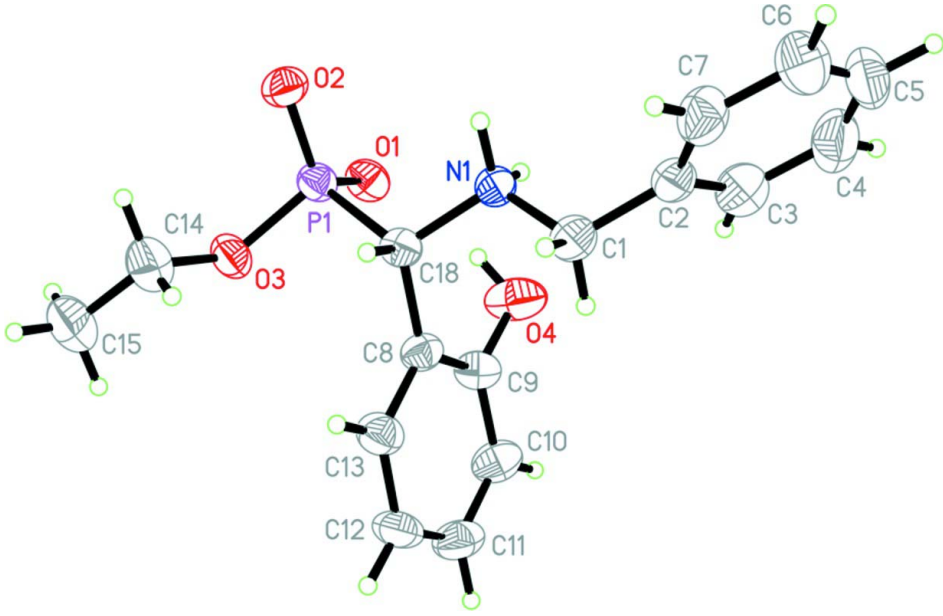

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Ethyl [(benzylazaniumyl)(2-hydroxyphenyl)methyl]phosphonate

Crystal data

$C_{16}H_{20}NO_4P$
$M_r = 321.30$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 28.069$ (3) Å
$b = 6.0927$ (7) Å
$c = 22.333$ (3) Å
$\beta = 124.464$ (2)°
$V = 3149.0$ (6) Å^3
$Z = 8$
$F(000) = 1360$
$D_x = 1.355$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 187 reflections
$\theta = 2.6$–22.5°
$\mu = 0.19$ mm^{-1}
$T = 296$ K
Block, colourless
0.28 × 0.25 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.955$, $T_{max} = 0.969$
8453 measured reflections
3104 independent reflections
1918 reflections with $I > 2\sigma(I)$
$R_{int} = 0.057$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.8°$
$h = -34 \rightarrow 33$
$k = -7 \rightarrow 7$
$l = -19 \rightarrow 27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.121$
$S = 1.00$
3104 reflections
209 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 atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ 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}
P1	0.17738 (3)	0.87761 (11)	0.22830 (4)	0.0315 (2)
O3	0.11541 (7)	0.8149 (3)	0.20942 (10)	0.0399 (5)
N1	0.23047 (10)	0.8251 (4)	0.15689 (12)	0.0303 (5)
O2	0.22470 (8)	0.7684 (3)	0.29547 (9)	0.0393 (5)
C18	0.17490 (11)	0.7697 (4)	0.14969 (13)	0.0301 (6)
H18	0.1735	0.6094	0.1520	0.036*
C8	0.12149 (11)	0.8387 (4)	0.07710 (13)	0.0315 (6)
O1	0.17840 (8)	1.1221 (3)	0.22305 (10)	0.0376 (5)
O4	0.15309 (10)	1.2130 (3)	0.08789 (11)	0.0586 (6)
H4A	0.1652	1.2047	0.1309	0.088*
C2	0.28397 (12)	0.7998 (5)	0.09779 (14)	0.0378 (7)
C10	0.06502 (13)	1.1018 (5)	−0.01849 (15)	0.0448 (7)
H10	0.0610	1.2415	−0.0376	0.054*
C1	0.23072 (12)	0.7379 (5)	0.09411 (15)	0.0424 (7)
H1A	0.2277	0.5791	0.0932	0.051*
H1B	0.1971	0.7936	0.0492	0.051*
C13	0.07828 (12)	0.6856 (5)	0.03567 (15)	0.0399 (7)
H13	0.0826	0.5438	0.0535	0.048*
C9	0.11301 (12)	1.0518 (4)	0.04989 (15)	0.0375 (7)
C11	0.02300 (13)	0.9456 (5)	−0.05861 (16)	0.0474 (8)
H11	−0.0095	0.9812	−0.1042	0.057*
C7	0.32831 (13)	0.6516 (5)	0.12183 (16)	0.0505 (8)
H7	0.3259	0.5144	0.1382	0.061*
C12	0.02899 (12)	0.7382 (5)	−0.03144 (16)	0.0451 (8)
H12	0.0002	0.6339	−0.0578	0.054*
C5	0.38007 (15)	0.9030 (7)	0.09799 (19)	0.0648 (10)
H5	0.4122	0.9375	0.0978	0.078*
C3	0.28884 (14)	1.0027 (5)	0.07376 (17)	0.0511 (8)
H3A	0.2595	1.1056	0.0572	0.061*
C14	0.10333 (14)	0.5962 (5)	0.2229 (2)	0.0629 (10)

H14A	0.1335	0.5506	0.2718	0.075*
H14B	0.1024	0.4934	0.1890	0.075*
C15	0.04702 (15)	0.5964 (7)	0.2139 (2)	0.0851 (13)
H15A	0.0485	0.6956	0.2483	0.128*
H15B	0.0386	0.4511	0.2220	0.128*
H15C	0.0173	0.6426	0.1655	0.128*
C4	0.33690 (16)	1.0534 (6)	0.07413 (19)	0.0635 (10)
H4	0.3399	1.1903	0.0581	0.076*
C6	0.37635 (15)	0.7037 (6)	0.12200 (19)	0.0648 (10)
H6	0.4060	0.6021	0.1386	0.078*
H1NA	0.2628 (13)	0.754 (4)	0.1997 (16)	0.050 (9)*
H1NB	0.2378 (11)	0.963 (4)	0.1627 (14)	0.033 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0278 (4)	0.0342 (4)	0.0320 (4)	−0.0001 (3)	0.0167 (3)	−0.0020 (3)
O3	0.0304 (11)	0.0432 (12)	0.0473 (12)	−0.0015 (9)	0.0227 (10)	0.0046 (9)
N1	0.0311 (14)	0.0301 (14)	0.0293 (13)	0.0002 (11)	0.0169 (12)	−0.0005 (11)
O2	0.0338 (11)	0.0488 (12)	0.0306 (10)	0.0068 (9)	0.0155 (9)	0.0055 (9)
C18	0.0285 (15)	0.0256 (14)	0.0321 (15)	0.0010 (11)	0.0147 (13)	0.0016 (11)
C8	0.0306 (15)	0.0350 (16)	0.0265 (14)	0.0032 (12)	0.0147 (12)	−0.0019 (12)
O1	0.0369 (11)	0.0333 (11)	0.0418 (11)	−0.0045 (9)	0.0218 (10)	−0.0078 (9)
O4	0.0591 (15)	0.0372 (12)	0.0452 (13)	−0.0073 (11)	0.0090 (12)	0.0035 (10)
C2	0.0379 (17)	0.0472 (18)	0.0300 (15)	−0.0016 (14)	0.0202 (14)	−0.0071 (13)
C10	0.0451 (19)	0.0437 (18)	0.0351 (16)	0.0101 (15)	0.0164 (15)	0.0067 (14)
C1	0.0429 (18)	0.0532 (19)	0.0337 (16)	−0.0061 (14)	0.0232 (15)	−0.0111 (14)
C13	0.0319 (17)	0.0465 (18)	0.0390 (16)	−0.0002 (14)	0.0187 (14)	−0.0035 (14)
C9	0.0333 (17)	0.0368 (17)	0.0346 (15)	0.0021 (13)	0.0145 (13)	−0.0016 (13)
C11	0.0347 (18)	0.066 (2)	0.0312 (16)	0.0105 (16)	0.0126 (14)	−0.0002 (16)
C7	0.058 (2)	0.055 (2)	0.0491 (19)	0.0096 (17)	0.0372 (18)	0.0018 (16)
C12	0.0272 (17)	0.060 (2)	0.0375 (17)	−0.0078 (14)	0.0117 (14)	−0.0112 (15)
C5	0.054 (2)	0.096 (3)	0.061 (2)	−0.021 (2)	0.043 (2)	−0.016 (2)
C3	0.057 (2)	0.0476 (19)	0.0494 (19)	0.0017 (16)	0.0301 (17)	−0.0012 (16)
C14	0.054 (2)	0.068 (2)	0.064 (2)	−0.0147 (18)	0.0314 (19)	0.0084 (19)
C15	0.046 (2)	0.119 (3)	0.081 (3)	−0.016 (2)	0.031 (2)	0.024 (2)
C4	0.078 (3)	0.068 (2)	0.062 (2)	−0.020 (2)	0.049 (2)	−0.0062 (19)
C6	0.054 (2)	0.087 (3)	0.066 (2)	0.016 (2)	0.042 (2)	−0.001 (2)

Geometric parameters (Å, º)

P1—O2	1.4841 (18)	C1—H1B	0.9700
P1—O1	1.4961 (18)	C13—C12	1.384 (4)
P1—O3	1.5874 (18)	C13—H13	0.9300
P1—C18	1.839 (3)	C11—C12	1.370 (4)
O3—C14	1.448 (3)	C11—H11	0.9300
N1—C1	1.503 (3)	C7—C6	1.383 (4)
N1—C18	1.513 (3)	C7—H7	0.9300

N1—H1NA	0.97 (3)	C12—H12	0.9300
N1—H1NB	0.86 (3)	C5—C6	1.355 (5)
C18—C8	1.517 (3)	C5—C4	1.364 (5)
C18—H18	0.9800	C5—H5	0.9300
C8—C13	1.388 (3)	C3—C4	1.380 (4)
C8—C9	1.396 (4)	C3—H3A	0.9300
O4—C9	1.367 (3)	C14—C15	1.477 (4)
O4—H4A	0.8200	C14—H14A	0.9700
C2—C7	1.377 (4)	C14—H14B	0.9700
C2—C3	1.384 (4)	C15—H15A	0.9600
C2—C1	1.498 (4)	C15—H15B	0.9600
C10—C11	1.380 (4)	C15—H15C	0.9600
C10—C9	1.382 (4)	C4—H4	0.9300
C10—H10	0.9300	C6—H6	0.9300
C1—H1A	0.9700		

O2—P1—O1	118.69 (11)	C8—C13—H13	119.1
O2—P1—O3	112.33 (10)	O4—C9—C10	118.2 (3)
O1—P1—O3	106.60 (10)	O4—C9—C8	121.3 (2)
O2—P1—C18	109.48 (11)	C10—C9—C8	120.4 (3)
O1—P1—C18	105.86 (11)	C12—C11—C10	120.2 (3)
O3—P1—C18	102.51 (11)	C12—C11—H11	119.9
C14—O3—P1	121.11 (18)	C10—C11—H11	119.9
C1—N1—C18	111.8 (2)	C2—C7—C6	121.0 (3)
C1—N1—H1NA	105.4 (16)	C2—C7—H7	119.5
C18—N1—H1NA	109.7 (16)	C6—C7—H7	119.5
C1—N1—H1NB	111.2 (17)	C11—C12—C13	119.3 (3)
C18—N1—H1NB	112.3 (17)	C11—C12—H12	120.3
H1NA—N1—H1NB	106 (2)	C13—C12—H12	120.3
N1—C18—C8	112.8 (2)	C6—C5—C4	120.4 (3)
N1—C18—P1	109.82 (16)	C6—C5—H5	119.8
C8—C18—P1	113.68 (17)	C4—C5—H5	119.8
N1—C18—H18	106.7	C4—C3—C2	120.6 (3)
C8—C18—H18	106.7	C4—C3—H3A	119.7
P1—C18—H18	106.7	C2—C3—H3A	119.7
C13—C8—C9	117.7 (2)	O3—C14—C15	109.2 (3)
C13—C8—C18	119.2 (2)	O3—C14—H14A	109.8
C9—C8—C18	123.0 (2)	C15—C14—H14A	109.8
C9—O4—H4A	109.5	O3—C14—H14B	109.8
C7—C2—C3	118.1 (3)	C15—C14—H14B	109.8
C7—C2—C1	120.6 (3)	H14A—C14—H14B	108.3
C3—C2—C1	121.2 (3)	C14—C15—H15A	109.5
C11—C10—C9	120.4 (3)	C14—C15—H15B	109.5
C11—C10—H10	119.8	H15A—C15—H15B	109.5
C9—C10—H10	119.8	C14—C15—H15C	109.5
C2—C1—N1	113.0 (2)	H15A—C15—H15C	109.5
C2—C1—H1A	109.0	H15B—C15—H15C	109.5
N1—C1—H1A	109.0	C5—C4—C3	120.1 (3)

C2—C1—H1B	109.0	C5—C4—H4	120.0
N1—C1—H1B	109.0	C3—C4—H4	120.0
H1A—C1—H1B	107.8	C5—C6—C7	119.8 (3)
C12—C13—C8	121.8 (3)	C5—C6—H6	120.1
C12—C13—H13	119.1	C7—C6—H6	120.1

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*NA*···O1^{i}	0.97 (3)	1.77 (3)	2.738 (3)	179 (2)
N1—H1*NB*···O4	0.86 (3)	2.50 (3)	2.982 (3)	116 (2)
N1—H1*NB*···O2^{ii}	0.86 (3)	2.08 (3)	2.915 (3)	164 (2)
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Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+1/2; (ii) −*x*+1/2, *y*+1/2, −*z*+1/2.