C12	0.5084 (6)	0.2260 (6)	0.1808 (6)	0.036 (2)
C13	0.6092 (6)	0.1548 (6)	0.2301 (6)	0.029 (2)
C14	0.7637 (6)	0.1026 (6)	0.1633 (6)	0.030 (2)
N4	0.7900 (5)	0.0763 (5)	0.0820 (4)	0.030 (2)
O	0.5999 (4)	0.0007 (4)	−0.0357 (4)	0.0404 (14)
C15	0.7015 (6)	0.0347 (7)	−0.0193 (6)	0.031 (2)
C16	0.7396 (6)	0.0333 (6)	−0.1109 (5)	0.028 (2)
C17	0.8559 (6)	0.0565 (6)	−0.0912 (5)	0.035 (2)
C18	0.8909 (6)	0.0484 (7)	−0.1774 (6)	0.041 (2)
C19	0.8059 (7)	0.0215 (7)	−0.2863 (6)	0.043 (2)
C20	0.6886 (7)	0.0013 (6)	−0.3056 (6)	0.046 (2)
C21	0.6545 (6)	0.0052 (6)	−0.2205 (6)	0.038 (2)

Table 2. *Selected geometric parameters* (*Å, °*)

S—C7	1.737 (6)	N3—C13	1.408 (8)
S—C8	1.768 (7)	C1—C6	1.382 (8)
N1—C7	1.301 (7)	C8—C13	1.382 (9)
N1—C1	1.406 (8)	C14—N4	1.294 (8)
N2—C6	1.399 (8)	N4—C15	1.380 (8)
N2—C14	1.406 (7)	O—C15	1.237 (7)
N2—C7	1.412 (7)	C15—C16	1.485 (8)
N3—C14	1.356 (8)		
C7—S—C8	97.7 (3)	N2—C7—S	123.6 (5)
C7—N1—C1	105.0 (5)	C13—C8—S	120.8 (5)
C6—N2—C14	125.7 (5)	C8—C13—N3	121.4 (7)
C6—N2—C7	105.6 (5)	N4—C14—N3	127.3 (6)
C14—N2—C7	127.1 (6)	N4—C14—N2	115.8 (6)
C14—N3—C13	130.8 (6)	N3—C14—N2	116.9 (6)
C6—C1—N1	111.0 (6)	C14—N4—C15	120.4 (5)
C1—C6—N2	105.5 (6)	O—C15—N4	125.3 (6)
N1—C7—N2	112.8 (5)	O—C15—C16	121.5 (6)
N1—C7—S	123.5 (5)	N4—C15—C16	113.2 (6)
C14—N2—C7—S	−11.3 (10)	C8—S—C7—N2	−55.4 (6)
C7—S—C8—C13	60.3 (6)	S—C8—C13—N3	1.0 (10)
C14—N3—C13—C8	−60.5 (12)	C13—N3—C14—N2	17.2 (12)
C7—N2—C14—N3	44.1 (10)		

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1101). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Ethyl {[*N*-(2-Hydroxyethyl)amino](2-hydroxy-1-naphthyl)methyl}phosphonate

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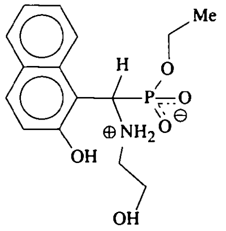
Abstract

The title molecule, $C_{15}H_{20}NO_5P$, has a zwitterionic structure with strong intermolecular N···O and O···O hydrogen bonds. The coordination around phosphorus is distorted tetrahedral, with a maximum bond-angle deviation of 12° from the ideal. The P—C bond length is 1.860 (4) Å and the average P—O value is 1.518 (3) Å.

Comment

Aminophosphonic acids are currently the subject of much interest because of their potential antibacterial (van Assche *et al.*, 1991), antifungal (Cameron, Hudson & Pianka, 1993) and anticancer activities (Lavielle *et al.*, 1991). These compounds may also be of use in metal extraction processes (Aguilar, Miralles & Sastre, 1989). The present structural analysis of the title compound, (I), has been performed in order to determine whether the molecule has distinct acidic -POH and basic -NH- groups or is a zwitterionic structure with $-NH_2^+$ and $-PO^-$ groups.



(I)

As can be seen from Fig. 1, the molecule is a zwitterion. The P1—O3 and P1—O4 bond lengths (Table 2) are almost equal within experimental error and are comparable to reported P—O bond lengths for similar structures (Gibson & Karaman, 1989). They are longer than the P═O double-bond values [1.466 (5) Å] given for some phosphonic acids (Toroš, Prodić & Šljukić 1978; Choi, Failla, Finocchiaro, McPartlin & Scowen, 1994). The P1—O5 and P1—C11 separations are 1.584 (3) and

1.860 (4) Å, respectively. The coordination around the P atom is distorted tetrahedral, with O—P—O angles in the range 105.8 (2)–121.5 (2)° and O—P—C angles in the range 105.0 (2)–108.4 (2)°. The naphthyl group is planar with normal bond lengths and angles. There are fairly strong intermolecular hydrogen bonds, details of which are given in Table 3.

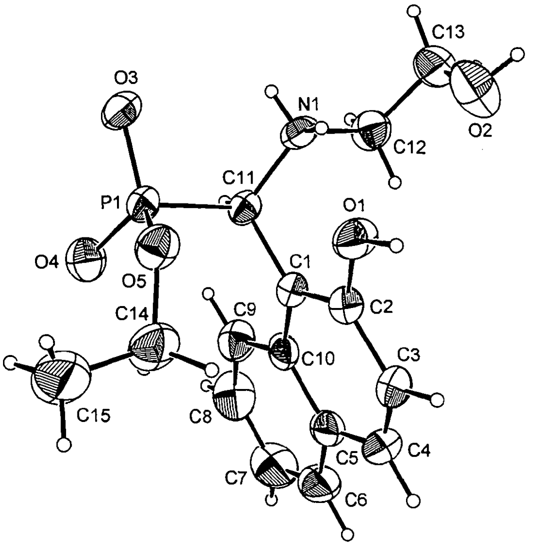


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of $C_{15}H_{20}NO_5P$ with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small circles with arbitrary radii.

Experimental

The title compound was prepared by refluxing a mixture of *N*-(2-hydroxyethyl)-2-hydroxynaphthaldimine (2.13 g, 10 mmol) and diethyl phosphite (1.41 g, 10 mmol, 98%) in 95% ethanol (30 h). The ethanol was then evaporated and the oily reaction product which solidified upon cooling was recrystallized from ethanol.

Crystal data

$C_{15}H_{20}NO_5P$
$M_r = 325.30$
Monoclinic
$P2_1/n$
$a = 6.990\,(3)$ Å
$b = 13.887\,(4)$ Å
$c = 16.389\,(3)$ Å
$\beta = 92.47\,(4)°$
$V = 1589.4\,(8)$ Å^3
$Z = 4$
$D_x = 1.359$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.13–18.14°$
$\mu = 0.19$ mm^{-1}
$T = 294$ K
Prismatic
0.35 × 0.20 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*MolEN*; Fair, 1990)
$T_{\min} = 0.977$, $T_{\max} = 0.999$
2932 measured reflections
2680 independent reflections

2036 observed reflections $[I > 3\sigma(I)]$
$R_{\rm int} = 0.018$
$\theta_{\max} = 25.01°$
$h = 0 \rightarrow 8$
$k = -16 \rightarrow 0$
$l = -19 \rightarrow 19$
3 standard reflections
frequency: 120 min
intensity decay: 0.28%

Refinement

Refinement on F
$R = 0.049$
$wR = 0.054$
$S = 1.24$
2036 reflections
199 parameters
H atoms: see below
Unit weights

$(\Delta/\sigma)_{\max} = 0.0004$
$\Delta\rho_{\max} = 0.283$ e Å^{-3}
$\Delta\rho_{\min} = -0.326$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
P1	0.9730 (1)	0.15377 (7)	0.92561 (6)	2.44 (2)
O1	0.4915 (3)	0.1428 (2)	0.8745 (2)	3.49 (6)
O2	0.3359 (5)	−0.0639 (2)	0.9130 (2)	5.34 (8)
O3	0.9978 (4)	0.1120 (2)	1.0090 (2)	3.34 (5)
O4	1.1396 (4)	0.1839 (2)	0.8789 (2)	3.64 (6)
O5	0.8351 (4)	0.2434 (2)	0.9341 (2)	3.37 (6)
N1	0.7249 (4)	−0.0041 (2)	0.9079 (2)	2.73 (6)
C1	0.7385 (5)	0.1062 (3)	0.7878 (2)	2.45 (7)
C2	0.5617 (5)	0.1463 (3)	0.7990 (2)	2.83 (7)
C3	0.4556 (5)	0.1900 (3)	0.7336 (3)	3.51 (9)
C4	0.5299 (6)	0.1940 (3)	0.6589 (3)	3.76 (9)
C5	0.7105 (6)	0.1542 (3)	0.6437 (2)	3.17 (8)
C6	0.7866 (7)	0.1564 (3)	0.5652 (2)	4.12 (9)
C7	0.9576 (7)	0.1165 (4)	0.5504 (3)	4.9 (1)
C8	1.0643 (7)	0.0728 (4)	0.6141 (3)	4.6 (1)
C9	0.9979 (6)	0.0698 (3)	0.6912 (3)	3.61 (9)
C10	0.8180 (5)	0.1090 (3)	0.7094 (2)	2.68 (7)
C11	0.8473 (5)	0.0620 (3)	0.8603 (2)	2.38 (7)
C12	0.6394 (6)	−0.0859 (3)	0.8595 (2)	3.34 (8)
C13	0.4824 (6)	−0.1326 (3)	0.9044 (3)	3.92 (9)
C14	0.8196 (7)	0.3195 (3)	0.8737 (3)	4.5 (1)
C15	0.9289 (9)	0.4054 (4)	0.9010 (4)	7.4 (2)

Table 2. *Selected geometric parameters (Å, °)*

P1—O3	1.488 (3)	C2—C3	1.415 (5)
P1—O4	1.482 (3)	C3—C4	1.350 (6)
P1—O5	1.584 (3)	C4—C5	1.411 (6)
P1—C11	1.860 (4)	C5—C6	1.414 (6)
O1—C2	1.351 (4)	C5—C10	1.431 (5)
O2—C13	1.411 (5)	C6—C7	1.349 (7)
O5—C14	1.449 (5)	C7—C8	1.396 (7)
N1—C11	1.497 (5)	C8—C9	1.365 (6)
N1—C12	1.495 (5)	C9—C10	1.415 (6)
C1—C2	1.375 (5)	C12—C13	1.496 (6)
C1—C10	1.421 (5)	C14—C15	1.476 (7)
C1—C11	1.514 (5)		
O3—P1—O4	121.5 (2)	C4—C5—C6	121.8 (4)
O3—P1—O5	105.8 (2)	C4—C5—C10	118.9 (4)
O3—P1—C11	107.2 (2)	C6—C5—C10	119.3 (4)
O4—P1—O5	108.5 (2)	C5—C6—C7	121.7 (4)
O4—P1—C11	105.0 (2)	C6—C7—C8	119.5 (4)
O5—P1—C11	108.4 (2)	C7—C8—C9	120.9 (5)
P1—O5—C14	122.7 (3)	C8—C9—C10	121.7 (4)

C11—N1—C12	114.4 (3)	C1—C10—C5	118.6 (4)
C2—C1—C10	119.9 (3)	C1—C10—C9	124.5 (4)
C2—C1—C11	118.9 (3)	C5—C10—C9	116.9 (4)
C10—C1—C11	121.2 (3)	P1—C11—N1	112.8 (2)
O1—C2—C1	118.2 (3)	P1—C11—C1	112.5 (3)
O1—C2—C3	120.7 (4)	N1—C11—C1	112.3 (3)
C1—C2—C3	121.1 (4)	N1—C12—C13	110.7 (3)
C2—C3—C4	119.7 (4)	O2—C13—C12	107.8 (4)
C3—C4—C5	121.8 (4)	O5—C14—C15	111.1 (4)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H12···O3^{i}	0.810	1.998	2.761 (4)	157.06
O1—H1···O4^{ii}	0.819	1.716	2.529 (3)	171.63
O2—H2···O3^{iii}	0.830	1.996	2.789 (5)	160.83

Symmetry codes: (i) $2-x, -y, 2-z$; (ii) $x-1, y, z$; (iii) $1-x, -y, 2-z$.

All non-H atoms were refined with anisotropic displacement parameters. The H atoms of the naphthyl group were placed geometrically 0.95 Å from their corresponding C atoms, while other H atoms were taken from a $\Delta\rho$ map. For all H atoms a riding model was used with $U_{\rm iso}(\rm H) = 1.3U_{\rm eq}(\rm C, N, O)$.

Data collection: *CAD*-4 *Express* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976) in *MolEN*. Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1384). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Diphenylphosphino-2-phenyl-1,2-dicarba-*closo*-dodecaborane(12)

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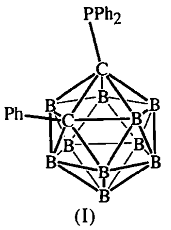
Abstract

The two crystallographically independent molecules of the title compound, $1\text{-}[(C_6H_5)_2P]\text{-}2\text{-}C_6H_5\text{-}1,2\text{-}C_2B_{10}H_{10}$ ($C_{20}H_{25}B_{10}P$), in the asymmetric fraction of the unit cell are highly congruent. The conformation of the cage phenyl groups is defined by $\theta = 1.15°$ and the average C1—C2 distance is 1.744 (8) Å, which is significantly longer than that usually found for $1,2\text{-}C_2B_{10}H_{10}$ compounds. The PPh_2 substituent is tilted away from an idealized radial position on C1 as a result of intramolecular crowding.

Comment

1-Diphenylphosphino-2-phenyl-1,2-dicarba-*closo*-dodecaborane(12), (I), is an exceptionally stable compound with the potential for extensive derivatization of both the phosphine moiety and the carborane polyhedron. It was synthesized and characterized in order to extend the series of structural determinations of carboryl phosphines (Kivekäs, Sillanpää, Teixidor, Viñas & Nuñez, 1994; Kivekäs, Teixidor, Viñas & Nuñez, 1995) and also to establish a structural basis for its subsequent use as a carborane-containing ligand (Reid, 1992).



(I)

There are two crystallographically independent molecules in the asymmetric fraction of the unit cell. A perspective view of one molecule (atom labels unprimed) is shown in Fig. 1 and Table 2 lists selected geometric parameters. There is a high degree of congruence between the two independent molecules, with