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Diethylammonium diphenylphosphinate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.106
Data-to-parameter ratio = 20.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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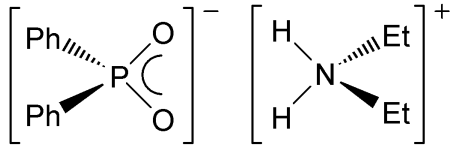
In the title compound, $C_4H_{12}N^+\cdot C_{12}H_{10}O_2P^-$, the $Et_2NH_2^+$ and $Ph_2PO_2^-$ moieties in the asymmetric unit are linked by P—O···H—N hydrogen bonds [O···N distance 2.6722 (15) Å]. These hydrogen-bonded pairs are in turn linked by intermolecular P—O···H—N hydrogen bonds [O···N distance 2.6943 (15) Å] to form infinite chains along 2_1 screw axes in the y direction.

Comment

Diethylammonium diphenylphosphinate, $[Et_2NH_2][Ph_2PO_2]$, (I) (Fig. 1), was synthesized during the course of our work on the preparation and reactivity of phosphinoborane compounds (Dorn *et al.*, 2000). The reaction between the phosphine–borane adduct Ph_2PHBH_3 and diethylamine (1:2 ratio) in dichloromethane under air yielded $[Et_2NH_2][Ph_2PO_2]$ as colourless crystals, along with the amine–borane adduct Et_2NHBH_3, which was detected by ^{11}B NMR spectroscopy. The breaking of the P—B bond in phosphine–borane adducts by amines is known; however, the subsequent oxidation of Ph_2PH and formation of the ammonium salt was unexpected. The title compound could also be synthesized directly from $Ph_2P(O)(OH)$ and Et_2NH in $CDCl_3$ solution, and was identified by ^{31}P NMR spectroscopy.



(I)

Experimental

Ph_2PHBH_3 (305 mg, 1.53 mmol) was dissolved in dichloromethane (5 ml) and Et_2NH (230 mg, 2.1 equivalents) was added *via* syringe at room temperature. The reaction mixture was stirred for 3 d, filtered and set aside for crystallization in air. Colourless crystals of $[Et_2NH_2][Ph_2PO_2]$ suitable for X-ray diffraction grew over a period of several days. ^1H NMR ($CDCl_3$, p.p.m.): δ 10.3 (*br*, 2H, NH), 7.76–7.22 (*m*, 10H, Ph), 2.77 (*q*, 4H, CH_2), 1.24 (*t*, 6H, CH_3); ^{31}P NMR: 19.3 p.p.m. (s).

Crystal data

$C_4H_{12}N^+\cdot C_{12}H_{10}O_2P^-$
M_r = 291.32
Monoclinic, $P2_1/c$
a = 12.4163 (2) Å
b = 8.3038 (2) Å
c = 16.4517 (3) Å
β = 106.949 (1)°
V = 1622.53 (6) Å^3
Z = 4
D_x = 1.193 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3745 reflections
θ = 2.6–27.5°
μ = 0.17 mm^{-1}
T = 150 (1) K
Needle, colourless
0.32 × 0.30 × 0.26 mm

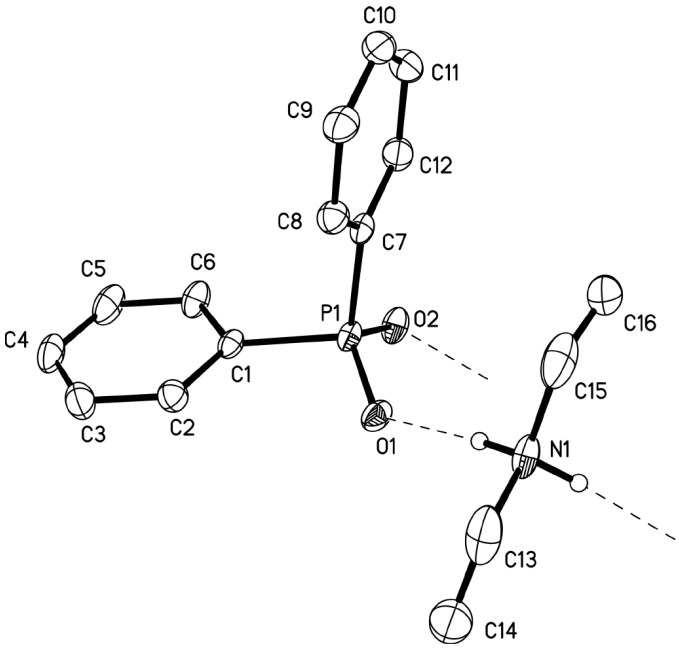


Figure 1
View of (I) showing the atom-labelling scheme. Displacement ellipsoids are at the 50% probability level and H atoms bonded to C atoms have been omitted for clarity.

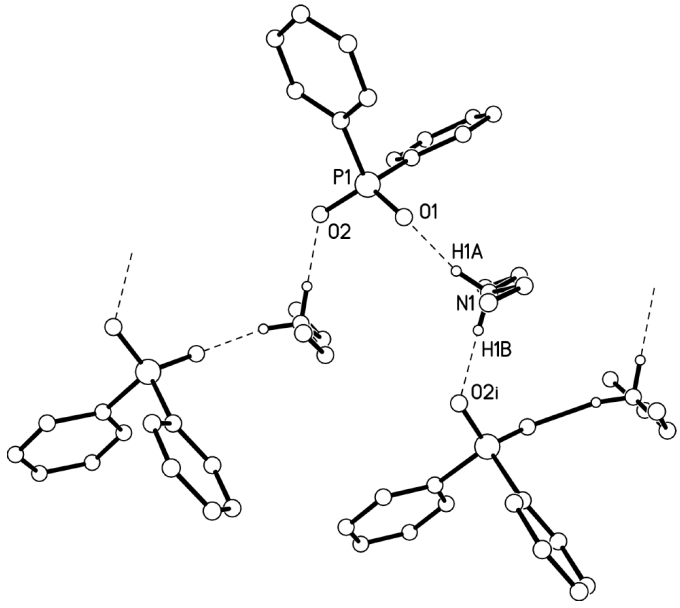


Figure 2
View of the hydrogen bonding in (I). H atoms bonded to C atoms have been omitted for clarity.

Data collection

Nonius Kappa–CCD diffractometer	3705 independent reflections
φ scans, and ω scans with κ offsets	3236 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)	$R_{int} = 0.026$
	$\theta_{max} = 27.5^\circ$
$T_{min} = 0.947$, $T_{max} = 0.957$	$h = -16 \rightarrow 16$
13 875 measured reflections	$k = -10 \rightarrow 10$
	$l = -21 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.7570P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.040$	
$wR(F^2) = 0.106$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.03$	$\Delta\rho_{max} = 0.36$ e Å^{-3}
3705 reflections	$\Delta\rho_{min} = -0.34$ e Å^{-3}
181 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

P1—O1	1.5027 (10)	N1—C13	1.475 (2)
P1—O2	1.5046 (10)	N1—C15	1.503 (2)
O1—P1—O2	118.54 (6)	O2—P1—C7	108.01 (6)
O1—P1—C1	107.63 (6)	C1—P1—C7	103.72 (6)
O2—P1—C1	109.08 (6)	C13—N1—C15	114.47 (16)
O1—P1—C7	108.86 (6)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1	0.92	1.77	2.6722 (15)	166
N1—H1B···O2^{i}	0.92	1.78	2.6943 (15)	176

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

H atoms were included in calculated positions, with C—H distances ranging from 0.95 to 0.99 Å and N—H distances of 0.92 Å.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1999); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

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