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Diethyl 5-Bis(diphenylphosphinoyl)methyl-2-hydroxy-3-*tert*-butylphenylphosphonate Hydrate at 173 K

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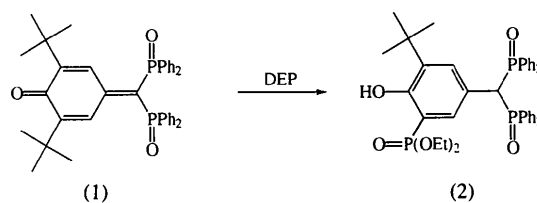
Abstract

The title compound, $C_{39}H_{43}O_6P_3 \cdot H_2O$, shows an intramolecular hydrogen bond between the O1 and O2 atoms. It interacts *via* two further intermolecular hydrogen bonds involving one water molecule.

Comment

The addition of diethylphosphite (DEP) to phosphino-oxidoquinonmethide, (1), by splitting off isobutene, led to the phosphorylated aromatic product, (2). The structure of (2) was proven by detailed NMR studies (Gross, Keitel & Costisella, 1991). Surprisingly, the ¹H NMR signal of the OH group was shifted strongly to lower field compared with very similar compounds (Gross, Keitel & Costisella, 1991). The shift to lower field can be explained by the neighbourhood of the electron-withdrawing phosphonate group and the possibility of an

intramolecular hydrogen bond between the OH proton and the phosphoryl O atom of the phosphonate group. The aim of the present structure analysis was therefore to prove whether the presumed intramolecular hydrogen bond exists or not.



Previous X-ray structure analyses of related compounds at room temperature (Ramm & Schulz, 1990; Ramm, 1993) showed that due to high thermal motion the positions of the O—C₂H₅ groups could not be determined accurately. The data collection was therefore performed at 173 K. The P atoms form distorted tetrahedra. They are coordinated either by three O atoms and one C atom (P1) or by one O atom and three C atoms (P2 and P3). The tetrahedral angles around P2 and P3 show the same systematic deviations from the ideal value. The three angles enclosing the P=O double bond are larger, the other three being smaller. This corresponds to other phosphorus-containing compounds (Kulpe & Seidel, 1979; Ramm, Costisella & Gross, 1995). Only the O2—P1—O3 bond angle deviates from this array. This can be attributed to the participation of the O2 atom in an intramolecular hydrogen bond with O1 resulting in a six-membered ring [O1...O2

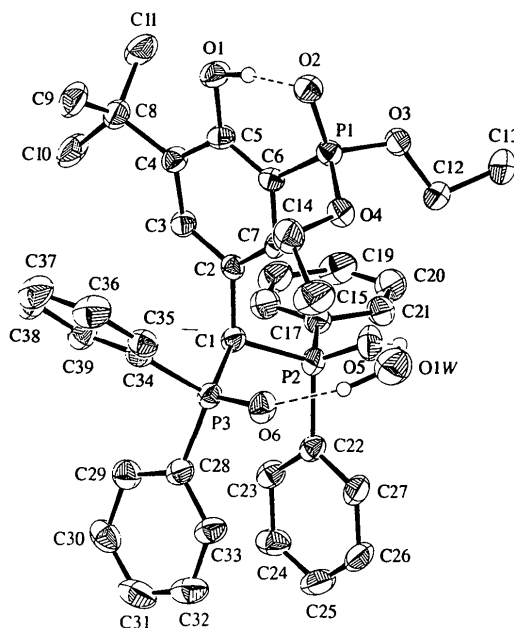


Fig. 1. ORTEP (Johnson, 1971) drawing of the asymmetric unit of the title compound showing 50% probability displacement ellipsoids. Only H atoms participating in hydrogen bonds are drawn. Dashed lines represent hydrogen bonds.

2.639 (4), H1O...O2 1.69 (5) Å and O1—H1O...O2 174 (6)°. The water molecule interacts *via* two intermolecular hydrogen bonds with the C₃₉H₄₃O₆P₃ molecule; O1W...O5 2.715 (5), H1OW...O5 1.739 (3) Å and O1W—H1OW...O5 145.1 (2)°; O1W...O6 2.825 (5), H2OW...O6 1.759 (3) Å and O1W—H2OW...O6 158.7 (2)°. An eight-membered ring is formed in this way. Surprisingly, these hydrogen bonds do not influence the systematic deviations of the bond angles around the P2 and P3 atoms.

Experimental

The title compound was recrystallized from ethyl acetate.

Crystal data

C₃₉H₄₃O₆P₃·H₂O

M_r = 718.70

Orthorhombic

P2₁2₁2₁

a = 16.233 (2) Å

b = 17.313 (2) Å

c = 13.300 (1) Å

V = 3737.7 (7) Å³

Z = 4

D_x = 1.277 Mg m⁻³

D_m not measured

Cu Kα radiation

λ = 1.54178 Å

Cell parameters from 25 reflections

θ = 36–46°

μ = 1.85 mm⁻¹

T = 173 K

Plate

0.32 × 0.32 × 0.14 mm

Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer

ω/2θ scans

Absorption correction: none

3774 measured reflections

3774 independent reflections

3443 observed reflections

[*F* > 3σ(*F*)]

θ_{max} = 70°

h = 0 → 19

k = -18 → 0

l = -16 → 0

2 standard reflections

frequency: 60 min

intensity decay: 4.0%

Refinement

Refinement on *F*

R = 0.044

wR = 0.059

S = 2.274

3443 reflections

617 parameters

All H atoms, located in the difference synthesis, were refined, except for H1OW and H2OW for which only *U*'s were refined

w = 1/σ²(*F*)

(Δ/σ)_{max} = 0.15

Δρ_{max} = 0.270 e Å⁻³

Δρ_{min} = -0.092 e Å⁻³

Extinction correction:

Zachariasen (1963)

Extinction coefficient:

5.47333 × 10⁻⁷

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

O1W	0.2803 (2)	0.5583 (2)	0.4178 (3)	0.068 (1)
O2	-0.0222 (2)	0.4045 (2)	0.2697 (2)	0.0350 (8)
O3	0.0322 (2)	0.4427 (2)	0.4369 (2)	0.0322 (7)
O4	0.1304 (2)	0.4412 (2)	0.2979 (2)	0.0320 (7)
O5	0.1816 (2)	0.6726 (2)	0.4886 (2)	0.0417 (9)
O6	0.2722 (2)	0.6654 (2)	0.2579 (2)	0.0368 (8)
C1	0.1302 (2)	0.7479 (2)	0.3126 (3)	0.028 (1)
C2	0.0638 (2)	0.6872 (2)	0.2941 (2)	0.026 (1)
C3	-0.0099 (2)	0.7078 (2)	0.2476 (3)	0.028 (1)
C4	-0.0698 (2)	0.6553 (2)	0.2182 (3)	0.029 (1)
C5	-0.0548 (2)	0.5762 (2)	0.2414 (3)	0.029 (1)
C6	0.0177 (2)	0.5547 (2)	0.2912 (3)	0.0264 (9)
C7	0.0775 (2)	0.6099 (2)	0.3150 (3)	0.027 (1)
C8	-0.1474 (3)	0.6789 (2)	0.1592 (3)	0.037 (1)
C9	-0.1467 (3)	0.6394 (3)	0.0566 (3)	0.050 (2)
C10	-0.1482 (3)	0.7665 (3)	0.1417 (4)	0.053 (2)
C11	-0.2257 (3)	0.6589 (3)	0.2174 (4)	0.050 (2)
C12	0.0898 (3)	0.4756 (3)	0.5092 (3)	0.042 (1)
C13	0.0713 (3)	0.4396 (4)	0.6081 (4)	0.067 (2)
C14	0.1608 (3)	0.4428 (3)	0.1950 (3)	0.046 (1)
C15	0.2531 (3)	0.4332 (4)	0.2004 (4)	0.065 (2)
C16	0.0797 (2)	0.7950 (3)	0.5145 (3)	0.034 (1)
C17	0.0251 (3)	0.8476 (3)	0.4754 (3)	0.040 (1)
C18	-0.0321 (3)	0.8830 (3)	0.5383 (3)	0.048 (1)
C19	-0.0331 (3)	0.8661 (3)	0.6395 (3)	0.052 (2)
C20	0.0218 (3)	0.8138 (3)	0.6795 (3)	0.051 (2)
C21	0.0780 (3)	0.7779 (3)	0.6173 (3)	0.042 (1)
C22	0.2467 (2)	0.8158 (2)	0.4593 (3)	0.030 (1)
C23	0.2372 (2)	0.8947 (3)	0.4410 (3)	0.033 (1)
C24	0.3036 (3)	0.9455 (3)	0.4554 (3)	0.043 (1)
C25	0.3777 (3)	0.9159 (3)	0.4888 (3)	0.048 (1)
C26	0.3877 (2)	0.8385 (3)	0.5065 (3)	0.046 (1)
C27	0.3227 (2)	0.7879 (3)	0.4911 (3)	0.038 (1)
C28	0.2737 (2)	0.8202 (2)	0.2144 (3)	0.032 (1)
C29	0.2373 (3)	0.8889 (3)	0.1809 (3)	0.037 (1)
C30	0.2852 (3)	0.9549 (3)	0.1707 (3)	0.043 (1)
C31	0.3685 (3)	0.9527 (3)	0.1947 (3)	0.050 (1)
C32	0.4032 (3)	0.8855 (3)	0.2282 (4)	0.047 (1)
C33	0.3574 (2)	0.8194 (3)	0.2376 (3)	0.038 (1)
C34	0.1699 (2)	0.7108 (2)	0.1063 (3)	0.031 (1)
C35	0.1929 (2)	0.6425 (3)	0.0588 (3)	0.035 (1)
C36	0.1599 (3)	0.6220 (3)	-0.0327 (3)	0.049 (1)
C37	0.1027 (3)	0.6720 (3)	-0.0780 (3)	0.052 (1)
C38	0.0788 (3)	0.7390 (3)	-0.0306 (3)	0.047 (1)
C39	0.1118 (3)	0.7594 (3)	0.0606 (3)	0.039 (1)

Table 2. Geometric parameters (Å, °)

P1—O2	1.488 (3)	C12—C13	1.486 (7)
P1—O3	1.564 (3)	C14—C15	1.509 (7)
P1—O4	1.562 (3)	C16—C17	1.373 (7)
P1—C6	1.774 (4)	C16—C21	1.399 (6)
P2—O5	1.475 (3)	C17—C18	1.392 (7)
P2—C1	1.851 (4)	C18—C19	1.377 (6)
P2—C16	1.803 (4)	C19—C20	1.377 (7)
P2—C22	1.801 (4)	C20—C21	1.379 (7)
P3—O6	1.494 (3)	C22—C23	1.396 (6)
P3—C1	1.847 (4)	C22—C27	1.391 (5)
P3—C28	1.806 (4)	C23—C24	1.404 (6)
P3—C34	1.810 (4)	C24—C25	1.381 (7)
O1—C5	1.351 (4)	C25—C26	1.370 (7)
O3—C12	1.457 (5)	C26—C27	1.387 (6)
O4—C14	1.455 (5)	C28—C29	1.401 (6)
C1—C2	1.525 (5)	C28—C33	1.393 (5)
C2—C3	1.393 (5)	C29—C30	1.389 (7)
C2—C7	1.385 (5)	C30—C31	1.390 (7)
C3—C4	1.387 (5)	C31—C32	1.367 (7)
C4—C5	1.425 (5)	C32—C33	1.370 (7)
C4—C8	1.539 (6)	C34—C35	1.392 (6)
C5—C6	1.401 (5)	C34—C39	1.402 (6)
C6—C7	1.398 (5)	C35—C36	1.376 (6)
C8—C9	1.526 (6)	C36—C37	1.405 (7)
C8—C10	1.534 (6)	C37—C38	1.376 (7)
C8—C11	1.528 (7)	C38—C39	1.372 (6)
O2—P1—O3	108.9 (2)	C4—C8—C11	111.2 (3)
O2—P1—O4	115.8 (2)	C9—C8—C10	107.9 (4)
O2—P1—C6	111.2 (2)	C9—C8—C11	111.0 (4)
O3—P1—O4	102.6 (1)	C10—C8—C11	107.1 (4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
P1	0.03744 (6)	0.45655 (6)	0.32086 (7)	0.0281 (3)
P2	0.16220 (6)	0.74919 (6)	0.44614 (7)	0.0297 (3)
P3	0.21762 (6)	0.73058 (6)	0.22661 (7)	0.0276 (3)
O1	-0.1130 (2)	0.52400 (2)	0.2160 (2)	0.0374 (8)

O3—P1—C6	110.9 (2)	O3—C12—C13	106.9 (4)
O4—P1—C6	107.1 (2)	O4—C14—C15	106.8 (3)
O5—P2—C1	114.6 (2)	P2—C16—C17	125.5 (3)
O5—P2—C16	111.2 (2)	P2—C16—C21	114.5 (3)
O5—P2—C22	112.0 (2)	C17—C16—C21	119.8 (4)
C1—P2—C16	106.3 (2)	C16—C17—C18	119.7 (4)
C1—P2—C22	108.4 (2)	C17—C18—C19	120.1 (4)
C16—P2—C22	103.6 (2)	C18—C19—C20	120.6 (4)
O6—P3—C1	114.0 (2)	C19—C20—C21	119.5 (4)
O6—P3—C28	112.0 (2)	C16—C21—C20	120.2 (4)
O6—P3—C34	110.9 (2)	P2—C22—C23	121.7 (3)
C1—P3—C28	107.7 (2)	P2—C22—C27	118.9 (3)
C1—P3—C34	104.4 (2)	C23—C22—C27	119.4 (3)
C28—P3—C34	107.4 (2)	C22—C23—C24	120.3 (4)
P1—O3—C12	123.8 (3)	C23—C24—C25	118.7 (5)
P1—O4—C14	120.5 (3)	C24—C25—C26	121.4 (4)
P2—C1—P3	112.4 (2)	C25—C26—C27	120.2 (4)
P2—C1—C2	111.2 (2)	C22—C27—C26	120.0 (4)
P3—C1—C2	109.3 (2)	P3—C28—C29	123.1 (3)
C1—C2—C3	120.1 (3)	P3—C28—C33	117.6 (3)
C1—C2—C7	121.3 (3)	C29—C28—C33	119.4 (4)
C3—C2—C7	118.3 (3)	C28—C29—C30	119.6 (4)
C2—C3—C4	124.0 (3)	C29—C30—C31	120.0 (5)
C3—C4—C5	116.7 (3)	C30—C31—C32	119.9 (5)
C3—C4—C8	122.9 (3)	C31—C32—C33	121.1 (4)
C5—C4—C8	120.4 (3)	C28—C33—C32	120.0 (4)
O1—C5—C4	118.0 (3)	P3—C34—C35	116.6 (3)
O1—C5—C6	121.9 (3)	P3—C34—C39	123.9 (3)
C4—C5—C6	120.1 (3)	C35—C34—C39	119.6 (4)
P1—C6—C5	120.8 (3)	C34—C35—C36	121.1 (4)
P1—C6—C7	118.6 (3)	C35—C36—C37	118.5 (4)
C5—C6—C7	120.6 (3)	C36—C37—C38	120.6 (4)
C2—C7—C6	120.2 (3)	C37—C38—C39	120.8 (4)
C4—C8—C9	109.3 (3)	C34—C39—C38	119.4 (4)
C4—C8—C10	110.3 (3)		

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN11/82* (Main *et al.*, 1982). Program(s) used to refine structure: *LSFM* in *MolEN*. Software used to prepare material for publication: *PLATON* (Spek, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: KA1188). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Isopropylamidinium Chloride

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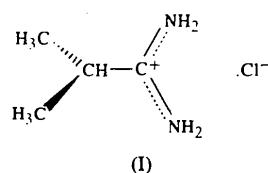
(Received 8 August 1994; accepted 27 February 1996)

Abstract

The amidine fragment in each of the two independent molecules of $C_4H_{11}N_2^+ \cdot Cl^-$ is essentially planar; the bond angles around the central C atom sum to $360.0(4)^\circ$ in each case. This, and the almost equal C—N bond lengths [1.299(6) to 1.320(5) Å], indicate that an amidinium salt has been formed.

Comment

A number of papers concerning the structures of amidines have appeared (Tykarska, Jaskolski & Kos-turkiewicz, 1986; Thailambal, Pattabhi & Guru Row, 1986; Barker, Gould & Kilner, 1987; Barker & Kilner, 1993; Alcock, Barker & Kilner, 1988; Alcock, Barker, Blacker, Errington & Wallbridge, 1994; Dehnicke, 1990), reflecting the widespread interest in such compounds because of their pharmaceutical and biological importance, bonding characteristics and ligand properties. The structural investigation of the title compound, (I), was undertaken to investigate the effect of alkyl substitution on the central amidine fragment.



The asymmetric unit consists of two molecules with N—H...Cl hydrogen bonding present (Fig. 1), as it is throughout the structure (Fig. 2). The skeletal C—N bond lengths are clearly not differentiated in terms of single- or double-bond character, with mean C—N bond lengths of 1.317(5) and 1.300(6) Å for each molecule, respectively. They are similar to those found for acetamidinium chloride [1.305(2) and 1.310(3) Å; Cannon, White & Willis, 1976] and bis(acetamidinium)-carbonate monohydrate [1.308(5) and 1.315 Å; Norrestam, 1984]. They differ somewhat to those found for acetamidine [1.298(1) and 1.344(1) Å; Norrestam, Mertz & Crossland, 1983], *N,N*-diphenylbenzamidine [1.302(7) and 1.360(8) Å; Alcock *et al.*, 1988] and