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N,N-Dimethyl-*N*′,*N*″-bis(4-methylphenyl)-phosphoramidate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.156
Data-to-parameter ratio = 18.5

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

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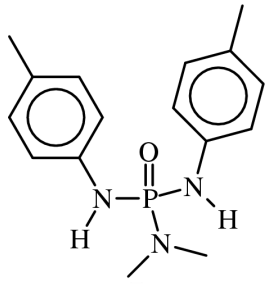
Adjacent molecules of the title compound [alternative name: phosphoric bis(4-methylbenzamide) dimethylamide], $C_{16}H_{22}N_3OP$, with tetrahedral phosphorus, are linked by twin N—H···O [N···O = 2.958 (3) Å] hydrogen bonds into a linear chain.

Comment

The title compound, (I), belongs to a class of amides of arylphosphonic acid that is expected to have antibacterial properties (Doak & Freedman, 1954). An earlier study of bis(4-nitrophenyl)-*N,N*-dimethylphosphoramidate confirmed the double-bond character of the P—N bond to the dimethylamino group [P—N = 1.603 (2) Å; Gholivand *et al.*, 2001]. The title compound (Fig. 1) possesses no electron-withdrawing groups, so that the P—N bond does not show this feature [P—N = 1.642 (2) Å] although the dimethylamino entity itself is planar, as it lies on a crystallographic mirror plane. Adjacent molecules are linked by a pair of N—H···O hydrogen bonds to give a linear chain running along the *a* axis (Fig. 2).



(I)

Experimental

The compound was synthesized from the reaction of 4-toluidine and *N,N*-dimethylaminophosphoryl dichloride in toluene at 268 K. Crystals were obtained by recrystallization of the product from chloroform/ethanol (1:1).

Crystal data

$C_{16}H_{22}N_3OP$
M_r = 303.34
Orthorhombic, *Pnam*
a = 9.305 (9) Å
b = 10.101 (2) Å
c = 17.454 (2) Å
V = 1641 (2) Å^3
Z = 4
D_x = 1.228 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 25 reflections
θ = 12.9–18.3°
μ = 0.17 mm^{-1}
T = 298 (2) K
Block, colorless
0.42 × 0.21 × 0.10 mm

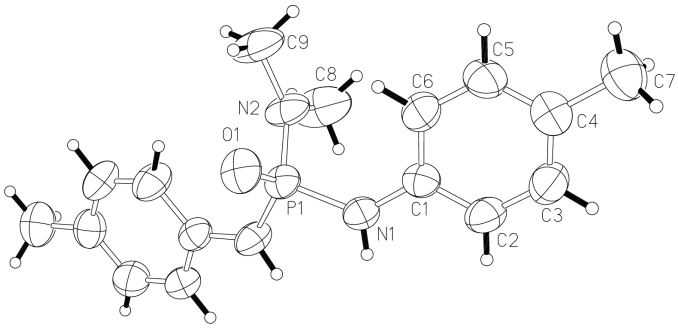


Figure 1
*ORTEP*II (Johnson, 1976) plot of the molecule, with ellipsoids at the 50% probability level.

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.881, T_{max} = 0.968
1939 measured reflections
1939 independent reflections
1213 reflections with $I > 2\sigma(I)$
θ_{max} = 27.5°
$h = -12 \rightarrow 0$
$k = 0 \rightarrow 13$
$l = -22 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.156
S = 1.03
1939 reflections
105 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0811P)^2 + 0.2692P]$ 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}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.021 (3)

Table 1
Selected geometric parameters (Å, °).

P1—O1	1.478 (2)	P1—N1	1.642 (2)
P1—N2	1.626 (3)		
O1—P1—N1	116.1 (1)	N1—P1—N1^{i}	98.4 (2)
O1—P1—N2	108.3 (1)	N1—P1—N2	108.7 (1)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.13	2.958 (3)	163

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

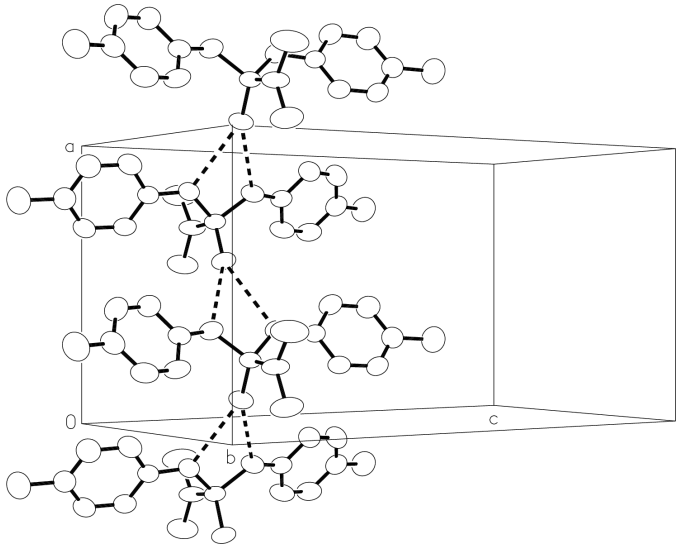


Figure 2
*ORTEP*II (Johnson, 1976) plot of the hydrogen-bonded chain structure.

The space group is an unconventional setting of *Pnma*.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CELDIM* (Enraf–Nonius, 1994); data reduction: *XCAD*4 (Harms, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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