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Diethyl phenyl(4-pyridylcarbonylamino)-methylphosphonate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.072
wR factor = 0.207
Data-to-parameter ratio = 14.3

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

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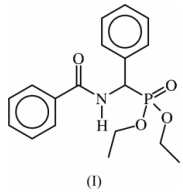
Two molecules of the title compound, $C_{17}H_{21}N_2O_4P$, are linked across a center of inversion by N—H···O=P amido–phosphoryl interactions [d(N···O) = 2.894 (3) Å], forming a hydrogen-bonded dimer.

Comment

The title compound, (I), was synthesized for a study of its cytotoxicity against the KB cancer cell line. The amido-phosphonic acid derivative was found to be moderately cytotoxic with an IC_{50} value of 95 μg ml^{-1}. The compound exists as a centrosymmetric hydrogen-bonded dimer (Fig. 1); the amido N atom interacts with the doubly bonded phosphoryl O atom (Table 2). The P1=O5 double bond is significantly shorter than the P1—O3 and P1—O4 single bonds (Table 1).



(I)

There are only a few related crystal structures having the $(CH_3CH_2O)_2P(O)$— fragment that can be used for comparison; one is 3-hydroxy-3-diethoxyphosphoryl-2-oxoindolinone, which is the alcoholysis product of 1,3,2-dioxaphospholanes having the 2-oxoindolinone unit (Gureivich *et al.*, 1998). The amide unit is also involved in hydrogen-bonding interactions with the phosphoryl O atom of a neighboring molecule [d(N···O) = 2.824 (3) Å], but for this compound the hydroxy group is also involved in hydrogen bonding.

Experimental

The hydrochloride of the α-aminophosphonate, $C_6H_5CH(NH_2)P(O)(OCH_2CH_3)_2$, was prepared according to the literature procedure of Takahashi *et al.* (1994). The reagent (2.79 g, 10 mmol) was dissolved in 1,2-dichloroethane (90 ml) to which triethylamine (4 ml) was added, and the solution was added dropwise to isonicotinic acid (1.23 g, 10 mmol) in the same solvent. After completion of the reaction, the solvent was removed to give the crude product, which was purified by recrystallization from a 1:1 mixture of hexane and dichloroethane. CHN analysis, calculated for $C_{17}H_{21}N_2O_4P$: C 58.62, H 6.08, N 8.04%; found: C 58.44, H 6.10 N 7.95%.

Crystal data

$C_{17}H_{21}N_2O_4P$
M_r = 348.33
Monoclinic, $C2/c$
a = 23.714 (1) Å
b = 8.0928 (4) Å
c = 20.012 (1) Å
β = 110.325 (1)°
V = 3601.4 (3) Å^3
Z = 8
D_x = 1.285 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4335 reflections
θ = 2.3–27.5°
μ = 0.18 mm^{-1}
T = 293 (2) K
Block, colorless
0.35 × 0.27 × 0.26 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: none
12057 measured reflections
3162 independent reflections
2774 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -28 \rightarrow 28$
$k = -9 \rightarrow 9$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.207
S = 1.05
3162 reflections
221 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1172P)^2 + 6.3036P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—O3	1.559 (3)	N2—C6	1.330 (4)
P1—O4	1.565 (3)	N2—C7	1.447 (4)
P1—O5	1.448 (2)	O2—C6	1.210 (4)
P1—C7	1.819 (3)	O3—C14	1.425 (5)
N1—C3	1.314 (6)	O4—C16	1.407 (5)
N1—C4	1.314 (7)		
O3—P1—O4	107.1 (2)	O5—P1—C7	113.2 (1)
O3—P1—O5	116.0 (2)	C3—N1—C4	115.9 (4)
O3—P1—C7	103.2 (2)	C6—N2—C7	121.6 (3)
O4—P1—O5	108.6 (2)	C14—O3—P1	126.8 (3)
O4—P1—C7	108.3 (1)	C16—O4—P1	125.9 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···O5^{i}	0.84 (1)	2.06 (1)	2.894 (3)	173 (3)

Symmetry code: (i) $1-x, 1-y, 1-z$.

The H atoms were positioned geometrically [C—H = 0.93 (aromatic), 0.93 (methine), 0.96 (methylene) and 0.97 Å (methyl)] and were included in the refinement in the riding-model approximation. The displacement parameters were set to $1.5U_{eq}$(C) for the methyl H atoms and to $1.2U_{eq}$ for the other H atoms. The amido H atom was located and refined with an N—H distance restraint of 0.85 (1) Å.

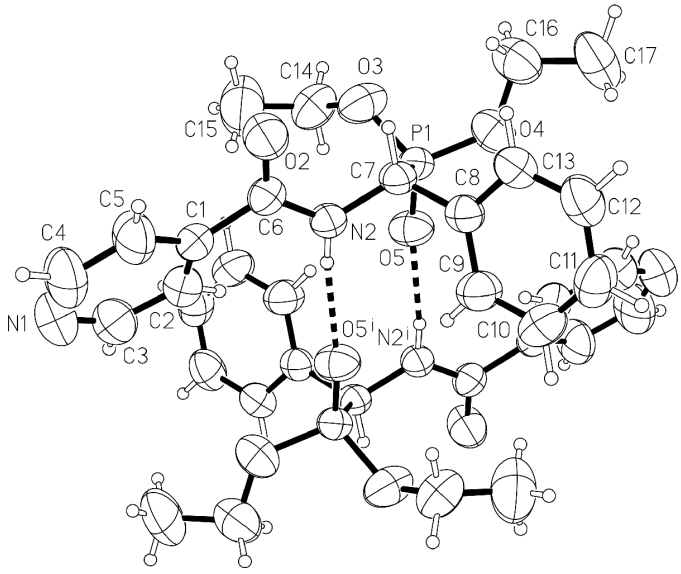


Figure 1
View of the hydrogen-bonded dimer in (I) (30% displacement ellipsoids). H atoms are drawn as spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines. [Symmetry code: (i) $1-x, 1-y, 1-z$.]

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); 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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References

Bruker (2001). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Gureivich, P. A., Akhmetova, G. Z., Gubaidullin, A. T., Moskva, V. V. & Litvinov, I. A. (1998). *Russ. J. Gen. Chem.* **68**, 1501–1506.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Takahashi, H., Yoshioka, M., Imai, N., Onimura, K. & Kobayashi, S. (1994). *Synthesis*, pp. 763–764.