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Diethylammonium ethyl (4-methylanilino)phosphonate chloroform solvate

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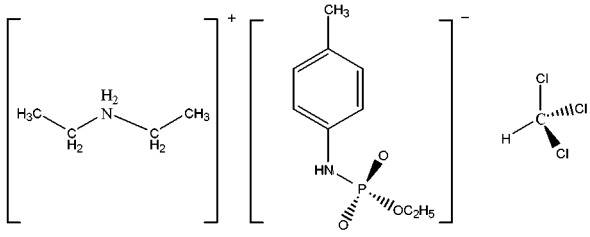
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; R factor = 0.052; wR factor = 0.155; data-to-parameter ratio = 18.0.

In the title compound, $C_4H_{12}N^+\cdot C_9H_{13}NO_3P^-\cdot CHCl_3$, there are two independent formula units in the asymmetric unit. In the crystal structure, intermolecular N—H···O hydrogen bonds form two independent one-dimensional chains along the *a* axis.

Related literature

For the corresponding dimeric complex with similar P—O and P—N bonds, see: Andrianov *et al.* (1977). For related literature, see: Quin & Jankowski (1994).



Experimental

Crystal data

$C_4H_{12}N^+\cdot C_9H_{13}NO_3P^-\cdot CHCl_3$
M_r = 407.69
Triclinic, $P\overline{1}$
a = 8.4030 (2) Å
b = 12.5575 (4) Å
c = 19.6980 (7) Å
α = 82.8707 (15)°
β = 83.869 (2)°
γ = 84.9637 (18)°
V = 2044.82 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.54 mm^{-1}
T = 173 (2) K
0.4 × 0.4 × 0.2 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.811, T_{max} = 0.895
14646 measured reflections
8048 independent reflections
5266 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.155
S = 1.04
8048 reflections
447 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.87 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O2	0.79 (3)	2.00 (3)	2.782 (3)	179 (4)
N4—H5···O5	0.88 (3)	1.91 (3)	2.790 (3)	177 (3)

Data collection: *SMART* (Bruker, 1996); cell refinement: *SAINT* (Bruker, 1994); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2482).

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supplementary materials

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Diethylammonium ethyl (4-methylanilino)phosphonate chloroform solvate

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Comment

Although O,*N*-substituted phosphoramidic acids have been reported as a useful agents in the synthesis of pyrophosphate groups, (Quin & Jankowski, 1994), crystal structures of these types of materials are not well characterized. As part of an investigation of the use of the O,*N*-substituted phosphoramidic acids as ligand in the synthesis of new metal phosphate frameworks, the title compound was obtained as a salt of the O,*N*-substituted phosphoramidic acid.

As shown in Figure 1, the asymmetric unit of the title compound is composed of two chloroform molecules, two diethylamine cations and two *N*-ethoxyphosphorl-p -tolylamide anions. The geometrical parameters of the two independent anions are similar. The phosphorus atoms have tetrahedral coordination geometry and there are three types of P—O bonds and one P—N bond existing in the [PO_3N] tetrahetra. The shortest bond lengths of 1.4945 (17)Å (P1—O3) and 1.4891 (17)Å (P2—O4) refer to the P=O double bonds at the [P1O_3N] and [P2O_3N] tetrahetra respectively, while the P—O bond lengths of 1.6002 (19) Å and 1.6055 (18)Å are attributed to the P-OEt connections. These longer P—O distances are due to the influence of the –OEt group, according to the literature report (Andrianov *et al.*, 1977). The P—N bond lengths in the [PO_3N] tetrahetra are 1.656 (2)Å (P1—N1)and 1.659 (2)Å (P2—N) respectively. The bond angles of O—P—O and O—P—N range from 103.44 (10)- 118.32 (10)° and 103.03 (10)- 119.28 (10)°, indicating that the tetrahetra are slightly distorted. Solvated chloroform molecules fill the voids in the crystal lattice.

In the crystal structure, intermolecular N—H···O hydrogen bonds form two independent one-dimensional chains along the *a* axis.

Experimental

A solution of toluidine 12.5 mmol and 12.5 mmol of Et_2NH in 15 ml of ether was added to a solution of 12.5 mmol of ethyl phosphorodichloridate in 15 ml of ether. After 20 h, the solution was filtrated and the filtrate was evaporated to give a solid powder. The powder was dissolved in 30 ml of acetone-water containing 1 g of NaOH. After 10 min, the solvent was evaporated and the residue dried over *in vacuo*. Recrystalization of a chloroform solution of the title compound gave crystals suitable for X-ray diffraction.

Refinement

H atoms bonded to N atoms and the C atoms of the chloroform molecules were located in a difference map and refined with distance restraints of N—H = 0.85 (3), and C—H = 0.95 (3) Å, and isotropic displacement parameters. Other H atoms were positioned geometrically and refined using a riding- model approximation, with C—H = 0.93–0.97 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups)U_{eq}(C).

Figures

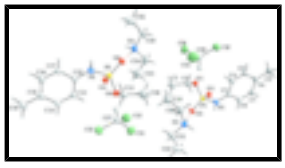

Fig. 1. The asymmetric unit with atom labels and 50% probability displacement ellipsoids for non-H atoms. Hydrogen bonds are shown as dashed lines.

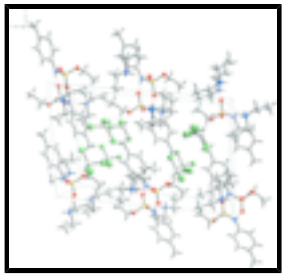

Fig. 2. The packing of the title compound viewed down the *a* axis, showing N—H···O hydrogen bonds as dashed lines.

Diethylammonium ethyl (4-methylanilino)phosphonate chloroform solvate

Crystal data

$C_4H_{12}N^+·C_9H_{13}NO_3P^−·CHCl_3$	$Z = 4$
$M_r = 407.69$	$F_{000} = 856$
Triclinic, $P\overline{1}$	$D_x = 1.326$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.4030$ (2) Å	Cell parameters from 14646 reflections
$b = 12.5575$ (4) Å	$\theta = 2.6$–$26.4°$
$c = 19.6980$ (7) Å	$\mu = 0.54$ mm^{-1}
$\alpha = 82.8707$ (15)°	$T = 173$ (2) K
$\beta = 83.869$ (2)°	Block, colourless
$\gamma = 84.9637$ (18)°	0.4 × 0.4 × 0.2 mm
$V = 2044.82$ (11) Å^3	

Data collection

Bruker SMART CCD diffractometer	8048 independent reflections
Radiation source: fine-focus sealed tube	5266 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.026$
$T = 173$(2) K	$\theta_{max} = 26.4°$
ω scans	$\theta_{min} = 2.6°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{min} = 0.811$, $T_{max} = 0.895$	$k = -15 \rightarrow 15$
14646 measured reflections	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.155$	$w = 1/[\sigma^2(F_o^2) + (0.0688P)^2 + 1.5923P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.002$
8048 reflections	$\Delta\rho_{max} = 0.87$ e Å^{-3}
447 parameters	$\Delta\rho_{min} = -0.37$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	1.29263 (7)	0.89735 (5)	1.05042 (3)	0.02205 (16)
P2	0.71031 (7)	1.10383 (5)	1.44730 (3)	0.02143 (16)
C1	1.5019 (4)	0.9023 (3)	1.19335 (18)	0.0534 (9)
H1B	1.6076	0.9100	1.2056	0.080*
H1C	1.4394	0.8677	1.2322	0.080*
H1F	1.4511	0.9721	1.1798	0.080*
C2	1.5139 (3)	0.8359 (2)	1.13512 (16)	0.0392 (7)
H2A	1.5784	0.8704	1.0961	0.047*
H2B	1.5667	0.7657	1.1485	0.047*
C3	1.4176 (3)	0.76561 (18)	0.95398 (13)	0.0244 (5)
C4	1.3400 (3)	0.6769 (2)	0.98657 (14)	0.0339 (6)
H4A	1.2809	0.6812	1.0290	0.041*
C5	1.3499 (3)	0.5826 (2)	0.95654 (15)	0.0372 (7)
H5A	1.2964	0.5248	0.9794	0.045*
C6	1.4366 (3)	0.5708 (2)	0.89340 (15)	0.0326 (6)
C7	1.5153 (3)	0.6589 (2)	0.86158 (14)	0.0348 (6)
H7A	1.5755	0.6538	0.8195	0.042*

C8	1.5066 (3)	0.7547 (2)	0.89096 (14)	0.0315 (6)
H8A	1.5609	0.8123	0.8682	0.038*
C9	1.4443 (4)	0.4666 (2)	0.86181 (17)	0.0469 (8)
H9A	1.5092	0.4733	0.8185	0.070*
H9B	1.3379	0.4511	0.8547	0.070*
H9C	1.4908	0.4093	0.8922	0.070*
C10	0.5322 (4)	1.1069 (3)	1.29435 (16)	0.0507 (8)
H10A	0.4308	1.0988	1.2779	0.076*
H10B	0.5844	1.0371	1.3068	0.076*
H10C	0.5986	1.1460	1.2588	0.076*
C11	0.5053 (3)	1.1669 (2)	1.35537 (15)	0.0384 (7)
H11A	0.4510	1.2370	1.3429	0.046*
H11B	0.4368	1.1280	1.3910	0.046*
C12	0.5763 (3)	1.23085 (18)	1.54516 (12)	0.0221 (5)
C13	0.6510 (3)	1.32177 (19)	1.51385 (14)	0.0314 (6)
H13A	0.7082	1.3204	1.4708	0.038*
C14	0.6406 (3)	1.4135 (2)	1.54627 (15)	0.0351 (6)
H14A	0.6919	1.4729	1.5245	0.042*
C15	0.5556 (3)	1.4207 (2)	1.61081 (14)	0.0313 (6)
C16	0.4814 (3)	1.3295 (2)	1.64106 (14)	0.0328 (6)
H16A	0.4234	1.3311	1.6839	0.039*
C17	0.4911 (3)	1.2363 (2)	1.60930 (13)	0.0287 (6)
H17A	0.4400	1.1768	1.6311	0.034*
C18	0.5453 (4)	1.5223 (2)	1.64501 (17)	0.0442 (7)
H18A	0.4825	1.5120	1.6887	0.066*
H18B	0.4955	1.5805	1.6164	0.066*
H18C	0.6514	1.5391	1.6518	0.066*
C19	0.9895 (4)	1.0396 (3)	1.18932 (18)	0.0625 (10)
H19A	0.9186	0.9997	1.2226	0.094*
H19B	1.0103	1.1049	1.2064	0.094*
H19C	1.0887	0.9969	1.1812	0.094*
C20	0.9133 (4)	1.0663 (3)	1.12371 (15)	0.0469 (8)
H20A	0.8118	1.1078	1.1325	0.056*
H20B	0.8907	1.0000	1.1072	0.056*
C21	1.0452 (4)	1.2393 (2)	1.07998 (19)	0.0513 (8)
H21A	1.1069	1.2367	1.1192	0.062*
H21B	0.9428	1.2791	1.0902	0.062*
C22	1.1338 (5)	1.2952 (3)	1.0177 (2)	0.0731 (12)
H22A	1.1507	1.3670	1.0259	0.110*
H22B	1.0720	1.2983	0.9791	0.110*
H22C	1.2356	1.2563	1.0081	0.110*
C23	1.1327 (4)	0.9797 (3)	1.36070 (18)	0.0609 (10)
H23A	1.1959	0.9904	1.3172	0.091*
H23B	1.1984	0.9845	1.3971	0.091*
H23C	1.0450	1.0342	1.3623	0.091*
C24	1.0701 (4)	0.8741 (3)	1.36913 (16)	0.0531 (9)
H24A	1.1589	0.8194	1.3668	0.064*
H24B	1.0060	0.8690	1.3317	0.064*
C25	0.9187 (4)	0.7405 (2)	1.4462 (2)	0.0550 (10)

H25A	0.8568	0.7291	1.4093	0.066*
H25B	1.0127	0.6897	1.4454	0.066*
C26	0.8194 (4)	0.7206 (3)	1.5135 (2)	0.0675 (11)
H26A	0.7872	0.6482	1.5194	0.101*
H26B	0.7258	0.7703	1.5141	0.101*
H26C	0.8814	0.7304	1.5501	0.101*
C27	0.9296 (3)	1.3268 (2)	1.31187 (16)	0.0370 (7)
C28	1.0752 (3)	0.6802 (2)	1.18552 (16)	0.0361 (6)
O1	1.3562 (2)	0.82291 (14)	1.11549 (9)	0.0304 (4)
O2	1.31494 (19)	1.01307 (13)	1.05514 (9)	0.0274 (4)
O3	1.12594 (18)	0.86509 (13)	1.04931 (9)	0.0263 (4)
O4	0.87397 (18)	1.13685 (13)	1.45287 (9)	0.0274 (4)
O5	0.68917 (19)	0.98893 (13)	1.43997 (9)	0.0273 (4)
O6	0.6545 (2)	1.18052 (13)	1.38125 (9)	0.0292 (4)
Cl1	0.81294 (9)	1.34074 (7)	1.24222 (4)	0.0466 (2)
Cl2	0.91751 (11)	1.44700 (7)	1.34968 (5)	0.0574 (2)
Cl3	1.13127 (9)	1.28867 (8)	1.28418 (5)	0.0570 (2)
Cl4	0.87684 (9)	0.72423 (8)	1.21407 (6)	0.0698 (3)
Cl5	1.07822 (12)	0.55915 (7)	1.14978 (5)	0.0607 (3)
Cl6	1.19441 (9)	0.66534 (7)	1.25426 (4)	0.0490 (2)
N1	1.4088 (3)	0.86350 (17)	0.98185 (11)	0.0276 (5)
H1	1.480 (4)	0.902 (3)	0.9688 (17)	0.047 (10)*
N2	0.5856 (2)	1.13562 (17)	1.51415 (11)	0.0260 (5)
H2	0.521 (4)	1.094 (3)	1.5298 (17)	0.044 (9)*
N3	1.0184 (3)	1.13005 (19)	1.06806 (12)	0.0304 (5)
H3	1.102 (4)	1.097 (3)	1.0637 (16)	0.039 (9)*
H4	0.970 (3)	1.132 (2)	1.0265 (17)	0.039 (8)*
N4	0.9706 (3)	0.85319 (19)	1.43514 (12)	0.0303 (5)
H5	0.882 (4)	0.896 (3)	1.4384 (16)	0.046 (9)*
H6	1.029 (3)	0.859 (2)	1.4702 (16)	0.035 (8)*
H7	0.889 (3)	1.272 (2)	1.3452 (15)	0.037 (8)*
H8	1.113 (3)	0.737 (2)	1.1534 (16)	0.042 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0208 (3)	0.0206 (3)	0.0255 (3)	−0.0042 (2)	−0.0029 (2)	−0.0034 (2)
P2	0.0196 (3)	0.0207 (3)	0.0247 (3)	−0.0046 (2)	−0.0021 (2)	−0.0035 (2)
C1	0.063 (2)	0.0459 (18)	0.059 (2)	0.0036 (16)	−0.0318 (18)	−0.0195 (16)
C2	0.0372 (15)	0.0373 (15)	0.0478 (18)	0.0049 (12)	−0.0223 (13)	−0.0129 (13)
C3	0.0212 (12)	0.0212 (12)	0.0309 (14)	−0.0012 (9)	−0.0039 (10)	−0.0026 (10)
C4	0.0374 (15)	0.0271 (13)	0.0353 (15)	−0.0060 (11)	0.0082 (12)	−0.0031 (11)
C5	0.0448 (16)	0.0203 (13)	0.0457 (18)	−0.0070 (12)	0.0035 (13)	−0.0043 (12)
C6	0.0327 (14)	0.0273 (13)	0.0392 (16)	0.0043 (11)	−0.0076 (12)	−0.0104 (12)
C7	0.0355 (14)	0.0371 (15)	0.0320 (15)	−0.0024 (12)	0.0030 (12)	−0.0099 (12)
C8	0.0320 (14)	0.0311 (14)	0.0317 (15)	−0.0078 (11)	0.0015 (11)	−0.0044 (11)
C9	0.0557 (19)	0.0325 (15)	0.055 (2)	0.0032 (14)	−0.0081 (15)	−0.0179 (14)
C10	0.060 (2)	0.0543 (19)	0.0435 (19)	−0.0111 (16)	−0.0163 (16)	−0.0156 (16)

C11	0.0352 (15)	0.0404 (16)	0.0428 (17)	0.0015 (12)	−0.0167 (13)	−0.0099 (13)
C12	0.0181 (11)	0.0225 (12)	0.0266 (13)	−0.0036 (9)	−0.0050 (9)	−0.0028 (10)
C13	0.0369 (14)	0.0239 (13)	0.0320 (15)	−0.0054 (11)	0.0047 (11)	−0.0026 (11)
C14	0.0418 (15)	0.0229 (13)	0.0407 (17)	−0.0077 (11)	0.0017 (13)	−0.0050 (12)
C15	0.0323 (14)	0.0262 (13)	0.0369 (15)	0.0006 (11)	−0.0062 (11)	−0.0091 (11)
C16	0.0331 (14)	0.0363 (14)	0.0288 (14)	−0.0022 (11)	0.0021 (11)	−0.0082 (12)
C17	0.0281 (13)	0.0286 (13)	0.0298 (14)	−0.0095 (10)	0.0011 (11)	−0.0030 (11)
C18	0.0507 (18)	0.0343 (15)	0.0504 (19)	0.0006 (13)	−0.0066 (15)	−0.0177 (14)
C19	0.070 (2)	0.067 (2)	0.046 (2)	−0.0003 (19)	0.0016 (18)	0.0014 (18)
C20	0.0357 (16)	0.067 (2)	0.0358 (17)	0.0067 (15)	−0.0047 (13)	−0.0022 (15)
C21	0.056 (2)	0.0374 (16)	0.066 (2)	0.0032 (15)	−0.0239 (17)	−0.0199 (16)
C22	0.085 (3)	0.049 (2)	0.091 (3)	−0.029 (2)	−0.042 (2)	0.015 (2)
C23	0.053 (2)	0.077 (3)	0.049 (2)	0.0022 (19)	0.0029 (16)	−0.0027 (19)
C24	0.0422 (17)	0.079 (2)	0.0377 (18)	0.0091 (17)	−0.0037 (14)	−0.0139 (17)
C25	0.0407 (17)	0.0396 (17)	0.094 (3)	0.0057 (14)	−0.0284 (18)	−0.0323 (18)
C26	0.060 (2)	0.047 (2)	0.097 (3)	−0.0234 (17)	−0.026 (2)	0.013 (2)
C27	0.0326 (14)	0.0365 (15)	0.0407 (17)	−0.0079 (12)	−0.0007 (12)	0.0019 (13)
C28	0.0315 (14)	0.0366 (15)	0.0395 (17)	−0.0062 (12)	−0.0034 (12)	0.0013 (13)
O1	0.0312 (9)	0.0303 (9)	0.0318 (10)	−0.0035 (7)	−0.0121 (8)	−0.0029 (8)
O2	0.0230 (8)	0.0217 (8)	0.0385 (10)	−0.0056 (7)	−0.0011 (7)	−0.0064 (7)
O3	0.0219 (8)	0.0301 (9)	0.0286 (10)	−0.0070 (7)	−0.0027 (7)	−0.0055 (7)
O4	0.0202 (8)	0.0328 (9)	0.0309 (10)	−0.0080 (7)	−0.0031 (7)	−0.0064 (8)
O5	0.0243 (8)	0.0210 (8)	0.0371 (10)	−0.0050 (7)	0.0014 (7)	−0.0070 (7)
O6	0.0313 (9)	0.0296 (9)	0.0280 (10)	−0.0069 (7)	−0.0084 (7)	−0.0008 (7)
Cl1	0.0401 (4)	0.0578 (5)	0.0423 (4)	−0.0038 (3)	−0.0082 (3)	−0.0038 (4)
Cl2	0.0731 (6)	0.0455 (4)	0.0558 (5)	−0.0118 (4)	−0.0042 (4)	−0.0107 (4)
Cl3	0.0326 (4)	0.0710 (6)	0.0650 (6)	−0.0005 (4)	−0.0016 (4)	−0.0038 (4)
Cl4	0.0325 (4)	0.0831 (7)	0.0890 (7)	0.0047 (4)	−0.0021 (4)	−0.0014 (6)
Cl5	0.0874 (7)	0.0465 (5)	0.0511 (5)	−0.0216 (4)	−0.0063 (5)	−0.0061 (4)
Cl6	0.0441 (4)	0.0613 (5)	0.0424 (5)	0.0017 (4)	−0.0093 (3)	−0.0087 (4)
N1	0.0252 (11)	0.0230 (11)	0.0350 (13)	−0.0080 (9)	0.0036 (9)	−0.0065 (9)
N2	0.0247 (11)	0.0224 (10)	0.0312 (12)	−0.0088 (9)	0.0036 (9)	−0.0048 (9)
N3	0.0269 (12)	0.0343 (12)	0.0304 (13)	0.0036 (10)	−0.0053 (10)	−0.0073 (10)
N4	0.0224 (11)	0.0387 (13)	0.0320 (13)	0.0027 (10)	−0.0075 (10)	−0.0125 (10)

Geometric parameters (Å, °)

P1—O3	1.4945 (17)	C16—H16A	0.9300
P1—O2	1.4959 (17)	C17—H17A	0.9300
P1—O1	1.6002 (19)	C18—H18A	0.9600
P1—N1	1.656 (2)	C18—H18B	0.9600
P2—O4	1.4891 (17)	C18—H18C	0.9600
P2—O5	1.4946 (17)	C19—C20	1.491 (5)
P2—O6	1.6055 (18)	C19—H19A	0.9600
P2—N2	1.659 (2)	C19—H19B	0.9600
C1—C2	1.489 (4)	C19—H19C	0.9600
C1—H1B	0.9600	C20—N3	1.523 (4)
C1—H1C	0.9600	C20—H20A	0.9700
C1—H1F	0.9600	C20—H20B	0.9700

C2—O1	1.447 (3)	C21—N3	1.460 (4)
C2—H2A	0.9700	C21—C22	1.495 (5)
C2—H2B	0.9700	C21—H21A	0.9700
C3—C4	1.392 (3)	C21—H21B	0.9700
C3—C8	1.394 (3)	C22—H22A	0.9600
C3—N1	1.399 (3)	C22—H22B	0.9600
C4—C5	1.379 (4)	C22—H22C	0.9600
C4—H4A	0.9300	C23—C24	1.453 (5)
C5—C6	1.390 (4)	C23—H23A	0.9600
C5—H5A	0.9300	C23—H23B	0.9600
C6—C7	1.385 (4)	C23—H23C	0.9600
C6—C9	1.510 (4)	C24—N4	1.476 (4)
C7—C8	1.390 (4)	C24—H24A	0.9700
C7—H7A	0.9300	C24—H24B	0.9700
C8—H8A	0.9300	C25—C26	1.493 (5)
C9—H9A	0.9600	C25—N4	1.500 (4)
C9—H9B	0.9600	C25—H25A	0.9700
C9—H9C	0.9600	C25—H25B	0.9700
C10—C11	1.483 (4)	C26—H26A	0.9600
C10—H10A	0.9600	C26—H26B	0.9600
C10—H10B	0.9600	C26—H26C	0.9600
C10—H10C	0.9600	C27—Cl1	1.752 (3)
C11—O6	1.433 (3)	C27—Cl2	1.754 (3)
C11—H11A	0.9700	C27—Cl3	1.767 (3)
C11—H11B	0.9700	C27—H7	0.95 (3)
C12—C17	1.390 (3)	C28—Cl5	1.749 (3)
C12—C13	1.394 (3)	C28—Cl6	1.750 (3)
C12—N2	1.401 (3)	C28—Cl4	1.764 (3)
C13—C14	1.376 (4)	C28—H8	0.94 (3)
C13—H13A	0.9300	N1—H1	0.80 (3)
C14—C15	1.398 (4)	N2—H2	0.80 (3)
C14—H14A	0.9300	N3—H3	0.79 (3)
C15—C16	1.389 (4)	N3—H4	0.95 (3)
C15—C18	1.506 (4)	N4—H5	0.88 (3)
C16—C17	1.385 (4)	N4—H6	0.90 (3)
O3—P1—O2	118.32 (10)	H18A—C18—H18C	109.5
O3—P1—O1	103.44 (10)	H18B—C18—H18C	109.5
O2—P1—O1	110.39 (10)	C20—C19—H19A	109.5
O3—P1—N1	110.78 (11)	C20—C19—H19B	109.5
O2—P1—N1	106.60 (11)	H19A—C19—H19B	109.5
O1—P1—N1	106.81 (11)	C20—C19—H19C	109.5
O4—P2—O5	119.28 (10)	H19A—C19—H19C	109.5
O4—P2—O6	103.03 (10)	H19B—C19—H19C	109.5
O5—P2—O6	110.12 (10)	C19—C20—N3	112.8 (3)
O4—P2—N2	110.52 (11)	C19—C20—H20A	109.0
O5—P2—N2	106.51 (11)	N3—C20—H20A	109.0
O6—P2—N2	106.80 (10)	C19—C20—H20B	109.0
C2—C1—H1B	109.5	N3—C20—H20B	109.0
C2—C1—H1C	109.5	H20A—C20—H20B	107.8

H1B—C1—H1C	109.5	N3—C21—C22	110.0 (3)
C2—C1—H1F	109.5	N3—C21—H21A	109.7
H1B—C1—H1F	109.5	C22—C21—H21A	109.7
H1C—C1—H1F	109.5	N3—C21—H21B	109.7
O1—C2—C1	110.7 (2)	C22—C21—H21B	109.7
O1—C2—H2A	109.5	H21A—C21—H21B	108.2
C1—C2—H2A	109.5	C21—C22—H22A	109.5
O1—C2—H2B	109.5	C21—C22—H22B	109.5
C1—C2—H2B	109.5	H22A—C22—H22B	109.5
H2A—C2—H2B	108.1	C21—C22—H22C	109.5
C4—C3—C8	117.5 (2)	H22A—C22—H22C	109.5
C4—C3—N1	122.7 (2)	H22B—C22—H22C	109.5
C8—C3—N1	119.8 (2)	C24—C23—H23A	109.5
C5—C4—C3	120.6 (2)	C24—C23—H23B	109.5
C5—C4—H4A	119.7	H23A—C23—H23B	109.5
C3—C4—H4A	119.7	C24—C23—H23C	109.5
C4—C5—C6	122.5 (2)	H23A—C23—H23C	109.5
C4—C5—H5A	118.7	H23B—C23—H23C	109.5
C6—C5—H5A	118.7	C23—C24—N4	112.1 (3)
C7—C6—C5	116.6 (2)	C23—C24—H24A	109.2
C7—C6—C9	122.2 (3)	N4—C24—H24A	109.2
C5—C6—C9	121.2 (3)	C23—C24—H24B	109.2
C6—C7—C8	121.7 (2)	N4—C24—H24B	109.2
C6—C7—H7A	119.2	H24A—C24—H24B	107.9
C8—C7—H7A	119.2	C26—C25—N4	110.9 (3)
C7—C8—C3	121.0 (2)	C26—C25—H25A	109.5
C7—C8—H8A	119.5	N4—C25—H25A	109.5
C3—C8—H8A	119.5	C26—C25—H25B	109.5
C6—C9—H9A	109.5	N4—C25—H25B	109.5
C6—C9—H9B	109.5	H25A—C25—H25B	108.0
H9A—C9—H9B	109.5	C25—C26—H26A	109.5
C6—C9—H9C	109.5	C25—C26—H26B	109.5
H9A—C9—H9C	109.5	H26A—C26—H26B	109.5
H9B—C9—H9C	109.5	C25—C26—H26C	109.5
C11—C10—H10A	109.5	H26A—C26—H26C	109.5
C11—C10—H10B	109.5	H26B—C26—H26C	109.5
H10A—C10—H10B	109.5	Cl1—C27—Cl2	110.84 (16)
C11—C10—H10C	109.5	Cl1—C27—Cl3	109.84 (17)
H10A—C10—H10C	109.5	Cl2—C27—Cl3	110.34 (16)
H10B—C10—H10C	109.5	Cl1—C27—H7	108.2 (18)
O6—C11—C10	110.9 (2)	Cl2—C27—H7	108.5 (18)
O6—C11—H11A	109.5	Cl3—C27—H7	109.1 (17)
C10—C11—H11A	109.5	Cl5—C28—Cl6	110.80 (16)
O6—C11—H11B	109.5	Cl5—C28—Cl4	110.50 (16)
C10—C11—H11B	109.5	Cl6—C28—Cl4	109.60 (17)
H11A—C11—H11B	108.0	Cl5—C28—H8	112.5 (19)
C17—C12—C13	118.1 (2)	Cl6—C28—H8	107.8 (19)
C17—C12—N2	119.9 (2)	Cl4—C28—H8	105.4 (18)
C13—C12—N2	122.1 (2)	C2—O1—P1	119.30 (17)

C14—C13—C12	120.4 (2)	C11—O6—P2	120.05 (17)
C14—C13—H13A	119.8	C3—N1—P1	126.97 (18)
C12—C13—H13A	119.8	C3—N1—H1	117 (2)
C13—C14—C15	122.5 (2)	P1—N1—H1	115 (2)
C13—C14—H14A	118.8	C12—N2—P2	126.94 (17)
C15—C14—H14A	118.8	C12—N2—H2	115 (2)
C16—C15—C14	116.3 (2)	P2—N2—H2	118 (2)
C16—C15—C18	122.3 (3)	C21—N3—C20	116.5 (3)
C14—C15—C18	121.4 (2)	C21—N3—H3	109 (2)
C17—C16—C15	122.0 (2)	C20—N3—H3	107 (2)
C17—C16—H16A	119.0	C21—N3—H4	109.6 (18)
C15—C16—H16A	119.0	C20—N3—H4	106.9 (18)
C16—C17—C12	120.8 (2)	H3—N3—H4	107 (3)
C16—C17—H17A	119.6	C24—N4—C25	111.7 (3)
C12—C17—H17A	119.6	C24—N4—H5	114 (2)
C15—C18—H18A	109.5	C25—N4—H5	106 (2)
C15—C18—H18B	109.5	C24—N4—H6	109.6 (19)
H18A—C18—H18B	109.5	C25—N4—H6	106.2 (18)
C15—C18—H18C	109.5	H5—N4—H6	109 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O2	0.79 (3)	2.00 (3)	2.782 (3)	179 (4)
N4—H5···O5	0.88 (3)	1.91 (3)	2.790 (3)	177 (3)

Fig. 1

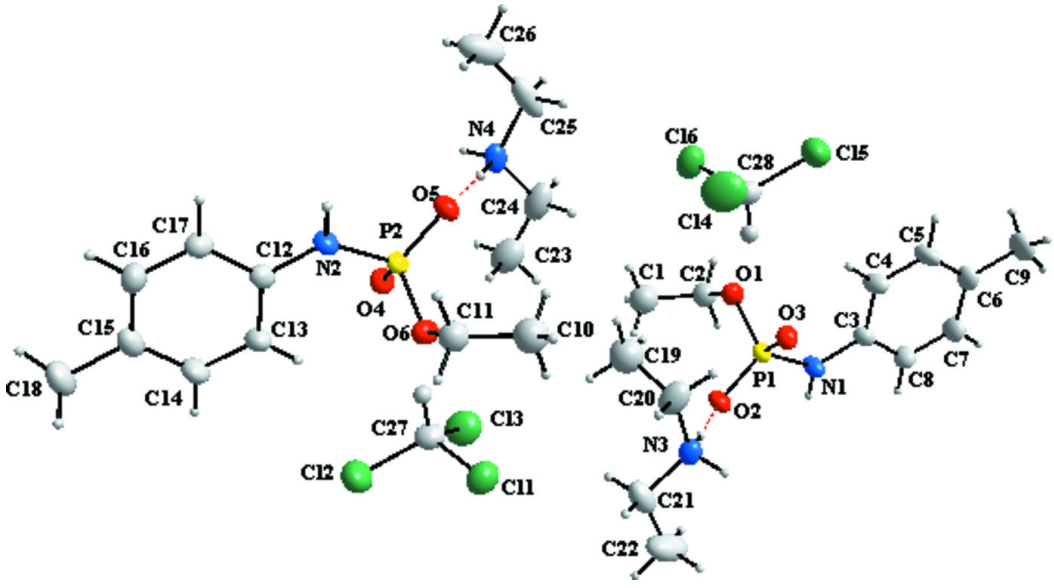
C26
N4
C25
C16
C28
Cl5
O5
C24
C14
C17
C16
C12
N2
P2
C23
C5
C4
C1
C2
O1
C9
O4
C11
O3
C6
C15
C13
C10
C19
C3
C18
C14
P1
N1
C7
C20
C8
O2
C27
Cl3
N3
Cl2
Cl1
C21
C22

Fig. 2