Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-Benzylpropan-2-aminium (benzylisopropylamido)(2,2,2-trifluoroacetamido)phosphate

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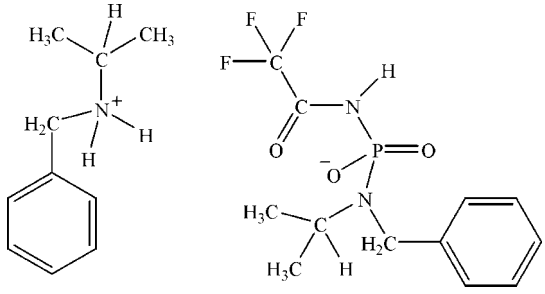
Received 29 September 2007; accepted 9 October 2007

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.103; data-to-parameter ratio = 21.4.

In the title compound, $C_{10}H_{16}N^+\cdot C_{12}H_{15}F_3N_2O_3P^-$, the two P—N distances are significantly different [1.740 (2) and 1.641 (2) Å]. In the crystal structure, cations and anions are connected *via* intermolecular N—H···O hydrogen bonds to form chains of centrosymmetric rings propagating in the *b*-axis direction.

Related literature

For related literature, see: Banaszczyk *et al.* (2002); Chivers *et al.* (2003); Cho *et al.* (1982); Corbridge (1995); Hamilton *et al.* (1995); Hammerschmidt & Hanbauer (2000); Hu *et al.* (2003); Pettit *et al.* (2005); Protsen *et al.* (1971); Shokol *et al.* (1969).



Experimental

Crystal data

$C_{10}H_{16}N^+\cdot C_{12}H_{15}F_3N_2O_3P^-$
M_r = 473.47
Triclinic, $P\overline{1}$
a = 9.6147 (18) Å
b = 10.027 (2) Å
c = 13.503 (3) Å
α = 68.355 (4)°
β = 75.939 (4)°
γ = 88.494 (4)°
V = 1170.9 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 150 (2) K
0.55 × 0.30 × 0.25 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: none
14154 measured reflections
6183 independent reflections
3060 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.103
S = 1.00
6183 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N1···O1^{i}	0.85	1.94	2.781 (3)	170
N3—H1N3···O2^{ii}	0.85	1.96	2.791 (3)	164
N3—H2N3···O2	0.85	2.10	2.932 (3)	166

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Support of this investigation by Ferdowsi University is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2523).

References

Banaszczyk, M. G., Carlo, A. T., Millan, V., Lindsey, A., Moss, R., Carlo, D. J. & Hendler, S. S. (2002). *Anesth. Analg.* **95**, 1285–1292.
Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chivers, T., Krahn, M., Schatte, G. & Parvez, M. (2003). *Inorg. Chem.* **42**, 3994–4005.
Cho, M. J., Kurtz, R. R., Lewis, C., Machkovech, S. M. & Houser, D. J. (1982). *J. Pharm. Sci.* **71**, 410–414.
Corbridge, D. E. C. (1995). *Phosphorus, an Outline of its Chemistry, Biochemistry and Technology*, 5th ed., p. 1179. New York: Elsevier Science.
Hamilton, R., Walker, B. & Walker, B. J. (1995). *Tetrahedron Lett.* **36**, 4451–4454.
Hammerschmidt, F. & Hanbauer, M. (2000). *J. Org. Chem.* **65**, 6121–6131.
Hu, L., Yu, C., Jiang, Y., Han, J., Li, Z., Browne, P., Race, P. R., Knox, R. J., Searle, P. F. & Hyde, E. I. (2003). *J. Med. Chem.* **46**, 4818–4821.
Pettit, G. R., Anderson, C. R., Gapud, E. J., Jung, M. K., Knight, J. C., Hamel, E. & Pettit, R. K. (2005). *J. Nat. Prod.* **68**, 1191–1197.
Protsen, L. D., Rodionov, P. V., Nikolaeva, S. V., Tarnavskaya, M. I. & Kopelnik, M. A. (1971). *Fiziol. Aktiv. Veshchestva*, **3**, 80–85.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Shokol, V. A., Kisilenko, A. A. & Derkach, G. I. (1969). *Zh. Obshch. Khim.* **39**, 1492–1497.

  doi:10.1107/S1600536807049586

supplementary materials

Acta Cryst. (2007). E**63**, o4318 [**doi:10.1107/S1600536807049586**]

N-Benzylpropan-2-aminium (benzylisopropylamido)(2,2,2-trifluoroacetamido)phosphate

M. Yazdanbakhsh and F. Sabbaghi

Comment

Phosphoro-azo (isoelectronic analogues of orthophosphate $[PO_4]^{3-}$, in which the oxygen atoms are replaced by imido (NRR') groups) derivatives of Beta-diketones are potentially versatile multidentate ligands (Chivers *et al.*, 2003). The presence of a peptide group in the compound causes it to possess diverse biological activity and some of these compounds have been proposed as anticancer drugs (Protsen *et al.*, 1971). Phosphate, phosphonate and phosphoramide functionalies are also used in enhancing the solubility of drugs and in some cases they function as pro-drugs (Cho *et al.*, 1982; Banaszczyk *et al.*, 2002; Pettit *et al.*, 2005; Hu *et al.*, 2003). They have also been used in the synthesis of aminophosphonic acids and aminoalkylphosphonic acids, respectively, as surrogates for the corresponding amino acids in biological systems (Hamilton *et al.*, 1995; Hammerschmidt & Hanbauer 2000). Here, we report on the synthesis, spectroscopic characterization and the crystal structure of the novel phosphate, $[(CH_3)_2CHNH_2CH_2C_6H_5][CF_3C(O)NHP(O)(O)NCH(CH_3)_2CH_2C_6H_5]$, which contains both amine and peptide moieties linked to a P atom (Figure 1). The C1—O3 bond length of 1.217 (3) Å is typical for a peptide C=O bond and the P1—O1 = 1.4824 (16) Å and P1—O2 = 1.4961 (16) Å bond lengths are shorter than normal P—O single bonds. The P1—N1 distance of 1.740 (2) Å is clearly consistent with a P—N single bond (Corbridge, 1995), whereas P1—N2 = 1.641 (2) Å shows partial double bond character. The sum of the surrounding angles around the atom N2 atom is close to 360° (357.9°), suggesting considerable sp^2 character. The P···P distance between two anions linked by two hydrogen bonds is 4.569 Å. In the crystal structure, cations and anions are connected *via* intermolecular N—H···O hydrogen bonds to form chains of centrosymmetric rings propagating in the *b* axis direction (Fig. 2). These chains, are in turn, connected *via* weak C15—H15A···O3 hydrogen bonds, C15···O3^{iii} = 3.402 Å; H15A···O3^{iii} = 2.53Å and C15—H15A···O3^{iii} = 153° [symmetry code: (iii) $x + 1, y, z$], see Figure 3.

Experimental

$CF_3C(O)N(H)P(O)Cl_2$ was prepared similar to the literature method (Shokol *et al.*, 1969) from the reaction of phosphorus pentachloride and 2,2,2-triflouoroacetamide in CCl_4 and then the treatment of formic acid. Synthesis of $[(CH_3)_2CHNH_2CH_2C_6H_5][CF_3C(O)NHP(O)(O)NCH(CH_3)_2CH_2C_6H_5]$ To a solution of (1.15 g, 5 mmol) triflouroacetyl phosphoramidic dichloride in CCl_4 (20 ml), a solution of *N*-isopropyl benzylamine (2.98 g, 20 mmol) in CCl_4 (10 ml) was added dropwise at 273 K. After 24 h, the solvent removed in vacuum and the solid washed with distilled water. The residue recrystallized in CH_3CN. Anal. Calc. for $C_{22}H_{31}F_3N_3O_3P$: C, 55.80; H, 6.55; N, 8.88. Found: C, 54.69; H, 6.14; N, 8.52%. ^{31}P NMR ([D$_6$]DMSO): −2.44. ^{13}C NMR ([D$_6$]DMSO): 18.44 (s, 2 C, CH_3), 21.54 (d, ^3J(P,*C*) = 2.6 Hz, 2 C, CH_3), 45.86 (*d*), 46.58 (d, ^2J(P,*C*) = 5.1 Hz), 47.20 (*s*), 49.09 (*s*), 125.47 (*s*), 127.19 (*s*), 127.34 (*s*), 128.41 (*s*), 128.51 (*s*), 129.78 (*s*), 132.50 (*s*). ^1H NMR ([D$_6$]DMSO): 0.84 (d, ^3J(H,H) = 6.7 Hz, 6H, $2CH_3$), 1.25 (d, ^3J(H,H) = 6.5 Hz, 6H, $2CH_3$), 3.19 (m, 1H, CH), 3.86 (m, 1H, CH), 4.04 (s, 2H, CH_2), 4.25 (d, ^3J(P,H) = 11.2 Hz, 2H, CH_2), 7.10 (t, ^3J(H,H) = 7.3 Hz, 1H, Ar—H),

7.20 (t, ^{3}J(H,H) = 7.5 Hz, 2H, Ar—H), 7.36–7.59 (m, 7H, Ar—H), 9.07 (b, 1H, NH), 9.22 (b, 2H, NH_2). IR (KBr, cm^{-1}): 3405, 3025, 2900, 2760, 2425, 1698 (C=O), 1595, 1480, 1450, 1366, 1312, 1244, 1213, 1171, 1092, 908, 928, 658.

Refinement

The hydrogen atoms of NH_2 and NH groups were found in difference Fourier maps but were subsequently placed in idealized geometry with N—H = 0.85 Å. The H(C) atom positions were calculated with C—H = 0.93–0.98 Å. All hydrogen atoms were refined in isotropic approximation in the riding-model with the U_{iso}(H) parameters equal to 1.2 U_{eq}(Ci) (or 1.5U_{eq}(Ci) for methyl C atoms) where U_{eq}(Ci) are the equivalent thermal parameters of the atoms to which corresponding H atoms are bonded.

Figures

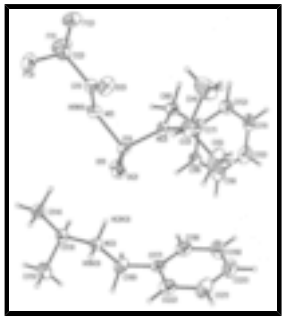


Fig. 1. The molecular structure of the title compound with thermal ellipsoid at 50% probability level.

Fig. 2. The crystal packing, showing hydrogen-bonded centrosymmetric rings propagating in the *b* axis direction. Hydrogen bonds are shown as dashed lines.

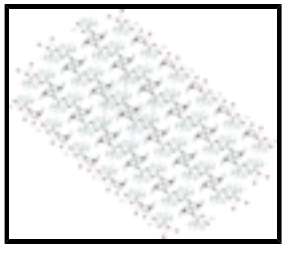


Fig. 3. Connection of the extended hydrogen bonded chains in crystal network *via* weak C15—H15A···O3 hydrogen bonds, H atoms are omitted for clarity. Red and blue dashed lines indicate hydrogen bonds.

N-Benzylpropan-2-aminium (benzylisopropylamido)(2,2,2-trifluoroacetamido)phosphate

Crystal data

$C_{10}H_{16}N^+ \cdot C_{12}H_{15}F_3N_2O_3P^-$	$Z = 2$
$M_r = 473.47$	$F_{000} = 500$
Triclinic, $P\overline{1}$	$D_x = 1.343$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å

a = 9.6147 (18) Å	Cell parameters from 1506 reflections
b = 10.027 (2) Å	θ = 5.8–45.6º
c = 13.503 (3) Å	μ = 0.17 mm^{-1}
α = 68.355 (4)º	T = 150 (2) K
β = 75.939 (4)º	Needle, colourless
γ = 88.494 (4)º	0.55 × 0.30 × 0.25 mm
V = 1170.9 (4) $Å^3$	

Data collection

Bruker APEXII CCD area-detector diffractometer	3060 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.081
Monochromator: graphite	θ_{max} = 29.0º
T = 150(2) K	θ_{min} = 1.7º
φ and ω scans	h = −13→13
Absorption correction: none	k = −13→13
14154 measured reflections	l = −18→18
6183 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)]$ = 0.054	H-atom parameters constrained
$wR(F^2)$ = 0.103	$w = 1/[\sigma^2(F_o^2) + (0.0285P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max} < 0.001$
6183 reflections	$\Delta\rho_{max}$ = 0.26 e $Å^{-3}$
289 parameters	$\Delta\rho_{min}$ = −0.46 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.44594 (7)	0.23272 (7)	0.42151 (5)	0.01853 (16)
F1	0.10818 (15)	−0.11604 (15)	0.63153 (13)	0.0370 (4)
F2	−0.06390 (14)	0.02192 (15)	0.60075 (12)	0.0349 (4)
F3	0.04486 (16)	0.02252 (16)	0.72108 (12)	0.0406 (4)
N1	0.3090 (2)	0.11036 (19)	0.51924 (16)	0.0195 (5)
H1N1	0.3407	0.0272	0.5404	0.023*
N2	0.4138 (2)	0.2526 (2)	0.30309 (16)	0.0209 (5)
N3	0.70019 (19)	0.41357 (19)	0.49447 (15)	0.0208 (5)
H1N3	0.6752	0.4876	0.5090	0.025*
H2N3	0.6316	0.3955	0.4707	0.025*
O1	0.57863 (16)	0.15789 (16)	0.43698 (13)	0.0220 (4)
O2	0.43257 (16)	0.37217 (16)	0.43854 (13)	0.0229 (4)
O3	0.12192 (18)	0.25182 (17)	0.49109 (14)	0.0319 (5)
C1	0.1712 (3)	0.1383 (3)	0.5349 (2)	0.0222 (6)
C2	0.0649 (3)	0.0146 (3)	0.6219 (2)	0.0245 (6)
C3	0.3566 (3)	0.3843 (3)	0.2365 (2)	0.0286 (6)
H3A	0.3152	0.4338	0.2862	0.034*
C4	0.2356 (3)	0.3524 (3)	0.1932 (3)	0.0594 (10)
H4A	0.1629	0.2885	0.2532	0.089*
H4B	0.2723	0.3082	0.1410	0.089*
H4C	0.1949	0.4405	0.1580	0.089*
C5	0.4741 (3)	0.4858 (3)	0.1473 (2)	0.0513 (9)
H5A	0.5476	0.5034	0.1789	0.077*
H5B	0.4352	0.5749	0.1118	0.077*
H5C	0.5146	0.4438	0.0942	0.077*
C6	0.4310 (3)	0.1295 (3)	0.2680 (2)	0.0245 (6)
H6A	0.3430	0.1109	0.2508	0.029*
H6B	0.4440	0.0453	0.3292	0.029*
C7	0.5554 (3)	0.1488 (2)	0.1694 (2)	0.0230 (6)
C8	0.6935 (3)	0.1853 (3)	0.1699 (2)	0.0284 (6)
H8A	0.7092	0.1982	0.2312	0.034*
C9	0.8073 (3)	0.2025 (3)	0.0806 (2)	0.0359 (7)
H9A	0.8992	0.2272	0.0821	0.043*
C10	0.7865 (3)	0.1833 (3)	−0.0115 (2)	0.0375 (7)
H10A	0.8635	0.1951	−0.0717	0.045*
C11	0.6502 (3)	0.1466 (3)	−0.0123 (2)	0.0358 (7)
H11A	0.6351	0.1332	−0.0736	0.043*
C12	0.5350 (3)	0.1293 (3)	0.0771 (2)	0.0296 (6)
H12A	0.4434	0.1045	0.0754	0.036*
C13	0.7003 (2)	0.2879 (2)	0.59973 (19)	0.0221 (6)
H13A	0.7228	0.2025	0.5815	0.026*
C14	0.5517 (3)	0.2611 (3)	0.6757 (2)	0.0284 (6)
H14A	0.4835	0.2438	0.6396	0.043*
H14B	0.5275	0.3438	0.6945	0.043*
H14C	0.5493	0.1788	0.7415	0.043*

C15	0.8153 (3)	0.3158 (3)	0.6505 (2)	0.0324 (7)
H15A	0.9073	0.3320	0.5987	0.049*
H15B	0.8163	0.2340	0.7158	0.049*
H15C	0.7950	0.3992	0.6690	0.049*
C16	0.8400 (3)	0.4436 (3)	0.4097 (2)	0.0296 (6)
H16A	0.8743	0.3534	0.4058	0.035*
H16B	0.9099	0.4862	0.4324	0.035*
C17	0.8300 (2)	0.5428 (3)	0.2971 (2)	0.0237 (6)
C18	0.8503 (3)	0.4934 (3)	0.2117 (2)	0.0347 (7)
H18A	0.8678	0.3976	0.2247	0.042*
C19	0.8447 (3)	0.5848 (3)	0.1078 (2)	0.0446 (8)
H19A	0.8589	0.5502	0.0512	0.054*
C20	0.8182 (3)	0.7265 (3)	0.0871 (2)	0.0398 (7)
H20A	0.8129	0.7872	0.0170	0.048*
C21	0.7997 (3)	0.7782 (3)	0.1697 (2)	0.0397 (7)
H21A	0.7829	0.8744	0.1556	0.048*
C22	0.8062 (3)	0.6868 (3)	0.2749 (2)	0.0326 (7)
H22A	0.7944	0.7225	0.3307	0.039*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0193 (3)	0.0139 (3)	0.0226 (4)	0.0020 (3)	−0.0048 (3)	−0.0074 (3)
F1	0.0271 (8)	0.0209 (8)	0.0559 (11)	0.0019 (7)	−0.0068 (8)	−0.0087 (8)
F2	0.0181 (8)	0.0375 (9)	0.0471 (10)	0.0025 (7)	−0.0071 (7)	−0.0142 (8)
F3	0.0442 (10)	0.0440 (10)	0.0257 (9)	−0.0071 (8)	−0.0006 (7)	−0.0086 (8)
N1	0.0200 (11)	0.0132 (10)	0.0259 (12)	0.0059 (8)	−0.0071 (9)	−0.0074 (9)
N2	0.0265 (12)	0.0183 (11)	0.0202 (12)	0.0063 (9)	−0.0086 (9)	−0.0083 (9)
N3	0.0195 (11)	0.0181 (11)	0.0271 (12)	0.0032 (9)	−0.0070 (9)	−0.0105 (9)
O1	0.0180 (9)	0.0180 (9)	0.0297 (10)	0.0035 (7)	−0.0073 (7)	−0.0080 (8)
O2	0.0264 (10)	0.0170 (9)	0.0268 (10)	0.0007 (7)	−0.0063 (8)	−0.0100 (8)
O3	0.0285 (10)	0.0235 (10)	0.0349 (11)	0.0099 (8)	−0.0054 (9)	−0.0030 (9)
C1	0.0217 (14)	0.0221 (13)	0.0241 (14)	0.0037 (11)	−0.0044 (11)	−0.0111 (12)
C2	0.0201 (14)	0.0254 (14)	0.0278 (16)	0.0041 (11)	−0.0060 (11)	−0.0098 (12)
C3	0.0346 (16)	0.0255 (14)	0.0265 (15)	0.0122 (12)	−0.0116 (13)	−0.0086 (12)
C4	0.059 (2)	0.053 (2)	0.079 (3)	0.0222 (18)	−0.047 (2)	−0.021 (2)
C5	0.059 (2)	0.0303 (17)	0.043 (2)	0.0094 (16)	−0.0025 (17)	0.0032 (15)
C6	0.0277 (14)	0.0194 (13)	0.0279 (15)	0.0012 (11)	−0.0070 (12)	−0.0107 (12)
C7	0.0322 (15)	0.0150 (12)	0.0218 (14)	0.0034 (11)	−0.0077 (11)	−0.0064 (11)
C8	0.0310 (15)	0.0296 (15)	0.0277 (16)	0.0044 (12)	−0.0082 (12)	−0.0137 (13)
C9	0.0337 (16)	0.0395 (17)	0.0344 (17)	0.0030 (13)	−0.0054 (14)	−0.0158 (14)
C10	0.0475 (19)	0.0290 (15)	0.0292 (17)	0.0082 (14)	−0.0002 (14)	−0.0096 (13)
C11	0.057 (2)	0.0293 (16)	0.0242 (16)	0.0030 (14)	−0.0102 (14)	−0.0132 (13)
C12	0.0374 (16)	0.0238 (14)	0.0303 (16)	0.0008 (12)	−0.0099 (13)	−0.0121 (13)
C13	0.0281 (14)	0.0159 (12)	0.0230 (14)	0.0059 (11)	−0.0092 (11)	−0.0067 (11)
C14	0.0295 (15)	0.0266 (14)	0.0278 (15)	0.0010 (12)	−0.0064 (12)	−0.0091 (13)
C15	0.0364 (16)	0.0348 (16)	0.0328 (16)	0.0110 (13)	−0.0135 (13)	−0.0177 (14)
C16	0.0200 (14)	0.0314 (15)	0.0307 (16)	0.0041 (12)	0.0017 (12)	−0.0095 (13)

C17	0.0175 (13)	0.0249 (14)	0.0282 (15)	−0.0031 (11)	−0.0017 (11)	−0.0116 (12)
C18	0.0377 (17)	0.0284 (15)	0.0384 (18)	−0.0005 (13)	−0.0028 (14)	−0.0171 (14)
C19	0.0464 (19)	0.059 (2)	0.0313 (18)	−0.0018 (17)	−0.0053 (15)	−0.0226 (17)
C20	0.0332 (17)	0.0457 (19)	0.0296 (17)	−0.0014 (15)	−0.0044 (13)	−0.0037 (15)
C21	0.0424 (18)	0.0256 (15)	0.0401 (19)	0.0064 (14)	−0.0057 (15)	−0.0031 (15)
C22	0.0321 (16)	0.0283 (15)	0.0391 (18)	0.0017 (13)	−0.0044 (13)	−0.0174 (14)

Geometric parameters (Å, °)

P1—O1	1.4824 (16)	C8—H8A	0.9300
P1—O2	1.4961 (16)	C9—C10	1.386 (4)
P1—N2	1.641 (2)	C9—H9A	0.9300
P1—N1	1.740 (2)	C10—C11	1.374 (4)
F1—C2	1.333 (3)	C10—H10A	0.9300
F2—C2	1.331 (3)	C11—C12	1.386 (4)
F3—C2	1.338 (3)	C11—H11A	0.9300
N1—C1	1.330 (3)	C12—H12A	0.9300
N1—H1N1	0.8500	C13—C14	1.507 (3)
N2—C6	1.469 (3)	C13—C15	1.515 (3)
N2—C3	1.477 (3)	C13—H13A	0.9800
N3—C16	1.492 (3)	C14—H14A	0.9600
N3—C13	1.512 (3)	C14—H14B	0.9600
N3—H1N3	0.8501	C14—H14C	0.9600
N3—H2N3	0.8500	C15—H15A	0.9600
O3—C1	1.217 (3)	C15—H15B	0.9600
C1—C2	1.540 (3)	C15—H15C	0.9600
C3—C5	1.503 (4)	C16—C17	1.504 (3)
C3—C4	1.511 (4)	C16—H16A	0.9700
C3—H3A	0.9800	C16—H16B	0.9700
C4—H4A	0.9600	C17—C18	1.384 (4)
C4—H4B	0.9600	C17—C22	1.387 (3)
C4—H4C	0.9600	C18—C19	1.378 (4)
C5—H5A	0.9600	C18—H18A	0.9300
C5—H5B	0.9600	C19—C20	1.373 (4)
C5—H5C	0.9600	C19—H19A	0.9300
C6—C7	1.514 (3)	C20—C21	1.368 (4)
C6—H6A	0.9700	C20—H20A	0.9300
C6—H6B	0.9700	C21—C22	1.394 (4)
C7—C12	1.388 (3)	C21—H21A	0.9300
C7—C8	1.388 (3)	C22—H22A	0.9300
C8—C9	1.378 (4)		
O1—P1—O2	117.01 (9)	C7—C8—H8A	119.7
O1—P1—N2	112.16 (10)	C8—C9—C10	120.7 (3)
O2—P1—N2	109.92 (10)	C8—C9—H9A	119.7
O1—P1—N1	103.82 (9)	C10—C9—H9A	119.7
O2—P1—N1	108.49 (9)	C11—C10—C9	119.0 (3)
N2—P1—N1	104.44 (10)	C11—C10—H10A	120.5
C1—N1—P1	123.04 (16)	C9—C10—H10A	120.5
C1—N1—H1N1	125.6	C10—C11—C12	120.7 (3)

P1—N1—H1N1	109.3	C10—C11—H11A	119.6
C6—N2—C3	119.4 (2)	C12—C11—H11A	119.6
C6—N2—P1	118.09 (16)	C11—C12—C7	120.5 (3)
C3—N2—P1	122.23 (16)	C11—C12—H12A	119.8
C16—N3—C13	113.40 (18)	C7—C12—H12A	119.8
C16—N3—H1N3	111.3	C14—C13—N3	108.73 (18)
C13—N3—H1N3	109.7	C14—C13—C15	113.1 (2)
C16—N3—H2N3	111.6	N3—C13—C15	110.31 (19)
C13—N3—H2N3	107.2	C14—C13—H13A	108.2
H1N3—N3—H2N3	103.1	N3—C13—H13A	108.2
O3—C1—N1	126.9 (2)	C15—C13—H13A	108.2
O3—C1—C2	117.4 (2)	C13—C14—H14A	109.5
N1—C1—C2	115.6 (2)	C13—C14—H14B	109.5
F2—C2—F1	107.4 (2)	H14A—C14—H14B	109.5
F2—C2—F3	106.4 (2)	C13—C14—H14C	109.5
F1—C2—F3	106.8 (2)	H14A—C14—H14C	109.5
F2—C2—C1	111.7 (2)	H14B—C14—H14C	109.5
F1—C2—C1	114.0 (2)	C13—C15—H15A	109.5
F3—C2—C1	110.1 (2)	C13—C15—H15B	109.5
N2—C3—C5	111.6 (2)	H15A—C15—H15B	109.5
N2—C3—C4	112.7 (2)	C13—C15—H15C	109.5
C5—C3—C4	112.6 (2)	H15A—C15—H15C	109.5
N2—C3—H3A	106.4	H15B—C15—H15C	109.5
C5—C3—H3A	106.4	N3—C16—C17	113.3 (2)
C4—C3—H3A	106.4	N3—C16—H16A	108.9
C3—C4—H4A	109.5	C17—C16—H16A	108.9
C3—C4—H4B	109.5	N3—C16—H16B	108.9
H4A—C4—H4B	109.5	C17—C16—H16B	108.9
C3—C4—H4C	109.5	H16A—C16—H16B	107.7
H4A—C4—H4C	109.5	C18—C17—C22	118.4 (2)
H4B—C4—H4C	109.5	C18—C17—C16	120.6 (2)
C3—C5—H5A	109.5	C22—C17—C16	121.0 (2)
C3—C5—H5B	109.5	C19—C18—C17	120.7 (3)
H5A—C5—H5B	109.5	C19—C18—H18A	119.6
C3—C5—H5C	109.5	C17—C18—H18A	119.6
H5A—C5—H5C	109.5	C20—C19—C18	120.5 (3)
H5B—C5—H5C	109.5	C20—C19—H19A	119.8
N2—C6—C7	114.8 (2)	C18—C19—H19A	119.8
N2—C6—H6A	108.6	C21—C20—C19	119.8 (3)
C7—C6—H6A	108.6	C21—C20—H20A	120.1
N2—C6—H6B	108.6	C19—C20—H20A	120.1
C7—C6—H6B	108.6	C20—C21—C22	120.0 (3)
H6A—C6—H6B	107.5	C20—C21—H21A	120.0
C12—C7—C8	118.5 (2)	C22—C21—H21A	120.0
C12—C7—C6	121.2 (2)	C17—C22—C21	120.5 (3)
C8—C7—C6	120.3 (2)	C17—C22—H22A	119.7
C9—C8—C7	120.6 (3)	C21—C22—H22A	119.7
C9—C8—H8A	119.7		
O1—P1—N1—C1	177.84 (18)	N2—C6—C7—C12	−126.2 (2)

O2—P1—N1—C1	52.7 (2)	N2—C6—C7—C8	54.5 (3)
N2—P1—N1—C1	−64.5 (2)	C12—C7—C8—C9	0.4 (4)
O1—P1—N2—C6	44.1 (2)	C6—C7—C8—C9	179.7 (2)
O2—P1—N2—C6	176.07 (16)	C7—C8—C9—C10	−0.2 (4)
N1—P1—N2—C6	−67.71 (19)	C8—C9—C10—C11	−0.1 (4)
O1—P1—N2—C3	−141.44 (18)	C9—C10—C11—C12	0.2 (4)
O2—P1—N2—C3	−9.4 (2)	C10—C11—C12—C7	0.0 (4)
N1—P1—N2—C3	106.77 (19)	C8—C7—C12—C11	−0.3 (4)
P1—N1—C1—O3	−5.3 (4)	C6—C7—C12—C11	−179.6 (2)
P1—N1—C1—C2	177.62 (17)	C16—N3—C13—C14	−176.1 (2)
O3—C1—C2—F2	30.5 (3)	C16—N3—C13—C15	59.4 (3)
N1—C1—C2—F2	−152.2 (2)	C13—N3—C16—C17	165.9 (2)
O3—C1—C2—F1	152.4 (2)	N3—C16—C17—C18	−113.5 (3)
N1—C1—C2—F1	−30.2 (3)	N3—C16—C17—C22	69.1 (3)
O3—C1—C2—F3	−87.5 (3)	C22—C17—C18—C19	−1.0 (4)
N1—C1—C2—F3	89.9 (2)	C16—C17—C18—C19	−178.4 (2)
C6—N2—C3—C5	−88.0 (3)	C17—C18—C19—C20	−0.3 (4)
P1—N2—C3—C5	97.6 (2)	C18—C19—C20—C21	1.1 (4)
C6—N2—C3—C4	39.9 (3)	C19—C20—C21—C22	−0.7 (4)
P1—N2—C3—C4	−134.5 (2)	C18—C17—C22—C21	1.4 (4)
C3—N2—C6—C7	73.6 (3)	C16—C17—C22—C21	178.8 (2)
P1—N2—C6—C7	−111.8 (2)	C20—C21—C22—C17	−0.6 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O1^{i}	0.85	1.94	2.781 (3)	170
N3—H1N3···O2^{ii}	0.85	1.96	2.791 (3)	164
N3—H2N3···O2	0.85	2.10	2.932 (3)	166

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+1.

Fig. 1

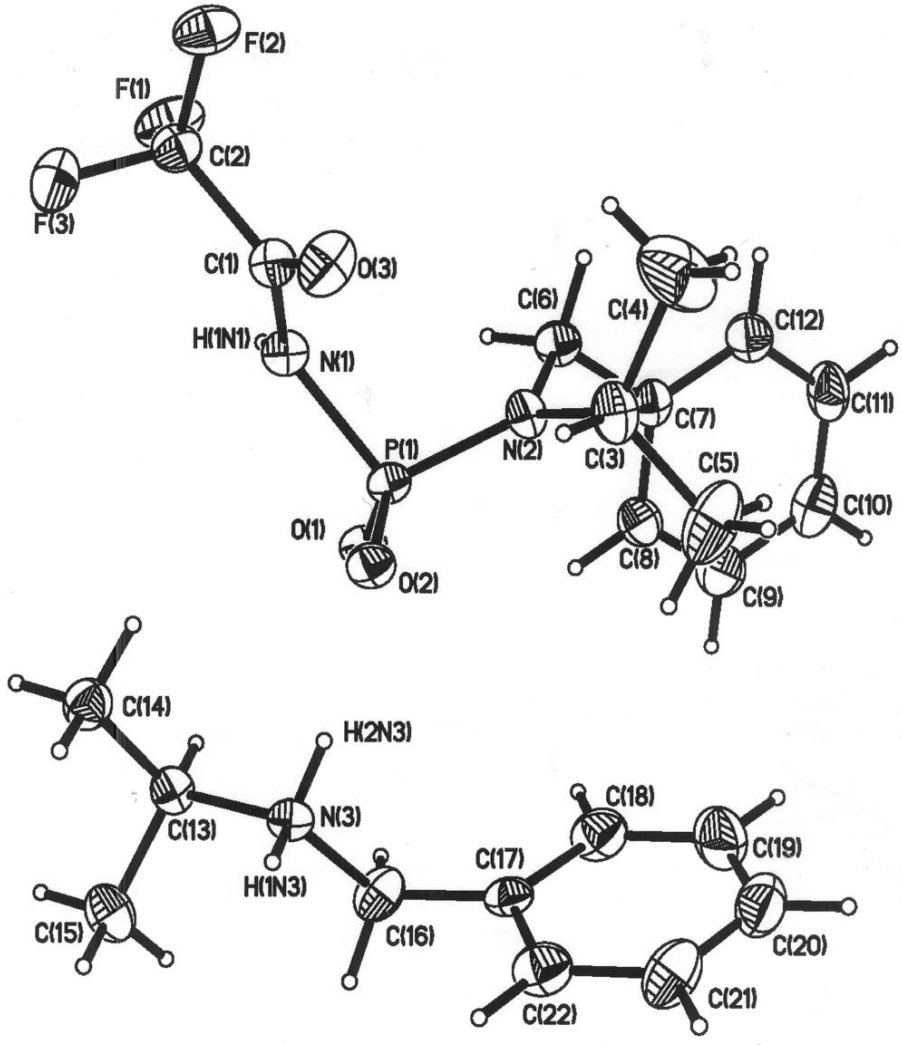

F(2)
F(1)
C(2)
F(3)
C(1)
O(3)
H(1N1)
N(1)
C(6)
C(4)
C(12)
C(11)
C(7)
P(1)
N(2)
C(3)
C(5)
C(10)
O(1)
C(8)
O(2)
C(9)
C(14)
H(2N3)
C(18)
C(13)
N(3)
C(19)
C(17)
H(1N3)
C(15)
C(16)
C(20)
C(22)
C(21)

Fig. 2

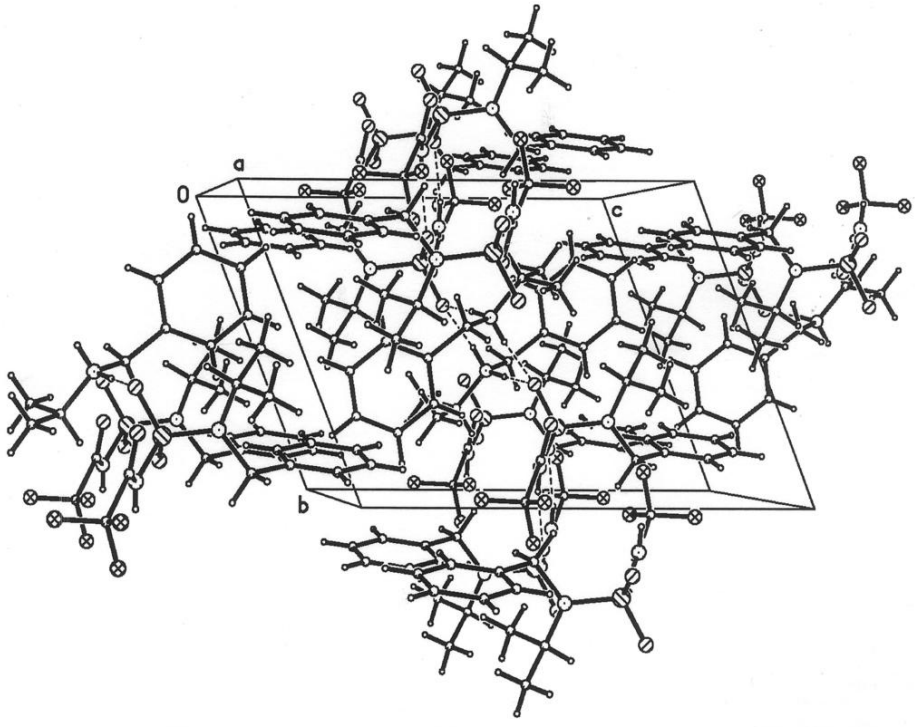


Fig. 3