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1,3-Bis(2,6-diisopropylanilino)-1-phenylbutylium hexafluoridophosphate

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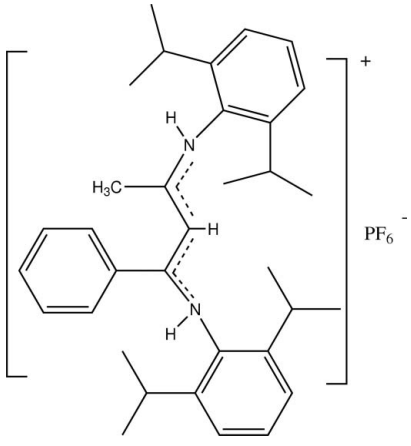
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.060; wR factor = 0.175; data-to-parameter ratio = 14.3.

The cation of the title salt, $C_{34}H_{45}N_2^+\cdot PF_6^-$, is a protonated form of an unsymmetrical overcrowded β-iminoamine. The observed bond lengths [C—N = 1.326 (4)–1.341 (4) Å and C—C = 1.383 (4)–1.391 (4) Å] suggest significant delocalization within the π system of the N···C···C···C···N backbone.

Related literature

For related literature, see: Allen *et al.* (1987); Bourget-Merle *et al.* (2002); Filipou *et al.* (1993); Landolsi *et al.* (2002, 2008); Mair *et al.* (1995); Parks & Holm (1968).



Experimental

Crystal data

$C_{34}H_{45}N_2^+\cdot F_6P^-$
M_r = 626.69
Monoclinic, $P2_1/n$
a = 12.4688 (2) Å
b = 15.6981 (2) Å
c = 17.4329 (3) Å
β = 95.563 (3)°
V = 3396.18 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 (2) K
0.31 × 0.21 × 0.16 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
Absorption correction: none
10101 measured reflections
5966 independent reflections
3165 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
2 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.175
S = 1.02
5966 reflections
416 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected bond lengths (Å).

C3—N2	1.341 (4)	C2—C1	1.391 (4)
C3—C2	1.383 (4)	C1—N1	1.326 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H*N*1···F2	0.86 (3)	2.28 (4)	3.094 (4)	156 (3)
N1—H*N*1···F4	0.86 (3)	2.49 (4)	3.187 (4)	139 (3)

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

The authors thank Chtioui Ahlem for the structure refinement.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2111).

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supplementary materials

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1,3-Bis(2,6-diisopropylanilino)-1-phenylbutylium hexafluoridophosphate

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Comment

β-Iminoamine complexes or β-diketiminato complexes (also known as diazapentadienyl complexes) were first reported in 1968 (Parks *et al.*, 1968) and a few reports of their structural characterizations appeared since then (Mair *et al.*, 1995; Filipou *et al.*, 1993). Attention to their structural chemistry is quite recent. The β-diketiminates have been recognized as ancillary ligands owing to their exceptional steric and electronic properties that can be readily modified by variation of the substituents in the main framework (Bourget-Merle *et al.*, 2002). Recently we have been interested in synthesis of new unsymmetrical β-iminoamines with bulky substituents attached to the nitrogen atoms and their coordination chemistry (Landolsi *et al.*, 2002). Here we report the crystal structure of the hexafluorophosphate salt of overcrowded β-iminoamine.

The PF_6^- anion possess an octahedral geometry with the P—F distances ranging from 1.525 (3) to 1.589 (3) Å. The cation shows the W-shaped open conformation whereas the neutral form exhibit the U-shaped closed conformation (Landolsi *et al.*, 2008). The N—C and the C—C bond distances of the N—C—C—C—N backbone (Table 1) are intermediate between single and double-bond lengths, that suggests significant delocalization within the π-system (Allen *et al.*, 1987). One N—C bond is longer than the other, this difference can possibly be attributed to the different groups attached to C1 and C3. This bis-(iminium) salt can be used as prospective starting materiels for organoelement and coordination chemistry.

Experimental

The title compound was obtained as a result of attempted recrystallization of the hexafluorophosphate methallyl β-diimine nickel complex, (C_4H_7) Ni ($C_{34}H_{44}N_2$) PF_6 (100 mg, 0.135 mmoles) from methylene chloride (15 ml)\acetic acid (0.05%) mixture, which resulted in decomposition of the complex. The Ni complex was prepared by an oxidative addition of the hexafluorophosphate methallyloxyphosphonium salt (54 mg, 0.143 mmoles) to the (β-diimine)Ni spieces generated *in situ* by the chemical reduction of (β-diimine)$NiBr_2$ (100 mg, 0.143 mmoles) with zinc (190 mg, 2.860 mmoles) in methylene chloride (20 ml). Crystals for X-ray analysis were obtained from a diluted solution of the title compound (10 mg, 0.013 mmoles) in methylene chloride /n-hexane (15 ml/15 ml) at 243 K.

Refinement

Hydrogen atoms H2, HN1 and HN2 were located in a Fourier map and refined freely. All the other H atoms were placed in calculated positions and allowed to ride on their parent atom. U_{iso} of H atoms are equal to 1.2 U_{eq} of the parent atom.

Figures

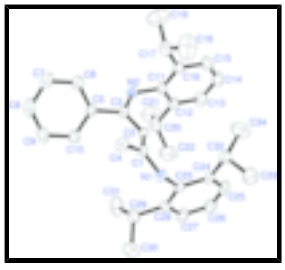


Fig. 1. View of the cation with displacement ellipsoids drawn at the 30% probability level. H atoms and the hexaflororphosphate anion are omitted for clarity.

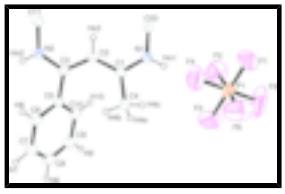


Fig. 2. View of the anion and a fragment of the cation with displacement ellipsoids are drawn at the 30% probability level.

1,3-Bis(2,6-diisopropylanilino)-1-phenylbutylium hexafluorophosphate

Crystal data

$C_{34}H_{45}N_2^+ \cdot F_6P^-$	F_{000} = 1328
M_r = 626.69	D_x = 1.226 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 25 reflections
a = 12.4688 (2) Å	θ = 9.3–11.3º
b = 15.6981 (2) Å	μ = 0.14 mm^{-1}
c = 17.4329 (3) Å	T = 293 (2) K
β = 95.563 (3)º	Prism, colourless
V = 3396.18 (9) Å^3	0.31 × 0.21 × 0.16 mm
Z = 4	

Data collection

Enraf–Nonius TurboCAD-4 diffractometer	R_{int} = 0.028
Radiation source: fine-focus sealed tube	θ_{max} = 25.0º
Monochromator: graphite	θ_{min} = 2.1º
T = 293(2) K	h = −14→14
non–profiled ω scans	k = 0→18
Absorption correction: none	l = −10→20
10101 measured reflections	2 standard reflections
5966 independent reflections	every 120 min
3165 reflections with $I > 2\sigma(I)$	intensity decay: 1%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.060$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.175$	$w = 1/[\sigma^2(F_o^2) + (0.0756P)^2 + 1.269P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.003$
5966 reflections	$\Delta\rho_{max} = 0.45$ e Å^{-3}
416 parameters	$\Delta\rho_{min} = -0.27$ 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	−0.00211 (8)	0.84236 (7)	0.17439 (7)	0.0746 (4)
F1	−0.0452 (2)	0.91779 (17)	0.22077 (19)	0.1208 (10)
F2	0.0651 (3)	0.8051 (2)	0.24702 (19)	0.1345 (11)
F3	0.0425 (3)	0.76825 (19)	0.1260 (2)	0.1384 (12)
F4	0.1024 (2)	0.8969 (2)	0.1625 (2)	0.1362 (12)
F5	−0.1013 (2)	0.7896 (2)	0.1879 (3)	0.198 (2)
F6	−0.0594 (3)	0.8823 (2)	0.1000 (2)	0.1797 (18)
N1	0.3134 (2)	0.81810 (16)	0.25332 (15)	0.0420 (6)
HN1	0.246 (3)	0.8131 (19)	0.2369 (18)	0.051*
N2	0.67098 (19)	0.85742 (16)	0.19588 (15)	0.0445 (7)
HN2	0.724 (3)	0.838 (2)	0.1770 (19)	0.054*
C1	0.3830 (2)	0.81205 (18)	0.20064 (17)	0.0393 (7)
C2	0.4909 (2)	0.83195 (19)	0.22012 (19)	0.0414 (7)
HC2	0.506 (2)	0.8615 (19)	0.2632 (18)	0.050*
C3	0.5788 (2)	0.81410 (18)	0.18015 (17)	0.0394 (7)
C4	0.3338 (2)	0.7888 (2)	0.12127 (18)	0.0532 (9)
H4A	0.3227	0.7283	0.1183	0.065*

H4B	0.3813	0.8058	0.0839	0.065*
H4C	0.2659	0.8174	0.1109	0.065*
C5	0.5816 (2)	0.74619 (19)	0.12192 (18)	0.0416 (7)
C6	0.6244 (3)	0.7605 (2)	0.0529 (2)	0.0557 (9)
H6	0.6522	0.8138	0.0425	0.067*
C7	0.6261 (3)	0.6956 (3)	−0.0011 (2)	0.0697 (11)
H7	0.6537	0.7054	−0.0480	0.084*
C8	0.5868 (3)	0.6168 (3)	0.0153 (3)	0.0714 (12)
H8	0.5879	0.5733	−0.0207	0.086*
C9	0.5461 (3)	0.6015 (2)	0.0836 (3)	0.0696 (11)
H9	0.5206	0.5475	0.0942	0.084*
C10	0.5424 (2)	0.6658 (2)	0.1372 (2)	0.0545 (9)
H10	0.5137	0.6553	0.1836	0.066*
C11	0.6796 (2)	0.93436 (19)	0.24075 (19)	0.0447 (8)
C12	0.7108 (2)	0.9293 (2)	0.3193 (2)	0.0526 (9)
C13	0.7167 (3)	1.0056 (3)	0.3608 (2)	0.0648 (10)
H13	0.7364	1.0046	0.4136	0.078*
C14	0.6939 (3)	1.0818 (3)	0.3247 (3)	0.0692 (11)
H14	0.6987	1.1318	0.3533	0.084*
C15	0.6640 (3)	1.0855 (2)	0.2471 (3)	0.0664 (10)
H15	0.6495	1.1380	0.2237	0.080*
C16	0.6552 (2)	1.0113 (2)	0.2029 (2)	0.0535 (9)
C17	0.6231 (3)	1.0165 (3)	0.1165 (2)	0.0688 (11)
HC17	0.601 (3)	0.963 (3)	0.102 (2)	0.083*
C18	0.5244 (4)	1.0711 (4)	0.0970 (3)	0.128 (2)
H18A	0.5387	1.1281	0.1151	0.154*
H18B	0.4650	1.0480	0.1214	0.154*
H18C	0.5068	1.0718	0.0422	0.154*
C19	0.7163 (4)	1.0459 (4)	0.0744 (3)	0.127 (2)
H19A	0.6949	1.0467	0.0200	0.154*
H19B	0.7758	1.0075	0.0849	0.154*
H19C	0.7375	1.1021	0.0913	0.154*
C20	0.7387 (3)	0.8462 (3)	0.3608 (2)	0.0644 (10)
HC20	0.731 (3)	0.799 (2)	0.329 (2)	0.078*
C21	0.8559 (3)	0.8428 (3)	0.3926 (3)	0.1132 (18)
H21A	0.8700	0.8866	0.4307	0.137*
H21B	0.9007	0.8514	0.3515	0.137*
H21C	0.8714	0.7881	0.4158	0.137*
C22	0.6704 (4)	0.8301 (3)	0.4258 (3)	0.1090 (17)
H22A	0.6898	0.8696	0.4669	0.132*
H22B	0.6819	0.7730	0.4443	0.132*
H22C	0.5958	0.8376	0.4076	0.132*
C23	0.3434 (2)	0.83308 (19)	0.33457 (17)	0.0417 (7)
C24	0.3415 (2)	0.9163 (2)	0.36250 (19)	0.0474 (8)
C25	0.3743 (3)	0.9285 (2)	0.4401 (2)	0.0617 (10)
H25	0.3755	0.9833	0.4604	0.075*
C26	0.4052 (3)	0.8608 (3)	0.4875 (2)	0.0643 (10)
H26	0.4274	0.8705	0.5392	0.077*
C27	0.4036 (3)	0.7793 (2)	0.4590 (2)	0.0570 (9)

H27	0.4230	0.7342	0.4920	0.069*
C28	0.3734 (2)	0.7634 (2)	0.38158 (19)	0.0486 (8)
C29	0.3744 (3)	0.6724 (2)	0.3519 (2)	0.0620 (10)
HC29	0.355 (3)	0.674 (2)	0.296 (2)	0.075*
C30	0.2925 (4)	0.6185 (3)	0.3876 (3)	0.1124 (18)
H30A	0.3088	0.6177	0.4426	0.136*
H30B	0.2945	0.5615	0.3679	0.136*
H30C	0.2219	0.6421	0.3751	0.136*
C31	0.4860 (4)	0.6328 (3)	0.3650 (3)	0.1013 (16)
H31A	0.5062	0.6273	0.4193	0.122*
H31B	0.5371	0.6686	0.3426	0.17 (3)*
H31C	0.4853	0.5776	0.3413	0.122*
C32	0.3040 (3)	0.9907 (2)	0.3117 (2)	0.0571 (9)
H32	0.2955	0.9709	0.2582	0.069*
C33	0.1952 (3)	1.0217 (3)	0.3322 (3)	0.0838 (13)
H33A	0.1455	0.9748	0.3298	0.102*
H33B	0.1687	1.0650	0.2963	0.102*
H33C	0.2026	1.0448	0.3834	0.102*
C34	0.3843 (4)	1.0635 (3)	0.3173 (3)	0.1030 (16)
H34A	0.3875	1.0884	0.3677	0.125*
H34B	0.3622	1.1059	0.2793	0.125*
H34C	0.4542	1.0423	0.3083	0.125*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C3	0.0382 (16)	0.0388 (17)	0.0404 (18)	0.0018 (13)	−0.0006 (13)	−0.0030 (14)
C2	0.0384 (16)	0.0449 (19)	0.0411 (19)	−0.0019 (14)	0.0051 (14)	−0.0135 (15)
C1	0.0402 (16)	0.0375 (17)	0.0405 (18)	0.0040 (13)	0.0047 (14)	−0.0042 (14)
C4	0.0411 (18)	0.071 (2)	0.047 (2)	0.0035 (16)	0.0004 (15)	−0.0106 (18)
C32	0.066 (2)	0.050 (2)	0.057 (2)	0.0066 (17)	0.0131 (17)	−0.0037 (17)
C29	0.077 (3)	0.048 (2)	0.060 (2)	0.0017 (18)	0.004 (2)	0.0001 (19)
C20	0.070 (2)	0.063 (2)	0.058 (2)	−0.001 (2)	−0.0056 (19)	−0.013 (2)
C17	0.082 (3)	0.054 (2)	0.068 (3)	0.000 (2)	−0.003 (2)	0.000 (2)
C21	0.068 (3)	0.136 (4)	0.132 (5)	0.017 (3)	−0.004 (3)	0.041 (4)
C33	0.080 (3)	0.079 (3)	0.094 (3)	0.029 (2)	0.017 (2)	0.018 (2)
C30	0.132 (4)	0.083 (3)	0.128 (4)	−0.036 (3)	0.041 (4)	−0.019 (3)
C31	0.103 (4)	0.072 (3)	0.127 (4)	0.032 (3)	0.000 (3)	−0.022 (3)
C34	0.098 (3)	0.081 (3)	0.130 (4)	−0.023 (3)	0.010 (3)	0.021 (3)
C22	0.103 (4)	0.109 (4)	0.118 (4)	−0.003 (3)	0.027 (3)	0.033 (3)
C18	0.119 (4)	0.140 (5)	0.114 (5)	0.049 (4)	−0.041 (3)	−0.015 (4)
C19	0.121 (4)	0.178 (6)	0.085 (4)	−0.002 (4)	0.019 (3)	0.031 (4)
P1	0.0465 (5)	0.0677 (7)	0.1073 (10)	0.0088 (5)	−0.0044 (5)	−0.0161 (6)
N1	0.0325 (13)	0.0514 (16)	0.0417 (16)	0.0009 (12)	0.0010 (12)	−0.0082 (12)
N2	0.0324 (14)	0.0480 (16)	0.0534 (17)	−0.0004 (12)	0.0067 (12)	−0.0127 (13)
C23	0.0344 (15)	0.050 (2)	0.0410 (19)	0.0009 (14)	0.0076 (13)	−0.0072 (16)
C11	0.0322 (15)	0.0454 (19)	0.056 (2)	−0.0033 (13)	0.0019 (14)	−0.0139 (17)
C5	0.0329 (15)	0.0454 (18)	0.047 (2)	0.0011 (13)	0.0034 (14)	−0.0103 (15)

C16	0.0459 (18)	0.050 (2)	0.065 (2)	−0.0049 (15)	0.0063 (16)	−0.0103 (18)
C12	0.0423 (17)	0.058 (2)	0.057 (2)	−0.0085 (16)	0.0013 (16)	−0.0156 (19)
C28	0.0481 (18)	0.050 (2)	0.049 (2)	0.0015 (15)	0.0086 (15)	−0.0029 (17)
C24	0.0465 (18)	0.051 (2)	0.045 (2)	0.0051 (15)	0.0101 (15)	−0.0068 (16)
C10	0.0442 (18)	0.053 (2)	0.068 (2)	−0.0022 (15)	0.0104 (16)	−0.0136 (19)
C13	0.061 (2)	0.072 (3)	0.060 (2)	−0.0096 (19)	−0.0020 (18)	−0.022 (2)
F1	0.122 (2)	0.0880 (18)	0.158 (3)	0.0191 (16)	0.042 (2)	−0.0302 (18)
C27	0.062 (2)	0.065 (2)	0.045 (2)	0.0079 (18)	0.0055 (17)	0.0036 (18)
C14	0.060 (2)	0.059 (3)	0.088 (3)	−0.0107 (19)	0.007 (2)	−0.031 (2)
C6	0.0501 (19)	0.064 (2)	0.054 (2)	−0.0014 (16)	0.0091 (17)	−0.0124 (19)
C25	0.068 (2)	0.060 (2)	0.057 (3)	0.0043 (19)	0.0095 (19)	−0.018 (2)
C15	0.066 (2)	0.050 (2)	0.084 (3)	−0.0076 (18)	0.006 (2)	−0.010 (2)
C7	0.059 (2)	0.096 (3)	0.056 (2)	0.006 (2)	0.0129 (18)	−0.027 (2)
C26	0.069 (2)	0.083 (3)	0.041 (2)	0.006 (2)	0.0045 (18)	−0.010 (2)
C8	0.051 (2)	0.076 (3)	0.087 (3)	0.003 (2)	0.003 (2)	−0.045 (2)
F2	0.120 (2)	0.158 (3)	0.121 (2)	0.029 (2)	−0.0131 (19)	0.019 (2)
F3	0.135 (2)	0.117 (2)	0.159 (3)	0.0506 (19)	−0.014 (2)	−0.059 (2)
F4	0.098 (2)	0.119 (2)	0.196 (3)	−0.0125 (17)	0.036 (2)	−0.003 (2)
C9	0.049 (2)	0.053 (2)	0.107 (3)	−0.0063 (17)	0.013 (2)	−0.032 (2)
F5	0.0667 (18)	0.093 (2)	0.442 (7)	−0.0233 (15)	0.056 (3)	−0.030 (3)
F6	0.224 (4)	0.150 (3)	0.146 (3)	0.086 (3)	−0.077 (3)	−0.028 (2)

Geometric parameters (Å, °)

C3—N2	1.341 (4)	C18—H18B	0.9600
C3—C2	1.383 (4)	C18—H18C	0.9600
C3—C5	1.475 (4)	C19—H19A	0.9600
C2—C1	1.391 (4)	C19—H19B	0.9600
C2—HC2	0.89 (3)	C19—H19C	0.9600
C1—N1	1.326 (4)	P1—F5	1.525 (3)
C1—C4	1.503 (4)	P1—F6	1.551 (3)
C4—H4A	0.9600	P1—F1	1.558 (3)
C4—H4B	0.9600	P1—F2	1.563 (3)
C4—H4C	0.9600	P1—F3	1.570 (3)
C32—C24	1.513 (5)	P1—F4	1.589 (3)
C32—C33	1.515 (5)	N1—C23	1.449 (4)
C32—C34	1.516 (5)	N1—HN1	0.86 (3)
C32—H32	0.9800	N2—C11	1.437 (4)
C29—C30	1.507 (6)	N2—HN2	0.83 (3)
C29—C28	1.520 (5)	C23—C24	1.395 (4)
C29—C31	1.521 (5)	C23—C28	1.396 (4)
C29—HC29	0.98 (4)	C11—C12	1.390 (5)
C20—C22	1.503 (6)	C11—C16	1.396 (4)
C20—C21	1.513 (5)	C5—C6	1.382 (4)
C20—C12	1.516 (5)	C5—C10	1.388 (4)
C20—HC20	0.94 (4)	C16—C15	1.395 (5)
C17—C19	1.506 (6)	C12—C13	1.397 (5)
C17—C18	1.511 (6)	C28—C27	1.389 (4)
C17—C16	1.522 (5)	C24—C25	1.388 (5)

C17—HC17	0.91 (4)	C10—C9	1.379 (5)
C21—H21A	0.9600	C10—H10	0.9300
C21—H21B	0.9600	C13—C14	1.369 (5)
C21—H21C	0.9600	C13—H13	0.9300
C33—H33A	0.9600	C27—C26	1.372 (5)
C33—H33B	0.9600	C27—H27	0.9300
C33—H33C	0.9600	C14—C15	1.368 (5)
C30—H30A	0.9600	C14—H14	0.9300
C30—H30B	0.9600	C6—C7	1.388 (5)
C30—H30C	0.9600	C6—H6	0.9300
C31—H31A	0.9600	C25—C26	1.377 (5)
C31—H31B	0.9600	C25—H25	0.9300
C31—H31C	0.9600	C15—H15	0.9300
C34—H34A	0.9600	C7—C8	1.370 (5)
C34—H34B	0.9600	C7—H7	0.9300
C34—H34C	0.9600	C26—H26	0.9300
C22—H22A	0.9600	C8—C9	1.360 (5)
C22—H22B	0.9600	C8—H8	0.9300
C22—H22C	0.9600	C9—H9	0.9300
C18—H18A	0.9600		
N2—C3—C2	120.0 (3)	C17—C19—H19A	109.5
N2—C3—C5	115.5 (2)	C17—C19—H19B	109.5
C2—C3—C5	124.5 (3)	H19A—C19—H19B	109.5
C3—C2—C1	128.8 (3)	C17—C19—H19C	109.5
C3—C2—HC2	114.4 (19)	H19A—C19—H19C	109.5
C1—C2—HC2	116.7 (19)	H19B—C19—H19C	109.5
N1—C1—C2	119.8 (3)	F5—P1—F6	91.9 (3)
N1—C1—C4	114.7 (3)	F5—P1—F1	90.42 (18)
C2—C1—C4	125.3 (3)	F6—P1—F1	88.41 (18)
C1—C4—H4A	109.5	F5—P1—F2	92.9 (2)
C1—C4—H4B	109.5	F6—P1—F2	175.0 (2)
H4A—C4—H4B	109.5	F1—P1—F2	92.79 (19)
C1—C4—H4C	109.5	F5—P1—F3	90.9 (2)
H4A—C4—H4C	109.5	F6—P1—F3	90.54 (19)
H4B—C4—H4C	109.5	F1—P1—F3	178.4 (2)
C24—C32—C33	110.0 (3)	F2—P1—F3	88.15 (18)
C24—C32—C34	112.3 (3)	F5—P1—F4	178.6 (3)
C33—C32—C34	110.2 (3)	F6—P1—F4	89.4 (2)
C24—C32—H32	108.1	F1—P1—F4	89.20 (17)
C33—C32—H32	108.1	F2—P1—F4	85.72 (19)
C34—C32—H32	108.1	F3—P1—F4	89.54 (18)
C30—C29—C28	111.0 (3)	C1—N1—C23	124.3 (2)
C30—C29—C31	110.9 (4)	C1—N1—HN1	116 (2)
C28—C29—C31	111.7 (3)	C23—N1—HN1	119 (2)
C30—C29—HC29	108 (2)	C3—N2—C11	123.7 (2)
C28—C29—HC29	108 (2)	C3—N2—HN2	116 (2)
C31—C29—HC29	107 (2)	C11—N2—HN2	120 (2)
C22—C20—C21	108.4 (4)	C24—C23—C28	122.8 (3)
C22—C20—C12	112.5 (3)	C24—C23—N1	118.8 (3)

C21—C20—C12	112.0 (3)	C28—C23—N1	118.4 (3)
C22—C20—HC20	107 (2)	C12—C11—C16	122.9 (3)
C21—C20—HC20	104 (2)	C12—C11—N2	119.1 (3)
C12—C20—HC20	113 (2)	C16—C11—N2	117.9 (3)
C19—C17—C18	111.5 (4)	C6—C5—C10	119.2 (3)
C19—C17—C16	111.0 (4)	C6—C5—C3	121.3 (3)
C18—C17—C16	112.5 (4)	C10—C5—C3	119.5 (3)
C19—C17—HC17	112 (3)	C15—C16—C11	117.3 (3)
C18—C17—HC17	104 (2)	C15—C16—C17	120.1 (3)
C16—C17—HC17	106 (3)	C11—C16—C17	122.6 (3)
C20—C21—H21A	109.5	C11—C12—C13	117.1 (3)
C20—C21—H21B	109.5	C11—C12—C20	123.3 (3)
H21A—C21—H21B	109.5	C13—C12—C20	119.5 (3)
C20—C21—H21C	109.5	C27—C28—C23	117.5 (3)
H21A—C21—H21C	109.5	C27—C28—C29	119.4 (3)
H21B—C21—H21C	109.5	C23—C28—C29	123.2 (3)
C32—C33—H33A	109.5	C25—C24—C23	117.1 (3)
C32—C33—H33B	109.5	C25—C24—C32	120.6 (3)
H33A—C33—H33B	109.5	C23—C24—C32	122.3 (3)
C32—C33—H33C	109.5	C9—C10—C5	120.0 (3)
H33A—C33—H33C	109.5	C9—C10—H10	120.0
H33B—C33—H33C	109.5	C5—C10—H10	120.0
C29—C30—H30A	109.5	C14—C13—C12	120.9 (4)
C29—C30—H30B	109.5	C14—C13—H13	119.6
H30A—C30—H30B	109.5	C12—C13—H13	119.6
C29—C30—H30C	109.5	C26—C27—C28	120.8 (3)
H30A—C30—H30C	109.5	C26—C27—H27	119.6
H30B—C30—H30C	109.5	C28—C27—H27	119.6
C29—C31—H31A	109.5	C15—C14—C13	121.1 (3)
C29—C31—H31B	109.5	C15—C14—H14	119.5
H31A—C31—H31B	109.5	C13—C14—H14	119.5
C29—C31—H31C	109.5	C5—C6—C7	120.2 (3)
H31A—C31—H31C	109.5	C5—C6—H6	119.9
H31B—C31—H31C	109.5	C7—C6—H6	119.9
C32—C34—H34A	109.5	C26—C25—C24	121.2 (3)
C32—C34—H34B	109.5	C26—C25—H25	119.4
H34A—C34—H34B	109.5	C24—C25—H25	119.4
C32—C34—H34C	109.5	C14—C15—C16	120.7 (4)
H34A—C34—H34C	109.5	C14—C15—H15	119.7
H34B—C34—H34C	109.5	C16—C15—H15	119.7
C20—C22—H22A	109.5	C8—C7—C6	119.5 (4)
C20—C22—H22B	109.5	C8—C7—H7	120.3
H22A—C22—H22B	109.5	C6—C7—H7	120.3
C20—C22—H22C	109.5	C27—C26—C25	120.6 (3)
H22A—C22—H22C	109.5	C27—C26—H26	119.7
H22B—C22—H22C	109.5	C25—C26—H26	119.7
C17—C18—H18A	109.5	C9—C8—C7	120.9 (4)
C17—C18—H18B	109.5	C9—C8—H8	119.5
H18A—C18—H18B	109.5	C7—C8—H8	119.5

C17—C18—H18C	109.5	C8—C9—C10	120.2 (4)
H18A—C18—H18C	109.5	C8—C9—H9	119.9
H18B—C18—H18C	109.5	C10—C9—H9	119.9
N2—C3—C2—C1	161.6 (3)	C24—C23—C28—C29	179.2 (3)
C5—C3—C2—C1	−21.5 (5)	N1—C23—C28—C29	−1.0 (4)
C3—C2—C1—N1	165.6 (3)	C30—C29—C28—C27	64.9 (5)
C3—C2—C1—C4	−18.7 (5)	C31—C29—C28—C27	−59.4 (5)
C2—C1—N1—C23	−9.0 (4)	C30—C29—C28—C23	−115.2 (4)
C4—C1—N1—C23	174.9 (3)	C31—C29—C28—C23	120.5 (4)
C2—C3—N2—C11	−13.6 (5)	C28—C23—C24—C25	2.0 (4)
C5—C3—N2—C11	169.2 (3)	N1—C23—C24—C25	−177.9 (3)
C1—N1—C23—C24	95.7 (4)	C28—C23—C24—C32	−177.1 (3)
C1—N1—C23—C28	−84.2 (4)	N1—C23—C24—C32	3.0 (4)
C3—N2—C11—C12	94.3 (4)	C33—C32—C24—C25	−71.1 (4)
C3—N2—C11—C16	−85.2 (4)	C34—C32—C24—C25	52.0 (4)
N2—C3—C5—C6	−47.3 (4)	C33—C32—C24—C23	108.0 (4)
C2—C3—C5—C6	135.7 (3)	C34—C32—C24—C23	−128.9 (4)
N2—C3—C5—C10	131.5 (3)	C6—C5—C10—C9	−0.3 (5)
C2—C3—C5—C10	−45.6 (4)	C3—C5—C10—C9	−179.1 (3)
C12—C11—C16—C15	0.6 (5)	C11—C12—C13—C14	−0.8 (5)
N2—C11—C16—C15	−179.9 (3)	C20—C12—C13—C14	178.8 (3)
C12—C11—C16—C17	178.8 (3)	C23—C28—C27—C26	−1.0 (5)
N2—C11—C16—C17	−1.7 (5)	C29—C28—C27—C26	179.0 (3)
C19—C17—C16—C15	76.5 (5)	C12—C13—C14—C15	0.4 (6)
C18—C17—C16—C15	−49.3 (5)	C10—C5—C6—C7	1.3 (5)
C19—C17—C16—C11	−101.7 (4)	C3—C5—C6—C7	−179.9 (3)
C18—C17—C16—C11	132.5 (4)	C23—C24—C25—C26	−1.3 (5)
C16—C11—C12—C13	0.3 (5)	C32—C24—C25—C26	177.8 (3)
N2—C11—C12—C13	−179.2 (3)	C13—C14—C15—C16	0.6 (6)
C16—C11—C12—C20	−179.3 (3)	C11—C16—C15—C14	−1.0 (5)
N2—C11—C12—C20	1.3 (5)	C17—C16—C15—C14	−179.3 (3)
C22—C20—C12—C11	−122.7 (4)	C5—C6—C7—C8	−1.2 (5)
C21—C20—C12—C11	114.8 (4)	C28—C27—C26—C25	1.6 (5)
C22—C20—C12—C13	57.7 (5)	C24—C25—C26—C27	−0.4 (5)
C21—C20—C12—C13	−64.7 (5)	C6—C7—C8—C9	0.1 (6)
C24—C23—C28—C27	−0.9 (4)	C7—C8—C9—C10	0.9 (6)
N1—C23—C28—C27	179.0 (3)	C5—C10—C9—C8	−0.8 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—HN1···F2	0.86 (3)	2.28 (4)	3.094 (4)	156 (3)
N1—HN1···F4	0.86 (3)	2.49 (4)	3.187 (4)	139 (3)

Fig. 1

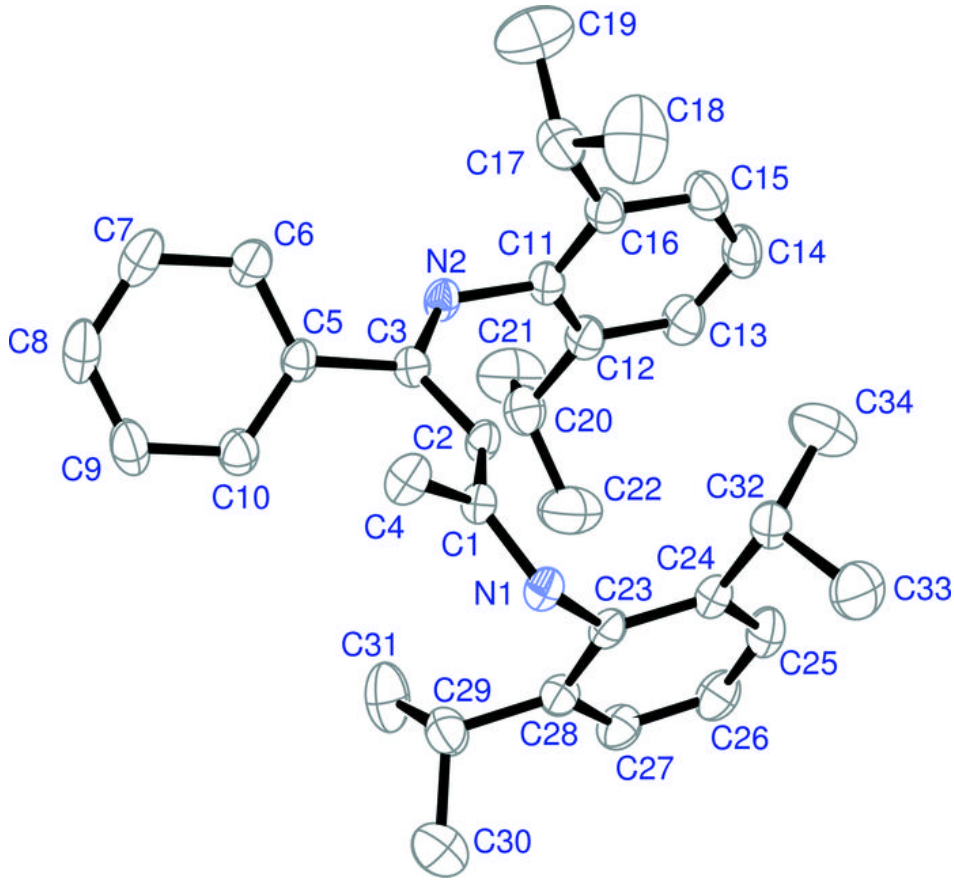
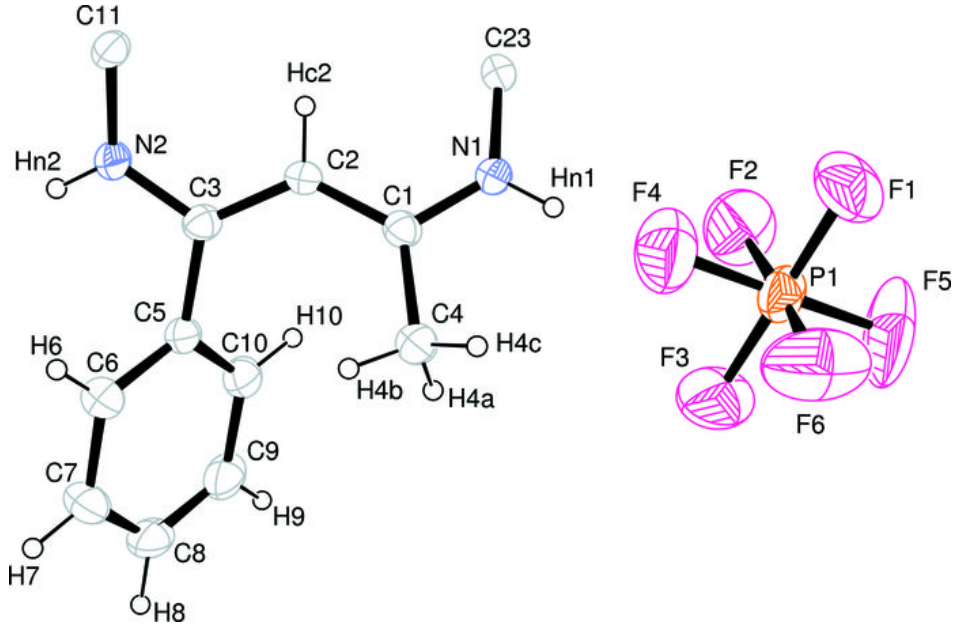

C19
C18
C17
C15
C7
C6
C11
C16
C14
N2
C8
C5
C3
C21
C13
C12
C34
C20
C2
C9
C10
C22
C32
C4
C1
C24
N1
C23
C33
C31
C25
C29
C28
C26
C27
C30

Fig. 2


C11
C23
Hc2
Hn2
N2
C2
N1
Hn1
C3
C1
F4
F2
F1
P1
F5
C5
H10
C4
H6
C6
C10
H4c
H4b
H4a
F3
F6
C9
C7
H9
C8
H7
H8