Supporting information for:
Crystallographic characterization of the geometry changes upon electron loss from 2-tert-butyl-3-aryl-2,3-diazabicyclo[2.2.2]octanes

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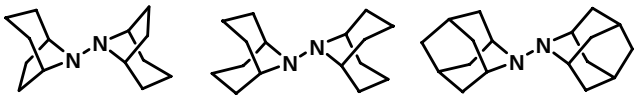
Discussion: R_4N_2 (0,+1) geometry changes characterized by x-ray crystallography

Although $\mathbf{R_4N_2}^+$ persist for hours in solution,[S1] they cannot be isolated as salts unless special alkyl groups are present that limit the rate of proton transfer between the tiny amount of the disproportionation products, $\mathbf{R_4N_2}^0$ and $\mathbf{R_4N_2}^{2+}$, formed upon intermolecular transfer between $\mathbf{R_4N_2}^+$, a process which is about 20 kcal/mol endoenergetic for many examples. This was first achieved using "Bredt's rule kinetic protection", placing the nitrogens in N,N-bicyclic systems so the α-hydrogens in the dications were at bridgehead carbons for which proton loss is kinetically inhibited.[S2] The large α-branched alkyl groups in these compounds destabilize the electronically preferred θ ~ 90° neutral conformation so much that they exist in *anti* θ = 180° conformations in both their 0 and +1 oxidation levels.[S3] Although Bredt's rule protection led to radical cation crystals that diffracted, undetected disorder led to incorrect assignment of the nitrogens as being planar, which misled us about the nature of these species for a few years. We finally realized our error when the dimeric diazabicyclooctane salt $\mathbf{1}^+PF_6^-$ was disordered in the same way as the $\mathbf{2}^+$ originally studied, and were able to characterize



S1 (32N)$_2$ **S2 (33N)$_2$** **S3 (AdN)$_2$**

the geometry change upon electron removal in three N,N'-bis-bicyclic systems (See Table 1). The most interesting thing conformationally about **1** is that it crystallizes in the diaxial conformation (each NR_2 axial to the adjacent six-membered ring) shown, despite

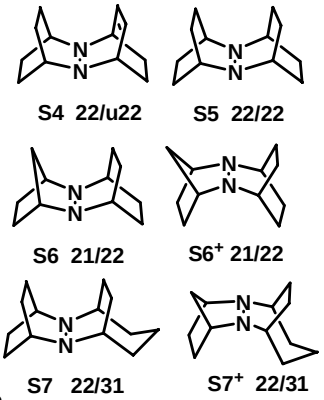
Table S1. N,N'-bis(bicyclic) hydrazines (θ_0 ~ 180°)

(**321N**)$_2$	$\mathbf{1}^0$ [a]	$\mathbf{1}^+NO_3^-H_2O$ [a]	Difference
d(NN), Å	1.469(2)	1.323(4)	-0.146 (9.9%)
$\Delta\alpha_{av}$,°	12.1(1)	4.4(2)	-7.7°(73%)
θ °	180	180	-
(**331N**)$_2$	$\mathbf{2}^{0}$ [b]	2^+OTs^- [c]	
d(NN), Å	1.505(3)	1.357(4)	-0.148 (9.8%)
$\Delta\alpha_{av}$,°	12.0(1)	3.7(3)	-8.3° (79%)
θ °	180	180	-
(**AdN**)$_2$	$\mathbf{3}^{0}$ [d]	$\mathbf{3}^+$ TsO^- [d]	
d(NN), Å	1.512(2)	1.353(5) A 1.360(6) B	-0.159(10.5%) -0.152 (10.1%)
$\Delta\alpha_{av}$,°	11.65(12)	3.4(3) A 3.3(4) B	-8.2_5(79%) -8.3_5 (80%)
θ °	180	180	-

a. Ref. S4. b. Ref. S3. c. previously unpublished. d. Ref. S5.

the fact that this is the least stable of the three θ = 180° conformations in solution.[S4] It is the most compact conformation according to calculations. The bis(azaadamantane) **3**,[S5] which has not been discussed previously shows rather similar geometrical changes to **2**.

Discovery of the proton-driven Diels-Alder reaction[S6] allowed preparation of bis(N,N'-bicyclic) (sequibicyclic) hydrazines, which have close to 0° θ values in their neutral forms, and x-ray characterization of both 0 and +1 oxidation levels of **4**-**6**.[S7,S8]



The dication $\mathbf{5}^{2+}$is uniquely stable,[S9] and not only could an x-ray structure be obtained,[S7] but its deprotonation and hydride reduction of the resulting aminoaziridinium cation allowed preparation of **7** and structural characterization of both of its oxidation levels (see Table 2).[S10] 6^+ and $\mathbf{7}^+$ are examples of several sesquibicyclic hydrazines for which

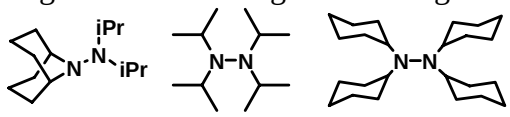
Table S2. N,N'-bis(bicyclic) hydrazines ($\theta_0 \sim 0°$)

22/u22	$\mathbf{4}^{\mathbf{0}}$ [a]	$\mathbf{4}^{+}NO_3^-$ [a]	
d(NN), Å	1.497(4)	1.349(1)	-0.148 (9.9%)
$\Delta\alpha_{av}$,°	7.8(3),8.0(3)	2.5(1), 2.2(1)	-5.6 (53%)
θ_{NN},°	0.2(4)	0.6(1)	+0.2°
22/22	$\mathbf{5}^{\mathbf{0}}$ [a]	$\mathbf{5}^{+}$ TsO^- [a]	
d(NN), Å	1.492(2)	1.339 (14)[A] 1.325(13)[B]	-0.160 (10.7%)
$\Delta\alpha_{av}$,°	6.5(1)	0.7(10),1.4(10)[A] 1.5(9),2.0(9)[B]	-5.45 (52%) -4.75(45%)
θ_N,°	15.0(2)	1.9(1.4) [A] 5.5(1.2) [B]	-13.1° -9.5°
21/22	$\mathbf{6}^{\mathbf{0}}$ [b]	$\mathbf{6}^{+}NO_3^-$ [c]	
d(NN), Å	1.514(2)	1.341(3) [A] 1.335(4) [B]	-0.173(11.4%)[A] -0.179(11.8%)[B]
$\Delta\alpha_{av}$,°	9.7(1) 10.0(1)	1.7(2),2.1(3)[A] 1.7(3),1.4(3)[B]	7.9_5(76%)[A] 8.3(79%[B]
θ_N,°	1.0(2)	2.0(3) [A],-1.2(4)[B]	(-1, -0.2)
22/13	$\mathbf{7}$ [d]	$\mathbf{7}^{+}$ TsO^- [d]	
d(NN), Å	1.500(3)	1.328(3)	-0.172 (11.5%)
$\Delta\alpha_{av}$,°	9.3 (2) (N_1) 9.7 (2) (N_2)	0.9 (2) (both)	-8.6 (82%)
θ_{NN},°	4.3 (2)	0.7 (3)	-3.6

a. Ref. S7. b. Ref. S8. c. Ref. S5. d. Ref. S10.

the neutral form and radical cation exist in different double nitrogen inversion forms. The flatter nitrogens of the radical cation decrease 1,3 interactions and increase 1,2 interactions (bridgehead hydrogen NR_2 interaction is the important interaction) enough that which ring reversal form is most stable changes upon electron removal. Because the two ring reversal forms of the radical cation have different nitrogen pyramidalities, properties like the optical absorption spectra, which are especially sensitive to $\Delta\alpha_{av}$ are rather different for the two double nitrogen inversion forms.

Chen's discovery[S11] that Bredt's rule protection is not necessary to allow radical cation isolation because tetra-α-branching suffices allows obtaining crystal structures for which the θ values of 0 and +1 oxidation levels are substantially different.[S12] Compounds S**8**-S**10** have been studied. These systems have significant flattening at the nitrogens



S8 33NNiPr$_2$ **S9 iPr$_2$NNiPr$_2$** **S10 cHx$_2$NNcHx$_2$**

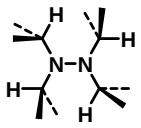
of the neutral oxidation level, leading to considerably smaller changes in both d(NN) and in $\Delta\alpha_{av}$ than for the $\theta_0 \sim 0$ or ~180 systems (see Table 3). The unlinked α-branched alkyl

Table S3. Neutral $\theta_0 \sim 90°$ systems

(**33N**)**NiPr$_2$**	**S8(0)** [a]	**S8(+)** NO_3^- [a]	difference
d(NN), Å	1.420(2)	1.346(3)	-0.074(-5.2%
$\Delta\alpha_{av}$,°	4.7 (33N) 2.3 ($NiPr_2$)	3.1 (33N) 1.2 ($NiPr_2$)	-1.6° (15%) -1.1° (10%)
ϕ_{NN},°	85.9	22.7	-63.2°
iPr$_2$NNiPr$_2$	**S9(0)** [b]	**S9(+)** $TsO^-\cdot CH_3CN$	
d(NN), Å	1.394(4) [major] 1.399(5) [minor]	1.333(4)	-0.061 (-4.4%
$\Delta\alpha_{av}$,°	4.2 (3) [major] 4.2 (5) [minor]	0.1;0.4	-3.9$_5$ (38%)
ϕ_{NN},°	88.5 (5) [major] 85.6 (8) [minor]	7.9	-80.6°[major 78.6 [minor]
cHx$_2$NNcHx$_2$	**S10(0)** [b]	**S10(+)** [c] $B[C_6H_3(CF_3)_2]_4^-$	
d(NN), Å	1.389(7)	1.333(9)	-0.056 (4.0%
$\Delta\alpha_{av}$,°	3.3(5) [N_1] 3.4(5) [N_2]	0.0(3)	3.3 (31%) 3.4 (32%)
ϕ_{NN},°	89.4(7)	0.1(6)	-89.3°

a. Ref. S11. b. Ref. S12. c. Ref. S5.

groups adopt conformations been studied. Despite θ values near 90°, that put one HCNN twist angle near 0° and the other at the same nitrogen near 180°, to minimize non-bonded steric interactions. For **S9**$^+$ each $C_\alpha H$ bond is tucked between the substitutents of the adjacent alkyl groups, as indicated diagramatically below, which makes **S9**$^+$ nearly isostructural with tetraisopropylethylene.[S13] Although the nearly colorless tetrakis-

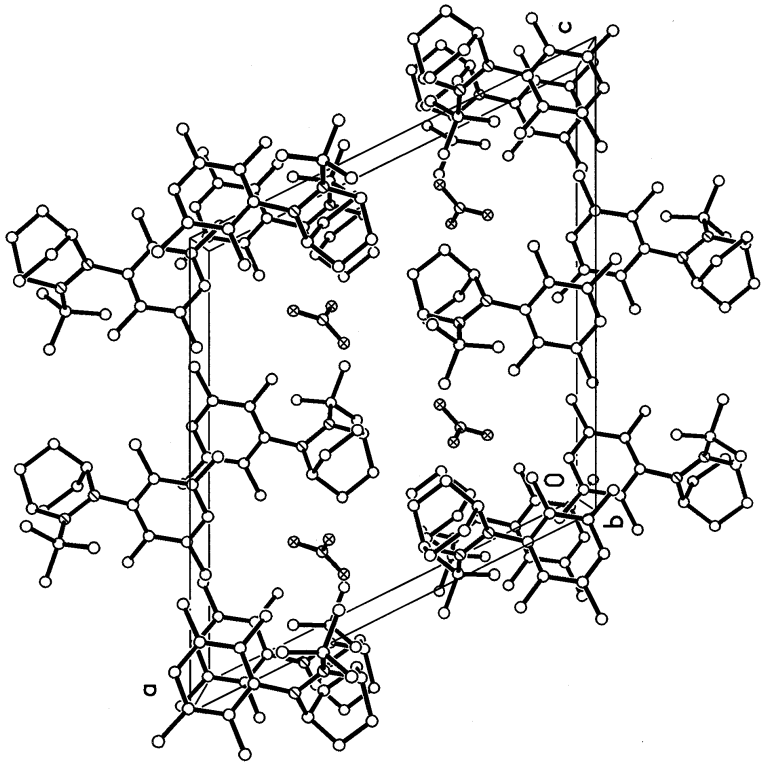


S9^{+}

(m,m'-bis(trifluoromethyl)phenyl)borate salt of **S10**$^{+}$ is in the same conformation, its dark red TsO^{-}, and NO_3^{-} and SbF_6^{-}salts are in a twisted conformation because the alkyl groups are in a different conformation than **S9**$^{+}$.[S5]

Crystallographic Experimental Section

HyDU^{+}NO$_3^{-}$ (97264)



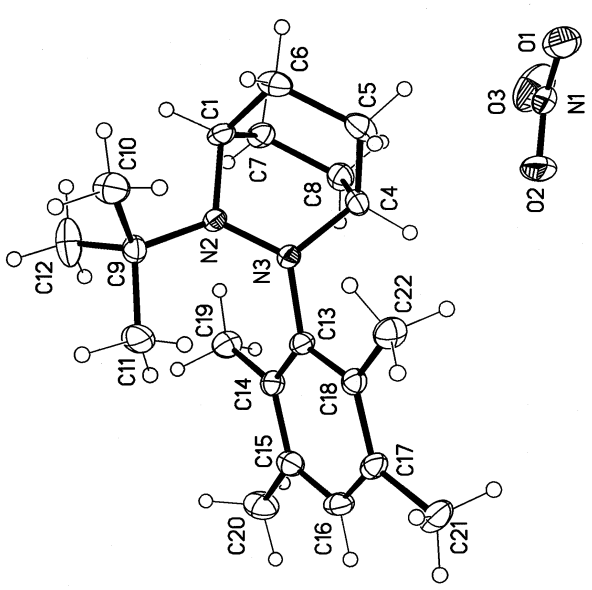


Table 1. Crystal data and structure refinement for 97264.

Identification code	97264	
Empirical formula	C20 H32 N3 O3	
Formula weight	362.49	
Temperature	133(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 15.9105(4) Å	α= 90°.
	b = 8.0279(2) Å	β= 116.514(2)°.
	c = 16.6895(5) Å	γ = 90°.
Volume	1907.51(9) $Å^3$	
Z	4	
Density (calculated)	1.262 Mg/m^3	
Absorption coefficient	0.085 mm^{-1}	
F(000)	788	
Crystal size	0.48 x 0.41 x 0.29 mm^3	
Theta range for data collection	2.45 to 29.06°.	
Index ranges	-20<=h<=21, -6<=k<=10, -12<=l<=21	
Reflections collected	8955	
Independent reflections	4393 [R(int) = 0.0164]	
Completeness to theta = 29.06°	86.1 %	
Absorption correction	Multi-scan with SADABS	
Max. and min. transmission	0.9757 and 0.9603	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4393 / 0 / 235	
Goodness-of-fit on F^2	1.065	
Final R indices [I>2sigma(I)]	R1 = 0.0383, wR2 = 0.1016	
R indices (all data)	R1 = 0.0469, wR2 = 0.1063	
Largest diff. peak and hole	0.297 and -0.229 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 97264. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)

N(1)	7609(1)	6453(1)	5598(1)	15(1)
N(2)	8315(1)	6936(1)	5407(1)	15(1)
C(1)	9220(1)	6105(1)	5989(1)	18(1)
C(2)	9484(1)	6482(2)	6977(1)	24(1)
C(3)	8673(1)	5910(2)	7177(1)	25(1)
C(4)	7896(1)	5176(1)	6320(1)	19(1)
C(5)	9090(1)	4219(1)	5833(1)	20(1)
C(6)	8263(1)	3649(1)	6019(1)	21(1)
C(7)	6618(1)	6552(1)	4978(1)	16(1)
C(8)	6263(1)	5843(1)	4119(1)	18(1)
C(9)	5286(1)	5951(1)	3575(1)	21(1)
C(10)	4716(1)	6693(1)	3910(1)	23(1)
C(11)	5072(1)	7329(1)	4777(1)	20(1)
C(12)	6045(1)	7292(1)	5324(1)	17(1)
C(13)	6860(1)	4952(2)	3760(1)	23(1)
C(14)	4846(1)	5253(2)	2636(1)	31(1)
C(15)	4401(1)	8041(2)	5108(1)	28(1)
C(16)	6458(1)	8047(2)	6253(1)	24(1)
C(17)	8337(1)	8544(1)	4938(1)	18(1)
C(18)	8704(1)	8108(2)	4257(1)	37(1)
C(19)	7389(1)	9410(2)	4477(1)	34(1)
C(20)	9024(1)	9738(1)	5641(1)	27(1)
N(3)	1787(1)	8127(2)	1950(1)	29(1)
O(1)	1246(1)	7504(1)	1209(1)	38(1)
O(2)	2516(1)	7368(2)	2445(1)	67(1)
O(3)	1585(1)	9487(2)	2177(1)	58(1)

Table 3. Bond lengths [Å] and angles [°] for 97264.

N(1)-N(2)	1.3548(12)	C(10)-C(11)	1.3935(17)
N(1)-C(7)	1.4503(13)	C(10)-H(10)	0.9500
N(1)-C(4)	1.4901(13)	C(11)-C(12)	1.4029(15)
N(2)-C(1)	1.4886(13)	C(11)-C(15)	1.5157(16)
N(2)-C(17)	1.5184(13)	C(12)-C(16)	1.5147(15)
C(1)-C(5)	1.5349(15)	C(13)-H(13A)	0.9819
C(1)-C(2)	1.5401(15)	C(13)-H(13B)	0.9806
C(1)-H(1)	1.0000	C(13)-H(13C)	0.9816
C(2)-C(3)	1.5399(17)	C(14)-H(14A)	0.9819
C(2)-H(2A)	0.9900	C(14)-H(14B)	0.9815
C(2)-H(2B)	0.9900	C(14)-H(14C)	0.9805
C(3)-C(4)	1.5304(16)	C(15)-H(15A)	0.9815
C(3)-H(3A)	0.9900	C(15)-H(15B)	0.9806
C(3)-H(3B)	0.9900	C(15)-H(15C)	0.9817
C(4)-C(6)	1.5359(15)	C(16)-H(16A)	0.9816
C(4)-H(4)	1.0000	C(16)-H(16B)	0.9814
C(5)-C(6)	1.5490(15)	C(16)-H(16C)	0.9811
C(5)-H(5A)	0.9900	C(17)-C(19)	1.5213(16)
C(5)-H(5B)	0.9900	C(17)-C(18)	1.5315(16)
C(6)-H(6A)	0.9900	C(17)-C(20)	1.5316(15)
C(6)-H(6B)	0.9900	C(18)-H(18A)	0.9800
C(7)-C(8)	1.4060(15)	C(18)-H(18B)	0.9800
C(7)-C(12)	1.4104(14)	C(18)-H(18C)	0.9800
C(8)-C(9)	1.4098(15)	C(19)-H(19A)	0.9800
C(8)-C(13)	1.5099(15)	C(19)-H(19B)	0.9800
C(9)-C(10)	1.3937(17)	C(19)-H(19C)	0.9799
C(9)-C(14)	1.5112(16)	C(20)-H(20A)	0.9800

C(20)-H(20B)	0.9800	N(3)-O(3)	1.2434(17)
C(20)-H(20C)	0.9800	N(3)-O(1)	1.2525(15)
N(3)-O(2)	1.2431(15)		

N(2)-N(1)-C(7)	124.53(8)	C(11)-C(10)-H(10)	118.8
N(2)-N(1)-C(4)	113.63(8)	C(9)-C(10)-H(10)	118.8
C(7)-N(1)-C(4)	117.26(8)	C(10)-C(11)-C(12)	119.15(10)
N(1)-N(2)-C(1)	112.38(8)	C(10)-C(11)-C(15)	119.36(10)
N(1)-N(2)-C(17)	125.34(8)	C(12)-C(11)-C(15)	121.49(11)
C(1)-N(2)-C(17)	118.59(8)	C(11)-C(12)-C(7)	118.02(10)
N(2)-C(1)-C(5)	108.25(8)	C(11)-C(12)-C(16)	120.41(10)
N(2)-C(1)-C(2)	109.41(9)	C(7)-C(12)-C(16)	121.56(10)
C(5)-C(1)-C(2)	109.06(9)	C(8)-C(13)-H(13A)	109.6
N(2)-C(1)-H(1)	110.0	C(8)-C(13)-H(13B)	109.6
C(5)-C(1)-H(1)	110.0	H(13A)-C(13)-H(13B)	109.4
C(2)-C(1)-H(1)	110.0	C(8)-C(13)-H(13C)	109.2
C(3)-C(2)-C(1)	108.62(9)	H(13A)-C(13)-H(13C)	109.5
C(3)-C(2)-H(2A)	110.0	H(13B)-C(13)-H(13C)	109.5
C(1)-C(2)-H(2A)	110.0	C(9)-C(14)-H(14A)	109.5
C(3)-C(2)-H(2B)	110.0	C(9)-C(14)-H(14B)	109.5
C(1)-C(2)-H(2B)	110.0	H(14A)-C(14)-H(14B)	109.5
H(2A)-C(2)-H(2B)	108.3	C(9)-C(14)-H(14C)	109.4
C(4)-C(3)-C(2)	108.58(9)	H(14A)-C(14)-H(14C)	109.4
C(4)-C(3)-H(3A)	110.0	H(14B)-C(14)-H(14C)	109.5
C(2)-C(3)-H(3A)	110.0	C(11)-C(15)-H(15A)	109.3
C(4)-C(3)-H(3B)	110.0	C(11)-C(15)-H(15B)	109.4
C(2)-C(3)-H(3B)	110.0	H(15A)-C(15)-H(15B)	109.5
H(3A)-C(3)-H(3B)	108.4	C(11)-C(15)-H(15C)	109.7
N(1)-C(4)-C(3)	108.57(9)	H(15A)-C(15)-H(15C)	109.5
N(1)-C(4)-C(6)	107.96(9)	H(15B)-C(15)-H(15C)	109.4
C(3)-C(4)-C(6)	110.28(9)	C(12)-C(16)-H(16A)	109.5
N(1)-C(4)-H(4)	110.0	C(12)-C(16)-H(16B)	109.4
C(3)-C(4)-H(4)	110.0	H(16A)-C(16)-H(16B)	109.5
C(6)-C(4)-H(4)	110.0	C(12)-C(16)-H(16C)	109.6
C(1)-C(5)-C(6)	108.71(9)	H(16A)-C(16)-H(16C)	109.5
C(1)-C(5)-H(5A)	109.9	H(16B)-C(16)-H(16C)	109.4
C(6)-C(5)-H(5A)	109.9	N(2)-C(17)-C(19)	113.73(9)
C(1)-C(5)-H(5B)	109.9	N(2)-C(17)-C(18)	107.04(9)
C(6)-C(5)-H(5B)	109.9	C(19)-C(17)-C(18)	110.46(11)
H(5A)-C(5)-H(5B)	108.3	N(2)-C(17)-C(20)	108.06(9)
C(4)-C(6)-C(5)	108.11(9)	C(19)-C(17)-C(20)	107.97(10)
C(4)-C(6)-H(6A)	110.1	C(18)-C(17)-C(20)	109.52(10)
C(5)-C(6)-H(6A)	110.1	C(17)-C(18)-H(18A)	109.5
C(4)-C(6)-H(6B)	110.1	C(17)-C(18)-H(18B)	109.5
C(5)-C(6)-H(6B)	110.1	H(18A)-C(18)-H(18B)	109.5
H(6A)-C(6)-H(6B)	108.4	C(17)-C(18)-H(18C)	109.5
C(8)-C(7)-C(12)	123.32(10)	H(18A)-C(18)-H(18C)	109.5
C(8)-C(7)-N(1)	121.29(9)	H(18B)-C(18)-H(18C)	109.5
C(12)-C(7)-N(1)	115.24(9)	C(17)-C(19)-H(19A)	109.5
C(7)-C(8)-C(9)	117.18(10)	C(17)-C(19)-H(19B)	109.5
C(7)-C(8)-C(13)	123.98(10)	H(19A)-C(19)-H(19B)	109.5
C(9)-C(8)-C(13)	118.83(10)	C(17)-C(19)-H(19C)	109.5
C(10)-C(9)-C(8)	119.77(10)	H(19A)-C(19)-H(19C)	109.5
C(10)-C(9)-C(14)	119.40(10)	H(19B)-C(19)-H(19C)	109.5
C(8)-C(9)-C(14)	120.82(11)	C(17)-C(20)-H(20A)	109.5
C(11)-C(10)-C(9)	122.48(10)	C(17)-C(20)-H(20B)	109.5

H(20A)-C(20)-H(20B)	109.5
C(17)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
O(2)-N(3)-O(3)	121.25(14)
O(2)-N(3)-O(1)	119.11(14)
O(3)-N(3)-O(1)	119.64(12)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 97264. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	14(1)	17(1)	14(1)	1(1)	7(1)	1(1)
N(2)	13(1)	16(1)	15(1)	0(1)	6(1)	0(1)
C(1)	13(1)	20(1)	19(1)	1(1)	5(1)	2(1)
C(2)	22(1)	25(1)	17(1)	-1(1)	1(1)	1(1)
C(3)	28(1)	30(1)	13(1)	2(1)	7(1)	6(1)
C(4)	20(1)	21(1)	18(1)	6(1)	10(1)	4(1)
C(5)	19(1)	18(1)	22(1)	0(1)	9(1)	4(1)
C(6)	23(1)	18(1)	24(1)	3(1)	11(1)	1(1)
C(7)	13(1)	15(1)	17(1)	1(1)	6(1)	0(1)
C(8)	19(1)	16(1)	18(1)	0(1)	8(1)	-1(1)
C(9)	20(1)	18(1)	20(1)	1(1)	4(1)	-3(1)
C(10)	14(1)	20(1)	29(1)	4(1)	4(1)	-1(1)
C(11)	17(1)	16(1)	30(1)	4(1)	12(1)	2(1)
C(12)	18(1)	15(1)	21(1)	1(1)	10(1)	1(1)
C(13)	23(1)	26(1)	19(1)	-6(1)	9(1)	-1(1)
C(14)	27(1)	34(1)	22(1)	-3(1)	1(1)	-4(1)
C(15)	21(1)	24(1)	45(1)	3(1)	20(1)	3(1)
C(16)	25(1)	26(1)	23(1)	-3(1)	13(1)	4(1)
C(17)	20(1)	18(1)	18(1)	2(1)	9(1)	-3(1)
C(18)	65(1)	29(1)	36(1)	-3(1)	39(1)	-9(1)
C(19)	22(1)	26(1)	43(1)	19(1)	5(1)	0(1)
C(20)	26(1)	18(1)	29(1)	-1(1)	5(1)	-4(1)
N(3)	19(1)	42(1)	27(1)	12(1)	11(1)	-4(1)
O(1)	28(1)	50(1)	34(1)	-3(1)	11(1)	1(1)
O(2)	22(1)	105(1)	59(1)	57(1)	6(1)	6(1)
O(3)	53(1)	57(1)	76(1)	-26(1)	39(1)	-20(1)

Table 5. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^{\ 3}$) for 97264.

	x	y	z	U(eq)
H(1)	9726	6511	5837	22
H(2A)	10068	5885	7371	28
H(2B)	9593	7692	7093	28
H(3A)	8900	5060	7657	29
H(3B)	8426	6868	7381	29
H(4)	7345	4855	6425	23
H(5A)	9672	3629	6239	23
H(5B)	8953	3952	5208	23
H(6A)	7756	3167	5470	26
H(6B)	8479	2787	6493	26
H(10)	4060	6767	3534	27
H(13A)	6823	5537	3229	35
H(13B)	6634	3806	3598	35
H(13C)	7515	4934	4223	35
H(14A)	5151	5742	2294	47

H(14B)	4173	5522	2342	47
H(14C)	4926	4040	2661	47
H(15A)	3758	8002	4622	42
H(15B)	4574	9199	5295	42
H(15C)	4436	7385	5618	42
H(16A)	7098	8437	6416	36
H(16B)	6474	7202	6685	36
H(16C)	6069	8989	6262	36
H(18A)	9328	7601	4568	56
H(18B)	8746	9124	3951	56
H(18C)	8273	7321	3816	56
H(19A)	7150	9632	4915	51
H(19B)	6946	8691	4001	51
H(19C)	7461	10463	4217	51
H(20A)	9652	9238	5918	40
H(20B)	8814	9950	6101	40
H(20C)	9046	10791	5353	40

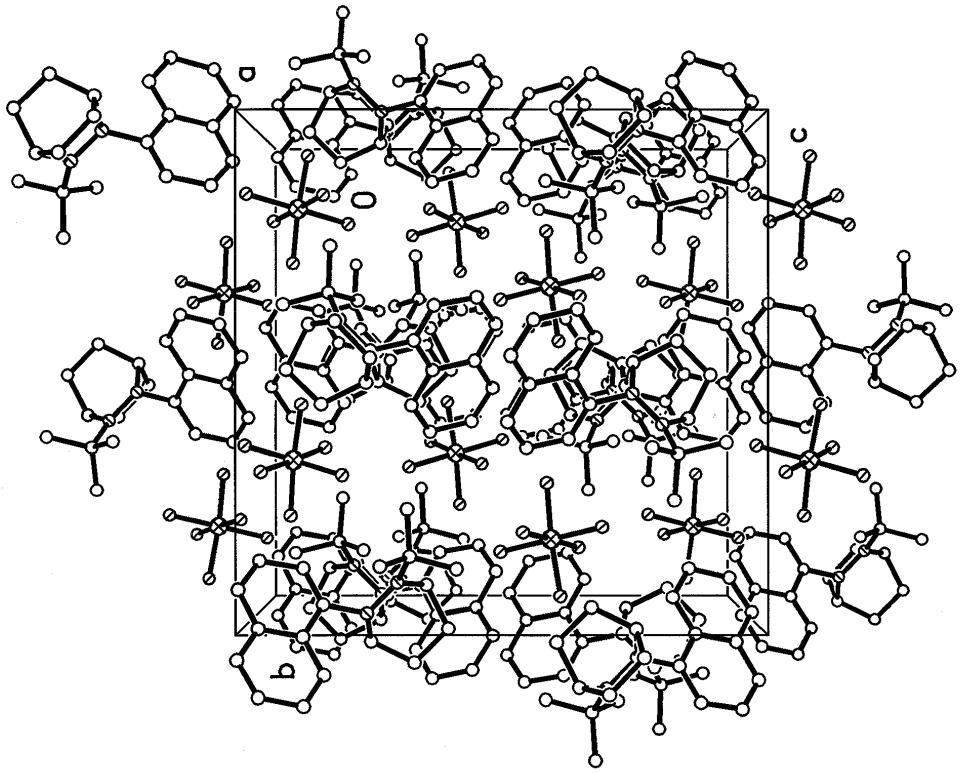
Table 6. Torsion angles [°] for 97264.

C(7)-N(1)-N(2)-C(1)	-157.00(9)
C(4)-N(1)-N(2)-C(1)	-1.79(11)
C(7)-N(1)-N(2)-C(17)	45.14(14)
C(4)-N(1)-N(2)-C(17)	-159.64(9)
N(1)-N(2)-C(1)-C(5)	60.96(11)
C(17)-N(2)-C(1)-C(5)	-139.53(9)
N(1)-N(2)-C(1)-C(2)	-57.77(11)
C(17)-N(2)-C(1)-C(2)	101.73(10)
N(2)-C(1)-C(2)-C(3)	56.59(12)
C(5)-C(1)-C(2)-C(3)	-61.64(11)
C(1)-C(2)-C(3)-C(4)	-0.10(13)
N(2)-N(1)-C(4)-C(3)	60.56(11)
C(7)-N(1)-C(4)-C(3)	-142.30(9)
N(2)-N(1)-C(4)-C(6)	-59.00(11)
C(7)-N(1)-C(4)-C(6)	98.14(10)
C(2)-C(3)-C(4)-N(1)	-56.38(12)
C(2)-C(3)-C(4)-C(6)	61.72(12)
N(2)-C(1)-C(5)-C(6)	-56.33(11)
C(2)-C(1)-C(5)-C(6)	62.64(11)
N(1)-C(4)-C(6)-C(5)	57.96(11)
C(3)-C(4)-C(6)-C(5)	-60.51(12)
C(1)-C(5)-C(6)-C(4)	-1.77(12)
N(2)-N(1)-C(7)-C(8)	53.23(14)
C(4)-N(1)-C(7)-C(8)	-101.18(11)
N(2)-N(1)-C(7)-C(12)	-131.14(10)
C(4)-N(1)-C(7)-C(12)	74.45(12)
C(12)-C(7)-C(8)-C(9)	2.52(16)
N(1)-C(7)-C(8)-C(9)	177.79(9)
C(12)-C(7)-C(8)-C(13)	-176.03(10)
N(1)-C(7)-C(8)-C(13)	-0.76(16)
C(7)-C(8)-C(9)-C(10)	-1.93(16)
C(13)-C(8)-C(9)-C(10)	176.70(10)
C(7)-C(8)-C(9)-C(14)	178.82(10)
C(13)-C(8)-C(9)-C(14)	-2.55(16)
C(8)-C(9)-C(10)-C(11)	-0.69(17)
C(14)-C(9)-C(10)-C(11)	178.57(11)

C(9)-C(10)-C(11)-C(12)	2.84(17)
C(9)-C(10)-C(11)-C(15)	-177.03(10)
C(10)-C(11)-C(12)-C(7)	-2.23(15)
C(15)-C(11)-C(12)-C(7)	177.65(10)
C(10)-C(11)-C(12)-C(16)	177.12(10)
C(15)-C(11)-C(12)-C(16)	-3.01(16)
C(8)-C(7)-C(12)-C(11)	-0.44(16)
N(1)-C(7)-C(12)-C(11)	-175.97(9)
C(8)-C(7)-C(12)-C(16)	-179.78(10)
N(1)-C(7)-C(12)-C(16)	4.69(14)
N(1)-N(2)-C(17)-C(19)	-14.76(15)
C(1)-N(2)-C(17)-C(19)	-171.37(10)
N(1)-N(2)-C(17)-C(18)	-137.04(11)
C(1)-N(2)-C(17)-C(18)	66.35(12)
N(1)-N(2)-C(17)-C(20)	105.09(11)
C(1)-N(2)-C(17)-C(20)	-51.52(12)

Symmetry transformations used to generate equivalent atoms:

Hy[1]**NA**$^{+}$**SbF**$_6^{-}$ (98001)



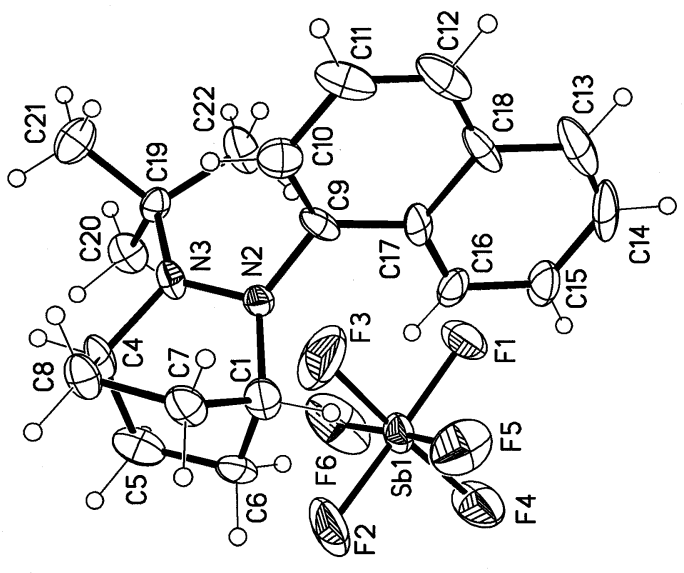


Table 1. Crystal data and structure refinement for 98001.

Identification code	98001	
Empirical formula	C20 H26 F6 N2 Sb	
Formula weight	530.18	
Temperature	133(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 15.8173(18) Å	α= 90°.
	b = 16.3724(16) Å	β= 90°.
	c = 16.4957(17) Å	γ = 90°.
Volume	4271.8(8) $Å^3$	
Z	8	
Density (calculated)	1.649 Mg/m^3	
Absorption coefficient	1.351 mm^{-1}	
F(000)	2120	
Crystal size	0.42 x 0.14 x 0.01 mm^3	
Theta range for data collection	2.78 to 25.00°.	
Index ranges	-15<=h<=18, -19<=k<=11, -19<=l<=13	
Reflections collected	15951	
Independent reflections	3763 [R(int) = 0.0814]	
Completeness to theta = 25.00°	99.9 %	
Absorption correction	Multi-scan with SADABS	
Max. and min. transmission	0.9866 and 0.6007	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3763 / 0 / 262	
Goodness-of-fit on F^2	1.020	
Final R indices [I>2sigma(I)]	R1 = 0.0617, wR2 = 0.1287	
R indices (all data)	R1 = 0.1036, wR2 = 0.1441	
Largest diff. peak and hole	1.160 and -1.333 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 98001. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Sb(1)	6854(1)	3277(1)	5962(1)	35(1)
F(1)	7854(3)	3500(3)	6520(3)	54(2)
F(2)	5845(4)	3083(4)	5409(4)	93(3)
F(3)	6529(5)	4368(4)	6137(5)	92(2)
F(4)	7144(4)	2179(4)	5884(4)	76(2)
F(5)	6313(4)	3001(4)	6933(3)	72(2)
F(6)	7330(4)	3554(5)	4981(3)	96(3)
C(1)	4156(4)	4810(5)	7491(5)	31(2)
N(2)	4857(4)	5405(3)	7539(3)	21(1)
N(3)	4810(4)	5992(4)	6966(3)	23(1)
C(4)	4068(5)	5895(5)	6433(4)	28(2)
C(5)	4103(5)	5050(5)	6018(5)	37(2)
C(6)	4187(5)	4403(5)	6663(5)	32(2)
C(7)	3318(5)	5263(5)	7598(5)	31(2)
C(8)	3270(4)	5941(5)	6964(4)	30(2)
C(9)	5181(5)	5601(5)	8346(4)	24(2)
C(10)	4879(5)	6270(5)	8734(5)	32(2)
C(11)	5188(5)	6430(6)	9532(5)	41(2)
C(12)	5756(6)	5929(6)	9886(5)	46(2)
C(13)	6670(5)	4694(7)	9828(5)	51(3)
C(14)	6967(6)	4037(7)	9419(6)	56(3)
C(15)	6668(5)	3840(5)	8619(6)	41(2)
C(16)	6087(5)	4358(5)	8274(5)	33(2)
C(17)	5768(4)	5040(5)	8686(4)	26(2)
C(18)	6068(5)	5222(6)	9487(4)	34(2)
C(19)	5544(4)	6530(4)	6712(4)	21(2)
C(20)	5774(5)	6311(5)	5838(4)	32(2)
C(21)	5243(5)	7415(5)	6767(5)	37(2)
C(22)	6322(5)	6395(5)	7234(5)	35(2)

Table 3. Bond lengths [Å] and angles [°] for 98001.

Sb(1)-F(6)	1.841(5)	C(10)-C(11)	1.428(11)
Sb(1)-F(4)	1.859(6)	C(10)-H(10)	0.9500
Sb(1)-F(1)	1.866(5)	C(11)-C(12)	1.349(12)
Sb(1)-F(2)	1.866(5)	C(11)-H(11)	0.9500
Sb(1)-F(5)	1.872(5)	C(12)-C(18)	1.420(13)
Sb(1)-F(3)	1.881(6)	C(12)-H(12)	0.9500
C(1)-N(2)	1.479(9)	C(13)-C(14)	1.354(14)
C(1)-C(6)	1.522(11)	C(13)-C(18)	1.403(12)
C(1)-C(7)	1.529(10)	C(13)-H(13)	0.9500
C(1)-H(1)	1.0000	C(14)-C(15)	1.437(14)
N(2)-N(3)	1.349(8)	C(14)-H(14)	0.9500
N(2)-C(9)	1.462(8)	C(15)-C(16)	1.374(10)
N(3)-C(4)	1.474(9)	C(15)-H(15)	0.9500
N(3)-C(19)	1.517(9)	C(16)-C(17)	1.401(11)
C(4)-C(8)	1.538(10)	C(16)-H(16)	0.9500
C(4)-C(5)	1.544(11)	C(17)-C(18)	1.436(10)
C(4)-H(4)	1.0000	C(19)-C(22)	1.517(10)
C(5)-C(6)	1.507(11)	C(19)-C(21)	1.528(10)
C(5)-H(5A)	0.9900	C(19)-C(20)	1.529(10)
C(5)-H(5B)	0.9900	C(20)-H(20A)	0.9799
C(6)-H(6A)	0.9900	C(20)-H(20B)	0.9800
C(6)-H(6B)	0.9900	C(20)-H(20C)	0.9801
C(7)-C(8)	1.527(10)	C(21)-H(21A)	0.9800
C(7)-H(7A)	0.9900	C(21)-H(21B)	0.9800
C(7)-H(7B)	0.9900	C(21)-H(21C)	0.9800
C(8)-H(8A)	0.9900	C(22)-H(22A)	0.9801
C(8)-H(8B)	0.9900	C(22)-H(22B)	0.9802
C(9)-C(10)	1.356(11)	C(22)-H(22C)	0.9799
C(9)-C(17)	1.421(10)		

F(6)-Sb(1)-F(4)	94.4(3)	N(2)-N(3)-C(4)	112.7(6)
F(6)-Sb(1)-F(1)	92.2(3)	N(2)-N(3)-C(19)	124.3(5)
F(4)-Sb(1)-F(1)	90.8(2)	C(4)-N(3)-C(19)	120.5(5)
F(6)-Sb(1)-F(2)	87.8(3)	N(3)-C(4)-C(8)	107.9(5)
F(4)-Sb(1)-F(2)	90.7(3)	N(3)-C(4)-C(5)	109.5(6)
F(1)-Sb(1)-F(2)	178.4(3)	C(8)-C(4)-C(5)	109.0(6)
F(6)-Sb(1)-F(5)	176.9(3)	N(3)-C(4)-H(4)	110.1
F(4)-Sb(1)-F(5)	86.5(3)	C(8)-C(4)-H(4)	110.1
F(1)-Sb(1)-F(5)	90.7(3)	C(5)-C(4)-H(4)	110.1
F(2)-Sb(1)-F(5)	89.2(3)	C(6)-C(5)-C(4)	108.7(6)
F(6)-Sb(1)-F(3)	90.7(4)	C(6)-C(5)-H(5A)	110.0
F(4)-Sb(1)-F(3)	174.8(3)	C(4)-C(5)-H(5A)	110.0
F(1)-Sb(1)-F(3)	88.3(3)	C(6)-C(5)-H(5B)	110.0
F(2)-Sb(1)-F(3)	90.2(3)	C(4)-C(5)-H(5B)	110.0
F(5)-Sb(1)-F(3)	88.5(3)	H(5A)-C(5)-H(5B)	108.3
N(2)-C(1)-C(6)	108.2(6)	C(5)-C(6)-C(1)	108.8(6)
N(2)-C(1)-C(7)	108.9(6)	C(5)-C(6)-H(6A)	109.9
C(6)-C(1)-C(7)	110.1(6)	C(1)-C(6)-H(6A)	109.9
N(2)-C(1)-H(1)	109.9	C(5)-C(6)-H(6B)	109.9
C(6)-C(1)-H(1)	109.9	C(1)-C(6)-H(6B)	109.9
C(7)-C(1)-H(1)	109.9	H(6A)-C(6)-H(6B)	108.3
N(3)-N(2)-C(9)	120.1(6)	C(8)-C(7)-C(1)	108.4(6)
N(3)-N(2)-C(1)	113.0(6)	C(8)-C(7)-H(7A)	110.0
C(9)-N(2)-C(1)	117.1(6)	C(1)-C(7)-H(7A)	110.0

C(8)-C(7)-H(7B)	110.0
C(1)-C(7)-H(7B)	110.0
H(7A)-C(7)-H(7B)	108.4
C(7)-C(8)-C(4)	108.3(6)
C(7)-C(8)-H(8A)	110.0
C(4)-C(8)-H(8A)	110.0
C(7)-C(8)-H(8B)	110.0
C(4)-C(8)-H(8B)	110.0
H(8A)-C(8)-H(8B)	108.4
C(10)-C(9)-C(17)	124.5(7)
C(10)-C(9)-N(2)	118.9(7)
C(17)-C(9)-N(2)	116.5(6)
C(9)-C(10)-C(11)	117.6(8)
C(9)-C(10)-H(10)	121.2
C(11)-C(10)-H(10)	121.2
C(12)-C(11)-C(10)	121.0(8)
C(12)-C(11)-H(11)	119.5
C(10)-C(11)-H(11)	119.5
C(11)-C(12)-C(18)	121.8(7)
C(11)-C(12)-H(12)	119.1
C(18)-C(12)-H(12)	119.1
C(14)-C(13)-C(18)	121.6(8)
C(14)-C(13)-H(13)	119.2
C(18)-C(13)-H(13)	119.2
C(13)-C(14)-C(15)	121.5(8)
C(13)-C(14)-H(14)	119.3
C(15)-C(14)-H(14)	119.3
C(16)-C(15)-C(14)	117.4(9)
C(16)-C(15)-H(15)	121.3
C(14)-C(15)-H(15)	121.3
C(15)-C(16)-C(17)	122.2(8)
C(15)-C(16)-H(16)	118.9
C(17)-C(16)-H(16)	118.9
C(16)-C(17)-C(9)	124.0(7)
C(16)-C(17)-C(18)	119.5(7)
C(9)-C(17)-C(18)	116.4(7)
C(13)-C(18)-C(12)	123.6(8)
C(13)-C(18)-C(17)	117.7(9)
C(12)-C(18)-C(17)	118.7(7)
C(22)-C(19)-N(3)	112.3(5)
C(22)-C(19)-C(21)	111.0(6)
N(3)-C(19)-C(21)	107.2(6)
C(22)-C(19)-C(20)	108.0(6)
N(3)-C(19)-C(20)	107.8(6)
C(21)-C(19)-C(20)	110.6(6)
C(19)-C(20)-H(20A)	109.5
C(19)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5
C(19)-C(20)-H(20C)	109.4
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
C(19)-C(21)-H(21A)	109.7
C(19)-C(21)-H(21B)	109.4
H(21A)-C(21)-H(21B)	109.5
C(19)-C(21)-H(21C)	109.3
H(21A)-C(21)-H(21C)	109.5
H(21B)-C(21)-H(21C)	109.5
C(19)-C(22)-H(22A)	109.7
C(19)-C(22)-H(22B)	109.2
H(22A)-C(22)-H(22B)	109.5
C(19)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 98001. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Sb(1)	27(1)	40(1)	39(1)	8(1)	-6(1)	5(1)
F(1)	47(3)	64(4)	50(3)	-10(3)	-19(3)	-3(3)
F(2)	47(4)	120(6)	112(5)	64(5)	-46(4)	-26(4)
F(3)	93(5)	47(4)	136(6)	19(4)	-3(5)	25(3)
F(4)	83(4)	59(4)	87(4)	-38(3)	-36(4)	26(3)
F(5)	86(5)	69(4)	60(4)	2(3)	29(3)	-13(3)
F(6)	68(4)	181(8)	39(3)	19(4)	5(3)	-44(5)
C(1)	22(4)	36(4)	33(4)	10(4)	-1(4)	-4(4)
N(2)	17(3)	20(3)	26(3)	-2(3)	-4(3)	3(2)
N(3)	18(3)	33(4)	19(3)	8(3)	-2(3)	1(3)
C(4)	22(4)	44(5)	16(4)	15(4)	-3(3)	-3(4)
C(5)	30(4)	54(5)	27(4)	-18(4)	5(4)	-5(4)
C(6)	23(4)	29(4)	43(5)	-4(4)	-10(4)	-9(4)
C(7)	24(5)	41(5)	29(4)	7(4)	-2(3)	-10(4)
C(8)	15(4)	42(5)	31(4)	5(4)	0(3)	3(4)
C(9)	23(4)	39(5)	10(3)	1(3)	-1(3)	-12(4)
C(10)	32(5)	33(5)	32(4)	-2(4)	3(4)	-3(4)
C(11)	33(5)	58(6)	32(5)	-20(4)	3(4)	-11(4)
C(12)	43(6)	76(7)	18(4)	-4(5)	-5(4)	-16(5)
C(13)	34(6)	93(8)	27(5)	28(5)	-10(4)	-15(5)
C(14)	31(5)	69(7)	68(6)	57(6)	-14(5)	-14(5)
C(15)	28(5)	33(5)	63(6)	17(4)	-13(4)	3(4)
C(16)	26(5)	23(4)	51(5)	18(4)	-8(4)	-8(4)
C(17)	19(4)	34(5)	25(4)	20(3)	0(3)	-8(3)
C(18)	18(4)	65(6)	20(4)	17(4)	-7(3)	-18(4)
C(19)	20(4)	20(4)	24(4)	7(3)	3(3)	-2(3)
C(20)	20(4)	43(5)	34(5)	2(4)	5(3)	-3(4)
C(21)	44(5)	29(5)	38(5)	9(4)	11(4)	0(4)
C(22)	17(4)	55(5)	34(4)	18(4)	-2(3)	-8(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 98001.

	x	y	z	U(eq)
H(1)	4220	4389	7927	37
H(4)	4059	6337	6016	33
H(5A)	3581	4959	5700	44
H(5B)	4592	5025	5644	44
H(6A)	4729	4108	6596	38
H(6B)	3720	4003	6613	38
H(7A)	2839	4879	7527	38
H(7B)	3284	5499	8149	38
H(8A)	3238	6480	7234	36
H(8B)	2759	5870	6625	36
H(10)	4476	6620	8485	39
H(11)	4990	6896	9818	49
H(12)	5951	6052	10416	55
H(13)	6874	4799	10360	62
H(14)	7381	3699	9665	67
H(15)	6865	3370	8340	50
H(16)	5896	4251	7739	40
H(20A)	5265	6332	5500	48
H(20B)	6013	5759	5822	48
H(20C)	6191	6702	5633	48
H(21A)	5077	7539	7326	56
H(21B)	4756	7492	6407	56
H(21C)	5702	7780	6601	56
H(22A)	6210	6584	7788	53
H(22B)	6797	6704	7007	53
H(22C)	6462	5812	7244	53

Table 6. Torsion angles [°] for 98001.

C(6)-C(1)-N(2)-N(3)	60.0(8)
C(7)-C(1)-N(2)-N(3)	-59.7(8)
C(6)-C(1)-N(2)-C(9)	-154.1(6)
C(7)-C(1)-N(2)-C(9)	86.2(7)
C(9)-N(2)-N(3)-C(4)	-144.8(6)
C(1)-N(2)-N(3)-C(4)	0.0(8)
C(9)-N(2)-N(3)-C(19)	53.4(9)
C(1)-N(2)-N(3)-C(19)	-161.9(6)
N(2)-N(3)-C(4)-C(8)	60.2(8)
C(19)-N(3)-C(4)-C(8)	-137.1(6)
N(2)-N(3)-C(4)-C(5)	-58.3(7)
C(19)-N(3)-C(4)-C(5)	104.4(7)
N(3)-C(4)-C(5)-C(6)	54.3(8)
C(8)-C(4)-C(5)-C(6)	-63.6(8)
C(4)-C(5)-C(6)-C(1)	3.3(8)
N(2)-C(1)-C(6)-C(5)	-59.2(8)
C(7)-C(1)-C(6)-C(5)	59.8(8)
N(2)-C(1)-C(7)-C(8)	55.4(8)
C(6)-C(1)-C(7)-C(8)	-63.0(8)
C(1)-C(7)-C(8)-C(4)	2.1(8)
N(3)-C(4)-C(8)-C(7)	-59.0(8)
C(5)-C(4)-C(8)-C(7)	59.8(8)
N(3)-N(2)-C(9)-C(10)	48.0(9)
C(1)-N(2)-C(9)-C(10)	-95.4(8)
N(3)-N(2)-C(9)-C(17)	-135.2(6)
C(1)-N(2)-C(9)-C(17)	81.5(8)
C(17)-C(9)-C(10)-C(11)	1.5(11)
N(2)-C(9)-C(10)-C(11)	178.1(6)
C(9)-C(10)-C(11)-C(12)	-0.4(12)
C(10)-C(11)-C(12)-C(18)	-0.3(13)
C(18)-C(13)-C(14)-C(15)	-1.1(13)
C(13)-C(14)-C(15)-C(16)	1.9(12)
C(14)-C(15)-C(16)-C(17)	-2.1(11)
C(15)-C(16)-C(17)-C(9)	178.6(7)
C(15)-C(16)-C(17)-C(18)	1.6(11)
C(10)-C(9)-C(17)-C(16)	-178.9(7)
N(2)-C(9)-C(17)-C(16)	4.5(10)
C(10)-C(9)-C(17)-C(18)	-1.8(10)
N(2)-C(9)-C(17)-C(18)	-178.4(6)
C(14)-C(13)-C(18)-C(12)	-178.3(8)
C(14)-C(13)-C(18)-C(17)	0.5(12)
C(11)-C(12)-C(18)-C(13)	178.8(8)
C(11)-C(12)-C(18)-C(17)	0.0(12)
C(16)-C(17)-C(18)-C(13)	-0.7(10)
C(9)-C(17)-C(18)-C(13)	-177.9(7)
C(16)-C(17)-C(18)-C(12)	178.2(7)
C(9)-C(17)-C(18)-C(12)	1.0(10)
N(2)-N(3)-C(19)-C(22)	-5.8(9)
C(4)-N(3)-C(19)-C(22)	-166.4(6)
N(2)-N(3)-C(19)-C(21)	-127.9(7)
C(4)-N(3)-C(19)-C(21)	71.6(8)
N(2)-N(3)-C(19)-C(20)	113.0(7)
C(4)-N(3)-C(19)-C(20)	-47.5(8)

$Hy^2NA^+SbF_6^-$ (Nel011)

Data Collection

A black crystal with approximate dimensions 0.41 x 0.38 x 0.35 mm^3 was selected under oil under ambient conditions and attached to the tip of a nylon loop. The crystal was mounted in a stream of cold nitrogen at 100(2) K and centered in the X-ray beam by using a video camera.

The crystal evaluation and data collection were performed on a Bruker CCD-1000 diffractometer with Mo K_α (λ = 0.71073 Å) radiation and the diffractometer to crystal distance of 4.9 cm.

The initial cell constants were obtained from three series of ω scans at different starting angles. Each series consisted of 20 frames collected at intervals of 0.3º in a 6º range about ω with the exposure time of 10 seconds per frame. A total of 54 reflections was obtained. The reflections were successfully indexed by an automated indexing routine built in the SMART program. The final cell constants were calculated from a set of 6454 strong reflections from the actual data collection.

The data were collected by using the hemisphere data collection routine. The reciprocal space was surveyed to the extent of a full sphere to a resolution of 0.80 Å. A total of 16605 data were harvested by collecting three sets of frames with 0.3º scans in ω with an exposure time eight seconds per frame. These highly redundant datasets were corrected for Lorentz and polarization effects. The absorption correction was based on fitting a function to the empirical transmission surface as sampled by multiple equivalent measurements.[14]

Structure Solution and Refinement

The systematic absences in the diffraction data were consistent for the space groups *Cc* and *C*2/*c*. The *E*-statistics strongly suggested the centrosymmetric space group *C*2/*c* that yielded chemically reasonable and computationally stable results of refinement [1]. A successful solution by the direct methods provided most non-hydrogen atoms from the *E*-map. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement coefficients. All hydrogen atoms were included in the structure factor calculation at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement

coefficients. There are two half-ions of SbF_6^- in the asymmetric unit. The Sb(1) anion occupies a crystallographic 2-fold axis while the Sb(2) anion resides on a crystallographic inversion center. Atoms F(2), F(3), and F(4) are equally disordered over two positions each; the Sb(1) moiety was refined with soft restraints.

The final least-squares refinement of 572 parameters against 6071 data resulted in residuals *R* (based on F^2 for $I \geq 2\sigma$) and *wR* (based on F^2 for all data) of 0.0259 and 0.0960, respectively. The final difference Fourier map was featureless.

Table 1. Crystal data and structure refinement for nel11.

Identification code	nel11	
Empirical formula	$C_{20}\ H_{26}\ F_6\ N_2\ Sb$	
Formula weight	530.18	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 22.587(3) Å	α= 90°.
	b = 14.2553(18) Å	β= 124.757(2)°.
	c = 16.202(2) Å	γ = 90°.
Volume	4285.9(10) $Å^3$	
Z	8	
Density (calculated)	1.643 Mg/m^3	
Absorption coefficient	1.347 mm^{-1}	
F(000)	2120	
Crystal size	0.41 x 0.38 x 0.35 mm^3	
Theta range for data collection	2.20 to 26.45°.	
Index ranges	-28<=h<=27, -17<=k<=17, -20<=l<=20	
Reflections collected	16605	
Independent reflections	4386 [R(int) = 0.0425]	
Completeness to theta = 26.45°	99.2 %	
Absorption correction	Multiscan with SADABS	
Max. and min. transmission	0.6500 and 0.6082	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4386 / 6 / 291	
Goodness-of-fit on F^2	1.135	
Final R indices [I>2sigma(I)]	R1 = 0.0447, wR2 = 0.1046	
R indices (all data)	R1 = 0.0525, wR2 = 0.1081	
Largest diff. peak and hole	1.804 and -1.631 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for nel11. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Sb(1)	0	1651(1)	2500	19(1)
Sb(2)	2500	2500	0	16(1)
F(1)	1008(1)	1636(2)	3204(2)	38(1)
F(2)	-43(9)	2923(2)	2256(9)	65(3)
F(3)	-73(6)	1916(6)	1329(5)	54(2)

F(3A)	-76(5)	1386(7)	1327(4)	67(3)
F(4)	-93(6)	391(2)	2184(6)	73(4)
F(5)	2793(1)	1245(2)	333(2)	30(1)
F(6)	2429(2)	2581(2)	1095(2)	31(1)
F(7)	3458(1)	2898(2)	815(2)	34(1)
N(1)	2824(1)	4270(2)	3464(2)	14(1)
N(2)	2684(2)	4687(2)	2624(2)	16(1)
C(1)	2347(2)	3512(2)	3457(3)	16(1)
C(2)	2771(2)	2586(2)	3764(3)	23(1)
C(3)	2186(2)	3795(3)	4221(3)	24(1)
C(4)	1654(2)	3377(2)	2412(3)	23(1)
C(5)	3586(2)	4363(2)	4329(3)	18(1)
C(6)	3735(2)	5417(3)	4503(3)	23(1)
C(7)	3628(2)	5855(2)	3555(3)	25(1)
C(8)	3336(2)	5086(3)	2741(3)	21(1)
C(9)	3876(2)	4292(3)	3055(3)	26(1)
C(10)	4084(2)	3915(3)	4077(3)	22(1)
C(11)	2011(2)	5162(2)	1958(2)	16(1)
C(12)	1693(2)	5655(2)	2332(3)	16(1)
C(13)	1023(2)	6105(2)	1662(3)	17(1)
C(14)	661(2)	6611(2)	2006(3)	21(1)
C(15)	14(2)	7033(3)	1339(3)	23(1)
C(16)	-306(2)	6972(3)	300(3)	25(1)
C(17)	31(2)	6500(2)	-57(3)	22(1)
C(18)	701(2)	6050(2)	609(3)	18(1)
C(19)	1065(2)	5549(2)	263(3)	20(1)
C(20)	1703(2)	5110(2)	912(3)	20(1)

Table 3. Bond lengths [Å] and angles [°] for nel11.

Sb(1)-F(2)#1	1.847(3)	N(2)-C(11)	1.437(4)
Sb(1)-F(2)	1.847(3)	N(2)-C(8)	1.486(4)
Sb(1)-F(3)#1	1.847(3)	C(1)-C(4)	1.527(5)
Sb(1)-F(3)	1.847(3)	C(1)-C(3)	1.529(5)
Sb(1)-F(3A)	1.847(3)	C(1)-C(2)	1.537(5)
Sb(1)-F(3A)#1	1.847(3)	C(5)-C(6)	1.531(5)
Sb(1)-F(4)	1.847(3)	C(5)-C(10)	1.538(5)
Sb(1)-F(4)#1	1.847(3)	C(6)-C(7)	1.544(5)
Sb(1)-F(1)	1.877(2)	C(7)-C(8)	1.544(5)
Sb(1)-F(1)#1	1.877(2)	C(8)-C(9)	1.523(5)
Sb(2)-F(7)#2	1.868(2)	C(9)-C(10)	1.537(5)
Sb(2)-F(7)	1.868(2)	C(11)-C(12)	1.368(5)
Sb(2)-F(6)	1.875(2)	C(11)-C(20)	1.421(5)
Sb(2)-F(6)#2	1.875(2)	C(12)-C(13)	1.417(5)
Sb(2)-F(5)#2	1.878(2)	C(13)-C(14)	1.419(5)
Sb(2)-F(5)	1.878(2)	C(13)-C(18)	1.425(5)
F(2)-F(2)#1	0.698(17)	C(14)-C(15)	1.366(5)
F(3)-F(3A)	0.757(12)	C(15)-C(16)	1.406(5)
F(4)-F(4)#1	0.858(18)	C(16)-C(17)	1.365(6)
N(1)-N(2)	1.347(4)	C(17)-C(18)	1.416(5)
N(1)-C(5)	1.482(4)	C(18)-C(19)	1.424(5)
N(1)-C(1)	1.521(4)	C(19)-C(20)	1.360(5)

F(2)#1-Sb(1)-F(2)	21.8(5)
F(2)#1-Sb(1)-F(3)#1	67.4(4)
F(2)-Sb(1)-F(3)#1	89.0(4)
F(2)#1-Sb(1)-F(3)	89.0(4)
F(2)-Sb(1)-F(3)	67.4(4)
F(3)#1-Sb(1)-F(3)	156.4(5)
F(2)#1-Sb(1)-F(3A)	112.6(4)
F(2)-Sb(1)-F(3A)	91.1(4)
F(3)#1-Sb(1)-F(3A)	179.8(6)
F(3)-Sb(1)-F(3A)	23.6(4)
F(2)#1-Sb(1)-F(3A)#1	91.1(4)
F(2)-Sb(1)-F(3A)#1	112.6(4)
F(3)#1-Sb(1)-F(3A)#1	23.6(4)
F(3)-Sb(1)-F(3A)#1	179.8(6)
F(3A)-Sb(1)-F(3A)#1	156.3(6)
F(2)#1-Sb(1)-F(4)	176.7(5)
F(2)-Sb(1)-F(4)	155.8(4)
F(3)#1-Sb(1)-F(4)	114.4(5)
F(3)-Sb(1)-F(4)	89.1(5)
F(3A)-Sb(1)-F(4)	65.5(5)
F(3A)#1-Sb(1)-F(4)	90.9(5)
F(2)#1-Sb(1)-F(4)#1	155.8(4)
F(2)-Sb(1)-F(4)#1	176.7(5)
F(3)#1-Sb(1)-F(4)#1	89.1(5)
F(3)-Sb(1)-F(4)#1	114.4(5)
F(3A)-Sb(1)-F(4)#1	90.9(5)
F(3A)#1-Sb(1)-F(4)#1	65.5(5)
F(4)-Sb(1)-F(4)#1	26.9(6)
F(2)#1-Sb(1)-F(1)	89.0(6)
F(2)-Sb(1)-F(1)	92.2(6)
F(3)#1-Sb(1)-F(1)	90.6(4)
F(3)-Sb(1)-F(1)	89.6(4)
F(3A)-Sb(1)-F(1)	89.6(3)
F(3A)#1-Sb(1)-F(1)	90.1(3)
F(4)-Sb(1)-F(1)	93.7(4)
F(4)#1-Sb(1)-F(1)	85.1(4)
F(2)#1-Sb(1)-F(1)#1	92.2(6)
F(2)-Sb(1)-F(1)#1	89.0(6)
F(3)#1-Sb(1)-F(1)#1	89.6(4)
F(3)-Sb(1)-F(1)#1	90.6(4)
F(3A)-Sb(1)-F(1)#1	90.1(3)
F(3A)#1-Sb(1)-F(1)#1	89.6(3)
F(4)-Sb(1)-F(1)#1	85.1(4)
F(4)#1-Sb(1)-F(1)#1	93.7(4)
F(1)-Sb(1)-F(1)#1	178.71(16)
F(7)#2-Sb(2)-F(7)	180.00(11)
F(7)#2-Sb(2)-F(6)	89.86(11)
F(7)-Sb(2)-F(6)	90.14(11)
F(7)#2-Sb(2)-F(6)#2	90.14(11)
F(7)-Sb(2)-F(6)#2	89.86(11)
F(6)-Sb(2)-F(6)#2	180.0(2)
F(7)#2-Sb(2)-F(5)#2	90.58(11)
F(7)-Sb(2)-F(5)#2	89.42(11)
F(6)-Sb(2)-F(5)#2	90.42(9)
F(6)#2-Sb(2)-F(5)#2	89.58(9)
F(7)#2-Sb(2)-F(5)	89.42(11)

F(7)-Sb(2)-F(5)	90.58(11)
F(6)-Sb(2)-F(5)	89.58(9)
F(6)#2-Sb(2)-F(5)	90.42(9)
F(5)#2-Sb(2)-F(5)	180.0
F(2)#1-F(2)-Sb(1)	79.1(3)
F(3A)-F(3)-Sb(1)	78.2(2)
F(3)-F(3A)-Sb(1)	78.2(2)
F(4)#1-F(4)-Sb(1)	76.6(3)
N(2)-N(1)-C(5)	113.1(3)
N(2)-N(1)-C(1)	123.3(3)
C(5)-N(1)-C(1)	120.4(3)
N(1)-N(2)-C(11)	120.3(3)
N(1)-N(2)-C(8)	113.4(3)
C(11)-N(2)-C(8)	117.5(3)
N(1)-C(1)-C(4)	111.8(3)
N(1)-C(1)-C(3)	107.5(3)
C(4)-C(1)-C(3)	111.4(3)
N(1)-C(1)-C(2)	107.5(3)
C(4)-C(1)-C(2)	107.8(3)
C(3)-C(1)-C(2)	110.9(3)
N(1)-C(5)-C(6)	106.1(3)
N(1)-C(5)-C(10)	110.0(3)
C(6)-C(5)-C(10)	109.9(3)
C(5)-C(6)-C(7)	108.7(3)
C(6)-C(7)-C(8)	108.2(3)
N(2)-C(8)-C(9)	107.7(3)
N(2)-C(8)-C(7)	106.9(3)
C(9)-C(8)-C(7)	111.8(3)
C(8)-C(9)-C(10)	108.4(3)
C(5)-C(10)-C(9)	108.6(3)
C(12)-C(11)-C(20)	122.1(3)
C(12)-C(11)-N(2)	120.5(3)
C(20)-C(11)-N(2)	117.4(3)
C(11)-C(12)-C(13)	119.7(3)
C(14)-C(13)-C(12)	122.1(3)
C(14)-C(13)-C(18)	118.9(3)
C(12)-C(13)-C(18)	119.0(3)
C(15)-C(14)-C(13)	120.6(3)
C(14)-C(15)-C(16)	120.5(4)
C(17)-C(16)-C(15)	120.5(3)
C(16)-C(17)-C(18)	120.7(3)
C(17)-C(18)-C(19)	122.2(3)
C(17)-C(18)-C(13)	118.8(3)
C(19)-C(18)-C(13)	119.0(3)
C(20)-C(19)-C(18)	121.5(3)
C(19)-C(20)-C(11)	118.7(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x,y,-z+1/2 #2 -x+1/2,-y+1/2,-z

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for nel11. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Sb(1)	20(1)	19(1)	20(1)	0	13(1)	0
Sb(2)	21(1)	12(1)	18(1)	2(1)	13(1)	3(1)
F(1)	18(1)	56(2)	36(1)	7(1)	13(1)	-3(1)
F(2)	59(5)	36(3)	111(12)	43(4)	55(10)	14(5)
F(3)	57(5)	89(6)	29(3)	10(4)	32(3)	-10(5)
F(3A)	25(3)	147(9)	27(3)	-28(4)	14(3)	7(6)
F(4)	33(6)	25(2)	139(11)	-12(3)	37(8)	1(3)
F(5)	47(2)	16(1)	35(1)	8(1)	28(1)	10(1)
F(6)	50(2)	27(1)	30(1)	5(1)	31(1)	7(1)
F(7)	22(1)	36(1)	34(1)	7(1)	9(1)	-5(1)
N(1)	15(1)	10(1)	16(1)	1(1)	9(1)	-1(1)
N(2)	19(2)	12(1)	19(2)	0(1)	12(1)	-1(1)
C(1)	19(2)	10(2)	21(2)	0(1)	12(2)	-4(1)
C(2)	28(2)	10(2)	30(2)	4(1)	16(2)	2(1)
C(3)	30(2)	23(2)	25(2)	-1(2)	20(2)	-3(2)
C(4)	23(2)	15(2)	26(2)	0(2)	12(2)	-7(1)
C(5)	16(2)	17(2)	20(2)	1(1)	9(2)	1(1)
C(6)	21(2)	18(2)	27(2)	-7(2)	11(2)	-4(2)
C(7)	23(2)	17(2)	32(2)	-1(2)	14(2)	-7(2)
C(8)	20(2)	21(2)	26(2)	4(2)	15(2)	-1(2)
C(9)	24(2)	30(2)	32(2)	1(2)	19(2)	4(2)
C(10)	18(2)	19(2)	32(2)	3(2)	15(2)	4(1)
C(11)	18(2)	10(2)	19(2)	1(1)	10(1)	-2(1)
C(12)	20(2)	11(2)	17(2)	-1(1)	10(1)	-3(1)
C(13)	19(2)	11(2)	18(2)	-1(1)	9(2)	-3(1)
C(14)	22(2)	20(2)	20(2)	0(1)	11(2)	0(1)
C(15)	22(2)	21(2)	30(2)	2(2)	16(2)	1(2)
C(16)	17(2)	19(2)	32(2)	7(2)	10(2)	-1(2)
C(17)	20(2)	20(2)	18(2)	4(2)	5(2)	-2(1)
C(18)	17(2)	11(2)	22(2)	0(1)	8(2)	-6(1)
C(19)	24(2)	15(2)	15(2)	-1(1)	8(2)	-4(1)
C(20)	25(2)	16(2)	24(2)	-3(1)	17(2)	-4(2)

Table 5. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for nel11.

	x	y	z	U(eq)
H(2A)	2880	2428	3276	34
H(2B)	2481	2084	3779	34
H(2C)	3221	2655	4432	34
H(3A)	2640	3873	4883	36
H(3B)	1897	3305	4257	36
H(3C)	1918	4387	4013	36
H(4A)	1342	3925	2238	34
H(4B)	1404	2814	2408	34
H(4C)	1772	3306	1920	34
H(5)	3661	4060	4939	22
H(6A)	4234	5526	5092	28
H(6B)	3401	5711	4637	28

H(7A)	4092	6098	3713	30
H(7B)	3282	6382	3312	30
H(8)	3207	5364	2091	25
H(9A)	4309	4526	3109	32
H(9B)	3658	3785	2547	32
H(10A)	4032	3224	4048	27
H(10B)	4593	4072	4603	27
H(12)	1920	5694	3037	19
H(14)	872	6656	2706	25
H(15)	-222	7369	1579	28
H(16)	-759	7262	-157	30
H(17)	-187	6474	-760	27
H(19)	856	5520	-438	23
H(20)	1939	4775	670	24

Table 6. Torsion angles [°] for nel11.

F(3)#1-Sb(1)-F(2)-F(2)#1	9(3)
F(3)-Sb(1)-F(2)-F(2)#1	-170(3)
F(3A)-Sb(1)-F(2)-F(2)#1	-171(3)
F(3A)#1-Sb(1)-F(2)-F(2)#1	9(3)
F(4)-Sb(1)-F(2)-F(2)#1	174.2(19)
F(4)#1-Sb(1)-F(2)-F(2)#1	-46(12)
F(1)-Sb(1)-F(2)-F(2)#1	-82(3)
F(1)#1-Sb(1)-F(2)-F(2)#1	99(3)
F(2)#1-Sb(1)-F(3)-F(3A)	178.8(16)
F(2)-Sb(1)-F(3)-F(3A)	-177.6(17)
F(3)#1-Sb(1)-F(3)-F(3A)	-179.5(15)
F(3A)#1-Sb(1)-F(3)-F(3A)	87(100)
F(4)-Sb(1)-F(3)-F(3A)	-3.9(15)
F(4)#1-Sb(1)-F(3)-F(3A)	5.4(17)
F(1)-Sb(1)-F(3)-F(3A)	89.8(15)
F(1)#1-Sb(1)-F(3)-F(3A)	-89.0(15)
F(2)#1-Sb(1)-F(3A)-F(3)	-1.3(17)
F(2)-Sb(1)-F(3A)-F(3)	2.2(16)
F(3)#1-Sb(1)-F(3A)-F(3)	94(100)
F(3A)#1-Sb(1)-F(3A)-F(3)	-179.5(15)
F(4)-Sb(1)-F(3A)-F(3)	175.7(16)
F(4)#1-Sb(1)-F(3A)-F(3)	-175.1(15)
F(1)-Sb(1)-F(3A)-F(3)	-90.1(15)
F(1)#1-Sb(1)-F(3A)-F(3)	91.2(15)
F(2)#1-Sb(1)-F(4)-F(4)#1	-144(9)
F(2)-Sb(1)-F(4)-F(4)#1	175.3(16)
F(3)#1-Sb(1)-F(4)-F(4)#1	-21(2)
F(3)-Sb(1)-F(4)-F(4)#1	161.1(19)
F(3A)-Sb(1)-F(4)-F(4)#1	159(2)
F(3A)#1-Sb(1)-F(4)-F(4)#1	-18.7(19)
F(1)-Sb(1)-F(4)-F(4)#1	71.5(19)
F(1)#1-Sb(1)-F(4)-F(4)#1	-108.2(19)
C(5)-N(1)-N(2)-C(11)	-142.7(3)
C(1)-N(1)-N(2)-C(11)	57.4(4)
C(5)-N(1)-N(2)-C(8)	3.9(4)
C(1)-N(1)-N(2)-C(8)	-156.0(3)
N(2)-N(1)-C(1)-C(4)	-7.2(4)
C(5)-N(1)-C(1)-C(4)	-165.8(3)
N(2)-N(1)-C(1)-C(3)	-129.7(3)
C(5)-N(1)-C(1)-C(3)	71.7(4)
N(2)-N(1)-C(1)-C(2)	110.9(3)
C(5)-N(1)-C(1)-C(2)	-47.7(4)
N(2)-N(1)-C(5)-C(6)	58.9(3)
C(1)-N(1)-C(5)-C(6)	-140.4(3)
N(2)-N(1)-C(5)-C(10)	-59.8(3)
C(1)-N(1)-C(5)-C(10)	100.8(3)
N(1)-C(5)-C(6)-C(7)	-61.7(4)
C(10)-C(5)-C(6)-C(7)	57.2(4)
C(5)-C(6)-C(7)-C(8)	5.6(4)
N(1)-N(2)-C(8)-C(9)	57.7(4)
C(11)-N(2)-C(8)-C(9)	-154.7(3)
N(1)-N(2)-C(8)-C(7)	-62.6(3)
C(11)-N(2)-C(8)-C(7)	85.1(4)
C(6)-C(7)-C(8)-N(2)	53.8(4)
C(6)-C(7)-C(8)-C(9)	-63.8(4)
N(2)-C(8)-C(9)-C(10)	-62.3(4)

C(7)-C(8)-C(9)-C(10)	54.7(4)
N(1)-C(5)-C(10)-C(9)	49.8(4)
C(6)-C(5)-C(10)-C(9)	-66.6(4)
C(8)-C(9)-C(10)-C(5)	9.0(4)
N(1)-N(2)-C(11)-C(12)	36.6(4)
C(8)-N(2)-C(11)-C(12)	-108.7(4)
N(1)-N(2)-C(11)-C(20)	-144.4(3)
C(8)-N(2)-C(11)-C(20)	70.3(4)
C(20)-C(11)-C(12)-C(13)	2.1(5)
N(2)-C(11)-C(12)-C(13)	-178.9(3)
C(11)-C(12)-C(13)-C(14)	178.8(3)
C(11)-C(12)-C(13)-C(18)	-1.3(5)
C(12)-C(13)-C(14)-C(15)	-179.7(3)
C(18)-C(13)-C(14)-C(15)	0.4(5)
C(13)-C(14)-C(15)-C(16)	0.1(5)
C(14)-C(15)-C(16)-C(17)	-0.8(6)
C(15)-C(16)-C(17)-C(18)	1.0(5)
C(16)-C(17)-C(18)-C(19)	179.7(3)
C(16)-C(17)-C(18)-C(13)	-0.6(5)
C(14)-C(13)-C(18)-C(17)	-0.1(5)
C(12)-C(13)-C(18)-C(17)	179.9(3)
C(14)-C(13)-C(18)-C(19)	179.6(3)
C(12)-C(13)-C(18)-C(19)	-0.4(5)
C(17)-C(18)-C(19)-C(20)	-179.1(3)
C(13)-C(18)-C(19)-C(20)	1.2(5)
C(18)-C(19)-C(20)-C(11)	-0.4(5)
C(12)-C(11)-C(20)-C(19)	-1.3(5)
N(2)-C(11)-C(20)-C(19)	179.6(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x,y,-z+1/2 #2 -x+1/2,-y+1/2,-z

$Hy_2^{15}NA^{2+}(BF_4^-)_2$ (*nel06*)

Data Collection

A crystal with approximate dimensions 0.21 x 0.14 x 0.10 mm^3 was selected under oil under ambient conditions and attached to the tip of a glass capillary. The crystal was mounted in a stream of cold nitrogen at 100(2) K and centered in the X-ray beam by using a video camera.

The crystal evaluation and data collection were performed on a Bruker CCD-1000 diffractometer with Mo K_α (λ = 0.71073 Å) radiation and the diffractometer to crystal distance of 4.9 cm.

The initial cell constants were obtained from three series of ω scans at different starting angles. Each series consisted of 20 frames collected at intervals of 0.3º in a 6º range about ω with the exposure time of 10 seconds per frame. A total of 44 reflections was obtained. The reflections were successfully indexed by an automated indexing routine built in the SMART program. The final cell constants were calculated from a set of 12142 strong reflections from the actual data collection.

The data were collected by using the hemisphere data collection routine. The reciprocal space was surveyed to the extent of a full sphere to a resolution of 0.80 Å. A total of 12234 data were harvested by collecting three sets of frames with 0.3º scans in ω with an exposure time 70 sec per frame. These highly redundant datasets were corrected for Lorentz and polarization effects. The absorption correction was based on fitting a function to the empirical transmission surface as sampled by multiple equivalent measurements.[15]

Structure Solution and Refinement

The systematic absences in the diffraction data were uniquely consistent for the space group $P2_1/c$ that yielded chemically reasonable and computationally stable results of refinement.[16]

A successful solution by the direct methods provided most non-hydrogen atoms from the *E*-map. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement coefficients. All hydrogen atoms were included in the structure factor calculation at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients. The cation occupies a crystallographic inversion center.

The final least-squares refinement of 382 parameters against 4700 data resulted in residuals *R* (based on F^2 for $I \geq 2\sigma$) and *wR* (based on F^2 for all data) of 0.0689 and 0.1716, respectively. The final difference Fourier map was featureless.

Table 1. Crystal data and structure refinement for nel06.

Identification code	nel06	
Empirical formula	$C_{78}\ H_{84}\ B_2\ N_4$	
Formula weight	1099.11	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 11.8549(16) Å	α= 90°.
	b = 13.3075(18) Å	β= 103.070(2)°.
	c = 19.512(3) Å	γ = 90°.
Volume	2998.5(7) $Å^3$	
Z	2	
Density (calculated)	1.217 Mg/m^3	
Absorption coefficient	0.069 mm^{-1}	
F(000)	1180	
Crystal size	0.21 x 0.14 x 0.10 mm^3	
Theta range for data collection	2.14 to 24.00°.	
Index ranges	-13<=h<=13, -15<=k<=15, -22<=l<=22	
Reflections collected	19948	
Independent reflections	4700 [R(int) = 0.0532]	
Completeness to theta = 24.00°	99.8 %	
Absorption correction	Empirical with SADABS	
Max. and min. transmission	0.9931 and 0.9856	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4700 / 0 / 382	
Goodness-of-fit on F^2	1.083	
Final R indices [I>2sigma(I)]	R1 = 0.0689, wR2 = 0.1642	
R indices (all data)	R1 = 0.0867, wR2 = 0.1716	
Largest diff. peak and hole	0.612 and -0.262 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for nel06. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor. (nel06)

	x	y	z	U(eq)
N(1)	1911(2)	6568(2)	524(1)	25(1)
N(2)	2040(2)	7511(2)	772(1)	20(1)
B(1)	7594(3)	7247(2)	2565(2)	16(1)
C(1)	456(2)	5335(2)	-31(2)	17(1)
C(2)	889(3)	5990(2)	548(2)	20(1)
C(3)	428(3)	6011(2)	1134(2)	20(1)
C(4)	-477(2)	5348(2)	1177(2)	18(1)
C(5)	-893(2)	4683(2)	649(2)	18(1)
C(6)	1162(3)	8339(2)	491(2)	27(1)
C(7)	1791(3)	9154(3)	165(2)	33(1)
C(8)	755(3)	8748(2)	1119(2)	27(1)
C(9)	163(3)	7953(3)	-70(2)	35(1)

C(10)	3230(3)	7760(2)	1153(2)	25(1)
C(11)	3545(3)	6999(2)	1756(2)	28(1)
C(12)	3527(3)	5938(3)	1438(2)	29(1)
C(13)	3048(3)	6013(2)	651(2)	25(1)
C(14)	3829(3)	6631(2)	287(2)	26(1)
C(15)	4079(3)	7648(2)	666(2)	27(1)
C(16)	7155(2)	8200(2)	2035(2)	17(1)
C(17)	7576(3)	8370(2)	1428(2)	20(1)
C(18)	7141(3)	9099(2)	935(2)	24(1)
C(19)	6263(3)	9732(2)	1034(2)	28(1)
C(20)	5841(3)	9621(2)	1640(2)	26(1)
C(21)	6283(3)	8872(2)	2124(2)	21(1)
C(22)	7155(2)	7418(2)	3302(2)	17(1)
C(23)	6103(2)	7043(2)	3409(2)	17(1)
C(24)	5726(3)	7197(2)	4022(2)	21(1)
C(25)	6389(3)	7762(2)	4564(2)	21(1)
C(26)	7413(3)	8172(2)	4475(2)	22(1)
C(27)	7784(3)	8008(2)	3856(2)	20(1)
C(28)	9022(3)	7163(2)	2752(1)	18(1)
C(29)	9746(3)	8007(2)	2785(2)	21(1)
C(30)	10949(3)	7945(2)	2971(2)	26(1)
C(31)	11488(3)	7025(3)	3137(2)	29(1)
C(32)	10813(3)	6166(3)	3110(2)	27(1)
C(33)	9615(3)	6248(2)	2922(2)	22(1)
C(34)	7053(2)	6191(2)	2169(2)	16(1)
C(35)	6862(2)	5327(2)	2540(2)	19(1)
C(36)	6435(3)	4434(2)	2209(2)	21(1)
C(37)	6198(3)	4353(2)	1486(2)	20(1)
C(38)	6395(2)	5180(2)	1100(2)	18(1)
C(39)	6802(2)	6071(2)	1437(2)	16(1)

Table 3. Bond lengths [Å] and angles [°] for nel06.

N(1)-N(2)	1.342(3)
N(1)-C(2)	1.444(4)
N(1)-C(13)	1.507(4)
N(2)-C(10)	1.476(4)
N(2)-C(6)	1.530(4)
B(1)-C(16)	1.645(4)
B(1)-C(22)	1.650(4)
B(1)-C(28)	1.653(4)
B(1)-C(34)	1.662(4)
C(1)-C(5)#1	1.418(4)
C(1)-C(2)	1.426(4)
C(1)-C(1)#1	1.428(6)
C(2)-C(3)	1.374(4)
C(3)-C(4)	1.406(4)
C(4)-C(5)	1.363(4)
C(5)-C(1)#1	1.418(4)
C(6)-C(9)	1.510(5)
C(6)-C(8)	1.516(4)
C(6)-C(7)	1.533(5)
C(10)-C(11)	1.533(4)
C(10)-C(15)	1.539(4)
C(11)-C(12)	1.540(5)
C(12)-C(13)	1.516(4)
C(13)-C(14)	1.529(4)
C(14)-C(15)	1.539(4)
C(16)-C(17)	1.405(4)
C(16)-C(21)	1.407(4)
C(17)-C(18)	1.381(4)
C(18)-C(19)	1.385(5)
C(19)-C(20)	1.391(5)
C(20)-C(21)	1.392(4)
C(22)-C(23)	1.402(4)
C(22)-C(27)	1.406(4)
C(23)-C(24)	1.384(4)
C(24)-C(25)	1.387(4)
C(25)-C(26)	1.378(4)
C(26)-C(27)	1.394(4)
C(28)-C(29)	1.406(4)
C(28)-C(33)	1.407(4)
C(29)-C(30)	1.392(4)
C(30)-C(31)	1.386(5)
C(31)-C(32)	1.390(5)
C(32)-C(33)	1.389(4)
C(34)-C(39)	1.401(4)
C(34)-C(35)	1.403(4)
C(35)-C(36)	1.392(4)
C(36)-C(37)	1.378(4)
C(37)-C(38)	1.384(4)
C(38)-C(39)	1.388(4)

N(2)-N(1)-C(2)	120.9(2)
N(2)-N(1)-C(13)	112.0(2)
C(2)-N(1)-C(13)	117.4(2)
N(1)-N(2)-C(10)	114.2(2)
N(1)-N(2)-C(6)	122.2(2)
C(10)-N(2)-C(6)	120.9(2)

C(16)-B(1)-C(22)	108.9(2)
C(16)-B(1)-C(28)	110.7(2)
C(22)-B(1)-C(28)	109.1(2)
C(16)-B(1)-C(34)	109.0(2)
C(22)-B(1)-C(34)	111.0(2)
C(28)-B(1)-C(34)	108.2(2)
C(5)#1-C(1)-C(2)	123.3(3)
C(5)#1-C(1)-C(1)#1	119.4(3)
C(2)-C(1)-C(1)#1	117.3(3)
C(3)-C(2)-C(1)	122.1(3)
C(3)-C(2)-N(1)	120.7(3)
C(1)-C(2)-N(1)	117.0(3)
C(2)-C(3)-C(4)	119.3(3)
C(5)-C(4)-C(3)	120.9(3)
C(4)-C(5)-C(1)#1	120.9(3)
C(9)-C(6)-C(8)	111.6(3)
C(9)-C(6)-N(2)	111.9(3)
C(8)-C(6)-N(2)	106.5(2)
C(9)-C(6)-C(7)	108.4(3)
C(8)-C(6)-C(7)	110.8(3)
N(2)-C(6)-C(7)	107.6(3)
N(2)-C(10)-C(11)	106.1(2)
N(2)-C(10)-C(15)	110.6(2)
C(11)-C(10)-C(15)	109.2(3)
C(10)-C(11)-C(12)	108.6(3)
C(13)-C(12)-C(11)	108.4(3)
N(1)-C(13)-C(12)	108.4(2)
N(1)-C(13)-C(14)	105.8(2)
C(12)-C(13)-C(14)	112.2(3)
C(13)-C(14)-C(15)	108.7(3)
C(14)-C(15)-C(10)	107.5(3)
C(17)-C(16)-C(21)	114.4(3)
C(17)-C(16)-B(1)	122.2(3)
C(21)-C(16)-B(1)	123.3(3)
C(18)-C(17)-C(16)	123.5(3)
C(17)-C(18)-C(19)	120.3(3)
C(18)-C(19)-C(20)	118.6(3)
C(19)-C(20)-C(21)	120.0(3)
C(20)-C(21)-C(16)	123.1(3)
C(23)-C(22)-C(27)	114.7(3)
C(23)-C(22)-B(1)	123.2(3)
C(27)-C(22)-B(1)	122.0(3)
C(24)-C(23)-C(22)	123.3(3)
C(23)-C(24)-C(25)	120.0(3)
C(26)-C(25)-C(24)	118.9(3)
C(25)-C(26)-C(27)	120.4(3)
C(26)-C(27)-C(22)	122.5(3)
C(29)-C(28)-C(33)	114.4(3)
C(29)-C(28)-B(1)	122.7(3)
C(33)-C(28)-B(1)	122.8(3)
C(30)-C(29)-C(28)	122.9(3)
C(31)-C(30)-C(29)	120.3(3)
C(30)-C(31)-C(32)	119.1(3)
C(33)-C(32)-C(31)	119.4(3)
C(32)-C(33)-C(28)	123.9(3)
C(39)-C(34)-C(35)	114.3(3)

C(39)-C(34)-B(1)	122.8(2)
C(35)-C(34)-B(1)	122.9(2)
C(36)-C(35)-C(34)	123.0(3)
C(37)-C(36)-C(35)	120.7(3)
C(36)-C(37)-C(38)	118.3(3)
C(37)-C(38)-C(39)	120.3(3)
C(38)-C(39)-C(34)	123.5(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for nel06. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	33(2)	22(1)	22(1)	-4(1)	11(1)	-8(1)
N(2)	20(1)	20(1)	20(1)	-8(1)	6(1)	-3(1)
B(1)	20(2)	16(2)	12(2)	0(1)	3(1)	-2(1)
C(1)	21(2)	17(2)	15(2)	2(1)	7(1)	5(1)
C(2)	24(2)	16(2)	21(2)	-1(1)	7(1)	-5(1)
C(3)	26(2)	18(2)	17(2)	-3(1)	6(1)	-1(1)
C(4)	21(2)	22(2)	12(2)	-1(1)	7(1)	1(1)
C(5)	16(2)	19(2)	18(2)	3(1)	5(1)	-2(1)
C(6)	30(2)	23(2)	27(2)	2(1)	5(1)	2(1)
C(7)	40(2)	27(2)	33(2)	8(2)	8(2)	1(2)
C(8)	26(2)	22(2)	30(2)	-1(1)	2(1)	5(1)
C(9)	43(2)	26(2)	31(2)	6(2)	0(2)	-3(2)
C(10)	25(2)	26(2)	21(2)	-3(1)	2(1)	-4(1)
C(11)	30(2)	30(2)	24(2)	1(1)	4(1)	2(2)
C(12)	26(2)	33(2)	31(2)	4(2)	9(2)	1(2)
C(13)	20(2)	23(2)	33(2)	-7(1)	7(1)	3(1)
C(14)	23(2)	31(2)	24(2)	-3(1)	5(1)	1(1)
C(15)	24(2)	29(2)	29(2)	-1(1)	6(1)	-4(1)
C(16)	21(2)	14(2)	16(2)	-4(1)	2(1)	-6(1)
C(17)	23(2)	13(2)	23(2)	-3(1)	3(1)	-4(1)
C(18)	33(2)	19(2)	20(2)	1(1)	7(1)	-8(1)
C(19)	41(2)	18(2)	23(2)	6(1)	-1(2)	-1(2)
C(20)	32(2)	17(2)	25(2)	-1(1)	1(1)	6(1)
C(21)	26(2)	19(2)	19(2)	-4(1)	3(1)	-2(1)
C(22)	19(2)	15(2)	16(2)	2(1)	2(1)	4(1)
C(23)	20(2)	16(2)	14(2)	0(1)	-1(1)	3(1)
C(24)	20(2)	20(2)	23(2)	4(1)	7(1)	4(1)
C(25)	25(2)	25(2)	15(2)	1(1)	6(1)	7(1)
C(26)	27(2)	20(2)	16(2)	-6(1)	1(1)	3(1)
C(27)	18(2)	21(2)	20(2)	-1(1)	4(1)	1(1)
C(28)	24(2)	22(2)	8(1)	-1(1)	7(1)	1(1)
C(29)	25(2)	21(2)	19(2)	-3(1)	7(1)	-2(1)
C(30)	24(2)	31(2)	25(2)	-4(1)	10(1)	-8(1)
C(31)	16(2)	44(2)	27(2)	-2(2)	6(1)	2(2)
C(32)	23(2)	29(2)	28(2)	1(1)	5(1)	8(1)
C(33)	25(2)	21(2)	20(2)	1(1)	6(1)	-1(1)
C(34)	14(1)	17(2)	17(2)	0(1)	4(1)	4(1)
C(35)	22(2)	18(2)	17(2)	2(1)	4(1)	3(1)
C(36)	25(2)	16(2)	24(2)	3(1)	7(1)	0(1)
C(37)	19(2)	14(2)	26(2)	-5(1)	5(1)	1(1)

C(38)	17(2)	23(2)	13(1)	-2(1)	3(1)	4(1)
C(39)	17(2)	16(2)	17(2)	1(1)	6(1)	2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for nel06.

	x	y	z	U(eq)
H(3)	718	6469	1505	24
H(4)	-802	5364	1580	21
H(5)	-1485	4225	697	21
H(7A)	1262	9716	8	50
H(7B)	2049	8873	-238	50
H(7C)	2463	9392	517	50
H(8A)	328	8224	1307	40
H(8B)	247	9327	973	40
H(8C)	1427	8956	1484	40
H(9A)	-309	7495	142	52
H(9B)	463	7593	-430	52
H(9C)	-315	8519	-288	52
H(10)	3260	8459	1344	29
H(11A)	2981	7041	2059	34
H(11B)	4325	7149	2047	34
H(12A)	4321	5657	1536	35
H(12B)	3036	5488	1651	35
H(13)	2930	5325	439	30
H(14A)	3442	6740	-213	31
H(14B)	4564	6268	305	31
H(15A)	3968	8203	319	32
H(15B)	4888	7669	945	32
H(17)	8194	7962	1352	24
H(18)	7444	9167	527	29
H(19)	5956	10231	695	34
H(20)	5252	10057	1723	31
H(21)	5981	8811	2533	26
H(23)	5626	6665	3043	21
H(24)	5012	6915	4072	25
H(25)	6140	7864	4988	26
H(26)	7869	8570	4839	26
H(27)	8490	8306	3806	23
H(29)	9399	8648	2675	26
H(30)	11402	8536	2984	31
H(31)	12309	6982	3268	34
H(32)	11168	5528	3219	32
H(33)	9169	5652	2908	26
H(35)	7031	5354	3039	23
H(36)	6306	3875	2484	25
H(37)	5907	3744	1259	24
H(38)	6251	5139	601	22
H(39)	6917	6628	1156	20

Table 6. Torsion angles [°] for nel06.

C(2)-N(1)-N(2)-C(10)	140.8(3)	C(22)-C(23)-C(24)-C(25)	1.3(4)
C(13)-N(1)-N(2)-C(10)	-4.3(3)	C(23)-C(24)-C(25)-C(26)	0.7(4)
C(2)-N(1)-N(2)-C(6)	-57.8(4)	C(24)-C(25)-C(26)-C(27)	-1.0(4)
C(13)-N(1)-N(2)-C(6)	157.1(3)	C(25)-C(26)-C(27)-C(22)	-0.7(5)
C(5)#1-C(1)-C(2)-C(3)	178.2(3)	C(23)-C(22)-C(27)-C(26)	2.5(4)
C(1)#1-C(1)-C(2)-C(3)	-2.3(5)	B(1)-C(22)-C(27)-C(26)	178.9(3)
C(5)#1-C(1)-C(2)-N(1)	-7.6(4)	C(16)-B(1)-C(28)-C(29)	31.1(4)
C(1)#1-C(1)-C(2)-N(1)	171.8(3)	C(22)-B(1)-C(28)-C(29)	-88.7(3)
N(2)-N(1)-C(2)-C(3)	-42.2(4)	C(34)-B(1)-C(28)-C(29)	150.4(3)
C(13)-N(1)-C(2)-C(3)	101.1(3)	C(16)-B(1)-C(28)-C(33)	-152.0(3)
N(2)-N(1)-C(2)-C(1)	143.6(3)	C(22)-B(1)-C(28)-C(33)	88.2(3)
C(13)-N(1)-C(2)-C(1)	-73.1(3)	C(34)-B(1)-C(28)-C(33)	-32.6(3)
C(1)-C(2)-C(3)-C(4)	1.8(5)	C(33)-C(28)-C(29)-C(30)	0.0(4)
N(1)-C(2)-C(3)-C(4)	-172.1(3)	B(1)-C(28)-C(29)-C(30)	177.1(3)
C(2)-C(3)-C(4)-C(5)	0.5(4)	C(28)-C(29)-C(30)-C(31)	-0.2(5)
C(3)-C(4)-C(5)-C(1)#1	-2.3(4)	C(29)-C(30)-C(31)-C(32)	0.4(5)
N(1)-N(2)-C(6)-C(9)	1.1(4)	C(30)-C(31)-C(32)-C(33)	-0.4(5)
C(10)-N(2)-C(6)-C(9)	161.3(3)	C(31)-C(32)-C(33)-C(28)	0.2(5)
N(1)-N(2)-C(6)-C(8)	123.3(3)	C(29)-C(28)-C(33)-C(32)	0.1(4)
C(10)-N(2)-C(6)-C(8)	-76.6(3)	B(1)-C(28)-C(33)-C(32)	-177.1(3)
N(1)-N(2)-C(6)-C(7)	-117.9(3)	C(16)-B(1)-C(34)-C(39)	29.2(4)
C(10)-N(2)-C(6)-C(7)	42.3(4)	C(22)-B(1)-C(34)-C(39)	149.1(3)
N(1)-N(2)-C(10)-C(11)	-57.8(3)	C(28)-B(1)-C(34)-C(39)	-91.3(3)
C(6)-N(2)-C(10)-C(11)	140.5(3)	C(16)-B(1)-C(34)-C(35)	-154.0(3)
N(1)-N(2)-C(10)-C(15)	60.5(3)	C(22)-B(1)-C(34)-C(35)	-34.1(4)
C(6)-N(2)-C(10)-C(15)	-101.1(3)	C(28)-B(1)-C(34)-C(35)	85.6(3)
N(2)-C(10)-C(11)-C(12)	62.4(3)	C(39)-C(34)-C(35)-C(36)	-1.3(4)
C(15)-C(10)-C(11)-C(12)	-56.8(3)	B(1)-C(34)-C(35)-C(36)	-178.4(3)
C(10)-C(11)-C(12)-C(13)	-7.6(4)	C(34)-C(35)-C(36)-C(37)	1.3(5)
N(2)-N(1)-C(13)-C(12)	62.3(3)	C(35)-C(36)-C(37)-C(38)	0.0(4)
C(2)-N(1)-C(13)-C(12)	-84.1(3)	C(36)-C(37)-C(38)-C(39)	-1.1(4)
N(2)-N(1)-C(13)-C(14)	-58.2(3)	C(37)-C(38)-C(39)-C(34)	1.0(4)
C(2)-N(1)-C(13)-C(14)	155.4(2)	C(35)-C(34)-C(39)-C(38)	0.2(4)
C(11)-C(12)-C(13)-N(1)	-52.2(3)	B(1)-C(34)-C(39)-C(38)	177.3(3)
C(11)-C(12)-C(13)-C(14)	64.3(3)	B(1)-C(16)-C(21)-C(20)	-174.0(3)
N(1)-C(13)-C(14)-C(15)	65.9(3)	C(16)-B(1)-C(22)-C(23)	91.9(3)
C(12)-C(13)-C(14)-C(15)	-52.1(3)	C(28)-B(1)-C(22)-C(23)	-147.2(3)
C(13)-C(14)-C(15)-C(10)	-13.0(3)	C(34)-B(1)-C(22)-C(23)	-28.1(4)
N(2)-C(10)-C(15)-C(14)	-47.5(3)	C(16)-B(1)-C(22)-C(27)	-84.3(3)
C(11)-C(10)-C(15)-C(14)	68.9(3)	C(28)-B(1)-C(22)-C(27)	36.6(4)
C(22)-B(1)-C(16)-C(17)	167.5(3)	C(34)-B(1)-C(22)-C(27)	155.8(3)
C(28)-B(1)-C(16)-C(17)	47.6(3)	C(19)-C(20)-C(21)-C(16)	0.0(5)
C(34)-B(1)-C(16)-C(17)	-71.3(3)		
C(22)-B(1)-C(16)-C(21)	-16.6(4)		
C(28)-B(1)-C(16)-C(21)	-136.5(3)		
C(34)-B(1)-C(16)-C(21)	104.6(3)		
C(21)-C(16)-C(17)-C(18)	-3.1(4)		
B(1)-C(16)-C(17)-C(18)	173.1(3)		
C(16)-C(17)-C(18)-C(19)	1.9(5)		
C(17)-C(18)-C(19)-C(20)	0.5(5)		
C(18)-C(19)-C(20)-C(21)	-1.4(5)		
C(17)-C(16)-C(21)-C(20)	2.2(4)		
C(27)-C(22)-C(23)-C(24)	-2.8(4)		
B(1)-C(22)-C(23)-C(24)	-179.2(3)		

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z

$Hy_2{}^{27}NA^{2+}(BF_4^-)_2$ (*00069*)

A dark red prism-shaped crystal of dimensions 0.36 x 0.26 x 0.24 mm was selected for structural analysis. Intensity data for this compound were collected using a Bruker SMART ccd area detector[17] mounted on a Bruker P4 goniometer using graphite-monochromated Mo K_α radiation (λ = 0.71073 Å). The sample was cooled to 133(2) K. The intensity data, which nominally covered one and a half hemispheres of reciprocal space, were measured as a series of ω oscillation frames each of 0.4 ° for 20 sec / frame. The detector was operated in 512 x 512 mode and was positioned 5.00 cm from the sample. Coverage of unique data was 99.4 % complete to 25.00 degrees in ω. Cell parameters were determined from a non-linear least squares fit of 7318 peaks in the range $2.66 < \theta < 28.86°$. The first 50 frames were repeated at the end of data collection and yielded 352 peaks showing a variation of 0.14 % during the data collection. A total of 9264 data were measured in the range $1.81 < \theta < 29.13°$. The data were corrected for absorption by the empirical method[18] giving minimum and maximum transmission factors of 0.6107 and 0.7118. The data were merged to form a set of 5611 independent data with R(int) = 0.0187.

The monoclinic space group Cc was determined by systematic absences and statistical tests and verified by subsequent refinement. The structure was solved by direct methods and refined by full-matrix least-squares methods on F^2.[19] Hydrogen atom positions were initially determined by geometry and were refined using a riding model. Non-hydrogen atoms were refined with anisotropic displacement parameters. A total of 589 parameters were refined against 1605 restraints and 5611 data to give $wR(F^2) = 0.0488$ and S = 0.968 for weights of $w = 1/[\sigma^2 (F^2) + (0.0240\ P)^2]$, where $P = [F_o^2 + 2F_c^2] / 3$. The final R(F) was 0.0236 for the 5170 observed, [$F > 4\sigma(F)$], data. The largest shift/s.u. was 0.002 in the final refinement cycle. The final difference map had maxima and minima of 0.401 and -0.343 e/Å^3, respectively. The absolute structure was determined by refinement of the Flack parameter.[20] The polar axis restraints were taken from Flack and Schwarzenbach.[21]

Comment

The B (Sb F6)- group is disordered and is modeled in three orientations with occupancies of 0.623(4), 0.285(4), and 0.092(2) for the unprimed, primed, and double primed atoms. Restraints on the positional and displacement parameters of the disordered anion were required for the refinement to achieve convergence. This structure was determined by Douglas R. Powell.

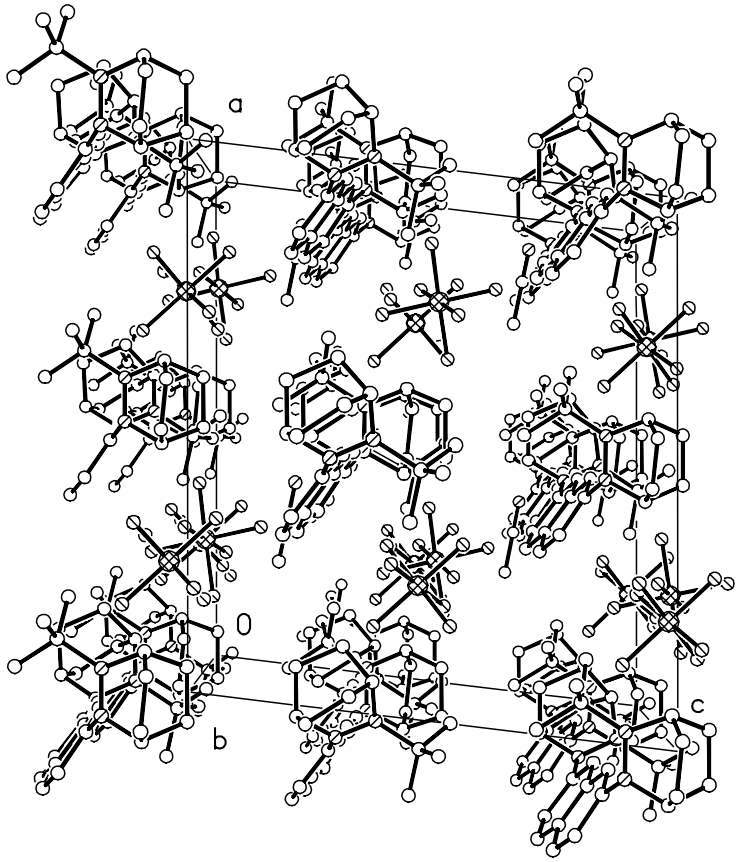


Table 1. Crystal data and structure refinement for 00069.

Identification code	00069
Empirical formula	(C30 H44 N4)2+ 2(Sb F6)- (C2 H3 N)
	C32 H47 F12 N5 Sb2
Formula weight	973.25

Crystal system	Monoclinic	
Space group	Cc	
Unit cell dimensions	*a* = 16.6690(9) Å	α= 90°
	b = 15.3167(8) Å	β= 96.964(2)°
	c = 14.9090(7) Å	γ= 90°
Volume	3778.4(3) Å^3	
Z	4	
Density (calculated)	1.711 Mg/m^3	
Wavelength	0.71073 Å	
Temperature	133(2) K	
F(000)	1936	
Absorption coefficient	1.520 mm^{-1}	
Absorption correction	Empirical	
Max. and min. transmission	0.7118 and 0.6107	
Theta range for data collection	1.81 to 29.13°.	
Reflections collected	9264	
Independent reflections	5611 [R(int) = 0.0187]	
Data / restraints / parameters	5611 / 1605 / 589	
wR(F^2 all data)	wR = 0.0488	
R(*F* obsd data)	R = 0.0236	
Goodness-of-fit on F^2	0.968	
Observed data [I > 2□(I)]	5170	
Absolute structure parameter	0.005(15)	
Largest and mean shift / s.u.	0.002and 0.000	
Largest diff. peak and hole	0.401 and -0.343 e/Å^3	

Table 2. Atomic coordinates and equivalent isotropic displacement parameters for 00069. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Sb(1A)	0.26730(5)	0.325689(19)	0.51653(4)	0.02347(7)
F(1A)	0.22979(15)	0.35635(17)	0.39740(12)	0.0442(7)
F(2A)	0.30427(14)	0.29622(15)	0.63667(11)	0.0342(6)
F(3A)	0.24272(14)	0.43843(11)	0.55452(16)	0.0364(7)

F(4A)	0.29019(15)	0.21275(11)	0.47931(17)	0.0453(8)
F(5A)	0.16313(11)	0.28909(15)	0.53506(16)	0.0382(7)
F(6A)	0.37185(11)	0.36197(16)	0.50098(17)	0.0435(7)
Sb(1B)	0.74803(13)	0.37210(14)	0.48881(15)	0.0320(4)
F(1B)	0.7336(3)	0.25333(18)	0.5130(3)	0.0435(15)
F(2B)	0.7617(3)	0.49068(18)	0.4647(3)	0.062(2)
F(3B)	0.6986(3)	0.4011(3)	0.5912(3)	0.080(2)
F(4B)	0.7974(3)	0.3422(3)	0.3868(3)	0.058(2)
F(5B)	0.84877(19)	0.3699(3)	0.5597(3)	0.0402(15)
F(6B)	0.6470(2)	0.3740(3)	0.4182(3)	0.066(2)
Sb(1')	0.7375(3)	0.3863(3)	0.4711(3)	0.0338(9)
F(1')	0.7001(6)	0.2871(5)	0.5268(6)	0.062(3)
F(2')	0.7748(6)	0.4853(5)	0.4151(6)	0.052(3)
F(3')	0.6734(5)	0.4574(6)	0.5358(6)	0.075(3)
F(4')	0.8015(6)	0.3152(6)	0.4061(6)	0.054(4)
F(5')	0.8230(5)	0.3962(6)	0.5644(5)	0.049(3)
F(6')	0.6518(5)	0.3763(6)	0.3778(5)	0.035(3)
Sb(1'')	0.7126(4)	0.3885(5)	0.4905(6)	0.0403(16)
F(1'')	0.7373(13)	0.2693(6)	0.4873(17)	0.048(5)
F(2'')	0.6878(13)	0.5077(6)	0.4936(17)	0.069(6)
F(3'')	0.7403(13)	0.3890(16)	0.6160(6)	0.061(6)
F(4'')	0.6849(13)	0.3881(16)	0.3649(6)	0.053(6)
F(5'')	0.8195(6)	0.4160(14)	0.4748(15)	0.053(4)
F(6'')	0.6056(6)	0.3611(15)	0.5060(16)	0.078(7)
C(1)	0.5945(2)	0.0733(3)	0.4477(3)	0.0260(10)
N(2)	0.5553(2)	0.1119(2)	0.3626(2)	0.0181(7)
N(3)	0.4744(2)	0.1201(2)	0.3614(2)	0.0188(7)
C(4)	0.4431(3)	0.0813(3)	0.4413(3)	0.0242(10)
C(5)	0.4685(3)	-0.0142(3)	0.4475(3)	0.0298(10)
C(6)	0.5612(3)	-0.0191(3)	0.4584(4)	0.0340(11)
C(7)	0.5735(3)	0.1311(4)	0.5242(3)	0.0328(12)
C(8)	0.4810(3)	0.1324(3)	0.5243(3)	0.0322(11)
C(9)	0.5959(3)	0.1142(3)	0.2778(3)	0.0255(10)
C(10)	0.5387(3)	0.1380(3)	0.1936(3)	0.0308(11)
C(11)	0.6654(3)	0.1802(3)	0.2918(4)	0.0361(14)
C(12)	0.6289(3)	0.0219(3)	0.2633(4)	0.0363(13)
C(13)	0.4721(2)	0.2774(3)	0.3413(3)	0.0197(9)
C(14)	0.4361(2)	0.1979(3)	0.3237(3)	0.0199(9)
C(15)	0.3606(2)	0.1898(3)	0.2702(3)	0.0235(9)
C(16)	0.3229(2)	0.2621(3)	0.2323(3)	0.0243(9)
C(17)	0.3204(2)	0.4227(3)	0.2098(3)	0.0235(9)
C(18)	0.3534(3)	0.5030(3)	0.2287(3)	0.0244(10)
C(19)	0.4265(3)	0.5100(3)	0.2867(3)	0.0229(9)
C(20)	0.4665(2)	0.4374(3)	0.3236(3)	0.0204(9)
C(21)	0.4326(2)	0.3540(3)	0.3050(3)	0.0191(9)
C(22)	0.3581(3)	0.3462(3)	0.2480(3)	0.0208(10)
C(23)	0.5712(3)	0.6704(3)	0.3776(3)	0.0353(11)
N(24)	0.4987(2)	0.6164(2)	0.3832(3)	0.0272(9)
N(25)	0.4595(2)	0.5956(2)	0.3027(3)	0.0261(8)
C(26)	0.4917(3)	0.6405(3)	0.2262(3)	0.0431(14)
C(27)	0.5776(4)	0.6105(4)	0.2240(4)	0.0551(18)
C(28)	0.6241(3)	0.6185(4)	0.3185(4)	0.0423(13)
C(29)	0.5478(4)	0.7574(3)	0.3326(4)	0.0520(16)

C(30)	0.4892(4)	0.7387(4)	0.2465(5)	0.070(2)
C(31)	0.4578(3)	0.6126(3)	0.4702(3)	0.0368(12)
C(32)	0.5051(4)	0.5511(5)	0.5347(4)	0.0597(18)
C(33)	0.3704(3)	0.5831(4)	0.4501(4)	0.0454(14)
C(34)	0.4582(4)	0.7052(4)	0.5083(4)	0.0569(17)
C(1S)	0.7519(4)	0.4073(3)	0.1829(4)	0.0526(15)
C(2S)	0.8359(4)	0.4312(4)	0.2032(4)	0.0462(14)
N(3S)	0.9013(4)	0.4503(4)	0.2184(4)	0.0633(16)

Table 3. Bond lengths [Å] and angles [°] for 00069.

Sb(1A)-F(1A)	1.8700(15)	C(5)-C(6)	1.535(6)
Sb(1A)-F(4A)	1.8701(15)	C(7)-C(8)	1.542(7)
Sb(1A)-F(6A)	1.8702(15)	C(9)-C(10)	1.525(6)
Sb(1A)-F(5A)	1.8767(15)	C(9)-C(11)	1.532(6)
Sb(1A)-F(2A)	1.8773(15)	C(9)-C(12)	1.542(6)
Sb(1A)-F(3A)	1.8780(15)	C(13)-C(14)	1.369(6)
Sb(1B)-F(2B)	1.8710(18)	C(13)-C(21)	1.420(6)
Sb(1B)-F(4B)	1.8717(18)	C(14)-C(15)	1.411(5)
Sb(1B)-F(5B)	1.8728(18)	C(15)-C(16)	1.363(6)
Sb(1B)-F(3B)	1.8741(18)	C(16)-C(22)	1.423(6)
Sb(1B)-F(6B)	1.8752(18)	C(17)-C(18)	1.364(6)
Sb(1B)-F(1B)	1.8757(18)	C(17)-C(22)	1.416(6)
Sb(1')-F(2')	1.8728(19)	C(18)-C(19)	1.410(5)
Sb(1')-F(3')	1.8730(19)	C(19)-C(20)	1.376(6)
Sb(1')-F(5')	1.8732(19)	C(19)-N(25)	1.430(5)
Sb(1')-F(4')	1.8733(19)	C(20)-C(21)	1.411(6)
Sb(1')-F(1')	1.8738(19)	C(21)-C(22)	1.422(6)
Sb(1')-F(6')	1.8748(19)	C(23)-N(24)	1.475(6)
Sb(1")-F(3")	1.8731(19)	C(23)-C(29)	1.521(7)
Sb(1")-F(5")	1.8734(19)	C(23)-C(28)	1.541(7)
Sb(1")-F(6")	1.8735(19)	N(24)-N(25)	1.334(5)
Sb(1")-F(2")	1.8735(19)	N(24)-C(31)	1.538(6)
Sb(1")-F(1")	1.8736(19)	N(25)-C(26)	1.487(6)
Sb(1")-F(4")	1.8737(19)	C(26)-C(27)	1.507(8)
C(1)-N(2)	1.478(5)	C(26)-C(30)	1.536(8)
C(1)-C(7)	1.518(7)	C(27)-C(28)	1.528(8)
C(1)-C(6)	1.537(6)	C(29)-C(30)	1.542(8)
N(2)-N(3)	1.353(4)	C(31)-C(32)	1.499(8)
N(2)-C(9)	1.505(6)	C(31)-C(33)	1.519(7)
N(3)-C(14)	1.434(5)	C(31)-C(34)	1.529(7)
N(3)-C(4)	1.481(5)	C(1S)-C(2S)	1.443(8)
C(4)-C(5)	1.523(6)	C(2S)-N(3S)	1.125(7)
C(4)-C(8)	1.534(6)		

F(1A)-Sb(1A)-F(4A)	90.41(10)
F(1A)-Sb(1A)-F(6A)	90.97(10)
F(4A)-Sb(1A)-F(6A)	90.65(10)
F(1A)-Sb(1A)-F(5A)	90.34(10)
F(4A)-Sb(1A)-F(5A)	89.60(9)
F(6A)-Sb(1A)-F(5A)	178.66(11)
F(1A)-Sb(1A)-F(2A)	179.24(12)
F(4A)-Sb(1A)-F(2A)	90.32(10)
F(6A)-Sb(1A)-F(2A)	89.23(9)
F(5A)-Sb(1A)-F(2A)	89.45(9)
F(1A)-Sb(1A)-F(3A)	89.74(10)
F(4A)-Sb(1A)-F(3A)	179.06(11)
F(6A)-Sb(1A)-F(3A)	90.27(10)
F(5A)-Sb(1A)-F(3A)	89.47(9)
F(2A)-Sb(1A)-F(3A)	89.53(9)
F(2B)-Sb(1B)-F(4B)	90.32(13)
F(2B)-Sb(1B)-F(5B)	90.21(12)
F(4B)-Sb(1B)-F(5B)	90.10(13)
F(2B)-Sb(1B)-F(3B)	90.12(13)
F(4B)-Sb(1B)-F(3B)	179.55(16)
F(5B)-Sb(1B)-F(3B)	89.83(13)
F(2B)-Sb(1B)-F(6B)	89.95(13)
F(4B)-Sb(1B)-F(6B)	90.06(13)
F(5B)-Sb(1B)-F(6B)	179.77(17)
F(3B)-Sb(1B)-F(6B)	90.01(13)
F(2B)-Sb(1B)-F(1B)	179.63(16)
F(4B)-Sb(1B)-F(1B)	89.89(13)
F(5B)-Sb(1B)-F(1B)	90.09(13)
F(3B)-Sb(1B)-F(1B)	89.67(13)
F(6B)-Sb(1B)-F(1B)	89.74(13)
F(2')-Sb(1')-F(3')	90.04(14)
F(2')-Sb(1')-F(5')	90.07(14)
F(3')-Sb(1')-F(5')	90.04(14)
F(2')-Sb(1')-F(4')	89.96(14)
F(3')-Sb(1')-F(4')	179.9(2)
F(5')-Sb(1')-F(4')	90.08(14)
F(2')-Sb(1')-F(1')	179.84(19)
F(3')-Sb(1')-F(1')	90.08(14)
F(5')-Sb(1')-F(1')	90.03(14)
F(4')-Sb(1')-F(1')	89.93(14)
F(2')-Sb(1')-F(6')	89.97(14)
F(3')-Sb(1')-F(6')	89.92(14)
F(5')-Sb(1')-F(6')	179.9(2)
F(4')-Sb(1')-F(6')	89.96(14)
F(1')-Sb(1')-F(6')	89.92(14)
F(3")-Sb(1")-F(5")	90.04(14)
F(3")-Sb(1")-F(6")	90.01(14)
F(5")-Sb(1")-F(6")	179.9(2)
F(3")-Sb(1")-F(2")	90.00(14)
F(5")-Sb(1")-F(2")	90.00(14)
F(6")-Sb(1")-F(2")	89.99(14)
F(3")-Sb(1")-F(1")	90.02(14)
F(5")-Sb(1")-F(1")	90.02(14)

F(6")-Sb(1")-F(1")	90.00(14)
F(2")-Sb(1")-F(1")	180.0(2)
F(3")-Sb(1")-F(4")	180.0(2)
F(5")-Sb(1")-F(4")	89.99(14)
F(6")-Sb(1")-F(4")	89.96(14)
F(2")-Sb(1")-F(4")	90.01(14)
F(1")-Sb(1")-F(4")	89.98(14)
N(2)-C(1)-C(7)	107.1(4)
N(2)-C(1)-C(6)	109.2(4)
C(7)-C(1)-C(6)	109.8(4)
N(3)-N(2)-C(1)	112.7(3)
N(3)-N(2)-C(9)	122.4(3)
C(1)-N(2)-C(9)	122.2(3)
N(2)-N(3)-C(14)	118.5(3)
N(2)-N(3)-C(4)	113.5(3)
C(14)-N(3)-C(4)	117.7(3)
N(3)-C(4)-C(5)	108.1(4)
N(3)-C(4)-C(8)	107.1(4)
C(5)-C(4)-C(8)	110.9(4)
C(4)-C(5)-C(6)	108.8(3)
C(5)-C(6)-C(1)	108.4(3)
C(1)-C(7)-C(8)	109.3(4)
C(4)-C(8)-C(7)	107.8(4)
N(2)-C(9)-C(10)	113.4(3)
N(2)-C(9)-C(11)	108.1(4)
C(10)-C(9)-C(11)	110.0(4)
N(2)-C(9)-C(12)	107.5(4)
C(10)-C(9)-C(12)	107.6(4)
C(11)-C(9)-C(12)	110.3(4)
C(14)-C(13)-C(21)	119.3(4)
C(13)-C(14)-C(15)	121.8(4)
C(13)-C(14)-N(3)	120.0(3)
C(15)-C(14)-N(3)	118.1(3)
C(16)-C(15)-C(14)	119.9(4)
C(15)-C(16)-C(22)	120.5(4)
C(18)-C(17)-C(22)	121.0(4)
C(17)-C(18)-C(19)	119.5(4)
C(20)-C(19)-C(18)	121.6(4)
C(20)-C(19)-N(25)	121.1(4)
C(18)-C(19)-N(25)	117.2(4)
C(19)-C(20)-C(21)	119.4(4)
C(20)-C(21)-C(13)	121.1(4)
C(20)-C(21)-C(22)	119.5(4)
C(13)-C(21)-C(22)	119.4(4)
C(17)-C(22)-C(21)	119.0(4)
C(17)-C(22)-C(16)	121.9(4)
C(21)-C(22)-C(16)	119.1(4)
N(24)-C(23)-C(29)	110.4(4)
N(24)-C(23)-C(28)	105.8(4)
C(29)-C(23)-C(28)	109.5(5)
N(25)-N(24)-C(23)	113.5(4)
N(25)-N(24)-C(31)	122.0(4)
C(23)-N(24)-C(31)	121.3(4)

N(24)-N(25)-C(19)	120.4(3)
N(24)-N(25)-C(26)	113.3(4)
C(19)-N(25)-C(26)	117.6(4)
N(25)-C(26)-C(27)	107.9(4)
N(25)-C(26)-C(30)	106.3(5)
C(27)-C(26)-C(30)	110.5(5)
C(26)-C(27)-C(28)	109.5(5)
C(27)-C(28)-C(23)	107.9(4)
C(23)-C(29)-C(30)	107.9(4)
C(26)-C(30)-C(29)	108.5(4)
C(32)-C(31)-C(33)	111.0(5)
C(32)-C(31)-C(34)	111.5(5)
C(33)-C(31)-C(34)	108.1(4)
C(32)-C(31)-N(24)	108.2(4)
C(33)-C(31)-N(24)	110.9(4)
C(34)-C(31)-N(24)	107.1(4)
N(3S)-C(2S)-C(1S)	179.5(8)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 00069. The anisotropic displacement factor exponent takes the form:

$-2\ \pi^2[\ h^2\ a^{*2}\ U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sb(1A)	19(1)	29(1)	23(1)	-5(1)	3(1)	0(1)
F(1A)	42(2)	65(2)	24(2)	1(2)	-3(1)	8(2)
F(2A)	36(2)	37(2)	29(2)	1(1)	2(1)	-1(1)
F(3A)	33(2)	29(2)	44(2)	-12(1)	-6(1)	6(1)
F(4A)	46(2)	37(2)	53(2)	-18(2)	8(2)	8(1)
F(5A)	25(1)	49(2)	42(2)	-16(1)	8(1)	-10(1)
F(6A)	23(1)	58(2)	52(2)	7(2)	11(1)	-6(1)
Sb(1B)	22(1)	32(1)	41(1)	2(1)	1(1)	-9(1)
F(1B)	48(3)	34(3)	50(3)	7(2)	12(3)	-11(2)
F(2B)	58(4)	31(3)	91(5)	17(3)	-16(4)	-10(3)
F(3B)	75(4)	72(4)	102(5)	-23(4)	55(4)	-10(4)
F(4B)	66(4)	81(5)	26(3)	6(3)	3(3)	-11(3)
F(5B)	35(3)	45(3)	37(3)	3(2)	-7(2)	-12(3)
F(6B)	31(3)	59(4)	102(6)	-2(4)	-21(3)	-7(3)
Sb(1')	26(2)	33(2)	43(2)	-1(1)	4(1)	-6(1)
F(1')	59(6)	53(6)	72(6)	23(5)	-1(5)	-21(5)
F(2')	40(6)	50(6)	65(8)	25(6)	-3(6)	-14(5)
F(3')	66(6)	79(7)	87(8)	-34(6)	33(6)	1(6)
F(4')	43(6)	66(8)	50(8)	-9(7)	-4(6)	29(6)
F(5')	45(6)	56(6)	43(6)	11(5)	-11(5)	-13(5)
F(6')	15(5)	40(6)	49(7)	-11(5)	1(5)	-8(4)
Sb(1")	31(3)	35(3)	57(3)	2(2)	12(3)	0(2)
F(1")	51(8)	38(8)	53(9)	11(8)	0(8)	-13(8)
F(2")	71(10)	50(11)	84(11)	-10(10)	4(10)	-11(10)
F(3")	60(11)	57(10)	67(10)	8(10)	6(10)	0(10)
F(4")	39(11)	43(10)	74(11)	-5(10)	1(10)	-13(10)

F(5")	46(6)	48(6)	62(6)	8(6)	-2(6)	-12(6)
F(6")	40(12)	78(13)	110(15)	-21(14)	-18(13)	7(12)
C(1)	18(2)	33(2)	27(2)	10(2)	2(2)	1(2)
N(2)	17(2)	18(2)	20(2)	3(1)	5(1)	2(1)
N(3)	17(2)	21(2)	19(2)	-1(1)	3(1)	1(1)
C(4)	20(2)	27(2)	27(2)	6(2)	8(2)	1(2)
C(5)	30(2)	20(2)	39(3)	12(2)	4(2)	1(2)
C(6)	32(2)	30(3)	42(3)	15(2)	12(2)	6(2)
C(7)	30(3)	45(3)	22(2)	3(2)	1(2)	-8(2)
C(8)	34(3)	41(3)	22(2)	-1(2)	8(2)	0(2)
C(9)	22(2)	26(3)	30(3)	-2(2)	10(2)	4(2)
C(10)	38(3)	36(3)	20(2)	0(2)	10(2)	5(2)
C(11)	33(3)	43(3)	35(3)	-4(3)	14(2)	-11(2)
C(12)	44(3)	30(3)	38(3)	-2(2)	18(3)	13(2)
C(13)	16(2)	21(2)	21(2)	0(2)	0(2)	4(2)
C(14)	20(2)	19(2)	21(2)	-2(2)	2(2)	3(2)
C(15)	23(2)	20(2)	26(2)	-3(2)	-1(2)	-1(2)
C(16)	21(2)	23(2)	28(2)	-2(2)	-4(2)	0(2)
C(17)	18(2)	28(2)	23(2)	-1(2)	-2(2)	2(2)
C(18)	24(2)	20(2)	28(2)	2(2)	-2(2)	7(2)
C(19)	25(2)	16(2)	27(2)	-2(2)	-2(2)	-1(2)
C(20)	19(2)	19(2)	22(2)	-1(2)	-1(2)	2(2)
C(21)	17(2)	22(2)	19(2)	2(2)	3(2)	2(2)
C(22)	21(2)	20(2)	21(2)	-2(2)	-1(2)	-1(2)
C(23)	32(2)	32(3)	39(3)	-2(2)	-7(2)	-7(2)
N(24)	31(2)	21(2)	27(2)	-5(2)	-7(2)	-2(2)
N(25)	31(2)	15(2)	29(2)	0(2)	-8(2)	2(2)
C(26)	56(3)	43(3)	26(3)	17(2)	-8(2)	-24(3)
C(27)	53(4)	69(5)	45(4)	-7(3)	17(3)	-22(3)
C(28)	35(3)	47(3)	44(3)	2(3)	6(2)	-12(2)
C(29)	60(4)	30(3)	60(4)	5(3)	-16(3)	-14(3)
C(30)	92(5)	39(4)	68(5)	24(3)	-43(4)	-27(3)
C(31)	38(3)	42(3)	31(3)	-11(2)	2(2)	0(2)
C(32)	59(4)	90(5)	29(3)	4(3)	0(3)	6(4)
C(33)	37(3)	61(4)	40(3)	-13(3)	15(2)	-2(3)
C(34)	62(4)	55(4)	56(4)	-31(3)	14(3)	-13(3)
C(1S)	64(4)	41(3)	54(4)	3(3)	9(3)	0(3)
C(2S)	69(4)	31(3)	42(3)	-2(2)	21(3)	-4(3)
N(3S)	69(4)	65(4)	61(4)	-20(3)	27(3)	-14(3)

Table 5. Hydrogen coordinates and isotropic displacement parameters for 00069.

	x	y	z	U(eq)
H(1)	0.6544	0.0712	0.4472	0.031
H(4)	0.3829	0.0861	0.4352	0.029
H(5A)	0.4460	-0.0456	0.3920	0.036
H(5B)	0.4476	-0.0421	0.4999	0.036
H(6A)	0.5792	-0.0584	0.4120	0.041
H(6B)	0.5814	-0.0427	0.5188	0.041
H(7A)	0.5934	0.1911	0.5161	0.039

H(7B)	0.5997	0.1084	0.5826	0.039
H(8A)	0.4663	0.1050	0.5803	0.039
H(8B)	0.4609	0.1933	0.5217	0.039
H(10A)	0.4963	0.0936	0.1830	0.046
H(10B)	0.5691	0.1408	0.1413	0.046
H(10C)	0.5140	0.1949	0.2024	0.046
H(11A)	0.7017	0.1646	0.3462	0.054
H(11B)	0.6435	0.2388	0.2989	0.054
H(11C)	0.6955	0.1792	0.2393	0.054
H(12A)	0.6655	0.0045	0.3168	0.054
H(12B)	0.6585	0.0221	0.2103	0.054
H(12C)	0.5839	-0.0194	0.2536	0.054
H(13)	0.5230	0.2813	0.3774	0.024
H(15)	0.3362	0.1340	0.2607	0.028
H(16)	0.2727	0.2564	0.1951	0.029
H(17)	0.2713	0.4180	0.1704	0.028
H(18)	0.3274	0.5539	0.2029	0.029
H(20)	0.5165	0.4434	0.3611	0.025
H(23)	0.6009	0.6800	0.4392	0.042
H(26)	0.4580	0.6267	0.1678	0.052
H(27A)	0.6038	0.6468	0.1810	0.066
H(27B)	0.5780	0.5491	0.2035	0.066
H(28A)	0.6759	0.6492	0.3155	0.051
H(28B)	0.6359	0.5598	0.3446	0.051
H(29A)	0.5966	0.7878	0.3167	0.062
H(29B)	0.5213	0.7951	0.3742	0.062
H(30A)	0.4337	0.7563	0.2558	0.085
H(30B)	0.5055	0.7724	0.1951	0.085
H(32A)	0.5034	0.4924	0.5086	0.089
H(32B)	0.4816	0.5500	0.5919	0.089
H(32C)	0.5614	0.5709	0.5459	0.089
H(33A)	0.3687	0.5240	0.4246	0.068
H(33B)	0.3412	0.6232	0.4065	0.068
H(33C)	0.3451	0.5831	0.5060	0.068
H(34A)	0.4252	0.7432	0.4657	0.085
H(34B)	0.5138	0.7272	0.5175	0.085
H(34C)	0.4359	0.7047	0.5662	0.085
H(1SA)	0.7364	0.3693	0.2308	0.079
H(1SB)	0.7439	0.3763	0.1250	0.079
H(1SC)	0.7185	0.4601	0.1792	0.079

Table 6. Torsion angles [°] for 00069.

C(7)-C(1)-N(2)-N(3)	-57.1(4)	C(19)-C(20)-C(21)-C(22)	0.5(6)
C(6)-C(1)-N(2)-N(3)	61.7(4)	C(14)-C(13)-C(21)-C(20)	178.2(4)
C(7)-C(1)-N(2)-C(9)	141.1(4)	C(14)-C(13)-C(21)-C(22)	-2.6(6)
C(6)-C(1)-N(2)-C(9)	-100.1(4)	C(18)-C(17)-C(22)-C(21)	-1.6(7)
C(1)-N(2)-N(3)-C(14)	139.2(4)	C(18)-C(17)-C(22)-C(16)	177.3(4)
C(9)-N(2)-N(3)-C(14)	-59.1(5)	C(20)-C(21)-C(22)-C(17)	1.2(7)
C(1)-N(2)-N(3)-C(4)	-5.4(5)	C(13)-C(21)-C(22)-C(17)	-178.0(4)
C(9)-N(2)-N(3)-C(4)	156.4(3)	C(20)-C(21)-C(22)-C(16)	-177.7(4)
N(2)-N(3)-C(4)-C(5)	-56.2(4)	C(13)-C(21)-C(22)-C(16)	3.1(7)
C(14)-N(3)-C(4)-C(5)	158.9(3)	C(15)-C(16)-C(22)-C(17)	-179.9(4)
N(2)-N(3)-C(4)-C(8)	63.4(4)	C(15)-C(16)-C(22)-C(21)	-1.0(7)
C(14)-N(3)-C(4)-C(8)	-81.5(4)	C(29)-C(23)-N(24)-N(25)	62.4(5)
N(3)-C(4)-C(5)-C(6)	60.5(5)	C(28)-C(23)-N(24)-N(25)	-55.9(5)
C(8)-C(4)-C(5)-C(6)	-56.7(5)	C(29)-C(23)-N(24)-C(31)	-97.8(5)
C(4)-C(5)-C(6)-C(1)	-6.7(5)	C(28)-C(23)-N(24)-C(31)	143.8(4)
N(2)-C(1)-C(6)-C(5)	-52.1(5)	C(23)-N(24)-N(25)-C(19)	139.8(4)
C(7)-C(1)-C(6)-C(5)	65.0(5)	C(31)-N(24)-N(25)-C(19)	-60.1(6)
N(2)-C(1)-C(7)-C(8)	60.8(5)	C(23)-N(24)-N(25)-C(26)	-7.2(5)
C(6)-C(1)-C(7)-C(8)	-57.6(5)	C(31)-N(24)-N(25)-C(26)	152.9(4)
N(3)-C(4)-C(8)-C(7)	-54.0(5)	C(20)-C(19)-N(25)-N(24)	-37.4(6)
C(5)-C(4)-C(8)-C(7)	63.8(5)	C(18)-C(19)-N(25)-N(24)	145.4(4)
C(1)-C(7)-C(8)-C(4)	-5.2(6)	C(20)-C(19)-N(25)-C(26)	108.2(5)
N(3)-N(2)-C(9)-C(10)	7.3(5)	C(18)-C(19)-N(25)-C(26)	-69.0(6)
C(1)-N(2)-C(9)-C(10)	167.3(4)	N(24)-N(25)-C(26)-C(27)	63.3(5)
N(3)-N(2)-C(9)-C(11)	129.5(4)	C(19)-N(25)-C(26)-C(27)	-84.7(5)
C(1)-N(2)-C(9)-C(11)	-70.5(5)	N(24)-N(25)-C(26)-C(30)	-55.3(5)
N(3)-N(2)-C(9)-C(12)	-111.5(4)	C(19)-N(25)-C(26)-C(30)	156.7(4)
C(1)-N(2)-C(9)-C(12)	48.5(5)	N(25)-C(26)-C(27)-C(28)	-49.5(6)
C(21)-C(13)-C(14)-C(15)	0.1(6)	C(30)-C(26)-C(27)-C(28)	66.4(6)
C(21)-C(13)-C(14)-N(3)	-178.7(4)	C(26)-C(27)-C(28)-C(23)	-10.6(6)
N(2)-N(3)-C(14)-C(13)	-41.2(5)	N(24)-C(23)-C(28)-C(27)	63.8(5)
C(4)-N(3)-C(14)-C(13)	101.9(5)	C(29)-C(23)-C(28)-C(27)	-55.1(5)
N(2)-N(3)-C(14)-C(15)	140.0(4)	N(24)-C(23)-C(29)-C(30)	-47.3(7)
C(4)-N(3)-C(14)-C(15)	-76.9(5)	C(28)-C(23)-C(29)-C(30)	68.7(6)
C(13)-C(14)-C(15)-C(16)	2.0(7)	N(25)-C(26)-C(30)-C(29)	64.6(6)
N(3)-C(14)-C(15)-C(16)	-179.2(4)	C(27)-C(26)-C(30)-C(29)	-52.2(7)
C(14)-C(15)-C(16)-C(22)	-1.5(7)	C(23)-C(29)-C(30)-C(26)	-13.1(7)
C(22)-C(17)-C(18)-C(19)	0.4(7)	N(25)-N(24)-C(31)-C(32)	121.7(5)
C(17)-C(18)-C(19)-C(20)	1.4(7)	C(23)-N(24)-C(31)-C(32)	-79.7(6)
C(17)-C(18)-C(19)-N(25)	178.6(4)	N(25)-N(24)-C(31)-C(33)	-0.2(6)
C(18)-C(19)-C(20)-C(21)	-1.9(7)	C(23)-N(24)-C(31)-C(33)	158.4(4)
N(25)-C(19)-C(20)-C(21)	-179.0(4)	N(25)-N(24)-C(31)-C(34)	-118.0(5)
C(19)-C(20)-C(21)-C(13)	179.7(4)	C(23)-N(24)-C(31)-C(34)	40.6(6)
C(23)-N(24)-C(31)-C(34)	40.6(6)		

[S1] Nelsen, S. F. *J. Am. Chem. Soc.* **1966**, *88*, 5666-5667.
[S2] Nelsen, S. F.; Kessel, C. R.; Brien, D. J. *J. Am. Chem. Soc.* **1980**, *102*, 702-711.
[S3] Nelsen, S. F.; Hollinsed, W. C.; Kessel, C. R.; Calabrese, J.C. *J. Am. Chem. Soc.* **1978**, *100*, 7876-7882.
[S4] Nelsen, S. F.; Cunkle, G. T.; Evans, D. H.; Haller, K. J.; Kaftory, M.; Kirste, B.; Clark, T. *J. Am. Chem. Soc.* **1985**, *107*, 3829.
[S5] Nelsen, S. F.; Tran, H. Q.; Ismagilov, R. F.; Chen, L.-J; Powell, D. R. *J. Org. Chem.* **1998**, *63*, 2536-2543.
[S6] Nelsen, S. F.; Blackstock, S. C.; Frigo, T. B. *J. Am. Chem. Soc.* **1984**, *106*, 3366-3367.
[S7] Nelsen, S. F.; Blackstock, S. C.; Haller, K. J. *Tetrahedron*, **1986**, *42*, 6101-6109.
[S8] Nelsen, S. F.; Wang, Y.; Powell, D. R.; Hiyashi, R. K. *J. Am. Chem. Soc.* **1993**, *115*, 5246-5253.
[S9] Nelsen, S. F.; Wang, Y. *J. Org. Chem.* **1994**, *59*, 3082-3090.
[S10] Nelsen, S. F.; Tran, H. Q.; Powell, D. R.; Neugebauer, F. A. *J. Org. Chem.* **1995**, *60*, 6756-6763.
[S11] Nelsen, S. F.; Chen, L.-J.; Powell, D. R.; Neugebauer, F. A. *J. Am. Chem. Soc.* **1995**, *117*, 11434-11440.
[S12] Nelsen, S. F.; Ramm, M. T.; Ismagilov, R. F.; Nagy, M. A.; Trieber, D. A., II; Powell, D. R.; Chen, X.; Gengler, J. J.; Qu, Q.; Brandt, J. L.; Pladziewicz, J. R. *J. Am. Chem. Soc.* **1997**, *119*, 5900-5907.
[S13] (a) Langler, R. F.; Tidwell, T. T. *Tetrahedron Lett.* **1975**, 777-80. (b) Bomse, D. S.; Morton, T. H. *Tetrahedron Lett.* **1975**, 781-84.
[S14] Bruker-AXS. (2000-2003) SADABS V.2.05, SAINT V.6.22, SHELXTL V.6.10 & SMART 5.622 Software Reference Manuals. Bruker-AXS, Madison, Wisconsin, USA.
[S15] Blessing, R.H. *Acta Cryst*. **1995**, *A51*, 33-38.
[S16] All software and sources of the scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-Ray Systems, Madison, WI).
[S17] (a) Data Collection: SMART Software Reference Manual (1994). Bruker-AXS, 6300 Enterprise Dr., Madison, WI 53719-1173, USA. (b) Data Reduction: SAINT Software Reference Manual (1995). Bruker-AXS, 6300 Enterprise Dr., Madison, WI 53719-1173, USA.
[18] G. M. Sheldrick (1996). SADABS. Program for Empirical Absorption Correction of Area Detector Data. University of Göttingen, Germany.
[19] (a) G. M. Sheldrick (1994). SHELXTL Version 5 Reference Manual. Bruker-AXS, 6300 Enterprise Dr., Madison, WI 53719-1173, USA. (b) *International Tables for Crystallography, Vol C*, Tables 6.1.1.4, 4.2.6.8, and 4.2.4.2, Kluwer: Boston (1995).
[20] H. D. Flack, *Acta Cryst. A39*, 876-881 (1983).
[21] H. D. Flack and D. Schwarzenbach, *Acta Cryst. A44*, 499-506 (1988).