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Tris(acetonitrile)(1,1,4,7,7-pentamethyldiethylenetriamine)nickel(II) bis(hexafluoridophosphate)

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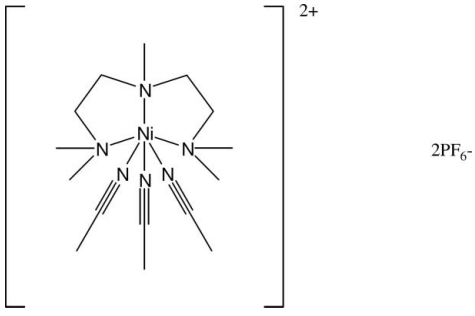
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.048; wR factor = 0.130; data-to-parameter ratio = 14.2.

The title compound, $[Ni(C_2H_3N)_3(C_9H_{23}N_3)](PF_6)_2$, has the Ni atom in an octahedral environment. It is coordinated by the three N atoms of one chelating 1,1,4,7,7-pentamethyldiethylenetriamine molecule and by three acetonitrile molecules. The charge is balanced by two hexafluoridophosphate anions. The ethylene chains and the central methyl group of the 1,1,4,7,7-pentamethyldiethylenetriamine are disordered over two sites with occupation factors 0.619 (6) and 0.381 (6).

Related literature

For related literature, see: Margraf *et al.* (2005, 2006).



Experimental

Crystal data

$[Ni(C_2H_3N)_3(C_9H_{23}N_3)](PF_6)_2$
M_r = 645.12
Monoclinic, $P2_1/c$
a = 17.4418 (8) Å
b = 10.5913 (6) Å
c = 15.7326 (8) Å
β = 110.921 (4)°
V = 2714.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.93 mm^{-1}
T = 173 (2) K
0.38 × 0.17 × 0.14 mm

Data collection

STOE IPDS II two-circle-diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.719, T_{max} = 0.881
33712 measured reflections
5076 independent reflections
4263 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.130
S = 1.04
5076 reflections
357 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.83 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2457).

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supplementary materials

Acta Cryst. (2007). E**63**, m2971 [**doi:10.1107/S1600536807054360**]

Tris(acetonitrile)(1,1,4,7,7-pentamethyldiethylenetriamine)nickel(II) bis(hexafluoridophosphate)

T. Morawitz, H.-W. Lerner and M. Bolte

Comment

We report here the X-ray crystal structure analysis of the nickel salt of 1,1,4,7,7-pentamethyldiethylenetriamine and hexafluoridophosphate, $[Ni(PMDTA)(CH_3CN)_3]^{2+}\cdot 2[PF_6]^-$ (PMDTA = $Me_2NHCH_2CH_2NMeCH_2CH_2NHMe_2$). We are interested in the synthesis of transition metal complexes with the PMDTA ligand (Margraf *et al.*, 2005) which are a convenient starting material for novel benzoquinone- and hydroquinone-substituted transition metal derivatives (Margraf *et al.*, 2006). The synthesis of the title compound was achieved by reaction of $NiCl_2$ with $AgPF_6$ in the presence of PMDTA, as shown in the equation below. Blue crystals of the title compound suitable for X-ray diffraction were grown from acetonitrile/diethyl ether solution at ambient temperature.

The title compound has the Ni atom in an octahedral environment. It is coordinated by the three N atoms of one chelating 1,1,4,7,7-pentamethyldiethylenetriamine molecule and three acetonitrile molecules. The charge is balanced by two hexafluoridophosphate anions.

Experimental

$AgPF_6$ (3.2 mmol, 0.799 g) was added to a solution of $NiCl_2$ (1.6 mmol, 0.205 g) and PMDTA (1.6 mmol, 0.274 g) in 20 ml acetonitrile. After filtering the reaction solution blue crystals of the title compound suitable for X-ray diffraction were grown by slow diffusion of diethyl ether (25 ml) to the acetonitrile solution at ambient temperature (Yield 85%).

Refinement

H atoms were were geometrically positioned and refined with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C) or U(H) = 1.5 $U_{eq}(C_{methyl})$] using a riding model with C—H = 0.99Å or 0.98Å for CH_2 and CH_3 groups, respectively. The methyl groups of the acetonitrile molecules were allowed to rotate but not to tip. The ethylene chains and the central methyl group of the 1,1,4,7,7-pentamethyldiethylenetriamine are disordered over two sites with the occupation factors of 0.619 (6) and 0.381 (6).

Figures

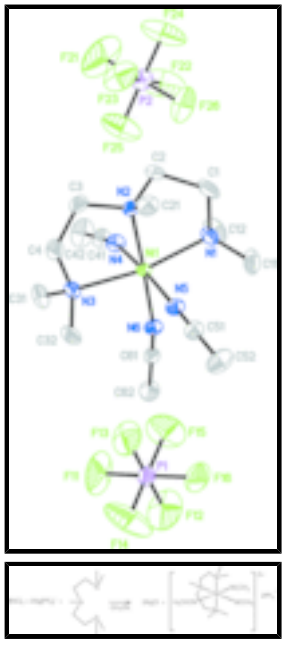

Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level; hydrogen atoms and the minor occupied sites of the disordered atoms are omitted for clarity.

Triacetonitrile(1,1,4,7,7-pentamethyldiethylenetriamine)nickel(II) bis(hexafluoridophosphate)

Crystal data

$[Ni(C_2H_3N)_3(C_9H_{23}N_3)](PF_6)_2$	F_{000} = 1320
M_r = 645.12	D_x = 1.578 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 31260 reflections
a = 17.4418 (8) Å	θ = 3.6–25.7º
b = 10.5913 (6) Å	μ = 0.93 mm^{-1}
c = 15.7326 (8) Å	T = 173 (2) K
β = 110.921 (4)º	Rod, light blue
V = 2714.7 (3) Å^3	0.38 × 0.17 × 0.14 mm
Z = 4	

Data collection

STOE IPDS II two-circle-diffractometer	5076 independent reflections
Radiation source: fine-focus sealed tube	4263 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.081
T = 173(2) K	θ_{max} = 25.7º
ω scans	θ_{min} = 3.6º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −21→20

T_{min} = 0.719, T_{max} = 0.881
33712 measured reflections

k = −12→12
l = −19→19

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.048

$wR(F^2)$ = 0.130

S = 1.04

5076 reflections

357 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0753P)^2 + 3.1233P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.83 e Å^{-3}

$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0019 (5)

Special details

Experimental. ;

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

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ni1	0.25753 (2)	0.72144 (3)	0.39571 (2)	0.01573 (14)	
N1	0.36863 (17)	0.6842 (3)	0.36422 (19)	0.0275 (6)	
C11	0.3512 (3)	0.6273 (4)	0.2723 (3)	0.0421 (9)	
H11A	0.3188	0.6866	0.2254	0.063*	
H11B	0.3204	0.5486	0.2676	0.063*	
H11C	0.4031	0.6094	0.2637	0.063*	
C12	0.4179 (3)	0.8012 (4)	0.3684 (3)	0.0502 (11)	
H12A	0.4306	0.8415	0.4279	0.075*	
H12B	0.3863	0.8597	0.3204	0.075*	
H12C	0.4691	0.7789	0.3595	0.075*	
C1	0.4183 (2)	0.5922 (4)	0.4352 (3)	0.0474 (11)	
H1A	0.4775	0.6060	0.4482	0.057*	0.619 (6)
H1B	0.4045	0.5048	0.4128	0.057*	0.619 (6)

H1A'	0.4527	0.6379	0.4906	0.057*	0.381 (6)
H1B'	0.4547	0.5426	0.4120	0.057*	0.381 (6)
C2	0.3981 (3)	0.6126 (6)	0.5261 (4)	0.0303 (13)	0.619 (6)
H2A	0.4297	0.5525	0.5740	0.036*	0.619 (6)
H2B	0.4119	0.6997	0.5494	0.036*	0.619 (6)
C2'	0.3532 (5)	0.4976 (8)	0.4591 (5)	0.0240 (19)	0.381 (6)
H2A'	0.3149	0.4568	0.4035	0.029*	0.381 (6)
H2B'	0.3824	0.4318	0.5035	0.029*	0.381 (6)
N2	0.30893 (16)	0.5893 (2)	0.49946 (16)	0.0213 (5)	
C21	0.2897 (4)	0.4536 (5)	0.4767 (4)	0.0311 (14)	0.619 (6)
H2C	0.2305	0.4401	0.4588	0.047*	0.619 (6)
H2D	0.3187	0.4016	0.5300	0.047*	0.619 (6)
H2E	0.3073	0.4299	0.4263	0.047*	0.619 (6)
C21'	0.3699 (6)	0.6356 (9)	0.5892 (5)	0.031 (2)	0.381 (6)
H2C'	0.3836	0.7240	0.5831	0.047*	0.381 (6)
H2D'	0.4198	0.5841	0.6062	0.047*	0.381 (6)
H2E'	0.3456	0.6286	0.6364	0.047*	0.381 (6)
C3	0.2762 (4)	0.6294 (5)	0.5703 (3)	0.0291 (13)	0.619 (6)
H3A	0.2993	0.7126	0.5950	0.035*	0.619 (6)
H3B	0.2925	0.5676	0.6209	0.035*	0.619 (6)
C3'	0.2374 (6)	0.5281 (8)	0.5133 (6)	0.029 (2)	0.381 (6)
H3A'	0.2562	0.4712	0.5667	0.035*	0.381 (6)
H3B'	0.2055	0.4781	0.4591	0.035*	0.381 (6)
C4	0.1832 (2)	0.6380 (3)	0.5295 (2)	0.0320 (7)	
H4A	0.1598	0.5541	0.5069	0.038*	0.619 (6)
H4B	0.1614	0.6655	0.5766	0.038*	0.619 (6)
H4A'	0.1333	0.6018	0.5359	0.038*	0.381 (6)
H4B'	0.2144	0.6824	0.5868	0.038*	0.381 (6)
N3	0.15869 (16)	0.7311 (2)	0.45229 (18)	0.0228 (5)	
C31	0.1484 (2)	0.8585 (3)	0.4872 (3)	0.0374 (8)	
H31A	0.2008	0.8866	0.5319	0.056*	
H31B	0.1071	0.8542	0.5161	0.056*	
H31C	0.1303	0.9186	0.4365	0.056*	
C32	0.0773 (2)	0.6948 (4)	0.3851 (3)	0.0337 (8)	
H32A	0.0812	0.6112	0.3602	0.051*	
H32B	0.0602	0.7569	0.3357	0.051*	
H32C	0.0368	0.6923	0.4151	0.051*	
N4	0.31638 (17)	0.8772 (3)	0.47852 (19)	0.0270 (6)	
C41	0.3362 (2)	0.9746 (3)	0.5098 (2)	0.0296 (7)	
C42	0.3589 (3)	1.0992 (4)	0.5502 (3)	0.0555 (12)	
H42A	0.3122	1.1365	0.5621	0.083*	
H42B	0.3740	1.1535	0.5082	0.083*	
H42C	0.4057	1.0916	0.6075	0.083*	
N5	0.19225 (16)	0.5870 (2)	0.29991 (17)	0.0236 (6)	
C51	0.1556 (2)	0.5185 (3)	0.2436 (2)	0.0255 (7)	
C52	0.1096 (3)	0.4311 (4)	0.1724 (2)	0.0412 (9)	
H52A	0.0684	0.4778	0.1233	0.062*	
H52B	0.0823	0.3684	0.1975	0.062*	
H52C	0.1473	0.3881	0.1482	0.062*	

N6	0.20712 (17)	0.8590 (2)	0.29706 (17)	0.0246 (6)
C61	0.1785 (2)	0.9455 (3)	0.2550 (2)	0.0248 (7)
C62	0.1419 (3)	1.0573 (3)	0.2014 (3)	0.0402 (9)
H62A	0.1184	1.1114	0.2364	0.060*
H62B	0.0986	1.0313	0.1448	0.060*
H62C	0.1842	1.1042	0.1871	0.060*
P1	0.06223 (6)	0.25769 (8)	0.91132 (6)	0.0285 (2)
F11	0.0825 (3)	0.1545 (4)	0.8516 (3)	0.1208 (17)
F12	0.0812 (3)	0.1592 (4)	0.9895 (3)	0.1160 (17)
F13	0.0419 (2)	0.3586 (3)	0.8304 (2)	0.0832 (11)
F14	0.1530 (2)	0.3044 (5)	0.9479 (4)	0.1351 (19)
F15	−0.0322 (2)	0.2210 (4)	0.8730 (3)	0.1009 (13)
F16	0.0399 (2)	0.3628 (3)	0.97109 (18)	0.0702 (9)
P2	0.60650 (6)	0.79625 (9)	0.70684 (7)	0.0366 (2)
F21	0.6226 (4)	0.8502 (5)	0.8033 (3)	0.154 (2)
F22	0.6603 (2)	0.9020 (3)	0.6859 (3)	0.1093 (16)
F23	0.5515 (2)	0.6870 (4)	0.7266 (2)	0.0805 (10)
F24	0.6838 (2)	0.7085 (3)	0.7493 (3)	0.1051 (15)
F25	0.5273 (2)	0.8800 (3)	0.6621 (3)	0.1024 (13)
F26	0.5853 (3)	0.7330 (4)	0.6089 (2)	0.0873 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0157 (2)	0.0130 (2)	0.0168 (2)	0.00060 (13)	0.00374 (14)	0.00083 (13)
N1	0.0241 (14)	0.0277 (14)	0.0349 (15)	0.0070 (11)	0.0155 (12)	0.0090 (12)
C11	0.053 (2)	0.045 (2)	0.043 (2)	0.0073 (18)	0.0347 (19)	0.0026 (17)
C12	0.034 (2)	0.050 (2)	0.078 (3)	−0.0101 (18)	0.033 (2)	0.005 (2)
C1	0.032 (2)	0.062 (3)	0.055 (2)	0.0274 (19)	0.0240 (19)	0.030 (2)
C2	0.023 (3)	0.036 (3)	0.028 (3)	0.011 (2)	0.003 (2)	0.005 (2)
C2'	0.024 (4)	0.022 (4)	0.023 (4)	0.007 (3)	0.006 (3)	0.003 (3)
N2	0.0240 (13)	0.0201 (12)	0.0176 (12)	0.0036 (10)	0.0050 (10)	0.0023 (10)
C21	0.047 (4)	0.014 (2)	0.031 (3)	0.009 (2)	0.011 (2)	0.007 (2)
C21'	0.033 (5)	0.036 (5)	0.016 (4)	0.005 (4)	−0.003 (3)	0.001 (3)
C3	0.039 (3)	0.028 (3)	0.020 (2)	0.010 (2)	0.010 (2)	0.007 (2)
C3'	0.032 (5)	0.027 (4)	0.029 (4)	−0.001 (4)	0.013 (4)	0.010 (3)
C4	0.0335 (19)	0.0374 (19)	0.0314 (17)	0.0028 (15)	0.0193 (15)	0.0078 (14)
N3	0.0201 (13)	0.0229 (13)	0.0263 (13)	0.0020 (10)	0.0093 (11)	−0.0010 (10)
C31	0.037 (2)	0.0314 (19)	0.053 (2)	0.0029 (15)	0.0269 (18)	−0.0076 (16)
C32	0.0155 (15)	0.044 (2)	0.0408 (19)	−0.0008 (14)	0.0096 (14)	−0.0038 (16)
N4	0.0266 (14)	0.0229 (14)	0.0292 (14)	−0.0036 (11)	0.0072 (11)	−0.0008 (11)
C41	0.0315 (18)	0.0292 (18)	0.0291 (16)	−0.0090 (14)	0.0121 (14)	−0.0062 (14)
C42	0.072 (3)	0.039 (2)	0.061 (3)	−0.029 (2)	0.030 (2)	−0.025 (2)
N5	0.0247 (14)	0.0220 (13)	0.0208 (13)	−0.0011 (11)	0.0040 (11)	−0.0007 (11)
C51	0.0306 (17)	0.0212 (15)	0.0239 (15)	0.0000 (13)	0.0086 (13)	0.0012 (13)
C52	0.054 (2)	0.0325 (19)	0.0303 (18)	−0.0126 (17)	0.0063 (17)	−0.0101 (15)
N6	0.0264 (14)	0.0220 (13)	0.0230 (13)	0.0022 (11)	0.0059 (11)	0.0043 (11)
C61	0.0272 (16)	0.0255 (16)	0.0215 (15)	0.0039 (13)	0.0085 (13)	0.0012 (13)

C62	0.055 (2)	0.0307 (19)	0.0362 (19)	0.0201 (17)	0.0184 (18)	0.0142 (15)
P1	0.0328 (5)	0.0281 (4)	0.0257 (4)	0.0039 (4)	0.0118 (4)	−0.0009 (3)
F11	0.194 (5)	0.094 (3)	0.097 (3)	0.061 (3)	0.079 (3)	−0.023 (2)
F12	0.187 (5)	0.092 (3)	0.084 (3)	0.062 (3)	0.066 (3)	0.063 (2)
F13	0.113 (3)	0.089 (2)	0.0639 (18)	0.032 (2)	0.0519 (19)	0.0411 (17)
F14	0.0301 (17)	0.150 (4)	0.206 (5)	−0.009 (2)	0.019 (2)	−0.003 (4)
F15	0.057 (2)	0.098 (3)	0.132 (3)	−0.0344 (19)	0.015 (2)	−0.023 (2)
F16	0.087 (2)	0.0728 (19)	0.0465 (15)	0.0167 (16)	0.0193 (14)	−0.0234 (13)
P2	0.0420 (6)	0.0273 (5)	0.0373 (5)	−0.0052 (4)	0.0102 (4)	−0.0038 (4)
F21	0.240 (6)	0.132 (4)	0.066 (2)	−0.021 (4)	0.024 (3)	−0.064 (3)
F22	0.063 (2)	0.064 (2)	0.173 (4)	−0.0286 (17)	0.008 (2)	0.052 (2)
F23	0.075 (2)	0.099 (2)	0.0649 (18)	−0.0377 (19)	0.0205 (16)	0.0212 (17)
F24	0.0484 (18)	0.063 (2)	0.177 (4)	0.0078 (15)	0.008 (2)	0.045 (2)
F25	0.065 (2)	0.066 (2)	0.151 (4)	0.0231 (17)	0.009 (2)	0.007 (2)
F26	0.117 (3)	0.096 (3)	0.065 (2)	−0.018 (2)	0.052 (2)	−0.0248 (18)

Geometric parameters (Å, °)

Ni1—N6	2.081 (3)	C3'—H3B'	0.9900
Ni1—N5	2.090 (3)	C4—N3	1.503 (4)
Ni1—N2	2.093 (2)	C4—H4A	0.9900
Ni1—N4	2.125 (3)	C4—H4B	0.9900
Ni1—N1	2.200 (3)	C4—H4A'	0.9900
Ni1—N3	2.207 (3)	C4—H4B'	0.9900
N1—C11	1.494 (5)	N3—C32	1.486 (4)
N1—C12	1.496 (5)	N3—C31	1.492 (4)
N1—C1	1.502 (4)	C31—H31A	0.9800
C11—H11A	0.9800	C31—H31B	0.9800
C11—H11B	0.9800	C31—H31C	0.9800
C11—H11C	0.9800	C32—H32A	0.9800
C12—H12A	0.9800	C32—H32B	0.9800
C12—H12B	0.9800	C32—H32C	0.9800
C12—H12C	0.9800	N4—C41	1.142 (4)
C1—C2	1.605 (7)	C41—C42	1.458 (5)
C1—C2'	1.655 (9)	C42—H42A	0.9800
C1—H1A	0.9900	C42—H42B	0.9800
C1—H1B	0.9900	C42—H42C	0.9800
C1—H1A'	0.9900	N5—C51	1.146 (4)
C1—H1B'	0.9900	C51—C52	1.455 (4)
C2—N2	1.480 (6)	C52—H52A	0.9800
C2—H2A	0.9900	C52—H52B	0.9800
C2—H2B	0.9900	C52—H52C	0.9800
C2'—N2	1.515 (8)	N6—C61	1.137 (4)
C2'—H2A'	0.9900	C61—C62	1.461 (4)
C2'—H2B'	0.9900	C62—H62A	0.9800
N2—C3	1.484 (6)	C62—H62B	0.9800
N2—C3'	1.489 (9)	C62—H62C	0.9800
N2—C21	1.490 (6)	P1—F12	1.556 (3)
N2—C21'	1.514 (8)	P1—F14	1.559 (4)

C21—H2C	0.9800	P1—F11	1.562 (3)
C21—H2D	0.9800	P1—F15	1.586 (3)
C21—H2E	0.9800	P1—F16	1.593 (3)
C21'—H2C'	0.9800	P1—F13	1.602 (3)
C21'—H2D'	0.9800	P2—F21	1.551 (4)
C21'—H2E'	0.9800	P2—F22	1.570 (3)
C3—C4	1.518 (7)	P2—F24	1.577 (3)
C3—H3A	0.9900	P2—F25	1.580 (3)
C3—H3B	0.9900	P2—F26	1.598 (3)
C3'—C4	1.576 (10)	P2—F23	1.602 (3)
C3'—H3A'	0.9900		
N6—Ni1—N5	87.88 (10)	N2—C3—H3B	109.7
N6—Ni1—N2	177.41 (10)	C4—C3—H3B	109.7
N5—Ni1—N2	94.48 (10)	H3A—C3—H3B	108.2
N6—Ni1—N4	84.08 (11)	N2—C3'—C4	106.5 (5)
N5—Ni1—N4	171.95 (10)	N2—C3'—H3A'	110.4
N2—Ni1—N4	93.56 (10)	C4—C3'—H3A'	110.4
N6—Ni1—N1	97.41 (10)	N2—C3'—H3B'	110.4
N5—Ni1—N1	90.71 (11)	C4—C3'—H3B'	110.4
N2—Ni1—N1	83.66 (10)	H3A'—C3'—H3B'	108.6
N4—Ni1—N1	90.32 (11)	N3—C4—C3	109.5 (3)
N6—Ni1—N3	94.90 (10)	N3—C4—C3'	111.6 (4)
N5—Ni1—N3	91.12 (10)	C3—C4—C3'	54.0 (4)
N2—Ni1—N3	83.99 (10)	N3—C4—H4A	109.8
N4—Ni1—N3	89.57 (10)	C3—C4—H4A	109.8
N1—Ni1—N3	167.61 (10)	C3'—C4—H4A	58.2
C11—N1—C12	106.9 (3)	N3—C4—H4B	109.8
C11—N1—C1	108.8 (3)	C3—C4—H4B	109.8
C12—N1—C1	109.6 (3)	C3'—C4—H4B	138.6
C11—N1—Ni1	113.5 (2)	H4A—C4—H4B	108.2
C12—N1—Ni1	112.4 (2)	N3—C4—H4A'	109.3
C1—N1—Ni1	105.6 (2)	C3—C4—H4A'	141.2
N1—C11—H11A	109.5	C3'—C4—H4A'	109.3
N1—C11—H11B	109.5	H4A—C4—H4A'	54.8
H11A—C11—H11B	109.5	H4B—C4—H4A'	56.6
N1—C11—H11C	109.5	N3—C4—H4B'	109.3
H11A—C11—H11C	109.5	C3—C4—H4B'	59.1
H11B—C11—H11C	109.5	C3'—C4—H4B'	109.3
N1—C12—H12A	109.5	H4A—C4—H4B'	140.8
N1—C12—H12B	109.5	H4B—C4—H4B'	54.0
H12A—C12—H12B	109.5	H4A'—C4—H4B'	108.0
N1—C12—H12C	109.5	C32—N3—C31	106.6 (3)
H12A—C12—H12C	109.5	C32—N3—C4	109.2 (3)
H12B—C12—H12C	109.5	C31—N3—C4	109.5 (3)
N1—C1—C2	108.9 (3)	C32—N3—Ni1	112.7 (2)
N1—C1—C2'	107.6 (4)	C31—N3—Ni1	113.9 (2)
C2—C1—C2'	59.5 (4)	C4—N3—Ni1	104.91 (19)
N1—C1—H1A	109.9	N3—C31—H31A	109.5
C2—C1—H1A	109.9	N3—C31—H31B	109.5

C2'—C1—H1A	142.4	H31A—C31—H31B	109.5
N1—C1—H1B	109.9	N3—C31—H31C	109.5
C2—C1—H1B	109.9	H31A—C31—H31C	109.5
C2'—C1—H1B	54.2	H31B—C31—H31C	109.5
H1A—C1—H1B	108.3	N3—C32—H32A	109.5
N1—C1—H1A'	110.2	N3—C32—H32B	109.5
C2—C1—H1A'	53.6	H32A—C32—H32B	109.5
C2'—C1—H1A'	110.2	N3—C32—H32C	109.5
H1A—C1—H1A'	59.1	H32A—C32—H32C	109.5
H1B—C1—H1A'	139.8	H32B—C32—H32C	109.5
N1—C1—H1B'	110.2	C41—N4—Ni1	166.1 (3)
C2—C1—H1B'	140.8	N4—C41—C42	178.0 (4)
C2'—C1—H1B'	110.2	C41—C42—H42A	109.5
H1A—C1—H1B'	52.7	C41—C42—H42B	109.5
H1B—C1—H1B'	58.5	H42A—C42—H42B	109.5
H1A'—C1—H1B'	108.5	C41—C42—H42C	109.5
N2—C2—C1	105.5 (4)	H42A—C42—H42C	109.5
N2—C2—H2A	110.6	H42B—C42—H42C	109.5
C1—C2—H2A	110.6	C51—N5—Ni1	176.1 (3)
N2—C2—H2B	110.6	N5—C51—C52	179.6 (4)
C1—C2—H2B	110.6	C51—C52—H52A	109.5
H2A—C2—H2B	108.8	C51—C52—H52B	109.5
N2—C2'—C1	101.6 (5)	H52A—C52—H52B	109.5
N2—C2'—H2A'	111.5	C51—C52—H52C	109.5
C1—C2'—H2A'	111.5	H52A—C52—H52C	109.5
N2—C2'—H2B'	111.5	H52B—C52—H52C	109.5
C1—C2'—H2B'	111.5	C61—N6—Ni1	168.5 (3)
H2A'—C2'—H2B'	109.3	N6—C61—C62	179.6 (4)
C2—N2—C3	112.8 (4)	C61—C62—H62A	109.5
C2—N2—C3'	151.8 (4)	C61—C62—H62B	109.5
C3—N2—C3'	56.4 (4)	H62A—C62—H62B	109.5
C2—N2—C21	110.6 (4)	C61—C62—H62C	109.5
C3—N2—C21	110.0 (4)	H62A—C62—H62C	109.5
C3'—N2—C21	59.2 (4)	H62B—C62—H62C	109.5
C2—N2—C21'	50.6 (4)	F12—P1—F14	91.1 (3)
C3—N2—C21'	62.5 (4)	F12—P1—F11	88.7 (2)
C3'—N2—C21'	110.5 (5)	F14—P1—F11	91.5 (3)
C21—N2—C21'	124.0 (4)	F12—P1—F15	92.0 (3)
C2—N2—C2'	65.4 (4)	F14—P1—F15	175.7 (3)
C3—N2—C2'	152.8 (4)	F11—P1—F15	91.6 (3)
C3'—N2—C2'	111.2 (5)	F12—P1—F16	91.7 (2)
C21—N2—C2'	52.1 (4)	F14—P1—F16	89.4 (2)
C21'—N2—C2'	107.8 (5)	F11—P1—F16	179.0 (2)
C2—N2—Ni1	102.9 (3)	F15—P1—F16	87.5 (2)
C3—N2—Ni1	102.8 (2)	F12—P1—F13	179.5 (3)
C3'—N2—Ni1	104.9 (4)	F14—P1—F13	89.4 (3)
C21—N2—Ni1	117.4 (2)	F11—P1—F13	91.3 (2)
C21'—N2—Ni1	118.3 (4)	F15—P1—F13	87.5 (2)
C2'—N2—Ni1	104.0 (3)	F16—P1—F13	88.34 (18)

N2—C21—H2C	109.5	F21—P2—F22	91.7 (3)
N2—C21—H2D	109.5	F21—P2—F24	88.5 (3)
H2C—C21—H2D	109.5	F22—P2—F24	91.44 (19)
N2—C21—H2E	109.5	F21—P2—F25	93.0 (3)
H2C—C21—H2E	109.5	F22—P2—F25	89.9 (2)
H2D—C21—H2E	109.5	F24—P2—F25	177.9 (2)
N2—C21'—H2C'	109.5	F21—P2—F26	175.6 (3)
N2—C21'—H2D'	109.5	F22—P2—F26	92.7 (2)
H2C'—C21'—H2D'	109.5	F24—P2—F26	91.7 (3)
N2—C21'—H2E'	109.5	F25—P2—F26	86.6 (2)
H2C'—C21'—H2E'	109.5	F21—P2—F23	89.3 (3)
H2D'—C21'—H2E'	109.5	F22—P2—F23	179.0 (2)
N2—C3—C4	109.8 (4)	F24—P2—F23	88.42 (19)
N2—C3—H3A	109.7	F25—P2—F23	90.2 (2)
C4—C3—H3A	109.7	F26—P2—F23	86.32 (19)
N6—Ni1—N1—C11	−65.5 (2)	N5—Ni1—N2—C21'	176.2 (5)
N5—Ni1—N1—C11	22.4 (2)	N4—Ni1—N2—C21'	−3.9 (5)
N2—Ni1—N1—C11	116.8 (2)	N1—Ni1—N2—C21'	86.0 (5)
N4—Ni1—N1—C11	−149.6 (2)	N3—Ni1—N2—C21'	−93.1 (5)
N3—Ni1—N1—C11	120.9 (5)	N5—Ni1—N2—C2'	56.8 (4)
N6—Ni1—N1—C12	56.0 (3)	N4—Ni1—N2—C2'	−123.4 (4)
N5—Ni1—N1—C12	143.9 (3)	N1—Ni1—N2—C2'	−33.5 (4)
N2—Ni1—N1—C12	−121.6 (3)	N3—Ni1—N2—C2'	147.4 (4)
N4—Ni1—N1—C12	−28.1 (3)	C2—N2—C3—C4	164.0 (4)
N3—Ni1—N1—C12	−117.6 (5)	C3'—N2—C3—C4	−45.7 (5)
N6—Ni1—N1—C1	175.4 (3)	C21—N2—C3—C4	−71.9 (4)
N5—Ni1—N1—C1	−96.7 (3)	C21'—N2—C3—C4	169.3 (6)
N2—Ni1—N1—C1	−2.2 (3)	C2'—N2—C3—C4	−115.8 (9)
N4—Ni1—N1—C1	91.3 (3)	Ni1—N2—C3—C4	53.9 (4)
N3—Ni1—N1—C1	1.9 (6)	C2—N2—C3'—C4	117.8 (9)
C11—N1—C1—C2	−150.4 (4)	C3—N2—C3'—C4	42.5 (4)
C12—N1—C1—C2	93.0 (4)	C21—N2—C3'—C4	−166.5 (7)
Ni1—N1—C1—C2	−28.2 (4)	C21'—N2—C3'—C4	75.4 (6)
C11—N1—C1—C2'	−87.4 (4)	C2'—N2—C3'—C4	−165.0 (5)
C12—N1—C1—C2'	156.0 (4)	Ni1—N2—C3'—C4	−53.1 (5)
Ni1—N1—C1—C2'	34.8 (4)	N2—C3—C4—N3	−58.9 (4)
N1—C1—C2—N2	59.8 (5)	N2—C3—C4—C3'	44.1 (4)
C2'—C1—C2—N2	−39.9 (4)	N2—C3'—C4—N3	56.1 (6)
N1—C1—C2'—N2	−64.0 (5)	N2—C3'—C4—C3	−42.9 (4)
C2—C1—C2'—N2	38.0 (4)	C3—C4—N3—C32	151.1 (3)
C1—C2—N2—C3	−168.2 (3)	C3'—C4—N3—C32	93.2 (5)
C1—C2—N2—C3'	130.9 (9)	C3—C4—N3—C31	−92.5 (4)
C1—C2—N2—C21	68.1 (4)	C3'—C4—N3—C31	−150.5 (4)
C1—C2—N2—C21'	−174.2 (7)	C3—C4—N3—Ni1	30.1 (3)
C1—C2—N2—C2'	41.6 (4)	C3'—C4—N3—Ni1	−27.9 (5)
C1—C2—N2—Ni1	−58.1 (4)	N6—Ni1—N3—C32	62.5 (2)
C1—C2'—N2—C2	−39.3 (4)	N5—Ni1—N3—C32	−25.4 (2)
C1—C2'—N2—C3	−131.7 (8)	N2—Ni1—N3—C32	−119.8 (2)
C1—C2'—N2—C3'	171.2 (5)	N4—Ni1—N3—C32	146.5 (2)

C1—C2'—N2—C21	172.8 (6)	N1—Ni1—N3—C32	−123.9 (5)
C1—C2'—N2—C21'	−67.6 (6)	N6—Ni1—N3—C31	−59.1 (2)
C1—C2'—N2—Ni1	58.8 (4)	N5—Ni1—N3—C31	−147.1 (2)
N5—Ni1—N2—C2	124.3 (3)	N2—Ni1—N3—C31	118.6 (2)
N4—Ni1—N2—C2	−55.9 (3)	N4—Ni1—N3—C31	24.9 (2)
N1—Ni1—N2—C2	34.0 (3)	N1—Ni1—N3—C31	114.5 (5)
N3—Ni1—N2—C2	−145.1 (3)	N6—Ni1—N3—C4	−178.8 (2)
N5—Ni1—N2—C3	−118.4 (3)	N5—Ni1—N3—C4	93.3 (2)
N4—Ni1—N2—C3	61.5 (3)	N2—Ni1—N3—C4	−1.1 (2)
N1—Ni1—N2—C3	151.4 (3)	N4—Ni1—N3—C4	−94.7 (2)
N3—Ni1—N2—C3	−27.7 (3)	N1—Ni1—N3—C4	−5.2 (6)
N5—Ni1—N2—C3'	−60.1 (4)	N6—Ni1—N4—C41	16.6 (12)
N4—Ni1—N2—C3'	119.7 (4)	N2—Ni1—N4—C41	−162.4 (12)
N1—Ni1—N2—C3'	−150.3 (4)	N1—Ni1—N4—C41	114.0 (12)
N3—Ni1—N2—C3'	30.5 (4)	N3—Ni1—N4—C41	−78.4 (12)
N5—Ni1—N2—C21	2.5 (3)	N5—Ni1—N6—C61	139.9 (14)
N4—Ni1—N2—C21	−177.6 (3)	N4—Ni1—N6—C61	−40.1 (14)
N1—Ni1—N2—C21	−87.7 (3)	N1—Ni1—N6—C61	−129.7 (14)
N3—Ni1—N2—C21	93.2 (3)	N3—Ni1—N6—C61	48.9 (14)

Fig. 1

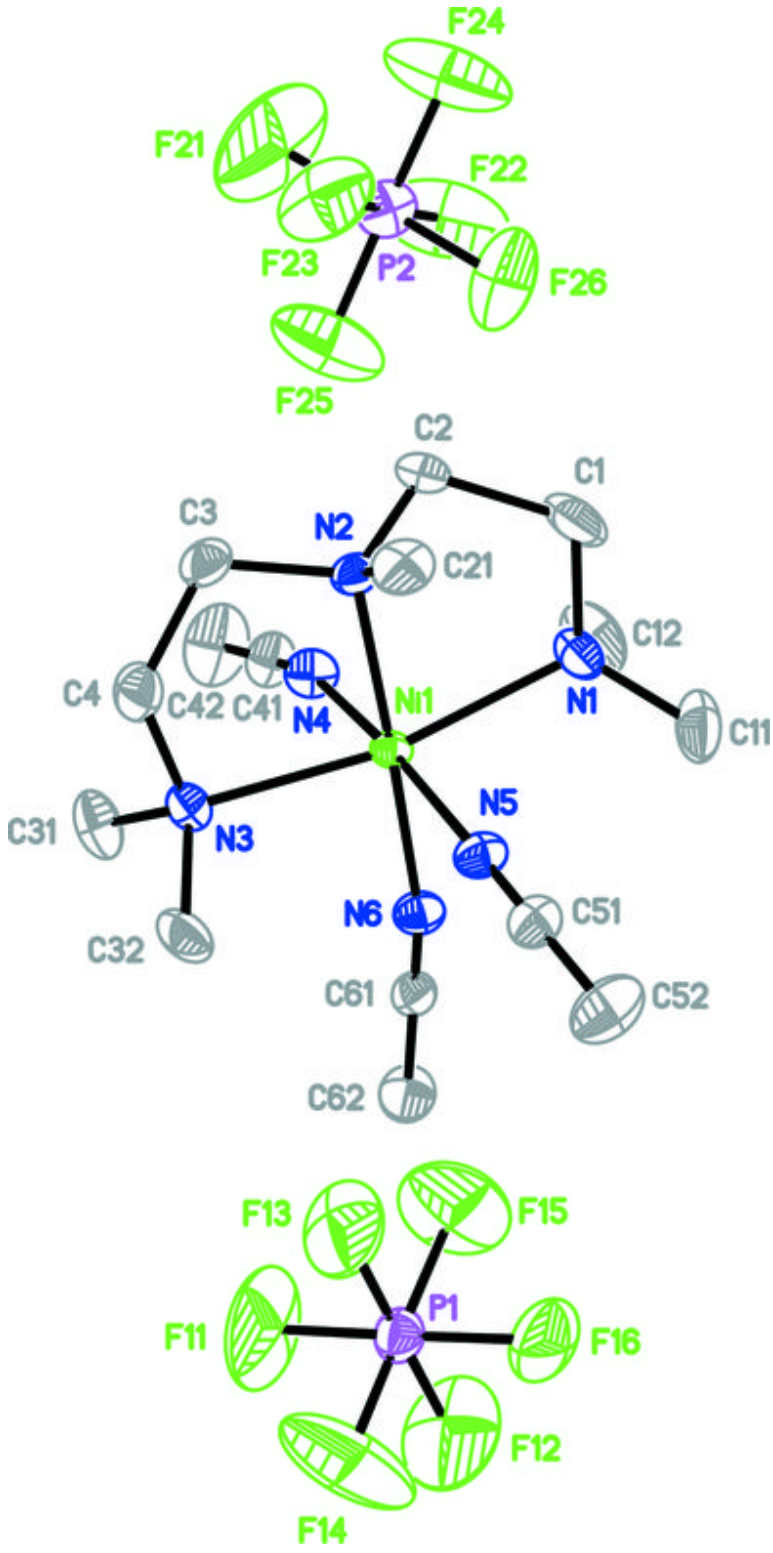
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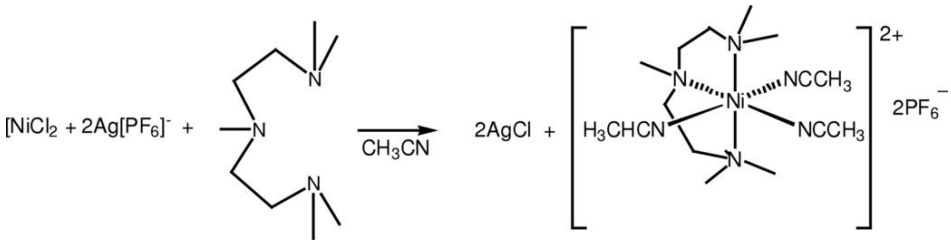
Fig. 2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