O3	1.0339 (2)	0.4914 (4)	0.6467 (2)	0.0444 (6)
O4	0.6128 (2)	0.4501 (3)	0.2306 (2)	0.0365 (5)
N	0.8141 (2)	0.8278 (3)	0.4350 (2)	0.0185 (5)
C1	0.9473 (2)	0.5842 (4)	0.5872 (2)	0.0264 (6)
C2	0.6692 (2)	0.5552 (4)	0.3126 (2)	0.0235 (6)
C3	0.9287 (2)	0.7614 (5)	0.5155 (2)	0.0315 (7)
C4	0.7530 (3)	0.7032 (6)	0.3345 (2)	0.0397 (8)
C5	0.8072 (3)	1.0603 (5)	0.4125 (3)	0.0386 (8)

Table 2. *Selected geometric parameters (Å, °)*

Cr—O2	1.948 (2)	O4—C2	1.223 (3)
Cr—O1	1.963 (2)	N—C3	1.484 (3)
Cr—N	2.060 (2)	N—C4	1.490 (4)
O1—C1	1.283 (3)	N—C5	1.490 (4)
O2—C2	1.280 (3)	C1—C3	1.517 (4)
O3—C1	1.222 (3)	C2—C4	1.510 (4)
O2—Cr—O1	90.84 (9)	C5—N—Cr	113.1 (2)
O2—Cr—N	85.11 (8)	O3—C1—O1	123.8 (3)
O1—Cr—N	83.42 (8)	O3—C1—C3	119.8 (3)
C1—O1—Cr	115.5 (2)	O1—C1—C3	116.4 (2)
C2—O2—Cr	116.5 (2)	O4—C2—O2	123.8 (2)
C3—N—C4	111.9 (2)	O4—C2—C4	119.1 (2)
C3—N—C5	109.7 (2)	O2—C2—C4	117.1 (2)
C4—N—C5	110.0 (3)	N—C3—C1	112.4 (2)
C3—N—Cr	105.3 (2)	N—C4—C2	114.2 (2)
C4—N—Cr	106.7 (2)		

The title structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for non-H atoms. All H atoms were found from difference Fourier syntheses and refined isotropically.

Data collection, cell refinement and data reduction: *SDP* (Frenz, 1985). Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *SHELXL*93 (Sheldrick, 1993). Preparation of materials for publication and molecular graphics: *ORTEP*II (Johnson, 1976) and *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Software used to prepare material for publication: *NRCVAX*.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: MU1280). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). C**53**, 434–436

trans-Bis[4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole-N^1,N']copper(II) Bis(tetrafluoroborate)

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Abstract

The title complex, $[Cu(C_{12}H_{10}N_6)_2](BF_4)_2$, contains square-planar mononuclear Cu^{II}–bis[4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole] cations counterbalanced by semi-coordinated BF_4^- anions. The Cu atoms reside on inversion centers and have a distorted octahedral coordination sphere. The amino group is hydrogen bridged to two of the F atoms of an adjacent BF_4^- anion.

Comment

Dipyridyl ligands give structural chemists access to a wide variety of transition metal complexes and unusual geometries. Simple variation of the central metal atom and associated counterions has resulted in the isolation of novel dimers (Sommerer, Westcott, Jircitano & Abboud, 1995) and metal-containing extended structures (Sommerer, Westcott, Jircitano & Abboud, 1996). The ligand 4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole (abpt) exemplifies this behavior and has been used to isolate dinuclear Ni^{II} (Keij, de Graaff, Haasnoot & Reedijk, 1984), Cu^{II} (van Koningsbruggen *et al.*, 1995) and Mn^{II} (Faulmann, van Koningsbruggen, de Graaff, Haasnoot & Reedijk, 1990) species, as well as mononuclear Rh^{II} (García *et al.*, 1986), Ru^{II} (Rheingold, Saisuwan & Thomas, 1993) and Cu^{II} (Cornelissen *et al.*, 1992). An

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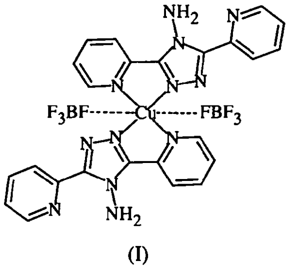
example of an elongated octahedral mononuclear Cu^{II}–abpt complex, (I), is reported here.



(I)

An X-ray diffraction analysis of the title complex showed the Cu^{II} atom to reside on an inversion center, with a distorted octahedral coordination sphere consisting of two abpt ligands forming a square plane and two BF_4^- ions in axial positions (Fig. 1). The bond distances within the coordination sphere are characteristic of Cu^{II}—N distances reported previously (Müller, Bernardinelli & Reedijk, 1996) and are unremarkable. The Cu—F bond distance of 2.515 (3) Å is similar to interactions of this type observed previously (Brown, Lee & Melsom, 1968; Driessen, de Graaff & Wiesmeijer, 1987) and is in the range which has been described as 'semi-coordinated' (Procter, Hathaway & Nicholls, 1968). The dihedral angle between the mean planes of the coordinated pyridine and triazole rings is 4.5 (2)°, while that between the non-coordinated pyridine and triazole rings is 15.2 (2)°. Thus, there is indeed a minimal deviation from true planarity.

Both amine H atoms are hydrogen bonded; the HN1 atom binds intramolecularly to N5, which appears to stabilize the non-coordinated pyridine ring in a *trans*-like conformation. The HN2 atom hydrogen bridges to two of the F atoms of a symmetry-related BF_4^- ion, apparently helping to keep the anion well ordered. The hydrogen-bond geometry is given in Table 2.

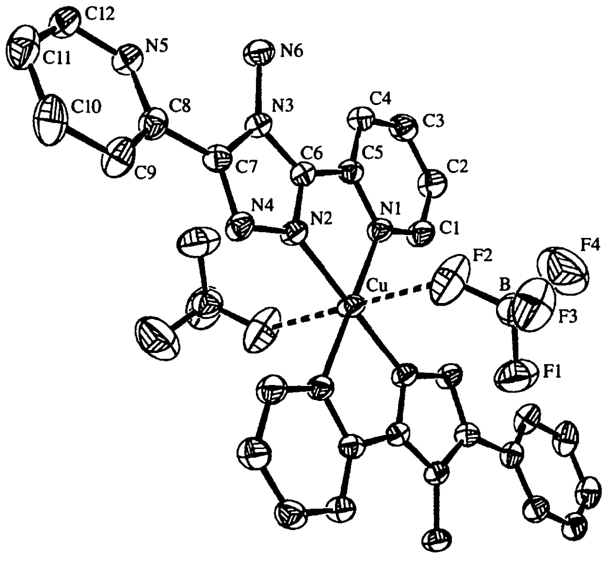


Fig. 1. Displacement ellipsoid drawing of the title compound with ellipsoids drawn at the 50% probability level.

Experimental

For the preparation of the title compound, copper(II) tetrafluoroborate was combined with abpt in a 2:1 stoichiometric ratio in methanol. The resulting solution was filtered and allowed to evaporate slowly. Clear blue crystals suitable for X-ray diffraction studies were isolated.

Crystal data

$[Cu(C_{12}H_{10}N_6)_2](BF_4)_2$
$M_r = 713.68$
Monoclinic
$P2_1/c$
$a = 8.814\,(2)$ Å
$b = 9.742\,(2)$ Å
$c = 16.516\,(3)$ Å
$\beta = 99.65\,(3)°$
$V = 1398.1\,(5)$ Å^3
$Z = 2$
$D_x = 1.695$ Mg m^{-3}
$D_m = 1.68\,(1)$ Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 10–11°$
$\mu = 0.877$ mm^{-1}
$T = 298$ K
Block
0.34 × 0.30 × 0.24 mm
Blue

Data collection

Siemens *P*3*m*/*V* diffractometer
ω scans
Absorption correction: analytical
$T_{min} = 0.776$, $T_{max} = 0.821$
3212 measured reflections
3212 independent reflections

2087 reflections with $I > 2\sigma(I)$
$\theta_{max} = 27.49°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 21$
4 standard reflections every 96 reflections intensity decay: 1%

Refinement

Refinement on F^2
$R(F) = 0.0578$
$wR(F^2) = 0.1467$
$S = 1.140$
3212 reflections
214 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0962P)^2 + 0.2265P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.696$ e Å^{-3}
$\Delta\rho_{min} = -0.648$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Cu—N2	1.968 (3)	N3—C7	1.377 (4)
Cu—N1	2.027 (3)	N3—N6	1.415 (4)
Cu—F2	2.515 (3)	N4—C7	1.318 (5)
N1—C1	1.342 (5)	N5—C12	1.338 (5)
N1—C5	1.353 (4)	N5—C8	1.339 (5)
N2—C6	1.315 (4)	C5—C6	1.466 (5)
N2—N4	1.367 (4)	C7—C8	1.461 (5)
N3—C6	1.338 (4)		

N2—Cu—N1	80.97 (12)	C7—N3—N6	129.9 (3)
N1—Cu—F2	92.13 (13)	C7—N4—N2	106.1 (3)
N2—Cu—F2	87.29 (11)	C12—N5—C8	116.8 (4)
C1—N1—C5	117.7 (3)	N1—C5—C6	110.9 (3)
C5—N1—Cu	115.0 (2)	N2—C6—N3	109.1 (3)
C6—N2—N4	109.2 (3)	N2—C6—C5	119.3 (3)
C6—N2—Cu	113.8 (2)	N4—C7—N3	109.8 (3)
C6—N3—C7	105.8 (3)	N4—C7—C8	124.9 (3)
C6—N3—N6	124.0 (3)	N3—C7—C8	125.3 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—HN1···N5	0.82	2.24	2.859 (5)	132
N6—HN2···F3^i	0.84	2.45	3.096 (6)	135
N6—HN2···F4^i	0.84	2.71	3.376 (6)	138

Symmetry code: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$.

The title structure was solved by the heavy-atom method. Amine H atoms were located from a difference synthesis. All other H atoms were included in calculated positions (C—H 0.93 Å) and displacement parameters were fixed at $1.2U_{iso}$ of the bound atom.

Data collection: *SHELXTL-Plus* (Sheldrick, 1990*b*). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEX* (McArdle, 1995). Software used to prepare material for publication: *SHELXL93*.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: FG1212). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Chelate-Stabilized Tungsten–Imido–Monoalkyl Complex, $[WCl(PhN)(CH_2CMe_3)\{o\text{-}C_6H_4(NSiMe_3)_2\}]$

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Abstract

The title compound, [*N*,*N*′-bis(trimethylsilyl)-*o*-phenylenediamido-*N*,*N*′](chloro)(neopentyl)(phenylimido-*N*)-tungsten(VI), $[WCl(C_5H_{11})(C_6H_5N)(C_{12}H_{22}N_2Si_2)]$, has been synthesized and its crystal structure determined. The geometry around the W atom, imposed by the bidentate *o*-phenylenediamido ligand and the bulky neopentyl ligand, is that of a distorted trigonal bipyramid. A relatively short W—N(imido) bond length of 1.741 (4) Å is interpreted as arising from the interaction of the lone pair of electrons on the imido N atom and the tungsten metal center.

Comment

Recently, the chemistry of high oxidation state metal–alkyl complexes has been studied extensively because of their role in olefin polymerization and metathesis reactions (Ivin, 1982; Jordan, 1991). The title compound, $[\{o\text{-}C_6H_4(NSiMe_3)_2\}(PhN)W(CH_2CMe_3)Cl]$, (I), is of great interest because it possesses only one alkyl group and one chloride ligand. The compound is thus an intermediate between dihalide complexes (catalyst precursors for Ziegler–Natta polymerization reactions) and dialkyl complexes (catalyst precursors for metathesis reactions). In spite of the great interest in these compounds, only a