

Table 2. Selected values of bond lengths (Å) and bond angles (°) with e.s.d.'s

Fe(1)—P(1)	2.276 (6)	P(2)—C(11)	1.84 (1)
Fe(1)—P(2)	2.282 (5)	P(2)—C(21)	1.84 (1)
Fe(1)—C(50)	1.81 (2)	N(1)—C(50)	1.18 (2)
Fe(1)—C(60)	1.84 (2)	N(1)—C(51)	1.39 (2)
Fe(1)—C(70)	1.84 (2)	N(2)—C(60)	1.16 (3)
Fe(1)—C(80)	1.86 (2)	N(2)—C(61)	1.40 (2)
P(1)—C(10)	1.73 (2)	N(3)—C(70)	1.15 (3)
P(1)—C(31)	1.83 (2)	N(3)—C(71)	1.38 (3)
P(1)—C(41)	1.84 (2)	N(4)—C(80)	1.13 (2)
P(2)—C(10)	1.69 (2)	N(4)—C(81)	1.38 (2)
P(1)—Fe1—P(2)	69.9 (2)	C(10)—P(1)—C(31)	111.7 (9)
P(1)—Fe1—C(50)	163.9 (6)	C(10)—P(1)—C(41)	116.5 (9)
P(1)—Fe1—C(60)	88.2 (7)	C(31)—P(1)—C(41)	100.9 (7)
P(1)—Fe1—C(70)	87.9 (7)	Fe(1)—P(2)—C(10)	95.9 (7)
P(1)—Fe1—C(80)	97.1 (6)	Fe(1)—P(2)—C(11)	116.5 (5)
P(2)—Fe1—C(50)	94.0 (6)	Fe(1)—P(2)—C(21)	119.5 (5)
P(2)—Fe1—C(60)	90.8 (7)	C(10)—P(2)—C(11)	114.0 (9)
P(2)—Fe1—C(70)	92.0 (7)	C(10)—P(2)—C(21)	113.8 (9)
P(2)—Fe1—C(80)	166.9 (6)	C(11)—P(2)—C(21)	98.3 (6)
C(50)—Fe1—C(60)	92. (1)	P(1)—C(10)—P(2)	99. (1)
C(50)—Fe1—C(70)	93.0 (9)	Fe(1)—C(50)—N(1)	178. (2)
C(50)—Fe1—C(80)	99.0 (8)	Fe(1)—C(60)—N(2)	178. (2)
C(60)—Fe1—C(70)	174.0 (9)	Fe(1)—C(70)—N(3)	176. (2)
C(60)—Fe1—C(80)	87.1 (9)	Fe(1)—C(80)—N(4)	173. (2)
C(70)—Fe1—C(80)	89.0 (9)	C(50)—N(1)—C(51)	174. (2)
Fe1—P(1)—C(10)	94.9 (7)	C(60)—N(2)—C(61)	170. (2)
Fe1—P(1)—C(31)	119.5 (5)	C(70)—N(3)—C(71)	174. (2)
Fe1—P(1)—C(41)	114.4 (5)	C(80)—N(4)—C(81)	178. (2)

the chelating dpmm-H ligand. The bond lengths Fe—C and Fe—P of the first coordination sphere of the Fe, as well as the bond angles among them, are in the expected range (see Table 2), the small bite of the bidentate dpmm-H ligand being the cause of the distortion of the octahedron around the Fe atom.

The bond lengths P(1)—C(10) and P(2)—C(10) are equal and show a shortening with respect to the P—C distance commonly associated with the dpmm derivatives, which is in accord with the expected multiple bond character for the anionic bis(diphenylphosphino)methanide (dpmm-H), present in our compound. The dihedral angle between the equatorial coordination plane of iron [defined by P(1), P(2), C(50) and C(80)] and that defined by P(1), P(2) and C(10) is 1(1)°.

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**Structure of 2,2'-[5-(2-Aminoethyl)-1-hydroxy-2,5,8-triazanon-3-ene-1,9-diyl]bis-(pyridine *N*-oxide)nickel Hexafluorophosphate Monohydrate, [Ni(C<sub>18</sub>H<sub>26</sub>N<sub>6</sub>O<sub>3</sub>)]<sup>2+</sup>·2PF<sub>6</sub><sup>-</sup>·H<sub>2</sub>O: a Novel Tripodal Nickel(II) Complex with Three Inequivalent Arms**

BY SHAO-HUA GOU,\* XIAO-ZENG YOU AND ZHENG XU

*Coordination Chemistry Institute, Nanjing University, Nanjing, People's Republic of China*

AND SHI-XIONG LIU AND CHI-CHANG LIN

*Department of Chemistry, Fuzhou University, Fuzhou, People's Republic of China*

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**Abstract.**  $M_r = 741.09$ , monoclinic,  $P2_1/c$ ,  $a = 13.014$  (2),  $b = 15.038$  (2),  $c = 14.329$  (2) Å,  $\beta = 97.98$  (1)°,  $V = 2777$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.77$  g cm<sup>-3</sup>, Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å,  $\mu = 9.30$  cm<sup>-1</sup>,  $T = 296$  K,  $F(000) = 1504$ ,  $R = 0.045$  and  $wR = 0.068$

for 3621 unique observed [ $I > 3.0\sigma(I)$ ] reflections. The nickel(II) atom is in a distorted octahedral environment of a tripodal ligand with three inequivalent arms. The compound is derived from the reaction of 2,2',2''-triaminotriethylamine with 2-pyridinecarboxaldehyde *N*-oxide in the presence of an Ni<sup>II</sup> salt.

\* To whom correspondence should be addressed.

**Introduction.** Unusual coordination polyhedra of complex compounds have often attracted the chemist's attention, since they can provide interesting models to explore the role of ligand configuration in determining the geometry of complexes (Wilson & Rose, 1968). A series of X-ray structure analyses of the transition metal compounds with tripodal ligands have been reported (Mealli & Lingafelter, 1970) and reviewed (Kichner, Mealli, Bailey, Howe, Torre, Wilson, Andrews, Rose & Lingafelter, 1987). All of them exhibited octahedral coordination polyhedra as well as 'special' physical and chemical properties. The tripodal ligand reported, which had three similar arms, was obtained from the Schiff-base condensation of 2,2',2''-triaminotriethylamine (tren) with 2-pyridinecarboxaldehyde. Here we report the first nickel(II) complex of a tripodal ligand with three inequivalent arms derived from the reaction of tren and 2-pyridinecarboxaldehyde *N*-oxide in the presence of an Ni<sup>II</sup> salt.

**Experimental.** The complex was prepared by an *in situ* reaction involving the sequential addition of NiCl<sub>2</sub> and KPF<sub>6</sub> to a stirred aqueous solution containing tren and 2-pyridinecarboxaldehyde *N*-oxide. Details of the preparation will be reported elsewhere. Single crystals grown from acetone/water (1:1). Cell dimensions and crystal orientation matrix determined on a AFC5R diffractometer by least-squares treatment of the setting angles of 20 reflections in the range of 10 <  $\theta$  < 15°. Crystal dimensions 0.38 × 0.32 × 0.42 mm; intensities of reflections with 2 $\theta$  < 48° measured; *hkl* ranges: *h* 0 to 14, *k* -16 to 16, *l* -16 to 16;  $\omega$ -2 $\theta$  scans;  $\omega$ -scan width (1.020 + tan $\theta$ )°, graphite-monochromatized Mo *K* $\alpha$  radiation. Three standard reflections were measured every 200 reflections, and showed no significant changes during data collection. 7553 reflections measured; 5476 unique; 3621 reflections with *I* > 3.0 $\sigma$ (*I*) were used in structure solution and refinement; *R*<sub>int</sub> = 0.030. Data corrected for Lorentz and polarization factors and absorption effects (max. and min. transmission factors 1.045 and 0.937). Gaussian integration grid 6 × 6 × 6. Space group *P*2<sub>1</sub>/*c* uniquely determined from systematic absences.

The crystal structure was solved by the heavy-atom method. Refinement was by full-matrix least-squares calculations, initially with isotropic and later with anisotropic thermal parameters. At an intermediate stage in the refinement, difference maps showed maxima in positions consistent with the expected locations of H atoms; in the final round of calculations the H atoms were positioned on geometrical grounds (C—H = 0.95 Å) and included (as riding atoms) in the structure factor calculations. Final refinement was on *F* by least-squares methods, refining 388 parameters, *R* = 0.045, *wR* = 0.068,

Table 1. *Positional parameters and B<sub>eq</sub> for [Ni(C<sub>18</sub>H<sub>26</sub>N<sub>6</sub>O<sub>3</sub>)](PF<sub>6</sub>)<sub>2</sub>·H<sub>2</sub>O*

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B <sub>eq</sub> (Å <sup>2</sup> )
Ni	0.25468 (5)	0.04533 (4)	0.23663 (4)	2.15 (3)
O1	0.1208 (3)	-0.0097 (2)	0.2819 (2)	2.9 (2)
O2	0.0972 (4)	0.2376 (3)	0.3892 (3)	4.4 (2)
O11	0.3384 (3)	0.0274 (2)	0.3656 (2)	3.1 (2)
N	0.1881 (4)	0.0714 (3)	0.1000 (3)	2.9 (2)
N1	0.1268 (3)	0.0023 (3)	0.3743 (3)	2.5 (2)
N2	0.1844 (3)	0.1664 (3)	0.2727 (3)	2.5 (2)
N11	0.4143 (3)	0.0809 (3)	0.4006 (3)	2.6 (2)
N12	0.3834 (4)	0.0938 (3)	0.1939 (3)	2.8 (2)
N22	0.2753 (4)	-0.0819 (3)	0.1844 (3)	3.8 (2)
C1	0.1371 (5)	-0.0700 (4)	0.4300 (4)	3.7 (3)
C2	0.1483 (5)	-0.0592 (5)	0.5266 (4)	4.7 (3)
C3	0.1497 (5)	0.0235 (5)	0.5639 (4)	4.6 (3)
C4	0.1385 (5)	0.0962 (4)	0.5042 (4)	4.0 (3)
C5	0.1258 (4)	0.0855 (4)	0.4082 (3)	2.7 (2)
C6	0.1057 (4)	0.1592 (4)	0.3372 (4)	3.1 (2)
C7	0.1406 (5)	0.2060 (4)	0.1801 (4)	3.6 (3)
C8	0.1029 (4)	0.1346 (4)	0.1093 (4)	3.5 (3)
C11	0.4298 (5)	0.0909 (4)	0.4957 (3)	3.2 (3)
C12	0.5112 (5)	0.1387 (4)	0.5390 (4)	4.2 (3)
C13	0.5801 (5)	0.1784 (5)	0.4879 (5)	4.7 (3)
C14	0.5638 (5)	0.1679 (4)	0.3927 (4)	3.6 (3)
C15	0.4812 (4)	0.1208 (3)	0.3475 (4)	2.6 (2)
C16	0.4657 (5)	0.1168 (4)	0.2448 (4)	3.0 (3)
C17	0.3779 (5)	0.0932 (4)	0.0910 (4)	4.2 (3)
C18	0.2671 (5)	0.1120 (5)	0.0468 (4)	4.1 (3)
C27	0.2313 (6)	-0.0859 (5)	0.0837 (4)	5.3 (4)
C28	0.1502 (5)	-0.0155 (5)	0.0608 (4)	4.3 (3)
Ow	0.0536 (4)	0.3950 (3)	0.3007 (4)	6.4 (3)

goodness-of-fit = 1.81,  $w = 1/\sigma^2(F_o)$ . Max.  $\Delta/\sigma = 0.02$ , electron density in final difference map  $\pm 0.55 e \text{ \AA}^{-3}$ ; no chemically significant features. Scattering factors and anomalous-dispersion corrections for non-H atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol IV). All calculations were performed on a MicroVAX II computer using *TEXSAN* software package (Molecular Structure Corporation, 1985). Atomic coordinates, and details of molecular geometry are given in Tables 1 and 2.\* Fig. 1 is a view of the molecule prepared using *TEXSAN*, and Fig. 2 is the packing view of the molecules in a unit cell.

**Discussion.** The nickel(II) ion of each molecule is in a distorted octahedral environment, see Fig. 1. None of the groups of atoms (O1, N2, N12, N22), (N, N2, N22, O11) and (N, N12, O1, O11), which may comprise the basal plane of an octahedron around the Ni<sup>II</sup> atom, are coplanar. The Ni—O and Ni—N bond lengths are different from those reported in the literature, presumably for steric reasons. Thus the Ni—N, Ni—N12 and Ni—O11 bond lengths of 2.065 (4), 1.999 (4) and 2.029 (4) Å respectively are

\* Lists of structure factors, anisotropic thermal parameters, PF<sub>6</sub> coordinates, complete bond lengths and angles, calculated hydrogen coordinates, intermolecular distances, mean plane data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53549 (44 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and bond angles (°)

Ni—N12	1.999 (4)	N11—C15	1.372 (7)	
Ni—O11	2.029 (4)	N12—C16	1.258 (7)	
Ni—N	2.065 (4)	N12—C17	1.467 (7)	
Ni—N22	2.085 (5)	N22—C27	1.478 (8)	
Ni—O1	2.110 (3)	C1—C2	1.382 (8)	
Ni—N2	2.132 (4)	C2—C3	1.35 (1)	
O1—N1	1.328 (5)	C3—C4	1.383 (9)	
O2—C6	1.408 (6)	C4—C5	1.371 (7)	
O11—N11	1.318 (6)	C5—C6	1.503 (8)	
N—C8	1.480 (7)	C7—C8	1.511 (8)	
N—C28	1.480 (8)	C11—C12	1.357 (9)	
N—C18	1.493 (7)	C12—C13	1.37 (1)	
N1—C5	1.343 (6)	C13—C14	1.360 (8)	
N1—C1	1.345 (7)	C14—C15	1.373 (8)	
N2—C6	1.476 (7)	C15—C16	1.458 (7)	
N2—C7	1.494 (7)	C17—C18	1.519 (9)	
N11—C11	1.357 (6)	C27—C28	1.50 (1)	
N12—Ni—O11	87.7 (2)	N—Ni—N2	86.0 (2)	
N12—Ni—N	84.2 (2)	N22—Ni—O1	84.4 (2)	
N12—Ni—N22	94.0 (2)	N22—Ni—N2	162.0 (2)	
N12—Ni—O1	178.3 (2)	O1—Ni—N2	82.0 (1)	
N12—Ni—N2	99.7 (2)	N1—O1—Ni	108.4 (3)	
O11—Ni—N	172.0 (2)	C28—N—C18	112.5 (4)	
O11—Ni—N22	97.2 (2)	N11—O11—Ni	123.0 (3)	
O11—Ni—O1	91.7 (1)	C8—N—C28	112.8 (5)	
O11—Ni—N2	95.0 (2)	C8—N—C18	111.1 (4)	
N—Ni—N22	83.8 (2)	C8—N—Ni	104.9 (3)	
N—Ni—O1	96.3 (2)	N1—C1—C2	119.1 (5)	
C28—N—Ni	105.4 (3)	C18—N—Ni	109.5 (3)	
O1—Ni—C5	119.1 (4)	C3—C2—C1	119.9 (6)	
O1—Ni—C1	117.9 (5)	C5—N1—C1	123.0 (4)	
C2—C3—C4	119.2 (6)	C5—C4—C3	121.1 (6)	
C6—N2—C7	111.9 (4)	N1—C5—C4	117.8 (5)	
C6—N2—Ni	116.6 (3)	N1—C5—C6	116.8 (4)	
C7—N2—Ni	104.4 (3)	C4—C5—C6	125.3 (5)	
O11—N11—C11	116.7 (4)	O11—N11—C15	123.7 (4)	
C11—N11—C15	119.5 (5)	C16—N12—C17	120.2 (4)	
C16—N12—Ni	127.2 (3)	O2—C6—N2	112.5 (5)	
C17—N12—Ni	112.3 (4)	O2—C6—C5	106.1 (4)	
N2—C6—C5	114.0 (4)	C27—N22—Ni	109.7 (4)	
N2—C7—C8	111.2 (5)	N12—C17—C18	109.2 (4)	
N—C8—C7	109.8 (5)	C12—C11—N11	121.0 (5)	
N—C18—C17	113.1 (4)	C11—C12—C13	120.9 (5)	
C14—C13—C12	117.6 (6)	C13—C14—C15	122.4 (5)	
N22—C27—C28	110.5 (5)	N11—C15—C16	122.2 (5)	
N11—C15—C14	118.7 (5)	C14—C15—C16	119.1 (5)	
N—C28—C27	110.7 (5)	N12—C16—C15	125.7 (5)	
X—H...Y	X—H (Å)	H...Y (Å)	X...Y (Å)	X—H...Y (°)
O2—HO4 Ow	1.05	1.76	2.708 (6)	148.4
Ow—HO5 O1	0.97	1.84	2.803 (6)	171.5

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

shorter, whereas Ni—N2 [2.132 (4) Å] and Ni—O1 [2.110 (3) Å] are longer than the relevant values listed by Orpen, Brammer, Allen, Kennard, Watson & Taylor (1989).

It has been reported (Kichner *et al.*, 1987) that the tren ligand can adjust the distance of the *M*—*N* (bridging atom) by rearranging its configuration from a tetrahedron to a square plane, and all three amino moieties of tren have been condensed with aromatic aldehydes to form a potential heptadentate ligand of which three arms are identical in transition metal complexes. Hence, the *M*—*N* (bridging atom) distance is usually greater than that of a normal *M*—*N* bond.

It is of interest that the three arms of the tripodal ligand in our complex are not equivalent despite the fact that the mole ratio of tren to aldehyde was 1:3 during complex formation. Generally, Schiff-base condensations are known to proceed by way of

nucleophilic attack by the amino group at the C atom of a carbonyl function. This yields a carbinol-amine intermediate (seldom isolated) followed by elimination of H<sub>2</sub>O to generate the imine (Patai, 1970). Normally the reaction is acid catalyzed, but this role may often be taken by a metal ion, in which case the imine product is generally, but not always, isolated as a metal complex. The X-ray structure studied here indicates:

(1) The coordinated N12 atom is part of the expected imine system. This is confirmed by the C16—N12 bond length of 1.258 (7) Å, normal for a C=N bond, and the N12—C16—C15 angle of

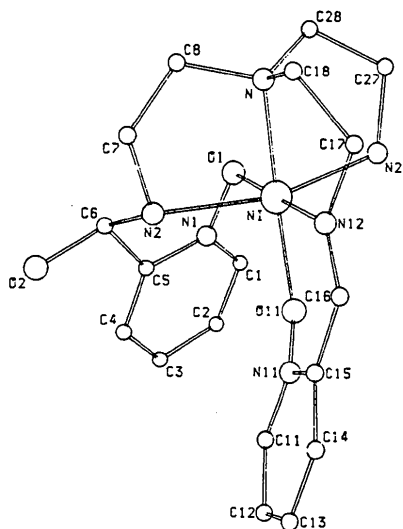
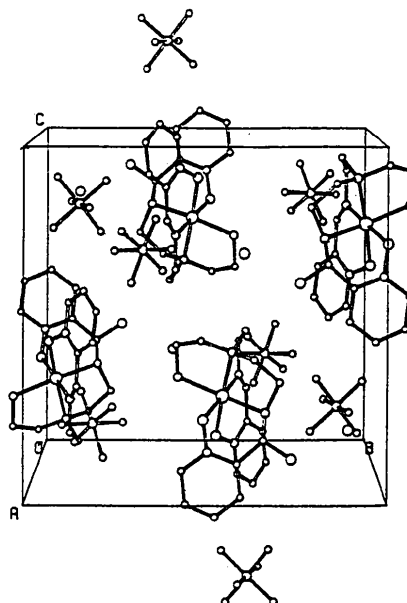
Fig. 1. Coordination geometry about the Ni<sup>II</sup> atom.

Fig. 2. Packing view of the molecules in a unit cell.

125.7 (5)° both of which indicate  $sp^2$  hybridization for C16.

(2) The coordinated N2 atom is part of a stable carbinolamine group HO2—C6HR—N2R'. The  $sp^3$  hybridization of C6 is confirmed by the single-bond values for C6—C5, C6—O2 and C6—N2 of 1.503 (8), 1.408 (6) and 1.476 (7) Å together with the normal tetrahedral angles.

(3) Coordination of N22, a primary amine function of tren which has not condensed with the aldehyde. This is presumably a consequence of the steric requirements imposed by the coordination of the tertiary nitrogen of tren.

In contrast to our complex the tertiary nitrogen in [Ni(py<sub>3</sub>tren)](PF<sub>6</sub>)<sub>2</sub> (Kichner *et al.*, 1987) is not coordinated (Ni—N 3.235 Å) and all the primary amine groups can undergo Schiff-base condensation with an aldehyde. Thus whether the tertiary N atom is coordinated to the metal or not has an important impact on the nature of the tripodal ligand formed.

Fig. 2 is a packing diagram of the unit-cell contents. The O1...Ow and O2...Ow distances, 2.803 (6)

and 2.708 (6) Å respectively, are indicative of hydrogen bonding; the formation of the latter may increase the stability of the carbinolamine intermediate. The Ow...F3 distance of 2.887 (7) Å is greater than that reported by Zhou (1984).

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## Structure of (Isothiocyanato)[3,3,9,9-tetramethyl-4,8-diazaundecane-2,10-dione dioximato(1-)]copper(II) Monohydrate

BY TSONG-JEN LEE, TSENG-YUH LEE AND CHUNG-SUN CHUNG

*National Tsing Hua University, Hsinchu, Taiwan 30043*

AND YUN-MING WANG

*Institute of Nuclear Energy Research, PO Box 3, Lung Tan 32500, Taiwan*

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**Abstract.** [Cu(C<sub>13</sub>H<sub>27</sub>N<sub>4</sub>O<sub>2</sub>)(NCS)].H<sub>2</sub>O,  $M_r = 411.02$ , orthorhombic,  $P2_12_12_1$ ,  $a = 7.236$  (2),  $b = 13.291$  (5),  $c = 20.802$  (3) Å,  $U = 2000.6$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.365$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 1.26$  mm<sup>-1</sup>,  $F(000) = 868$ ,  $T = 297$  (5) K,  $R = 0.058$  for 1412 observed reflections [ $I > 3\sigma(I)$ ]. The coordination about the Cu<sup>II</sup> atom is a distorted square pyramid with the deprotonated diazadioxime molecule equatorial and the N atom of the isothiocyanato group axial. The Cu atom is significantly (0.349 Å) out of the basal plane of the four N atoms towards the isothiocyanato group. The central six-membered chelate ring is in a stable chair conformation; one of the two terminal five-membered rings is in a stable *gauche* conformation, while the other is in an unstable eclipsed conformation.

**Introduction.** It is now well established that copper(II) coordinates with a deprotonated diazadioxime and a monodentate ligand to form a five-coordinate square-pyramidal complex having a strong intramolecular hydrogen bond between *cis* oxime groups (Gavel & Schlemper, 1979). A comprehensive study of this hydrogen bond is in progress in order to examine the effect of the O...O distance on the H-atom position and the important factors that influence the O...O distance. Previously, three important factors have been reported (Lee, Chang, Chung & Wang, 1990; Pal, Murmann, Schlemper, Fair & Hussain, 1986); (1) the constraint imposed by the methylene C atoms bridging the amine N atoms; (2) changing from an  $sp^3$  amine N atom to an  $sp^2$  imine N atom; and (3) the steric effect of the C—