Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Bis{2-[*N*,*N*-bis(2-aminoethyl)amino]ethanaminium}-nickel(II) hexacyanidoferrate(II)

Yue-Juan Yao, Hui-Ming Shu, Chuan-Qin Du and Huai-Ming Hu*

Department of Chemistry, Northwest University, Xi'an 710069, People's Republic of China

Correspondence e-mail: chemhu1@nwu.edu.cn

Received 14 March 2007
Accepted 21 March 2007

Key indicators

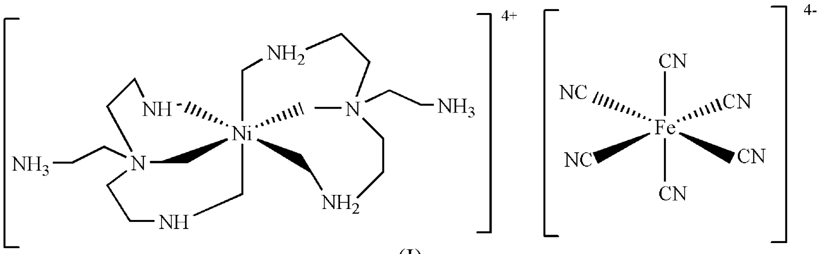
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.083
Data-to-parameter ratio = 16.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $[Ni(C_6H_{19}N_4)_2][Fe(CN)_6]$, the Ni^{2+} and Fe^{2+} cations are both octahedrally coordinated. The complex cations and anions, both of which are centrosymmetric, are linked by a network of N—H···N hydrogen bonds to form a three-dimensional network.

Comment

The title compound, (I), is a molecular salt containing complex cations and anions (Fig. 1). It complements related materials containing hexacyanidometalate anions that show novel magnetic properties (Kou *et al.*, 2002).



(I)

The general synthetic approach for these types of bimetallic assemblies is to utilize ligands in the cation that can make hydrogen bonds to the anion. In particular, chelating ligands such as 2,2′-bipyridine (Brinker & König, 1979) and diethylenetriamine (Razak *et al.*, 2000) have been studied. Here we use a tripodal ligand, which has attracted more and more attention recently (Hajela *et al.*, 2001, and references therein).

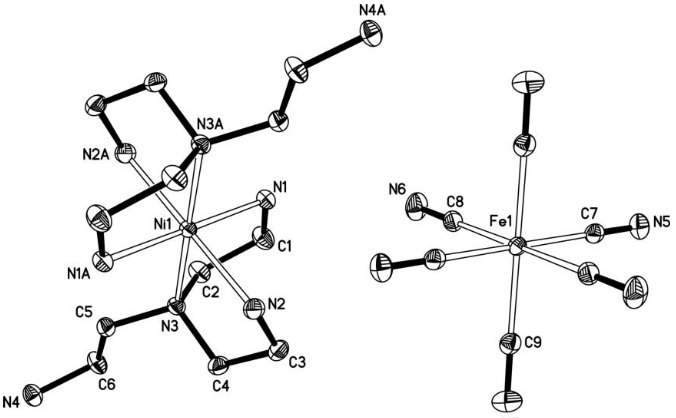


Figure 1
The molecular structure of (I) with displacement ellipsoids drawn at the 30% probability level. All H atoms have been omitted for clarity. Atoms with the suffix A are generated by the symmetry operation (1 − *x*, 1 − *y*, 1 − *z*). Unlabelled atoms in the anion are related to labelled atoms by (1 − *x*, 2 − *y*, 2 − *z*).

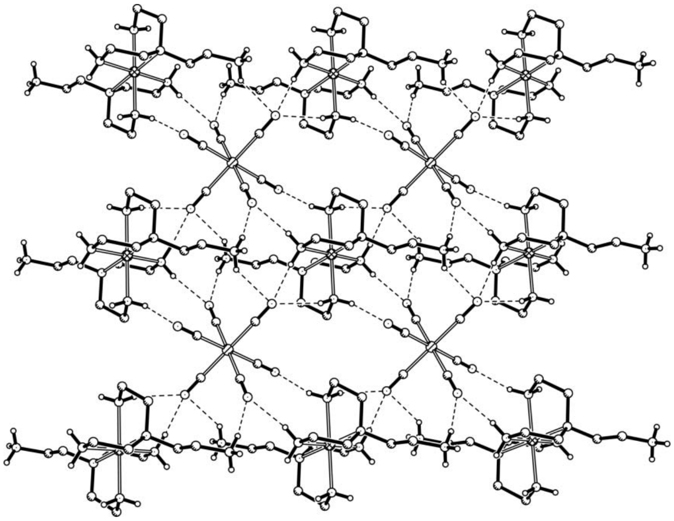


Figure 2
The layer structure in (I) arising from hydrogen bonds (dashed lines). C-bound H atoms have been omitted.

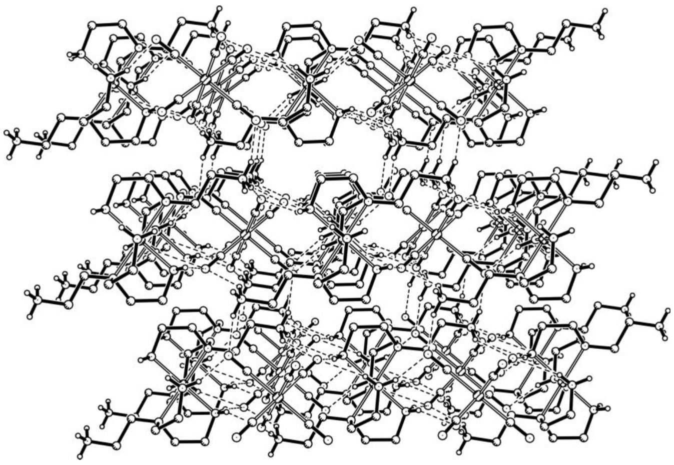


Figure 3
The three-dimensional hydrogen-bonding network in (I), with the hydrogen bonds shown as dashed lines. C-bound H atoms have been omitted.

The structure of (I) consists of one [Ni(Htren)$_2$]$^{4+}$ [Htren = tris(2-aminoethyl)ammonium cation, C$_6$H$_{19}$N$_4$$^+$] complex cation and one [Fe(CN)$_6$]$^{4-}$ complex anion. Both metal atoms have site symmetry $\overline{1}$. The Ni^{II} ion is coordinated by the two *N*,*N*,*N*-tridentate ligands in a distorted octahedral geometry (Table 1). The mean Ni—N bond length of of 2.129 (2)Å is in the normal range (Barkigia *et al.*, 1999). The N—Ni—N chelate bite angles in (I) are comparable to those of other Ni^{II} complexes with the tris(2-aminoethyl)amine ligand (Calatayud *et al.*, 2003). The Fe—C bond lengths in (I) are slightly shorter than those observed in [Zn(C$_4$H$_{13}$N$_3$)$_2$]$_2$ [Fe(CN)$_6$]·4H$_2$O (Razak *et al.*, 2000).

In this complex, the protonated Htren can provide numerous N—H groups for hydrogen bonding (Table 2), resulting in a three-dimensional network (Figs. 2 and 3). Each complex cation is surrounded by six adjacent complex anions and *vice versa*.

Experimental

The tren ligand (146 mg, 1 mmol) was added to a 50 ml aqueous solution of Ni(ClO$_4$)$_2$·6H$_2$O (183 mg, 0.5 mmol), forming a green solution, which was then added dropwise to a 50 ml aqueous solution of K$_4$[Fe(CN)$_6$] (183 mg, 0.5 mmol) at room temperature. The resulting solution was allowed to stand for several days to obtain red crystals of (I). Elemental analysis found: C 23.10, H 1.86, N 15.80%; calculated for C$_{18}$H$_{38}$FeN$_{14}$Ni: C 22.50, H 1.89, N 15.75%.

Crystal data

[Ni(C$_6$H$_{19}$N$_4$)$_2$][Fe(CN)$_6$]
M_r = 565.18
Triclinic, $P\overline{1}$
a = 8.6744 (17) Å
b = 8.7883 (18) Å
c = 9.3802 (19) Å
α = 92.45 (3)°
β = 99.70 (3)°
γ = 119.56 (3)°
V = 606.5 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.41 mm^{-1}
T = 293 (2) K
0.48 × 0.35 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min}$ = 0.551, $T_{\max}$ = 0.765
2632 measured reflections
2632 independent reflections
2350 reflections with $I > 2\sigma(I)$
3 standard reflections every 97 reflections intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.083
S = 0.97
2632 reflections
159 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.39 e Å^{-3}
$\Delta\rho_{\min}$ = −0.67 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N1	2.0956 (19)	Fe1—C9	1.907 (2)
Ni1—N2	2.1369 (17)	Fe1—C7	1.912 (2)
Ni1—N3	2.1558 (18)	Fe1—C8	1.906 (2)
N1—Ni1—N2	91.77 (8)	N2—Ni1—N3	81.67 (7)
N1—Ni1—N3	82.23 (7)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···N7^{i}	0.90	2.06	2.946 (3)	168
N1—H1*B*···N6	0.90	2.20	3.014 (3)	151
N2—H2*A*···N5^{ii}	0.90	2.30	3.153 (3)	158
N2—H2*B*···N7^{iii}	0.90	2.46	3.307 (3)	157
N4—H4*A*···N6^{iv}	0.89	1.92	2.791 (3)	166
N4—H4*B*···N5^{v}	0.89	1.94	2.792 (3)	161
N4—H4*C*···N7^{vi}	0.89	2.27	3.016 (3)	141

H atoms were placed at calculated positions (C—H = 0.93–0.97 Å, N—H = 0.89–0.90 Å) and refined as riding with $U_{\rm iso}$(H) = 1.2 $U_{\rm eq}$(carrier) or 1.5$U_{\rm eq}$(N).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997);

Acta Cryst. (2007). E**63**, m1208–m1210 Yao *et al.* • [Ni(C$_6$H$_{19}$N$_4$)$_2$][Fe(CN)$_6$] **m1209**

molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXTL* (Siemens, 1995).

References

Barkigia, K., Nelson, N., Renner, M., Smith, K. M. & Fajer, J. (1999). *J. Phys. Chem. B*, **103**, 8643–8646.

Brinker, U. H. & König, L. (1979). *J. Am. Chem. Soc.* **101**, 4738–4739.

Calatayud, M. L., Sletten, J., Castro, I., Julve, M., Seitz, G. & Mann, K. (2003). *Inorg. Chim. Acta*, **353**, 159–167.

Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft, The Netherlands.

Hajela, S., Johnson, A., Xu, J., Sunderland, C. J., Cohen, S. M., Caulder, D. L. & Raymond, K. N. (2001). *Inorg. Chem.* **40**, 3208–3216.

Kou, H. Z., Zhou, B. C., Liao, D. Z., Wang, R. J. & Li, Y. D. (2002). *Inorg. Chem.* **47**, 6887–6891.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Razak, I. A., Shanmuga Sundara Raj, S., Fun, H.-K., Tong, Y.-X., Lu, Z.-L. & Kang, B.-S. (2000). *Acta Cryst.* C**56**, 291–292.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

Siemens (1994). *XP*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1995). *SHELXTL*. Version 5.0, Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

m1210 Yao *et al.* • $[Ni(C_6H_{19}N_4)_2][Fe(CN)_6]$ *Acta Cryst.* (2007). E**63**, m1208–m1210