

N11—Cu1—N14	169.1 (4)	Cu2—C1—N1	176.1 (6)
N11—Cu1—N13	92.4 (4)	Cu2—C2—N2	175.8 (8)
N11—Cu1—N12	80.2 (3)	N3—C3—S3	178.1 (9)

Table 3. *Hydrogen-bonding geometry* (Å, °)

<i>D</i>	<i>H</i>	<i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
O2	H21	O1	0.80 (1)	2.33 (1)	2.717 (7)	111 (1)
O2	H22	N3	1.12 (2)	1.74 (2)	2.854 (10)	173 (1)
O1	H11	N2 ⁱ	0.73 (1)	2.23 (2)	2.822 (8)	139 (2)
O1	H12	O2 ⁱⁱ	0.89 (1)	2.29 (2)	2.818 (9)	118 (1)

Symmetry codes: (i) $x, y, z - 1$; (ii) $-x - 1, -y + 1, -z + 1$.

Intensities were corrected for Lorentz and polarization factors using *XP21* (Pavelčík, 1986). The structure was solved by direct methods with *XFPS* (Pavelčík, Rizzoli & Andreetti, 1990) and subsequent Fourier syntheses using *SHELX76* (Sheldrick, 1976). Anisotropic displacement parameters were refined for all non-H atoms. All H atoms of water and phenanthroline molecules except H123 were located from difference Fourier maps and refined with isotropic displacement parameters fixed to U_{eq} of the respective bonded O or C atoms plus 0.01 \AA^2 . The position of H123 was calculated and then refined as for the other H atoms. At the final stage of the refinement, $(\Delta\sigma)_{max}$ was smaller than 0.1 for non-H atoms (0.446 for H atoms). Geometric analysis was performed using *PARST* (Nardelli, 1983). *ORTEP* (Johnson, 1965) and *MOLDRAW* (Ugliengo, Borzani & Viterbo, 1988) were used to draw the structure.

The authors thank Mrs. Danková for assistance with the synthetic work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71552 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: VS1008]

References

- Anderson, O. P. (1973). *J. Chem. Soc. Dalton Trans.* pp. 1237–1241.
- Anderson, O. P. (1975). *Inorg. Chem.* **14**, 730–734.
- Dunaj-Jurčo, M. (1993). In preparation.
- Dunaj-Jurčo, M., Kabešová, M., Kettmann, V., Čišařová, I. & Mikloš, D. (1993). *Acta Cryst.* **C49**, 1476–1479.
- Dunaj-Jurčo, M., Potočnák, I., Cibik, J., Kabešová, M., Kettmann, V. & Mikloš, D. (1993). *Acta Cryst.* **C49**, 1479–1482.
- Harrison, W. D. & Hathaway, B. J. (1980). *Acta Cryst.* **B36**, 1069–1074.
- Jeter, D. Y., Casteel, W. J. Jr, Condren, S. M., Hobson, A. M., Stiles, T. E. & Cordes, A. W. (1988). *Acta Cryst.* **C44**, 1303–1305.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Marriquer, V., Brito, I., Andrade, A., Wittke, O., von Schnering, H. G. & Peters, K. (1988). *Acta Cryst.* **C44**, 1191–1193.
- Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
- Pavelčík, F. (1986). *Program XP21*. Comenius Univ., Pharmaceutical Faculty, Bratislava, Slovakia.
- Pavelčík, F., Rizzoli, C. & Andreetti, G. D. (1990). *XFPS. A Program for Automatic Structure Determination by Fourier, Patterson and Superposition Methods*. MS-DOS PC version, November 1990. Univ. of Parma, Italy, and J. A. Komenský Univ., Bratislava, Slovakia.

- Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
- Stephens, F. S. (1972). *J. Chem. Soc. Dalton Trans.* pp. 1350–1353.
- Tyagi, S. & Hathaway, B. J. (1981). *J. Chem. Soc. Dalton Trans.* pp. 2029–2053.
- Ugliengo, P., Borzani, G. & Viterbo, D. (1988). *J. Appl. Cryst.* **21**, 75.

Acta Cryst. (1994). **C50**, 383–386

A Binuclear Silver Complex of a Tetrapyrazolyl Ligand

WILLIAM CLEGG,* PAUL J. COOPER, JOYCE C. LOCKHART AND DAVID J. RUSHTON

Department of Chemistry, University of Newcastle, Newcastle upon Tyne NE1 7RU, England

(Received 5 August 1993; accepted 7 September 1993)

Abstract

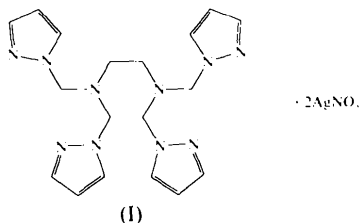
Silver(I) nitrate forms a binuclear complex with the potentially multidentate ligand *N,N',N'',N'''*-tetrakis(1-pyrazolylmethyl)-1,2-ethanediamine, [μ -*N,N',N'',N'''*-tetrakis(1-pyrazolyl- κ *N*²-methyl)-1,2-ethanediamine]-disilver(I) dinitrate, $[\text{Ag}_2(\text{C}_{18}\text{H}_{24}\text{N}_{10})]2\text{NO}_3$. Each Ag ion is linearly coordinated by two pyrazole *N*² atoms. The aliphatic amine *N* atoms of the ligand play no primary coordination role. The two Ag ions are separated by 3.1599 (9) Å. There are additional weak interactions with the nitrate anions.

Comment

Polynuclear Ag complexes are currently of interest in redox chemistry (Che, Yip, Li, Peng, Lee, Wang & Liu, 1991; Gubelmann, Harriman, Lehn & Sessler, 1988) and provide experimental data with which to test bonding theories (Cotton, Feng, Matusz & Poli, 1988; Perreault, Drouin, Michel & Harvey, 1993). Our own investigations into force fields for molecular modelling of Ag chelates (Lockhart & Rushton, 1991) found crystallographic data on linear complexes of Ag ions with aromatic *N* ligands (related to imidazole, benzimidazole, pyrazole, *etc.*) to be scarce.

The tetrakis(1-pyrazolylmethyl)-1,2-ethanediamine ligand was first synthesized by Driessen (1982). It has been shown to form 1:1 complexes with most first-row transition-metal divalent ions (Hulsbergen, Driessen, Reedijk & Verschoor, 1984). In these complexes, the ligand is hexadentate, coordinating through both aliphatic amine *N* atoms and one *N* atom of each pyrazole ring. The crystal structure of the complex with $\text{Mn}(\text{ClO}_4)_2$

has been reported (Hulsbergen, Driessen, Reedijk & Verschoor, 1984). The Mn atom is seven-coordinate, with an additional bond to one perchlorate O atom. Complexes of Fe, Cd and Zn appear to be isostructural, while Co, Ni and Cu complexes are probably octahedrally coordinated, without the additional bond.



The Ag complex (I) reported here has a completely different structure (Fig. 1), with a 2:1 rather than a 1:1 metal-ligand stoichiometry. Each of the two Ag ions is coordinated by two pyrazole N atoms, with Ag—N bond lengths in the range 2.136 (4)–2.166 (5) Å. The coordination is essentially linear [N—Ag—N angles 174.3 (2) and 169.2 (2)°], as is commonly found for silver(I). There are, however, some secondary interactions approximately in an equatorial belt for each Ag ion, of types Ag···O, Ag···Ag and Ag···N.

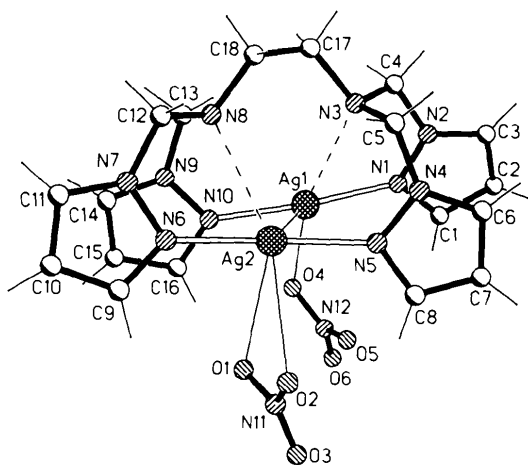


Fig. 1. Structure of the disilver complex. Primary coordination is shown by thick bonds, secondary interactions by thin and dashed bonds.

Each Ag ion interacts weakly and asymmetrically with two O atoms of one of the nitrate anions. The degree of asymmetry is different for the two ion pairs: Ag1—O4 2.725 (5) and Ag1···O5 3.239 (11) Å; Ag2—O1 2.839 (6) and Ag2—O2 2.924 (6) Å. These distances are too great to be considered normal metal-ligand bonds. Silver nitrate itself has Ag—O contacts in the range 2.48–2.99 Å and these are regarded as largely ionic (Lindley & Woodward, 1966).

The Ag···Ag separation of 3.1599 (9) Å is less than the van der Waals contact distance of 3.44 Å (Bondi, 1964) but considerably greater than the closest contact of 2.88 Å in Ag metal (Wells, 1984). Cotton, Feng, Matusz & Poli (1988) concluded from theoretical studies that at such distances there is very little direct metal-metal bonding. Symmetrical binuclear Ag complexes with Ag···Ag separations even as great as the van der Waals contact distance, however, do display Raman frequencies which have been attributed to the Ag—Ag stretching vibration; correlation of the observed frequencies with Ag···Ag separations has been attempted for a range of binuclear Ag chelates (Perreault, Drouin, Michel & Harvey, 1993).

The aliphatic amine N atoms of the ligand play no primary role in the coordination of Ag; the closest Ag···N distances are 2.906 and 2.859 Å (each aliphatic N atom being positioned closer to one Ag ion than the other), which are considerably greater than the primary Ag—N bond lengths.

Ag complexes of other N ligands display features similar to some of those found here. A binuclear complex of pyrazine has two-coordinate Ag somewhat distorted from linearity and has secondary Ag···O interactions with nitrate ions (2.72–2.94 Å), but no Ag···Ag interactions (Vranka & Amma, 1966). In binuclear complexes of naphthyridine (Munakata, Maekama, Kitegawa, Adachi & Masuda, 1990), 1,2,4-triazole and 2-aminoquinoline (Schmidbauer, Mair, Müller, Lachmann & Gamper, 1991), unidentate coordination of a nitrate or perchlorate anion to Ag is weak but significant and distorts the primary linear coordination towards trigonal, with a correlation among Ag—N and Ag—O distances and the N—Ag—N angle. In complexes containing two or more silver(I) atoms with a primary linear or almost linear coordination by two N atoms, Ag···Ag distances have been found ranging upwards from 2.65 Å, many of them considerably shorter than that in the present structure and interpreted as direct metal-metal bonding (Beck & Strähle, 1986; Hass & Bergerhoff, 1974; Hartmann & Strähle, 1988, 1990; Eastland, Mazid, Russell & Symons, 1980; Lee & Peng, 1991; Fenske, Baum, Zinn & Dehnicke, 1990; Tsuda, Ohba, Takahashi & Ito, 1989; Hartmann, Schmid & Strähle, 1989; van Stein, van Koten, Blank, Taylor, Vrieze, Spek, Duisenberg, Schreurs, Kojic Prodic & Brevard, 1985; Cotton, Feng, Matusz & Poli, 1988; Munakata, Maekama, Kitagawa, Adachi & Masuda, 1990).

Although the structure of only one complex of the tetrakis(1-pyrazolylmethyl)-1,2-ethanediamine ligand has been reported previously, there are other similar ligands in which a pyridine or imidazole ring takes the place of pyrazole. In principle, these have the same multidentate capability and several structures are known in which all six N atoms do indeed coordinate, either to a single Cu, Ni, Fe or Co ion (Birker, Hendriks, Reedijk & Verschoor, 1981; Holt, Piggott, Hursthouse & Short,

1987; Chang, McCusker, Toftlund, Wilson, Trautwein, Winkler & Hendrickson, 1990; Mandel & Douglas, 1989) or to two Cu, Mo, V, Cr or Mn ions (Gagne, Kreh, Dodge, Marsh & McCool, 1982; Birker, Hendriks & Reedijk, 1981; Hendriks, Birker, van Rijn, Verschoor & Reedijk, 1982; Carr, Piggott & Wong, 1986; Neves, Wieghardt, Nuber & Weiss, 1988; Toftlund, Simonsen & Pedersen, 1990; Pal, Gohdes, Wilisch & Armstrong, 1992; Driessen, Haanstra & Reedijk, 1992).

The previously reported structure most closely related to the present Ag complex is that of a binuclear copper(I) complex of a tetrakis(benzimidazolyl) ligand similar to the ligand here (Cagnon, Hubert, Rivest & Beauchamp, 1977). It has an N—Cu—N angle of 170.9° and the Cu···Cu distance of 3.043 Å was regarded as a possible model for the copper(I) coordination in deoxyhaemocyanin.

Experimental

The ligand was prepared by the method of Driessen (1982). Mixing a concentrated aqueous solution of the ligand (0.11 g, 0.289 mmol) and a concentrated aqueous solution of silver nitrate (0.0978 g, 0.0579 mmol) gave an immediate precipitate. Colourless crystals of the title complex were deposited slowly by the supernatant liquid. Satisfactory elemental analysis results were obtained.

Crystal data

[Ag₂(C₁₈H₂₄N₁₀)]₂NO₃
M_r = 720.23
 Triclinic
P $\bar{1}$
a = 9.507 (2) Å
b = 9.865 (2) Å
c = 13.262 (3) Å
 α = 98.774 (14)°
 β = 93.82 (2)°
 γ = 97.97 (2)°
V = 1212.5 (4) Å³
Z = 2

D_x = 1.973 Mg m⁻³
 Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 32 reflections
 θ = 10.11–11.46°
 μ = 1.678 mm⁻¹
T = 295 (2) K
 Block
 0.38 × 0.18 × 0.14 mm
 Colourless

Data collection

Stoe Siemens diffractometer
 ω/θ scans with on-line profile fitting (Clegg, 1981)
 Absorption correction: empirical
 $T_{\min} = 0.596$, $T_{\max} = 0.645$
 5747 measured reflections
 4286 independent reflections

3456 observed reflections
 $[I > 2\sigma(I)]$
 $R_{\text{int}} = 0.0353$
 $\theta_{\text{max}} = 25.02^\circ$
 $h = -11 \rightarrow 11$
 $k = -11 \rightarrow 11$
 $l = -15 \rightarrow 15$
 3 standard reflections
 frequency: 60 min
 intensity variation: 4%

Refinement

Refinement on *F*²
R(F) = 0.0399

$\Delta\rho_{\text{max}} = 0.633 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.461 \text{ e } \text{Å}^{-3}$

$wR(F^2) = 0.1138$
 $S = 1.072$
 4272 reflections
 344 parameters
 H atoms riding
 Calculated weights
 $w = 1/[\sigma^2(F_o^2) + (0.0176P)^2 + 2.0375P]$,
 where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction:
SHELXL (Sheldrick, in preparation)
 Extinction coefficient:
 0.0048 (5)
 Atomic scattering factors from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Ag1	0.51576 (4)	0.42152 (4)	0.35997 (3)	0.0650 (2)
Ag2	0.60328 (5)	0.49061 (4)	0.14564 (3)	0.0648 (2)
N1	0.3272 (5)	0.2716 (5)	0.3596 (3)	0.0640 (12)
N2	0.1971 (5)	0.2963 (5)	0.3271 (3)	0.0636 (12)
N3	0.2704 (4)	0.4957 (4)	0.2493 (4)	0.0600 (11)
N4	0.3052 (4)	0.3013 (4)	0.1225 (3)	0.0544 (10)
N5	0.4491 (4)	0.3072 (4)	0.1262 (3)	0.0578 (10)
N6	0.7518 (5)	0.6740 (5)	0.1344 (4)	0.0639 (12)
N7	0.7240 (5)	0.8022 (5)	0.1667 (3)	0.0661 (12)
N8	0.5101 (5)	0.7358 (5)	0.2448 (4)	0.0645 (12)
N9	0.6969 (4)	0.7185 (4)	0.3741 (3)	0.0545 (10)
N10	0.6967 (4)	0.5814 (4)	0.3742 (3)	0.0536 (10)
C1	0.3089 (7)	0.1380 (6)	0.3711 (5)	0.075 (2)
C2	0.1678 (8)	0.0803 (8)	0.3475 (5)	0.086 (2)
C3	0.0989 (7)	0.1839 (8)	0.3205 (5)	0.081 (2)
C4	0.1742 (6)	0.4339 (7)	0.3152 (5)	0.071 (2)
C5	0.2421 (6)	0.4282 (6)	0.1432 (5)	0.0682 (15)
C6	0.2401 (7)	0.1707 (6)	0.0974 (4)	0.0658 (15)
C7	0.3418 (7)	0.0870 (6)	0.0856 (4)	0.0683 (15)
C8	0.4700 (7)	0.1745 (6)	0.1039 (4)	0.0645 (14)
C9	0.8892 (6)	0.6912 (7)	0.1187 (5)	0.071 (2)
C10	0.9483 (8)	0.8293 (8)	0.1393 (5)	0.085 (2)
C11	0.8393 (8)	0.8970 (7)	0.1689 (5)	0.084 (2)
C12	0.5796 (7)	0.8259 (6)	0.1817 (5)	0.076 (2)
C13	0.5641 (6)	0.7722 (6)	0.3515 (5)	0.068 (2)
C14	0.8285 (6)	0.7896 (6)	0.3979 (4)	0.0636 (14)
C15	0.9178 (6)	0.6967 (7)	0.4132 (4)	0.069 (2)
C16	0.8329 (6)	0.5690 (6)	0.3972 (4)	0.0617 (13)
C17	0.2565 (6)	0.6453 (6)	0.2629 (7)	0.091 (2)
C18	0.3544 (7)	0.7390 (7)	0.2285 (8)	0.103 (3)
N11	0.8398 (5)	0.2877 (5)	0.0882 (4)	0.0652 (12)
O1	0.8508 (8)	0.3635 (8)	0.1709 (5)	0.147 (3)
O2	0.7736 (8)	0.3193 (8)	0.0191 (4)	0.143 (3)
O3	0.8891 (7)	0.1829 (6)	0.0838 (5)	0.129 (2)
N12	0.7057 (5)	0.1800 (5)	0.4255 (5)	0.0734 (13)
O4	0.6933 (7)	0.2925 (5)	0.4699 (4)	0.106 (2)
O5	0.6751 (13)	0.1502 (10)	0.3364 (6)	0.206 (5)
O6	0.7513 (14)	0.1038 (7)	0.4659 (9)	0.267 (7)

Table 2. Selected geometric parameters (Å, °)

Ag1—N10	2.144 (4)	Ag2—N6	2.166 (5)
Ag1—N1	2.159 (4)	Ag1—Ag2	3.1599 (9)
Ag2—N5	2.136 (4)		
N10—Ag1—N1	174.3 (2)	N5—Ag2—N6	169.2 (2)

Refinement was on *F*² for all reflections except for 14 with very negative *F*_o². Isotropic H atoms were constrained to give C—H 0.93 (aromatic, on ring-angle external bisectors) or 0.97 Å (aliphatic, with H—C—H 109.5°) and *U*(H) = 1.2*U*_{eq}(C). All e.s.d.'s were estimated using the full covariance matrix. Data collection: *DIF4* (Stoe & Cie, 1988). Cell refinement: *DIF4*. Data reduction: local programs. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to re-

fine structure: *SHELXL93* (Sheldrick, in preparation). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93* and local programs.

We thank the SERC for financial support, including research studentships to PJC and DJR, and ICI for a CASE award to PJC.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71611 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1072]

References

- Beck, J. & Strähle, J. (1986). *Z. Naturforsch. Teil B*, **41**, 4–9.
- Birker, P. J. M. W. L., Hendriks, H. M. J. & Reedijk, J. (1981). *Inorg. Chim. Acta*, **55**, L17–L18.
- Birker, P. J. M. W. L., Hendriks, H. M. J., Reedijk, J. & Verschoor, G. C. (1981). *Inorg. Chem.* **20**, 2408–2414.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- Cagnon, C., Hubert, J., Rivest, R. & Beauchamp, A. L. (1977). *Inorg. Chem.* **16**, 2469–2473.
- Carr, P., Piggott, B. & Wong, S. F. (1986). *Inorg. Chim. Acta*, **122**, 221–224.
- Chang, H.-R., McCusker, J. K., Toftlund, H., Wilson, S. R., Trautwein, A. X., Winkler, H. & Hendrickson, D. N. (1990). *J. Am. Chem. Soc.* **112**, 6814–6827.
- Che, C.-M., Yip, H.-K., Li, D., Peng, S.-M., Lee, G.-H., Wang, Y.-M. & Liu, S.-T. (1991). *J. Chem. Soc. Chem. Commun.* pp. 1615–1617.
- Clegg, W. (1981). *Acta Cryst.* **A37**, 22–28.
- Cotton, F. A., Feng, X., Matusz, M. & Poli, R. (1988). *J. Am. Chem. Soc.* **110**, 7077–7083.
- Driessen, W. L. (1982). *Recl Trav. Chim. Pays-Bas*, **101**, 441–443.
- Driessen, W. L., Haanstra, W. G. & Reedijk, J. (1992). *Acta Cryst.* **C48**, 1585–1587.
- Eastland, G. W., Mazid, M. A., Russell, D. R. & Symons, M. C. R. (1980). *J. Chem. Soc. Dalton Trans.* pp. 1682–1687.
- Fenske, D., Baum, G., Zinn, A. & Dehnicke, K. (1990). *Z. Naturforsch. Teil B*, **45**, 1273–1278.
- Gagne, R. R., Kreh, R. P., Dodge, J. A., Marsh, R. E. & McCool, M. (1982). *Inorg. Chem.* **21**, 254–261.
- Gubelmann, M., Harriman, A., Lehn, J.-M. & Sessler, J. I. (1988). *J. Chem. Soc. Chem. Commun.* pp. 77–79.
- Hartmann, E., Schmid, R. & Strähle, J. (1989). *Z. Naturforsch. Teil B*, **44**, 778–785.
- Hartmann, E. & Strähle, J. (1988). *Z. Naturforsch. Teil B*, **43**, 525–528.
- Hartmann, E. & Strähle, J. (1990). *Z. Anorg. Allg. Chem.* **583**, 31–40.
- Hass, D. & Bergerhoff, G. (1974). *Acta Cryst.* **B30**, 1361–1362.
- Hendriks, H. M. J., Birker, P. J. M. W. L., van Rijn, J., Verschoor, G. C. & Reedijk, J. (1982). *J. Am. Chem. Soc.* **104**, 3607–3617.
- Holt, S. D., Piggott, B., Hursthouse, M. B. & Short, R. L. (1987). *Polyhedron*, **6**, 1457–1461.
- Hulsbergen, F. B., Driessen, W. L., Reedijk, J. & Verschoor, G. C. (1984). *Inorg. Chem.* **23**, 3588–3592.
- Lee, C.-F. & Peng, S.-M. (1991). *J. Chin. Chem. Soc. (Taipei)*, **38**, 559–564.
- Lindley, P. F. & Woodward, P. (1966). *J. Chem. Soc. A*, pp. 123–126.
- Lockhart, J. C. & Rushton, D. J. (1991). *J. Chem. Soc. Dalton Trans.* pp. 2633–2638.
- Mandel, J. B. & Douglas, B. E. (1989). *Inorg. Chim. Acta*, **155**, 55–69.
- Munakata, M., Maekama, M., Kitegawa, S., Adachi, M. & Masuda, H. (1990). *Inorg. Chim. Acta*, **167**, 181–188.
- Neves, A., Wieghardt, C., Nuber, B. & Weiss, J. (1988). *Inorg. Chim. Acta*, **150**, 183–187.
- Pal, S., Gohdes, J. W., Wilisch, W. C. A. & Armstrong, W. H. (1992). *Inorg. Chem.* **31**, 713–716.
- Perreault, D., Drouin, M., Michel, A. & Harvey, P. D. (1993). *Inorg. Chem.* **32**, 1903–1912.
- Schmidbaur, H., Mair, A., Müller, G., Lachmann, J. & Gamper, S. (1991). *Z. Naturforsch. Teil B*, **46**, 912–918.
- Sheldrick, G. M. (1990). *SHELXTL/PC User's Manual*. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.
- Stein, G. C. van, van Koten, G., Blank, F., Taylor, L. C., Vrieze, K., Spek, A. L., Duisenberg, A. J. M., Schreurs, A. M. M., Kojic Prodic, B. & Brevard, C. (1985). *Inorg. Chim. Acta*, **98**, 107–120.
- Stoe & Cie (1988). *DIF4. Diffractometer Control Program*. Version 7.04. Stoe & Cie, Darmstadt, Germany.
- Toftlund, H., Simonsen, O. & Pedersen, E. (1990). *Acta Chem. Scand.* **44**, 676–682.
- Tsuda, T., Ohba, S., Takahashi, M. & Ito, M. (1989). *Acta Cryst.* **C45**, 887–890.
- Vranka, R. G. & Amma, E. L. (1966). *Inorg. Chem.* **5**, 1020–1025.
- Wells, A. F. (1984). *Structural Inorganic Chemistry*, 5th ed., p. 1279. Oxford: Clarendon Press.

Acta Cryst. (1994). **C50**, 386–388

Synthesis and Structure of the New Complex Et₃NH[Fe(tben)]

IVO VENCATO AND ADEMIR NEVES

Depto. Física and Depto. Química – UFSC,
88040-900 Florianópolis, SC, Brazil

BEVERLY R. VINCENT

MSC, 3200A Research Forest Drive, The Woodlands,
Texas 77381-4238, USA

CLAUDIA ERASMUS-BUHR AND WOLFGANG HAASE

Institut für Physikalische Chemie, THD,
D-6100 Darmstadt, Germany

(Received 26 May 1993; accepted 15 September 1993)

Abstract

Triethylammonium (2,2',2'',2'''-{1,2-ethanediylbis[nitri-*l*obis(methylene)]}tetraphenolato-*N,N',O,O',O'',O'''*-ferrate(III)), [NH(C₂H₅)₃][Fe(C₃₀H₂₈N₂O₄)], has been synthesized and its structure determined by X-ray diffraction. The anion, [Fe(tben)]⁻, shows pseudo-octahedral geometry in which the hexadentate ligand is coordinated to the high-spin ferric center by four phenolate O and two N donor atoms in facial NO₂ arrangements. The O(1)—Fe—O(2) and the N(1)—Fe—N(2) bond angles of 103.6 (1) and 80.7 (1)°, respectively, deviate significantly from 90° as a result of the large bite angle required by the