$a = 9.247$ (2) Å
$b = 13.434$ (6) Å
$c = 16.367$ (3) Å
$\beta = 105.60$ (2)°
$V = 1958$ (1) Å^3
$Z = 4$
$D_x = 1.60$ Mg m^{-3}
$D_m = 1.59$ (1) Mg m^{-3}

$\mu = 2.066$ mm^{-1}
$T = 293$ K
Plate
0.20 × 0.20 × 0.15 mm
Yellow

Data collection

Rigaku AFC-5*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.87$, $T_{\max} = 1.00$
3217 measured reflections
2565 independent reflections
1997 observed reflections [$F > 4\sigma(F)$]

$R_{\rm int} = 0.018$
$\theta_{\max} = 30.00°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 18$
$l = -23 \rightarrow 22$
3 standard reflections monitored every 150 reflections
intensity decay: 1.3%

Refinement

Refinement on *F*
$R = 0.0431$
$wR = 0.0443$
$S = 1.172$
1997 reflections
157 parameters
All H-atom parameters refined except for H9 and H10, for which only coordinates were refined
$w = 1/[\sigma^2(F_o) + 0.0003F_o^2]$
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.81$ e Å^{-3}
$\Delta\rho_{\min} = -0.79$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELX*76 (Sheldrick, 1976) (C, H, N, O, ON) and *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B) (Cd, Ni)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cd	0	0.51843 (4)	1/4	0.0310 (2)
Ni	1/2	1/2	1/2	0.0287 (3)
O	1/2	0.3133 (6)	1/4	0.076 (4)
ON	0.0521 (5)	0.3778 (4)	0.1765 (4)	0.058 (2)
N1	0.2439 (5)	0.5193 (4)	0.3420 (3)	0.051 (2)
N2	0.7006 (5)	0.4357 (4)	0.3921 (3)	0.058 (2)
N3	0.0895 (6)	0.6249 (4)	0.1669 (3)	0.043 (2)
C1	0.3423 (5)	0.5130 (3)	0.4019 (3)	0.035 (1)
C2	0.6271 (5)	0.4615 (3)	0.4340 (3)	0.038 (2)
C3	0.0011 (7)	0.6501 (4)	0.0802 (4)	0.046 (2)
C4	0.0685 (7)	0.7324 (4)	0.0387 (4)	0.048 (2)
C5	0.2166 (8)	0.7077 (4)	0.0202 (4)	0.047 (2)
C6	0.0635 (9)	0.2905 (5)	0.2302 (7)	0.087 (4)

Table 4. *Selected geometric parameters (Å, °) for (2)*

Cd—N1	2.352 (4)	C3—C4	1.517 (7)
Cd—N3	2.277 (5)	C4—C5	1.516 (8)
Cd—ON	2.359 (5)	C5—C5$^{\rm i}$	1.526 (9)
Ni—C1	1.865 (5)	ON—C6	1.452 (10)
Ni—C2	1.870 (5)	C6—C6$^{\rm ii}$	1.486 (15)
C1—N1	1.146 (6)	O···N2$^{\rm iii}$	3.038 (6)
C2—N2	1.142 (6)	O···N3$^{\rm iv}$	3.090 (8)
N3—C3	1.474 (7)	N2···ON$^{\rm iii}$	2.911 (8)
N1—Cd—N3	86.5 (2)	Ni—C1—N1	178.6 (4)
N1—Cd—ON	92.2 (2)	Ni—C2—N2	177.5 (4)
N3—Cd—ON	92.4 (2)	N3—C3—C4	114.3 (5)
C1—Ni—C2	89.8 (2)	C3—C4—C5	115.6 (5)
Cd—N1—C1	161.9 (4)	C4—C5—C5$^{\rm i}$	114.6 (5)
Cd—N3—C3	121.5 (4)	ON—C6—C6$^{\rm ii}$	109.1 (6)
Cd—ON—C6	109.3 (5)		

Symmetry codes: (i) $\frac{1}{2}-x, \frac{3}{2}-y, -z$; (ii) $-x, y, \frac{1}{2}-z$; (iii) $1-x, y, \frac{1}{2}-z$; (iv) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$.

All calculations were carried out on a HITAC M-680H computer at the Institute for Molecular Science, Okazaki.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; program(s) used to solve structures: *SHELX*76 (Sheldrick, 1976); program(s) used to refine structures: *SHELX*76; molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1281). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$[\{(C_5Me_4H)Fe(C_5Me_4)CH_2C_5H_4\}_2Fe]^{2+}.-[C_2(CN)_4]^-.[C_3(CN)_5]^-$

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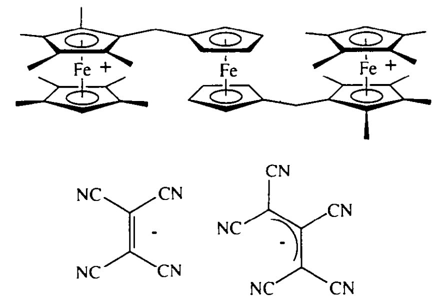
Abstract

The structure of the organometallic charge-transfer salt 1,1′-bis[(octamethylferroceniumyl)methyl]ferrocene tet-

racyanoethenide pentacyanopropenide, $[Fe\{(C_9H_{13})Fe(C_9H_{12})CH_2C_5H_4\}_2][C_2(CN)_4][C_3(CN)_5]$, has been determined; the asymmetric unit contains the 1,1′-bis[(octamethylferroceniumyl)methyl]ferrocene dication, one tetracyanoethenide ($TCNE^-$) radical anion and one pentacyanopropenide ($PCNP^-$) anion. The structure analysis shows that the dication adopts an unusual conformation and that, as expected, the octamethylferrocene units are oxidized, and the central ferrocene unit is not.

Comment

The radical cation–radical anion salts formed by redox-active metallocenes with polycyano acceptors have attracted considerable interest since the discovery of ferromagnetism in decamethylferrocenium tetracyanoethenide (Miller & Epstein, 1994). In a study of tetracyanoethenide salts of trimetallocene species, we prepared $[\{(C_5Me_4H)Fe(C_5Me_4)CH_2C_5H_4\}_2Fe][TCNE]_2$. On recrystallization, in addition to iridescent purple–green plate-like microcrystals, shown by magnetic susceptibility, IR and microanalytical data to be $[\{(C_5Me_4H)Fe(C_5Me_4)CH_2C_5H_4\}_2Fe][TCNE]_2$, a small number of green single crystals were obtained. X-ray crystallography revealed these crystals to be the title compound, (I). Useful structures for comparison are those of octamethylferrocene (Struchkov, Adrianov, Sal'nikova, Lyatifov & Materikova, 1978), decamethylferrocene (Struchkov, Adrianov, Sal'nikova, Lyatifov & Materikova, 1978; Freyberg, Robbins, Raymond & Smart, 1979), $[Fe(C_5Me_5)_2][TCNE]$ and $[Fe(C_5Me_5)_2][PCNP]$ (Miller *et al.*, 1987), and $[Fe(C_5Me_4H)_2][TCNE]$ (Miller *et al.*, 1989).



(I)

In the dication, the Fe—C distances for the terminal octamethylferrocenyl groups range from 2.060 (7) to 2.112 (6) Å, with an average of 2.086 Å. Similar values have been found for octamethylferrocenium (average 2.084 Å) and decamethylferrocenium ions (average 2.09 Å), whereas octamethylferrocene and decamethylferrocene have average Fe—C distances of 2.054 and 2.050 Å, respectively. The Fe—C distances of the central ferrocene range from 2.023 (6) to 2.039 (5) Å, with an average of 2.029 Å; these values are indicative of an unoxidized ferrocene moiety (Dunitz, Orgel & Rich, 1956), rather than a ferrocenium ion (Sullivan & Foxman, 1983).

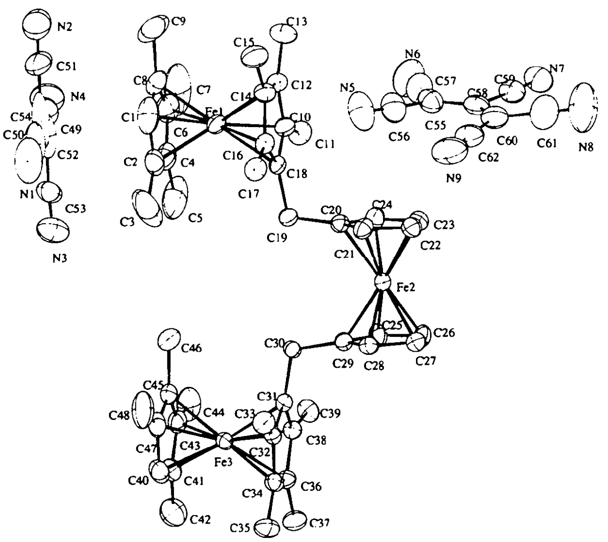


Fig. 1. View of the structure of $[Fe\{(C_5Me_4H)Fe(C_5Me_4)CH_2C_5H_4\}_2][TCNE][PCNP]$. Displacement ellipsoids are shown at the 50% level; H atoms are omitted for clarity.

The conformation of the cation is remarkable and may be contrasted with the range of conformations found for related trimetallocenes and trimetallocenium salts (Barlow, Murphy, Evans & O'Hare, 1995; Pannell *et al.*, 1994; Lough, Manners & Rulkens, 1994). The two bridging substituents on the central ferrocene are only 2.23° away from being perfectly eclipsed. Presumably the adoption of this conformation is due to the packing requirements of the counterions.

The structural parameters of the pentacyanopropenide anion are similar to those reported by Miller *et al.* (1987) for $[Fe(C_5Me_5)_2][PCNP]$. However, some C—C bond lengths and some angles in the $TCNE^-$ ion differ significantly from those found in other $TCNE^-$ salts (Miller *et al.*, 1987, 1989). Most notably, the C═C bond length of 1.13 (2) Å contrasts to corresponding values of 1.392 (9) and 1.385 (6) Å in $[Fe(C_5Me_5)_2][TCNE]$ and $[Fe(C_5Me_4H)_2][TCNE]$, respectively. Fourier maps indicate this anomaly may be due to a degree of disorder whereby a small proportion of the anions are coplanar with the majority, but rotated by approximately 90°. However, this disorder could not be satisfactorily resolved; the disorder was therefore modelled by refining the coordinates and displacement parameters for each of six C and four N atoms, each representing a composite of the sites of the different orientations believed to be present.

The origin of the pentacyanopropenide ion is unclear but it may arise from the presence of adventitious oxygen or water. Several related conversions of TCNE and its compounds have been reported: basic hydrolysis of neutral TCNE yields $PCNP^-$ salts (Middleton, Little, Coffman & Engelhardt, 1958), the exposure of $Fe(C_5H_5)_2$.TCNE to air yields a mixture of products including $[Fe(C_5H_5)_2]$[PCNP] (Rosenblum, Fish & Bennett, 1964), and the recrystallization of $[Fe(C_5Me_5)_2]$[TCNE] in air yields $[Fe(C_5Me_5)_2]$[PCNP] (Miller *et al.*, 1987).

Experimental

The synthesis of $[\{(C_5Me_4H)Fe(C_5Me_4)CH_2C_5H_4\}_2Fe]$-$[TCNE]_2$ has been described elsewhere (Barlow, Murphy, Evans & O'Hare, 1995). Layering of a tetrahydrofuran solution of this material with diethyl ether afforded microcrystalline $[\{(C_5Me_4H)Fe(C_5Me_4)CH_2C_5H_4\}_2Fe][TCNE]_2$ and a small number of single crystals of the title compound. Suitable crystals were sealed in Lindemann capillaries under a nitrogen atmosphere.

Crystal data

$[Fe(C_{24}H_{31}Fe)_2](C_6N_4)$-$(C_8N_5)$
$M_r = 1100.77$
Triclinic
$P\overline{1}$
$a = 13.866$ (2) Å
$b = 14.0228$ (8) Å
$c = 15.316$ (1) Å
$\alpha = 100.160$ (6)°
$\beta = 97.712$ (9)°
$\gamma = 104.641$ (7)°
$V = 2785.8$ (5) $Å^3$
$Z = 2$
$D_x = 1.312$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 6.1$–$13.7°$
$\mu = 0.82$ mm^{-1}
$T = 293$ K
Plate
0.96 × 0.45 × 0.15 mm
Green

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.47$, $T_{max} = 0.75$
9851 measured reflections
8698 independent reflections

4869 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.015$
$\theta_{max} = 24°$
$h = -15 \rightarrow 15$
$k = -16 \rightarrow 16$
$l = -1 \rightarrow 17$
3 standard reflections
frequency: 360 min
intensity decay: 17.5%

Refinement

Refinement on F
$R = 0.051$
$wR = 0.060$
$S = 1.129$
4869 reflections
668 parameters
H-atom parameters not refined
Weights: Chebyshev polynomial (Carruthers & Watkin, 1975)

$(\Delta/\sigma)_{max} = 0.072$
$\Delta\rho_{max} = 0.48$ e $Å^{-3}$
$\Delta\rho_{min} = -0.40$ e $Å^{-3}$
Extinction correction: Larson (1967)
Extinction coefficient: 13 (5)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ($Å^2$)

$$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Fe(1)	0.13717 (6)	0.33963 (6)	0.18545 (5)	0.0446 (5)
Fe(2)	−0.04711 (5)	0.20579 (6)	0.52655 (5)	0.0392 (5)
Fe(3)	−0.46985 (5)	−0.01739 (6)	0.24597 (5)	0.0403 (4)
N(1)	−0.084 (1)	0.3568 (8)	−0.1349 (6)	0.13 (1)
N(2)	0.2089 (9)	0.335 (1)	−0.1915 (7)	0.15 (1)
N(3)	−0.1703 (6)	0.0545 (7)	−0.1484 (6)	0.107 (7)
N(4)	0.1036 (6)	0.0295 (7)	−0.2286 (6)	0.114 (8)
N(5)	0.2461 (7)	0.5575 (6)	0.5101 (6)	0.113 (9)
N(6)	0.3842 (8)	0.3156 (7)	0.5082 (6)	0.127 (7)
N(7)	0.4716 (5)	0.3825 (5)	0.7662 (5)	0.09 (1)
N(8)	0.417 (1)	0.5998 (8)	0.9263 (7)	0.16 (2)
N(9)	0.2176 (6)	0.6581 (7)	0.7212 (7)	0.114 (9)
C(1)	0.0960 (9)	0.3933 (6)	0.0735 (5)	0.08 (1)
C(2)	0.0139 (6)	0.3148 (8)	0.0837 (4)	0.075 (8)
C(3)	−0.0889 (8)	0.326 (2)	0.0954 (8)	0.15 (1)
C(4)	0.0469 (7)	0.2289 (6)	0.0763 (4)	0.071 (6)
C(5)	−0.019 (1)	0.1278 (9)	0.0818 (7)	0.13 (2)
C(6)	0.1448 (7)	0.2531 (7)	0.0631 (5)	0.078 (7)
C(7)	0.202 (1)	0.177 (1)	0.0493 (7)	0.13 (2)
C(8)	0.1758 (7)	0.3521 (9)	0.0606 (5)	0.082 (8)
C(9)	0.278 (1)	0.416 (2)	0.0478 (8)	0.16 (1)
C(10)	0.1910 (4)	0.2873 (4)	0.2963 (4)	0.047 (3)
C(11)	0.2039 (5)	0.1843 (4)	0.2961 (4)	0.057 (4)
C(12)	0.2657 (4)	0.3729 (4)	0.2866 (4)	0.051 (4)
C(13)	0.3721 (5)	0.3756 (6)	0.2754 (5)	0.072 (6)
C(14)	0.2238 (4)	0.4558 (4)	0.2938 (4)	0.050 (5)
C(15)	0.2771 (5)	0.5617 (5)	0.2897 (5)	0.070 (5)
C(16)	0.1226 (4)	0.4220 (4)	0.3082 (3)	0.044 (3)
C(17)	0.0538 (5)	0.4871 (4)	0.3232 (4)	0.055 (4)
C(18)	0.1020 (4)	0.3179 (4)	0.3103 (3)	0.040 (3)
C(19)	0.0100 (4)	0.2545 (4)	0.3367 (3)	0.043 (3)
C(20)	0.0351 (4)	0.2603 (4)	0.4362 (4)	0.043 (3)
C(21)	0.0317 (5)	0.3404 (4)	0.5050 (4)	0.052 (4)
C(22)	0.0732 (5)	0.3262 (5)	0.5891 (4)	0.058 (4)
C(23)	0.1042 (4)	0.2393 (6)	0.5732 (4)	0.058 (5)
C(24)	0.0804 (5)	0.1976 (5)	0.4787 (4)	0.056 (4)
C(25)	−0.1430 (5)	0.0646 (5)	0.5009 (4)	0.053 (4)
C(26)	−0.1156 (5)	0.1062 (6)	0.5957 (4)	0.059 (5)
C(27)	−0.1454 (5)	0.1940 (6)	0.6132 (4)	0.062 (6)
C(28)	−0.1917 (4)	0.2086 (5)	0.5325 (4)	0.052 (4)
C(29)	−0.1910 (4)	0.1294 (4)	0.4609 (4)	0.044 (3)
C(30)	−0.2442 (4)	0.1095 (4)	0.3643 (3)	0.040 (3)
C(31)	−0.3536 (4)	0.0510 (4)	0.3574 (3)	0.038 (3)
C(32)	−0.4355 (4)	0.0946 (4)	0.3655 (3)	0.041 (3)
C(33)	−0.4311 (5)	0.2028 (4)	0.3754 (4)	0.055 (4)
C(34)	−0.5229 (4)	0.0142 (4)	0.3669 (4)	0.047 (4)
C(35)	−0.6258 (5)	0.0255 (5)	0.3788 (5)	0.066 (5)
C(36)	−0.4951 (4)	−0.0766 (4)	0.3601 (4)	0.045 (3)
C(37)	−0.5614 (5)	−0.1766 (5)	0.3655 (5)	0.063 (4)
C(38)	−0.3898 (4)	−0.0540 (4)	0.3543 (4)	0.042 (3)
C(39)	−0.3279 (5)	−0.1279 (4)	0.3517 (4)	0.054 (4)
C(40)	−0.5817 (5)	−0.0305 (6)	0.1373 (4)	0.066 (6)
C(41)	−0.5566 (5)	−0.1231 (5)	0.1309 (4)	0.061 (4)
C(42)	−0.6301 (7)	−0.2254 (6)	0.1248 (6)	0.097 (7)
C(43)	−0.4538 (5)	−0.1051 (5)	0.1246 (4)	0.056 (4)
C(44)	−0.3977 (7)	−0.1824 (7)	0.1133 (5)	0.085 (7)
C(45)	−0.4165 (5)	−0.0010 (6)	0.1267 (4)	0.056 (5)
C(46)	−0.3096 (6)	0.0521 (7)	0.1164 (5)	0.085 (8)
C(47)	−0.4954 (5)	0.0446 (5)	0.1336 (4)	0.061 (5)
C(48)	−0.4896 (8)	0.1528 (6)	0.1329 (6)	0.093 (9)
C(49)	0.024 (1)	0.240 (1)	−0.1666 (6)	0.097 (9)
C(50)	−0.037 (1)	0.3021 (7)	−0.1478 (6)	0.095 (8)
C(51)	0.1346 (9)	0.2918 (9)	−0.1821 (7)	0.105 (8)
C(52)	0.012 (1)	0.155 (1)	−0.1759 (6)	0.105 (7)
C(53)	−0.0996 (6)	0.1034 (7)	−0.1585 (6)	0.078 (6)
C(54)	0.0672 (6)	0.0899 (7)	−0.2064 (5)	0.085 (7)
C(55)	0.3340 (6)	0.4586 (6)	0.6022 (6)	0.075 (6)
C(56)	0.2838 (7)	0.5135 (6)	0.5538 (6)	0.087 (7)
C(57)	0.3628 (7)	0.3780 (7)	0.5513 (6)	0.089 (7)
C(58)	0.3577 (5)	0.4821 (5)	0.6950 (5)	0.064 (5)
C(59)	0.4207 (6)	0.4250 (5)	0.7365 (6)	0.072 (5)

C(60)	0.3360 (5)	0.5545 (5)	0.7555 (5)	0.072 (6)
C(61)	0.3815 (9)	0.5773 (7)	0.8511 (8)	0.105 (9)
C(62)	0.2708 (6)	0.6097 (6)	0.7352 (6)	0.079 (7)

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

Fe(1)—C(1)	2.060 (7)	Fe(3)—C(41)	2.088 (6)
Fe(1)—C(2)	2.064 (7)	Fe(3)—C(43)	2.112 (6)
Fe(1)—C(4)	2.077 (7)	Fe(3)—C(45)	2.088 (6)
Fe(1)—C(6)	2.076 (7)	Fe(3)—C(47)	2.089 (6)
Fe(1)—C(8)	2.079 (7)	N(1)—C(50)	1.13 (1)
Fe(1)—C(10)	2.087 (6)	N(2)—C(51)	1.10 (1)
Fe(1)—C(12)	2.092 (6)	N(3)—C(53)	1.09 (1)
Fe(1)—C(14)	2.091 (6)	N(4)—C(54)	1.12 (1)
Fe(1)—C(16)	2.089 (5)	N(5)—C(56)	1.15 (1)
Fe(1)—C(18)	2.091 (5)	N(6)—C(57)	1.13 (1)
Fe(2)—C(20)	2.039 (5)	N(7)—C(59)	1.133 (9)
Fe(2)—C(21)	2.035 (6)	N(8)—C(61)	1.15 (1)
Fe(2)—C(22)	2.031 (6)	N(9)—C(62)	1.145 (9)
Fe(2)—C(23)	2.028 (6)	C(49)—C(50)	1.39 (2)
Fe(2)—C(24)	2.024 (6)	C(49)—C(51)	1.59 (2)
Fe(2)—C(25)	2.024 (6)	C(49)—C(52)	1.13 (2)
Fe(2)—C(26)	2.023 (6)	C(52)—C(53)	1.61 (2)
Fe(2)—C(27)	2.024 (6)	C(52)—C(54)	1.40 (1)
Fe(2)—C(28)	2.029 (6)	C(55)—C(56)	1.40 (1)
Fe(2)—C(29)	2.035 (5)	C(55)—C(57)	1.43 (1)
Fe(3)—C(31)	2.082 (5)	C(55)—C(58)	1.38 (1)
Fe(3)—C(32)	2.102 (5)	C(58)—C(59)	1.48 (1)
Fe(3)—C(34)	2.100 (5)	C(58)—C(60)	1.37 (1)
Fe(3)—C(36)	2.097 (5)	C(60)—C(61)	1.46 (1)
Fe(3)—C(38)	2.085 (5)	C(60)—C(62)	1.37 (1)
Fe(3)—C(40)	2.065 (6)		
C(50)—C(49)—C(51)	117.0 (13)	C(57)—C(55)—C(58)	120.6 (7)
C(50)—C(49)—C(52)	133.0 (20)	N(5)—C(56)—C(55)	175.9 (11)
C(51)—C(49)—C(52)	110.0 (14)	N(6)—C(57)—C(55)	177.3 (10)
N(1)—C(50)—C(49)	175.8 (15)	C(55)—C(58)—C(59)	115.9 (7)
N(2)—C(51)—C(49)	174.5 (16)	C(55)—C(58)—C(60)	129.6 (7)
C(49)—C(52)—C(53)	109.6 (14)	C(59)—C(58)—C(60)	114.4 (7)
C(49)—C(52)—C(54)	133.5 (18)	N(7)—C(59)—C(58)	177.3 (8)
C(53)—C(52)—C(54)	116.5 (11)	C(58)—C(60)—C(61)	120.7 (7)
N(3)—C(53)—C(52)	168.6 (10)	C(58)—C(60)—C(62)	125.5 (8)
N(4)—C(54)—C(52)	172.3 (12)	C(61)—C(60)—C(62)	113.8 (7)
C(56)—C(55)—C(57)	117.4 (8)	N(8)—C(61)—C(60)	176.8 (11)
C(56)—C(55)—C(58)	122.0 (8)	N(9)—C(62)—C(60)	177.6 (11)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1987). Cell refinement: *CAD*-4 *Software*. Data reduction: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1975). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *CRYSTALS*. Molecular graphics: *CAMERON* (Pearce & Watkin, 1993). Software used to prepare material for publication: *CRYSTALS*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1195). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Monoclinic Form of [Cu(C$_{10}$H$_{24}$N$_4$)]CuCl$_4$

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Abstract

The monoclinic form of 1,4,8,11-tetraazacyclotetradecanecopper(II) tetrachlorocuprate, [Cu(14-ane)]CuCl$_4$, has a chain structure consisting of Cu(14-ane)$^{2+}$ cations linked by CuCl$_4^{2-}$ anions. The Cu$^{\rm II}$ ion has square-planar coordination in the cation [average Cu—N distance = 2.020 (7) Å] while the anion has flattened tetrahedral coordination geometry [average Cu—Cl distance = 2.246 (13) Å with average *trans* Cl—Cu—Cl angles = 130 (1)°]. The cations and anions are linked by semi-