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Synthesis and Structure of 4-Ferrocenyl-2′-methyl-4′-nitroazobenzene*

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Abstract. [Fe($C_{23}H_{19}N_3O_2$)], $M_r = 425{\cdot}3$, monoclinic, $P2_1/c$, $a = 15{\cdot}020\,(9)$, $b = 7{\cdot}555\,(2)$, $c = 18{\cdot}134\,(15)$ Å, $\beta = 112{\cdot}22\,(6)^\circ$, $V = 1905$ Å^3, $Z = 4$, $D_x = 1{\cdot}483$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 8{\cdot}28$ cm^{-1}, $F(000) = 880$, $T = 293$ K, $R = 0{\cdot}052$ for 2252 observed reflections. The azobenzene residue has the *trans* configuration, C—N—N—C torsion angle 177·2 (7)°, N=N bond length 1·238 (6) Å. The cyclopentadienyl rings of the ferrocenyl residue are eclipsed with Fe—C distances 2·005 (7) to 2·053 (6) Å, mean 2·034 (4) Å. The rings are inclined at 2·7 (6)° to one another. There does not appear to be any significant electron delocalization along the length of the molecule.

Introduction. There has recently been considerable interest in materials which exhibit non-linear optical (NLO) properties (*e.g.* Chemla & Zyss, 1987) in connection with optical data transmission and processing technologies. The necessary (but not sufficient) condition for a material to exhibit the desired NLO properties is that it should consist of polarizable dipolar molecules which crystallize in a non-centrosymmetric space group. The title compound contains a ferrocenyl residue which acts as electron donor and a nitro group which acts as electron acceptor linked by a polarizable chain of atoms, thus meeting the first criterion for NLO behaviour. Unfortunately, however, it crystallizes in the centrosymmetric space group $P2_1/c$. The related compound *cis*-[(η^5-C_5H_5)Fe(η^5-C_5H_4)—CH=CHC_6H_4-4-NO_2], however, crystallizes in the non-centrosymmetric space group *Cc* and shows relatively high NLO properties of 62 times that of urea taken as standard (Green, Marder, Thompson, Bandy, Bloor, Kolinsky & Jones, 1987).

Experimental. The synthesis of the title compound followed the method of Little & Clark (1960). A mixture of 2-nitroso-5-nitrotoluene (3·6 g, 22 mmol) and 4-aminophenylferrocene (5·0 g, 18 mmol) in glacial acetic acid (400 cm^3) was stirred at room temperature for 5 d. Water (1000 cm^3) and dichloromethane (100 cm^3) were added and the organic layer separated and evaporated to dryness to yield the crude product. This was purified by column chromatography using gradient elution from 50% dichloromethane/*n*-hexane to 100% dichloromethane. The major purple band collected in 70% dichloromethane/*n*-hexane afforded deep purple crystals. A crystal of size 0·4 × 0·4 × 0·1 mm selected from this material was mounted on an Enraf—Nonius CAD-4 diffractometer and cell dimensions were determined from the setting angles of 24 reflections in the range $10 < \theta < 15^\circ$. Intensity data were

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and equivalent isotropic temperature factors* ($Å^2 \times 10^3$)

$U_{eq} = \frac{1}{3}$(trace of orthogonalized U_{ij} tensor).

	x	y	z	U_{eq}
Fe	−3135 (1)	1240 (1)	802 (1)	37
N(1)	4346 (4)	3476 (8)	7658 (3)	59
N(2)	859 (3)	2285 (6)	5072 (3)	45
N(3)	906 (3)	1728 (6)	4446 (3)	44
O(1)	4323 (3)	4445 (8)	8188 (3)	77
O(2)	5069 (3)	2712 (10)	7682 (3)	90
C(1)	3485 (4)	2216 (9)	6330 (3)	51
C(2)	2633 (4)	1945 (8)	5687 (3)	48
C(3)	1773 (4)	2603 (7)	5697 (3)	39
C(4)	1744 (4)	3526 (7)	6352 (3)	42
C(5)	2601 (4)	3851 (8)	6985 (3)	46
C(6)	3450 (4)	3174 (8)	6962 (3)	45
C(7)	789 (4)	4120 (9)	6391 (4)	59
C(8)	−7 (4)	1328 (8)	3832 (3)	41
C(9)	22 (4)	802 (8)	3116 (3)	47
C(10)	−813 (4)	364 (9)	2478 (3)	46
C(11)	−1697 (4)	510 (7)	2555 (3)	38
C(12)	−1724 (4)	1067 (8)	3273 (3)	45
C(13)	−888 (4)	1461 (8)	3916 (3)	47
C(14)	−3668 (4)	−1281 (8)	744 (3)	51
C(15)	−2681 (4)	−1205 (8)	1263 (3)	46
C(16)	−2590 (4)	24 (7)	1885 (3)	41
C(17)	−3527 (4)	733 (8)	1745 (3)	46
C(18)	−4184 (4)	−96 (8)	1033 (4)	50
C(19)	−2286 (8)	1983 (14)	212 (7)	95
C(20)	−3192 (8)	1982 (12)	−296 (5)	84
C(21)	−3735 (5)	3146 (11)	−24 (5)	77
C(22)	−3077 (8)	3901 (10)	701 (5)	85
C(23)	−2183 (6)	3113 (13)	816 (5)	87

measured with graphite-monochromated Mo $K\alpha$ radiation. 4546 reflections were scanned by $\omega/2\theta$ scans up to $\theta = 27°$ of which 4157 were unique (R_{int} 0·075). 2252 [$I > 2{\cdot}5\sigma(I)$] reflections were considered observed and used in the analysis, index range h −19 to 19, k 0 to 9, l 0 to 23. Three standard reflections measured every 2 h showed no significant variation in intensity. The structure was determined by Patterson and Fourier methods and refined by least squares using anisotropic thermal parameters for the heavier atoms. H atoms were included in calculated positions, riding on their respective bonded atoms. Weights, $w = 1/[\sigma^2(F) + 0{\cdot}0005F^2]$, which resulted in a satisfactory weighting analysis, were used in the least-squares refinement. The refinement was carried out on F values and converged to $R = 0{\cdot}052$ and $wR = 0{\cdot}067$ with maximum shift/e.s.d. ratio < 0·015. The residual electron density in a final difference map was within ±0·5 e Å^{-3}. An empirical absorption correction using *DIFABS* (Walker & Stuart, 1983) was applied (minimum and maximum transmission factors were 0·526 and 0·719). No correction was made for secondary extinction. The final atomic coordinates are listed in Table 1.*

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Fe—C(14)	2·053 (6)	C(4)—C(5)	1·385 (7)
Fe—C(15)	2·036 (6)	C(4)—C(7)	1·530 (8)
Fe—C(16)	2·039 (5)	C(5)—C(6)	1·389 (8)
Fe—C(17)	2·044 (6)	C(8)—C(9)	1·374 (7)
Fe—C(18)	2·044 (6)	C(8)—C(13)	1·391 (7)
Fe—C(19)	2·029 (8)	C(9)—C(10)	1·388 (7)
Fe—C(20)	2·038 (7)	C(10)—C(11)	1·391 (7)
Fe—C(21)	2·027 (6)	C(11)—C(12)	1·384 (7)
Fe—C(22)	2·024 (7)	C(11)—C(16)	1·476 (7)
Fe—C(23)	2·005 (7)	C(12)—C(13)	1·384 (7)
N(1)—O(1)	1·220 (7)	C(14)—C(15)	1·425 (8)
N(1)—O(2)	1·216 (7)	C(14)—C(18)	1·408 (9)
N(1)—C(6)	1·473 (7)	C(15)—C(16)	1·428 (8)
N(2)—N(3)	1·238 (6)	C(16)—C(17)	1·436 (7)
N(2)—C(3)	1·432 (6)	C(17)—C(18)	1·438 (8)
N(3)—C(8)	1·434 (6)	C(19)—C(20)	1·324 (12)
C(1)—C(2)	1·382 (8)	C(19)—C(23)	1·350 (13)
C(1)—C(6)	1·374 (8)	C(20)—C(21)	1·410 (12)
C(2)—C(3)	1·390 (7)	C(21)—C(22)	1·431 (11)
C(3)—C(4)	1·392 (7)	C(22)—C(23)	1·411 (12)

O(1)—N(1)—O(2)	123·2 (5)	C(8)—C(9)—C(10)	121·1 (5)
O(1)—N(1)—C(6)	118·5 (5)	C(9)—C(10)—C(11)	119·5 (5)
O(2)—N(1)—C(6)	118·2 (6)	C(10)—C(11)—C(12)	119·2 (5)
N(3)—N(2)—C(3)	114·3 (4)	C(10)—C(11)—C(16)	120·1 (5)
N(2)—N(3)—C(8)	114·5 (4)	C(12)—C(11)—C(16)	120·7 (5)
C(2)—C(1)—C(6)	117·9 (5)	C(11)—C(12)—C(13)	121·2 (5)
C(1)—C(2)—C(3)	120·2 (5)	C(8)—C(13)—C(12)	119·3 (5)
N(2)—C(3)—C(2)	123·2 (5)	C(15)—C(14)—C(18)	108·1 (5)
N(2)—C(3)—C(4)	115·3 (5)	C(14)—C(15)—C(16)	108·2 (5)
C(2)—C(3)—C(4)	121·3 (5)	C(11)—C(16)—C(15)	126·8 (5)
C(3)—C(4)—C(5)	118·5 (5)	C(11)—C(16)—C(17)	125·3 (5)
C(3)—C(4)—C(7)	121·3 (5)	C(15)—C(16)—C(17)	107·9 (5)
C(5)—C(4)—C(7)	120·2 (5)	C(16)—C(17)—C(18)	106·9 (5)
C(4)—C(5)—C(6)	119·1 (5)	C(14)—C(18)—C(17)	108·9 (5)
N(1)—C(6)—C(1)	119·0 (5)	C(20)—C(19)—C(23)	109·7 (9)
N(1)—C(6)—C(5)	118·0 (5)	C(19)—C(20)—C(21)	109·6 (8)
C(1)—C(6)—C(5)	122·9 (5)	C(20)—C(21)—C(22)	106·2 (7)
N(3)—C(8)—C(9)	115·6 (5)	C(21)—C(22)—C(23)	104·8 (7)
N(3)—C(8)—C(13)	124·7 (5)	C(19)—C(23)—C(22)	109·7 (7)
C(9)—C(8)—C(13)	119·7 (5)		

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computations were carried out largely on the University of Birmingham's IBM 3090 computer using *SHELXS*86 (Sheldrick, 1986) and *SHELX*76 (Sheldrick, 1976). Diagrams were drawn with *PLUTO* (Motherwell & Clegg, 1978), as implemented at the Manchester Computing Centre.

Discussion. Bond lengths and bond angles are listed in Table 2. The atomic numbering scheme is shown in Fig. 1. The molecule can be described in terms of six planar groupings, the nitro group, the C(1)—C(6) phenyl ring, the C(3)—N(2)—N(3)—C(8) azo group, the C(8)—C(13) phenyl ring and the two cyclopentadienyl rings of the ferrocene residue. Within each grouping, deviations from planarity are within the limits of experimental error [maximum 0·018 Å in the C(1)—C(6) phenyl ring and in the azo group].

The nitro group is twisted by 8·2 (6)° from the plane of the phenyl ring to which it is bonded. The two N—O bond lengths are identical at 1·22 (1) Å; the N—C(phenyl) bond length of 1·473 (7) Å indicates that this is essentially a pure single bond. The azo group is *trans*, torsion angle C(3)—N(2)—N(3)—C(8) 177·2 (7)° and the C(1)-C(6) and C(8)-C(13) phenyl rings are inclined to the azo plane at

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and spectroscopic and analytical data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54109 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angles of 14·7 (7) and 3·4 (7)°, respectively. The C—N and N—N bond lengths (Table 2) agree well with mean values of 1·428 and 1·244 Å, respectively, found in the most recent X-ray study of *trans*-azobenzene (Bouwstra, Schouten & Kroon, 1983) and also with the values of 1·443 and 1·251 Å in *cis*-azobenzene (Mostad & Romming, 1971).

The C(14)–C(18) cyclopentadienyl ring of the ferrocene residue is rotated by 27·9 (6)° relative to the plane of the C(8)–C(13) phenyl ring about the C(16)—C(11) linking bond. The length of this bond, 1·476 (7) Å, corresponds approximately to that of a single bond between sp^2-hybridized carbon atoms, so that any electron delocalization across this bond must be relatively small. Nevertheless the C—C bonds of the C(14)–C(18) cyclopentadienyl ring are slightly longer [mean length 1·427 (5) Å] than those of the other cyclopentadienyl ring, which is not linked directly to the azobenzene system, where the corresponding mean is 1·385 (2) Å. This mean, however, includes the two rather anomalously short bonds C(19)—C(20) and C(19)—C(23) of 1·324 and 1·350 Å. If these are excluded from the averaging, the mean length becomes 1·417 (7) Å, still smaller than the mean length for the bonded cyclopentadienyl ring, but not significantly so. Such a relative lengthening of bonds in the cyclopentadienyl ring bonded to another residue may be indicative of electron delocalization from the ferrocenyl residue to the bonded system (Roberts, Silver, Yamin, Drew & Eberhardt, 1988).

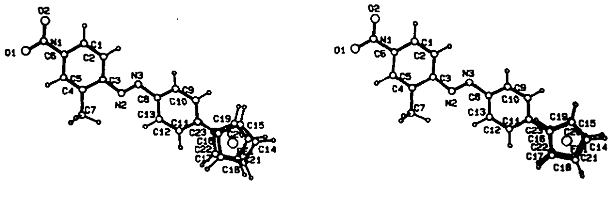

Fig. 1. Stereoscopic view of the molecule showing the atomic numbering.

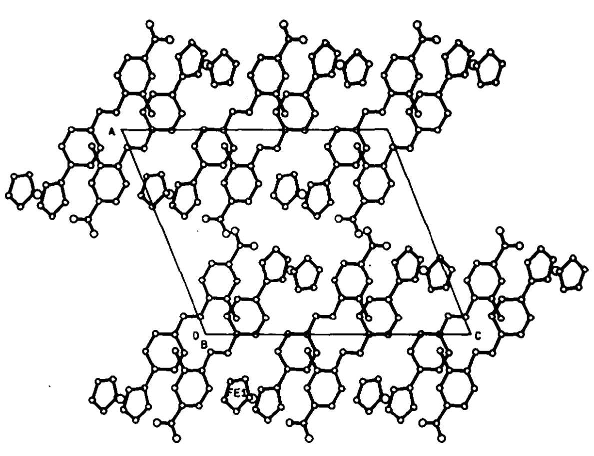

Fig. 2. View of the crystal structure along the *y* axis.

The two cyclopentadienyl rings are virtually eclipsed (see Fig. 1), angle of rotation from eclipsed 0·6°. The rings are nearly parallel, interplanar angle 2·7 (6)°, with an average separation of 3·29 (2) Å. The Fe—C distances are in the range 2·005 to 2·053 Å, mean 2·034 (4) Å. The distances between the Fe atom and the ring centroids are 1·643 and 1·647 Å and the angle subtended at Fe by the ring centroids is 177·7°, so that the Fe atom lies very close to the centroid of the ferrocenyl residue.

These parameters are similar to those found in, *inter alia*, ferrocene itself (Seiler & Dunitz, 1979, 1982), 4-biphenylferrocene (Allen, Trotter & Williston, 1970), ferrocenecarboxylic acid (Cotton & Reid, 1985; Iwai, Katada, Motoyama & Sano, 1987), 1,1′-ferrocenedicarboxylic acid (Takusagawa & Koetzle, 1979) and *o*-nitrophenyl-, *m*-nitrophenyl- and *p*-nitrophenylferrocenes (Roberts *et al.*, 1988). In all of these the two cyclopentadienyl rings are eclipsed, or nearly so, with a maximum rotation from eclipsed of *ca* 15° in one of the two independent molecules in the crystal structure of 4-biphenylferrocene. The relative rotation of the rings in the other molecule is, however, only *ca* 5°. The eclipsed orientation of the rings in ferrocene has been shown to be the preferred orientation by approximately 4 kJ $\mathrm{mol^{-1}}$ (Haaland & Nilsson, 1968). However, in [η^5-mesityl(hydroxymethyl)tetramethylcyclopentadienyl]-(η^5-pentamethylcyclopentadienyl)iron (Shubina, Epstein, Yanovskii, Timofeeva, Struchkov, Kreindlin, Fadeeva & Rybinskaya, 1988) and decamethylferrocene (Freyberg, Robbins, Raymond & Smart, 1979), where all ten H atoms of the cyclopentadienyl rings are substituted by bulkier groups, the orientation is fully staggered. Presumably, here the fully staggered orientation is energetically the most favourable due to the steric interactions of the substituents.

The crystal packing (Fig. 2) involves a centrosymmetric head-to-tail pairing of molecules, so that individual molecular dipoles are opposed and the bulk solid shows no NLO properties.

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Structure of Diperchlorato[(2*SR*,5*RS*,8*RS*,11*SR*)-2,5,8,11-tetraazadodecane]-nickel(II)

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Abstract. $[Ni(ClO_4)_2(C_8H_{22}N_4)]$, $M_r = 431{\cdot}90$, orthorhombic, $C222_1$, $a = 7{\cdot}661\,(3)$, $b = 15{\cdot}344\,(3)$, $c = 14{\cdot}323\,(3)$ Å, $V = 1683{\cdot}7\,(8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}704$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7093 Å, $\mu = 1{\cdot}52$ mm^{-1}, $F(000) = 895{\cdot}82$, $T = 295\,(3)$ K, $R = 0{\cdot}054$, $wR = 0{\cdot}074$ for 746 observed reflections. The central Ni^{II} atom and the two Cl atoms of the perchlorate groups are located on twofold axes. The coordination about Ni^{II} is a distorted square bipyramid with the tetraamine ligand equatorial and two O atoms of the perchlorate ions axial. The important bond distance is Ni—O(1) = 2·840 (11) Å. The bond angle of N(2)—Ni—N(2^i) is 93·4 (4)° and the *trans* N—Ni—N angles are 173·3 (5)°. The hydrogen bonds among the tetraamine and perchlorate groups stabilize the crystal structure.

Introduction. The crystal structures of transition metal complexes of $H_2N(CH_2)_lNH(CH_2)_mNH(CH_2)_nNH_2$ (*l,m,n*-tet) have been extensively studied (Marongiu, Lingafelter & Paoletti, 1969; Fawcett, Rudich, Toby, Lalancette, Potenza & Schugar, 1980; Lee, Lee, Hong, Hsieh, Wu & Chung, 1986; Boggs & Donohue, 1975). However, the crystal structures of transition metal complexes of $CH_3NH(CH_2)_lNH(CH_2)_mNH(CH_2)_nNHCH_3$ (Me$_2$-*l,m,n*-tet) have received little attention. An earlier report from this laboratory described the structure of the copper(II) complex of Me$_2$-2,2,2-tet (Wu, Wang, Liou & Chung, 1989). In this study, the structure of the nickel(II) complex has been determined.

Experimental. 2,5,8,11-Tetraazadodecane tetrahydrochloride was synthesized according to the method of Clay, Corr, Micheloni & Paoletti (1985). It (2·0 g) was dissolved in 10 cm^3 of water and the solution was passed through an anion exchange column (Amberlite IR400, OH$^-$form) directly into a hot aqueous solution of $Ni(ClO_4)_2.6H_2O$ (2·67 g). The solution was refluxed for two hours and was slowly evaporated to give reddish-orange crystals. The single crystals used in the X-ray analysis were obtained by recrystallization from methanol at room temperature.

A crystal of size 0·50 × 0·52 × 0·30 mm, cut from orange parallelepiped shape, was used for the X-ray structure determination; Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$. Unit-cell parameters from 25 reflections with $20 < 2\theta < 27°$, data collected by ω–2θ scans with scan width of $2(0{\cdot}60 + 0{\cdot}25\tan\theta)°$ and scan speed of 2·0 to 8·0° min^{-1}. Empirical absorption correction based on azimuthal rotation from three reflections (402, $5\bar{1}3$, 404) (North, Phillips & Mathews, 1968); the minimum, maximum and average correction factors are 0·9128, 0·9986 and 0·9590, respectively. Max.

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