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Crystal and molecular structure of *N*-(ferrocenylcarbonyl)-*N*′-naphthyl thiourea

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Abstract

The crystal structure of the title compound is monoclinic, space group $C2/c$ with unit cell parameters of $a = 38.703(3)$, $b = 12.989(2)$, $c = 15.271(4)$ Å, $\beta = 95.48(2)°$, $V = 7643(4)$ Å^3, $M_r = 414.31$, $Z = 16$, $D_x = 1.44$ g/cm^3, $\mu = 9.06$ cm^{-1}, $F(000) = 3424$. The final refinement converges with $R = 0.047$ and $R_w = 0.050$ for 2410 observed independent reflections. In this crystal, there are two independent molecules in an asymmetric unit each in a different conformation, both having intramolecular hydrogen bonding between the amino and carbonyl groups of –NH–C(S)–NH–C(O)–. This group forms a planar six-membered ring parallel to the Cp ring of the ferrocene moiety, but the dihedral angles between the planar six-membered ring and the naphthyl ring differ. One is 45.3°, designated as *endo* form, and the other 81.6°, the *exo* form.

Key words: Iron; Naphthyl; Thiourea

1. Introduction

Several thiourea derivatives have been found to have strong anti-thyroid activity. This has been interpreted as due to the formation of disulfide by the reaction of I_2 and –SH group (formed by enolization), thus interrupting the synthesis of thyroxine [1]. At the same time, the anti-bacterial activity of penicillins and cephalosporins is enhanced by introduction of the ferrocenyl moiety [2,3]. In a search for potentially biological active materials, a series of thiourea derivatives containing ferrocenyl mioety was prepared by treating with various amines the products of the reaction of ferrocenylcarbonyl chloride and potassium thiocyanate [4]. The title compound is one such compound and its crystal structure determined by X-ray diffraction, is reported here. X-ray analysis reveals that both *endo* and *exo* conformation isomers of the title compound co-exist in a monoclinic cell.

2. Discussion

2.1. Synthesis and reaction

The following equation shows the preparation of the title compound:

$$\mathrm{Fc{-}C(O)Cl} \xrightarrow[\text{acetone}]{\text{KSCN}} \mathrm{FcC(O){-}NCS} \xrightarrow{\alpha\text{-naphthylamine}}$$

$$\mathrm{FcC(O){-}\overset{\alpha}{NH}{-}C(S){-}\overset{\beta}{NH}{-}Np}$$

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where, Fc = ferrocenyl group, Np = α-naphthyl.

The product is red, yield 80%, melting point 146–147°C and was recrystallized from benzene/ligroin solution.

2.2. Infra-red investigation

A weak absorption at 1663.3 cm^{-1} is attributed to the stretching vibrations of carbonyl group attached to ferrocenyl. The ν_{CO} is apparently lower than that of ordinary carbonyl absorption [5], because of its proximity to the Cp ring of ferrocenyl and the formation of an

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intramolecular hydrogen bond with NH attached to naphthyl. A strong band at 1515.6 cm^{-1} is attributed to δ_{N-H}. A weak absorption band is observed at 1261.3 cm^{-1}, which is considered to arise from $\nu_{-N-C=O}$ vibration, and this is significantly weaker than either ν_{CO} or δ_{N-H}. It is certainly due to a mixed vibration involving -N-C=O and N-H modes [5] which also supports the hypothesis of the presence of the intramolecular hydrogen bond. The medium intensity band at 3394.1 cm^{-1} is due to stretching vibration of the NH group attached to carbonyl. The broad and weak band in the region 3160-2900 cm^{-1} is assigned to the stretching vibration of NH attached to naphthyl group. The medium band at 1343.0 cm^{-1} is assigned to $\nu_{C=S}$ and the strong band at 1146.5 cm^{-1} is assigned to $\nu_{-N-C=S}$ vibration.

TABLE 1. Fractional coordinates and equivalent isotropic thermal parameters of non-hydrogen atoms

	x	y	z	B_{eq} (Å^2)
Fe(1)	0.90472(3)	0.0188(1)	0.68068(9)	4.02(3)
S(1)	1.00483(7)	0.1444(2)	0.4249(2)	5.20(6)
O(1)	0.9218(2)	0.2757(5)	0.5819(4)	5.3(2)
N(1)	0.9631(2)	0.1602(6)	0.5490(5)	4.5(2)
N(2)	0.9526(2)	0.2801(6)	0.4402(5)	4.7(2)
C(11)	0.8893(2)	−0.1294(8)	0.6571(8)	6.2(3)
C(12)	0.9037(3)	−0.0884(8)	0.5826(7)	6.1(3)
C(13)	0.8838(2)	0.001(1)	0.5533(6)	6.2(3)
C(14)	0.8570(2)	0.0142(9)	0.6118(7)	5.5(3)
C(15)	0.8604(2)	−0.0673(8)	0.6755(7)	5.6(3)
C(21)	0.9357(2)	0.1415(7)	0.6839(6)	3.9(2)
C(22)	0.9550(2)	0.0538(7)	0.7167(6)	4.6(2)
C(23)	0.9393(2)	0.0206(8)	0.7924(6)	5.2(2)
C(24)	0.9090(3)	0.0850(8)	0.8035(6)	5.2(3)
C(25)	0.9069(2)	0.1623(7)	0.7361(6)	4.7(2)
C(26)	0.9393(2)	0.1984(7)	0.6027(6)	4.3(2)
C(27)	0.9718(2)	0.2001(7)	0.4695(6)	4.2(2)
C(30)	0.9556(2)	0.3374(7)	0.3598(6)	4.0(2)
C(31)	0.9563(2)	0.2898(8)	0.2815(6)	4.7(2)
C(32)	0.9565(3)	0.3510(8)	0.2054(7)	5.6(3)
C(33)	0.9562(2)	0.4571(8)	0.2091(6)	5.5(3)
C(34)	0.9561(3)	0.6150(8)	0.2971(7)	6.0(3)
C(35)	0.9567(3)	0.6639(9)	0.3766(8)	7.9(4)
C(36)	0.9558(4)	0.601(1)	0.4551(8)	8.9(4)
C(37)	0.9553(3)	0.4962(8)	0.4520(7)	6.6(3)
C(38)	0.9554(2)	0.4461(7)	0.3693(6)	4.3(2)
C(39)	0.9559(2)	0.5067(7)	0.2919(6)	4.4(2)
Fe(2)	0.66889(3)	0.9676(1)	0.38768(9)	4.21(3)
S(2)	0.63609(7)	1.2838(3)	0.6258(2)	6.38(8)
O(2)	0.7187(2)	1.2094(5)	0.4449(5)	6.3(2)
N(3)	0.6633(2)	1.2171(6)	0.4867(5)	4.8(2)
N(4)	0.7019(2)	1.3048(6)	0.5885(5)	4.4(2)
C(41)	0.6748(3)	0.8156(9)	0.4132(8)	7.8(3)
C(42)	0.6433(3)	0.8517(9)	0.4478(8)	8.1(3)
C(43)	0.6525(4)	0.9308(9)	0.5092(8)	8.3(4)
C(44)	0.6894(4)	0.9437(9)	0.5147(8)	8.8(4)
C(45)	0.7037(3)	0.8712(9)	0.4540(9)	8.5(4)
C(51)	0.6741(2)	0.1155(7)	0.3609(6)	4.3(2)
C(52)	0.6382(2)	1.0841(7)	0.3385(6)	4.6(2)
C(53)	0.6387(2)	1.0056(8)	0.2728(6)	5.3(2)
C(54)	0.6748(2)	0.9899(8)	0.2573(6)	5.4(3)
C(55)	0.6968(2)	1.0566(7)	0.3113(6)	4.8(2)
C(56)	0.6884(2)	1.1848(7)	0.4327(6)	4.4(2)
C(57)	0.6697(2)	1.2712(7)	0.5667(6)	4.3(2)
C(60)	0.7103(2)	1.3615(7)	0.6682(6)	4.3(2)
C(61)	0.7243(2)	1.3123(8)	0.7437(6)	4.9(2)
C(62)	0.7334(2)	1.3694(9)	0.8217(7)	5.7(3)
C(63)	0.7287(2)	1.4745(9)	0.8225(6)	5.8(3)
C(64)	0.7084(3)	1.6354(8)	0.7454(8)	6.8(3)
C(65)	0.6935(3)	1.6848(9)	0.6728(9)	8.1(4)
C(66)	0.6856(3)	1.6289(9)	0.5918(9)	7.6(3)
C(67)	0.6916(2)	1.5241(8)	0.5876(7)	6.1(3)
C(68)	0.7057(2)	1.4695(7)	0.6656(6)	4.3(2)
C(69)	0.7148(2)	1.5274(8)	0.7452(6)	4.6(2)

Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as: $(4/3)\times[a^2\times\beta(1, 1)+b^2\times\beta(2, 2)+c^2\times\beta(3, 3)+ab(\cos\ r)\times\beta(1, 2)+ac(\cos\ \beta)\times\beta(1, 3)+bc(\cos\ a)\times\beta(2, 3)]$.

TABLE 2. Selected bond distances (Å) and angles (°) between non-hydrogen atoms

Fe(1)–Cp(1) [a]	1.638(5)	Fe(2)–Cp(4) [a]	1.644(6)
Fe(1)–Cp(2) [a]	1.627(5)	Fe(2)–Cp(5) [a]	1.618(4)
S(1)–C(27)	1.670(4)	S(2)–C(57)	1.660(4)
O(1)–C(26)	1.235(4)	O(2)–C(56)	1.215(4)
N(1)–C(26)	1.384(5)	N(3)–C(56)	1.397(5)
N(1)–C(27)	1.389(5)	N(3)–C(57)	1.409(5)
N(2)–C(27)	1.331(5)	N(4)–C(57)	1.332(4)
N(2)–C(30)	1.449(5)	N(4)–C(60)	1.435(5)
C(21)–C(22)	1.429(5)	C(51)–C(52)	1.455(5)
C(21)–C(25)	1.455(6)	C(51)–C(55)	1.434(5)
C(21)–C(26)	1.461(5)	C(51)–C(56)	1.485(5)
Cp(1)–Fe(1)–Cp(2)	177.9(3)	Cp(4)–Fe(2)–Cp(5)	179.0(3)
C(26)–N(1)–C(27)	127.9(4)	C(56)–N(3)–C(57)	125.9(3)
C(27)–N(2)–C(30)	126.8(3)	C(57)–N(4)–C(60)	120.9(3)
C(22)–C(21)–C(25)	111.0(4)	C(52)–C(51)–C(55)	110.2(4)
C(22)–C(21)–C(26)	127.8(4)	C(52)–C(51)–C(56)	128.5(4)
C(25)–C(21)–C(26)	120.7(4)	C(55)–C(51)–C(56)	120.6(4)
O(1)–C(26)–N(1)	121.2(4)	O(2)–C(56)–N(3)	123.2(4)
O(1)–C(26)–C(21)	122.4(4)	O(2)–C(56)–C(51)	124.1(4)
N(1)–C(26)–C(21)	116.5(4)	N(3)–C(56)–C(51)	112.8(4)
S(1)–C(27)–N(1)	116.9(3)	S(2)–C(57)–N(3)	116.1(3)
S(1)–C(27)–N(2)	128.7(3)	S(2)–C(57)–N(4)	126.4(3)
N(1)–C(27)–N(2)	114.4(3)	N(3)–C(57)–N(4)	117.5(3)
N(2)–C(30)–C(31)	121.7(5)	N(4)–C(60)–C(61)	120.6(4)
N(2)–C(30)–C(38)	115.0(5)	N(4)–C(60)–C(68)	117.9(4)
C(31)–C(30)–C(38)	123.2(4)	C(61)–C(60)–C(68)	121.4(4)
Hydrogen bonding			
O(1) ··· N(2)	2.568(7)	O(2) ··· N(4)	2.654(7)
C(26)–O(1) ··· N(2)	87.1(5)	C(56)–O(2) ··· N(4)	86.5(5)
C(27)–N(2) ··· O(1)	89.3(5)	C(57)–N(4) ··· O(2)	86.5(5)
C(30)–N(2) ··· O(1)	143.7(5)	C(60)–N(4) ··· O(2)	152.5(5)

Numbers in parentheses are estimated standard deviations in the last significant digits.

[a] Cp(1), Cp(2), Cp(4), and Cp(5) are the centres of five-membered rings composed of C(11)–C(15), C(21)–C(25), C(41)–C(45), and C(51)–C(55), respectively.

2.3. ^1H NMR investigation

The protons on the substituted Cp give rise to AA'BB' type resonance. The NH between naphthyl and thiocarbonyl gives δ 12.8 ppm at lower field and the NH between the carbonyl and thiocarbonyl δ 8.88 ppm at high field, which might be due to the deshielding effect of the intramolecular hydrogen bond. The multiple peaks at 7.44–8.2 ppm are assigned as signals of the naphthyl group.

2.4. Results of X-ray diffraction analysis

The fractional coordinates and thermal parameters for non-hydrogen atoms are listed in Table 1 and selected bond distances and angles in Table 2.

X-ray diffraction analysis reveals that in each asymmetric unit, there are two independent molecules of the title compound with different conformations, which are shown in Figs. 1(a) and 1(b), respectively. In each molecule the atoms of the bridge between Fc and Np, –NH–C(S)–NH–C(O)–, form an intramolecular hydrogen bond to give a planar six-membered ring including the hydrogen atom. This planar ring is almost parallel to the cyclopentadienyl ring of Fc in both conformations, but it forms different dihedral angles with Np ring, one being 45° and the other 82°. The former conformation is called the *endo* form and the latter the *exo* form.

Table 3 lists the least-squares planes of relevant groups and the dihedral angles between them, and Table 4 shows selected torsion angles. This crystal consists of discrete molecules and the contact distances between the molecules are normal van der Waals distances. Fig. 2 shows the packing of 16 molecules of the title compound including both *endo* and *exo* forms in a

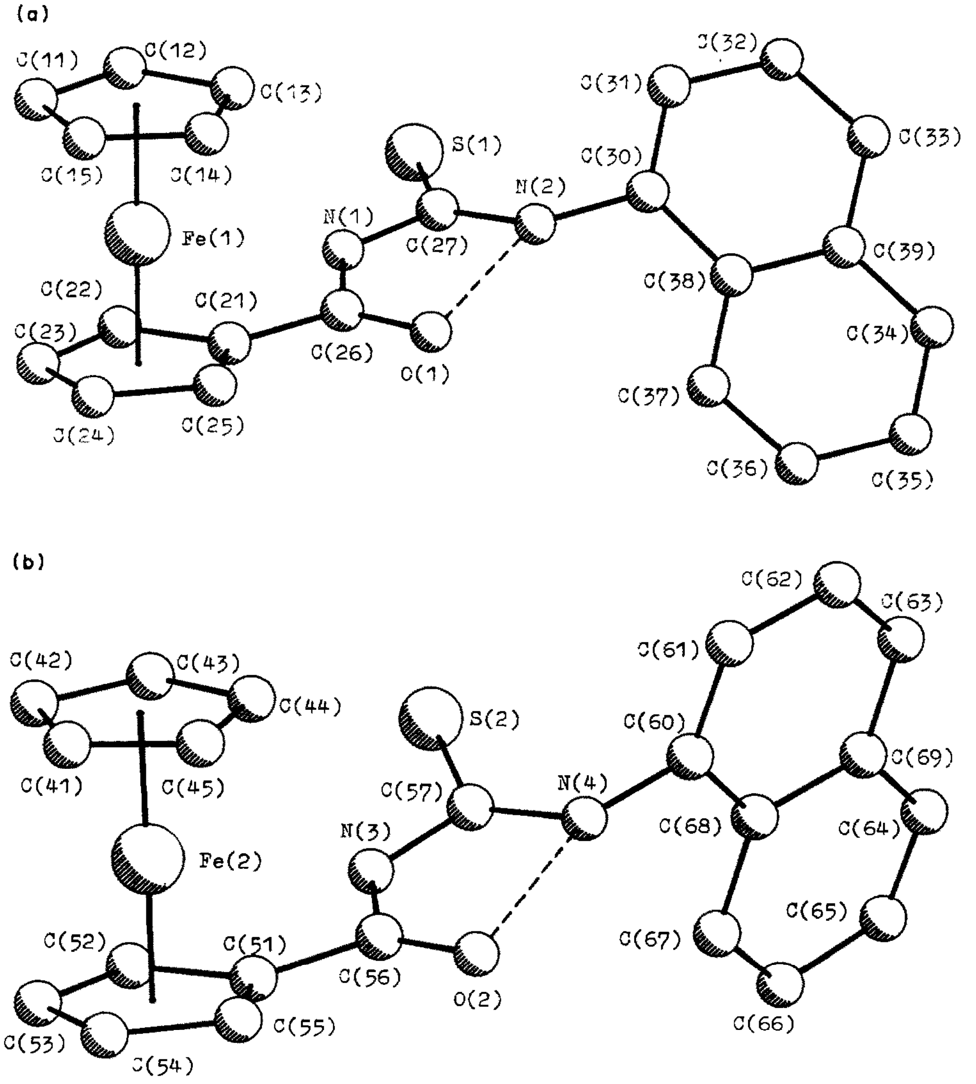

Fig. 1. The molecular structure of the title compound showing the atomic labelling scheme with (a) for *endo* form and (b) for *exo* form.

unit cell. It is interesting that conformation isomers with different Np group orientations co-exist within the same crystal. The fact that the ^{1}H NMR signal of the NH between thiocarbonyl and Np group is a sharp singlet suggests that the *N*-substituted Np group can rotate freely around the N–C bond in solution.

In Fig. 3, (a) and (b) are the projections along the normal of the unsubstituted cyclopentadienyl for the conformation isomers of the title compound, respectively. It can be seen that the ferrocenyl group in both conformations are almost eclipsed, which are similar to ferrocene in the triclinic phase (at 101 K) [6] but different from those in the monoclinic phase (stagger as disorder model, at 173 K) [7] and orthorhombic (exactly eclipsed, at 98 K) [8]. The bulky substituent on the cyclopentadienyl ring prevents the rotation of cyclopentadienyl about its normal as it does in the unsubstituted ferrocene so that the vibrational tensors obtained from X-ray data at room temperature for the carbons composing the cyclopentadienyl rings are close

TABLE 3. Least-squares planes

Equation of plane 1: $-0.557(4)X - 0.575(4)Y - 0.599(4)Z + 23.7(1) = 0$										
Atom	C(11)	C(12)	C(13)	C(14)	C(15)	Fe(1)				
Distance to plane (Å)	0.00(1)	0.00(1)	0.00(1)	0.00(1)	0.00(1)	−1.638(1)				
Equation of plane 2: $-0.535(4)X - 0.606(4)Y - 0.589(4)Z + 26.1(1) = 0$										
Atom	C(21)	C(22)	C(23)	C(24)	C(25)	C(26)	Fe(1)			
Distance to plane (Å)	−0.01(1)	0.01(1)	−0.02(1)	0.01(1)	−0.00(1)	0.13(1)	1.627(1)			
Equation of plane 3: $-0.661(3)X - 0.592(3)Y - 0.462(3)Z + 29.20(8) = 0$										
Atom	O(1)	N(1)	N(2)	C(26)	C(27)	S(1)	C(21)	C(30)		
Distance to plane (Å)	−0.02(1)	0.02(1)	0.02(1)	0.01(1)	−0.03(1)	−0.177(3)	0.05(1)	−0.01(1)		
Equation of plane 4: $-0.9944(3)X + 0.005(2)Y - 0.106(3)Z + 36.82(1) = 0$										
Atom	C(30)	C(31)	C(32)	C(33)	C(34)	C(35)	C(36)	C(37)	C(38)	C(39)
Distance to plane (Å)	0.02(1)	−0.02(1)	−0.01(1)	0.01(1)	0.01(1)	−0.02(1)	−0.00(1)	0.01(1)	0.01(1)	0.01(1)
Equation of plane 5: $-0.043(6)X + 0.683(5)Y - 0.729(4)Z - 1.6(2) = 0$										
Atom	C(41)	C(42)	C(43)	C(44)	C(45)	Fe(2)				
Distance to plane (Å)	0.00(1)	−0.01(1)	0.00(1)	−0.00(1)	−0.00(1)	1.643(1)				
Equation of plane 6: $-0.021(5)X + 0.695(4)Y - 0.719(4)Z - 5.6(1) = 0$										
Atom	C(51)	C(52)	C(53)	C(54)	C(55)	C(56)	Fe(2)			
Distance to plane (Å)	0.01(1)	−0.00(1)	0.00(1)	0.00(1)	−0.00(1)	−0.16(1)	−1.617(1)			
Equation of plane 7: $-0.126(4)X + 0.842(2)Y - 0.524(3)Z - 6.3(1) = 0$										
Atom	O(2)	N(3)	N(4)	C(56)	C(57)	S(2)	C(51)	C(60)		
Distance to plane (Å)	0.00(1)	0.03(1)	0.01(1)	−0.02(1)	−0.03(1)	−0.187(3)	−0.15(1)	−0.03(1)		
Equation of plane 8: $0.9514(7)X + 0.148(2)Y - 0.270(3)Z - 25.14(7) = 0$										
Atom	C(60)	C(61)	C(62)	C(63)	C(64)	C(65)	C(66)	C(67)	C(68)	C(69)
Distance to plane (Å)	−0.03(1)	−0.02(1)	−0.01(1)	0.02(1)	0.00(1)	−0.05(1)	−0.00(1)	0.04(1)	0.02(1)	0.03(1)
Planes	1–2	1–3	1–4	2–3	2–4	3–4				
Dihedral angle (°)	2.28	9.91	52.07	10.29	53.75	45.33				
Planes	5–6	5–7	5–8	6–7	6–8	7–8				
Dihedral angle (°)	1.53	15.65	75.09	15.28	73.91	81.58				

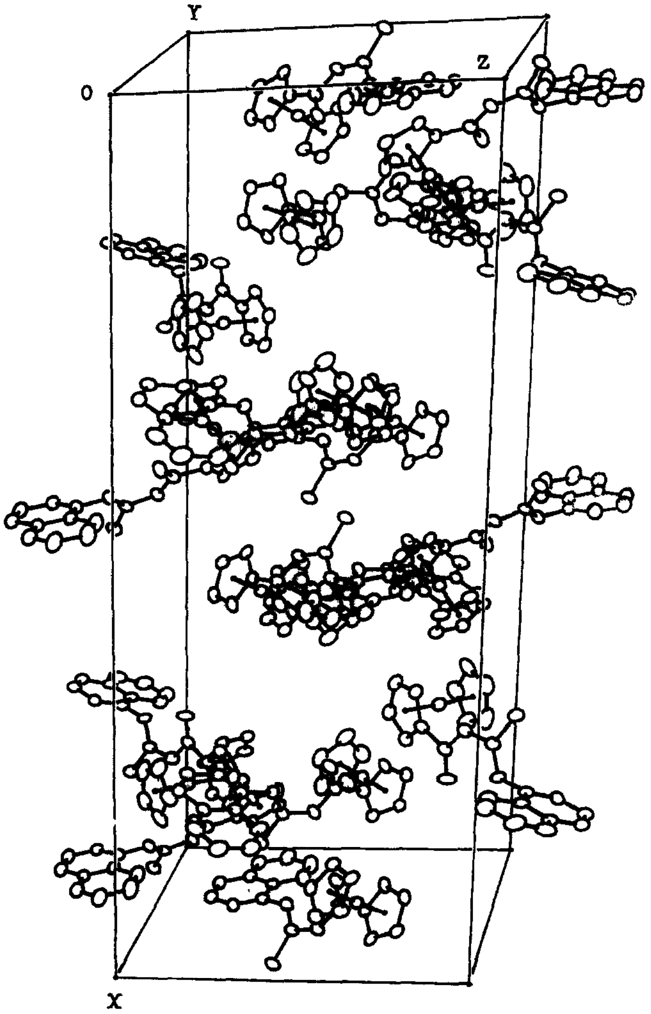

Fig. 2. Unit cell of the title compound containing both endo and exdo forms.

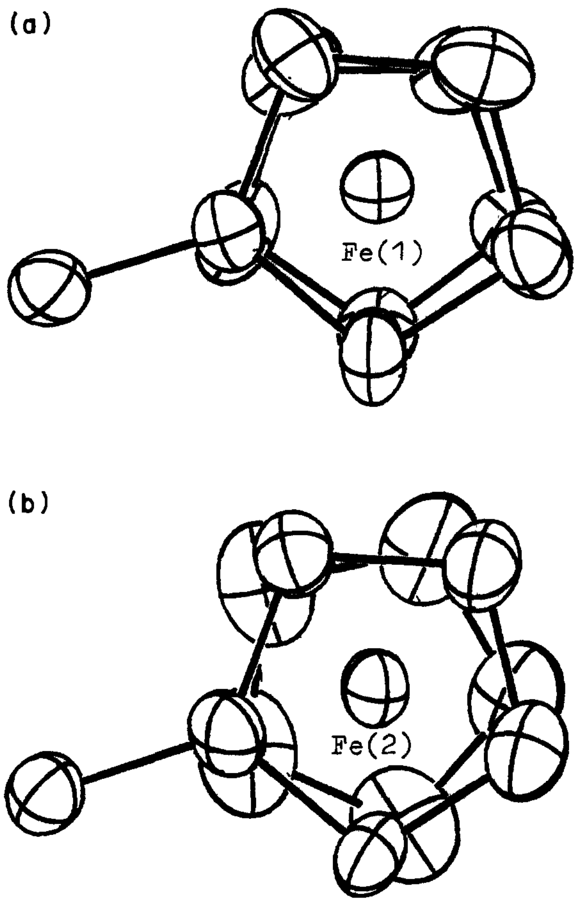

Fig. 3. ORTEP drawings of the ferrocenyl groups showing the thermal motion ellipsoids (35%) with (a) for *endo* form and (b) for *exo* form, where the substituent is omitted except for the first carbon atom.

to or even smaller than those obtained at low temperature for the corresponding carbons in the unsubstituted ferrocene [6–8].

3. Experimental section

The title compound was prepared according to the literature [4]. Anal. Found: C, 64.14; H, 4.39; N, 6.73. $C_{22}H_{18}FeN_2OS$ Calcd.: C, 63.78; H, 4.38; N, 6.76%.

TABLE 4. Selected torsion angles (°)

C(27)-N(1)-C(26)-O(1)	2(1)	C(57)-N(3)-C(56)-O(2)	-8(1)
C(27)-N(1)-C(26)-C(21)	-179.1(8)	C(57)-N(3)-C(56)-C(51)	170.7(8)
C(26)-N(1)-C(27)-S(1)	173.3(7)	C(56)-N(3)-C(57)-S(2)	-170.5(7)
C(26)-N(1)-C(27)-N(2)	-5(1)	C(56)-N(3)-C(57)-N(4)	8(1)
C(30)-N(2)-C(27)-S(1)	1(1)	C(60)-N(4)-C(57)-S(2)	-3(1)
C(30)-N(2)-C(27)-N(1)	-180.0(7)	C(60)-N(4)-C(57)-N(3)	179.2(8)
C(27)-N(2)-C(30)-C(31)	50(1)	C(57)-N(4)-C(60)-C(61)	96(1)
C(27)-N(2)-C(30)-C(38)	-133.3(9)	C(57)-N(4)-C(60)-C(68)	-87(1)
C(22)-C(21)-C(26)-O(1)	177.6(9)	C(52)-C(51)-C(56)-O(2)	-177.6(9)
C(22)-C(21)-C(26)-N(1)	3(1)	C(52)-C(51)-C(56)-N(3)	4(1)
C(25)-C(21)-C(26)-O(1)	11(1)	C(55)-C(51)-C(56)-O(2)	13(1)
C(25)-C(21)-C(26)-N(1)	-167.8(8)	C(55)-C(51)-C(56)-N(3)	-166.0(8)
N(2)-C(30)-C(31)-C(32)	175.2(8)	N(4)-C(60)-C(61)-C(62)	178.4(8)
N(2)-C(30)-C(38)-C(37)	5(1)	N(4)-C(60)-C(68)-C(67)	3(1)
N(2)-C(30)-C(38)-C(39)	-175.9(7)	N(4)-C(60)-C(68)-C(69)	-178.9(7)

Elemental analysis was performed by a CHN-CORDERM7-3 auto-analyzer. Melting points were determined on a YANACO melting point apparatus and are uncorrected.

3.1. Spectroscopic data

KBr disk infrared spectra were obtained using a Nicolet-FTIR 5DX infrared spectrophotometer, ^1H NMR spectra were recorded on a JEOL FX-90Q NMR spectrometer with $CDCl_3$ solution using TMS as an internal standard. Mass spectra were obtained with a HP 5988A spectrometer operating at 70 eV.

IR data (cm^{-1}): ν^{α}_{N-H} 3394.1m, ν^{β}_{N-H} 3160–2900w, $\nu_{C=O}$ 1663.3w, $\nu_{C=S}$ 1343.0m, $\nu_{N-C=O}$ 1216.3w, δ_{N-H}, 1515.6s, $\nu_{N-C=S}$ 1146.5s. ^1H NMR (δ, ppm): 4.36 (s, 5H, C_5H_5), 4.60 (s, 2H, H^3, H^4-C_5H_4), 4.88 (s, 2H, H^2, H^5-C_5H_4), 7.44–8.20 (m, 7H, Np), 8.88 [s, 1H, N*H*–C(O)], 12.8 (s, 1H, N*H*–Np). MS m/z: 414 $(M)^+$, 271 $[FcC(O)N{=}C{=}S]^+$, 213 $(FcC\equiv O)^+$, 185 $(Fc)^+$, 121 $(C_5H_5Fe)^+$, 56 $(Fe)^+$

3.2. X-ray crystallography

Crystals suitable for X-ray examination were obtained by the slow evaporation of a benzene/ligroin solution. A single crystal with dimensions of about $0.15 \times 0.2 \times 0.2$ mm^3 was mounted on an Enraf-Nonius CAD4 diffractometer with graphite monochromatic Mo $K\alpha$ radiation ($\lambda = 0.071073$ nm). A total of 5129 independent reflections was collected within $2 \leq \theta \leq 22°$ of ω–2θ scan, at room temperature (23 ± 1°C), among which 2410 were accepted as observable $[I \geq 3\sigma(I)]$. the intensities were fully corrected for Lp factors and empirical absorption [9].

The crystal belongs to monoclinic space group $C2/c$, $a = 38.708(3)$, $b = 12.989(2)$, $c = 15.271(4)$ Å, $\beta = 95.48(2)°$, $V = 7643(4)$ Å^3, $M_r = 414.3$, $Z = 16$, $D_x = 1.44$ g/cm^3, $\mu = 9.06$ cm^{-1}, $F(000) = 3424$.

The crystal structure was solved by direct phase determination method (MULTAN 82). Two independent Fe atoms were located on an E-map. The other non-hydrogen atoms were found by successive difference Fourier syntheses. The hydrogen atoms were not included in the refinement or the calculations of structure factors. The coordinates and anisotropic thermal parameters of non-hydrogen atoms were refined by full-matrix least-squares method and converged to $R = 0.047$ and $R_w = 0.050$ with unit weights. The highest electron density on the final difference Fourier map was found to be 0.41 e Å^3.

All calculations were performed on a PDP11/44 computer using the SDP-PLUS program system.

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