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Synthesis, spectra and crystal structure of $(CO)_2(NO)Cr(\eta^5\text{-}C_5H_4)C(O)(\eta^5\text{-}C_5H_4)Fe(\eta^5\text{-}C_5H_5)$

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Abstract

Friedel–Crafts ferrocenoylation of (η^5-cyclopentadienyl)dicarbonylnitrosylchromium (**10**) (hereafter called cynichrodene) has afforded a new metallocenyl ketone ferrocenoylcyclopentadienyldicarbonylnitrosylchromium (**7**) in 72% yield. The structure of **7** has been solved by an X-ray diffraction study: space group $P2_1/n$, a 13.530(3), b 6.387(2), c 18.933(4) Å, β 101.09(2)° and $Z = 4$. **7** adopts a transoid conformation at the organic carbonyl carbon. The nitrosyl group of the cynichrodenyl moiety is located at the site away from the exocyclic organic carbonyl carbon with a twist angle 177.2°.

Introduction

Several metallocenyl ketones which contain two metallo-aromatic substituents are known including diferrocenyl ketone (**1**) [1], ferrocenyl ruthenocenyl ketone (**2**) [1], dicymantrenyl ketone (**3**) [2] and cymantrenyl ferrocenyl ketone (**4**) [2]. Compounds such as **1** have served as important precursors for the synthesis and structural elucidation of metallocenyl-stabilized carbonium ions [3–6]. X-ray structural investigations of both ketone **1** [7] and **2** [8] have been undertaken.

Since (η^5-carboxycyclopentadienyl)dicarbonylnitrosylchromium (**5**) (hereafter called cynichrodenoic acid) has been prepared easily from hydrolysis of (η^5-$C_5H_4COOCH_3$)$Cr(CO)_2NO$ (**6**) with potassium hydroxide in aqueous methanol [9], it is of interest to synthesize a new dimetallocenyl ketone: cynichrodenyl ferrocenyl ketone (**7**) by the Friedel–Crafts acylation of cynichrodenoyl chloride (**8**) with

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highly active metallo-aromatic ferrocene (9). Further, while the chemistry of the well-known dicarbonylcyclopentadienylnitrosyl complexes of chromium has become the subject of considerable study, the crystal structure and ^{13}C NMR of these complexes have almost been completely ignored [9–12]. Herein, we reported thorough spectral studies on compounds **7** and **8** and the crystal structure of **7**.

Experimental

All operations were carried out under a nitrogen atmosphere by means of Schlenk techniques. Trace oxygen in the nitrogen was removed by BASF catalyst and the deoxygenated nitrogen was dried with molecular sieve 3A and P_2O_5. Benzene and dichloromethane were dried over calcium hydride and freshly distilled under argon from calcium hydride. All other solvents were used as commercially obtained.

Column chromatography was carried out under nitrogen using Merck Kiesel-gel 60. The silica gel was heated with a heat gun while mixing on a rotary evaporator attached to a vacuum pump for 2 h to remove water and oxygen. The silica gel was stored under nitrogen until use. Cynichrodenoic acid was prepared according to the literature procedure [9].

^1H and ^{13}C (300 and 400 MHz) NMR were obtained on a Bruker AM-300-WB or AM-400 spectrometer. ^1H and ^{13}C were referenced to tetramethylsilane. Infrared spectra were recorded on a Perkin–Elmer 682 spectrophotomer. Microanalyses were performed by the Microanalytical Laboratory at National Taiwan University.

Preparation of [η^5-(ferrocenoyl)cyclopentadienyl)]dicarbonylnitrosylchromium (cynichrodenyl) ferrocenyl ketone) (7)

Cynichrodenoic acid (1.0 g, 4.05 mmol) was stirred with phosphorus pentachloride (0.93 g, 4.47 mmol) in 50 ml of dry methylene chloride for 2 h at room temperature. Subsequently, aluminium chloride (0.54 g, 4.05 mmol) was added and the mixture stirred for another 2 h. The solution was filtered from excess $AlCl_3$ and the filtrate was added dropwise to a solution of ferrocene (0.87 g, 4.67 mmol) in 25 ml methylene chloride at 0 ° C. After the addition was completed, the reaction mixture was stirred at room temperature for 12 h. The reaction was then cooled to 0 ° C and slowly hydrolyzed with 50 ml of ice followed by 5 drops of concentrated hycrochloric acid. The aqueous and organic layers were separated, and the aqueous layer was extracted twice with methylene chloride. The combined organic layer was washed one with water, one with sodium bicarbonate solution, once again with water, and dried with anhydrous magnesium sulfate. The solution was filtered, concentrated to 50 ml under vacuum, silica gel (20 g) added, and the solvent removed under vacuum. The residue was added to a dry-packed column (2 × 15 cm) of silica gel. Elution of column with benzene gave a dark red band which upon removal of the solvent under vacuum gave cynichrodenyl ferrocenyl ketone, 1.21 g (72%). An analytical sample, m.p. 139–140 ° C, was obtained by vacuum sublimation at 120 ° C/0.1 torr. An X-ray sample was obtained by solvent expansion from pentane/hexane/methylene chloride at 0 ° C.

Analysis: Found: C, 51.77; H, 3.44; N, 3.32. $C_{18}H_{13}CrFeNO_4$ calcd. 52.08; H, 3.16; N, 3.37%. Proton NMR ($CDCl_3$): δ (relative intensity, multiplicity, assignment): 4.23 (5H, s, Cp^2(Fe) unsubstituted ring protons): 4.54(2H, t, Cp^1(Fe) H(3,4));

4.83(2H, t, Cp^1(Fe) H(2,5)); 5.15(2H, t, Cp(Cr) H(3,4)); 5.82(2H, t, Cp(CR) H(2,5)). Carbon-13 NMR ($CDCl_3$): δ (assignment): 70.24 (Cp^2(Fe)); 70.43 (Cp^1(Fe), C(2,5)); 72.25 (Cp^1(Fe), C(3,4)); 78.39 (Cp^1(Fe), C(1)); 91.06 (Cp(Cr), C(3,4)); 93.89 (Cp(Cr), C(2,5)); 103.03 (Cp(Cr), C(1)); 192.52 (C(O)); 234.76 ($Cr{-}C{\equiv}O$). IR spectrum (CH_2Cl_2); cm^{-1} (intensity): 2030(s), 1965(s), 1710(s), 1630(m), 1490(w), 1469(m), 1382(m), 1300(m), 1258(m), 1205(w), 1178(w), 1120(w), 1055(w), 830(w), 630(m). Mass spectrum: $m/e = 415$ (M^+).

Table 1

Summary of crystal data and intensity collection

Empirical formula	$C_{18}H_{13}NO_4CrFe$
Color; habit	Red; rod
Crystal size (mm)	0.42 mm × 0.20 mm × 0.18 mm
Space group	Monoclinic; $P2_1/n$
Unit cell dimensions	a 13.530(3) Å
	b 6.387(2) Å
	c 18.993(4) Å
	β 101.09(2)°
Volume	1610.7(6) Å^3
Z	4
Formula weight	415.1
Density (calc.)	1.712 Mg/m^3
Absorption coefficient	15.81 cm^{-1}
$F(000)$	840
Diffractometer used	Nicolet R3m/V
Radiation	Mo-K_α (λ 0.71073 Å)
Temperature (K)	297
Monochromator	Highly oriented graphite crystal
2θ range	2.0 to 50.0°
Scan type	$\theta/2\theta$
Scan speed	Variable; 2.93 to 14.65°/min. in ω
Scan range (ω)	1.10° plus K_α-separation
Background measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 50.0% of total scan time
Standard reflections	3 measured every 50 reflections
Index ranges	$-1 \leqslant h \leqslant 15$, $0 \leqslant k \leqslant 7$, $-22 \leqslant l \leqslant 22$
Reflections collected	3539 ($2196 > 3.0\sigma(I)$)
Independent reflections	2851 ($1879 > 3.0\sigma(I)$)
Absorption correction	N/A
Extinction correction	$\chi = 0.00036(8)$, where $F^* = F\,[1+0.002\chi F^2/\sin(2\theta)]^{-1/4}$
Hydrogen atoms	Riding model, fixed isotropic U
Weighting scheme	$w^{-1} = \sigma^2(F) + 0.0005F^2$
Final R indices (obs. data)	R 3.25%, R_w 3.24%
Goodness-of-fit	1.09
Largest and mean Δ/σ	0.061, −0.001
Data-to-parameter ratio	7.1 : 1
Largest difference peak	0.30 eÅ^{-3}
Largest difference hole	−0.31 eÅ^{-3}

Preparation of (η^5-chloroformylcyclopentadienyl)dicarbonylnitrosylchromium (cynichrodenoyl chloride) (8)

Cynichrodenoic acid (1.0 g, 4.05 mmol) was stirred with phosphorus pentachloride (0.93 g, 4.47 mmol) in 50 ml of dry benzene for 2 h at room temperature. The reaction mixture was filtered and the filtrate concentrated in vacuum at 50 ° C to remove the benzene and phosphorus oxychloride, leaving 1.01 g (94%) of cynichrodenoyl chloride as a red-brown residue. An analytical samples,. m.p. 42.5 ° C, was obtained by vacuum sublimation at 50 ° C/0.1 torr.

Analysis: Found: C, 36.29; H, 1.62;N. 5.15. $C_8H_4ClCrNO_4$ calcd.: C, 36.18; H, 1.52; N, 5.27%. Proton NMR ($CDCl_3$): 8gd (relative intensity, multiplicity, assignment): 5.23 (2H, t, Cp H(3,4)); 5.91 (2H, t, Cp H(2,5)). Carbon-13 NMR ($CDCl_3$): δ (assignment): 92.12 (Cp, C(3,4)); 93.96 (Cp, C(1)): 95.35 (Cp, C(2,5)); 161.99 (C(O)); 231.27 (Cr–*C*–O). IR spectrum ($CDCl_3$): cm^{-1} (intensity): 3120(w), 2036(vs), 1980(vs), 1765(s), 1720(vs), 1550(w), 1446(m), 1370(m), 1256(s), 1050(m), 1040(m), 938(m), 790(s), 620(m). Mass spectrum: $m/e = 265$ (M^+).

X-ray diffraction analysis of 7

The intensity data were collected on a Nicolet R3m/V diffractometer with a graphite monochromator (Mo-K_α radiation). $\theta/2\theta$ scan data were collected at room temperature (24 ° C). The data were corrected for Lorentz and polarization effects. The details of crystal data and intensity collection are summarized in Table 1.

The structure was solved by direct methods using SHELXTL PLUS program [13]. Full matrix least-squares refinement on F used anomalous-dispersion corrections for atoms with atomic numbers greater than 16. Neutral atom scattering form factors from International Tables for X-ray Crystallography [14] were used. The non-hydrogen atoms were refined anisotropically and all hydrogen atoms were positioned at calculated coordinates and with fixed isotropic U of 0.08 Å^2. All calculations were performed on a MicroVAX II based Nicolet SHELXTL PLUS system.

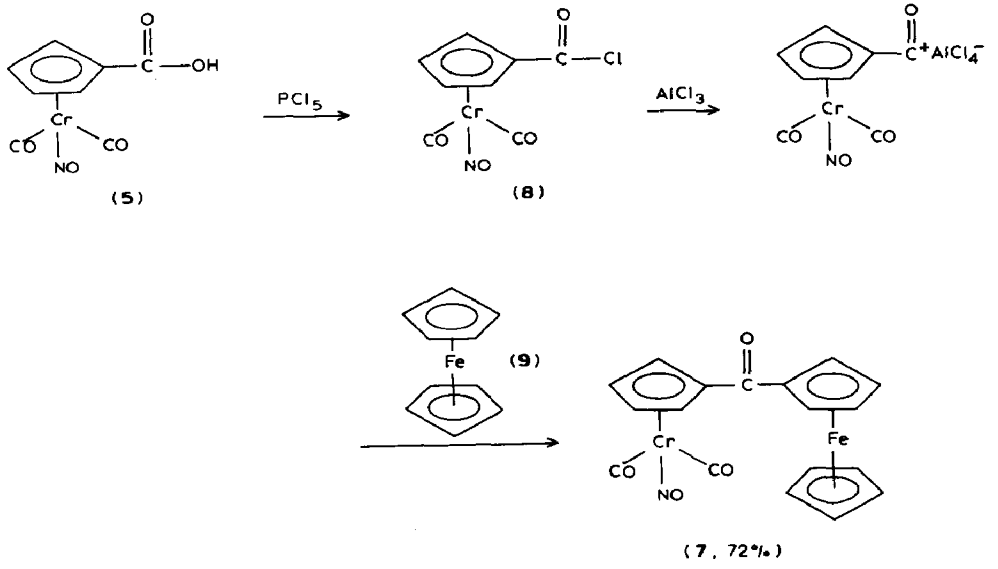
Results and discussion

By reacting with phosphorus pentachloride, cynichrodenoic acid (**5**) [9] was transformed into the acid chloride **8** which formed the Perrier-type complex [10] with aluminum chloride that subsequently reacted with ferrocene **9** to result in the cynichrodenyl ferrocenyl ketone (**7**) in 72% yield.

The infrared and ^{1}H NMR spectra of complex **7** are consistent with its assigned structure and are similar to other metallocenyl systems [10,15–17].

It is of interest to compare the ^{1}H NMR spectrum of **7** with those of ferrocene (**9**) and cynichrodene (**10**). The chemical shifts of protons on both Cp^1(Fe) (δ4.54, H(3,4); 4.83, H(3,5)) and Cp(Cr) (δ 5.15, H(3,4); 5.82 (H(2,5)) occur at a much lower field than those of the corresponding protons of **9** (δ 4.18) and **10** (δ 5.07). This reflects the strong electron-withdrawing effect of the bridging $\rangle$C=O groups.

The assignments of ^{13}C NMR spectra for compound **7** are based on standard ^{13}C NMR correlations [18]; 2D-HetCOR; DEPT technique and compared with other metallo-aromatic system [19]. Four relatively less intense signals were observed at δ 234.76, 192.52, 103.03 and 78.39 corresponding to terminal carbonyl carbon, organic carbonyl carbon, C(1) of Cp(Cr) and C(1) of Cp^1(Fe) respectively. Carbons of



Cp^2(Fe) resonate at δ 70.24. The line assignments for C(2–5) of Cp(Cr) and Cp^1(Fe) were more difficult to make. Based on 2D-HetCOR (Fig. 1) chemical shifts at δ 91.06 and 93.89 were assigned to C(3,4) and C(2,5) of Cp(Cr) ring respectively, at δ 70.43 and 72.25 were assigned to C(2,5) and C(3,4) of Cp^1(Fe) respectively.

One surprising finding in the study of ^{13}C spectra of 7 is that the highfield and lowfield chemical shifts are assigned to C(2,5) and C(3,4), respectively, for electron-

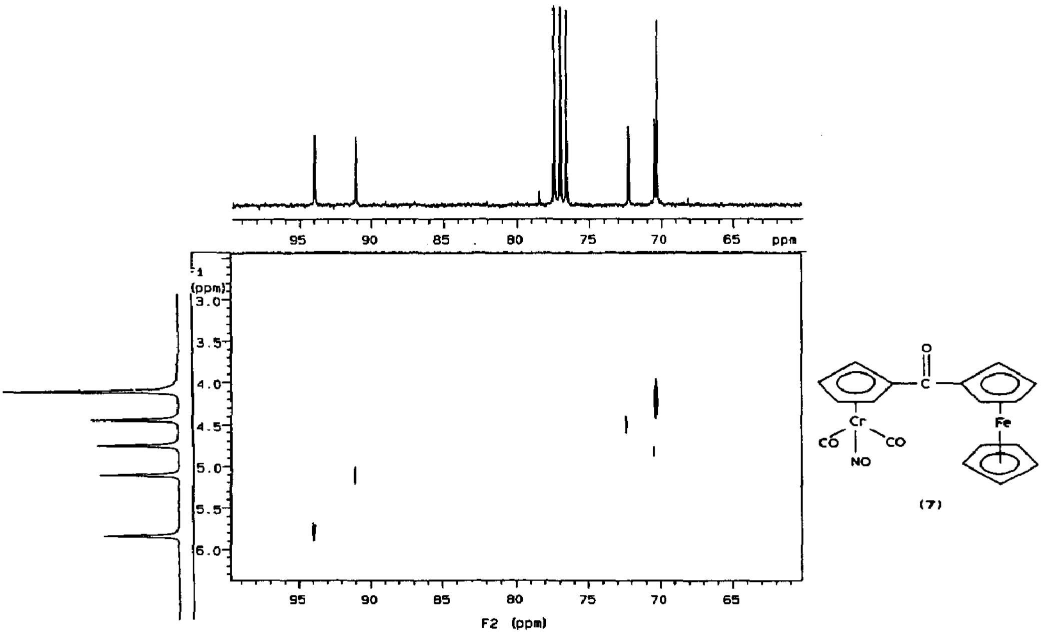


Fig. 1. Two-dimensional ^{1}H-^{13}C HetCOR NMR spectrum of 7 in CDCl$_3$.

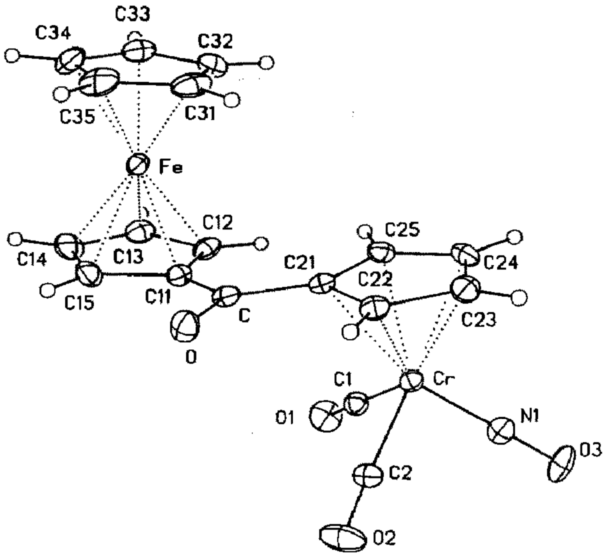


Fig. 2. Molecular configuration of 7.

Table 2

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($Å^2 \times 10^3$)

	x	y	z	U_{eq} [a]
Fe	−3313(1)	2625(1)	5755(1)	33(1)
Cr	975(1)	1630(1)	6566(1)	33(1)
O	−1449(2)	−1660(5)	6389(2)	52(1)
O(1)	478(3)	2671(6)	4997(2)	66(1)
O(2)	1464(3)	−2811(5)	6293(2)	74(2)
O(3)	3058(2)	2832(6)	6674(2)	70(1)
N(1)	2209(3)	2349(6)	6633(2)	46(1)
C	−1354(3)	209(7)	6258(2)	37(2)
C(1)	659(3)	2266(7)	5590(2)	39(1)
C(2)	1259(3)	−1121(7)	6382(2)	39(2)
C(11)	−2024(3)	1154(7)	5639(2)	36(1)
C(12)	−2066(3)	3220(8)	5341(2)	42(2)
C(13)	−2933(3)	3372(9)	4793(2)	48(2)
C(14)	−3427(4)	1427(9)	4741(2)	56(2)
C(15)	−2885(4)	55(8)	5254(3)	50(2)
C(21)	−577(3)	1421(7)	6744(2)	34(1)
C(22)	81(3)	438(7)	7334(2)	37(1)
C(23)	748(3)	1959(8)	7685(2)	46(2)
C(24)	518(4)	3885(8)	7328(3)	46(2)
C(25)	−290(3)	3585(7)	6748(2)	41(2)
C(31)	−3252(4)	2937(11)	6830(3)	65(2)
C(32)	−3370(4)	4927(9)	6497(3)	56(2)
C(33)	−4267(4)	4883(9)	5988(3)	57(2)
C(34)	−4704(4)	2930(10)	6001(3)	62(2)
C(35)	−4087(5)	1720(10)	6520(3)	69(2)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 3

Selected bond distances (Å) and angles (deg) of 7

Bond distances			
Fe–C(11)	2.029(4)	Fe–C(12)	2.030(5)
Fe–C(13)	2.048(5)	Fe–C(14)	2.050(5)
Fe–C(15)	2.036(5)	Fe–C(31)	2.036(5)
Fe–C(32)	2.047(6)	Fe–C(33)	2.040(6)
Fe–C(34)	2.034(6)	Fe–C(35)	2.031(7)
Cr–C(21)	2.194(4)	Cr–C(22)	2.203(5)
Cr–C(23)	2.216(5)	Cr–C(24)	2.213(5)
Cr–C(25)	2.199(5)	C(11)–C(12)	1.432(6)
C(11)–C(15)	1.433(6)	C(12)–C(13)	1.414(6)
C(13)–C(14)	1.405(8)	C(14)–C(15)	1.407(7)
C(21)–C(22)	1.435(5)	C(21)–C(25)	1.435(6)
C(22)–C(23)	1.404(6)	C(23)–C(24)	1.411(7)
C(24)–C(25)	1.407(6)	C(31)–C(32)	1.415(8)
C(31)–C(35)	1.404(8)	C(32)–C(33)	1.398(7)
C(33)–C(34)	1.383(8)	C(34)–C(35)	1.396(8)
Cr–N(1)	1.712(4)	Cr–C(2)	1.846(4)
Cr–C(1)	1.865(4)	N(1)–O(3)	1.178(5)
C(2)–O(2)	1.135(5)	C(1)–O(1)	1.136(5)
C–C(11)	1.470(5)	C–C(21)	1.477(5)
C–O	1.231(6)	Cr–cen.Cp(Cr)	1.846
Cr...C	3.223	Fe...C	3.057
cen.(Cp^1(Fe))...cen.(Cp^2(Fe))			3.297
H(C(12))...H(C(25))			2.146
Bond angles			
C(11)–C(12)–C(13)	108.5(4)	C(12)–C(13)–C(14)	107.9(4)
C(12)–C(11)–C(15)	106.5(3)	C(13)–C(14)–C(15)	108.9(4)
C(11)–C(15)–C(14)	108.2(4)	C(21)–C(22)–C(23)	108.3(4)
C(22)–C(21)–C(25)	106.9(3)	C(22)–C(23)–C(24)	108.2(4)
C(23)–C(24)–C(25)	108.9(4)	C(21)–C(25)–C(24)	107.7(4)
C(32)–C(31)–C(35)	107.3(5)	C(31)–C(32)–C(33)	107.4(5)
C(32)–C(33)–C(34)	108.9(5)	C(33)–C(34)–C(35)	108.2(5)
C(31)–C(35)–C(34)	108.2(5)	N(1)–Cr–C(2)	92.0(2)
N(1)–Cr–C(1)	92.9(2)	C(1)–Cr–C(2)	92.1(2)
C(11)–C–C(21)	122.7(4)	Cr–N(1)–O(3)	179.4(3)
Cr–C(2)–O(2)	177.2(4)	Cr–C(1)–O(1)	179.0(4)
C–C(11)–C(12)	131.7(4)	C–C(11)–C(15)	121.5(4)
C–C(21)–C(22)	121.2(4)	C–C(21)–C(25)	131.9(4)
C(11)–C–O	119.1(3)	C(21)–C–O	118.2(3)
cen.–Cr–C(1)	121.1°	cen.–Cr–C(2)	120.4°
cen.–Cr–N(1)	129.1°		

withdrawing substituents on a Cp ring of ferrocene derivatives which is analogous to literature reported [19]. However, the opposite assignment in which the downfield shifts and upfield shifts are assigned to C(2,5) and C(3,4) respectively was made for those substituents on Cp ring of cynichrodene derivatives.

The molecular structure of compound 7 is shown in Fig. 2. The atomic coordinates of the non-hydrogen atoms are listed in Table 2. Selected bond distances and angles are given in Table 3.

Compound 7 adopts a transoid conformation at the organic carbonyl carbon. The coordination geometry about the Cr center is approximately a distorted

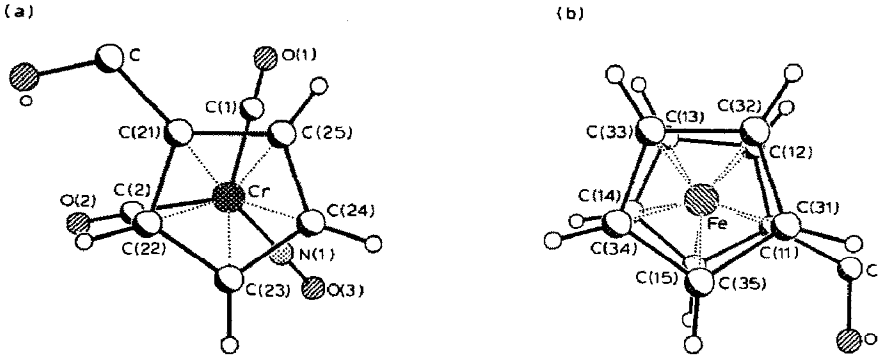


Fig. 3. (a) View of part of 7 along the normal of Cp (Cr) ring and Cr. (b) View of part of 7 along the normal to planes of Cp^1(Fe) and Cp^2(Fe) rings.

tetrahedron with two carbonyl groups, the Cp group and nitrosyl group as the four coordination sites. The nitrosyl group of the cynichrodenyl moiety is located at the site away the exocyclic organic carbonyl carbon with a twist angle 177.2° (Fig. 3). The twist angle is defined as the torsional angle between the nitrogen atom, the chromium atom, the Cp ring center and the ring carbon atom bearing exocyclic carbon.

In the cynichrodene moiety, the observed average bond lengths of Cr–C(ring) 2.205 Å, compare favorably with the 2.188(5) Å average found in $(\eta^5\text{-}C_5H_5)Cr(CO)_2NO$ [11] and with the 2.20(1) Å average value in $[(\eta^5\text{-}C_5H_5)Cr(CO)_3]_2$ [20] and in $(\eta^5\text{-}C_5H_5)Cr(NO)_2Cl$ [21]. The Cr–N length 1.712(4) Å, falls in the range of reported values, 1.687(7) Å in $(\eta^5\text{-}C_{13}H_9)Cr(CO)_2NO$ [11] to 1.72(1) Å in $(\eta^5\text{-}C_5H_5)Cr(NO)_2(NCO)$ [22]. The Cr–C (carbonyl) distance: 1.864(4) Å (Cr–C(1)); 1.846(4) Å (Cr–C(2)) agree well with the 1.864(6) Å found in $(\eta^5\text{-}C_{13}H_9)Cr(CO)_2NO$ and 1.86 Å found in $[(\eta^5\text{-}C_5H_5)Cr(CO)_3]_2$ [21]. The N≡O length of 1.178(5) Å (N(1)–O(3)) is longer than the C≡O distances of 1.135(5) (C(1)–O(1)), 1.135(5) (C(2)–O(2)), in keeping with the greater antibonding population in the nitrosyl ligand. The Cr–N–O angle of 179.4(3)° (Cr–N(1)–O(3)) is consistent with the NO^+ formalism typical of linear M–NO linkage, while the Cr–C–O angle of 179.0(4) (Cr–C(1)–O(1)), 177.2(4) (Cr–C(2)–O(2)) indicate the usual mode of bonding in the terminal metal carbonyl complexes. The Cr–centroid(Cp(Cr)) distance is 1.846 Å, agrees with the 1.844 Å in $[(\eta^5\text{-}C_5H_5)Cr(CO)_2NO]$ and 1.884 Å in $(\eta^5\text{-}C_{13}H_9)Cr(CO)_2NO$ [11]. The average C–C distance in the ring (Cp(Cr)) is 1.418 Å. The mean angle in the ring is 108°.

The two cyclopentadienyl rings of ferrocene moiety exhibit an average twist angle of 5.6° (Fig. 3b). The twist angle is defined by Palenik [23] as the torsional angle between a ring carbon, the two ring centers and the corresponding carbon on the opposite ring. It is apparent that compound 7 is close to the eclipsed configuration which is in good agreement with other ferrocenyl compounds [7,24]. There is a 2.5° offset from parallelism between the two Cp rings of the ferrocene moiety and those are separated by 3.30 Å. The mean bond distances of ferrocenyl moiety in compound 7 are very similar to those in related molecules [24]. The average Fe–C(ring) distance is 2.04 Å, the average C–C distances in the rings are C–C(Cp^1(Fe)) 1.42 Å, C–C(Cp^2(Fe)) 1.40 Å, the exocyclic C–C bond measures 1.470(5) (C–C(11)), 1.477(5) (C–C(21)) and the mean angle in the rings is 108°.

The organic C=O bond length is 1.231(6) Å, and the angles at this carbonyl group (119, 118 and 123°) do not differ from normal values. The exocyclic carbon (C) is bent toward both metals, Cr and Fe, with θ angles of 1.04 and 5.02° respectively. The θ angle is defined as the angle between the exocyclic C–C bond and the Cp ring with positive angle toward metal. The carbonyl plane (C(11), C, O, C(21)) turns away from the corresponding ring planes Cp^1(Fe) and Cp(Cr) by 9.4 and 1.6°, respectively. These rotations are the result of intramolecular steric interference between atoms H(C(12)) and H(C(25)). This is supported by the enlargement of bond angles (C(12)–C(11)–C) and (C(25)–C(21)–C) to 131.7 and 131.9, respectively.

Supplementary material available. List of anisotropic temperature factors of non-hydrogen atoms and the coordinates with isotropic temperature factors of hydrogen atoms as well as list of structure amplitudes (11 pages) are deposited. Ordering information can be obtained from the authors.

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