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Iron dithiocarbonate complexes: Structure of CpFe(CO)₂SC(S)O-4-C₆H₄Cl

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Abstract

A group of dithiocarbonate iron complexes $CpFe(CO)_2SC(S)OAr$ (1) were prepared by the reaction of $(\mu - S_x)[CpFe(CO)_2]_2$ with arylchlorothionoformates (Ar = Ph (1a), 4-C₆H₄Cl (1b), 4-C₆H₄F (1c), C₆F₅ (1d), 4-C₆H₄CH₃ (1e)). The conversion of 1 into $CpFe(CO)(\kappa^2 S, S-SC(S)OAr)$ (2), in which the dithiocarbonate ligand is bonded to the iron through the two S-atoms, can be achieved photolytically. The structure of $CpFe(CO)_2SC(S)O-4-C_6H_4Cl$ (1b) is reported. © 2005 Elsevier Ltd. All rights reserved.

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1. Introduction

Dithioacid ligands, $X-CS_2^-$ (X = OR, SR, NR₂), have a rich coordination and organometallic chemistry [1–3]. These ligands are obtained easily by the addition of the nucleophiles (RO⁻, RS⁻, NHR₂) to carbon disulfide [4–10]. Dithioacid ligands and their metal complexes have obtained continuous attraction due to their interesting structural and chemical properties as well as their wide range of application in biological systems [11–13].

Transition metal complexes with dithioacid ligands are very common in the literature [4–16]. They have been generally prepared by metatheses reaction of transition metal halides with the free ligands. In most cases the ligand is bonded to the metal in a chelate fashion through the two S-atoms. However, several complexes in which the mode of coordination of the dithioacid ligands is monodentate were obtained and characterized [14,15].

A variety of complexes containing dithioacid ligands with cyclopentadienyl iron system has been reported. Treatment

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of the iron chloride complex, CpFe(CO)₂Cl, with sodium dialkyldithiocarbamates in acetone gave $CpFe(CO)_2(\kappa S S_2CNR_2$) (R = Me, Et), which contains a unidentate dialkyldithiocarbamates group [16,17]. The same products were also obtained from the reaction of the iron dimer, [CpFe(CO)₂]₂, amines (NHR₂) and CS₂ in refluxing THF, or by concerted CS₂ insertion into the Fe-N bond of the $[CpFe(CO)_2(NHR_2)]^+$ salt in the presence of a base [14]. The chelate complex CpFe(CO)($\kappa^2 S$, S-S₂CNMe₂) is obtained from the reaction of the dimer with tetramethylthiuram disulfide, [(CH₃)₂NCS₂]₂. The ethyl-dithiocarbonate complexes $Cp'Fe(CO)_2(\kappa S-S_2COEt)$ ($Cp' = C_5H_5$, C_5Me_5) were prepared from the reaction of the corresponding iron dimers [Cp'Fe(CO)₂]₂ with [EtOC(S)S]₂ in cyclohexane [18,19]. Photolysis of these complexes gave $Cp'Fe(CO)(\kappa^2 S, S-S_2COEt)$ which have a bidentate ligand. Treatment of $Cp'Fe(CO)_2(\kappa S-S_2COEt)$ with phosphine or phosphite ligands gave the mixed carbonyl-phosphine or phosphite complexes $Cp'Fe(CO)(L)(\kappa S-S_2COEt)$ (L = PBu₃, PPh₃, P(OEt)₃, P(OPh)₃) [20]. The trithiocarbonate complexes $CpFe(CO)_2(\kappa S-S_2CSR)$ have been prepared from CpFe(CO)₂Cl and the in situ generated anions RSCS₂⁻ (R = Me, Et, Ph). These complexes can be converted to the chelated form, CpFe(CO)($\kappa^2 S$, S-S₂CSR) by photolysis [21].

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In the framework of synthetic studies of cyclopentadienyl iron sulfur complexes, we have studied the reactions of the iron sulfides $(\mu$ -S_x)[CpFe(CO)₂]₂ with a variety of nucleophiles such as acid chlorides, sulfonyl chlorides and chloroformates. These reactions give the sulfur containing complexes, thiocarboxylates (CpFe(CO)₂SCOR) [22–24], thiosulfonates (CpFe(CO)₂SSO₂R) [25] and thiocarbonates (CpFe(CO)₂SCO₂R) [26]. We hereby report the synthesis of dithiocarbonate complexes of iron by the reaction of the iron sulfides, (μ -S_x)[CpFe(CO)₂]₂, with aryl chlorothionoformates.

2. Results and discussion

2.1. Synthesis and characterization

The reaction of $(\mu$ -S_x)[CpFe(CO)₂]₂ with various aryl chlorothionoformates proceeds smoothly to give the corresponding dithiocarbonate complexes CpFe(CO)₂SC(S)OAr (1) as shown in Eq. (1). The iron chloride, CpFe(CO)₂Cl is obtained as a by-product of this reaction and can be separated from the products 1 by column chromatography.



Ar = Ph (a), $4-C_6H_4Cl$ (b), $4-C_6H_4F$ (c), C_6F_5 (d), $4-C_6H_4CH_3$ (e)

The new organoiron S-bonded dithiocarbonates (1) are stable solids and are also stable in solution. Structural features of 1 were identified on the basis of IR and ¹H NMR spectroscopy as well as by elemental analysis. The IR spectra of 1 in the carbonyl stretching region show two bands due to the terminal carbonyl groups in the ranges 2044-2049 and 1998-2006 cm⁻¹. These bands are higher than those reported for the corresponding thiocarboxylates CpFe(CO)₂SCOR (2027-2060, $1984-1998 \text{ cm}^{-1}$) [22-24] and are similar to those of thiocarbonate complexes CpFe(CO)₂SCO₂R (2044-2045 and 1997–1999 cm⁻¹) [26], suggesting that the mono- and di-thiocarbonate ligands have similar basicity. In the ¹H NMR spectra of 1, the peak for the Cp-protons is found in the range 5.05–5.10 ppm. This chemical shift range is similar to that observed for the thiocarboxylate (4.98–5.13 ppm) [22–24] and thiocarbonate (4.98–5.12 ppm) analogues [26]. These results suggest a similar electron density around the iron center in all of these complexes.

When a THF solution containing $CpFe(CO)_2SCSOAr$ (1) was irradiated by UV-light for short time (ca. 30 min), the chelate complexes $CpFe(CO)(\kappa^2 S, S-S_2COAr)$ (2) were obtained in high yields (Eq. (2)).



Compounds 2 were characterized by IR, ¹H NMR spectroscopy and elemental analysis. The IR spectrum of each of complexes 2 in the carbonyl stretching region exhibits a singlet band in the range 1953-1961 cm⁻¹ for the terminal carbonyl group coordinated to the iron center. This range is comparable to those found for iron complexes containing one CO-group such as CpFe- $(CO)(EPh_3)SCOR (1950-1970 \text{ cm}^{-1})$. ¹H NMR spectra of 2 in CDCl₃ show singlet peak in the range 4.63-4.69 ppm for the cyclopentadienyl protons. This peak has lower chemical shift compared to the corresponding peak of the starting dicarbonyl complexes 1. This shift may attributed to the higher electron-density around the Fe-atom in 2 compared to that in 1. Comparing this peak to that of CpFe(CO)(EPh₃)SCOR, it is obvious that these values are comparable to each other.

2.2. Crystal structure

The structure of $CpFe(CO)_2SC(S)O-4-C_6H_4Cl$ (1b) was determined by single-crystal X-ray analysis and is shown in Fig. 1. Selected bond lengths and angles are shown in Table 1. The relevant bond parameters are listed in Table 2. As expected, the analysis proved that the dithiocarbonate ligand is bonded to the iron in a monodentate fashion through one sulfur atom with an Fe-S bond distance of 2.2765(5) A. This distance is longer than the corresponding Fe-S distance in CpFe(CO)₂SCO₂Et (2.2675(10) Å) and in $CpFe(CO)_2SCO-2-C_6H_4NO_2$ (2.266(1) Å) The Cp ligand is bound to the metal in an η^5 -fashion with Fe–C bond distances comparable to those observed for Cp-iron complexes. The Fe-C (of carbonyl ligands) bond distances of 1.7745(19) and 1.7810(17) Å are in the normal range for CpFe(CO)₂X complexes [24,26–28]. The Fe–S1–C8 angle is 113.50°.

3. Experimental

3.1. General

All reactions were carried out under an inert atmosphere of nitrogen using standard Schlenk line techniques. Solvents were dried by standard methods and were distilled under nitrogen prior to use. The complexes $(\mu$ -S_x)-[CpFe(CO)₂]₂ (x = 2, 3) were prepared by published method [29]. The reagents: aryl-chlorothionoformates,



Fig. 1. ORTEP drawing of CpFe(CO)₂SC(S)O-4-C₆H₄Cl (1b).

Table 1 Selected bond length (Å) and angles (°) of $CpFe(CO)_2SC(S)O$ -4-C₆H₄Cl (1b)

Fe-S1	2.2765(5)	C8–S1–Fe	113.50(5)
Fe-C1	1.77745(19)	C1-Fe-S1	89.25(6)
Fe–C2	1.7810(17)	C2-Fe-S1	94.92(5)
Fe–C3	2.0785(19)	C2-Fe-C1	92.24(9)
Fe–C4	2.1003(19)	C8–O3–C9	120.75(12)
Fe–C5	2.110(2)	O3–C8–S2	124.53(11)
Fe–C6	2.089(2)	O3-C8-S1	114.36(11)
Fe–C7	2.0818(19)	S1-C8-S2	121.10(9)
S1-C8	1.7095(16)		
S2–C8	1.6541(15)		
C1-O1	1.139(2)		
C2–O2	1.139(2)		
C3–O3	1.3553(18)		
C9–O3	1.4036(18)		

[CpFe(CO)₂]₂, and sulfur were obtained from Acros and were used as received.

Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker 200 MHz spectrometer. Chemical shifts are in ppm relative to TMS at 0 ppm. Infrared (IR) spectra were recorded on a Nicolat Nexus FT-IR spectrometer using NaCl solvent cells. Elemental analyses were done in Laboratoire d'Analyse Elémentaire, Université de Montréal, Montréal, Que., Canada. Melting points were reported on an electrothermal melting point apparatus and are uncorrected. The photolytic reactions were carried out in a medium pressure mercury lamp (150 W) with a quartz immersion cell.

3.2. General procedure for the preparation of $CpFe(CO)_2SC(S)OAr(1)$

To a solution of $(\mu$ -S_x)[CpFe(CO)₂]₂ (0.50 mmol) in diethylether (100 mL), aryl chlorothionoformate (0.55 mmol) was added and the resulting mixture was stirred overnight at room temperature. The volatiles were removed under reduced pressure and the resulting solid was redissolved in a minimum amount of CH₂Cl₂ and transferred to a silica gel column made up in hexane. The column was first

Table 2 Selected crystal data and refinement parameters for CpFe(CO)₂SC(S)O-4-C₄H₄Cl (1b)

Empirical formula	C ₁₄ H ₉ ClFeO ₃ S ₂	
Formula weight	380.63	
Crystal size (mm)	$0.13 \times 0.10 \times 0.08$	
Crystal system	monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions		
<i>a</i> (Å)	11.4680 (10)	
$b(\mathbf{A})$	9.7400 (10)	
c (Å)	13.678 (10)	
α (°)	90.00	
β (°)	103.21(5)	
γ (°)	90.00	
$V(\dot{A}^3)$	1487.4 (2)	
Z	4	
Index ranges	$-16 \leqslant h \leqslant 9$,	
	$0 \leqslant k \leqslant 13, \ 0 \leqslant l \leqslant 19$	
Number of total reflections	4334	
Number of unique	3711	
reflections ($\geq 2\sigma(I)$)		
$D_{\rm calc} ({\rm Mg}{\rm m}^{-3})$	1.855	
Radiation type	Μο Κα	
$\mu (\mathrm{mm}^{-1})$	1.478	
λ (Å)	0.71069	
θ Range (°)	1.82-30.05	
$R[F^2 > 2\sigma(F^2)]$	0.0280	
$\omega R(F^2)^{\rm a}$	0.0765	
$1/[2/(\pi^2) + (0.042(\pi)^2)]$	$1 - D = (D^2 + 2D^2)/2$	

^a $\omega = 1/[\sigma^2 (F_o^2) + (0.0426P)^2]$, where $P = (F_o^2 + 2F_c^2)/3$.

eluted with hexane to remove any unreacted aryl chlorothionoformates. Then, it was eluted with hexane/dichloromethane solution (1:1 v:v ratio) to give a red-orange band which was collected and identified as $CpFe(CO)_2SC(S)OAr$, followed by a red band which was also collected and identified as $CpFe(CO)_2Cl$. The $CpFe(CO)_2SC(S)OAr$ (1) were recrystallized from dichloromethane/hexane.

3.2.1. $CpFe(CO)_2SC(S)OC_6H_5$ (1a)

Yield, 79%; m.p. 132–133 °C. *Anal.* Required for $C_{14}H_{10}FeO_3S_2$: C, 48.57; H, 2.91; S, 18.53. Found: C, 47.78; H, 2.65; S, 18.03%. IR (CH₂Cl₂, cm⁻¹): 2045s,

1998s $v(C \equiv O)$. ¹H NMR (CDCl₃, δ , ppm): 5.05 (s, 5H, C₅H₅), 7.07 (m, 3H, C₆H₅), 7.31 (m, 2H, C₆H₅).

3.2.2. $CpFe(CO)_2SC(S)O-4-C_6H_4Cl$ (1b)

Yield, 87%; m.p. 134–136 °C. *Anal.* Required for $C_{14}H_9CIFeO_3S_2$: C, 44.18; H, 2.38; S, 16.58. Found: C, 44.10; H, 2.23; S, 15.98%. IR (CH₂Cl₂, cm⁻¹): 2046s, 1999s ν (C=O). ¹H NMR (CDCl₃, δ , ppm): 5.05 (s, 5H, C_5H_5), 6.95 (d, 2H, C_6H_4), 7.32 (d, 2H, C_6H_4).

3.2.3. $CpFe(CO)_2SC(S)O-4-C_6H_4F(1c)$

Yield, 86%; m.p. 128–129 °C. *Anal.* Required for $C_{14}H_9FFeO_3S_2$: C, 46.17; H, 2.49; S, 17.61. Found: C, 45.88; H, 2.28; S, 17.01%. IR (CH₂Cl₂, cm⁻¹): 2046s, 2000s ν (C=O). ¹H NMR (CDCl₃, δ , ppm): 5.09 (s, 5H, C₅H₅), 7.00 (d, 2H, C₆H₄), 7.40 (d, 2H, C₆H₄).

3.2.4. $CpFe(CO)_2SC(S)OC_6F_5$ (1d)

Yield, 90%; m.p. 115–116 °C. *Anal.* Required for $C_{14}H_5F_5FeO_3S_2$: C, 38.55; H, 1.16; S, 14.70. Found: C, 38.22; H, 0.99; S, 14.13%. IR (CH₂Cl₂, cm⁻¹): 2049s, 2006s $v(C\equiv O)$. ¹H NMR (CDCl₃, δ , ppm): 5.10 (s, H's, C₅H₅).

3.2.5. $CpFe(CO)_2SC(S)O-4-C_6H_4Me$ (1e)

Yield, 85%; m.p. 138–140 °C. *Anal.* Required for $C_{15}H_{12}FeO_3S_2$: C, 50.01; H, 3.24; S, 17.58. Found: C, 49.79; H, 3.36; S, 17.58%. IR (CH₂Cl₂, cm⁻¹): 2044s, 2000s ν (C=O). ¹H NMR (CDCl₃, δ , ppm): 2.31 (s, 3H, CH₃), 5.05 (s, 5H, C₅H₅), 6.91 (d, 2H, C₆H₄), 7.22 (d, 2H, C₆H₄).

3.3. General procedure for the preparation of $CpFe(CO)(\kappa^2 S, S-SC(S)OAr)$ (2)

A THF solution (30 mL) of CpFe(CO)₂SC(S)OAr (0.25 mmol) was irradiated under a stream of N₂ for 30 min. The products were separated by column chromatography. The column was eluted with (2:1 v:v ratio) CH₂Cl₂/hexane to separate the products which were recrystallized from dichloromethane/hexane.

3.3.1. $CpFe(CO)(\kappa^2 S, S-SC(S)OC_6H_5)$ (2a)

Yield, 80%; m.p. 89–91 °C. *Anal.* Required for $C_{13}H_{10}FeO_2S_2$: C, 49.07; H, 3.17; S, 20.16. Found: C, 48.51; H, 3.11; S, 19.77%. IR (CH₂Cl₂, cm⁻¹): 1954s ν (C=O). ¹H NMR (CDCl₃, δ , ppm): 4.65 (s, 5H, C₅H₅), 7.41 (m, 5H, C₆H₅).

3.3.2. $CpFe(CO)(\kappa^2 S, S-SC(S)O-4-C_6H_4Cl)$ (2b)

Yield, 82%; m.p. 102–103 °C. *Anal.* Required for $C_{13}H_9ClFeO_2S_2$: C, 44.28; H, 2.57; S, 18.19. Found: C, 44.01; H, 2.54; S, 17.50%. IR (CH₂Cl₂, cm⁻¹): 1956s $\nu(C\equivO)$. ¹H NMR (CDCl₃, δ , ppm): 4.66 (s, 5H, C₅H₅), 7.09 (d, 2H, C₆H₄), 7.37 (d, 2H, C₆H₄).

3.3.3. $CpFe(CO)(\kappa^2 S, S-SC(S)O-4-C_6H_4F)$ (2c)

Yield, 68%; m.p. 92–93 °C. *Anal.* Required for C₁₃H₉FFeO₂S₂: C, 46.44; H, 2.70; S, 19.08. Found: C,

46.12; H, 2.29; S, 18.56%. IR (CH₂Cl₂, cm⁻¹): 1956s ν (C=O). ¹H NMR (CDCl₃, δ , ppm): 4.67 (s, 5H, C₅H₅), 7.19 (d, 2H, C₆H₄), 7.40 (d, 2H, C₆H₄).

3.3.4. $CpFe(CO)(\kappa^2 S, S-SC(S)OC_6F_5)$ (2d)

Yield, 87%; m.p. 78–79 °C. *Anal.* Calc. for $C_{13}H_5F_5FeO_2S_2$: C, 38.25; H, 1.23; S, 15.71. Found: C, 37.90; H, 1.11; S, 15.07%. IR (CH₂Cl₂, cm⁻¹): 1961s ν (C=O). ¹H NMR (CDCl₃, δ , ppm): 4.69 (s, H's, C₅H₅).

3.3.5. $CpFe(CO)(\kappa^2 S, S-SC(S)O-4-C_6H_4Me)$ (2e)

Yield, 75%; m.p. 95–96 °C. *Anal.* Required for $C_{14}H_{12}FeO_2S_2$: C, 50.61; H, 3.64; S, 19.30. Found: C, 50.31; H, 3.78; S, 19.50%. IR (CH₂Cl₂, cm⁻¹): 1953s ν (C=O). ¹H NMR (CDCl₃, δ , ppm): 2.33 (s, 3H, CH₃), 4.63 (s, 5H, C₅H₅); 6.97 (d, 2H, C₆H₄), 7.17 (d, 2H, C₆H₄).

3.4. X-ray structure analysis

Crystals suitable for X-ray diffraction of **1b** were obtained from CH₂Cl₂/hexane mixture. The data were collected on a KappaCCD diffractometer with graphite monochromated Mo K α radiation at 175 K. The crystallographic data are shown in Table 2. The cell parameters were determined from 4335 reflections collected in the range $1.82 \le \theta \le 30.05^{\circ}$. There were 4334 independent reflections with 3711 observed reflections (>2 $\sigma(I)$). The structure was solved by direct method using SHELXS-97 [30] and DIFMAP synthesis using SHELXTL-96 [31].

4. Supplementary data

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 277850 for compound **1b**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44 1233 336 033; e-mail: deposit@ ccdc.cam.ac.uk or www:http://www.ccdc.cam.ac.uk).

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