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Synthesis and structures of CpFe(CO)₂(κE -ECS₂Ph) and [CpFe(CO)($\kappa^2 S$, *E*-ECS₂Ph)] (E = S, Se)

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Abstract

The iron trithiocarbonato complex CpFe(CO)₂(κ S-SCS₂Ph) (1a) and its selenodithiocarbonato analogue CpFe(CO)₂(κ Se-SeCS₂Ph) (1b) were generated by room temperature reactions of (μ -E_x)[CpFe(CO)₂]₂ (E = S; x = 2, 3. E = Se; x = 1) with PhSC(S)Cl. These compounds can be converted into the complexes CpFe(CO)(κ^2 S,E-ECS₂Ph) [E = S (2a), Se (2b)], in which the trithiocarbonato or the selenodithiocarbonato ligand is bonded to the iron in a chelate form, under photolytic conditions. The composition and structure of all products have been verified by elemental analyses, IR and ¹H NMR spectroscopies. The crystal structures for compounds 1a, 1b, and 2b show a three-legged piano-stool configuration at Fe in each complex. The spectroscopic and structural data in this work are commensurate with the electronic factor of the S- and Se-donor ligands. © 2007 Elsevier Ltd. All rights reserved.

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1. Introduction

The reactions of the iron chalcogenides $(\mu$ -E_x)-[CpFe(CO)₂]₂ (E = S; x = 2, 3. E = Se; x = 1) with electrophiles have been the subject of multiple investigations by us and others [1–10]. The reactions of these chalcogenides with acid chlorides were reported to give chalcogeno-carboxylato complexes with the general formula CpFe(CO)₂-ECOR (E = S, Se) [1–4]. Starting from oxalyl chloride, the chalcogeno-oxalato dimers [CpFe(CO)₂ECO]₂ have been prepared. On the other hand, the reaction of *O*-alkyl oxalyl chlorides with these chalcogenides gave the expected *O*-alkyl chalcogeno-oxalato complexes CpFe(CO)₂ECO-CO₂R [5]. The chalcogenosulfonato complexes CpFe-(CO)₂ESO₂R [6,7] were obtained from the corresponding chalcogenides and sulfonyl chlorides. It was found that the sulfides (μ -S_x)[CpFe(CO)₂]₂ react exclusively with sulfonyl chlorides bearing strong electron-withdrawing groups such as Cl_3CSO_2Cl , F_3CSO_2Cl and $C_6F_5SO_2Cl$ [6], while the selenide (μ -Se)[CpFe(CO)₂]₂ can react with simple alkyl and aryl sulfonyl chlorides [7].

Recent work in our laboratory has shown that the reactions of these chalcogenides with chloroformates, ROCOCI, produce the iron mono-chalcogenocarbonato complexes CpFe(CO)₂ECO₂R [8,9]. The dithiocarbonato complexes $CpFe(CO)_2(\kappa S-SC(S)OR)$ and the selenothiocarbonato complexes $CpFe(CO)_2(\kappa Se-SeC(S)OR)$ were obtained from the reactions of the chalcogenides with alkyl or aryl chlorothioformates ROC(S)Cl [10,11]. Photolysis of THF solutions of the latter dithiocarbonato or selenothiocarbonato complexes lead to the dissociation of one carbonyl group and the generation of the chelate complexes CpFe- $(CO)(\kappa^2 E, S-EC(S)OR)$ [10,11]. Reactions of the iron dimers $[Cp'Fe(CO)_2]_2$ (Cp' = C₅H₅, C₅Me₅) with $[EtOC(S)S]_2$ in cyclohexane were reported to produce the ethyl-dithiocarcomplexes $Cp'Fe(CO)_2(\kappa S-S_2COEt)$ [12,13]. bonato Photolysis of these complexes gave $Cp'Fe(CO)(\kappa^2 S, S-S_2)$ -COEt), which exhibit bidentate coordination of the ethyl-

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dithiocarbonato ligand [14]. Treatment of the latter complexes with phosphine or phosphite ligands gave the mixed carbonyl-phosphine or phosphite complexes Cp'Fe(CO)-(L)(κ S-S₂COEt) (L = PBu₃, PPh₃, P(OEt)₃, P(OPh)₃) [14].

The trithiocarbonato complexes $CpFe(CO)_2(\kappa S-SCS_2R)$ have been made by substitution of the iodide ligand of $CpFe(CO)_2I$ by the trithiocarbonate anions $RSCS_2^-$ (R = Me, Et, Ph) [15]. The chelated forms, CpFe(CO)-($\kappa^2 S, S-S_2CSR$) were obtained by photolysis of the dicarbonyl analogs [15].

In continuation of our work on the area of organoiron complexes containing thio- and selocarbonate ligands, this paper describes the syntheses and X-ray structures of iron trithiocarbonate and selenodithiocarbonate complexes.

2. Results and discussion

2.1. Synthesis and characterization

The trithio- and the dithioselenocarbonate complexes are obtained in good yields by the reactions of the iron chalcogenide complexes $(\mu$ -E_x)[CpFe(CO)₂]₂ (E = S; x = 2, 3. E = Se; x = 1) with PhSC(S)Cl, as shown in Eq. (1).



E=S(a), Se(b).

The orange complex 1a and the brown complex 1b are air stable as solids and in solution. They are soluble in common polar organic solvents but insoluble in hydrocarbons. The identities of 1 and 2 have been confirmed by IR, ¹H NMR spectroscopy, elemental analysis and crystal structure determination. The ¹H NMR spectra of 1 reveal one singlet in the range of 5.06–5.11 ppm for the Cp-proton resonances. These chemical shifts fall within the typical range reported for analogous thiocarbonato and selenocarbonato complexes of iron [8-11]. The protons of the phenyl group appear as two multiplets in the ratio of 3:2, as expected. The bands at 2050 and 2002 cm^{-1} in the IR spectrum of 1a are due to v(CO) of the two terminal carbonyl groups. For 1b. these bands are found at lower wavenumbers (2040, 1990 cm⁻¹) due to stronger σ -donor and/or weaker π -acceptor properties of the selenodithiocarbonate ligand compared to the trithiocarbonate ligand. A similar shift for simple thiocarboxylato versus selenocarboxylato complexes has been reported [2-5].

Photolyzing THF solutions of complexes 1 in the absence of added ligand produces the chelate complexes $CpFe(CO)(\kappa^2 E, S-ECS_2Ph)$ (2) as shown in Eq. (2). The trithiocarbonate ligand of 2a is bonded to the metal through the two sulfur atoms. In a similar fashion, the dithioseleno-carbonate ligand of 2b is bonded through the selenium and sulfur atoms. These dark red complexes are soluble in most organic solvents, including hydrocarbons.



Complexes 2 have been characterized by IR, ¹H NMR, as well as elemental analysis. The IR spectra of 2 in CH₂Cl₂ show the characteristic vCO absorption in the range of 1947–1954 cm⁻¹. This absorption is similar to those of CpFe(CO)($\kappa^2 S$,*E*-EC(S)OR) (1941–1957 cm⁻¹) [10,11] and are lower than those of the starting complexes 1. The latter difference might be attributed to the weak π -acid character of the chelate trithio- or selenodithio-carbonate ligand compared to that of carbonyl ligand. Complexes 2 exhibit ¹H NMR spectra showing a singlet in the range of 4.65–4.66 ppm for the Cp-protons. This chemical shift range is lower than that for the corresponding Cp resonances in complexes 1 and is similar to the analogous range reported for CpFe(CO)(κ^2 S,E-EC(S)OR) complexes (4.63–4.69 ppm) [10,11].



Fig. 1. An ORTEP representation of the structure of CpFe(CO)₂- $(\kappa S$ -SCS₂Ph) (1a). The thermal ellipsoids are drawn at the 50% probability level.



Fig. 2. An ORTEP representation of the structure of $CpFe(CO)_2(\kappa Se-SeCS_2Ph)$ (1b). The thermal ellipsoids are drawn at the 50% probability level.

Table 1	l
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Selected bond lengths (Å) and bond angles (°) in CpFe(CO)₂(κ S-SCS₂Ph) (1a), CpFe(CO)₂(κ Se-SeCS₂Ph) (1b) and CpFe(CO)(κ ²Se,S-SeCS₂Ph) (2b)

1a		1b		2b	
Fe–C1	2.0871(17)	Fe-C1	2.0928(17)	Fe–C8	2.070(11)
Fe–C2	2.1035(16)	Fe–C2	2.0830(19)	Fe–C9	2.063(12)
Fe-C3	2.1111(16)	Fe–C3	2.0840(18)	Fe–C10	2.095(14)
Fe-C4	2.0960(16)	Fe–C4	2.0985(17)	Fe–C11	2.045(14)
Fe-C5	2.0871(172)	Fe–C5	2.1072(17)	Fe–C12	2.051(14)
Fe–C6	1.7879(16)	Fe–C6	1.7783(18)	Fe–C13	1.732(14)
Fe–C7	1.7831(17)	Fe–C7	1.7834(17)	Fe–Se1	2.3872(18)
Fe-S1	2.2706(4)	Fe–Se1	2.3844(3)	Fe–S1	2.310(2)
S1-C8	1.7232(16)	Se1–C8	1.8908(3)	Se1–C1	1.800(9)
S2–C8	1.6379(16)	S2–C8	1.6293(16)	S1-C1	1.734(9)
S3-C9	1.7675(18)	S1–C8	1.7568(17)	S2-C1	1.736(8)
S3–C8	1.7733(16)	S1-C9	1.7692(14)	S2-C2	1.792(10)
C6O1	1.134(2)	C6O1	1.137(2)	C13–O1	1.129(16)
C7–O2	1.134(2)	C7–O2	1.133(2)		
C6–Fe–C7	94.16(8)	C6–Fe–C7	94.21(8)	C13-Fe-Se1	95.8(4)
C7–Fe–S1	89.86(6)	C7–Fe–Se1	89.26(6)	C13–Fe–S1	92.8(4)
C6–Fe–S1	89.93(5)	C6–Fe–Se1	88.95(6)	Se1–Fe–S	77.63(7)
C8–S1–Fe	110.37(7)	C8–Se1–Fe	106.98(5)	Se1-C1-S1	112.8(5)
C9–S3–C8	103.73(7)	C8-S1-C9	104.03(8)	Se1-C1-S2	121.8(5)
S2-C8-S1	128.91(9)	S2-C8-Se1	128.14(10)	S1-C1-S2	125.4(5)
S2-C8-S3	123.37(10)	S2-C8-S1	124.59(10)	C1-S2-C2	102.2(4)
S1-C8-S3	107.72(8)	S1-C8-Se1	107.26(8)		

2.2. Crystal structures

The structures of complexes **1a** and **1b** are shown in Figs. 1 and 2, respectively. Selected bond distances and angles for these complexes are collected in Table 1. These complexes display a three-legged piano-stool configuration at Fe. The Fe–C(Cp) and Fe–C(CO) bond lengths in **1a** and **1b** are similar to those found in CpFe(CO)₂-containing complexes [5,8–10]. The Fe–S bond distance of 2.2677(4) Å in **1a** is comparable to that found in CpFe(CO)₂SX complexes [X = SO₂CCl₃ (2.2803(13) Å), CO₂Et (2.2675(10) Å), CO-2-C₆H₄NO₂ (2.266(1) Å), C(S)O–C₆H₄Cl (2.2765 (5) Å)] [5,8,16,17]. The Fe–Se bond distance in **1b** of 2.3839(4) Å is also comparable to that found in CpFe(CO)₂SeY complexes [Y = SO₂Ph (2.394(3) Å), CO₂Et (2.3829(8) Å), C(S)O-4-C₆H₄Cl (2.3985(2) Å)] [7,10,18].

The crystal structure of **2b** is presented in Fig. 3. Selected bond distances and angles of **2b** are listed in Table 1. The Fe–Se bond distance in **2b** is 2.3872(18) Å, which is



Fig. 3. An ORTEP representation of the structure of CpFe(CO)($\kappa^2 Se,S$ -SeCS₂Ph) (**2b**). The thermal ellipsoids are drawn at the 30% probability level.

similar to that of **1b**. The Fe–S bond length in **2b** of 2.310(2) Å is longer than that of **1a**. The Fe–CO bond length of **2b** (1.732(14) Å) is shorter than the corresponding lengths observed for **1a** and **1b** in line with higher π -back donation from the Fe-*d* orbitals to the empty π^* -orbital of CO in **2b** compared to those of **1a** and **1b**.

3. Experimental

3.1. General

All manipulations were performed using standard Schlenk and inert-atmosphere techniques under an argon atmosphere. Diethyl ether, hexane, and benzene were dried over sodium and benzophenone, and were freshly-distilled under nitrogen prior to use. Dichloromethane was heated under reflux over P_2O_5 , and was freshly-distilled under nitrogen prior to use. The compounds $(\mu-S_x)[CpFe(CO)_2]_2$ [1] and $(\mu-Se)[CpFe(CO)_2]_2$ [2] were prepared by previously published procedures. The iron dimer $[CpFe(CO)_2]_2$, the PhSC(S)Cl precursor, selenium, and sulfur were purchased from Aldrich and were used as received.

Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker 400 MHz spectrometer. Chemical shifts are given in ppm relative to TMS at 0 ppm. Infrared (IR) spectra were recorded on a Nicolat-Impact 410 FT-IR spectrometer. Elemental analyses of C, H and S were performed in the Institute of Organic Chemistry, Vienna University, Austria. Melting points were reported on an electrothermal melting point apparatus and are uncorrected. The photolytic reactions were carried out using a conventional tungsten lamp (100 W).

3.2. General procedure for the preparation of $CpFe(CO)_2(\kappa E-ECS_2Ph)$ (1)

A solution of PhSC(S)Cl (1.20 mmol) in 10.0 mL of diethyl ether was dropwise added to a solution of the iron chalcogenides $(\mu - E_x)$ [CpFe(CO)₂]₂ (1.00 mmol) in 50.0 mL of diethyl ether. The deep red solution was stirred for 2 h under protection of light. The solution was stripped to dryness and the resulting solid was redissolved in a minimum amount of dichloromethane and introduced to a silica gel column made up in hexane. Elution with hexane removes the unreacted PhSC(S)Cl. A red band of the product was eluted with diethyl ether: hexane solution (1:1 v:v ratio), followed by another red band which was also collected and identified as CpFe(CO)₂Cl. The products CpFe(CO)₂-(KE-ECS₂Ph) were recrystallized from dichloromethane/ hexane.

3.2.1. $CpFe(CO)_2(\kappa S-SCS_2Ph)$ (1a)

Yield: 85%. m.p. = 57–59 °C. IR (CH₂Cl₂, cm⁻¹): 2044(s), 1999(s) ($\nu_{C=O}$). ¹H NMR (CDCl₃): δ 5.11 (s, 5H, C₅H₅); 7.45 (m, 3H, C₆H₅); 7.55 (m, 2H, C₆H₅). Anal. Calc. for C₁₄H₁₀FeO₂S₃: C, 46.42; H, 2.78; S, 26.55. Found: C, 46.31; H, 2.62; S, 26.80%.

3.2.2. $CpFe(CO)_2(\kappa Se-SeCS_2Ph)$ (1b)

Yield: 83%. m.p. = 130–131 °C. IR (CH₂Cl₂, cm⁻¹): 2038(s), 1993(s) ($v_{C=0}$). ¹H NMR (CDCl₃): δ 5.14 (s, 5H, C₅H₅); 7.48 (m, 3H, C₆H₅); 7.56 (m, 2H, C₆H₅). Anal. Calc. for C₁₄H₁₀FeO₂S₂Se: C, 41.10; H, 2.46; S, 15.67. Found: C, 41.00; H, 2.36; S, 15.71%.

3.3. General procedure for the preparation of $CpFe(CO)(\kappa^2 E, S-ECS_2Ph)$ (2)

A THF solution (30 mL) of $CpFe(CO)_2(\kappa E-ECS_2Ph)$ (0.25 mmol) was irradiated under a stream of N₂ for 15 min. The colour of the reaction mixture turned from orange to dark red. The volatile components were removed by vacuum and the resulting solids were purified by column chromatography using silica gel columns. The columns were eluted with (1:5 v:v ratio) of CH₂Cl₂/hexane to separate the products, which were subsequently recrystallized by slow evaporation of hexane solutions.

3.3.1. $CpFe(CO)(\kappa^2 S, S-SCS_2Ph)$ (2a)

Yield: 75%. m.p. = 121-122 °C. IR (CH₂Cl₂, cm⁻¹): 1955(s) $(v_{C=0})$. ¹H NMR (CDCl₃): δ 4.64 (s, 5H, C₅H₅); 7.30 (m, 3H, C₆H₅); 7.51 (m, 2H, C₆H₅). Anal. Calc. for C13H10FeOS3: C, 46.71; H, 3.02; S, 28.78. Found: C, 46.31; H, 3.09; S, 27.80%.

3.3.2. $CpFe(CO)(\kappa^2 S, Se-SeCS_2Ph)$ (2b)

Yield: 78%. m.p. = 98–99 °C. IR (CH₂Cl₂, cm⁻¹): 2049(s) ($v_{C=0}$). ¹H NMR (CDCl₃): δ 4.61 (s, 5 H, C₅H₅); 7.48 (m, 3H, C₆H₅). Anal. Calc. for C₁₃H₁₀FeOS₂Se: C, 40.96; H. 2.64; S. 16.83. Found: C. 41.25; H. 2.62; S. 15.21%.

Table 2

Crystallographic data and refinement details for CpFe(CO)₂(κS-SCS₂Ph) (1a), CpFe(CO)₂(κSe-SeCS₂Ph) (1b), and CpFe(CO)(κ²Se,S-SeCS₂Ph) (2b)

Compound	1a	1b	2b
Empirical formula	$C_{14}H_{10}FeO_2S_3$	C ₁₄ H ₁₀ FeO ₂ S ₂ Se	C ₁₃ H ₁₀ FeOS ₂ Se
Formula weight	362.25	409.15	381.14
Crystal size (mm)	$0.60 \times 0.40 \times 0.40$	$0.40 \times 0.30 \times 0.28$	$0.30 \times 0.14 \times 0.12$
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/c$	$P2_1/c$
Volume $(Å^3)$	1464.56(7)	1488.16(9)	1423.32(11)
Ζ	4	4	4
Unit cell dimensions			
a (Å)	10.3722(3)	10.3802(4)	14.6540(6)
b (Å)	12.0814(4)	12.0808(4)	8.1790(4)
<i>c</i> (Å)	11.7589(3)	11.9470(4)	12.8734(5)
β (°)	96.320(1)	96.626(2)	112.710(2)
Index ranges	$-14 \leqslant h \leqslant 14,$	$-14 \leqslant h \leqslant 14,$	$-17 \leqslant h \leqslant 17$,
	$-17 \leqslant k \leqslant 17,$	$-17 \leqslant k \leqslant 17,$	$-9 \leqslant k \leqslant 9$,
	$-16 \leqslant l \leqslant 16$	$-16 \leqslant l \leqslant 16$	$-15 \leqslant l \leqslant 15$
Data/parameters	4302/181	4348/181	2504/163
<i>F</i> (000)	736	808	752
$D_{\rm calc} ({\rm g/cm}^3)$	1.643	1.826	1.779
$\mu ({\rm mm}^{-1})$	1.453	3.735	3.893
λ (Å)	0.71073	0.71073	0.71073
R_1^{a}	0.0267	0.0218	0.0817
wR_2^{b}	0.0796	0.0551	0.2780
Goodness-of-fit ^c	1.036	1.016	1.057

 $R_1 = \sum ||F_0| - |F_c|| / \sum |F_0|.$

^b $wR_2 = \{\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2] \}^{1/2}$. ^c Goodness-of-fit = $\{\sum [w(F_o^2 - F_c^2)^2] / (n-p)\}^{1/2}$, where *n* is the number of reflections and *p* is the total number of parameters refined.

3.4. X-ray crystal structure analysis

Details of data collections and structure refinement for complexes 1a, 1b and 2b are collected in Table 2. Single crystals suitable for X-ray structure determination were obtained by re-crystallization of 1a and 1b from THF/ hexane mixtures, and from hexane for 2b. X-ray diffraction measurements were performed on X8APEXII CCD diffractometer with graphite monochromated Mo Ka radiation, $\lambda = 0.71073$ Å at 100, 100 and 296 K for **1a**, 1b and 2b, correspondingly. Single crystals were positioned at 40 mm from the detector and 3099, 1936 and 1487 frames were measured, each for 5, 5 and 40 s over 1° scan width. The data were processed using SAINT software while absorption corrections were performed using the program SADABS [16]. The structures were solved by direct methods and refined by full-matrix least-squares techniques. Non-hydrogen atoms were refined with anisotropic displacement parameters. H atoms were placed in geometrically-calculated positions and were refined as riding atoms in subsequent least-squares model refinements. The isotropic thermal parameters were estimated to be 1.2 times the values of the equivalent isotropic thermal parameters of the atoms to which hydrogens were bonded. The following computer programs were used: structure solution, shelxs-97 [17]; refinement, shelxl-97 [18]; molecular diagrams, ORTEP [19]. Scattering factors were taken from the literature [20].

4. Supplementary material

CCDC 632056, 632055 and 632057 contain the supplementary crystallographic data for **1a**, **1b** and **2b**. These data can be obtained free of charge via http://www.ccdc.ac.uk/ conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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