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Synthesis and characterization of cyclopentadienyldicarbonyliron *S*-bonded monothiocarboxylates, $FeCp(CO)_2SCOR$. Crystal structure of $FeCp(CO)_2SCO(2\text{-}NO_2C_6H_4)$

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Abstract

The reactions of organoiron sulfanes, $(\mu\text{-}S_x)[FeCp(CO)_2]_2$ ($x = 3$, 4) with acid chlorides, RCOCl, produce the new organoiron thiocarboxylates, $FeCp(CO)_2SCOR$, where $R = 2\text{-}CH_3C_6H_4$, I; $R = 2\text{-}CH_3COOC_6H_4$, II; $R = 3,5\text{-}(O_2N)_2C_6H_3$, III; $R = 2\text{-}O_2NC_6H_4$, IV and $R = 2\text{-}FC_6H_4$, V. The organoiron thioterephthalate $(CO)_2CpFeSCOC_6H_4COSFeCp(CO)_2$ (VI) has also been prepared. The new complexes were characterized by IR, ^1H NMR and elemental analysis and the crystal structure of complex IV was determined by X-ray diffraction.

Complex IV crystallises in the monoclinic system, space group $P2_1/c$ with a 923.9(4), b 1132.2(4), c 1497.6(7) pm; β 106.87(4)°; d_{calc} 1.64 g cm^{-3}; μ 11.3 cm^{-1}; $Z = 4$; $R_1 = 0.0414$ and $R_2 = 0.0455$. The thiocarboxylate group is bonded to the iron atom through sulfur.

Introduction

During our studies on the reactivity of the organoiron sulfane complexes, $(\mu\text{-}S_x)[FeCp(CO)_2]_2$, ($x = 3$, 4) we observed that these iron sulfanes react with acid chloride, RCOCl in ether at room temperature to give stable dicarbonylcyclopentadienyliron S-bonded monothiocarboxylates, $FeCp(CO)_2SCOR$, in good yield [1]. Complexes of monothiocarboxylic acids, RCOSH are not very common [2–10]. The acid, RCOSH or its salt has usually been used in the preparation of thiocarboxylate

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complexes [2,4,10]. In other reports, the reaction of RCOCl with LnMSH [3] or the sulfide $LnMS^-$ [1,11] was used.

The reaction of the organoiron sulfanes with acid chloride provide a novel and convenient route to new class of dicarbonylcyclopentadienyliron derivatives. We previously described the preparation of $FeCP(CO)_2SCOR$, where R = CH_3, C_6H_5, 2-C_4H_3O (2-furyl), 3-$MeOC_6H_4$, $C(CH_3)_3$, 4-$O_2NC_6H_4$, 1-$C_{10}H_7$(1), and we present here details of the preparation of $FeCp(CO)_2SCOR$ where R = 2-$O_2NC_6H_4$, 2-FC_6H_4, 2-$CH_3COOC_6H_4$; 3,5-$(O_2N)_2C_6H_3$, 2-$CH_3C_6H_4$, and of the dinuclear iron complex $Cp(CO)_2FeSCOC_6H_4COSFe(CO)_2Cp$. The molecular structure of $FeCp(CO)_2SCO(2\text{-}O_2NC_6H_4)$ is also reported.

Experimental

The organoiron sulfanes, $(\mu\text{-}S_x)[FeCp(CO)_2]_2$ ($x = 3, 4$) were prepared as previously described [12]. Acid chlorides were commercial samples (Aldrich) and used as purchased, except for 2-$CH_3COOC_6H_4COCl$ which was prepared from *O*-acetylsalicylic acid [13]. The solvents used were dried as previously described [12]. All reactions were performed under an inert atmosphere. ^1H NMR spectra were recorded on a Bruker WP-80 SY spectrometer. Infrared spectra were recorded on Pye-Unicam SP3-100 spectrophotometer. Elemental analyses were performed by M–H–W Laboratories, Phoenix, Arizona.

Reactions of organoiron sulfanes, $(\mu\text{-}S_x)[FeCp(CO)_2]_2$ ($x = 3$ or 4) with acid chlorides

A solution of the acid chloride (2.5 mmol for a monoacid chloride and 1.2 mmol for $ClCOC_6H_4COCl$ in diethyl ether (10 cm^3) was added slowly at room temperature to a reddish brown solution of 0.9 g (2.0 mmol) of $(\mu\text{-}S_3)[FeCp(CO)_2]_2$ in diethyl ether (10 cm^3). The color changed from reddish-brown to orange within one hour. After 2–4 hours a sample of the mixture was examined by TLC (Silica, CH_2Cl_2), and found to contain an orange-yellow product along with a red product with a lower R_f value. The two products were separated by column chromatography (70–230 mesh silica gel, CH_2Cl_2). The red product was obtained as red crystals (yield ~ 10%, based on RCOCl) from a mixture of CH_2Cl_2/petroleum ether and identified as $FeCp(CO)_2Cl$ [14] (m.p. 87–88 °C). ^1H NMR ($CDCl_3$); singlet at δ 5.05 ppm. IR (CH_2Cl_2) ν(CO) 2050s and 2004s cm^{-1}. Analysis. Found: C, 39.38; H, 2.47; Cl, 16.54. $C_7H_5FeO_2Cl$ calcd.: C, 39.6; H, 2.35; Cl, 16.7%. The yields, melting points, color and analytical data for the reported organoiron-*S*-monothiocarboxylates, $FeCp(CO)_2SCOR$ are shown in Table 1. Their spectral data are listed in Table 2.

X-ray study

Single crystals suitable for X-ray work were obtained by recrystallization of $FeCp(CO)_2SCO(2\text{-}O_2NC_6H_4)$ (IV) from CH_2Cl_2/n-hexane at −30 °C. A single crystal of dimensions ca. 0.65 × 0.35 × 0.31 mm was selected for data collections. Compound IV crystallises in the monoclinic system, space group $P2_1/c$ with a 923.9(4), b 1132.2(4), c 1497.6(7) pm, β 106.87(4)°, d_{calc} 1.64 g cm^{-3}, μ 11.3 cm^{-1}, $Z = 4$, 2θ range 2–48°. Data were collected with an Enraf–Nonius CAD-4 diffractometer with Mo-K_α radiation (λ 0.71069 Å). A total of 2587 reflections were collected and 2227 reflections with $I \geqslant 3\sigma(I)$ were used to solve the structure. No

Table 1

Analytical data, yields and properties of $FeCp(CO)_2SCOR$. Compounds I–VI

R	Yield (%)	M.p. (°C)	Color	Analysis (Found (Calcd) (%))				
				C	H	S	Fe	N
$2\text{-}CH_3C_6H_4$	40–45	102–103	Orange	55.07	3.91	9.73	–	–
(I)				(54.88)	(3.66)	(9.76)		
$2\text{-}CH_3COOC_6H_4$	45–50	84– 85	Light	52.02	3.44	8.76	14.96	–
(II)			brown	(51.64)	(3.22)	(8.60)	(15.05)	
$3,5\text{-}(O_2N)_2C_6H_3$	50–55	~180 with	Reddish	41.62	2.16	7.8	–	–
(III)		decomposition	Orange	(41.58)	(1.98)	(7.92)		
$2\text{-}O_2NC_6H_4$	50–55	85– 86	Orange	47.01	2.76	8.66	15.24	3.96
(IV)				(46.80)	(2.51)	(8.91)	(15.60)	(3.90)
$2\text{-}FC_6H_4$	50–55	114–115	Orange	50.73	2.97	9.55	–	–
(V)				(50.60)	(2.71)	(9.64)		
$Cp(CO)_2FeSCOC_6H_4$	45–50	>170	Orange	47.89	2.71	11.51	19.57	–
(VI)		decomposition		(48.00)	(2.54)	(11.63)	(20.36)	

Table 2

^{1}H NMR and IR spectral data for FeCp(CO)$_2$SCOR. Compounds I–VI

R	^{1}H NMR (CDCl$_3$) [a] (ppm)	IR(CH$_2$Cl$_2$) [b] (cm^{-1})
2-CH$_3$C$_6$H$_4$ (I)	2.37(s, 3H, C*H*$_3$) 5.04(s, 5H, C$_5$*H*$_5$) 7.17(m, 3H, 3-,4,5-Ar*H*) 7.74(m, 1H, 6-Ar*H*)	2027 vs, 1987 vs (ν(C≡O)) 1604 s (ν(C=O)) 1204 m, 1188 m 913 s (ν(C—S)) 845 m
2-CH$_3$COOC$_6$H$_4$ (II)	2.25(s, 3H, C*H*$_3$) 5.00(s, 5H, C$_5$*H*$_5$) 6.95–7.48(m, 3H, 3-,4-, 5-Ar*H*) 7.89–8.00(m, 1H, 6-Ar*H*)	2037 vs, 1984 vs (ν(C≡O)) 1753, (ν(C=O)) 1598 s, (ν(C=O)) 1467 w, 1367 m 1187, 1197 b, s 930 s(ν(C=S)) 910 sh, 842 m
3,5-(O$_2$N)$_2$C$_6$H$_3$ (III)	5.12(s, 5H, C$_5$*H*$_5$) 9.09(t, 1H, 4-Ar*H*) 9.24(d, 2H, 2-,6-Ar*H*)	2060 vs, 1998 vs (ν(C=O)) 1613 s, (ν(C=O)) 1541 s, 1347 s (ν(N=O)) 1108 m, 1000 m 925 m (ν(C=S))
2-O$_2$NC$_6$H$_4$ (IV)	5.13(s, 5H, C$_5$*H*$_5$) 7.3–7.7 (m, 3H, 3-,4- 5-Ar*H*) 7.8–9.0 (m, 1H, 6-Ar*H*)	2040 vs, 1986 vs (ν(C=O)) 1600 s, (ν(C=O)) 1522 s, 1345 s (ν(N=O)) 1195 m 925 s (ν(C=S)) 848 m
2-FC$_6$H$_4$ (V)	5.06(s, 5H, C$_5$*H*$_5$) 6.9–7.5(m, 3H, 3-,4-, 5-Ar*H*) 7.8–8.0(m, 1H, 6-Ar*H*)	2045 vs, 1990 vs (ν(C=O)) 1605 s (ν(C=O)) 1478 m, 1220 w, 1190 m, 1150w, 1100w 925 s(ν(C=S))
Cp(CO)$_2$FeSCOC$_6$H$_4$ (VI)	5.07(s, 5H, C$_5$*H*$_5$) 8.08(s, 4H, 2-,3-,5-, 6-Ar*H*)	2050 vs, 1995 vs (ν(C≡O)) 1595 vs (ν(C=O)) 1380 m, b, 1195 m 1100 w, b 910 s, b (ν(C=S)) 848 m

[a] s, singlet; d, doublet; t, triplet; m, multiplet. [b] vs, very strong; s, strong; m, medium; sh, shoulder; w, weak; b, broad.

absorption correction was applied. The structure was solved by Patterson synthesis, and refined with the SHELX program. All non-hydrogen atoms were refined with anisotropic thermal parameters. Hydrogen atoms were located in final difference synthesis and freely refined with isotropic thermal parameters. The refinement converged at $R_1 = 0.0414$ and $R_2 = 0.0455$. A list of atomic coordinates is given in Table 3. Further details of the structure determination are available from Crystallographic Data Centre, University Chemistry Laboratory, Lensfield Road, Cambridge CB2 1EW, England.

Results and discussion

The reactions of acid chlorides with the organoiron sulfane, Cp(CO)$_2$FeSSSFe(CO)$_2$Cp, in diethyl ether at room temperature produce stable organoiron thiocar-

Table 3

Atomic coordinates for $FeCp(CO)_2SCO(2\text{-}O_2NC_6H_4)$

Atom	x	y	z	U_{eq}
Fe	0.1984(1)	0.0468(1)	0.3431(0)	0.0448(3)
S	0.4064(1)	0.1632(1)	0.3714(1)	0.0501(6)
C(1)	0.3465(5)	0.3086(4)	0.3516(3)	0.046(2)
C(2)	0.4724(5)	0.3988(4)	0.3734(3)	0.045(2)
C(3)	0.4540(5)	0.5058(4)	0.3245(3)	0.050(2)
C(4)	0.5536(6)	0.5989(5)	0.3526(4)	0.061(3)
C(5)	0.6796(6)	0.5841(5)	0.4281(4)	0.069(3)
C(6)	0.7029(6)	0.4786(6)	0.4751(4)	0.069(3)
C(7)	0.6006(5)	0.3868(5)	0.4491(3)	0.057(3)
C(10)	0.1057(6)	0.1411(5)	0.4046(3)	0.058(3)
C(11)	0.1089(5)	0.1118(4)	0.2320(3)	0.052(2)
C(20)	0.2778(4)	−0.1110(3)	0.2991(2)	0.062(3)
C(21)	0.3513(4)	−0.0939(3)	0.3955(2)	0.066(3)
C(22)	0.2387(4)	−0.0896(3)	0.4429(2)	0.072(3)
C(23)	0.0955(4)	−0.1040(3)	0.3757(2)	0.071(3)
C(24)	0.1197(4)	−0.1172(3)	0.2868(2)	0.069(3)
N	0.3283(5)	0.5213(4)	0.2388(3)	0.068(3)
O(1)	0.2161(3)	0.3406(3)	0.3252(3)	0.066(2)
O(2)	0.2509(5)	0.6079(4)	0.2317(4)	0.119(3)
O(3)	0.3152(5)	0.4477(4)	0.1785(3)	0.086(2)
O(10)	0.0444(5)	0.1962(4)	0.4453(3)	0.086(3)
O(11)	0.0476(4)	0.1457(3)	0.1602(2)	0.075(2)
H(4)	0.536(6)	0.666(5)	0.309(3)	0.07(2)
H(5)	0.762(6)	0.648(5)	0.441(4)	0.10(2)
H(6)	0.784(6)	0.467(5)	0.523(4)	0.08(2)
H(7)	0.614(5)	0.319(4)	0.487(3)	0.05(1)
H(10)	0.336(6)	−0.116(4)	0.250(3)	0.08(2)
H(11)	0.467(7)	−0.084(5)	0.442(4)	0.11(2)
H(12)	0.253(6)	−0.082(5)	0.508(4)	0.10(2)
H(13)	0.002(5)	−0.100(4)	0.400(3)	0.08(2)
H(14)	0.044(6)	−0.133(5)	0.231(4)	0.08(2)

boxylates $FeCp(CO)_2SCOR$, in moderate yields, in addition to FpCl, where Fp = $Fe(CO)_2Cp$, in low yield. The two products were separated by column chromatography (silica, CH_2Cl_2).

The reactivity of organoiron sulfanes towards acid chlorides is attributed to the presence of reactive sulfur atom(s) in the bridging S_x group ($x = 3$ or 4). The course of reaction of the organoiron sulfane FpS_3Fp with acid chloride may be as shown in eq. 1 and 2 followed by 3–5 or 6 and 7.

$$FpS_3Fp + RCOCl \rightarrow FpSCOR + FpS_2Cl \tag{1}$$

$$FpS_2Cl \rightarrow FpCl + \tfrac{1}{4}S_8 \tag{2}$$

$$FpS_3Fp + RCOCl \rightarrow FpS_2COR + FpSCl \tag{3}$$

$$FpSCl \rightarrow FpCl + \tfrac{1}{8}S_8 \tag{4}$$

$$FpS_2COR \rightarrow FpSCOR + \tfrac{1}{8}S_8 \tag{5}$$

$$FpS_3Fp + RCOCl \rightarrow FpS_3COR + FpCl \tag{6}$$

$$FpS_3COR \rightarrow FpSCOR + \tfrac{1}{4}S_8 \tag{7}$$

Such reactivity of the bridging, S_x, group towards electrophilic reagents is known for organic sulfides [15,16a,b]. However the reaction of acid chloride with organic sulfides, RS_xR, with $x \geqslant 1$ has not been investigated. On the other hand, acid chlorides, RCOCl, were converted to monothioesters. RCOSR, by reaction with thiols, RSH or the corresponding metal mercaptides, especially thallium [17], tin [18], or copper [19] mercaptide. The trimethyl thioethers also react with acid chlorides to give monothioesters [20].

In this work we have prepared a new series of the organoiron thiocarboxylates, $FeCp(CO)_2SCOR$, where $R = 2\text{-}CH_3C_6H_4, 2\text{-}CH_3COOC_6H_4$; $3,5\text{-}(O_2N)_2C_6H_3$; $2\text{-}O_2NC_6H_4$; $2\text{-}FC_6H_4$. The organoiron thioterephthalate, $(CO)_2CpFeSCOC_6H_4COS\text{-}FeCp(CO)_2$ has also been prepared. The products were purified by column chromatography under nitrogen and recrystallised from CH_2Cl_2/n-hexane. Table 1 shows the analytical data, yields, and some properties of the products. Their infrared spectra and ^1H NMR spectra are shown in Table 2. The infrared spectra of the above new iron thiocarboxylate derivatives show the two characteristic strong terminal metal carbonyl bands in the ranges 2060–2027 and 1998–1984 cm^{-1}, as generally found for the mononuclear cyclopentadienyldicarbonyliron derivatives, $FeCp(CO)_2X$ (X = Cl, Br) [21]. Second the spectra show the C=O and the C—S stretching frequencies for the S-bonded monthiocarboxylate group at ca. 1600 and 920 cm^{-1}, respectively [4,5]. Their ^1H NMR spectra show a singlet due to Cp protons in the range, 5.0–5.13 ppm. The two largest chemical shifts, 5.12 and 5.13 ppm, are for the iron thiocarboxylates, $FeCp(CO)_2SCOR$ with $R = 3,5(O_2N)C_6H_3$ and $2\text{-}O_2NC_6H_4$, respectively, which reflects a relatively low electron density on the Fe atom in these compounds. The spectra also show the characteristic peaks due to the protons in the R group (see Table 2). The iron thiocarboxylate compounds containing R groups with electron-withdrawing substituents like NO_2 or F show the greatest thermal stability, and so other products were isolated in lower yields (see Table 1 in ref. 1 and Table 1 in this paper). This enhanced thermal stability can be attributed to relatively stronger iron–sulfur bonds in the nitro and fluoro compounds.

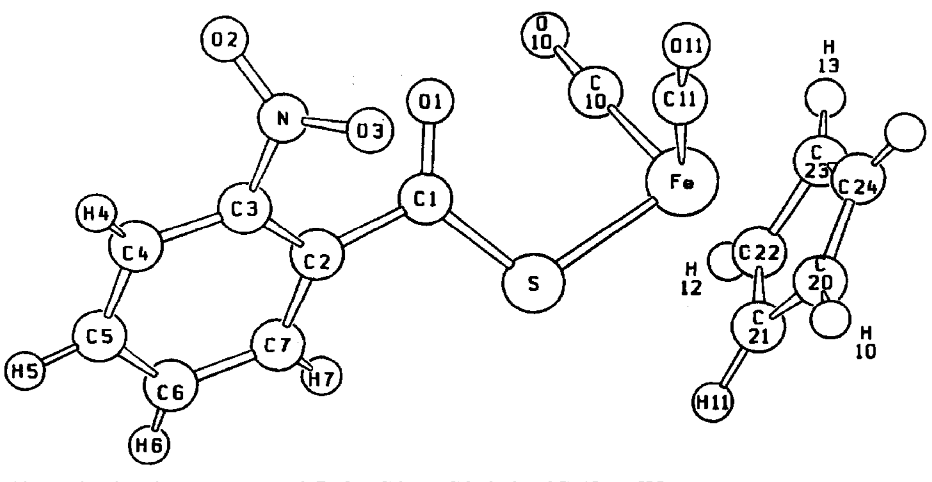

Fig. 1. Molecular structure of $FeCp(CO)_2SCO(2\text{-}O_2NC_6H_4)$ (IV).

Table 4

Selected bond distances (Å) for $FeCp(CO)_2SCO(2\text{-}O_2NC_6H_4)$

Fe–S	2.266(1)	N–O(2)	1.200(6)
Fe–C(10)	1.783(6)	N–O(3)	1.209(6)
Fe–C(11)	1.788(5)	N–C(3)	1.470(6)
Fe–C(20)	2.108(3)	C(2)–C(3)	1.400(6)
Fe–C(21)	2.123(4)	C(2)–C(7)	1.388(6)
Fe–C(22)	2.106(4)	C(3)–C(4)	1.382(6)
Fe–C(23)	2.080(4)	C(4)–C(5)	1.378(7)
Fe–C(24)	2.081(4)	C(5)–C(6)	1.372(2)
C(1)–S	1.734(4)	C(6)–C(7)	1.382(7)
C(1)–O(1)	1.209(5)	C(11)–O(11)	1.127(5)
C(1)–C(2)	1.511(5)	C(10)–O(10)	1.132(6)

Molecular structure of $FeCp(CO)_2SCO(2\text{-}O_2NC_6H_4)$ *(IV)*

The molecular structure of compound IV is shown in Fig. 1. Bond distance and selected bond angles are shown in Table 4 and 5, respectively. It can be seen that the thiocarboxylate ligand is S-bonded to the Fe atom in the $FeCp(CO)_2$ unit, with a *cis* configuration of Fe–S bond relative to C=O bond. The same planar *cis* conformation is found in organic esters such as crystalline methyl acetate [22], and silylmonothioacetate [23]. The structure also shows a *trans* configuration of Fe–Cp bond relative to C–S bond. The NO_2 group lies in a plane approximately perpendicular to the plane of the benzene ring and in the same direction as the thiocarboxylate C=O group. The Fe–S bond distance of 2.266 Å is in the range of single Fe–S bond distance reported for organoiron sulfur complexes [12]. The Fe–S–C(1) angle of 108 seems to be smaller than the reported Ru–S–C angle (114.9–115.8) in the S-bonded thiocarboxylate ruthenium complex $Ru(SCOph)_2(Phen)(PMe_2Ph)_2$ and related complexes [10b], and also smaller than the corresponding angle in organic esters or thioesters [22,23].

Table 5

Selected bond angles (°) for $FeCp(CO)_2SCO(2\text{-}O_2NC_6H_4)$

Fe–S–C(1)	108.0(1)	C(3)–C(2)–C(7)	117.2(4)
C(11)–Fe–C(10)	94.2(2)	C(3)–C(4)–C(5)	119.1(5)
O(1)–C(1)–S	125.3(3)	C(4)–C(5)–C(6)	119.8(5)
O(1)–C(1)–C(2)	120.0(4)	C(5)–C(6)–C(7)	121.2(5)
C(2)–C(1)–S	114.7(3)	C(6)–C(7)–C(2)	120.5(5)
O(3)–N–O(2)	124.7(5)	C(11)–Fe–C(20)	99.6(2)
O(3)–N–C(3)	116.9(4)	C(10)–Fe–C(20)	158.7(2)
O(2)–N–C(3)	118.3(5)	C(11)–Fe–C(22)	154.3(2)
C(11)–Fe–S	94.1(2)	C(10)–Fe–C(22)	94.9(2)
C(10)–Fe–S	93.6(2)	C(11)–Fe–C(21)	137.0(2)
N–C(3)–C(2)	120.3(4)	C(10)–Fe–C(21)	128.8(2)
C(3)–C(2)–C(1)	120.2(4)	C(11)–Fe–C(23)	115.9(2)
C(7)–C(2)–C(1)	122.1(4)	C(10)–Fe–C(23)	92.8(2)
N–C(3)–C(4)	117.5(4)	C(11)–Fe–C(24)	88.7(2)
C(4)–C(3)–C(2)	122.2(4)	C(10)–Fe–C(24)	125.4(2)

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