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Synthesis and characterization of organoiron bifunctional complexes $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COX$ (X = RS, RO, RCOO, R_2N)†

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Abstract—The reactions of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ with thiols (RSH), carboxylic acids (RCOOH), secondary amines (R_2NH) and phenols in the presence of pyridine afforded the bifunctional complexes $(C_5H_4)Fe(CO)_2SCO(C_6H_4)COX$ (X = RS, RCOO, R_2N, ArO) in fairly good yields. The synthesized complexes were characterized by elemental analysis, IR and ^1H NMR spectra. © 1997 Elsevier Science Ltd

Keywords: iron; cyclopentadienyl; carbonyl; sulfur; terephthaloyl chloride; thiocarboxylate.

Metal chalcogen complexes are of special interest due to their important biological and catalytic applications [1–4]. Beside their synthetic and structural aspects, the reactivity of metal chalcogen complexes attract the attention of many chemists [5–12]. We have previously shown that organoiron and organoruthenium sulfides and selenides $[Cp'M(CO)_2]_2(\mu\text{-Ex})$ ($Cp' = C_5H_5$, $Bu^t\text{-}C_5H_4$, $1,3Bu^t_2\text{-}C_5H_3$; M = Fe, Ru; E = S, Se; x = 1–5) react smoothly with monoacid chlorides RCOCl to give the corresponding monothio- or monoselenocarboxylate derivatives $Cp'M(CO)_2ECOR$ (R = alkyl, aryl) [13–16]. Recently we reported a convenient synthesis of organoiron thio- and selenoterephthaloyl chloride complexes $Cp'Fe(CO)_2CO(C_6H_4)COCl$ through the reaction of organoiron sulfides or selenides $[Cp'Fe(CO)_2]_2(\mu\text{-Ex})$ with terephtaloyl chloride ($ClCO(C_6H_4)COCl$) [17].

The presence of an acid chloride group in these organoiron thio- and selenoterephthaloyl chloride complexes subjects them to many reactions, especially with nucleophiles. In accordance with this assumption, we report here the results of the reactions of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ with thiols, carboxylic acids, *N*-methylaniline and 2-chlorophenol.

EXPERIMENTAL

All reactions were conducted under dinitrogen using Schlenk techniques. Thiols, carboxylic acids, *N*-methylaniline and 2-chlorophenol were commercial samples (Aldrich) and used as purchased. $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ was prepared from the reaction of $[(C_5H_5)Fe(CO)_2]_2(\mu\text{-}S_3)$ with terephthaloyl chloride as recently reported [17]. IR spectra were recorded on a Pye–Unicam SP_3-100 spectrophotometer and ^1H NMR spectra on a Bruker WP 80SY spectrometer with Me_4Si as internal standard. Elemental analyses were performed by M-H-W Laboratories, Phoenix, AZ, U.S.A.

Reactions of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ with thiols

Reactions of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ with triphenylmethane thiol. Preparation of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COSC(C_6H_5)_3$ (**1**). To a tetrahydrofuran (THF) solution (50 cm^3) containing triphenylmethane thiol (0.22 g, 0.80 mmol) at 0°C, a solution of Bu^nLi (0.80 mmol) in THF (50 cm^3) was added dropwise.

† Dedicated to Professor G. Huttner on the occasion of his 60th birthday.

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The reaction mixture was left to warm gradually to room temperature and stirring was continued for a further 30 min. A THF (100 cm^3) solution containing $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ (0.30 g, 0.80 cm^3) was then added slowly. After 30 min of stirring at room temperature the color changed from yellow to brown. The solvent was evaporated *in vacuo* at 20°C and the residue was taken in CH_2Cl_2 and transferred to a chromatography column made up in *n*-hexane. A yellow band was eluted with CH_2Cl_2–*n*-hexane (2:1). The solvent was evaporated *in vacuo* and the remaining solid was washed with hexane to give the analytically pure yellow powder of compound **I**. Yield 81%; m.pt (decomposition) 154–156°C. Found: C, 66.0; H, 4.1; S, 10.3. Calc. for $C_{34}H_{24}O_4S_2Fe$: C, 66.2; H, 3.9; S, 10.4%.

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with thiophenol. Preparation of* $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COS(C_6H_5)$ (**II**). In a similar procedure to that described above, a THF (100 cm^3) solution of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ (0.4 g, 1.0 mmol) was added to the reaction mixture of thiophenol (0.10 cm^3, 1.0 mmol) and Bu^nLi (1.0 mmol). Column chromatography afforded an orange band which was eluted with CH_2Cl_2–*n*-hexane (2:1) and from which compound **II** was obtained. Yield 70%; m.pt (decomposition) 147–149°C. Found: C, 55.3; H, 3.3; S, 14.7. Calc. for $C_{21}H_{14}O_4S_2Fe$: C, 56.0; H, 3.1; S, 14.2%.

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with* p-*chloro-thiophenol. Preparation of* $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COS—C_6H_4—Cl$ (**III**). A toluene solution (100 cm^3) containing *p*-chlorothiophenol (0.12, 0.80 mmol), $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ and 5 drops of pyridine was refluxed with stirring for 2 h. The reaction mixture was then cooled to room temperature and filtered. The solvent was evaporated *in vacuo* at 20°C and the residue was taken in CH_2Cl_2 and transferred to a chromatography column made up in *n*-hexane. An orange band was eluted with CH_2Cl_2–*n*-hexane (2:1). The solvent was evaporated and the remaining solid was washed several times with hexane to give the analytically pure orange powder of compound **III**. Yield 77%; m.pt (decomposition) 158–159°C. Found: C, 51.8; H, 2.8; S, 12.8. Calc. for $C_{21}H_{13}O_4S_2ClFe$: C, 52.0; H, 2.7; S, 13.2%.

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with carboxylic acids*

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with nicotinic acid. Preparation of* $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CO—O—CO—C_5H_4N$ (**IV**). A toluene–THF (5:1) solution (100 cm^3) containing nicotinic acid ($3\text{-}COOHC_5H_4N$; 0.10 g, 0.80 mmol), $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ (0.30 g, 0.80 mmol) and 5 drops of pyridine was refluxed with stirring for 3 h. The reaction mixture was then cooled to room temperature and filtered. The solvent was evaporated *in vacuo* and the residue was taken in CH_2Cl_2 and transferred to a chromatography column made up in *n*-hexane. A yellow band was eluted with CH_2Cl_2–ether (9:1). The solvent was evaporated and the residue was dissolved in benzene and filtered. Benzene was then evaporated *in vacuo* and the residue was finally washed several times with hexane to give the analytically pure orange powder of compound **IV**. Yield 86%; m.pt (decomposition) 140–142°C. Found: C, 53.7; H, 2.9; N, 3.1. Calc. for $C_{21}H_{13}O_6NSFe$: C, 54.4; H, 2.8; N, 3.0%.

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with thiophene* 3-*carboxylic acid. Preparation of* $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CO—O—CO—C_4H_3S$ (**V**). In a similar procedure to that described above, a toluene–THF (5:1) solution (100 cm^3) containing thiophene 3-carboxylic acid ($3\text{-}COOHC_4H_3S$; 0.10 g, 0.80 mmol), $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ (0.30 g, 0.80 mmol) and 5 drops of pyridine was refluxed with stirring for 90 min. Column chromatography afforded a yellow band which was eluted with CH_2Cl_4–ether (9:1) and from which compound **V** was obtained. Yield 80%; m.pt (decomposition) 118–120°C. Found: C, 52.0; H, 2.7; S, 12.9. Calc. for $C_{20}H_{12}O_6S_2Fe$: C, 51.3; H, 2.6; S, 13.7%.

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with pyrrole* 2-*carboxylic acid. Preparation of* $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CO—O—CO—C_4H_4N$ (**VI**). In a similar procedure to that described for $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CO—O—CO—C_5H_4N$, a toluene–THF (5:1) solution (100 cm^3) containing pyrrole-2-carboxylic acid ($2\text{-}COOHC_4H_4N$; 0.09 g, 0.80 mmol), $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ (0.30 g, 0.80 mmol) and 5 drops of pyridine was refluxed for 3 h. Column chromatography afforded an orange band which was eluted with CH_2Cl_2–ether (9:1) and from which compound **VI** was obtained. Yield 83%; m.pt (decomposition) 128–130°C. Found: C, 52.7; H, 2.8; N, 3.3; S, 6.8. Calc. for $C_{20}H_{13}O_6NSFe$: C, 53.2; H, 2.9; N, 3.1; S, 7.1%.

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with* p-*bromo-cinnamic acid. Preparation of* $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CO—O—CO—CH—CHC_6H_4Br$ (**VII**). In a similar procedure to that described for **IV**, a toluene–THF (5:1) solution (100 cm^3) containing *p*-bromocinnamic acid (0.18 g, 0.80 mmol), $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ (0.30 g, 0.80 mmol) and 5 drops of pyridine was refluxed for 3 h. Column chromatography afforded an orange–red band which was eluted with CH_2Cl_2–ether (9:1) and from which compound **VII** was obtained. Yield 78%; m.pt (decomposition) 153–155°C. Found: C, 50.4; H, 2.8; S, 5.8. Calc. for $C_{24}H_{15}O_6SBrFe$: C, 50.8; H, 2.7; S, 5.6%.

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with* N-*methylaniline. Preparation of* $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CON(CH_3)C_6H_5$ (**VIII**).

A benzene solution (80 cm^3) containing *N*-methylaniline (0.09 g, 0.8 mmol), $(C_5H_5)Fe(CO)_2$

$SCO(C_6H_4)COCl$ (0.30 g, 0.8 mmol) and 5 drops of pyridine was refluxed for 2 h. The reaction mixture was cooled to room temperature and filtered. The solvent was evaporated *in vacuo* at 20°C and the residue was transferred to a chromatography column made up in *n*-hexane. An orange–red band was eluted with CH_2Cl_2–ether (9:1). Evaporation of the solvent *in vacuo* and washing the remaining solid with hexane afforded the analytically pure orange powder of compound **VIII**. Yield 85%; m.pt (decomposition) 134–136°C. Found: C, 59.1; H, 4.0; N, 3.2; S, 7.4. Calc. for $C_{22}H_{17}O_4NSFe$: C, 59.1; H, 3.8; N, 3.1; S, 7.2%.

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with 2-chlorophenol. Preparation of* $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CO_2(C_6H_4Cl)$ **(IX)**.

In a similar procedure to that described above, a benzene solution (80 cm^3) containing 2-chlorophenol (0.10 g, 0.8 mmol), $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ (0.3 g, 0.8 mmol) and 5 drops of pyridine was refluxed for 2 h. Column chromatography afforded an orange band which was eluted with CH_2Cl_2–ether (9:1) and from which compound **IX** was obtained. Yield 85%; m.pt (decomposition) 81–83°C. Found: C, 53.3; H, 2.7; S, 6.5. Calc. for $C_{21}H_{13}O_5SClFe$: C, 53.8; H, 2.8; S, 6.8%.

DISCUSSION

The organoiron thioterephthaloyl chloride complex $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ is an interesting organometallic compound that possesses a reactive center, namely the acid chloride group which can be subjected to many reactions. Thus, the reaction with organic nucleophiles such as carboxylic acids, amines, alcohols, thiols, etc., facilitates the synthesis of a large variety of interesting bifunctional complexes. In this work, we have conducted some representative reactions using different nucleophiles (Scheme I).

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with thiols*

The reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ with triphenylmethane thiol $[(C_6H_5)_3C—SH]$, thiophenol $(C_6H_5—SH)$ and *p*-chlorothiophenol (*p*-$ClC_6H_4—SH$) afforded, respectively, the unsymmetrical bithioterephthalate complexes $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COSC(C_6H_5)_3$ **(I)**, $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COSC_6H_5$ **(II)** and $(C_5H_5)Fe(CO)_2SCO(C_6H_4Cl)$ **(III)** in very good yields. Two different routes were utilized in the preparation of these complexes.

In the first method, Bu^nLi was reacted first with the organic thiol (RSH) in stoichiometric ratios at 0°C giving presumably the organic sulfide RS^- which was then treated *in situ* with the terephthaloyl chloride complex as presented in the following equations:

$$RSH + Bu^nLi \longrightarrow RS^-Li^+ + C_4H_{10}$$

$$RS^-Li^+ + (C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl \longrightarrow (C_5H_5)Fe(CO)_2SCO(C_6H_4)COSR + LiCl$$

In the second method, the terephthaloyl chloride complex $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ was reacted with organic thiols RSH in refluxing toluene in the presence of pyridine as shown in the following equation:

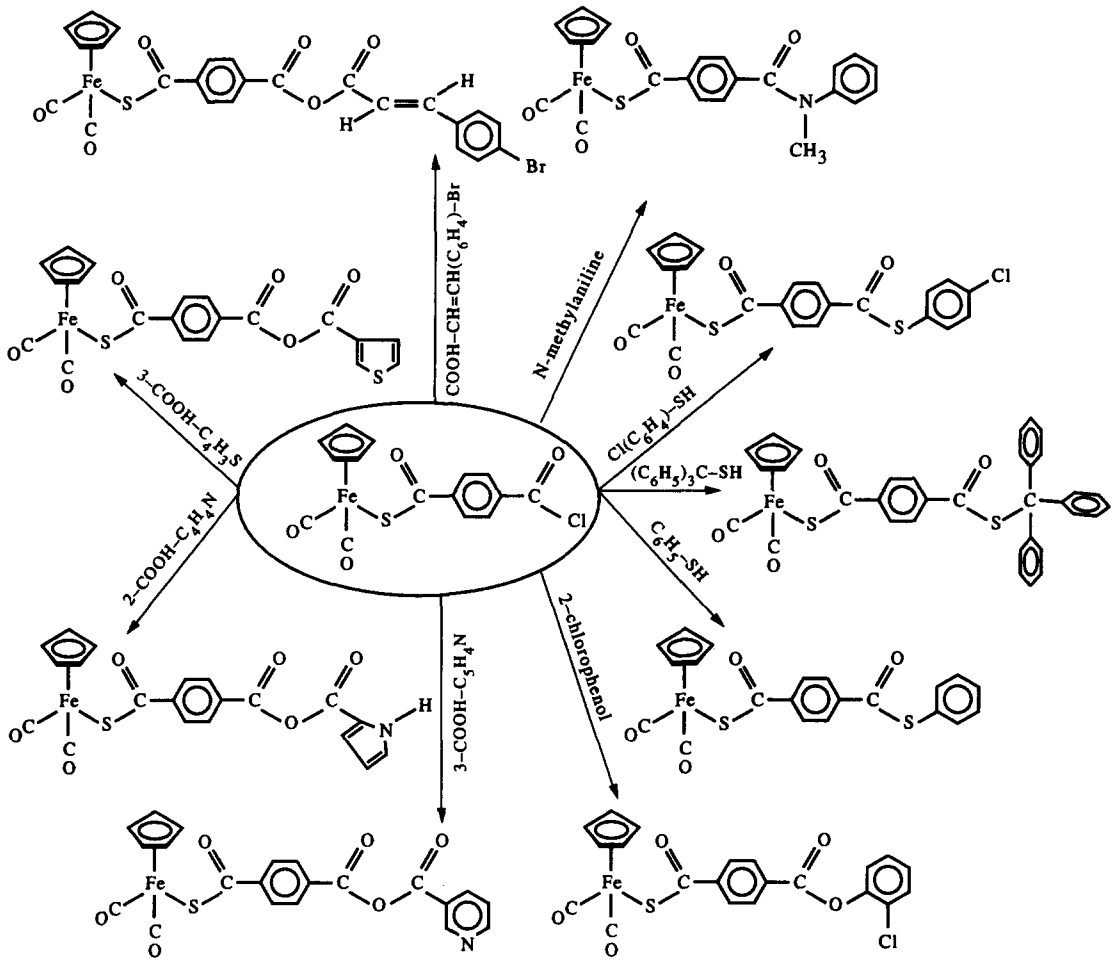
$$(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl + RSH \xrightarrow{\text{Pyridine}} (C_5H_5)Fe(CO)_2SCO(C_6H_4)COSR + HCl$$

Both methods when utilized in the reaction with triphenylmethane thiol gave almost the same yield. However, the first method is neater and gives products that can be easily purified, but it cannot be used when interfering substituents, that can be attacked by Bu^nLi, are present in the organic thiols. Moreover, the reactions with thiols can be easily followed by IR spectroscopy. The disappearance of the band at 1750 cm^{-1} corresponding to the (ClC=O) group of the starting material and the appearance of a new band in the range 1645–1650 cm^{-1} is a clear indication of the completion of the reaction.

The bithioterephthalate complexes (**I–III**) were characterized by elemental analysis, IR and ^1H NMR spectra. Their IR spectra (Table 1) show two bands in the ranges 2020–2030 and 1970–1980 cm^{-1}, corresponding to the two terminal carbonyl groups. The strong band in the range 1645–1650 cm^{-1} is assigned for ν(C=O) of the organo thiocarboxylate part (RS—C=O) and the other band in the range 1580–1590 cm^{-1} is assigned for the ν(C=O) of the organoiron thiocarboxylate part (Fe—S—C=O). The assignment of the latter band was made on the basis of the reported results of the organoiron thiocarboxylate derivatives [18]. Their ^1H NMR spectra (Table 1) show the characteristic protons in their expected chemical shift regions.

Reactions of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with carboxylic acids*

The organoiron terephthaloyl chloride complex $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ reacts readily with carboxylic acids in the presence of pyridine to afford the bifunctional complex, namely, the thioanhydride terephthalate complexes $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CO—O—COR$ in fairly good yields. Four sample reactions have been conducted in this work, using three different heterocyclic carboxylic acids (nicotine acid, thiophene 3-carboxylic acid and pyrrole 2-carboxylic acid) in addition to *p*-bromocinnamic acid. These reactions afforded the expected thioanhydride



Scheme I.

terephthalate complexes $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CO—O—COR$ [R = C_5H_4 (**IV**), C_4H_3S (**V**), C_4H_4N (**VI**) and $CH{=}CHC_6H_4{-}Br$ (**VII**)] in good yields. The complexes **IV–VII** were characterized by elemental analysis, IR and ^1H NMR spectra (Table 1). Their IR spectra show two bands in the ranges 2020–2030 and 1970–1980 cm^{-1} due to the two terminal carbonyl groups and two strong bands in the ranges 1650–1670 and 1580–1590 cm^{-1} assigned for $\nu(C{=}O)$ of the anhydride and the thiocarboxylate moieties, respectively. Although the IR spectra of organic anhydrides usually show two bands for stretching vibrations in the ranges 1830–1800 and 1775–1740 cm^{-1} for the two (C=O) groups [19], only one band in the range 1650–1670 cm^{-1} has been observed for the anhydride (C=O) moiety in the four complexes prepared in this work. This result might be attributed to possible conjugation on both sides of (C=O) groups that have unsaturated rings. The ^1H NMR spectra of complexes **IV–VII** show the characteristic protons in their expected chemical shift regions.

Reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ *with phenols and secondary amines*

The reaction of $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COCl$ with *N*-methylaniline (secondary amine) and 2-chlorophenol in the presence of pyridine afforded the expected bifunctional complexes, namely the thioamide $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CON(CH_3)C_6H_5$ (**VIII**) and the thioester $(C_5H_5)Fe(CO)_2SCO(C_6H_4)CO_2C_6H_4Cl$ (**IX**) complexes, respectively, in almost quantitative yields. These complexes were characterized by elemental analysis, IR and ^1H NMR spectra. Besides the bands corresponding to the terminal carbonyl groups and the thio carboxylate (SC=O) group, which are in full agreement with the previously mentioned complexes, $\nu(C{=}O)$ of the amide appears at 1610 cm^{-1} and that of the ester at 1680 cm^{-1}. Their ^1H NMR spectra show the characteristic protons in their chemical shift regions.

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Table 1. IR and ^1H NMR spectra of the bifunctional complexes $(C_5H_5)Fe(CO)_2SCO(C_6H_4)COX$ (I–IX)

	Complex X	IR (KBr) (cm^{-1})	^1H NMR (CD_2Cl_2) [δ (ppm)]
I	$SC(C_6H_5)_3$	2020(s), 1970(vs) ν(CO); 1645(s), 1580(s) ν(C=O); 890(s) ν(C—S)	4.98 (s, 5H, C_5H_5) 7.21 [s, 15H, $C(C_5H_5)_3$] 7.90 (m, 4H, Ar—H)
II	$S(C_6H_5)$	2030(s), 1980(vs) ν(CO); 1650(s), 1585(s) ν(C=O); 890(s) ν(C—S)	5.00 (s, 5H, C_5H_5) 7.41 (s, 5H, C_6H_5) 8.00 (m, 4H, Ar—H)
III	S—C_6H_4—Cl	2020(s), 1970(vs) ν(CO); 1650(s), 1580(s) ν(C=O); 905(s) ν(C—S)	5.01 (s, 5H, C_5H_5) 7.21 (s, 4H, C_6H_4Cl) 8.00 (m, 4H, Ar—H)
IV	OCO—C_5H_4N	2020(s), 1975(vs) ν(CO); 1670(s), 1580(s) ν(C=O); 1260(s) ν(C—O); 920(s) ν(C—S)	5.09 (s, 5H, C_5H_5) 8.15 (m, 8H, Ar—H)
V	OCO—C_4H_3S	2020(s), 1970(vs) ν(CO); 1760(s), 1585(s) ν(C=O); 1275(s) ν(C—O)	5.09 (s, 5H, C_5H_5) 7.50–8.14 (m, 7H, Ar—H)
VI	OCO—C_4H_4N	2020(s), 1970(vs) ν(CO); 1650(s), 1585(s) ν(C=O); 1275(s) ν(C—O) 920(s) ν(C—S)	5.08 (s, 5H, C_5H_5) 7.06 (m, 3H, C_4H_3N) 8.13 (s, 4H, Ar—H) 9.53 (s, 1H, NH)
VII	OCO—CH=CHC_6H_4Br	2020(s), 1970(vs) ν(CO); 1670(s), 1580(s) ν(C=O); 920(s) ν(C—S)	5.09 (s, 5H, C_5H_5) 7.50 (m, 2H, CH=CH) 8.12 (s, 8H,m Ar—H)
VIII	$N(CH_3)(C_6H_5)$	2020(s), 1970(vs) ν(CO); 1610(s), 1580(s) ν(C=O); 920(s) ν(C—S)	3.45 (s, 3H, CH_3) 5.03 (s, 5H, C_5H_5) 7.10–7.90 (m, 9H, Ar—H)
IX	OCO—C_6H_4—Cl	2020(s), 1970(vs ν(CO); 1680(s), 1580(s) ν(C=O); 920(s) ν(C—S)	5.00 (s, 5H, C_5H_5) 7.35 (m, 4H, C_6H_4Cl) 8.15 (m, 4H, Ar—H)

S: strong, vs: very strong, Ar—H: SCO—C_6H_4—COX.

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