

## OXIDATIVE ADDITION REACTIONS TO $(C_5H_4CH_3)Co(CO)_2$ AND PREPARATION OF THE MONO- $\mu$ -HYDRIDO COMPLEXES $[(C_5H_4CH_3)CoX]_2(\mu-H)(\mu-X)$ (X = Cl, Br, I)

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### Summary

Oxidative additions of the pseudohalogens  $(SCN)_2$ ,  $S(CN)_2$  and the halogens  $Cl_2$ ,  $Br_2$  and  $I_2$  to  $(C_5H_4CH_3)Co(CO)_2$  have been carried out in ether. The mono- $\mu$ -hydrido complexes  $[(C_5H_4CH_3)CoX]_2(\mu-H)(\mu-X)$  (X = Cl, Br, I) have also been made. All the compounds have been characterized by elemental analysis, IR, electronic and  $^1H$  NMR spectroscopy.

### Introduction

We previously reported the oxidative addition of a variety of pseudohalogens to  $(C_5Me_5)Co(CO)_2$ ; in all cases pseudohalogen derivatives of  $Co^{III}$  were obtained in high yield [1-3]. The complex  $(C_5H_4CH_3)Co(CO)_2$  reacts with neutral ligands such as  $PPh_3$  and  $PPhMe_2$  to give  $(C_5H_4CH_3)Co(CO)L$  (L =  $PPh_3$ ,  $PPhMe_2$ ) complexes [4,5] but, its oxidative addition reactions have not been studied. We describe below a study of its reactions with the halogens  $Cl_2$ ,  $Br_2$ , and  $I_2$  and the pseudohalogens  $(SCN)_2$  and  $S(CN)_2$ .

The mono- $\mu$ -hydrido-complexes  $[(C_5H_4CH_3)CoX]_2(\mu-H)(\mu-X)$  (X = Cl, Br, I), analogous to those previously synthesized from the dicarbonyl pentamethylcyclopentadienyl cobalt [6], have also been made so that they can be examined as possible catalysts for hydrogenation of olefins.

### Discussion of results

Addition of a solution of  $(SCN)_2$  or  $S(CN)_2$  in ether to solutions of  $(C_5H_4CH_3)Co(CO)_2$  in the same solvent under  $N_2$  results in loss of CO and precipitation of solids of the stoichiometry  $(C_5H_4CH_3)Co(CO)(NCS)_2$  and  $(C_5H_4CH_3)Co(CO)(CN)(NCS)$ .

Similar reactions with  $Br_2$  and  $I_2$  gave solids of composition  $(C_5H_4CH_3)Co(CO)X_2$  (X = Br, I), but reaction with chlorine resulted in loss of both

TABLE 1  
IR SPECTRAL DATA FOR THE COMPLEXES <sup>a</sup>

$\text{Cp}'\text{Co}(\text{CO})(\text{NCS})_2$	$\text{Cp}'\text{Co}(\text{CO})(\text{CN})(\text{NCS})$	Assignments
2144vs (2.2)	2152vs (2.0)	$\nu(\text{CN})$ of (CN) and (NCS)
2110sh	2112vs	
2056vs	2065vs	$\nu(\text{CO})$
	490m	$\nu(\text{Co}-\text{CO})$
472m	464m,br.	$\nu(\text{Co}-\text{CN})$
456m	440sh	$\delta(\text{Co}-\text{CO})$
395w	395w	$\delta(\text{Co}-\text{CN})$
		$\delta(\text{NCS})$
315w	315m	$\nu(\text{Co}-\text{ring})$
280m	278w	$\nu(\text{Co}-\text{NCS})$

<sup>a</sup> In  $\text{cm}^{-1}$ ;  $\text{Cp}' = \text{C}_5\text{H}_4\text{CH}_3$ . The internal standard ratio determined by Bailey method is given in parentheses [10].

carbonyl groups, as was evidenced from the IR spectrum, and the dimeric compounds  $[(\text{C}_5\text{H}_4\text{CH}_3)\text{CoCl}(\mu\text{-Cl})_2]$  was obtained. The analogous bromo and iodo complexes,  $[(\text{C}_5\text{H}_4\text{CH}_3)\text{CoX}(\mu\text{-X})_2]$  ( $\text{X} = \text{Br}, \text{I}$ ) were obtained by refluxing  $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})\text{X}_2$  ( $\text{X} = \text{Br}, \text{I}$ ) respectively, in light petroleum (b.p. 100–120°C).

Following a standard procedure for preparation of transition metal hydrides [7], the mono- $\mu$ -hydrido complexes  $[(\text{C}_5\text{H}_4\text{CH}_3)\text{CoX}]_2(\mu\text{-H})(\mu\text{-X})$  ( $\text{X} = \text{Cl}, \text{Br}, \text{I}$ ) were obtained by treatment of  $[(\text{C}_5\text{H}_4\text{CH}_3)\text{CoX}(\mu\text{-X})_2]$  with KOH in isopropyl alcohol.

In all cases the IR spectrum displays, in the 4000–200  $\text{cm}^{-1}$  range, the characteristic bands of the methylcyclopentadiene ligand with  $C_5$  symmetry [8,9]. For the complexes  $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})(\text{NCS})_2$  and  $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})(\text{CN})(\text{NCS})$  (Table 1), the bands corresponding to terminal  $\nu(\text{CN})$  and the  $\nu(\text{CN})$  of NCS appears in the range 2152–2110  $\text{cm}^{-1}$ ; determination of the internal standard ratio of  $\nu(\text{CN})$  of NCS bands by the Bailey method [10] indicates that in both complexes the NCS ligand is coordinated via the N atom. The range of stretching vibrations  $\nu(\text{CO})$  is typical of terminal ligands [11]. The  $\nu(\text{C}-\text{S})$  band of the isothiocyanate ligands should appear at ca. 800  $\text{cm}^{-1}$  [12], but it is masked by the strong band  $\gamma(\text{CH})$  from the methylcyclopentadiene ligand. In the range 490–395  $\text{cm}^{-1}$  there appear bands due to Co–CO and Co–CN stretchings, and also the  $\delta(\text{NCS})$  band. A band at 280  $\text{cm}^{-1}$  can be assigned to  $\nu(\text{Co}-\text{NCS})$ .

The more characteristic frequencies for the complexes  $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})\text{X}_2$  ( $\text{X} = \text{Br}, \text{I}$ ),  $[(\text{C}_5\text{H}_4\text{CH}_3)\text{CoX}(\mu\text{-X})_2]$  and  $[(\text{C}_5\text{H}_4\text{CH}_3)\text{CoX}]_2(\mu\text{-H})(\mu\text{-X})$  ( $\text{X} = \text{Cl}, \text{Br}, \text{I}$ ), are listed in Table 2. The range of frequencies of the  $\nu(\text{CO})$  bands is characteristic of terminal ligands and the vibration frequencies  $\nu(\text{Co}-\text{CO})$  and  $\delta(\text{Co}-\text{CO})$  are observed at 495–460  $\text{cm}^{-1}$ , in agreement with literature data [11].

The spectrum of the di- $\mu$ -halo-bis-halo(methylcyclopentadienyl)cobalt complexes  $[(\text{C}_5\text{H}_4\text{CH}_3)\text{CoX}(\mu\text{-X})_2]$  ( $\text{X} = \text{Cl}, \text{Br}$ ) show bands at 302 and 353  $\text{cm}^{-1}$  ( $\text{X} = \text{Cl}$ ) and 240 and 270  $\text{cm}^{-1}$  ( $\text{X} = \text{Br}$ ), which can be ascribed to bridging (b) and terminal (t) Co–X bonds, respectively. The ratios  $\nu(\text{Co}-\text{Cl})_b/\nu(\text{Co}-\text{Cl})_t = 0.85$ ,  $\nu(\text{Co}-\text{Br})_b/\nu(\text{Co}-\text{Br})_t = 0.88$  and  $\nu(\text{Co}-\text{Br})_t/\nu(\text{Co}-\text{Cl})_t = 0.76$  are in accord with literature data [13].

TABLE 2  
IR SPECTRAL DATA FOR THE COMPLEXES <sup>a</sup>

Complex	$\nu(\text{CO})$	$\nu(\text{Co}-\text{CO})$ $\delta(\text{Co}-\text{CO})$	$\nu(\text{Co}-\text{X})$	$\nu(\text{Co}-\text{H}-\text{Co})$
$\text{Cp}'\text{Co}(\text{CO})\text{Br}_2$	2060s	485s 460s	250w	
$\text{Cp}'\text{Co}(\text{CO})\text{I}_2$	2050vs	495s 470vs		
$[\text{Cp}'\text{CoCl}_2]_2$			353m (t) 302m (b)	
$[\text{Cp}'\text{CoBr}_2]_2$			270m (t) 240w (b)	
$(\text{Cp}'\text{Co})_2\text{HCl}_3$			352w (t) 314m (b)	1140vw
$(\text{Cp}'\text{Co})_2\text{HBr}_3$			265m (t) 240w (b)	1115w
$(\text{Cp}'\text{Co})_2\text{HI}_3$				1130vw

<sup>a</sup> In  $\text{cm}^{-1}$ ;  $\text{Cp}' = \text{C}_5\text{H}_4\text{CH}_3$ ; t = terminal; b = bridge.

The mono- $\mu$ -hydrido-complexes  $[(\text{C}_5\text{H}_4\text{CH}_3)\text{CoX}]_2(\mu\text{-H})(\mu\text{-X})$  ( $\text{X} = \text{Cl}, \text{Br}, \text{I}$ ) show bands at 314 and 352  $\text{cm}^{-1}$  ( $\text{X} = \text{Cl}$ ) and 240 and 265  $\text{cm}^{-1}$  ( $\text{X} = \text{Br}$ ), which are assigned to bridging and terminal Co-X bonds, respectively, and the frequency ratios are similar to those indicated above. No  $\nu(\text{Co}-\text{H})$  bands are observed in the 2200–1600  $\text{cm}^{-1}$  range, which is characteristic of terminal Co-H bonds, but a very weak band appear at 1140–1115  $\text{cm}^{-1}$  and can be assigned to the  $\nu(\text{Co}-\text{H}-\text{Co})$  vibration in the light of literature data [14,15]. The presence of the (Co-H-Co) bond in these hydrido complexes was verified by their reaction with  $\text{ICH}_3$  [16].

Electronic spectral data for the complexes in acetone are shown in Table 3. These spectra display poorly resolved broad bands in the range 13440–18726  $\text{cm}^{-1}$  due to  $d-d$  transitions, as a consequence of the low symmetry resulting from the simulta-

TABLE 3  
ELECTRONIC SPECTRA OF THE COMPLEXES <sup>a</sup>

Complex	$\tilde{\nu}_{\text{max.}} (\text{cm}^{-1})$
$\text{Cp}'\text{Co}(\text{CO})(\text{NCS})_2$	15384, 15872, 17543, 29760.
$\text{Cp}'\text{Co}(\text{CO})(\text{CN})(\text{NCS})$	15576, 16025, 17543, 29760.
$\text{Cp}'\text{Co}(\text{CO})\text{Br}_2$	13815, 14285, 14792, 18867, 22522, 29760.
$\text{Cp}'\text{Co}(\text{CO})\text{I}_2$	13440, 14204, 15060, 18726, 26596, 27472, 29760.
$[\text{Cp}'\text{CoCl}_2]_2$	14925, 15723, 16233, 18518, 30120.
$[\text{Cp}'\text{CoBr}_2]_2$	14792, 15625, 16129, 16949, 24390, 30120.
$[\text{Cp}'\text{CoI}_2]_2$	14705, 15384, 16949, 26596, 27027.
$(\text{Cp}'\text{Co})_2\text{HCl}_3$	14792, 15873, 16891, 29940.
$(\text{Cp}'\text{Co})_2\text{HBr}_3$	14204, 15625, 16129, 27027, 27472.
$(\text{Cp}'\text{Co})_2\text{HI}_3$	14705, 27027, 27173.

<sup>a</sup>  $\text{Cp}' = \text{C}_5\text{H}_4\text{CH}_3$ . Acetone solution.

neous presence of different ligands in the molecule, and a very intense peak in the vicinity of  $29000\text{ cm}^{-1}$  due to charge transfer transitions from the metal to the  $\pi^*$  orbital of CO, CN, or NCS, or even to the  $\pi^*$  orbitals of methylcyclopentadiene ligand. The spectra of the complexes  $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})(\text{NCS})\text{X}$  ( $\text{X} = \text{NCS}, \text{CN}$ ) and  $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})\text{X}_2$  ( $\text{X} = \text{Br}, \text{I}$ ) are in accord with the position of the ligands in the spectrochemical series:  $\text{I}^- < \text{Br}^- < \text{NCS}^- < \text{CN}^-$ .

For most of the complexes reported, the  $^1\text{H}$  NMR spectra could not be obtained because they are insoluble in  $\text{CDCl}_3$  and decompose in solvents of higher polarity (e.g. DMSO). The spectra of the complexes  $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})\text{X}_2$  ( $\text{X} = \text{Br}, \text{I}$ ) show singlets at  $\delta$  1.59 ( $\text{X} = \text{Br}$ ) and  $\delta$  1.58 ppm ( $\text{X} = \text{I}$ ), arising from the three equivalent methyl hydrogens, and two multiplets, at  $\delta$  4.04 (2H, BB'), 4.95 (2H, AA') ( $\text{X} = \text{Br}$ ) and  $\delta$  4.05 (2H, BB'), 4.95 ppm (2H, AA') ( $\text{X} = \text{I}$ ), arising from the four ring hydrogens.

The dimeric complex  $[(\text{C}_5\text{H}_4\text{CH}_3)\text{CoBr}(\mu\text{Br})]_2$  shows a singlet at  $\delta$  1.60 (2 $\text{CH}_3$ ) and a multiplet at  $\delta$  3.35–3.70 ppm (8H), and the spectrum of the  $\mu$ -hydrido complex  $[(\text{C}_5\text{H}_4\text{CH}_3)\text{CoCl}]_2(\mu\text{-H})(\mu\text{-Cl})$  shows a singlet at  $\delta$  1.58 (2 $\text{CH}_3$ ) and a multiplet at  $\delta$  3.90–4.20 ppm (8H). This assignment is in accord with that for the methylcyclopentadienyl metal derivatives [4,17,18].

## Experimental

All the reactions were carried out under oxygen-free  $\text{N}_2$ . The  $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})_2$  was prepared by the procedure described for  $(\text{C}_5\text{H}_5)\text{Co}(\text{CO})_2$  [19]. The pseudohalogens  $(\text{SCN})_2$  [20] and  $\text{S}(\text{CN})_2$  [21] were prepared by published procedures.

The cobalt was determined by titration of the Co-EDTA complex in the presence of NET as indicator. The microanalyses were performed by the Dpto. Química Inorgánica de la Facultad de Ciencias de la Universidad de Alcalá de Henares (Madrid, Spain).

The IR spectra were recorded in the range  $4000\text{--}200\text{ cm}^{-1}$  on a Nicolet 5DX spectrometer using Nujol and hexachlorobutadiene mulls between CsI windows.  $^1\text{H}$  NMR spectra were recorded on a Bruker WM-200-SY. The visible spectra were recorded on a Pye Unicam SP8-100 ultraviolet spectrophotometer.

### *Preparation of $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})(\text{NCS})_2$*

A solution of  $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})_2$  (1, g, 5.2 mmol) in  $\text{Et}_2\text{O}$  ( $30\text{ cm}^3$ ), in a 100 ml two-neck flask fitted with an  $\text{N}_2$  inlet, magnetic stirrer and pressure equalized dropping funnel was saturated with oxygen-free  $\text{N}_2$  then cooled to  $-20^\circ\text{C}$ . A solution of freshly prepared  $(\text{SCN})_2$  (0.6 g, 5.2 mmol) in  $\text{Et}_2\text{O}$  ( $15\text{ cm}^3$ ) was slowly added dropwise. A green solid immediately appeared, and evolution of CO was observed. The solid was filtered off on a cooled sintered glass disc ( $-20^\circ\text{C}$ ), washed several times with cooled  $\text{Et}_2\text{O}$ , and dried in vacuum. The yield was 1.24 g (85%). Found: C, 36.95; H, 3.02; N, 10.71; Co, 20.09.  $\text{C}_9\text{H}_7\text{CoON}_2\text{S}_2$  calcd.: C, 38.31; H, 2.48; N, 9.93; Co, 20.90%.

### *Preparation of $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})(\text{CN})(\text{NCS})$*

The procedure described above was used starting with solutions of  $(\text{C}_5\text{H}_4\text{CH}_3)\text{Co}(\text{CO})_2$  (1 g, 5.2 mmol) in  $\text{Et}_2\text{O}$  ( $30\text{ cm}^3$ ) and  $\text{S}(\text{CN})_2$  (0.43 g, 5.2 mmol) in  $\text{Et}_2\text{O}$  ( $20\text{ cm}^3$ ). A deep green solid was obtained. The yield was 1.03 g

(80%). Found: C, 42.23; H, 2.95; N, 10.81; Co, 23.01.  $C_9H_7CoON_2S$  calcd.: C, 43.18; H, 2.80; N, 11.20; Co, 23.56%.

*Preparation of  $(C_5H_4CH_3)Co(CO)X_2$  ( $X = Br, I$ )*

On mixing solutions of  $(C_5H_4CH_3)Co(CO)_2$  (1 g, 5.2 mmol) in light petroleum (30 cm<sup>3</sup>; b.p. 65–70°C), with liquid bromine (0.3 cm<sup>3</sup>; 5.8 mmol), with I<sub>2</sub> (1.32 g, 5.2 mmol) in the same solvent (30 cm<sup>3</sup>), deep green (1.51 g, 90%) and deep violet (1.80 g, 85%) solids were obtained, respectively. Found: C, 24.58; H, 2.40; Br, 48.33; Co, 18.08.  $C_7H_7OBr_2Co$  calcd.: C, 25.79; H, 2.15; Br, 49.06; Co, 18.9%. <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 1.59 (s, 3H); 4.04 (m, BB', 2H), 4.95 (m, AA', 2H) ppm. Found: C, 18.15; H, 1.85; I, 58.25; Co, 14.45.  $C_7H_7OI_2Co$  calcd.: C, 20.01; H, 1.65; I, 60.47; Co, 14.04%. <sup>1</sup>H NMR (CDCl<sub>3</sub>): 1.58 (s, 3H); 4.05 (m, BB' 2H); 4.95 (m, AA' 2H) ppm.

*Preparation of  $[(C_5H_4CH_3)CoCl(\mu-Cl)]_2$*

Chlorine gas was bubbled through a solution of  $(C_5H_4CH_3)Co(CO)_2$  (1 g, 5.2 mmol) in Et<sub>2</sub>O (30 cm<sup>3</sup>) cooled to –15°C. A blue-green solid separated, and this was filtered off, washed with Et<sub>2</sub>O, and dried in vacuum. The yield was 1.02 g (95%). Found: C, 32.95; H, 3.80; Cl, 32.24; Co, 27.70.  $C_{12}H_{14}Cl_4Co_2$  calcd.: C, 34.48; H, 3.35; Cl, 33.95; Co, 28.22%.

*Preparation of  $[(C_5H_4CH_3)CoX(\mu-X)]_2$  ( $X = Br, I$ )*

A suspension of  $(C_5H_4CH_3)Co(CO)X_2$  ( $X = Br$  or  $I$ ) (1 g) in light petroleum (30 cm<sup>3</sup>, b.p. 100–120°C) was refluxed. After 2 ( $X = Br$ ) or 3 h ( $X = I$ ), a black solid was obtained. The yields were 1.46 g ( $X = Br$ ) (95%) and 1.82 g ( $X = I$ ) (90%). Found: C, 23.63; H, 2.78; Br, 51.39; Co, 19.26.  $C_{12}H_{14}Br_4Co_2$  calcd.: C, 24.19; H, 2.35; Br, 53.68; Co, 19.76%. <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 1.60 (s, 2CH<sub>3</sub>); 3.35–3.70 (m, 8H). Found: C, 17.57; H, 1.95; I, 63.10; Co, 14.92.  $C_{12}H_{14}I_4Co_2$  calcd.: C, 18.38; H, 1.79; I, 64.79; Co, 15.04%.

*Preparation of  $[(C_5H_4CH_3)CoCl]_2(\mu-H)(\mu-Cl)$*

To a solution of KOH (0.20 g, 3.59 mmol) in isopropyl alcohol (30 cm<sup>3</sup>) was added  $[(C_5H_4CH_3)CoCl(\mu-Cl)]_2$  (1 g, 2.40 mmol). The suspension was stirred for 4 h. The green solid was filtered off, washed with isopropyl alcohol, and dried in vacuum. The yield is 0.68 g (75%). Found: C, 36.85; H, 4.10; Cl, 26.96; Co, 30.24.  $C_{12}H_{15}CoCl_3$  calcd.: C, 37.57; H, 3.91; Cl, 27.78; Co, 30.73%. <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 1.58 (s, 2CH<sub>3</sub>); 3.90–4.20 (m, 8H).

*Preparation of  $[(C_5H_4CH_3)CoBr]_2(\mu-H)(\mu-Br)$*

By a procedure similar to that described above a brown solid was obtained from KOH (0.14 g, 2.50 mmol) and  $[(C_5H_4CH_3)CoBr(\mu-Br)]_2$  (1 g, 1.68 mmol) in isopropyl alcohol. The yield was 0.65 g (75%). Found: C, 26.84; H, 3.12; Br, 45.91; Co, 21.98.  $C_{12}H_{15}CoBr_3$  calcd.: C, 27.88; H, 2.90; Br, 46.41; Co, 22.81%.

*Preparation of  $[(C_5H_4CH_3)CoI]_2(\mu-H)(\mu-I)$*

By a similar procedure a green solid was obtained from KOH (0.12 g, 1.90 mmol) and  $[(C_5H_4CH_3)CoI(\mu-I)]_2$  (1 g, 1.28 mmol) in isopropyl alcohol. The yield was 0.88 g (70%). Found: C, 20.94; H, 2.55; I, 56.86; Co, 18.24.  $C_{12}H_{15}CoI_3$  calcd.: C, 21.90; H, 2.28; I, 57.90; Co, 17.92%.

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