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Synthesis and mass spectra of derivatives of (η^5-cyclopentadienyl)(η^4-cyclopentadiene)cobalt

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Abstract

For the first time ever the interaction of cobaltocene with organocadmium compounds has been shown to be a convenient route to the derivatives of (η^5-cyclopentadienyl)(η^4-cyclopentadiene)cobalt. The reaction of Cp_2Co with R_2Cd (R = Me, Et, n-Pr, Ph, $GeEt_3$, CH_2SiMe_3) gave (η^5-Cp)(η^4-CpR)Co, and metallic cadmium and R-radical conversion products would be isolated. (η^5-Cp)(η^4-CpR)Co decomposes in three directions during electron impact: (i) loss of R substituents, (ii) the loss of *endo*-hydrogen and (iii) migration of *endo*-hydrogen to Cp ligand and subsequent cleavage of the Co–C_5H_4R (or Co–C_5H_6) bond.

Introduction

The first ever cyclopentadienyl-cyclopentadiene cobalt complexes, (η^5-Cp)(η^4-CpR)Co, were prepared by the reaction of Cp_2Co with RI [1]. Later on the interaction of cobalticinium salts with nucleophilic agents (RLi, RMgX) was shown to result in the formation of similar derivatives [2]. While we were investigating the 3*d*-series metallocene reactions with organocadmium compounds [3–6] we found yet another method to synthesize cyclopentadienyl-cyclopentadiene compounds: the interaction of Cp_2Co with R_2Cd. The lack of systematic data on mass spectrometric characteristics for (η^5-Cp)(η^4-CpR)Co induced us to study the mass spectra of some derivatives with various substituents to find some possible rearrangement processes during dissociative ionization.

Results and discussion

The interaction of cobaltocene with organocadmium compounds

When a liquid organocadmium compound AlK_2Cd (Alk = Me, Et, Pr) is added to cobaltocene the reaction proceeds readily at room temperature. After 24 h the

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deposition of metallic cadmium onto the walls of reaction ampule is observed. The reaction is complete after 4–6 d and is indicated by quantitative formation of cadmium. The hydrocarbons methane and ethane (Alk = Me), alkane and alkene (Alk = Et, Pr) were found in a gas phase. Reaction of a 1/1 molar ratio of Cp_2Co with Alk_2Cd gives (η^5-Cp)(η^4-CpAlk)Co (in 30–35% yields based on the initial quantity of Cp_2Co) (Table 1). Increasing the quantity of organocadmium compound relative to the stoichiometric quantity results in an increased yield of (η^5-Cp)(η^4-CpAlk)Co. If a 7–10-fold excess of organocadmium compound is used cobalt diene complex is obtained in 80–90% yield.

The reaction between Cp_2Co and R_2Cd occurs in benzene or toluene solution. This reaction is particularly convenient when the organocadmium compound is in solid state.

The reaction between Cp_2Co and Ph_2Cd was conducted in toluene solution at room temperature. We did not observe substantial variations of reaction rate when phenyl derivatives were used. The yield of the final reaction product, (η^5-Cp)(η^4-CpPh)Co, did not change significantly. In addition to the product mentioned, metallic cadmium, diphenyl, and benzene were also isolated.

The interaction of cobaltocene with organocadmium compounds is a novel method for the preparation of cobalt compounds bearing an organoelement substituent on the cyclopentadiene ring. Thus the new (η^5-Cp)(η^4-CpR)Co (R = $GeEt_3$, CH_2SiMe_3) compounds were obtained from the reaction of Cp_2Co with R_2Cd (R = $GeEt_3$, CH_2SiMe_3). Cadmium, $(GeEt_3)_2$ (R = $GeEt_3$), and Me_4Si (R = CH_2SiMe_3) were also formed.

This reaction permits the easy introduction of bulky organoelement groups into the cyclopentadienyl ring of cobaltocene and offers rich synthesis possibilities for the preparation of substituted cyclopentadienyl cobalt compounds.

The (η^5-Cp)(η^4-CpAlk)Co compounds have also been obtained by boiling cobaltocene in alkyl iodide solution.

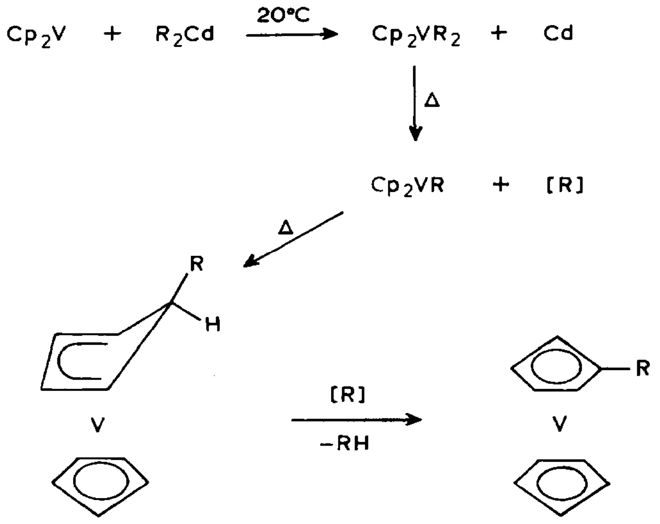
PMR spectra of the compounds, which were prepared by the various procedures suggest that the R group in all cases is in the *exo*-position with respect to the plane of the cyclopentadiene ring.

The oxidative addition of organocadmium compound to electron-deficient vanadocene was previously found to result in the formation of stable covalent d^1- and d^2-vanadocene sandwich complexes, Cp_2VR_n [3]. When these compounds were heated the σ-bonded R groups split off, with some of them migrating to the cyclopentadienyl ring of vanadocene to form substituted vanadocenes. This reaction is presumed to proceed via an intermediate diene complex (1) which is similar to the cobalt derivatives [7,8].

At the same time the coordination of organocadmium compound to the electron supersaturated nickelocene results in displacement of one cyclopentadienyl ligand and the formation of a polynuclear compound with a nickel–cadmium bond [4].

Comparison of the results of the reactions between metallocenes and organocadmium compounds indicates that such interactions are due to coordinated unsaturation of a central metallocene atom and the ability of organocadmium compound to undergo complex formation.

An attempt was made to record the presence of the adduct intermediate of cobaltocene and organocadmium compound by IR spectroscopy. It was carried out as follows: Cp_2Co was deposited on the Me_2Cd film of the optical substrate cooled



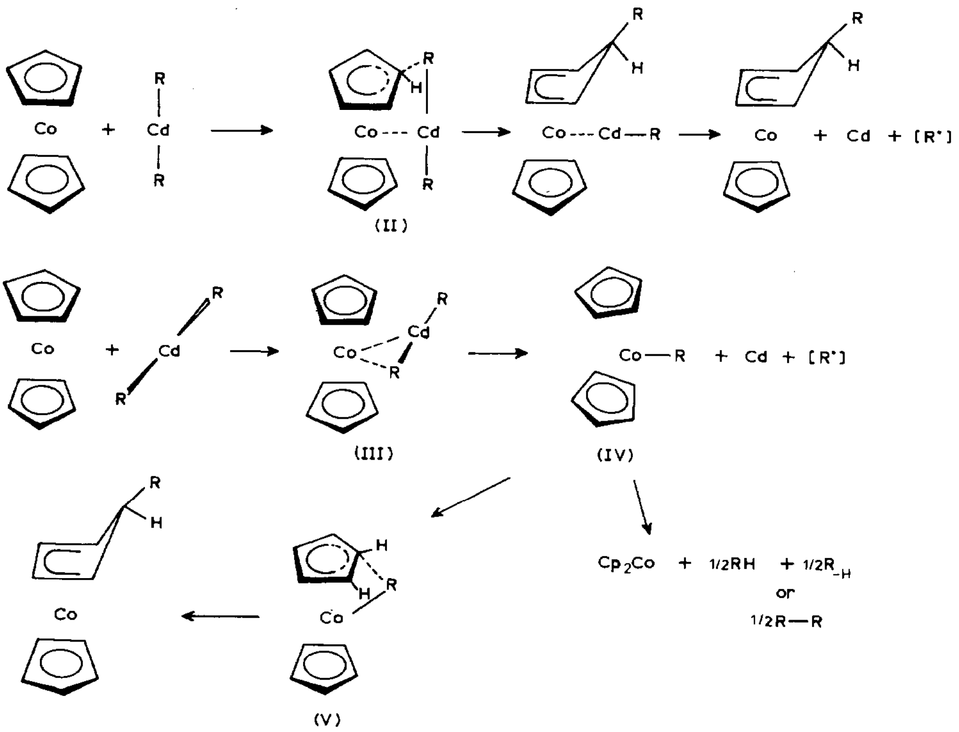
(I)

up to 80 K. IR spectra of the product of the low-temperature reaction showed as absorption bands of initial Me_2Cd. At the same time some new absorption bands (2962, 2943, 2918, 2900 and 2850 cm^{-1}), absent from spectra of the initial components are observed in the spectrum. We assigned these bands to stretching vibrations of C–H bonds of methyl group substituent on the cyclopentadiene ring. In addition, the splitting of absorption bands of the plane and out-of-plane vibrations of the C–H bonds of the cyclopentadienyl ring into two components of similar intensity is observed in the 1000–800 cm^{-1} region. Such splitting is not observed with cobaltocene.

The experimental data obtained and the available literature data do not give sufficient information as to the reaction mechanism. Cobaltocene is able to add free radicals to give cyclopentadiene-cyclopentadienyl complex [9]. But the conversion product of R-radicals by the reaction between Cp_2Co and R_2Cd cannot be explained merely in terms of free-radical processes. Although we did not succeed in fixing coordination of cadmium atom to that of cobalt, we think that the initial state of the reaction is the activation of the Cd–R bond within the coordination sphere of the cobalt atom of cobaltocene. In this case, depending on the spatial orientation of R_2Cd, two transition states, II and III, are possible (Scheme 1).

In transition state II coordination of Cd atom to that of cobalt results in the shift of electron density onto one of the cyclopentadienyl rings. The Cd–R bond is somewhat longer than that of Co–Cp, so the interaction of one R group with a carbon atom of the ring occurs over the ring plane. The introduction of R group into the *exo*-position of the η^4-Cp ring becomes energetically favourable.

In transition state III not only is the interaction between metal atoms realized, but the interaction between cobalt atom and R group also takes place. This process results in an unstable covalent sandwich complex (IV). The ratio of alkanes to alkenes being 1/1, and the fact that dimerization products are formed by reaction of Cp_2Co with R_2Cd is up to now the only evidence of the existence of an intermediate Co–R bond.



Scheme 1

The possible conversion of IV into cyclopentadienyl-cyclopentadiene complex cannot be excluded. The formation of substituted vanadocene from covalent sandwich vanadium compounds, Cp_2VR_n, indicates that R-group migration into the ring can take place [7,8]. However, to form the *exo*-isomer, R-group attack must occur between carbon atoms of the cyclopentadienyl ring of complex V with simultaneous rotation of R group over the ring plane to form the final complex (see Scheme 1).

Table 1

The reaction between Cp_2Co and R_2Cd

R_2Cd	Reaction conditions		Reaction products (mol/mol Cp_2Co)					
	Ratio Co/Cd	Solvent	Cp(CpR)Co	Cp_2Co	RH	R_{-H}	R-R	Cd
Me_2Cd	1/1	[a]	0.30	0.70	0.32	–	0.68	1.0
Et_2Cd	1/1	[a]	0.34	0.62	0.80	0.73	–	1.0
Pr_2Cd	1/1	[a]	0.35	0.64	0.86	0.74	–	1.0
	1/2	[a]	0.44	0.55	1.79	1.62	–	2.0
Ph_2Cd	1/1	toluene	0.36	0.62	0.31	–	0.64	1.0
	1/2	toluene	0.47	0.50	0.50	–	1.40	2.0
$(Et_3Ge)_2CdCd$	1/1	benzene	0.34	0.65	0.10	–	0.75	1.0
$(Me_3SiCH_2)_2Cd$		[a]	0.35	0.60	0.75	–	[b]	1.0

[a] Without solvent. [b] Not determined.

Fragmentation of (η^5-Cp)(η^4-CpR)Co by electron impact

Mass spectrometry under electron impact of all investigated Cp(CpR)Co compounds (except R = Ph) showed that they readily lost the R substituent to give Cp_2Co^+ ion, as the line of greatest intensity in the spectrum (Table 2). The peaks of the metastable ions which confirm the occurrence of these processes (Table 3, conversion 1) are observed in mass spectra of most of the compounds studied.

The main direction taken during Cp_2Co^+ ion fragmentation is consistent with the decomposition of a molecular cobaltocene ion (Table 3, conversions 5–8, 12) [10].

The second route of decomposition of molecular ions is an elimination of *endo*-hydrogen from an η^4-cyclopentadiene ligand. As a result of this a pseudomolecular ion of substituted cobaltocene, $(C_5H_5)(C_5H_4R)Co^+$ (R = Alk, CH_2SiMe_3, Ph, $GeEt_3$) occurs.

The decomposition of $(C_5H_5)(C_5H_4R)Co^+$ obeys the fragmentation regularities which were found for alkylmetallocenes [11,12]. $(C_5H_5)(C_5H_4R)Co^+$ ions lose a neutral R_{-CH_2} (R = Me, Et, n-Pr, n-Bu, i-Am) fragment to form $(C_5H_5)(C_6H_6)Co^+$ or a neutral fragment Me (R = i-Pr) to form $(C_5H_5)(C_7H_8)Co^+$ because of the cleavage of the C–C bond in the alkyl substituent (Table 3, conversions 3, 4).

Furthermore $(C_5H_5)(C_5H_4R)Co^+$ ion loses cyclopentadiene molecule to form $(C_5H_4R_{-H})Co^+$ ion (Table 3, conversion 2). This process occurs with transfer of hydrogen atom from alkylcyclopentadienyl ligand to cyclopentadienyl. Hydrogen migrates from carbon α-atom of alkyl group of cyclopentadienyl ring [12]. The intermediate formation of σ-bond between hydrogen atom and that of cobalt is assumed during this migration. Prior to the elimination of cyclopentadiene molecule the hydride ion isomerizes from having bis-cyclopentadienyl structure to one having fulvenecyclopentadiene structure:

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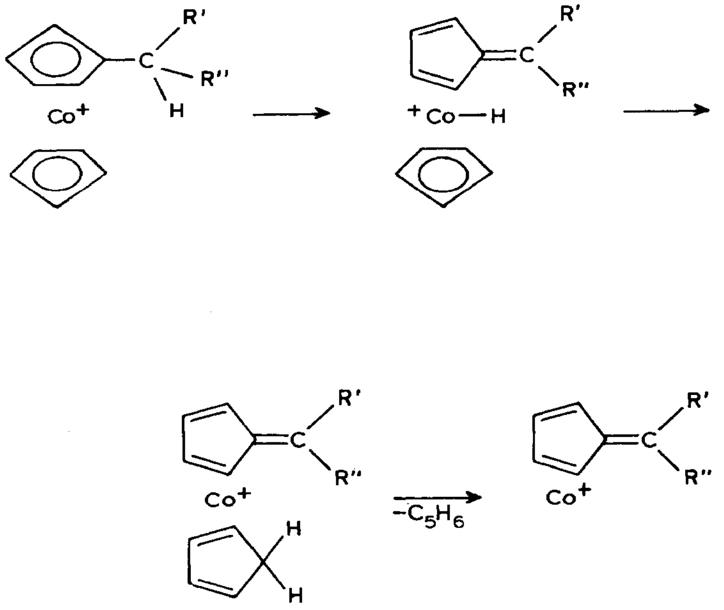


(1)

(R = CHR'R'')

Table 2

Mass spectra of Cp(CpR)Co

m/e	Ion	Relative intensity								
		R = Me	R = Et	R = n-Pr	R = i-Pr	R = n-Bu	R = i-Am	R = CH_2SiMe_3	R = $GeEt_3$	R = Ph
	$(C_5H_5)(C_5H_5R)Co^+$	16.4	4.0	4.7	1.2	2.5	0.5	4.4	–	8.8
	$(C_5H_5)(C_5H_4R)Co^+$	53.3	8.8	11.1	8.5	15.8	3.2	21.6	0.1	100.0
232	$(C_5H_5)(C_8H_{12})Co^+$	–	–	–	–	–	–	–	3.8	–
231	$(C_5H_5)(C_8H_{11})Co^+$	–	–	–	–	–	–	–	5.7	–
216	$(C_5H_5)(C_7H_8)Co^+$	–	–	–	5.2	0.9	0.3	–	–	–
203	$(C_5H_5)(C_6H_7)Co^+$	–	–	–	–	–	–	11.3	0.5	1.3
202	$(C_5H_5)(C_6H_6)Co^+$	1.1	4.4	6.3	–	12.2	2.1	2.9	3.5	2.9
200	$PhC_5H_4Co^+$	–	–	–	–	–	–	–	–	3.6
189	$(C_5H_5)_2Co^+$	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	7.5
187	$C_{10}H_8Co^+$	0.5	1.4	0.9	0.6	2.7	2.2	0.2	2.1	–
174	$C_9H_7Co^+$	0.4	0.6	3.0	0.8	1.6	1.0	0.1	0.3	1.0
166	$C_8H_{11}Co^+$	–	–	0.2	–	–	–	0.2	–	–
165	$C_8H_{10}Co^+$	–	–	0.4	0.3	–	–	0.3	0.3	0.8
163	$C_8H_8Co^+$	0.2	0.5	1.0	0.6	1.0	0.9	0.1	0.7	0.2
151	$C_7H_8Co^+$	–	0.9	0.3	1.2	0.7	–	0.7	0.1	0.2
150	$C_7H_7Co^+$	–	2.3	2.4	1.1	0.7	–	0.3	0.4	0.2
138	$C_6H_7Co^+$	–	–	3.4	0.3	–	–	0.7	–	0.2
137	$C_6H_6Co^+$	15.5	1.8	4.2	1.2	4.3	1.9	5.2	4.5	0.5
136	$C_6H_5Co^+$	0.7	–	0.2	–	2.0	3.5	0.2	0.5	0.3
135	$C_6H_4Co^+$	–	–	1.0	1.9	–	–	1.0	0.6	0.4
125	$C_5H_6Co^+$	1.2	2.3	1.5	3.5	2.0	1.2	1.0	1.8	1.8
124	$C_5H_5Co^+$	21.5	18.6	19.3	16.3	31.3	25.3	14.5	23.2	9.7
99	$C_3H_4Co^+$	0.5	0.4	0.6	0.7	0.4	0.6	0.3	1.0	0.2
98	$C_3H_3Co^+$	7.4	4.8	4.8	3.9	6.3	6.7	3.3	6.7	2.2
97	$C_3H_2Co^+$	2.8	2.0	1.5	1.5	2.6	2.6	1.2	2.6	1.6

96	C_3HCo^+	0.7	0.5	0.3	0.2	0.6	0.7	0.2	0.5	0.3
85	$C_2H_2Co^+$	1.8	1.2	1.0	0.9	1.3	1.1	0.7	1.1	0.6
84	C_2HCo^+	2.1	1.2	1.0	0.8	1.6	1.6	0.6	1.5	0.9
74	CH_3Co^+	0.3	0.3	0.3	0.2	0.2	0.2	0.7	–	0.2
73	Me_3Si^+	–	–	–	–	–	–	9.4	–	–
60	HCo^+	1.0	0.3	0.5	0.2	0.4	0.5	0.4	0.5	0.2
59	Co^+	25.6	15.1	16.2	12.1	23.2	22.4	13.8	21.0	14.8
133	$(C_5H_5)(C_5H_5R)Co^{2+}$	–	–	–	–	–	–	–	–	0.7
132.5	$(C_5H_5)(C_5H_4R)Co^{2+}$	–	–	–	–	–	–	–	–	3.0
94.5	$(C_5H_5)_2Co^{2+}$	0.4	0.7	0.5	0.5	2.1	2.3	0.5	0.1	0.1

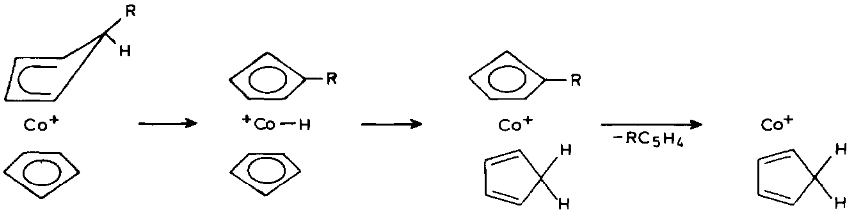
Table 3

Electron impact conversions. The apparent mass (m^*) of metastable ions in mass spectra of Cp(CpR)Co

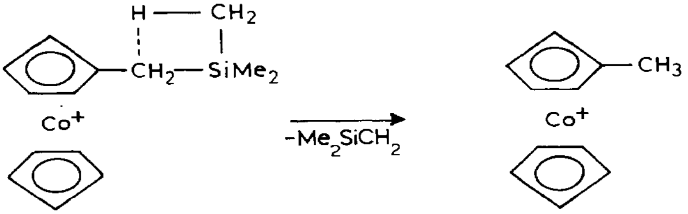
Nr.	Conversion	m^*							
		R = Me	R = Et	R = n-Pr	R = i-Pr	R = n-Bu	R = i-Am	R = CH_2SiMe_3	R = $GeEt_3$
1	$(C_5H_5)(C_5H_5R)Co^+ \rightarrow R + (C_5H_5)_2Co^+$	175.1	163.9	154.0	154.0	145.2	–	129.4	102.1
2	$(C_5H_5)(C_5H_4R)Co^+ \rightarrow C_5H_6 + R_{-H}C_5H_4Co^+$	95.2	–	–	–	–	–	–	–
3	$(C_5H_5)(C_5H_4R)Co^+ \rightarrow R_{-CH_2} + (C_5H_5)(C_6H_6)Co^+$	–	–	176.6	–	166.5	–	–	–
4	$(C_5H_5)(C_5H_4R)Co^+ \rightarrow CH_3 + (C_5H_5)(C_7H_8)Co^+$	–	–	–	202.0	–	–	–	–
5	$(C_5H_5)_2Co^+ \rightarrow C_5H_5 + C_5H_5Co^+$	81.3	81.3	81.3	81.3	81.3	81.3	81.3	81.3
6	$(C_5H_5)_2Co^+ \rightarrow H_2 + C_{10}H_8Co^+$	185.0	185.0	185.0	185.0	185.0	185.0	185.0	185.0
7	$(C_5H_5)_2Co^+ \rightarrow CH_3 + C_9H_7Co^+$	160.2	160.2	160.2	160.2	160.2	160.2	160.2	160.2
8	$(C_5H_5)_2Co^+ \rightarrow C_2H_2 + C_8H_8Co^+$	140.6	140.6	140.6	140.6	140.6	140.6	140.6	140.6
9	$C_7H_8Co^+ \rightarrow C_2H_2 + C_5H_6Co^+$	–	103.5	–	–	–	–	–	–
10	$C_7H_7Co^+ \rightarrow C_2H_2 + C_5H_5Co^+$	–	102.5	–	–	–	–	–	–
11	$C_6H_6Co^+ \rightarrow C_6H_6 + Co^+$	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4
12	$C_5H_5Co^+ \rightarrow C_5H_5 + Co^+$	28.1	28.1	28.1	28.1	28.1	28.1	28.1	28.1
13	$(C_5H_5)(C_5H_4R)Co^+ \rightarrow C_3H_7Ge + (C_5H_5)(C_8H_{12})Co^+$	–	–	–	–	–	–	–	154.2
14	$(C_5H_5)(C_8H_{11})Co^+ \rightarrow C_2H_5 + (C_5H_5)(C_6H_6)Co^+$	–	–	–	–	–	–	–	176.6

Rearrangement (1) is most likely to occur in $(C_5H_5)(C_5H_4R)Co^+$ ions for $R = Me$ (Tables 2 and 3, conversion 2) with increasing n in $R = C_nH_{2n+1}$ the role of this rearrangement in the decomposition of ions becomes negligible.

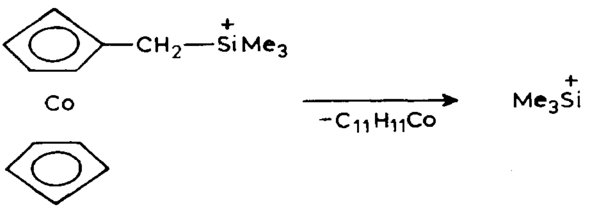
Lastly a third direction of decomposition of the molecular ion is possible. It results in the formation of $C_5H_6Co^+$ or $C_5H_4RCo^+$. It can be seen as a process involving the migration of *endo*-hydrogen from substituted ligand to the unsubstituted via the metal centre with the formation of a Co–H bond. The coordination of bonds between cobalt and ligands changes accordingly with each step of *endo*-hydrogen migration:



Some peculiarties in the fragmentation of $Cp(CpCH_2SiMe_3)Co$ are observed. Loss of the *endo*-hydrogen from the molecular ion results in $(C_5H_5)(C_5H_4CH_2SiMe_3)Co^+$. This ion undergoes rearrangement to the $(C_5H_5)(C_6H_7)Co^+$ ion, in addition to the fragmentation processes typical of other alkylcobaltocenes. Such a process probably proceeds via an intermediate four-membered cycle formed by interaction of methylene group with trimethylsilyl one:



If a positive charge is localized on the Me_3Si group the migration of hydrogen does not occur and Me_3Si^+ ion gives rise to a line of moderate intensity in the mass spectrum.



The compound $(C_5H_5)(C_5H_5GeEt_3)Co$ is unstable under electron impact, so that in the mass spectrum, the molecular ion line is absent (Table 2) but the peak of a metastable ion is observed which confirms that the elimination of Et_3Ge substituent from a molecular ion takes place (Table 3, conversion 1). The elimination of triethyl germyl substituent is a dominant direction in the decomposition of a molecular ion and results in the formation of $(C_5H_5)_2Co^+$ ion, which gives rise to a line of the greatest intensity in the mass spectrum. The line of $(C_5H_5)(C_5H_4GeEt_3)$-Co^+ ion which forms as a result of hydrogen atom elimination from molecular ion has negligible intensity. The decomposition of $(C_5H_5)(C_5H_4GeEt_3)Co^+$ ion proceeds with elimination of neutral C_3H_7Ge fragment, which leads to the rearrangement ion $(C_5H_5)(C_8H_{12})Co^+$, (Table 3, conversion 13) which loses a hydrogen atom to give $(C_5H_5)(C_8H_{11})Co^+$. The loss of an ethyl group from this ion (Table 3, conversion 14) is consistent with conversion 3 (Table 3) and the structure of propylcobaltocene ion was assigned to $(C_5H_5)(C_8H_{11})Co^+$.

The fragmentation of $(C_5H_5Ph)Co^+$ compound can take one of two directions, one is the initial elimination of phenyl group, and the second elimination of *endo*-hydrogen from a molecular ion, which is the more favourable because of the greater stability of $(C_5H_5)(C_5H_4Ph)Co^+$ ion compared with that of cobaltocene. Naturally further decomposition of phenylcobaltocene ion results in a relatively small quantity of products smaller than that obtained for the decomposition of cobaltocene ion (Table 2).

Experimental

Mass spectra of the investigated compounds were recorded with a MI-1305 mass-spectrometer, at an accelerating voltage of 2 kV, electron ionization energy of 70 eV and an emission current of 1.5 mA. The introduction of compounds was carried out through a measuring valve for compounds with R = alkyl or by placing them directly into the ion source for compounds with R = CH_2SiMe_3, $GeEt_3$, Ph.

Reaction between Cp_2Co and R_2Cd

The mixture of Cp_2Co and R_2Cd was kept at room temperature for 4–6 d. The reaction mixture was extracted with hexane, and solution was passed through a column containing Al_2O_3. After removal of the solvent, Cp(CpR)Co was obtained

Table 4

Analytical data of Cp(CpR)Co

Compound	Analysis (Found (calc)(%))		PMR spectrum [a]				
	C	H	Cp	H(1,4)	H(2,3)	H(5)	R
$(C_5H_5)(C_5H_5Me)Co$	64.53	6.40	4.66	2.67	5.09	2.22	0.36, 0.43
	(64.71)	(6.41)					
$(C_5H_5)(C_5H_5Et)Co$	66.01	6.85	4.65	2.76	5.09	2.61	0.71, 0.74
	(66.06)	(6.92)					
$(C_5H_5)(C_5H_5Pr)Co$	67.11	7.29	4.62	2.69	5.07	2.38	0.88
	(67.24)	(7.38)					
$(C_5H_5)(C_5H_5Ph)Co$	72.01	5.60	4.65	2.89	5.11	3.84	
	(72.18)	(5.68)					
$(C_5H_5)(C_5H_5GeEt_3)Co$	55.0	7.12	4.71	2.70	5.16	2.53	0.50
	(55.09)	(7.22)					
$(C_5H_5)(C_5H_5CH_2SiMe_3)Co$	60.50	7.52	4.70	2.71	5.16	2.52	−0.10
	(60.85)	(7.66)					

[a] 80 MHz, TMS ($\delta \equiv 0$ ppm); solvent C_6H_6 (for R = Me, Et, Pr, Ph) and $CHCl_3$ (for R = $GeEt_3$, CH_2SiMe_3).

as red liquid. Stereochemistry of the reactions and the yields of the reaction products are described under results and discussion. The characteristics of Cp(CpR)Co are listed in Table 4.

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