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ORGANOCOBALT CLUSTER COMPLEXES

XXVI *. THERMOLYSIS OF ALKYLIDYNETRICOBALT NONACARBONYL COMPLEXES. EVIDENCE FOR CARBYNE INTERMEDIATES

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Summary

The thermolysis of methylidynetricobalt nonacarbonyl, $HCCo_3(CO)_9$, in refluxing xylene solution gives a mixture of $CH_3CCo_3(CO)_9$ and $C_2H_5CCo_3(CO)_9$. A reaction course involving the generation of a carbyne, $H\dot{C}:$, and its addition to the CCo_3 core of the $HCCo_3(CO)_9$ molecule, followed by extrusion of $Co(CO)_3$ to give acetylenehexacarbonyldicobalt and finally decomposition of the latter to form $CH_3CCo_3(CO)_9$, is suggested. A separate experiment confirmed that the last step can occur.

Introduction

The decomposition of organic derivatives of the carbon tricobaltcarbon nonacarbonyl cluster, I, in principle could proceed by release of the organic fragment as a carbyne (eq. 1). There are in the literature some fragmentary reports which

$$RCCo_3(CO)_9 \rightarrow R\dot{C}: + 3\ Co + 9\ CO \qquad (1)$$

provide evidence that such may indeed be the case. In earlier work [2], we had shown that $PhCCo_3(CO)_9$ decomposes to give diphenylacetylenehexacarbonyldicobalt when it was heated for 24 h in refluxing methanol solution under argon or under air (eq. 2) The acetylene complex was obtained in 39% conversion, or, based on unrecovered $PhCCo_3(CO)_9$, in 63% yield. Other substituted benzylidynetricobalt nonacarbonyls, $ArCCo_3(CO)_9$ (Ar = m-$CH_3C_6H_4$, p-$CH_3OC_6H_4$, p-$CH_3C(O)C_6H_4$) decomposed in similar fashion under these conditions. More drastic conditions produced the free acetylene [3]. Thus when a diglyme

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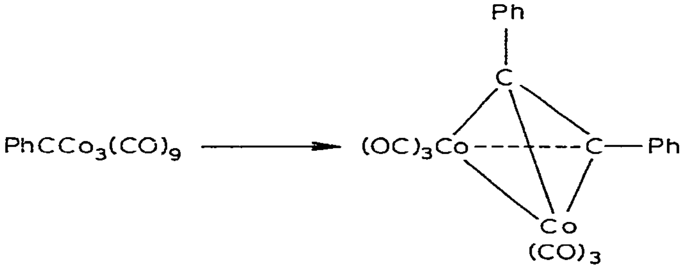
* For part XXV see ref. 1.



(2)

solution of benzylidynetricobalt nonacarbonyl was heated at reflux for 8 h, cobalt metal was formed, and a small amount of diphenylacetylene was isolated. The thermal decomposition of $PhCCo_3(CO)_8PPh_3$ under these conditions was more rapid and also gave some diphenylacetylene [3]. Similarly, thermolysis of $PhCH_2CCo_3(CO)_9$ in diglyme at reflux resulted in formation of metallic cobalt and $PhCH_2C{\equiv}CCH_2Ph$ [3].

The formation of acetylene or acetylenehexacarbonyldicobalt complexes in these thermolyses is suggestive of the intermediacy of carbynes. Indeed, the $RCCo_3(CO)_9$ compounds may be viewed as μ_3-carbyne complexes of the $Co_3(CO)_9$ unit [2], so their decomposition to give this fragment would not be surprising. Simple carbyne complexes, e.g., $PhC{\equiv}Cr(CO)_4Br$, also decompose, at about 30°C, to give acetylenes, $PhC{\equiv}CPh$ in the case cited [4]. Furthermore, the decomposition of $CH_3C{\equiv}Cr(CO)_4Br$ in the presence of dicobalt octacarbonyl was found by Fischer and Däweritz [5] to give the μ_3-carbyne complex, $CH_3CCo_3(CO)_9$. In none of these processes in which acetylenes are formed is the mechanism necessarily a case of carbyne dimerization. In fact, the dimerization of such reactive fragments in a solution process is a rather remote possibility. Much more likely is a reaction course in which the carbyne fragment adds to or inserts into an intact cluster molecule, with subsequent release of one or all of the $Co(CO)_3$ units, depending on the reaction conditions.

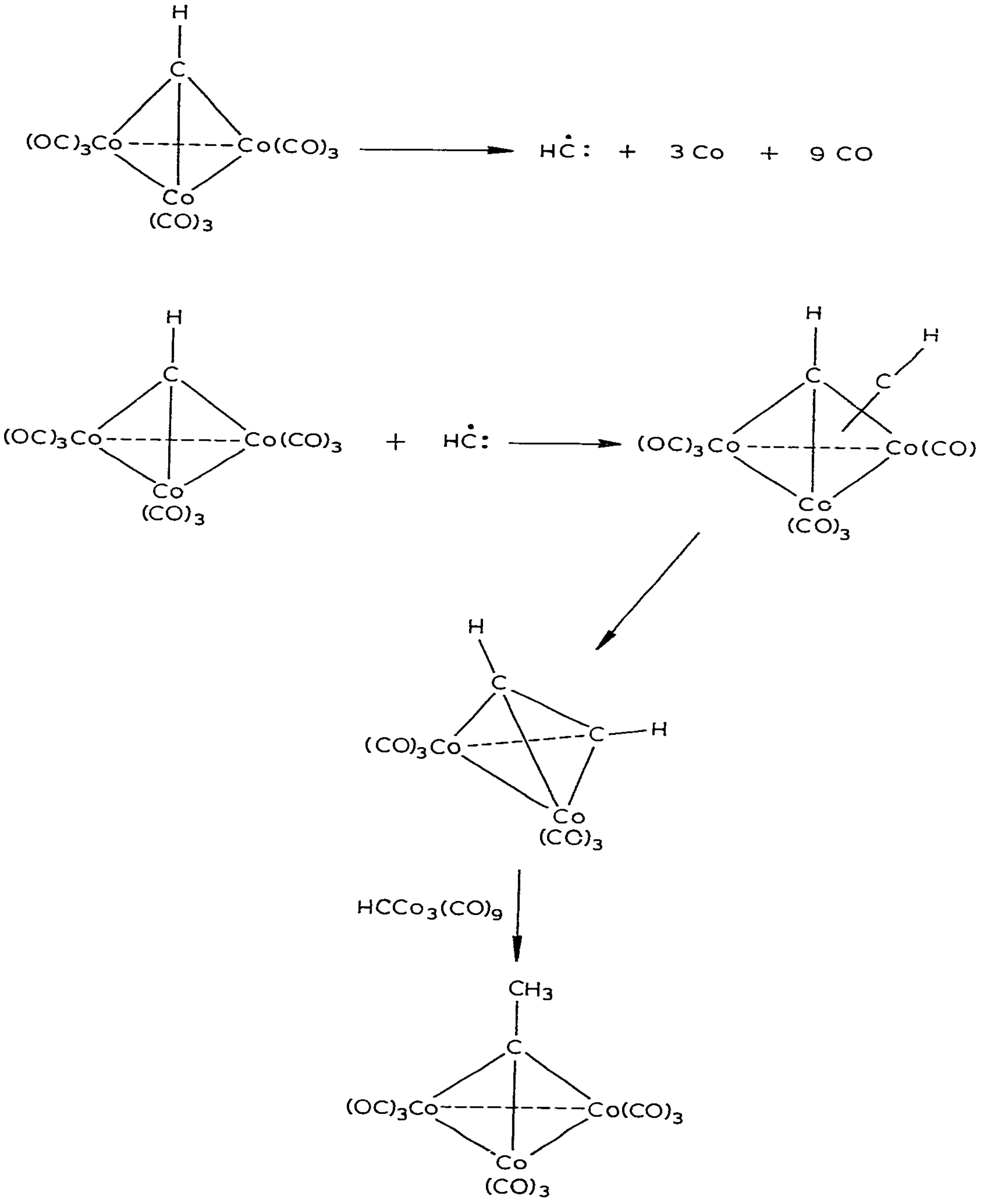
We report here some thermolysis reactions the results of which agree well with the general picture indicated above.

Results and discussion

The thermolysis of the parent cluster complex itself, methylidynetricobalt nonacarbonyl, $HCCo_3(CO)_9$, in xylene solution at reflux was examined. Rather than acetylenehexacarbonyldicobalt, the product expected on the basis of the considerations above, the products were alkylidynetricobalt nonacarbonyl complexes, $CH_3CCo_3(CO)_9$ and $C_2H_5CCo_3(CO)_9$. To establish the provenence of the hydrogen atoms in the products, the thermolysis of $DCCo_3(CO)_9$ in refluxing xylene was examined. The products obtained were $CD_3CCo_3(CO)_9$ and $C_2D_5CCo_3(CO)_9$.

One may rationalize the formation of these products in the following way: Decomposition of a molecule of $HCCo_3(CO)_9$ leads to the generation of the carbyne $H\dot{C}$: which adds to an as yet undecomposed molecule of $HCCo_3(CO)_9$, giving $(HC_2H)Co_2(CO)_9$. One could picture this process as an addition of $H\dot{C}$: to a face of the CCo_3 tetrahedral core or as an insertion into a C—Co bond. The adduct thus formed loses a $Co(CO)_3$ unit, forming the known acetylenehexacarbonyldicobalt, $(HC_2H)Co_2(CO)_6$. The latter, under the reaction conditions, then undergoes conversion to $CH_3CCo_3(CO)_9$. The second product, $C_2H_5CCo_3(CO)_9$, is formed by a similar sequence, the first step of which involves $H\dot{C}$: addition to $CH_3CCo_3(CO)_9$ and/or of $CH_3\dot{C}$: to $HCCo_3(CO)_9$. The postulated reaction course is shown in Scheme 1. Inspection of this Scheme will make

SCHEME 1



apparent that only low yields of $CH_3CCo_3(CO)_9$ can be expected. Two molecules of $HCCo_3(CO)_9$ are required to provide the carbon atoms of one molecule of $CH_3CCo_3(CO)_9$ and, furthermore, one molecule of $HCCo_3(CO)_9$ must be sacrificed (as the $DCCo_3(CO)_9$ experiment showed) to provide the third hydrogen atom of the methyl group of the $CH_3CCo_3(CO)_9$.

An alternative mechanism also must be mentioned. Whenever one postulates the generation of a reactive intermediate from an organometallic starting material, one cannot assume, in the absence of experimental support, that one is dealing with this reactive intermediate as the free species. The possibility must be considered that the "reactive intermediate" is transferred directly from the organometallic reagent to the substrate *. Thus, in the present reactions processes in which the $R\dot{C}$: fragment is transferred directly from one cluster to another or from a cluster fragment to an intact cluster cannot be discounted. We shall continue our discussion in terms of "carbyne" intermediates, with the proviso, however, that these processes could just as well be "carbynoid".

In support of these general ideas, we note that Dickson and Michel [8] found $PhCH_2CCo_3(CO)_9$ among the products of the thermolysis of phenylacetylenehexacarbonyldicobalt, $(PhC_2H)Co_2(CO)_6$, at 165°C and that the reaction of phenylacetylene with dicobalt octacarbonyl, when carried out at higher temperatures without isolation of the acetylene complex, also gave $PhCH_2CCo_3(CO)_9$ [9]. Furthermore, our own experiments have shown that acetylenehexacarbonyldicobalt gives $CH_3CCo_3(CO)_9$ when it is heated in refluxing xylene under nitrogen.

The insertion of the organic fragment of one $RCCo_3(CO)_9$ complex into another, $R'CCo_3(CO)_9$, was demonstrated in two different reactions. When $HCCo_3(CO)_9$ was heated in refluxing xylene in the presence of an equimolar quantity of $PhCCo_3(CO)_9$, the isolated products consisted of $CH_3CCo_3(CO)_9$ and $C_2H_5CCo_3(CO)_9$ from the decomposition of the former, as well as a small amount of $PhCH_2CCo_3(CO)_9$. The latter product must have been formed by way of an initial addition of $H\dot{C}$: to the $PhCCo_3(CO)_9$ cluster. Additionally, the thermolysis of a mixture of $DCCo_3(CO)_9$ and $CH_3CCo_3(CO)_9$ gave not only the $CD_3CCo_3(CO)_9$ and $C_2D_5CCo_3(CO)_9$ expected from the decomposition of $DCCo_3(CO)_9$ but also some $CH_3CD_2CCo_3(CO)_9$ and $CH_3CHDCD_2CCo_3(CO)_9$. The latter two complexes must have been formed in processes involving $D\dot{C}$: insertion into $CH_3CCo_3(CO)_9$.

Attempts to trap $R\dot{C}$: intermediates with other substrates were unsuccessful. One must conclude that either the $RCCo_3(CO)_9$ are more effective traps for $R\dot{C}$: species or that a direct cluster—cluster or cluster fragment—cluster interaction is involved rather than a free carbyne. When $PhCCo_3(CO)_9$ was heated at 150° C in the presence of a large excess of triethylsilane, a compound known to be an excellent trap for carbenes [10], no Et_3SiCH_2Ph was obtained. This was the expected product by way of $Ph\dot{C}$: insertion into the Si—H bond followed by H atom abstraction by the intermediate $Et_3Si\dot{C}HPh$ radical. Instead, the $PhCCo_3(CO)_9$-derived product was toluene, which presumably was formed by

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* In carbene chemistry, for instance, both types of processes are known. Thus in the transfer of CH_2 to olefins from ICH_2ZnI a direct, bimolecular reaction is involved [6], while in the transfer of CCl_2 to olefins using $PhHgCCl_2Br$, free CCl_2 is an intermediate [7].

cluster hydrogenolysis by the silicon hydride. The organosilicon product was hexaethyldisilane. An attempt also was made to trap $\mathrm{Ph\dot{C}}$: by reaction with cyclohexene, a reagent which had been used successfully to trap a photochemically generated carbyne [11]. However, in our experiment, thermolysis of $PhCCo_3(CO)_9$ in cyclohexene solution in a sealed tube at 150°C for 20 h resulted in a 40% recovery of the starting cobalt complex. The volatile fraction of the reaction mixture did not contain 7-phenylnorcarane, the expected phenylcarbyne-derived product, but rather dicyclohexyl ketone and another ketone believed to be dicyclohexenyl ketone were found to be present. Thermolysis of $HCCo_3(CO)_9$ in cyclohexene solution, carried out in the same manner, also gave some dicyclohexyl ketone, as well as some $CH_3CCo_3(CO)_9$.

Experimental

General comments

1. Procedures

The standard apparatus consisted of a flame-dried, 100 ml three-necked flask equipped with a magnetic stirring bar, a thermometer and a reflux condenser. The reactions were carried out under an atmosphere of dry nitrogen.

The standard apparatus was charged with methylidynetricobalt nonacarbonyl and the appropriate solvent. The reaction mixture was heated to reflux while it was stirred under nitrogen, thin-layer chromatography (TLC) being used to monitor the progress of the decomposition (Eastman Chromagram Sheet No. 6060). Upon completion of the decomposition the reaction mixture was cooled to room temperature. The solvent was removed at reduced pressure (a tedious process when a high-boiling solvent is used since the $RCCo_3(CO)_9$ complexes are rather volatile). Filtration chromatography (silicic acid) was used to separate the products, $RCCo_3(CO)_9$, from the decomposition residue. It is imperative that all product materials be sublimed prior to their spectroscopic identification and determination. Sublimation should be carried out in vacuum at low enough temperature (ca. 40°C) so that less volatile products are not decomposed before they can sublime. Also, the sublimation should be carried out for a sufficiently long time so that the less volatile products can sublime. It has been found that as the length of the carbon chain attached to the apical carbon atom increases, the sublimation time generally increases. Care must be taken to ensure that spectra are recorded of a homogeneous mixture, so all products were ground together well in an agate mortar and pestle after sublimation.

Care also must be taken in the disposal of the residues from the thermolysis reactions. In these experiments either a mirror of metallic cobalt or a finely divided cobalt powder precipitate is formed; both are pyrophoric in air when dry.

2. Spectroscopy

NMR and IR spectroscopy were carried out using the most concentrated samples possible for easier detection of low yield products. Product distribution yields for each reaction mixture were calculated for the NMR integrations. Product indentification was facilitated by comparison of the IR and NMR spectra of the mixtures with those of known samples which were suspected to be products. This is not easy and straightforward, and IR and NMR spectra had to be used in combination.

Infrared spectra. The spectra generally were determined using samples dissolved in carbon tetrachloride in 0.1 mm sodium chloride cavity cells. Listed below are the bands for each compound which were used in its identification (values in cm^{-1}).

$HCCo_3(CO)_9$: 865m and 720w.
$DCCo_3(CO)_9$: 680m.
$CH_3CCo_3(CO)_9$: four weak bands in the 3000—2800 region; 1420w, 1360w, 1165m, and 1010m.
$C_2H_5CCo_3(CO)_9$: four weak bands in the 3000—2800 region; 1450w, 1420w, 1370w, 1155m, 1050w, 1040w, and 640m.

^1H Nuclear magnetic resonance spectra. Carbon tetrachloride was used as solvent and chloroform was added as internal standard. Listed below are the resonances (in δ units, downfield from internal tetramethylsilane) for each compound which were used in its identification.

$HCCo_3(CO)_9$: 12.0 (s).
$CH_3CCo_3(CO)_9$: 3.6 (s).
$C_2H_5CCo_3(CO)_9$: 1.5 (t, $J = 7$ Hz), 3.6 (q, $J = 7$ Hz).

^2H Nuclear magnetic resonance spectra. Carbon tetrachloride was used as solvent with $DCCl_3$ as reference. The deuterium resonance of $DCCo_3(CO)_9$ occurs at 4.81 ppm downfield from internal $DCCl_3$. Authentic samples of $CD_3CCo_3(CO)_9$ and $C_2D_5CCo_3(CO)_9$ were not available, but a sample of CHD_2-$CCo_3(CO)_9$ from a previous project [12] was on hand and this compound showed its deuterium resonance at 3.53 ppm upfield from internal $DCCl_3$. The ^2H NMR spectra were obtained using a modified Bruker HFX 90 spectrometer which was interfaced with a Digilab FTS/NMR-3 data system operating at 13.82 MHz in the FT mode.

Thermolysis of methylidynetricobalt nonacobalt in xylene at 135° C

The standard apparatus was charged with 1.00 g (2.26 mmol) of methylidynetricobalt nonacarbonyl and 30 ml of xylene (mixed isomers). The solution was heated at 135° C under nitrogen for 30 min. The standard work-up procedure was followed by filtration chromatography. Elution with hexane followed by sublimation in vacuo at 40° C gave 0.34 g of a mixture of alkylidynetricobalt nonacarbonyl complexes. IR and NMR spectra of the mixture established the presence of $HCCo_3(CO)_9$, $CH_3CCo_3(CO)_9$ and $C_2H_5CCo_3(CO)_9$ and possibly even of n-$C_3H_7CCo_3(CO)_9$. The total yield of the mixture was 80%.[*] The composition of the mixture was: $HCCo_3(CO)_9$, 18%; $CH_3CCo_3(CO)_9$, 63%; $C_2H_5CCo_3(CO)_9$, 19%. IR (CCl_4, cm^{-1}): 2965w, 2915w, 2875w, 2810w, 2110—1980vs, 1450w, 1420w, 1350w, 1160m, 1150(sh), 1050vw, 1040vw, 1005s, 860m, 720w, 640m. NMR (CCl_4): 1.25 (s), 1.53 (t), 3.70 (s), 3.80 (s), 11.83 (s) ppm.

The mass spectrum (70 eV, inlet temperature about 250° C) also served to

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[*] Relative ratios of products were calculated from the NMR integration of the protons of each component. Absolute yields were calculated by assuming that all protons came from $HCCo_3(CO)_9$ and by knowing the relative ratios of products in the mixture and the total weight of the mixture.

TABLE 1

SIGNIFICANT FRAGMENT IONS IN THE MASS SPECTRUM OF THE THERMOLYSIS PRODUCT MIXTURE

Fragment ion	Mass (m/e)	Relative intensity
$(OC)_9Co_3CC_2H_5$	470	1.33
$(OC)_9Co_3CCH_3$	456	9.78
$(OC)_9Co_3CH/(OC)_8Co_3CC_2H_5$	442	24.80
$(OC)_5Co_3CH/(OC)_4Co_3CC_2H_5$	330	70.25
$(OC)_4Co_3CCH_3$	316	69.73
$(OC)Co_3CH/Co_3CC_2H_5$	218	42.53
Co_3CCH_3	204	28.98
Co_3CH	190	70.67
$Co_2CC_2H_5$	159	5.36
Co_2CH	131	48.55
$(OC)Co$	87	44.63
CoC	71	8.08

identify the three components of the mixture. The mass difference between $C_2H_5CCo_3(CO)_9$ and $HCCo_3(CO)_9$ is 28, which is also the mass of CO. This serves to complicate a discussion of fragmentation patterns since $C_2H_5CCo_3(CO)_{n-1}$ and $HCCo_3(CO)_n$ (n 1 through 9) have the same mass. A selection of the peaks of significance is given in Table 1. The mass spectroscopy of alkylidynetricobalt nonacarbonyl complexes has been discussed [13].

Thermolysis of methylidynetricobalt nonacarbonyl-d in xylene at 135°C

The standard apparatus was charged with 1.00 g (2.26 mmol) of $DCCo_3(CO)_9$ [14] and 30 ml of xylene (mixed isomers). The solution was heated under nitrogen for 30 min. The standard work-up procedure was followed by filtration chromatography (hexane). Sublimation in vacuo at 40°C gave 0.35 g of a mixture of alkylidynetricobalt nonacarbonyls. IR and NMR spectroscopy was used to determine the product composition. The complexes $DCCo_3(CO)_9$, $CD_3CCo_3(CO)_9$ and $C_2D_5CCo_3(CO)_9$ were present. One partially deuterated product (^{1}H NMR: δ 1.27 (s)) also was present. The total yield of the mixture was 83%. The product composition was $DCCo_3(CO)_9$, 13%; $CD_3CCo_3(CO)_9$, 56%; $C_2D_5CCo_3(CO)_9$, 14%. IR (in CCl_4, cm^{-1}): 2950w, 2915w, 2850w, 2190w, 2110—1980vs, 1165m, 680s. ^{2}H NMR (CCl_4); +4.95 (s), —3.53 (s), —5.65 (s) ppm, vs. internal $DCCl_3$. Tentative assignments are: —3.53 resonance: $CD_3CCo_3(CO)_9$ + CD_2 of $C_2D_5CCo_3(CO)_9$: —5.65 resonance: CD_3 of $C_2D_5CCo_3(CO)_9$. The intensities of the signals in the ^{2}H NMR spectrum were used to calculate the product composition; thus these figures are less accurate than those for the $HCCo_3(CO)_9$ experiment which were based on the integrations obtained using a Varian Associates T-60 spectrometer.

Thermolysis of acetylenehexacarbonyldicobalt in xylene at 135°C

The standard apparatus (200 ml flask) was charged with 1.50 g (4.8 mmol) of acetylenehexacarbonyldicobalt [15] and 100 ml of xylene (mixed isomers). The solution was heated at reflux for 30 min. Subsequently the solvents was removed at reduced pressure and the residue was subjected to column chromatography (silicic acid, hexane eluent). The major product (0.2 g) was $CH_3CCo_3(CO)_9$, m.p. 182—184°C (lit. [14] m.p. 183—184°C), which also was identified by its IR spectrum. Two additional minor products were eluted with hexane and benzene, but these were not identified.

Thermolysis of methylidynetricobalt nonacarbonyl in the presence of benzylidynetricobalt nonacarbonyl

The standard apparatus was charged with 1.00 g (2.26 mmol) of $HCCo_3(CO)_9$, 1.09 g (2.11 mmol) of $PhCCo_3(CO)_9$ and 30 ml of xylene. The solution was stirred and heated at reflux under nitrogen for 30 min. Subsequent workup was as described above. Column chromatography gave two product fractions: (1) a purple solid, 0.37 g, identified by NMR and IR as being composed of $HCCo_3(CO)_9$ (63%), $CH_3CCo_3(CO)_9$ (28%) and $C_2H_5CCo_3(CO)_9$ (9%), and (2) a brown solid, 0.99 g, identified by NMR as a mixture of $PhCCo_3(CO)_9$ (95%) and $PhCH_2CCo_3(CO)_9$ (5%). The methylene proton resonance in the NMR spectrum of the latter was observed at 4.80 ppm (in CCl_4), in agreement with the literature [16].

Cothermolysis of methylidynetricobalt nonacarbonyl-d and ethylidynetricobalt nonacarbonyl

The standard apparatus was charged with 1.00 g (2.26 mmol) of $DCCo_3(CO)_9$ and 1.00 g (2.20 mmol) of $CH_3CCo_3(CO)_9$ and 30 ml of xylenes. The reaction mixture was heated at reflux under nitrogen for 30 min. Subsequently, the solvent was removed at reduced pressure and the residue was sublimed (40°C at 0.03 Torr) for 48 h in order to insure that all material had sublimed. The 0.8 g of solid which was obtained was shown to be a mixture of $DCCo_3(CO)_9$, $CH_3CCo_3(CO)_9$, $CH_3CD_2CCo_3(CO)_9$, $CD_3CD_2CCo_3(CO)_9$ and $CH_3CHDCD_2 \cdot CCo_3(CO)_9$. The proton NMR spectrum of the sublimate was useful only in that it showed that the alkyl chains on the clusters had been extended and that there were methyl groups which no longer were attached directly to the Co_3C cluster. NMR ($CDCl_3$): δ 1.40 (s, broad, due to D coupling, $Co_3C(CD_2)_nCH_3$, 23%) and 3.63 ppm (s, Co_3CCH_3, 77%). The mass spectrum served in the identification of the products, but not in their quantitative assay. The following is not a total listing of all of the fragments ions which were observed, rather only the parent ions and those fragments ions due to loss of CO ligands are given. Mass spectrum: *m/e* 456, 428, 400, 372, 344, 316, 288, 260, 232, 204 ($CH_3CCo_3(CO)_9^+$ and successive loss of 9 CO to $CH_3CCo_3^+$); 442, 414, 386, 358, 330, 302, 274, 246, 218, 190 ($DCCo_3(CO)_9^+$ and successive loss of 9 CO to $DCCo_3^+$); 472 to 220 ($CH_3CD_2CCo_3(CO)_9^+$ and successive loss of 9 CO to $CH_3CD_2CCo_3^+$); 475 to 223 ($CD_3CD_2CCo_3(CO)_9^+$ and successive loss of 9 CO to $CD_3CD_2CCo_3^+$); 487 to 235 ($CH_3CHDCD_2CCo_3(CO)_9^+$ and successive loss of 9 CO to $CH_3CHDCD_2CCo_3^+$).

Thermolysis of benzylidynetricobalt nonacarbonyl in the presence of triethylsilane

A thick-walled Pyrex tube was charged with 2.00 g (3.86 mmol) of $PhCCo_3(CO)_9$ and 6.03 ml (38 mmol) of triethylsilane in 20 ml of benzene. The solution was degassed by several freeze-thaw cycles and then the tube was evacuated and sealed. Subsequently it was heated at 150°C for 20 h in a tube furnace. At the end of this time, a cobalt mirror had formed and the solution was pale yellow. GLC analysis of the volatiles (6 ft. Apiezon L, 100° C) showed the presence of two products: toluene (58% yield) and hexaethyldisilane. These were identified by comparison of their GLC retention times and proton NMR spectra with those of authentic samples.

Thermolysis of benzylidynetricobalt nonacarbonyl in the presence of cyclohexene

A thick-walled Pyrex tube was charged with 2.00 g (3.8 mmol) of $PhCCo_3(CO)_9$ and 10 ml of cyclohexene. The procedure described in the experiment above was followed. The tube was heated at 145° C for 20 h. At the end of this time, the solution still was quite deeply colored, so further heating at 200° C was applied for 5 h. Subsequently the reaction solution was filtered and the residue was washed with pentane until the washings were colorless. The filtrate and washings were trap-to-trap distilled at room temperature and 0.03 Torr. The volatiles were examined by GLC and shown to contain only cyclohexene. The residue from the distillation was chromatographed on a silicic acid column. Hexane eluted a single brown band from which 0.4 g (20%) of $PhCCo_3(CO)_9$ was recovered. Elution with dichloromethane removed a yellow oil. GLC analysis of the latter (6 ft. DC 710, 180° C) showed the presence of two components. The first was identified as dicyclohexyl ketone, n_D^{25} 1.4830 (lit. [17] n_D^{20} 1.4847), with NMR and IR spectra consistent with this assignment (ν (C=O) 1670 cm^{-1}; neat film). The second component, n_D^{25} 1.5294, was not identified conclusively, but on the basis of its spectra may be dicyclohexenyl ketone (NMR (CCl_4): δ 1.0—2.2 (m, broad, 14H, CH_2) and 7.2—8.0 ppm (M, 4H, =CH); IR (film) ν (C=O) 1670 cm^{-1}).

A similar reaction of $HCCo_3(CO)_9$ (130° C for 24 h) on a 1.00 g (2.26 mmol) scale gave 0.4 g of a mixture of $CH_3CCo_3(CO)_9$ (17%) and starting material (83%) as well as some dicyclohexyl ketone.

Acknowledgments

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