Table III. ^{95}Mo NMR Spectral Results for Nitrosyl Iodide Complexes

allyl	compd	δ	
allyl	**1c**	−1093	(endo)
1-Me	**2c**	−974	(endo)
2-Me	**5c**	−1042	(endo)
2-Cl	**6c**	−1021	(endo)
2-Br	**7c**	−1028	(endo)
cyclohexenyl	**8c**	−895	(exo)
cycloheptenyl	**9c**	−878	(exo)
cyclooctenyl	**10c**	−884	(exo)
1,1,2-Me_3	**11c**	−795	(exo)
		−791	(exo)

assign the ^{95}Mo resonance observed at −884 ppm for complex **10c** to its exo conformation. It is also most likely that the cyclohexenyl and cycloheptenyl complexes exist mainly in the exo conformation, and so the ^{95}Mo resonances at −895 and −878 ppm for these complexes are also assigned to the exo conformation.

It is apparent from these complexes that the general trend of the exo isomers exhibiting resonances upfield of the endo isomers is no longer followed. In fact, the trend seems to be reversed, i.e., exo isomers being downfield of the endo isomers which leads to the assignments shown in Table III.

Conclusion

We have shown that for the dicarbonyl complexes and the carbonyl nitrosyl cations the exo isomers of a given allyl ligand yield resonances upfield of their corresponding endo isomers. In addition, for the dicarbonyl complexes the exo conformations of nearly all the allyl ligands examined yield resonances upfield of the endo complexes as a whole.

In conclusion, we can see that the use of ^{95}Mo NMR may prove to be a valuable tool in the stereochemical analyses of closely related complexes.

Experimental Section

Complexes. The molybdenum dicarbonyl complexes $CpMo(CO)_2$(allyl) and nitrosyl carbonyl cations [CpMo(CO)(NO)(allyl)]PF_6 were prepared by published methods.[4,5] The nitrosyl iodide complexes[7] CpMo(NO)(I)(allyl) were prepared by adding 0.2 M solutions of NaI in acetone directly to the NMR samples of the corresponding nitrosyl cations and were not isolated.

Physical Measurements. The ^{95}Mo NMR spectra were obtained on a Bruker WM500 spectrometer using a multinuclear probe and operating at 32.59 MHz with a deuterium lock. The spectrometer was calibrated by using an external standard of 2 M Na_2MoO_4 in D_2O. Complex concentrations ranged from 0.1 to 0.5 M in acetone-d_6 or 1:1 $(CH_3)_2CO/(CD_3)_2CO$. Spectra of acceptable signal to noise could be obtained in 15 min. The shifts relative to Na_2MoO_4 were obtained indirectly by measuring the shifts relative to the lock on $(CD_3)_2CO$. The shifts were corrected for the field shift in locking on D_2O by subtracting 2.5 ppm. All measurements were conducted at 303 K.

Acknowledgment. We wish to thank the National Science Foundation for support of the NSF Northeast Regional NMR Facility (Grant CHE79-16210). The work was supported by NSF Grant CHE85-04516.

Synthesis and Characterization of $[Co_4(CO)_{11}C(O)Me]^-$. The X-ray Crystal Structure of Tetraphenylphosphonium Tris(μ-carbonyl)octacarbonylacetyl-*tetrahedro*-tetracobaltate(1−)

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Received August 26, 1985

The alkylidyne complex $Co_3(CO)_9COC(O)Me$ decomposes in THF to produce $[Co(CO)_4]^-$, $Co_4(CO)_{12}$, and an unidentified intermediate which reacts with $[Co(CO)_4]^-$ to produce $[Co_4(CO)_{11}C(O)Me]^-$ (I). Complex I is also produced by the reaction of $Co_4(CO)_{12}$ with MeLi and has been characterized by IR and NMR spectroscopy and by a single-crystal X-ray diffraction study, as a tetraphenylphosphonium salt. The salt crystallizes in the monoclinic space group $P2_1/c$, with a = 12.026 (1) Å, b = 23.835 (3) Å, c = 12.788 (2) Å, β = 90.492 (9)°, and Z = 4. The structure has been solved from 4949 reflections with $I > 3\sigma(I)$ and refined by least-squares calculations to R = 3.7%. The structure of the anion is derived from that of $Co_4(CO)_{12}$, with a mean cobalt–cobalt distance of 2.512 (3) Å. Three of the carbonyl groups are edge bridging, defining a basal triangle, while the remaining eight are terminal. The acetyl ligand is terminally bound in an axial position on the basal triangle, with a cobalt–carbon distance of 1.953 (4) Å.

Introduction

Recently in this laboratory several anionic ketenylidene cluster complexes have been synthesized via reductive cleavage of μ_3-C–O bonds. For example, reduction of the anions $[M_3(CO)_{10}COC(O)Me]^-$ (M = Fe, Ru, or Os) with Na/Ph_2CO affords the dianions $[M_3(CO)_9CCO]^{2-}$, containing face-capping ketenylidene ligands.[1,2] These complexes possess a rich and varied chemistry,[2-4] one aspect of which is metal fragment substitution reactions to form new mixed-metal ketenylidene complexes. For example, reaction of $[PPN]_2[Fe_3(CO)_9CCO]$ with $Co_2(CO)_8$ affords $[PPN][CoFe_2(CO)_9CCO]$, a monoanionic ketenylidene complex containing cobalt.[5] For many years the homo-

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nuclear $[Co_3(CO)_9CCO]^+$ cation has been known and its synthetic utility amply demonstrated.[6] We attempted to prepare $[Co_3(CO)_8CCO]^-$ by reduction of $Co_3(CO)_9COC(O)Me$.[7] The X-ray crystal structure of $Co_3(CO)_9COC(O)Me$ suggests that the μ_3-C–O bond (1.37 Å) is activated,[8] but reduction using either Na/Ph_2CO or Na/Hg amalgam in THF or Et_2O leads to $Na[Co(CO)_4]$ as the only stable product. It was observed, however, that $Co_3(CO)_9COC(O)Me$ decomposes rapidly in THF to give a mixture of $[Co(CO)_4]^-$, $Co_4(CO)_{12}$, and an unidentified intermediate which reacts with $[Co(CO)_4]^-$ to afford the new cluster anion $[Co_4(CO)_{11}C(O)Me]^-$ (I). In this paper the formation, characterization, and X-ray structure of I are described, as well as a more rational synthesis of I via treatment of $Co_4(CO)_{12}$ with MeLi.

Experimental Section

Materials and Methods. All manipulations were performed under an atmosphere of prepurified N_2 by using standard Schlenk techniques or in a Vacuum Atmospheres drybox. THF and Et_2O were dried over sodium benzophenone ketyl, pentane, hexane, and toluene over sodium and CH_2Cl_2 over P_2O_5. CD_2Cl_2 (99.6 + atom % D, Aldrich) was vacuum distilled from P_2O_5 prior to use. IR spectra were recorded on either a Perkin-Elmer 399 or 283 spectrophotometer using 0.1-mm path length CaF_2 solution cells or as Nujol mulls between KBr plates. ^1H NMR spectra were recorded on a JEOL FX-90Q specterometer operating at 89.55 MHz, and ^{13}C NMR spectra were recorded on a JEOL FX-270 spectrometer operating at 67.80 MHz. All chemical shifts are reported positive if downfield relative to Me_4Si, using residual solvents protons (^1H) or the solvent (^{13}C) as internal references. ^{13}C NMR were recorded with $Cr(acac)_3$ as a relaxation agent. $Co_2(CO)_8$ (Strem), Florisil (Aldrich), and low halide 1.2 M MeLi–diethyl ether solution (Aldrich) were used without further purification. $Co_3(CO)_9COC(O)Me$,[7] $[PPN][Co(CO)_4]$,[9] $Co_4(CO)_{12}$,[10] and $Li[Co_3(CO)_{11}]$[11] were prepared by literature procedures, and $[PPN][BF_4]$ was prepared by metathesis of [PPN]Cl and $Na[BF_4]$.

Preparation of ^{13}C-Enriched $Co_3(^*CO)_9{}^*COC(O)Me$. A 100-mL gas bulb was charged with $Li[Co_3(CO)_{10}]$[11] (0.400 g, 0.86 mmol) and Et_2O (25 mL). The resulting solution was degassed and stirred under 100 torr of ^{13}CO for 24 h. The solvent was then removed under vacuum and the residue dissolved in toluene (20 mL) before addition of MeCOCl (0.1 mL, 1.4 mmol). After the solution was stirred for 16 h, the toluene was removed under vacuum. The residue was dissolved in CH_2Cl_2 (ca. 10 mL) and adsorbed onto Florisil (ca. 1 g) which was transferred onto a Florisil chromatography column (ca. 1.5 × 15 cm). Elution with hexane produced a brown followed by a purple band. The first band was identified as $Co_4(CO)_{12}$, while the second was $Co_3(CO)_9COC(O)Me$. Collection of the second band and removal of the hexane under vacuum afforded microcrystals of $Co_3(CO)_9COC(O)Me$ (0.200 g, 0.40 mmol) in 46% yield, ^{13}C enriched to ca. 20% as estimated from the mass spectrum. ^{13}C NMR (CD_2Cl_2/CH_2Cl_2, −60 °C): 281.0 (s), 199.1 (s) ppm (ca. intensities 1:9).

$[PPN][Co_4(CO)_{11}C(O)Me]$ (I). (i) A solution of $Co_3(CO)_9COC(O)Me$ (0.175 g, 0.35 mmol) and [PPN] $[Co(CO)_4]$ (0.250 g, 0.35 mmol) in THF (10 mL) was stirred for 6 h, after which time the solution IR showed the presence of $Co_4(CO)_{12}$, $[Co(CO)_4]^-$, and $[Co_4(CO)_{11}C(O)Me]^-$. The solvent was removed under vacuum and the residue washed with pentane (3 × 20 mL), before being extracted into Et_2O (60 mL) and filtered through a medium-porosity frit. Reduction of the Et_2O under vacuum to ca. 20 mL in volume and precipitation by addition of pentane (60 mL) afforded $[PPN][Co_4(Co)_{11}C(O)Me]$ (I) as a purple powder (0.150 g, 0.13 mmols) in 37% yield.

Table I. Crystal Structure Data for $[PPh_4][Co_4(CO)_{11}C(O)Me]$

formula	$C_{37}H_{23}Co_4O_{12}P$
mol wt	926.3
cryst color habit	black lozenge
approx cryst dimens, mm	0.18 × 0.42 × 0.20
space group	$P2_1/c$
a, Å	12.026 (1)
b, Å	23.835 (3)
c, Å	12.788 (2)
β, deg	90.492 (9)
V, Å^3	3665 (1)
$F(000)$	1856
Z	4
D(calcd), g cm^{-3}	1.679
λ(Mo Kα), Å	0.71073
monochromator	graphite
2θ range, deg	3–55
scan mode	ω–2θ
scan width	$\theta = 0.7 + 0.35$ tan + 50% extension either side for backgrounds
scan speed range, deg min^{-1}	0.63–5.49
octants measd	$\pm h,+k,+l$
data $I > 3\sigma(I)$	4949
final no. of variables	487
abs correctn	cryst faced; bounding planes {100}, {011}
trans coeff	0.62–0.83
T, K	163 (2)
R, R_w	3.7, 4.6%
weighting scheme	$w = 4F_o^2/[\sigma^2(F_o^2) + (0.04F_o^2)^2]$
est observn of unit wt	1.28

(ii) A solution of $Co_4(CO)_{12}$ (0.200 g, 0.350 mmol) in Et_2O (30 mL) was treated with a 1.2 M Et_2O solution of MeLi (0.3 mL, 0.36 mmol). The solution was stirred for 5 min and filtered and then the solvent removed under vacuum. The residue was dissolved in CH_2Cl_2 (20 mL) containing $[PPN][BF_4]$ (0.220 g, 0.352 mmol) and stirred for 10 min. Removal of the solvent under vacuum and workup as described in (i) afforded I (0.080 g, 0.071 mmol) in 20% yield: IR ν_{CO} (THF) 2057 (w), 2010 (vs), 1986 (s), 1818 (m), 1665 (w)cm^{-1}; IR ν_{CO} (Nujol) 2055 (w), 2010 (s), 1992 (s), 1980 (m), 1952 (m), 1812 (m), 1804 (m), 1655 (w) cm^{-1}; ^1H NMR (CD_2Cl_2, +16 °C) 2.39 (s) ppm; ^{13}C NMR (CD_2Cl_2/CH_2Cl_2, +30 °C) 239.5 (s), 215.2 (s) ppm (ca. intensities 1:11).

Attempted Methylation of $[CO_4(CO)_{11}C(O)Me]^-$. Both the Li^+ and PPN^+ salts of I react with excess $MeOSO_2CF_3$ in CH_2Cl_2 over a period of several hours to give a mixture of neutral products, one of which was identified by IR and mass spectral analysis as $Co_4(CO)_{12}$. The other products were thermally unstable even in the solid state and were not characterized.

X-ray Crystal Structure of $[PPh_4][Co_4(CO)_{11}C(O)Me]$. A summary of the procedures used in data collection, structure solution and refinement is presented in Table I.[12,13] Crystals of $[PPh_4][Co_4(CO)_{11}C(O)Me]$, prepared by method i using $[PPh_4][Co(CO)_4]$, were grown by slow diffusion of an Et_2O/pentane mixture (2:1) into a concentrated THF solution of the complex at 5 °C. A suitable crystal was mounted in air on a glass fiber and and transferred to the cold stream of an Enraf-Nonius CAD4 diffractometer. A total of 22 reflections (21° < 2θ < 29°) were used to align the crystal, and a least-squares refinement of their diffraction geometry showed the crystal to belong to a monoclinic system. Systematic absences indicated the space group to be $P2_1/c$. Data were collected by using a variable scan rate, ω–2θ scan mode, to a maximum 2θ value of 55°. The intensities of three standard reflections were remeasured after every 97 reflections and were observed to vary by less than 0.6%. Data were corrected for Lorentz, polarization, background, and adsorption effects, but not for extinction. The four cobalt atoms and the phosphorous atom were located by direct methods and

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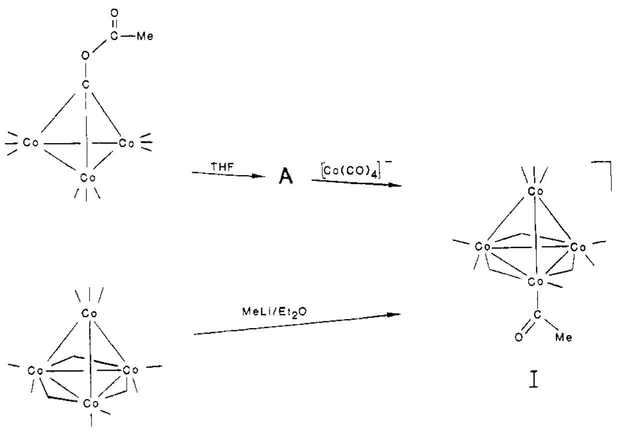

Figure 1. Methods of synthesis of the anion [Co$_4$(CO)$_{11}$C(O)Me]$^-$ (I) (A = uncharacterized intermediate).

the remaining non-hydrogen atoms by successive difference syntheses. Aromatic hydrogen atoms were included as fixed contributions after idealization (r_{C-H} = 1.00 Å; *B*'s 1 Å^2 greater than the B_{equiv} of the attached carbon atoms). The hydrogen atoms of the methyl group were not included in the refinement. Refinement of positional and anisotropic thermal parameters for all non-hydrogen atoms was carried out to convergence (*R* = 3.7%, R_w = 4.6%). During the final cycle of refinement, no parameter shifted by more than 0.01 σ_p where σ_p is the estimated standard deviation of that parameter. A final difference Fourier synthesis showed no peaks greater than 1.0 e/Å^3, the largest peaks being located in the vicinity of the cobalt tetrahedron.

Results and Discussion

Syntheses. The complex Co$_3$(CO)$_9$COC(O)Me can be prepared in high yield by the reaction of Li[Co$_3$(CO)$_{10}$] with MeCOBr in benzene, followed by recrystallization from pentane.[7] Although Co$_3$(CO)$_9$COC(O)Me apparently is stable in hydrocarbon solvents, Et$_2$O and CH$_2$Cl$_2$, we were surprised to observe that it decomposes rapidly (ca. $^1/_2$ h) in THF at room temperature to afford Co$_4$(CO)$_{12}$, [Co(THF)$_6$][Co(CO)$_4$]$_2$, and a third product which we were unable to isolate. The IR spectrum of this unidentified complex has similar terminal CO stretching bands to Co$_3$(CO)$_9$COC(O)Me,[7] although exact assignments were made difficult by overlap with Co$_4$(CO)$_{12}$ adsorptions. More noticeable, however, is a shift of the band assigned to the acetate C=O in Co$_3$(CO)$_9$COC(O)Me at 1775–1712 cm^{-1}. The reaction of equimolar quantities of Co$_3$(CO)$_9$COC(O)Me and [PPN][Co(CO)$_4$] in THF results first in decomposition of the alkylidyne complex followed by slow reaction with [PPN][Co(CO)$_4$] to produce a new anionic cluster (see Figure 1). Over the time of the reaction, ca. 6 h, the adsorption at 1712 cm^{-1}, which is characteristic of the unidentified complex, disappears and is replaced by a new C=O adsorption at 1665 cm^{-1}. The final product can be isolated by removing the THF under vacuum, extracting with Et$_2$O to separate most of the excess [PPN][Co(CO)$_4$], and precipitating with pentane to form a microcrystalline purple powder while the Co$_4$(CO)$_{12}$ remains in solution. The product is formulated as [PPN][Co$_4$(CO)$_{11}$C(O)Me] (I) on the basis of IR, NMR, and X-ray crystallographic data and can be isolated in ca. 37% yield. Stirring Co$_3$(CO)$_9$COC(O)Me in THF for prolonged periods of time (ca. 2 days) affords trace amounts of [Co(THF)$_6$][Co$_4$(CO)$_{11}$C(O)Me]$_2$ since some [Co(THF)$_6$][Co(CO)$_4$]$_2$ is formed in the initial decomposition of Co$_3$(CO)$_9$COC(O)Me.

IR spectra of I consistently revealed contamination with [Co(CO)$_4$]$^-$, which upon careful crystallization could be seen as separate pale green crystals of [PPN][Co(CO)$_4$]. Spectra recorded by dissolving a small number of purple crystals were clean. Similar problems with the purification of other [Co$_4$(CO)$_{11}$X]$^-$ derivatives have been noted previously,[14] and a satisfactory analysis for the bulk material was not obtained. Separation of the PPh$_4^+$ salts of I and [Co(CO)$_4$]$^-$ was found to be more difficult and impossible when the NMe$_3$Bz$^+$ cation was employed due to the low solubility of [NMe$_3$Bz][Co$_4$(CO)$_{11}$C(O)Me] in Et$_2$O.

Compound I can be prepared more directly by methylation of Co$_4$(CO)$_{12}$ with MeLi, followed by metathesis with [PPN][BF$_4$) in an analogous manner to the synthesis of [PPN][Co$_4$(CO)$_{11}$C(O)OMe].[14]

Spectroscopy. The IR spectrum (THF) of I has three terminal CO stretching adsorptions at 2057 (w), 2010 (vs), and 1986 (s) cm^{-1}, a bridging CO band at 1818 (m) cm^{-1}, and a band due to the acetyl ligand at 1665 (w) cm^{-1}. The spectrum is similar in appearance to that reported for [PPN][Co$_4$(CO)$_{11}$C(O)OMe],[14] 2070 (w), 2020 (s), 2000 (ms), 1980 (sh), 1830 (m), and 1640 (w) cm^{-1}; a lowering of the CO stretching frequencies (ca. 10 cm^{-1}) apparently is caused by the greater electron-donating ability of the C(O)Me vs. the C(O)OMe ligand. Similarity of IR spectra and color between the C(O)Me and C(O)OMe derivatives suggests that they have the same structure. In the case of the anions [Co$_4$(CO)$_{11}$C(O)R]$^-$ (R = OMe, O-*i*-Pr, and NH-*n*-Pr) it has been postulated that the C(O)R substituent is coordinated to a basal cobalt.[14] The only X-ray crystallographic study of a four metal [Co$_4$(CO)$_{11}$X]$^-$ derivative reported is that of the yellow salt [NEt$_4$][Co$_4$(CO)$_{11}$I], which reveals the iodine to be attached to an apical cobalt although a basal isomer was also detected in the crystal lattice.[15] The anion [Ge{Co$_7$(CO)$_{20}$}]$^-$ is a derivative is a derivative of Co$_4$(CO)$_{12}$ with the axial carbonyl ligand of one basal cobalt replaced by a GeCo$_3$(CO)$_9$ fragment, although accurate structural details were not obtained due to disorder of the Co$_4$ tetrahedron.[16] The solid-state structure of I as the PPh$_4^+$ salt has been unambiguous determined by an X-ray crystallographic study (see below) which shows the acetyl group to be located in an axial position of the basal cobalt plane.

The ^{1}H NMR spectrum of I contains a signal assigned to the acetyl methyl group at δ 2.39. ^{13}C NMR spectra were recorded by using samples prepared from ^{13}C-enriched (ca. 20%) Co$_3$(CO)$_9$COC(O)Me. This was prepared by stirring an Et$_2$O solution of Li[Co$_3$(CO)$_{10}$][11] under a ^{13}CO atmosphere for 24 h before reaction with MeC(O)Cl to afford Co$_3$(CO)$_9$COC(O)Me.[7] The degree of enrichment could be conveniently checked by mass spectroscopy, and the ^{13}C NMR, not previously reported, contains resonances due to the μ_3-C at 281.0 ppm and nine equivalent CO ligands at 199.1 ppm. Enrichment of the μ_3-C is not obtained when complexes of the form Co$_3$(CO)$_9$CY are stirred under a ^{13}CO atmosphere.[17] At +30 °C the ^{13}C NMR spectrum of I contains a resonance of 239.5 ppm assigned to acetyl carbon *C*(O)Me and a resonance at 215.2 ppm due to the carbonyl ligands in an approximate ratio of 1:11. The acetyl resonance is in the usual range (δ 280–200) found for terminal acetyl metal complexes.[18] The carbonyl ligands are evidently more fluxional than those in the neutral Co$_4$(CO)$_{11}$L (L = CO or POMe)$_3$) derivatives,[19] and

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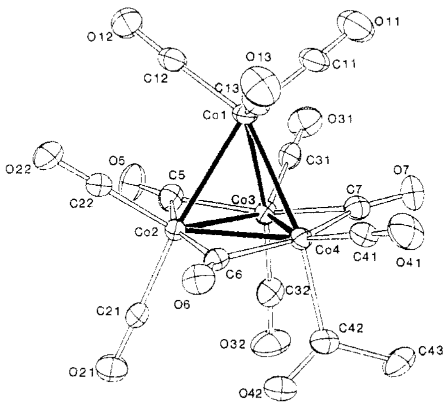

Figure 2. ORTEP drawing and atomic numbering scheme of $[Co_4(CO)_{11}C(O)Me]^-$ with thermal ellipsoids drawn at the 50% probability level.

even at −90 °C, the lowest temperature at which the ^{13}C NMR was recorded, the CO signal remains a singlet, although considerably broadened, while the *C*(O)Me signal remains sharp. It is of interest that the acetyl carbon is enriched to approximately the same extent as the carbonyl ligands suggesting that either migration of the Me group to the μ_3-C of $Co_3(CO)_9COC(O)Me$ has occurred during the formation of complex I or reversible methyl migration scrambles the carbonyl ligands once complex I has been formed. In contrast to the formation of I, treatment of $Co_3(CO)_9CCl$ with $Na[Co(CO)_4]$ has been shown to afford $Na_2[Co_6C(CO)_{15}]$ which in turn reacts with $Co_4(CO)_{12}$ to produce $Na_2[Co_8C(CO)_{18}]$ and $Na[Co_6C(CO)_{14}]$.[20] No evidence for the formation of higher nuclearity clusters was found in this system, however.

X-ray Structure of $[Ph_4P][Co_4(CO)_{11}C(O)Me]$. This compound crystallizes in the monoclinic space group $P2_1/c$ with four discrete $[Co_4(CO)_{11}C(O)Me]^-$ anions and Ph_4P^+ cations per unit cell. The structure of the anion $[Co_4(CO)_{11}C(O)Me]^-$ is shown in Figure 2. Final positional parameters are given in Table II, and derived distances and angles are listed in Table III.

The anion contains a central tetrahedron of cobalt atoms bridged on three edges by carbonyl ligands with the acetyl group occupying an axial site on the basal plane. The basal–basal, cobalt–cobalt bond distances are very similar ranging from 2.454 (1) to 2.469 (1) Å while the apical–basal cobalt–cobalt bonds have a greater range, the distance Co(1)–Co(4) [2.616 (1) Å] being significantly longer than the bonds from the apical cobalt to the unsubstituted basal cobalt atoms Co(2) [2.533 (1) Å] and Co(3) [2.537 (1) Å]. A similar asymmetry is observed in the anion $[Ir(CO)_{11}C(O)OMe]^-$.[21] The mean cobalt–cobalt distance [2.512 (3) Å] is comparable with the average distances in $Co_4(CO)_{11}P(C_6H_5)_3$ (2.506 Å),[22] $Co_4(CO)_{11}PMe_3$ (2.500 Å),[23] $CO_4(CO)_{12}$ (2.492 Å),[24] and $[Co_4(CO)_{11}I]^-$ [2.493 (16) Å],[14] the only other anionic tetrahedral cobalt cluster for which an accurate structure has been determined.

The mean Co–C(term) distance is 1.779 (5) Å and is intermediate in length between that found in $Co_4(CO)_{11}P(C_6H_5)_3$ (1.797 Å)[22] and $[Co_4(CO)_{11}I]^-$ [1.755 (7) Å].[15] The shortest Co–C(term) bond length is Co(4)–C(41) [1.749 (4) Å], and this reflects the higher charge density on Co(4) caused by the presence of the acetyl substituent. The effect of the acetyl ligand also is observed on the bridging carbonyl groups. One of the carbonyl ligands which bridges to the acetyl bearing cobalt, Co(4), is markedly asymmetric [C(7)–Co(4) = 1.879 (4), C(7)–Co(3) = 1.988 (4) Å], while this asymmetry is not present in the other carbonyl bridging to Co(4) [C(6)–Co(4) = 1.943 (4), C(6)–Co(2) = 1.952 (4) Å]. The third bridging carbonyl also shows slight asymmetry [C(5)–Co(3) = 1.924 (4), C(5)–Co(2) = 1.962 (4) Å]. C–O bond distances, including both terminal and bridging carbonyls, range from 1.128 (5) to 1.164 (4) Å.

Table II. Positional Parameters and Their Estimated Standard Deviations for $[Co_4(CO)_{11}C(O)Me]^-$

atom	*x*	*y*	*z*
Co(1)	0.37119 (4)	0.12482 (2)	0.79207 (5)
Co(2)	0.24187 (4)	0.17923 (2)	0.90843 (5)
Co(3)	0.19011 (4)	0.14972 (2)	0.71984 (5)
Co(4)	0.19991 (4)	0.08001 (2)	0.86971 (4)
O(5)	0.2619 (3)	0.2658 (1)	0.7362 (3)
O(6)	0.2382 (3)	0.1062 (1)	1.1048 (3)
O(7)	0.1793 (3)	0.0313 (1)	0.6492 (2)
O(11)	0.4299 (3)	0.0617 (2)	0.5937 (3)
O(12)	0.5261 (3)	0.2150 (2)	0.7821 (3)
O(13)	0.4608 (3)	0.0547 (2)	0.9662 (3)
O(21)	0.0672 (3)	0.2401 (2)	1.0055 (4)
O(22)	0.4102 (3)	0.2372 (2)	1.0267 (3)
O(31)	0.2555 (3)	0.1575 (2)	0.4884 (3)
O(32)	−0.0313 (3)	0.1801 (2)	0.7073 (3)
O(41)	0.2311 (3)	−0.0324 (1)	0.9533 (3)
O(42)	0.0115 (3)	0.1007 (1)	0.9732 (3)
C(5)	0.2434 (4)	0.2212 (2)	0.7681 (4)
C(6)	0.2302 (3)	0.1162 (2)	1.0111 (3)
C(7)	0.1861 (3)	0.0664 (2)	0.7164 (3)
C(11)	0.4066 (4)	0.0855 (2)	0.6707 (4)
C(12)	0.4632 (4)	0.1814 (2)	0.7832 (4)
C(13)	0.4197 (4)	0.0812 (2)	0.8998 (4)
C(21)	0.1338 (3)	0.2159 (2)	0.9674 (4)
C(22)	0.3445 (3)	0.2142 (2)	0.9806 (4)
C(31)	0.2302 (4)	0.1548 (2)	0.5784 (4)
C(32)	0.0550 (4)	0.1672 (2)	0.7108 (4)
C(41)	0.2186 (3)	0.0116 (2)	0.9189 (4)
C(42)	0.0513 (3)	0.0726 (2)	0.9026 (4)
C(43)	−0.0137 (4)	0.0296 (2)	0.8370 (5)

The acetyl group is in an axial position, approximately trans to the apical cobalt. The non-hydrogen portion of the $-C(O)CH_3$ group is planar within experimental error. The distance Co(4)–C(42) [1.953 (4) Å] is comparable with the cobalt–carbon bond distance of 1.90 (2) Å found in the acyl derivative $Co_2\{\mu-(\eta-C_5H_4CH_2CO)\}\{\mu-(\eta-C_5H_4CH_2)\}(CO)_2$.[25] The C(42)–O(42) distance [1.197 (5) Å] is similar to the carbon–oxygen distances found in acyl anions $[Rh_6(CO)_{15}C(O)Et]^-$ [1.19 (2) Å],[26] $[Mn(CO)_4\{C(O)Me\}\{C(O)Ph\}]^-$ [1.20 (1) and 1.22 (1) Å],[27] and $[Fe(CO)_3P(OMe)_3C(O)Me]^-$ [1.226 (5) Å][28] and indicates a carbon–oxygen double bond. The distance C(42)–C(43) [1.533 (6) Å] is as expected for a carbon–carbon single bond, and the angle C(43)–C(42)–O(42) [120.4 (4)°] is also reasonable.

Summary

This paper describes two synthetic routes to the anion I which is considerably more stable than the previously reported tetranuclear cobalt anions $[Co_4(CO)_{11}C(O)R]^-$ (R = OMe, O-*i*-Pr, or NH-*n*-Pr). The structure of these an-

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Table III. Bond Distances (Å) and Angles (deg) for $[PPh_4][Co_4(CO)_{11}C(O)Me]^-$[a]

Bond Lengths

atom 1	atom 2	dist	atom 1	atom 2	dist
Co(1)	Co(2)	2.533 (1)	Co(4)	C(6)	1.943 (4)
Co(1)	Co(3)	2.537 (1)	Co(4)	C(7)	1.879 (4)
Co(1)	Co(4)	2.616 (1)	Co(4)	C(41)	1.749 (4)
Co(1)	C(11)	1.795 (4)	Co(4)	C(42)	1.953 (4)
Co(1)	C(12)	1.793 (5)	O(5)	C(5)	1.154 (4)
Co(1)	C(13)	1.770 (4)	O(6)	C(6)	1.155 (4)
Co(2)	Co(3)	2.460 (1)	O(7)	C(7)	1.164 (4)
Co(2)	Co(4)	2.469 (1)	O(11)	C(11)	1.129 (4)
Co(2)	C(5)	1.962 (4)	O(12)	C(12)	1.136 (5)
Co(2)	C(6)	1.952 (4)	O(13)	C(13)	1.141 (5)
Co(2)	C(21)	1.788 (4)	O(21)	C(21)	1.128 (5)
Co(2)	C(22)	1.775 (4)	O(22)	C(22)	1.143 (5)
Co(3)	Co(4)	2.454 (1)	O(31)	C(31)	1.134 (5)
Co(3)	C(5)	1.924 (4)	O(32)	C(32)	1.147 (5)
Co(3)	C(7)	1.988 (4)	O(41)	C(41)	1.139 (5)
Co(3)	C(31)	1.785 (4)	O(42)	C(42)	1.197 (5)
Co(3)	C(32)	1.779 (5)	C(42)	C(43)	1.533 (6)

Bond Angles

atom 1	atom 2	atom 3	angle	atom 1	atom 2	atom 3	angle
Co(2)	Co(1)	Co(3)	58.06 (1)	C(5)	Co(3)	C(32)	98.7 (2)
Co(2)	Co(1)	Co(4)	57.27 (1)	C(7)	Co(3)	C(31)	93.2 (2)
Co(2)	Co(1)	C(11)	152.5 (1)	C(7)	Co(3)	C(32)	102.0 (2)
Co(2)	Co(1)	C(12)	94.6 (1)	C(31)	Co(3)	C(32)	102.2 (2)
Co(2)	Co(1)	C(13)	97.1 (1)	Co(1)	Co(4)	Co(2)	59.66 (1)
Co(3)	Co(1)	Co(4)	56.85 (1)	Co(1)	Co(4)	Co(3)	59.94 (1)
Co(3)	Co(1)	C(11)	94.6 (1)	Co(1)	Co(4)	C(6)	88.3 (1)
Co(3)	Co(1)	C(12)	113.7 (1)	Co(1)	Co(4)	C(7)	78.0 (1)
Co(3)	Co(1)	C(13)	134.6 (1)	Co(1)	Co(4)	C(41)	112.9 (1)
Co(4)	Co(1)	C(11)	107.2 (1)	Co(1)	Co(4)	C(42)	158.2 (1)
Co(4)	Co(1)	C(12)	151.6 (1)	Co(2)	Co(4)	Co(3)	59.96 (1)
Co(4)	Co(1)	C(13)	77.8 (1)	Co(2)	Co(4)	C(6)	50.8 (1)
C(11)	Co(1)	C(12)	100.0 (2)	Co(2)	Co(4)	C(7)	111.7 (1)
C(11)	Co(1)	C(13)	101.5 (2)	Co(2)	Co(4)	C(41)	143.1 (1)
C(12)	Co(1)	C(13)	105.0 (2)	Co(2)	Co(4)	C(42)	105.0 (1)
Co(1)	Co(2)	Co(3)	61.05 (1)	Co(3)	Co(4)	C(6)	110.6 (1)
Co(1)	Co(2)	Co(4)	63.07 (1)	Co(3)	Co(4)	C(7)	52.6 (1)
Co(1)	Co(2)	C(5)	77.0 (1)	Co(3)	Co(4)	C(41)	152.3 (1)
Co(1)	Co(2)	C(6)	90.5 (1)	Co(3)	Co(4)	C(42)	99.6 (1)
Co(1)	Co(2)	C(21)	168.7 (1)	C(6)	Co(4)	C(7)	162.2 (2)
Co(1)	Co(2)	C(22)	91.5 (1)	C(6)	Co(4)	C(41)	95.3 (2)
Co(3)	Co(2)	Co(4)	59.72 (1)	C(6)	Co(4)	C(42)	92.9 (2)
Co(3)	Co(2)	C(5)	50.0 (1)	C(7)	Co(4)	C(41)	100.5 (2)
Co(3)	Co(2)	C(6)	110.0 (1)	C(7)	Co(4)	C(42)	95.7 (2)
Co(3)	Co(2)	C(21)	107.6 (1)	C(41)	Co(4)	C(42)	88.7 (2)
Co(3)	Co(2)	C(22)	141.0 (1)	C(42)	O(42)	C(43)	33.8 (2)
Co(4)	Co(2)	C(5)	109.3 (1)	Co(2)	C(5)	Co(3)	78.5 (1)
Co(4)	Co(2)	C(6)	50.5 (1)	Co(2)	C(5)	O(5)	139.4 (3)
Co(4)	Co(2)	C(21)	112.1 (1)	Co(3)	C(5)	O(5)	142.0 (3)
Co(4)	Co(2)	C(22)	134.4 (1)	Co(2)	C(6)	Co(4)	78.7 (1)
C(5)	Co(2)	C(6)	159.8 (2)	Co(2)	C(6)	O(6)	140.2 (3)
C(5)	Co(2)	C(21)	96.0 (2)	Co(4)	C(6)	O(6)	141.1 (3)
C(5)	Co(2)	C(22)	99.7 (2)	Co(3)	C(7)	Co(4)	78.7 (2)
C(6)	Co(2)	C(21)	93.6 (2)	Co(3)	C(7)	O(7)	137.1 (3)
C(6)	Co(2)	C(22)	96.4 (2)	Co(4)	C(7)	O(7)	144.1 (3)
C(21)	Co(2)	C(22)	98.5 (2)	Co(1)	C(11)	O(11)	178.7 (4)
Co(1)	Co(3)	Co(2)	60.89 (1)	Co(1)	C(12)	O(12)	175.2 (4)
Co(1)	Co(3)	Co(4)	63.21 (1)	Co(1)	C(13)	O(13)	173.1 (4)
Co(1)	Co(3)	C(5)	77.5 (1)	Co(2)	C(21)	O(21)	178.3 (4)
Co(1)	Co(3)	C(7)	78.3 (1)	Co(2)	C(22)	O(22)	179.3 (4)
Co(1)	Co(3)	C(31)	94.2 (1)	Co(3)	C(31)	O(31)	179.3 (4)
Co(1)	Co(3)	C(32)	163.5 (1)	Co(3)	C(32)	O(32)	177.6 (4)
Co(2)	Co(3)	Co(4)	60.31 (1)	Co(4)	C(41)	O(41)	178.4 (4)
Co(2)	Co(3)	C(5)	51.4 (1)	Co(4)	C(41)	C(42)	48.9 (1)
Co(2)	Co(3)	C(7)	108.2 (1)	O(41)	C(41)	C(42)	131.1 (3)
Co(2)	Co(3)	C(31)	141.4 (1)	Co(4)	C(42)	O(42)	120.9 (3)
Co(2)	Co(3)	C(32)	104.1 (1)	Co(4)	C(42)	C(41)	42.4 (1)
Co(4)	Co(3)	C(5)	111.3 (1)	Co(4)	C(42)	C(43)	118.7 (3)
Co(4)	Co(3)	C(7)	48.7 (1)	O(42)	C(42)	C(41)	128.0 (3)
Co(4)	Co(3)	C(31)	137.1 (1)	O(42)	C(42)	C(43)	120.4 (4)
Co(4)	Co(3)	C(32)	104.3 (1)	C(41)	C(42)	C(43)	96.2 (3)
C(5)	Co(3)	C(7)	154.3 (2)	O(42)	C(43)	C(42)	25.8 (2)
C(5)	Co(3)	C(31)	97.1 (2)				

[a] Numbers in parentheses are estimated standard deviations in the least significant digits.

ions, which previously was inferred by comparison with the corresponding rhodium and iridium derivatives, has been confirmed by a single-crystal X-ray diffaction study. The acetyl, which occupies an axial site, is terminally bonded to one cobalt atom unlike many polynuclear acyl complexes containing transition metals from earlier in the periodic table where coordination of the acyl oxygen to adjacent metal centers is often observed.[29] Despite the apparent stability of I toward CO loss and formation of a cluster containing a μ-O-CMe group,[30] reaction with $MeOSO_2CF_3$ in CH_2Cl_2, in an attempt to form a tetranuclear Fischer-type carbene complex, is slow and results in the formation of $Co_4(CO)_{12}$ as the only stable product. Other products could be detected in the solution IR of the reaction mixture but appeared to be thermally unstable and were not characterized.

Acknowledgment. This research was supported by the NSF. M.J.W. is a recipient of a NATO/SERC postdoctoral fellowship. Joseph A. Hriljac is thanked for helpful discussions.

Registry No. I, 100682-41-5; $Li[CO_4(CO)_{11}C(O)Me]$, 100837-21-6; $CO_3(*CO)_9*COC(O)Me$, 100682-39-1; $Li[Co_3(CO)_{10}]$, 26248-45-3; MeCOCl, 75-36-5; $Co_3(CO)_9COC(O)Me$, 13682-05-8; $[PPN][Co(CO)_4]$, 53433-12-8; $Co_4(CO)_{12}$, 17786-31-1; Co, 7440-48-4.

Supplementary Material Available: Complete listings of positional parameters, bond lengths, anisotropic thermally parameters, and observed and calculated structure factors for $[PPh_4][Co_4(CO)_{11}C(O)Me]$ and bond angles for $[Co_4(CO)_{11}C(O)Me]^-$ (65 pages). Ordering information is given on any current masthead page.

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Indenylmetallacarboranes. 1. The 18-Valence-Electron Complex 3-(η^5-C_9H_7)-3,1,2-$CoC_2B_9H_{11}$ and Comparative Molecular Structures of This Complex and 3-(η^5-C_5H_5)-3,1,2-$CoC_2B_9H_{11}$

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Received August 14, 1985

The molecular structures of 3-(η^5-C_9H_7)-3,1,2-$CoC_2B_9H_{11}$ (**1**) (two crystalline forms) and 3-(η^5-C_5H_5)-3,1,2-$CoC_2B_9H_{11}$ (**2**) have been determined in the solid state. The conformation of the former is cisoid, and the η^5-bonded indenyl ligand is slipped such that the junction carbons are ca. 0.07 Å further from the metal atom. Both the conformation and the direction of slipping have been rationalized by the results of molecular orbital calculations at the extended Hückel level. **1** undergoes two reversible 1e reductions at $E_{1/2} = -0.8$ and -1.82 V. Crystals of **1α** are monoclinic, $C2/c$, with $a = 23.882$ (4) Å, $b = 6.8415$ (24) Å, and $c = 22.824$ (6) Å, $\beta = 130.926$ (16)°, and $Z = 8$. With use of 4111 data collected at 185 K, the structure has been refined to 0.0273. The orthorhombic form **1β** crystallizes in $P2_12_12_1$ with $a = 7.1142$ (6) Å, $b = 10.7926$ (15) Å, and $c = 19.1767$ (21) Å and $Z = 4$. A total of 1012 data collected at 291 K have been refined to 0.0393. For **2** the cell is also orthorhombic, $P2_12_12$, with $a = 15.8260$ (24) Å, $b = 10.2226$ (22) Å, and $c = 7.4928$ (19) Å and $Z = 4$. A total of 2948 low-temperature data have afforded a final R index of 0.0475.

Introduction

The slipping distortion in metallacarboranes has been a subject of considerable interest over the last few years. Structural and theoretical studies have identified two classes of metallacarborane that display the distortion, those of the type[1] $L_2M(C_2B_xH_{2+x})$ where L_2M is an angular 14e fragment and those of the type[1d,2] $M(C_2B_xH_{2+x})_2^{n-}$, i.e., symmetrical bis(carborane) complexes in which the electronic configuration of the metal atom is d^8 or greater.

The electronically induced translational slip of a metal fragment across an η-bonded ligand face is not, however, restricted to metallacarboranes. Notably it has also been observed in a number of structural studies of η^5-indenyl complexes,[3,4] and, moreover, facile η^5-η^3 slipping of the indenyl ligand has been suggested[4] to be the process re-

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