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Structure of $(PhC{\equiv}CH)_3Co_2(CO)_6$: A New $Co_2(CO)_8$ Catalyzed Oligomerization Sequence of an Alkyne and Carbon Monoxide

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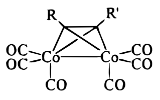
Hübel's tris(ethynylbenzene)hexacarbonyldicobalt complex, $(PhC{\equiv}CH)_3Co_2(CO)_6$, a byproduct in cobaltacyclopentadiene formation, has been characterized by spectroscopic methods and a single-crystal X-ray diffraction analysis. The organic ligand, derived from the ester [(1,2,3,4,5-η)-2,5-diphenylcyclopenta-2,4-dienyl][(2-η)(3-η:3-η)]-2*H*-3-phenylpropynoate, forms a η^1:η^2-bridge between the two cobalt atoms linked by a "semibridging" carbonyl.

Introduction

Alkyne−metal chemistry was initiated by the pioneering work of Reppe[1] in the 1940s, but it was the comprehensive studies by Hübel et al.[2] in the 1960s that revealed the structural diversity available from alkyne oligomerization at metal centers. For example, Hübel describes the isolation of more than 10 types of cobalt complexes and 20 types of iron complexes arising from simple reactions between alkynes and the metal carbonyls. Although most of the complexes possess the empirical formulas $(alkyne)_xM_y(CO)_z$, subsequent X-ray structural studies have revealed examples where CO groups have been incorporated into carbon skeletons as anhydride, ether, ketone, lactone, or quinone groups. Several complexes of each metal still remain to be identified unambiguously including, surprisingly, one readily accessible[3] cobalt complex, **1**, which has apparently not been reinvestigated, although it was highlighted in a subsequent review.[2]

Most alkynes react with $Co_2(CO)_8$ at room temperature to give the well-known dicobaltatetrahedranes $(RC{\equiv}CR^1)Co_2(CO)_6$, **2**. At temperatures around 100 °C these complexes react further with excess alkyne, causing cyclotrimerization to benzene derivatives,[4,5] sometimes linear trimerization to give the so-called "fly over" complexes $(RC{\equiv}CR^1)_3Co_2(CO)_4$,[3,6,7] **3**, and, variously, incorporation of carbon monoxide to yield cyclopenta-2,4-dienones and dimeric derivatives $[(cyclopentadienone)Co(CO)_2]_2$, **4**.[2,4]



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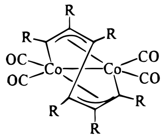

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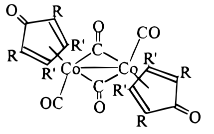

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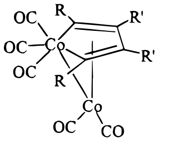
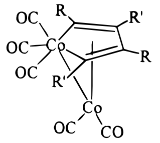
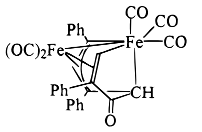
2a, 4, 5a, R = Ph, R' = H



5a



5b



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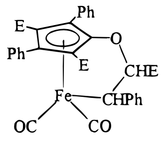

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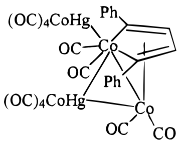

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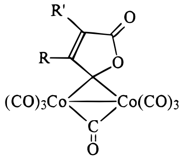

9

Uniquely, a prolonged reaction at 20 °C between $(PhC{\equiv}CH)Co_2(CO)_6$ (**2a**, R = Ph; R^1 = H) and excess ethynylbenzene yields a dark blue-violet complex $(PhC{\equiv}CH)_3Co_2(CO)_6$, **1**.[8] We became interested in determining the identity of **1** when we also isolated this as a byproduct from a reaction between $(PhC{\equiv}CH)Co_2(CO)_6$, ethynylbenzene, and trimethylamine *N*-oxide, which gives a cobaltacyclopentadiene, **5a**, as the major product.[9]

We were also interested in any possible structural relationship between the *penta*carbonyl **5a**, which *is* an intermediate in the cyclotrimerization of alkynes[9] and the *tetra*carbonyl **3** and the apparent *hexa*carbonyl **1**, which Hübel states categorically[10] are *not* catalysts for arene production, even though both the latter complexes are derived from three alkyne units.

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X-ray investigations have revealed numerous different arrangements by which three alkynes can be incorporated into organometallic structures. Considering only examples derived from ethynylbenzene, an Os_3 complex $(PhC{\equiv}CH)_3Os_3(CO)_7$ contains one alkyne bonded to an Os_2 unit, while the other two alkynes form an osmacyclopentadiene.[11] Oligomerization of three alkynes with one carbon monoxide is found in the tropone complexes $[(RC{\equiv}CH)_3CO]Fe_2(CO)_6$[12] and the 1-ferrabicyclo[3.3.0]octane complex, $[(PhC{\equiv}CH)_3CO]Fe_2(CO)_5$,[13] **6**. Hübel et al tentatively suggested[3] that similarities between the molecular formulas, color, thermal stability, and IR spectrum of **1** and **6** might indicate similar structural features.

This paper describes the structural characterization of **1** and identifies a previously unknown oligomerization assembly derived from three alkynes, two carbon monoxide units, and two cobalt atoms.

Results and Discussion

The air and solution stable dark blue-violet, diamagnetic, crystalline complex **1** was most easily obtained in 12% yield after separation from other products by reacting 1 equiv of **2a** with 10 equiv of PhC≡CH in hexane at room temperature for 7 days. It was also obtained as a byproduct in ~3% yield together with known[2,4] tetracarbonylbis-η^4-[2,5-diphenyl(cyclopenta-2,4-dienone)]dicobalt **4** from a large-scale preparation of the cobaltacyclopentadiene complex **5a**.[9] Purification was achieved by a combination of crystallization and chromatography. In agreement with previous spectroscopic characterization of **1**, IR spectroscopy showed the presence of an ester group [ν_{CO} (CH_2Cl_2): 1712(m) cm^{-1}] and terminal metal carbonyls [ν_{CO} (CH_2Cl_2): 2045(s), 2020(vs), 1951(w) cm^{-1}]. Mass spectroscopy indicated that the organic fragment corresponded to $\{(PhC{\equiv}CH)_3(CO)_2\}$; hence the molecule may be represented as $[(PhC{\equiv}CH)_3(CO)_2]Co_2(CO)_4$.

^1H NMR spectroscopy showed the presence of more than one type of phenyl group, a unique −CH−C(O)- group (δ 3.36), and an AB system (δ 4.21, 4.45; ^3J 3.07 Hz). ^{13}C NMR spectroscopy showed three different phenyl groups, one sp^3 CH and two sp^2 CH groups, one ester group, cobalt carbonyls, and four other carbon atoms not substituted by hydrogens.

Collectively, these data are inconsistent with any of the previously known $Co_2(CO)_8$-catalyzed oligomerization arrangements of three alkynes and prompted a single-crystal X-ray study.

X-ray Crystal Structure of 1. The results of a single-crystal diffraction analysis of $[(PhC{\equiv}CH)_3(CO)_2]Co_2(CO)_4$ are shown in Figures 1−3 and Tables 1 and 2. It reveals an assembly of alkynes and CO which condense to give a ligand derived from the 2,5-diphenylhydroxycyclopenta-2,4-dienyl ester of 3-phenylpropynoic acid. Overall, the new ligand is generated by two PhC≡CH molecules adding to **2a**. The skeletal residue of **2a** can be seen in the Ph−C(5)−C(6)−$Co_2(CO)_4$ unit (Figure 1). Without implying any particular mechanistic sequence, it can be envisaged that C(7)−O(6) has inserted into the C(6)−Co(2) bond and become an acyl group. Another CO (C8), also originally bonded to Co(2) has condensed with two additional PhC≡CH mol-

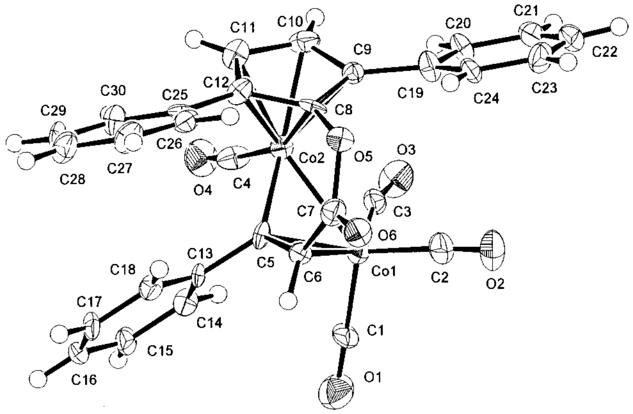

Figure 1. ORTEP plot of **1** with thermal ellipsoids at the 50% level and showing the atom-labeling scheme.

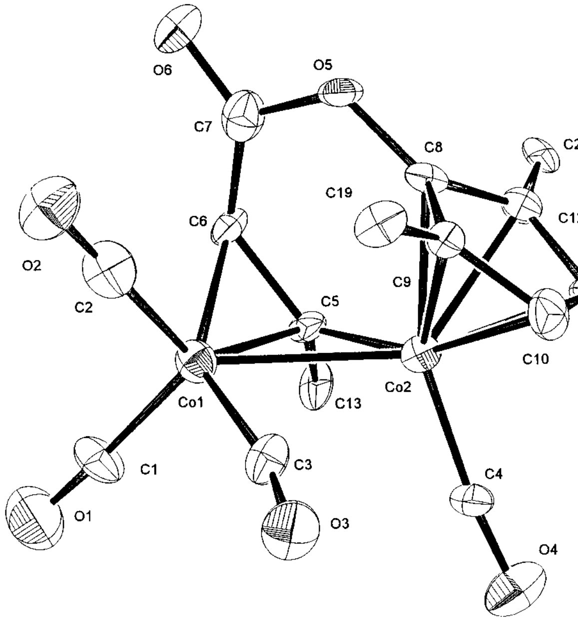

Figure 2. View of **1** with phenyl groups attached to C(5), C(9), and C(12) omitted for clarity.

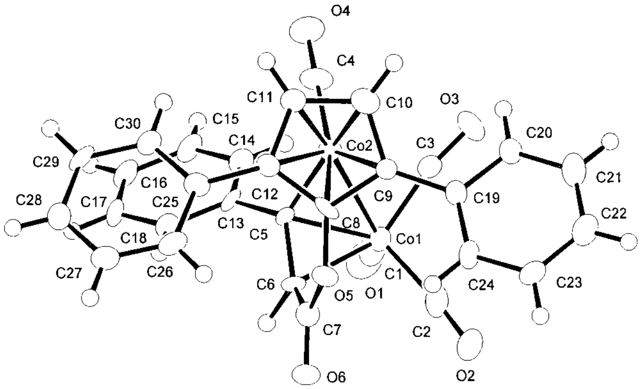

Figure 3. "Aerial" view of **1** along the Co(2)−cyclopentadienyl ring axis.

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Table 1. Selected Bond Lengths and Angles for 1

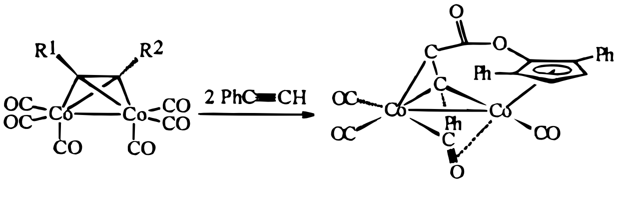
atoms	length (Å)	atoms	angle (deg)
Co(1)−Co(2)	2.512(2)	C(1)−Co(1)−C(2)	109.4(5)
Co(1)−C(1)	1.782(11)	C(1)−Co(1)−C(3)	102.2(5)
Co(1)−C(2)	1.801(13)	C(1)−Co(1)−C(5)	97.5(5)
Co(1)−C(3)	1.768(11)	C(1)−Co(1)−C(6)	103.5(5)
Co(1)−C(5)	1.975(9)	C(2)−Co(1)−C(3)	101.3(5)
Co(1)−C(6)	2.078(10)	C(2)−Co(1)−C(5)	133.6(5)
Co(2)−C(4)	1.790(11)	C(2)−Co(1)−(6)	94.8(5)
Co(2)−C(5)	1.937(10)	C(3)−Co(1)−C(5)	109.2(4)
Co(2)−C(8)	2.031(10)	C(3)−Co(1)−C(6)	142.9(4)
Co(2)−C(9)	2.131(10)	C(5)−Co(1)−C(6)	41.2(4)
Co(2)−C(10)	2.088(11)	Co(1)−Co(2)−C(4)	106.7(4)
Co(2)−C(11)	2.038(11)	C(4)−Co(2)−C(5)	98.8(5)
Co(2)−C(12)	2.073(9)	C(7)−O(5)−C(8)	119.3(8)
O(1)−C(1)	1.155(12)	Co(1)−C(1)−O(1)	175.9(11)
O(2)−C(2)	1.149(14)	Co(1)−C(2)−O(2)	174.6(11)
O(3)−C(3)	1.167(12)	Co(1)−C(3)−O(3)	166.2(10)
O(4)−C(4)	1.133(13)	Co(2)−C(4)−O(4)	173.1(10)
O(5)−C(7)	1.425(13)	Co(1)−C(5)−Co(2)	79.9(4)
O(5)−C(8)	1.371(12)	C(6)−C(5)−C(13)	117.9(9)
O(6)−C(7)	1.218(13)	Co(1)−C(6)−C(7)	110.0(7)
C(5)−C(6)	1.430(13)	C(5)−C(6)−C(7)	131.4(9)
C(5)−C(13)	1.474(13)	O(5)−C(7)−O(6)	110.9(10)
C(6)−C(7)	1.432(15)	O(5)−C(7)−C(6)	122.8(9)
C(8)−C(9)	1.434(12)	O(6)−C(7)−C(6)	126.3(10)
C(8)−C(12)	1.447(14)	O(5)−C(8)−C(9)	124.3(9)
C(9)−C(10)	1.435(14)	O(5)−C(8)−C(12)	125.4(8)
C(9)−C(19)	1.479(13)	C(9)−C(8)−C(12)	109.7(8)
C(10)−C(11)	1.433(14)	C(8)−C(9)−C(10)	106.1(9)
C(11)−C(12)	1.398(14)	C(8)−C(9)−C(19)	127.4(9)
C(12)−C(25)	1.474(13)	C(10)−C(9)−C(19)	126.3(9)
		C(9)−C(10)−C(11)	107.7(9)
Co(2)···C(3)	2.408(11)	C(10)−C(11)−C(12)	110.4(9)
Co(2)···Cp (centroid)	1.677(9)	C(8)−C(12)−C(11)	105.8(8)
		C(8)−C(12)−C(25)	127.7(9)

Table 2. Experimental Details of the Crystal Structure Determination of 1

Crystal Data	
molecular formula	$C_{30}H_{18}Co_2O_6$
mol wt	592.34
cryst color	black
cryst size, mm	0.2 × 0.15 × 0.03
space group	$P2_1/n$ (No. 14)
a, Å	14.736(3)
b, Å	8.615(3)
c, Å	19.839(5)
β, deg	100.29(2)
V, Å^3	2478(1)
Z	4
ρ_{calcd}, g cm^{-3}	1.588
μ (Mo kα), cm^{-1}	13.79
$F(000)$	1200.00
Data Collection	
diffractometer	Rigaku AFC7S
radiation	Mo Kα
wavelength, Å	0.710 69
T, K	123
scan type	$\omega-2\theta$
scan rate, deg min^{-1}	16 (in ω)
scan width	$1.00 + 0.35 \tan \theta$
θ range	2.5−27
index ranges	h, 0 to 18; k, 0 to 10; l, 23 to −24
Solution and Refinement	
no. of reflcns measd	5981
no. of unique reflcns	5763 ($R_{int} = 0.104$)
no. of obsd reflcns ($I > 3.00\sigma(I)$)	2367
no. of variables	339
GOF	1.07
R	0.063
R_w	0.072
residual electron density, e/Å^3	0.82 to −0.98

ecules to give, by well-established precedent, a 2,5-diphenylcyclopentadienone structure π-bonded to Co(2). O-acylation of O(5) by C(7)−O(6) generates the organic ligand. C(5) is σ-bonded to both Co(1) and Co(2); C(6) is σ-bonded to Co(1) only while the trisubstituted cyclopentadienyl ligand is π-bonded only to Co(2). Three carbonyls bonded to Co(1), one carbonyl bonded to Co(2), and a Co−Co bond complete 18-electron configurations at both metal atoms.

Scheme 1



1

Four features of the molecular structure merit discussion: the regiochemistry and origin of magnetic asymmetry of the oxycyclopentadienyl ring, the bonding of C(5), and the abnormal geometry of the Co(1)−C(3)−O(3) moiety.

The symmetrical 2,5-diphenyl substitution pattern of the oxycyclopentadienyl ring of **1** mirrors that of the cyclopentadienone complex **4**, the cobaltacyclopentadiene **5a** formed from PhC≡CH in the same reaction, and the mercury−cobaltacycle **8**.[14] By contrast, the oxycyclopentadienyl ring of the iron complex **7** from $PhC{\equiv}CCO_2Me$ is asymmetrically disubstituted by phenyl groups,[15] but this alkyne also yields an asymmetric cobaltacyclopentadiene **5b**.[9] However $PhC{\equiv}CCO_2Me$ does not appear to give an analogue of **1**, and we have not, so far, detected similar derivatives from any other alkyne, although it might be expected from the terminal alkyne, tBuC≡CH, which favors cyclopentadienone formation.[3] There is, however, one report[16] of a similar trimerization of an alkyne: a chromium carbene complex reacts with three $MeC{\equiv}CNEt_2$ units to give a mononuclear chromium derivative with a symmetrical oxycyclopentadienyl ring, but this is presumed to arise via the dimerization of a ketene complex.

Inspection of the Cp ring environment in Figure 3 reveals the origin of the magnetic distinction between H(10) and H(11) manifested in the ^{1}H and ^{13}C NMR spectra. In the solid state the proximate carbonyl CO-(4) is asymmetrically located with respect to these two protons, while the C(5)−C(6)−C(7)−O(5) bridge is offset from the plane orthogonal to the Cp ring.

There are some unusual aspects of the bonding of C(5). The C(5)−C(6) bond length [1.43(1) Å] is not significantly different from the similar bond in **2** (e.g., $R = R^1 = Ph$; 1.46 Å),[17] and the Co(2)−C(5)−C(13) bond angle of 123.0(1)° suggests sp^2 hybridization at C(5). However C(5)−C(6) is not a symmetrical η^2-donor because the Co(1)−C(6) bond [2.08 Å] is longer than Co(1)−C(5) [1.976 Å]. C(5) is at the apex of an equilateral triangle with a Co(1)−C(5)−Co(2) angle of 79.8°, and Co(2)−C(5) [1.94 Å] is very similar to Co(1)−C(5). Furthermore the coordinates of this triangle are similar (Scheme 2) to those of the lactone complex **9**,[18] which is best considered as a bridging carbene complex.

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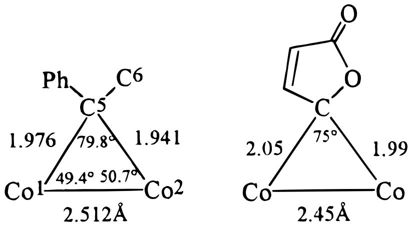
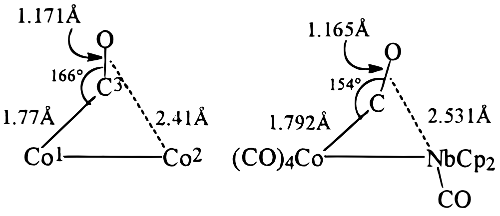
Scheme 2



1 **9**

Scheme 3



1 **10**

Finally the molecular structure suggests that carbonyl CO(3) is "semibridging". While the other two carbonyl ligands attached to Co(1) and the single carbonyl attached to Co(2) are normal, there are abnormalities associated with CO(3). The Co(1)−C(3) bond is rather shorter [1.77 Å] and the C(3)−O(3) bond is rather longer than normal, but the Co(1)−C(3)−O(3) bond angle at 166° is some 8° more acute than normal and is noticeably "bent" (see Figures 1−3). Also since the Co(2)−C(3) distance [2.41 Å] is approximately 75% of the sum of the van der Waals radii of the carbon and cobalt, there must be some bonding interaction. This carbonyl group is thus reasonably described as a "semi−bridging" carbonyl and can be rationalized within the context of such an interaction proposed by Cotton;[19] that is, carbonyl CO(3) moves toward Co(2) to assist the delocalization of "excess" negative charge on that metal, created by the strongly π-donating oxycyclopentadienyl ring and the relative paucity of other ligands able to accept back-bonding. "Semibridging" carbonyl groups are encountered widely in alkyne−iron complexes[20] but are less common in cobalt chemistry. Examples include $Cp_3Co_3(CO)_3$[21] and the mixed metal complex $Cp_2Nb(CO)Co(CO)_4$,[22] **10**; a comparison between the relevant structural parameters of **1** and **10** is shown in Scheme 3.

Experimental Section

Preparation of 1 from 2. Complex **2a**, $(PhC{\equiv}CH)Co_2(CO)_6$ (10.58 g, 0.027 mol), and ethynylbenzene (10.58 g, 11.02 mL, 0.104 mol) were combined in hexane (100 mL) and left to stir at room temperature under N_2 for 10 days. The reaction solution was removed by decantation to leave a brown amorphous solid, which was washed with hexane until the washings were clean. The brown solid was chromatographed in portions on neutral alumina, and the dark blue/black band eluted with toluene; the solvent was removed to afford **1** as a black crystalline product (3.10 g, 12%). Crystals suitable for X-ray analysis were obtained from *n*-hexane/benzene by slow evaporation at 0 °C: ^1H NMR (400 MHz, C_6D_6) δ 3.36(s, 1H), 4.21(d, $J = 3.03$ Hz, 1H), 4.46(d, $J = 3.11$ Hz, 1H), 6.45(d, $J = 7.61$ Hz, 2H), 7.21−7.67(m, 11H), 7.605(d, $J = 7.05$ Hz, 2H); ^{13}C NMR (100 MHz, C_6D_6) δ 44.94, 76.69, 95.02, 105.51, 112.92, 126.88, 127.13, 127.53, 127.58, 127.78, 127.82, 128.01, 128.05, 128.13, 128.18, 128.23, 128.57, 128.90, 128.96, 129.42, 129.70, 152.02, 172.49, 203.81 {M-CO}; MS *m/z* 362.1377 $[M^+ - Co_2(CO)_4]$; $C_{26}H_{18}O_2$, $M_r = 362.1387$. Anal. Calcd for $C_{30}H_{18}Co_2O_6$: C, 60.83; H, 3.11. Found: C, 61.02; H, 3.11.

Chromatography of the reaction solution (neutral alumina, petrol/ether mixtures) yielded unchanged **2** (8.36 g, 79% recovery), a trace of **1**, 1,2,4-triphenylbenzene (white crystals, 802 mg, 6%), and the bis(cyclopentadienone)dicobalt complex **4** (orange crystals, 203 mg), all identified by spectroscopic comparisons with authentic compounds.

Details of the isolation of **1** from a large-scale preparation of **5a** are given in the preceding paper.[9]

Crystallographic Analysis. Black plate crystals of **1** suitable for X-ray diffraction studies were obtained by slow evaporation of an *n*-hexane/benzene solution at 0 °C. The complex crystallizes in the monoclinic space group $P2_1/c$. A crystal having dimensions 0.20 × 0.15 × 0.03 mm was coated in oil, mounted on a glass fiber, and cooled to −150 °C, at which temperature data collection was performed. Accurate cell dimensions were obtained from 20 precisely centered reflections in the range $20.45° \leq 2\theta \leq 27.88°$. Data collection in the range $2.5° \leq \theta \leq 27°$ was performed using $\omega-2\theta$ scans on a Rigaku AFC7S diffractometer with graphite-monchromated Mo Kα radiation. A total of 5981 reflections were recorded, of which 5763 were unique ($R_{int} = 0.104$) and were corrected for Lorentz and polarization effects. The structure was solved by direct methods (SIR92)[23] and expanded using Fourier techniques (DIRDIF92).[24] An empirical absorption correction (trans: min. 0.449, max. 1.000) was applied to the isotropic model (DIFABS).[25] Full-matrix least-squares refinement based on 2367 observed reflections ($I > 3.00\sigma(I)$) with all non-H atoms except C(9) anisotropic converged with $R = 0.064$, $R_w = 0.073$, GOF = 1.07. H atoms were included in calculated positions. All calculations were performed using the teXsan crystallographic software package of Molecular Structure Corporation.[26] The experimental data are summarized in Table 2.

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Supporting Information Available: Additional experimental details for the structure determination and tables of atomic coordinates, anisotropic displacement parameters, and interatomic distances and bond angles (20 pages). Ordering information is given on any current masthead page.

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