Transition-Metal Complexes with Silicon-Containing Cyclic Ligands. 2. Crystal and Molecular Structure of Mono- and Binuclear Complexes of 9,9,10,10-Tetramethyl-9,10-disiladihydroanthracene

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X-ray crystal analyses of mononuclear $[Cr(CO)_3][C_6H_4(SiMe_2)_2C_6H_4]$ (II) and two binuclear trans and cis complexes $[Cr(CO)_3]_2[C_6H_4(SiMe_2)_2C_6H_4]$ (III and IV) have shown that coordination of 9,9,10,10-tetramethyl-9,10-disiladihydroanthracene (I) both by one (in II) and by two (in III and IV) Cr atoms causes only relatively small changes of its conformation. The most significant ligand bending along the Si···Si line (145.2°) is found in the less sterically hindered mononuclear complex II. In the binuclear complex IV both $Cr(CO)_3$ groups are situated on that side of the average plane of ligand I where the distance between them is decreased. Crystals of II are monoclinic of space group $P2_1/c$ with a = 16.941 (5) Å, b = 6.954 (2) Å, c = 17.489 (6) Å, β = 105.93 (3)°, and Z = 4. Crystals of III are orthorhombic of space group Cmc_1 with a = 13.093 (3) Å, b = 13.871 (3) Å, c = 12.926 (2), and Z = 4. Crystals of IV are orthorhombic of space groups $Pbc2_1$ with a = 8.783 (2) Å, b = 14.893 (4) Å, c = 18.226 (4) Å, and Z = 4.

Introduction

$Cr(CO)_6$ reacts with a polycyclic silicon-containing system, viz., 9,9,10,10-tetramethyl-9,10-disiladihydroanthracene (I), to form three types of metal-containing complexes: mononuclear $[Cr(CO)_3][C_6H_4(SiMe_2)_2C_6H_4]$ (II) and two binuclear compounds $[Cr(CO)_3]_2[C_6H_4(SiMe_2)_2C_6H_4]$ (III and IV).[1] On the basis of spectral data the structure of (arene)chromium tricarbonyl complexes with a Si-bonded benzene ring of the heterocyclic ligand was proposed for compounds II–IV. The purpose of this work is an X-ray study of complexes II–IV to prove unambiguously their structure and to determine the effect of complex formation with one and two metal atoms on the geometry of silicon-containing heterocyclic ligands I, whose crystal structure in the free state was determined earlier.[4]

Experimental Section

Suitable lemon yellow needle crystals of $[Cr(CO)_3][C_6H_4(SiMe_2)_2C_6H_4]$ (II) were obtained by crystallization from heptane, and bright yellow isometric crystals of both III and IV, $[Cr(CO)_3]_2[C_6H_4(SiMe_2)_2C_6H_4]$, were obtained by crystallization from an acetone–heptane solution. Unit cell parameters and intensities of diffraction reflections were measured with an automated Nicolet R3m X-ray diffractometer (T = 20 °C; Mo Kα radiation; λ = 0.710 69 Å^{-1}; graphite parallel monochromator; $2\theta/\theta$ scan; absorbtion corrections were neglected because of the small dimensions of the crystal, about 0.2 × 0.3 × 0.3 mm^3).

Crystals of II are monoclinic of space group $P2_1/c$ with a = 16.941 (5) Å, b = 6.954 (2) Å, c = 17.489 (6) Å, β = 105.93 (3)°, d_{calcd} = 1.36 g·cm^{-3}, μ = 7.4 cm^{-1}, and Z = 4.

Crystals of III are orthorhombic of space group $Cmc2_1$ with a = 13.093 (3) Å, b = 13.871 (3) Å, c = 12.926 (2) Å, d_{calcd} = 1.59 g·cm^{-3}, μ = 11.0 cm^{-1}, and Z = 4 (the molecule lies in the symmetry plane m).

Crystals of IV are orthorhombic of space group $Pbc2_1$ with a = 8.783 (2) Å, b = 14.893 (4) Å, c = 18.226 (4) Å, d_{calcd} = 1.51 g·cm^{-3}, μ = 10.9 cm^{-1}, and Z = 4.

A total of 2350 (II), 1147 (III), and 1734 (IV) reflections were obtained, 2274, 1062, and 1607, respectively, with $I > 2\sigma(I)$ being considered "observed" and used in the solution by the heavy-atom method and the full-matrix least-squares anisotropic refinement of the structures. In cases III and IV the refinement was carried out for both "direct" and "inverted" structures; however, the R factor values were, in fact, very close. All calculations were made with a Nova-3 computer using the XTL program package.

The atomic scattering factors were calculated from the analytical expression for the scattering factors curves.[2] The f' and f'' components of anomalous dispersion were those of Cromer and Liberman[3] and were included in the calculations for all atoms.

The final R and R_w values are 0.037 and 0.025 for II, 0.026 and 0.030 for III, 0.025 and 0.030 for IV, respectively. In all three structures difference syntheses revealed all hydrogen atoms that were included in the structure factor calculation with fixed temperature parameters.

Final atomic coordinates for non-hydrogen atoms are given in Table I. Bond lengths in molecules II, III, and IV are given in Table II, and the geometry of molecule IV is shown in the Figure 1. Atom numbering of molecules II and III coincides with that of IV.

Results and Discussion

Chromium atoms in all three structures of II–IV are in fact symmetrically coordinated by six carbon atoms of aromatic η^6 rings. Individual Cr–C(arene) distances (Table II) differ only slightly from the average values that are 2.221, 2.217, and 2.216 Å in II, III, and IV, respectively. In the other known structures of typical π-arene complexes these distances equal 2.221 Å (average) in $(C_6H_6)Cr(CO)_3$,[5] 2.211–2.253 Å (average 2.233 Å) in $[(CH_3)_6C_6]Cr(CO)_3$,[6] 2.218–2.239 Å (average 2.227 Å) in $(SiMe_3)(C_6H_5)$ $Cr(CO)_3$,[7] and 2.206–2.289 Å (average 2.236 Å) in the orthorhomibc modification of (phenanthrene)chromium tricarbonyl.[8]

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Table I. Fractional Atomic Coordinates (×10^5 for Cr and ×10^4 for the Other Atoms) for the Non-Hydrogen Atoms with Esd's in Parentheses for Molecules II, III, and IV

atom	*x*	*y*	*z*	atom	*x*	*y*	*z*
			II				
Cr	14662 (3)	890 (8)	38804 (3)	C(11)	4458 (2)	235 (6)	3390 (3)
Si(1)	3376 (1)	2793 (2)	3865 (1)	C(12)	3677 (2)	1075 (5)	3169 (2)
Si(2)	2091 (1)	1646 (1)	2054 (1)	C(13)	3916 (3)	2251 (9)	4921 (4)
C(1)	2230 (2)	2593 (4)	3700 (2)	C(14)	3623 (3)	5264 (7)	3631 (3)
C(2)	1876 (2)	2995 (5)	4331 (2)	C(15)	2069 (3)	3979 (7)	1538 (3)
C(3)	1044 (3)	2881 (6)	4240 (2)	C(16)	1433 (3)	87 (8)	1362 (2)
C(4)	518 (2)	2329 (6)	3505 (3)	C(17)	1413 (2)	−2006 (5)	3239 (2)
C(5)	845 (2)	1958 (5)	2874 (2)	C(18)	2315 (3)	−1134 (5)	4605 (2)
C(6)	1697 (2)	2063 (4)	2950 (2)	C(19)	789 (2)	−1218 (6)	4346 (2)
C(7)	3148 (2)	681 (5)	2407 (2)	O(1)	1367 (2)	−3351 (4)	2842 (2)
C(8)	3444 (3)	−498 (6)	1895 (3)	O(2)	2824 (2)	−1970 (4)	5052 (2)
C(9)	4222 (4)	−1253 (8)	2137 (4)	O(3)	364 (2)	−2068 (5)	4641 (2)
C(10)	4724 (3)	−926 (8)	2861 (4)				
			III				
Cr(1)	0	42373 (5)	−36460 (8)	C(14)	2429 (4)	2566 (5)	−1222 (4)
Cr(2)	0	10072 (5)	0	C(17)	988 (3)	4757 (3)	−4485 (4)
Si	1363 (1)	2296 (1)	−2129 (1)	C(18)	0	3211 (4)	−4544 (5)
C(1)	550 (2)	3409 (2)	−2261 (3)	C(21)	0	2297 (5)	340 (6)
C(2)	1056 (3)	4305 (3)	−2326 (3)	C(22)	−970 (3)	848 (3)	1015 (4)
C(3)	−528 (3)	5185 (3)	−2374 (4)	O(1)	1626 (2)	5077 (3)	−5006 (3)
C(7)	556 (3)	1294 (3)	−1598 (3)	O(2)	0	2608 (4)	−5159 (4)
C(8)	1069 (4)	484 (3)	−1196 (3)	O(4)	−1560 (3)	762 (3)	1667 (4)
C(9)	−539 (4)	−320 (4)	−798 (4)	O(5)	0	3090 (4)	588 (6)
C(13)	1886 (4)	1887 (4)	−3396 (4)				
			IV				
Cr(1)	91708 (7)	57529 (5)	−25490 (5)	C(13)	7402 (7)	3523 (3)	−534 (3)
Cr(2)	67962 (8)	69271 (5)	−213 (5)	C(14)	10059 (7)	4786 (4)	−296 (3)
Si(1)	8146 (1)	4669 (1)	−707 (1)	C(15)	4670 (6)	6791 (3)	−2159 (4)
Si(2)	5237 (1)	5736 (1)	−1696 (1)	C(16)	3719 (6)	4869 (4)	−1785 (4)
C(1)	8176 (5)	4801 (3)	−1740 (3)	C(17)	10827 (6)	5996 (4)	−3136 (3)
C(2)	9337 (5)	4361 (3)	−2135 (3)	C(18)	10219 (5)	6303 (3)	−1801 (3)
C(3)	9341 (6)	4335 (3)	−2894 (3)	C(19)	8472 (6)	6871 (3)	−2809 (3)
C(4)	8149 (6)	4732 (4)	−3295 (3)	C(20)	8761 (6)	7021 (3)	304 (3)
C(5)	6999 (5)	5170 (3)	−2917 (3)	C(21)	6424 (6)	8050 (3)	340 (3)
C(6)	6960 (5)	5222 (3)	−2131 (3)	C(22)	7433 (5)	7506 (3)	−849 (3)
C(7)	5563 (5)	5904 (3)	−682 (3)	O(1)	11866 (4)	6157 (3)	−3495 (2)
C(8)	4474 (5)	6428 (3)	−301 (3)	O(2)	10898 (4)	6657 (3)	−1347 (2)
C(9)	4573 (6)	6552 (4)	461 (3)	O(3)	8054 (4)	7575 (3)	−2980 (2)
C(10)	5716 (6)	6125 (4)	857 (3)	O(4)	10009 (5)	7085 (3)	520 (3)
C(11)	6768 (6)	5597 (3)	485 (3)	O(5)	6206 (6)	8778 (3)	550 (3)
C(12)	6736 (5)	5468 (3)	−288 (2)	O(6)	7894 (4)	7915 (2)	−1343 (2)

Table II. Relevant Bond Lengths (Å) in Molecules II, III, and IV

bond	II	III	IV	bond	II	III	IV
Cr(1)-C(1)	2.243 (3)	2.246 (4)	2.224 (5)	C(17)-O(1)	1.155 (5)	1.160 (5)	1.148 (7)
Cr(1)-C(2)	2.211 (4)	2.199 (4)	2.212 (5)	C(18)-O(2)	1.150 (5)	1.154 (8)	1.148 (6)
Cr(1)-C(3)	2.219 (4)	2.216 (4)	2.209 (5)	C(19)-O(3)	1.156 (6)	1.160 (5)[a]	1.153 (6)
Cr(1)-C(4)	2.204 (4)	2.216 (4)[a]	2.228 (5)	C(20)-O(4)		1.149 (6)	1.169 (7)
Cr(1)-C(5)	2.208 (4)	2.199 (4)[a]	2.201 (5)	C(21)-O(5)		1.146 (9)	1.166 (6)
Cr(1)-C(6)	2.244 (3)	2.246 (4)[a]	2.231 (4)	C(22)-O(6)		1.149 (6)[a]	1.160 (6)
Cr(2)-C(7)		2.226 (4)	2.223 (4)	average	1.154 (7)	1.153 (7)	1.157 (6)
Cr(2)-C(8)		2.208 (4)	2.230 (5)				
Cr(2)-C(9)		2.225 (4)	2.213 (5)	Si(1)-C(13)	1.860 (6)	1.864 (5)	1.855 (5)
Cr(2)-C(10)		2.225 (4)[a]	2.211 (5)	Si(1)-C(14)	1.841 (5)	1.862 (6)	1.848 (6)
Cr(2)-C(11)		2.208 (4)[a]	2.185 (5)	Si(2)-C(15)	1.852 (5)	1.864 (5)[a]	1.851 (6)
Cr(2)-C(12)		2.226 (4)[a]	2.228 (4)	Si(2)-C(16)	1.849 (5)	1.862 (6)[a]	1.864 (5)
average	2.222 (4)	2.220 (4)	2.216 (5)	average	1.850 (5)	1.863 (6)	1.854 (6)
Cr(1)-C(17)	1.826 (4)	1.836 (4)	1.842 (5)	Si(1)-C(1)	1.889 (4)	1.883 (4)	1.893 (5)
Cr(1)-C(18)	1.843 (4)	1.836 (5)	1.838 (5)	Si(1)-C(12)	1.873 (4)	1.876 (4)	1.879 (5)
Cr(1)-C(19)	1.823 (4)	1.836 (4)[a]	1.837 (5)	Si(2)-C(6)	1.886 (3)	1.883 (3)[a]	1.872 (4)
Cr(2)-C(20)		1.839 (4)	1.830 (6)	Si(2)-C(7)	1.852 (4)	1.876 (4)[a]	1.887 (5)
Cr(2)-C(21)		1.842 (6)	1.826 (5)	average	1.875 (4)	1.879 (4)	1.882 (5)
Cr(2)-C(22)		1.839 (4)[a]	1.826 (5)				
average	1.831 (4)	1.838 (5)	1.833 (5)				

[a] Symmetrically independent distance.

In the binuclear complex III two Cr(CO)$_3$ groups are situated on the opposite sides of the average ligand plane (trans configuration). In complex IV two Cr(CO)$_3$ groups are situated on one side of the average ligand plane (Figure 1), the distance Cr···Cr of 5.351 Å being considerably greater than the doubled covalent radius of the Cr atom

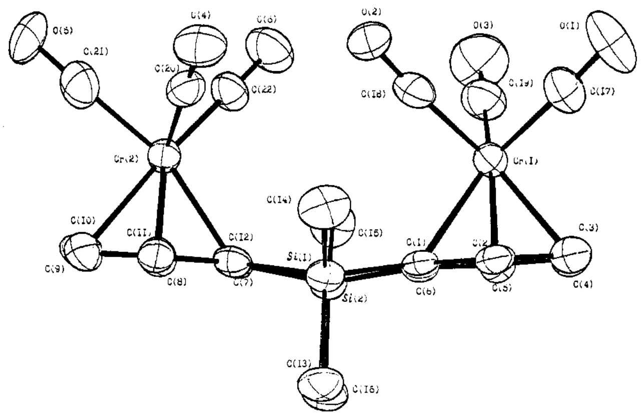

Figure 1. Structure of molecule IV.

that equals 2.96 Å,[9] so as there is no direct bond between metal atoms.

In the free state the molecule of 9,9,10,10-tetramethyl-9,10-disiladihydroanthracene (I) has a boat conformation[4] wherein the central six-membered heterocycle is bent along the Si···Si line with a dihedral angle of 155°. In mononuclear as well as in both binuclear complexes a boat conformation of the ligand is retained, but its deviation from planarity is different. Thus in the mononuclear complex II the ligand is bent along the Si···Si line forming a dihedral angle of 145.2°, and in the binuclear complexes III and IV the ligands are bent by angles of 160.0° and 160.7°, respectively.

It is interesting to note that in IV both $Cr(CO)_3$ groups are situated on that side of the average ligand plane where the distance between them is decreased (both groups are located under a "house roof") in comparison with other possible conformations (both groups are located over a "house roof") (Figure 1). In complex II the $Cr(CO)_3$ group is also situated under a "house roof". Apparenlty this space arrangement is most favorable energetically.

At the same time, on the basis of an X-ray analysis of some phenaza- and phenoxasilines and phenaza- and phenoxastannines it was found that the value of the dihedral angle of bending of the central heterocycle is strongly dependent both on the nature of heteroatoms and on the substituent's character[10] and varies in the compounds studied from 125° to 165°. In the complexes investigated here the greatest cycle bending is found in the less sterically hindered mononuclear complex II.

In all three complexes endocyclic Si–C bonds (average 1.875 (4), 1.879 (4) and 1.882 (5) Å in II, III, and IV, respectively) are longer than exocyclic $Si-CH_3$ bonds (average 1.850 (5), 1.863 (6), and 1.854 (6) Å respectively), but the lengths of both bond types differ little from the standard value of an ordinary bond of 1.87 Å.[11] In the free Ligand I the average lengths of endo- and exocyclic bonds are 1.861 and 1.889 Å. Differences in Si–C bond lengths found in I, II, III, and IV and their deviations from the standard value of 1.87 Å do not exceed 3σ and therefore cannot be considered as statistically significant.

As a rule, chromium tricarbonyl complexes of condensed polyaromatic systems where the chromium atom interacts with one of the rings have a staggered orientation of the $Cr(CO)_3$ group relative to the aromatic fragment.[12] Only in the case of some additional interactions (e.g., strongly orienting substituents in benzene ring[13]), an eclipsed orientation is found. In the present investigation a practically ideal staggered conformation is established in the mononuclear complex II and the binuclear complex III. The same conformation was found in $(C_6H_6)Cr(CO)_3$[5] and $(SiMe_3)(C_6H_5)Cr(CO_3$.[7] The orientation of the $Cr(CO)_3$ fragment that is characterized by an angle of rotation (α) of CO groups relative to carbon atoms of the coordinated benzene ring is shown in the Figure 2. In the cases of the ideal eclipsed and staggered conformations α is equal to 0° and 30°, respectively. As can be seen in Table III the mean value of α is 28.0° in II and 29.8° in III. At the same time orientation of the $Cr(CO)_3$ fragments in complexes II and III is different. In the binuclear complex III both fragments are rotated by 60° relative to the position of the $Cr(CO)_3$ fragment in the mononuclear complex II (Figure 2).

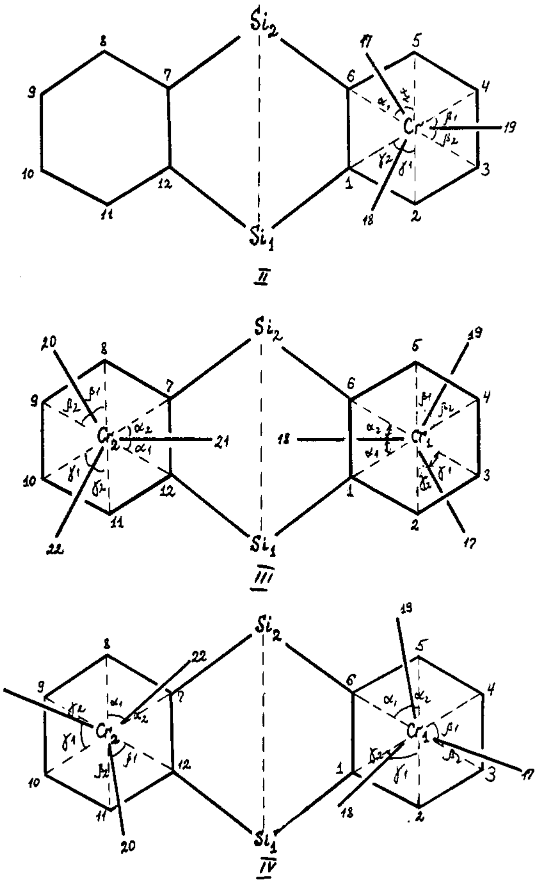

Figure 2. Projections of molecules II, III, and IV on the average plane of the disiladihydroanthracene fragment.

Table III. Angles of Rotation of CO Groups in Molecules II, III, and IV Relative to the Coordinated Benzene Ring

angle of rotatn	II at Cr	III at Cr(1)	III at Cr(2)	IV at Cr(1)	IV at Cr(2)
α_1	29.8	30.2	30.5	47.0	56.0
α_2	30.8	30.2	30.5	14.0	4.6
β_1	27.3	30.6	30.9	44.7	57.8
β_2	32.5	29.7	29.4	15.2	2.9
γ_1	27.0	29.7	29.4	45.2	56.2
γ_2	33.9	30.6	30.9	15.0	2.9

It should be noted that in complexes II and III where coordinated benzene rings have two Si atoms in ortho-positions the orientation of the $Cr(CO)_3$ group is the same as in the nonsubstituted $(C_6H_6)Cr(CO)_3$ and monosub-

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stituted $(SiMe_3)(C_6H_5)Cr(CO_3$ complexes.

In the binuclear cis complex IV the orientation of the $Cr(CO)_3$ fragment is close to the eclipsed conformation (the mean value of the α angle is 14.7° and 3.5° for the Cr(I) and Cr(2) atoms). Probably such mutual orientation of the $Cr(CO)_3$ groups is determined by a tendency to minimize steric repulsions between CO groups at adjacent Cr atoms. Indeed, there are some short nonbonding contacts in molecule IV (C(18)···C(22) = 3.49, C(18)···O(6) = 3.26, C(19)···O(6) = 3.13, and C(20)···O(2) = 3.24 Å with the sums of van der Waals radii being C···C = 3.60 and C···0 = 3.20 Å).

Registry No. II, 84179-62-4; III, 84179-63-5; IV, 84236-68-0.

Supplementary Material Available: Anisotropic temperature factors, hydrogen atom coordinates, bond lengths and angles, planes and deviations therefrom, and structure factor (121 pages). Ordering information is given on any current masthead page.

Communications

New Bridged Ruthenium Carboxylate Complexes. Structure and Catalytic Activity

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Summary: The reaction of $Ru_3(CO)_{12}$ with benzoic acid and 2-methylbutyric acid gives two structurally different and unexpected complexes. X-ray crystallographic analyses gave the structure of $[Ru(\mu\text{-}PhCO_2)(\eta^1\text{-}PhCO_2H)(CO)_2]_2$ and $[(Ru_2(\mu\text{-}sec\text{-}C_4H_9CO_2)_2(\eta^1\text{-}sec\text{-}C_4H_9CO_2H)(CO)_4]_2$ for the complexes obtained with the above two acids. The latter complex is a logical structural model for the previously reported ruthenium carboxylate polymers. Both complexes are catalytically active in the addition reactions of carboxylic acids to acetylenes.

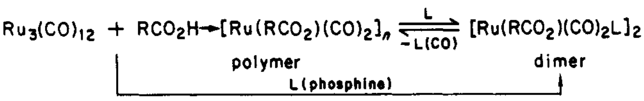
In a recent communication[1] we reported on a new catalytic reaction of $Ru_3(CO)_{12}$, described in a general form, by eq 1. Three isomeric vinyl esters are produced in good yields and selectivity.

$$R'CO_2H + RC\equiv CR \xrightarrow{Ru_3(CO)_{12}} \underset{E+Z}{R'CO_2(R)C=CHR} + R'CO_2(H)C=CR_2 \quad (1)$$

R = H, alkyl, aryl; R′ = alkyl, aryl

In conjunction with the above reaction we are studying the chemistry of $Ru_3(CO)_{12}$ and carboxylic acids. This subject was previously investigated by the groups of Lewis[2] and Schumann.[3,4] Their findings are summarized in Scheme I. The molecular structure of the insoluble polymer is not known. An X-ray crystallographic analysis[3] of a complex obtained from $Ru_3(CO)_{12}$, butyric acid, and tri-*tert*-butylphosphine revealed a structure of bis(μ-*n*-butyrato)bis[dicarbonyl(tri-*tert*-butylphosphine)ruthenium(I)]. Consequently, all the reported dimers[2,4] most probably possess similar bridging dicarboxylato core structure, $Ru\text{–}[O\text{–}C(R)\text{–}O]_2\text{–}Ru$.

Scheme I



R = H, CH_3, C_2H_5, C_3H_7, C_9H_{19}, $C_{17}H_{35}$; L = CO, phosphines, amines, arsines

In the present work we have studied the reactions of benzoic and 2-methylbutyric acids with $Ru_3(CO)_{12}$, which were carried out in toluene in a glass-lined reactor at 145 °C under nitrogen[5] (in eq 2 and 3 the number in parentheses represents moles of reactants).

$$PhCO_2H\ (15) + Ru_3(CO)_{12}\ (1) \rightarrow \underset{\mathbf{1}\ (48\%)}{[Ru(PhCO_2)(CO)_2(PhCO_2H)]_2} \quad (2)$$

$$sec\text{-}C_4H_9CO_2H\ (6) + Ru_3(CO)_{12}\ (1) \rightarrow \underset{\mathbf{2}\ (67\%)}{[Ru_2(sec\text{-}C_4H_9CO_2)_2(CO)_4(sec\text{-}C_4H_9CO_2H)]_2} \quad (3)$$

Both **1** and **2** were found to catalyze reaction 1 in the presence of their respective acids. However the structures of **1** and **2** are intriguing. The infrared spectra of **1**[6] and **2**[7] exhibit a broad O–H stretching band in the region of 2700–3200 cm^{-1} and a carbonyl band at 1670 (1675) cm^{-1}, which are indicative of carboxylic acids. Lower frequency bands (1560–1600 cm^{-1}) indicate metal-bridged carboxylato type of bonding. The presence of both carboxylato and carboxylic acid ligands in the same transition-metal complex is certainly a surprising structural feature. The ^1H NMR spectrum of **1**[6] gives rise to two chemically shifted Ph resonances (1:1). Two sets of *sec*-butyl resonances (1:2) were observed in the ^1H NMR spectrum of **2**.[7] Elemental analyses dictate a 1:2 ratio of Ru to benzoic acid in **1**[6] and a 2:3 ratio of Ru to 2-methylbutyric acid in **2**.[7]

On the basis of the spectral data and the elemental analyses we have assigned the molecular formula $Ru(PhCO_2)(CO)_2(PhCO_2H)$ to **1** and $Ru_2(sec\text{-}C_4H_9CO_2)_2\text{-}$

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(5) The same reaction conditions prevail during catalysis (eq 1).

(6) Anal. Calcd for $C_{16}H_{11}O_6Ru$: C, 48.00, H, 2.75. Found: C, 47.70; H, 2.69. IR (CH_2Cl_2): 3200–2700 (br), 2040, 1995, 1962, 1670, and 1600 cm^{-1}. ^1H NMR ($CDCl_3$): δ 7.36 (t, J = 7.74 Hz, 2 H), 7.50 (t, J = 7.74 Hz, 1 H)8 7.53 (t, J = 7.37 Hz, 2 H), 7.68 (t, J = 7.37 Hz, 1 H), 7.88 (d, J = 7.74 Hz, 2 H), 8.20 (d, J = 7.37 Hz, 2 H).

(7) Anal. Calcd for $C_{19}H_{28}O_{10}Ru_1$: C, 36.89; H, 4.53; Ru, 32.7. Found: C, 36.87; H, 4.63; Ru, 32. IR (CH_2Cl_2): 3200–2700 (br), 2040, 1990, 1965, 1675, and 1560 cm^{-1}. ^1H NMR (acetone-d_4) δ 0.87 (t, J = 7 Hz, 6 H), 0.91 (t, J = 7 Hz, 3 H), 1.04 (d, J = 7 Hz, 6 H), 1.12 (d, J = 7 Hz, 3 H), 1.41 (m, 2 H), 1.61 (m, 4 H), 2.33 (m, 3 H).

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