tion, giving back the square-planar Rh(I) complex 5. The enantioselection is made at the cyclometalation step, and the atomic arrangement postulated earlier[4] explains the prevailing chirality.

Acknowledgment. We warmly thank Dr. Nobuaki Koga, Institute for Molecular Science, for his extremely valuable advice and discussions.

Supplementary Material Available: Tables giving calculated bond lengths and angles for 7–13 and related compounds (22 pages). Ordering information is given on any current masthead page.

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Mercuration of a Ruthenocene. Synthesis, Characterization, and Halogenation of $[(C_5(HgO_2CCH_3)_5)(C_5(CH_3)_5)Ru]$

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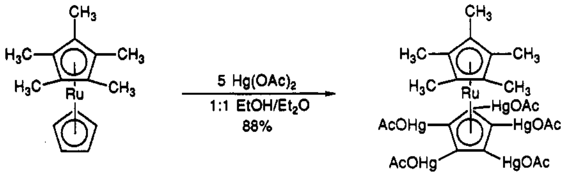
Summary: Treatment of pentamethylruthenocene with 5 equiv of mercuric acetate affords pentakis(acetoxymecurio)pentamethylruthenocene (88%), which can be halogenated with potassium triiodide or potassium tribromide to afford pentaiodopentamethylruthenocene (60%) and pentabromopentamethylruthenocene (35%). The crystal structure of the pentaiodopentamethylruthenocene derivative is described.

Substantial effort has been directed toward both the synthesis of mercurated cyclopentadienyl complexes and their subsequent transformation to functionalized species.[1] Unfortunately, the oxidizing nature of the mercuration process often destroys electron-rich organometallic fragments, so useful chemistry has really only been developed for ferrocene and substituted derivatives. While the mercuration of ruthenocene was reported in a 1960 publication,[2] subsequent work has shown that the interaction of ruthenocene with mercury(II) salts produces ruthenium–mercury-bonded adducts, rather than carbon–mercury bonds.[3] Herein we report that pentamethylruthenocene is preferentially pentamercurated at the unsubstituted cyclopentadienyl ring upon treatment with mercuric acetate in ethanol/diethyl ether. This represents the first well-characterized mercuration of a ruthenocene derivative and suggests that permercuration, rather than monomercuration, may be a general process in metallocene chemistry. Furthermore, the pentamercurated ruthenocene yields the pentahalo derivatives upon treatment with potassium triiodide or potassium tribromide. The crystal structure of pentaiodopentamethylruthenocene is reported and constitutes the first structural evaluation of a complex bearing an η^5-pentaiodocyclopentadienyl ligand.

Treatment of pentamethylruthenocene with mercuric acetate (5 equiv) in 1:1 ethanol/diethyl ether at ambient temperature for 18 h afforded a white precipitate of pentakis(acetoxymecurio)pentamethylruthenocene (1), which was obtained as an analytically pure powder in 88% yield after filtration and vacuum drying (eq 1).[4] The structure of 1 was established by ^{1}H NMR, infrared spectroscopy, and microanalysis and from reactivity studies (vide infra). In particular, the ^{1}H NMR spectrum showed no cyclopentadienyl C–H resonances, which indicated that 1 was fully pentamercurated. An experiment was performed in which pentamethylruthenocene was reacted with 1 equiv of mercuric acetate in 1:1 ethanol/diethyl ether for 18 h. Workup afforded 1 (18%) and recovered pentamethylruthenocene (78%). This indicates that the rates of mercuration for the intermediate mercurated species are all faster than the initial mercuration of pentamethylruthenocene.



1

(1)

The ^{1}H NMR spectrum of 1 was complicated. At ambient temperature in dichloromethane-d_2, four singlets (δ 2.07, 1.98 (br), 1.95 (br), 1.92) were observed. Upon cooling of the sample to −80 °C, seven resonances were obtained (δ 2.02, 1.97, 1.93, 1.90, 1.88, 1.80, 1.72). The 60 °C spectrum in benzene–d_6 showed two equal intensity singlets at δ 2.22 and 2.12. We propose that this behavior arises from acetate isomerism that occurs when the oxygen or methyl is either pointed up toward the Cp* ring or away from it. At low temperature, this would result in a sta-

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(4) Preparation of 1: A 250-mL round-bottomed flask was charged with pentamethylruthenocene (0.904 g, 3.00 mmol), mercuric acetate (4.78 g, 15.0 mmol), ethanol (100 mL), diethyl ether (50 mL), and a stir bar and was fitted with a rubber septum. The solution was stirred at ambient temperature for 18 h, during which time an off-white solid precipitated. The precipitate was collected on a medium glass frit and was washed succesively with ethanol (20 mL), ether (20 mL), and hexane (20 mL). Vacuum drying afforded 1 as an analytically pure off-white powder (4.19 g, 88%): decomposition range, with evolution of elemental mercury, 230–250 °C; IR (KBr) ν_{CO} 1570 (vs); ^{1}H NMR (CD_2Cl_2, 23 °C, δ) 2.07 (s), 1.98 (s, broad), 1.95 (s, broad), 1.92 (s). Anal. Calcd for $C_{25}H_{30}Hg_5O_{10}Ru$: C, 18.83; H, 1.90. Found: C, 18.84; H, 1.93.

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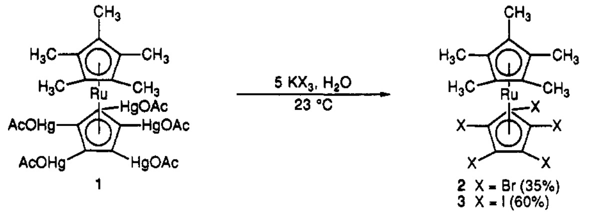
tistical mixture of all eight possible isomers and would account for the complicated nature of the observed spectrum.[5] At 60 °C carbonyl rotation is fast on the NMR time scale. This proposed model is based upon the crystal structure of phenylmercuric acetate,[6] which shows a nearly linear C–Hg–O bond (170°) with the plane of the acetate group approximately perpendicular to the plane of the aromatic ring. The observed hindered rotation indicates that substantial steric interactions are present in the permercurated cyclopentadienyl ligand. Interconversion of the isomers probably occurs by an "interlocking" mechanism, similar to that proposed for hexaisopropylbenzene[7a] and pentaisopropylcobaltocenium hexafluorophosphate.[7b]

Complex 1 can be halogenated with either potassium tribromide or potassium triodide (prepared from X_2 and KX at 25 °C) in aqueous medium to afford off-white pentabromopentamethylruthenocene (2, 35%) and bright yellow pentaiodopentamethylruthenocene (3, 60%), respectively, upon workup (eq 2). The structures of 2 and 3 were evident from their spectral and analytical data.[8,9]



1

2 X = Br (35%)
3 X = I (60%)

(2)

The crystal structure of 3·0.25CCl_4 was determined in order to understand its molecular geometry.[10] Figure 1 shows a perspective view of the molecule, along with selected bond lengths and angles. The cyclopentadienyl ligands are bonded to the ruthenium atom with idealized η^5-geometry and are staggered.[11] The ruthenium–carbon distances averaged 2.16 Å (C_5I_5) and 2.15 Å (Cp*), while the ruthenium–cyclopentadienyl centroid were 1.7854 (9) (C_5I_5) and 1.803 (9) Å (Cp*). Complex 3·0.25CCl_4 represents the first structurally characterized complex containing an η^5-pentaiodocyclopentadienyl ligand.[12]

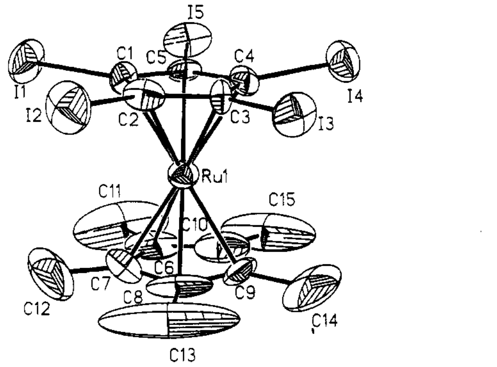


Figure 1. Perspective view of 3·0.25CCl_4. Selected bond lengths (Å) and angle (deg): Ru–C(1), 2.17 (1); Ru–C(2), 2.18 (1); Ru–C(3), 2.14 (1); Ru–C(4), 2.15 (1); Ru–C(5), 2.16 (1); Ru–C(6), 2.15 (2); Ru–C(7), 2.13 (1); Ru–C(8), 2.16 (2); Ru–C(9), 2.16 (1); Ru–C(10), 2.16 (2); Ru–C_5I_5(centroid), 1.7854 (9); Ru–Cp*(centroid), 1.803 (9); I(1)–C(1), 2.07 (1); I(2)–C(2), 2.07 (1); I(3)–C(3), 2.09 (1); I(4)–C(4), 2.07 (1); I(5)–C(5), 2.05 (1); C(1)–C(2), 1.42 (2); C(1)–C(5), 1.45 (2); C(2)–C(3), 1.40 (2); C(3)–C(4), 1.41 (2); C(4)–C(5), 1.46 (1); C(6)–C(7), 1.36 (3); C(6)–C(10), 1.41 (3); C(6)–C(11), 1.52 (3); C(7)–C(8), 1.42 (3); C(7)–C(8), 1.42 (3); C(7)–C(12), 1.44 (2); C(8)–C(9), 1.38 (3); C(8)–C(13), 1.51 (3); C(9)–C(10), 1.35 (3); C(9)–C(14), 1.50 (3); C(10)–C(15), 1.51 (3); C_5I_5(centroid)–Ru–Cp*(centroid), 179.1 (3).

In summary, pentamethylruthenocene is preferentially pentamercurated upon treatment with mercuric acetate to afford the mercurated derivative 1. Seemingly conflicting reports have suggested that the HgOAc group is a strong electron-withdrawing substituent on an arene ring (σ_p = 0.40)[13] and also that the HgX group possesses a neglible electronic effect as an aromatic substituent.[14] If the HgOAc group is electron withdrawing, then a possible rationale for the observed permercuration could be the strong σ,π-hyperconjugation associated with a carbon–mercury bond,[15] which would stabilize the adjacent positive charge involved in each mercuration step. A small group of organic aromatic compounds have been permercurated with mercuric trifluoracetate;[16a] ferrocene has even been

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(5) A chart showing the possible isomers of 1 is contained in the supplementary material.

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(8) Preparation of 2: A 100-mL round-bottomed flask was charged with potassium bromide (0.192 g, 1.61 mmol), bromine (0.082 mL, 1.59 mmol), water (25 mL), methanol (25 mL), and a stir bar. The resultant mixture was stirred at ambient temperature for 0.5 h, and then pentakis(acetoxymercurio)pentamethylruthenocene (0.427 g, 0.268 mmol) was added. The mixture was stirred at ambient temperature for 3 h, during which time a white precipitate formed. The crude product was collected on a medium-porosity glass frit and was successively washed with saturated aqueous sodium thiosulfate (250 mL) and saturated aqueous potassium iodide (250 mL) to remove mercury impurities. The precipitate was then extracted with dichloromethane (50 mL) to afford a colorless solution. This solution was applied to a 2-cm pad of silica gel on a coarse glass frit, and elution with dichloromethane (50 mL) afforded a colorless solution. Removal of the volatiles under reduced pressure afforded an analytically pure off-white powder (0.065 g, 35%): decomposition point (sealed tube) 270 °C; ^1H NMR ($CDCl_3$, δ) 1.65 (s, $C_5(CH_3)_5$); $^{13}C\{^1H\}$ NMR ($CDCl_3$, ppm) 91.21 (s, C–CH_3), 80.09 (s, C–Br), 8.25 (s, CH_3). HRMS: calcd for $C_{15}H_{15}Br_5Ru$, m/e 691.6130; found, m/e 691.6139. Anal. Calcd for $C_{15}H_{15}Br_5Ru$: C, 25.89; H, 2.17. Found: C, 25.70; 2.05.

(9) Preparation of 3: In analogy with the preparation of 2, potassium triiodide (prepared from potassium iodide (0.311 g, 1.87 mmol) and iodine (0.237 g, 1.87 mmol) in water (25 mL) and methanol (25 mL)) was reacted with pentakis(acetoxymercurio)pentamethylruthenocene (0.427 g, 0.268 mmol) to afford a yellow powder (0.150 g, 60%). An analytical sample was crystallized from hexane/carbon tetrachloride to provide bright yellow needles of 3·0.25CCl_4: decomposition point (sealed tube) 190 °C; ^1H NMR ($CDCl_3$, δ) 1.58 (s, $C_5(CH_3)_5$); $^{13}C\{^1H\}$ NMR ($CDCl_3$, ppm) 90.71 (s, C–CH_3), 62.45 (s, C–I), 7.06 (s, CH_3). Anal. Calcd for $C_{15}H_{15}I_5Ru\cdot0.25CCl_4$: C, 18.90; H, 1.56. Found: C, 18.92; 1.52.

(10) Crystallization of 3 from hexane/carbon tetrachloride afforded suitable crystals of the formulation 3·0.25CCl_4. Crystal data for 3·0.25CCl_4: $C_{15.25}H_{15}ClI_5Ru$, tetragonal crystal system, space group $I\bar{4}$, a = 22.434 (5) Å, b = 22.434 (5) Å, c = 9.167 (1) Å, V = 4614 (2) Å^3, Z = 8, d_{calcd} = 2.791 g/cm^3, $\theta/2\theta$ scanning technique, refined in a full matrix with the programs of SHELX-76, R = 0.038. Further information is contained in the supplementary material.

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reported to be decamercurated upon treatment with mercuric trifluoroacetate.[16b] It should be anticipated that pentamercuration of cyclopentadienyl complexes will be frequently observed, provided that the complexes bear groups that impart sufficient solubility to the partially mercurated intermediates.

Complex 1 serves as a versatile synthon for pentahalocyclopentadienyl ligands and affords the pentaiodo and pentabromo derivatives upon treatment with aqueous KX_3. Moreover, the structure of 3 provides the first structural data for the pentaiodocyclopentadienyl ligand. We are continuing to work on the synthesis of mercurated metallocenes, their halogenated derivatives, and mechanistic studies aimed at probing the electronic character of mercury substituents in cyclopentadienyl complexes. These studies will be published in due course.

Supplementary Material Available: Tables listing positional parameters, thermal parameters, complete bond lengths and angles, and full experimental details for data collection and refinement for $3\cdot0.25CCl_4$, text giving experimental details for the preparation of 1–3 and their spectroscopic and analytical data, and a chart illustrating all of the possible isomers for 1 (11 pages). Ordering information is given on any current masthead page.

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Structure and Bonding of *cis*-$[Ru(bpy)_2(CO)(C(O)OCH_3)](B(C_6H_5)_4)\cdot CH_3CN$ (bpy = 2,2′-Bipyridine)

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Summary: The molecular structure of the title compound as a model for $[Ru(bpy)_2(CO)(C(O)OH)]^+$ was determined in order to elucidate the structural difference between $[Ru(bpy)_2(CO)(C(O)OH)]^+$ and $[Ru(bpy)_2(CO)(\eta^1\text{-}CO_2)]$, both of which are possible reaction intermediates in electrochemical and photochemical CO_2 reduction.

Electrochemical[2] and photochemical[3] CO_2 reductions catalyzed by transition-metal complexes are of much interest in connection with the utilization of carbon resources. Elucidation of the bonding modes of CO_2 to those metal centers may afford fundamental information on the activation of CO_2 on the metal atoms. Among the η^1-,[4] η^2-,[5] μ_2-,[6] and μ_3-CO_2[7] metal complexes reported so far, η^1-CO_2

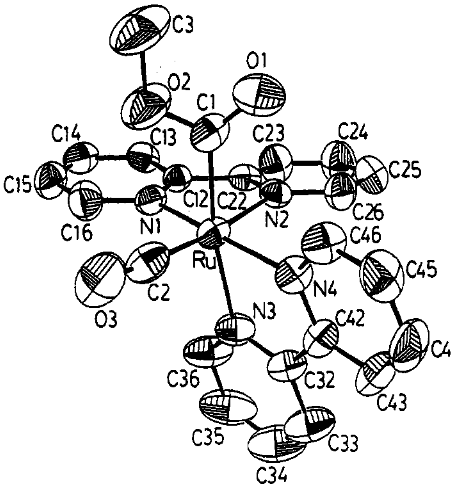

Figure 1. Molecular structure of $[Ru(bpy)_2(CO)(C(O)OCH_3)]^+$. Selected bond distances (Å) and angles (deg): Ru–C1, 2.042 (6); Ru–C2, 1.800 (7); Ru–N1, 2.070 (5); Ru–N2, 2.105 (5); Ru–N3, 2.151 (5); Ru–N4, 2.093 (5); C1–O1, 1.191 (8); C1–O2, 1.344 (8); O2–C3, 1.463 (9); C2–O3, 1.154 (8); Ru–C1–O1, 125.4 (5); Ru–C1–O2, 115.4 (4); O1–C1–O2, 119.2 (6); C1–O2–C3, 116.4 (6); Ru–C2–O3, 176.5 (6).

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