

by an approximately tetrahedral array of four carbon atoms, each of which in turn occupies an apical position in one of the two $C_2B_8H_{10}$ carborane moieties. The four aluminum-carbon interatomic distances average 2.06 Å while the eight aluminum-boron distances average 2.53 Å.

The bonding in $[2]^-$ can be rationalized in a manner similar to that applied to complex 1.¹ The geometry and interatomic distances in $[2]^-$ suggest that the aluminum atom participates in four electron precise bonds with the nearby tetrahedrally disposed carbon atoms. The relatively long aluminum-boron distances are indicative of a lack of bonding interaction between aluminum and boron. In this bonding description, each of the 6,9- $C_2B_8H_{10}$ cluster fragments in $[2]^-$ are formally regarded as dianionic *nido*-carborane cages, each of which donate four electrons via two carbon-based orbitals to sp^3 hybrid orbitals of the formal aluminum(III) metal center having suitable symmetry. It may be noted that this bonding description allows the $[2]^-$ anion to be regarded as a spiro-aluminate complex.

The mechanism by which $[2]^-$ is formed is not known but may involve successive loss of NaCl and cleavage of the two ethyl groups from aluminum concomitant with deprotonation of each carborane cage resulting in formation of ethane. Migration of the carbon atoms to the 6,9-positions of the $C_2B_8H_{10}$ cage is, as with compound 1, the expected thermal rearrangement for this system.⁷ This rearrangement also appears to result in the most favorable arrangement of carbon-based orbitals for bonding to the aluminum center. Continuous heating of Na[2] in refluxing toluene for 3 weeks resulted neither in decomposition nor further rearrangement.

Synthesis of other new aluminacarborane compounds are under investigation and will be reported later.

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Supplementary Material Available: Tables of positional and thermal parameters and interatomic distances and angles (12 pages); a listing of observed and calculated structure factors (10 pages). Ordering information is given on any current masthead page.

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Formation of Metallo Hydride, Formyl, and Alkyl Complexes of Rh(TMTAA)

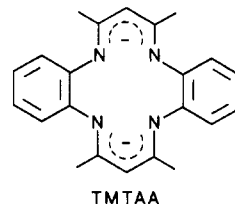
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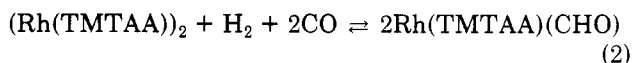
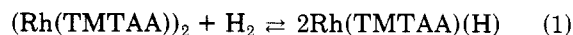
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Summary: The dimeric complex $(Rh^{II}(TMTAA))_2$ (1) (TMTAA = dibenzotetramethylaza[14]annulene dianion) reacts with mixtures of hydrogen and carbon monoxide gases to produce a metallo hydride, $Rh^{III}(TMTAA)(H)$ (2), and an η^1 -metallo formyl complex, $Rh^{III}(TMTAA)(CHO)$. Compounds 1 and 2 react with ethene to produce bridging and terminal alkyl complexes, respectively.

Reactions of metallo hydrides with carbon monoxide that result in the transfer of hydrogen to the carbon center have been accomplished with the transition-metal hydride species $(Cp^*_2)V(H)$,¹ $(Cp^*_2)Zr(H)_2$,² $Rh(\text{porphyrin})(H)$,³ and actinide complexes $(Cp^*_2)Th(OR)(H)^4$ [$Cp^* = \eta^5-C_5(CH_3)_5$]. Metal-site catalyzed hydrogenation of CO to organic products is thought to require the formation of a metallo hydride complex, through the reaction of molecular hydrogen, and the formation of either an η^1 -carbon-bonded or η^2 -carbon- and oxygen-bonded, metallo formyl complex.⁵ Rhodium porphyrin dimer complexes are at present the only molecular species reported to react with hydrogen and carbon monoxide to produce both the metallo hydride and metallo formyl complexes.³ This communication reports on several key features of the organometallic chemistry of a Rh-Rh bonded macrocyclic complex, $(Rh(TMTAA))_2$



(TMTAA = dibenzotetramethylaza[14]annulene dianion), including the reactions with H_2 and CO to produce both metallo hydride and η^1 -formyl complexes (reactions 1 and 2).



$(Rh(TMTAA))_2$ (1) is prepared by refluxing rhodium acetate dimer with the free base ligand H_2TMTAA ⁶ in ethanol (24 h) and collecting the precipitate by filtration under nitrogen gas. The product obtained is an ethanol solvate and can be converted to the ethanol free form by recrystallization from benzene followed by vacuum drying. Complex 1 has been characterized by 1H NMR as well as mass spectral analyses.⁷

Solutions of 1 (5×10^{-3} M) in THF- d_8 react with hydrogen gas ($P = 0.2-0.8$ atm), over a period of 48 h at 298 K, to form a monomeric metallo hydride complex, $Rh(TMTAA)(H)$ (2) (reaction 1). Formation of 2 is conveniently observed by the appearance of a diagnostic high-field doublet in the 1H NMR ($\delta_{Rh-H} -20.2$, $J_{103Rh-1H} = 47.6$ Hz).⁸

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(7) (a) 1H NMR (C_6D_6): $\delta +7.09$ (d of d, 8 H, *m*-phenyl), $+6.76$ (d of d, 8 H, *o*-phenyl), $+3.68$ (s, 4 H, methene), $+2.00$ (s, 24 H, methyl). MS: m/e 890 (exptl) vs. 890.6 (calcd) (M^+). (b) Footnote in ref 6 alludes to an unpublished structure determination.

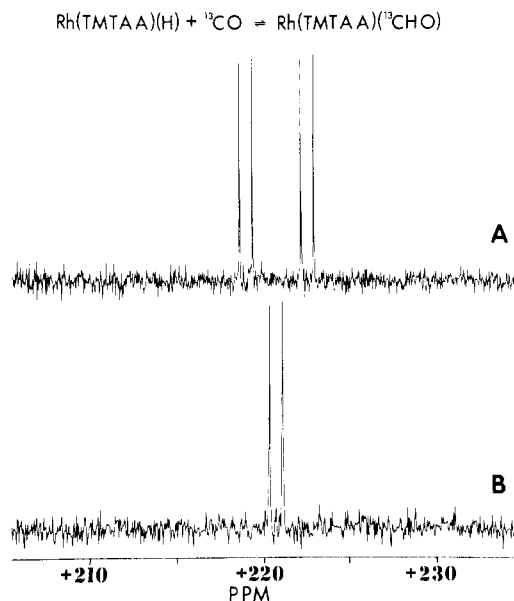


Figure 1. ^{13}C NMR of the formyl group in the $\text{Rh}(\text{TMTAA})({}^{13}\text{CHO})$ complex: (A) undecoupled ^{13}C NMR spectrum in $\text{THF-}d_8$ (δ_{CHO} 220.1, $J_{^{13}\text{C}-^1\text{H}} = 178.2$ Hz, $^1J_{^{103}\text{Rh}-^{13}\text{C}} = 36.2$ Hz); (B) ^1H -decoupled ^{13}C NMR spectrum in $\text{THF-}d_8$.

Removal of excess hydrogen gas results in the reformation of **1** and demonstrates the reversibility of reaction 1.

Solutions of $(\text{Rh}(\text{TMTAA}))_2$ in $\text{THF-}d_8$ react with CO and H_2 gases ($P_{\text{CO}} = 0.4$ atm; $P_{\text{H}_2} = 0.3$ atm) to produce the formyl complex $\text{Rh}(\text{TMTAA})(\text{CHO})$ which is formulated as an η^1 -carbon-bonded formyl on the basis of ^1H NMR (δ_{CHO} 11.9), ^{13}C NMR ($\delta_{^{13}\text{CHO}}$ 220.1, $J_{^{13}\text{C}-^1\text{H}} = 178.2$ Hz, $J_{^{103}\text{Rh}-^{13}\text{C}} = 36.2$ Hz; Figure 1a), and IR spectra of the solid ($\nu_{\text{CO}} = 1697$ cm^{-1}).⁹ Removal of excess hydrogen and carbon monoxide gases results in the reformation of **1** and demonstrates the reversibility of reaction 2. The metallo formyl complex can also be prepared by using water as a source of hydrogen (reaction 3). Using ^{13}CO in reaction $(\text{Rh}(\text{TMTAA}))_2 + 3\text{CO} + \text{H}_2\text{O} \rightarrow 2\text{Rh}(\text{TMTAA})(\text{CHO}) + \text{CO}_2$ (3)

3 results in observation by ^{13}C NMR of both the ^{13}C -labeled metallo formyl and $^{13}\text{CO}_2$ ($\delta_{^{13}\text{CO}_2}$ 124) consistent with the occurrence of a water gas shift type reaction.

Solutions of **1** in pyridine when exposed to H_2 gas ($P = 0.6$ atm) react to form a hydride complex with NMR parameters ($\delta_{\text{Rh-H}} -13.6$, $J_{^{103}\text{Rh}-^1\text{H}} = 35.7$ Hz). The magnitude and sign of the changes in the ^1H NMR parameters for the hydride complex in pyridine from those observed in THF ($\delta_{\text{Rh-H}} -20.2$, $J_{^{103}\text{Rh}-^1\text{H}} = 47.6$ Hz) are comparable to those observed for rhodium porphyrin hydride species¹⁰ and are compatible with the formation of a six-coordinate pyridine complex, $\text{Rh}(\text{TMTAA})(\text{py})(\text{H})$. Solutions of $\text{Rh}(\text{TMTAA})(\text{H})$ in pyridine react with carbon monoxide ($P_{\text{CO}} = 0.5$ atm) to form a metallo formyl complex (δ_{CHO} 12.9).

Organometallic reactions of the $\text{Rh}(\text{TMTAA})$ system, producing metallo alkyl complexes, closely parallel the reactions observed with the rhodium porphyrin system.¹¹

(8) (a) ^1H NMR ($\text{THF-}d_8$) δ +6.84 (d of d, 4 H, *m*-phenyl), +6.45 (d of d, 4 H, *o*-phenyl), +4.51 (s, 2 H, methene), +2.13 (s, 12 H, methyl), -20.3 (d, 1 H, hydride). (b) IR (KBr): $\nu_{\text{Rh-H}}$ 2121 cm^{-1} .

(9) (a) ^1H NMR ($\text{THF-}d_8$) δ +6.99 (d of d, 4 H, *m*-phenyl), +6.60 (d of d, 4 H, *o*-phenyl), +4.62 (s, 2 H, methene), +2.22 (s, 12 H, methyl), +11.93 (s, 1 H, formyl). (b) IR (KBr): $\nu_{\text{C-H}}$ 2681 cm^{-1} , $\nu_{\text{C=O}}$ 1697 cm^{-1} .

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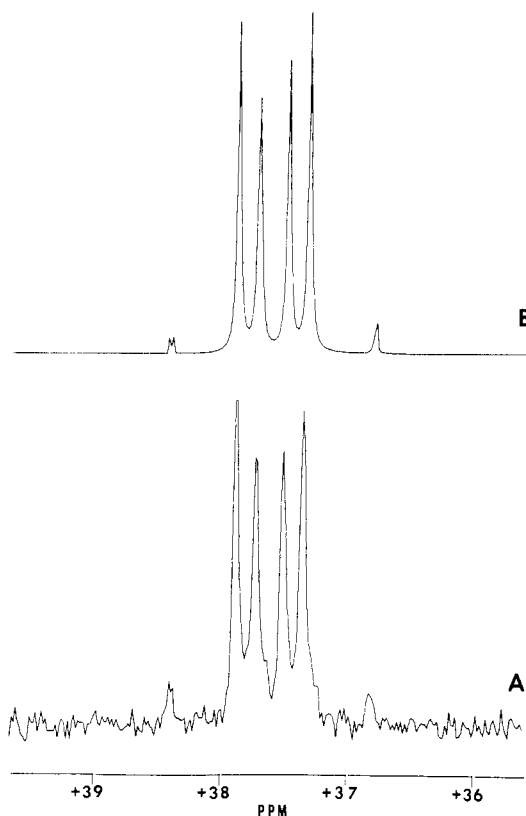
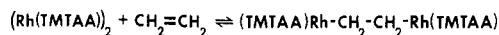
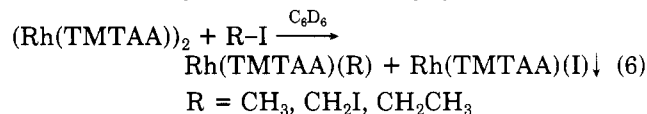


Figure 2. ^{13}C NMR of the ethylene bridge in the $(\text{TMTAA})\text{Rh}-\text{CH}_2-\text{CH}_2-\text{Rh}(\text{TMTAA})$ complex: (A) observed ^1H -decoupled ^{13}C NMR spectrum; (B) simulated ^1H -decoupled ^{13}C NMR spectrum ($\delta_{^{13}\text{C}}$ 37.6, $^1J_{^{103}\text{Rh}-^{13}\text{C}} = 28.7$ Hz, $^2J_{^{103}\text{Rh}-^{13}\text{C}} = -2.2$ Hz, $^1J_{^{13}\text{C}-^{13}\text{C}} = 33.7$ Hz, $^3J_{^{103}\text{Rh}-^{103}\text{Rh}} = 0.3$ Hz).

Ethene ($P_{\text{C}_2\text{H}_4} = 0.3$ – 0.8 atm) reacts reversibly with **1** to produce an alkyl-bridged complex (reaction 4).¹² The $(\text{Rh}(\text{TMTAA}))_2 + \text{CH}_2=\text{CH}_2 \rightleftharpoons (\text{TMTAA})\text{Rh}-\text{CH}_2-\text{CH}_2-\text{Rh}(\text{TMTAA})$ (4)

^1H -decoupled ^{13}C NMR spectrum of this complex, prepared with ^{13}C ethene, appears as an AA'XX' multiplet (Figure 2a) and can be simulated by using the following parameters: $\delta_{^{13}\text{C}}$ 37.6, $^1J_{^{103}\text{Rh}-^{13}\text{C}} = 28.7$ Hz, $^2J_{^{103}\text{Rh}-^{13}\text{C}} = -2.2$ Hz, $^1J_{^{13}\text{C}-^{13}\text{C}} = 33.7$ Hz, $^3J_{^{103}\text{Rh}-^{103}\text{Rh}} = 0.3$ Hz (Figure 2b). The metallo ethyl complex can be prepared by reacting **1** with a mixture of hydrogen and ethene gases ($P_{\text{H}_2} = 0.3$ atm; $P_{\text{C}_2\text{H}_4} = 0.4$ atm) (reaction 5). Alkyl complexes can $(\text{Rh}(\text{TMTAA}))_2 + \text{H}_2 + 2\text{CH}_2=\text{CH}_2 \rightarrow 2\text{Rh}(\text{TMTAA})(\text{CH}_2\text{CH}_3)$ (5)

be alternatively prepared by reacting stoichiometric amounts of alkyl iodide with **1** in C_6D_6 (reaction 6).¹³



(12) ^1H NMR (C_6D_6): δ +6.82 (d of d, 8 H, *m*-phenyl), +6.61 (d of d, 8 H, *o*-phenyl) +4.53 (s, 4 H, methene), +1.97 (s, 24 H, methyl), +3.75 (m, 4 H, bridging ethylene).

(13) (a) $\text{Rh}(\text{TMTAA})(\text{C}_6\text{H}_5)$: ^1H NMR (C_6D_6) δ +6.96 (d of d, 4 H, *m*-phenyl), +6.63 (d of d, 4 H, *o*-phenyl), +4.71 (s, 2 H, methene), +2.09 (s, 12 H, methyl), +3.08 (q of d, 2 H, CH_2-CH_3), +0.75 (t of d, 3 H, CH_2-CH_3); $J_{^{103}\text{Rh}-\text{CH}_2\text{CH}_3} = 3.4$ Hz, $J_{^{103}\text{Rh}-\text{CH}_2\text{CH}_3} = 2.5$ Hz. (b) $\text{Rh}(\text{TMTAA})(\text{CH}_2\text{I})$: ^1H NMR (C_6D_6) δ +6.93 (d of d, 4 H, *m*-phenyl), +6.61 (d of d, 4 H, *o*-phenyl), +4.04 (s, 2 H, methene), +2.06 ppm (s, 12 H, methyl), +2.20 (d, 3 H, $\text{Rh}-\text{CH}_3$); $J_{^{103}\text{Rh}-\text{CH}_3} = 3.6$ Hz. (c) $\text{Rh}(\text{TMTAA})(\text{CH}_2\text{I})$: ^1H NMR (C_6D_6) δ +6.97 (d of d, 4 H, *m*-phenyl), +6.65 (d of d, 4 H, *o*-phenyl), +4.72 (s, 2 H, methene), +2.07 (s, 12 H, methyl), +5.22 (d, 2 H, $\text{Rh}-\text{CH}_2\text{I}$); $J_{^{103}\text{Rh}-\text{CH}_2} = 3.4$ Hz.

Both the porphyrin and the TMTAA ligand systems are tetradentate N_4 -macrocycles that coordinate in the form of dianions. While the porphyrin dianion is a planar 18π -electron aromatic species, TMTAA is a 16π -electron antiaromatic dianion that can significantly deviate from planarity by rotation about the N-C bonds of the *o*-phenylenediamine portion of the macrocycle.¹⁴ Close parallels between the organometallic chemistry of $(Rh-(TMTAA))_2$ and $(Rh(Por))_2$ indicate that the conjugation and planarity of the porphyrin system are not essential features for the unusual reactivity that occurs at the rhodium center. The observed reaction of $Rh-(TMTAA)(py)(H)$ in pyridine with CO to produce a metallo formyl complex further extends the scope of complexes capable of this type of reaction to include six-coordinate rhodium(III) hydride species. The general reaction of a metal hydride with CO to produce observable concentrations of an η^1 -carbon-bonded formyl complex is rare because of the thermodynamic requirement that the (M-H)-(M-C) bond energies must be relatively small (<10 kcal).^{10,15} Accumulating evidence suggests that a large class of rhodium(III) hydride complexes may fulfill this thermodynamic criterion and could be effective in producing the initial steps in the hydrogenation of carbon monoxide.

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Synthesis of Chelating Ditelluroether Ligands, $RTeCH_2CH_2CH_2TeR$ (R = Me, Ph)

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Summary: $RTeLi$ (R = Me, Ph), prepared in situ in tetrahydrofuran from RLi and Te, react at low temperatures with 1,3-dihalopropanes, $X(CH_2)_3X$ (X = Cl, Br), to give high yields of $RTe(CH_2)_3TeR$. At ambient or higher temperatures these reactions yield mainly R_2Te_2 . The $RTeCH_2CH_2CH_2TeR$ are made analogously, but 1,2-dichloroethane gave only R_2Te_2 .

Despite the recent interest in organotellurium chemistry and its applications in synthesis,¹ nuclear medicine,² photography,³ and new conducting materials,⁴ the coordination chemistry of tellurium ligands remains underdeveloped in comparison with the selenium and sulfur analogues.⁵ We were interested in extending our recent studies⁶ on diselenoether complexes of the platinum metals

and ⁷⁷Se NMR spectroscopy to the tellurium analogues. From the coordination chemists viewpoint, ligands with two- or three-carbon backbones leading to stable five- or six-membered chelate rings are preferable, but for tellurium all attempts to prepare $RTe(CH_2)_nTeR$ ($n = 2, 3$) are reported to have failed. In particular reaction of $RTeM$ (M = alkali metal) with $X(CH_2)_nX$ (X = Cl, Br; $n = 2, 3$) at room temperature or above gave either R_2Te_2 + olefin or telluronium salts $RTe(CH_2)_nX$.⁷

We have found that $RTeLi$ (R = Me, Ph) are very conveniently obtained by an analogous method to that described for $MeSeLi$,⁶ essentially adding RLi to a frozen mixture of tetrahydrofuran (THF) and elemental tellurium and allowing the mixture to warm to room temperature. If 1,3-dichloropropane is subsequently added at room temperature or above, the product is R_2Te_2 , consistent with literature reports. However, if the THF solution is refrozen (-196 °C), then treated with 1,3- $Cl(CH_2)_3Cl$, and allowed to warm slowly, after hydrolysis, drying, and removal of the solvent in vacuo the $RTe(CH_2)_3TeR$ are obtained in high yield⁸ (85%, R = Ph; 73%, R = Me) and acceptable purity. The products appear to be stable at ambient temperatures. Characterization was by ¹H, ¹³C, and ¹²⁵Te NMR and mass spectra⁹ and by the preparation of derivatives.¹⁰ In particular the ¹²⁵Te NMR chemical shifts of $\delta +104$ (R = Me) and $+465$ (R = Ph) should be compared with those for $MeTe-n-Pr$ and $PhTe-n-Pr$ ($\delta +95$ and 460 , respectively).¹¹

Similar reactions of $RTeLi$ with CH_2Cl_2 provide good yields of $RTeCH_2TeR$, which have been independently obtained recently from R_2Te_2 and CH_2N_2 ,¹² $\delta(^{125}Te)$ 212 (R = Me) and 583.5 (R = Ph) (lit.¹² δ 213.5 and 587.6, respectively), but 1,2-dichloroethane affords only R_2Te_2 and C_2H_4 even at low temperatures. The greatest tendency to elimination is also observed with 1,2-dichloroalkanes and the corresponding element of group VB (15¹⁴) (antimony), although the $R_2Sb(CH_2)_nSbR_2$ ($n = 1, 3$; R = Me, Ph) can be prepared.¹³

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(8) $PhTe(CH_2)_3TePh$. Under a dinitrogen atmosphere, dry tetrahydrofuran (100 cm³) was distilled onto tellurium powder (12.8 g, 0.1 mol) and frozen (-196 °C). Phenyllithium (55.6 cm³, 1.8 M, 0.1 mol) was syringed in and the mixture allowed to thaw. When all the Te had dissolved, the clear solution was refrozen (-196 °C) and 1,3-dibromopropane (5.1 cm³, 0.05 mol) added. The mixture was allowed to warm to room temperature and hydrolyzed (saturated aqueous NaCl solution), the organic phase separated and dried ($MgSO_4$), and the solvent removed in vacuo to leave a pale orange oil. $MeTe(CH_2)_3TeMe$ was made similarly from $MeLi$.

(9) $PhTe(CH_2)_3TePh$: ¹H NMR 2.2 (q, 2 H), 2.85 (t, 4 H), 6.9-7.8 ppm (m, 10 H); ¹³C{¹H} NMR 138.2, 128.9, 127.3, 111.7, 33.2, 10.5 ppm (¹J_{Te-C} = 158 Hz); ¹²⁵Te{¹H} NMR +465 ppm; mass spectrum (only ¹⁸⁰Te peak quoted), *m/z* 456 (M), 414 (M - (CH₂)₃), 379 (M - Ph), 337 (PhTe₂). $MeTe(CH_2)_3TeMe$: ¹H NMR 1.85 (s, 6 H), 2.05 (q, 2 H), 2.6 ppm (t, 4 H); ¹³C{¹H} NMR 33.6, 5.2 (¹J_{Te-C} = 153 Hz), -23.3 ppm (¹J_{Te-C} = 162 Hz); ¹²⁵Te{¹H} NMR +104 ppm. All in CDCl₃ relative to Me₄Si (¹H, ¹³C) or external neat TeMe₂ (¹²⁵Te).

(10) Dimethioides were prepared by stirring the ligand with MeI in acetone. $IPhMeTe(CH_2)_3TeMePhI$: ¹²⁵Te{¹H} NMR +609.7, +610.3 ppm. Anal. Calcd for C₁₇H₂₂I₂Te₂: C, 27.8; H, 3.0. Found: C, 28.3; H, 3.1. $IME_2Te(CH_2)_3TeMe_2I$: ¹²⁵Te{¹H} +494 ppm. Anal. Calcd for C₇H₁₈I₂Te₂: C, 13.7; H, 2.9. Found: C, 13.5; H, 3.0.

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