Synthesis and Reactivity of [TpRh(PPh$_3$)$_2$] (Tp = Hydridotris(pyrazol-1-yl)borate)

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Summary: The new complex [TpRh(PPh$_3$)$_2$], readily obtained from [RhCl(PPh$_3$)$_3$] and KTp, is a particularly convenient and versatile entry point into the organometallic chemistry of the "TpRh" fragment including complexes bearing alkyne, alkene, carbene, thiocarbamoyl, dithioalkoxycarbonyl, carbon disulfide, and vinyl ligands.

The complex [TpRuCl(PPh$_3$)$_2$] (Tp = hydridotris(pyrazol-1-yl)borate) is readily obtained from the simple reaction of [RuCl$_2$(PPh$_3$)$_3$] with KTp.[1] This useful complex provides a convenient entry point into the organometallic chemistry of the "TpRu" fragment, by virtue of the lability of the phosphine ligands.[2] Wishing to extend the chemistry of the Tp ligand within group 9, we have sought an easily accessible starting material. In marked contrast to the group 6 metals, the organometallic chemistry of the Tp scorpionate bound to rhodium has been somewhat slow to develop,[3] despite Graham's inspiring demonstration that the complex [Tp^{Me2}Ir(CO)$_2$] is capable of thermally activating hydrocarbons.[4] The principal point of access to the TpRh fragment has been [TpRh(η-C$_2$H$_4$)$_2$] (and related olefin complexes), obtained *via* the reaction of [Rh$_2$(μ-Cl)$_2$(η-C$_2$H$_4$)$_4$] with KTp.[5] Recently, we reported the synthesis and reactivity of [Rh(PPh$_3$)$_2$([9]aneS$_3$)]PF$_6$ from the simple reaction of Wilkinson's complex [RhCl(PPh$_3$)$_3$] with 1,4,7-trithiacyclononane.[6] This complex was the first well-defined sulfur macrocycle complex to be deployed in (and recovered from) a catalytic process. Herein we report (i) that the novel and related complex [TpRh(PPh$_3$)$_2$] (**1**) may be similarly obtained in high yield from the reaction of [RhCl(PPh$_3$)$_3$] with KTp, (ii) that **1** is a valuable synthon for developing the organometallic chemistry of the TpRh fragment, (iii) that **1** has potential for catalysis, and (iv) three structural studies on organometallic derivatives of **1**.

Combining equimolar amounts of [RhCl(PPh$_3$)$_3$] and KTp in a refluxing methanolic suspension provides the new complex [TpRh(PPh$_3$)$_2$] (**1**)[7] in 66% yield. Complex **1** is air sensitive in solution, providing [TpRh(O$_2$)(PPh$_3$)] (**2**) and initially PPh$_3$, which is then catalytically converted to OPPh$_3$ by **2** (Scheme 1). Under anaerobic conditions, however, **1** is indefinitely stable. One phosphine ligand in **1** is labile and is readily replaced by C$_2$(CO$_2$Me)$_2$ to provide the alkyne complex [TpRh{η^2-C$_2$(CO$_2$Me)$_2$}(PPh$_3$)] (**3**)[7] (Figure 1).[8] Considering the alkyne as a single ligand, the geometry at rhodium may

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(7) [RhCl(PPh$_3$)$_3$] (0.50 g) and K[HB(pz)$_3$] (0.14 g) were heated under reflux in degassed methanol (20 mL) for 30 min. The resulting yellow precipitate of **1** was filtered off, washed with methanol, and anaerobically recrystallized from a mixture of dichloromethane and methanol. Yield: 0.30 g (66%). Selected data for new complexes are as follows (IR, Nujol; NMR, CDCl$_3$, 25 °C; FAB-MS, nba matrix; satisfactory microanalytical data). **1**: IR, 2442, 2409 [ν(BH)] cm^{-1}; ^{1}H NMR, δ 5.75 [s(br), 1 H, BH], 5.77, 5.80, 5.95 [t × 3, 3 H, H^4(pz)], 6.54, 6.81 [d × 2, 2 H, H^{3,5}(pz)], 7.01−7.65 [m, 32 H, PC$_6$H$_5$ + 2 H^{3,5}(pz)], 8.07, 8.11 [d × 2, 2 H, H^{3,5}(pz)]; ^{31}P NMR, 52.3 [d, J(RhP) = 171 Hz] ppm; FAB-MS m/z 841 [M]$^+$. **2**: quantitative; IR, 2493 [ν(BH)], 883 [ν(RhO$_2$)] cm^{-1}; ^{1}H NMR, δ 4.89 [m(br), 1 H, BH], 5.90 [t, 2 H, H^4(pz)], 6.19 [dd, 1 H, H^4(pz)], 6.87 [d, 2 H, H^{3,5}(pz)], 7.33−7.69 [m, 19 H, C$_6$H$_5$ + 4 H^{3,5}-(pz)]; ^{31}P NMR, 37.0 [d, J(RhP) = 129 Hz] ppm; FAB-MS, m/z 609 [M − H]$^+$. **3**: 65%; IR, 2460 [ν(BH)], 1789, 1697, 1675 [ν(C=O): 1779, 1687 in CH$_2$Cl$_2$ solution] cm^{-1}; ^{1}H NMR, δ 3.5 [s(br), 1 H, BH], 3.63 [s, 6 H, CH$_3$], 5.95 [t, 2 H, H^4(pz)], 6.00 [dd, 1 H, H^4(pz),], 7.1−7.4 [m(br), 15 H, C$_6$H$_5$], 7.25 [d, 2 H, H^{3,5}(pz)], 7.38 [d, 1 H, H^{3,5}(pz)], 7.50 [dd, 1 H, H^{3,5}(pz)], 7.67 [d, 2 H, H^{3,5}(pz)] ppm; ^{13}C{^{1}H} NMR, δ 161.6 [CO$_2$], 98.7 [dd, RhC, J(PC) = 7.6, J(RhC) = 19.4 Hz], 51.8 [OCH$_3$] ppm; ^{31}P NMR, 45.3 [d, J(RhP) = 139 Hz] ppm; FAB-MS, m/z 721 [M]$^+$. **4**: 85%; IR, 2483 [ν(BH)], 1718 [ν(C=O)], 1575 [ν(C=O)] cm^{-1}; ^{1}H NMR, δ 3.26, 3.61 [s × 2, 6 H, CH$_3$], 5.35 [s(br), 1 H, RhC=CH], 5.84, 5.86 [t × 2, 2 H, H^4(pz)], 6.12 [dd, 1 H, H^4(pz)], 6.25 [d(br), 1 H, H^{3,5}(pz)], 7.06 [d, 1 H, H^{3,5}(pz)], 7.22−7.41 [m(br) x 2, 15 H, C$_6$H$_5$], 7.53 [m, 1 H, H^{3,5}(pz)], 7.60, 7.73, 7.90 [d × 3, 3 H, H^{3,5}(pz)] ppm; ^{31}P NMR, 24.0 [d, J(RhP) = 117 Hz] ppm; FAB-MS, m/z 721 [M − Cl]$^+$. **5**: 88%; IR, 2510, 2459 [ν(BH): 2483 in CH$_2$Cl$_2$ solution], 1167 [ν(SCS)] cm^{-1}; ^{1}H NMR, δ 4.6 [br, 1 H, BH], 5.91, 6.03, 6.04 [s × 3, 3 H, H^4(pz)], 6.87, 7.39, 7.52 [s × 3, 3 H, H^{3,5}(pz)], 7.25−7.52 [m, 15 H, C$_6$H$_5$], 7.63, 7.68, 7.77 [d × 3, 3 × H^{3,5}(pz)]; ^{13}C{^{1}H} NMR, 256.9 [dd, SCS, J(RhC) = 24.1 Hz, J(PC) = 6.2 Hz]; ^{31}P NMR, 36.3 [d, J(RhP) = 129 Hz] ppm; FAB-MS, m/z 578 [M + nba − PPh$_3$]$^+$. **6**: 73%; IR, 2498 [ν(BH)], 920 [ν(CS)] cm^{-1}; ^{1}H NMR, δ 1.27 [t, 3 H, CH$_3$], 3.37 [q, 2 H, CH$_2$], 4.6 [s(br), 1 H, BH], 6.15, 6.17, 6.20 [t × 3, 3 H, H^4(pz)], 6.85, 7.20, 7.68, 7.82, 7.93 [d × 5, 5 H, H^{3,5}(pz)], 7.00−7.70 [m, 16 H, C$_6$H$_5$ + H^{3,5}(pz)] ppm; ^{31}P NMR, 32.2 [d, J(RhP) = 112 Hz] ppm; FAB-MS, m/z 683 [M]$^+$. **7a**: 48%; IR, 2482 [ν(BH)] cm^{-1}; ^{1}H NMR, δ 4.2 [s(br), 1 H, BH], 0.96, 3.05 [s(vbr), 6 H, SCH$_3$], 6.20 [t, 1 H, H^4(pz)], 6.25 [t, 2 H, H^4(pz)], 7.60 [d, 1 H, H^{3,5}(pz)], 7.75 [d, 2 H, H^{3,5}(pz)], 7.77 [s(br), 2 H, H^{3,5}(pz)], 8.63 [s(br), 1 H, H^{3,5}(pz)]; FAB-MS, 676 [M]$^+$. **8**: 89%; IR, 2502 [ν(BH)], 1661 [ν-(NC)], 917 [ν(C−S)] cm^{-1}; ^{1}H NMR, δ 3.36, 3.63 [s × 2, 6 H, CH$_3$], 5.96, 6.14, 6.21 [t × 3, 3 H, H^4(pz)], 6.90, 7.18, 7.73, 7.88 [d × 4, 4 H, H^{3,5}(pz)], 7.10−8.00 [m, 17 H, C$_6$H$_5$ + 2 H^{3,5}(pz)]; ^{13}C{^{1}H} NMR, 208.1 [dd, SCN, J(RhC) = 31.2 Hz, J(PC) = 8.0 Hz], 50.1, 46.1 [s × 2, 2 × CH$_3$]; ^{31}P NMR, 33.2 [d, J(RhP) = 112 Hz] ppm; FAB-MS, m/z 666 [M]$^+$. **10**: ^{31}P NMR (C$_6$H$_6$), 58.3 [d, J(RhP) = 156 Hz (cf. ref 10: 57.9 ppm, 156 Hz in thf). **11**: 60%; IR, 2472 [ν(BH)] cm^{-1}; ^{1}H NMR, δ 1.87, 2.55 [m × 2, 8 H, CH$_2$], 3.93 [s(br), 4 H, HC=CH], 6.19 [t, 3 H, H^4-(pz)], 7.56, 7.74 [d × 2, 6 H, H^{3,5}(pz)] (cf. ref 11); FAB-MS, m/z 424 [M]$^+$.

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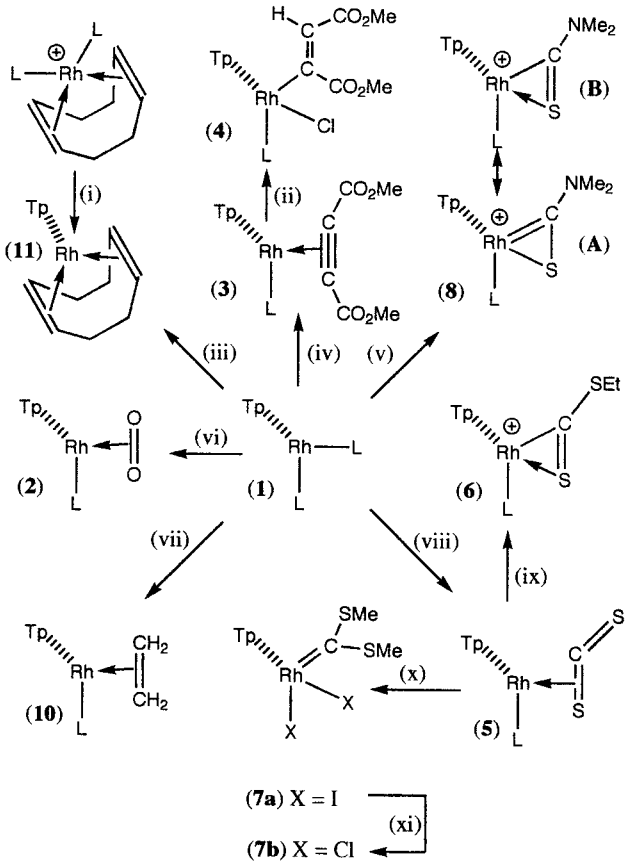
Scheme 1[a]



[a] Reagents and conditions (L = PPh_3, Tp = η^3-$HB(pz)_3$, 25 °C): (i) KTp; (ii) HCl; (iii) cod, toluene (110 °C); (iv) $C_2(CO_2Me)_2$; (v) $Me_2NC(=S)Cl$; (vi) O_2; (vii) C_2H_4; (viii) CS_2; (ix) $[Et_3O]BF_4$; (x) MeI; (xi) $CHCl_3$.

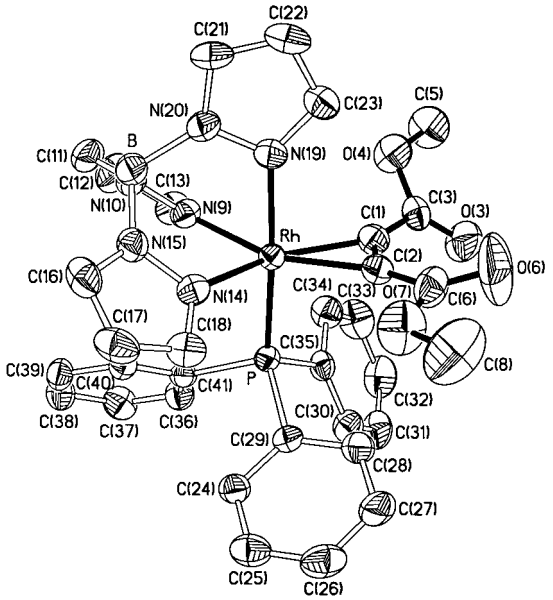


Figure 1. Molecular geometry of **3**. Phenyl substituents are omitted for clarity.

be described as trigonal bipyramidal with an axially coordinated phosphine ligand (Rh−P = 2.2628(11) Å) and an equatorially coordinated alkyne (Rh−C(1) 2.055(5) Å; Rh−C(2) = 2.040(5) Å). The two pyrazolyl groups trans to the alkyne have somewhat longer (10 and 16σ) Rh−N bond lengths (Rh−N(9) = 2.166(4); Rh−N(14) = 2.180(4) Å) than that trans to the phosphine (Rh−N(19) = 2.125(4) Å). Superficially, this might suggest a differential trans influence by the two ligands; however, such an interpretation is clouded by the observation that one *ortho* C−H group of a phenyl group is involved in a C−H···π interaction with the face of the pyrazolyl ring based on N(14) (C−H···π = 2.73 Å; C−H···π = 174°). Within the alkyne, one CO_2Me group aligns in conjugation with the alkyne, while the other (based on C(3)) lies at a dihedral angle of 45° to the Rh−C(1)−C(2) plane. The reaction of **3** with HCl provides the rhodium(III) σ-vinyl complex [Rh{C(CO_2Me)=CHCO_2Me}Cl(PPh_3){$HB(pz)_3$}] (**4**),[7] for which a cis arrangement of the two ester groups is assumed, consistent with (i) the lack of resolvable Rh−H or P−H coupling to the broadened ^{1}H NMR resonance for the vinylic proton and (ii) initial protonation at the rhodium center followed by insertion of the alkyne into the rhodium−hydride bond.

Complex **1** reacts readily with carbon disulfide to provide the adduct [TpRh(η^2-SCS)(PPh_3)] (**5**).[7] The desulfurization of CS_2 complexes by phosphines is a common, though not general, route to thiocarbonyl complexes. The complex **5** does *not*, however, react with excess PPh_3 in refluxing toluene to produce [TpRh(CS)(PPh_3)]; however, it is readily alkylated by $[Et_3O]BF_4$ to provide the dithioethoxycarbonyl salt [TpRh(η^2-SCSEt)(PPh_3)]BF_4 (**6**).[7] A similar reaction of **5** with methyl iodide, however, provides the red bis(thiolato)carbene complex [TpRh{=C($SMe)_2$}I_2] (**7a**) via a double-alkylation sequence, analogous to that of [Os(η^2-SCS)($CO)_2(PPh_3)_2$] with MeI.[9] The identity of **7a** was established from spectroscopic data;[7] however, attempts to obtain crystallographic grade crystals from chloroform solution led surprisingly to halide metathesis and formation of the structurally characterized yellow complex [TpRh{=C($SMe)_2$}Cl_2] (**7b**)[7] (Figure 2)[8]. This transformation may be effected on a preparative scale with excess $[Bu_4N]Cl$.

The geometry of **7b** at rhodium may be described as distorted octahedral with angles between cis ligands in the range 86.2(2)−93.65(8)°, the largest of these being between the two chloride ligands. The Rh−N bond lengths are contracted relative to those for **3**, consistent with the higher oxidation state of the central rhodium(III). Of these, that trans to the carbene ligand (Rh−N(13) = 2.107(6) Å) is significantly elongated (13σ)

(8) *Crystal data for* **3**: $C_{33}H_{31}N_6O_4BPRh$, M_r = 720.3, monoclinic, $P2_1/n$ (No. 14), a = 13.238(2) Å, b = 15.665(2) Å, c = 15.370(3) Å, β = 93.10(1)°, V = 3182.7(7) Å^3, Z = 4, D_c = 1.503 g cm^{-3}, μ(Cu−Kα) = 52.1 cm^{-1}, F(000) = 1472. An orange columnal needle of dimensions 0.60 × 0.18 × 0.18 mm was used. Crystal data for **7b**: $C_{12}H_{16}N_6BS_2Cl_2Rh$, M_r = 493.1, monoclinic, $P2_1/n$ (No. 14), a = 10.018(2) Å, b = 12.638(2) Å, c = 14.550(4) Å, β = 98.08(2)°, V = 1823.9(6) Å^3, Z = 4, D_c = 1.796 g cm^{-3}, μ(Mo Kα) = 14.7 cm^{-1}, F(000) = 984. A yellow prism of dimensions 0.22 × 0.18 × 0.13 mm was used. Crystal data for **8**: $C_{30}H_{31}N_7BPSClRh\cdot CHCl_3$, M_r = 821.2, triclinic, $P\bar{1}$ (No. 2), a = 10.563(2) Å, b = 12.845(2) Å, c = 13.987(1) Å, α = 96.59(1)°, β = 103.51(1)°, γ = 99.82(1)°, V = 1794.4(5) Å^3, Z = 2, D_c = 1.520 g cm^{-3}, μ(Mo Kα) = 9.10 cm^{-1}, F(000) = 832. A yellow plate of dimensions 0.40 × 0.33 × 0.17 mm was used. Totals of 4727/2389/6285 independent reflections were measured on Siemens P4/PC diffractometers with Cu Kα/Mo Kα/Mo Kα radiation using ω-scans for **3**/**7b**/**8**, respectively. The structures were solved by direct methods, and all of the major occupancy non-hydrogen atoms were refined anisotropically using full matrix least-squares based on F^2 to give R1 = 0.041/0.046/0.044 and wR2 = 0.093/0.110/0.096 for 3853/1852/4863 independent, observed, absorption-corrected reflections ($|F_o| > 4\sigma(|F_o|)$, $2\theta \le 120/45/45°$) and 380/218/391 parameters for **3**/**7b**/**8**, respectively.

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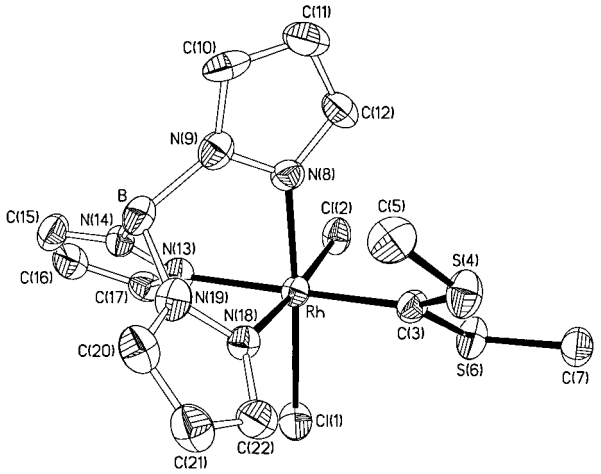

Figure 2. Molecular geometry of **7b**.

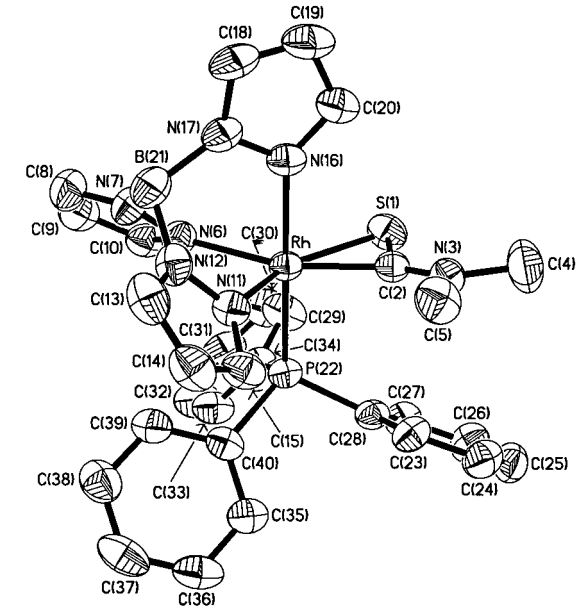

Figure 3. Molecular geometry for the cation of **8**. Phenyl substituents are omitted for clarity.

relative to those trans to the chloride ligands (Rh−N(8) = 2.027(6) Å; Rh−N(18) = 2.020(6) Å), consistent with the well-established trans influence of carbene ligands. The carbene ligand shows a comparatively long Rh−C(3) bond length of 2.009(8) Å, as might be expected for such a ligand bearing two π-dative substituents. This interpretation is supported by the observation that the bond lengths between sulfur and C(3) possess significant multiple-bond character, being clearly shorter (C(3)−S(4) = 1.692(8) Å; C(3)−S(6) = 1.689(8) Å), than those between the sulfur atoms and the methyl groups (S(6)−C(7) = 1.814(8) Å; S(4)−C(5) = 1.788(8) Å).

Although we have not yet obtained structural data for complex **6**, the analogous thiocarbamoyl salt [TpRh(η^2-SCNMe$_2$)(PPh$_3$)]Cl (**8**)[7] (Figure 3)[8] can be obtained via oxidative addition of *N*,*N*-dimethylthiocarbamoyl chloride to **1**. The pseudo-trigonal-bipyramidal geometry at rhodium is comparable to that of **3**, with the notable exception that the two pyrazolyl groups trans to the thiocarbamoyl ligand are now quite different (0.1 Å) in their bond lengths to rhodium. The pyrazolyl group which is pseudo-trans to (π-acidic) C(2) is considerably displaced (Rh−N(6) 2.174(4) Å) relative to N(11) (Rh−N(11) = 2.076(3) Å) which is pseudo-trans to the π-basic sulfur donor S(1), while Rh−N(16) at 2.116(4) Å is intermediate. The structural features of the thiocarbamoyl ligand comprise (i) a Rh−C(2) bond length of 1.945(4) Å which is clearly multiple in nature (cf. 2.009(8) Å for the carbene ligand in **7b**), (ii) a short C(2)−S(1) bond length of 1.685(5) Å, (iii) a short C(2)−N(3) bond length of 1.282(6) Å, consistent with the π-dative role of the trigonal N(3), and (iv) a Rh−S(1) bond length of 2.3943(13) Å. These data taken together indicate that both the "metallathiirene" (**A**, Scheme 1) and bidentate thioacyl (**B**, Scheme 1) resonance descriptions contribute to the conjugated system.

The reaction of **1** with hydrogen was investigated and found not to provide [TpRhH$_2$(PPh$_3$)] (**9**)[10] but rather a complex mixture of species (^{31}P NMR), which is currently under investigation. The ethene complex [TpRh(η-C$_2$H$_4$)(PPh$_3$)] (**10**) results from **1** and ethene, thus illustrating why **1** is not synthetically accessible from **10**. The reaction of **1** with 1,4-cyclooctadiene (cod), however, results in loss of both phosphines and formation of [Rh(cod){HB(pz)$_3$}] (**11**),[7,11] which may also be obtained from [Rh(cod)(PPh$_3$)$_2$]PF$_6$ and K[HB(pz)$_3$].

The complex [Rh(PPh$_3$)$_2$([9]aneS$_3$)]PF$_6$ (**12**) catalyzes the demercuration of bis(alkynyl)mercurials to provide 1,3-diynes in refluxing thf.[6] We find that **1** will effect the same catalysis at ambient temperatures. While **12** could be recovered after the catalysis, ^{31}P NMR investigation of the post-catalysis mixture from **1** (5 mol %) and [Hg(C≡CC$_6$H$_4$Me)$_2$] revealed the presence of at least six as yet unidentified rhodium phosphine complexes, including **2**. This might suggest that while the two mechanisms are globally similar, there may be subtle differences in the nature of the catalytically active species and the relative facilities of related steps in the two processes.

The above results together illustrate that the complex **1** is both conveniently accessible and highly versatile and should greatly facilitate the development of [hydridotris(pyrazolyl)borato]rhodium chemistry. Already **1** has provided access to the first TpRh complexes bearing carbon disulfide, alkyne, carbene, thiocarbamoyl, and dithioalkoxycarbonyl ligands. Furthermore, **1** has been successfully deployed in two catalytic processes, *viz.* (a) the catalytic demercuration of bis(alkynyl)mercurials under mild conditions and (b) the more mundane aerial oxidation of triphenylphosphine.

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Supporting Information Available: Figures giving additional views and tables giving crystal data and structure refinement details, atomic coordinates, thermal parameters, and bond distances and angles for **3**, **7b**, and **8** (19 pages). Ordering information is given on any current masthead page.

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