Table II. Observed and Calculated X-ray Diffraction Data for $\alpha\text{-LiAlO}_2$

hkl	d_{cal}, Å	d_{obs}, Å	I_{cal}	I_{obs}
003	4.73	4.75	85.3	84.0
101	2.39	2.40	19.0	18.6
006	2.36	2.37	0.0	0.0
012	2.29	2.30	0.0	0.0
104	2.00	2.01	100.0	100.0
015	1.843	1.845	3.6	3.9
009	1.576	1.577	0.3	0.0
107	1.555	1.555	11.4	11.9
018	1.431	1.431	23.2	22.9
110	1.400	1.399	23.2	23.2
113	1.342	1.341	8.4	8.3
1,0,10	1.224	1.222	0.6	0.6
021	1.208	1.206	1.7	1.8
116	1.204	1.202	0.8	0.8

BJH method.[7] The measurements were done by nitrogen adsorption/desorption with an Omnisorp-360 instrument interfaced with an IBM PC/AT computer, which automatically controlled the experiments followed by data acquisition, reduction, and display. Outgassing at 200 °C for 4 h was performed on each sample before the measurement. The Scherrer equation[8] was employed to obtain the average particle size. Results corresponding to different calcination conditions are shown in Table I. The lithium-based oxide $\alpha\text{-LiAlO}_2$ derived from the precursor, $\text{LiAl(OH)}_4\cdot n\text{H}_2\text{O}$ ($n \approx 1.0$), develops significant surface area and porosity that are stable with time and temperature.

Structural details of $\alpha\text{-LiAlO}_2$ have not been improved since an estimate of atomic positions based on X-ray diffraction photographs.[9] Powder data shown in Figure 1b were refined by the Rietveld method.[10,11] The results confirmed that $\alpha\text{-LiAlO}_2$ is isostructural with $\alpha\text{-NaFeO}_2$ with sheets of alternating $\text{LiO}_{6/6}$ and $\text{AlO}_{6/6}$ octahedra that result in a superlattice of the NaCl structure elongated along the 3-fold axis ($c/2a_{\text{obsd}} = 2.54$ vs $c/2a_{\text{cubic}} = 2.45$).

Powder data were obtained with Ni-filtered Cu Kα radiation on a Rigaku Geigerflex powder diffractometer over the range $10° \leq 2\theta \leq 80°$ in increments of 0.05°. The structure was determined with the full-matrix least-squares program DBW3.2.[12,13] $\alpha\text{-LiAlO}_2$ has the space group $R\bar{3}m$ (No. 166, hexagonal setting) with $a = b = 2.7993$ (5) Å, $c = 14.180$ (3) Å, and $Z = 3$. In the unit cell, lithium and aluminum cations take (3a) and (3b) positions, respectively, while the oxygen anions are in the special position (6c) with $z = 0.2406$ (3). This agrees with the estimated range 0.235–0.240 reported earlier.[9] A pseudo Voigt function, which is the sum of Gaussian and Lorentzian functions with adjustable mixing parameters, was employed for profile fitting. The refinement converged with background and half-width parameters as variables. Final *R* factors[12,13] were $R_p = 8.82\%$, $R_{wt} = 11.22\%$, and Bragg $R = 1.63\%$ ($R_{ex} = 9.11\%$, $R_{wt}/R_{ex} = 1.23$). Bragg reflections with their relative intensities are given in Table II.

Interatomic distances obtained are in good agreement with those reported in the literature.[9] All metal ions in $\alpha\text{-LiAlO}_2$ have coordination number 6 (D_{3d}) with equidistant oxygen neighbors. Bond distances predicted from the ionic radii of Shannon[14] for octahedral Li^+, Al^{3+}, and O^{2-} are Li–O = 2.16 Å and Al–O = 1.94 Å, compared well with the observed values 2.084 (1) and 1.926 (1) Å, respectively. In-plane oxygen–oxygen separations corresponding to the *a*-axis length are 2.799 (1) Å. As expected, there are slightly shorter interplane oxygen–oxygen distances of 2.647 (1) Å for the Al–O sheet with longer 3.087 (1) Å oxygen–oxygen separations in the Li–O sheet.

The compound precursor $\text{LiAl(OH)}_4\cdot n\text{H}_2\text{O}$ ($n \approx 1.0$) effectively overcomes the intrinsic barrier associated with lithium ion diffusion encountered when lithium salts react with alumina.[15] Decomposition results in high-surface-area $\alpha\text{-LiAlO}_2$ because of the short reaction time and relatively low temperature. Future work will explore the use of high-surface-area $\alpha\text{-LiAlO}_2$ as a catalyst support and for the preparation of delithiated transitional alumina compounds.[3]

Acknowledgment. This research was supported by the National Science Foundation, Solid State Chemistry (Grant DMR-8610659), and the Alcoa Foundation. We also thank Lorraine A. Townsend for her assistance with the BET/BJH measurements. The purchase of the instrument was made possible by DOE (Contract No. DE-FG05-86ER75295).

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Dinuclear Mixed Alkyl Phosphido and Alkyl Aryloxy Complexes of Manganese(II). Crystal Structures of $[\text{Mn(CH}_2\text{CMe}_3)(\mu\text{-}t\text{-Bu}_2\text{P})]_2$ and $[\text{Mn(CH}_2\text{CMe}_2\text{Ph})(\mu\text{-O-2,4,6-(}t\text{-Bu)}_3\text{C}_6\text{H}_2)]_2$

Sir:

Apart from binary (homoleptic) complexes,[1] most phosphido and arsenido (R_2E^-, E = P, As) complexes of the d-block transition metals also bear stabilizing ligands such as cyclopentadienyl, CO, phosphine, and NR_2^-.[2] During the course of our studies on the steric effects of bulky phosphido complexes of the d-block transition metals,[3] we examined reactions of the manganese(II) dialkyls

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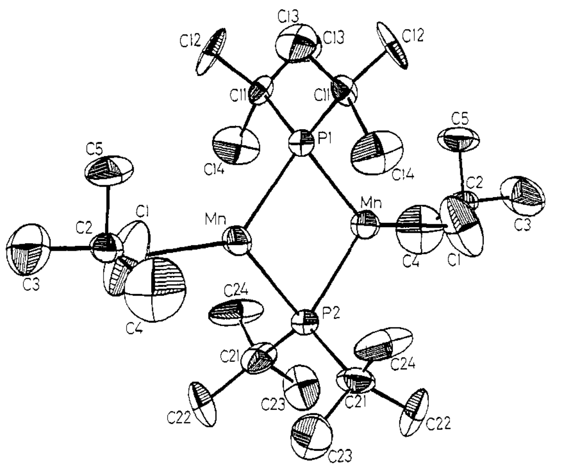

Figure 1. ORTEP diagram of $[Mn(CH_2CMe_3)(\mu\text{-}t\text{-}Bu_2P)]_2$ (**1**) showing the atom-numbering scheme. Key bond angles are as follows: P(1)–Mn–P(2) = 97.6 (1)°, Mn–P(1)–Mn = 81.1 (2)°, Mn–P(2)–Mn = 83.6 (2)°, Mn–C(1)–C(2) = 111.1 (1)°. See text for key bond lengths; a complete listing is given in the supplementary material.

$(MnR_2)_n$ (R = CH_2CMe_3, $n = 4$; CH_2CMe_2Ph, $n = 2$; CH_2SiMe_3, $n = \infty$), reported by Wilkinson and co-workers in 1976,[4] with the bulky secondary phosphine $t\text{-}Bu_2PH$. These reactions result in loss of alkane (RH) and give compounds of formula $[MnR(\mu\text{-}t\text{-}Bu_2P)]_2$, which, to our knowledge, are the first d-block transition-metal phosphido complexes that bear only σ-bound alkyl groups as ancillary ligands.

In order to probe the reactions of the manganese dialkyls further, we examined their reactivity with other protic sources. Reaction with HOAr (Ar = $2,4,6\text{-}(t\text{-}Bu)_3C_6H_2$) yields the analogous aryloxy-bridged complex $[MnR(OAr)]_2$ in high yield. These complexes represent the first mixed alkyl aryloxy complexes of Mn(II). For the compounds $[Mn(CH_2CMe_3)(\mu\text{-}t\text{-}Bu_2P)]_2$ (**1**) and $[Mn(CH_2CMe_2Ph)(OAr)]_2$ (**2**) the solid-state structures have been determined by single-crystal X-ray diffraction studies. These complexes are noteworthy for several reasons. Unlike the Mn(II) alkyls $[(MnR_2)_n]$ they contain three-coordinate Mn atoms with no Mn–Mn bonds. In addition, despite the very electron-deficient nature of the Mn atoms (each Mn has a formal electron count of 11), there appears to be no agostic CH···Mn interaction in either complex.[5] This may be due to the steric demands of the bridging $t\text{-}Bu_2P^-$ and OAr^- ligands.

The reaction of the dialkylmanganese reagents $(MnR_2)_n$ with di-*tert*-butyl phosphine ($t\text{-}Bu_2PH$) in refluxing hexane affords the red phosphido-bridged dimers $[MnR(\mu\text{-}t\text{-}Bu_2P)]_2$ in good yields.[6] They may be recrystallized from hexane and are extremely air-sensitive, in both solution and the solid state. There is no further reaction when excess phosphine is employed, presumably due to the steric demands of the reactants.

The neopentyl derivative $[Mn(CH_2CMe_3)(\mu\text{-}t\text{-}Bu_2P)]_2$ (**1**) crystallized in the monoclinic space group $C2$ (No. 5) with two dimers in the unit cell.[7] An ORTEP view of the molecule is shown in Figure 1. The two Mn atoms are separated by a nonbonding distance of 3.293 (2) Å and are part of a planar Mn_2P_2 core that results from a crystallographically imposed 2-fold axis of symmetry passing through the phosphorus atoms. The geometry of the three-coordinate Mn atoms is essentially trigonal planar, and the Mn–Mn separation is much longer than that found in $[Mn(CH_2CMe_2Ph)_2]_2$ (2.719 Å)[4] or in the phosphine-stabilized dimers $[MnR_2(PR_3)]_2$, in which the Mn–Mn distances range from 2.667 (1) to 2.828 (1) Å.[8] The terminal $-CH_2CMe_3$ units adopt a configuration in which the *t*-Bu moiety lies approximately in the Mn–Mn′–C(1)–C(1)′ plane. The Mn–C(1) distance of 2.28 (2) Å is somewhat longer that the $Mn\text{–}C_{terminal}$ distances in $[MnR_2(PR_3)]_2$,[8] where Mn–C distances range from 2.111 (3) to 2.19 (3) Å. It is also longer than that found in the monomeric Mn(II) alkyl $Mn(CH_2CMe_2Ph)_2(PMe_3)_2$ (2.149 (6) Å).[9] The longer Mn–C bond in **1** may be due to the steric effects of the bulky bridging $t\text{-}Bu_2P^-$ units.

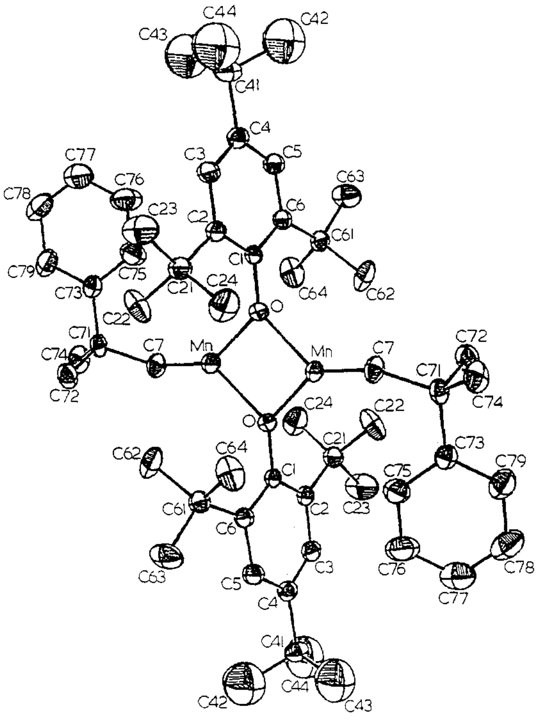

Figure 2. ORTEP diagram of $[Mn(CH_2CMe_2Ph)(\mu\text{-}O\text{-}2,4,6\text{-}(t\text{-}Bu)_3C_6H_2)]_2$ (**2**) showing the atom-numbering scheme. Key bond angles are as follows: O–Mn–O = 82.2 (2)°, Mn–O–Mn = 97.8 (2)°, O–Mn–C(7) = 144.0 (2)°, O–Mn–C(7) = 131.3 (3)°. See text for key bond lengths; a complete listing is given in the supplementary material.

The two Mn–P distances are significantly different (Mn–P(1) = 2.532 (4) Å, Mn–P(2) = 2.470 (4) Å). They are both considerably longer than those found in other dinuclear manganese phosphido complexes stabilized by CO ligands. For example, in $Mn_2(\mu\text{-}H)(\mu\text{-}Cy_2P)(CO)_7(Cy_2PH)$ and $Mn_2(\mu\text{-}H)(\mu\text{-}Cy_2P)(CO)_6(PMe_3)_2$ the $Mn\text{–}P_{bridge}$ distances range from 2.267 (3) to 2.312 (4) Å.[10] Also, we note that the methyl groups C(5) and C(5)′ of the $-CH_2C\mathit{Me}_3$ units occupy the P(1) side of the molecule. It may be that the steric effects of these methyl groups are responsible, at least in part, for the differences in the Mn–P distances.

The bulky phenol $HO\text{-}2,4,6\text{-}(t\text{-}Bu)_3C_6H_2$ (HOAr) reacts readily with the dialkylmanganese reagents to give aryloxy derivatives analogous to the phosphido complexes. Thus, reaction of $[Mn(CH_2CMe_2Ph)_2]_2$ with HOAr at room temperature in hexane leads to a color change from light brown to light pink. Light pink air-sensitive crystals of $[Mn(CH_2CMe_2Ph)(OAr)]_2$ (**2**) may be isolated in 80% yield following evaporation of the solvent to dryness and recrystallization of the residue from hexane at −20 °C. Crystals of **2** crystallize in the triclinic space group $P\bar{1}$ (No. 2) with one molecule per unit cell.[7] There is a crystallographically

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imposed inversion point at the center of the molecule. An ORTEP view of **2** is shown in Figure 2. The Mn–Mn distance of 3.113 (2) Å is again nonbonding, and the terminal alkyl groups and phenolic groups are arranged so as to minimize steric interactions. The aryl rings are at an angle of 64.7° to the Mn_2O_2 plane, and presumably this is the optimum geometry to maximize the distance between the ortho *t*-Bu groups and the neophyl units. The Mn–C(7) distance is 2.104 (8) Å, which is shorter than that found in **1**, while the Mn–O distances are similar at 2.058 (5) and 2.073 (4) Å. The latter bond length is very similar to the Mn–O distances in $Mn_3(OCH\text{-}t\text{-}Bu_2)_6$[11] and $Li\{Mn(N(SiMe_3)_2)(OC\text{-}t\text{-}Bu_3)_2\}$.[12]

Both complexes are paramagnetic (**1**, $\mu_{eff} = 3.1\ \mu_B$ per Mn atom; **2**, $\mu_{eff} = 5.2\ \mu_B$ per Mn atom; room temperature, Evans method). The lower magnetic moment for **1** suggests some degree of cooperative magnetic behavior through the bridging phosphorus atoms. The ESR spectra at 125 K in frozen toluene solutions of both **1** and **2** consist of a single broad signal (**1**, $g = 1.98$; **2**, $g = 1.98$); no fine coupling to P is observed in the spectrum of **1**. All compounds gave satisfactory elemental analysis (C, H).

Mention should also be made of the absence of agostic[5] CH···Mn interactions in both **1** and **2**. Although H atoms were not located in the X-ray structures of either **1** or **2**, there are no close Mn–C distances that might indicate the presence of an agostic C–H···Mn interaction.[13] Also, IR spectra of both complexes do not contain any particularly low ν_{C-H} bands in the 2700–2800 cm^{-1} region indicative of agostic interactions.[5] In most other Mn alkyl complexes in which such interactions occur, they involve bridging rather than terminal alkyl units.[4,8,9] In **1** and **2** the phosphido and aryloxy ligands clearly prefer bridging positions, forcing the alkyl units to occupy terminal sites. These alkyl groups are then prevented from close interaction with the metal centers by the steric bulk of the bridging units. Further studies are in progress.

Acknowledgment. We thank the National Science Foundation (Grant No. CHE-8517759), the Robert A. Welch Foundation (Grant No. F-816), and the Texas Advanced Technology Research Program for support. R.A.J. thanks the Alfred P. Sloan Foundation for a fellowship (1985–1989).

Supplementary Material Available: Tables of all bond lengths and angles, positional parameters, and thermal parameters and experimental details and crystal structure details for **1** and **2** (14 pages); tables of observed and calculated structure factors (24 pages). Ordering information is given on any current masthead page.

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(13) For **1** the closest nonbonded Mn–C distances (Å) are as follows: Mn–C(4), 3.20 (3); Mn–C(14), 3.62 (2); Mn–C(24), 3.69 (2). If hydrogen atoms are placed in idealized positions on these carbon atoms with C–H distances of 0.95 Å, then the closest Mn···H distance is 2.608 Å in Mn···H–C(4). For **2** the closest nonbonded Mn–C distances (Å) are as follows: Mn–C(22), 3.33 (1); Mn–C(24), 3.46 (1); Mn–C(62), 3.29 (9); Mn–C(64), 3.528 (9). If the hydrogen atoms are placed in idealized locations on these C atoms, the closest Mn···H distance is 2.360 Å in Mn···H–C(64).

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Sequential Synthesis of "FePt", "$FePt_2$", and "$FePt_3$" Complexes from the Reaction of $Fe(CO)_4(PR_2H)$ with $Pt(C_2H_4)_2(PCy_3)$. A Detailed Look at a Cluster Assembly Process

Sir:

While $Pt(C_2H_4)_n(PR_3)_{3-n}$ (n = 1–3) complexes are excellent precursors to a range of chemically interesting "Pt_x" and "M_yPt_x" cluster compounds,[1] very little is known with respect to the mechanisms by which these cluster compounds are assembled. Herein we report preliminary observations concerning the preferred reaction pathways in "FePt", "$FePt_2$", and "$FePt_3$" cluster synthesis from the reaction of $(OC)_4Fe(PR_2H)$ with $Pt(C_2H_4)_2(PCy_3)$—a system in which the serendipitous occurrence of appropriate relative rates and sequential isomerization processes provide considerable insight into and detail of a platinum cluster assembly process.

Oxidative addition of the P–H bond of $(OC)_4Fe(PR_2H)$[2], (**1**, R = Cy, Ph, *n*-Pr) to $Pt(C_2H_4)_2(PCy_3)$ in CD_2Cl_2 (ca. 20 °C) gives rise to a range of "FePt", "$FePt_2$", and "$FePt_3$" clusters depending on the initial Fe:Pt stoichiometry (Scheme I). Reaction of **1** with 1 molar equiv of $Pt(C_2H_4)_2(PCy_3)$ results in the rapid formation of $(OC)_4Fe(\mu\text{-}PR_2)PtH(PCy_3)$ (**2**), identified by ^1H and $^{31}P\{^1H\}$ NMR.[3] Complex **2**, which may be formulated as octahedral Fe (valency II) and square-planar Pt (valency II) linked by a phosphido bridge and a "bent" Fe–Pt bond, quickly rearranges (ca. 2–3 min, 20 °C) to the μ-hydrido complex **4**, presumably via the μ-carbonyl intermediate **3**[4] (not observed spectroscopically). Within 10–15 min the complex **4** equilibrates with the terminal (Fe–H) hydrido isomer **5**. (At equilibrium, 20 °C, in C_6D_6 the **2**:**4**:**5** ratio (μ-PPh_2) is ca. 1:3:6.) Particularly diagnostic of the terminal Fe–H structure in **5** is the rather small value of $^2J_{^{195}Pt-^{1}H}$ (ca. 15 Hz).[3,5] Complex **5** crystallizes from solution on addition of hexanes. When **5** is redissolved, the **5** ⇄ **4** ⇄ **2** equilibria are reestablished.

Reaction of **1** with 2 molar equiv of $Pt(C_2H_4)_2(PCy_3)$ in CD_2Cl_2 solution (an NMR study) gives rise to the same sequence of events documented above except that now the resonances due to **5** are of much lower intensity, suggestive of a "steady state concentration", and new signals assignable to the trinuclear "$FePt_2$" complex **6**, in which a CO bridges two platinums, are observed.[3] Complex **6** does *not* form prior to the formation of **5**. Isolated **5**, on redissolving in CD_2Cl_2, reacts rapidly with $Pt(C_2H_4)_2(PCy_3)$

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(3) Spectroscopic data for representative examples of "$FePt_x(\mu\text{-}PPh_2)$" complexes (Scheme I). **2**: ^1H NMR (hydrido region) $\delta(H)$ −5.3 ($J_{^{195}Pt-^{1}H}$ = 1770 Hz, $J_{^{31}P-^{1}H}$ = 24, 2 Hz); $^{31}P\{^1H\}$ NMR (relative to 85% H_3PO_4) $\delta(P_\mu)$ 96 ($J_{^{195}Pt-^{31}P_\mu}$ = 2140 Hz, $J_{^{31}P-^{31}P_\mu}$ = 311 Hz), $\delta(PCy_3)$ 41 ($J_{^{195}Pt-^{31}P}$ = 2920 Hz, $J_{^{31}P-^{31}P}$ = 311 Hz). **4**: IR (ν(CO) region, cm^{-1}) 2036 (m) (PtCO), 1976 (s), 1908 (m), 1895 (m); ^1H NMR (hydrido region) $\delta(H)$ −10.3 ($J_{^{195}Pt-^{1}H}$ = 525 Hz, $J_{^{31}P-^{1}H}$ = 35, 12 Hz); $^{31}P\{^1H\}$ NMR $\delta(P_\mu)$ 178 ($J_{^{195}Pt-^{31}P_\mu}$ = 1584 Hz, $J_{^{31}P-^{31}P}$ = 166 Hz), $\delta(PCy_3)$ 41 ($J_{^{195}Pt-^{31}P}$ = 2520 Hz, $J_{^{31}P-^{31}P}$ = 166 Hz). **5**: IR (ν(CO), cm^{-1}) 2037 (vs) (PtCO), 2000 (s), 1958 (m), 1940 (m); ^1H NMR (hydrido region) $\delta(H)$ −11.2 ($J_{^{195}Pt-^{1}H}$ = 16 Hz, $J_{^{31}P-^{1}H}$ = 59, 10 Hz); $^{31}P\{^1H\}$ NMR $\delta(P_\mu)$ 164 ($J_{^{195}Pt-^{31}P_\mu}$ = 2819 Hz, $J_{^{31}P-^{31}P_\mu}$ = 12 Hz), $\delta(PCy_3)$ 46 ($J_{^{195}Pt-^{31}P}$ = 3290 Hz, $J_{^{31}P-^{31}P}$ = 12 Hz). **6**: IR (ν(CO), cm^{-1}) 2014 (m), 1948 (s, br), 1753 (μ-CO); ^1H NMR (hydrido region) $\delta(H)$ −7.1 ($^1J_{^{195}Pt-^{1}H}$ = 580, $^2J_{^{195}Pt-^{1}H}$ = 75 Hz, $J_{^{31}P-^{1}H}$ = 10, 0, 0 Hz); $^{31}P\{^1H\}$ NMR $\delta(P_\mu)$ 282 ($^1J_{^{195}Pt-^{31}P_\mu}$ = 2032 Hz, $^2J_{^{195}Pt-^{31}P_\mu}$ = 131 Hz, $J_{^{31}P-^{31}P_\mu}$ = 18, 8 Hz), $\delta(PCy_3)$ (cis μ-PPh_2)) 71 ($^1J_{^{195}Pt-^{31}P}$ = 5480 Hz, $^2J_{^{195}Pt-^{31}P}$ = 320 Hz, $J_{^{31}P-^{31}P_\mu}$ = 18 Hz, $J_{^{31}P-^{31}P}$ = 18 Hz), $\delta(PCy_3$ (cis μ-H)) 53 ($^1J_{^{195}Pt-^{31}P}$ = 4360 Hz, $^2J_{^{195}Pt-^{31}P}$ = 230 Hz, $J_{^{31}P-^{31}P_\mu}$ = 8 Hz, $J_{^{31}P-^{31}P}$ = 18 Hz). **8**: IR (ν(CO), cm^{-1}) 2019 (m), 1950 (s), 1926 (s), 1755 (μ-CO); ^1H (hydrido region, −64 °C) $\delta(H)$ −7.3 ($^1J_{^{195}Pt-^{1}H}$ = 720 Hz, $^2J_{^{195}Pt-^{1}H}$ = 70 Hz, $J_{^{31}P-^{1}H}$ = 87, 20, 10 Hz); $^{31}P\{^1H\}$ NMR $\delta(P_\mu)$ 327 ($J_{^{195}Pt-^{31}P_\mu}$ = 2217, 1815 Hz, $J_{^{31}P-^{31}P_\mu}$ = 0, 0 Hz), $\delta(PCy_3$ (cis μ-H)) 42 $^1J_{^{195}Pt-^{31}P}$ = 3780 Hz, $^2J_{^{195}Pt-^{31}P}$ = 0 Hz, $J_{^{31}P-^{31}P_\mu}$ = 0 Hz, $J_{^{31}P-^{31}P}$ = 59 Hz), $\delta(PCy_3$ (cis μ-CO)) 55 ($^1J_{^{195}Pt-^{31}P}$ = 4032 Hz, $^2J_{^{195}Pt-^{31}P}$ = 70 Hz, $J_{^{31}P-^{31}P_\mu}$ = 0 Hz, $J_{^{31}P-^{31}P}$ = 59 Hz). **11**: IR (ν(CO), cm^{-1}, CH_2Cl_2) 1981 (s), 1931 (m), 1827 (m), 1780 (s); NMR $\delta(H)$ −18.4 ($J_{^{195}Pt-^{1}H}$ = 350 Hz, $^2J_{^{195}Pt-^{1}H} \approx$ 50 Hz (br resonances, $\Delta\delta_{1/2} \approx 50$ Hz), $\delta(P_\mu)$ 470 ($^1J_{^{195}Pt-^{31}P_\mu}$ = 1726, 1134 Hz, $^2J_{^{195}Pt-^{31}P_\mu}$ = 80 Hz, $J_{^{31}P-^{31}P_\mu}$ = 297, 7, 0 Hz), $\delta(PCy_3$ (1) (cis to μ-PPh_2)) 57 ($^1J_{^{195}Pt-^{31}P}$ = 3948 Hz, $^2J_{^{195}Pt-^{31}P}$ = 268, 0 Hz, $J_{^{31}P-^{31}P}$ = 39, 7 Hz), $\delta(PCy_3$ (2) (cis to μ-PPh_2)) 74 ($^1J_{^{195}Pt-^{31}P}$ = 4904 Hz, $^2J_{^{195}Pt-^{31}P}$ = 340, 0 Hz, $J_{^{31}P-^{31}P}$ = 39, 0, 0 Hz), $\delta(PCy_3$ (3)) 23 ($^uJ_{^{195}Pt-^{31}P}$ = 2885 Hz, $^2J_{^{195}Pt-^{31}P}$ = 0, 0 Hz, $J_{^{31}P-^{31}P}$ = 297, 0, 0 Hz). Anal. Calcd (found) for **11** (μ-PPh_2) ($C_{70}H_{110}FeO_4P_4Pt_3$): C, 47.19 (46.91); H, 6.18 (6.04).

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