distance to the second molecular unit of the dimer is slightly longer at 2.407 (4) Å. Additionally, the C(2)-Be-C(3) angle is distorted from the expected trigonal planar 120° to 117.1 (2)°, apparently due to the interaction with the lithium atom. The exact nature of this interaction is not understood. While the trigonal-planar arrangement of the alkyl groups around beryllium suggests that lithium has an electrostatic interaction with the organoberyllium anion, the fact that the compound sublimes and dissolves in hydrocarbon solvents suggests a more covalent interaction.

We are currently investigating the reaction chemistry of $Li[Be(t\text{-}C_4H_9)_3]$ and related organometallic compounds of beryllium and lithium and will report on these at a later date.

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Supplementary Material Available: Tables of crystallographic data, positional and thermal parameters, and bond distances and angles (8 pages); a listing of structure factor amplitudes (7 pages). Ordering information is given on any current masthead page.

Thermodynamic Control of Stereochemistry in Alkylation of Chiral Transition-Metal β-Oxoacyl Compounds: Enolization without Epimerization

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Summary: Deprotonation of $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(\mu\text{-}\eta^1,\eta^2\text{-}COCH_2CO)Re(CO)_4$ (**1**) in THF solution and addition of CH_3I lead to the C-alkylation product $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(\mu\text{-}\eta^1,\eta^2\text{-}COCH(CH_3)CO)Re(CO)_4$ (**2-D**), isolated as a single diastereomer (91% yield). In THF-d_8 solution, **2-D** exists in equilibrium with its enol form $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(\mu\text{-}\eta^1,\eta^2\text{-}COC(CH_3)C(OH))Re(CO)_4$ (**4**); K_{eq} = 0.10 at 23 °C. Deprotonation of **2-D** and alkylation with CD_3I generate $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(\mu\text{-}\eta^1,\eta^2\text{-}COC(CH_3)(CD_3)CO)Re(CO)_4$ (**5-D**-d_3), with >97% diastereoselectivity.

The alkylation chemistry of chiral β-oxoacyl transition metal complexes offers intriguing possibilities for asymmetric bond formation. This is particularly true in light of the role that the 1,3-dicarbonyl functionality plays in organic chemistry. However, application of metallaenolate methodology to the β-oxoacyl system raises interesting and important questions concerning enolization phenomena as well as alkylation regiochemistry.[1] Specifically, the formation of new chiral centers which bear an acidic hydrogen will be significant only if thermodynamic control of stereochemistry is operative or if the rate of enolization is sufficiently slow on the laboratory time scale.

We report herein that stereoselective methylation of the metallaenolate derived from chiral β-oxoacyl complex $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(\mu\text{-}\eta^1,\eta^2\text{-}COCH_2CO)Re(CO)_4$ (1) is indeed under thermodynamic control. In addition, the newly formed tertiary carbon center is readily deprotonated and C-alkylated a second time to generate a new quaternary carbon center with excellent diastereoselectivity.[2]

We recently reported the synthesis of the first stable β-oxoacyl complex $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(\mu\text{-}\eta^1,\eta^2\text{-}COCH_2CO)Re(CO)_4$ (1) from reaction of $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(COCH_2Li)$ and $Re(CO)_5(OSO_2CF_3)$.[3] Deprotonation of 1 (310 mg, 0.32 mmol, ~0.01 M) in THF with t-BuOK (0.44 mmol) and quenching the resultant enolate anion with excess CH_3I (~32 mmol) led to isolation of the C-alkylated product $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(\mu\text{-}\eta^1,\eta^2\text{-}COCH(CH_3)CO)Re(CO)_4$ (**2-D**) as a single diastereomer in 91% yield. In addition to **2-D**, the O-alkylation product $(\eta^5\text{-}C_5Me_2)Re(NO)(PPh_3)(\mu\text{-}\eta^1,\eta^2\text{-}COCHC(OCH_3))Re(CO)_4$ (**3**) is formed in ~4% yield as determined by ^1H NMR spectroscopy on the crude reaction mixture.[4] Complex **3** was synthesized in 18% isolated yield from similar reaction of the enolate from 1 with $(CH_3)_3O^+BF_4^-$.

We were unable to assign the stereochemistry for **2-D** on the basis of spectroscopic data; however, by single-

(1) (a) Constable, A. G.; Gladysz, J. A. *J. Organomet. Chem.* **1980**, *202*, C21. Kiel, W. A.; Lin, G.-Y.; Constable, A. G.; McCormick, F. B.; Strouse, C. E.; Eisenstein, O.; Gladysz, J. A. *J. Am. Chem. Soc.* **1982**, *104*, 4862. Kiel, W. A.; Lin, G.-Y.; Bodner, G. S.; Gladysz, J. A. *J. Am. Chem. Soc.* **1983**, *105*, 4958. Crocco, G. L.; Gladysz, J. A. *J. Am. Chem. Soc.* **1985**, *107*, 4103. O'Connor, E. J.; Kobayashi, M.; Floss, H. G.; Gladysz, J. A. *J. Am. Chem. Soc.* **1987**, *109*, 4837. Bodner, G. S.; Smith, D. E.; Hatton, W. G.; Heah, P. C.; Georgiou, S.; Rheingold, A. L.; Geib, S. J.; Hutchinson, J. P.; Gladysz, J. A. *J. Am. Chem. Soc.* **1987**, *109*, 7688. Buhro, W. E.; Zwick, B. D.; Georgiou, S.; Hutchinson, J. P.; Gladysz, J. A. *J. Am. Chem. Soc.* **1988**, *110*, 2427. (b) Liebeskind, L. S.; Welker, M. E. *Organometallics* **1983**, *2*, 194. Liebeskind, L. S.; Welker, M. E.; Goedken, V. *J. Am. Chem. Soc.* **1984**, *106*, 441. Liebeskind, L. S.; Fengl, R. W.; Welker, M. E. *Tetrahedron Lett.* **1985**, *26*, 3075. Liebeskind, L. S.; Welker, M. E.; Fengl, R. W. *J. Am. Chem. Soc.* **1986**, *108*, 6328. (c) Baird, G. J.; Davies, S. G. *J. Organomet. Chem.* **1983**, *248*, C1. Ambler, P. W.; Davies, S. G. *Tetrahedron Lett.* **1985**, *26*, 2129. Baird, G. J.; Davies, S. G.; Maberly, T. R. *Organometallics* **1984**, *3*, 1964. Ayscough, A. P.; Davies, S. G. *J. Chem. Soc., Chem. Commun.* **1986**, 1648. Davies, S. G.; Dordor-Hedgecock, I. M.; Warner, P. *J. Organomet. Chem.* **1985**, *285*, 213. Davies, S. G.; Dordor-Hedgecock, I. M.; Warner, P. *Tetrahedron Lett.* **1985**, *26*, 2125. Brown, S. L.; Davies, S. G.; Warner, P.; Jones, R. H.; Prout, K. *J. Chem. Soc., Chem. Commun.* **1985**, 1446. Davies, S. G.; Walker, J. C. *J. Chem. Soc., Chem. Commun.* **1986**, 609. Brown, S. L.; Davies, S. G.; Foster, D. F.; Seeman, J. I.; Warner, P. *Tetrahedron Lett.* **1986**, *27*, 623. Seeman, J. I.; Davies, S. G. *J. Am. Chem. Soc.* **1985**, *107*, 6522. Davies, S. G. *Pure Appl. Chem.* **1988**, *60*, 13 and references therein. (d) Theopold, K. H.; Becker, P. N.; Bergman, R. G. *J. Am. Chem. Soc.* **1982**, *104*, 5250. Doney, J. J.; Bergman, R. G.; Heathcock, C. H. *J. Am. Chem. Soc.* **1985**, *107*, 3724. Burkhardt, E. R.; Doney, J. J.; Stack, J. G.; Heathcock, C. H.; Bergman, R. B. *J. Mol. Catal.* **1987**, *41*, 41. Burkhardt, E. R.; Doney, J. J.; Slough, G. A.; Stack, J. M.; Heathcock, C. H.; Bergman, R. G. *Pure Appl. Chem.* **1988**, *60* and references therein. (e) Brown-Wensley, K. A.; Buchwald, S. L.; Canizzo, L.; Clawson, L.; Ho, S.; Meinhardt, D.; Stille, J. R.; Straus, D.; Grubbs, R. H. *Pure Appl. Chem.* **1983**, *55*, 1733. (f) Brinkmen, K.; Helquist, P. *Tetrahedron Lett.* **1985**, *26*, 2845. (g) Rusik, C. A.; Tonker, T. L.; Templeton, J. L. *J. Am. Chem. Soc.* **1986**, *108*, 1652.

(2) Mononuclear metallaenolates are generally not directly applicable to the synthesis of quaternary carbon centers due to the low acidity of disubstituted acyl complexes: Davies, S. G.; Walker, J. C. *J. Chem. Soc., Chem. Commun.* **1986**, 495.

(3) O'Connor, J. M.; Uhrhammer, R.; Rheingold, A. L. *Organometallics* **1987**, *6*, 1987.

(4) Characterization data for complexes **2-D**, **3**, **4**, **5**, **5-D**-d_3, **5-P**-d_3, and **6** is provided as supplementary material.

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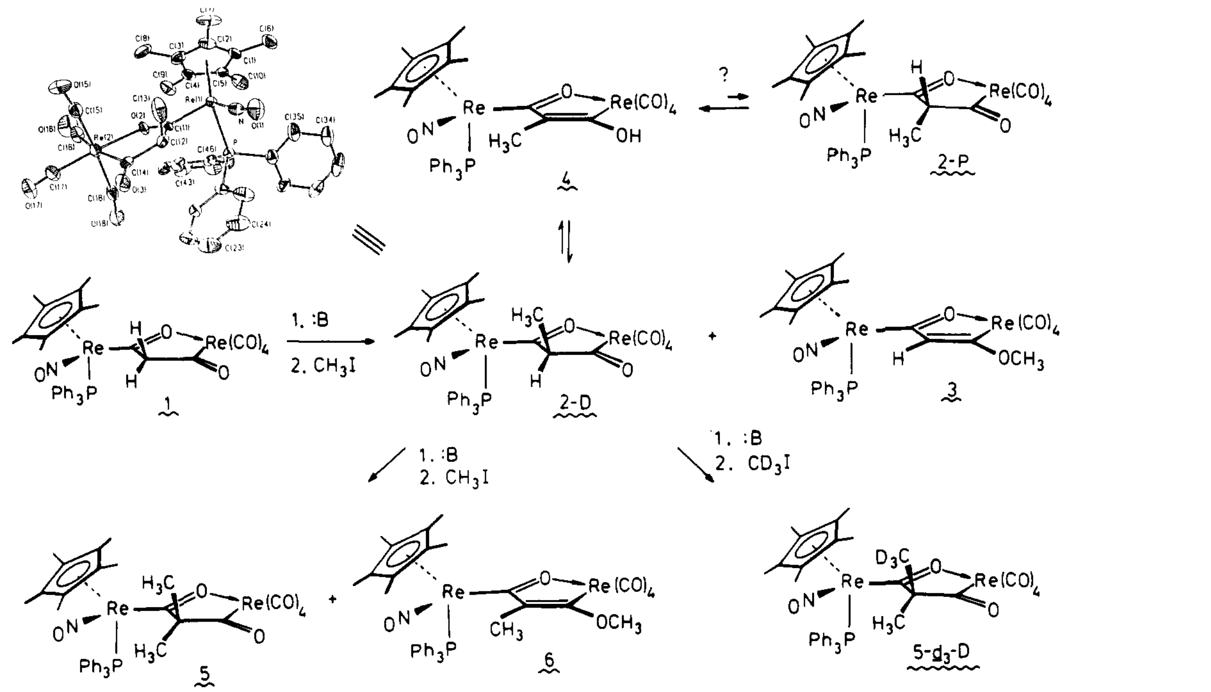
Scheme I



crystal X-ray analysis we have now established the stereochemistry for **2-D** as *SR,RS* (Scheme I).[5,6] The ON–Re–C_α–O torsion angle (θ) is 179°, which places the C(11)–O(2) oxygen anti to the NO ligand. The methyl substituent C(13) is distal to the bulky PPh_3 ligand, thereby occupying the less congested face of the nearly planar bridging oxoacyl ligand.

In mononuclear acetyl complexes (η^5-C_5H_5)M(L)(PPh_3)(COR) (M = Fe, L = CO; M = Re, L = NO), the observed alkylation stereoselectivity is consistent with approach of the electrophile from opposite the PPh_3 ligand.[1a–c] In contrast, alkylation of the pentamethylcyclopentadienyl derivative (η^5-C_5Me_5)Re(NO)(PPh_3)(COCHPhLi) is thought to give the isomer which would arise from approach of the electrophile from the same side as the PPh_3 ligand and opposite the C_5Me_5 ligand.[7] This raises the possibility that the kinetic alkylation product derived from (pentamethylcyclopentadienyl)rhenium acyl **1** is actually the *SS,RR* diastereomer **2-P** and that subsequent isomerization occurs to give the isolated *SR,RS* complex **2-D**.

In $CDCl_3$ solution there is no spectroscopic evidence for the enol form of **2-D**. However, in THF-d_8 new resonances are observed in the ^{1}H NMR spectrum at δ 8.92 (s, 1 H), 1.97 (s, 3 H), and 1.75 (s, 15 H), in addition to those resonances assigned to **2-D**.[4,8] We attribute these new resonances to the enol complex (η^5-C_5Me_5)Re(NO)(PPh_3)(μ-η^1,η^2-COC(CH_3)C(OH))Re$(CO)_4$ (**4**); K_{eq} = 0.10 at 23 °C.[4,8] The parent complex **1** was previously observed in equilibrium with its enol form in THF-d_8; K_{eq} = 0.66 at 23 °C.[9] The lower value of K_{eq} for methyl-substituted complex **2-D** may be due to an unfavorable steric interaction between the methyl group and the nitrosyl ligand when **4** occupies the conformation with θ = 180°. Parent complex **1** was observed to incorporate deuterium from added D_2O into the methylene hydrogen position distal to the PPh_3 ligand more rapidly ($t_{1/2}$ = 3 min) than deuterium incorporation into the proximal hydrogen site ($t_{1/2}$ = 2 h).[9] By comparison, in THF-d_8 solution containing D_2O (175 M excess), compound **2** undergoes exceedingly slow deuterium incorporation ($t_{1/2} \gg$ 2 weeks). Significantly, there is no evidence by ^{1}H NMR spectroscopy for formation of **2-P**-d_1. Addition of *t*-BuOK (8.4 × 10^{-3} M) to a THF-d_8/D_2O (3.8 M) solution of **2-D** (12 mg, 0.022 M) accelerates the rate of deuterium incorporation ($t_{1/2}$ = 4.5 h); again with no evidence for formation of **2-P**-d_1. When a THF-d_8 solution of the enolate anion derived from deprotonation of **2-D** is reprotonated with trifluoroacetic acid at −76 °C, initially only **4** is observed by ^{1}H NMR spectroscopy. Upon warming the NMR sample to 23 °C, slow formation of **2-D** is observed to the exclusion of **2-P**. Attempts to observe **2-P** by following the alkylation of **1** by low-temperature ^{1}H NMR spectroscopy resulted in observation of only **2-D** and **4**. The fact that this enolization process does not result in epimerization at carbon indicates that the observed diastereoselectivity is under thermodynamic control; i.e., keto–enol equilibration is established without loss of stereochemical integrity at carbon.

Introduction of a second methyl group at carbon proceeds with excellent regioselectivity. Thus, the methyl-

(5) Crystal data for **2-D**: triclinic, $P\bar{1}$; a = 10.295 (3) Å, b = 11.096 (3) Å, c = 17.367 (6) Å, α = 73.30 (2)°, β = 87.39 (2)°, γ = 70.62 (2)°; V = 1790.1 (9) Å; Z = 2; D(calcd) = 1.848 g cm^{-3}; μ(Mo Kα) = 72.33 cm^{-1}; T(max)/T(min) = 2.08; temp = 23 °C, Nicolet R3m/μ, Mo Kα. Of 6617 empirically absorption-corrected data (40 ≤ 2θ ≤ 50°), 6289 were independent (R_{int} = 2.58%), and 4468 were observed (5σ(F_o)). The structure was solved by heavy-atom methods. Refinement: all non-hydrogen atoms anisotropic, all hydrogen atoms idealized, phenyl rings constrained to rigid hexagons, $R(F)$ = 4.95% $R(wF)$ = 5.19%, GOF = 1.153, N_o/N_v = 11.5, Δ/σ(final) = 0.06 ($\Delta(\rho)$ = 2.3 e Å^{-3} (Re noise). SHELXTL software used for all computations (Nicolet XRD, Madison, WI).

(6) Absolute configurations are assigned according to the Baird/Sloan modification of the Cahn-Ingold-Prelog priority rules as employed by Gladysz: Stanely, K.; Baird, M. C. *J. Am. Chem. Soc.* **1975**, *97*, 6598. Sloan, T. E. *Top. Stereochem.* **1981**, *12*, 1. See ref 1a.

(7) On the basis of trends in NMR spectra the stereochemistry of the major alkylation product (η^5-C_5Me_5)Re(NO)(PPh_3)(COC(H)(Ph)(CH_3)) was *tentatively* assigned as *SS,RR*. Gladysz points out that this isomer would also result from electrophilic attack on the intermediate enolate from opposite the PPh_3 ligand on the *Z* isomer of the enolate: Heah, P. C.; Patton, A. T.; Gladysz, J. A. *J. Am. Chem. Soc.* **1986**, *108*, 1185.

(8) For **2-D**: ^{1}H NMR (THF-d_8) δ 1.01 (d, J = 7.9 Hz, 1 H), 1.80 (s, 15 H), 2.07 (q, J = 7.9 Hz, 3 H), 7.41 (m, br). For **3**: ^{1}H NMR (THF-d_8) δ 1.75 (s, 15 H), 1.97 (s, 3 H), 8.92 (s, 1 H), 7.41 (m, br).

(9) O'Connor, J. M.; Uhrhammer, R. *J. Am. Chem. Soc.* **1988**, *110*, 4448.

substituted acyl **2-D** is converted to the dimethyl complex **5** by deprotonation with *t*-BuOK followed by CH_3I addition (85% isolated yield).[4] In the ^1H NMR spectrum of **5** ($CDCl_3$) a singlet at δ 0.03 (3 H) is assigned to the methyl group proximal to the PPh_3 ligand and a singlet at δ 0.95 (3 H) is assigned to the methyl group distal to PPh_3.[10] Once again, only 4% of the O-alkylation product (η^5-C_5Me_5)Re(NO)(PPh_3)(μ-η^1,η^2-$\overline{COC(CH_3)C(OCH_3)}$)Re-$(CO)_4$ (**6**) is observed by ^1H NMR spectroscopy of the crude reaction mixture.

To probe the kinetic stereoselectivity of alkylation, the enolate of **2-D** was quenched with CD_3I to give **5-D**-d_3 as a >38:1 mixture of nonenolizable diastereomers favoring product with the CD_3 group distal to the PPh_3 ligand.[4] In a similar fashion methylation (CH_3I) of the enolate anion of **2**-d_3 leads to a >38:1 ratio of diastereomers favoring the product with the CD_3 group proximal to the PPh_3 ligand (**5-P**-d_3).[4] It is a reasonable assumption that product development control also dictates the alkylation stereochemistry in conversion of **1** to **2-D**.

The regio- and stereoselective alkylation chemistry described here indicates that chiral β-oxoacyl complexes undergo stereoselective formation of tertiary carbon centers and that stereochemical integrity at carbon is maintained even when keto-enol equilibration is established. In addition, β-oxoacyl complexes allow for direct stereoselective conversion of secondary carbon atoms to quaternary centers. We are currently exploring routes to analogous chelating and nonchelating β-oxoacyl complexes of the first-row metals.

Acknowledgment. Partial support from the Universitywide Energy Research Group at the University of California is gratefully acknowledged.

Supplementary Material Available: Listings of fractional coordinates, bond distances, bond angles, hydrogen atom coordinates, thermal parameters, and characterization of all new compounds (7 pages); a listing of structure factors (27 pages). Ordering information is given on any current masthead page.

(10) For **5**: ^1H NMR ($CDCl_3$) δ 0.03 (s, 3 H), 0.95 (s, 3 H), 1.68 (s, 15 H), 7.4 (s, br, 15 H). In NOE experiments, saturation of the C_5Me_5 resonance at δ 1.68 results in a 3.2% enhancement of the 0.95 singlet and a 1.4% increase in the intensity of the 0.03 singlet. Saturation of the PPh_3 resonance results in a 0.8% increase in the δ 0.95 resonance and a 3.5% enhancement of the δ 0.03 resonance. Saturation of the δ 0.03 resonance results in a 0.5% increase in the δ 1.68 resonance and a 2.4% increase in the δ 7.4 resonance. Saturation of the δ 0.95 resonance leads to a 2.8% increase in the δ 1.68 resonance and a δ 2.1% increase in the PPh_3 resonance at δ 7.4. While the observed intensity changes are not large, they do support the indicated assignments and are consistent with PPh_3 shielding of the methyl group proximal to the PPh_3 ligand.

Organoaluminum Chemistry of Bidentate Phosphine Ligands. Reaction of Diisobutylaluminum Hydride with Bis(diphenylthiophosphinoyl)methane: Synthesis and Molecular Structure of $[Al(C_4H_9)]_2[(C_6H_5)_2P(S)CP(C_6H_5)_2(S)_2][Al(C_4H_9)_2]_2$

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Summary: Reaction of bis(diphenylthiophosphinoyl)methane with diisobutylaluminum hydride affords the crystalline complex $[Al(C_4H_9)]_2[(C_6H_5)_2P(S)CP(C_6H_5)_2(S)_2][Al(C_4H_9)_2]_2$. The title compound, isolated from a condensation reaction involving the cleavage of Al–R, C–H, S–H, and Al–H bonds, crystallizes in the monoclinic space group $P2_1/n$ with unit cell parameters a = 19.703 (9) Å, b = 13.462 (8) Å, c = 19.924 (9) Å, β = 94.41 (4)°, V = 5269.47 Å^3, and D_{calcd} = 1.17 g cm^{-3} for Z = 4. Least-squares refinement based on 4120 observed reflections ($I > 3\sigma(I)$) converged at R = 0.0481 (R_w = 0.0544). The central core of the title compound contains an unusual S_2Al_4 fragment.

Although the organometallic chemistry of bis(diphenylphosphino)methane has been extensively investigated,[1-6] the corresponding organometallic chemistry of sulfur and oxygen derivatives of this ligand remains largely unexplored. To this end, we recently endeavored to investigate the organoaluminum chemistry of such bidentate phosphine ligands. Herein, we report the synthesis[7] and molecular structure of the novel organoaluminum main-group compound $[Al(C_4H_9)]_2[(C_6H_5)_2P(S)CP(C_6H_5)_2(S)_2][Al(C_4H_9)_2]_2$ isolated from reaction of bis(diphenylthiophosphinoyl)methane, $[(C_6H_5)_2P(S)CH_2P(S)(C_6H_5)_2]$, with diisobutylaluminum hydride. Particularly noteworthy is the fact that the central core of the compound contains an unusual S_2Al_4 fragment. The X-ray crystal structure of the title compound is shown in Figure 1.

X-ray intensity data were collected on a Nicolet R3m/V diffractometer using an $\omega/2\theta$ scan technique with Mo Kα radiation (λ = 0.71073 Å) at 21 °C. The title compound crystallizes in the monoclinic space group $P2_1/n$ with unit cell parameters a = 19.703 (9) Å, b = 13.462 (8) Å, c = 19.924 (9) Å, β = 94.41 (4)°, V = 5269 (5) Å^3, and D_{calcd} = 1.17 g cm^{-3} for Z = 4. The structure was solved by direct methods and refined, based on 4120 observed reflections ($I > 3\sigma(I)$), using SHELXTL.[8] Least-squares refinement converged at R = 0.0481 (R_w = 0.0544). Anisotropic thermal parameters were used for all non-hydrogen atoms. Hydrogen atoms were located by standard difference Fourier techniques. Phenyl hydrogen atoms were refined with isotropic temperature factors while the alkyl hydrogen atoms were constrained to idealized positions (d_{C-H} = 0.96 Å) with a refined isotropic group thermal parameter.

Previous studies in this laboratory concerned the organoaluminum chemistry of oxygen-,[9-11] sulfur-,[12-14] and

(1) Van Order, N., Jr.; Geiger, W. E.; Bitterwolf, T. E.; Rheingold, A. L. *J. Am. Chem. Soc.* **1987**, *109*, 5680.
(2) Albers, M. O.; Liles, D. C.; Robinson, D. J.; Singleton, E. *Organometallics* **1987**, *6*, 2179.
(3) Delavaux, B.; Chaudret, B.; Dahan, F.; Pollblanc, R. *Organometallics* **1985**, *4*, 935.
(4) Chaudret, B.; Dahan, F.; Saba, S. *Organometallics* **1985**, *4*, 1490.
(5) Iggo, J. A.; Markham, D. P.; Shaw, B. L.; Thornton-Pett, M. *J. Chem. Soc., Chem. Commun.* **1985**, 432.
(6) Puddephatt, R. J. *Chem. Rev.* **1983**, *112*, 99.

(7) Inside the drybox a reaction vessel was charged with bis(diphenylthiophosphinoyl)methane (2.2 mmol) and toluene/heptane (5 mL:5 mL mixture). Diisobutylaluminum hydride (Aldrich Chemical Co.) (5.6 mmol) was slowly added via syringe. Reaction was immediate and exothermic. The sealed reaction vessel (under argon) was removed from the drybox and heated (160 °C) in an oil bath for 24 h. The reaction vessel was vented periodically to release H_2, H_2S, and isobutane, which was eliminated during the course of the reaction. Removal of solvent and subsequent cooling to −5 °C resulted in X-ray quality crystals (35% yield based on bis(diphenylthiophosphinoyl)methane): mp 123 °C; ^1H NMR (IBM, 200 MHz, $CDCl_3$) δ 0.730–1.00 (m, 54 H, Al(C_4H_9)), 7.14–7.40 (m, 20 H, P(C_6H_5)$_2$); $^{31}P\{^1H\}$ NMR ($CDCl_3$, H_3PO_4 external reference) δ −8.66 (d, P:), 46.0 (d, P(S)), $^2J_{(P(S))-(P)}$ = 54.8 Hz.

(8) Sheldrick, G. M. SHELXTL, Crystallographic Computing System, Revision 5.1; Nicolet Instruments, Division: Madison, WI, 1986.
(9) Robinson, G. H.; Bott, S. G.; Elgamal, H.; Hunter, W. E.; Atwood, J. L. *J. Inclusion Phenom.* **1985**, *3*, 65.
(10) Atwood, J. L.; Elgamal, H.; Robinson, G. H.; Bott, S. G.; Weeks, J. L.; Hunter, W. E. *J. Inclusion Phenom.* **1984**, *2*, 367.

