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 $(\eta^5\text{-}C_5Me_5)Re(NO)(PPh_3)(\mu\text{-}\eta^1,\eta^2\text{-}C\overline{OC(CH_3)C(OCH_3))}Re(CO)_4$ (**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.

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[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

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[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.

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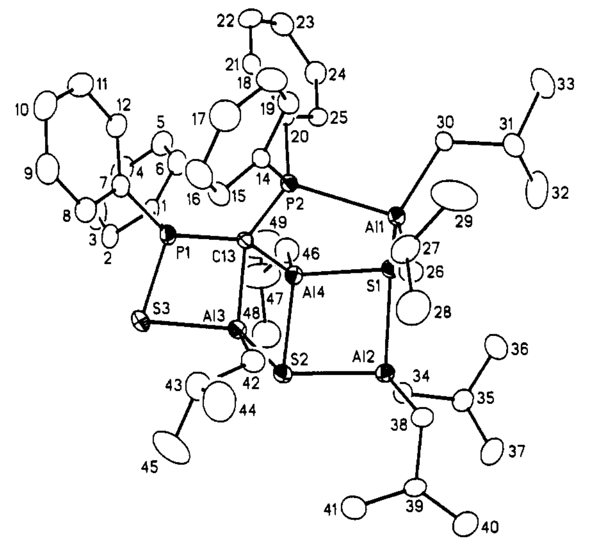

Figure 1. A view of the $[Al(C_4H_9)]_2[(C_6H_5)_2P(S)CP(C_6H_5)_2(S)_2][Al(C_4H_9)_2]_2$ molecule showing the atom labeling scheme. Thermal ellipsoids show 20% probability levels; hydrogen atoms have been omitted. Selected bond distances (Å) and angles (deg): Al(1)–P(2), 2.458 (3); Al(1)–S(1), 2.291 (3); Al(2)–S(1), 2.350 (3); Al(2)–S(2), 2.342 (3); Al(4)–S(1), 2.288 (3); Al(4)–S(2), 2.297 (3); Al(3)–S(2), 2.254 (3); Al(3)–C(13), 2.059 (5); Al(4)–C(13), 2.007 (6); Al(2)–S(1)–Al(4), 83.7 (1); Al(2)–S(2)–Al(4), 83.7 (1); S(1)–Al(1)–P(2), 94.0 (1); P(2)–C(13)–Al(4), 108.7 (3); P(1)–C(13)–P(2), 124.9 (3); P(2)–C(13)–Al(3), 123.3 (3); P(1)–C(13)–Al(3), 92.4 (2); P(1)–S(3)–Al(3), 79.6 (1).

nitrogen-based multidentate ligands.[15-21] These studies have revealed a particularly rich and varied chemistry. An examination of the corresponding chemistry of multidentate phosphine ligands is a logical extension of this work. Thus, we recently initiated a probe into the organoaluminum chemistry of bidentate phosphine ligands. This report represents the initial examination of the organoaluminum chemistry of the sulfur-based bidentate phosphine ligand bis(diphenylthiophosphinoyl)methane.

Several points are worthy of note regarding structure and bonding in this unusual compound. As can be seen from the X-ray crystal structure (Figure 1), the title compound contains four organoaluminum moieties: two isobutylaluminum fragments and two diisobutylaluminum fragments. Particularly significant is the fact that the central methylene carbon, C(13), of the bidentate phosphine ligand, initially bonded to two hydrogen atoms, is now bonded to two aluminum atoms (in addition to the two phosphorus atoms) and thus is the central atom of an Al_2P_2 distorted tetrahedron. The compound contains three sulfur atoms. Interestingly, only one sulfur atom, S(3), remains bonded to a phosphorus atom (P(1)–S(3) = 2.014 (3) Å). Each of the two remaining sulfur atoms, S(1) and S(2), are found to interact exclusively with aluminum atoms resulting in a S_2Al_4 fragment. Moreover, the Al–S bonds of this fragment are quite short having a mean Al–S distance of 2.286 (3) Å. The combination of the S_2Al_4 fragment and the bidentate phosphine ligand results in three adjoining four-membered rings in addition to one five-membered ring.

The title compound results from a unique condensation reaction involving the cleavage of Al–H, Al–R, S–H, and C–H bonds. It is reasonable to consider the reduction of one P=S bond of the ligand by diisobutylaluminum hydride, thus providing H_2S, as an initial step in the process (eq 1). Subsequent participation of the products of eq

$$[(C_6H_5)_2P(S)CH_2P(S)(C_6H_5)_2] \xrightarrow{(i\text{-}C_4H_9)_2AlH} [(C_6H_5)_2P(S)CH_2P(C_6H_5)_2] + H_2S \quad (1)$$

1 in a high-temperature (160 °C) condensation reaction with diisobutylaluminum hydride results in the title compound accompanied by H_2 and isobutane elimination (from the cleavage of Al–H and S–H bonds and Al–R and C–H bonds, respectively (eq 2).

$$[(C_6H_5)_2P(S)CH_2P(C_6H_5)_2] + 2H_2S + 4(i\text{-}C_4H_9)_2AlH \rightarrow [Al(C_4H_9)]_2[(C_6H_5)_2P(S)CP(C_6H_5)_2(S)_2][Al(C_4H_9)_2]_2 + 4H_2 + 2i\text{-Bu-H} \quad (2)$$

The initial report from this laboratory regarding the organoaluminum chemistry of bidentate phosphine ligands concerned the $[Al(CH_3)_3]_2[(C_6H_5)_2PCH_2P(C_6H_5)_2]$[22] complex isolated from reaction of trimethylaluminum with bis(diphenylphosphino)methane. The most notable comparison between the title compound and this complex concerns the central methylene hydrogen atoms of the bidentate phosphine ligand. The methylene hydrogen atoms of bis(diphenylphosphino)methane proved to be unreactive with respect to trimethylaluminum as only the 2:1 (AlR_3:ligand) adduct was isolated. This is in stark contrast with the title compound where both C–H bonds of the central methylene carbon atom were cleaved, along with Al–R bonds, resulting in a P_2CAl_2 fragment. The cleavage of the C–H bonds in bis(diphenylthiophosphinoyl)methane can thus be attributed to an increase in acidity of the methylene hydrogen atoms. The steric bulk of the isobutyl groups on the aluminum atoms notwithstanding, the Al(1)–P(2) bond distance of 2.458(3) Å in the title compound is shorter than the corresponding distances observed in $[Al(CH_3)_3]_2[(C_6H_5)_2PCH_2P(C_6H_5)_2]$ (2.585 (2) and 2.521 (2) Å).

The organoaluminum chemistry of the oxygen analogue of bis(diphenylthiophosphinoyl)methane has been previously examined. Reaction of bis(diphenylphosphinoyl)methane with trimethylaluminum resulted in $[Al(CH_3)][(C_6H_5)_2P(O)CP(O)(C_6H_5)_2]_2[Al(CH_3)_2]_2$.[23] This compound was noteworthy in that the central methylene hydrogen atoms of two bidentate phosphine ligand proved reactive toward $Al(CH_3)_3$ resulting in an $Al(CH_3)$ bridge between the two ligands in addition to two dimethylaluminum fragments.

While the transition-metal chemistry of oxygen and sulfur derivatives of bis(diphenylphosphino)methane has been examined,[24-26] the literature reveals a paucity of

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studies concerning the main-group organometallic chemistry of these ligands. The interaction of aluminum species with multidentate ligands containing phosphorus and sulfur represents an important extension of organoaluminum chemistry. While organoaluminum compounds containing Al–S bonds, characterized by X-ray diffraction techniques, are relatively rare, the title compound is unique, inter alia, in that it contains an S_2Al_4 moiety.

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Supplementary Material Available: Tables of crystal data, bond distances and angles, final fractional coordinates, thermal parameters, and a summary of data collection and refinement (14 pages); a listing of observed and calculated structure factors (25 pages). Ordering information is given on any current masthead page.

Electrochemical Reduction of $Fe(CO)_5$ Revisited

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Summary: The electrochemical reduction of $Fe(CO)_5$ in THF has been studied by cyclic voltammetry with microelectrodes. The electron stoichiometry of the reduction varies with potential scan rates because the initial two-electron reduction leading to $Fe(CO)_4^{2-}$ is followed rapidly by the reaction of the latter with $Fe(CO)_5$ to yield $Fe_2(CO)_8^{2-}$, the major product of electrolysis. An analysis of the cyclic voltammograms yields the rate of this reaction (6×10^6 M^{-1} s^{-1}). The anodic waves following reduction of $Fe(CO)_5$ have been assigned to specific electrode processes by comparison with the cyclic voltammograms of several iron carbonylate anions.

The electrochemical reduction of $Fe(CO)_5$ has attracted considerable attention.[1-6] There is general agreement that this reduction is chemically irreversible and that it is followed by fast chemical reactions. Several anodic peaks for the oxidation of reaction products have been observed after reduction in cyclic voltammetry which have not been positively assigned to specific electrode processes. In aprotic media, the electrolytic process was shown to involve an overall *one-electron* consumption and the major product was the $Fe_2(CO)_8^{2-}$ carbonylate anion.[3,6] It has been implicitly assumed that this product arises from the dimerization of the transient 17-electron $Fe(CO)_4^{\bullet-}$ radical anion intermediates formed by CO loss from the initial 19-electron $Fe(CO)_5^{\bullet-}$ radical anion. During its fleeting life, the latter behaves as an organic acyl radical[7] which can be intercepted by adding $(n\text{-Bu})_3SnH$ from which it abstracts a hydrogen atom affording the formyl anion $(CO)_4FeCOH^-$.[8] An apparently conflicting result was obtained when the cyclic voltammetry was performed in acetone at a Hg drop electrode[4] or in the presence of protic impurities:[6] in these cases the reduction was shown to be an overall *two-electron* process.[9] Furthermore, in synthetic work using Na naphthalenide as a one-electron carrier, it was found that the reduction of $Fe(CO)_5$ using 2 equiv of this reagent afforded Collman's reagent $Fe(CO)_4^{2-}$ whereas 1 equiv yielded $Fe_2(CO)_8^{2-}$.[10] The discrepancies concerning the observed electron stoichiometries could be related to the fate of the initially formed radical intermediates. Since we have recently studied by ESR the transient $Fe(CO)_4^{\bullet-}$ radical anion and we did not find it to be exceedingly short-lived under the photochemical conditions used for its generation (photolysis of $Fe_2(CO)_8^{2-}$ in THF),[11] we decided to reexamine the cyclic voltammetry of $Fe(CO)_5$ at faster potential scan rates than have been used so far in hopes of observing it directly by electrochemical techniques. We also felt that a complete understanding of the chemistry following reduction demands the positive identification of the associated anodic peaks. For this purpose we examined by cyclic voltammetry several iron carbonylate anions which could be involved in this chemistry (Table I). In this communication we report that the electron stoichiometry of the electrochemical reduction of $Fe(CO)_5$ in THF is dependent on the potential scan rates and that the $Fe(CO)_4^{\bullet-}$ radical anion, contrary to accepted views, is not a significant intermediate in this reduction at conventional scan rates.

Table I. Standard Potentials of Iron Carbonylate Anions[a]

electrochemical couple	$E°$ (V)
$Fe(CO)_4^{2-}/Fe(CO)_4^{\bullet-}$	-1.95[b]
$Fe_2(CO)_8^{2-}/Fe_2(CO)_8^{\bullet-}$	-1.68
$Fe_2(CO)_8^{\bullet-}/Fe_2(CO)_8$	-0.53[c]
$Fe_3(CO)_{11}^{2-}/Fe_3(CO)_{11}^{\bullet-}$	-1.55[d]
$Fe_4(CO)_{13}^{2-}/Fe_4(CO)_{13}^{\bullet-}$	-0.96[d]

[a] In THF with 0.3 M $(n\text{-Bu})_4N^+BF_4^-$ (except $Fe(CO)_4^{2-}/Fe(CO)_4^{\bullet-}$, cf. footnote *b*). $E°$ are given vs $Ag/AgBF_4$, 0.02 M in THF. In this medium and with this reference electrode, $E°$ for ferrocene/ferrocenium(1+) is -0.226 V. [b] In THF/HMPA (95:5 v/v) without supporting electrolyte and with Au electrode of 0.1-mm diameter. [c] Irreversible one-electron oxidation. Peak potential value at 5 V s^{-1}. [d] Additional oxidation waves are observed at more positive potentials.

Two cyclic voltammograms for the reduction of $Fe(CO)_5$ in THF are shown in parts A and B of Figure 1. The

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