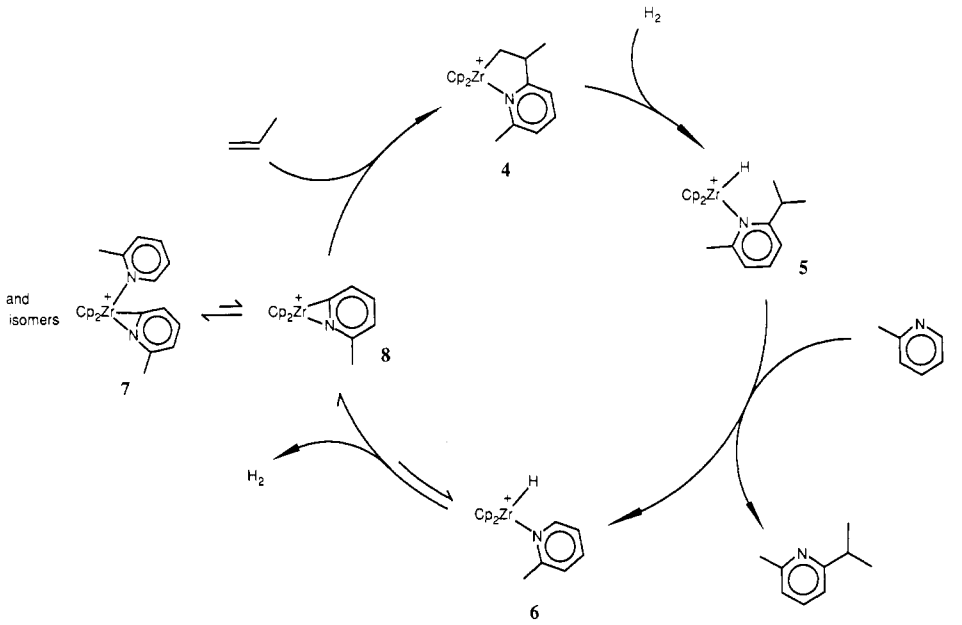
Scheme I



The results of stoichiometric model reactions and NMR monitoring experiments support the essential features of the proposed mechanism in Scheme I. Key observations are as follows. (1) Complex **4** is stable in the presence of excess picoline (i.e., no 6-Me, 2-^{i}Pr-pyridine is evolved), and H_2 is required for catalysis. These results imply that Zr–C bond cleavage in **4** occurs by hydrogenolysis and not by a C–H abstraction reaction of a ring-opened $Cp_2Zr\{CH_2CH(Me)(6\text{-Me-pyrid-2-yl})\}(picoline)^+$ species. (2) Reaction of **4** with H_2 in CH_2Cl_2 (<30 min, 20 °C, 1 atm) produces 6-Me,2-^{i}Pr-pyridine and Cp_2ZrCl_2. Hydrogenolysis of **4** in the presence of ethylene results in rapid formation of polyethylene. These results are consistent with the formation of **5** which contains a highly labile disubstituted pyridine ligand and which thus undergoes rapid reaction with solvent or ethylene.[14] (3) Reaction of **4** with H_2 in the presence of 3 equiv of picoline yields 6-Me,2-^{i}Pr-pyridine and **7** (two isomers, 3/1, 80% NMR). No intermediates are observed when this reaction is monitored by ^{1}H NMR. This is consistent with generation of **5** followed by rapid ligand substitution and H_2 elimination/C–H abstraction. The analogous reaction with D_2 produces 6-Me,2-^{i}Pr-pyridine labeled in the isopropyl methyl position. Catalytic H/D exchange of the ortho and methyl hydrogens of the excess picoline (ca. 5 and 1 t.o./h, respectively at 23 °C) is also observed, indicating that the conversion of **6** to **7** is reversible and that activation of methyl C–H bonds also occurs. (4) Complex **7**, like **2**, inserts propene to yield **4** (100% NMR, <10 min, 23 °C, 1 atm) and 1 equiv of α-picoline. By analogy to eq 2, picoline dissociation to yield **8** likely precedes insertion.[15] (5) Both **4** and **7** are effective catalysts. (6) Minor amounts of propane (ca. 10 mol % vs 6-Me,2-^{i}Pr-pyridine) are formed in the catalytic reactions, consistent with the intermediacy of Zr–H species. (7) ^{1}H NMR monitoring of catalytic reactions reveals that the only significant Zr species present are **4** and/or **7**.[15] This is consistent with the relative rates of the hydrogenolysis (**4** to **5**) and propene insertion (**7** to **4**) reactions (slow) and ligand exchange (**5** to **6**) and H_2 elimination (**6** to **7**) reactions (fast) established above.

(14) (a) In CH_2Cl_2 solution $Cp_2Zr(R)(THF)^+$ complexes and "naked" $Cp_2Zr(R)^+$ complexes decompose to yield $Cp_2Zr(R)Cl$ as initial products and are efficient ethylene polymerization catalysts.[8b] (b) $Cp_2Zr(H)Cl$ undergoes Cl/H exchange with CH_2Cl_2 to yield Cp_2ZrCl_2. Buchwald, S. L.; LaMaire, S. J.; Nielson, R. B.; Watson, B. T.; King, S. M. *Tetrahedron Lett.* **1987**, 3895.

(15) Reaction of **2** with picoline also yields **7**. In this case significant amounts of two additional isomers or oligomers of **7** are also formed. These species are the sole products when **2** is reacted with neat picoline, are minor products in the reaction of **4** with H_2 in the presence of a large excess of picoline, and are observed as minor species in catalytic runs containing high concentrations of α-picoline. These additional isomers/oligomers do not react rapidly with propene.

Further mechanistic studies of the current system and extensions to other substrates are in progress.[16]

Acknowledgment. This work was supported by the National Science Foundation (Grant CHE-8706294) and the donors of the Petroleum Research Fund, administered by the American Chemical Society (Grant 19467-AC3). We thank Professor Rob Ronald for valuable discussions and suggestions and Sam Borkowsky and Don Appel for timely assistance with several experiments.

Supplementary Material Available: A listing of characterization data for **2**, **4**, **7**, and 6-Me,2-^{i}Pr-pyridine (4 pages). Ordering information is given on any current masthead page.

(16) Ethylene and 1-butene also are catalytically coupled with picoline. Pyridine is not a suitable substrate due to the formation of an unreactive nonlabile $Cp_2Zr(pyridyl)(pyridine)^+$ species analogous to **7**.

Ramberg–Bäcklund Syntheses and Chemodirected Annulations of Exocyclic Allylsilanes[1]

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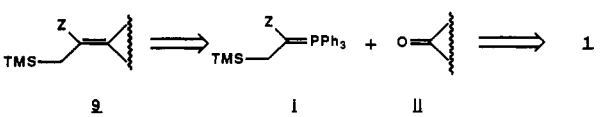
In connection with our synthetic program we required a means to convert an α-sulfonyl anion **1** to a series of vinyl-functionalized allylsilanes **9a–e**.[2] Guided by the observations of Henderickson[3a,b]

(1) Syntheses via Vinyl Sulfones. 36. For a review of this area, see: Fuchs, P. L.; Braish, T. F. *Chem. Rev.* **1986**, *86*, 903.

(2) Although oxidation of α-sulfonyl anion **1** (see: Baudin, J.-B.; Julia, M.; Rolando, C. *Tetrahedron Lett.* **1985**, *26*, 2333) to ketone *i* followed by Wittig reaction with substituted trimethylsilylethyl phosphorane reagents *ii* (the parent reagent Z=H is used in this manner, see: Fleming, I.; Marchi, D., Jr. *Synthesis* **1981**, 560) represented a formal solution for synthesis of **9**, we desired a more general method.



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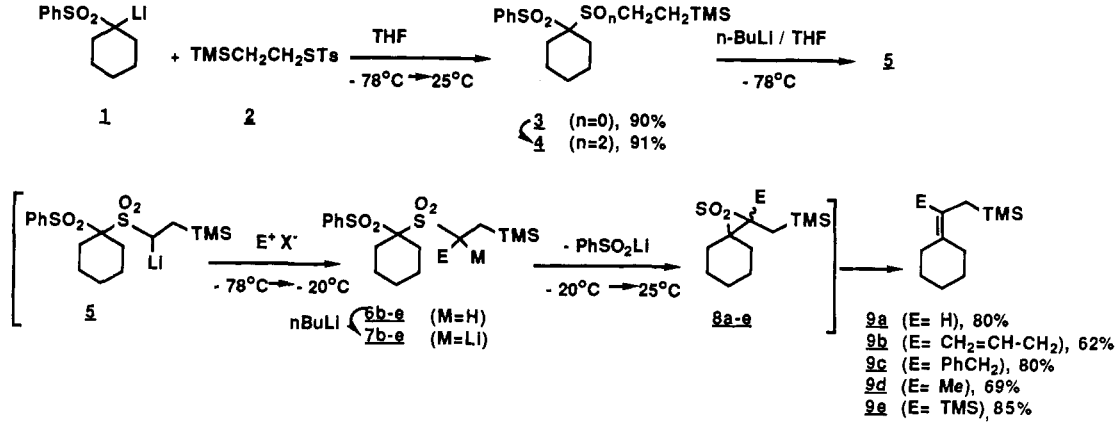
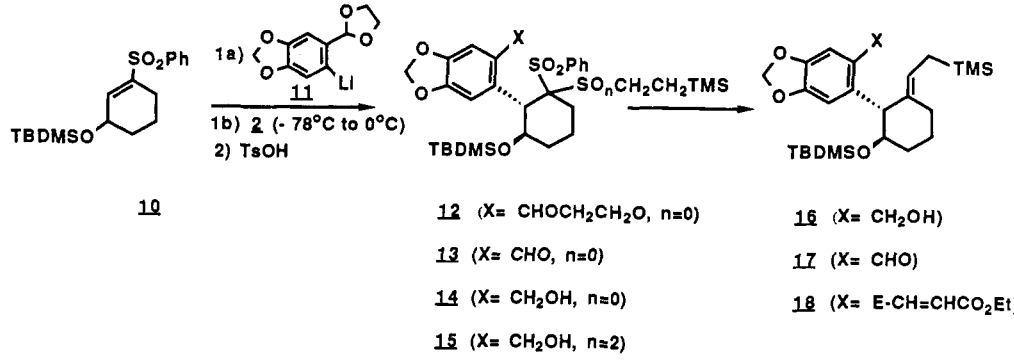
Scheme I



Scheme II



and Matsuyama[3c] that α-sulfonyl sulfones undergo the Ramberg–Bäcklund[3d,e] olefination reaction, we speculated that β-silylethyl bis-sulfone **4** should undergo metalation to provide α-sulfonyl anion **5** which could be converted to allylsilanes **9a–e**.

Treatment of cyclohexylphenylsulfone with *n*-butyllithium in THF at −78 °C generates **1** to which is added 1 equiv of thiosulfonate **2**,[4] and the solution is warmed to room temperature to afford a 90% yield of α-sulfenylated sulfone **3**. Transformation of **3** to bis-sulfone **4** is accomplished via MCPBA oxidation (91%).[5] Reaction of **4** with *n*-butyllithium in THF at −78 °C affords the yellow anion **5**, which fades upon warming to 0 °C accompanied by the formation of allylsilane **9a** (80%). Intermediate **5** can be intercepted by addition of an electrophile in the presence of 4% HMPA, followed by warming to −20 °C for 1 h. Alkylation of **5** with allyl bromide, benzyl bromide, methyl iodide, and chlorotrimethylsilane provides functionalized bis-sulfones **6b–e** in 70–87% yield. These intermediates need not be isolated; standard protocol simply involves following the functionalization step with a second equivalent of *n*-butyllithium to produce a new α-sulfonyl anion **7**, which decomposes upon warming to allylsilanes **9b–e**.[6,7]

Combination of the above strategy with the previously established conjugate-addition chemistry of vinyl sulfones[1] provides an opportune setting to expand the established annulation methodology[8] of the allylsilane moiety. Reaction of vinyl sulfone **10**[9] with aryllithium reagent **11**[10] at −78 °C in a 2:3 mixture of THF and ether followed by addition of thiosulfonate **2**,[3] warming to 0 °C, and aqueous workup provides a mixture of acetal **12** and aldehyde **13**. Completion of the hydrolysis of the sensitive acetal by stirring this mixture in acetone with *p*-toluenesulfonic acid for 2 h at 25 °C provides aldehyde **13** in 81% yield as a single (unassigned) diastereomer at the α-sulfonyl center. Treatment of this aldehyde with sodium borohydride in 1:1 methanol/THF for 10 min at room temperature affords alcohol **14** in 93% yield. Oxidation of sulfide **14** to bis-sulfone **15** was smoothly accomplished by MCPBA in methylene chloride for 1 h at 0 °C (80%). Reaction of **15** with 2.5 equiv of *n*-BuLi in THF at −78 °C followed by warming to 0 °C provides a 91% yield of allylsilane **16**. This material is a single stereoisomer at the exocyclic olefin and is assigned the E-configuration based upon NOE measurements.[11] Reaction of **16** with MnO_2 (5 equiv) in hexane for 1 h at room temperature affords aldehyde **17** in 93% yield. Wittig olefination of **17** with carbethoxytriphenylphosphorane in toluene at reflux for 12 h gives *trans*-vinyl ester **18** (86%).

Treatment of allylsilane **17** with titanium tetrachloride in methylene chloride at −78 °C for 2 min affords a 4:1 mixture of cis fused alcohols **19ax-x** and **19ax-n** in 70% yield. Repetition

(3) (a) Hendrickson, J. B.; Palumbo, P. S. *J. Org. Chem.* **1985**, *50*, 2110. (b) Hendrickson, J. B.; Boudreaux, G. J.; Palumbo, P. S. *J. Am. Chem. Soc.* **1986**, *108*, 2358. (c) Matsuyama, H.; Miyazawa, Y.; Takei, Y.; Kobayashi, M. *J. Org. Chem.* **1987**, *52*, 1703. (d) Paquette, L. A. *Organic Reactions*, John Wiley: New York, 1977; Vol. 25, p 1. (e) Block, E.; Aslam, M.; Eswarakrishnan, V.; Gebreyes, K.; Hutchinson, J.; Iyer, R.; Laffitte, J.-A.; Wall, *J. Am. Chem. Soc.* **1986**, *108*, 4568.

(4) Ranasinghe, M. G.; Fuchs, P. L. *Synth. Commun.* **1988**, *18*, 227.

(5) Anderson, M. A.; Ranasinghe, M. G.; Palmer, J. T.; Fuchs, P. L. *J. Org. Chem.* **1988**, *53*, 3125.

(6) These products are usually accompanied by ca. 5–8% of **9a** which arises from incomplete functionalization of [**5**]. It is interesting to note that use of larger excesses of *n*-butyllithium during reaction with **4** effects partial metalation of the arylsulfonyl moiety, although this does not adversely affect the yield of **9** providing that a sufficient excess of functionalization reagent is employed to effect alkylation of the aryl group. In those instances where small amounts of **9a** are not acceptable, purification of **6** can be accomplished by silica chromatography.

(7) For alternative approaches to bis-silyl olefins related to **9e**, see: Smith, J. G.; Drozda, S. E.; Petraglia, S. P.; Quinn, N. R.; Rice, E. M.; Taylor, B. S.; Viswanathan, M. *J. Org. Chem.* **1984**, *49*, 4112.

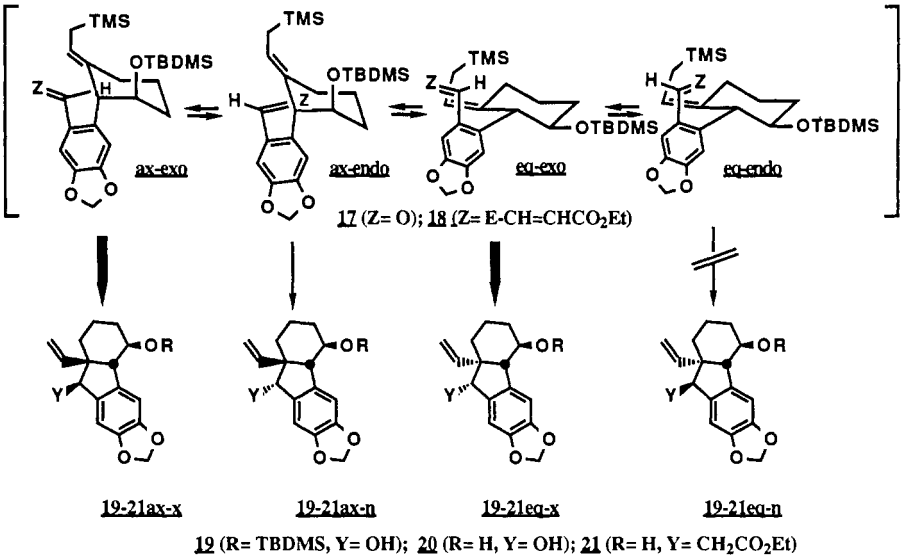
(8) (a) Schinzer, D. *Synthesis* **1988**, 263. (b) Sarkar, T. K.; Andersen, N. H. *Tetrahedron Lett.* **1978**, 3513. (c) Denmark, S. E.; Weber, E. J. *J. Am. Chem. Soc.* **1984**, *106*, 7970. (d) Majetich, G.; Desmond, R. W., Jr.; Soria, J. J. *J. Org. Chem.* **1986**, *51*, 1753. (e) Majetich, G.; Casares, A.; Chapman, D.; Behnke, M. *J. Org. Chem.* **1986**, *51*, 1745. (f) Majetich, G.; Defauw, J.; Ringold, C. *J. Org. Chem.* **1988**, *53*, 50 and references contained therein.

(9) Saddler, J. C.; Conrad, P. C.; Fuchs, P. L. *Tetrahedron Lett.* **1978**, 5079.

(10) Conrad, P. C.; Kwiatkowski, P. L.; Fuchs, P. L. *J. Org. Chem.* **1987**, *52*, 586.

(11) The assignment of stereochemistry of **16** and **19–21** is based upon extensive spectral and chemical correlation experiments; these are described in detail in the Supplementary Material.

Scheme III



Substrate	Reagent	cis-Fused	trans-Fused	C/T
17	$TiCl_4$	19ax-x (56%) + 19ax-n (14%)	--	70/0
17	BF_3-Et_2O	19ax-x (41%) + 19ax-n (4.5%)	--	45/0
17	n-Bu_4NF 25°C,18h	19ax-x (4%) + 20ax-x (7%) +	20eq-x (79%)	11/79
17	n-Bu_4NF 0°C,1h	19ax-x (13%) +	19eq-x (51%) + 20eq-x (15%)	13/65
18	n-Bu_4NF	--	21eq-x (65%)	0/65
18	$TiCl_4$ or BF_3-Et_2O... no cyclization products; only protodesilylation observed.			

of this experiment using boron trifluoride etherate (−78 °C, 4 h) as the Lewis acid provided the same two alcohols in a 9:1 ratio (45% yield). As expected, no trans-fused products (**19eq-x**/**19eq-n**) were formed. Attempts to cyclize vinyl ester **18** under comparable conditions simply afforded protodesilylation of the allylsilane without any annulation taking place. All these observations are well in accord with a wealth of precedent that had been previously established in related systems.[8]

Accordingly, we were exceedingly surprised to observe that reaction of **17** with excess tetrabutylammonium fluoride in THF at room temperature for 18 h provided a 10:1 mixture of diols **20eq-x** and **20ax-x** in 86% yield; in addition, 4% of **19ax-x**, the monosilyl ether of **20ax-x**, was isolated. Repeating this reaction for 1 h at 0 °C provided **19eq-x** (51%), **19ax-x** (13%) as well as diol **20eq-x** (15%). Thus, the selectivity for the fluoride-induced trans fusion process is between 5–7:1. Moreover, conducting this fluoride-promoted reaction for 3 h in THF at room temperature with vinyl ester **18** as substrate affords **21eq-x** as a single stereo- and regioisomer in 65% yield.[11]

A commonly held dictum in synthetic planning is that *all annulation reactions which generate small rings (≤7) are expected to form cis ring fusions*;[12] that is, bond formation will occur syn to the tethering arc. Allyl-, vinyl-, and alkylsilanes and -stannanes have served as terminators in cation–olefin cyclizations, affording trans-fused products,[13] but we feel that the fluoride-induced annulations described above are prototypical examples of a rare, but potentially predictable process.[14] Examination of the literature reveals some additional (although less stereospecific) examples of formation of trans fused 6/5 ring systems.[15] The common feature in all these cases appears to involve conformational bias toward maintaining the reacting arc in an *equatorial* orientation (cf. **17,18eq**). Those factors which are responsible for the reagent-based annulation specificity observed are yet to be determined. Experiments along these lines are underway.

Acknowledgment. We thank the National Institutes of Health (AI 13073) for their generous support of this work. We thank the Purdue University Biological Magnetic Resonance Laboratory (NIH RR01077) for access to the 470 MHz high field proton spectrometer and Mark Anderson and David Scarpetti for spectral assistance. Arlene Rothwell provided mass spectral data. We

(12) For leading referenes to kinetic annulation processes forming trans fused rings of ≥7, see: (a) Silverton, J. V.; Ziffer, M. *J. Org. Chem.* **1979**, *44*, 3959. (b) Berthiaume, G.; Lavallee, J.-F.; Deslongchamps, P. *Tetrahedron Lett.* **1986**, *27*, 5451.

(13) (a) Grieco, P. A.; Fobare, W. F. *Chem. Commun.* **1987**, 185. (b) Burke, S. D.; Saunders, J. O.; Oplinger, J. A.; Murtiashaw, C. W. *Tetrahedron Lett.* **1985**, *26*, 1131. (c) Macdonald, T. L.; Mahalingam, S. *Tetrahedron Lett.* **1981**, *22*, 2077. (d) Macdonald, T. L.; Mahalingam, S.; O'Dell, D. E. *J. Am. Chem. Soc.* **1981**, *103*, 6767.

(14) For an example of formation of a trans fused bicyclic ring system via "antitether" delivery of an acyl nitroso group to an exocyclic 1,3-diene, see: Burkholder, T. B.; Fuchs, P. L. *J. Am. Chem. Soc.* **1988**, *110*, 2341.

(15) (a) Beal, R. B.; Dombroski, M. A.; Snider, B. B. *J. Org. Chem.* **1986**, *51*, 4391. (b) Molander, G. A.; Etter, J. B. *J. Org. Chem.* **1986**, *51*, 1778. (c) Crandall, J. K.; Magaha, H. S.; Henderson, M. A.; Widener, R. K.; Tharp, G. A. *J. Org. Chem.* **1982**, *47*, 5372. (d) Garst, M. E.; McBride, B. J.; Johnson, A. T. *J. Org. Chem.* **1983**, *48*, 8.

are grateful for helpful discussions with Professors B. Snider (Brandeis), J. Crandall (Indiana), and S. Denmark (Illinois) on the subject of the fluoride-based cyclizations.

Supplementary Material Available: Listings of chemical and spectral data for compounds **17-21** (5 pages). Ordering information is given on any current masthead page.

Synthesis of $(C_5Me_5)Ta(S)_3^{2-}$ and the Structure of a Hexagonal-Prismatic $Ta_2Li_4S_6$ Core

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The attention that has been paid to synthesis and structures of sulfido- and polysulfido-metal complexes derives both from the well-established significance of such compounds in metalloenzymatic catalysis and also from the wealth of their own chemistry. Our attempt to prepare sulfido complexes of pentamethylcyclopentadienyltantalum, Cp*Ta, was prompted by the two motives. One is the beautiful chemistry developed recently from the closely related complexes $Cp'_2Mo_2S_4$[1] and $Cp'_2V_2S_4$ (Cp' = C_5H_4Me).[2] The other is the EI fragmentation pattern of $Cp^*Ta(SCH_2CH_2S)_2$ which shows sets of peaks associated with $Cp^*Ta(SCH_2CH_2S)(S_2)^+$ and $Cp^*Ta(S_2)_2^+$.[3]

Aiming to synthesize relevant disulfido(2-) complexes, we ran a reaction between Cp^*TaCl_4 and 2 equiv of Li_2S_2[4a] in THF, only to find that an uncharacterizable orange-colored gum was formed. However, when the amount of Li_2S_2 was increased by 2-2.5 times we were able to isolate the unexpected title complex **1** which carries three terminal sulfides at a Ta center. Thus, the reaction of Cp^*TaCl_4 with 4-5 equiv of Li_2S_2 in dry THF under Ar gave **1** as light yellow crystals (ca. 50% yield), after evaporation of the solvent, extraction of the residue with benzene, and recrystallization from THF/hexane.[5a] Formation of **1** was also noted when $Cp^*Ta(SCH_2CH_2S)_2$ was treated with 2 equiv of Li_2S_2 in THF, which is an intriguing reaction by itself. Upon introducing a THF solution of $Cp^*Ta(SCH_2CH_2S)_2$ to a THF suspension of Li_2S_2, the color of the solution turned gradually from red to yellow, and, after filtrating off the insoluble yellow residue, **1** (crystals, 42% yield) was readily isolated from the filtrate by concentrating it in vacuo to ca. one-seventh of its original volume.

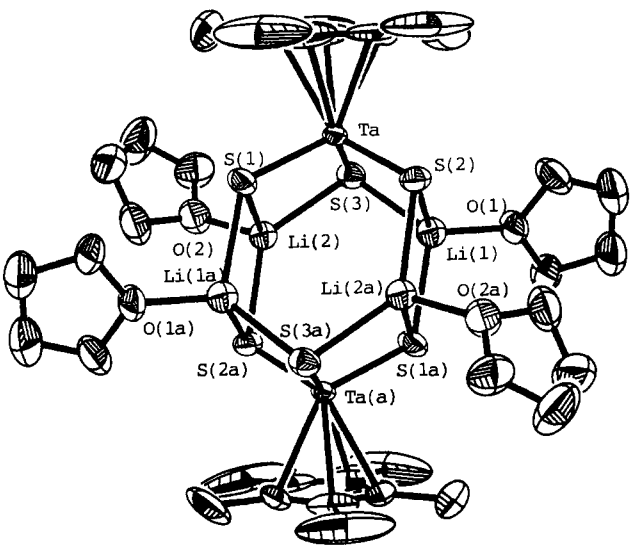
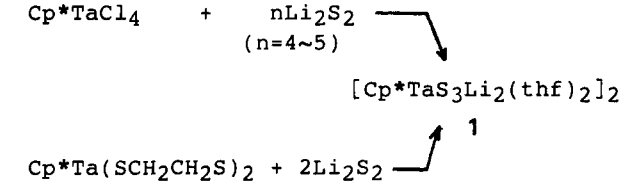


Figure 1. Molecular structure and atom-labeling scheme for $[Cp^*TaS_3Li_2(thf)_2]_2$ (**1**) drawn with 50% thermal ellipsoids. Selected bond distances (Å) and angles (deg) are as follows: Ta-S(1), 2.270 (2); Ta-S (2), 2.268 (3); Ta-S(3), 2.300 (3); Ta-C(1), 2.48 (1); Ta-C(2), 2.47 (1), Ta-C(3), 2.45 (1); Ta-C(4), 2.46 (1); Ta-C(5), 2.45 (2); Ta-Ta(a), 5.490 (1); Li(1)-S(1a), 2.45 (2); Li(1)-S(2), 2.54 (2); Li(1)-S(3), 2.44 (2); Li(2)-S(1), 2.50 (2); Li(2)-S(2a), 2.49 (2); Li(2)-S(3), 2.44 (2); Li(1)-O(1), 1.96 (2); Li(2)-O(2), 1.90 (2); S(1)-Ta-S-(2), 107.1 (1); S(1)-Ta-S(3), 103.4 (1); S(2)-Ta-S(3), 104.1 (1); S-(1a)-Li(1)-S(2), 104.7 (6); S(1a)-Li(1)-S(3), 133.1 (7); S(2)-Li(1)-S(3), 92.7 (6); S(1)-Li(2)-S(2a), 104.9 (7); S(1)-Li(2)-S(3), 93.1 (5); S(2a)-Li(2)-S(3), 118.4 (7).



With the stoichiometry of **1** being established, we attempted the reaction between Cp^*TaCl_4 and 3 equiv of Li_2S.[4b] However, isolation of **1** from the resultant red powder[6] has not been successful. Instead, we noticed formation of **1** (ca. 50% yield) when the amount of Li_2S was increased to 5 equiv as in the case of the reaction with Li_2S_2. On the other hand, addition of tetramethylethylenediamine (tmeda) (excess) into a THF solution of **1** afforded a stoichiometric amount of yellow crystalline powder formulated as $Cp^*TaS_3Li_2(tmeda)_2$ **2**.[5b] The sulfide complexes **1** and **2** are moderately air-sensitive and hygroscopic.

Figure 1 shows an ORTEP view of the structure determined for **1** by X-ray crystallography,[7] along with the salient intramolecular

† Present address: Department of Chemistry, Calvin College, Grand Rapids, MI 49506.

(1) (a) Rakowski DuBois, M.; DuBois, D. L.; VanDerveer, M. C.; Haltiwanger, R. C. *Inorg. Chem.* **1981**, *20*, 3064-3071. (b) Casewit, C. J.; Rakowski DuBois, M. *J. Am. Chem. Soc.* **1986**, *108*, 5482-5489, and references therein. (c) Wright, L. L.; Haltiwanger, R. C.; Noordik, J.; Rakowski DuBois, M. *Ibid.* **1987**, *109*, 282-283. (d) Brunner, H.; Meier, W.; Wachter, J.; Guggolz, E.; Zahn, T.; Ziegler, M. L. *Organometallics* **1982**, *1*, 1107-1113. (e) Beck, W.; Danzer, W.; Thiel, G. *Angew. Chem., Int. Ed. Engl.* **1973**, *12*, 582-583.

(2) (a) Bolinger, C. M.; Rauchfuss, T. B.; Wilson, S. R. *J. Am. Chem. Soc.* **1982**, *104*, 7313-7314. (b) Rauchfuss, T. B.; Weatherill, T. D.; Wilson, S. R.; Zebrowski, J. P. *Ibid.* **1983**, *105*, 6508-6509. (c) For $Cp^*_2Cr_2S_5$, see: Brunner, H.; Wachter, J.; Guggolz, E.; Ziegler, M. L. *Ibid.* **1982**, *104*, 1765-1766. (d) For $Cp'_4Ti_4S_8O_x$, see: Zank, G. A.; Jones, C. A.; Rauchfuss, T. B.; Rheingold, A. L. *Inorg. Chem.* **1986**, *25*, 1886-1891.

(3) Tatsumi, K.; Takeda, J.; Sekiguchi, Y.; Kohsaka, M.; Nakamura, A. *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 332-333.

(4) (a) "Li_2S_2" (light yellow powder) was prepared by reaction of lithium metal with elemental sulfur (1:1) in liquid ammonia. This "Li_2S_2" was nearly identical with that prepared from the reaction between $LiEt_3BH$ (Super Hydride) and $^1/_8S_8$ according to their Raman spectra. Further characterization of the lithium sulfide(s) is underway. Before use, the resulting yellow powder was washed by THF under Ar until the filtrate became nearly colorless. See: Letoffe, J. M.; Thourey, J.; Perachon, G.; Bousquet, J. *Bull. Soc. Chim. Fr.* **1976**, 424-426. Dubois, P.; Lelieur, J. P.; Lepoutre, G. *Inorg. Chem.* **1988**, *27*, 73-80; Gladysz, J. A.; Wong, V. K.; Jick, B. S. *Tetrahedron* **1978**, *35*, 2329-2335. (b) "Li_2S" (yellowish white powder) was prepared similarly from the $Li/^1/_{16}S_8$ reaction system.

(5) (a) $[Cp^*TaS_3Li_2(thf)_2]_2$ **1**: ^1H NMR (100 MHz, THF-d_8) δ 2.10 (s, Cp*); UV (λ_{max} (10^{-3} ϵ_{max}, M^{-1} cm^{-1}), THF) 302 (16.6) nm; Raman (crystals) 619 w, 597 m, 552 w, 434 s (Ta=S), 418 sh, 368 w, 354 w. (b) $Cp^*TaS_3Li_2(tmeda)_2$ **2**: ^1H NMR (100 MHz, C_6D_6) δ 2.49 (s, Cp*), 2.44 (s, $(CH_3)_2NCH_2CH_2N(CH_3)_2$), 2.18 (s, $(CH_3)_2NCH_2CH_2N(CH_3)_2$); UV ($\lambda_{max}$ (10^{-3} ϵ_{max}, M^{-1} cm^{-1}), C_6H_6) 305 (7.2) nm; Raman (crystals) 618 w, 595 w, 552 w, 432 s (Ta=S), 423 sh, 401 m, 350 w. The molecular weight estimated by cryoscopy in benzene is not accurate because of relatively low solubility of **2**, ranging from 500-700 which however might indicate the monomeric nature of **2**.

(6) Characterization of the product(s) is in progress.

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