s-Bu_2Zn are stirred with solid MeOK, solutions are produced within a few minutes that have a MeOK to R_2Zn ratio of 0.5, which does not change on longer stirring, with sonication, or with heating.

(3) In some systems, liquids or solids with R′OK to R_2Zn ratios of 0.5 or 1.0 remain from removing R_2Zn at reduced pressure from preparations made from R′OK and R_2Zn. For example, subjecting a benzene solution containing *t*-BuOK and excess Bu_2Zn to 0.5 Torr at 30–40 °C for 12 h left a viscous liquid (solidified at 0 °C) that had a *t*-BuOK to Bu_2Zn ratio of 0.5. Subjecting that oil to 0.01 Torr and 75 °C for 12 h left a solid that had a ratio of 1.0.

(4) More ^1H and ^{13}C NMR absorptions for R (Bu and Me_3SiCH_2) and R′ (*t*-Bu) are seen at R to R′OK ratios in the 0,5–1.0 range when 18-crown-6 is added.[23] A possible explanation of these observations is that coordination of the crown ether with K^+ slows exchange between the 1:1 species and other species in solution.

We conclude that solutions prepared from R_2Zn and R′OK form zincate species, probably similar to the magnesiate species formed from R_2Mg and R′OK. Equilibration of R and OR′ groups between all species in the solutions is more rapid for Zn than for Mg, however, most likely reflecting a lesser tendency of R_2Zn than of R_2Mg to form bonds to oxygens.

Acknowledgment. We are indebted to the National Science Foundation for support of this work and for aiding in the purchase of the NMR spectrometers. We thank referees for particularly helpful comments.

Registry No. $[Et_2Zn(t\text{-}BuO)_2ZnEt_2]^{2-}2K^+$, 131130-36-4; *t*-BuOK, 865-47-4; Et_2Zn, 557-20-0.

Supplementary Material Available: Tables of atomic coordinates, bond lengths and angles, and anisotropic temperature parameters for $[Et_2Zn(t\text{-}BuO)_2ZnEt_2]^{2-}2K^+$ (7 pages); table of observed and calculated structure factor amplitudes for $[Et_2Zn(t\text{-}BuO)_2ZnEt_2]^{2-}2K^+$ (7 pages). Ordering information is given on any current masthead page.

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[23] Bu_2Zn and 18-crown-6 in benzene do not form significant amounts of a "threaded" [Pajerski, A. D.; BergStresser, G. L.; Parvez, M.; Richey, H. G., Jr. *J. Am. Chem. Soc.* **1988**, *110*, 4844] Bu_2Zn(crown) species.

Lithium–Metalloid Exchange.[1] Dynamics and Equilibrium in the Li/I and Li/Te Exchange in Tetrahydrofuran: Iodine, Tellurium, and Mercury Ate Complexes

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The metal halogen exchange[2] and related transmetalations involving selenium,[1e,3] tellurium,[4] tin,[1d,5] and mercury[6] are powerful methods for the preparation of functionalized and unstable organolithium reagents. The mechanisms of these transformations have been variously described in terms of single electron transfer or four-center processes or ate complex intermediates.[1a,b,d,5,7] We have previously reported kinetic[1a] and spectroscopic[1b] studies which demonstrated that phenyllithium and iodobenzene combine to form the ate complex $Ph_2I^-Li^+$ at low temperatures in THF/HMPA. Described herein are results from spectroscopic studies which implicate ate complex intermediates in the Li/Hg, Li/Te, and Li/I exchanges in THF as well and extend and complement our earlier work. The exchange in pure THF is complicated by the interplay between the monomer–dimer equilibrium of phenyllithium (eq 1), the ate complex equilibrium (eq 2), and the unexpected degenerate process of eq 3. For M = I (n = 1), all three processes occur on the DNMR time scale between −78 and −115 °C.

$$2(\mathrm{PhLi})_1 \overset{K_1}{\rightleftharpoons} (\mathrm{PhLi})_2 \tag{1}$$

$$(\mathrm{PhLi})_1 + (\mathrm{Ph})_n\mathrm{M} \overset{K_2}{\rightleftharpoons} (\mathrm{Ph})_{n+1}\mathrm{M}^-\mathrm{Li}^+ \tag{2}$$

$$(\mathrm{Ph})_n\mathrm{M} + (\mathrm{Ph})_{n+1}\mathrm{M}^-\mathrm{Li}^+ \overset{K_3}{\rightleftharpoons} (\mathrm{Ph})_{n+1}\mathrm{M}^-\mathrm{Li}^+ + (\mathrm{Ph})_n\mathrm{M} \tag{3}$$

^7Li and ^{13}C NMR spectra of phenyllithium solutions that contain increasing amounts of metalloid species are presented in Figures 1 and 2. These define the exchange processes in qualitative terms. The addition of diphenylmercury to phenyllithium (Figure 1A) produced a new sharp signal in the ^7Li NMR at δ −0.9,[8] which has been assigned to tetrahedrally coordinated $Li^+(THF)_4$[9] of $Ph_3Hg^-Li^+$.[10] Since only this signal was present when exactly 1 equiv of diphenylmercury had been added, the formation constant of $Ph_3Hg^-Li^+$ (K_2 of eq 2) is large. With less than 1 equiv, the remaining $(PhLi)_n$ signals were identical with those of phenyllithium itself at the appropriate concentration. Hence the exchange processes depicted in eqs 2 and 3, where M = Hg and n = 2, are both slow on the NMR time scale at −105 °C.

The addition of 1 equiv of diphenyl telluride (Figure 1B) or iodobenzene (Figure 1C) produced a similar signal at δ −0.8, which we have assigned to the lithium of $Ph_3Te^-Li^+$ and $Ph_2I^-Li^+$. As with $Ph_3Hg^-Li^+$, the formation constant (K_2) for each is large, since no phenyllithium signals were visible. At intermediate points in the titrations, the spectra contained broad signals indicative of rapid exchange. While the $(PhLi)_2$ signal at δ 1.5 was unaffected, as it was in the mercury case, $(PhLi)_1$ and ate complex signals had become part of the broad signal between δ −1 and 1. We assign this averaged signal to $(PhLi)_1$ and $Ph_3Te^-Li^+$ (Figure

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[9] Lithium-7 is quadrupolar ($I = {}^3/_2$) and gives sharp lines only in highly symmetrical (tetrahedral or octahedral) environments.

[10] The rate of reaction of phenyllithium in THF with butyl iodide to form butylbenzene is reduced when diphenylmercury is added due to the formation of $Ph_3Hg^-Li^+$.[1a] Diphenylmercury does not form significant amounts of $Ph_3Hg^-Li^+$ in *ether* at room temperature: Wittig, G.; Meyer, F. J.; Lange, G. *Justus Liebigs Ann. Chem.* **1951**, *571*, 167. No simple triorganomercury ate complexes have been observed, but they have been suggested as intermediates: Seitz, L. M.; Hall, S. D. *J. Organomet. Chem.* **1968**, *15*, C7. Trisilyl complexes are well-known: Sadurski, E. A.; Ilsley, W. H.; Thomas, R. D.; Glick, M. D.; Oliver, J. P. *J. Am. Chem. Soc.* **1978**, *100*, 7761. See also: Schmidbauer, H.; Gasser, O.; Fraser, T. E.; Ebsworth, E. A. V. *J. Chem. Soc., Chem. Commun.* 1977, 334.

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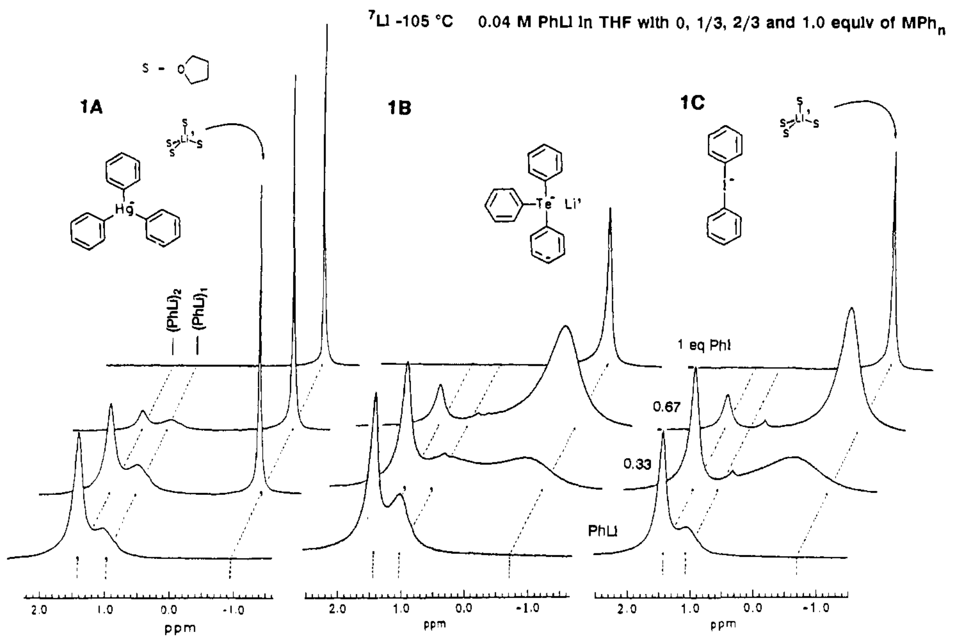

Figure 1. ^7Li NMR spectra[8] (194.32 MHz) in THF at −105 °C of 0.04 M PhLi solutions with 0, 0.33, 0.67, and 1.0 equiv of (A) Ph_2Hg, (B) Ph_2Te, and (C) PhI added.

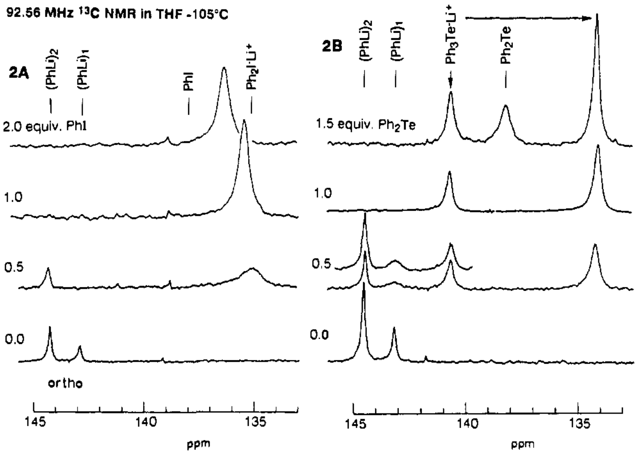

Figure 2. Ortho carbons in the ^{13}C NMR spectra: 0.08 M PhLi with 0, 0.5, and 1.0 equiv and an excess of (A) PhI and (B) Ph_2Te. The inset in B is 0.12 M PhLi, 0.04 M Ph_2Te.

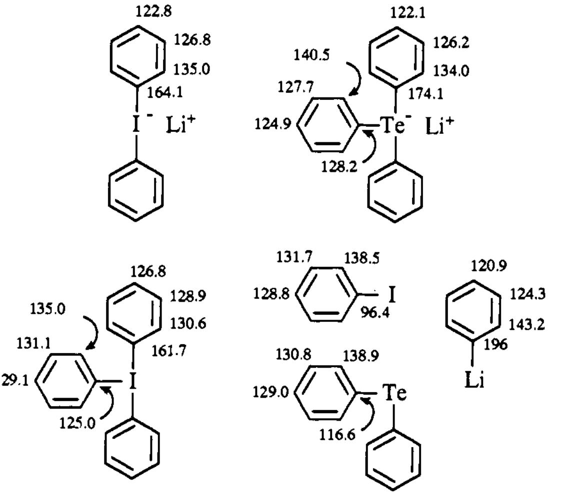

Figure 3. ^{13}C NMR δ in THF at −105 °C.

1B)[11] or $(PhLi)_1$ or $Ph_2I^-Li^+$ (Figure 1C)[7a] in dynamic equilibrium, with the tellurium being slightly below coalescence, and the iodine case slightly above it.

The ^{13}C NMR spectra of parts A and B of Figure 2 (ortho carbons only shown) fully support the interpretation above and provide additional information about the structure of the ate complexes and the dynamics of the Li/I and Li/Te exchange reactions. The aryl resonances for $Ph_3Te^-Li^+$ are nonequivalent in a 2:1 ratio, as expected for a T-shaped structure.[1c,12] The pattern of "apical" phenyl chemical shifts (Figure 3), particularly for the strongly downfield ipso carbons, is strikingly similar to that of $Ph_2I^-Li^+$ and to that of the isoelectronic Ph_3I first prepared by Wittig.[1c,13]

The $(PhLi)_1$ and $Ph_3Te^-Li^+$ signals in Figure 2 (0.5 equiv) are below coalescence, and those of $Ph_2I^-Li^+$ are near coalescence, as in the ^7Li NMR spectra of Figure 1. When excess diphenyl telluride had been added, its signals could be seen. In contrast, addition of excess iodobenzene produced only a single broad resonance, which is the dynamic average of PhI and $Ph_2I^-Li^+$. When more PhI was added, the signal moved as expected for rapid exchange. In this respect, the behavior is similar to that observed in THF/HMPA solution,[1b] and for the same reason: there is an exchange process rapid on the NMR time scale in which Ph_2I^- reversibly donates Ph^- to PhI (eq 3). Thus $Ph_2I^-Li^+$ is a more reactive Ph^- donor than $Ph_3Te^-Li^+$. In fact these spectra provide no direct evidence that the tellurium ate complex reacts at all with diphenyl telluride (eq 3).

The spectroscopic results of Figures 1 and 2 demonstrate in a most direct fashion that the monomer–dimer PhLi equilibrium is slow at −105 °C and that the *dimer does not participate in the exchange of PhI or Ph_2Te on the NMR time scale, but that the monomer does.*[14] They also show that the ate complex equilibrium (eq 2) is fastest for PhI, slightly slower for Ph_2Te, and much slower

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(11) No triorganotellurium ate complexes have been observed. Some evidence for lithium pentaphenyltellurate has been reported: Wittig, G.; Fritz, H. *Justus Liebigs. Ann. Chem.* **1952**, *577*, 39. We have been able to confirm that $Ph_3Te^+Cl^-$ reacts with 2 equiv of PhLi in THF to produce $Ph_5Te^-Li^+$.

(12) The ^{13}C NMR spectrum of $Ph_3Hg^-Li^+$, on the other hand, shows only a single set of resonances, as expected for a trigonal structure.[10]

(13) Wittig, G.; Clauss, K. *Justus Liebigs Ann. Chem.* **1952**, *578*, 136. Clauss, K. *Chem. Ber.* **1955**, *88*, 268.

(14) The lower aggregates of organolithium reagents are usually more reactive: McGarrity, J. F.; Ogle, C. A.; Brich, Z.; Loosli, H.-R. *J. Am. Chem. Soc.* **1985**, *107*, 1810. Holm, T. *Acta Chem. Scand.* **1978**, *B32*, 162. Exceptions have been reported: West, P.; Waack, R.; Purmort, J. I. *J. Am. Chem. Soc.* **1970**, *92*, 840. Frequently the dominant aggregate is the reactive species[7b]: Bartlett, P. D.; Goebel, C. V.; Weber, W. P. *J. Am. Chem. Soc.* **1969**, *91*, 7425.

for Ph_2Hg, K_2 is large for all three at −105 °C.[15]

We have carried out a quantitative measurement of K_2 for PhI. Between −75 and −20 °C, all species [$(PhLi)_2$, $(PhLi)_1$, PhI, $Ph_2I^-Li^+$] are in rapid equilibrium (above coalescence). We have measured values for $\Delta H°$ and $\Delta S°$ for the phenyllithium monomer–dimer equilibrium,[16] so the population of the other species (PhI, $Ph_2I^-Li^+$) can be calculated from the average chemical shift of the ortho carbon in the ^{13}C NMR spectrum. We obtained $\Delta H° = -9.9 \pm 0.6$ kcal/mol and $\Delta S° = -44 \pm 9$ eu for eq 2.[17] Thus K_2 is 0.02 M^{-1} at 0 °C (a solution 0.1 M in PhLi and PhI is 0.1% associated), 30 M^{-1} at −78 °C (44% associated), and 1780 M^{-1} at −105 °C (92% associated). The corresponding association constant K_2 for Ph_2Te is almost the same as for PhI, as shown by an experiment in which 1 equiv of PhI was added to a solution of $Ph_3Te^-Li^+$ in THF at −105 °C.[20] Half of the $Ph_3Te^-Li^+$ was converted to $Ph_2I^-Li^+$.

Summary. Iodine, tellurium, and mercury ate complexes have been characterized as intermediates in the PhLi/Ph_nM (M = I, Te, Hg) exchange reaction in THF solution at low temperatures. Monomeric phenyllithium is the reactive species in the Li/I and Li/Te exchanges. The formation constant for $Ph_2I^-Li^+$ is strongly temperature dependent, consistent with the powerful solvent-ordering capacity of a solvent-separated ion pair.[18]

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(15) No PhLi or Ph_2Te could be detected in the ^{13}C NMR spectrum of 1:1 PhLi/Ph_2Te solution at −105 °C (5–10% would have been seen).

(16) For $2(PhLi)_1 \rightleftharpoons (PhLi)_2$, $\Delta H^\ddagger = -7.4 \pm 0.1$ kcal/mol, $\Delta S^\ddagger = -2.5 \pm 1$ eu, $\Delta H° = 0.5 \pm 0.7$ kcal/mol, and $\Delta S° = 10 \pm 6$ eu. Green, D. P. Ph.D. Thesis, University of Wisconsin, 1989.

(17) For the equilibration of lithium fluorenide contact and separated ion pairs in THF: $\Delta H° = -7.5$ kcal/mol and $\Delta S° = -22$ eu.[18] Rate studies for the PhLi/ArBr exchange in ether ($\Delta H^\ddagger = 14$ kcal/mol, $\Delta S^\ddagger = -24$ eu)[7b] and THF (223 °K, $\Delta H^\ddagger = 9.5$ kcal/mol, $\Delta S^\ddagger = -30$ eu)[19] show negative entropies of activation.

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Desulfurization of Thiophene and Thiophenol by a Sulfido–Cobalt–Molybdenum Cluster: Toward a Homogeneous Hydrodesulfurization Catalyst

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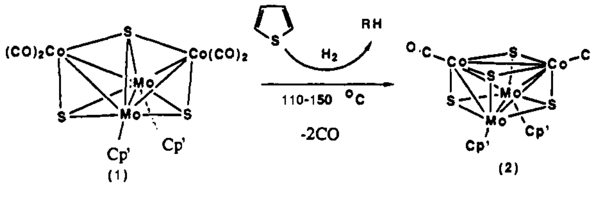
Conventional hydrodesulfurization (HDS)[1] catalysts are prepared by the coimpregnation of Mo (or W) and Co (or Ni) salts on an alumina support, followed by sulfidation with H_2S/H_2. The active site is thought to consist of small crystallites of MoS_2 with Co atoms coordinated to sulfur at the edges of the basal planes.[2] The mechanism of HDS has been studied through the aid of surface science and organometallic modeling studies,[3] but as yet there is no clear consensus on the detailed structure of the active site or on the mechanistic details of the HDS reaction itself.[4]

We have prepared sulfided bimetallic clusters[5] which mimic the metal composition of commerical HDS catalysts and have shown that one such cluster, $Cp'_2Mo_2Co_2(CO)_4S_3$ ($Cp' = CH_3C_5H_4$) (**1**), when supported on alumina and sulfided, produces the same active site as found in commercial Mo/Co/S catalysts.[6] We have begun an investigation of the solution chemistry of cluster **1** to see if a homogeneous analogue of the surface-bound HDS catalyst may be constructed.

Cluster **1** reacts under N_2 with thiophene[7] at temperatures ranging from 110 to 150 °C to give a >90% isolated yield of $Cp'_2Mo_2Co_2(CO)_2S_4$[9] (**2**), with the production of some black, insoluble material. If **1** is heated to 150 °C in toluene under N_2, no conversion to **2** takes place and **1** can be recovered unchanged. This indicates that **2** is not a decomposition product of **1**. The high yields (quantitative by NMR) of **2** obtained also indicate that **1** does not decompose to **2**. If the reaction is conducted under 200 psi (ca. 15 atm) of H_2, cluster **2** is again produced in high yields, the black solid is not formed, and analysis of the gases revealed the presence of ethane, propane, and butanes along with significant amounts of ethene, propene, and butene (eq 1). Compared to the hydrocarbon product slate from heterogeneous HDS catalysis, cracking and hydrogenation are more prevalent in the homogeneous reaction.



(1)

Reactions of thiophene with mononuclear organometallics result in the formation of either π-complexes or S-bound thiophene complexes, but the desulfurization of these complexes has not been reported.[11] There is only one other example of the reaction of thiophene with a transition-metal cluster: the reaction of $Fe_3(CO)_{12}$ with thiophene gives FeS and low yields of the dimetallic species $Fe_2(CO)_6(C_4H_4)$.[12] Thus, eq 1 represents the first desulfurization of thiophene accompanied by a clean, high-yield organometallic conversion.[13]

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(7) The reaction was carried out in a glass-lined Parr stainless steel reactor with thiophene as the solvent. Thiophene was purified according to the procedure outlined by Angelici (see ref 8) and was found to be stable when heated in the reactor to 150 °C in the presence of H_2. In a typical reaction, 100--200 mg of **1**, 30–40 mL of thiophene, and 200 psig of H_2 were used.

(8) Spies, G. H.; Angelici, R. J. *Organometallics* **1987**, *6*, 1897.

(9) Complete conversion of **1** to **2** was obtained within 2 h at 150 °C and 6 h at 110 °C. Cluster **2** was isolated by decanting the reaction mixture from the reactor (inside a N_2-filled glovebag) into a Schlenk flask, followed by removal of the thiophene under vacuum. The solid residue was extracted with dichloromethane and filtered. Evaporation of the dichloromethane under vacuum resulted in pure **2** (as evidenced by ^1H NMR). The Cp analogue of **2** has been synthesized previously (see ref 10), and **2** could be identified on the basis of IR, ^1H NMR, MS, and elemental analysis. For **2**: IR (KBr) 1960, 1935 cm^{-1} (ν_{CO}); ^1H NMR (C_6D_6) δ 5.08 (m, 4 H), 4.86 (m, 4 H), 1.64 (s, 6 H); MS (EI) 652 (P^+), 624 ($P^+ - CO$), 596 ($P^+ - 2CO$); Anal. Calcd for $C_{14}H_{14}Mo_2Co_2O_2S_4$: C, 25.68; H, 1.93. Found: C, 25.78; H, 2.16.

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