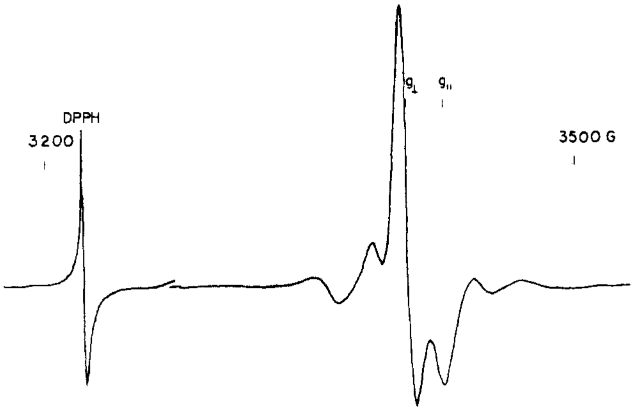

**Figure 2.** EPR spectrum of a $10^{-3}$ M frozen THF solution of $\{Mo_2[O_2P(OC_6H_5)_2]_4\}[PF_6]$ at 77 K (9.141 GHz) with diphenylpicrylhydrazyl (DPPH) standard.

duced the mixed-valence species $[Mo_2(DPP)_4]PF_6$ in 65% isolated yield. The cyclic and linear sweep voltammograms of this species showed a reduction wave at −0.52 V vs the ferrocene standard.

Physical properties of $Mo_2(DPP)_4$ and $[Mo_2(DPP)_4]PF_6$ resemble those of isoelectronic partners $Mo_2(HPO_4)_4^{4-}$–$Mo_2(HPO_4)_4^{3-}$[13,15] and $Mo_2(SO_4)_4^{4-}$–$Mo_2(SO_4)_4^{3-}$.[16,17] The quadruply bonded complex exhibits a weak absorption assigned to the $\delta \rightarrow \delta^*$ transition at 520 nm ($\epsilon$ = 160 $M^{-1}$ $cm^{-1}$). On oxidation the $\delta \rightarrow \delta^*$ transition red-shifts in the mixed-valence complex to 1530 nm ($\epsilon$ = 140 $M^{-1}$ $cm^{-1}$), and vibronic structure corresponding to a 303-$cm^{-1}$ progression (six members observed) in the Mo–Mo stretch is observed on the absorption in solution at room temperature. This is similar to $Mo_2(SO_4)_4^{3-}$, which absorbs at 1405 nm ($\epsilon$ = 143 $M^{-1}$ $cm^{-1}$) and exhibits a 350-$cm^{-1}$ vibrational progression in solution, and to $Mo_2(HPO_4)_4^{3-}$, which absorbs at 1438 nm ($\epsilon$ = 180 $M^{-1}$ $cm^{-1}$) and exhibits a 334-$cm^{-1}$ vibrational progression. The large charge difference between $Mo_2(DPP)_4$ and $Mo_2(SO_4)_4^{4-}$ or $Mo_2(HPO_4)_4^{4-}$ allows the former complex to be dissolved in organic solvents, which may be useful in studies of the photoredox behavior[15] of these complexes.

The magnetic properties of $Mo_2(DPP)_4^+$ also resemble those of the other mixed-valence Mo(II)–Mo(III) dimers.[13,15-17] In THF solution the $\mu_{eff}$ of $[Mo_2(DPP)_4]PF_6$ was 1.58 $\mu_B$, determined by the Evans method. At 77 K in a THF glass an EPR spectrum (9.141 GHz) similar to that of $Mo_2(SO_4)_4^{3-}$ was observed (Figure 2) whose line shape could be fit by assuming $g_\parallel$ = 1.899 and $g_\perp$ = 1.911 and by assuming average hyperfine splitting constants from both $^{95}Mo$ and $^{97}Mo$ of $A_\parallel$ = 22.7 G and $A_\perp$ = 12.8 G.

This work establishes the ability of $PO_2(OPh)_2^-$ to bridge two metal centers in a dinuclear metal–metal-bonded complex. Qualitative observations suggest the ligand binds more weakly than acetate, but better than triflate. Nucleophilic attack by $OH^-$ at phosphorus in the bound diester was not observed because of rapid cleavage of the ligand from the dinuclear center. In the context of using a dinuclear metal complex to introduce strain into a phosphate diester complex, this seems unlikely given the opening of the bridging O–P–O angle on binding. However, expanding the O–P–O angle toward 120° may stabilize a trigonal-bipyramidal transition state (with $MO_2P$ equatorial), which is often invoked[6] for nucleophilic substitutions at phosphorus. This strategy is being explored with less labile metal complexes.

**Acknowledgment.** This research was supported by the Army Research Office (Grant DAAG29-85-K0263). We thank the DoD University Research Instrumentation Program (Grant DAAL03-87-G-0071) for funds to purchase the X-ray diffractometer. We thank Prof. T. Don Tilley for helpful discussions and Prof. William H. Armstrong for providing additional crystallographic data for the structure in ref 1.

**Supplementary Material Available:** Tables of crystallographic data, bond distances, bond angles, anisotropic displacement parameters, and hydrogen atom coordinates (7 pages); a table of observed and calculated structure factors (19 pages). Ordering information is given on any current masthead page.

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# Solid-State Structure of $[Li(\mu\text{-}OAr\text{-}2,6\text{-}Ph_2)_3Sn]$ ($OAr\text{-}2,6\text{-}Ph_2$ = 2,6-Diphenylphenoxide): A Cage Compound Containing a Pyramidal Lithium Atom

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*Received July 6, 1988*

There has been a rapid growth of interest over the last few years in the solid-state structures adopted by main-group-element compounds.[2] Two areas of particular interest are the structural impact of sterically demanding functional groups[3] and the importance of $\pi$-bonding between main-group elements and unsaturated organic systems.[4] During our studies of the transition-metal chemistry associated with the sterically demanding aryloxide ligand 2,6-diphenylphenoxide ($OAr\text{-}2,6\text{-}Ph_2$), we have demonstrated an ability for this group to chelate to metal centers either through formation of a metal–carbon $\sigma$-bond or else via an $\eta^6$-interaction involving one of the side-chain aryl groups.[5] We have recently extended our studies of the chemistry of this and related ligands to derivatives of the main-group metals, and some complementary reactions involving CH bond activation have been uncovered.[6] However, the purpose of this note is to describe the isolation and structure of the title compound, a five-membered-cage material that contains a formally three-coordinate lithium atom apparently stabilized by weak interactions with the side-chain aryl groups of three 2,6-diphenylphenoxide ligands.

## Results and Discussions

The addition of 2 equiv of $LiOAr\text{-}2,6\text{-}Ph_2$ to $SnCl_4$ in benzene or toluene results in the formation of the cyclometalated dimer $[Cl_2Sn(OC_6H_3Ph\text{-}C_6H_4)]_2$ held together by bridging oxygen atoms.[6] However, a second only slightly hydrocarbon-soluble minor product was also obtained as large colorless blocks from hot toluene. A solid-state X-ray diffraction analysis of this minor product (vide infra) showed it to have the stoichiometry $[LiSn(OAr\text{-}2,6\text{-}Ph_2)_3]$ (**1**) containing a Sn(II) metal center formed by reduction of Sn(IV) in the reaction mixture. Mass spectrometric data for this component showed a strong parent molecular ion, **1**$^+$, with no evidence for higher oligomers. A much more rational

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**Table I.** Positional and Thermal Parameters and Their Estimated Standard Deviations[a]

| atom | *x* | *y* | *z* | *B*, Å$^2$ |
|---|---|---|---|---|
| Sn | 0.00520 | 0.00520 | 0.00520 | 3.937 (2) |
| Li | 0.1785 (8) | 0.1785 | 0.1785 | 5.39 (7) |
| O(10) | 0.2075 (2) | 0.0308 (3) | 0.0598 (2) | 4.37 (5) |
| C(11) | 0.2743 (3) | −0.0671 (4) | 0.0444 (4) | 4.25 (7) |
| C(12) | 0.2648 (4) | −0.1414 (4) | −0.0737 (4) | 4.94 (9) |
| C(13) | 0.3280 (5) | −0.2477 (4) | −0.0833 (5) | 6.1 (1) |
| C(14) | 0.4007 (5) | −0.2781 (4) | 0.0202 (7) | 7.1 (1) |
| C(15) | 0.4122 (4) | −0.2038 (5) | 0.1344 (5) | 6.2 (1) |
| C(16) | 0.3517 (4) | −0.0960 (4) | 0.1502 (4) | 5.09 (9) |
| C(121) | 0.1862 (4) | −0.1125 (4) | −0.1876 (4) | 5.21 (9) |
| C(122) | 0.1895 (5) | 0.0128 (5) | −0.2146 (4) | 5.50 (9) |
| C(123) | 0.1113 (6) | 0.0406 (6) | −0.3167 (5) | 7.6 (1) |
| C(124) | 0.0318 (8) | −0.0543 (7) | −0.3956 (6) | 9.3 (2) |
| C(125) | 0.0283 (8) | −0.1779 (8) | −0.3719 (7) | 10.8 (2) |
| C(126) | 0.1045 (7) | −0.2066 (5) | −0.2689 (6) | 8.1 (1) |
| C(161) | 0.3671 (4) | −0.0156 (5) | 0.2740 (4) | 5.9 (1) |
| C(162) | 0.4097 (5) | 0.1120 (6) | 0.2888 (5) | 6.9 (1) |
| C(163) | 0.4306 (7) | 0.1850 (8) | 0.4075 (7) | 9.4 (2) |
| C(164) | 0.4094 (8) | 0.1345 (9) | 0.5130 (7) | 12.0 (2) |
| C(165) | 0.3667 (7) | 0.008 (1) | 0.5004 (6) | 11.3 (2) |
| C(166) | 0.3437 (5) | −0.0695 (7) | 0.3822 (6) | 8.2 (2) |

[a] Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as $(4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

**Table II.** Selected Bond Distances (Å) and Angles (deg) for [Li($\mu$-OAr-2,6-Ph$_2$)$_3$Sn] (**1**)

| | | | |
|---|---|---|---|
| Sn–Li | 2.784 (4) | Li–O(10) | 1.988 (4) |
| Sn–O(10) | 2.150 (2) | O(10)–C(11) | 1.351 (5) |
| O(10)–Sn–O(10) | 76.0 (1) | Sn–O(10)–C(11) | 121.5 (2) |
| O(10)–Li–O(10) | 83.5 (1) | Li–O(10)–C(11) | 145.2 (3) |
| Sn–O(10)–Li | 84.5 (2) | | |

and higher yield synthesis of **1** was then achieved by using $SnCl_2$ as the initial tin substrate (eq 1).

$$SnCl_2 + 3LiOAr\text{-}2,6\text{-}Ph_2 \rightarrow \underset{\mathbf{1}}{[LiSn(OAr\text{-}2,6\text{-}Ph_2)_3]} + 2LiCl \qquad (1)$$

An ORTEP view of **1** generated by X-ray diffraction analysis along with the labeling scheme is shown in Figure 1, while Tables I and II contain positional and thermal parameters and selected bond distances and angles, respectively. The $\sigma$-bonding framework of the molecule can be seen to consist of a central cage of five atoms. The pyramidal lithium and tin metal atoms are bridged by three aryloxide oxygen atoms that are identical due to a crystallographically imposed $C_3$ axis of symmetry running along the Sn–Li vector. The tin atom in **1** appears to occupy a coordination environment very similar to that found in the related compound [Tl($\mu$-OBu$^t$)$_3$Sn] reported by Veith and Rosler.[7] However, the coordination environment about the lithium atom in **1** does not appear normal for this metal. Numerous compounds reported in the literature have been shown to contain three-coordinate lithium atoms surrounded by oxygen donor atoms.[8,9] In these cases a trigonal-planar array of oxygen atoms about the Li center is seen. In compound **1**, a pyramidal array of oxygen atoms is observed with O–Li–O angles of only 83.5 (1)° being present. Under normal circumstances one would expect this situation to lead to the formation of a four-coordinate tetrahedral lithium center by either the coordination of a neutral donor ligand or more extensive oligomerization through either aryloxide bridging or Sn→Li bonding. However, the steric demands of the bulky aryloxide group apparently preclude either of these from taking place. Examining the region around the lithium atom, one finds that a relatively close contact occurs between one of the ortho carbons of one of the aryloxide side-chain phenyl groups (Figure 1). The distance of the aryl ring to this carbon, 2.828 (8) Å, is at the outer fringes of distances typically found between electrostatically bound lithium ions and carbon-based $\pi$-clouds.[2,4,10] Hence, the bending in of these three aryl rings into the gap around the lithium atom may reflect purely packing effects, although it is also possible that these groups are helping to stabilize the electron-deficient lithium metal center.

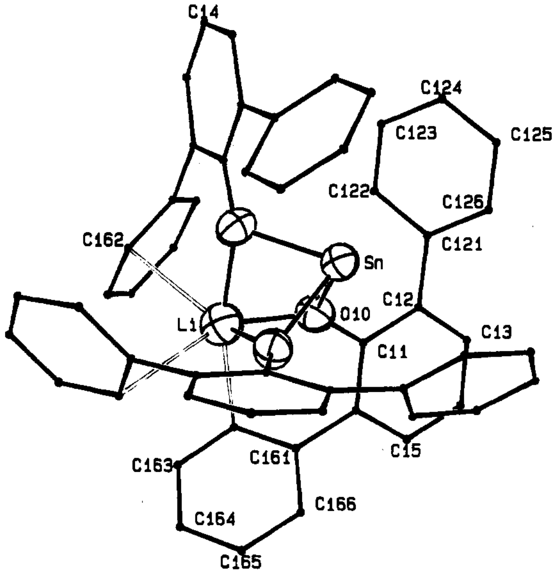

**Figure 1.** ORTEP view of **1**, emphasizing the central cage and indicating the Li–C(162) contact.

**Table III.** Crystallographic Data

| | |
|---|---|
| $SnO_3C_{60}LiH_{45}$ | $T$ = 19 °C |
| fw = 939.66 | $\lambda$ = 0.71073 Å |
| space group: $R_3$ (No. 146) | $\rho_{calc}$ = 3.880 g cm$^{-3}$ |
| $a$ = 10.741 (2) Å | $\mu$ = 17.18 cm$^{-1}$ |
| $\alpha$ = 97.31 (1)° | transmission coeff = 0.991–0.400 |
| $V$ = 1206.2 (8) Å$^3$ | $R(F_o)$ = 0.021 |
| $Z$ = 3 | $R_w(F_o)$ = 0.027 |

Another intriguing possibility is the notion that the electron-deficient lithium center is being stabilized not by interaction with the arene $\pi$-cloud but by *agostic* interactions with the three *o*-CH bonds.[11] Although typically associated with high-valent, electron-deficient d-block, lanthanide, and actinide metal centers,[11,12] close contacts between hydrocarbon fragments of ligands and main-group metals have also been noted.[13] However, the Li–H(162) distance of 2.695 (9) Å is long, indicating that any *agostic* interactions present must be very weak.

## Experimental Section

**Synthesis of [Li($\mu$-OAr-2,6-Ph$_2$)$_3$Sn].** To a suspension of LiOAr-2,6-Ph$_2$ (5.6 g, 22 mmol) in benzene (50 mL) was added solid $SnCl_2$ (1.34 g, 7 mmol), and the mixture was refluxed for 10 h. The resulting orange suspension was cooled and the solvent removed in vacuo. The orange mass was washed with hexane to remove the orange color before being extracted with hot benzene. Concentration and cooling of the colorless extract resulted in the formation of the product as large crystalline blocks. Yield: 0.55 g (9%). IR (cm$^{-1}$, Nujol mull): 1598 (m), 1578 (m), 1494 (m), 1378 (w), 1310 (m), 1280 (s), 1250 (s), 1182 (w), 1082 (m), 1068

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**Crystallographic Studies.** Details of the data collection procedures and structure refinement methods have been given previously.[12a] Table III contains some pertinent crystallographic data. The crystals were examined under dry, deoxygenated Nujol and mounted in an appropriately sized capillary with epoxy resin. The hydrogen atom positions were calculated after several cycles of anisotropic refinement assuming idealized geometries and a bond distance of 0.95 Å. The monitoring of three intensity standards every 5000 s of beam time indicated no decay. No correction for extinction was performed.

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**Supplementary Material Available:** Tables of crystal data and data collection parameters, non-hydrogen and hydrogen positional and thermal parameters, anisotropic temperature factors, and full bond distances and angles (9 pages); tables of observed and calculated structure factors (8 pages). Ordering information is given on any current masthead page.

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# Synthesis and Characterization of the Gadolinium(3+) Complex of DOTA-Propylamide: A Model DOTA-Protein Conjugate

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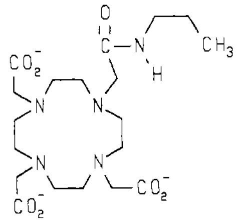
Several types of paramagnetic metal ion–chelate complexes have been proposed for use as contrast-enhancing agents in magnetic resonance imaging (MRI). The chemical properties and the distinct advantages of these different, largely inorganic, species have been extensively discussed in a recent review.[1] Gd(DTPA)$^{2-}$ is the first of these agents to be approved for use in humans and the standard to which newer agents are compared. The macrocyclic complex Gd(DOTA)$^-$ has several properties that make it a better candidate than Gd(DTPA)$^{2-}$, including a more favorable thermodynamic stability constant ($\log K = 24.7$ for Gd(DOTA)$^-$ versus 22.3 for Gd(DTPA)$^{2-}$),[2] a significantly greater in vitro stability in blood serum,[3] which apparently translates into a lower toxicity in animals,[4] and a significantly greater solvent (water) proton relaxivity at low fields.[5] Both complexes contain one inner-sphere water molecule that exchanges rapidly with solvent water, thereby providing an efficient mechanism for relaxing water protons with a relatively small concentration of agent.

The utility of these paramagnetic agents would be greatly expanded if they could be covalently attached to macromolecules that could target or concentrate the agent to specific tissues, lesions, or cells. Several DTPA-conjugated macromolecules have been reported[6-10] and their advantages outlined.[1] In most cases, the chelate was conjugated to the macromolecule via amide or ester linkages by simply mixing the readily available DTPA-dianhydride with the macromolecule of interest. However, this reaction is known to result in a mixture of mono- and diconjugated chelates[11] that have considerably reduced affinities[12] for Gd$^{3+}$. Interestingly, we have also found that the conversion of one or even two terminal carboxyl groups of DTPA into a propylamine or ester gives ligands which form complexes with Gd$^{3+}$ that have on average fewer inner-sphere water molecules than Gd(DTPA)$^{2-}$ at room temperature and below.[13] This suggests that the amide or ester moieties in these complexes, although weakly bound,[12] interact well enough to preclude the entrance of additional water molecules into the inner coordination sphere of Gd$^{3+}$.

**Chart I**



We now report the synthesis, rate of formation, thermodynamic stability, and field dependence of the longitudinal relaxation rate ($1/T_1$) of solvent water protons in solutions of Gd(DOTA–PA), which serves as a model of a monoconjugated DOTA macromolecule. The data indicate that monoconjugated DOTA macromolecules could offer certain advantages over the corresponding DTPA-conjugated systems in the design of in vivo targeted MRI contrast agents.

## Materials and Methods

**DOTA-PA.** The monopropylamide of DOTA was synthesized by using methods reported by Krejcarek and Tucker,[14] with minor modifications. In a typical synthesis, the free-acid form of DOTA (0.17 g, 0.29 mmol) and triethylamine (1.15 mmol) were dissolved in dry DMSO (15 mL) by gentle warming. The resulting clear solution was cooled to room temperature, and isobutyl chloroformate (0.29 mmol) was added dropwise, followed by addition of an excess (2.07 mmol) of dry *n*-propylamine. The resulting mixture was stirred for 30 min and filtered and the DMSO distilled off under vacuum. The residue was dissolved into water, and the solution was loaded onto a 1.5 × 20 cm Dowex-1 anion-exchange column (acetate form), eluted first with water to remove excess amines, followed by a linear 0–0.5 M acetic acid gradient (300 mL total) as previously described.[12] The fractions corresponding to the first peak were combined, the pH was adjusted to 2 with HCl to protonate the acetate, and the sample was freeze-dried and identified by $^1$H and $^{13}$C NMR and elemental analysis as the monopropylamide of DOTA (see Chart I). The overall yield was about 30%. Anal. Calcd for $C_{19}H_{31}N_5O_7Cl\cdot 5H_2O$ (white solid): C, 40.2; H, 7.3; N, 12.4; Cl, 6.6; O, 33.9. Found: C, 40.2;

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