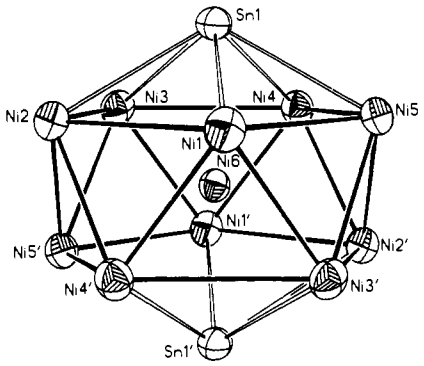

Figure 1. Ni-centered 1,12-$Ni_{10}Sn_2$ icosahedral cage in $[Ni_{11}(SnMe)_2(CO)_{18}]^{2-}$ (**3**) of crystallographic C_i-$\bar{1}$ site symmetry. This pentagonal antiprism of 10 surface Ni(s) with an interstitial Ni(i) and two capping Sn atoms has the following mean distances under assumed D_{5d}-$\bar{1}02m$ symmetry: Ni(i)-Sn, 2.351 (1) Å; Ni(i)-Ni(s), 2.58 Å; Ni(s)-Sn, 2.72 Å; intrapentagonal Ni(s)-Ni(s'), 2.79 Å; interpentagonal Ni(s)-Ni(s'), 2.51 Å. Atomic thermal ellipsoids are drawn at the 35% probability level.

that the bonding CVOs are occupied but the corresponding antibonding CVOs are empty. In the case of **2**, **3**, or **6**, the "extra" 8 electrons would then necessarily populate *antibonding tangential* cage LUMOs.[22,23] However, the structural parameters provide evidence that the antibonding tangential cage LUMOs in **7** remain empty in **2**, **3**, or **6** when the Ni(i) is added to the icosahedral cavity.

The only reasonable electronic scheme involves a breakdown of the CVO model[14] with the 10 valence d electrons of Ni(i) in **2**, **3**, or **6** occupying the five *antibonding radial* Ni(i)-cage CVOs. One major consequence is that the "net" bonding effects due to the 3d Ni(i) AOs are essentially nullified; the unusually short Ni(i)-E bonds in **2**, **3**, or **6** must then be attributed to strong interactions involving the 4s,4p Ni(i) AOs. This experimentally deduced proposal is consistent with the view that the d^{10} Ni(i) contributes its empty 4s,4p AOs but no "net" bonding skeletal electron pairs in stabilizing the $Ni_{10}E_2$ cage. Weak radial interactions between the filled 3d Ni(i) AOs and appropriate cage orbitals in **2**, **3**, or **6** (producing *occupied* bonding and antibonding MOs) are readily rationalized for late-first-row transition metals because their high effective nuclear charges give rise to relatively small, low-energy d AOs. This structural-bonding analysis is in harmony with that reported[13] for **6**.

Electrochemical measurements indicate that **2** and **3**, which do not conform to the PSEP model,[24] can be reversibly oxidized and reduced. Work in progress includes attempts to isolate these redoxed species for further structural-bonding studies.

Acknowledgment. We are grateful to the National Science Foundation for support of this research (Grant CHE-9013059).

Supplementary Material Available: Tables listing the atomic parameters, interatomic distances, and bond angles for **2-5** (32 pages). Ordering information is given on any current masthead page.

(22) The 13 skeletal electron pairs in a regular icosahedral I_h cage (e.g., $[B_{12}H_{12}]^{2-}$) were shown[23] to occupy bonding a_g, t_{1u}, h_g, and g_u MOs; the quadruply degenerate g_g LUMOs, which are composed of symmetry-adapted antibonding combinations of tangential surface orbitals, transform as e_{1g} + e_{2g} under the lower pseudo-D_{5d} symmetry of the 1,12-$Ni_{10}E_2$ cage.

(23) (a) Hoffmann, R.; Lipscomb, W. N. *J. Chem. Phys.* **1962**, *36*, 2179-2189. (b) Teo, B. K. *Inorg. Chem.* **1985**, *24*, 1627-1638. (c) Johnston, R. L.; Mingos, D. M. P. *J. Chem. Soc., Dalton Trans.* **1987**, 647-656. (d) Mingos, D. M. P.; Zhenyang, L. *J. Organomet. Chem.* **1988**, *341*, 523-534.

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Lithium Pentakis(dimethylsilyl)cyclopentadienide and Formation of Isolable Coordination Complexes with Ketones: $[(R_2C{=}O)Li{\cdot}\{C_5(SiMe_2H)_5\}]$[1]

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Although polysilylated cyclopentadienide anions have been of interest for a long time,[2] persilylated cyclopentadienide has been elusive.[3] As a part of the study on persilylated π-electron systems,[4] we report herein the preparation and interesting properties of lithium pentakis(dimethylsilyl)cyclopentadienide as the first example of persilylated cyclopentadienide anions.

Treatment of hexakis(dimethylsilyl)cyclopentadiene (**1**, 200 mg, 0.48 mmol), prepared by the reaction of hexabromocyclopentadiene and dimethylchlorosilane in the presence of magnesium,[5] with *n*-BuLi (0.63 mmol) in dry oxygen-free hexane/THF at room temperature led to the formation of [pentakis(dimethylsilyl)cyclopentadienyl]lithium by cleavage of an Si-C bond. Removal of the solvent afforded a THF complex of the anion, $[(THF)Li{\cdot}\{C_5(SiMe_2H)_5\}]$ (**2**), as colorless solids.[6]

Quite expectedly, the reaction of **2** with benzaldehyde and formaldehyde gave the corresponding fulvene derivatives **3a** and **3b**, respectively (Scheme I).[7] With acetone and acetophenone, **2** gave complex mixtures. However, the reaction of **2** with benzophenone gave interesting results. Thus, addition of an equivalent amount of benzophenone (90 mg, 0.49 mmol) to a solution of **2** produced a benzophenone adduct **4a** as air- and moisture-sensitive yellow crystals. Pure **4a** appears to be thermally quite stable, with no change observed on heating at 90 °C for 2 h. The adduct **4a** also reacted with benzaldehyde to give **3a**.

NMR data of the adduct of **4a** are fully consistent with the proposed structure: ^{1}H NMR (C_7D_8, δ) 0.57 (d, J = 3.9 Hz, 30 H, $SiMe_2$), 5.09 (sept, J = 3.9 Hz, 5 H, SiH), 7.05 (t, J = 7.2 Hz, 4 H, *m*-H), 7.15 (t, J = 7.2 Hz, 2 H, *p*-H), 7.44 (d, J = 7.2 Hz, 4 H, *o*-H); ^{13}C NMR (C_7D_8, δ) 0.66, 128.8, 130.7, 133.9, 135.8 (CpC), 136.9, 200.9 (C=O), ^{29}Si NMR (C_7D_8, δ) -26.5. Of particular interest is the chemical shift of ^{7}Li appearing at -7.51 ppm. A large high-field shift of the ^{7}Li NMR resonance indicates a structure in which the Li^+ ion is located at the center of the cyclopentadienyl ring.[8] The low-field shift of the carbonyl carbon

(1) Chemistry of Organosilicon Compounds. 297.

(2) For a review, see: Jutzi, P. *J. Organomet. Chem.* **1990**, *400*, 1.

(3) Miftakhov, M. S.; Tolstikov, G. A.; Lomakina, S. I. *Zh. Obshch. Khim.* **1976**, *46*, 2754; *Chem. Abstr.* **1977**, *86*, 121430z. Miftakhov et al. reported the synthesis of $C_5(SiMe_3)_6$ by the reaction of hexachlorocyclopentadiene and trimethylchlorosilane with lithium in THF, but unfortunately we could not reproduce the results under various conditions ourselves.

(4) Reviews: (a) Sakurai, H. *J. Synth. Org. Chem. Jpn.* **1982**, *40*, 472. (b) Sakurai, H. *Nippon Kagaku Kaishi* (*J. Chem. Soc. Jpn., Chem. Ind.*) **1990**, 439. (c) Sekiguchi, A. *J. Synth. Org. Chem. Jpn.* **1992**, *50*, 225.

(5) Compound **1**: colorless crystals, mp 138 °C; ^{1}H NMR (C_7D_8, 263 K, δ) 0.01 (d, J = 3.8 Hz, 12 H), 0.41 (d, J = 3.8 Hz, 12 H), 0.49 (d, J = 3.8 Hz, 12 H), 4.74 (sept, J = 3.8 Hz, 2 H), 4.80-4.92 (m, 4 H); ^{13}C NMR (C_7D_8, 263 K, δ) -3.85, -1.01, -0.66, 75.2, 159.8, 163.9; ^{29}Si NMR (C_7D_8, 263 K, δ) -25.5, -25.1, -20.1; high-resolution MS calcd for $C_{17}H_{42}Si_6$ 414.1902, found 414.1896.

(6) Compound **2**: ^{1}H NMR (C_7D_8, δ) 0.56 (d, J = 3.7 Hz, 30 H, $SiMe_2$), 1.14-1.22 (m, 4 H, THF), 3.16-3.24 (m, 4 H, THF), 5.04 (sept, J = 3.7 Hz, 5 H, SiH); ^{13}C NMR (C_7D_8, δ) 0.61, 25.4 (THF), 69.0 (THF), 135.7; ^{29}Si NMR (C_7D_8, δ) -26.5; ^{7}Li NMR (C_7D_8, δ) -8.49.

(7) **3a** (94% yield, red-orange crystals) and **3b** (93% yield, yellow-orange crystals) were characterized by NMR and mass spectroscopic analyses. Details will be reported elsewhere.

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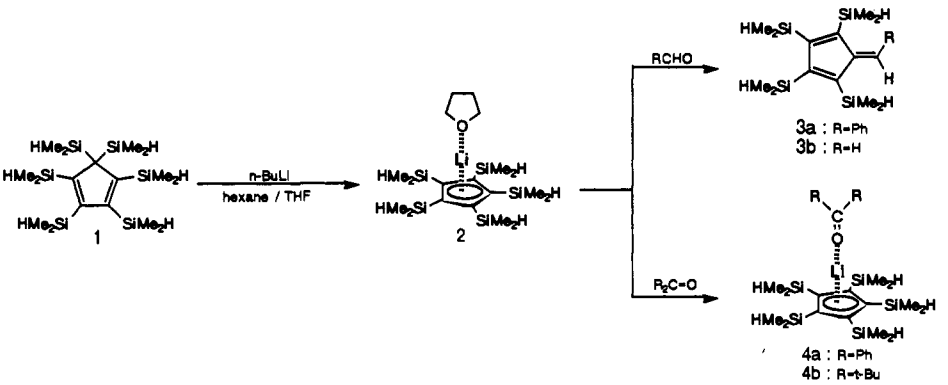
Scheme I



(200.9 ppm) compared to that of free benzophenone (195.6 ppm) is suggestive of coordination by the Li^+ ion. The ν(CO) of **4a** appeared at 1650.8 cm^{-1} (free benzophenone, 1670.1 cm^{-1}). The ORTEP drawing of **4a** determined by the X-ray diffraction method is shown in Figure 1.[9]

The Li^+ ion is coordinated by the oxygen of benzophenone as well as by the cyclopentadienide anion.[10] The cyclopentadienyl ring is planar, as shown by the internal bond angles of 107.3–108.7° (av 108.0°) and the sum of the angles (540.1°). The C—C distances of the ring, 1.417–1.435 Å (av 1.424 Å), are somewhat longer than those of [(12-crown-4)Li(C_5H_5)] (1.395 Å)[8j] but in the same range of magnitude found for [(THF)Li·{1,2,4-(Me_3Si)$_3C_5H_2$}] (av 1.411 Å).[8g,11] The lithium atom is found 1.818 Å above the center of the cyclopentadienyl ring. The distances between the lithium atom and the cyclopentadienyl carbons are approximately equal (av 2.186 Å). The distance between lithium and oxygen is 1.822 Å, and the Li—O═C angle is 163°. The bond distance of C═O (1.237 Å) is not stretched on coordination.[12]

The reaction of **2** with another hindered ketone, di-*tert*-butyl ketone, also led to the quantitative formation of an adduct **4b**: ^{1}H NMR (C_6D_6, δ) 0.61 (d, J = 4.0 Hz, 30 H, $SiMe_2$), 0.75 (s, 18 H, *t*-Bu), 5.12 (sept, J = 4.0 Hz, 5 H, SiH); ^{13}C NMR (C_6D_6, δ) 0.58, 28.0, 46.4, 135.8 (CpC), 229.9 (C═O); ^{29}Si NMR (C_6D_6, δ) −26.1; ^{7}Li NMR (C_6D_6, δ) −7.55. The carbonyl carbon was observed at 229.9 ppm (*t*-Bu_2CO: 218.8 ppm), and the ^{7}Li NMR resonance was found at −7.55 ppm. The free di-*tert*-butyl ketone showed the carbonyl frequency at 1689.4 cm^{-1}, whereas the adduct exhibited it at 1672.0 cm^{-1}.

On the basis of the molecular geometry of **4a**, it is obvious that the remarkable high-field shift of the ^{7}Li NMR resonance is caused by the strong shielding effect by the diatropic ring current resulting from the 6π-electron aromatic system of the cyclopentadienide anion.[13,14]

In the usual reactions of a lithium reagent with ketones, nucleophilic addition, otherwise reduction or electron-transfer reactions, takes place readily. Nucleophilic addition can be hindered by steric bulkiness. The electron-transfer reaction was also inhibited in the present case due to electronic reasons because the removal of one electron from the stable 6π-electron system leading to the cyclopentadienyl radical is unfavorable.[15] The formation

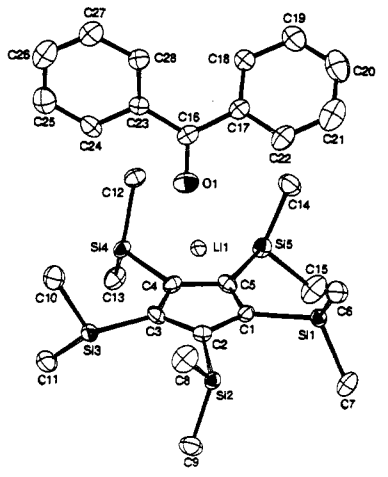


Figure 1. ORTEP drawing of **4a**. Selected bond lengths (Å): C1–C2 1.417(9), C1–C5 1.424(9), C2–C3 1.424(9), C3–C4 1.435(9), C4–C5 1.420(9), C1–Si1 1.875(7), C2–Si2 1.887(6), C3–Si3 1.879(7), C4–Si4 1.884(7), C5–Si5 1.885(7), O1–C16 1.237(9), O1–Li1 1.823(11). Selected bond angles (deg): C2–C1–C5 107.8(5), C2–C1–Si1 133.1(4), C5–C1–Si1 119.1(4), O1–C16–C17 119.2(6), O1–C16–C23 119.0(6), C17–C16–C23 121.8(6).

(8) For theoretical studies on cyclopentadienyllithium, see: (a) Alexandratos, S.; Streitwieser, A.; Schaefer, H. F., III. *J. Am. Chem. Soc.* **1976**, *98*, 7959. (b) Jemmis, E. D.; Schleyer, P. v. R. *J. Am. Chem. Soc.* **1982**, *104*, 4781. (c) Blom, R.; Faegri, K., Jr.; Midtgaard, T. *J. Am. Chem. Soc.* **1991**, *113*, 3230. For crystal structure of cyclopentadienyllithium derivatives, see; (d) Jutzi, P.; Schlüter, E.; Krüger, C.; Pohl, S. *Angew. Chem., Int. Ed. Engl.* **1983**, *22*, 994. (e) Lappert, M. F.; Singh, A.; Engelhardt, L. M.; White, A. H. *J. Organomet. Chem.* **1984**, *262*, 271. (f) Jutzi, P.; Schlüter, E.; Pohl, S.; Saak, W. *Chem. Ber.* **1985**, *118*, 1959. (g) Jutzi, P.; Leffers, W.; Pohl, S.; Saak, W. *Chem. Ber.* **1989**, *122*, 1449. (h) Jutzi, P. *Pure Appl. Chem.* **1989**, *61*, 1731. (i) Hammel, A.; Schwarz, W.; Weidlein, J. *Acta Crystallogr.* **1990**, *C46*, 2337. (j) Chen, H.; Jutzi, P.; Leffers, W.; Olmstead, M. M.; Power, P. P. *Organometallics* **1991**, *10*, 1282.

(9) A single crystal (0.5 × 0.3 × 0.2 mm) of **4a** was sealed in a capillary glass tube for data collection. Diffraction data were collected at 170 K on a Rigaku Denki AFC-5R diffractometer with a rotating anode (45 kV, 200 mA) with graphite-monochromated Mo Kα radiation (λ = 0.71069 Å). A total of 7360 reflections with 2θ = 3–52° were collected. Crystal data: MF = $Si_5C_{28}H_{45}OLi$, MW = 545.0; monoclinic; a = 9.157(4), b = 19.188(7), c = 19.497(3) Å, β = 98.04(2)°, V = 3392.0(21) Å^3, space group $P2_1/c$; Z = 4; D_c = 1.067 g/cm^3. The dimethylsilyl groups are arranged around the five-membered ring in a gear-meshed form, and the opposite arrangement results in a crystallographic orientational disorder. The structure was refined by splitting the population of the orientation (0.8/0.2). The final R factor was 0.0749 (R_w = 0.0909) for 4240 reflections with $F_o > 3\sigma(F_o)$.

(10) Two Si methyls should be magnetically nonequivalent. However, ^{1}H NMR of **4a** and **4b** at low temperature (298–178 K) in toluene-d_8 showed only a broadened signal for the Si methyl protons, and thus the rotational barriers could not be determined. For a sample of [(HMPA)^{6}Li·C_5($SiMe_2H$)$_5$], the coupling between ^{6}Li and ^{31}P was observable at 210 K [^{6}Li NMR δ = −7.93 (d, $^2J_{Li-P}$ = 6 Hz); ^{31}P NMR δ = 24.9 (t, $^2J_{Li-P}$ = 6 Hz) ppm], but the Si methyl protons also remained broadened.

(11) Owing to steric reasons, the distances of Si–C(ring) (av 1.882 Å) are somewhat stretched compared to other silyl-substituted cyclopentadienyllithiums (1.825–1.856 Å).

(12) The distance of C═O for benzophenone was reported to be 1.23 Å by X-ray diffraction. Fleischer, E. B.; Sung, N.; Hawkinson, S. *J. Phys. Chem.* **1968**, *72*, 4311.

(13) Treatment of **1** with *n*-BuLi in the presence of a base such as DME, quinuclidine, tetramethylethylenediamine (TMEDA), and 1,4-diazabicyclo-[2.2.2]octane (DABCO) also afforded the corresponding complexes, [(base)Li·C_5($SiMe_2H$)$_5$]. The ^{7}Li NMR chemical shifts of these complexes are as follows: −8.24 (DME), −8.54 (quinuclidine), −7.70 (TMEDA), and −8.69 (DABCO) ppm. These are less shielded than the corresponding values of ^{7}Li NMR chemical shifts of [(base)Li·1,2,4-(Me_3Si)$_3C_5H_2$] (−8.2 to −12.5 ppm)[8g] and [(base)Li·1,3-(Me_3Si)$_3C_5H_3$] (−10.1 to −10.8 ppm).[8g]

(14) A large deshielding effect on ^{7}Li NMR for bis[(dimethoxyethane)-lithium(1)] 1,2,4,5-tetrakis(trimethylsilyl)benzenide as a 6C-8π antiaromatic system has been reported recently. Sekiguchi, A.; Ebata, K.; Kabuto, C.; Sakurai, H. *J. Am. Chem. Soc.* **1991**, *113*, 7081.

of a complex between the lithium reagent and ketone, through which further reactions should take place, was the only fate of the present reactions. The X-ray and NMR studies evidently provide the first structural characterization of the intermediate adduct of the lithium reagent and ketones.[16]

Acknowledgment. We are grateful for the financial support of the Ministry of Education, Science, and Culture of Japan (Specially Promoted Research No. 02102004) and for a Kurata Research Grant.

Supplementary Material Available: Tables of X-ray experimental data, atomic parameters, anisotropic temperature factors, bond distances, and bond angles (10 pages); a table of observed and calculated structure factors for **4a** (21 pages). Ordering information is given on any current masthead page.

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(16) Very recently, stable complexes between esters and lithium hexamethyldisilazide were characterized by X-ray diffraction; see: Williard, P. G.; Liu, Q.-Y.; Lochmann, L. *J. Am. Chem. Soc.* **1992**, *114*, 348. For $(Ph_2P)_2CHLi\cdot Ph_2CO$, see: Issleib, V. K.; Abicht, H. P. *J. Prakt. Chem.* **1970**, *B312*, 456.

Polymerized Liposomes Containing *C*-Glycosides of Sialic Acid: Potent Inhibitors of Influenza Virus in Vitro Infectivity

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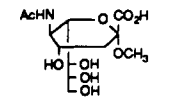
The surface lectin of the influenza virus, hemagglutinin, binds to terminal α-glycosides of *N*-acetylneuraminic acid (NeuAc) on cell-surface glycoproteins and glycolipids.[1] Viral binding to cells expressing terminal NeuAc residues can be inhibited by α-*O*-glycosides of NeuAc (*O*-sialosides).[2-5] Recently, dramatic enhancements in the inhibition of viral adhesion to erythrocytes have been achieved using synthetic polyvalent sialosides.[6-9] The inhibitory potencies of these polyvalent materials approach those of the most potent naturally occurring hemagglutination inhibitors, equine and guinea pig α_2-macroglobulins.[5,6] Despite intensive efforts in designing polyvalent sialosides to inhibit hemagglutination, no evidence exists that these synthetic *O*-sialoside materials can be used to arrest viral infectivity.[9] In this communication, we report that polymerized liposomes containing α-*C*-glycosides of sialic acid are potent inhibitors of influenza virus in vitro infectivity. Our results also indicate that the capacity to inhibit hemagglutination does not necessarily reflect the capacity to inhibit in vitro infectivity.

Table I. Hemagglutination Inhibition (HAI) and Plaque Reduction Assays of Liposome Preparations I-VI

entry	inhibitor	HAI[a] [3], M	plaque reduction [3], mM	plaque reduction, reduction,[b] %
1	liposome I (0%, **3**)	0 (-)	0.000	0
2	liposome II (1%, **3**)	4.0×10^{-6} (-)	0.003	96
3	liposome III (5%, **3**)	5.7×10^{-7} (+)	0.016	97
4	liposome IV (10%, **3**)	3.3×10^{-7} (+)	0.030	46
5	liposome V (30%, **3**)	8.0×10^{-5} (-)	3.750	0
6	liposome VI (60%, **3**)	1.5×10^{-4} (-)	7.500	0

[a] A (+) indicates complete inhibition while a (-) indicates that no inhibition was observed at the given concentrations of **3**. [b] The values represent the percent reduction in the number of plaques per well due to viral lysis of infected cells.



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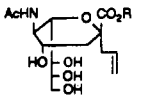
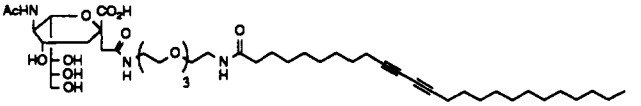
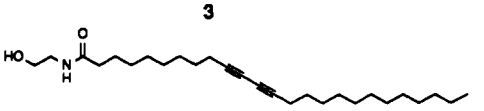


2a R=Et
2b R=H



3



4

Sialoside lipid **3** was synthesized from **2a**,[10] and mixed liposomes[11] composed of compounds **3** and **4** were prepared using a modified probe sonication method.[12] The liposome preparations

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[§] Glycomed, Inc.

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