Another phase of our collaborative venture simultaneously led to optimized chromatographic purifications of C_{60} and higher fullerenes.[12] In the course of these studies, an anomaly in the UV-vis spectrum of the C_{60} fraction led to the detection and isolation of the same oxide $C_{60}O$. The elemental composition was revealed by thermospray mass spectrometry.[13] The spectrum of a slightly impure sample appears in Figure 1a. The most intense peak, at 736 amu, corresponds to $C_{60}O$; the only other strong peak, at 720 amu, derives from C_{60}.[14]

The UV-vis absorption spectra for C_{60} and $C_{60}O$ in toluene are quite similar except for subtle differences in the 400-700-nm region (Figure 1b). $C_{60}O$ exhibits a new band at 424 nm but lacks the C_{60} band at 408 nm.[15] Relative to C_{60}, $C_{60}O$ displays stronger absorption at 496 nm and weaker absorptions at 540 and 600 nm.

To establish a vibrational fingerprint of the new material, FTIR spectra of triply chromatographed $C_{60}O$ were recorded with 0.5-cm^{-1} resolution (Figure 1c). The spectra contain no detectable absorptions above 1600 cm^{-1}, consistent with the absence of C—H or C=O bonds. Seventeen relatively strong bands and 25 weaker ones are observed between 450 and 1600 cm^{-1}. Four of the stronger bands (1427.8, 1184.6, 575.4, and 526.0 cm^{-1}) resemble the principal absorptions of C_{60} (1429.0, 1182.7, 575.9, and 526.9 cm^{-1} as recorded on the same instrument).[16]

The ^{13}C NMR spectrum of $C_{60}O$ (Figure 1d) was acquired at 125 MHz in benzene-d_6 with $Cr(acac)_3$ added as a relaxant. Sixteen lines are resolved, one at 90.18 ppm and the remainder between 140 and 146 ppm, referenced to the central peak of the benzene triplet (128 ppm).[17] The chemical shifts are consistent with the values reported for C_{60} (142.68[18a] or 143.2[18b] ppm), C_{70} (130-151 ppm),[12,18a,b] and C_{76} (129-150 ppm).[18c]

The ^{13}C NMR, FTIR, and UV-vis spectra of $C_{60}O$ contain a number of unique features, but also suggest that this new fullerene retains the essential electronic and structural character of C_{60}. The epoxide structure **1**, of C_{2v} symmetry, would derive from oxidation of one of the 30 equivalent C_{60} double bonds.[3,4] Oxidoannulene **2**, analogous to the structure proposed for $C_{70}O$,[4] could arise via isomerization of **1**. Pioneering studies of the parent oxidoannulene and related species by Vogel[19a] suggest that **2** should contain a delocalized annulene moiety[19b] and thus should also embody C_{2v} symmetry.

Both **1** and **2** contain 17 sets of inequivalent carbons: 13 groups of four carbons each and four comprising two carbons each. The relative intensities in the ^{13}C NMR spectrum of $C_{60}O$ follow the predicted pattern. The chemical shift of the two-carbon signal at 90.18 ppm is fully consistent with expectations for the epoxide carbons in **1**.[20] Although a priori the vinyl ether β carbons of **2** might also be expected to resonate near 90 ppm, **2** contains four such carbons rather than two. In contrast, the ^{13}C NMR spectrum of the parent 1,6-oxido[10]annulene comprises three lines between 124 and 131 ppm.[21] Thus, the room temperature NMR data cannot be reconciled with oxidoannulene **2**, but strongly support the isomeric epoxide structure **1**.

Finally, we have demonstrated that $C_{60}O$ is efficiently converted to C_{60} (ca. 91% yield) during chromatography on neutral alumina. The widespread use of alumina for purification of the fullerenes may explain why $C_{60}O$ has not been isolated previously.

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Supplementary Material Available: Detailed procedures for the preparation of $C_{60}O$ and tables of IR and NMR data (2 pages). Ordering information is given on any current masthead page.

(10) Heating C_{60} to 350 °C in air (10 °C/min) resulted in a 2% weight increase, consistent with the formation of $C_{60}O$. However, the product was insoluble in toluene and $C_{60}O$ was not detected by HPLC analysis. Unpublished results of Dr. Andrew R. McGhie, University of Pennsylvania. See also ref 5.

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(13) Thermospray mass spectra were measured by Dr. Robert T. Rosen, Food Sciences Department, Rutgers University, New Brunswick, NJ, using a Vestec 201 LC-MS instrument operated in the negative ion discharge mode with benzene as eluant. The benzene solution was transferred directly into the heated (200 °C) capillary for analysis.

(14) A significant fraction of the C_{60} ion signal likely results from thermal decomposition of $C_{60}O$. Thermal desorption mass spectrometry ($T \geq 300$ °C) of similar $C_{60}O$ samples shows C_{60} (720 amu) signals markedly stronger than those of $C_{60}O$ (736 amu); ca. 2.5 and 100 times larger for TD-FAB-MS and TD-CI-MS, respectively.

(15) Bathochromatic shifts (2-10 nm) and changes in relative absorption in the optical spectra of fullerenes are observed in different aromatic solvents. Unpublished results of K. Creegan.

(16) Thermal decomposition of $C_{60}O$ in air at 175 °C as monitored by IR absorption furnished an as-yet-unidentified product (not C_{60}). Unpublished results of Dr. John Robbins.

(17) (a) ^{13}C chemical shift values (and relative intensities) for $C_{60}O$ in benzene-d_6: 145.47 (3.6), 145.41 (3.6), 145.34 (3.6), 145.20 (1.7), 144.54 (4.1), 144.54 (4.2), 144.16 (8.5),[17b] 143.78 (2.2), 143.27 (4.1), 143.25 (4.1), 142.70 (2.4), 142.56 (4.0), 142.41 (4.0), 141.18 (4.0), 141.00 (3.6), 90.18 ppm (2.0). (b) Resolves into two resonances in CS_2.

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(21) The spectrum, measured in $CDCl_3$ containing 0.02 M $Cr(acac)_3$ as relaxant, consists of resonances at 123.60 (4), 128.11 (4), and 131.37 (2) ppm.

Accommodation of Polar Guests in Unimolecular Polyamine–Polyhydroxy Cores: Solubilization of Sugars in Apolar Organic Media via Intramolecular Polar Microsolvation[1]

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Highly polar compounds as guests can be solubilized in apolar organic media upon selective complexation with *rigid* hosts having preorganized binding sites.[3] A three-dimensional encapsulation

(1) Molecular Recognition. 19. Part 18: Kikuchi, Y.; Kobayashi, K.; Aoyama, Y. *J. Am. Chem. Soc.*, in press.

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(3) For examples of the extraction of amino acids, nucleotides, vitamins, and sugars, see: (a) Tabushi, I.; Imuta, J.; Seko, N.; Kobuke, Y. *J. Am. Chem. Soc.* **1978**, *100*, 6287-6288. (b) Rebek, J., Jr.; Askew, B.; Nemeth, D.; Parris, K. *Ibid.* **1987**, *109*, 2432-2434. (c) Aoyama, Y.; Tanaka, Y.; Toi, H.; Ogoshi, H. *Ibid.* **1988**, *110*, 634-635. (d) Aoyama, Y.; Tanaka, Y.; Sugahara, S. *Ibid.* **1989**, *111*, 5397-5404. (e) Aoyama, Y.; Asakawa, M.; Yamagishi, A.; Toi, H.; Ogoshi, H. *Ibid.* **1990**, *112*, 3145-3151.

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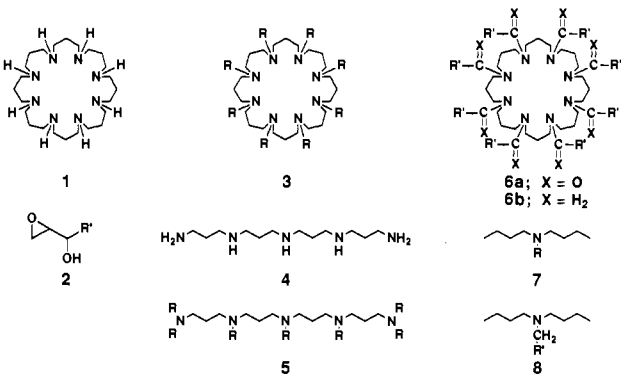
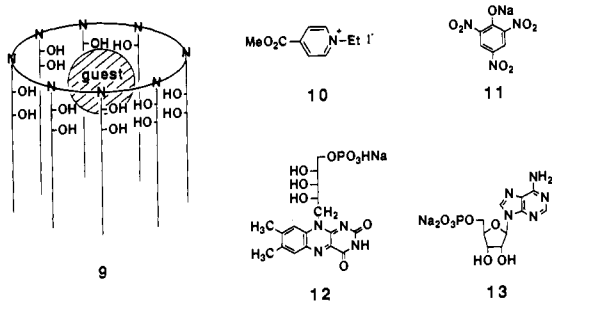
Chart I[a]



[a] R = $CH_2CH(OH)CH(OH)(CH_2)_{10}CH_3$, R′ = $(CH_2)_{10}CH_3$.

Chart II



of a guest may be achieved by modeling solvation shells for polar solutes in water. In the present work, we have prepared covalently-linked polyamine–polyhydroxy clusters along this line. We wish to report here that sugars and other polar guests can be solubilized in CCl_4 via what may be called *flexible* intramolecular polar microsolvation.[4]

Alkylation of octaaza macrocycle **1**[5] with long-chain epoxy alcohol **2** in DMF at 140 °C for 6–8 h afforded the octakis(dihydroxyalkyl) derivative **3** (60%).[6,7] A similar reaction of linear pentaamine **4**[5] with **2** gave the noncyclic heptakis(dihydroxyalkyl) derivative **5** (32%).[7] On the other hand, reaction of macrocycle **1** with dodecanoyl chloride in CH_2Cl_2, followed by reduction of the resulting octaamide **6a** with diborane in THF, gave the cyclic octaalkyl derivative **6b** (40% total) having no OH groups (Chart I).

Vigorous stirring for 24 h of a two-phase mixture of a CCl_4 solution of host **3** (1.0×10^{-2} M, 2 mL) and water (2 mL) resulted in the transfer of ~40 molecules (by ^{1}H NMR integration) of the latter into the former solution.[8] The resulting supramolecular complex **3**·$n$$H_2O$ ($n \approx 40$) was found to be nearly monomeric as such[8] and exhibited a large (by ~1 ppm) downfield shift (from δ 2.1–2.7 in free **3** to 2.7–3.8 in the complex) of the NCH_2 resonances. These results indicate that the polar core of host **3** composed of 16 OH groups and 8 tertiary amino groups can accommodate a water pool (structure **9** in a schematic representation; guest is ~40H_2O) with an amine-protonation equilibrium (amine + $H_2O \rightleftharpoons$ ammonium + OH^-)[9] (Chart II).

D-Glucose as well as D-fructose and D-ribose (3 M in water) could be extracted into CCl_4 containing host **3**. The number of coextracted water molecules, if any, was unmeasurably small in this case.[10] The stoichiometry **3**/glucose ≈ 1 was established directly by ^{1}H NMR integration[10] or after reextraction of the sugar back into water. Sugar extraction as well as water-pool accommodation was also observed with the noncyclic heptakis(dihydroxyalkyl) reference host **5**, but *never* with other references such as **6b**, **7**, and **8**. Thus, the presence of clustering dihydroxyalkyl chains is essential for the present sugar extraction. This suggests that the sugar, possibly with a limited number of coextracted water molecules, is encapsulated in the polar core as shown in structure **9** (guest is sugar).

Pyridinium iodide **10** is a probe for solvent polarity.[11] Guest **10** as a solid, otherwise insoluble in CCl_4, could be solubilized with host **3** in that solvent under anhydrous conditions, giving λ_{max} 350 nm. This λ_{max}, in light of the correlation between λ_{max} and solvent polarity,[11] indicates that the micropolarity of the **10**-binding polar core of **3** (structure **9**; guest is **10**) corresponds to ethanol (λ_{max} 359 nm) or formamide (λ_{max} 343 nm).

The extraction of organic anions took place readily.[12] From an aqueous solution (pH ≤ 12) of sodium picrate (**11**, Na^+Pic^-, 0.005–0.1 M) was instantaneously extracted Pic^-, with little pH dependence, into a CCl_4 solution of host **3** (0.01 M) upon formation of a monomeric 1:1 ammonium picrate salt (structure **9** with one nitrogen atom being protonated as a countercation; guest is Pic^-).[13,14] The extraction of nucleotide anions of FMN (**12**) and AMP (**13**) took place similarly.[15] Simple anions such as carbonate in water (0.01 M) could also be extracted.[16] Competitive extraction indicated the decreasing extractabilities in the following order: picrate (lipophilic anion) > carbonate (hydrophilic anion) > sugar (hydrophilic neutral species).

To summarize, compound **3** exhibits such features (monomeric nature and definite stoichiometry of binding) as are characteristic of a *host*. Most current hosts are designed so as to exhibit *selectivity*, a general strategy for which is the preorganization of limited but multiple and convergent binding sites using *rigid* skeletons.[17] The binding site or the polar core of host **3**, on the other hand, is an intramolecularly associated polyamine/ammonium–polyhydroxy cluster with a limited amount of H_2O. In this respect, host **3** can also be regarded as a unimolecular reversed micelle.[18] The polar core is adjustable to and hence incorporates

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(6) Compound **3** was purified by means of chromatography on silica gel (CH_3OH) and on Sephadex LH-20 (CH_3OH), followed by preparative HPLC on a column of TSK GEL G2000 HXL (THF): ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 24 H, CH_3), 1.0–1.7 (m, 176 H, CCH_2C), 2.1–2.7 (m, 48 H, NCH_2), 3.2–3.7 (m, 16 H, C*H*OH), 3.6–4.8 (br, 16 H, OH; disappeared on deuteriation); IR (CCl_4) 3350 cm^{-1} (ν_{OH}). Molecular weight by VPO for a $CHCl_3$ solution was 3.2×10^3 at infinity dilution (calcd 2280), indicative of some aggregation of compound **3**.

(7) Compounds **3** and **5**–**8** as well as acetylated **3** (prepared for an identification purpose) gave satisfactory spectral data.

(8) Water complex: δ ($CDCl_3$–CCl_4) 1.9 (bound H_2O; absent when D_2O was used); molecular weight (VPO, $CHCl_3$) 3.4×10^3 (calcd for 3·40H_2O 3000).

(9) For the pK_a values of compound **1** in water, see ref 5a.

(10) The ^{1}H NMR spectrum of the glucose complex in $CDCl_3$–CCl_4 indicates that glucose is bound as a 1:2 mixture of α- (δ_{1-H} 5.20) and β-pyranose (δ_{1-H} 4.60). That little coextraction of water had taken place came from the comparison of the ^{1}H NMR spectra of the complexes derived from glucose in H_2O and in D_2O.

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(12) The extraction of picrate and FMN anions was not specific to hosts **3** and **5** but also took place, although less effectively, with references **6b**, **7**, and **8**.

(13) The picrate complex in $CDCl_3$ or CCl_4 showed a molecular weight by VPO of 2.5×10^3 (calcd for ($3H^+$)Pic^- 2531), λ_{max} 344 nm (ε 19 210), and δ_H 8.73 as a sharp singlet for the Pic^- moiety. The ^{1}H NMR spectra also showed partial protonation of the nitrogen atoms (NCH_2 resonances being ~0.3 ppm downfield shifted) and possible involvement of ~5 molecules of coextracted H_2O.

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various monosaccharides and stabilizes various ammonium–anion salts by the induced-fit mechanism or what may be called *flexible* intramolecular polar microsolvation, in a similar manner as solvent water dissolves various polar solutes. This may also be why noncyclic host **5** works fairly well too. Thus, *versatility* is an important aspect here.[19]

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Registry No. 1, 63681-43-6; **2**, 138093-67-1; **3**, 138093-68-2; **4**, 13274-42-5; **5**, 138093-69-3; **6a**, 138093-70-6; **6b**, 138093-71-7; **10**, 1199-65-1; **11**, 3324-58-1; **12**, 130-40-5; **13**, 4578-31-8.

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Molecular Recognition in Aqueous Micellar Solution: Adenine–Thymine Base-Pairing in SDS Micelles

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Hydrogen bonding is a fundamental force in molecular recognition by biological macromolecules. It is central to nucleic acid base-pairing, yet does not occur significantly between individual nucleotides or nucleic acid bases in aqueous solution.[1] Model systems generally require noncompetitive organic solvents, such as $CDCl_3$, to achieve hydrogen bonding between uncharged receptors and substrates.[2,3] Here, we report that self-assembling molecular receptors, comprising (thyminyloctyl)ammonium groups in sodium dodecyl sulfate (SDS) micelles, bind adenine derivatives by means of hydrogen bonding in aqueous solution.[4]

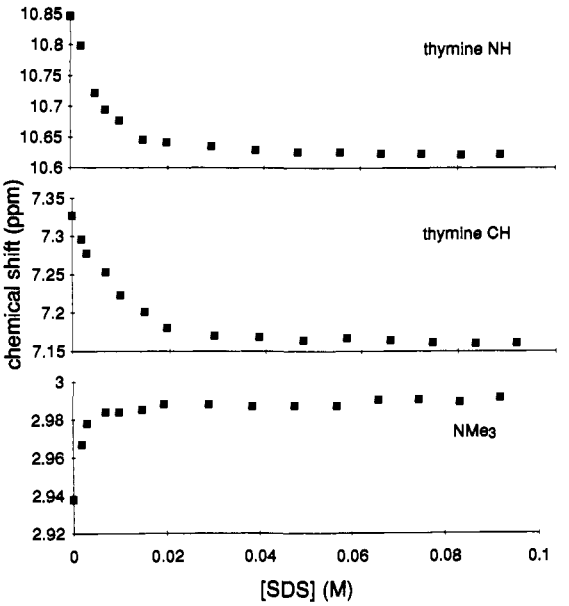
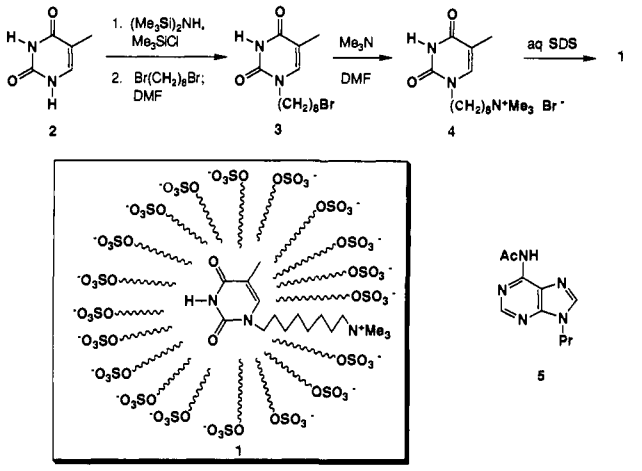


Figure 1. Effect of SDS concentration on chemical shift of protons of thymine **4**. Titrations were performed on a 300-MHz NMR instrument at 22 ± 1 °C by addition of 1 M SDS solution to a 1.0 mM solution of **4** in D_2O (CH protons) or 10% H_2O/D_2O (NH proton, 1.0 mM HOAc added). HOD or H_2O was used as a reference (δ 4.65).

Scheme I



The receptors (represented by structure **1**) were prepared from thymine as shown in Scheme I.[3b,5] ^1H NMR studies indicate that ammonium salt **4**, which is complementary in charge and structure to SDS, readily incorporates in SDS micelles (Figure 1). Increasing the SDS concentration from 0 to 20 mM results in large changes in the spectrum of **4**, suggesting that the environment of **4** changes drastically as the SDS forms micelles (CMC = 8.2 mM).[6] Incorporation is complete above 20 mM SDS. On the

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