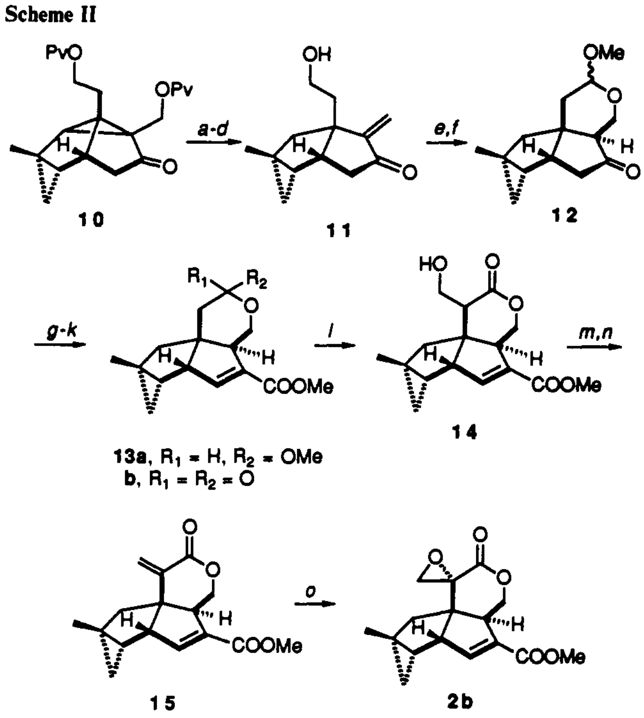
Scheme II



[a]NaOH, EtOH, H_2O, room temperature. [b]Li, NH_3. [c]Ac_2O, Et_3N, DMAP, CH_2Cl_2, -20 °C. [d]Na_2CO_3, MeOH, H_2O, room temperature. [e]Swern oxidation. [f]NaOMe, MeOH, room temperature. [g]LDA, THF, -78 °C; $PhNTf_2$, -78 °C → 0 °C. [h]$Pd(OAc)_2$, Ph_3P, Et_3N, MeOH, DMF, CO atmosphere, room temperature. [i]CH_2N_2. [j]HCl, THF, room temperature. [k]$(n\text{-}Pr)_4NRuO_4$ (TPAP), NMO, 4-Å sieves, CH_2Cl_2, room temperature. [l]LDA, THF, -78 °C; CH_2O. [m]CH_3SO_2Cl, Et_3N, CH_2Cl_2. [n]DBU, C_6H_6, room temperature. [o]MCPBA, CH_2Cl_2, reflux.

made available alcohol **8b** (70%).

Perruthenate oxidation[8] of **8b** delivered the pivotal tricyclic β,γ-unsaturated ketone **9** (97%). At this point, reliance was placed on the oxadi-π-methane rearrangement[11] for establishing a large segment of the structural network of **2**. Indeed, irradiation of **9** in acetone with 3000-Å light proceeded without event to introduce a second three-membered ring as in **10** (91%). Any ambiguity in structure was removed by X-ray analysis[12] of the highly crystalline diol produced by saponification of **10**.

Stereoelectronic factors present in **10** allowed for fully regiocontrolled cleavage of a single cyclopropane bond under dissolving metal conditions (Scheme II). The resultant dihydro diol and **11** (1:2) were separated; the diol was acetylated and treated with Na_2CO_3 in aqueous MeOH to give additional **11** (62% overall). The latter was oxidized to the keto aldehyde and exposed directly to methoxide ion. Intramolecular Michael addition of the hemiacetal anion[13] delivered **12** in 62% yield.

Proper attachment of the carbomethoxy group was realized by conversion of **12** to its enol triflate,[14] $Pd(OAc)_2$-catalyzed carbonylation,[15] and esterification with CH_2N_2 (66% of **13a**). Once **13b** was in hand (88%), several probe experiments indicated that introduction of the remaining carbon atom would be difficult. Recourse to monomeric formaldehyde dissolved in THF[16] as electrophile did, however, result in efficient conversion to **14** (86%, 10:1 α/β). The synthesis of **2b** was completed by formation of methylene lactone **15** (93%)[17] and peracid oxidation (18%). Modest improvement in yield was seen when the three-step sequence[17] involving Dibal-H/t-BuOOH, $VO(acac)_2$/TPAP, and NMO was utilized instead. The synthetic product proved identical in all respects with an authentic sample.[18]

Acknowledgment. We gratefully acknowledge the National Institutes of Health (Grant GM-28468) for support of this work.

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Chlorination and Bromination of Fullerenes. Nucleophilic Methoxylation of Polychlorofullerenes and Their Aluminum Trichloride Catalyzed Friedel–Crafts Reaction with Aromatics to Polyarylfullerenes[1a]

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Since the development of a practical synthesis of buckminsterfullerene, C_{60}, and related fullerene C_{70}, by resistive evaporation of graphite in early 1990,[2] their chemistry has seen an explosive growth. The majority of the work pertains to their redox behavior.[3-10] Recently, we were able to quench fullerene diamagnetic polyanions (probably hexaanions) with methyl iodide to polymethylated fullerenes.[7a] Poly(trimethyl)silylation was also achieved, albeit in poor yield.[7a] Wudl has achieved alkylation and arylation of fullerene,[7b] and Wood et al. reported sequential

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(1) (a) Considered Polycarbon Supercage Chemistry. 2. Part 1: Reference 7a. (b) Department of Chemistry and Biochemistry, University of California, Los Angeles, CA 90024-1569.

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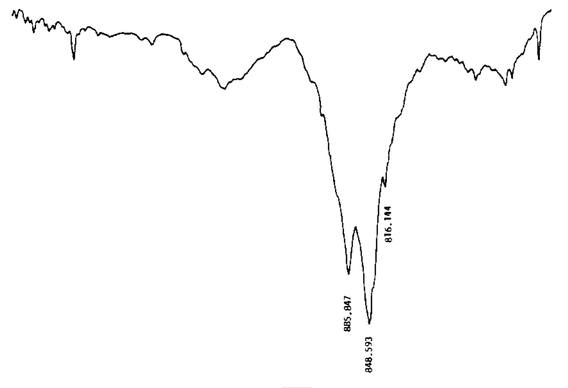

Figure 1. FT-IR spectrum of a polychlorinated C_{60}/C_{70} mixture.

methylene addition.[7c] Several transition-metal complexes of C_{60} have also been isolated.[11,12] Selig et al.[13] and Holloway and coworkers[14] have reported partial as well as complete fluorination of C_{60}. In continuation of our studies, we now report chlorination and bromination of fullerenes and some remarkable reactions of halofullerenes.

Treatment of a C_{60}/C_{70} mixture or pure C_{60}[15] with chlorine in chloro organic solvents at various temperatures did not result in any detectable reaction. However, in a hot glass tube under a slow stream of chlorine gas, both the C_{60}/C_{70} mixture and pure C_{60} reacted readily. At 250 °C, 50 mg of C_{60}/C_{70} was chlorinated (chlorine flow rate 10 mL/min) and maximum uptake of chlorine was achieved within 5 h. The weight increase of the dark brown product indicated that on average 24 chlorine atoms (also confirmed by elemental analysis[16a]) were added onto the skeleton. At higher temperatures (300, 350, and 400 °C), the chlorination occurred more rapidly. The highly chlorinated fullerene is light orange in color and soluble in many organic solvents. Under these conditions, complete chlorination of C_{60} was not achieved.

$$C_{60} \xrightarrow[\Delta]{Cl_2(g)} C_{60}Cl_n$$

The IR spectrum (Figure 1) shows a strong C–Cl stretch and is distinctly different from the simple IR spectrum of the C_{60}/C_{70} mixture. The 75-MHz ^{13}C solution NMR spectrum did not show any discernible peak even after 48 h of time averaging (due to chemical shift anisotropy and long carbon relaxation times). The 22.49-MHz solid-state ^{13}C NMR spectrum, however, showed broad absorptions at 50–150 ppm. Efforts to obtain mass spectra of polychlorinated fullerenes by FAB[16b] (fast atom bombardment) or FIMS (field-ionization mass spectrometry) were unsuccessful. Even under these mild ionization conditions, polychlorofullerenes lose chlorine (only peaks due to the C_{60} (M^+, 720) and C_{70} (M^+, 840) were observed). Indeed, polychlorofullerenes at 400 °C under argon completely dechlorinate to the parent fullerene mixture. The nature of polychlorinated C_{70} could not be ascertained since the percentage of C_{70} in the fullerene mixture is small ($\leq$10%).

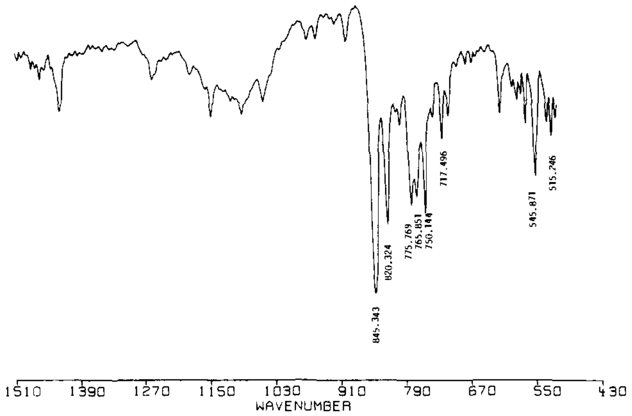

Figure 2. FT-IR spectrum of a polybrominated C_{60}/C_{70} mixture.

The polychlorofullerene mixture was treated with excess methanol/KOH[17] under reflux for 3 h. Quenching by aqueous ammonium chloride and workup provided a brownish material. The ^1H NMR spectrum ($CDCl_3$) indicated a broad envelop of methoxy groups centered at $\delta(^1H)$ 3.7. The FT-IR spectrum also shows a C–O stretching frequency at 1091 cm^{-1}. Evidence for the replacement of all the chlorine atoms by methoxy groups came from FAB mass spectrometry (polymethoxylation up to 26 methoxy groups, M^+, 1526). Further, sequential loss of methoxy groups was observed, indicating fragmentation from $C_{60}(OMe)_{26}$ all the way to $C_{60}(OMe)$. Mass spectrometric evidence was also obtained for some hydroxylation. The C_{60}/C_{70} mixture itself does not react with methanol/KOH. Observation of a product component with 26 methoxy groups implies that more than 24 chlorine atoms may have added to the fullerene skeleton. The replacement of chlorine by methoxy groups can be rationalized by a unique front-side substitution. Polychlorinated pure C_{60} behaved similarly.

$$C_{60}Cl_n \xrightarrow[\text{reflux}]{\text{MeOH, KOH}} C_{60}(OMe)_n$$

The polychlorinated fullerenes also undergo Friedel–Crafts reaction. Stirring a benzene solution of $C_{60}Cl_n$ with a catalytic amount of aluminum chloride at ambient temperature for 2 h gave a dark reddish-brown solution, which upon aqueous workup yielded a brown solid (soluble in organic solvents). The ^1H NMR spectrum ($CDCl_3$) showed a broad absorption centered at $\delta(^1H)$ 7.2. The ^{13}C NMR spectrum also showed a broad absorption, at $\delta(^{13}C)$ 128, indicating phenylation. The FAB mass spectrum indicates substitution by at least 22 phenyl groups (M^+, 2414) with no residual chlorine remaining in the product (by IR analysis). Further, consecutive loss of phenyl groups, demonstrating the fragmentation from $C_{60}(C_6H_5)_{22}$ to $C_{60}C_6H_5$, was observed. Polytolylation with toluene occurred similarly.[18]

$$C_{60}Cl_n \xrightarrow[AlCl_3]{C_6H_6} C_{60}(Ph)_n$$

Bromination of the C_{60}/C_{70} mixture or pure C_{60} in neat bromine at 20 °C and 50 °C gave evidence for the uptake of two to four bromine atoms (based on the weight increase). The FT-IR spectrum (Figure 2) shows a C–Br stretch at 515–545 cm^{-1}. Upon heating to 150 °C, the brominated fullerenes lose bromine readily.

$$C_{60} \xrightarrow[\text{room temp or 50 °C}]{Br_2\text{ (neat)}} C_{60}Br_n \qquad n = 2 \text{ or } 4$$

The solution ^{13}C NMR spectrum again did not indicate discernible absorptions due to lack of symmetry. However, the solid-state ^{13}C NMR spectrum showed broad peaks between +30 and +150 ppm. The FAB mass spectrum shows a loss of bromine, giving mass peaks only due to C_{60}. Brominated C_{60} also undergoes

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(15) The C_{60}/C_{70} mixture was prepared in house by a gravity-fed multiple graphite arcing technique (the details will be published elsewhere). Pure C_{60} was separated by published methods.

(16) (a) Elemental analysis was performed by Galbraith Laboratories, Knoxville, TN. (b) We thank Professor A. R. Battersby of the University of Cambridge for his initial help in obtaining the FAB spectrum. The FAB measurements reported were carried out on a VG Analytical Ltd. ZAB-SE high-resolution reverse-geometry mass spectrometer.

(17) Excess methanol with dry KOH was used. The reaction also works with NaOMe/MeOH.

(18) Proton and carbon NMR spectra and IR analysis clearly showed polytolylation (mostly para substitution).

Friedel–Crafts type fullerenation of benzene catalyzed by aluminum trichloride (or related catalysts). In addition to phenyl substitution, benzene (C_6H_6) addition (i.e., C_6H_5 and H) was also observed (see accompanying communication[19]).

In conclusion, we have found that fullerenes readily undergo chlorination and bromination. The halogenated fullerenes can be thermally dehalogenated. Polychlorinated fullerenes undergo nucleophilic methoxylation and Friedel–Crafts type reactions with benzene and toluene. Further studies are underway for selective functionalization of fullerenes.

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Polyarenefullerenes, $C_{60}(H-Ar)_n$, Obtained by Acid-Catalyzed Fullerenation of Aromatics[1a]

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Compared to the redox chemistry,[2-9] the functionalization chemistry[10] of fullerenes has received relatively little attention.[11-15] We have reported the polymethylation as well as trimethylsilylation of diamagnetic fullerene anions.[6] Reports on hydrogenation, oxygenation, metalation, methylenation, and fluorination have also appeared.[2,11-15] In the preceding communication[16a] we reported polychlorination and polybromination of fullerene C_{60} and C_{60}/C_{70} mixtures. Polychlorofullerenes undergo facile nucleophilic polymethoxylation. Furthermore, we found that polychlorinated C_{60} undergoes remarkable polyphenylation to polyphenylfullerene with benzene and aluminum trichloride under typical Friedel–Crafts reaction conditions.

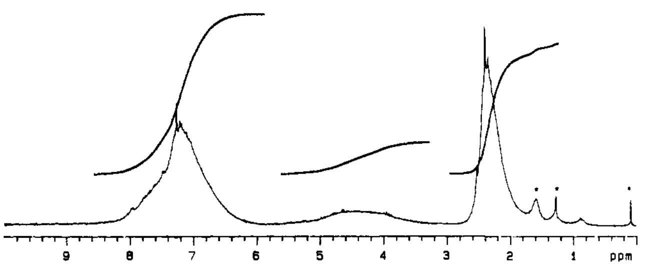

Figure 1. ^1H NMR spectrum (300 MHz) of fullerenated toluene ($C_{60}(C_6H_5-CH_3)_{12}$) in $CDCl_3$ solution at ambient temperature. The asterisks denote peaks due to unidentified impurities.

We report now that both pure C_{60} and C_{60}/C_{70} mixtures undergo $AlCl_3$-catalyzed reaction (as well as reaction catalyzed by other strong acids) with aromatics such as benzene and toluene to polyarenefullerenes. Since Ar and H are added across fullerene double bonds, the reaction can be characterized as fullerenation of aromatics.

$$C_{60} \xrightarrow[\text{AlCl}_3]{\text{H-Ar (excess)}} C_{60}(\text{H-Ar})_n$$

Reaction of 30 mg of the C_{60}/C_{70} mixture[16b] with 30 mg of aluminum trichloride in 20 mL of benzene at room temperature for 2 h gave a dark reddish-brown homogeneous solution. Quenching the solution with water and the usual workup (using chloroform) gave upon evaporation of solvent a brown colored solid. The FT-IR spectrum showed aromatic C–H stretching at 3056 and 3022 cm^{-1}. The 300-MHz ^1H NMR spectrum (in $CDCl_3$) indicated not only broad aromatic absorption centered at $\delta(^1H)$ 7.4 but also a broad C–H absorption at $\delta(^1H)$ 4.5. Integration of the peaks shows roughly 5:1 relative intensities (implying monosubstitution on the phenyl ring). The 75-MHz ^{13}C NMR spectrum showed a broad absorption centered around $\delta(^{13}C)$ 128, with extremely broad absorptions centered at $\delta(^{13}C)$ 146 and $\delta(^{13}C)$ 54. The most useful information was obtained from FAB (fast atom bombardment) mass spectrometry.[17] The FAB mass spectrum of the product indicated a strong mass peak at M^+ 1656, supporting the formation of $C_{60}(C_6H_6)_{12}$. Some evidence for the formation of $C_{60}(C_6H_6)_{16}$ was also obtained in the mass spectrum (M^+, 1968, weak absorption). Other mass peaks corresponding to various species containing varying amounts of benzene adducts were also detected all the way down to $C_{60}(C_6H_6)_6$. These peaks could be due to constant neutral C_6H_6 loss from higher species. These results support direct fullerenation of benzene by initial protonation of fullerene to the corresponding fullerene cation and subsequent Friedel–Crafts alkylation (vide infra). The addition of $H-C_6H_5$ is also supported by the observation of the C–H fullerene skeletal hydrogens at $\delta(^1H)$ 4.5. The Friedel–Crafts type fullerenation was also confirmed by carrying out the reaction with C_6D_6. Analysis of the product by FAB showed M^+ at 1728, which corresponds to $C_{60}(C_6D_6)_{12}$, and peaks due to progressive loss of mass 84 (C_6D_6). The FT-IR spectrum showed a characteristic C–D stretching frequency at 2271 cm^{-1}. In all deuterated and nondeuterated samples, peaks corresponding to C_{60} were also observed. However, we could not obtain any evidence for the phenylation of C_{70} in the fullerene mixture.

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(17) The FAB measurements were carried out on a VG Analytical Ltd. ZAB-SE high-resolution reverse-geometry mass spectrometer.