There are also implications in this work for the possibilities of asymmetric induction in cholesteric media. Eskanazi et al.[17] critically reexamined such studies up to 1979 and concluded that such induction can generally be expected to result in only low optical yields *except* for cases where strong solute–solvent interactions exist. Several additional studies since 1979 have claimed small enantiomeric excesses for reactions in cholesteric media;[18-20] one claim of a rather large (16%) excess has been made for some hydrogenation reactions.[21] The very large values of HTP for QP implies that this may be a case of a very strong solute–solvent interaction, and an attempt to capture these twist states in appropriate chemical reactions is therefore an intriguing possibility.

Acknowledgment. This work was supported by the national Science Foundation, Solid State Chemistry, under Grant No. DMR89-17833.

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Triplet States of Fullerenes C_{60} and C_{70}: Electron Paramagnetic Resonance Spectra, Photophysics, and Electronic Structures

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The recent macroscopic preparation of C_{60} and C_{70} (buckminsterfullerenes) has resulted in renewed interest in these materials from both theoretical and practical standpoints.[1-4] As yet, no detailed X-ray structures exist for these molecules. Estimates of the sizes and symmetries of these molecules from theoretical work[5-13] combined with recent scanning tunneling microscopy[14,15] and ^{13}C NMR[16,17] observations. While the photophysics of these molecules have been studied in the gas phase,[18] only one report has appeared recently that presents data on the photophysics of C_{60} in condensed media.[19] We now report optical and magnetic resonance measurements on the lowest excited triplet states of C_{60} and C_{70} that yield structural information.

C_{60} and C_{70} solutions in degassed toluene[20] (5×10^{-4} M) at 295 K were excited directly within their low-lying, weak optical transitions with 4-ps laser flashes at 515 and 450 nm, respectively.[21]

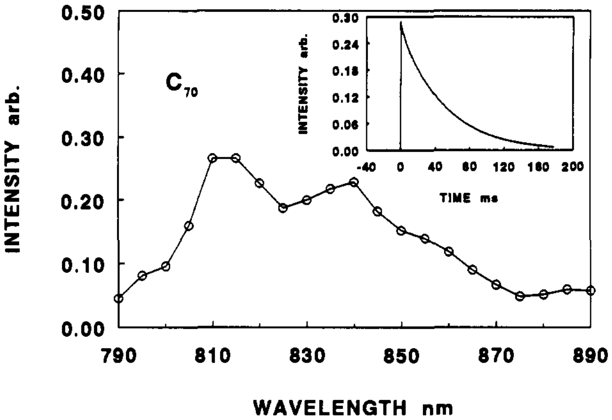

Figure 1. Phosphorescence spectrum of C_{70} in toluene/10% poly(α-methylstyrene) at 77 K obtained 10 ms after a 5-ns, 532-nm laser flash. A Burle (RCA) 8852 photomultiplier tube and a Jarrell-Ash 0.25-m monochromator were used for detection. Inset: phosphorescence decay at 810 nm.

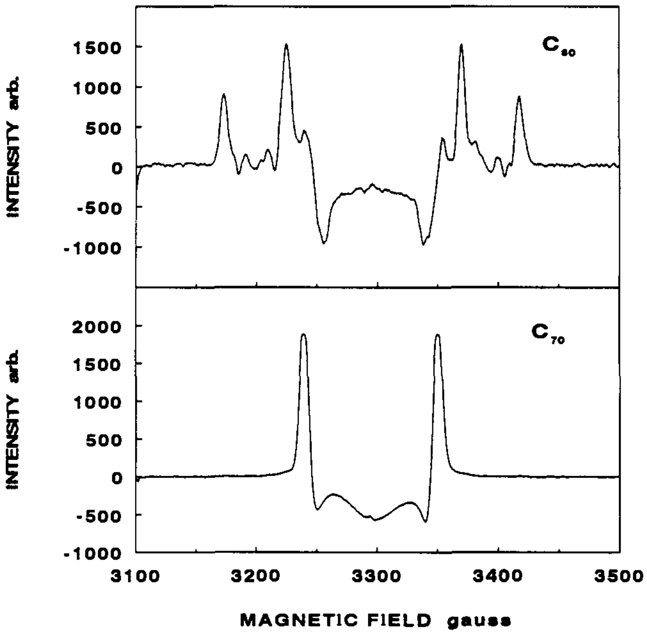

Figure 2. EPR spectra of $^3C_{60}$ and $^3C_{70}$ at 5 K, $\nu = 9.248$ GHz, microwave power = 1 mW, $\lambda > 400$ nm, 1-kHz light modulation, phase-sensitive detection, four field scans averaged, Varian E-9 spectrometer.

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Transient absorption spectra, measured as a function of time between 400 and 580 nm, show that the initially formed lowest excited singlet states, $^1C_{60}$ and $^1C_{70}$, decay to long-lived (>1 μs) transient states, presumably the corresponding lowest excited triplet states, $^3C_{60}$ and $^3C_{70}$. This conjecture was confirmed by measurements at longer times (vide infra). The rate constants for the decay of $^1C_{60}$ and $^1C_{70}$ are 3.0×10^{10} and 8.7×10^9 s^{-1}, respectively, and match those for the formation of $^3C_{60}$ and $^3C_{70}$, respectively.

Figure 1 shows the phosphorescence spectrum of $^3C_{70}$ at 77 K in degassed toluene containing 10% poly(α-methylstyrene) (39 kD) as a glassing agent. If the 810-nm emission band is ascribed to the (0,0) transition, the energy of T_1 is 1.53 eV. The phosphorescence quantum yield is 0.0013 and was measured by using zinc tetraphenylporphyrin ($\phi_p = 0.012$) as a reference.[22] The phosphorescence lifetime of $^3C_{70}$ is $\tau = 53.0 \pm 0.1$ ms, inset to Figure 1. Interestingly, phosphorescence was not observed from $^3C_{60}$. A low phosphorescence quantum yield may be due to a low triplet yield, dominant nonradiative decay of the triplet state, or emission beyond our 900-nm detection limit. Our magnetic resonance experiments on $^3C_{60}$ and $^3C_{70}$ address this issue.

To investigate the electronic structures of $^3C_{60}$ and $^3C_{70}$, we measured their EPR spectra at 5 K in degassed toluene by exciting the samples with light >400 nm from a 1 kHz modulated Xe lamp and using phase-sensitive detection of the EPR signal.[23] The spin-polarized EPR spectra of both $^3C_{60}$ and $^3C_{70}$, Figure 2, are very intense, exhibiting an aaa eee polarization pattern,[24] which implies that intersystem crossing from $^1C_{60}$ and $^1C_{70}$ results in dominant population of a single zero-field triplet sublevel defined as the z direction in the molecular axis system. This axis is probably aligned along the unique long dimension in $^3C_{70}$, while a structural distortion in $^3C_{60}$ may result in selective population of its triplet sublevels. The zero-field splittings due to the dipolar interaction of the two unpaired electron spins in the π systems of $^3C_{60}$ and $^3C_{70}$ are $|D| = 0.0114$ cm^{-1}, $|E| = 0.00069$ cm^{-1}, and $|D| = 0.0052$ cm^{-1}, $|E| = 0.00069$ cm^{-1}, respectively.

The value of $|D|$ measures the average distance between the two unpaired electrons, while the value of $|E|$ indicates the degree to which the π-electron distributions in $^3C_{60}$ and $^3C_{70}$ deviate from axial symmetry.[25] Importantly, $|E|$ for both $^3C_{60}$ and $^3C_{70}$ is very small, which shows that $^3C_{60}$ and $^3C_{70}$ are very nearly axially symmetric. This is consistent with the structures of C_{60} and C_{70} suggested by recent ^{13}C NMR measurements.[16,17] On the other hand, $^3C_{60}$ has a $|D|$ value that is about twice that of $^3C_{70}$. The average distance between the unpaired electrons within $^3C_{60}$ and $^3C_{70}$ can be calculated analytically from their respective $|D|$ values. For spins separated by an average distance r, the dipolar spin Hamiltonian is diagonalized to yield $|D|$ and $|E|$. For the case of unit spins, $|E| = 0$, and the calculated values of $|D|$ fit the experimentally determined numbers when $r = 6.1$ Å for $^3C_{60}$ and $r = 7.9$ Å for $^3C_{70}$.

These calculated average values show that the two spins within both $^3C_{60}$ and $^3C_{70}$ reside primarily at the farthest ends of their respective π systems. The value of 6.1 Å for $^3C_{60}$ correlates well with the assumed 7.1-Å nuclear diameter of C_{60} obtained via theoretical modeling.[9] Intuitively, one expects that electron correlation effects will distribute the spins of $^3C_{70}$ at opposite ends of the long axis in this molecule. The longer, 7.9-Å dimension and axial symmetry for $^3C_{70}$ are consistent with the prolate spheroidal (rugby ball) shape assumed for C_{70} and with its ^{13}C NMR spectrum.[16,17]

Since we could not detect phosphorescence from $^3C_{60}$, and since the EPR spectra for both $^3C_{60}$ and $^3C_{70}$ are very intense, we measured the triplet state lifetime for these molecules using time-resolved EPR. Samples of C_{60} and C_{70} in degassed toluene/10% poly(α-methylstyrene) at 9 K were subjected to 10-μs, λ > 400 nm light flashes at a 5-Hz repetition rate. The transient EPR signal was recorded with a digitizing oscilloscope. The data for $^3C_{60}$ and $^3C_{70}$ are shown in Figure 3. Data for $^3C_{70}$ were obtained for the line at 3240 G, which consists of the overlapping lines due to molecules oriented with their canonical z and x directions along the magnetic field (assuming $D > 0$). The decay of this signal occurs with $\tau = 51 \pm 2$ ms and agrees very well with the triplet lifetime of $^3C_{70}$ obtained from our phosphorescence decay measurement. The data for $^3C_{60}$ shows that its triplet lifetime is 0.41 ± 0.01 ms for the x direction (3224 G) and 0.29 ± 0.01 ms for the z direction (3172 G).[26] Our EPR results indicate that the yield of $^3C_{60}$ is significant, yet we observe no phosphorescence from $^3C_{60}$. Triplet–triplet energy transfer studies on $^3C_{60}$ suggest that the energy of T_1 is about 1.6 eV.[19] Thus, phosphorescence from C_{60} should be well within the wavelength range of our photomultiplier. If the radiative decay rate of $^3C_{60}$ is similar to that of $^3C_{70}$, the fact that the average lifetime of $^3C_{60}$ is about 160 times shorter than that of $^3C_{70}$ implies that decay of $^3C_{60}$ to ground state is dominated by nonradiative processes, even at 9 K.[27]

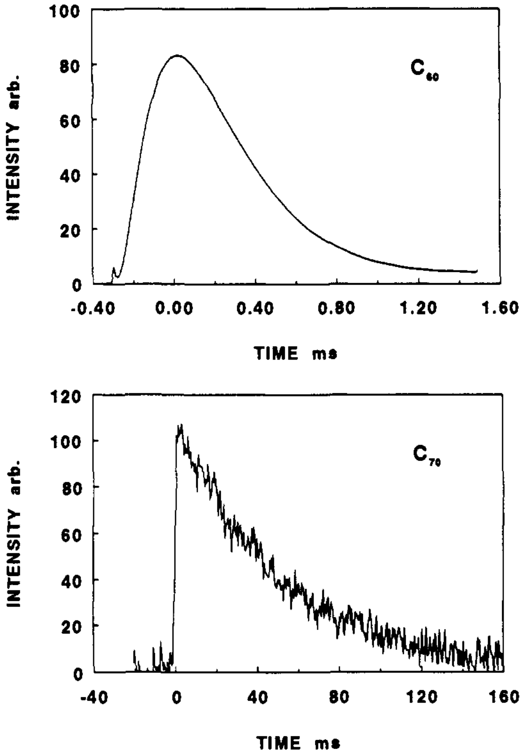

Figure 3. Decay of the EPR signal at 9 K for $^3C_{60}$ at 3224 G ($\nu = 9.248$ GHz), 1000 flashes averaged with a TEK 7912AD programmable digitizer, and for $^3C_{70}$ at 3240 G ($\nu = 9.248$ GHz), 128 flashes averaged with a TEK 11401 digitizing oscilloscope. The finite rise observed in the $^3C_{60}$ signal is due to the instrument response.

Our data show that the lowest excited triplet states of C_{60} and C_{70} in condensed media exhibit substantially different photophysical and structural characteristics. Besides being excellent vehicles for studying the fundamental photophysics of molecules with extended π systems, these materials may find use as photosensitizers, e.g., in the production of singlet oxygen[19] for photobiological or photopolymerization applications.

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$AlMe_2(BHT)NH_3$: An Unusually Stable Organoaluminum–Ammonia Complex and Its Extended Coordination Sphere Solvate

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The elimination–condensation reaction sequence (eq 1) that occurs between an aluminum alkyl and a primary or secondary amine has been widely explored as a route to aluminum nitrogen ring and cluster compounds.[1] Similar condensation reactions have been recently exploited with ammonia as the nitrogen source for the low-temperature preparation of aluminum nitride (eq 2).[2,3]

$$AlR_3 + HNR'_2 \rightarrow [R_2AlNR'_2]_n + RH \qquad (1)$$

$$AlR_3 + NH_3 \xrightarrow[>400\ °C]{\Delta} AlN + 3RH \qquad (2)$$

Reaction of aluminum alkyls with ammonia results in the initial formation of the simple Lewis acid–base adducts (eq 3), which are ordinarily unstable with respect to condensation.[3-5] Furthermore, no organoaluminum–ammonia adducts have been structurally characterized.

$$AlR_3 + NH_3 \rightarrow R_3AlNH_3 \qquad (3)$$

We have previously reported[6] that no condensation reactions are observed between primary and secondary amines and organoaluminum compounds of BHT (2,6-di-*tert*-butyl-4-methylphenoxide, BHT-H from the trivial name butylated hydroxytoluene). We have proposed that this lack of reactivity is due not to any steric hindrance but to the reduced nucleophilicity of the aluminum alkyls, as a consequence of π-donation from the aryloxide to aluminum.[6] We report herein the synthesis and structure of the thermally robust complex $AlMe_2(BHT)(NH_3)$ (**1**) as well as its stable complex with excess ammonia.

The addition of excess NH_3 to $AlMe_2(BHT)(OEt_2)$[7] results in the quantitative formation of the Lewis acid–base complex $AlMe_2(BHT)(NH_3)$ (**1**) (eq 4).[8] Compound **1** may also be synthesized[2] via the reaction of $[AlMe_2NH_2]_3$[9] with BHT-H (eq 5). Given that the reaction of both aluminum alkyls, $(AlR_3)_n$,

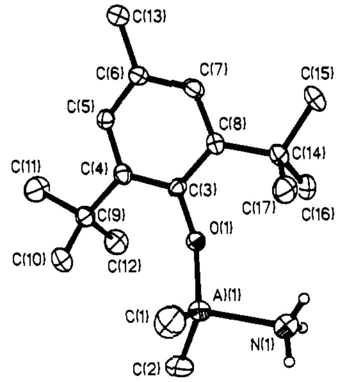

Figure 1. A view of the $AlMe_2(BHT)(NH_3)$ molecule showing the atom-labeling scheme. Thermal ellipsoids show 40% probability levels. Hydrogen atoms, except those bonded to N(1), have been omitted.

$$AlMe_2(BHT)(OEt_2) + NH_3\ (excess) \xrightarrow[pentane]{-78\ °C} \underset{\mathbf{1}}{AlMe_2(BHT)(NH_3)} \qquad (4)$$

$$\tfrac{1}{3}[AlMe_2NH_2]_3 + BHT\text{-}H \xrightarrow[pentane]{25\ °C} \mathbf{1} \qquad (5)$$

and amides, $[Al(NR_2)_3]_n$, with alcohols has been extensively utilized for the synthesis of alkoxides,[10] it is perhaps contrary to expectations that the aluminum–nitrogen bond is the exclusive site of reactivity in the present case.

The IR and multinuclear NMR spectra of **1** are consistent[11] with a 1:1 complex, the structure of which has been confirmed by X-ray crystallography.[12] The molecular structure of compound **1** is shown in Figure 1. The Al–N distance [2.015 (5) Å] is within the range observed for amine adducts of $AlMe_3$ (1.88–2.09 Å),[13] while the aryloxide Al–O distance [1.743 (4) Å] and Al–O–C angle [149.6 (3)°] are in the ranges observed previously and are consistent with a small degree of π-bonding between oxygen and aluminum.[14]

In contrast to previously reported organoaluminum–ammonia complexes, compound **1** shows no propensity for alkane elimination, giving a parent ion in the mass spectrum,[15] and may be sublimed without decomposition at 130 °C (10^{-2} mmHg). Heating under an inert atmosphere beyond its melting point (170 °C)

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[8] To a rapidly stirred pentane (60 mL) solution of $AlMe_2(BHT)(OEt_2)$ (1.00 g, 2.85 mmol) at −78 °C was added NH_3 (excess) from a lecture cylinder. The reaction mixture was allowed to warm to room temperature, at which point a large amount of precipitate had formed. Removal of solvent in vacuo led to a white powder, which was crystallized from pentane/toluene. Yield: 60–80% isolated; mp = 170 °C.

[9] To a suspension of $[AlMe_2NH_2]_3$ (0.50 g, 2.28 mmol) in pentane (40 mL) was added at room temperature BHT-H as a solid (1.51 g, 6.86 mmol). The reaction mixture was stirred for 36 h, after which the volatiles were removed under vacuum. Proton NMR of the crude material indicated **1** as the only product.

[10] See, for example: (a) Oliver, J. P.; Kumar, R. *Polyhedron* **1990**, *9*, 409. (b) Chisholm, M. H.; DiStasi, V. F.; Streib, W. E. *Polyhedron* **1990**, *9*, 253.

[11] NMR (ppm, toluene-d_8): ^1H 7.15 (2 H, s, C_6H_2), 2.34 (3 H, s, CH_3), 1.44 [18 H, s, $C(CH_3)_3$], 1.10 (3 H, br s, NH_3), −0.55 (6 H, s, $AlCH_3$); ^{13}C 155.22 (OC), 138.11 (*o*-C), 126.08 (*m*-C), 125.35 (*p*-C), 34.80 [$C(CH_3)_3$], 31.12 [$C(CH_3)_3$], 21.45 (CH_3), −6.09 ($AlCH_3$); ^{14}N 91 ($W_{1/2}$ = 4200 Hz); ^{27}Al 136 ($W_{1/2}$ = 4400 Hz). IR (cm^{-1}, Nujol, KBr): 3360, 3320, 3230, 3150 ν(N–H).

[12] Crystal data for $AlMe_2(BHT)(NH_3)$ (**1**): monoclinic, $P2_1/n$, a = 9.287 (11) Å, b = 12.096 (5) Å, c = 16.836 (9) Å, β = 96.70 (7)°, V = 1878 (3) $Å^3$, Z = 4, D(calcd) = 1.297 g cm^{-3}, λ(Mo Kα) = 0.71073 Å (graphite monochromator), T = −80 °C. A Nicolet R3m/V diffractometer, equipped with an LT-1 low-temperature device, was used to collect 5163 reflections ($4° < 2\theta < 45°$) on a colorless crystal 0.21 × 0.32 × 0.29 mm. Of these, 2456 were independent and 1666 observed [$F_o > 4\sigma F_o$]. A semiempirical absorption correction based on four ψ scans and Lorentz and polarization corrections were applied to the data. All the non-hydrogen atoms were located by direct methods, and they were refined anisotropically. The hydrogen atoms were included as idealized contributions. R = 0.083, R_w = 0.091, GOF = 0.90, final residual = 0.36 e $Å^{-3}$. All computations used SHELXTL-PLUS: Sheldrick, G., Nicolet XRD, Madison, WI, 1987.

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[15] Mass spectrum, 293 (M^+), 276 ($M^+ - NH_3$), 261 [AlMe(BHT)].

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