STUDIES OF C60, C70 AND OTHER FULLERENES:
SYNTHESIS, DERIVATIZATION, ELECTROCHEMISTRY,
LUBRICANT PROPERTIES AND NEW COMPOSITE MATERIALS

FINAL REPORT

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New designs for fullerene generators have been developed, including systems for producing endohedral species with metals inside the C60 or C70 cages. Also, focussed solar energy has been utilized for fullerene production. Electrochemical studies have led to the identification of C60⁻¹, C60⁻²...C60⁻⁶ ions and utilized for preparation of superconducting K₃C₆₀. Birch reductions of C60 and C70 led to C₆₀H₃₀ and C₇₀H₃₀ respectively. Adducts of C₆₀ with tetraphenylcyclopentadienone and with cyclopropananaphthalene are described, and low-temperature reactions of C₆₀ with H₂ and with energetic nitroben ion beams are reported. C₆₀ does not appear to enhance the lubricity of motor oil formulations. The vapor pressure has been measured and it is estimated that the total pressure reaches one atmosphere at ca. 1523K.
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INTRODUCTION

The goal of this research was to investigate various materials applications, materials properties and the chemistry of the newly available soccer ball-shaped buckminsterfullerene, C_{60}, and related substances ("fullerenes"). We proposed to explore research areas that could lead to important advances in materials science, energy technology, and, possibly, even in biomedicine. The results of this research effort at Rice University are documented in the Ph.D. theses of 10 students (6 more in progress) and in many publications, including a definitive book edited by W.E. Billups and M.A. Ciufolini. In the various sections of this report and the related publications, we describe advances in the preparation and characterization of fullerenes and metal endohedral fullerenes; the electrochemical synthesis of superconducting fullerides; chemical reactions of fullerenes with hydrogen, helium, and organic molecules; and measurements of vapor pressures, lubricity and other properties.


Since the original discovery in 1985 that fullerenes are produced spontaneously in laser-generated carbon vapors,\(^1\) extensive research has been devoted to finding ways of making these species in large amounts at high yield. Triggered 5 years later by the success of Kratschmer et al.\(^2\) in producing the first milligram amounts by resistive heating of graphite in an atmosphere of helium, the manufacture of these new materials has now
become something of a cottage industry. Even with subsequent improvements using carbon arcs, production of fullerenes remains a highly expensive process which has resisted all attempts to scale-up beyond several tens of grams per hour. This figure applies to the most abundant fullerenes, C₆₀ and C₇₀. Higher fullerenes are produced by current techniques in very small amounts and they are even more expensive.

Buckminsterfullerene (C₆₀) has now been prepared in multigram quantities by contact arc vaporization of a graphite rod in a 100-Torr atmosphere of helium, followed by extraction of the resultant graphitic soot with toluene. The dominance of C₆₀ in this extract has been verified by mass, FTIR and NMR Spectroscopy.³,⁴

In the course of an extensive series of experiments aimed at increasing the production of both empty fullerenes and endohedrally doped metallofullerenes, Smalley and Chibante⁵ may have discovered the principal mechanism which has prevented efficient scaling of the carbon arc technique to large rod sizes, i.e., photochemical destruction of the fullerenes by light from the arc. After considering ways this problem can be overcome in a fashion that scales well to large rod sizes, they proposed that the best answer may also be the simplest: sunlight. A solar furnace has been successfully used for C₆₀ preparation.⁵


By operating the carbon arc in a temperature-controlled flowing environment, fullerenes remain in the gas phase and condense on cold surfaces downstream. By using metal-doped graphite electrodes in such a configuration, and by providing a filtering medium, metallofullerenes can be extracted from the non-sublimable soot. Deposition onto a retractable collection surface allows these materials to be transported from the apparatus without ever contacting air or moisture. By vacuum distilling away the empty fullerenes, one can produce metallofullerenes without the use of solvents while maintaining an inert environment. Characterization and utilization of these unique materials is now feasible.

Macroscopic quantities of fullerenes with a metal atom inside were first produced for lanthanum by laser vaporization of a La₃O₃/graphite composite rod in a high-temperature tube furnace.⁶ In the sublimed film, La@C₆₀, La@C₇₀, La@C₇₄, and La@C₈₂ were all present, while La@C₈₂ was found to be abundant and uniquely soluble in toluene. Many rare earth elements have been found to be readily trapped inside fullerenes with one or more atoms both by the laser vaporization/high temperature furnace technique and by the carbon arc technique.
Recently, the electronic structure of Ca@C60 in the gas phase was probed by ultraviolet photoelectron spectroscopy (UPS) of Ca@C60. The electron affinity of Ca@C60 was found to be 3.0 eV. The spectrum suggests that Ca donates its two 4s electrons to the C60I1u LUMO. Ab initio self-consistent field Hartree-Fock calculations were performed on Ca@C60 at four different symmetries, Ih, D3d, D5d, and C5v. It was found that the C5v symmetry has the lowest energy with the central Ca2+ ion 0.7 Å away from the center of C60 and that Ca@C60 has a 3A2 triplet electronic ground state in C5v symmetry.


In November of 1990, we reported the first electrochemical reduction of C60 to the C60− and C602− ions in solution. Subsequently, electrochemical reductions to the C603−, C604−, C605−, and C606− anion levels were also reported. Shortly thereafter, this observed production of fulleride ions in solution by electrochemistry took on new importance with the discovery that K3C60 (containing discrete C603− ions) formed a metallic superconducting phase with Tc = 18 K.12 Our DARPA-sponsored research has explored the possibility of using electrochemistry to electrosynthesize and electrocrystallize superconducting salts of C603− from solution. An electrosynthetic approach to such superconducting materials has potential advantage over chemical vapor deposition (CVD) methods in that pure, single-phase materials are often obtained by electrosynthetic methods and large single crystals also often result from electrocrystallization techniques. Our methods and initial results are documented below. A recent publication describing these results in more detail is also available.13

Electrochemistry of C60

C60 reduction processes. Electrochemical analysis of C60 reveals the existence of at least eight oxidation states, (C60)n: n = 0, -1, -2, -3, -4, -5, -6, and +x, where x has yet to be definitely established) by use of cyclic voltammetry and differential pulse polargraphy. The six known reductions of C60 are all reversible by cyclic voltammetry at -10°C as shown in Figure 1. Historically, these six reduction processes for C60 were uncovered literally "one electron at a time" with the two-, three-, four- and five- and finally six-electron processes being reported.
All six electrochemical reductions of Figure 1 are reversible at a scan rate of 100 mV s⁻¹ in a CH₃CN/toluene solvent system at -10°C. Even more remarkable, the reductions occur at fairly evenly-spaced potentials (ca. Δ 200 mV). Initial M.O. calculations on C₆₀ predicted a triply-degenerate LUMO (lowest unoccupied molecular orbital) of t₁u symmetry,¹⁴ and the observed electrochemistry of C₆₀ supports this view.

Other solvents such as THF or CH₂Cl₂ reveal less reversible electrochemical behavior, especially for the more negative reduction processes.⁸ Bulk electrolysis in benzonitrile verifies the first four reductions of C₆₀ as one-electron transfers.¹⁰a The C₆₀⁻, C₆₀²⁻, and C₆₀³⁻ fulleride ions remain stable in benzonitrile for up to several days, and neutral C₆₀ is quantitatively recovered from these solutions after reoxidation. Epr spectroelectrochemical studies of these fulleride ions in frozen solution indicated S = 1/2 ground states for C₆₀⁻ and C₆₀³⁻ and an S = 1 ground state for the C₆₀²⁻ ion.¹⁴ C₆₀⁴⁻ has so far only been produced by bulk electrolysis in either benzonitrile or pyridine using the wide reduction windows of these solvents, however, quantitative recovery of C₆₀ was not achieved upon reoxidation of the C₆₀⁴⁻ solutions.¹⁵

Electrosynthesis of K₃C₆₀

The discovery of conditions under which all six reductions of C₆₀ are reversible at slow scan rates indicated that the C₆₀⁻ → C₆₀⁶⁻ anions could be generated in bulk by controlled-potential electrolysis (CPE),¹⁰ and we initiated such studies and selected K₃C₆₀ as an initial target compound because of its superconducting properties. Previous syntheses of superconducting K₃C₆₀ employed a chemical vapor deposition (CVD) approach, but we believe that an electrosynthetic method offers real advantages over CVD.
Specifically, we anticipated being able to prepare literally gram quantities of crystalline $K_3C_{60}$ and hopefully to grow large single crystals from solution in order to directly measure electrical transport properties (e.g., current density) in single crystals. By comparison, a CVD synthetic approach offers less hope of obtaining large single crystals for such measurements. Finally, an electro-synthetic approach also offers the prospect of producing new materials such as $K_nC_{60}$ (with $n = 1$) and $K_2C_{60}$ (with $n = 2$), whereas these less-highly-reduced phases of $C_{60}$ have remained elusive with high-temperature CVD syntheses.

After much trial and error, we settled on a solvent/electrolyte system of CH$_3$CN/KPF$_6$ for our electrosynthesis of $K_3C_{60}$. A flow diagram of the synthesis is shown in the Figure 2 where the potential is held at -1.6 V (v.s. Ag/AgCl) during the electrolysis at 40 cm$^2$ Pt gauze working electrode. The advantage offered by the CH$_3$CN/KPF$_6$ system is that only the $K_3C_{60}$ product crystallized from the bulk solution, leaving the supporting electrolyte (KPF$_6$) in solution as a clean method of separating $K_3C_{60}$ from KPF$_6$. Finally, washing the solid $K_3C_{60}$ with toluene removed any unreduced C$_{60}$ from the product. These preliminary results indicate that an electrosynthetic approach to fulleride-based materials should be a general success. Electrocryrstallization attempts are continuing for $K_3C_{60}$ and other $C_{60}^{3-}$ materials, and when large single crystals become available, their superconducting properties will be characterized and compared to the crystalline phases obtained by CVD methods. We hope to have such crystals soon.
4. **Birch, Reduction of C\textsubscript{60} and C\textsubscript{70} (W.E. Billups and Associates).**

The isolation of buckminsterfullerenes C\textsubscript{60} and C\textsubscript{70}\textsuperscript{1,16} in bulk quantities\textsuperscript{2,5,16} has led to a flurry of activity directed at the chemical modification of these new materials. The objective of this research was the development of a process for the production of fullerene hydrides to be used as hydrogen storage materials. In principle, it should be possible to store 30 moles of dihydrogen per mole of C\textsubscript{60}. However, C\textsubscript{60}H\textsubscript{60} is a highly energetic compound and it has not been possible to prepare this fully reduced material using conventional chemistry.

In this study, we have investigated the Birch reduction of C\textsubscript{60} as a route to new fullerene hydrides. The major product formed in the Birch reduction of C\textsubscript{60} is a species in which 36 hydrogen atoms are bound chemically to the carbon skeleton. Although mass spectral analyses are inconclusive with regard to other hydrides that are formed during the Birch reduction, there is evidence for C\textsubscript{60}H\textsubscript{18} as well as C\textsubscript{60}H\textsubscript{28} and C\textsubscript{60}H\textsubscript{32}. The mass spectrum recorded under FD conditions is shown as Figure 3. The peak at m/e 755.6 corresponds to C\textsubscript{60}H\textsubscript{36}.

A mechanism which would account for the formation of C\textsubscript{60}H\textsubscript{36} is shown in the scheme presented below.

![Birch reduction scheme](image)

It is interesting that 36 is the number of hydrogen atoms required to leave a single, non-conjugated double bond in each of the 12 cyclopentane subunits of C\textsubscript{60}. A possible structure of this product is presented as Figure 4.
Figure 3
FDMS of the Birch Reduction of 99% C\textsubscript{60}

calibration shifted -0.5 amu, C\textsubscript{60}H\textsubscript{36} = 756 amu
Figure 4
Structure of $\text{C}_{60}\text{H}_{36}$ from the Birch Reduction of $\text{C}_{60}$
Treatment of a toluene solution of the Birch-reduced product with dichlorodicyanoquinone (DDQ) led to the rapid formation of the starting fullerene C$_{60}$. Thus, the reduction of C$_{60}$ is fully reversible. This facile activation of the C-H bonds of C$_{60}$H$_{36}$ is consistent with theoretical calculations dealing with the fullerene hydrides.$^{16}$

\[
\begin{align*}
C_{60} & \xrightleftharpoons{\text{DDQ}} C_{60}H_{36} \\
\text{Li/t-BuOH/NH$_3$} & \xrightleftharpoons{\text{DDQ}} C_{60}H_{36}
\end{align*}
\]

Due to the limited availability of C$_{70}$, the chemistry of this fullerene has been somewhat less developed; however, we have isolated enough C$_{70}$ (95% purity by HPLC) to demonstrate that the Birch reduction of C$_{70}$ using Li metal in liquid ammonia and t-butyl alcohol yields a nearly white solid which also darkens irreversibly to a beige colored material upon exposure to the atmosphere. Analysis of this material by FDMS showed that the major product was C$_{70}$H$_{36}$, in strict analogy to the Birch reduction of C$_{60}$ under the same reaction conditions. Since both fullerenes contain 12 cyclopentanoid rings it seems reasonable that a similar mechanism accounts for both results. Treatment of a benzene solution of the reduced fullerene with DDQ for 5 hr at room temperature afforded a quantitative yield of the starting fullerene, as monitored by HPLC. Thus the reduction of both fullerenes appears to be fully reversible.

\[
\begin{align*}
C_{70} & \xrightleftharpoons{\text{DDQ}} C_{70}H_{36} \\
\text{Li/t-BuOH/NH$_3$} & \xrightleftharpoons{\text{DDQ}} C_{70}H_{36}
\end{align*}
\]

Calculations by Dr. Gus Scuseria suggest that the structure presented as Figure 5 may depict the most stable form of C$_{70}$H$_{36}$.

If the dihydrogen (18 moles per mole of C$_{60}$) contained in these Birch products could be removed both easily and reversibly, then these Birch products might provide a method to safely store hydrogen as a fuel source. It has been demonstrated that the Birch products of C$_{60}$ and C$_{70}$ may be removed by oxidation with DDQ, but this type of reaction would be impractical for a fuel storage device. One of the first observations made about the Birch products of the fullerenes is their relative instability, as evidenced by the rapid color change from white to beige to yellow upon exposure to air or heat. Therefore, it was decided to investigate the thermal behavior of the Birch product of C$_{60}$ by thermo-gravimetric analysis (TGA) to determine the decomposition pattern of the product.
Figure 5
Structure of C\(_{70}\)H\(_{36}\) from the Birch Reduction of C\(_{70}\)
The constraints of time have prevented detailed studies of the thermal behavior of these materials. However, some preliminary investigations using thermo-gravimetric analysis of Birch reduced C\textsubscript{60} from 25-800 °C at 5 °C/min under an argon blanket showed a 7% weight loss from 25-110 °C, a 6% weight loss from 120-260 °C, and finally a 43% weight loss from 300-750 °C. The weight loss of 7% from 25-110 °C is greater than would be expected for a simple loss of dihydrogen. If only hydrogen had been removed from the Birch product, the weight loss would have been 5%. This product had been dried \textit{in vacuo} for 24 h, and TGA of pure C\textsubscript{60} dried in this manner verified that the technique was effective for removing detectable residual solvents from the sample. Since the TGA instrument employed was not equipped for the analysis of outgases (gaseous decomposition products), this study is still inconclusive and the determination of the products produced during this process will be a long tedious task.

The characterization of these fullerene hydrides has presented a major obstacle during this investigation. As indicated above, FDMS shows a range of peaks with the major one at the position expected for C\textsubscript{60}H\textsubscript{36}. At the moment, we not able to determine whether these peaks correspond to discrete compounds or whether they are fragment peaks. We believe that some of the problems associated with characterization of these materials can be solved using \textsuperscript{3}He@C\textsubscript{60}.\textsuperscript{17,18,19}
5. Adducts of C$_{60}$

(a) Diels-Alder Reaction (J. L. Margrave, V. N. Khabashesku and R. H. Hauge)

We have studied the Diels-Alder [4+2] cycloadDITION reaction of tetraphenylcyclopentadienone (TPC) to fullerene C$_{60}$ with the aim of getting a new hard carbon material, which might be accessed by further cross-linking polymerization of the product 1, obtained via thermal or photochemical elimination of CO molecule from [C$_{60}$.TPC] adduct. The reaction is shown as follows:

![Diels-Alder Reaction Diagram]

To date we have succeeded in isolation of milligram quantities of the C$_{60}$.TPC adduct by liquid chromatography with spectroscopic characterization of this new compound by $^{13}$C NMR (Fig.6), UV-Vis (Fig. 7), IR (Fig. 8) and FAB MS (m/z 1104; M$^+$) techniques. A similar synthesis of the [4+2] adduct of isobenzofuran to fullerene C$_{60}$ was published by F. Wudl and coworkers.

Large scale synthesis of the [C$_{60}$.TPC] adduct as well as studies of [4+2] cycloaddition of C$_{60}$ to a-pyrone are now in progress. Our thin film deposition studies have shown some molecular hydrogen adsorption by neat C$_{60}$ and nickel doped C$_{60}$ films. To understand the mechanism of this process, which may be caused by initial molecular hydrogen complexation or a bond insertion hydrogenation of C$_{60}$ metal complexes, matrix isolation spectroscopic studies are now under way.
Figure 6. $^{13}$C NMR spectrum of C$_{60}$·TPC adduct in C$_6$D$_6$.

Figure 7. UV-vis spectrum of C$_{60}$·TPC adduct in benzene solution (solvent is subtracted).
Figure 8. IR spectrum of C₆₀-TPC adduct pressed into KBr pellet.
5. **Adducts of He@C_{60} (W.E. Billups and Associates).**

The recent discovery that $^3$He can be introduced into the fullerenes C60 and C70 by heating at 620°C with the helium at high pressure represents an important advance in the characterization of fullerene derivatives since the $^3$He NMR spectrum of each product should yield a single sharp peak and no non-fullerene products or impurities would give signals.\textsuperscript{17-19} The feasibility of using this technique was demonstrated by carrying out a simple reaction in which a diradical was condensed with $^3$He@C_{60} (see Figure 9). We are currently carrying out the Birch reductions using $^3$He@C_{60}. Examination of the $^3$He NMR spectrum of our products should provide a good indication of the number of isomers produced in this reactions.
$^3\text{He}$ NMR spectrum of the Reaction Product of C60 and Cyclopropanaphthalene
6. **Reactions of C$_{60}$-Thin Films with Hydrogen and with Nitrogen**

Our thin film deposition studies have shown some molecular hydrogen adsorption by neat C$_{60}$ and nickel doped C$_{60}$ films at low temperatures. This process may be caused by initial molecular hydrogen complexation or a bond insertion hydrogenation of C$_{60}$ metal complexes.

Both empirical and ab initio calculations$^{21}$ have shown that covalent carbon-nitrogen solids such as b-C$_3$N$_4$ may possess hardness comparable to diamond. Such predictions provide a strong reason for experimental efforts$^{22-24}$ to synthesize this superhard material. Such a material offers exciting prospects for new hard coating applications. Recently, C. Niu et al.$^{25}$ reported the synthesis of hard C-N films, containing up to 40 percent nitrogen. These films were prepared by laser ablation of graphite targets to produce carbon beams which were combined with atomic nitrogen from a radio frequency discharge source. The films were grown at substrate temperatures up to 800° C.

The goal of this work was to explore the possibility of growing hard carbon-nitrogen thin films at cryogenic temperatures. As a carbon source we have chosen the fullerene C$_{60}$, which can be vaporized by simple resistive heating at much lower temperatures than graphite. The films were prepared by co-deposition of a mixture of about 90% C$_{60}$ and 10% C$_{70}$ with highly energetic nitrogen beams, created in a Kauffman ion source.$^{26}$ Both beams were directed at a Si wafer which is held at various temperatures from 300 down to 18 K. The nitrogen source was located inside a vacuum chamber such that the distance between the nozzle of beam focusing cone and the substrate was about 10-15 mm. Samples were deposited at a variety of nitrogen flux energies (from 300 to 700 V) and controlled C$_{60}$ deposition rates (from about 500 to 1200 Å/hour). The deposits were studied by FTIR and Raman spectroscopy.

With the nitrogen beam present no C$_{60}$ features remained in the spectra of all deposited films. This demonstrated complete chemical transformation of the fullerenes.
Formation of graphite was found to be the main process occurring at high C$_{60}$ deposition rates (> 1000 Å/hour). At lower C$_{60}$ deposition rates (~ 500 - 600 Å/hour) we observed an increase of the amorphous carbon/graphite ratio as indicated by relative peak intensities in the Raman spectra. The nitrogen beam voltage was varied from 300 to 700 V and substrate temperature from 300 K (Fig. 10a down to 80 K (Fig. 10b).

Transformation of the C$_{60}$ into a new nongraphitic material was observed at substrate deposition temperatures of 18-20 K. The Raman spectrum of this sample (Fig. 10c) did not show characteristic bands of C$_{60}$ or those for amorphous carbon and graphite. The FTIR spectrum of the film deposited under the same conditions showed several new bands. None of those bands belong to the C$_{60}$/C$_{70}$ precursor. Two of them, at 1030 and 1270 cm$^{-1}$, were located in 1000-1280 cm$^{-1}$ C-N stretching vibrations range.

The above results demonstrate that energetic nitrogen atoms at cryogenic temperatures physically destroy fullerene molecules to form graphite or amorphous carbon at temperatures where molecular nitrogen does not condense. At lower temperatures (<30 K) where molecular nitrogen forms a film complete reaction of the deposited fullerene with nitrogen is observed. This strongly suggests that hard carbon films can be obtained by controlling the nitrogen deposition and reaction rate by simply controlling the substrate temperature. The use of high purity fullerene carbon beam sources, free of hydrogen impurities which are often found in graphite samples, along with a high purity nitrogen sources in a UHV system should be an excellent route to highly pure carbon-nitrogen films of variable stoichiometry with high nitrogen contents.
Figure 10. Raman spectra of carbon films formed by co-deposition of C$_{60}$ (vaporized at 470° C) with high-energy nitrogen flux (beam voltage 700 V) on Si substrate at temperatures a - 300 K, b - 80 K, c - 18 K.
7. **C₆₀ as a Lubricant** (John L. Margrave and Associates)

In collaborative studies with one of the Houston Petrochemical Research Laboratories, we have evaluated C₆₀ as an additive to various motor oils, mineral oils and thermally stable organics but there has been no indication of enhanced lubricity. Among the negative factors observed were:

1. Low solubility of C₆₀
2. High vapor pressure of C₆₀
3. Ease of oxidation of C₆₀

There does not appear to be any special advantage in using C₆₀ as a lubricant additive for standard motor oils.


The transpiration method is one of the oldest and most versatile ways of studying heterogeneous equilibria involving solids and gases. In the transpiration experiment, an inert gas, a carrier, is passed over a condensed sample at a flow rate sufficiently low for equilibrium conditions to be established. The vapor of the sample is transported by the carrier gas to some point downstream from the sample and is collected and analyzed to determine the vapor pressures of components in the sample.

The quartz crystal microbalance (QCM) has also proved to be a useful tool in conjunction with the Langmuir free evaporation technique for measuring the vapor pressures of solids and liquids. Its high mass sensitivity allows the measurement of vapor pressures several decades lower than other methods. By employing a Knudsen effusion cell, this technique can be used with elimination of ambiguities relating to evaporation coefficients. Two major requirements have to be met: (1) the sticking coefficient of the vapor on the QCM must be near unity, and (2) the condensation of impurities from the sample container and the heating device must be negligible. A molecular beam, characteristic of the equilibrium vapors in the Knudsen cell, is formed by the species effusing from the cell orifice. This molecular beam can then be analyzed either with a mass spectrometer or with a deposition monitor (e.g., QCM).

The sublimation pressures in equilibrium with a polycrystalline C₆₀/C₇₀ solid solution have been measured with a quartz crystal microbalance (QCM) and by transpiration methods, in the temperature ranges 772-857 and 806-929 K, respectively. The results from the two independent methods show good agreement. The solid solution was
found to have a total vapor pressure of $8.1 \times 10^{-4}$ Torr at 800 K. It is estimated that the total vapor pressure of the C$_{60}$/C$_{70}$ solid solution could reach 1 atm at ca. 1523 K. The analyses of the compositions of C$_{60}$ and C$_{70}$ in the solid and vapor phases also reveal that C$_{60}$ is more volatile than C$_{70}$. 
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19. "Probing the Interior of Fullerenes by $^3$He NMR of $^3$He@C\textsubscript{60} and $^3$He@C\textsubscript{70}," M.A. Saunders, H.A. Jimenez-Vasquez, R.J. Cross, S. Mroczkowski, D.I. Freedberg, F.A.L. Anet, *Nature*, 1994, 367, 256.

SELECTED PUBLICATIONS FROM PRINCIPAL INVESTIGATORS

W. E. BILLUPS


MARCO A. CIUFOLINI


"Efficient Production of C$_{60}$ (Buckminsterfullerene), C$_{60}$H$_{36}$, and the Solvated Buckide Ion. "Haufler, R. E.; Conceicao, J.; Chai, J.; Chibante, L.P.F.; Byrne, N. E. Flanagan, S.; Haley, M.M.; O'Brien, S.C.; Pan, C.; Xiao, Z.; Billups, W.E.; Ciufolini, M.A.;

ROBERT H. HAU GE


JOHN L. MARGRAVE


R. E. SMALLEY


In addition Professor Smalley has a number of other publications concerned with Fullerenes:


III. STUDENTS, POSTDOCTORAL RESEARCHERS AND OTHER COLLABORATORS

(a) Students Who Have Completed Ph.D. Theses Involving C_{60} - Research

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<td>J. Michael Alford</td>
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<td>Chenyu Pan</td>
<td>(J. L. Margrave)</td>
</tr>
<tr>
<td>Z. Xiao</td>
<td>(J. L. Margrave)</td>
</tr>
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(b) Other Researchers

<table>
<thead>
<tr>
<th>Name</th>
<th>Details</th>
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<tbody>
<tr>
<td>Chad Fargason</td>
<td>(L. J. Wilson) (currently pursuing a Ph.D. in mathematics at another university)</td>
</tr>
<tr>
<td>Michael Diener</td>
<td>(R. E. Smalley) (working at Los Alamos)</td>
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<tr>
<td>Ting Guo</td>
<td>(R. E. Smalley) (Ph.D. expected 5/95)</td>
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<tr>
<td>Eric Roche</td>
<td>(L. J. Wilson) (currently pursuing a Ph.D. in chemistry at another university)</td>
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<tr>
<td>Andreas Thess</td>
<td>(R. E. Smalley) (Ph.D. expected 5/97)</td>
</tr>
<tr>
<td>J. Zhang</td>
<td>(R. E. Smalley) (received M.S. 5/94)</td>
</tr>
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(c) Post-Doctoral Researchers

<table>
<thead>
<tr>
<th>Name</th>
<th>Mentor</th>
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<tbody>
<tr>
<td>Dr. V. N. Khabashesku</td>
<td>(J. L. Margrave)</td>
</tr>
<tr>
<td>L. S. Wang</td>
<td>(R. E. Smalley)</td>
</tr>
</tbody>
</table>
(d) Other Personnel

Dr. M. S. Chandrasekharaiyah, Research Chemist, Houston Advanced Research Center, The Woodlands, Texas.

Mr. D. Agan, Summer Research Student from the Conroe Independent School District, Conroe, Texas, at the Houston Advanced Research Center, The Woodlands, Texas.

Dr. Al Schultz, Ionwerks, Houston, Texas.