DOI: 10.1002/ejoc.200600675

Synthesis of a 2D Lander

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Keywords: Molecular devices / Nanotechnology / Arenes / Cycloadditions / Diels–Alder reactions

A method for the preparation of an asymmetric superbenzene is presented. The synthesis proceeds through the 12-fold cyclodehydrogenation of a polyaromatic precursor obtained by successive Diels–Alder additions of cyclopentadienones and alkynes.

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Introduction

The development of scanning-tunneling microscopes has allowed not only for imaging at the atomic scale, but also manipulation of atoms or molecules. Submolecular spectroscopy has opened the way to new experiments and new measurements on single molecules. In order to carry out such experiments,[1] we have in the past few years designed a series of molecular devices, named landers, for which the active polyaromatic hydrocarbon part is maintained above the metallic surface by spacers so that the molecular board is electronically decoupled from the surface. These spacers also increase the molecular mobility on metallic surfaces,[2] which permits a controlled molecular manipulation.[3,4] Furthermore, some of these landers have been designed in order to dock along surface step edges so that one of the platforms at the end of the polyaromatic plateau is physisorbed to the upper terrace. Such geometry has, for instance, allowed the measurement of the electronic contact between the molecular wire and the electrode (Figure 1).[5]

Moreover, these molecular landers have revealed interesting surface restructuring properties. For instance, these molecules can create trenches in the surface by stabilization or creation of vacancies in the substrate.[6,7] Alternatively, we have also shown that some landers could be used as molecular molds or templates for the fabrication of small metallic wires at the Cu(110) step edges,[8] and that the shape of these nanostructures could be controlled by the design of the molecular molds.[9] This molding property can also be used to transport metallic atoms on a surface.[10] These concepts have been recently extended to 2D landers from the hexaperi-benzocoronene (HBC) family[11] with the objective to gain insight into the electron transport in a 2D molecule and to study molding effects on larger polyaromatic hydrocarbons.

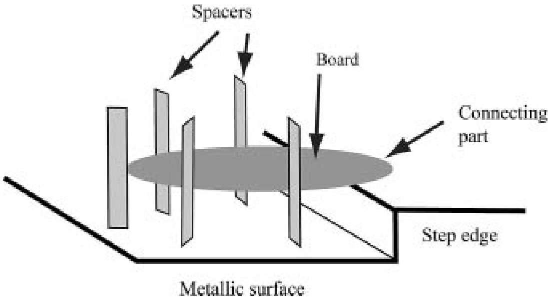

Figure 1. General concept of a 2D-lander connected at the step edge of a metallic surface.

In order to further extend these experiments of contact and molding to very large systems, we have designed a polycyclic aromatic hydrocarbon (PAH) shown in Figure 2. The "superacene" lander[12] comprises a 72 carbon atom planar main board partially surrounded by 6 *tert*-butyl spacers. The connecting part, left unsubstituted to allow contact with the upper terrace, is now larger than in previous landers in order to reduce the molecule-to-metal surface contact resistance.

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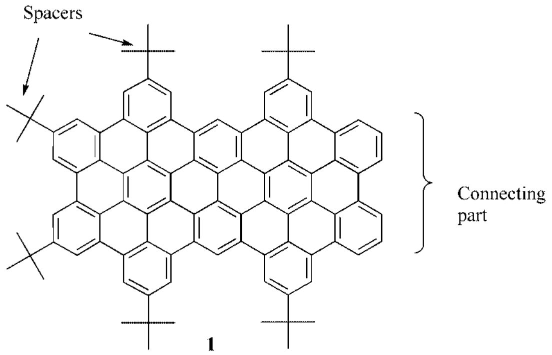

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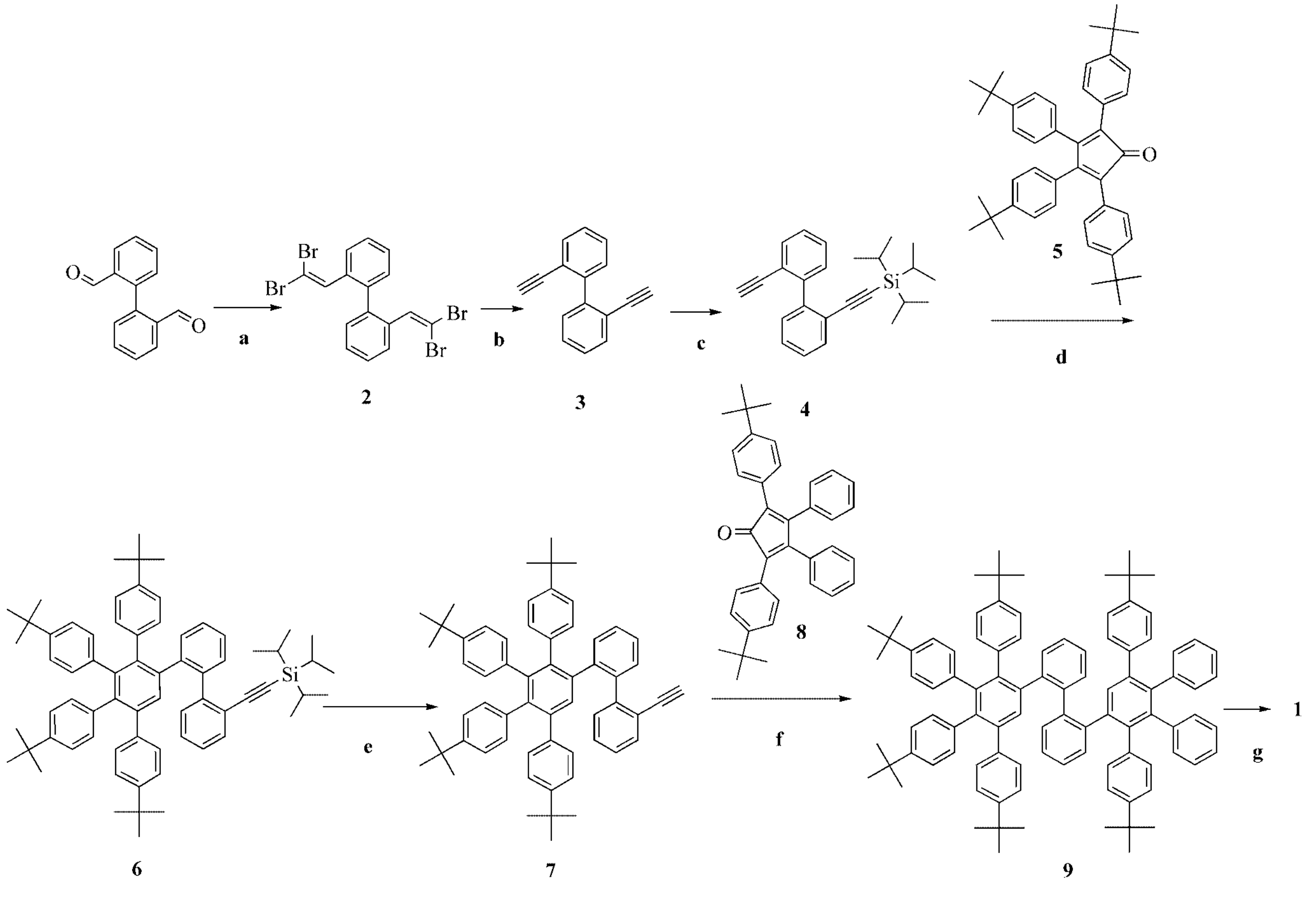
Figure 2. Superacene 2D lander with polyaromatic main board and *tert*-butyl spacers.

Results and Discussion

The synthesis of **1**, outlined in Scheme 1, uses the synthetic concept reported by Müllen et al. for the preparation of extremely extended PAH.[13] The key step of their synthetic routes is the intramolecular oxidative cyclodehydrogenation of suitable oligophenylene precursors (in our case **9**) under modified Kovacic[14] conditions (Scheme 1, Step g). Similar polyaromatic "superacenes", unsubstituted or with 8 *tert*-butyl or dodecyl substituents, have previously been described.[12] In the case of **1**, some supplementary difficulties arise: First, the molecule is partially asymmetric which increases the number of steps of the synthesis. Indeed, the elegant new route proposed by Mullen et al.[12c] which requires a double Diels–Alder reaction on a thermally unstable biscyclopentadienone cannot be extrapolated to asymmetric systems. Second, as the connecting part of the molecule must be left unsubstituted, the cyclodehydrogenation conditions cannot be too harsh to prevent scrambling of the *tert*-butyl groups around the polyaromatic core. Finally, the reduced number of bulky substituents increases the compound's insolubility.

The synthesis requires the preparation of known 2,2′-diethynylbiphenyl (**3**) which was described so far by tedious and low yield routes. For instance, the preparation from phenanthrenequinone requires 5 steps with a total yield of 8.5%.[15] This is why we have devised an alternative route for its preparation from biphenyl-2,2′-dicarbaldehyde which can be obtained in nearly quantitative yield by Swern oxidation of commercial (2′-hydroxymethyl-biphenyl-2-yl)-methanol.[16] Attempts to directly prepare alkyne **3** by a one-pot Wittig-type condensation with dibromomethyltriphenylphosphonium bromide in the presence of potassium *tert*-butoxide[17] was unsuccessful and gave only low yields of the target product; therefore, the Corey–Fuchs conver-



Scheme 1. Route to **1**: (a) CBr_4, PPh_3, Zn, CH_2Cl_2 (98%); (b) LDA, THF (78%); (c) i) *n*BuLi, THF ii) TIPSCl (57%); (d) Diphenyl ether, microwave irradiation (70%); (e) TBAF/THF, CH_2Cl_2 (84%); (f) Diphenyl ether (60%); (g) $FeCl_3$, CH_3NO_2, CH_2Cl_2, argon bubbling (61%).

sion was preferred. This reaction gave 2,2′-bis(2,2-dibromoethenyl)biphenyl **2** in nearly quantitative yield. The second step of the sequence afforded diethynyl **3** in 78% yield; the total yield over the 3-step procedure is now 69% from the commercial product. The next reaction is the protection of one of the ethynyl groups with the bulky triisopropylsilyl group which is known to prevent Diels–Alder addition. The silylation was carried out by metalation with *n*-butyllithium followed by the reaction with triisopropylsilyl chloride to give the monoprotected adduct in 57% yield. Then, Diels–Alder addition under microwave irradiation with activated diene **5**, with subsequent in situ decarbonylation and aromatization, furnished protected alkyne **6** in 70% yield. The remaining ethynyl group was then deprotected by moist tetrabutylammonium fluoride in THF to obtain free alkyne **7** in 84% yield. The second Diels–Alder reaction furnishing polyphenyl **9** (microwave irradiation, 60% yield) required the preparation of 2,5-bis(4-*tert*-butylphenyl)-3,4-diphenylcyclopenta-2,4-dien-1-one **8**, which was made by condensation of benzil with 1,3-bis(4-*tert*-butylphenyl)-2-propenone (85% yield). The final step is the cyclodehydrogenation of **9**, with the creation of 12 C–C bonds and the corresponding removal of 24 hydrogen atoms. It was carried out by iron chloride in a 1:1 mixture of dry dichloromethane and nitromethane. This final step proved difficult because of the very low solubility of the reaction intermediates and the risks of chlorination of the PAH plateau. The reaction was therefore carried out at small concentrations, with a large excess of iron chloride, and a constant and efficient flow of argon was bubbled through the mixture during the 5 d reaction. As final product **1** is significantly less soluble than the reaction byproducts, namely mono- and dichlorinated adducts and incompletely cyclodehydrogenated intermediates, the purification was done by successive washing/centrifugation steps. The final purity of the product was confirmed by comparison of the experimental and calculated DCI mass spectra, and the absence of peaks at $m/z > 1231$ (Figure 3). Indeed, traces of these more soluble and more easily desorbed impurities are immediately apparent in the mass spectra when present. Because of dynamic aggregation of this compound in solution, the proton NMR spectra showed overlapping broad resonances which prevents the use of this technique to attest the compound's purity.

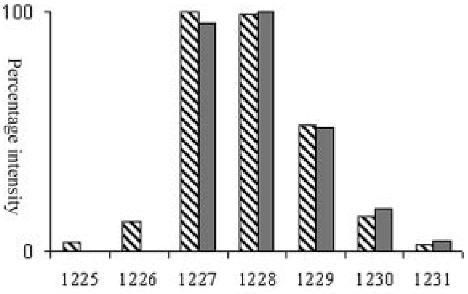


Figure 3. Comparison of the experimental (dashed bars) and calculated (grey bars) DCI mass spectrum (parent ion $[M]^+$) of compound **1**.

Finally, although the cyclodehydrogenation conditions have been maintained as mild as possible, one cannot exclude some scrambling of the *tert*-butyl group during this last step. Although there is not absolute proof, the weakness of the mass spectrum peaks $[M - C_4H_9]^+$ (<5% of the maximum intensity, similar to what was observed in the same DCI MS conditions for fully substituted octa-*tert*-butyl analogue of **1** and corresponding to fragmentation of the molecular parent ion) indicates that the mobility of the *tert*-butyl groups is not too high in this last step of the experimental sequence.

Work in progress is engaged toward UHV STM studies of the electronic and nanopatterning properties of this compound.

Experimental Section

General:. ^{1}H- and ^{13}C NMR spectra were recorded with a Bruker WF-250 in $CDCl_3$ or CD_2Cl_2 solutions at 20 °C and internal standard ($CDCl_3$ at δH: 7.25 ppm, δC: 77.0 ppm, CD_2Cl_2 at δH: 5.30 ppm, δC: 53.8 ppm). Mass Spectroscopy was performed with a Nermag R10-R10 (DCI). Elemental analyses were done by the Service d'Analyse de l'ICSN (Paris) and by the Service d'Analyse du LCC (Toulouse). 2,3,4,5-Tetrakis(4-*tert*-butylphenyl)cyclopenta-2,4-dien-1-one (**5**) was prepared according to the literature.[11] THF was distilled from Na/benzophenone. Dichloromethane was distilled from calcium hydride and nitromethane was dried with molecular sieves. Other solvents and reagents were used as obtained in the best quality available. The microwave heating was carried out in closed vials with a CEM-Discover monomode microwave apparatus at 300 watts under the conditions (temperature, time) given here. After completion of the reaction, the vessel was cooled down rapidly to 60 °C

Compound 2,2′-Bis(2,2-dibromoethenyl)biphenyl (2): A mixture of CBr_4 (10 g, 30.15 mmol), PPh_3 (7.909 g, 30.15 mmol), and Zn dust (1.972 g, 30.15 mmol) in dry dichloromethane (100 mL) was stirred at room temp. for 24 h. The suspension was then cooled to 0 °C and biphenyl-2,2′-dicarbaldehyde (600 mg, 2.85 mmol) in dry dichloromethane (20 mL) was added. The suspension was then stirred for 24 h at room temp. under an atmosphere of argon. The suspension was then filtered and the precipitate was washed with dichloromethane (25 mL). After evaporation of the combined filtrates, the residue was dissolved in dichloromethane (30 mL) and then hexane was added to precipitate the remaining P-containing products. The suspension was filtered through celite and the residue washed with hexane (2 × 75 mL). The filtrates were evaporated and the oily residue was chromatographed on silica gel with the use of dichloromethane/petroleum ether (1:1) as the eluent. Yield: 1.481 g (98%). ^{1}H NMR (250 MHz, CD_2Cl_2): $\delta = 7.04$ (s, 2 H, vinyl), 7.20–7.26 (m, 2 H, arom), 7.40–7.48 (m, 4 H, arom), 7.71–7.78 (m, 2 H, arom) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): $\delta = 140.18$, 137.06, 135.68, 130.90, 129.58, 129.17, 128.52, 92.14 ppm. MS (DCI, NH_3) $m/z = 539.8$ $[M + NH_4]^+$. $C_{16}H_{10}Br_4$ (521.87): calcd. C 36.82, H 1.93; found C 36.5, H 1.7.

Compound 2,2′-Diethynylbiphenyl (3): LDA (1.8 M in THF, 3.4 mL) was slowly added to a solution of **2** (400 mg, 0.77 mmol) in THF (30 mL) at –78 °C. After one hour stirring at this temperature, the mixture was left to warm up to room temp, and the stirring was then continued for another 2 h. The reaction was then quenched by the addition of a saturated NH_4Cl solution (20 mL), and the mixture was diluted with hexane (100 mL). The organic phase was

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then washed with water (2 × 50 mL), dried with magnesium sulfate, filtered, and the solvents evaporated. Preparative chromatography over silica gel with the use of dichloromethane/hexane (1:2) as the eluent gave 120 mg of **3** (yield: 78%). ^{1}H- and ^{13}C NMR, MS, melting point, and chemical analysis are consistent with those presented in ref.[15]

[(2′-Ethynylbiphenyl-2-yl)ethynyl](triisopropyl)silane (4): *n*BuLi (2.5 M in hexane, 0.68 mL,1.36 mmol) was added to a stirred solution of 2,2′-diethynylbiphenyl **3** (250 mg, 1.23 mmol) in THF at –78 °C. The reaction mixture was then stirred at this temperature for 2 h. Triisopropylsilyl chloride (0.4 mL, 1.85 mmol) was then added, and the solution was warmed up to room temp. and stirred for another 2 h. The reaction was quenched by the dropwise addition of water, and the solvent was removed under vacuum. The residue was then extracted with dichloromethane. The organic layer was washed with water, dried with $MgSO_4$, and the solvents evaporated. The residue was purified by flash chromatography (5% dichloromethane in petroleum ether) to afford the pure product. Yield: 252 mg, 57%. ^{1}H NMR (250 MHz, $CDCl_3$): δ = 0.95 (s, 21 H, TIPS), 2.99 (s, 1 H, CC-H), 7.33–7.39 (m, 6 H, arom), 7.54–7.59 (m, 2 H, arom) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 11.14, 18.50, 79.95, 82.74, 93.94, 105.68, 121.54, 122.99, 127.16, 127.28, 127.72, 128.22, 129.75, 130.13, 132.70, 132.92, 143.21, 143.65 ppm. $C_{25}H_{30}Si$ (358.59): calcd. C 83.74, H 8.43; found C 84.1, H 8.2. MS (DCI, NH_3): *m*/*z* = 376 [M + NH_4]$^+$.

Compound 6: A mixture of **4** (50 mg, 0.14 mmol) and tetrakis(4-*tert*-butylphenyl)cyclopenta-2,4-dien-1-one (**5**) (84.9 mg, 0.14 mmol) in diphenyl ether (1.5 g) was stirred at 160 °C under microwave irradiation for 45 min. The cooled reaction mixture was then poured in stirred methanol (50 mL). The precipitated white product was filtered, washed with methanol, and recrystallized from 10% dichloromethane in methanol. Yield: 92 mg, 70%. ^{1}H NMR (250 MHz, CD_2Cl_2): δ = 0.84–0.87 (m, 21 H, TIPS), 1.09 (s, 9 H, *t*Bu), 1.12 (s, 9 H, *t*Bu), 1.16 (s, 9 H, *t*Bu), 1.21 (s, 9 H, *t*Bu), 6.72 (d, 4 H, *J* = 8.5 Hz, arom), 6.96–7.12 (m, 10 H, arom), 7.14–7.36 (m, 10 H, arom), 7.61–7.66 (m, 1 H, arom) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 11.14, 18.47, 31.17, 34.06, 94.13, 106.46, 122.77, 123.18, 123.84, 125.83, 126.15, 126.37, 127.34, 129.41, 129.78, 130.47, 131.10, 131.82, 132.12, 132.38, 137.14, 137.80, 138.58, 138.80, 139.33, 140.05, 142.06, 147.72, 147.88 ppm. $C_{69}H_{82}Si$ (939.47): calcd. C 88.21, H 8.80; found C 88.11, H 8.80. MS (DCI, NH_3): *m*/*z* = 956 [M + NH_4]$^+$.

Compound 7: Tetrabutylammonium fluoride [1 M solution in THF (5% water), 0.5 mmol] was added to a solution of **6** (200 mg, 0.212 mmol) in CH_2Cl_2 (15 mL), heated under reflux for 2 h, cooled, diluted with CH_2Cl_2 (10 mL), and washed with water (3 × 20 mL). The organic layer was dried with $MgSO_4$ and the solvents evaporated. The residue was purified by flash chromatography (silica gel, 5% CH_2Cl_2 in petroleum ether) to afford the pure product. Yield: 140 mg, 84%. ^{1}H NMR (250 MHz, CD_2Cl_2): δ = 1.04 (s, 9 H, *t*Bu), 1.08 (s, 9 H, *t*Bu), 1.13 (s, 9 H, *t*Bu), 1.24 (s, 9 H, *t*Bu), 3.9 (s, 1 H, C2-H), 6.41–6.50 (m, 2 H, arom), 6.64–6.96 (m, 10 H, arom), 6.97–7.44 (m, 11 H, arom), 7.46–7.54 (m, 2 H, arom) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 31.66, 34.76, 80.83, 84.09, 122.79, 123.69, 124.16, 124.91, 126.82, 127.26, 127.57, 128.51, 130.17, 131.40, 131.74, 132.77, 133.37, 133.74, 137.47, 138.19, 138.47, 139;69, 140.21, 141.60, 142.66, 148.45, 148.72, 149.08, 149.52 ppm. $C_{60}H_{62}$ (783.13): calcd. C 92.02, H 7.98; found C 91.8, H 8.2. MS (DCI, NH_3): *m*/*z* = 800 [M + NH_4]$^+$.

2,5-Bis(4-*tert*-butylphenyl)-3,4-diphenylcyclopenta-2,4-dien-1-one (8): To a boiling solution of benzil (0.911 g, 4.34 mmol) and 1,3-bis(4-*tert*-butylphenyl)-2-propenone (1.4 g, 4.34 mmol) in dry ethanol (5 mL) was added a solution of KOH (2 M in dry ethanol, 2 mL). The solution was heated at reflux for 5 h and then cooled to 0 °C. The dark crystalline product was filtered, washed with cold ethanol (2 × 1 mL), and dried under vacuum to afford the pure product. Yield: 1.8 g, 85%. ^{1}H NMR (250 MHz, CD_2Cl_2): δ = 1.29 (s, 18 H, *t*Bu), 6.94–7.00 (m, 4 H, arom), 7.13–7.21 (m, 6 H, arom), 7.21–7.33 (m, 8 H, arom) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 31.24, 34.56, 124.84, 125.00, 127.75, 128.28, 129.32, 129.66, 133.39, 150.29, 153.92, 201.10 ppm. $C_{37}H_{36}O$ (496.68): calcd. C 89.47, H 7.31; Found C 89.1, H 7.7. MS (DCI, NH_3): *m*/*z* = 514 [M + NH_4]$^+$.

Compound 9: A mixture of **6** (50 mg, 0.0638 mmol) and 2,5-bis(4-*tert*-butylphenyl)-3,4-diphenylcyclopenta-2,4-dien-1-one (**8**) (38.4 mg, 0.052 mmol) in diphenyl ether (1.5 g) was stirred at 160 °C under microwave irradiation (300 w) for 45 min. The reaction mixture was then cooled, diluted in dichloromethane (1 mL), and poured into stirred methanol (50 mL). The solid was filtered and washed with methanol, dried under vacuum, and recrystallized from dichloromethane/ethanol (1:9). Yield: 60%. ^{1}H NMR (250 MHz, CD_2Cl_2): δ = 1.01 (s, 9 H, *t*Bu), 1.08 (s, 9 H, *t*Bu), 1.13 (s, 9 H, *t*Bu), 1.23 (s, 9 H, *t*Bu), 1.53 (s, 18 H, *t*Bu), 6.56–7.20 (m, 40 H, arom), 7.30–7.36 (m, 1 H, arom), 7.44–7.54 (m, 3 H, arom) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 31.18, 34.23, 118.86, 122.70, 123.06, 123.75, 123.89, 124.04, 125.01, 125.30, 125.46, 125.63, 125.95, 126.09, 126.69, 130.17, 130.61, 130.72, 131.06, 131.35, 131.96, 132.39, 132.96, 133.19, 133.82, 133.90, 134.16, 134.53, 136.85, 136.99, 138.00, 138.53, 138.90, 139.49, 139.64, 140.28, 140.36, 141.03, 141.62, 142.93, 147.63, 147.86, 148.11, 148.34, 148.60 ppm. $C_{96}H_{98}$ (1251.81): calcd. C 92.11, H 7.89; found C 91.9, H 7.5. MS (DCI, NH_3): *m*/*z* = 1269 [M + NH_4]$^+$.

Superacene 1: A solution of $FeCl_3$ (607 mg, 3.74 mmol) in dry nitromethane (10 mL) was added dropwise to a solution of **9** (20 mg, 0.0159 mmol) in dry dichloromethane (10 mL) with a strong flow of argon in the solution. The reaction mixture was stirred for 5 d with regular addition of a 1:1 mixture of dry dichloromethane and dry nitromethane to maintain a constant volume. The reaction was quenched with methanol (20 mL), and the solvent was evaporated under vacuum. A few drops of water were added to the deep reddish gummy material. The brownish–yellow solid was suspended and centrifuged successively with methanol and water to give the desired product as a yellow powder. Yield: 30 mg, 61%. MS (DCl, NH_3): *m*/*z* (%) = 1225 (3.7), 1226 (12.4), 1227 (100), 1228 (99.1), 1229 (52.9), 1230 (14.5), 1231 (2.5). $C_{96}H_{74}$ (1227.61): calcd. C 93.92, H 6.08; found C 93.6, H 5.8.

Acknowledgments

This work was supported by the CNRS, the European Community FET-NID projects BUN (Bottom-Up Nanomachines) IST-1999-11565 and CHIC (Consortium for Hamiltonian Intramolecular Computing) IST-2001-33578.

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Received: August 1, 2006
Published Online: October 13, 2006

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