Supporting Information

Liquid-Crystalline Bis-Adducts of [60]Fullerene

Stéphane Campidelli,[†] Ester Vázquez,[‖] Dragana Milic,[†] Julie Lenoble,[§] Carmen Atienza Castellanos,[‡] Ginka Sarova,[‡] Dirk M. Guldi,*[,‡] Robert Deschenaux*[,§] and Maurizio Prato*[,†]

[†]*Dipartimento di Scienze Farmaceutiche, Università degli Studi di Trieste, Piazzale Europa 1, 34127 Trieste, Italy. E-mail: prato@units.it*

[‖]*Departamento de Química Inorgánica, Orgánica y Bioquímica, Facultad de Químicas, Universidad de Castilla-La Mancha, Campus Universitario, 13071 Ciudad Real, Spain.*

[§]*Institut de Chimie, Université de Neuchâtel, Avenue de Bellevaux 51, CP 158, 2009 Neuchâtel, Switzerland. E-mail: robert.deschenaux@unine.ch*

[‡]*Friedrich-Alexander-Universität Erlangen-Nürnberg Universität Erlangen, Institute for Physical and Theoretical Chemistry, Egerlandstrasse 3, 91058 Erlangen, Germany. E-mail: dirk.guldi@chemie.uni-erlangen.de*

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Experimental section

Materials

C_{60} was purchased from Bucky-USA (99.5%). All other reagents and solvents were purchased and used as received. Compounds **12**[1] and **13**[2] were prepared according to literature procedures.

Techniques

Transition temperatures (onset point) and enthalpies were determined with a differential scanning under N_2/He, at a rate of 10°C/min. Optical studies were conducted using a polarizing microscope equipped with a variable-temperature stage, under N_2. For column chromatography, silica gel 60 (0.015-0.040 mm and 0.063-0.200 mm) were used. ^{1}H and ^{13}C NMR spectra were recorded with the solvent as internal reference. Femtosecond transient absorption studies were performed with 387 nm laser pulses (1 kHz, 150 fs pulse width) from an amplified Ti:Sapphire laser system. Nanosecond Laser Flash Photolysis experiments were performed with 355 nm of excitation with 5 ns laser pulse width from Nd:YAG laser. Fluorescence lifetimes were measured with a Laser Strope Fluorescence Lifetime Spectrometer with 337 nm laser pulses from a nitrogen laser fiber-coupled to a lens-based T-formal sample compartment equipped with a stroboscopic detector. Details of the Laser Strobe systems are described on the manufacture's web site. For emission spectra, the experiments were performed at room temperature. Each spectrum represents an average of at least 5 individual scans, and appropriate corrections were applied whenever necessary.

Synthesis

Compound 6. To a solution of the aldehyde derivative[3] (1.00 g, 0.366 mmol) in a mixture of THF (50 mL) and water (20 mL) was added $NaClO_2$ (50 mg, 0.553 mmol) and H_2NSO_3H (53 mg, 0.546 mmol). The reaction mixture was stirred at room temperature for 2h, and water (100 mL) was added. The solution was extracted with CH_2Cl_2 (3 × 100 mL), dried with anhydrous $MgSO_4$ and then the solvent was removed. Purification of the residue by precipitation (dissolution in CH_2Cl_2 and precipitation by pouring the solution into methanol) gave pure **6** (1.00 g, quantitative yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.93 (t, 1H, arom. H), 8.64 (t, 2H, arom. H), 8.36 (d, 2H, arom. H), 8.18-8.13 (m, 14H, arom. H), 8.11 (d, 4H, arom. H), 7.77-7.61 (m, 24H, arom. H), 7.32 (d, 8H, arom. H), 6.99 (d, 2H, arom. H), 6.97 (d, 8H, arom. H), 4.37 (t, 10H, CO_2CH_2), 4.03 (t, 10H, CH_2O), 1.85-1.76 (m, 20H, $CO_2CH_2CH_2$ and CH_2CH_2O), 1.47-1.26 (m, 60H, aliph. H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 166.15, 165.32, 165.25, 164.86, 164.50, 164.09, 163.49, 152.08, 151.97, 150.94, 145.25, 137.10, 135.34, 133.24, 133.06, 133.01, 132.96, 132.75, 131.51, 130.51, 130.00, 129.49, 128.85, 128.75, 128.08, 127.43, 122.97, 121.59, 120.70, 119.29, 114.95, 114.76, 111.39, 68.82, 68.74, 66.31, 66.05, 29.88, 29.83, 29.73, 29.64, 29.49, 29.04, 26.36. IR-DRIFT (KBr): ν (cm^{-1}) 3081, 2928, 2852, 2225, 1730, 1604, 1509, 1253, 1067, 1005, 847, 762, 533, 426. Anal. calcd. for $C_{169}H_{166}N_4O_{31}$ (2749.18): C, 73.84, H, 6.09, N, 2.04%; found C, 73.72, H, 6.07, N, 2.15%.

Compound 7. To a solution of **6** (100 mg, 0.036 mmol) in dry CH_2Cl_2 (5 mL), was added $SOCl_2$ (150 mg, 1.300 mmol). The reaction mixture was stirred under reflux for 5 h, then the solvent and the excess of thionyl chloride were removed (quantitative yield). Compound **7** was dried under high vacuum and used directly in the next step.

Compound 1. To a solution of **8b** (15 mg, 0.016 mmol) and Et_3N (100 μl) in CH_2Cl_2 (3 mL), was added a solution of freshly prepared **7** (45 mg, 0.016 mmol) in CH_2Cl_2 (3 mL). The reaction was stirred for 16 h, and the solvent was removed. Purification of the crude material by column chromatography (silica gel, 63-200 μm, toluene/ethyl acetate 9:1 to 7:3) and precipitation of the product from CH_2Cl_2 with MeOH gave pure **1** (11 mg, 19%). ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) 8.95 (t, 1H, arom. H), 8.66 (t, 2H, arom. H), 8.38 (d, 2H, arom. H), 8.24-8.05 (m, 16H, arom. H), 8.01 (d, 2H, arom. H), 7.82-7.58 (m, 24H, arom. H), 7.46 (br s, 1H, NH), 7.39-7.27 (m, 8H, arom. H), 7.00 (d, 10H, arom. H), 4.53 (s, 4H, pyrrolidine), 4.45-4.27 (m, 10H, CO_2CH_2), 4.13-3.98 (m, 12H, CH_2O and C*H*$_2$NH), 3.45 (t, 2H, NCH_2), 1.92-1.70 (m, 20H, $CO_2CH_2CH_2$ and C*H*$_2CH_2O$), 1.58-1.26 (m, 60H, aliph. H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 165.52, 164.73, 164.68, 163.97, 163.55, 162.92, 154.40, 151.46, 150.41, 146.18, 145.99, 145.80, 145.38, 145.19, 144.73, 144.41, 142.56, 142.04, 141.93, 141.77, 140.14, 138.22, 136.58, 135.92, 133.08, 132.54, 132.24, 131.01, 129.75, 128.97, 128.23, 127.58, 126.93, 122.47, 121.12, 120.52, 118.78, 114.50, 114.28, 110.92, 70.51 68.30, 65.84, 65.47, 51.84, 38.05, 29.42, 29.32, 29.22, 29.08, 28.64, 25.96. IR-DRIFT (KBr): ν (cm^{-1}) 3077, 2928, 2851, 2225, 1730, 1604, 1508, 1252, 1065, 1005, 843, 762, 527, 426. UV-Vis (CH_2Cl_2): λ_{max} (nm) 274, 431, 703.

Compound 4. To a solution of **11b** (19 mg, 0.017 mmol) and Et_3N (300 μl) in dry CH_2Cl_2 (3 mL) at 0°C, was added a solution of freshly prepared **7** (100 mg, 0.036 mmol) in dry CH_2Cl_2 (4 mL). The reaction was stirred for 1 h at room temperature, and the solvent was removed. Purification of the crude material by column chromatography (silica gel, 63-200 μm, toluene/ethyl acetate 15:1 to 8:2) and precipitation of the product from CH_2Cl_2 with MeOH gave pure **4** (18 mg, 17%). ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) 8.95 (t, 2H, arom. H), 8.65 (t, 4H, arom. H), 8.37 (d, 4H, arom. H), 8.23-8.10 (m, 32H, arom. H), 8.04-7.87 (m, 6H, arom. H and

NH), 7.80-7.59 (m, 48H, arom. H), 7.40-7.26 (m, 16H, arom. H), 7.00 (d, 20H, arom. H), 4.37 (t, 20H, CO_2CH_2), 4.32-3.94 (series of m, 28H, CH_2O and pyrrolidine), 3.95-3.80 (m, 4H, C*H*$_2$NH), 3.32-3.15 (m, 4H, NCH_2), 1.92-1.72 (m, 40H, $CO_2CH_2CH_2$ and C*H*$_2$CH$_2$O), 1.56-1.24 (m, 120H, aliph. H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 166.33, 164.76, 164.70, 164.32, 163.99, 163.58, 162.96, 158.82, 151.48, 150.44, 148.03, 147.67, 147.14, 146.61, 145.18, 144.75, 144.57, 144.34, 143.04, 142.14, 141.66, 140.52, 138.89, 136.59, 132.57, 132.26, 131.63, 131.05, 129.75, 129.62, 128.99, 128.25, 127.61, 126.94, 122.48, 121.14, 120.24, 118.80, 114.51, 114.31, 110.95, 69.68, 68.32, 65.86, 54.65, 29.72, 29.48, 29.44, 29.34, 29.25, 29.10, 28.67, 25.99. IR-DRIFT (KBr): ν (cm^{-1}) 30713, 2931, 2852, 2225, 1733, 1603, 1508, 1251, 1065, 1005, 846, 761, 529, 435. UV-Vis (CH_2Cl_2): λ_{max} (nm) 277, 421, 552.

Compound 5. To a solution of **6** (96 mg, 0.035 mmol), HOBt (9 mg, 0.070mmol) and EDC (13 mg, 0.070 mmol) in CH_2Cl_2 (3 mL), was added after 15 min a solution of **13**[2] (20 mg, 0.017 mmol) and Et_3N (300 μl) in CH_2Cl_2 (2 mL). The reaction was stirred at room temperature 3h, and the solvent was removed. Purification of the crude material by column chromatography (silica gel, 63-200 μm, toluene/ethyl acetate 10:0.5 to 10:1) and precipitation of the product from CH_2Cl_2 with MeOH gave pure **3** (17 mg, 15%). ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) 8.91 (t, 2H, arom. H), 8.61 (t, 4H, arom. H), 8.33 (d, 4H, arom. H), 8.18-8.01 (m, 32H, arom. H), 7.85 (d, 4H, arom. H), 7.78-7.54 (m, 48H, arom. H), 7.37-7.21 (m, 16H, arom. H), 7.03-6.89 (m, 22H, arom. H and NH), 4.44-4.21 (m, 24H, CO_2CH_2 and pyrrolidine), 4.18-3.90 (m 28H, CH_2O, pyrrolidine and ethylene glycol), 3.84-3.59 (m, 16H, ethylene glycol), 3.28-3.15 (m, 4H, ethylene glycol), 1.87-1.67 (m, 40H, $CO_2CH_2CH_2$ and C*H*$_2$CH$_2$O), 1.54-1.17 (m, 120H, aliph. H). ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 166.60, 165.89, 164.88, 164.83, 164.46, 164.08, 163.67, 163.07, 158.79, 155.55, 154.87, 151.67, 151.57, 150.53, 148.98, 146.53, 145.28, 145.12, 144.86,

143.93, 143.59, 141.54, 141.23, 140.98, 139.72, 138.52, 138.71, 136.35, 135.49, 132.99, 132.68, 132.63, 132.37, 131.14, 129.77, 129.15, 128.38, 127.72, 127.24, 127.07, 122.60, 121.22, 120.32, 118.93, 114.59, 114.40, 111.03, 97.21, 70.60, 70.42, 70.02, 69.81, 68.42, 65.98, 65.60, 54.41, 40.13, 29.84, 29.60, 29.56, 29.46, 29.37, 29.21, 28.76, 26.11. IR-DRIFT (KBr): ν (cm^{-1}) 3052, 2927, 2860, 2225, 1727, 1602, 1500, 1252, 1064, 998, 843, 756, 533, 433. UV-Vis (CH_2Cl_2): λ_{max} (nm) 277, 462.

Spectroscopic data for 9a-11a and 9b-11b

9a (trans-2): (49.4 mg, 3.6%). ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) 5.43-5.25 (br. s, 2H, NH), 4.68 (d, 2H, pyrrolidine), 4.50 (d, 2H, pyrrolidine), 4.37 (d, 2H, pyrrolidine), 4.34 (d, 2H, pyrrolidine), 3.80-3.60 (m, 4H, C*H*$_2$NHBoc), 3.27 (t, 4H, NCH_2), 1.51 (s, 18H, Boc). ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 158. 87, 156.16, 153.24, 153.04, 152,45, 148.44, 147.74, 147.13, 147.09, 146.48, 146.30, 146.12, 145.70, 145.60, 145.38, 145.21, 144.26, 143.86, 143.72, 142.64, 142.60, 142.55, 142.44, 141.60, 141.51, 139.64, 134.55, 133.80, 79.67, 69.43, 69.26, 67.93, 67.77, 54.27, 39.40, 28.66. IR-DRIFT (KBr): ν (cm^{-1}) 3345, 2971, 2786, 1710, 1514, 1168, 771, 525. UV-Vis (CH_2Cl_2): λ_{max} (nm) 284, 478, 627, 660, 722, 775. ES-MS: *m/z* 1093 (MH^+).

10a (trans-3): (79.8 mg, 5.8%). ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) 5.30-5.10 (br. s, 2H, NH), 4.43 (d, 2H, pyrrolidine), 4.35 (d, 2H, pyrrolidine), 4.18 (d, 2H, pyrrolidine), 4.09 (d, 2H, pyrrolidine), 3.70-3.53 (m, 4H, C*H*$_2$NHBoc), 3.15 (t, 4H, NCH_2), 1.47 (s, 18H, Boc). ^{13}C NMR (50 MHz, $CDCl_3$): δ (ppm) 158. 18, 156.09, 155.54, 154.87, 149,13, 148.94, 148.83, 148.26, 146.66, 145.36, 145.23, 144.90, 144.66, 143.68, 142.60, 141.64, 141.51, 141.32, 141.07, 139.82,

136.46, 135.59, 79.62, 70.02, 69.78, 67.89, 67.02, 54.20, 39.05, 28.61. IR-DRIFT (KBr): ν (cm^{-1}) 3346, 2973, 2781, 1692, 1516, 1181, 794, 472. UV-Vis (CH_2Cl_2): λ_{max} (nm) 285, 467, 704, 743, 775. ES-MS: *m/z* 1093 (MH^+).

11a (equatorial): (42.9, 3.1%). ^1H NMR (200 MHz, $CDCl_3$): δ (ppm) 5.21-5.8 (br. s, 2H, NH), 4.09 (d, 2H, pyrrolidine), 4.07 (s, 2H, pyrrolidine), 3.97 (d, 2H, pyrrolidine), 3.93 (s, 2H, pyrrolidine), 3.62-3.43 (m, 4H, C*H*$_2$NHBoc), 3.10-2.95 (m, 4H, NCH_2), 1.47 (s, 9H, Boc), 1.44 (s, 9H, Boc). ^{13}C NMR (50 MHz, $CDCl_3$) δ (ppm): 158. 93, 156.02, 153.53, 152.92, 152,59, 149,81, 148.91, 148.08, 147.78, 147.25, 147.21, 146.67, 146.65, 145.77, 145.21, 145.04, 144.67, 144.42, 143.75, 143.23, 142.29, 141.82, 141.65, 141.51, 140.69, 139.16, 136.80, 135.58, 79.58, 69.87, 69.67, 69.54, 67.72, 67.31, 66.81, 54.25, 54.04, 39.18, 28.62, 28.59. IR-DRIFT (KBr): ν (cm^{-1}) 3336, 2970, 2790, 1703, 1515, 1169, 771, 462. UV-Vis (CH_2Cl_2): λ_{max} (nm) 283, 317, 423, 704, 744, 775. ES-MS: *m/z* 1093 (MH^+).

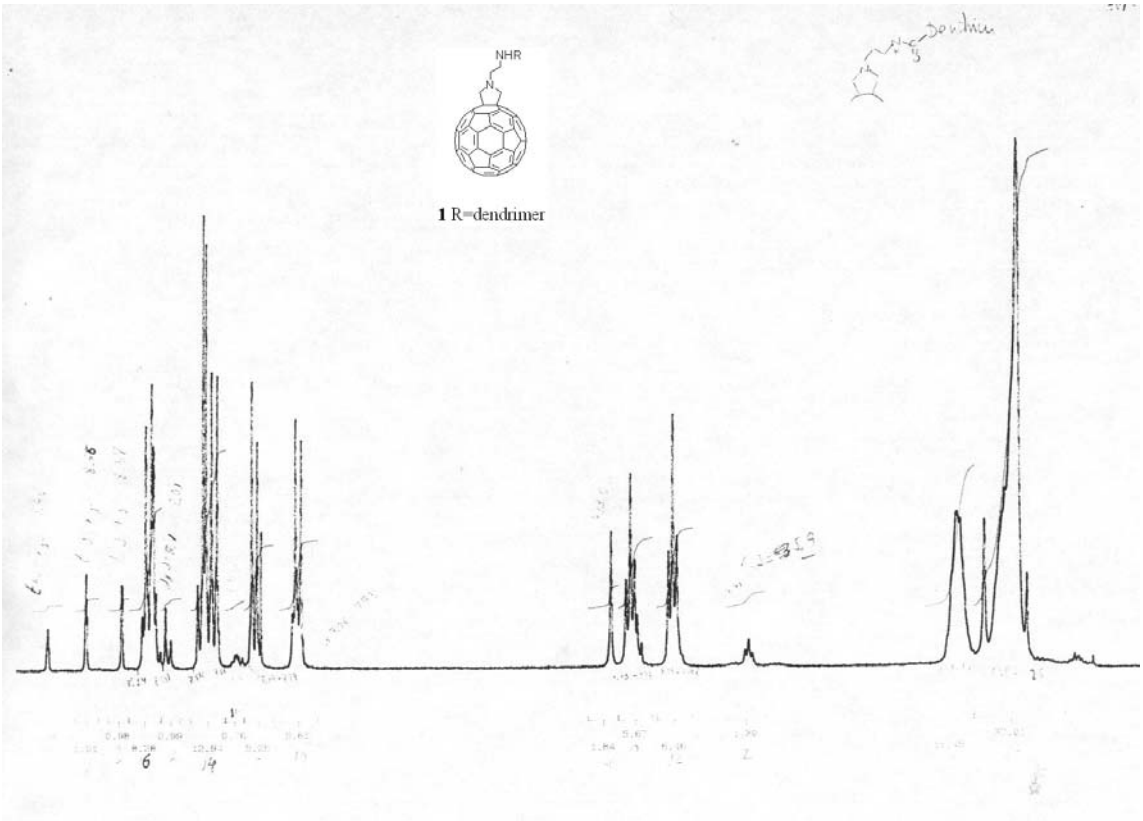
9b: from **9a** (20 mg, 0.018 mmol), TFA (1.5 mL), CH_2Cl_2 (1.5 mL); **9b** (21 mg, quantitative yield). IR-DRIFT (KBr): ν (cm^{-1}) 3330-2850, 1679, 1528, 1195, 1132, 727. UV-Vis (H_2O): λ_{max} (nm) 208, 244, 260, 298, 428, 473, 624, 651, 684, 718. ES-MS: *m/z* 894 (MH^+), 447 (MH_2^{2+}/2).

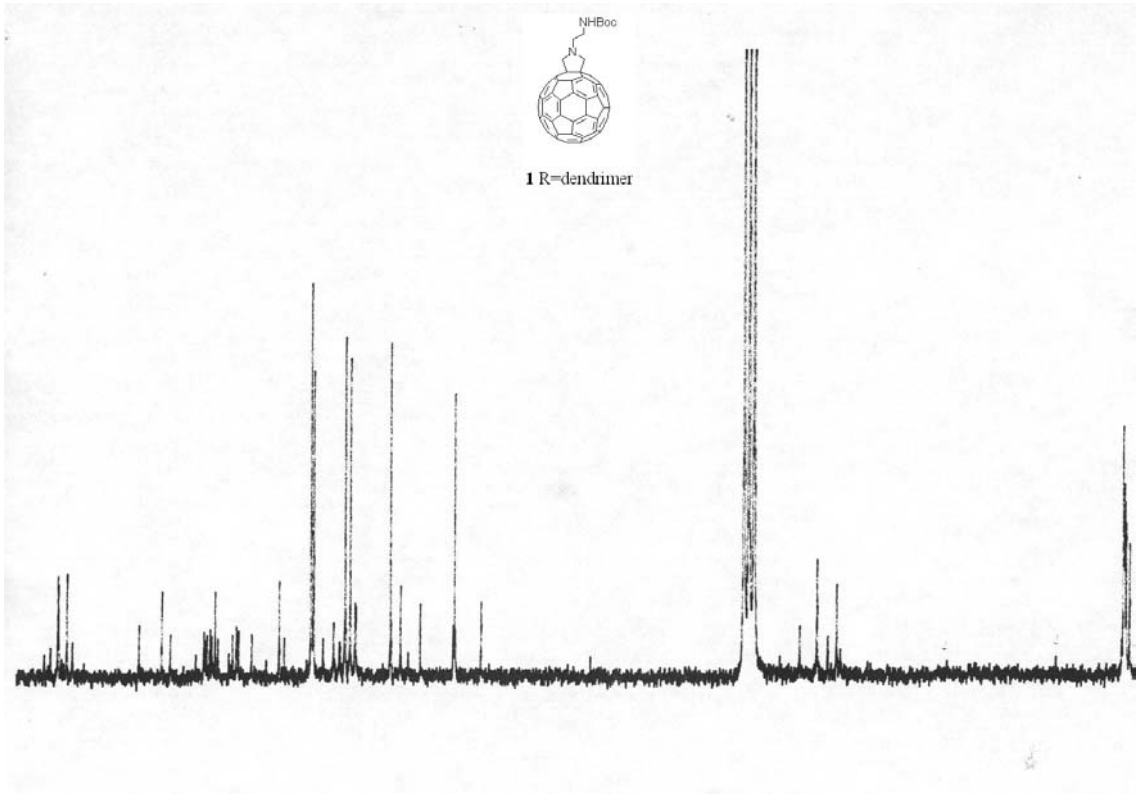
10b: from **10a** (43 mg, 0.039 mmol), TFA (3 mL), CH_2Cl_2 (3 mL); **10b** (43 mg, quantitative yield). IR-DRIFT (KBr): ν (cm^{-1}) 3640-2792, 1677, 1528, 1195, 1053. UV-Vis (H_2O): λ_{max} (nm) 244, 412, 460, 492. ES-MS: *m/z* 894 (MH^+), 447 (MH_2^{2+}/2).

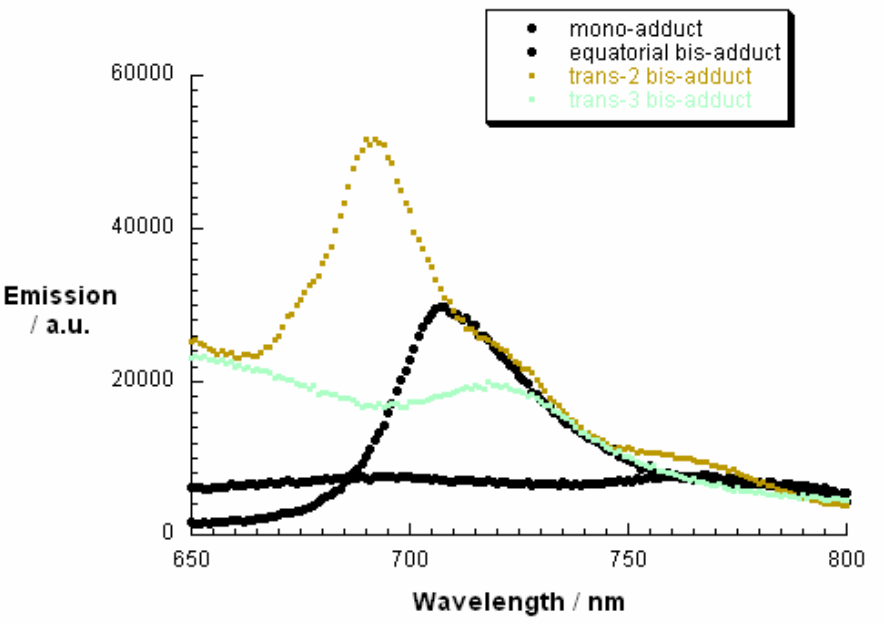
11b: from **11a** (42 mg, 0.038 mmol), TFA (3 mL), CH_2Cl_2 (3 mL); **11b** (41 mg, quantitative yield). IR-DRIFT (KBr): ν (cm^{-1}) 3640-2780, 1684, 1528, 1196, 1133. UV-Vis (H_2O): λ_{max} (nm) 239, 316, 420. ES-MS: *m/z* 894 (MH^+), 447 (MH_2^{2+}/2).

Analysis

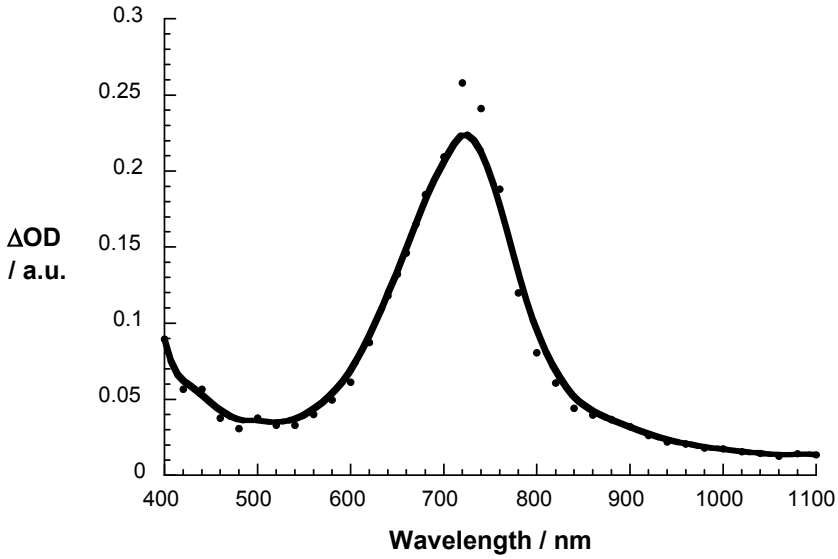
Compound 1



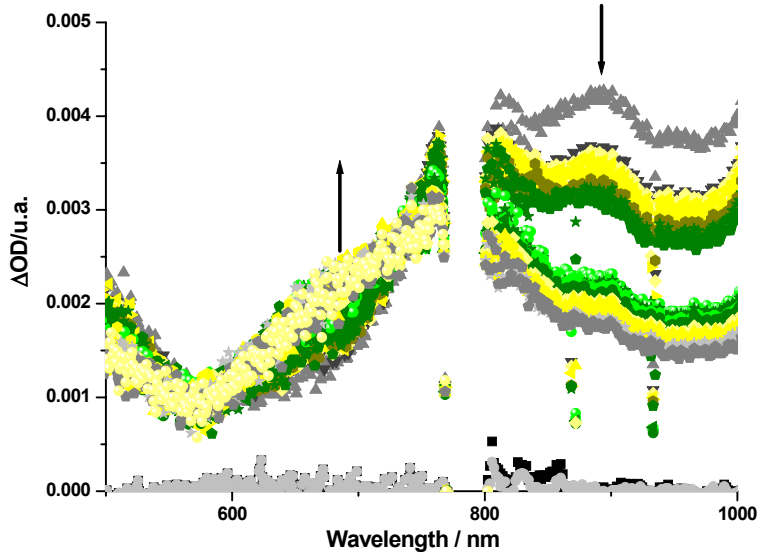




Fluorescence spectra of mono-adduct **1**, trans-2 **2**, trans-3 **3** and equatorial **4** bis-adducts.

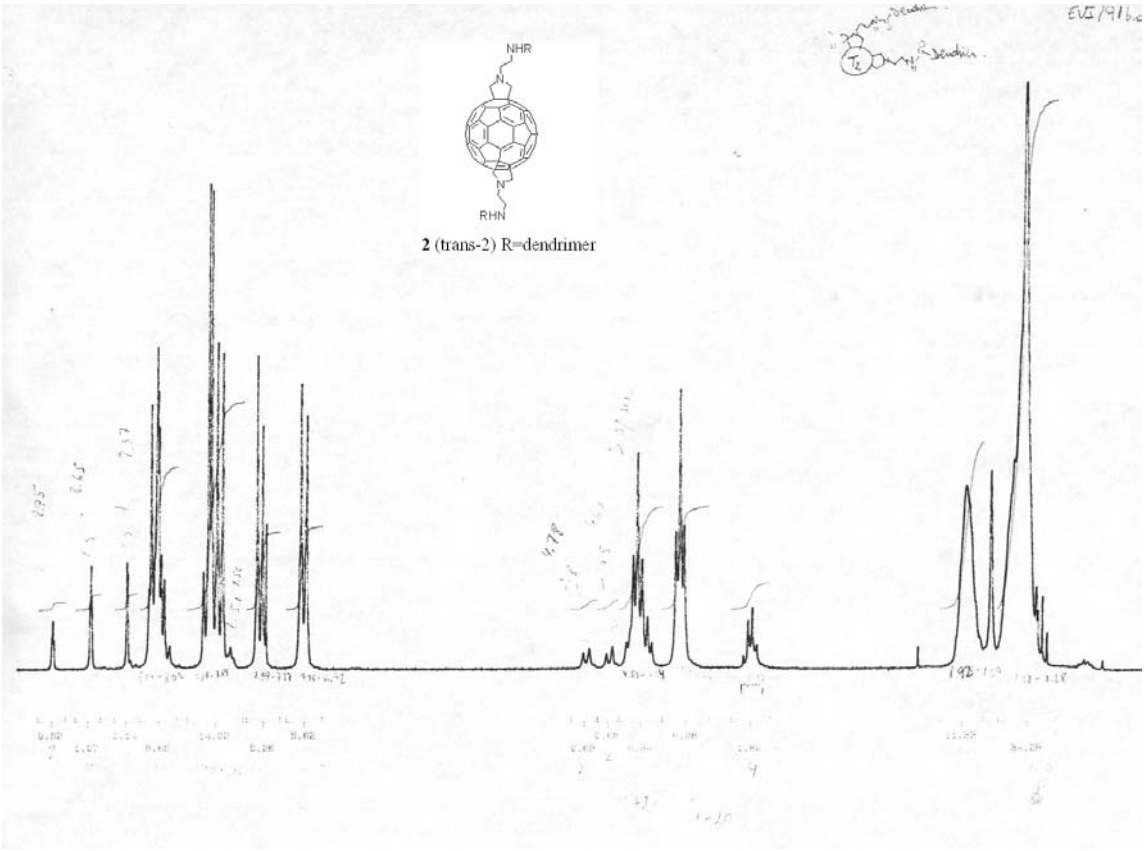


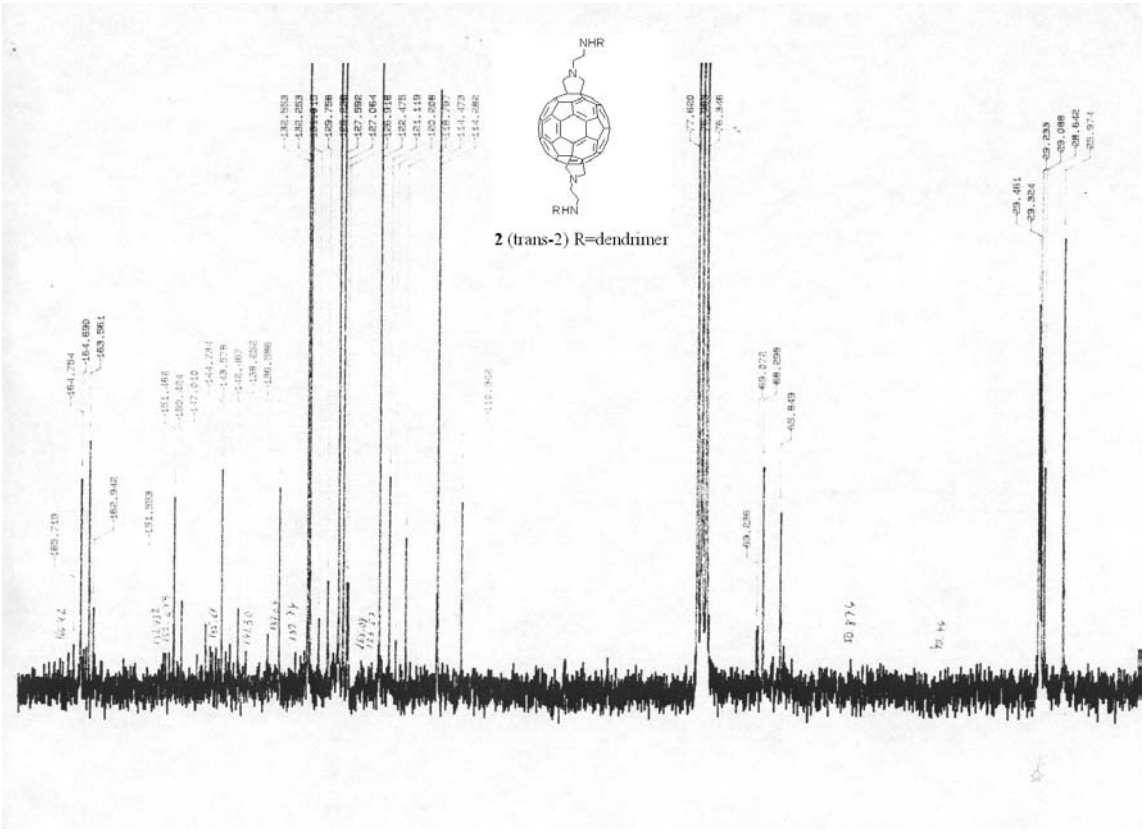
Transient absorption spectrum of the triplet excited state of **1**.

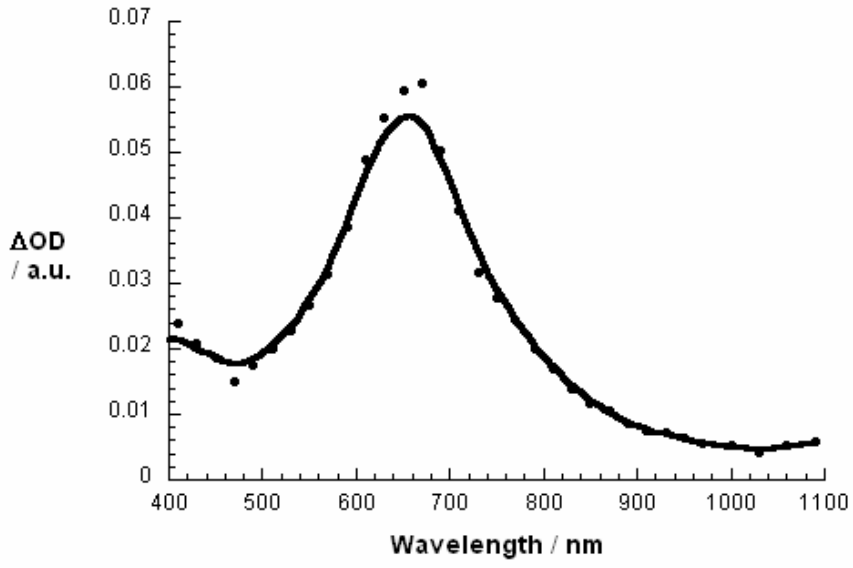


Transient absorption spectrum of **1** (i.e. visible-near-infrared part) obtained upon femtosecond flash photolysis at 387 nm in deoxygenated CH_2Cl_2.

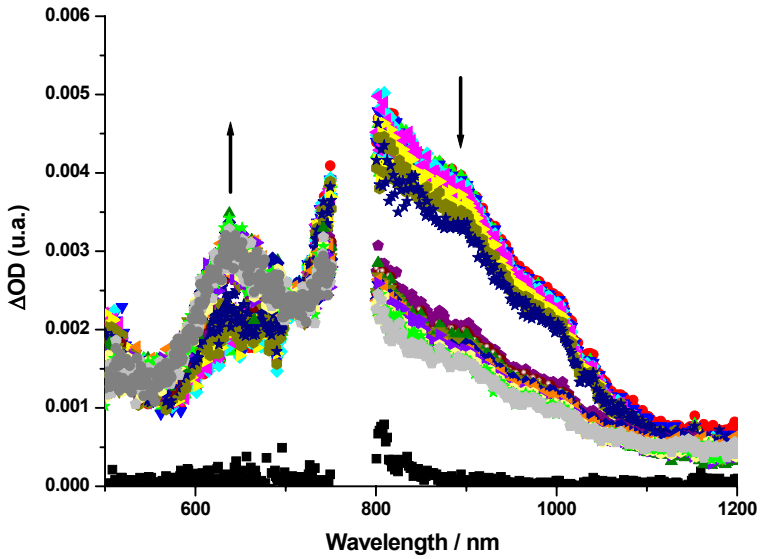
Compound 2





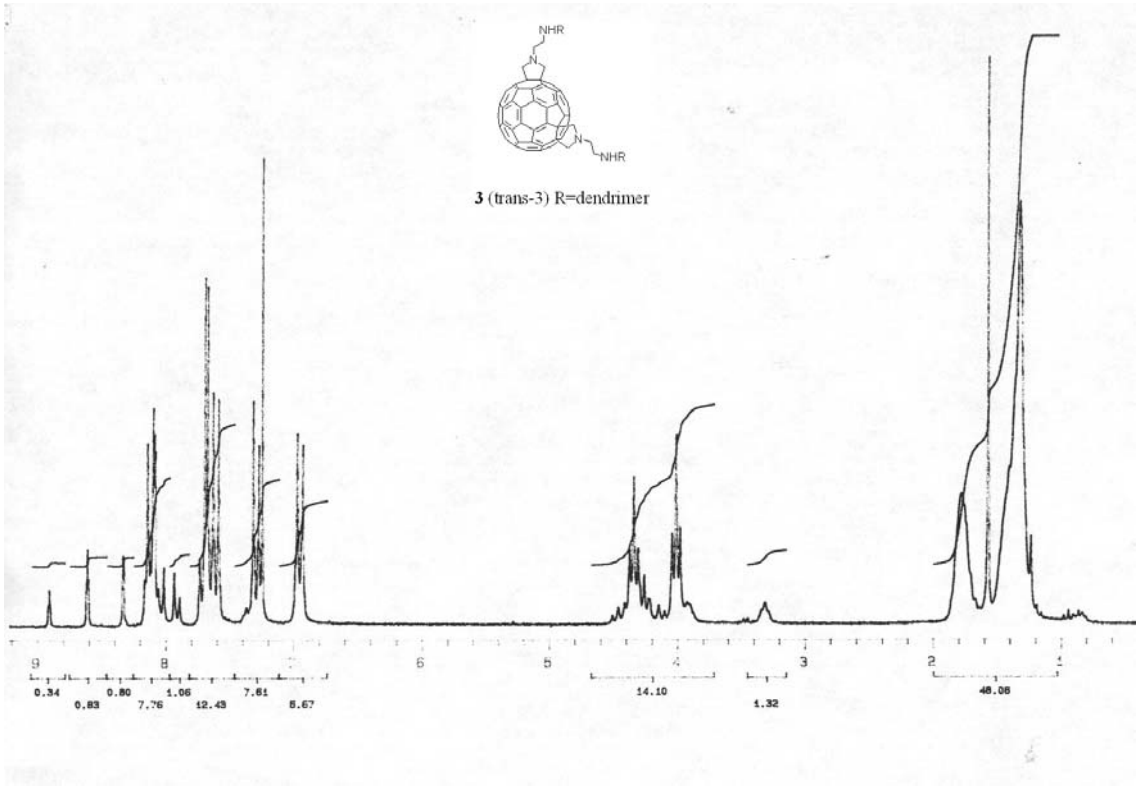


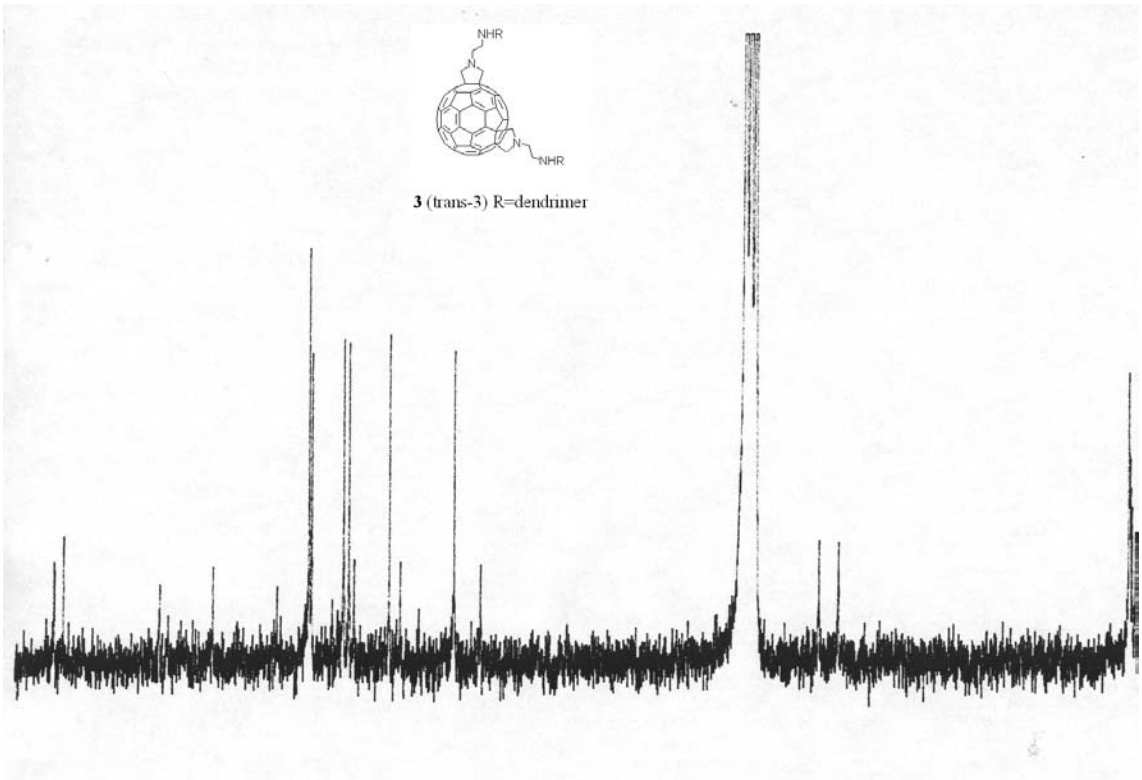
Transient absorption spectrum of the triplet excited state of **2**.



Transient absorption spectrum of **2** (i.e. visible-near-infrared part) obtained upon femtosecond flash photolysis at 387 nm in deoxygenated CH_2Cl_2.

Compound 3


NHR
N
N
NHR
3 (trans-3) R=dendrimer
9
8
7
6
5
4
3
2
1
0.34
0.83
0.80
7.76
1.06
12.43
7.61
8.67
14.10
1.32
48.08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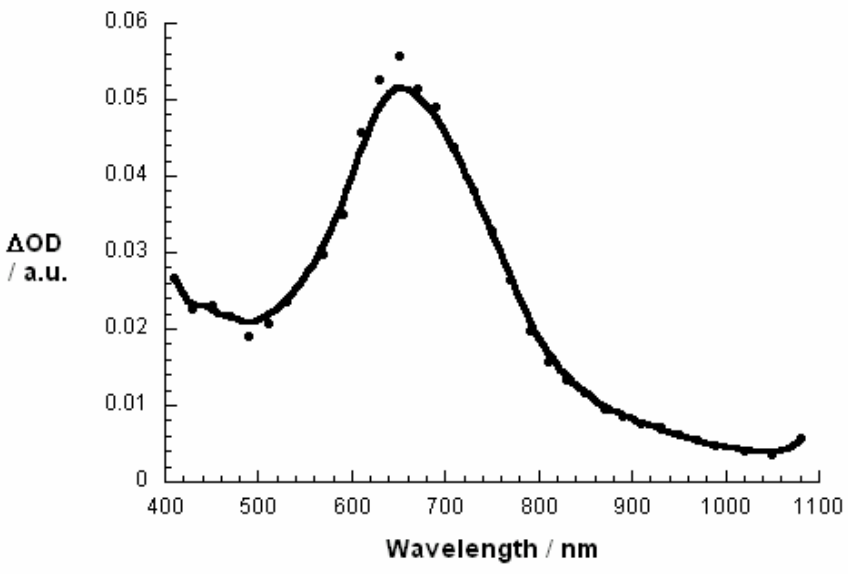
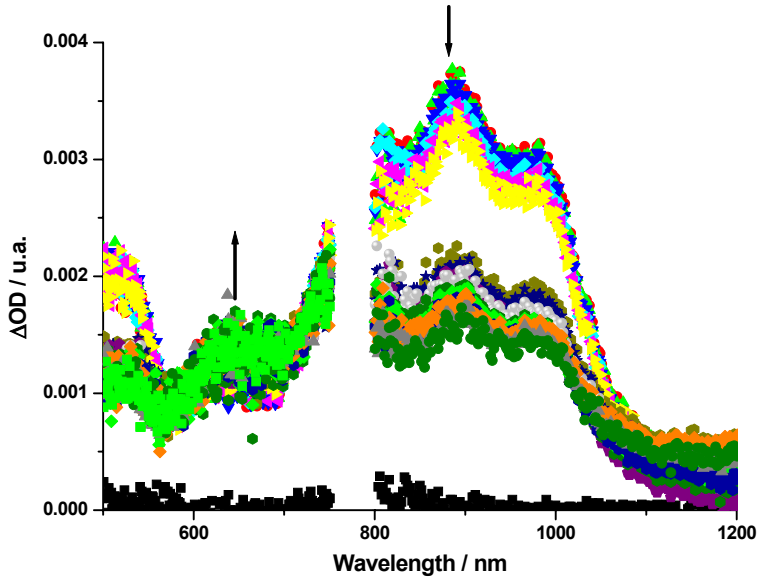
N
N
NHR
3 (trans-3) R=dendrimer

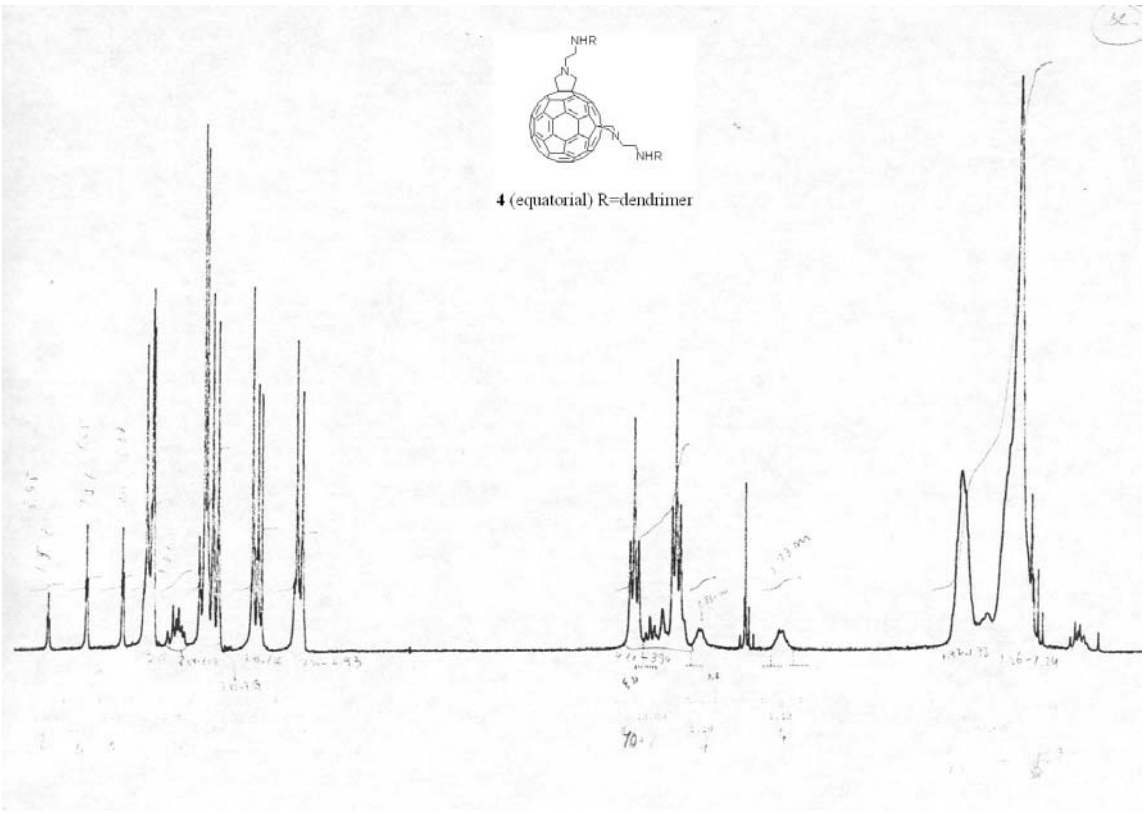


Transient absorption spectrum of the triplet excited state of **3**.

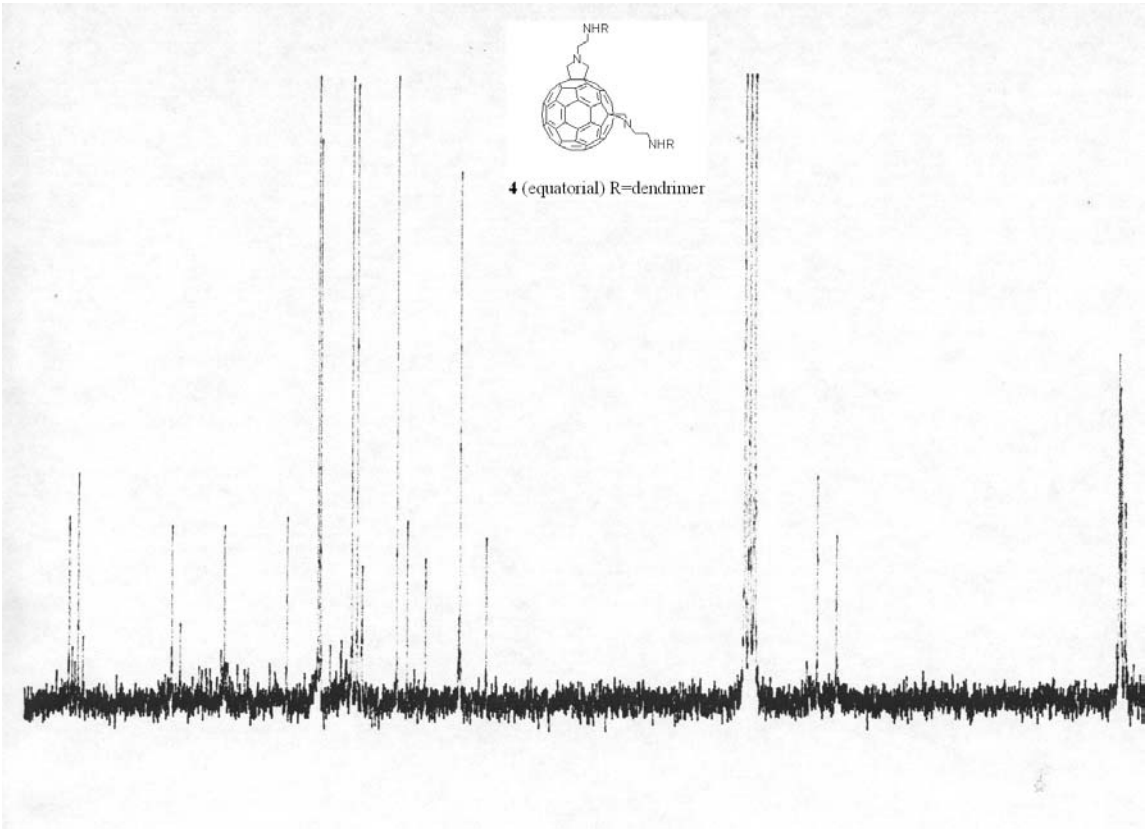


Transient absorption spectrum of **3** (i.e. visible-near-infrared part) obtained upon femtosecond flash photolysis at 387 nm in deoxygenated CH_2Cl_2.

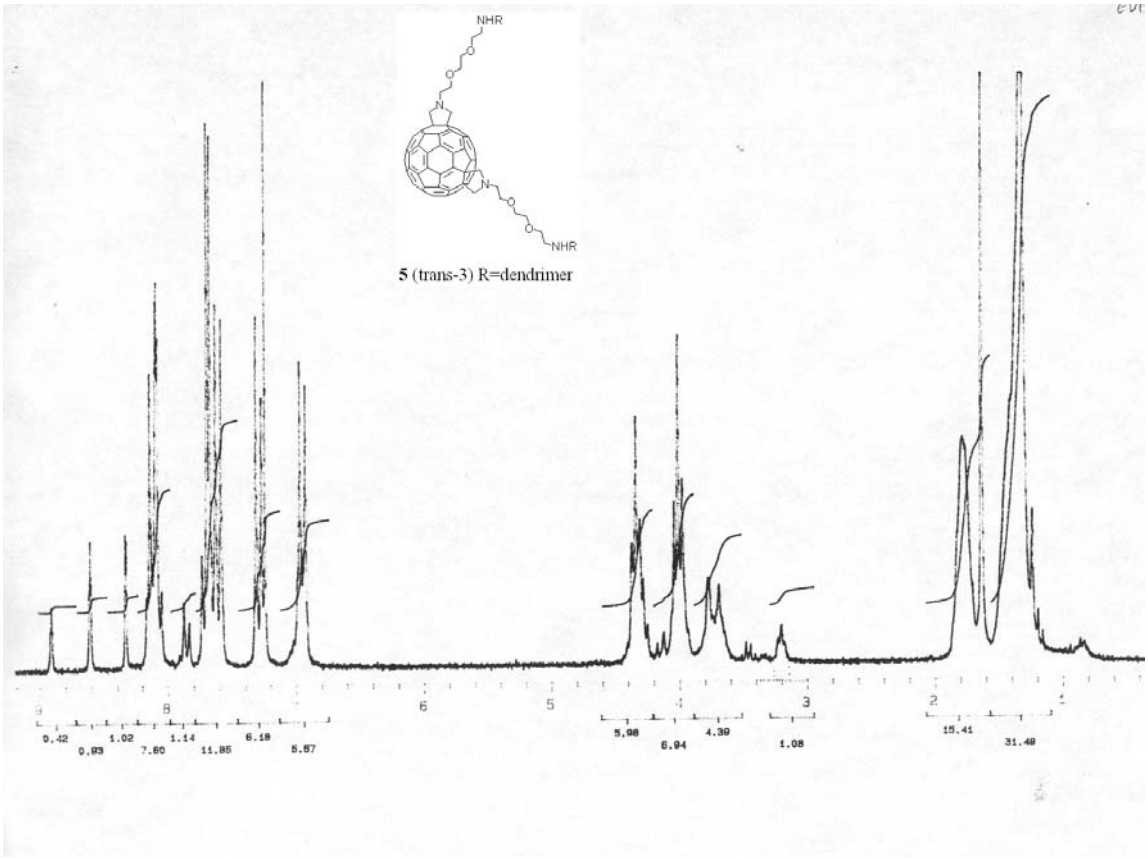
Compound 4



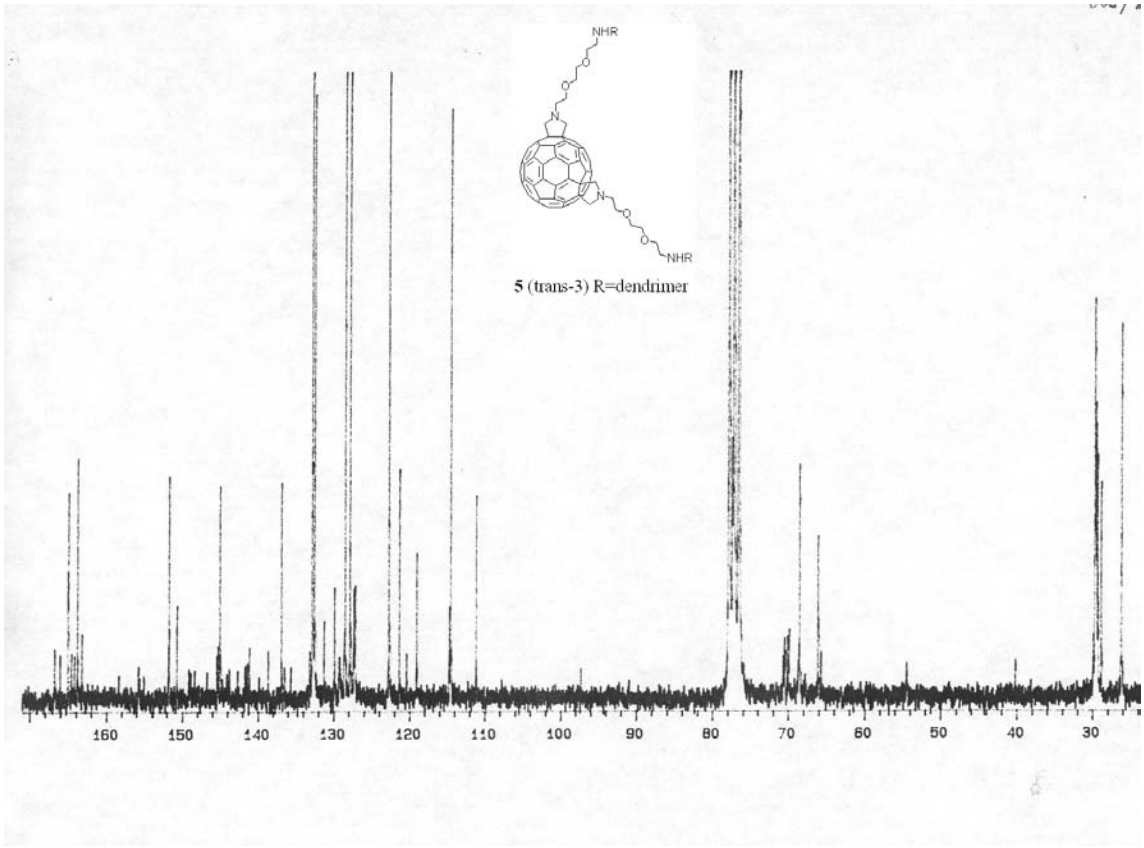
4 (equatorial) R=dendrimer



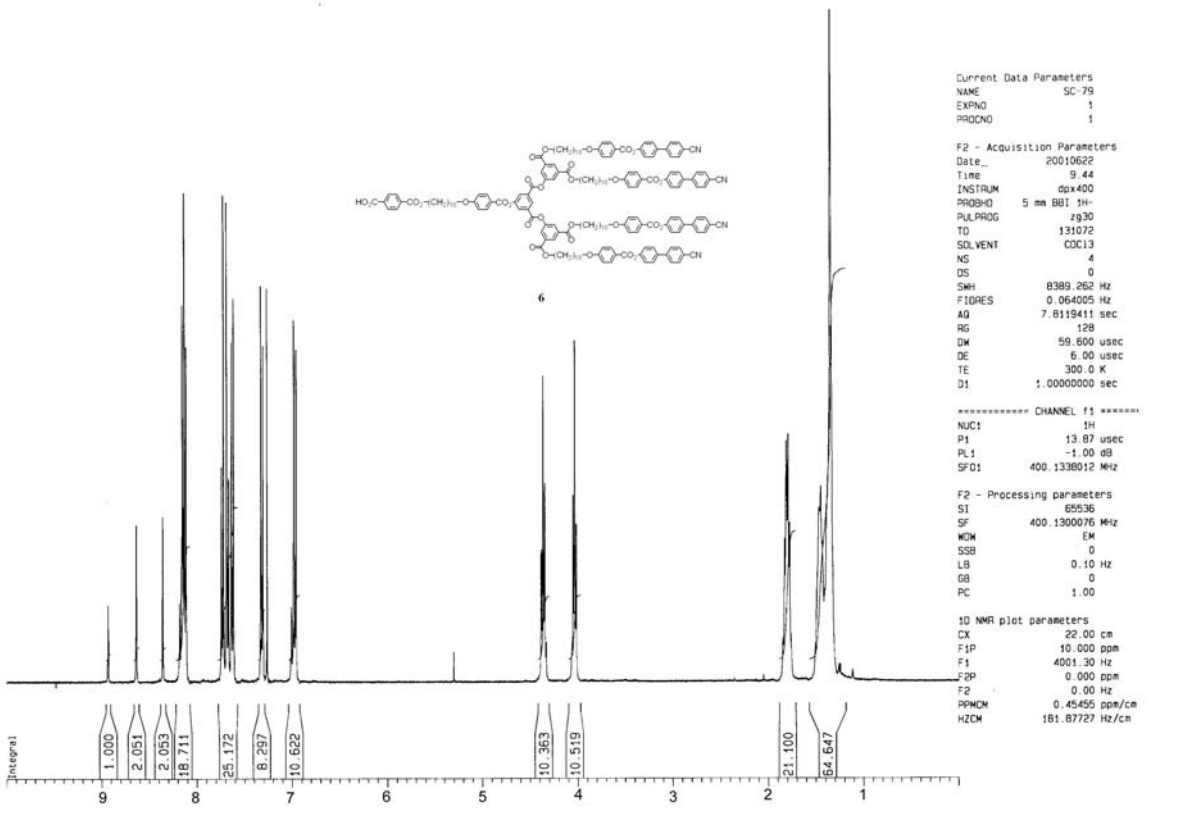
Compound 5

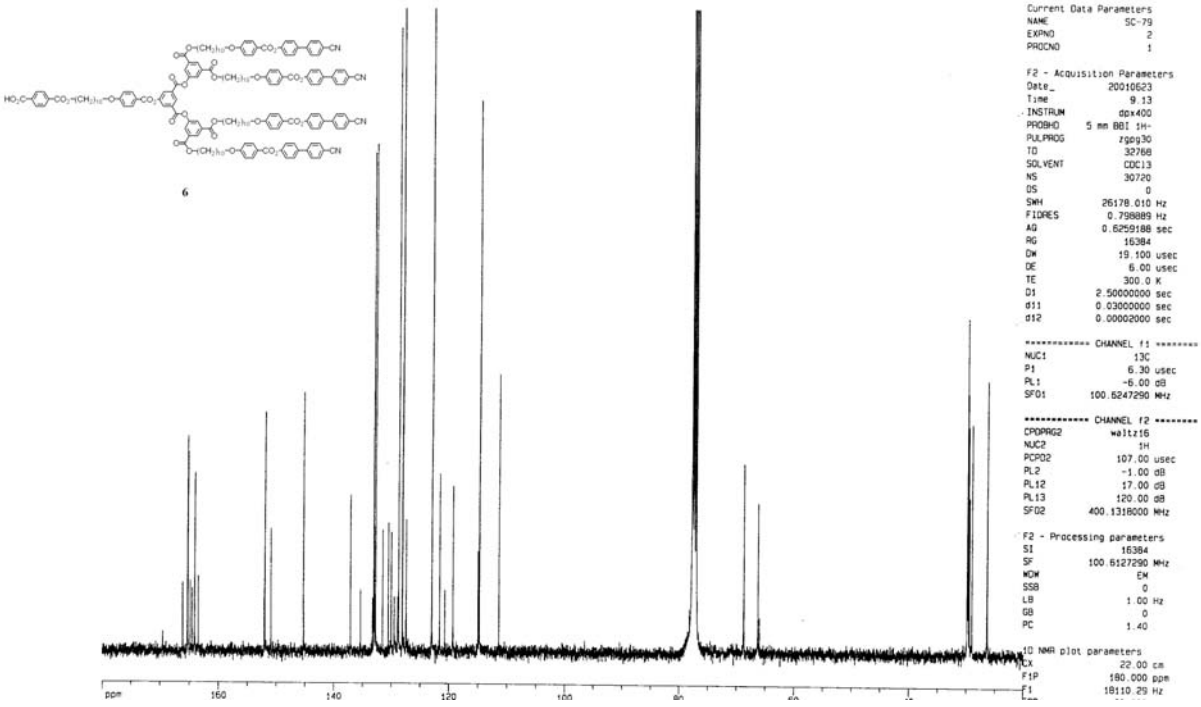


5 (trans-3) R=dendrimer

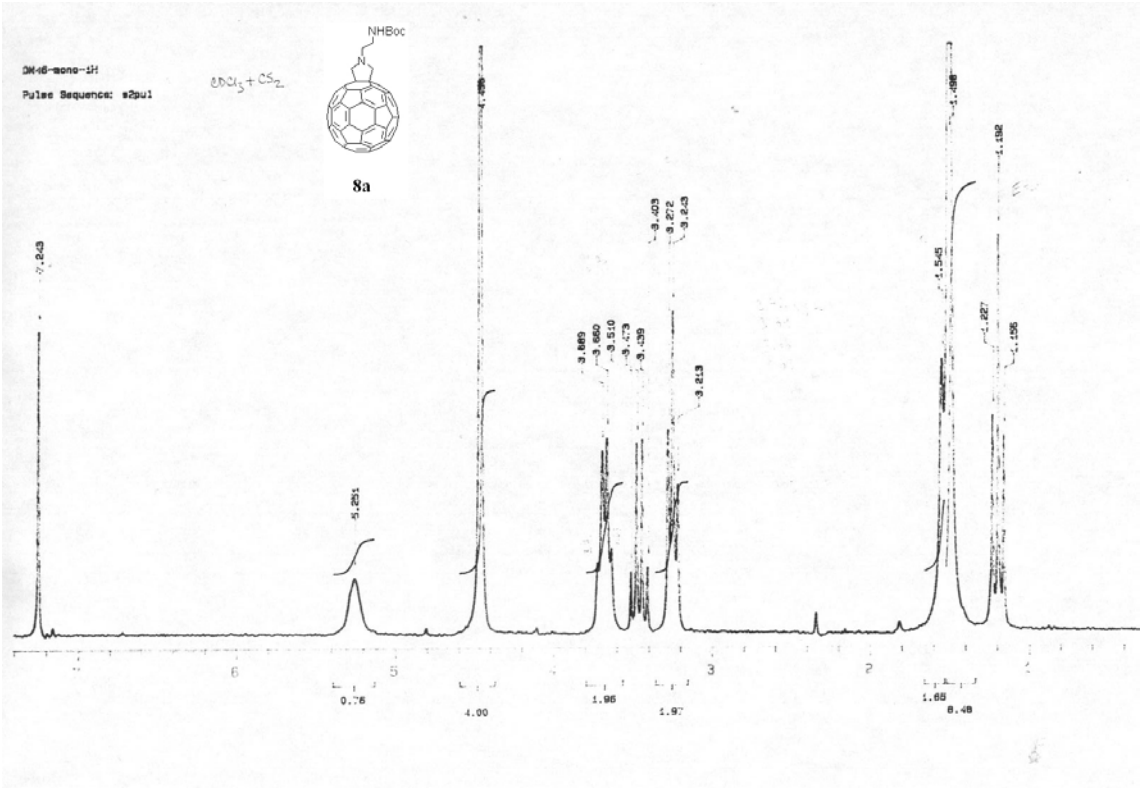

NHR
NHR
5 (trans-3) R=dendrimer
160
150
140
130
120
110
100
90
80
70
60
50
40
30

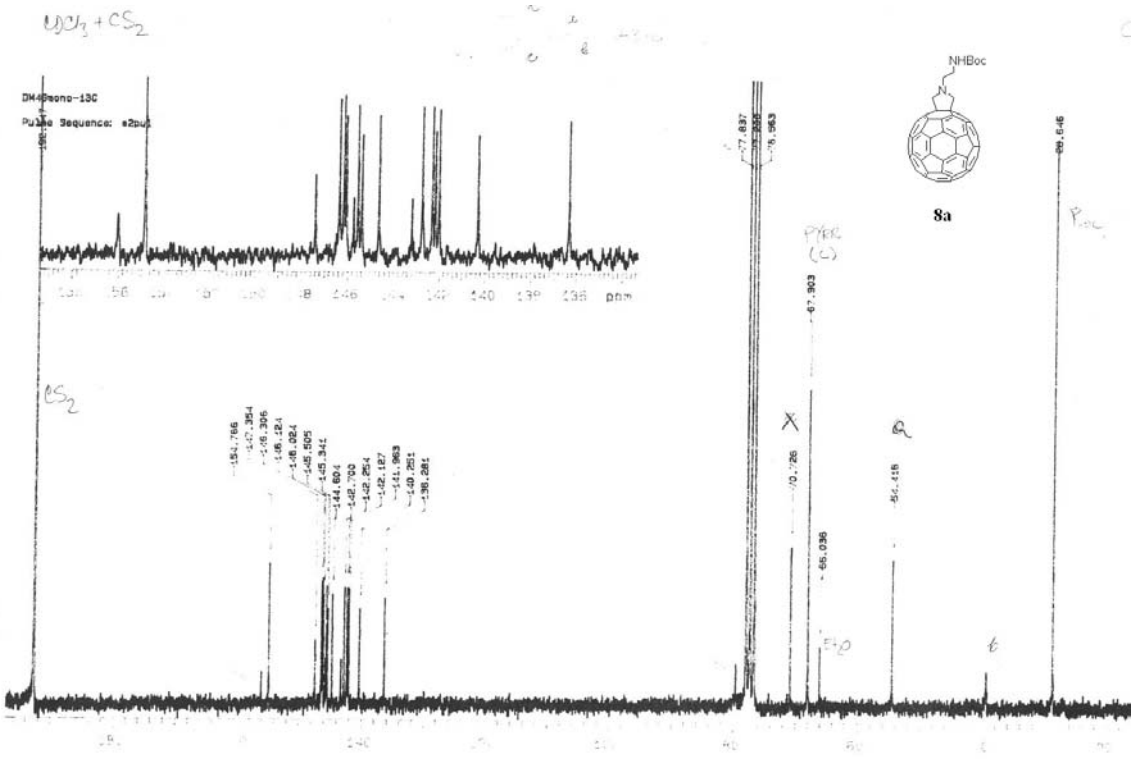
Compound 6

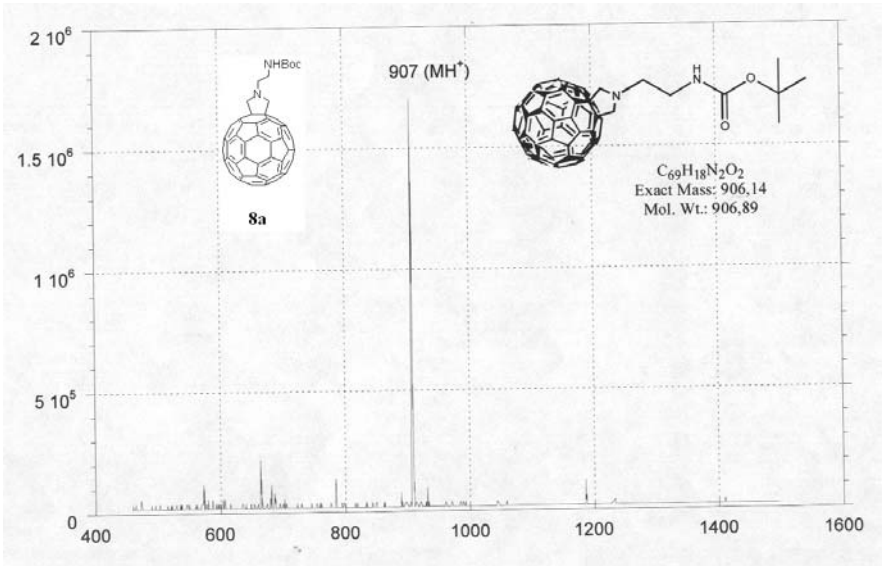

6
Integral
1.000
2.051
2.053
18.711
25.172
8.297
10.622
10.363
10.519
21.100
64.647
9
8
7
6
5
4
3
2
1
Current Data Parameters
NAME SC-79
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010622
Time 9.44
INSTRUM dpx400
PROBHD 5 mm BBI 1H-
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 4
DS 0
SWH 8389.262 Hz
FIDRES 0.064005 Hz
AQ 7.8119411 sec
RG 128
DW 59.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.87 usec
PL1 -1.00 dB
SFO1 400.1338012 MHz
F2 - Processing parameters
SI 65536
SF 400.1300076 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.45455 ppm/cm
HZCM 181.87727 Hz/cm


6
ppm
160
140
120
Current Data Parameters
NAME SC-79
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010623
Time 9.13
INSTRUM dpx400
PROBHD 5 mm BBI 1H-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 30720
DS 0
SWH 26178.010 Hz
FIDRES 0.798889 Hz
AQ 0.6259188 sec
RG 16384
DW 19.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.50000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 6.30 usec
PL1 -6.00 dB
SFO1 100.6247290 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 107.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 120.00 dB
SFO2 400.1318000 MHz
F2 - Processing parameters
SI 16384
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
F1P 180.000 ppm
F1 18110.29 Hz

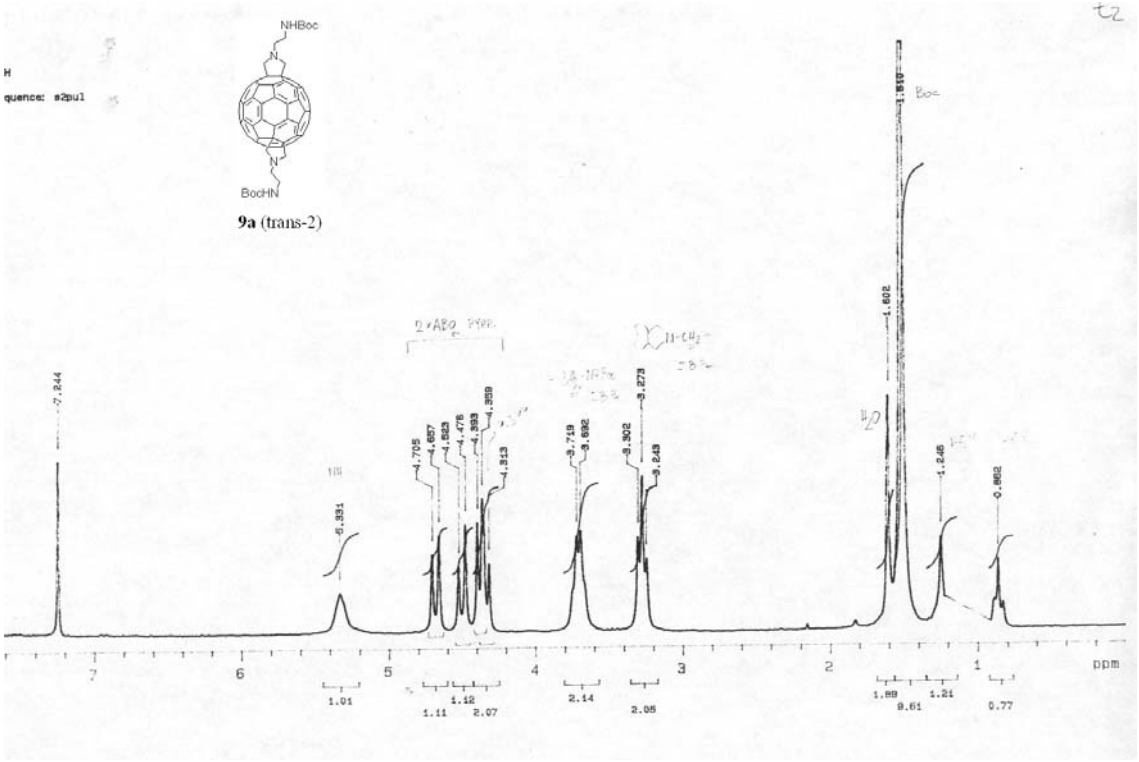
Compound 8a

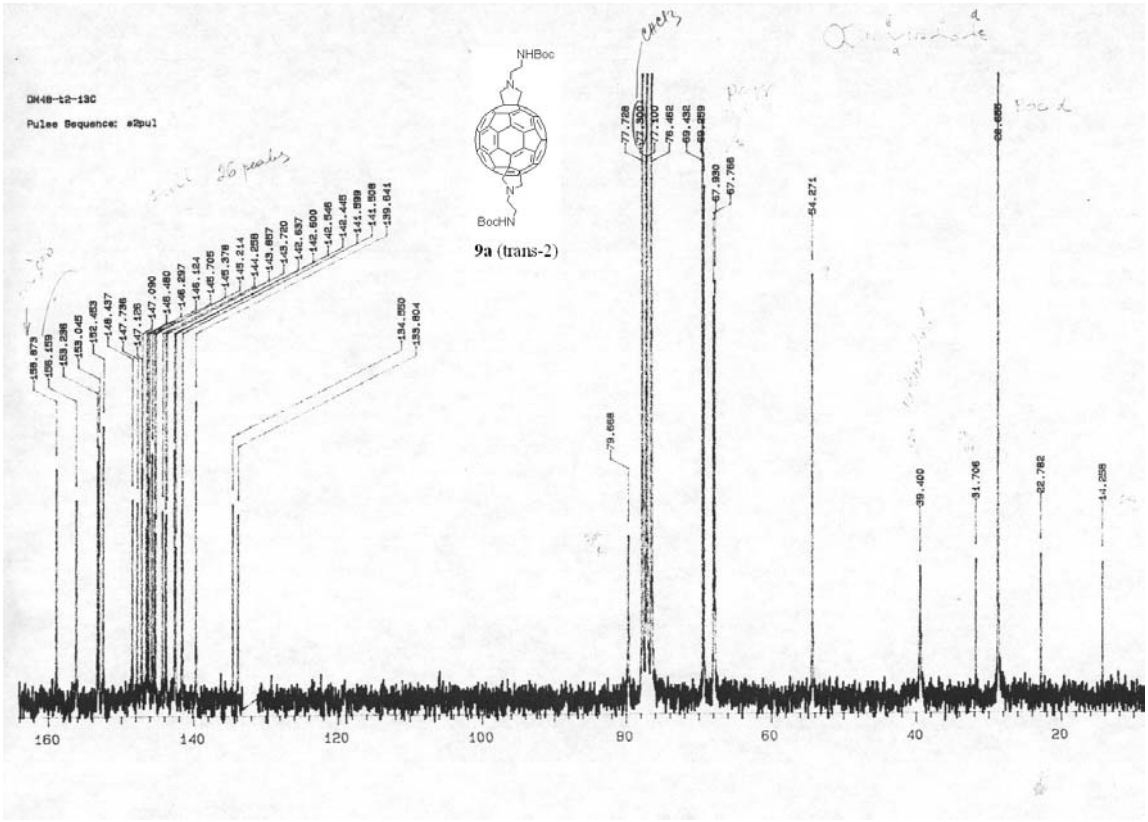

NHBoc
8a
7.243
5.251
3.689
3.660
3.510
3.473
3.439
3.403
3.272
3.243
3.213
1.545
1.498
1.192
1.227
1.195
0.78
4.00
1.96
1.97
1.65
8.48

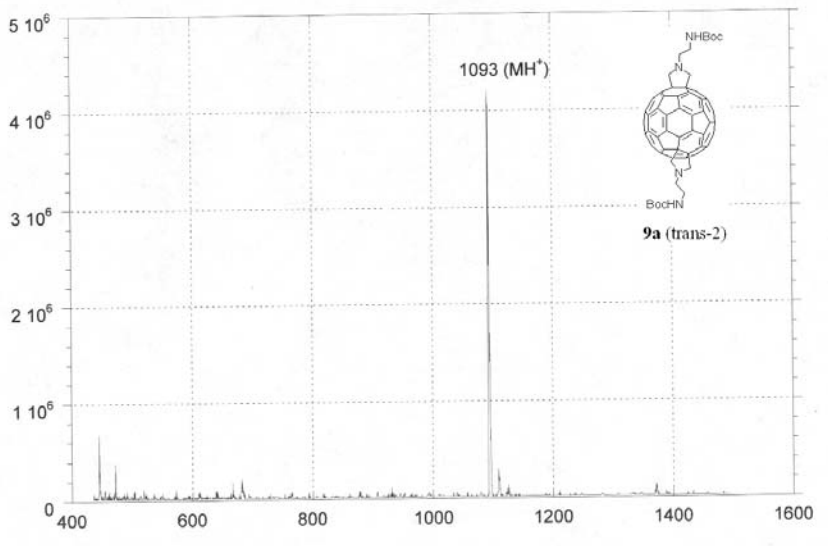

NHBoc
8a


907 (MH+)
NHBoc
8a
C69H18N2O2
Exact Mass: 906,14
Mol. Wt.: 906,89
2 10^6
1.5 10^6
1 10^6
5 10^5
0
400
600
800
1000
1200
1400
1600

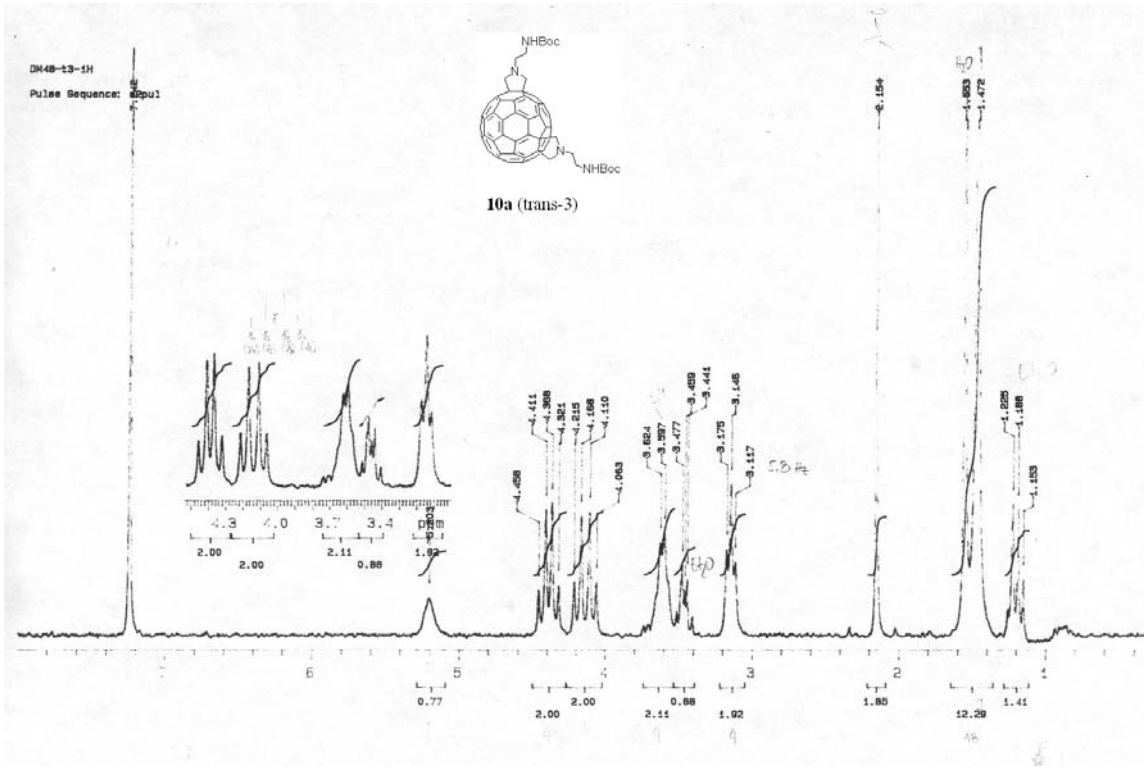
Compound 9a

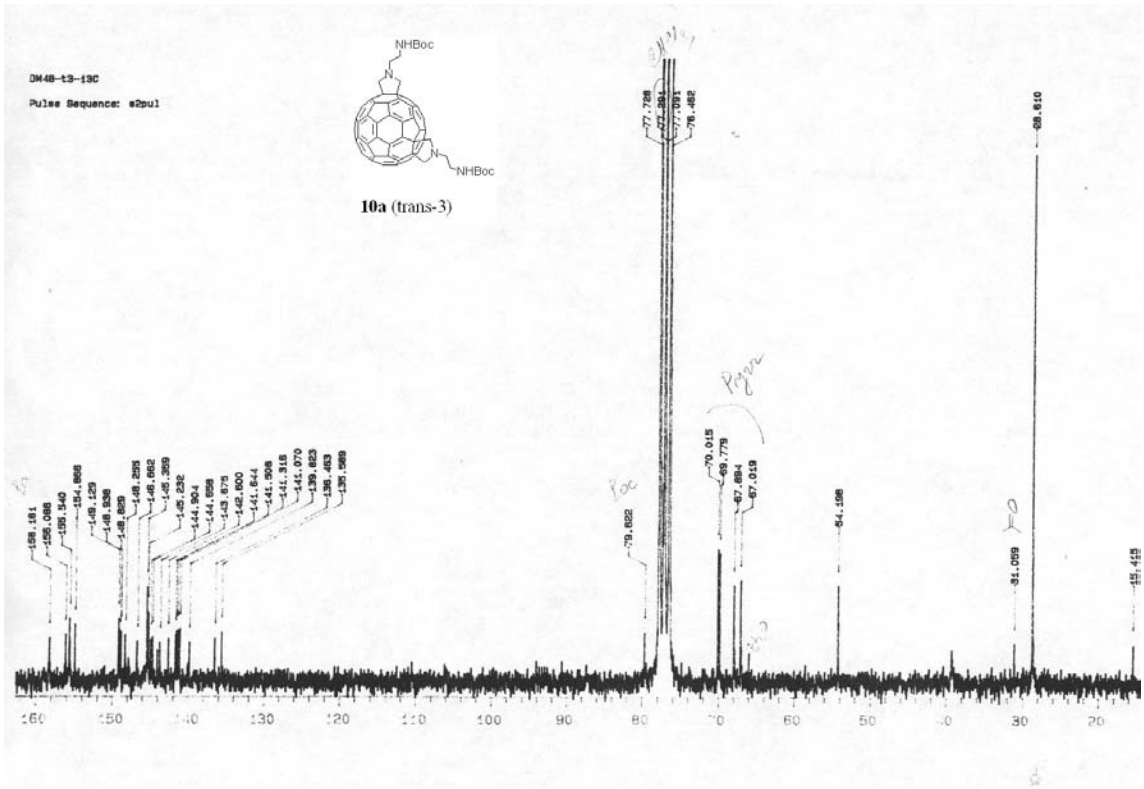

NHBoc
BocHN
9a (trans-2)
7.844
6.931
4.705
4.657
4.623
4.476
4.393
4.359
4.313
3.719
3.692
3.302
3.279
3.243
1.808
1.510
Boc
1.245
0.862
H2O
1.01
1.11
1.12
2.07
2.14
2.08
1.88
0.61
1.21
0.77
7
6
5
4
3
2
1
ppm

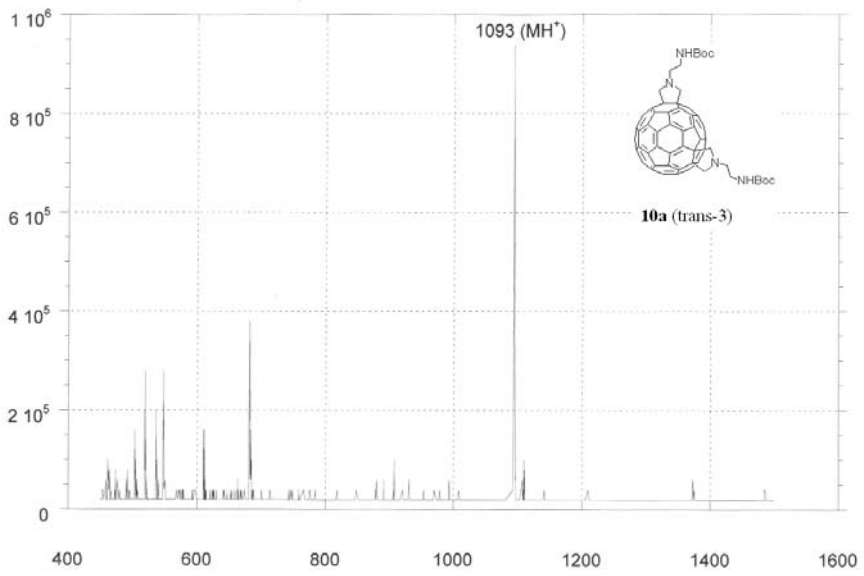




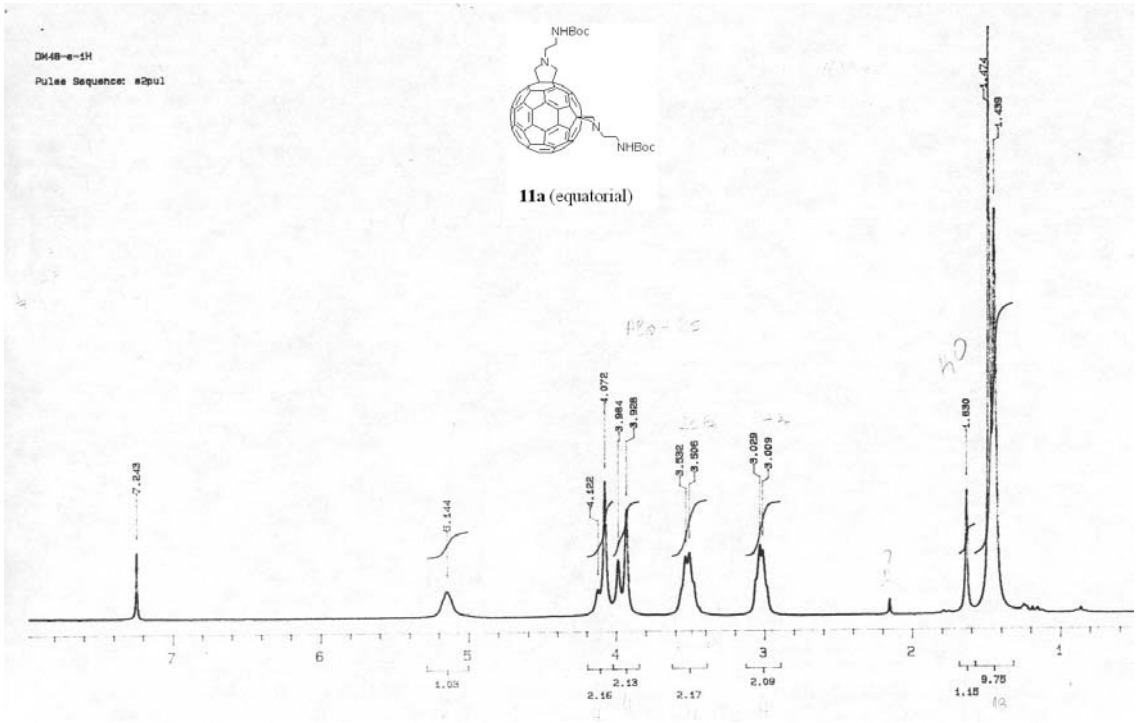
Compound 10a

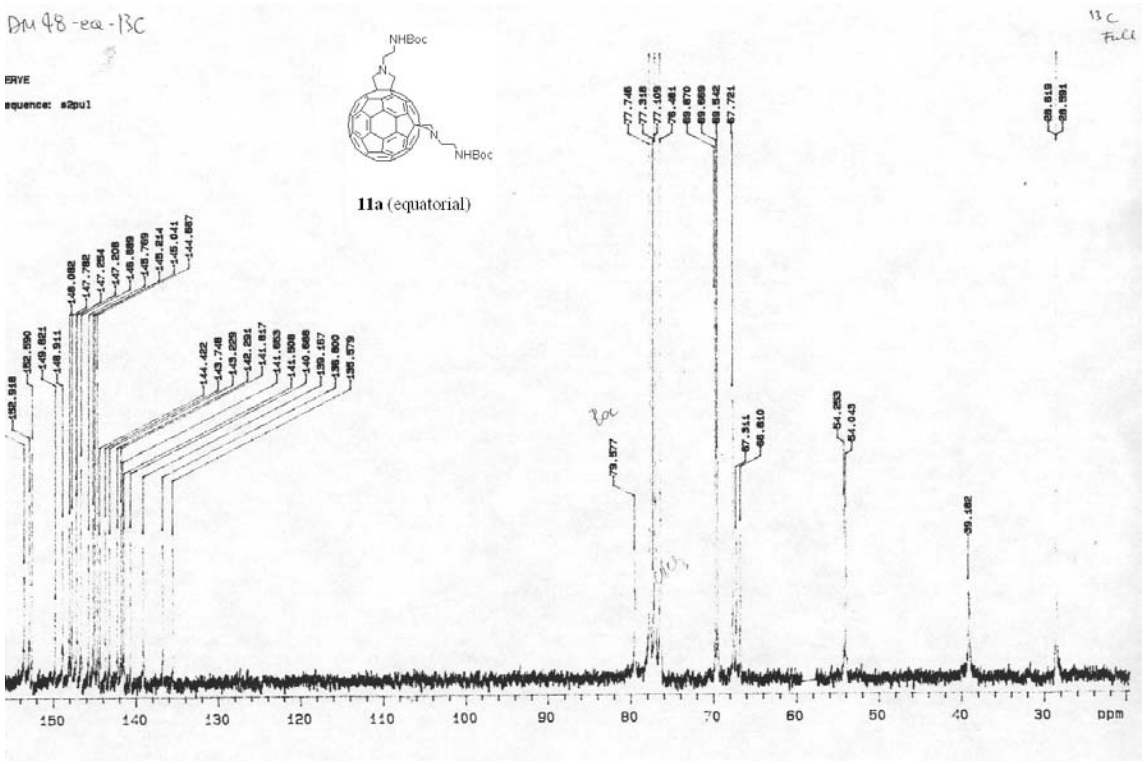

DM4B-t3-1H
Pulse Sequence: s2pul
NHBoc
NHBoc
10a (trans-3)

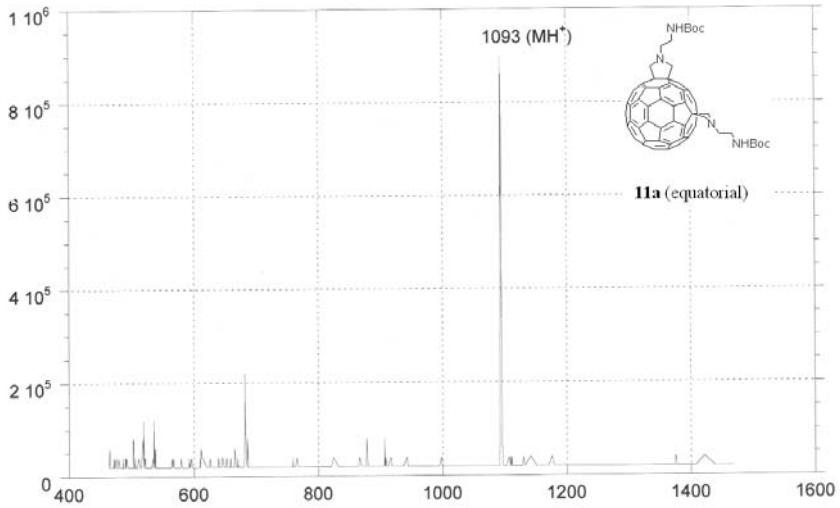

DM4B-t3-13C
Pulse Sequence: s2pul
NHBoc
NHBoc
10a (trans-3)



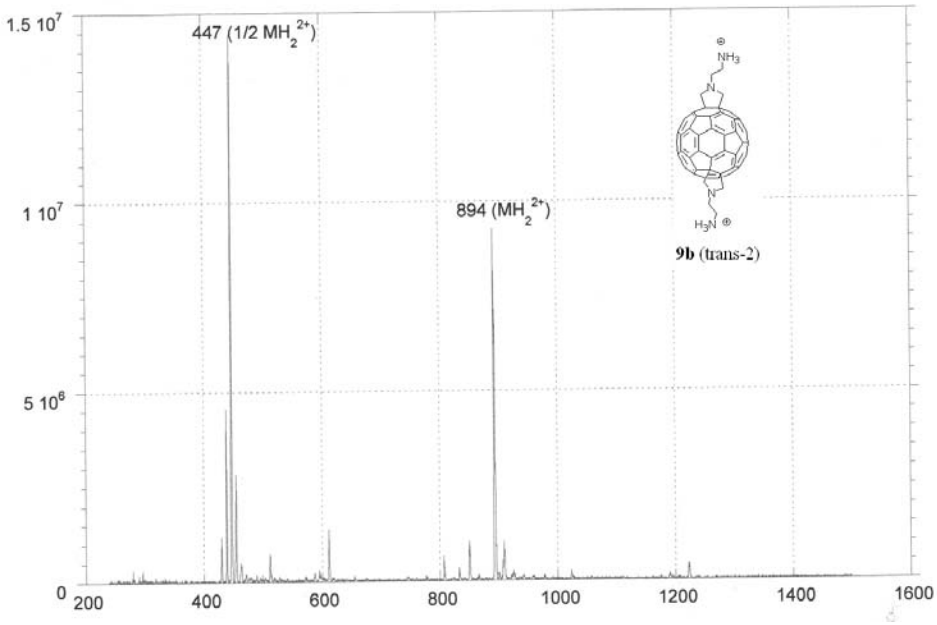
Compound 11a



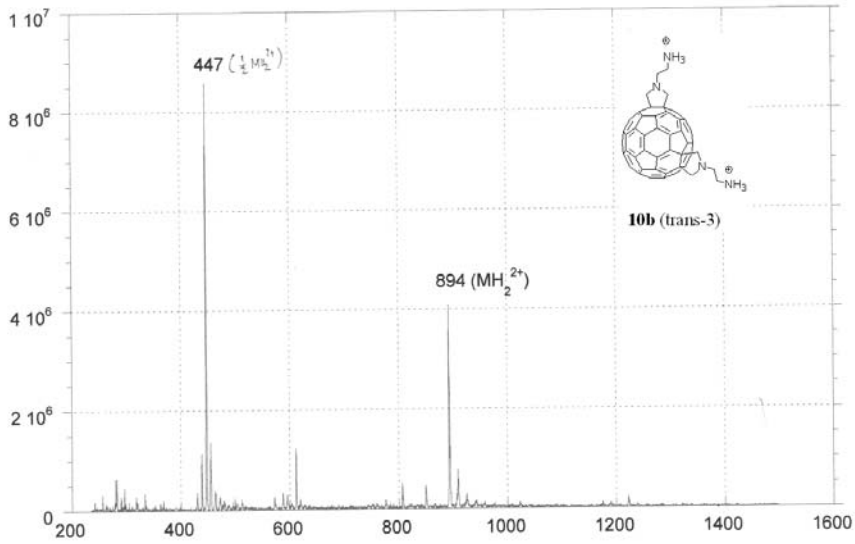

NHBoc
NHBoc
11a (equatorial)
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm


1093 (MH+)
NHBoc
NHBoc
11a (equatorial)
1 10^6
8 10^5
6 10^5
4 10^5
2 10^5
0
400
600
800
1000
1200
1400
1600

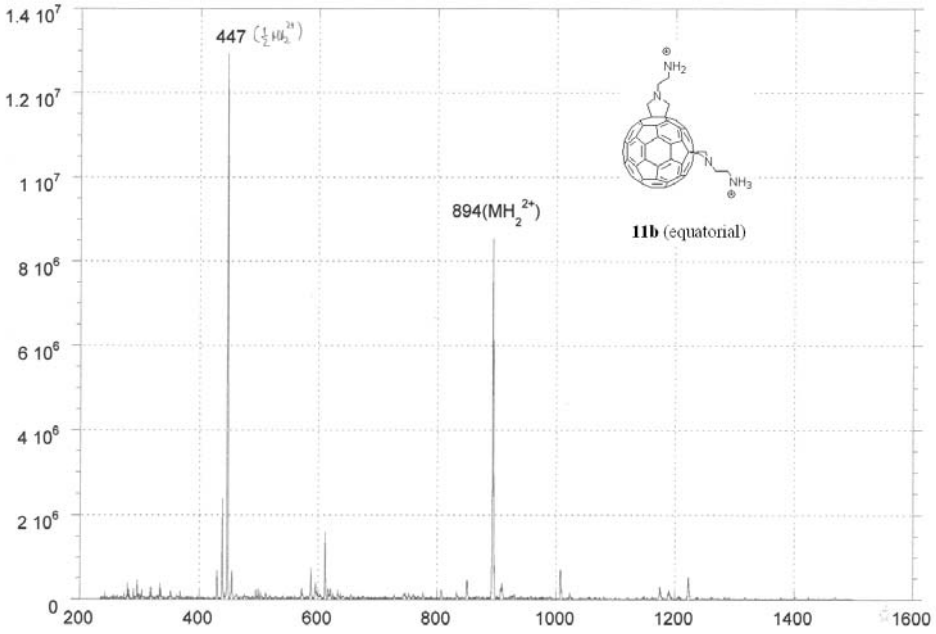
Compound 9b



Compound 10b



Compound 11b



References

(1) Kordatos, K.; Da Ros, T.; Bosi, S.; Vázquez, E.; Bergamin, M.; Cusan, C.; Pellarini, F.; Tomberli, V.; Baiti, B.; Pantarotto, D.; Georgakilas, V.; Spalluto, G.; Prato, M. *J.Org.Chem.* **2001**, *66*, 4915.

(2) Bosi, S.; Feruglio, L.; Milic, D.; Prato, M. *Eur.J.Org.Chem.* **2003**, 4741.

(3) Campidelli, S.; Lenoble, J.; Barberá, J.; Paolucci, F.; Marcaccio, M.; Paolucci, D.; Deschenaux, R. *Macromolecules* **2005**, *38*, 7915.