Carving two adjacent holes on [60]fullerene through two consecutive epoxide to diol to dione transformations

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All the reagents were used as received. Reactions were carried out under lab light in air at r.t. Chromatographic purifications were carried out with 200-300 mesh silica gel. ESI-MS spectra were recorded with $CHCl_3/CH_3OH$ or $CDCl_3/CH_3OH$ as the solvent.

Caution: a large amount of peroxide is involved in some of the reactions, care must be taken to avoid possible explosion.

Compound 2. To a stirred solution of compound **1** (444 mg, 0.429 mmol) in CH_2Cl_2 (148 mL) was added $B(C_6F_5)_3$ (210 mg, 0.410 mmol) at 30 °C. After 10 min, the solution was directly transferred to a silica gel column (35x50 mm) and eluted with toluene/petroleum ether/AcOEt (5:5:1). The first band was collected as the unreacted **1** (trace). The second band was collected and evaporated to give compound **2** (251 mg, 0.238 mmol, 56%).
^{1}H NMR: (400 MHz, $CDCl_3$): δ6.50 (1H), 6.10 (1H), 5.34 (1H), 1.42 (9H), 1.39 (9H), 1.35 (9H) ^{13}C NMR (100 MHz, $CS_2/CDCl_3$): all signals represent 1C except noted 157.48, 151.98, 150.33, 150.19 (2C), 149.37, 149.24, 149.22, 149.13, 148.81, 148.77, 148.75, 148.45, 148.31, 147.67(2C) , 147.46 (2C), 146.87, 146.71, 146.45, 146.41, 145.96, 145.72, 145.65, 145.59, 145.31, 144.95, 144.82, 144.56, 144.44 (2C), 144.35, 144.05, 143.78, 143.41, 142.93, 142.88, 142.72, 142.41, 142.06, 141.86, 140.73, 140.62, 140.32, 140.29, 140.11 (2C), 139.92, 138.10, 136.95, 135.03, 125.04, 122.48, 107.61 (1C, sp3), 82.86 (1C, sp3), 81.88 (1C, sp3), 81.76 (1C, sp3), 78.27 (1C, sp3), 77.57 (1C-$(CH_3)_3$), 77.25 (1C-$(CH_3)_3$), 76.93 (1C-$(CH_3)_3$), 76.53 (1C, sp3), 26.81 (6CH_3), 26.74 (3CH_3) FT-IR (microscope): 3423, 2978, 2928, 1477, 1387, 1364, 1193, 1116, 1073, 1051, 1023, 1003, 676 cm^{-1} ESI-HRMS: $C_{72}H_{34}NO_{10}$ ($M+NH_4^+$) calc. 1072.2177, found 1072.2165.

Compound 3 To a stirred solution of compound **2** (250 mg, 0.237 mmol) in benzene (83 mL) and methanol (0.83 ml) was added DIB (118 mg, 0.366 mmol) at 30 °C. After 1min, the solution was transferred to a silica gel column (35x50 mm) and eluted with toluene/petroleum ether/AcOEt (5:5:1). The first band was collected and evaporated to give compound **3** (218 mg, 0.2072 mmol, 87%).
^{1}H NMR: (400 MHz, $CDCl_3$): δ4.90 (1H), 1.45 (9H), 1.41 (9H), 1.39 (9H) ^{13}C NMR (100 MHz, $CDCl_3$): all signals represent 1C except noted 190.21, 161.22, 156.46, 153.20, 150.43, 150.02, 149.97, 149.89, 149.67, 149.56, 149.25, 149.23, 148.95, 148.63, 148.38, 148.13, 147.97, 147.62, 147.51, 147.12 (2C), 147.09, 146.81, 146.28, 146.23, 146.00, 145.68, 145.43, 145.25, 145.11, 144.98, 144.89, 144.24, 143.84, 143.63, 143.59, 143.43, 143.15 (2C), 142.30, 142.14, 141.99, 141.83 (2C), 141.42, 141.08, 140.24, 140.04, 138.32, 136.85, 136.32, 136.11, 128.10, 125.93, 123.69, 122.69, 85.58 (1C, sp3), 83.49 (1C-$(CH_3)_3$), 83.20 (1C-$(CH_3)_3$), 82.33 (1C-$(CH_3)_3$), 81.18 (1C, sp3), 79.45 (1C, sp3), 78.07 (1C, sp3), 26.70 (3CH_3), 26.66 (3CH_3), 26.33 (3CH_3) FT-IR (microscope):: 3481, 2961, 2925, 1788, 1712, 1364, 1190, 1105, 1094, 1071, 1057, 1014, 993, 867, 734 cm^{-1} ESI-HRMS: $C_{72}H_{32}NO_{10}$ ($M+NH_4^+$) calc. 1070.2021, found 1070.2004

Compound 4 and 5. To a stirred solution of compound **3** (218 mg, 0.2072 mmol) in CH_2Cl_2 (36 mL) was added mCPBA (361 mg, 2.093 mmol) at 30 °C. After 70 min, the solution was transferred to a silica gel column (35x50 mm) and eluted with toluene/petroleum ether/AcOEt (10:10:1). The first band was compound **4** (yield was less than 10%) the second band was collected and the solution was washed with Na_2SO_3-K_2CO_3 and water each for three times, then dried with Na_2SO_4 and evaporated to give compound **5** (155 mg, 0.1430 mmol, 69%)
Compound 4 ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.71 (s, 1H), 1.43 (s, 9H), 1.38 (s, 9H), 1.34 (s, 9H). ^{13}C NMR

(100 MHz, $CDCl_3$): all signals represent 1C except noted. δ186.71, 160.64, 155.92, 152.60, 151.24, 150.89, 150.71, 150.18 150.81, 149.90, 149.78, 149.32, 148.88, 148.71, 148.68, 148.64, 148.56, 148.45, 148.29, 148.07, 147.84, 147.40, 147.08, 146.99, 146.95(2C), 146.69, 146.17, 145.81, 145.71, 145.36, 145.30, 145.23, 144.82, 144.29, 143.09, 143.00, 142.59, 142.49, 142.42, 141.70, 141.53, 141.11, 140.97, 140.69, 140.52, 140.19, 139.41, 138.55, 135.27, 132.92, 126.65, 123.45, 123.30, 85.03 (1C, sp^3), 83.50 (1*C*-$(CH_3)_3$), 83.16(1*C*-$(CH_3)_3$), 82.29(1*C*-$(CH_3)_3$) , 80.13 (1C, sp^3), 80.04 (1C, sp^3), 78.99 (1C, sp^3), 78.82 (1C, sp^3), 76.82 (1C, sp^3), 26.60 ($3CH_3$), 26.54 ($3CH_3$), 26.20 ($3CH_3$). FT-IR (microscope):3476, 3361, 2956, 2923, 2852, 1789, 1717, 1659, 1239, 1190, 1163, 1113, 1097, 1075, 800cm^{-1}. ESI-HRMS: $C_{72}H_{32}NO_{11}$ ($M+NH_4^+$) calc. 1086.1970, found 1086.1949

Compound 5 ^1H NMR (400 MHz, $CDCl_3$): δ 3.56 (s, 1H), 1.44 (s, 9H), 1.35 (s, 18H). ^{13}C NMR (100 MHz, $CDCl_3$): all signals represent 1C except noted. δ186.36, 160.27, 154.22, 151.60, 151.42, 150.50, 150.36, 150.19, 150.18, 150.15, 149.37, 148.54, 148.39, 148.34, 148.23, 147.97, 147.90, 147.88, 147.39, 146.88, 146.80, 146.67, 146.62, 146.52, 146.23, 146.12, 146.10, 145.88, 145.82, 145.73, 145.48, 145.03, 144.96, 144.88, 144.61, 143.62, 142.91, 142.86, 142.75, 142.28, 140.84, 140.51, 140.14, 139.93, 139.84, 139.64, 137.47, 132.74, 127.20, 125.15, 124.33, 85.10 (1C, sp^3), 83.57 (1C, sp^3), 83.45 (1*C*-$(CH_3)_3$), 82.82(1*C*-$(CH_3)_3$) , 82.43(1*C*-$(CH_3)_3$) , 80.40 (1C, sp^3), 79.12 (1C, sp^3), 78.64 (1C, sp^3), 78.33 (1C, sp^3), 75.51 (1C, sp^3), 68.34 (1C, sp^3), 26.73 ($3CH_3$), 26.56 ($3CH_3$), 26.20 ($3CH_3$). FT-IR (microscope):3527, 2981, 2932, 1789, 1715, 1366, 1190, 1106, 911, 731cm^{-1}. ESI-HRMS: $C_{72}H_{29}O_{12}$ ($M+H^+$) calc. 1085.1653, found 1085.1627.

Crystal data and structure refinement for **5**. Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication CCDC-716212. Copies of these data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Identification code	07oct12a
Empirical formula	C74 H28 Cl6 O12
Formula weight	1325.68
Temperature	200(2) K
Measurement device	NONIUS KappaCCD
Measurement method	CCD
Wavelength	0.71073 A
Program for data collection	KappaCCD (Nonius B. V., 1998)
Program for cell refinement	HKL Scalepack (Otwinowski & Minor, 1997)
Program for data reduction	HKL Denzo (Otwinowski & Minor, 1997) & maXus (Mackay et al., 1998)
Program for structure solution	SHELXS-97 (Sheldrick, 1997)
Program for structure refinement	SHELXL-97 (Sheldrick, 1997)
Program for molecular graphics	Bruker SHELXTL V5.1 (Sheldrick, 1998)
Program for publication material	SHELXL-97 (Sheldrick, 1997)
Crystal system, space group	Monoclinic, P 2(1)/c
Unit cell dimensions	a = 19.2672(2) A alpha = 90 deg. b = 15.0967(2) A beta = 105.3484(7) deg. c = 20.0629(3) A gamma = 90 deg.
Volume	5627.58(13) A^3
Refls. No. for cell measurement	42975
Theta range for cell measurement	3.395 to 26.022 deg.
Z, Calculated density	4, 1.562 Mg/m^3

Absorption coefficient	0.379 mm^-1
F(000)	2688
Crystal shape / Crystal colour	plate / red
Crystal size	0.32 x 0.30 x 0.11 mm
Theta range for data collection	3.40 to 26.01 deg.
Limiting indices	-23<=h<=23, -18<=k<=18, -24<=l<=24
Reflections collected / unique	77704 / 11032 [R(int) = 0.0636]
Reflections with I>2sigma(I)	7260
Completeness to theta = 26.01	99.6 %
Decay correction (%)	none
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.968 and 0.787
Method for primary solution	direct
Method for secondary solution	difmap
Hydrogen addition / treatment	geom / constr.
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	11032 / 150 / 957
Goodness-of-fit on F^2	1.040
Final R indices [I>2sigma(I)]	R1 = 0.0819, wR2 = 0.2422
R indices (all data)	R1 = 0.1166, wR2 = 0.2660
Largest diff. peak and hole	1.157 and -0.628 e.A^-3
Max. and mean shift/sigma	0.000 and 0.000

In an independent molecule of compound **5** in the solid state, there should be two disordered $CHCl_3$ molecules. The solved $CHCl_3$ molecule was assigned as 50% occupancy. The other 1.5 molecules of $CHCl_3$ were difficult to locate by difference Fourier syntheses due to insufficient quality of crystals. The data has been run through the PLATON program in the absence of the 1.5 molecules of chloroforms by use of Squeeze program. The hydrogen atoms of $CHCl_3$ was not located, other hydrogen atoms were placed at the calculated positions and were included in the structure calculation without further refinement of the parameters.

Compound 6. To a stirred solution of compound **5** (139 mg, 0.1282 mmol) in CH_2Cl_2 (70 mL) was added TFA (0.816 ml) at 30 ^{o}C. After 30min, the solution was washed with water twice. The organic layer was transferred to a silica gel column (15x30 mm) and eluted with toluene/petroleum ether/AcOEt (3:3:1). The first band was collected as unreacted **5** (trace). The second band was collected and evaporated to give compound **6** (103 mg, 0. 0935 mmol, 73%).

^{1}H NMR (600 MHz, $CDCl_3$): δ5.30 (s, 1H), 5.08 (s, 1H), 2.05 (s, 1H), 1.40 (s, 9H), 1.38 (s, 9H), 1.30 (s, 9H)
^{13}C NMR (150 MHz, $CDCl_3$): all signals represent 1C except as noted; δ 194.95, 158.88, 153.34, 151.70, 151.52, 151.27, 150.89, 150.55, 150.32, 149.95, 148.97, 148.83, 148.78, 148.66, 148.23, 148.20, 147.87, 147.82, 147.53, 147.27, 147.22, 146.91, 146.65, 146.61, 146.53, 146.17(2C), 145.78(2C), 145.74(2C), 145.46, 145.20, 144.60, 143.55, 143.45, 143.39, 143.31, 143.07, 142.54, 141.93, 141.89, 140.95, 140.02, 139.90, 138.62, 138.55, 138.10, 137.25, 131.52(2C), 125.42, 99.30 (1C, sp^3), 88.08 (1C, sp^3), 82.91(1*C*-$(CH_3)_3$), 82.69 (1*C*-$(CH_3)_3$), 82.57(1*C*-$(CH_3)_3$), 81.57(1C, sp^3), 80.17(1C, sp^3), 79.90(1C, sp^3), 78.12(1C, sp^3), 78.03(1C, sp^3), 74.71(1C, sp^3), 26.72 ($3CH_3$), 26.66 ($3CH_3$), 26.53 ($3CH_3$). FT-IR (microscope): 3482, 2978, 2928, 2853, 1759, 1388, 1365, 1189, 1155, 1112, 1084, 1027, 732 cm^{-1}. ESI-HRMS: $C_{72}H_{34}NO_{13}$ ($M+NH_4^+$) calc.1120.2025, found 1120.1995

Compound 7 and 9. To a stirred solution of compound **6** (32 mg, 0.0290 mmol) in CH_2Cl_2 (5 mL) was added PIFA (54 mg, 0.126 mmol) at 30 °C. After 40min, the solution was directly transferred to a silica gel column (15 x 20 mm) and eluted with toluene/petroleum ether/AcOEt (3:3:1). The first band was collected and evaporated to give compound **7** (29 mg, 0.02636 mmol, 91%). The second band was collected and evaporated to give compound **9** (trace).

Compound 7 ^{1}H NMR (400 MHz, $CDCl_3$): δ6.47(1H), 1.43(9H), 1.30(9H), 1.25(9H). ^{13}C NMR (100 MHz, $CDCl_3$): all signals represent 1C except as noted; δ 192.86, 181.70, 180.92, 158.36, 151.99, 151.50, 151.42, 151.04, 150.95, 150.77, 149.62, 149.20, 149.00, 148.78, 148.48, 148.28, 148.20, 148.09, 147.91, 147.77, 147.66, 147.50, 147.07, 147.02, 146.84, 146.73, 146.68, 146.25, 146.06, 145.99, 145.88, 145.32, 144.85, 144.82, 144.68, 144.45, 144.36, 144.06, 142.14, 141.78, 141.44, 141.38, 140.99, 140.18, 138.80, 138.78, 138.43, 138.07, 137.91, 136.73, 136.38, 134.98, 132.16, 122.56, 99.17(1C, sp^3), 90.35(1C, sp^3), 88.95(1C, sp^3), 88.36(1*C*-$(CH_3)_3$), 83.05(1*C*-$(CH_3)_3$), 82.63(1*C*-$(CH_3)_3$), 81.88(1C, sp^3), 81.21(1C, sp^3), 76.37(1C, sp^3), 26.77 ($3CH_3$), 26.61 ($3CH_3$), 26.25 ($3CH_3$) FT-IR (microscope): 3430, 2980, 2927, 2853, 1761, 1719, 1459, 1389, 1365, 1189, 1151, 1080, 1013, 949, 910, 865, 792, 730. ESI-HRMS: $C_{72}H_{32}NO_{13}$ ($M+NH_4^+$) calc.1118.1868, found 1118.1835.

Compound 9 ^{1}H NMR (400 MHz, $CDCl_3$): δ6.09(1H), 5.96(1H), 5.83(1H), 1.44(9H), 1.35(9H), 1.30(9H) ^{13}C NMR (100 MHz, $CDCl_3$): all signals represent 1C except as noted; δ 188.43, 159.47, 151.91, 151.34, 151.29, 151.04, 150.91, 150.75, 150.03, 149.20, 149.09, 148.75, 148.51, 148.41, 148.07, 148.01, 147.82, 147.55, 147.22, 147.00, 146.81, 146.75, 146.72, 146.45, 146.39, 146.22, 145.76, 145.55, 145.46, 145.09, 144.86, 144.77, 144.41, 144.18, 143.52, 143.23, 142.26, 141.44, 141.21(2C), 140.39, 139.82(2C), 139.22, 138.80, 138.46, 137.90, 137.33, 136.43, 136.22, 132.23, 123.78, 99.63(1C, sp^3), 96.19(1C, sp^3), 94.77(1C, sp^3), 92.54(1C, sp^3), 88.85(1C, sp^3), 83.54(1*C*-$(CH_3)_3$), 82.68(1*C*-$(CH_3)_3$), 82.05(1*C*-$(CH_3)_3$), 81.45(1C, sp^3), 80.96 (1C, sp^3), 77.68(1C, sp^3), 26.81CH_3), 26.77 CH_3), 26.66 CH_3) FT-IR (microscope): 3446, 2978, 2928, 1761, 1365, 1192, 1158, 1087,, 1025, 917, 866, 792, 732. ESI-HRMS: $C_{72}H_{34}NO_{14}$ ($M+NH_4^+$) calc.1136.1974, found 1136.1942

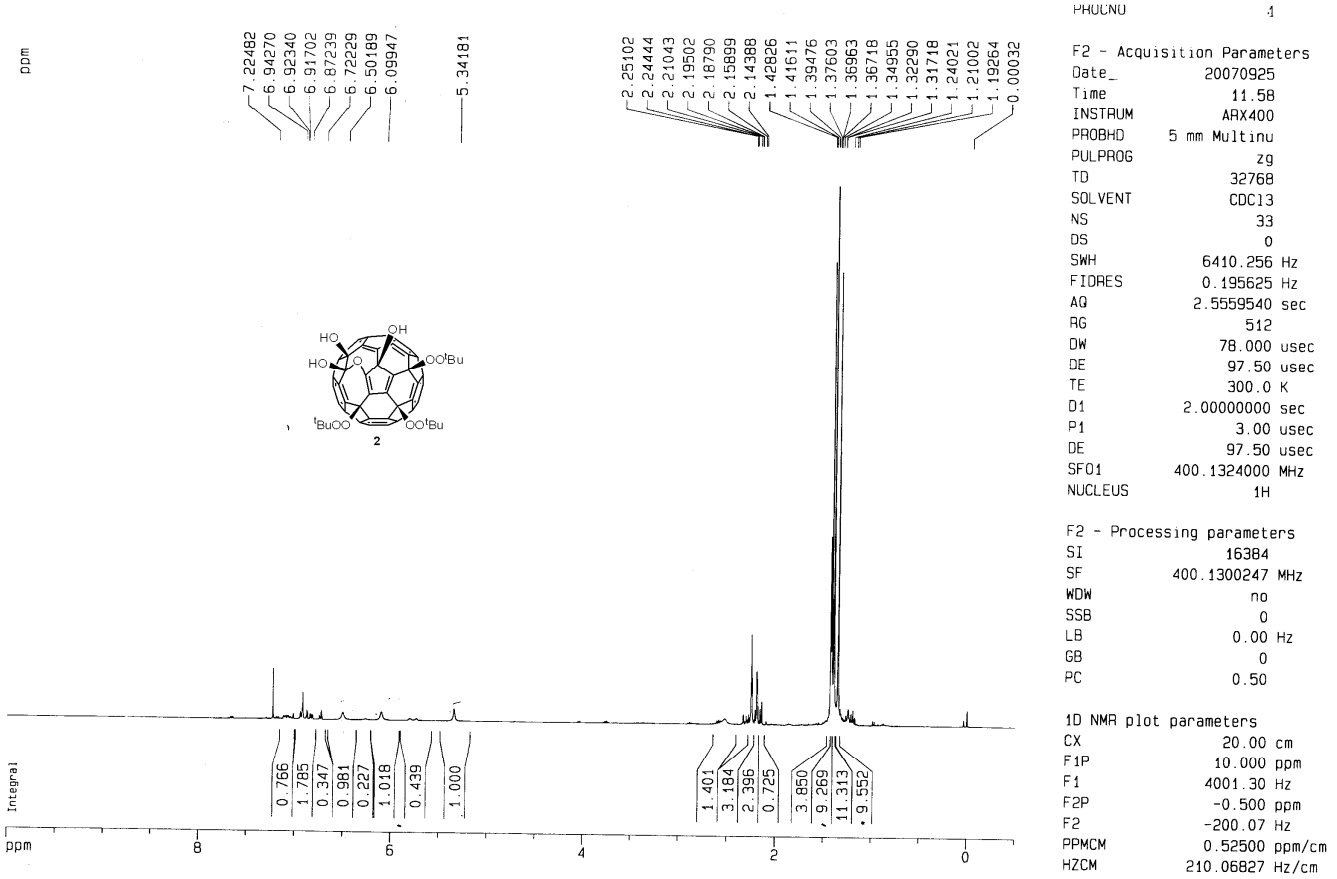
Compound 8. To a stirred solution of compound **7** (10 mg, 0.00909 mmol) in CH_2Cl_2 (2.5 mL) was added PCl_5 (150 mg, 0.7194 mmol) at 30 °C. After 60min, the solution was directly transferred to a silica gel column (10 x15 mm) and eluted with toluene/petroleum ether/AcOEt (3:3:1). The first band was collected and evaporated to give compound **8** (10 mg, 0.00894 mmol, 98%). The second band was collected and evaporated to give compound **10** (trace).

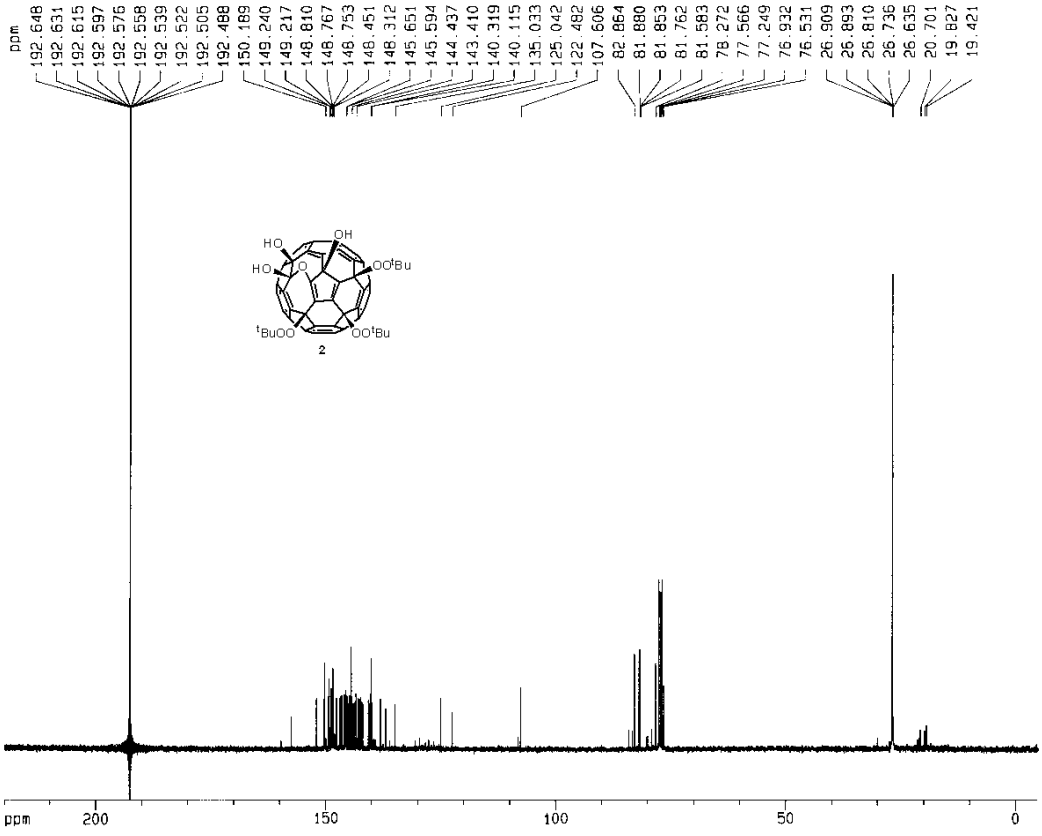
^{1}H NMR (400 MHz, $CDCl_3$): δ 1.44(9H), 1.31(9H), 1.26(9H). ^{13}C NMR (100 MHz, $CDCl_3$): all signals represent 1C except as noted; δ 192.67, 180.87, 180.84, 156.13, 151.75, 151.60, 151.06, 151.04, 150.99, 150.86, 149.72, 149.24, 149.00, 148.83, 148.57, 148.43, 148.24, 148.09(2C), 147.81, 147.76, 147.61, 147.15, 147.00, 146.97, 146.76, 146.67, 146.15, 146.13, 146.03, 145.60, 144.94, 144.74, 144.53, 144.42, 144.05, 143.74, 142.79, 142.26, 142.19, 141.61, 141.46, 141.02, 140.52, 139.14, 138.92, 138.65, 138.11, 137.59, 136.26, 135.83, 134.99, 131.26, 120.47, 91.55(1C, sp^3), 90.41(1C, sp^3), 88.98(1C, sp^3), 83.45(1*C*-$(CH_3)_3$), 83.18(1*C*-$(CH_3)_3$), 82.70(1*C*-$(CH_3)_3$), 82.01(1C, sp^3), 81.11(1C, sp^3), 75.71(1C, sp^3), 26.74($3CH_3$), 26.57($3CH_3$), 26.22($3CH_3$). FT-IR (microscope): 2979, 2928, 1785, 1720, 1365, 1191, 1145, 1105, 1079, 1024, 864. ESI-HRMS: $C_{72}H_{31}ClNO_{12}$ ($M+NH_4^+$) calc.1136.1529, found 1136.1527

Compound 10 To a stirred solution of compound **9** (20 mg, 0.01789 mmol) in CH_2Cl_2 (3 mL) was added PCl_5 (160 mg, 0.7674 mmol) at 30 °C. After 2 min, the solution was directly transferred to a silica gel column (10x15 mm) and eluted with CH_2Cl_2/AcOEt (10:1). The first band was collected and evaporated to give

compound **10** (19 mg, 0.01672 mmol, 93%).

^{1}H NMR (400 MHz, $CDCl_3$): δ1.44(9H), 1.35(9H), 1.30(9H). ^{13}C NMR (150 MHz, $CDCl_3$): all signals represent 1C except as noted; δ 188.53, 157.36, 151.68, 151.45, 151.01, 150.91(2C), 150.78, 150.10, 149.22, 149.09, 148.75, 148.53, 148.41, 148.15, 148.11, 147.82, 147.61, 147.31, 147.10, 146.73, 146.71, 146.68, 146.64, 146.45, 146.42, 145.58(2C), 145.34, 145.00, 144.62, 144.42, 144.28, 143.92, 143.74, 143.20, 142.34, 141.21, 141.14, 141.07, 140.36, 140.26, 139.32, 138.67, 138.25, 138.09, 138.85, 137.44, 136.32, 136.31, 131.45, 121.73, 96.06(1C, sp^3), 94.82(1C, sp^3), 92.77(1C, sp^3), 92.43(1C, sp^3), 88.89(1C, sp^3), 83.54(1*C*-$(CH_3)_3$), 82.78(1*C*-$(CH_3)_3$), 82.11(1*C*-$(CH_3)_3$), 81.48(1C, sp^3), 80.95(1C, sp^3), 26.77($3CH_3$), 26.72($3CH_3$), 26.60($3CH_3$). FT-IR (microscope): 3505 , 3450, 2977, 2926, 2855, 1786, 1727, 1459, 1388, 1366, 1192, 1150, 1109, 1084, 1025, 863 cm^{-1}. ESI-HRMS: $C_{72}H_{33}ClNO_{13}$ ($M+NH_4^+$) calc.1154.1635, found 1154.1598.





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Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

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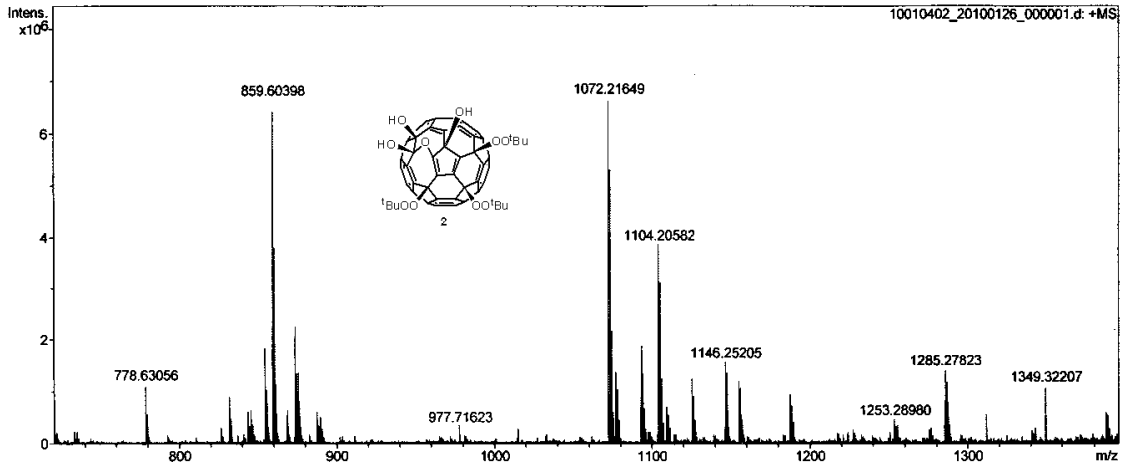

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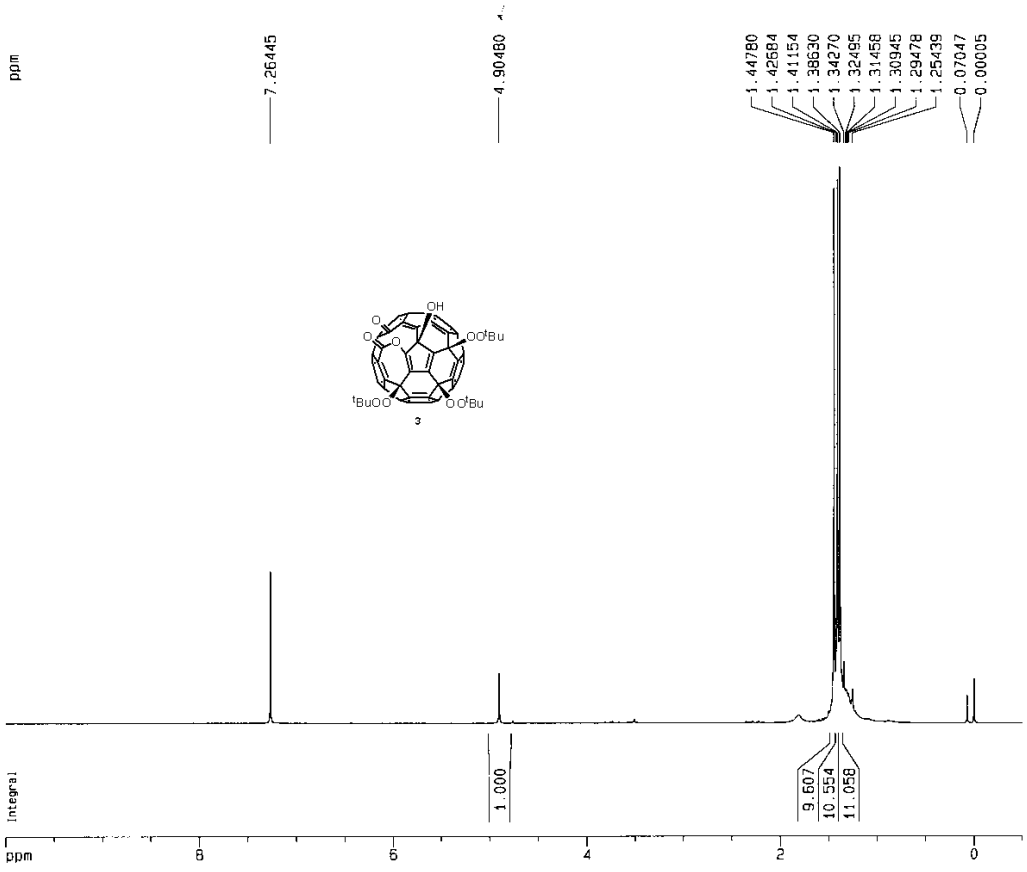

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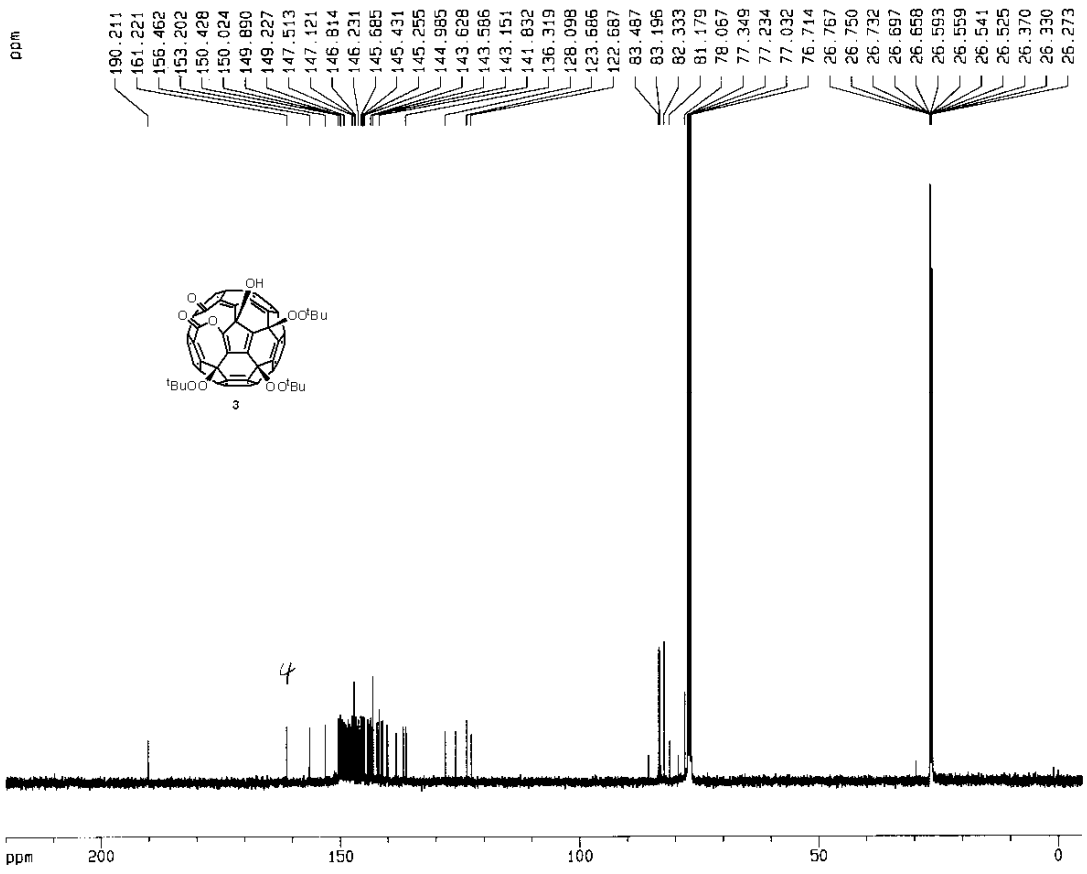
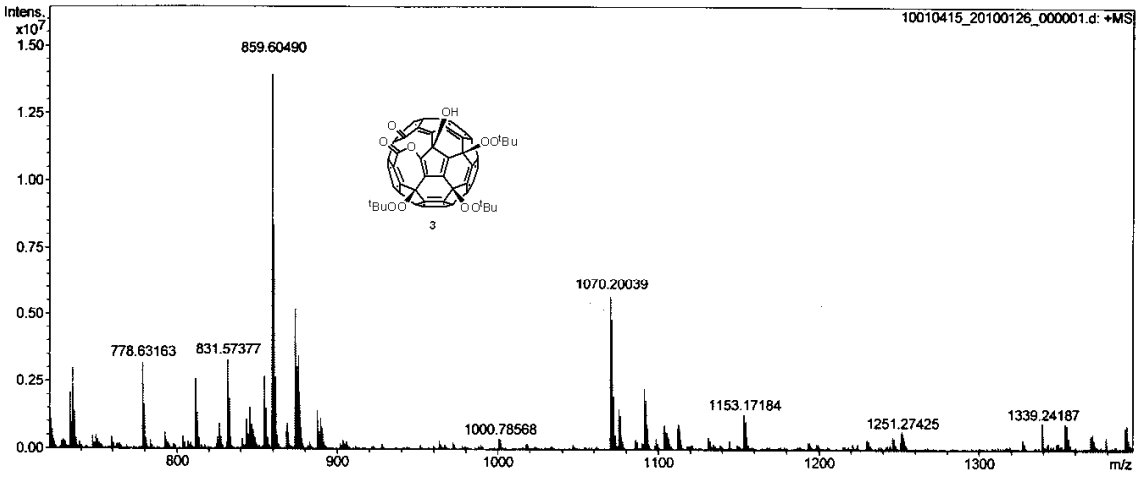
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm


ppm
190.211
161.221
156.462
153.202
150.428
150.024
149.890
149.227
147.513
147.121
146.814
146.231
145.685
145.431
145.255
144.985
143.628
143.586
143.151
141.832
136.319
128.098
123.686
122.687
83.487
83.196
82.333
81.179
78.067
77.349
77.234
77.032
76.714
26.767
26.750
26.732
26.697
26.658
26.593
26.559
26.541
26.525
26.370
26.330
26.273
OH
OO^{t}Bu
^{t}BuOO
OO^{t}Bu
3
4
ppm
200
150
100
50
0

Peking University Mass Spectrometry Sample Analysis Report

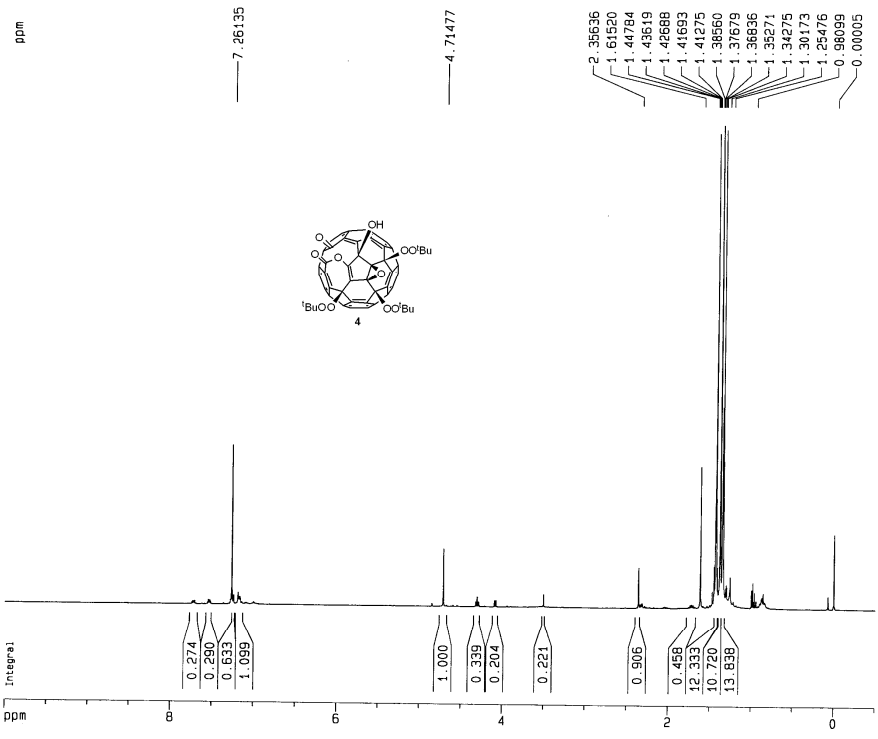
Analysis Info

Analysis Name	10010415_20100126_000001.d	Acquisition Date	1/26/2010 10:09:18 AM
Sample	yao2	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University

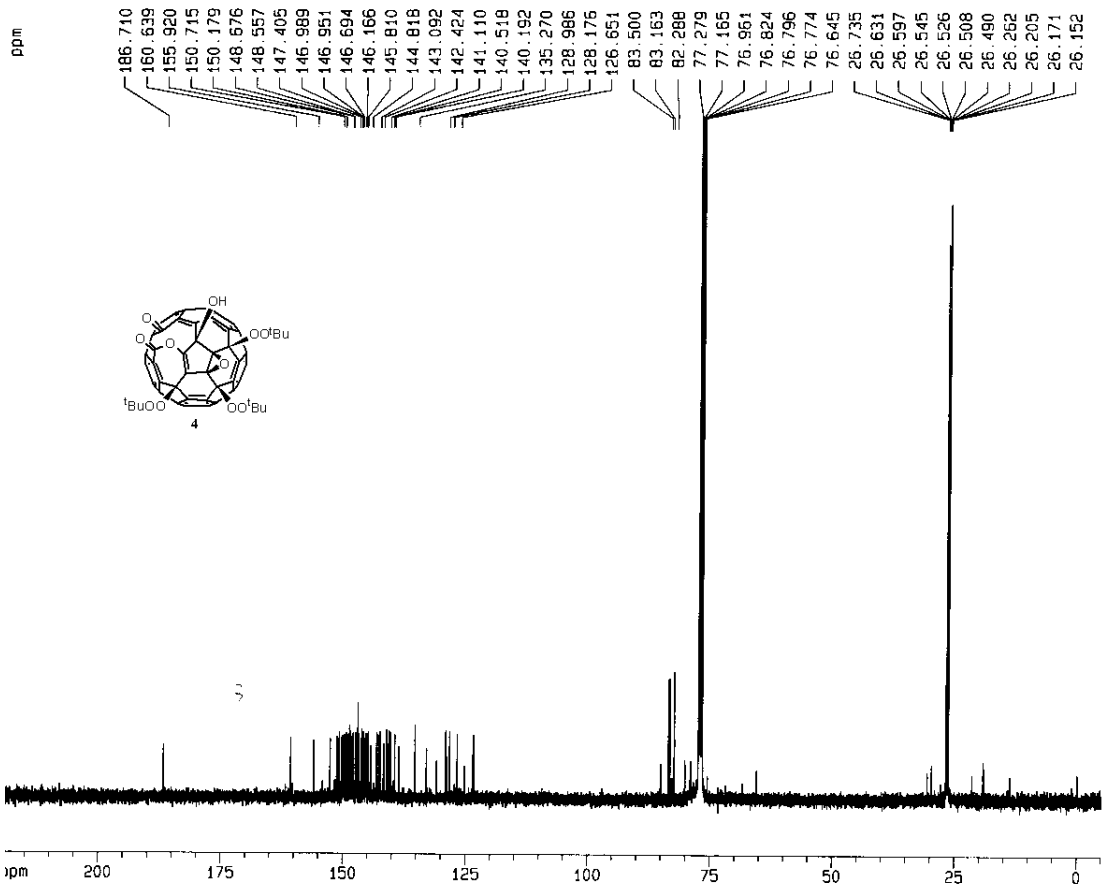


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
1070.20039	1	C 72 H 32 N O 10	100.00	1070.20207	1.7	1.6	23.0	57.5	even	ok

Bruker Compass DataAnalysis 4.0 printed: 1/26/2010 10:10:05 AM Page 1 of 1



EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	20071016
Time	17.05
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	58
DS	0
SWH	6410.256 Hz
FIDRES	0.195625 Hz
AQ	2.5559540 sec
RG	2048
DW	78.000 usec
DE	97.50 usec
TE	300.0 K
D1	2.00000000 sec
P1	3.00 usec
DE	97.50 usec
SFO1	400.1324000 MHz
NUCLEUS	1H
F2 - Processing parameters	
SI	16384
SF	400.1300091 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.50
1D NMR plot parameters	
CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.52500 ppm/cm
HZCM	210.06825 Hz/cm



Current Data Parameters

NAME	C36799
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20071016
Time	17.13
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zgdc
TD	32768
SOLVENT	CDCl3
NS	16000
DS	0
SWH	27777.777 Hz
FIDRES	0.847710 Hz
AQ	0.5898740 sec
RG	8192
DW	18.000 usec
DE	25.71 usec
TE	300.0 K
D12	0.00002000 sec
DL5	22.20 dB
CPDPRG	waltz16
P31	100.00 usec
D1	2.00000000 sec
P1	2.50 usec
DE	25.71 usec
SFO1	100.6240000 MHz
NUCLEUS	13C
D11	0.03000000 sec

F2 - Processing parameters

SI	32768
SF	100.6127747 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	0.30

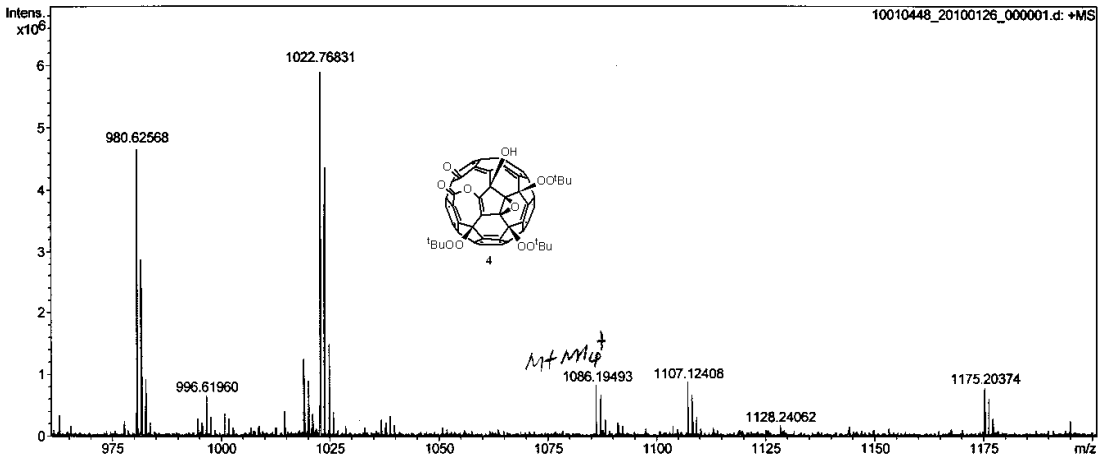
1D NMR plot parameters

CX	20.00 cm
F1P	220.000 ppm
F1	22134.81 Hz
F2P	-5.000 ppm
F2	-503.07 Hz
PPMCM	11.25000 ppm/cm
HZCM	1131.89380 Hz/cm

Peking University Mass Spectrometry Sample Analysis Report

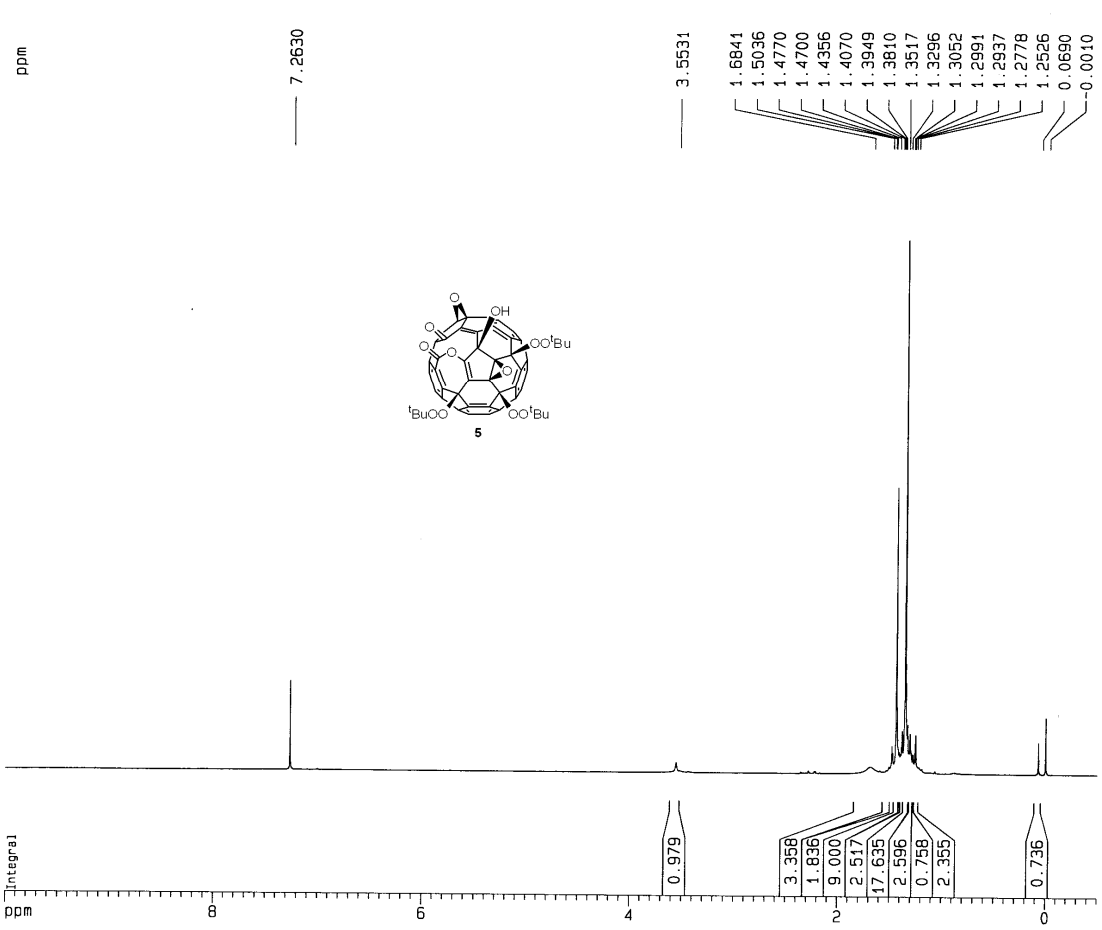
Analysis Info

Analysis Name	10010448_20100126_000001.d	Acquisition Date	1/26/2010 2:37:55 PM
Sample	yao11	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University

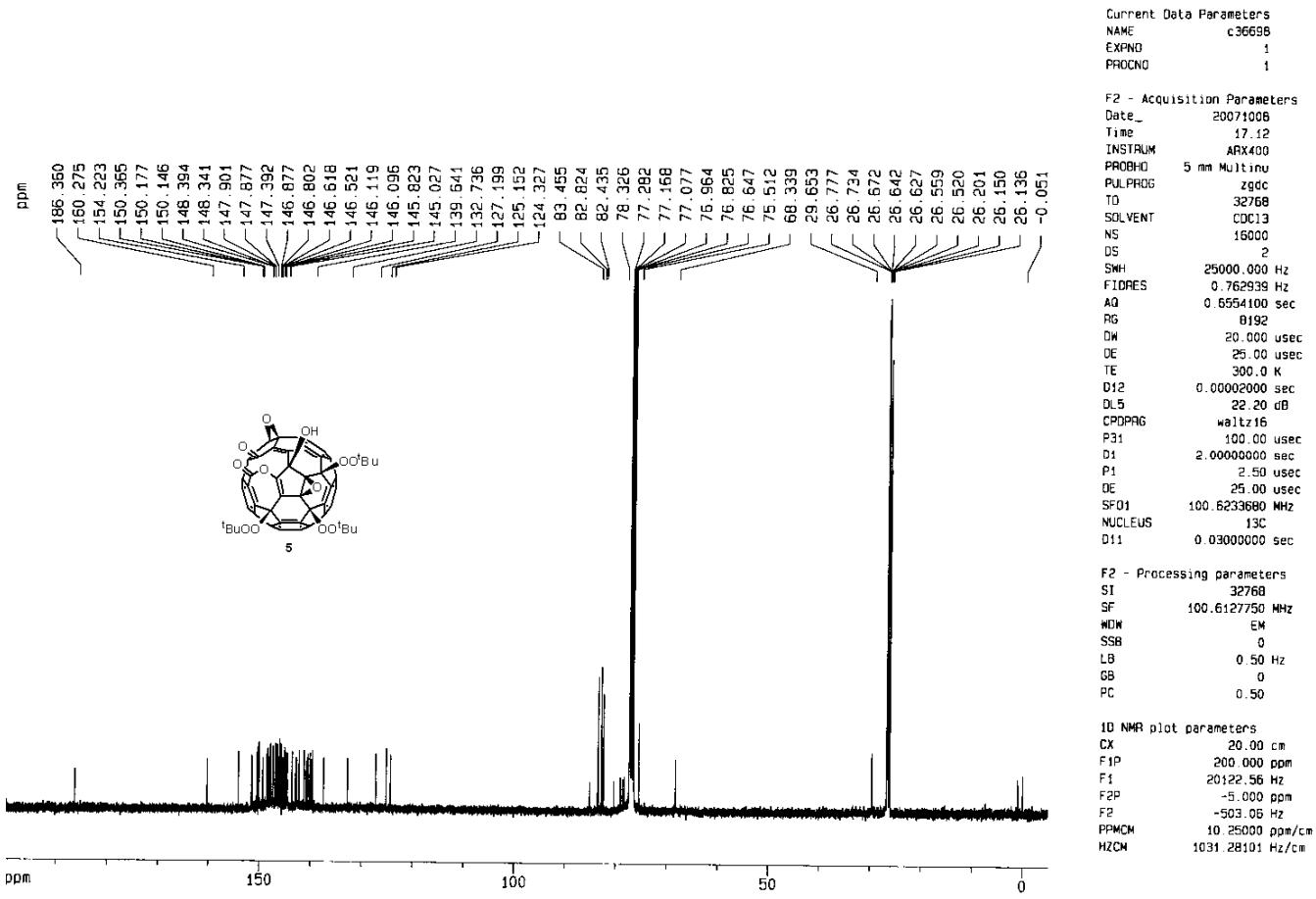


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
1086.19493	1	C 72 H 32 N O 11	100.00	1086.19699	2.1	1.9	45.9	57.5	even	ok

Bruker Compass DataAnalysis 4.0 printed: 1/26/2010 2:50:49 PM Page 1 of 1



Date_	20071008
Time	17.08
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	1430
DW	69.000 usec
DE	98.57 usec
TE	300.0 K
D1	2.00000000 sec
P1	3.00 usec
DE	98.57 usec
SFO1	400.1318844 MHz
NUCLEUS	1H
F2 - Processing parameters	
SI	16384
SF	400.1300082 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	4.00
1D NMR plot parameters	
CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.52500 ppm/
HZCM	210.06825 Hz/c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150.177
150.146
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147.901
147.877
147.392
146.877
146.802
146.618
146.521
146.119
146.096
145.823
145.027
139.641
132.736
127.199
125.152
124.327
83.455
82.824
82.435
78.326
77.282
77.168
77.077
75.964
76.825
76.647
75.512
68.339
29.653
26.777
26.734
26.672
26.642
26.627
26.559
26.520
26.201
26.150
26.136
-0.051
OH
OO^{t}Bu
^{t}BuOO
OO^{t}Bu
5
150
100
50
0
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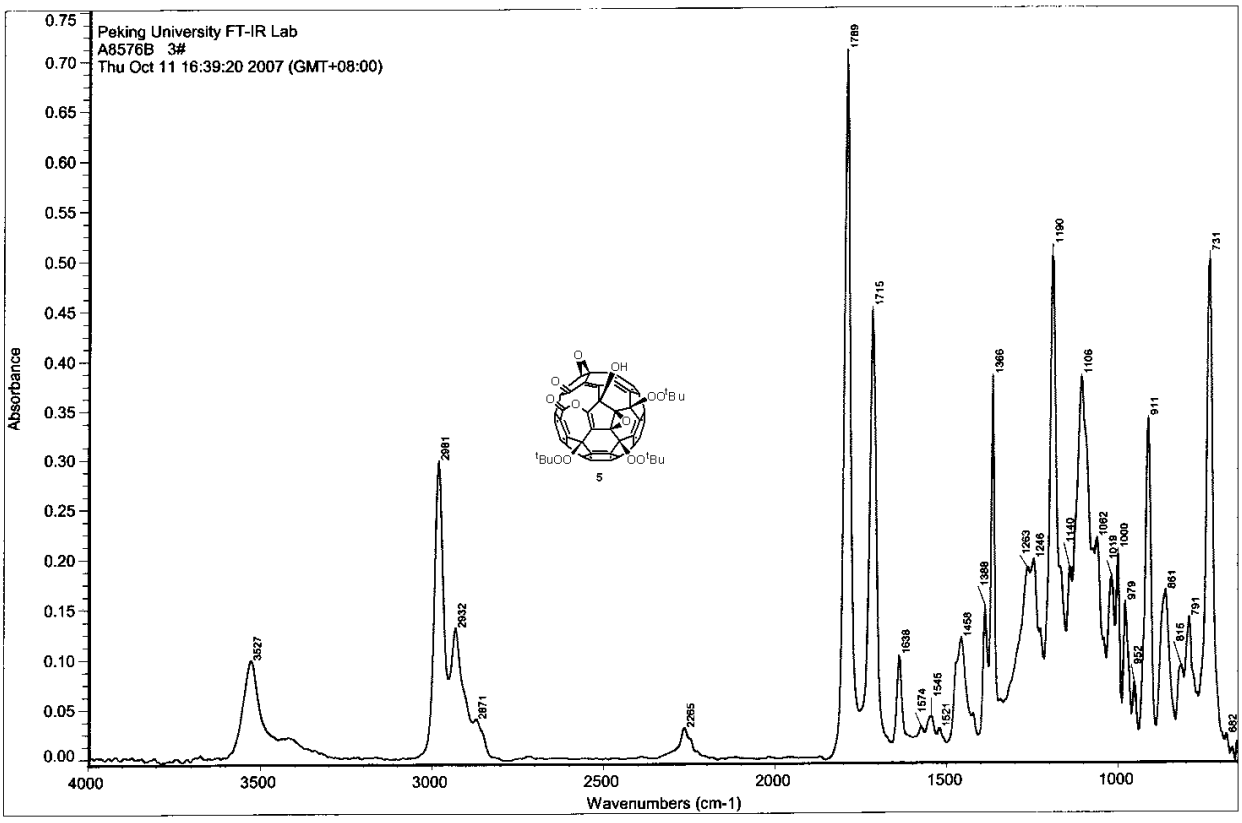
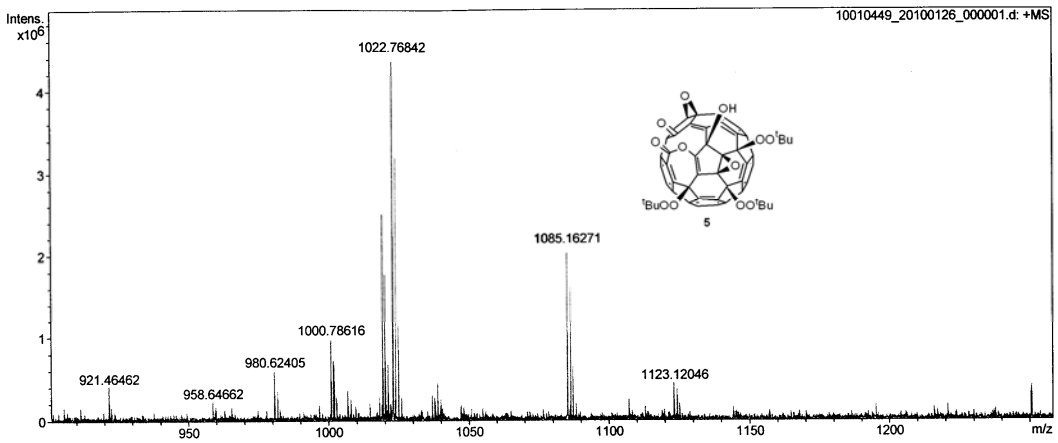
NAME c36698
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071008
Time 17.12
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zgdc
TD 32768
SOLVENT CDCl3
NS 16000
DS 2
SWH 25000.000 Hz
FIDRES 0.762939 Hz
AQ 0.6554100 sec
RG 8192
DW 20.000 usec
DE 25.00 usec
TE 300.0 K
D12 0.00002000 sec
DL5 22.20 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 2.50 usec
DE 25.00 usec
SFO1 100.6233680 MHz
NUCLEUS 13C
D11 0.03000000 sec
F2 - Processing parameters
SI 32768
SF 100.6127750 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 0.50
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.56 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 10.25000 ppm/cm
HZCM 1031.28101 Hz/cm


Peking University FT-IR Lab
A8576B 3#
Thu Oct 11 16:39:20 2007 (GMT+08:00)
Absorbance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
0.75
0.70
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
3527
2961
2932
2871
2265
1789
1715
1638
1574
1545
1521
1458
1389
1366
1263
1246
1190
1140
1106
1062
1018
1000
979
911
861
852
816
791
731
682
OH
OO^{t}Bu
^{t}BuOO
OO^{t}Bu
5

Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

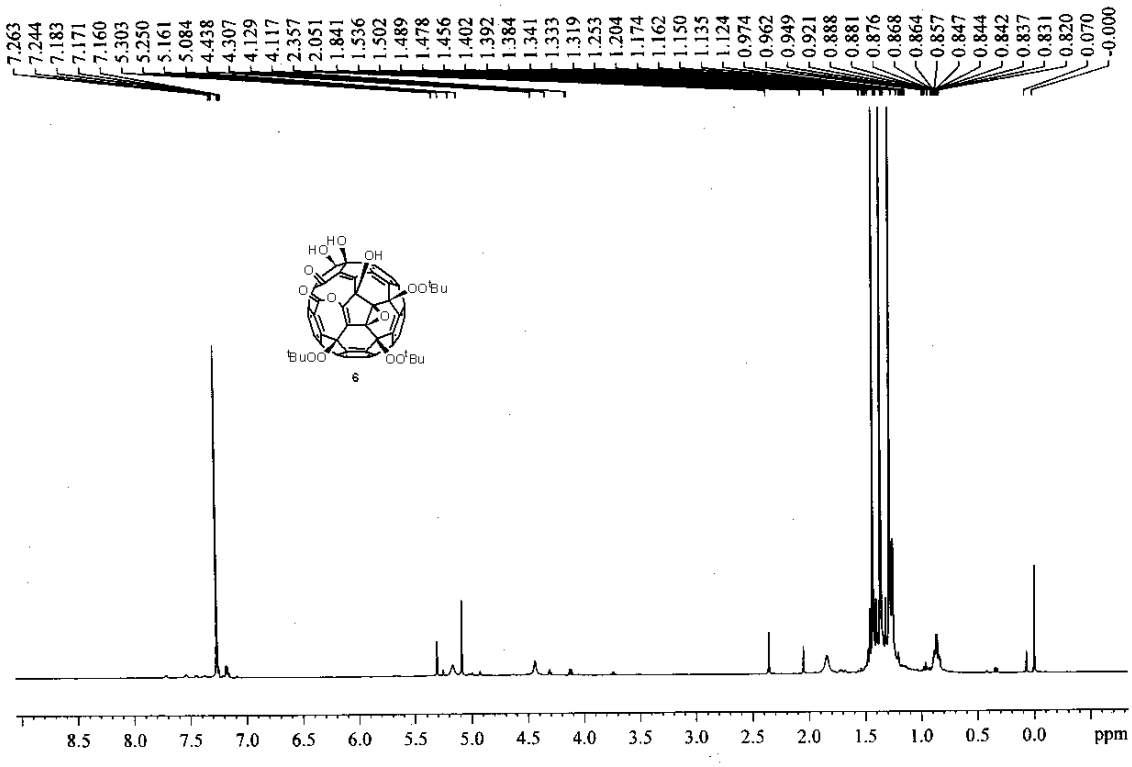
Analysis Name	10010449_20100126_000001.d	Acquisition Date	1/26/2010 2:40:27 PM
Sample	yao12	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
1085.16271	1	C 72 H 29 O 12	100.00	1085.16535	2.6	2.4	11.7	58.5	even	ok

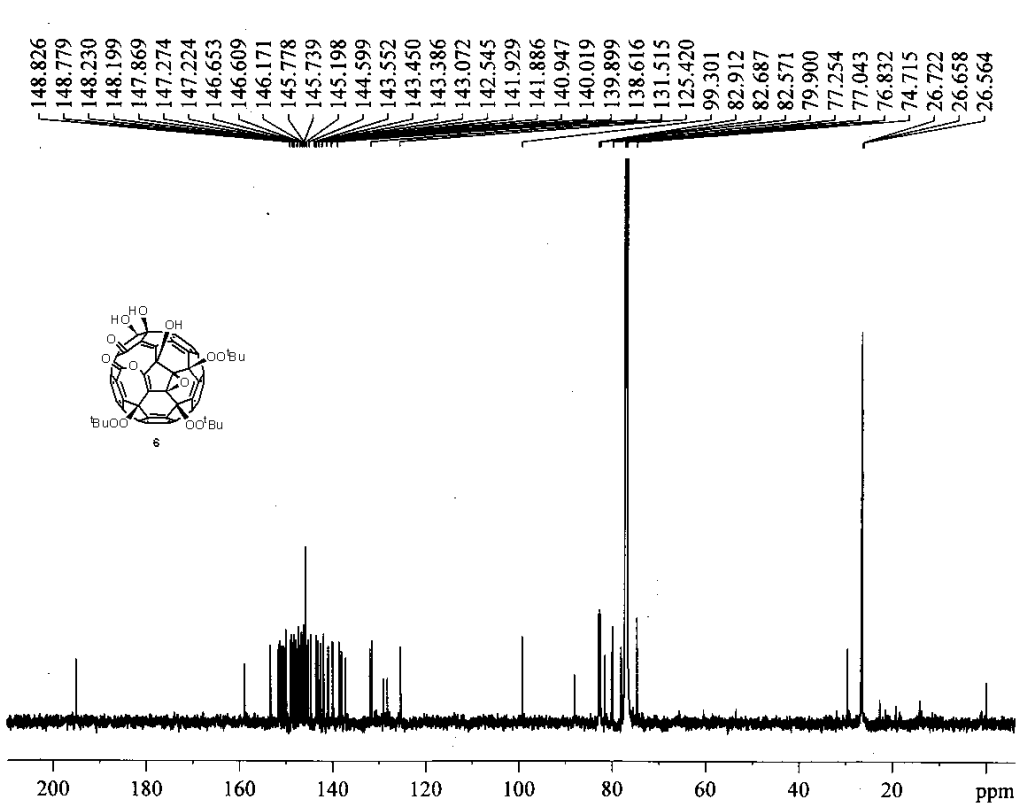
Bruker Compass DataAnalysis 4.0 printed: 1/26/2010 2:45:48 PM Page 1 of 1

Ganliangbing CDCl3 1H-NMR
Yao
20080305



NAME	1h
EXPNO	81
PROCNO	1
Date_	20080310
Time	16.54
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219786 sec
RG	181
DW	27.800 usec
DE	6.00 usec
TE	294.1 K
D1	5.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.33 usec
PL1	-1.00 dB
PL1W	31.62277603 W
SFO1	600.1348010 MHz
SI	32768
SF	600.1300211 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Gan Liangbing 13C-NMR 20080310
CdCl3 Yao

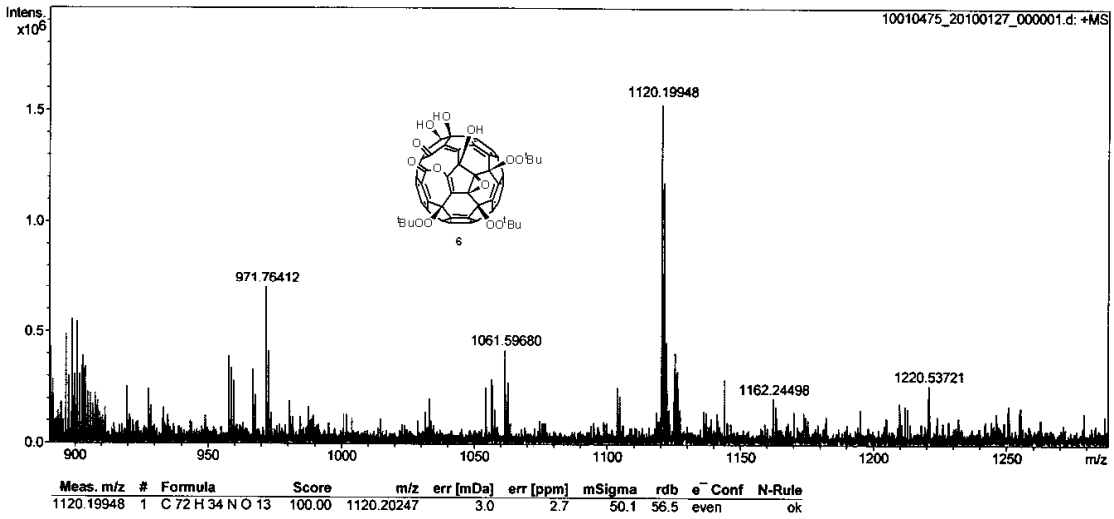


NAME	13c
EXPNO	54
PROCNO	1
Date_	20080310
Time	17.33
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	zgig30
TD	65536
SOLVENT	CDCl3
NS	18432
DS	0
SWH	37593.984 Hz
FIDRES	0.573639 Hz
AQ	0.8716921 sec
RG	13004
DW	13.300 usec
DE	6.50 usec
TE	294.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	14.50 usec
PL1	-2.00 dB
PL1W	119.43215179 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	88.00 usec
PL2	-1.00 dB
PL12	19.40 dB
PL2W	31.62277603 W
PL12W	0.28840318 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028090 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

Peking University Mass Spectrometry Sample Analysis Report

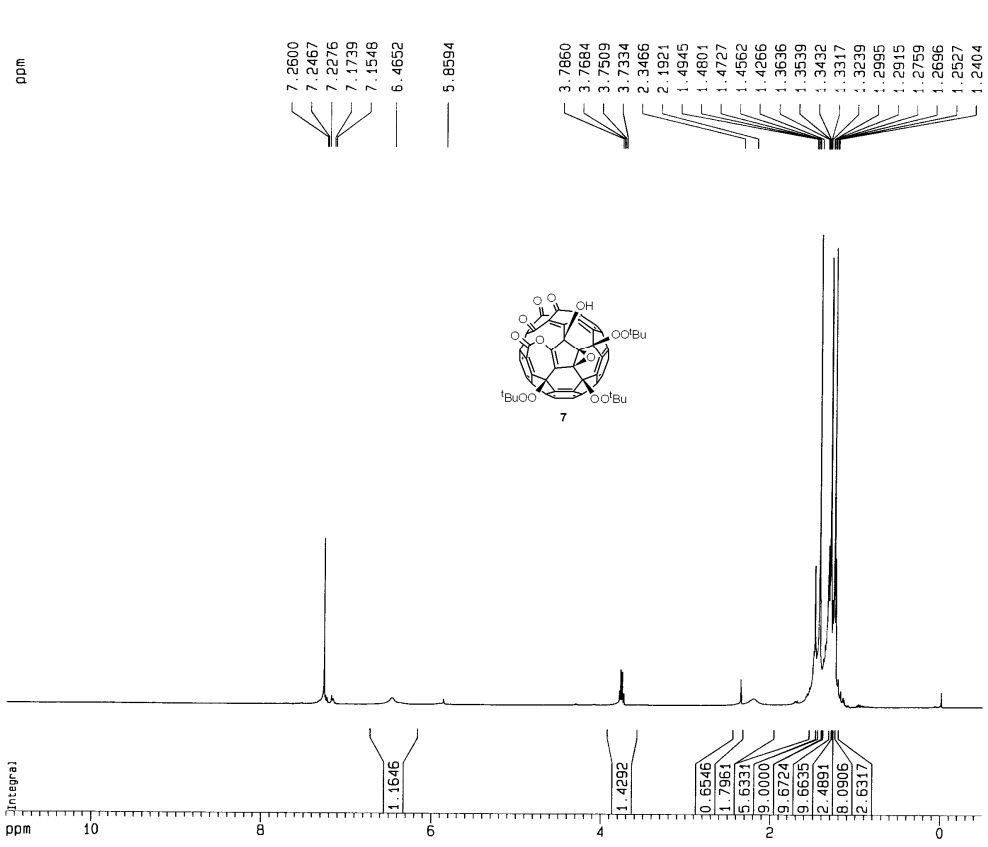
Analysis Info

Analysis Name	10010475_20100127_000001.d	Acquisition Date	1/27/2010 3:50:00 PM
Sample	yao	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
1120.19948	1	C 72 H 34 N O 13	100.00	1120.20247	3.0	2.7	50.1	56.5	even	ok

Bruker Compass DataAnalysis 4.0 printed: 1/27/2010 3:52:43 PM Page 1 of 1



F2 - Acquisition Parameters

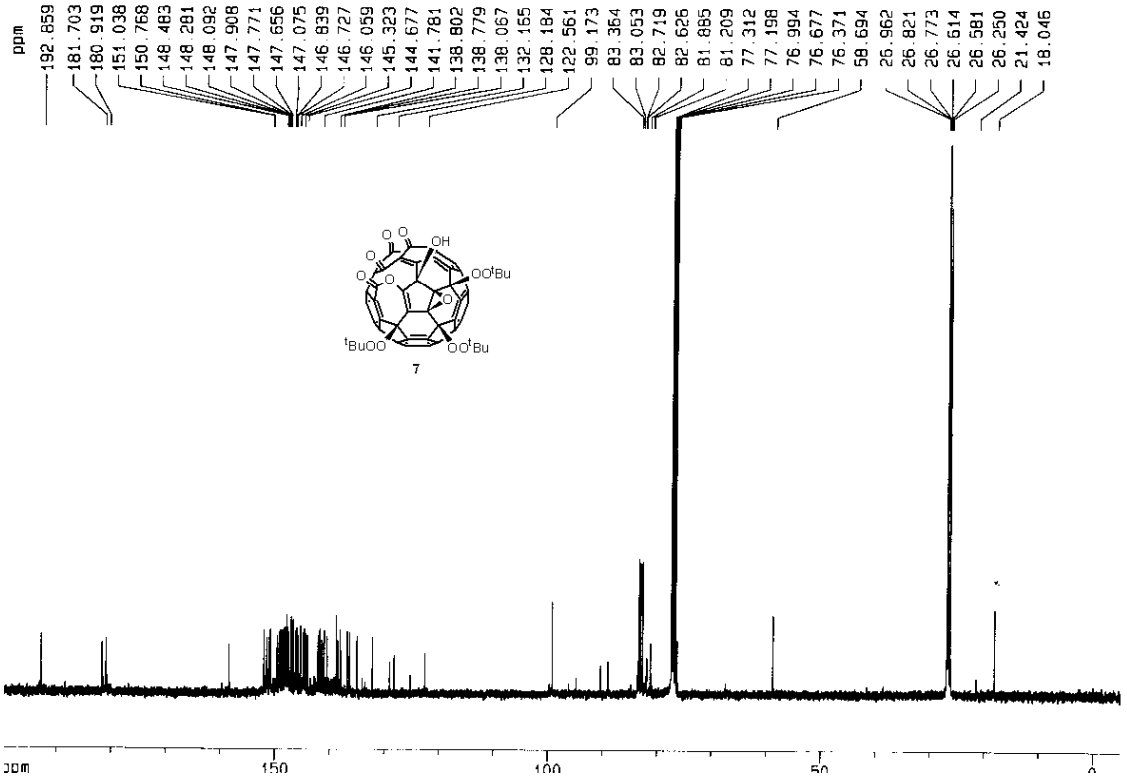
Date_	20080519
Time	11.15
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	360
DW	69.000 usec
DE	98.57 usec
TE	300.0 K
D1	1.50000000 sec
P1	3.00 usec
DE	98.57 usec
SFO1	400.1318844 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	400.1300091 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	11.000 ppm
F1	4401.43 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.57500 ppm/cm
HZCM	230.07474 Hz/cm

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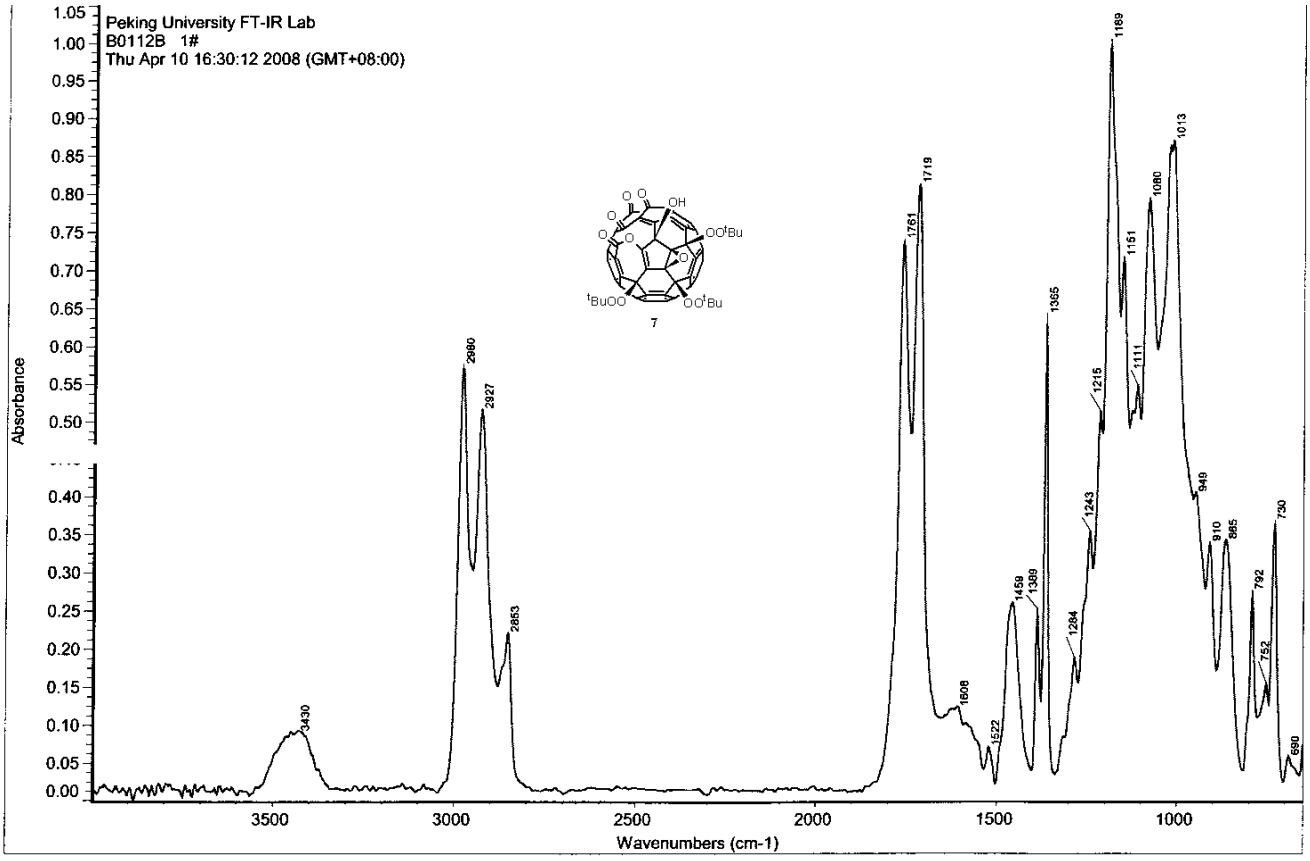
Current Data Parameters
NAME                  c38664
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20080519
Time                  11.19
INSTRUM              ARX400
PROBHD     5 mm Multinu
PULPROG               zgdc
TD                   32768
SOLVENT              CDCl3
NS                    3553
DS                       2
SWH           25000.000 Hz
FIDRES          0.762939 Hz
AQ            0.6554100 sec
RG                    8192
DW              20.000 usec
DE              25.00 usec
TE                300.0 K
D12           0.00002000 sec
DL5             22.20 dB
CPDPRG              waltz16
P31             100.00 usec
D1            2.00000000 sec
P1              2.50 usec
DE              25.00 usec
SFO1         100.6233680 MHz
NUCLEUS               13C
D11           0.03000000 sec

F2 - Processing parameters
SI                   32768
SF          100.6127750 MHz
WDW                     EM
SSB                      0
LB                    0.50 Hz
GB                       0
PC                    1.00

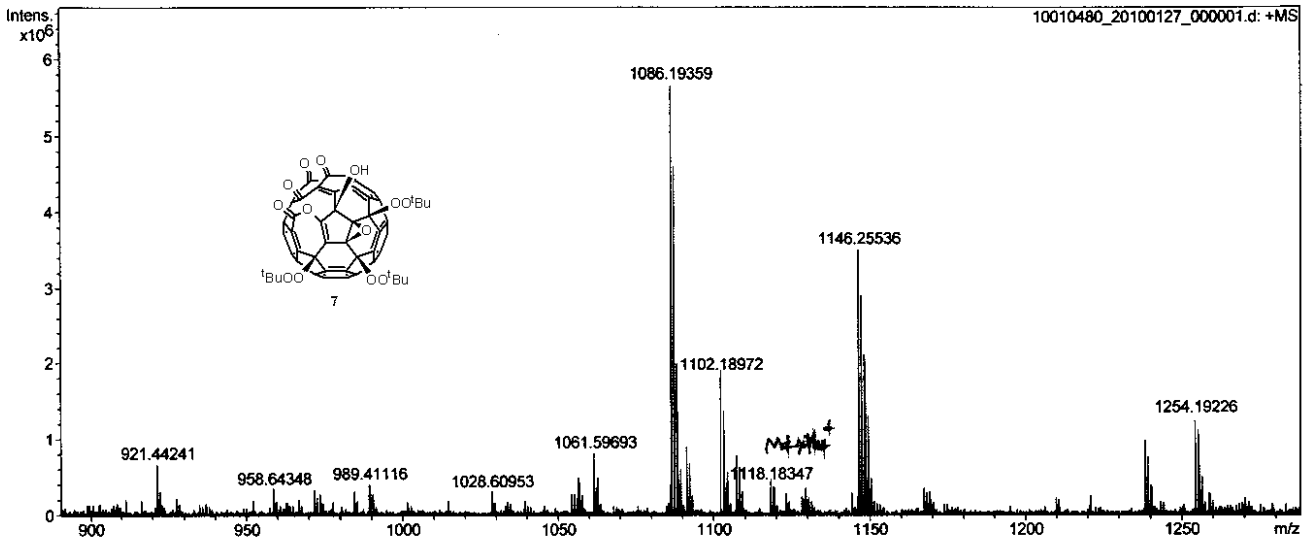
1D NMR plot parameters
CX                   20.00 cm
F1P                 200.000 ppm
F1                 20122.56 Hz
F2P                 -5.000 ppm
F2                 -503.06 Hz
PPMCM              10.25000 ppm/cm
HZCM             1031.28101 Hz/cm
```



Peking University Mass Spectrometry Sample Analysis Report

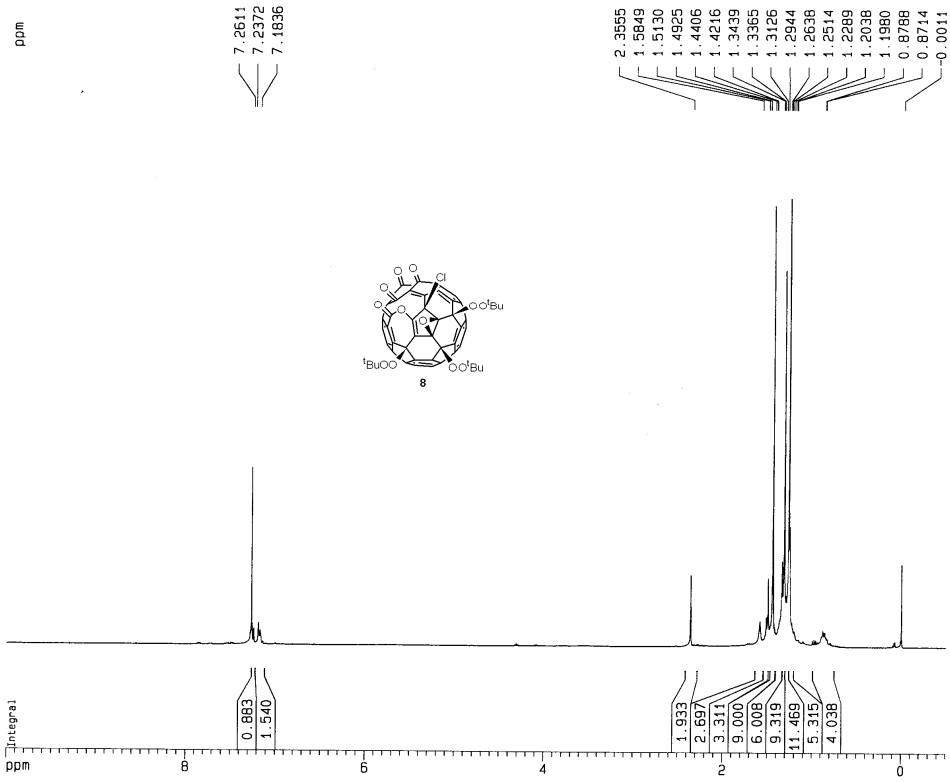
Analysis Info

Analysis Name	10010480_20100127_000001.d	Acquisition Date	1/27/2010 3:54:27 PM
Sample	yao1	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
1086.19359	1	C 72 H 32 N O 11	100.00	1086.19699	3.4	3.1	16.4	57.5	even	ok
1102.18972	1	C 72 H 32 N O 12	100.00	1102.19190	2.2	2.0	47.0	57.5	even	ok
1118.18347	1	C 72 H 32 N O 13	100.00	1118.18682	3.3	3.0	182.7	57.5	even	ok

Bruker Compass DataAnalysis 4.0 printed: 1/27/2010 3:57:21 PM Page 1 of 1



F2 - Acquisition Parameters

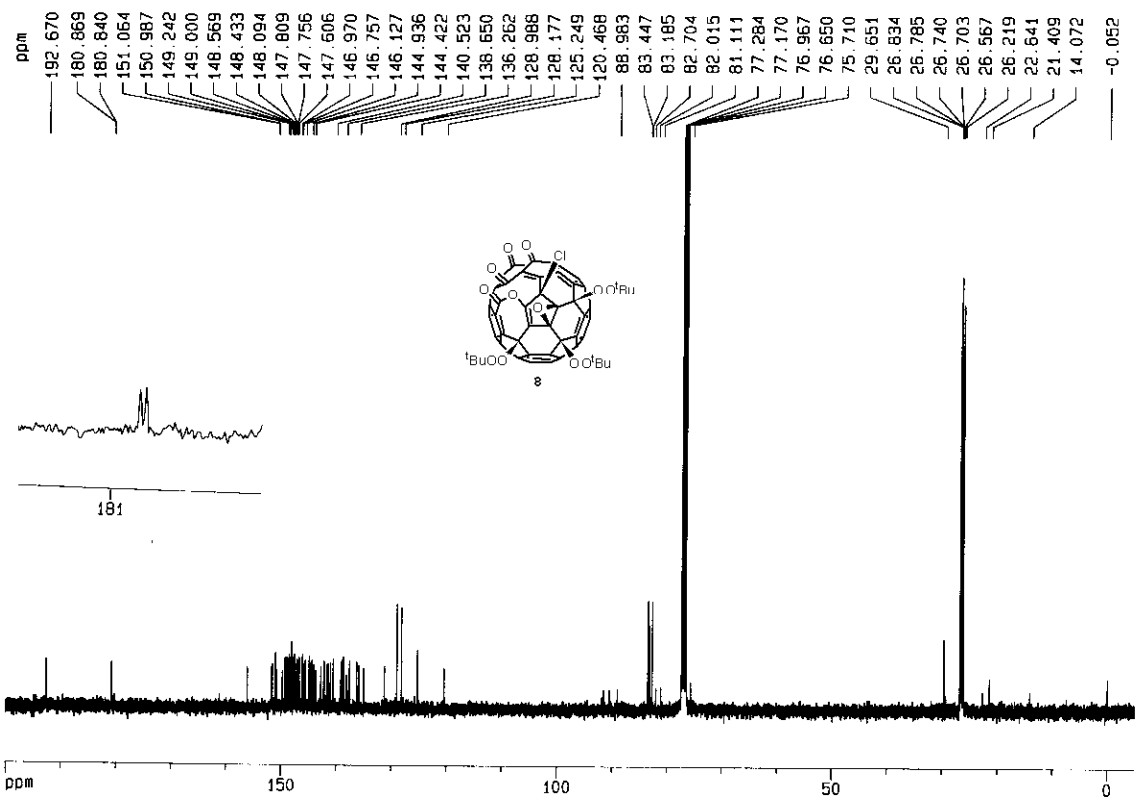
Date_	20080428
Time	14.21
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	1024
DW	69.000 usec
DE	98.57 usec
TE	300.0 K
D1	1.50000000 sec
P1	3.00 usec
DE	98.57 usec
SFO1	400.1318844 MHz
NUCLEUS	1H

F2 - Processing parameters

SI	16384
SF	400.1300096 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.52500 ppm/cm
HZCM	210.06825 Hz/cm



Current Data Parameters

NAME	c38528
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

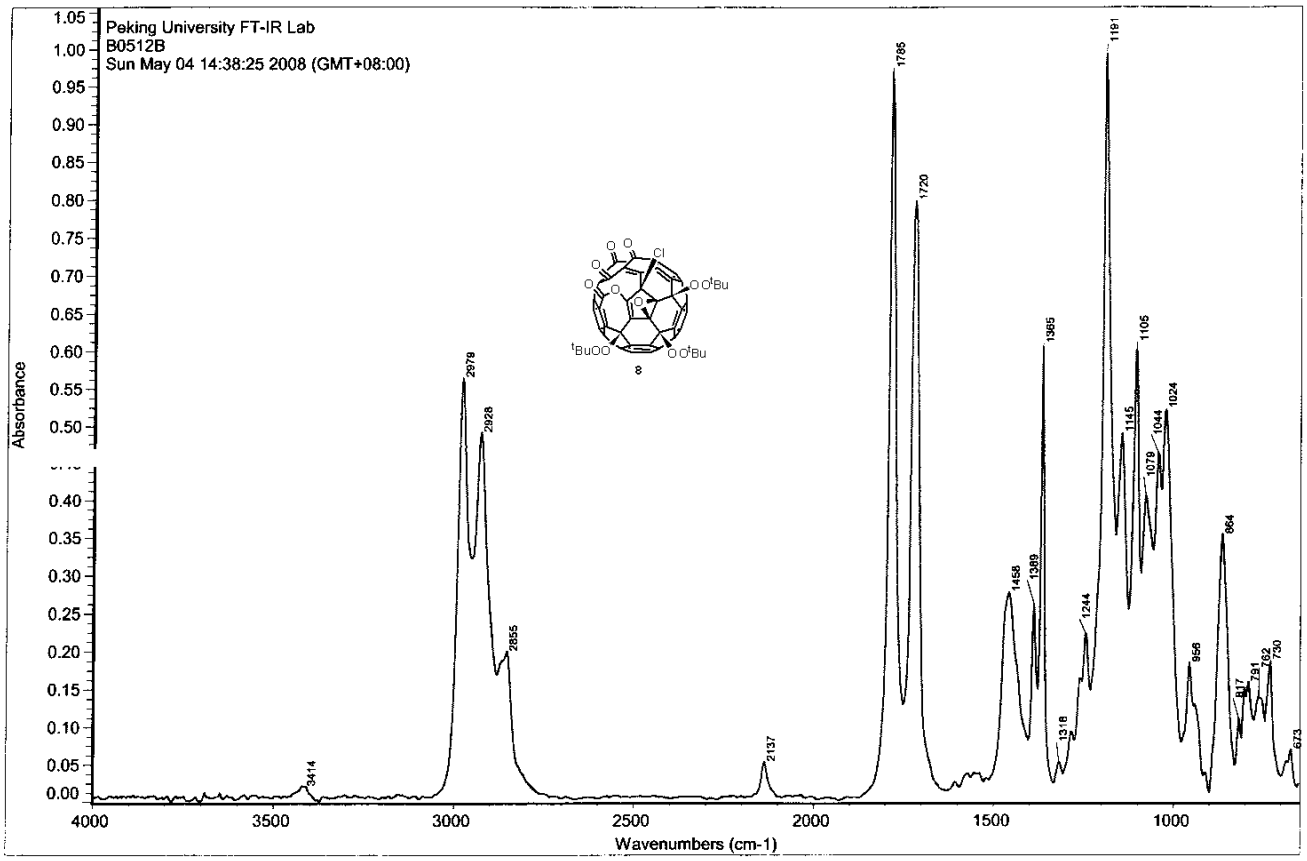
Date_	20080429
Time	10.52
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zgdc
TD	32768
SOLVENT	CDCl3
NS	4196
DS	2
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554100 sec
RG	8192
DW	20.000 usec
DE	25.00 usec
TE	300.0 K
D12	0.00002000 sec
DL5	22.20 dB
CPDPRG	waltz16
P31	100.00 usec
D1	2.00000000 sec
P1	2.50 usec
DE	25.00 usec
SFO1	100.6233680 MHz
NUCLEUS	13C
D11	0.03000000 sec

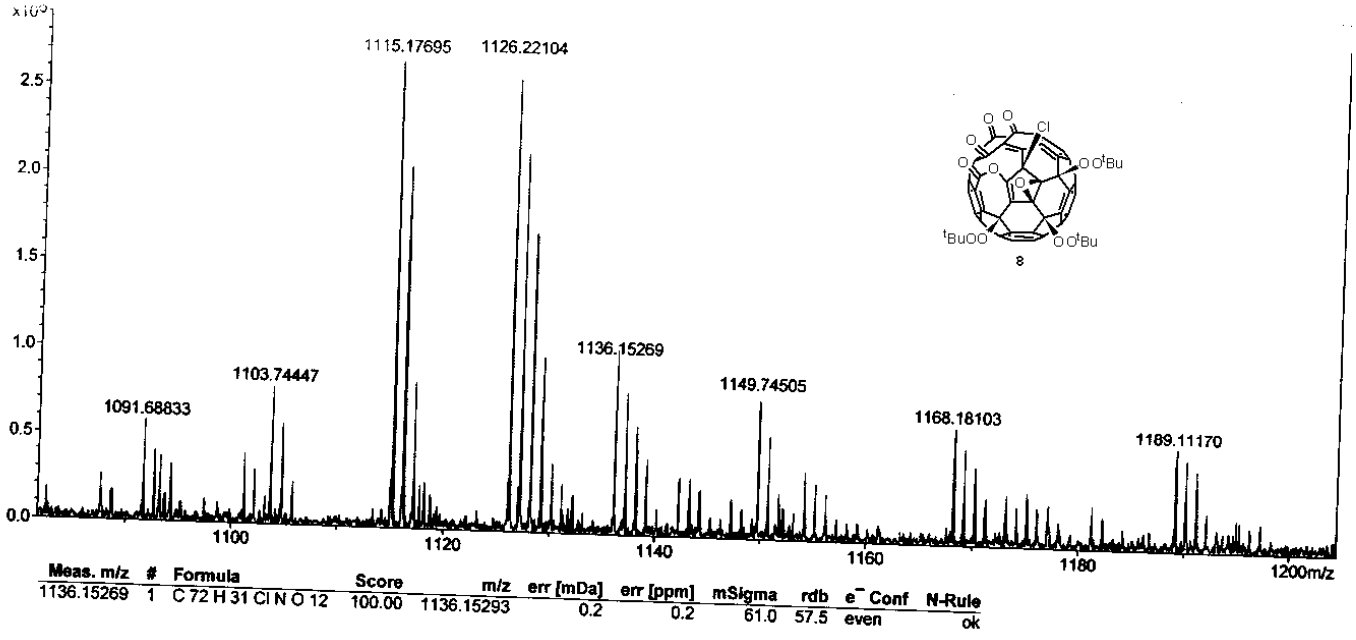
F2 - Processing parameters

SI	32768
SF	100.6127750 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

1D NMR plot parameters

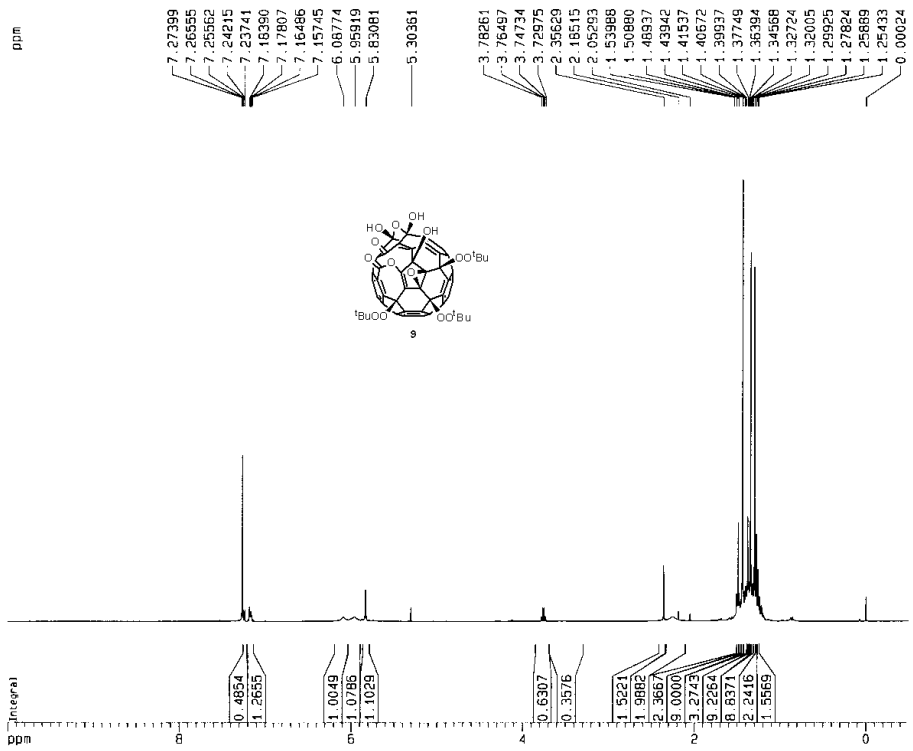
CX	20.00 cm
F1P	200.000 ppm
F1	20122.56 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	10.25000 ppm/cm
HZCM	1031.28101 Hz/cm





Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
1136.15269	1	C 72 H 31 Cl N O 12	100.00	1136.15293	0.2	0.2	61.0	57.5	even	ok

Bruker Compass DataAnalysis 4.0 printed: 1/29/2010 4:37:06 PM Page 1 of 1



9

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EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080520
Time               8.09
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PROBHD     5 mm Multinu
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   64
DS                    0
SWH            7246.377 Hz
FIDRES        0.221142 Hz
AQ            2.2610421 sec
RG                  512
DW               69.000 usec
DE               98.57 usec
TE               300.0 K
D1            1.50000000 sec
P1                3.00 usec
DE               98.57 usec
SFO1         400.1318844 MHz
NUCLEUS              1H

F2 - Processing parameters
SI                16384
SF          400.1300082 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 4.00

1D NMR plot parameters
CX                20.00 cm
F1P               10.000 ppm
F1              4001.30 Hz
F2P               -0.500 ppm
F2              -200.07 Hz
PPMCM          0.52500 ppm/cm
HZCM          210.06825 Hz/cm
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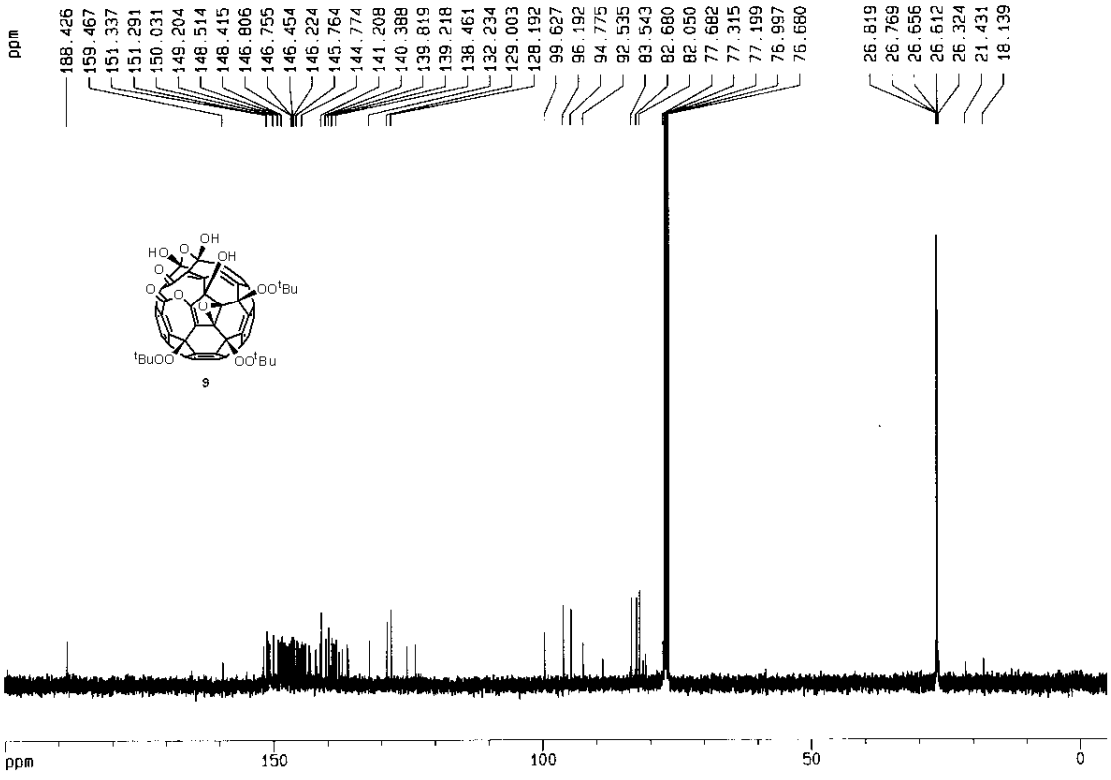

9

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Current Data Parameters
NAME              c38666
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
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Time               9.19
INSTRUM          ARX400
PROBHD     5 mm Multinu
PULPROG          zgdc
TD                32768
SOLVENT           CDCl3
NS                 1129
DS                    2
SWH           25000.000 Hz
FIDRES        0.762939 Hz
AQ            0.6554100 sec
RG                 8192
DW               20.000 usec
DE               25.00 usec
TE               300.0 K
D12           0.00002000 sec
DL5               22.20 dB
CPDPRG           waltz16
P31              100.00 usec
D1            2.00000000 sec
P1                2.50 usec
DE               25.00 usec
SFO1         100.6233680 MHz
NUCLEUS              13C
D11           0.03000000 sec

F2 - Processing parameters
SI                32768
SF          100.6127735 MHz
WDW                  EM
SSB                   0
LB                 0.50 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
F1P              200.000 ppm
F1             20122.55 Hz
F2P               -5.000 ppm
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HZCM          1031.28101 Hz/cm
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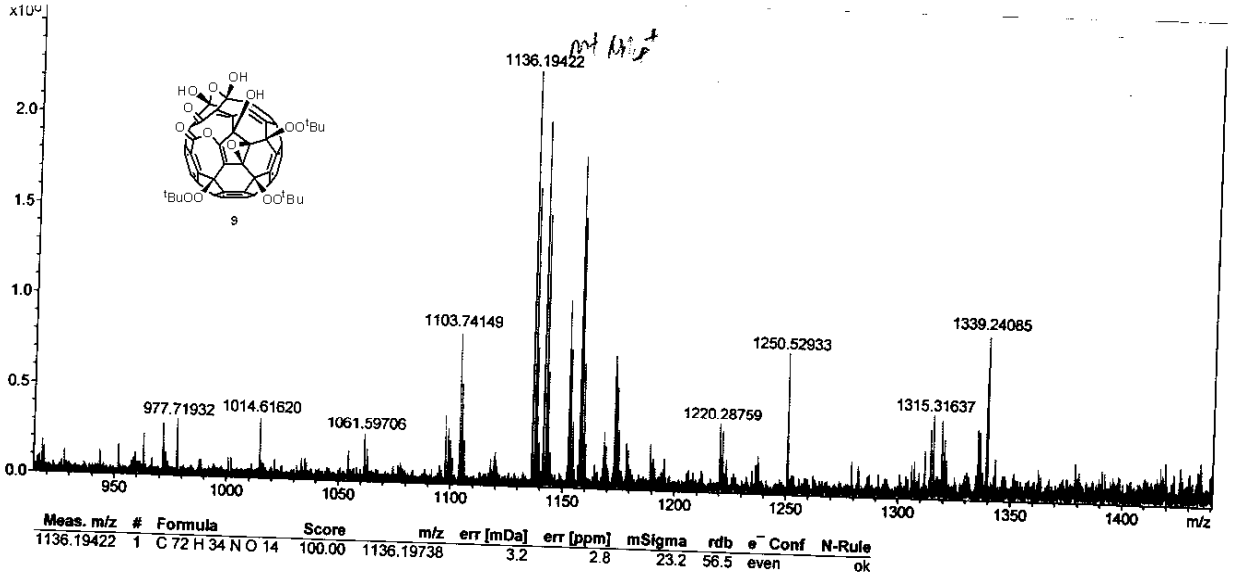
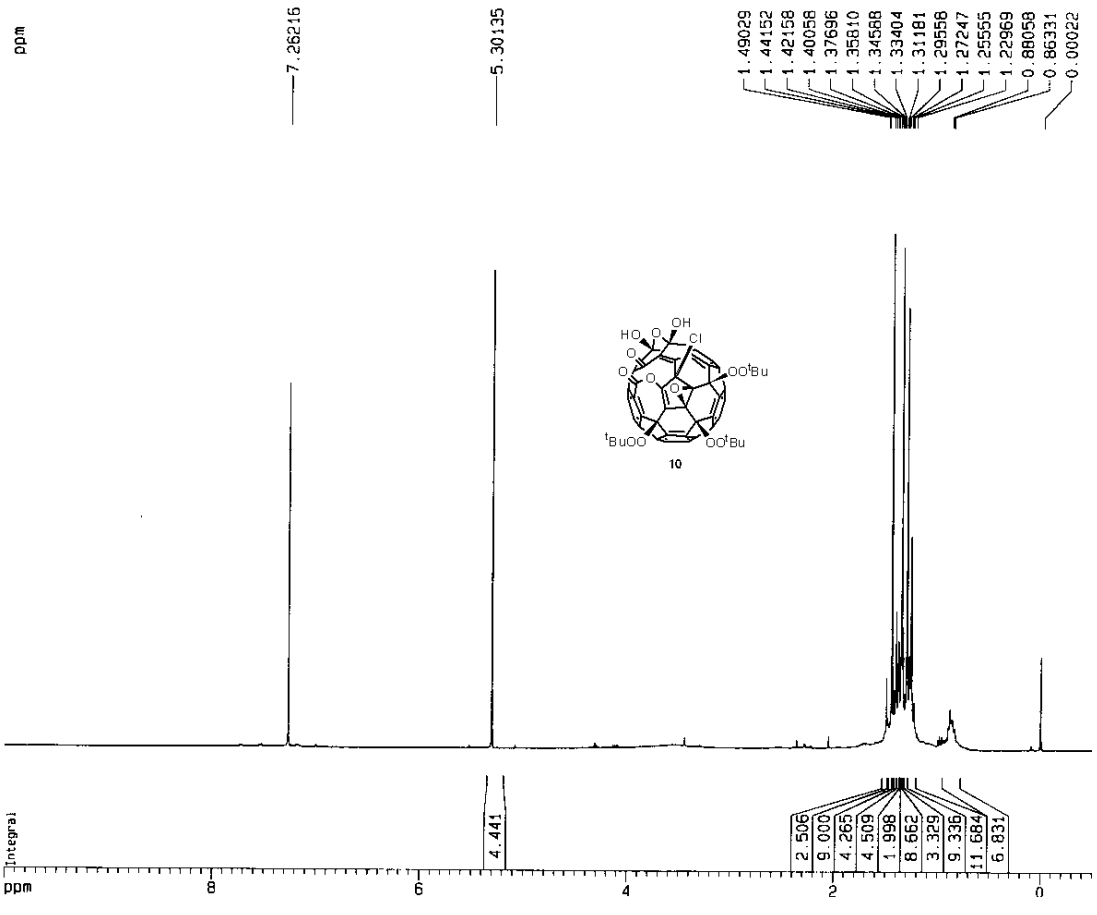


Bruker Compass DataAnalysis 4.0 printed: 1/29/2010 4:30:45 PM Page 1 of 1



Time	15.41
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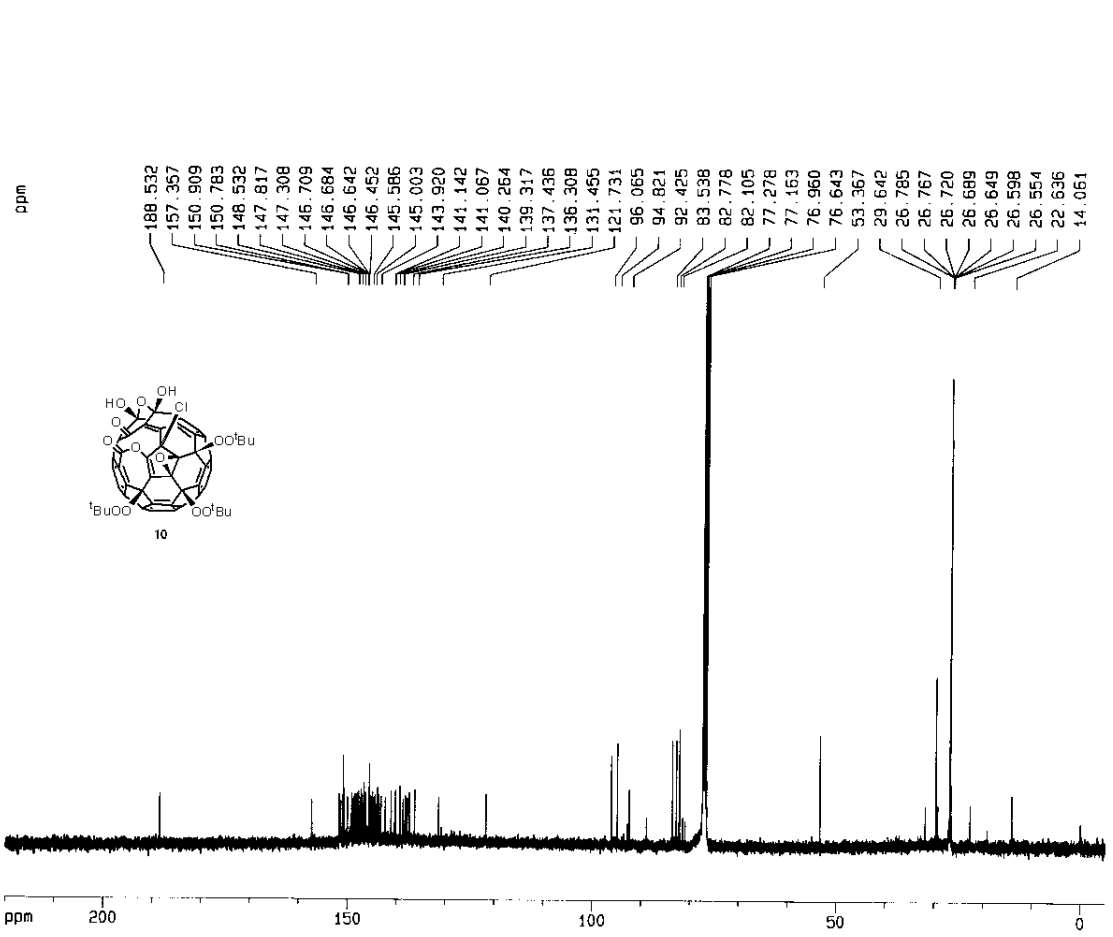
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	7246.377 Hz
FIDRES	0.221142 Hz
AQ	2.2610421 sec
RG	1430
DW	69.000 usec
DE	98.57 usec
TE	300.0 K
D1	1.50000000 sec
P1	3.00 usec
DE	98.57 usec
SFO1	400.1318844 MHz
NUCLEUS	1H
F2 - Processing parameters	
SI	16384
SF	400.1300087 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	4.00
1D NMR plot parameters	
CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.52500 ppm/cm
HZCM	210.06825 Hz/cm

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Current Data Parameters
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EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
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Time                  15.47
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PROBHD     5 mm Multinu
PULPROG              zgdc
TD                    32768
SOLVENT               CDCl3
NS                    20000
DS                        2
SWH               25000.000 Hz
FIDRES            0.762939 Hz
AQ                0.6554100 sec
RG                     8192
DW                  20.000 usec
DE                  25.00 usec
TE                  300.0 K
D12               0.00002000 sec
DL5                  22.20 dB
CPDPRG              waltz16
P31                 100.00 usec
D1                2.00000000 sec
P1                   2.50 usec
DE                  25.00 usec
SF01           100.6233680 MHz
NUCLEUS                 13C
D11               0.03000000 sec

F2 - Processing parameters
SI                    32768
SF             100.6127750 MHz
WDW                      EM
SSB                       0
LB                   0.50 Hz
GB                        0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
F1P                220.000 ppm
F1                 22134.81 Hz
F2P                 -5.000 ppm
F2                 -503.07 Hz
PPMCM              11.25000 ppm/cm
HZCM             1131.89380 Hz/cm
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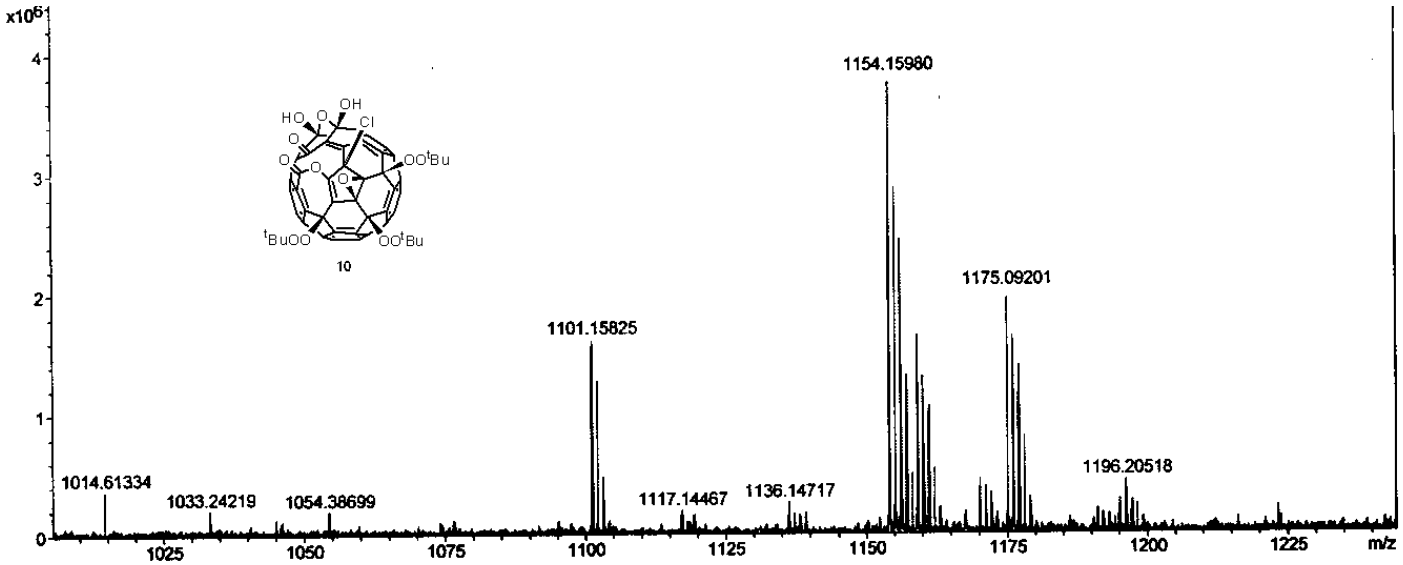

Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
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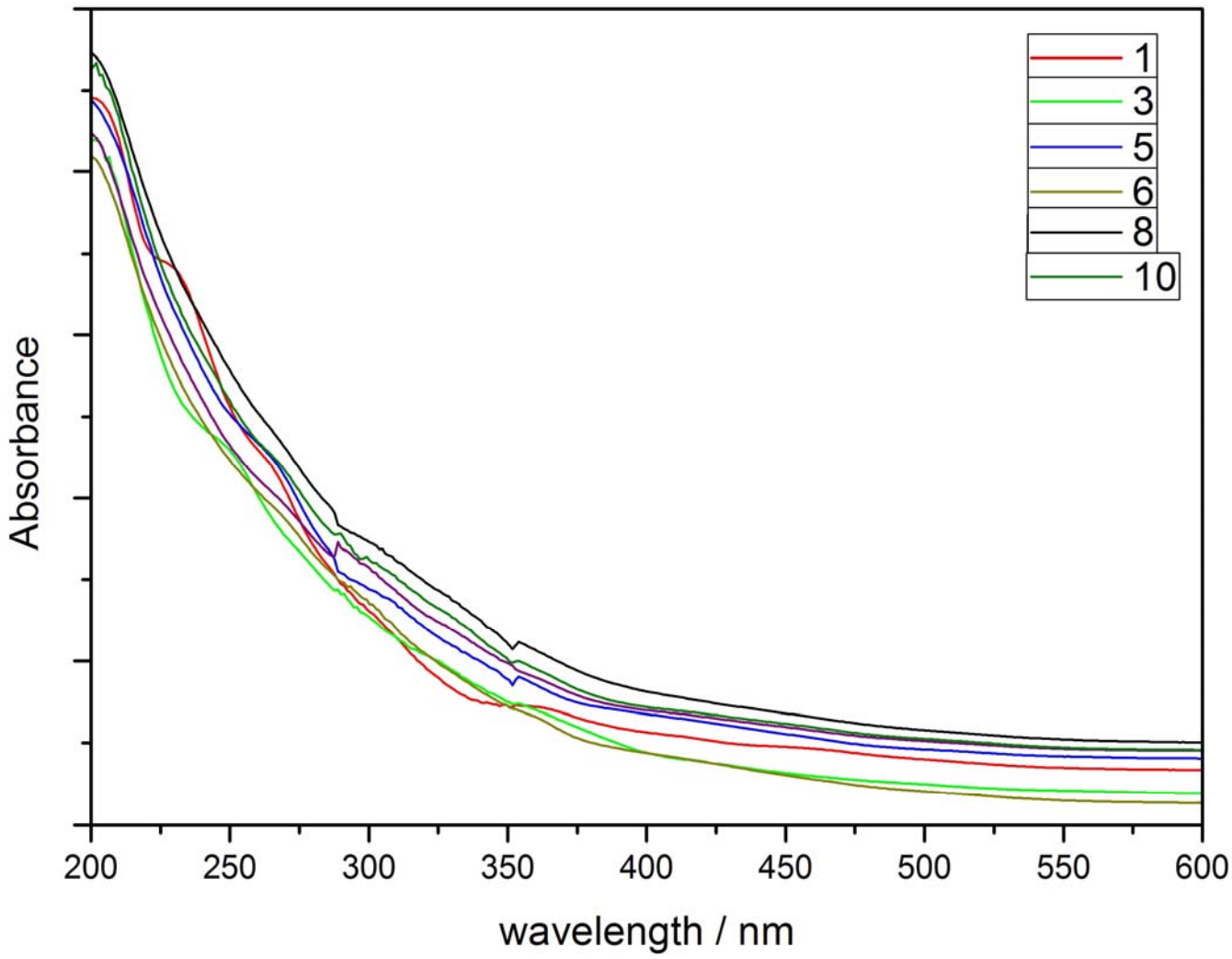

UV-Vis spectra of compounds 1, 3, 5, 6, 8 and 10 in $CHCl_3$