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Supporting Information

Palladium-catalysed heteroannulation of [60]fullerene with *N*-benzyl sulfonamides and subsequent functionalisation

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General procedure for the synthesis of C_{60}-fused tetrahydroisoquinolines 2a-2h: To a solution of C_{60} (36.0 mg, 0.05 mmol) in *o*-dichlorobenzene (4 mL) was sequentially added *N*-benzyl sulfonamide **1a** (**1b-1h**, 0.25 mmol), $K_2S_2O_8$ (67.5 mg, 0.25 mmol), mesitylenesulfonic acid dihydrate (24.6 mg, 0.10 mmol) and $Pd(OAc)_2$ (1.1 mg, 0.005 mmol; 2.2 mg, 0.01 mmol for **1d**). The reaction mixture was stirred at 100 °C (80 °C for **1d**) for a desired time (monitored by TLC to prevent overreaction). The reaction solution was filtered through a silica gel plug to remove any insoluble material. After evaporation *in vacuo*, the residue was separated on a silica gel column with carbon disulfide as the eluent to give unreacted C_{60}, subsequent elution with carbon disulfide/dichloromethane to give product **2a** (**2b-2h**).

General procedure for the synthesis of C_{60}-fused indanes 3a-3h: To a solution of C_{60}-fused tetrahydroisoquinoline **2a** (**2b-2h**, 0.0125 mmol) in *o*-dichlorobenzene (4 mL) was added mesitylenesulfonic acid dihydrate (8.9 mg, 0.0375 mmol). After being stirred at 100 °C for a designated time (monitored by TLC to prevent overreaction), the reaction mixture was filtered through a silica gel plug in order to remove any insoluble material and evaporated *in vacuo*. The residue was separated on a silica gel column with carbon disulfide/dichloromethane as the eluent to give unreacted C_{60}-fused tetrahydroisoquinoline **2a** (**2b-2h**) and C_{60}-fused indane **3a** (**3b-3h**).

Synthesis of C_{60}-fused indanes 4a and 4b: To a solution of C_{60}-fused fulleroindane **3a** (14.7 mg, 0.015 mmol) in a mixture solvent of *o*-dichlorobenzene (4.5 mL)/benzene or mesitylene (1.5 mL) was added $FeCl_3$ (7.3 mg, 0.045 mmol). The reaction mixture was stirred in a 15 mL sealed tube at 130 °C for 1 h. The reaction solution was filtered through a silica gel plug to remove any insoluble material. After evaporation *in vacuo*, the product was separated on a silica gel column with carbon disulfide as the eluent to afford C_{60}-fused indane **4a** (9.0 mg, 68%) or C_{60}-fused indane **4b** (11.4 mg, 82%).

Synthesis of C_{60}-fused indane 4c: To a solution of C_{60}-fused fulleroindane **3a** (14.7 mg, 0.015 mmol) in *o*-dichlorobenzene (6 mL) was added $FeCl_3$ (7.3 mg, 0.045 mmol) and Et_3SiH (24.0 μL, 0.15 mmol). The reaction mixture was stirred in a 15 mL sealed tube at 130 °C for 12 h. After the reaction solution was evaporated *in vacuo*, the product was separated on a silica gel column with carbon disulfide as the eluent to afford C_{60}-fused indane **4c** (8.5 mg, 70%).

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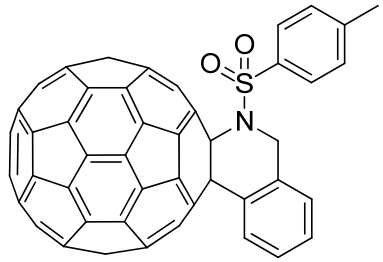
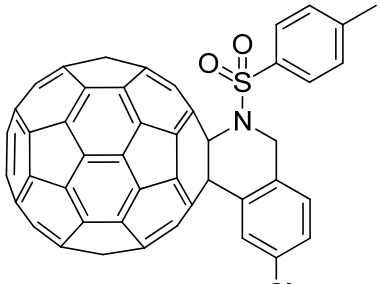


2a

Spectral data of **2a**: ^{1}H NMR (300 MHz, $CS_2/CDCl_3$) δ 8.14 (d, 7.5 Hz, 1H), 7.74 (d, J = 7.5 Hz, 1H), 7.69 (d, J = 8.3 Hz, 2H), 7.54 (td, J = 7.5, 1.0 Hz, 1H), 7.47 (td, J = 7.5, 1.5 Hz, 1H), 7.08 (d, J = 8.3 Hz, 2H), 6.02 (d, J = 16.4 Hz, 1H), 5.77 (d, J = 16.4 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR [75 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 158.76, 151.86, 150.70, 148.20, 147.98, 147.49, 147.09, 146.36 (2C), 146.28, 145.97 (3C), 145.94 (2C), 145.69, 145.60, 145.37 (3C), 145.31, 145.20, 145.16, 145.06, 144.99, 144.62, 144.46, 144.42, 144.32 (2C), 143.89, 143.28, 142.85, 142.79 (aryl *C*), 142.68, 142.55, 142.53, 142.47, 142.37, 142.12 (4C), 142.01, 141.92, 141.61, 141.39 (2C), 141.09, 140.27, 140.05 (2C), 139.79, 138.92, 138.38 (aryl *C*), 138.37, 138.07, 138.04, 136.68 (aryl *C*), 136.65, 136.19, 135.84 (aryl *C*), 131.66, 128.82 (2C, aryl *C*), 128.74 (aryl *C*), 128.24 (2C, aryl *C*), 127.81 (aryl *C*), 127.62 (aryl *C*), 127.51 (aryl *C*), 79.99 (sp^3-*C* of C_{60}), 63.74 (sp^3-*C* of C_{60}), 48.84 (*C*H_2), 21.42 (*C*H_3); FT-IR ν/cm^{-1} (KBr) 2919, 1505, 1434, 1333, 1154, 1083, 1037, 888, 808, 745, 667, 524; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 257 (4.96), 319 (4.55), 434 (3.38), 693 (2.39); (-)ESI MS m/z calcd for $C_{74}H_{13}NO_2S$ [M^-] 979.0667, found 979.0673.



2b

Spectral data of **2b**: ^{1}H NMR (300 MHz, $CS_2/CDCl_3$) δ 8.07 (d, J = 1.8 Hz, 1H), 7.74 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.1 Hz, 1H), 7.51 (dd, J = 8.1, 1.8 Hz, 1H), 7.12 (d, J = 8.4 Hz, 2H), 5.99 (d, J = 16.4 Hz, 1H), 5.76 (d, J = 16.4 Hz, 1H), 2.36 (s, 3H); ^{13}C NMR [75 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 157.95, 151.06, 150.29, 148.17, 148.10, 147.61, 146.76, 146.46 (2C), 146.41, 146.12, 146.08 (4C), 145.77, 145.62, 145.47, 145.44 (2C), 145.32, 145.28, 145.21, 145.15 (2C), 144.87, 144.54, 144.44, 144.42, 144.38, 143.82, 143.25 (aryl *C*), 143.13, 142.96, 142.77, 142.68, 142.63, 142.57, 142.49, 142.21 (3C), 142.09, 142.00 (2C), 141.72, 141.52, 141.48, 141.23, 140.31, 140.21, 140.16, 139.93, 139.22, 138.77 (aryl *C*), 138.27, 138.05 (aryl *C*), 137.92, 136.51, 136.48, 134.95 (aryl *C*), 134.28 (aryl *C*), 131.91, 129.08 (aryl *C*), 129.01 (2C, aryl *C*), 128.24 (2C, aryl *C*), 127.69 (aryl *C*), 127.66 (aryl *C*), 79.99 (sp^3-*C* of C_{60}), 63.43 (sp^3-*C* of C_{60}), 48.49 (*C*H_2), 21.47 (*C*H_3); FT-IR ν/cm^{-1} (KBr) 2921,

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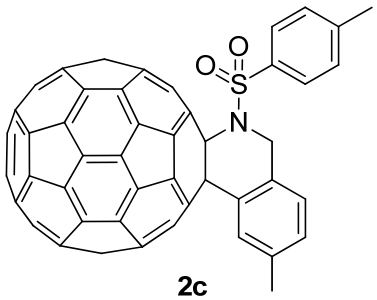
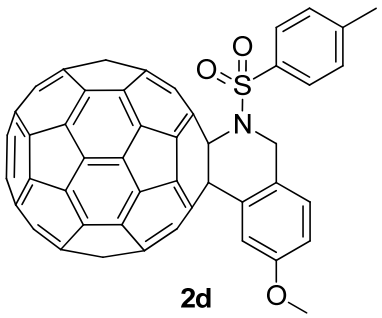
1510, 1345, 1160, 1084, 1039, 813, 668, 563, 526; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 257 (4.99), 317 (4.58), 435 (3.33), 693 (2.44); (-)ESI MS m/z calcd for $C_{74}H_{12}{}^{35}ClNO_2S$ [M^-] 1013.0277, found 1013.0282.



2c

Spectral data of **2c**: ^{1}H NMR (300 MHz, $CS_2/CDCl_3$) δ 7.92 (s, 1H), 7.73 (d, J = 7.8 Hz, 2H), 7.65 (d, J = 7.6 Hz, 1H), 7.36 (d, J = 7.6 Hz, 1H), 7.09 (d, J = 7.8 Hz, 2H), 6.01 (d, J = 16.4 Hz, 1H), 5.76 (d, J = 16.4 Hz, 1H), 2.45 (s, 3H), 2.34 (s, 3H); ^{13}C NMR [75 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 158.91, 151.96, 150.87, 148.47, 148.03, 147.54, 147.23, 146.40 (2C), 146.33, 146.02 (3C), 145.99 (2C), 145.72, 145.66, 145.41 (4C), 145.24, 145.20, 145.11, 145.02, 144.65, 144.52, 144.48, 144.37 (2C), 143.95, 143.41, 142.91, 142.73, 142.72 (aryl *C*), 142.60, 142.58, 142.51, 142.42, 142.25, 142.17 (3C), 142.11, 141.97, 141.65, 141.44 (2C), 141.11, 140.32, 140.13, 140.07, 139.81, 138.96, 138.53 (aryl *C*), 138.46 (aryl *C*), 138.11, 138.08, 136.70, 136.43 (aryl *C*), 136.22, 132.95 (aryl *C*), 131.68, 128.81 (2C, aryl *C*), 128.30 (2C, aryl *C*), 128.27 (aryl *C*), 128.21 (aryl *C*), 127.85 (aryl *C*), 80.04 (sp^3-*C* of C_{60}), 63.80 (sp^3-*C* of C_{60}), 48.73 (*C*H_2), 21.59 (*C*H_3), 21.45 (*C*H_3); FT-IR ν/cm^{-1} (KBr) 2922, 1510, 1343, 1159, 1086, 1035, 810, 665, 562, 523; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 257 (4.98), 318 (4.55), 435 (3.33), 693 (2.50); (-)ESI MS m/z calcd for $C_{75}H_{15}NO_2S$ [M^-] 993.0823, found 993.0819.



2d

Spectral data of **2d**: ^{1}H NMR (300 MHz, $CS_2/CDCl_3$) δ 7.67 (d, J = 8.3 Hz, 2H), 7.60 (d, J = 8.2 Hz, 1H), 7.57 (d, J = 2.4 Hz, 1H), 7.06 (d, J = 8.3 Hz, 2H), 7.01 (dd, J= 8.2, 2.4 Hz), 5.91 (d, J = 16.4 Hz, 1H), 5.68 (d, J = 16.4 Hz, 1H), 3.83 (s, 3H), 2.33 (s, 3H); ^{13}C NMR [75 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 160.00 (aryl *C*), 158.75, 151.80, 150.76, 148.36, 148.11, 147.62, 147.07, 146.46 (2C), 146.38, 146.08 (5C), 145.79, 145.69, 145.47, 145.45, 145.42, 145.36, 145.28 (2C), 145.17, 145.10, 144.73, 144.58, 144.51, 144.42 (2C), 143.94, 143.46, 143.15 (aryl *C*), 142.94, 142.78, 142.63, 142.61, 142.55, 142.47, 142.20 (4C), 142.12,

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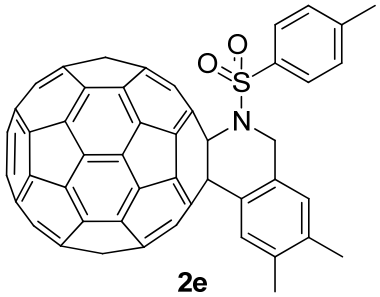
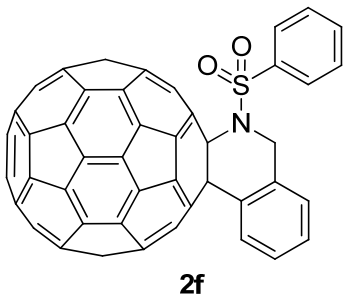
142.01, 141.70, 141.49, 141.47, 141.13, 140.35, 140.17, 140.11, 139.84, 139.11, 138.18 (aryl *C*), 138.18, 138.11, 138.01 (aryl *C*), 136.75, 136.24, 131.87, 128.97 (2C, aryl *C*), 128.82 (aryl *C*), 128.34 (2C, aryl *C*), 128.04 (aryl *C*), 114.80 (aryl *C*), 112.11 (aryl *C*), 79.96 (sp^3-*C* of C_{60}), 63.96 (sp^3-*C* of C_{60}), 55.27 (OCH_3), 48.55 (CH_2), 21.42 (CH_3); FT-IR ν/cm^{-1} (KBr) 2921, 1500, 1436, 1332, 1244, 1154, 1086, 1037, 808, 665, 558, 524; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 257 (5.00), 317 (4.54), 435 (3.38), 692 (2.46); (-)ESI MS m/z calcd for $C_{75}H_{15}NO_3S$ [M^-] 1009.0773, found 1009.0777.



2e

Spectral data of **2e**: ^{1}H NMR (300 MHz, CS_2/$CDCl_3$) δ 7.83 (s, 1H), 7.71 (d, J = 8.3 Hz, 2H), 7.49 (s, 1H), 7.08 (d, J = 8.3 Hz, 2H), 5.95 (d, J = 16.2 Hz, 1H), 5.69 (d, J = 16.2 Hz, 1H), 2.46 (s, 3H), 2.34 (s, 3H), 2.33 (s, 3H); ^{13}C NMR [75 MHz, CS_2/$CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 159.12, 152.04, 150.85, 148.32, 147.97, 147.47, 147.16, 146.32 (2C), 146.23, 145.93 (5C), 145.65, 145.59, 145.32 (4C), 145.12 (2C), 145.03, 144.93, 144.49, 144.45 (2C), 144.28, 144.27, 143.84, 143.38, 142.87 (aryl *C*), 142.82, 142.63, 142.48 (2C), 142.42, 142.32, 142.20, 142.11, 142.07 (3C), 141.88, 141.55, 141.36 (2C), 141.02, 140.23, 140.04 (2C), 139.66, 138.90, 138.25 (aryl *C*), 138.08, 138.01, 137.12 (aryl *C*), 136.71, 136.06, 136.01 (aryl *C*), 133.79 (aryl *C*), 133.06 (aryl *C*), 131.48, 129.07 (aryl *C*), 128.71 (2C, aryl *C*), 128.60 (aryl *C*), 128.24 (2C, aryl *C*), 80.03 (sp^3-*C* of C_{60}), 63.50 (sp^3-*C* of C_{60}), 48.55 (CH_2), 21.34 (CH_3), 19.78 (CH_3), 19.28 (CH_3); FT-IR ν/cm^{-1} (KBr) 2922, 1508, 1443, 1342, 1158, 1080, 1032, 882, 808, 664, 563, 525; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 258 (5.04), 318 (4.63), 437 (3.43), 695 (2.46); (-)ESI MS m/z calcd for $C_{76}H_{17}NO_2S$ [M^-] 1007.0980, found 1007.0983.



2f

Spectral data of **2f**: ^{1}H NMR (300 MHz, CS_2/$CDCl_3$) δ 8.15 (d, 7.8 Hz, 1H), 7.84 (d, J = 7.5 Hz, 2H), 7.76 (d, J = 7.2 Hz, 1H), 7.56 (td, J = 7.5, 1.0 Hz, 1H), 7.48 (td, J = 7.5, 1.5 Hz, 1H), 7.41 (t, J = 7.5 Hz, 1H), 7.30 (t, J = 7.5 Hz, 2H), 6.06 (d, J = 16.5 Hz, 1H), 5.82 (d, J = 16.5 Hz, 1H); ^{13}C NMR [75 MHz, CS_2/$CDCl_3$ with $Cr(acac)_3$ as relaxation

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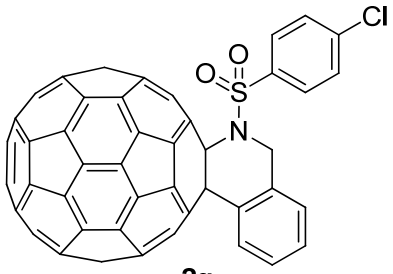
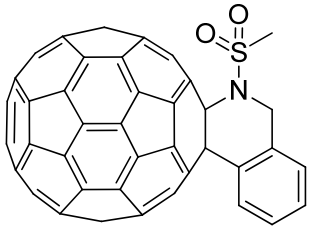
reagent] (all 1C unless indicated) δ 158.77, 151.93, 150.65, 148.11, 148.05, 147.62, 147.17, 146.49 (2C), 146.39, 146.11 (5C), 145.84, 145.69, 145.50 (2C), 145.42, 145.35, 145.32, 145.29, 145.19, 145.11, 144.74, 144.58, 144.54, 144.41 (2C), 143.89, 143.36, 142.98, 142.80, 142.68, 142.66, 142.60, 142.51, 142.24 (4C), 142.12, 142.04, 141.75, 141.52 (2C), 141.22 (aryl *C*), 141.22, 140.37, 140.18, 140.12, 139.91, 139.06, 138.23 (2C), 136.82 (aryl C), 136.82, 136.21, 135.85 (aryl *C*), 132.30 (aryl *C*), 131.77, 129.03 (aryl *C*), 128.34 (2C, aryl *C*), 128.27 (2C, aryl *C*), 127.90 (aryl *C*), 127.78 (aryl *C*), 127.66 (aryl *C*), 80.10 (sp^3-*C* of C_{60}), 63.88 (sp^3-*C* of C_{60}), 48.96 (*C*H_2); FT-IR ν/cm^{-1} (KBr) 2924, 1509, 1439, 1333, 1162, 1085, 1036, 808, 742, 523; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 258 (5.03), 318 (4.62), 434 (3.43), 693 (2.44); (-)ESI MS m/z calcd for $C_{73}H_{11}NO_2S$ [M^-] 965.0510, found 965.0512.



2g

Spectral data of **2g**: ^{1}H NMR (300 MHz, CS_2/$CDCl_3$) δ 8.13 (d, *J* = 7.5 Hz, 1H), 7.76-7.72 (m, 3H), 7.55 (td, *J* = 7.5, 1.2 Hz, 1H), 7.49 (td, *J* = 7.5, 1.5 Hz, 1H), 7.26 (d, *J* = 8.7 Hz, 2H), 6.06 (d, *J* = 16.5 Hz, 1H), 5.78 (d, *J* = 16.5 Hz, 1H); ^{13}C NMR [75 MHz, CS_2/$CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 158.39, 151.74, 150.34, 148.03, 147.93, 147.55, 146.93, 146.41 (2C), 146.32, 146.05 (3C), 146.00 (2C), 145.80, 145.53, 145.45, 145.44, 145.27 (2C), 145.22 (2C), 145.11, 145.02, 144.67, 144.48, 144.43, 144.35 (2C), 143.70, 143.18, 142.91, 142.73, 142.62, 142.58, 142.54, 142.47, 142.19 (3C), 142.14, 141.99, 141.95, 141.71, 141.54, 141.44, 141.11, 140.26, 140.12, 140.04, 139.92, 139.42 (aryl *C*), 139.05, 138.96 (aryl *C*), 138.29, 137.78, 136.68, 136.65 (aryl *C*), 135.89, 135.68 (aryl *C*), 131.66, 129.50 (2C, aryl *C*), 128.98 (aryl *C*), 128.45 (2C, aryl *C*), 127.88 (aryl *C*), 127.74 (aryl *C*), 127.61 (aryl *C*), 80.01 (sp^3-*C* of C_{60}), 63.81 (sp^3-*C* of C_{60}), 49.00 (*C*H_2); FT-IR ν/cm^{-1} (KBr) 2921, 1508, 1336, 1157, 1086, 1036, 889, 810, 748, 619, 525; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 258 (5.00), 319 (4.57), 434 (3.44), 693 (2.58); (-)ESI MS m/z calcd for $C_{73}H_{10}{}^{35}ClNO_2S$ [M^-] 999.0121, found 999.0114.



2h

Spectral data of **2h**: ^{1}H NMR (300 MHz, CS_2/$CDCl_3$) δ 8.41-8.38 (m, 1H), 7.77-7.74

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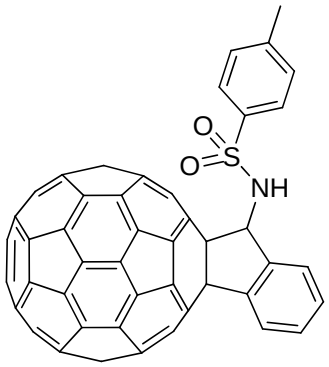
(m, 1H), 7.64–7.56 (m, 2H), 6.02 (d, J = 16.5 Hz, 1H), 5.60 (d, J = 16.5 Hz, 1H), 2.95 (s, 3H); ^{13}C NMR [75 MHz, CS_2/$CDCl_3$ with Cr(acac)$_3$ as relaxation reagent] (all 1C unless indicated) δ 157.86, 151.92, 150.32, 148.91, 148.12, 147.75, 146.73, 146.49 (3C), 146.19 (3C), 146.12 (2C), 145.80, 145.59 (2C), 145.52, 145.38 (4C), 145.19 (2C), 144.91, 144.55 (2C), 144.46, 144.39, 144.04, 143.43, 143.01, 142.79 (2C), 142.63 (3C), 142.49, 142.37 (2C), 142.17, 142.07, 142.01, 141.87, 141.79, 141.60, 141.11, 140.96, 140.35, 140.25, 140.17, 139.25, 139.08, 136.62 (aryl *C*), 136.33, 135.83, 135.68 (aryl *C*), 135.23, 132.21, 129.27 (aryl *C*), 128.27 (aryl *C*), 127.99 (aryl *C*), 127.95 (aryl *C*), 79.97 (sp^3-*C* of C_{60}), 63.54 (sp^3-*C* of C_{60}), 49.06 (*C*H$_2$), 42.36 (*C*H$_3$); FT-IR ν/cm^{-1} (KBr) 2923, 2854, 1505, 1449, 1332, 1152, 1072, 1036, 953, 801, 745, 520; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 257 (4.99), 317 (4.57), 433 (3.37), 692 (2.37); (-)ESI MS m/z calcd for $C_{68}H_9NO_2S$ [M$^-$] 903.0354, found 903.0351.



3a

Spectral data of **3a**: ^{1}H NMR (400 MHz, CS_2/DMSO-d_6/$CDCl_3$) δ 9.32 (d, J = 9.6 Hz, 1H), 8.03 (d, J = 7.6 Hz, 1H), 7.78 (d, J = 8.0 Hz, 2H), 7.59 (t, J = 7.6 Hz, 1H), 7.51 (t, J = 7.5, 0.9 Hz, 1H), 7.45 (d, J = 7.6, 1.5 Hz, 1H), 7.15 (d, J = 8.0 Hz, 2H), 6.75 (d, J = 9.6 Hz, 1H), 2.36 (s, 3H); D_2O exchange experiment: ^{1}H NMR (400 MHz, CS_2/DMSO-d_6/$CDCl_3$) δ 8.31 (d, J = 9.6 Hz, 0.18H, *NH*), 8.07 (d, J = 7.6 Hz, 1H), 7.82 (d, J = 8.0 Hz, 2H), 7.65-7.54 (m, 3H), 7.15 (d, J = 8.0 Hz, 2H), 6.81 (m, 1H), 2.36 (s, 3H); ^{13}C NMR [75 MHz, CS_2/DMSO-d_6/$CDCl_3$ with Cr(acac)$_3$ as relaxation reagent] (all 1C unless indicated) δ 156.38, 154.57, 152.30, 152.94, 146.69, 146.59, 146.58, 145.87, 145.57, 145.54, 145.53, 145.48, 145.40, 145.30, 145.28 (3C), 145.11, 145.08, 144.88, 144.65, 144.61, 144.52 (3C), 144.48, 144.40, 144.36, 143.91, 143.88, 143.71, 143.64, 142.49 (aryl *C*), 142.36, 142.30, 141.97, 141.89, 141.83 (2C), 141.68, 141.61 (2C), 141.40, 141.38, 141.31, 141.26, 141.09, 141.02 (2C), 140.97, 140.91, 140.80, 139.86, 139.68 (aryl *C*), 139.57, 139.27 (aryl *C*), 138.55, 138.35, 135.99, 134.91, 134.57, 133.71, 129.81 (aryl *C*), 129.21 (2C, aryl *C*), 129.15 (aryl *C*), 126.63 (aryl *C*), 126.45 (2C, aryl *C*), 125.13 (aryl *C*), 74.93 (sp^3-*C* of C_{60}), 73.52 (sp^3-*C* of C_{60}), 68.36 (*C*H), 20.98 (*C*H$_3$); FT-IR ν/cm^{-1} (KBr) 3243, 2921, 2852, 1452, 1420, 1337, 1288, 1161, 1083, 810, 747, 666, 558, 527; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 258 (4.98), 312 (4.52), 430 (3.56), 700 (2.46); MALDI FT-ICR MS m/z calcd for $C_{67}H_6N$ [M-MePhSO$_2$] 824.0500, found 824.0495.

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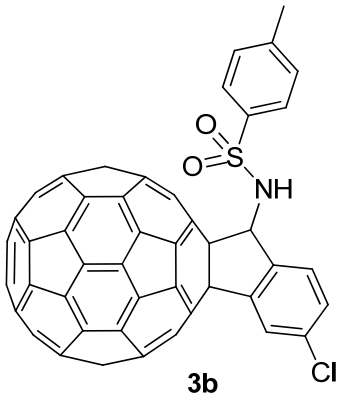
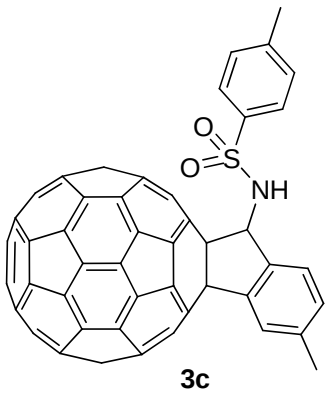


3b

Spectral data of **3b**: ^{1}H NMR (400 MHz, CS_2/DMSO-d_6) δ 9.52 (d, J = 9.6 Hz, 1H), 8.00 (d, J = 2.0 Hz, 1H), 7.75 (d, J = 8.4 Hz, 2H), 7.49 (dd, J = 8.0, 2.0 Hz, 1H), 7.41 (d, J = 8.0 Hz), 7.15 (d, J = 8.4 Hz, 2H), 6.67 (d, J = 9.6 Hz, 1H), 2.36 (s, 3H); ^{13}C NMR [75 MHz, CS_2/DMSO-d_6/$CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 155.55, 153.41, 152.23, 152.00, 146.41, 146.33, 146.08, 145.32, 145.30, 145.26, 145.24, 145.14, 145.03 (2C), 145.01 (2C), 144.99, 144.76, 144.71, 144.51, 144.38, 144.34, 144.31, 144.28, 144.23 (2C), 144.14, 144.12, 143.61 (2C), 143.39, 143.32, 142.72 (aryl *C*), 142.09, 142.03, 141.82 (aryl *C*), 141.74, 141.63, 141.57 (2C), 141.40, 141.33, 141.25, 141.08, 141.03 (2C), 140.99, 140.86, 140.72, 140.65, 140.62, 140.51, 139.66, 139.32, 139.11 (aryl *C*), 138.33, 138.22 (aryl *C*), 138.09, 135.72, 135.14 (aryl *C*), 134.99, 134.43, 133.28, 129.06 (aryl *C*), 128.77 (2C, aryl *C*), 127.86 (aryl *C*), 126.32 (2C, aryl *C*), 124.66 (aryl *C*), 74.70 (sp^3-*C* of C_{60}), 72.63 (sp^3-*C* of C_{60}), 67.46 (*C*H), 20.81 (*C*H_3); FT-IR ν/cm^{-1} (KBr) 3269, 2921, 1428, 1336, 1160, 1084, 811, 662, 564, 525; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 255 (4.97), 312 (4.50), 430 (3.52), 698 (2.48); MALDI FT-ICR MS m/z calcd for $C_{67}H_5{}^{35}ClN$ [M-$MePhSO_2$] 858.0111, found 858.0116.



3c

Spectral data of **3c**: ^{1}H NMR ((400 MHz, CS_2/DMSO-d_6/$CDCl_3$) δ 9.34 (d, J = 9.4 Hz, 1H), 7.83 (s, 1H), 7.76 (d, J = 8.0 Hz, 2H), 7.31 (dd, J = 7.8, 0.8 Hz, 1H), 7.28 (d, J = 7.8 Hz), 7.16 (d, J = 8.0 Hz, 2H), 6.67 (d, J = 9.4 Hz, 1H), 2.51 (s, 3H), 2.36 (s, 3H); FT-IR ν/cm^{-1} (KBr) 3260, 2922, 1434, 1325, 1261, 1157, 1093, 1026, 805, 662, 525; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 256 (4.97), 312 (4.50), 430 (3.48), 700 (2.52); MALDI FT-ICR MS m/z calcd for $C_{68}H_8N$ [M-$MePhSO_2$] 838.0657, found 838.0663.

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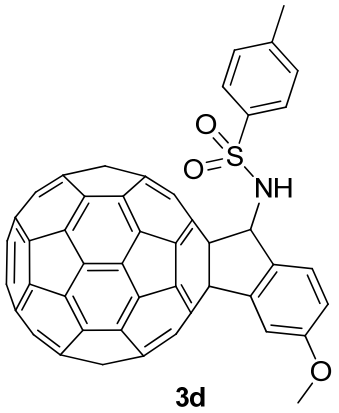
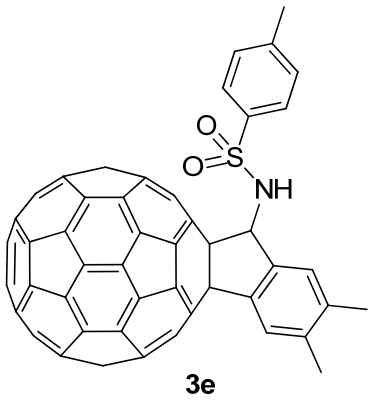


3d

Spectral data of **3d**: ^{1}H NMR (400 MHz, CS_2/DMSO-d_6) δ 9.25 (d, J = 9.6 Hz, 1H), 7.74 (d, J = 8.2 Hz, 2H), 7.43 (d, J = 2.4 Hz, 1H), 7.30 (d, J = 8.6 Hz, 1H), 7.13 (d, J = 8.2 Hz, 2H), 7.03 (dd, J = 8.6, 2.4 Hz, 1H), 6.60 (d, J = 9.6 Hz, 1H), 3.87 (s, 3H), 2.37 (s, 3H); ^{13}C NMR [75 MHz, CS_2/DMSO-d_6 with Cr(acac)$_3$ as relaxation reagent] (all 1C unless indicated) δ 160.19 (aryl *C*), 155.60, 153.52, 152.46, 152.16, 145.96, 145.89, 145.83, 145.05, 144.87, 144.84 (2C), 144.80, 144.69, 144.59, 144.57 (2C), 144.55, 144.38, 144.35, 144.19, 143.97, 143.88, 143.86, 143.81 (2C), 143.76, 143.71, 143.68, 143.23 (2C), 142.96, 142.91, 141.67, 141.61, 141.61 (aryl *C*), 141.30, 141.18, 141.14, 141.12, 141.00, 140.94 (3C), 140.74 (2C), 140.58, 140.54, 140.37, 140.25, 140.24, 140.21, 140.07, 139.19, 139.07 (aryl *C*), 138.83, 137.93, 137.64, 135.29, 134.48, 133.86, 132.88, 130.48 (aryl *C*), 128.23 (2C, aryl *C*), 127.00 (aryl *C*), 126.00 (2C, aryl *C*), 115.97 (aryl *C*), 107.93 (aryl *C*), 74.64 (sp^3-*C* of C_{60}), 72.61 (sp^3-*C* of C_{60}), 67.17 (*C*H), 54.34 (O*C*H$_3$), 20.52 (*C*H$_3$); FT-IR ν/cm^{-1} (KBr) 3254, 2924, 2853, 1457, 1263, 1158, 1090, 1025, 804, 662, 526; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 256 (4.98), 312 (4.52), 435 (3.48), 698 (2.40); MALDI FT-ICR MS m/z calcd for $C_{68}H_8NO$ [M-MePhSO$_2$] 854.0606, found 854.0612.



3e

Spectral data of **3e**: ^{1}H NMR (400 MHz, CS_2/DMSO-d_6/$CDCl_3$) δ 9.08 (d, J = 9.6 Hz, 1H), 7.82 (d, J = 8.0 Hz, 2H), 7.78 (s, 1H), 7.20 (d, J = 8.0 Hz, 2H), 7.06 (s, 1H), 6.70 (d, J = 9.6 Hz, 1H), 2.41 (s, 3H), 2.39 (s, 3H), 2.36 (s, 3H); ^{13}C NMR [75 MHz, CS_2/DMSO-d_6/$CDCl_3$ with Cr(acac)$_3$ as relaxation reagent] (all 1C unless indicated) δ 156.41, 154.50, 152.30, 152.95, 146.47, 146.42, 146.38, 145.69, 145.34, 145.31 (3C), 145.20, 145.10, 145.08 (3C), 144.89, 144.86, 144.73, 144.43, 144.39, 144.30 (3C),

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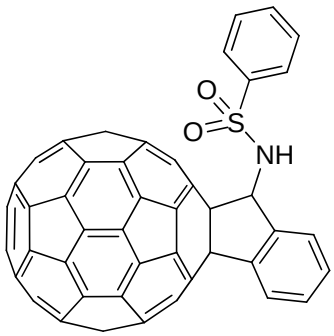
144.26, 144.19 (2C), 143.72, 143.70, 143.53, 143.46, 142.17, 142.12, 141.84 (aryl *C*), 141.79, 141.69, 141.63 (2C), 141.53, 141.48, 141.41, 141.32, 141.22, 141.10, 141.06, 140.90, 140.79, 140.78, 140.73, 140.61, 139.66, 139.47 (aryl *C*), 139.33, 138.44 (aryl C), 138.44, 138.16, 138.08 (aryl *C*), 137.34 (aryl *C*), 136.94 (aryl C), 135.81, 134.65, 134.22, 133.48, 128.83 (2C, aryl *C*), 127.07 (aryl *C*), 126.46 (2C, aryl *C*), 125.47 (aryl *C*), 75.03 (sp^3-*C* of C_{60}), 73.06 (sp^3-*C* of C_{60}), 68.06 (*C*H), 20.85 (*C*H_3), 19.45 (*C*H_3), 19.38 (*C*H_3); FT-IR ν/cm^{-1} (KBr) 3250, 2923, 2854, 1447, 1328, 1156, 1089, 806, 663, 563, 524; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 256 (4.99), 311 (4.51), 430 (3.50), 700 (2.46); MALDI FT-ICR MS m/z calcd for $C_{69}H_{10}N$ [M-MePhSO_2] 852.0813, found 852.0820.



3f

Spectral data of **3f**: ^{1}H NMR (400 MHz, CS_2/DMSO-d_6) δ 9.51 (d, *J* = 9.2 Hz, 1H), 8.02 (d, *J* = 7.6 Hz, 1H), 7.92 (d, *J* = 7.2 Hz, 2H), 7.58 (t, *J* = 7.0 Hz, 1H), 7.50-7.34 (m, 5H), 6.74 (d, *J* = 9.2 Hz, 1H); ^{13}C NMR [75 MHz, CS_2/DMSO-d_6/$CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 156.04, 154.14, 152.87, 152.59, 146.35, 146.31, 146.28, 145.47, 145.25, 145.21, 145.18 (2C), 145.07, 144.99, 145.96 (2C), 145.94, 144.75, 144.71, 144.48, 144.34 (2C), 144.20 (3C), 144.15, 144.13, 144.08, 143.62, 143.60, 143.37, 143.29, 142.04, 141.99 (aryl *C*), 141.99, 141.64, 141.56, 141.51 (2C), 141.37, 141.32, 141.30, 141.10, 141.07, 140.98, 140.93, 140.78, 140.70, 140.66 (3C), 140.59, 139.56, 139.42 (aryl *C*), 139.23, 138.42, 138.16, 135.68, 134.70, 134.26, 133.34, 131.38 (aryl *C*), 129.36 (aryl *C*), 128.72 (aryl *C*), 128.23 (2C, aryl *C*), 126.41 (aryl *C*), 126.15 (2C, aryl *C*), 124.70 (aryl *C*), 74.55 (sp^3-*C* of C_{60}), 73.11 (sp^3-*C* of C_{60}), 68.08 (*C*H); FT-IR ν/cm^{-1} (KBr) 3245, 2923, 2854, 1446, 1330, 1261, 1157, 1091, 1027, 804, 745, 676, 550, 521; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 257 (4.97), 312 (4.47), 430 (3.49), 700 (2.50).MALDI FT-ICR MS m/z calcd for $C_{67}H_6N$ [M-PhSO_2] 824.0500, found 824.0506.

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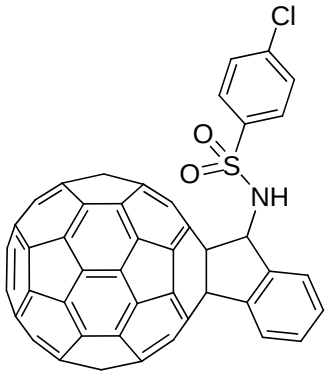
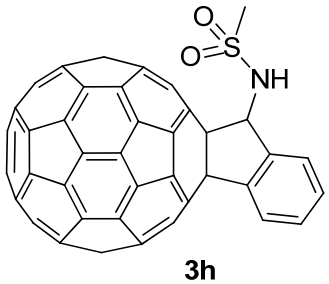


3g

Spectral data of **3g**: ^{1}H NMR (400 MHz, CS_2/DMSO-d_6) δ 9.55 (d, J = 10.0 Hz, 1H), 8.03 (d, J = 7.5 Hz, 1H), 7.85 (d, J = 8.6 Hz, 2H), 7.60 (t, J = 7.8 Hz, 1H), 7.54 (t, J = 7.4 Hz, 1H), 7.50 (d, J = 7.6 Hz, 1H), 7.31 (d, J = 8.6 Hz, 2H), 6.74 (d, J = 10 Hz, 1H); ^{13}C NMR [75 MHz, CS_2/DMSO-d_6/$CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 156.09, 154.22, 153.02, 152.53, 146.52, 146.44, 146.19, 145.80, 145.60, 145.41, 145.38, 145.35, 145.24, 145.15 (2C), 145.13 (2C), 145.11, 144.90, 144.87, 144.67, 144.50, 144.47, 144.37 (3C), 144.31, 144.25, 143.77, 143.75, 143.55, 143.48, 142.23, 142.23 (aryl *C*), 142.17, 141.82, 141.74, 141.69 (2C), 141.56, 141.47 (2C), 141.25, 141.22, 141.15, 141.11, 140.94, 140.85 (3C), 140.81, 140.73, 139.73, 139.44 (aryl *C*), 139.42, 138.51, 138.20, 137.70 (aryl *C*), 135.94, 134.85, 134.38, 133.57, 129.65 (aryl *C*), 129.03 (aryl *C*), 128.60 (2C, aryl *C*), 127.99 (2C, aryl *C*), 126.55 (aryl *C*), 124.93 (aryl *C*), 74.68 (sp^3-*C* of C_{60}), 73.32 (sp^3-*C* of C_{60}), 68.27 (*C*H); FT-IR ν/cm^{-1} (KBr) 3251, 2924, 2854, 1458, 1417, 1262, 1164, 1091, 1026, 804, 751, 554, 524; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 256 (4.98), 312 (4.49), 430 (3.56), 698 (2.53). MALDI FT-ICR MS m/z calcd for $C_{67}H_6N$ [M-ClPhSO_2] 824.0500, found 824.0496



3h

Spectral data of **3h**: ^{1}H NMR (400 MHz, CS_2/DMSO-d_6) δ 8.84 (d, J = 9.8 Hz, 1H), 8.08-8.05 (m, 1H), 7.96-7.93 (m, 1H), 7.66-7.61 (m, 2H), 6.83 (d, J = 9.8 Hz, 1H), 3.07 (s, 3H); ^{13}C NMR [75 MHz, CS_2/DMSO-d_6/$CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 156.32, 154.21, 152.90, 152.68, 146.48, 146.43, 146.40, 145.51, 145.33, 145.28 (2C), 145.17, 145.07 (4C), 144.95, 144.80, 144.78, 144.53, 144.50, 144.46, 144.34, 144.30 (4C), 144.19, 143.70, 143.67, 143.51, 143.41, 142.20, 142.17, 141.76, 141.69, 141.65 (2C), 141.46, 141.43, 141.40, 141.26, 141.18, 141.12, 141.05, 140.98, 140.91 (2C), 140.85, 140.82, 140.79, 139.87 (aryl *C*), 139.72, 139.40, 139.00, 138.63, 136.00, 134.79, 134.39, 133.55, 129.35 (aryl *C*), 128.78 (aryl *C*), 126.78 (aryl *C*), 124.76 (aryl *C*), 74.68 (sp^3-*C* of C_{60}), 73.26 (sp^3-*C* of C_{60}), 67.97 (*C*H), 41.93

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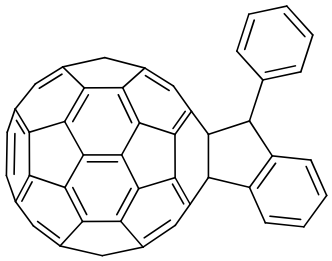
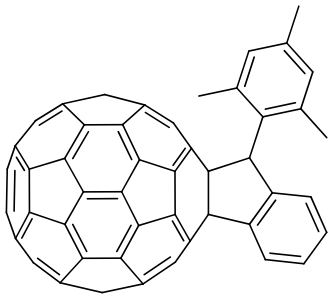
(CH_3); FT-IR ν/cm^{-1} (KBr) 3272, 2925, 2854, 1455, 1262, 1095, 1023, 802, 521; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 257 (4.99), 317 (4.57), 433 (3.37), 692 (2.37). MALDI FT-ICR MS m/z calcd for $C_{67}H_6N$ [M-$MeSO_2$] 824.0500, found 824.0498.



4a

Spectral data of **4a**: ^{1}H NMR (400 MHz, CS_2/$CDCl_3$) δ 8.17 (d, 7.6 Hz, 1H), 7.68 (bs, 1H), 7.62 (tt, 7.4, 1.2 Hz, 1H), 7.51 (td, 7.4, 1.2 Hz, 1H), 7.50 (d, 7.6 Hz, 2H), 7.38 (bs, 2H), 7.28 (tt, 7.4, 1.2 Hz, 1H), 6.50 (s, 1H); ^{13}C NMR [75 MHz, CS_2/$CDCl_3$ with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 157.90, 154.85, 154.62, 153.97, 147.32, 147.20, 146.20, 146.12, 146.05, 145.96, 145.92 (3C), 145.89, 145.84, 145.80, 145.68, 145.58, 145.42, 145.25, 145.16, 145.12, 145.07, 145.04, 144.95 (2C), 144.93 (2C), 144.45, 144.31, 144.28, 144.25, 143.21, 143.10, 143.00 (2C), 142.47 (2C), 142.43, 142.41, 142.24, 142.10, 142.09, 142.06, 142.02, 141.97, 141.92 (2C), 141.73 (2C), 141.63 (2C), 141.56, 140.50, 140.27, 139.91, 139.15, 135.87, 135.24, 135.00, 134.22, 129.36 (aryl *C*), 128.95 (aryl *C*), 127.70 (aryl *C*), 127.52 (aryl *C*), 125.66 (aryl *C*), 76.14 (sp^3-*C* of C_{60}), 75.68 (sp^3-*C* of C_{60}), 64.56 (*C*H) (Note: 4C of the phenyl ring were absent due to slow rotation); FT-IR ν/cm^{-1} (KBr) 2921, 2853, 1451, 1425, 1182, 1029, 743, 698, 579, 524; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 257 (4.96), 308 (4.54), 430 (3.53), 702 (2.45); MALDI FT-ICR MS m/z calcd for $C_{73}H_{10}$ [M$^-$] 886.8605, found 886.8610.



4b

Spectral data of **4b**: ^{1}H NMR (400 MHz, CS_2/$CDCl_3$) δ 8.17 (d, 7.6 Hz, 1H), 7.58 (t, 7.4 Hz, 1H), 7.51 (dt, 7.6, 1.2 Hz, 1H), 7.41 (d, 8.0 Hz, 2H), 7.04 (s, 1H), 6.91 (s, 1H), 6.82 (s, 1H), 2.67 (s, 1H), 2.29 (s, 1H), 2.26 (s, 1H), ^{13}C NMR [75 MHz, CS_2/DMSO-d_6 with $Cr(acac)_3$ as relaxation reagent] (all 1C unless indicated) δ 156.99, 153.38, 153.35, 153.26, 146.16, 146.06, 145.44, 145.19, 145.01, 144.96, 144.86, 144.83 (2C), 144.79, 144.77, 144.72, 144.54, 144.51, 144.46, 144.19, 144.08, 144.03, 144.00 (3C), 143.96, 143.90, 143.85, 143.39, 143.26, 143.21, 143.16, 141.94 (2C), 141.87, 141.46, 141.43 (2C), 141.38, 141.21, 141.02, 140.99 (2C), 140.89, 140.83, 140.81, 140.75, 140.72,

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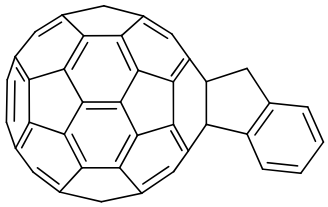
140.67, 140.56, 140.42, 139.58, 139.55, 139.34, 138.44, 138.35, 136.87 (aryl *C*), 136.46 (aryl *C*), 136.02 (aryl *C*), 135.35, 134.57, 133.85, 133.42, 132.52 (aryl *C*), 131.07 (aryl *C*), 128.80 (aryl *C*), 128.35 (aryl *C*), 127.43 (aryl *C*), 125.10 (aryl *C*), 125.04 (aryl *C*), 75.17 (sp^3-*C* of C_{60}), 74.85 (sp^3-*C* of C_{60}), 57.99 (*C*H), 22.87 (*C*H_3), 21.56 (*C*H_3), 20.11 (*C*H_3); FT-IR ν/cm^{-1} (KBr) 2919, 2850, 1451, 1428, 1374, 1263, 1184, 1028, 852, 746, 592, 526; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 257 (4.96), 308 (4.54), 430 (3.53), 700 (2.45); MALDI FT-ICR MS m/z calcd for $C_{76}H_{16}$ [M^-] 928.1252, found 928.1256.



4c

Spectral data of **4c:** ^{1}H NMR (400 MHz, CS_2/$CDCl_3$) δ 8.13-8.08 (m, 1H), 7.77-7.74 (m, 1H), 7.60-7.56 (m, 2H), 5.14 (s, 2H); ^{13}C NMR [75 MHz, CS_2/DMSO-d_6 with Cr(acac)$_3$ as relaxation reagent] (all 2C unless indicated) δ 156.26, 152.73, 146.30 (1C), 146.18 (1C), 145.07, 145.03, 145.01, 144.91 (4C), 144.77, 144.33, 144.17, 144.12 (4C), 143.45, 143.32, 142.09, 141.96, 141.54, 141.48, 141.28, 141.19, 141.09, 140.97, 140.85, 140.67, 140.43 (1C, aryl *C*), 139.52, 139.99, 138.67 (1C, aryl *C*), 134.08, 133.84, 128.46 (1C, aryl *C*), 127.77 (1C, aryl *C*), 125.79 (1C, aryl *C*), 125.11 (1C, aryl *C*), 75.36 (sp^3-*C* of C_{60}), 68.04 (sp^3-*C* of C_{60}), 46.69 (*C*H_2); FT-IR ν/cm^{-1} (KBr) 2920, 2851, 1454, 1428, 1183, 1103, 1032, 740, 524; UV-vis ($CHCl_3$) λ_{max}/nm (log ε) 257 (4.99), 317 (4.57), 433 (3.37), 692 (2.37); MALDI FT-ICR MS m/z calcd for $C_{67}H_6$ [M^-] 810.0470, found 810.0466.

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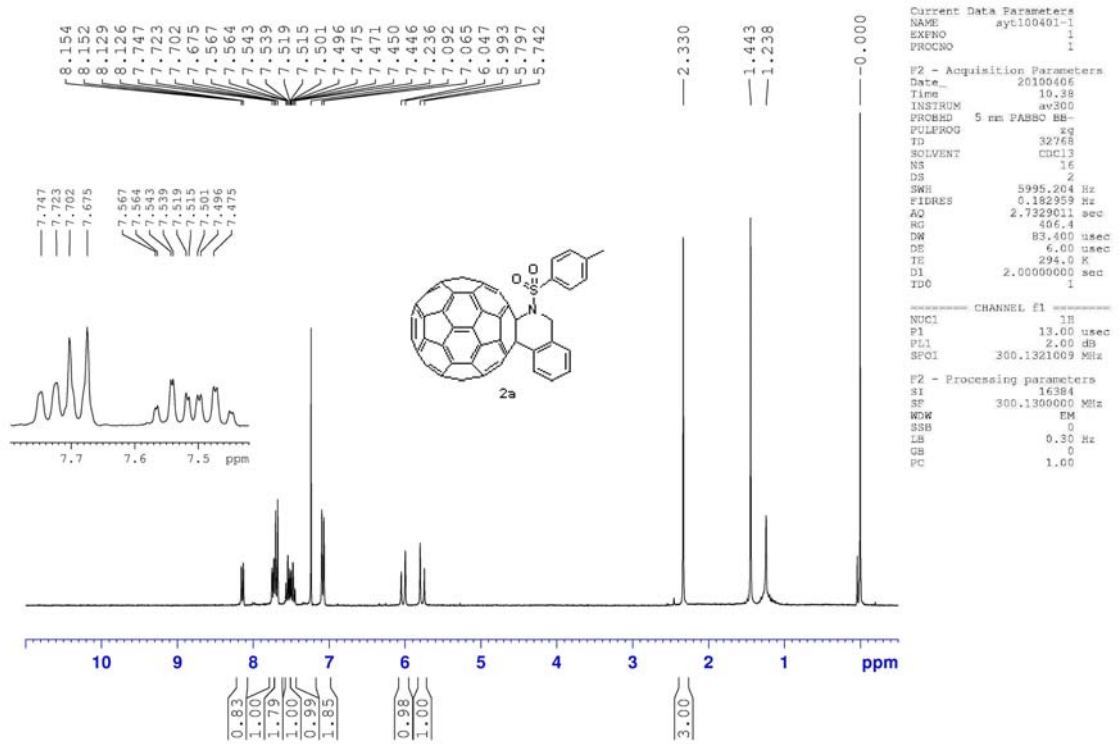

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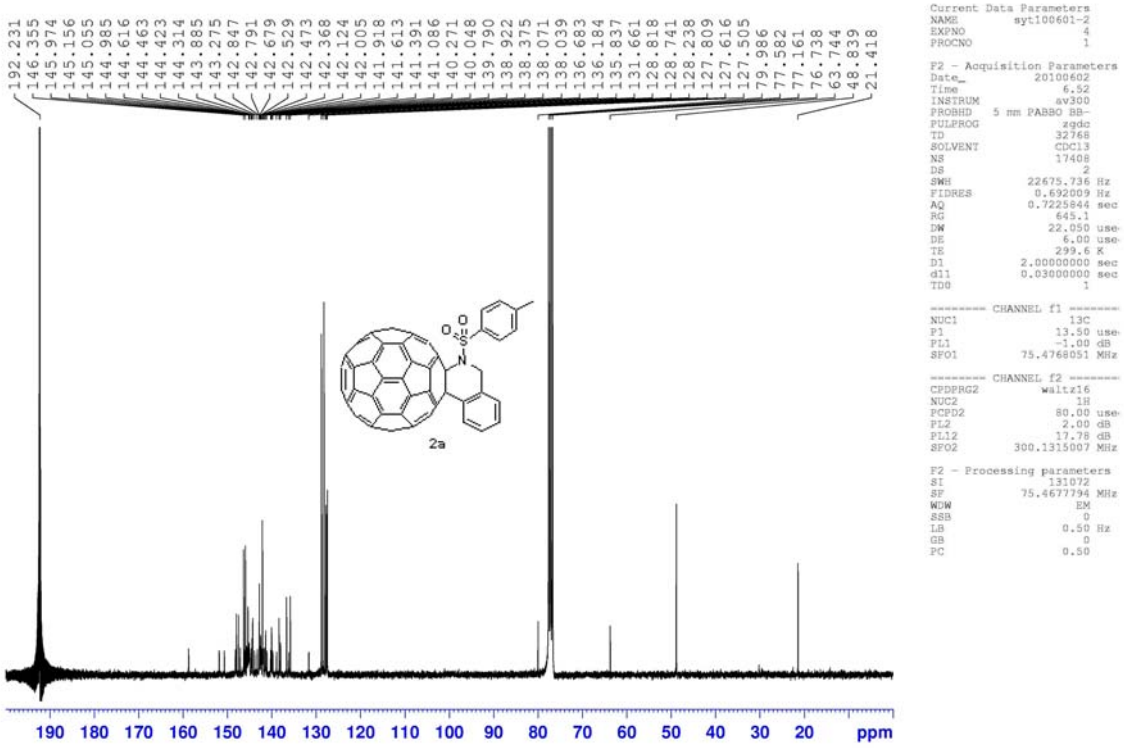

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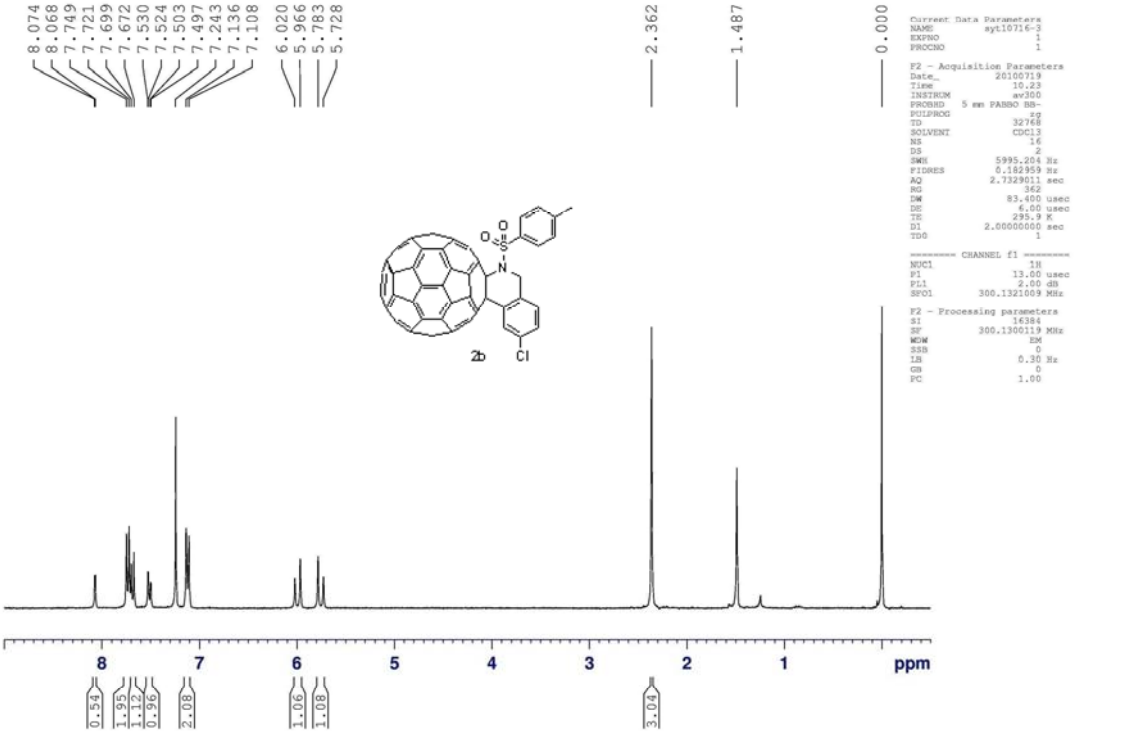

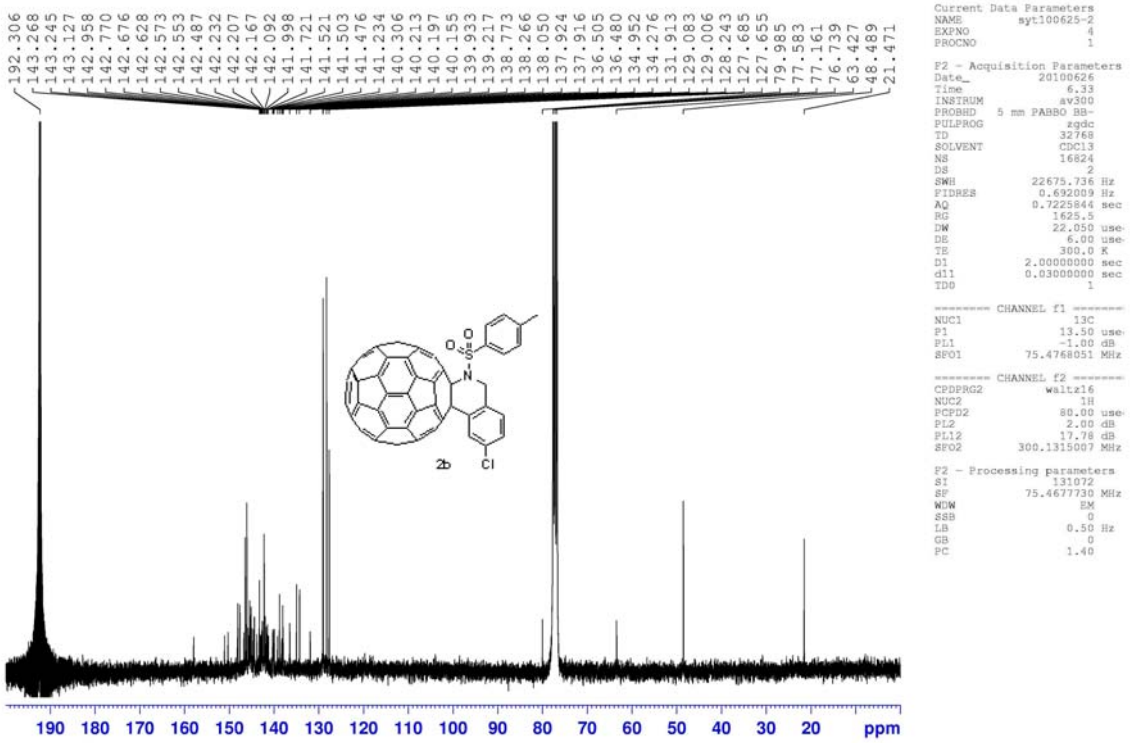

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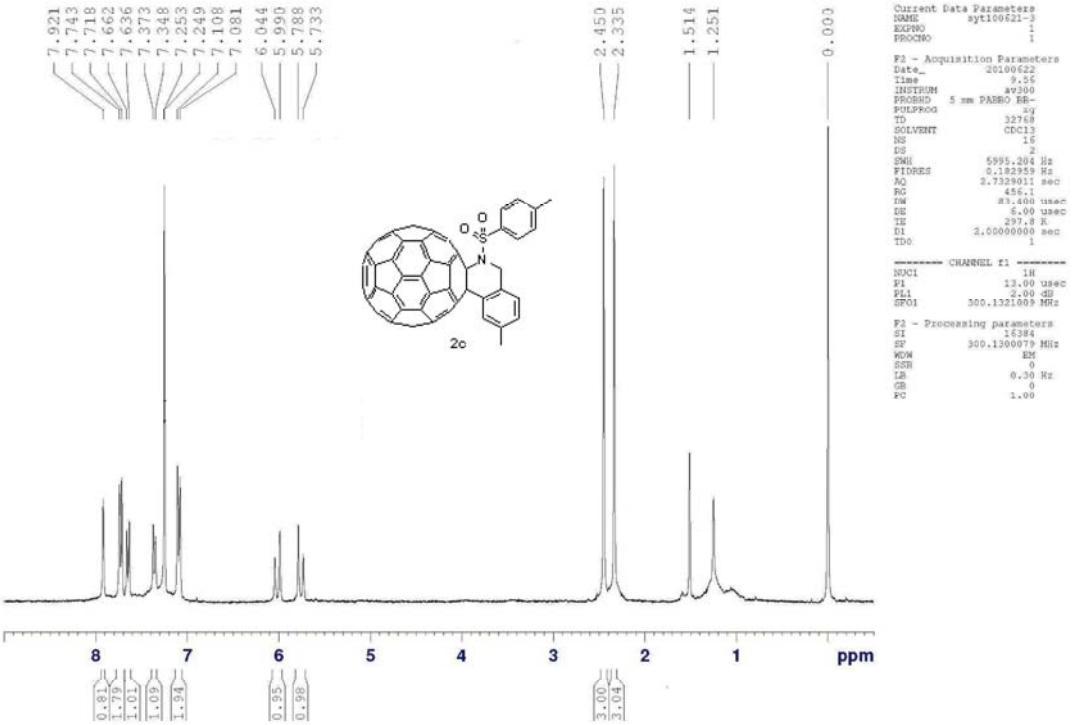

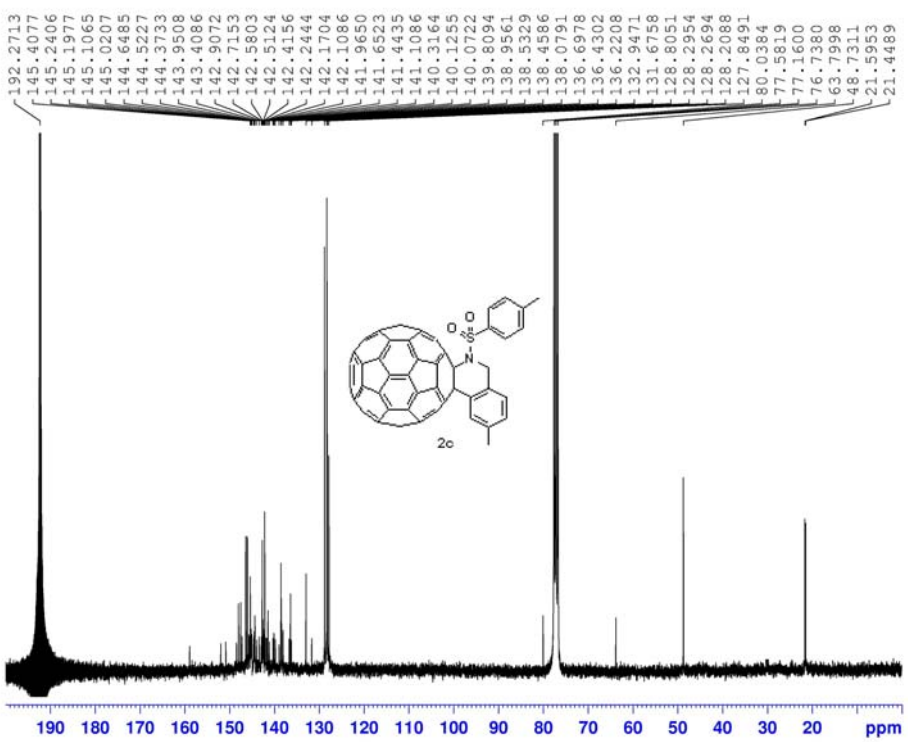

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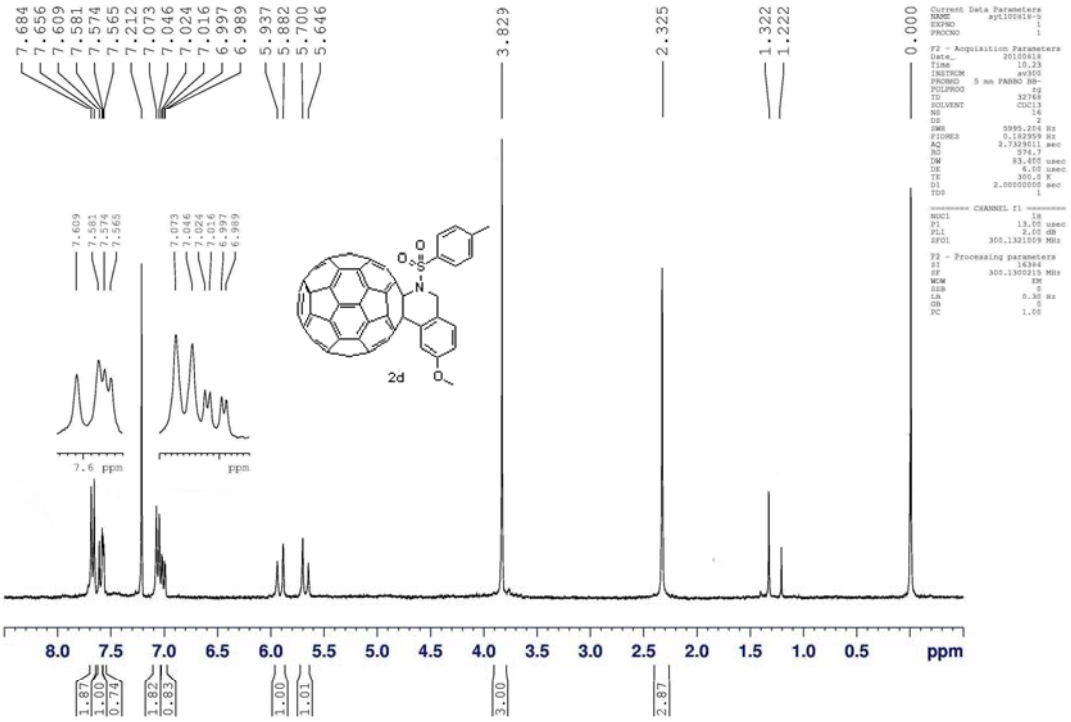

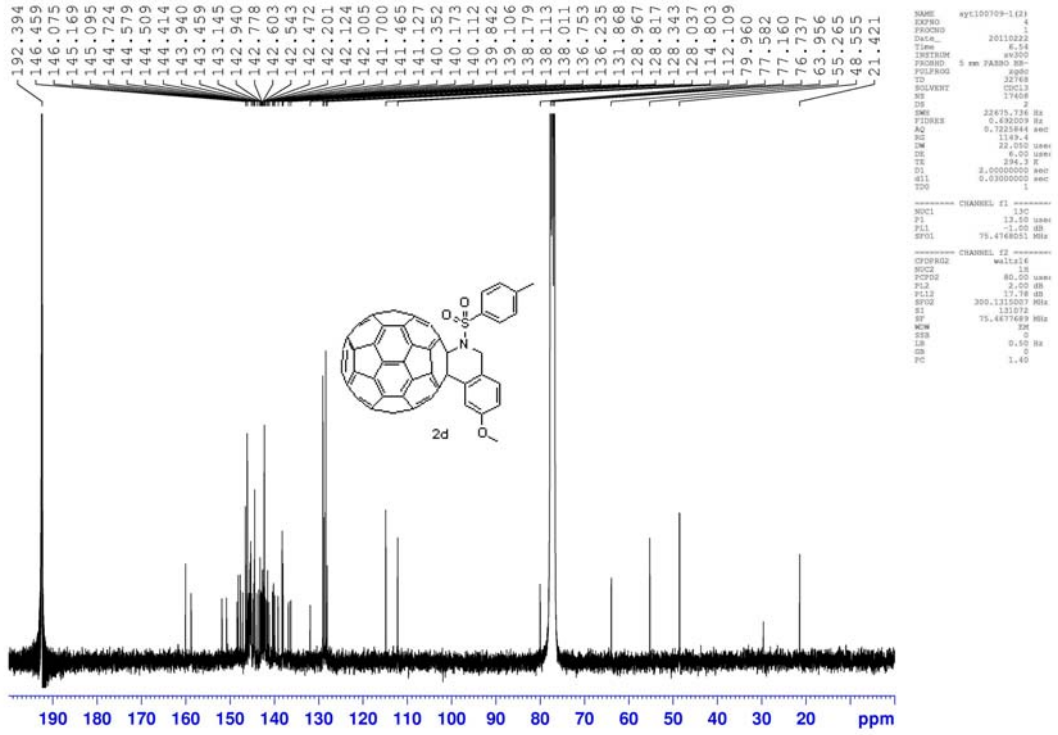

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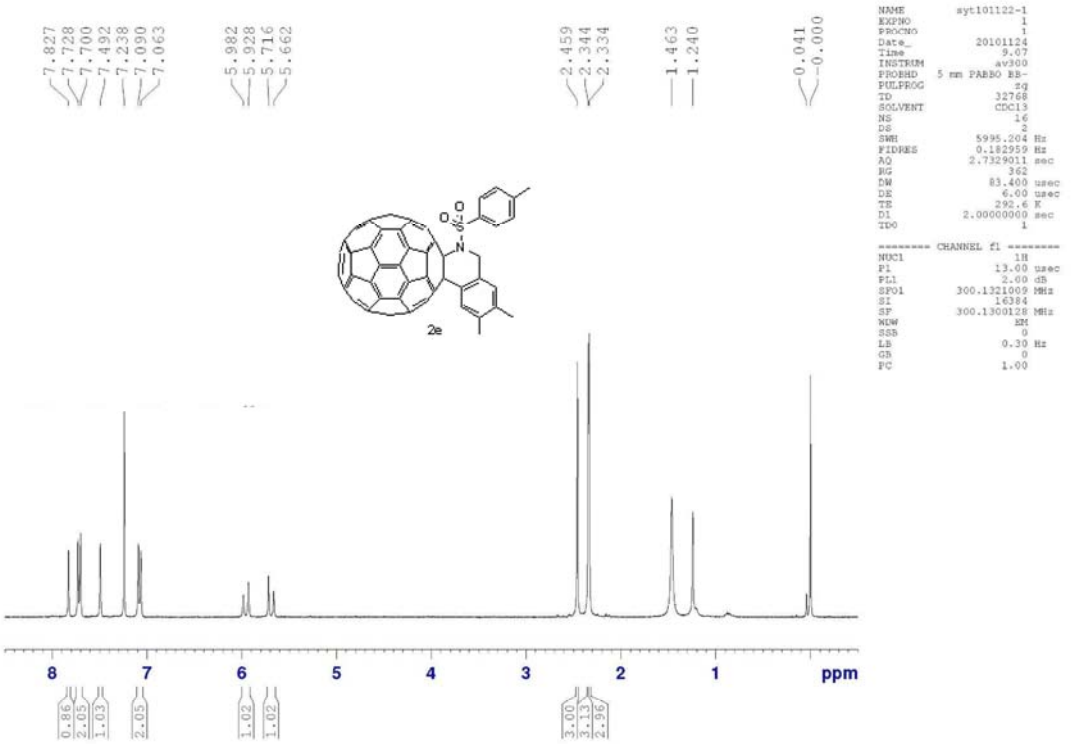

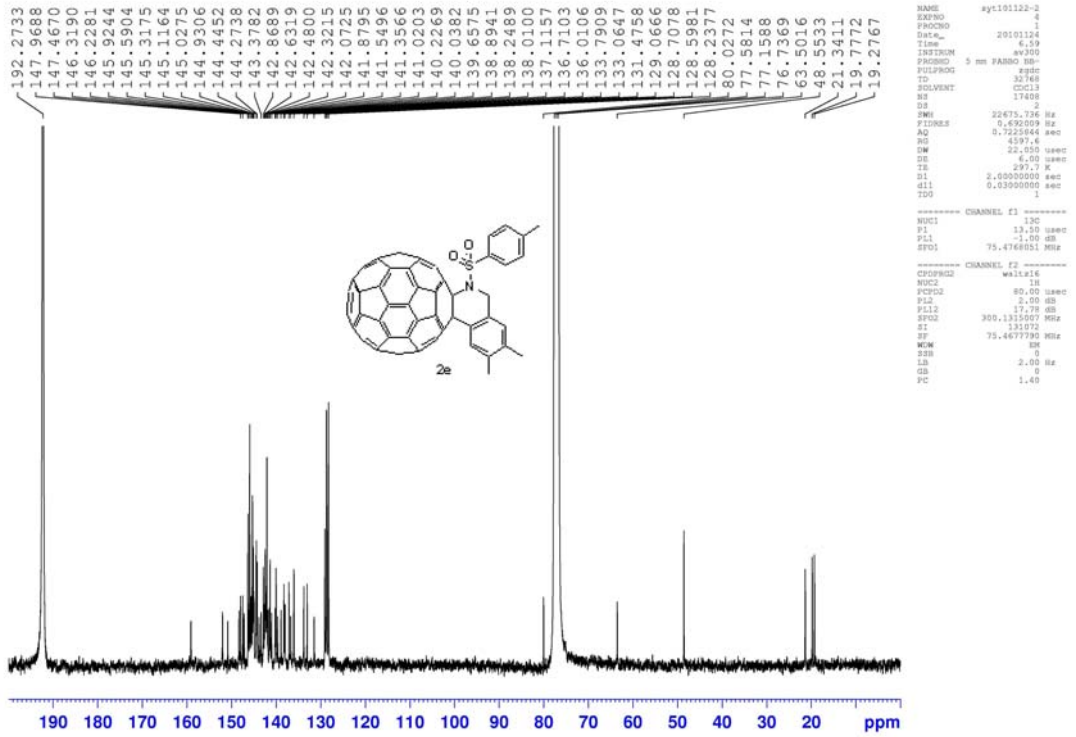

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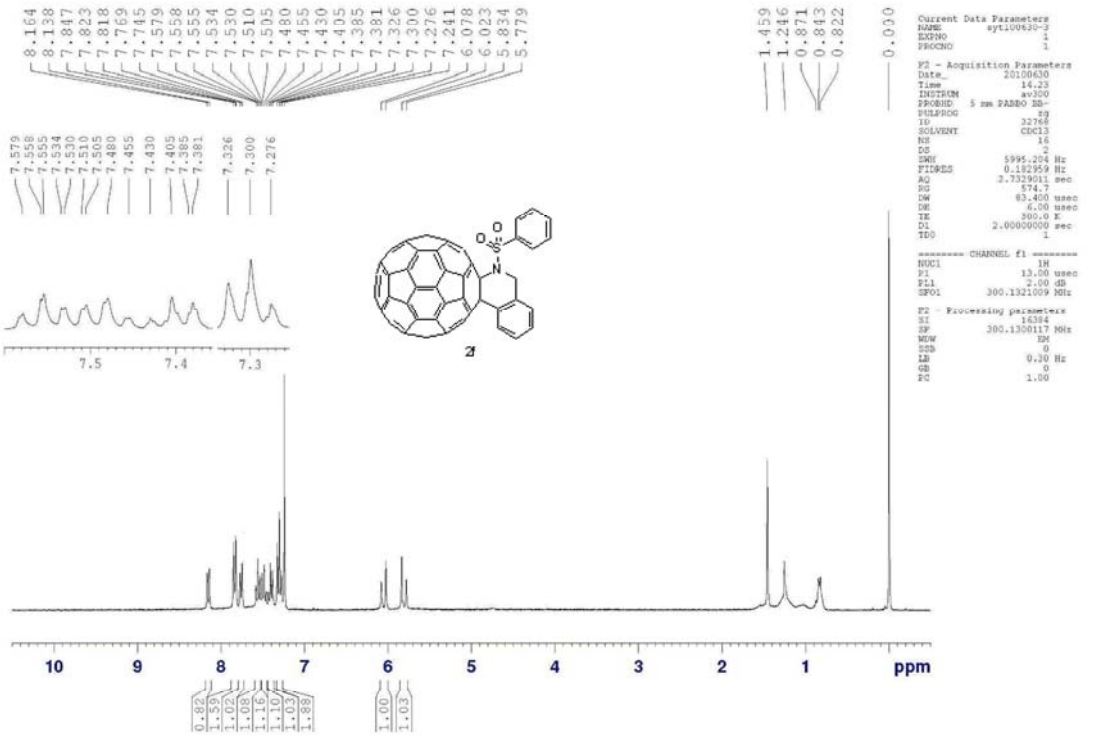

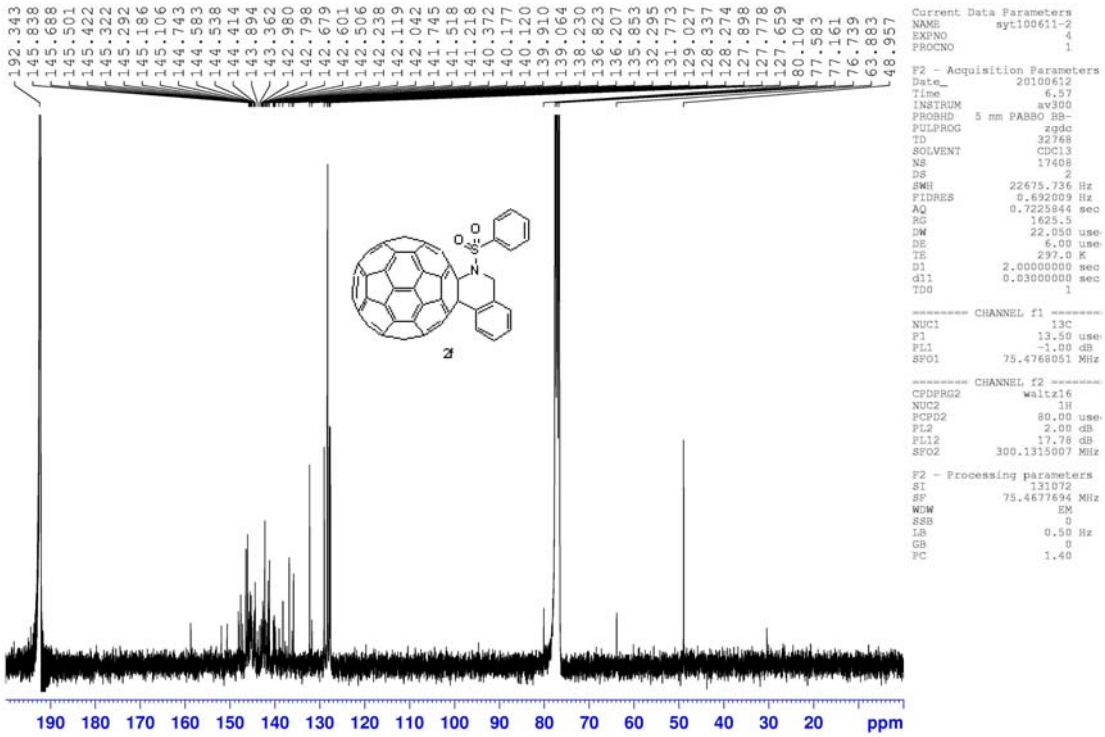

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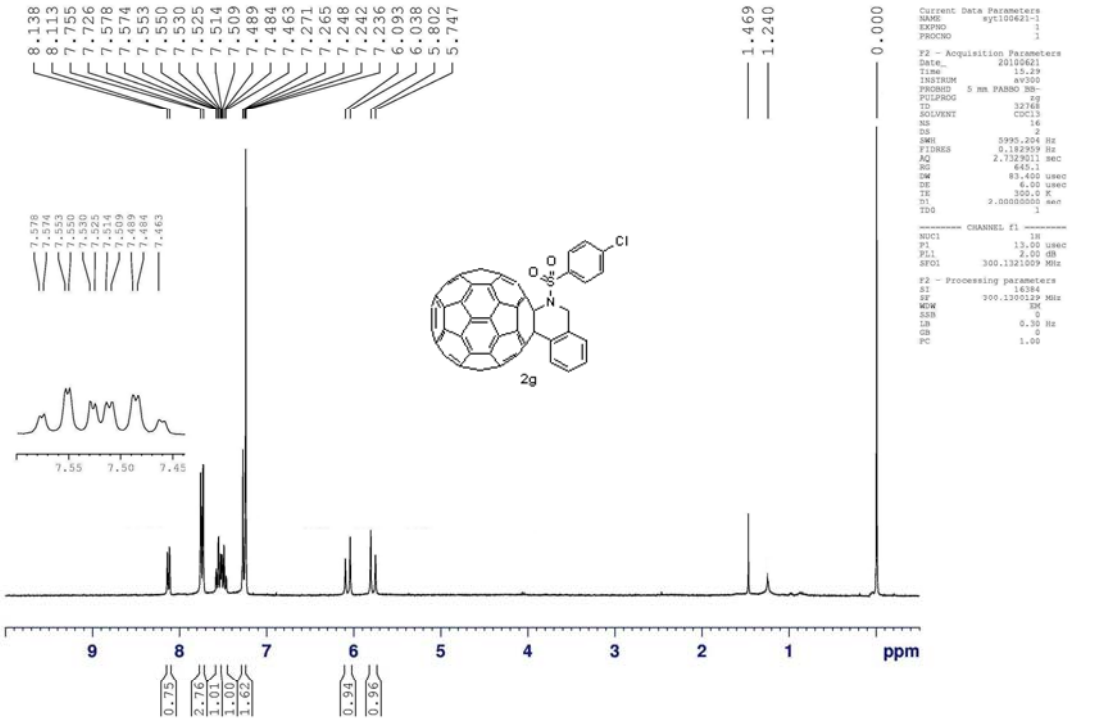

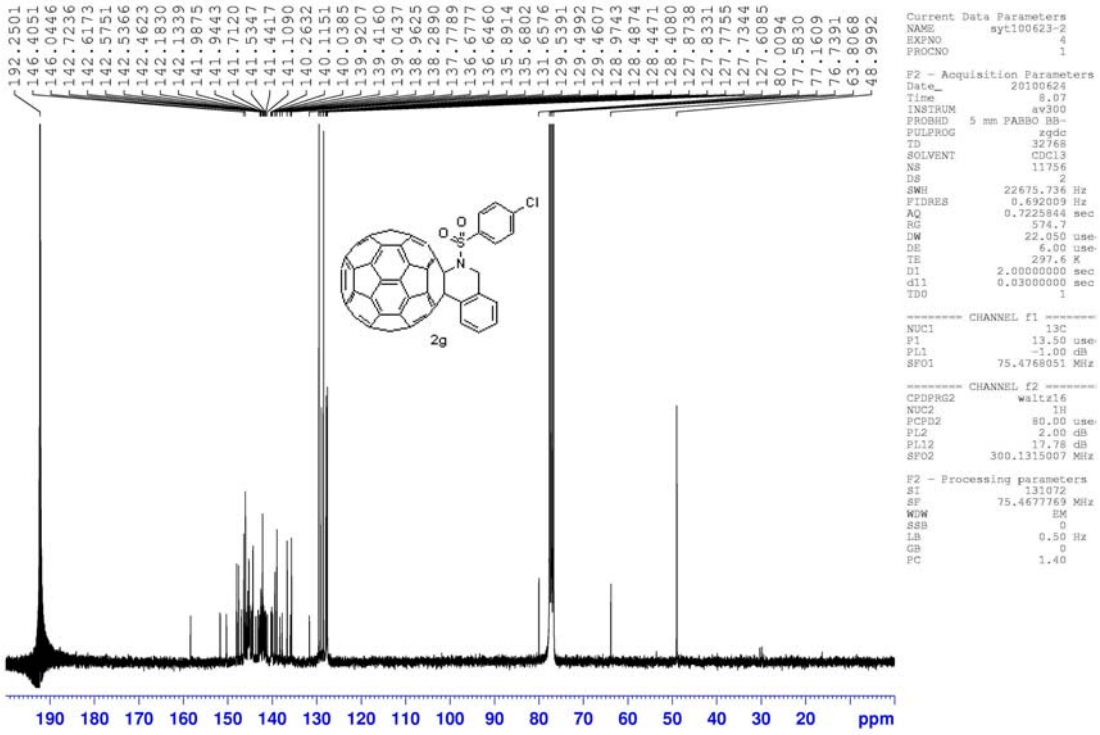

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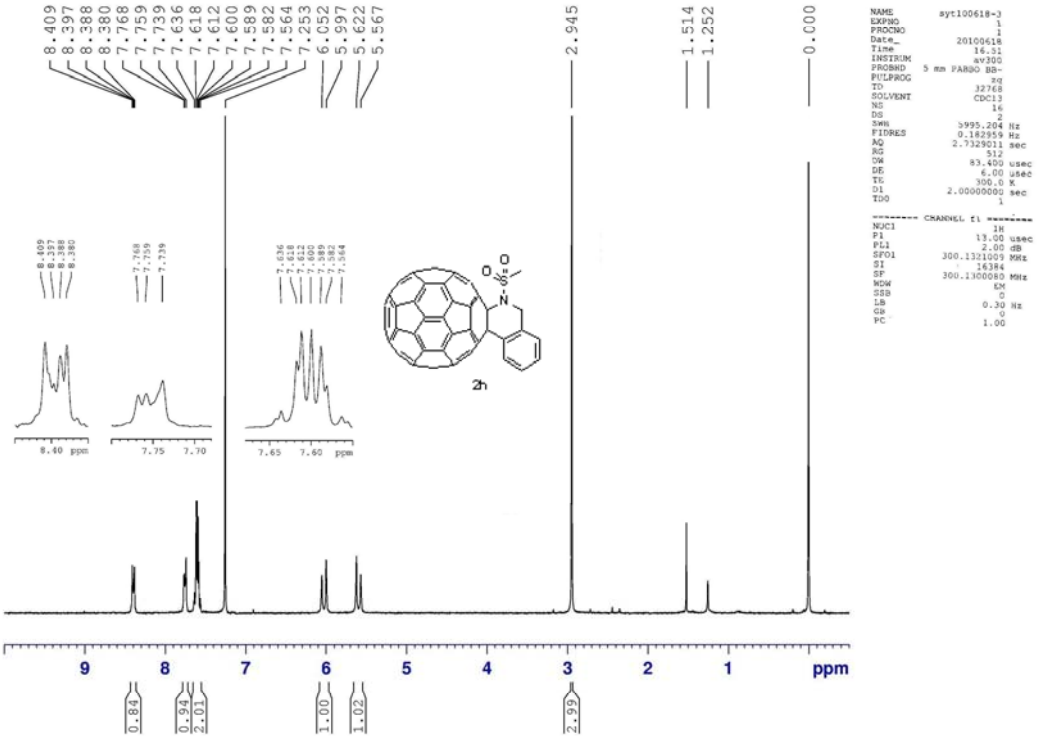

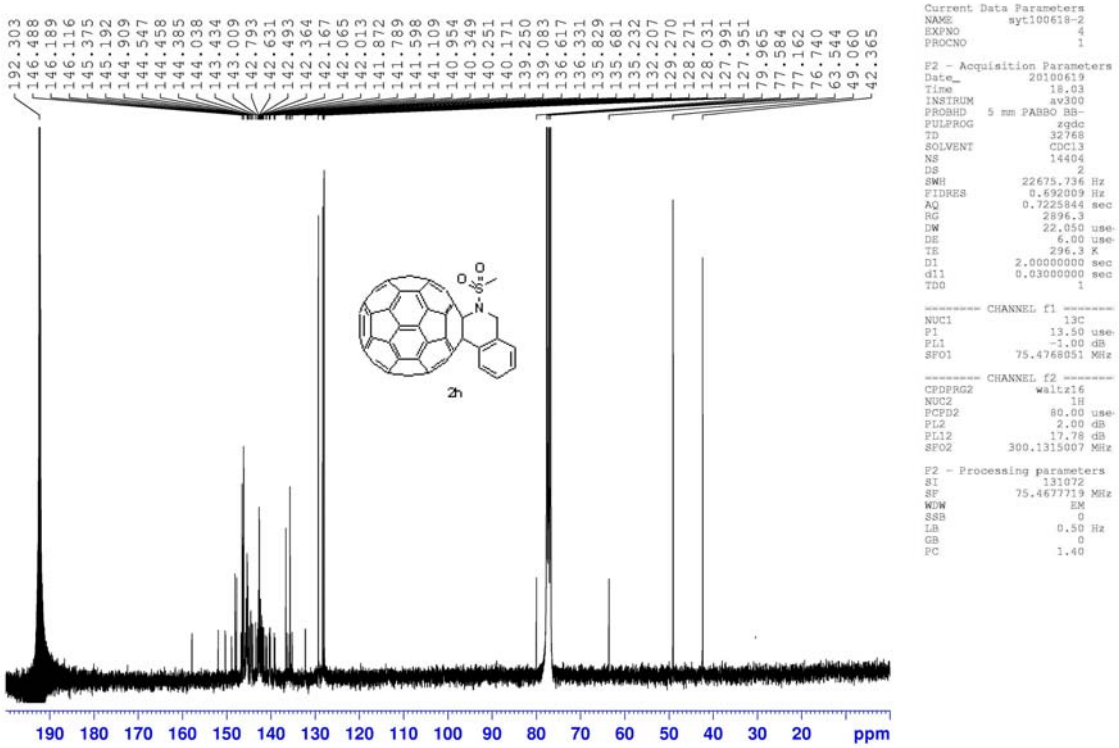

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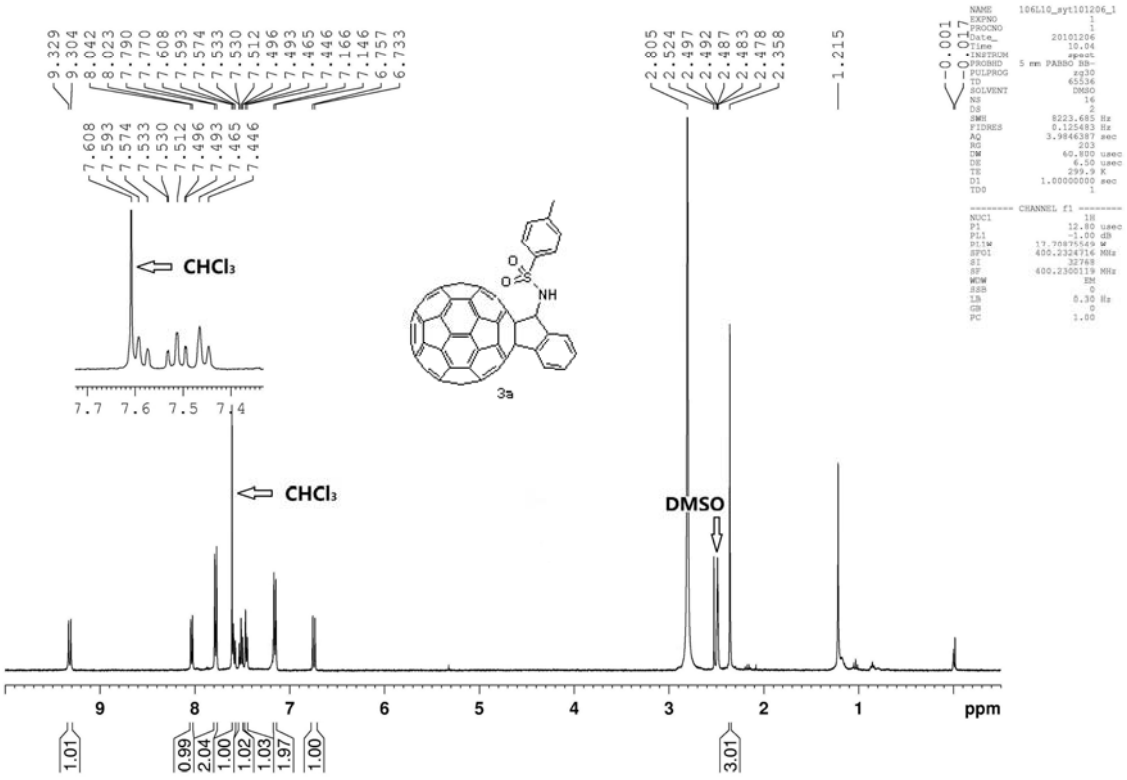
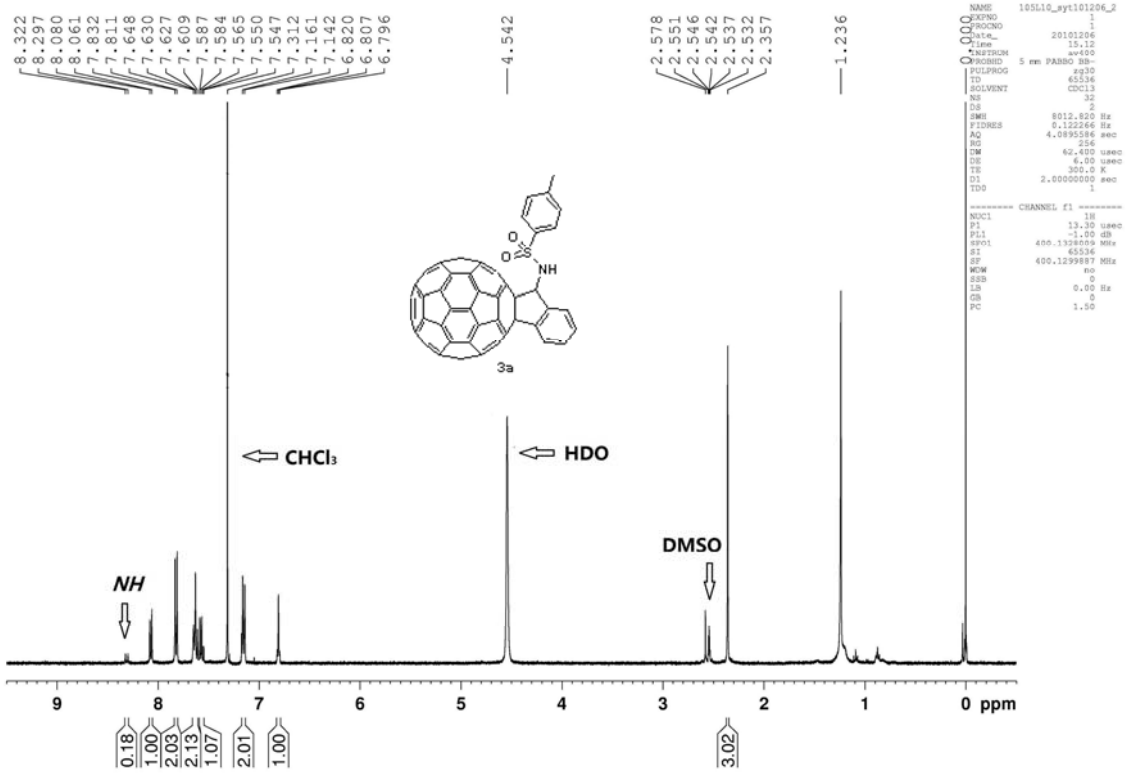


D_2O exchange experiment



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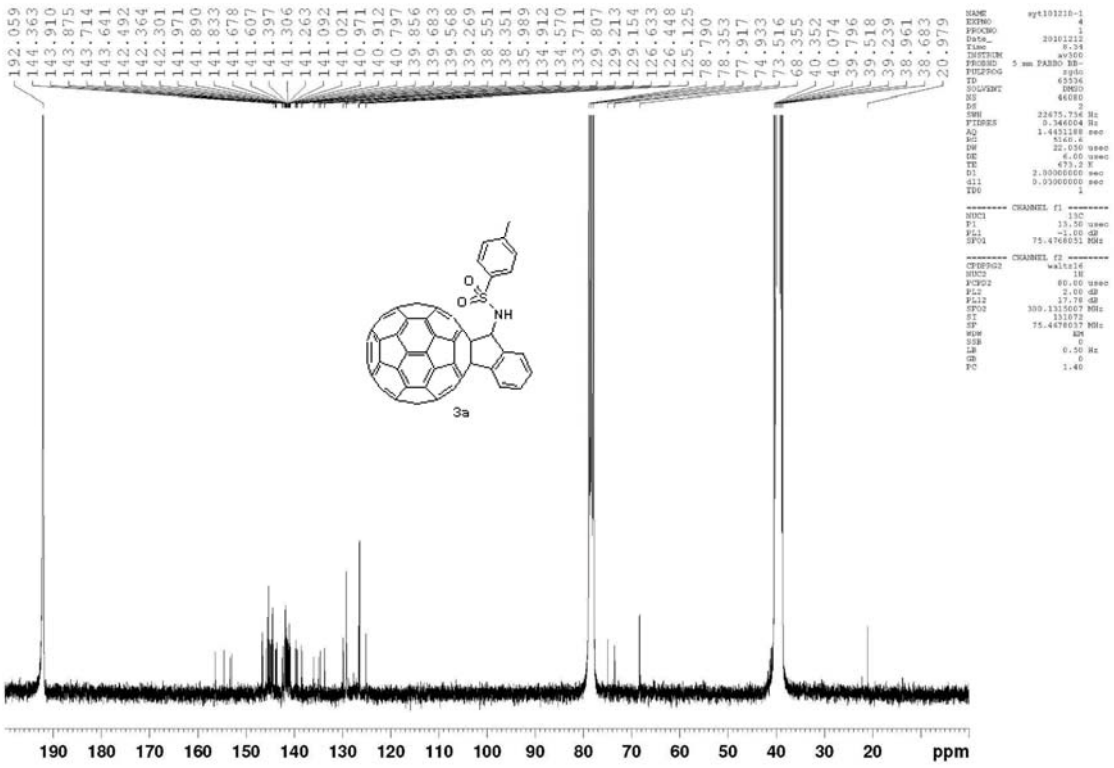

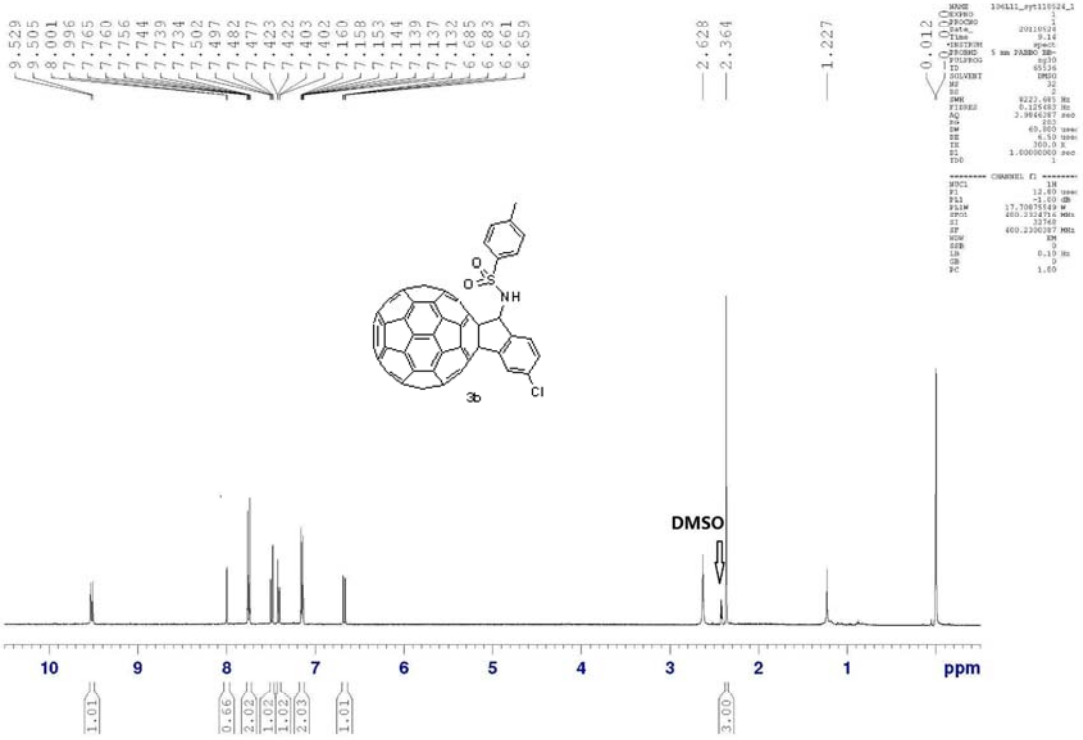

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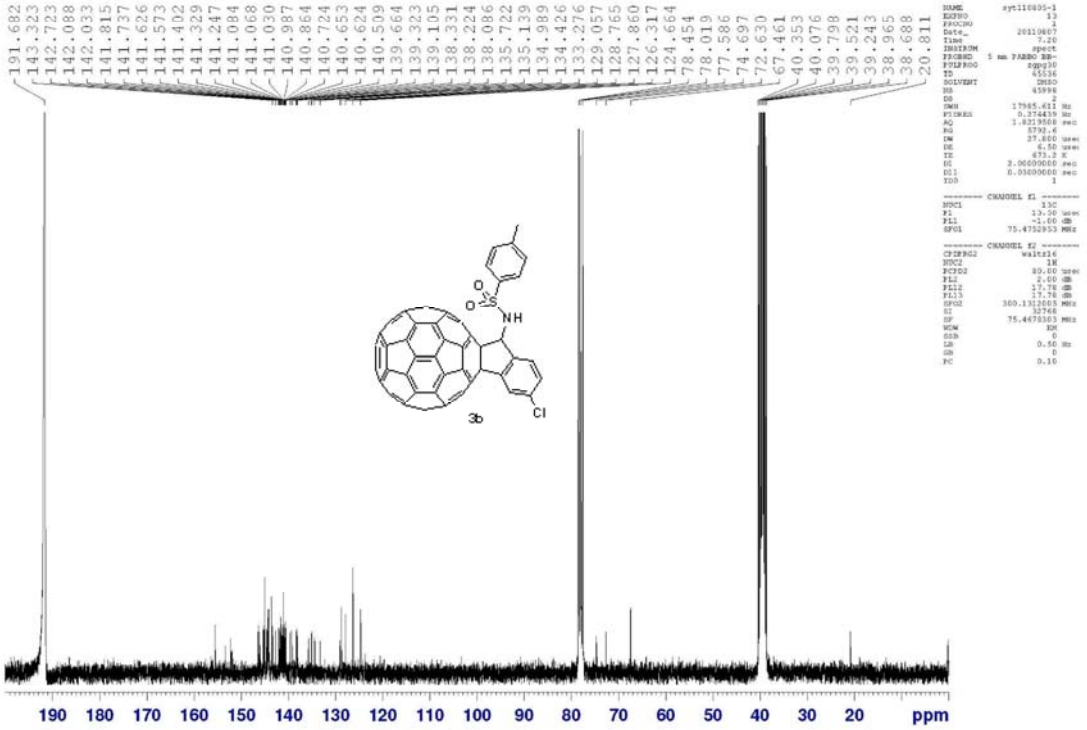

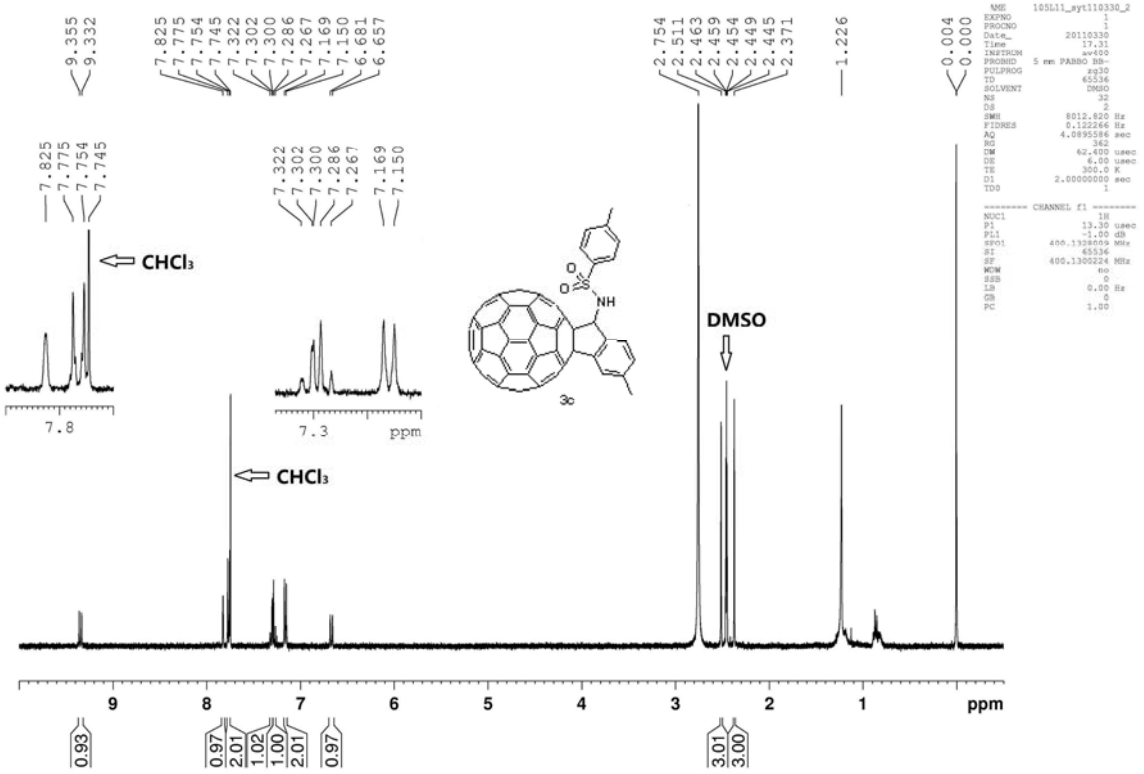

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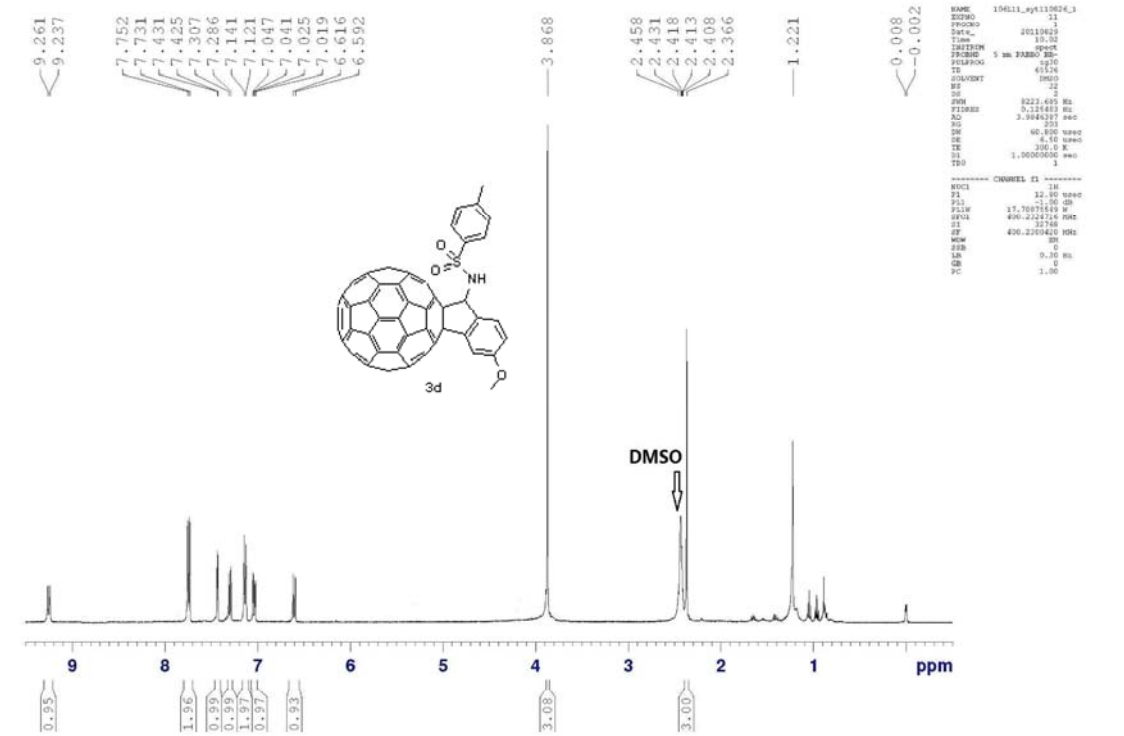

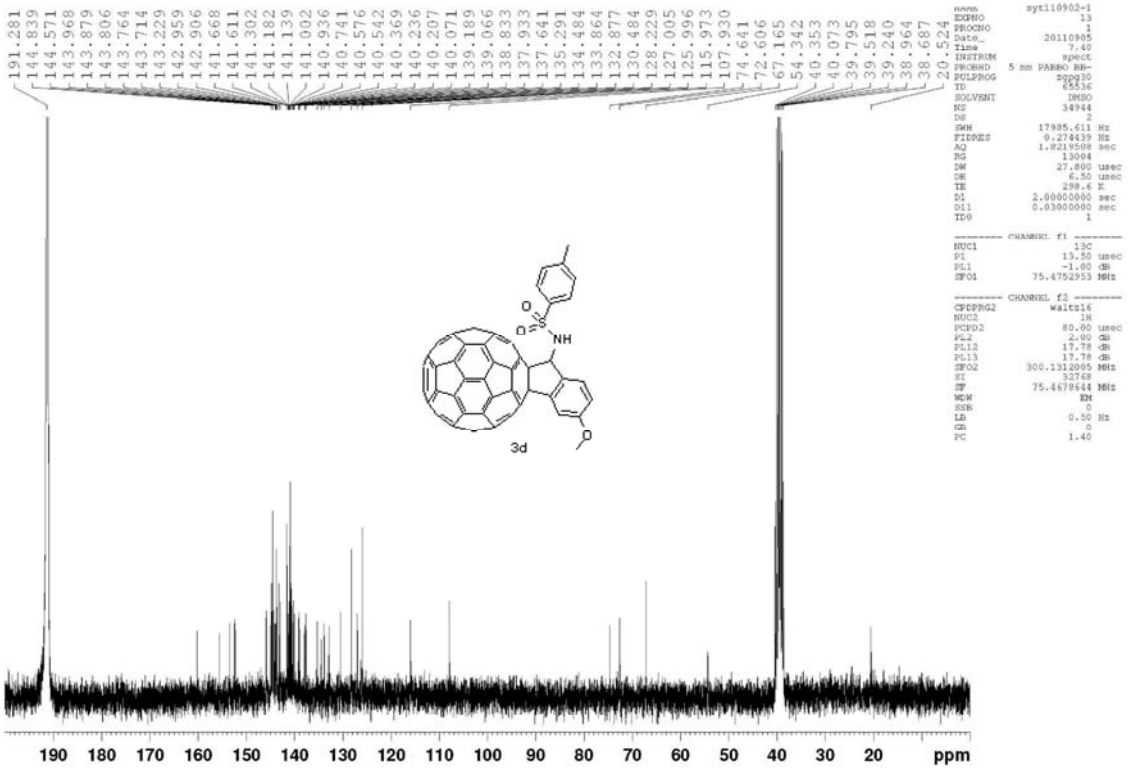

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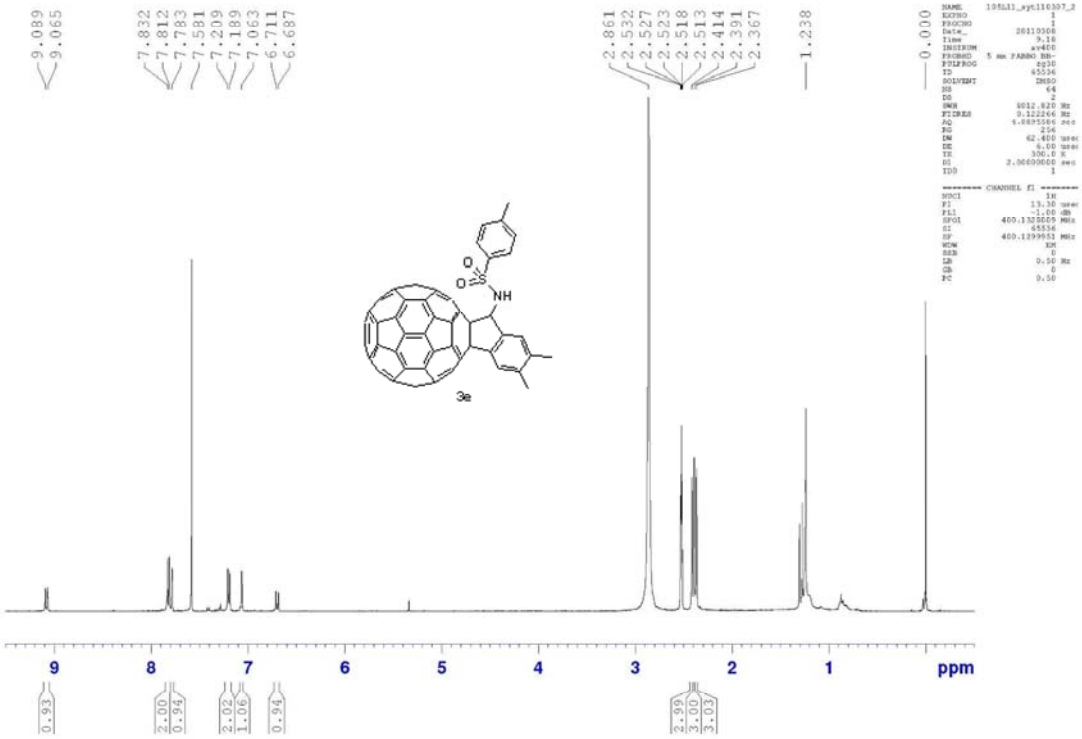

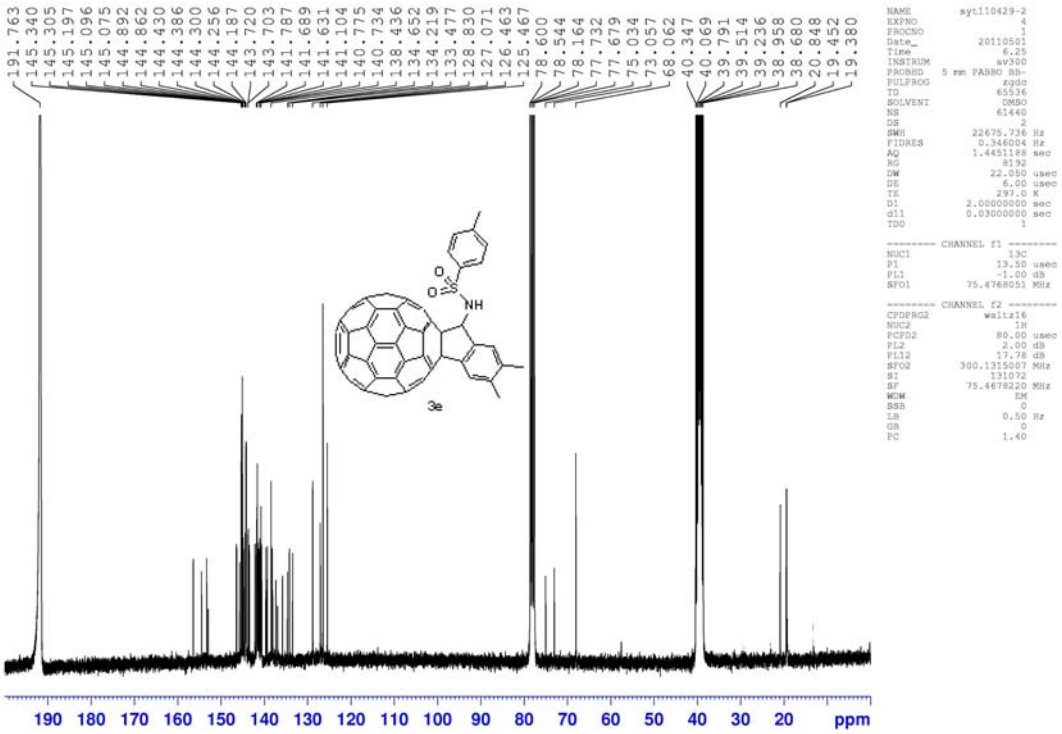

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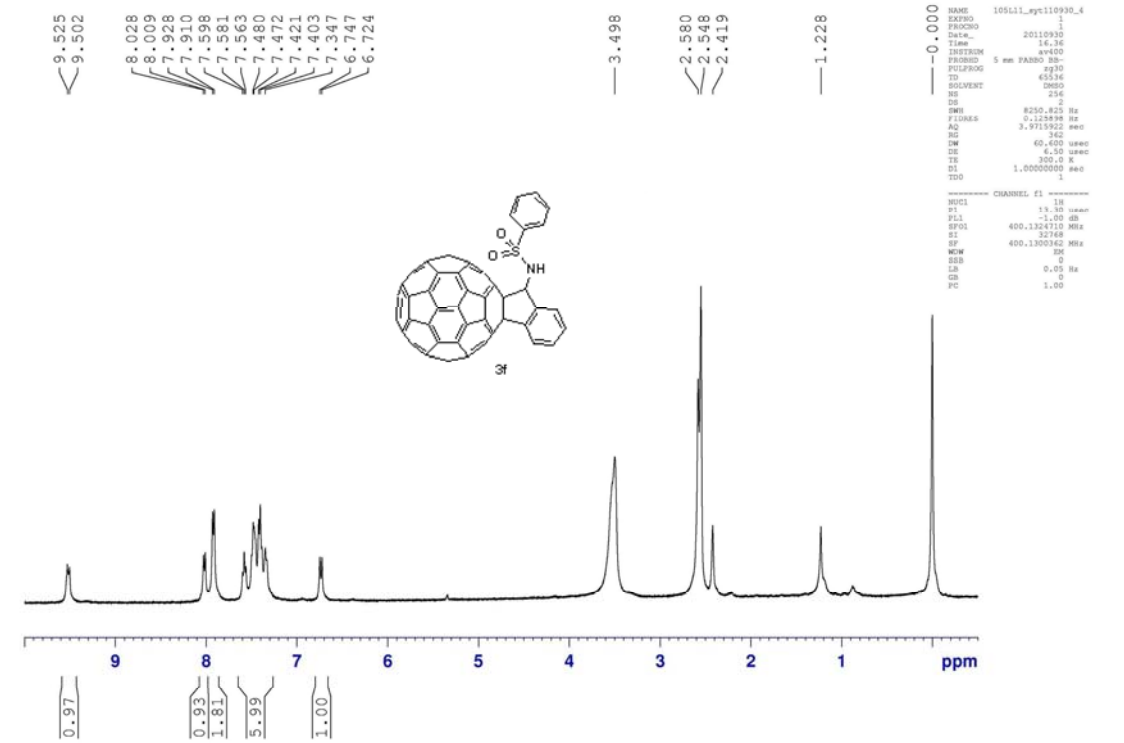

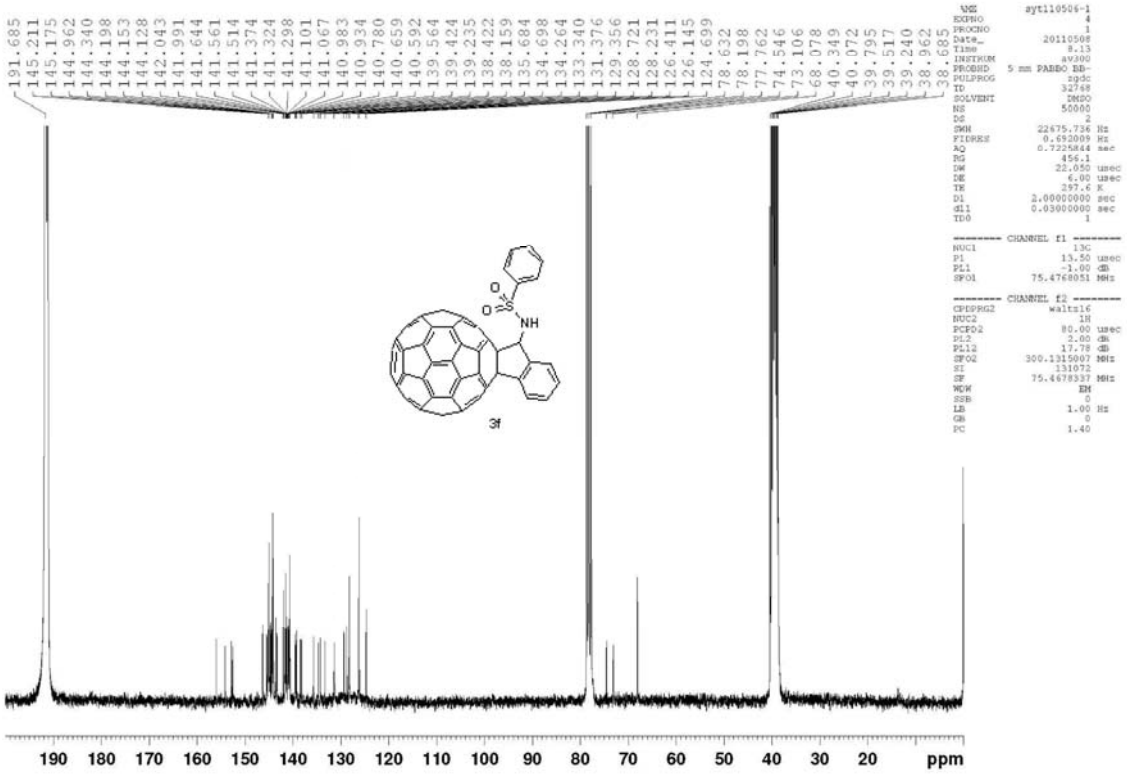

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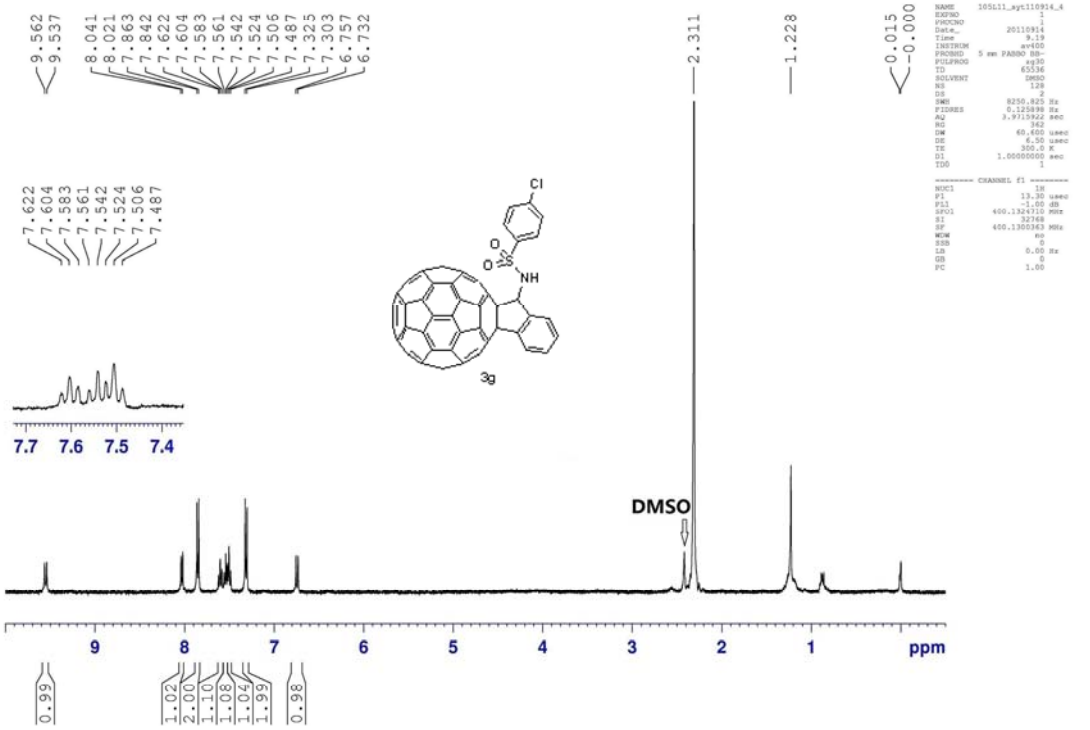

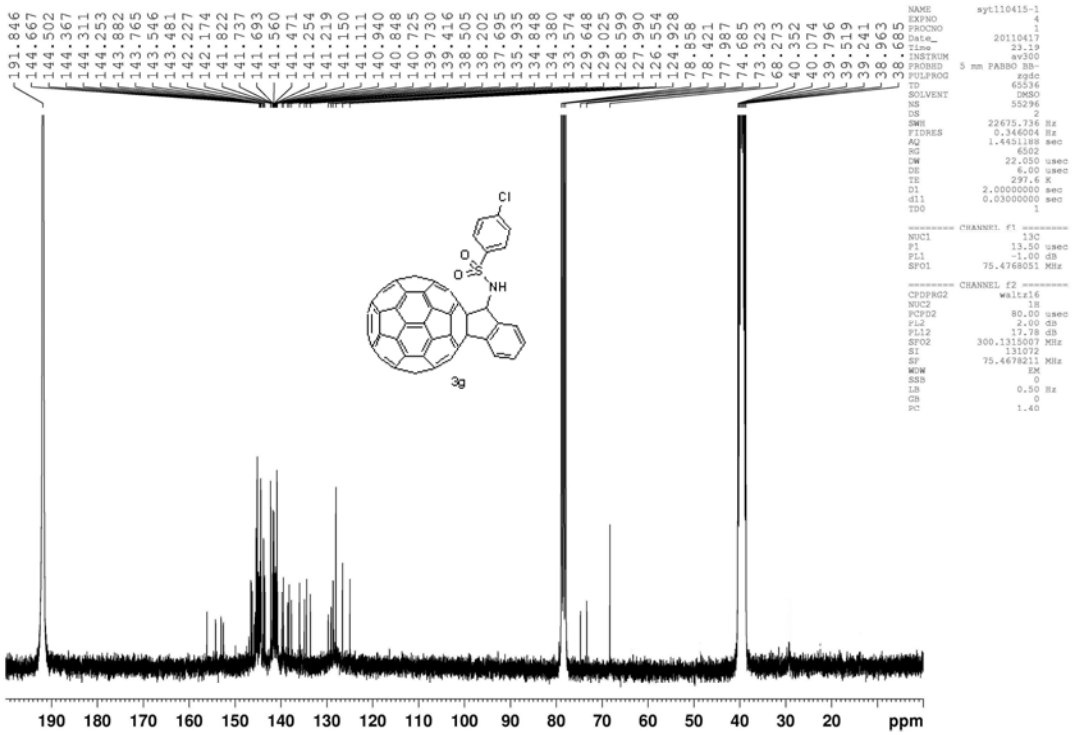

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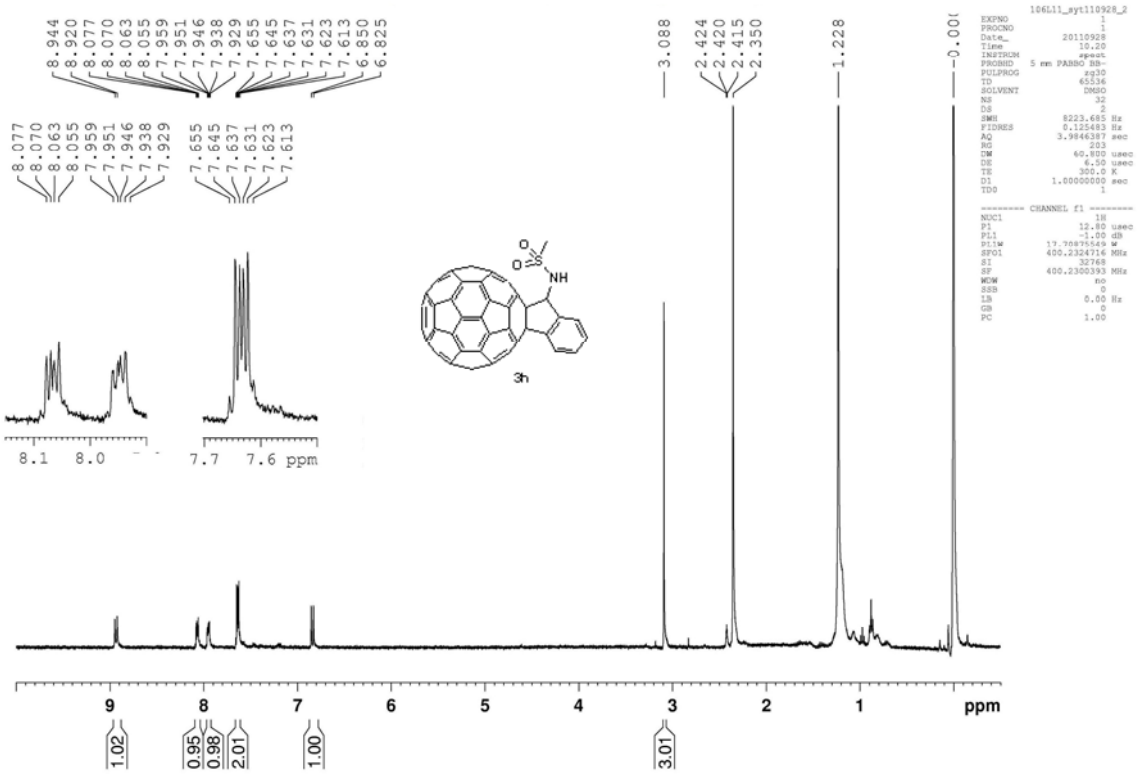

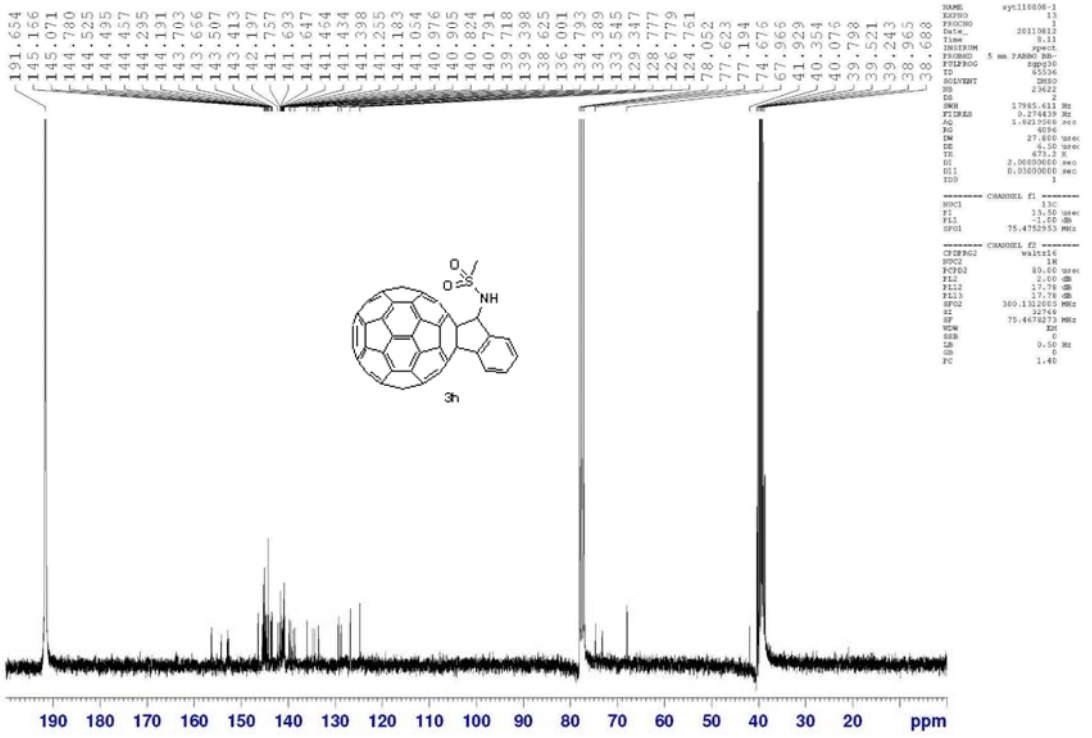

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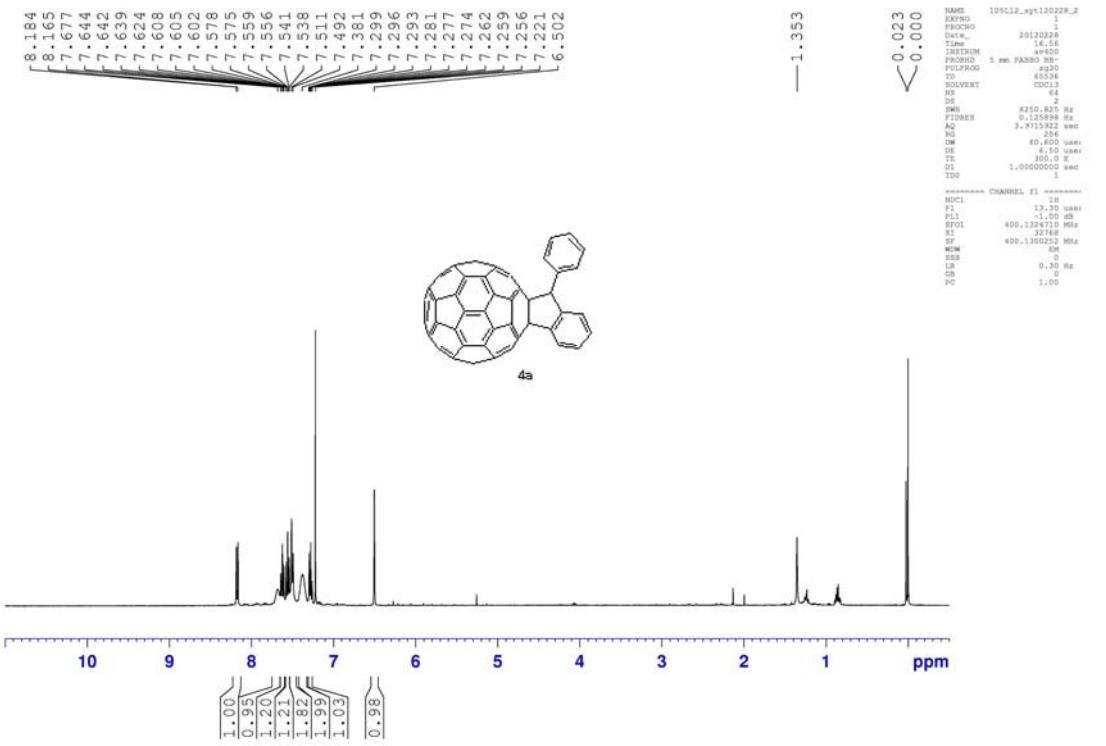

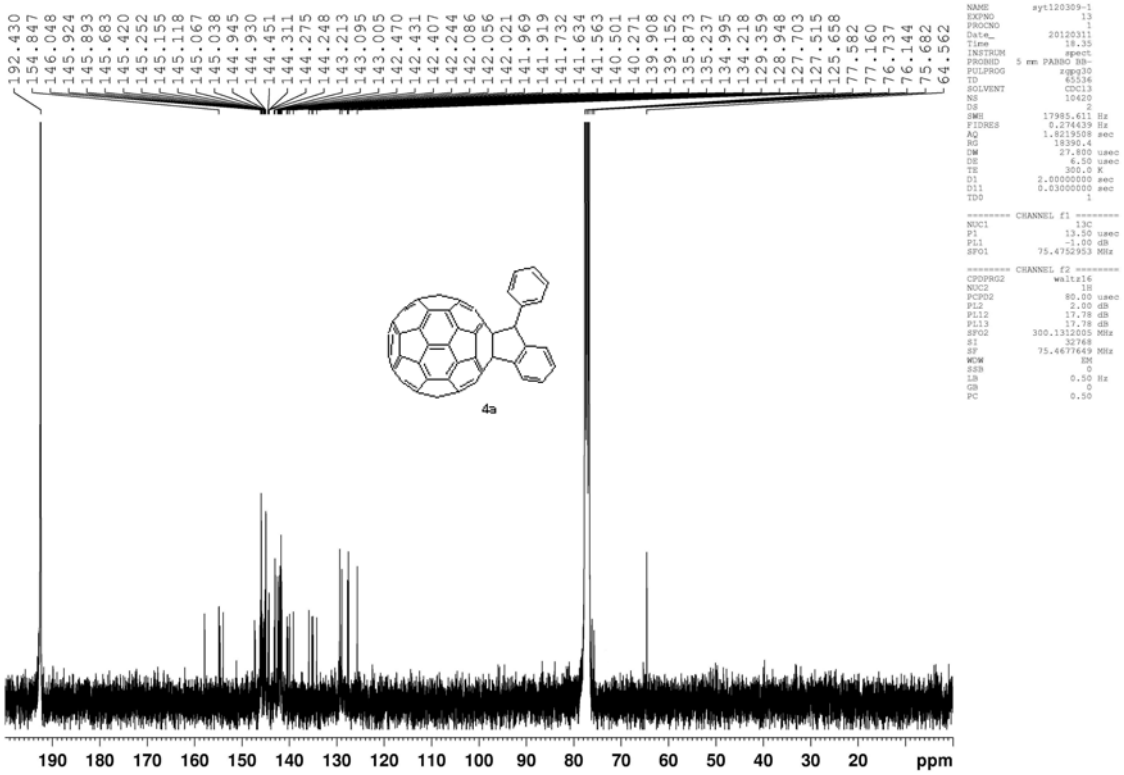

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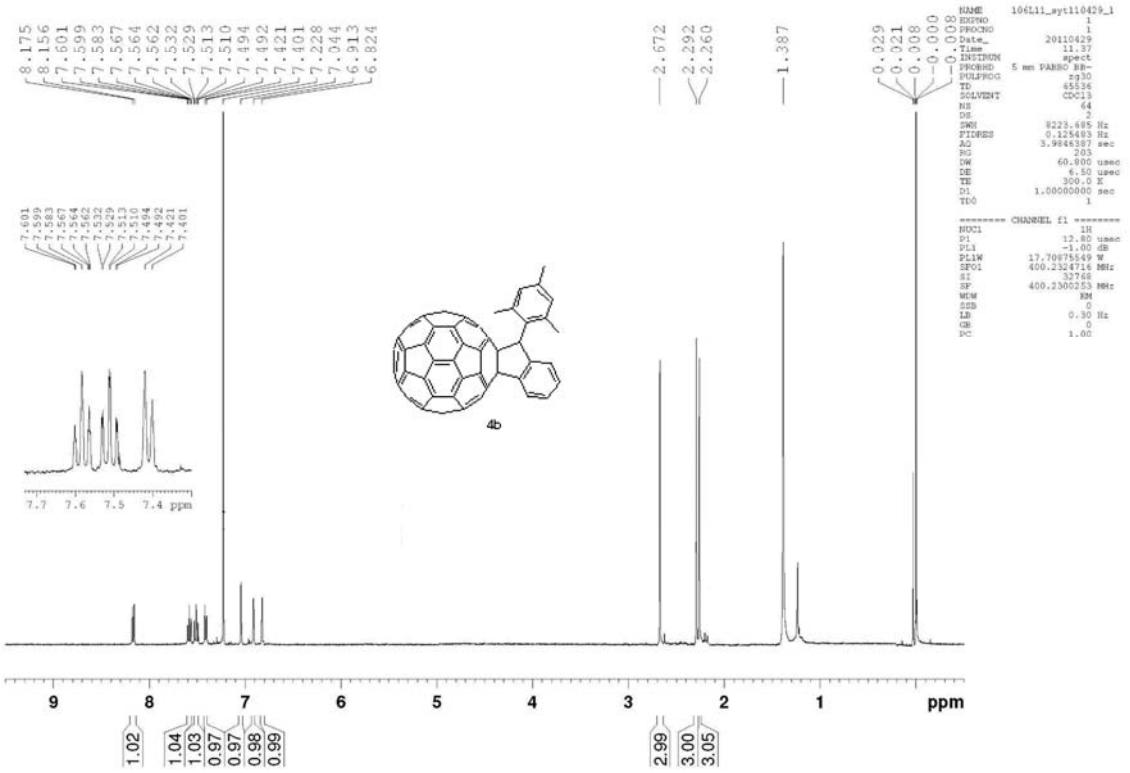

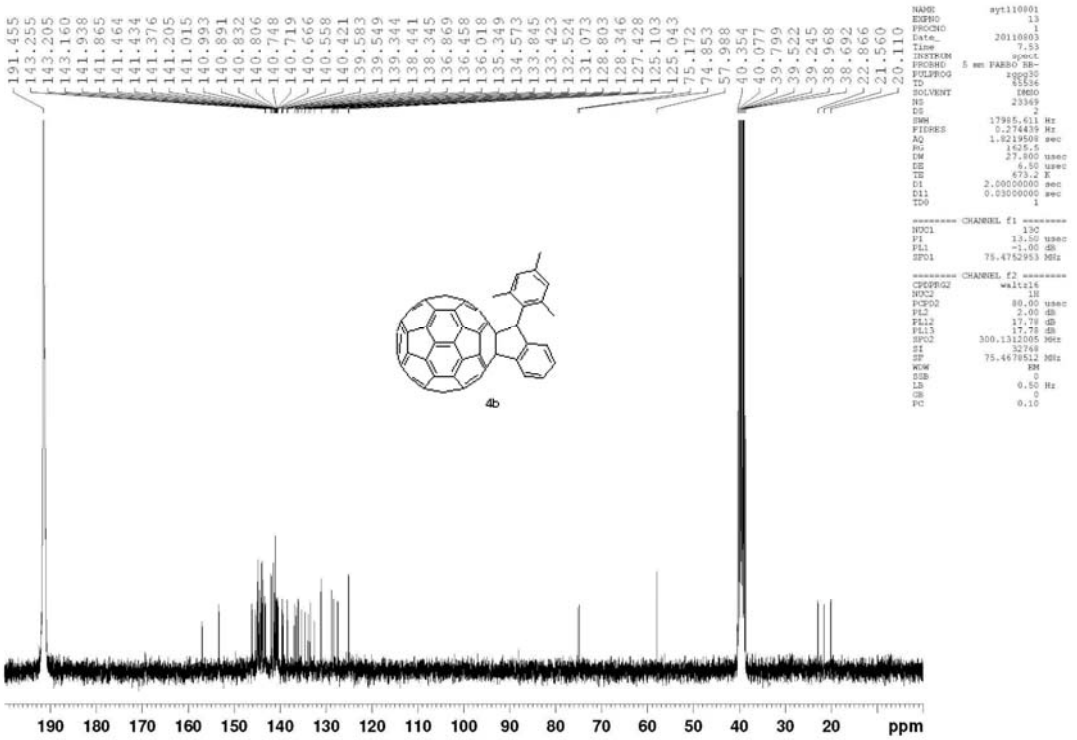

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