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Electronic Supplementary Information

One-pot, Three-component Regioselective Coupling Reaction of Triphenylamine/Carbazole Derivatives with [60]Fullerene and Indoles via an "Umpolung Relay" Strategy

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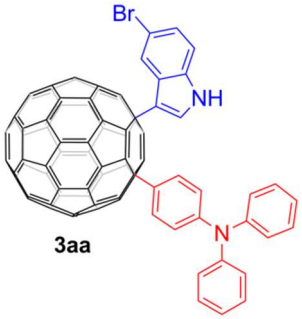
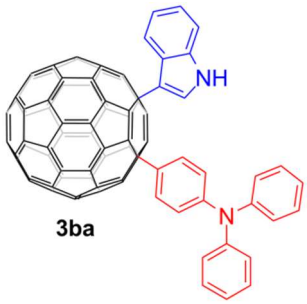
Synthesis of 3aa



3aa

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and triphenylamine (62 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (20:1) as the eluent to give unreacted C_{60} (5.2 mg, 14%) and **3aa** (38.4 mg, 66%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.55 (s, 1H), 8.41 (s, 1H), 7.92 (d, J = 8.4 Hz, 2H), 7.74 (d, J = 2.4 Hz, 1H), 7.38 (d, J = 8.8 Hz, 1H), 7.28 - 7.22 (m, 5H), 7.09 (t, J = 6.8 Hz, 6H), 7.01 (t, J = 7.2 Hz, 2H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.10, 157.00, 151.71, 151.60, 149.14(2C), 149.01, 148.85, 148.05, 147.72(3C), 147.66, 147.60(2C), 147.50(3C), 147.36, 147.30, 146.09, 146.04, 145.92(aryl C), 145.63(2C), 145.32(3C), 145.27, 145.24, 144.99, 144.94(2C), 144.87, 144.84, 144.79, 144.72, 144.62, 144.54(3C), 144.39, 143.82(aryl C), 143.72(2C), 143.67, 143.65(2C), 143.27, 143.15(4C), 142.94(aryl C), 142.74, 142.58, 141.48, 139.27, 139.14, 138.10, 137.97, 136.58(aryl C), 134.48(aryl C), 130.00(aryl C, 4C), 129.17(aryl C, 2C), 128.12(aryl C), 125.97(aryl C), 125.83(aryl C), 125.16(aryl C, 4C), 124.06(aryl C, 2C), 123.87(aryl C, 2C), 123.55(aryl C), 115.90(aryl C), 114.52(aryl C), 114.17(aryl C), 61.85(sp^3-C of C_{60}), 56.77(sp^3-C of C_{60}). UV-vis ($CHCl_3$) λmax 448, 542, 683 nm; HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{86}H_{19}BrN_2$ 1160.0724; found 1160.0718.

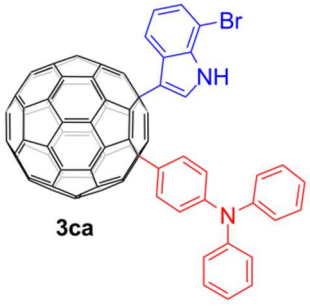
Synthesis of 3ba



3ba

C_{60} (36.0 mg, 0.05 mmol), indole (7.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and triphenylamine (62 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (20:1) as the eluent to give unreacted C_{60} (10.2 mg, 28%) and **3ba** (27.9 mg, 52%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.25 (s, 1H), 8.34 (d, J = 8.0 Hz, 1H), 7.94 (d, J = 8.4 Hz, 2H), 7.70 (s, 1H), 7.43 (d, J = 8.4 Hz, 1H), 7.25 (t, J = 7.6 Hz, 3H), 7.18 (t, J = 7.6 Hz, 2H), 7.10 (t, J = 7.6 Hz, 6H), 7.01 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.69, 157.15, 152.08, 151.84, 149.28, 149.21, 149.15, 148.97, 148.07(2C), 147.81(5C), 147.72(2C), 147.65, 147.60(2C), 147.40(2C), 146.12(aryl C, 2C), 145.92, 145.77, 145.71, 145.63, 145.43(4C), 145.02(3C), 144.95(2C), 144.83(2C), 144.60(3C), 144.51, 144.43, 143.89, 143.74(3C), 143.69, 143.25(4C), 142.92(aryl C), 142.79, 142.66, 141.45, 139.34, 139.23, 138.21, 137.98, 137.93(aryl C), 134.75(aryl C), 130.01(aryl C, 4C), 129.31(aryl C, 2C), 126.53(aryl C), 125.13(aryl C, 4C), 124.46(aryl C), 124.25(aryl C, 2C), 123.84(aryl C, 2C), 123.17(aryl C), 121.31(aryl C), 120.79(aryl C), 116.26(aryl C), 112.64(aryl C), 61.93(sp^3-C of C_{60}), 57.26(sp^3-C of C_{60}). UV-vis ($CHCl_3$) λmax 448, 542, 682 nm; HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z [M]$^-$ calcd for $C_{86}H_{20}N_2$ 1080.1632; found 1080.1641.

Synthesis of 3ca

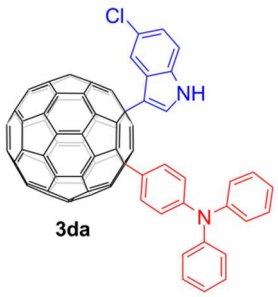


3ca

C_{60} (36.0 mg, 0.05 mmol), 7-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and triphenylamine (62 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature

and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (20:1) as the eluent to give unreacted C_{60} (10 mg, 28%) and **3ca** (38 mg, 66%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.43 (s, 1H), 8.36 (d, *J* = 8.0 Hz, 1H), 7.96 (d, *J* = 8.8 Hz, 2H), 7.84 (d, *J* = 2.4 Hz, 1H), 7.39 (d, *J* = 7.2 Hz, 1H), 7.27 (t, *J* = 7.8 Hz, 4H), 7.15 - 7.08 (m, 6H), 7.06 - 7.01 (m, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.20, 157.15, 151.79, 151.69, 149.18(2C), 149.11, 148.96, 148.18, 147.76(3C), 147.69, 147.66(3C), 147.56(2C), 147.39, 147.34, 146.12, 146.09, 145.99(aryl C), 145.66(2C), 145.46, 145.38, 145.35, 145.33, 145.30, 144.98(2C), 144.97, 144.94, 144.89, 144.87, 144.84, 144.67, 144.65, 144.62, 144.53, 144.52, 144.46, 143.84(aryl C), 143.78, 143.74, 143.72, 143.70, 143.68, 143.31, 143.23, 143.21, 143.19(2C), 142.88(aryl C), 142.81, 142.63, 141.48, 139.36, 139.20, 138.09, 136.42(aryl C), 134.36(aryl C), 130.07(aryl C, 4C), 129.20(aryl C, 2C), 127.98(aryl C), 125.68(aryl C), 125.62(aryl C), 125.20(aryl C, 4C), 124.15(aryl C, 2C), 123.96(aryl C, 2C), 121.92(aryl C), 120.67(aryl C), 117.41(aryl C), 106.32(aryl C), 61.91(sp^3-C of C_{60}), 57.02(sp^3-C of C_{60}). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{86}H_{19}BrN_2$ 1160.0724; found 1160.0729.

Synthesis of 3da

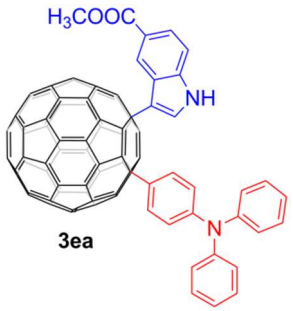


3da

C_{60} (36.0 mg, 0.05 mmol), 5-chloroindole (9 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and triphenylamine (62 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (20:1) as the eluent to give unreacted C_{60} (6.7 mg, 19%) and **3da** (35 mg, 63%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.58 (s, 1H), 8.28 (s, 1H), 7.94 (d, *J* = 8.8 Hz, 2H), 7.79 (d, *J* = 2.4 Hz, 1H), 7.44 (d, *J* = 8.8 Hz, 1H), 7.26 (t, *J* = 7.8 Hz, 4H), 7.16 - 7.09 (m, 7H), 7.03 (t, *J* = 7.2 Hz, 2H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.22, 157.13, 151.80, 151.72, 149.20, 149.18, 149.09, 148.94, 148.15, 147.78(3C),

147.71, 147.66(2C), 147.59(3C), 147.42, 147.36, 146.14, 146.10, 145.98(aryl C), 145.68(2C), 145.48, 145.38(2C), 145.34, 145.31, 145.04, 145.00(2C), 144.93(3C), 144.85, 144.77, 144.66, 144.62, 144.59(2C), 144.46, 143.87(aryl C), 143.79, 143.76, 143.74, 143.71(2C), 143.32, 143.21(3C), 142.98(aryl C), 142.80, 142.64, 141.51, 139.34, 139.22, 138.15, 138.02, 136.40(aryl C), 134.48(aryl C), 130.04(aryl C, 4C), 129.21(aryl C, 2C), 127.50(aryl C), 126.60(aryl C), 126.12(aryl C), 125.24(aryl C, 4C), 124.05(aryl C, 2C), 123.94(aryl C, 2C), 123.42(aryl C), 120.56(aryl C), 116.00(aryl C), 113.84(aryl C), 61.94(sp^3-C of C_{60}), 56.87(sp^3-C of C_{60}). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{86}H_{19}ClN_2$ 1114.1242; found 1114.1238.

Synthesis of 3ea

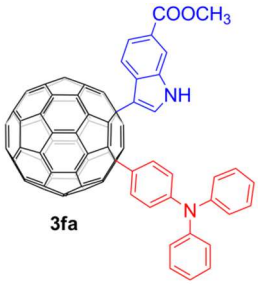


3ea

C_{60} (36.0 mg, 0.05 mmol), methyl indole-5-carboxylate (10.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and triphenylamine (62 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (5.3 mg, 15%) and **3ea** (37 mg, 65%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.43 (s, 1H), 9.04 (s, 1H), 7.90 (d, J = 8.4 Hz, 2H), 7.86 (d, J = 8.8 Hz, 1H), 7.75 (s, 1H), 7.45 (d, J = 8.8 Hz, 1H), 7.23 (t, J = 7.6 Hz, 4H), 7.07 (t, J = 8.4 Hz, 6H), 7.00 (t, J = 7.6 Hz, 2H), 3.81 (s, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 166.78(acyl C), 157.10, 156.97, 151.68, 151.55, 149.15, 149.12, 149.03, 148.81, 147.97, 147.69(2C), 147.66, 147.60, 147.57, 147.53, 147.52(2C), 147.38, 147.30, 146.10, 146.04, 145.84(aryl C), 145.63(2C), 145.40, 145.34, 145.32, 145.29, 145.21, 144.99, 144.94(2C), 144.88, 144.85, 144.84, 144.78, 144.70, 144.63, 144.54(2C), 144.52, 144.38, 143.84(aryl C), 143.72(2C), 143.68, 143.65(2C), 143.27, 143.18, 143.14(3C), 142.91(aryl C), 142.74, 142.59, 141.48, 140.28, 139.24, 139.21, 138.08, 138.03(aryl C), 134.55(aryl C), 129.96(aryl C, 4C), 129.16(aryl C, 2C), 125.98(aryl C), 125.74(aryl C), 125.07(aryl C, 4C), 124.54(aryl C), 124.13(aryl C, 2C), 124.10(aryl C), 123.80(aryl C, 2C), 122.93(aryl C),

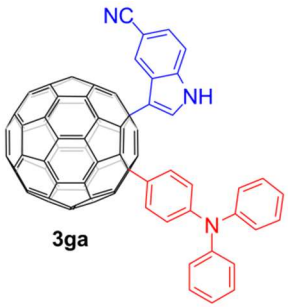
117.76(aryl C), 112.13(aryl C), 61.80(sp^3-C of C_{60}), 56.73(sp^3-C of C_{60}), 51.81(-OCH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{88}H_{22}O_2N_2$ 1138.1687; found 1138.1694.

Synthesis of 3fa



C_{60} (36.0 mg, 0.05 mmol), methyl indole-6-carboxylate (10.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and triphenylamine (62 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (3.4 mg, 9%) and **3fa** (45 mg, 79%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.73 (s, 1H), 8.35 (d, J = 8.4 Hz, 1H), 8.15 (s, 1H), 7.95 - 7.92 (m, 3H), 7.76 (dd, J = 8.4, 1.2 Hz, 1H), 7.25 (t, J = 7.8 Hz, 4H), 7.11 (t, J = 8.4 Hz, 6H), 7.02 (t, J = 7.2 Hz, 2H), 3.88 (s, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 166.96(acyl C), 157.27, 157.21, 151.80, 151.75, 149.21, 149.19, 149.15, 148.99, 148.19, 147.75(2C), 147.72, 147.68, 147.63(2C), 147.59, 147.57, 147.42, 147.37, 146.14, 146.12, 145.89(aryl C), 145.69(2C), 145.55, 145.39(2C), 145.35, 145.31, 145.00(3C), 144.95, 144.94, 144.91, 144.86, 144.72, 144.65, 144.63, 144.58, 144.56, 144.48, 143.86(aryl C), 143.80, 143.77, 143.75, 143.72(2C), 143.34, 143.22(4C), 142.94(aryl C), 142.83, 142.66, 141.51, 139.34, 139.25, 138.08, 138.01, 137.29(aryl C), 134.37(aryl C), 130.04(aryl C, 4C), 129.65(aryl C), 129.16(aryl C, 2C), 127.86(aryl C), 125.19(aryl C, 4C), 124.80(aryl C), 124.15(aryl C, 2C), 123.92(aryl C, 2C), 121.59(aryl C), 120.71(aryl C), 116.58(aryl C), 115.08(aryl C), 61.91(sp^3-C of C_{60}), 56.88(sp^3-C of C_{60}), 51.84(-OCH_3). UV-vis ($CHCl_3$) λmax 449, 538, 683 nm; HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{88}H_{22}O_2N_2$ 1138.1687; found 1138.1679.

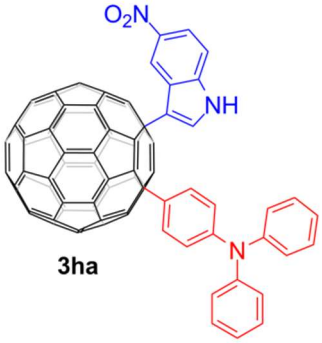
Synthesis of 3ga



3ga

C_{60} (36.0 mg, 0.05 mmol), indole-5-carbonitrile (8.6 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and triphenylamine (62 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (11.4 mg, 32%) and **3ga** (30.2 mg, 55%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-DMSO) δ 11.89 (s, 1H), 8.59 (s, 1H), 7.96 (d, J = 8.8 Hz, 2H), 7.91 (d, J = 2.0 Hz, 1H), 7.65 (d, J = 8.4 Hz, 1H), 7.44 (d, J = 8.4 Hz, 1H), 7.29 (t, J = 7.8 Hz, 4H), 7.15 - 7.12 (m, 6H), 7.05 (t, J = 7.2 Hz, 2H). ^{13}C NMR (100 MHz, CS_2/d_6-DMSO) (all 1C unless indicated) δ 155.82, 155.74, 150.42, 150.26, 147.89(2C), 147.69, 147.63, 146.82, 146.40(4C), 146.36, 146.29, 146.27(2C), 146.14(2C), 146.05(aryl C), 144.84, 144.79, 144.67, 144.37(2C), 144.06(2C), 144.03, 143.97(aryl C), 143.86, 143.73, 143.68(2C), 143.63, 143.56(3C), 143.41, 143.37, 143.30(3C), 143.16(aryl C), 142.55, 142.47(2C), 142.43(3C), 142.03, 141.89(4C), 141.71, 141.50, 141.32, 140.26, 138.35, 138.05, 137.84, 136.76(aryl C), 136.64(aryl C), 132.96(aryl C), 128.83(aryl C, 4C), 127.88(aryl C, 2C), 126.22(aryl C), 124.81(aryl C), 124.64(aryl C), 123.96(aryl C, 4C), 122.83(aryl C, 2C), 122.69(aryl C, 2C), 119.01(-CN), 115.07(aryl C), 112.93(aryl C), 102.62(aryl C), 60.57(sp^3-C of C_{60}), 55.34(sp^3-C of C_{60}). UV-vis ($CHCl_3$) λmax 448, 543, 682 nm; HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{87}H_{19}N_3$ 1105.1579; found 1105.1572.

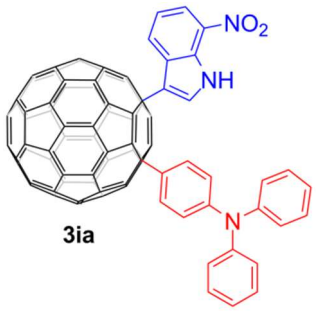
Synthesis of 3ha



3ha

C_{60} (36.0 mg, 0.05 mmol), 5-nitroindole (9.8 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and triphenylamine (62 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (3:1) as the eluent to give unreacted C_{60} (12.7 mg, 35%) and **3ha** (27.4 mg, 49%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 11.02 (s, 1H), 9.29 (s, 1H), 8.10 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.98 - 7.89 (m, 3H), 7.60 (d, *J* = 8.8 Hz, 1H), 7.25 (t, *J* = 7.8 Hz, 4H), 7.09 (t, *J* = 8.8 Hz, 5H), 7.02 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.13, 156.79, 151.82, 151.24, 149.28(2C), 148.98, 148.91, 148.25, 147.76(5C), 147.64(4C), 147.52, 147.41(2C), 146.24(3C), 145.80(aryl C), 145.73, 145.41(2C), 145.18, 145.04(5C), 144.92(2C), 144.81(2C), 144.69(4C), 144.54, 143.94(aryl C), 143.81(4C), 143.40, 143.28(2C), 143.24(2C), 143.08(aryl C), 142.87, 142.64(3C), 141.70, 140.81, 139.50, 139.15(aryl C), 138.10(aryl C), 134.32(aryl C), 130.05(aryl C, 4C), 129.13(aryl C, 2C), 127.79(aryl C), 125.72(aryl C), 125.26(aryl C, 4C), 124.08(aryl C, 2C), 123.99(aryl C, 2C), 118.85(aryl C), 118.48(aryl C), 118.17(aryl C), 112.72(aryl C), 61.96(sp^3-C of C_{60}), 56.47(sp^3-C of C_{60}). UV-vis ($CHCl_3$) λmax 447, 539, 684 nm; HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{86}H_{19}O_2N_3$ 1125.1483; found 1125.1475.

Synthesis of 3ia

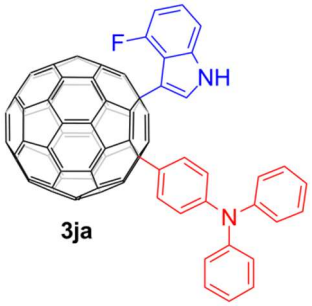


3ia

C_{60} (36.0 mg, 0.05 mmol), 7-nitroindole (9.8 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and triphenylamine (62 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water

and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (16.0 mg, 44%) and **3ia** (24 mg, 43%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-DMSO) δ 12.16 (s, 1H), 8.80 (d, *J* = 8.0 Hz, 1H), 8.20 (d, *J* = 7.6 Hz, 1H), 7.97-7.93 (m, 3H), 7.31-7.27 (m, 5H), 7.17-7.12 (m, 6H), 7.05 (t, *J* = 7.2 Hz, 2H). ^{13}C NMR (100 MHz, CS_2/d_6-DMSO) δ 155.85, 155.52, 150.41, 150.12, 147.87(2C), 147.63(2C), 146.89, 146.38(5C), 146.27, 146.24(3C), 146.20, 146.09, 146.05(aryl C), 144.82(2C), 144.39, 144.36, 144.05, 144.02, 143.95, 143.91(aryl C), 143.82, 143.66(4C), 143.55, 143.51, 143.38, 143.30(aryl C), 143.25(2C), 143.19(2C), 142.52(2C), 142.49, 142.45, 142.42(2C), 142.38, 142.03, 141.91(2C), 141.86(2C), 141.60, 141.53, 141.31, 140.24(aryl C), 138.13, 137.72, 136.77, 136.69, 132.76(aryl C), 129.37(aryl C), 129.33(aryl C), 128.84(aryl C, 4C), 127.82(aryl C, 2C), 127.60(aryl C), 127.47(aryl C), 123.95(aryl C, 4C), 122.90(aryl C, 2C), 122.72(aryl C, 2C), 118.62(aryl C), 118.32(aryl C), 115.72(aryl C), 60.57(sp^3-C of C_{60}), 55.14(sp^3-C of C_{60}). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{86}H_{19}O_2N_3$ 1125.1483; found 1125.1492.

Synthesis of 3ja

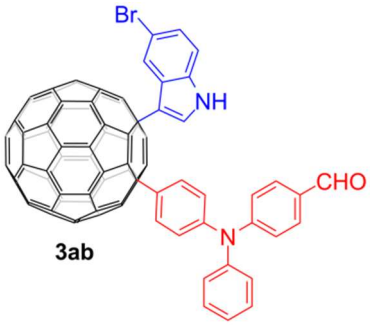


3ja

C_{60} (36.0 mg, 0.05 mmol), 4-fluoroindole (8 μL, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and triphenylamine (62 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (8.0 mg, 22%) and **3ja** (27.5 mg, 50%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.68 (s, 1H), 7.95 (d, *J* = 8.4 Hz, 2H), 7.81 (d, *J* = 2.4 Hz, 1H), 7.29 - 7.25 (m, 5H), 7.16 (dd, *J* = 7.8, 4.9 Hz, 1H), 7.11 (t, J = 8.0 Hz, 6H), 7.03 (t, *J* = 7.2 Hz, 2H), 6.81 (dd, J = 11.6, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) δ 157.63, 156.83(aryl C, d, J = 247.6 Hz), 157.21, 152.45, 151.82, 149.82, 149.20, 149.17, 148.78(2C), 148.10, 147.86(3C), 147.81, 147.68, 147.66, 147.60(2C), 147.46, 147.34, 146.51, 146.16(2C), 145.87(aryl C), 145.74(2C), 145.48, 145.42, 145.35,

145.14, 145.05(3C), 145.02, 144.92, 144.78, 144.70, 144.64, 144.53, 144.45, 144.43, 144.08, 143.97(aryl C), 143.86, 143.75(2C), 143.50, 143.39, 143.37, 143.20(aryl C), 143.15, 143.08, 142.78(2C), 142.20, 141.32, 141.07, 140.95, 139.93(aryl C), 139.35(aryl C), 139.28, 138.20, 134.61, 130.07(aryl C, 4C), 129.30(aryl C, 2C), 126.25(aryl C), 125.17(aryl C, 4C), 124.20(aryl C, 2C), 123.91(aryl C, 2C), 123.79(aryl C, d, J = 7.4 Hz), 116.00(aryl C), 115.30(aryl C, d, J = 21.1 Hz), 108.94(aryl C), 106.01(aryl C, d, J = 20.8 Hz), 61.86(sp^3-C of C_{60}), 56.86(sp^3-C of C_{60}). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{86}H_{19}FN_2$ 1098.1538; found 1098.1546.

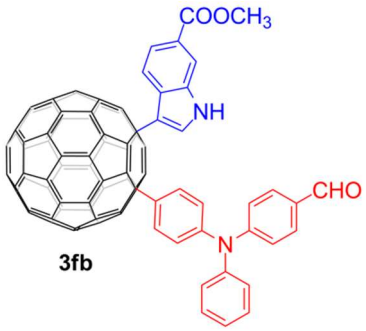
Synthesis of 3ab



C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4-(N, N-diphenylamino) benzaldehyde (69 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 100 °C for 120 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (1:1) as the eluent to give unreacted C_{60} (6.3 mg, 18%) and **3ab** (30.2 mg, 51%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.50 (s, 1H), 9.78 (s, 1H), 8.41 (s, 1H), 8.03 (d, *J* = 8.8 Hz, 2H), 7.74 (d, *J* = 2.4 Hz, 1H), 7.65 (d, *J* = 8.8 Hz, 2H), 7.38 – 7.33 (m, 3H), 7.29 - 7.26 (m, 1H), 7.23 (d, *J* = 8.4 Hz, 2H), 7.19-7.17 (m, 3H), 7.09 (d, *J* = 8.8 Hz, 2H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 188.36(acyl C), 157.01, 156.52, 152.79(aryl C), 151.50, 151.34, 149.19, 149.13, 148.95, 148.74, 147.69, 147.61, 147.53, 147.50, 147.42, 147.39, 147.33, 146.56, 146.41, 146.12, 146.07(aryl C, 2C), 145.70, 145.64, 145.57, 145.49, 145.28(3C), 145.19, 145.11, 145.03, 144.93(2C), 144.87(2C), 144.74(2C), 144.62, 144.58, 144.54, 144.30, 143.84, 143.74(3C), 143.69(2C), 143.30, 143.20, 143.16, 143.11, 143.09, 142.96(aryl C), 142.73, 142.57, 141.90, 141.57, 139.27, 139.16, 138.23, 137.92, 137.04, 136.53(aryl C), 131.49(aryl C, 2C), 130.67(aryl C), 130.42(aryl C, 2C), 129.58(aryl C, 2C), 128.04(aryl C), 126.77(aryl C, 2C), 126.50(aryl C, 2C), 126.08(aryl C), 125.72(aryl C), 125.66(aryl C), 123.54(aryl C),

120.91(aryl C, 2C), 115.85(aryl C), 114.62(aryl C), 114.10(aryl C), 61.72(sp^3-C of C_{60}), 56.75(sp^3-C of C_{60}). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{87}H_{19}BrON_2$ 1188.0673; found 1188.0665.

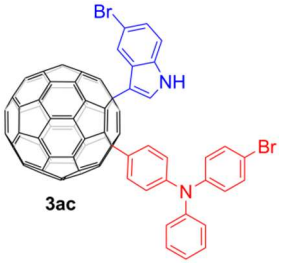
Synthesis of 3fb



3fb

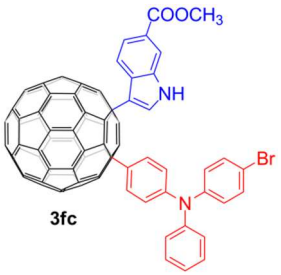
C_{60} (36.0 mg, 0.05 mmol), methyl indole-6-carboxylate (10.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in 1,2-dichlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4-(N, N-diphenylamino) benzaldehyde (69 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 150 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (1:1) as the eluent to give unreacted C_{60} (4.5 mg, 15%) and **3fb** (20.2 mg, 35%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-DMSO) δ 11.71(s, 1H), 9.98(s, 1H), 8.30 (d, J = 8.4 Hz, 1H), 8.21 (s, 1H), 8.07 (d, J = 8.4 Hz, 2H), 7.89 (s, 1H), 7.72-7.70 (m, 2H), 7.71 (d, J = 2.0 Hz, 1H), 7.39 - 7.37 (m, 2H), 7.26 (d, J = 8.4 Hz, 2H), 7.23-7.21 (m, 3H), 7.11 (d, J = 8.8 Hz, 2H) 3.92 (s, 3H). ^{13}C NMR (100 MHz, d_6-DMSO) δ 187.59(acyl C), 165.88(acyl C), 156.02, 155.49, 151.57(aryl C), 150.58, 150.10, 147.92, 147.85, 147.78, 147.62, 146.41, 146.36, 146.28(3C), 146.23, 146.10(aryl C), 146.06(aryl C), 145.32, 145.14, 144.81(2C), 144.67, 144.44, 144.34, 144.05(2C), 144.02(2C), 143.91, 143.75, 143.67(2C), 143.62(3C), 143.48, 143.45, 143.30(3C), 143.26, 143.07(aryl C), 142.56, 142.47(4C), 142.38, 142.03, 141.92(2C), 141.89, 141.86, 141.64, 141.50, 141.32, 140.24, 138.08, 137.87, 136.90, 136.63, 136.16, 135.67(aryl C), 130.47(aryl C, 2C), 129.34(aryl C), 129.28(aryl C, 2C), 128.34(aryl C, 2C), 128.28(aryl C), 127.05(aryl C), 125.62(aryl C, 2C), 125.35(aryl C, 2C), 124.55(aryl C), 122.97(aryl C), 119.97(aryl C), 119.61(aryl C, 2C), 119.16(aryl C), 114.39(aryl C), 114.26(aryl C), 60.45(sp^3-C of C_{60}), 55.68(sp^3-C of C_{60}), 50.69(-OCH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{89}H_{22}O_3N_2$ 1166.1636; found 1166.1651.

Synthesis of 3ac



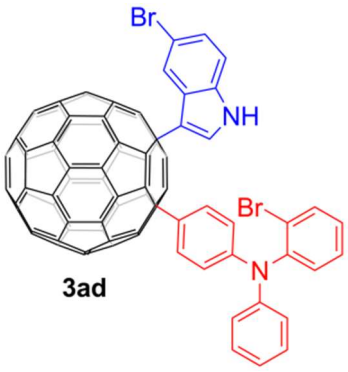
C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4-bromotriphenylamine (81 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 110 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (4 mg, 11%) and **3ac** (31 mg, 50%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.47 (s, 1H), 8.41 (s, 1H), 7.95 (d, J = 8.4 Hz, 2H), 7.73 (d, J = 2.8 Hz, 1H), 7.37 (d, J = 8.8 Hz, 1H), 7.33 (d, J = 8.8 Hz, 2H), 7.29 - 7.25 (m, 3H), 7.13-7.09 (m, 4H), 7.05 (t, J = 7.2 Hz, 1H), 6.99 (d, J = 8.8 Hz, 2H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.03, 156.76, 151.53, 151.50, 149.13, 149.09, 148.95, 148.76, 147.64, 147.57, 147.52, 147.50, 147.47(2C), 147.42, 147.34, 147.28, 147.20, 146.87, 146.07, 146.02(aryl C), 145.73, 145.62, 145.60, 145.37, 145.27(2C), 145.24, 145.13, 144.97, 144.90(2C), 144.83(2C), 144.76, 144.74, 144.68, 144.58, 144.51(3C), 144.32, 143.79(aryl C), 143.69(3C), 143.63(2C), 143.25, 143.16, 143.12, 143.09(2C), 142.92(aryl C), 142.70, 142.54, 141.48, 139.18, 139.16, 138.10, 137.89, 136.49(aryl C), 135.19(aryl C), 132.84(aryl C, 2C), 130.15(aryl C, 2C), 129.33(aryl C, 2C), 128.04(aryl C), 126.02(aryl C), 125.91(aryl C, 2C), 125.67(aryl C), 125.33(aryl C, 2C), 124.51(aryl C, 2C), 124.35(aryl C), 123.53(aryl C), 116.21(aryl C), 115.90(aryl C), 114.56(aryl C), 114.07(aryl C), 61.74(sp^3-C of C_{60}), 56.70(sp^3-C of C_{60}). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z [M]$^-$ calcd for $C_{86}H_{18}Br_2N_2$ 1237.9838; found 1237.9826.

Synthesis of 3fc



C_{60} (36.0 mg, 0.05 mmol), methyl indole-6-carboxylate (10.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4-bromotriphenylamine (81 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 110 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (5.3 mg, 15%) and **3fc** (35 mg, 58%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.79 (s, 1H), 8.35 (d, J = 8.4 Hz, 1H), 8.17 (s, 1H), 7.99 (d, J = 8.0 Hz, 2H), 7.95 (d, J = 2.4 Hz, 1H), 7.76 (d, J = 8.4 Hz, 1H), 7.35 (d, J = 8.4 Hz, 2H), 7.29 (t, J = 7.8 Hz, 2H), 7.15 (d, J = 8.8 Hz, 2H), 7.12 (d, J = 8.0 Hz, 2H), 7.07 (t, J = 7.6 Hz, 1H), 7.01 (d, J = 8.8 Hz, 2H), 3.89 (s, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 167.02(acyl), 157.32, 157.12, 151.79, 151.71, 149.26, 149.23, 149.17, 149.00, 147.76, 147.73(2C), 147.63(4C), 147.46, 147.42, 147.33(2C), 147.03, 146.18, 146.17, 145.78(aryl C), 145.74, 145.70(2C), 145.43, 145.41, 145.38, 145.31, 145.07, 145.03(2C), 144.99(aryl C), 144.95(2C), 144.88, 144.76, 144.68, 144.65, 144.61(2C), 144.49, 143.90, 143.80(3C), 143.76(2C), 143.38, 143.28(2C), 143.24(2C), 142.98(aryl C), 142.86, 142.69, 141.56, 139.34, 138.13, 138.00, 137.33(aryl C), 135.13(aryl C), 132.95(aryl C, 2C), 130.27(aryl C, 2C), 129.66(aryl C), 129.37(aryl C, 2C), 127.94(aryl C), 126.05(aryl C, 2C), 125.48(aryl C, 2C), 124.83(aryl C), 124.64(aryl C, 2C), 124.50(aryl C), 121.59(aryl C), 120.71(aryl C), 116.5(aryl C), 116.25(aryl C), 115.13(aryl C), 61.90(sp^3-C of C_{60}), 56.92(sp^3-C of C_{60}), 51.89(-OCH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{88}H_{21}BrO_2N_2$ 1218.0779; found 1218.0765.

Synthesis of 3ad

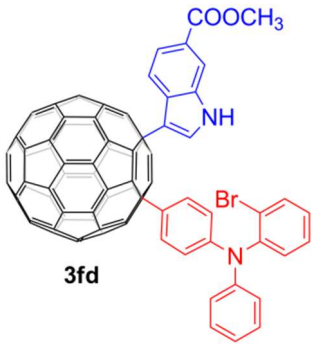


3ad

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed

from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 2-bromotriphenylamine (81 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (4 mg, 11%) and **3ad** (40 mg, 65%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.48 (s, 1H), 8.41 (s, 1H), 7.90 (d, *J* = 8.8 Hz, 2H), 7.71 (d, *J* = 2.4 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.41 - 7.35 (m, 2H), 7.28 - 7.26 (m, 2H), 7.24 - 7.16 (m, 3H), 7.01 - 6.96 (m, 5H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.05(2C), 151.72, 151.58, 149.11, 149.08, 148.97, 148.85, 147.62, 147.58(2C), 147.50(2C), 147.47, 147.33, 147.26, 147.17, 146.62, 146.05, 146.00(aryl C), 145.91, 145.60(2C), 145.43, 145.32, 145.30(2C), 145.25, 145.22, 144.95(2C), 144.92(2C), 144.84(3C), 144.76, 144.67, 144.57, 144.53(2C), 144.48, 144.37, 143.79(aryl C), 143.70, 143.67, 143.64, 143.61(2C), 143.23, 143.15, 143.12(2C), 142.89(aryl C), 142.71, 142.55, 141.43, 139.24, 139.09, 138.04, 137.98, 136.50(aryl C), 135.04(aryl C), 133.81(aryl C), 132.19(aryl C), 129.75(aryl C, 2C), 129.47(aryl C), 128.99(aryl C, 2C), 128.06(aryl C, 2C), 125.93(aryl C), 125.77(aryl C), 124.55(aryl C), 123.50(aryl C), 123.34(aryl C), 123.24(aryl C, 2C), 122.03(aryl C, 2C), 115.84(aryl C), 114.49(aryl C), 114.11(aryl C), 61.79(sp^3-C of C_{60}), 56.72(sp^3-C of C_{60}). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{86}H_{18}Br_2N_2$ 1237.9838; found 1237.9847.

Synthesis of 3fd

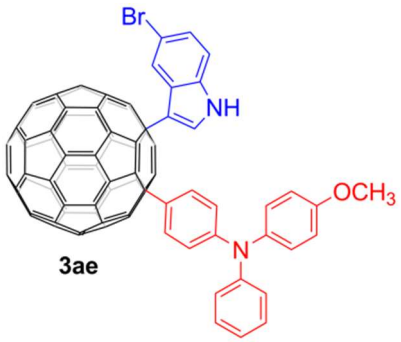


3fd

C_{60} (36.0 mg, 0.05 mmol), methyl indole-6-carboxylate (10.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 2-bromotriphenylamine (81 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted

C_{60} (6 mg, 17%) and **3fd** (40.2 mg, 66%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.74 (s, 1H), 8.34 (d, *J* = 8.4 Hz, 1H), 8.15 (s, 1H), 7.93 - 7.89 (m, 3H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.40 (t, *J* = 7.2 Hz, 1H), 7.29 (d, *J* = 7.6 Hz, 1H), 7.24 - 7.17 (m, 3H), 7.01 (d, *J* = 8.0 Hz, 2H), 6.98 (d, *J* = 8.4 Hz, 3H), 3.88 (s, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 167.02(acyl C), 157.30, 157.25, 151.85, 151.77, 149.19(2C), 149.14, 149.02, 147.70, 147.66(2C), 147.58(2C), 147.41, 147.36(2C), 146.69, 146.13(2C), 145.94(aryl C), 145.67(2C), 145.55, 145.48, 145.40(2C), 145.34(2C), 145.01(3C), 144.95(2C), 144.90, 144.86, 144.70, 144.64(2C), 144.58, 144.55, 144.50, 143.85(aryl C), 143.80, 143.71(4C), 143.32, 143.23(4C), 142.92(aryl C), 142.83, 142.65, 141.49, 139.36, 139.23, 138.05, 137.29, 135.11, 133.70(aryl C), 132.29(aryl C), 129.82(aryl C, 3C), 129.66(aryl C), 129.60(aryl C), 129.01(aryl C, 2C), 128.21(aryl C), 127.92(aryl C), 124.75(aryl C), 124.63(aryl C), 123.45(aryl C), 123.38(aryl C, 3C), 122.05(aryl C, 2C), 121.59(aryl C), 120.71(aryl C), 116.55(aryl C), 115.09(aryl C), 61.89(sp^3-C of C_{60}), 56.88(sp^3-C of C_{60}), 51.85(-OCH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{88}H_{21}BrO_2N_2$ 1218.0779; found 1218.0791.

Synthesis of 3ae

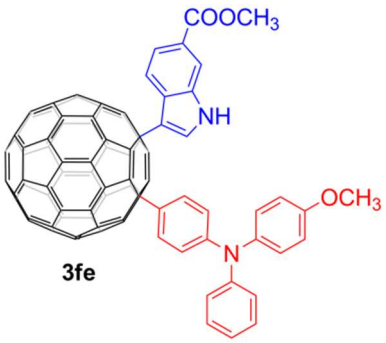


3ae

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4-methoxytriphenylamine (68 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (5 mg, 14%) and **3ae** (45 mg, 76%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.51 (s, 1H), 8.41 (s, 1H), 7.87 (d, *J* = 8.4 Hz, 2H), 7.74 (d, *J* = 2.4 Hz, 1H), 7.38 (d, *J* = 8.4 Hz, 1H), 7.27 (dd, J = 8.8, 1.2 Hz, 1H), 7.20 (t, *J* = 7.8 Hz, 2H), 7.04 (dd, *J* = 8.0, 2.4 Hz, 6H), 6.95 (t, *J* = 7.2 Hz, 1H), 6.83 (d, *J* = 8.8 Hz, 2H), 3.79 (s, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.14(2C), 157.01(aryl C), 151.84, 151.63, 149.15(2C), 149.05, 148.89, 148.41,

147.95, 147.66, 147.62(2C), 147.54(2C), 147.51, 147.37, 147.31, 146.10, 146.05(aryl C, 2C), 145.65, 145.63, 145.34(2C), 145.29(3C), 145.00, 144.96(2C), 144.89, 144.86(2C), 144.81, 144.73, 144.64, 144.59, 144.56, 144.53, 144.43, 143.83, 143.74, 143.72, 143.66(3C), 143.28, 143.17(4C), 142.95, 142.76, 142.59, 141.47, 140.44, 139.31, 139.12, 138.07, 138.00(aryl C), 136.59(aryl C), 133.54(aryl C), 129.86(aryl C, 2C), 129.05(aryl C, 2C), 128.14(aryl C), 128.00(aryl C, 2C), 125.98(aryl C), 125.87(aryl C), 124.14(aryl C, 2C), 123.57(aryl C), 123.18(aryl C), 122.77(aryl C, 2C), 115.95(aryl C), 115.51(aryl C, 2C), 114.50(aryl C), 114.19(aryl C), 61.88(sp^3-C of C_{60}), 56.77(sp^3-C of C_{60}), 55.53(-OCH_3). UV-vis ($CHCl_3$) λmax 449, 540, 683 nm;HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{87}H_{21}BrON_2$ 1190.0829; found 1190.0819.

Synthesis of 3fe

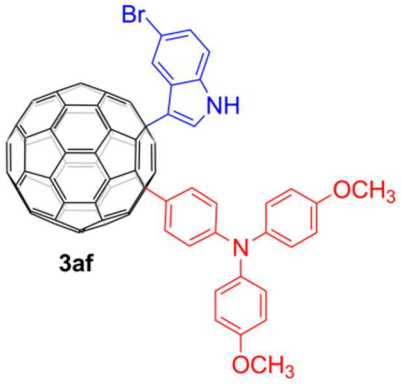


3fe

C_{60} (36.0 mg, 0.05 mmol), methyl indole-6-carboxylate (10.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4-methoxytriphenylamine (68 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane /ethyl acetate(12:5:1) as the eluent to give unreacted C_{60} (5 mg, 14%) and **3fe** (45 mg, 77%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.69 (s, 1H), 8.33 (d, J = 8.4 Hz, 1H), 8.15 (s, 1H), 7.89 (d, J = 2.0 Hz, 2H), 7.87 (s, 1H), 7.75 (d, J = 8.4 Hz, 1H), 7.20 (t, J = 7.8 Hz, 2H), 7.05 (d, J = 8.0 Hz, 6H), 6.94 (t, J = 7.2 Hz, 1H), 6.82 (d, J = 8.8 Hz, 2H), 3.88 (s, 3H), 3.79 (s, 3H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 167.02(acyl C), 157.28, 157.22, 157.03, 151.85, 151.71, 149.15(2C), 149.12, 148.97, 148.50, 147.96, 147.67, 147.64, 147.60(2C), 147.54(2C), 147.38, 147.33, 146.08(2C), 145.96(aryl C), 145.64(2C), 145.44, 145.36(2C), 145.31, 145.28, 144.98(3C), 144.92, 144.89, 144.87, 144.83, 144.67, 144.63, 144.59, 144.54, 144.51, 144.47, 143.82, 143.76, 143.73, 143.68(3C), 143.29, 143.20(4C), 142.90(aryl C), 142.81, 142.62, 141.46, 140.46, 139.34, 139.18(aryl C), 137.99, 137.27(aryl C),

133.41(aryl C), 129.86(aryl C, 2C), 129.66(aryl C), 128.99(aryl C, 2C), 127.99(aryl C, 3C), 127.85(aryl C), 124.76(aryl C), 124.16(aryl C, 2C), 123.20(aryl C), 122.84(aryl C, 2C), 121.60(aryl C), 120.73(aryl C), 116.63(aryl C), 115.53(aryl C, 2C), 115.07(aryl C), 61.89(sp^3-C of C_{60}), 56.83(sp^3-C of C_{60}), 55.53(-OCH_3), 51.84(-OCH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{89}H_{24}O_3N_2$ 1168.1792; found 1168.1788.

Synthesis of 3af

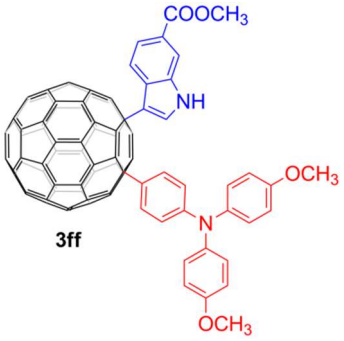


3af

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4,4′-dimethoxytriphenylamine (76 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (3.8 mg, 11%) and **3af** (48 mg, 79%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-DMSO) δ 11.47 (s, 1H), 8.38 (s, 1H), 7.85 (d, J = 7.2 Hz, 2H), 7.72 (s, 1H), 7.44 (d, J = 8.8 Hz, 1H), 7.28 (d, J = 8.8 Hz, 1H), 7.07 (d, J = 7.6 Hz, 4H), 6.97 (d, J = 7.2 Hz, 2H), 6.84 (d, J = 7.6 Hz, 4H), 3.80 (s, 6H). ^{13}C NMR (100 MHz, CS_2/d_6-DMSO) (all 1C unless indicated) δ 156.02(2C), 155.27(aryl C, 2C), 150.63, 150.55, 147.83, 147.81, 147.79, 147.65(2C), 146.35(2C), 146.29(2C), 146.23, 146.20, 146.06, 145.99(aryl C), 144.78, 144.75, 144.73, 144.32, 144.31, 144.10, 144.04(3C), 144.01, 143.68(3C), 143.59(2C), 143.56, 143.52, 143.45, 143.30(2C), 143.27, 143.20, 143.15, 142.53, 142.44, 142.40, 142.34(3C), 141.97, 141.90(3C), 141.86, 141.66, 141.48, 141.29, 140.11, 139.44(aryl C, 2C), 137.99, 137.80, 136.71, 136.66, 135.44(aryl C), 131.02(aryl C), 127.73(aryl C, 2C), 126.82(aryl C), 126.07(aryl C, 4C), 124.99(aryl C), 124.28(aryl C), 122.02(aryl C), 119.69(aryl C, 2C), 114.26(aryl C, 4C), 113.92(aryl C), 113.37(aryl C), 112.91(aryl C), 60.58(sp^3-C of C_{60}), 55.60(sp^3-C of C_{60}), 54.44(-OCH_3, 2C). UV-vis ($CHCl_3$) λmax 449, 541, 682

nm;HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{88}H_{23}BrO_2N_2$ 1220.0935; found 1220.0926.

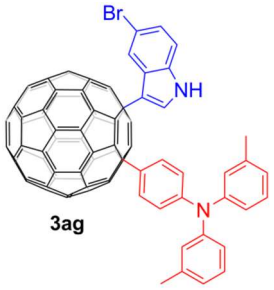
Synthesis of 3ff



3ff

C_{60} (36.0 mg, 0.05 mmol), methyl indole-6-carboxylate (10.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4,4′-dimethoxytriphenylamine (76 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane /ethyl acetate(12:5:1) as the eluent to give unreacted C_{60} (4.2 mg, 12%) and **3ff** (47.4 mg, 79%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.30 (s, 1H), 8.24 (d, J = 8.4 Hz, 1H), 8.09 (s, 1H), 7.68 - 7.45 (m, 4H), 6.94 (d, J = 8.8 Hz, 4H), 6.87 (d, J = 8.4 Hz, 2H), 6.70 (d, J = 8.4 Hz, 4H), 3.83 (s, 3H), 3.69 (s, 6H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 166.95(acyl C), 157.34, 157.16, 156.53(aryl C, 2C), 151.92, 151.64, 149.12(2C), 149.08, 149.00, 148.93, 147.61(2C), 147.56(2C), 147.51(2C), 147.35, 147.29, 146.04(3C), 145.62, 145.59, 145.33(2C), 145.27(3C), 144.94(3C), 144.90, 144.82(3C), 144.60(2C), 144.56, 144.51, 144.46(2C), 143.78, 143.70(2C), 143.65(3C), 143.26, 143.17(4C), 142.87(aryl C), 142.79, 142.58, 141.41, 140.74(aryl C, 2C), 139.32, 139.10, 137.97, 137.90, 137.19(aryl C), 132.20(aryl C), 129.61(aryl C), 128.84(aryl C, 2C), 127.70(aryl C), 127.16(aryl C, 4C), 124.75(aryl C), 121.59(aryl C), 121.16(aryl C, 2C), 120.74(aryl C), 116.71(aryl C), 115.34(aryl C, 4C), 115.00(aryl C), 61.84(sp^3-C of C_{60}), 56.73(sp^3-C of C_{60}), 55.47(-OCH_3, 2C), 51.82(-OCH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{90}H_{26}O_4N_2$ 1198.1887; found 1198.1893.

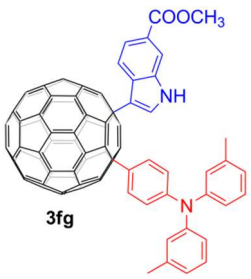
Synthesis of 3ag



3ag

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 3,3′-dimethyltriphenylamine (68 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (6.6 mg, 19%) and **3ag** (40.6 mg, 68%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.40 (s, 1H), 8.36 (s, 1H), 8.06 (dd, J = 8.4 , 2.8 Hz, 1H), 7.65 (d, J = 2.8 Hz, 1H), 7.35 (dd, J = 8.4 , 2.8 Hz, 1H), 7.26 - 7.21 (m, 3H), 7.12 (td, J = 7.6, 2.8 Hz, 1H), 7.05 (d, J = 7.6 Hz, 2H), 7.00 - 6.95 (m, 2H), 6.89 (s, 3H), 6.82 (d, J = 5.6 Hz, 1H), 3.03 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) δ 157.02, 156.19, 152.06, 150.76, 149.19, 149.03, 148.77, 148.54, 147.97(2C), 147.77(3C), 147.64(4C), 147.52(2C), 147.32(2C), 146.46, 146.15, 146.10, 146.01(aryl C), 145.74(2C), 145.32(3C), 145.10, 145.00(4C), 144.85, 144.75, 144.70, 144.59, 144.25(2C), 144.15, 143.95(3C), 143.75, 143.70(2C), 143.51, 143.42, 143.24(2C), 143.13, 143.04, 142.84, 142.64, 142.44, 141.31(aryl C), 139.40, 139.27(aryl C, 2C), 138.95(aryl C), 138.85(aryl C), 138.11, 136.42(aryl C), 132.99(aryl C), 131.52(aryl C), 129.89(aryl C, 2C), 128.23(aryl C), 127.40(aryl C), 126.16(aryl C), 125.93(aryl C), 125.73(aryl C), 125.14(aryl C, 2C), 124.84(aryl C), 123.64(aryl C), 123.49(aryl C), 122.68(aryl C), 121.32(aryl C), 115.32(aryl C), 114.49(aryl C), 113.96(aryl C), 62.42(sp^3-C of C_{60}), 56.64(sp^3-C of C_{60}), 24.74(-CH_3), 22.07(-CH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z [M]$^-$ calcd for $C_{88}H_{23}BrN_2$ 1188.1037; found 1186.1045.

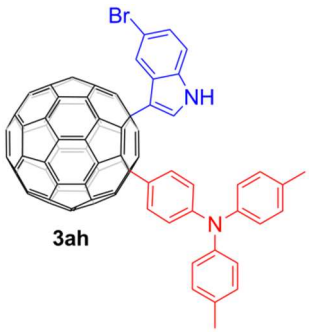
Synthesis of 3fg



3fg

C_{60} (36.0 mg, 0.05 mmol), methyl indole-6-carboxylate (10.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 3,3′-dimethyltriphenylamine (68 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (6.7 mg, 19%) and **3fg** (35.3 mg, 61%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.76 (s, 1H), 8.32 (d, J = 8.4 Hz, 1H), 8.16 (s, 1H), 8.11 (d, J = 8.4 Hz, 1H), 7.87 (d, J = 2,4 Hz, 1H), 7.76 (dd, J = 8.4, 1.2 Hz, 1H), 7.26 (t, J = 8.4 Hz, 2H), 7.15 (t, J = 8.0 Hz, 1H), 7.08 (d, J = 8.0 Hz, 2H), 7.04 - 6.98 (m, 2H), 6.92 (t, J = 8.4 Hz, 3H), 6.85 (d, J = 7.2 Hz, 1H), 3.88 (s, 3H), 3.04 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 166.74(acyl C), 156.91, 156.04, 151.86, 150.63, 148.90, 148.76, 148.56, 148.36, 147.82, 147.67, 147.53, 147.45(2C), 147.33(3C), 147.24(2C), 147.05, 146.99, 146.30, 145.90(aryl C), 145.79, 145.74, 145.44(2C), 145.08(3C), 144.87, 144.74, 144.71, 144.69, 144.66, 144.57, 144.47, 144.43, 144.32, 144.28, 143.97, 143.95, 143.90, 143.64(2C), 143.57, 143.46, 143.42, 143.35, 143.22, 143.13, 142.96(2C), 142.86, 142.69, 142.58, 142.38, 142.18(aryl C), 140.97(aryl C), 139.16(aryl C), 139.02(aryl C), 138.94, 138.61, 137.82, 136.87(aryl C), 132.47(aryl C), 131.26(aryl C), 129.65(aryl C, 2C), 129.61(aryl C), 129.45(aryl C), 127.64(aryl C), 126.85(aryl C), 125.90(aryl C), 124.94(aryl C, 2C), 124.62(aryl C), 124.33(aryl C), 123.46(aryl C), 122.45(aryl C), 121.19(aryl C), 120.94(aryl C), 120.26(aryl C), 115.68(aryl C), 114.67(aryl C), 62.18(sp^3-C of C_{60}), 56.45(sp^3-C of C_{60}), 51.55(-OCH_3), 24.51(-CH_3), 21.71(-CH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{90}H_{26}N_2O_2$ 1166.2001; found 1166.2011.

Synthesis of 3ah

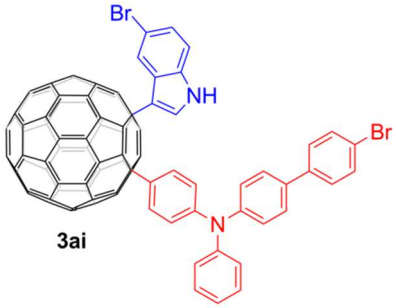


3ah

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30

minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4,4′-dimethyltriphenylamine (68 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 120 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (5.8 mg, 16%) and **3ah** (42 mg, 71%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.38 (s, 1H), 8.40 (s, 1H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.71 (d, *J* = 2.4 Hz, 1H), 7.36 (d, *J* = 8.4 Hz, 1H), 7.27 (d, *J* = 8.8 Hz, 1H), 7.04 (d, *J* = 2.8 Hz, 2H), 7.04-7.01 (m, 4H), 6.97 (d, *J* = 8.0 Hz, 4H), 2.34 (s, 6H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.00, 156.96, 151.71, 151.46, 149.03, 149.01, 148.91, 148.74, 148.23, 147.55, 147.50(2C), 147.42(3C), 147.26, 147.19, 145.99(aryl C), 145.93(2C), 145.54, 145.51, 145.22(2C), 145.17, 145.13(5C), 144.88, 144.84(2C), 144.77, 144.73(2C), 144.70. 144.60, 144.51, 144.46, 144.44. 144.39, 144.30, 143.72, 143.62(2C), 143.54(3C), 143.16, 143.08, 143.05(aryl C, 2C), 142.83, 142.64, 142.47, 141.36, 139.17, 138.99, 137.94, 137.89, 136.37(aryl C), 133.33(aryl C), 132.93(aryl C, 2C), 130.52(aryl C, 4C), 128.90(aryl C, 2C), 127.99(aryl C), 125.94(aryl C), 125.59(aryl C), 125.32(aryl C, 4C), 123.49(aryl C), 122.81(aryl C, 2C), 115.91(aryl C), 114.52(aryl C), 113.95(aryl C), 61.74(sp^3-C of C_{60}), 56.61(sp^3-C of C_{60}), 21.58(-CH_3, 2C). UV-vis ($CHCl_3$) λmax 449, 543, 685 nm;HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{88}H_{23}BrN_2$ 1188.1037; found 1188.1028.

Synthesis of 3ai

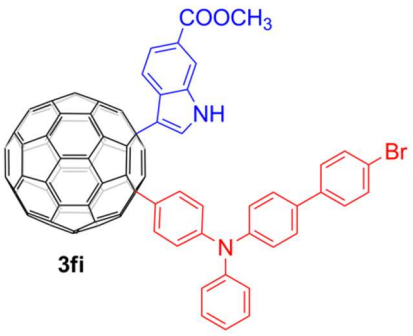


3ai

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4-bromo-4'-(diphenylamino)biphenyl (100 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the

eluent to give unreacted C_{60} (8 mg, 22%) and **3ai** (32.7 mg, 50%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.59 (s, 1H), 8.43 (s, 1H), 7.97 (d, J = 8.0 Hz, 2H), 7.79 (s, 1H), 7.53-7.45 (m, 6H), 7.40 (d, J = 8.4 Hz, 1H), 7.29 (t, J = 8.0 Hz, 3H), 7.20-7.12 (m, 6H), 7.07 (t, J = 7.2 Hz, 1H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.19, 156.97, 151.71, 151.64, 149.20, 149.16, 149.08, 148.89, 147.79, 147.72, 147.64(2C), 147.57, 147.54, 147.53, 147.48(2C), 147.41, 147.35, 146.14, 146.09, 145.92(aryl C), 145.69, 145.67, 145.44, 145.36(2C), 145.32, 145.28, 145.04(aryl C), 144.98(2C), 144.92, 144.90, 144.88, 144.83, 144.77, 144.66, 144.59(3C), 144.42, 143.87, 143.77(2C), 143.73, 143.70(2C), 143.32, 143.24, 143.19(3C), 142.99(aryl C), 142.78, 142.62, 141.53, 139.86, 139.30, 139.21, 138.18(aryl C), 137.97(aryl C), 136.63, 135.00(aryl C), 134.65(aryl C), 132.46(aryl C, 2C), 130.17(aryl C, 2C), 129.34(aryl C 2C), 128.76(aryl C, 2C), 128.35(aryl C, 2C), 128.14(aryl C), 125.99(aryl C), 125.91(aryl C), 125.51(aryl C, 2C), 124.84(aryl C, 2C), 124.57(aryl C, 2C), 124.30(aryl C), 123.53(aryl C), 121.93(aryl C), 115.88(aryl C), 114.49(aryl C), 114.28(aryl C), 61.90(sp^3-C of C_{60}), 56.83(sp^3-C of C_{60}). UV-vis ($CHCl_3$) λmax 447, 537, 683 nm; HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{92}H_{22}Br_2N_2$ 1314.0152; found 1312.0146.

Synthesis of 3fi

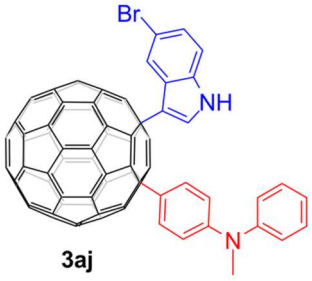


3fi

C_{60} (36.0 mg, 0.05 mmol), methyl indole-6-carboxylate (10.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4-bromo-4'-(diphenylamino)biphenyl (100 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (8 mg, 22%) and **3fi** (44.7 mg, 69%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.73 (s, 1H), 8.33 (d, J = 8.4Hz, 1H), 8.15 (s, 1H), 7.97 (d, J = 7.2 Hz, 2H), 7.91 (s, 1H), 7.76 (d, J = 8.4 Hz, 1H), 7.51 - 7.43 (m, 6H), 7.28 (t, J = 7.0 Hz, 2H), 7.18 - 7.13 (m, 6H), 7.05 (t, J = 6.8 Hz, 1H), 3.87 (s, 3H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ

166.97(acyl C), 157.21, 157.06, 151.68, 151.66, 149.17, 149.14, 149.08, 148.92, 147.82, 147.68, 147.64, 147.54(4C), 147.44(3C), 147.38, 147.33, 146.10, 146.08, 145.74(aryl C), 145.66, 145.63, 145.55, 145.33(2C), 145.29, 145.21, 144.99, 144.94(2C), 144.91(aryl C), 144.86(2C), 144.80, 144.67, 144.62, 144.57, 144.53(2C), 144.42, 143.81, 143.74(2C), 143.71, 143.69(2C), 143.30, 143.20(2C), 143.16(2C), 142.91(aryl C), 142.80, 142.60, 141.49, 139.82, 139.28, 139.24(aryl C), 138.05(aryl C), 137.93, 137.25(aryl C), 134.87(aryl C), 134.66(aryl C), 132.43(aryl C, 2C), 130.14(aryl C, 2C), 129.61(aryl C), 129.24(aryl C, 2C), 128.72(aryl C, 2C), 128.32(aryl C, 2C), 127.81(aryl C), 125.48(aryl C, 2C), 124.79(aryl C, 2C), 124.61(aryl C, 2C), 124.27(aryl C), 121.98(aryl C), 121.59(aryl C), 120.70(aryl C), 116.56(aryl C), 115.07(aryl C), 61.84(sp^3-C of C_{60}), 56.82(sp^3-C of C_{60}), 51.85(-OCH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{94}H_{25}BrN_2O_2$ 1294.1092; found 1294.1084.

Synthesis of 3aj

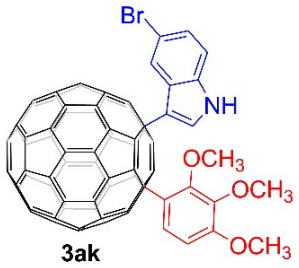


3aj

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and N-methyldiphenylamine (44 μL, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 120 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (4.9 mg, 14%) and **3aj** (23.3 mg, 43%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.20 (s, 1H), 8.38 (s, 1H), 7.87 (dd, J = 8.8, 3.2 Hz, 2H), 7.65 (t, J = 2.8 Hz, 1H), 7.34 (dd, J = 8.4, 2.8 Hz, 1H), 7.28 - 7.23 (m, 3H), 7.09-7.07 (m, 2H), 6.98 (dd, J = 8.4, 2.8 Hz, 3H), 3.38 (s, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.23, 156.98, 151.83, 151.48, 149.08(2C), 148.97(2C), 148.84(aryl C), 148.70, 147.60, 147.55(2C), 147.45(3C), 147.32, 147.25, 146.04(2C), 145.99(aryl C), 145.61, 145.55, 145.28(2C), 145.24, 145.17, 145.14, 144.94, 144.91(2C), 144.84(2C), 144.79(2C), 144.65, 144.58, 144.51(2C), 144.46, 144.40, 143.78, 143.68(2C), 143.60(3C), 143.23, 143.12(4C), 142.89, 142.72, 142.53, 141.43, 139.26, 139.04, 137.96, 137.90, 136.39(aryl C), 132.21(aryl C), 129.93(aryl C, 2C), 128.93(aryl C, 2C), 128.03(aryl C), 126.07(aryl

C), 125.56(aryl C), 123.63(aryl C), 123.33(aryl C), 122.92(aryl C, 2C), 119.39(aryl C, 2C), 116.09(aryl C), 114.64(aryl C), 113.86(aryl C), 61.76(sp^3-C of C_{60}), 56.62(sp^3-C of C_{60}), 40.60(-CH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{81}H_{17}BrN_2$ 1098.0567; found 1098.0575.

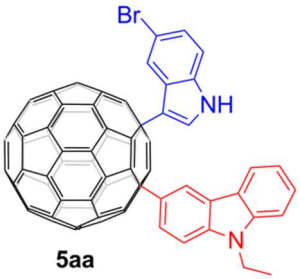
Synthesis of 3ak



3ak

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in 1,2-dichlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 1,2,3-trimethoxybenzene (42 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 150 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (2:1) as the eluent to give unreacted C_{60} (5.8 mg, 16%) and **3ak** (18.5 mg, 36%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-DMSO) δ 11.33 (s, 1H), 8.32 (s, 1H), 7.68 (d, J = 8.8 Hz, 1H), 7.61 (s, 1H), 7.38 (d, J = 8.4 Hz, 1H), 7.24 (d, J = 8.4 Hz, 1H), 6.74 (d, J = 8.4 Hz, 1H), 4.10 (s, 3H), 3.92 (s, 6H). ^{13}C NMR (100 MHz, CS_2/d_6-DMSO) (all 1C unless indicated) δ 155.88, 155.60, 153.54(aryl C), 152.33(aryl C), 150.59, 150.09, 147.85, 147.78, 147.70, 147.28, 146.84, 146.44, 146.22(4C), 145.99, 145.93, 145.21, 144.71, 144.63(2C), 144.48, 144.36, 144.29, 144.15, 144.08(2C), 143.83, 143.70(4C), 143.50(2C), 143.35, 143.27, 143.23, 143.05(2C), 142.68, 142.54, 142.45, 142.38(2C), 142.24, 142.09, 141.98, 141.93, 141.86, 141.79(2C), 141.61, 141.57, 141.36, 140.96, 139.96, 137.86(2C), 137.73, 136.74(aryl C), 135.26(aryl C), 126.80(aryl C), 125.64(aryl C), 124.80(aryl C), 124.10(aryl C), 123.19(aryl C), 121.89(aryl C), 113.56(aryl C), 113.16(aryl C), 112.69(aryl C), 106.71(aryl C), 59.90(sp^3-C of C_{60}), 59.43(sp^3-C of C_{60}), 58.85(-OCH_3), 55.40(-OCH_3), 55.03(-OCH_3). UV-vis ($CHCl_3$) λmax 447, 539, 683 nm; HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{77}H_{16}BrNO_3$ 1083.0305; found 1083.0311.

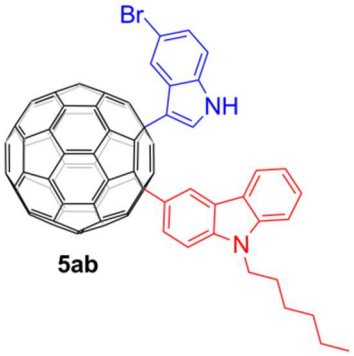
Synthesis of 5aa



5aa

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and N-ethylcarbazole (49 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 120 °C for 120 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (8.6 mg, 24%) and **5aa** (31.2 mg, 56%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.51 (s, 1H), 8.64 (d, J = 1.6 Hz, 1H), 8.48 (s, 1H), 8.18 (dd, J = 8.4, 2.0 Hz, 1H), 7.75 (d, J = 2.8 Hz, 1H), 7.69 (d, J = 8.0 Hz, 1H), 7.50 (d, J = 8.4 Hz, 1H), 7.44 - 7.39 (m, 3H), 7.31 (dd, J = 8.8, 1.6 Hz, 1H), 7.15 - 7.11 (m, 1H), 4.44 (q, J = 7.2 Hz, 2H), 1.49 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.34, 157.16, 152.48, 151.54, 149.19, 149.13(2C), 148.89, 147.90, 147.67, 147.62, 147.59, 147.52(2C), 147.36, 147.29, 146.71, 146.09, 146.02, 145.68(2C), 145.40(3C), 145.30, 145.14, 145.00(4C), 144.91, 144.85, 144.83, 144.73, 144.68, 144.60, 144.57, 144.52, 144.49, 143.85, 143.77, 143.72, 143.65(3C), 143.28, 143.21(3C), 143.14, 142.98, 142.79, 142.61, 141.49, 140.92(aryl C), 140.07, 139.43, 139.12, 138.37, 138.13(aryl C), 136.63(aryl C), 131.78(aryl C), 128.19(aryl C), 126.71(aryl C), 126.11(aryl C), 126.01(aryl C), 125.81(aryl C), 124.27(aryl C), 123.69(aryl C), 123.42(aryl C), 121.32(aryl C), 120.69(aryl C), 119.90(aryl C), 116.14(aryl C), 114.60(aryl C), 114.16(aryl C), 109.74(aryl C), 109.20(aryl C), 62.59(sp^3-C of C_{60}), 56.83(sp^3-C of C_{60}), 38.33(-CH_2), 14.65(-CH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z [M]$^-$ calcd for $C_{82}H_{17}BrN_2$ 1110.0557; found 1110.0554.

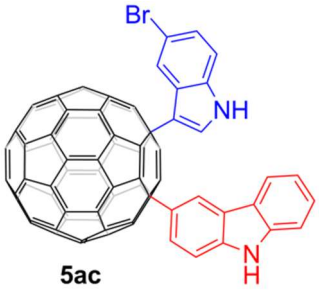
Synthesis of 5ab



5ab

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and N-hexylcarbazole (64 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 120 °C for 60 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (11 mg, 31%) and **5ab** (33.4 mg, 57%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) 10.53 (s, 1H), 8.74 (s, 1H), 8.61 (s, 1H), 8.30 (d, J = 8.4 Hz, 1H), 7.86 (d, J = 2.4 Hz, 1H), 7.78 (d, J = 7.6 Hz, 1H), 7.61 (d, J = 8.4 Hz, 1H), 7.56 - 7.59 (m, 3H), 7.44 (d, J = 8.4 Hz, 1H), 7.25 (t, J = 7.2 Hz, 1H), 4.49 (t, J = 7.2 Hz, 2H), 1.57 - 1.42 (m, 8H), 1.06(t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.22, 157.09, 152.42, 151.44, 149.14, 149.11(2C), 148.81, 147.85, 147.65, 147.56(2C), 147.49(2C), 147.33, 147.26, 146.64, 146.07, 145.99, 145.66(2C), 145.35(3C), 145.27, 145.06, 144.95(4C), 144.88, 144.82, 144.79, 144.70, 144.66, 144.56(2C), 144.46(2C), 143.82, 143.73, 143.69, 143.62(3C), 143.25, 143.18(3C), 143.11, 142.96, 142.75, 142.57, 141.47, 141.29(aryl C), 140.44, 139.37, 139.08, 138.34, 138.08(aryl C), 136.51(aryl C), 131.72(aryl C), 128.11(aryl C), 126.61(aryl C), 126.05(aryl C), 125.91(aryl C), 125.73(aryl C), 124.07(aryl C), 123.70(aryl C), 123.24(aryl C), 121.20(aryl C), 120.62(aryl C), 119.83(aryl C), 116.18(aryl C), 114.66(aryl C), 113.99(aryl C), 109.80(aryl C), 109.27(aryl C), 62.51(sp^3-C of C_{60}), 56.74(sp^3-C of C_{60}), 43.72(-CH_2), 32.56(-CH_2), 29.92(-CH_2), 27.87(-CH_2), 23.68(-CH_2), 14.97(-CH_3). UV-vis ($CHCl_3$) λmax 448, 540, 682 nm;HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z [M]$^-$ calcd for $C_{86}H_{25}BrN_2$ 1166.1193; found 1166.1184.

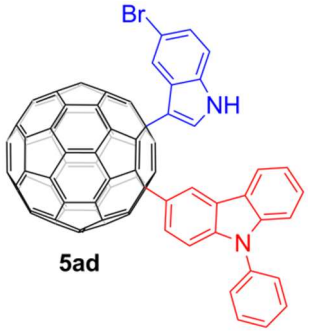
Synthesis of 5ac



5ac

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and carbazole (42 mg, 0.25 mmol) were added to the mixture and stirred at in an oil bath 110 °C for 120 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (3.1 mg, 9%) and **5ac** (30.1 mg, 56%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.45 (s, 1H), 9.46 (d, *J* = 9.2 Hz, 1H), 8.49 (s, 1H), 8.27 (dd, J = 7.2, 1.6 Hz, 1H), 8.12 (d, *J* = 7.6 Hz, 1H), 8.04 (d, *J* = 7.6 Hz, 1H), 7.64-7.62 (m, 1H), 7.37 - 7.27 (m, 4H), 7.18 (t, *J* = 7.6 Hz, 1H), 7.02-6.97 (m, 1H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.17, 156.77, 152.00, 151.20, 149.37, 149.27, 149.11, 148.92, 147.84, 147.81, 147.70(3C), 147.53, 147.49(2C), 146.43, 146.29, 146.26, 145.85(2C), 145.46, 145.42, 145.39(aryl C), 145.21(2C), 145.14(2C), 145.06(4C), 144.93(2C), 144.80, 144.73, 144.65(2C), 144.42, 144.05, 143.92(3C), 143.83(2C), 143.49, 143.38, 143.32, 143.21(2C), 143.09, 142.94, 142.76, 141.67, 140.28, 139.46, 139.10, 138.22, 138.03, 137.91(aryl C), 136.74(aryl C), 128.09(aryl C), 126.94(aryl C), 126.86(aryl C), 126.31(aryl C), 126.24(aryl C), 126.01(aryl C), 123.37(aryl C), 123.27(aryl C), 123.16(aryl C), 121.13(aryl C), 120.80(aryl C), 120.62(aryl C), 120.52(aryl C), 115.05(aryl C), 114.52(aryl C, 2C), 111.98(aryl C), 61.10(sp^3-C of C_{60}), 57.12(sp^3-C of C_{60}). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{80}H_{13}BrN_2$ 1082.0254; found 1082.0246.

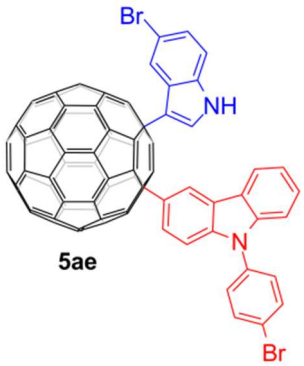
Synthesis of 5ad



5ad

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and N-phenylcarbazole (61 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 120 °C for 120 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (6 mg, 17%) and **5ad** (21.8 mg, 38%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.57 (s, 1H), 8.72 (s, 1H), 8.48 (s, 1H), 8.16 (dd, J = 8.8, 1.6 Hz, 1H), 7.80 - 7.75 (m, 2H), 7.68 - 7.59 (m, 4H), 7.52 - 7.39 (m, 5H), 7.33 - 7.30 (m, 1H), 7.23-7.19 (m, 1H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.26(2C), 152.41, 151.63, 149.23(4C), 148.95, 147.95, 147.75, 147.68(2C), 147.62(2C), 147.44, 147.38, 146.62, 146.17, 146.12(aryl C), 145.78(2C), 145.46(3C), 145.32, 145.06(4C), 144.99, 144.94, 144.89, 144.82, 144.75, 144.67(2C), 144.58(2C), 143.93, 143.85, 143.79, 143.74(3C), 143.36, 143.28(3C), 143.06, 142.86, 142.69, 141.81, 141.58, 140.91, 139.47, 139.23, 138.48, 138.16, 138.03(aryl C), 136.70(aryl C), 133.00(aryl C), 130.66(aryl C, 3C), 128.22(aryl C), 127.42(aryl C, 3C), 127.10(aryl C), 126.20(aryl C, 2C), 126.07(aryl C), 124.82(aryl C), 123.85(aryl C), 123.70(aryl C), 121.30(aryl C), 121.08(aryl C), 120.69(aryl C), 116.14(aryl C), 114.62(aryl C), 114.24(aryl C), 110.98(aryl C), 110.40(aryl C), 62.60(sp^3-C of C_{60}), 56.92(sp^3-C of C_{60}). UV-vis ($CHCl_3$) λmax 448, 542, 683 nm; HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z [M]$^-$ calcd for $C_{86}H_{17}BrN_2$ 1158.0567; found 1156.0578.

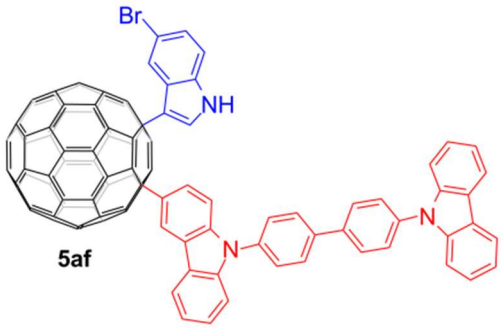
Synthesis of 5ae



5ae

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 9-(4-bromophenyl)carbazole (81 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 120 °C for 120 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (5.4 mg, 15%) and **5ae** (19.5 mg, 32%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.72 (s, 1H), 8.77 (s, 1H), 8.50 (s, 1H), 8.20 (d, J = 8.4 Hz, 1H), 7.88 (d, J = 2.0 Hz, 1H), 7.82 - 7.80 (m, 3H), 7.58 (d, J = 8.4 Hz, 2H), 7.50 (t, J = 8.8 Hz, 2H), 7.42 (s, 2H), 7.33 (d, J = 8.8 Hz, 1H), 7.25 (t, J = 5.6 Hz, 1H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.44, 157.28, 152.46, 151.73, 149.37, 149.31(2C), 149.03, 148.03, 147.83, 147.73(2C), 147.70(2C), 147.52, 147.46, 146.69, 146.24, 146.19, 145.85, 145.81, 145.55(2C), 145.46(2C), 145.13(4C), 145.06, 145.02, 144.96(aryl C), 144.91, 144.81, 144.74(2C), 144.65(2C), 144.00, 143.92, 143.82(4C), 143.43, 143.35(4C), 143.14, 142.92, 142.76, 141.63, 140.74, 139.54, 139.32, 138.57, 138.16, 137.20, 136.84(aryl C), 133.94(aryl C, 3C), 133.40(aryl C), 129.22(aryl C, 3C), 128.28(aryl C), 127.35(aryl C), 126.42(aryl C), 126.36(aryl C), 126.07(aryl C), 125.06(aryl C), 124.03(aryl C), 123.67(aryl C), 121.87(aryl C), 121.43(aryl C, 2C), 120.84(aryl C), 116.12(aryl C), 114.55(aryl C), 114.47(aryl C), 110.99(aryl C), 110.37(aryl C), 62.66(sp^3-C of C_{60}), 57.03(sp^3-C of C_{60}). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z [M]$^-$ calcd for $C_{86}H_{16}Br_2N_2$ 1235.9680; found 1235.9674.

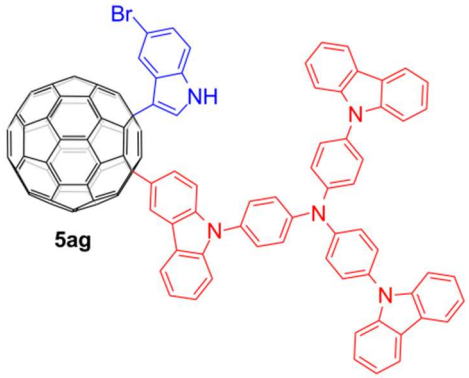
Synthesis of 5af



5af

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and 4,4′bis(N-carbazolyl)-1,1′-biphenyl (121 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 130 °C for 120 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (5 mg, 14%) and **5af** (35 mg, 50%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.69 (s, 1H), 8.78 (s, 1H), 8.52 (s, 1H), 8.23 (dd, *J* = 8.4, 1.6 Hz, 1H), 8.11 (d, *J* = 8.0 Hz, 2H), 8.04 - 8.01 (m, 3H), 7.88 (d, *J* = 2.4 Hz, 1H), 7.83 - 7.74 (m, 5H), 7.62 (d, *J* = 8.4 Hz, 1H), 7.53 - 7.46 (m, 5H), 7.42 (t, *J* = 8.0 Hz, 3H), 7.34 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.29 - 7.24 (m, 3H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.41, 157.33, 152.49, 151.72, 149.36, 149.30, 149.28, 149.04, 148.03, 147.83, 147.74, 147.70(2C), 147.51, 147.45, 146.71, 146.24, 146.18, 145.85, 145.81, 145.55(3C), 145.45, 145.41, 145.13(3C), 145.06, 145.01, 144.96, 144.90, 144.82, 144.73(2C), 144.65, 143.99, 143.92, 143.86, 143.81(3C), 143.43, 143.35(3C), 143.29, 143.13, 142.93, 142.75, 141.80, 141.64, 141.19(aryl C, 2C), 140.90(aryl C), 139.97 139.59, 139.55, 139.30, 138.55, 138.17, 137.91(aryl C), 137.56(aryl C), 136.80(aryl C), 133.21(aryl C), 129.33(aryl C, 2C), 129.24(aryl C, 2C), 128.27(aryl C), 127.98(aryl C, 3C), 127.90(aryl C, 2C), 127.26(aryl C), 126.80(aryl C, 3C), 126.32(aryl C, 2C), 126.07(aryl C), 125.01(aryl C), 124.18(aryl C, 2C), 124.01(aryl C), 123.68(aryl C), 121.43(aryl C), 121.28(aryl C), 121.08(aryl C, 2C), 121.00(aryl C, 3C), 120.81(aryl C), 116.13(aryl C), 114.51(aryl C), 114.40(aryl C), 111.17(aryl C), 110.56(aryl C), 110.38(aryl C, 2C), 62.69(sp^3-C of C_{60}), 57.02(sp^3-C of C_{60}). UV-vis ($CHCl_3$) λmax 448, 542, 683 nm;HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z [M]$^-$ calcd for $C_{104}H_{28}BrN_3$ 1399.1460; found 1399.1451.

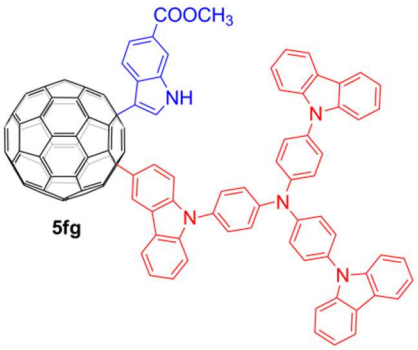
Synthesis of 5ag



5ag

C_{60} (36.0 mg, 0.05 mmol), 5-bromoindole (12 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and tris(4-carbazoyl-9-ylphenyl)amine (186 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 120 °C for 120 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (10:1) as the eluent to give unreacted C_{60} (5.5 mg, 15%) and **5ag** (36.3 mg, 44%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.38 (s, 1H), 8.70 (s, 1H), 8.47 (s, 1H), 8.19 - 8.16 (m, 1H), 8.08 – 8.05 (m, 4H), 7.74 (d, J = 2.8 Hz, 2H), 7.61 - 7.56 (m, 13H), 7.47-7.44 (m, 5H), 7.41 - 7.37 (m, 5H), 7.31 - 7.29 (m, 2H), 7.23 (td, J = 7.2, 2.8 Hz, 5H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 157.05(2C), 152.22, 151.42, 149.07(3C), 148.76, 147.77, 147.59, 147.45(4C), 147.28, 147.22, 146.78, 146.42(4C), 146.00(2C), 145.63(2C), 145.29(4C), 144.87(5C), 144.63(2C), 144.49(4C), 143.75(aryl C), 143.66(2C), 143.58(2C), 143.18(aryl C), 143.10(3C), 142.91, 142.71, 142.52, 141.74, 141.44, 141.14(aryl C, 4C), 140.83, 139.31, 139.09, 138.34, 137.98, 136.54, 133.48(aryl C), 133.01(aryl C), 132.91(aryl C), 128.55(aryl C, 5C), 128.42(aryl C, 2C), 128.11(aryl C), 127.04(aryl C), 126.62(aryl C, 5C), 126.13(aryl C), 126.08(aryl C), 125.95(aryl C, 5C), 125.66(aryl C, 2C), 124.76(aryl C), 123.95(aryl C, 6C), 123.80(aryl C), 123.71(aryl C), 121.31(aryl C), 120.99(aryl C, 5C), 120.84(aryl C, 5C), 116.19(aryl C), 114.68(aryl C), 114.06(aryl C), 110.97(aryl C), 110.37(aryl C), 110.21(aryl C, 5C), 62.44(sp^3-C of C_{60}), 56.74(sp^3-C of C_{60}). UV-vis ($CHCl_3$) λmax 448, 539, 682 nm; HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{122}H_{40}BrN_5$ 1655.2461; found 1653.2453.

Synthesis of 5fg



C_{60} (36.0 mg, 0.05 mmol), methyl indole-6-carboxylate (10.2 mg, 0.06 mmol), KO^{t}Bu (11.2 mg, 0.1 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then DMSO (2 mL) was added to the mixture and the color of the solution changed gradually from purple to dark green. After being stirred for 30 minutes, CF_3SO_3H (90 μL, 1 mmol) was added to the mixture and the color changed from dark green to brown. And after being stirred for 10 minutes, DDQ (56 mg, 0.25 mmol) and tris(4-carbazoyl-9-ylphenyl)amine (186 mg, 0.25 mmol) were added to the mixture and stirred in an oil bath at 120 °C for 120 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (1:1) as the eluent to give unreacted C_{60} (5.8 mg, 16%) and **5fg** (27.5 mg, 35%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.67 (s, 1H), 8.68 (s, 1H), 8.44 (d, J = 8.4 Hz, 1H), 8.20 (s, 1H), 8.16 (d, J = 8.4 Hz, 1H), 8.05 (d, J = 7.6 Hz, 4H), 7.91 (s, 1H), 7.80 (d, J = 8.4 Hz, 1H), 7.65 - 7.55 (m, 14H), 7.45 (d, J = 8.4 Hz, 5H), 7.40-7.36 (m, 5H), 7.25 - 7.17 (m, 5H), 3,86 (s, 3H).^{13}C NMR (100 MHz, CS_2/d_6-acetone) (all 1C unless indicated) δ 166.98(acyl C), 157.23, 157.03, 152.33, 151.49, 149.26, 149.16, 148.82, 147.90, 147.69, 147.61(2C), 147.54, 147.36(3C), 146.83, 146.52, 146.47(2C), 146.10(2C), 145.73, 145.67, 145.37(4C), 145.22, 144.97(5C), 144.87, 144.81, 144.71(2C), 144.48(3C), 143.67(5C), 143.30(aryl C), 143.19(3C), 142.95(aryl C), 142.79, 142.60, 141.76, 141.52, 141.17(aryl C, 4C), 140.83, 139.41, 139.21, 138.47, 138.07, 137.30, 133.52(aryl C), 132.99(aryl C), 132.91(aryl C), 129.67(aryl C), 128.60(aryl C, 5C), 128.47(aryl C, 2C), 127.91(aryl C), 127.09(aryl C), 126.63(aryl C, 5C), 125.99(aryl C, 5C), 125.66(aryl C, 2C), 124.89(aryl C), 124.77(aryl C), 123.96(aryl C, 5C), 123.73(aryl C), 121.77(aryl C), 121.29(aryl C), 121.11(aryl C), 120.99(aryl C, 5C), 120.85(aryl C, 5C), 120.72(aryl C), 116.78(aryl C), 115.10(aryl C), 110.96(aryl C), 110.38(aryl C), 110.22(aryl C, 5C), 62.51(sp^3-C of C_{60}), 56.85(sp^3-C of C_{60}), 51.86(-OCH_3). UV-vis ($CHCl_3$) λmax 448, 540, 682 nm; HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{124}H_{43}O_2N_5$ 1634.3455; found 1634.3463.

2. Experimental Procedure, Spectral Data, MALDI-TOF-MS spectrum and expanded ^{13}C NMR (100 MHz) of 6

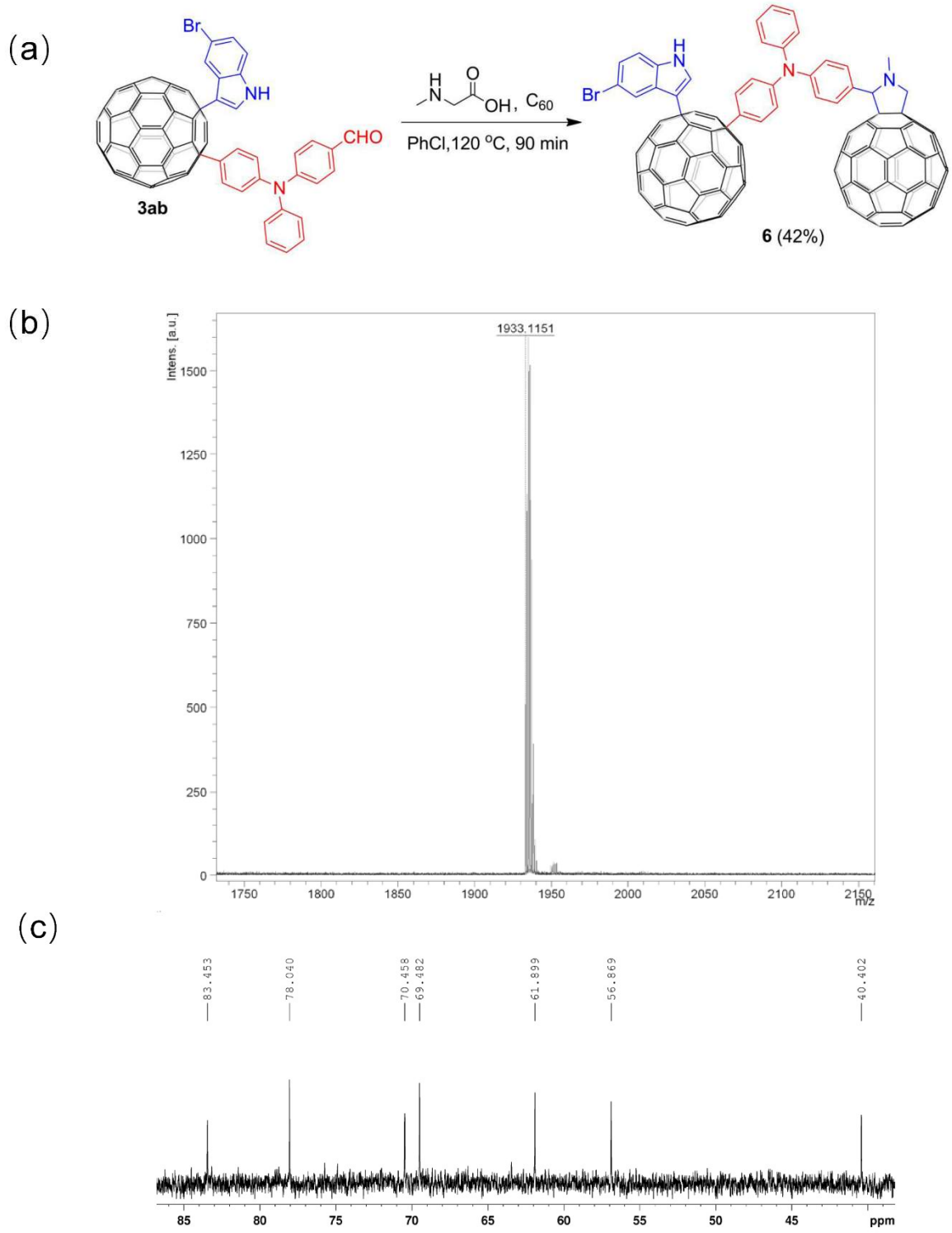
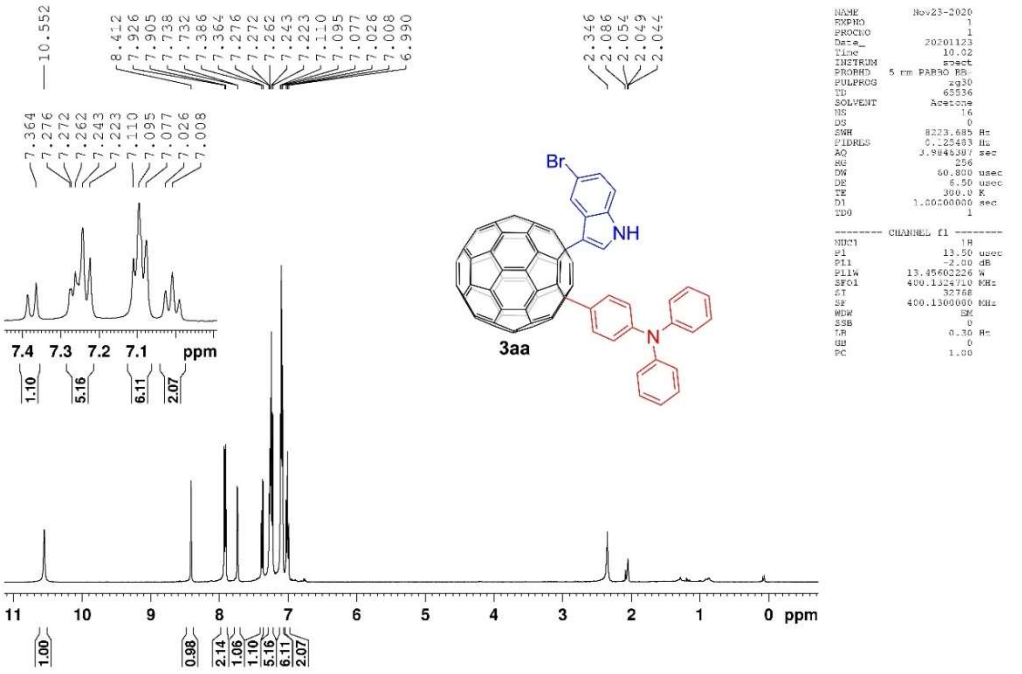


Figure S1 (a) Synthesis of dimer **6**. (b) The MALDI-TOF-MS spectrum of **6**: m/z $[\mathbf{6}]^-$ calcd for $C_{149}H_{24}BrN_3$ 1933.1154; found 1933.1151. (c) Expanded ^{13}C NMR (100 MHz) of **6**.

3ab (12 mg, 0.01 mmol), sarcosine (3 mg, 0.03 mmol), C_{60} (22 mg, 0.03 mmol) were dissolved in chlorobenzene (8 mL) at room temperature under Ar atmosphere. Then the mixture were stirred in an oil bath at 120 °C for 90 minutes. The mixture was cooled to room temperature and then added 20 mL carbon disulfide. Resulting solution was washed with water and then evaporated in vacuo. The residue was separated on a silica gel column with carbon disulfide/dichloromethane (5:1) as the eluent to give **6** (8 mg, 42%) as black amorphous solid. ^{1}H NMR (400 MHz, CS_2/d_6-acetone) δ 10.54 (s, 1H), 8.41 (s, 1H), 7.92 (d, J = 8.4 Hz, 2H), 7.74 (d, J = 2.4 Hz, 3H), 7.38 (d, J = 8.8 Hz, 1H), 7.28 - 7.22 (m, 3H), 7.17 (d, J = 8.4 Hz, 2H), 7.08 - 7.00 (m, 5H), 5.01 (d, J = 9.4 Hz, 1H), 4.98 (s, 1H), 4.33 (d, J = 9.2 Hz, 1H), 3.27 (s, 3H). ^{13}C NMR (100 MHz, CS_2/d_6-acetone) δ 157.23, 157.14, 156.99, 154.54(aryl C), 154.15, 153.99, 151.77, 151.67, 149.26, 149.23, 149.09, 149.00, 148.08(2C), 147.87(2C), 147.80(2C), 147.76, 147.71, 147.64(4C), 147.52, 147.48, 147.42, 147.35, 147.07, 146.89, 146.81(2C), 146.74(2C), 146.62(2C), 146.46(2C), 146.29, 146.19(aryl C), 146.15(2C), 146.05(2C), 145.88(2C), 145.82(2C), 145.76, 145.73(2C), 145.66(2C), 145.51, 145.43(2C), 145.39, 145.32, 145.26, 145.19, 145.10, 145.05(2C), 144.94(4C), 144.90(2C), 144.82, 144.70, 144.65(2C), 144.49, 143.93, 143.79(3C), 143.76(2C), 143.71, 143.56, 143.38, 143.25(4C), 143.17, 143.13(2C), 143.04(aryl C), 142.85, 142.82(2C), 142.72, 142.69(2C), 142.65(2C), 142.58, 142.54(2C), 142.30, 142.22, 142.17(aryl C), 141.58, 140.75, 140.69, 140.50, 139.75, 139.35, 139.26(2C), 138.12, 138.03, 137.35, 137.12, 136.64, 136.52(2C), 136.37(aryl C), 134.92(aryl C), 132.27(aryl C), 130.13(aryl C, 3C), 129.24(aryl C, 2C), 128.16(aryl C), 126.03(aryl C, 2C), 125.92(aryl C), 125.15(aryl C, 3C), 124.58(aryl C, 2C), 124.12(aryl C), 123.59(aryl C), 115.88(aryl C), 114.55(aryl C), 114.25(aryl C), 83.45, 78.04, 70.46, 69.48, 61.90(sp^3-C of C_{60}), 56.87(sp^3-C of C_{60}), 40.40(-CH_3). HRMS (MALDI-TOF-MS, DCTB as matrix, negative mode): m/z $[M]^-$ calcd for $C_{149}H_{24}BrN_3$ 1933.1154; found 1933.1151.

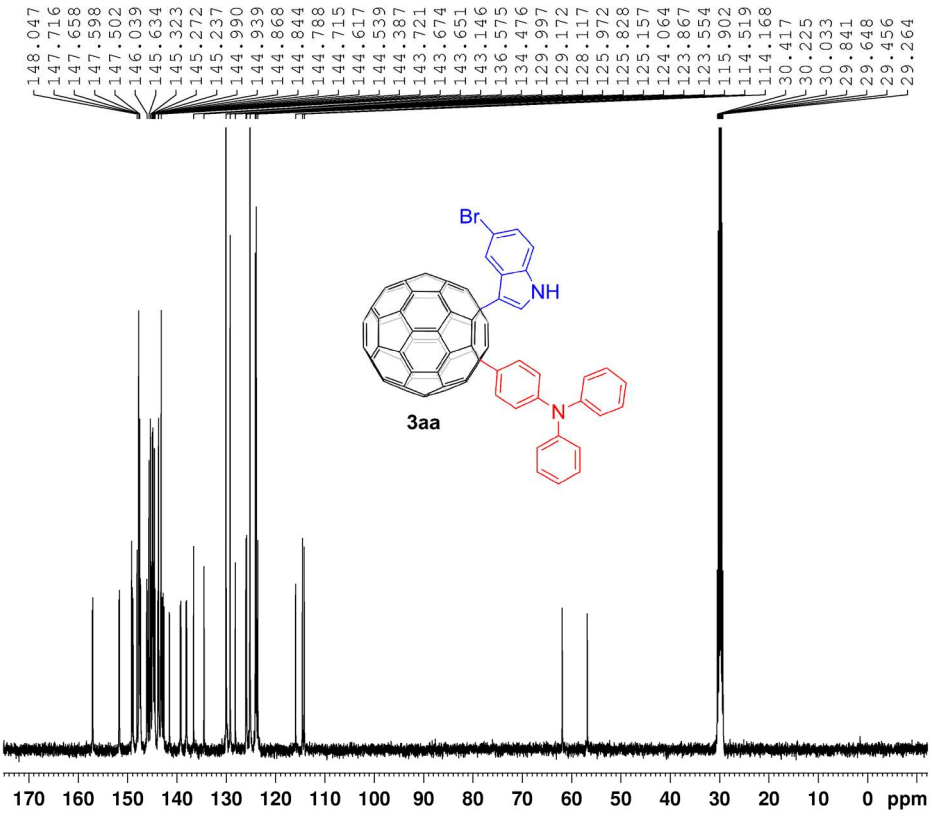
3. NMR Spectra of 3aa-3ak, 5aa-5fg and 6

^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3aa



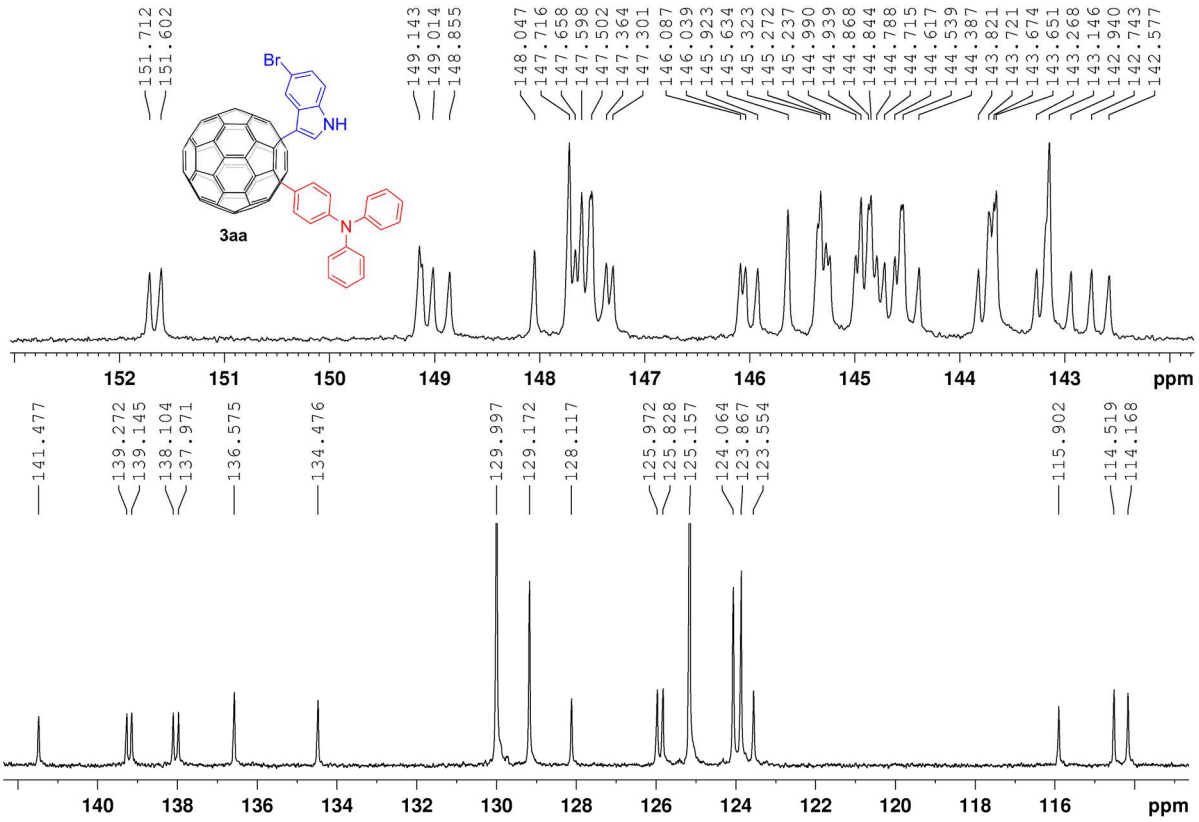
NAME	Nov23-2020
EXPNO	1
PROCNO	1
Date_	20201123
Time	10.02
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	Acetone
NS	16
DS	0
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9845397 sec
RG	256
DW	60.800 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.50 usec
PL1	-2.00 dB
PL1W	13.45602226 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3aa

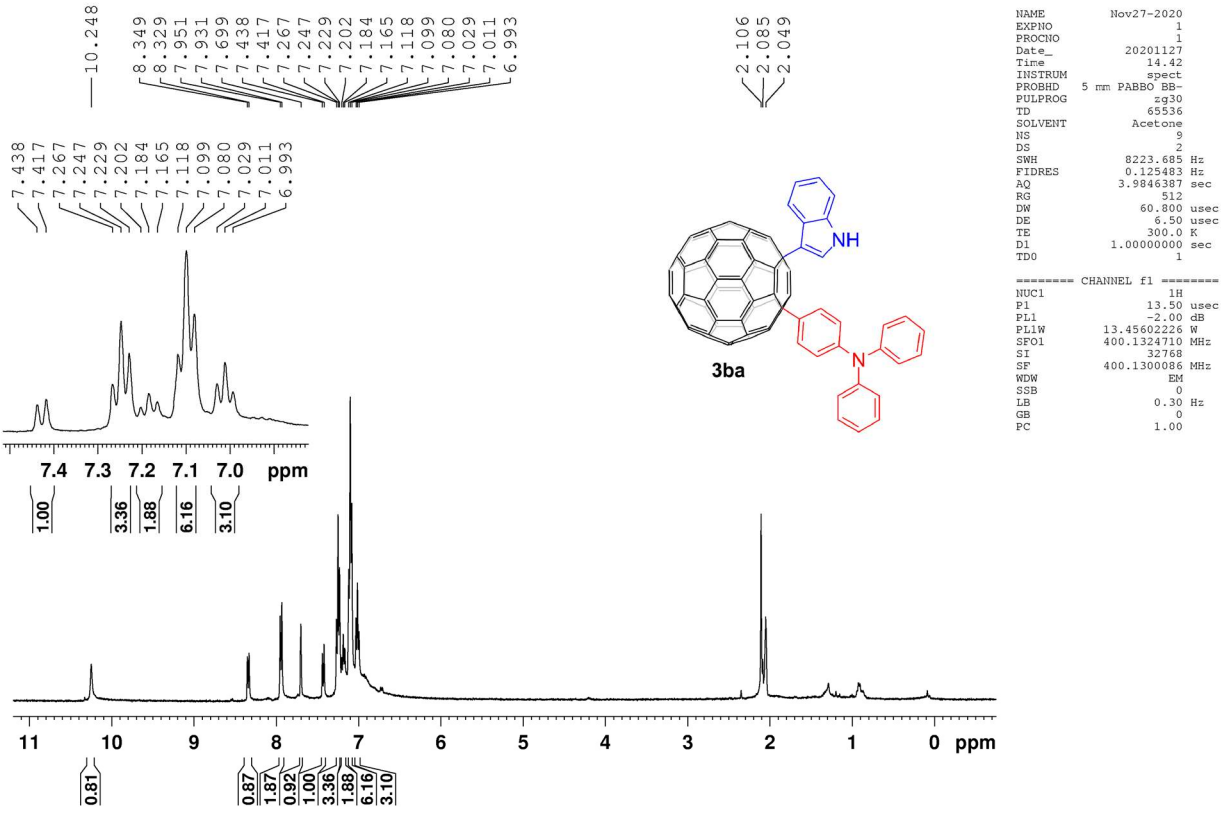


NAME	Nov23-2020
EXPNO	2
PROCNO	1
Date_	20201123
Time	17.42
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	Acetone
NS	16672
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	181
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.30 usec
PL1	-2.00 dB
PL1W	54.14257431 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	13.46 dB
PL13	120.00 dB
PL2W	13.45602226 W
PL12W	0.38275132 W
PL13W	0.00000000 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127308 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3aa



^1H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ba

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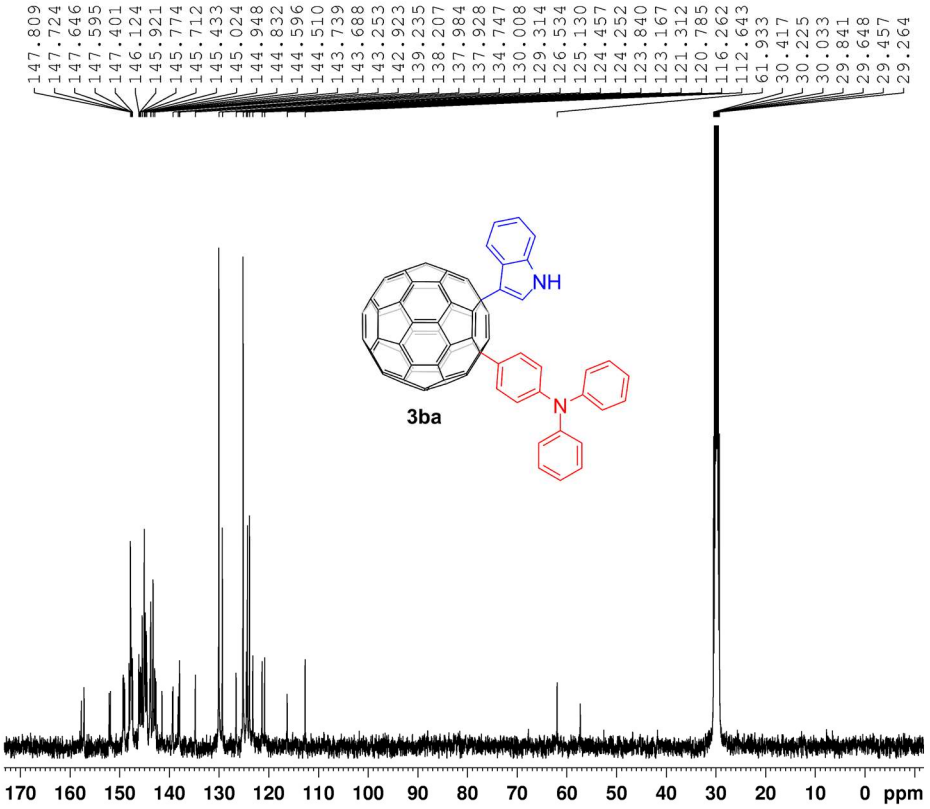
NAME                Nov27-2020
EXPNO                        1
PROCNO                       1
Date_                 20201127
Time                     14.42
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT               Acetone
NS                           9
DS                           2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                         512
DW                    60.800 usec
DE                     6.50 usec
TE                     300.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                    13.50 usec
PL1                   -2.00 dB
PL1W            13.45602226 W
SFO1            400.1324710 MHz
SI                       32768
SF              400.1300086 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                      1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ba

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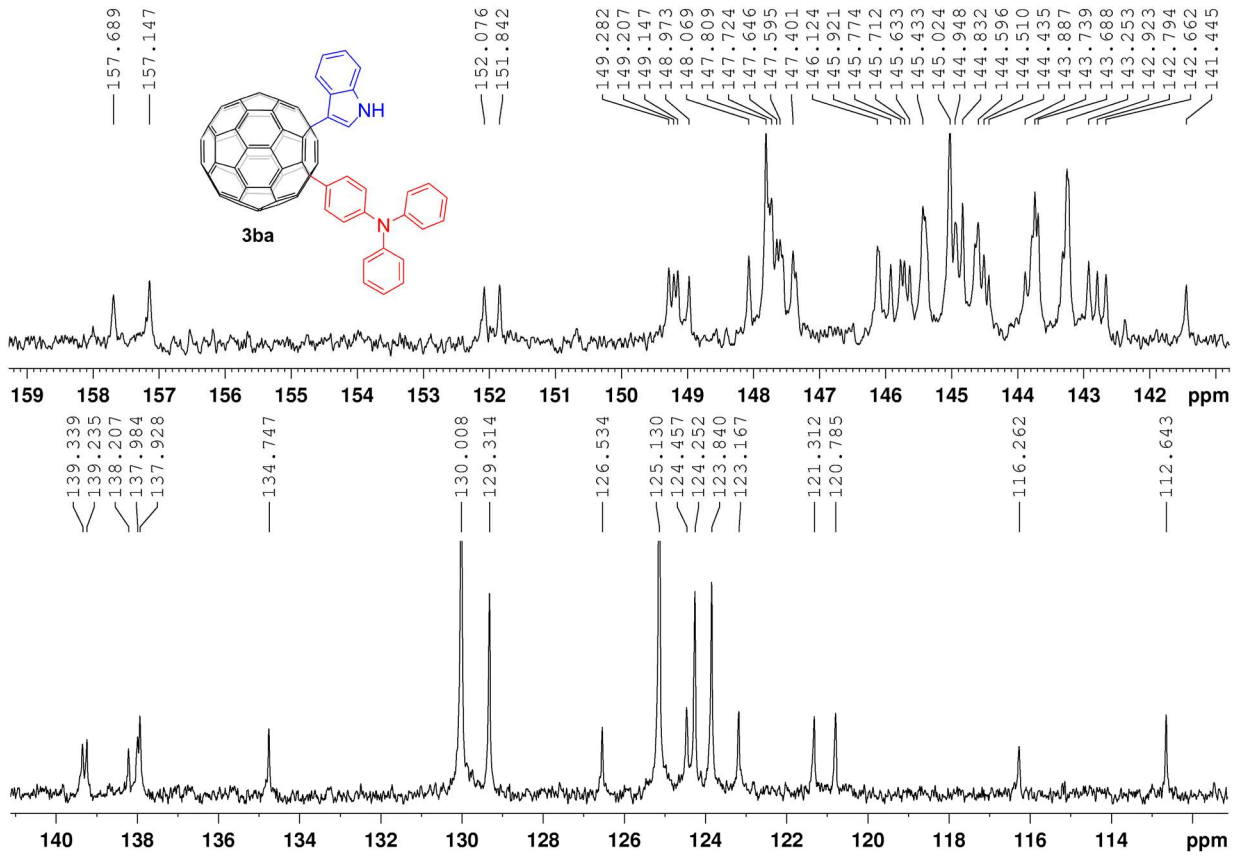
NAME          Nov27-2020
EXPNO                  2
PROCNO                 1
Date_           20201127
Time               17.25
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                 20480
DS                     4
SWH            24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  80.6
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

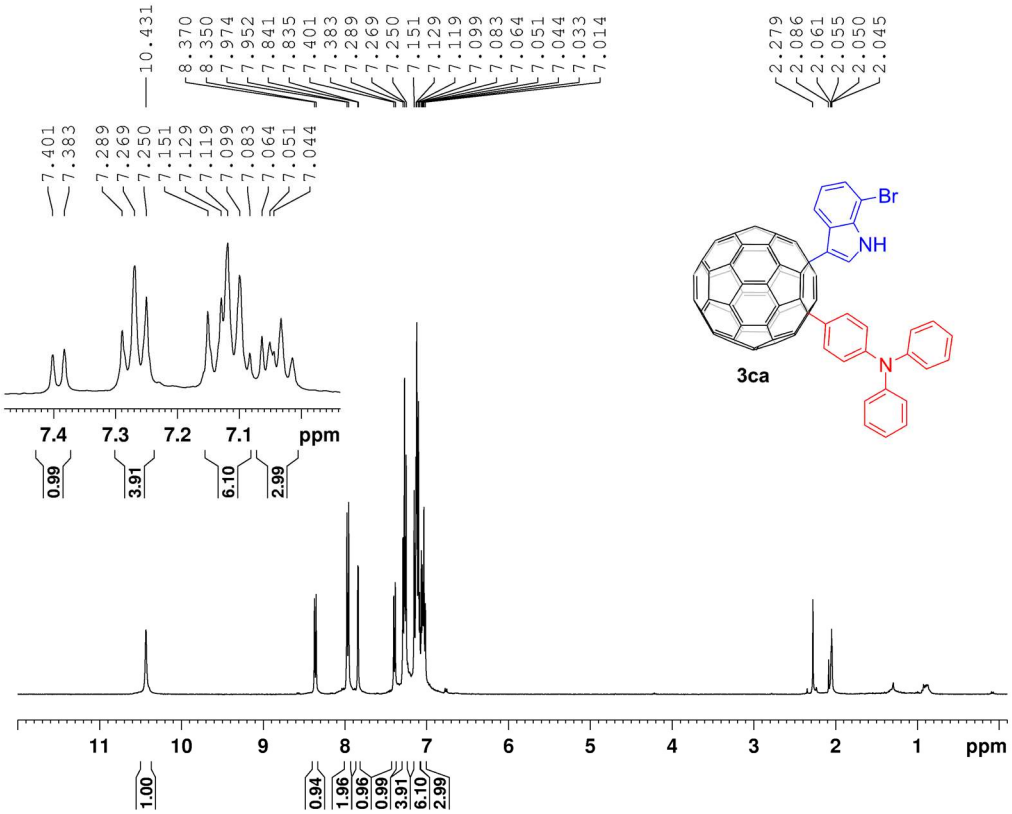
======== CHANNEL f1 ========
NUC1                 13C
P1                 9.30 usec
PL1                -2.00 dB
PL1W         54.14257431 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                -2.00 dB
PL12               13.46 dB
PL13              120.00 dB
PL2W         13.45602226 W
PL12W         0.38275132 W
PL13W         0.00000000 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127271 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ba



^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ca

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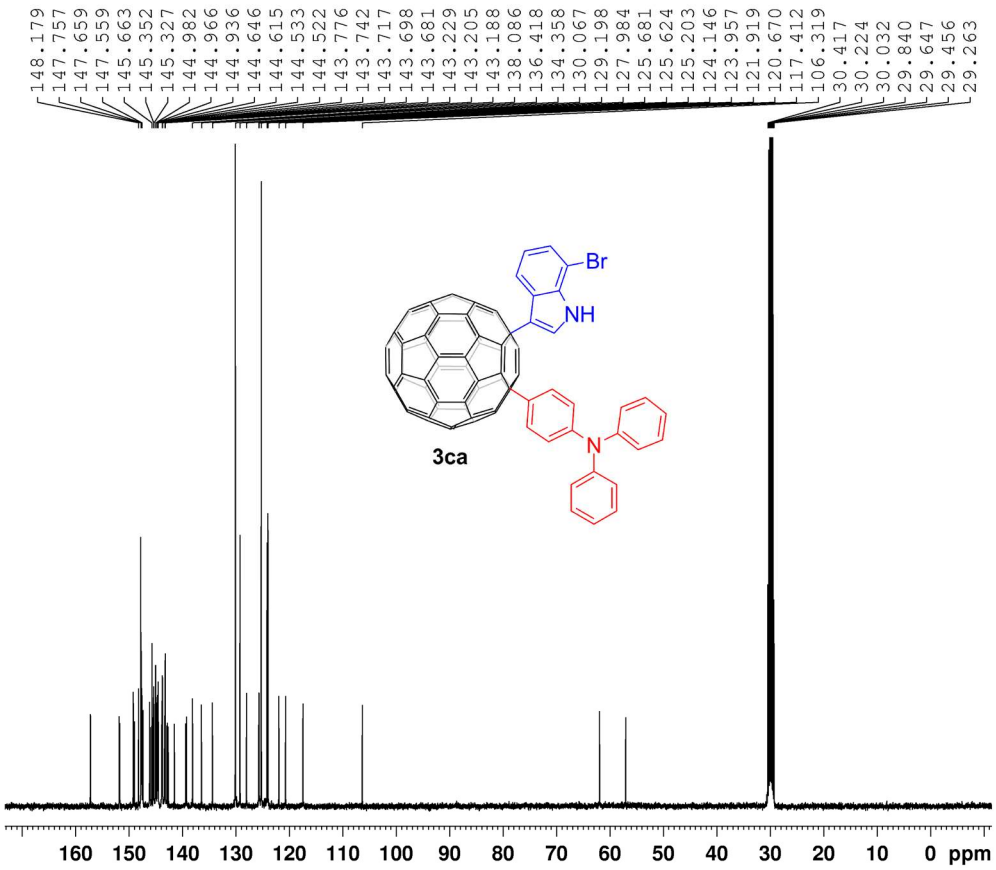
NAME          Dec03-2020
EXPNO                  1
PROCNO                 1
Date_           20201203
Time               10.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   362
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.50 usec
PL1               -2.00 dB
PL1W        13.45602226 W
SFO1       400.1324710 MHz
SI                 32768
SF         400.1300075 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ca

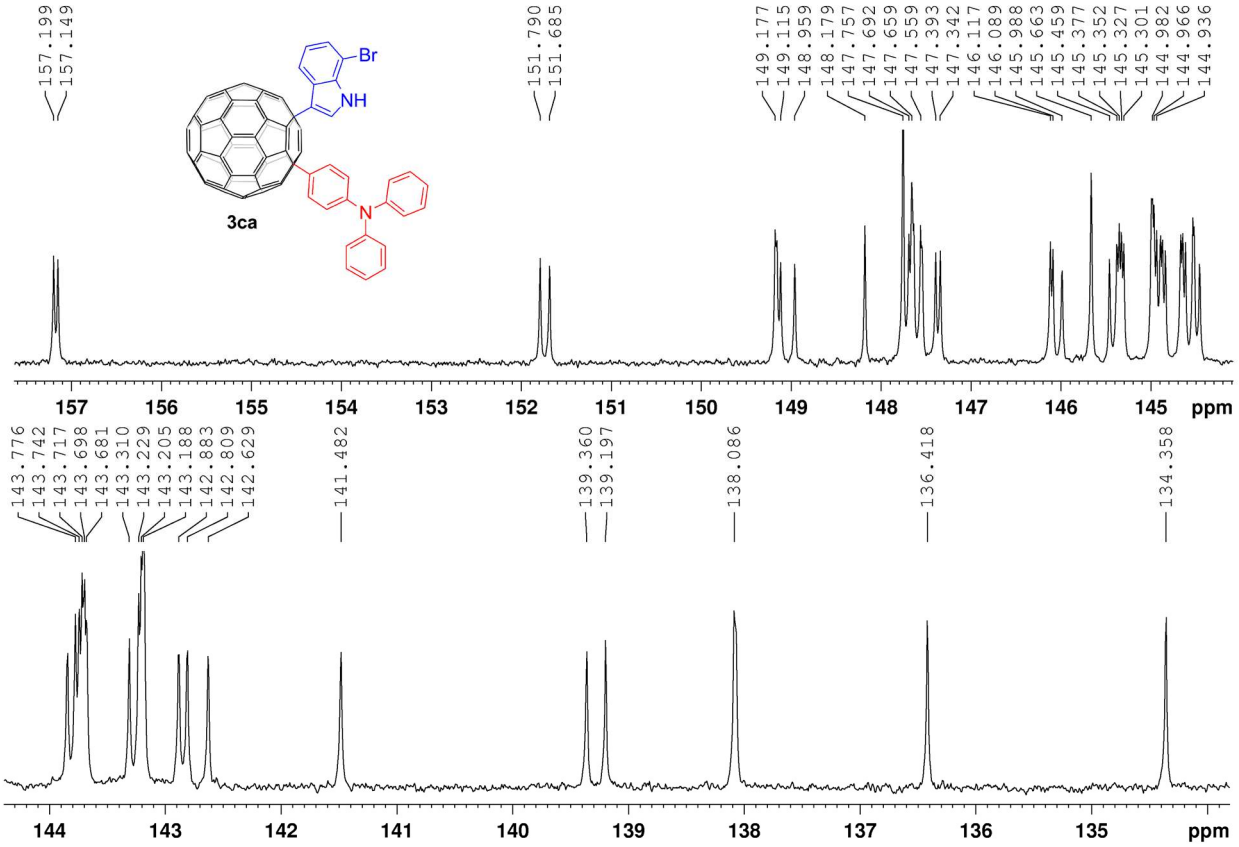



```
NAME          Dec03-2020
EXPNO                  5
PROCNO                 1
Date_           20201204
Time               8.35
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                 15769
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  80.6
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

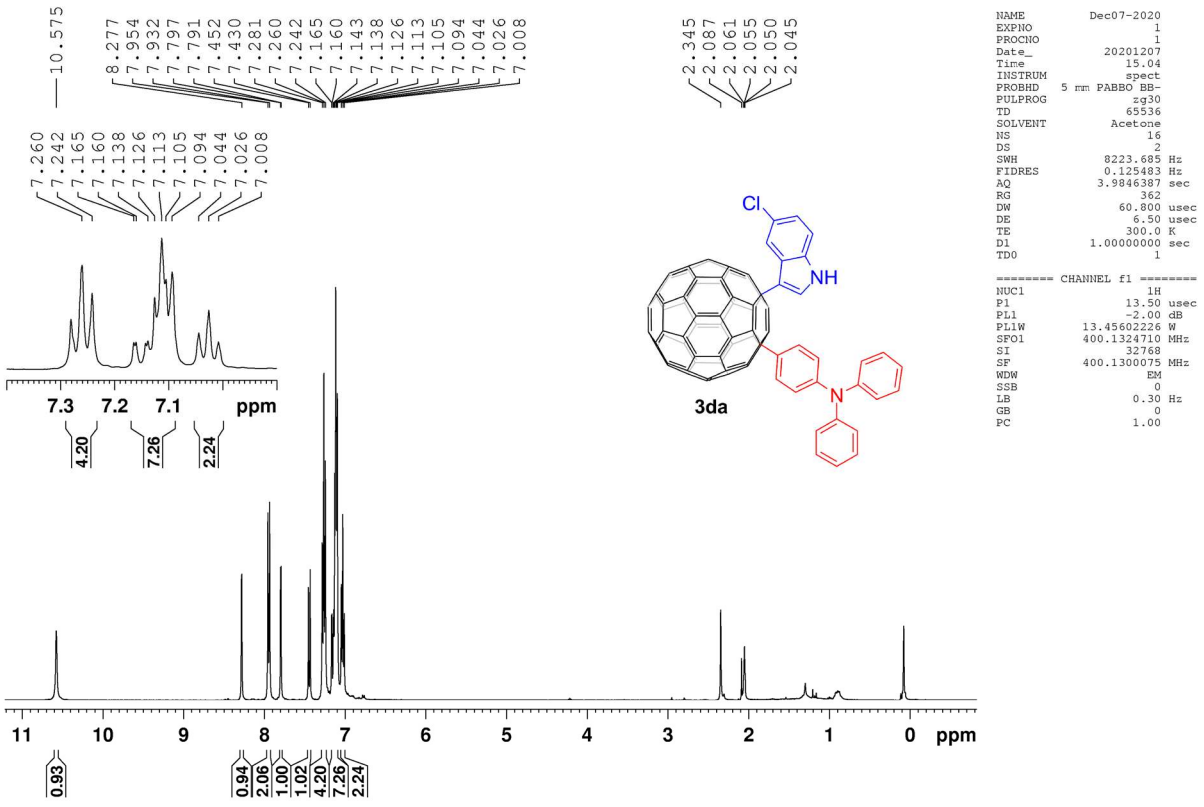
======== CHANNEL f1 ========
NUC1                 13C
P1                 9.30 usec
PL1               -2.00 dB
PL1W        54.14257431 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              13.46 dB
PL13             120.00 dB
PL2W        13.45602226 W
PL12W        0.38275132 W
PL13W        0.00000000 W
SFO2       400.1316005 MHz
SI                 32768
SF         100.6127271 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ca



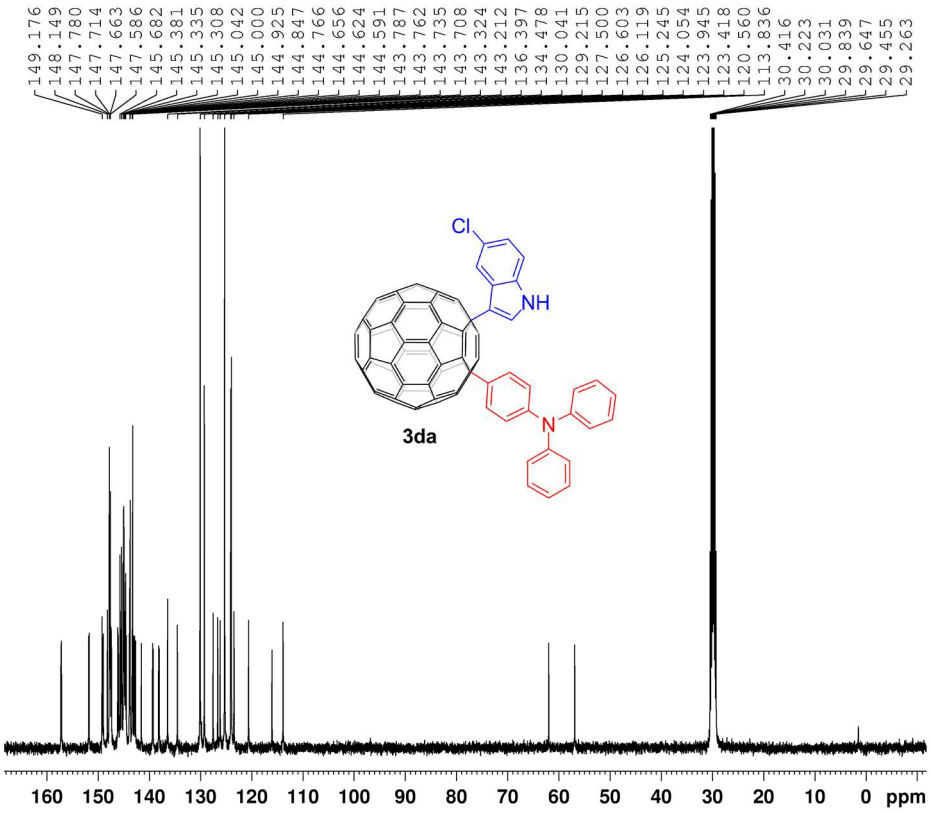
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3da




```
NAME                Dec07-2020
EXPNO                        1
PROCNO                       1
Date_                 20201207
Time                     15.04
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT               Acetone
NS                           16
DS                            2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9846387 sec
RG                          362
DW                     60.800 usec
DE                      6.50 usec
TE                     300.0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     13.50 usec
PL1                    -2.00 dB
PL1W            13.45602226 W
SFO1           400.1324710 MHz
SI                       32768
SF             400.1300075 MHz
WDW                         EM
SSB                          0
LB                     0.30 Hz
GB                           0
PC                     1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3da

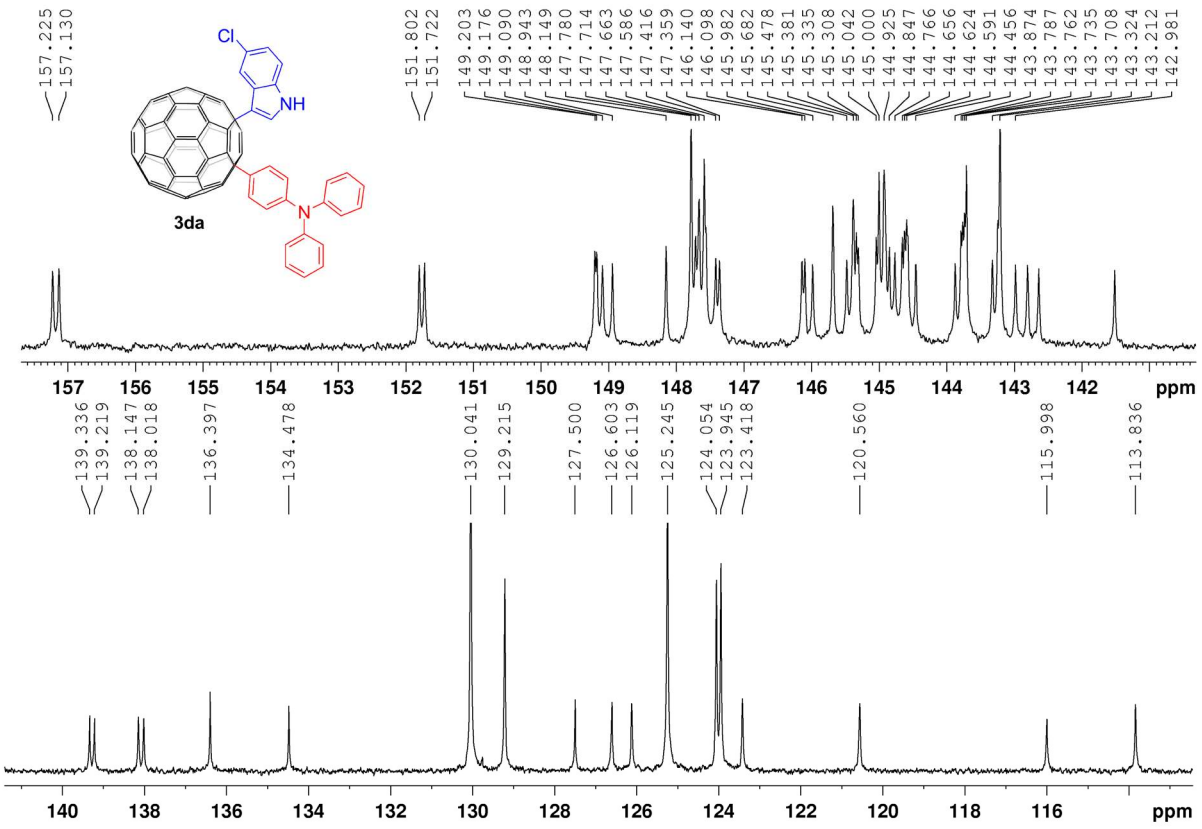



```
NAME            Dec07-2020
EXPNO                    2
PROCNO                   1
Date_             20201207
Time                19.35
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                   15204
DS                       4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631888 sec
RG                    80.6
DW                  20.800 usec
DE                   6.50 usec
TE                  300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

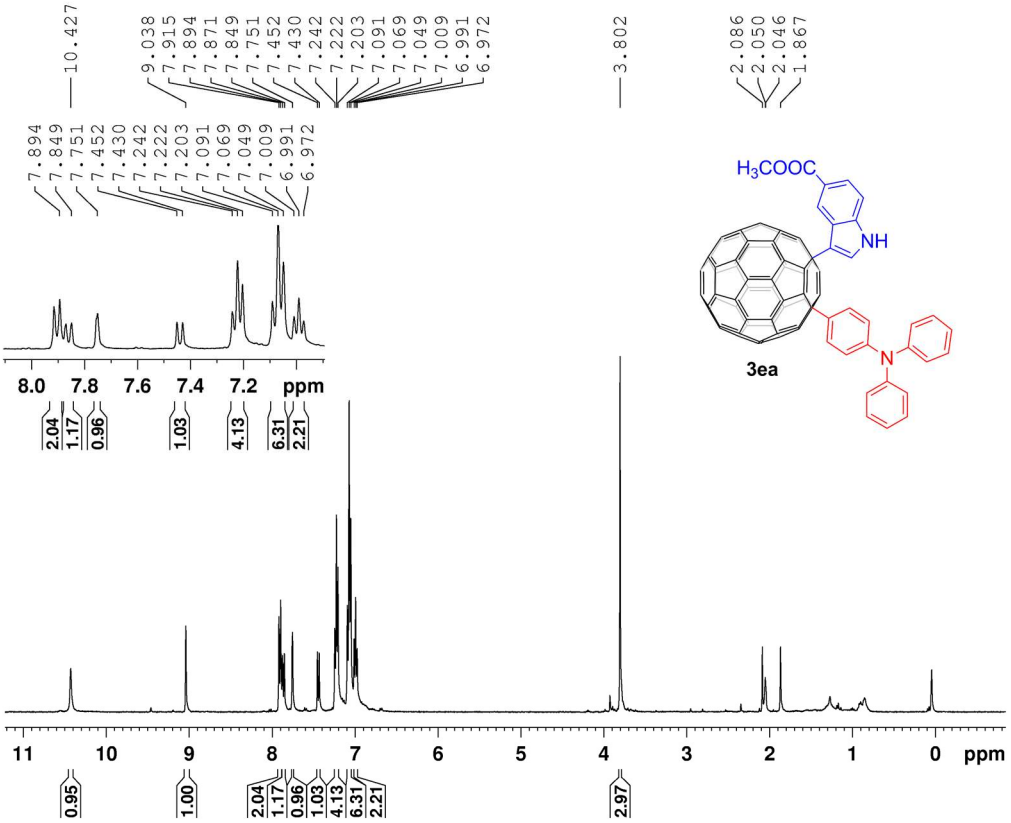
======== CHANNEL f1 ========
NUC1                   13C
P1                    9.30 usec
PL1                  -2.00 dB
PL1W          54.14257431 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 13.46 dB
PL13                120.00 dB
PL2W          13.45602226 W
PL12W          0.38275132 W
PL13W          0.00000000 W
SFO2          400.1316005 MHz
SI                   32768
SF            100.6127273 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3da



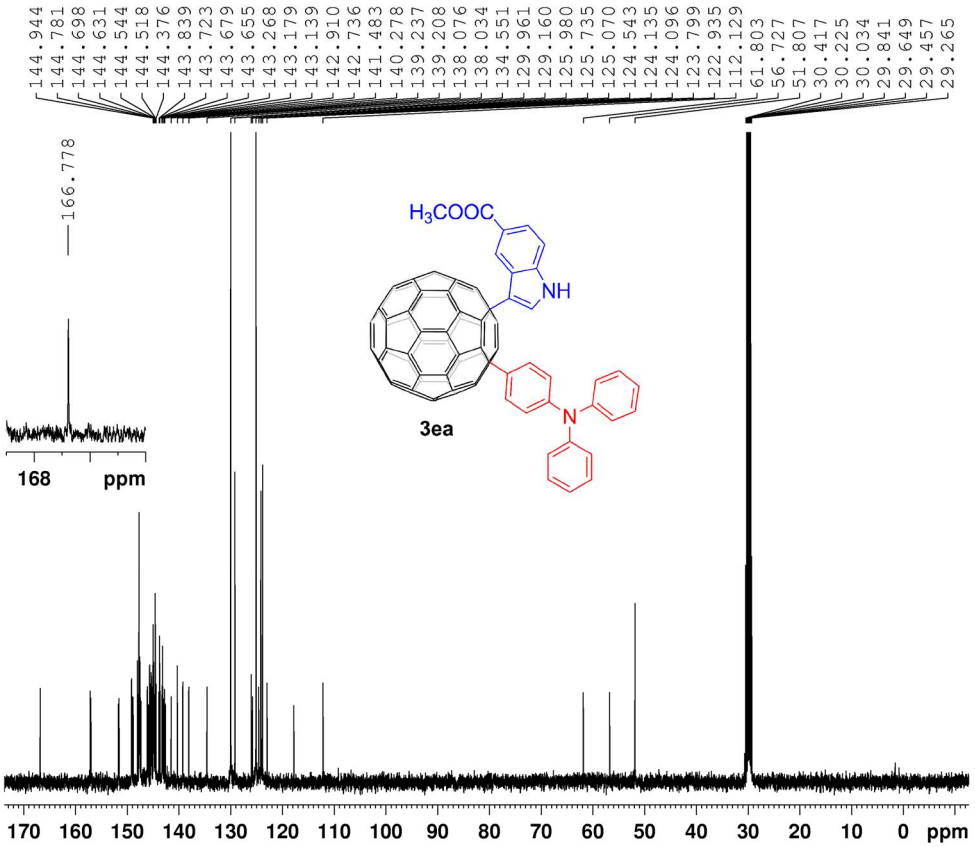
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ea




```
NAME            Dec04-2020
EXPNO                    1
PROCNO                   1
Date_             20201204
Time                 14.33
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT            Acetone
NS                       16
DS                        2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      362
DW                 60.800 usec
DE                  6.50 usec
TE                  300.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.50 usec
PL1                  -2.00 dB
PL1W           13.45602226 W
SFO1           400.1324710 MHz
SI                    32768
SF             400.1300080 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ea

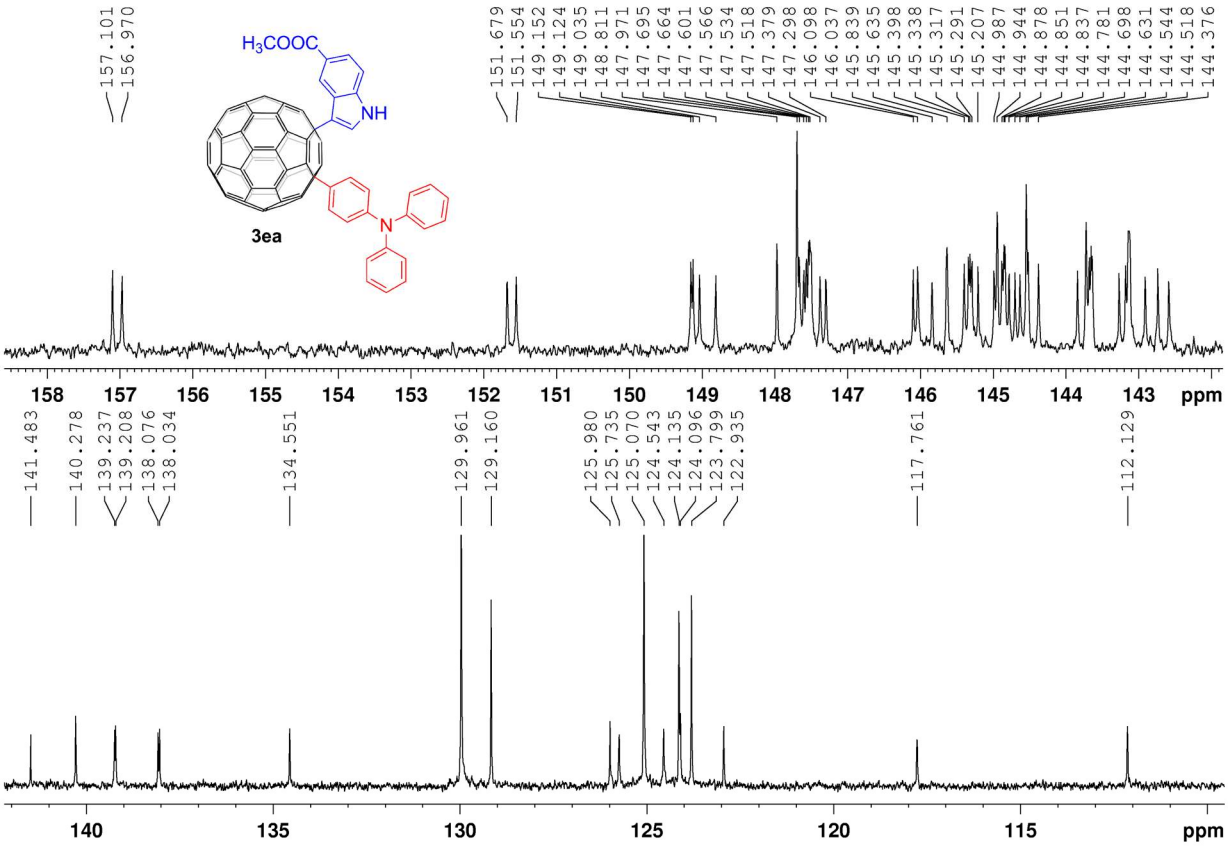



```
NAME            Dec04-2020
EXPNO                    2
PROCNO                   1
Date_             20201204
Time                 20.17
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                   20480
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                    71.8
DW                 20.800 usec
DE                  6.50 usec
TE                  300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

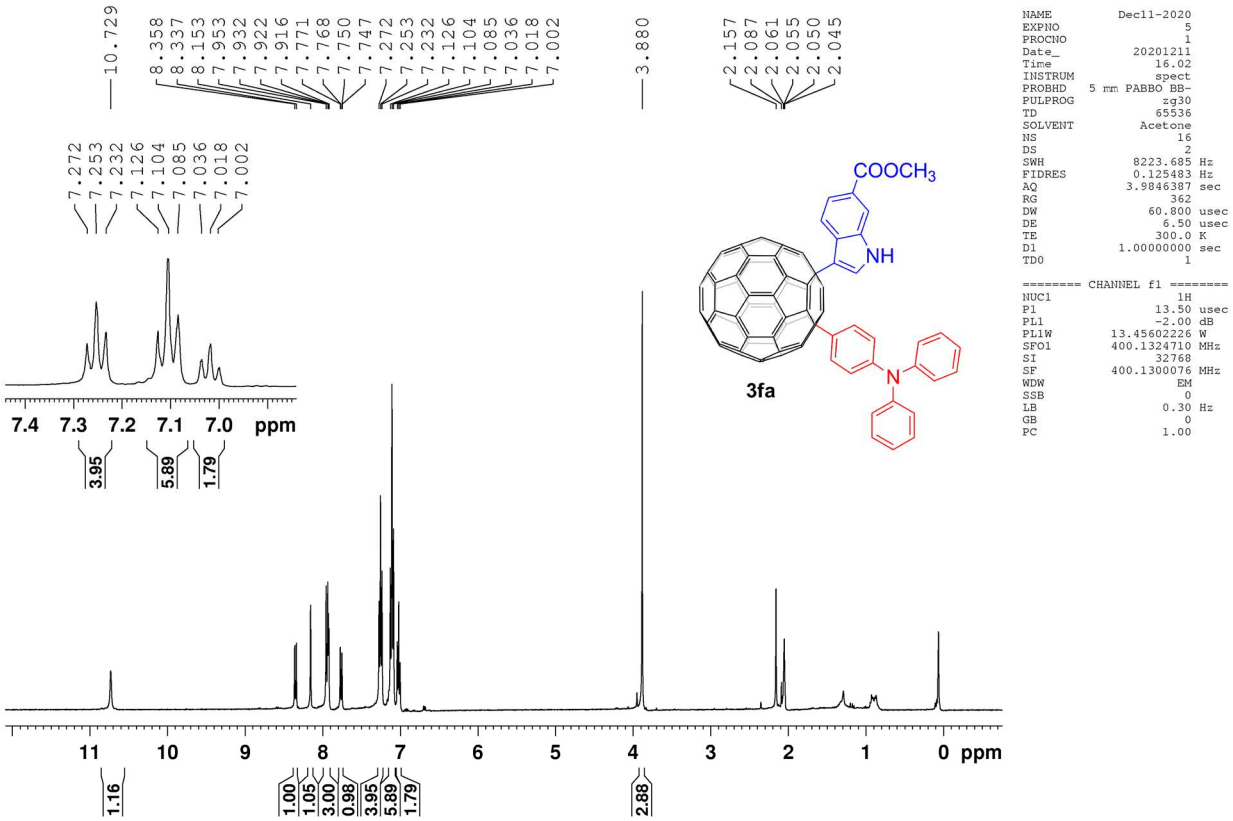
======== CHANNEL f1 ========
NUC1                   13C
P1                   9.30 usec
PL1                  -2.00 dB
PL1W           54.14257431 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                  -2.00 dB
PL12                 13.46 dB
PL13                120.00 dB
PL2W           13.45602226 W
PL12W           0.38275132 W
PL13W           0.00000000 W
SFO2           400.1316005 MHz
SI                    32768
SF             100.6127320 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ea



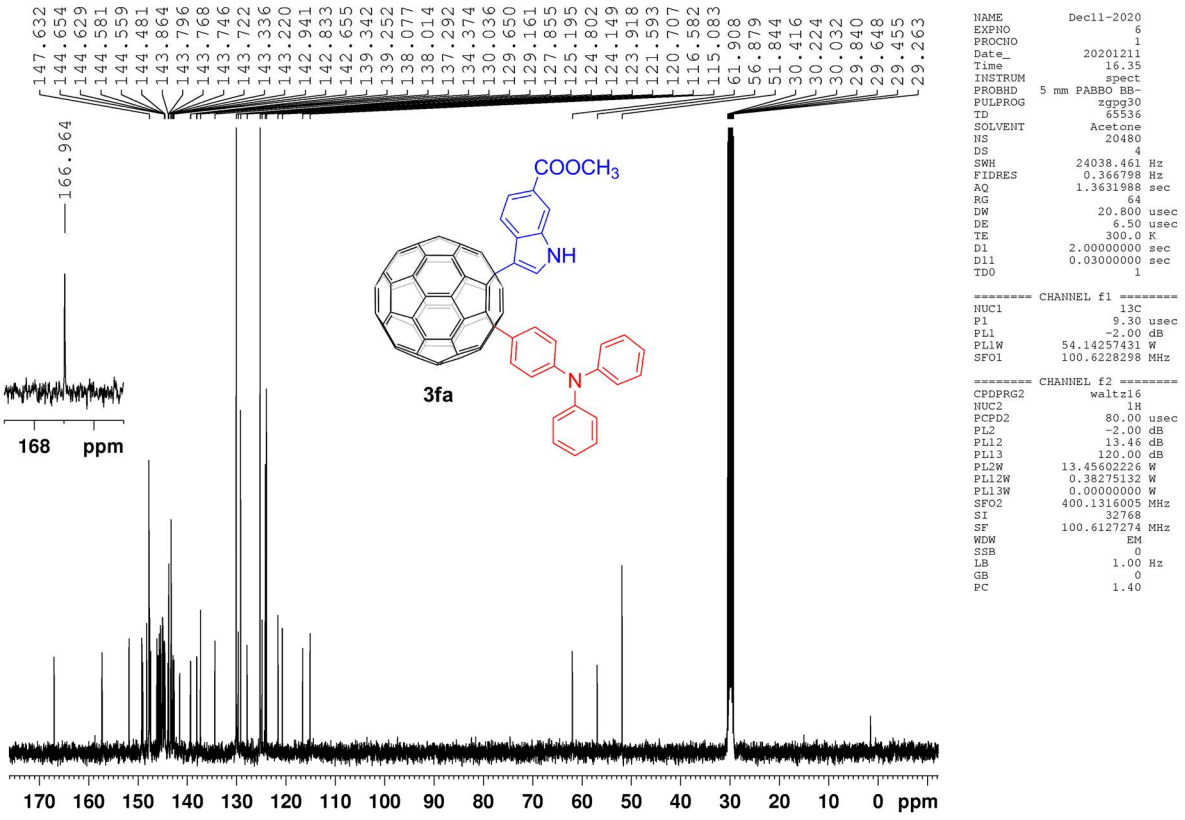
^1H NMR (400 MHz, CS_2/d_6-acetone) of compound 3fa



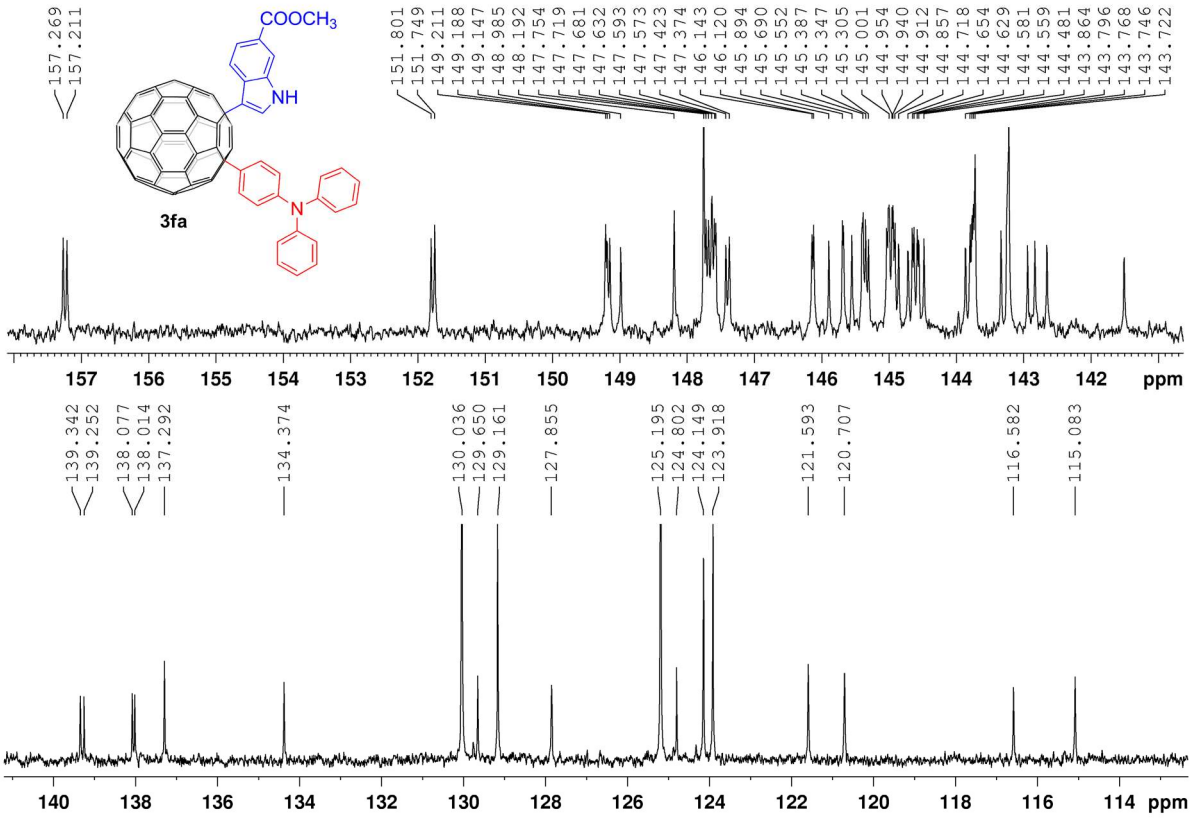

```
NAME            Dec11-2020
EXPNO                    5
PROCNO                   1
Date_             20201211
Time                 16.02
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                      16
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     362
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W          13.45602226 W
SFO1          400.1324710 MHz
SI                   32768
SF            400.1300076 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

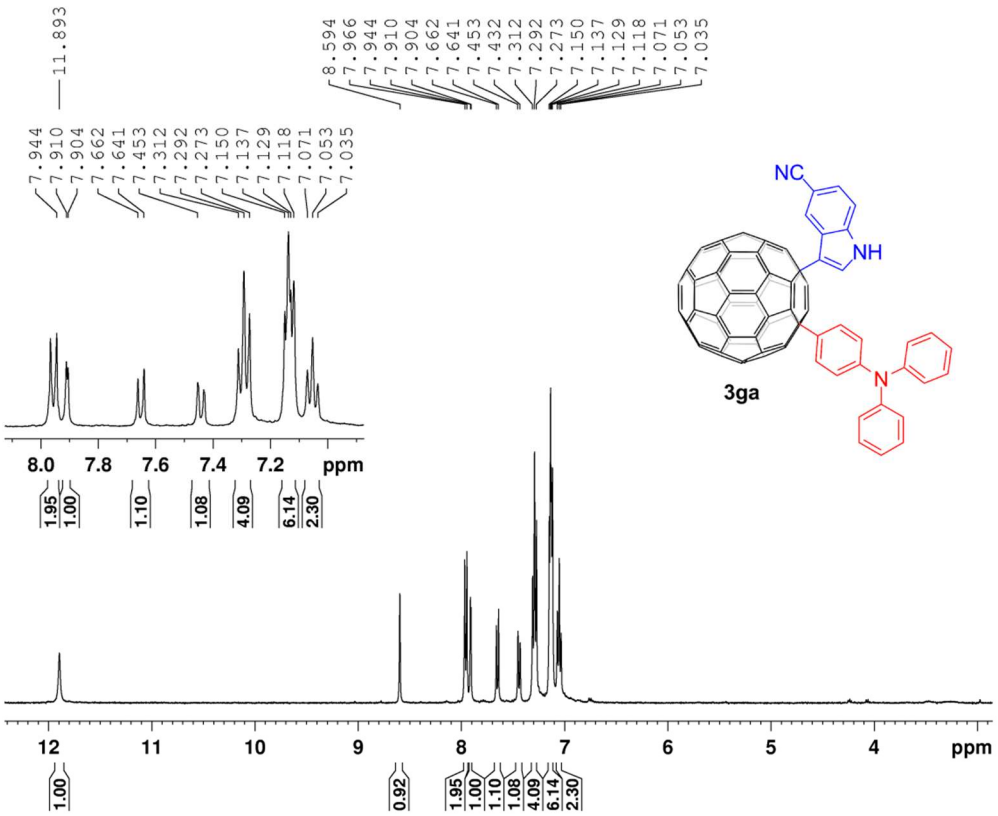
^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fa



Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fa



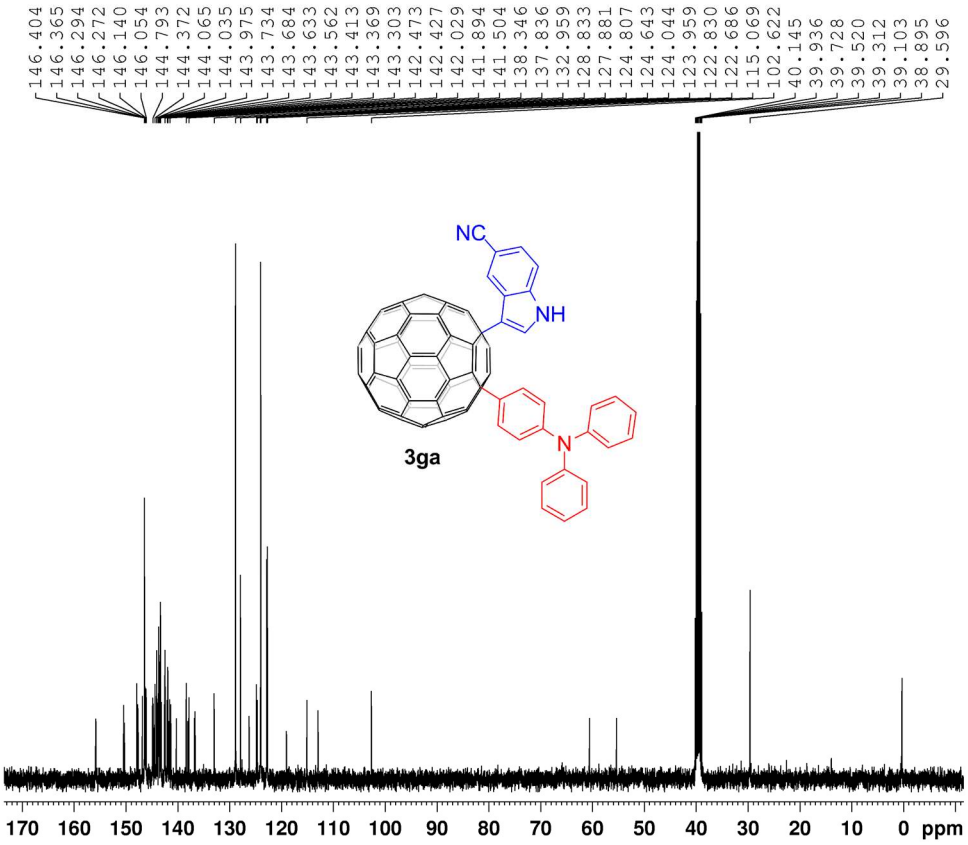
^{1}H NMR (400 MHz, CS_2/d_6-DMSO) of compound 3ga




```
NAME           Dec23-2020
EXPNO                   1
PROCNO                  1
Date_            20201223
Time                16.53
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   362
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                1H
P1                13.50 usec
PL1               -2.00 dB
PL1W        13.45602226 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300037 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-DMSO) of compound 3ga

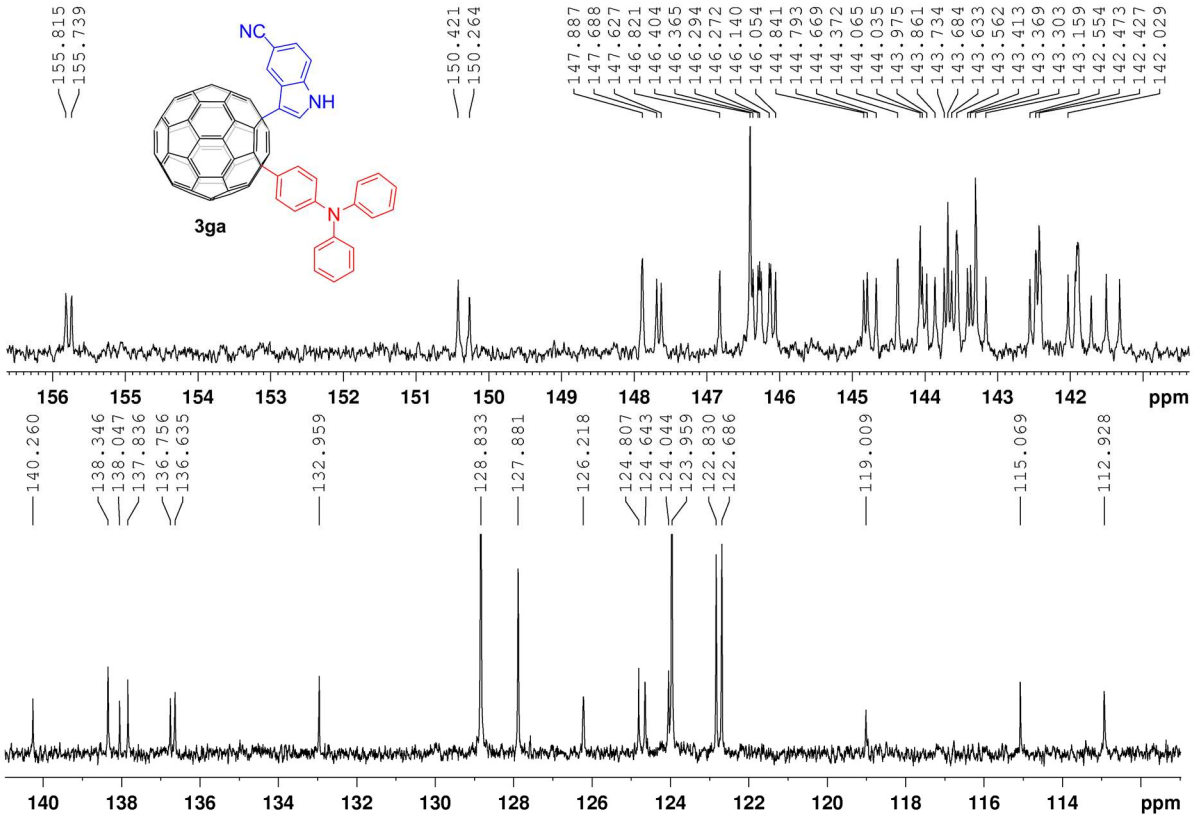



```
NAME           Dec23-2020
EXPNO                   2
PROCNO                  1
Date_            20201223
Time                18.42
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                 16359
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  80.6
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

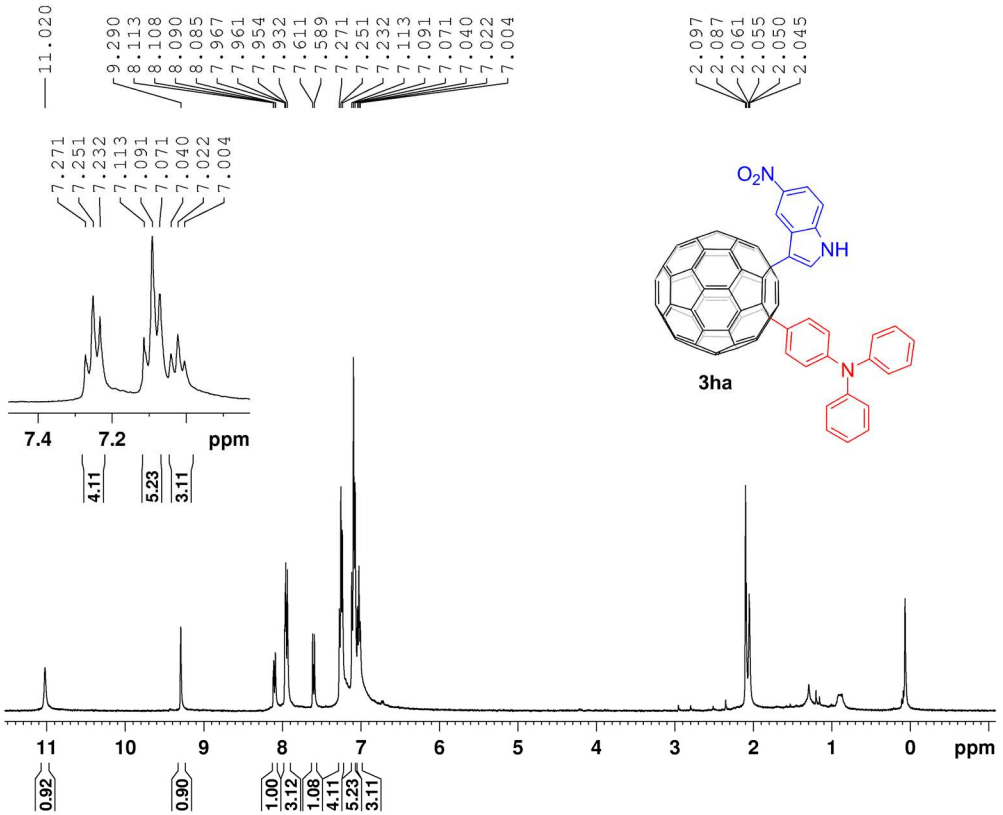
======== CHANNEL f1 ========
NUC1                13C
P1                 9.30 usec
PL1               -2.00 dB
PL1W        54.14257431 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              13.46 dB
PL13             120.00 dB
PL2W        13.45602226 W
PL12W        0.38275132 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6128626 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-DMSO) of compound 3ga

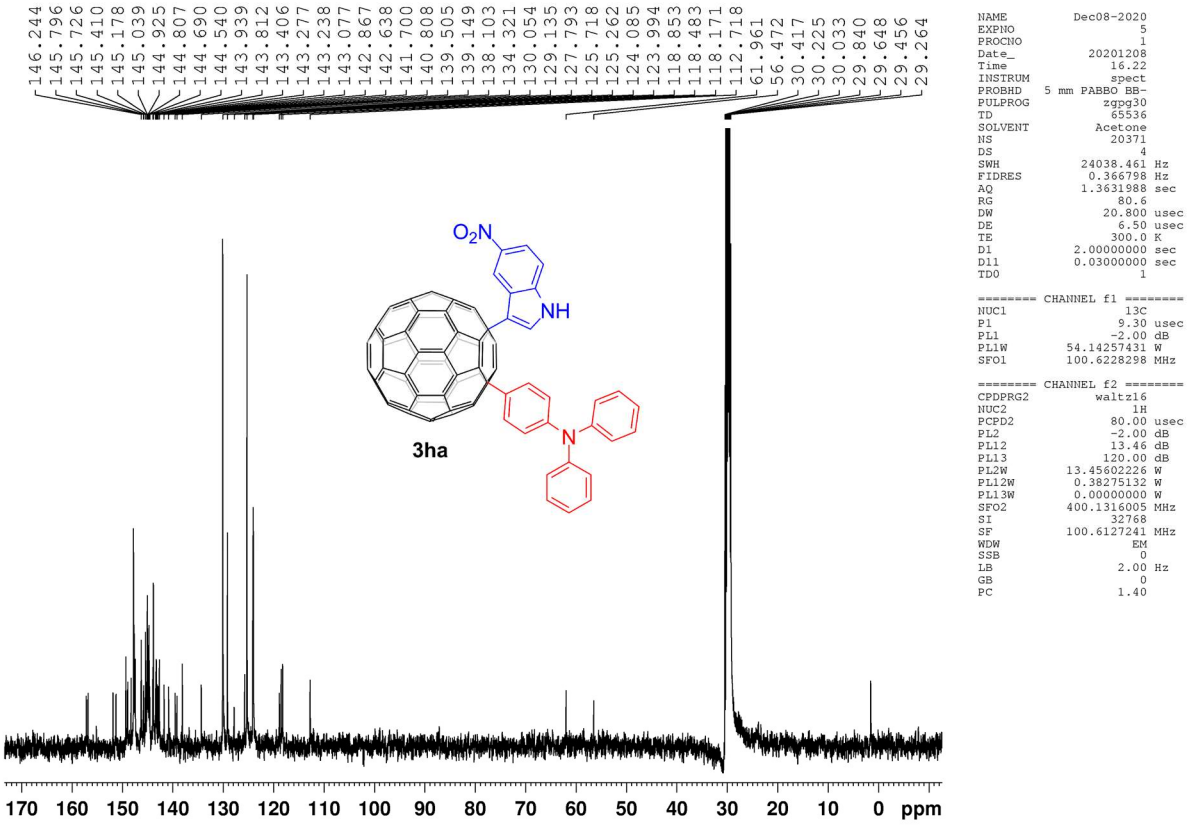


^1H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ha

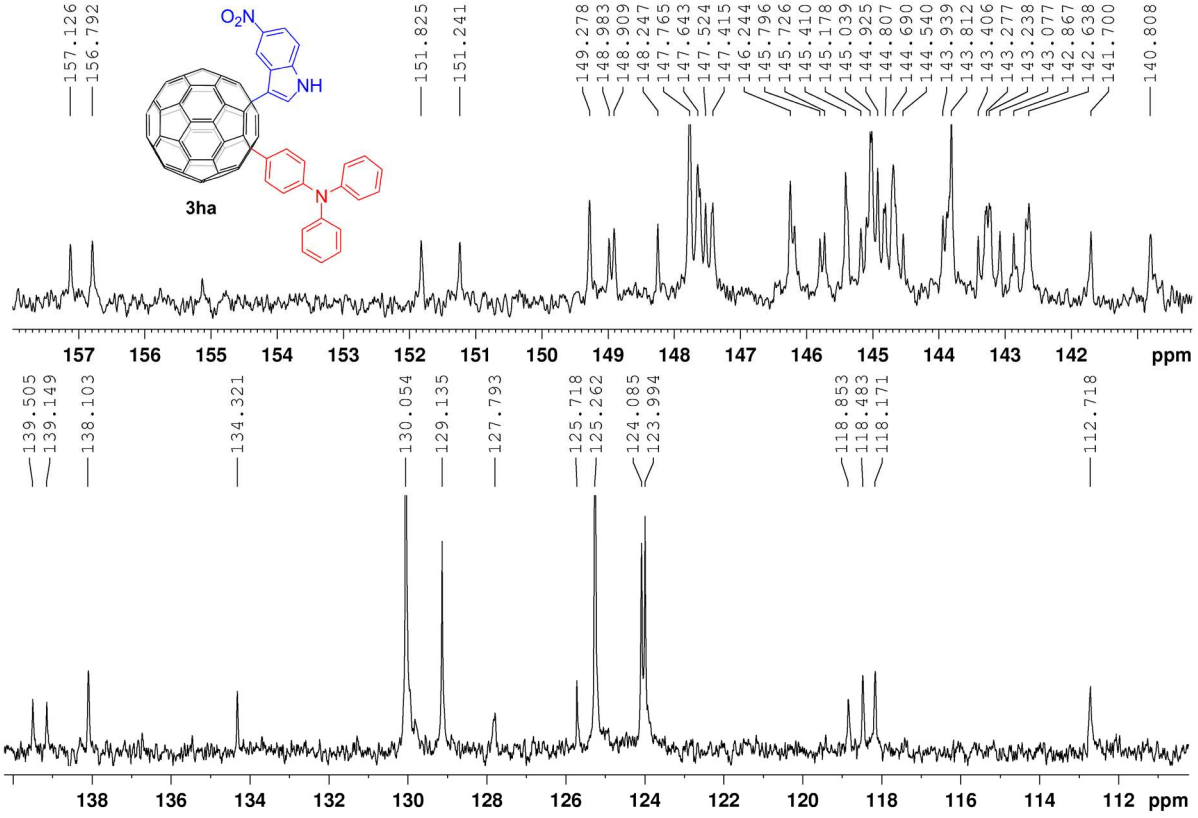


NAME	Dec08-2020
EXPNO	4
PROCNO	1
Date_	20201208
Time	11.17
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	Acetone
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	362
DW	60.800 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.50 usec
PL1	-2.00 dB
PL1W	13.45602226 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300075 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

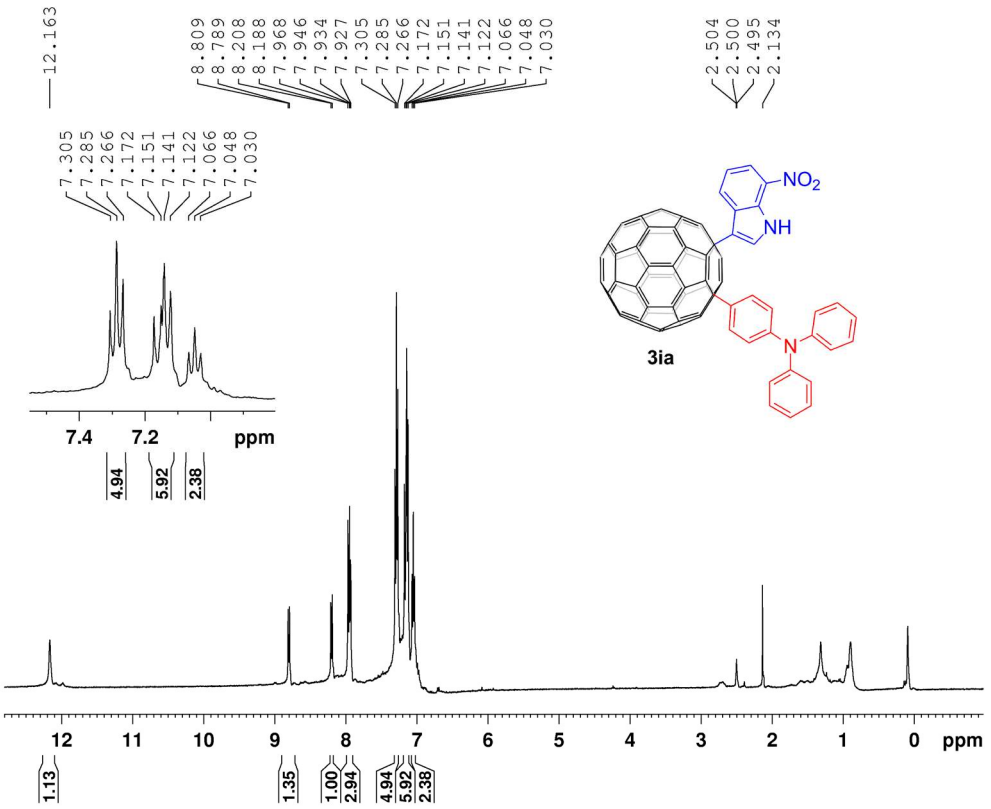
^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ha



Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ha



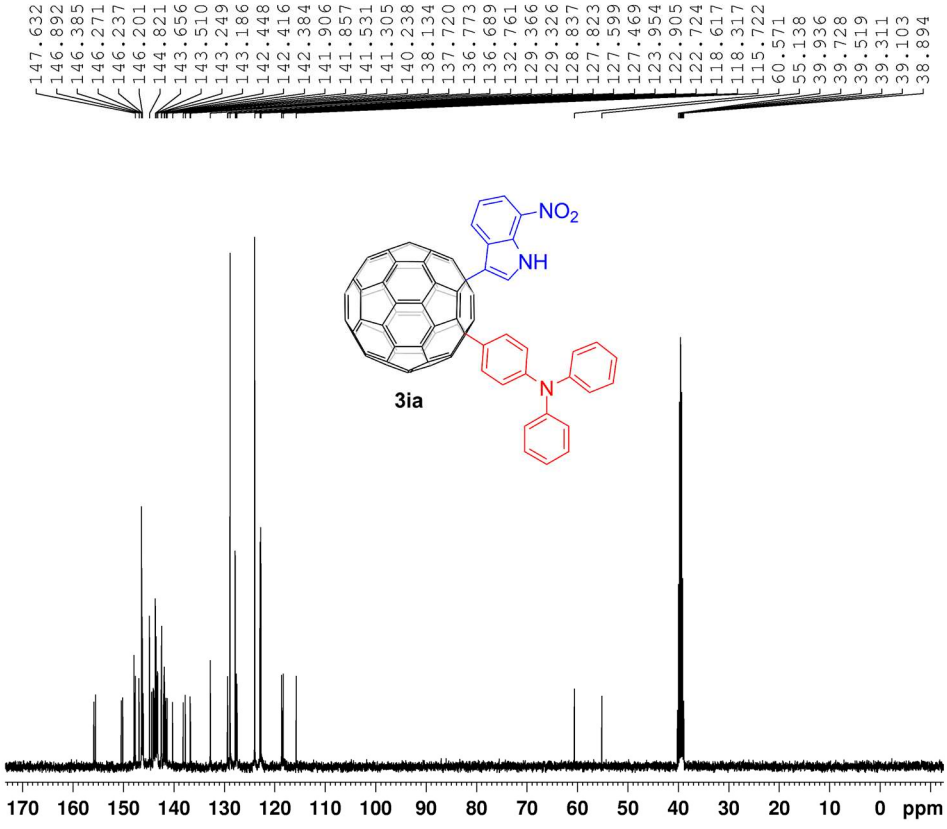
^{1}H NMR (400 MHz, CS_2/d_6-DMSO) of compound 3ia




```
NAME            Dec22-2020
EXPNO                    1
PROCNO                   1
Date_             20201222
Time                 10.35
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH               8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     362
DW                60.800 usec
DE                 6.50 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W          13.45602226 W
SFO1          400.1324710 MHz
SI                  32768
SF            400.1300046 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-DMSO) of compound 3ia

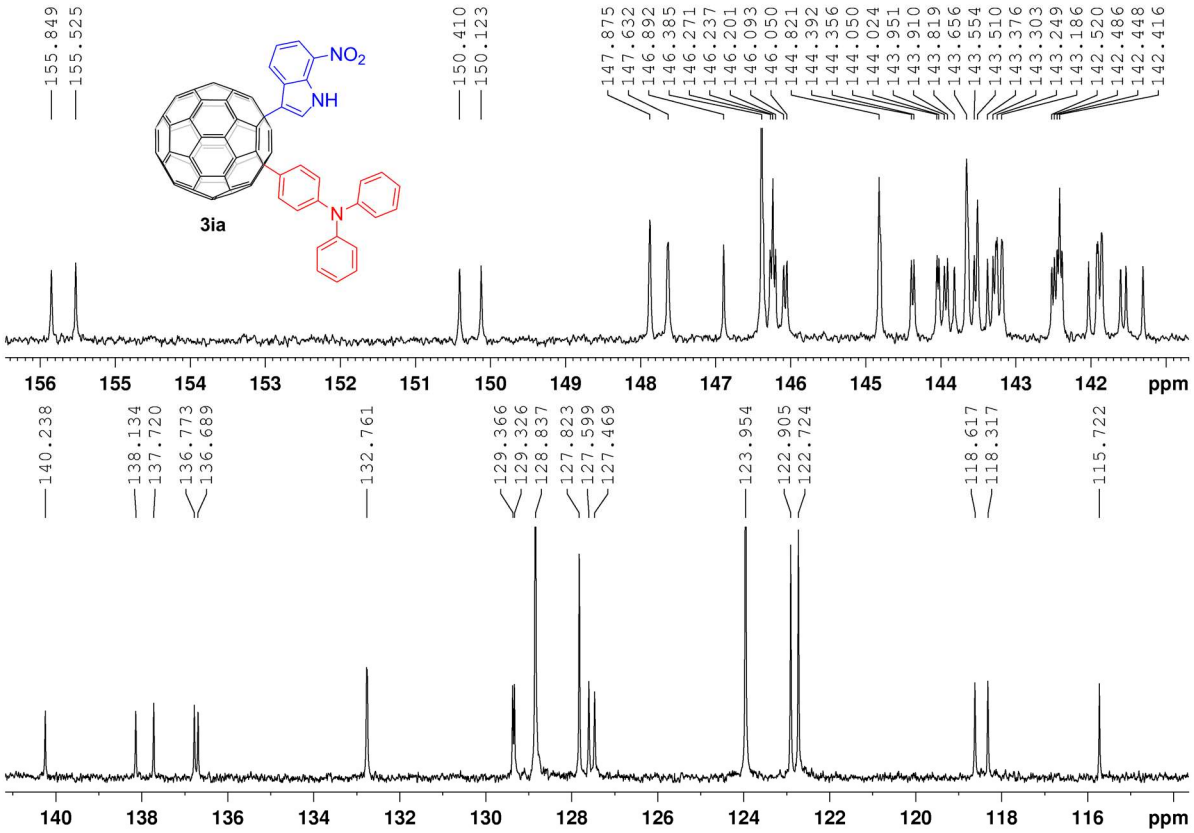



```
NAME            Apr07-2021
EXPNO                    3
PROCNO                   1
Date_             20210407
Time                 16.34
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                  10240
DS                      4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                    90.5
DW                20.800 usec
DE                 6.50 usec
TE                 300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

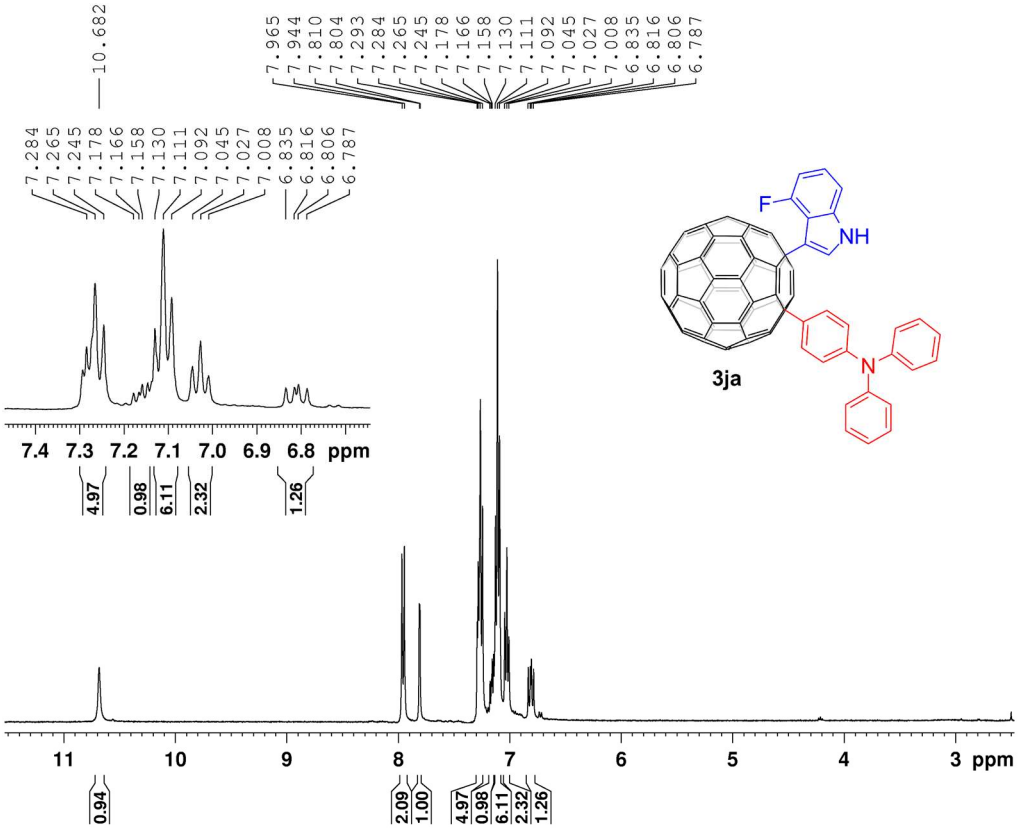
======== CHANNEL f1 ========
NUC1                   13C
P1                  9.30 usec
PL1                -2.00 dB
PL1W          54.14257431 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.46 dB
PL13              120.00 dB
PL2W          13.45602226 W
PL12W          0.38275132 W
PL13W          0.00000000 W
SFO2          400.1316005 MHz
SI                  32768
SF            100.6128618 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-DMSO) of compound 3ia



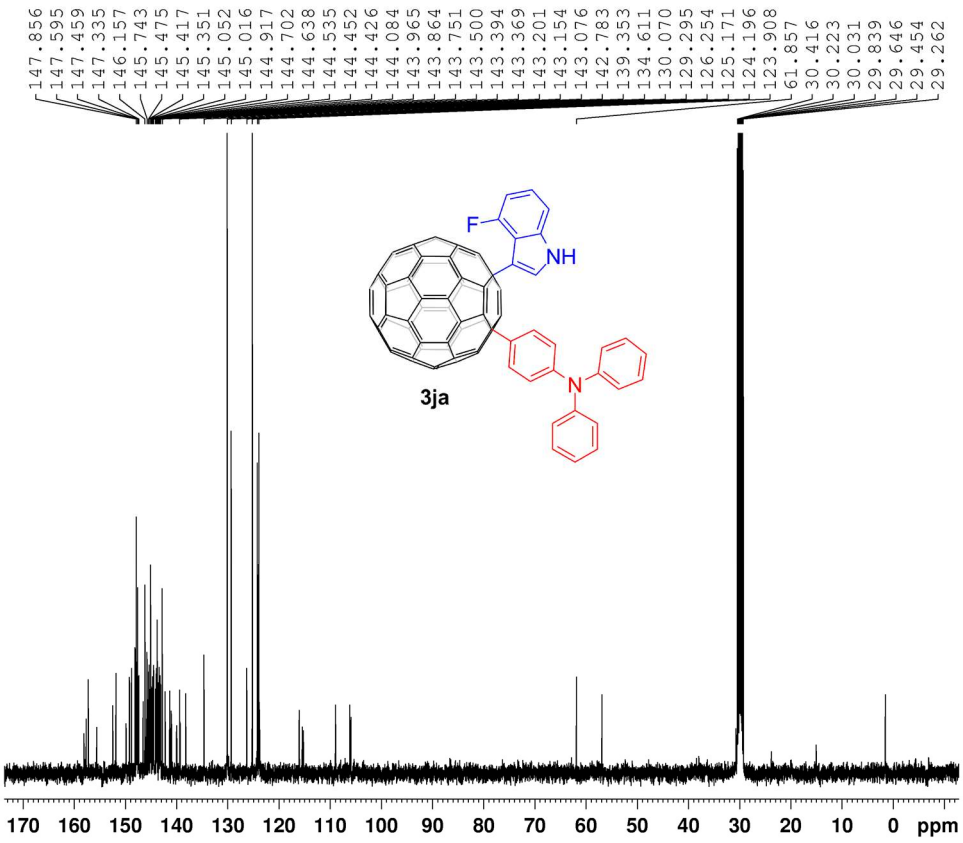
^1H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ja




```
NAME            Apr21-2021
EXPNO                    2
PROCNO                   1
Date_             20210421
Time                 9.37
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                      16
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     287
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W         13.45602226 W
SFO1         400.1324710 MHz
SI                   32768
SF           400.1300074 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ja

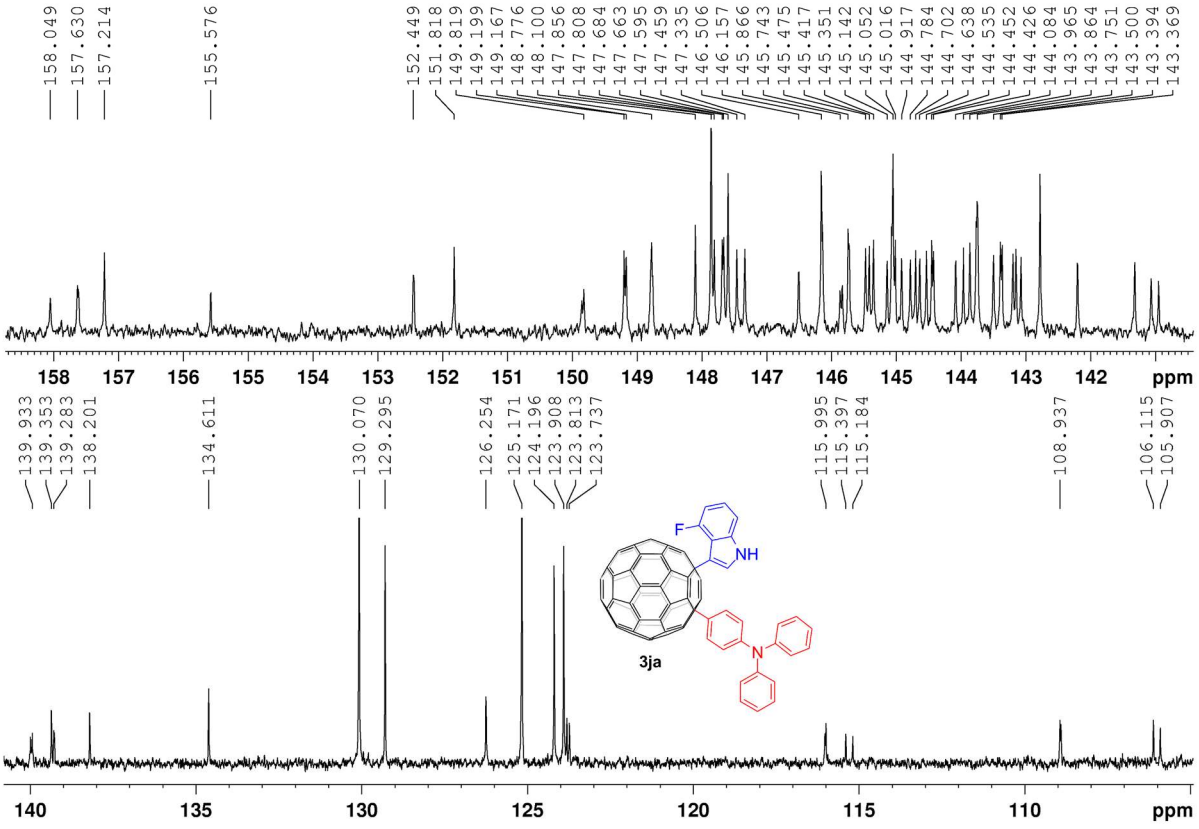



```
NAME            Apr21-2021
EXPNO                    3
PROCNO                   1
Date_             20210421
Time                16.37
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                 10240
DS                     4
SWH            24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                      80.6
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

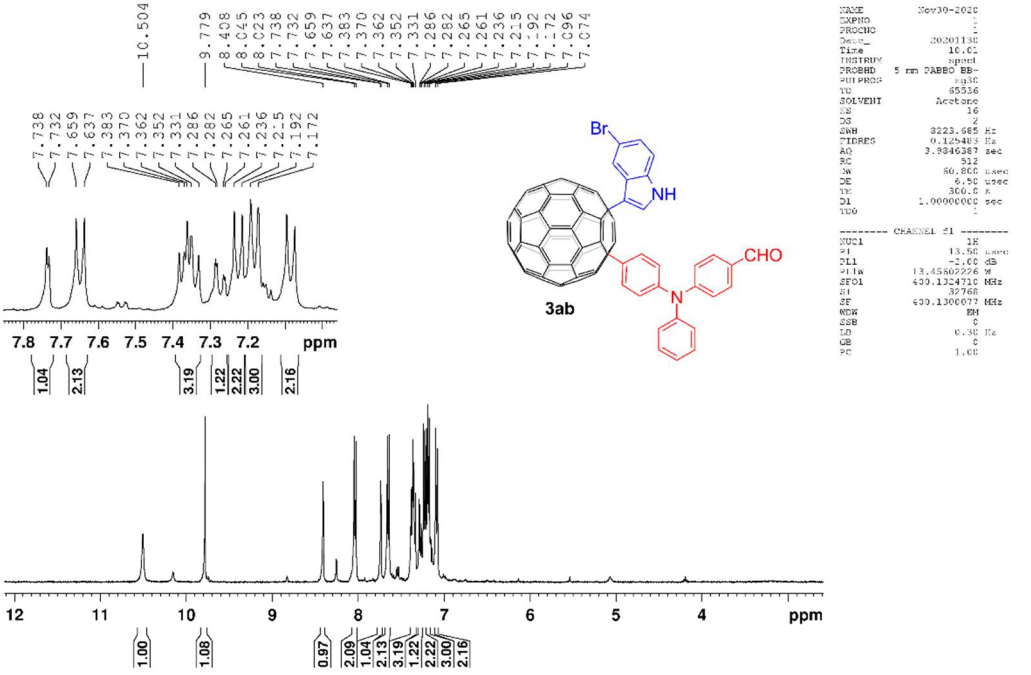
======== CHANNEL f1 ========
NUC1                    13C
P1                    9.30 usec
PL1                  -2.00 dB
PL1W           54.14257431 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 13.46 dB
PL13                120.00 dB
PL2W           13.45602226 W
PL12W           0.38275132 W
PL13W           0.00000000 W
SFO2          400.1316005 MHz
SI                   32768
SF            100.6127221 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

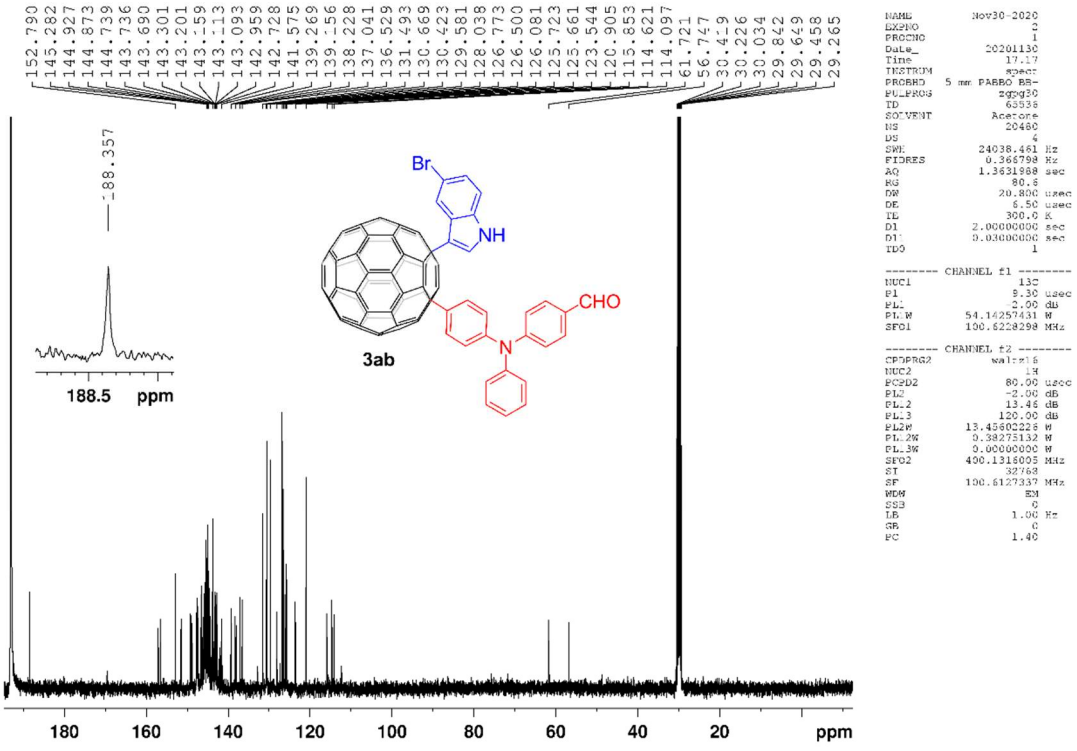
Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ja



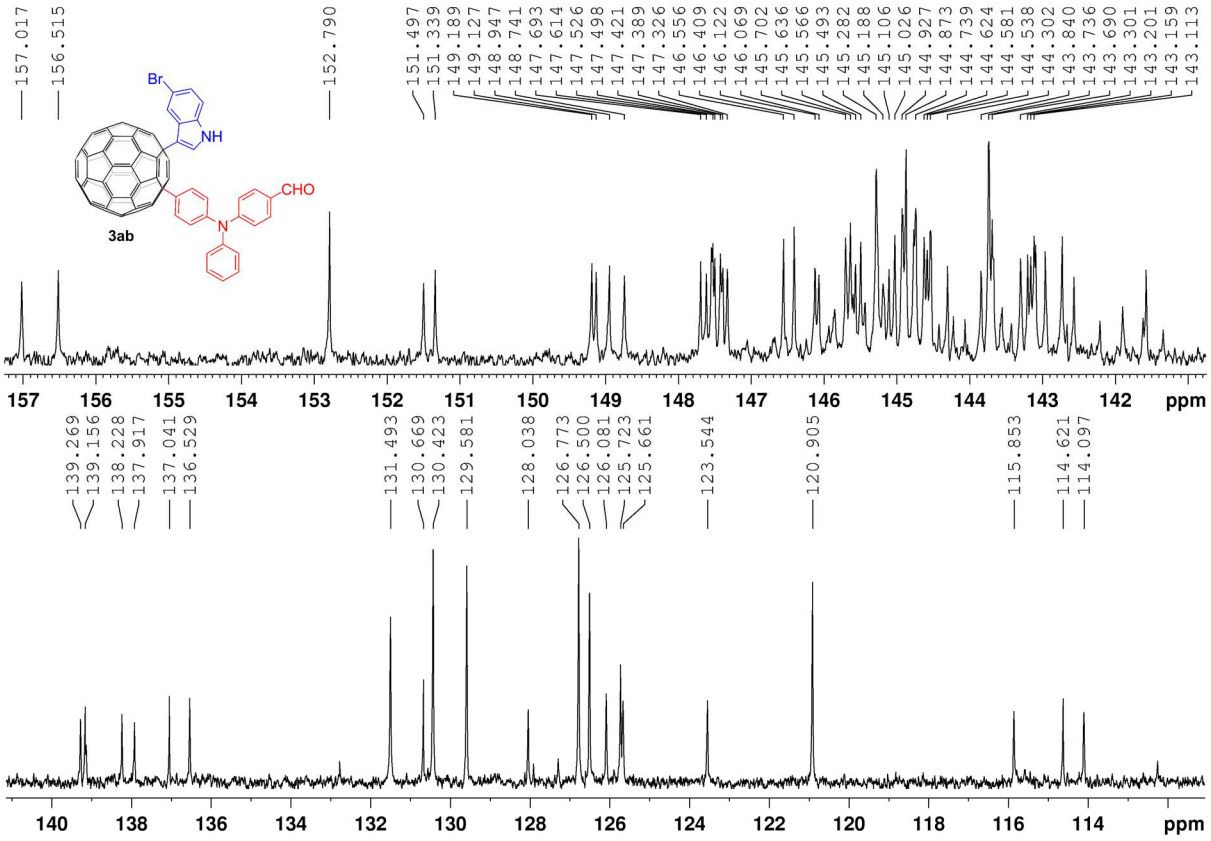
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ab



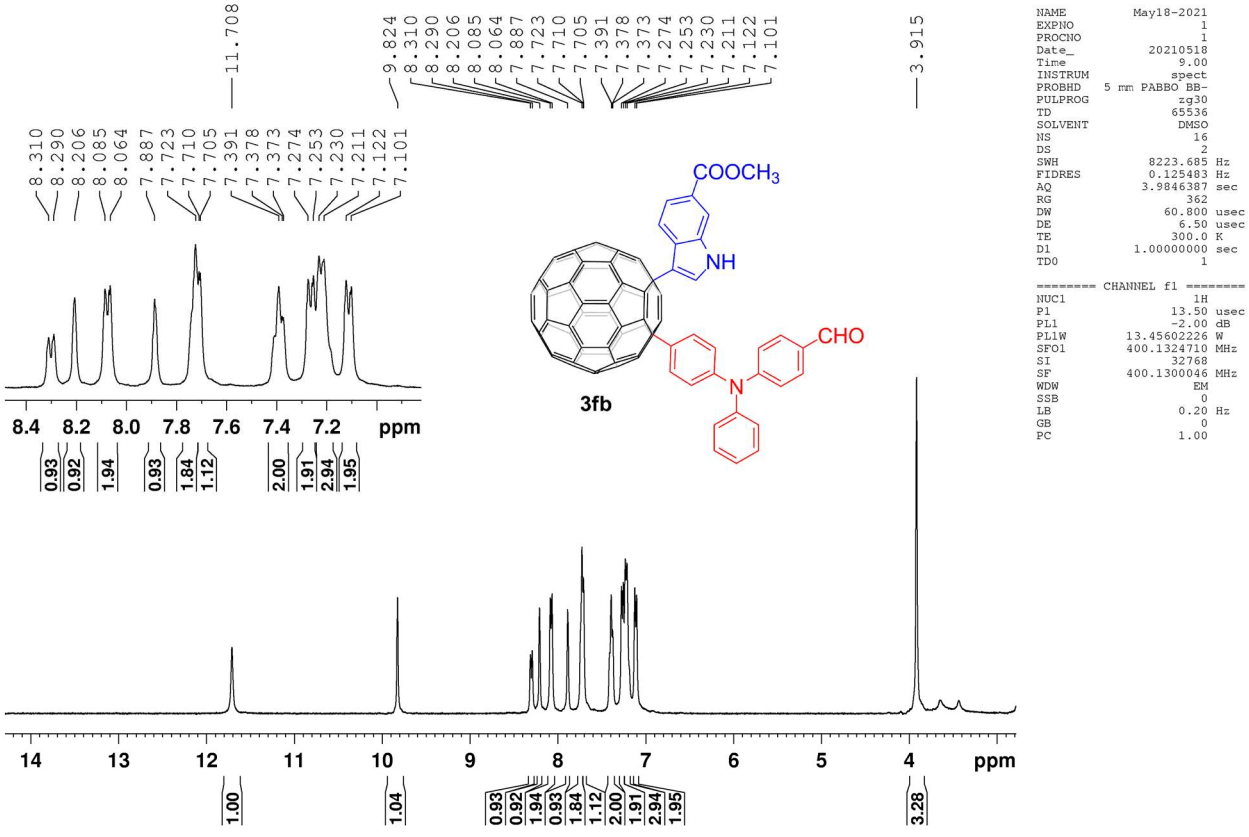
^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ab



Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ab



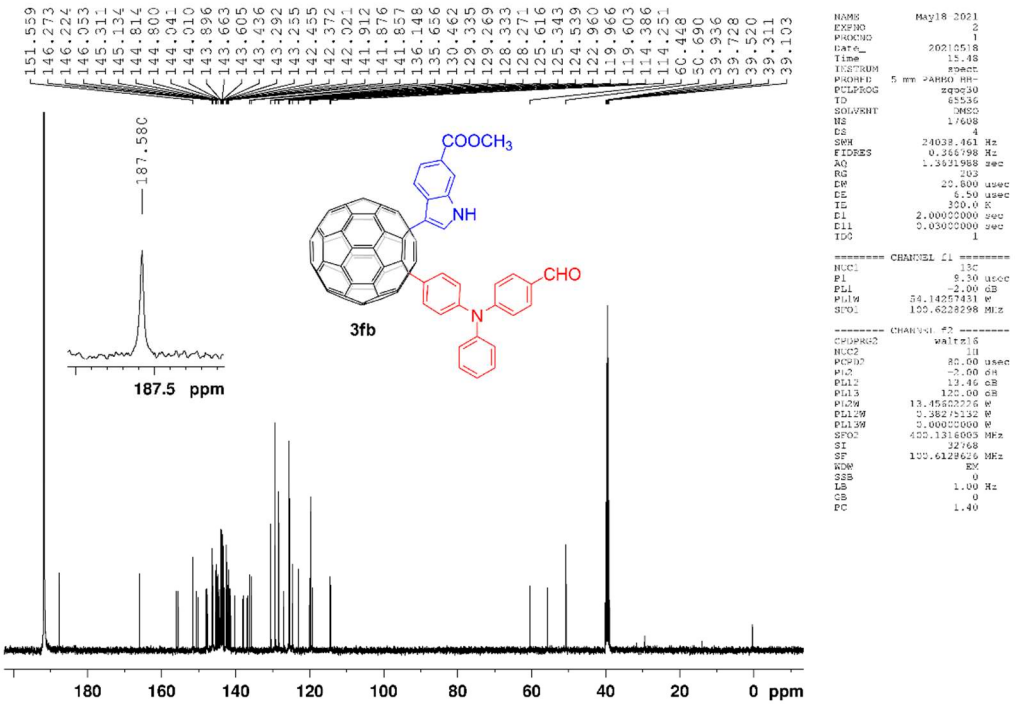
^1H NMR (400 MHz, CS_2/d_6-DMSO) of compound 3fb



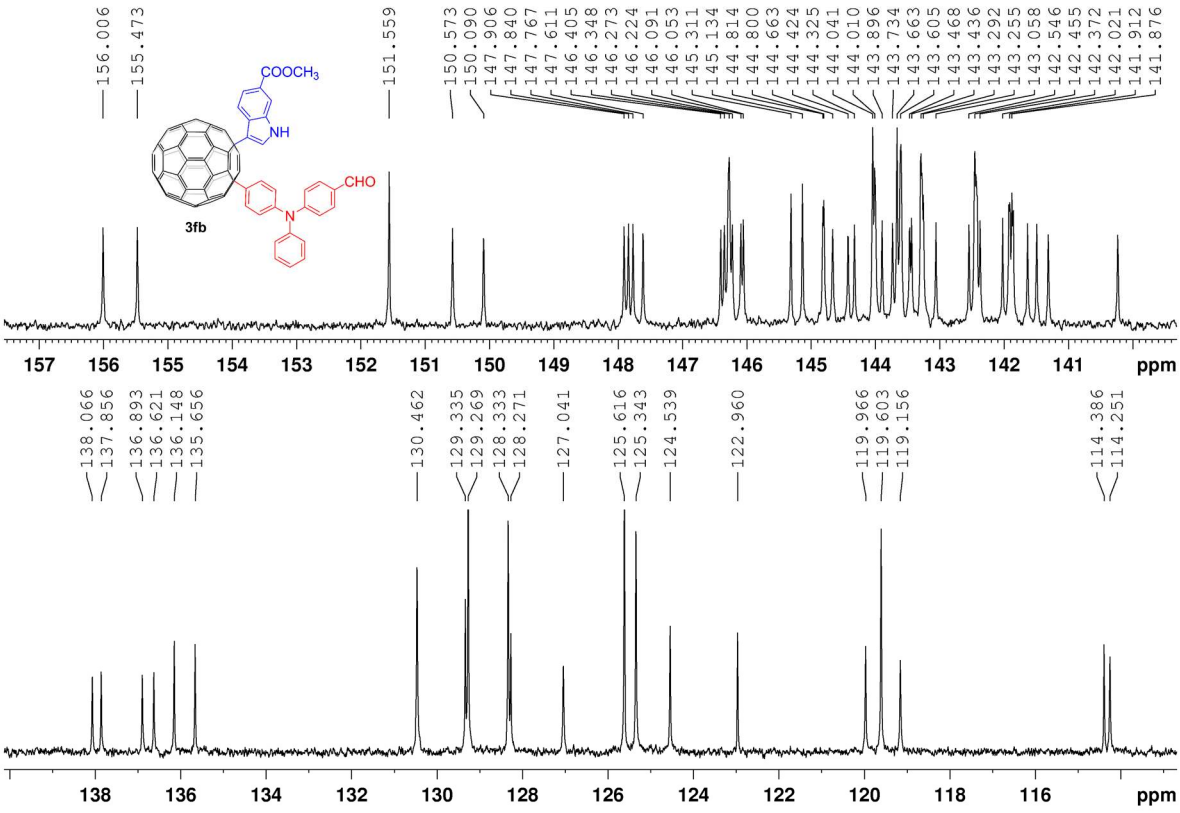

```
NAME            May18-2021
EXPNO                    1
PROCNO                   1
Date_             20210518
Time                 9.00
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                    362
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                13.50 usec
PL1               -2.00 dB
PL1W        13.45602226 W
SFO1       400.1324710 MHz
SI                 32768
SF         400.1300046 MHz
WDW                   EM
SSB                    0
LB                0.20 Hz
GB                     0
PC                1.00
```

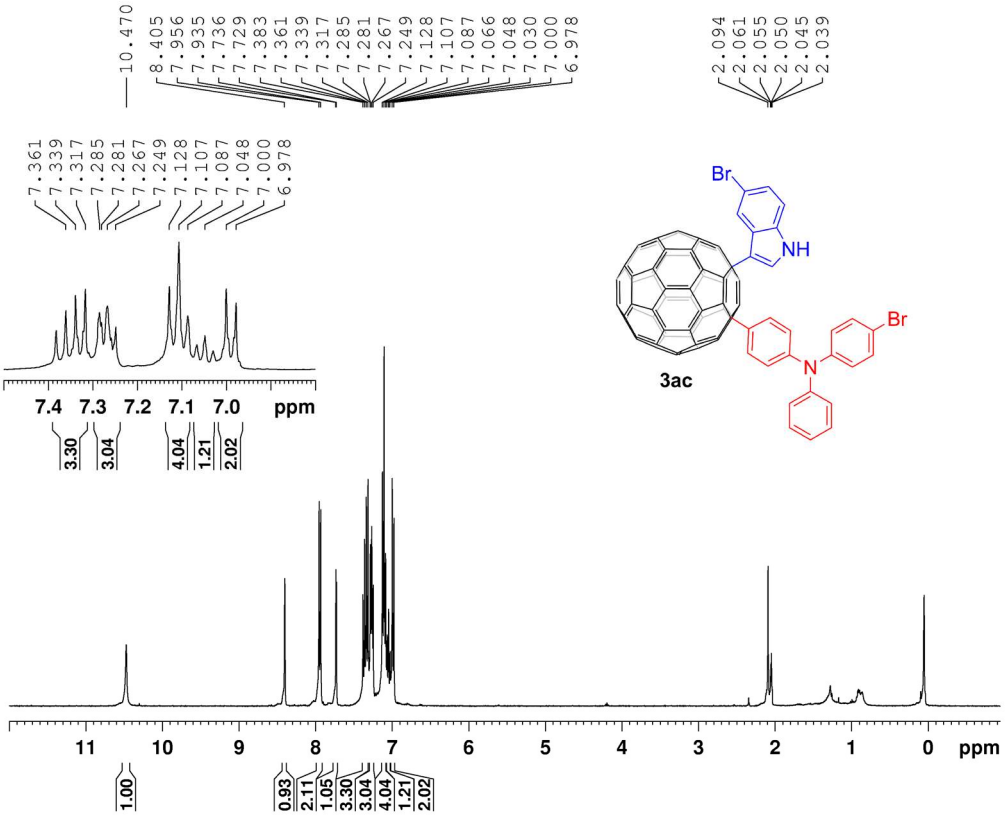
^{13}C NMR (100 MHz, CS_2/d_6-DMSO) of compound 3fb



Expanded ^{13}C NMR (100 MHz, CS_2/d_6-DMSO) of compound 3fb



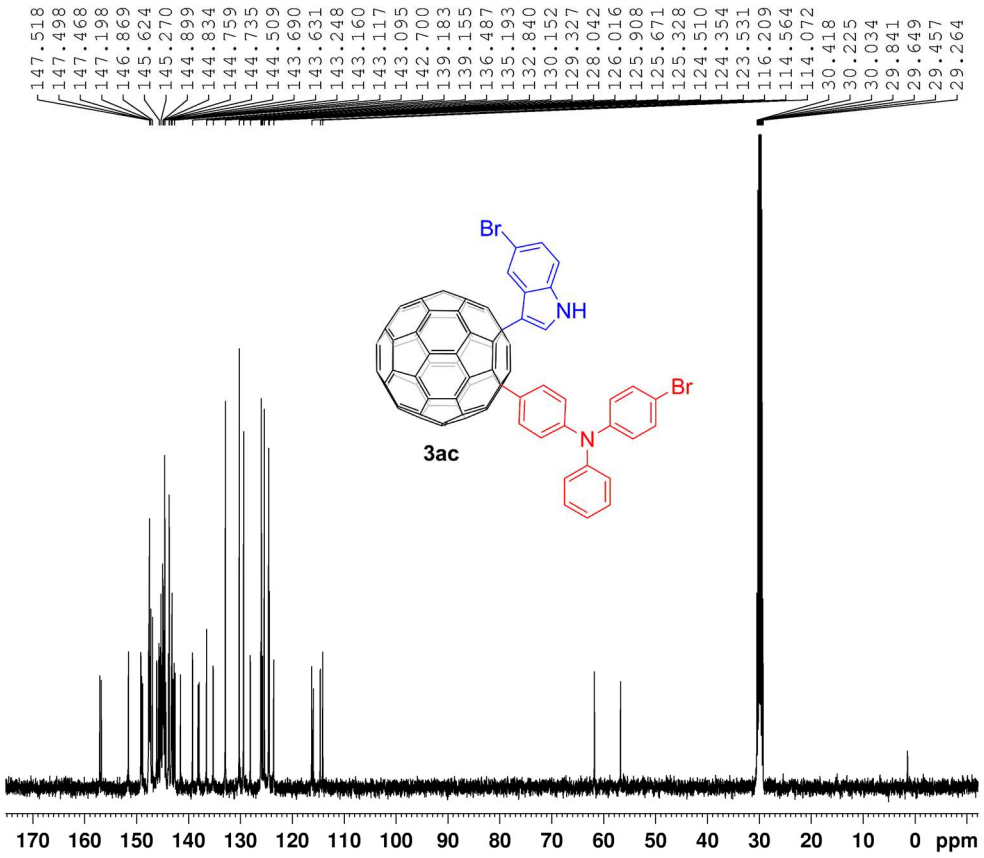
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ac




```
NAME          Dec09-2020
EXPNO                  1
PROCNO                 1
Date_           20201209
Time               19.22
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   362
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W         13.45602226 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300076 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ac

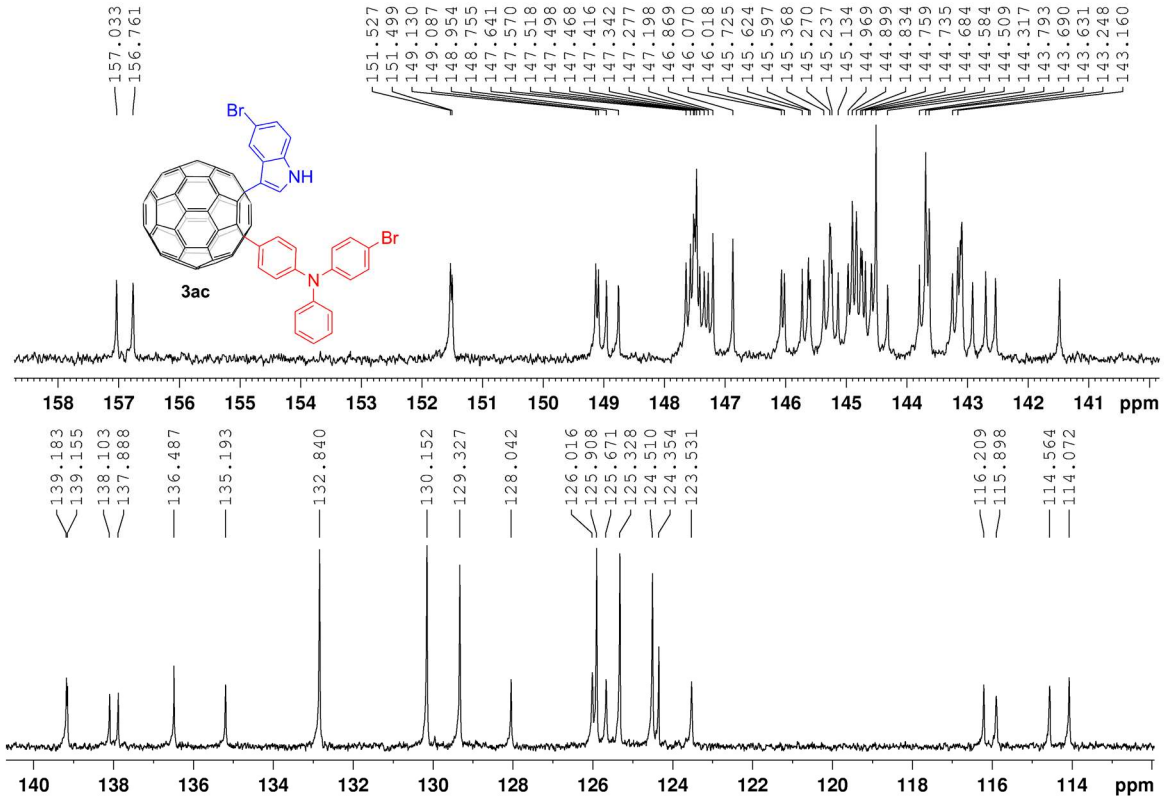



```
NAME          Dec09-2020
EXPNO                  2
PROCNO                 1
Date_           20201209
Time               19.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                 15360
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  80.6
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

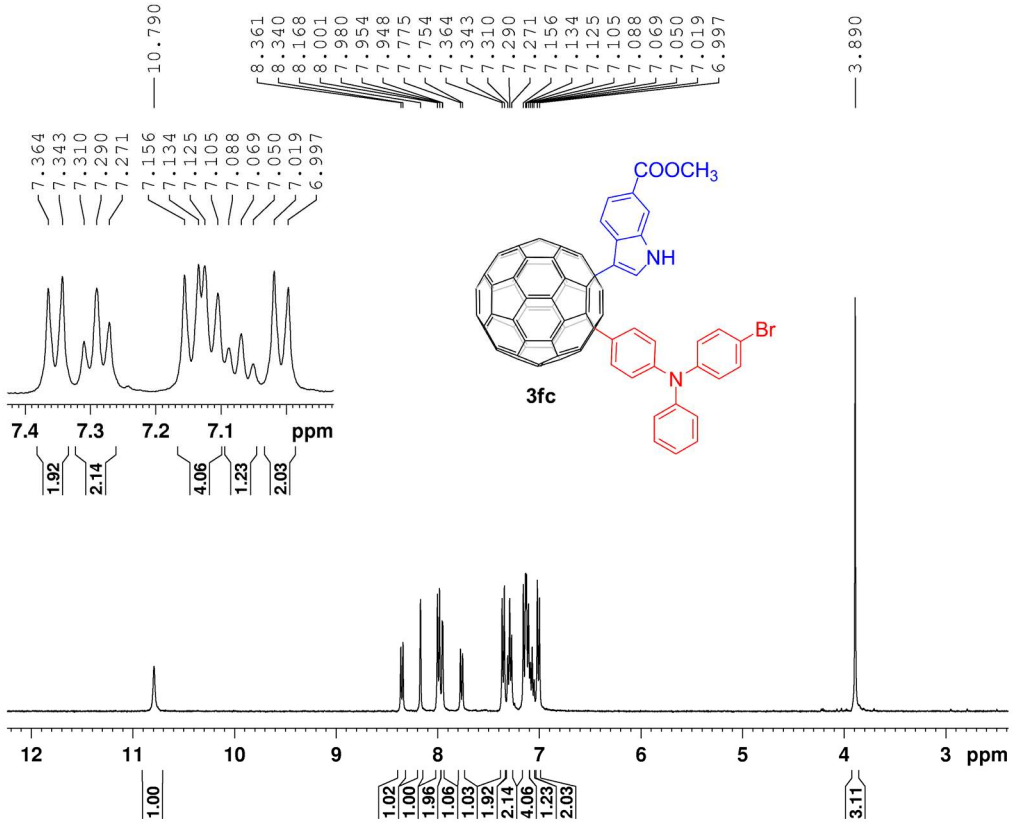
======== CHANNEL f1 ========
NUC1                  13C
P1                  9.30 usec
PL1                -2.00 dB
PL1W         54.14257431 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.46 dB
PL13              120.00 dB
PL2W         13.45602226 W
PL12W         0.38275132 W
PL13W         0.00000000 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127365 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ac



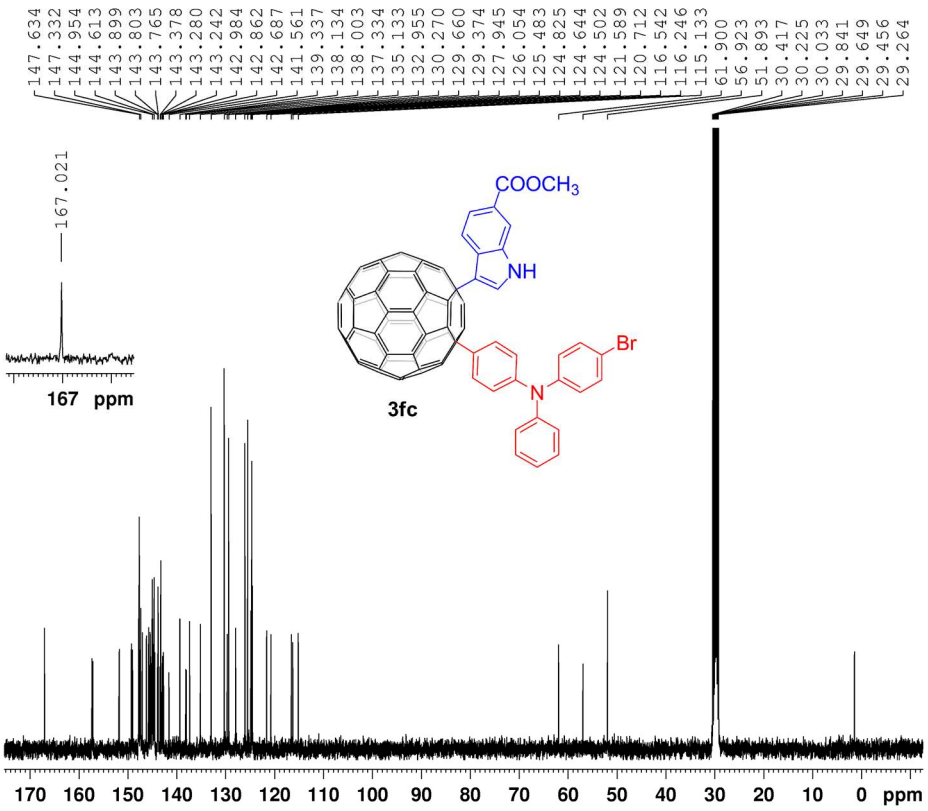
^1H NMR (400 MHz, CS_2/d_6-acetone) of compound 3fc




```
NAME                Jan15-2021
EXPNO                        1
PROCNO                       1
Date_                 20210115
Time                     10.03
INSTRUM                  spect
PROBHD   5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT               Acetone
NS                           16
DS                            2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                          322
DW                   60.800 usec
DE                    6.50 usec
TE                    300.0 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                    13.50 usec
PL1                   -2.00 dB
PL1W             13.45602226 W
SFO1             400.1324710 MHz
SI                       32768
SF               400.1300073 MHz
WDW                        EM
SSB                         0
LB                    0.30 Hz
GB                          0
PC                       1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fc

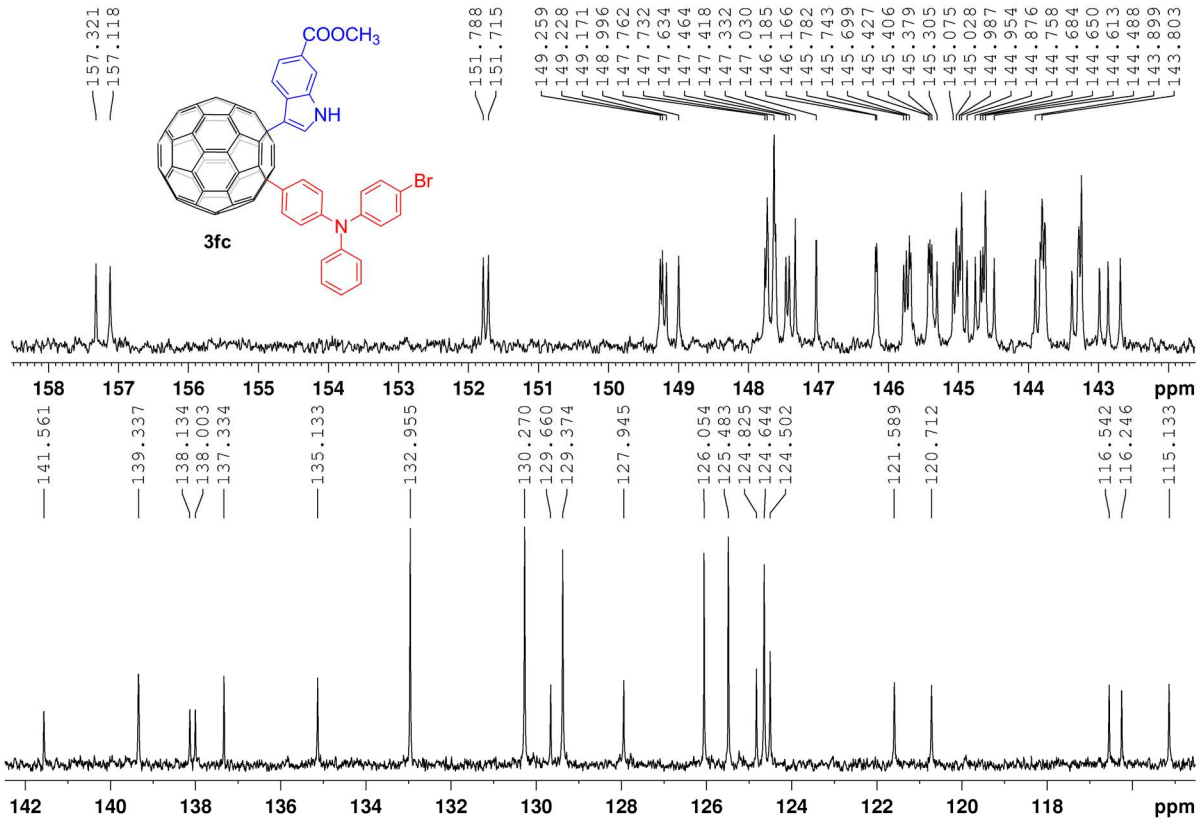



```
NAME            Jan15-2021
EXPNO                    2
PROCNO                   1
Date_             20210115
Time                 16.14
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                    20480
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                      144
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

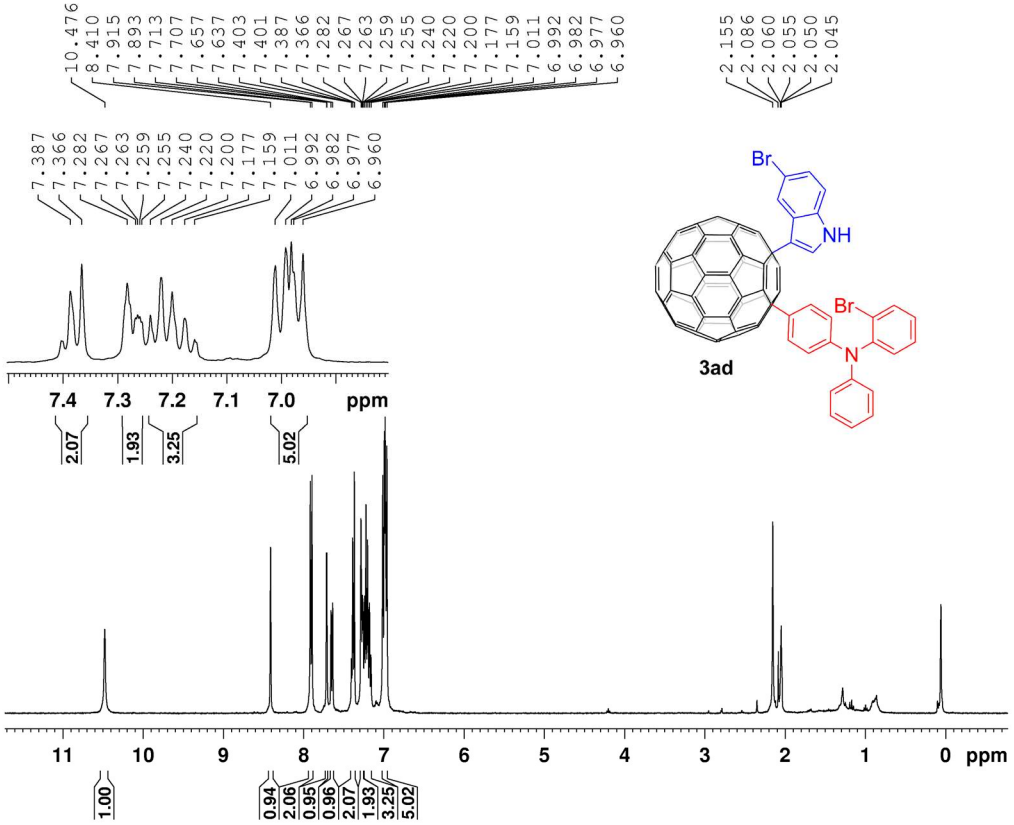
======== CHANNEL f1 ========
NUC1                    13C
P1                    9.30 usec
PL1                  -2.00 dB
PL1W           54.14257431 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 13.46 dB
PL13                120.00 dB
PL2W           13.45602226 W
PL12W           0.38275132 W
PL13W           0.00000000 W
SFO2          400.1316005 MHz
SI                    32768
SF            100.6127244 MHz
WDW                      EM
SSB                       0
LB                 1.00 Hz
GB                        0
PC                    1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fc



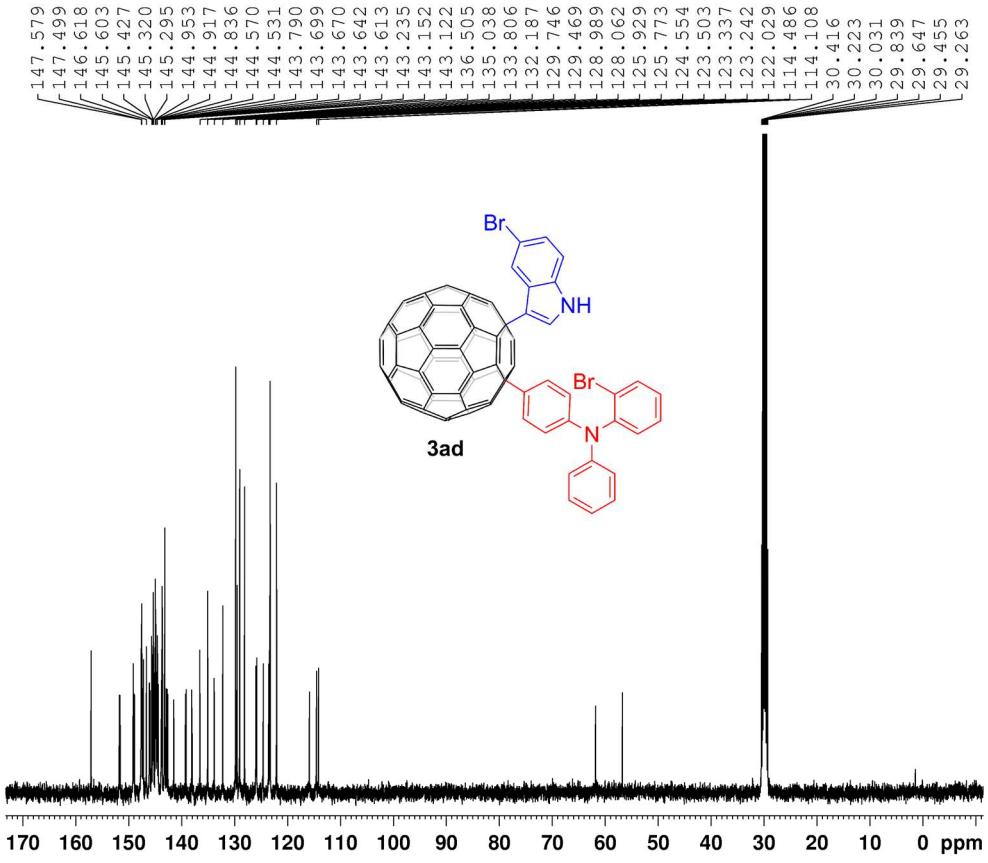
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ad




```
NAME            Dec21-2020
EXPNO                    1
PROCNO                   1
Date_             20201221
Time                 20.03
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT            Acetone
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     362
DW                  60.800 usec
DE                   6.50 usec
TE                  300.0 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.50 usec
PL1                 -2.00 dB
PL1W          13.45602226 W
SFO1          400.1324710 MHz
SI                   32768
SF            400.1300077 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ad

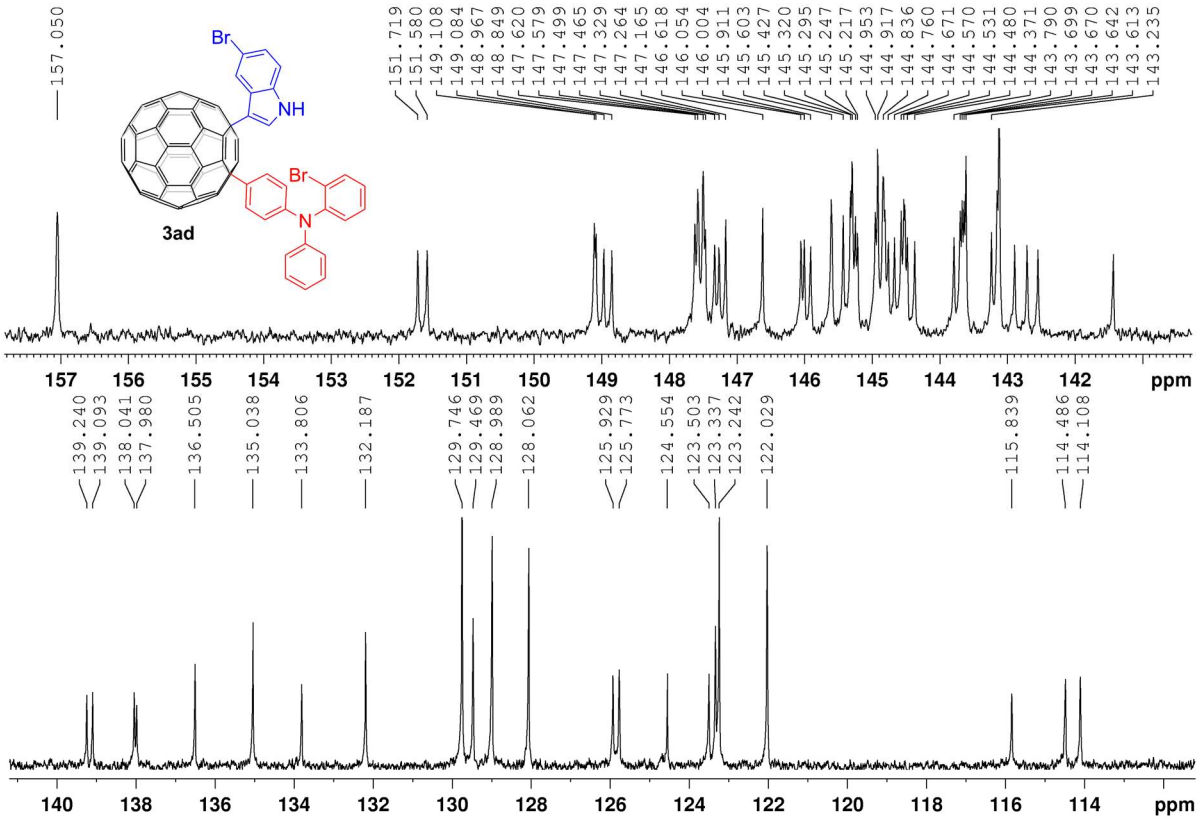



```
NAME            Dec21-2020
EXPNO                    2
PROCNO                   1
Date_             20201221
Time                 20.14
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                   14671
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                      57
DW                  20.800 usec
DE                   6.50 usec
TE                  300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

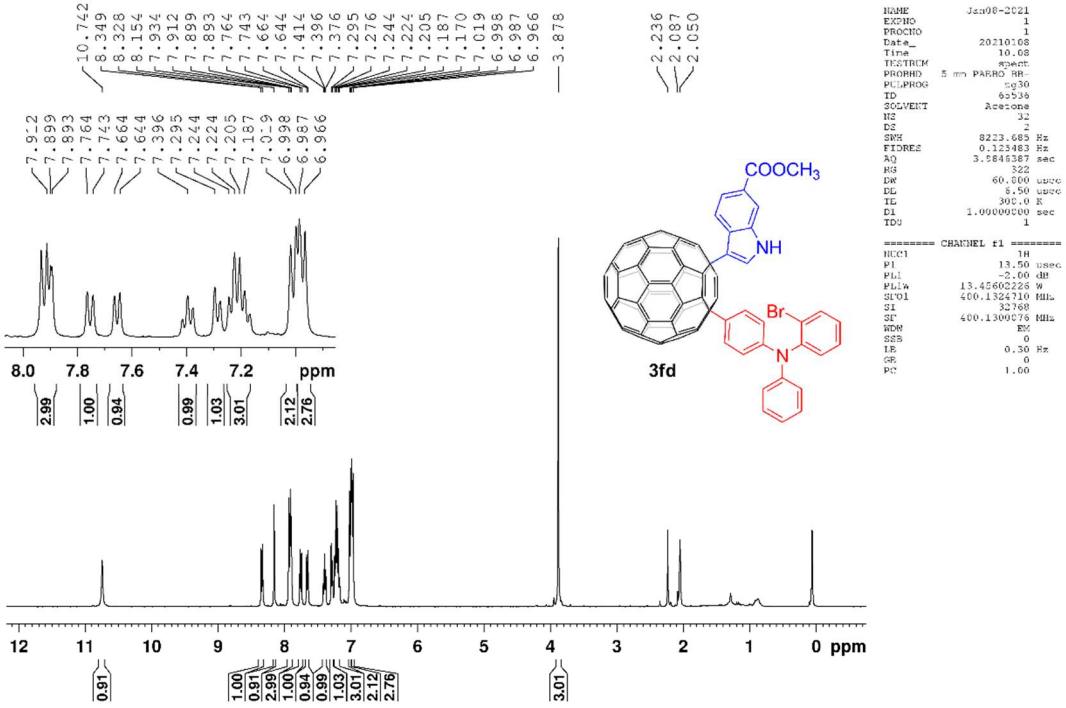
======== CHANNEL f1 ========
NUC1                   13C
P1                   9.30 usec
PL1                 -2.00 dB
PL1W          54.14257431 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -2.00 dB
PL12                13.46 dB
PL13                120.00 dB
PL2W          13.45602226 W
PL12W          0.38275132 W
PL13W          0.00000000 W
SFO2          400.1316005 MHz
SI                   32768
SF            100.6127362 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

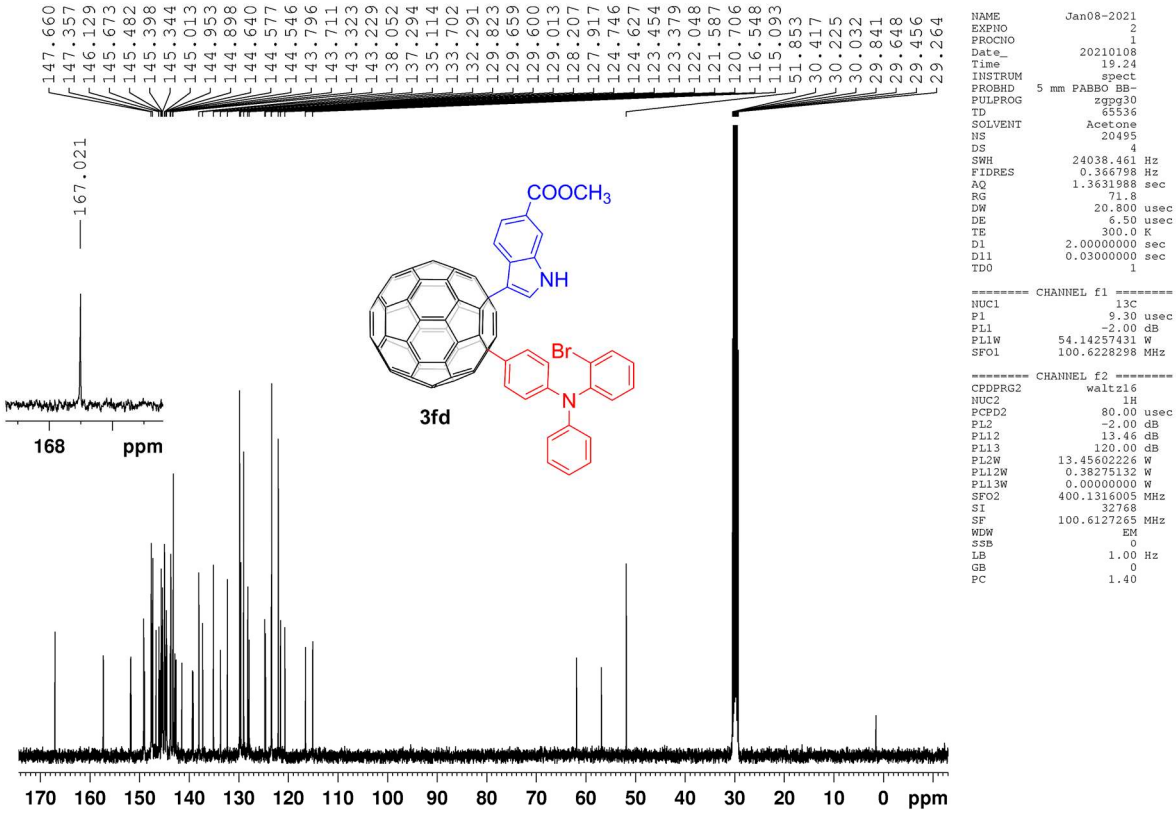
Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ad



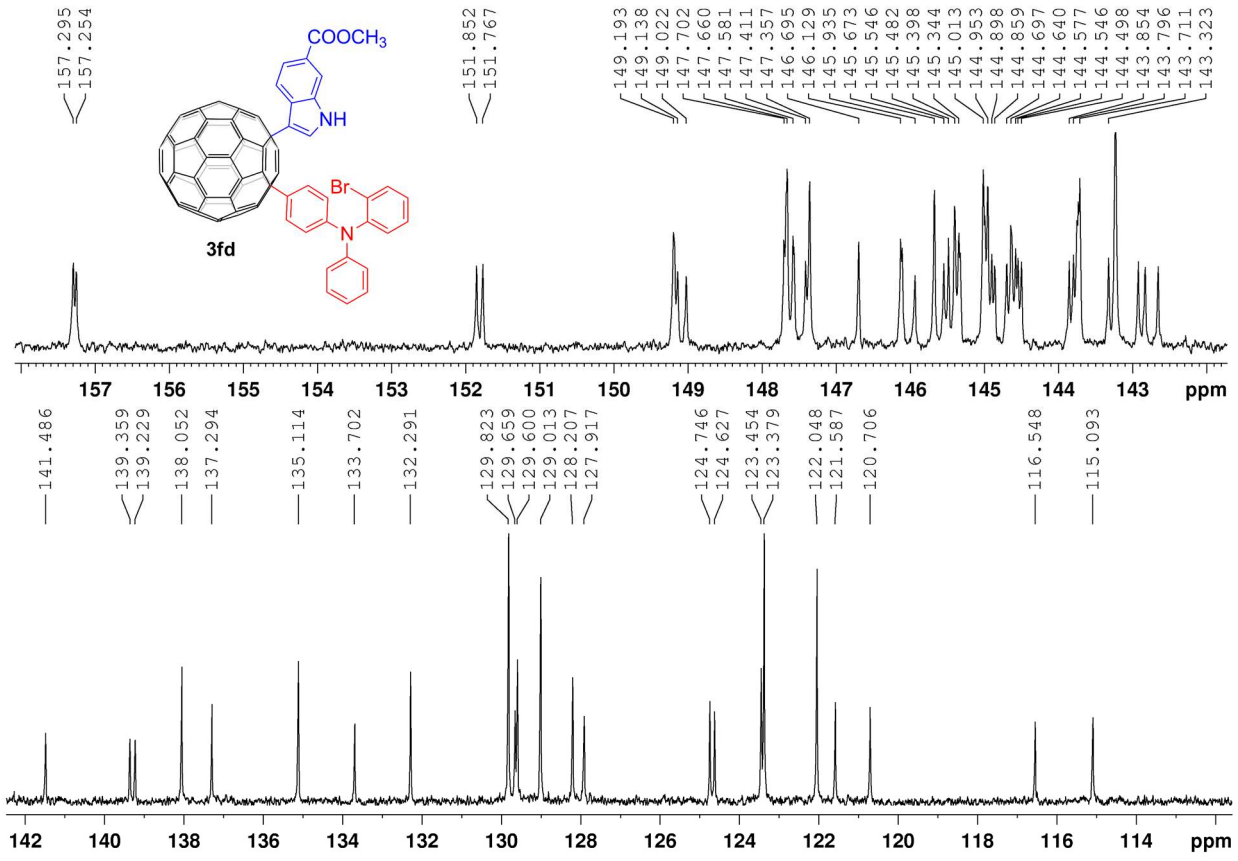
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3fd



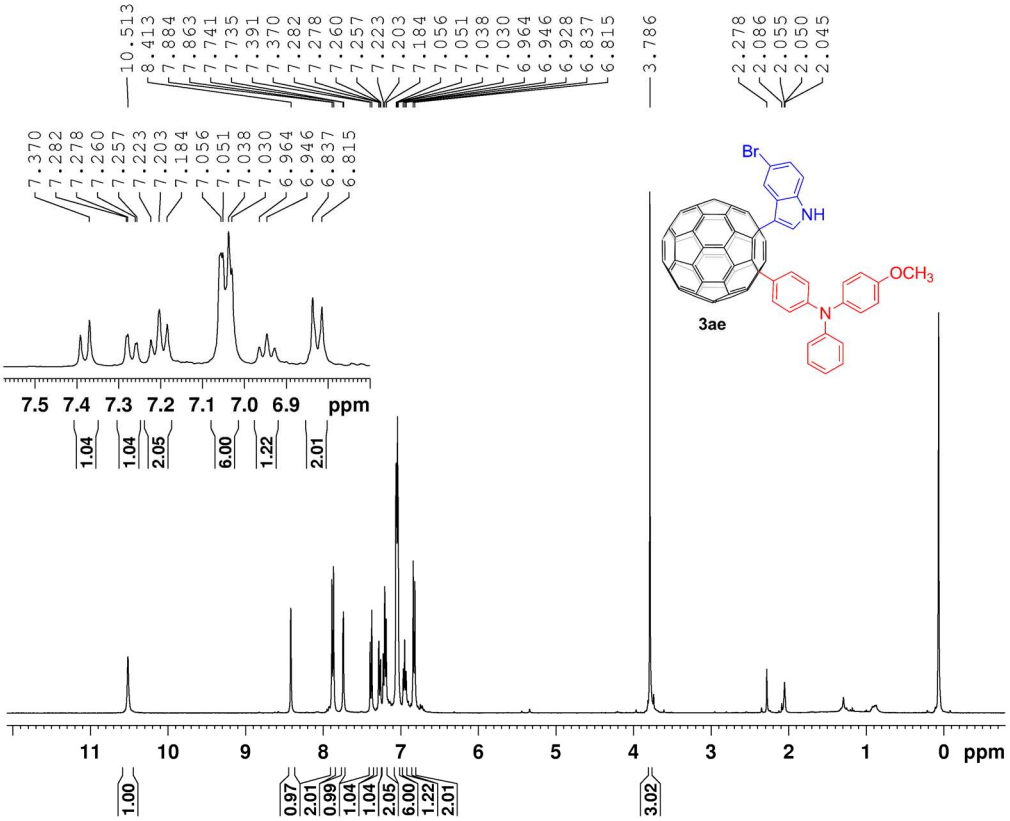
^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fd



Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fd



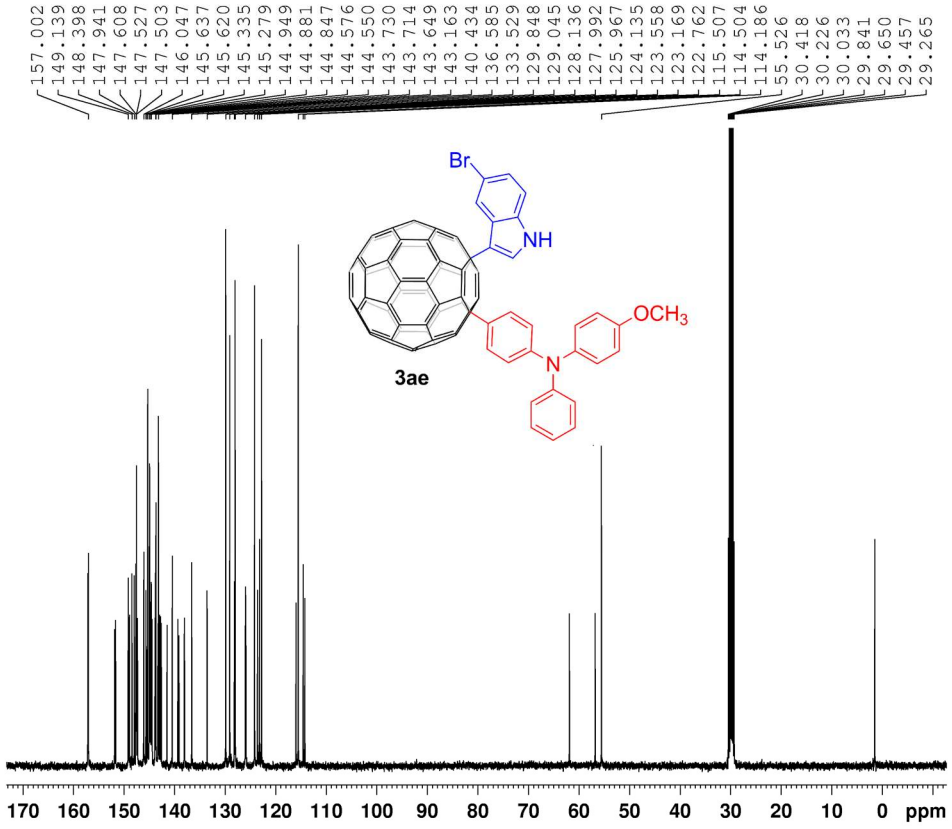
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ae




```
NAME          Dec31-2020
EXPNO                  2
PROCNO                 1
Date_           20201231
Time               10.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   256
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 1H
P1                13.50 usec
PL1               -2.00 dB
PL1W        13.45602226 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300078 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ae

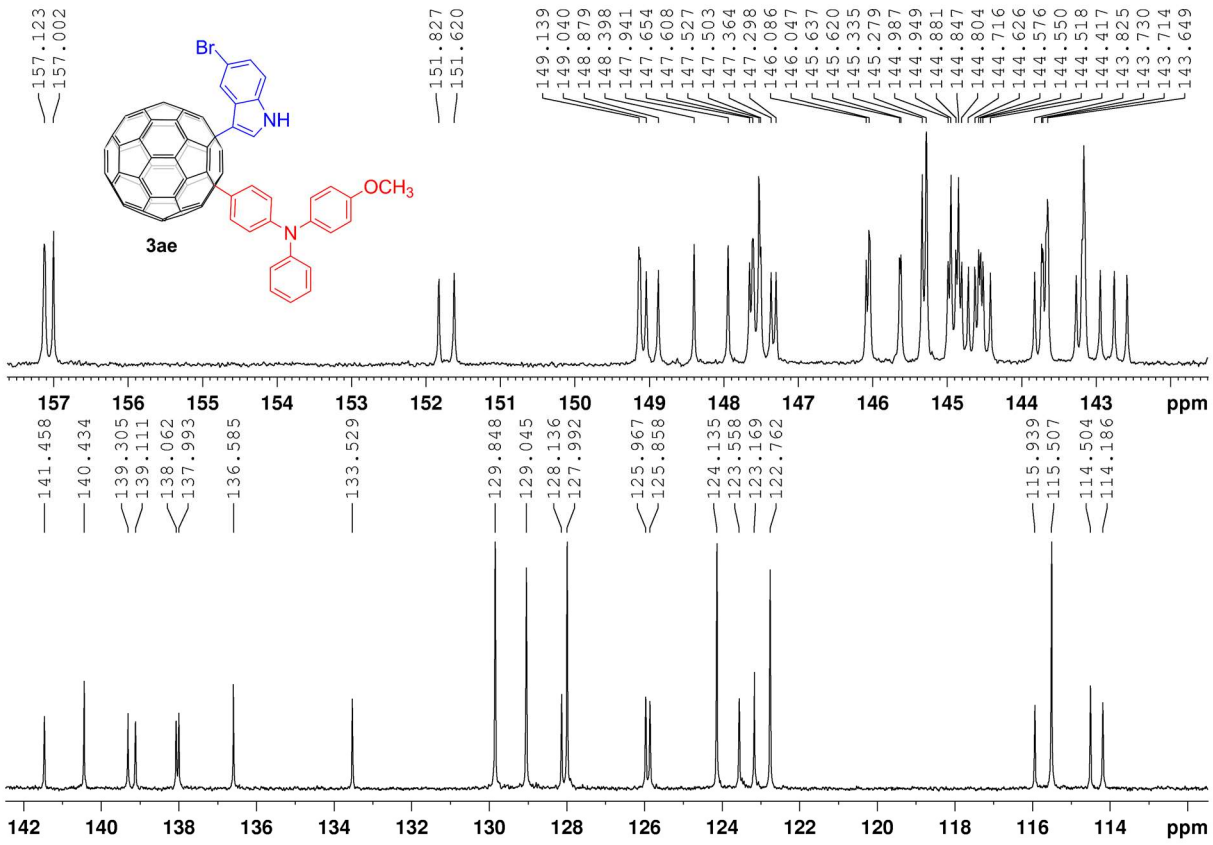



```
NAME          Dec31-2020
EXPNO                  3
PROCNO                 1
Date_           20201231
Time               17.36
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                 20480
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                    64
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

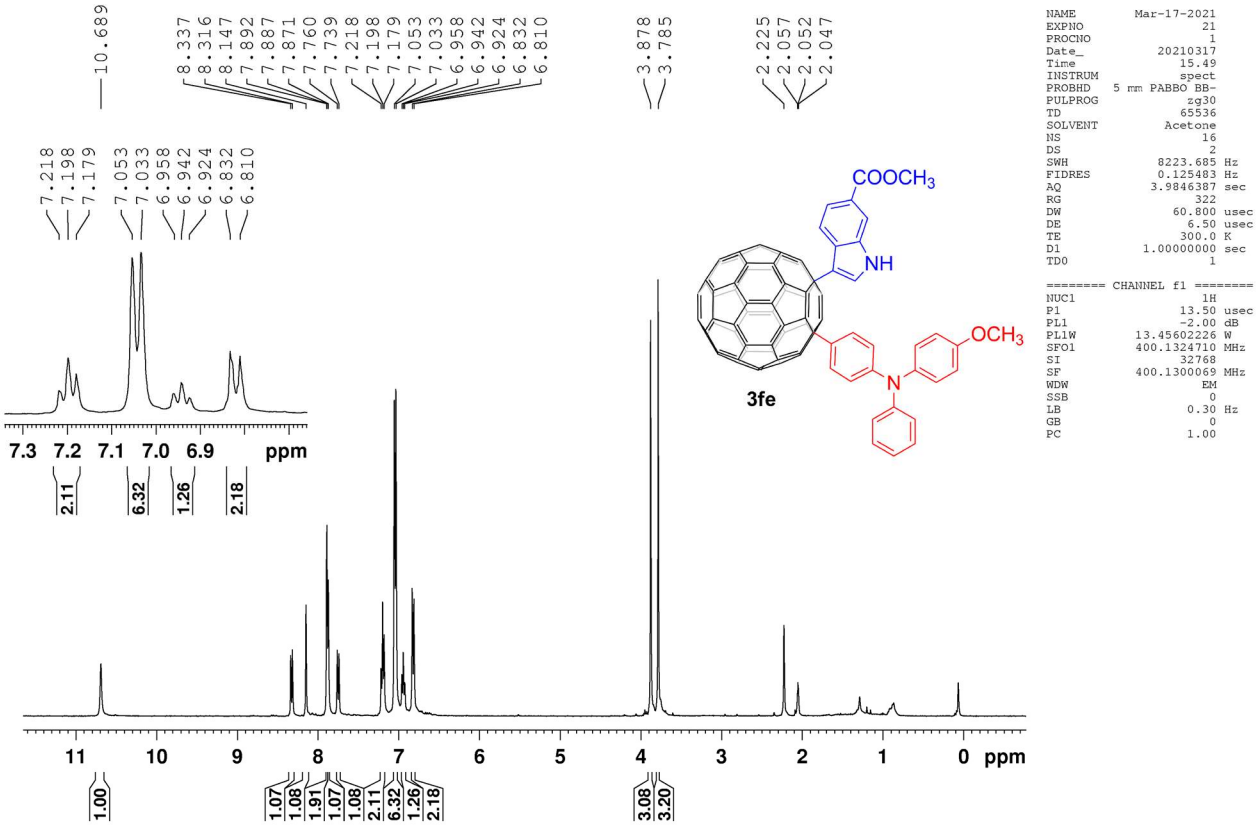
======== CHANNEL f1 ========
NUC1                13C
P1                 9.30 usec
PL1               -2.00 dB
PL1W        54.14257431 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              13.46 dB
PL13             120.00 dB
PL2W        13.45602226 W
PL12W        0.38275132 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127295 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ae



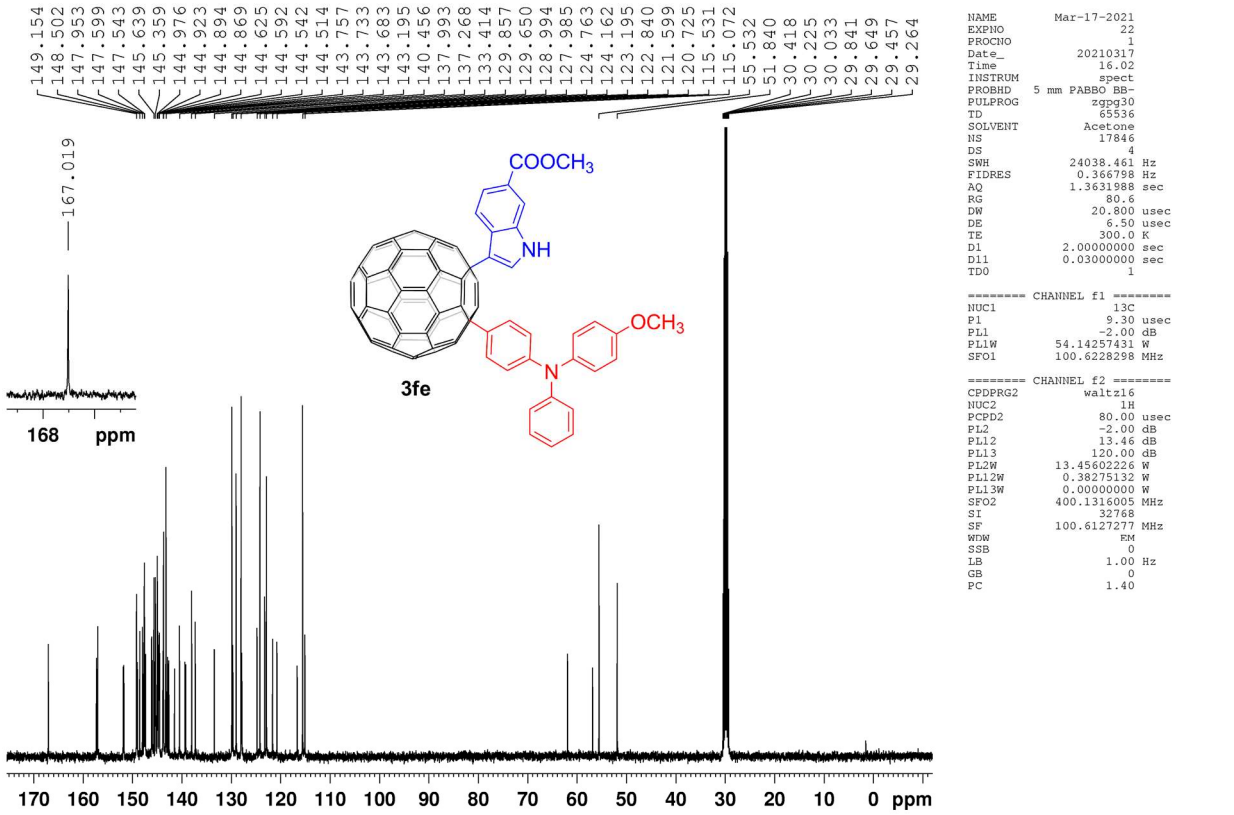
^1H NMR (400 MHz, CS_2/d_6-acetone) of compound 3fe



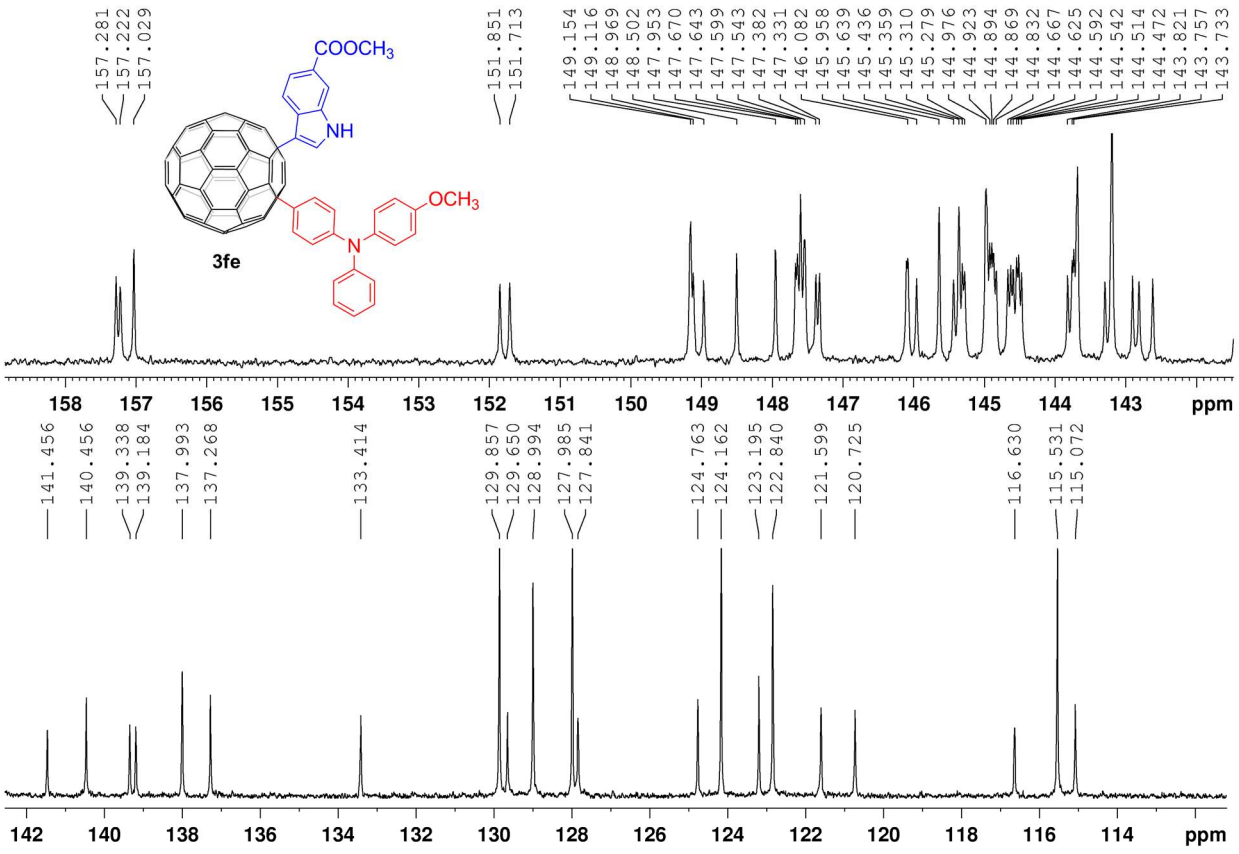

```
NAME            Mar-17-2021
EXPNO                    21
PROCNO                    1
Date_              20210317
Time                  15.49
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT             Acetone
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      322
DW                   60.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   13.50 usec
PL1                  -2.00 dB
PL1W            13.45602226 W
SFO1            400.1324710 MHz
SI                    32768
SF              400.1300069 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```

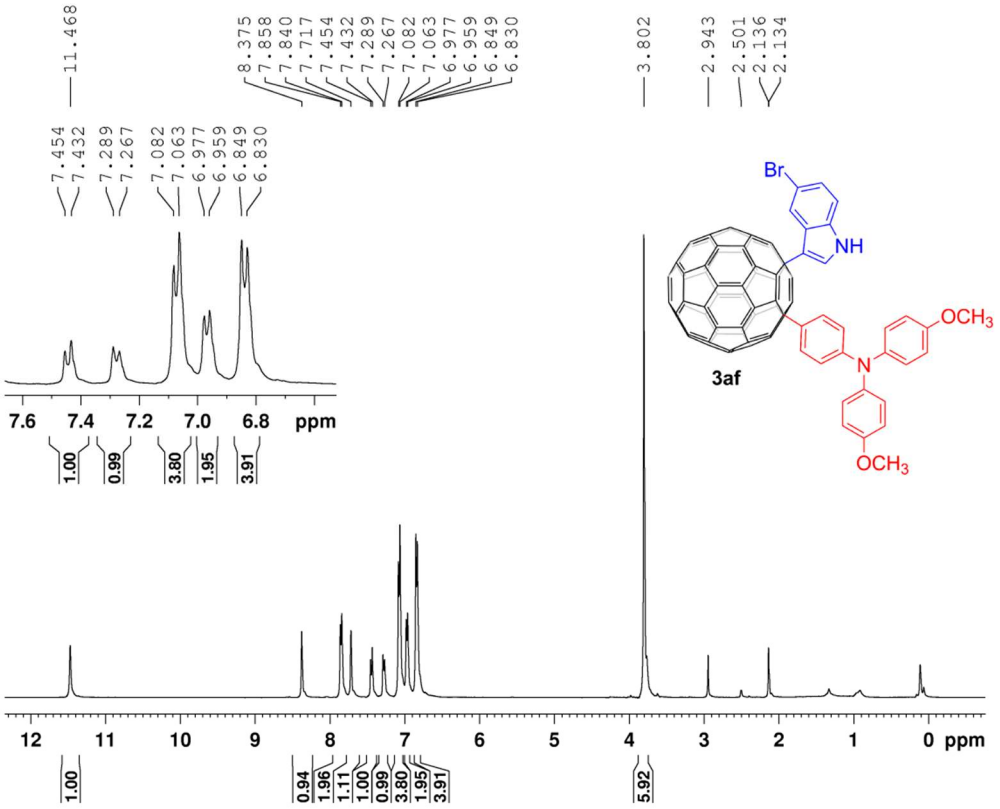
^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fe



Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fe



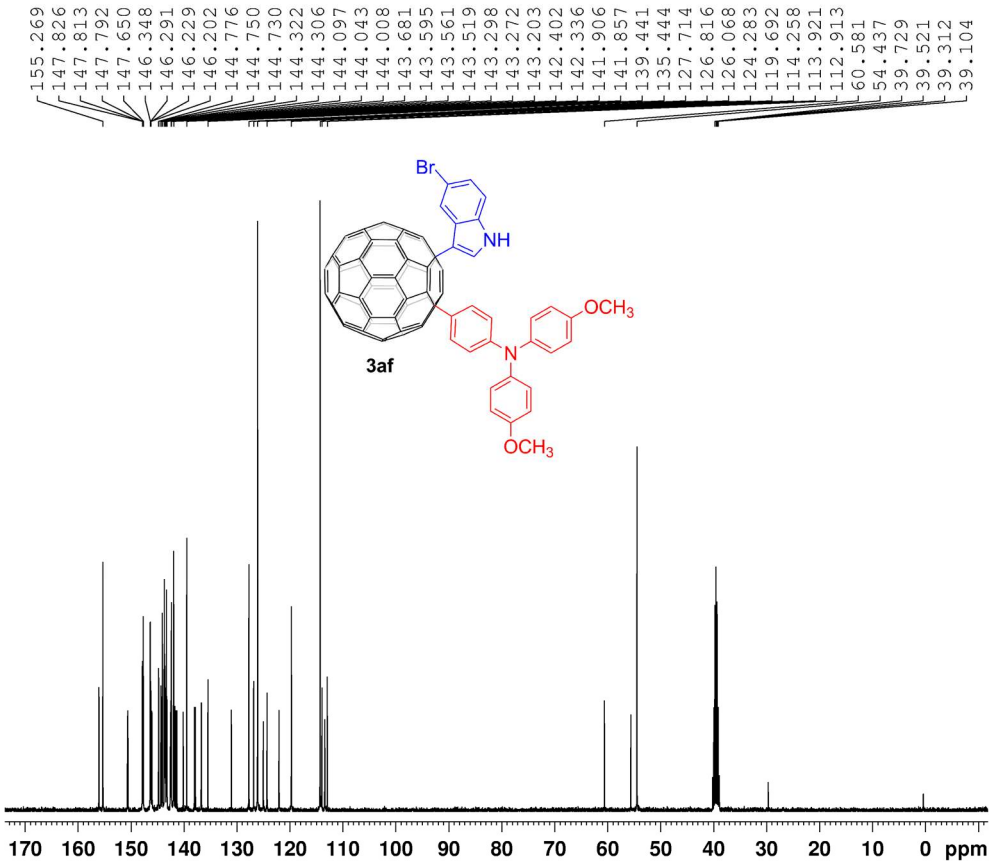
^{1}H NMR (400 MHz, CS_2/d_6-DMSO) of compound 3af




```
NAME          Dec22-2020
EXPNO                  3
PROCNO                 1
Date_           20201222
Time               11.23
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   228
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.50 usec
PL1               -2.00 dB
PL1W         13.45602226 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300034 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-DMSO) of compound 3af

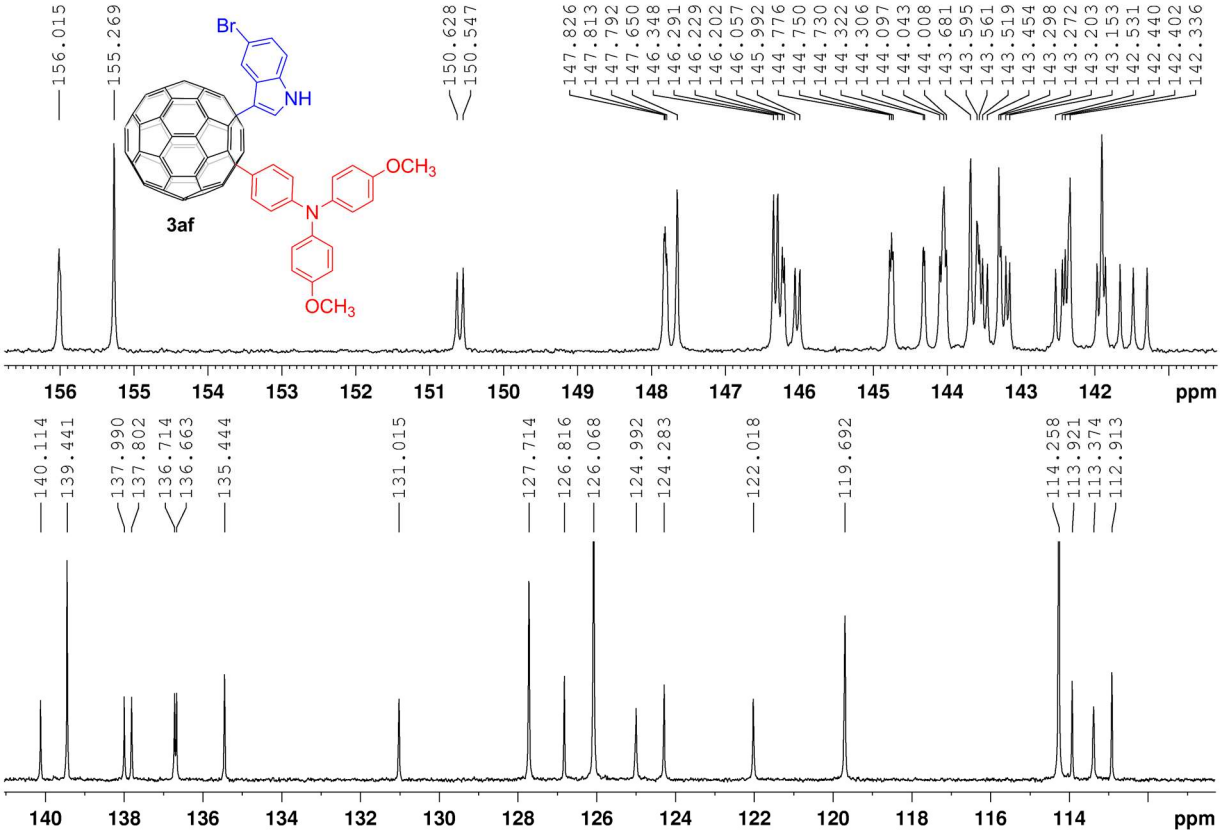



```
NAME          Dec22-2020
EXPNO                  4
PROCNO                 1
Date_           20201222
Time               18.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                 16094
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  80.6
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

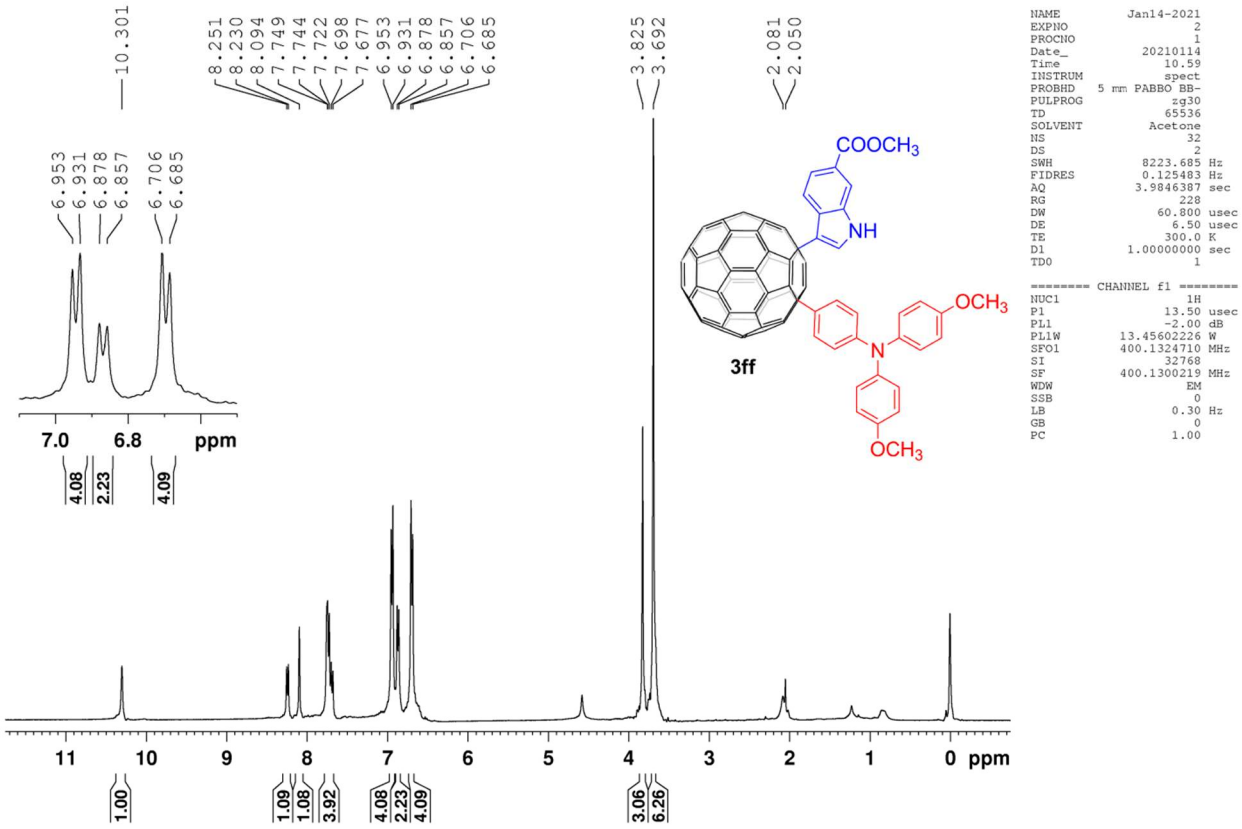
======== CHANNEL f1 ========
NUC1                 13C
P1                 9.30 usec
PL1               -2.00 dB
PL1W         54.14257431 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              13.46 dB
PL13             120.00 dB
PL2W         13.45602226 W
PL12W         0.38275132 W
PL13W         0.00000000 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6128617 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-DMSO) of compound 3af



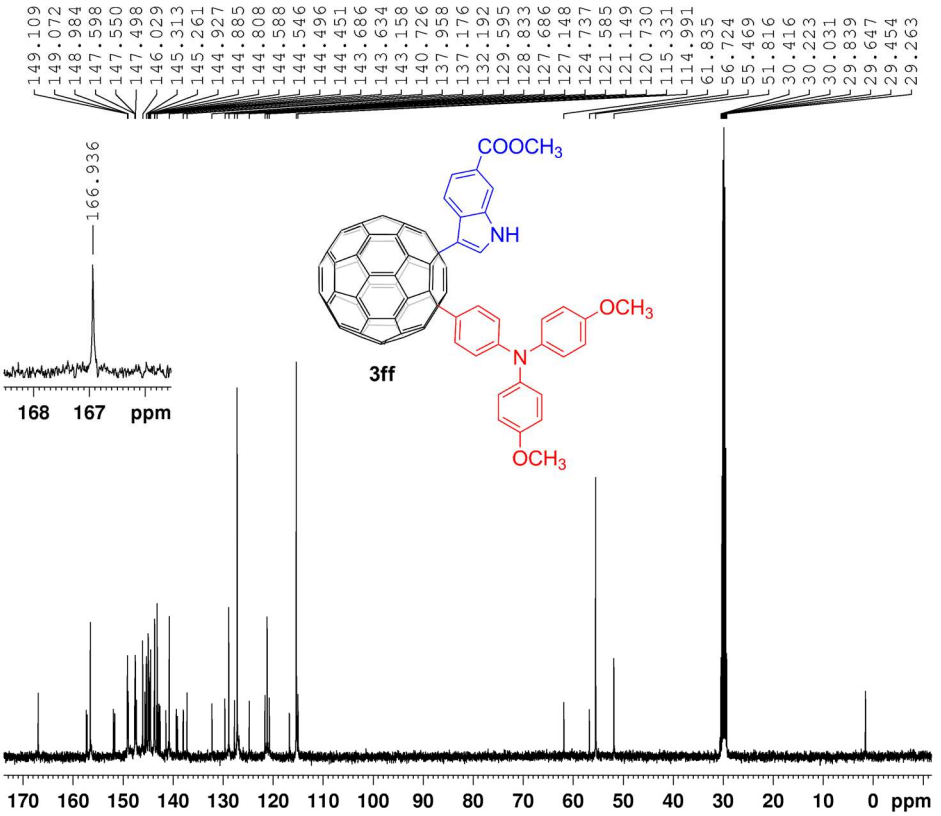
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ff




```
NAME            Jan14-2021
EXPNO                    2
PROCNO                   1
Date_             20210114
Time                 10.59
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                      32
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     228
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W         13.45602226 W
SFO1         400.1324710 MHz
SI                   32768
SF           400.1300219 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ff

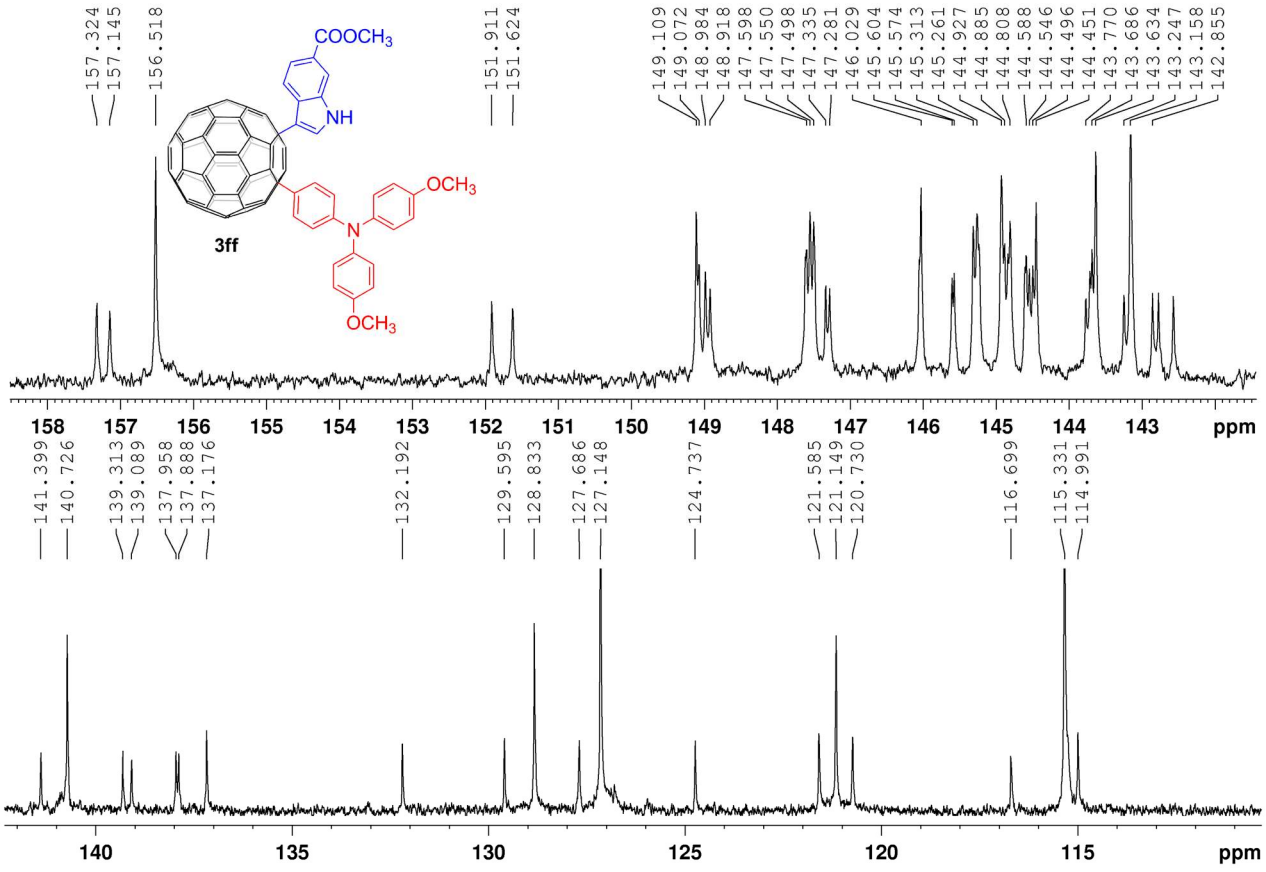



```
NAME            Jan19-2021
EXPNO                    1
PROCNO                   1
Date_             20210119
Time                 17.29
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                    20480
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                     71.8
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

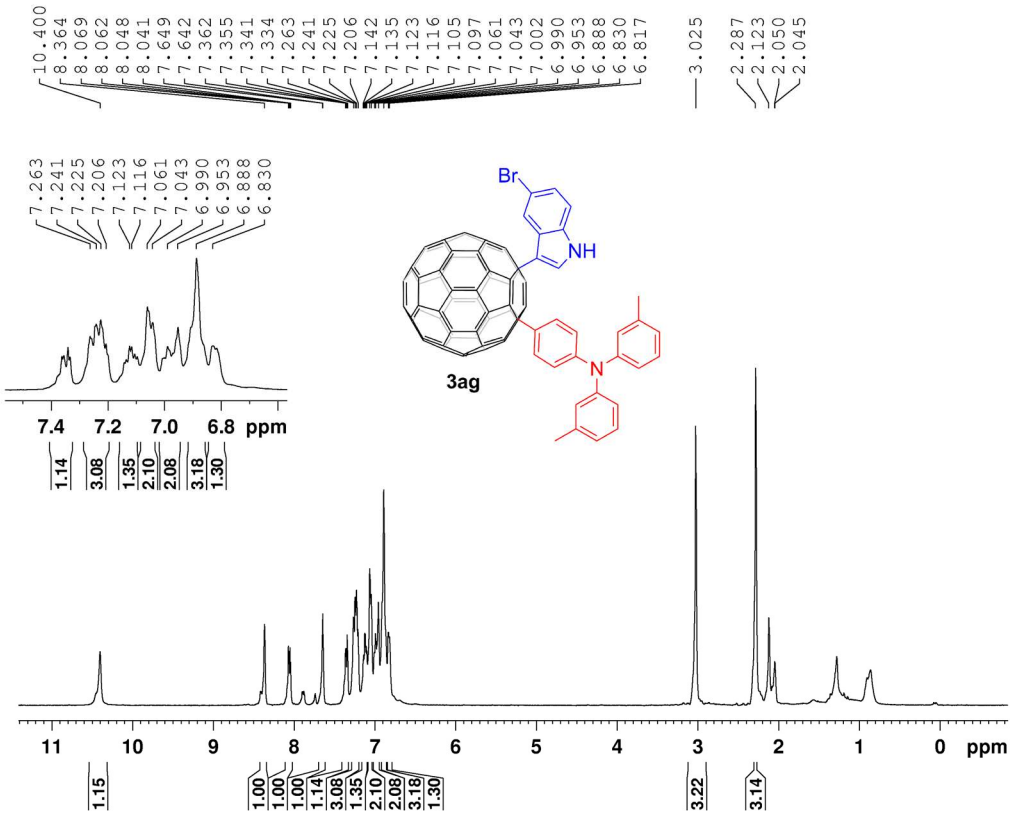
======== CHANNEL f1 ========
NUC1                    13C
P1                     9.30 usec
PL1                   -2.00 dB
PL1W             54.14257431 W
SFO1            100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                   -2.00 dB
PL12                  13.46 dB
PL13                 120.00 dB
PL2W             13.45602226 W
PL12W             0.38275132 W
PL13W             0.00000000 W
SFO2            400.1316005 MHz
SI                     32768
SF              100.6127318 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ff



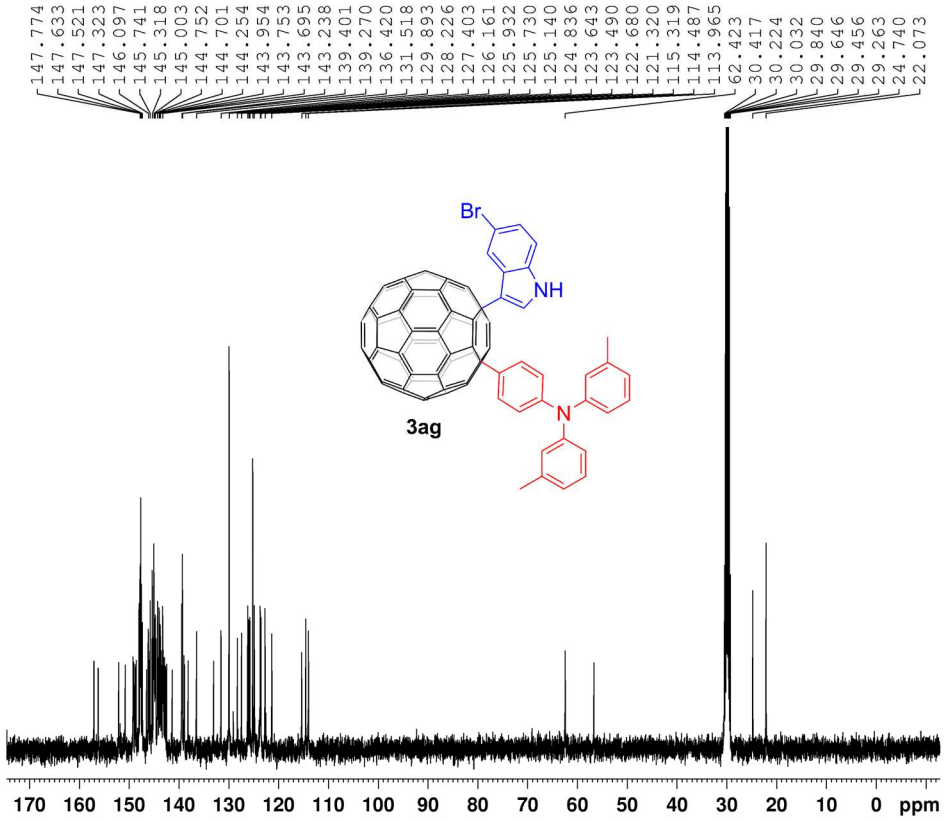
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ag




```
NAME            Apr19-2021
EXPNO                    1
PROCNO                   1
Date_             20210419
Time                 11.06
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT            Acetone
NS                       16
DS                        2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      287
DW                   60.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.50 usec
PL1                  -2.00 dB
PL1W            13.45602226 W
SFO1           400.1324710 MHz
SI                    32768
SF             400.1300078 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ag

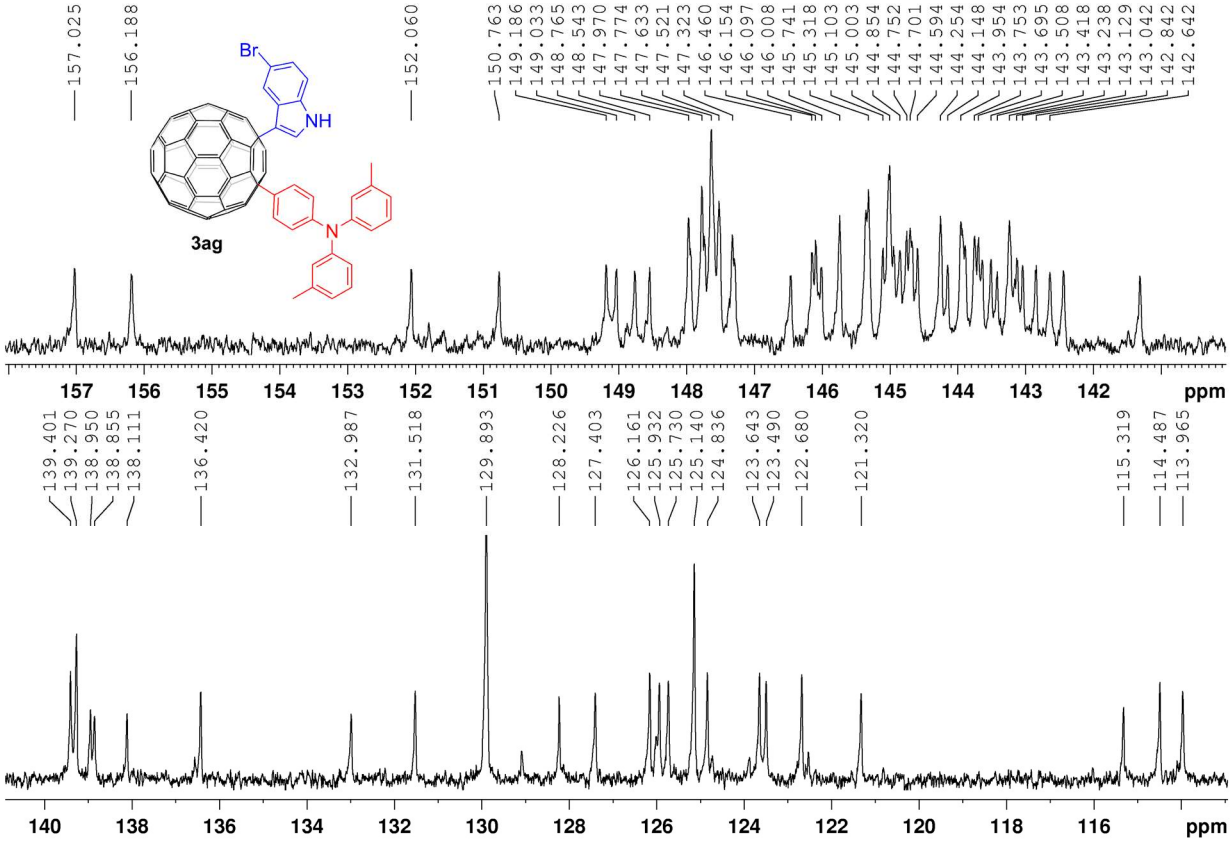



```
NAME            Apr19-2021
EXPNO                    2
PROCNO                   1
Date_             20210419
Time                 17.26
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                   10240
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                     80.6
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

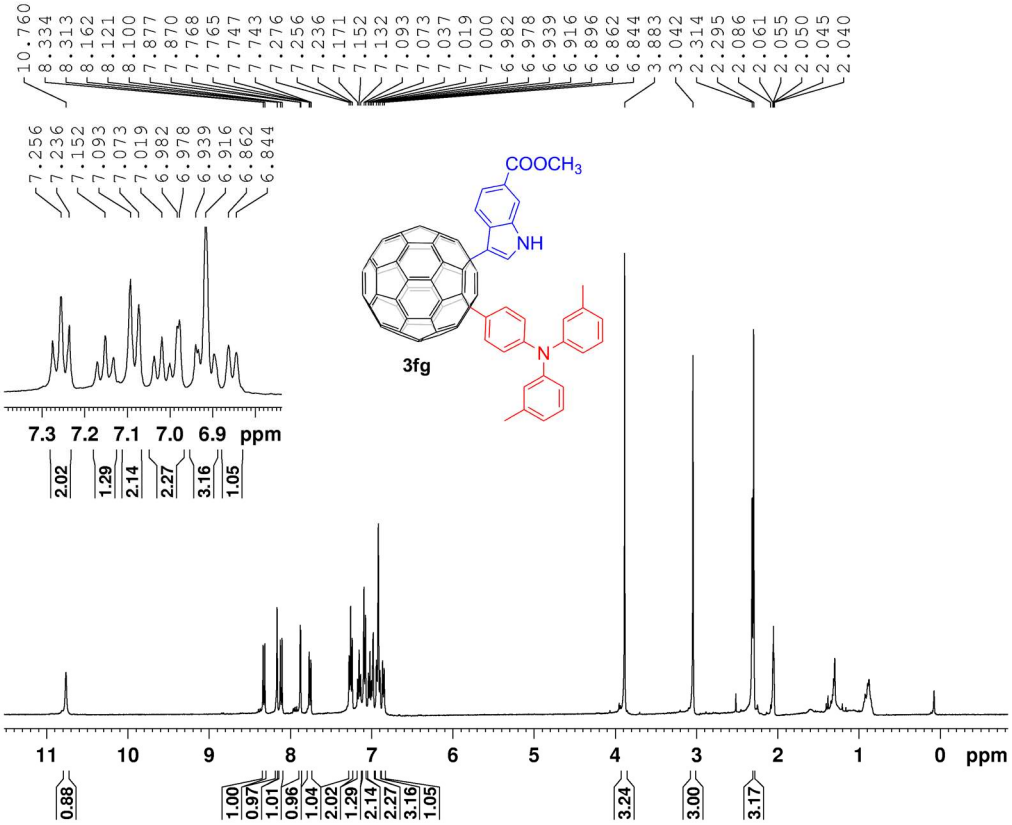
======== CHANNEL f1 ========
NUC1                   13C
P1                    9.30 usec
PL1                  -2.00 dB
PL1W            54.14257431 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 13.46 dB
PL13                120.00 dB
PL2W            13.45602226 W
PL12W            0.38275132 W
PL13W            0.00000000 W
SFO2           400.1316005 MHz
SI                    32768
SF             100.6127296 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ag



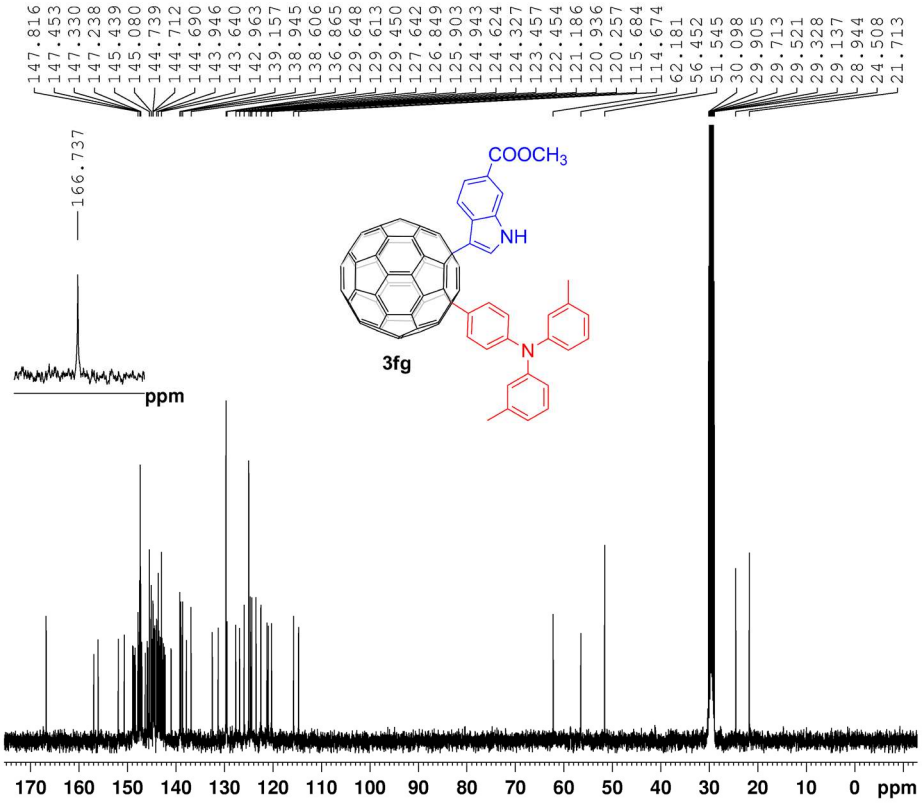
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3fg




```
NAME            Apr13-2021
EXPNO                    1
PROCNO                   1
Date_             20210413
Time                 10.22
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                      16
DS                       2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                     456
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W         13.45602226 W
SFO1         400.1324710 MHz
SI                   32768
SF           400.1300076 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fg

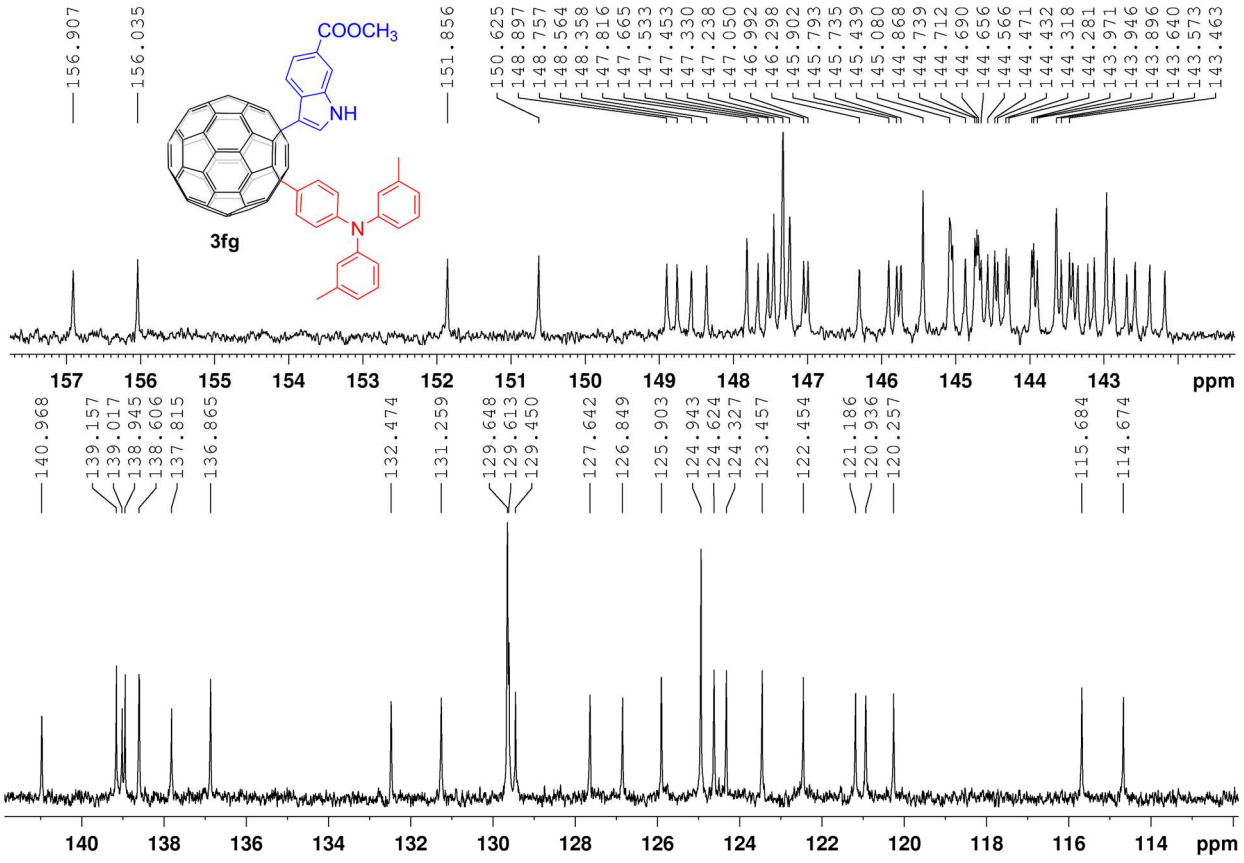



```
NAME            Apr13-2021
EXPNO                    2
PROCNO                   1
Date_             20210413
Time                 16.24
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                 10240
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  80.6
DW                 20.800 usec
DE                  6.50 usec
TE                 300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

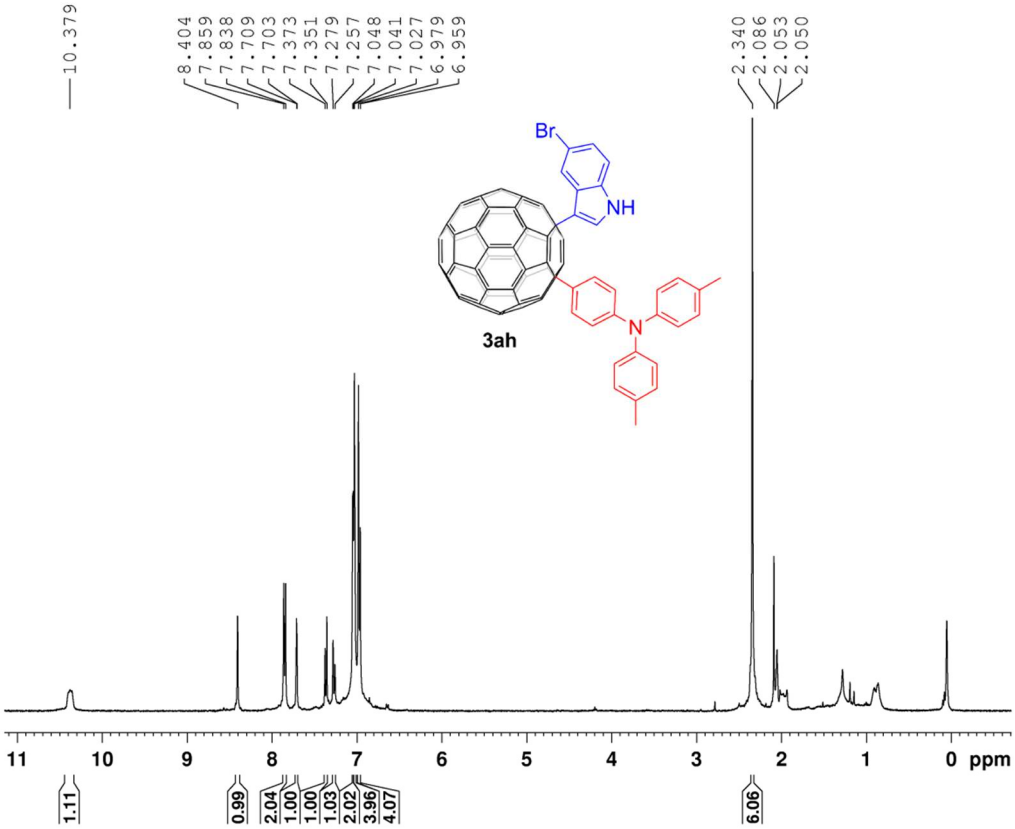
======== CHANNEL f1 ========
NUC1                 13C
P1                  9.30 usec
PL1                -2.00 dB
PL1W         54.14257431 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.46 dB
PL13              120.00 dB
PL2W         13.45602226 W
PL12W         0.38275132 W
PL13W         0.00000000 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127614 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fg



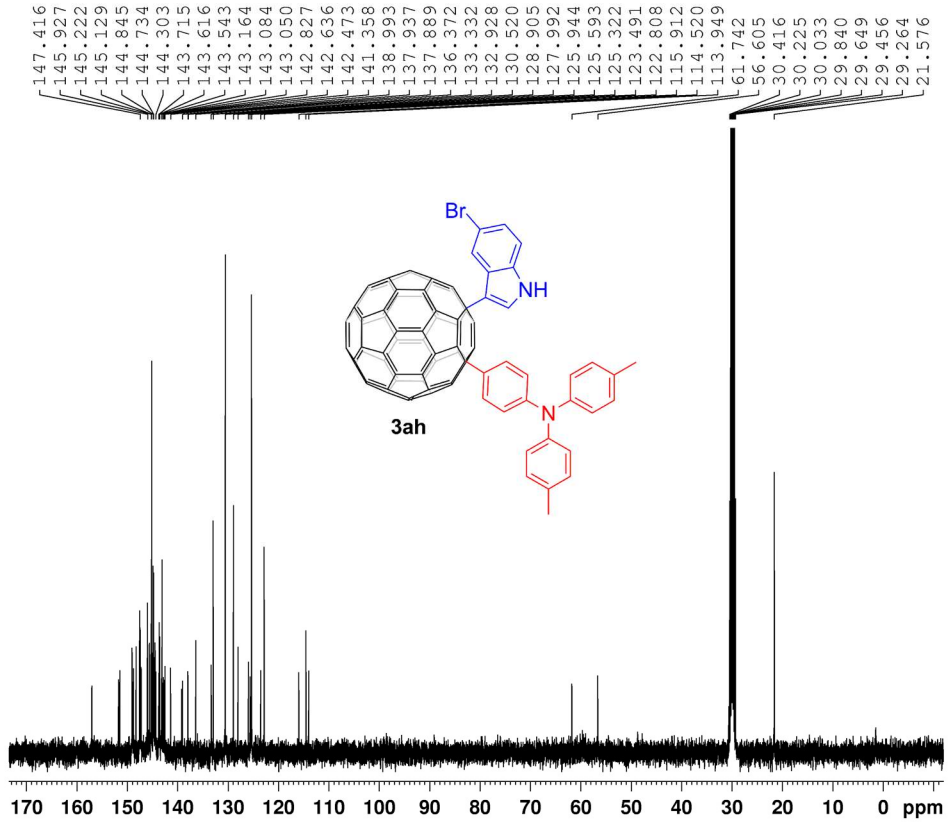
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ah




```
NAME          Dec16-2020
EXPNO                  1
PROCNO                 1
Date_           20201216
Time               15.39
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   362
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W          13.45602226 W
SFO1          400.1324710 MHz
SI                 32768
SF            400.1300076 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ah

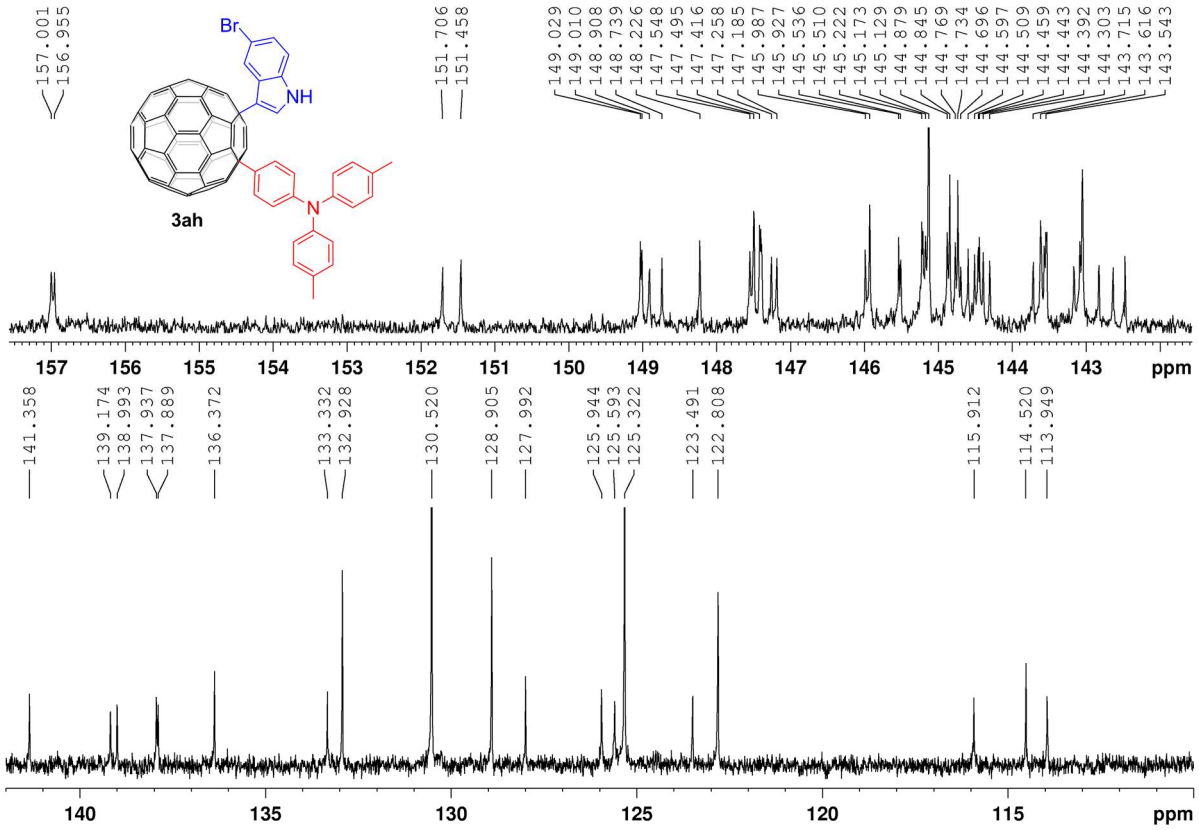



```
NAME          Dec16-2020
EXPNO                  2
PROCNO                 1
Date_           20201216
Time               16.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                 15360
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                    64
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

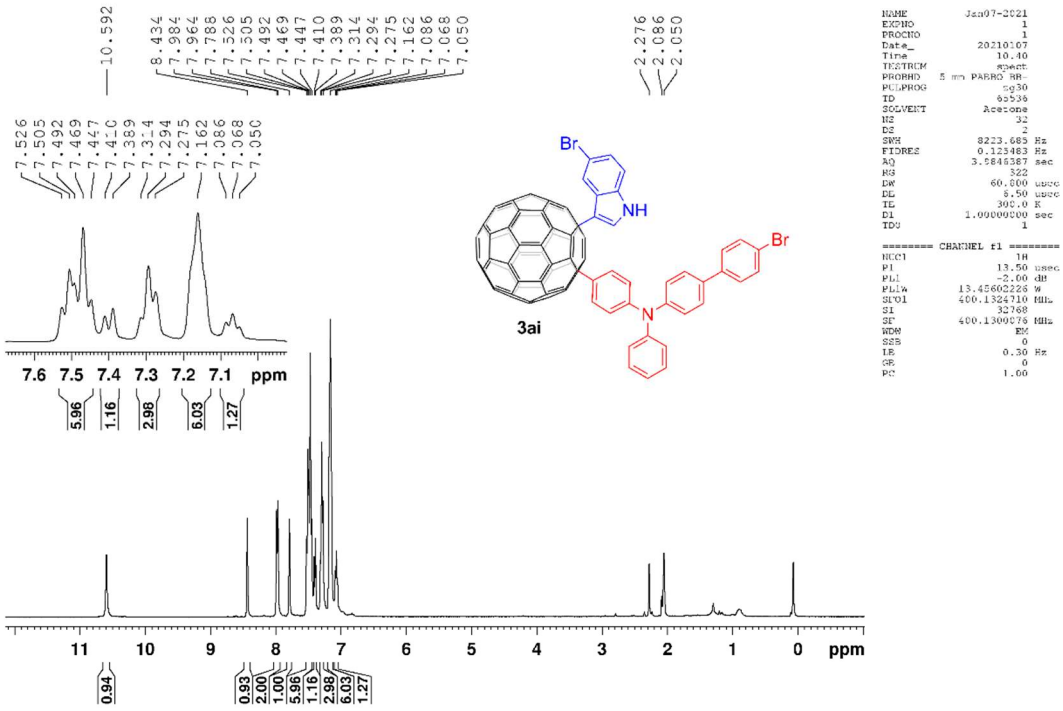
======== CHANNEL f1 ========
NUC1                  13C
P1                  9.30 usec
PL1                -2.00 dB
PL1W          54.14257431 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.46 dB
PL13              120.00 dB
PL2W          13.45602226 W
PL12W          0.38275132 W
PL13W          0.00000000 W
SFO2          400.1316005 MHz
SI                 32768
SF            100.6127445 MHz
WDW                   EM
SSB                    0
LB                  0.50 Hz
GB                     0
PC                  1.40
```

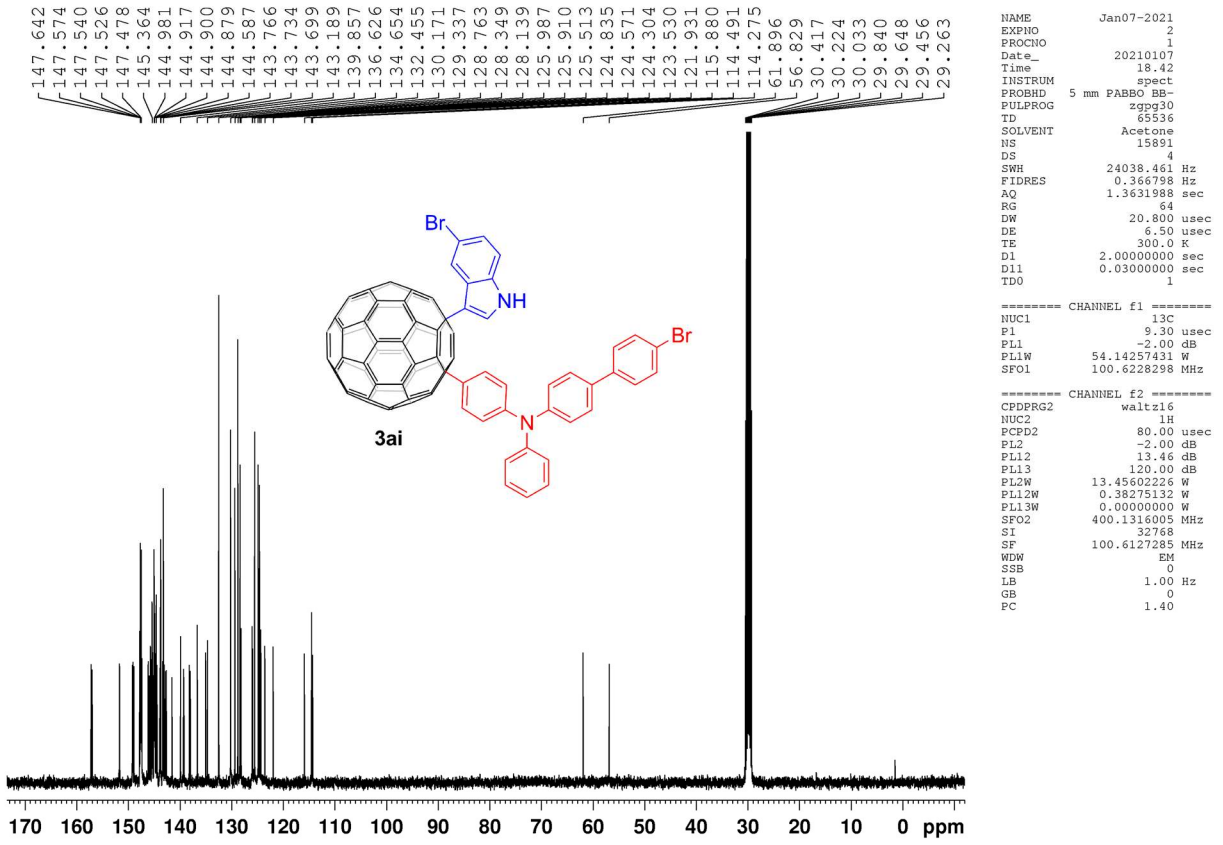
Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ah



^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3ai



^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ai

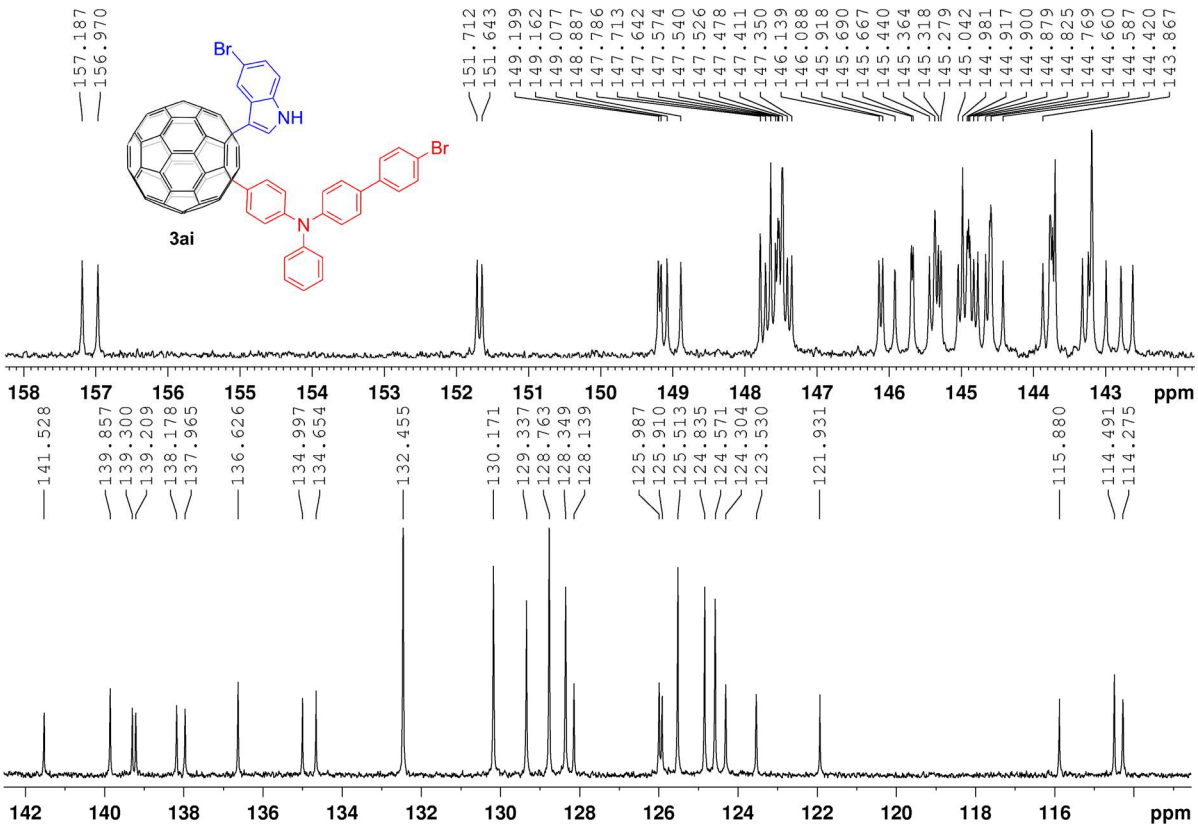



```
NAME            Jan07-2021
EXPNO                    2
PROCNO                   1
Date_             20210107
Time                18.42
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                65536
SOLVENT         Acetone
NS                15891
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                   64
DW               20.800 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

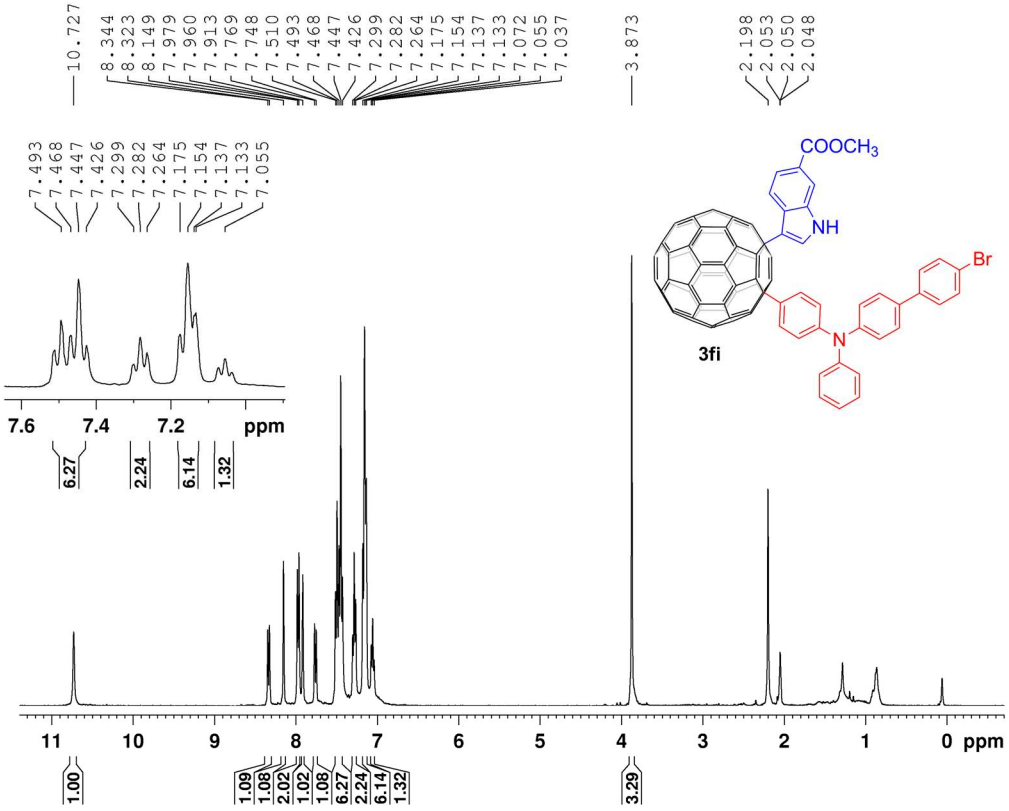
======== CHANNEL f1 ========
NUC1                13C
P1                9.30 usec
PL1              -2.00 dB
PL1W        54.14257431 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -2.00 dB
PL12             13.46 dB
PL13            120.00 dB
PL2W        13.45602226 W
PL12W        0.38275132 W
PL13W        0.00000000 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127285 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3ai



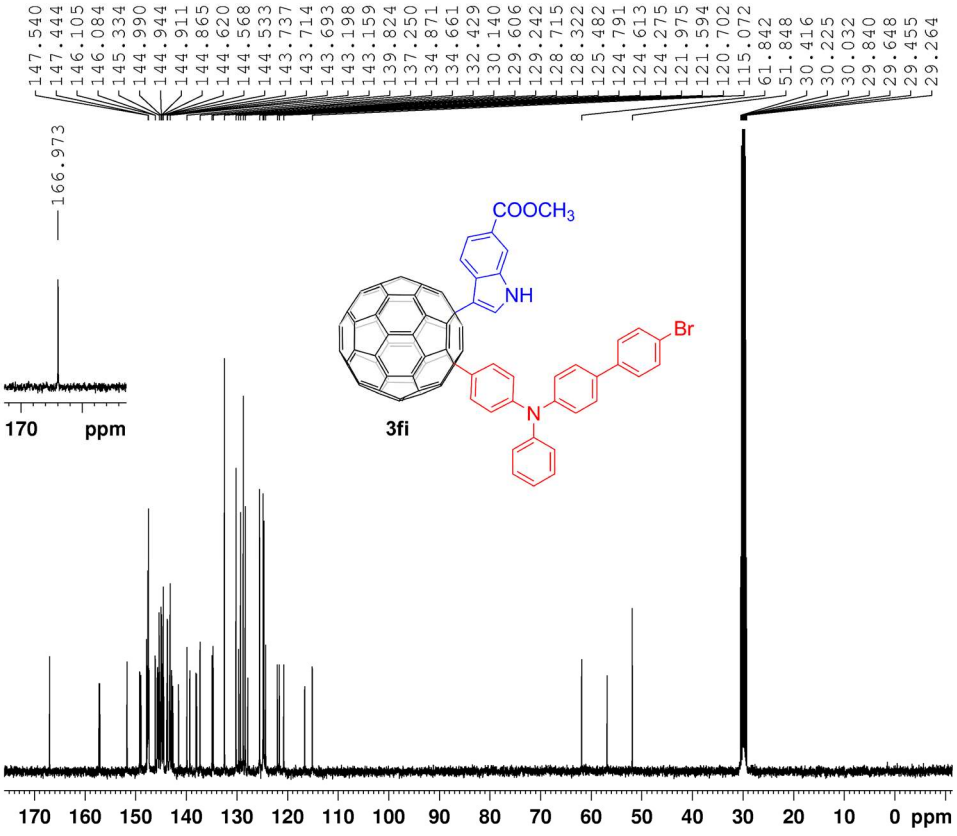
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3fi




```
NAME          Mar-16-2021
EXPNO                   2
PROCNO                  1
Date_            20210316
Time                10.06
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                     16
DS                      2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                    406
DW               60.800 usec
DE                6.50 usec
TE                300.0 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                13.50 usec
PL1               -2.00 dB
PL1W         13.45602226 W
SFO1         400.1324710 MHz
SI                  32768
SF           400.1300069 MHz
WDW                    EM
SSB                     0
LB                0.30 Hz
GB                      0
PC                1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fi

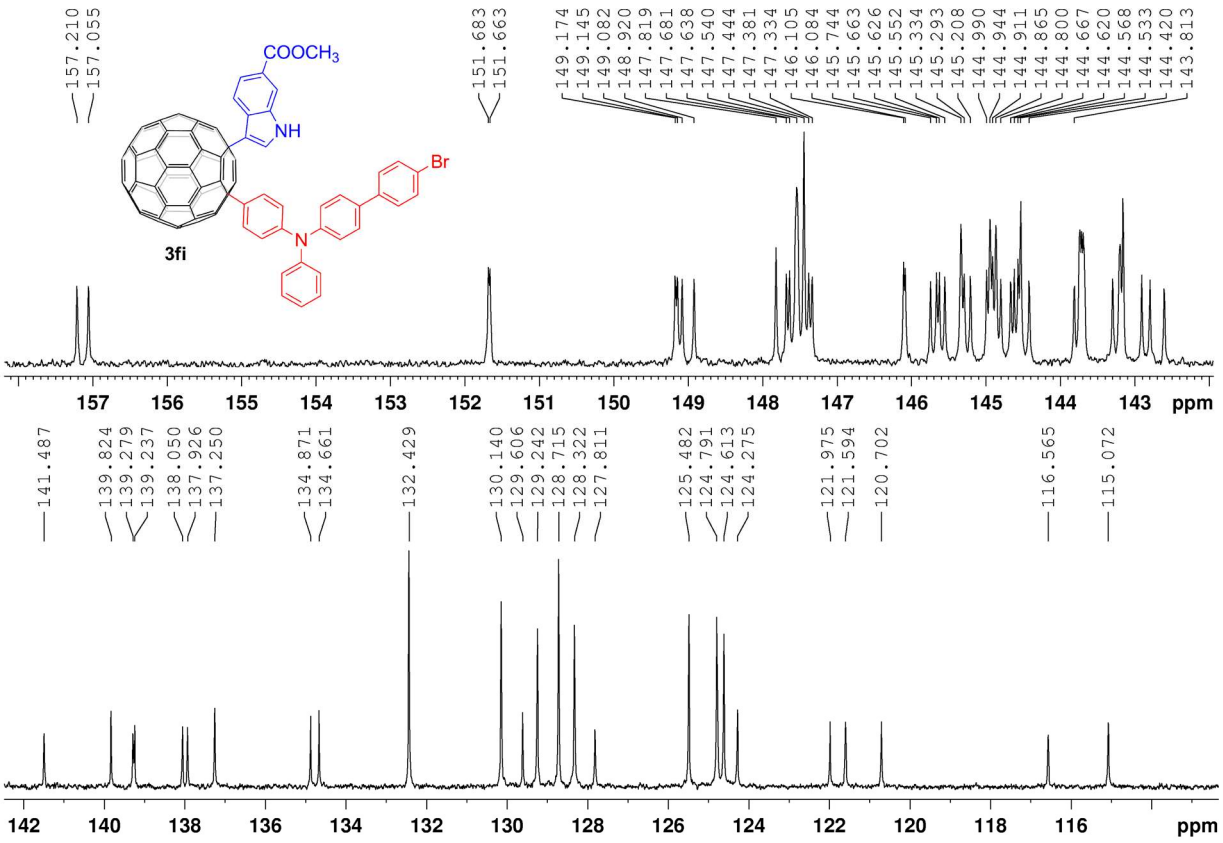



```
NAME          Mar-16-2021
EXPNO                   3
PROCNO                  1
Date_            20210316
Time                16.38
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT           Acetone
NS                  18136
DS                      4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                   80.6
DW               20.800 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

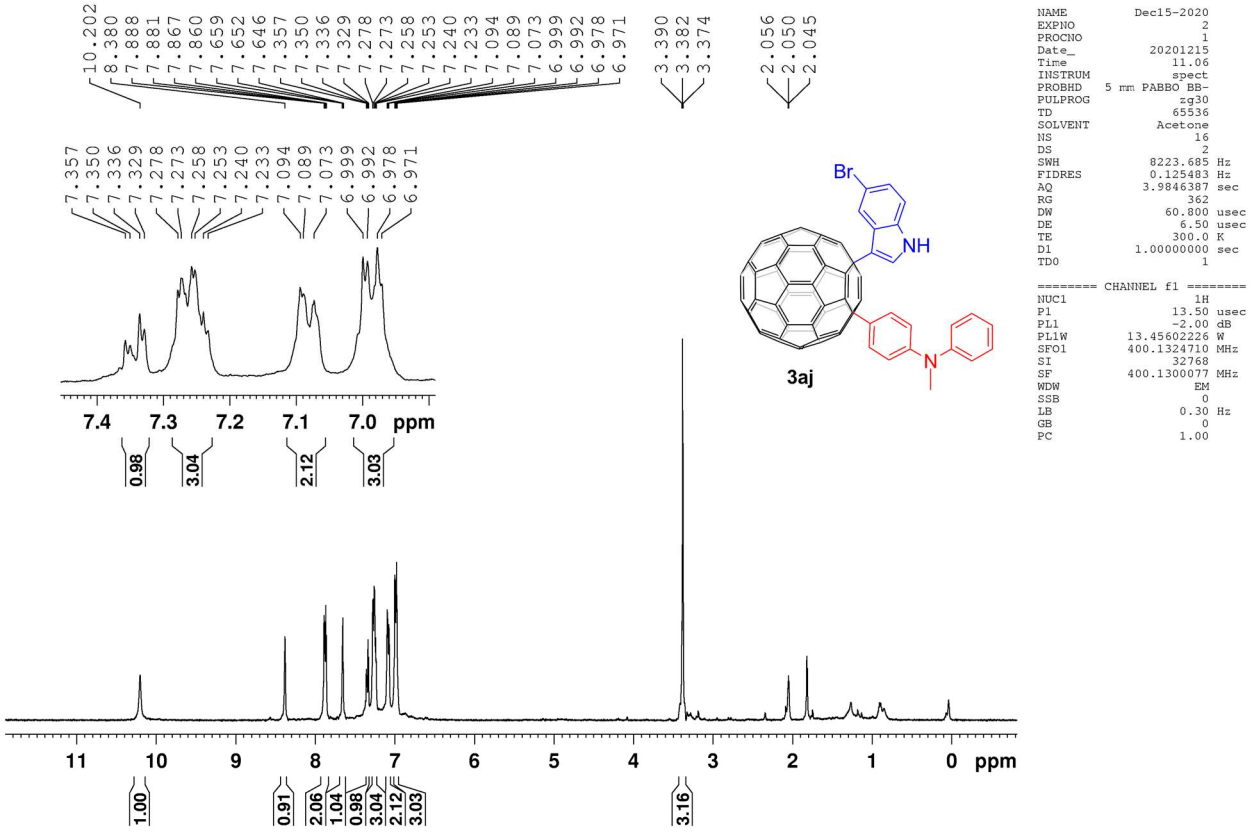
======== CHANNEL f1 ========
NUC1                  13C
P1                9.30 usec
PL1               -2.00 dB
PL1W        54.14257431 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2            80.00 usec
PL2               -2.00 dB
PL12              13.46 dB
PL13             120.00 dB
PL2W        13.45602226 W
PL12W        0.38275132 W
PL13W        0.00000000 W
SFO2        400.1316005 MHz
SI                  32768
SF          100.6127304 MHz
WDW                    EM
SSB                     0
LB                1.00 Hz
GB                      0
PC                1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3fi



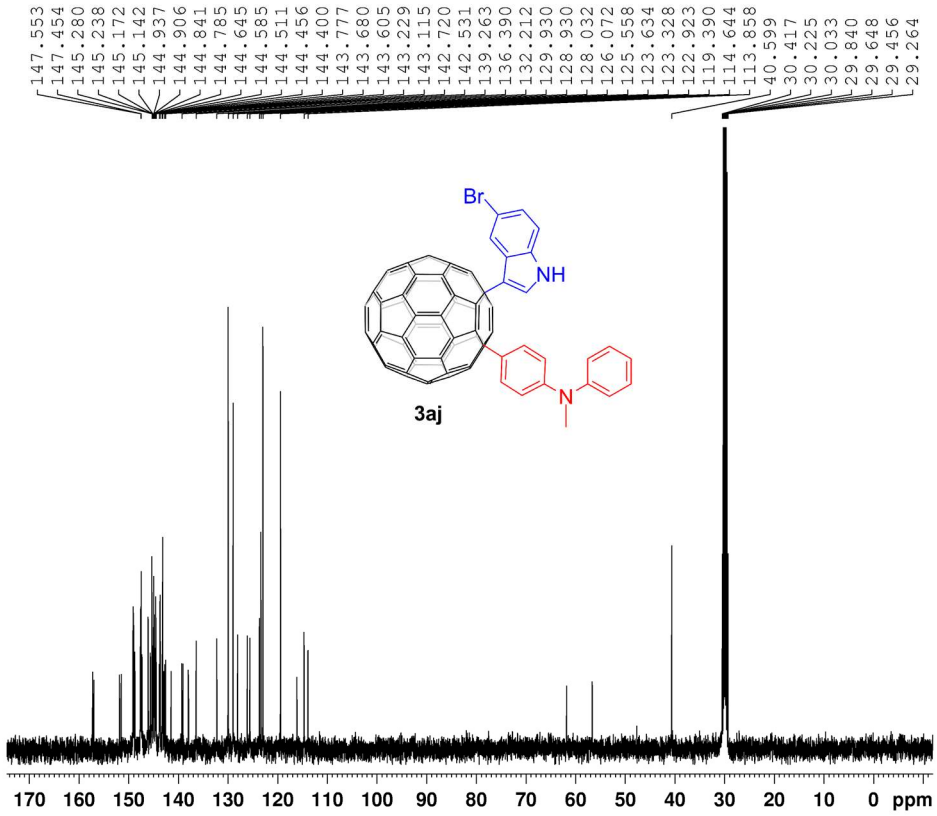
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 3aj




```
NAME            Dec15-2020
EXPNO                    2
PROCNO                   1
Date_             20201215
Time                11.06
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                      16
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     362
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W         13.45602226 W
SFO1         400.1324710 MHz
SI                   32768
SF           400.1300077 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3aj

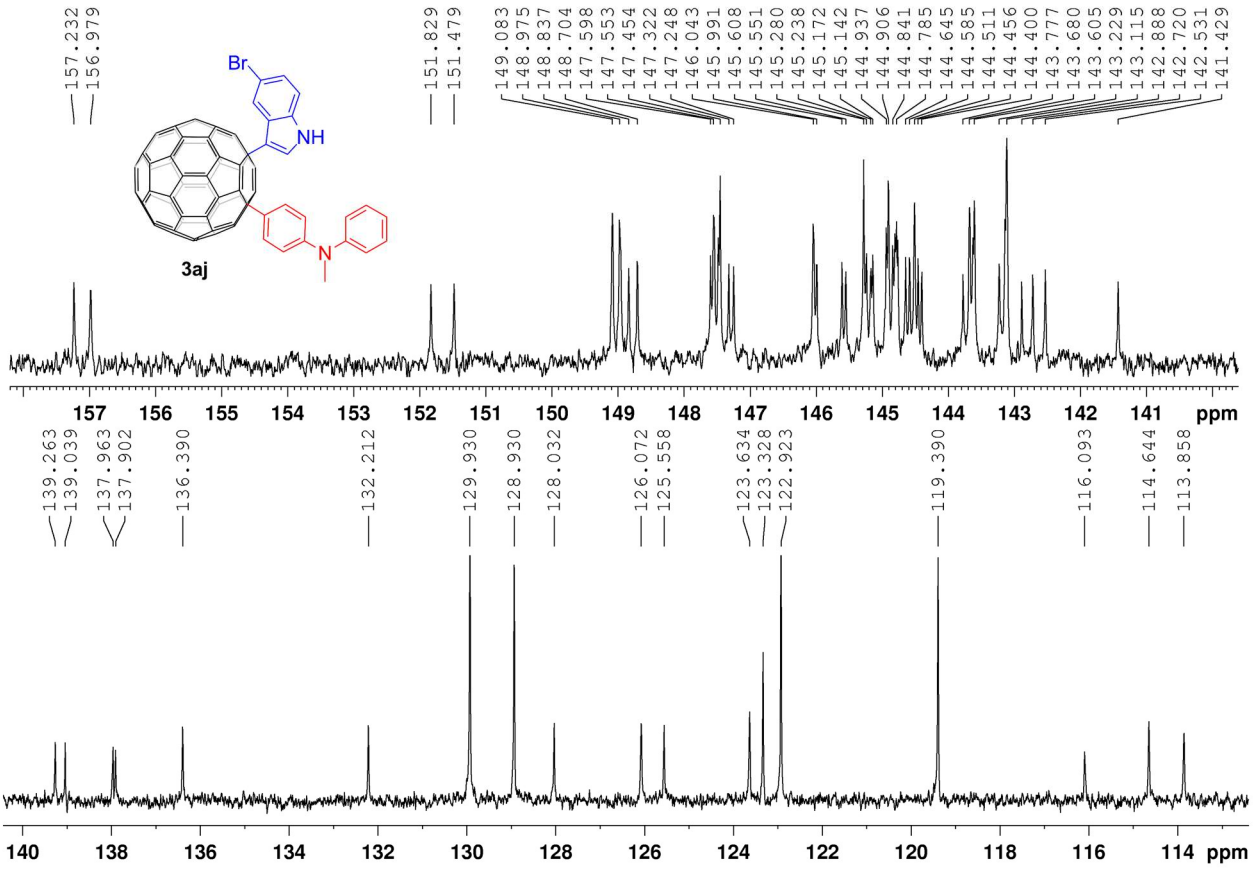



```
NAME            Dec15-2020
EXPNO                    3
PROCNO                   1
Date_             20201215
Time                 17.14
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                   18472
DS                       4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                      64
DW                 20.800 usec
DE                  6.50 usec
TE                 300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

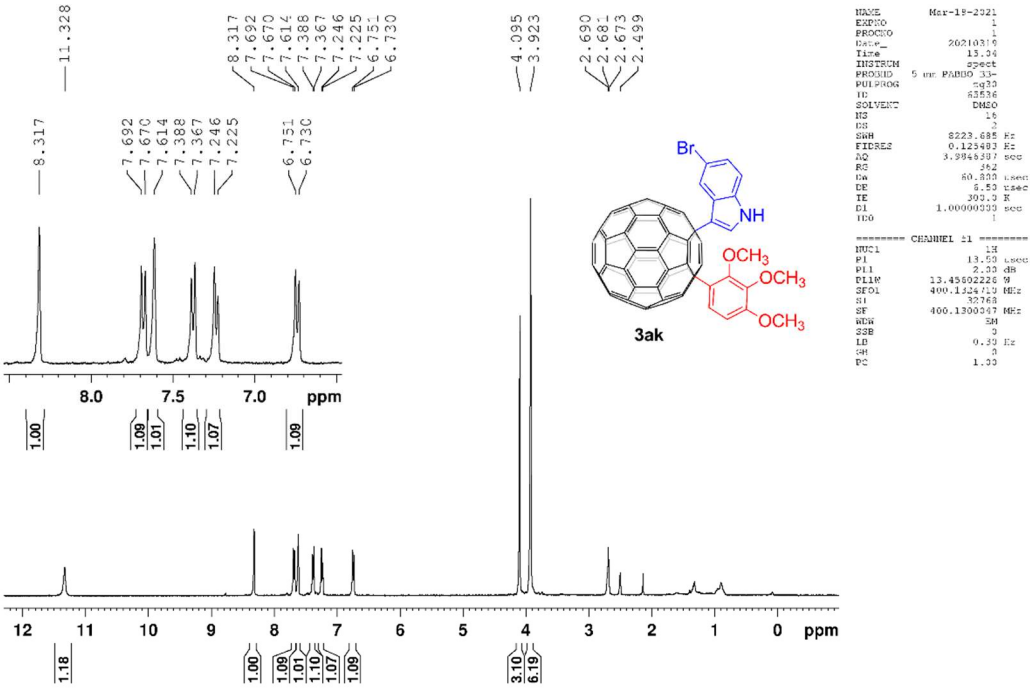
======== CHANNEL f1 ========
NUC1                   13C
P1                  9.30 usec
PL1                -2.00 dB
PL1W          54.14257431 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.46 dB
PL13               120.00 dB
PL2W          13.45602226 W
PL12W          0.38275132 W
PL13W          0.00000000 W
SFO2          400.1316005 MHz
SI                   32768
SF            100.6127365 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

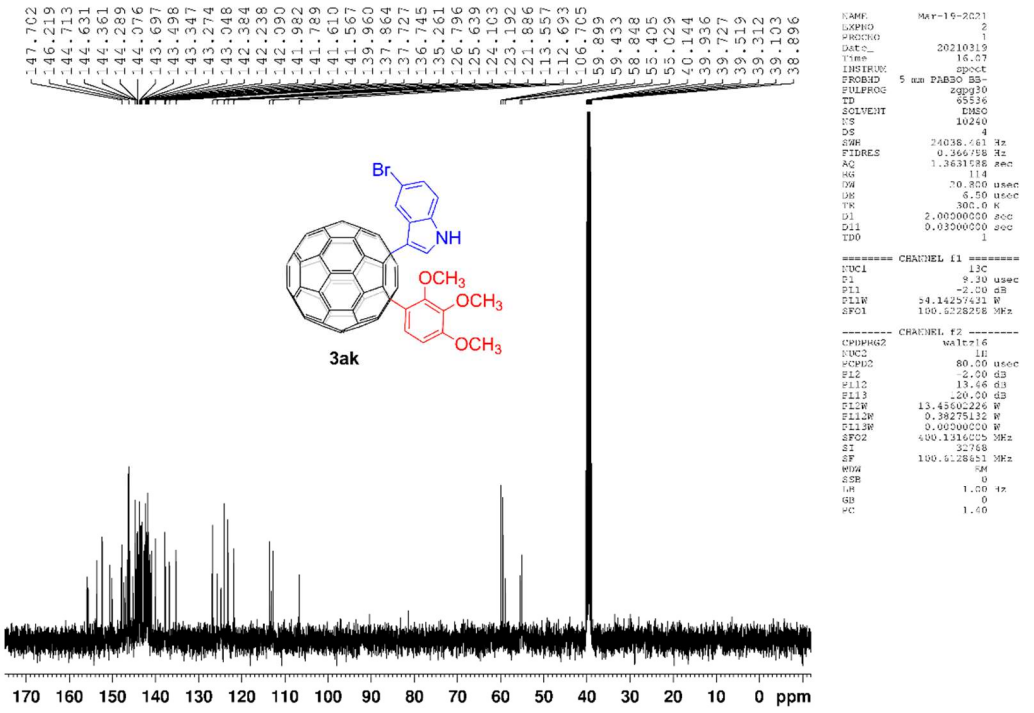
Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 3aj



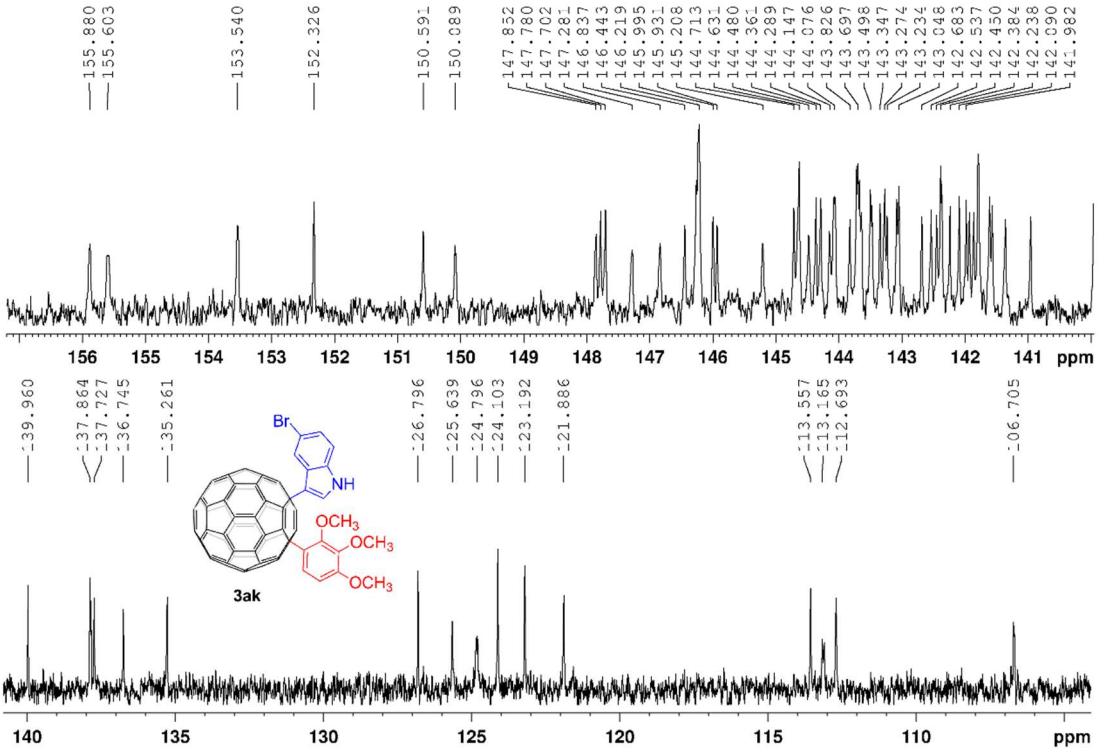
^{1}H NMR (400 MHz, CS_2/d_6-DMSO) of compound 3ak



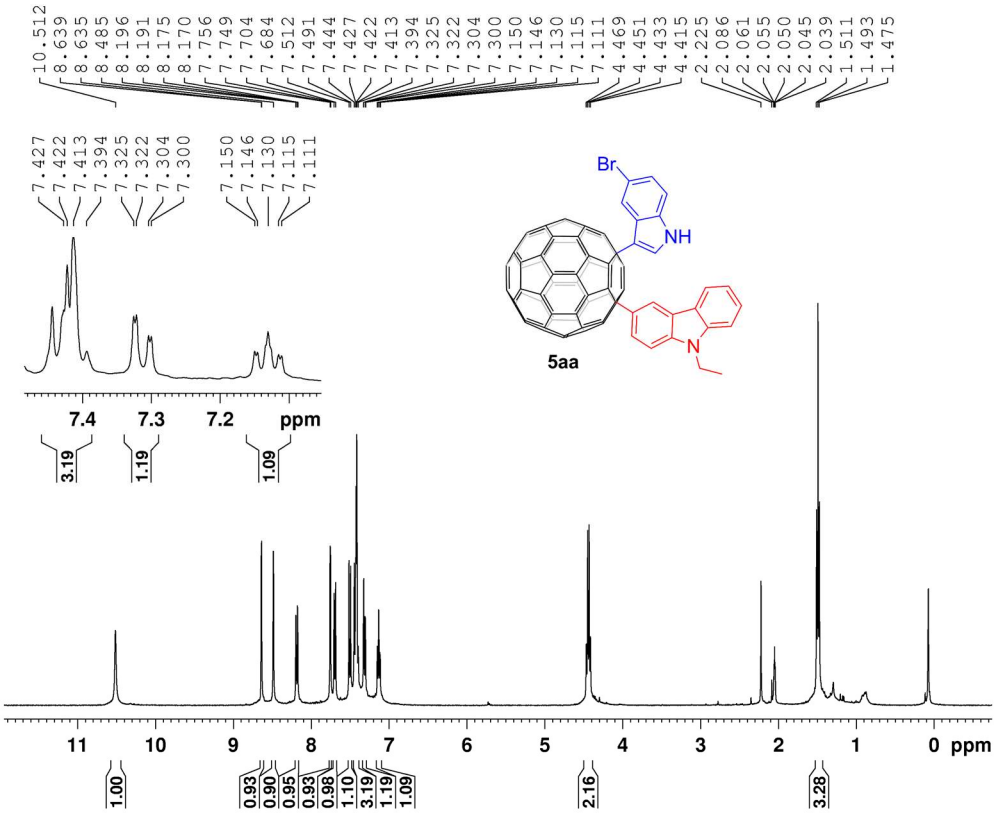
^{13}C NMR (100 MHz, CS_2/d_6-DMSO) of compound 3ak



Expanded ^{13}C NMR (100 MHz, CS_2/d_6-DMSO) of compound 3ak



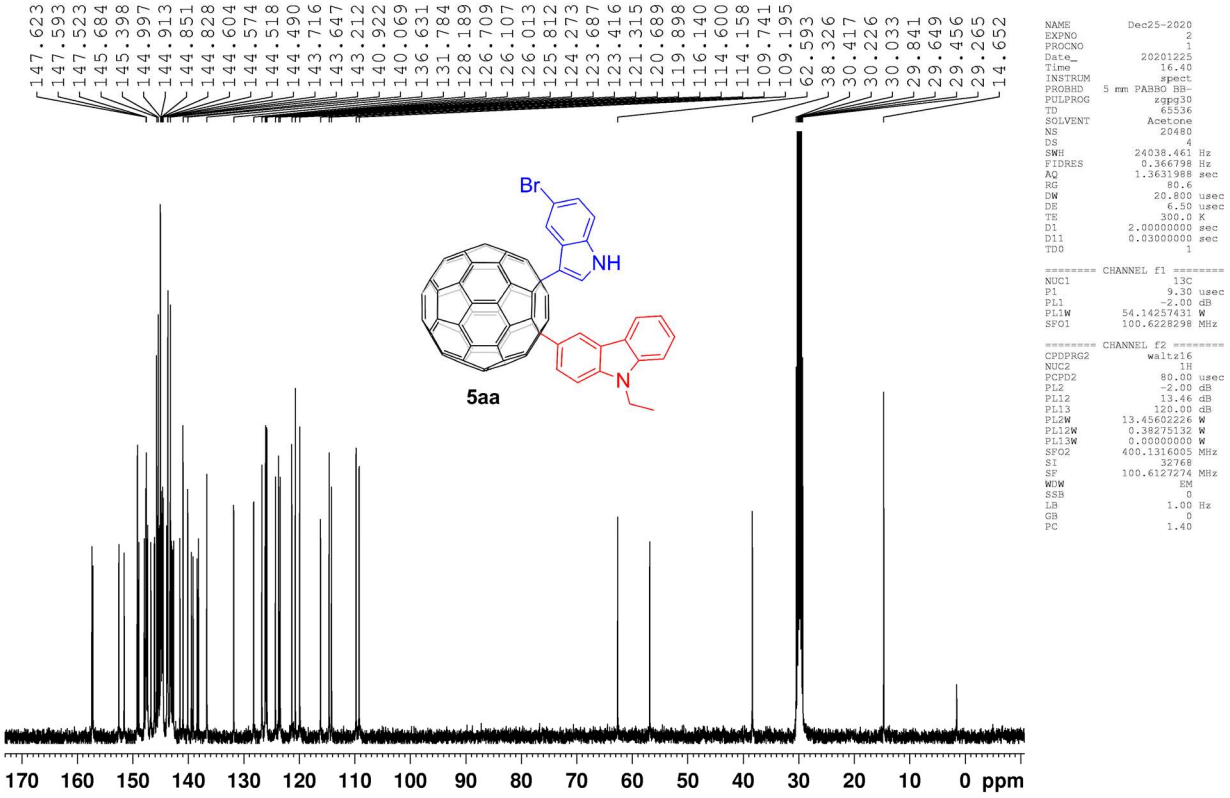
^1H NMR (400 MHz, CS_2/d_6-acetone) of compound 5aa




```
NAME                Dec25-2020
EXPNO                        1
PROCNO                       1
Date_                 20201225
Time                     15.14
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT               Acetone
NS                           16
DS                            2
SWH                   8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9846387 sec
RG                          362
DW                       60.800 usec
DE                        6.50 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                          1

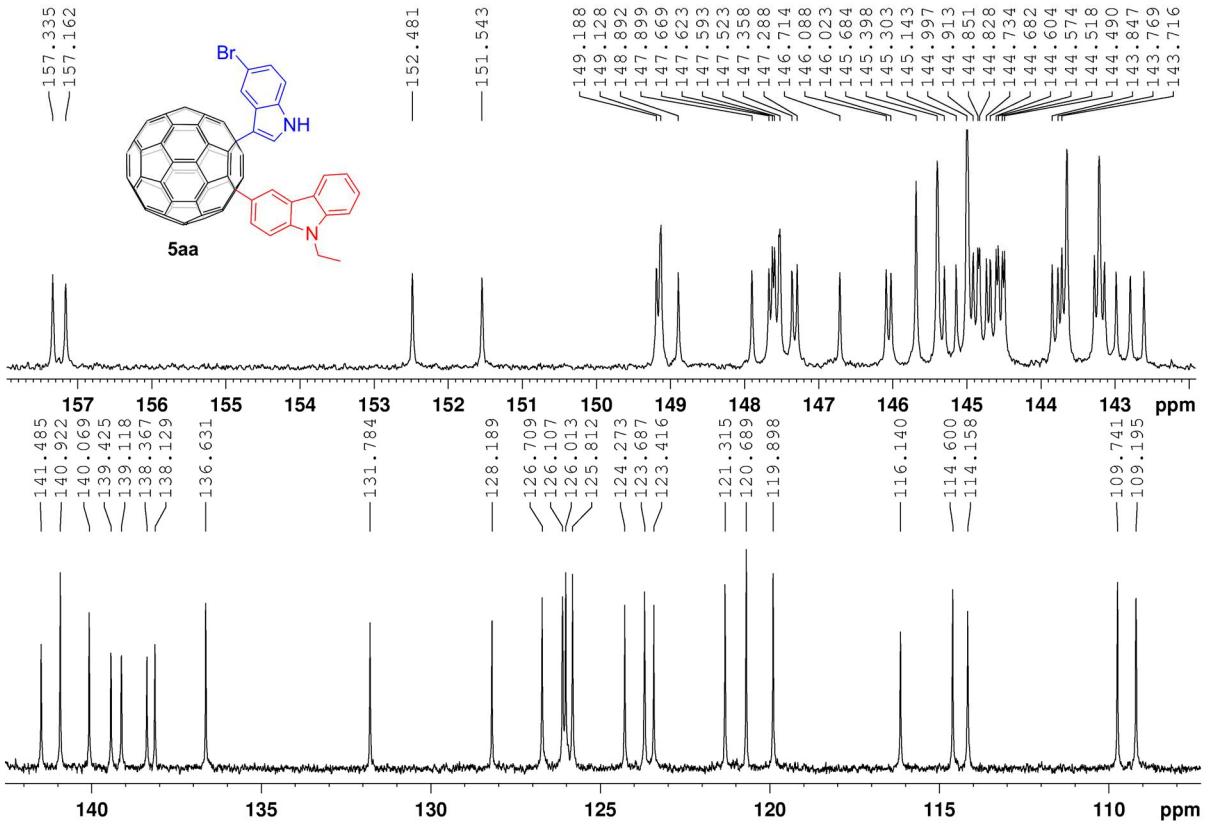
======== CHANNEL f1 ========
NUC1                        1H
P1                       13.50 usec
PL1                      -2.00 dB
PL1W                13.45602226 W
SFO1                400.1324710 MHz
SI                        32768
SF                  400.1300075 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5aa

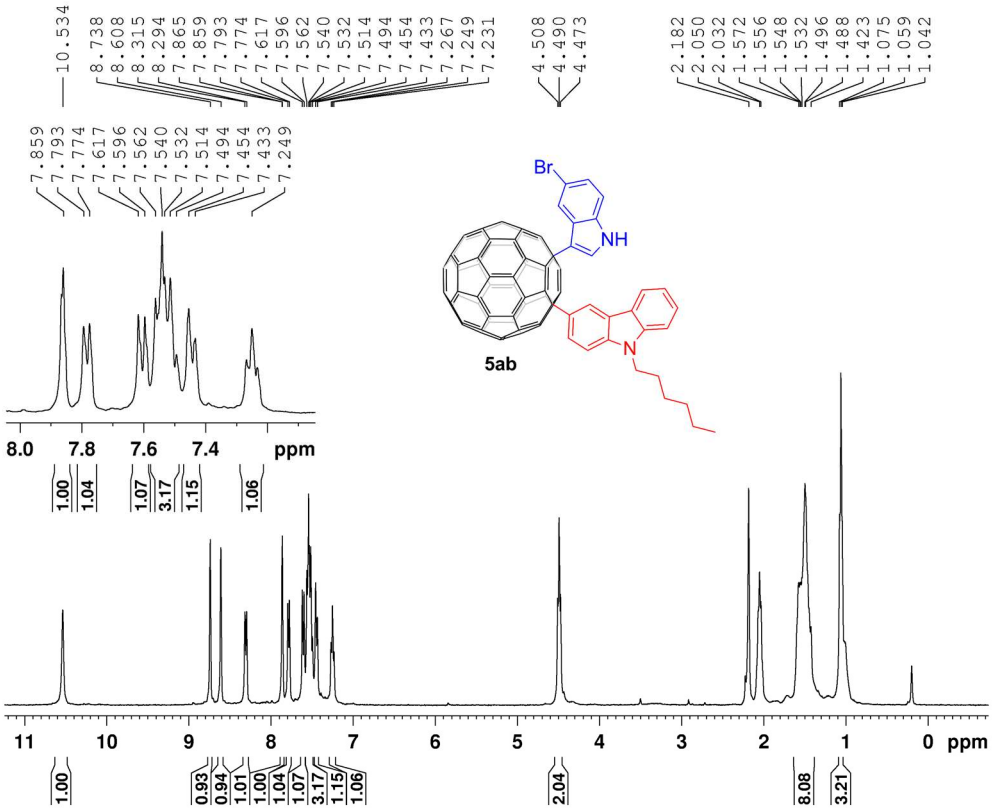


NAME	Dec25-2020
EXPNO	2
PROCNO	1
Date_	20201225
Time	16.40
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	Acetone
NS	20480
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	80.6
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.30 usec
PL1	-2.00 dB
PL1W	54.14257431 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	13.46 dB
PL13	120.00 dB
PL2W	13.45602226 W
PL12W	0.38275132 W
PL13W	0.00000000 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127274 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5aa



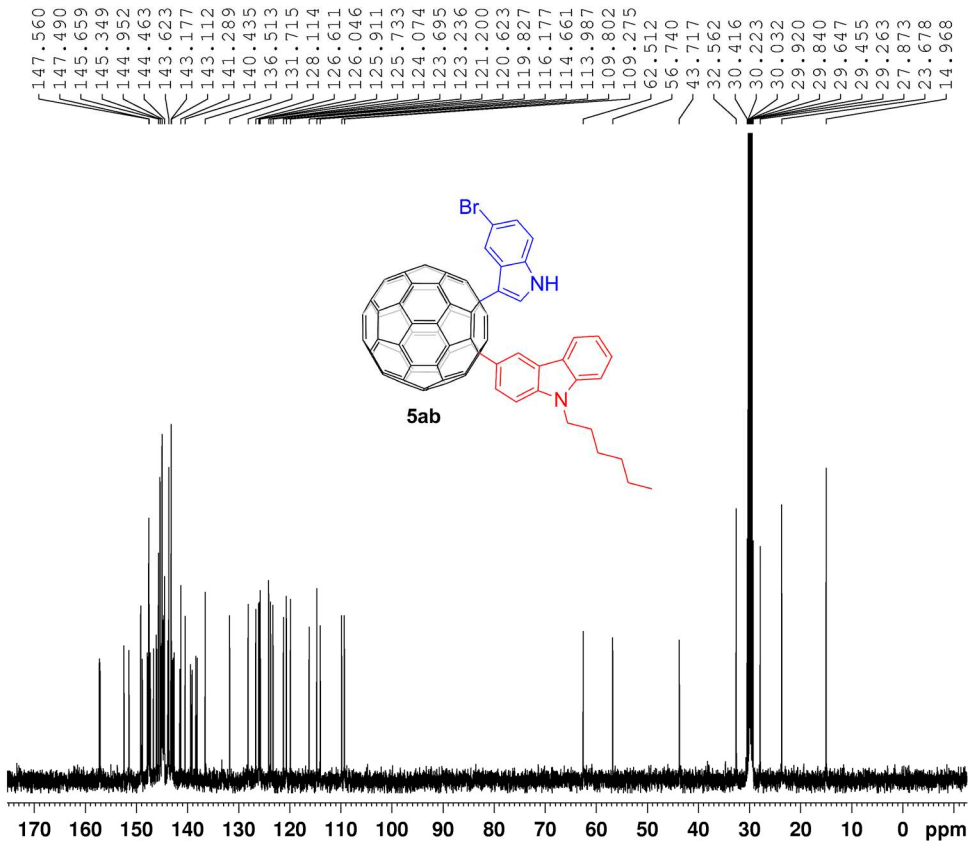
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 5ab




```
NAME            Mar30-2021
EXPNO                    1
PROCNO                   1
Date_             20210330
Time                 10.03
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT             Acetone
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES                0.125483 Hz
AQ                 3.9846387 sec
RG                      362
DW                   60.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.50 usec
PL1                  -2.00 dB
PL1W            13.45602226 W
SFO1           400.1324710 MHz
SI                    32768
SF             400.1299527 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5ab

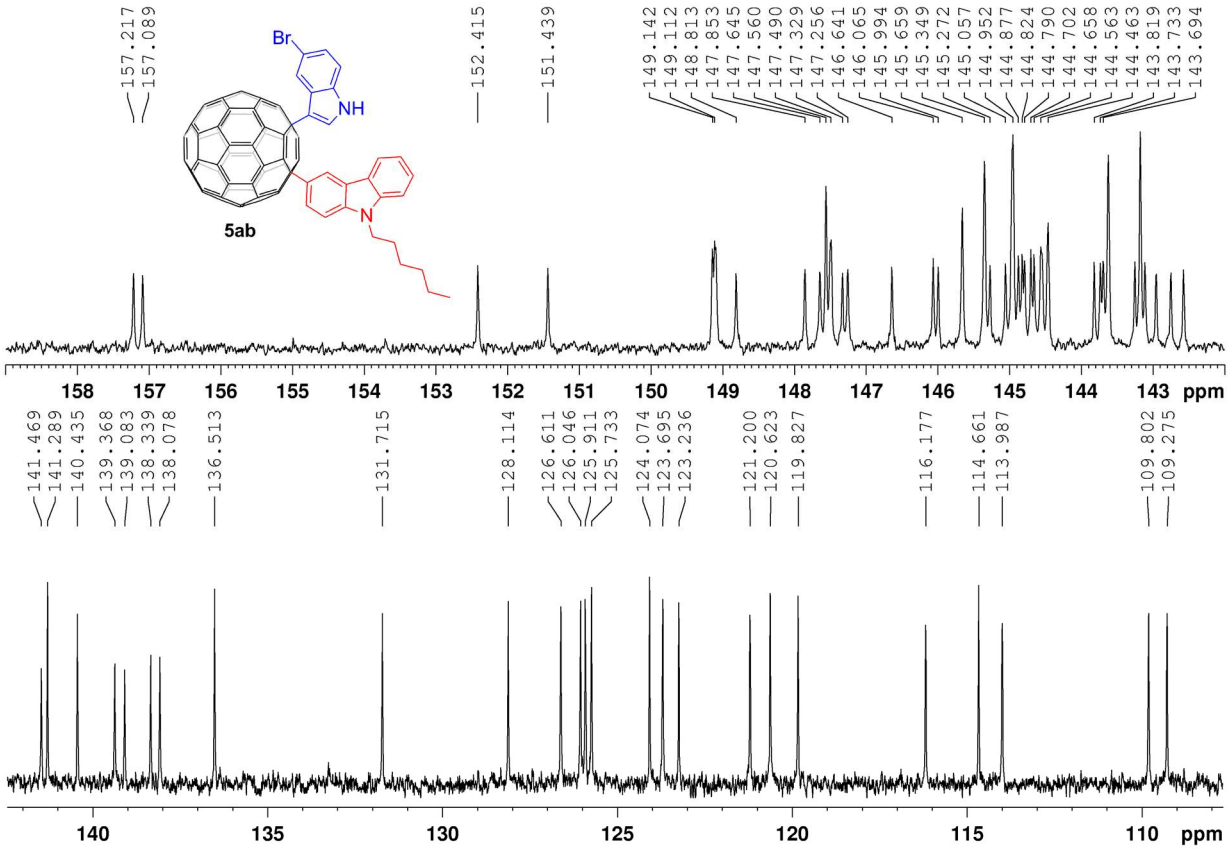



```
NAME            Mar30-2021
EXPNO                    2
PROCNO                   1
Date_             20210330
Time                 17.08
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT             Acetone
NS                    10240
DS                        4
SWH               24038.461 Hz
FIDRES                0.366798 Hz
AQ                 1.3631988 sec
RG                      181
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

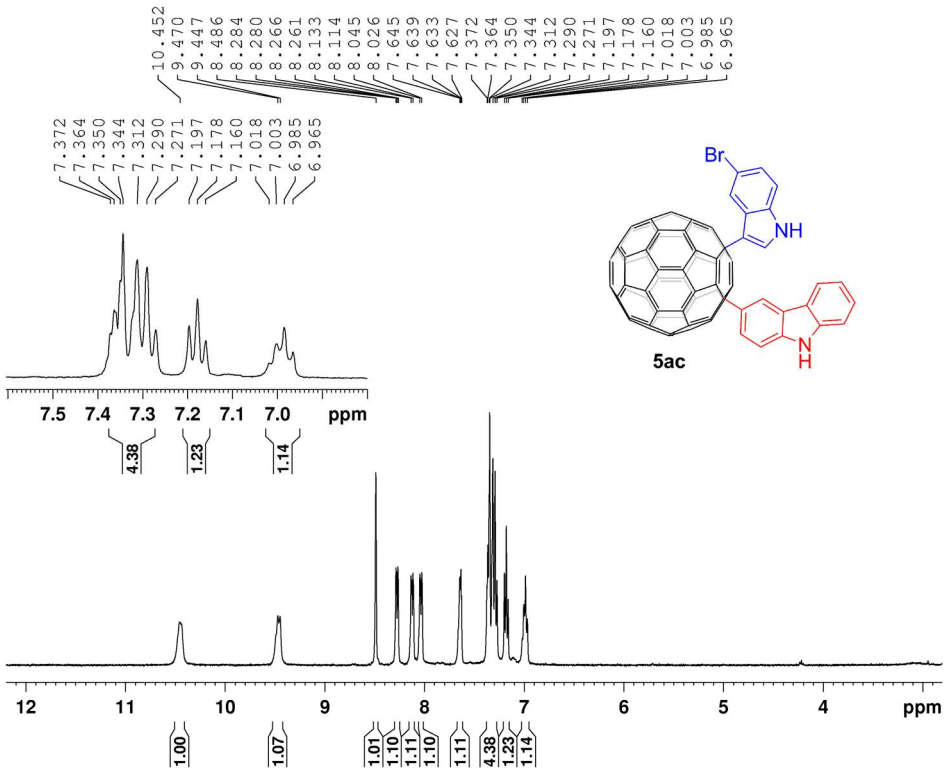
======== CHANNEL f1 ========
NUC1                    13C
P1                    9.30 usec
PL1                  -2.00 dB
PL1W            54.14257431 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 13.46 dB
PL13                120.00 dB
PL2W            13.45602226 W
PL12W            0.38275132 W
PL13W            0.00000000 W
SFO2           400.1316005 MHz
SI                    32768
SF             100.6127330 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5ab



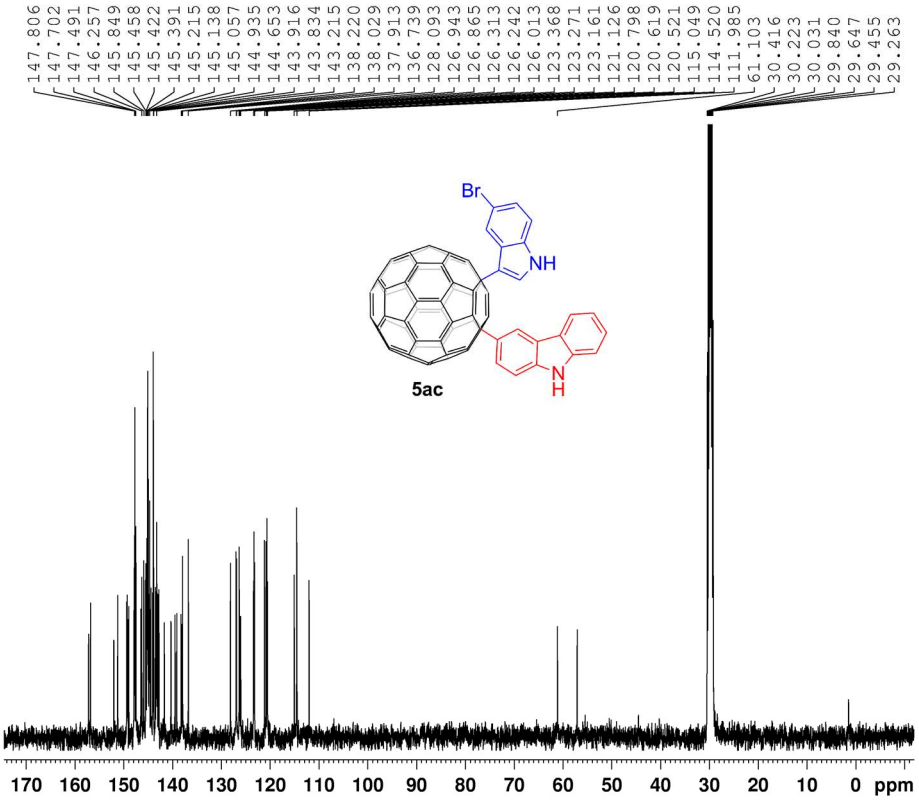
^1H NMR (400 MHz, CS_2/d_6-acetone) of compound 5ac




```
NAME                Jan04-2021
EXPNO                        3
PROCNO                       1
Date_                 20210105
Time                      9.42
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                Acetone
NS                          16
DS                           2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                         512
DW                    60.800 usec
DE                     6.50 usec
TE                     300.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                    13.50 usec
PL1                   -2.00 dB
PL1W               13.45602226 W
SFO1              400.1324710 MHz
SI                       32768
SF                400.1300074 MHz
WDW                        EM
SSB                         0
LB                    0.30 Hz
GB                          0
PC                       1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5ac

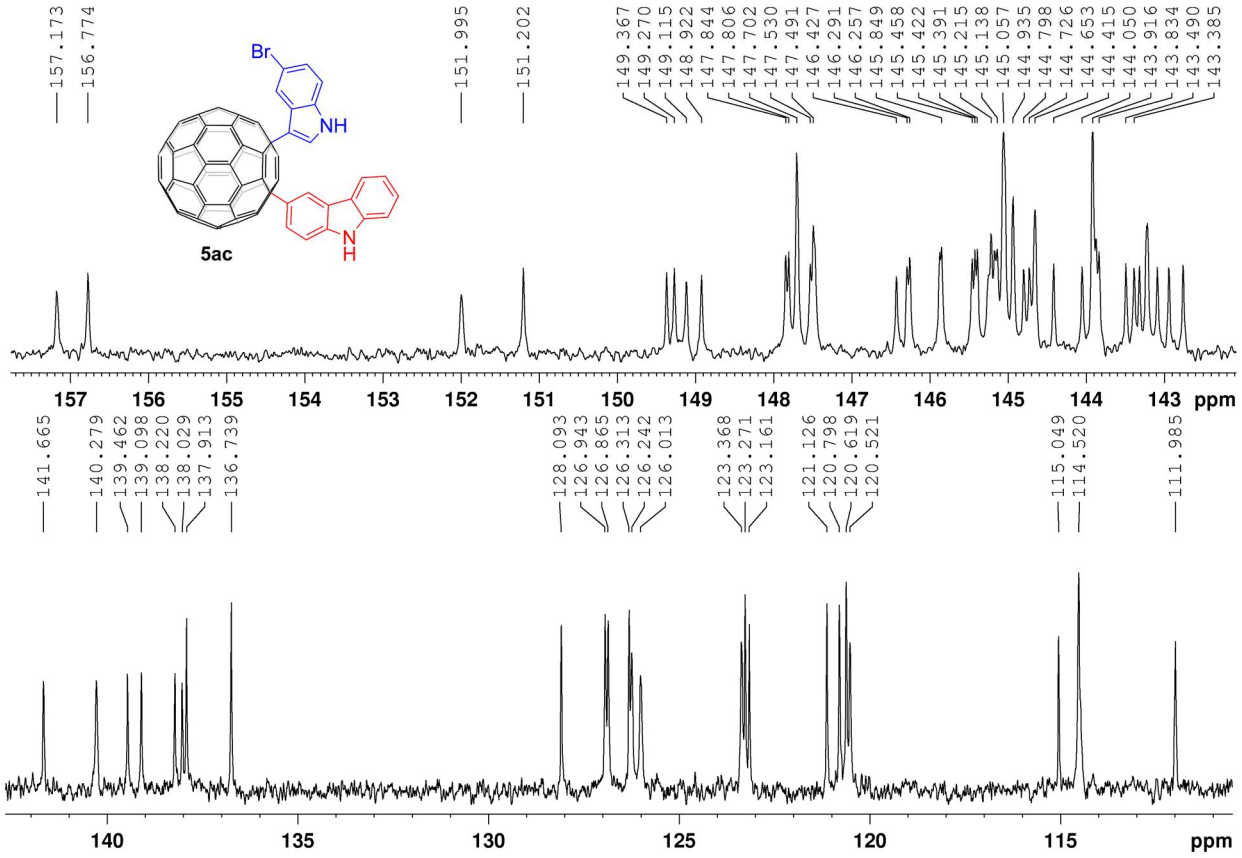



```
NAME            Jan04-2021
EXPNO                    2
PROCNO                   1
Date_             20210104
Time                 17.01
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                    17439
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                      71.8
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

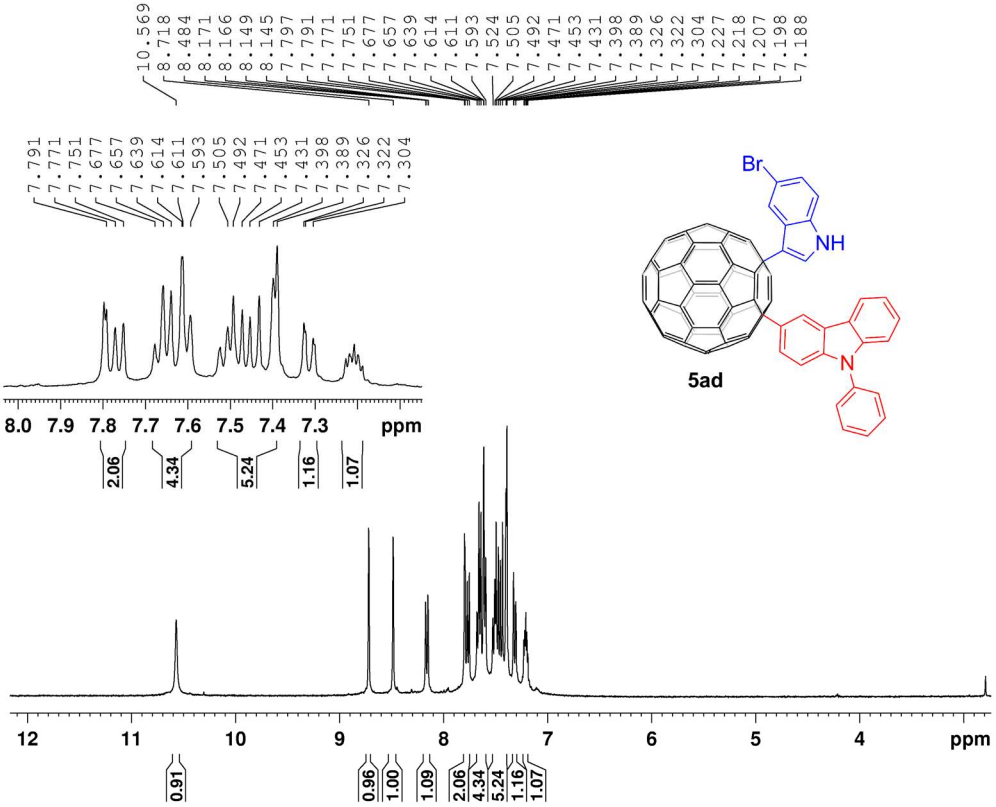
======== CHANNEL f1 ========
NUC1                    13C
P1                    9.30 usec
PL1                  -2.00 dB
PL1W             54.14257431 W
SFO1             100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 13.46 dB
PL13                120.00 dB
PL2W             13.45602226 W
PL12W             0.38275132 W
PL13W             0.00000000 W
SFO2             400.1316005 MHz
SI                      32768
SF               100.6127208 MHz
WDW                     EM
SSB                      0
LB                    2.00 Hz
GB                       0
PC                    1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5ac



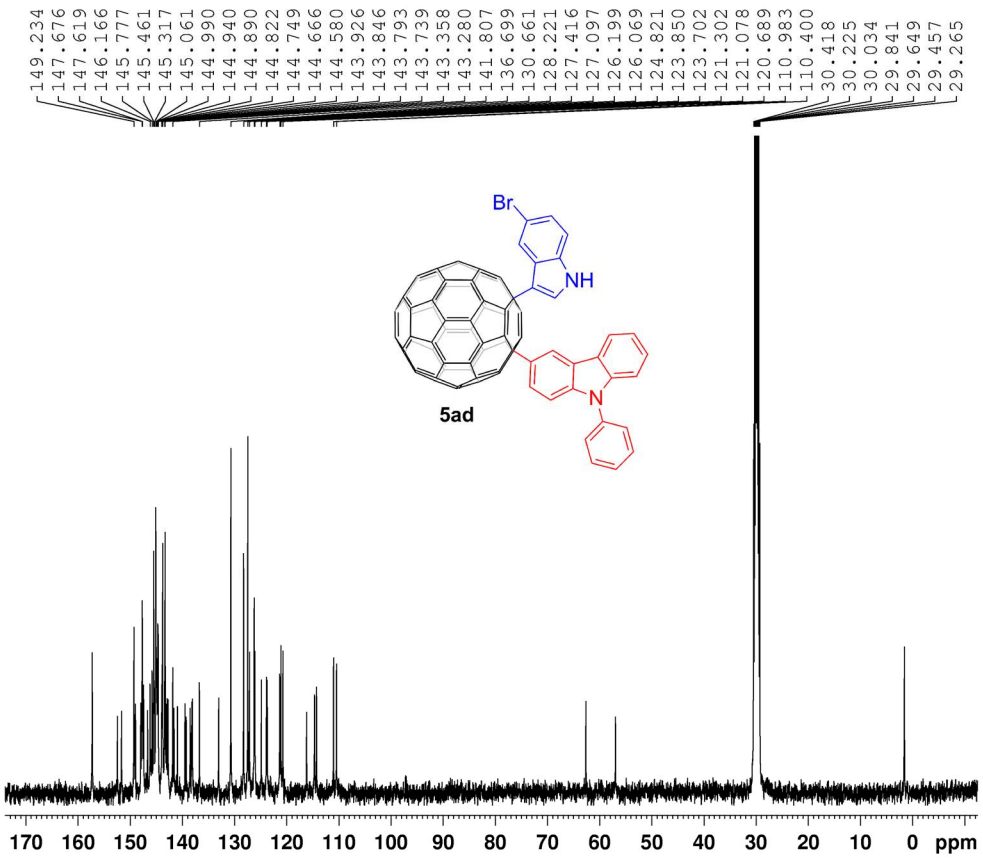
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 5ad




```
NAME          Dec24-2020
EXPNO                  1
PROCNO                 1
Date_           20201224
Time               19.29
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   362
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W        13.45602226 W
SFO1       400.1324710 MHz
SI                 32768
SF         400.1300073 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5ad

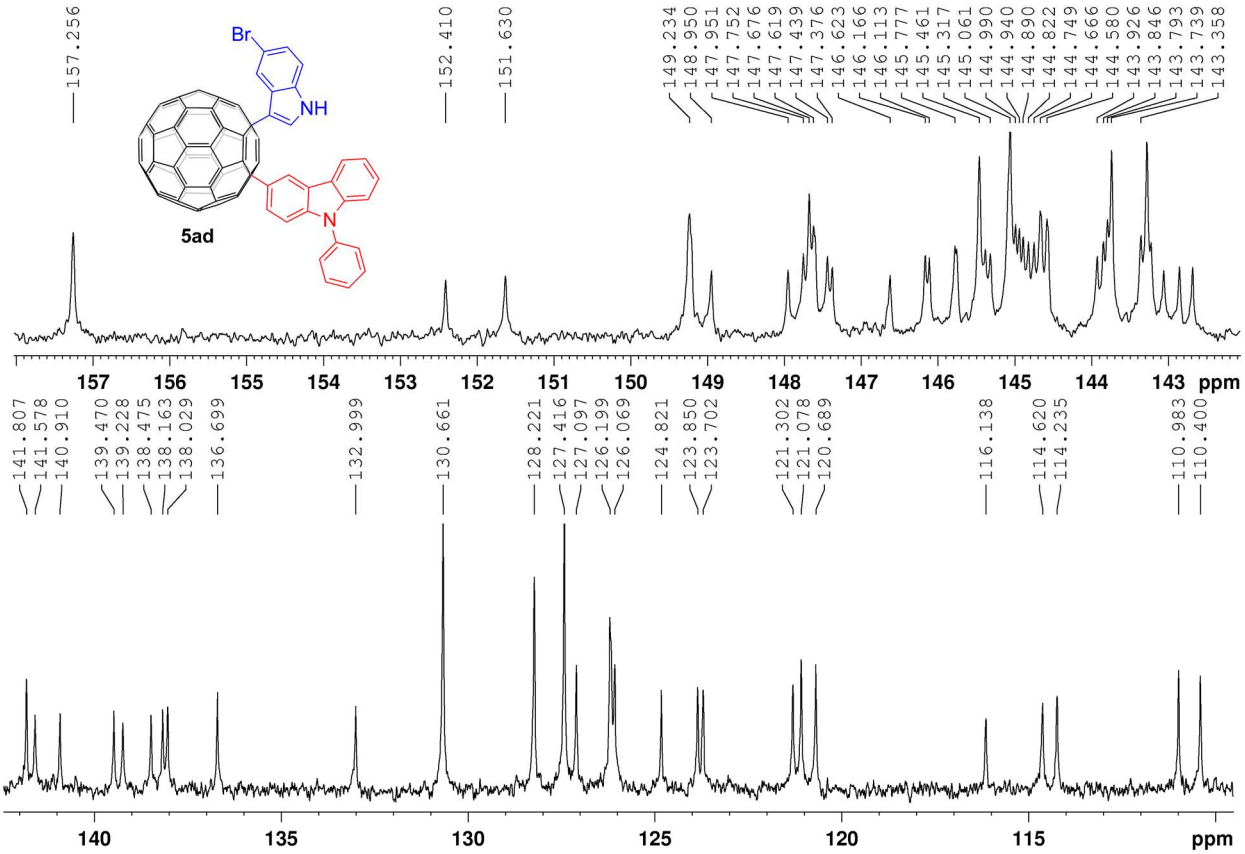



```
NAME          Dec24-2020
EXPNO                  2
PROCNO                 1
Date_           20201224
Time               19.34
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                 16305
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                   114
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

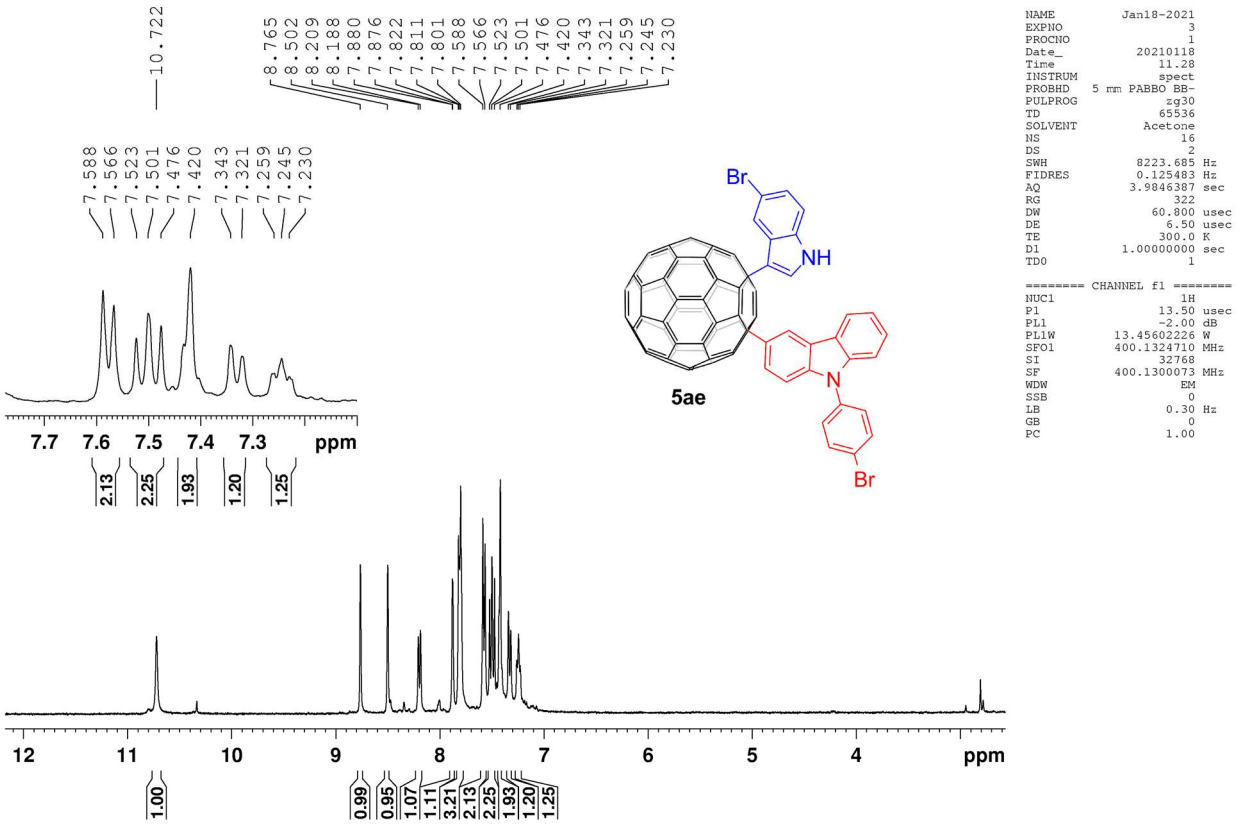
======== CHANNEL f1 ========
NUC1                 13C
P1                 9.30 usec
PL1                -2.00 dB
PL1W        54.14257431 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               13.46 dB
PL13              120.00 dB
PL2W        13.45602226 W
PL12W        0.38275132 W
PL13W        0.00000000 W
SFO2       400.1316005 MHz
SI                 32768
SF         100.6127225 MHz
WDW                   EM
SSB                    0
LB                 2.00 Hz
GB                     0
PC                  1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5ad



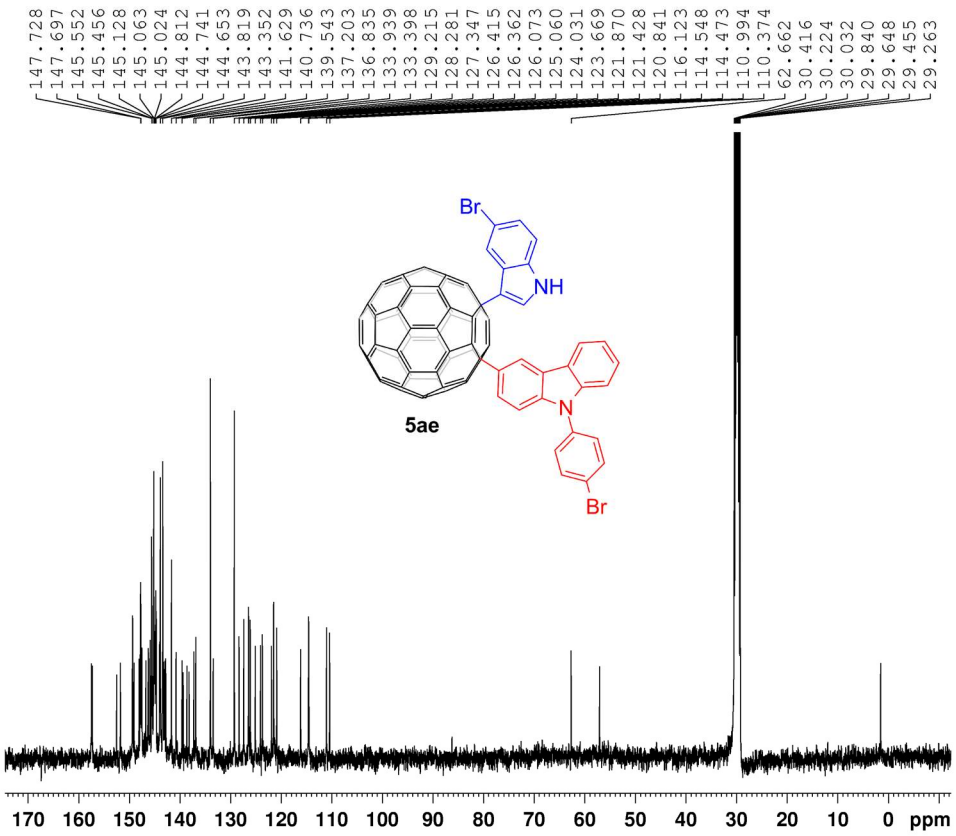
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 5ae




```
NAME            Jan18-2021
EXPNO                    3
PROCNO                   1
Date_             20210118
Time                11.28
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                      16
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     322
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W          13.45602226 W
SFO1         400.1324710 MHz
SI                   32768
SF           400.1300073 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5ae

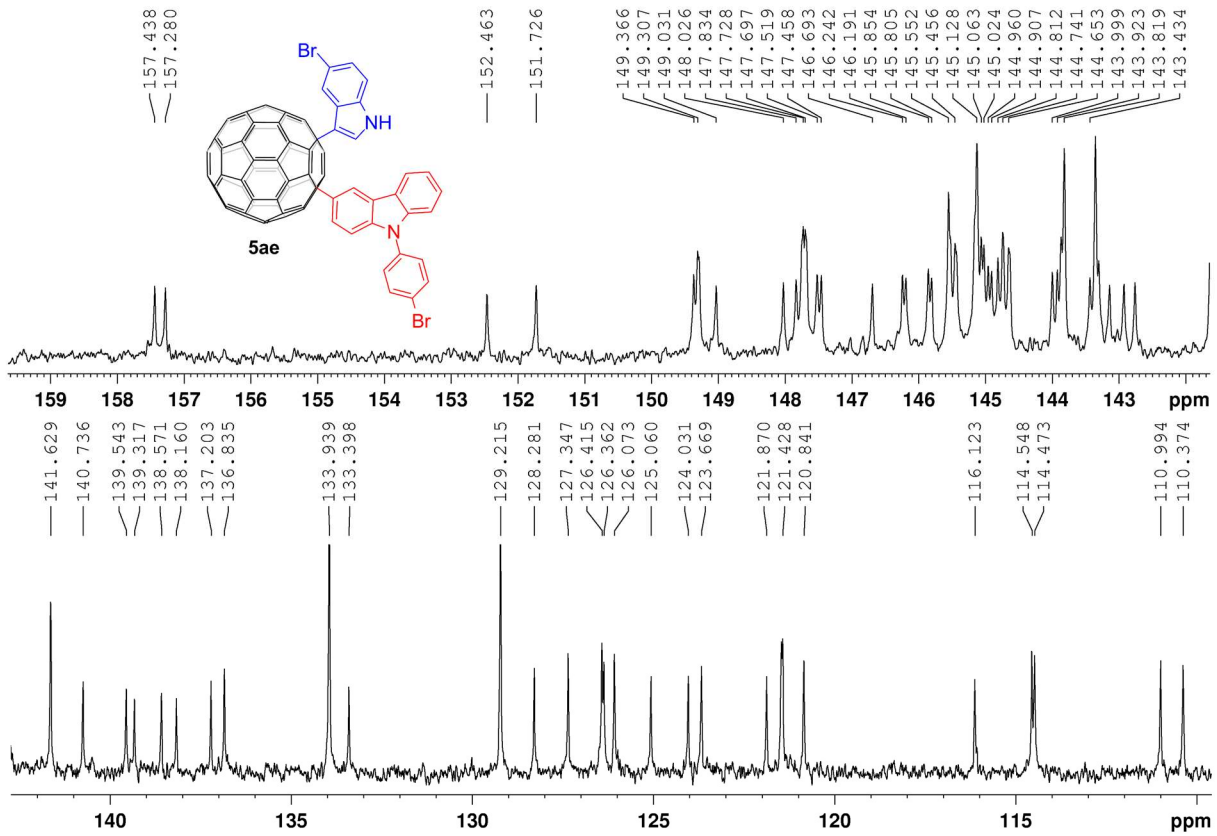



```
NAME            Jan18-2021
EXPNO                    4
PROCNO                   1
Date_             20210118
Time                11.38
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                   20480
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                     71.8
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

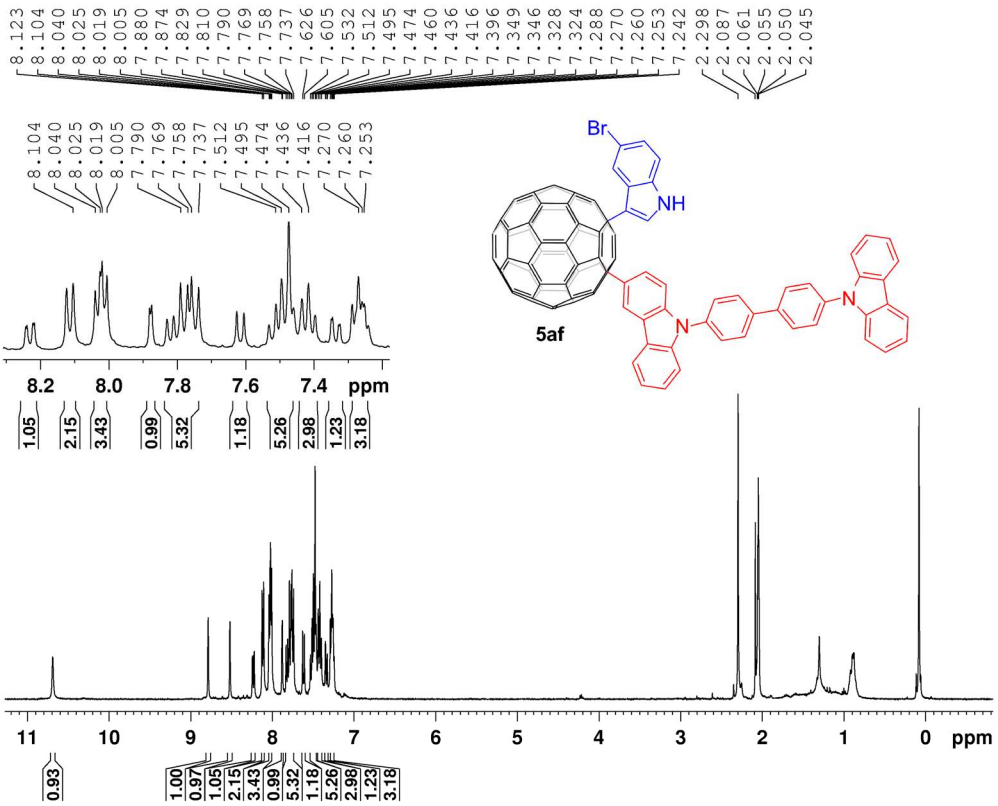
======== CHANNEL f1 ========
NUC1                   13C
P1                    9.30 usec
PL1                  -2.00 dB
PL1W          54.14257431 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 13.46 dB
PL13                120.00 dB
PL2W          13.45602226 W
PL12W          0.38275132 W
PL13W          0.00000000 W
SFO2         400.1316005 MHz
SI                   32768
SF           100.6127155 MHz
WDW                     EM
SSB                      0
LB                    2.00 Hz
GB                       0
PC                    1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5ae



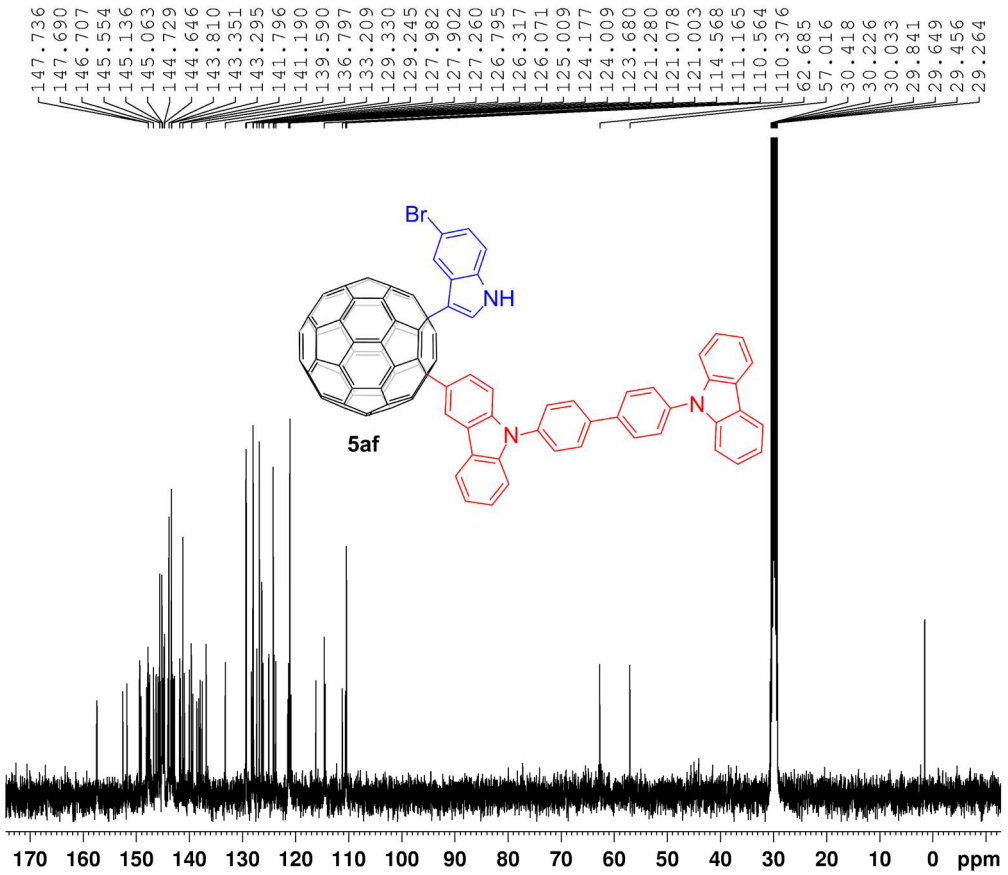
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 5af




```
NAME            Jan05-2021
EXPNO                    1
PROCNO                   1
Date_             20210105
Time                 9.56
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                      16
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     512
DW                  60.800 usec
DE                   6.50 usec
TE                  300.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.50 usec
PL1                 -2.00 dB
PL1W           13.45602226 W
SFO1           400.1324710 MHz
SI                   32768
SF             400.1300075 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5af

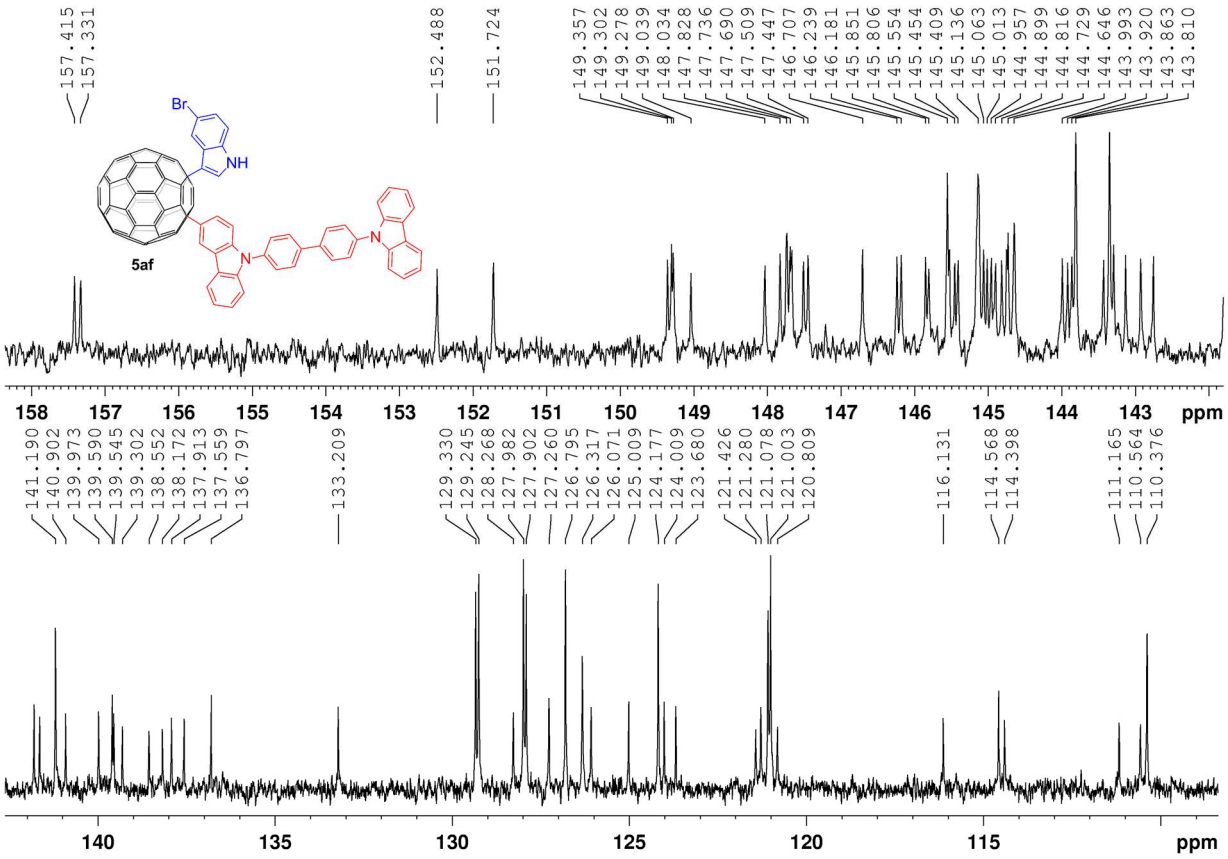



```
NAME            Jan05-2021
EXPNO                    2
PROCNO                   1
Date_             20210105
Time                16.57
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT           Acetone
NS                   18348
DS                       4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                     114
DW                  20.800 usec
DE                   6.50 usec
TE                  300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

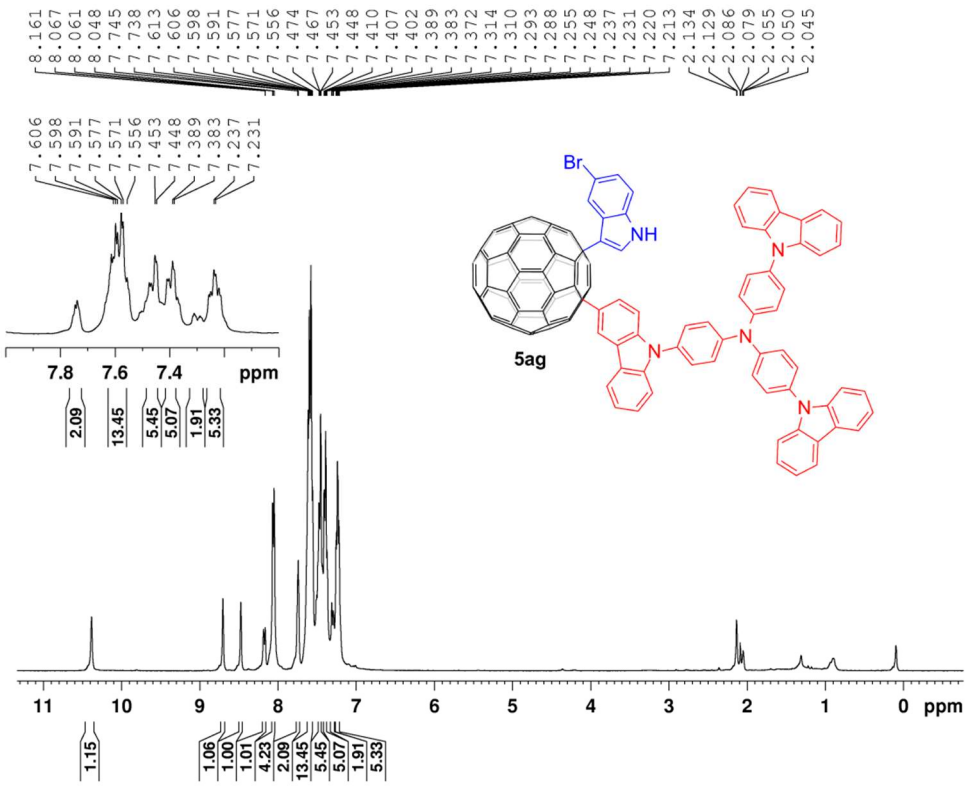
======== CHANNEL f1 ========
NUC1                  13C
P1                   9.30 usec
PL1                 -2.00 dB
PL1W           54.14257431 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -2.00 dB
PL12                13.46 dB
PL13               120.00 dB
PL2W           13.45602226 W
PL12W           0.38275132 W
PL13W           0.00000000 W
SFO2           400.1316005 MHz
SI                   32768
SF             100.6127186 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5af



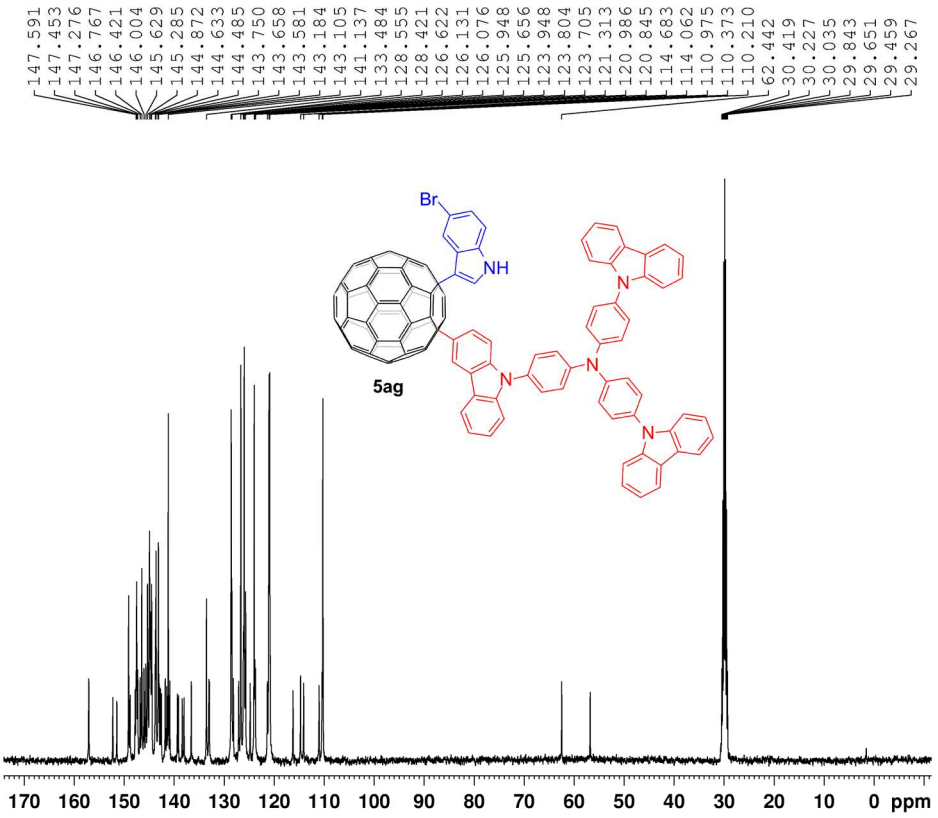
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 5ag




```
NAME            Mar-10-2021
EXPNO                     1
PROCNO                    1
Date_              20210310
Time                   9.42
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT             Acetone
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      228
DW                   60.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                 1.00000000 sec
TD0                       1

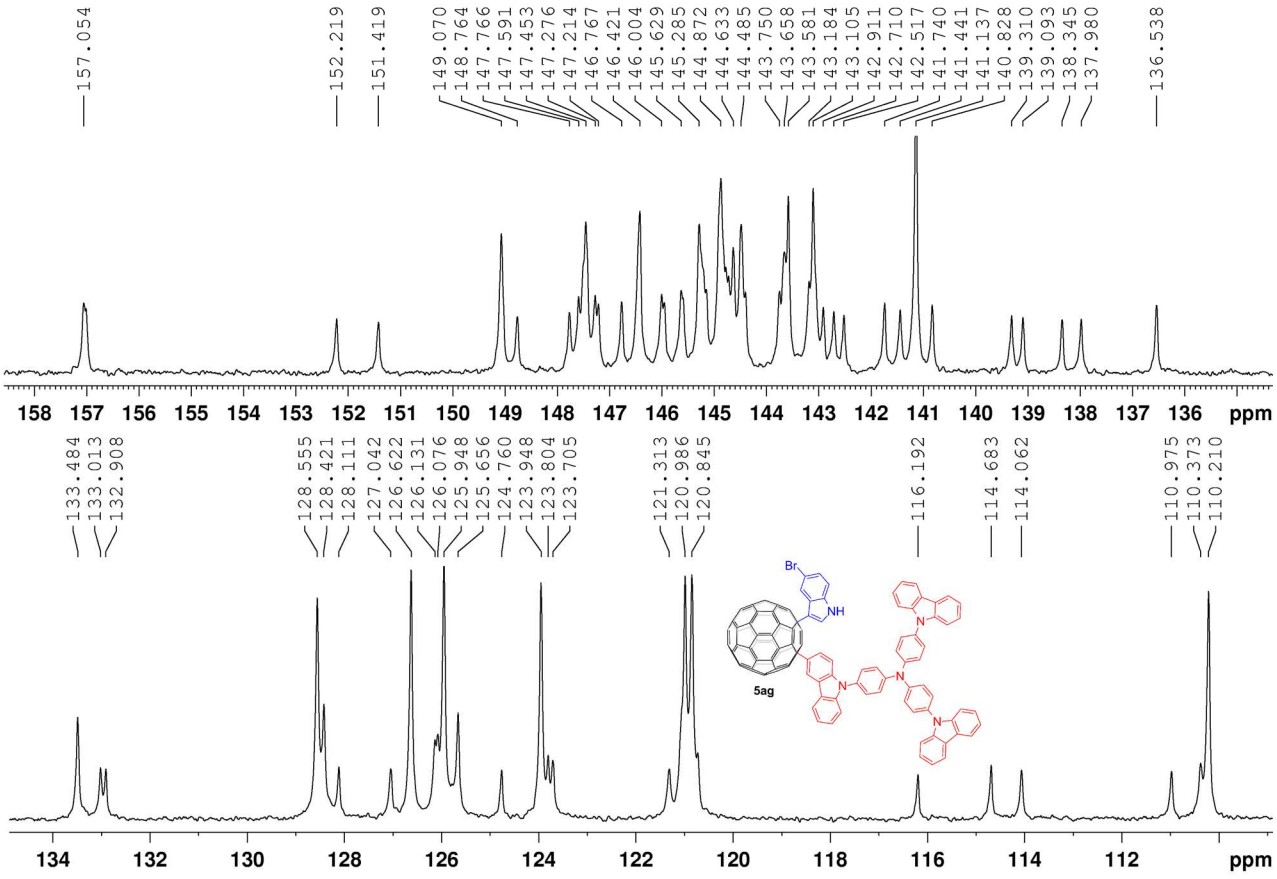
======== CHANNEL f1 ========
NUC1                      1H
P1                   13.50 usec
PL1                   -2.00 dB
PL1W            13.45602226 W
SFO1           400.1324710 MHz
SI                    32768
SF             400.1300077 MHz
WDW                      EM
SSB                       0
LB                   0.50 Hz
GB                        0
PC                     1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5ag

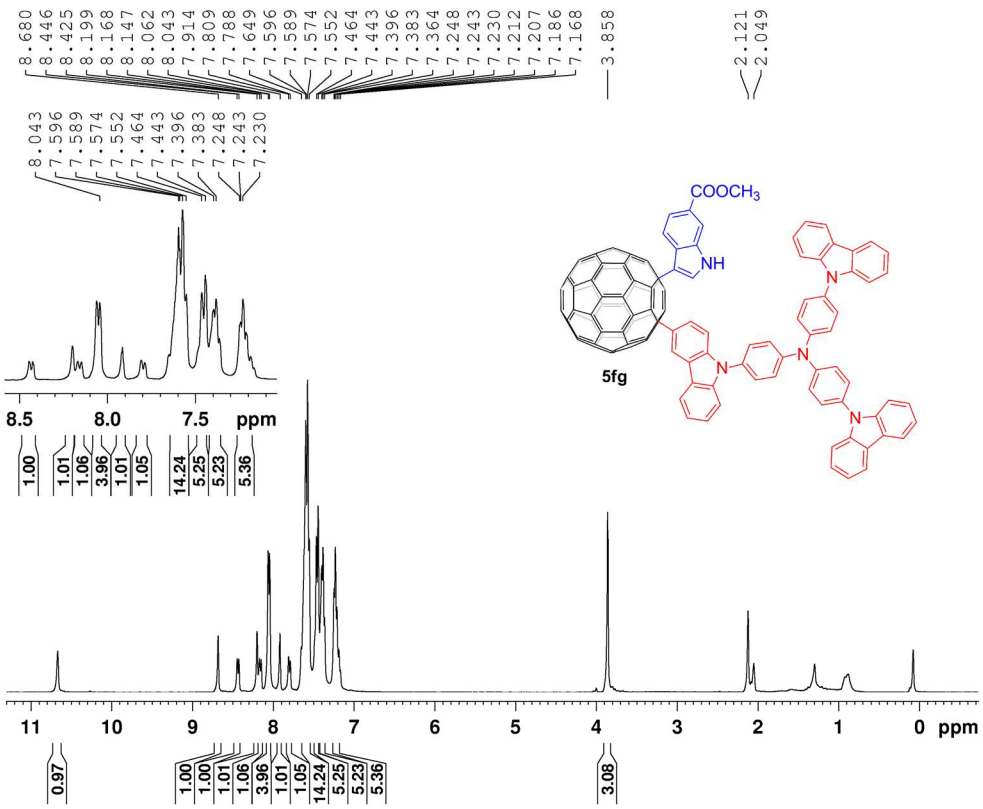


NAME	Mar-10-2021
EXPNO	2
PROCNO	1
Date_	20210310
Time	15.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	Acetone
NS	18208
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	80.6
DW	20.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.30 usec
PL1	-2.00 dB
PL1W	54.14257431 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	13.46 dB
PL13	120.00 dB
PL2W	13.45602226 W
PL12W	0.38275132 W
PL13W	0.00000000 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127325 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5ag



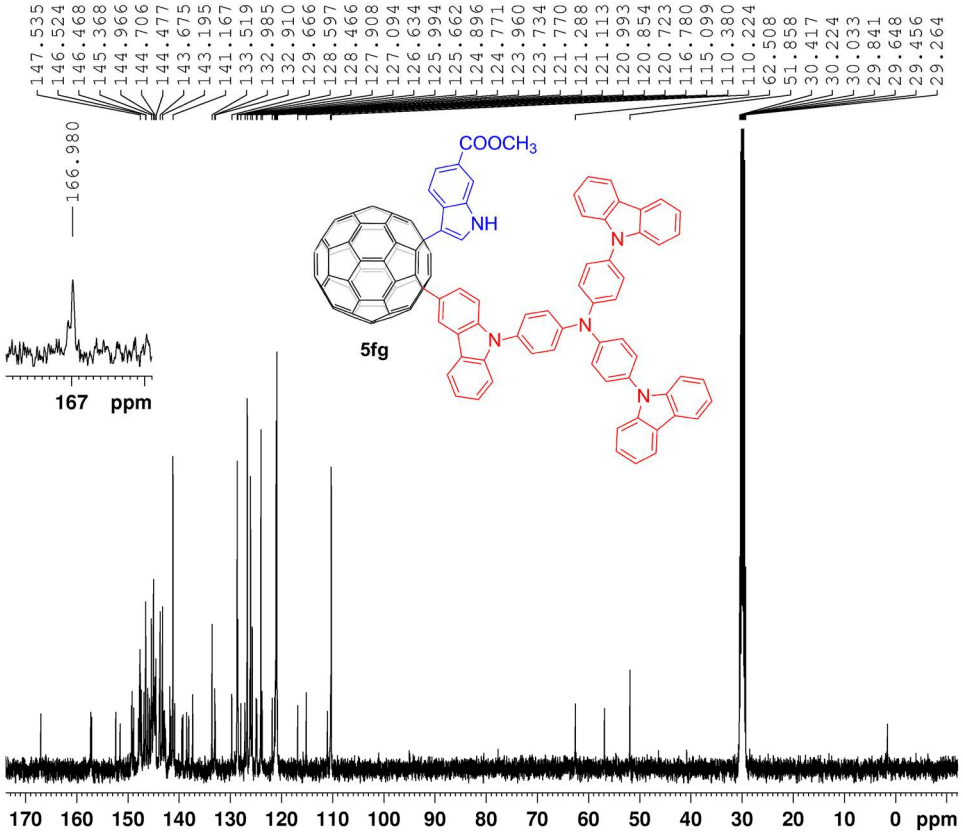
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 5fg




```
NAME            Mar31-2021
EXPNO                    1
PROCNO                   1
Date_             20210331
Time                 10.59
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT            Acetone
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      362
DW                   60.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.50 usec
PL1                  -2.00 dB
PL1W           13.45602226 W
SFO1          400.1324710 MHz
SI                    32768
SF            400.1300079 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5fg

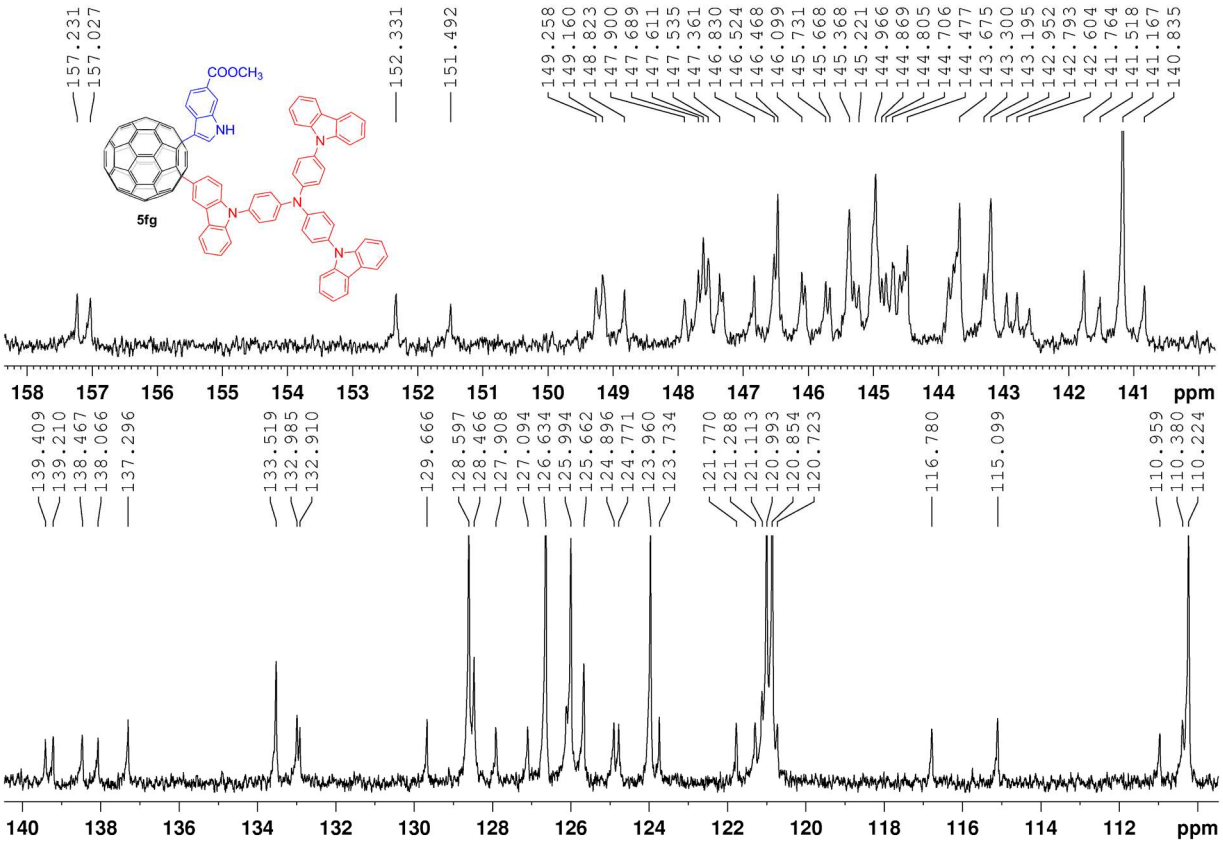



```
NAME            Mar31-2021
EXPNO                    2
PROCNO                   1
Date_             20210401
Time                 10.17
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                   20504
DS                       4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                      80.6
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

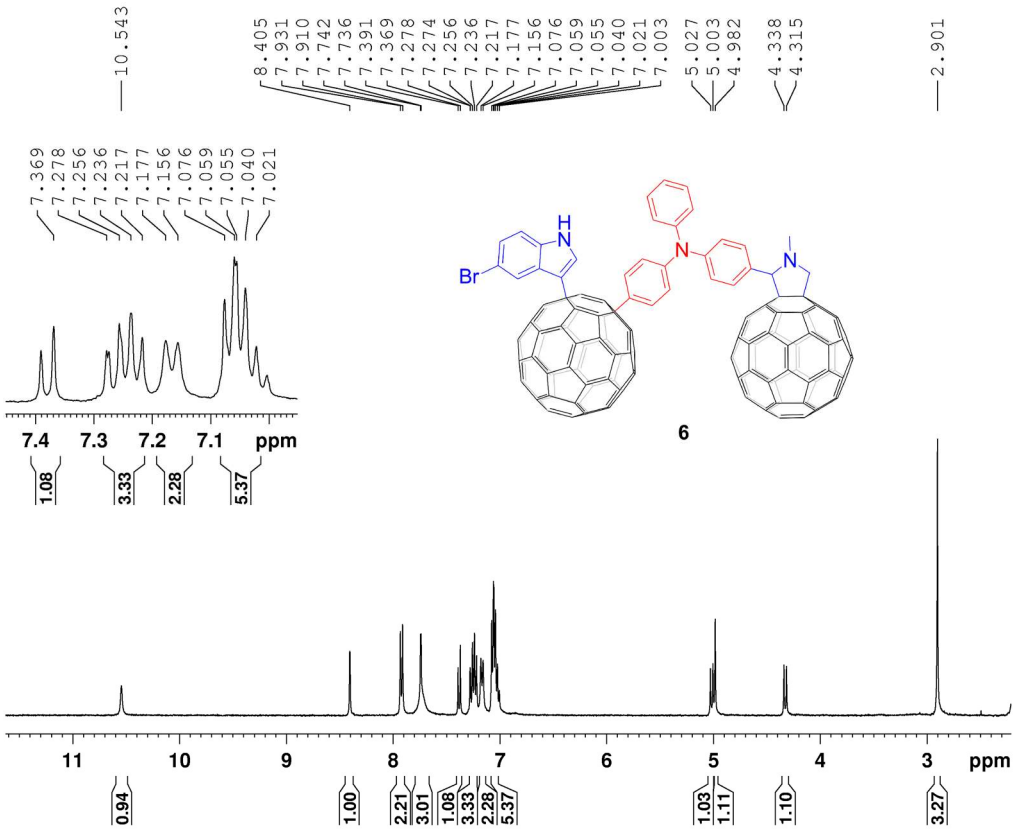
======== CHANNEL f1 ========
NUC1                   13C
P1                    9.30 usec
PL1                  -2.00 dB
PL1W           54.14257431 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 13.46 dB
PL13                120.00 dB
PL2W           13.45602226 W
PL12W           0.38275132 W
PL13W           0.00000000 W
SFO2          400.1316005 MHz
SI                    32768
SF            100.6127287 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 5fg



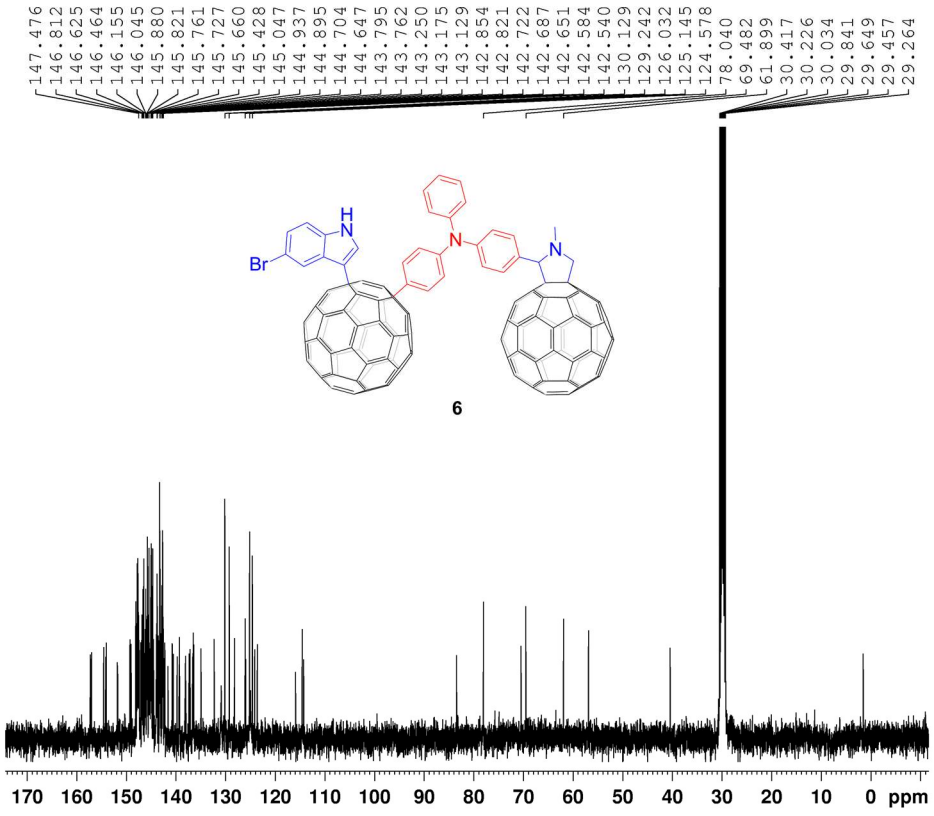
^{1}H NMR (400 MHz, CS_2/d_6-acetone) of compound 6




```
NAME            May14-2021
EXPNO                    1
PROCNO                   1
Date_             20210514
Time                16.39
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                      16
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     362
DW                60.800 usec
DE                 6.50 usec
TE                300.0 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.50 usec
PL1                -2.00 dB
PL1W         13.45602226 W
SFO1         400.1324710 MHz
SI                   32768
SF           400.1300076 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```

^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 6

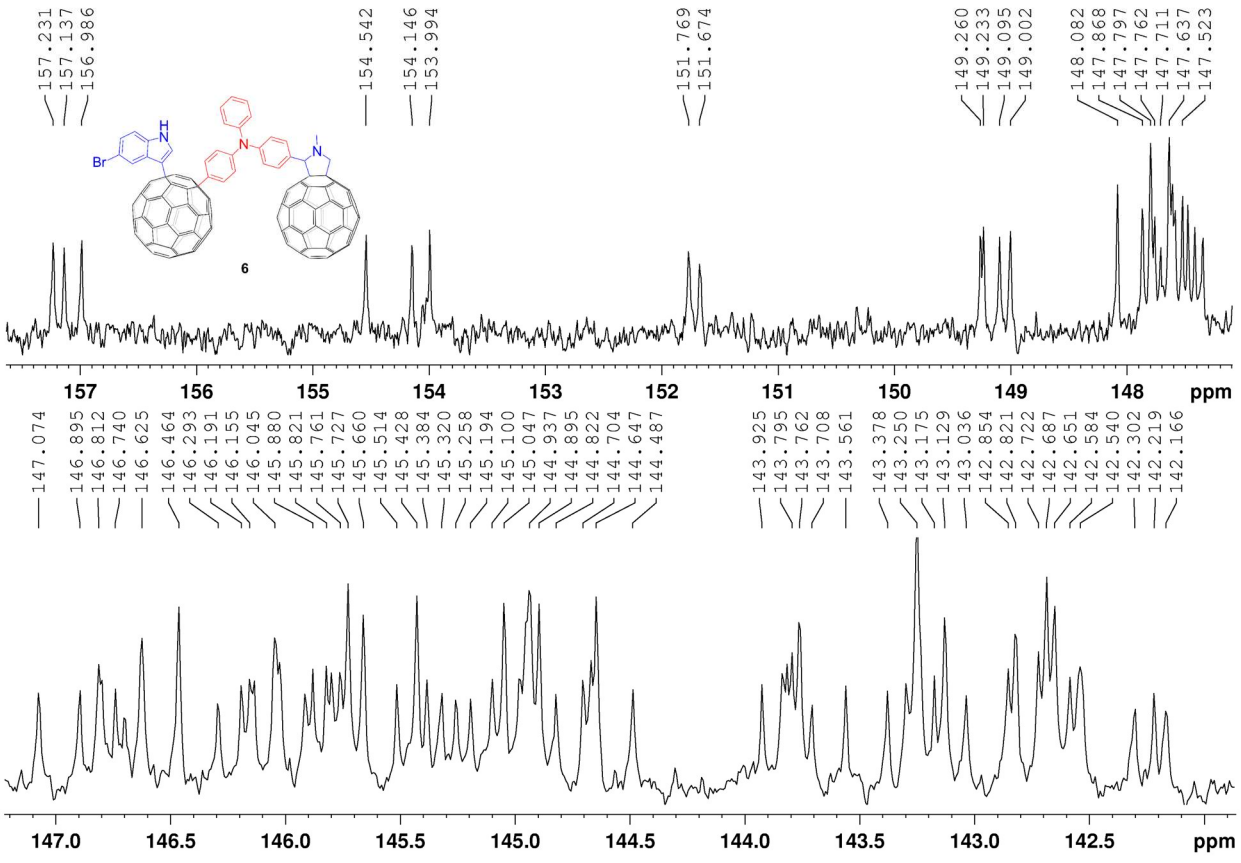



```
NAME          May14-2021
EXPNO                  2
PROCNO                 1
Date_           20210514
Time               16.42
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                 20480
DS                     4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                  80.6
DW               20.800 usec
DE                6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

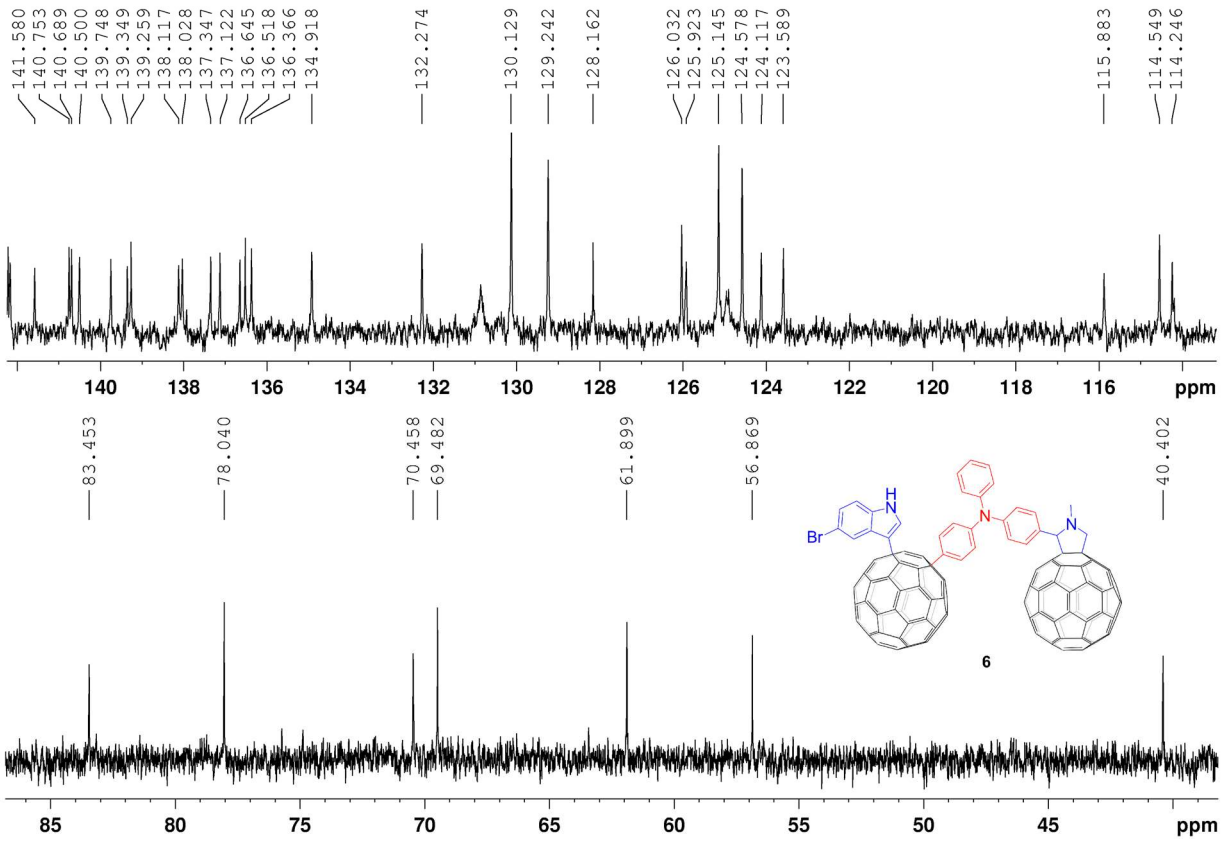
======== CHANNEL f1 ========
NUC1                 13C
P1                 9.30 usec
PL1                -2.00 dB
PL1W        54.14257431 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              13.46 dB
PL13             120.00 dB
PL2W        13.45602226 W
PL12W        0.38275132 W
PL13W        0.00000000 W
SFO2       400.1316005 MHz
SI                 32768
SF         100.6127224 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40
```

Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 6



Expanded ^{13}C NMR (100 MHz, CS_2/d_6-acetone) of compound 6



4. Single-Crystal X-Ray Crystallography of 3aa, 3ca, 5ad and 5af

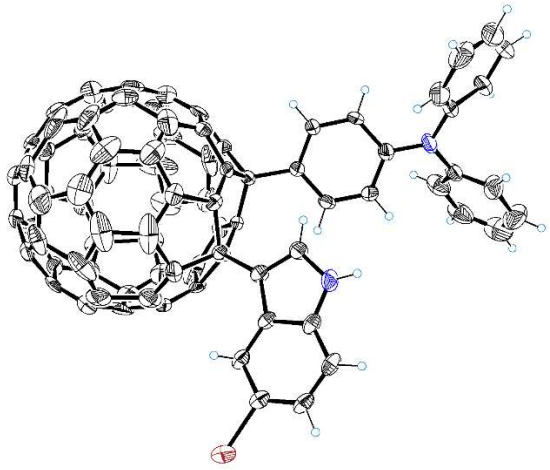


Figure S2 ORTEP diagrams of **3aa** with 30% thermal ellipsoids. The solvent molecules are omitted for clarity.

Black block crystals of **3aa** suitable for X-ray diffraction were obtained from slow evaporation of its solution in a mixture of CS_2 and toluene at room temperature. Single-crystal X-ray diffraction data were collected on a diffractometer (DECTRIS PILATUS 300K, STOE & Cie GmbH) equipped with a CCD area detector using

graphite-monochromated Cu Kα radiation (λ = 1.54184 Å) in the scan range 8.90° < 2θ < 140.22°. The structure was solved with direct methods using SHELXS-97 and refined with full-matrix least-squares refinement using the SHELXL-97 program within OLEX2. Crystallographic data have been deposited in the Cambridge Crystallographic Data Centre as deposition number CCDC 2080448.

Table S1 Crystal data and structure refinement for 3aa	
Identification code	3aa
Empirical formula	$C_{86}H_{19}BrN_2$
Formula weight	1159.94
Temperature/K	293(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	10.4943(4)
b/Å	16.3104(4)
c/Å	34.6451(9)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	5930.1(3)
Z	4
ρ_{calc}g/cm^3	1.299
μ/mm^{-1}	1.342
F(000)	2336.0
Crystal size/mm^3	0.28 × 0.25 × 0.16
Radiation	CuKα (λ = 1.54186)
2Θ range for data collection/°	12.624 to 139.126
Index ranges	-12 ≤ h ≤ 10, -19 ≤ k ≤ 19, -41 ≤ l ≤ 20
Reflections collected	21649
Independent reflections	10685 [R_{int} = 0.0274, R_{sigma} = 0.0320]
Data/restraints/parameters	10685/1/780
Goodness-of-fit on F^2	1.078
Final R indexes [I>=2σ (I)]	R_1 = 0.0802, wR_2 = 0.2334
Final R indexes [all data]	R_1 = 0.0900, wR_2 = 0.2504
Largest diff. peak/hole / e Å^{-3}	0.88/-0.60
Flack parameter	0.030(14)

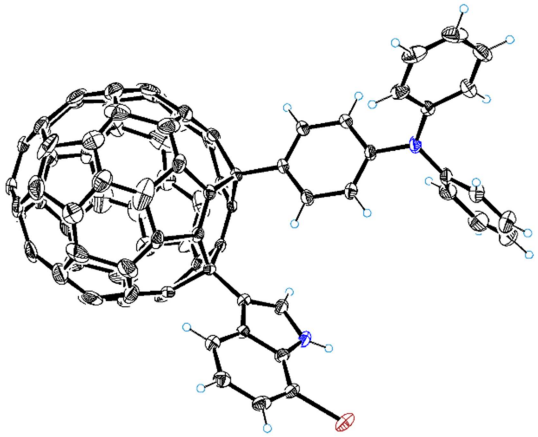


Figure S3 ORTEP diagrams of **3ca** with 30% thermal ellipsoids. The solvent molecules are omitted for clarity.

Black block crystals of **3ca** suitable for X-ray diffraction were obtained from slow evaporation of its solution in a mixture of CS_2 and isopropyl ether at room temperature. Single-crystal X-ray diffraction data were collected on a diffractometer (DECTRIS PILATUS 300K, STOE & Cie GmbH) equipped with a CCD area detector using graphite-monochromated Cu Kα radiation (λ = 1.54184 Å) in the scan range 8.90° < 2θ < 140.22°. The structure was solved with direct methods using SHELXS-97 and refined with full-matrix least-squares refinement using the SHELXL-97 program within OLEX2. Crystallographic data have been deposited in the Cambridge Crystallographic Data Centre as deposition number CCDC 2080456.

Table S2 Crystal data and structure refinement for 3ca	
Identification code	3ca
Empirical formula	$C_{92}H_{33}BrN_2O$
Formula weight	1262.11
Temperature/K	293(2)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	15.1762(2)
b/Å	23.7457(3)
c/Å	16.64667(19)
α/°	90
β/°	115.1948(15)
γ/°	90
Volume/Å^3	5428.24(13)
Z	4
ρ_{calc}g/cm^3	1.544
μ/mm^{-1}	1.528

F(000)	2568.0
Crystal size/mm^3	0.3 × 0.2 × 0.1
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	6.95 to 140.086
Index ranges	-12 ≤ h ≤ 18, -26 ≤ k ≤ 28, -20 ≤ l ≤ 14
Reflections collected	21686
Independent reflections	9852 [R_{int} = 0.0247, R_{sigma} = 0.0293]
Data/restraints/parameters	9852/31/857
Goodness-of-fit on F^2	1.056
Final R indexes [I>=2σ (I)]	R_1 = 0.0963, wR_2 = 0.2625
Final R indexes [all data]	R_1 = 0.1056, wR_2 = 0.2733
Largest diff. peak/hole / e Å^{-3}	1.59/-0.88

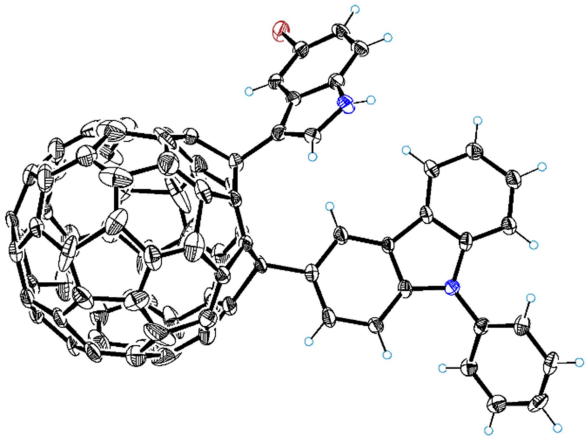


Figure S4 ORTEP diagrams of **5ad** with 30% thermal ellipsoids. The solvent molecules are omitted for clarity.

Black block crystals of **5ad** suitable for X-ray diffraction were obtained from slow evaporation of its solution in a mixture of CS_2 and *n*-hexane at room temperature. Single-crystal X-ray diffraction data were collected on a diffractometer (DECTRIS PILATUS 300K, STOE & Cie GmbH) equipped with a CCD area detector using graphite-monochromated Cu Kα radiation (λ = 1.54184 Å) in the scan range 8.90° < 2θ < 140.22°. The structure was solved with direct methods using SHELXS-97 and refined with full-matrix least-squares refinement using the SHELXL-97 program within OLEX2. Crystallographic data have been deposited in the Cambridge Crystallographic Data Centre as deposition number CCDC 2080460.

Table S3 Crystal data and structure refinement for 5ad	
Identification code	5ad
Empirical formula	$C_{173}H_{34}Br_2N_4S_2$
Formula weight	2391.98
Temperature/K	293(2)
Crystal system	triclinic
Space group	P-1
a/Å	13.8089(3)
b/Å	19.3737(5)
c/Å	23.3923(6)
α/°	66.123(2)
β/°	79.414(2)
γ/°	70.079(2)
Volume/Å^3	5372.5(3)
Z	2
ρ_{calc}g/cm^3	1.479
μ/mm^{-1}	1.854
F(000)	2404.0
Crystal size/mm^3	0.23 × 0.19 × 0.14
Radiation	CuKα (λ = 1.54186)
2Θ range for data collection/°	7.126 to 139.408
Index ranges	-14 ≤ h ≤ 16, -19 ≤ k ≤ 23, -12 ≤ l ≤ 27
Reflections collected	51212
Independent reflections	19362 [R_{int} = 0.0221, R_{sigma} = 0.0292]
Data/restraints/parameters	19362/26/1630
Goodness-of-fit on F^2	1.047
Final R indexes [I>=2σ (I)]	R_1 = 0.0749, wR_2 = 0.1994
Final R indexes [all data]	R_1 = 0.0938, wR_2 = 0.2201
Largest diff. peak/hole / e Å^{-3}	1.10/-1.00

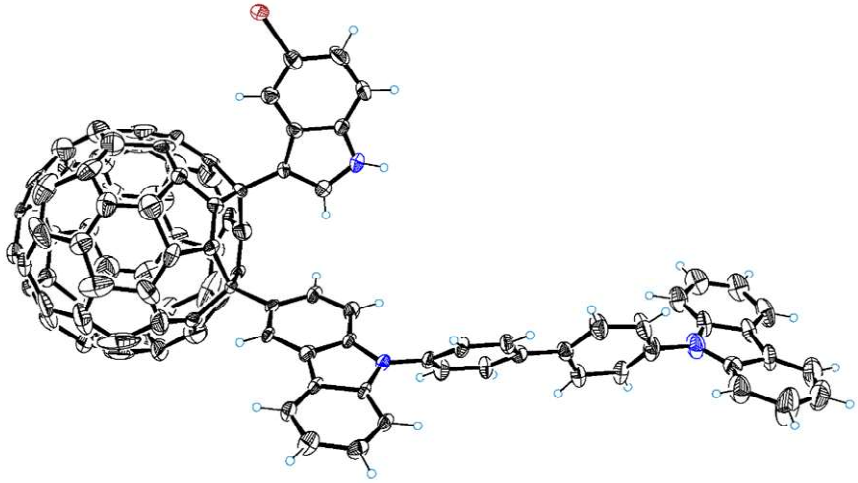


Figure S5 ORTEP diagrams of **5af** with 30% thermal ellipsoids. The solvent molecules are omitted for clarity.

Black block crystals of **5af** suitable for X-ray diffraction were obtained from slow evaporation of its solution in a mixture of CS_2 and toluene at room temperature. Single-crystal X-ray diffraction data were collected on a diffractometer (DECTRIS PILATUS 300K, STOE & Cie GmbH) equipped with a CCD area detector using graphite-monochromated Cu Kα radiation (λ = 1.54184 Å) in the scan range 8.90° < 2θ < 140.22°. The structure was solved with direct methods using SHELXS-97 and refined with full-matrix least-squares refinement using the SHELXL-97 program within OLEX2. Crystallographic data have been deposited in the Cambridge Crystallographic Data Centre as deposition number CCDC 2080463.

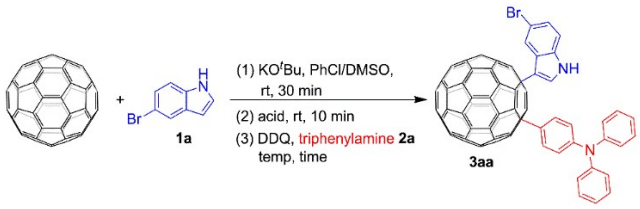
Table S4 Crystal data and structure refinement for 5af	
Identification code	5af
Empirical formula	$C_{104}H_{28}BrN_3$
Formula weight	1399.20
Temperature/K	298
Crystal system	triclinic
Space group	P-1
a/Å	13.1196(10)
b/Å	17.4041(17)
c/Å	17.8966(15)
α/°	95.025(7)

β/°	99.017(6)
γ/°	100.517(7)
Volume/Å^3	3939.6(6)
Z	2
ρ_{calc}g/cm^3	1.180
μ/mm^{-1}	1.101
F(000)	1416.0
Crystal size/mm^3	0.20 × 0.15 × 0.13
Radiation	CuKα (λ = 1.54186)
2Θ range for data collection/°	12.402 to 136.718
Index ranges	-15 ≤ h ≤ 15, -20 ≤ k ≤ 6, -20 ≤ l ≤ 21
Reflections collected	29646
Independent reflections	13723 [R_{int} = 0.0341, R_{sigma} = 0.0312]
Data/restraints/parameters	13723/56/521
Goodness-of-fit on F^2	1.215
Final R indexes [I>=2σ (I)]	R_1 = 0.1089, wR_2 = 0.2642
Final R indexes [all data]	R_1 = 0.1280, wR_2 = 0.2920
Largest diff. peak/hole / e Å^{-3}	1.04/-0.42

5. Optimization of the Reaction Conditions

The three-component cross-coupling of 5-bromoindole (**1a**) with C_{60} and triphenylamine (**2a**) was selected as a model reaction for the optimization of reaction conditions. On the basis of our previous research work, which has realized the KO^{t}Bu promoted C3-coupling of indole with C_{60}, we commenced our study by examining whether the following regioselective C-C cross-coupling between triphenylamine and C_{60} could be achieved in one-pot (Table S5). To our delight, the desired 1,4-adducted indole-C_{60}-TPA compound **3aa** was obtained at 100 °C for 1 hour in 35% yield when **2a** was added with DDQ as the oxidant in the presence of CF_3SO_3H (Table S5, entry 1). Further condition optimization revealed that increasing the temperature could improved the yield obviously (Table S5, entry 2-5), especially carrying the reaction at 130 °C for 1 hour resulted in raising the yield of **3aa** to 66% (Table S5, entry 5). Then the solvent effect was investigated, and we found that change the ratio of solvents PhCl/DMSO (v/v) from 4:1 to 2:1 or 1:1 could not provide the desired product in superior yields (Table S5, entry 5 vs 6, 7). Different acids, such as CF_3COOH, CH_3COOH and H_2SO_4 were screened (Table S5, entries 8–10). The results revealed that CF_3SO_3H was an efficient acid for the transformation.

Table S5. Optimization of Reaction Conditions[a]



entry[a]	acid	solvent (v/v) (PhCl/DMSO)	temp[b] (°C)	time (h)	yield (%)[c]
1	CF_3SO_3H	4:1	100	1	35 (44)
2	CF_3SO_3H	4:1	110	1	45 (52)
3	CF_3SO_3H	4:1	120	1	54 (70)
4	CF_3SO_3H	4:1	120	2	63 (70)
5	**CF_3SO_3H**	**4:1**	**130**	**1**	**66 (77)**
6	CF_3SO_3H	2:1	130	1	53 (66)
7	CF_3SO_3H	1:1	130	1	50 (58)
8	CF_3COOH	4:1	130	1	17(20)
9	CH_3COOH	4:1	130	1	trace
10	H_2SO_4	4:1	130	1	19(23)

[a]Reactions were carried out using C_{60} (0.05 mmol, 1 equiv), 5-bromoindole (0.06 mmol, 1.2 equiv), KO^{t}Bu (0.1 mmol, 2.0 equiv) in Schlenk tubes under Ar atmosphere for 30 min, and acid (1.0 mmol, 20 equiv) was added and stirred for another 10 min, then DDQ (0.25 mmol, 5 equiv) and triphenylamine (0.25 mmol, 5 equiv) were added and stirred in an oil bath. [b]Oil temperature. [c]Isolated yield by column chromatography. Values in parentheses were based on consumed C_{60}.

6. UV-vis absorption spectra of compounds 3aa, 3ba, 3fa, 3ga, 3ha, 3ae, 3af, 3ah, 3ai, 3ak, 5ab, 5ad, 5af, 5ag and 5fg

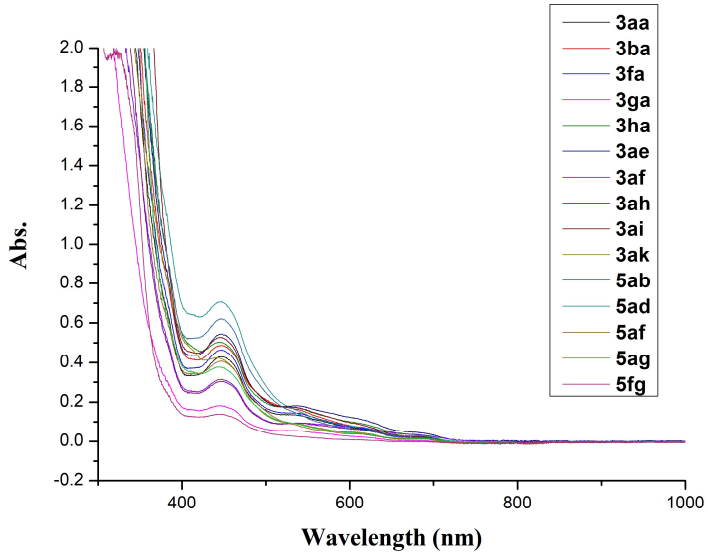
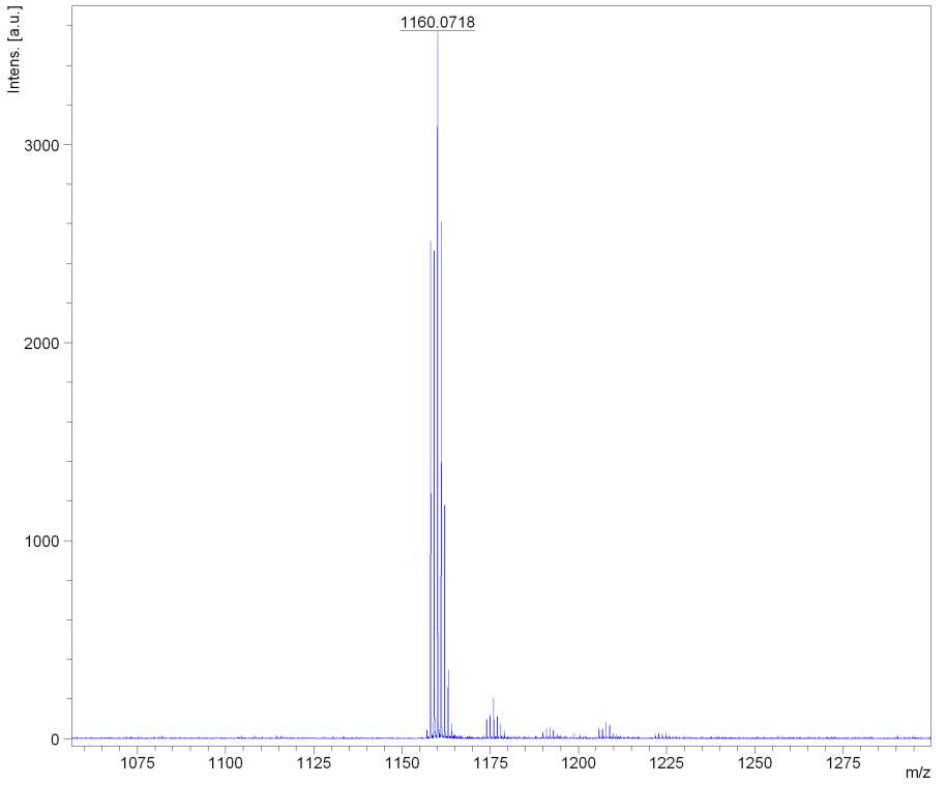


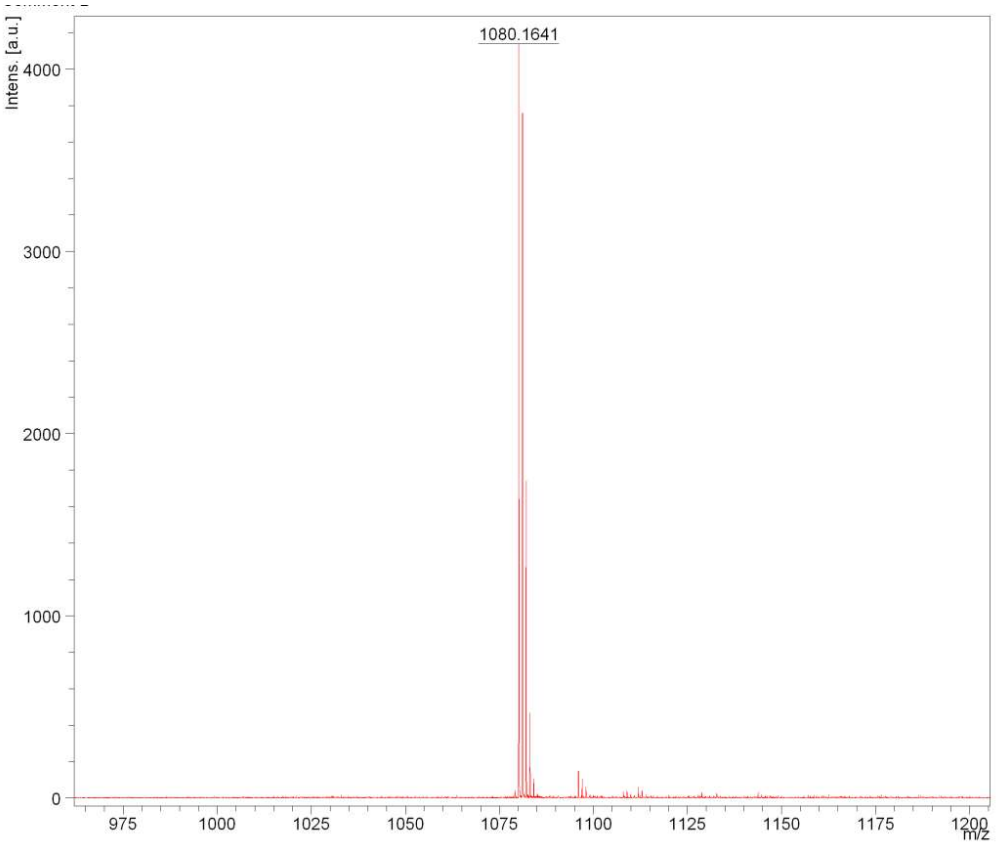
Figure S6 UV-vis spectra of representative TPA/carbazole-fullerene-indole products

7. The MALDI-TOF-MS spectra of 3aa-3ak, 5aa-5fg

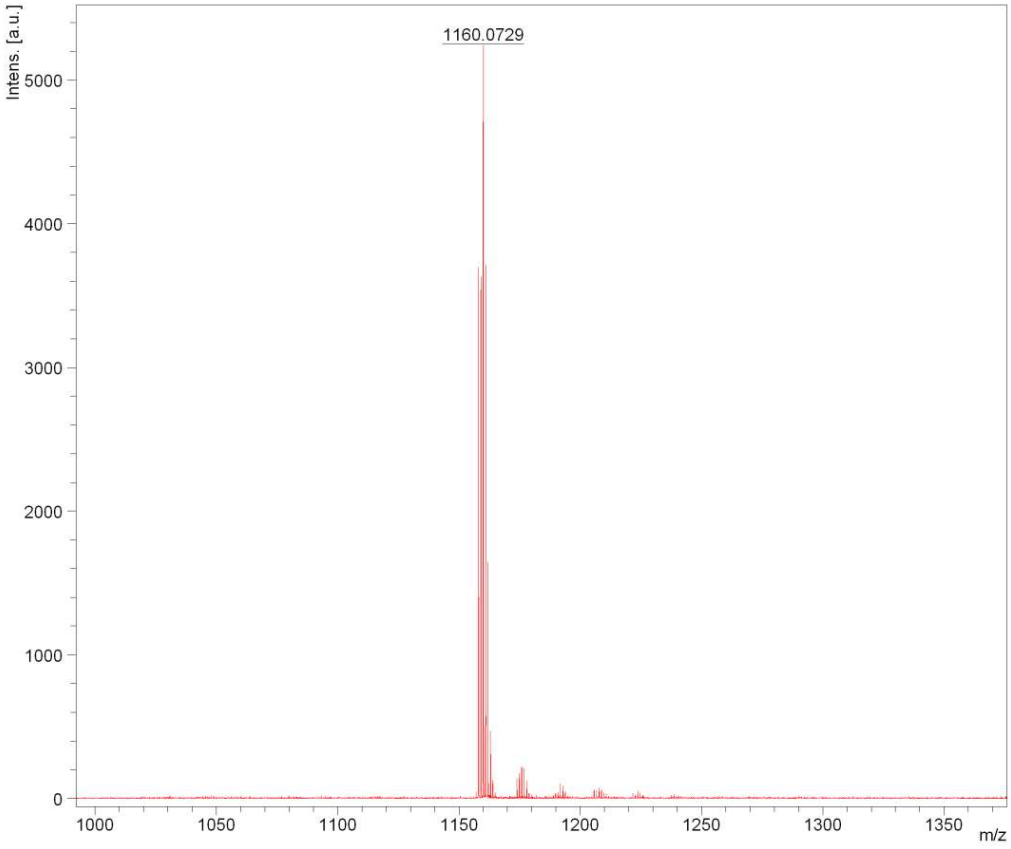
The MALDI-TOF-MS spectrum of **3aa**



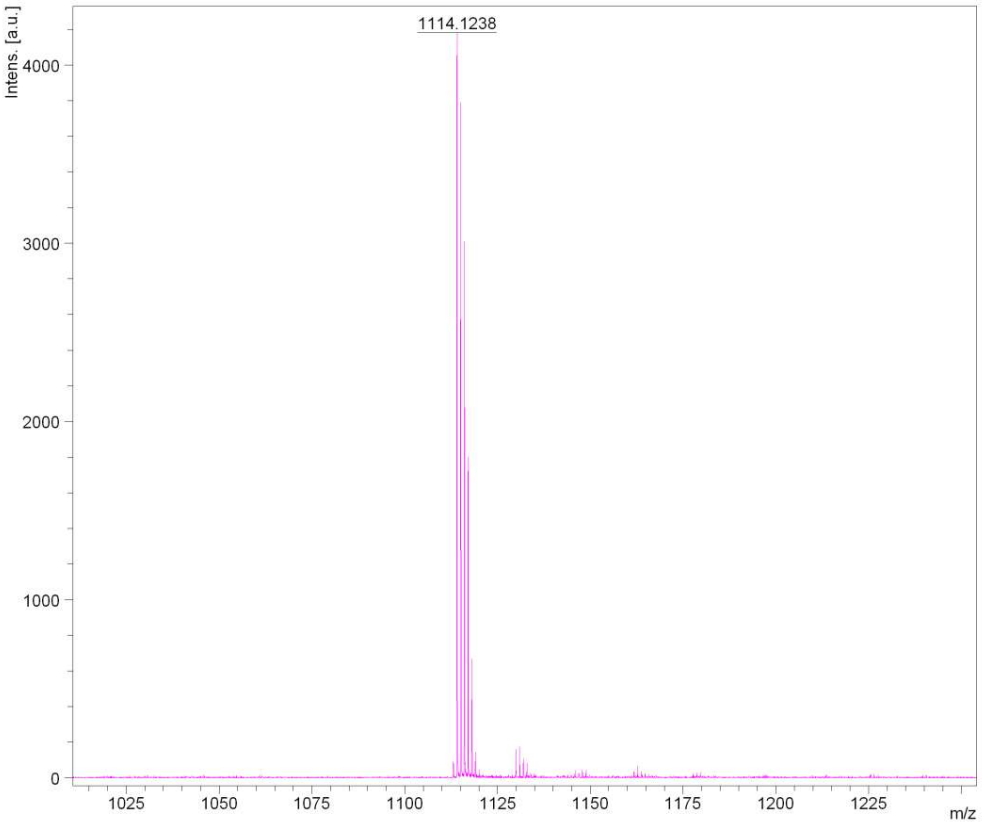
The MALDI-TOF-MS spectrum of **3ba**



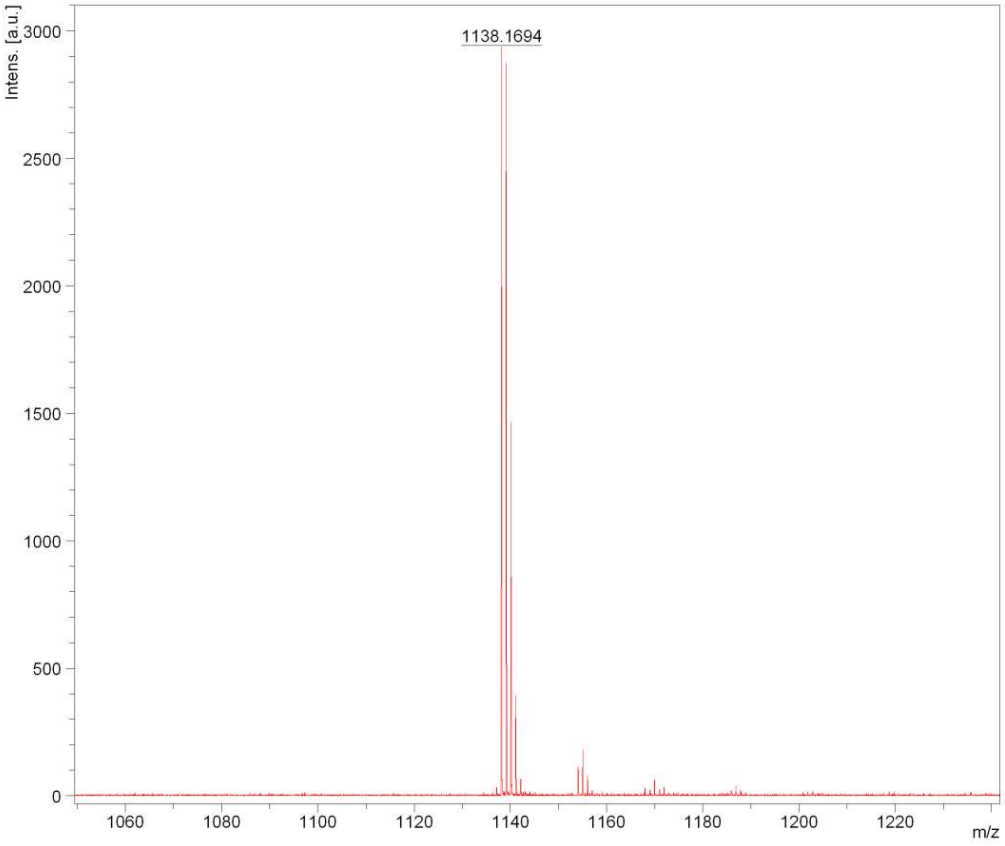
The MALDI-TOF-MS spectrum of **3ca**



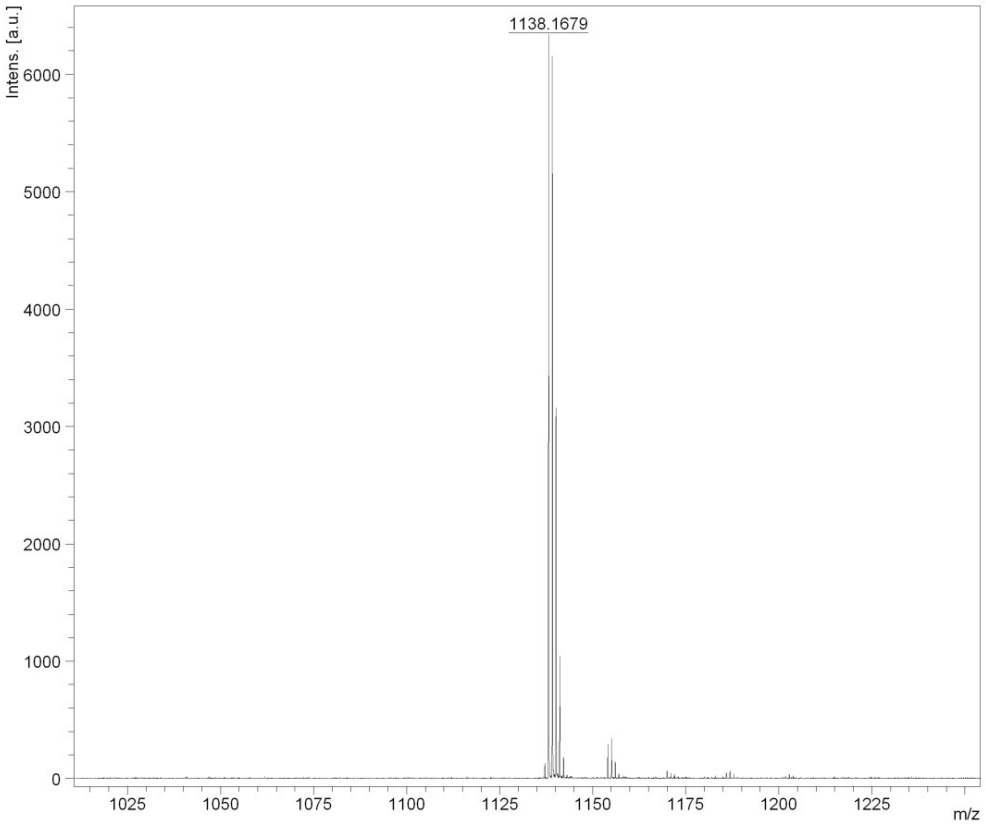
The MALDI-TOF-MS spectrum of **3da**



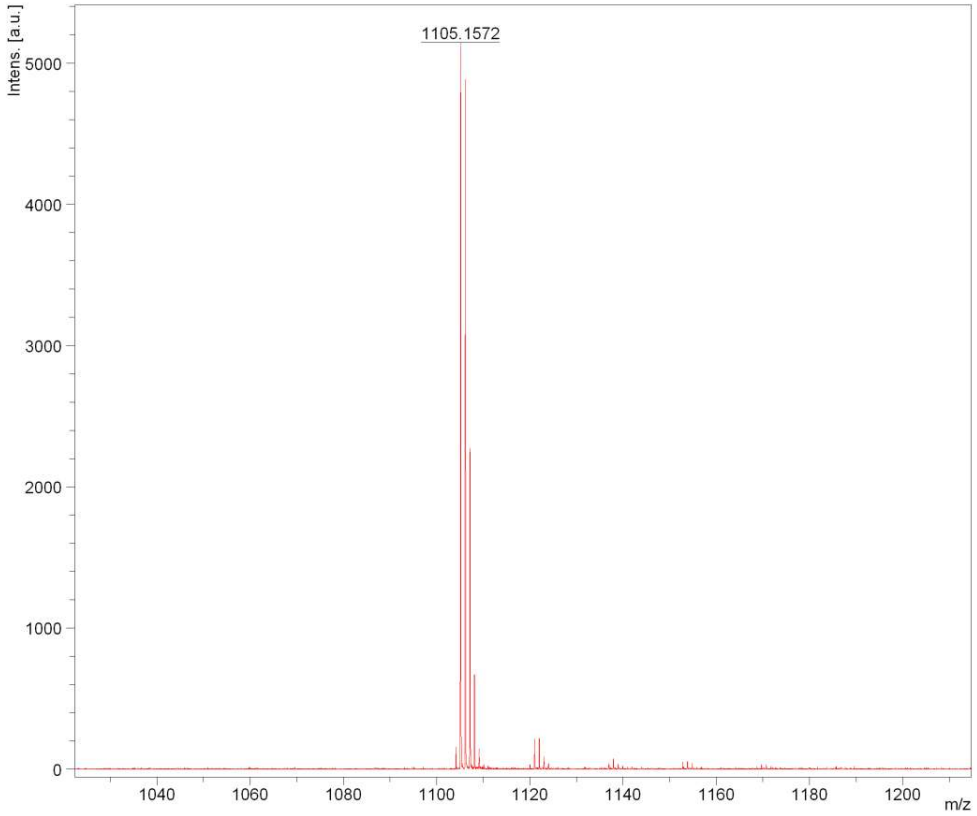
The MALDI-TOF-MS spectrum of **3ea**



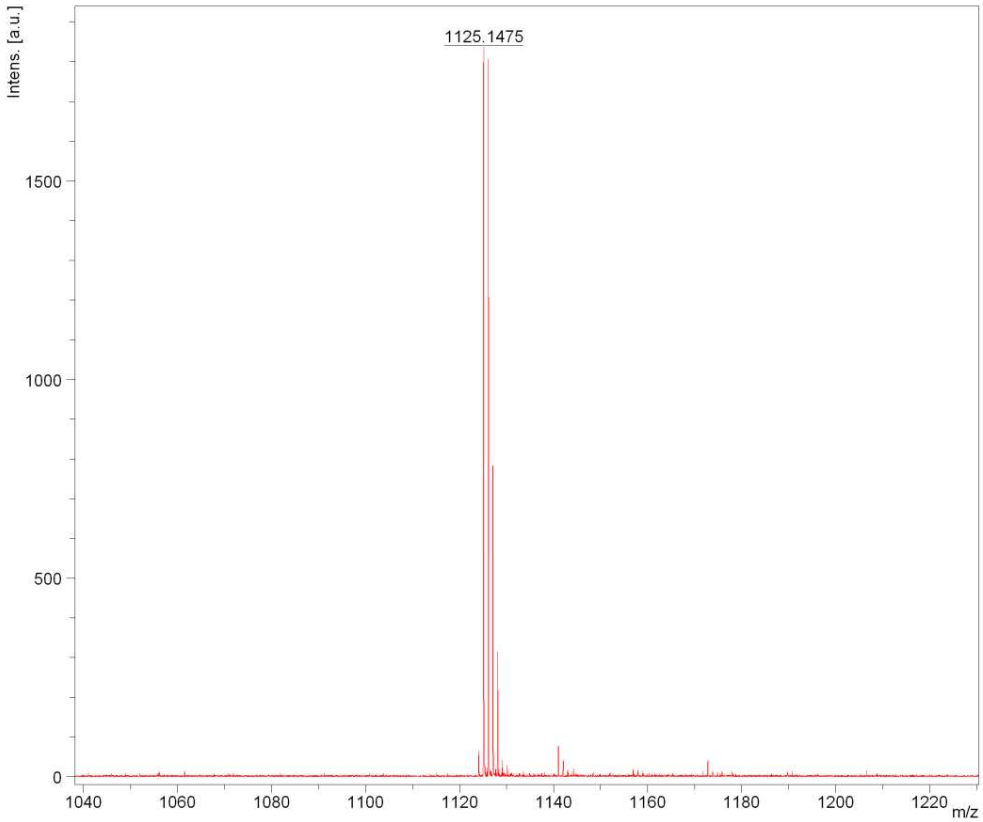
The MALDI-TOF-MS spectrum of **3fa**



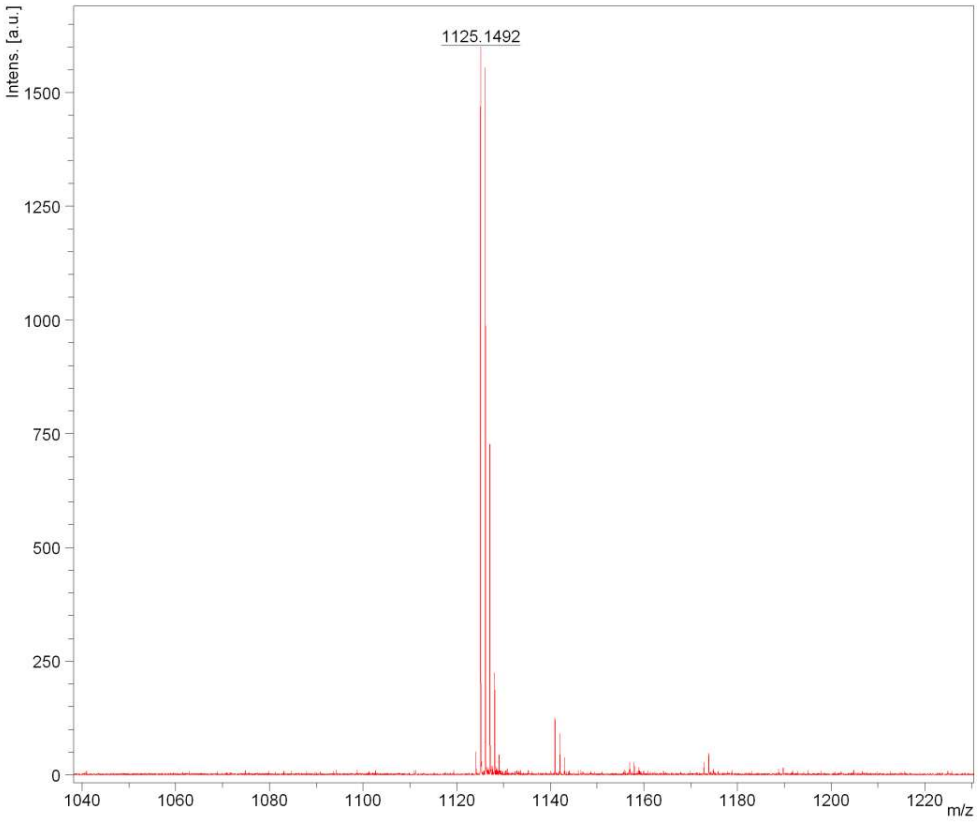
The MALDI-TOF-MS spectrum of **3ga**



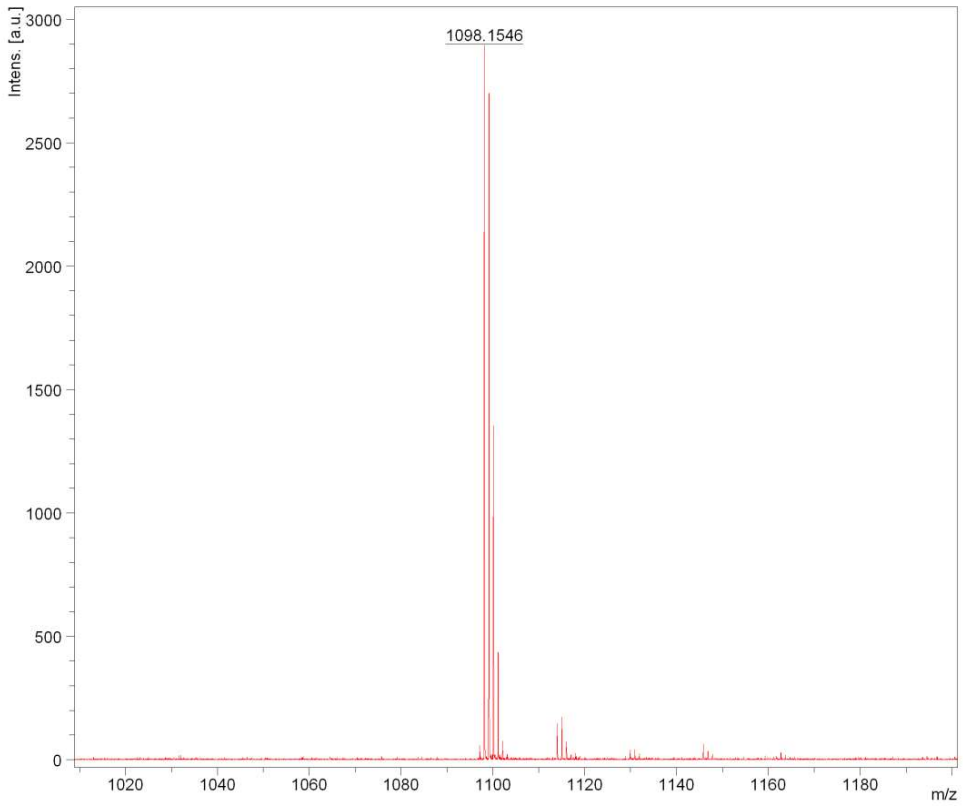
The MALDI-TOF-MS spectrum of **3ha**



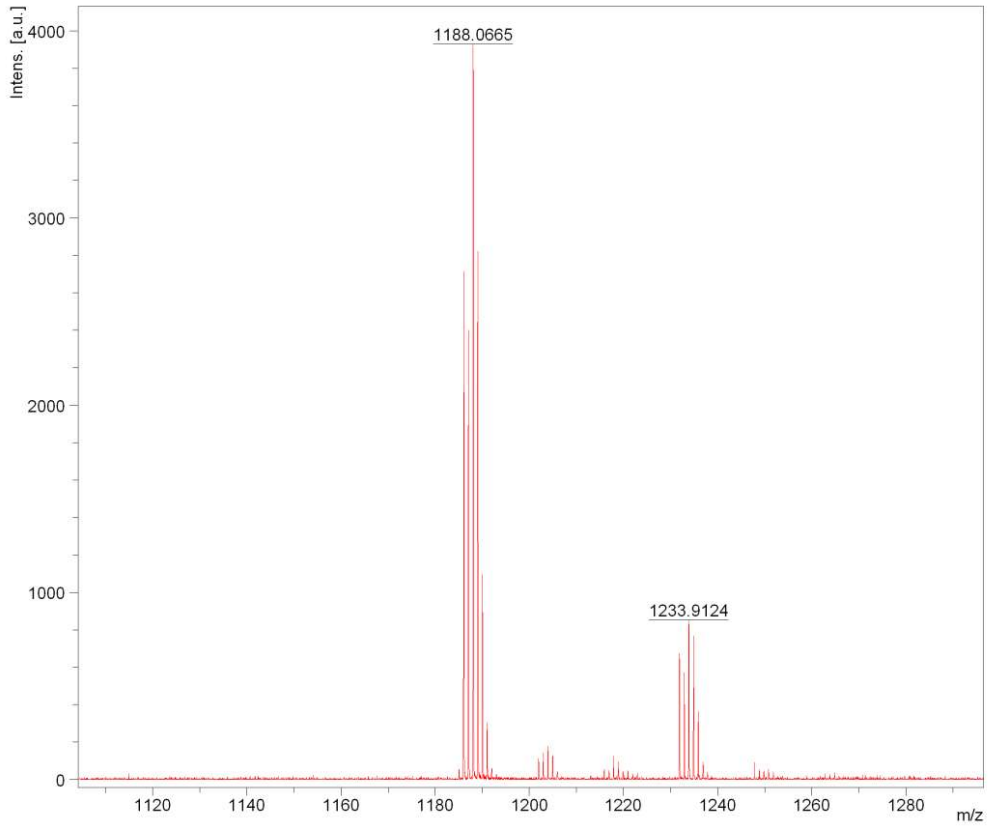
The MALDI-TOF-MS spectrum of **3ia**



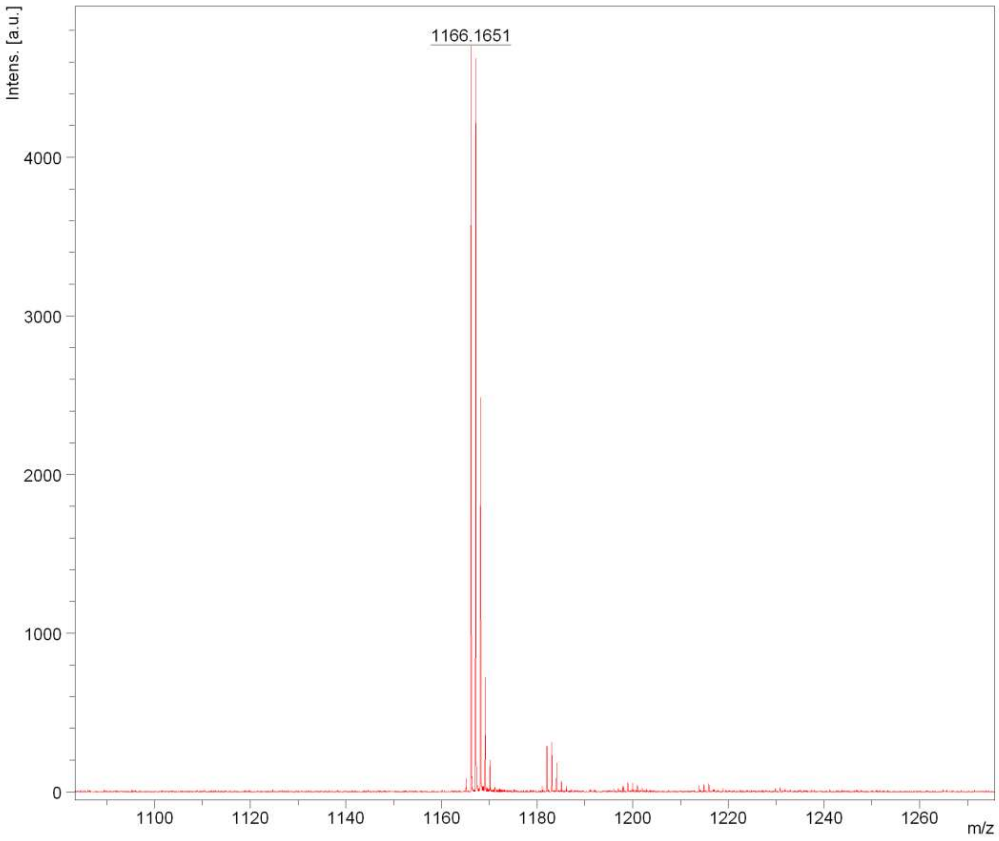
The MALDI-TOF-MS spectrum of **3ja**



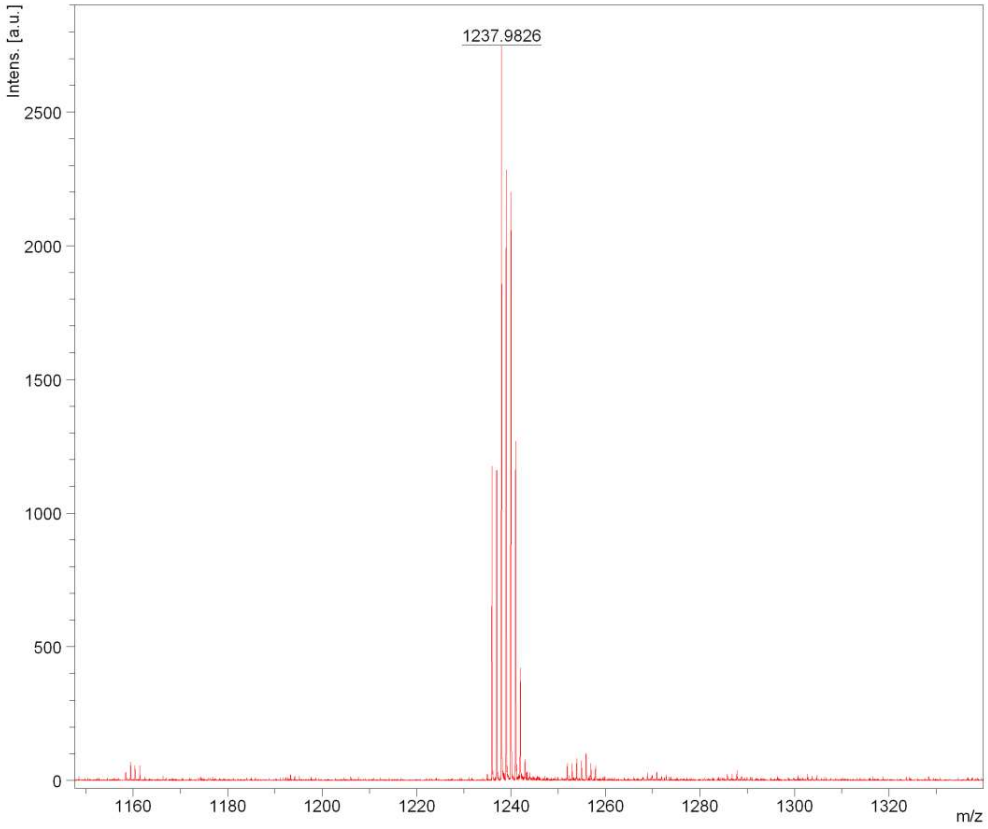
The MALDI-TOF-MS spectrum of **3ab**



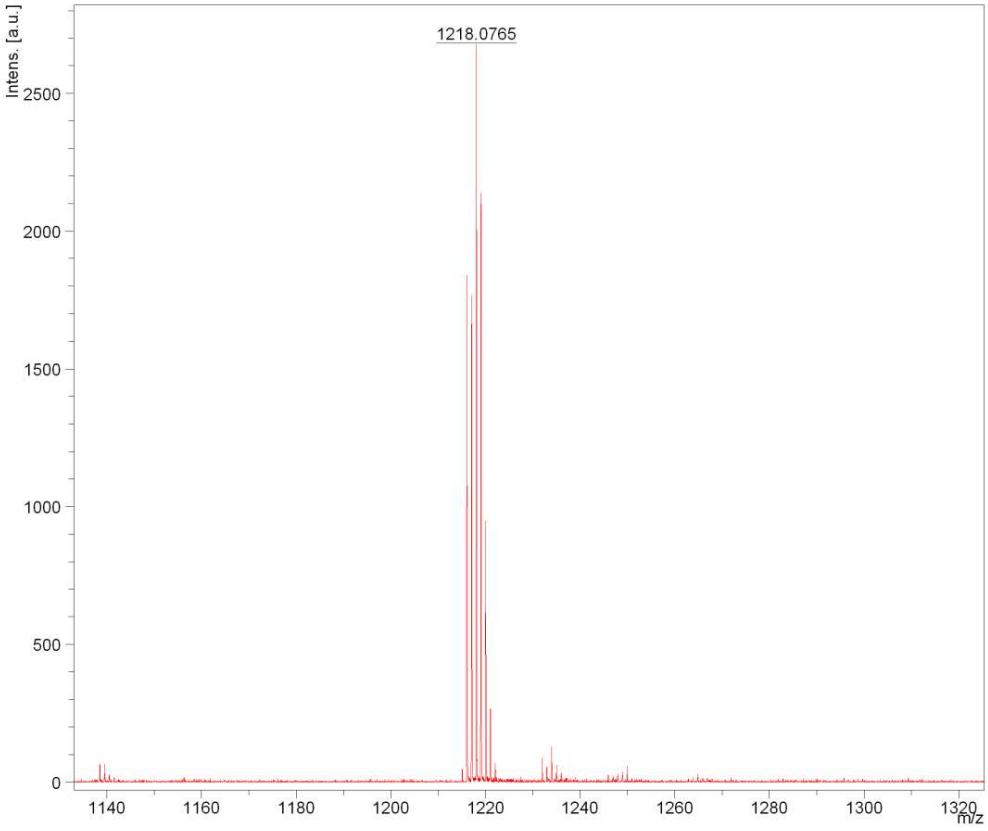
The MALDI-TOF-MS spectrum of **3fb**



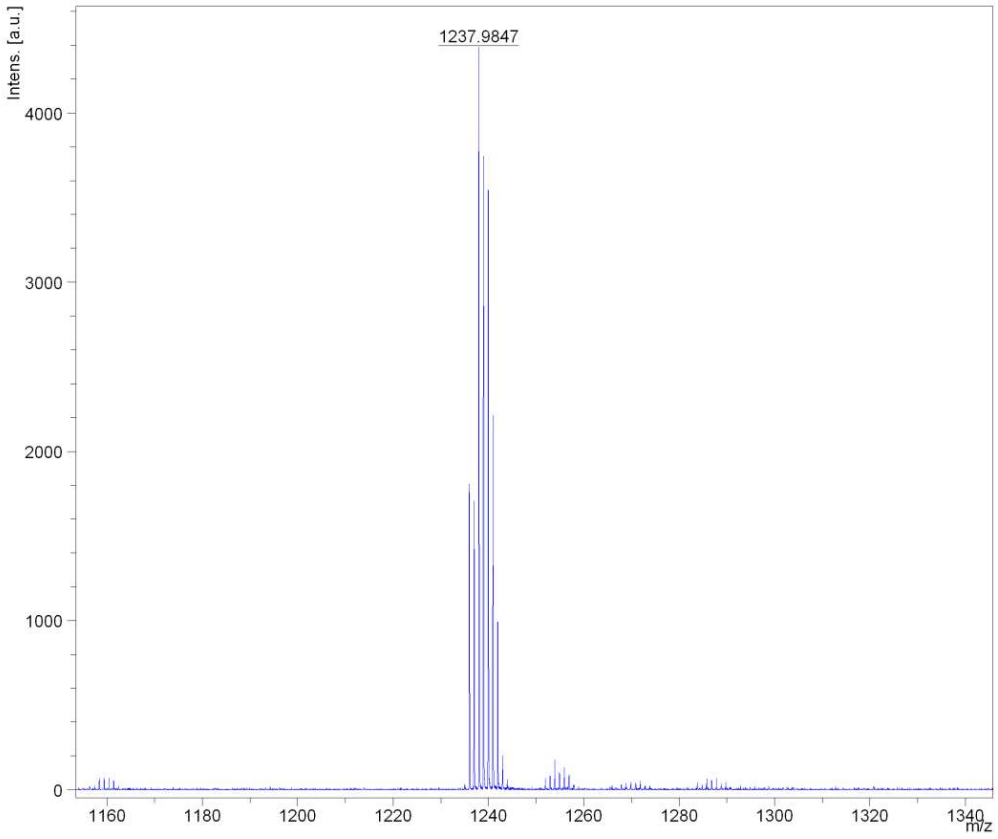
The MALDI-TOF-MS spectrum of **3ac**



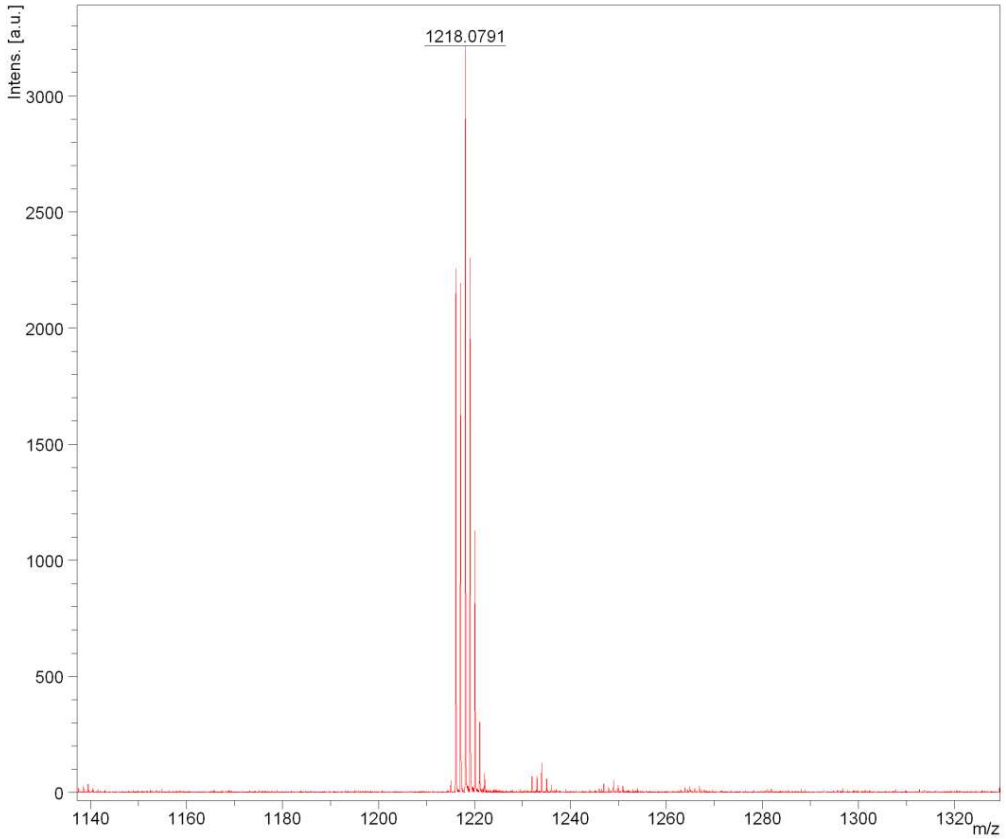
The MALDI-TOF-MS spectrum of **3fc**



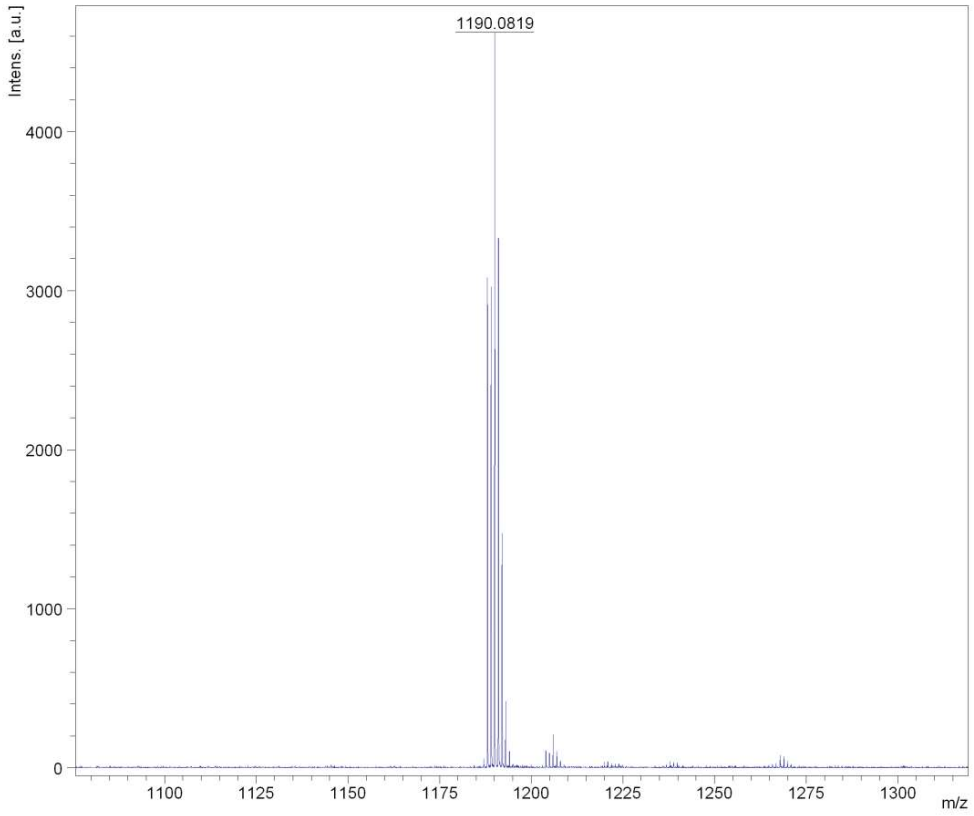
The MALDI-TOF-MS spectrum of **3ad**



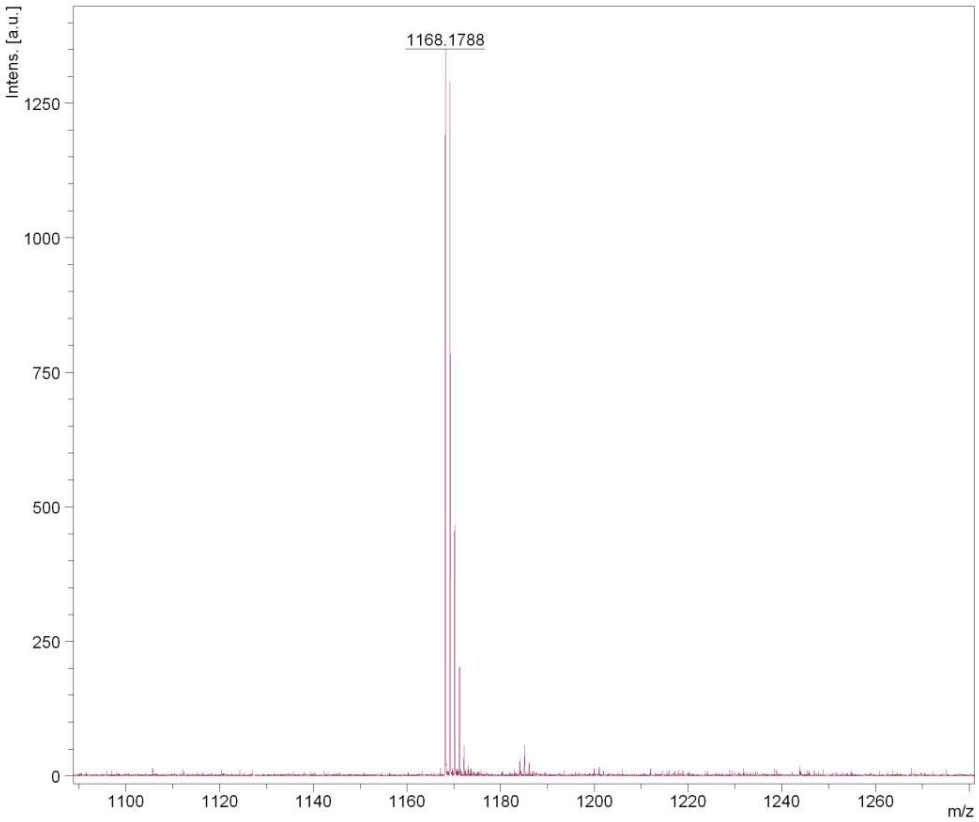
The MALDI-TOF-MS spectrum of **3fd**



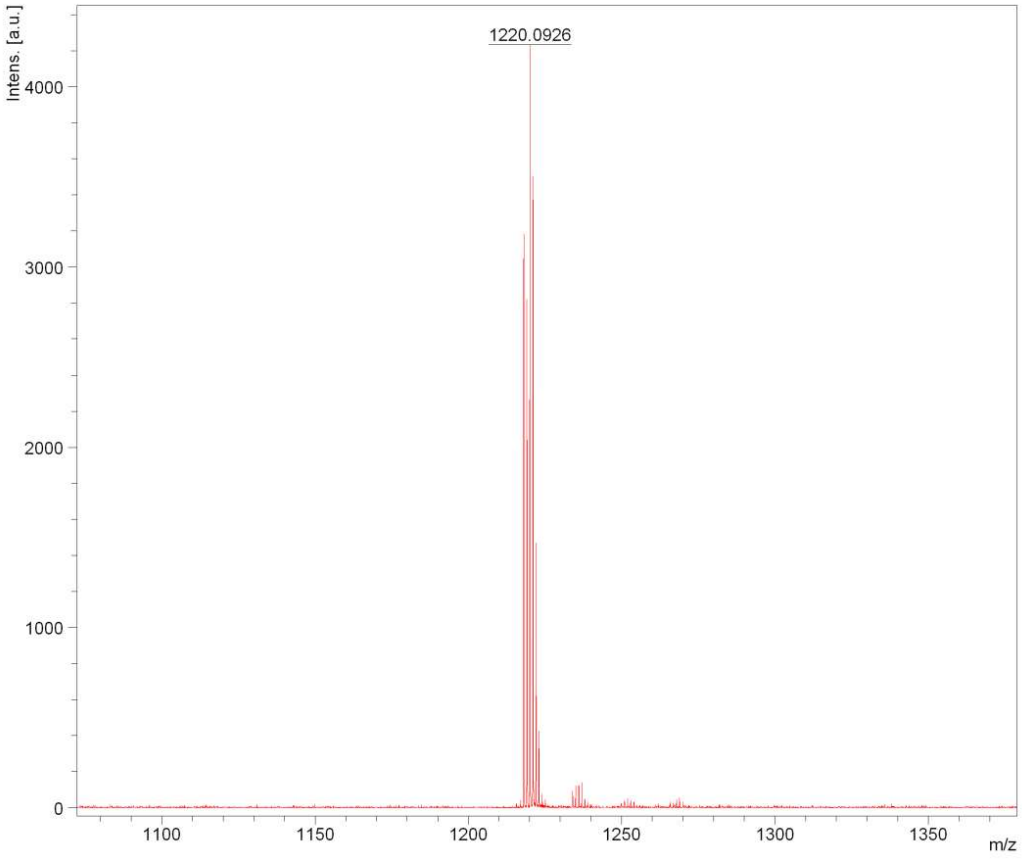
The MALDI-TOF-MS spectrum of **3ae**



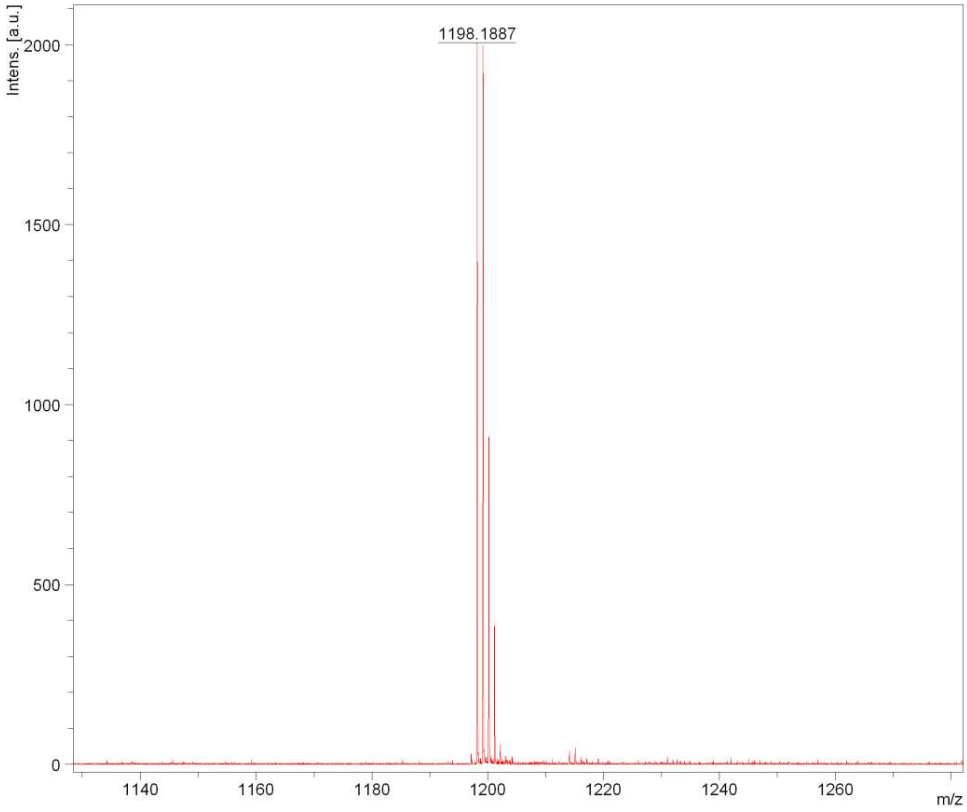
The MALDI-TOF-MS spectrum of **3fe**



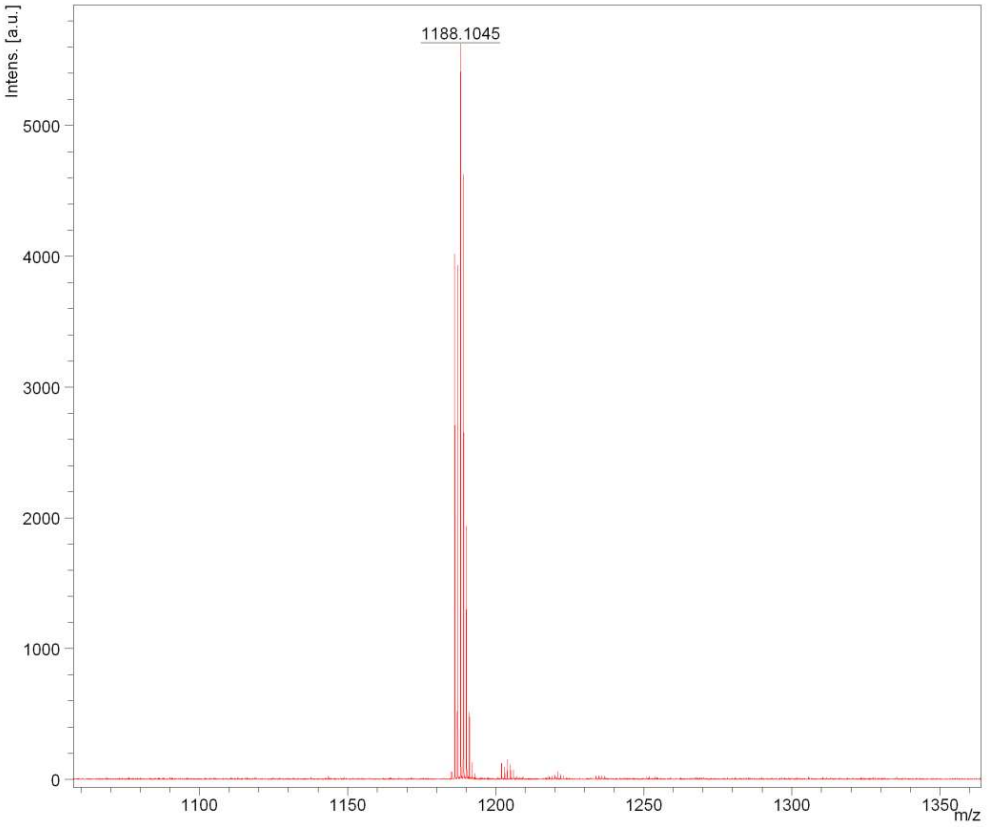
The MALDI-TOF-MS spectrum of **3af**



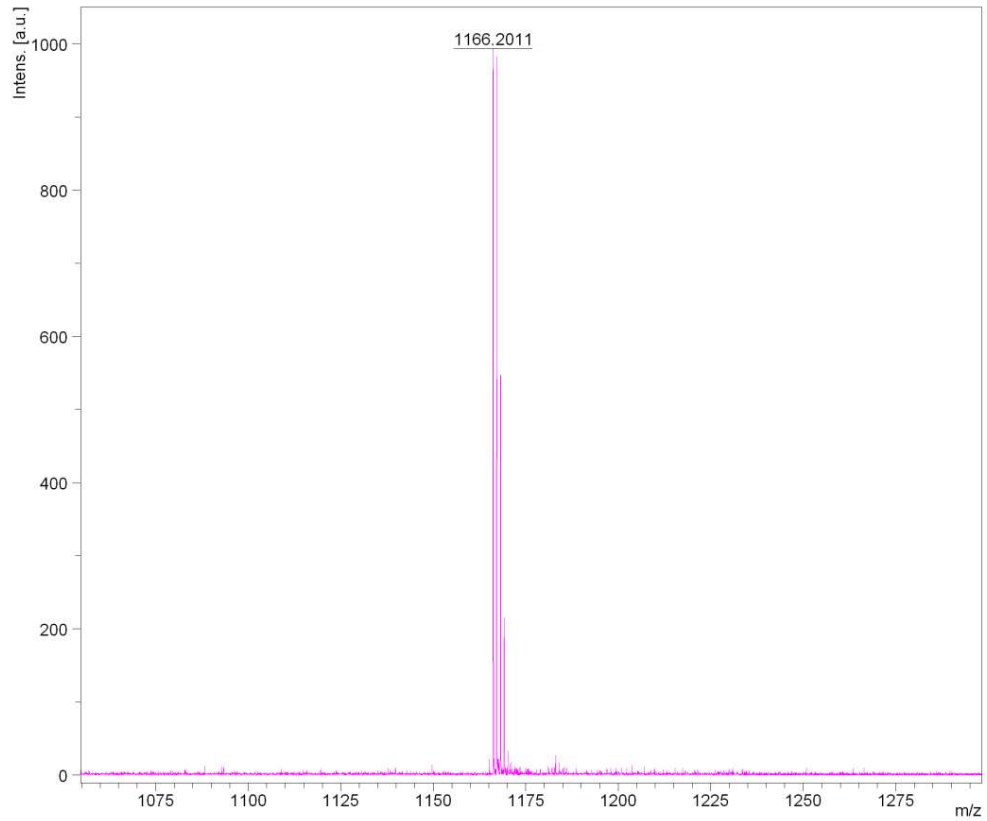
The MALDI-TOF-MS spectrum of **3ff**



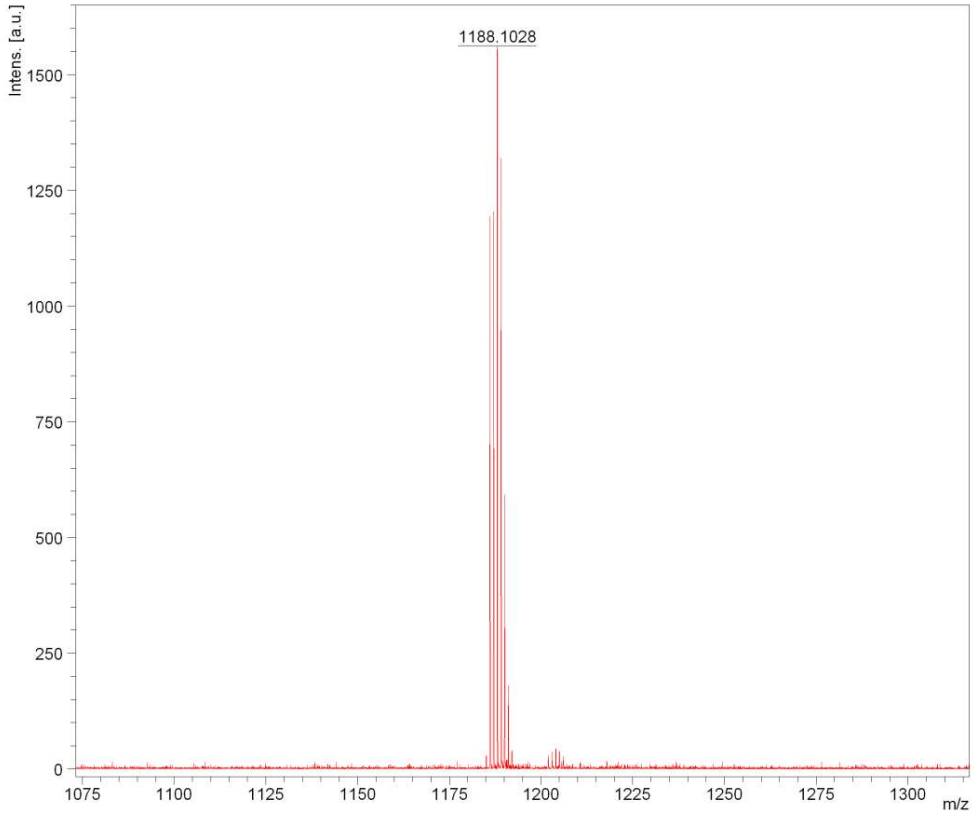
The MALDI-TOF-MS spectrum of **3ag**



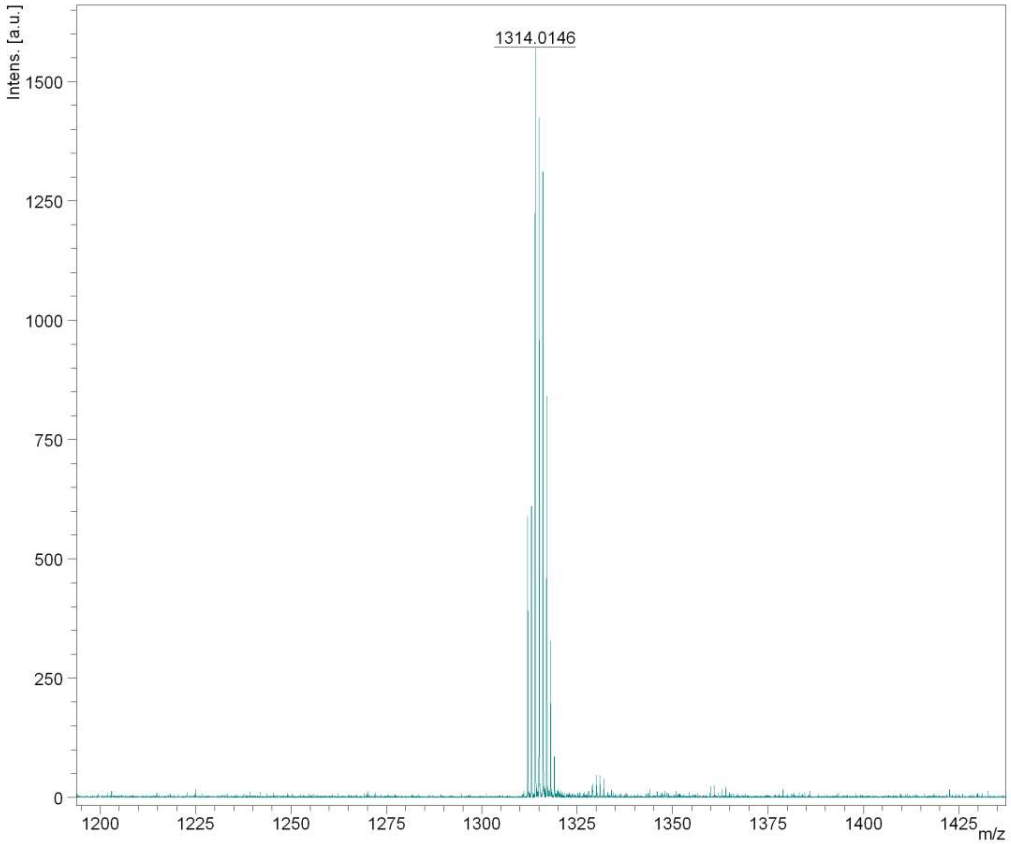
The MALDI-TOF-MS spectrum of **3fg**



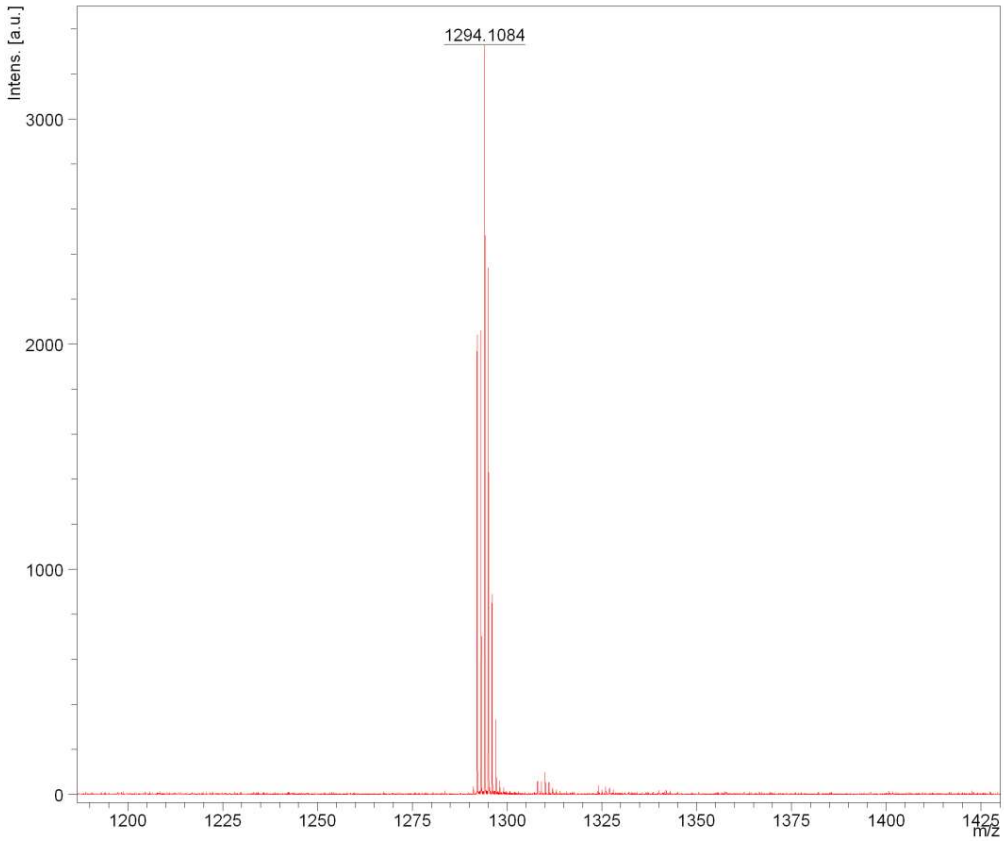
The MALDI-TOF-MS spectrum of **3ah**



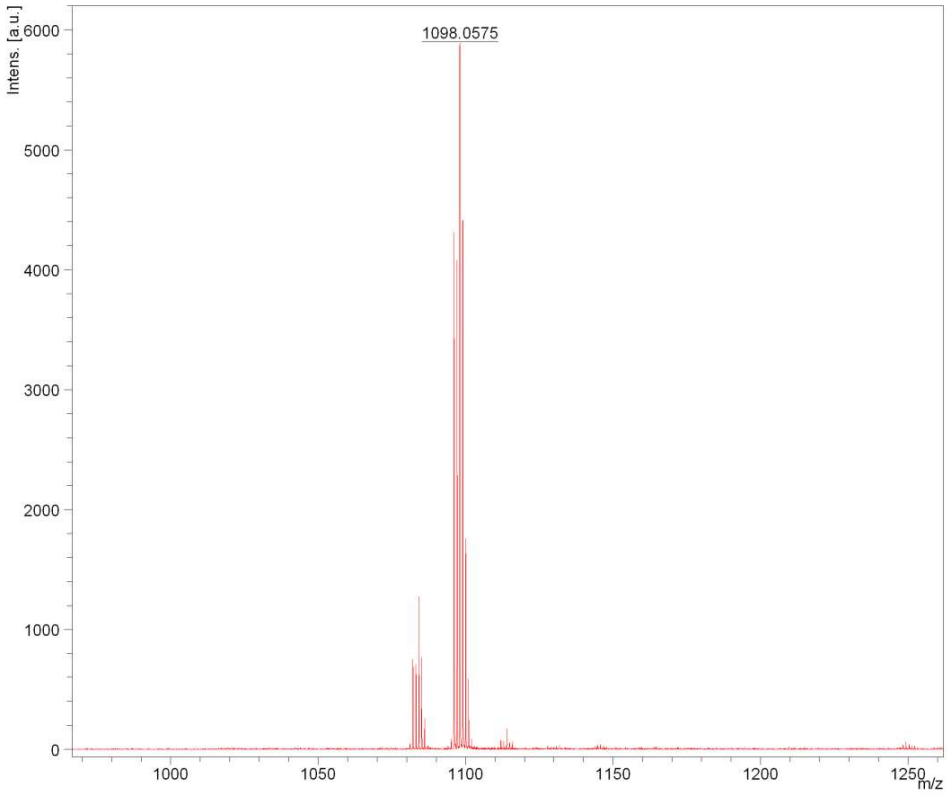
The MALDI-TOF-MS spectrum of **3ai**



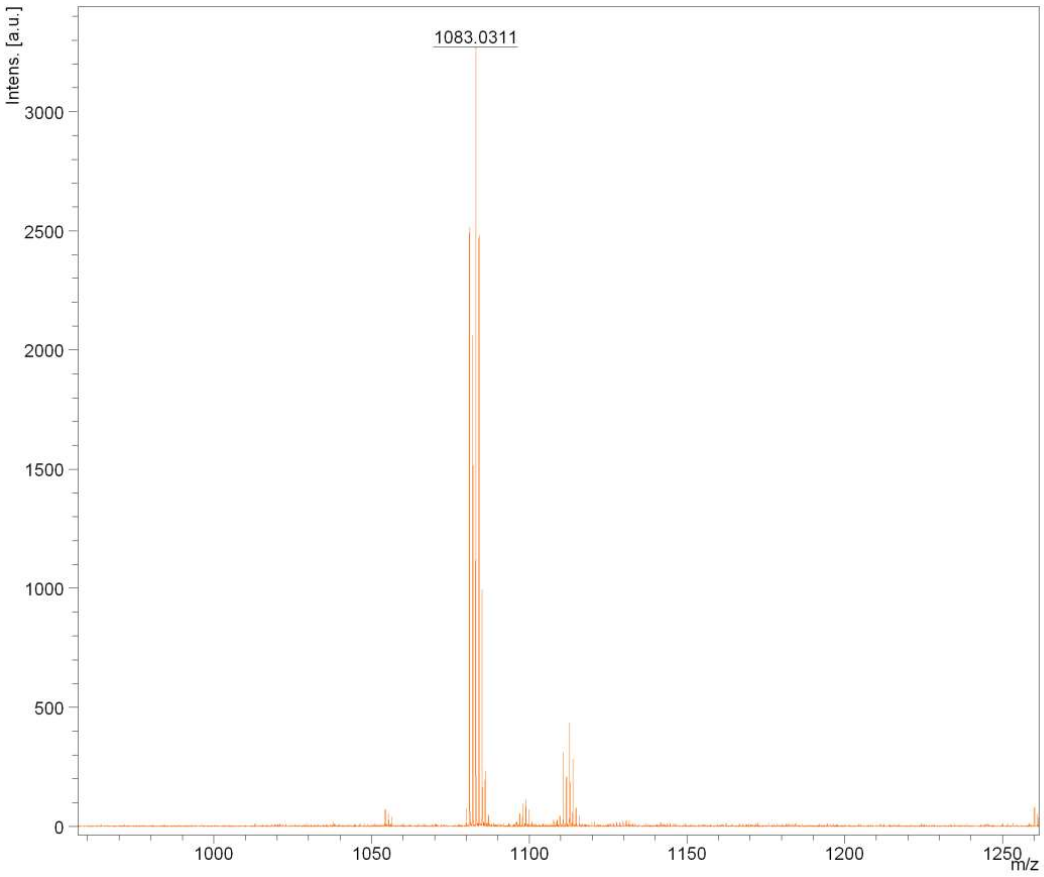
The MALDI-TOF-MS spectrum of **3fi**



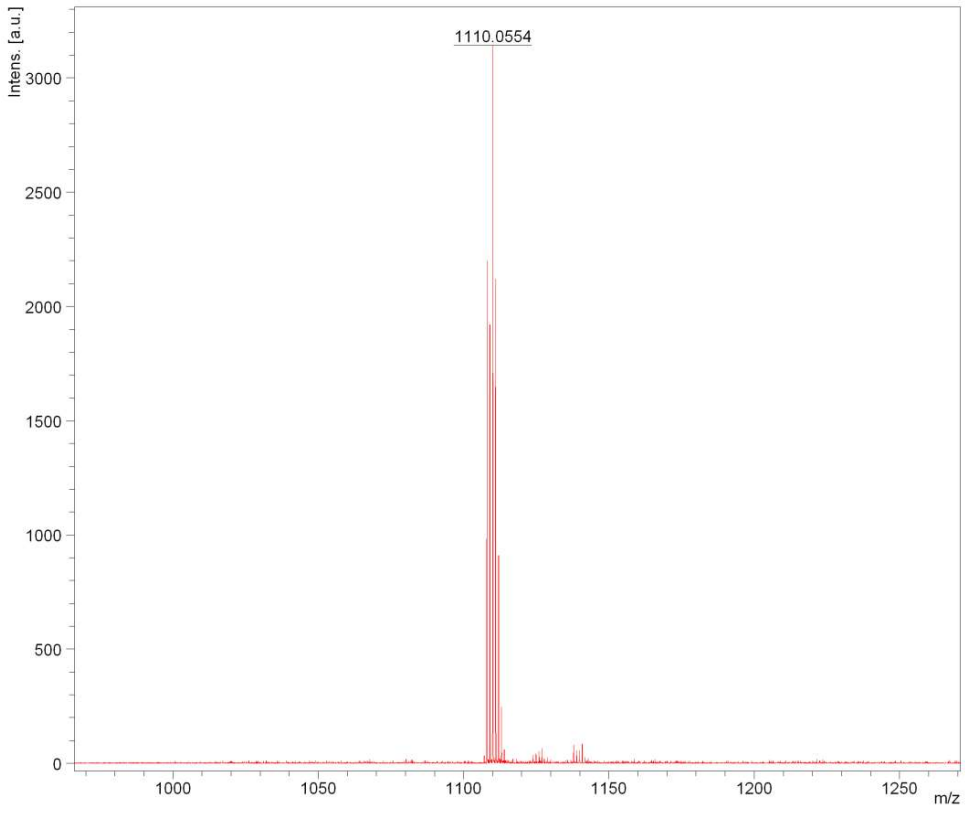
The MALDI-TOF-MS spectrum of **3aj**



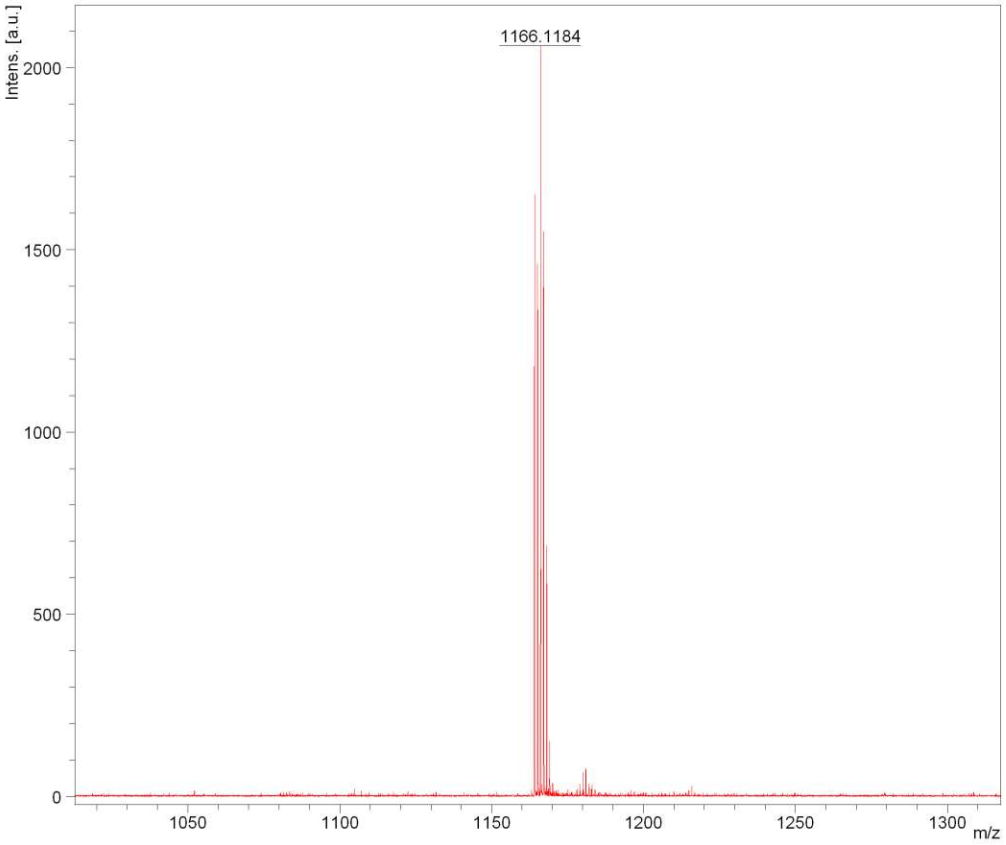
The MALDI-TOF-MS spectrum of **3ak**



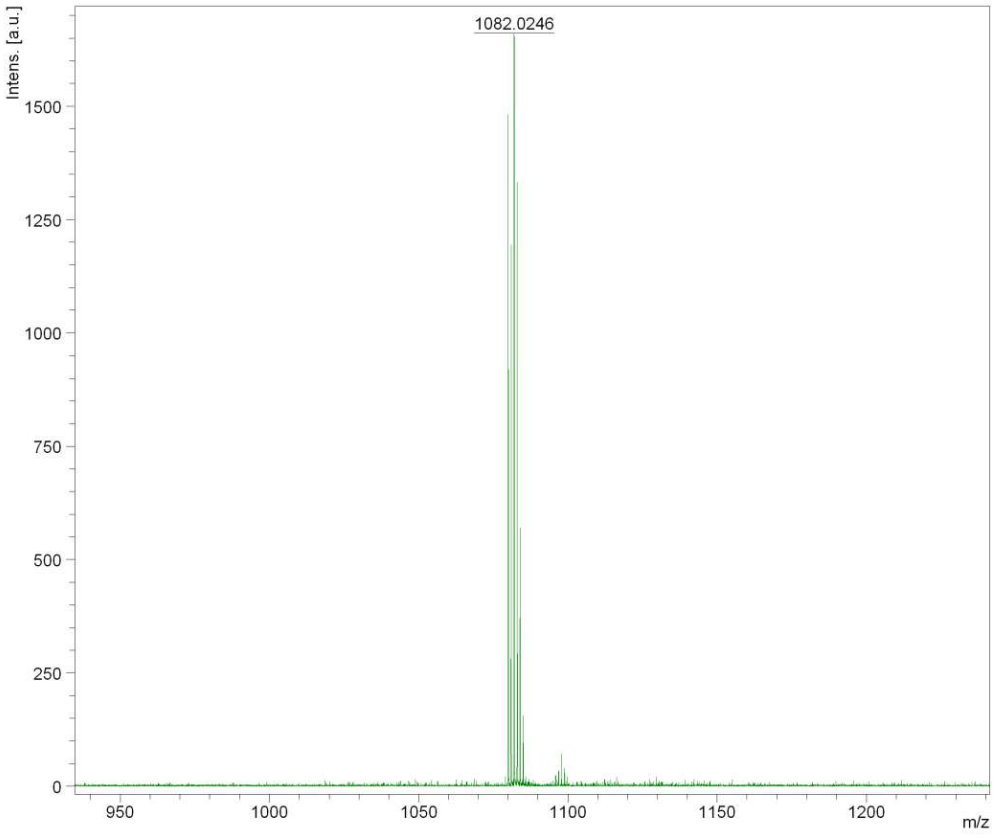
The MALDI-TOF-MS spectrum of **5aa**



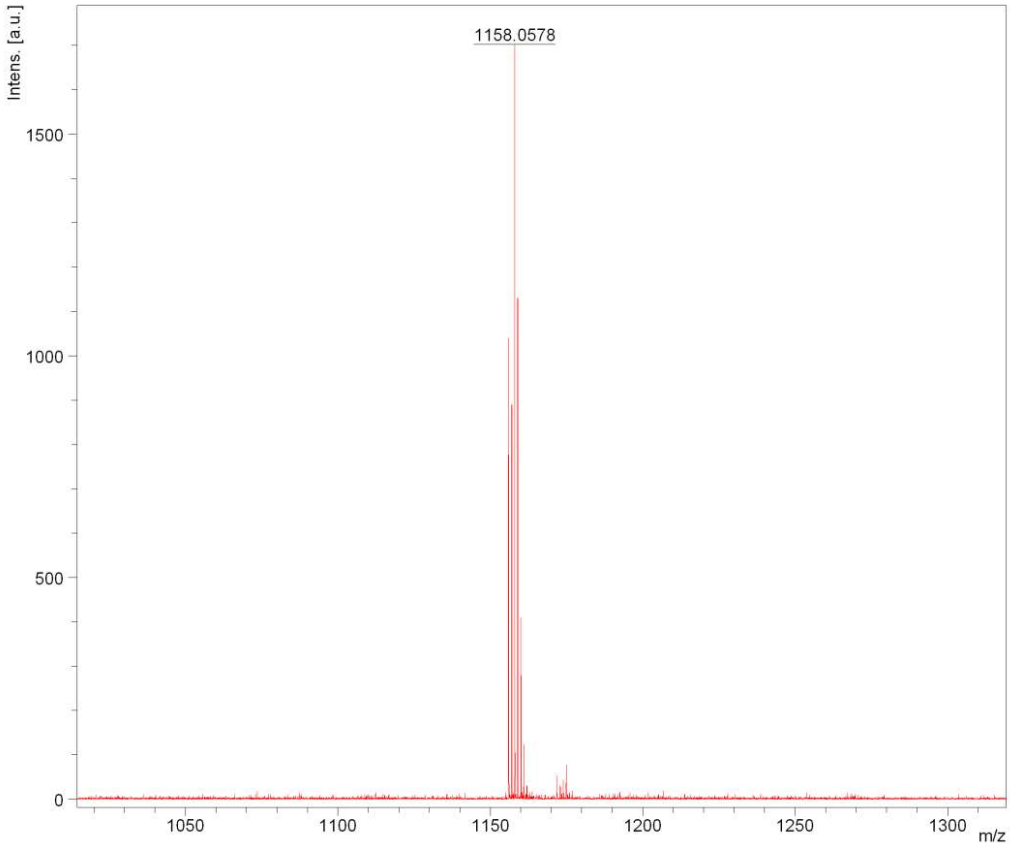
The MALDI-TOF-MS spectrum of **5ab**



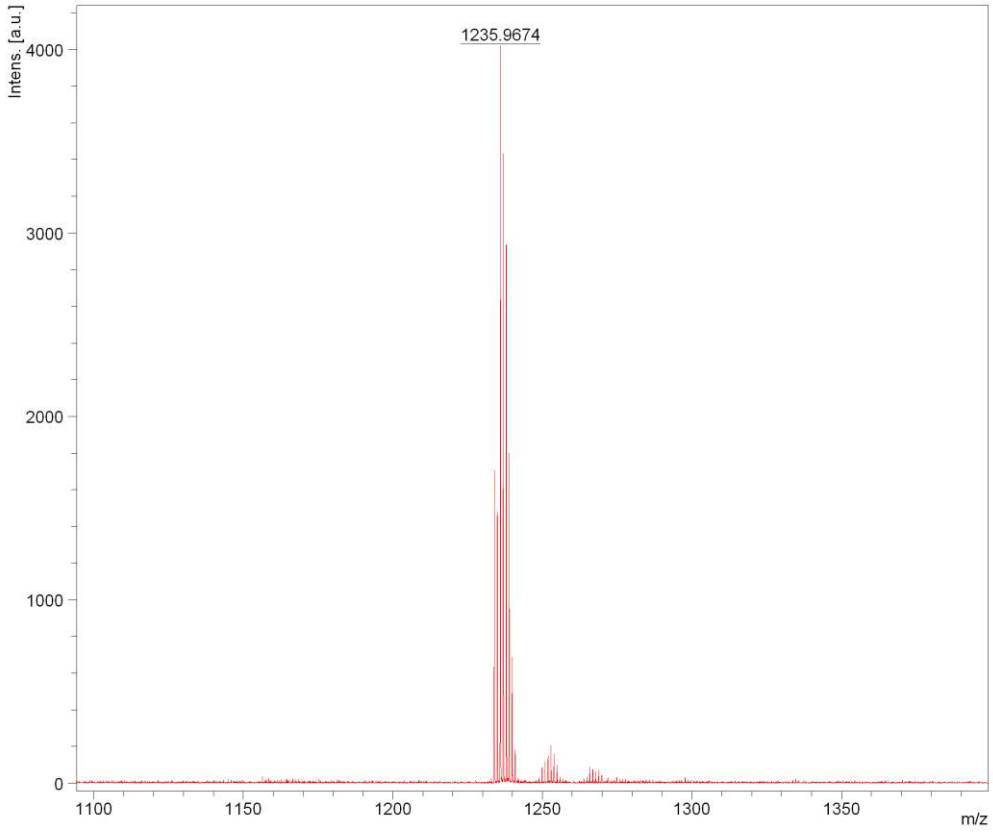
The MALDI-TOF-MS spectrum of **5ac**



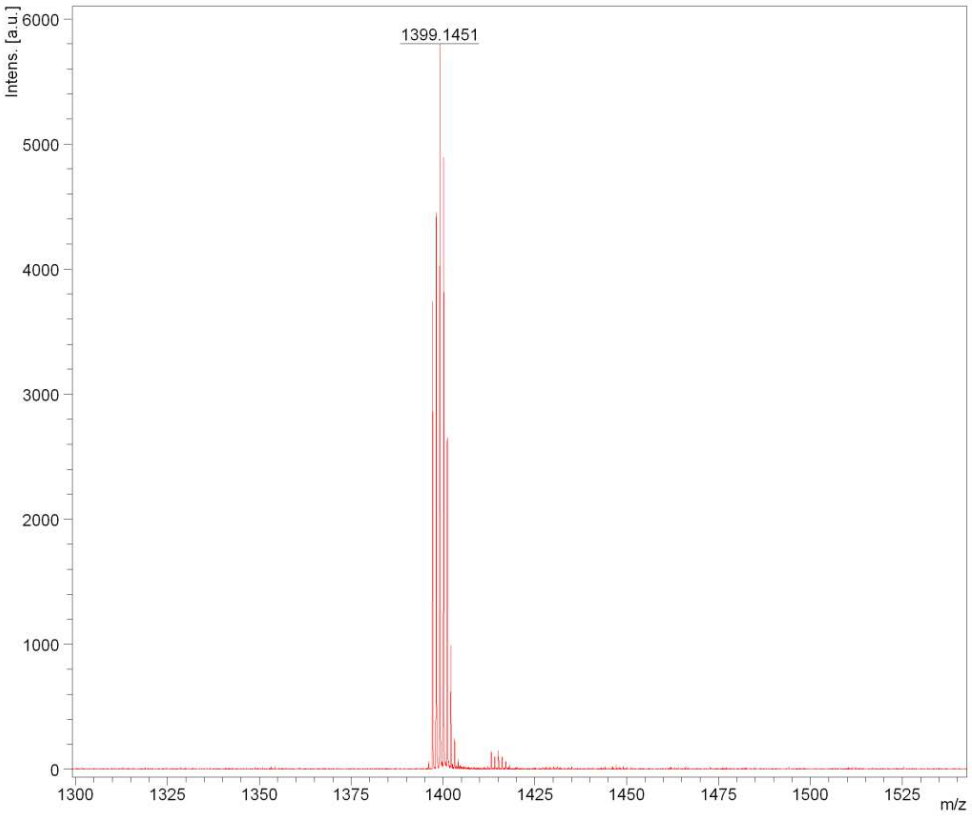
The MALDI-TOF-MS spectrum of **5ad**



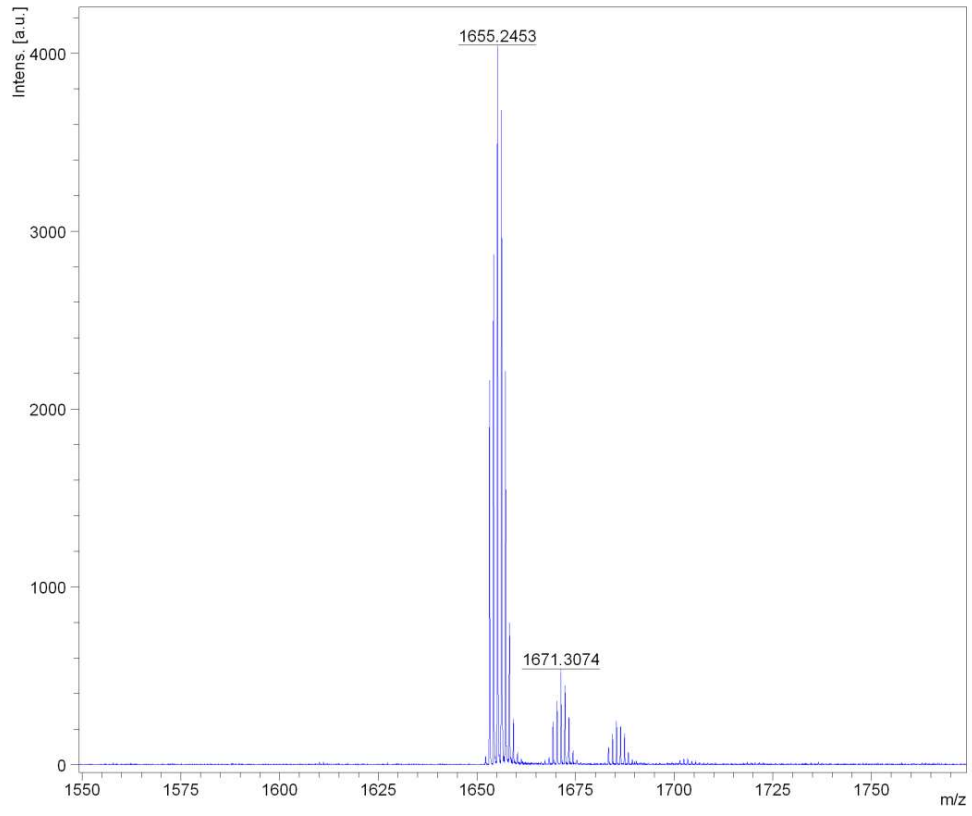
The MALDI-TOF-MS spectrum of **5ae**



The MALDI-TOF-MS spectrum of **5af**



The MALDI-TOF-MS spectrum of **5ag**



The MALDI-TOF-MS spectrum of **5fg**