Supporting Information

Synthesis of C_{60}-fused tetrahydrothiophene derivatives *via* nucleophilic cycloaddition of thiocyanates

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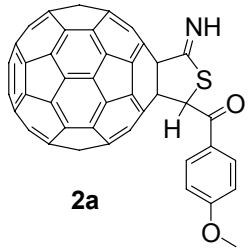
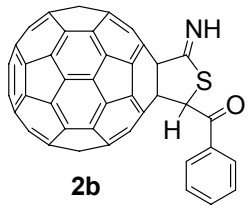


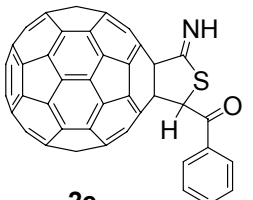
2a

Spectral data of **2a**: ^{1}H NMR (300 MHz, CS_2/DMSO-d_6): δ 11.33 (s, 1H), 8.21 (d, J = 8.9 Hz, 2H), 6.96 (d, J = 8.9 Hz, 2H), 6.93 (s, 1H), 3.85 (s, 3H); ^{13}C NMR (75 MHz, CS_2/DMSO-d_6 with Cr(acac)$_3$ as relaxation reagent, all 1C unless indicated): δ 192.46 (*C*=O), 173.26 (*C*=N), 163.00 (aryl *C*), 153.46, 152.05, 151.68, 151.26, 147.95, 146.04, 145.79, 145.00 (2C), 144.90, 144.83, 144.60, 144.58, 144.56, 144.46, 144.36, 144.31, 144.19, 144.14, 143.90, 143.88, 143.82 (5C), 143.74, 143.54, 143.39, 143.21, 142.99, 142.89, 141.60, 141.55, 141.29, 141.27, 141.23, 141.16, 141.08, 140.76 (2C), 140.63, 140.54, 140.50, 140.47, 140.34 (4C), 140.20, 138.92, 138.62, 138.50, 137.93, 137.75, 136.53, 134.90, 132.06, 130.66 (2C, aryl *C*), 127.03 (aryl *C*), 113.29 (2C, aryl *C*), 77.60 (sp^3-*C* of C_{60}), 70.50 (sp^3-*C* of C_{60}), 58.41 (S*C*H), 54.72 (O*C*H$_3$); FT-IR (KBr) ν (cm^{-1}): 3277, 2951, 2922, 2851, 1671, 1624, 1597, 1572, 1509, 1461, 1422, 1322, 1295, 1265, 1232, 1218, 1171, 1027, 973, 847, 764, 715, 609, 575, 554, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 257 (5.01), 315 (4.63), 429 (3.42), 693 (2.33); MS (-ESI): *m/z* 927 (M^-).



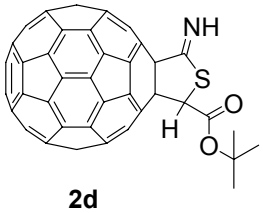
2b

Spectral data of **2b**: ^{1}H NMR (300 MHz, CS_2/$CDCl_3$): δ 10.54 (bs, 1H), 8.19 (d, J = 8.4 Hz, 2H), 7.68 (t, J = 7.4 Hz 1H), 7.57 (t, J = 7.6 Hz, 2H), 6.65 (s 1H); ^{13}C NMR (75 MHz, CS_2/$CDCl_3$ with Cr(acac)$_3$ as relaxation reagent, all 1C unless indicated): δ 195.18 (*C*=O), 178.52 (*C*=N), 153.30, 151.57 (2C), 151.38, 148.27, 147.44, 147.17, 146.36, 146.34, 146.26, 146.18, 145.99, 145.96, 145.93, 145.83, 145.79, 145.71, 145.46, 145.36, 145.30, 145.26, 145.25, 145.21 (2C), 145.12, 144.64, 144.54, 144.48, 144.21 (3C), 144.12, 142.94, 142.89, 142.64 (2C), 142.61, 142.48, 142.44, 142.05, 142.03, 142.02, 141.89, 141.84, 141.69 (4C), 141.67, 141.59, 140.48, 140.27, 140.24, 139.40, 139.01, 137.95 (aryl *C*), 136.40, 135.27, 134.30 (aryl *C*), 133.53, 129.14 (2C, aryl *C*), 128.89 (2C, aryl *C*), 78.58 (sp^3-*C* of C_{60}), 71.94 (sp^3-*C* of C_{60}), 60.05 (S*C*H); FT-IR (KBr) ν (cm^{-1}): 3276, 2950, 2920, 2861, 1680, 1625, 1595, 1446, 1429, 1322, 1295, 1216, 1186, 1094, 1005, 972, 921, 849, 809, 765, 746, 684, 651, 557, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε) 257 (5.02), 314 (4.61), 429 (3.44), 692 (2.35); MS (+ESI): *m/z* 897 (M^+).



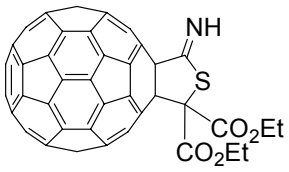
2c

Spectral data of **2c**: ^{1}H NMR (300 MHz, CS_2/DMSO-d_6): δ 11.16 (s, 1H), 8.34 (d, J = 8.4 Hz, 2H), 7.58 (d, J = 8.4 Hz, 2H), 7.03 (s, 1H); FT-IR (KBr) ν (cm^{-1}): 3278, 2950, 2921, 2852, 1680, 1626, 1588, 1568, 1487, 1462, 1429, 1400, 1317, 1290, 1216, 1188, 1093, 1011, 973, 920, 848, 812, 765, 710, 627, 589, 575, 554, 546, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 257 (4.98), 314 (4.60), 429 (3.42), 692 (2.30); MS (-ESI) *m/z* 931 (M^-).



2d

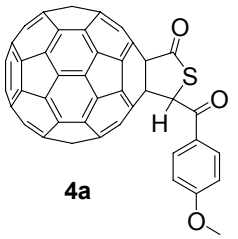
Spectral data of **2d**: ^{1}H NMR (300 MHz, CS_2/$CDCl_3$): δ 10.54 (bs, 1H), 5.57 (s, 1H), 1.61 (s, 9H); ^{13}C NMR (75 MHz, CS_2/DMSO-d_6 with $Cr(acac)_3$ as relaxation reagent, all 1C unless indicated): δ 174.44 (*C*=N), 166.66 (*C*=O), 152.12, 151.84, 150.54, 149.82, 147.32, 145.88, 145.63, 144.84, 144.81 (2C), 144.68, 144.48, 144.40 (2C), 144.37, 144.25, 144.17, 144.01, 143.99, 143.85, 143.74, 143.71, 143.67, 143.62, 143.57, 143.49, 143.33, 143.10, 142.92, 142.71 (3C), 141.40, 141.36, 141.13 (2C), 141.07, 140.98, 140.89, 140.60, 140.57, 140.51, 140.39, 140.31 (2C), 140.23 (2C), 140.14, 140.11, 139.75, 138.81, 138.71, 138.45, 137.89, 136.38, 135.11, 134.53, 132.01, 81.75 (O*C*(CH_3)$_3$), 76.62 (sp^3-*C* of C_{60}), 69.74 (sp^3-*C* of C_{60}), 61.10 (S*C*H), 26.56 (3C, OC(*C*H_3)$_3$); FT-IR (KBr) ν (cm^{-1}): 3272, 2960, 2920, 2852, 1729, 1622, 1454, 1428, 1390, 1366, 1312, 1282, 1247, 1217, 1183, 1140, 849, 819, 765, 742, 574, 548, 526; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 257 (5.09), 313 (4.74), 428 (3.68), 692 (2.49); MS (-ESI): *m/z* 893 (M^-).



2e

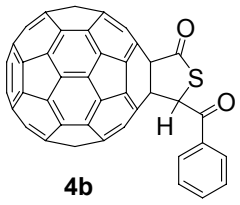
Spectral data of **2e**: ^{1}H NMR (300 MHz, CS_2/$CDCl_3$): δ 11.31 (s, 1H), 4.49-4.31 (m, 4H), 1.38 (t, J = 7.1 Hz, 6H); ^{13}C NMR (75 MHz, CS_2/DMSO-d_6 with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated): δ 173.12 (1C, *C*=N), 164.63 (*C*=O), 152.43, 148.04, 146.30 (1C), 145.93 (1C), 145.73, 145.32, 145.20, 144.80, 144.75, 144.58, 144.44, 144.15, 144.12 (4C), 143.95, 143.52, 143.05, 141.69, 141.60, 141.49, 141.25, 141.13, 140.63, 140.60, 140.32, 140.06, 139.01, 137.45, 137.10, 134.26, 79.91 (1C, sp^3-*C* of C_{60}),

75.05 (1C, S*C*(COEt)$_2$), 71.83 (1C, sp^3-*C* of C_{60}), 62.28 (O*C*H$_2$CH$_3$), 13.19 (OCH$_2$*C*H$_3$); FT-IR ν/cm^{-1} (KBr): 3277, 2956, 2921, 2852, 1732, 1625, 1571, 1520, 1459, 1431, 1381, 1365, 1244, 1217, 1178, 1079, 1013, 932, 849, 819, 771, 762, 676, 590, 575, 554, 546, 526; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 260 (5.07), 318 (4.64), 428 (3.40), 686 (2.50); MS (+ESI): *m/z* 937 (M^+).



4a

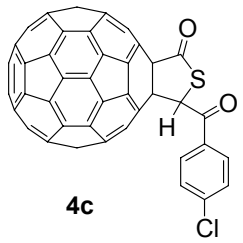
Spectral data of **4a**: ^{1}H NMR (300 MHz, CS_2/DMSO-d_6): δ 8.33 (d, J = 9.0 Hz), 7.28 (s, 1H), 7.03 (d, J = 9.0 Hz), 3.93 (s 1H); ^{13}C NMR (75 MHz, CS_2/DMSO-d_6 with Cr(acac)$_3$ as relaxation reagent, all 1C unless indicated): δ 194.60 (*C*=O), 193.68 (*C*=O), 163.52 (aryl *C*), 150.53, 150.45, 150.23, 149.49, 147.34, 146.21, 145.87, 145.20, 145.16, 145.05 (2C), 144.81, 144.79, 144.77, 144.66, 144.60, 144.58, 144.43, 144.37, 144.20, 144.16, 144.10 (2C), 144.07, 144.03, 143.99 (2C), 143.39, 143.21, 143.15, 143.04, 142.93, 141.77, 141.68, 141.48, 141.44, 141.41, 141.33, 141.29, 140.88, 140.85, 140.81, 140.77, 140.73, 140.58, 140.54, 140.48, 140.47, 140.38, 140.23, 139.30, 139.16, 139.00, 138.20, 136.78, 136.61, 134.41, 132.85, 131.09 (2C, aryl *C*), 127.12 (aryl *C*), 113.48 (2C, aryl *C*), 80.38 (sp^3-*C* of C_{60}), 68.66 (sp^3-*C* of C_{60}), 56.73 (S*C*H), 54.61 (O*C*H$_3$); FT-IR ν/cm^{-1} (KBr): 2951, 2925, 2850, 1715, 1709, 1667, 1596, 1571, 1509, 1460, 1422, 1320, 1293, 1266, 1233, 1170, 1098, 1023, 969, 922, 837, 827, 791, 764, 715, 608, 588, 554, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 259 (5.02), 315 (4.70), 430 (3.48), 691 (2.51); MS (-ESI): *m/z* 928 (M^-).



4b

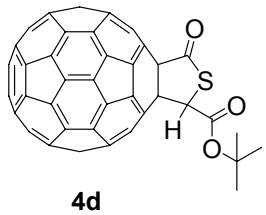
Spectral data of **4b**: ^{1}H NMR (300 MHz, CS_2/DMSO-d_6): δ 8.38 (dt, J = 7.0 Hz, 2H), 7.74 (t, J = 7.4 Hz, 1H), 7.63 (t, J = 7.5 Hz, 2H) 7.28 (s, 1H); ^{13}C NMR (75 MHz, CS_2/DMSO-d_6 with Cr(acac)$_3$ as relaxation reagent, all 1C unless indicated): δ 195.26 (*C*=O), 194.10 (*C*=O), 150.20, 150.02 (2C), 149.26, 147.27, 146.24, 145.87, 145.20, 145.19, 145.07, 145.06, 144.83, 144.81, 144.79, 144.67, 144.63 (2C), 144.34 (2C), 144.23, 144.14, 144.12, 144.07 (2C), 144.02, 144.00, 143.85, 143.40, 143.22, 142.99 (2C), 142.90, 141.78, 141.69, 141.49, 141.47, 141.43, 141.34, 141.31, 140.90, 140.83, 140.78 (2C), 140.71, 140.56, 140.54, 140.50, 140.43, 140.40, 140.21, 139.34, 139.22, 139.08, 138.21, 136.92, 136.51, 134.50, 134.23, 133.39, 132.80, 128.45 (2C, aryl *C*), 128.10 (2C, aryl *C*), 80.27 (sp^3-*C* of C_{60}), 68.44 (sp^3-*C* of C_{60}), 56.98 (S*C*H); FT-IR ν/cm^{-1}

(KBr): 2921, 2852, 1720, 1676, 1593, 1462, 1445, 1428, 1320, 1291, 1218, 1183, 1162, 1098, 1021, 969, 922, 815, 764, 745, 683, 650, 587, 556, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 257 (5.07), 315 (4.65), 430 (3.48), 691 (2.56); MS (-ESI): *m/z* 898 (M^-).



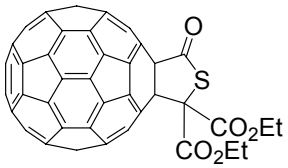
4c

Spectral data of **4c**: ^{1}H NMR (300 MHz, CS_2/DMSO-d_6): δ 8.33 (d, J = 6.7 Hz, 2H), 7.50 (d, J = 6.7 Hz, 2H), 7.35 (s, 1H); ^{13}C NMR (75 MHz, CS_2/DMSO-d_6 with $Cr(acac)_3$ as relaxation reagent, all 1C unless indicated): δ 194.60 (*C*=O), 194.29 (*C*=O), 150.19, 149.98, 149.96, 149.22, 147.20, 146.18, 145.82, 145.16, 145.14, 145.02, 145.01, 144.78, 144.76, 144.74, 144.63, 144.58, 144.54, 144.28 (2C), 144.18, 144.07 (2C), 144.02 (2C), 143.99, 143.96 (2C), 143.34, 143.16, 142.95 (2C), 142.86, 141.73, 141.64, 141.44, 141.42, 141.39, 141.31, 141.25, 140.84, 140.79, 140.74, 140.72, 140.64, 140.48 (2C), 140.44, 140.35 (2C), 140.22, 140.16, 139.27, 139.17, 138.91, 138.17, 136.81, 136.44, 134.37, 132.75, 132.52, 130.17 (2C, aryl *C*), 128.34 (2C, aryl *C*), 80.27 (sp^3-*C* of C_{60}), 68.39 (sp^3-*C* of C_{60}), 56.87 (S*C*H); FT-IR ν/cm^{-1} (KBr): 2949, 2922, 2852, 1716, 1679, 1587, 1461, 1429, 1400, 1315, 1286, 1232, 1217, 1183, 1094, 1015, 970, 922, 836, 765, 710, 656, 610, 588, 550, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 259 (5.00), 316 (4.57), 430 (3.40), 691 (2.50); MS (-ESI): *m/z* 932 (M^-).



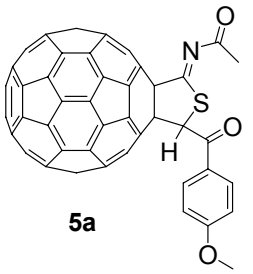
4d

Spectral data of **4d**: ^{1}H NMR (300 MHz, CS_2/DMSO-d_6): δ 5.89 (s 1H), 1.58 (s, 9H); ^{13}C NMR (75 MHz, CS_2/DMSO-d_6 with $Cr(acac)_3$ as relaxation reagent, all 1C unless indicated): δ 194.55 (*C*=O), 166.67 (*C*=O), 149.71, 149.14, 148.91 (2C), 146.57, 145.85, 145.53, 144.85, 144.80, 144.78, 144.73, 144.48, 144.42, 144.39, 144.38, 144.25 (2C), 144.01, 143.98, 143.90, 143.83, 143.73, 143.71, 143.68 (2C), 143.64, 143.31, 143.27, 142.90, 143.75, 142.67, 142.62, 141.39, 141.35, 141.16, 141.10, 141.06, 141.02, 140.81, 140.54 (3C), 140.29, 140.28, 140.21, 140.15, 140.10, 140.06, 139.91, 139.70, 138.96, 138.80, 138.79, 137.88, 135.62, 135.31, 133.80, 132.67, 82.51 (O*C*CH_3)$_3$), 79.53 (sp^3-*C* of C_{60}), 67.14 (sp^3-*C* of C_{60}), 59.08 (S*C*H), 26.45 (3C, C(*C*H_3)$_3$); FT-IR ν/cm^{-1} (KBr): 2975, 2925, 2852, 1735, 1715, 1514, 1454, 1429, 1392, 1368, 1316, 1247, 1183, 1144, 1098, 1022, 997, 827, 765, 590, 574, 554, 545, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 258 (5.08), 316 (4.61), 429 (3.46), 690 (2.55); MS (-ESI): *m/z* 894 (M^-).



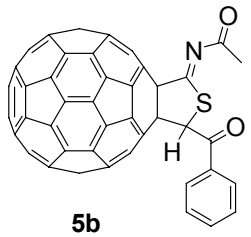
4e

Spectral data of **4e**: ^{1}H NMR (300 MHz, CS_2/DMSO-d_6): δ 4.49-4.35 (m, 4H), 1.38 (t, J = 7.0 Hz, 6H); ^{13}C NMR (75 MHz, CS_2/DMSO-d_6 with Cr(acac)$_3$ as relaxation reagent, all 2C unless indicated): δ 191.72 (1C, *C*=O), 164.55 (*C*=O), 149.23, 146.99, 146.18 (1C), 145.74 (1C), 145.51, 145.21, 145.16, 145.14, 144.75, 144.68, 144.51, 144.22, 144.19, 144.02, 143.93, 143.22, 142.88, 141.58, 141.50, 141.45, 141.09, 141.04, 140.57, 140.22, 140.15, 139.91, 139.21, 137.08, 136.57, 134.61, 83.27 (1C, sp^3-*C* of C_{60}), 74.17 (1C, S*C*(COEt)$_2$), 69.77 (1C, sp^3-*C* of C_{60}), 62.77 (O*C*H$_2$CH$_3$), 13.06 (OCH$_2$*C*H$_3$); FT-IR ν/cm^{-1} (KBr): 2973, 2956, 2923, 2853, 1748, 1720, 1453, 1429, 1366, 1297, 1249, 1227, 1178, 1090, 1091, 1009, 866, 763, 598, 583, 574, 544, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 260 (5.06), 318 (4.63), 428 (3.40), 686 (2.48); MS (-ESI): *m/z* 938 (M^-).



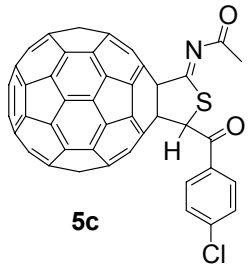
5a

Spectral data of **5a**: ^{1}H NMR (300 MHz, CS_2/$CDCl_3$): δ 8.17 (d, J = 8.8 Hz, 2H), 7.01 (d, J = 8.8 Hz, 2H), 6.79 (s, 1H), 3.90 (s, 3H), 2.52 (s, 3H); ^{13}C NMR (75 MHz, CS_2/$CDCl_3$ with Cr(acac)$_3$ as relaxation reagent, all 1C unless indicated): δ 194.41 (*C*=O), 184.14 (*C*=O), 176.62 (*C*=N), 164.62 (aryl *C*), 152.22, 151.84, 151.46, 151.12, 148.54, 147.42, 147.19, 146.33, 146.32, 146.21, 146.19, 145.98, 145.95, 145.91, 145.80, 145.72, 145.71, 145.44, 145.30, 145.28, 145.25, 145.20, 145.18, 145.16 (2C), 145.13, 144.58, 144.52, 144.39, 144.18, 144.16, 144.07, 142.88, 142.82, 142.61 (2C), 142.59, 142.50, 142.42, 142.09, 141.91, 141.89 (2C), 141.77, 141.70 (2C), 141.64, 141.56, 141.51 (2C), 140.34, 140.30, 139.89, 139.45, 138.34, 137.62, 136.06, 133.74, 131.38 (2C, aryl *C*), 128.19 (aryl *C*), 114.43 (2C, aryl *C*), 79.73 (sp^3-*C* of C_{60}), 69.66 (sp^3-*C* of C_{60}), 61.10 (S*C*H), 55.34 (O*C*H$_3$), 25.77 (CO*C*H$_3$); FT-IR ν/cm^{-1} (KBr): 2951, 2920, 2850, 1713, 1668, 1643, 1597, 1572, 1510, 1461, 1422, 1358, 1326, 1266, 1216, 1171, 1115, 1029, 975, 930, 845, 764, 715, 604, 554, 543, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 258 (5.01), 314 (4.67), 429 (3.38), 693 (2.43); MS (-ESI): *m/z* 969 (M^-).



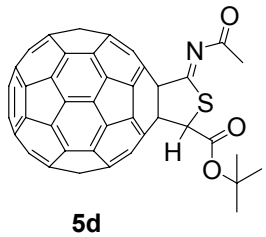
5b

Spectral data of **5b**: ^{1}H NMR (300 MHz, CS_2/$CDCl_3$): δ 8.21 (d, J = 7.5 Hz, 2H), 7.69 (t, J = 7.3 Hz, 1H), 7.58 (t, J = 7.6 Hz, 2H), 6.87 (s, 1H), 2.55 (s, 3H); ^{13}C NMR (75 MHz, CS_2/$CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 1C unless indicated): δ 196.49 (*C*=O), 184.63 (*C*=O), 176.29 (*C*=N), 152.03, 151.63, 151.19, 150.84, 148.39, 147.42, 147.19, 146.32, 146.31, 146.20, 146.18, 145.97, 145.94, 145.90, 145.80, 145.72, 145.69, 145.40, 145.25 (3C), 145.21, 145.18, 145.15, 145.13, 144.99, 144.54, 144.43, 144.35, 144.12, 144.10, 144.05, 142.87, 142.80, 142.59 (2C), 142.57, 142.47, 142.41, 142.05, 141.89, 141.87, 141.86, 141.73, 141.70, 141.65, 141.63, 141.52, 141.50, 141.47, 140.33, 140.30, 139.92, 139.45, 138.35, 137.55, 136.04, 135.24, 134.56, 133.75, 129.19 (2C, aryl *C*), 128.89 (2C, aryl *C*), 79.66 (sp^3-*C* of C_{60}), 69.60 (sp^3-*C* of C_{60}), 61.29 (S*C*H), 25.80 (CO*C*H_3); FT-IR ν/cm^{-1} (KBr): 2919, 2850, 1697, 1677, 1637, 1608, 1594, 1446, 1426, 1359, 1323, 1296, 1218, 1182, 1167, 1051, 999, 974, 930, 889, 764, 685, 651, 609, 554, 544, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 2.58 (5.08), 316 (4.58), 429 (3.40), 688 (2.46); MS (-ESI): *m/z* 939 (M^-).



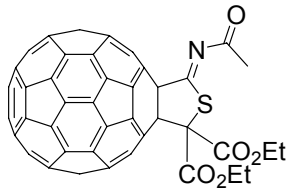
5c

Spectral data of **5c**: ^{1}H NMR (300 MHz, CS_2/DMSO-d_6): δ 8.39 (d, J = 8.7 Hz, 2H), 7.57 (d, J = 8.7 Hz, 2H), 7.40 (s, 1H), 2.52 (s, 3H); ^{13}C NMR (75 MHz, CS_2/DMSO-d_6 with $Cr(acac)_3$ as relaxation reagent, all 1C unless indicated): δ 194.59 (*C*=O), 182.09 (*C*=O), 175.64 (*C*=N), 151.63, 151.21, 151.11, 150.45, 147.82, 146.46, 146.25, 145.43, 145.40, 145.30, 145.28, 145.04, 145.01, 144.98, 144.89, 144.82, 144.75, 144.58, 144.43, 144.36 (3C), 144.31 (2C), 144.27, 144.23 (2C), 144.19, 143.65, 143.60, 143.46, 143.31, 143.23, 141.97, 141.90, 141.70, 141.68, 141.66, 141.56, 141.53, 141.14, 141.06, 141.03, 140.88, 140.86, 140.78, 140.77, 140.71, 140.63, 140.57 (2C), 140.21, 139.30, 139.11, 138.89, 138.49, 137.52, 136.91, 134.88, 132.80 (aryl *C*), 130.26 (2C, aryl *C*), 128.44 (2C, aryl *C*), 79.01 (sp^3-*C* of C_{60}), 68.65 (sp^3-*C* of C_{60}), 60.07 (S*C*H), 24.80 (CO*C*H_3); FT-IR ν/cm^{-1} (KBr): 2951, 2921, 2853, 1725, 1680, 1639, 1588, 1568, 1428, 1400, 1358, 1317, 1287, 1217, 1180, 1093, 975, 930, 850, 764, 710, 608, 587, 547, 528; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 258 (5.09), 316 (4.63), 429 (3.41), 690 (2.51); MS (-ESI): *m/z* 973 (M^-).



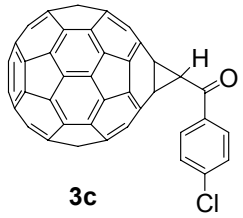
5d

Spectral data of **5d**: ^{1}H NMR (300 MHz, $CS_2/CDCl_3$): δ 5.70 (s, 1H), 2.53 (s, 3H), 1.57 (s, 9H); ^{13}C NMR (75 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 1C unless indicated): 184.52 (*C*=O), 177.54 (*C*=N), 169.14 (*C*=O), 151.89, 151.74, 151.35, 150.53, 148.24, 147.51, 147.31, 146.41, 146.39, 146.37, 146.31, 146.08, 146.01 (2C), 145.97, 145.78, 145.77, 145.54, 145.48, 145.36, 145.33, 145.32 (2C), 145.26, 145.25, 145.20, 144.84, 144.69, 144.54, 144.35, 144.27, 144.22, 142.94, 142.89, 142.72, 142.69, 142.66, 142.56, 142.46, 142.15, 142.09, 142.07, 141.92, 141.81, 141.76, 141.74, 141.68, 141.61, 141.60, 141.22, 140.40, 140.37, 140.00, 139.59, 137.54, 136.83, 135.69, 133.98, 84.36 ($OC(CH_3)_3$), 79.46 (sp^3-*C* of C_{60}), 69.01 (sp^3-*C* of C_{60}), 63.58 (S*C*H), 27.95 (3C, $OC(CH_3)_3$), 25.84 ($COCH_3$); FT-IR ν/cm^{-1} (KBr): 2975, 2924, 1734, 1637, 1453, 1429, 1392, 1368, 1316, 1217, 1185, 1144, 1098, 1022, 996, 828, 765, 589, 575, 554, 546, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 2.55 (5.06), 316 (4.59), 429 (3.46), 689 (2.50); MS (-ESI): *m/z* 935 (M^-).



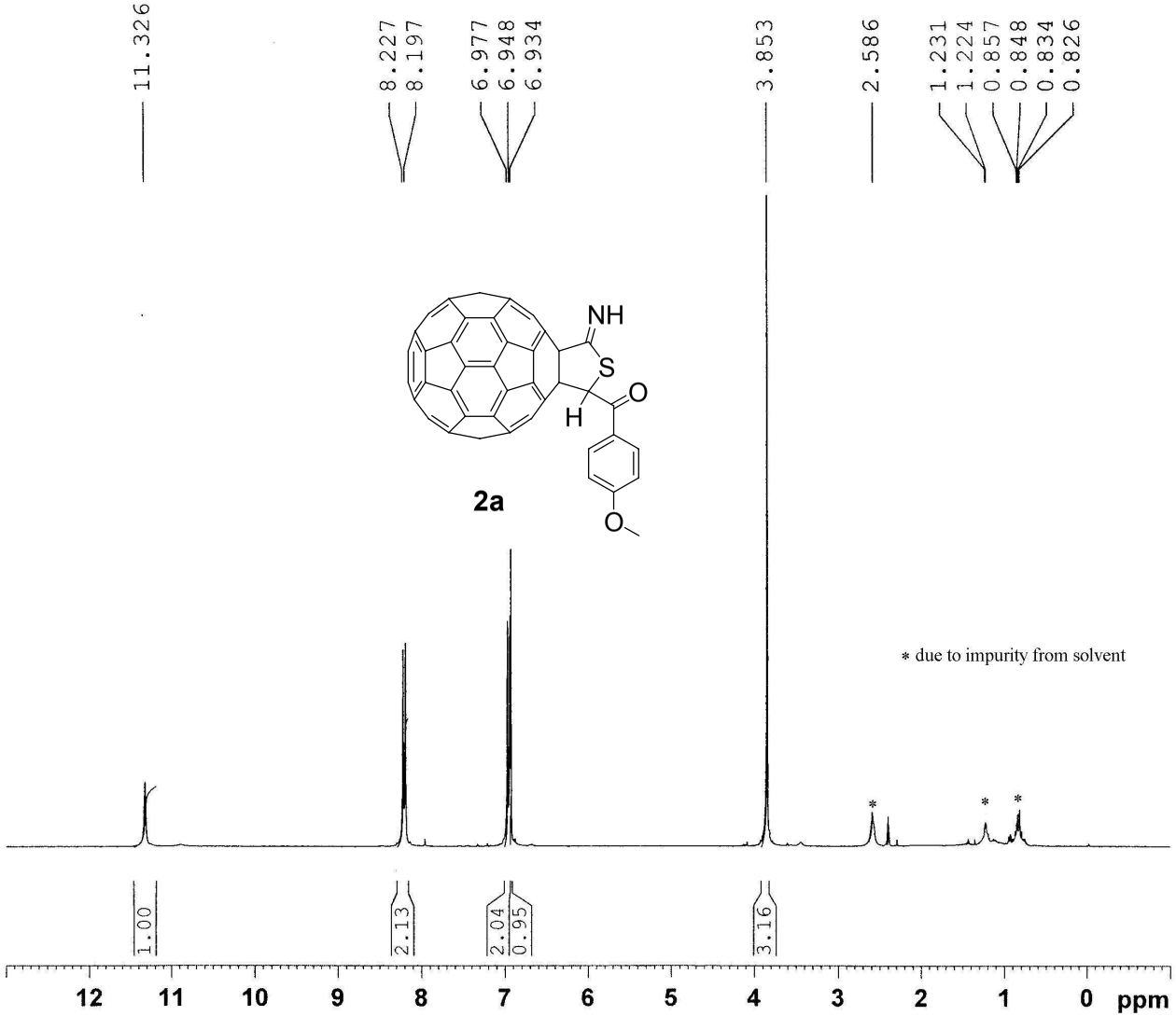
5e

Spectral data of **5e**: ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.56-4.39 (m, 4H), 2.54 (s, 3H), 1.38 (t, J = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated): δ 185.00 (1C, *C*=O), 175.08 (1C, *C*=N), 166.80 (*C*=O), 151.97, 148.41, 147.62 (1C), 147.32 (1C), 146.74, 146.58, 146.50, 146.41, 146.12, 146.05, 145.80, 145.57, 145.48, 145.41, 145.28, 144.62, 144.27, 142.90, 142.85, 142.76, 142.48, 142.35, 141.83, 141.63, 141.61, 141.26, 140.29, 138.57, 138.14, 135.78, 82.31 (1C, sp^3-*C* of C_{60}), 78.11 (1C, S*C*$(COEt)_2$), 71.04 (1C, sp^3-*C* of C_{60}), 63.86 (OCH_2CH_3), 25.95 (1C, $COCH_3$) , 13.88 (OCH_2CH_3); FT-IR ν/cm^{-1} (KBr): 2975, 2953, 2922, 2865, 1732, 1609, 1463, 1430, 1424, 1361, 1245, 1217, 1167, 1115, 1080, 1064, 1048, 1027, 872, 762, 707, 676, 633, 589, 575, 546, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 256 (5.05), 317 (4.58), 429 (3.31), 684 (2.42); MS (-ESI): *m/z* 979 (M^-).



3c

Spectral data of **3c**: ^{1}H NMR (300 MHz, $CS_2/CDCl_3$): δ 8.37 (d, J = 8.4 Hz, 2H), 7.62 (d, J = 8.4 Hz, 2H), 5.55 (s, 1H); ^{13}C NMR (75 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated): δ 187.51 (1C, *C*=O), 147.57, 146.10, 145.25, 145.11 (4C), 145.03 (4C), 144.91, 144.63 (1C), 144.55 (2C), 144.49 (6C), 144.38 (1C), 144.24, 143.76, 143.51, 143.18, 142.99 (1C), 142.88, 142.82 (4C), 142.62, 142.27, 142.09, 141.93 (5C), 141.09 (3C), 140.83, 139.49, 136.48, 134.12 (1C, aryl C), 130.07 (aryl *C*), 129.52 (aryl *C*), 71.89 (sp^3-*C* of C_{60}), 43.75 (1C, *C*H); FT-IR (KBr) ν (cm^{-1}): 2971, 2922, 2865, 1685, 1640, 1588, 1427, 1404, 1319, 1242, 1216, 1183, 1092, 1007, 945, 874, 842, 816, 738, 703, 575, 527; UV-vis ($CHCl_3$) λ_{max} nm (log ε): 261 (4.97), 327 (4.41), 427 (3.24), 496 (3.00), 690 (2.27); MS (+ESI): *m/z* 872 (M^+).

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PROCNO                1

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PULPROG              zg
TD                65536
SOLVENT            DMSO
NS                   17
DS                    2
SWH            5995.204 Hz
FIDRES         0.091480 Hz
AQ             5.4657526 sec
RG                287.4
DW               83.400 usec
DE                6.00 usec
TE                300.0 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.50 usec
PL1               -3.00 dB
SFO1         300.1321009 MHz

F2 - Processing parameters
SI                16384
SF          300.1300348 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
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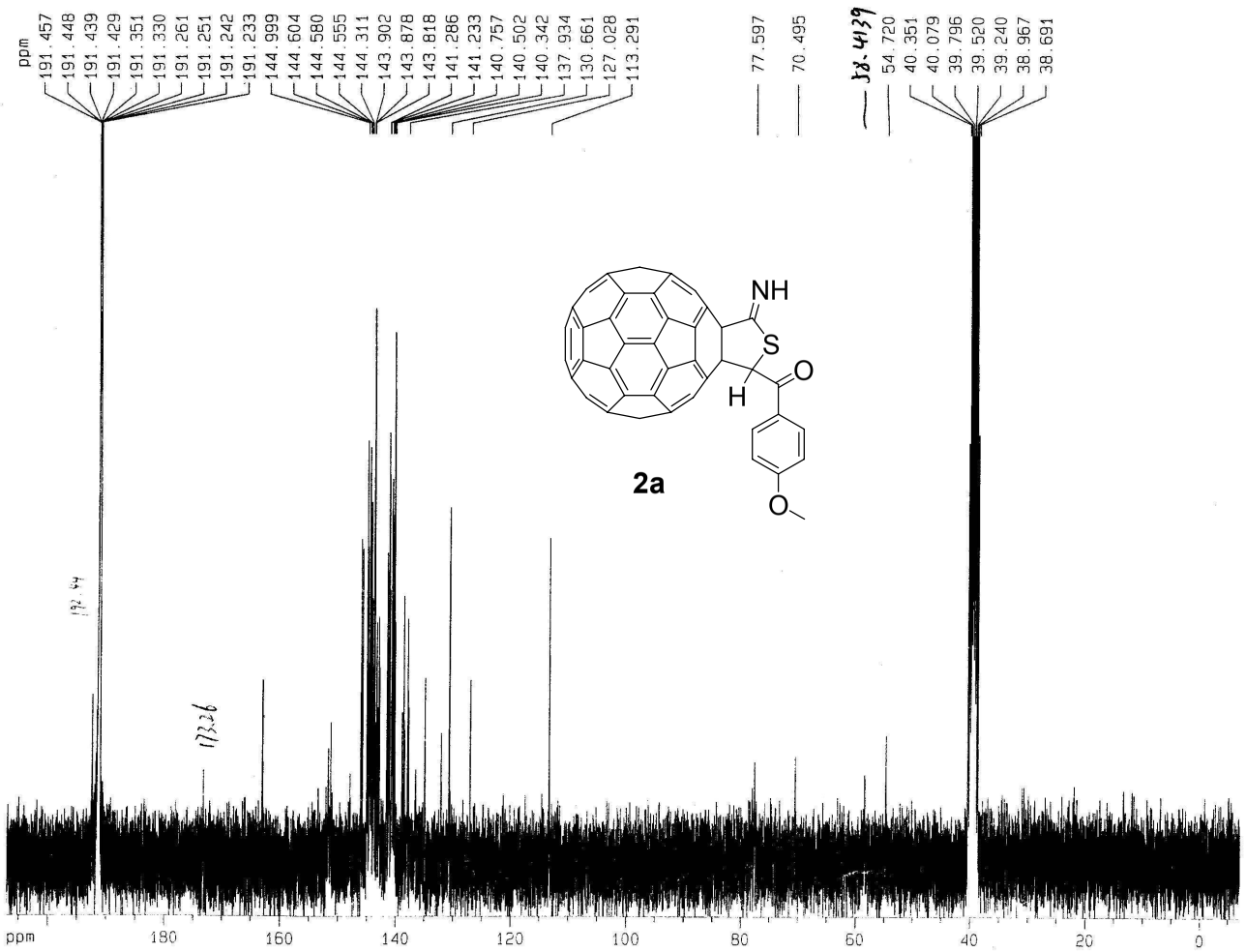


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Current Data Parameters
NAME                 ljx051123
EXPNO                        4
PROCNO                       1

F2 - Acquisition Parameters
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Time                     7.49
INSTRUM                av300
PROBHD   5 mm BBO BB-1H
PULPROG                 zgdc
TD                     65536
SOLVENT                DMSO
NS                     29701
DS                         4
SWH                22675.736 Hz
FIDRES             0.346004 Hz
AQ                 1.4451188 sec
RG                   11585.2
DW                    22.050 usec
DE                     6.00 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     3.00 usec
PL1                   -1.00 dB
SFO1               75.4768051 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                   -3.00 dB
PL12                  16.00 dB
SFO2              300.1315007 MHz

F2 - Processing parameters
SI                       131072
SF                 75.4678665 MHz
WDW                        EM
SSB                         0
LB                     0.20 Hz
GB                          0
PC                      1.40

1D NMR plot parameters
CX                     22.00 cm
CY                      0.00 cm
F1P                   207.068 ppm
F1                  15627.00 Hz
F2P                    -7.047 ppm
F2                   -531.85 Hz
PPMCM                 9.73252 ppm/cm
HZCM                734.49286 Hz/cm
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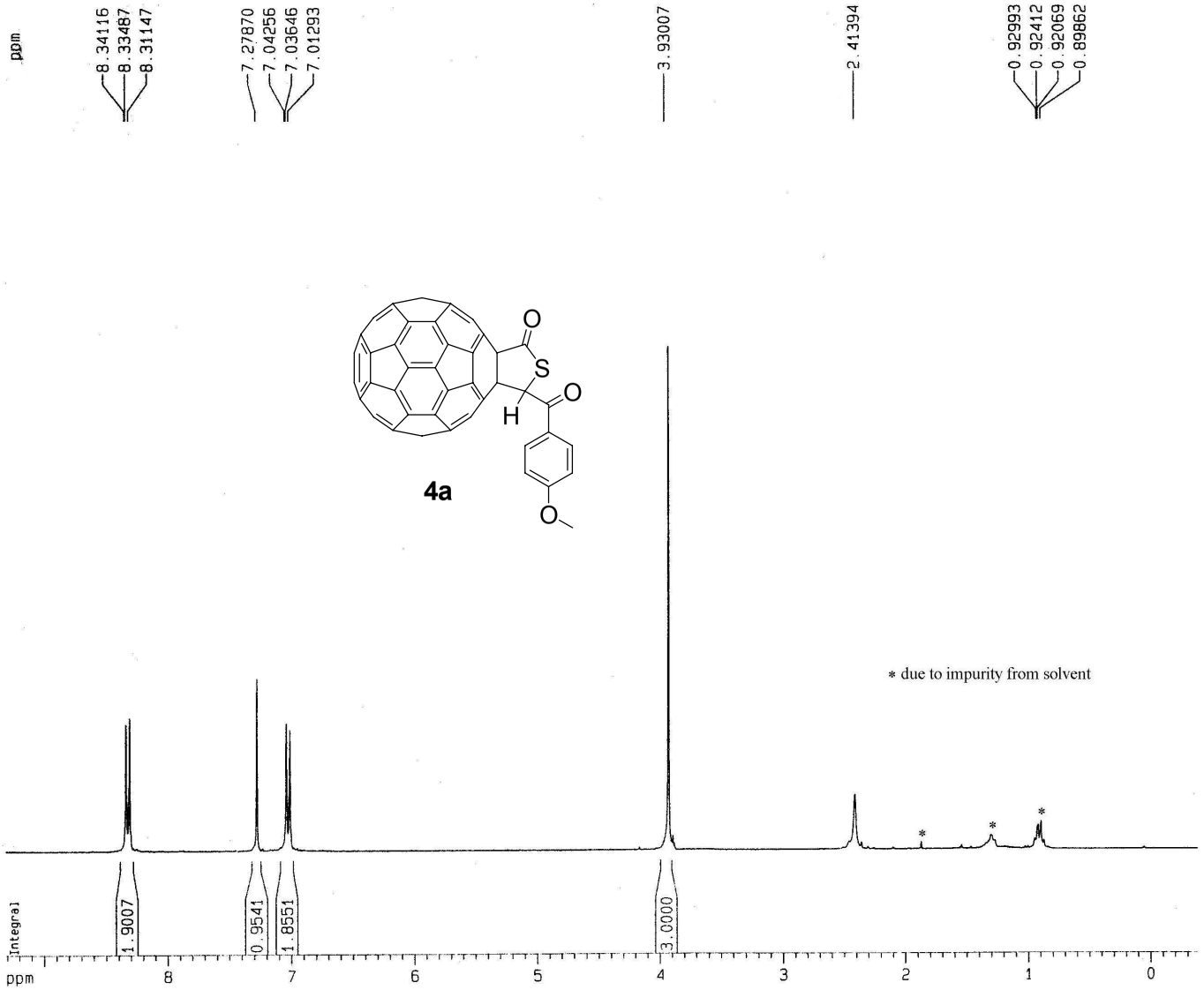


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Current Data Parameters
NAME            1jx051020
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20051020
Time                16.48
INSTRUM             av300
PROBHD    5 mm BBO BB-1H
PULPROG                zg
TD                  65536
SOLVENT              DMSO
NS                     25
DS                      2
SWH             5995.204 Hz
FIDRES          0.091480 Hz
AQ              5.4557526 sec
RG                    362
DW                 83.400 usec
DE                  6.00 usec
TE                 300.0 K
D1             2.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  7.50 usec
PL1                -3.00 dB
SFO1          300.1321009 MHz

F2 - Processing parameters
SI                  16384
SF            300.1300133 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
CY                   0.00 cm
F1P                  9.311 ppm
F1                2794.64 Hz
F2P                 -0.378 ppm
F2                -113.33 Hz
PPMCM             0.44041 ppm/cm
HZCM            132.18057 Hz/cm
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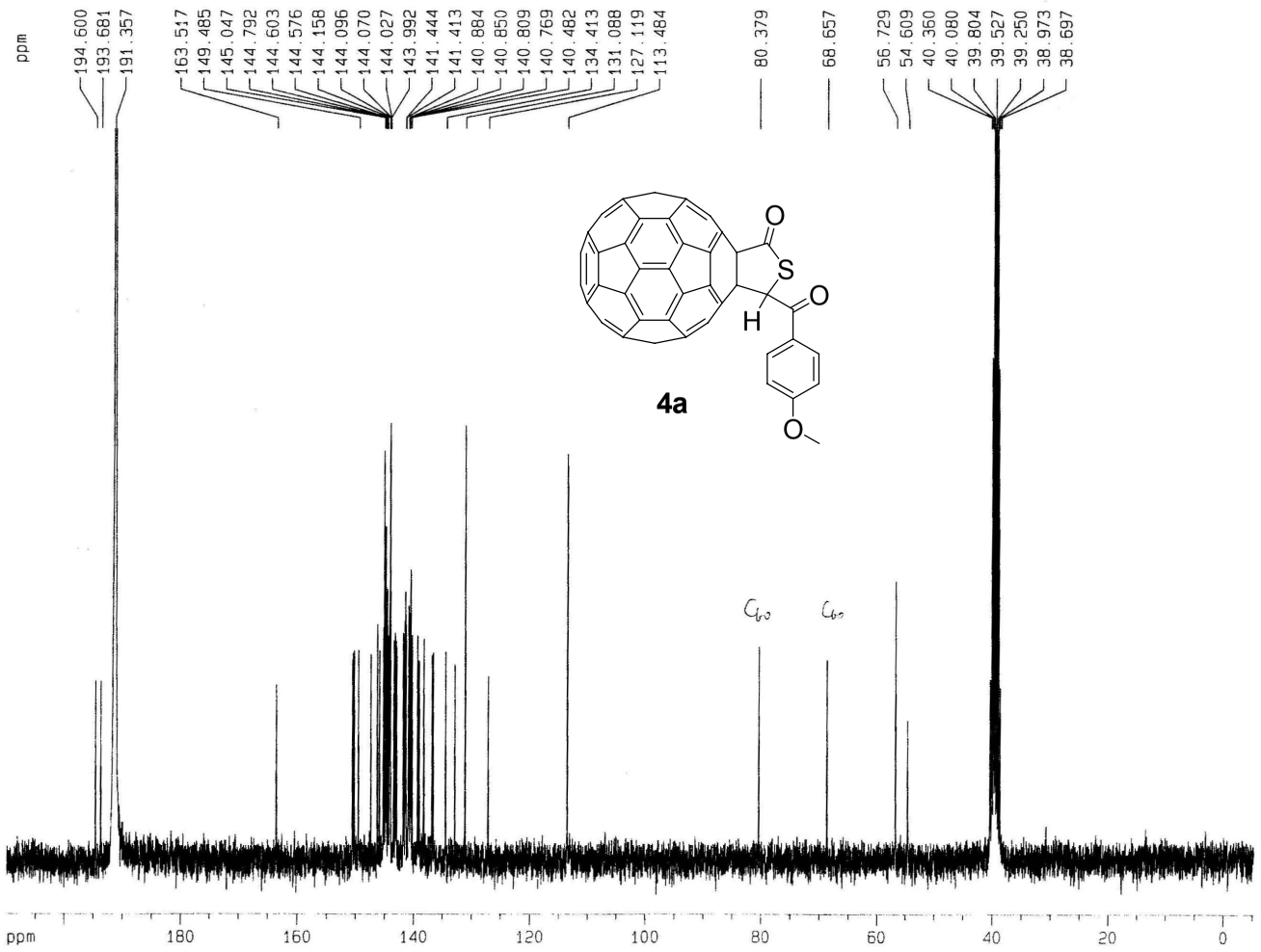


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Current Data Parameters
NAME            ljx051020
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20051021
Time                7.55
INSTRUM             av300
PROBHD   5 mm BBO BB-1H
PULPROG             zgdc
TD                 65536
SOLVENT             DMSO
NS                 14297
DS                     4
SWH          22675.736 Hz
FIDRES        0.346004 Hz
AQ            1.4451188 sec
RG                  4096
DW               22.050 usec
DE                6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                3.00 usec
PL1              -1.00 dB
SFO1         75.4768051 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2              -3.00 dB
PL12             16.00 dB
SFO2        300.1315007 MHz

F2 - Processing parameters
SI                 131072
SF          75.4678563 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                22.00 cm
CY                 0.00 cm
F1P              209.916 ppm
F1             15841.94 Hz
F2P               -5.203 ppm
F2              -392.68 Hz
PPMCM          9.77817 ppm/cm
HZCM         737.93738 Hz/cm
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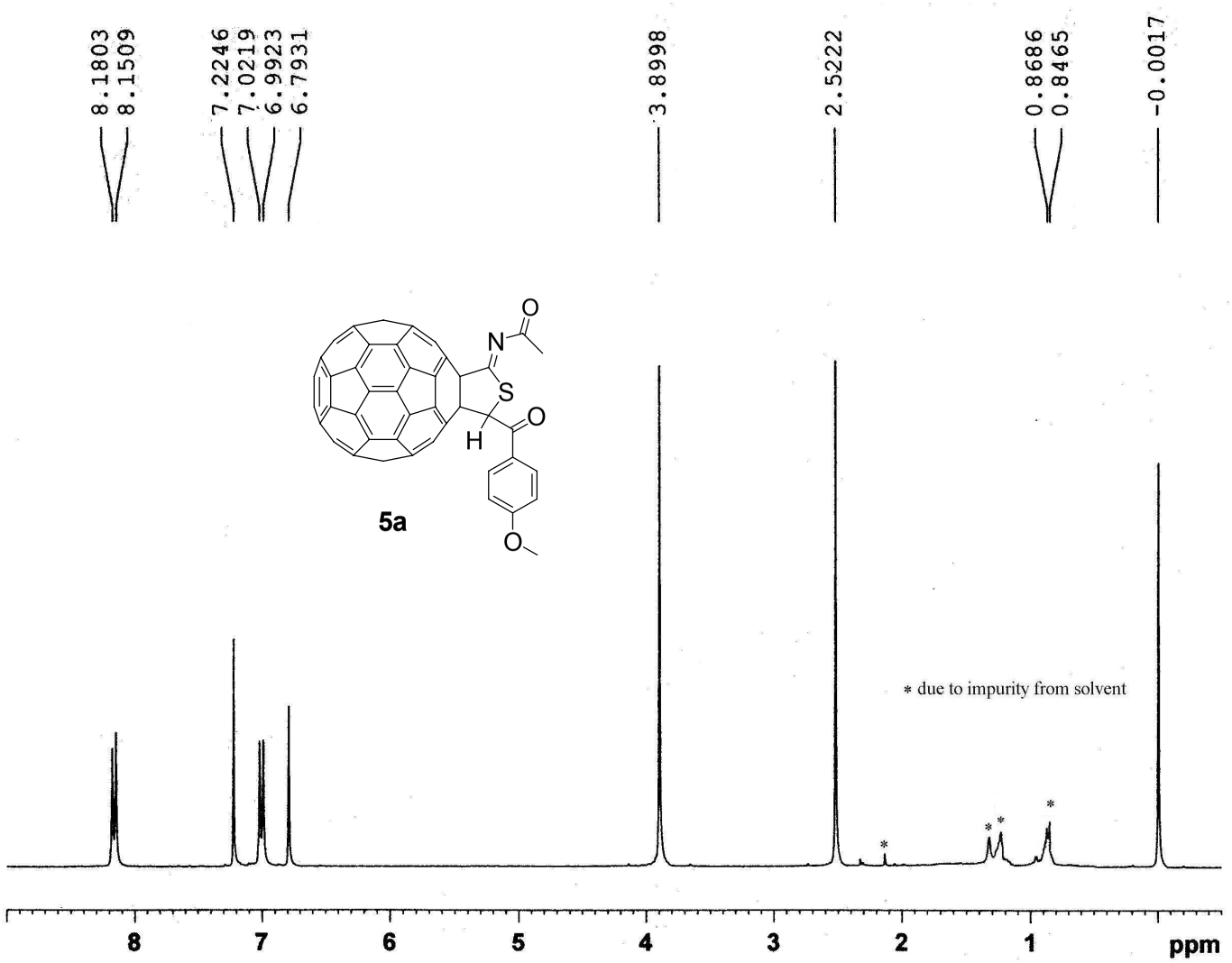


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Current Data Parameters
NAME                  1jx060118
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20060118
Time                      15.44
INSTRUM                   av300
PROBHD    5 mm BBO BB-1H
PULPROG                     zg
TD                       65536
SOLVENT                  CDCl3
NS                          41
DS                           2
SWH                   5995.204 Hz
FIDRES                0.091480 Hz
AQ                    5.4657526 sec
RG                        406.4
DW                       83.400 usec
DE                        6.00 usec
TE                       300.0 K
D1                    2.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                         7.50 usec
PL1                       -3.00 dB
SFO1                300.1321009 MHz

F2 - Processing parameters
SI                       16384
SF                  300.1300168 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
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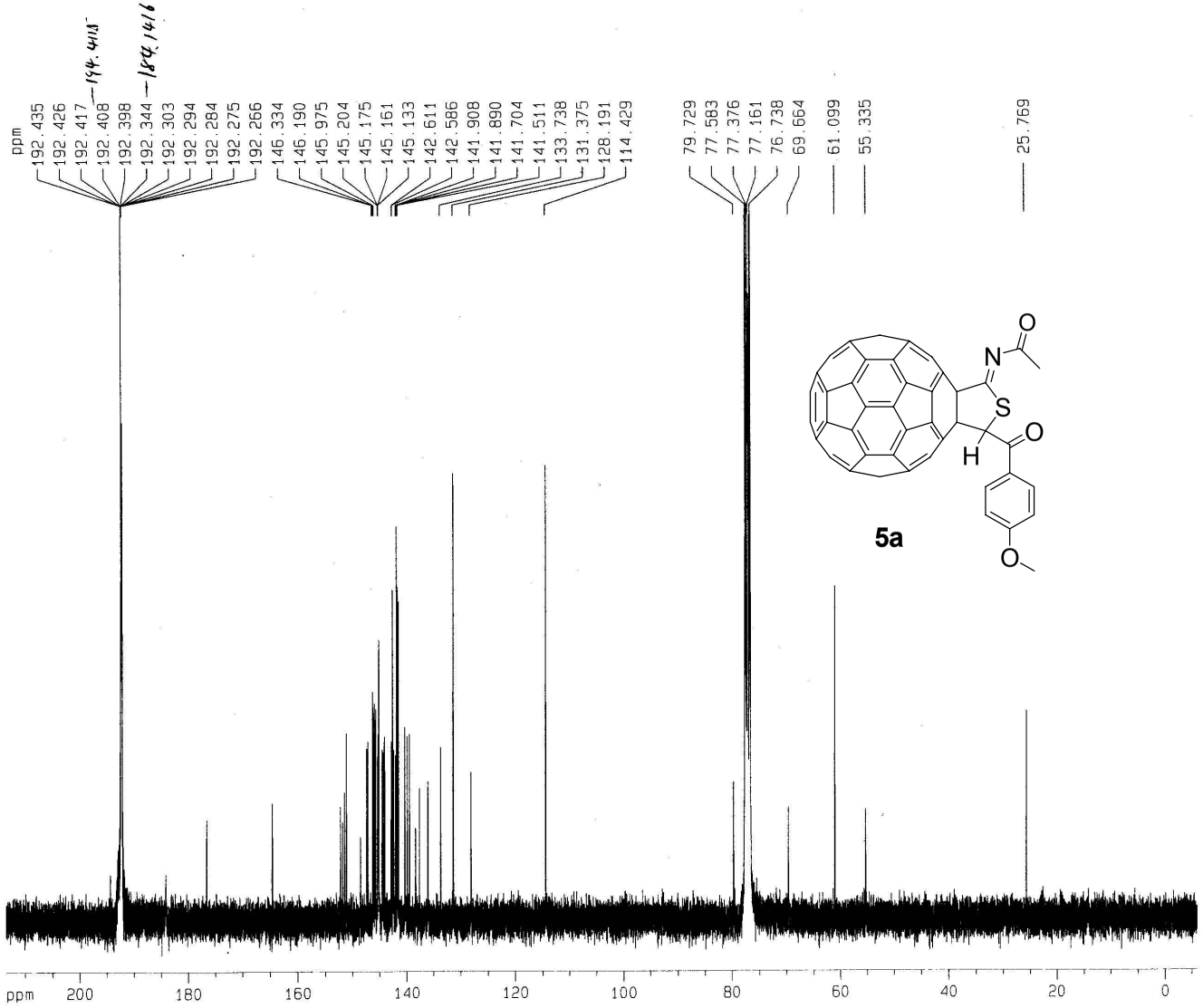


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Current Data Parameters
NAME                 ljx060119
EXPNO                        4
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20060121
Time                      8.38
INSTRUM                  av300
PROBHD    5 mm BBO BB-1H
PULPROG                   zgdc
TD                       65536
SOLVENT                  CDCl3
NS                       47310
DS                           4
SWH                22675.736 Hz
FIDRES             0.346004 Hz
AQ                 1.4451188 sec
RG                        6502
DW                   22.050 usec
DE                    6.00 usec
TE                   300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                   3.00 usec
PL1                  -1.00 dB
SFO1               75.4768051 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                80.00 usec
PL2                  -3.00 dB
PL12                 16.00 dB
SFO2              300.1315007 MHz

F2 - Processing parameters
SI                       131072
SF                 75.4677696 MHz
WDW                        EM
SSB                         0
LB                   0.20 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                   22.00 cm
CY                    0.00 cm
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HZCM               752.76959 Hz/cm
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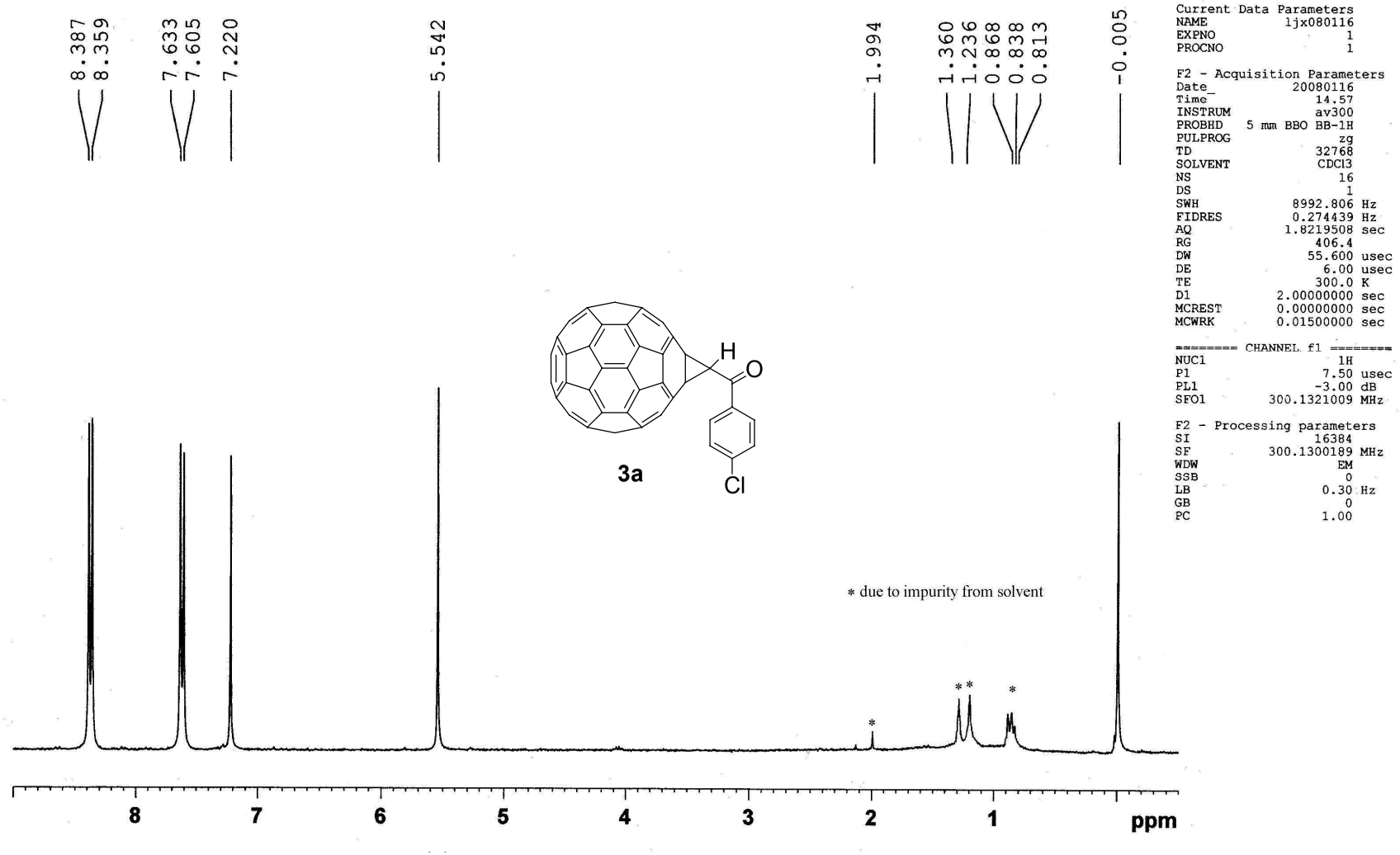


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Current Data Parameters
NAME                  ljx080116
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20080116
Time                      14.57
INSTRUM                   av300
PROBHD    5 mm BBO BB-1H
PULPROG                      zg
TD                        32768
SOLVENT                   CDCl3
NS                           16
DS                            1
SWH                    8992.806 Hz
FIDRES                 0.274439 Hz
AQ                     1.8219508 sec
RG                         406.4
DW                        55.600 usec
DE                         6.00 usec
TE                        300.0 K
D1                     2.00000000 sec
MCREST                 0.00000000 sec
MCWRK                  0.01500000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                         7.50 usec
PL1                       -3.00 dB
SFO1                  300.1321009 MHz

F2 - Processing parameters
SI                        16384
SF                    300.1300189 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
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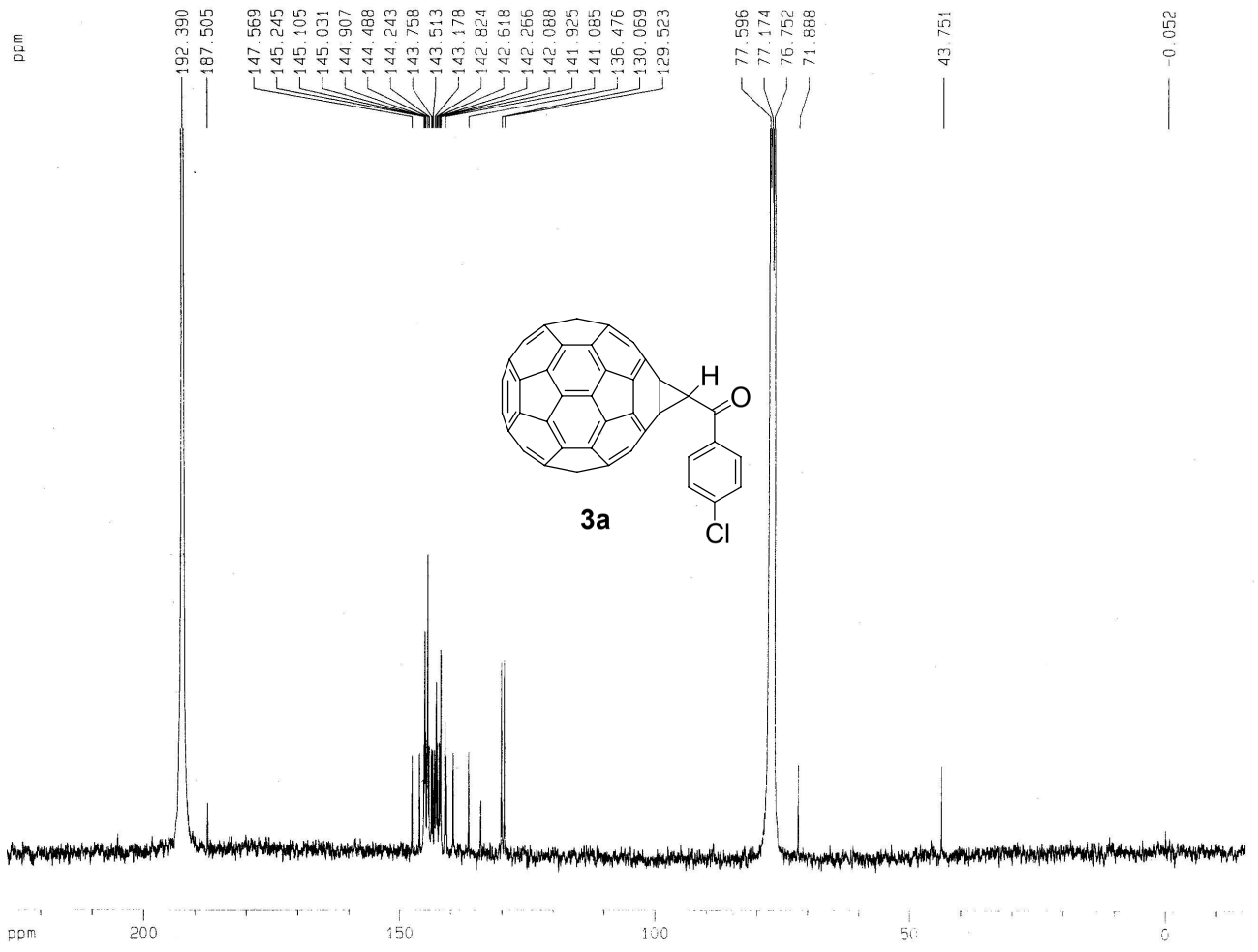


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Current Data Parameters
NAME                  ljx080116
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20080201
Time                       8.21
INSTRUM                   av300
PROBHD    5 mm BBO BB-1H
PULPROG                    zgdc
TD                        65536
SOLVENT                   CDCl3
NS                        22566
DS                            4
SWH                37878.789 Hz
FIDRES              0.577984 Hz
AQ                  0.8651252 sec
RG                         8192
DW                    13.200 usec
DE                     6.00 usec
TE                     300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                    3.00 usec
PL1                  -1.00 dB
SFO1               75.4768051 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                  -3.00 dB
PL12                 16.00 dB
SFO2              300 1315007 MHz

F2 - Processing parameters
SI                       131072
SF                 75.4677667 MHz
WDW                        EM
SSB                         0
LB                     4.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
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CY                      0.00 cm
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F1                  17095.18 Hz
F2P                  -15.673 ppm
F2                  -1182.84 Hz
PPMCM               11.00892 ppm/cm
HZCM               830.81897 Hz/cm
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