Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Electronic Supporting Information

New Synthesis of Spirocycles by Utilizing *in situ* Forming Hypervalent Iodine Species

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(1) General Information. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ with tetramethylsilane as an internal standard. Data are reported as follows: chemical shift in ppm (δ), integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad singlet, m = multiplet), coupling constant (Hz). Absorptions for infrared spectra (IR) are reported in reciprocal centimeters with the following relative intensities: s (strong), m (medium), or w (weak).

(2) Materials. Aryl alkynes **1a** and **1b** were obtained according to the literature procedures.[1)] Alkynes **1g**, **1h**, and **1k** were prepared from corresponding aliphatic bromides and lithium acetylides by substitutions.[2),3)] Other alkynes were obtained by general esterification methods from corresponding phenols and alkyne carboxylic acids.[4)] Regarding the bis(iodoarene)s,

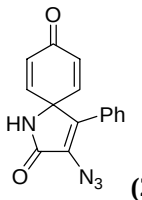
simple **3h** is commercially available and used without purification. The alkylated bis(iodoarene)s **3i** and **3l** were readily prepared in single step from 3,5-dimethyliodobenzene and 3,4-dimethyliodobenzene by the oxidative biaryl coupling method developed by us.[5),6)] Other **3j** and **3k** were prepared according to the literatures.[7),8)] We used commercial nucleophiles ($Bu_4N^+N_3^-$, $NaNO_2$, KSCN, $NaSO_2Tol$, or $Bu_4N^+Br^-$), solvents, and *p*-toluenesulfonic acid monohydrate (*p*-$TsOH{\cdot}H_2O$). Other chemicals are commercially available and used as recieved.

(3) Azidative Spirocyclization of Aryl Alkyns 1 Using Bis(iodoarene) 3h: The General Procedure (Tables 1 and 2)

To a stirred solution of bis(iodoarene) **3h** (22.3 mg, 0.055 mmol) in 2,2,2-trifluoroethanol (3.6 mL) was added *m*-chloroperbenzoic acid (ca. 69% purity with abt. 25% water, 25.3 mg, 0.11 mmol) and *p*-toluenesulfonic acid monohydrate (20.9 mg, 0.11 mmol) at room temperature. After 30 minutes, aryl alkyne **1b** (25.2 mg, 0.10 mmol) was then added to the solution, and the mixture was stirred for additional 3 hours to prepare spirocyclized iodonium salt. The solvent was removed under vaccum, and chloroform (3 mL) was added to dissolve the *in situ* prepared salt. To the solution $Bu_4N^+N_3^-$ (31.3 mg, 0.11 mmol) in small amount of chloroform was dropwise added, and it was stirred for 12 hours at room temperature. After the completion of the reaction, the mixture was quenched with saturated aqueous sodium hydrogen carbonate and the aqueous phase was extracted with dichloromethane. The extract was treated with anhydrous Na_2SO_4 and then evaporated to dryness. The crude residue was purified by column chromatography on silica gel [hexane/AcOEt (5/1)] to give a pure **2b/N$_3$** (14.0 mg) in >99% yield.

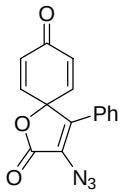
The reactions were typically carried out using 0.1 - 1.0 mmol of the aryl alkynes **1**.

(4) Chemical Data of Spirocyclized Azides 2a-k/N$_3$



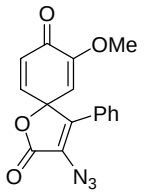
(2a/N$_3$)

Slightly yellow solid; mp. 268-269 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.99 (1H, s), 6.42 (2H, d, *J* = 10.0 Hz), 6.64 (2H, d, *J* = 10.0 Hz), 7.31-7.33 (3H, m), 7.56-7.59 (2H, m) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 59.6, 127.6, 128.7, 129.5, 129.6, 129.8, 131.3, 133.1, 146.3, 166.9, 184.2 ppm; IR (KBr): 3304 m, 2234 w, 2123 s, 1669 s, 1632 s, 1546 w, 1513 m, 1495 m, 1445 m, 1374 s, 1301 s, 1139 m, 1097 m, 1062 m, 1003 w, 966 w, 883 s, 831 m, 689 s, 651 m, 627 m cm^{-1}; HRFABMS: calcd for $C_{15}H_{10}N_4O_2$ $[M + H]^+$ 279.0882, found 279.0901.



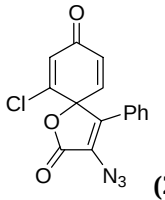
(2b/N_3)

Slightly yellow solid; mp 101-102 ^{o}C; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 6.47 (2H, d, *J* = 10.0 Hz), 6.64 (2H, d, *J* = 10.0 Hz), 7.33-7.38 (3H, m), 7.62 (2H, d, *J* = 8.0 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 79.0, 124.6, 127.8, 128.1, 128.9, 130.9, 132.0, 138.5, 143.3, 166.2, 183.8 ppm; IR (KBr): 3057 m, 2453 w, 2184 m, 2129 s, 1872 s, 1768 s, 1632 s, 1495 m, 1445 m, 1363 s, 1283 m, 1218 s, 1175 m, 1131 m, 1093 m, 1068 m, 1007 m, 894 m, 755 s, 689 m cm^{-1}; HRFABMS: calcd for $C_{15}H_9N_3O_3$ $[M]^+$ 279.0644, found 279.0641.



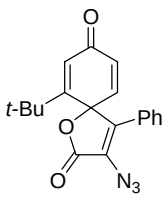
(2c/N_3)

Slightly yellow solid; mp 136-137 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.67 (3H, s), 5.50 (1H, s), 6.46 (1H, d, *J* = 10.0 Hz), 6.63 (1H, d, *J* = 10.0 Hz), 7.34-7.38 (3H, m), 7.64-7.66 (2H, m) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.5, 81.7, 110.2, 124.1, 127.8, 128.2, 128.9, 130.7, 131.1, 139.0, 144.0, 152.8, 166.2, 179.4 ppm; IR (KBr): 2124 s, 1767 s, 1683 s, 1646 m, 1618 m,1446 w, 1362 m, 1209 m, 1128 w, 979 w, 906 w, 841 w, 762 m , 700 w, 647 w, 617 w, 600 w cm^{-1}; HRFABMS: calcd for $C_{16}H_{11}N_3O_4$ $[M + Na]^+$ 332.0647, found 332.0651.



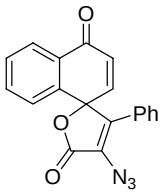
(2d/N_3)

Slightly yellow solid; mp 116-117 ^{o}C ; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 6.49 (1H, d, *J* = 9.8 Hz), 6.67 (1H, s), 6.70 (1H, d, *J* = 10.0 Hz), 7.35-7.41 (3H, m), 7.54-7.60 (2H, m) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 80.5, 126.0, 127.5, 127.6, 129.1, 131.0, 131.2, 131.4, 137.0, 142.6, 150.1, 165.8, 182.7 ppm; IR (KBr): 3062 m, 2925 m, 2853 w, 2131 s, 1774 s, 1666 s, 1572 s, 1446 m, 1371 m, 1274 m, 1104 m, 1011 m, 982 m, 911 s, 845 s, 819 m, 778 w, 742 s, 688 m cm^{-1}.



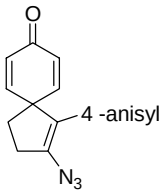
(2e/N_3)

Slightly yellow solid; mp 115-116 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.13 (9H, s), 6.31-6.38 (2H, m), 6.56 (1H, s), 7.34-7.36 (3H, m) 7.57-7.59 (2H, m) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.9, 38.1, 82.9, 124.8, 127.9, 128.5, 128.9, 128.9, 130.8, 130.8, 140.4, 147.8, 161.8, 166.9, 185.5 ppm; IR (KBr): 3059 m, 2971 s, 2432 w, 2358 m, 2129 s, 1770 s, 1668 s, 1632 s, 1574 m, 1482 m, 1446 m, 1363 s, 1324 m, 1286 m, 1221 s, 1127 m, 1103 m, 1033 m, 998 s, 903 m, 866 w, 819 m, 786 m, 750 s, 690 m cm^{-1}; HRFABMS: calcd for $C_{19}H_{17}N_3O_3$ $[M + Na]^+$ 358.1168, found 358.1149.



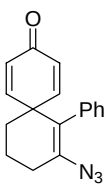
(2f/N_3)

Slightly yellow solid; mp 135-136 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.62 (1H, d, *J* = 10.0 Hz), 6.69 (1H, d, *J* = 10.0 Hz), 7.20-7.33 (6H, m), 7.53-7.61 (2H, m), 8.21 (1H, d, *J* = 7.6 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 80.5, 124.1, 126.3, 127.6, 127.9, 128.0, 128.8, 130.3, 130.5, 130.8, 132.2, 134.1, 137.1, 140.4, 143.3, 166.9, 183.0 ppm; IR (KBr): 2130 s, 1764 s, 1671 s, 1445 m, 1363 m, 1297 m, 1218 s, 1119 m, 981 m, 913 m, 758 s, 689 w cm^{-1}; HRFABMS: calcd for $C_{19}H_{12}O_3N_3$ $[M+H]^+$ 330. 0879, found 330.0882.



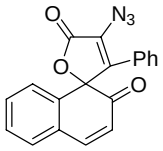
(2g/N_3)

Slightly yellow solid; mp 88-89 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.22 (2H, t, *J* = 7.4 Hz), 2.90 (2H, t, *J* = 7.4 Hz), 3.74 (3H, s), 6.28 (2H, d, *J* = 10.0 Hz), 6.75 (2H, d, *J* = 9.0 Hz), 6.94 (2H, d, *J* = 10.0 Hz), 7.18 (2H, d, *J* = 9.0 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.0, 34.4, 55.2, 55.4, 113.5, 125.5, 125.6, 128.7, 128.8, 135.6, 154.0, 158.9, 185.6 ppm; IR (KBr): 2932 w, 2851 w, 2361 m, 2109 s, 1662 s, 1608 m, 1572 w, 1510 m, 1463 w ,1399 w, 1340 m, 1292 m, 1250 m, 1181 m, 1071 w, 1030 w, 913 m, 859 w, 833 w, 743 m, 697 w, 668 w, 615 w cm^{-1}; HRFABMS: calcd for $C_{17}H_{15}N_3O_2$ $[M + Na]^+$ 316.1062, found 316.1060.



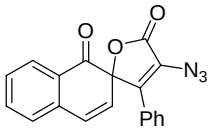
(2h/N_3)

Slightly yellow solid; mp 106-107 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.84-1.87 (2H, m), 2.03-2.09 (2H, m), 2.56 (2H, t, *J* = 6.5 Hz), 6.09 (2H, d, *J* = 10.0 Hz), 6.87-6.94 (4H, m), 7.17-7.21 (3H, m) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.7, 25.5, 34.3, 45.6, 124.3, 127.6, 127.7, 128.9, 128.9, 133.9, 136.5, 153.4, 185.5 ppm; IR (KBr): 3052 w, 2930 m, 2835 m, 2094 s, 1663 s, 1622 m, 1630 s, 1604 m ,1491 w, 1442 w, 1399 w, 1363 w, 1281 m, 1178 w, 1087 w, 1029 w, 913 m, 880 w, 858 m, 752 m, 700 m, 669 m cm^{-1}; HRFABMS: calcd for $C_{17}H_{15}N_3O$ $[M + H]^+$ 278.1293, found 278.1302.



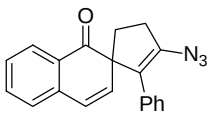
(2i/N$_3$)

Slightly yellow solid; mp 134-135 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.32 (1H, d, *J* = 10.0 Hz), 7.20-7.27 (6H, m), 7.36-7.45 (3H, m),7.57 (1H, d, *J* = 10.0 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 82.9, 124.1, 124.2, 128.1, 128.3, 128.6, 128.8, 130.1, 130.2, 130.6, 130.7, 131.3, 135.5, 139.5, 146.3, 167.6, 190.8 ppm; IR (KBr): 2930 w, 2107 s, 1660 s, 1563 w, 1445 w, 1347 w, 1277 w, 763 m, 692 w cm^{-1}; HRFABMS: calcd for $C_{19}H_{11}N_3O_3$ $[M + H]^+$ 330.0879, found 330.0818.



(2j/N$_3$)

Slightly yellow solid; mp 136-137 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.01 (1H, d, *J* = 9.8 Hz), 6.92 (1H, d, *J* = 9.8 Hz), 7.22-7.30 (3H, m), 7.34 (1H, d, *J* =7.6 Hz), 7.44-7.49 (3H, m), 7.69 (1H, t, *J* = 7.8 Hz), 8.07 (1H, d, *J* = 7.8 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 81.2, 124.0, 127.8, 127.9, 128.4, 128.6, 128.8, 128.9, 129.7, 130.3, 131.9, 136.1, 136.2, 138.8, 167.5, 190.4 ppm; IR (KBr): 3062 w, 2929 w, 2360 w, 2124 s, 1767 s, 1691 s, 1628 m, 1496 w, 1447 m, 1368 s ,1289 m, 1217 s, 1154 m, 1106 w, 1069 w, 1021 w, 1000 m, 999, w, 816 w, 745 m, 755 m, 692 m, 656 m cm^{-1}; HRFABMS: calcd for $C_{19}H_{11}N_3O_3$ $[M + H]^+$ 330.0879, found 330.0873.



(2k/N$_3$)

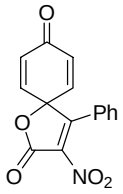
Slightly orange solid; mp 92-93 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.05-2.13 (1H, m), 2.31-2.38 (1H, m), 2.82-2.89 (1H, m), 3.02-3.10 (1H, m), 6.20 (1H, d, *J* = 9.5 Hz), 6.59 (1H, d, *J* = 9.8Hz), 7.08-7.17 (5H, m), 7.26 (1H, d, *J* =8.0 Hz), 7.37 (1H, t, *J* =7.7 Hz), 7.59 (1H, t, *J* = 7.5 Hz), 8.07 (1H, d, *J* = 7.6 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.4, 34.9, 63.1, 124.2, 127.0, 127.1, 127.4, 127.5, 127.6, 128.0, 128.1, 128.9, 133.3, 134.7, 137.1, 138.1, 138.9, 200.7 ppm; IR (KBr): 3055 w, 3030 w, 2937

m, 2848 w, 2360 w, 2111 s, 1672 s, 1596 s, 1496 m, 1450 m ,1396 w, 1346 s, 1283 s, 1217 m, 1158 m, 1022 m, 990 m, 965 w, 912 s, 868 m, 795 s, 740 s, 666 m cm^{-1}; HRFABMS: calcd for $C_{20}H_{15}N_3O$ $[M + Na]^+$ 336.113, found 336.1106.

(5) Introduction of Other Ttypes of Nucleophiles (Scheme 2)

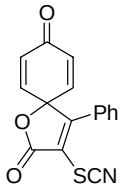
By the similar treatments of $NaNO_2$, KSCN, $NaSO_2Tol$, or $Bu_4N^+Br^-$ instead of $Bu_4N^+N_3^-$ mentioned above, the corresponding functionalized spirocycles **2** produced from aryl alkynes **1** in good yields. Insoluble salts to chloroform, such as, $NaNO_2$, KSCN, and $NaSO_2Tol$, were added in one portion as fine powder. The introduction of bromo functionality was performed in acetonitrile at 60 °C.

(6) Chemical Data of the Spirocyclized Products in Scheme 2



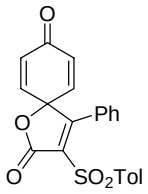
(2b/NO_2)

Colorless solid; mp 174-175 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.49 (2H, d, *J* = 10.0 Hz), 6.72 (2H, d, *J* = 10.0 Hz), 7.31 (2H, d, *J* = 8.0 Hz), 7.42 (2H, t, *J* = 8.0 Hz), 7.53 (1H, t, *J* = 8.0 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 80.0, 125.2, 127.4, 127.4, 129.5, 133.0, 133.2, 139.6, 158.6, 160.3, 182.7 ppm; IR (KBr): 3060 w, 2926 w, 1795 s, 1674 s, 1636 m, 1536 s, 1445 m, 1363 s, 1281 m, 1229 m, 1177 w, 1118 w, 1082 m, 1032 m, 897 m, 863 m, 773 w, 743 m, 695 m cm-1; HRFABMS: calcd for $C_{15}H_9NO_5$ [M]+ 283.0481, found 283.0478.



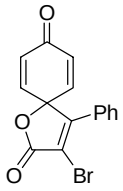
(2b/SCN)

Slightly yellow solid; mp 107-108 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.44 (2H, d, *J* = 10.0 Hz), 6.65 (2H, d, *J* = 10.0 Hz), 7.31 (2H, d, *J* = 8.0 Hz), 7.46 (2H, t, *J* = 8.0 Hz), 7.51 (1H, t, *J* = 8.0 Hz) ppm; ^{13}C NMR (100 MHz, CD_3OD): δ 83.0, 105.1, 116.2, 127.4, 127.5, 127.6, 129.5, 132.3, 132.7, 140.6, 165.9, 183.0 ppm; IR (KBr): 3058 w, 2924 w, 2164 m, 1777 s, 1672 s, 1632 m, 1612 m, 1491 w, 1444 w, 1389 w, 1282 m, 1219 s, 1175 w, 1068 m, 1005 s, 912 w, 894 w, 862 m, 776 w, 738 s, 695 m cm^{-1}; HRFABMS: calcd for $C_{16}H_9NO_3S$ $[M + H]^+$ 295.0303, found 295.0310.



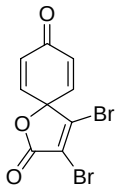
(2b/SO_2Tol)

Colorless solid; mp 208-209 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.44 (3H, s), 6.33 (2H, d, *J* = 9.8 Hz), 6.55 (2H, d, *J* = 9.8 Hz), 7.16 (2H, d, *J* = 7.3 Hz), 7.33-7.41 (4H, m), 7.47 (1H, t, *J* = 7.4 Hz) 7.86 (2H, d, *J* = 8.0 Hz) ppm; ^{13}C NMR (100 MHz, CD_3OD): δ 21.8, 82.4, 127.1, 127.5, 128.2, 129.3, 130.0, 131.3, 131.6, 132.9, 135.3, 139.9, 146.2, 164.3, 170.3, 182.9 ppm; IR (KBr): 3059 w, 2925 w, 2255 m, 1780 s, 1674 s, 1634 s, 1595 m, 1489 m, 1443 m, 1389 m, 1337 s, 1275 m, 1235 s, 1208 m, 1158 s, 1091 m, 1066 m, 1001 m, 913 s, 861 m, 830 m, 814 m, 777 m, 741 s, 703 m, 669 m, 650 m, 620 s, 604 m cm^{-1}; HRFABMS: calcd for $C_{22}H_{16}O_5S$ $[M + H]^+$ 393.0797, found 393.0796.



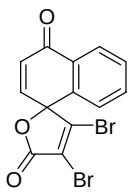
(2b/Br)

Colorless solid; mp 163-164 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.41 (2H, d, *J* = 10.0 Hz), 6.65 (2H, d, *J* = 10.0 Hz), 7.38-7.47 (5H, m) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 83.0, 112.3, 127.4, 128.6, 129.0, 131.4, 132.2, 141.6, 159.8, 166.9, 183.4 ppm; IR (KBr): 1782 s, 1672 s, 1635 m, 1581 m, 1450 w, 1387 w, 1190 s, 1131 m, 1043 m, 1013 m, 971 m, 894 m, 857 m, 816 m, 742 m, 697 m cm^{-1}; HRFABMS: calcd for $C_{15}H_{10}O_3Br$ $[M + H]^+$ 316.9813, found 316.9818.



(2l/Br)

Colorless solid; mp 185-186 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.47 (2H, d, *J* = 10.5 Hz), 6.51 (2H, d, *J* = 10.5 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 83.8, 117.4, 133.1, 140.1, 147.1, 164.9, 183.0 ppm; IR (KBr): 1780 s, 1673 s, 1635 w, 1608 w, 1389 w, 1275 w, 1216 w, 1067 w, 998 m, 897 m, 859 w, 747 m cm^{-1}; HRFABMS: calcd for $C_9H_4Br_2O_3$ $[M + H]^+$ 318.8605, found 318.8591.



(2m/Br)

Slightly yellow solid; mp 213-214 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.59 (1H, d, *J* = 10.0 Hz), 6.70 (1H, d, *J* = 10.0 Hz), 7.20 (1H, d, *J* = 7.8 Hz), 7.57-7.67 (2H, m), 8.19 (1H, d, *J* = 7.6 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 85.1, 115.8, 125.3, 127.5, 130.6, 131.0, 133.4, 133.8, 134.5, 140.3, 150.5, 165.8, 182.5 ppm; IR (KBr): 1775 s, 1673 s, 1633 w, 1602 m, 1386 w, 1302 m, 1223 m, 1150 w, 1122 w, 996 w, 979 m, 912 w, 895 w, 763 s, 743 s , 682 w cm^{-1}; HRFABMS: calcd for $C_{13}H_6O_3Br_2$ $[M+H]^+$ 368.8762, found 368.8773.

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(8) Spectrum Charts of the Spirocyclized Products 2

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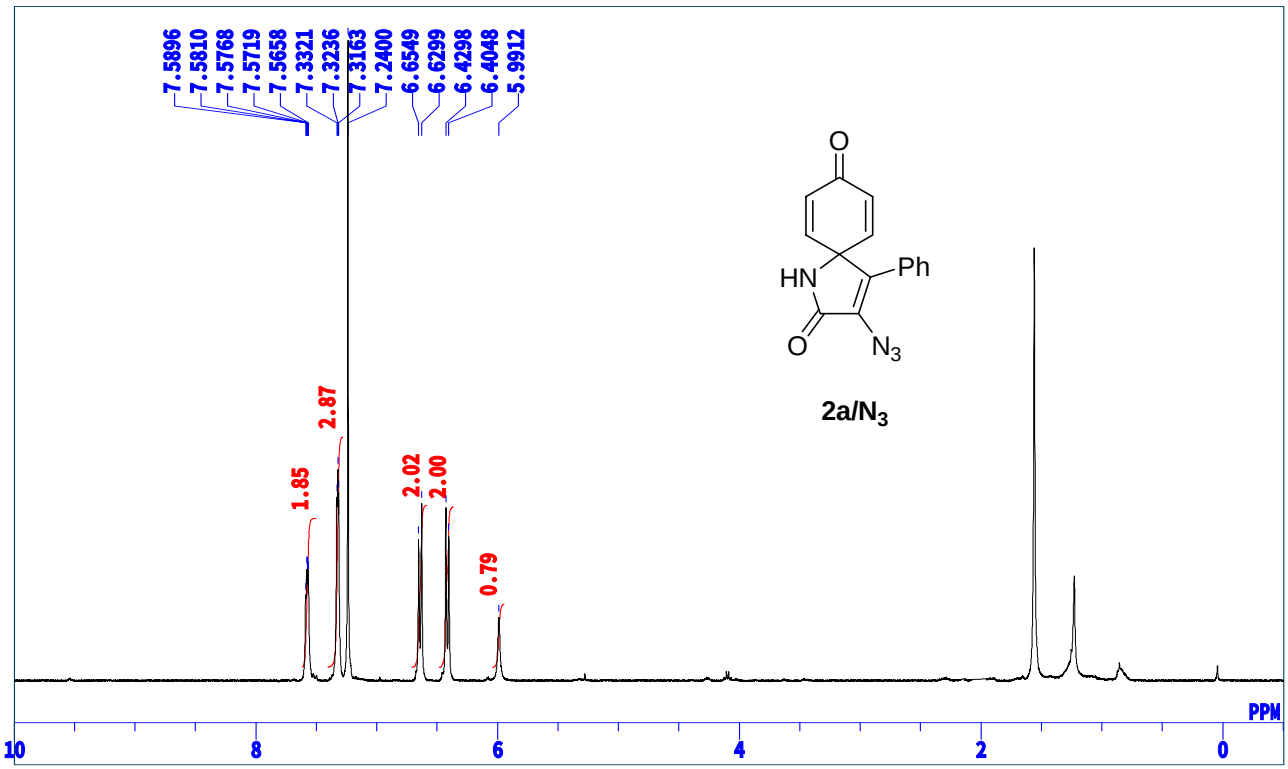

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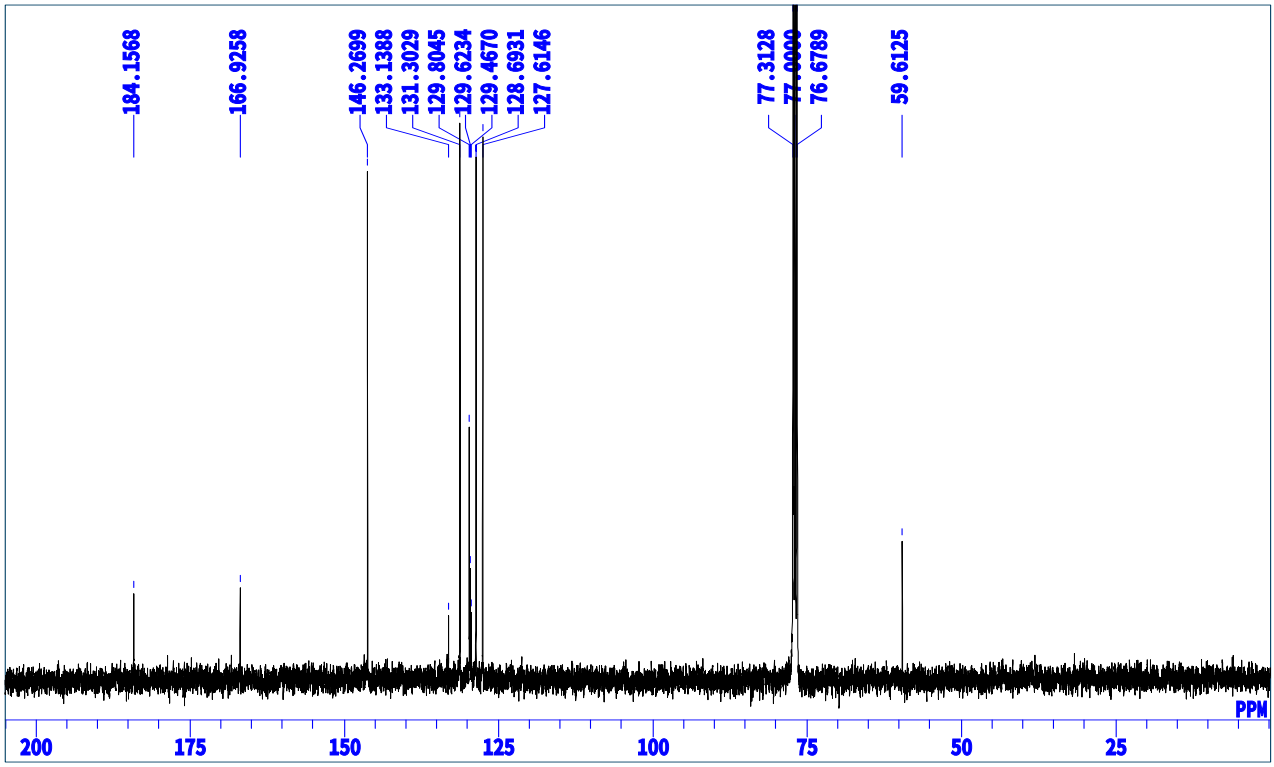

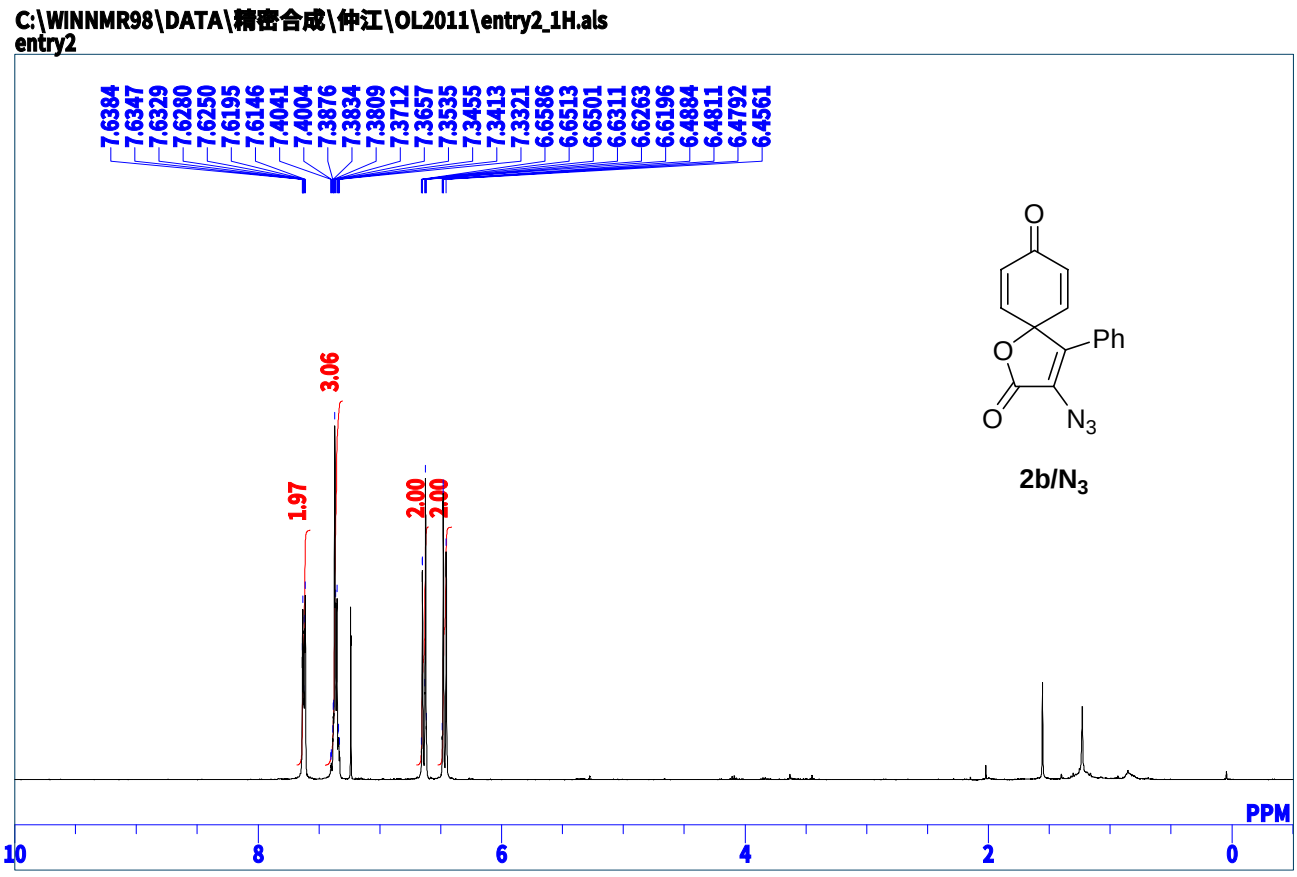
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6.4811
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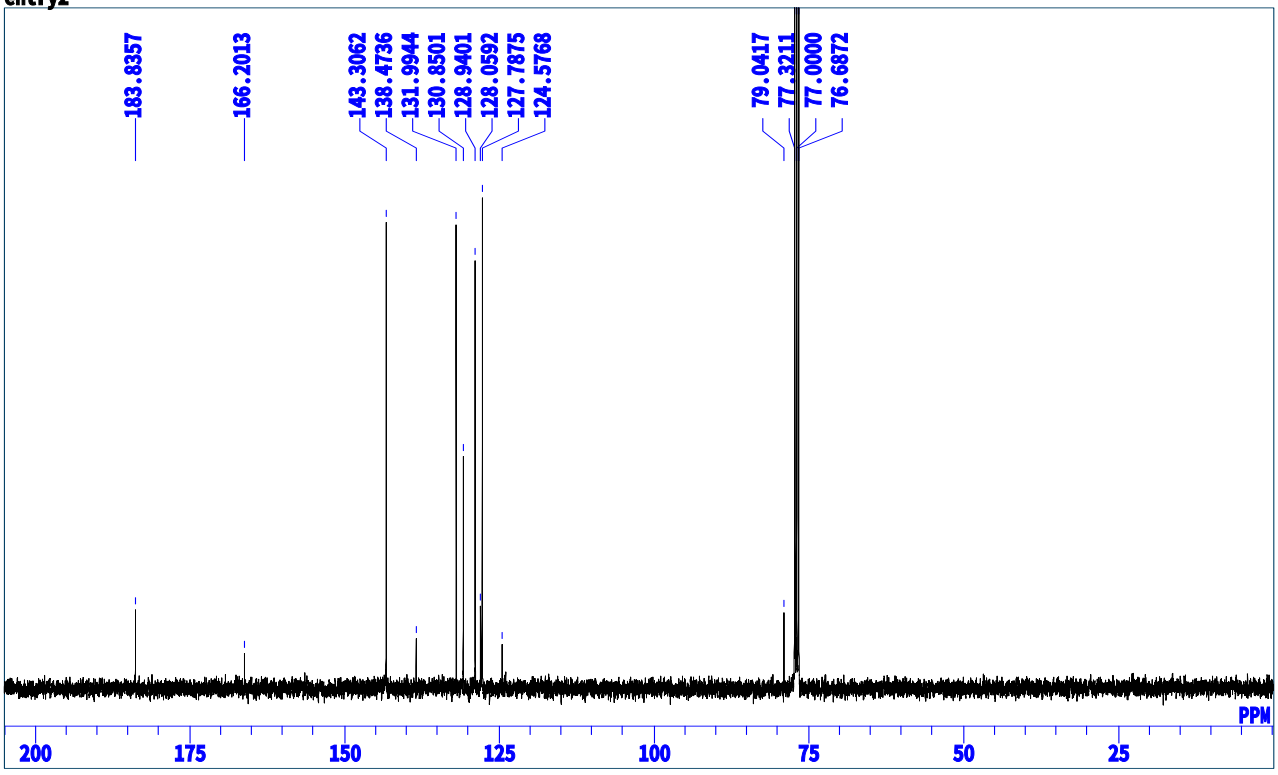
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PPM
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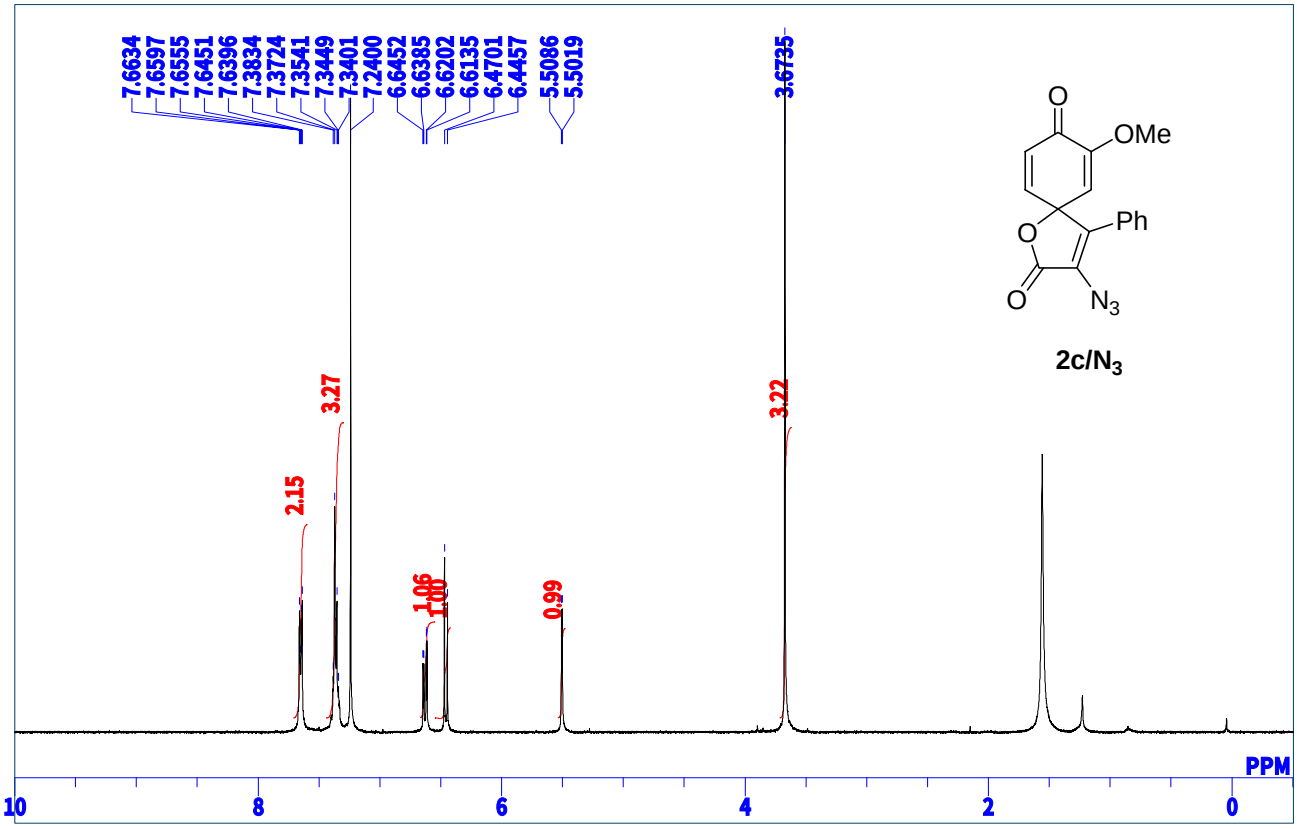

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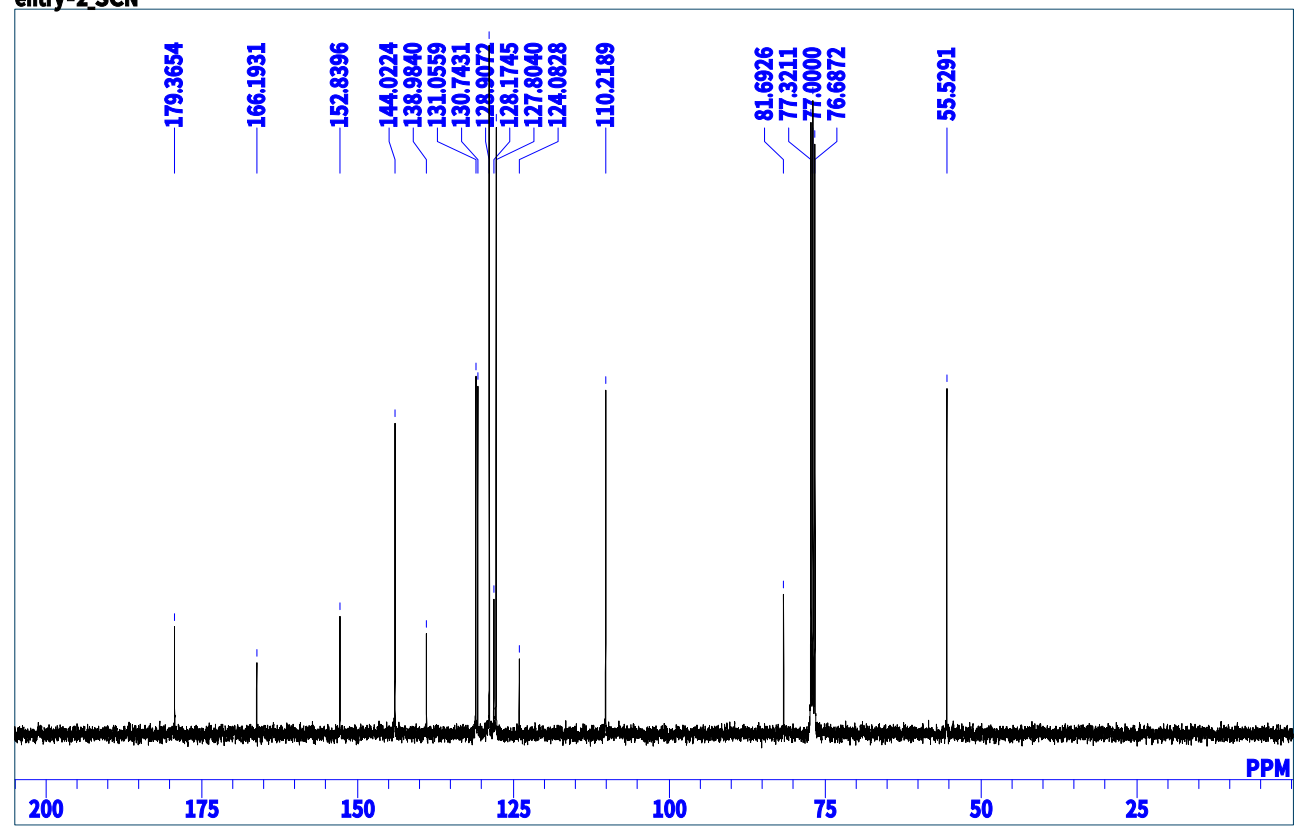

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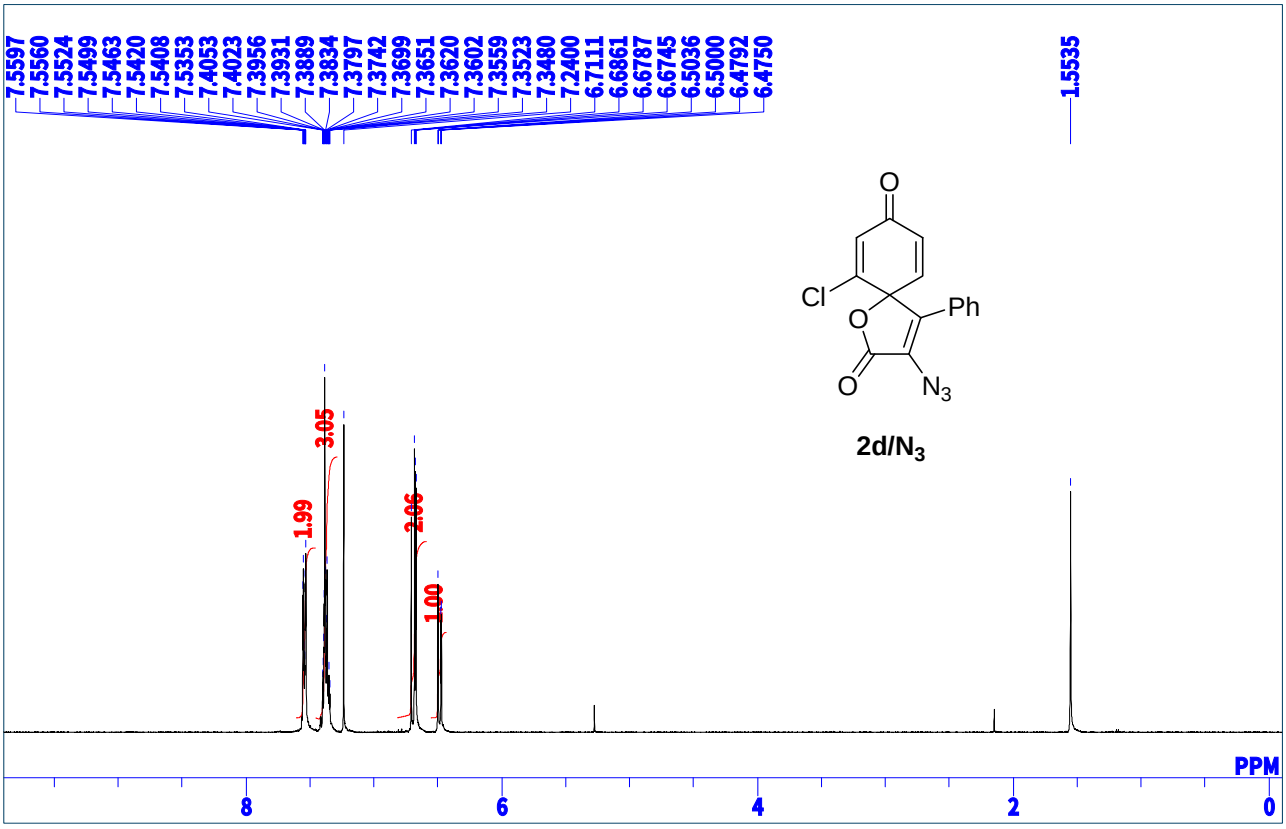

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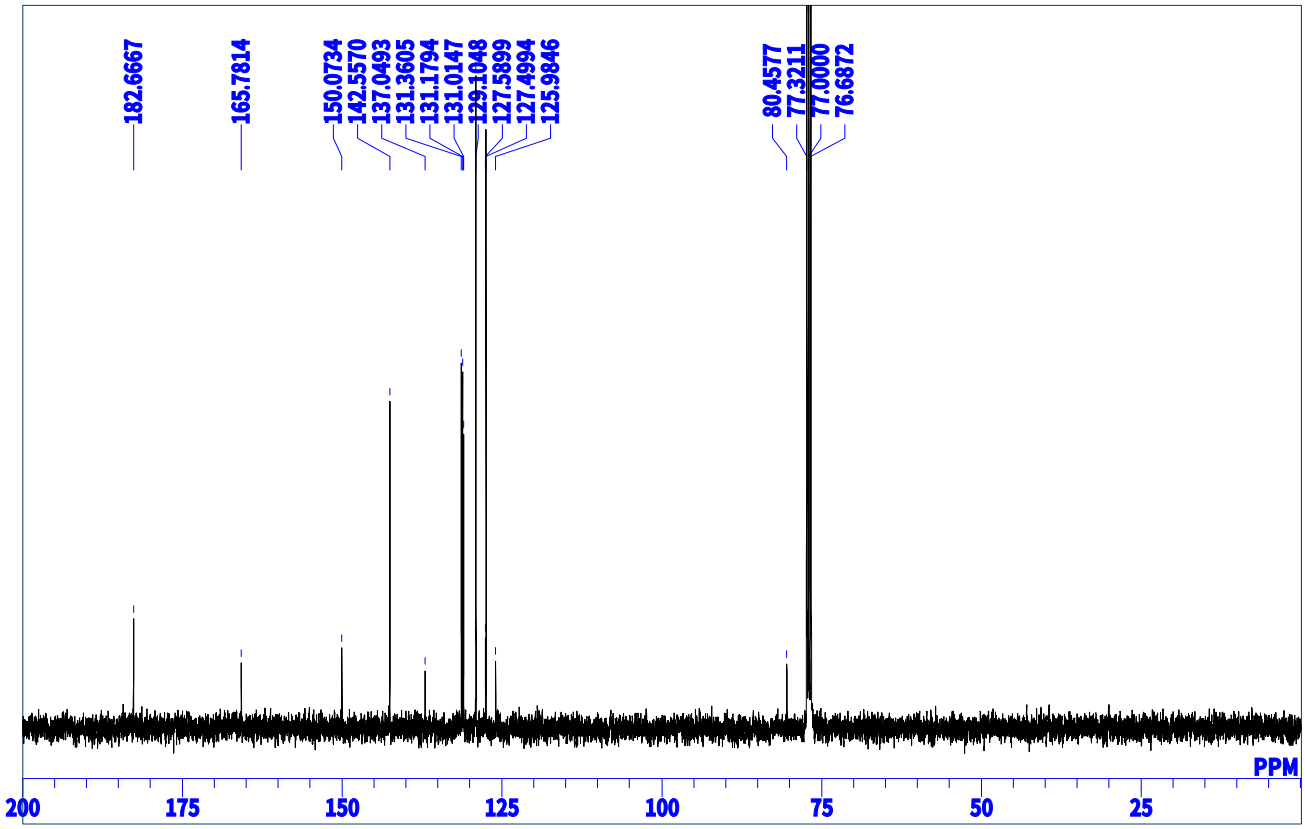

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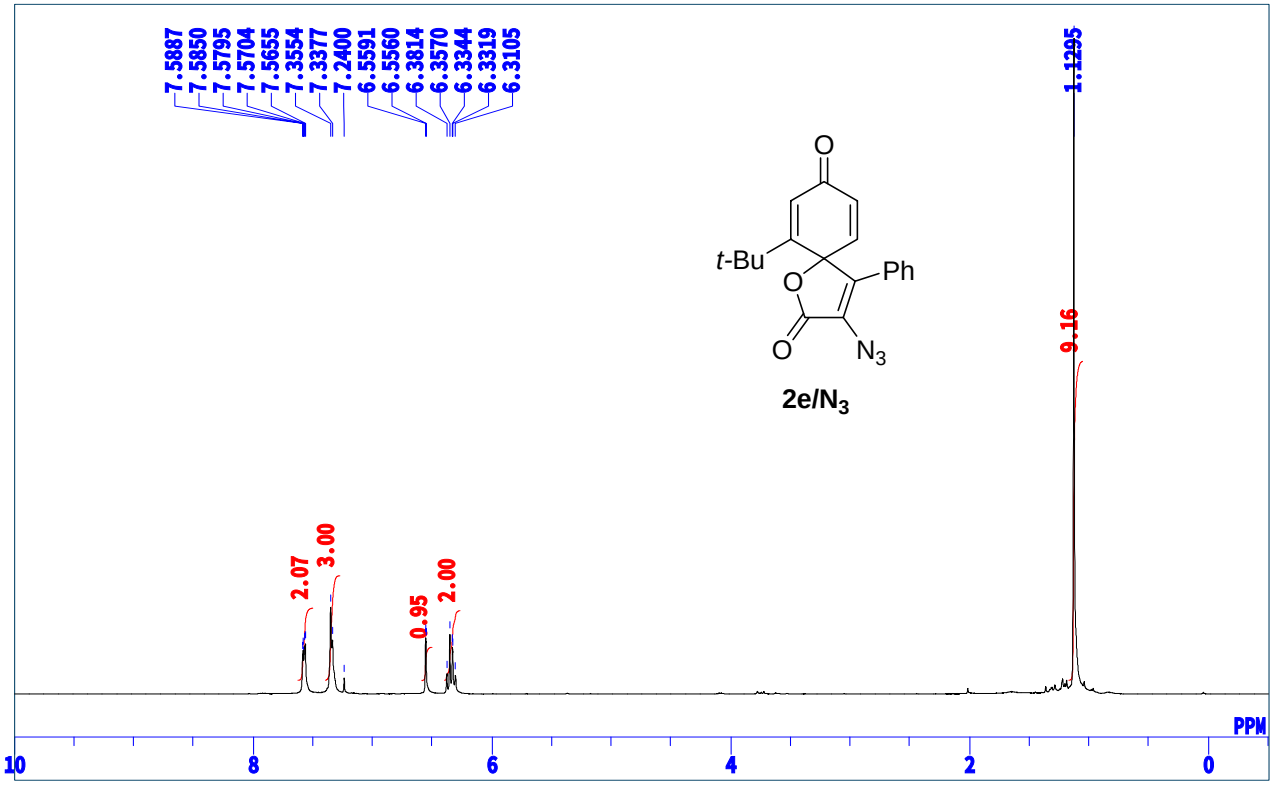

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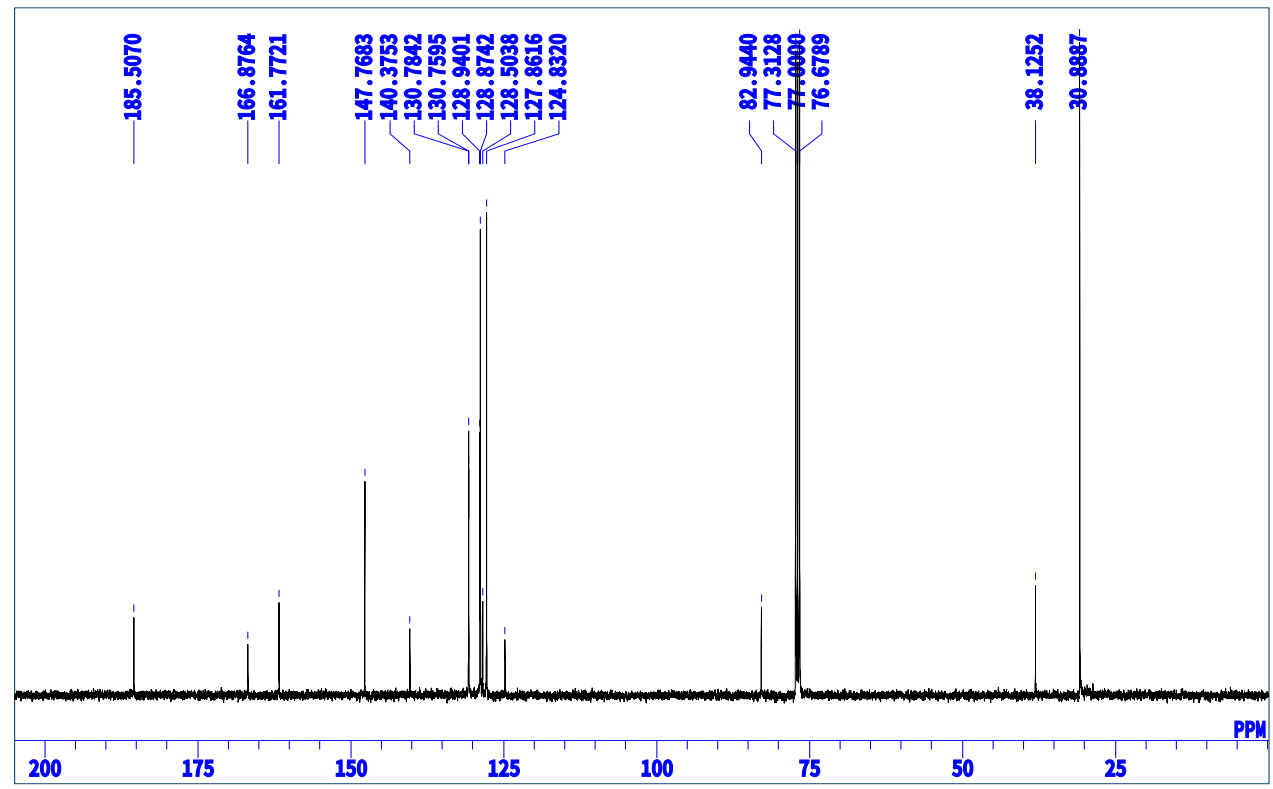

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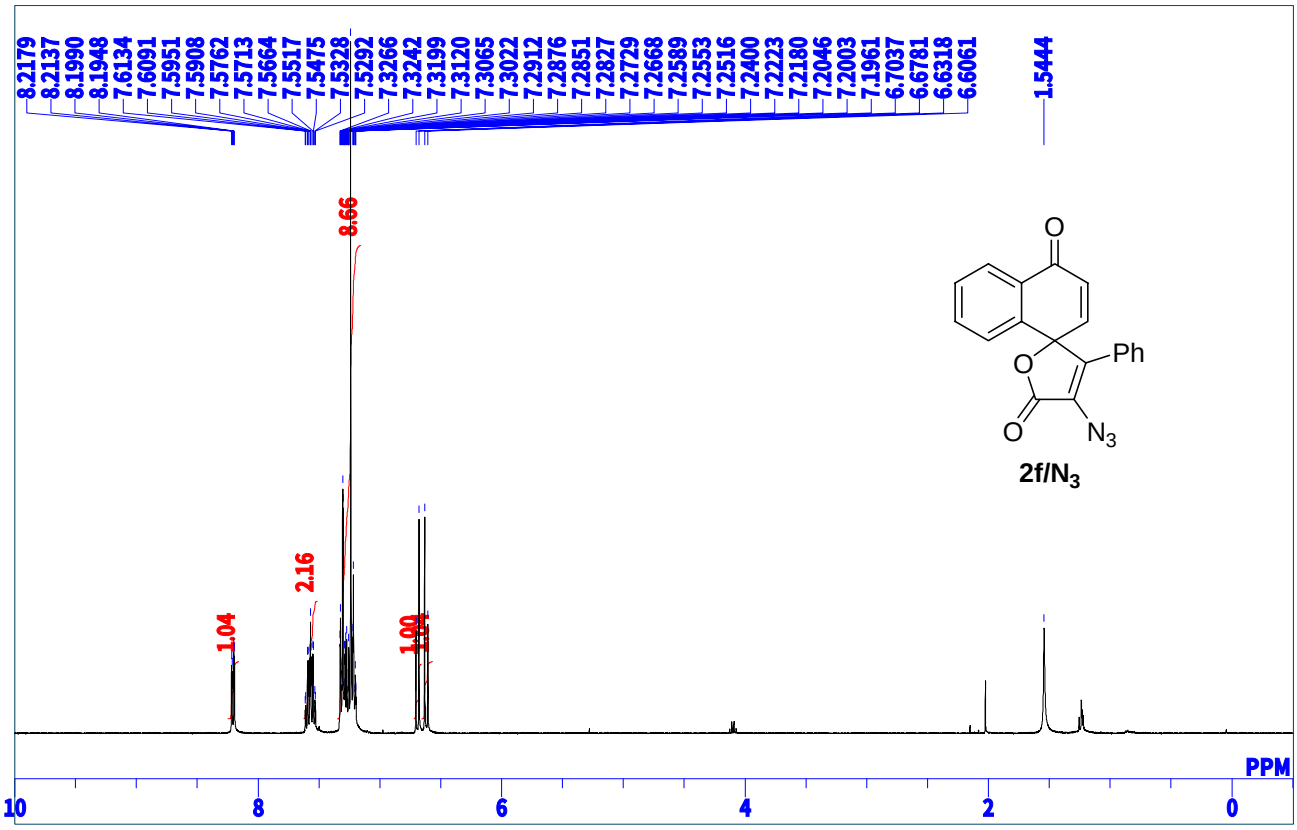

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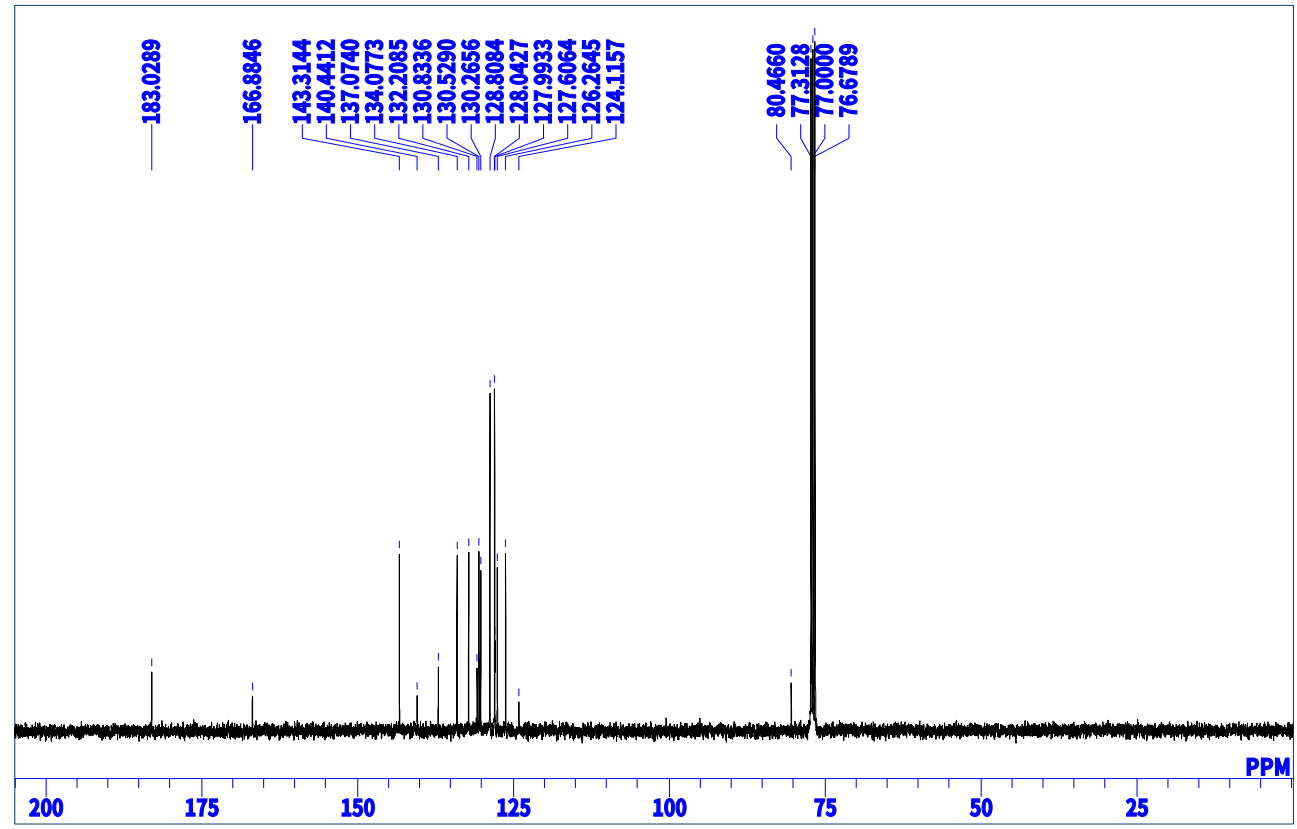

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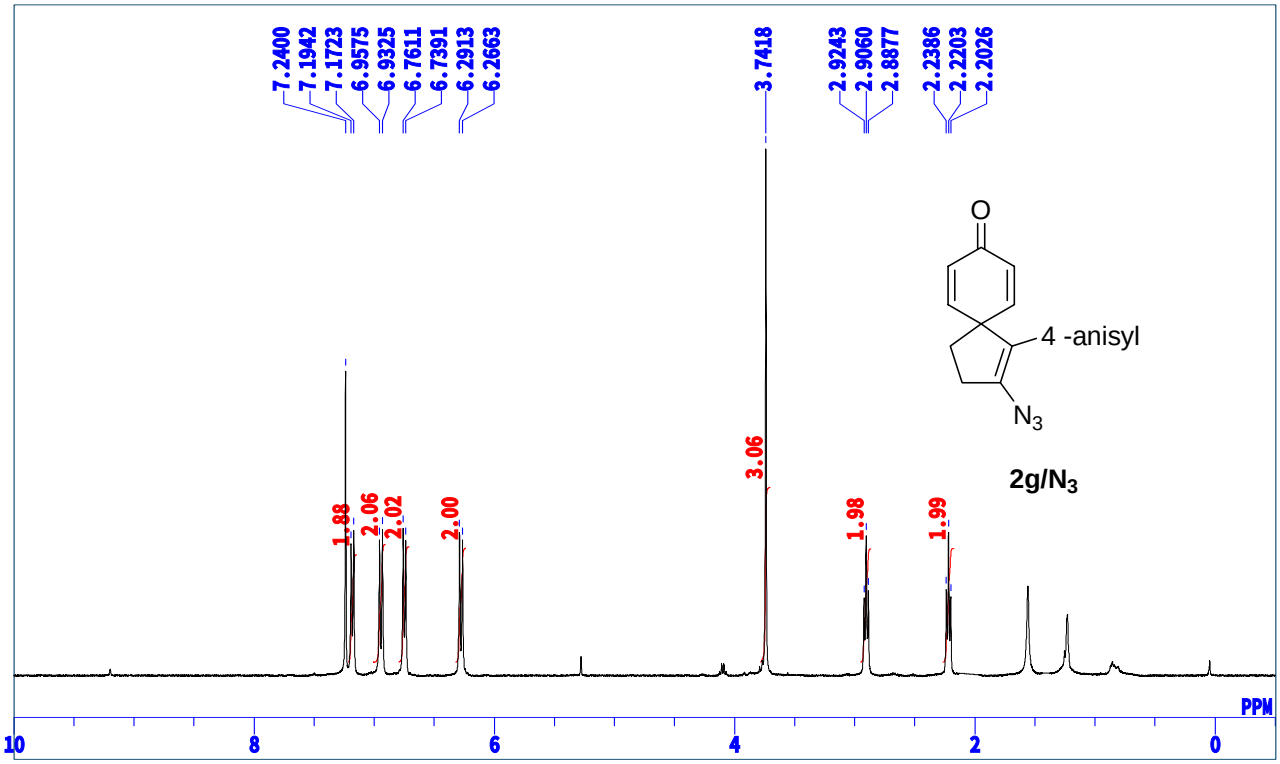

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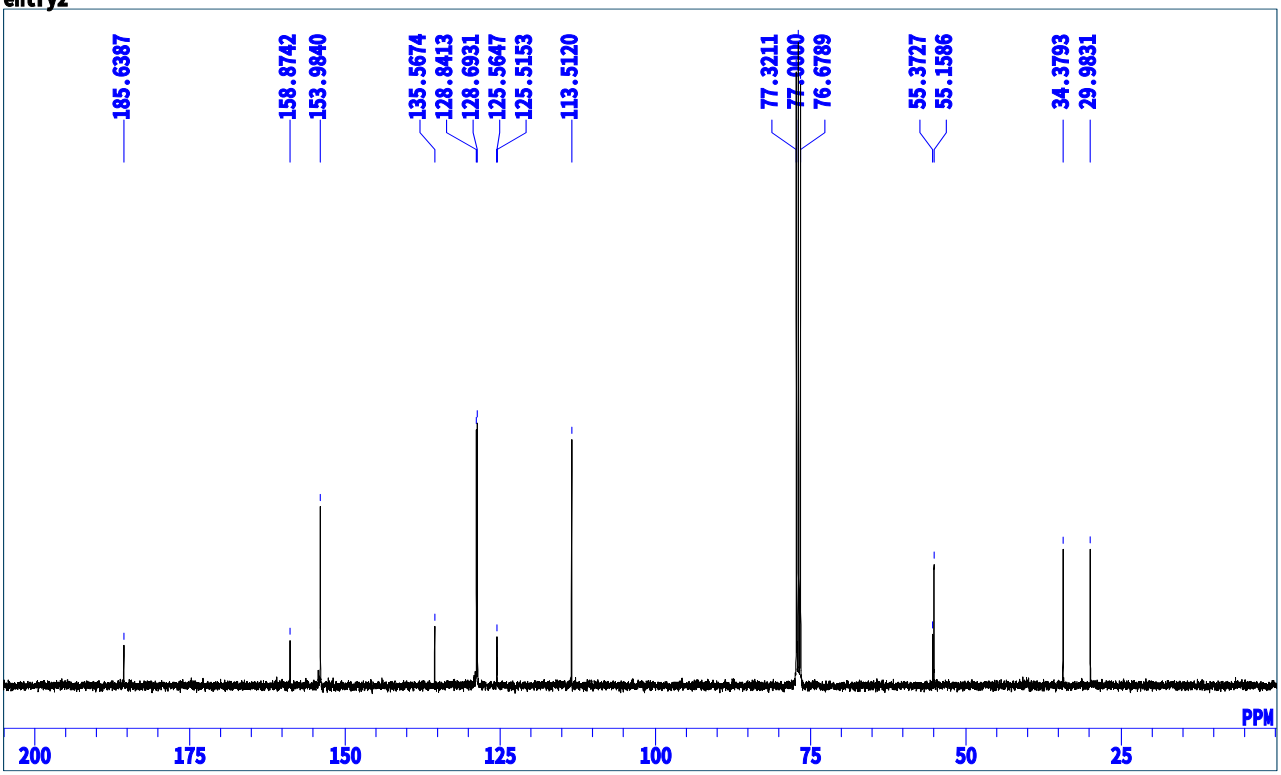

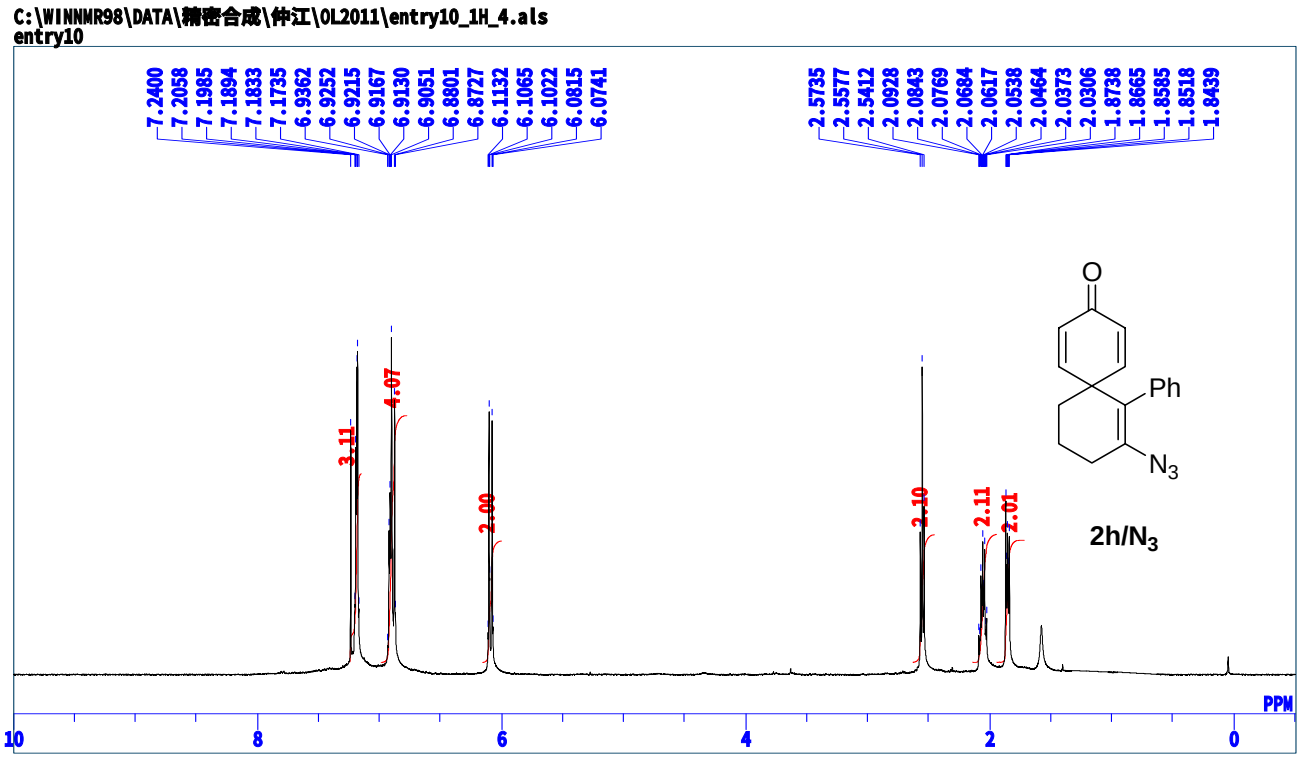
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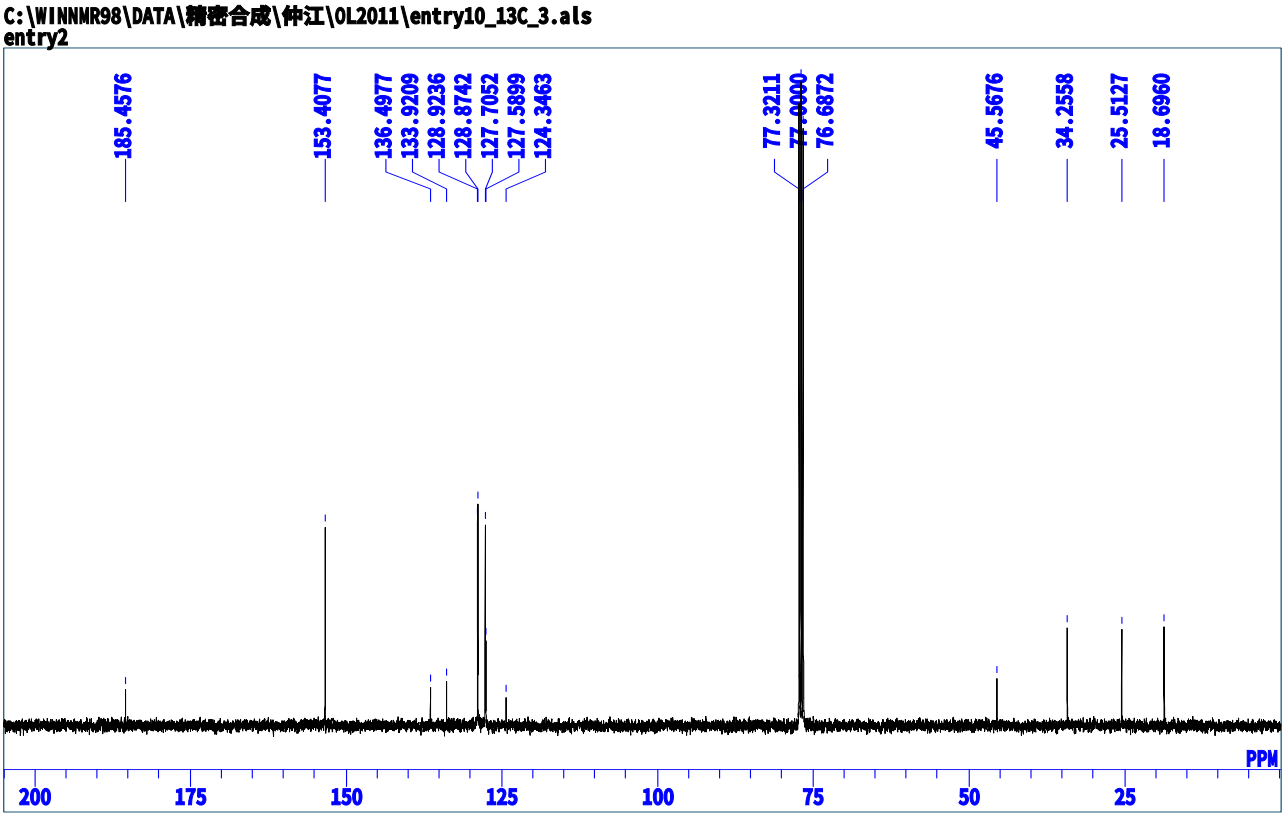
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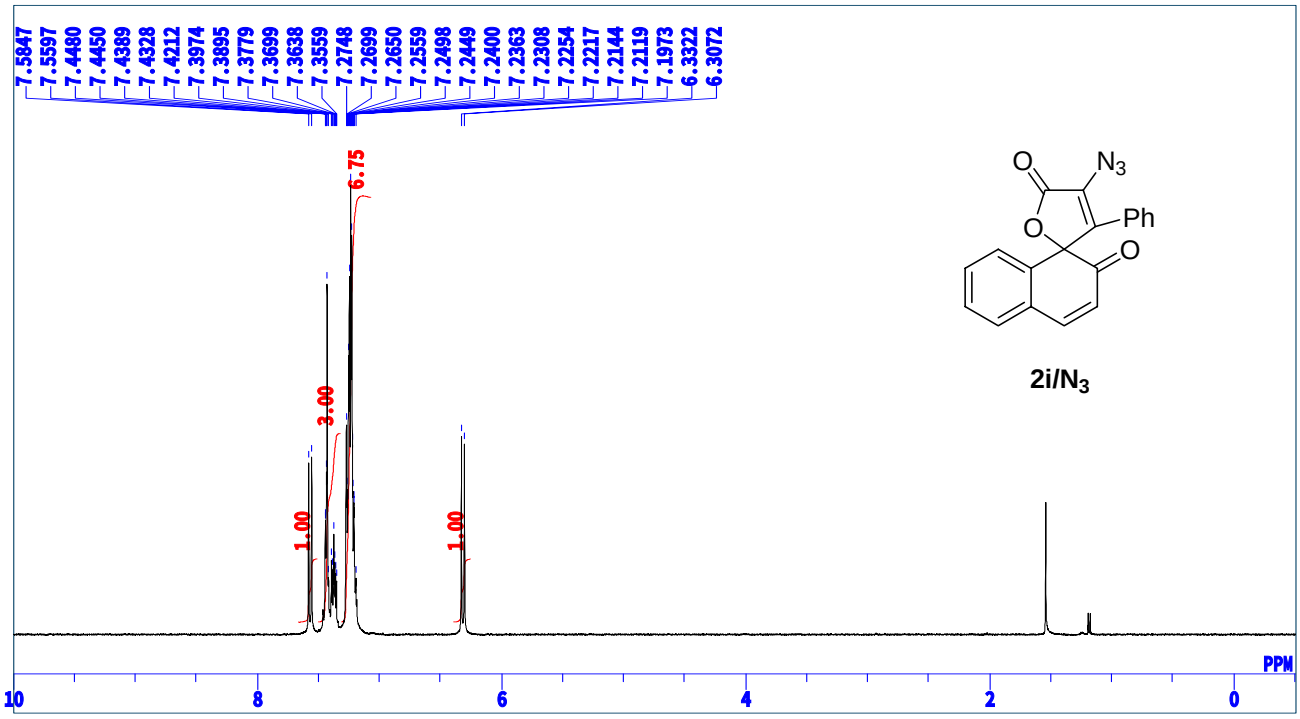

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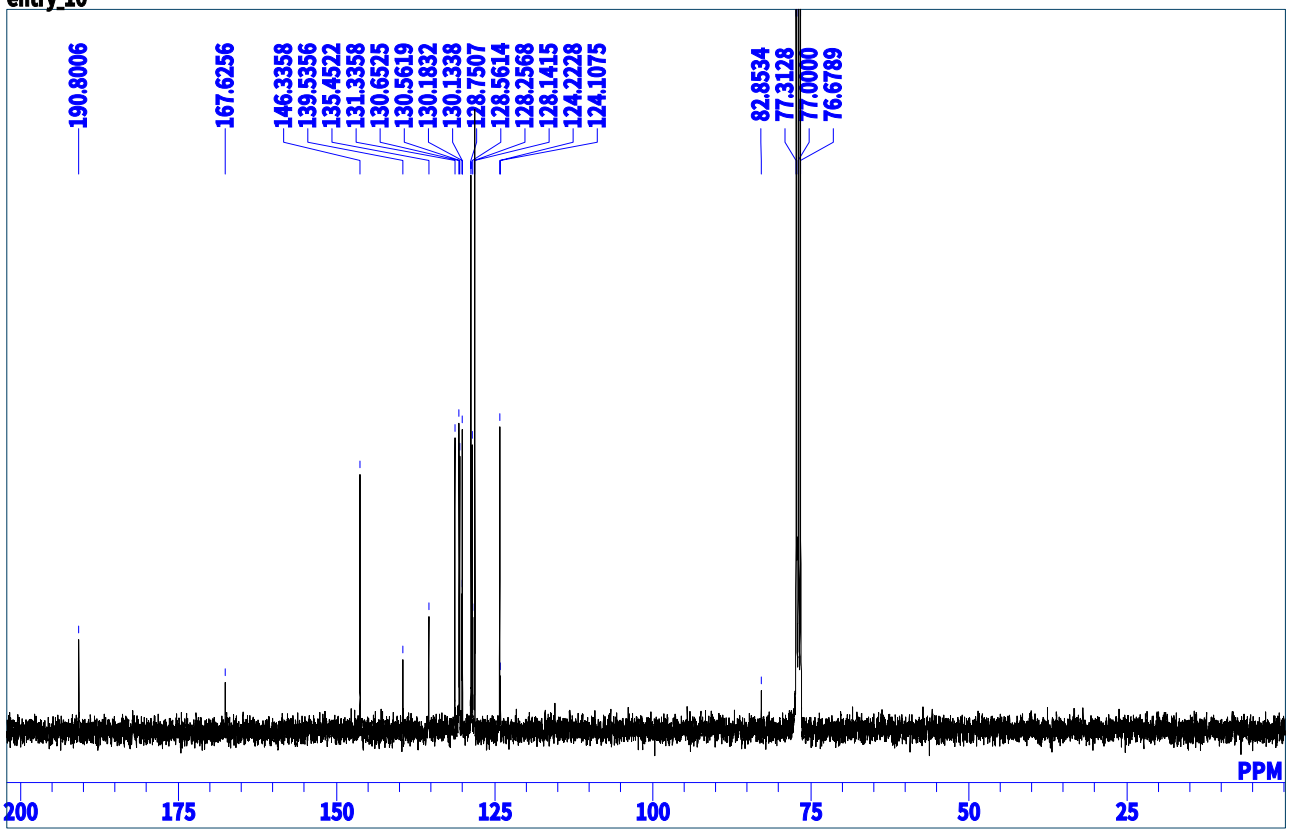

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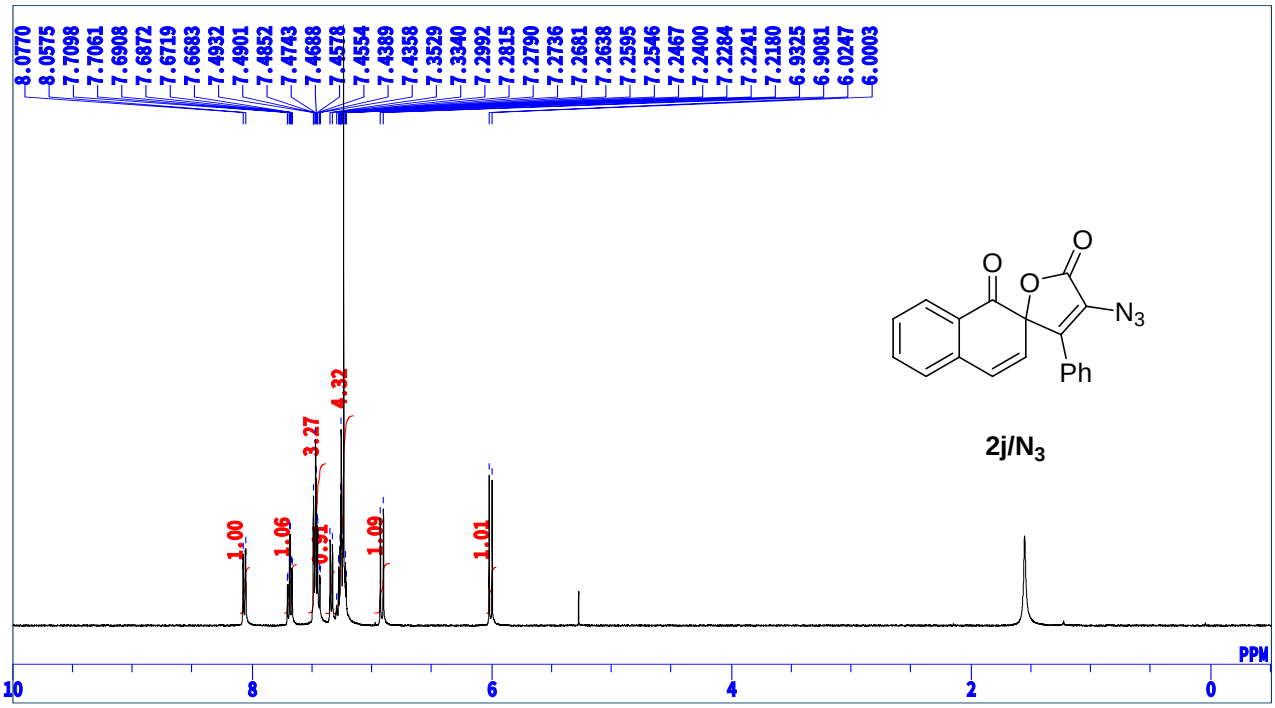

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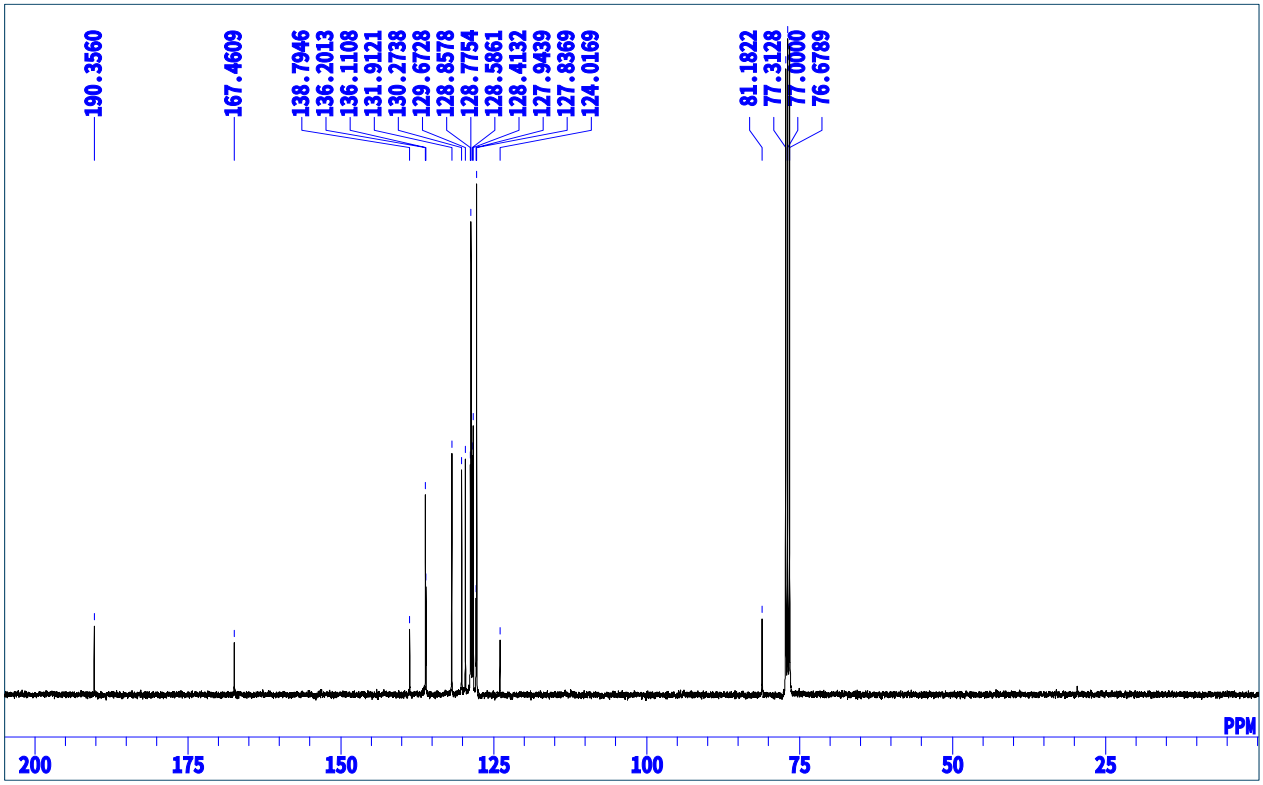

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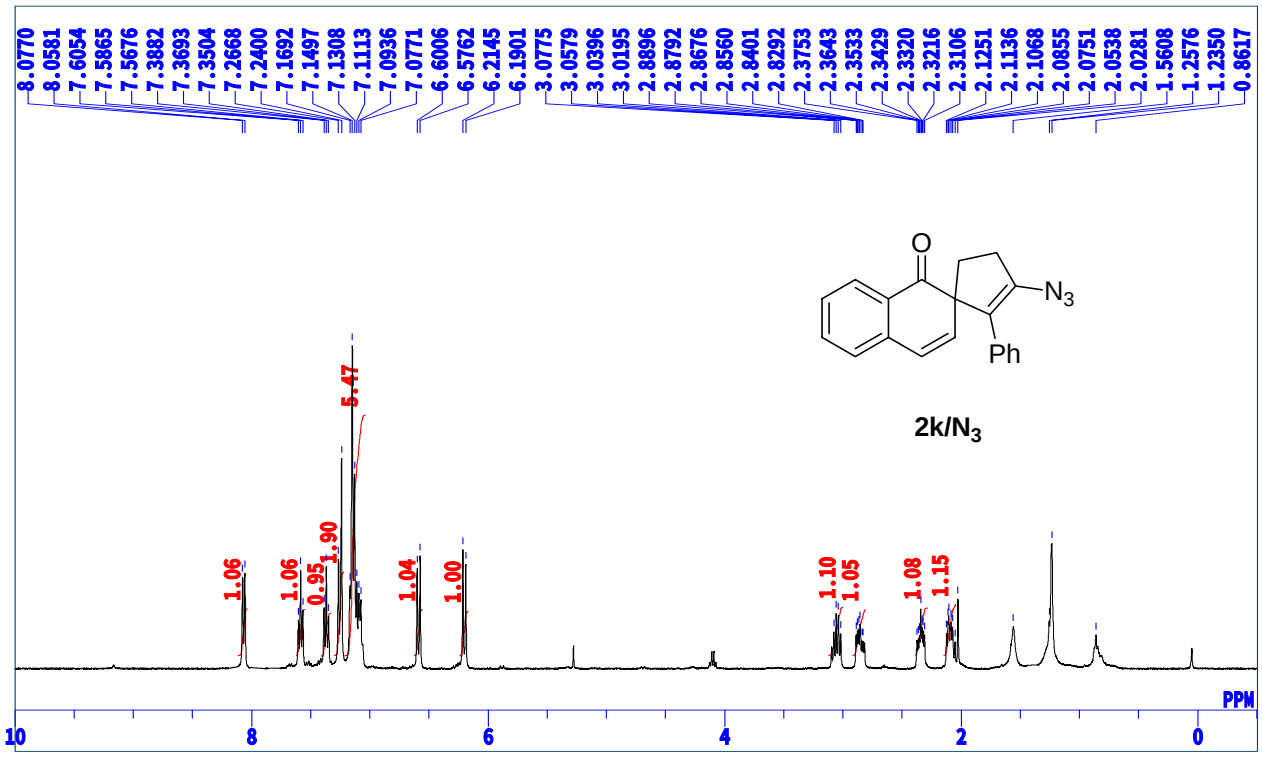

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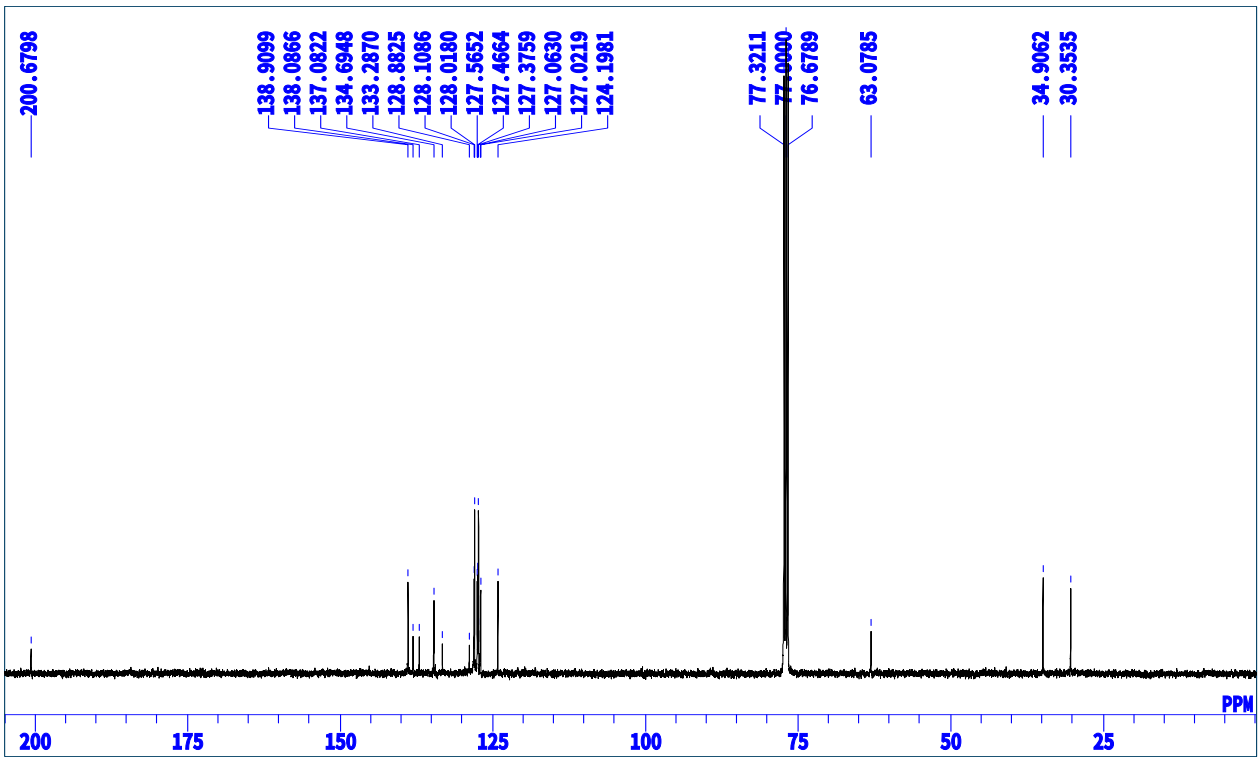

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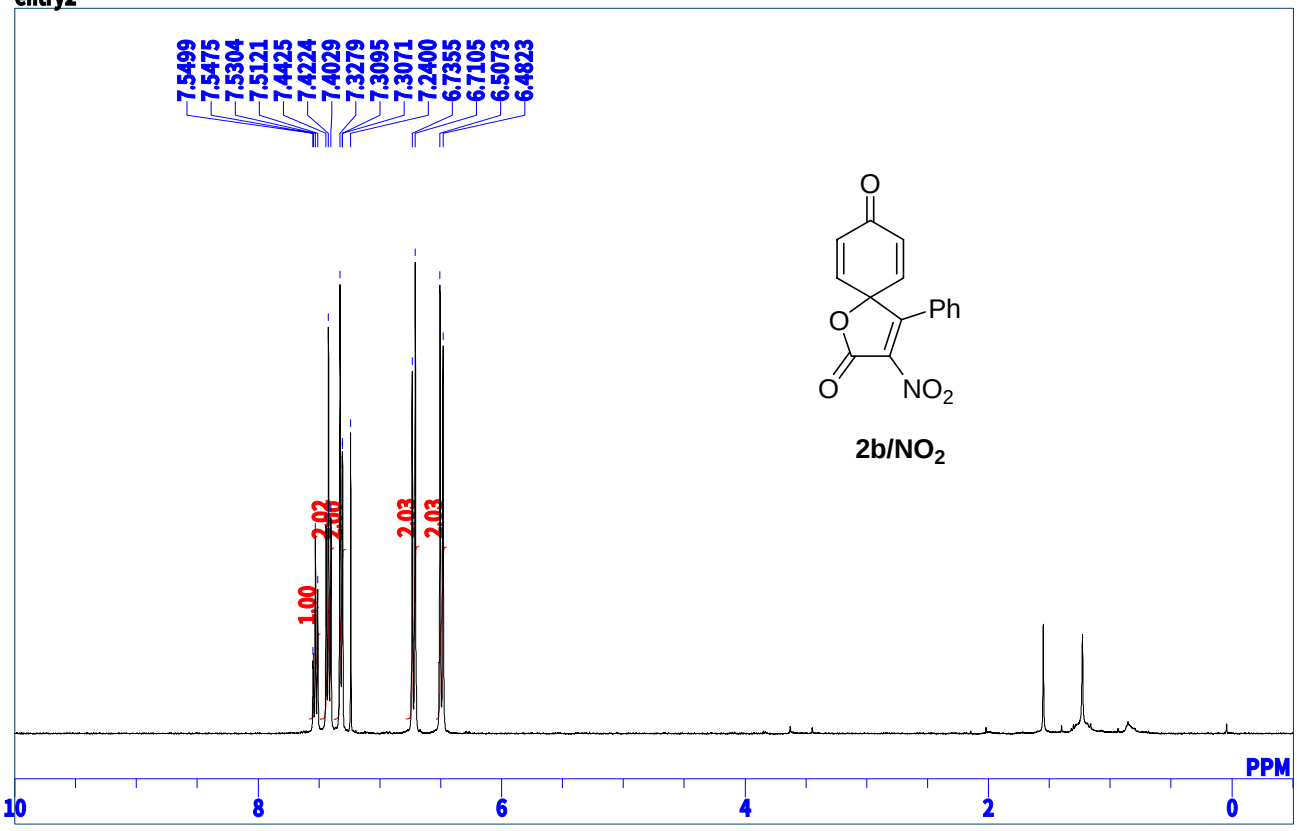

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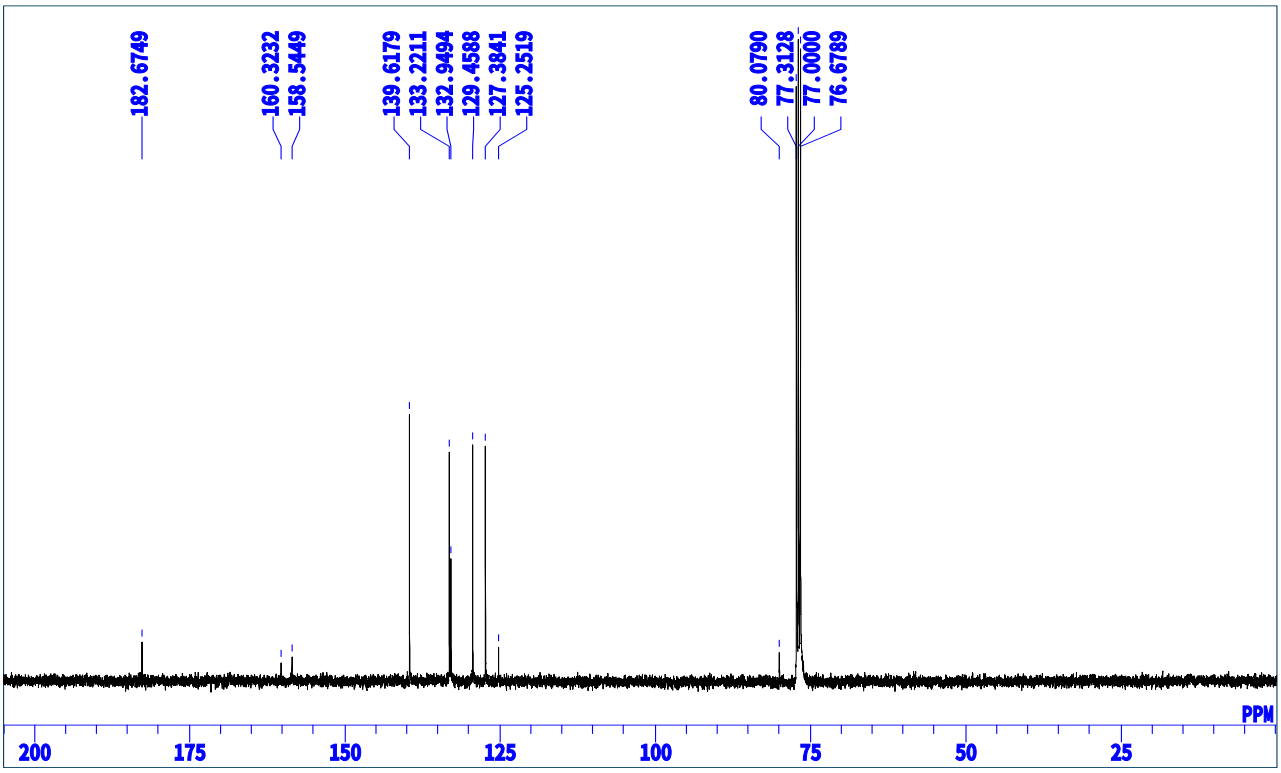

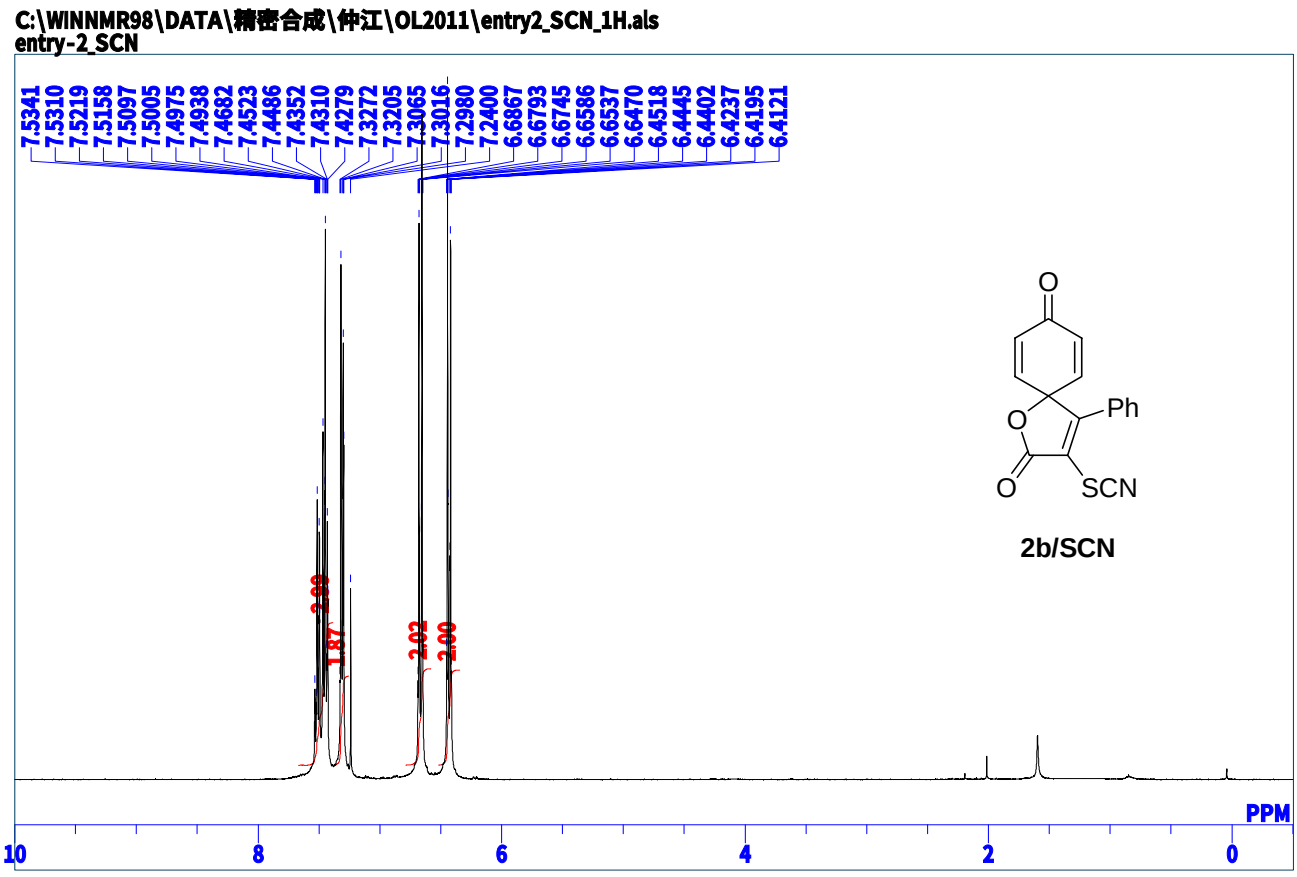
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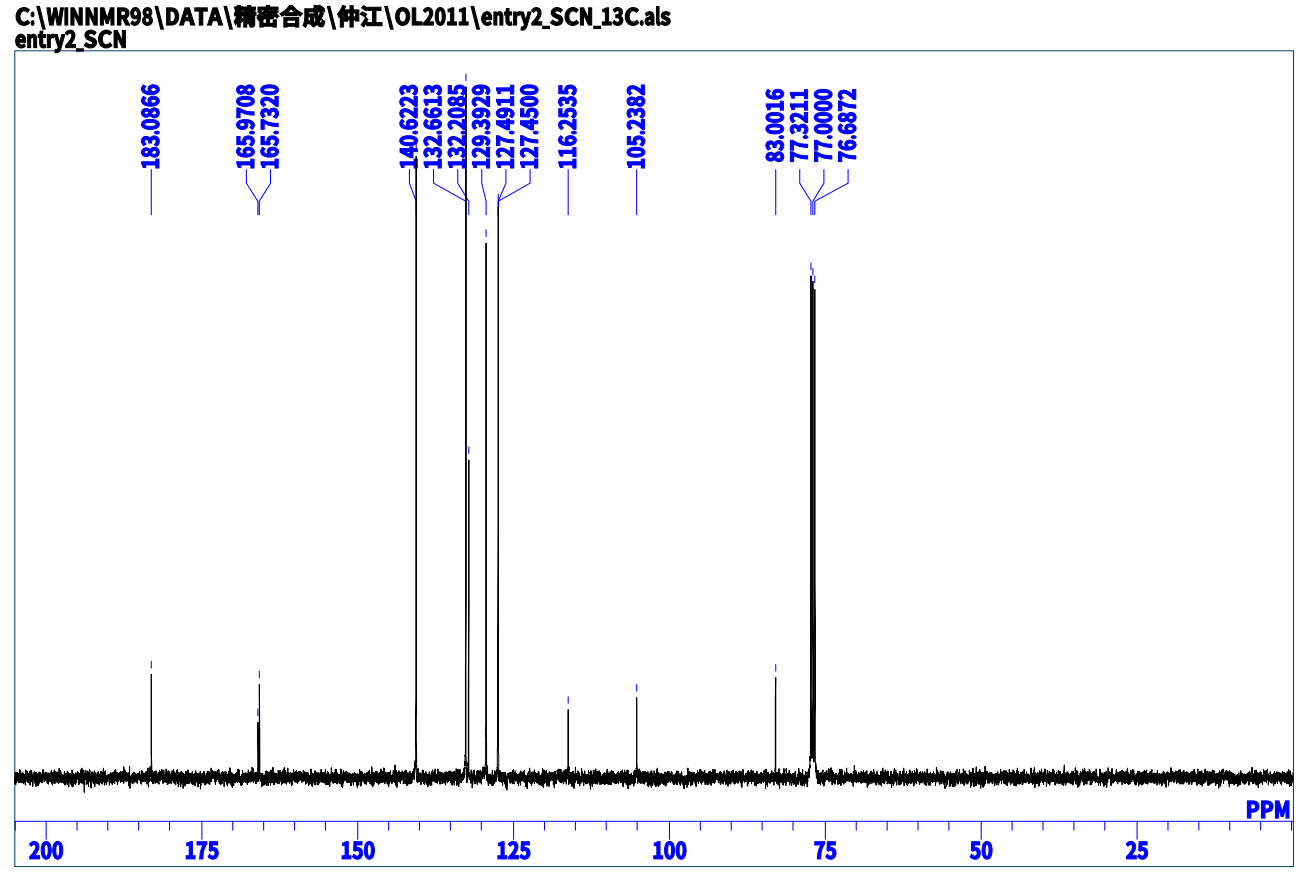
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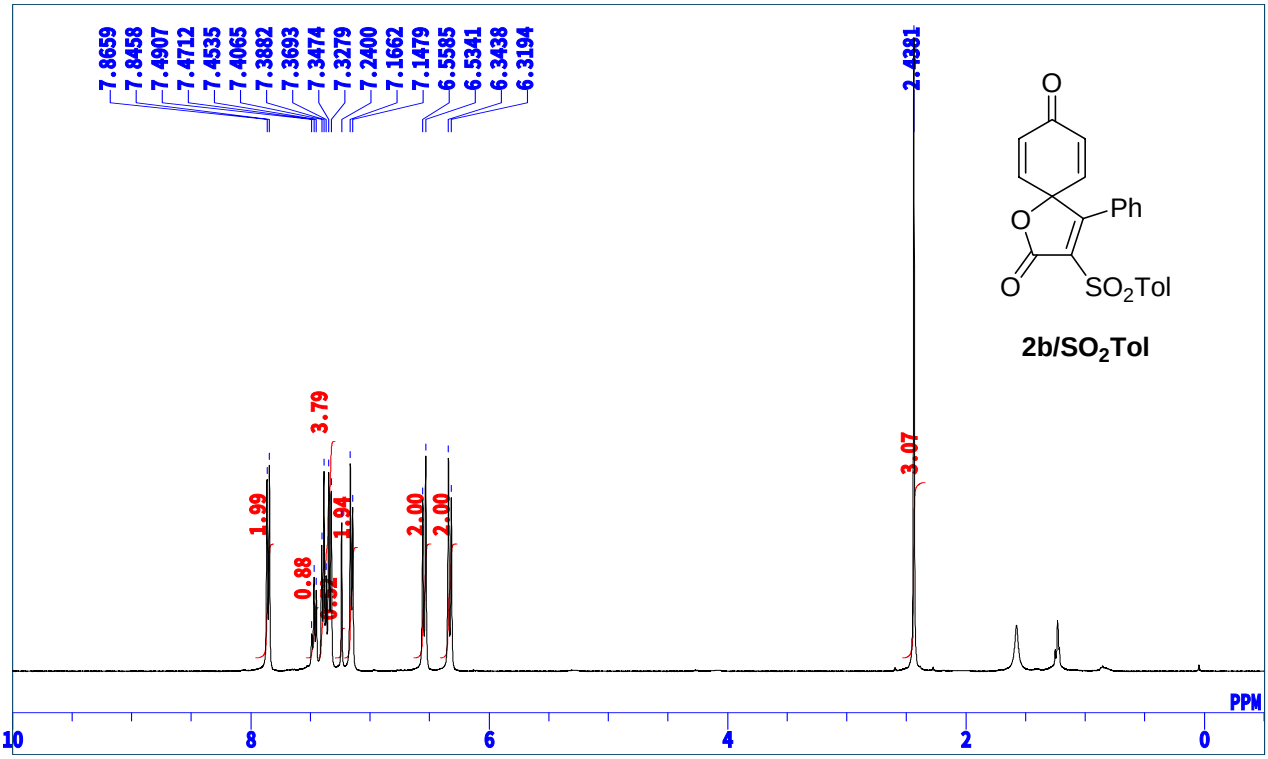

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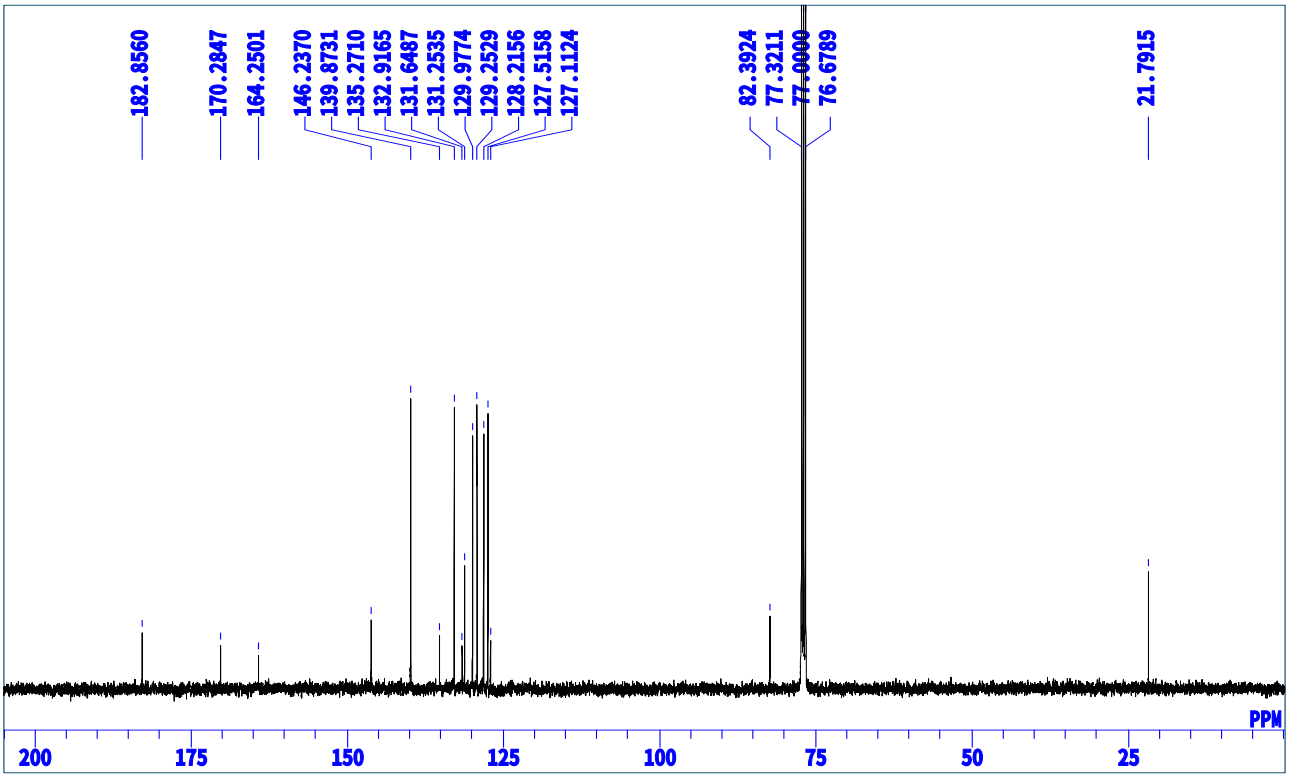

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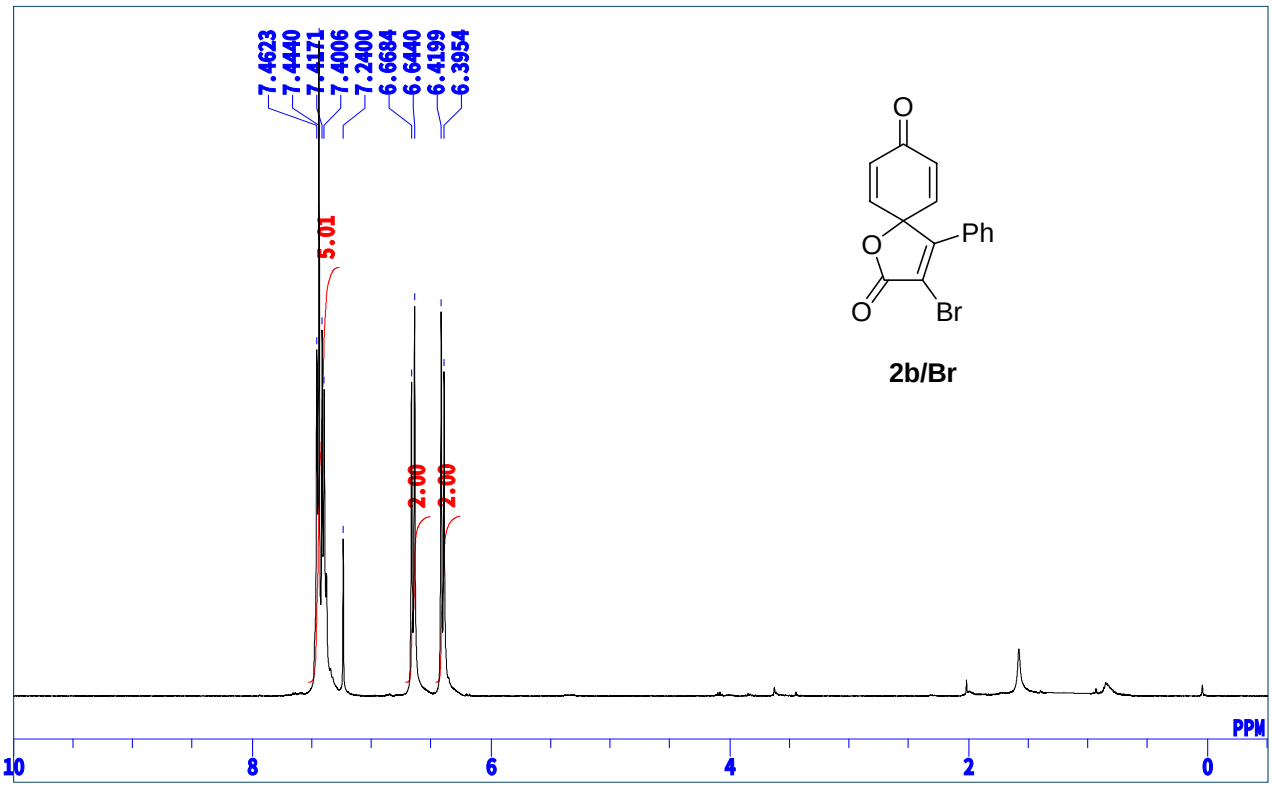

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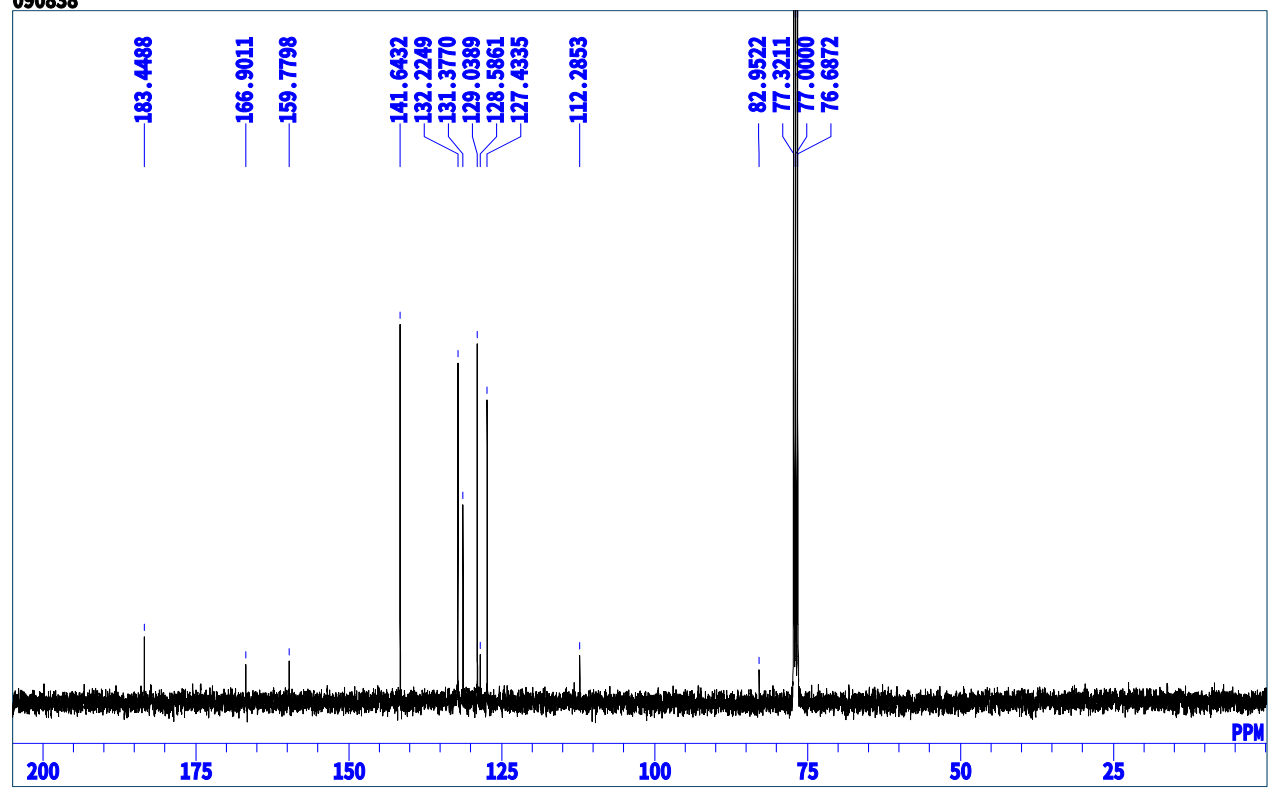

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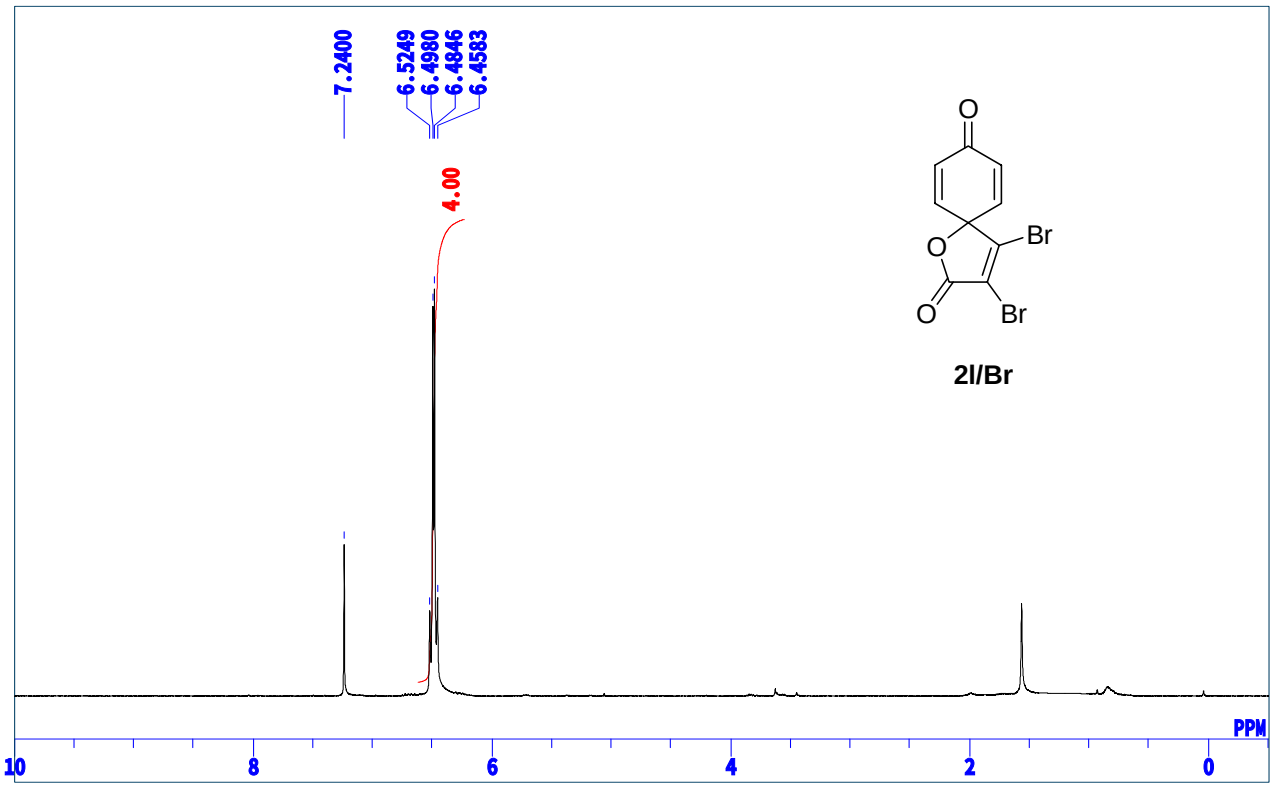

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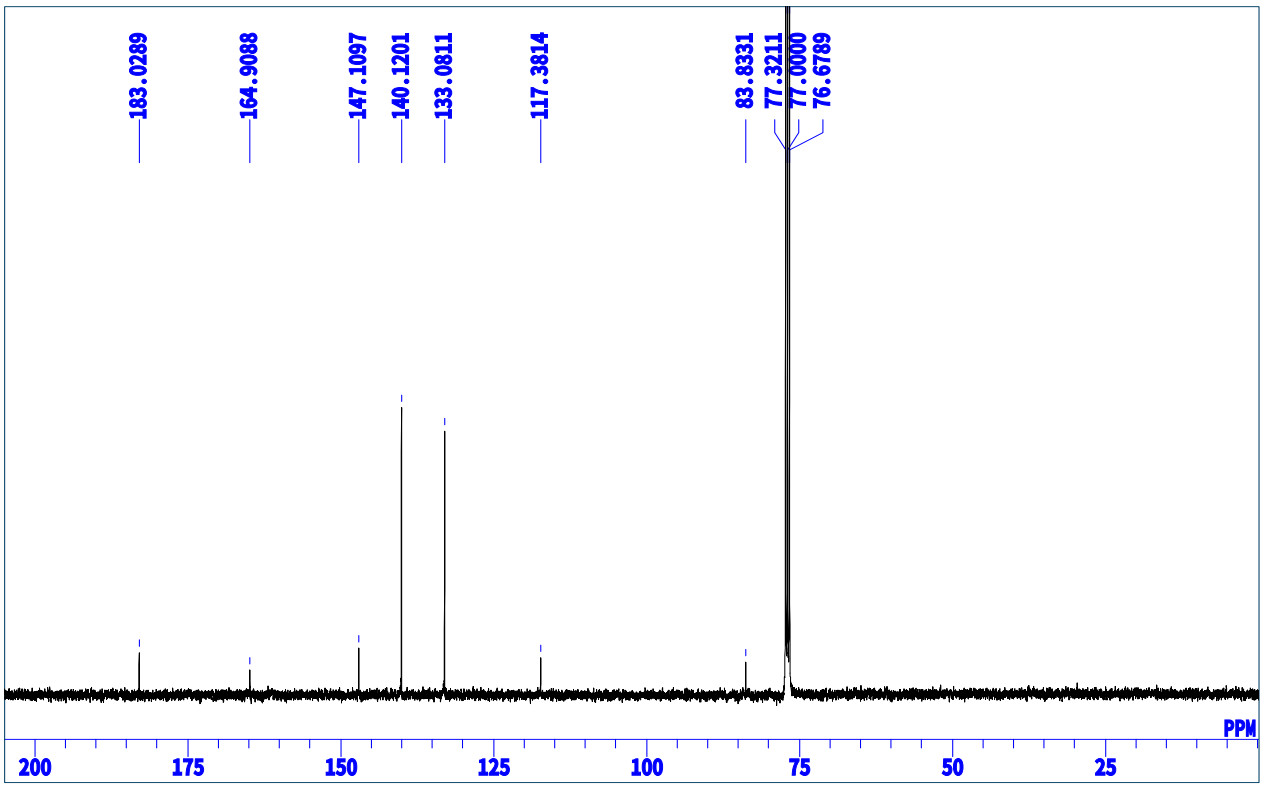

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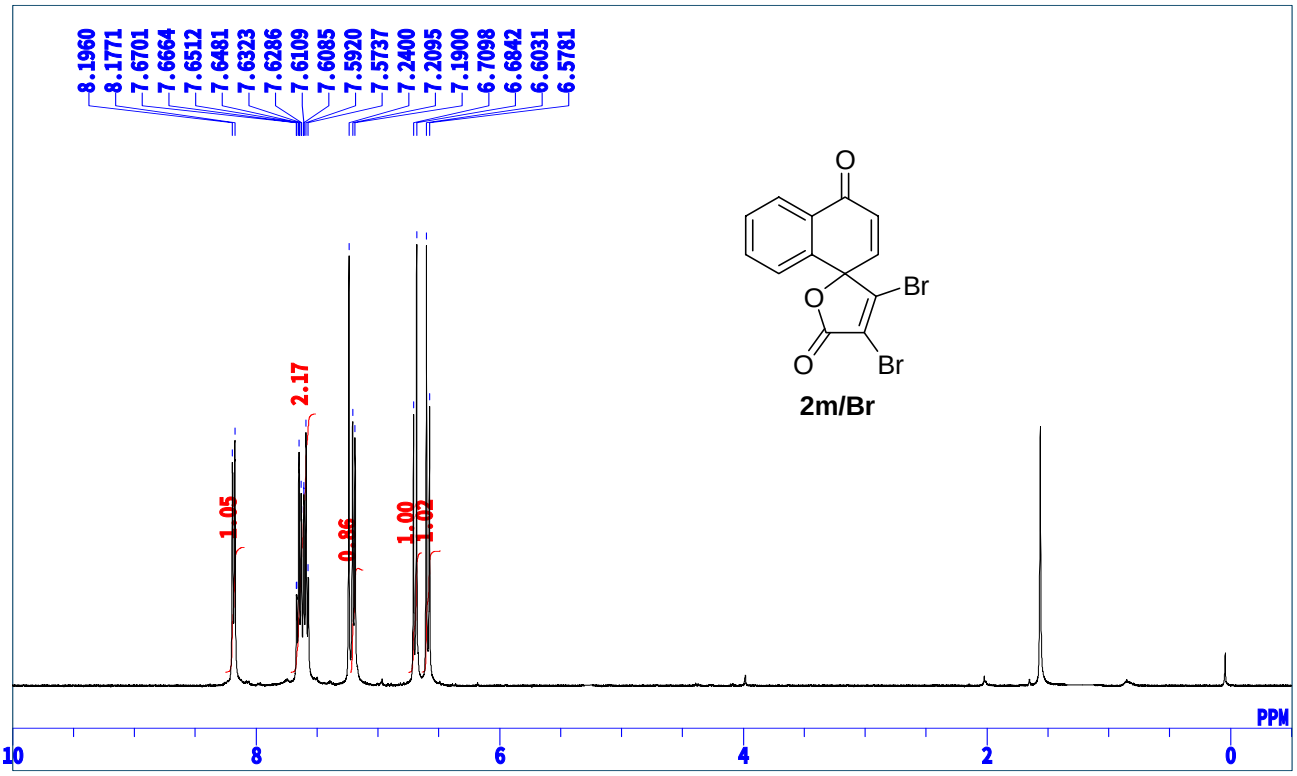

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