Supporting Information for

Gold-catalyzed Benzannulation of 3-Alkoxy-1,5-Enynes: Efficient Access to Functionalized Benzenes

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Contents:

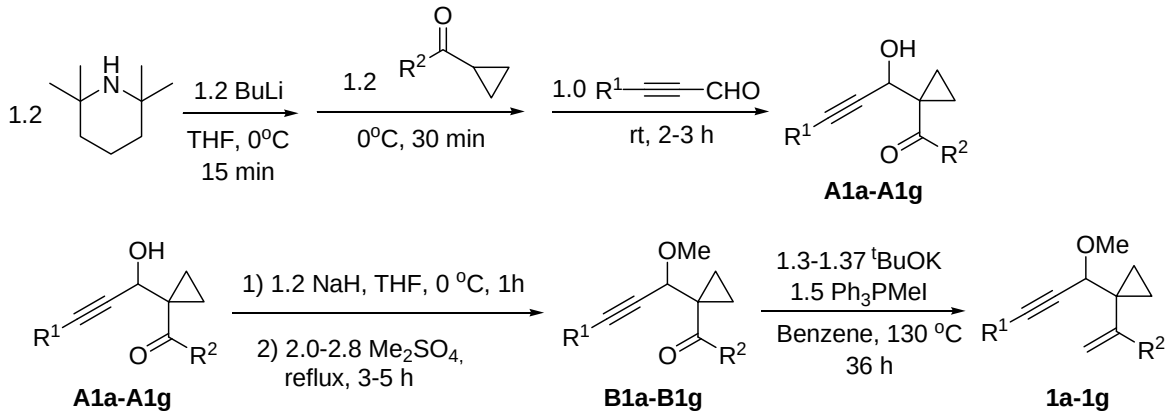
General Methods. All reactions were carried out under nitrogen. Dichloromethane and 1,2-dichloroethane were distilled from P_2O_5. THF, Et_2O and toluene were distilled from sodium and benzophenone. CH_3CN was distilled from CaH_2. CH_3NO_2 was dried over anhydrous Na_2SO_4. Unless noted, all commercial reagents were used as purchased without further purification. Ph_3PAuCl[1] and $Ph_3PAuNTf_2$[2] were prepared according to the published methods. $AgSbF_6$ was used as a 0.05 M solution in CH_3CN. $AgBF_4$ was used as a 0.05 M solution in toluene. Methanol and ethanol were dried before use by standard methods.[3] Prop-2-en-1-ol was dried by K_2CO_3 and distilled before use. Prop-2-yn-1-ol was distilled before use.

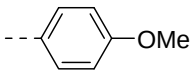
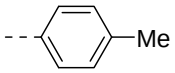
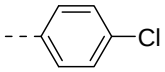
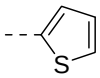
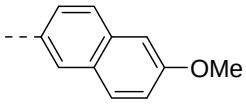
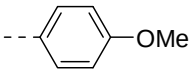
^1H NMR spectra was recorded at 300 or 400 MHz, ^{13}C NMR spectra was recorded at 75 or 100 MHz, and in $CDCl_3$ (containing 0.03% TMS) solutions. ^1H NMR spectra was recorded with tetramethylsilane (δ = 0.00 ppm) as internal reference; ^{13}C NMR spectra was

recorded with $CDCl_3$ (δ = 77.00 ppm) as internal reference.

General procedure for the synthesis of 1,5-enynes 1a-1g:

Table 1. Preparation of 3-Methoxy-1,5-Enynes.



entry	R^1	R^2	yields of **A**[a]	yields of **B**[a]	yields of **1**[a]
1	Ph	Ph	**A1a**, 91%	**B1a**, 72%	**1a**, 93%
2		Ph	**A1b**, 76%	**B1b**, 79%	**1b**, 56% (29%)[b]
3		Ph	**A1c**, 74%	**B1c**, 82%	**1c**, 92%
4		Ph	-	**B1d**, 61%[c]	**1d**, 77% (14%)[b]
5		Ph	**A1e**, 78%	**B1e**, 89%	**1e**, 79%
6		Ph	-	**B1f**, 66%[c]	**1f**, 63% (26%)[b]
7	Ph		**A1g**, 71%	**B1g**, 90%	**1g**, 90%

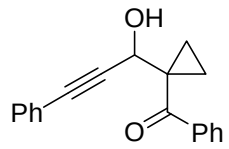
[a] Isolated yields; [b] Yields of the recovered starting material are shown in the parentheses. [c]Overall yields.

A typical procedure for the synthesis of **1c**: To a solution of 2,2,6,6-tetramethylpiperidine (16.25 mmol, 2.77 mL) in THF was added n-BuLi (16.25 mmol, 6.5 mL, 2.5 M in hexane) slowly at 0 °C. The reaction mixture was stirred for 15 min, then cyclopropyl phenyl ketone (16.25 mmol, 2.24 mL) was added at the same temperature and stirred for 30 min. 3-*p*-Tolylpropiolaldehyde (13.5 mmol, 1.95 g) was added and the mixture was warmed up to room temperature. The reaction mixture was

stirred for 3 hours and quenched with a saturated aqueous solution of NH_4Cl, extracted with EtOAc. The extract was washed with brine and dried over Na_2SO_4. The solvent was evaporated *in vacuo* and the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 5:1) to yield the desired alkynyl ketone **A1c** in 74% yield.

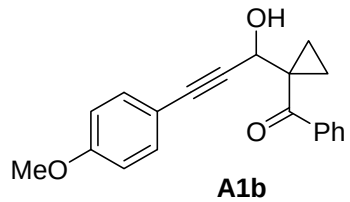
To a solution of **A1c** (4.0 mmol, 1.16 g) in THF was added NaH (4.8 mmol, 0.192 g, 60% dispersion in mineral oil) slowly at 0 ^oC. The reaction mixture was stirred for 1 hour at 0 ^oC and then Me_2SO_4 (11.2 mmol, 1.06 mL) was added slowly. The reaction mixture was warmed up to room temperature, and then refluxed for 4 hours until the starting material was consumed as monitored by TLC analysis. The reaction mixture was cooled to room temperature and quenched with a saturated aqueous solution of NH_4Cl, extracted with EtOAc. The extract was washed with brine and dried over Na_2SO_4. The solvent was evaporated *in vacuo* and the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 20:1) to yield the desired product **B1c** in 82% yield.

Methylenation:[4] To a suspension of Ph_3PMeI (2.12 mmol, 0.856 g) in benzene (2.5 mL) was added ^tBuOK (1.9 mmol, 0.216 g) at room temperature under the nitrogen atmosphere. The mixture was heated to130 ^oC and stirred for 1 h, then most of the benzene was vaporized via a needle until the reaction mixture became slurry (It was recommended to use distillation to remove the benzene when the reaction was carried out on a larger scale). The mixture was cooled to room temperature and a solution of **B1c** (1.48 mmol, 0.45 g) in benzene (1.5 mL) was added. The mixture was heated to 130 ^oC again and stirred for 15 min, then most of the benzene was vaporized via a needle until the reaction mixture became slurry. The Schlenk tube was sealed and the reaction mixture was continued to stir for about 36 hours. The mixture was cooled to room temperature and quenched with H_2O, extracted with Et_2O. The extract was dried over Na_2SO_4. The solvent was evaporated *in vacuo* and the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 40:1) to afford the desired **1c** in 92% yield.



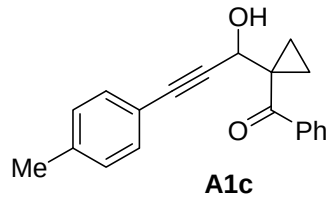
A1a

Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1-5:1) afforded the title compound as a brown solid in 91% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.11-1.16 (m, 1H), 1.26-1.35 (m, 1H), 1.37-1.50 (m, 2H), 2.85 (d, *J* = 7.2 Hz, 1H), 5.32 (d, *J* = 7.5 Hz, 1H), 7.25-7.54 (m, 8H), 7.76-7.78 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75Mz): δ 8.4, 13.1, 35.3, 65.4, 86.0, 86.1, 121.9, 128.0 (2C), 128.2 (2C), 128.3 (2C), 128.6, 131.7 (2C), 131.9, 136.9, 202.6; IR (KBr): 3447, 3060, 2222, 1671, 1598, 1578, 1490, 1446, 1346, 1256, 1196, 1177, 1038, 1002, 980, 917, 757, 691 cm^{-1}; HRMS (EI) for $C_{19}H_{16}O_2$: calcd 276.1150, found 276.1148.



A1b

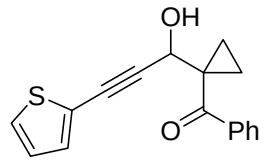
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1-5:1) afforded the title compound as a brown sticky liquid in 76% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300Mz): δ 1.08-1.14 (m, 1H), 1.28-1.34 (m, 1H), 1.36-1.48 (m, 2H), 2.93 (d, *J* = 6.9 Hz, 1H), 3.78 (s, 3H), 5.32 (d, *J* = 6.6 Hz, 1H), 6.79-6.84 (m, 2H), 7.26-7.34 (m, 2H), 7.40-7.46 (m, 2H), 7.49-7.54 (m, 1H), 7.76-7.79 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75Mz): δ 8.3, 13.2, 35.3, 55.2, 65.5, 84.5, 86.1, 113.8 (2C), 113.9, 128.0 (2C), 128.3 (2C), 131.9, 133.2 (2C), 136.8, 159.7, 202.6; IR (KBr): 3459, 3062, 3004, 2935, 2838, 2222, 1673, 1606, 1510, 1447, 1346, 1291, 1249, 1174, 1107, 1035, 980, 833, 702 cm^{-1}; HRMS (EI) for $C_{20}H_{18}O_3$: calcd 306.1256, found 306.1258.



A1c

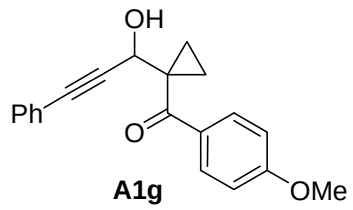
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum

ether/ethyl acetate = 20:1-5:1) afforded the title compound as a yellow solid in 74% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.09-1.14 (m, 1H), 1.28-1.48 (m, 3H), 2.33 (s, 3H), 2.84 (d, *J* = 7.2 Hz, 1H), 5.32 (d, *J* = 7.2 Hz, 1H), 7.10 (d, *J* = 7.8 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 7.41-7.54 (m, 3H), 7.77 (d, *J* = 6.9 Hz, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 8.3, 13.2, 21.4, 35.3, 65.5, 85.1, 86.3, 118.8, 128.0 (2C), 128.4 (2C), 129.0 (2C), 131.6 (2C), 131.9, 136.9, 138.8, 202.6; IR (KBr): 3447, 3058, 3027, 2921, 2224, 1669, 1597, 1578, 1509, 1448, 1417, 1346, 1257, 1198, 1177, 1038, 1003, 981, 933, 919, 817, 706, 640 cm^{-1}; HRMS (EI) for $C_{20}H_{18}O_2$: calcd 290.1307, found 290.1312.



A1e

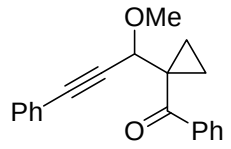
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) afforded the title compound as a brown yellow sticky liquid in 78% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.09-1.16 (m, 1H), 1.25-1.31 (m, 1H), 1.33-1.40 (m, 1H), 1.42-1.48 (m, 1H), 3.04 (d, *J* = 7.2 Hz, 1H), 5.30 (d, *J* = 7.2 Hz, 1H), 6.93-6.97 (m, 1H), 71.6-7.18 (m, 1H), 7.24-7.26 (m, 1H), 7.40-7.45 (m, 2H), 7.49-7.54 (m, 1H), 7.74-7.78 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75Mz): δ 8.6, 13.1, 35.2, 65.6, 79.4, 89.9, 121.7, 126.9, 127.6, 128.0 (2C), 128.4 (2C), 132.0, 132.6, 136.8, 202.5; IR (KBr): 3441, 3103, 3023, 2896, 2220, 1668, 1597, 1578, 1517, 1447, 1422, 1343, 1257, 1190, 1036, 970, 850, 702 cm^{-1}; HRMS (EI) for $C_{17}H_{14}O_2S$: calcd 282.0715, found 282.0718.



A1g

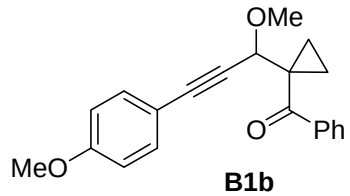
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) afforded the title compound as a brown sticky liquid in 71% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.03-1.11 (m, 1H), 1.24-1.29 (m, 1H), 1.33-1.41 (m,

2H), 2.91 (dd, *J* = 7.8, 1.2 Hz, 1H), 3.85 (s, 3H), 5.29 (d, *J* = 7.2 Hz, 1H), 6.90-6.95 (m, 2H), 7.26-7.33 (m, 3H), 7.37-7.40 (m, 2H), 7.87-7.92 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.8, 12.4, 34.8, 55.4, 66.0, 86.0, 86.2, 113.6 (2C), 122.0, 128.2 (2C), 128.6, 129.1, 130.9 (2C), 131.7 (2C), 163.0, 199.8; IR (KBr): 3446, 3062, 3007, 2935, 2840, 2222, 1666, 1599, 1574, 1509, 1490, 1443, 1419, 1345, 1309, 1255, 1167, 1032, 980, 917, 842, 758, 692 cm^{-1}; HRMS (EI) for $C_{20}H_{18}O_3$: calcd 306.1256, found 306.1247.



B1a

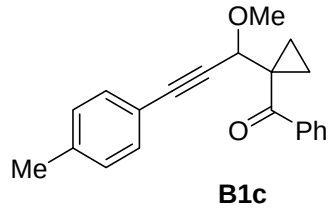
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) afforded the title compound as a light yellow sticky liquid in 72% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.02-1.07 (m, 1H), 1.31-1.42 (m, 2H), 1.52-1.57 (m, 1H), 3.27 (s, 3H), 5.13 (s, 1H), 7.28-7.33 (m, 3H), 7.39-7.55 (m, 5H), 7.81-7.83 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 8.0, 12.8, 33.2, 56.6, 73.8, 83.5, 87.6, 122.0, 128.0 (2C), 128.3 (4C), 128.7, 131.7 (2C), 131.8, 137.3, 201.4; IR (KBr): 3061, 2996, 2934, 2900, 2822, 2215, 1680, 1598, 1578, 1490, 1446, 1348, 1257, 1190, 1177, 1088, 1031, 1004, 995, 969, 920, 780, 757, 722, 692 cm^{-1}; HRMS (EI) for $C_{20}H_{18}O_2$: calcd 290.1307, found 290.1303.



B1b

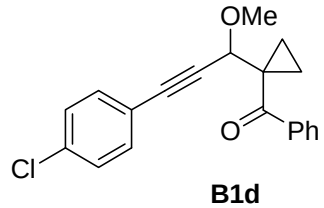
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1) afforded the title compound as a light yellow sticky liquid in 79% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.01-1.06 (m, 1H), 1.30-1.41 (m, 2H), 1.51-1.56 (m, 1H), 3.26 (s, 3H), 3.80 (s, 3H), 5.11 (s, 1H), 6.82-6.86 (m, 2H), 7.33-7.38 (m, 2H), 7.42-7.56 (m, 3H), 7.80-7.84 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.9, 12.8, 33.3, 55.2, 56.5, 73.9, 82.0, 87.5, 113.9 (2C), 114.0, 128.0 (2C), 128.3 (2C), 131.8, 133.2

(2C), 137.3, 159.8, 201.4; IR (KBr): 3059, 3000, 2935, 2838, 2219, 1679, 1606, 1570, 1509, 1446, 1418, 1348, 1291, 1248, 1174, 1088, 1032, 1002, 968, 923, 833, 804, 779, 714 cm^{-1}; HRMS (EI) for $C_{21}H_{20}O_3$: calcd 320.1412, found 320.1406.



B1c

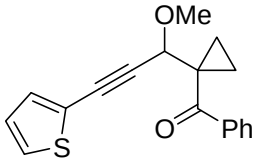
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20:1) afforded the title compound as a light yellow sticky liquid in 82% yield. ^1H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.00-1.06 (m, 1H), 1.33-1.41 (m, 2H), 1.51-1.57 (m, 1H), 2.34 (s, 3H), 3.26 (s, 3H), 5.12 (s, 1H), 7.12 (d, *J* = 7.8 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.42-7.55 (m, 3H), 7.80-7.83 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.9, 12.8, 21.4, 33.2, 56.5, 73.8, 82.7, 87.7, 118.9, 128.0 (2C), 128.3 (2C), 129.0 (2C), 131.6 (2C), 131.8, 137.3, 138.9, 201.4; IR (KBr): 3077, 3056, 3032, 2993, 2942, 2923, 2880, 2819, 2220, 1680, 1597, 1578, 1509, 1448, 1418, 1353, 1308, 1257, 1188, 1086, 1002, 969, 816, 721, 642 cm^{-1}; HRMS (EI) for $C_{21}H_{20}O_2$: calcd 304.1463, found 304.1465.



B1d

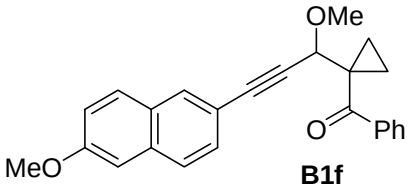
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 40:1) afforded the title compound as a colorless sticky liquid in 61% yield (overall yield for two steps). ^1H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.04-1.09 (m, 1H), 1.28-1.39 (m, 2H), 1.51-1.56 (m, 1H), 3.27 (s, 3H), 5.10 (s, 1H), 7.26-7.36 (m, 4H), 7.43-7.56 (m, 3H), 7.80-7.83 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 8.1, 12.6, 33.2, 56.6, 73.7, 84.6, 86.4, 120.4, 128.0 (2C), 128.3 (2C), 128.6 (2C), 131.9, 133.0 (2C), 134.7, 137.2, 201.2; IR (KBr): 3064, 2994, 2933, 2900, 2822, 2223, 1680, 1597, 1578, 1489, 1447, 1420, 1397, 1347, 1256, 1191, 1089, 1015, 1003, 968, 922, 829, 780, 765, 699,

655 cm^{-1}; HRMS (EI) for $C_{20}H_{17}O_2Cl$: calcd 324.0917, found 324.0915.



B1e

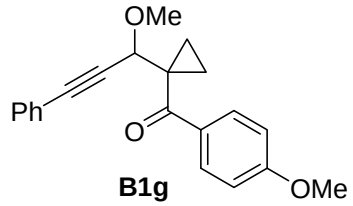
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 15:1) afforded the title compound as a light brown sticky liquid in 89% yield (overall yield for two steps). ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.03-1.08 (m, 1H), 1.28-1.39 (m, 2H), 1.52-1.57 (m, 1H), 3.26 (s, 3H), 5.12 (s, 1H), 6.97 (t, *J* = 3.9 Hz, 1H), 7.21 (d, *J* = 3.3 Hz, 1H), 7.27 (d, *J* = 5.4 Hz, 1H), 7.43-7.56 (m, 3H), 7.81 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 8.1, 12.8, 33.2, 56.7, 74.0, 80.8, 87.5, 121.7, 127.0, 127.6, 128.0 (2C), 128.3 (2C), 131.8, 132.6, 137.2, 201.2; IR (KBr): 3103, 3083, 2994, 2933, 2900, 2822, 2218, 1679, 1598, 1578, 1447, 1422, 1344, 1322, 1303, 1259, 1190, 1088, 1035, 1007, 991, 970, 851, 779, 703, 645 cm^{-1}; HRMS (EI) for $C_{18}H_{16}O_2S$: calcd 296.0871, found 296.0870.



B1f

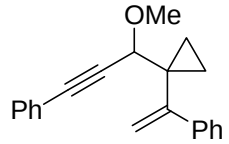
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20:1) afforded the title compound as a brown red sticky liquid in 66% yield (overall yield for two steps). ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.04-1.09 (m, 1H), 1.34-1.47 (m, 2H), 1.55-1.60 (m, 1H), 3.30 (s, 3H), 3.89 (s, 3H), 5.17 (s, 1H), 7.08 (d, *J* = 1.5 Hz, 1H), 7.15 (dd, *J* = 9.0, 2.4 Hz, 1H), 7.40-7.55 (m, 4H), 7.65 (d, *J* = 5.2 Hz, 1H), 7.67 (d, *J* = 5.8 Hz, 1H), 7.82-7.86 (m, 3H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 8.0, 12.8, 33.3, 55.2, 56.6, 73.9, 82.8, 88.1, 105.6, 116.7, 119.5, 126.8, 128.0 (2C), 128.2, 128.3 (2C), 128.8, 129.2, 131.6, 131.8, 134.2, 137.3, 158.4, 201.4; IR (KBr): 3059, 2999, 2935, 2822, 2221, 1678, 1629, 1601, 1499, 1483, 1447, 1390, 1347, 1246, 1198, 1163, 1122, 1087, 1029, 1004, 968, 892, 853, 806, 779, 700, 644 cm^{-1}; HRMS (EI) for $C_{25}H_{22}O_3$: calcd

370.1569, found 370.1568.



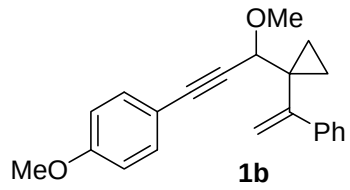
B1g

Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20:1-10:1) afforded the title compound as a brown yellow sticky liquid in 90% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.95-1.00 (m, 1H), 1.27-1.38 (m, 2H), 1.43-1.48 (m, 1H), 3.32 (s, 3H), 3.86 (s, 3H), 5.12 (s, 1H), 6.92-6.97 (m, 2H), 7.26-7.35 (m, 3H), 7.41-7.44 (m, 2H), 7.90-7.94 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.4, 11.7, 32.7, 55.3, 56.5, 74.3, 83.5, 87.6, 113.5 (2C), 122.0, 128.3 (2C), 128.6, 129.3, 130.8 (2C), 131.7 (2C), 162.8, 198.8; IR (KBr): 3062, 2999, 2935, 2839, 2822, 2222, 1671, 1601, 1575, 1509, 1490, 1443, 1418, 1347, 1309, 1254, 1167, 1088, 1030, 995, 971, 842, 758, 691, 608, 589 cm^{-1}; HRMS (EI) for $C_{21}H_{20}O_3$: calcd 320.1412, found 320.1414.



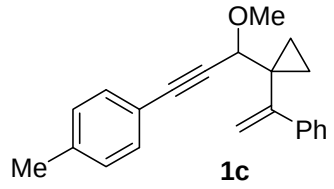
1a

Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 50:1) afforded the title compound as a light yellow sticky liquid in 93% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.76-0.81 (m, 1H), 0.99-1.05 (m, 1H), 1.18-1.30 (m, 2H), 3.32 (s, 3H), 4.45 (s, 1H), 5.29 (d, J = 0.9 Hz, 1H), 5.43 (d, J = 0.9 Hz, 1H), 7.25-7.42 (m, 8H), 7.57-7.61 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.7, 12.3, 28.2, 56.8, 74.7, 85.3, 86.6, 115.1, 122.5, 127.0 (2C), 127.5, 128.2 (2C), 128.2 (2C), 128.4, 131.7 (2C), 140.1, 148.8; IR (KBr): 3082, 3055, 3020, 2995, 2962, 2929, 2901, 2851, 2820, 2214, 1624, 1599, 1573, 1490, 1443, 1325, 1261, 1188, 1088, 1028, 905, 798, 777, 756, 704, 691 cm^{-1}; HRMS (EI) for $C_{21}H_{20}O$: calcd 288.1514, found 288.1517.



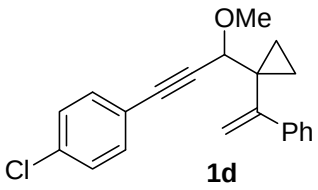
1b

Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 50:1) afforded the title compound as a brown sticky liquid in 56% yield (the starting material of **B1b** was recovered in 29% yield). ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.74-0.80 (m, 1H), 0.99-1.05 (m, 1H), 1.17-1.29 (m, 2H), 3.31 (s, 3H), 3.80 (s, 3H), 4.44 (s, 1H), 5.28 (d, *J* = 1.2 Hz, 1H), 5.42 (d, *J* = 1.2 Hz, 1H), 6.80-6.85 (m, 2H), 7.25-7.38 (m, 5H), 7.57-7.61 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.6, 12.4, 28.2, 55.3, 56.7, 74.8, 83.8, 86.5, 113.8 (2C), 114.6, 115.1, 127.1 (2C), 127.5, 128.2 (2C), 133.2 (2C), 140.2, 148.8, 159.6; IR (KBr): 3083, 2931, 2838, 2222, 1606, 1571, 1509, 1493, 1464, 1443, 1290, 1248, 1173, 1088, 1033, 905, 832, 777, 704 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O_2$: calcd 318.1620, found 318.1630.



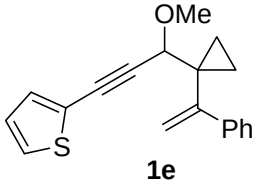
1c

Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 40:1) afforded the title compound as a brown sticky liquid in 92% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.74-0.80 (m, 1H), 0.98-1.03 (m, 1H), 1.18-1.26 (m, 2H), 2.32 (s, 3H), 3.31 (s, 3H), 4.45 (s, 1H), 5.28 (d, *J* = 0.6 Hz, 1H), 5.42 (s, 1H), 7.09 (d, *J* = 7.8 Hz, 2H), 7.21-7.37 (m, 5H), 7.59 (dd, *J* = 8.1, 1.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.7, 12.3, 21.4, 28.2, 56.7, 74.7, 84.6, 86.7, 115.1, 119.5, 127.0 (2C), 127.5, 128.1 (2C), 129.0 (2C), 131.6 (2C), 138.4, 140.2, 148.8; IR (KBr): 3082, 3053, 3027, 2993, 2925, 28219, 2223, 1624, 1573, 1510, 1493, 1445, 1419, 1324, 1257, 1188, 1088, 1023, 1012, 904, 816, 777, 704 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O$: calcd 302.1671, found 302.1668.



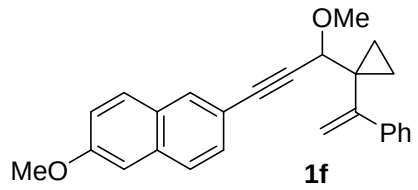
1d

Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 50:1) afforded the title compound as a light brown sticky liquid in 77% yield (the starting material of **B1d** was recovered in 14% yield). ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.78-0.82 (m, 1H), 0.99-1.04 (m, 1H), 1.16-1.23 (m, 2H), 3.31 (s, 3H), 4.42 (s, 1H), 5.29 (s, 1H), 5.43 (s, 1H), 7.25-7.37 (m, 7H), 7.57-7.60 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.9, 12.1, 28.3, 56.9, 74.7, 85.5, 86.5, 115.3, 121.0, 127.0 (2C), 127.6, 128.2 (2C), 128.6 (2C), 132.9 (2C), 134.4, 140.1, 148.6; IR (KBr): 3084, 3055, 2990, 2929, 2853, 2820, 2221, 1489, 1464, 1445, 1397, 1324, 1254, 1188, 1091, 1016, 906, 828, 777, 705 cm^{-1}; HRMS (EI) for $C_{21}H_{19}OCl$: calcd 322.1124, found 322.1124.



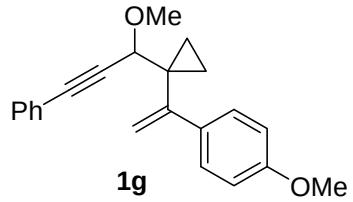
1e

Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1) afforded the title compound as a brown red sticky liquid in 79% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.76-0.81 (m, 1H), 0.99-1.03 (m, 1H), 1.15-1.26 (m, 2H), 3.30 (s, 3H), 4.44 (s, 1H), 5.28 (d, *J* = 1.5 Hz, 1H), 5.43 (d, *J* = 1.5 Hz, 1H), 6.94 (dd, *J* = 5.4, 3.3 Hz, 1H), 7.16 (dd, *J* = 3.3, 1.5 Hz, 1H), 7.22 (dd, *J* = 5.4, 1.2 Hz, 1H), 7.25-7.37 (m, 3H), 7.56-7.60 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.9, 12.2, 28.3, 56.9, 74.9, 79.8, 89.5, 115.2, 122.4, 126.9, 127.0 (2C), 127.1, 127.5, 128.2 (2C), 132.2, 140.0, 148.6; IR (KBr): 3083, 3055, 2995, 2930, 2900, 2820, 2216, 1623, 1573, 1517, 1493, 1444, 1422, 1355, 1323, 1187, 1088, 1028, 996, 906, 850, 777, 703 cm^{-1}; HRMS (EI) for $C_{19}H_{18}OS$: calcd 294.1078, found 294.1075.



1f

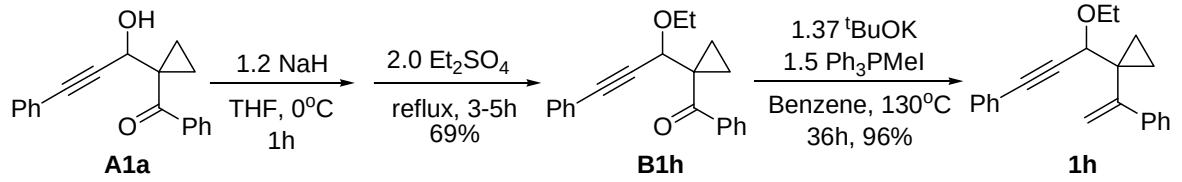
Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 25:1) afforded the title compound as a brown sticky liquid in 63% yield (the starting material of **B1f** was recovered in 26% yield). ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.78-0.84 (m, 1H), 1.02-1.08 (m, 1H), 1.23-1.33 (m, 2H), 3.35 (s, 3H), 3.91 (s, 3H), 4.50 (s, 1H), 5.31 (d, *J* = 0.9 Hz, 1H), 5.44 (d, *J* = 1.2 Hz, 1H), 7.05 (d, *J* = 2.4 Hz, 1H), 7.15 (dd, *J* = 8.7, 2.4 Hz, 1H), 7.24-7.43 (m, 4H), 7.60-7.69 (m, 4H), 7.84 (s, 1H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.8, 12.4, 28.3, 55.3, 56.8, 74.9, 84.9, 87.1, 105.7, 115.2, 117.4, 119.4, 126.7, 127.1 (2C), 127.5, 128.2 (2C), 128.3, 129.0, 129.2, 131.4, 134.1, 140.2, 148.8, 158.3; IR (KBr): 3082, 3057, 3000, 2935, 2901, 2840, 2820, 2221, 1629, 1601, 1498, 1483, 1462, 1390, 1333, 1270, 1244, 1198, 1162, 1122, 1087, 1029, 891, 853, 806, 777, 705, 663 cm^{-1}; HRMS (EI) for $C_{26}H_{24}O_2$: calcd 368.1776, found 368.1769.



1g

Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 15:1) afforded the title compound as a brown liquid in 90% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.73-0.79 (m, 1H), 0.97-1.03 (m, 1H), 1.16-1.26 (m, 2H), 3.33 (s, 3H), 3.81 (s, 3H), 4.46 (s, 1H), 5.21 (d, *J* = 0.9 Hz, 1H), 5.36 (d, *J* = 0.9 Hz, 1H), 6.87-6.91 (m, 2H), 7.28-7.31 (m, 3H), 7.38-7.42 (m, 2H), 7.52-7.56 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.7, 12.2, 28.2, 55.2, 56.8, 74.8, 85.4, 86.6, 113.5 (2C), 113.7, 122.6, 128.1 (2C), 128.2 (2C), 128.3, 131.7 (2C), 132.5, 148.1, 159.1; IR (KBr): 3084, 3034, 2998, 2933, 2902, 2835, 2821, 2222, 1607, 1573, 1510, 1490, 1463, 1443, 1324, 1292, 1248, 1177, 1087, 1032, 900, 836, 757, 691 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O_2$: calcd 318.1620, found 318.1620.

Synthese of the substrate 1h:

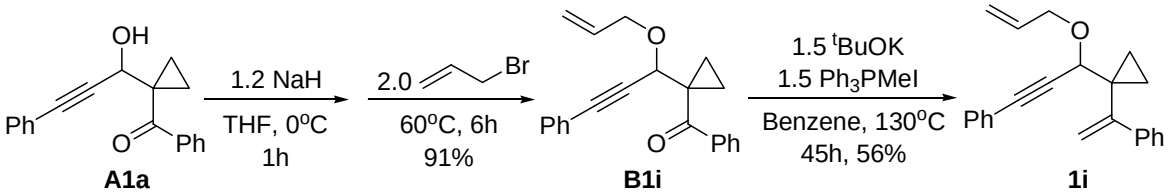


To a solution of **A1a** (3.0 mmol, 0.83 g) in THF (10 mL) was added NaH (3.6 mmol, 0.14 g, 60% dispersion in mineral oil) slowly at 0 ^{o}C. The reaction mixture was stirred for 1 hour at 0 ^{o}C and then Et_2SO_4 (6.0 mmol, 0.79 mL) was added slowly at the same temperature. The reaction mixture was warmed up to room temperature and then refluxed for about 5 hours until the reaction was complete as monitored by thin-layer chromatography. The reaction mixture was cooled to room temperature and quenched with a saturated aqueous solution of NH_4Cl, extracted with EtOAc. The extract was washed with brine and dried over Na_2SO_4. The solvent was evaporated *in vacuo* and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20:1-10:1) to afford the title compound **B1h** as a brown yellow sticky liquid in 69% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.98-1.06 (m, 4H), 1.32-1.40 (m, 2H), 1.54-1.59 (m, 1H), 3.14-3.24 (m, 1H), 3.64-3.72 (m, 1H), 5.20 (s, 1H), 7.27-7.32 (m, 3H), 7.39-7.54 (m, 5H), 7.80 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 8.0, 12.7, 14.7, 33.2, 64.5, 71.9, 84.2, 87.0, 122.1, 127.9 (2C), 128.2 (2C), 128.3 (2C), 128.6, 131.68, 131.73 (2C), 137.7, 201.7; IR (KBr): 3080, 3060, 3022, 2976, 2929, 2871, 2221, 1681, 1598, 1578, 1490, 1446, 1347, 1316, 1304, 1258, 1198, 1176, 1122, 1084, 1032, 986, 922, 757, 721, 692, 646 cm^{-1}; HRMS (EI) for $C_{21}H_{20}O_2$: calcd 304.1463, found 304.1467.

Methylenation:[4] To a suspension of Ph_3PMeI (2.91 mmol, 1.18 g) in benzene (5.8 mL) was added ^{t}BuOK (2.65 mmol, 0.30 g) at room temperature under the nitrogen atmosphere. The reaction mixture was heated to 130 ^{o}C and stirred for 1h, then most of the benzene was vaporized via a needle until the reaction mixture became slurry. The mixture was cooled to room temperature and a solution of **B1h** (1.94 mmol, 0.59 g) in benzene (1.9 mL) was added. The mixture was heated to 130 ^{o}C again and then most of the benzene was vaporized via a needle until the reaction mixture became slurry. The Schlenk tube was

sealed and the reaction mixture was continued to stir for 36 hours. The mixture was cooled to room temperature and quenched with H_2O, extracted with Et_2O. The extract was dried over Na_2SO_4. The solvent was evaporated *in vacuo* and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1) to yield the desired **1h** as a brown yellow sticky liquid in 96% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.75-0.79 (m, 1H), 0.99-1.03 (m, 1H), 1.09 (t, *J* = 6.9 Hz, 3H), 1.19-1.28 (m, 2H), 3.23-3.33 (m, 1H), 3.66-3.77 (m, 1H), 4.53 (s, 1H), 5.29 (d, *J* = 1.2 Hz, 1H), 5.41 (d, *J* = 0.9 Hz, 1H), 7.25-7.42 (m, 8H), 7.56-7.60 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 8.0, 11.8, 14.9, 28.3, 64.6, 72.6, 85.9, 86.2, 115.1, 122.7, 127.1 (2C), 127.4, 128.1 (2C), 128.2 (2C), 128.2, 131.7 (2C), 140.4, 149.0; IR (KBr): 3082, 3055, 3021, 2975, 2869, 2221, 1600, 1491, 1443, 1318, 1121, 1087, 1029, 905, 777, 756, 704, 691 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O$: calcd 302.1671, found 302.1676.

Synthese of the substrate 1i:

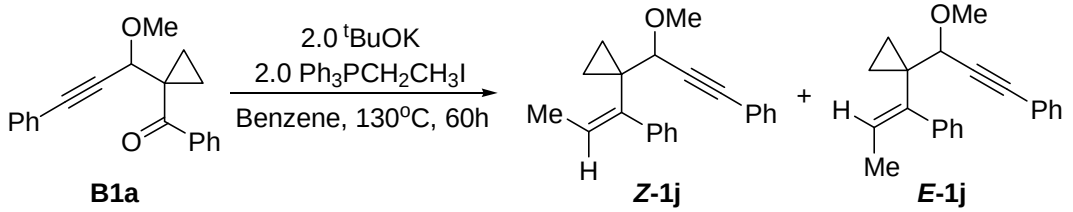


To a solution of **A1a** (10.0 mmol, 2763 mg) in THF (30 mL) was added NaH (12.0 mmol, 0.48 g, 60% dispersion in mineral oil) slowly at 0 °C. The reaction mixture was stirred for 1 hour at 0 °C and then 3-bromo-1-propene (20.0 mmol, 1.73 mL) was added slowly at the same temperature. The reaction mixture was warmed up to 60 °C and stirred for 6 hours. Then the mixture was cooled to room temperature and quenched with a saturated aqueous solution of NH_4Cl, extracted with EtOAc. The extract was washed with brine and dried over Na_2SO_4. The solvent was evaporated *in vacuo* and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1) to afford the title compound **B1i** as a yellow liquid in 91% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 400 MHz): δ 1.03-1.07 (m, 1H), 1.34-1.40 (m, 2H), 1.55-1.59 (m, 1H), 3.76-3.82 (m, 1H), 4.12-4.17 (m, 1H), 5.01 (dq, *J* = 14.4, 1.6 Hz, 1H), 5.11 (dq, *J* = 17.2, 2.0 Hz, 1H), 5.25 (s, 1H), 5.50-5.59 (m, 1H), 7.28-7.35 (m, 3H), 7.39-7.46 (m, 4H), 7.49-7.54 (m, 1H), 7.79-7.82 (m,

2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 100 MHz): δ 8.0, 12.8, 33.2, 69.7, 71.2, 83.8, 87.3, 117.5, 122.0, 128.0 (2C), 128.2 (2C), 128.3 (2C), 128.7, 131.7 (2C), 133.7, 137.5, 201.4. One carbon overlapped with other signals; IR (KBr): 3081, 3056, 3019, 2917, 2856, 2222, 1646, 1624, 1599, 1573, 1491, 1443, 1421, 1352, 1318, 1253, 1130, 1070, 1030, 993, 910, 777, 756, 704, 691cm^{-1}; HRMS (EI) for $C_{22}H_{20}O_2$: calcd 316.1463, found 316.1464.

Methylenation:[4] To a suspension of Ph_3PMeI (13.27 mmol, 5.36 g) in benzene (15 mL) was added ^{t}BuOK (13.27 mmol, 1.49 g) at room temperature under the nitrogen atmosphere. The reaction mixture was heated to 130 ^{o}C and stirred for 1h, then most of the benzene was vaporized via a needle until the reaction mixture became slurry. The mixture was cooled to room temperature and a solution of **B1i** (8.85 mmol, 2.8 g) in benzene was added. The mixture was heated to 130 ^{o}C again and stirred for 15 min, then most of the benzene was vaporized via a needle until the reaction mixture became slurry. The Schlenk tube was sealed and the reaction mixture was continued to stir for 45 hours. The mixture was cooled to room temperature and quenched with H_2O, extracted with Et_2O. The extract was dried over Na_2SO_4. The solvent was evaporated *in vacuo* and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 50:1-25:1) to yield the desired **1i** as a yellow sticky liquid in 56% yield (the starting material of **B1i** was recovered in 33% yield). ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.73-0.84 (m, 1H), 0.96-1.07 (m, 1H), 1.21-1.29 (m, 2H), 3.85 (dd, *J* = 12.9, 6.0 Hz, 1H), 4.16-4.22 (m, 1H), 4.58 (s, 1H), 5.04 (dd, *J* = 10.5, 1.2 Hz, 1H), 5.07 (dd, *J* = 17.7, 1.5 Hz, 1H), 5.30 (s, 1H), 5.43 (s, 1H), 5.66-5.79 (m, 1H), 7.25-7.42 (m, 8H), 7.58-7.60 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 8.1, 12.0, 28.3, 69.8, 72.1, 85.8, 86.3, 115.3, 117.0, 122.6, 127.1 (2C), 127.5, 128.1 (2C), 128.2 (2C), 128.3, 131.7 (2C), 134.3, 140.2, 148.8; IR (KBr): 3081, 3056, 3019, 2917, 2856, 2222, 1646, 1624, 1599, 1573, 1491, 1443, 1421, 1352, 1318, 1253, 1130, 1070, 1030, 993, 910, 777, 756, 704, 691cm^{-1}; HRMS (EI) for $C_{23}H_{22}O$: calcd 314.1671, found 314.1667.

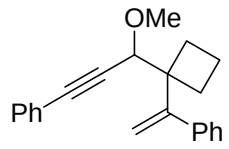
Synthese of the substrates *Z*-1j and *E*-1j:



B1a ***Z*-1j** ***E*-1j**

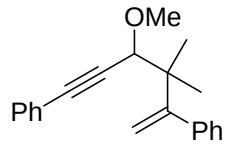
To a suspension of $Ph_3PCH_2CH_3I$ (10.0 mmol, 4.18 g) in benzene (10 mL) was added ^tBuOK (10.0 mmol, 1.12 g) at room temperature under the nitrogen atmosphere. The reaction mixture was heated to 130 ^oC and stirred for 1h, then most of the benzene was vaporized via a needle until the reaction mixture became slurry. The mixture was cooled to room temperature and a solution of **B1a** (5.0 mmol, 1.45 g) in benzene (5 mL) was added. The mixture was heated to 130 ^oC again, then most of the benzene was vaporized via a needle until the reaction mixture became slurry. The Schlenk tube was sealed and the reaction mixture was continued to stir for 60 hours. The mixture was cooled to room temperature and quenched with H_2O, extracted with Et_2O. The extract was dried over Na_2SO_4. The solvent was evaporated *in vacuo* and the residue was purified by flash chromatography on silica gel to give the desired ***Z*-1j** (eluent: petroleum ether/CH_2Cl_2 = 15:1) as a light brown sticky liquid in 24% yield, and ***E*-1j** (eluent: petroleum ether/CH_2Cl_2 = 5:1-2:1) as a brown sticky liquid in 23% yield. ***Z*-1j :** ^1H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.58-0.66 (m, 2H), 1.24-1.35 (m, 2H), 2.01 (d, *J* = 7.2 Hz, 3H), 3.47 (s, 3H), 4.62 (s, 1H), 6.12 (q, *J* = 7.2 Hz, 1H), 7.19-7.22 (m, 1H), 7.25-7.32 (m, 5H), 7.41-7.44 (m, 2H), 7.60-7.62 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 10.7, 13.4, 15.4, 24.1, 57.5, 76.7, 85.8, 86.5, 122.6, 126.3, 127.2 (2C), 127.7 (2C), 128.2 (2C), 128.3, 128.5, 131.7 (2C), 141.2, 142.2; IR (KBr): 3081, 3055, 3019, 3001, 2930, 2904, 2855, 2819, 2222, 1598, 1490, 1443, 1320, 1189, 1090, 1031, 1014, 963, 914, 892, 840, 757, 691 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O$: calcd 302.1671, found 302.1672. ***E*-1j:** ^1H NMR ($CDCl_3$, Me_4Si, 300Mz): δ 0.69-0.75 (m, 1H), 1.02-1.14 (m, 3H), 1.54 (d, *J* = 7.2 Hz, 3H), 3.33 (s, 3H), 5.29 (s, 1H), 5.75 (q, *J* = 6.9 Hz, 1H), 7.24-7.31 (m, 6H), 7.33-7.40 (m, 4H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 7.3, 11.7, 14.8, 30.2, 56.6, 74.6, 85.6, 86.2, 122.7, 123.6, 126.7, 128.0 (2C), 128.2, 128.2 (2C), 129.2 (2C), 131.7 (2C), 140.1, 141.3; IR (KBr): 3080, 3054, 3019, 2983, 2930, 2856, 2819, 2221, 1599, 1491, 1442, 1323, 1189, 1090, 1028, 1006, 996, 962, 914, 756, 702, 691 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O$: calcd 302.1671, found 302.1668. The

structures of ***Z*-1j** and ***E*-1j** were confirmed by 2D NOESY spectra.



3a

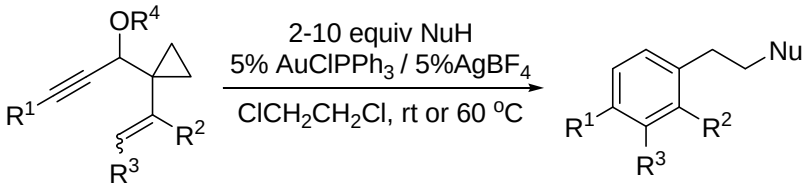
It was synthesized from cyclobutyl(phenyl)methanone using LDA as base according to the procedure described for **1a**. Overall yield: 50%. ^{1}H NMR ($CDCl_3$, Me_4Si, 300MHz): δ 1.78-1.92 (m, 1H), 1.99-2.14 (m, 1H), 2.29-2.57 (m, 4H), 3.42 (s, 3H), 4.20 (s, 1H), 5.340 (s, 1H), 5.344 (s, 1H), 7.25-7.37 (m, 8H), 7.43-7.46 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 15.4, 28.9, 30.1, 50.9, 57.4, 77.4, 86.5, 86.9, 114.8, 123.0, 126.9, 127.8 (2C), 127.9 (2C), 128.2, 128.3 (2C), 131.7 (2C), 141.4, 153.3; IR (KBr): 3080, 3055, 2987, 2939, 2820, 2214, 1599, 1572, 1490, 1442, 1323, 1195, 1093, 903, 777, 756, 691cm^{-1}; HRMS (EI) for $C_{22}H_{22}O$: calcd 302.1671, found 302.1672.



3b

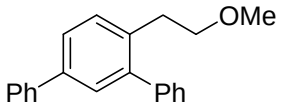
It was synthesized from 2-methyl-1-phenylpropan-1-one using LDA as base according to the procedure described for **1a**. Overall yield: 13%. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.26 (s, 3H), 1.35 (s, 3H), 3.41 (s, 3H), 3.92 (s, 1H), 4.95 (s, 1H), 5.33 (s, 1H), 7.20-7.24 (m, 2H), 7.27-7.33 (m, 6H), 7.42-7.45 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 22.1, 24.4, 44.4, 57.0, 77.6, 86.7, 87.1, 114.9, 122.9, 126.6, 127.5 (2C), 128.20, 128.23 (2C), 129.2 (2C), 131.7 (2C), 142.4, 155.7; IR (KBr): 3079, 3055, 2974, 2934, 2819, 2215, 1599, 1490, 1442, 1378, 1333, 1178, 1147, 1094, 1029, 910, 756, 704, 691 cm^{-1}; HRMS (EI) for $C_{21}H_{22}O$: calcd 290.1671, found 290.1674.

General procedure for Au(I)-catalyzed benzannulation of 3-alkoxy-1,5-enynes.



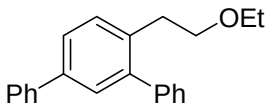
1 **2**

To a solution of 1,5-enyne **1** (0.3 mmol) and alcohol (2-10 equiv) in $ClCH_2CH_2Cl$ (6 mL) was added Ph_3PAuCl (0.015 mmol, 7.4 mg) and $AgBF_4$ (0.015 mmol, 300 uL, used as a 0.05 M solution in toluene). The resulting solution was stirred at room temperature or 60 ^oC until the reaction was complete as monitored by thin-layer chromatography. The solvent was evaporated under the reduced pressure and the residue was purified by chromatography on silica gel (eluent: petroleum ether / ethyl acetate) to afford the benzene derivatives **2**.



2a

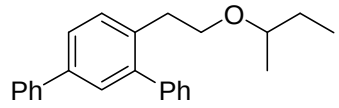
(**2a**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 50:1) afforded the title compound **2a** as a brown sticky liquid in 83% yield. ^1H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.91 (t, J = 7.2 Hz, 2H), 3.24 (s, 3H), 3.47 (t, J = 7.2 Hz, 2H), 7.28-7.47 (m, 10H), 7.54 (dd, J = 7.8, 2.1 Hz, 1H), 7.60-7.62 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 32.8, 58.5, 73.0, 126.0, 127.0 (3C), 127.2, 128.1 (2C), 128.7 (2C), 128.8, 129.2 (2C), 130.1, 135.2, 139.0, 140.6, 141.5, 142.7; IR (KBr): 3057, 3027, 2976, 2925, 2871, 2824, 1600, 1479, 1443, 1383, 1180, 1113, 1027, 1012, 1000, 968, 895, 833, 762, 700 cm^{-1}; HRMS (EI) for $C_{21}H_{20}O$: calcd 288.1514, found 288.1513.



2b

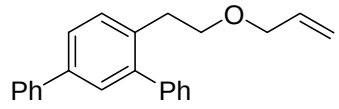
(**2b**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1) afforded the title compound **2b** as a light yellow

sticky liquid in 69% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.13 (t, *J* = 7.2 Hz, 3H), 2.91 (t, *J* = 6.9 Hz, 2H), 3.38 (q, *J* = 7.2 Hz, 2H), 3.51 (t, *J* = 7.5 Hz, 2H), 7.28-7.42 (m, 10H), 7.52-7.55 (m, 1H), 7.59 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 15.1, 33.0, 66.0, 71.0, 125.9, 126.95, 126.97 (2C), 127.1, 128.1 (2C), 128.7 (2C), 128.8, 129.2 (2C), 130.2, 135.3, 138.9, 140.6, 141.5, 142.7; IR (KBr): 3058, 3027, 2971, 2865, 1600, 1479, 1442, 1376, 1354, 1261, 1106, 1075, 1025, 894, 800, 762, 700 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O$: calcd 302.1671, found 302.1674.



2c

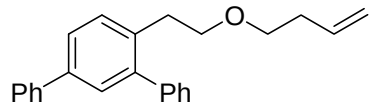
(**2c**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 50:1) afforded the title compound **2c** as a light yellow sticky liquid in 77% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.81 (t, *J* = 7.2 Hz, 3H), 1.02 (d, *J* = 6.0 Hz, 3H), 1.26-1.50 (m, 2H), 2.90 (t, *J* = 7.5 Hz, 2H), 3.16-3.23 (m, 1H), 3.40-3.48 (m, 1H), 3.51-3.59 (m, 1H), 7.28-7.47 (m, 10H), 7.53 (dd, *J* = 8.1, 2.1 Hz, 1H), 7.58-7.62 (m, 2H),; ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 9.7, 19.1, 29.0, 33.5, 68.7, 76.6, 125.9, 126.9, 127.0 (2C), 127.1, 128.1 (2C), 128.69 (2C), 128.74, 129.2 (2C), 130.3, 135.5, 138.9, 140.7, 141.6, 142.7; IR (KBr): 3058, 3028, 2966, 2931, 2873, 1600, 1479, 1464, 1443, 1373, 1340, 1261, 1172, 1139, 1113, 1083, 1026, 1012, 895, 830, 762, 700 cm^{-1}; HRMS (EI) for $C_{24}H_{26}O$: calcd 330.1984, found 330.1981.



2d

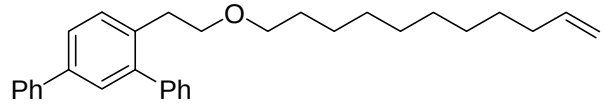
(**2d**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 80:1) afforded the title compound **2d** as a light yellow sticky liquid in 84% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.93 (t, *J* = 7.2 Hz, 2H), 3.52 (t, *J* = 7.2 Hz, 2H), 3.87 (dd, *J* = 5.1, 0.9 Hz, 2H), 5.11 (d, *J* = 10.5 Hz, 1H), 5.18 (d, *J* = 17.4 Hz, 1H), 5.76-5.89 (m, 1H), 7.28-7.46 (m, 10H), 7.53 (d, *J* = 7.8 Hz, 1H), 7.59 (d, *J*

= 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 32.9, 70.6, 71.6, 116.7, 125.9, 127.0 (3C), 127.2, 128.1 (2C), 128.7 (2C), 128.8, 129.2 (2C), 130.2, 134.7, 135.2, 139.0, 140.6, 141.5, 142.7; IR (KBr): 3058, 3027, 2963, 2856, 1600, 1479, 1443, 1261, 1097, 1025, 923, 895, 800, 762, 700 cm^{-1}; HRMS (EI) for $C_{23}H_{22}O$: calcd 314.1671, found 314.1667.



2e

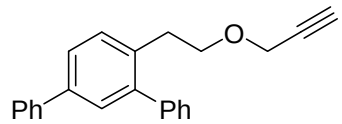
(**2e**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1) afforded the title compound **2e** as a colorless sticky liquid in 91% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.26 (qt, *J* = 6.9, 1.5 Hz, 2H), 2.91 (t, *J* = 7.5 Hz, 2H), 3.36 (t, *J* = 6.6 Hz, 2H), 3.52 (t, *J* = 7.2 Hz, 2H), 4.98-5.07 (m, 2H), 5.69-5.82 (m, 1H), 7.27-7.46 (m, 10H), 7.53 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.57-7.61 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 32.9, 34.1, 70.0, 71.3, 116.2, 125.9, 127.0 (3C), 127.1, 128.1 (2C), 128.7 (2C), 128.8, 129.2 (2C), 130.2, 135.2, 135.3, 138.9, 140.6, 141.5, 142.6; IR (KBr): 3059, 3027, 2931, 2859, 1641, 1600, 1479, 1442, 1391, 1362, 1260,1108, 1026, 1012, 996, 914, 831, 762, 700 cm^{-1}; HRMS (EI) for $C_{24}H_{24}O$: calcd 328.1827, found 328.1828.



2f

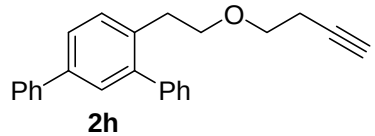
(**2f**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1) afforded the title compound **2f** as a colorless sticky liquid in 90% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.25-1.43 (m, 12H), 1.47-1.59 (m, 2H), 1.99-2.06 (m, 2H), 2.91 (t, *J* = 7.2, Hz, 2H), 3.30 (t, *J* = 6.6 Hz, 2H), 3.50 (t, *J* = 7.2 Hz, 2H), 4.90-5.02 (m, 2H), 5.73-5.86 (m, 1H), 7.27-7.46 (m, 10H), 7.53 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.58-7.61 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 26.1, 28.9, 29.1, 29.4(2C), 29.5, 29.6, 33.0, 33.8, 70.8, 71.2, 114.1, 125.9, 126.9, 127.0 (2C), 127.1, 128.1 (2C), 128.7 (2C), 128.8, 129.2 (2C), 130.2, 135.4, 138.9, 139.2, 140.7, 141.6, 142.6; IR

(KBr): 3060, 3028, 2926, 2854, 1640, 1600, 1479, 1465, 1442, 1366, 1111, 1027, 1012, 994, 910, 831, 761, 700 cm^{-1}; HRMS (EI) for $C_{31}H_{38}O$: calcd 426.2923, found 426.2926.



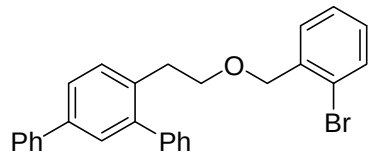
2g

(**2g**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1) afforded the title compound **2g** as a light yellow sticky liquid in 54% yield. ^1H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.38 (s, 1H), 2.94 (t, *J* = 7.2 Hz, 2H), 3. 63 (t, *J* = 7.2 Hz, 2H), 4.05 (s, 2H), 7.30-7.47 (m, 10H), 7.50 (d, *J* = 7.5 Hz, 1H), 7.61 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 32.7, 57.9, 70.3, 74.3, 79.7, 126.0, 127.0 (2C), 127.2, 128.2 (2C), 128.7 (2C), 128.9, 129.2 (2C), 130.2, 134.8, 139.1, 140.6, 141.4, 142.7. One carbon overlapped with other signals; IR (KBr): 3291, 3057, 3027, 2936, 2861, 2114, 1599, 1479, 1442, 1389, 1356, 1267, 1138, 1356, 1267, 1097, 1026, 1011, 896, 834, 762, 700 cm^{-1}; HRMS (EI) for $C_{23}H_{20}O$: calcd 312.1514, found 312.1525.



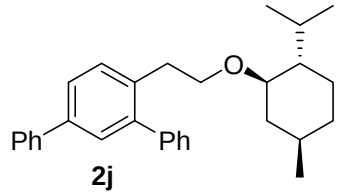
2h

(**2h**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1) afforded the title compound **2h** as a light yellow sticky liquid in 61% yield. ^1H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.95 (t, *J* = 2.4 Hz, 1H), 2.38 (td, *J* = 6.9, 2.4 Hz, 2H), 2.92 (t, *J* = 7.5 Hz, 2H), 3.45 (t, *J* = 6.9 Hz, 2H), 3.56 (t, *J* = 7.5 Hz, 2H), 7.29-7.47 (m, 10H), 7.54 (dd, *J* = 8.1, 1.8 Hz, 1H), 7.60 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 19.7, 32.8, 68.6, 69.2, 71.4, 81.3, 126.0, 127.0 (3C), 127.2, 128.2 (2C), 128.7 (2C), 128.8, 129.2 (2C), 130.2, 135.1, 139.0, 140.6, 141.5, 142.7; IR (KBr): 3296, 3057, 3027, 2916, 2865, 2116, 1599, 1479, 1442, 1391, 1364, 1110, 1075, 1026, 1012, 895, 832, 762, 700, 638 cm^{-1}; HRMS (EI) for $C_{24}H_{22}O$: calcd 326.1671, found 326.1672.



2i

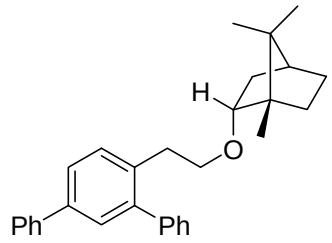
(**2i**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1) afforded the title compound **2i** as a light yellow sticky liquid in 75% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.99 (t, *J* = 6.9 Hz, 2H), 3.62 (t, *J* = 7.5 Hz, 2H), 4.45 (s, 2H), 7.06 (td, *J* = 7.2, 1.5 Hz, 1H), 7.18-7.23 (m, 1H), 7.28-7.43 (m, 10H), 7.45-7.48 (m, 2H), 7.53 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.57-7.60 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 33.0, 71.2, 71.9, 122.4, 125.9, 126.96 (2C), 126.98, 127.2, 127.2, 128.1 (2C), 128.65, 128.69 (2C), 128.8, 129.2 (2C), 130.3, 132.3, 135.1, 137.6, 139.0, 140.6, 141.4, 142.7. One carbon overlapped with other signals; IR (KBr): 3057, 3027, 2926, 2862, 1599, 1568, 1479, 1441, 1390, 1357, 1263, 1205, 1122, 1101, 1027, 895, 832, 751, 700 cm^{-1}; HRMS (EI) for $C_{27}H_{23}OBr$: calcd 442.0932 found 442.0940.



2j

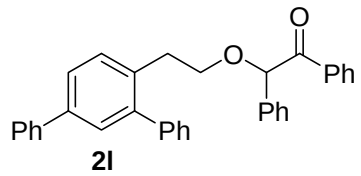
(**2j**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1) afforded the title compound **2j** as a brown sticky liquid in 66% yield. $[a]_D^{20}$ = -37.7 (*c* = 1.22, $CHCl_3$); ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 0.63 (d, *J* = 6.9 Hz, 3H), 0.69-0.94 (m, 9H), 1.10-1.28 (m, 2H), 1.52-1.61 (m, 2H), 1.90 (d, *J* = 9.3 Hz, 1H), 2.02-2.08 (m, 1H), 2.82-2.93 (m, 3H), 3.29-3.37 (m, 1H), 3.64-3.72 (m, 1H), 7.27-7.46 (m, 10H), 7.51 (dd, *J* = 7.8, 2.1 Hz, 1H), 7.59 (d, *J* = 7.2, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 100 MHz): δ 16.1, 20.9, 22.3, 23.3, 25.4, 31.5, 33.7, 34.5, 40.4, 48.1, 68.9, 79.2, 125.9, 126.9, 127.0 (2C), 127.1, 128.1 (2C), 128.68 (2C), 128.74, 129.2 (2C), 130.3, 135.4, 139.0, 140.7, 141.6, 142.6; IR (KBr): 3058, 3027, 2955, 2922, 2867, 1599, 1479, 1453, 1384, 1369, 1343, 1262, 1179, 1107, 1089, 1013, 895, 831, 761, 700 cm^{-1}; HRMS

(EI) for $C_{30}H_{36}O$: calcd 412.2766, found 412.2762.



2k

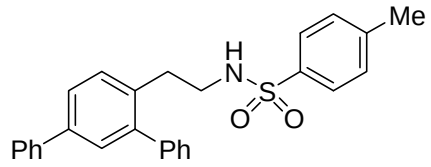
(**2k**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1) afforded the title compound **2k** as a brown sticky liquid in 72% yield. $[a]_D^{20}$ = -25.4 (*c* = 0.674, $CHCl_3$); ^1H NMR ($CDCl_3$, Me_4Si, 400 MHz): δ 0.77-0.88 (m, 10H), 1.10-1.17 (m, 2H), 1.43-1.57 (m, 1H), 1.61-1.66 (m, 1H), 1.87-2.00 (m, 2H), 2.86-2.91 (m, 2H), 3.39-3.55 (m, 3H), 7.29-7.46 (m, 10H), 7.53 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.59-7.62 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 100 MHz): δ 14.0, 18.8, 19.8, 26.6, 28.2, 33.4, 36.2, 44.9, 47.7, 49.1, 70.4, 84.6, 125.8, 126.9, 127.0 (2C), 127.1, 128.1 (2C), 128.7 (3C), 129.3 (2C), 130.5, 135.8, 138.8, 140.8, 141.7, 142.6; IR (KBr): 3058, 3027, 2949, 2873, 1600, 1479, 1452, 1387, 1369, 1358, 1232, 1139, 1117, 1094, 1076, 1026, 1013, 895, 830, 761, 700 cm^{-1}; HRMS (EI) for $C_{30}H_{34}O$: calcd 410.2610, found 410.2609.



2l

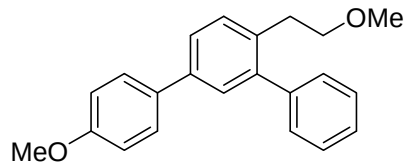
(**2l**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 25:1) afforded the title compound **2l** as a brown sticky liquid in 33% yield. ^1H NMR ($CDCl_3$, Me_4Si, 400 MHz): δ 2.98-3.02 (m, 2H), 3.60-3.64 (m, 2H), 5.40 (s, 1H), 7.22-7.46 (m, 18H), 7.50 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.59-7.61 (m, 2H), 7.87-7.89 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 33.0, 70.2, 85.5, 125.9, 126.95 (3C), 127.12 (2C), 127.19, 128.1 (2C), 128.2, 128.3 (2C), 128.6 (2C), 128.7 (2C), 128.8, 129.1 (4C), 130.5, 133.1, 134.6, 134.9, 136.2, 139.1, 140.6, 141.3, 142.6, 197.4; IR (KBr): 3059, 3027, 2928, 2866, 1694, 1677, 1597, 1578, 1479, 1448, 1391, 1308, 1275, 1239, 1214, 1180, 1107, 1075, 1027, 968, 895, 831, 762, 697 cm^{-1}; HRMS (EI) for

$C_{34}H_{28}O_2$: calcd 468.2089, found 468.2093.



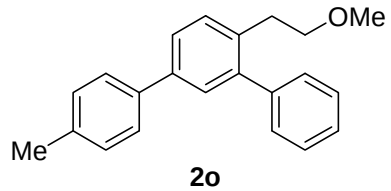
2m

(**2m**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) afforded the title compound **2m** as a brown sticky liquid in 23% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.40 (s, 3H), 2.81 (t, J = 6.9 Hz, 2H), 3.00 (q, J = 6.9 Hz, 2H), 4.23 (t, J = 6.0 Hz, 1H), 7.20-7.23 (m, 5H), 7.25-7.46 (m, 7H), 7.51 (dd, J = 7.8, 1.8 Hz, 1H), 7.58 (d, J = 7.5 Hz, 4H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 21.5, 32.7, 43.4, 126.2, 126.96 (2C), 127.00 (2C), 127.2, 127.4, 128.3 (2C), 128.8 (2C), 129.0 (2C), 129.1, 129.6 (2C), 130.1, 134.0, 136.7, 139.6, 140.3, 141.0, 142.6, 143.2; IR (KBr): 3285, 3058, 3028, 2926, 2872, 1710, 1598, 1479, 1443, 1328, 1159, 1093, 1076, 896, 814, 762, 701, 662, 550 cm^{-1}; HRMS (EI) for $C_{27}H_{25}NO_2S$: calcd 427.1606, found 427.1603.



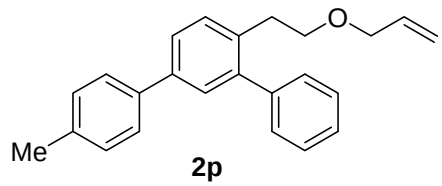
2n

(**2n**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20:1) afforded the title compound **2n** as a light brown sticky liquid in 91% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.90 (t, J = 7.2 Hz, 2H), 3.23 (s, 3H), 3.46 (t, J = 7.2 Hz, 2H), 3.79 (s, 3H), 6.92-6.96 (m, 2H), 7.34-7.43 (m, 7H), 7.47-7.54 (m, 3H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 32.7, 55.2, 58.4, 73.0, 114.1 (2C), 125.5, 126.9, 127.9 (2C), 128.1 (2C), 128.3, 129.2 (2C), 130.1, 133.1, 134.4, 138.6, 141.6, 142.6, 159.0; IR (KBr): 3053, 3024, 2930, 2894, 2834, 2808, 1609, 1580, 1519, 1499, 1484, 1463, 1442, 1384, 1287, 1249, 1179, 1112, 1041, 1029, 967, 896, 823, 773, 704 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O_2$: calcd 318.1620, found 318.1618.



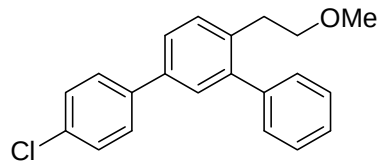
2o

(**2o**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20:1) afforded the title compound **2o** as a light brown sticky liquid in 88% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.35 (s, 3H), 2.90 (t, *J* = 7.2 Hz, 2H), 3.22 (s, 3H), 3.46 (t, *J* = 7.2 Hz, 2H), 7.20 (t, *J* = 7.8 Hz, 2H), 7.31-7.53 (m, 10H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 21.0, 32.7, 58.4, 73.0, 125.8, 126.8 (2C), 126.9, 128.1 (2C), 128.6, 129.2 (2C), 129.4 (2C), 130.1, 134.8, 136.9, 137.7, 138.9, 141.6, 142.6; IR (KBr): 3052, 3024, 2976, 2922, 2870, 2824, 1600, 1518, 1484, 1444, 1383, 1185, 1113, 1021, 1011, 967, 897, 811, 772, 703 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O$: calcd 302.1671, found 302.1662.



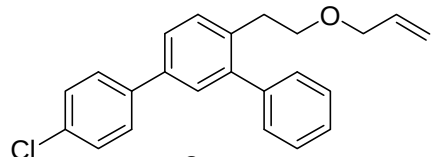
2p

(**2p**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1) afforded the title compound **2p** as a light yellow sticky liquid in 86% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.36 (s, 3H), 2.92 (t, *J* = 7.5 Hz, 2H), 3.52 (t, *J* = 7.5 Hz, 2H), 3.85-3.88 (m, 2H), 5.09-5.21 (m, 2H), 5.76-5.89 (m, 1H), 7.21 (d, *J* = 7.8 Hz, 2H), 7.31-7.38 (m, 7H), 7.40-7.53 (m, 3H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 21.0, 32.9, 70.6, 71.6, 116.7, 125.7, 126.8 (2C), 126.9, 128.1 (2C), 128.56 129.2 (2C), 129.4 (2C), 130.2, 134.7, 134.8, 136.9, 137.7, 138.9, 141.6, 142.6; IR (KBr): 3054, 3023, 2921, 2858, 1600, 1518, 1498, 1484, 1443, 1385, 1347, 1249, 1138, 1099, 991, 922, 811, 772, 703 cm^{-1}; HRMS (EI) for $C_{24}H_{24}O$: calcd 328.1827, found 328.1824.



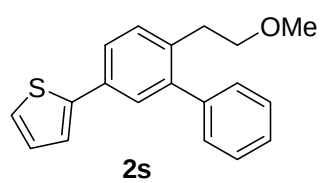
2q

(**2q**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 40:1) afforded the title compound **2q** as a light yellow sticky liquid in 84% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.91 (t, *J* = 7.2 Hz, 2H), 3.24 (s, 3H), 3.47 (t, *J* = 7.2 Hz, 2H), 7.34-7.45 (m, 9H), 7.48-7.53 (m, 3H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 32.7, 58.4, 72.9, 125.7, 127.1, 128.2 (4C), 128.6, 128.8 (2C), 129.1 (2C), 130.2, 133.2, 135.6, 137.7, 139.0, 141.3, 142.8; IR (KBr): 3056, 3026, 2977, 2925, 2871, 2825, 1599, 1575, 1556, 1479, 1444, 1179, 1113, 1093, 1010, 968, 898, 816, 771, 752, 734, 703 cm^{-1}; HRMS (EI) for $C_{21}H_{19}OCl$: calcd 322.1124, found 322.1121.



2r

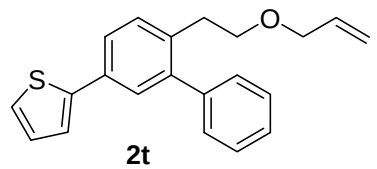
(**2r**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1) afforded the title compound **2r** as a colorless sticky liquid in 77% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.92 (t, *J* = 7.2 Hz, 2H), 3.52 (t, *J* = 7.2 Hz, 2H), 3.88 (dt, *J* = 5.4, 1.2 Hz, 2H), 5.10-5.22 (m, 2H), 5.77-5.90 (m, 1H), 7.33-7.45 (m, 9H), 7.48-7.54 (m, 3H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 33.0, 70.6, 71.6, 116.8, 125.8, 127.1, 128.2 (4C), 128.6, 128.9 (2C), 129.2 (2C), 130.4, 133.2, 134.7, 135.7, 137.7, 139.1, 141.3, 142.9; IR (KBr): 3058, 3025, 2927, 2856, 1599, 1479, 1443, 1381, 1348, 1137, 1094, 1015, 1000, 924, 817, 771, 703 cm^{-1}; HRMS (EI) for $C_{23}H_{21}OCl$: calcd 348.1281, found 348.1285.



2s

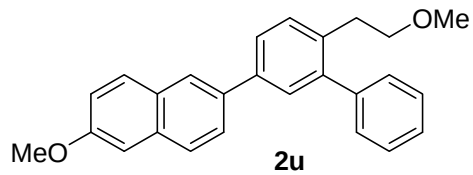
(**2s**). Purification of the crude product by flash chromatography on silica gel (eluent:

petroleum ether/ethyl acetate = 20:1) afforded the title compound **2s** as a brown black sticky liquid in 91% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.86 (t, *J* = 7.2 Hz, 2H), 3.21 (s, 3H), 3.43 (t, *J* = 7.5 Hz, 2H), 7.02 (t, *J* = 5.1 Hz, 1H), 7.20 (d, *J* = 5.1 Hz, 1H), 7.27 (d, *J* = 3.6 Hz, 1H), 7.31-7.43 (m, 6H), 7.47 (d, *J* = 1.8 Hz, 1H), 7.53 (dd, *J* = 7.8, 1.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 32.8, 58.4, 72.9, 122.9, 124.5, 124.8, 127.0, 127.5, 127.9, 128.1 (2C), 129.1 (2C), 130.2, 132.3, 135.4, 141.1, 142.7, 143.9; IR (KBr): 3104, 3059, 2923, 2868, 1599, 1560, 1483, 1442, 1401, 1380, 1188, 1110, 1024, 962, 889, 856, 817, 779, 748, 702 cm^{-1}; HRMS (EI) for $C_{19}H_{18}OS$: calcd 294.1078, found 294.1079.



2t

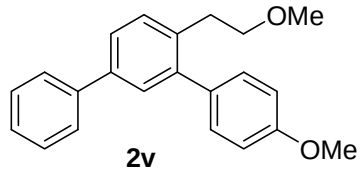
(**2t**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1) afforded the title compound **2t** as a light yellow sticky liquid in 81% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.89 (t, *J* = 7.2 Hz, 2H), 3.49 (t, *J* = 7.2 Hz, 2H), 3.84-3.87 (m, 2H), 5.09-5.20 (m, 2H), 5.76-5.88 (m, 1H), 7.04 (dd, *J* = 5.1, 3.6 Hz, 1H), 7.23 (dd, *J* = 5.1, 0.9Hz, 1H), 7.28 (dd, *J* = 3.9, 0.6 Hz, 1H), 7.33-7.45 (m, 6H), 7.47 (d, *J* = 1.8 Hz, 1H), 7.54 (dd, *J* = 7.8, 2.1 Hz, 1H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 33.0, 70.5, 71.6, 116.7, 122.9, 124.6, 124.8, 127.1, 127.5, 127.9, 128.1 (2C), 129.2 (2C), 130.3, 132.3, 134.7, 135.5, 141.2, 142.8, 144.0; IR (KBr): 3071, 3023, 2918, 2856, 1601, 1485, 1442, 1434, 1401, 1347, 1267, 1240, 1209, 1136, 1097, 990, 924, 891, 854, 818, 771, 701 cm^{-1}; HRMS (EI) for $C_{21}H_{20}OS$: calcd 320.1235, found 320.1237.



2u

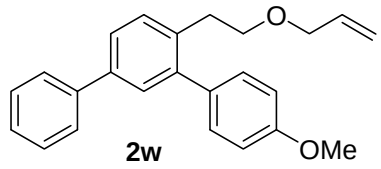
(**2u**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20:1) afforded the title compound **2u** as a white solid in

96% yield. M.p.: 94-95 ^{o}C; ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.93 (t, *J* = 7.5 Hz, 2H), 3.25 (s, 3H), 3.48 (t, *J* = 7.2 Hz, 2H), 3.89 (s, 3H), 7.13-7.16 (m, 2H), 7.36-7.46 (m, 6H), 7.58 (s, 1H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.70-7.78 (m, 3H), 7.98 (s, 1H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 32.8, 55.2, 58.4, 73.0, 105.4, 119.1, 125.3, 125.8, 126.0, 127.0, 127.2, 128.1 (2C), 128.8, 129.1, 129.2 (2C), 129.6, 130.2, 133.7, 134.9, 135.7, 138.9, 141.5, 142.7, 157.6; IR (KBr): 3052, 2993, 2954, 2920, 2871, 2817, 2807, 1626, 1606, 1490, 1388, 1239, 1201, 1168, 1114, 1029, 1016, 969, 893, 859, 837, 821, 775, 703 cm^{-1}; HRMS (EI) for $C_{26}H_{24}O_2$: calcd 368.1776, found 368.1777.



2v

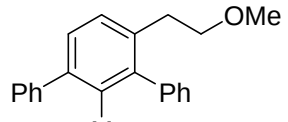
(**2v**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 40:1) afforded the title compound **2v** as a colorless sticky liquid in 75% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.92 (t, *J* = 7.5 Hz, 2H), 3.25 (s, 3H), 3.48 (t, *J* = 7.2 Hz, 2H), 3.83 (s, 3H), 6.93-6.97 (m, 2H), 7.26-7.33 (m, 3H), 7.37-7.46 (m, 4H), 7.51 (dd, *J* = 7.8, 2.4 Hz, 1H), 7.58-7.61 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 32.8, 55.2, 58.5, 73.0, 113.5 (2C), 125.7, 127.0 (2C), 127.1, 128.7 (2C), 129.0, 130.1, 130.2 (2C), 133.8, 135.3, 139.0, 140.7, 142.3, 158.6; IR (KBr): 3056, 3030, 2930, 2871, 2834, 1610, 1574, 1514, 1481, 1463, 1384, 1290, 1248, 1177, 1112, 1030, 835, 763, 699, 579 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O_2$: calcd 318.1620, found 318.1622.



2w

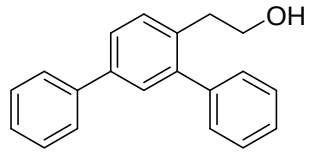
(**2w**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 50:1) afforded the title compound **2w** as a colorless sticky liquid in 67% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 2.94 (t, *J* = 7.5 Hz, 2H), 3.53 (t, *J* = 7.5 Hz, 2H), 3.84 (s, 3H), 3.89 (dt, *J* = 4.2, 1.2 Hz, 2H), 5.10-5.23 (m, 2H), 5.78-5.89

(m, 1H), 6.94-6.98 (m, 2H), 7.27-7.34 (m, 3H), 7.38-7.45 (m, 4H), 7.52 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.58-7.61 (m, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 33.0, 55.3, 70.7, 71.6, 113.5 (2C), 116.7, 125.7, 127.0 (2C), 127.1, 128.7 (2C), 129.0, 130.2, 130.3 (2C), 133.9, 134.7, 135.4, 139.0, 140.7, 142.3, 158.6; IR (KBr): 3058, 3030, 2955, 2932, 2907, 2854, 2830, 1610, 1574, 1515, 1481, 1463, 1290, 1245, 1177, 1097, 1030, 924, 834, 762, 698 cm^{-1}; HRMS (EI) for $C_{24}H_{24}O_2$: calcd 344.1776, found 344.1778.



2x

(**2x**). Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100:1-50:1) afforded the title compound **2x** as a white solid in 66% (form ***Z*-1j**) or 67% (from ***E*-1j**) yield. M.p. 60-62 ^{o}C; ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.88 (s, 3H), 2.66 (t, *J* = 7.2 Hz, 2H), 3.22 (s, 3H), 3.43 (t, *J* = 7.2 Hz, 2H), 7.18-7.23 (m, 4H), 7.31-7.42 (m, 8H); ^{13}C NMR ($CDCl_3$, Me_4Si, 100 MHz): δ 18.9, 33.7, 58.3, 73.0, 126.5, 126.6, 126.8, 128.0 (2C), 128.4 (2C), 128.9, 129.3 (2C), 129.4 (2C), 133.7, 135.6, 140.4, 140.9, 142.4, 142.6; IR (KBr): 3060, 3022, 2970, 2958, 2928, 2864, 2827, 1600, 1469, 1442, 1405, 1382, 1184, 1111, 1101, 1070, 1023, 1003, 970, 925, 833, 763, 702 cm^{-1}; HRMS (EI) for $C_{22}H_{22}O$: calcd 302.1671, found 302.1669.



2-(1,1',3',1'')terphenyl-4'-yl-ethanol: Purification of the crude product by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) afforded the title compound as a brown sticky liquid in 15% yield. ^{1}H NMR ($CDCl_3$, Me_4Si, 300 MHz): δ 1.29 (t, *J* = 6.0 Hz, 1H), 2.93 (t, *J* = 6.9 Hz, 2H), 3.69-3.76 (m, 2H), 7.31-7.49 (m, 10H), 7.55-7.63 (m, 3H); ^{13}C NMR ($CDCl_3$, Me_4Si, 75 MHz): δ 35.9, 63.2, 126.1, 127.0 (2C), 127.1, 127.3, 128.2 (2C), 128.8 (2C), 129.1, 129.2 (2C), 130.3, 134.7, 139.2, 140.6, 141.5, 142.9; IR (KBr): 3569, 3388, 3057, 3027, 2933, 2877, 1599, 1479, 1442, 1390, 1043, 895,

830, 762, 700 cm^{-1}; HRMS (EI) for $C_{20}H_{18}O$: calcd 274.1358, found 274.1360.

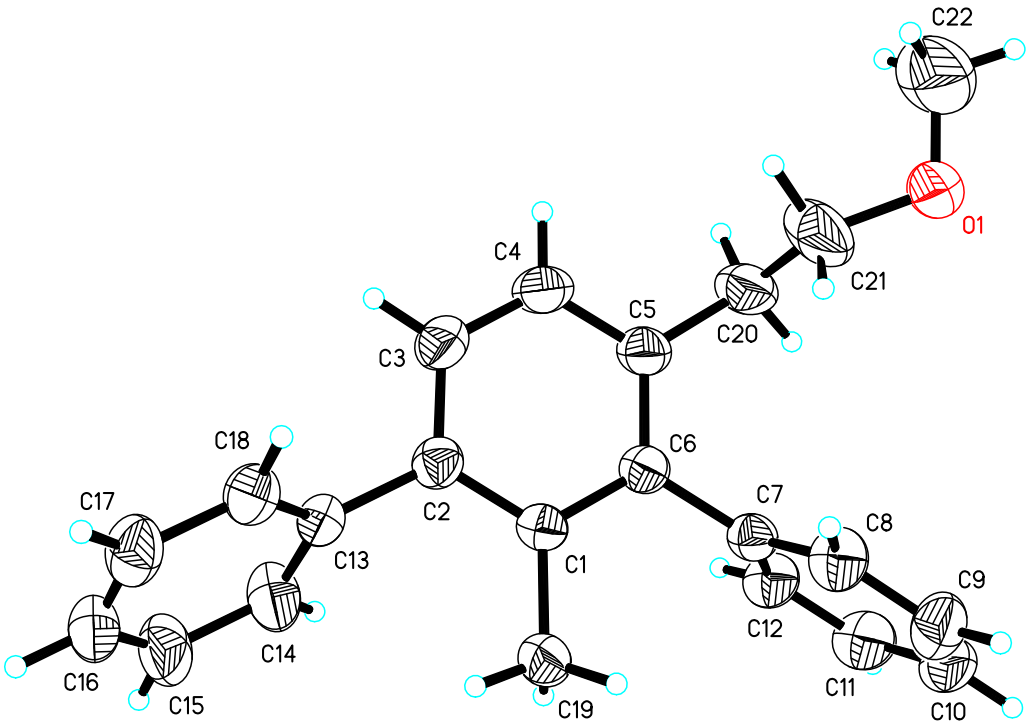
References:

(1) Braunstein, P.; Lehner, H.; Matt, D. *Inorg. Synth.* **1990**, *27*, 218.

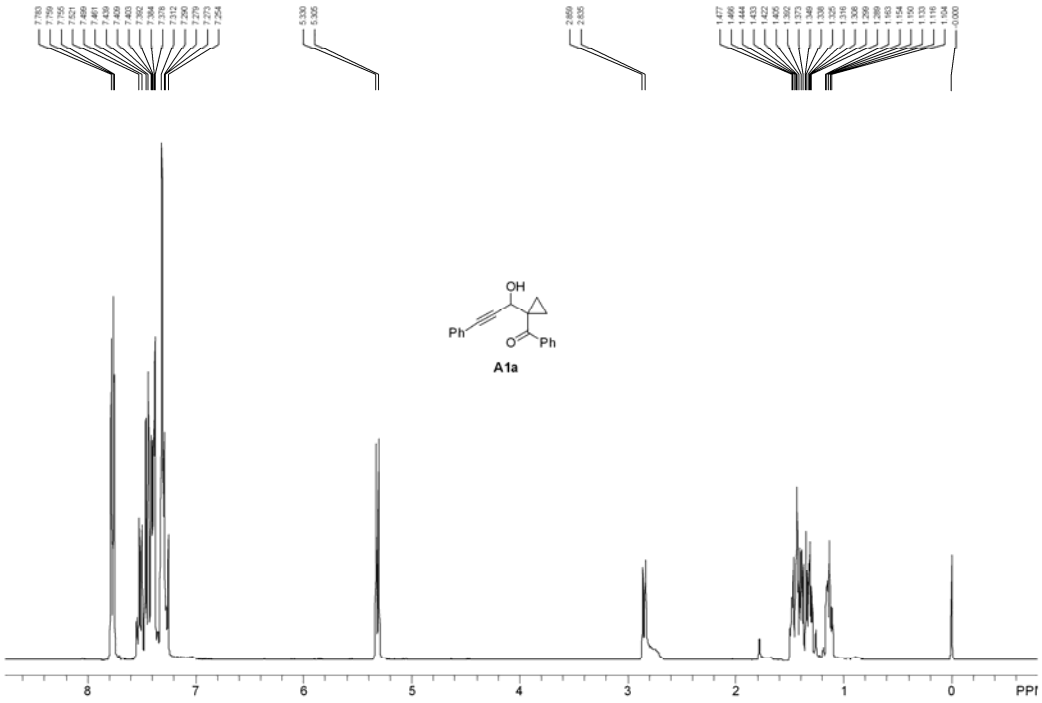
(2) Mezailles, N.; Ricard, L.; Gagosz, F. *Org. Lett.* **2005**, *7*, 4133. b) Vij, A.; Zheng, Y. Y.; Kirchmeier, R. L.; Shreeve, J. M. *Inorg. Chem.* **1994**, *33*, 3281.

(3) *Purification of Laboratory Chemicals*, Fourth edition, Armarego, W. L. F.; Perrin, D. D. Butterworth Heinemann, 1997.

(4) Fitjer, L.; Quabeck, U.; *Synth. Commun.* **1985**, *15*, 855-864.



X-ray crystal structure of compound **2x**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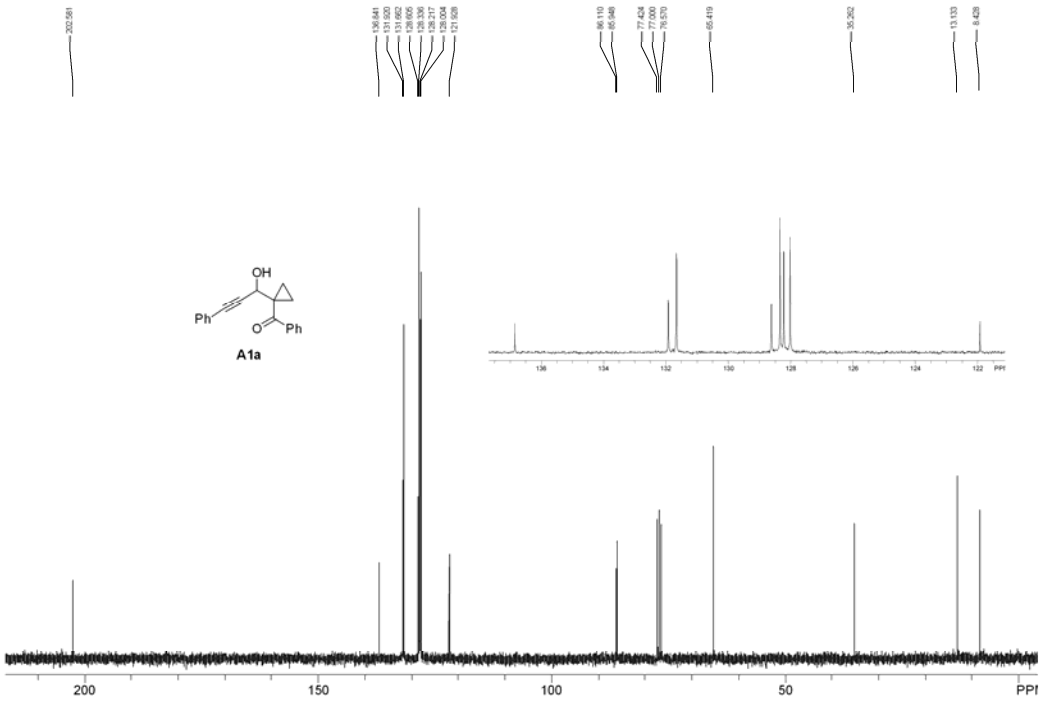
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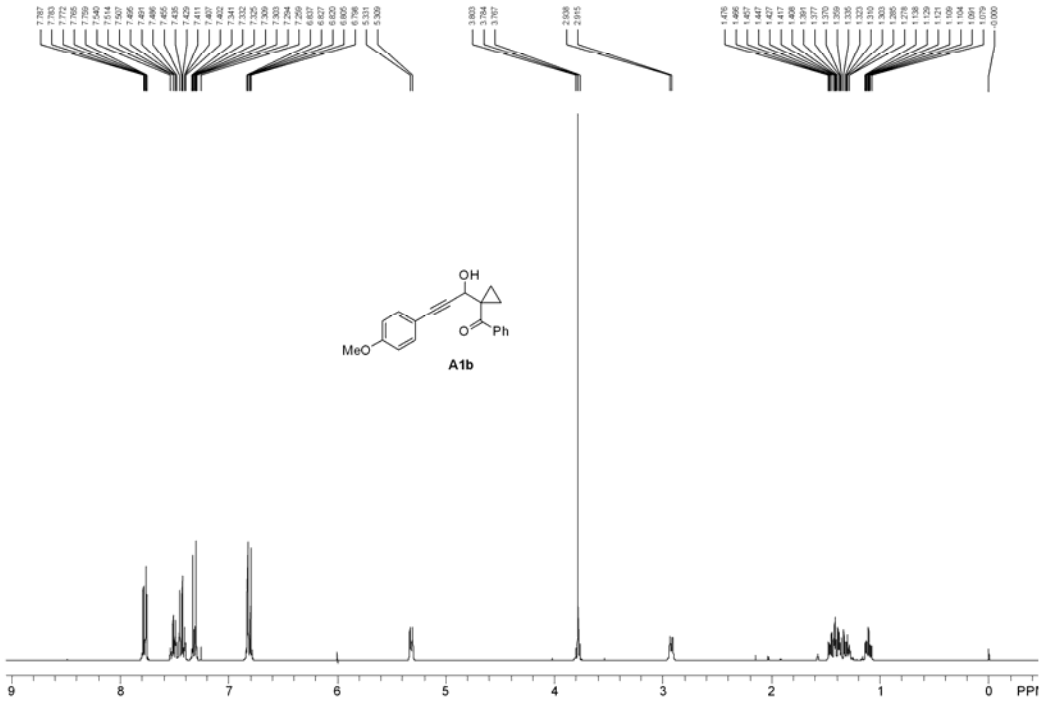
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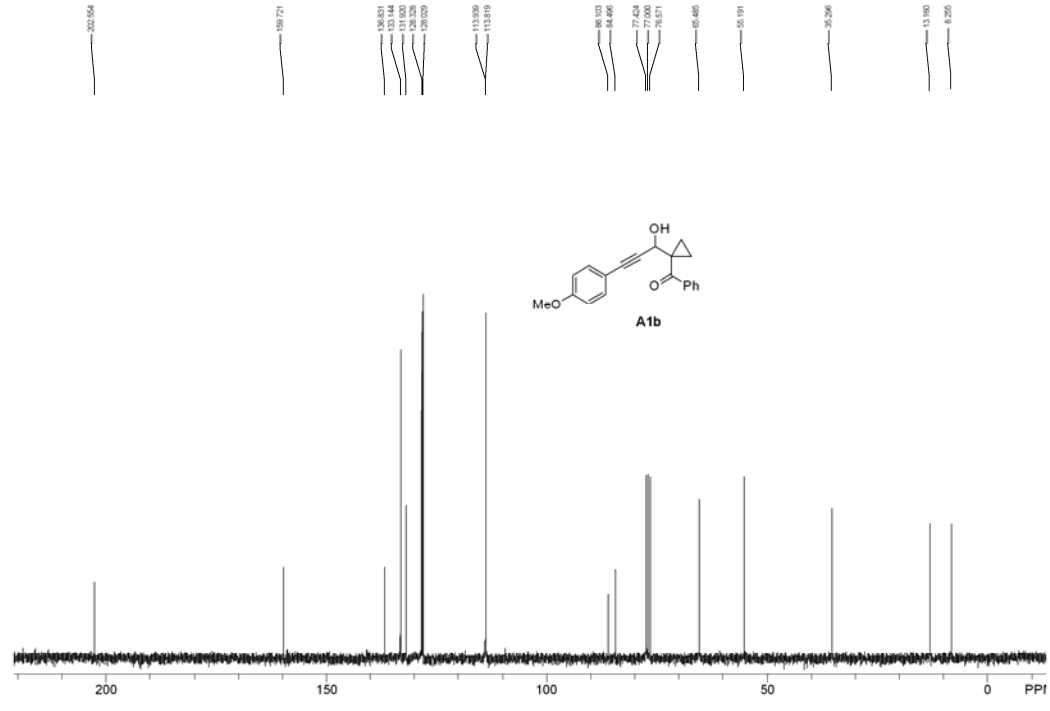
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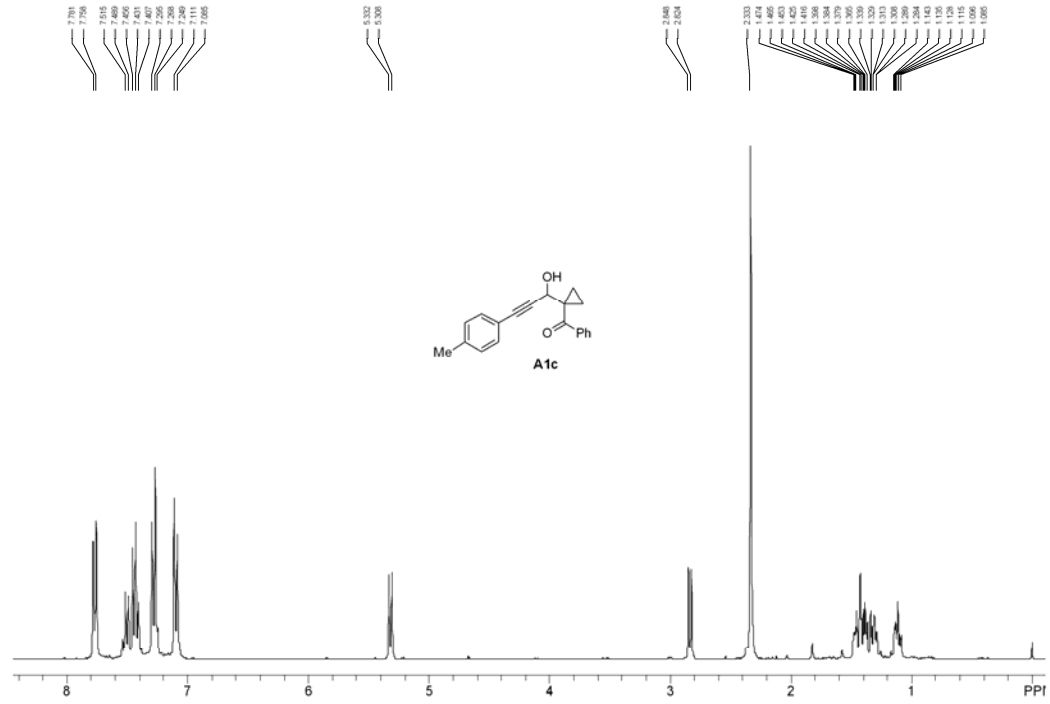
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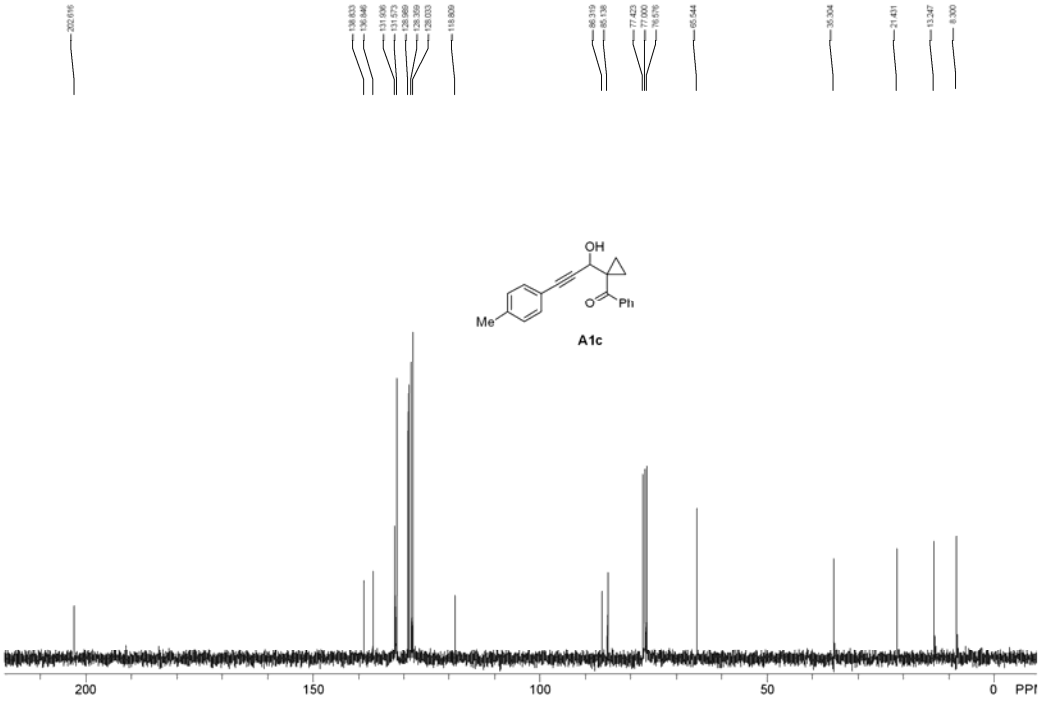
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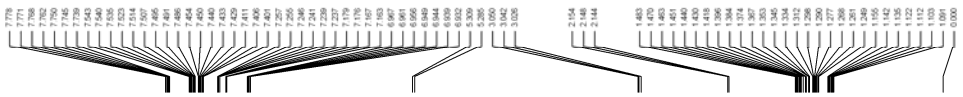

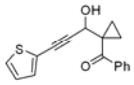

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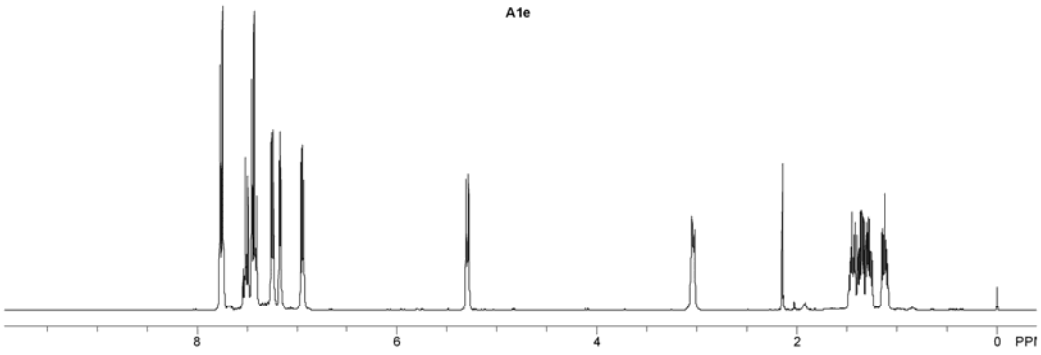

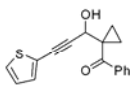

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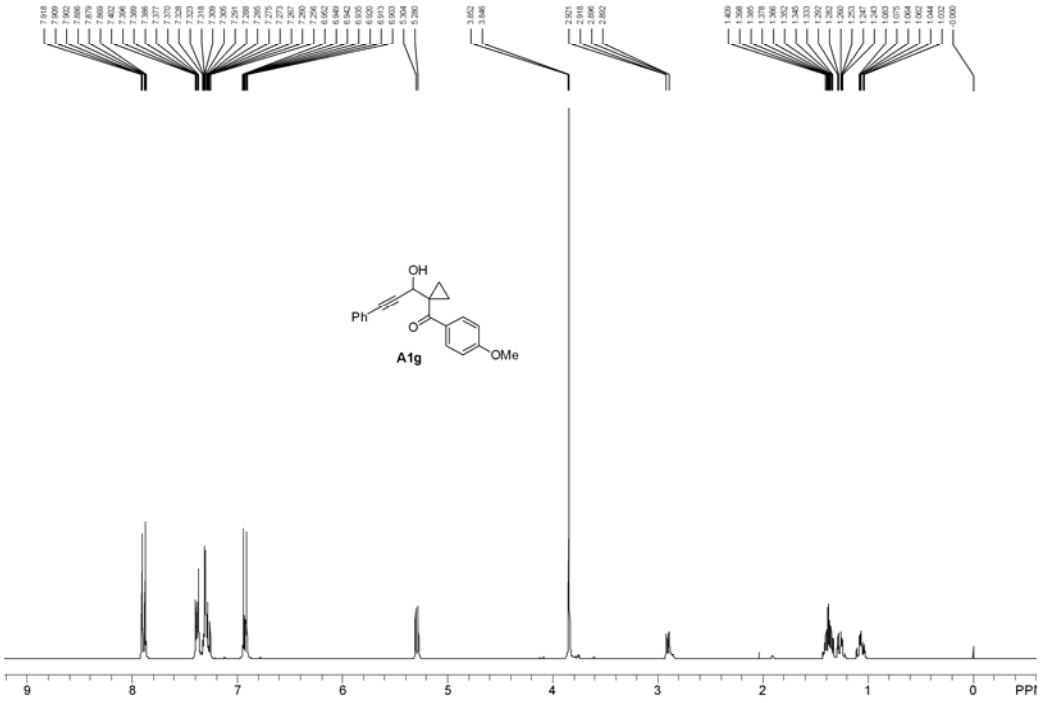
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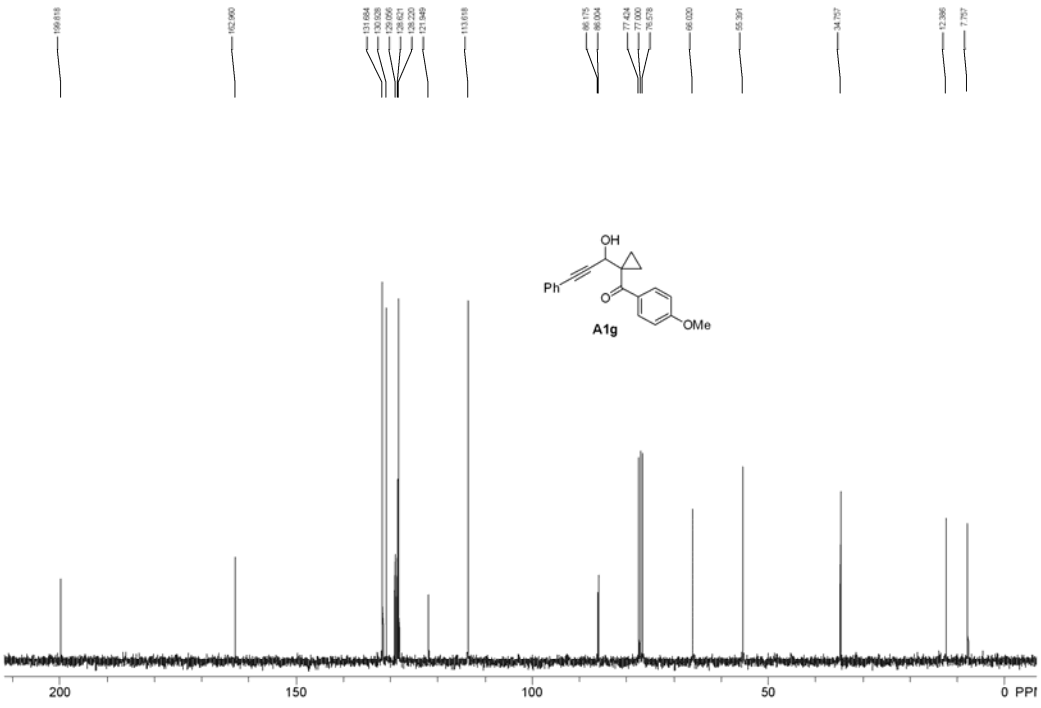
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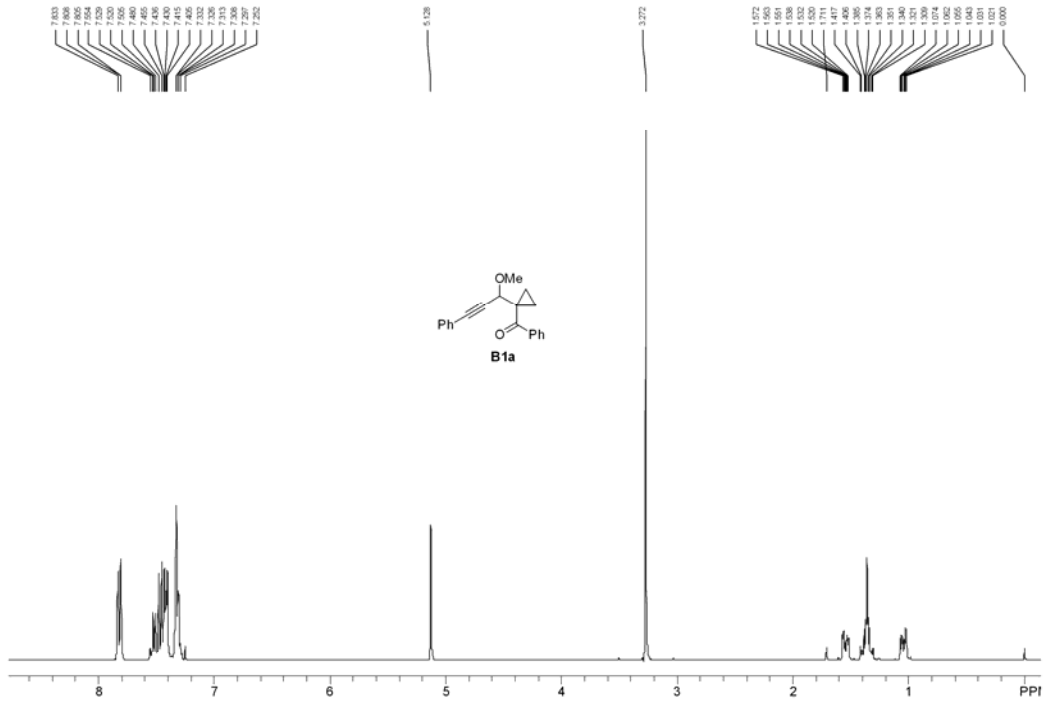
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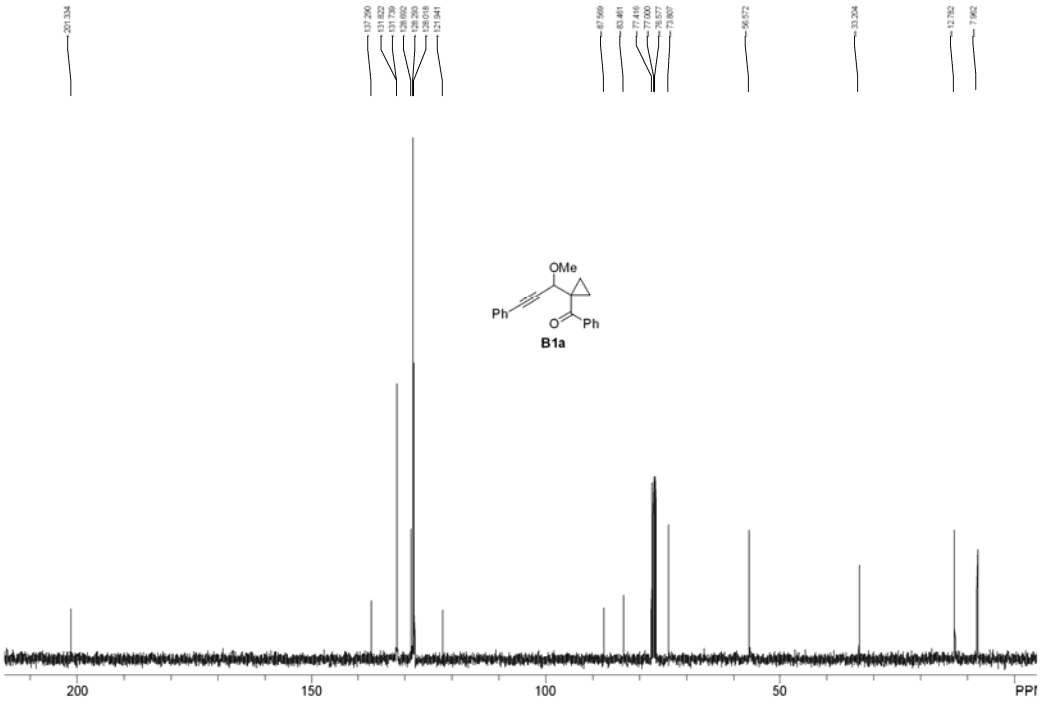
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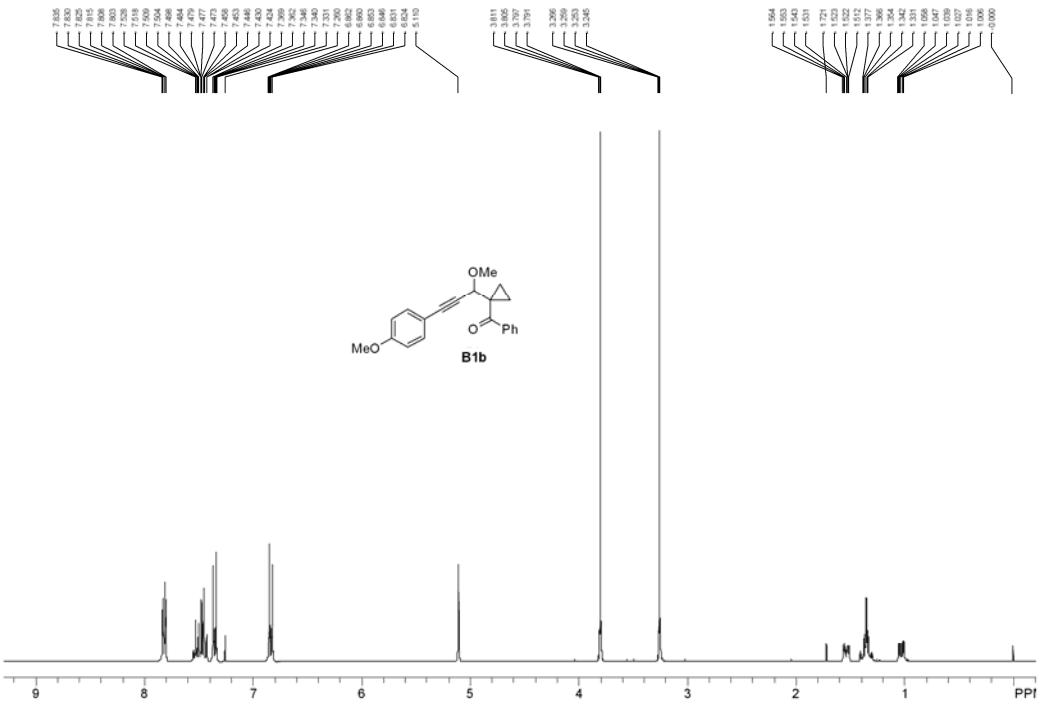
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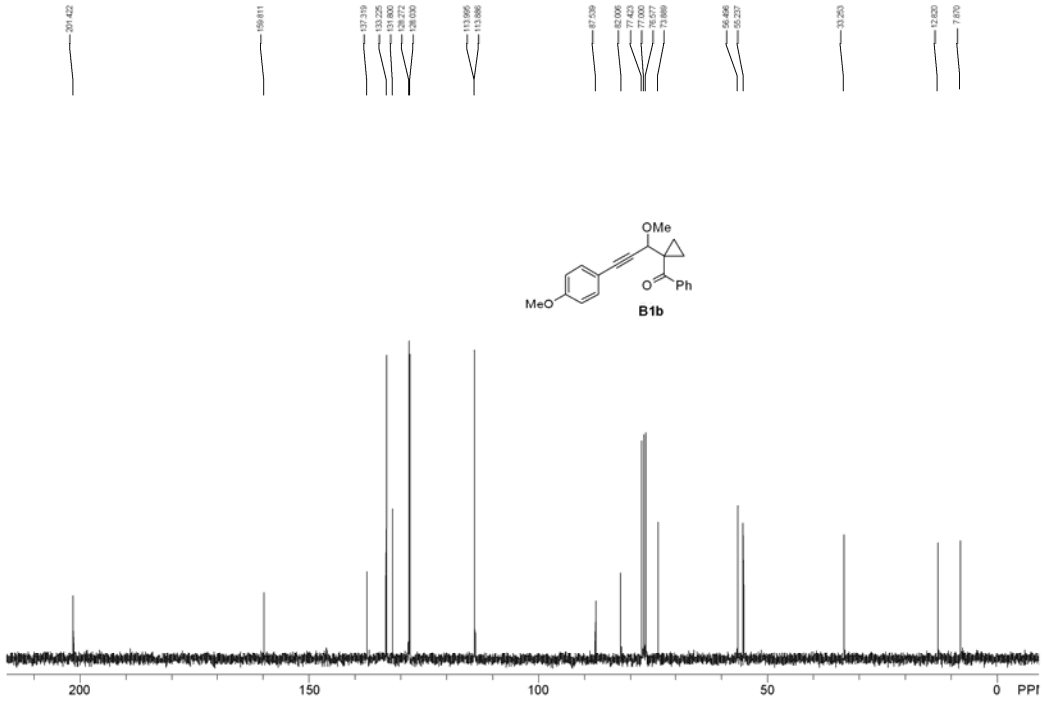
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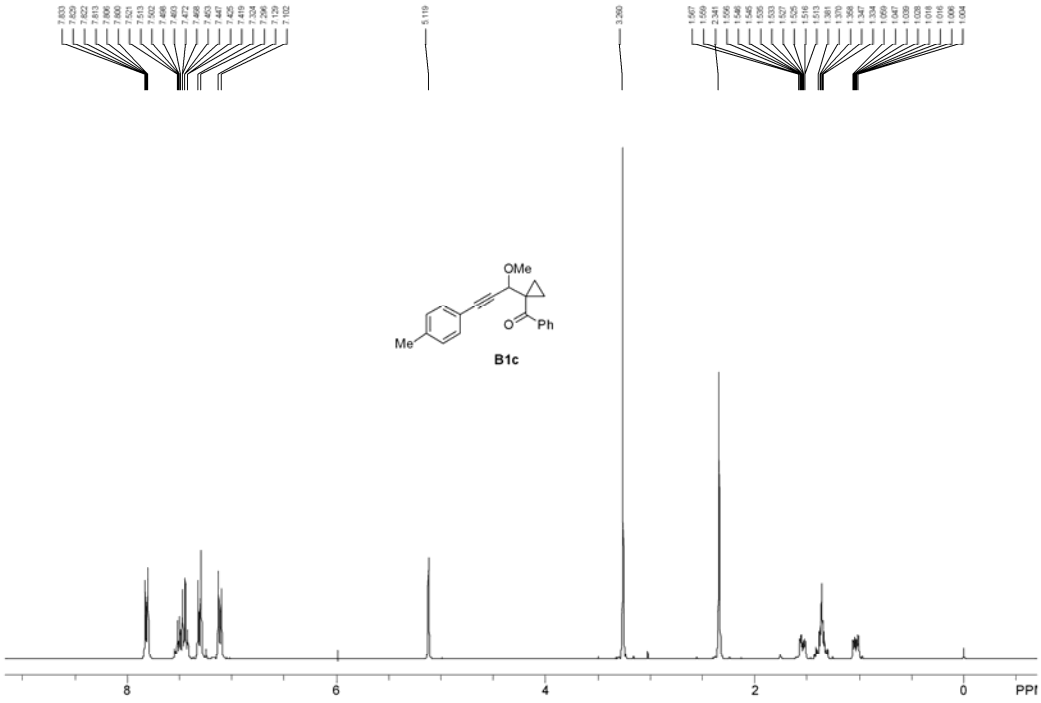
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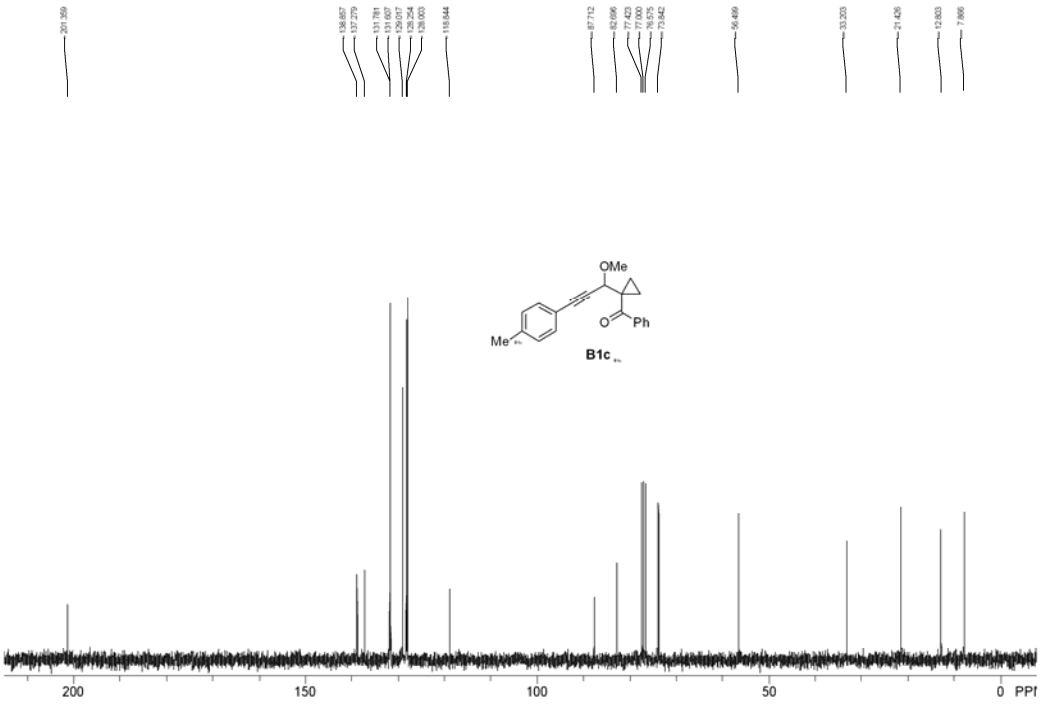
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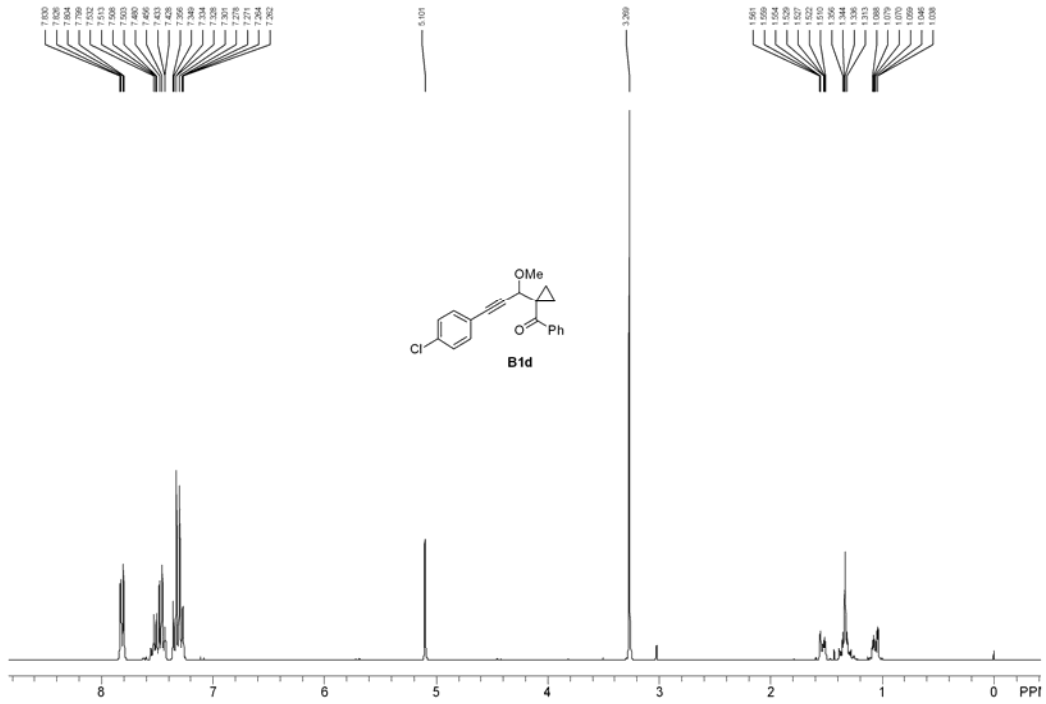
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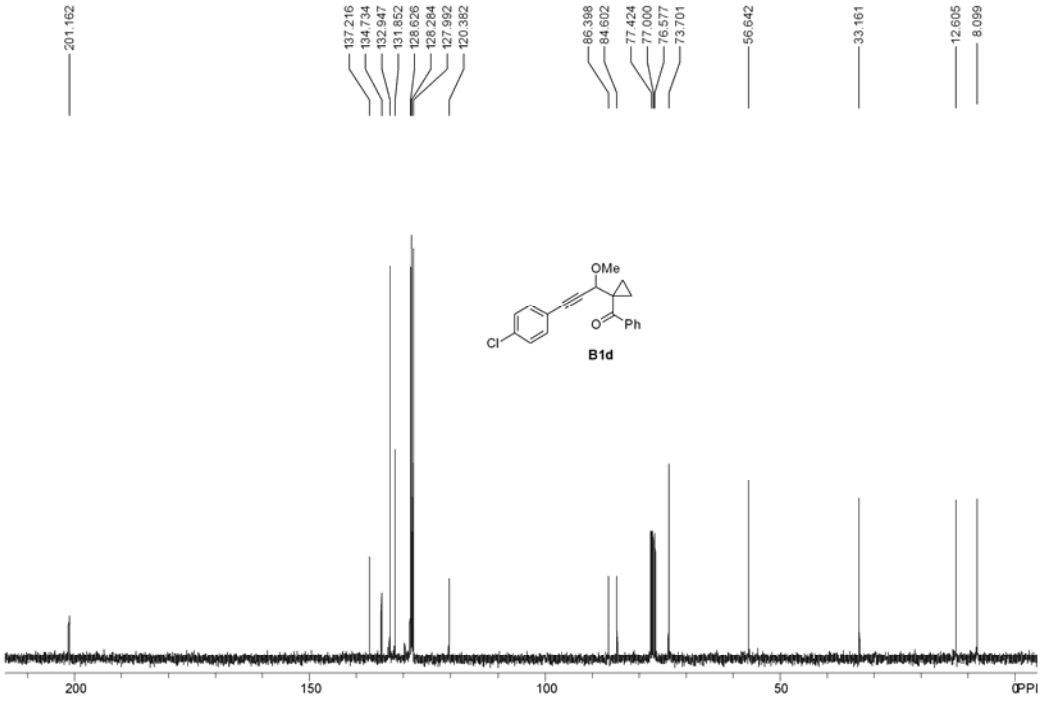
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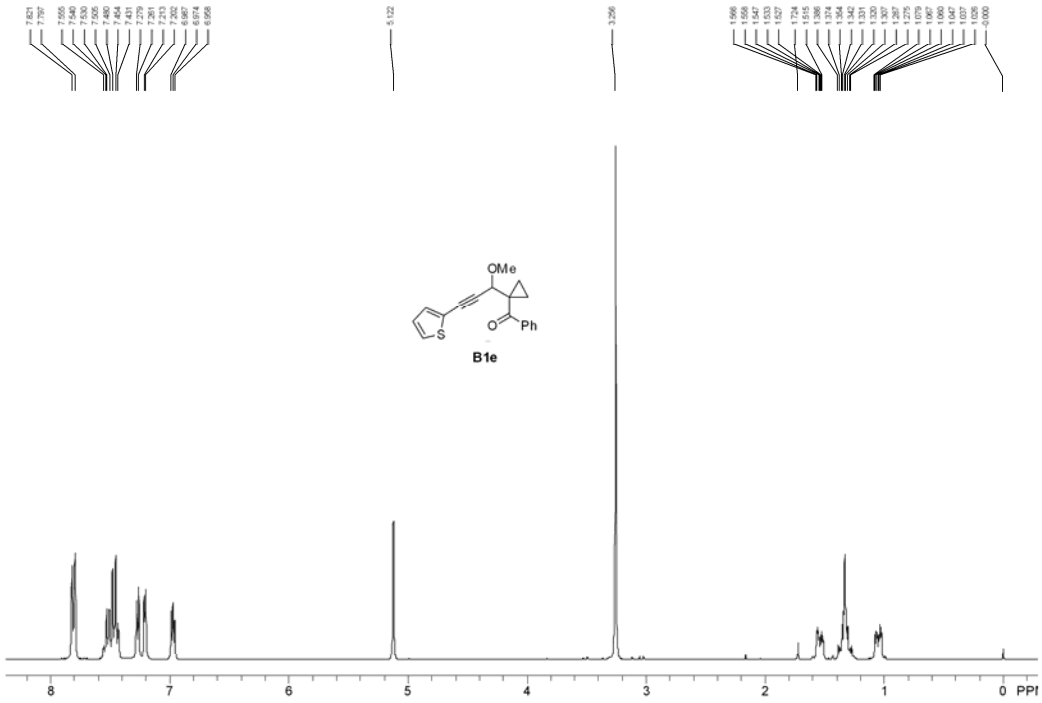
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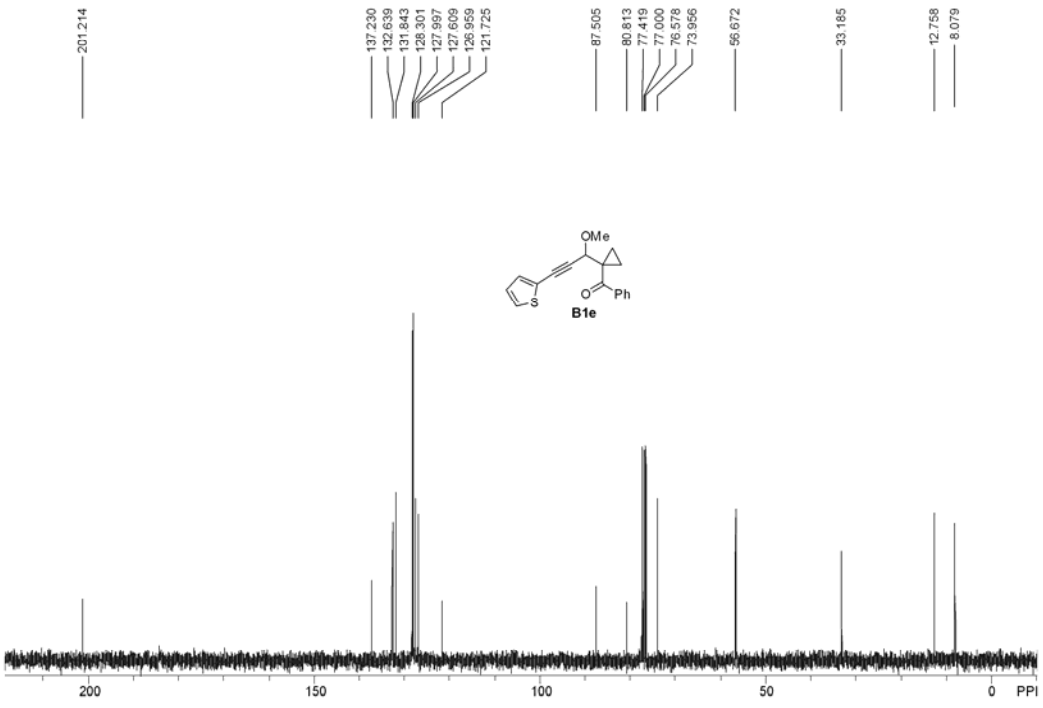
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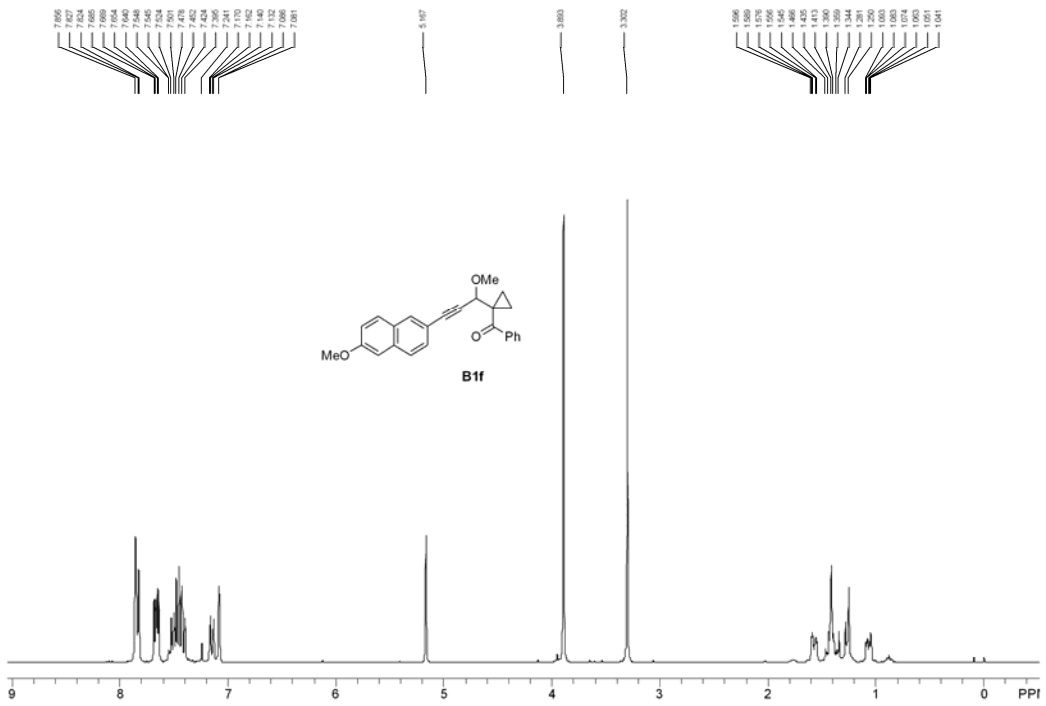
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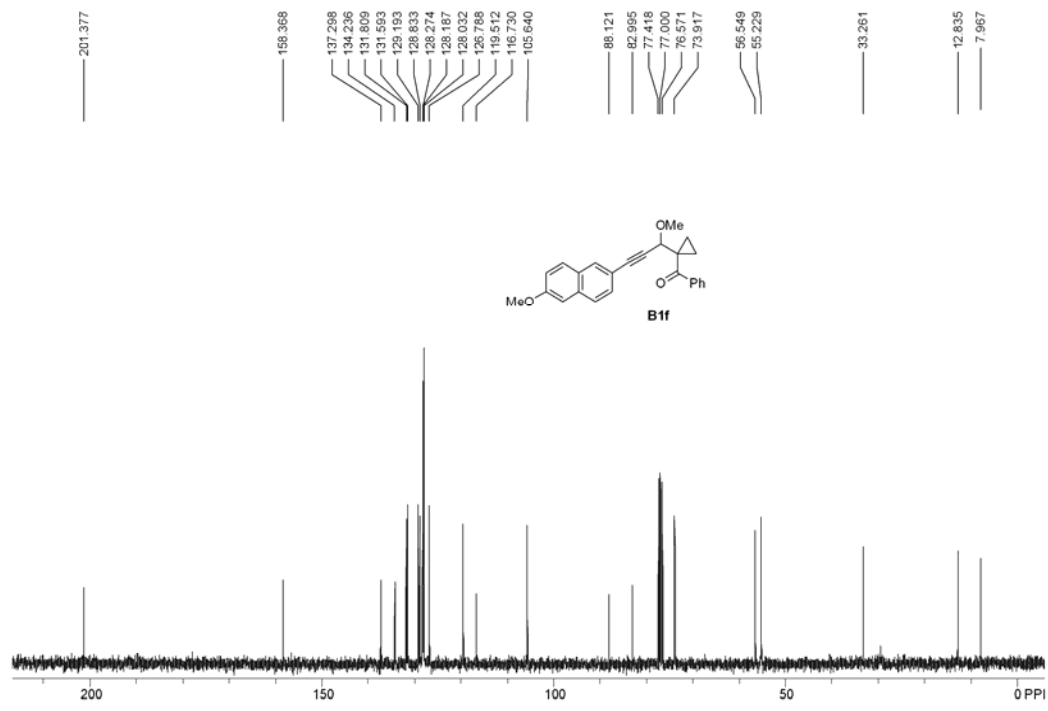
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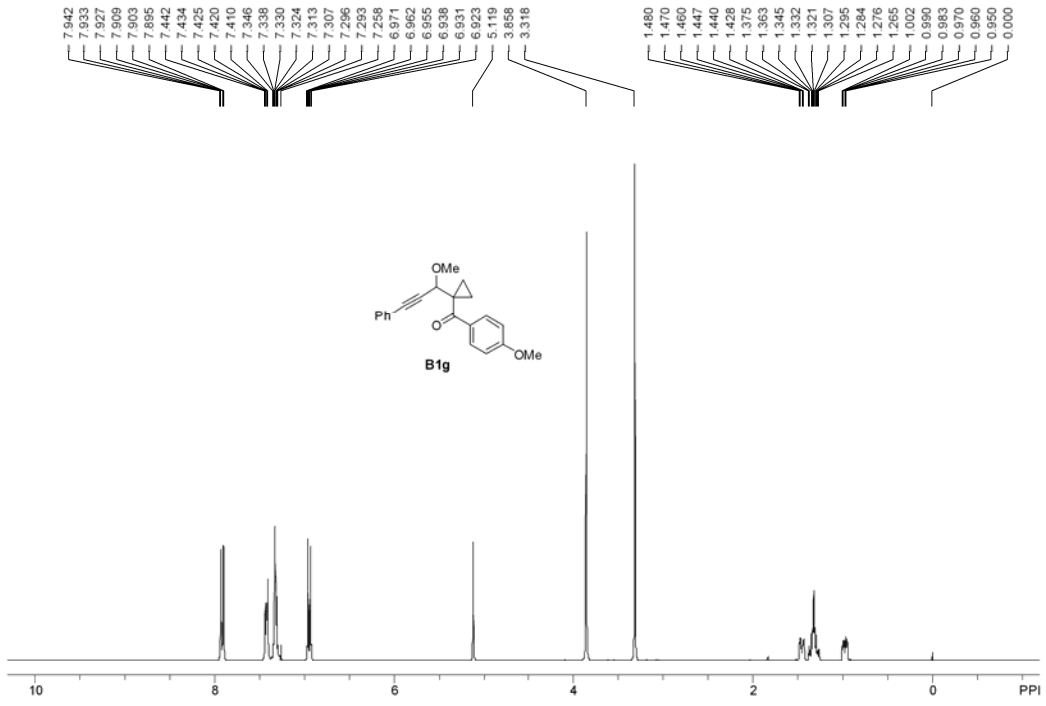
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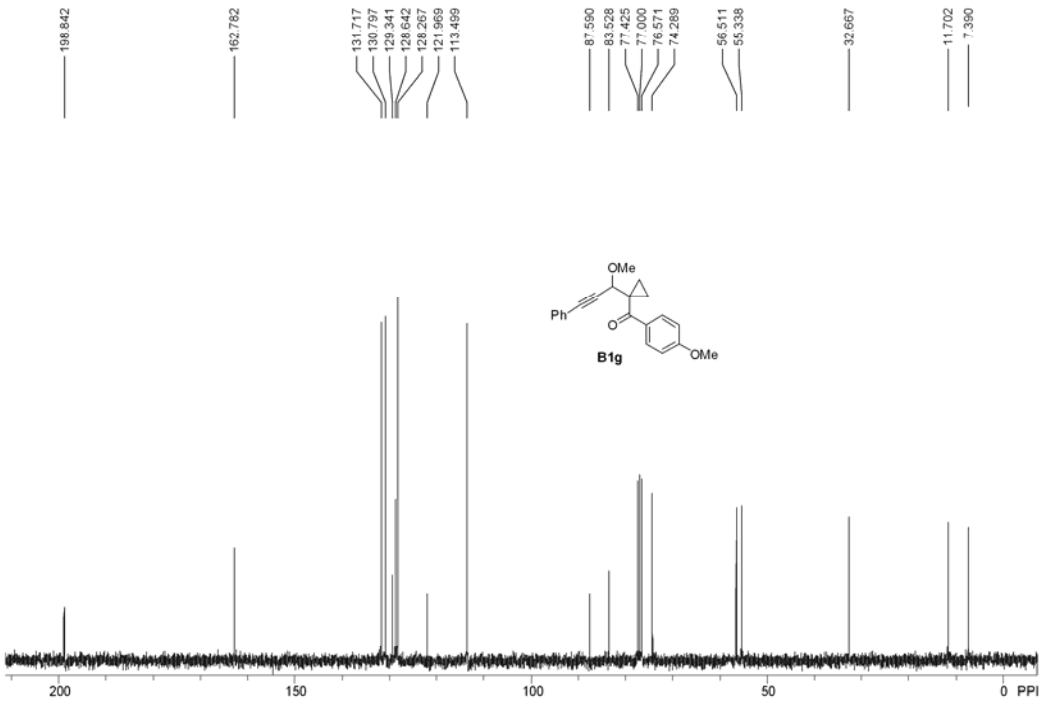
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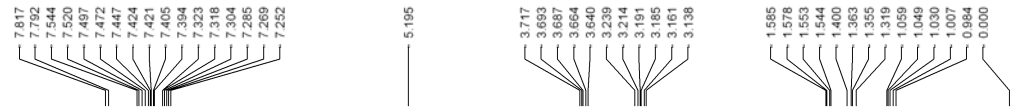
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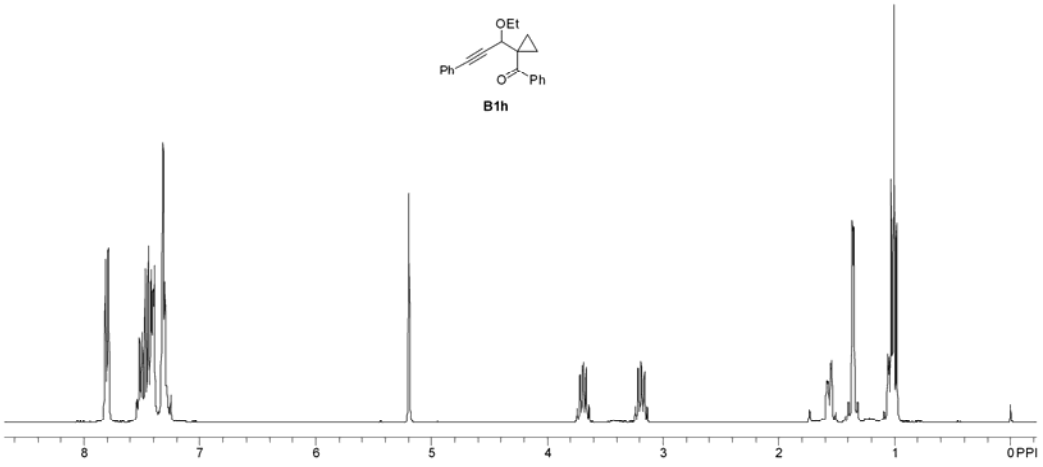
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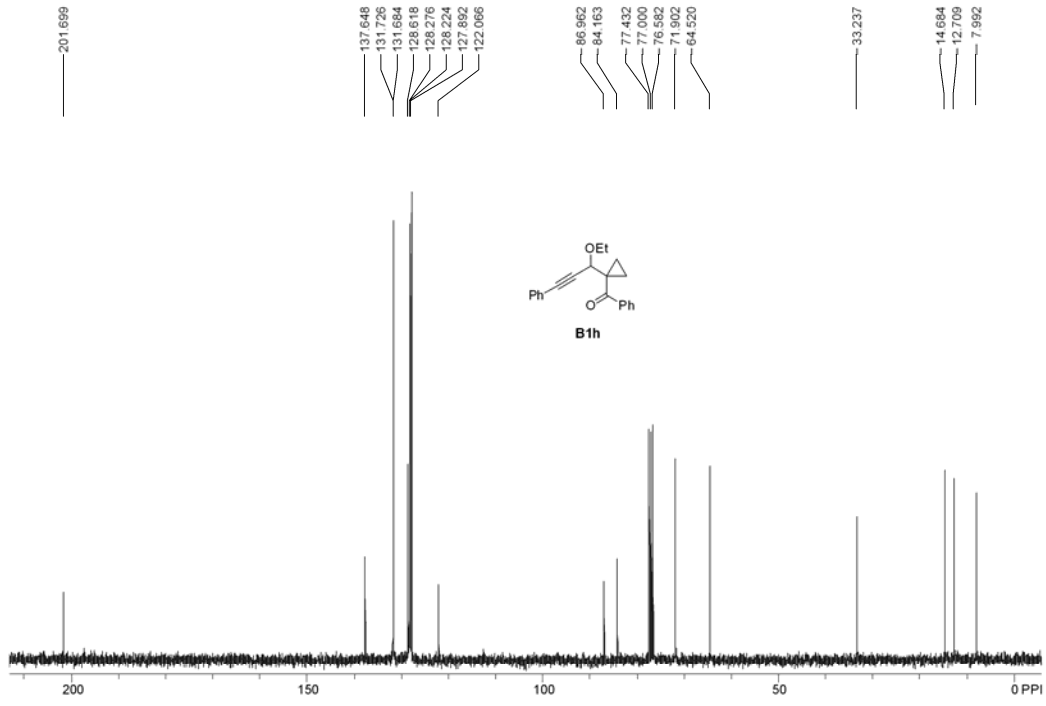
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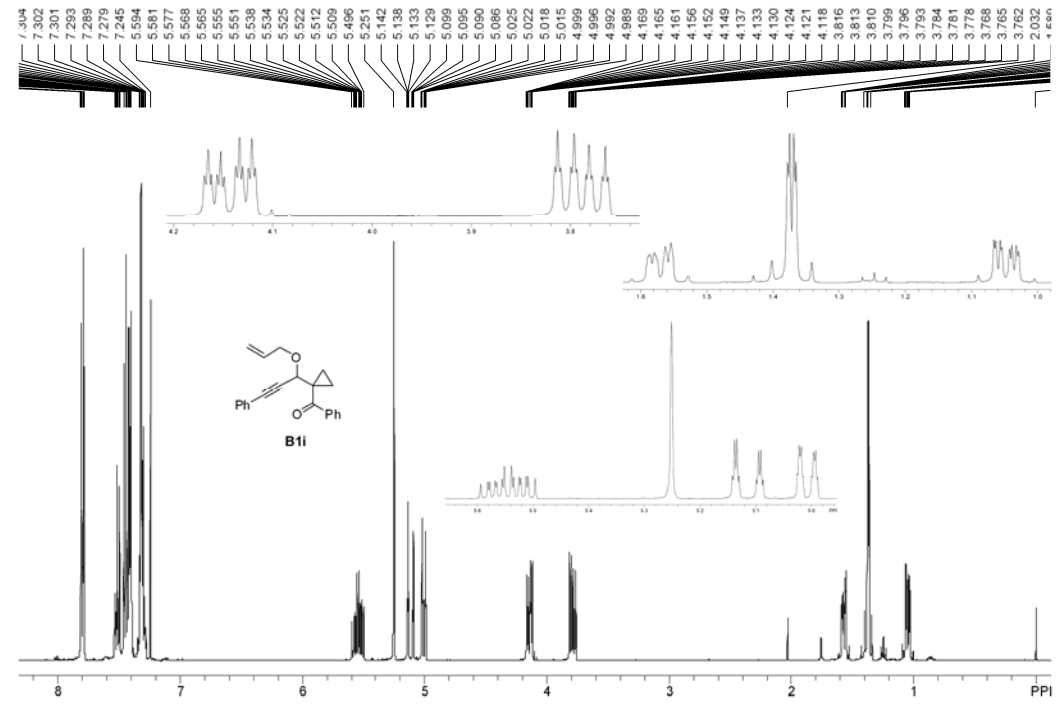
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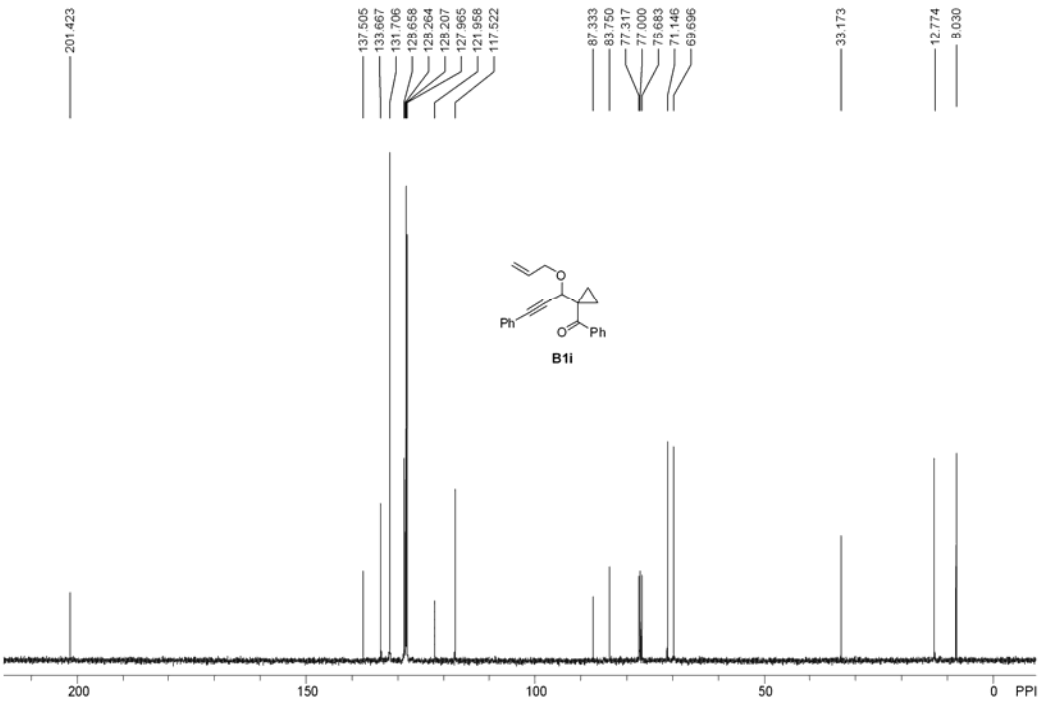
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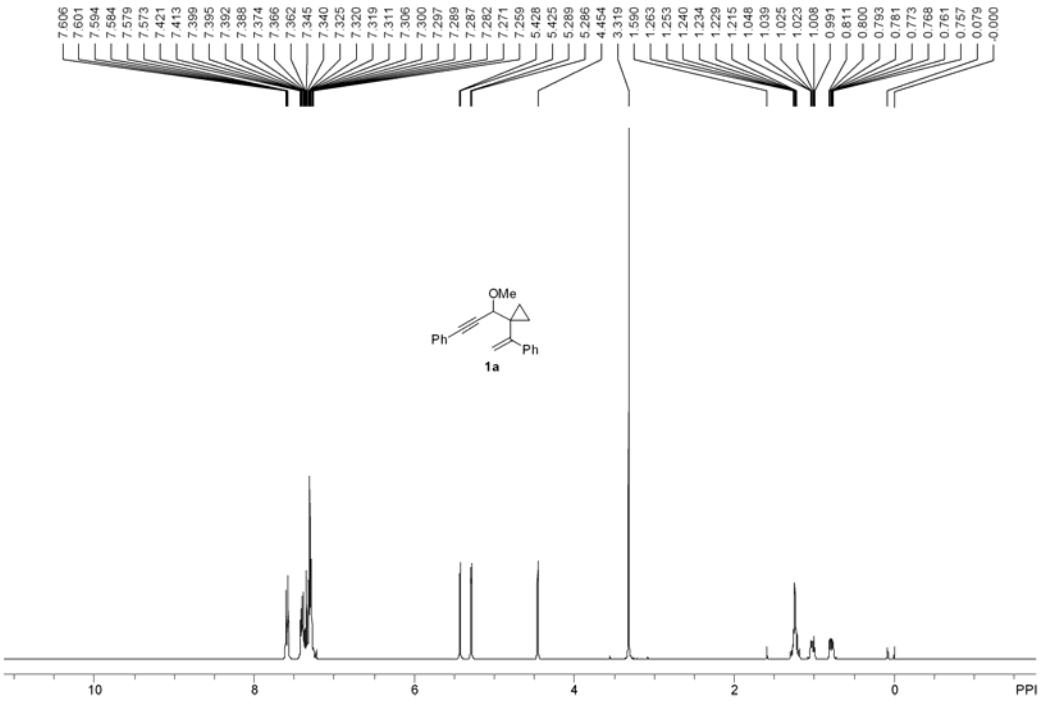
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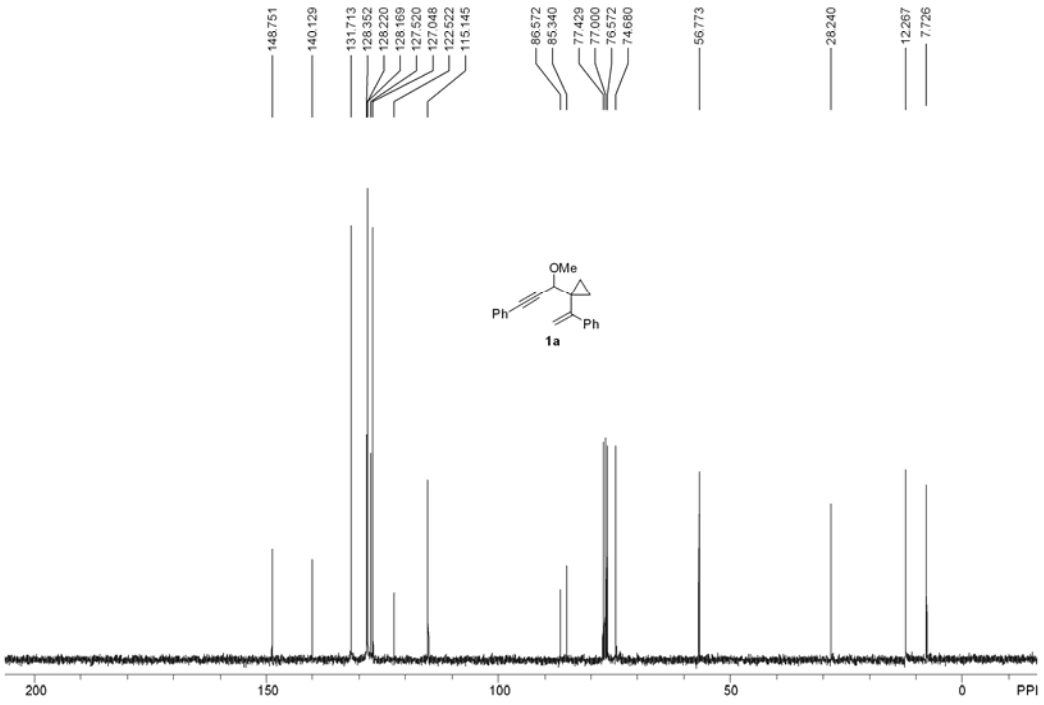
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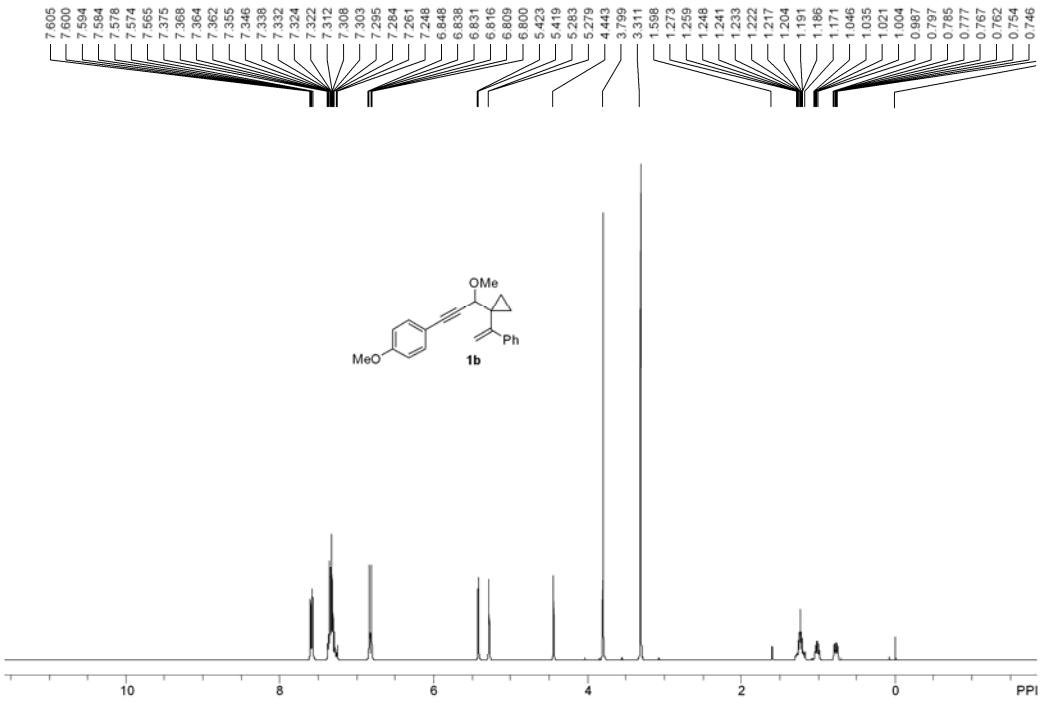
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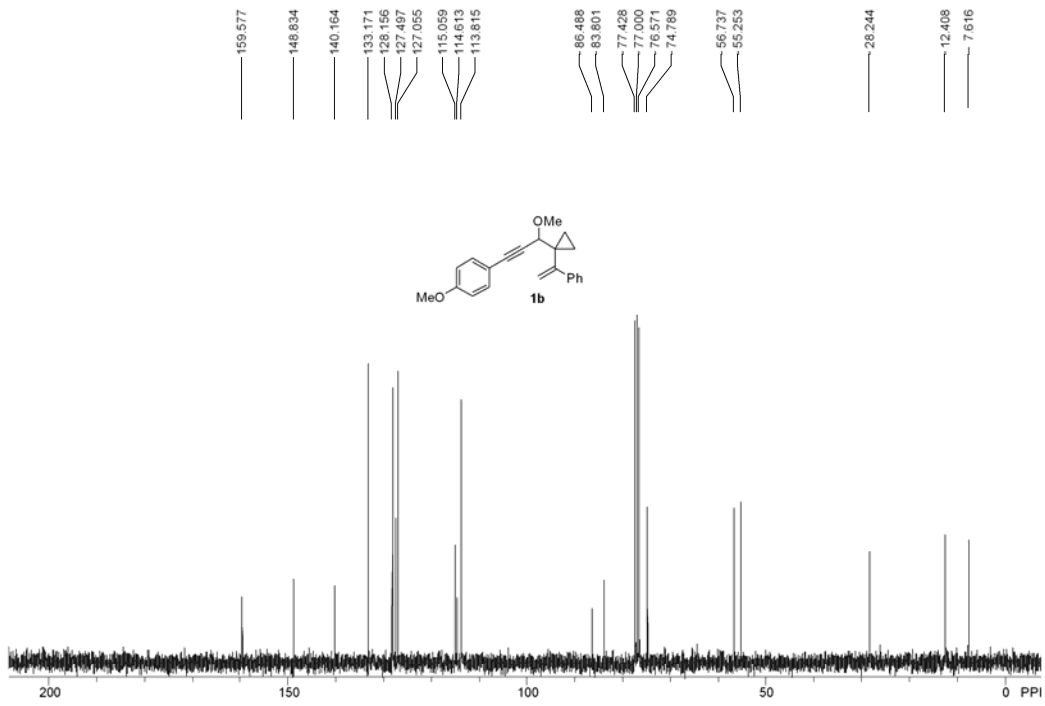
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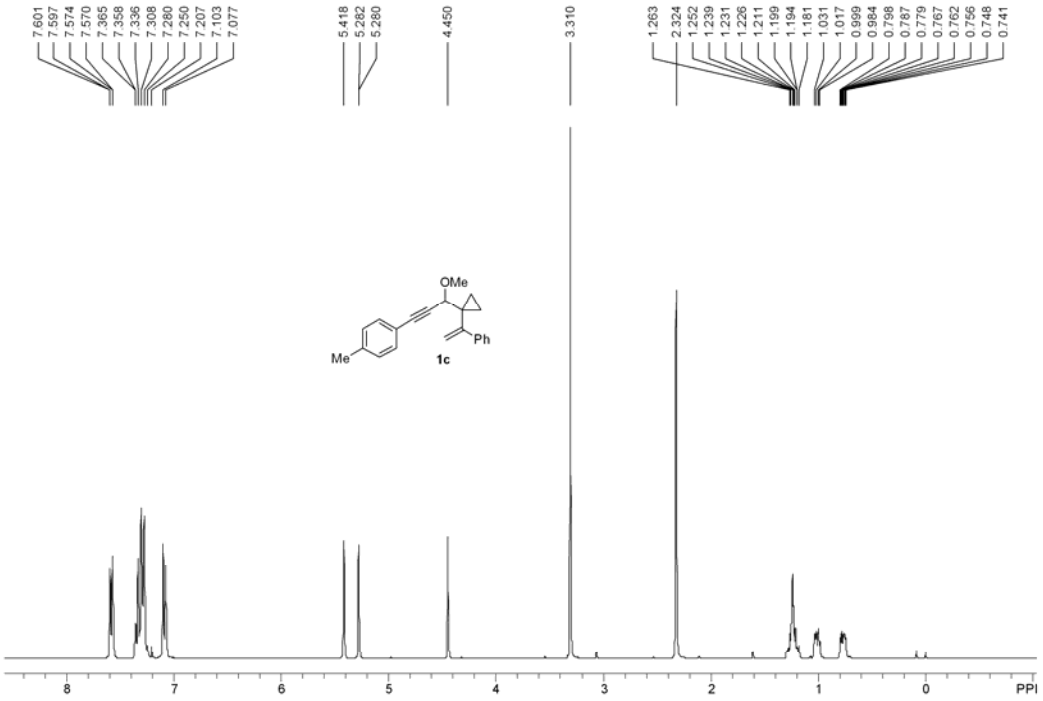
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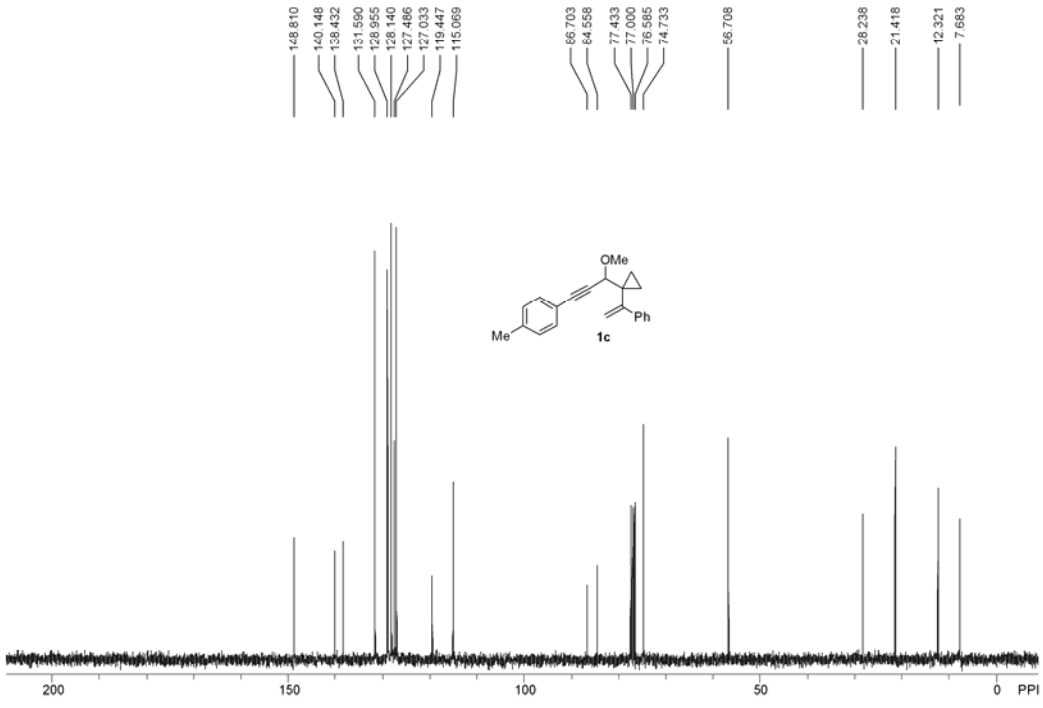
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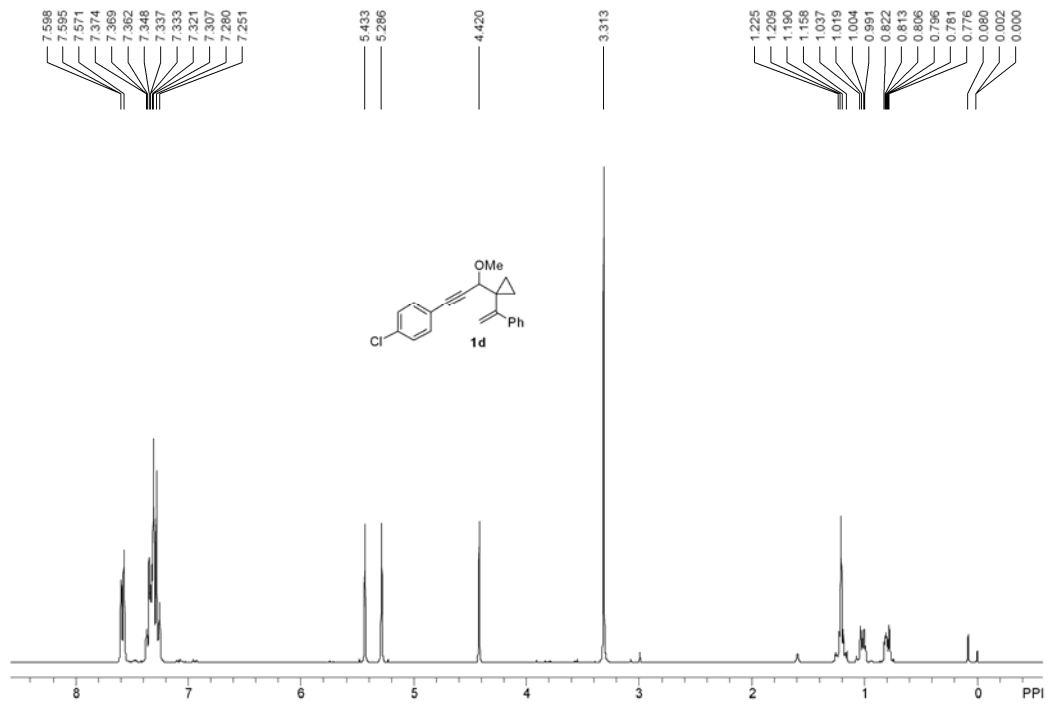
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7.251
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1.225
1.209
1.190
1.158
1.037
1.019
1.004
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0.822
0.813
0.806
0.796
0.781
0.776
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Cl
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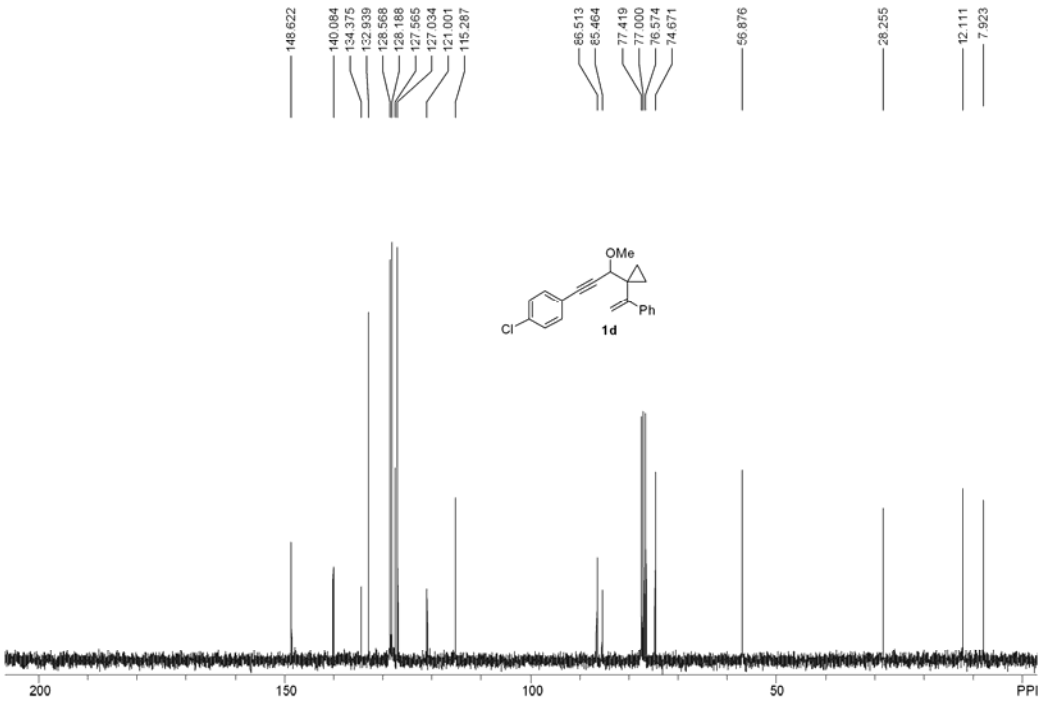
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Ph
Cl
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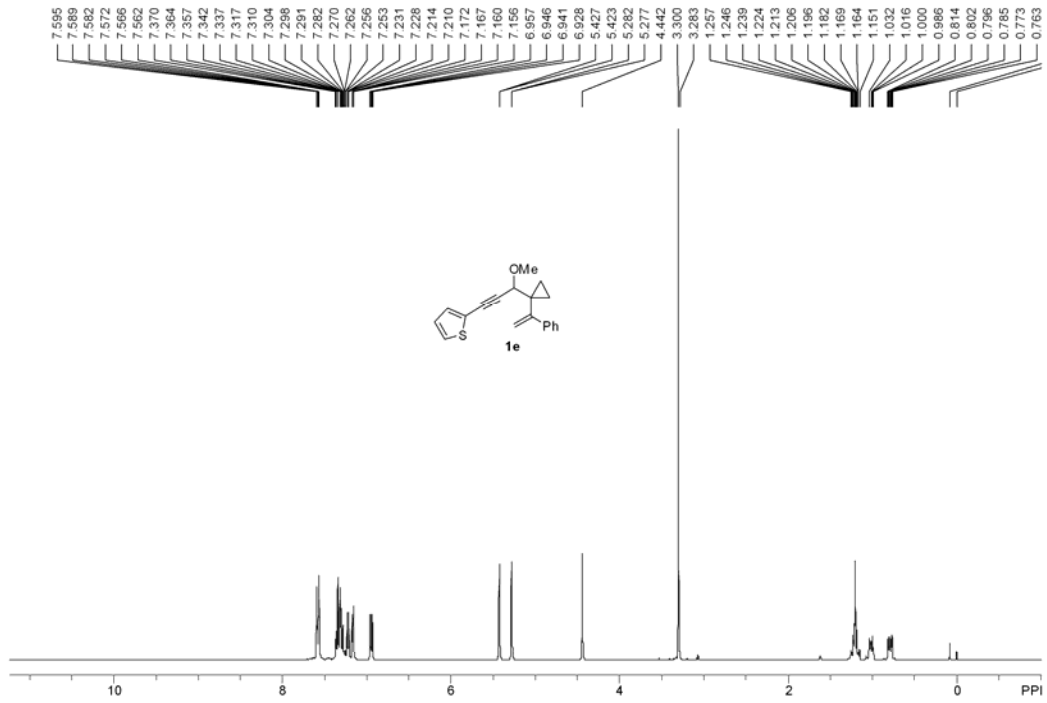
200
150
100
50
PPI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7.304
7.298
7.291
7.282
7.270
7.262
7.256
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7.210
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7.167
7.160
7.156
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6.928
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1.246
1.239
1.224
1.213
1.206
1.196
1.182
1.169
1.164
1.151
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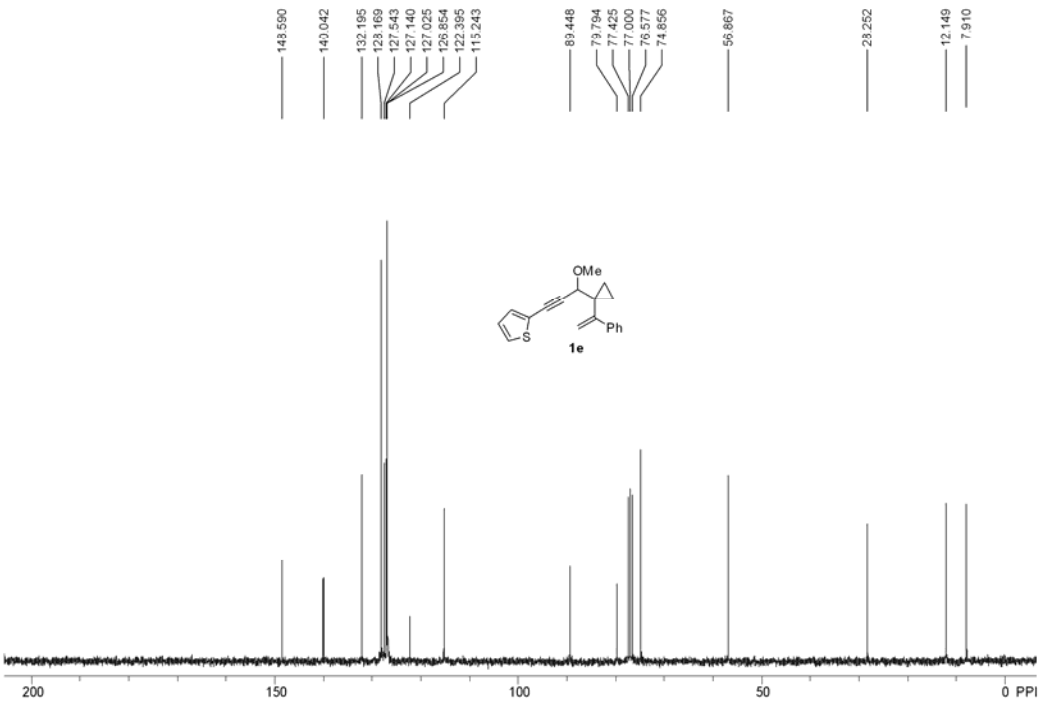
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127.025
126.854
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77.000
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28.252
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OMe
Ph
S
1e
200
150
100
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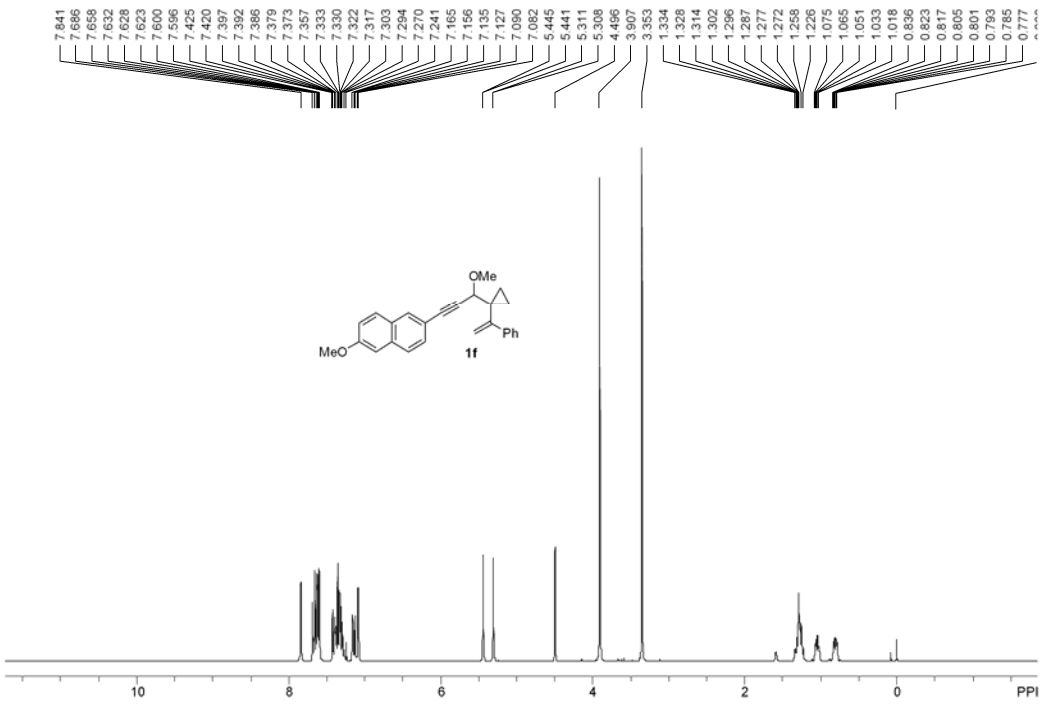
0 PPI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7.373
7.357
7.333
7.330
7.322
7.317
7.303
7.294
7.270
7.241
7.165
7.156
7.135
7.127
7.090
7.082
5.445
5.441
5.311
5.308
4.496
3.907
3.353
1.334
1.328
1.314
1.302
1.296
1.287
1.277
1.272
1.258
1.226
1.075
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1.051
1.033
1.018
0.836
0.823
0.817
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0.785
0.777
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MeO
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10
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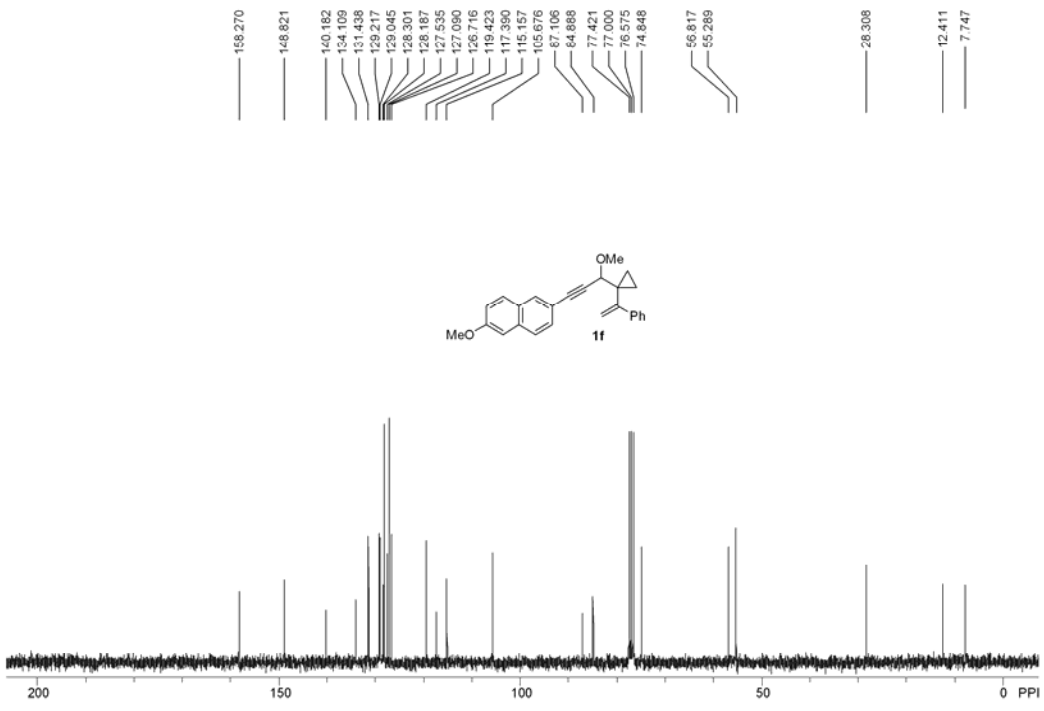
4
2
0
PPI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77.000
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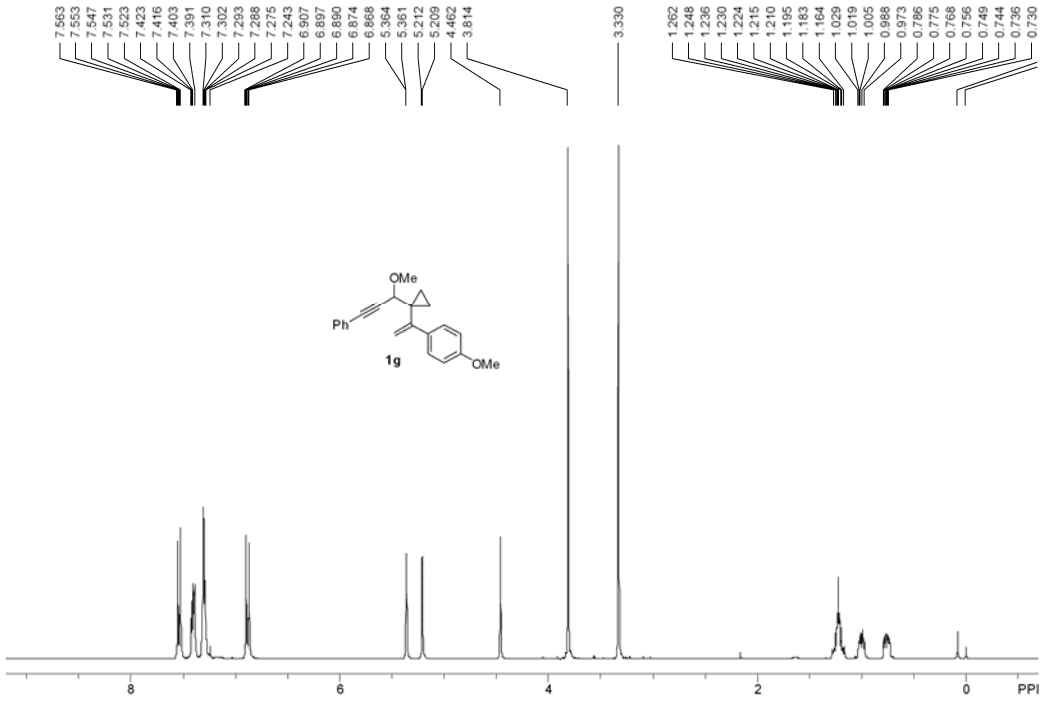
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7.310
7.302
7.293
7.288
7.275
7.243
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6.897
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6.874
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5.364
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5.212
5.209
4.462
3.814
3.330
1.262
1.248
1.236
1.230
1.224
1.215
1.210
1.195
1.183
1.164
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1.019
1.005
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0.775
0.768
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1g
OMe
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4
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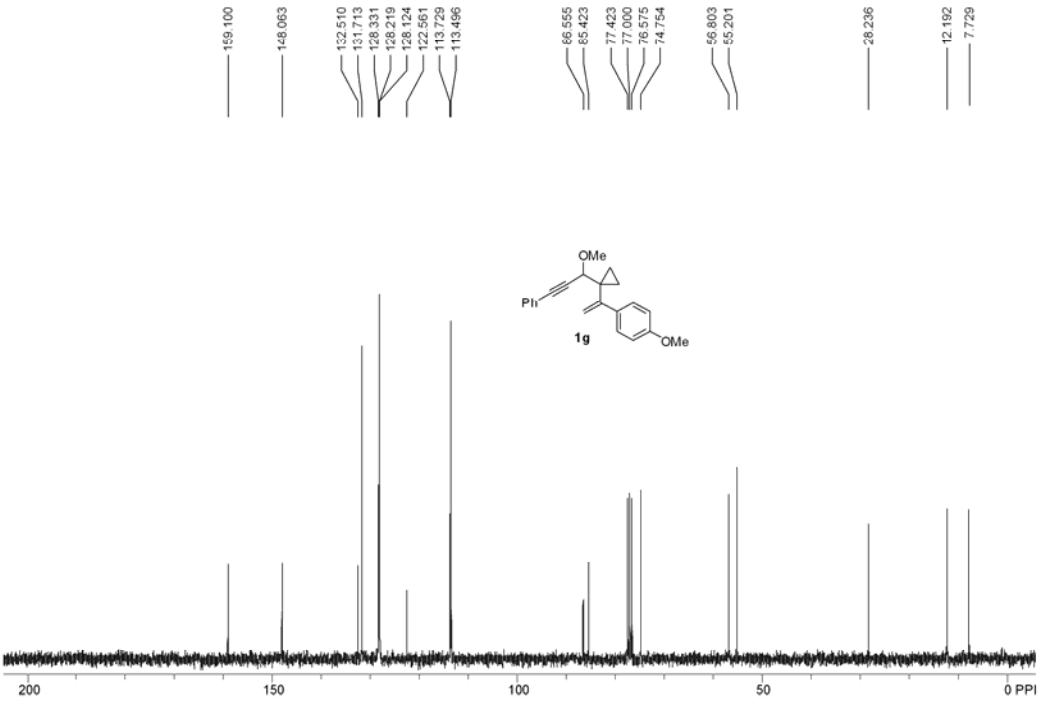
PPI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Ph
1g
OMe
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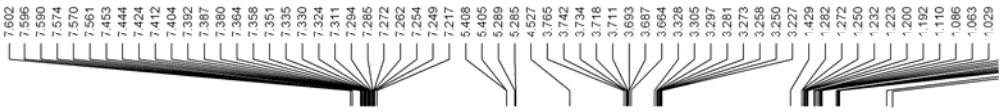
150
100
50
0 PPI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7.335
7.330
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7.285
7.272
7.262
7.254
7.249
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5.405
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5.285
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3.250
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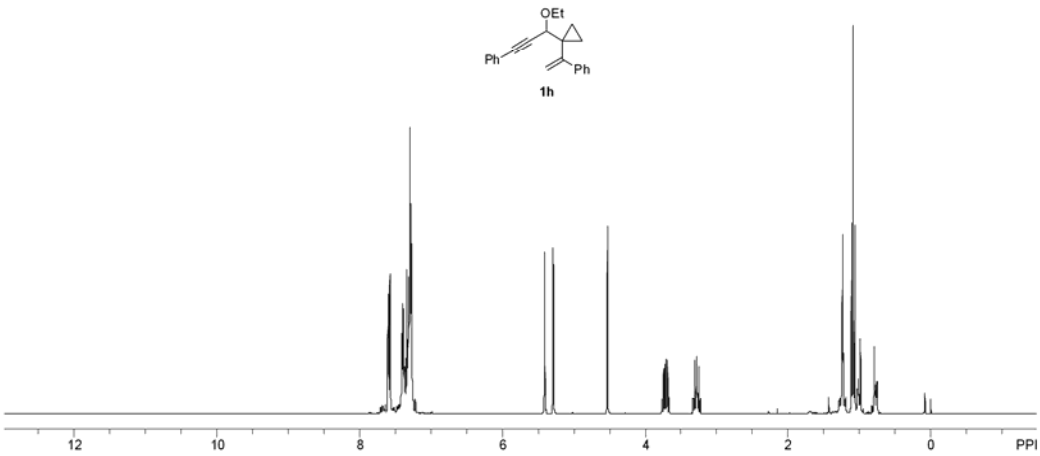
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Ph
Ph
1h
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8
6
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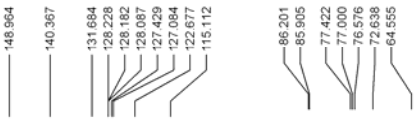
PPI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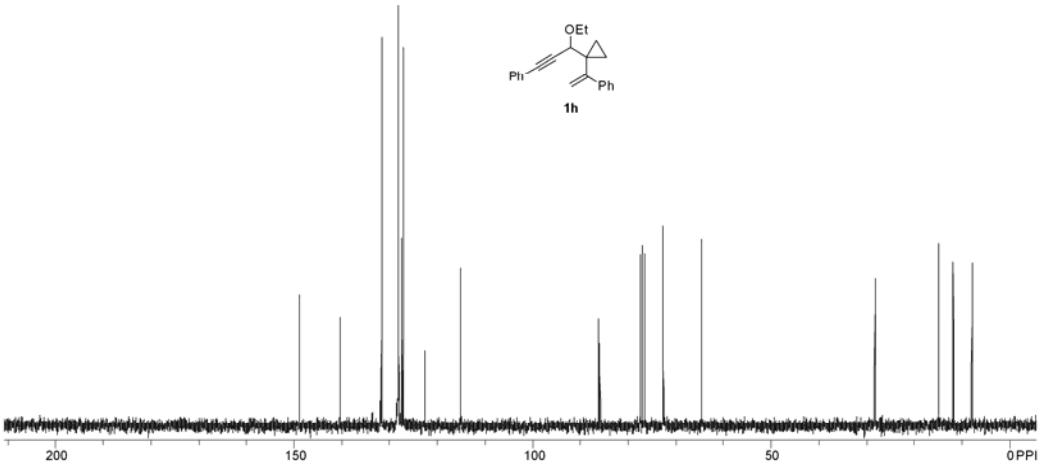
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Ph
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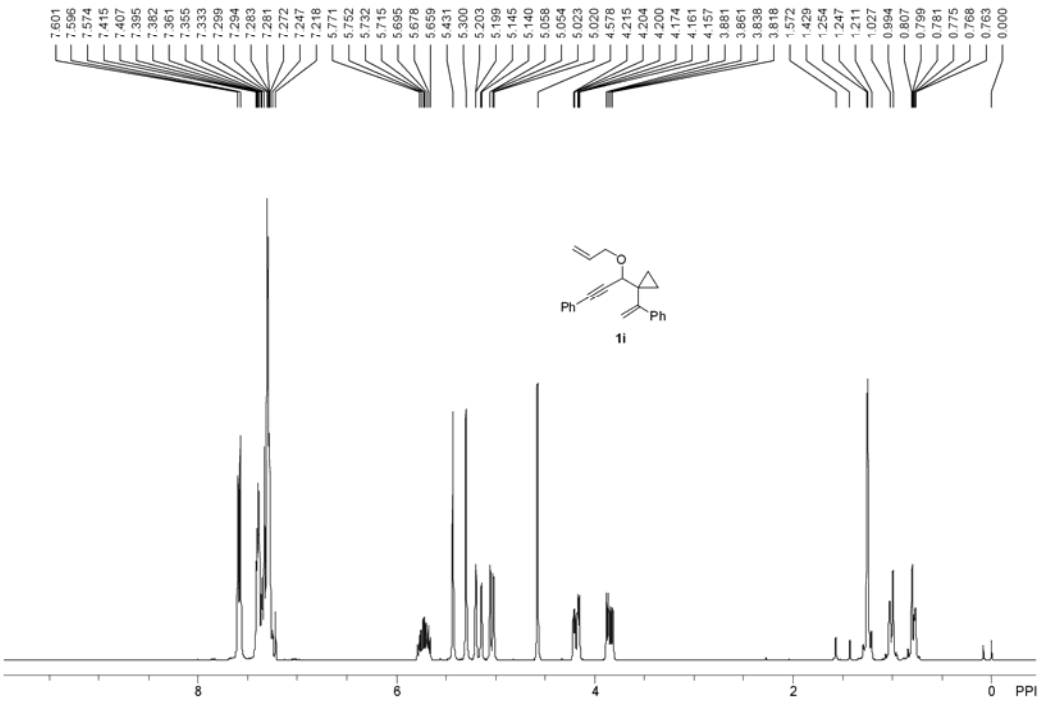
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7.283
7.281
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7.218
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5.732
5.715
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5.678
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5.431
5.300
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5.140
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5.054
5.023
5.020
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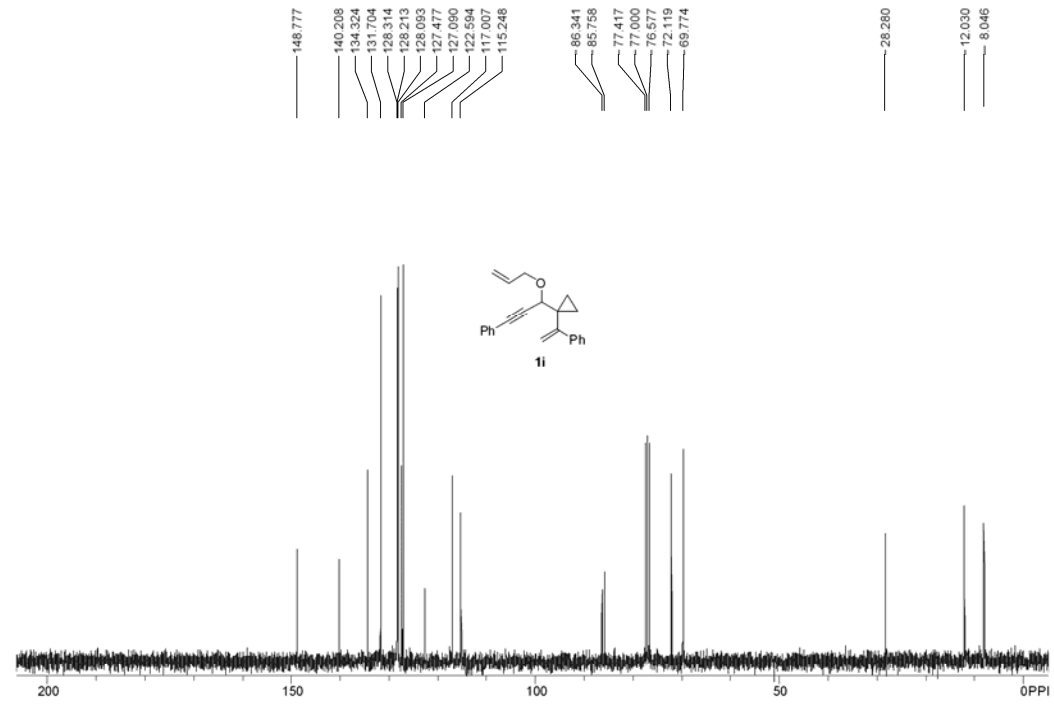
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Ph
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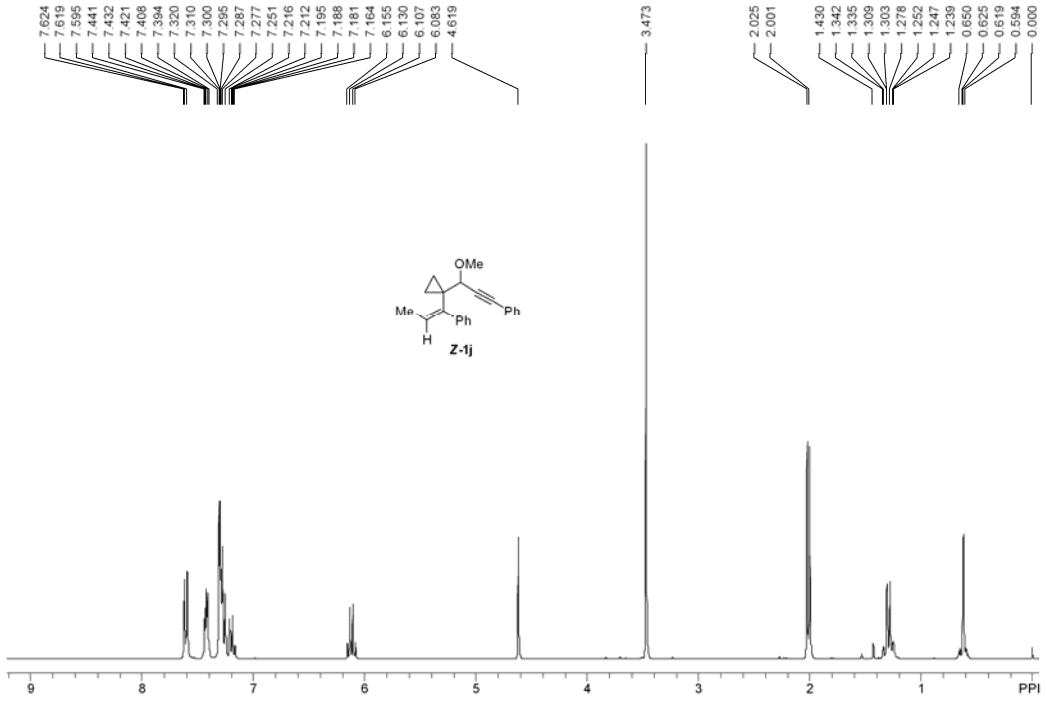
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7.188
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7.164
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6.130
6.107
6.083
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0.619
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Ph
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1
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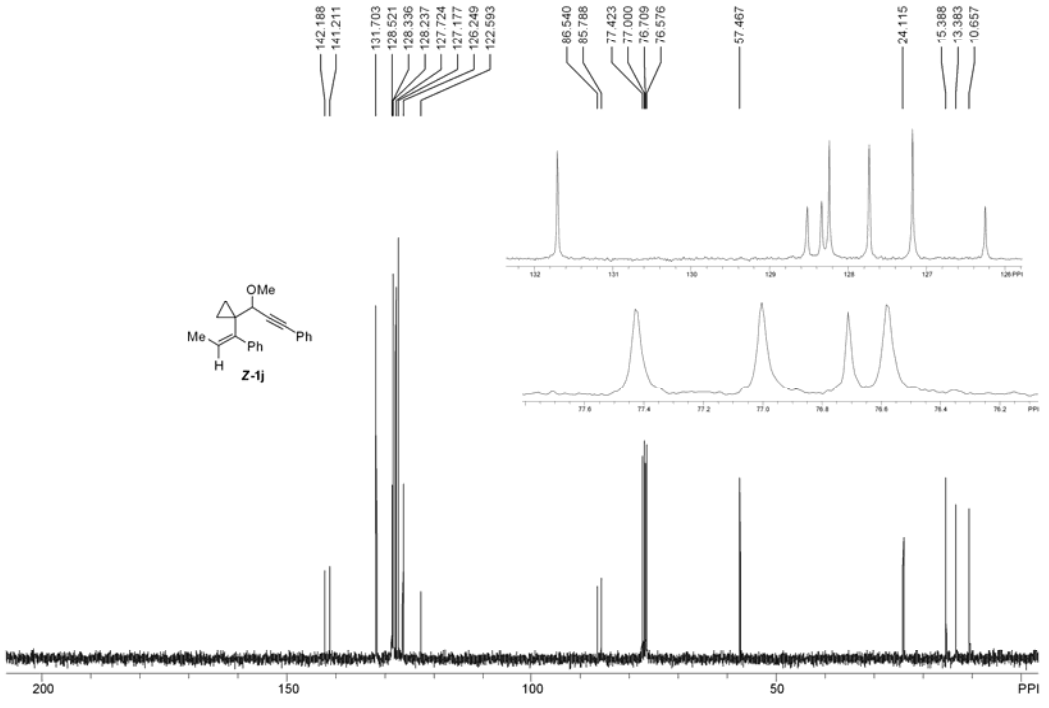
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57.467
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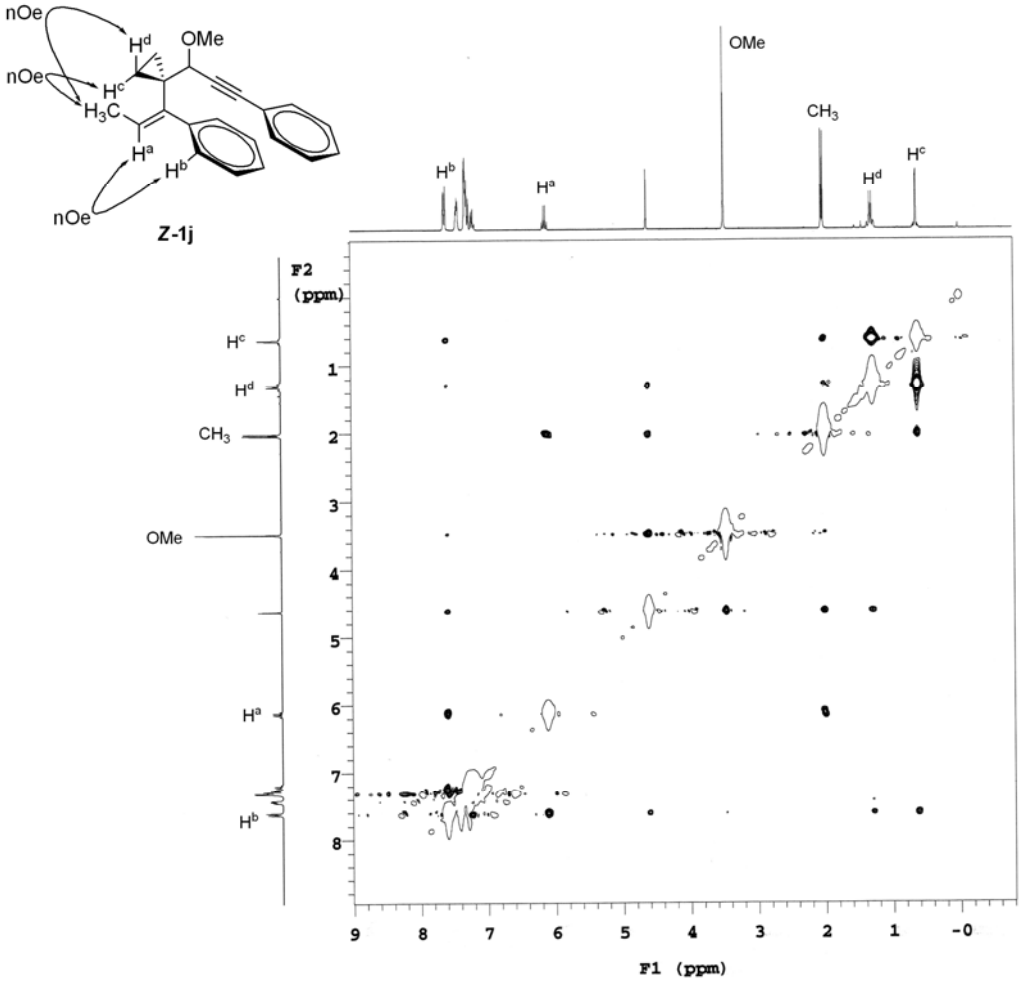
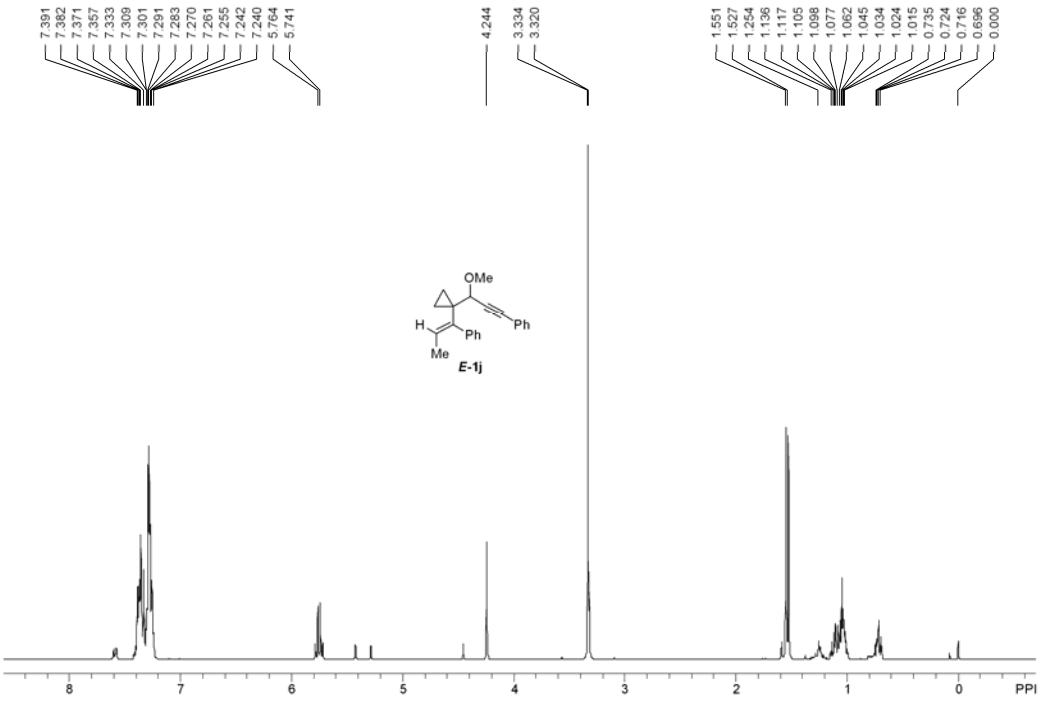
OMe
Me
Ph
Ph
H
Z-1j
200
150
100
50
PPI


nOe
nOe
nOe
H^d
OMe
H^c
H_3C
H^a
H^b
Z-1j
OMe
CH_3
H^b
H^a
H^d
H^c
F2 (ppm)
F1 (ppm)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7.255
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7.240
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4.244
3.334
3.320
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Ph
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Ph
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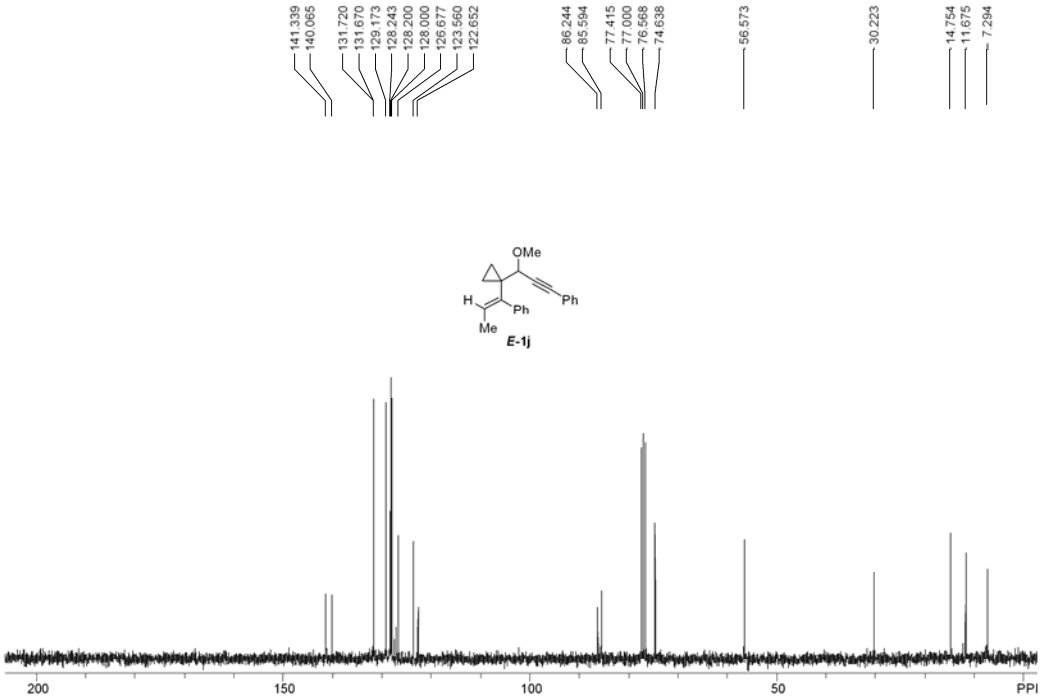
E-1j
PPM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123.560
122.652
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77.000
76.588
74.638
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11.675
7.294
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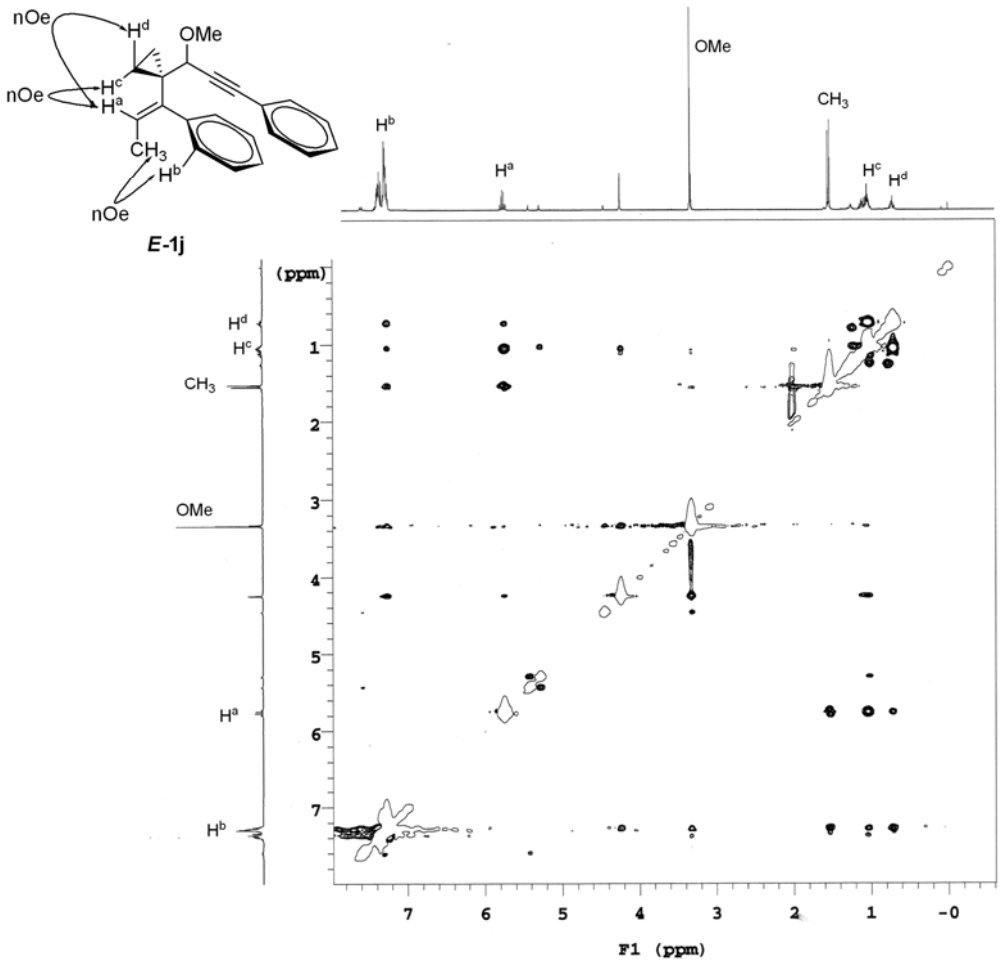
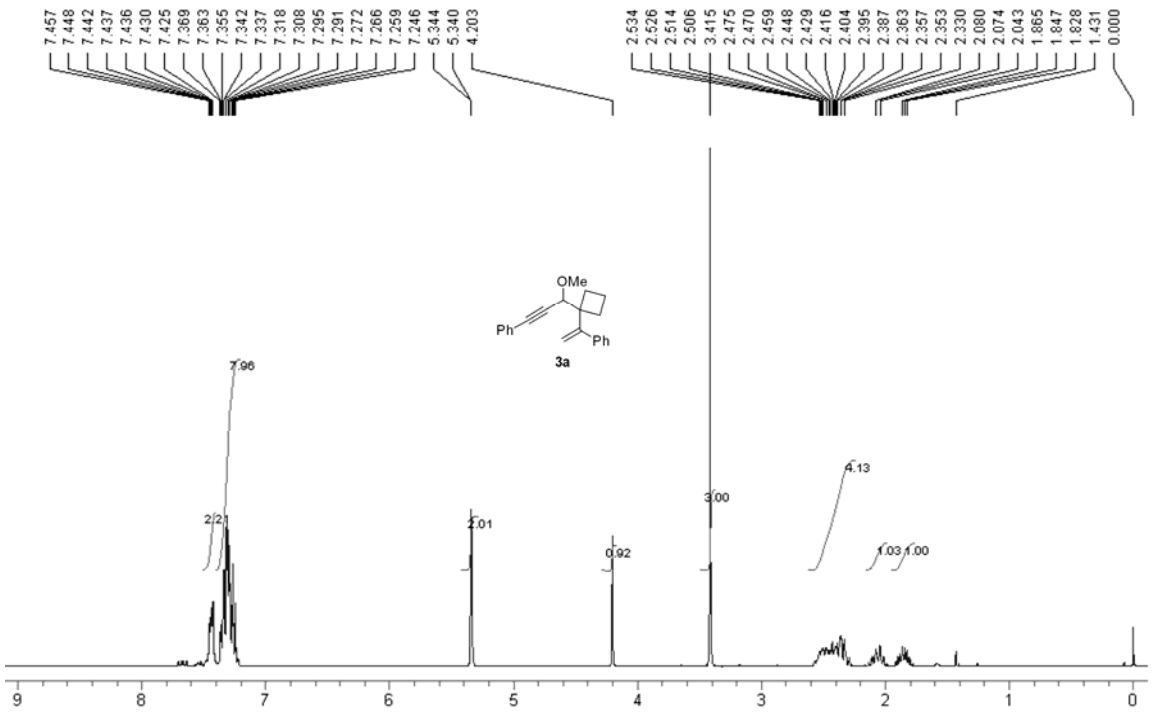
Ph
Ph
H
Me
E-1j
200
150
100
50
PPI


nOe
Hd
OMe
nOe
Hc
Ha
CH3
Hb
nOe
E-1j
OMe
CH3
Hb
Ha
Hc
Hd
(ppm)
Hd
Hc
CH3
OMe
Ha
Hb
1
2
3
4
5
6
7
7
6
5
4
3
2
1
-0
F1 (ppm)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7.291
7.272
7.266
7.259
7.246
5.344
5.340
4.203
2.534
2.526
2.514
2.506
3.415
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2.429
2.416
2.404
2.395
2.387
2.363
2.357
2.353
2.330
2.080
2.074
2.043
1.865
1.847
1.828
1.431
0.000
OMe
Ph
Ph
3a
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2.21
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1.03
1.00
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8
7
6
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1
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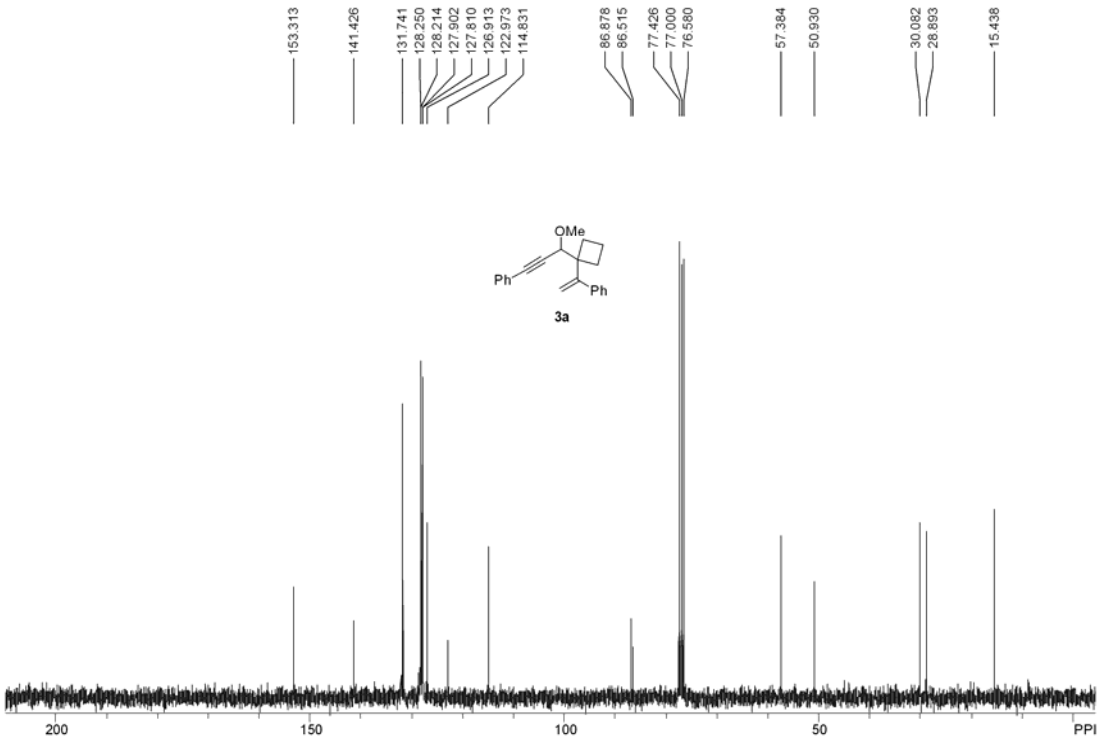
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86.515
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77.000
76.580
57.384
50.930
30.082
28.893
15.438
OMe
Ph
Ph
3a
200
150
100
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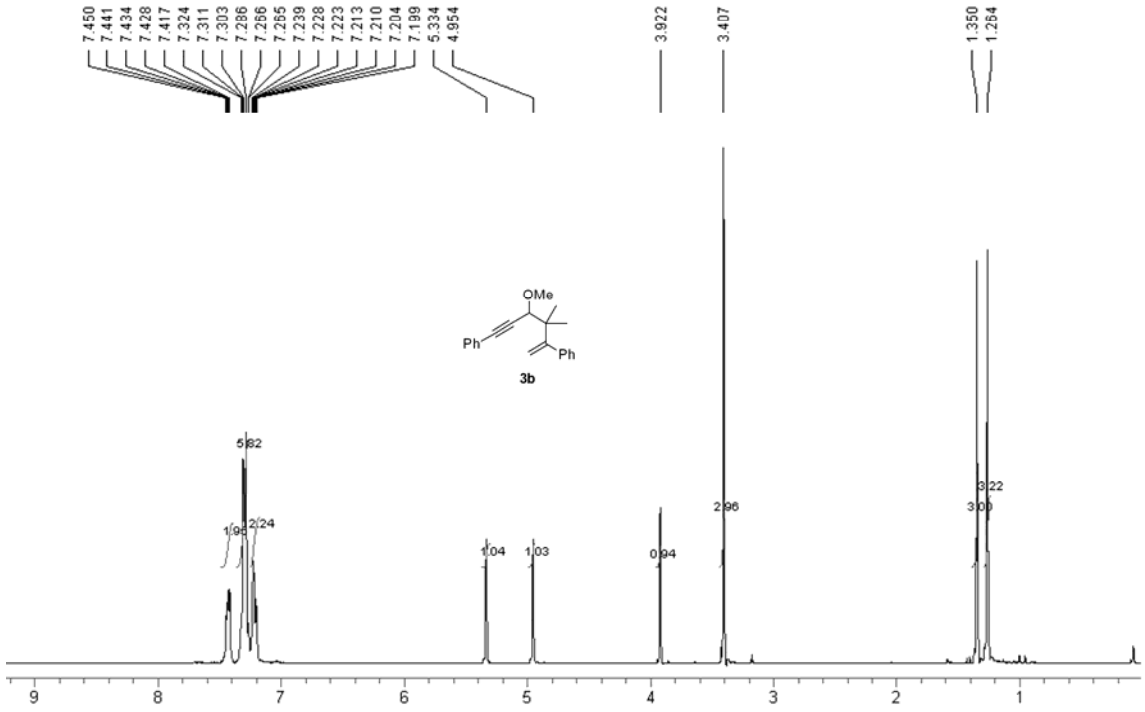
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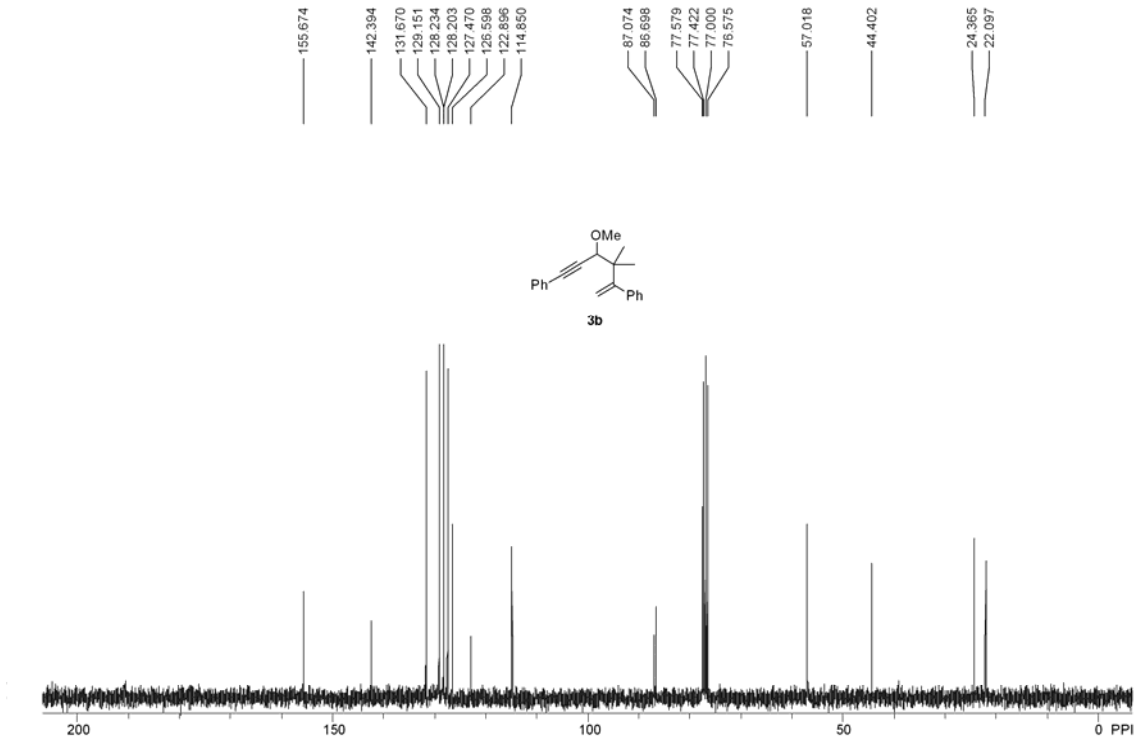
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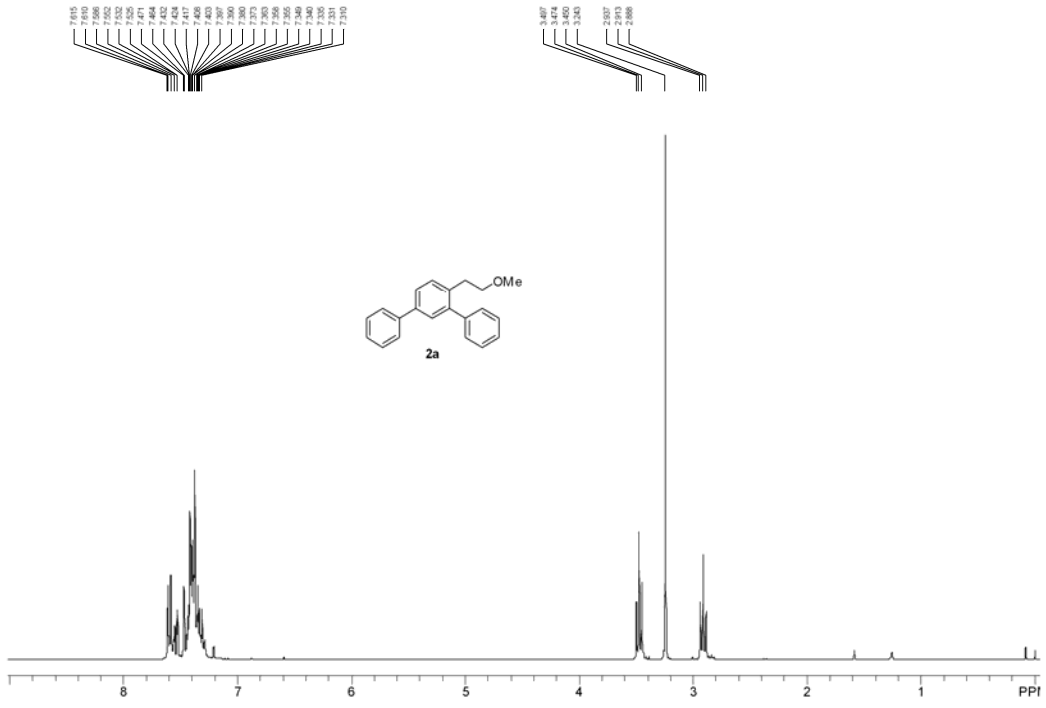
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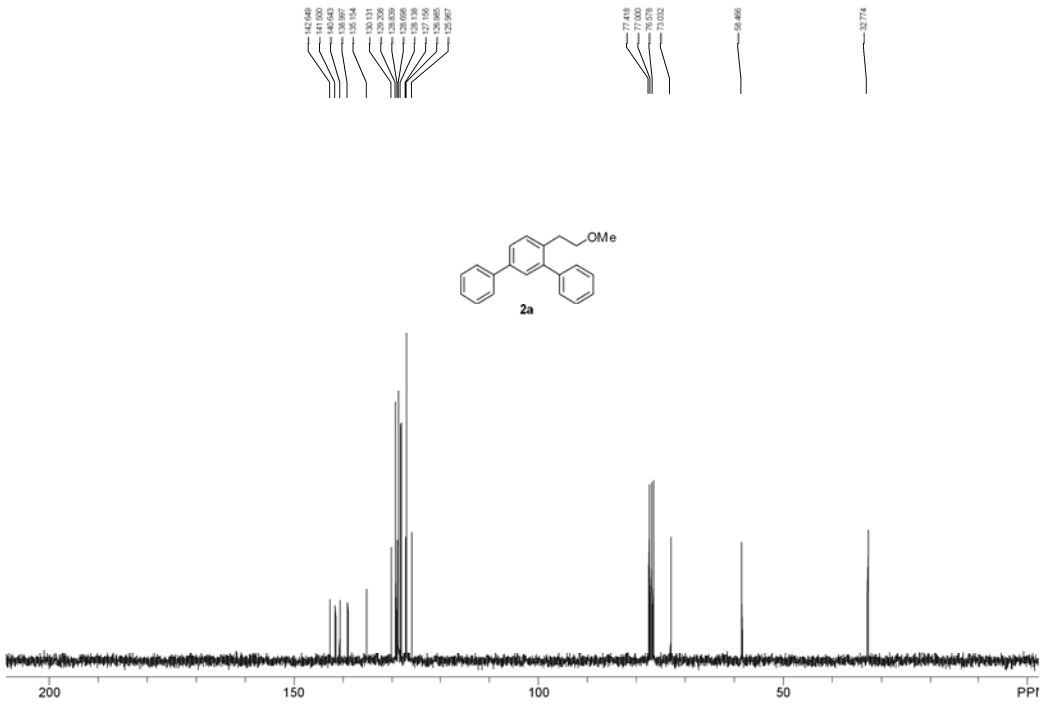
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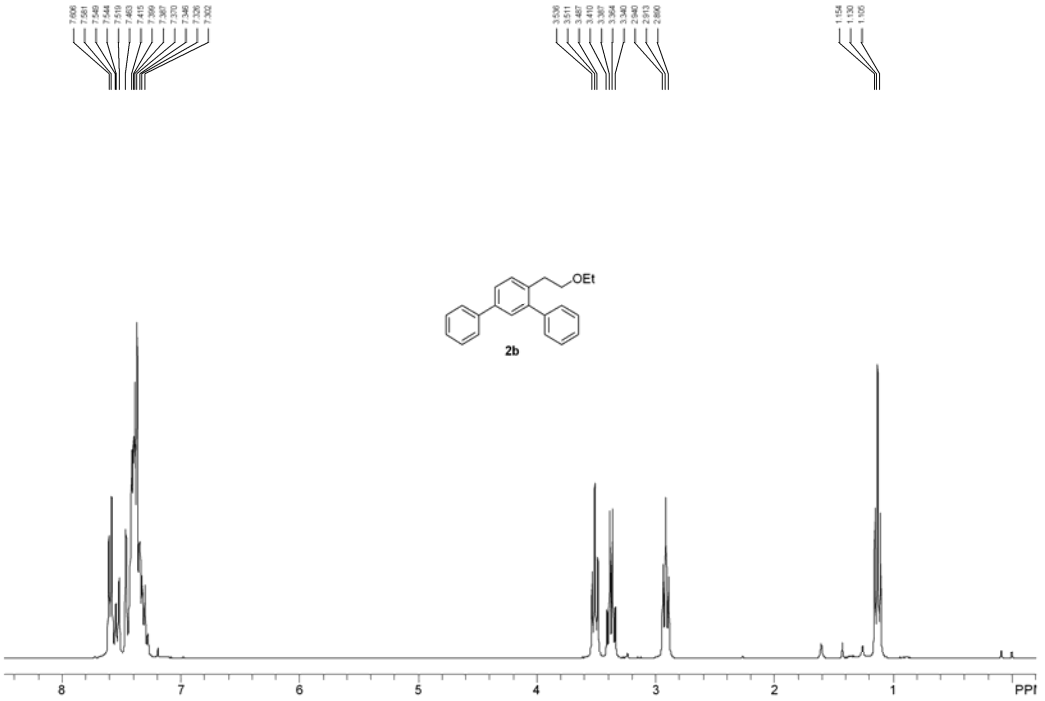
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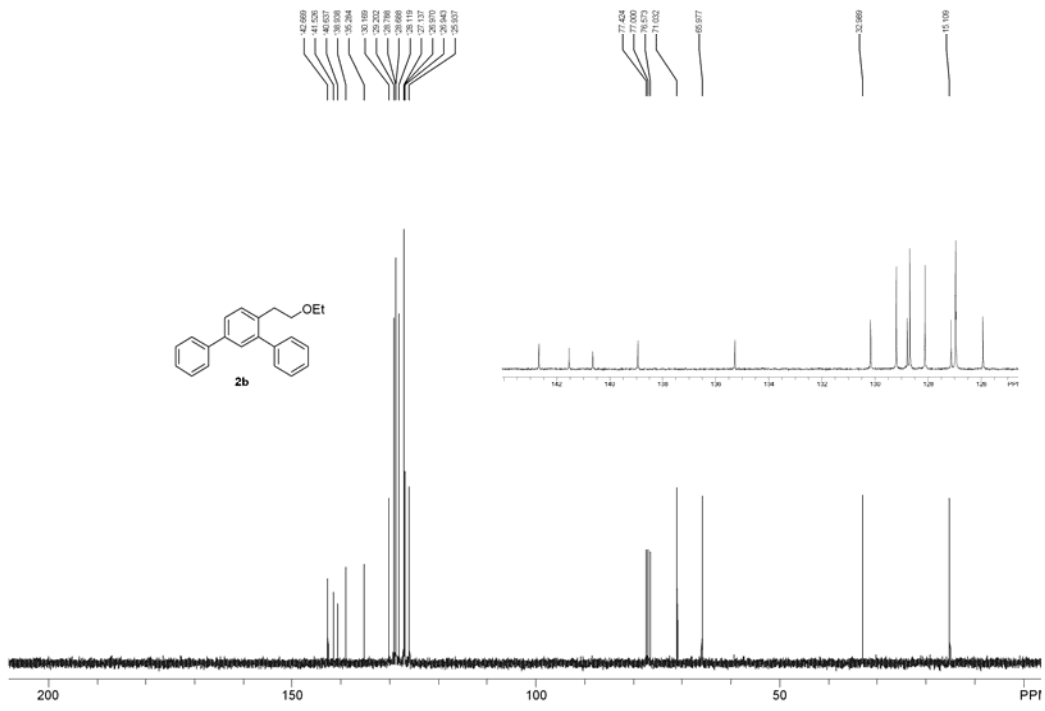
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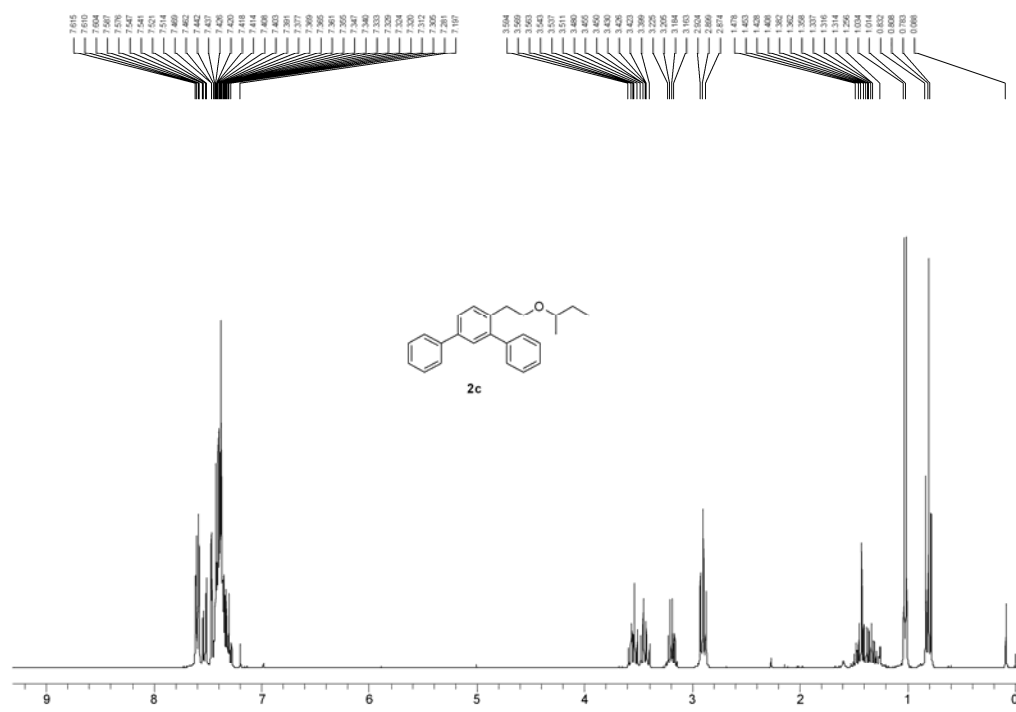
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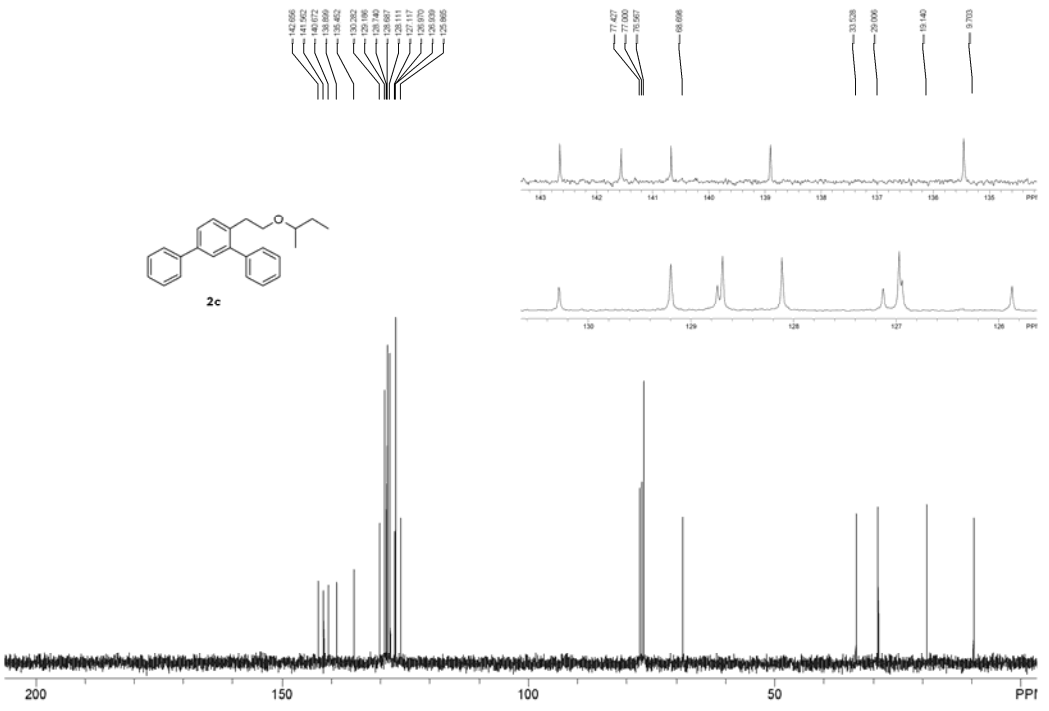
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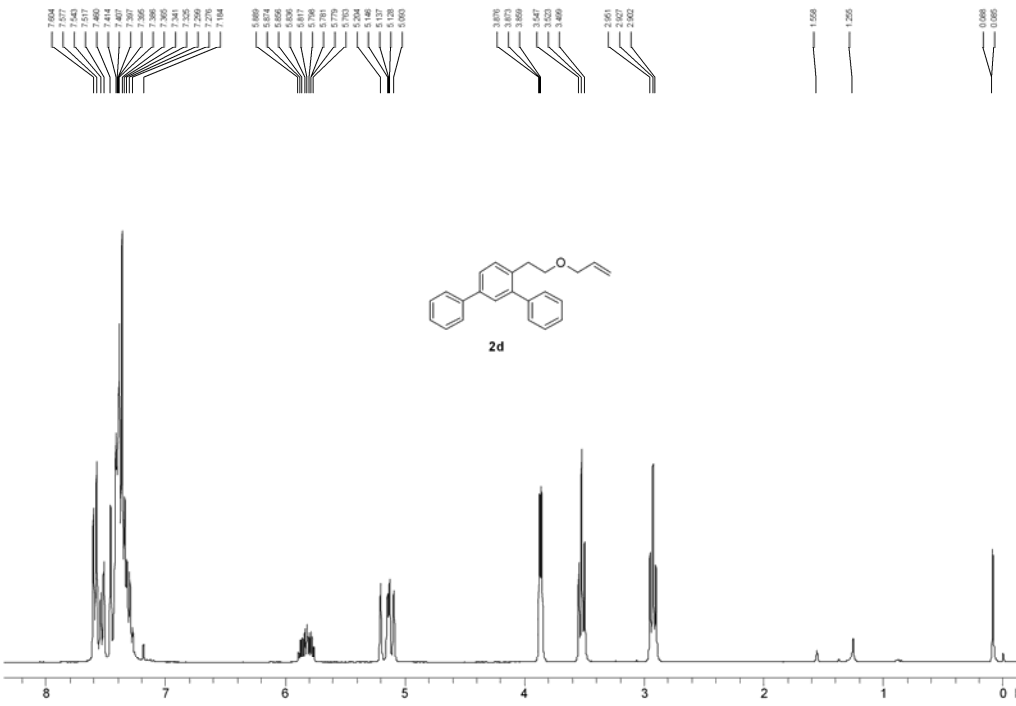
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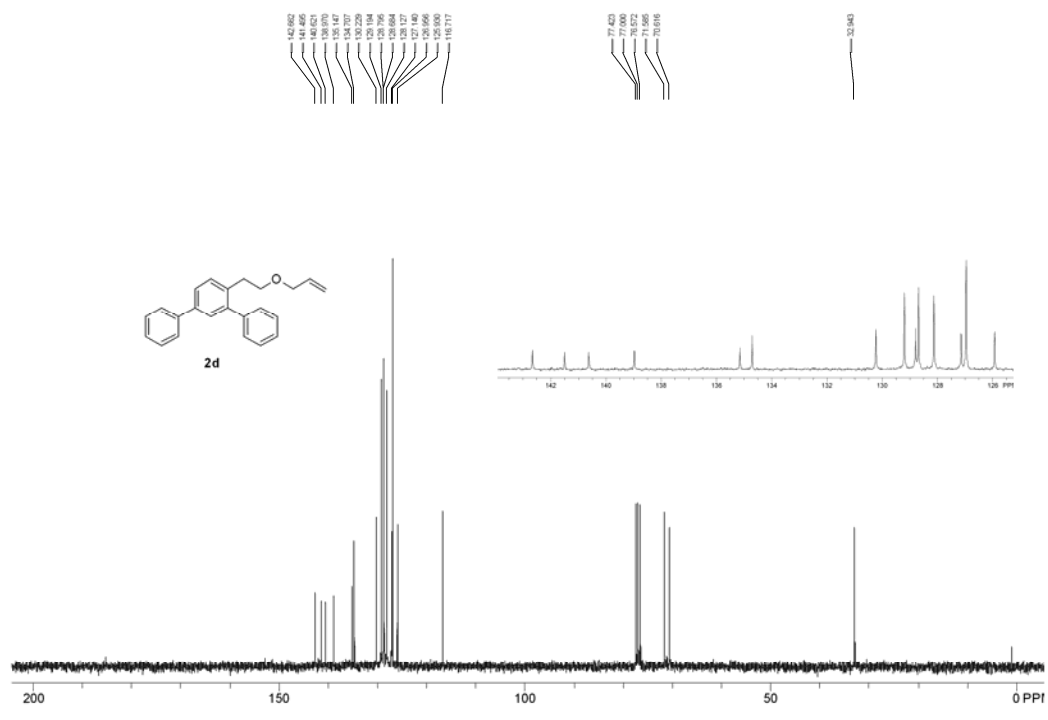
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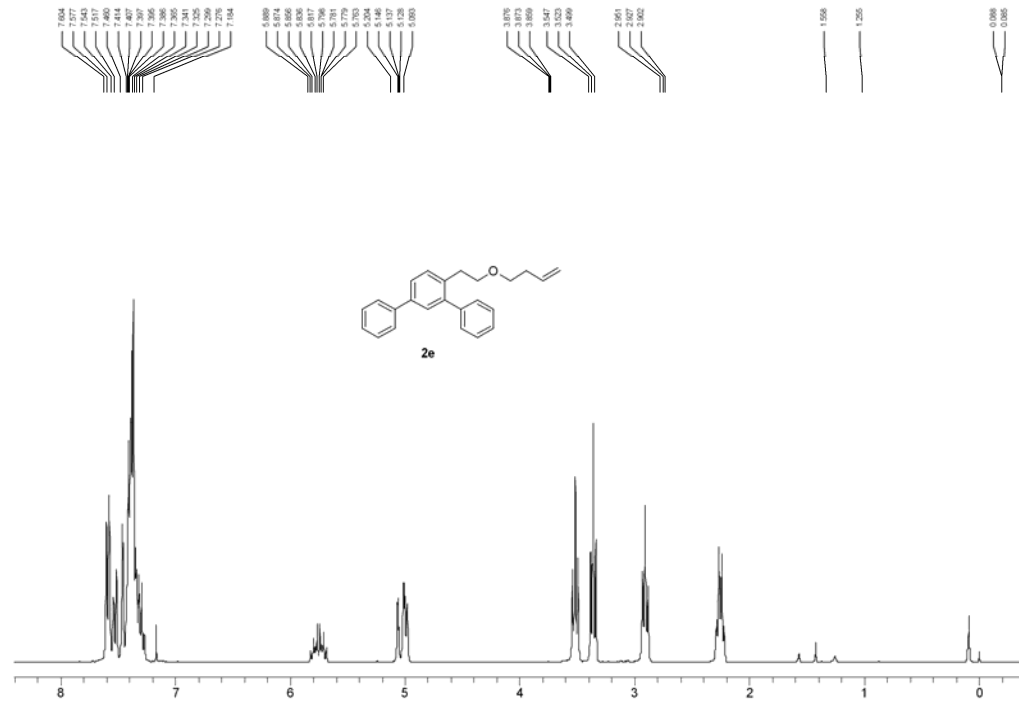
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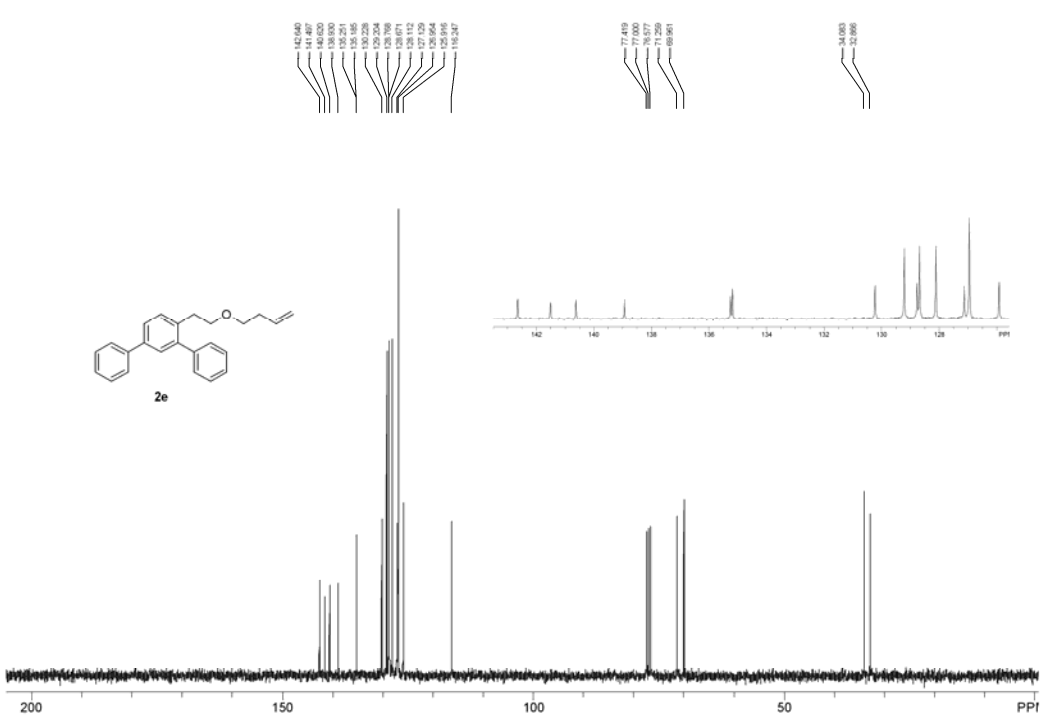
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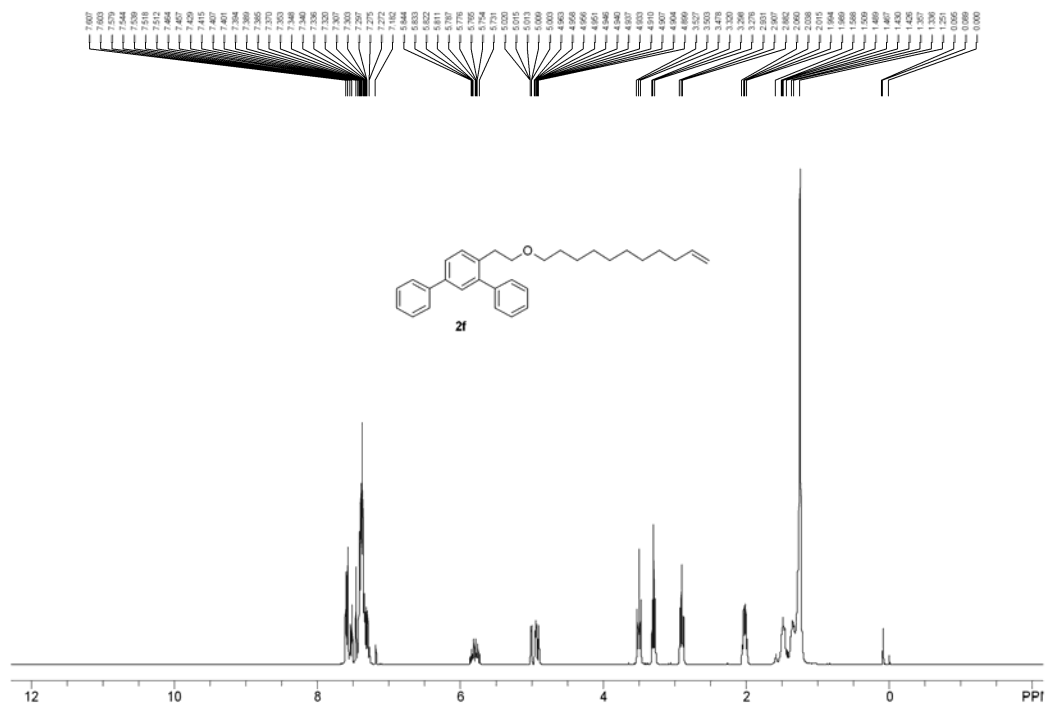
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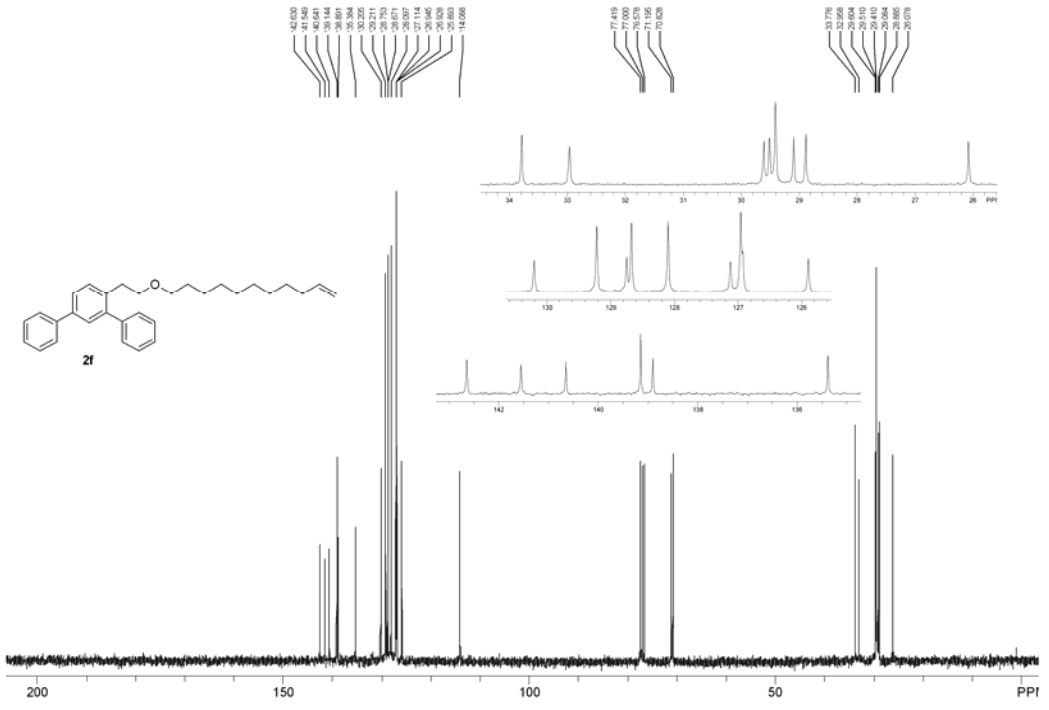
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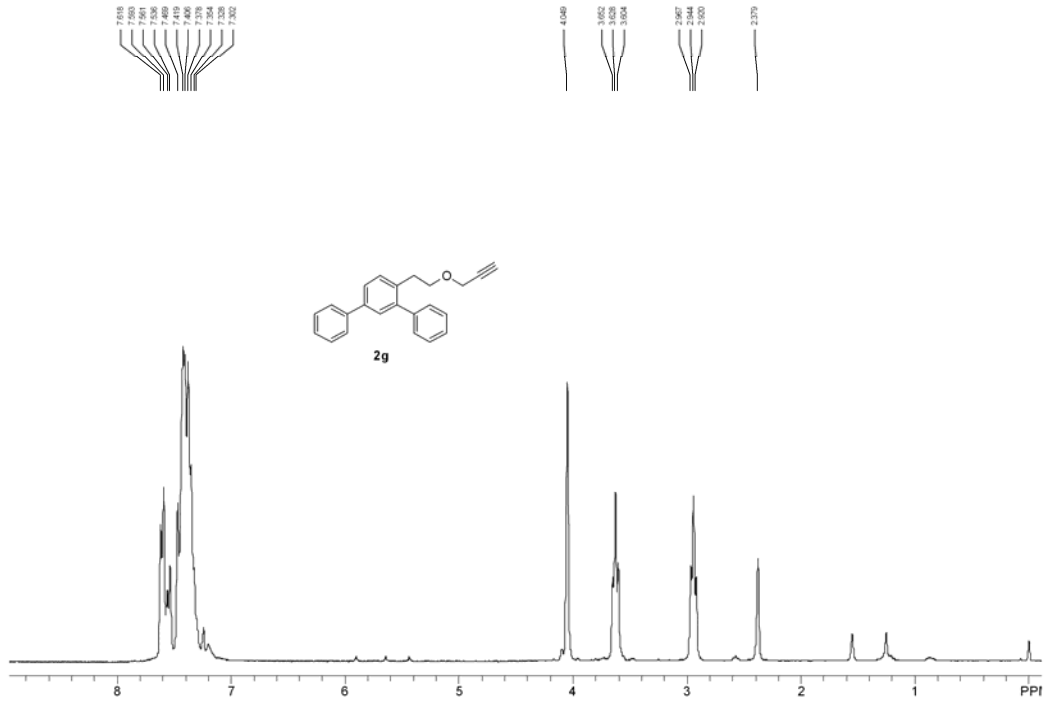
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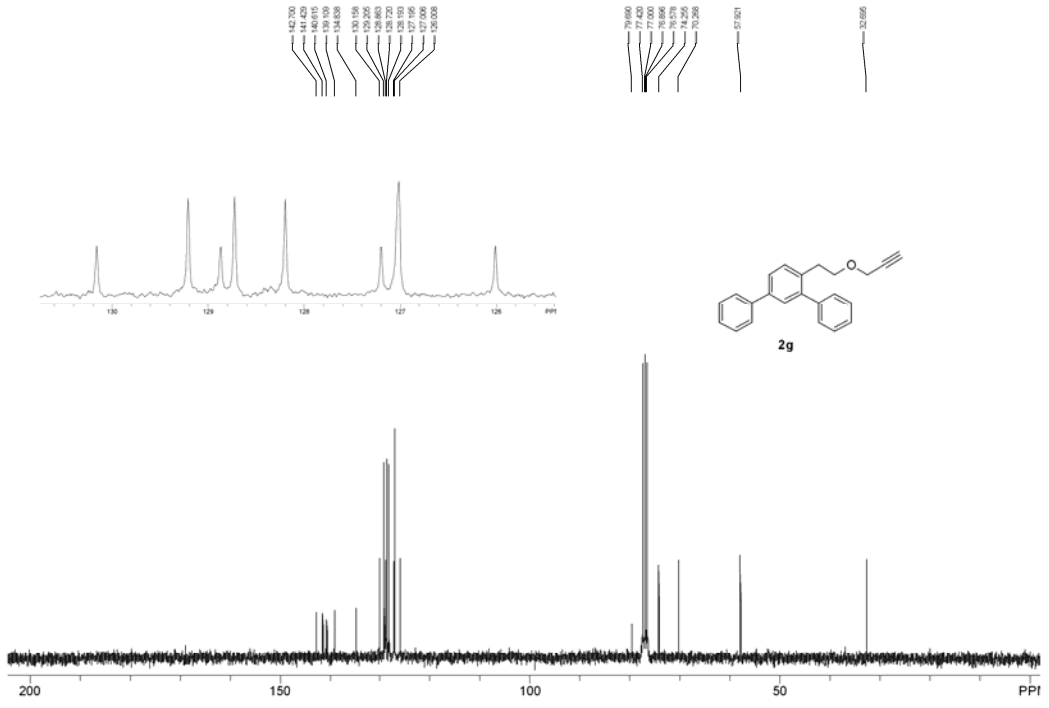
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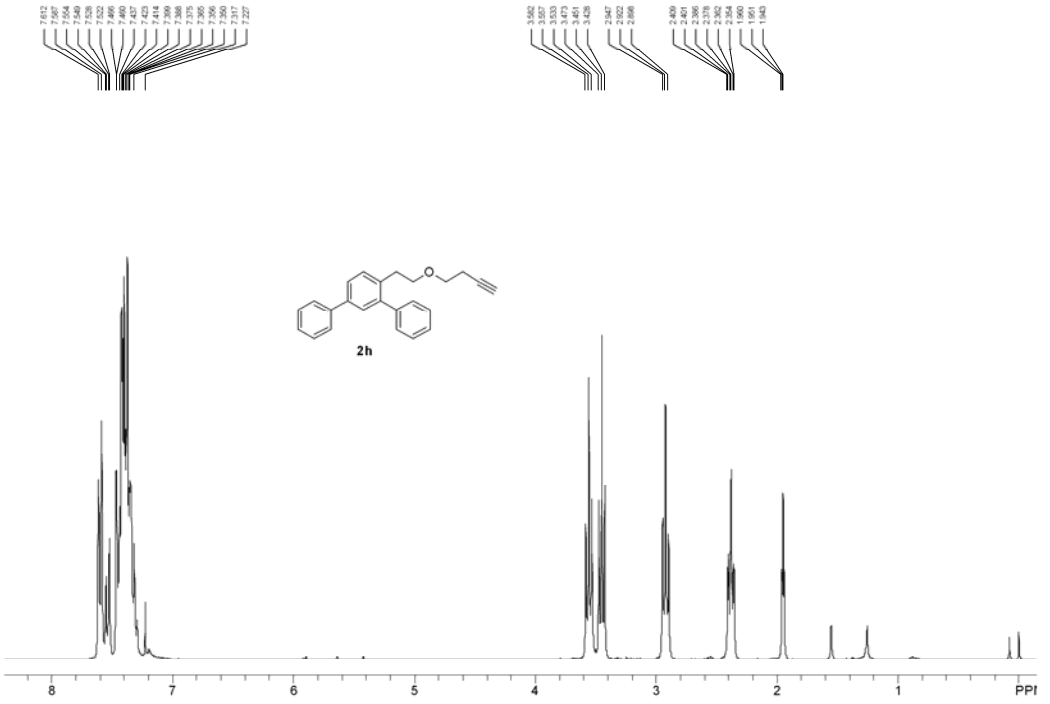
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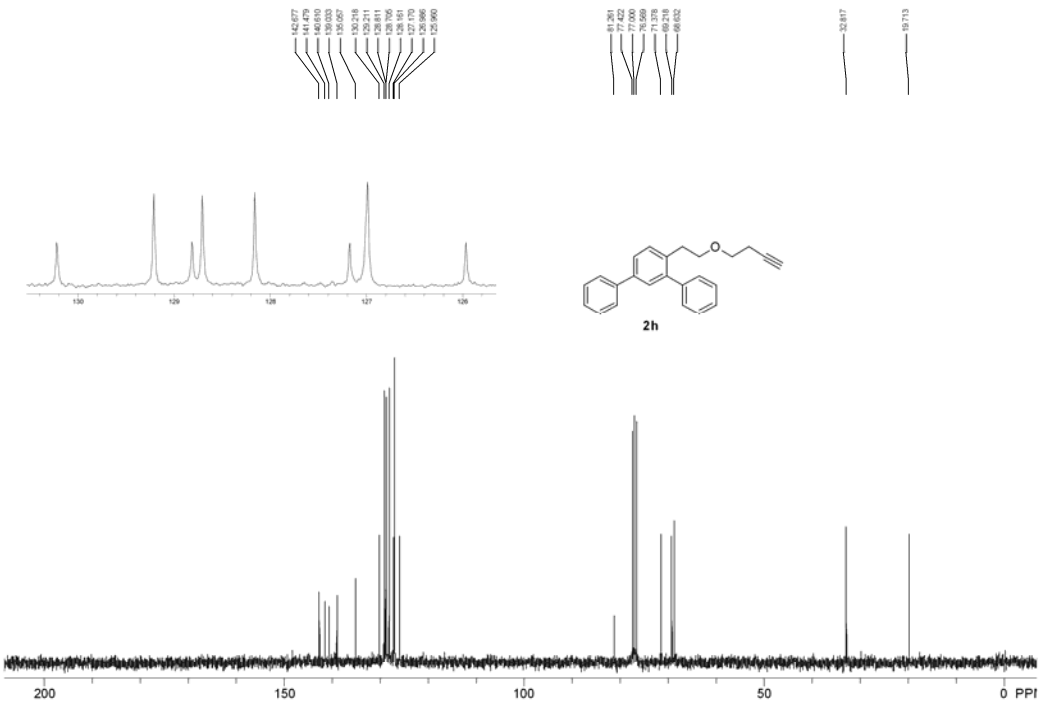
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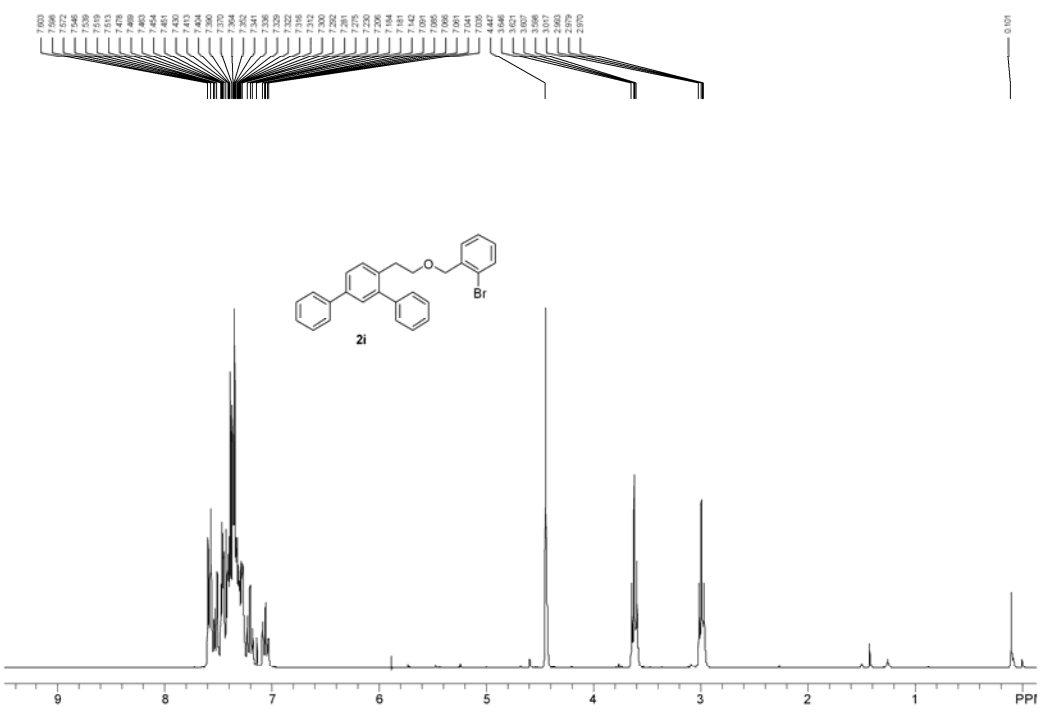
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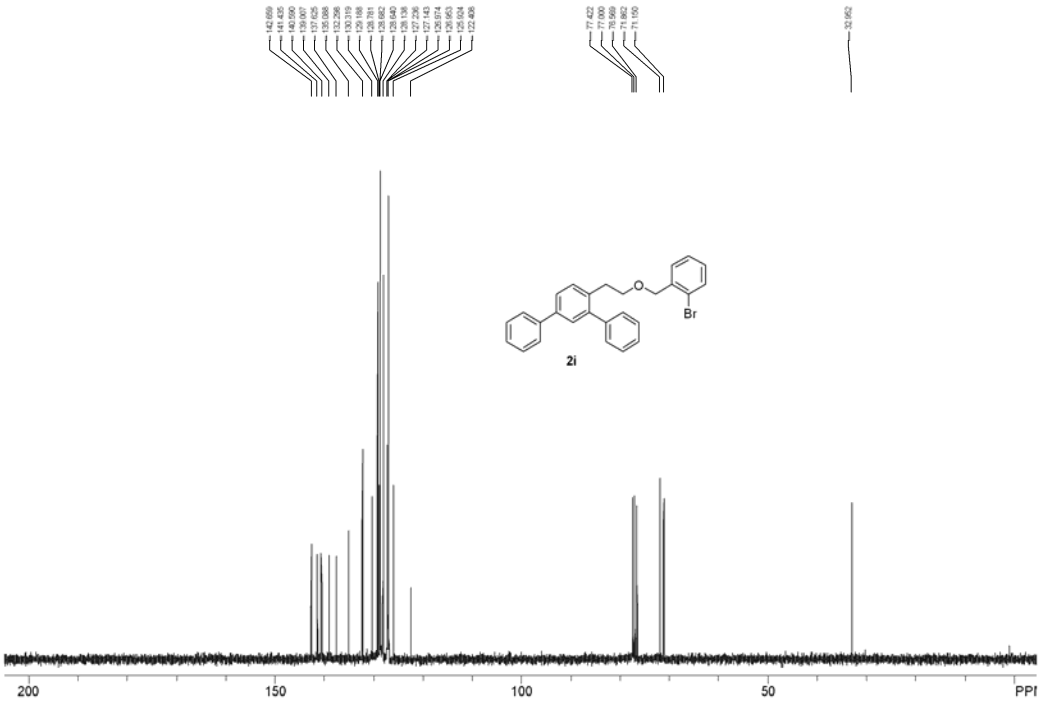
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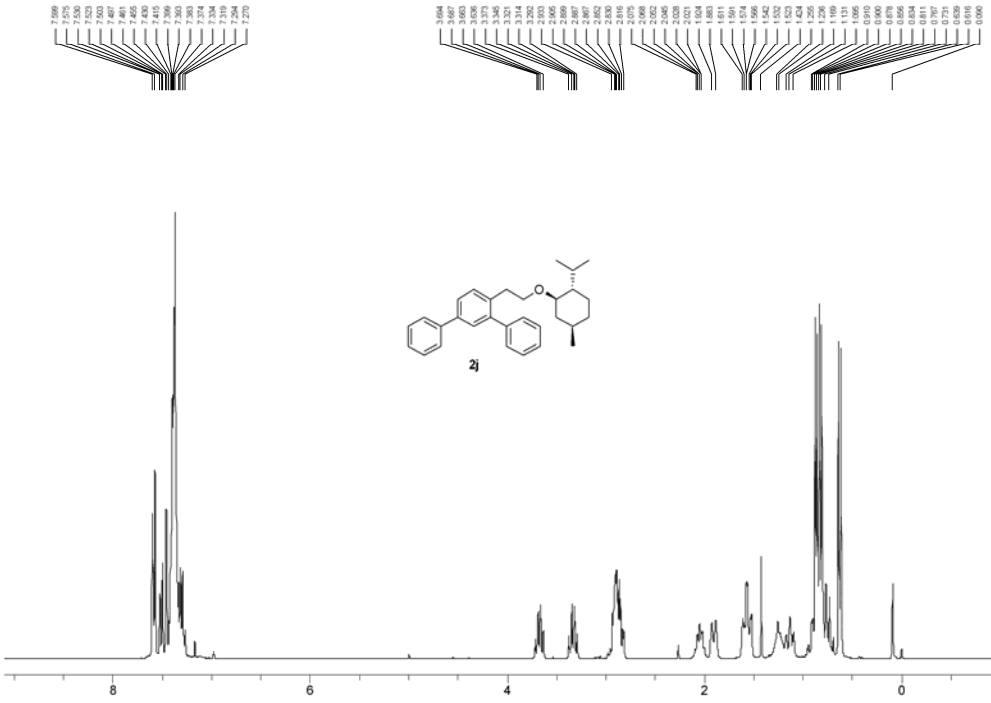
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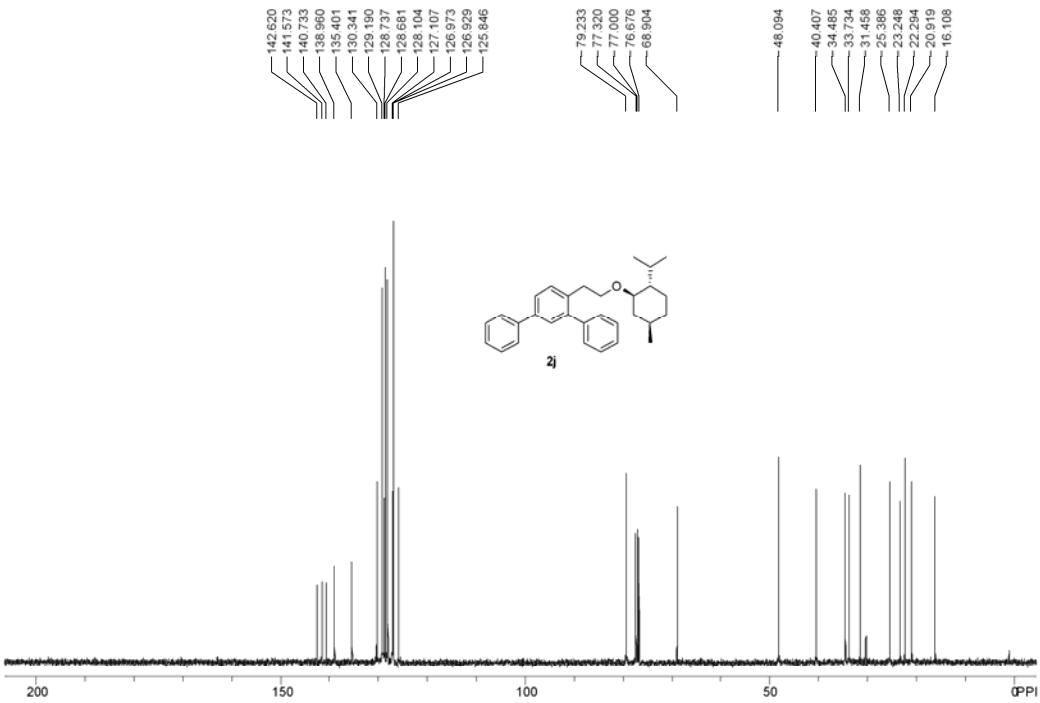
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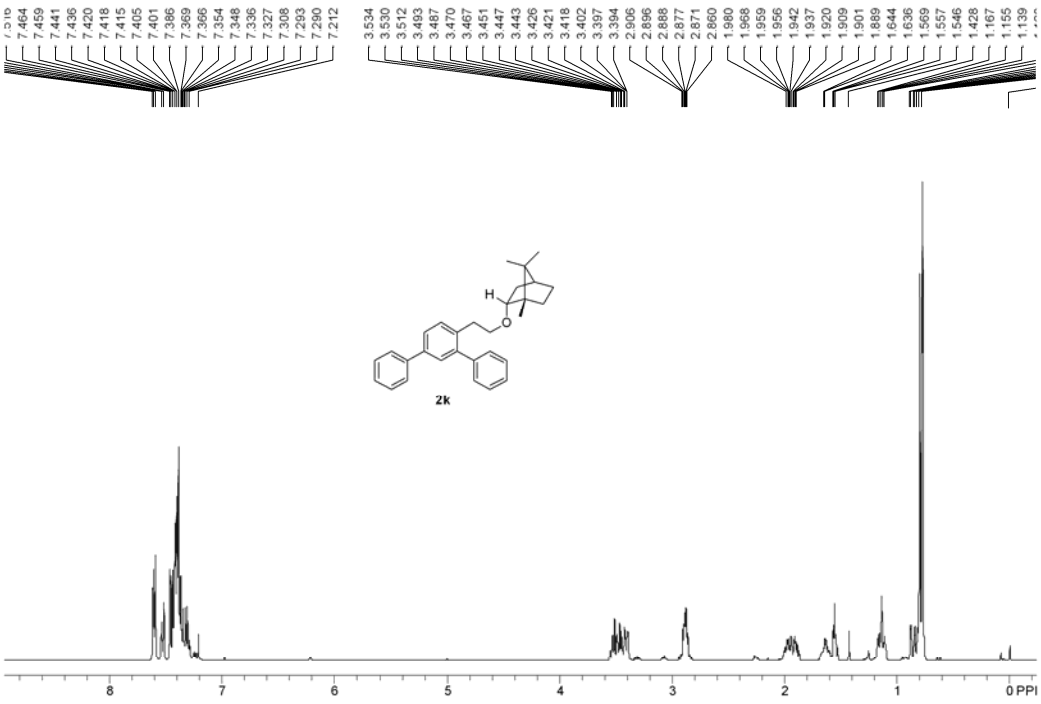
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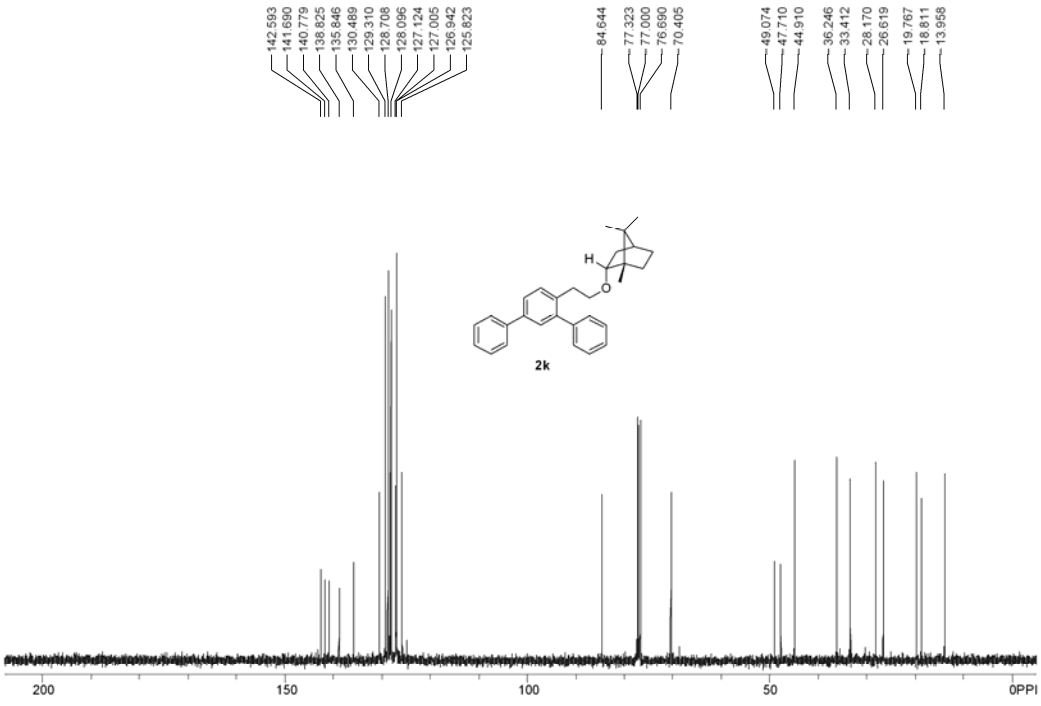
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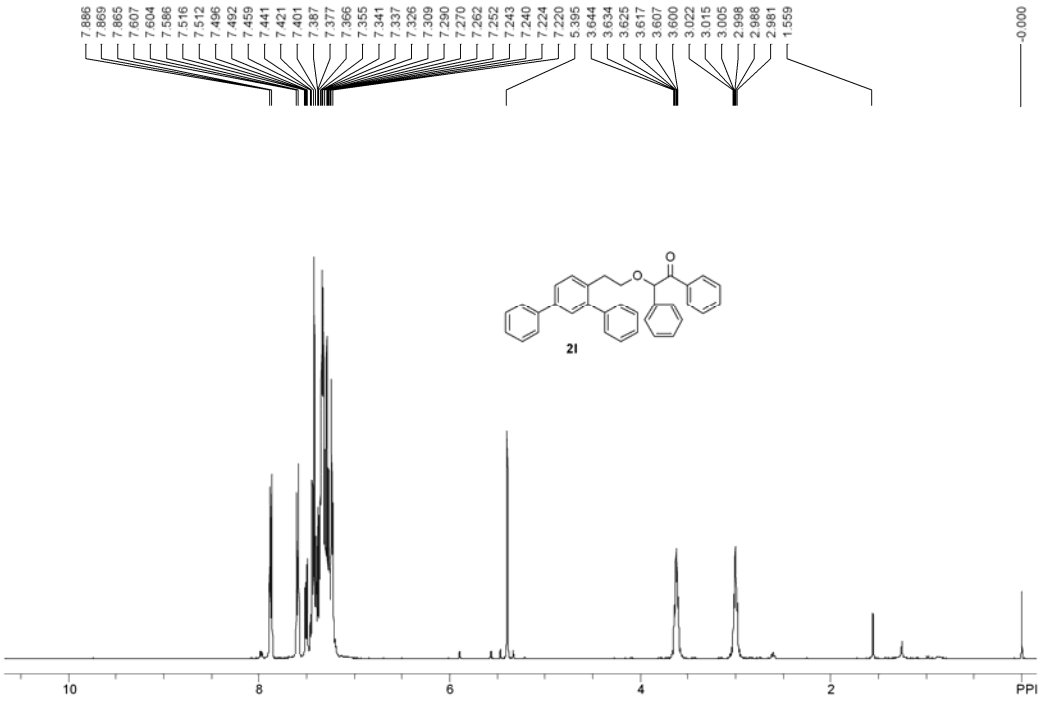
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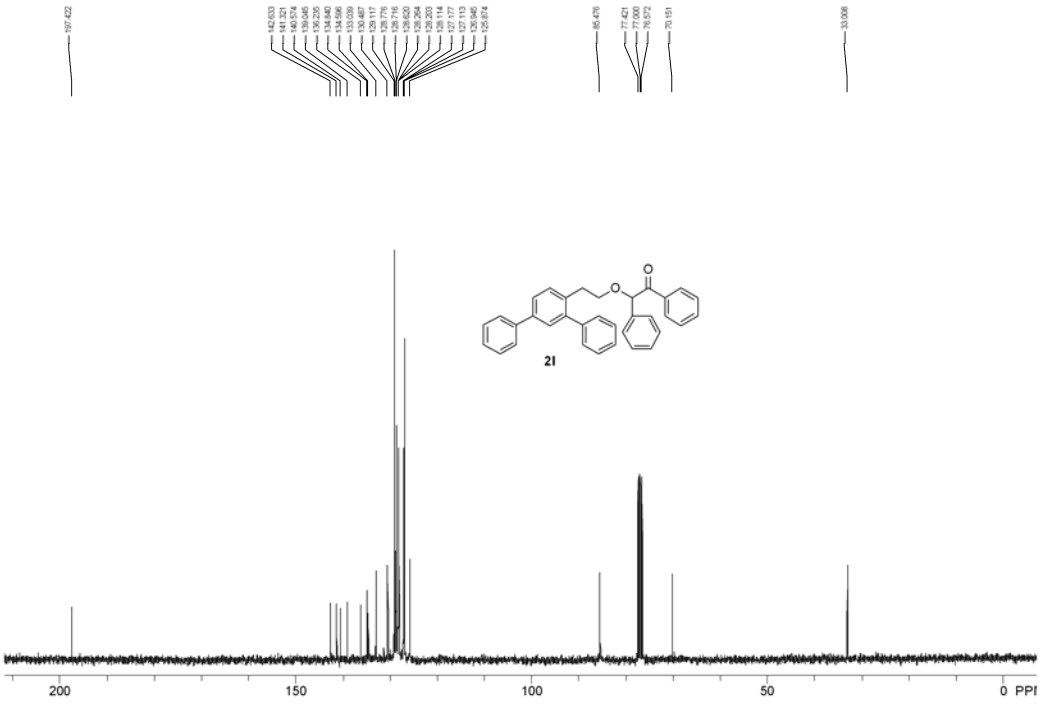
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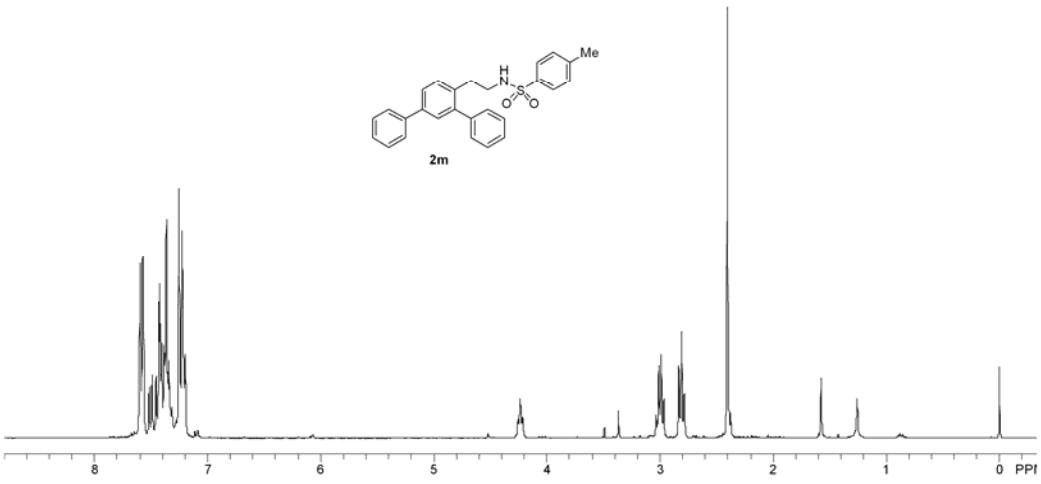
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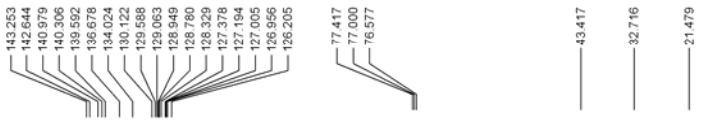
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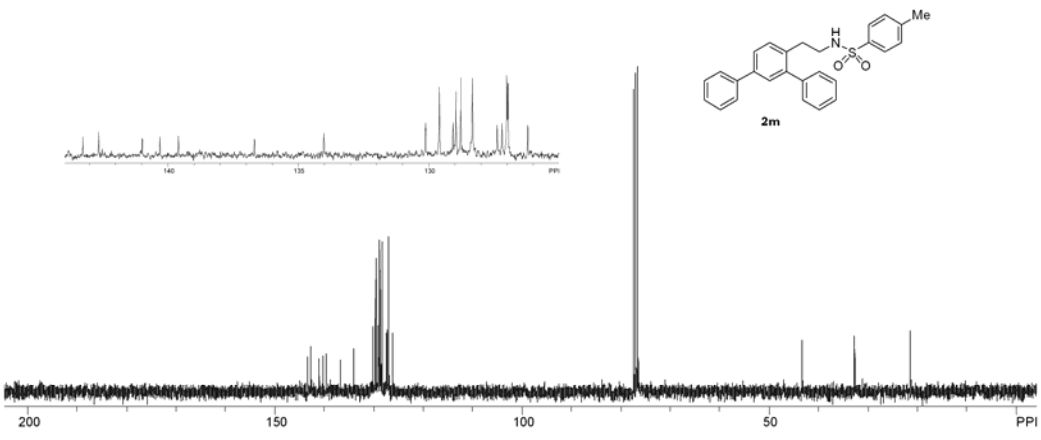
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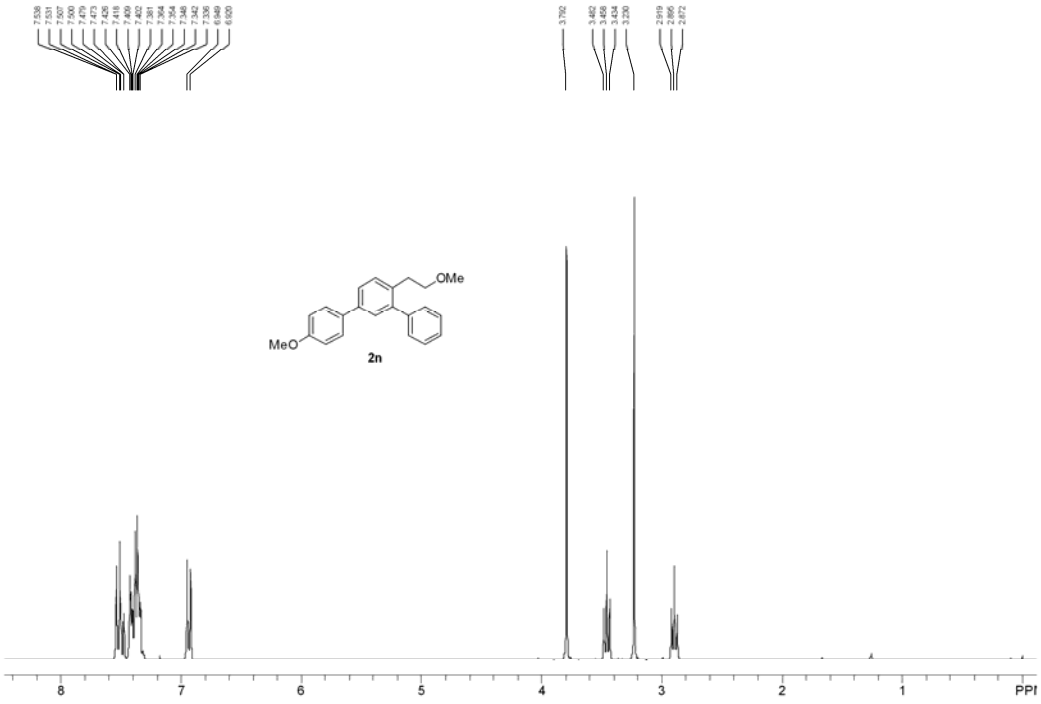
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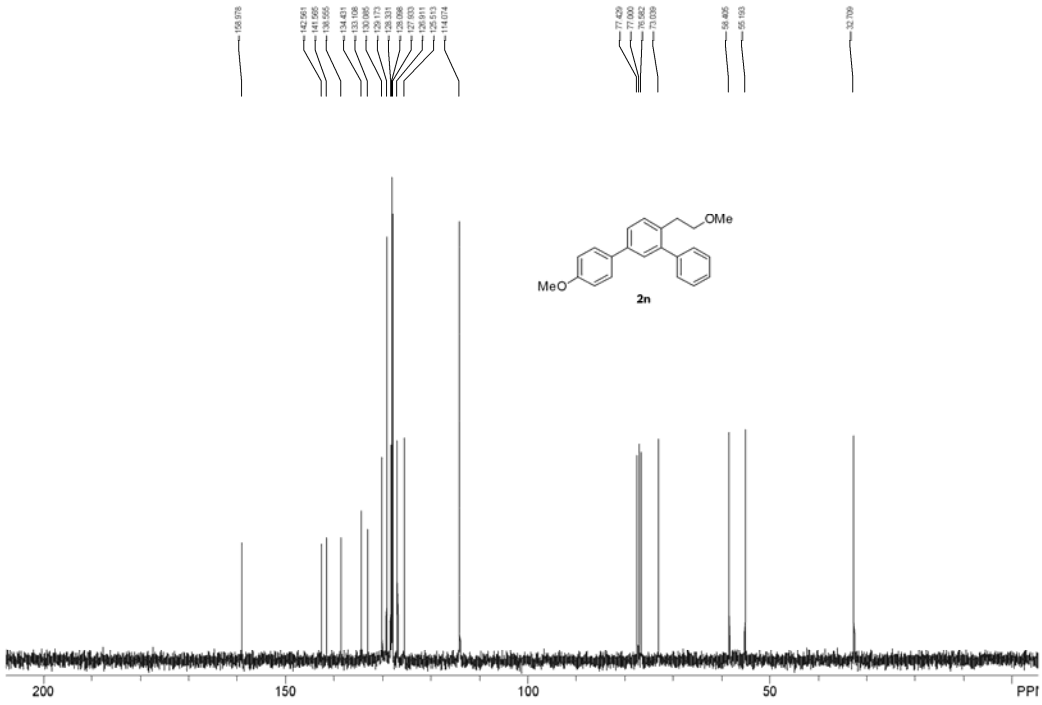
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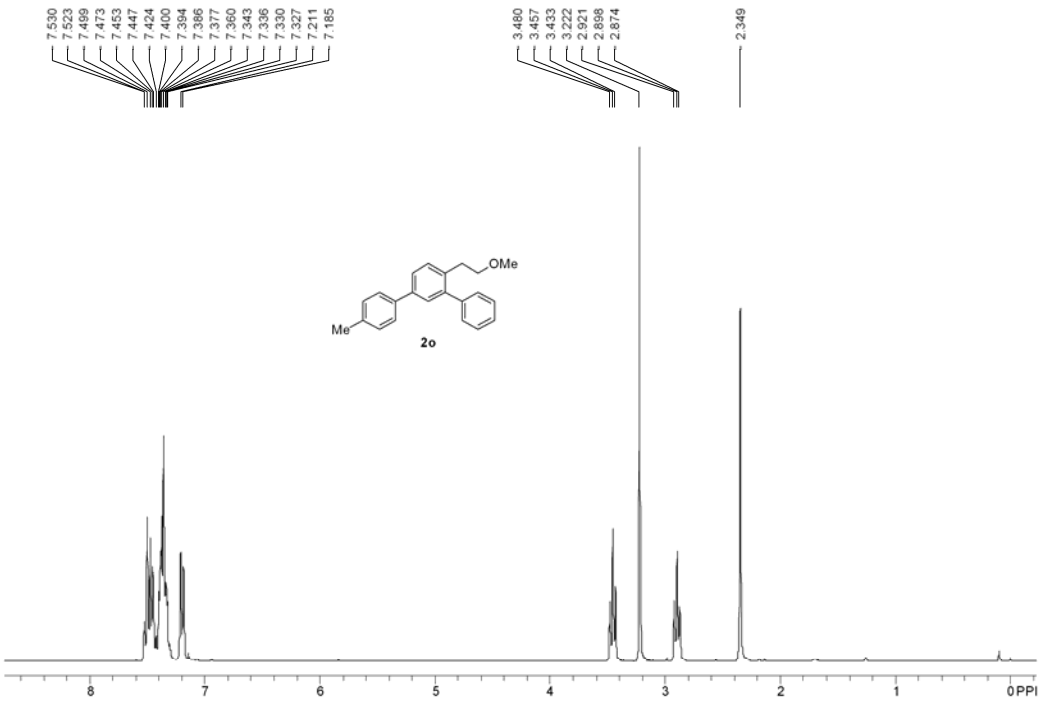
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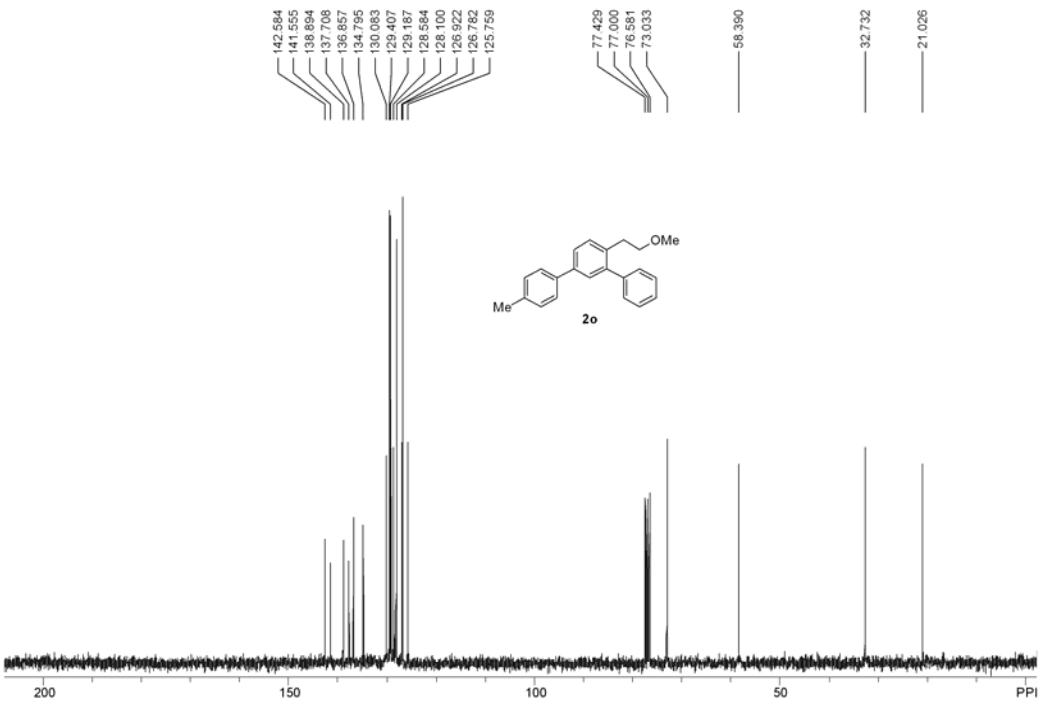
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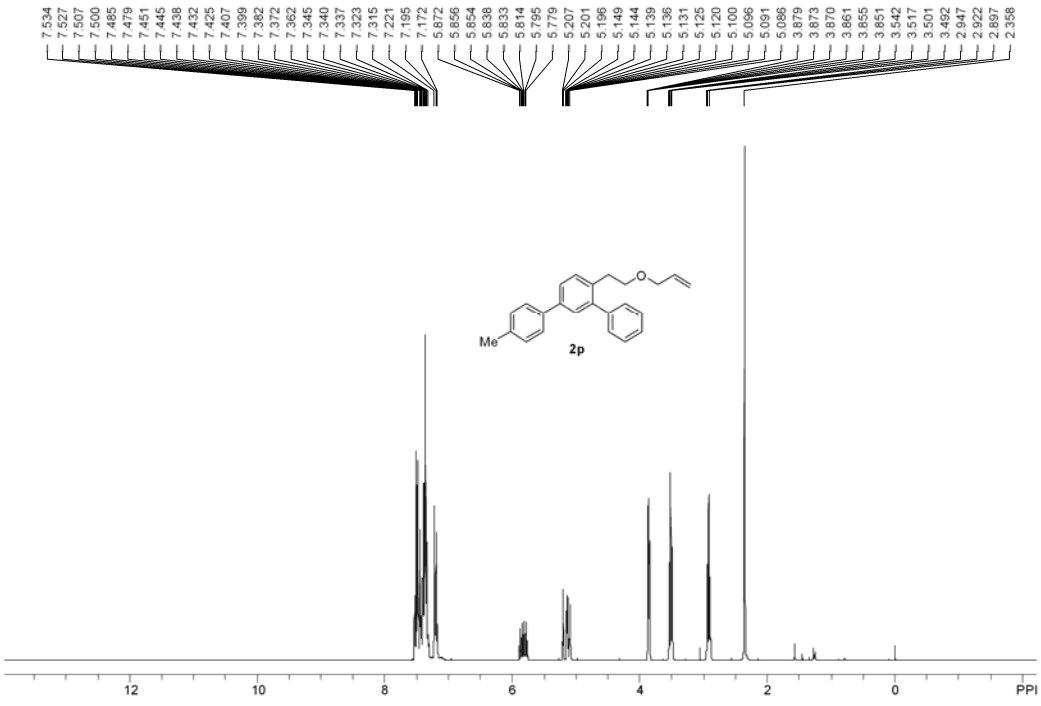
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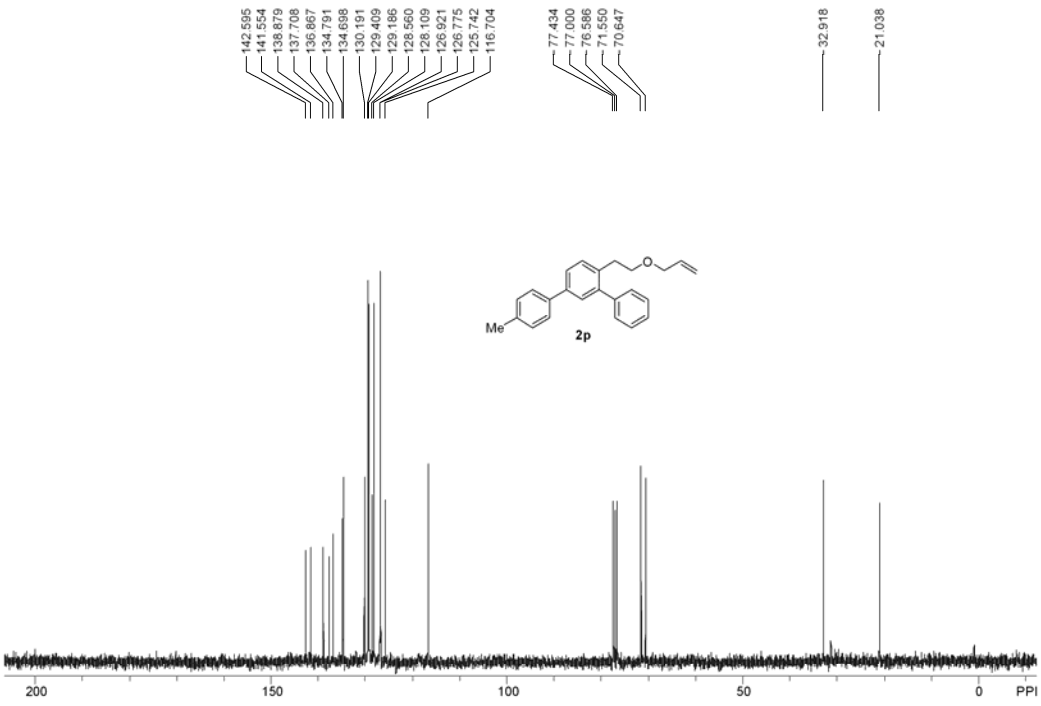
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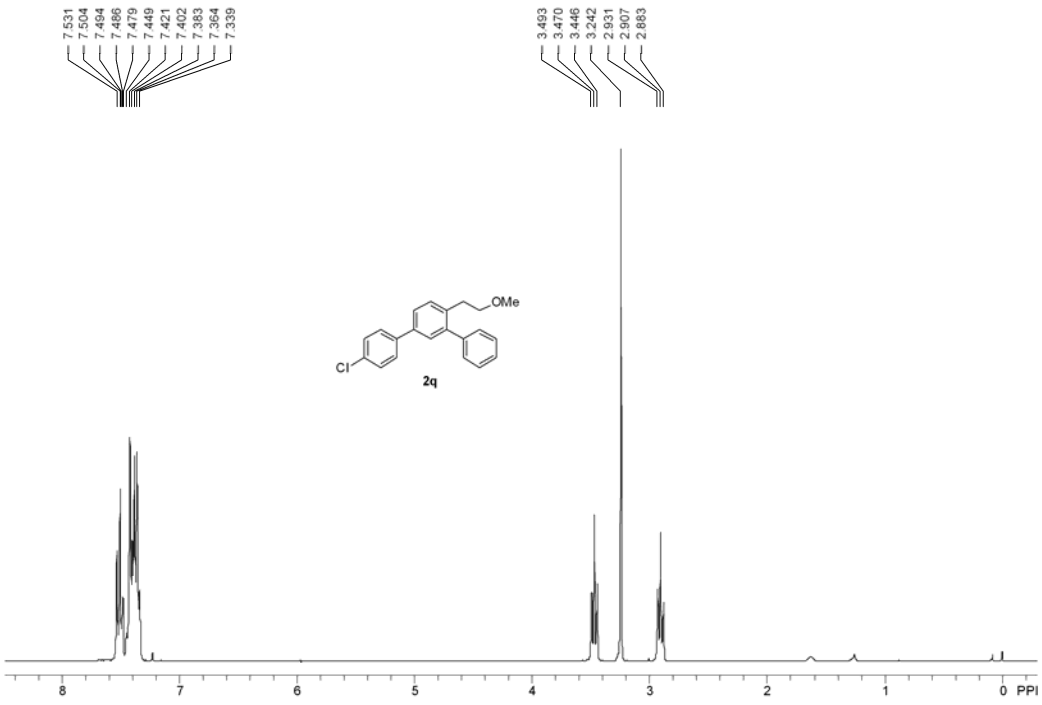
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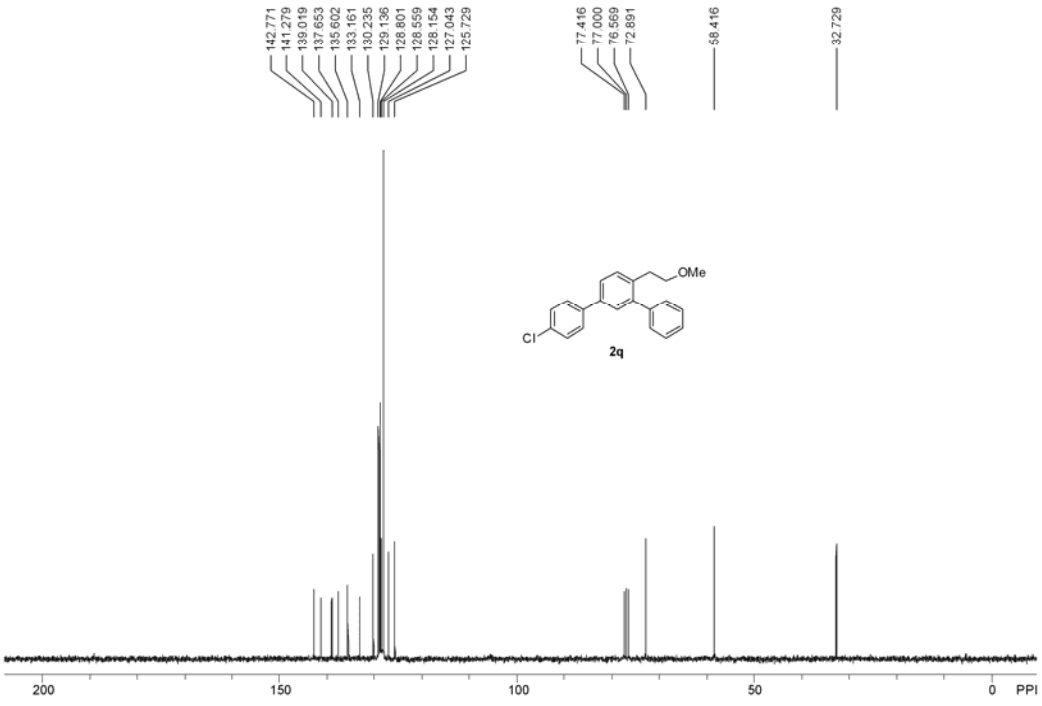
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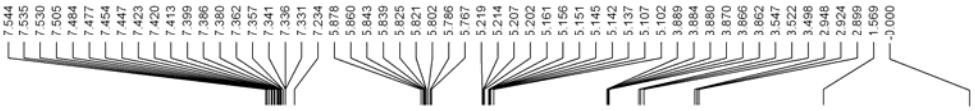
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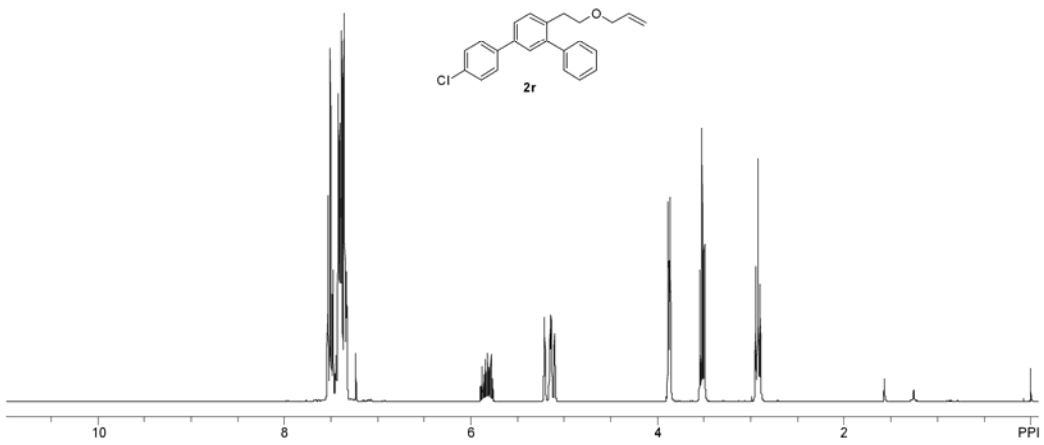
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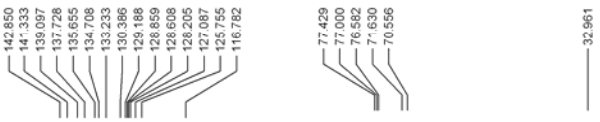
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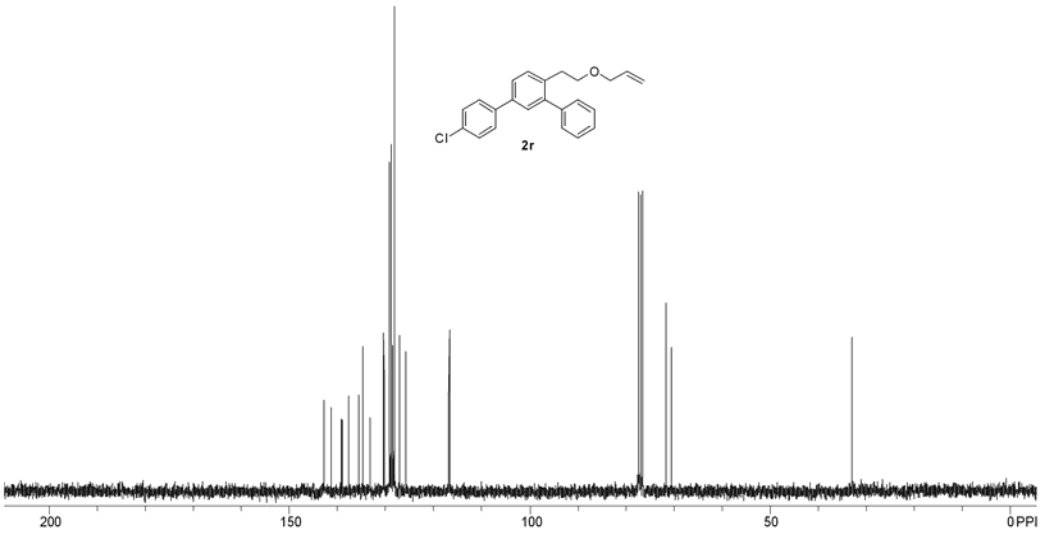
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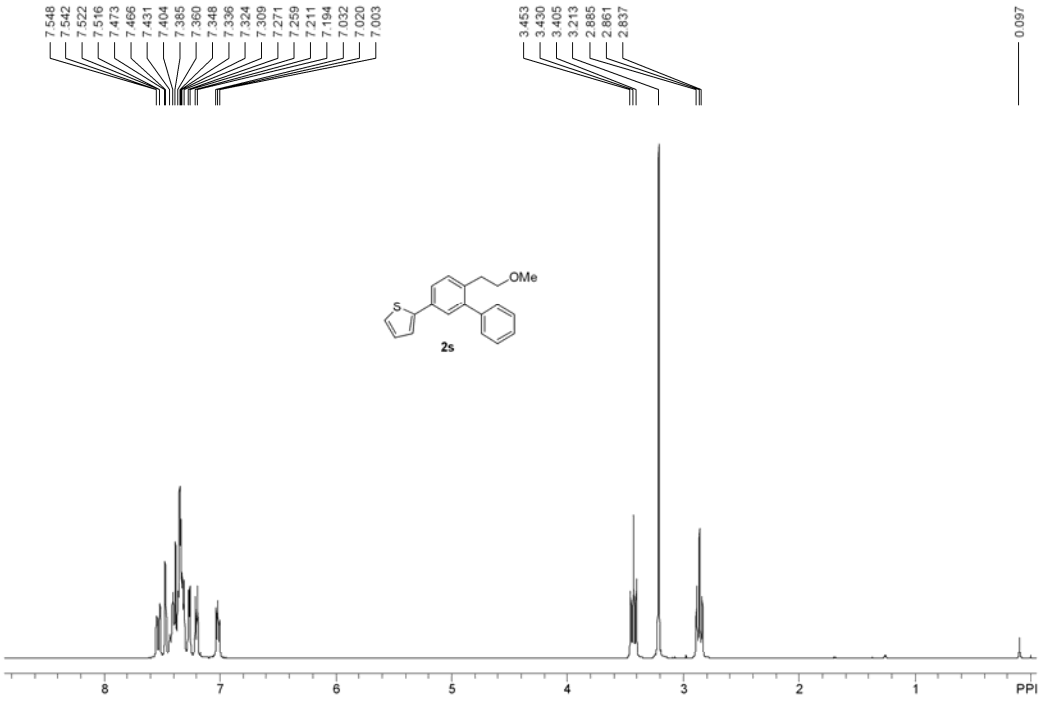
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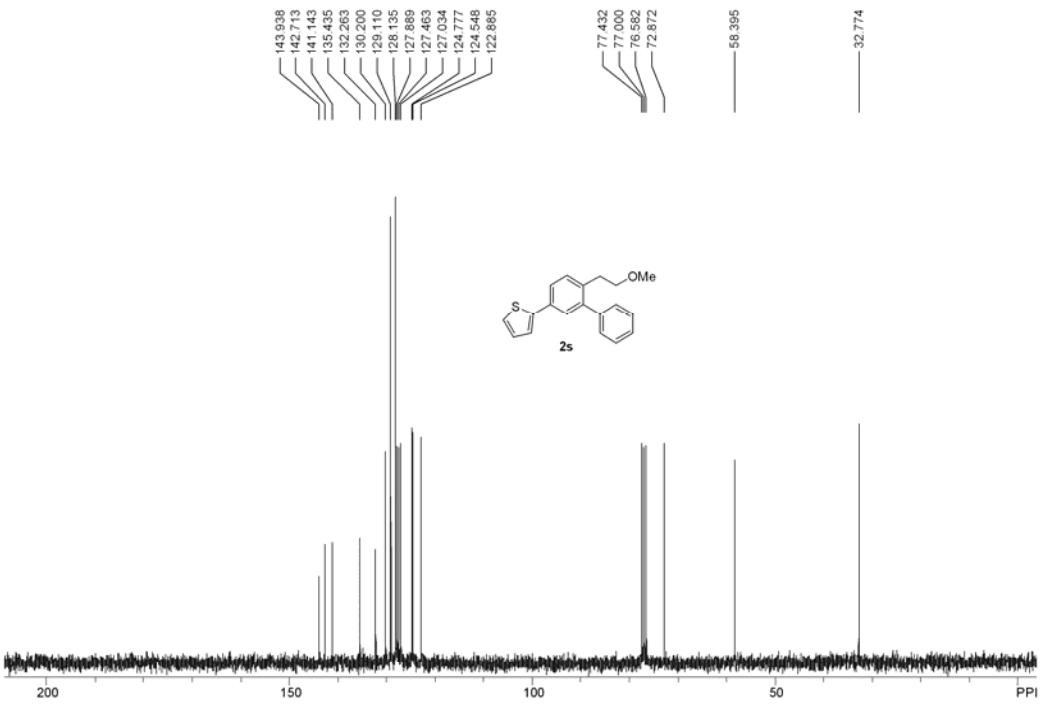
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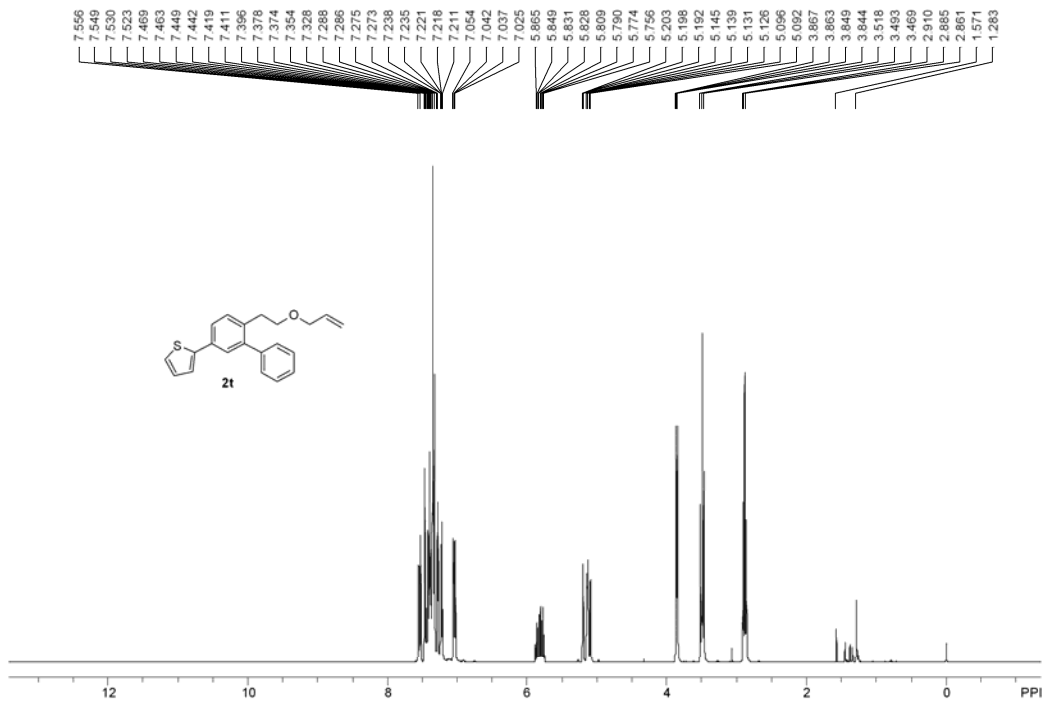
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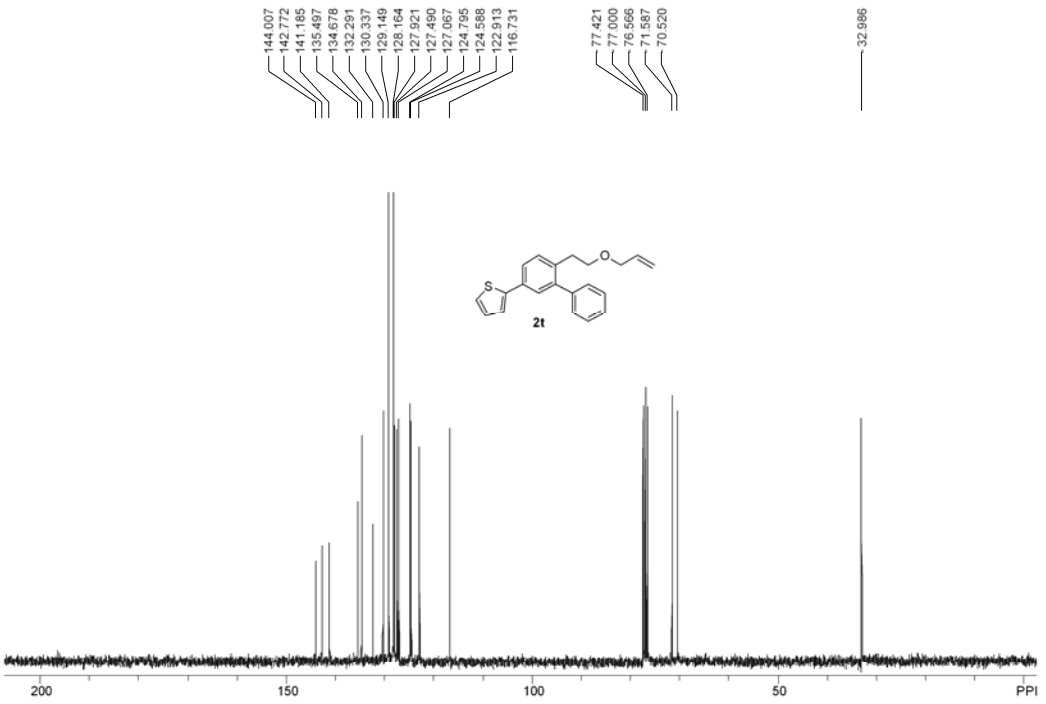
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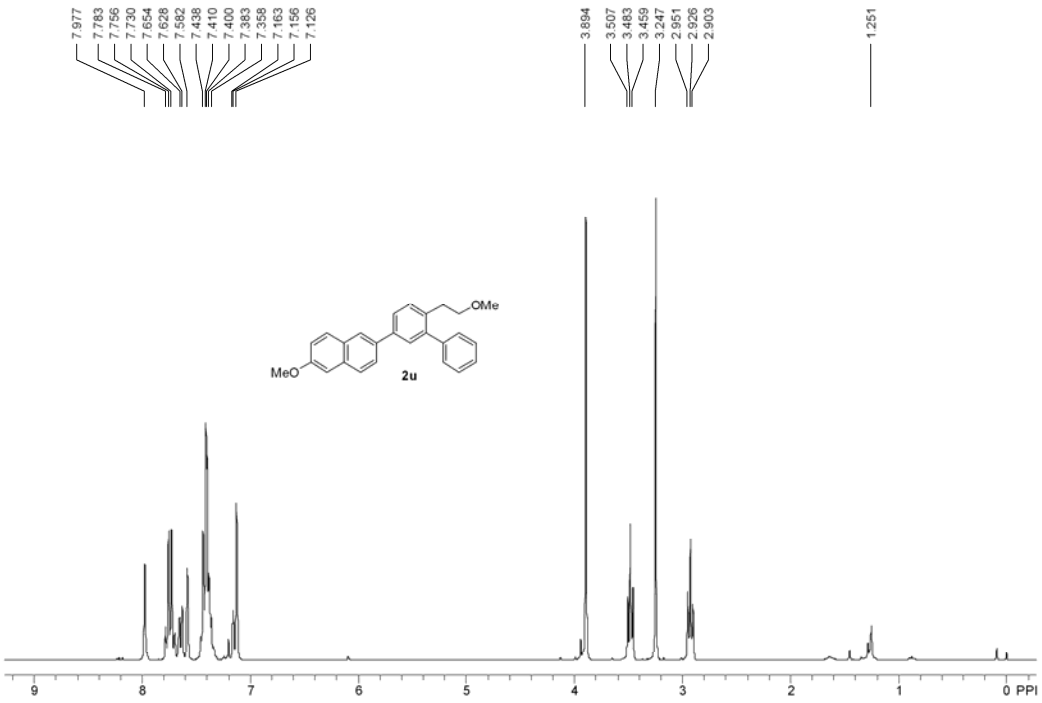
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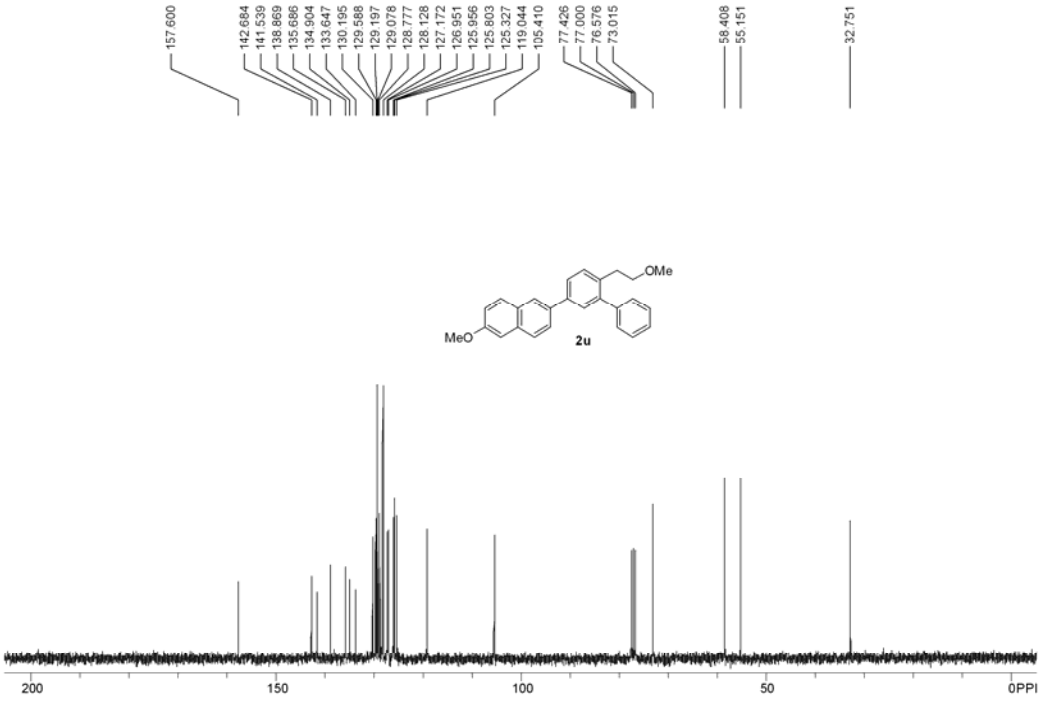
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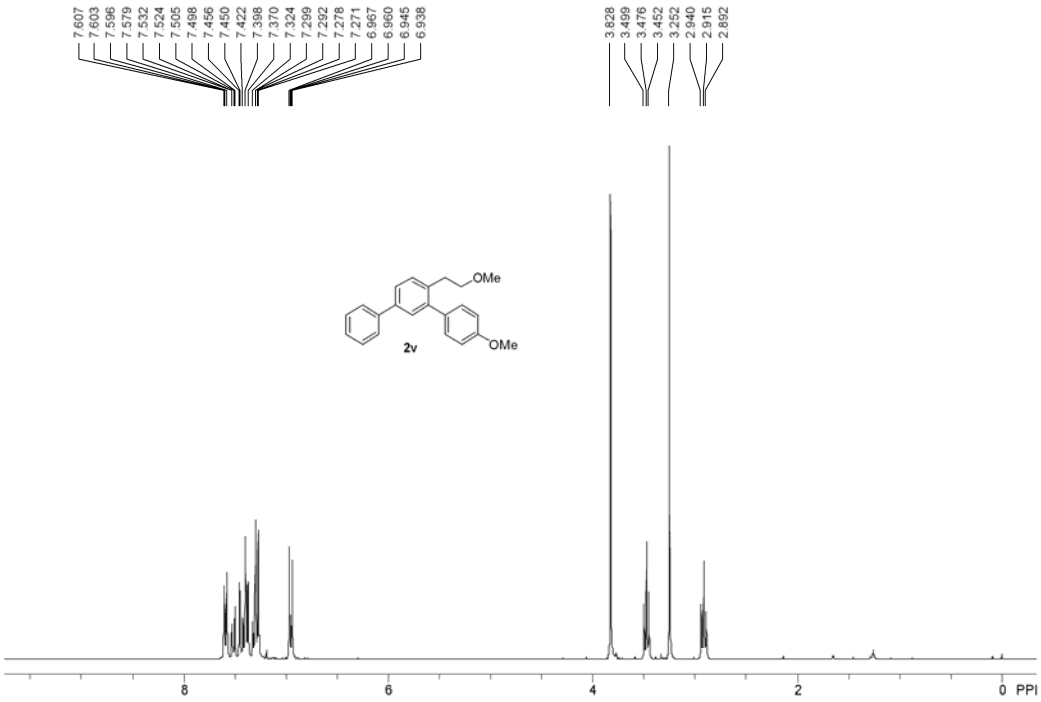
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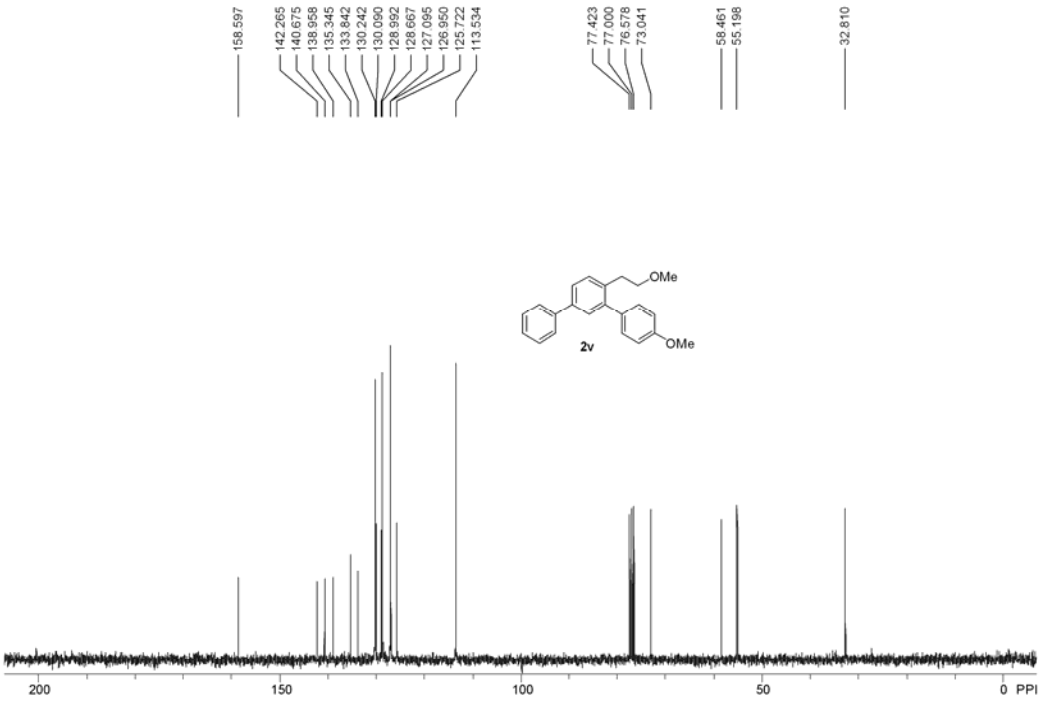
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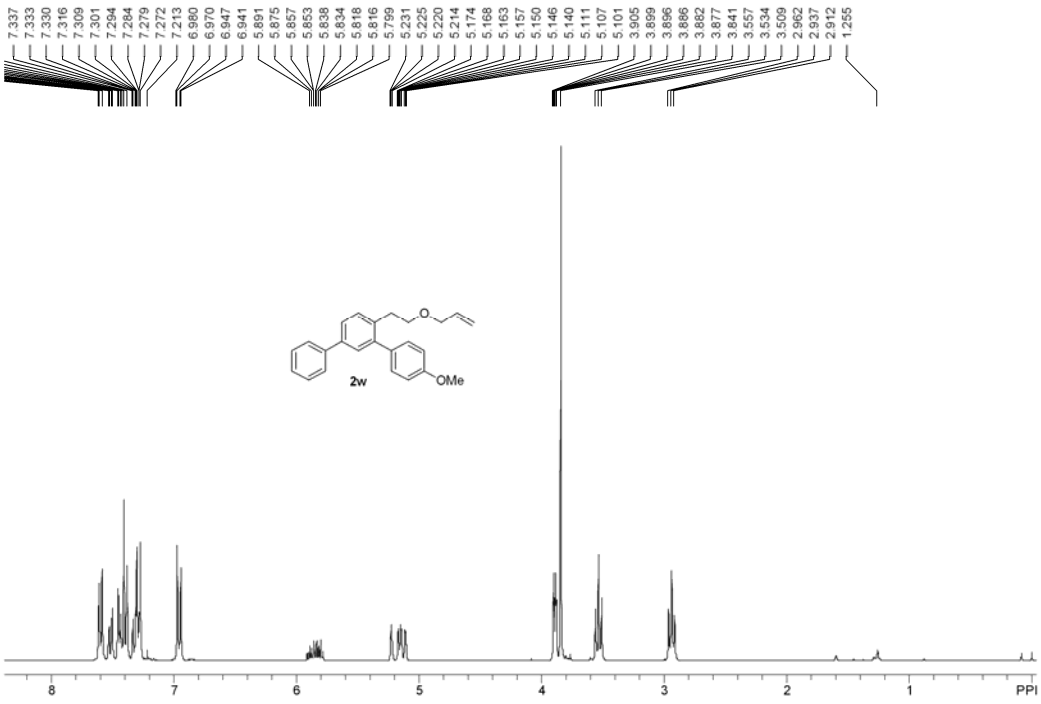
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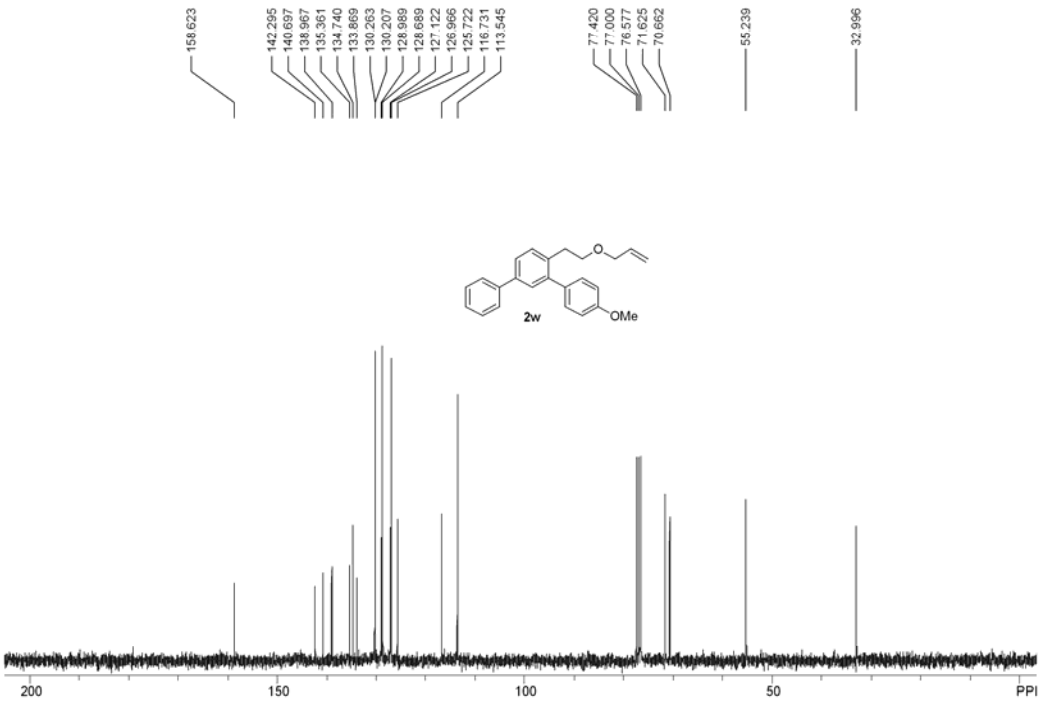
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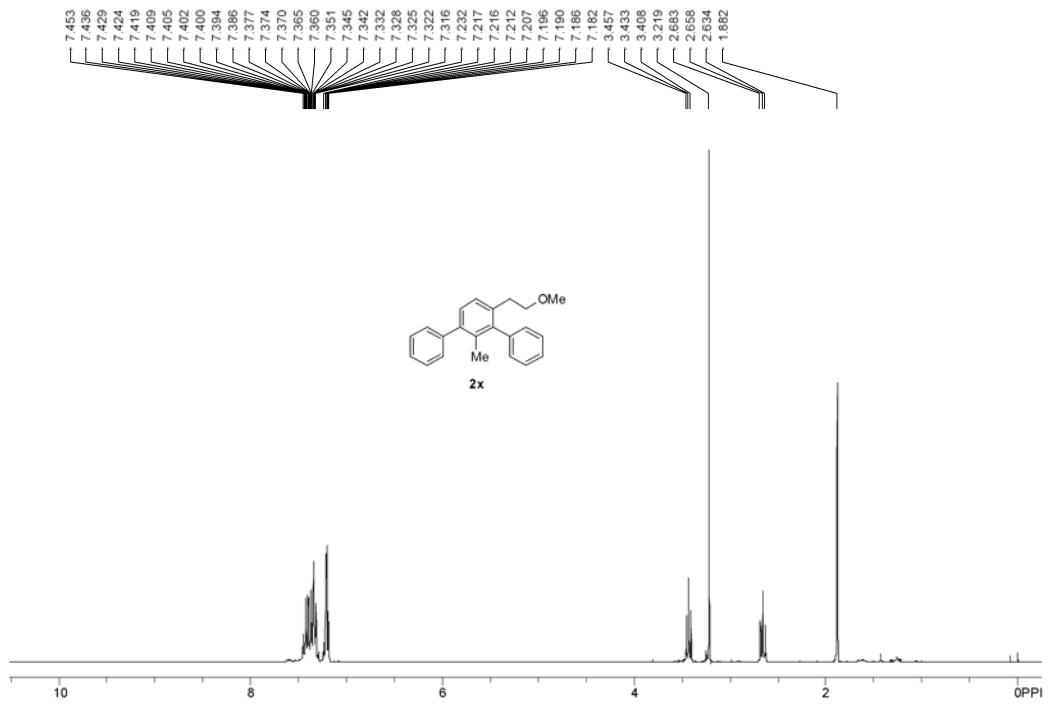
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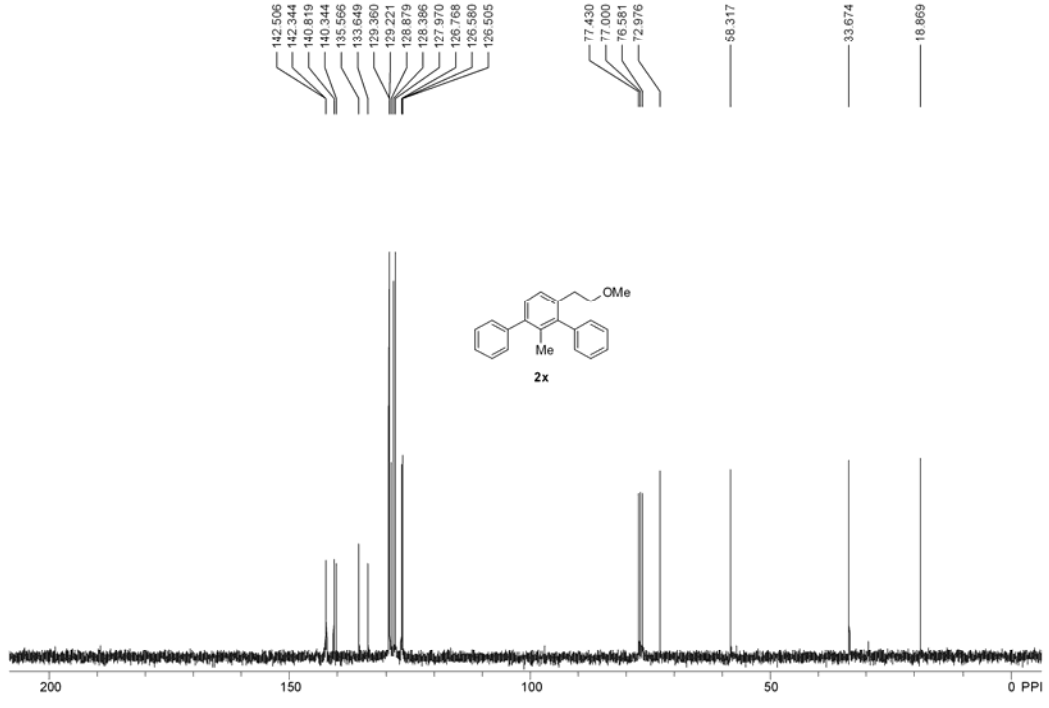
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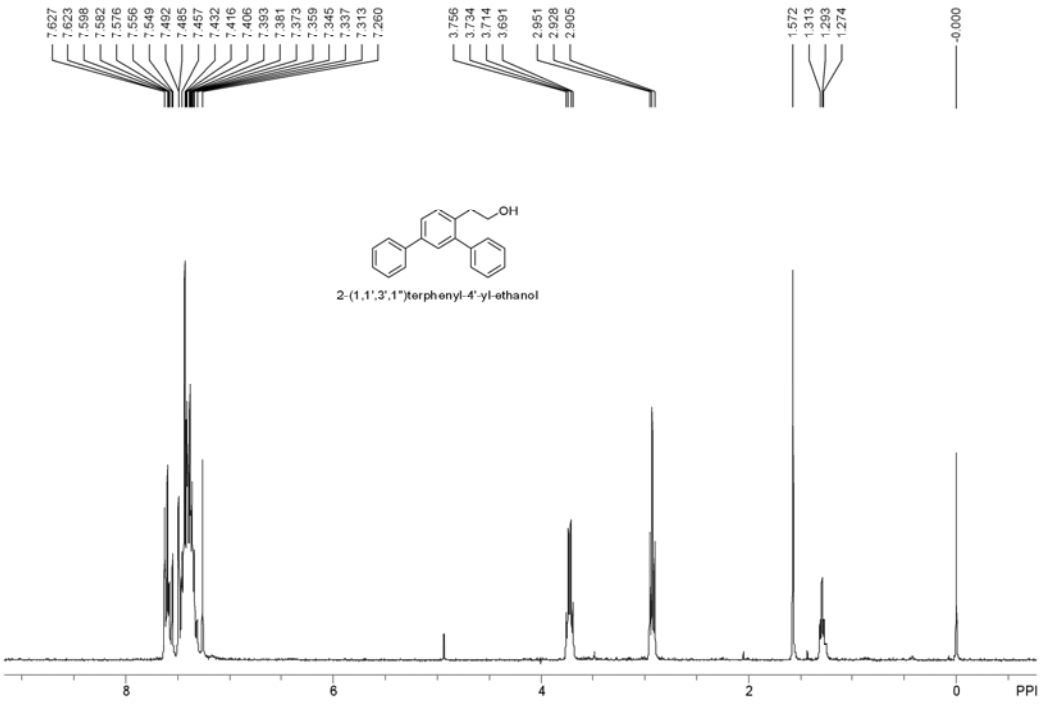
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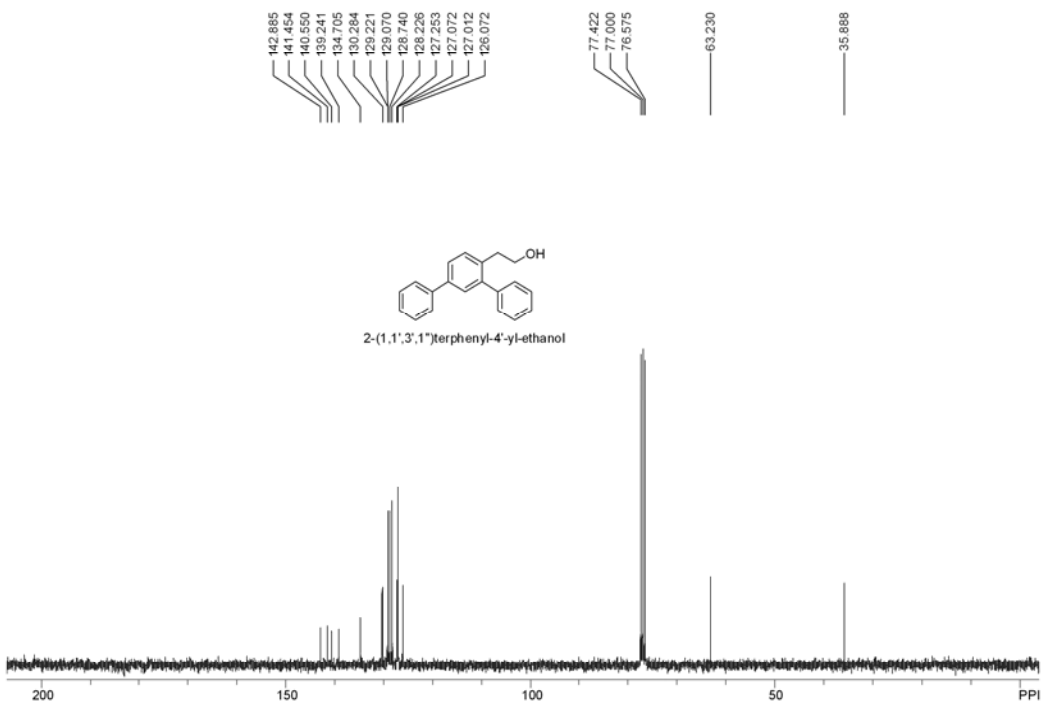
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35.888
OH
2-(1,1',3',1")terphenyl-4'-yl-ethanol
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150
100
50
PPI