Supporting Information

Substrate-dependent selectivity in $Sc(OTf)_3$-catalyzed cyclization of alkenoic acids and *N*-protected alkenamides

Hailong Zhang, Romain Carlino, Régis Guillot, Richard Gil *, Sophie Bezzenine * and Jérôme Hannedouche *

Institut de Chimie Moléculaire et des Matériaux d'Orsay (ICMMO), Site Henri Moissan, Université Paris-Saclay, CNRS, 91400 Orsay, France

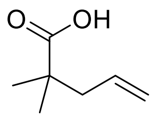
* Correspondence: richard.gil@universite-paris-saclay.fr (R.G.); sophie.bezzenine@universite-paris-saclay.fr (S.B.); jerome.hannedouche@universite-paris-saclay.fr (J.H.)

1-Preparation of starting materials

1.1-Preparation of alkenoic acids

Representative procedure for the preparation of 2,2-dimethylpent-4-enoic acid (1b):[1] To a solution of diisopropylamine (4.92 mL, 28.2 mmol) in THF (20 mL) at -78 °C was slowly added n-BuLi (2.5 M in hexane, 11.28 mL, 28.2 mmol), and the mixture was stirred at -78 °C for 0.5 h. To the solution was added isobutyric acid (0.82 g, 9.4 mmol) in THF (10 mL) and the mixture was stirred at 45 °C for 1.5 h. Allyl bromide (2.28 g, 18.8 mmol) was added to the solution at -78 °C and then the mixture was stirred at 45 °C overnight. H_2O was added to the mixture and the aqueous layer was washed with Et_2O. The aqueous layer was acidified with aq. HCl, and it was extracted with Et_2O three times. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated under vacuum. The residue was washed with pentane to give **1b**. Compounds **1c**, **1d**, **1f**, **1h**, **1i, 1l**, **1m**, **1p** were prepared according to the representative procedure for **1b**.

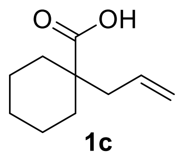
2,2-dimethylpent-4-enoic acid (1b)[1]



1b

Colorless liquid, 86% yield. ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 11.35 (b, 1H, OH), 5.85-5.66 (m, 1H, <u>CH</u>=CH_2), 5.06 (d, J = 12.7 Hz, 2H, CH=<u>CH_2</u>), 2.29 (d, J = 7.7 Hz, 2H, CH_2), 1.18 (s, 6H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 184.7, 134.0, 118.4, 44.5, 42.3, 24.7. HRMS (ESI) *m/z* calcd for $C_7H_{11}O_2$ $[M-H]^-$ 127.0765, found 127.0758. These data are in agreement with literature data.[1]

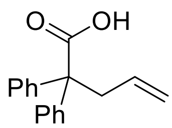
1-allylcyclohexane-1-carboxylic acid (1c)[1]



1c

Colorless liquid, 48% yield. ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 5.83-5.70 (m, 1H, <u>CH</u>=CH_2), 5.10-5.01 (m, 2H, CH=<u>CH_2</u>), 2.29 (d, J = 7.2 Hz, 1H, C<u>H_2</u>CH=CH_2), 2.09-2.00 (m, 2H, Cy-H), 1.86-1.51 (m, 3H, Cy-H), 1.47-1.35 (m, 2H, Cy-H), 1.33-1.19 (m, 3H, Cy-H). ^{13}C NMR (63 MHz, $CDCl_3$) δ (ppm) 183.1, 133.5, 118.1, 47.3, 44.5, 33.6, 26.9, 23.2. HRMS (ESI) *m/z* calcd for $C_{10}H_{16}O_2Na$ $[M+Na]^+$ 191.1043, found 191.1036. These data are in agreement with literature data.[1]

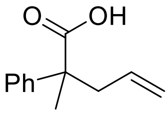
2,2-dimethylpent-4-enoic acid (1d)[2]



1d

White solid, 94% yield. m.p. 140-142 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.34-7.22 (m, 10H, Ar-H), 5.59 (ddt, J = 15.5, 11.9, 6.9 Hz, 1H, <u>CH</u>=CH_2), 4.93 (d, J = 15.5 Hz, 1H, CH=<u>CH_2</u>), 4.92 (d, J = 11.9 Hz, 1H, CH=<u>CH_2</u>), 3.16 (d, J = 6.9 Hz, 2H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 179.9, 142.2, 134.0, 129.2, 128.1, 127.3, 116.8, 60.4, 42.7. HRMS (ESI) *m/z* calcd for $C_{17}H_{16}O_2Na$ $[M+Na]^+$ 275.1043, found 275.1038. These data are in agreement with literature data.[2]

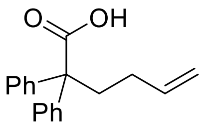
2-methyl-2-phenylpent-4-enoic acid (1f)[3]



1f

Yellow liquid, 68% yield. ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 10.65 (b, 1H, OH), 7.44 - 7.25 (m, 5H, Ar-H), 5.64 (ddt, J = 15.7, 10.8, 7.8 Hz, 1H, CH=CH_2), 5.11 (d, J = 15.7 Hz, 1H, CH=CH_2), 5.07 (d, J = 10.8 Hz 1H, CH=CH_2), 2.85 (dd, J = 14.1, 7.8 Hz, 1H, CH_2), 2.70 (dd, J = 14.1, 7.8 Hz, 1H, CH_2), 1.58 (s, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 182.2, 142.5, 134.0, 128.7, 127.2, 126.3, 118.9, 49.8, 43.6, 22.3. HRMS (ESI) m/z calcd for $C_{12}H_{14}O_2Na$ $[M+Na]^+$ 213.0886, found 213.0888. These data are in agreement with literature data.[3]

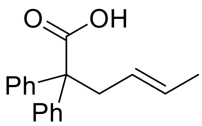
2,2-diphenylhex-5-enoic acid (1h)[4]



1h

White solid, 78% yield, m.p. 134-136 °C. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 7.45-7.20 (m, 10H, Ar-H), 5.86-5,68 (m, 1H, CH=CH_2), 4.96 (d, J = 18.8 Hz, 1H, CH=CH_2), 4.92 (d, J = 11.6 Hz, 1H, CH=CH_2), 2.54-2.40 (m, 2H, CH_2), 1.90-1.75 (m, 2H, CH_2). ^{13}C NMR (63 MHz, $CDCl_3$) δ (ppm) 180.9, 142.3, 138.3, 129.1, 128.1, 127.2, 114.8, 60.2, 37.2, 29.7. HRMS (ESI) m/z calcd for $C_{18}H_{18}O_2Na$ $[M+Na]^+$ 289.1199, found 289.1188. These data are in agreement with literature data.[4]

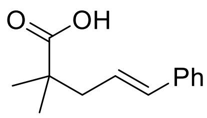
(*E*)-2,2-diphenylhex-4-enoic acid (1i)[5]



1i

White solid, 84% yield, (E:Z) isomer = 83:17. ^{1}H NMR (E isomer, 250 MHz, $CDCl_3$) δ (ppm) 7.41-7.18 (m, 10H, Ar-H), 5.35-5.18 (m, 2H, CH=CH_2), 3.08 (d, J = 5.6 Hz, 2H, CH_2), 1.50 (d, J = 5.1 Hz, 3H, CH_3). ^{13}C NMR (63 MHz, $CDCl_3$) δ (ppm) 179.7, 142.3, 129.5, 129.3, 127.9, 127.1, 126.1, 60.6, 41.5, 18.1). These data are in agreement with literature data.[5]

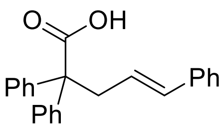
(*E*)-2,2-dimethyl-5-phenylpent-4-enoic acid (1l)[6]



1l

Light yellow solid, 38% yield. m.p. 172-174 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.38-7.22 (m, 5H, Ar-H), 6.45 (d, J = 15.8 Hz, 1H, CH=CH), 6.19 (dt, J = 15.6, 7.5Hz, 1H, CH=CH), 2.45 (d, J = 7.5 Hz, 2H, CH_2), 1.24 (s, 6H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 184.4, 137.5, 133.5, 128.6, 127.4, 126.3, 125.8, 43.6, 42.7, 24.9. HRMS (ESI) m/z calcd for $C_{13}H_{16}O_2Na$ $[M+Na]^+$ 227.1043, found 227.1040. These data are in agreement with literature data.[6]

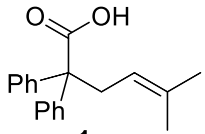
(*E*)-2,2,5-triphenylpent-4-enoic acid (1m)[6]



1m

Light yellow solid, 57% yield. m.p. 146-148 ℃, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.46-7.22 (m, 15H, Ar-H), 6.31 (d, J = 15.8 Hz, 1H, CH=CH), 6.03 (dt, J = 15.8, 7.0 Hz 1H, CH=CH), 3.39 (d, J = 7.0 Hz, 2H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 180.1, 142.0, 137.6, 133.7, 129.3, 128.5, 128.1, 127.3, 127.2, 126.2, 125.8, 60.8, 41.7. HRMS (ESI) *m/z* calcd for $C_{23}H_{20}O_2Na$ $[M+Na]^+$ 351.1356, found 351.1342. These data are in agreement with literature data.[6]

5-methyl-2,2-diphenylhex-4-enoic acid (1p)[4]

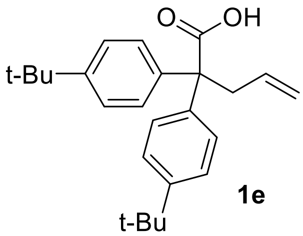


1p

White solid, 91% yield. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 7.40-7.11 (m, 10H, Ar-H), 5.13-4.99 (m, 1H, CH=C), 3.11 (d, J = 7.2 Hz, 2H, CH_2), 1.56 (s, 3H, CH_3), 1.26 (s, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 180.6, 142.2, 135.3, 129.2, 127.8, 126.9, 119.3, 60.6, 36.7, 25.9, 17.6. HRMS (ESI) *m/z* calcd for $C_{23}H_{20}O_2Na$ $[M+Na]^+$ 351.1356, found 351.1346.

Representative procedure for the preparation of 2,2-bis(4-(*tert*-butyl)phenyl)pent-4-enoic acid (1e). A solution of glyoxylic acid (50 mmol) and *tert*-butylbenzene (100 mmol) in acetic acid (100 mL) was cooled in an ice bath. Concentrated sulfuric acid (6 mL) was added dropwise under stirring. The solution was then stirred at 40 °C overnight. Then to the reaction was added water and the product was extracted by dichloromethane. The combined organic layer was washed with water, and brine and dried over Na_2SO_4, filtered, and concentrated under vacuum The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 5:1) to give the intermediate (5.04 mg, 37%) as a white solid. To a solution of diisopropylamine (4.92 mL, 28.2 mmol) in THF (20 mL) at -78 °C was slowly added *n*-BuLi (2.5 M in hexane, 11.28 mL, 28.2 mmol), and the mixture was stirred at -78 °C for 0.5 h. To the solution was added the intermediate (2.56 g, 9.4 mmol) in THF (10 mL), and the mixture was stirred at 45 °C for 1.5 h. Allyl bromide (3.70 g, 18.8 mmol) was added to the solution at -78 °C and then the mixture was stirred at 45 °C overnight. H_2O was added to the mixture and the aqueous layer was washed with Et_2O. The aqueous layer was acidified with $HCl_{aq.}$, and it was extracted with Et_2O. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated under vacuum. The solid residue was washed with pentane to give **1e**. Compounds **1n**, **1o**, **1r** and **1s** were prepared according to the representative procedure for **1e**.

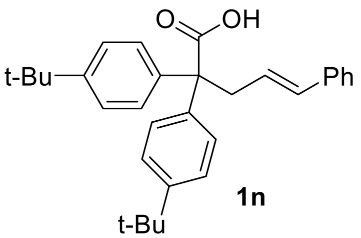
2,2-bis(4-(*tert*-butyl)phenyl)pent-4-enoic acid (1e)[7]



1e

White solid, 88% yield. m.p. 126-128 ℃, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.31 (d, 4H, J = 8.3 Hz, 4H, Ar-H), 7.22 (d, J = 8.3 Hz, 4H, Ar-H), 5.58 (ddt, J = 16.9, 10.2, 7.0 Hz, 1H, $CH=CH_2$), 4.98 (d, J = 16.9 Hz, 1H, $CH=CH_2$), 4.94 (d, J =10.2 Hz, 1H, $CH=CH_2$), 3.14 (d, J = 7.0 Hz, 2H, CH_2), 1.31 (s, 18H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 180.0, 149.8, 139.0, 134.4, 128.8, 124.9, 118.4, 59.5, 42.6, 34.6, 31.5. HRMS (ESI) *m/z* calcd for $C_{25}H_{32}O_2Na$ $[M+Na]^+$ 387.2295, found 387.2281. These data are in agreement with literature data.[7]

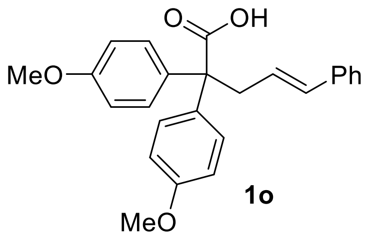
(*E*)-2,2-bis(4-(*tert*-butyl)phenyl)-5-phenylpent-4-enoic acid (1n)



1n

White solid, 81% yield. m.p. 218-220 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.34 (d, J = 8.7 Hz 4H, Ar-H), 7.30-7.23 (m, 6H, Ar-H), 7.22-7.16 (m, 3H, Ar-H), 6.22 (d, 1H, J = 15.8 Hz, CH=<u>CH</u>), 5.99 (dt, J = 15.8, 7.2 Hz, 1H, <u>CH</u>=CH), 3.30 (d, J = 7.2 Hz, 2H, CH_2), 1.34 (s, 18H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 179.6, 149.9, 139.0, 137.8, 133.5, 128.8, 128.5, 127.1, 126.3, 126.2, 124.9, 60.1, 41.8, 34.6, 31.5. HRMS (ESI) *m/z* calcd for $C_{31}H_{36}O_2Na$ [M+Na]$^+$ 463.2608, found 463.2585.

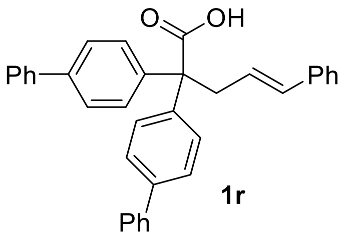
(*E*)-2,2-bis(4-methoxyphenyl)-5-phenylpent-4-enoic acid (1o)



1o

Yellow solid, 68% yield. m.p. 142-144 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.32-7.16 (m, 9H, Ar-H), 6.87 (d, 4H, J = 8.9 Hz, Ar-H), 6.29 (d, 1H, J = 16.1 Hz, CH=<u>CH</u>), 5.9 (dt, 1H, J = 7.0, 16.1, Hz, <u>CH</u>=CH), 3.84 (s, 6H, OCH_3), 3.28 (d, 2H, J = 7.0 Hz, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 180.3, 158.6, 137.7, 134.3, 133.6, 130.3, 128.5, 127.2, 126.2, 126.1, 113.4, 59.4, 55.4, 42.0. HRMS (ESI) *m/z* calcd for $C_{25}H_{24}O_4Na$ [M+Na]$^+$ 411.1567, found 411.1551.

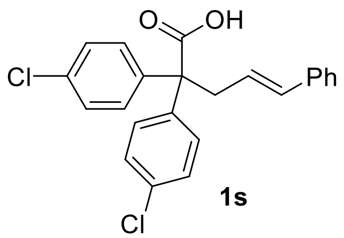
(*E*)-2,2-di([1,1'-biphenyl]-4-yl)-5-phenylpent-4-enoic acid (1r)



1r

White solid, 19% yield. m.p. 100-102 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.64-7.54 (m, 8H, Ar-H), 7.48-7.41 (m, 8H, Ar-H),), 7.37-7.32 (m, 2H, Ar-H),), 7.25-7.13 (m, 5H, Ar-H), 6.31 (d, 1H, J = 15.8 Hz, CH=<u>CH</u>), 6.02 (dt, J = 15.8, 7.1 Hz 1H, <u>CH</u>=CH), 3.38 (d, 2H, J = 7.1 Hz, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 178.9, 141.0, 140.6, 140.1, 137.6, 134.0, 129.7, 128.9, 128.6, 127.5, 127.3, 127.2, 126.8, 126.3, 125.6, 60.4, 41.8. HRMS (ESI) *m/z* calcd for $C_{35}H_{28}O_2Na$ [M+Na]$^+$ 503.1982, found 503.1957.

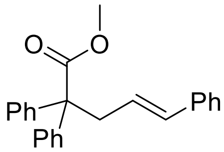
(*E*)-2,2-bis(4-chlorophenyl)-5-phenylpent-4-enoic acid (1s)



1s

Orange solid, 37% yield. m.p. 184 - 186 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.37-7.16 (m, 13H, Ar-H), 6.26 (d, 1H, J = 15.4 Hz, CH=<u>CH</u>-Ph), 5.90 (dt, 1H, J = 15.4 Hz, 7.1 Hz, CH_2-<u>CH</u>=CH), 3.26 (d, 2H, J = 7.1 Hz, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 178.9, 140.2, 137.3, 134.6, 133.6, 130.6, 128.7, 128.4, 127.6, 126.2, 124.6, 60.0, 41.7. HRMS (ESI) *m/z* calcd for $C_{23}H_{18}Cl_2O_2Na$ $[M+Na]^+$ 419.0576, found 419.0564.

Procedure for the preparation of methyl (*E*)-2,2,5-triphenylpent-4-enoate (1q)[5]



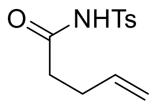
1q

To a solution of 2,2,5-triphenylpent-4-enoic acid (**1m**, 0.328 g, 1 mmol) in acetone (3 mL) at 25 °C was added K_2CO_3 in one portion, and the mixture was stirred at 25 °C for 20 min. To the solution was added iodomethane (0.85 g, 6 mmol), and the mixture was stirred at 25 °C for 18 h. After the reaction, the volatiles were removed under decreased pressure, then H_2O was added to the mixture and it was extracted with EtOAc. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated under vacuum to give **1q** (0.32g, 0.93 mmol, 94% yield). ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.46 – 7.20 (m, 15H, Ar-H), 6.32 (d, J = 15.8 Hz, 1H, CH=<u>CH</u>), 6.05 (dt, J = 15.8 Hz, 7.3 Hz, 1H, <u>CH</u>=CH), 3.78 (s, 3H, CH_3), 3.40 (d, J = 7.3 Hz, 2H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 174.6, 142.6, 137.7, 133.5, 129.1, 128.5, 128.0, 127.1, 127.0, 126.1, 60.9, 52.5, 42.0. HRMS (ESI) *m/z* calcd for $C_{24}H_{22}O_2Na$ $[M+Na]^+$ 365.1512, found 365.1500. These data are in agreement with literature data.[5]

1.2- Preparation of alkenamides

Representative procedure for the preparation of *N*-tosylpent-4-enamide (5d):[8] To a solution of **1a** (1.56 g, 15.6 mmol) in THF (30 mL) was added *p*-toluenesulfonyl isocyanate (2.4 mL, 15.6 mmol), and the mixture was stirred at room temperature for 10 min. To the solution was added triethylamine (2.2 mL, 15.6 mmol) and the mixture was stirred at room temperature for 2 h. Aqueous HCl was added to the mixture, and extraction with EtOAc was done. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated under vacuum. The residue was subjected to short column chromatography on silica gel with EtOAc/hexane (1/5) as an eluent to give **5d**. Compounds **5a, 5b**, **5c**, and **5e** were prepared according to the representative procedure for **5d**.

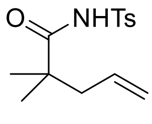
***N*-tosylpent-4-enamide (5d)**[1]



5d

White solid, 75% yield. m.p. 89-91 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 8.25 (b, 1H, NH), 7.94 (d, J = 8.5 Hz, 2H, Ar), 7.35 (d, J = 8.5 Hz, 2H, Ar), 5.72 (ddt, J = 16.6, 10.7, 6.8 Hz 1H, <u>CH</u>=CH_2), 4.99 (d, J = 16.6 Hz, 1H, CH=<u>CH_2</u>), 4.98 (d, J = 10.7 Hz, 1H, CH=<u>CH_2</u>), 2.45 (s, 3H, CH_3), 2.38 – 2.28 (m, 4H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 170.0, 145.4, 135.9, 135.6, 129.8, 128.5, 116.6, 35.6, 28.3, 21.9. HRMS (ESI) *m/z* calcd for $C_{12}H_{15}O_3NSNa$ $[M+Na]^-$ 276.0665, found 276.0662. These data are in agreement with literature data.[1]

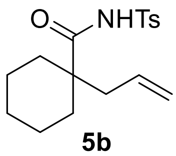
2,2-dimethyl-*N*-tosylpent-4-enamide (5a)[1]



5a

White solid, 62% yield. m.p. 148-150 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 8.76 (b, 1H, NH), 7.94 (d, *J* = 8.3 Hz, 2H, Ar-H), 7.33 (d, *J* = 8.3 Hz, 2H, CH_2), 5.59-5.47 (m, 1H, CH=CH_2), 4.96 (dd, *J* = 8.3, 1.5 Hz, 1H, CH=CH_2), 4.95 (dd, *J* = 18.0, 1,5 Hz, 1H, CH=CH_2) 2.44 (s, 3H, CH_3), 2.18 (d, *J* = 7.3 Hz, 2H, CH_2), 1.12 (s, 6H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 175.3, 145.1, 135.5, 132.9, 129.6, 128.5, 119.1, 44.5, 43.5, 24.4, 21.8. HRMS (ESI) *m/z* calcd for $C_{14}H_{19}NO_3SNa$ $[M+Na]^+$ 304.0978, found 304.0979. These data are in agreement with literature data.[1]

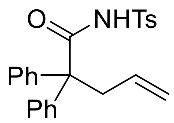
1-allyl-*N*-tosylcyclohexane-1-carboxamide (5b)[1]



5b

White solid, 55% yield. m.p. 148-150 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 8.47 (b, 1H, NH), 7.94 (d, *J* = 8.6 Hz, 2H, Ar-H), 7.33 (d, *J* = 8.6 Hz, 2H, Ar-H), 5.51 - 5.39 (m, 1H, CH=CH_2), 4.91 (dd, *J* = 10.3, 1,3 Hz, 1H, CH=CH_2), 4.86 (dd, *J* = 16.9, 1,3 Hz, 1H, CH=CH_2), 2.44 (s, 3H, CH_3), 2.13 (d, *J* = 7.6Hz, 2H, CH_2), 1.90-1.85 (m, 2H, CH_2), 1.55-1.46 (m, 3H, 1CH_2, CHH), 1.36-1.20 (m, 5H, 2CH_2, CHH). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 174.2, 145.1, 135.7, 132.1, 129.6, 128.7, 119.1, 48.1, 44.0, 33.6, 25.7, 22.7, 21.8. HRMS (ESI) *m/z* calcd for $C_{17}H_{24}NO_3S$ $[M+H]^+$ 322.1471, found 322.1463. These data are in agreement with literature data.[1]

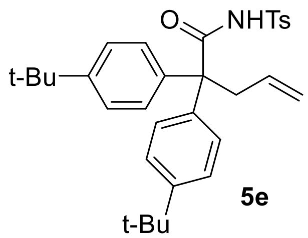
2,2-diphenyl-*N*-tosylpent-4-enamide (5c)[5]



5c

White solid, 82% yield. m.p. 118-120 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.78 (b, 1H, NH), 7.66 (d, *J* = 8.4 Hz, 2H, Ar-H), 7.24-7.18 (m, 8H, Ar-H), 7.07-7.04 (m, 4H, Ar-H), 5.50-5.39 (m, 1H, CH=CH_2), 4.80 (dd, *J* = 8.5, 1.9 Hz, 1H, CH=CH_2), 4.78 (dd, *J* = 18.6, 1,9 Hz, 1H, CH=CH_2), 2.98 (d, *J* = 6.8 Hz, 2H, CH_2), 2.37 (s, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 171.5, 145.1, 140.2, 135.0, 133.5, 129.54, 128.8, 128.5, 127.8, 119.2, 61.6, 42.8, 21.8. HRMS (ESI) *m/z* calcd for $C_{24}H_{24}NO_3S$ $[M+H]^+$ 406.1471, found 406.1461. These data are in agreement with literature data.[5]

2,2-bis(4-(*tert*-butyl)phenyl)-*N*-tosylpent-4-enamide (5e)

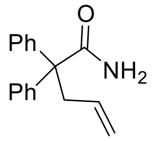


5e

White solid, 60% yield. m.p. 176-178 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.86 (b, 1H, NH), 7.7 (d, *J* = 7.2, 2H, Ar-H), 7.33-7.26 (m, 5H, Ar-H), 7.26-7.20 (m, 1H, Ar-H), 7.08 (m, 4H, Ar-H), 5.62-5.46 (m, 1H, CH=CH_2), 4.92-4.87 (m, 2H, CH=CH_2), 3.05 (d, *J* = 7.2Hz, 2H, CH_2), 2.46 (s, 3H, CH_3), 1.34 (s, 18H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 171.9, 150.7, 145.0, 137.2, 135.2, 133.7, 129.5, 128.7, 128.6, 125.7, 124.9, 118.9, 60.9, 42.7, 31.4, 31.3, 21.8. HRMS (ESI) *m/z* calcd for $C_{32}H_{40}NO_3S$ $[M+H]^+$ 518.2729, found 518.2704.

Representative procedure for the preparation of compound 5i:[9] 2,2-diphenylpent-4-enoic acid (3.85 g 15.3 mmol) was dissolved in dry dichloromethane (30 mL), followed by addition of two drops of DMF and $SOCl_2$ (1.5 mL, 20.7 mmol). After 2 h, the volatiles and solvent were evaporated and the resulting acyl chloride was obtained. To the acyl chloride was then added NH_4OH (35% in aqueous solution, 27 mL, 270 mmol), and the resulting mixture was stirred for 16 h at r.t. and extracted with ethyl acetate. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 5:1) to give **5i**.

2,2-diphenylpent-4-enamide (5i)[10]

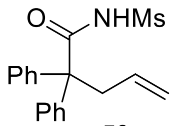


5i

White solid, 86% yield. m.p. 78-80 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.27-7.18 (m, 10H, Ar-H), 5.68 (ddt, J = 17.1, 10.3, 6,8 Hz, 1H, CH=CH_2), 5.48 (b, 1H, NH), 4.96-4.87 (m, 2H, CH=CH_2), 3.16-3.13 (dt, J = 6.8, 1.3 Hz, 2H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 176.8, 142.9, 135.1, 129.1, 128.4, 127.2, 118.1, 60.6, 43.3. HRMS (ESI) m/z calcd for $C_7H_{18}NO$ $[M+H]^+$ 252.1383, found 252.1377. These data are in agreement with literature data.[10]

Representative procedure for the preparation of compound 5f. 2,2-diphenylpent-4-enoic acid (3.85 g 15.3 mmol) was dissolved in dry dichloromethane (30 mL), followed by addition of two drops of DMF and $SOCl_2$ (1.5 mL, 20.7 mmol). After 2 h, the volatiles and solvent were evaporated and the resulting acyl chloride was obtained. To the acyl chloride was then added methanesulfonamide (1.75 g, 18.4 mmol), and the resulting mixture was stirred for 16 h at r.t. and extracted with ethyl acetate. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 5:1) to give **5f**. Compounds **5g, 5h, 5j**, and **5k**, were prepared according to the representative procedure from **5f**.

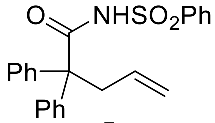
***N*-(methylsulfonyl)-2,2-diphenylpent-4-enamide (5f)**[8]



5f

Colourless oil, 46% yield. ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.71 (b, 1H, NH), 7.36-7.21 (m, 10H, Ar-H), 5.65-5.54 (m, 1H, CH=CH_2), 4.97-4.92 (m, 2H, CH=CH_2), 3.16 (d, J = 7,5 Hz, 2H, CH_2), 3.15 (s, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 173.2, 140.4, 133.5, 129.0, 128.9, 128.0, 119.4, 61.7, 42.9, 41.4. HRMS (ESI) m/z calcd for $C_{18}H_{19}NO_3NaS$ $[M+Na]^+$ 352.0978, found 352.0965. These data are in agreement with literature data.[8]

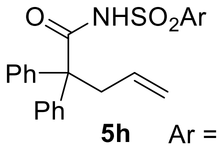
2,2-diphenyl-*N*-(phenylsulfonyl)pent-4-enamide (5g)



5g

White solid, 66% yield. m.p. 148 - 150 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.86 (d, J = 7.5 Hz 2H, Ar-H), 7.65 (t, J = 7.5 Hz, 1H, Ar-H), 7.50 (t, J = 7.8 Hz, 2H, Ar-H), 7.31-7.24 (m, 6H, Ar-H), 7.16-7.11 (m, 4H, Ar-H), 5.56-5.46 (ddt, J = 16.9, 10.3, 7 Hz, 1H, CH=CH_2), 4.89-4.83 (m, 2H, CH=CH_2), 3.06 (d, J = 7.0 Hz, 2H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 171.5, 140.2, 138.0, 134.0, 133.4, 128.9, 128.5, 127.9, 119.2, 61.7, 42.8. HRMS (ESI) m/z calcd for $C_{23}H_{21}NO_3NaS$ $[M+Na]^+$ 414.1134, found 414.1113.

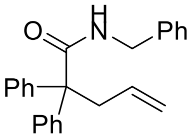
***N*-((4-methoxyphenyl)sulfonyl)-2,2-diphenylpent-4-enamide (5h)**



5h Ar = 4-MeO-C_6H_4

White solid, 49% yield. m.p. 148- 52 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.72 (d, J = 8.6 Hz, 1H, Ar-H), 7.36 (d, J = 8.6 Hz, 1H, Ar-H). 7.20-7.12 (m, 10H, Ar-H), 5.51-5.40 (m, 1H, CH=CH_2), 4.82-4.78 (m, 2H, CH=CH_2), 4.78 (s, 3H, CH_3) 3.03 (d, J = 7.0 Hz, 2H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 171.5, 164.0, 140.3, 133.5, 130.9, 129.4, 128.9, 127.9, 119.2, 114.1, 61.6, 55.9, 42.8. HRMS (ESI) *m/z* calcd for $C_{23}H_{21}NO_3NaS$ $[M+Na]^+$ 444.1245, found 444.1258.

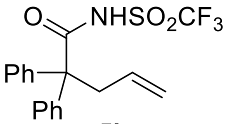
***N*-benzyl-2,2-diphenylpent-4-enamide (5j)**[10]



5j

White solid, 89% yield. m.p. 138-140 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.20-7.05 (m, 13H, Ar-H), 6.94-6.91 (m, 2H, Ar-H), 5.70 (b, 1H, NH), 5.61 (ddt, J = 17.1, 10.2, 6.8 Hz 1H, CH=CH_2), 4.92-4.84 (m, 2H, CH=CH_2), 4.30 (d, J = 5.6 Hz, 2H, NHCH_2), 3.17 (d, J = 6.8 Hz, 2H, CH_2CH=CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 174.1, 143.0, 138.3, 135.3, 129.2, 128.7, 128.4, 127.5, 127.4, 127.2, 118.0, 60.8, 44.0, 43.5. HRMS (ESI) *m/z* calcd for $C_{24}H_{24}NO$ $[M+H]^+$ 342.1852, found 342.1842. These data are in agreement with literature data.[10]

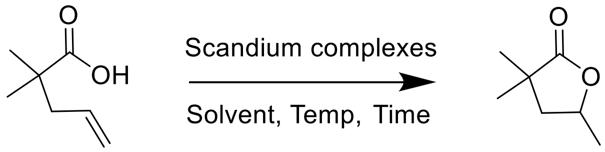
2,2-diphenyl-*N*-((trifluoromethyl)sulfonyl)pent-4-enamide (5k)



5k

White solid, 64% yield. m.p. 70-76 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.20-7.15 (m, 10H, Ar-H), 5.54 (ddt, J = 15.3, 12.0, 6.9 Hz, 1H, CH=CH_2), 4.87 (d, J = 15,3 Hz, 2H, CH=CH_2), 4.86 (d, J = 12.0 Hz, 2H, CH=CH_2), 3.09 (d, J = 6.9 Hz, 2H, CH_2), 1.73 (b, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 184.1, 143.0, 134.9, 129.3, 128.0, 126.8, 117.9, 63.2, 43.0. HRMS (ESI) *m/z* calcd for $C_{23}H_{21}NO_3NaS$ $[M+Na]^+$ 406.0695, found 406.0685.

2-Optimization studies for the $Sc(OTf)_3$-catalyzed cyclization of alkenoic acid 1b by heating



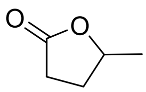
1b **2b**

Entry	catalyst	Equiv (%)	Solvent	Temp	Time	Conv (%)	Yield (%)[a]
1	-	-	Tol	110°C	16 h	0	0
2	CF_3COOH	10	Tol	110°C	16 h	0	0
3	$Sc(OAc)_3$	10	Tol	110°C	16 h	0	0
4	$ScCl_3$	10	Tol	110°C	16 h	0	0
5	**$Sc(OTf)_3$**	**10**	**Tol**	**110°C**	**16 h**	**99**	**91**
6	$Sc(OTf)_3$	5	Tol	110°C	16 h	69	46
7	$Sc(OTf)_3$	1	Tol	110°C	16 h	12	4
8	$Sc(OTf)_3$	10	H_2O	100°C	16 h	0	0
9	$Sc(OTf)_3$	10	1,4-dioxane	101°C	16 h	93	78
10	$Sc(OTf)_3$	10	EtOH	78°C	16 h	62	0
11	$Sc(OTf)_3$	10	CH_3CN	82°C	16 h	32	26
12	$Sc(OTf)_3$	10	DCE	84°C	16 h	87	59
13	$Sc(OTf)_3$	10	Tol	80°C	16 h	24	22
14	$Sc(OTf)_3$	10	Tol	40°C	16 h	0	0
18	$Sc(OTf)_3$	10	Tol	110°C	8h	81	59

Table S1. Optimization studies for the $Sc(OTf)_3$-catalyzed cyclization of **1b**. [a] yields were determined by ^1H NMR using 1,3,5-trimethoxybenzene as internal standard.

3-Cyclization Products

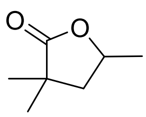
5-methyldihydrofuran-2(3*H*)-one (2a)[11]



2a

Colorless oil, 82 mg, 79% isolated yield. ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 4.64-4.55 (m, 1H, CH), 2.56-2.44 (m, 2H, CH_2), 2.39-2.25 (m, 1H, CH_2), 1.85-1.71 (m, 1H, CH_2), 1.36 (d, J = 6.2 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 177.3, 77.3, 29.7, 29.1, 21.0. HRMS (ESI) m/z calcd for $C_5H_9O_2$ $[M+H]^+$ 101.0597, found 101.0598. These data are in agreement with literature data.[11]

3,3,5-trimethyldihydrofuran-2(3*H*)-one (2b)[12]

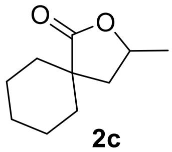


2b

Colorless oil, 88 mg, 69% isolated yield (under thermal conditions) and 80 mg, 62% yield (under microwave conditions). ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 4.74-4.60 (m, 1H, CH), 2.20 (dd, J = 12.8 Hz, 5.7 Hz, 1H, CH_2), 1.74 (dd, J = 12.8 Hz, 9.8Hz, 1H, CH_2), 1.40 (d, J = 6.1 Hz, 3H, CH_3), 1.27

(s, 3H, CH_3), 1.25 (s, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 182.7, 73.9, 45.2, 41,1, 25.1, 24.3, 21.1. HRMS (ESI) *m/z* calcd for $C_7H_{13}O_2$ $[M+H]^+$ 129.0910, found 129.0907. These data are in agreement with literature data.[12]

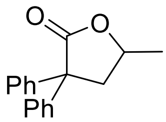
3-methyl-2-oxaspiro[4.5]decan-1-one (2c)[12]



2c

Slightly yellow solid, 70 mg, 83% isolated yield. m.p. 66-68 °C, ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 4.51 (pd, J = 6.1, 3.8 Hz, 1H, CH), 2.37 (dd, J = 12.5, 6.1 Hz, 1H, CH_2), 1.81 (dd, J = 12.5, 3.8 Hz, 1H, CH_2), 1.78-1.65 (m, 2H, Cy-CH_2), 1.63-1.51 (m, 4H, CH_2, Cy-CH_2), 1.50-1.42 (m, 1H, CH_2, Cy-CH_2), 1.37 (d, J = 6.1 Hz, 3H, CH_3), 1.34-1.15 (m, 3H, Cy-CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 181.7, 737, 45.5, 41.3, 34.4, 31.6, 25.4, 22.2, 22.2, 21.5. HRMS (ESI) *m/z* calcd for $C_{10}H_{17}O_2$ $[M+H]^+$ 169.1223, found 169.1218. These data are in agreement with literature data. [12]

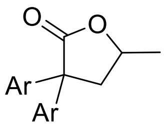
5-methyl-3,3-diphenyldihydrofuran-2(3*H*)-one (2d)[12]



2d

White solid, 156 mg, 62% yield (under thermal conditions) and 174 mg, 69% yield (under microwave conditions). m.p. 110-112 °C. ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.36-7.10 (m, 10H, Ar-H), 4.48-4.34 (m, 1H, CH), 2.99 (dd, J = 12.9, 4.4 Hz, 1H, CH_2), 2.52 (dd, J = 12.9, 11.1 Hz, 1H, CH_2), 1.39 (d, J = 6 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 177.3, 142.2, 140.1, 129.0, 128.5, 127.8, 127.6, 127.5, 127.3, 73.7, 58.8, 45.5, 20.5. HRMS (ESI) *m/z* calcd for $C_{17}H_{17}O_2$ $[M+H]^+$ 253.1223, found 253.1222. These data are in agreement with literature data.[12]

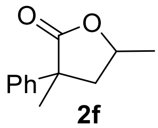
3,3-bis(4-(*tert*-butyl)phenyl)-5-methyldihydrofuran-2(3*H*)-one (2e)



2e Ar = 4-t-BuC_6H_4

Slightly yellow solid, 37 mg, 51% isolated yield. m.p. 165-167 °C, ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.31-7.12 (m, 8H, Ar-H), 4.46-4.35 (m, 1H, CH), 2.97 (dd, J = 12.8, 4.7 Hz, 1H, CH_2), 2.47 (dd, J = 12.8, 10.6 Hz, 1H, CH_2), 1.38 (d, J = 6.2 Hz, 3H, CH_3), 1.23 (s, 9H, CH_3), 1.21 (s, 9H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 177.8, 150.6, 149.9, 139.4, 136.7, 127.4, 127.1, 125.9, 125.3, 73.7, 58.1, 45.7, 34.6, 34.5, 31.4, 20.5. HRMS (ESI) *m/z* calcd for $C_{25}H_{33}O_2$ $[M+H]^+$ 365.2481, found 365.2460.

3,5-dimethyl-3-phenyldihydrofuran-2(3*H*)-one (2f)

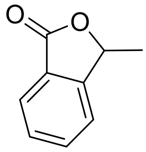


2f

Yellow oil, 89 mg, 94% isolated yield, mixture of 2 diastereomers. **2f-(diastereomer 1)** 56 mg, 59% isolated yield. ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.38-7.25 (m, 5H, Ar-H), 4.40-4.28 (m, 1H, CH), 2.78 (dd, J = 12.9, 4.9 Hz, 1H, CH_2), 1.97 (dd, J = 12.9, 10.7 Hz, 1H, CH_2), 1.59 (s, 3H, CH_3), 1.41 (d, J = 6.1 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 179.6, 141.1, 129.0, 127.5, 125.8, 73.7, 50.5, 46.1, 26.5, 20.5. **2f-(diastereomer 2)** 33 mg, 35% isolated yield. ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.51-7.45 (m, 2H, Ar-H), 7.40-7.33 (m, 2H, Ar-H), 7.31-7.24 (m, 1H, Ar-H), 4.71 (pd,

J =8,9, 6.1 Hz 1H, CH), 2.55-2.53 (dd, J = 12.8, 6.1 Hz, 1H, CH_2), 2.29 (dd, J = 12.8, 8.9 Hz, 1H, CH_2), 1.64 (s, 3H, CH_3), 1.40 (d, J = 6.2 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 179.7, 142.9, 128.8, 127.2, 126.2, 73.7, 48.7, 46.0, 25.4, 21.2. HRMS (ESI) *m/z* calcd for $C_{12}H_{15}O_2$ $[M+H]^+$ 191.1067, found 191.1063.

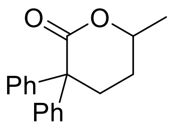
3-methylisobenzofuran-1(3*H*)-one (2g)[13]



2g

Colorless oil, 89% yield. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 7.90 (d, J = 7.6 Hz, 1H), 7.68 (td, J = 7.5, 1.0 Hz, 1H), 7.53 (t, J = 7.4 Hz, 1H), 7.44 (d, J = 7.6 Hz, 1H), 5.57 (q, J = 6.7 Hz, 1H), 1.64 (d, J = 6.7 Hz, 3H). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 170.5, 151.3, 134.1, 129.1, 125.8, 127.7, 121.7, 77.8, 20.5. HRMS (ESI) *m/z* calcd for $C_9H_9O_2$ $[M+H]^+$ 149.0601, found 149.0597. These data are in agreement with literature data.[13]

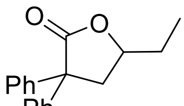
6-methyl-3,3-diphenyltetrahydro-2*H*-pyran-2-one (2h)[5]



2h

White solid, 73% yield. mp = 101 °C, ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 7.46-7.17 (m, 10H), 4.45-4.14 (m, 1H), 2.83-2.73 (m, 1H), 2.69-2.62 (m, 1H), 1.98-1.93 (m, 1H), 1.79-1.68 (m, 1H), 1.38 (d, J = 7.0 Hz, 3H). These data are in agreement with literature data.[5]

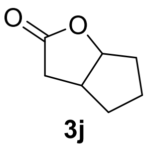
5-ethyl-3,3-diphenyldihydrofuran-2(3*H*)-one (2i)[5]



2i

Colorless oil, 74% yield. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 7.51-7.14 (m, 10H), 4.39-4.22 (m, 1H), 2.92-2.78 (m, 1H), 2.72-2.55 (m, 1H), 1.89-1.66 (m, 2H), 1.05 (t, J = 7.4 Hz, 3H). These data are in agreement with literature data.[5]

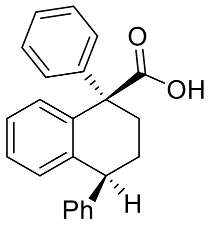
Hexahydro-2*H*-cyclopenta[b]furan-2-one (3j)[14]



3j

Colorless oil, 79% yield. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 5.00 (t, J = 5.6 Hz, 1H), 2.93-2.74 (m, 2H), 2.28 (d, J = 15.6 Hz, 1H), 2.09-1.98 (m, 1H), 1.92-1.61 (m, 4H), 1.59-1.46 (m, 1H). These data are in agreement with literature data.[14]

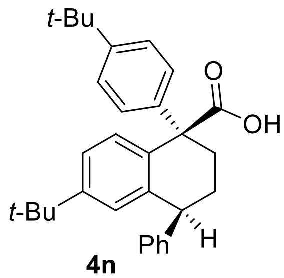
1,4-diphenyl-1,2,3,4-tetrahydronaphthalene-1-carboxylic acid (4m)



4m

White solid, 91% yield (under thermic conditions) and 58% yield (under microwave conditions). m.p. 203-205 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 11.75 (b, 1H, OH), 7.33-7.13 (m, 13H, Ar-H), 6.98-6.96 (m, 1H, Ar-H), 4.22 (t, J = 6.5 Hz, 1H, CH), 2.88-2.83 (m, 1H, CH_2), 2.13-1.91 (m, 3H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 181.0, 147.1, 145.7, 140.3, 131.2, 130.6, 129.0, 128.5, 128.4, 128.1, 127.4, 126.7, 126.2, 57.8, 45.8, 34.6, 29.8. HRMS (ESI) *m/z* calcd for $C_{23}H_{20}O_2Na$ $[M+Na]^+$ 351.1355, found 351.1351.

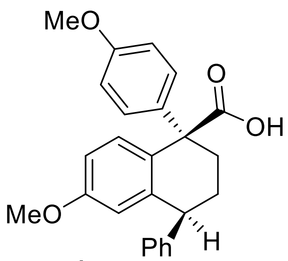
6-(tert-butyl)-1-(4-(tert-butyl)phenyl)-4-phenyl-1,2,3,4-tetrahydronaphthalene-1-carboxylic acid (4n)



4n

White solid, 86 mg, 98% isolated yield. m.p. 230-232 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 11.63 (b, 1H, OH), 7.35-7.28 (m, 5H, Ar-H), 7.24 (td, J = 7.8, 1.9, 2H, Ar-H), 7.17 (d, J = 7.3 Hz, 2H, Ar-H), 7.07 (d, J = 8,7 Hz, 2H, Ar-H), 6.99 (d, J = 1.9 Hz, 1H, Ar-H), 4.23 (t, J = 6.4 Hz, 1H, CH), 2.85-2.78 (m, 1H, CH_2), 1.98-1.69 (m, 3H, CH_2), 1.35 (s, 9H, CH_3), 1.24 (s, 9H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 181.3, 150.1, 149.4, 147.3, 142.4, 139.6, 134.0, 130.6, 129.1, 128.4, 128.0, 127.5, 126.1, 125.0, 123.5, 56.7, 45.8, 34.5, 34.2, 31.5, 31.4, 29.7. HRMS (ESI) *m/z* calcd for $C_{31}H_{36}O_2Na$ $[M+Na]^+$ 463.2613, found 463.2585.

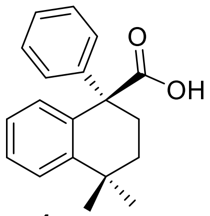
6-methoxy-1-(4-methoxyphenyl)-4-phenyl-1,2,3,4-tetrahydronaphthalene-1-carboxylic acid (4o)



4o

Slightly yellow solid, 73 mg, 95% isolated yield. m.p. 176-178 °C, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 11.72 (b, 1H, OH), 7.32-7.20 (m, 4H, Ar-H), 7.15 (d, J = 7.5 Hz, 2H, Ar-H), 7.05 (d, J = 8.3 Hz, 2H, Ar-H), 6.84 (d, J = 8.3 Hz, 2H, Ar-H), 6.76 (dd, J = 8.7, 2.5 Hz, 1H, Ar-H), 6.47 (d, J = 2.3 Hz, 1H, Ar-H), 4.16 (t, J = 6.2 Hz, 1H, CH), 3.80 (s, 3H, OCH_3), 3.68 (s, 3H, OCH_3), 2.83-2.76 (m, 1H, CH_2), 2.05-1.99 (m, 2H, CH_2), 1.95-1,86 (m, 1H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 181.7, 158.6, 158.3, 146.8, 141.7, 137.7, 132.2, 129.4, 129.1, 128.5, 126.3, 114.9, 113.5, 112.9, 56.2, 55.4, 55.2, 45.9, 34.4, 29.5. HRMS (ESI) *m/z* calcd for $C_{25}H_{24}O_4Na$ $[M+Na]^+$ 411.1567, found 411.1561.

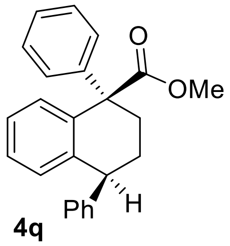
4,4-dimethyl-1-phenyl-1,2,3,4-tetrahydronaphthalene-1-carboxylic acid (4p)



4p

Colourless oil, 90% yield. ^{1}H RMN (400 MHz, $CDCl_3$) δ (ppm) 10.25 (b, 1H, COOH), 7.35 (d, 1H, *J* = 7.7 Hz, Ar-H), 7.22-7.20 (m, 6H, Ar-H), 7.02 (t, 1H, *J* = 7.2 Hz, Ar-H), 6.96 (d, 2H, *J* = 7.2 Hz, Ar-H), 2.70-2.62 (m 1H, CH_2), 2.10-2.03 (m, 1H, CH_2), 1.66-1.58 (m, 1H, CH_2), 1.44-1.37 (m, 1H, CH_2), 1.28 (s, CH_3), 1.21 (s, CH_3). ^{13}C RMN (100.6 MHz, $CDCl_3$) δ (ppm) 181.6, 146.9, 145.6, 135.1, 131.2, 128.4, 128.1, 127.8, 127.0, 126.7, 125.6, 57.9, 35.2, 34.1, 33.3, 32.1 31.8. HRMS (ESI) *m/z* calcd for $C_{19}H_{20}O_2Na$ $[M+Na]^+$ 303.1356 found 303.1353.

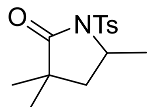
Methyl 1,4-diphenyl-1,2,3,4-tetrahydronaphthalene-1-carboxylate (4q)



4q

Colourless oil, 93% yield, ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.35-7.20 (m, 7H, Ar-H), 7.17-7.14 (m, 4H, Ar-H), 7.09-7.07 (m, 2H, Ar-H), 6.96-6.93 (m, 1H, Ar-H), 4.22 (dd, *J* = 8.1, 6.1 Hz, 1H, CH), 3.83 (s, 3H, CH_3), 2.90-2.83 (m, 1H, CH_2), 2.13-2.05 (m, 2H, CH_2), 1.97-1.87 (m, 1H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 175.7, 147.2, 146.2, 140.5, 137.7, 131.4, 130.5, 129.0, 128.5, 128.2, 127.3, 126.7, 126.2, 126.1, 57.8, 52.7, 45.9, 35.1, 30.1. HRMS (ESI) *m/z* calcd for $C_{24}H_{23}O_2$ $[M+H]^+$ 343.1693, found 343.1678.

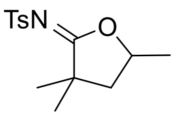
3,3,5-trimethyl-1-tosylpyrrolidin-2-one (6a)[15]



6a

White solid, 41 mg, 28% isolated yield. m.p. 87-89 °C, IR (neat, cm^{-1}) ν 1727 (C=O). ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.92 (d, *J* = 8.4 Hz, 2H, Ar-H), 7.31 (d, *J* = 8.4 Hz, 2H, Ar-H), 4.34-4.25 (m, 1H, CH), 2.42 (s, 3H, CH_3), 2.13 (dd, *J* = 13.0, 7.9 Hz, 1H, CH_2), 1.58-1.52 (m, 1H, CH_2), 1,55 (d, *J* = 6.1 Hz, 3H, CH_3), 1.14 (s, 3H, CH_3), 1.00 (s, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 179.1, 144.9, 136.1, 129.6, 128.3, 53.3, 42.4, 41.3, 25.3, 23.1, 21.8. HRMS (ESI) *m/z* calcd for $C_{14}H_{20}NO_3S$ $[M+H]^+$ 282.1158, found 282.1155. These data are in agreement with literature data.[15]

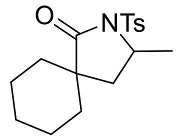
4-methyl-*N*-(3,3,5-trimethyldihydrofuran-2(3*H*)-ylidene)benzenesulfonamide (7a)[16]



7a

Yellow solid, 89 mg, 61% isolated yield. m.p. 143-145 °C. IR (neat, cm^{-1}) ν 1618 (C=N). ^{1}H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.85 (d, *J* = 8.2 Hz, 2H, Ar-H), 7.26 (d, *J* = 8.2 Hz, 2H, Ar-H), 4.84-4.75 (m, 1H, CH), 2.41 (s, 3H, CH_3), 2.13 (dd, *J* = 12.8, 5.8 Hz, 1H, CH_2), 1.64 (dd, *J* = 12.7, 10.1 Hz, 1H, CH_2), 1.40 (d, *J* = 6.2 Hz, 3H, CH_3), 1.26 (s, 3H, CH_3), 1.25 (s, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 180.0, 143.2, 138.7, 129.1, 127.6, 81.3, 45.1, 44.8, 25.9, 25.7, 21.7, 20.7. HRMS (ESI) *m/z* calcd for $C_{14}H_{20}NO_3S$ $[M+H]^+$ 282.1158, found 282.1147. These data are in agreement with literature data.[16]

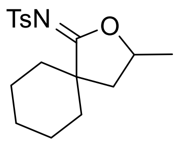
3-methyl-2-tosyl-2-azaspiro[4.5]decan-1-one (6b)[15]



6b

Yellow oil, 7 mg, 11% isolated yield. IR (neat, cm^{-1}) ν 1731 (C=O). ^1H NMR (360 MHz, $CDCl_3$) *δ* (ppm) 7.91 (d, *J* = 8.3 Hz, 2H, Ar-H), 7.30 (d, *J* = 8.3 Hz, 2H, Ar-H), 4.30 (dqd, *J* = 8.3, 6.1, 1,8 Hz, 1H, CH), 2.42 (s, 3H, CH_3), 2.23 (dd, *J* = 13.1, 8.3 Hz, 1H, CH_2), 1.69-1.60 (m, 5H, CH_2), 1,54 (d, J = 6.1 Hz, 3H, CH_3), 1. 41-1.36 (m, 2H, CH_2), 1.28-1.22 (m, 4H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) *δ* (ppm) 178.8, 155.0, 144.9, 136.2, 129.6, 128.4, 53.5, 45.7, 38.4, 33.8, 33.1, 25.3, 23.6, 21.9, 21.8, 21.8. HRMS (ESI) *m/z* calcd for $C_{17}H_{24}NO_3S$ $[M+H]^+$ 322.1471, found 322.1468. These data are in agreement with literature data.[15]

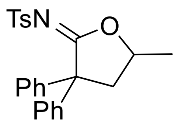
4-methyl-*N*-(3-methyl-2-oxaspiro[4.5]decan-1-ylidene)benzenesulfonamide (7b)[16]



7b

Slightly yellow solid, 34 mg, 52% isolated yield. m.p. 114-116 ℃. IR (neat, cm^{-1}) ν 1625 (C=N). ^1H NMR (360 MHz, $CDCl_3$) *δ* (ppm) 7.85 (d, *J* = 6.8 Hz, 2H, Ar-H), 7.26 (d, *J* = 6.8 Hz, 2H, Ar-H), 4.82-4.73 (m, 1H, CH), 2.41 (s, 3H, CH_3), 2.37 (dd, *J* = 13.2, 6.2 Hz, 1H, CH_2), 1.85-1.35 (m, 7H, CH_2), 1.40 (d, *J* = 6.2 Hz, 3H, CH_3), 1.33-1.14 (m, 4H, CH_2). ^{13}C NMR (91 MHz, $CDCl_3$) *δ* (ppm) 179.9, 143.1, 138.9, 129.1, 127.5, 81.7, 49.6, 40.2, 35.0, 32.7, 25.1, 22.4, 21.7, 21.0. HRMS (ESI) *m/z* calcd for $C_{17}H_{24}NO_3S$ $[M+H]^+$ 322.1471, found 322.1464. These data are in agreement with literature data.[16]

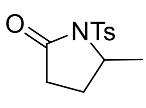
4-methyl-*N*-(5-methyl-3,3-diphenyldihydrofuran-2(3*H*)-ylidene)benzenesulfonamide (7c)[16]



7c

White solid, 74 mg, 79% isolated yield. m.p. 138-140 ℃. IR (neat, cm^{-1}) ν 1618 (C=N). ^1H NMR (360 MHz, $CDCl_3$) *δ* (ppm) 7.85 (d, *J* = 8.3 Hz, 2H, Ar-H), 7.39-7.20 (m, 12H, Ar-H), 4.77-4.67 (m, 1H, CH), 3.00 (dd, *J* = 13.00, 4.6 Hz, 1H, CH_2), 2.54-2.48 (dd, *J* = 13.00, 10.6 Hz, 1H, CH_2), 2.45 (s, 3H, CH_3), 1.52 (d, *J* = 6.1 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) *δ* (ppm) 175.5, 143.2, 141.7, 140.2, 138.8, 129.2, 129.0, 128.3, 128.0, 127.6, 127.5, 80.9, 61.4, 45.7, 21.7, 19.9. HRMS (ESI) *m/z* calcd for $C_{24}H_{24}NO_3S$ $[M+H]^+$ 406.1471, found 406.1452. These data are in agreement with literature data.[16]

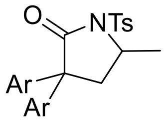
5-methyl-1-tosylpyrrolidin-2-one (6d)[15]



6d

Slightly yellow solid, 21 mg, 38% isolated yield. m.p. 95-97 ℃, IR (neat, cm^{-1}) ν 1733 (C=O). ^1H NMR (360 MHz, $CDCl_3$) *δ* (ppm) 7.93 (d, *J* = 8.4 Hz, 2H, Ar-H), 7.31 (d, *J* = 8.4 Hz, 2H, Ar-H), 4.55-4.47 (m, 1H, CH), 2.60-2.50 (m, 1H, CH_2), 2.42 (s, 3H, CH_3), 2.38-2.22 (m, 2H, CH_2), 1.74-1.68 (m, 1H, CH_2), 1.45 (d, *J* = 6.4 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) *δ* (ppm) 173.4, 145.0, 136.2, 129.6, 128.4, 56.5, 30.7, 26.7, 21.8, 21.6. HRMS (ESI) *m/z* calcd for $C_{12}H_{16}NO_3S$ $[M+H]^+$ 254.0845, found 254.0844. These data are in agreement with literature data.[15]

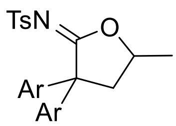
3,3-bis(4-(*tert*-butyl)phenyl)-5-methyl-1-tosylpyrrolidin-2-one (6e)



6e Ar = 4-t-BuC_6H_4

White solid, 7 mg, 7% isolated yield. m.p. 151-155 °C. IR (neat, cm^{-1}) ν 1755 (C=O). ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.84 (d, *J* = 8.3Hz, 2H, Ar-H), 7.24 (d, *J* = 8.3Hz, 2H, Ar-H), 7.19 (d, *J* = 8.3Hz, 2H, Ar-H), 7,07 (d, *J* = 8,3 Hz, 2H, Ar-H), 6.99 (d, *J* = 8,3 Hz, 2H, Ar-H), 4.26-4.17 (m, 1H, CH), 3.01 (dd, J = 13.2, 6.5 Hz, 1H, CH_2), 2.43 (s, 3H, CH_3), 2.38 (dd, J = 13,2, 8.0 Hz, 1H, CH_2), 1.53 (d, *J* = 6.1 Hz, 3H, CH_3), 1.27 (s, 9H, CH_3), 1.25 (s, 9H, CH_3). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 175.2, 150.2, 150.0, 144.8, 139.1, 137.1, 135.7, 129.4, 128.6, 127.5, 127.1, 125.6, 125.4, 57.3, 53.8, 42.7, 34.5, 31.4, 31.1, 22.2, 21.8. HRMS (ESI) *m/z* calcd for $C_{32}H_{40}NO_3S$ $[M+H]^+$ 518.2723, found 518.2701.

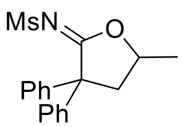
***N*-(3,3-bis(4-(*tert*-butyl)phenyl)-5-methyldihydrofuran-2(3*H*)-ylidene)-4-methylbenzenesulfonamide (7e)**



7e Ar = 4-t-BuC_6H_4

White solid, 77 mg, 75% isolated yield. m.p. 94-96 °C. IR (neat, cm^{-1}) ν 1629 (C=N). ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.83 (d, *J* = 8.1Hz, 2H, Ar-H), 7.32 (d, *J* = 8.2Hz, 2H, Ar-H), 7.26-7.23 (m, 6H, Ar-H), 7.08 (d, *J* = 8.2Hz, 2H, Ar-H), 4.73-4.63 (m, 1H, CH), 2.97 (dd, *J* = 12.8, 4.6 Hz, 1H, CH_2), 2.46-2.41 (m, 1H, CH_2), 2.43 (s, 3H, CH_3), 1.44 (d, *J* = 6.1 Hz, 3H, CH_3), 1.29 (s, 9H, CH_3), 1.28 (s, 9H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 176.1, 150.8, 150.1, 143.1, 139.0, 138.9, 136.5, 129.1, 127.9, 127.6, 127.3, 125.9, 125.2, 80.9, 60.8, 46.0, 34.6, 34.5, 31.4, 21.7, 20.0. HRMS (ESI) *m/z* calcd for $C_{32}H_{40}NO_3S$ $[M+H]^+$ 518.2723, found 518.2700.

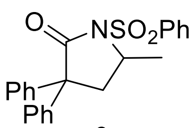
***N*-(5-methyl-3,3-diphenyldihydrofuran-2(3*H*)-ylidene)methanesulfonamide (7f)**



7f

White solid, 57 mg, 87% isolated yield. m.p. 132-134 °C, IR (neat, cm^{-1}) ν 1643 (C=N). ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.26-7.14 (m, 10H, Ar-H), 4.68-4.59 (m, 1H, CH), 2.93 (s, 3H, CH_3), 2.89 (dd, *J* = 13.0, 4.5 Hz, 1H, CH_2), 2.56 (dd, *J* = 13.0, 10.7 Hz, 1H, CH_2), 1.49 (d, *J* = 6.2 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 175.5, 141.5, 140.3, 129.1, 129.0, 128.9, 128.3, 128.09, 127.5, 81.0, 61.3, 45.7, 42.6, 20.0. HRMS (ESI) *m/z* calcd for $C_{18}H_{20}NO_3S$ $[M+H]^+$ 330.1158, found 330.1150.

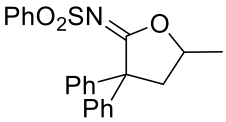
5-methyl-3,3-diphenyl-1-(phenylsulfonyl)pyrrolidin-2-one (6g)



6g

Yellow oil, 9 mg, 12% isolated yield. IR (neat, cm^{-1}) ν 1733 (C=O). ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.96 (d, J = 7.7 Hz, 2H, Ar-H), 7.61 (t, *J* = 7.5 Hz 1H, Ar-H), 7.45 (t, *J* = 8.0 Hz, 2H, Ar-H), 7.24-7.13 (m, 8H, Ar-H), 7.07-7.04 (m, 2H, Ar-H), 4.32-4.23 (m, 1H, CH), 3.06 (dd, *J* = 13.2, 6.5 Hz, 1H, CH_2), 2.44 (dd, *J* = 13.2, 7.4 Hz, 1H, CH_2), 1.53 (d, *J* = 6.2 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 74.7, 142.1, 140.0, 138.3, 133.9, 128.9, 128.8, 128.6, 128.5, 127.8, 127.5, 127.4, 127.3, 58.0, 53.9, 42.4, 22.1. HRMS (ESI) *m/z* calcd for $C_{23}H_{22}NO_3S$ $[M+H]^+$ 392.1315, found 392.1296.

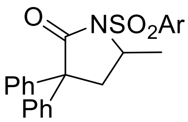
_N_-(5-methyl-3,3-diphenyldihydrofuran-2(3_H_)-ylidene)benzenesulfonamide (7g)



7g

White solid, 56 mg, 72% isolated yield. m.p. 121-123 ℃, IR (neat, cm^{-1}) ν 1630 (C=N). ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.87-7.84 (m, 2H, Ar-H), 7.48-7.44 (m, 1H, Ar-H), 7.40-7.35 (m, 2H, Ar-H), 7.24-7.09 (m, 10H, Ar-H), 4.65-4.56 (m, 1H, CH), 2.93 (dd, *J* = 13.0, 4.6 Hz, 1H, CH_2), 2.50 (dd, *J* = 13.0, 10.7 Hz, 1H, CH_2), 1.39 (d, *J* = 6.2 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 175.8, 141.6, 141.5, 140.0, 132.5, 129.0, 128.5, 128.2, 128.0, 127.5, 127.3, 127.2, 81.0, 61.4, 45.6, 19.9. HRMS (ESI) *m/z* calcd for $C_{23}H_{22}NO_3S$ $[M+H]^+$ 392.1315, found 392.1298.

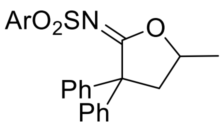
1-((4-methoxyphenyl)sulfonyl)-5-methyl-3,3-diphenylpyrrolidin-2-one (6h)



6h Ar = 4-MeOC$_6$H$_4$

White solid, 8 mg, 9% isolated yield. m.p. 176-178 ℃, IR (neat, cm^{-1}) ν 1731 (C=O). ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.88 (d, *J* = 8.9 Hz, 2H, Ar-H), 7.25-7.14 (m, 8H, Ar-H), 7.08-7.05 (m, 2H, Ar-H), 6.89 (d, *J* = 8.9 Hz, 2H, Ar-H), 4.30-4.19 (m, 1H, CH), 3.87 (s, 3H, CH_3), 3.05 (dd, *J* = 13.2, 6.6 Hz, 1H, CH_2), 2.42 (dd, *J* = 13.2, 7.3 Hz, 1H, CH_2), 1.52 (d, *J* = 6.1 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 174.3, 163.9, 142.3, 140.1, 130.8, 128.8, 128.6, 127.9, 127.6, 127.5, 127.3, 114.0, 58.0, 55.8, 53.8, 42.4, 22.2. HRMS (ESI) *m/z* calcd for $C_{24}H_{24}NO_3S$ $[M+H]^+$ 422.1421, found 422.1406.

4-methoxy-_N_-(5-methyl-3,3-diphenyldihydrofuran-2(3_H_)-ylidene)benzenesulfonamide (7h)



7h Ar = 4-MeOC$_6$H$_4$

White solid, 74 mg, 88% isolated yield. m.p. 138-140 ℃, IR (neat, cm^{-1}) ν 1630 (C=N). ^1H NMR (360 MHz, $CDCl_3$) δ (ppm) 7.83 (d, *J* = 8.8 Hz, 2H, Ar-H), 7.30-7.12 (m, 10H, Ar-H), 6.88 (d, *J* = 8.8 Hz, 2H, Ar-H), 4.67-4.58 (m, 1H, CH), 3.80 (s, 3H, CH_3), 2.88 (dd, *J* = 13.0, 4.6 Hz, 1H, CH_2), 2.45 (dd, *J* = 13.0, 10.7 Hz, 1H, CH_2), 1.42 (d, *J* = 6.2 Hz, 3H, CH_3). ^{13}C NMR (91 MHz, $CDCl_3$) δ (ppm) 175.3, 162.9, 141.6, 140.0, 133.3, 129.6, 128.9, 128.2, 127.9, 127.5, 127.4, 114.2, 113.6, 80.8, 61.3, 55.7, 45.6, 19.9. HRMS (ESI) *m/z* calcd for $C_{24}H_{24}NO_3S$ $[M+H]^+$ 422.1421, found 422.1404.

4-Crystallographic Data

X-ray diffraction data for compounds **4m**, **6a**, **7a** & **7c** were collected by using a Kappa X8 APPEX II Bruker diffractometer with graphite-monochromated MoK_{α} radiation (λ = 0.71073 Å). Crystals were mounted on a CryoLoop (Hampton Research) with Paratone-N (Hampton Research) as cryoprotectant and then flashfrozen in a nitrogen-gas stream at 200 K. For compounds, the temperature of the crystal was maintained at the selected value by means of a 700 series Cryostream cooling device to within an accuracy of ±1K. The data were corrected for Lorentz polarization, and absorption effects. The structures were solved by direct methods using SHELXS-97[17] and refined against F^2 by full-matrix least-squares techniques using SHELXL-2018[18] with anisotropic displacement parameters for all non-hydrogen atoms. Hydrogen atoms were located on a difference Fourier map and introduced into the calculations as a riding model with isotropic thermal parameters. All calculations were performed by using the Crystal Structure crystallographic software package WINGX[19].

The crystal data collection and refinement parameters are given in Table S2.

CCDC 2213993-2213996 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe via http://www.ccdc.cam.ac.uk/structures/.

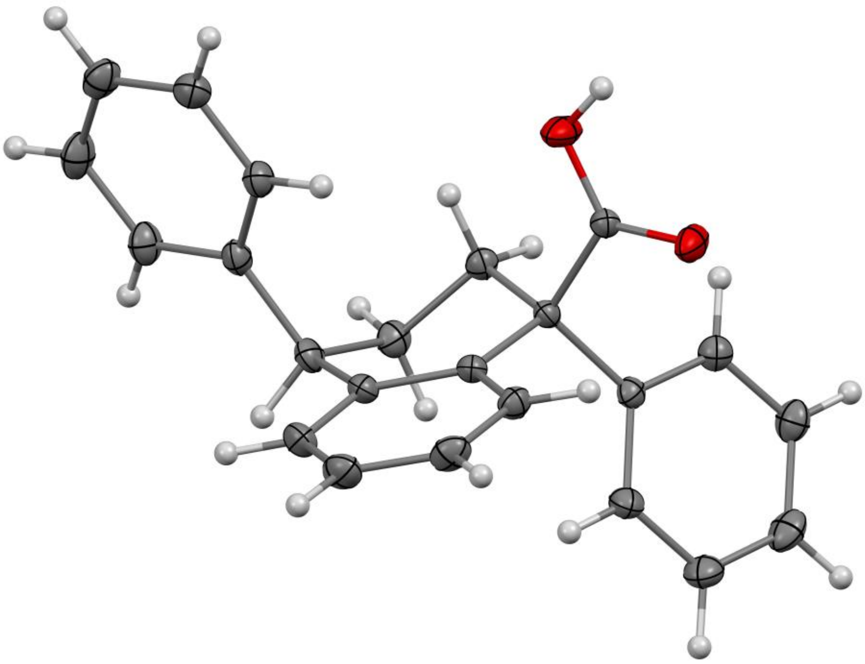


Figure S1. An ORTEP drawing of compound **4m.** Thermal ellipsoids are shown at the 30% level.

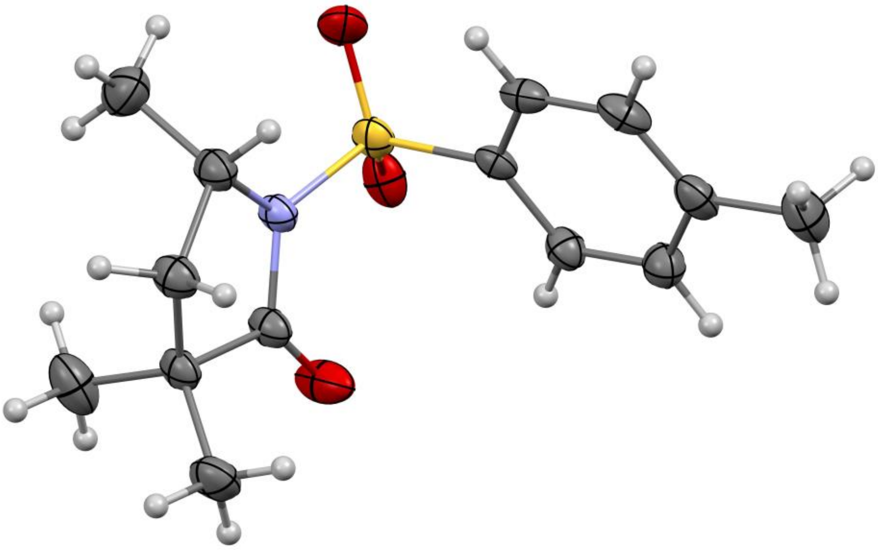


Figure S2. An ORTEP drawing of compound **6a**. Thermal ellipsoids are shown at the 30% level.

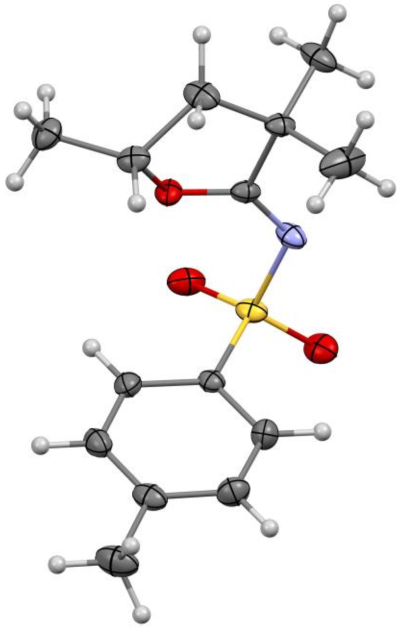


Figure S3. An ORTEP drawing of compound **7a**. Thermal ellipsoids are shown at the 30% level. Only one disorder component is shown.

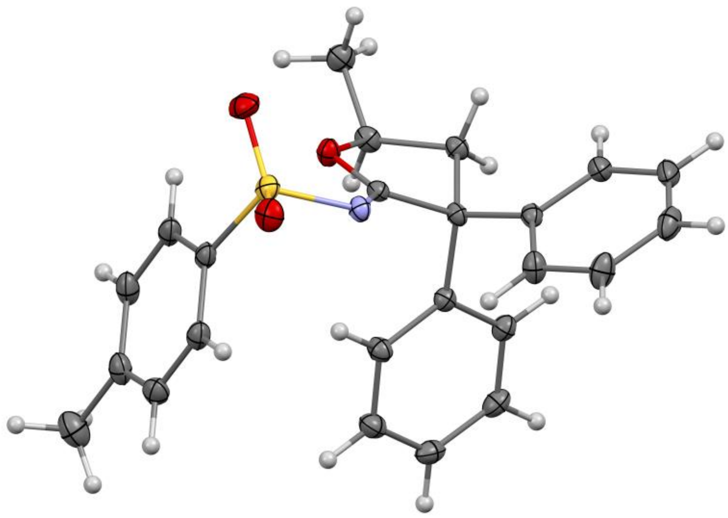
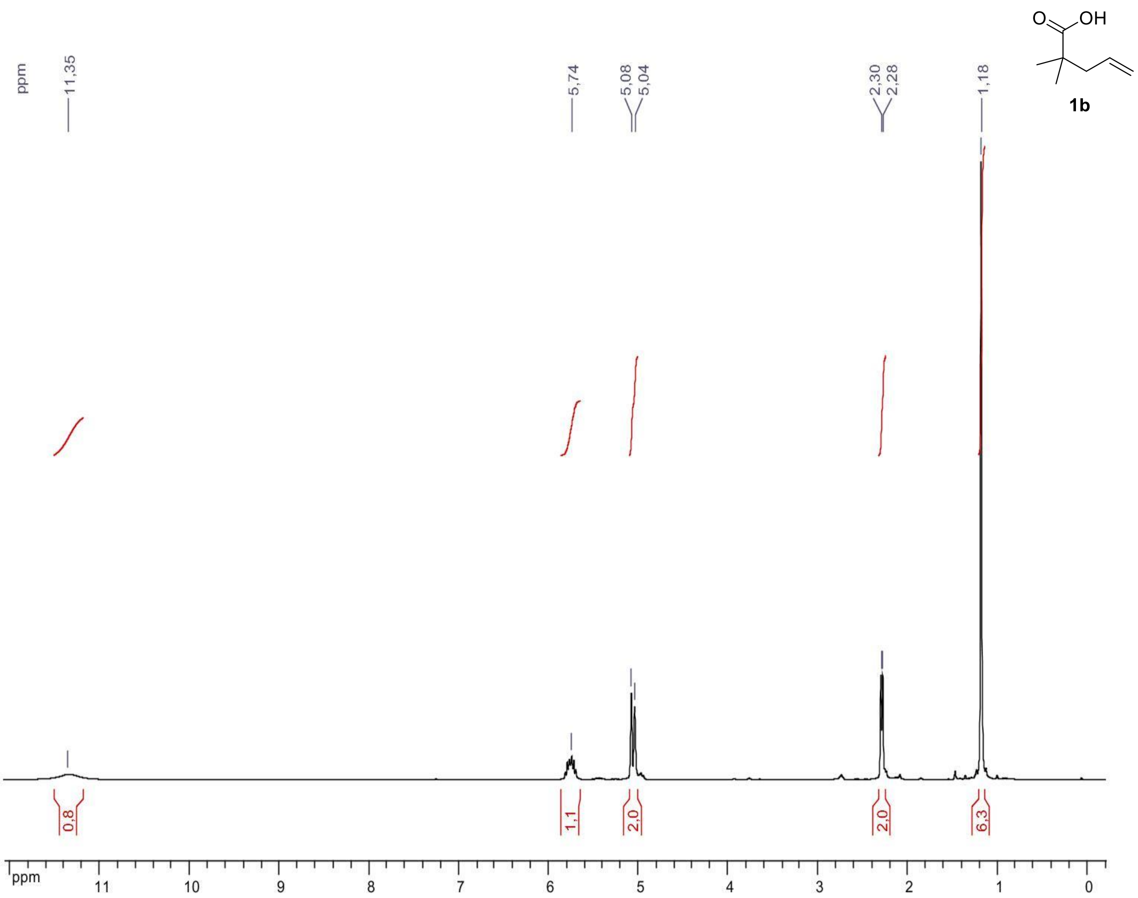


Figure S4. An ORTEP drawing of compound **7c**. Thermal ellipsoids are shown at the 30% level.

Compound	**4m**	**6a**	**7a**	**7c**
CCDC	2213993	2213994	2213996	2213995
Empirical Formula	C_{23} H_{20} O_2	C_{14} H_{19} N O_3 S	C_{14} H_{19} N O_3 S	C_{24} H_{23} N O_3 S
M_r	328.39	281.36	281.36	405.49
Crystal size, mm^3	0.14 x 0.07 x 0.02	0.28 x 0.09 x 0.03	0.28 x 0.18 x 0.13	0.33 x 0.12 x 0.03
Crystal system	triclinic	monoclinic	triclinic	orthorhombic
Space group	*P* -1	$P\ 2_1/c$	*P* -1	*P b c a*
a, Å	8.611(2)	8.0427(9)	8.0664(4)	10.3347(3)
b, Å	9.741(2)	15.333(2)	8.1885(4)	19.6916(6)
c, Å	11.193(3)	11.9935(15)	12.3567(7)	20.1928(7)
α, °	95.479(10)	90	89.870(3)	90
β, °	96.784(10)	99.561(7)	88.621(3)	90
γ, °	105.599(10)	90	64.810(2)	90
Cell volume, Å^3	889.9(4)	1458.4(3)	738.32(7)	4109.4(2)
Z ; Z’	2 ; 1	4 ; 1	2 ; 1	8 ; 1
T, K	200(1)	200(1)	200(1)	200(1)
Radiation type ; wavelength Å	MoKα ; 0.71073	MoKα ; 0.71073	MoKα ; 0.71073	MoKα ; 0.71073
F_{000}	348	600	300	1712
μ, mm^{-1}	0.077	0.225	0.223	0.183
range, °	1.849 - 30.495	2.175 - 30.498	1.649 - 30.723	1.621 - 30.712
Reflection collected	24 263	24 815	27 372	104 894
Reflections unique	5 197	4 348	4 487	6 265
R_{int}	0.0585	0.0581	0.0376	0.0837
GOF	1.036	1.075	1.023	1.013
Refl. obs. (I>2(I))	3 085	2 326	3 599	3 978
Parameters ; restraints	227 ; 0	176 ; 0	205 ; 0	264 ; 0
wR_2 (all data)	0.1346	0.2557	0.1463	0.1163
R value (I>2(I))	0.0540	0.0823	0.0525	0.0452
Largest diff. peak and hole (e-.Å^{-3})	0.232 ; -0.260	0.872 ; -0.471	0.439 ; -0.433	0.261 ; -0.439

Table S2. Crystallographic data and structure refinement details.

5-Spectra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2,0
6,3
ppm
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9
8
7
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2
1
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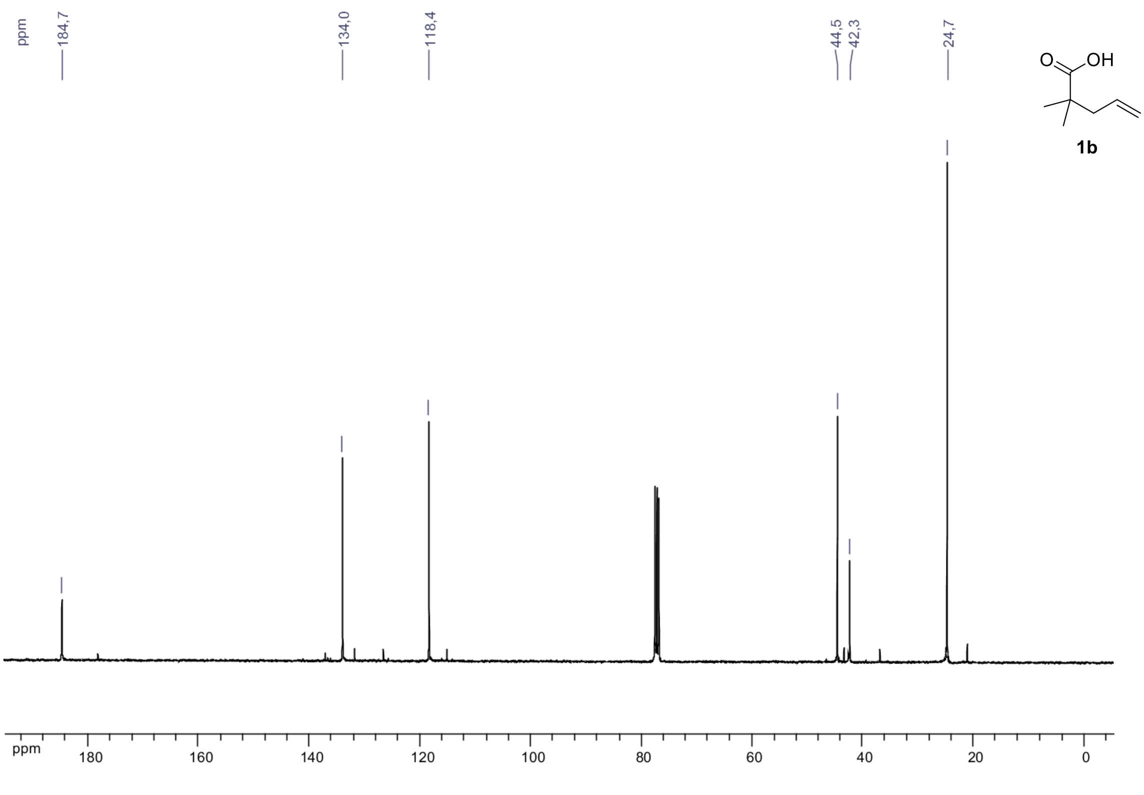
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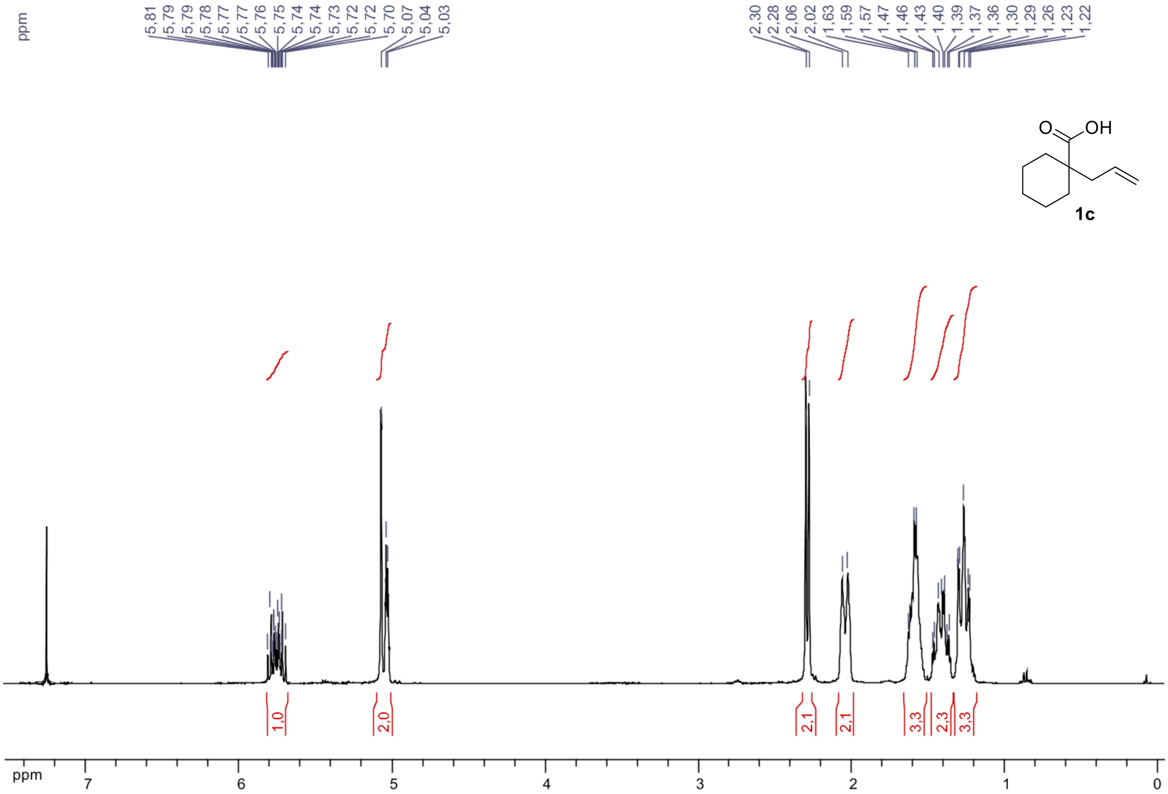

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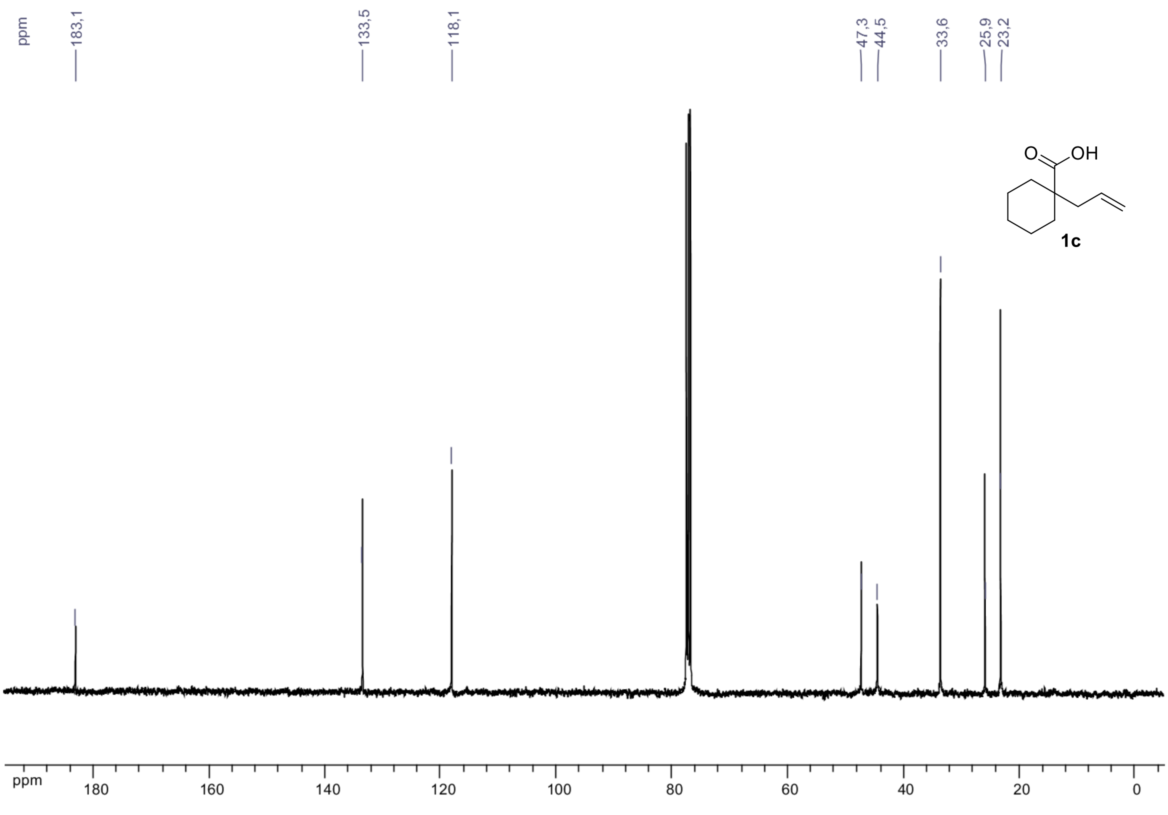
ppm 7 6 5 4 3 2 1 0



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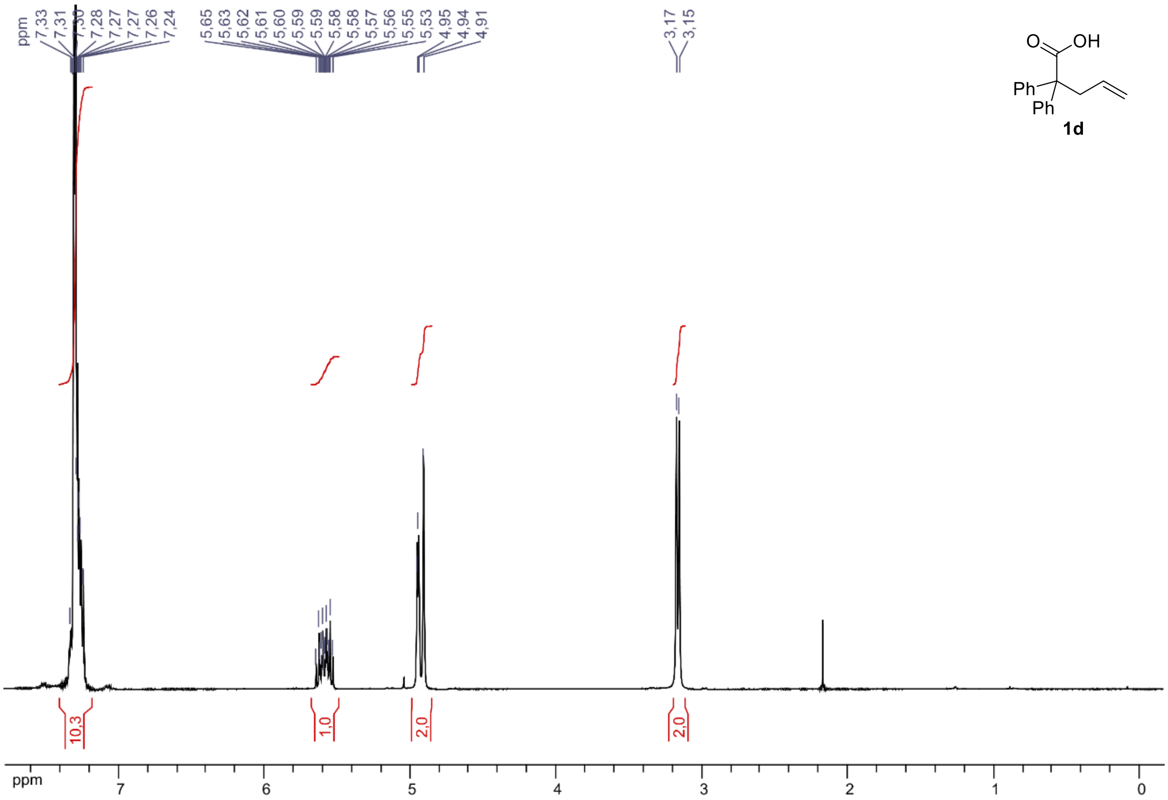

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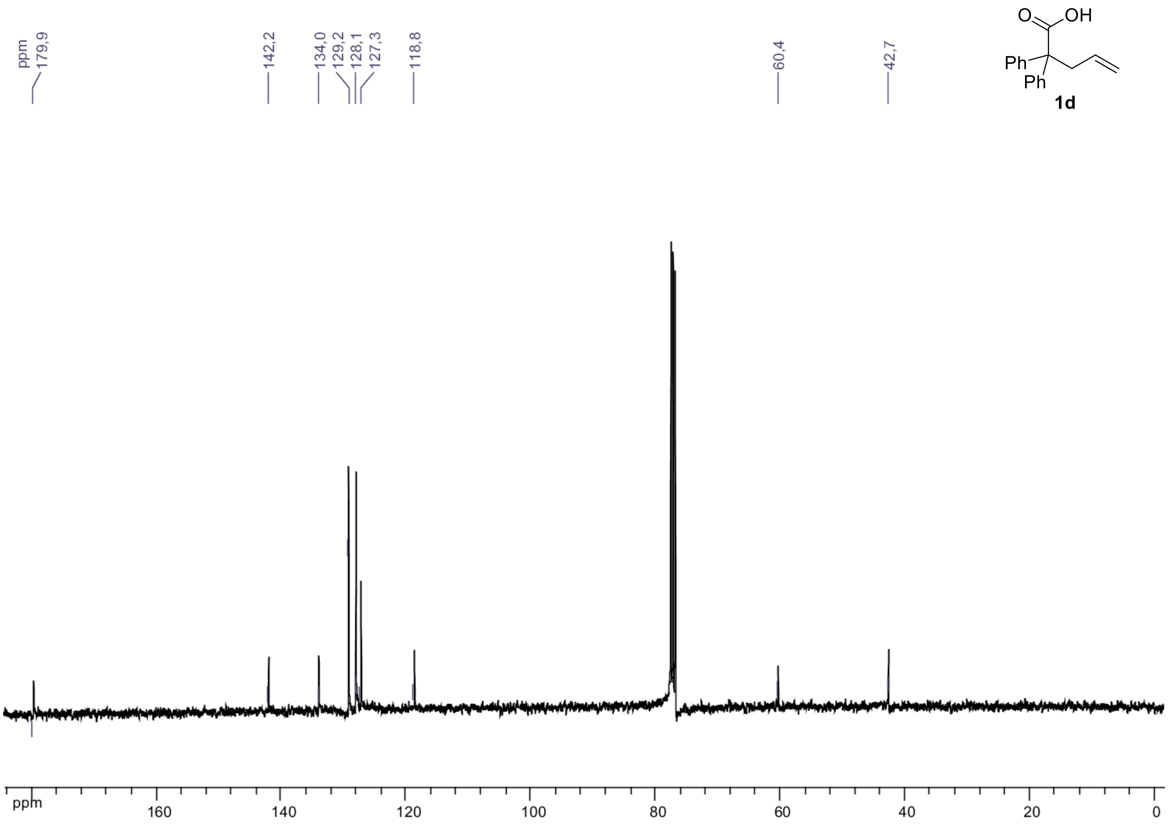

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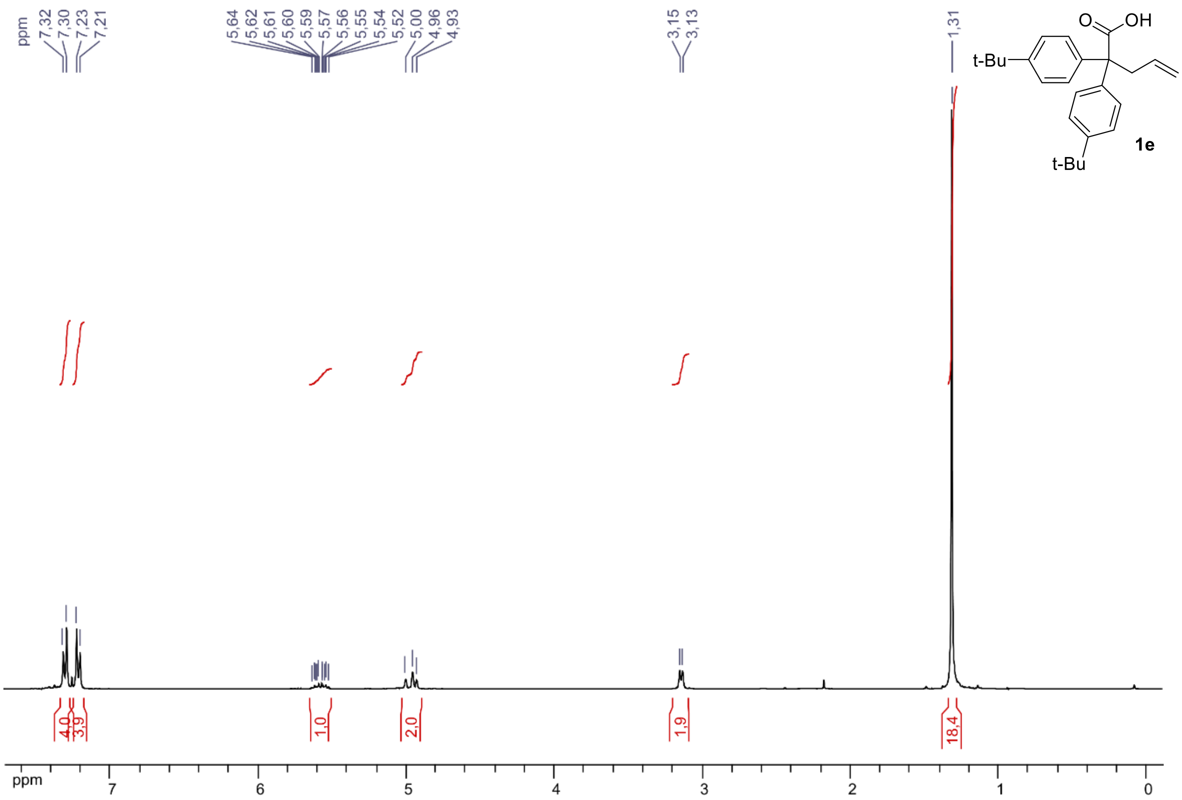
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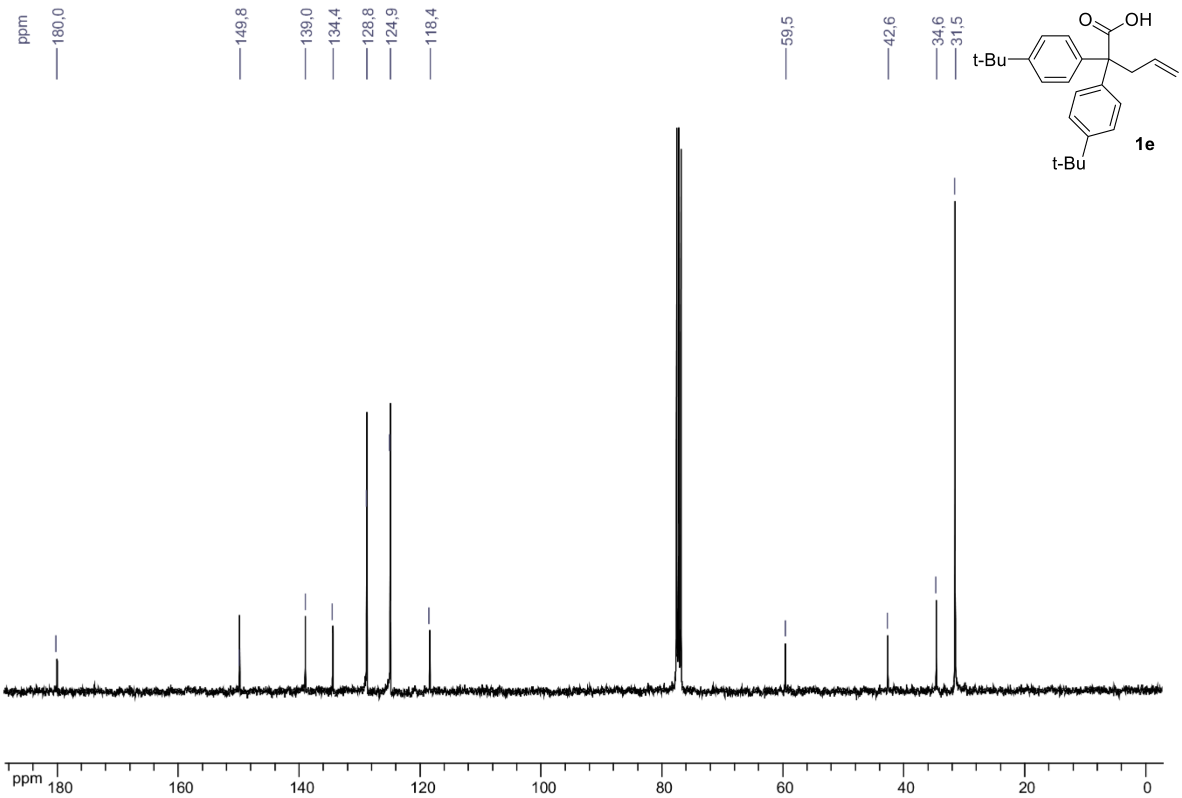
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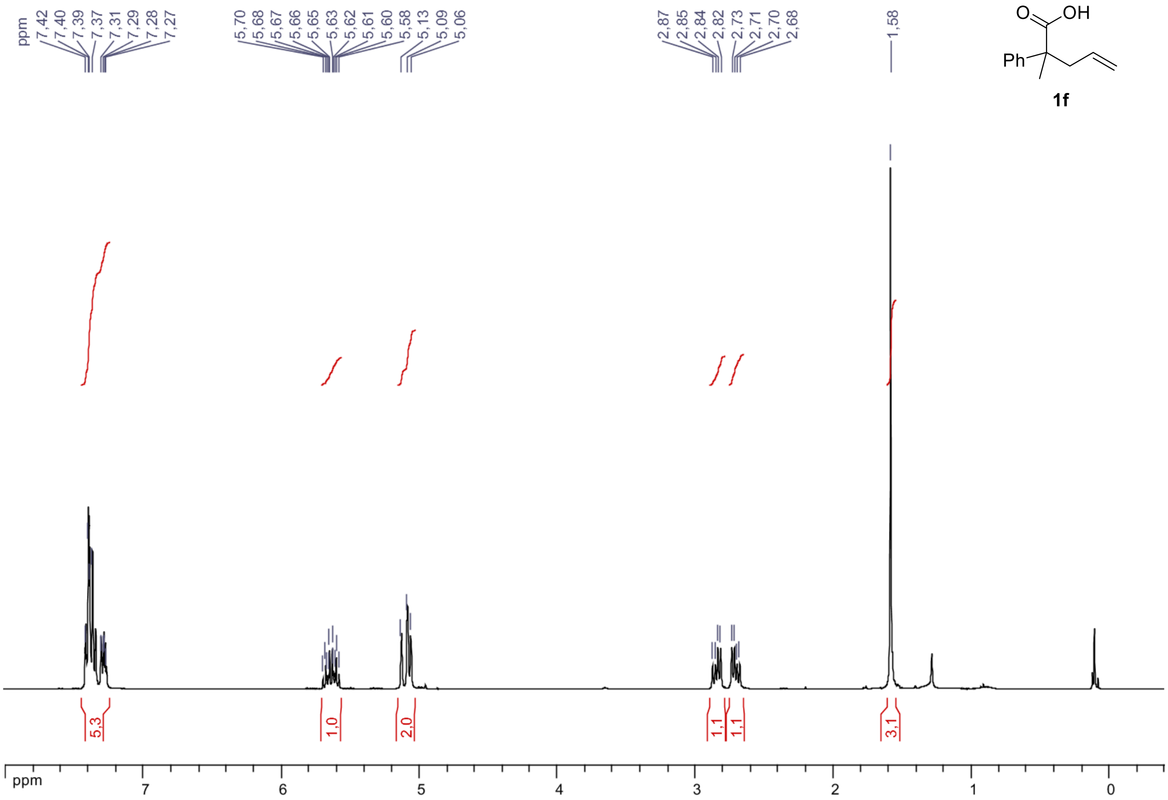
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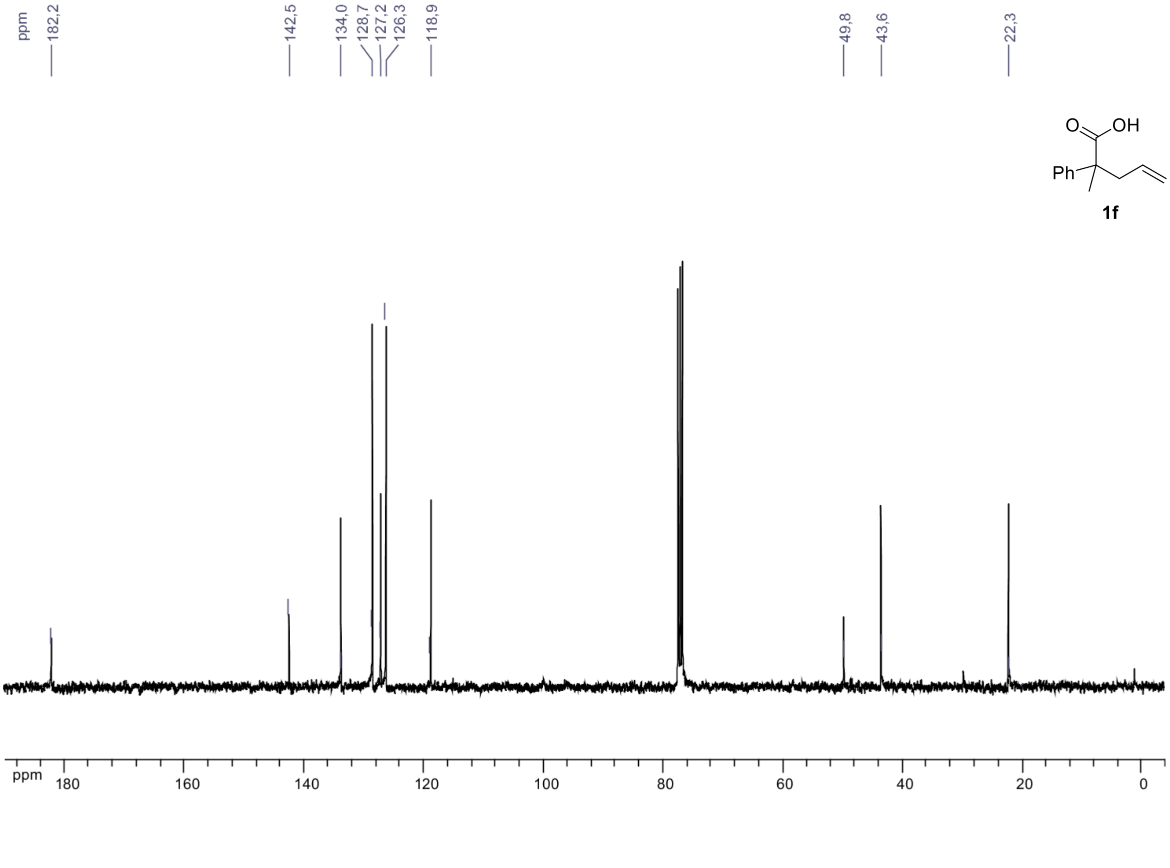
Ph

1f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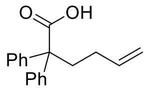

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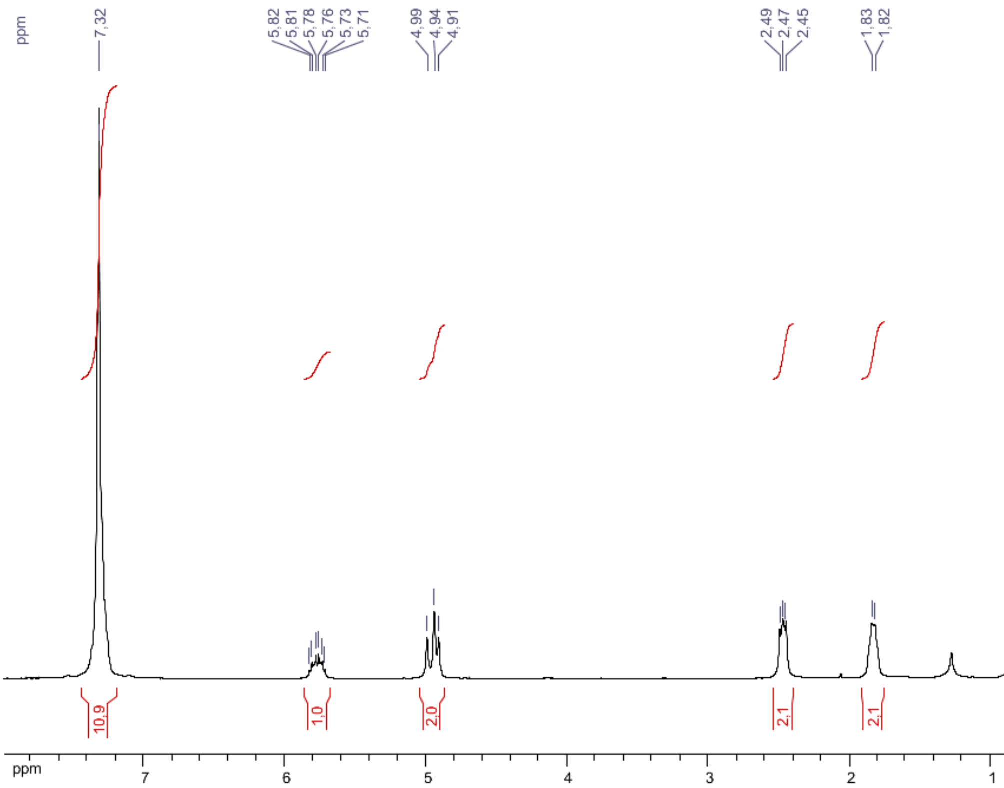

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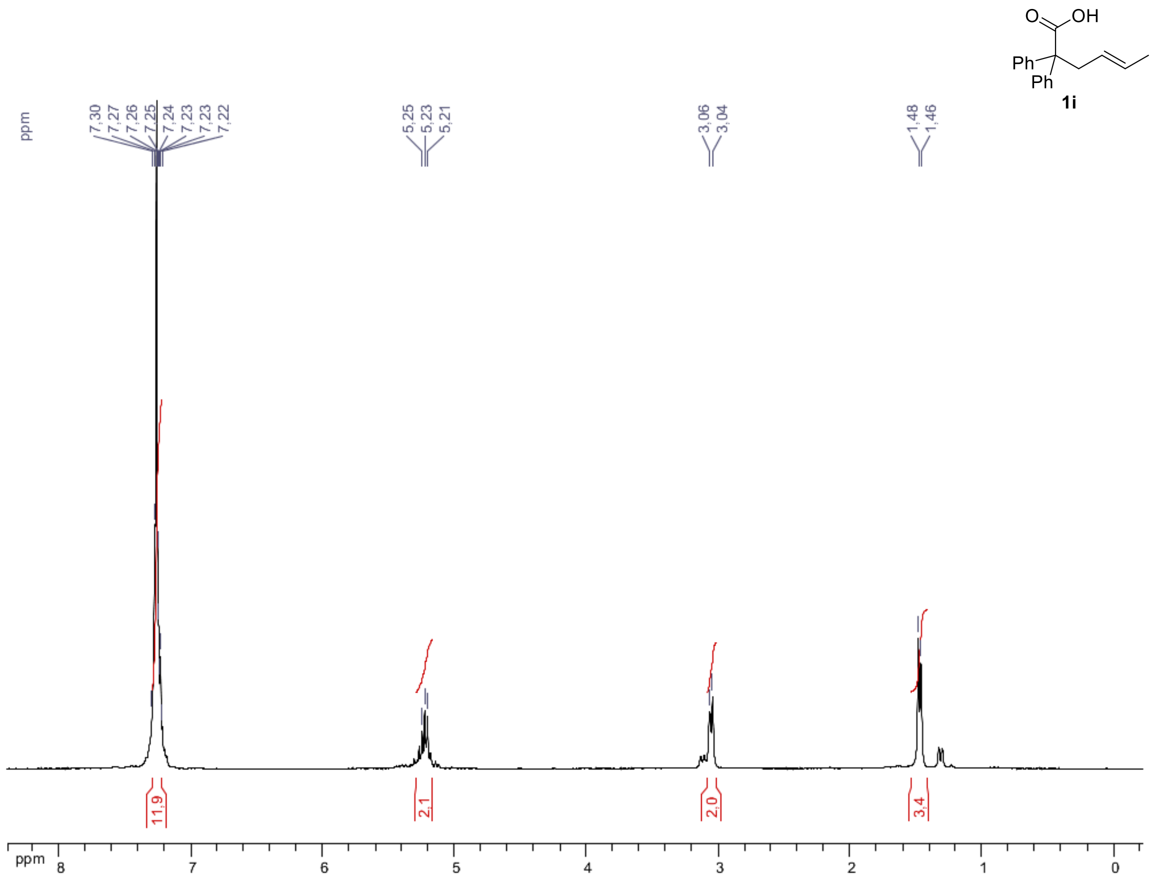
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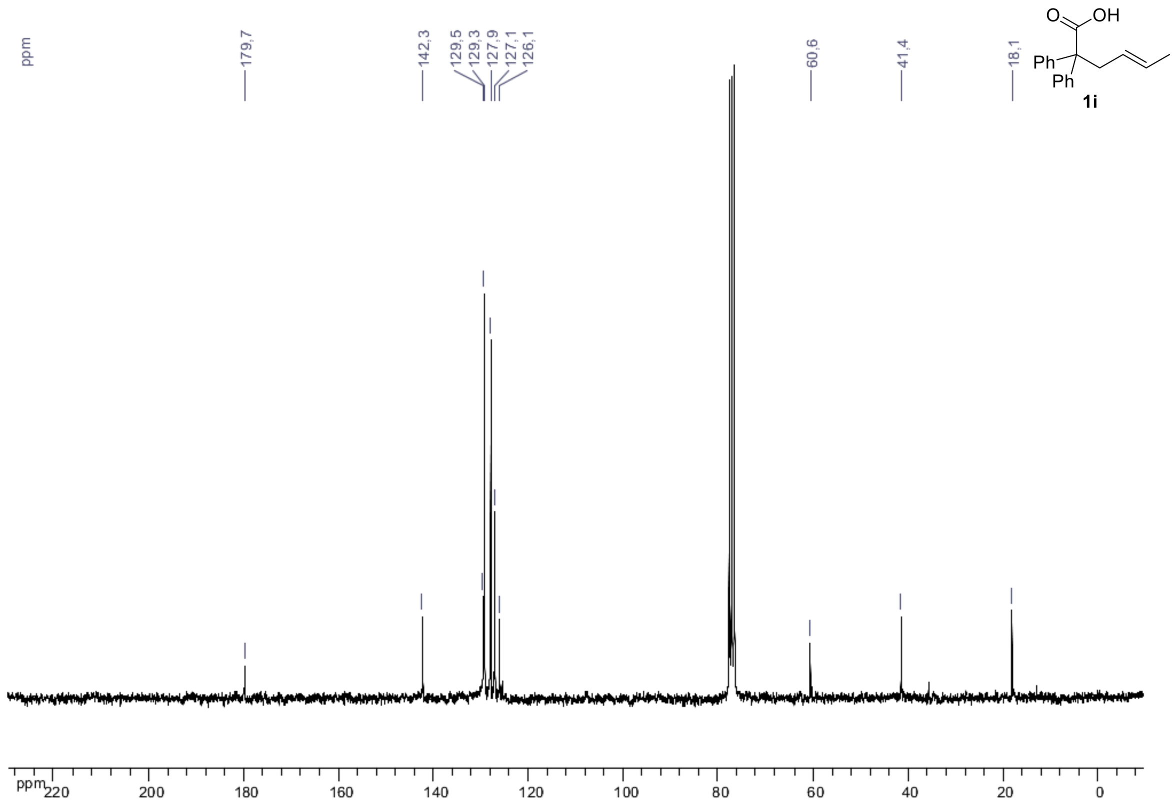
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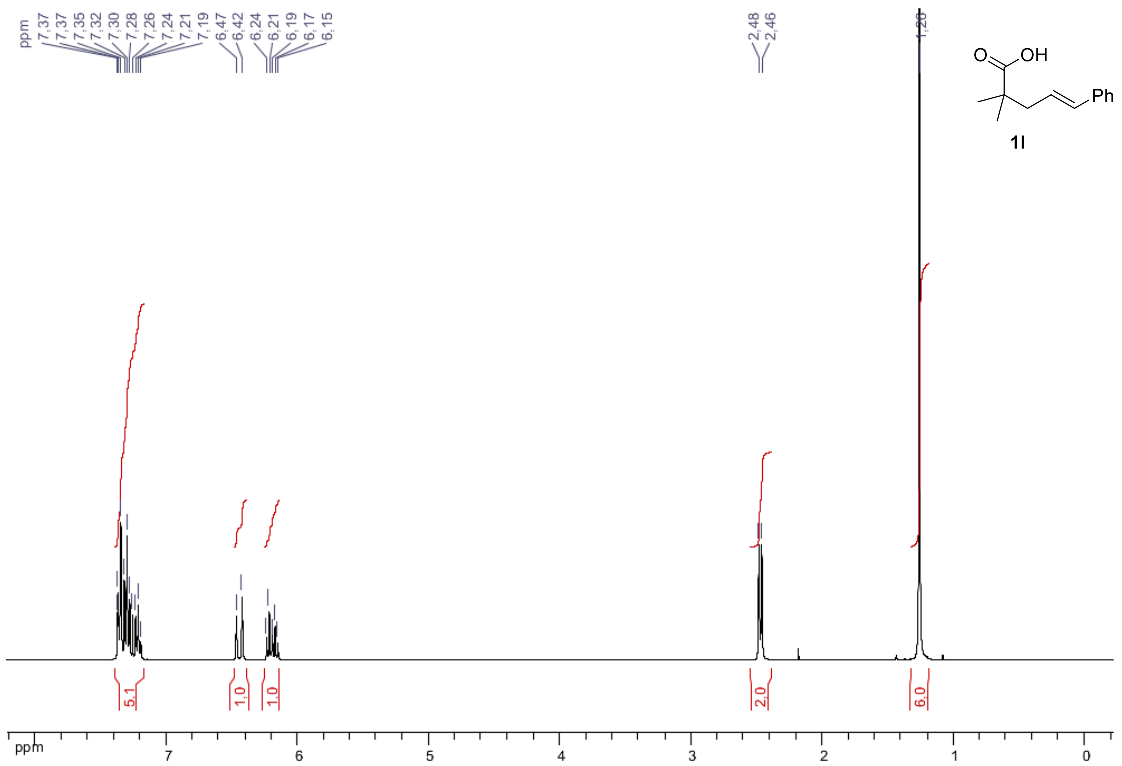
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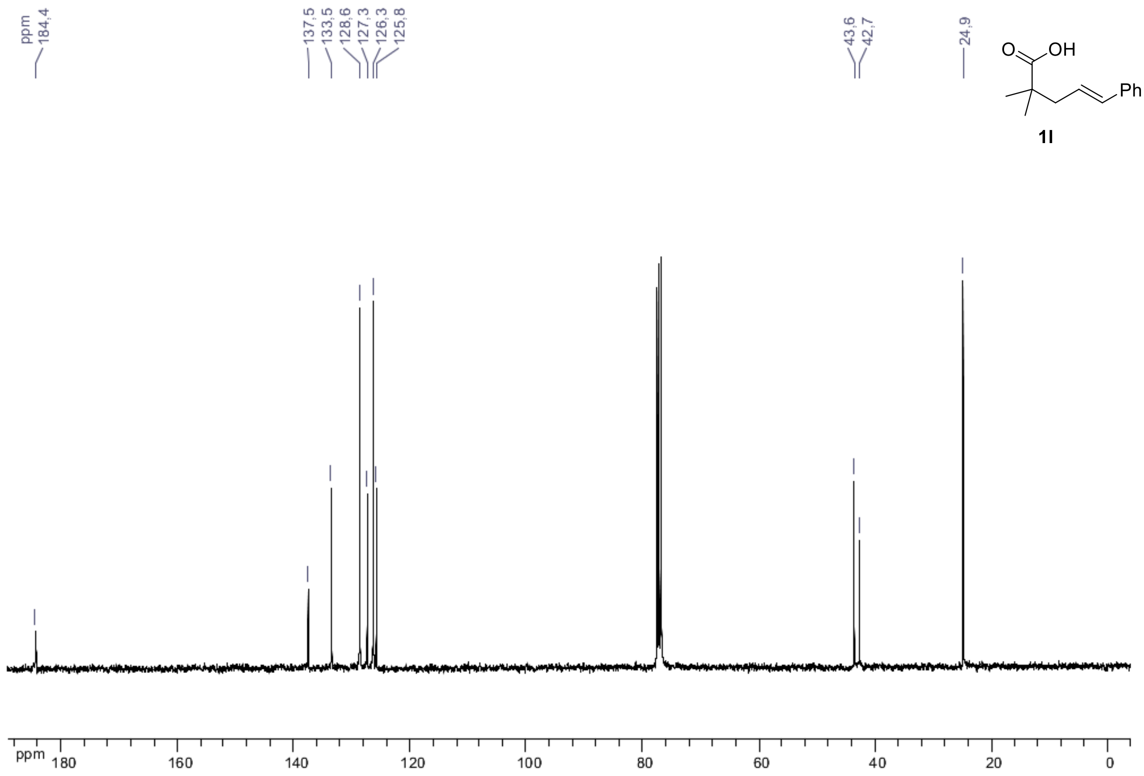
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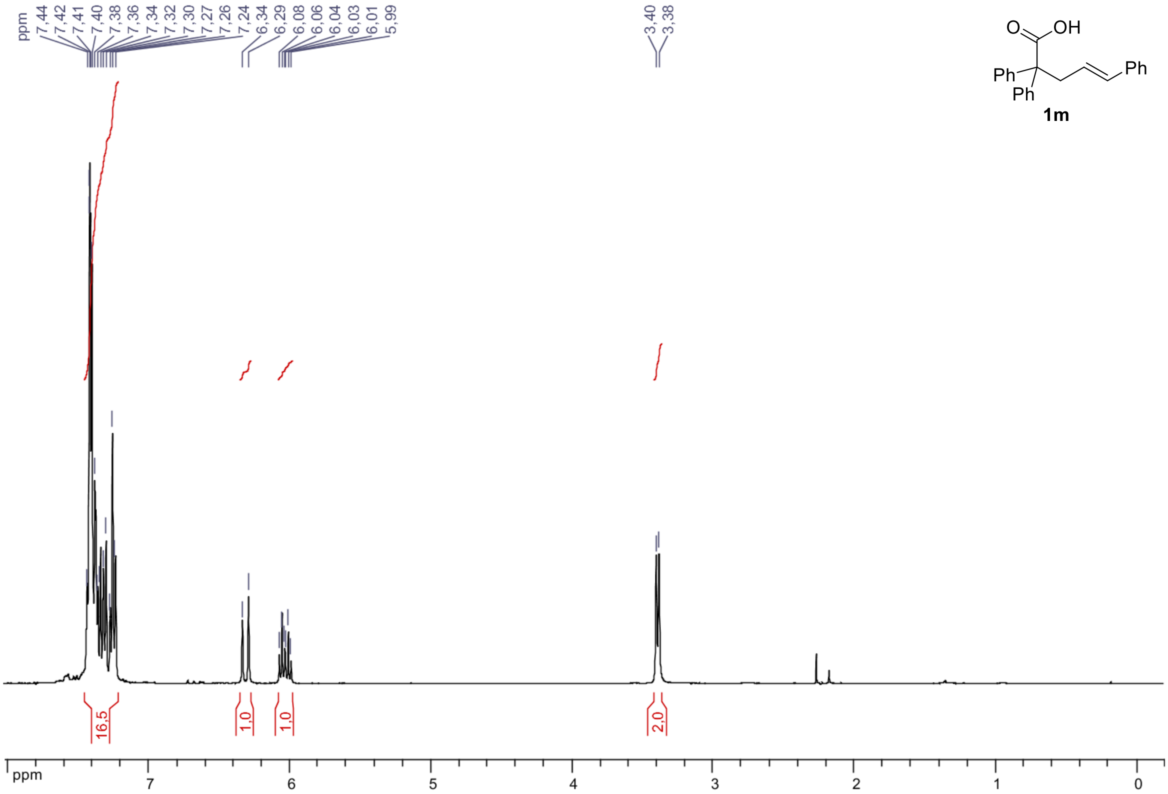
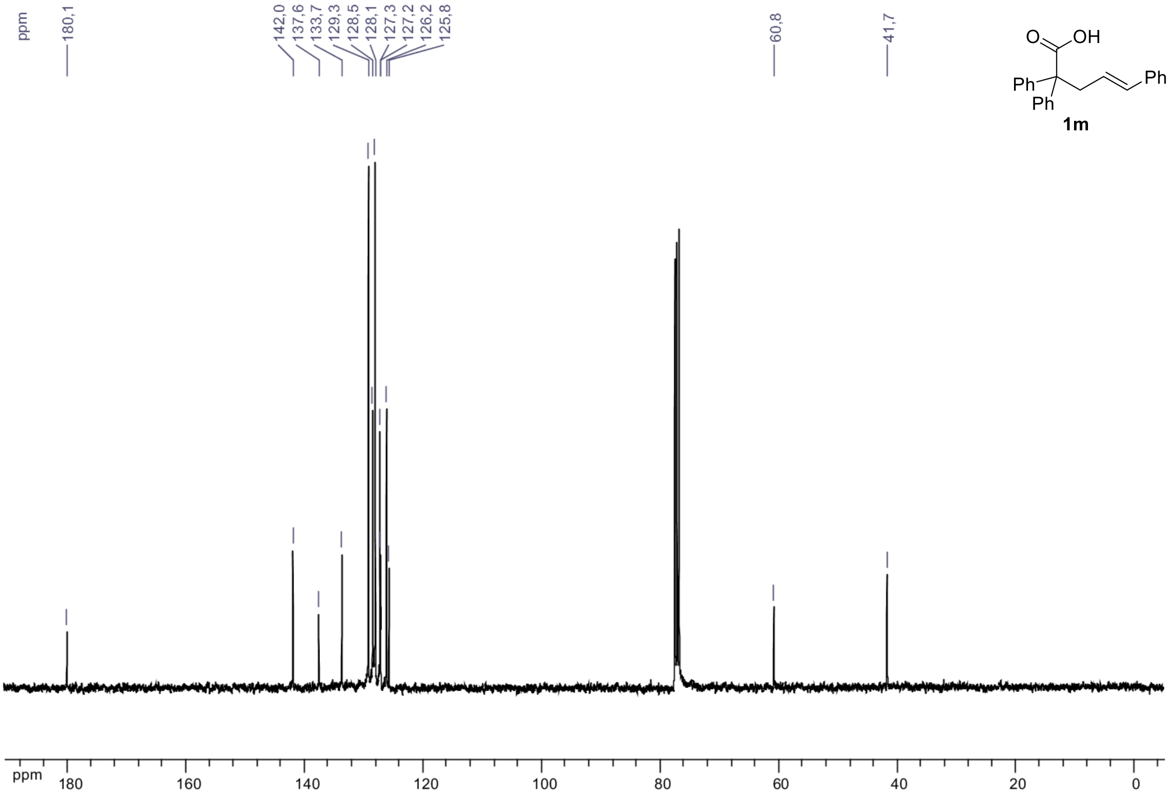
128,6
127,3
126,3
125,8
43,6
42,7
24,9
ppm
180
160
140
120
100
80
60
40
20
0
O
OH
Ph

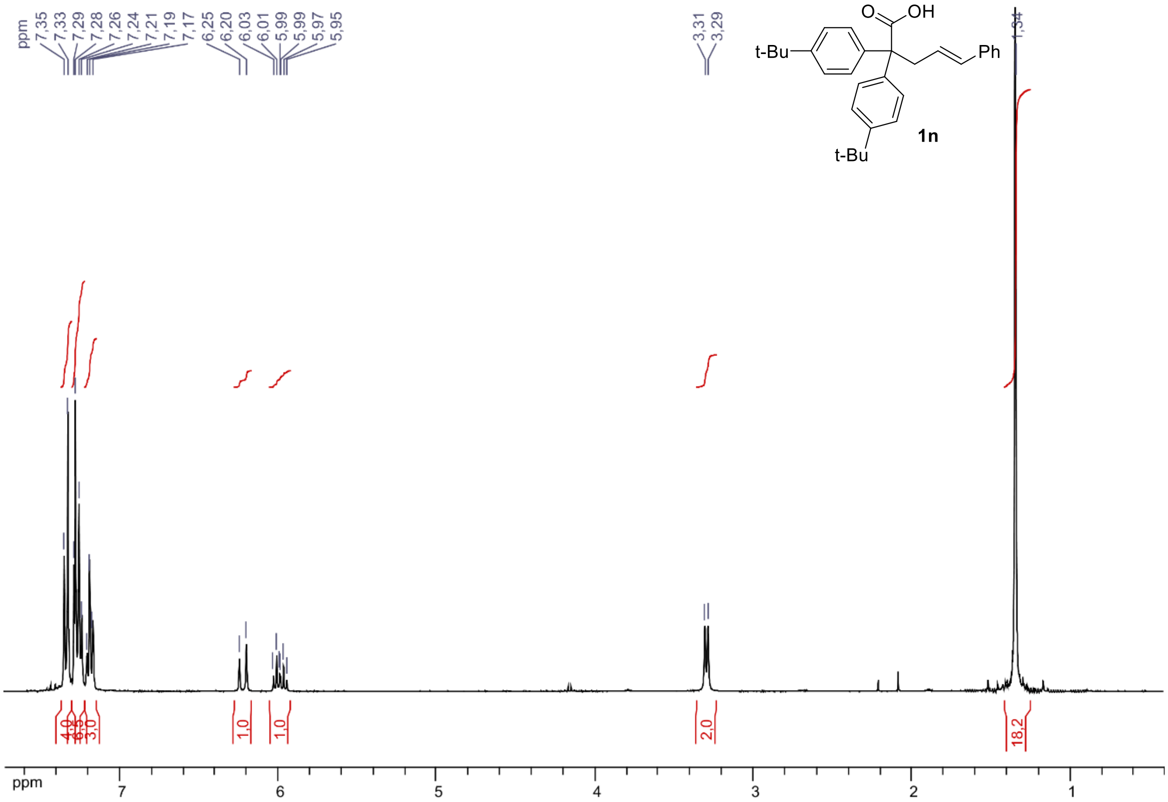
1l

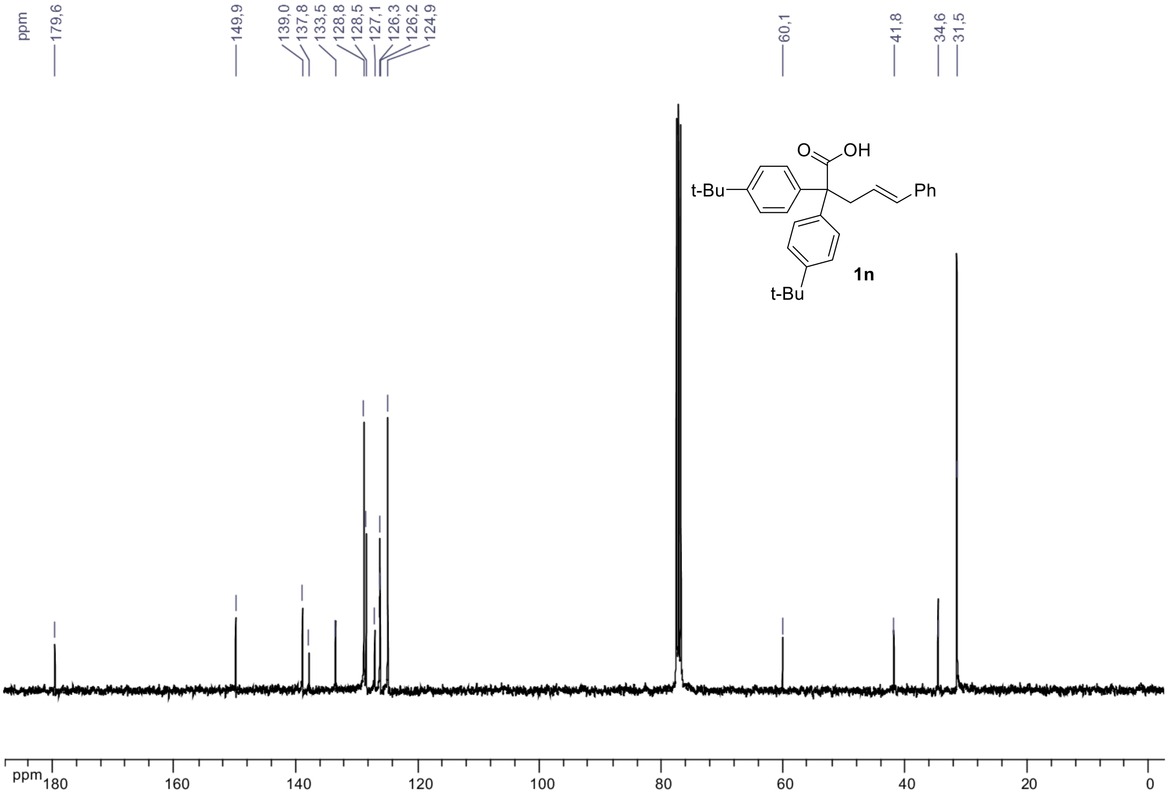

ppm
7,44 7,42 7,41 7,40 7,38 7,36 7,34 7,32 7,30 7,27 7,26 7,24 6,34 6,29 6,08 6,06 6,04 6,03 6,01 5,99
3,40 3,38
16,5
1,0
1,0
2,0
ppm 7 6 5 4 3 2 1 0
O
OH
Ph
Ph
Ph

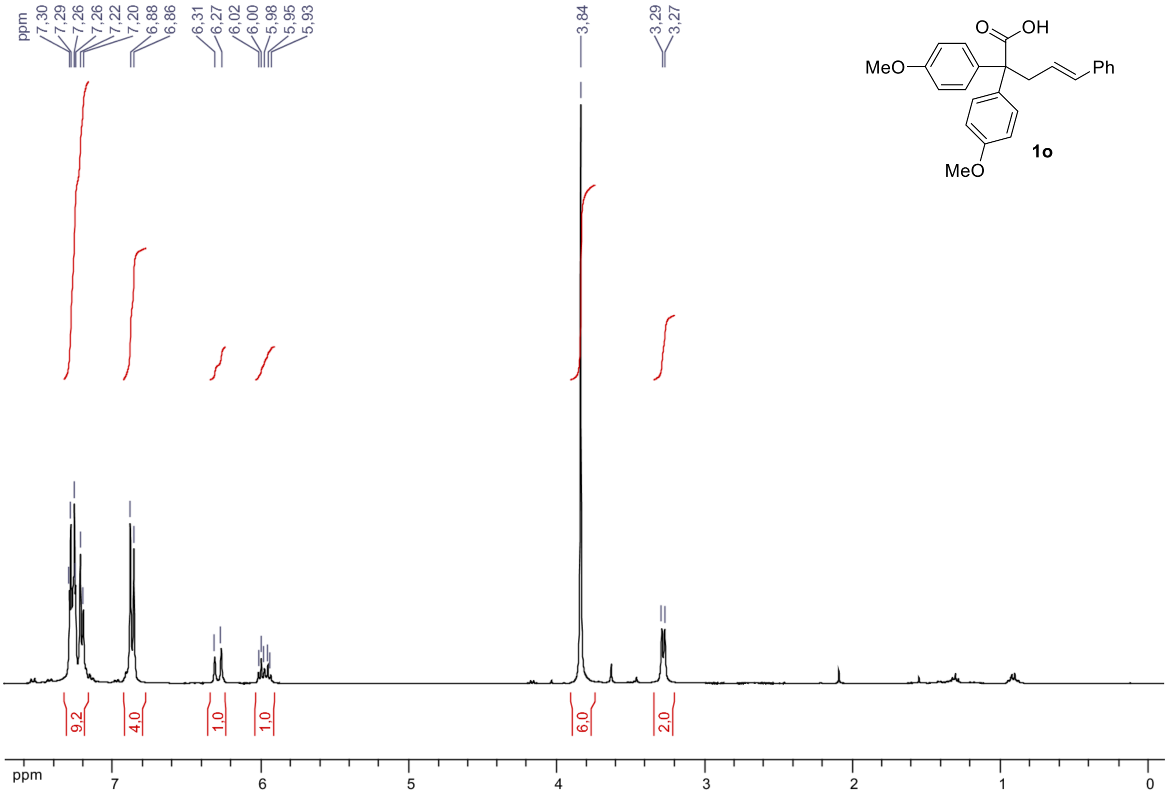
1m

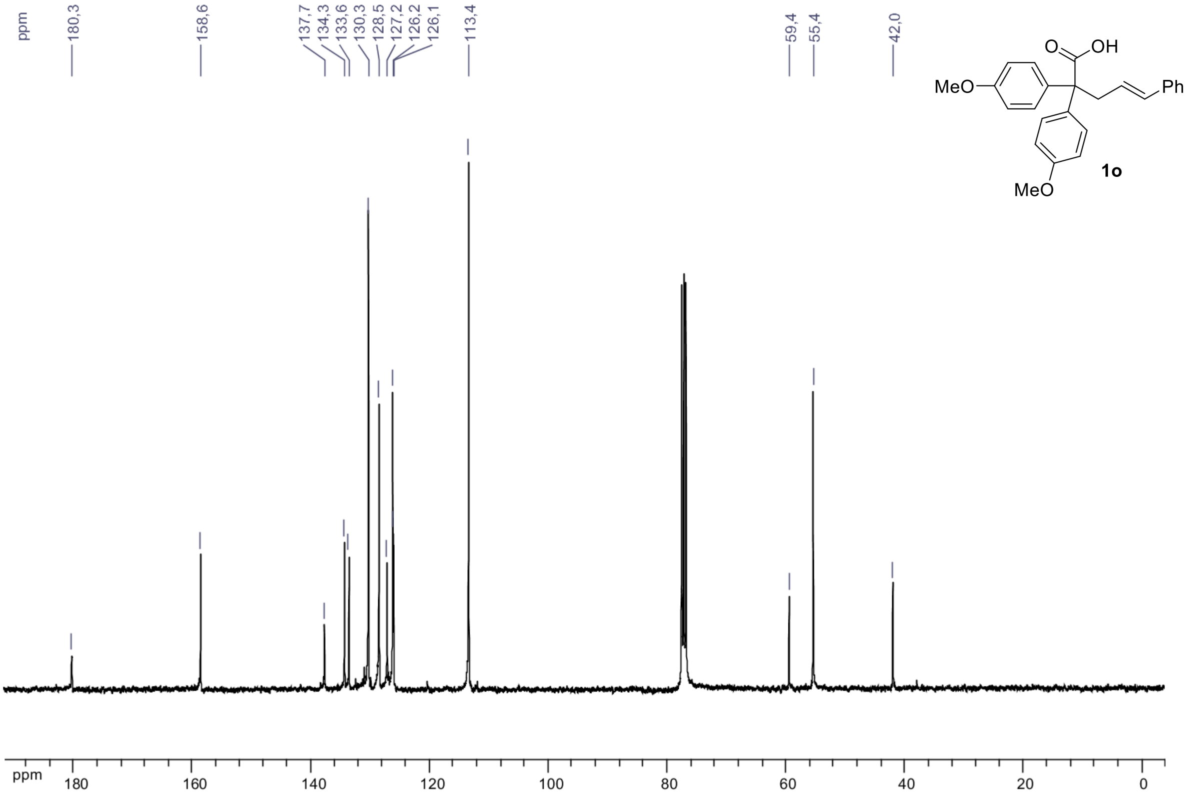

ppm
180,1
142,0 137,6 133,7 129,3 128,5 128,1 127,3 127,2 126,2 125,8
60,8
41,7
ppm 180 160 140 120 100 80 60 40 20 0
O
OH
Ph
Ph
Ph

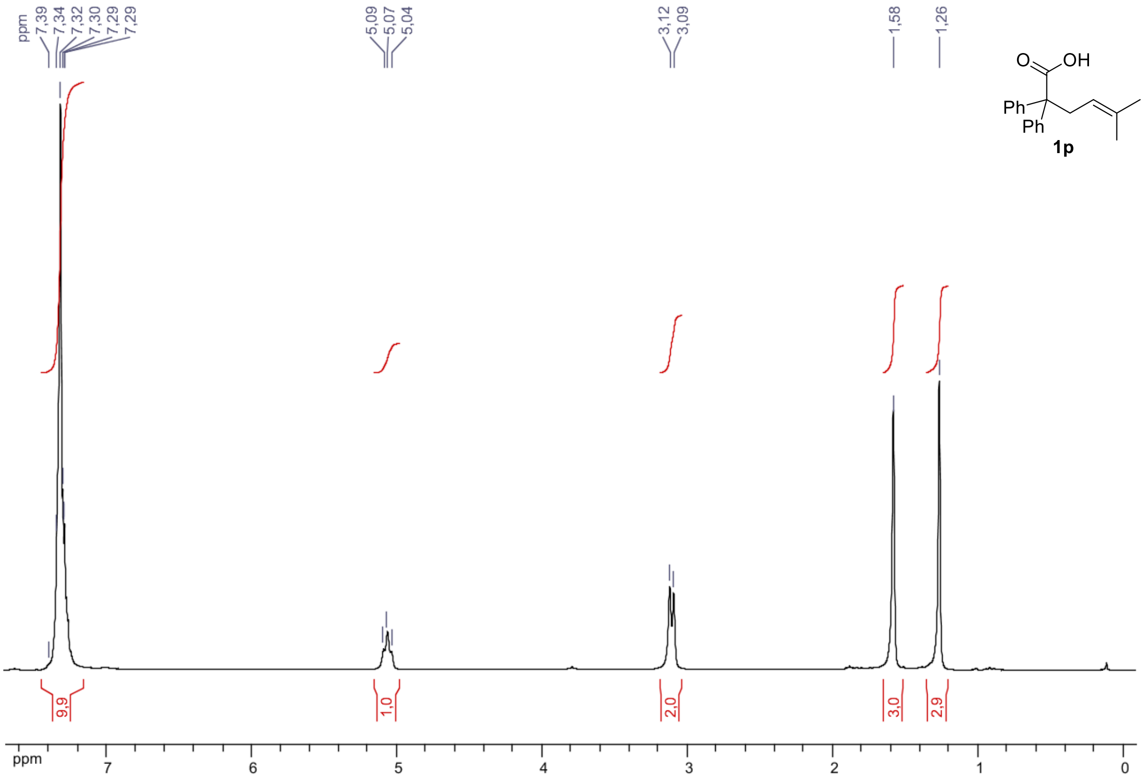
1m

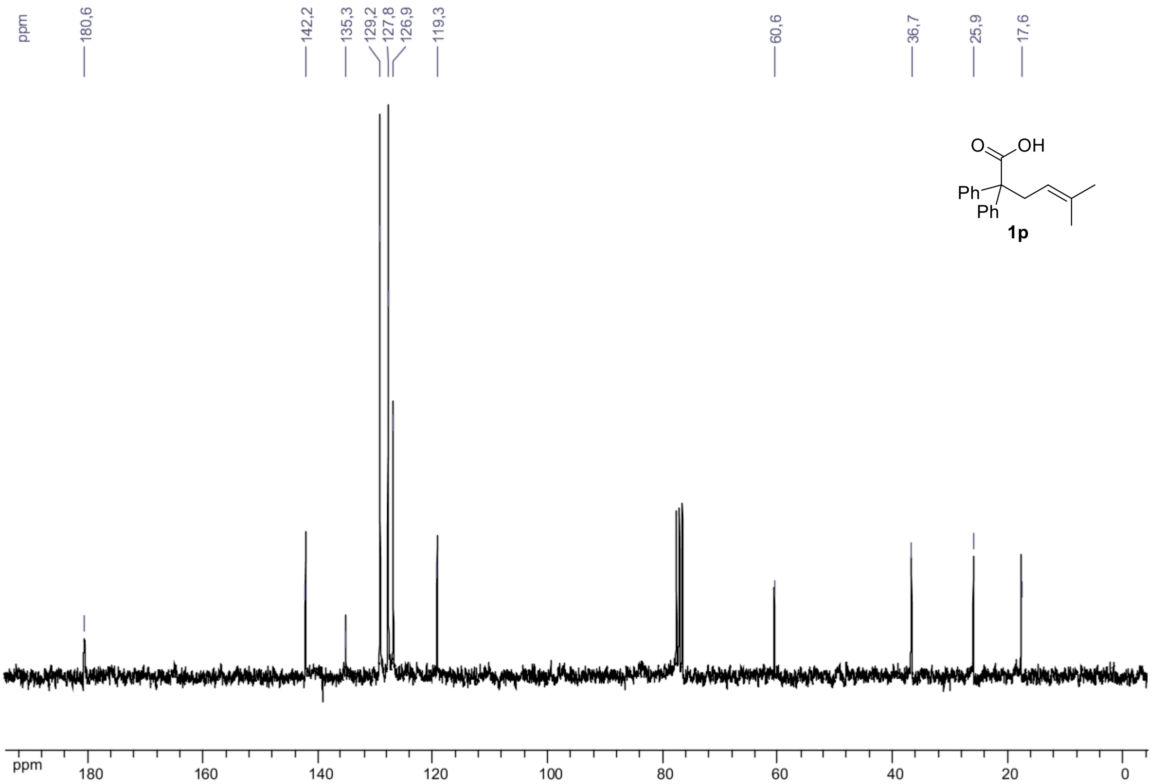

ppm
7,35
7,33
7,29
7,28
7,26
7,24
7,21
7,19
7,17
6,25
6,20
6,03
6,01
5,99
5,99
5,97
5,95
3,31
3,29
1,34
O
OH
t-Bu
Ph
t-Bu
1n
3,0
1,0
1,0
2,0
18,2
ppm
7
6
5
4
3
2
1

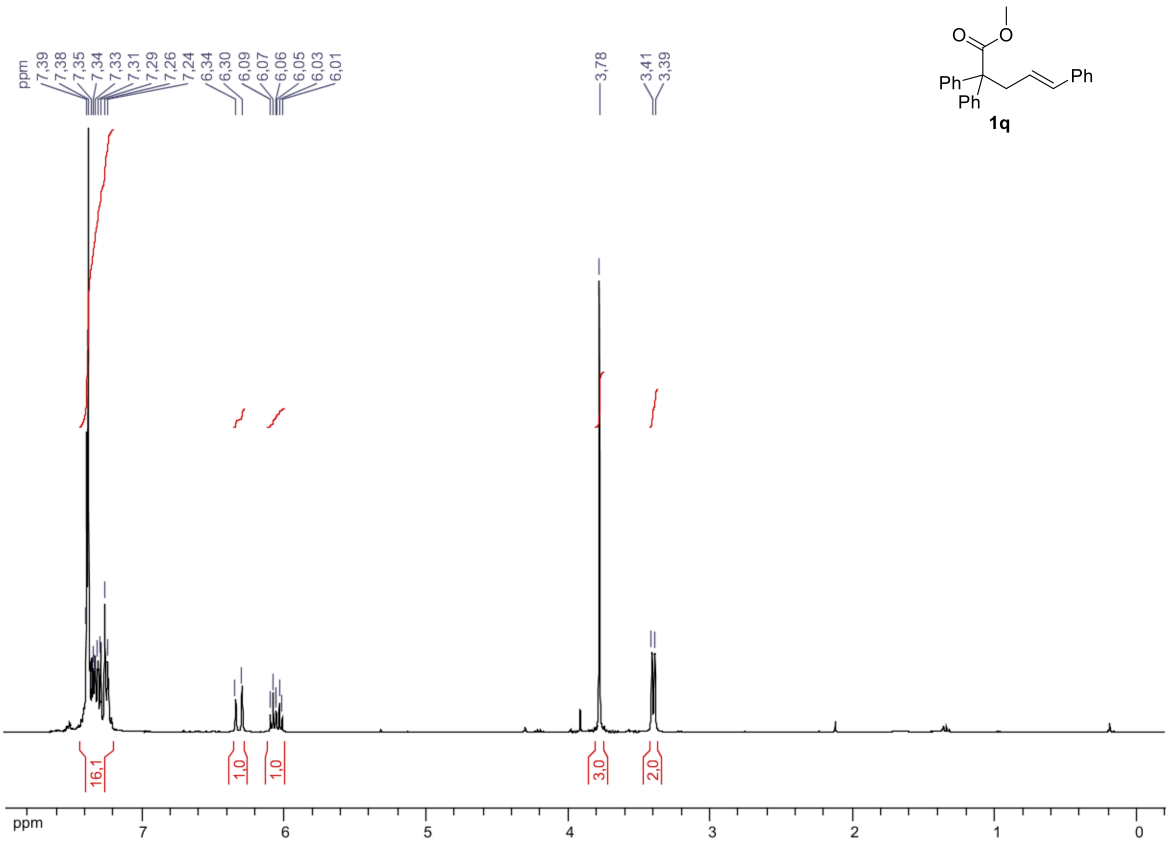

ppm
179,6
149,9
139,0
137,8
133,5
128,8
128,5
127,1
126,3
126,2
124,9
60,1
41,8
34,6
31,5
O
OH
t-Bu
Ph
t-Bu
1n
ppm
180
160
140
120
100
80
60
40
20
0

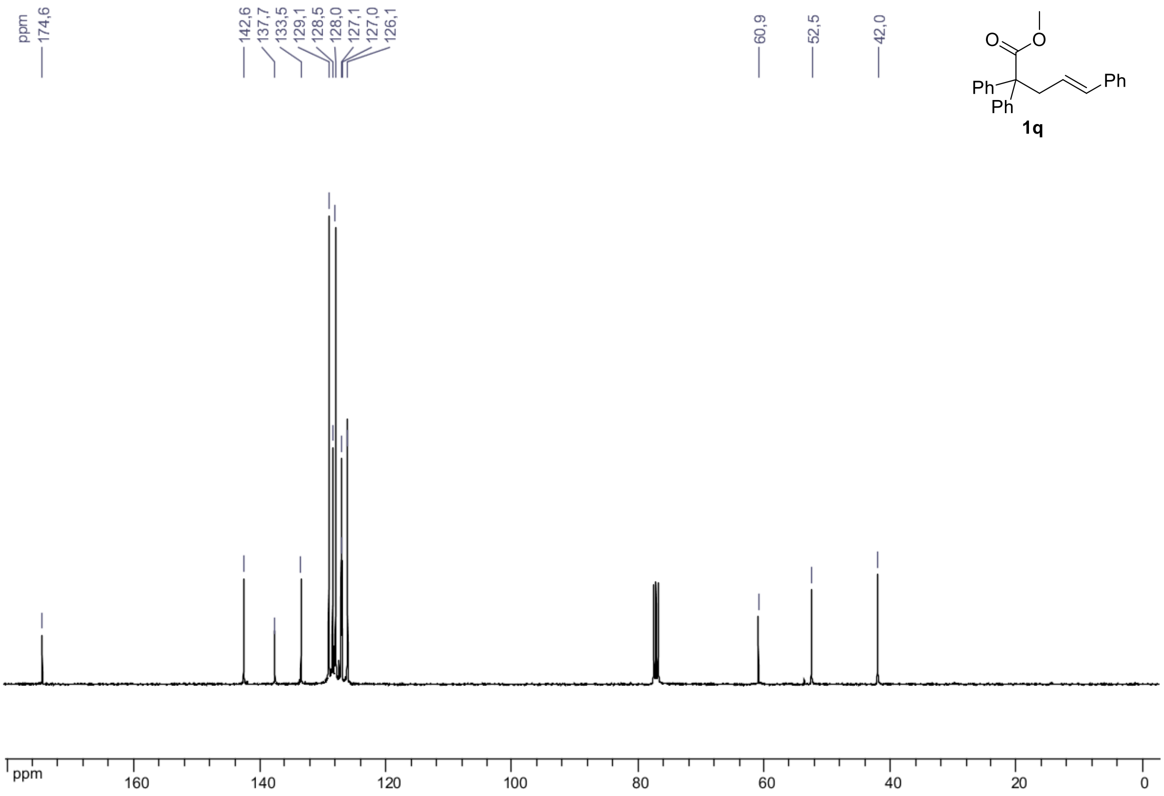

ppm 7,30 7,29 7,26 7,26 7,22 7,20 6,88 6,86 6,31 6,27 6,02 6,00 5,98 5,95 5,93
3,84
3,29 3,27
O OH
MeO
Ph
MeO
1o
9,2
4,0
1,0
1,0
6,0
2,0
ppm 7 6 5 4 3 2 1 0

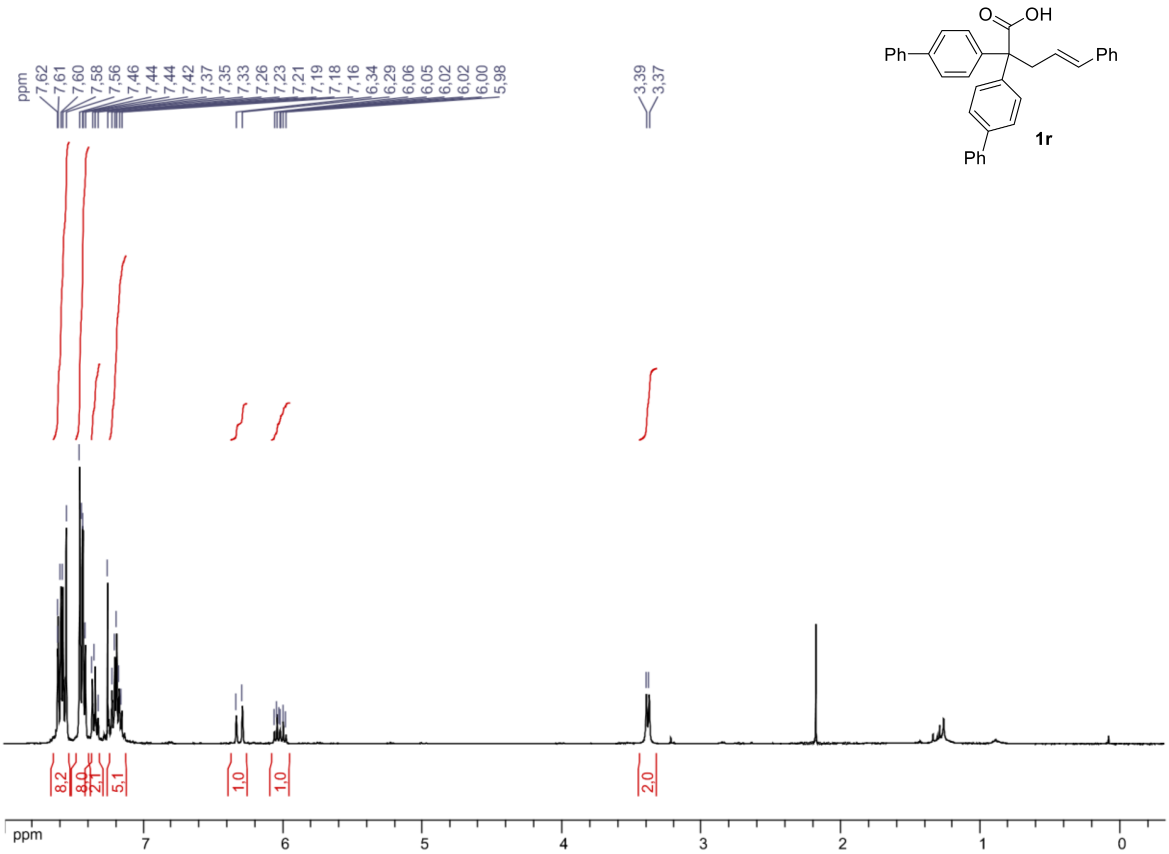

ppm 180,3
158,6
137,7 134,3 133,6 130,3 128,5 127,2 126,2 126,1
113,4
59,4 55,4
42,0
O OH
MeO
Ph
MeO
1o
ppm 180 160 140 120 100 80 60 40 20 0

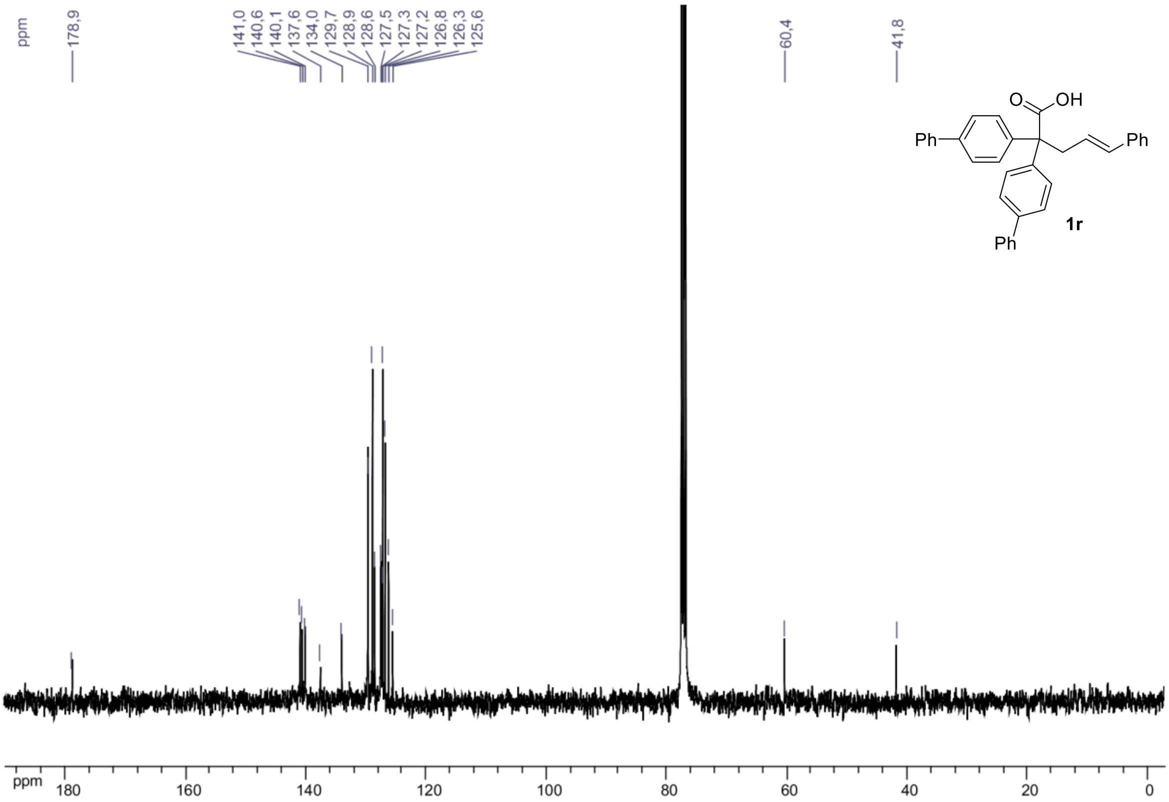

ppm
7,39
7,34
7,32
7,30
7,29
7,29
5,09
5,07
5,04
3,12
3,09
1,58
1,26
O
OH
Ph
Ph
1p
9,9
1,0
2,0
3,0
2,9
ppm
7
6
5
4
3
2
1
0

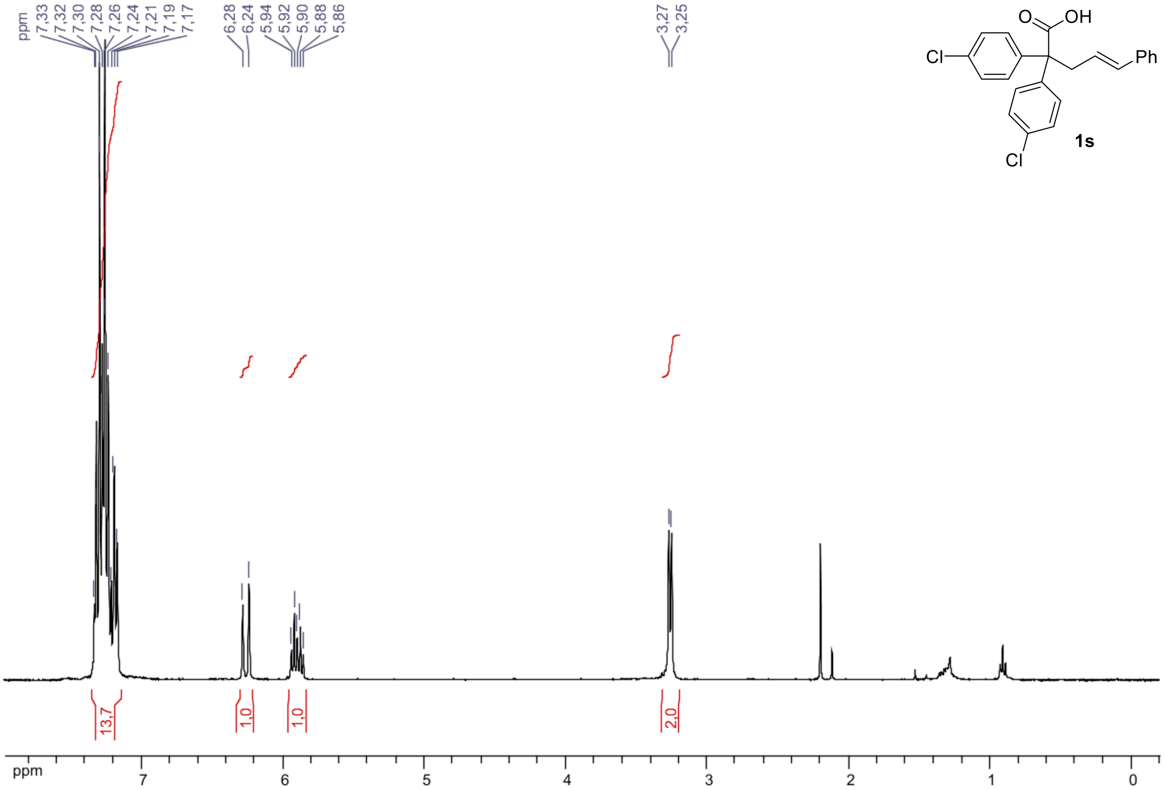

ppm
180,6
142,2
135,3
129,2
127,8
126,9
119,3
60,6
36,7
25,9
17,6
O
OH
Ph
Ph
1p
ppm
180
160
140
120
100
80
60
40
20
0


ppm
7,39
7,38
7,35
7,34
7,33
7,31
7,29
7,26
7,24
6,34
6,30
6,09
6,07
6,06
6,05
6,03
6,01
3,78
3,41
3,39
16,1
1,0
1,0
3,0
2,0
ppm
7
6
5
4
3
2
1
0
O
O
Ph
Ph
Ph
1q

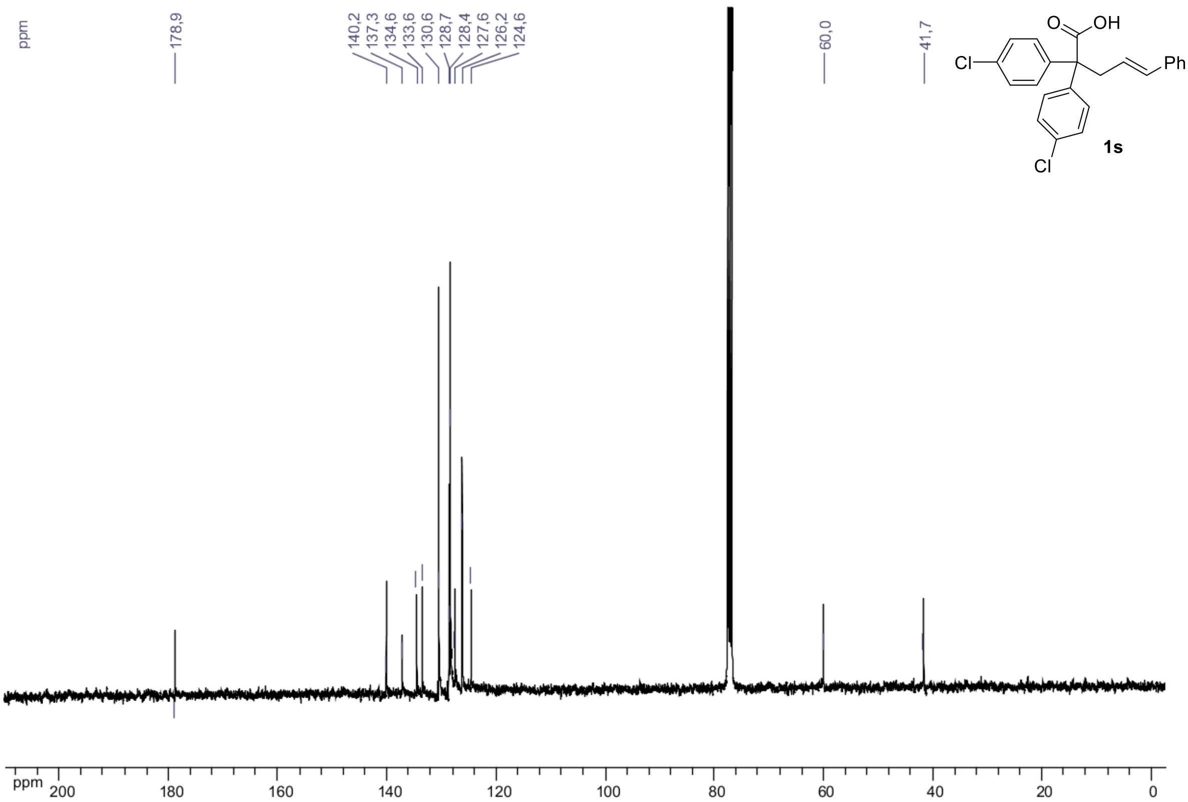

ppm
174,6
142,6
137,7
133,5
129,1
128,5
128,0
127,1
127,0
126,1
60,9
52,5
42,0
ppm
160
140
120
100
80
60
40
20
0
O
O
Ph
Ph
Ph
1q


ppm
7,62
7,61
7,60
7,58
7,56
7,46
7,44
7,44
7,42
7,37
7,35
7,33
7,26
7,23
7,21
7,19
7,18
7,16
6,34
6,29
6,06
6,05
6,02
6,02
6,00
5,98
3,39
3,37
8,2
8,0
2,1
5,1
1,0
1,0
2,0
ppm
7
6
5
4
3
2
1
0
Ph
O
OH
Ph
Ph
1r

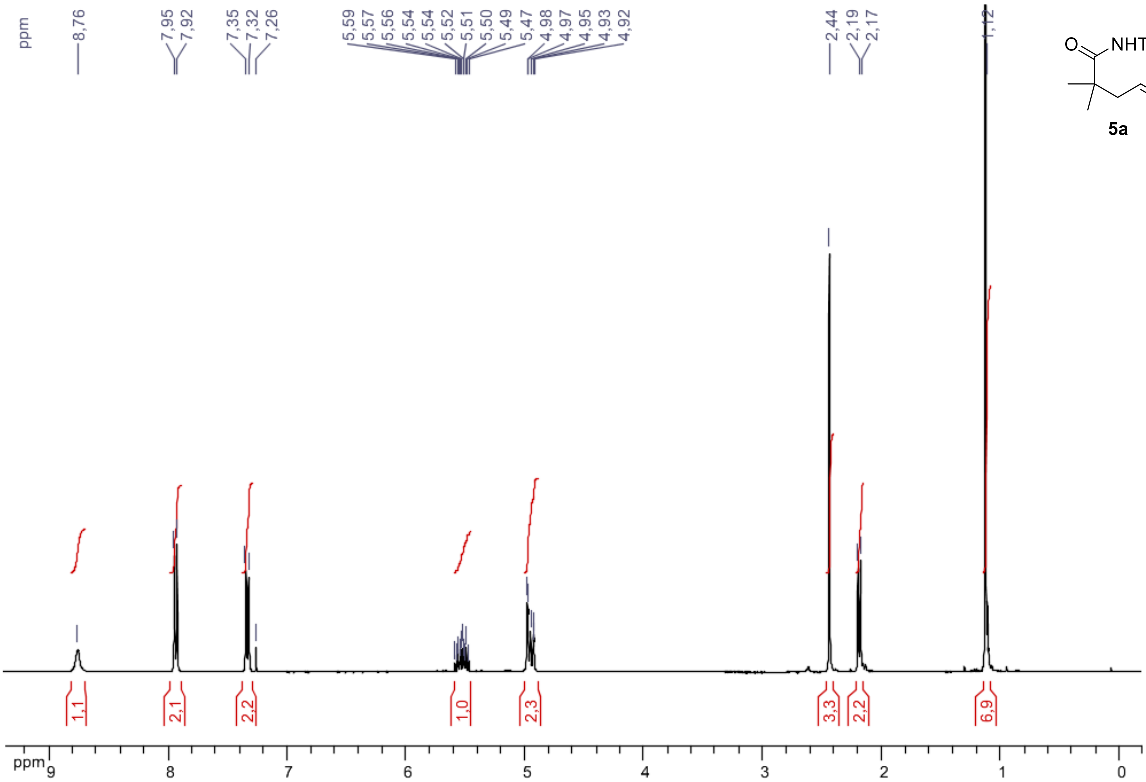

ppm
178,9
141,0
140,6
140,1
137,6
134,0
129,7
128,9
128,6
127,5
127,3
127,2
126,8
126,3
125,6
60,4
41,8
ppm
180
160
140
120
100
80
60
40
20
0
Ph
O
OH
Ph
Ph
1r



ppm
7,33
7,32
7,30
7,28
7,26
7,24
7,21
7,19
7,17
6,28
6,24
5,94
5,92
5,90
5,88
5,86
3,27
3,25
13,7
1,0
1,0
2,0
ppm
7
6
5
4
3
2
1
0
O
OH
Cl
Ph
Cl
1s

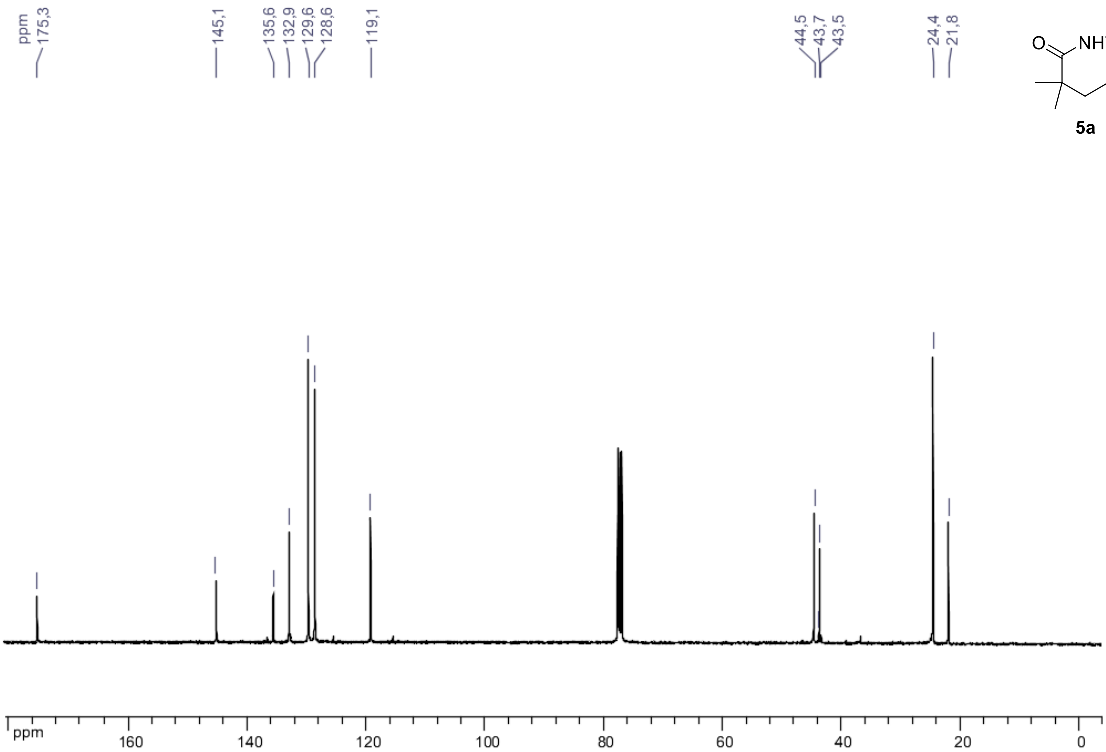


ppm
178,9
140,2
137,3
134,6
133,6
130,6
128,7
128,4
127,6
126,2
124,6
60,0
41,7
ppm
200
180
160
140
120
100
80
60
40
20
0
O
OH
Cl
Ph
Cl
1s


ppm
8,76
7,95
7,92
7,35
7,32
7,26
5,59
5,57
5,56
5,54
5,54
5,52
5,51
5,50
5,49
5,47
4,98
4,97
4,95
4,93
4,92
2,44
2,19
2,17
1,12
1,1
2,1
2,2
1,0
2,3
3,3
2,2
6,9
ppm 9 8 7 6 5 4 3 2 1 0

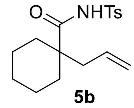

O
NHTs

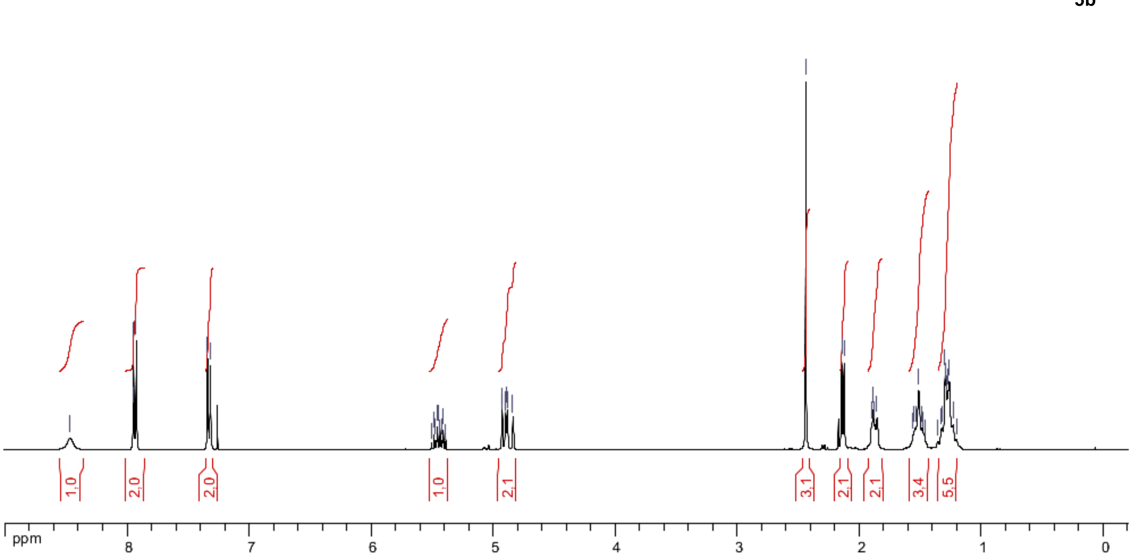
5a

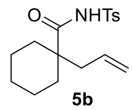

ppm
175,3
145,1
135,6
132,9
129,6
128,6
119,1
44,5
43,7
43,5
24,4
21,8
ppm 160 140 120 100 80 60 40 20 0

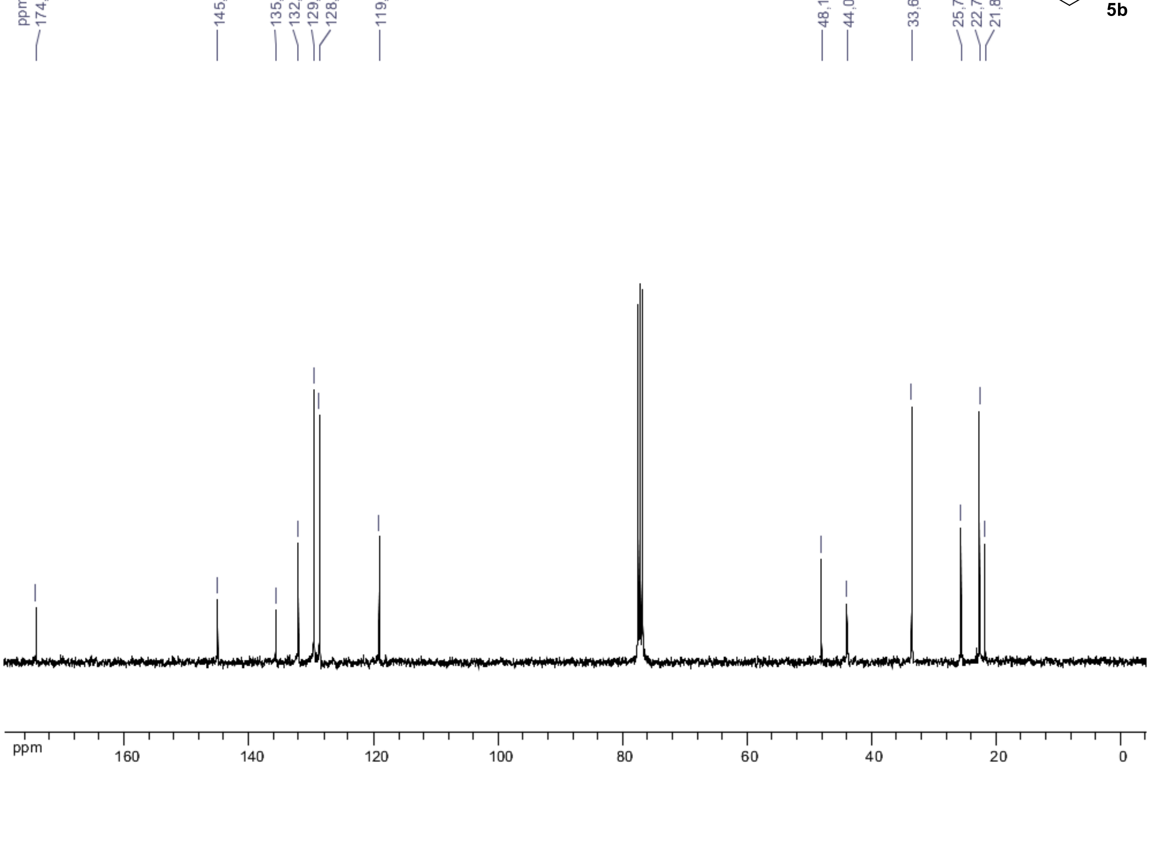

O
NHTs

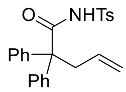
5a


O
NHTs
5b

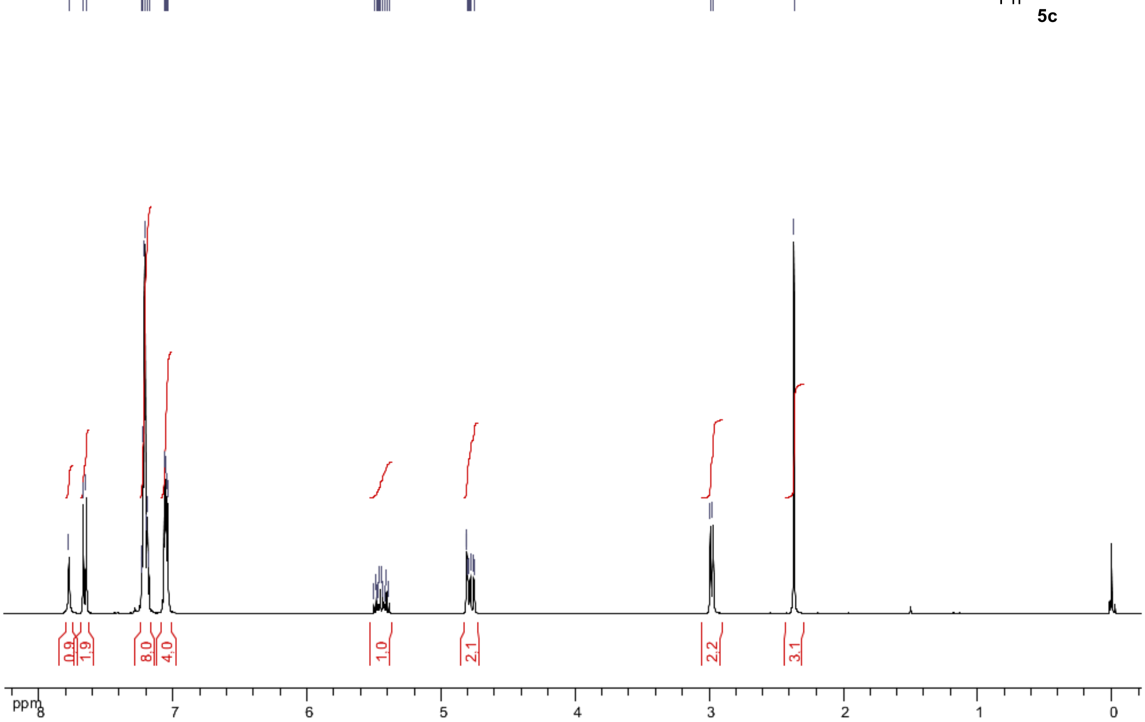

ppm
8,47
7,95
7,94
7,93
7,34
7,32
5,51
5,48
5,48
5,46
5,44
5,43
5,42
5,41
5,39
4,93
4,93
4,90
4,90
4,89
4,88
4,84
4,83
2,44
2,15
2,12
1,90
1,89
1,87
1,85
1,56
1,55
1,54
1,51
1,48
1,47
1,46
1,36
1,33
1,32
1,30
1,29
1,28
1,27
1,26
1,23
1,20
1,0
2,0
2,0
1,0
2,1
3,1
2,1
2,1
3,4
5,5
ppm
8
7
6
5
4
3
2
1
0

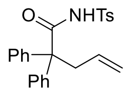

O
NHTs
5b


ppm
174,2
145,1
135,7
132,1
129,6
128,7
119,1
48,1
44,0
33,6
25,7
22,7
21,8
ppm
160
140
120
100
80
60
40
20
0

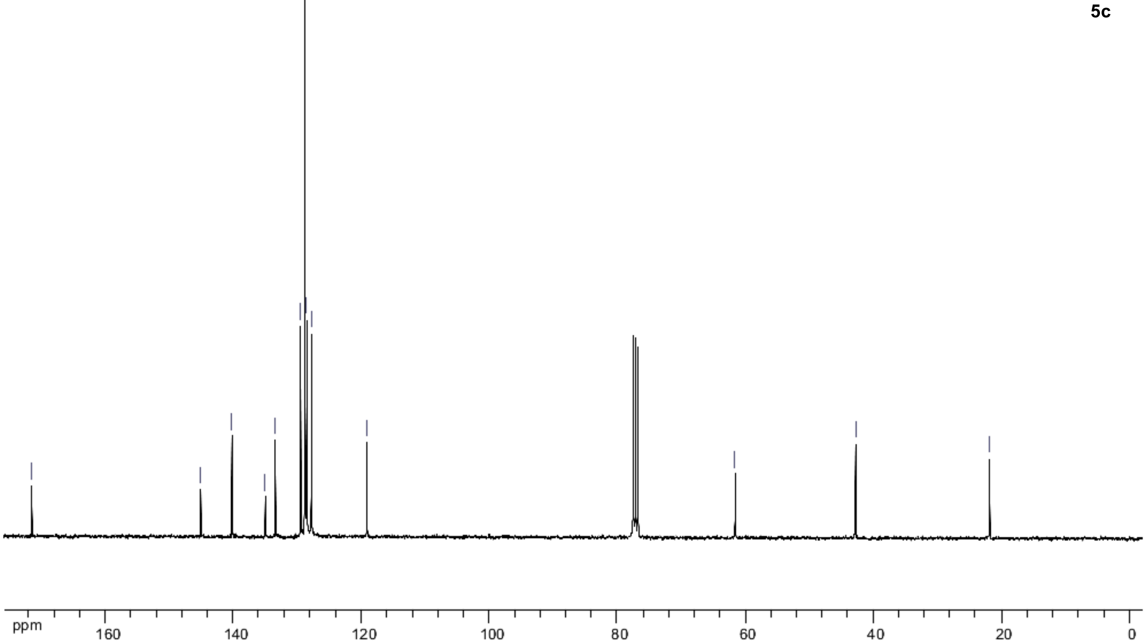

O
NHTs
Ph
Ph

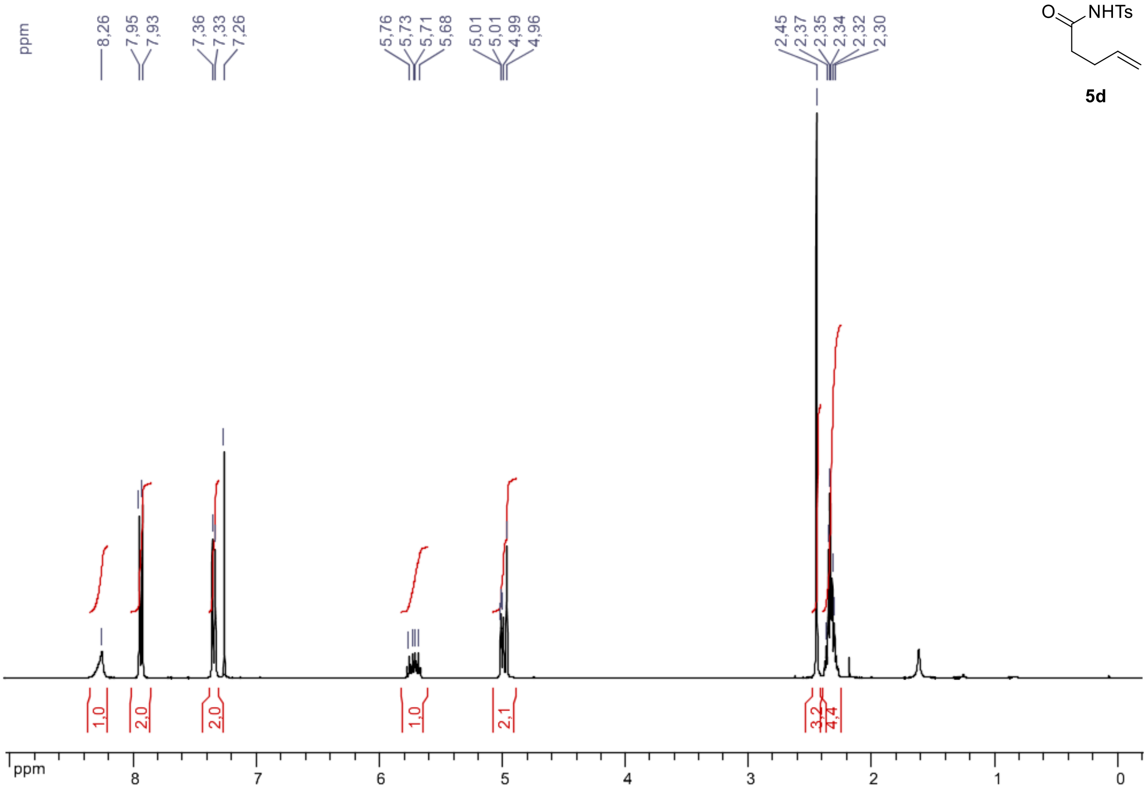
5c

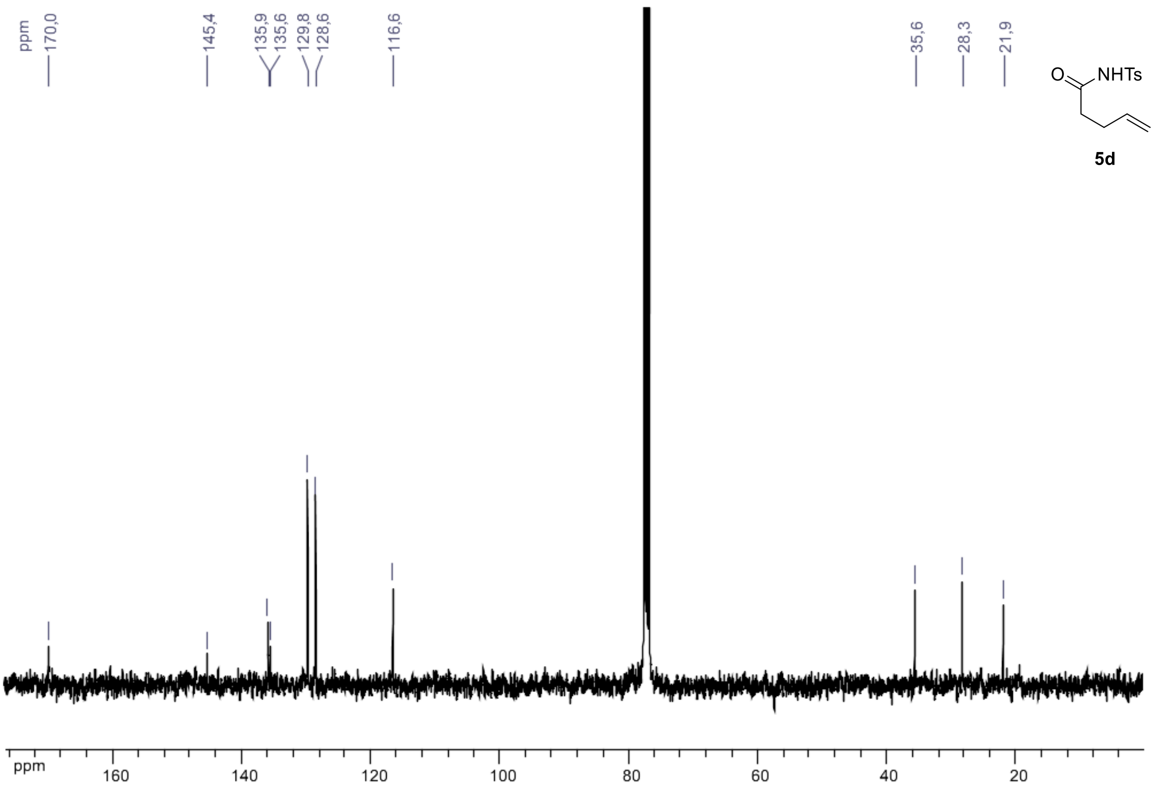

ppm
7,78
7,67
7,65
7,24
7,23
7,23
7,22
7,21
7,20
7,19
7,18
7,07
7,06
7,05
7,04
5,50
5,48
5,48
5,47
5,46
5,44
5,43
5,42
5,41
5,39
4,81
4,81
4,80
4,79
4,78
4,76
4,75
2,99
2,97
2,37
0,9
1,9
8,0
4,0
1,0
2,1
2,2
3,1
ppm
8
7
6
5
4
3
2
1
0

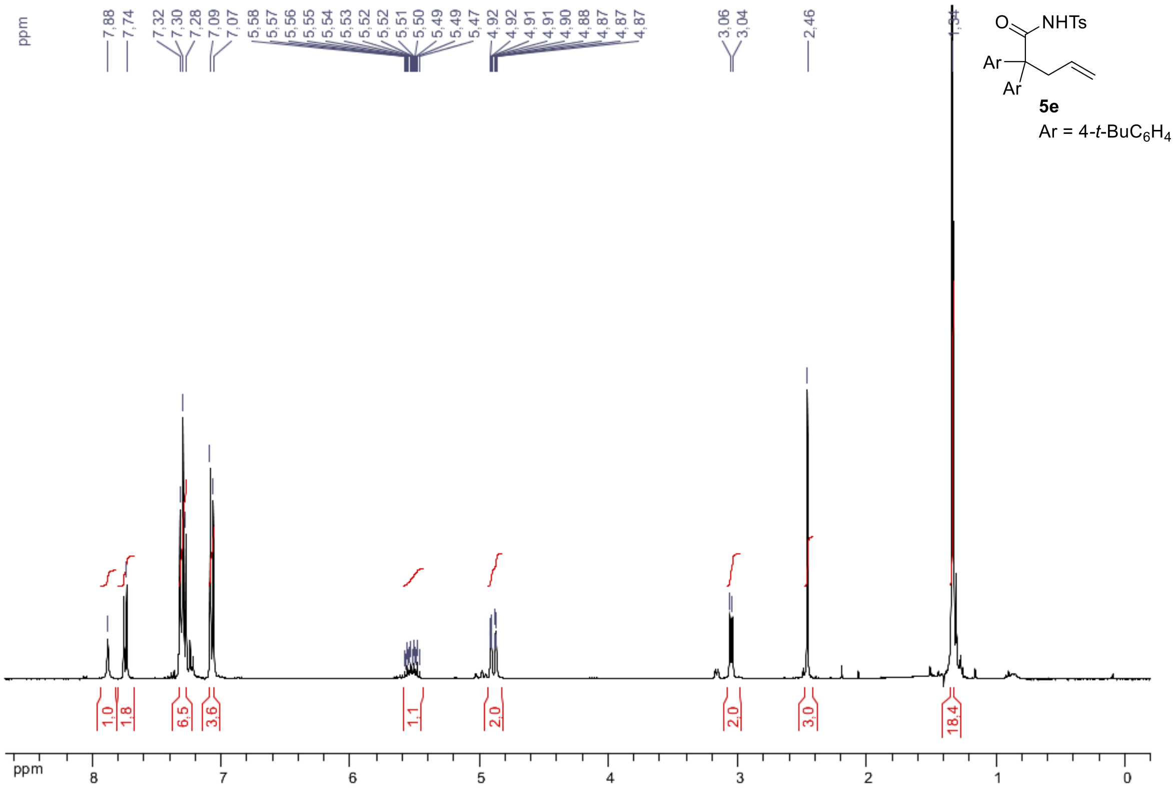

O
NHTs
Ph
Ph

5c

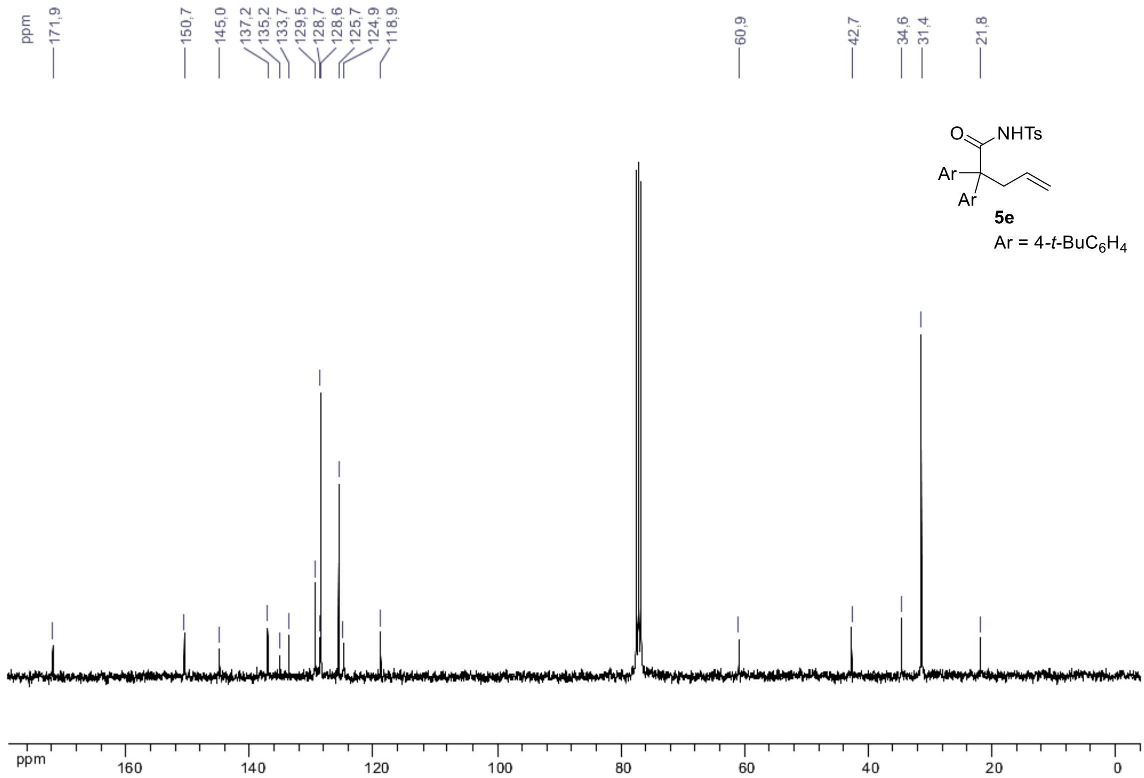

ppm
171,5
145,1
140,2
135,0
133,5
129,5
128,8
128,5
127,8
119,2
61,6
42,8
21,8
ppm
160
140
120
100
80
60
40
20
0


ppm
8,26
7,95
7,93
7,36
7,33
7,26
5,76
5,73
5,71
5,68
5,01
5,01
4,99
4,96
2,45
2,37
2,35
2,34
2,32
2,30
O
NHTs
5d
1,0
2,0
2,0
1,0
2,1
3,2
4,4
ppm
8
7
6
5
4
3
2
1
0

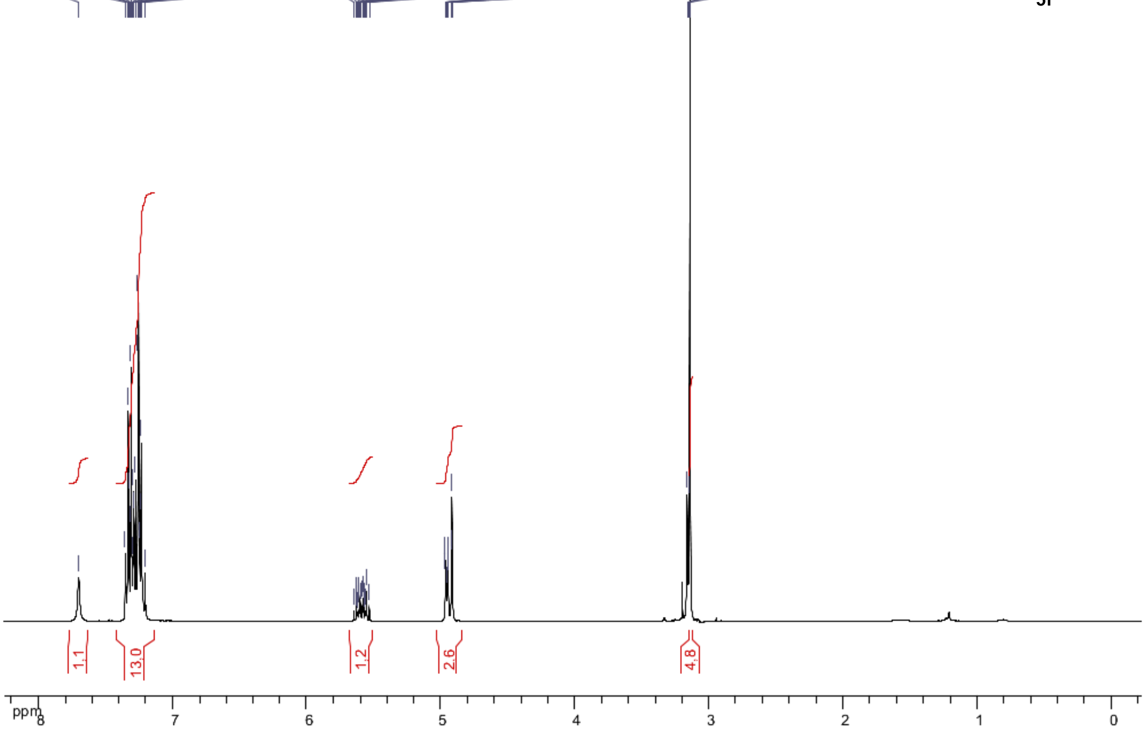

ppm
170,0
145,4
135,9
135,6
129,8
128,6
116,6
35,6
28,3
21,9
O
NHTs
5d
ppm
160
140
120
100
80
60
40
20

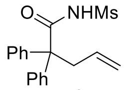


ppm
7,88
7,74
7,32
7,30
7,28
7,09
7,07
5,58
5,57
5,56
5,55
5,54
5,53
5,52
5,52
5,51
5,50
5,49
5,49
5,47
4,92
4,92
4,91
4,91
4,90
4,88
4,87
4,87
4,87
3,06
3,04
2,46
1,34
O
NHTs
Ar
Ar
5e
Ar = 4-t-BuC6H4
1,0
1,8
6,5
3,6
1,1
2,0
2,0
3,0
18,4
ppm 8 7 6 5 4 3 2 1 0

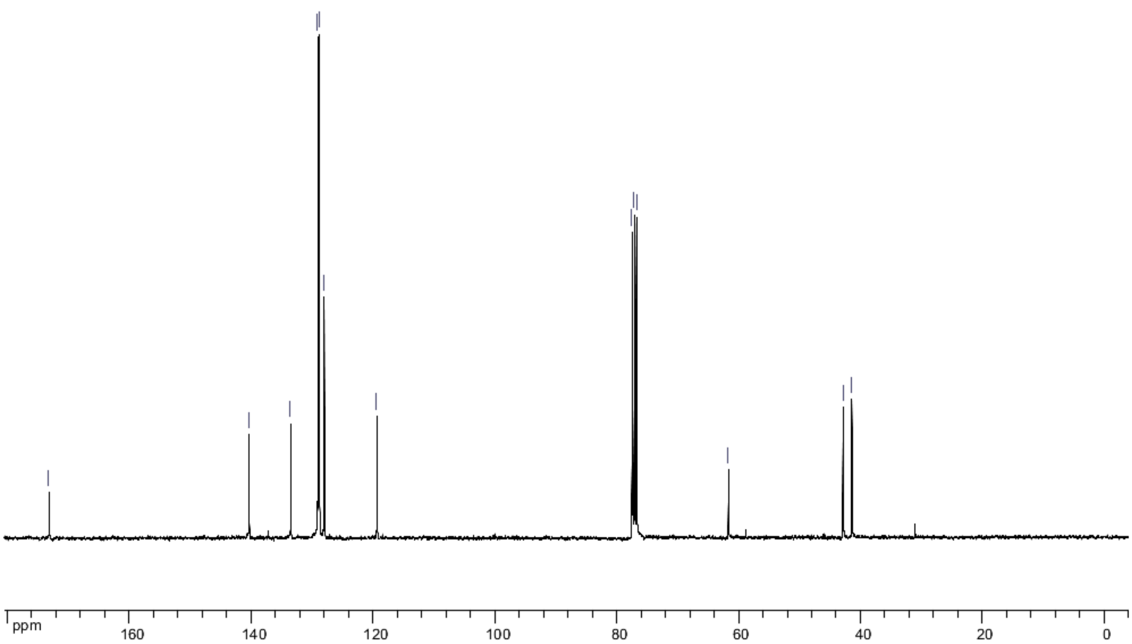


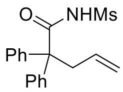
ppm
171,9
150,7
145,0
137,2
135,2
133,7
129,5
128,7
128,6
125,7
124,9
118,9
60,9
42,7
34,6
31,4
21,8
O
NHTs
Ar
Ar
5e
Ar = 4-t-BuC6H4
ppm 160 140 120 100 80 60 40 20 0


ppm 7,70 7,35 7,33 7,33 7,32 7,31 7,30 7,29 7,29 7,28 7,26 7,26 7,25 7,24 7,24 7,21 5,65 5,63 5,62 5,62 5,61 5,60 5,60 5,59 5,58 5,57 5,57 5,56 5,55 5,54 4,97 4,96 4,96 4,95 4,95 4,92 4,92 4,91 3,16 3,15 3,14
1,1
13,0
1,2
2,6
4,8
ppm 8 7 6 5 4 3 2 1 0

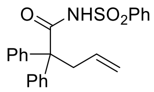

O
NHMs
Ph
Ph

5f

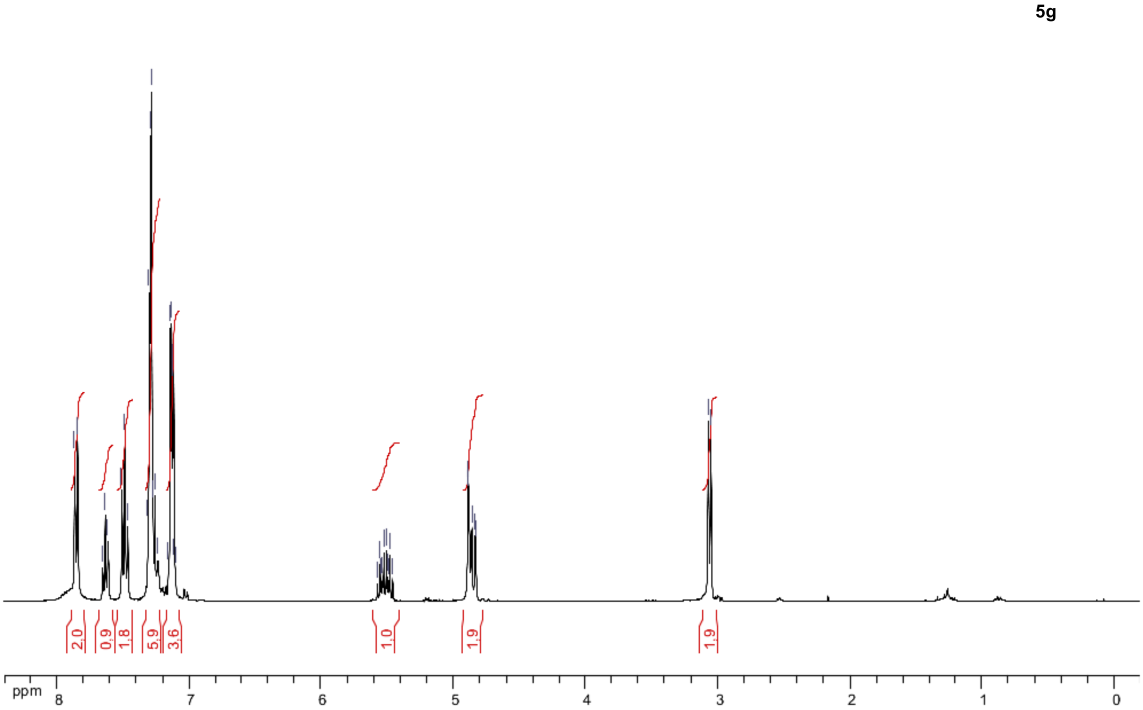

ppm 173,2 140,4 133,5 129,0 128,9 128,0 119,4 77,5 77,2 76,8 61,7 42,9 41,4
ppm 160 140 120 100 80 60 40 20 0

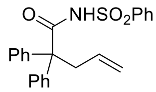

O
NHMs
Ph
Ph

5f

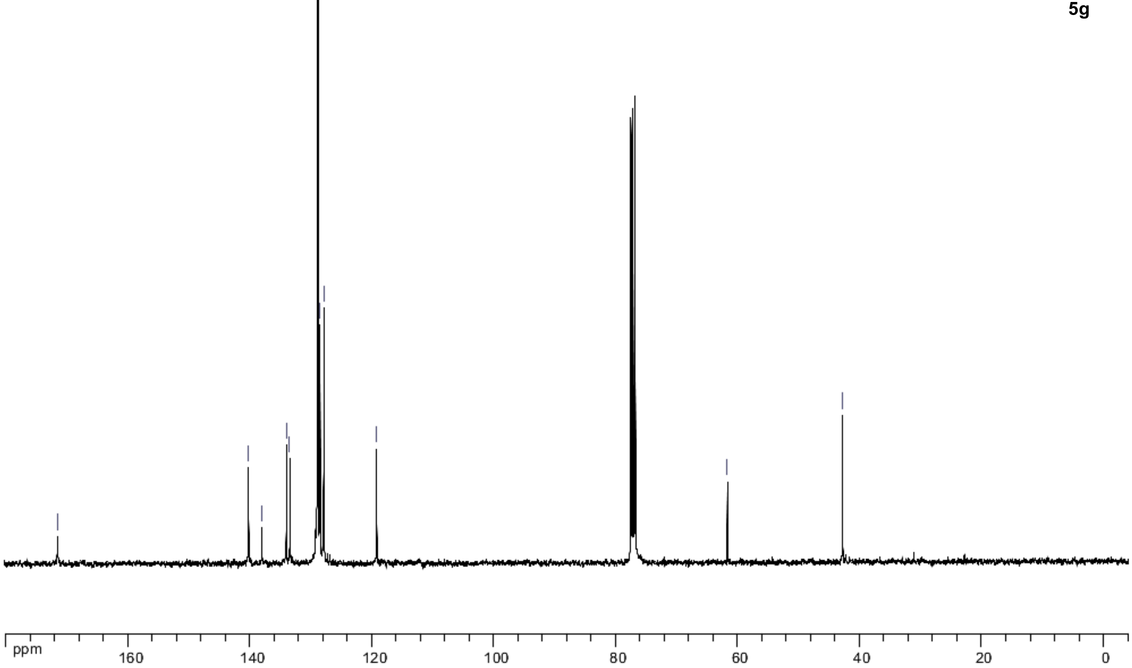

O
NHSO2Ph
Ph
Ph

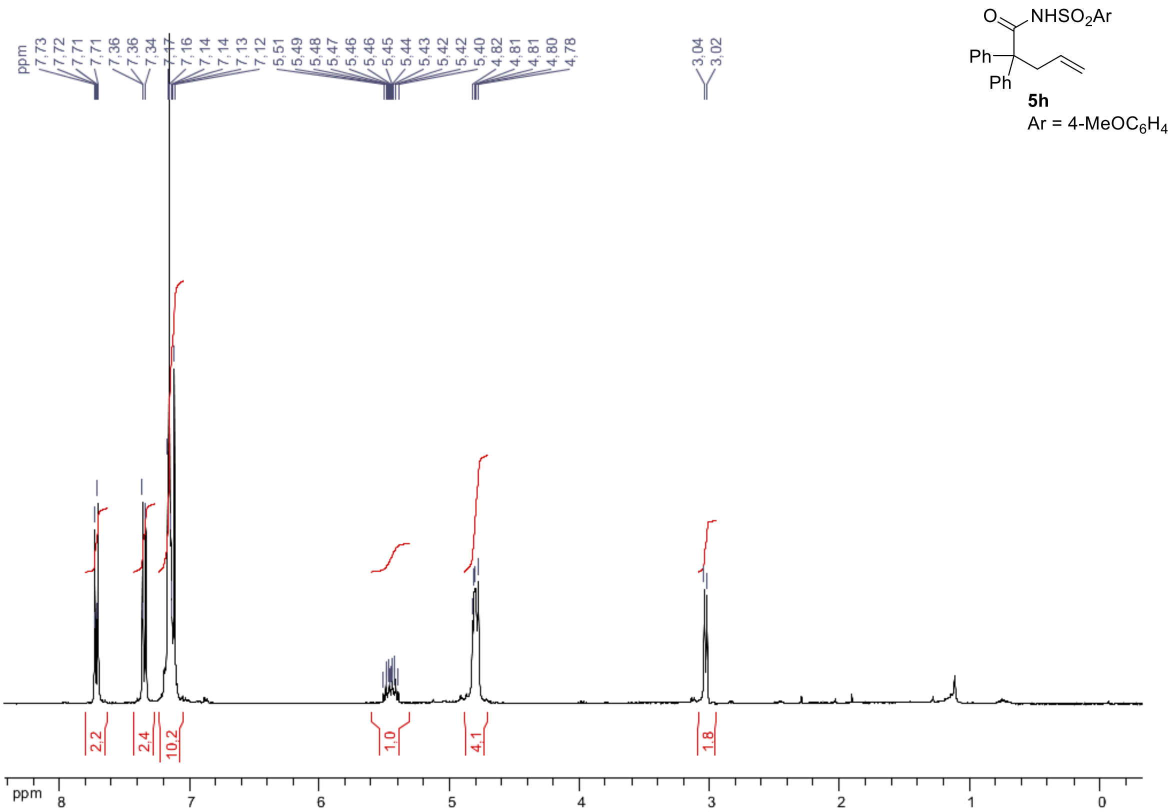
5g


ppm
7,86 7,84 7,65 7,63 7,61 7,51 7,49 7,46 7,31 7,30 7,29 7,28 7,27 7,26 7,24 7,16 7,14 7,13 7,12 7,12 7,11 7,10 5,57 5,55 5,55 5,54 5,53 5,51 5,50 5,49 5,48 5,46 4,89 4,88 4,86 4,84 4,83 3,07 3,05
2,0 0,9 1,8 5,9 3,6 1,0 1,9 1,9
ppm 8 7 6 5 4 3 2 1 0

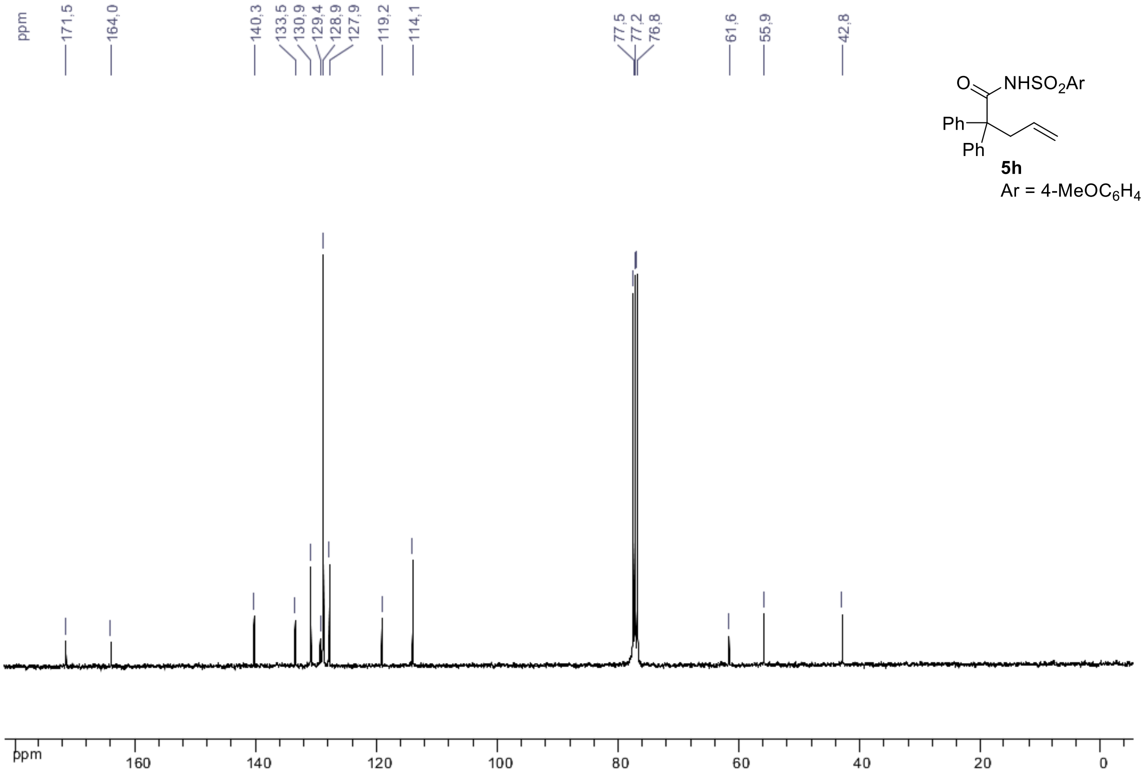

O
NHSO2Ph
Ph
Ph

5g

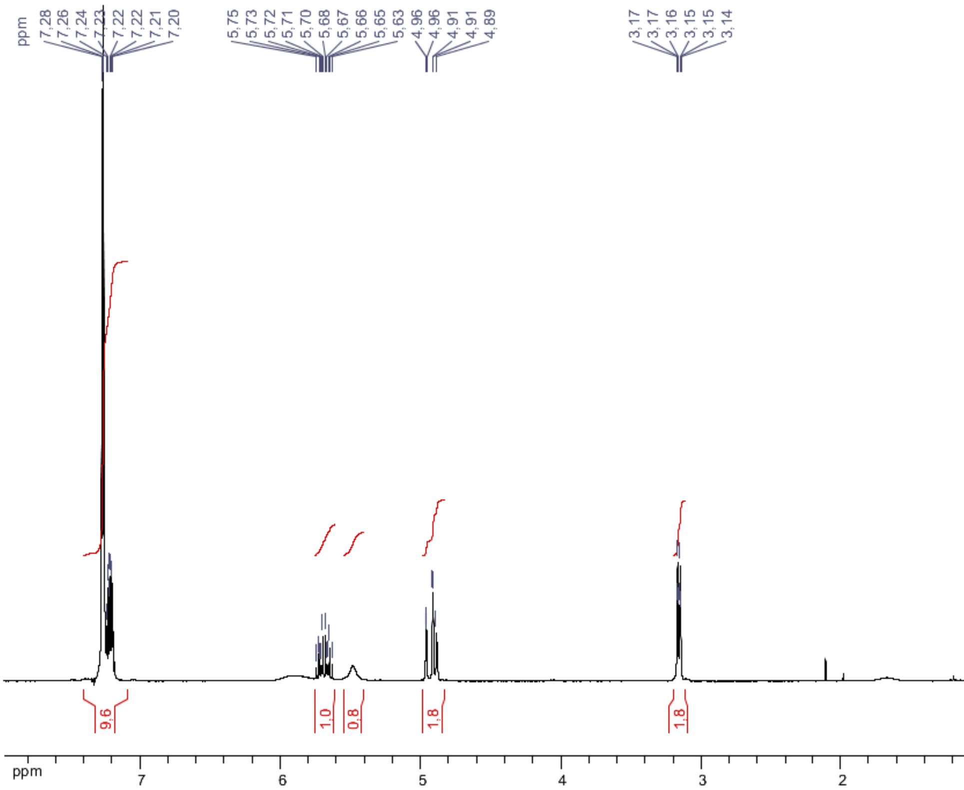

ppm
171,5
140,2 138,0 134,0 133,4 128,9 128,5 127,9 119,2
61,7
42,8
ppm 160 140 120 100 80 60 40 20 0

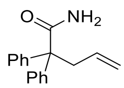


5h
Ar = 4-MeOC$_6$H$_4$



5h
Ar = 4-MeOC$_6$H$_4$





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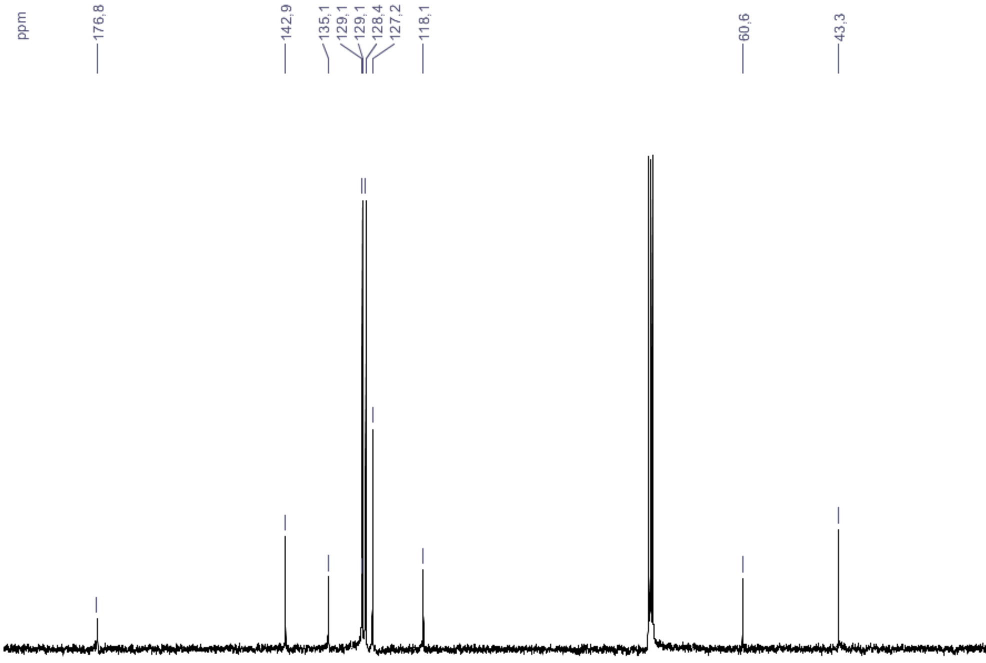

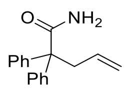

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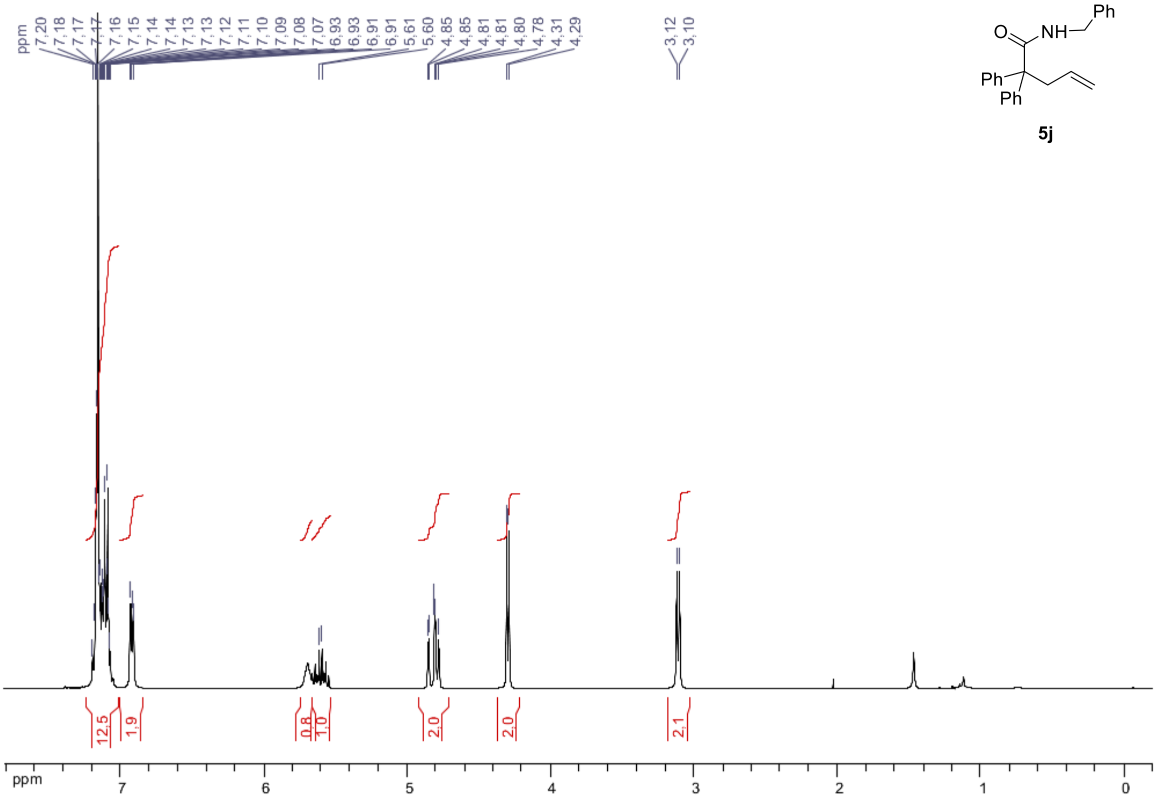
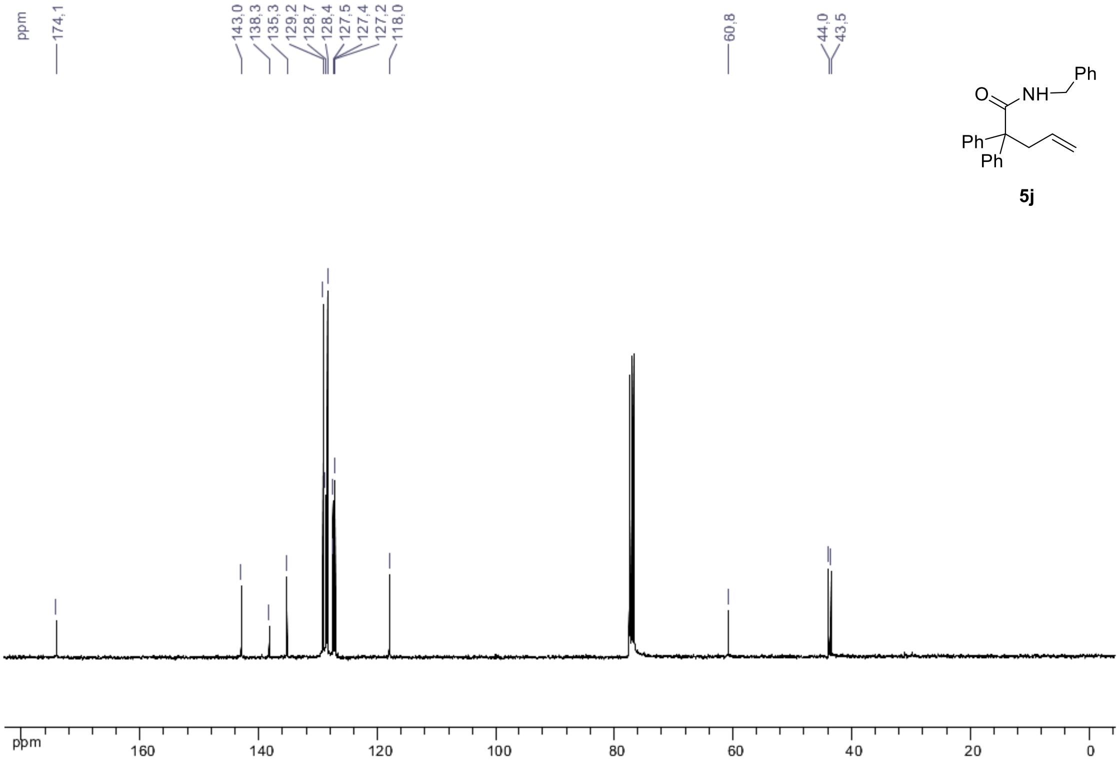


5j



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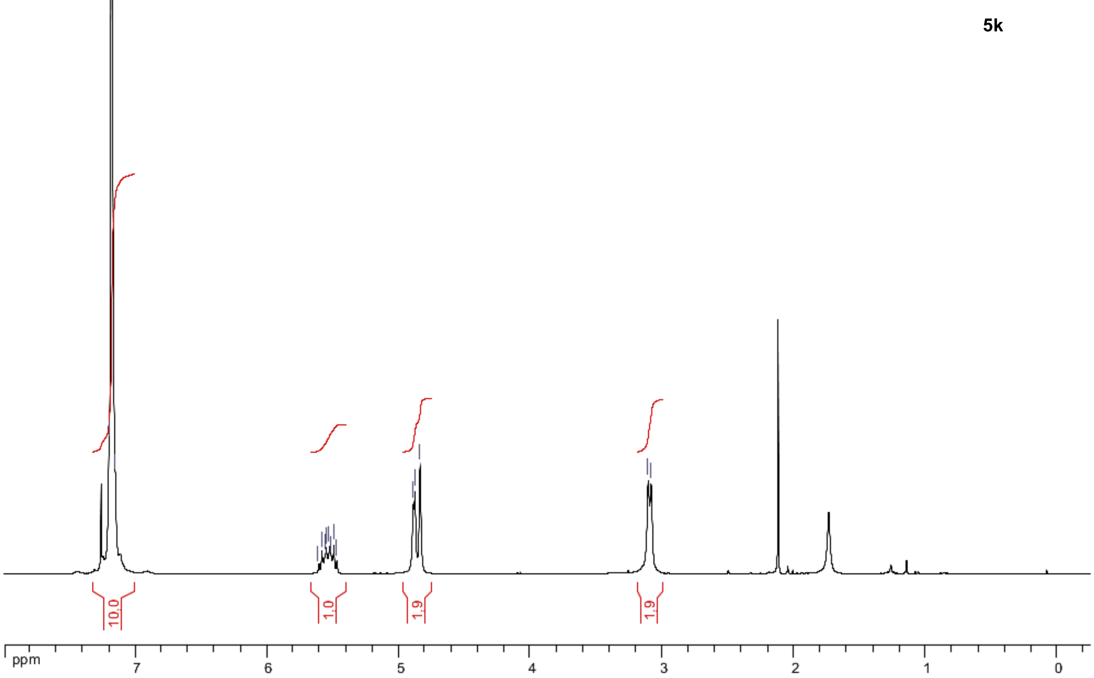
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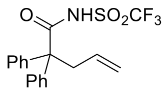
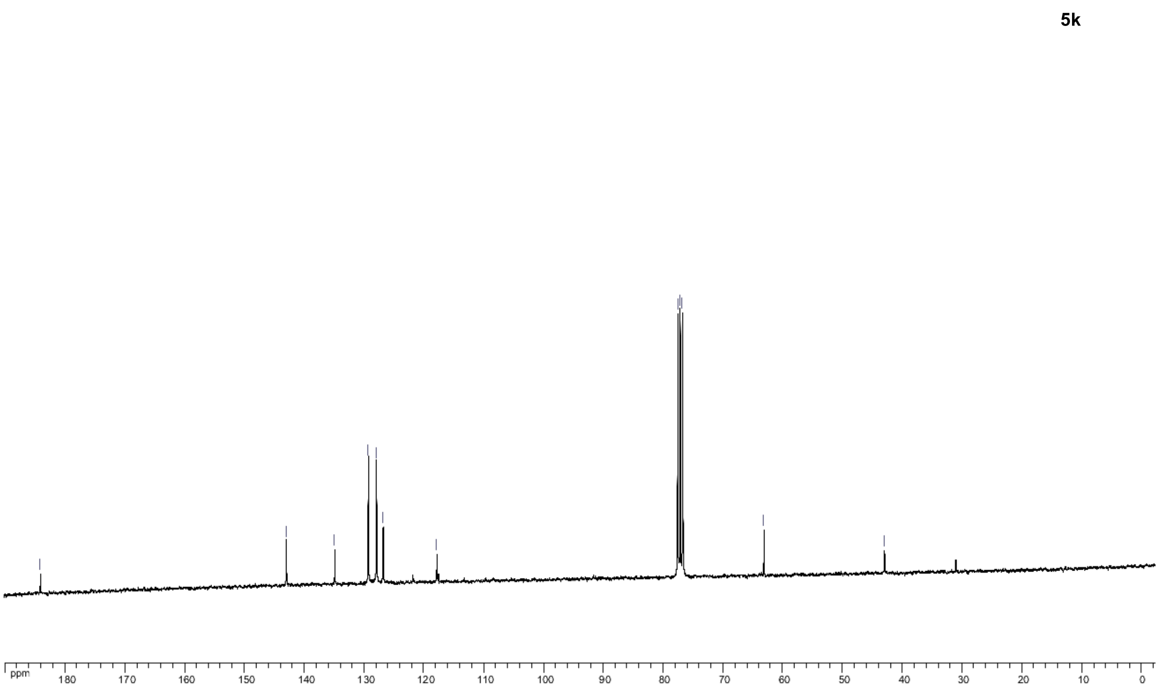
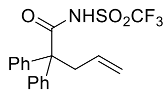
7,15
5,61
5,58
5,56
5,55
5,53
5,51
5,49
5,47
4,89
4,88
4,84
3,10
3,08
10,0
1,0
1,9
1,9
ppm
7
6
5
4
3
2
1
0

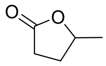

O
NHSO2CF3
Ph
Ph

5k

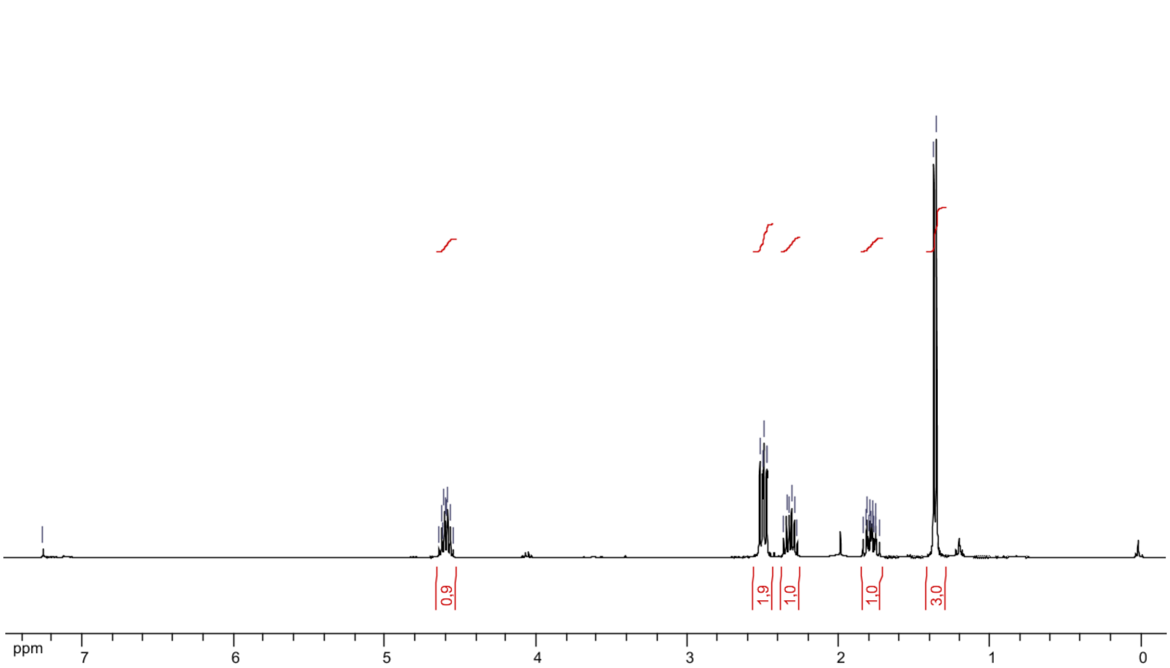

ppm
184,2
143,0
134,9
129,3
128,0
126,8
117,9
77,6
77,2
76,7
63,2
43,0
ppm
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


O
NHSO2CF3
Ph
Ph

5k



2a



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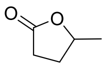
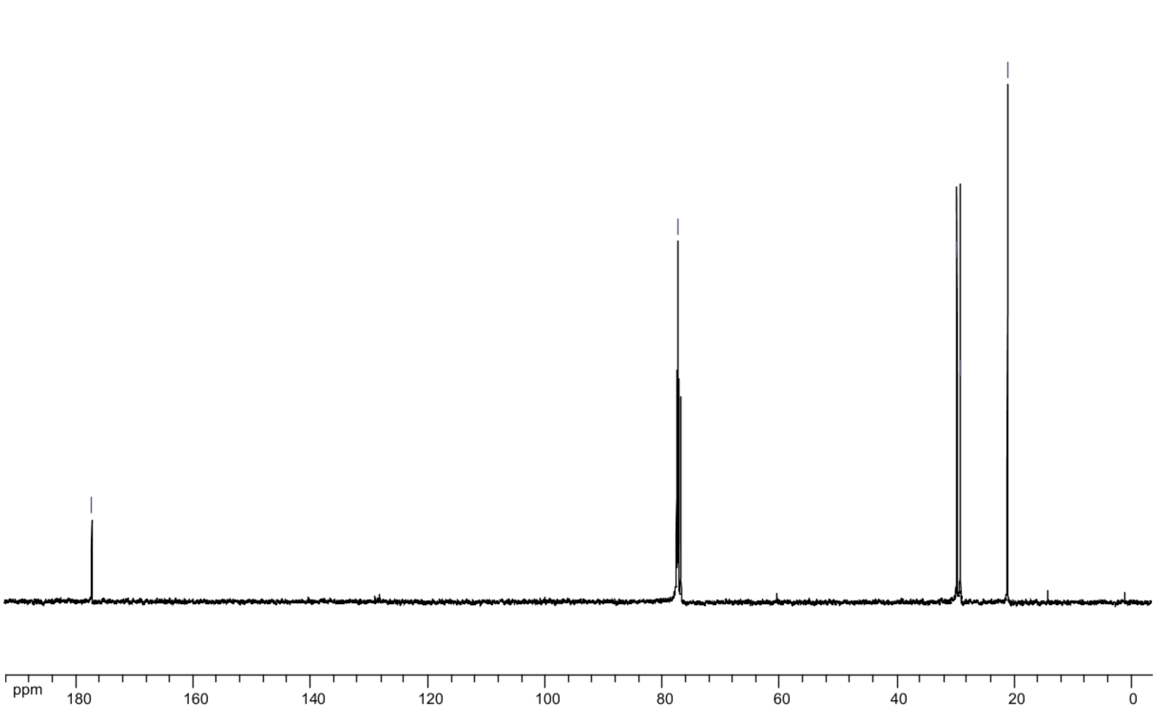
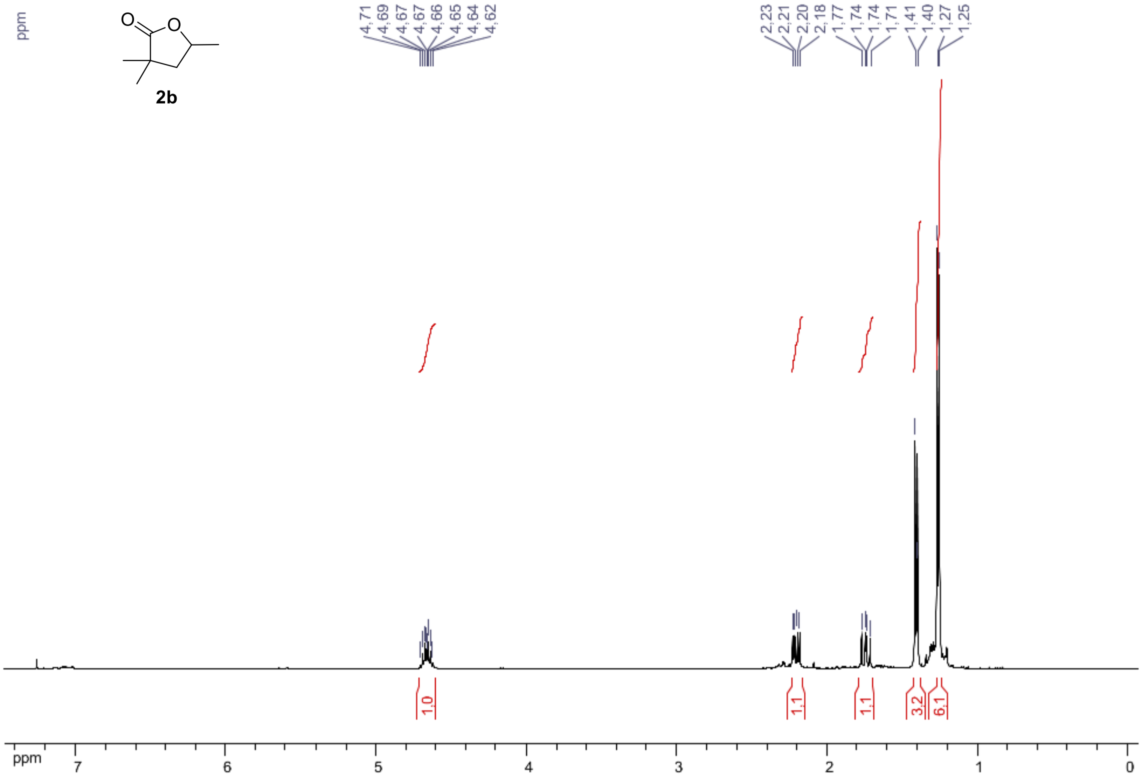
4,62
4,62
4,61
4,60
4,59
4,58
4,57
4,55
2,52
2,50
2,50
2,49
2,48
2,47
2,36
2,34
2,33
2,31
2,29
2,27
1,84
1,81
1,81
1,80
1,79
1,78
1,78
1,78
1,76
1,75
1,75
1,73
1,37
1,35
0,9
1,9
1,0
1,0
3,0
ppm 7 6 5 4 3 2 1 0

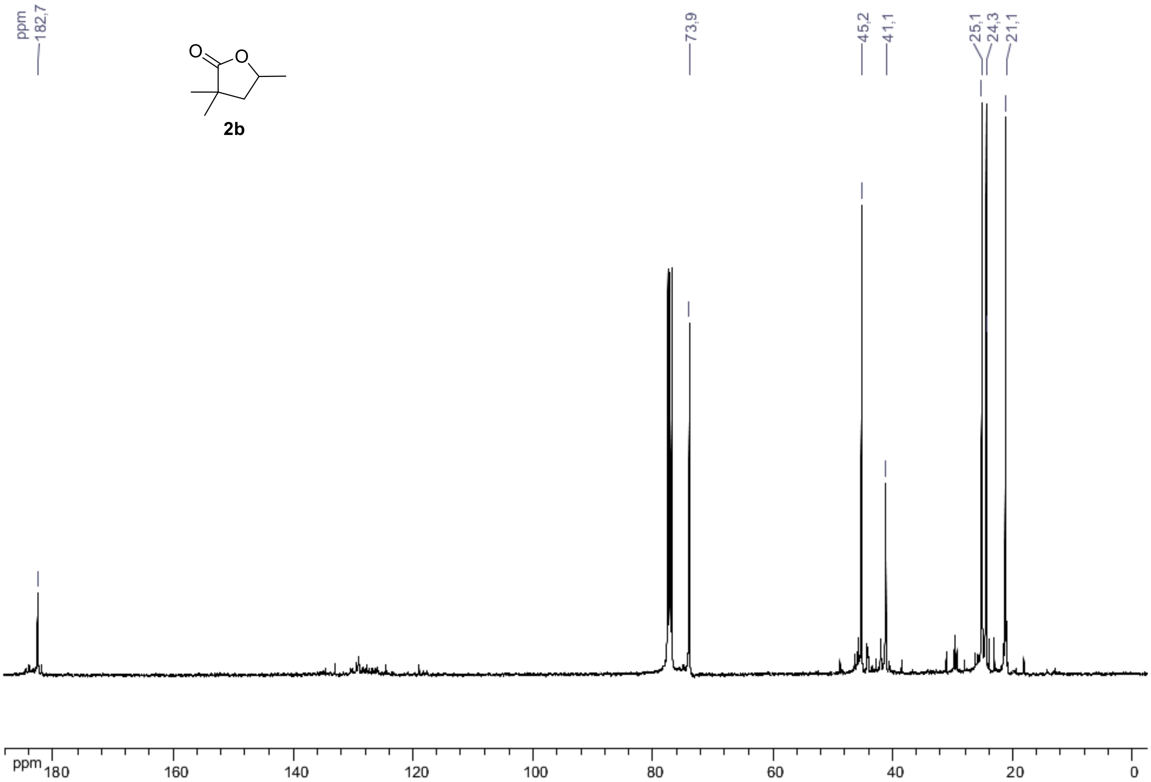


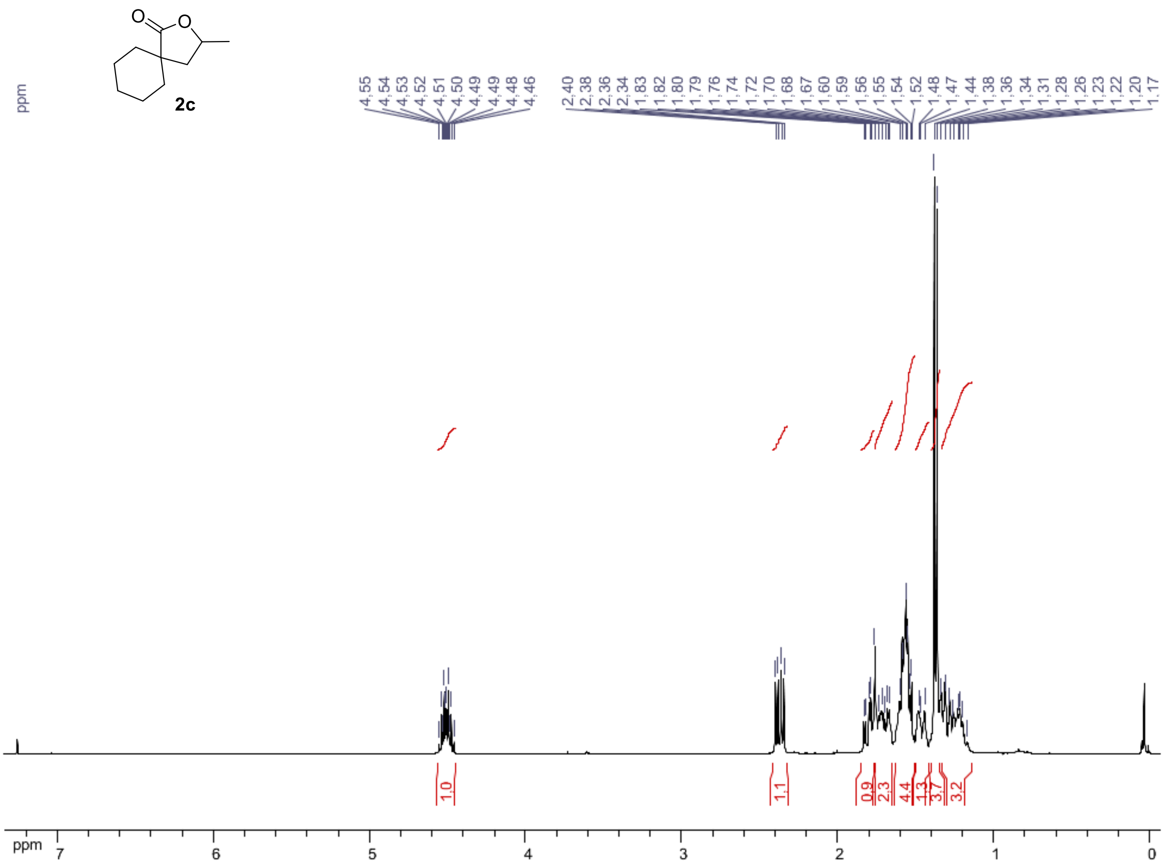
2a

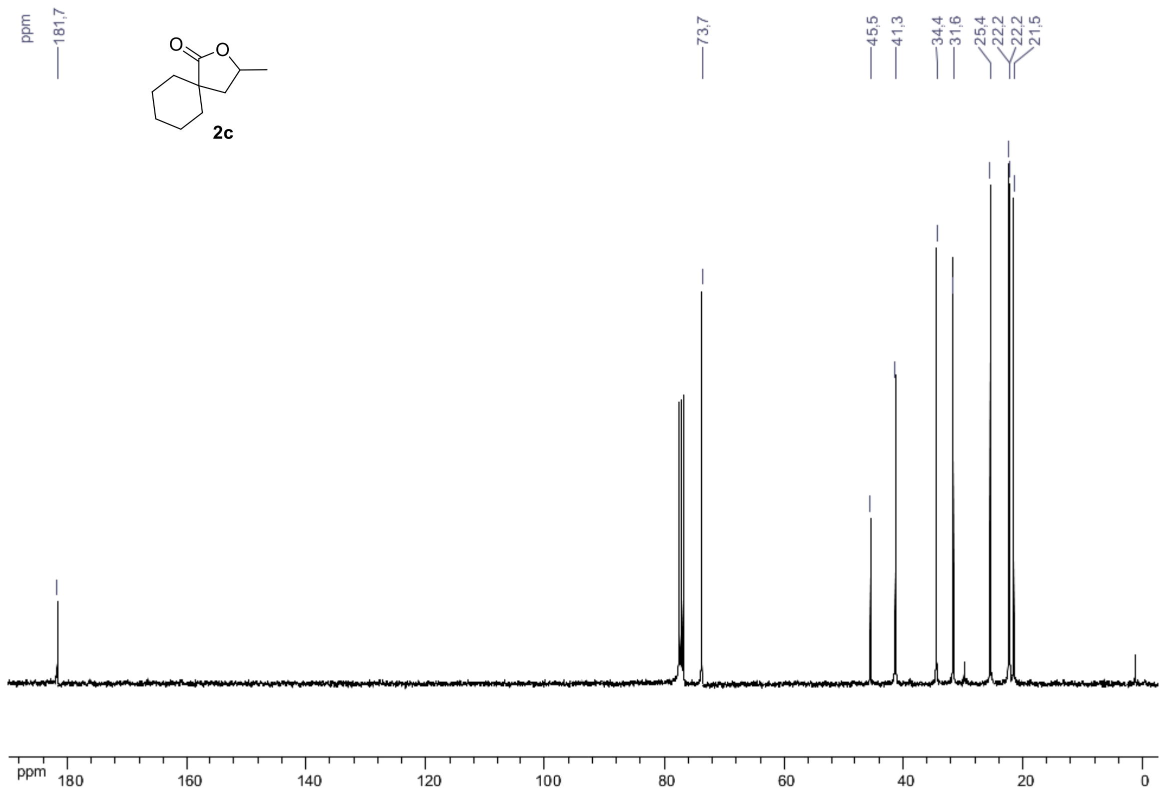


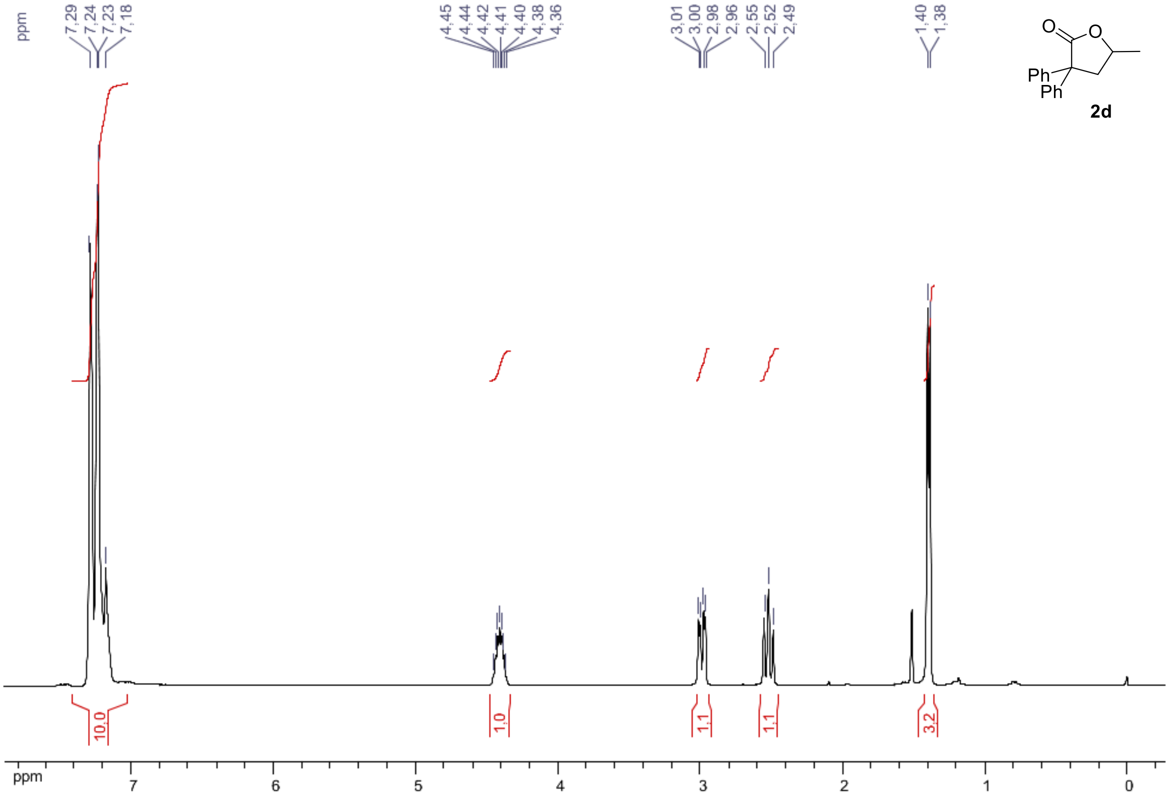
ppm
177,3
77,3
29,7
29,1
21,0
ppm 180 160 140 120 100 80 60 40 20 0


ppm
O
O
2b
4,71
4,69
4,67
4,67
4,66
4,65
4,64
4,62
2,23
2,21
2,20
2,18
1,77
1,74
1,74
1,71
1,41
1,40
1,27
1,25
1,0
1,1
1,1
3,2
6,1
ppm 7 6 5 4 3 2 1 0


ppm
182,7
O
O
2b
73,9
45,2
41,1
25,1
24,3
21,1
ppm 180 160 140 120 100 80 60 40 20 0


ppm
2c
4,55 4,54 4,53 4,52 4,51 4,50 4,49 4,49 4,48 4,46
2,40 2,38 2,36 2,34 1,83 1,82 1,80 1,79 1,76 1,74 1,72 1,70 1,68 1,67 1,60 1,59 1,56 1,55 1,54 1,52 1,48 1,47 1,44 1,38 1,36 1,34 1,31 1,28 1,26 1,23 1,22 1,20 1,17
1,0
1,1
0,9
2,3
4,4
1,3
3,7
3,2
ppm 7 6 5 4 3 2 1 0


ppm
181,7
73,7
45,5
41,3
34,4
31,6
25,4
22,2
22,2
21,5
2c
ppm 180 160 140 120 100 80 60 40 20 0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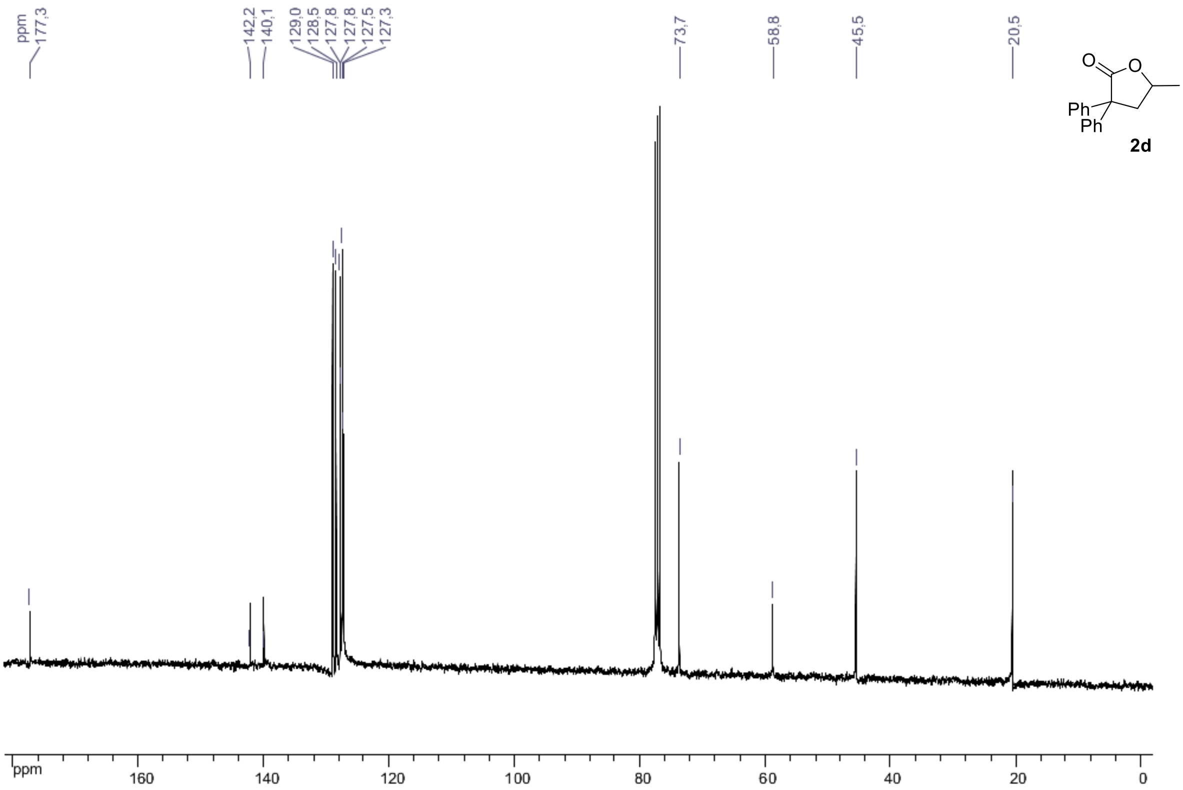
7,24
7,23
7,18
4,45
4,44
4,42
4,41
4,40
4,38
4,36
3,01
3,00
2,98
2,96
2,55
2,52
2,49
1,40
1,38
O
O
Ph
Ph
2d
10,0
1,0
1,1
1,1
3,2
ppm
7
6
5
4
3
2
1
0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177,3
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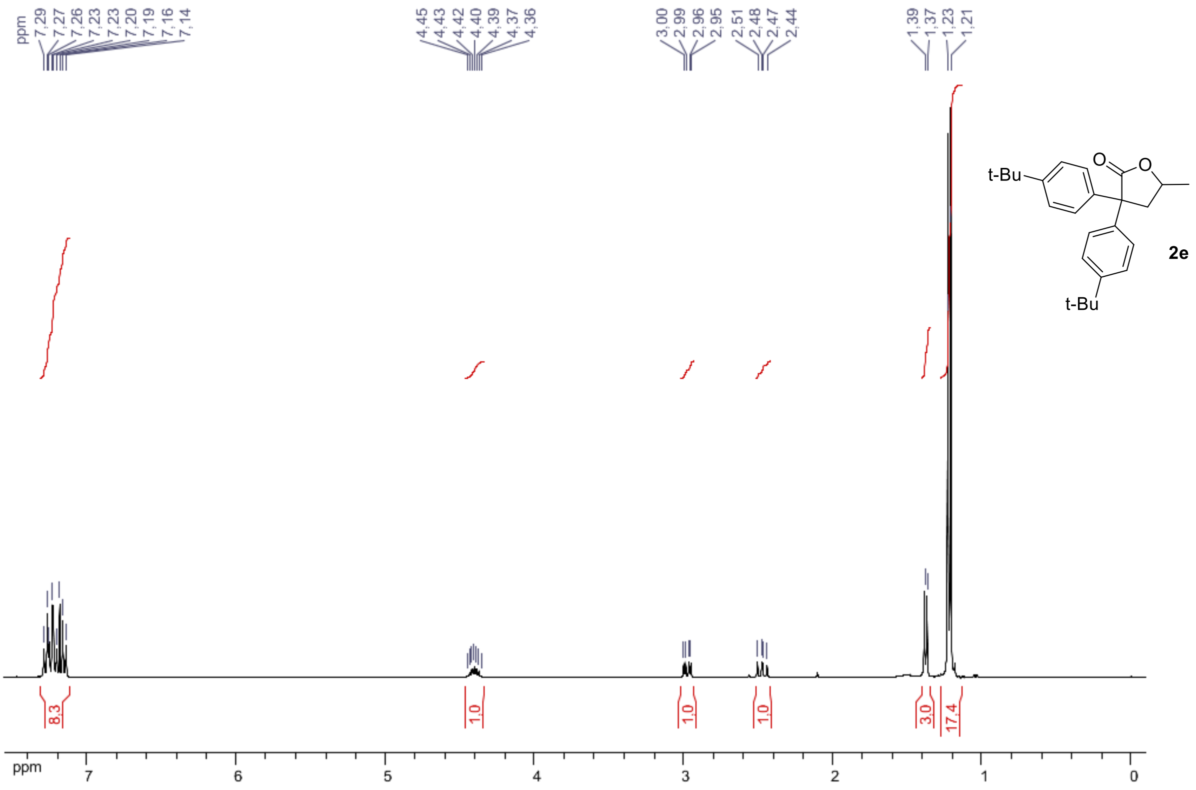
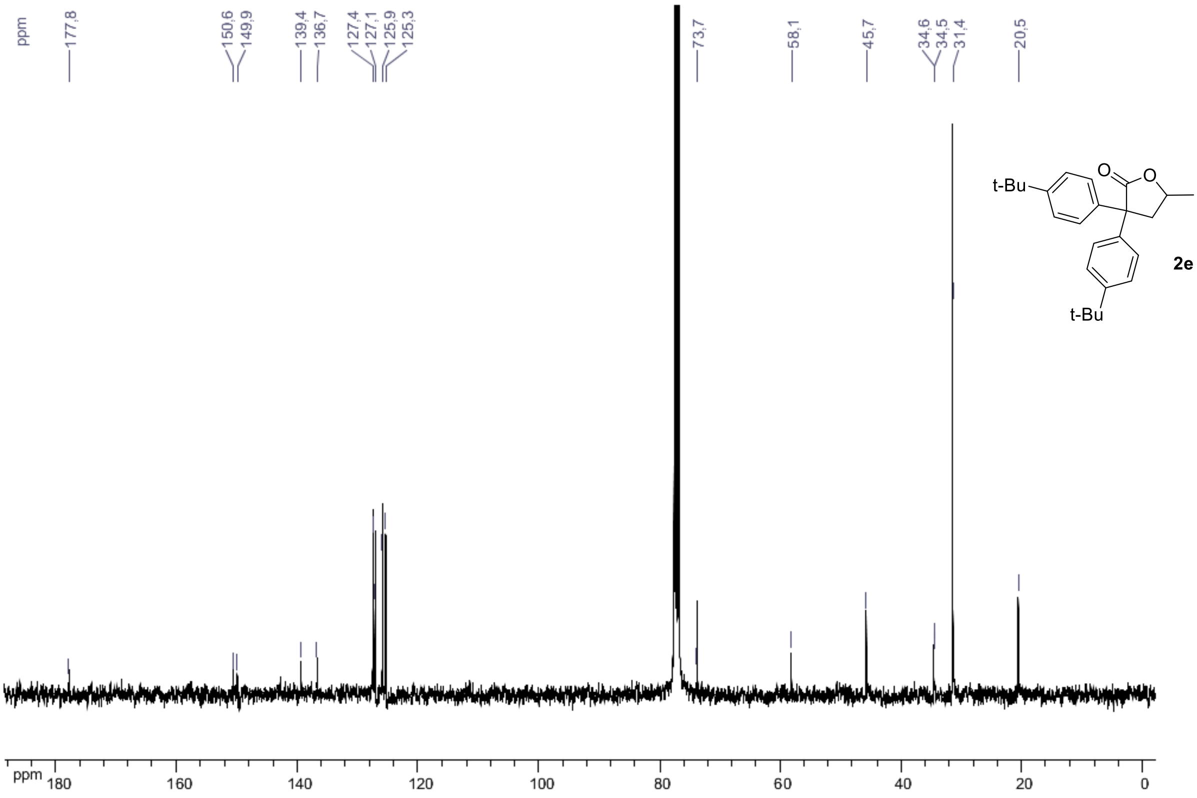
140,1
129,0
128,5
127,8
127,8
127,5
127,3
73,7
58,8
45,5
20,5
O
O
Ph
Ph
2d
ppm
160
140
120
100
80
60
40
20
0


ppm
7,29
7,27
7,26
7,23
7,23
7,20
7,19
7,16
7,14
4,45
4,43
4,42
4,40
4,39
4,37
4,36
3,00
2,99
2,96
2,95
2,51
2,48
2,47
2,44
1,39
1,37
1,23
1,21
t-Bu
O
O
t-Bu
2e
8,3
1,0
1,0
1,0
3,0
17,4
ppm
7
6
5
4
3
2
1
0


ppm
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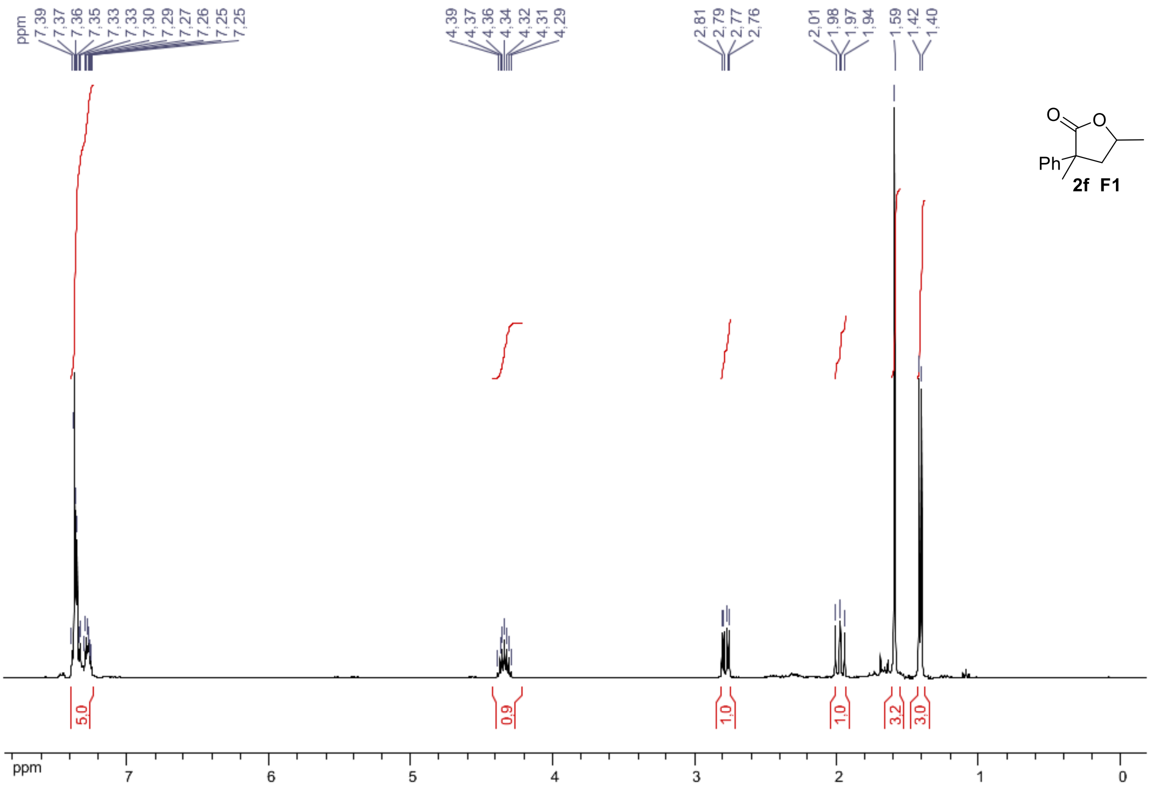
149,9
139,4
136,7
127,4
127,1
125,9
125,3
73,7
58,1
45,7
34,6
34,5
31,4
20,5
t-Bu
O
O
t-Bu
2e
ppm
180
160
140
120
100
80
60
40
20
0


ppm
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7,36
7,35
7,33
7,33
7,30
7,29
7,27
7,26
7,25
7,25
4,39
4,37
4,36
4,34
4,32
4,31
4,29
2,81
2,79
2,77
2,76
2,01
1,98
1,97
1,94
1,59
1,42
1,40
O
O
Ph
2f F1
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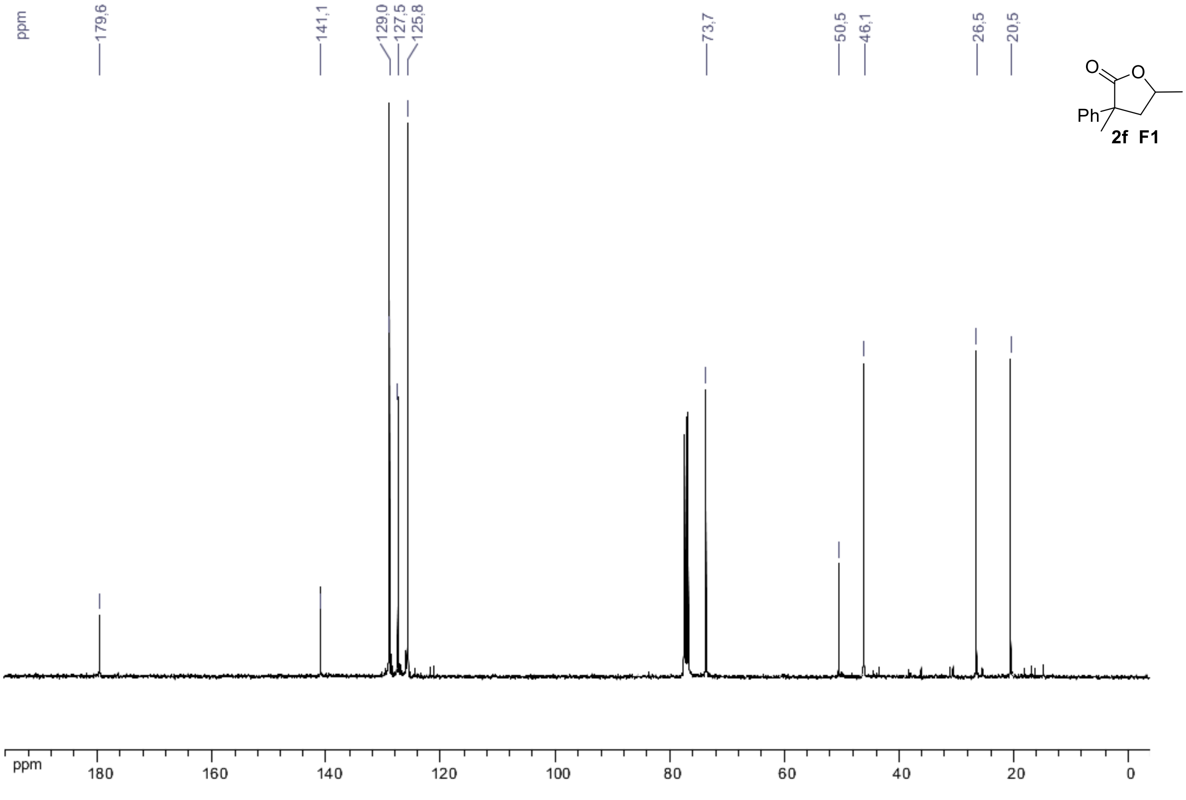
0,9
1,0
1,0
3,2
3,0
ppm
7
6
5
4
3
2
1
0


ppm
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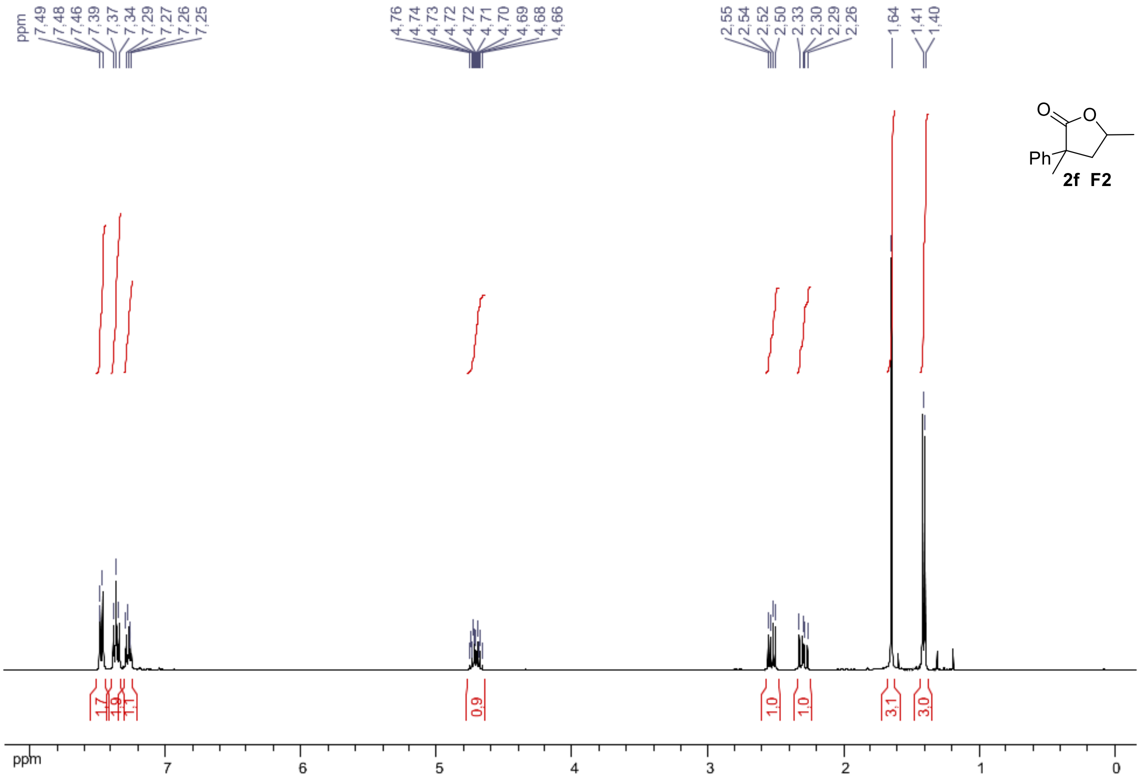
129,0
127,5
125,8
73,7
50,5
46,1
26,5
20,5
O
O
Ph
2f F1
ppm
180
160
140
120
100
80
60
40
20
0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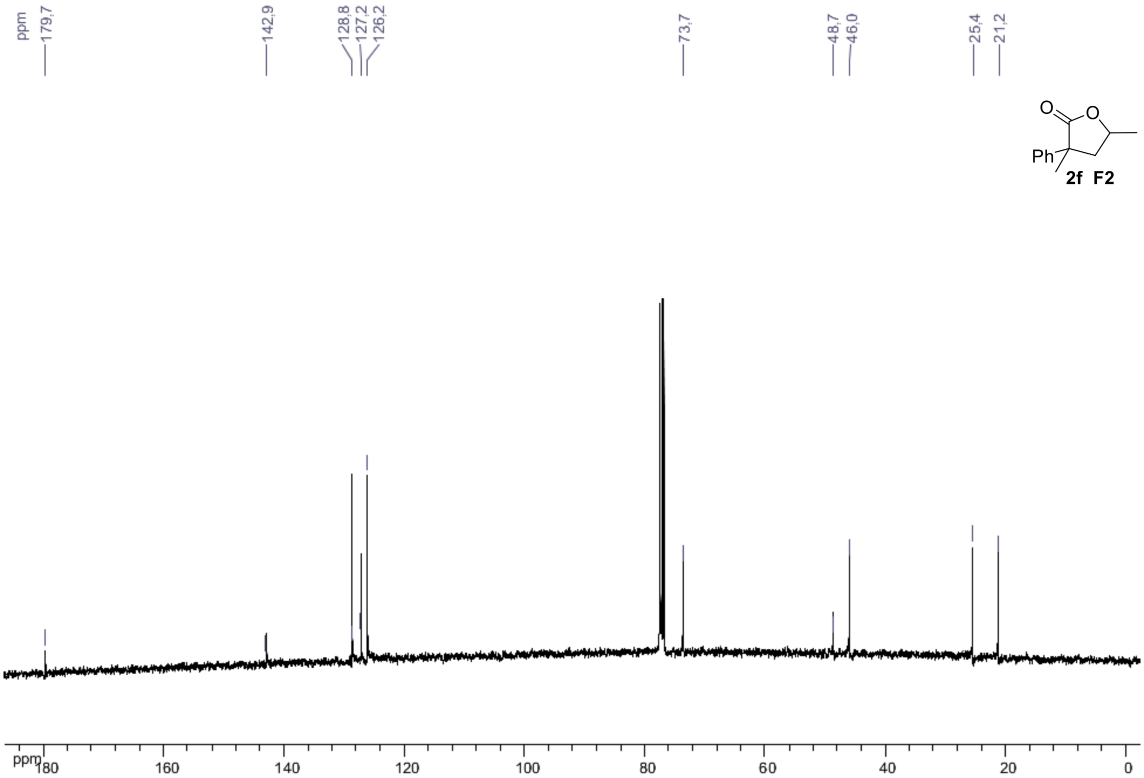
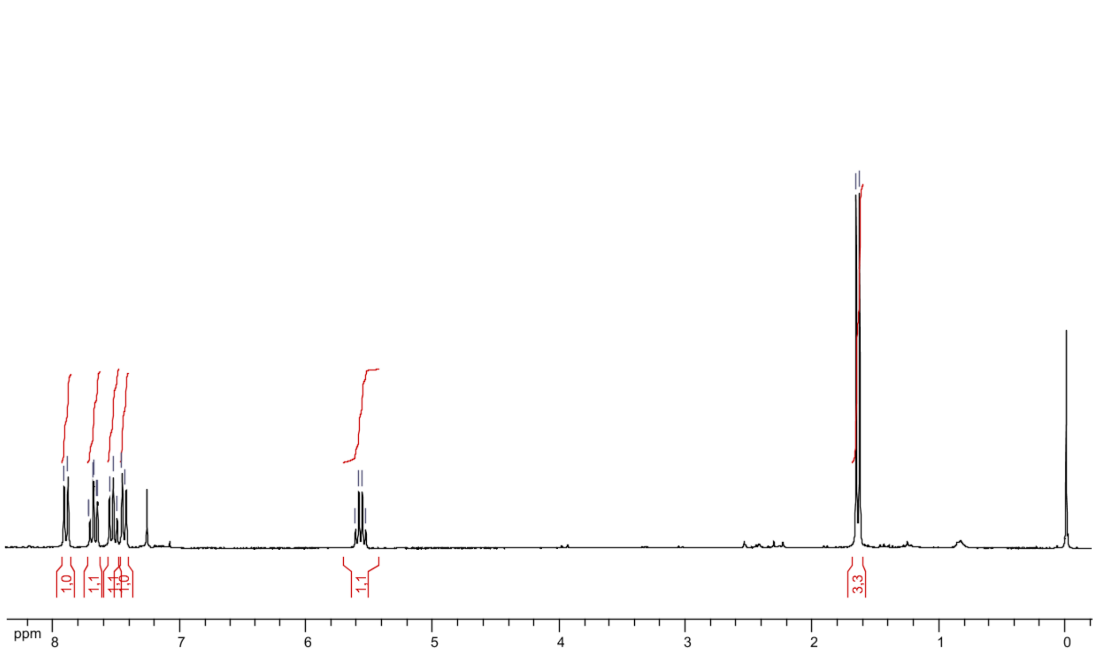
7,46
7,39
7,37
7,34
7,29
7,27
7,26
7,25
4,76
4,74
4,73
4,72
4,72
4,71
4,70
4,69
4,68
4,66
2,55
2,54
2,52
2,50
2,33
2,30
2,29
2,26
1,64
1,41
1,40
Ph
O
O
2f F2
1,7
1,9
1,1
0,9
1,0
1,0
3,1
3,0
ppm
7
6
5
4
3
2
1
0


ppm
179,7
142,9
128,8
127,2
126,2
73,7
48,7
46,0
25,4
21,2
Ph
O
O
2f F2
ppm
180
160
140
120
100
80
60
40
20
0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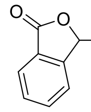
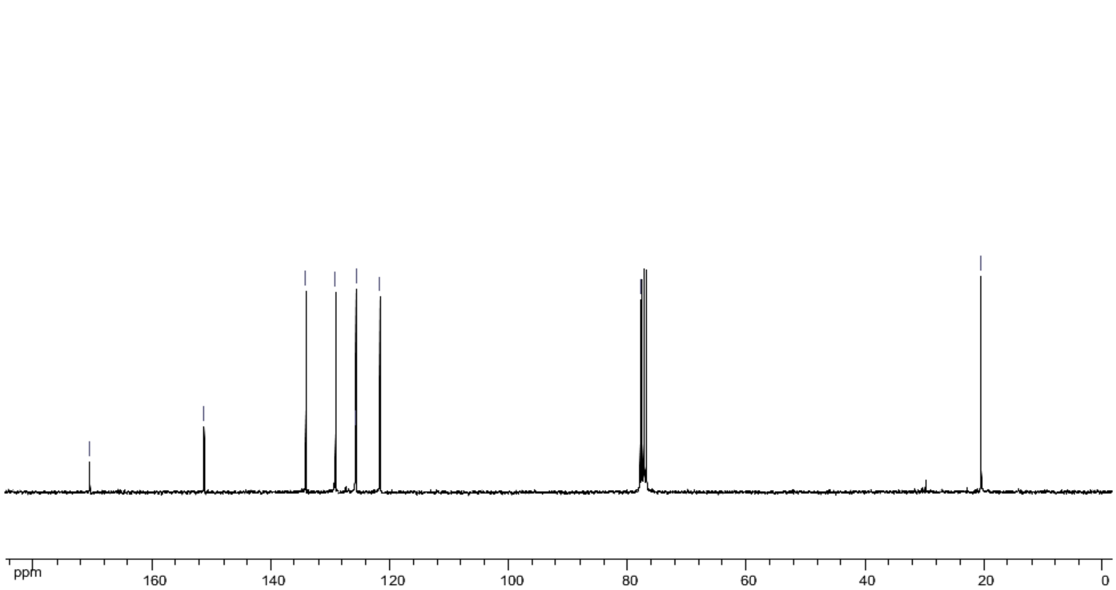
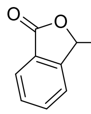
7,71
7,71
7,68
7,68
7,65
7,65
7,56
7,53
7,50
7,45
7,42
5,61
5,58
5,56
5,53
1,65
1,63
1,0
1,1
1,1
1,0
1,1
3,3
ppm
8
7
6
5
4
3
2
1
0

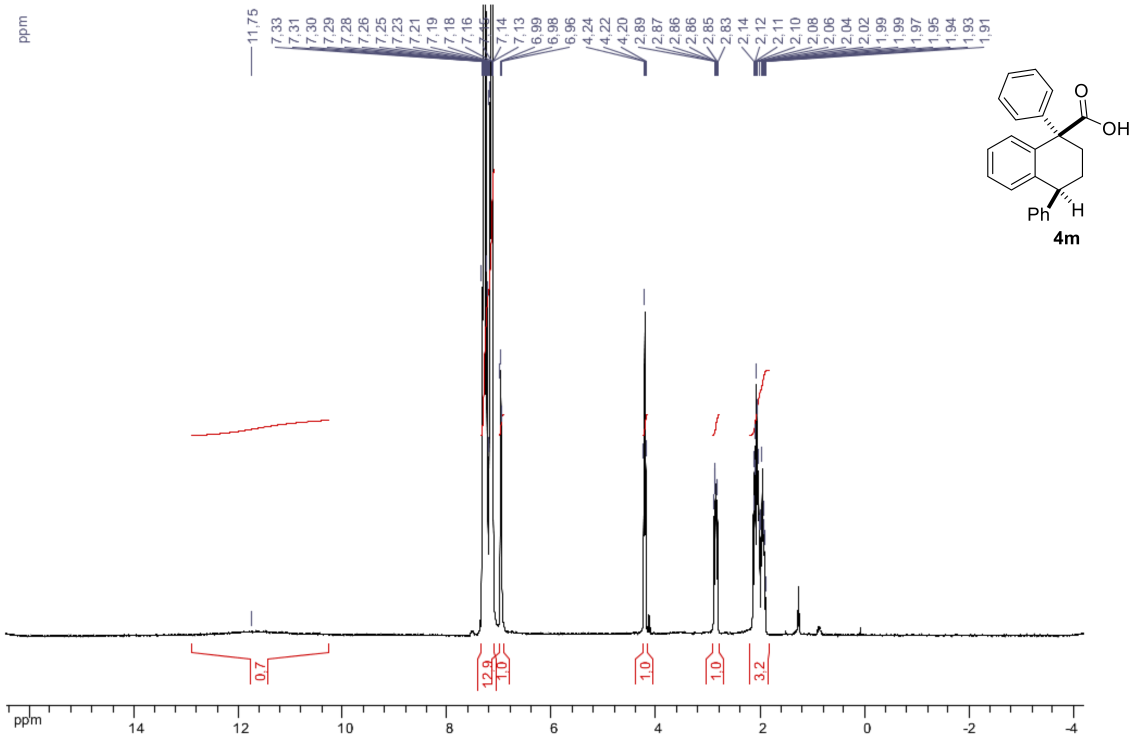


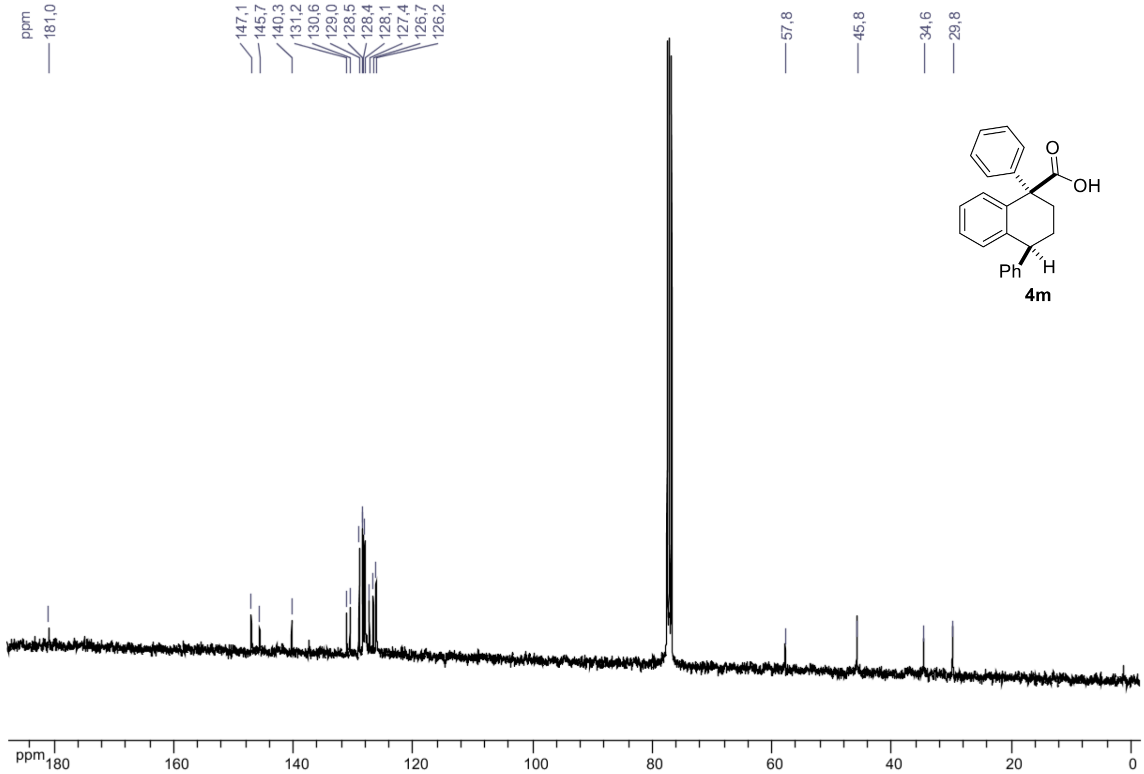
2g

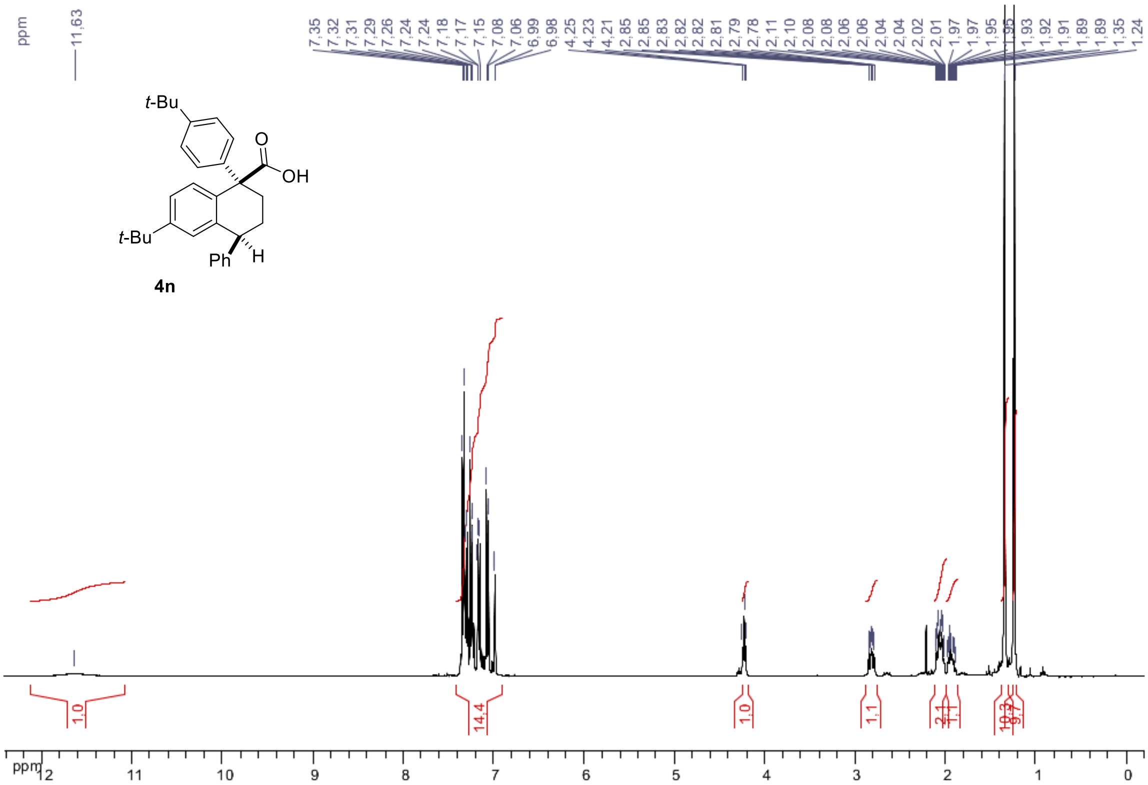

ppm
170,5
151,3
134,1
129,1
125,8
125,7
121,7
77,8
20,5
ppm
160
140
120
100
80
60
40
20
0

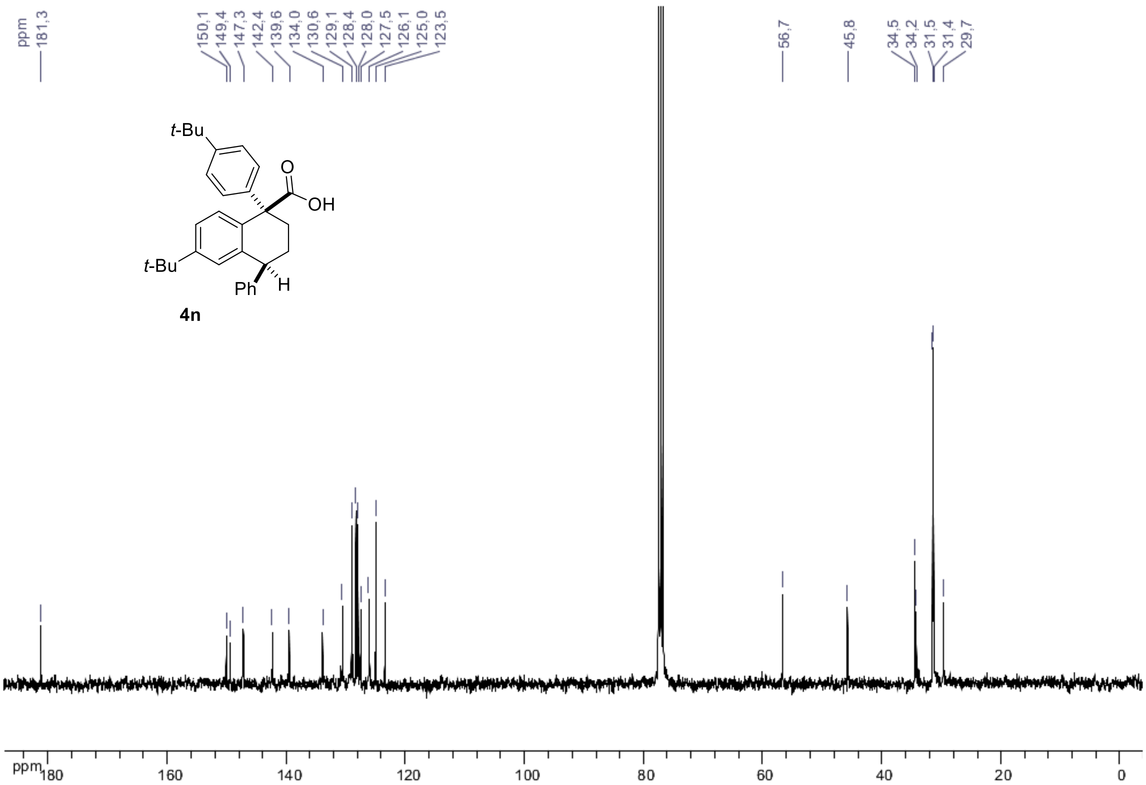


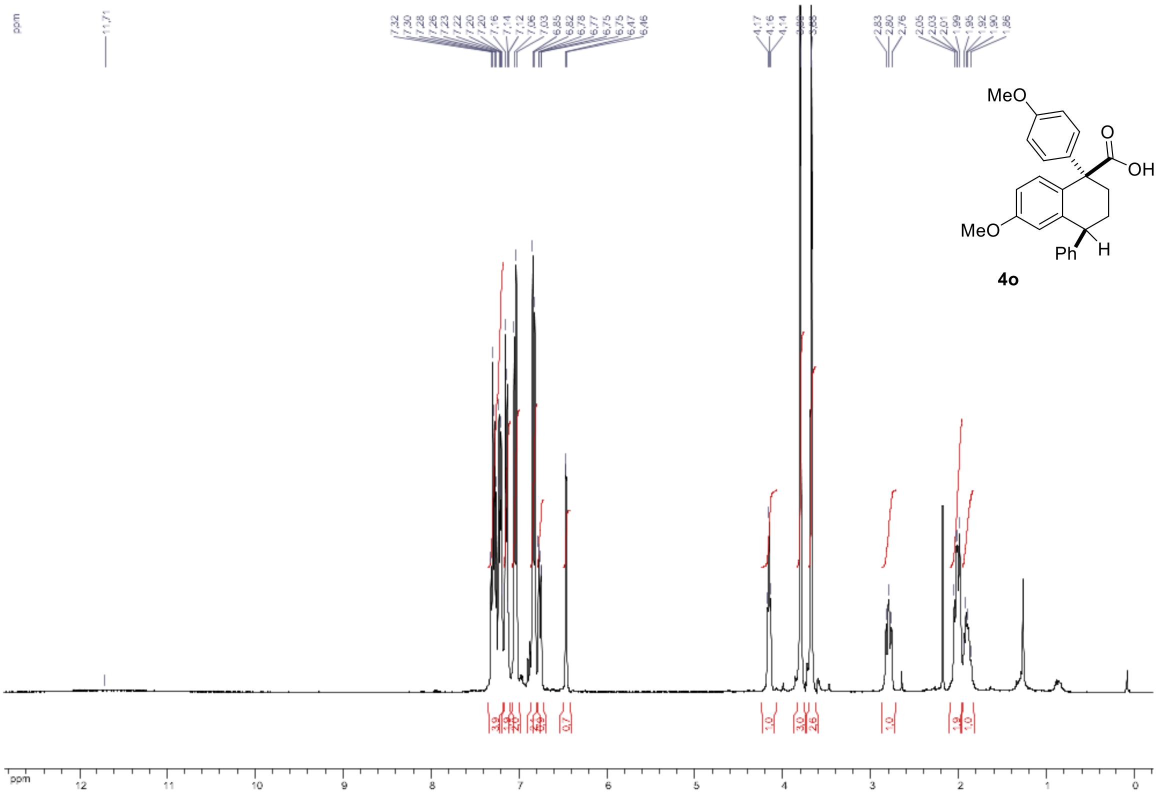
2g

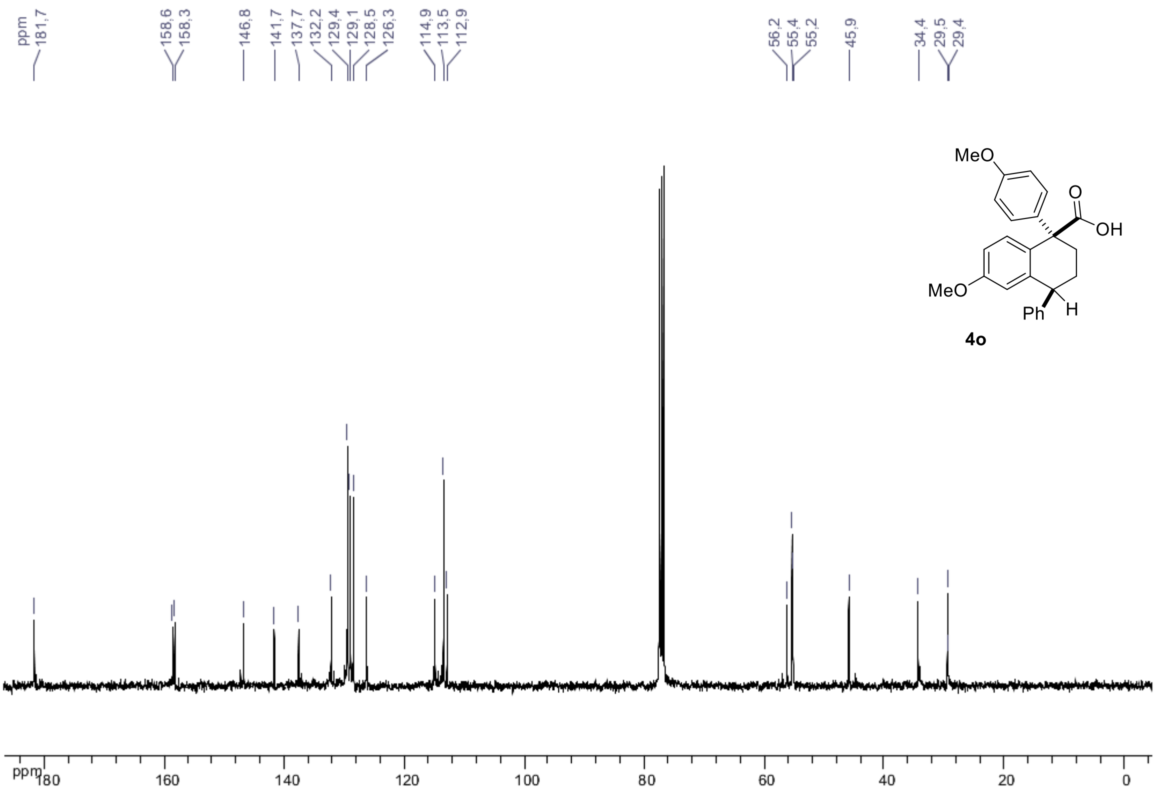

ppm
11,75
7,33 7,31 7,30 7,29 7,28 7,26 7,25 7,23 7,21 7,19 7,18 7,16 7,14 7,13 6,99 6,98 6,96 4,24 4,22 4,20 2,89 2,87 2,86 2,86 2,85 2,83 2,14 2,12 2,11 2,10 2,08 2,06 2,04 2,02 1,99 1,99 1,97 1,95 1,94 1,93 1,91
O
OH
Ph
H
4m
0,7
12,9
1,0
1,0
1,0
3,2
ppm
14
12
10
8
6
4
2
0
-2
-4


ppm
181,0
147,1 145,7 140,3 131,2 130,6 129,0 128,5 128,4 128,1 127,4 126,7 126,2
57,8
45,8
34,6
29,8
O
OH
Ph
H
4m
ppm
180
160
140
120
100
80
60
40
20
0


ppm
11,63
7,35 7,32 7,31 7,29 7,26 7,24 7,24 7,18 7,17 7,15 7,08 7,06 6,99 6,98
4,25 4,23 4,21
2,85 2,85 2,83 2,82 2,82 2,81 2,79 2,78
2,11 2,10 2,08 2,08 2,08 2,06 2,06 2,04 2,04 2,02 2,01
1,97 1,97 1,95 1,93 1,92 1,91 1,89 1,89 1,35 1,24
t-Bu
O
OH
t-Bu
Ph
H
4n
1,0
14,4
1,0
1,1
2,1
1,1
10,3
9,7
ppm 12 11 10 9 8 7 6 5 4 3 2 1 0


ppm
181,3
150,1 149,4 147,3 142,4 139,6 134,0 130,6 129,1 128,4 128,0 127,5 126,1 125,0 123,5
56,7
45,8
34,5 34,2 31,5 31,4 29,7
t-Bu
O
OH
t-Bu
Ph
H
4n
ppm 180 160 140 120 100 80 60 40 20 0


MeO
O
OH
MeO
Ph
H
4o
ppm


ppm
181,7
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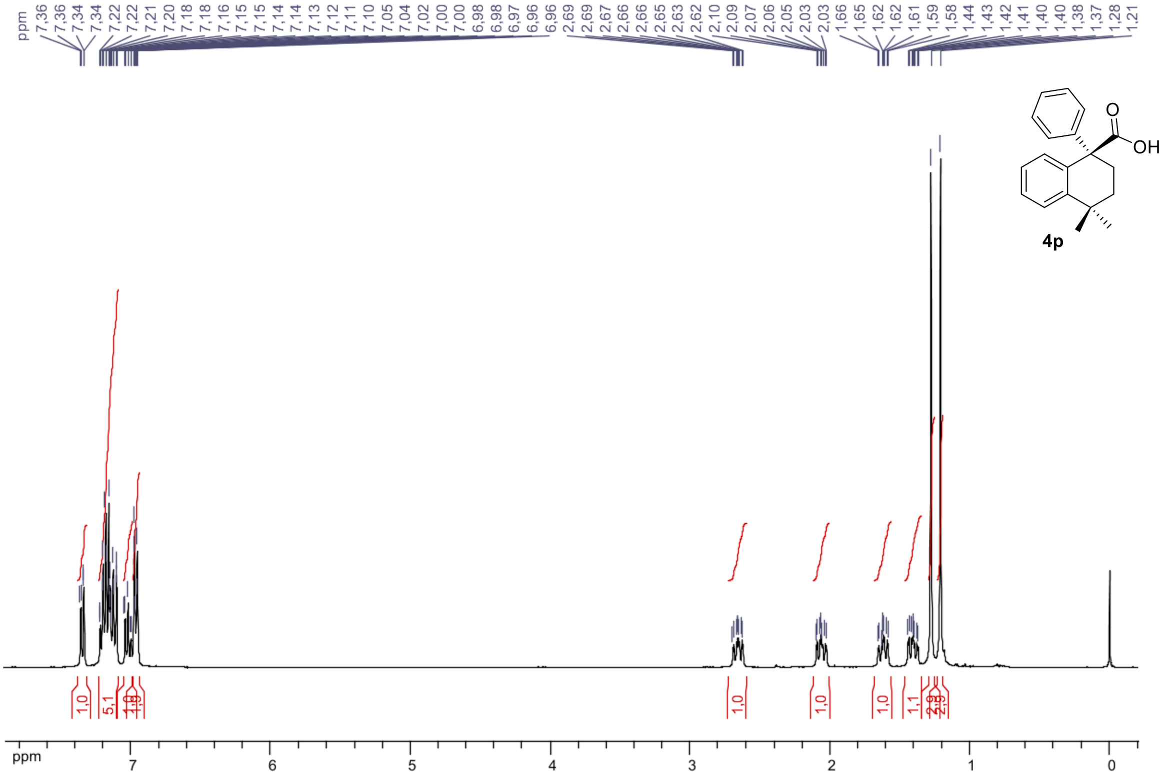
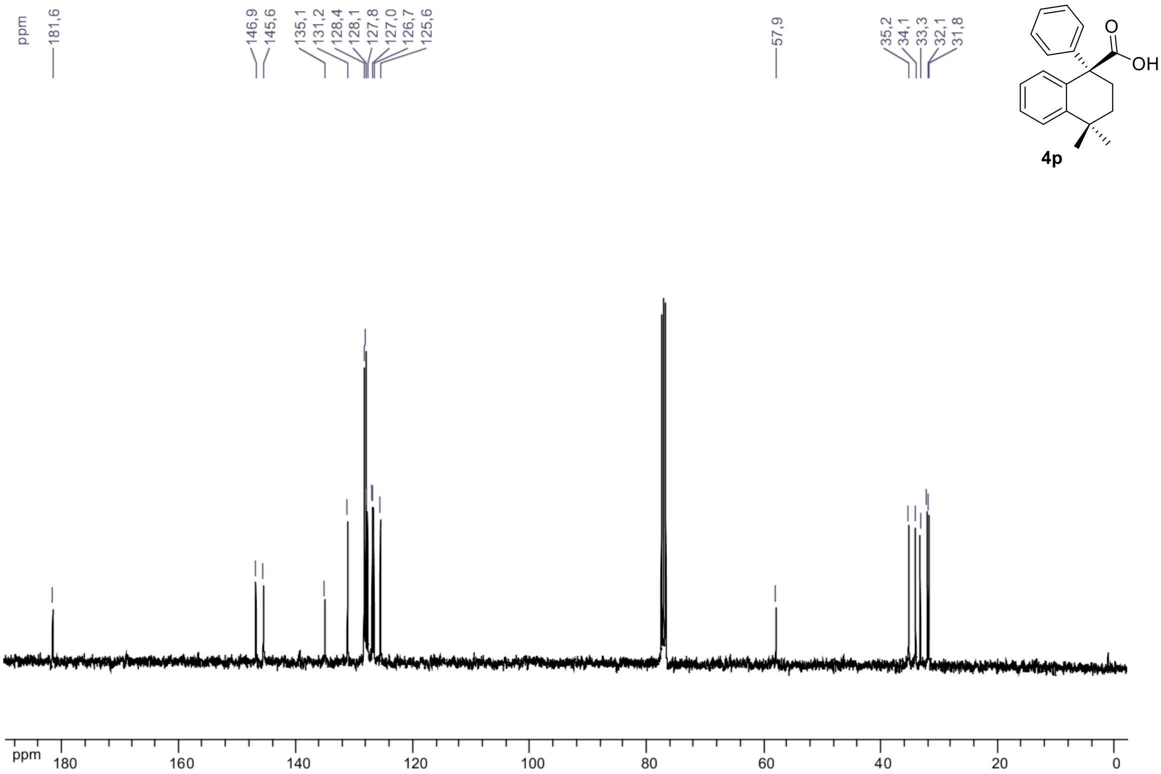
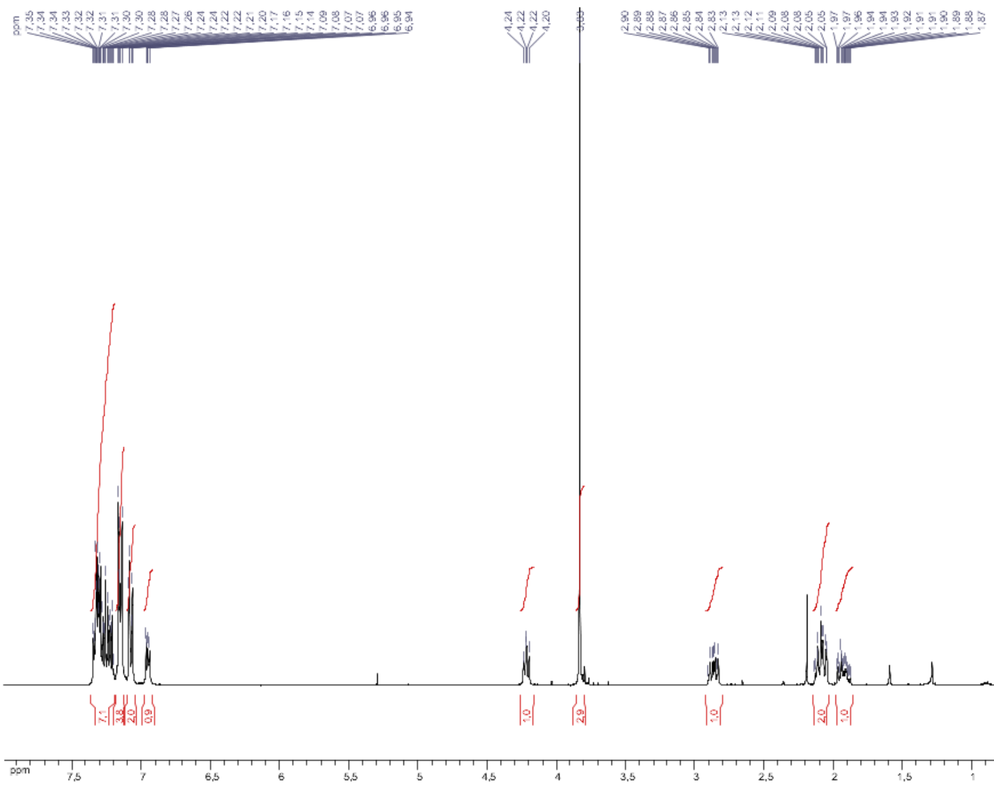
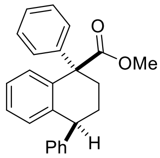
158,3
146,8
141,7
137,7
132,2
129,4
129,1
128,5
126,3
114,9
113,5
112,9
56,2
55,4
55,2
45,9
34,4
29,5
29,4
MeO
O
OH
MeO
Ph
H
4o
ppm

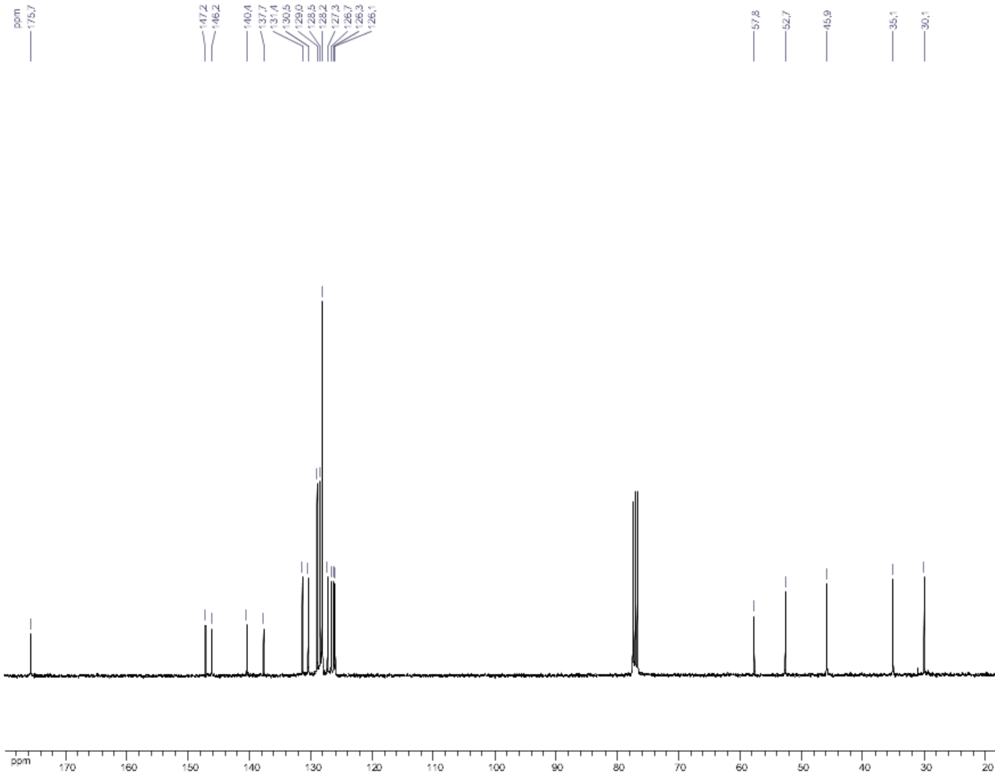

ppm
7,36 7,36 7,34 7,34 7,22 7,22 7,21 7,20 7,18 7,18 7,16 7,15 7,15 7,14 7,14 7,13 7,12 7,11 7,10 7,05 7,04 7,02 7,00 7,00 6,98 6,98 6,97 6,96 6,96 2,69 2,69 2,67 2,66 2,66 2,65 2,63 2,62 2,10 2,09 2,07 2,06 2,05 2,03 2,03 1,66 1,65 1,62 1,62 1,61 1,59 1,58 1,44 1,43 1,42 1,41 1,40 1,40 1,38 1,37 1,28 1,21
O
OH
4p
1,0
5,1
1,0
1,0
1,0
1,0
1,0
1,1
2,9
2,9
ppm 7 6 5 4 3 2 1 0


ppm
181,6
146,9 145,6
135,1 131,2 128,4 128,1 127,8 127,0 126,7 125,6
57,9
35,2 34,1 33,3 32,1 31,8
O
OH
4p
ppm 180 160 140 120 100 80 60 40 20 0




O
OMe
Ph
H

4q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146,2
140,4
137,7
131,4
130,5
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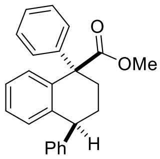
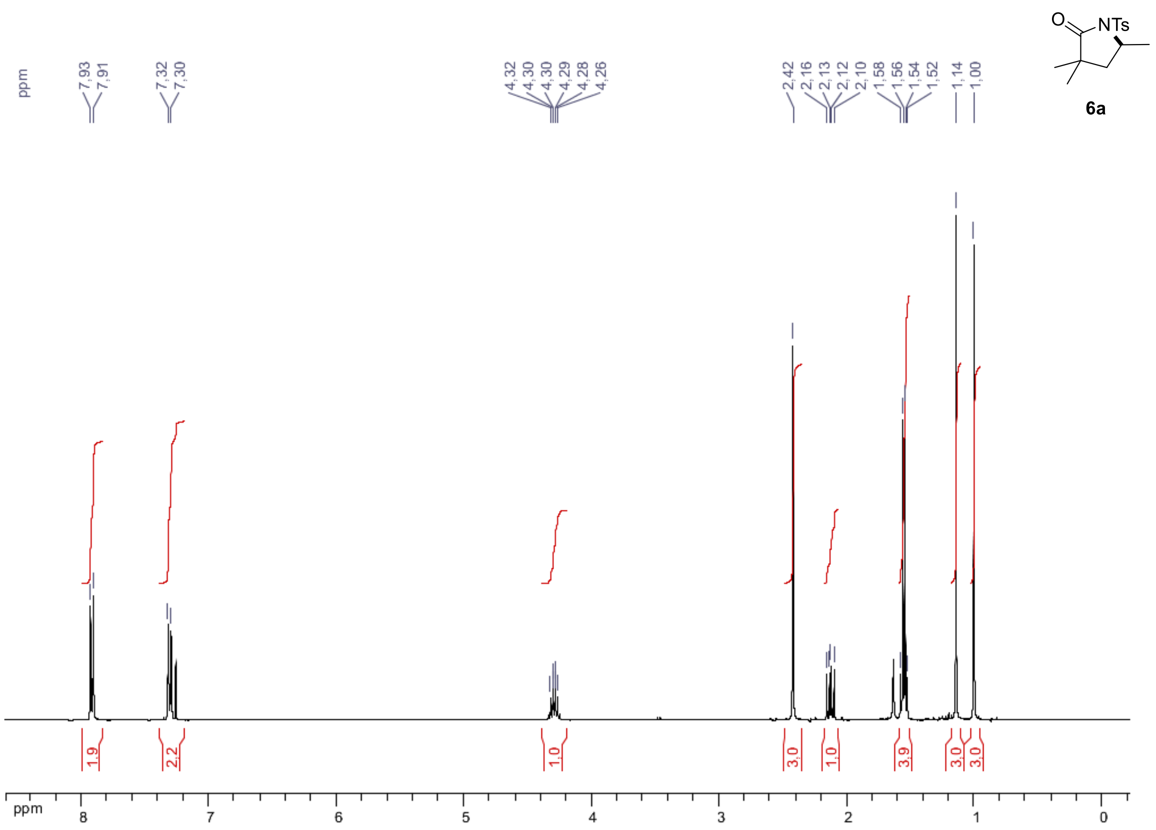
128,2
127,3
126,7
126,3
126,1
57,8
52,7
45,9
35,1
30,1


O
OMe
Ph
H

4q



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7,91
7,32
7,30
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4,30
4,30
4,29
4,28
4,26
2,42
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2,12
2,10
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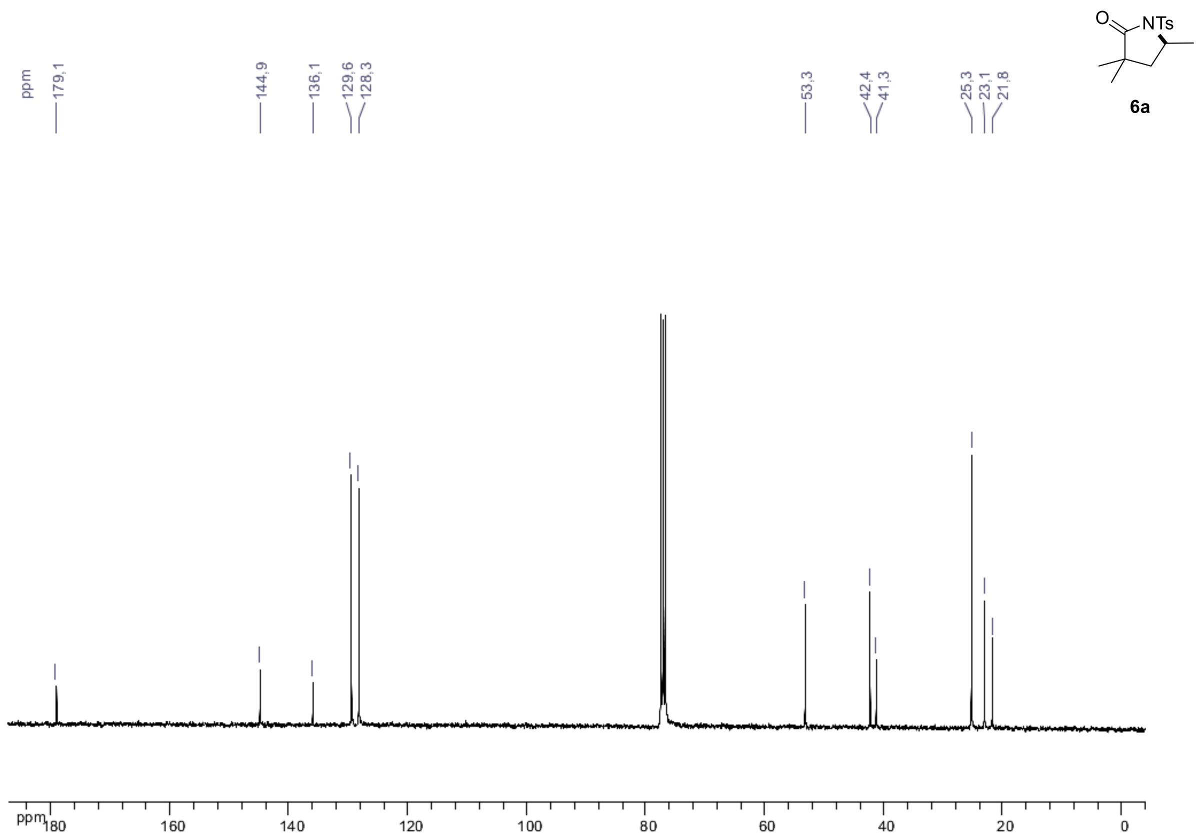
1,56
1,54
1,52
1,14
1,00
O
NTs
6a
1,9
2,2
1,0
3,0
1,0
3,9
3,0
3,0
ppm
8
7
6
5
4
3
2
1
0



ppm
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136,1
129,6
128,3
53,3
42,4
41,3
25,3
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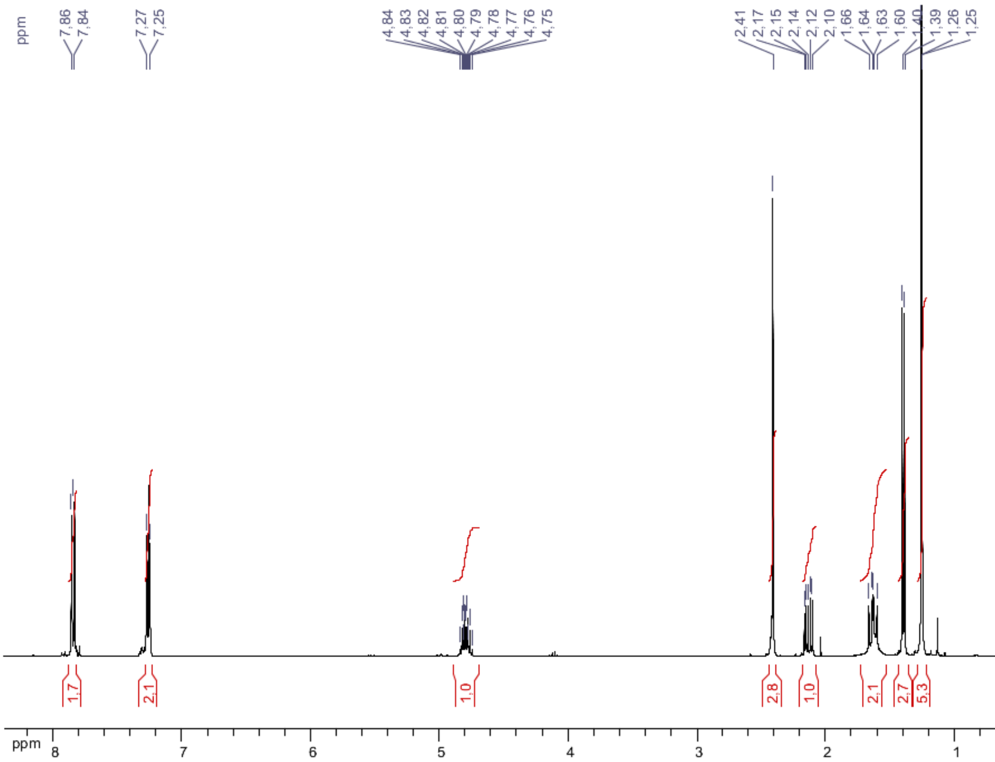
21,8
O
NTs
6a
ppm
180
160
140
120
100
80
60
40
20
0



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4,80
4,79
4,78
4,77
4,76
4,75
2,41
2,17
2,15
2,14
2,12
2,10
1,66
1,64
1,63
1,60
1,40
1,39
1,26
1,25
1,7
2,1
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2,8
1,0
2,1
2,7
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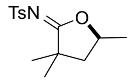
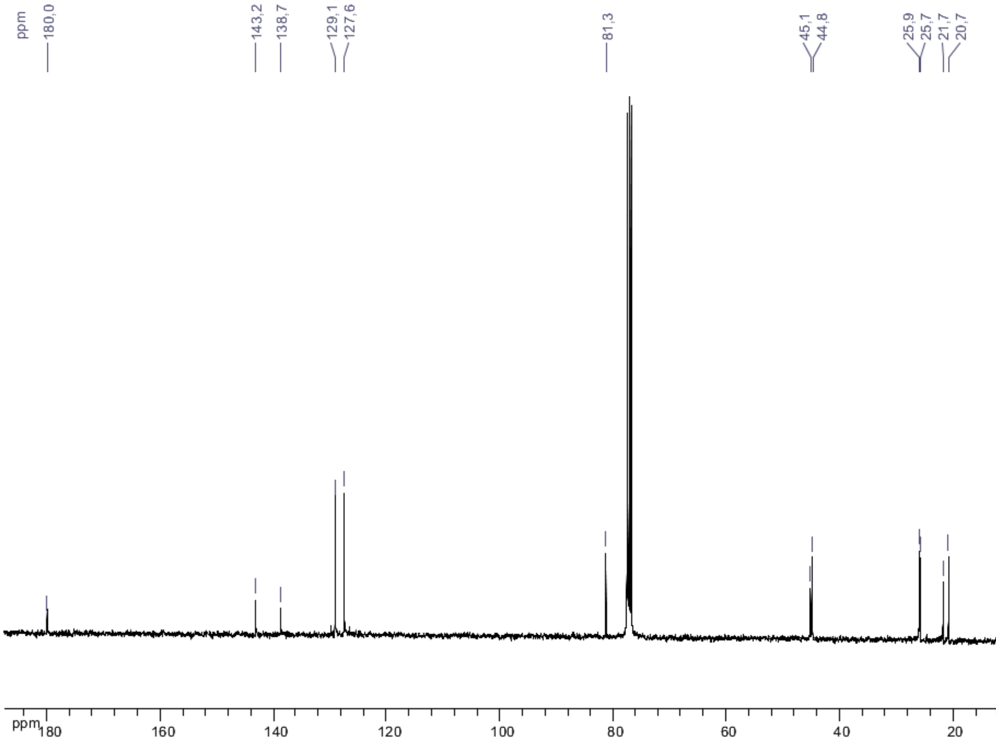
ppm
8
7
6
5
4
3
2
1
0



TsN
O

7a



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129,1
127,6
81,3
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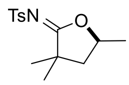
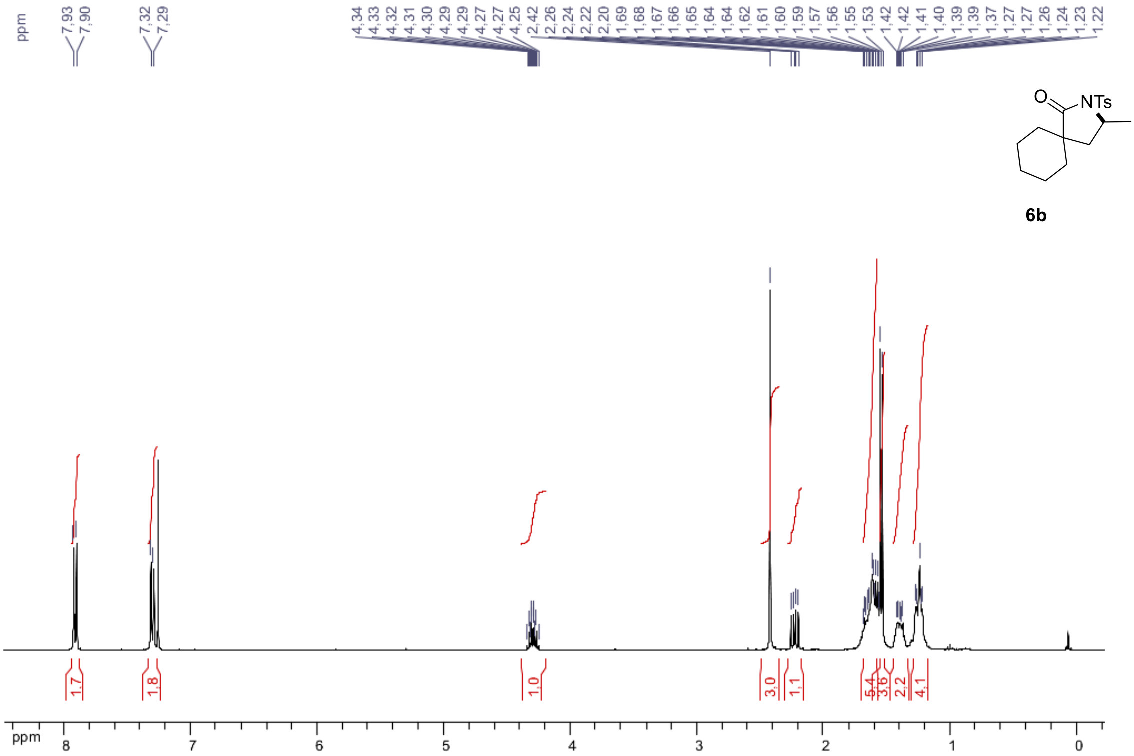
44,8
25,9
25,7
21,7
20,7
ppm
180
160
140
120
100
80
60
40
20
0



TsN
O

7a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4,33
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4,30
4,29
4,29
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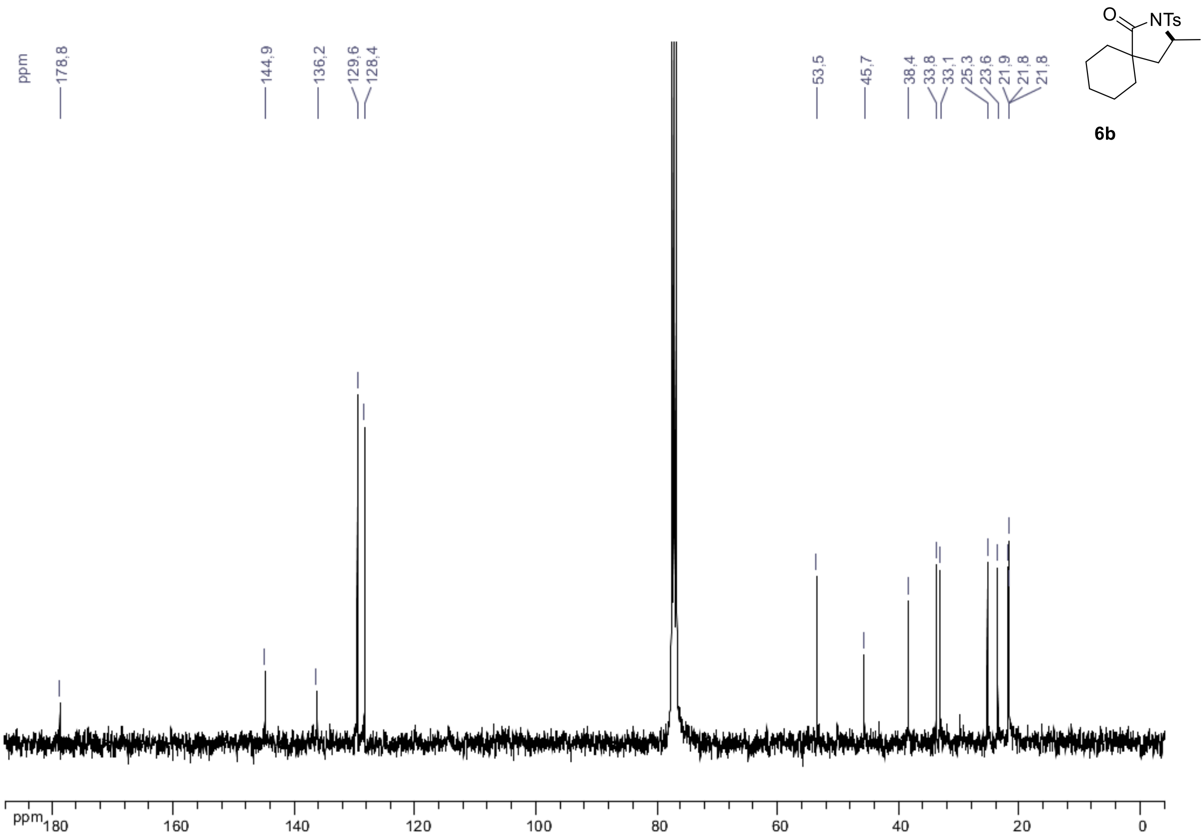
4,25
2,42
2,26
2,24
2,22
2,20
1,69
1,68
1,67
1,66
1,65
1,64
1,64
1,62
1,61
1,60
1,59
1,57
1,56
1,55
1,53
1,42
1,42
1,41
1,40
1,39
1,39
1,37
1,27
1,27
1,26
1,24
1,23
1,22
O
NTs
1,7
1,8
1,0
3,0
1,1
5,4
3,6
2,2
4,1
ppm
8
7
6
5
4
3
2
1
0

6b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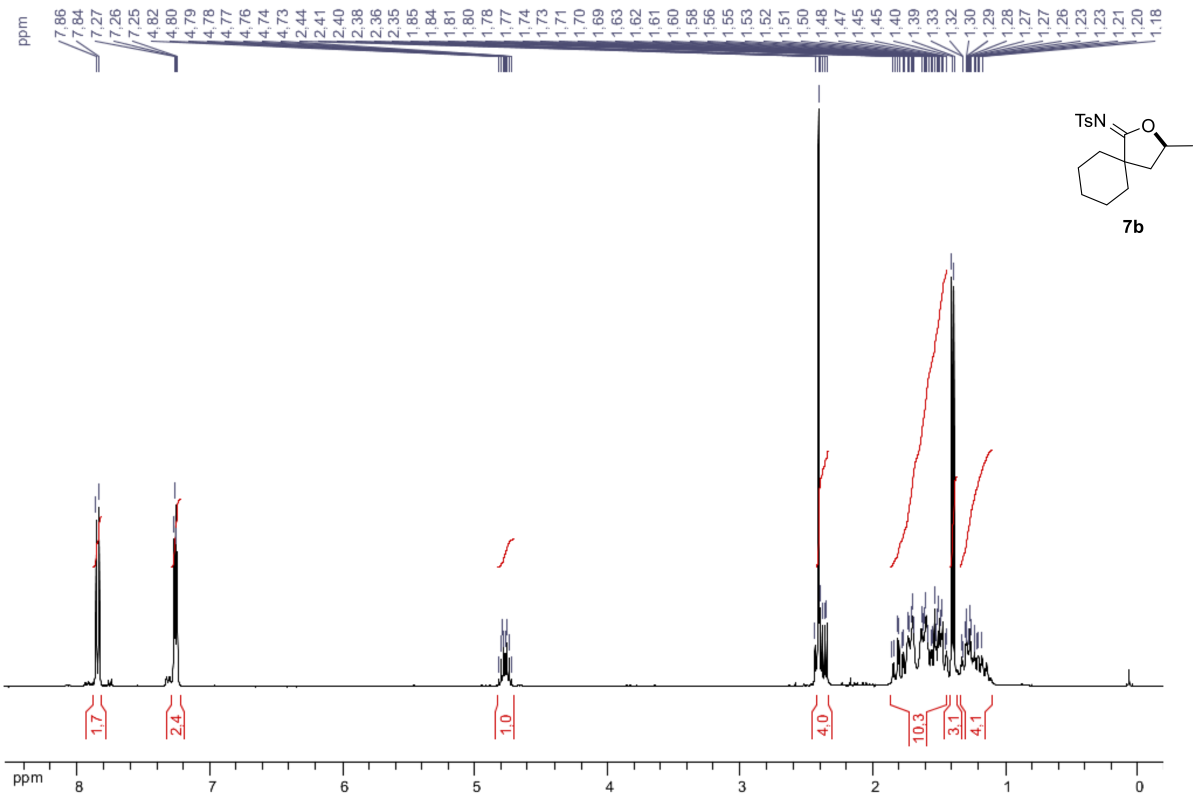
136,2
129,6
128,4
53,5
45,7
38,4
33,8
33,1
25,3
23,6
21,9
21,8
21,8
O
NTs
ppm
180
160
140
120
100
80
60
40
20
0

6b


TsN
O
7b
ppm
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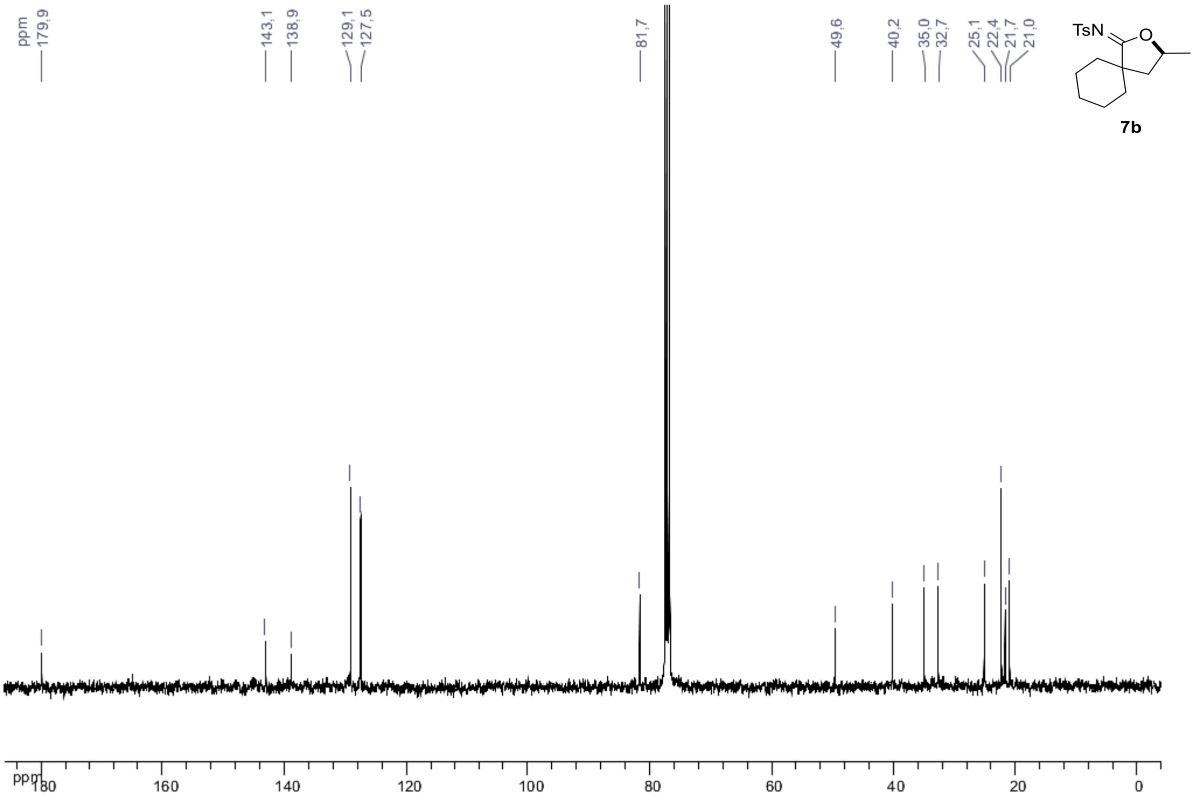
4,0
10,3
3,1
4,1
8
7
6
5
4
3
2
1
0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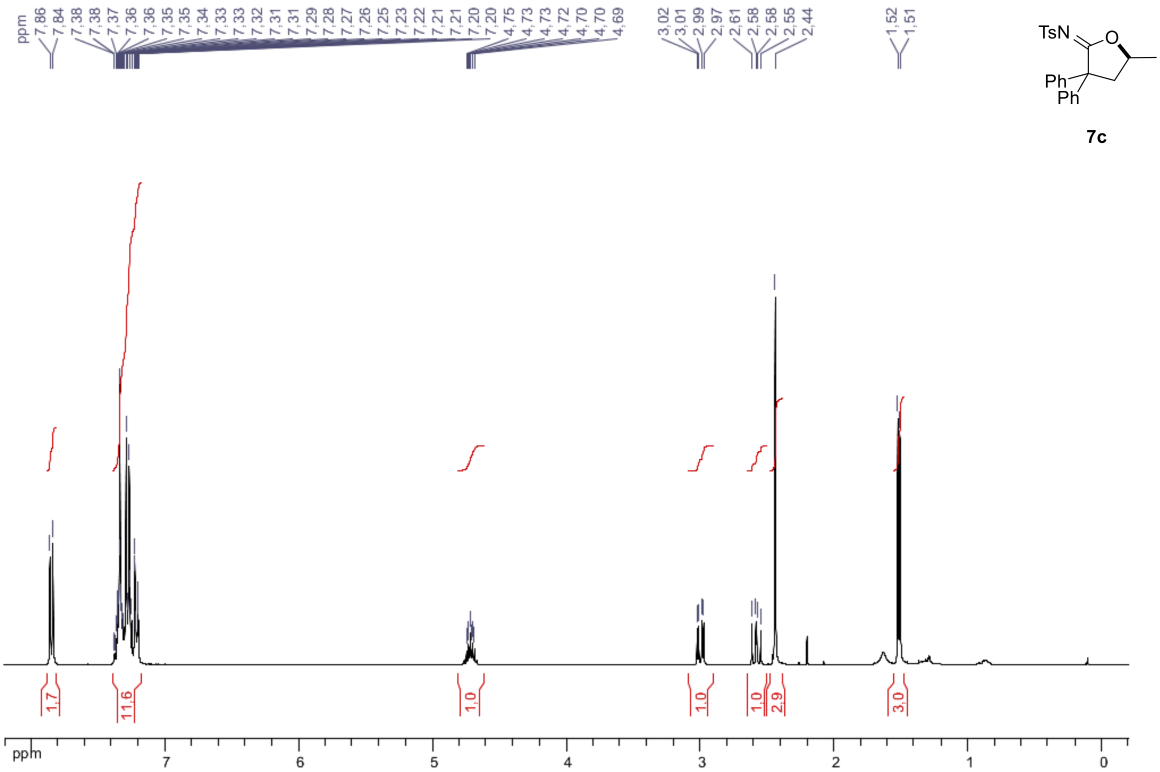
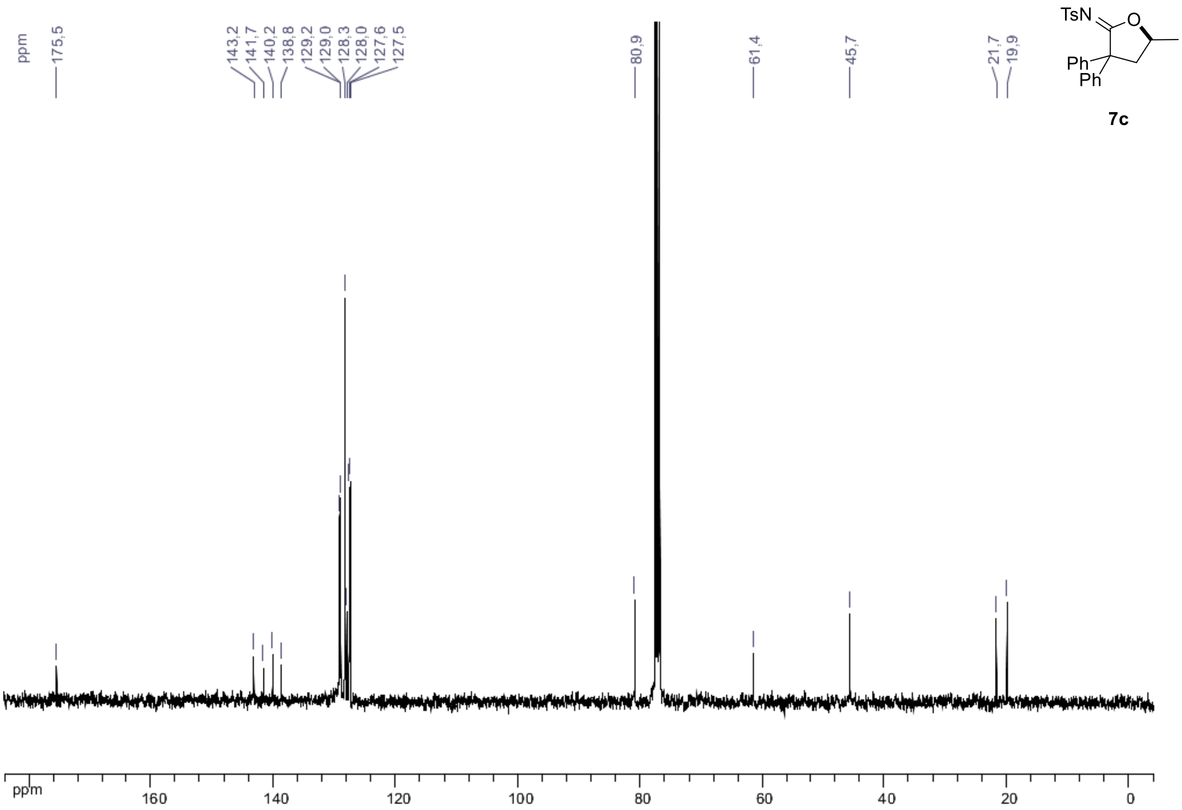
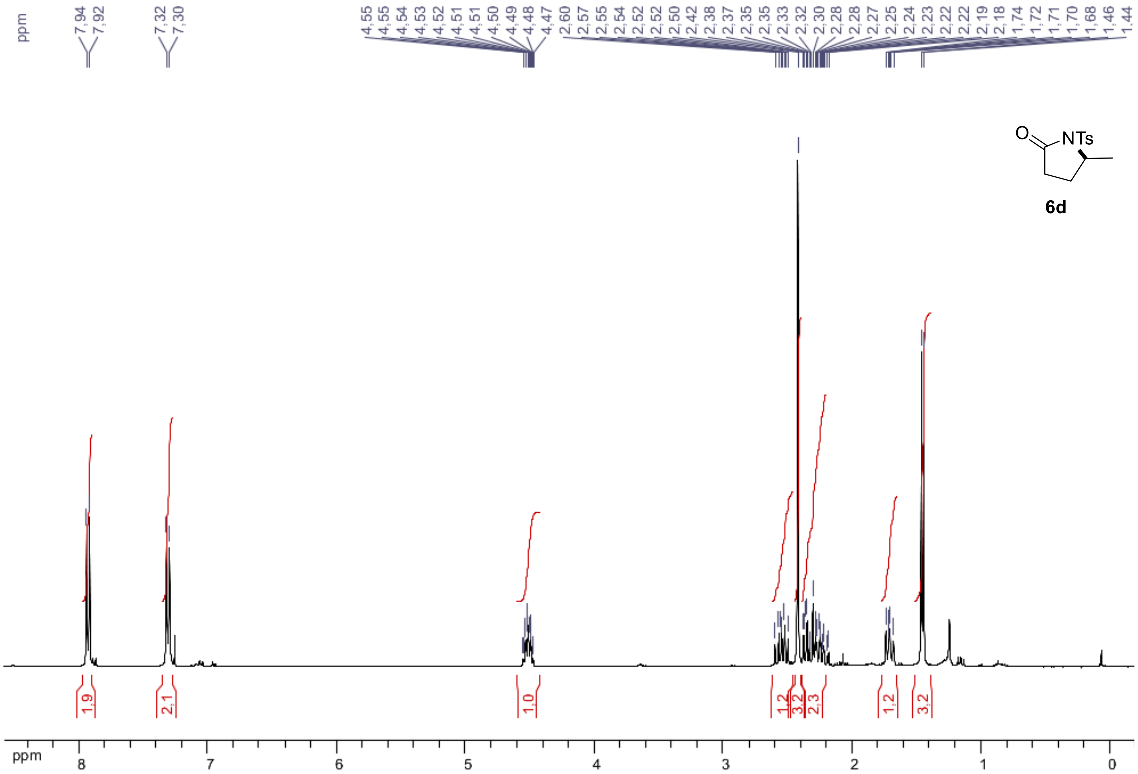
138,9
129,1
127,5
81,7
49,6
40,2
35,0
32,7
25,1
22,4
21,7
21,0
TsN
O
7b
180
160
140
120
100
80
60
40
20
0



7c



7c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4,51
4,51
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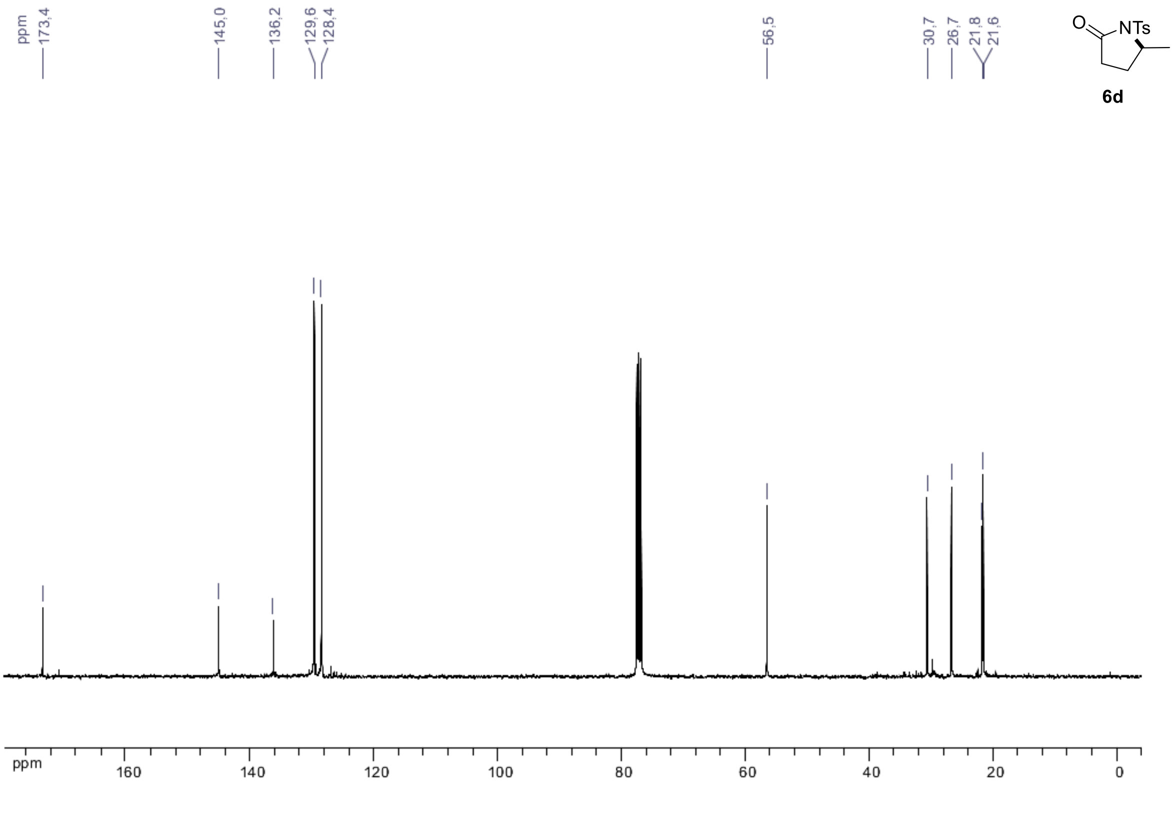
2,38
2,37
2,35
2,35
2,33
2,32
2,30
2,28
2,28
2,27
2,25
2,24
2,23
2,22
2,22
2,19
2,18
1,74
1,72
1,71
1,70
1,68
1,46
1,44
O
NTs
6d
1,9
2,1
1,0
1,2
3,2
2,3
1,2
3,2
ppm
8
7
6
5
4
3
2
1
0


ppm
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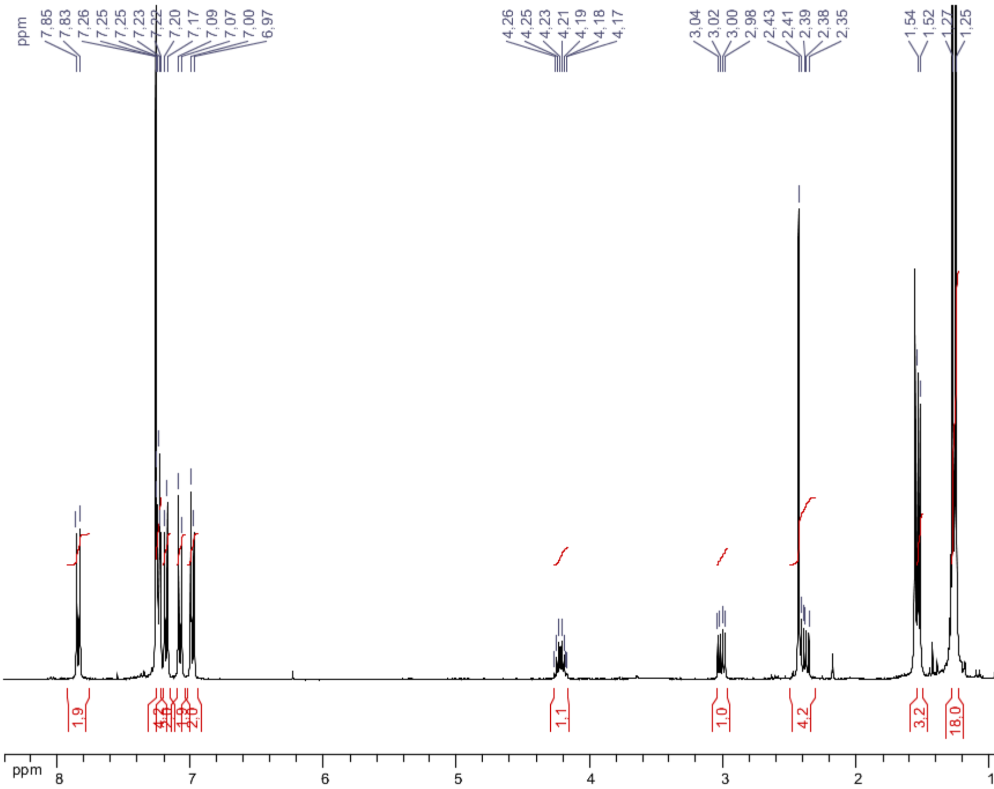
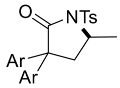
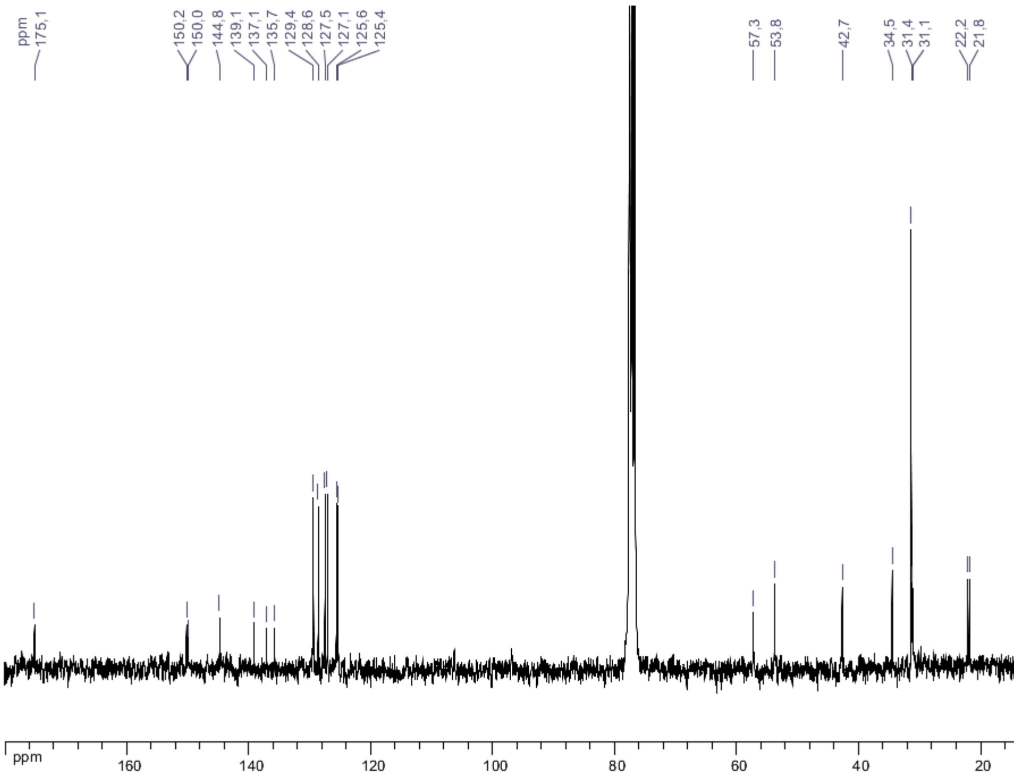
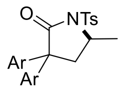
136,2
129,6
128,4
56,5
30,7
26,7
21,8
21,6
O
NTs
6d
ppm
160
140
120
100
80
60
40
20
0



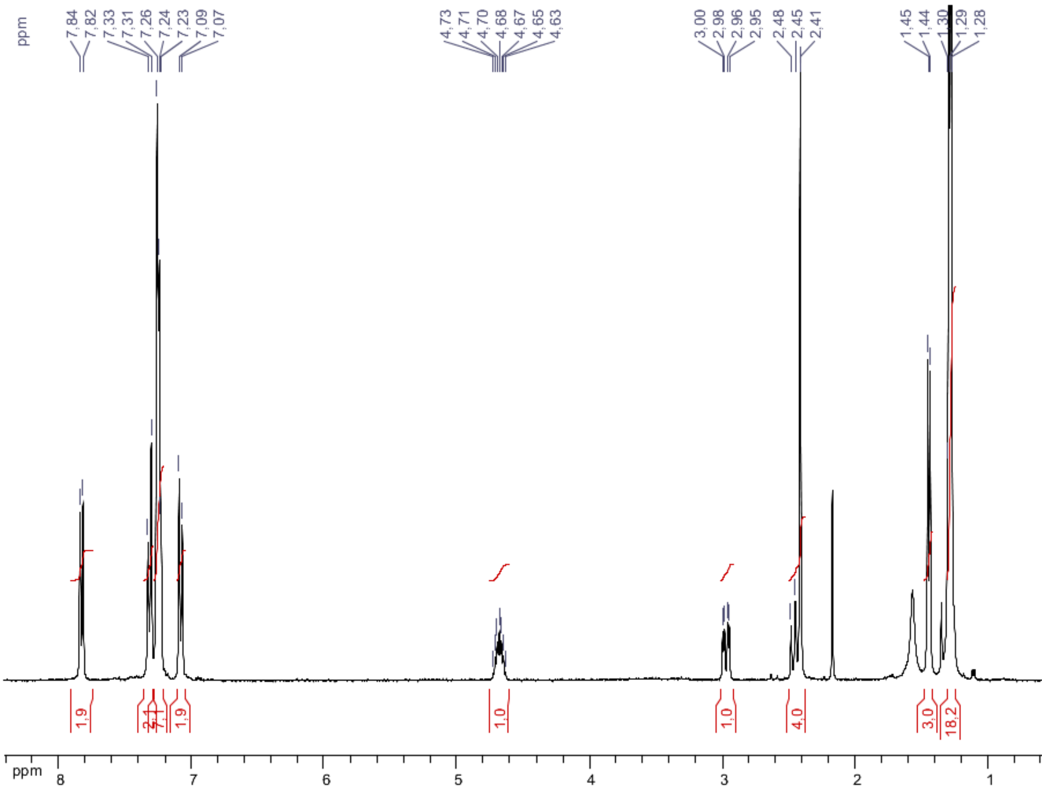


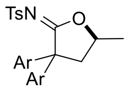
6e
Ar = 4-t-BuC_6H_4



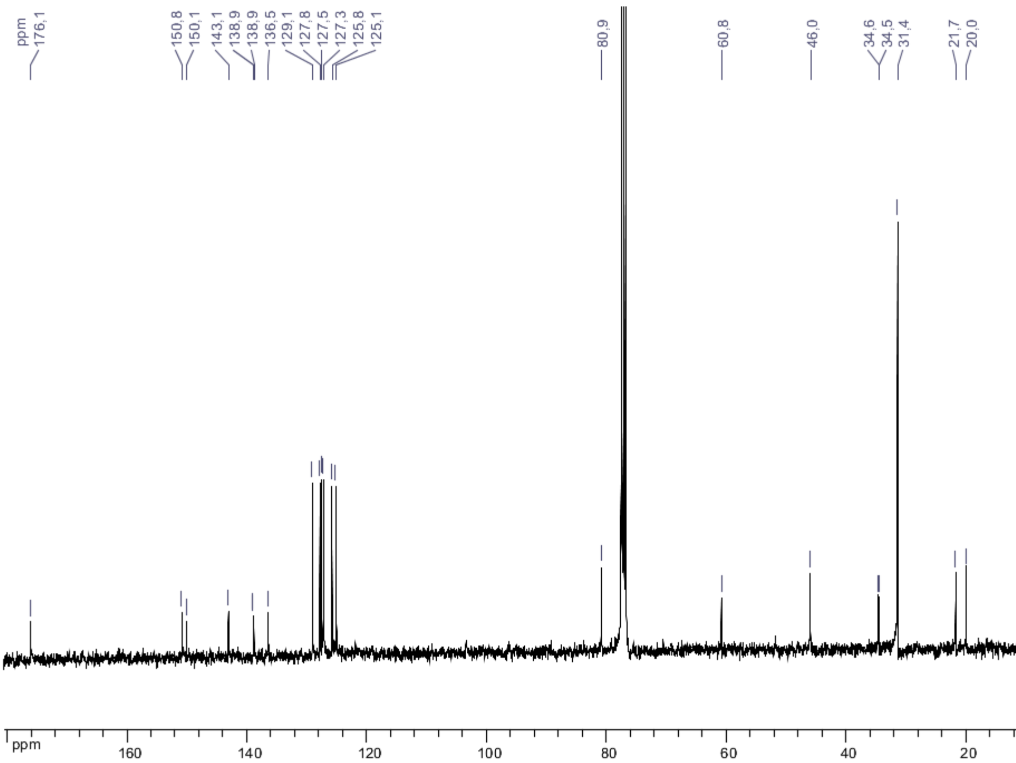


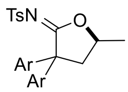
6e
Ar = 4-t-BuC_6H_4



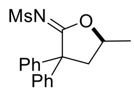


7e
Ar = 4-*t*-BuC$_6$H$_4$

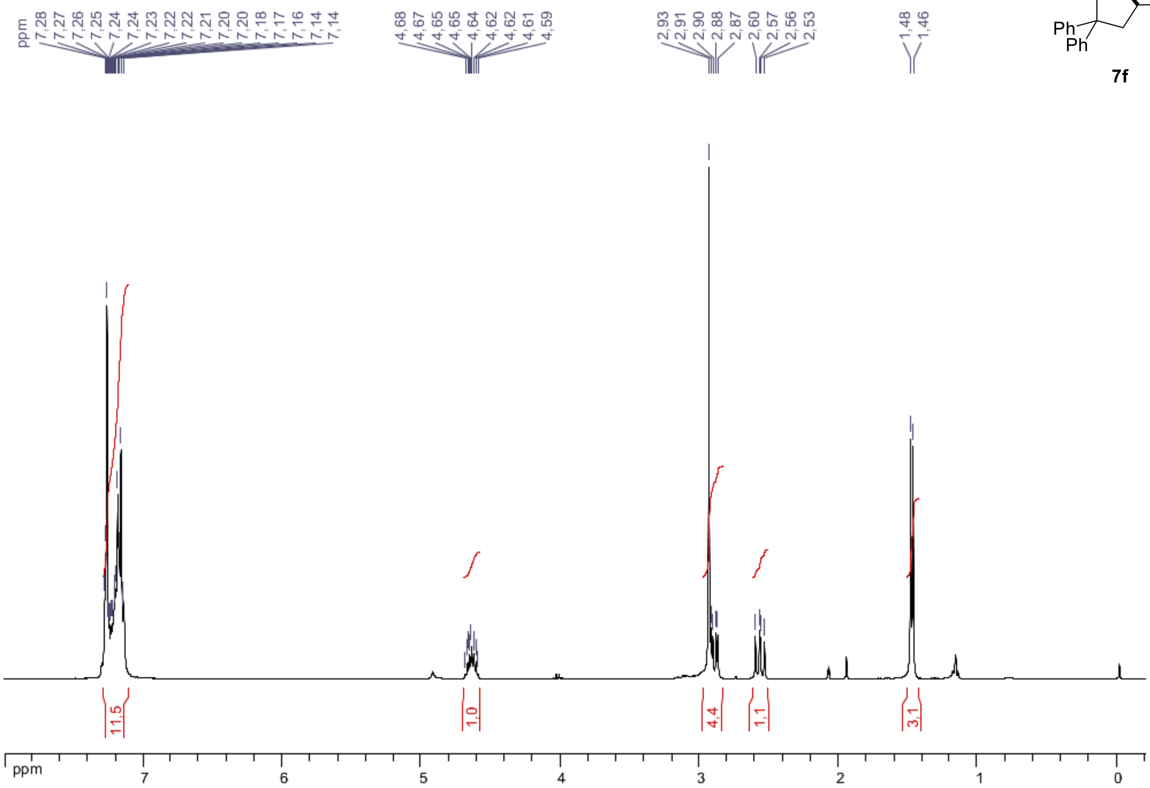


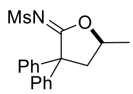


7e
Ar = 4-*t*-BuC$_6$H$_4$

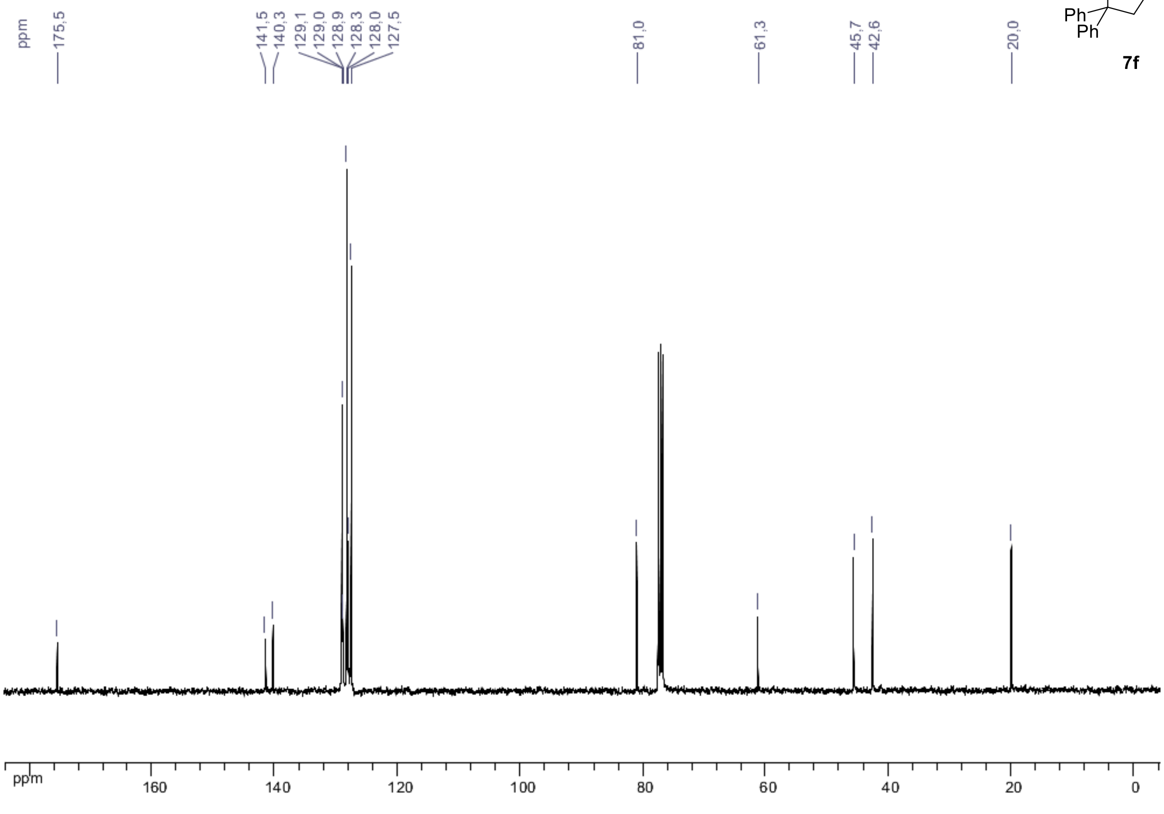

MsN
O
Ph
Ph

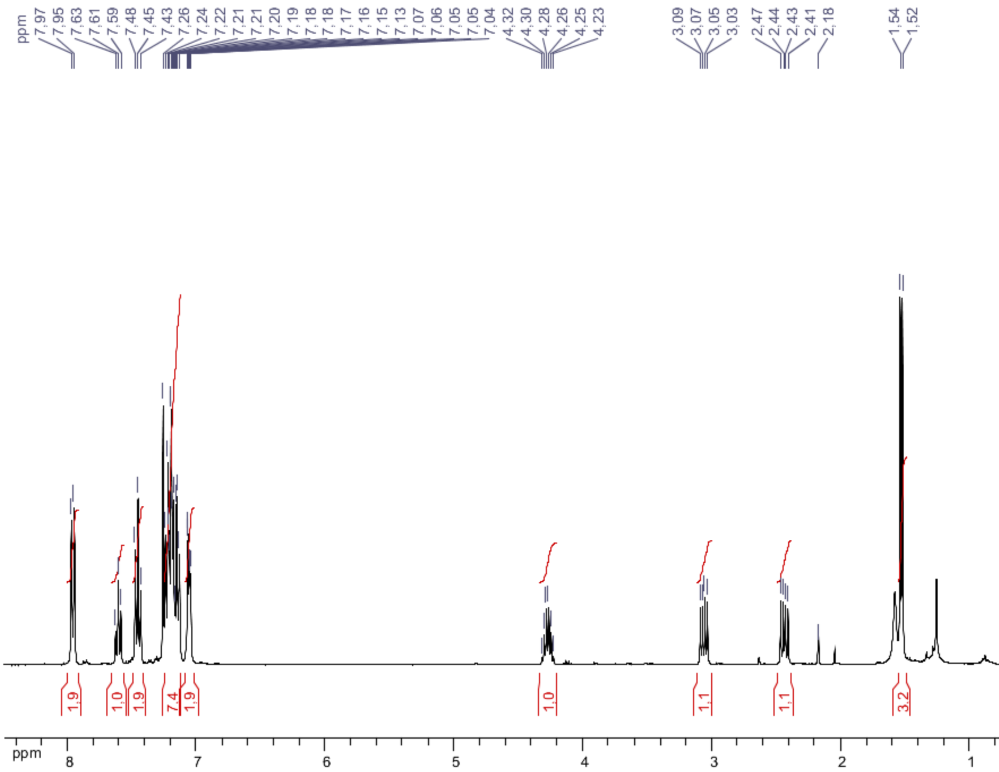
7f

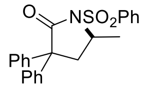

ppm 7,28 7,27 7,26 7,25 7,24 7,24 7,23 7,22 7,22 7,21 7,20 7,20 7,18 7,17 7,16 7,14 7,14
4,68 4,67 4,65 4,65 4,64 4,62 4,62 4,61 4,59
2,93 2,91 2,90 2,88 2,87 2,60 2,57 2,56 2,53
1,48 1,46
11,5
1,0
4,4
1,1
3,1
ppm 7 6 5 4 3 2 1 0


MsN
O
Ph
Ph

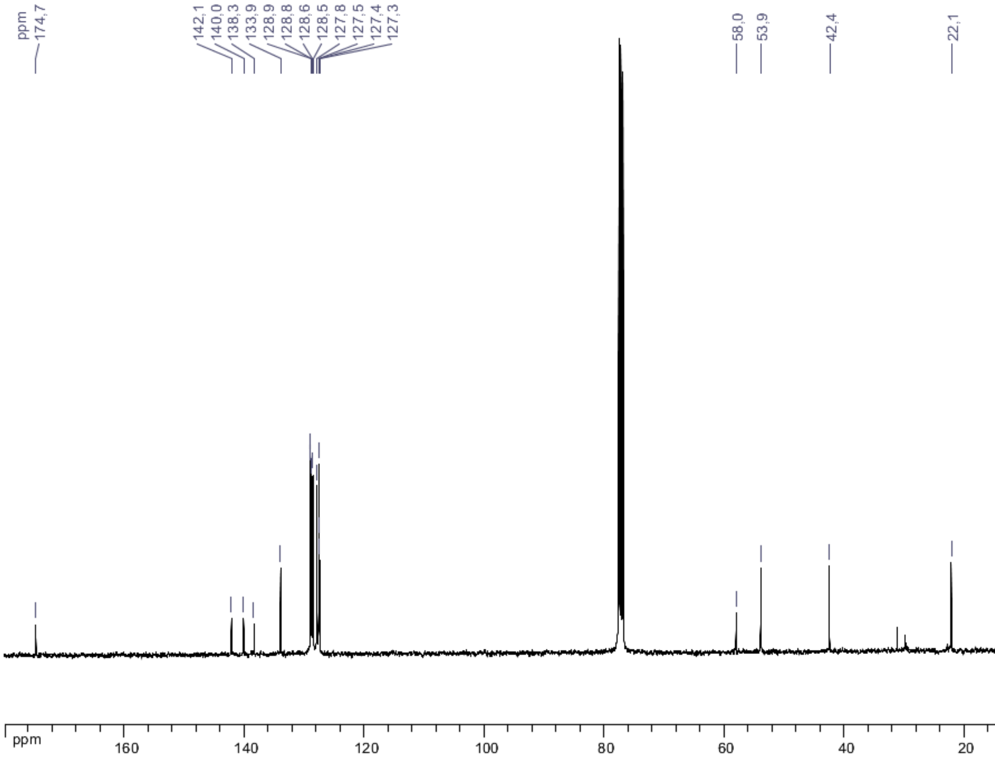
7f

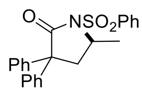

ppm 175,5
141,5 140,3 129,1 129,0 128,9 128,3 128,0 127,5
81,0
61,3
45,7 42,6
20,0
ppm 160 140 120 100 80 60 40 20 0


ppm
7,97 7,95 7,63 7,61 7,59 7,48 7,45 7,43 7,26 7,24 7,22 7,21 7,21 7,20 7,19 7,18 7,18 7,17 7,16 7,15 7,13 7,07 7,06 7,05 7,05 7,04
4,32 4,30 4,28 4,26 4,25 4,23
3,09 3,07 3,05 3,03
2,47 2,44 2,43 2,41 2,18
1,54 1,52
1,9 1,0 1,9 7,4 1,9 1,0 1,1 1,1 3,2
ppm 8 7 6 5 4 3 2 1 0

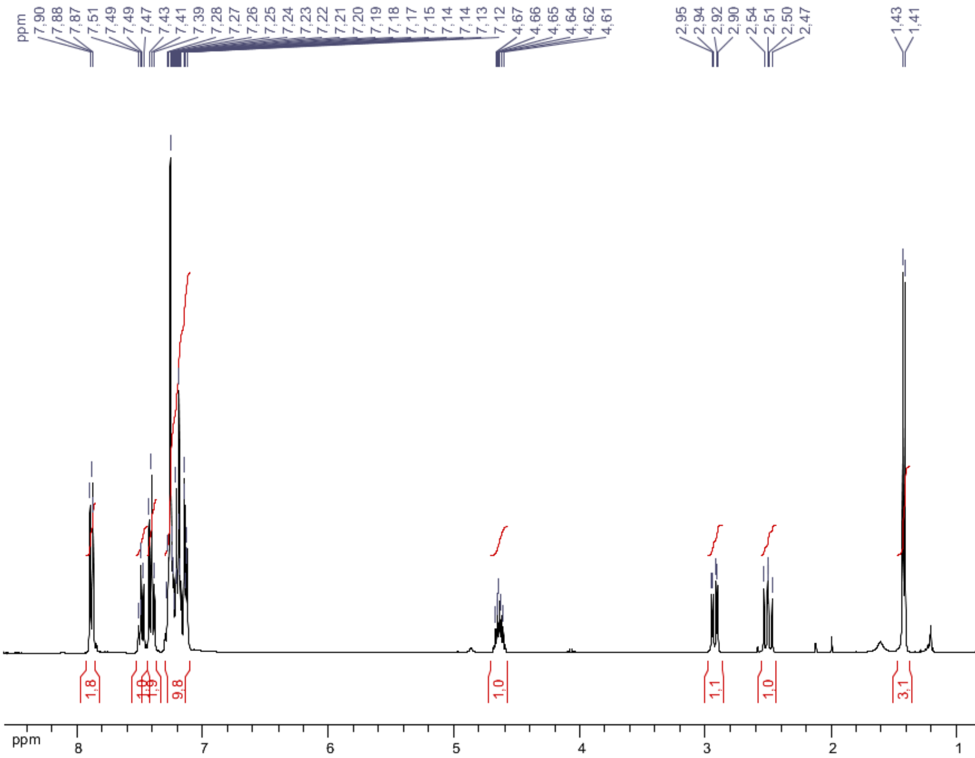

O
NSO2Ph
Ph
Ph

6g


ppm
174,7
142,1 140,0 138,3 133,9 128,9 128,8 128,6 128,5 127,8 127,5 127,4 127,3
58,0 53,9 42,4 22,1
ppm 160 140 120 100 80 60 40 20 0


O
NSO2Ph
Ph
Ph

6g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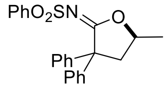
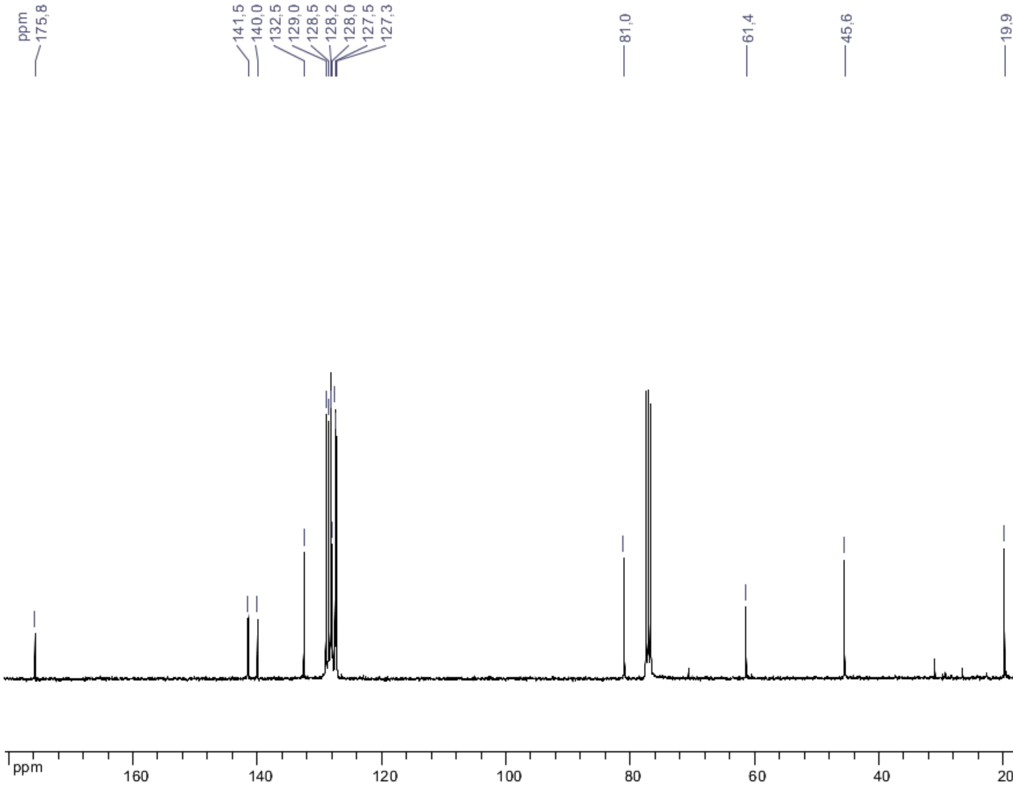
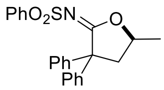
2,95 2,94 2,92 2,90 2,54 2,51 2,50 2,47
1,43 1,41
1,8 1,0 1,9 9,8 1,0 1,1 1,0 3,1
ppm 8 7 6 5 4 3 2 1 0

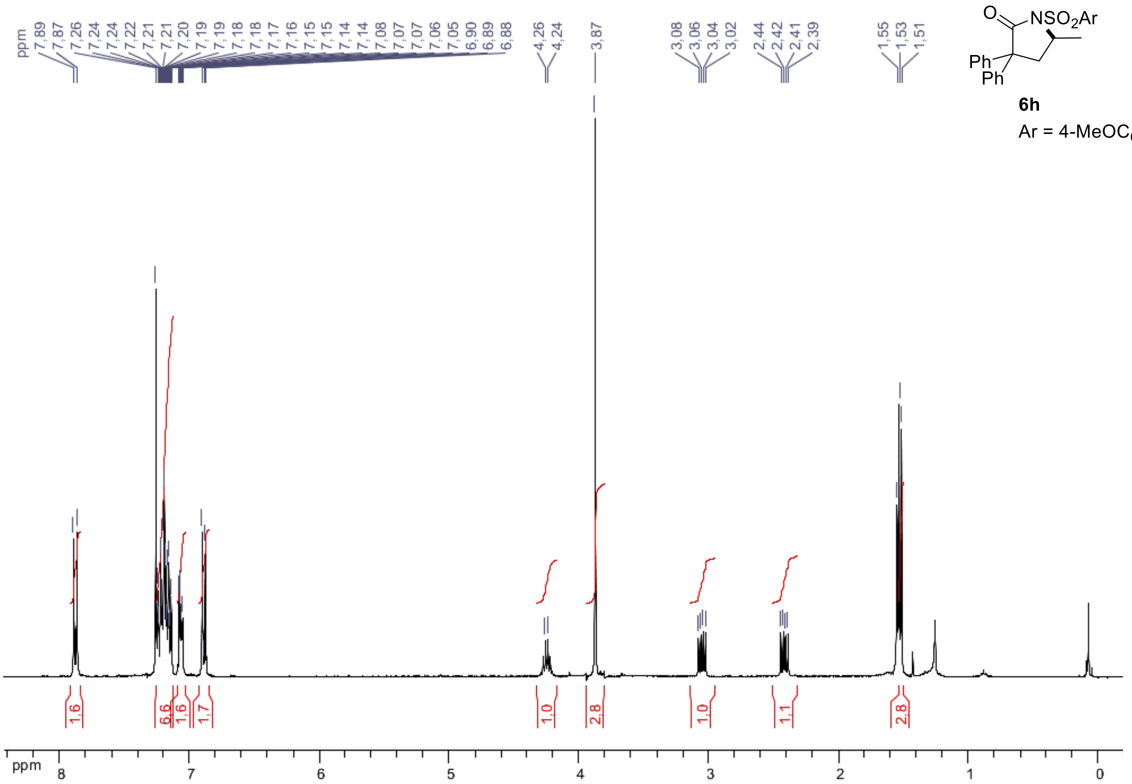

PhO2SN
O
Ph
Ph

7g


ppm 175,8
141,5 140,0 132,5 129,0 128,5 128,2 128,0 127,5 127,3
81,0
61,4
45,6
19,9
ppm 160 140 120 100 80 60 40 20 0

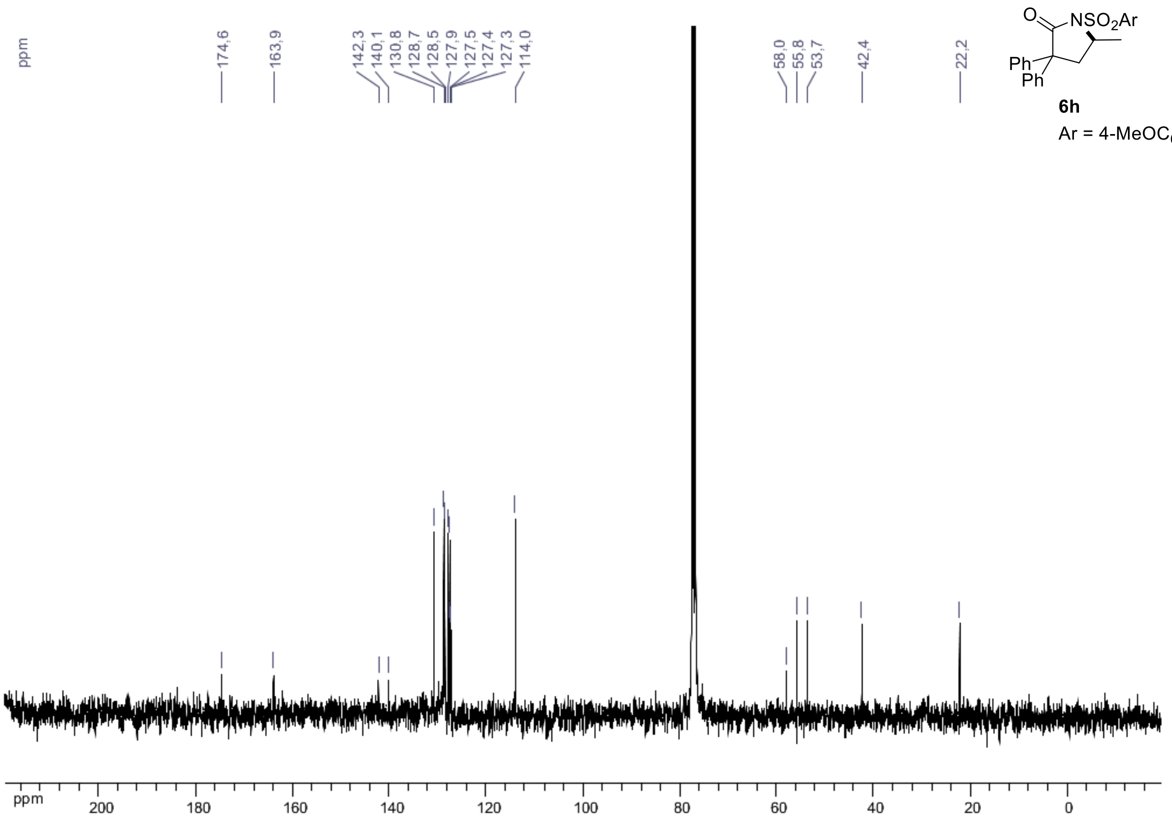

PhO2SN
O
Ph
Ph

7g



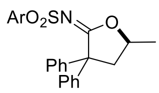
6h

Ar = 4-MeOC$_6$H$_4$



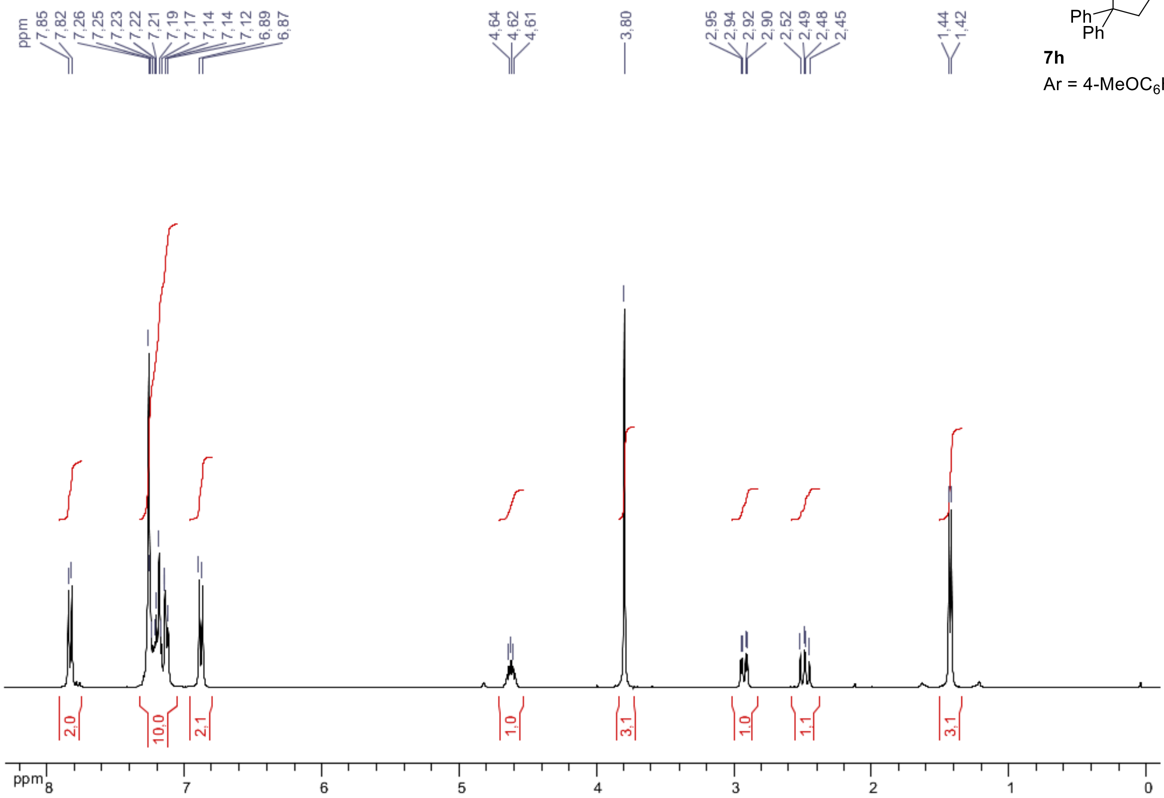
6h

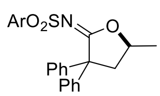
Ar = 4-MeOC$_6$H$_4$



7h

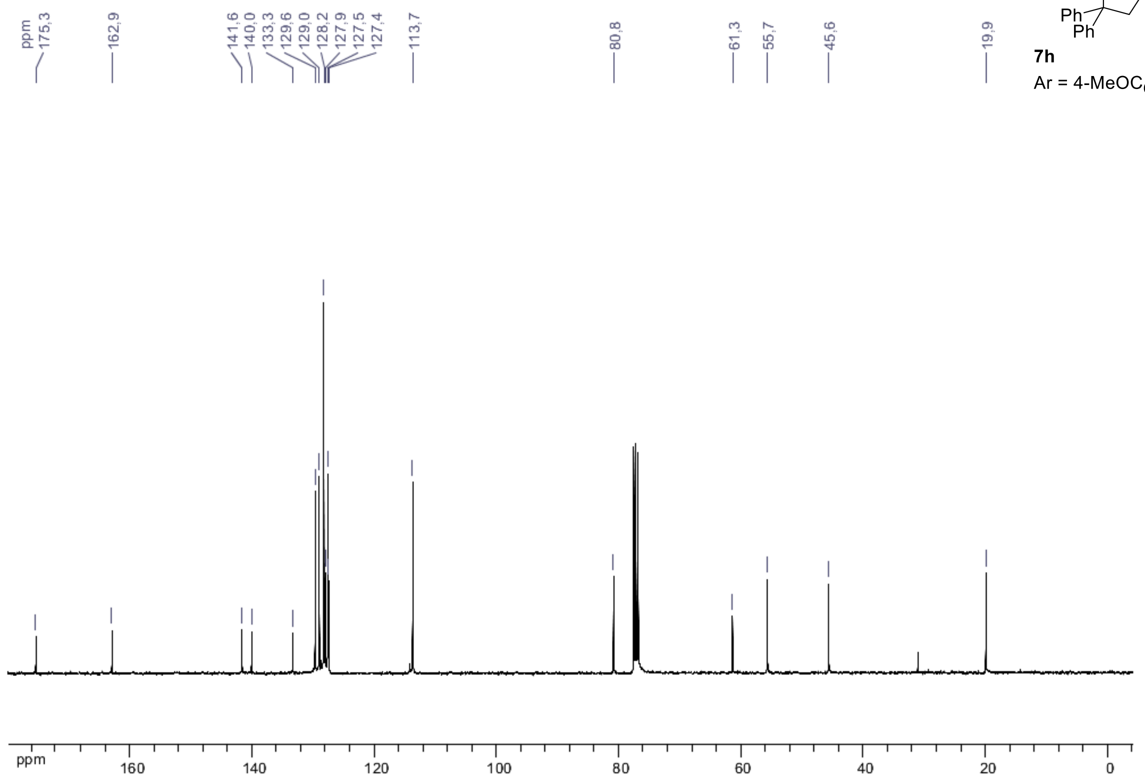
Ar = 4-MeOC$_6$H$_4$





7h

Ar = 4-MeOC$_6$H$_4$



6-References

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