Supplementary Information

Copper-Catalysed Oxidative C-H/C-H Coupling between Olefins and Simple Ethers

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General information

All reactions were isolated from moisture and oxygen by a nitrogen atmosphere in a sealed tube. All glassware was fully dried at 110 °C in oven for hours and cooled down under vacuum. Tetrahydrofuran was purified by distillation with sodium as the drying agent. CuI was purchased from Aladdin Chemical Reagent Co., Ltd. KI and DTBP (Di-*tert*-butyl peroxide, Chemical Purity) was purchased from Sinopharm Chemical Reagent Co., Ltd. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. Substituted 1,1-diphenylethene derivatives were all prepared following literature reports[1]. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Chromatography columns were packed with 200-300 mesh silica gel in petroleum ether (bp. 60-90 °C). Gas chromatographic analyses were performed on Varian GC 2000 gas chromatography instrument with a FID detector and biphenyl was added as an internal standard. GC-MS spectra were recorded on a Varian GC-MS 3900-2100T. ^{1}H and ^{13}C NMR data were recorded with Bruker ADVANCE III (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. All chemical shifts are reported relative to tetramethylsilane and *d*-solvent peaks (77.3 ppm, chloroform), respectively.

General procedure

General procedure for the copper-catalyzed oxidative alkenylation of simple ethers

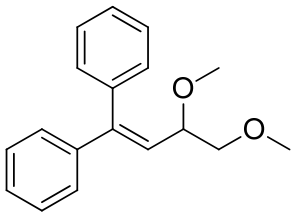
CuI (5.7 mg, 0.03 mmol) and KI (10.0 mg, 0.06 mmol) were added in a dried sealed tube. The sealed tube was then filled with dry N_2. Under the protection of N_2, ether (2.0 mL) was injected into the tube *via* a syringe. After stirring for a several minutes, *t*-BuOO*t*-Bu (87.6 mg, 0.6 mmol) was injected into the reaction tube by a microsyringe followed by the addition of alkene (0.3 mmol). The reaction was then heated up to 120 ^{o}C (*Warning: Heating simple ethers such as tetrahydrofuran with peroxide to 120 ^{o}C is of potential danger, please did this reaction in high qualified sealed tube and put the heater in fuming cupboard!*) and kept stirring for 24 hours. After completion of the reaction, the mixture was quenched with diluted hydrochloric acid. The solution was extracted with ethyl acetate (3 × 5 mL). The organic layers were combined and dried over sodium sulfate. The pure product was obtained by flash column chromatography on silica gel (petroleumether : ethyl acetate 100 : 1).

Regioselectivity Experiment

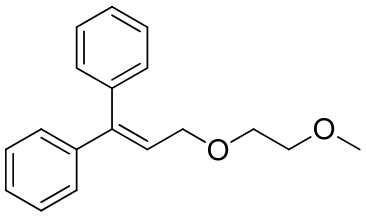
1. General procedure

CuI (5.7 mg, 0.03 mmol) and KI (10.0 mg, 0.06 mmol) were added in a dried sealed tube. The sealed tube was then filled with dry N_2. Under the protection of N_2, glycol dimethyl ether (2.0 mL) was injected into the tube *via* a syringe. After stirring for a several minutes, *t*-BuOO*t*-Bu (87.6 mg, 0.6 mmol) was injected into the reaction tube by a microsyringe followed by the addition of 1,1-diphenylethene (0.3 mmol). The reaction was then heated up to 120 ^oC (*Warning: Heating simple ethers such as tetrahydrofuran with peroxide to 120 ^oC is of potential danger, please did this reaction in high qualified sealed tube and put the heater in fuming cupboard!*) and kept stirring for 24 hours. After completion of the reaction, the pure product was obtained by flash column chromatography on silica gel (petroleum ether : ethyl acetate 100 : 1). Total isolated yield of the product is 46%. While the ratio of **3s** and **3s'** is 10 : 1 detected by ^1H NMR analysis.

2. Detailed description of products 3s and 3s'



3r



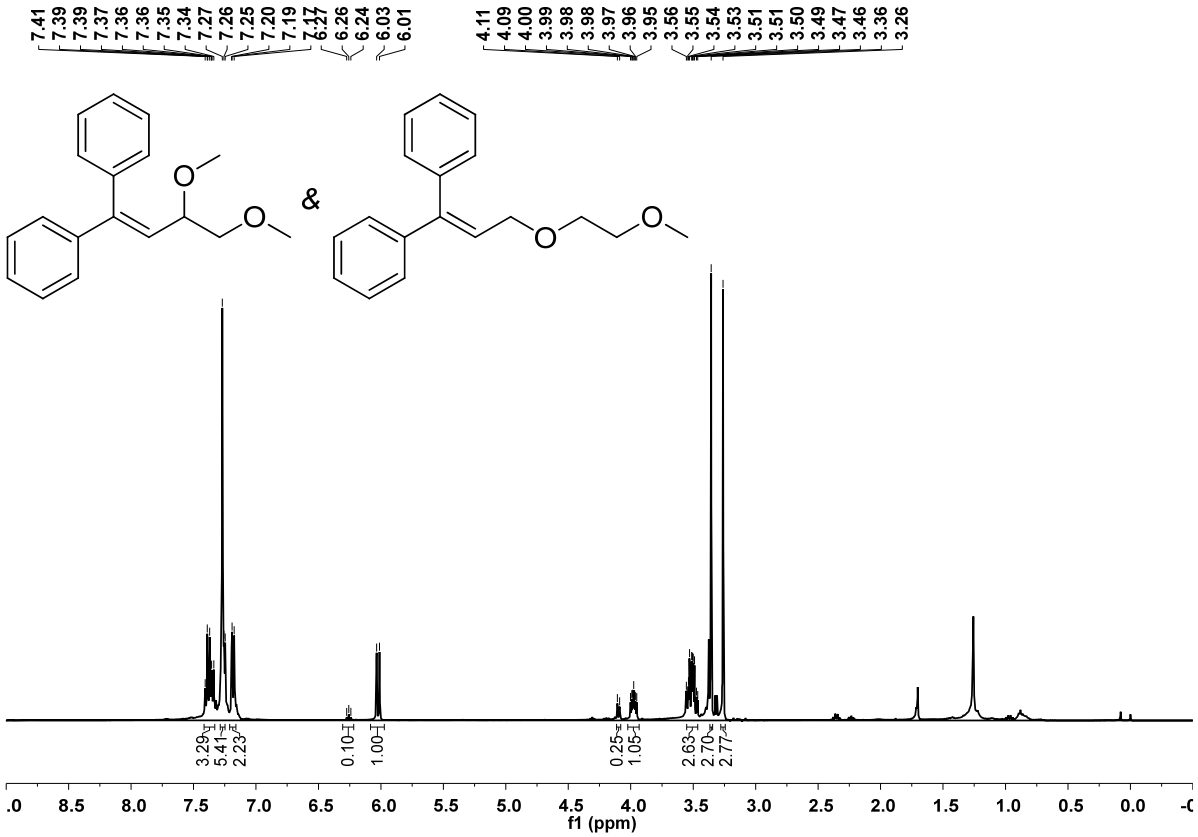
3r'

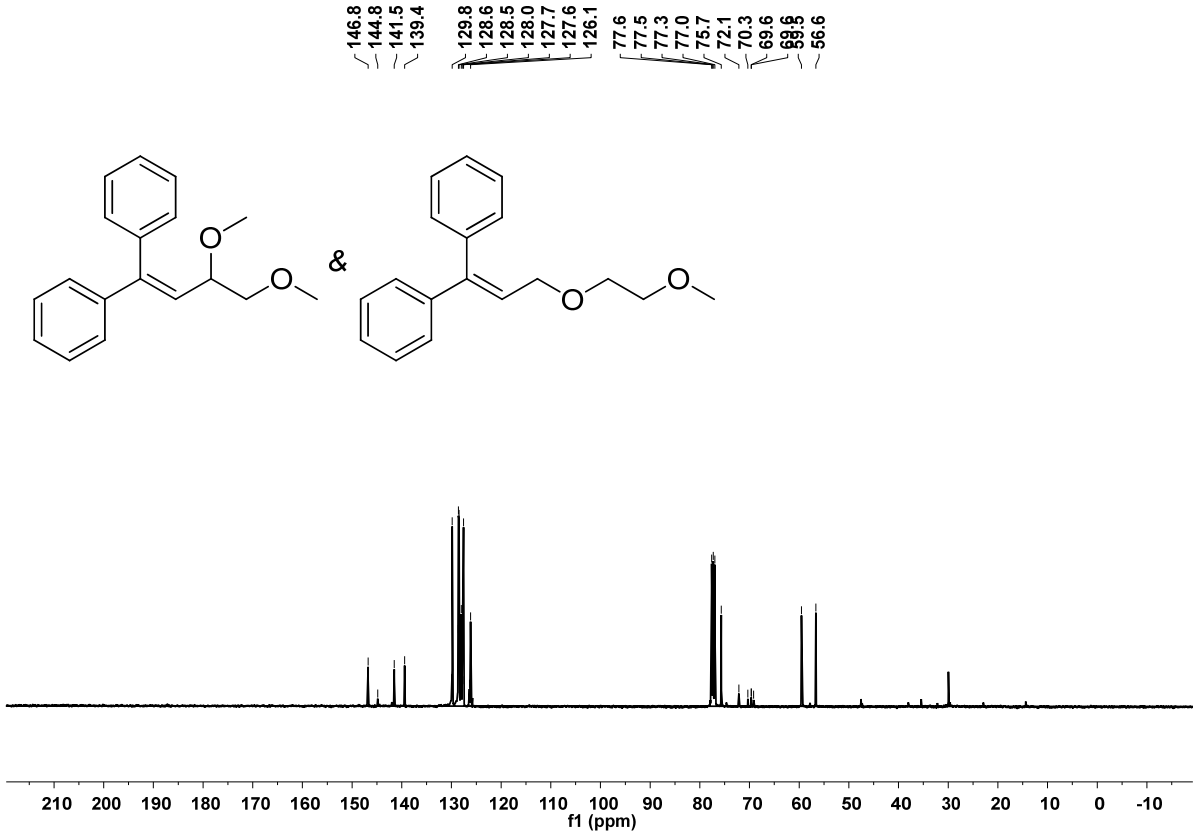
(3,4-dimethoxybut-1-ene-1,1-diyl)dibenzene (3r) and (3-(2-methoxyethoxy)prop-1-ene-1,1-diyl)dibenzene (3r'): Total isolated yield = 46%. **3r** : **3r'** = 10 : 1.

^1H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.33 (m, 3H), 7.29 – 7.24 (m, 5H), 7.21 – 7.16 (m, 2H), 6.26 (t, *J* = 6.6 Hz, 0.1H), 6.02 (d, *J* = 9.5 Hz, 1H), 4.10 (d, *J* = 6.7 Hz, 0.25H), 4.02 – 3.93 (m, 1H), 3.55 – 3.46 (m, 2H), 3.36 (s, 3H), 3.26 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 146.8, 144.8, 141.5, 139.4, 130.0, 129.8, 128.6(4), 128.6(1), 128.5, 128.3(9),

128.3(5), 128.1, 128.0, 127.8, 127.7(4), 127.6(9), 127.6, 126.4, 126.1, 77.6, 77.5, 77.3, 77.0, 75.7, 72.1, 70.3, 69.6, 69.2, 59.5, 56.6.



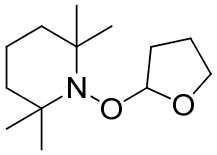


Radical-Trapping Experiment

1. General procedure

CuI (5.7 mg, 0.03 mmol) and KI (10.0 mg, 0.06 mmol) were added in a dried sealed tube. The sealed tube was then filled with dry N_2. Under the protection of N_2, THF (2.0 mL) was injected into the tube *via* a syringe. After stirring for a several minutes, *t*-BuOO*t*-Bu (87.6 mg, 0.6 mmol) was injected into the reaction tube by a microsyringe followed by the addition of 1,1-diphenylethene (0.3 mmol). Finally, radical-trapping reagent TEMPO was added into the reaction tube which was then heated up to 120 °C (*Warning: Heating simple ethers such as tetrahydrofuran with peroxide to 120 °C is of potential danger, please did this reaction in high qualified sealed tube and put the heater in fuming cupboard!*) and kept stirring for 24 hours. After completion of the reaction, the pure product **3I** was obtained in 53% yield by flash column chromatography on silica gel (petroleum ether : ethyl acetate 100 : 1) without quenching.

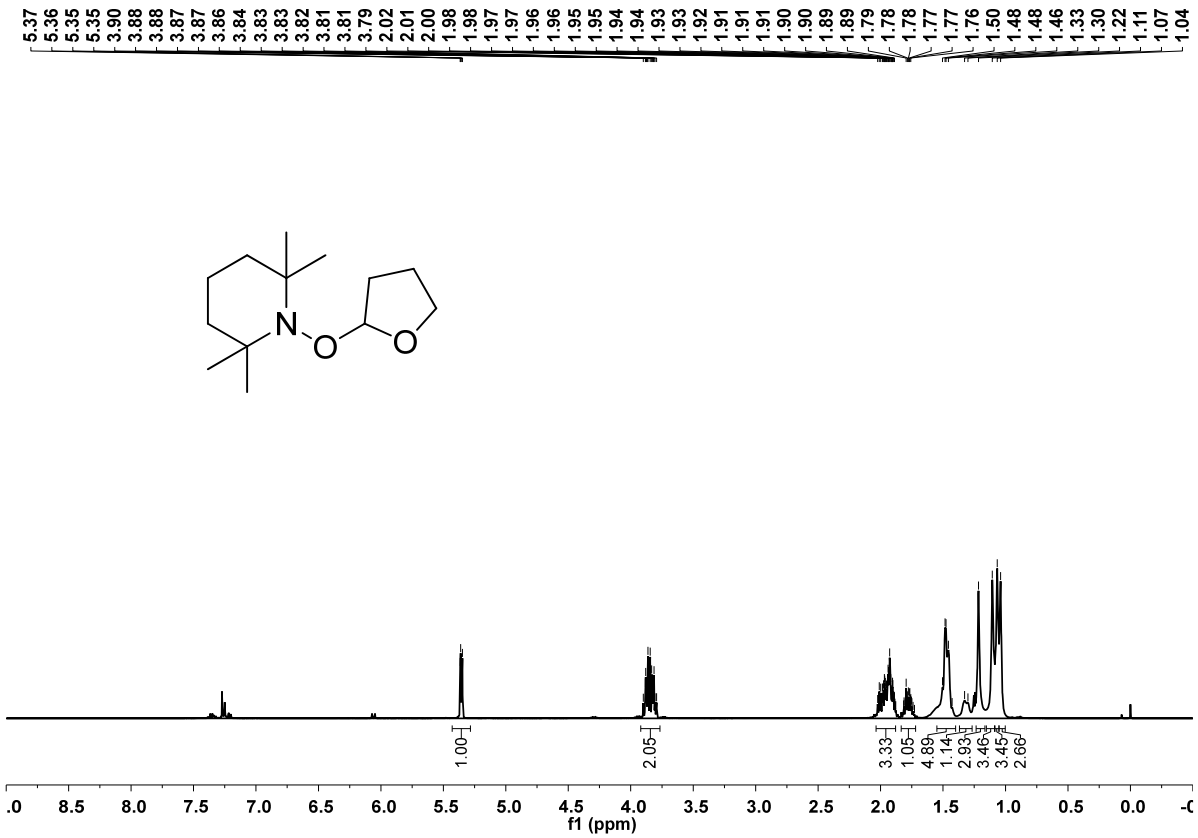
2. Radical-Trapping Product Description



2,2,6,6-tetramethyl-1-((tetrahydrofuran-2-yl)oxy)piperidine (3I)[2]: Isolated yield = 53%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.36 (dd, *J* = 5.4, 1.9 Hz, 1H), 3.92 – 3.77 (m, 2H), 2.04 – 1.88 (m, 3H), 1.83 – 1.72 (m, 1H), 1.55 – 1.40 (m, 5H), 1.35 – 1.28 (m, 1H), 1.22 (s, 3H), 1.11 (s, 3H), 1.07 (s, 3H), 1.04 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 109.8, 77.6, 77.3, 77.0, 66.9, 60.4, 58.9, 40.3, 39.9, 34.2, 33.6, 31.5, 24.2, 20.7, 20.3, 17.5.


5.37 5.36 5.35 5.35 3.90 3.88 3.88 3.87 3.87 3.86 3.84 3.83 3.82 3.81 3.81 3.79 2.02 2.01 2.00 1.98 1.98 1.97 1.97 1.96 1.96 1.95 1.95 1.94 1.94 1.93 1.93 1.92 1.91 1.91 1.91 1.90 1.90 1.89 1.89 1.79 1.78 1.78 1.77 1.77 1.76 1.50 1.48 1.48 1.46 1.33 1.30 1.22 1.11 1.07 1.04
N
O
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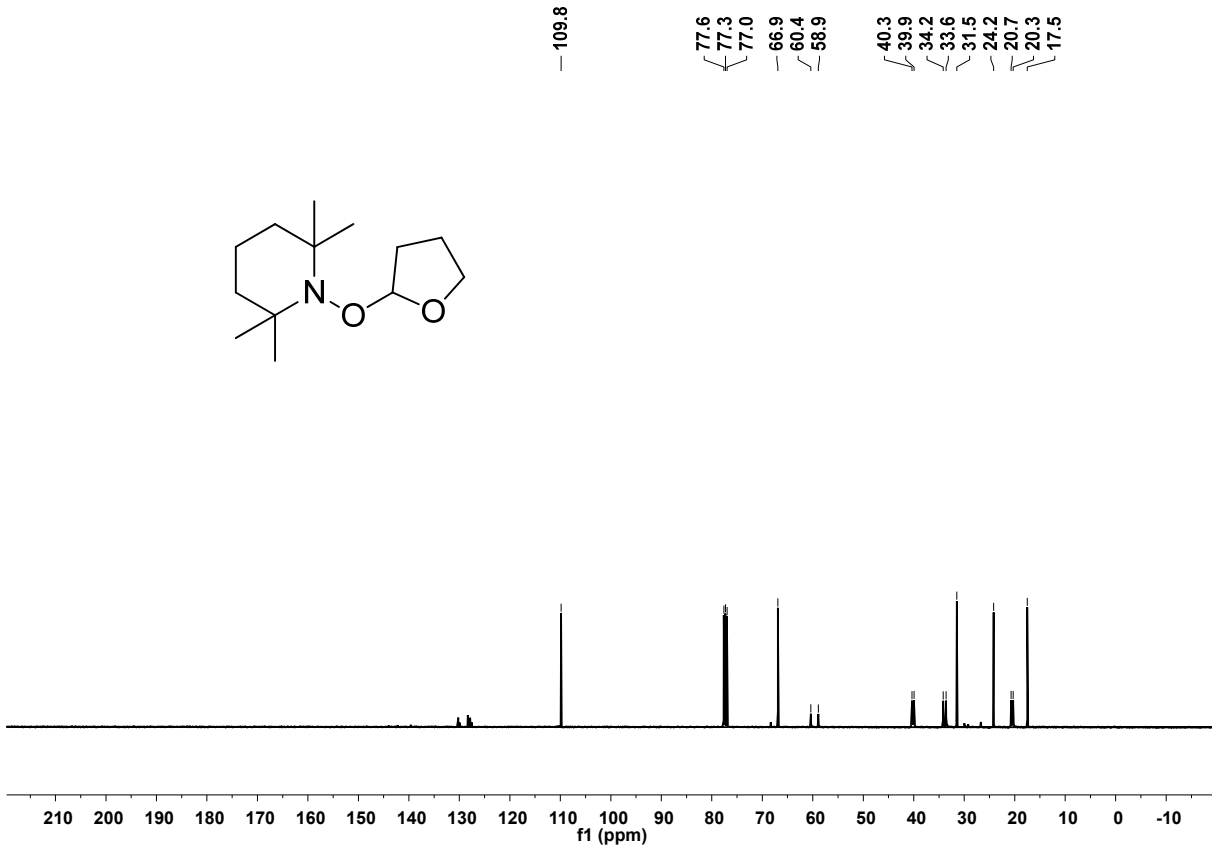
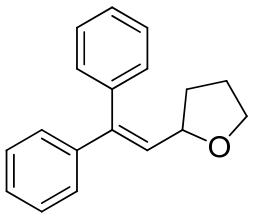
3.45
2.66
.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


109.8
77.6
77.3
77.0
66.9
60.4
58.9
40.3
39.9
34.2
33.6
31.5
24.2
20.7
20.3
17.5
N
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

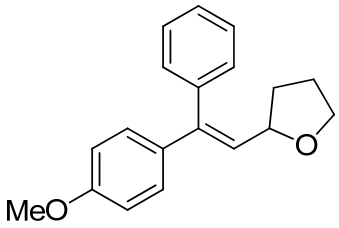
Detailed Descriptions for products:



2-(2,2-diphenylvinyl)tetrahydrofuran (3a): Isolated yield = 87%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.30 (m, 3H), 7.27 – 7.23 (m, 5H), 7.23 – 7.19 (m, 2H), 6.06 (d, *J* = 9.0 Hz, 1H), 4.38 – 4.19 (m, 1H), 3.98 – 3.89 (m, 1H), 3.76 – 3.69 (m, 1H), 2.10 – 1.92 (m, 2H), 1.91 – 1.79 (m, 1H), 1.78 – 1.67 (m, 1H).

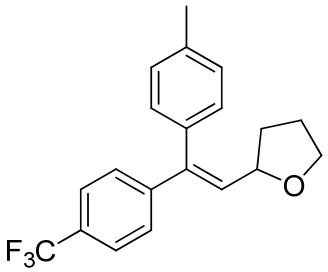
^{13}C NMR (101 MHz, $CDCl_3$) δ 143.9, 142.2, 139.7, 130.2, 130.0, 128.3(3), 128.3(2), 127.8, 127.7, 127.6, 77.6, 77.3, 77.0, 76.9, 68.3, 33.3, 26.7. HRMS (ESI) calcd for $C_{18}H_{19}O^+$ $[M+H]^+$: 251.1430; found: 251.1417.



(*E, Z*)-2-(2-(4-methoxyphenyl)-2-phenylvinyl)tetrahydrofuran (3b): Isolated yield = 94%. *E/Z* isomers. *E/Z* = 5:3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 2H), 7.28 – 7.23 (m, 2H), 7.22 – 7.13 (m, 3H), 6.89 (d, *J* = 8.6 Hz, 1H), 6.79 (d, *J* = 8.8 Hz, 1H), 6.03 – 5.95 (m, 1H), 4.38 – 4.21 (m, 1H), 3.98 – 3.89 (m, 1H), 3.82 (s, 1.2H), 3.77 (s, 2H), 3.75 – 3.68 (m, 1H), 2.10 – 1.93 (m, 2H), 1.91 – 1.78 (m, 1H), 1.78 – 1.65 (m, 1H).

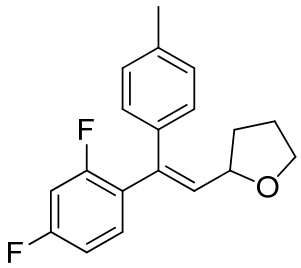
^{13}C NMR (101 MHz, $CDCl_3$) δ 159.3, 159.1, 143.7, 143.5, 142.6, 139.9, 134.8, 132.8, 131.4, 130.2, 129.9, 129.6, 129.0, 128.3, 128.2, 127.9, 127.6, 127.5, 113.6(7), 113.6(5), 77.6, 77.3, 77.0, 76.9, 68.3, 68.2, 55.5, 55.4, 33.3, 26.6(9), 26.6(6). HRMS (ESI) calcd for $C_{19}H_{21}O_2^+$ $[M+H]^+$: 281.1536; found: 281.1520.



(*E,Z*)-2-(2-(*p*-tolyl)-2-(4-(trifluoromethyl)phenyl)vinyl)tetrahydrofuran (3c): Isolated yield = 78%. E/Z isomers. E/Z = 2:1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.1 Hz, 1H), 7.51 (d, *J* = 8.2 Hz, 1H), 7.35 (dd, *J* = 8.0, 4.0 Hz, 2H), 7.19 (d, *J* = 7.8 Hz, 1H), 7.11 – 7.05 (m, 3H), 6.09 (d, *J* = 9.2 Hz, 1H), 4.37 – 4.28 (m, 0.5H), 4.26 – 4.15 (m, 0.7H), 3.99 – 3.90 (m, 1H), 3.78 – 3.70 (m, 1H), 2.38 (s, 1H), 2.33 (s, 2H), 2.08 – 1.95 (m, 2H), 1.93 – 1.81 (m, 1H), 1.77 – 1.68 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 146.0, 143.6(5), 143.6(4), 142.8, 142.7, 138.6, 138.0, 137.8, 135.9, 131.8, 130.6, 130.1, 130.0, 129.9, 129.6, 129.3, 128.1, 127.7, 125.8, 125.4 – 125.3 (m), 123.1, 77.6, 77.3, 77.0, 76.8, 76.7, 68.5, 68.4, 33.3, 26.7(2), 26.6(9), 21.5, 21.3. HRMS (EI^+) calcd for $C_{20}H_{19}F_3O^+$ (M^+): 332.1388; found: 332.1386.

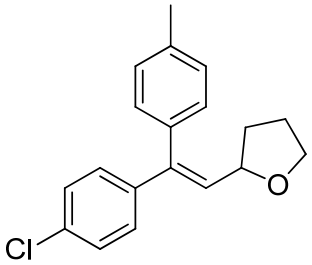


(*E,Z*)-2-(2-(2,4-difluorophenyl)-2-(*p*-tolyl)vinyl)tetrahydrofuran (3d): Isolated yield = 83%. E/Z isomers. E/Z = 2:1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 – 7.05 (m, 8H), 6.95 – 6.81 (m, 2H), 6.81 – 6.70 (m, 1H), 6.19 (d, *J* = 8.9 Hz, 1H), 5.90 (d, *J* = 9.1 Hz, 0.5H), 4.41 (dd, *J* = 15.5, 7.9 Hz, 0.5H), 4.16 (dd, *J* = 15.1, 7.9 Hz, 1H), 3.98 – 3.88 (m, 1.7H), 3.79 – 3.68 (m, 1.7H), 2.35 (s, 1.7H), 2.31 (s, 3.1H), 2.06 – 1.93 (m, 2.8H), 1.92 –

1.80 (m, 1.7H), 1.80 – 1.64 (m, 2H).

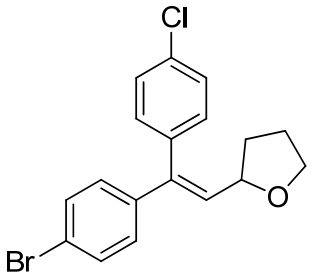
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.0 (d, *J* = 11.5 Hz), 163.7 (d, *J* = 11.8 Hz), 161.6 (d, *J* = 3.7 Hz), 161.5 (d, *J* = 3.2 Hz), 161.2 (d, *J* = 11.9 Hz), 159.1, 159.0, 137.9, 137.8, 137.5, 136.4, 136.2, 133.5 (d, *J* = 3.0 Hz), 133.0 (dd, *J* = 9.4, 5.2 Hz), 132.2 (dd, *J* = 9.4, 4.8 Hz), 131.5, 129.4, 129.3, 129.0, 126.7, 123.0 (dd, *J* = 16.9, 3.9 Hz), 111.5 (dd, *J* = 21.1, 3.7 Hz), 111.1 (dd, *J* = 20.9, 3.8 Hz), 104.3 (td, *J* = 25.6, 6.9 Hz), 77.6, 77.3, 77.1, 77.0, 76.4, 68.4, 33.3, 32.7, 26.7, 26.6, 21.5, 21.3. HRMS (ESI) calcd for $C_{19}H_{19}F_2O^+$ $[M+Na]^+$: 301.1398; found: 301.1377.



(*E,Z*)-2-(2-(4-chlorophenyl)-2-(*p*-tolyl)vinyl)tetrahydrofuran (3e): Isolated yield = 80%. E/Z isomers. E/Z = 3:2.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.2 Hz, 1H), 7.26 – 7.20 (m, 1H), 7.15 (dd, *J* = 15.4, 7.0 Hz, 3H), 7.08 (t, *J* = 7.9 Hz, 3H), 6.05 – 5.98 (m, 1H), 4.27 (ddd, *J* = 23.3, 14.9, 8.4 Hz, 1H), 3.93 (dd, *J* = 14.6, 7.2 Hz, 1H), 3.73 (dd, *J* = 13.7, 7.9 Hz, 1H), 2.37 (s, 1.5H), 2.32 (s, 2H), 2.09 – 1.94 (m, 2H), 1.91 – 1.80 (m, 1H), 1.76 – 1.69 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 142.9, 142.8, 141.0, 139.0, 138.2, 137.8, 137.5, 136.2, 133.5(0), 133.4(6), 131.6, 130.2, 130.0, 129.6, 129.1(7), 129.1(5), 129.1, 128.5, 128.4, 127.7, 77.6, 77.3, 77.0, 76.9, 76.8, 68.4, 68.3, 33.3, 26.7(1), 26.6(9), 21.5, 21.3. HRMS (ESI) calcd for $C_{19}H_{20}ClO^+$ $[M+H]^+$: 299.1197; found: 299.1234.

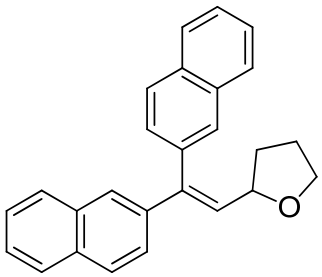


(*E,Z*)-2-(2-(4-bromophenyl)-2-(4-chlorophenyl)vinyl)tetrahydrofuran (3f): Isolated yield = 83%. E/Z isomers.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.2 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 1H), 7.34 (d, *J* = 8.2 Hz, 1H), 7.28 – 7.20 (m, 1H), 7.14 (dd, *J* = 8.3, 4.9 Hz, 2H), 7.08 (dd, *J* = 8.3, 3.6 Hz, 2H), 6.07 – 6.00 (m, 1H), 4.23 (dd, *J* = 15.0, 8.1 Hz, 1H), 3.93 (dd, *J* = 14.7, 7.0 Hz, 1H), 3.74 (dd, *J* = 13.7, 7.8 Hz, 1H), 2.08 – 1.95 (m, 2H), 1.93 – 1.82 (m, 1H), 1.76 – 1.68 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.9(4), 141.9(0), 140.8, 140.2, 138.1, 137.5, 133.9, 133.8, 131.8, 131.7, 131.6, 131.5, 131.0, 130.9, 129.4, 129.1, 128.8, 128.6, 122.1, 122.0, 77.6, 77.3, 77.0, 76.7, 68.5, 33.3, 26.7.

HRMS (EI^+) calcd for $C_{18}H_{16}BrClO^+$ (M^+): 362.0073; found: 362.0069.

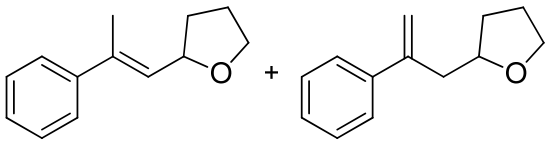


2-(2,2-di(naphthalen-2-yl)vinyl)tetrahydrofuran (3g): Isolated yield = 63%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.82 (m, 3H), 7.81 – 7.72 (m, 3H), 7.71 – 7.65 (m, 1H), 7.62 (s, 1H), 7.55 – 7.47 (m, 3H), 7.45 – 7.37 (m, 2H), 7.34 (d, *J* = 8.4 Hz, 1H), 6.30 (d, *J* = 8.9 Hz, 1H), 4.39 (dd, *J* = 15.5, 7.3 Hz, 1H), 3.97 (dd, *J* = 14.5, 6.9 Hz, 1H), 3.73 (dd, *J* = 13.8, 7.4 Hz, 1H), 2.14 – 1.95 (m, 2H), 1.91 – 1.75 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 143.9, 139.5, 137.1, 133.5, 133.4, 133.1, 133.0, 130.9, 129.2, 128.4(9),

128.4(7), 128.4, 128.0(4), 127.9(8), 127.9(7), 127.8, 127.4, 126.5, 126.4, 126.2, 125.8, 77.6, 77.3, 77.1, 77.0, 68.4, 33.4, 26.7. HRMS (ESI) calcd for $C_{26}H_{23}O^+$ $[M+H]^+$: 351.1743; found: 351.1751.

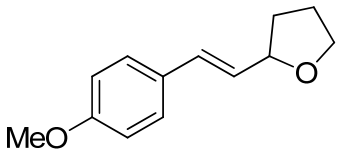


A (E/Z) **B**

(*E,Z*)-2-(2-phenylprop-1-en-1-yl)tetrahydrofuran (3h-A) & 2-(2-phenylallyl)tetrahydrofuran (3h-B): mixtures. total isolated yield = 55%. **A/B** = 4:5. **A** (E/Z = 1:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.39 (m, 3H), 7.35 – 7.30 (m, 3H), 7.30 – 7.22 (m, 3H), 5.81 (dd, *J* = 8.1, 1.3 Hz, 0.4H), 5.34 (d, *J* = 1.4 Hz, 1H), 5.15 (d, *J* = 1.3 Hz, 1H), 4.69 (dd, *J* = 14.6, 8.0 Hz, 0.4H), 4.01 – 3.86 (m, 3H), 3.86 – 3.77 (m, 1H), 3.73 – 3.65 (m, 1H), 2.88 (ddd, *J* = 14.3, 6.4, 1.0 Hz, 1H), 2.60 (ddd, *J* = 14.3, 6.9, 0.9 Hz, 1H), 2.17 – 2.06 (m, 2H), 2.04 – 1.74 (m, 5H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.9, 143.3, 141.3, 137.5, 129.3, 128.5, 128.4, 127.7, 127.3, 126.4, 126.1, 114.5, 77.8, 77.6, 77.3, 77.0, 76.4, 68.2, 68.1, 41.9, 32.8, 31.4, 26.5, 25.8, 16.6. HRMS (ESI) calcd for $C_{13}H_{17}O^+$ $[M+H]^+$: 189.1274; found: 189.1259.

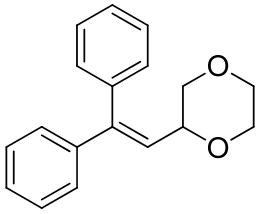


(*E*)-2-(4-methoxystyryl)tetrahydrofuran (3i)[3]: Isolated yield = 42%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.7 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.52 (d, *J* = 15.8 Hz, 1H), 6.06 (dd, *J* = 15.8, 6.8 Hz, 1H), 4.44 (q, *J* = 6.9 Hz, 1H), 3.96 (dd, *J* = 14.5, 7.4 Hz, 1H), 3.87 – 3.80 (m, 1H), 3.79 (s, 3H), 2.17 – 2.05 (m, 1H), 2.02 – 1.89 (m, 2H), 1.76 – 1.64 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.4, 130.4, 129.8, 128.5, 127.9, 114.1, 80.2, 77.6, 77.3, 77.0, 68.3, 55.5,

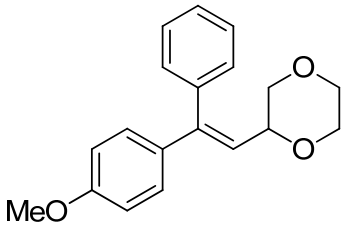
32.7, 26.2.



2-(2,2-diphenylvinyl)-1,4-dioxane (3j): Isolated yield = 81%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.34 (m, 3H), 7.28 – 7.22 (m, 5H), 7.22 – 7.18 (m, 2H), 5.93 (d, *J* = 8.9 Hz, 1H), 4.13 (td, *J* = 9.8, 2.8 Hz, 1H), 3.80 – 3.75 (m, 1H), 3.72 (dd, *J* = 11.5, 2.6 Hz, 1H), 3.67 – 3.61 (m, 3H), 3.48 (dd, *J* = 11.5, 10.0 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 147.1, 141.7, 139.3, 129.8, 128.6, 128.4, 128.1, 128.0, 127.8, 124.4, 77.6, 77.3, 77.0, 74.0, 70.6, 66.4(0), 66.3(6). HRMS (ESI) calcd for $C_{18}H_{19}O_2^+$ $[M+H]^+$: 267.1380; found: 267.1363.

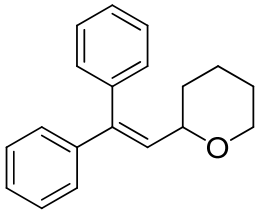


(*E,Z*)-2-(2-(4-methoxyphenyl)-2-phenylvinyl)-1,4-dioxane (3k): Isolated yield = 61%. E/Z isomers. E/Z = 2:1.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.33 (m, 2H), 7.28 – 7.22 (m, 2H), 7.22 – 7.16 (m, 2H), 7.16 – 7.12 (m, 1H), 6.93 – 6.89 (m, 1H), 6.83 – 6.77 (m, 1H), 5.88 – 5.82 (m, 1H), 4.16 (td, *J* = 9.8, 2.7 Hz, 0.4H), 4.08 (td, *J* = 9.8, 2.8 Hz, 0.7H), 3.84 (s, 1H), 3.81 – 3.74 (m, 3H), 3.71 (dd, *J* = 11.5, 2.4 Hz, 1H), 3.69 – 3.60 (m, 3H), 3.48 (ddd, *J* = 11.5, 10.0, 5.7 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.7, 159.4, 147.0, 146.6, 142.1, 139.5, 134.2, 131.6, 131.0, 129.8, 129.0,

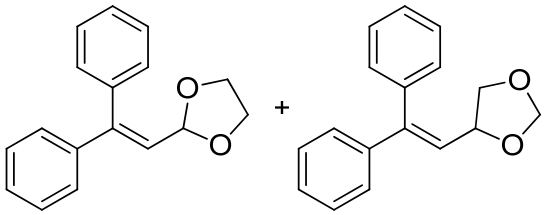
128.5, 128.4, 128.1, 128.0, 127.9, 124.0, 122.5, 113.9, 113.8, 77.6, 77.3, 77.0, 74.1, 74.0, 70.7(1), 70.6(7), 66.4, 55.5. HRMS (ESI) calcd for $C_{19}H_{21}O_3^+$ $[M+H]^+$: 297.1485; found: 297.1463.



2-(2,2-diphenylvinyl)tetrahydro-*2H*-pyran (3l): Isolated yield = 79%. Isomers.

^{1}H NMR (400 MHz, $CDCl_3$) (Major isomer) δ 7.40 – 7.29 (m, 4H), 7.25 – 7.21 (m, 6H), 6.04 (d, *J* = 8.9 Hz, 1H), 4.01 – 3.93 (m, 1H), 3.88 – 3.76 (m, 1H), 3.36 (td, *J* = 11.8, 2.2 Hz, 1H), 1.85 – 1.75 (m, 1H), 1.68 – 1.54 (m, 3H), 1.48 – 1.37 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) (Major isomer) δ 143.7, 142.3, 139.9, 130.1, 130.0, 128.3(4), 128.3(2), 127.9, 127.7, 127.6, 77.6, 77.3, 77.0, 75.9, 68.2, 32.5, 26.0, 23.4. HRMS (ESI) calcd for $C_{19}H_{21}O^+$ $[M+H]^+$: 265.1587; found: 265.1569.



2-(2,2-diphenylvinyl)-1,3-dioxolane (3m-A) & 4-(2,2-diphenylvinyl)-1,3-dioxolane (3m-B): total isolated yield = 97%. **A**/**B** = 3:2.

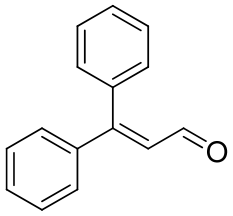
A: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.33 (m, 3H), 7.30 – 7.25 (m, 7H), 6.01 (d, *J* = 7.9 Hz, 1H), 5.21 (d, *J* = 7.9 Hz, 1H), 4.09 – 3.99 (m, 2H), 3.90 – 3.80 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 148.5, 141.6, 138.7, 130.3, 128.4, 128.3(4), 128.2(9), 128.2, 128.1, 124.5,

101.3, 77.6, 77.3, 77.0, 65.4. HRMS (ESI) calcd for $C_{17}H_{17}O_2^+$ $[M+H]^+$: 253.1223; found: 253.1204.

B: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.34 (m, 3H), 7.30 – 7.23 (m, 5H), 7.22 – 7.15 (m, 2H), 6.07 (d, J = 9.0 Hz, 1H), 5.11 (s, 1H), 4.89 (s, 1H), 4.47 (dd, J = 15.8, 6.9 Hz, 1H), 3.99 (t, J = 7.3 Hz, 1H), 3.63 (t, J = 7.5 Hz, 1H).

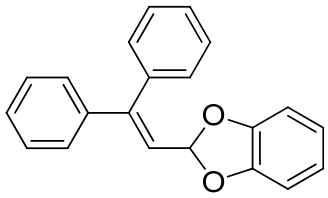
^{13}C NMR (101 MHz, $CDCl_3$) δ 146.9, 141.6, 139.1, 130.1, 128.6, 128.5, 128.2, 128.1, 127.9, 125.6, 95.7, 77.6, 77.3, 77.0, 74.2, 70.3. HRMS (ESI) calcd for $C_{17}H_{17}O_2^+$ $[M+H]^+$: 253.1223; found: 253.1206.



3,3-diphenylacrylaldehyde (3m')

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.53 (d, *J* = 8.0 Hz, 1H), 7.49 – 7.40 (m, 4H), 7.39 – 7.35 (m, 4H), 7.33 – 7.28 (m, 2H), 6.60 (d, *J* = 8.0 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 193.8, 162.5, 140.0, 136.9, 131.0, 130.7, 129.7, 128.9(3), 128.8(8), 128.6, 127.5, 77.6, 77.3, 77.0.

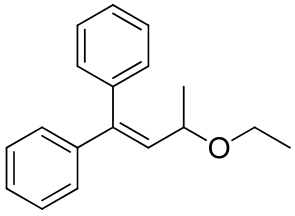


2-(2,2-diphenylvinyl)benzo[*d*][1,3]dioxole (3n): Isolated yield = 89%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.37 (m, 3H), 7.35 – 7.32 (m, 2H), 7.30 (s, 5H), 6.79 (s, 4H), 6.41 (d, *J* = 8.3 Hz, 1H), 6.27 (d, *J* = 8.3 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 150.2, 147.9, 140.9, 138.0, 130.3, 128.9, 128.7, 128.5(8), 128.5(7), 128.4,

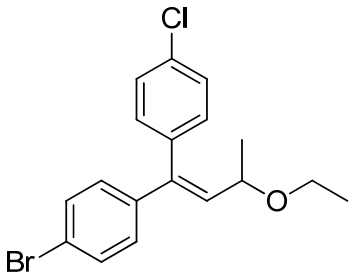
121.7(3), 121.6(9), 108.7, 108.2, 77.6, 77.3, 77.0. HRMS (EI^+) calcd for $C_{21}H_{16}O_2^+$ (M^+): 300.1150; found: 300.1151.



(3-ethoxybut-1-ene-1,1-diyl)dibenzene (3o): Isolated yield = 90%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.30 (m, 3H), 7.28 – 7.23 (m, 5H), 7.18 – 7.13 (m, 2H), 6.03 (d, *J* = 9.2 Hz, 1H), 3.96 (dq, *J* = 9.2, 6.3 Hz, 1H), 3.53 (dq, *J* = 9.1, 7.0 Hz, 1H), 3.22 (dq, *J* = 9.1, 7.0 Hz, 1H), 1.30 (d, *J* = 6.3 Hz, 3H), 1.13 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 143.4, 141.8, 139.8, 131.9, 129.9, 128.5, 128.4, 127.7, 127.5(2), 127.4(8), 77.6, 77.3, 77.0, 72.6, 63.7, 22.0, 15.8. HRMS (EI^+) calcd for $C_{18}H_{20}O^+$ (M^+): 252.1514; found: 252.1516.

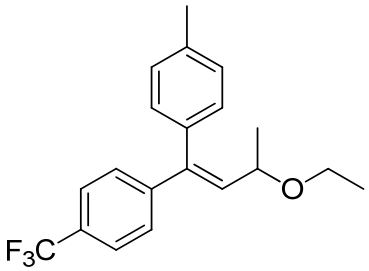


(*E,Z*)-1-bromo-4-(1-(4-chlorophenyl)-3-ethoxybut-1-en-1-yl)benzene (3p): Isolated yield = 87%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.49 (m, 1H), 7.41 – 7.34 (m, 2H), 7.27 – 7.21 (m, 1H), 7.17 – 7.12 (m, 1H), 7.11 – 7.06 (m, 2H), 7.05 – 6.99 (m, 1H), 6.02 (dd, *J* = 9.2, 2.4 Hz, 1H), 3.91 (dq, *J* = 9.2, 6.3 Hz, 1H), 3.47 (dq, *J* = 9.1, 7.0 Hz, 1H), 3.23 (dq, *J* = 9.1, 7.0 Hz, 1H), 1.29 (d, *J* = 6.3 Hz, 3H), 1.13 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.2(5), 141.2(0), 140.3, 139.8, 138.2, 137.7, 133.8(4), 133.8(1), 133.0,

132.9, 131.9, 131.6, 131.5, 131.2, 129.0, 128.9, 128.7(2), 128.6(7), 122.0, 121.9, 77.6, 77.3, 77.0, 72.5, 63.8, 21.7(4), 21.7(1), 15.7. HRMS (EI^+) calcd for $C_{18}H_{18}BrClO^+$ (M^+): 364.0230; found: 364.0229.



(*E,Z*)-1-(3-ethoxy-1-(4-(trifluoromethyl)phenyl)but-1-en-1-yl)-4-methylbenzene (3q): Isolated yield = 85%. E/Z isomers. E/Z = 3:2.

^1H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.0 Hz, 1H), 7.42 (d, *J* = 8.1 Hz, 1H), 7.27 (d, *J* = 8.1 Hz, 1H), 7.20 (d, *J* = 7.9 Hz, 1H), 7.12 (d, *J* = 7.8 Hz, 1H), 7.01 (s, 2H), 6.94 (d, *J* = 7.8 Hz, 1H), 5.98 (dd, *J* = 9.2, 4.9 Hz, 1H), 3.96 – 3.87 (m, 0.4H), 3.84 – 3.73 (m, 0.6H), 3.47 – 3.36 (m, 1H), 3.22 – 3.06 (m, 1H), 2.30 (s, 1.2H), 2.24 (s, 1.8H), 1.21 (dd, *J* = 5.8, 4.6 Hz, 3H), 1.05 (td, *J* = 7.0, 2.1 Hz, 3H).

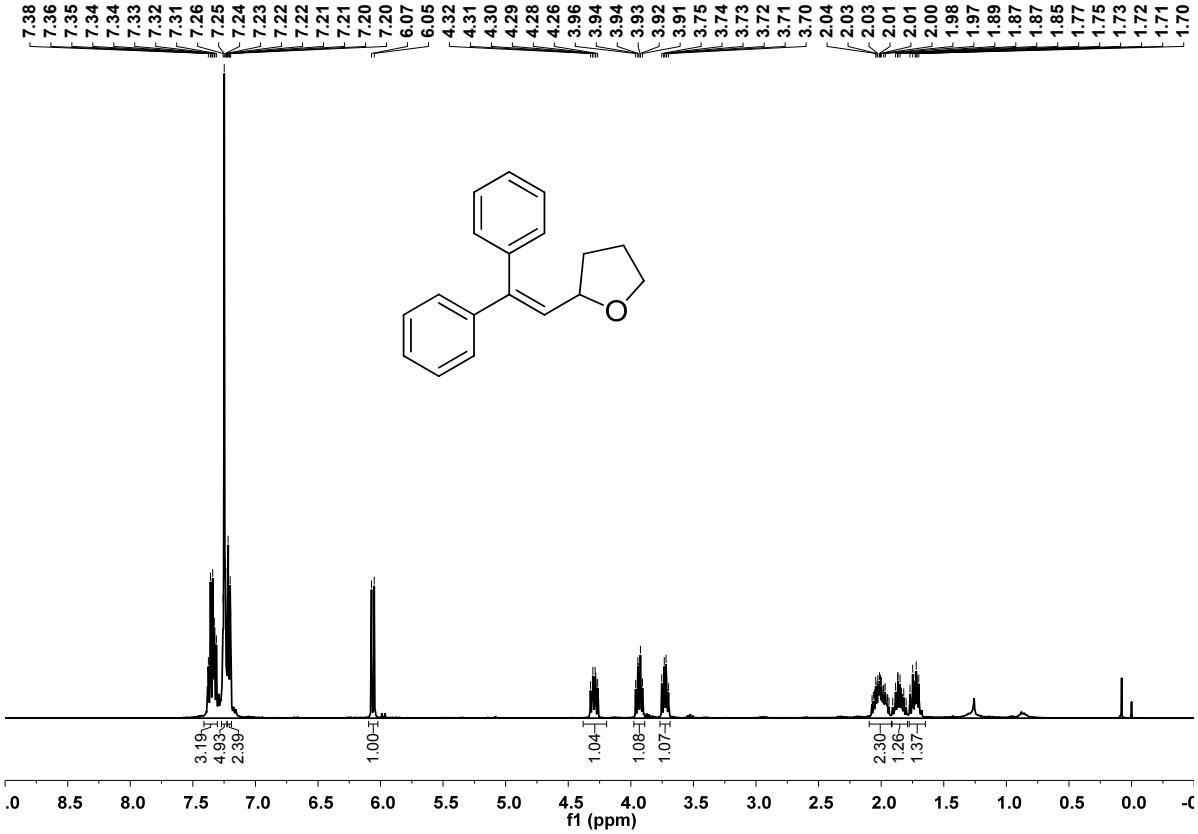
^{13}C NMR (101 MHz, $CDCl_3$) δ 145.6, 143.9, 142.3, 142.1, 138.3, 138.0, 137.7, 136.0, 133.9, 132.1, 130.3, 129.7, 129.4, 129.3, 127.8, 127.3, 125.5(4), 125.5(0), 125.4, 125.3, 123.1(4), 123.1(2), 77.6, 77.3, 77.0, 72.6(2), 72.6(0), 63.8, 63.7, 21.9, 21.8, 21.5, 21.3, 15.7. HRMS (EI^+) calcd for $C_{20}H_{21}F_3O^+$ (M^+): 334.1544; found: 334.1541.

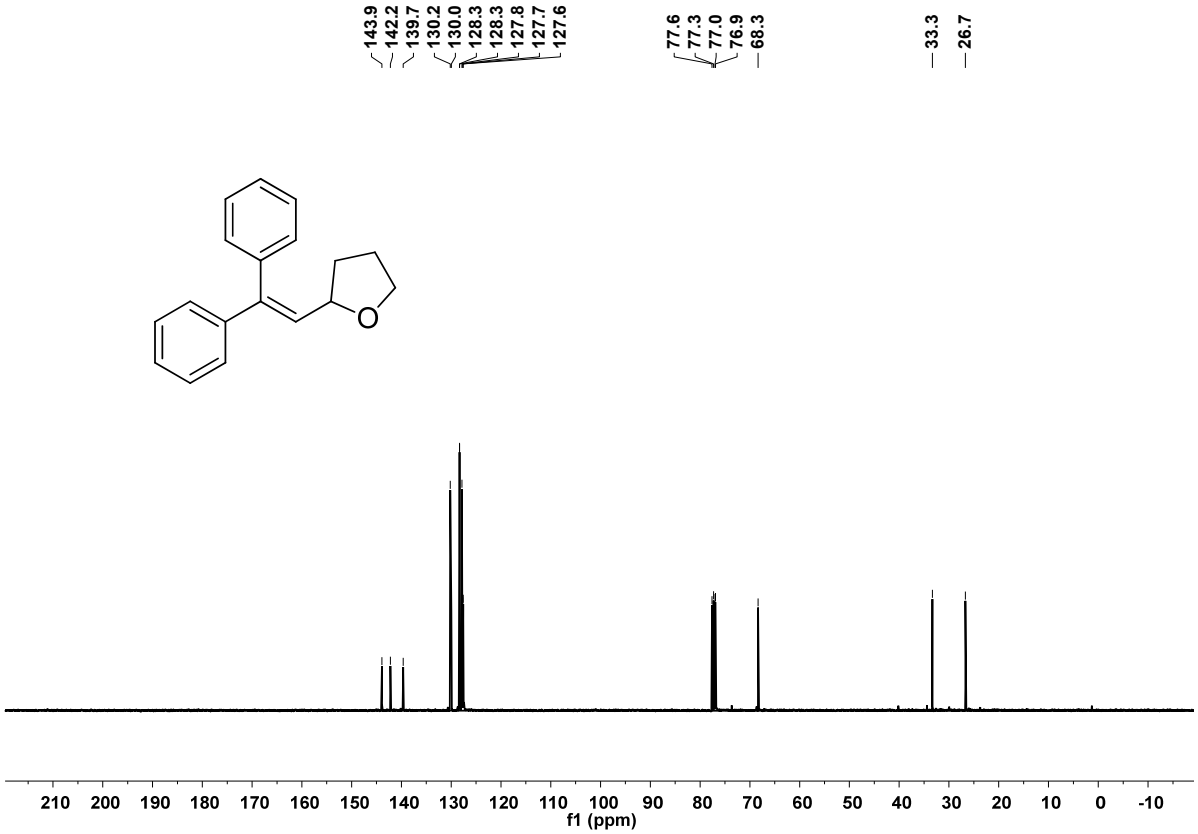
References:

1. T. Wang, Y. Hu and S. Zhang, *Org. Biomol. Chem.*, 2010, **8**, 2312.
2. S. Pan, J. Liu, H. Li, Z. Wang, X. Guo and Z. Li, *Org. Lett.*, 2010, **12**, 1932.
3. Y.-J. Jang, Y.-K. Shih, J.-Y. Liu, W.-Y. Kuo and C.-F. Yao, *Chem. Eur. J.*, 2003, **9**, 2123.

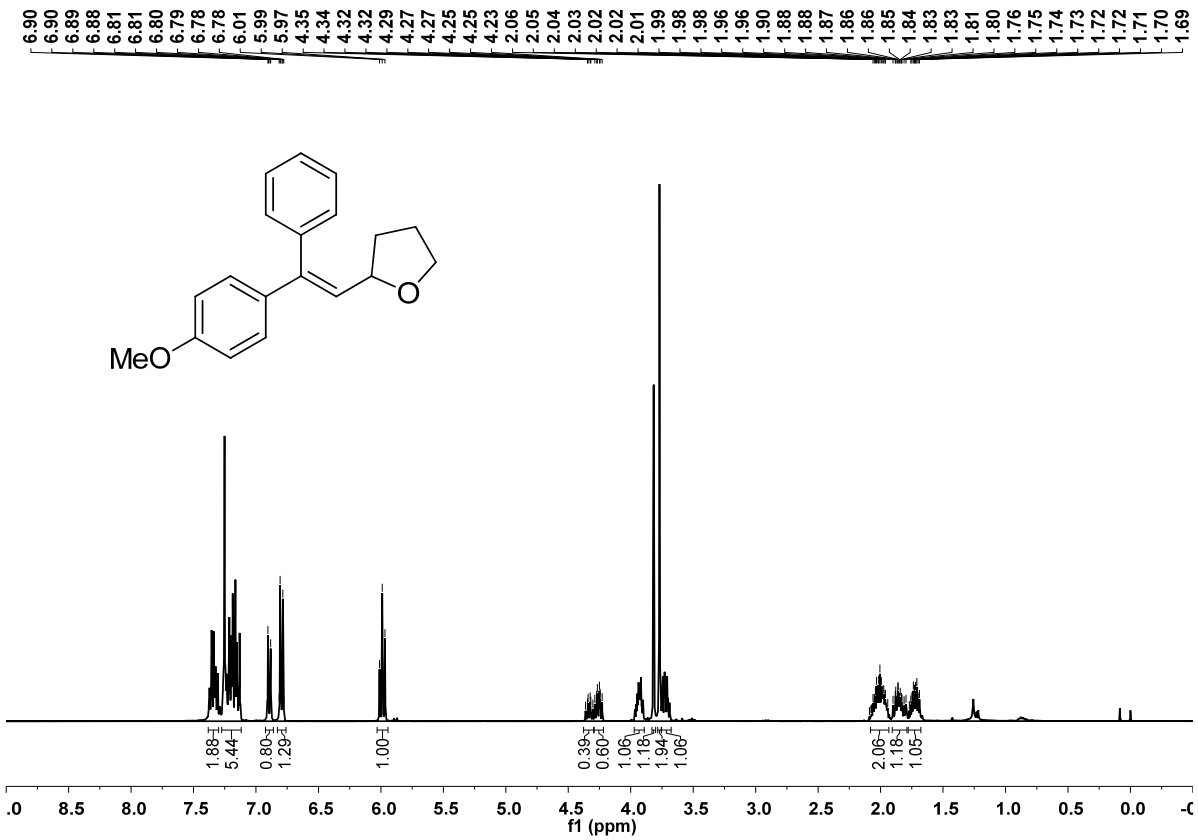
Copies of product ^{1}H NMR and ^{13}C NMR

3a





3b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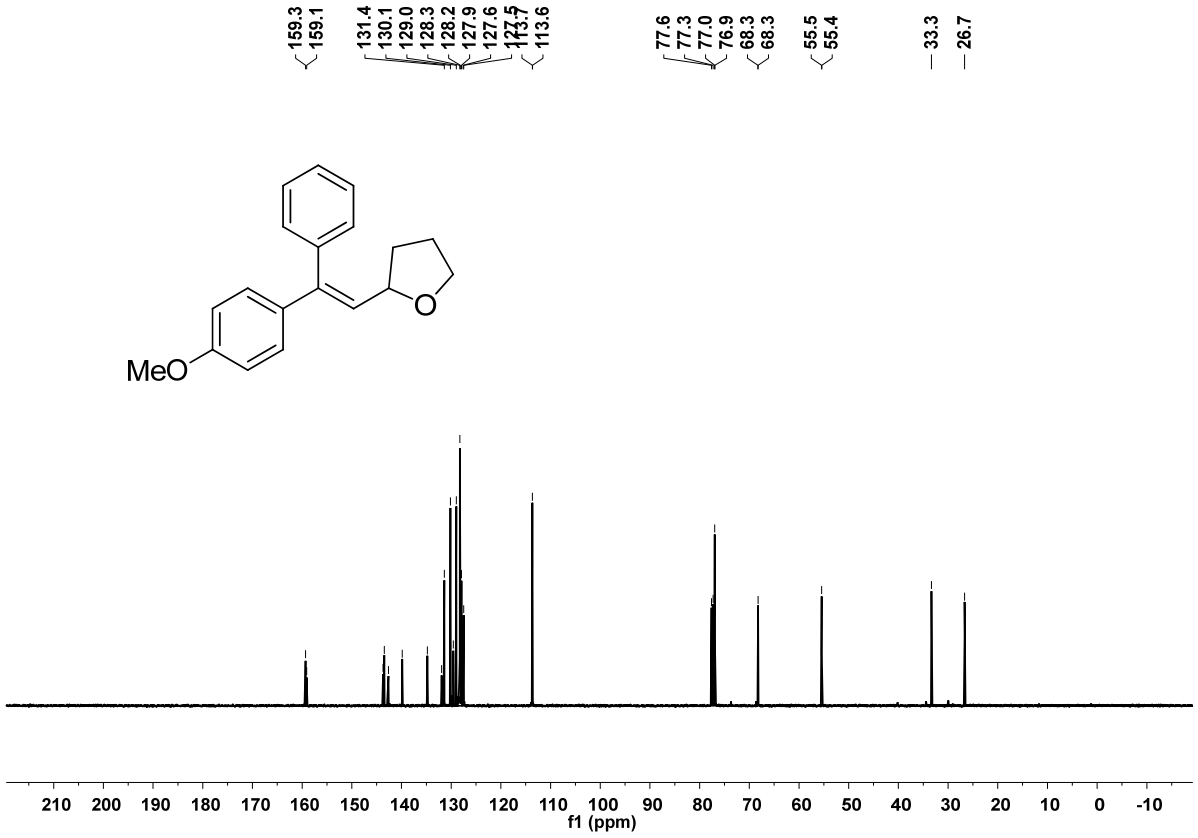
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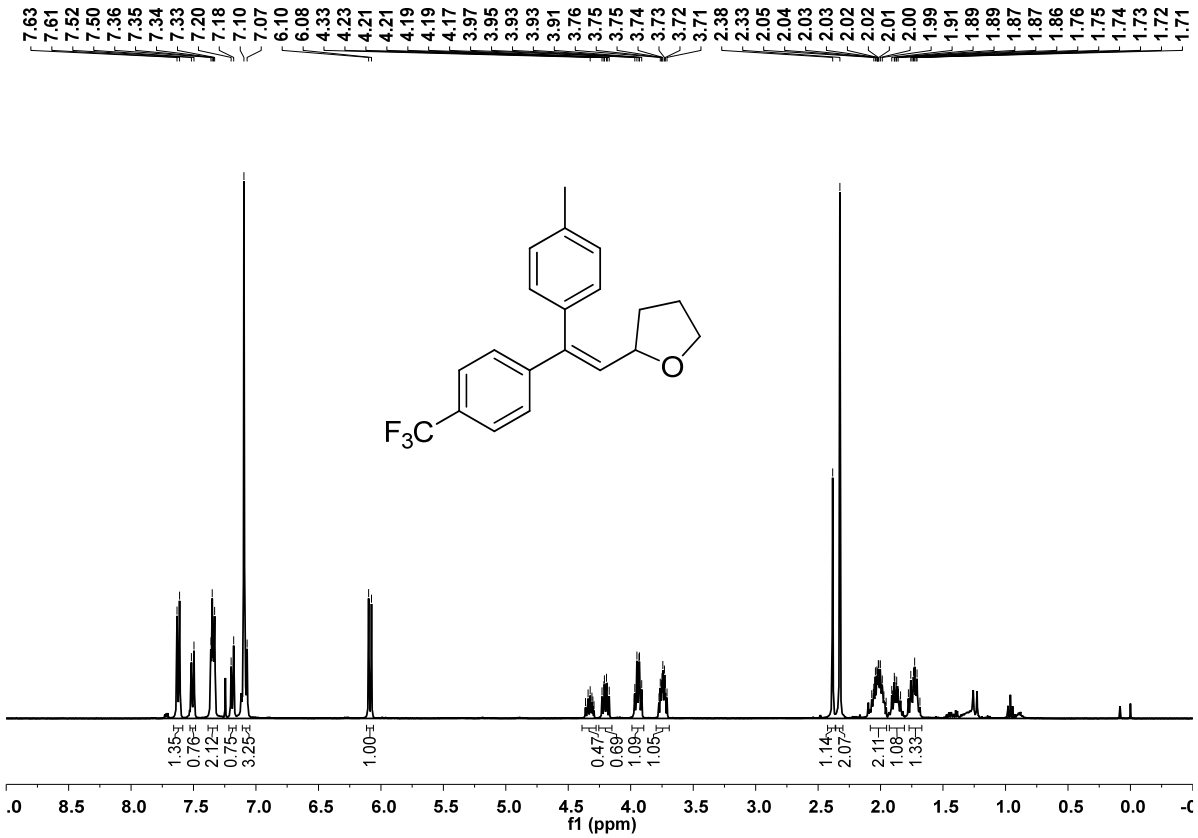
f1 (ppm)

3c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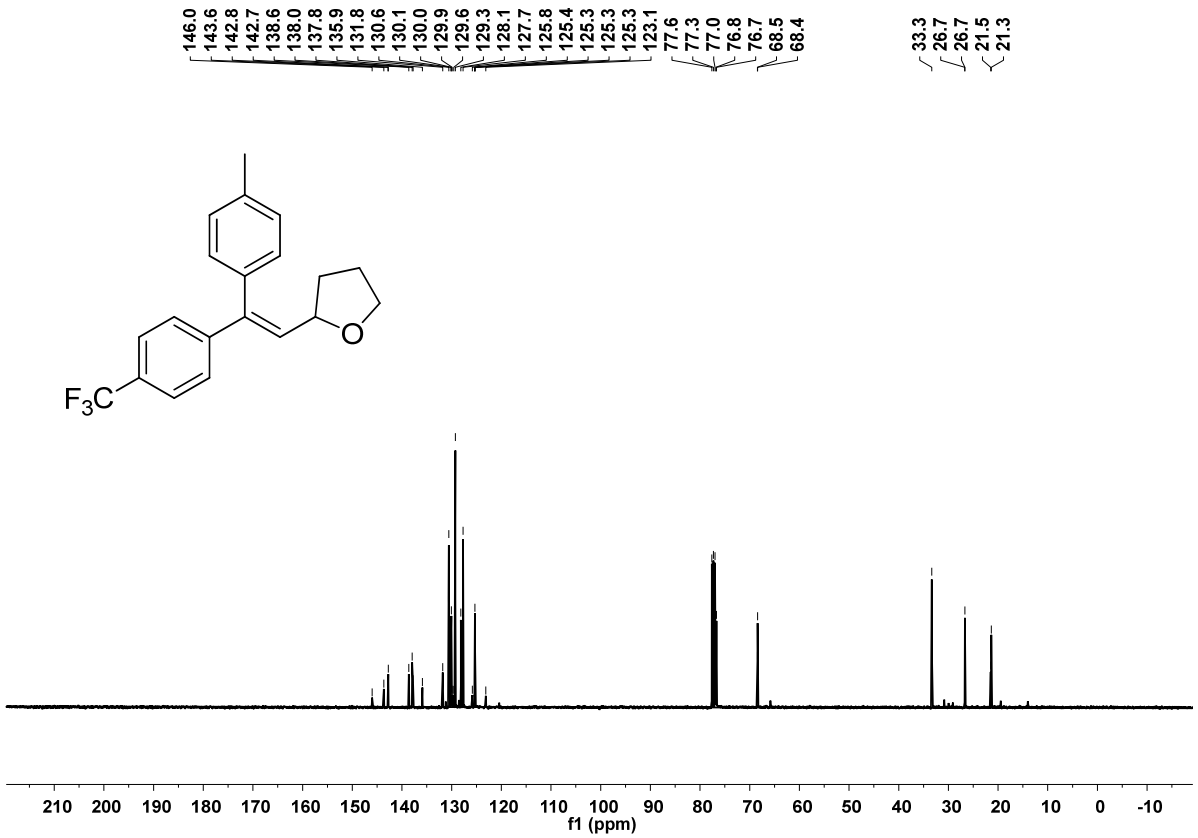
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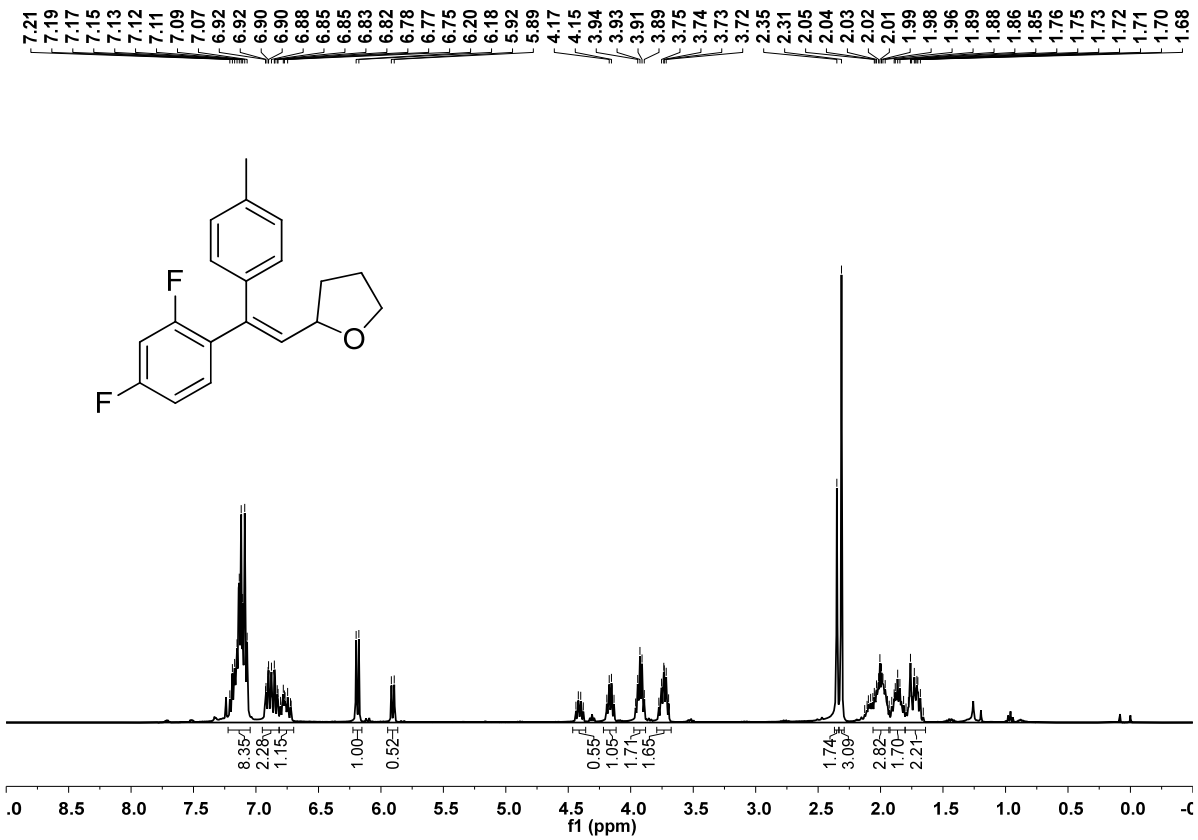
F3C
O
f1 (ppm)

3d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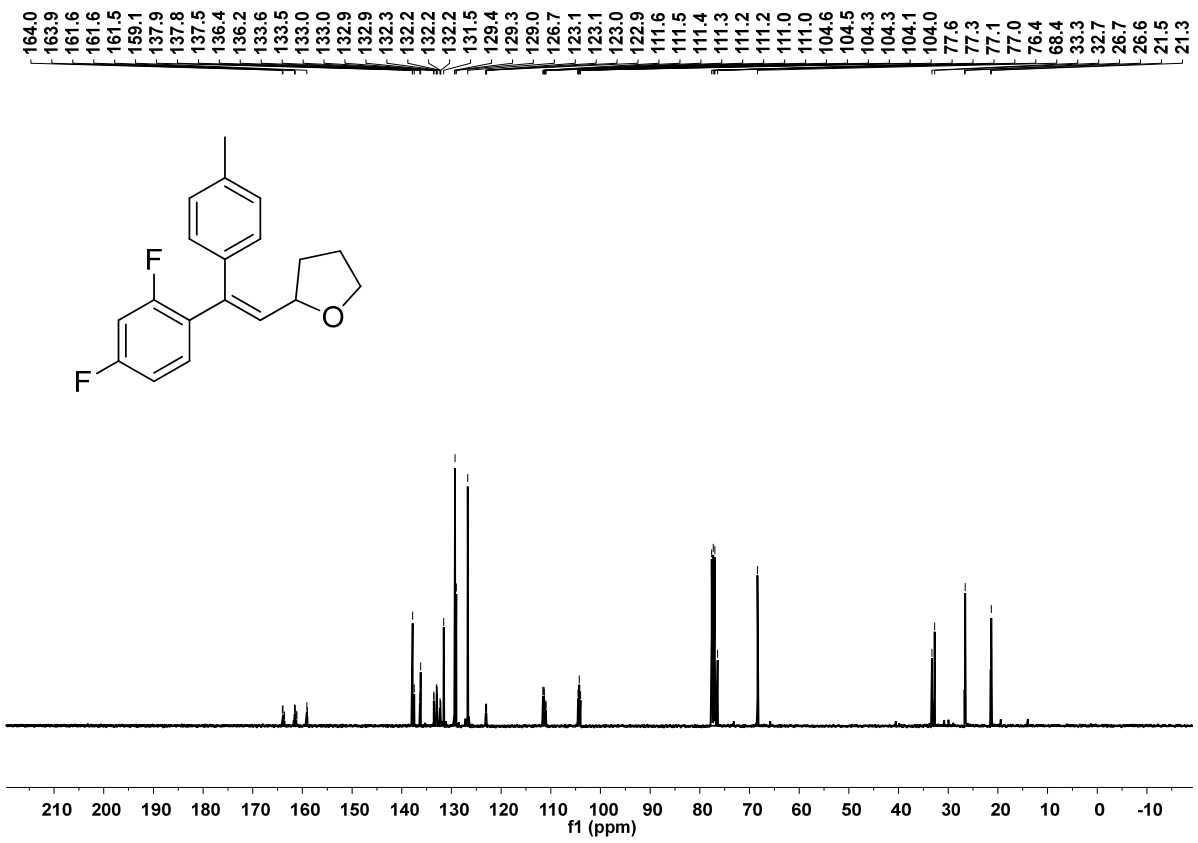
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F
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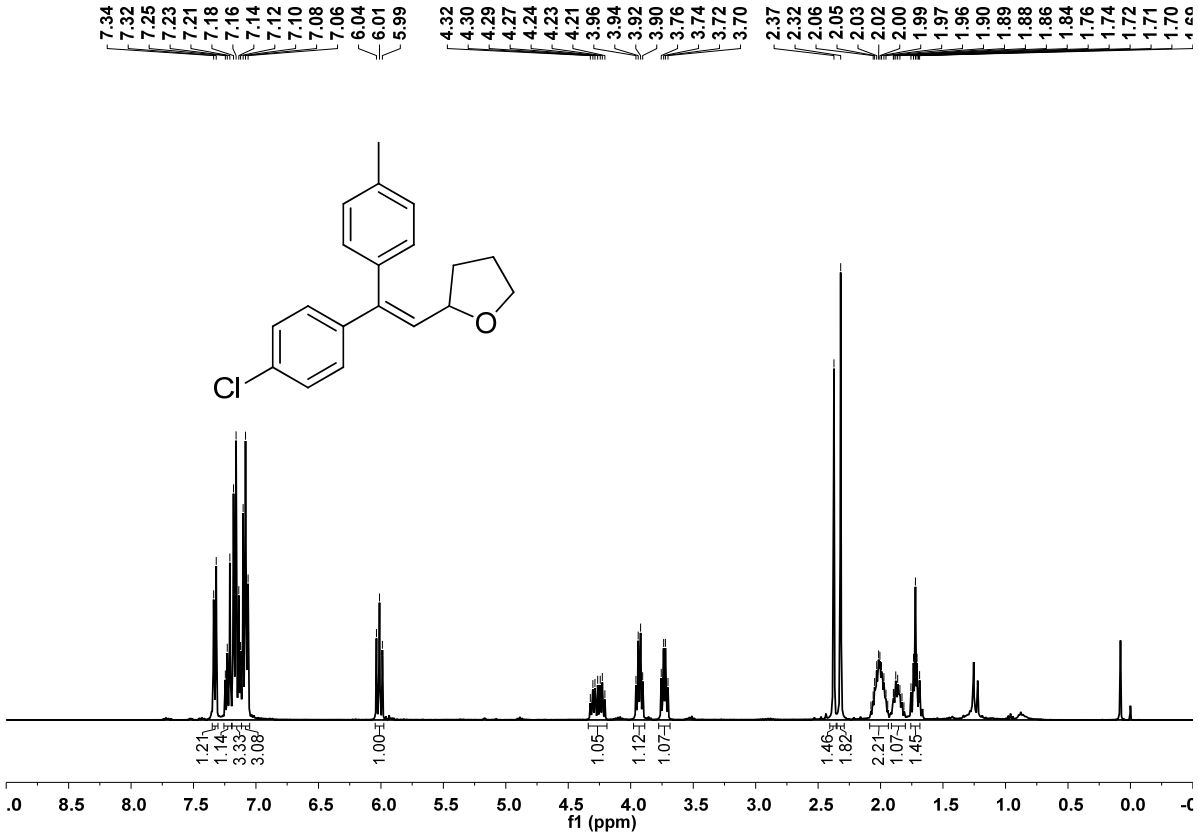
f1 (ppm)

3e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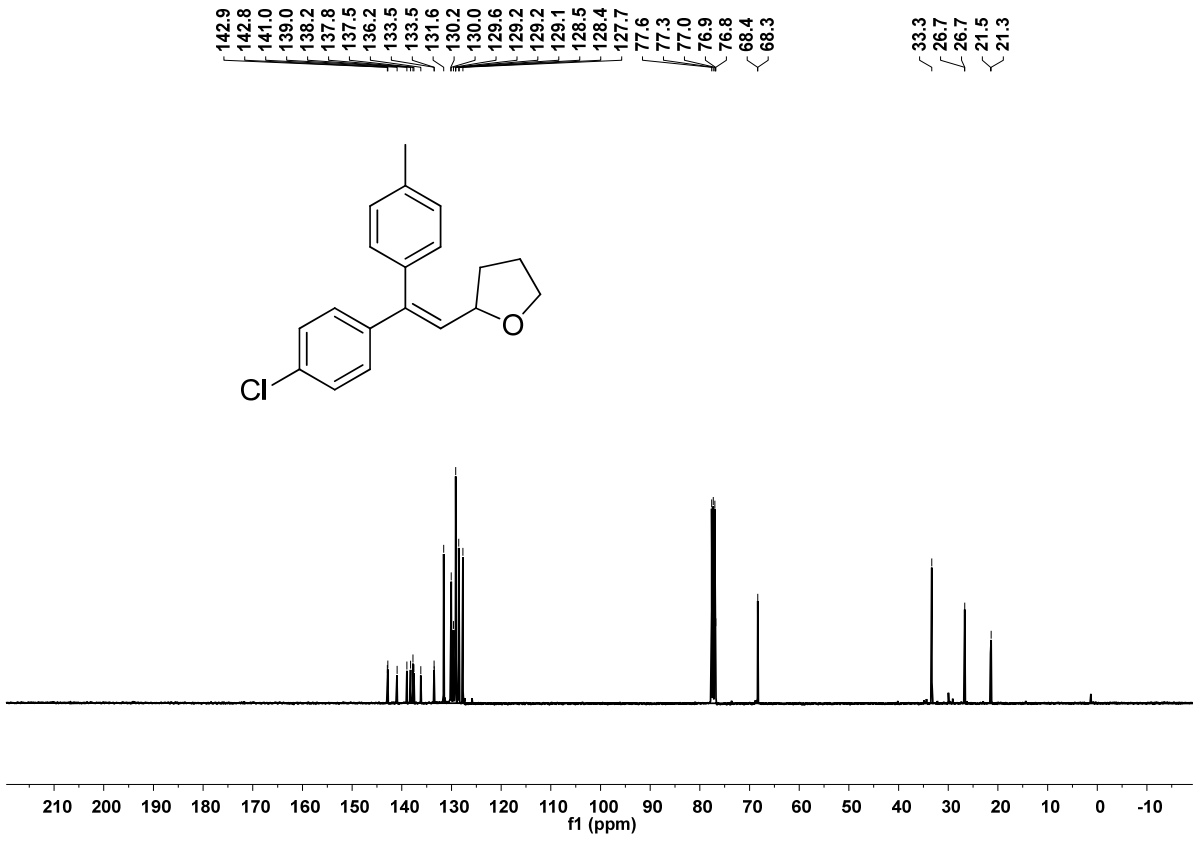
3.74
3.72
3.70
2.37
2.32
2.06
2.05
2.03
2.02
2.00
1.99
1.97
1.96
1.90
1.89
1.88
1.86
1.84
1.76
1.74
1.72
1.71
1.70
Cl
O
1.21
1.14
3.33
3.08
1.00
1.05
1.12
1.07
1.46
1.82
2.21
1.07
1.45
f1 (ppm)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139.0
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137.8
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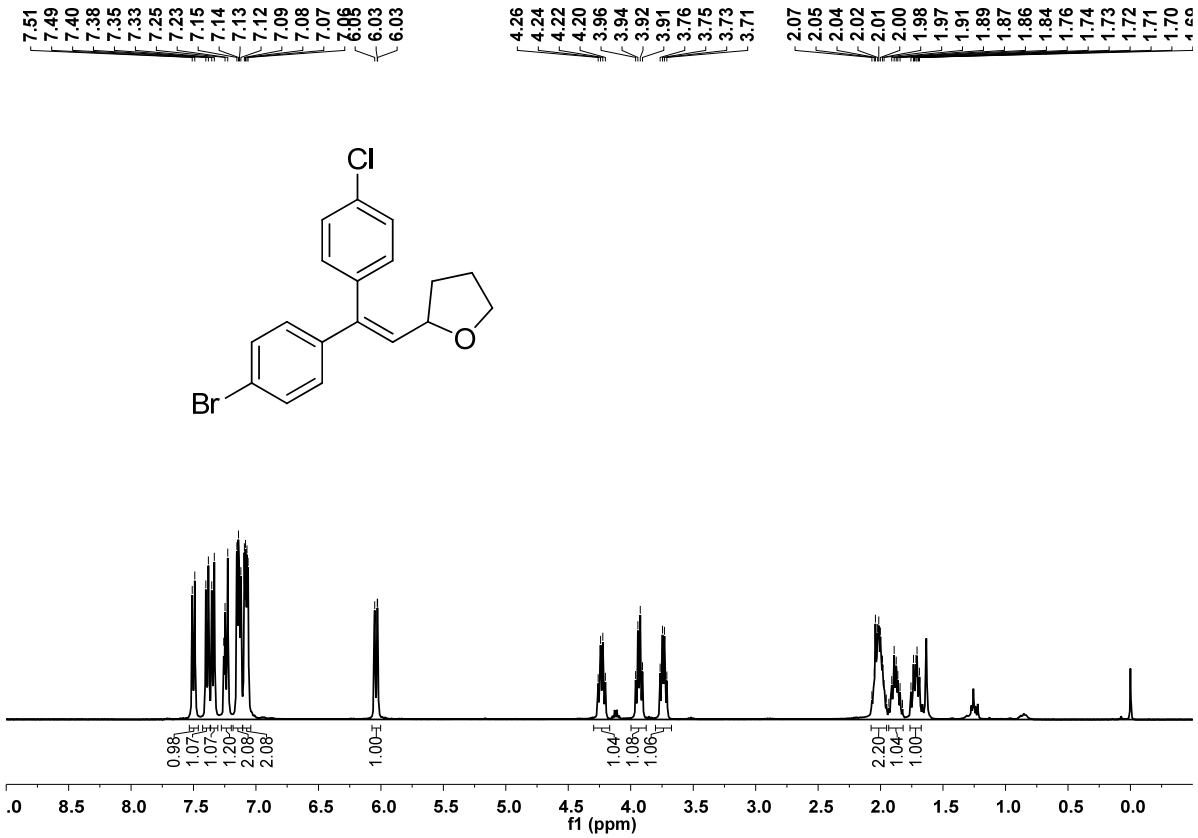
133.5
131.6
130.2
130.0
129.6
129.2
129.2
129.1
128.5
128.4
127.7
77.6
77.3
77.0
76.9
76.8
68.4
68.3
33.3
26.7
26.7
21.5
21.3
Cl
O
f1 (ppm)

3f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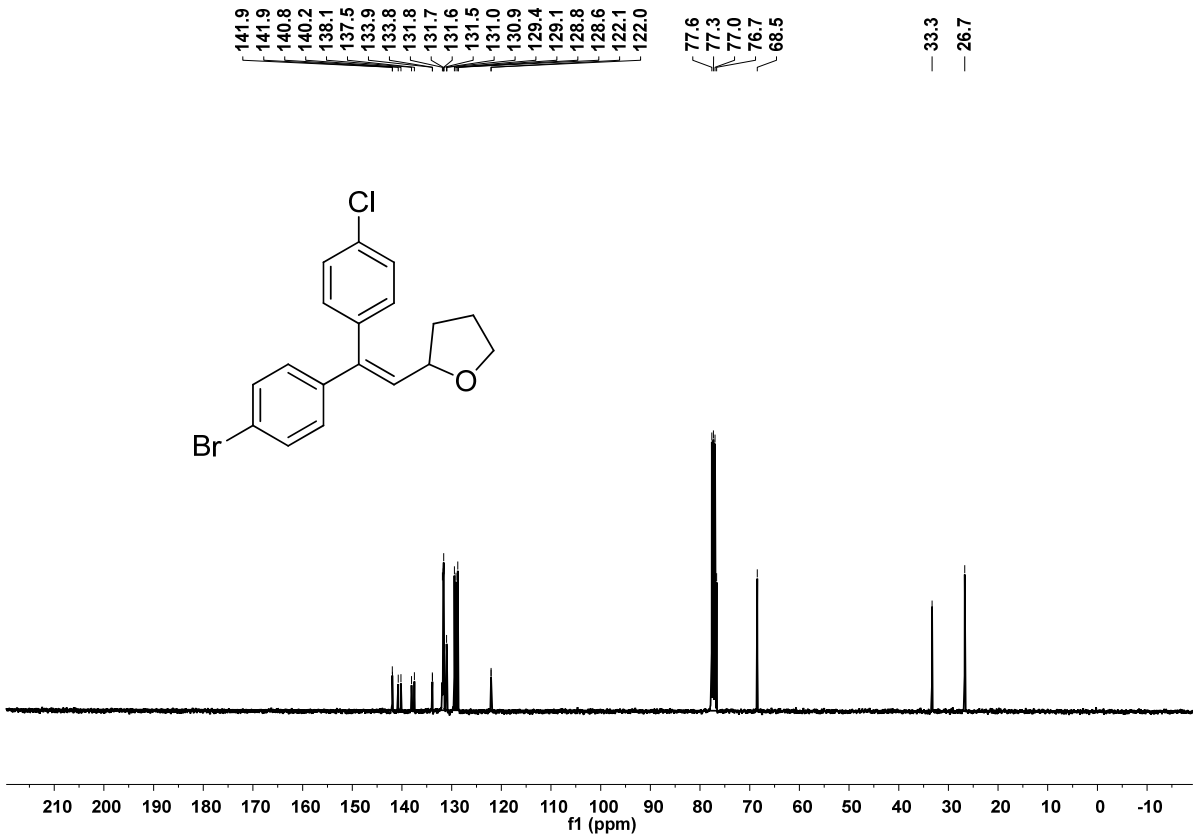
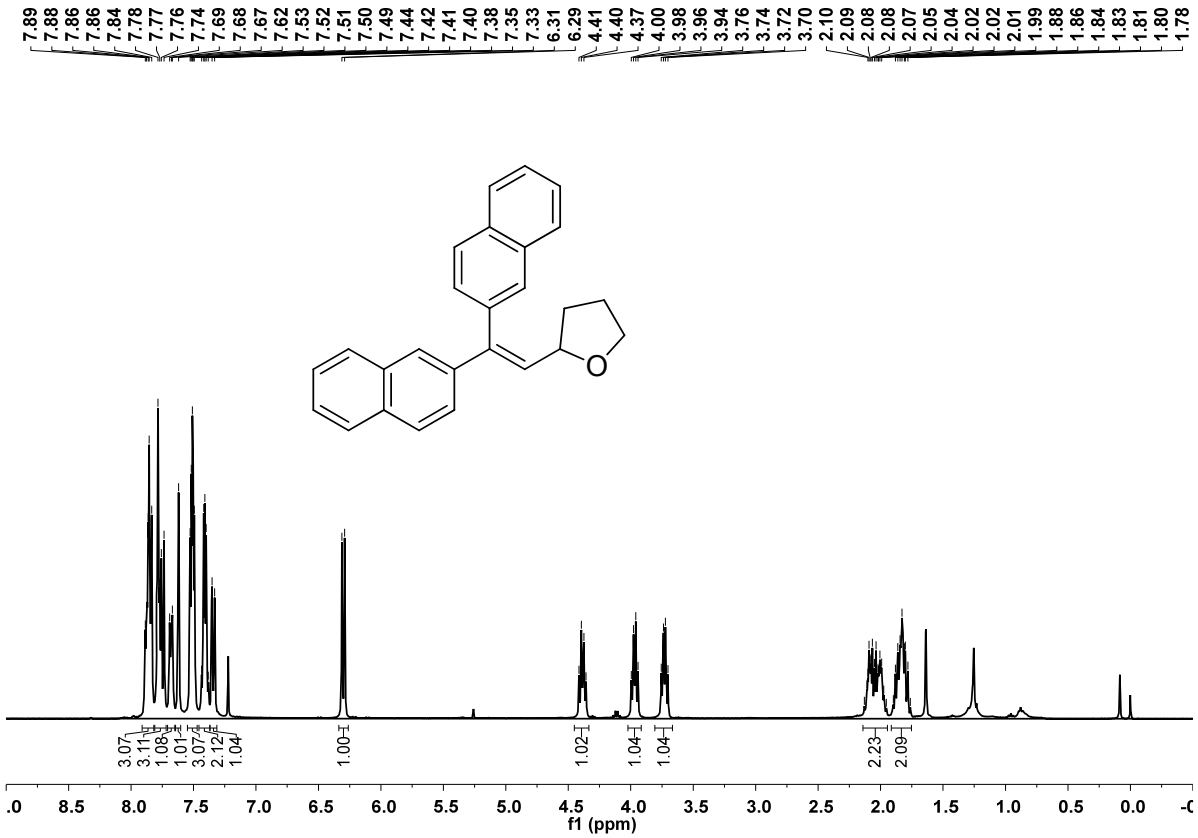
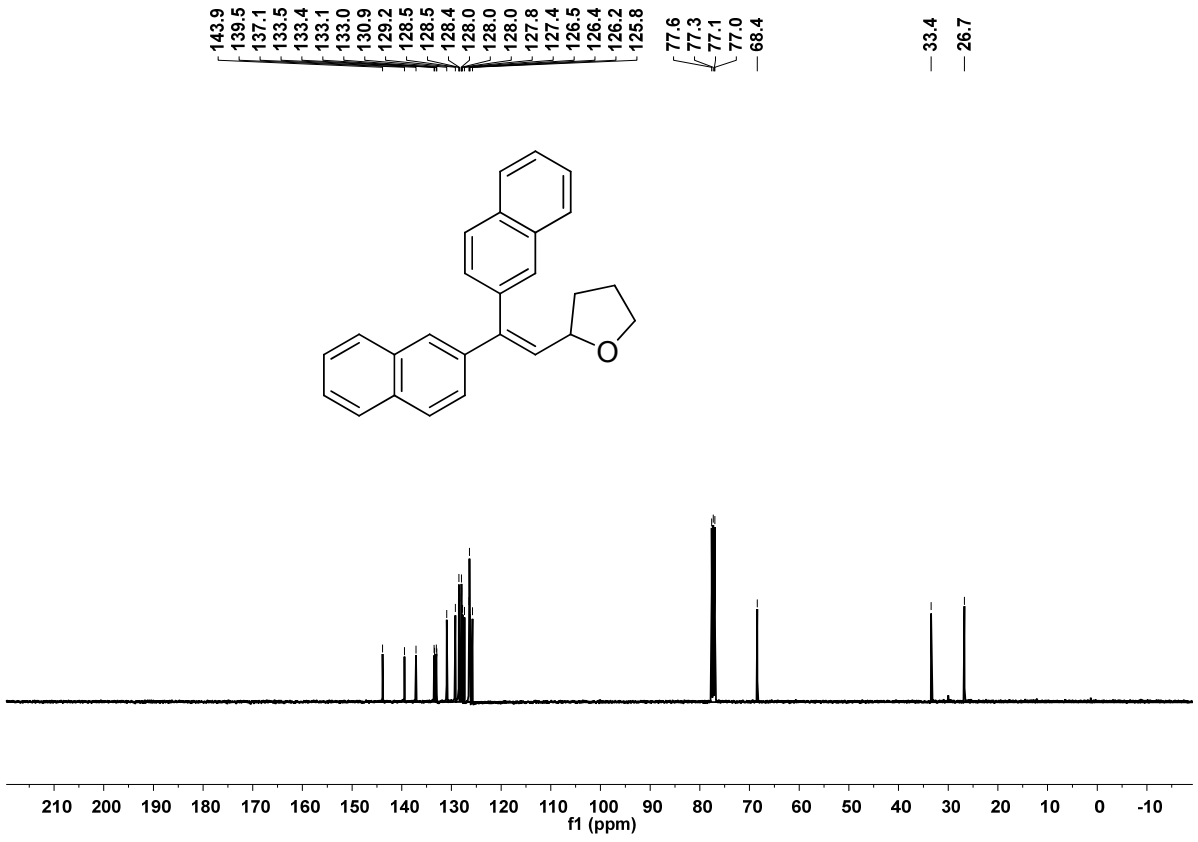
7.23
7.15
7.14
7.13
7.12
7.09
7.08
7.07
6.03
6.03
4.26
4.24
4.22
4.20
3.96
3.94
3.92
3.91
3.76
3.75
3.73
3.71
2.07
2.05
2.04
2.02
2.01
2.00
1.98
1.97
1.91
1.89
1.87
1.86
1.84
1.76
1.74
1.73
1.72
1.71
1.70
Cl
O
Br
0.98
1.07
1.07
1.20
2.08
2.08
1.00
1.04
1.08
1.06
2.20
1.04
1.00
f1 (ppm)

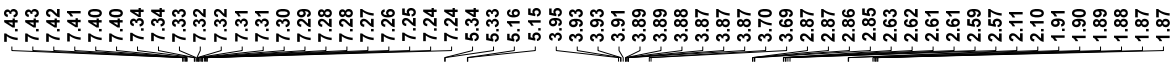

141.9
141.9
140.8
140.2
138.1
137.5
133.9
133.8
131.8
131.7
131.6
131.5
131.0
130.9
129.4
129.1
128.8
128.6
122.1
122.0
77.6
77.3
77.0
76.7
68.5
33.3
26.7
Cl
O
Br
f1 (ppm)

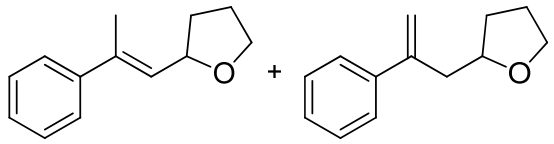
3g


7.89 7.88 7.86 7.86 7.84 7.78 7.77 7.76 7.74 7.69 7.68 7.67 7.62 7.53 7.52 7.51 7.50 7.49 7.44 7.42 7.41 7.40 7.38 7.35 7.33 6.31 6.29
4.41 4.40 4.37 4.00 3.98 3.96 3.94 3.76 3.74 3.72 3.70
2.10 2.09 2.08 2.08 2.07 2.05 2.04 2.02 2.02 2.01 1.99 1.88 1.86 1.84 1.83 1.81 1.80 1.78
O
3.07 3.11 1.08 1.01 3.07 2.12 1.04
1.00
1.02 1.04 1.04
2.23 2.09
f1 (ppm)

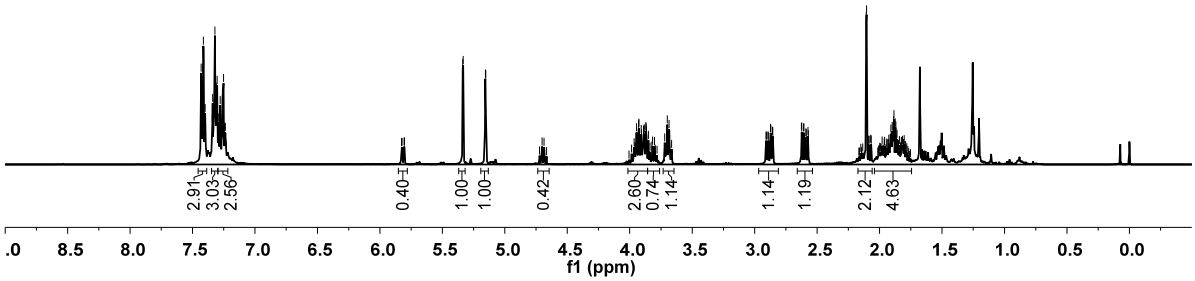

143.9 139.5 137.1 133.5 133.4 133.1 133.0 130.9 129.2 128.5 128.5 128.4 128.0 128.0 128.0 127.8 127.4 126.5 126.4 126.2 125.8
77.6 77.3 77.1 77.0 68.4
33.4 26.7
O
f1 (ppm)

3h

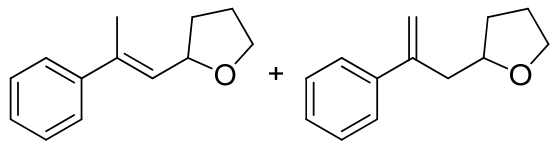

7.43 7.43 7.42 7.41 7.40 7.40 7.34 7.34 7.33 7.32 7.32 7.31 7.31 7.30 7.29 7.28 7.28 7.27 7.26 7.25 7.24 7.24 5.34 5.33 5.16 5.15
3.95 3.93 3.93 3.91 3.89 3.89 3.88 3.87 3.87 3.87 3.70 3.69 2.87 2.87 2.86 2.85 2.63 2.62 2.61 2.61 2.59 2.57 2.11 2.10 1.91 1.90 1.89 1.88 1.87 1.87


O
+
O

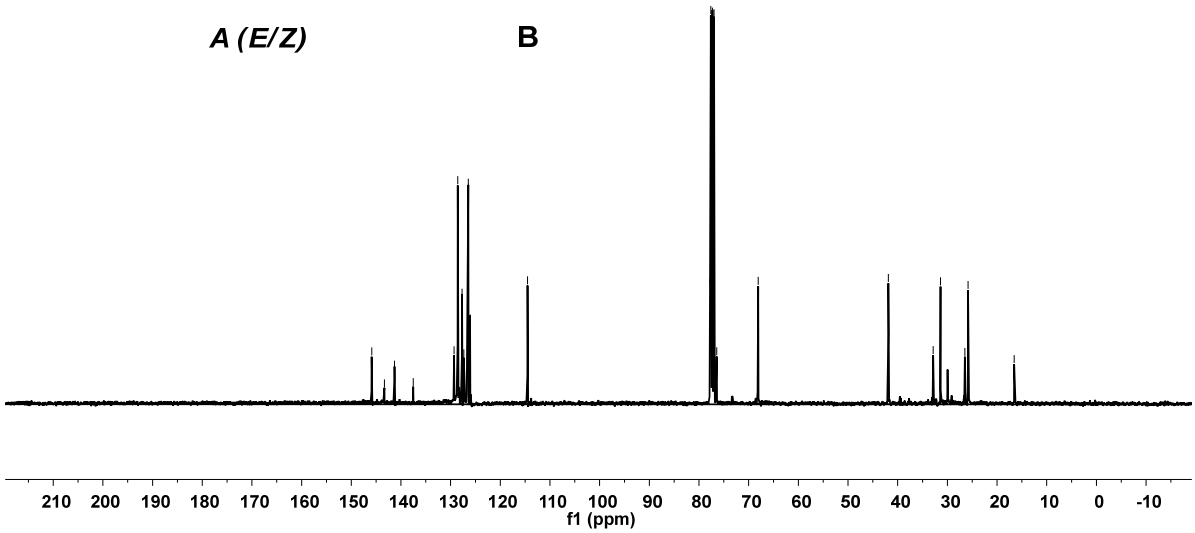
A (E/Z) **B**


2.91
3.03
2.56
0.40
1.00
1.00
0.42
2.60
0.74
1.14
1.14
1.19
2.12
4.63
.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

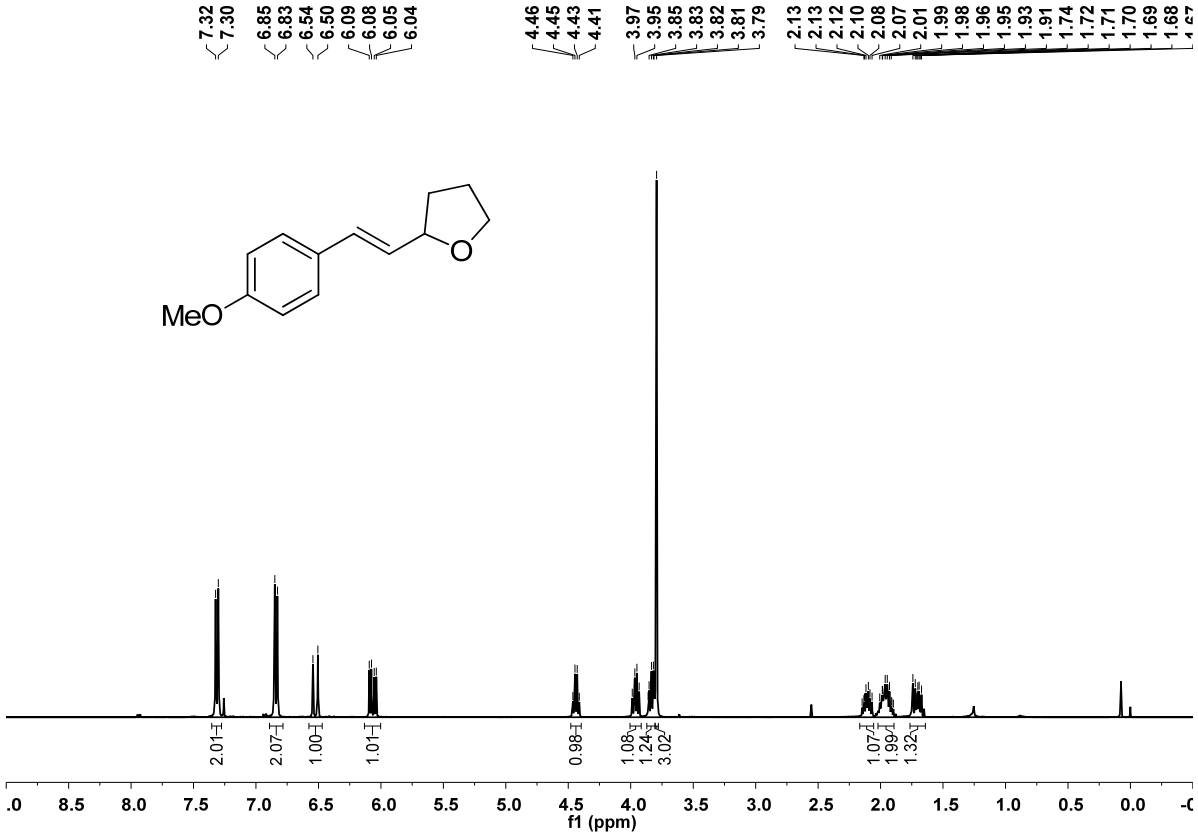

145.9 143.3 141.3 137.5 129.3 128.5 128.4 127.7 127.3 126.4 126.1 114.5
77.8 77.6 77.3 77.0 76.4 68.2 68.1
41.9 32.8 31.4 26.5 25.8 16.6

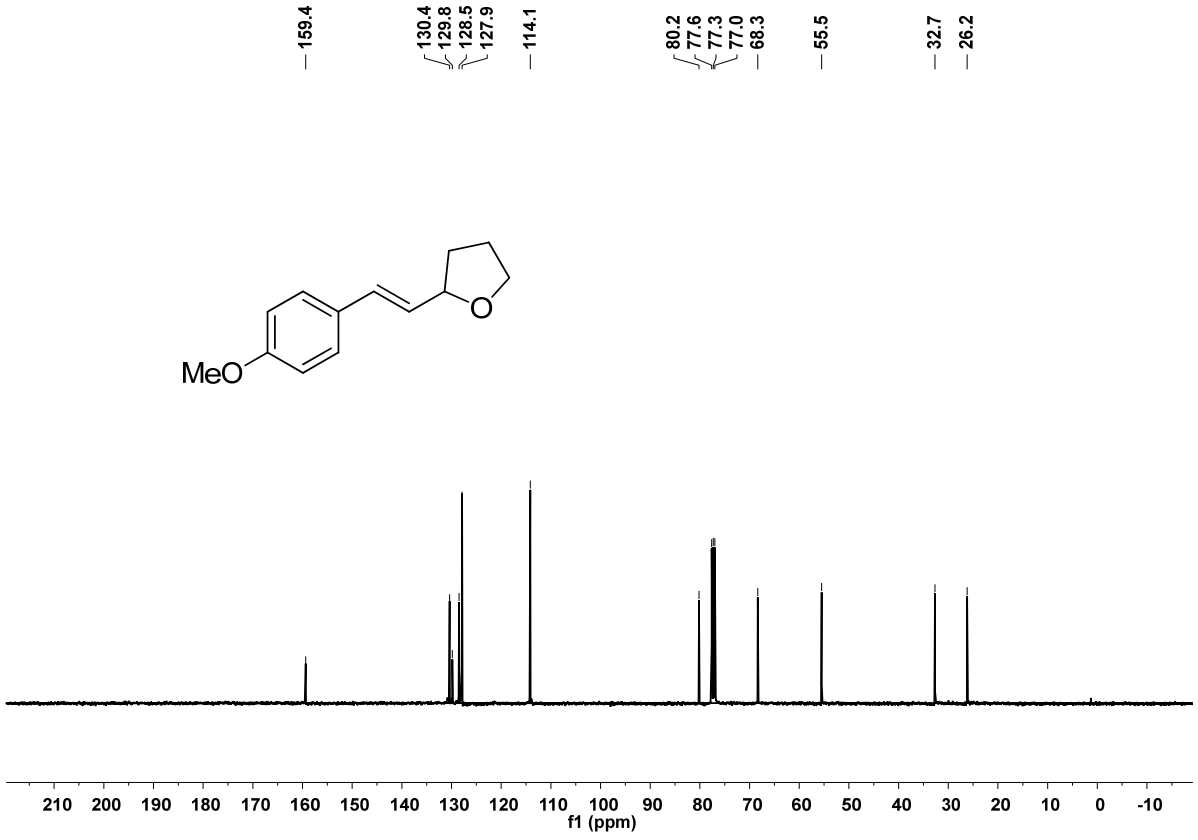

O
+
O

A (E/Z) **B**

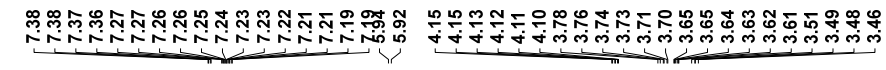

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3i


7.32
7.30
6.85
6.83
6.54
6.50
6.09
6.08
6.05
6.04
4.46
4.45
4.43
4.41
3.97
3.95
3.85
3.83
3.82
3.81
3.79
2.13
2.12
2.10
2.08
2.07
2.01
1.99
1.98
1.96
1.95
1.93
1.91
1.74
1.72
1.71
1.70
1.69
1.68
MeO
O
2.01
2.07
1.00
1.01
0.98
1.08
1.24
3.02
1.07
1.99
1.32
f1 (ppm)


159.4
130.4
129.8
128.5
127.9
114.1
80.2
77.6
77.3
77.0
68.3
55.5
32.7
26.2
MeO
O
f1 (ppm)

3j


7.38
7.38
7.37
7.36
7.27
7.27
7.26
7.26
7.25
7.24
7.23
7.23
7.22
7.21
7.21
7.19
5.94
5.92
4.15
4.15
4.13
4.12
4.11
4.10
3.78
3.76
3.74
3.73
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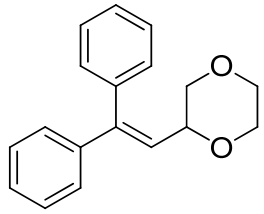
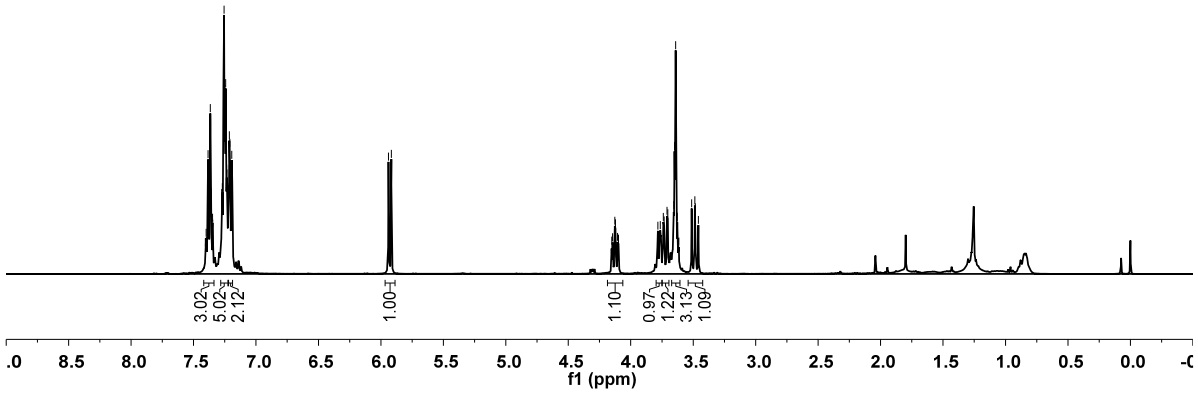
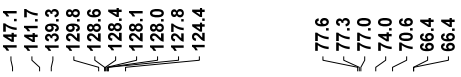
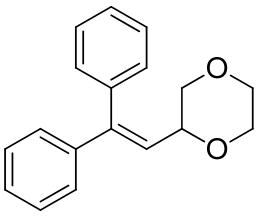
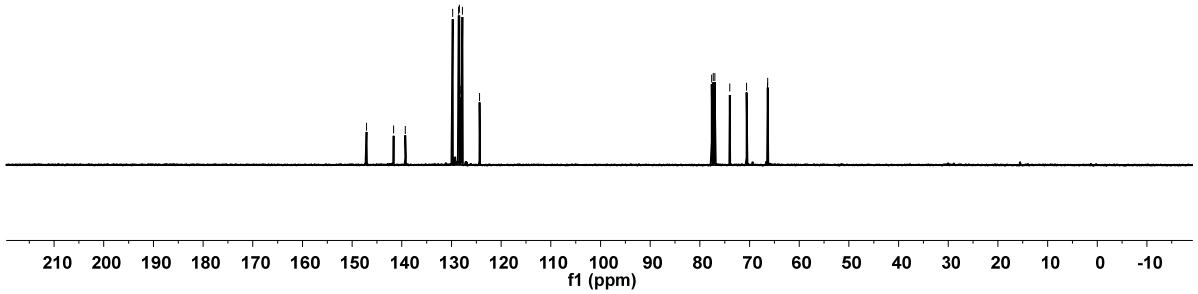
3.62
3.61
3.51
3.49
3.48
3.46


O
O

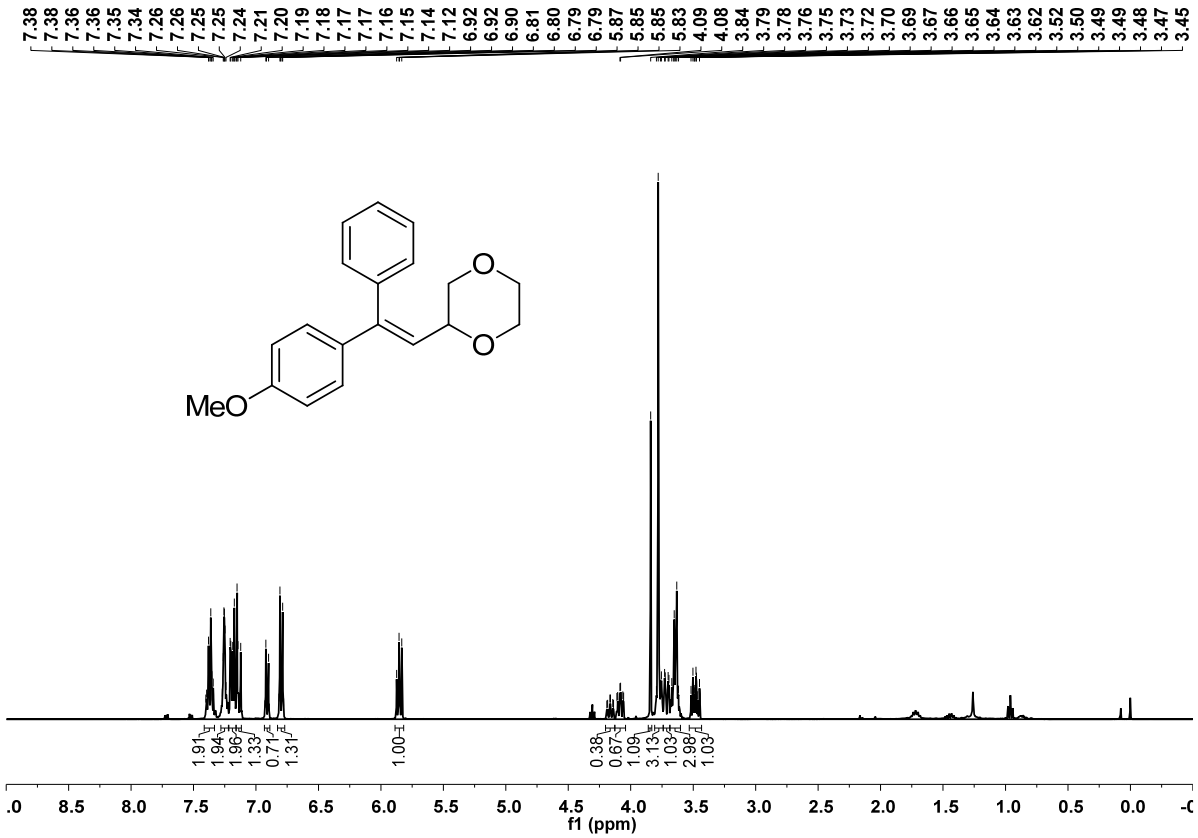

3.02
5.02
2.12
1.00
1.10
0.97
1.22
3.13
1.09
.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

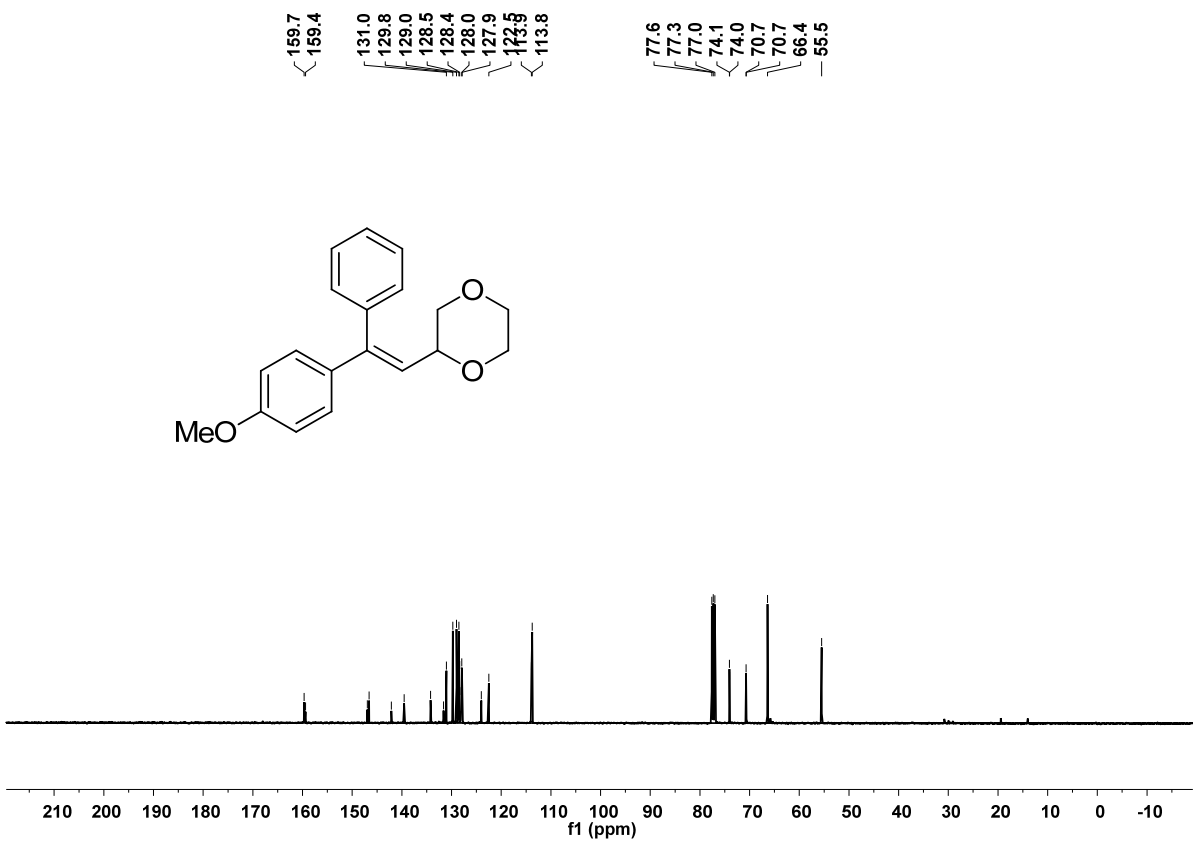

147.1
141.7
139.3
129.8
128.6
128.4
128.1
128.0
127.8
124.4
77.6
77.3
77.0
74.0
70.6
66.4
66.4


O
O

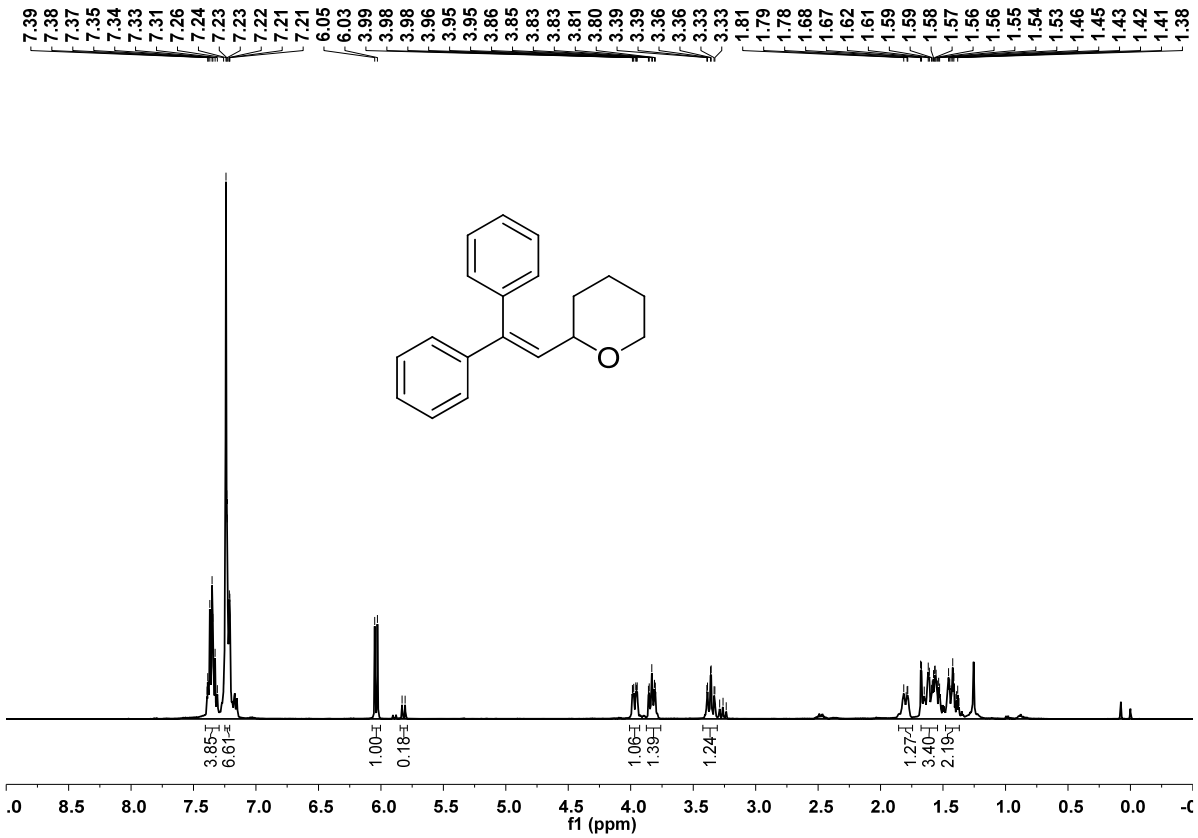

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

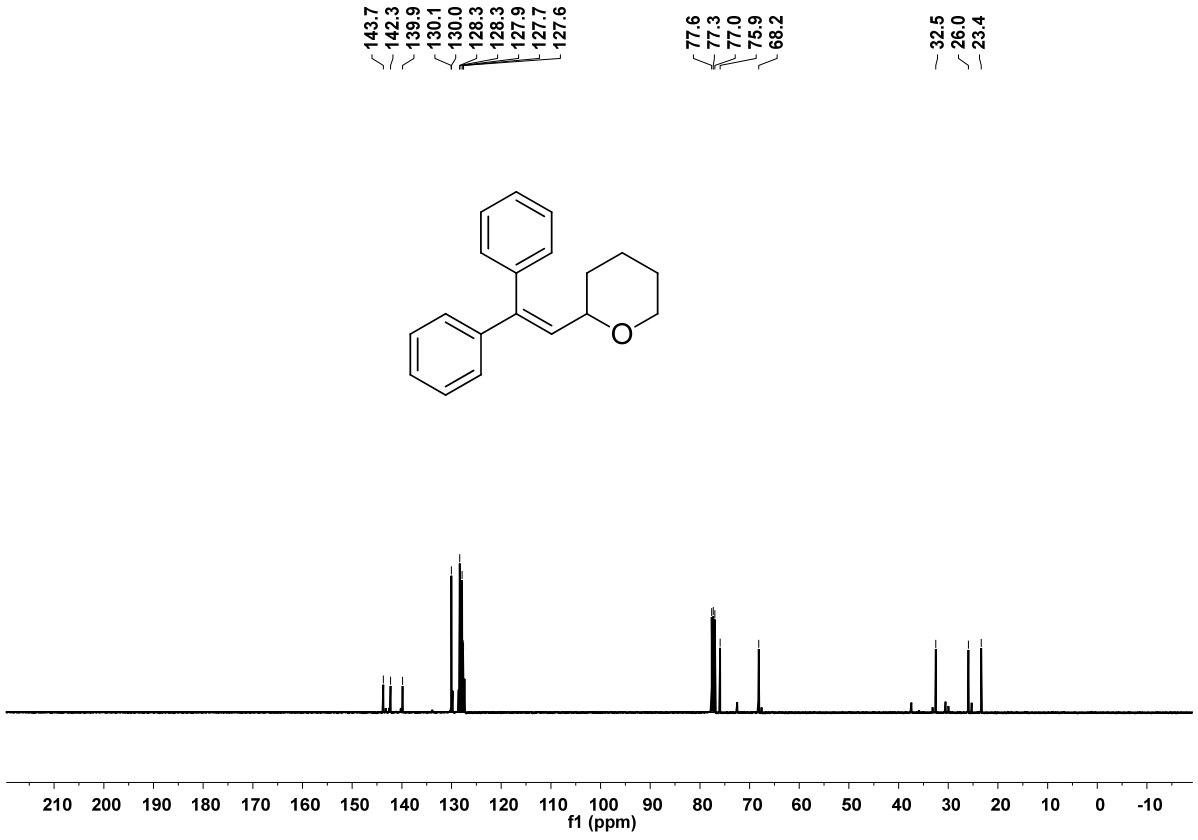
3k


7.38 7.38 7.36 7.36 7.35 7.34 7.26 7.26 7.25 7.25 7.24 7.21 7.20 7.19 7.18 7.17 7.17 7.16 7.15 7.14 7.12 6.92 6.92 6.90 6.81 6.80 6.79 6.79 5.87 5.85 5.85 5.83 4.09 4.08 3.84 3.79 3.78 3.76 3.75 3.73 3.72 3.70 3.69 3.67 3.66 3.65 3.64 3.63 3.62 3.52 3.50 3.49 3.49 3.48 3.47 3.45
MeO
O
O
1.91 1.94 1.96 1.33 0.71 1.31 1.00 0.38 0.67 1.09 3.13 1.03 2.98 1.03
f1 (ppm)

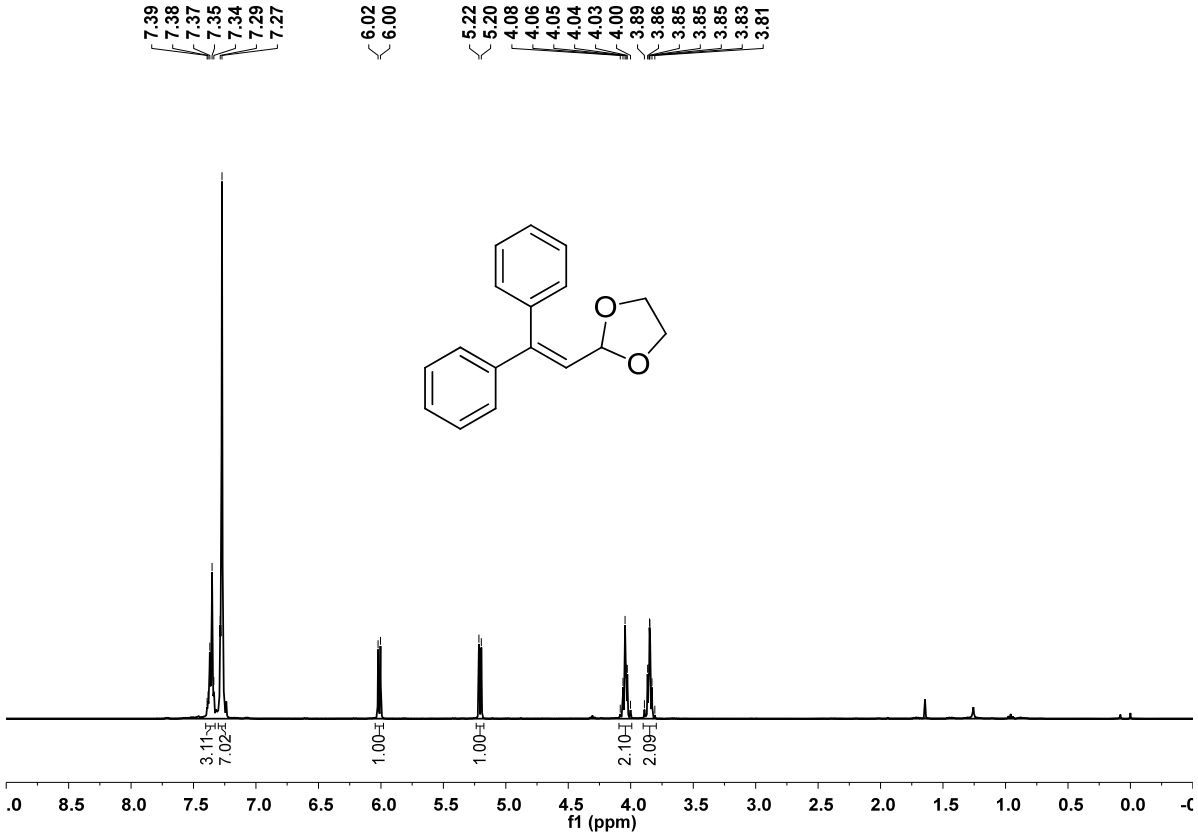

159.7 159.4 131.0 129.8 129.0 128.5 128.4 128.0 127.9 113.8 77.6 77.3 77.0 74.1 74.0 70.7 70.7 66.4 55.5
MeO
O
O
f1 (ppm)

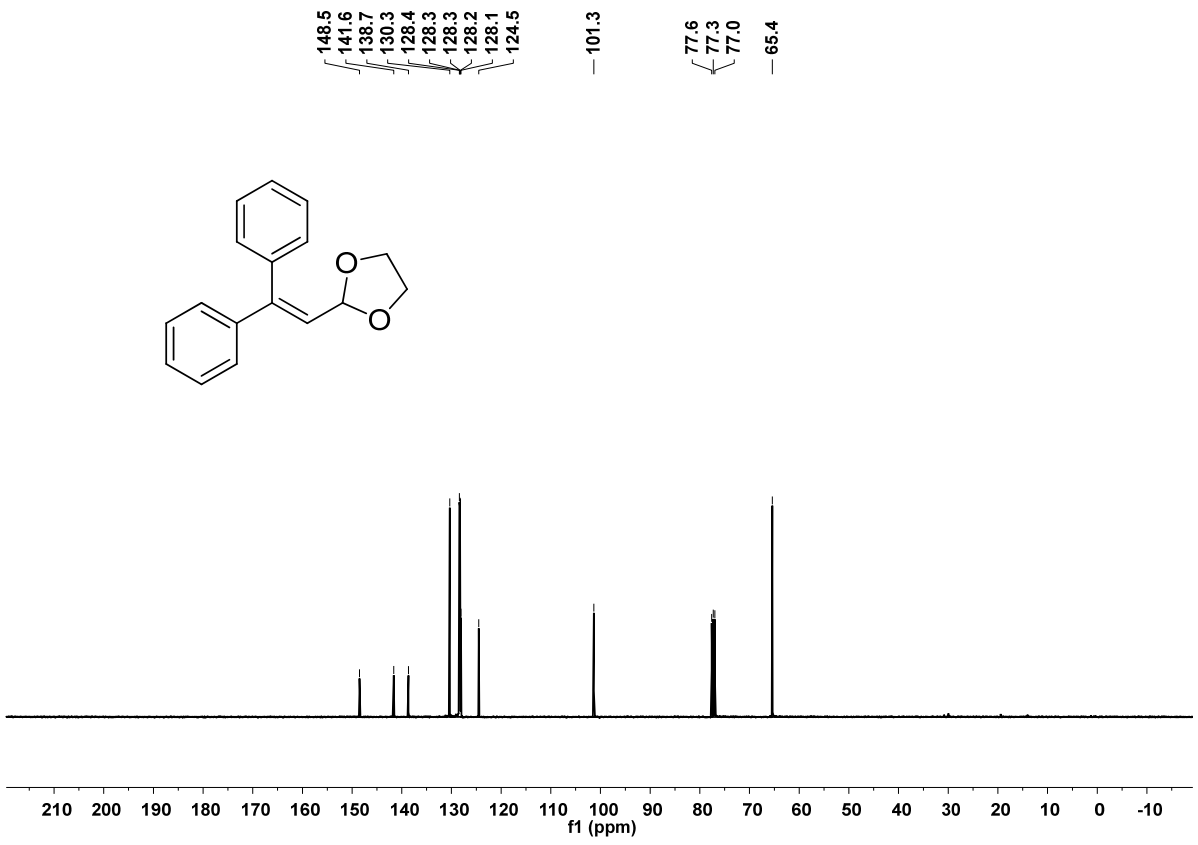
3l


7.39 7.38 7.37 7.35 7.34 7.33 7.31 7.26 7.24 7.23 7.23 7.22 7.21 7.21
6.05 6.03 3.99 3.98 3.98 3.96 3.95 3.95 3.86 3.85 3.83 3.83 3.81 3.80 3.39 3.39 3.36 3.36 3.33 3.33
1.81 1.79 1.78 1.68 1.67 1.62 1.61 1.59 1.59 1.58 1.57 1.56 1.56 1.55 1.54 1.53 1.46 1.45 1.43 1.42 1.41 1.38
O
3.85 6.61 1.00 0.18 1.06 1.39 1.24 1.27 3.40 2.19
f1 (ppm)

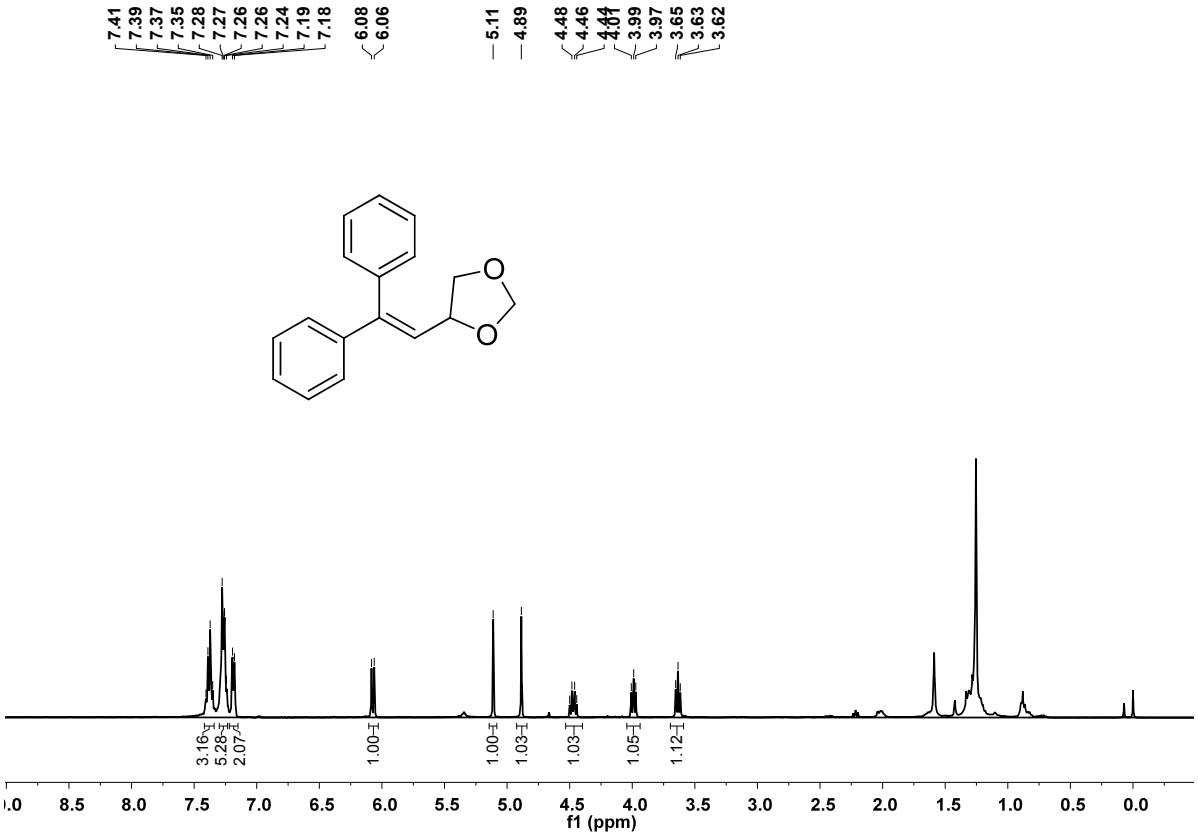

143.7 142.3 139.9 130.1 130.0 128.3 128.3 127.9 127.7 127.6
77.6 77.3 77.0 75.9 68.2
32.5 26.0 23.4
O
f1 (ppm)

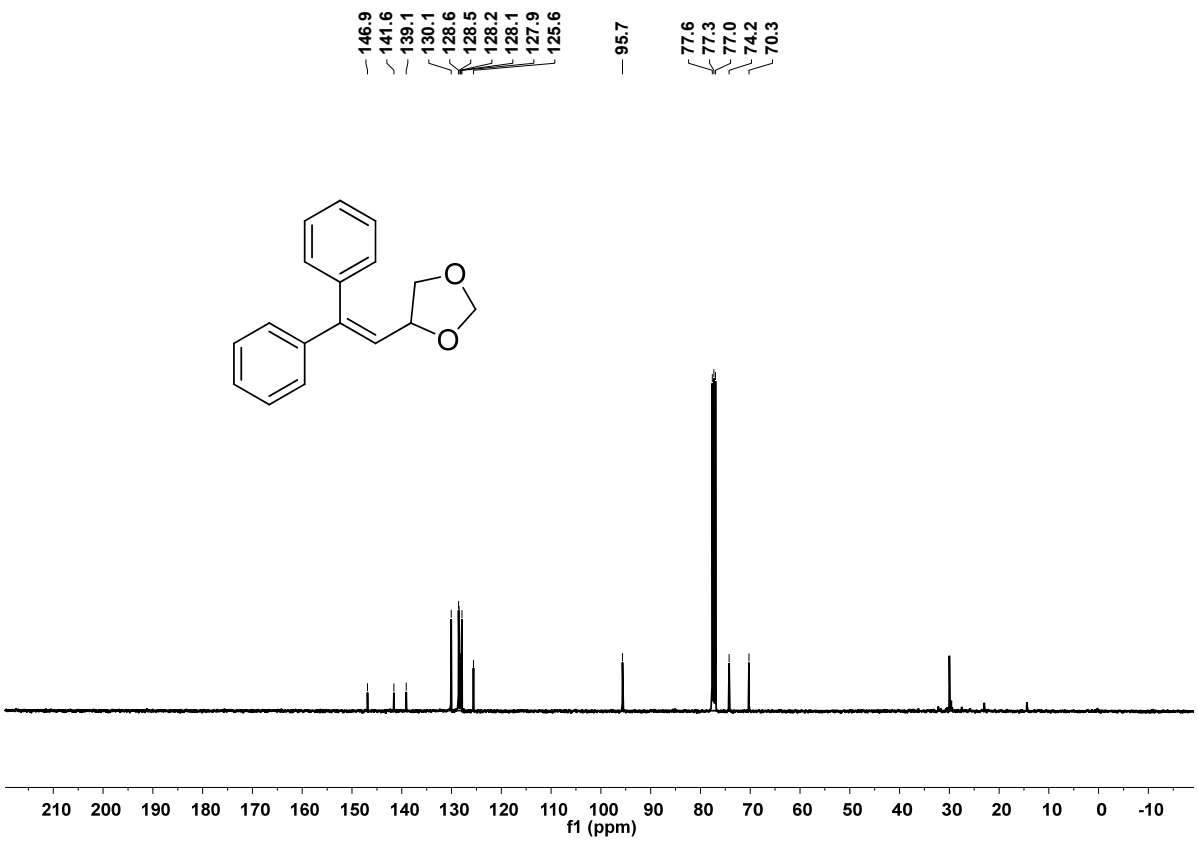
3m-A


7.39
7.38
7.37
7.35
7.34
7.29
7.27
6.02
6.00
5.22
5.20
4.08
4.06
4.05
4.04
4.03
4.00
3.89
3.86
3.85
3.85
3.85
3.83
3.81
O
O
3.11
7.02
1.00
1.00
2.10
2.09
f1 (ppm)

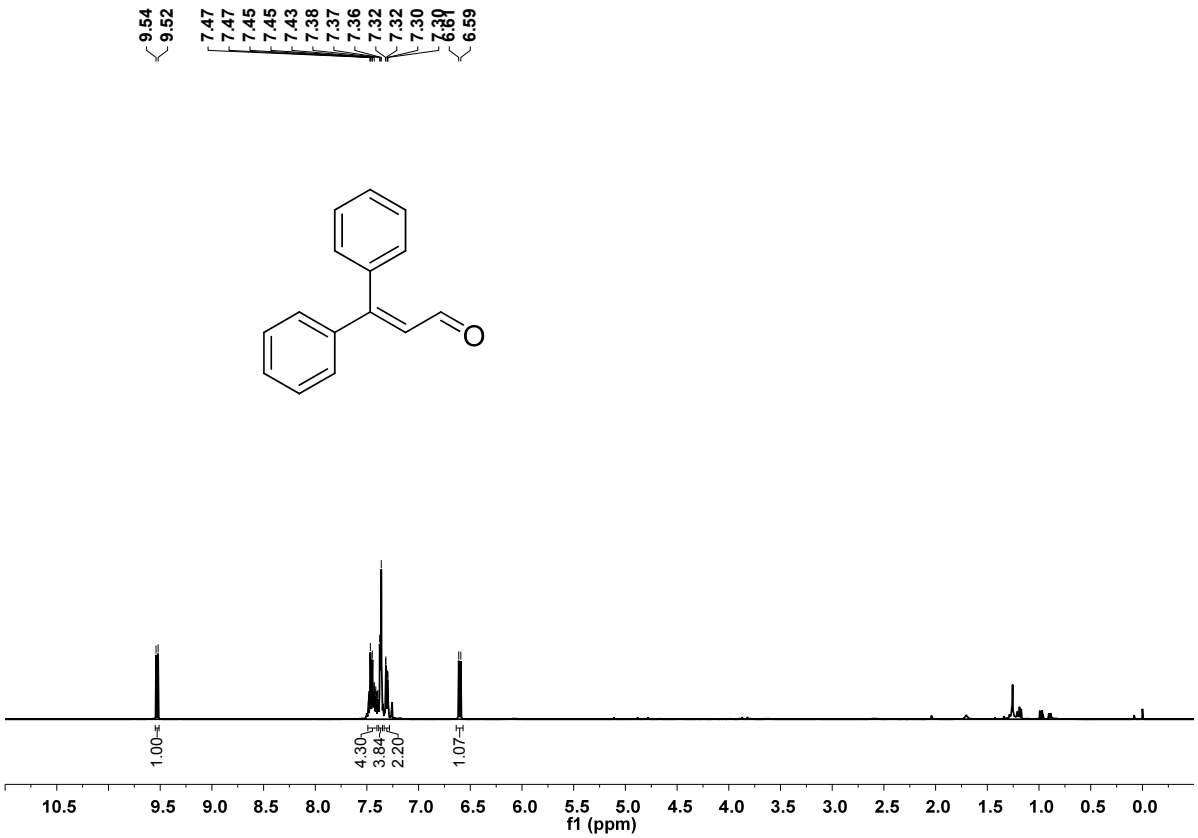

148.5
141.6
138.7
130.3
128.4
128.3
128.3
128.2
128.1
124.5
101.3
77.6
77.3
77.0
65.4
O
O
f1 (ppm)

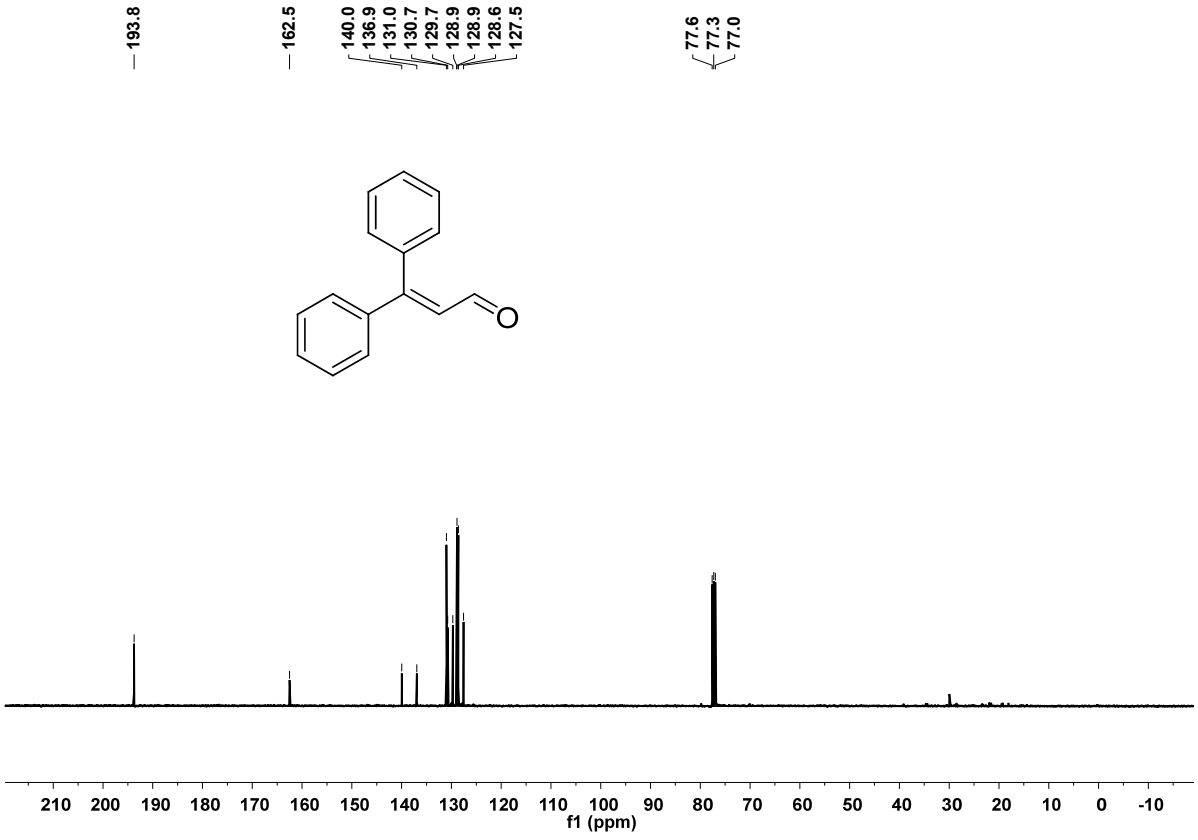
3m-B


7.41
7.39
7.37
7.35
7.28
7.27
7.26
7.26
7.24
7.19
7.18
6.08
6.06
5.11
4.89
4.48
4.46
4.44
4.01
3.99
3.97
3.65
3.63
3.62
O
O
3.16
5.28
2.07
1.00
1.00
1.03
1.03
1.05
1.12
f1 (ppm)

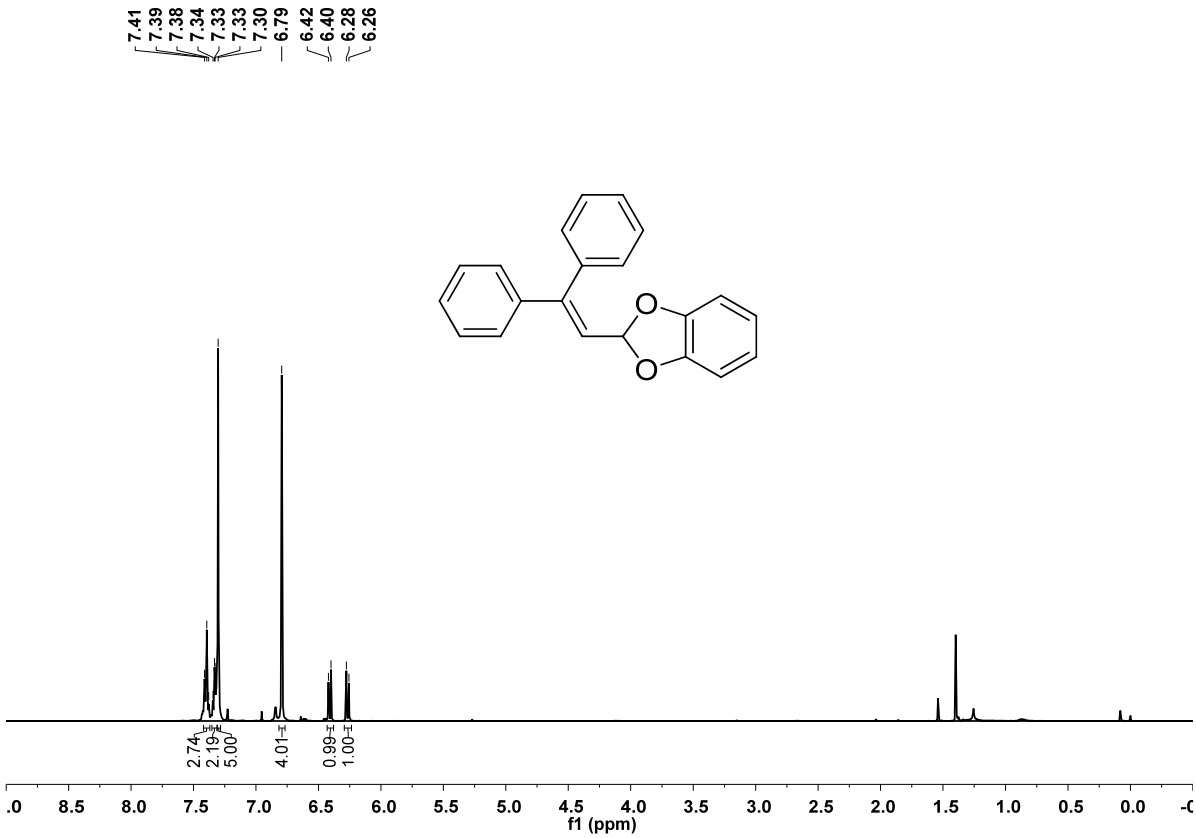

146.9
141.6
139.1
130.1
128.6
128.5
128.2
128.1
127.9
125.6
95.7
77.6
77.3
77.0
74.2
70.3
O
O
f1 (ppm)

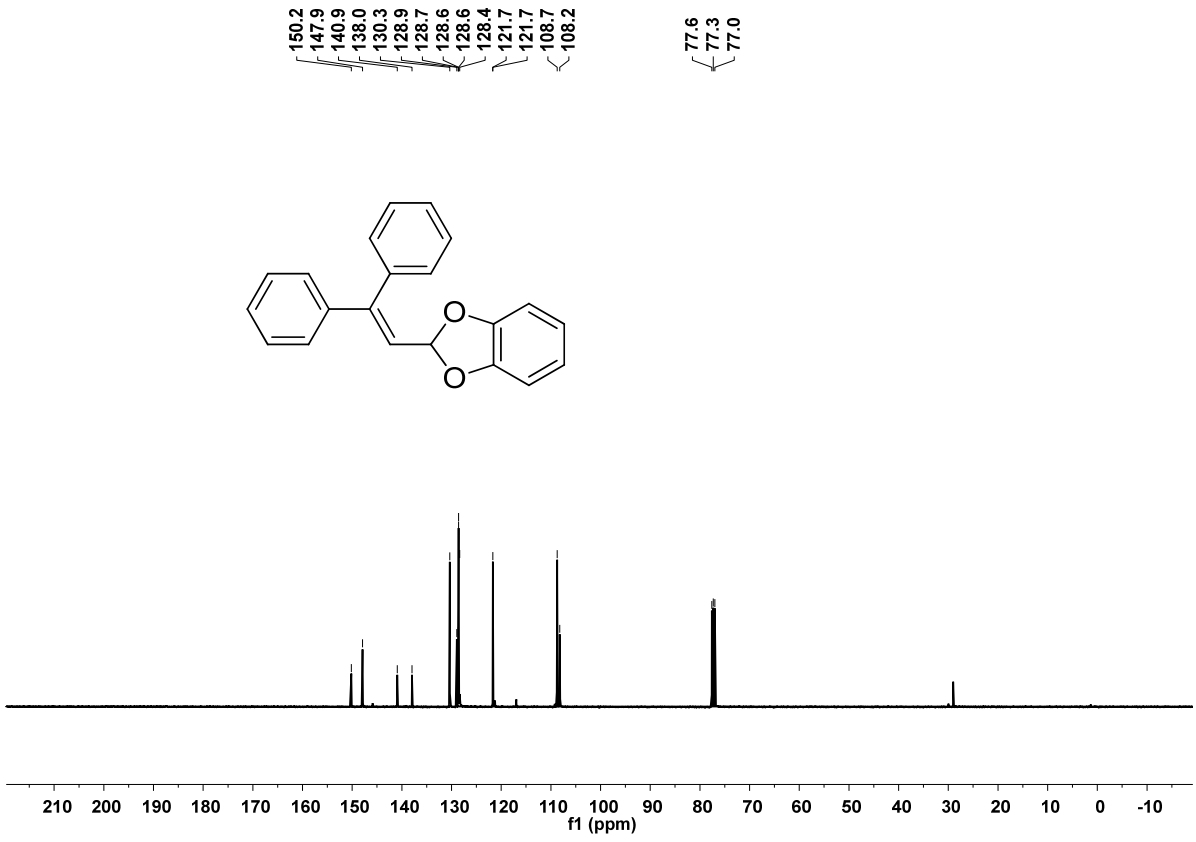
3m’


9.54
9.52
7.47
7.47
7.45
7.45
7.43
7.38
7.37
7.36
7.32
7.32
7.30
6.81
6.59
O
1.00
4.30
3.84
2.20
1.07
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

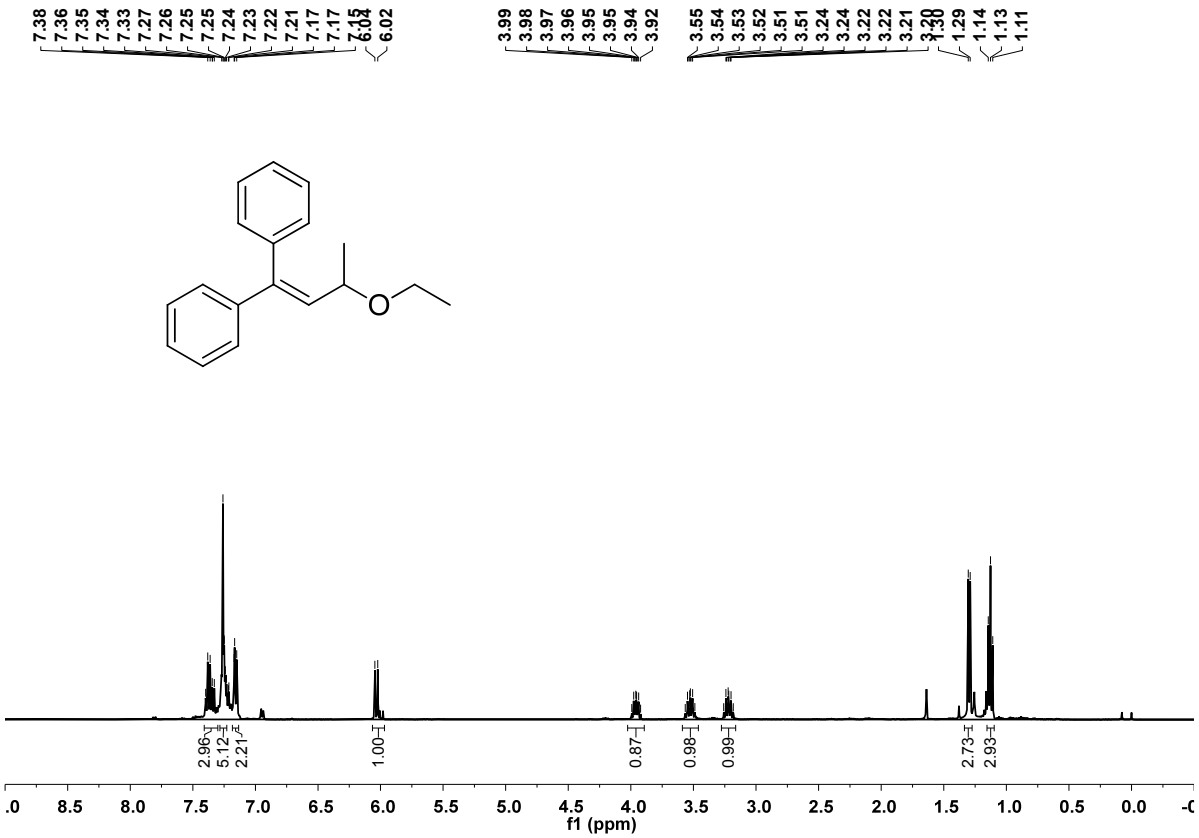

193.8
162.5
140.0
136.9
131.0
130.7
129.7
128.9
128.9
128.6
127.5
77.6
77.3
77.0
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

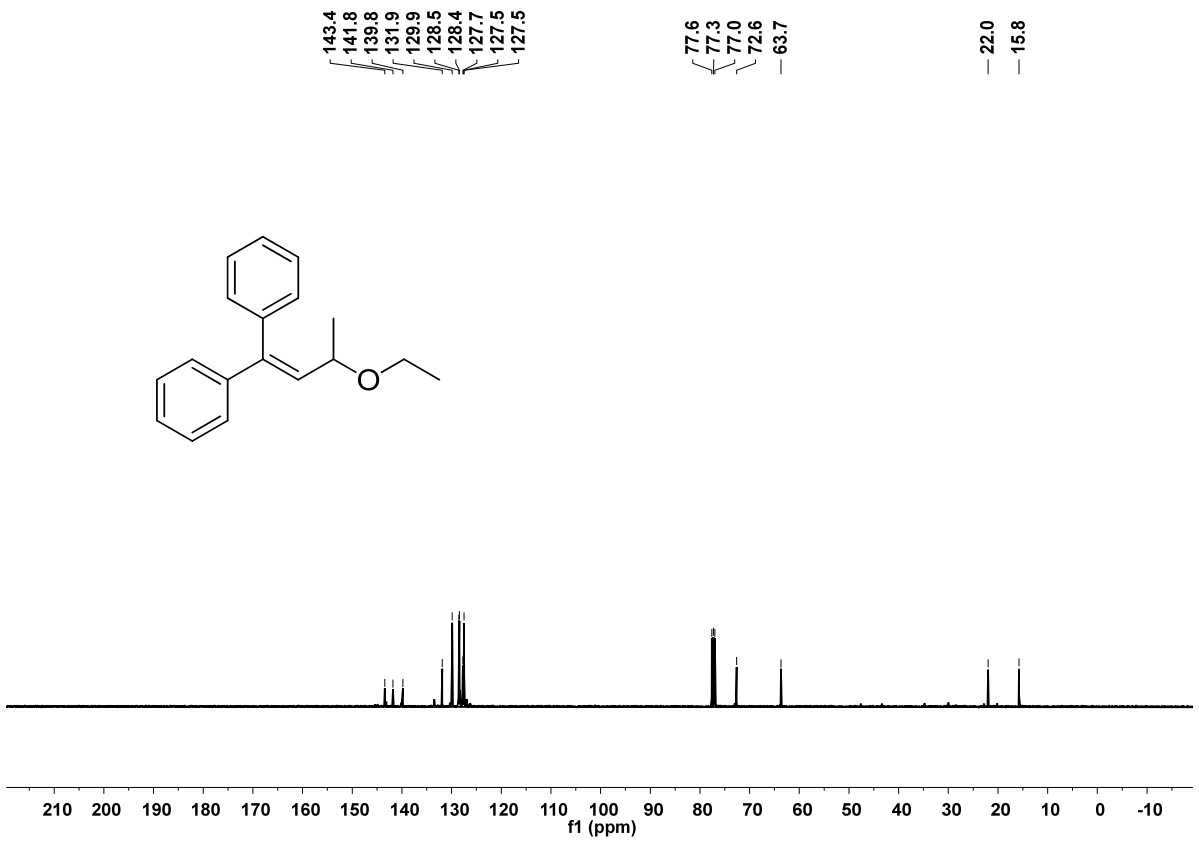
3n


7.41
7.39
7.38
7.34
7.33
7.33
7.30
6.79
6.42
6.40
6.28
6.26
2.74
2.19
5.00
4.01
0.99
1.00
f1 (ppm)

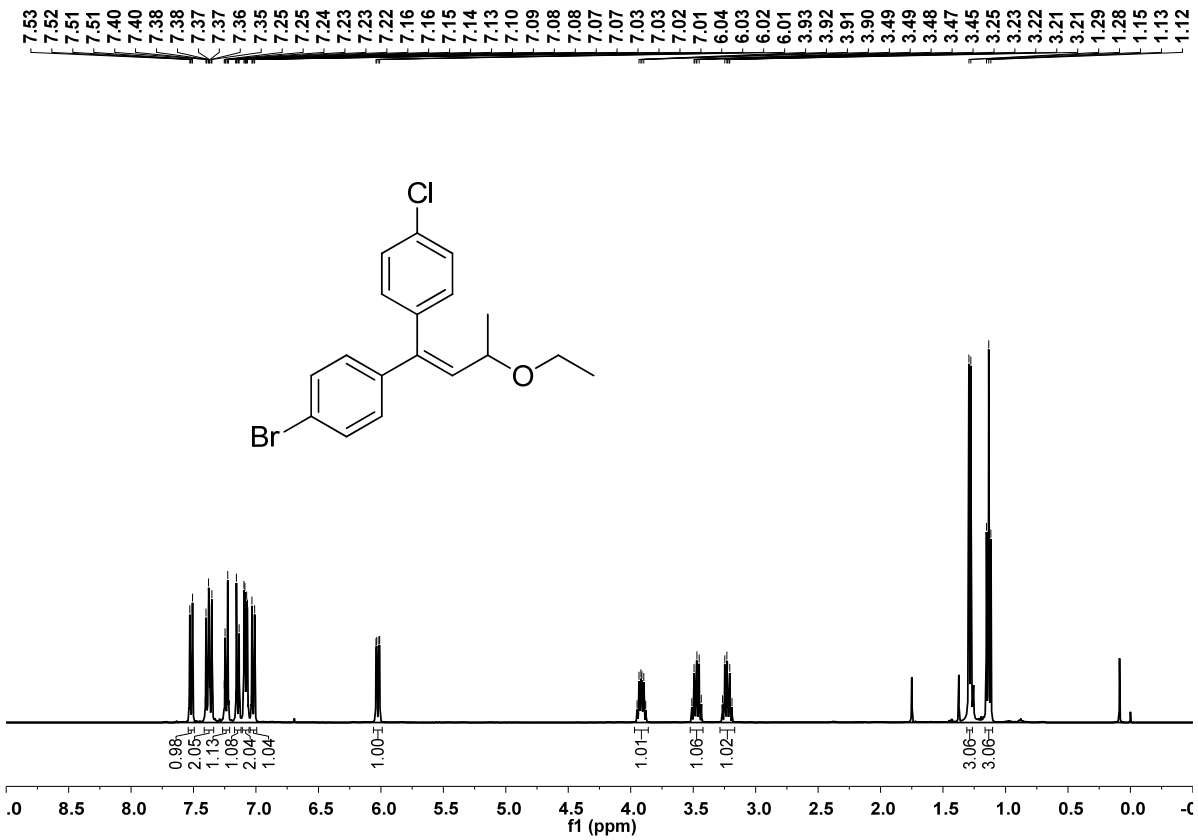

150.2
147.9
140.9
138.0
130.3
128.9
128.7
128.6
128.6
128.4
121.7
121.7
108.7
108.2
77.6
77.3
77.0
f1 (ppm)

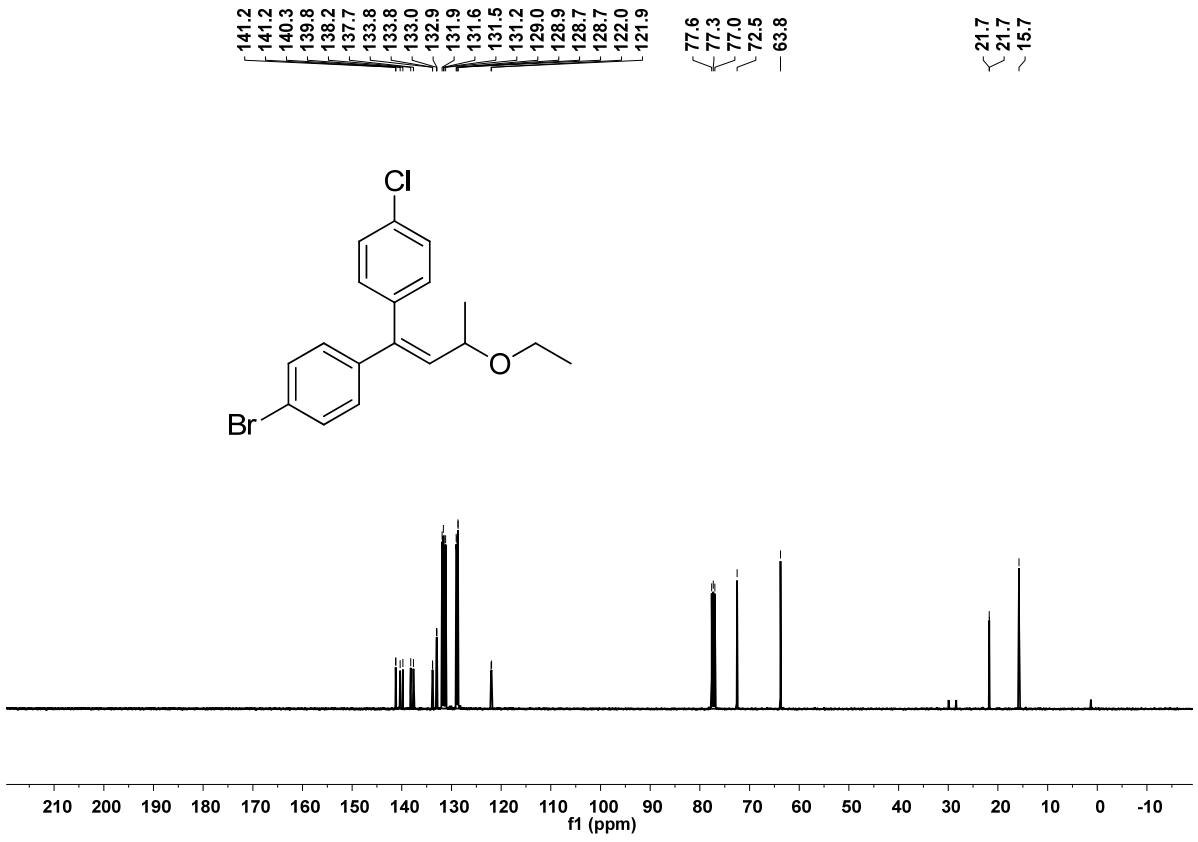
3o


7.38
7.36
7.35
7.34
7.33
7.27
7.26
7.25
7.25
7.24
7.23
7.22
7.21
7.17
7.17
6.04
6.02
3.99
3.98
3.97
3.96
3.95
3.95
3.94
3.92
3.55
3.54
3.53
3.52
3.51
3.51
3.24
3.24
3.22
3.22
3.21
1.30
1.29
1.14
1.13
1.11
2.96
5.12
2.21
1.00
0.87
0.98
0.99
2.73
2.93
f1 (ppm)

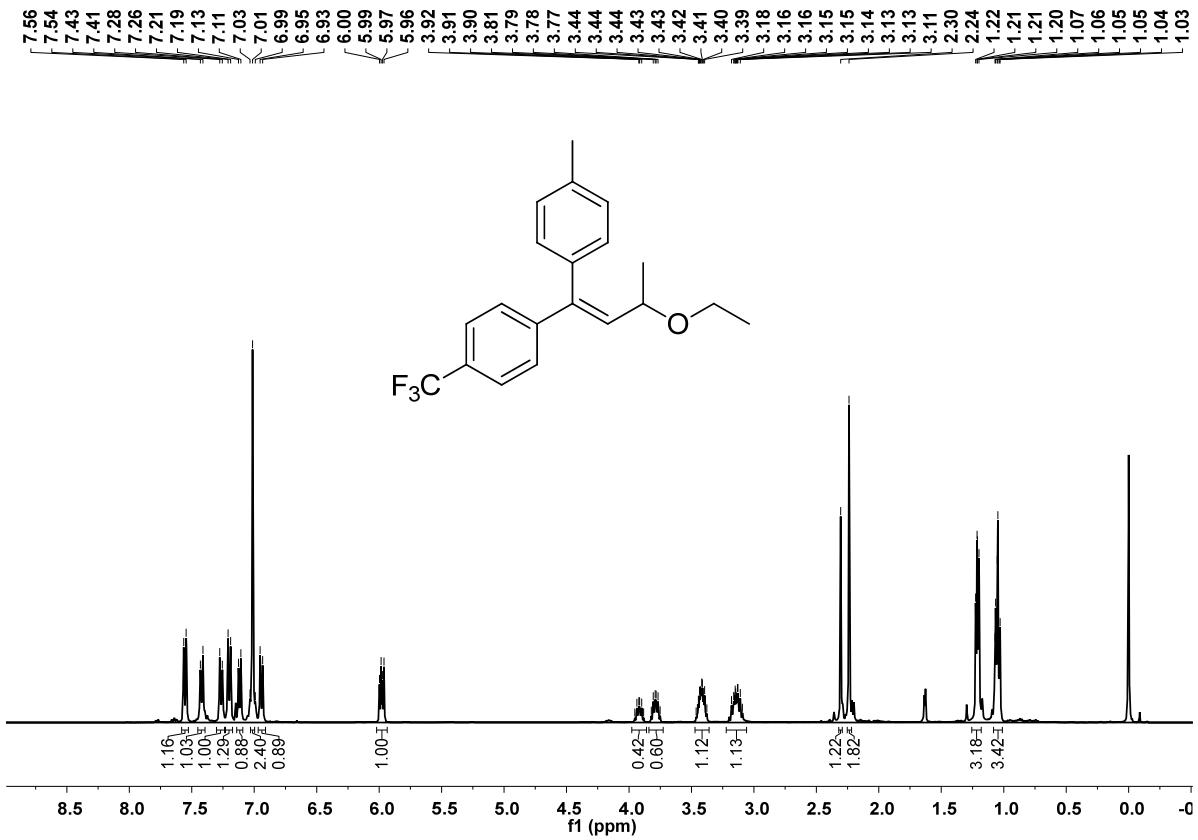

143.4
141.8
139.8
131.9
129.9
128.5
128.4
127.7
127.5
127.5
77.6
77.3
77.0
72.6
63.7
22.0
15.8
f1 (ppm)

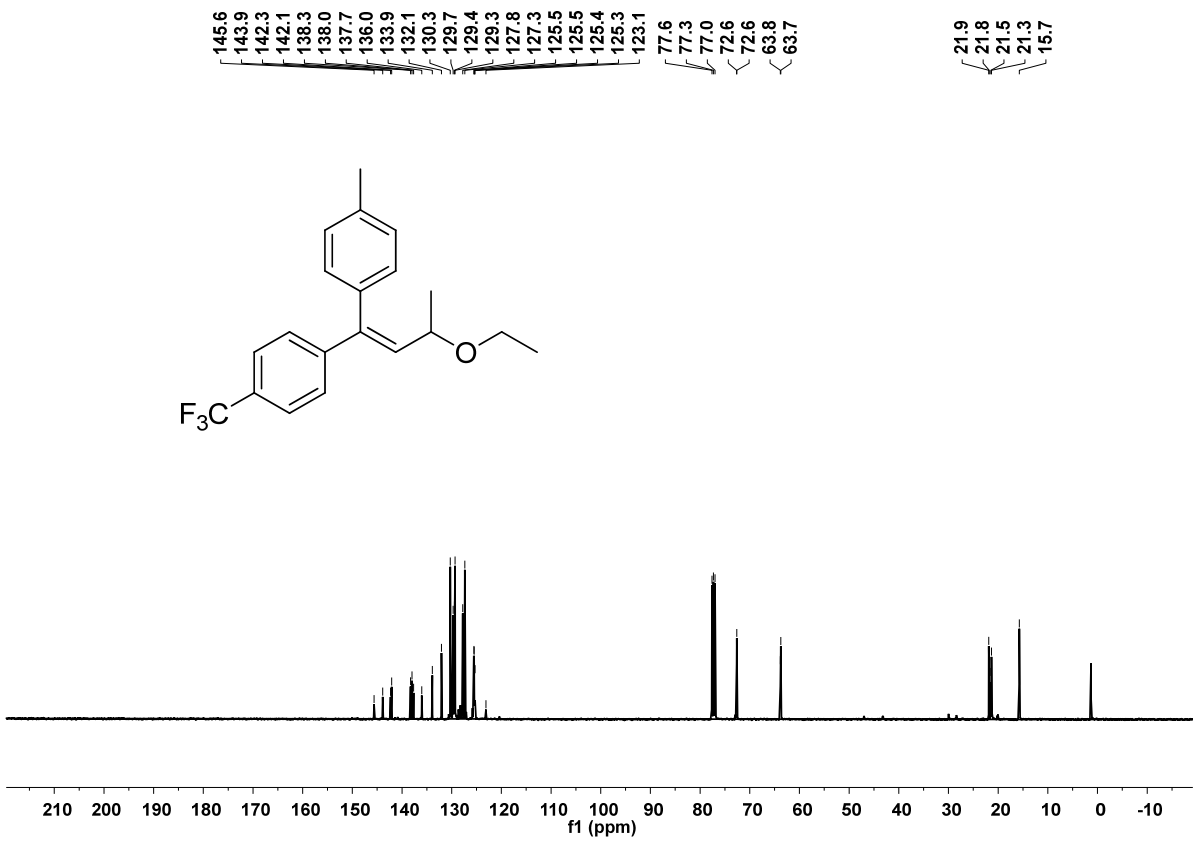
3p


7.53 7.52 7.51 7.51 7.40 7.40 7.38 7.38 7.37 7.37 7.36 7.35 7.25 7.25 7.24 7.23 7.23 7.22 7.16 7.16 7.15 7.14 7.13 7.10 7.09 7.08 7.08 7.07 7.07 7.03 7.03 7.02 7.01 6.04 6.03 6.02 6.01 3.93 3.92 3.91 3.90 3.49 3.49 3.48 3.47 3.45 3.25 3.23 3.22 3.21 3.21 1.29 1.28 1.15 1.13 1.12
Cl
O
Br
0.98 2.05 1.13 1.08 2.04 1.04 1.00 1.01 1.06 1.02 3.06 3.06
f1 (ppm)


141.2 141.2 140.3 139.8 138.2 137.7 133.8 133.8 133.0 132.9 131.9 131.6 131.5 131.2 129.0 128.9 128.7 128.7 122.0 121.9
77.6 77.3 77.0 72.5 63.8
21.7 21.7 15.7
Cl
O
Br
f1 (ppm)

3q


7.56 7.54 7.43 7.41 7.28 7.26 7.21 7.19 7.13 7.11 7.03 7.01 6.99 6.95 6.93 6.00 5.99 5.97 5.96 3.92 3.91 3.90 3.81 3.79 3.78 3.77 3.44 3.44 3.44 3.43 3.43 3.42 3.41 3.40 3.39 3.18 3.16 3.16 3.15 3.15 3.14 3.13 3.13 3.11 2.30 2.24 1.22 1.21 1.21 1.20 1.07 1.06 1.05 1.05 1.04 1.03
F3C
O
1.16 1.03 1.00 1.29 0.88 2.40 0.89 1.00 0.42 0.60 1.12 1.13 1.22 1.82 3.18 3.42
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


145.6 143.9 142.3 142.1 138.3 138.0 137.7 136.0 133.9 132.1 130.3 129.7 129.4 129.3 127.8 127.3 125.5 125.5 125.4 125.3 123.1 77.6 77.3 77.0 72.6 72.6 63.8 63.7 21.9 21.8 21.5 21.3 15.7
F3C
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)