Supporting Information

Cobalt-Catalyzed Carbonylative Cross-Coupling of Alkyl Tosylates and Dienes: Stereospecific Synthesis of Dienones at Low Pressure

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General Methods and Materials

Proton and carbon magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were recorded on a Bruker Avance III 600 CryoProbe (^{1}H NMR 600 MHz and ^{13}C at 151 MHz) spectrometer with solvent resonance as the internal standard (^{1}H NMR: $CHCl_3$ at 7.28 ppm; ^{13}C NMR: $CDCl_3$ at 77.0 ppm). ^{1}H NMR data are reported as follows: chemical shift (ppm), multiplicity (s = singlet, br s = broad singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. Mass spectra were obtained using a Thermo LTqFT mass spectrometer with electrospray introduction and external calibration. These samples were prepared in methanol. HPLC analysis was performed on a Shimadzu SPD-M20A photodiode array (PDA) system equipped with Daicel Chiralpak IE, IF, IG, and OJ-H columns or on an Agilent 1200 Series PDA system equipped with Chiralpak IA and IC using a flow rate of 1 mL/min with hexanes and isopropanol as eluent, unless otherwise indicated. Enantiomeric excess of starting materials that were insoluble in this mobile phase were determined from their corresponding alcohol intermediates.

Analytical thin layer chromatography (TLC) was performed on SiliaPlate 250μm thick silica gel purchased from Silicycle. Visualization was accomplished with short-wave UV light (254 nm), iodine, Hanessian's stain, or ethanolic acidic p-anisaldehyde solution followed by heating when necessary. Purification of the reaction products was carried out by flash chromatography using Siliaflash P60 silica gel (40-63μm) purchased from Silicycle. Carbon monoxide, Research Purity 99.999% (part number G2119118) was purchased from Matheson Tri-Gas. Tetrahydrofuran, diethyl ether, *N,N*-dimethylformamide, acetonitrile, and dichloromethane were dried by passage through a column of neutral alumina under nitrogen prior to use. *t*-Amyl alcohol was sparged with argon before storage over 3Å molecular sieves in the glovebox. $K[Co(CO)_4]$ was synthesized according to Ellis and co-workers.[1] 1,3-Cyclohexadiene was distilled prior to being stored in the glovebox at -30 ºC. All other reagents were obtained from commercial sources and used without further purification, unless otherwise noted. In addition, all reactions were carried out under an atmosphere of dry argon in flame or oven-dried glassware with magnetic stirring. The glass tubes for carbonylations were purchased from Ace Glass and the gas quick-connect adapters were obtained from Swagelok. An example of the carbonylation setup is shown below.

Swagelok setup for pressurizing reactions

List of Abbreviations

DCM = dichloromethane
DEAD = diethyl azodicarboxylate
DHP = 3,4-dihydro-*2H*-pyran
DIAD = diisopropyl azodicarboxylate
DMF = *N,N*-dimethylformamide
EtOAc = ethyl acetate
MeCN = acetonitrile
TEA = triethylamine
THF = tetrahydrofuran
TMP = 2,2,6,6-tetramethylpiperidine
TMS = trimethylsilyl
TsCl = 4-toluenesulfonyl chloride

Substrate Preparation

General Procedure A: Tosylation of Secondary Alcohols.

Tosylates were synthesized using a modified procedure from Tanabe, et. al.[2] To a 0 ºC ice bath cooled solution of TsCl (1.5 equiv) and trimethylamine hydrochloride (0.1 equiv) in DCM (1 M with respect to the alcohol) was added TEA (2.5 equiv) dropwise. To this solution was added a solution of the alcohol in DCM (1 M), the solution was then stirred for 1 hour at 0 ºC, monitoring by TLC. The reaction was quenched by addition of *N,N*-dimethyldiaminopropane (2.0 equiv) and stirred for 20 additional minutes while warming to room temperature. The reaction mixture was poured into water and separated. The organic layer was washed sequentially with 1 M HCl, a saturated aqueous solution of $NaHCO_3$, and brine, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash chromatography.

General Procedure B: Mitsunobu of Phenols

The corresponding alcohol (1 equiv), substituted phenol (1 equiv), and triphenylphosphine (1 equiv), were dissolved in THF (0.5 M) and cooled to 0 ºC. DIAD or DEAD (1 equiv) was added dropwise and the reaction was warmed to room temperature and stirred for 24-48 hours until complete consumption of the alcohol. The mixture was concentrated under reduced pressure and the crude product was purified by flash chromatography.

General Procedure C: THP Deprotection

The corresponding THP ether (1 equiv) was combined with pyridinium *p*-toluenesulfonate (0.1 equiv) in ethanol (0.4 M) and stirred overnight at 55 ºC. The mixture was concentrated under reduced pressure and the crude product was purified by flash chromatography.

General Procedure D: Olefination for Diene Synthesis

To a 0 ºC ice bath cooled suspension of $MePPh_3I$ (1.0 equiv) in THF (0.4 M) was added KO*t*Bu (1.5 equiv) and the bright yellow solution was stirred for 10 minutes. To this solution was added the corresponding aldehyde (1.02 equiv) and the reaction mixture

was stirred overnight at room temperature. Hexanes was then added and the mixture was filtered through sequential beds of celite and silica gel, rinsing with hexanes. The filtrate was then concentrated under reduced pressure and the crude product was purified by flash chromatography or distillation.

General Procedure E: CBS Reduction

To a room temperature solution of the enone (1 equiv) in PhMe (0.5 M) was added a solution of (*R*)-methyl oxazaborolidine (0.1 equiv) in PhMe (1 M) and stirred for 5 minutes. This mixture was then cooled to -78 °C and a solution of catecholborane (2 equiv) in THF (1 M) was added dropwise. The reaction was stirred overnight at -78 °C, then quenched with a saturated solution of sodium bicarbonate and warmed to room temperature. The mixture was extracted 3 times with EtOAc and the combined organic layers were washed 3-5 times with 1 M NaOH. The organic layer was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash chromatography.

General Procedure F: Olefin and Dieneone Hydrogenation

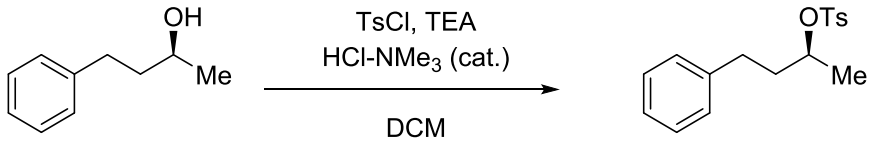
To a mixture of 1% w/w Pd (using 10% Pd/C) in MeOH (0.1 M) was added a solution of olefin or dieneone (1 equiv) in MeOH (1 M). The reaction was placed under a balloon of hydrogen, purging for 30 seconds, and stirred overnight at room temperature. The reaction mixture was then filtered over celite and concentrated under reduced. The products were either used without further purification or purified by flash chromatography.

General Procedure G: Propylene Oxide Opening for Non-Racemic Secondary Alcohol Synthesis

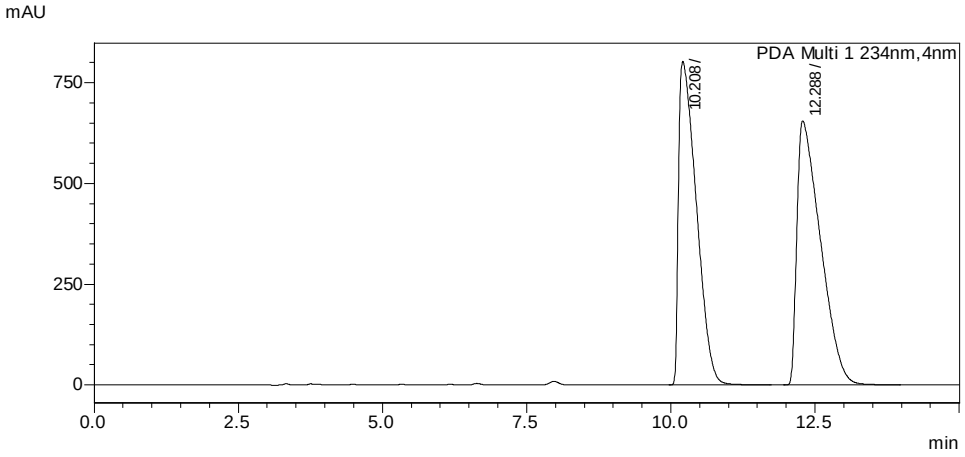
To a room temperature suspension of magnesium (1.5 equiv) in THF (2 M) with a crystal of I_2 was added the corresponding bromide (0.3 equiv) and stirred until Grignard reaction initiated (a heat gun was employed if no initiation after 10 minutes). The rest of the bromide was added (1.2 equiv) and stirred for 1 hour until complete consumption of magnesium. This solution was cannula transferred to a -30 °C suspension of CuI (1.5 equiv) in THF (1 M) and stirred for 5 minutes. (*S*)-Propylene oxide (1 equiv) was rapidly

added and the reaction was warmed to 0 °C and stirred for an additional 2 hours. The reaction was then quenched with an aqueous saturated solution of NH_4Cl and warmed to room temperature. Ether was added and the layers were separated. The organic layer was washed sequentially with a saturated aqueous solution of $NaHCO_3$, then brine, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash chromatography.

Tosylates:



(*S*)-4-phenylbutan-2-yl 4-methylbenzenesulfonate (1) was synthesized by tosylating (*S*)-4-phenylbutan-2-ol (1 g, 6.7 mmol) according to General Procedure A. The crude product was purified via flash chromatography in 10% EtOAc/hexanes to provide 2 g (100%) of tosylate **1** as a colorless, viscous oil. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 7.9 Hz, 2H), 7.36 (d, *J* = 7.9 Hz, 2H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.21 (t, *J* = 7.4 Hz, 1H), 7.12 – 7.07 (m, 2H), 4.72 – 4.63 (m, 1H), 2.64 (ddd, *J* = 14.0, 10.2, 5.8 Hz, 1H), 2.53 (ddd, *J* = 14.0, 10.1, 5.9 Hz, 1H), 2.48 (s, 3H), 1.96 (dddd, *J* = 13.6, 10.0, 7.4, 5.9 Hz, 1H), 1.88 – 1.79 (m, 1H), 1.33 (d, *J* = 6.3 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 144.6, 140.8, 134.4, 129.8, 128.5, 128.3, 127.8, 126.1, 79.9, 38.2, 31.2, 21.7, 20.9. **HRMS** (ESI) calculated for $[C_{17}H_{20}O_3S+H]^+$ 305.1211, found 305.1218. **Chiral HPLC** (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 99%.



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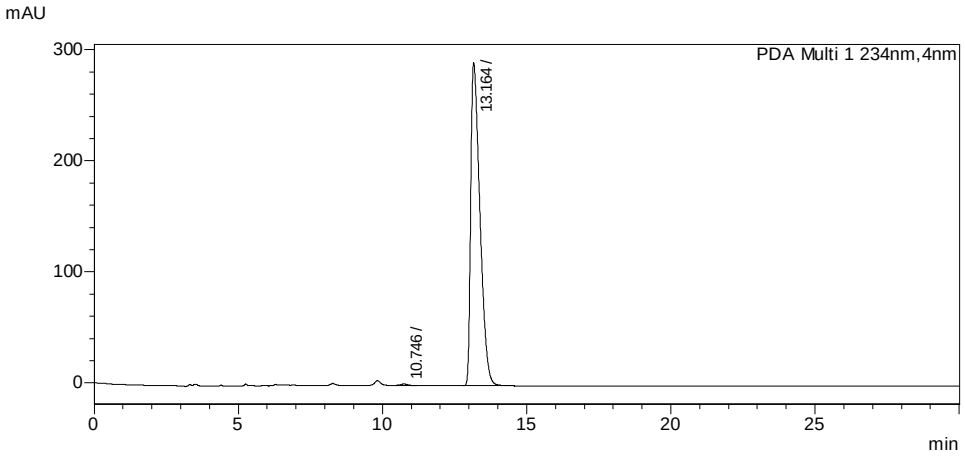

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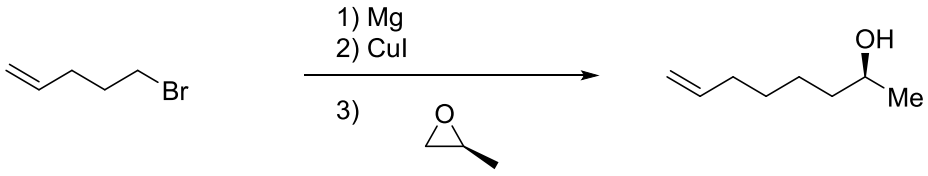
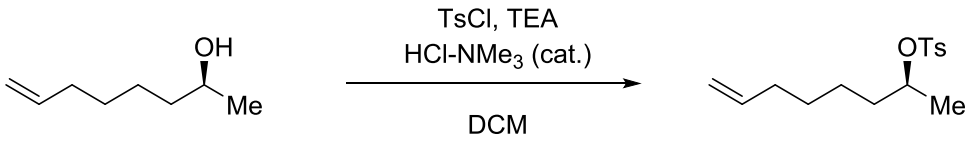
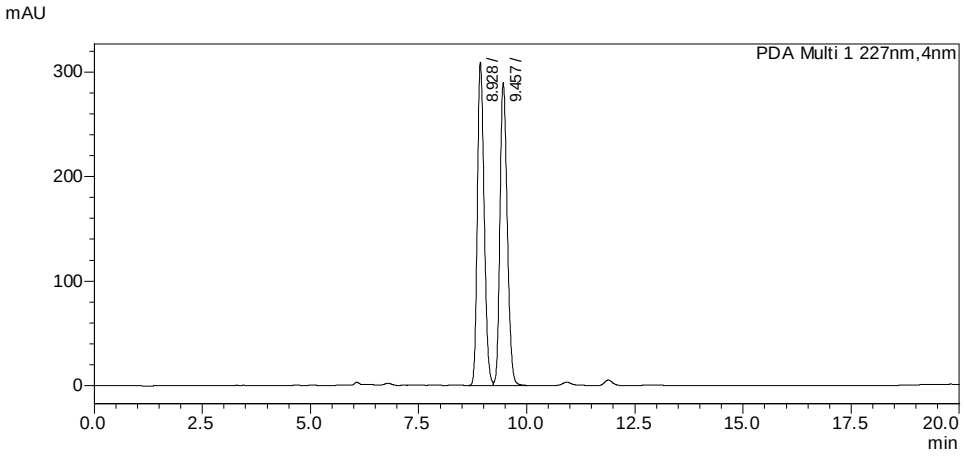


(*S*)-oct-7-en-2-ol (SI-1) was synthesized from 5-bromopent-1-ene (4.8 g, 32 mmol) according to General Procedure G. The crude product was purified via flash chromatography in 20% EtOAc/hexanes to provide 2.3 g (84%) of **SI-1** as a colorless liquid. Physical and spectral data were in accordance with literature data.[3]



(*S*)-oct-7-en-2-yl 4-methylbenzenesulfonate (SI-2) was synthesized by tosylating **SI-1** (1.2 g, 6.9 mmol) according to General Procedure A. The crude product was purified via flash chromatography in 5% EtOAc/hexanes to provide 3.2 g (73%) of **SI-1** as a colorless liquid. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.81 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.1 Hz, 2H), 5.74 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 4.99 – 4.91 (m, 2H), 4.65 – 4.56 (m, 1H), 2.46 (s, 3H), 1.96 (dtd, *J* = 8.5, 6.9, 1.5 Hz, 2H), 1.68 – 1.57 (m, 1H), 1.50 (ddt, *J* = 14.0, 9.7, 5.6 Hz, 1H), 1.35 – 1.22 (m, 6H), 1.25 – 1.11 (m, 1H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 144.5, 138.5, 134.5, 129.7, 127.7, 114.5, 80.6, 36.3, 33.5, 28.4, 24.3, 21.7, 20.9. **HRMS** (ESI) calculated for $[C_{15}H_{22}O_3S+Na]^+$ 305.1187, found 305.1185. **Chiral HPLC** (Daicel OJ-H, 95:5 hexanes:isopropanol): *ee* = 97%.



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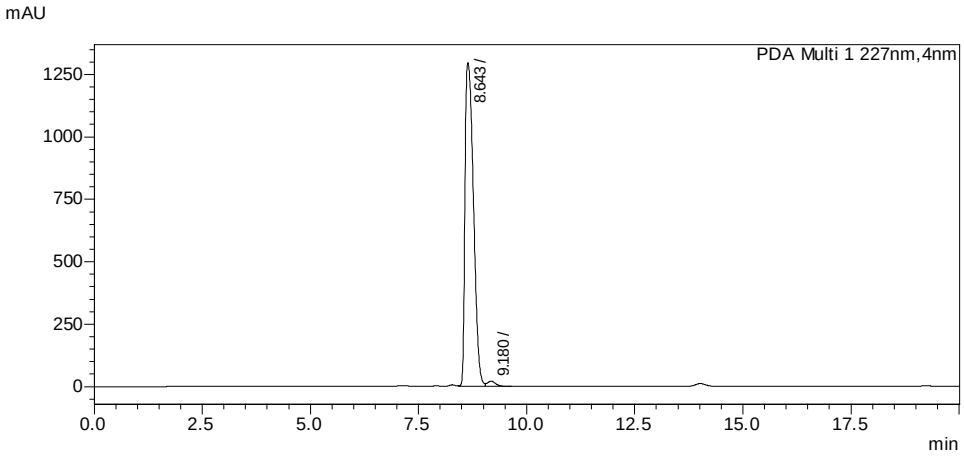

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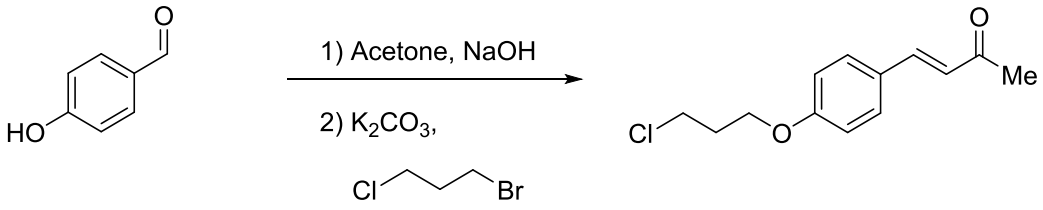
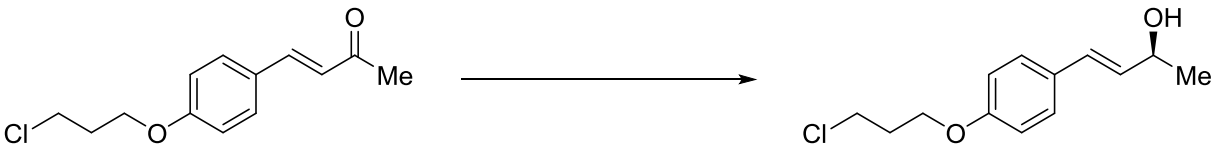
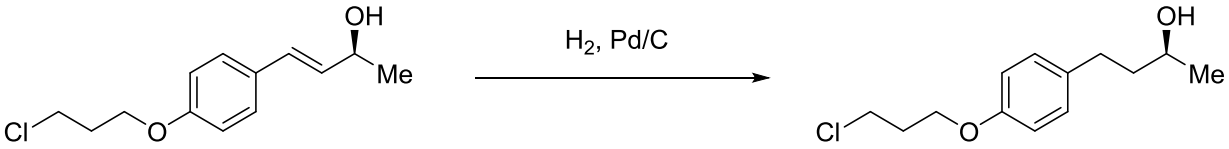


(*E*)-4-(4-(3-chloropropoxy)phenyl)but-3-en-2-one (SI-3) was prepared by first synthesizing (*E*)-4-(4-hydroxyphenyl)but-3-en-2-one via aldol condensation.[4] To a stirring solution of (*E*)-4-(4-hydroxyphenyl)but-3-en-2-one (1 g, 6.2 mmol) and 1-bromo-3-chloropropane (0.61 mL, 6.2 mmol) in MeCN (13 mL) was added K_2CO_3 (2.4 g, 17.4). The mixture was then refluxed for 2 hours, then cooled to room temperature and quenched by addition of water. This solution was extracted 3 times with EtOAc and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash chromatography in 10% EtOAc/hexanes to provide 0.9 g (61%) of **SI-3** as a cream colored solid. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.55 – 7.47 (m, 3H), 6.97 – 6.92 (m, 2H), 6.64 (dd, *J* = 16.3, 2.2

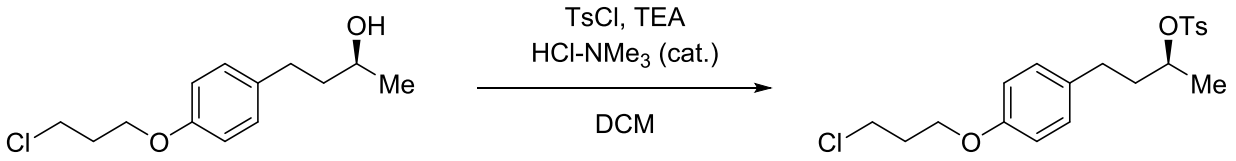
Hz, 1H), 4.19 (td, *J* = 5.9, 1.8 Hz, 2H), 3.78 (td, *J* = 6.3, 1.9 Hz, 2H), 2.39 (d, *J* = 2.1 Hz, 3H), 2.28 (qt, *J* = 6.1, 3.2 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 198.5, 160.7, 143.2, 130.0, 127.2, 125.1, 114.9, 64.3, 41.4, 32.1, 27.5. **HRMS** (ESI) calculated for $[C_{13}H_{15}ClO_2+Na]^+$ 261.0658, found 261.0654.



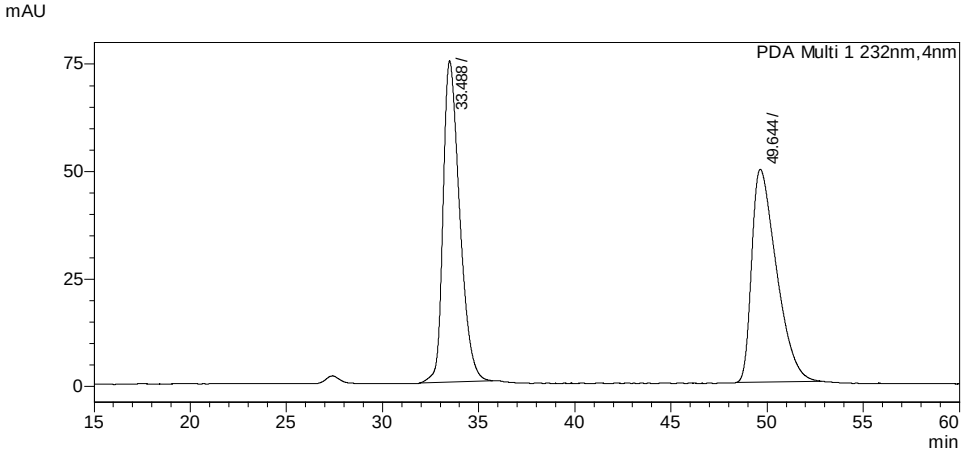
(*S*,*E*)-4-(4-(3-chloropropoxy)phenyl)but-3-en-2-ol (SI-4) was synthesized by reducing **SI-3** (0.9 g, 3.8 mmol) according to General Procedure E. The crude product was plugged through silica with 20% EtOAc/hexanes and used without further purification.



(*S*)-4-(4-(3-chloropropoxy)phenyl)butan-2-ol (SI-5) was synthesized by hydrogenating **SI-4** according to General Procedure F. The crude product was purified by flash chromatography in 20% EtOAc/hexanes to provide 0.22 g (24% over 2 steps) of **SI-5** as a colorless oil. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.14 (d, *J* = 8.5 Hz, 2H), 6.88 – 6.83 (m, 2H), 4.12 (t, *J* = 5.8 Hz, 2H), 3.88 – 3.81 (m, 1H), 3.77 (t, *J* = 6.4 Hz, 2H), 2.72 (ddd, *J* = 13.8, 9.5, 6.0 Hz, 1H), 2.64 (ddd, *J* = 13.8, 9.3, 6.9 Hz, 1H), 2.25 (p, *J* = 6.1 Hz, 2H), 1.83 – 1.72 (m, 2H), 1.43 – 1.35 (m, 1H), 1.25 (d, *J* = 6.2 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 156.9, 134.3, 129.3, 114.4, 67.5, 64.2, 41.6, 41.1, 32.3, 31.2, 23.7. **HRMS** (ESI) calculated for $[C_{13}H_{19}ClO_2+Na]^+$ 265.0971, found 265.0967.



(*S*)-4-(4-(3-chloropropoxy)phenyl)butan-2-yl 4-methylbenzenesulfonate (SI-6) was synthesized by tosylating **SI-5** (0.22 g, 0.9 mmol) according to General Procedure A. The crude product was purified via flash chromatography in 10% EtOAc/hexanes to provide 0.24 g (67%) of **SI-6** as a colorless, viscous oil. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.1 Hz, 2H), 7.00 (d, *J* = 8.5 Hz, 2H), 6.82 (d, *J* = 8.5 Hz, 2H), 4.70 – 4.61 (m, 1H), 4.11 (t, *J* = 5.8 Hz, 2H), 3.77 (t, *J* = 6.3 Hz, 2H), 2.57 (ddd, *J* = 14.1, 10.0, 5.8 Hz, 1H), 2.52 – 2.40 (m, 4H), 2.28 – 2.18 (m, 2H), 1.92 (dddd, *J* = 13.5, 10.0, 7.4, 5.9 Hz, 1H), 1.79 (ddt, *J* = 14.8, 10.5, 5.5 Hz, 1H), 1.31 (d, *J* = 6.3 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 157.0, 144.5, 134.4, 133.1, 129.8, 129.3, 127.8, 114.4, 79.9, 64.2, 41.6, 38.4, 32.3, 30.3, 21.7, 20.9. **HRMS** (ESI) calculated for $[C_{20}H_{25}ClO_4S+Na]^+$ 419.1060, found 419.1056. **Chiral HPLC** (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 44%.



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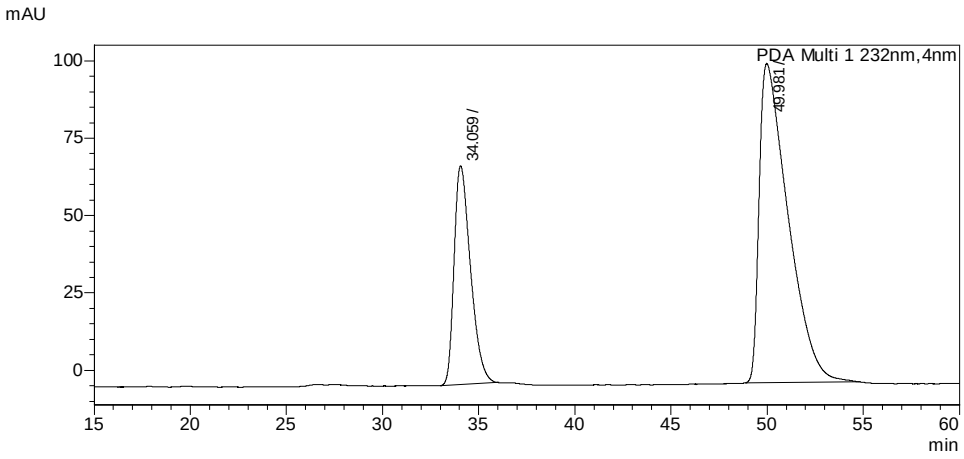

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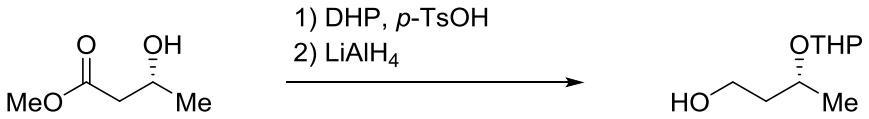
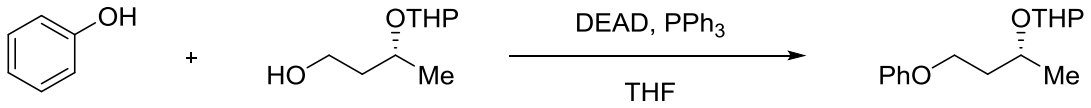
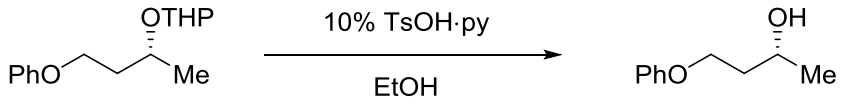


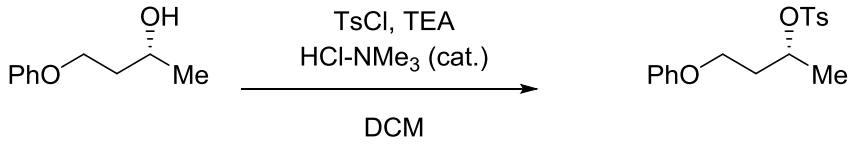
(*R*)-3-((tetrahydro-2H-pyran-2-yl)oxy)butan-1-ol (SI-7) was synthesized by the 2 step sequence shown above. (*R*)-methyl 3-hydroxybutanoate (20 g, 169 mmol), 3,4-dihydro-*2H*-pyran (18.5 mL, 203 mmol), and *p*-toluenesulfonic acid monohydrate (0.3 g, 1.7 mmol) were combined in Et_2O and allowed to stir overnight at room temperature. The reaction was quenched via addition of a saturated sodium bicarbonate solution. The organic layer was separated and the aqueous layer was extracted twice with Et_2O and the organic layers were combined and dried over $MgSO_4$, filtered, and concentrated under reduced pressure to provide 34.1 g of the crude product (99%). A solution of the crude product (24.1 g, 119 mmol) in Et_2O (100 mL) was added dropwise to a suspension of $LiAlH_4$ (4.5 g, 119 mmol) in Et_2O (150 mL) at 0 ºC. The reaction was allowed to warm to room temperature and stirred overnight. The reaction was cooled to 0 ºC and quenched by sequential dropwise addition of water (4.5 mL), 3 M NaOH (4.5 mL), and water (13.5 mL). The slurry was warmed to room temperature, dried with $MgSO_4$, filtered through a pad of Celite, and concentrated. Crude product **SI-7** (21 g, 100%) was used without further purification.



2-(((*R*)-4-phenoxybutan-2-yl)oxy)tetrahydro-2H-pyran (SI-8) was synthesized by subjecting **SI-7** (1.6 g, 9 mmol) and phenol (0.85 g, 9 mmol) to General Procedure B. The crude product was purified via flash chromatography in 10% EtOAc/hexanes to provide 1.3 g (58%) of **SI-8** as a clear oil in a 1:1 mixture of diastereomers. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.39 – 7.25 (m, 4H), 7.00 – 6.90 (m, 6H), 4.78 (t, *J* = 3.5 Hz, 1H), 4.65 (dd, *J* = 5.2, 2.8 Hz, 1H), 4.21 – 3.93 (m, 7H), 3.81 (ddd, *J* = 11.5, 8.4, 3.3 Hz, 1H), 3.56 – 3.43 (m, 2H), 2.09 – 1.92 (m, 4H), 1.83 (dtd, *J* = 12.3, 6.3, 5.2, 3.0 Hz, 2H), 1.78 – 1.66 (m, 2H), 1.62 – 1.46 (m, 8H), 1.34 (d, *J* = 6.3 Hz, 3H), 1.22 (d, *J* = 6.1 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 159.0, 158.9, 129.5, 129.4, 120.6, 120.5, 114.52, 114.46, 99.6, 95.3, 71.5, 67.9, 64.7, 64.3, 63.0, 62.3, 37.0, 36.4, 31.2, 31.1, 25.50, 25.46, 22.1, 20.1, 19.6, 19.4. **HRMS** (ESI) calculated for $[C_{15}H_{22}O_3+Na]^+$ 273.1467, found 273.1457.

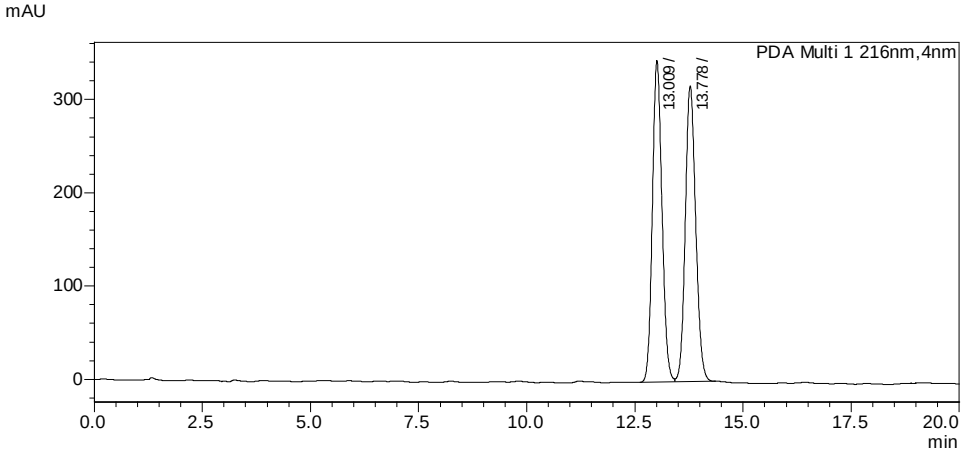


(*R*)-4-phenoxybutan-2-ol (SI-9) was synthesized by subjecting **SI-8** (0.72 g, 2.9 mmol) to General Procedure C. The crude product was purified via flash chromatography in 15% EtOAc/hexanes to provide 0.36 g (75%) of **SI-9** as a colorless oil. Physical and spectral data were in accordance with literature data.[5]

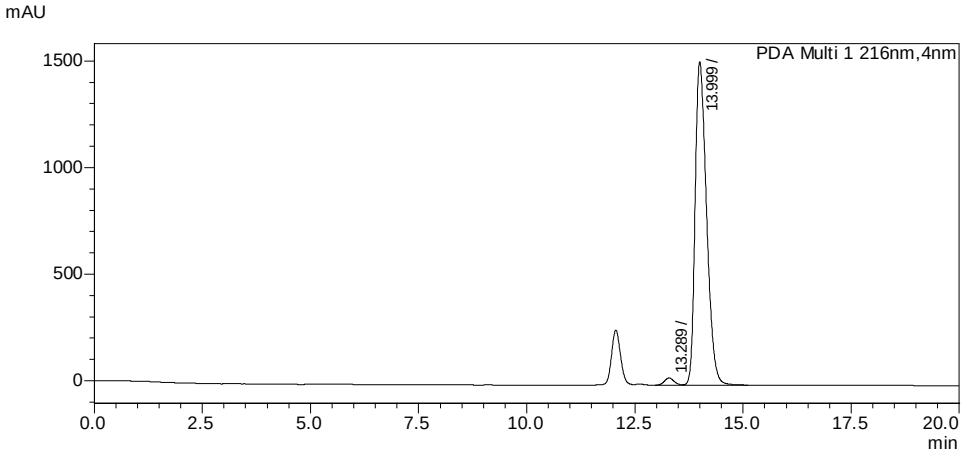


(*R*)-4-phenoxybutan-2-yl 4-methylbenzenesulfonate (SI-10) was synthesized by tosylating **SI-9** (1 g, 6 mmol) according to General Procedure A. The crude product was purified via flash chromatography in 20% EtOAc/hexanes to provide 1.5 g (78%) of tosylate **SI-10** as a white solid. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.78 – 7.73 (m, 2H), 7.29 – 7.23 (m, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.95 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.71 – 6.66

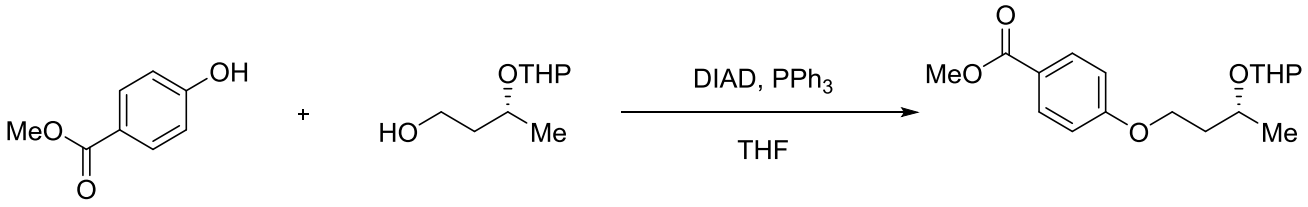
(m, 2H), 4.93 – 4.85 (m, 1H), 3.81 (dt, *J* = 9.8, 5.0 Hz, 1H), 3.65 (td, *J* = 9.1, 4.5 Hz, 1H), 2.28 (s, 3H), 2.08 – 1.93 (m, 2H), 1.47 (d, *J* = 6.3 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 158.4, 144.6, 133.7, 129.7, 129.3, 127.6, 120.7, 114.2, 77.3, 62.8, 36.2, 21.7, 21.6. **HRMS** (ESI) calculated for $[C_{17}H_{20}O_4S+Na]^+$ 343.0980, found 343.0985. **Chiral HPLC**: (ChiralPak IF, 95:5 hexanes:isopropanol): *ee* = 96%.



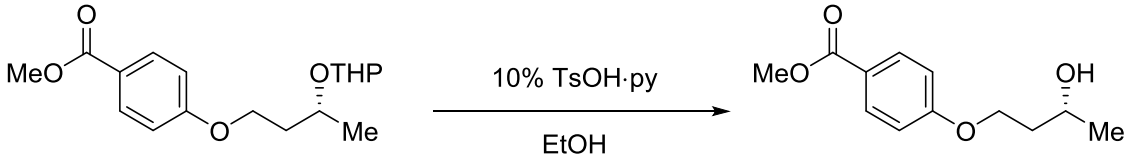
Peak#	Ret. Time	Area	Area%
1	13.009	5352979	49.752
2	13.778	5406434	50.248
Total		10759413	100



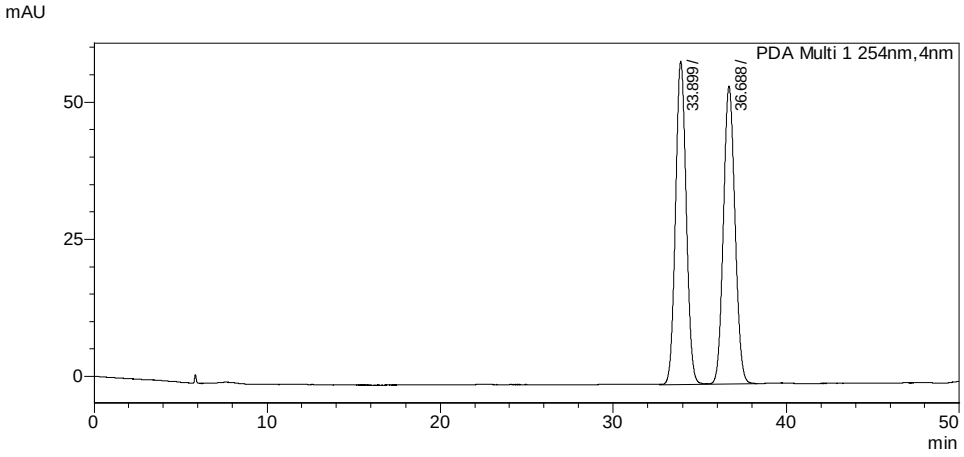
Peak#	Ret. Time	Area	Area%
1	13.289	517633	1.824
2	13.999	27865833	98.176
Total		28383465	100



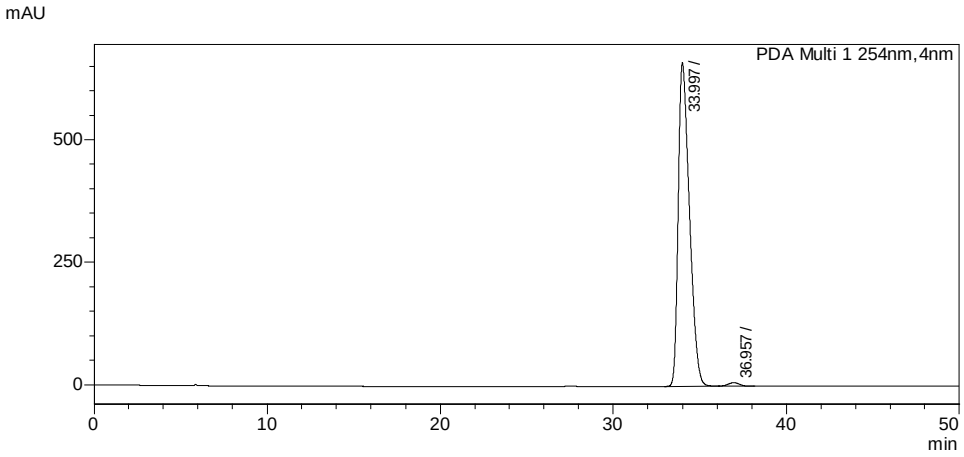
Methyl 4-((3*R*)-3-((tetrahydro-2H-pyran-2-yl)oxy)butoxy)benzoate (SI-11) was synthesized by subjecting **SI-7** (2 g, 11.5 mmol) and methyl 4-hydroxylbenzoate (1.75 g, 11.5 mmol) to General Procedure B. The crude product was purified via flash chromatography in 15% EtOAc/hexanes to provide 1.8 g (51%) of **SI-11** as a colorless oil in a 1:1 mixture of diastereomers. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 8.04 – 7.97 (m, 4H), 6.97 – 6.90 (m, 4H), 4.75 (dd, *J* = 4.5, 2.8 Hz, 1H), 4.63 (dd, *J* = 5.1, 2.8 Hz, 1H), 4.24 (ddd, *J* = 9.3, 7.5, 6.3 Hz, 1H), 4.20 – 4.05 (m, 4H), 4.07 – 3.94 (m, 2H), 3.91 (d, *J* = 2.2 Hz, 6H), 3.76 (ddd, *J* = 11.3, 8.1, 3.2 Hz, 1H), 3.51 (dtd, *J* = 10.6, 4.5, 4.0, 2.3 Hz, 1H), 3.47 – 3.40 (m, 1H), 2.09 – 1.94 (m, 4H), 1.86 – 1.65 (m, 4H), 1.60 – 1.43 (m, 8H), 1.34 (d, *J* = 6.3 Hz, 3H), 1.22 (d, *J* = 6.2 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 167.0, 166.9, 162.9, 162.7, 131.6, 131.5, 122.5, 122.3, 114.1, 114.0, 99.5, 95.6, 71.4, 67.8, 65.0, 64.7, 63.0, 62.5, 51.90, 51.88, 36.8, 36.2, 31.14, 31.12, 25.45, 25.43, 22.1, 20.1, 19.7, 19.5. **HRMS** (ESI) calculated for $[C_{17}H_{24}O_5+Na]^+$ 331.1521, found 331.1512.



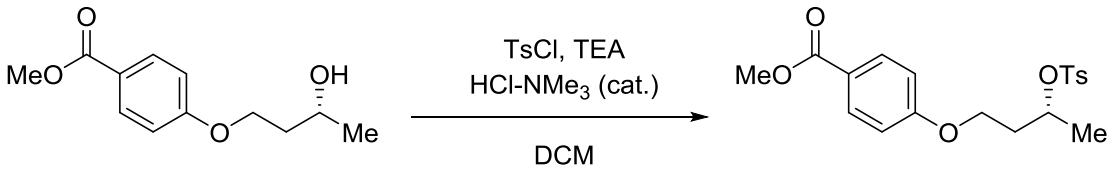
Methyl (*R*)-4-(3-hydroxybutoxy)benzoate (SI-12) was synthesized by subjecting **SI-11** (1.8 g, 5.8 mmol) to General Procedure C. The crude product was purified via flash chromatography in 40% EtOAc/hexanes to provide 1 g (77%) of **SI-12** as a clear oil. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 8.03 – 7.97 (m, 2H), 6.97 – 6.91 (m, 2H), 4.23 (ddd, *J* = 9.4, 7.2, 5.2 Hz, 1H), 4.20 – 4.09 (m, 2H), 3.90 (s, 3H), 2.03 – 1.89 (m, 2H), 1.31 (d, *J* = 6.2 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 166.9, 162.5, 131.6, 122.7, 114.1, 65.78, 65.75, 51.9, 38.0, 23.9. **HRMS** (ESI) calculated for $[C_{12}H_{16}O_4+Na]^+$ 247.0946, found 247.0943. **Chiral HPLC**: (Daicel OJ-H, 95:5 hexanes:isopropanol): *ee* = 98%.



Peak#	Ret. Time	Area	Area%
1	33.899	2524871	50.035
2	36.688	2521357	49.965
Total		5046228	100

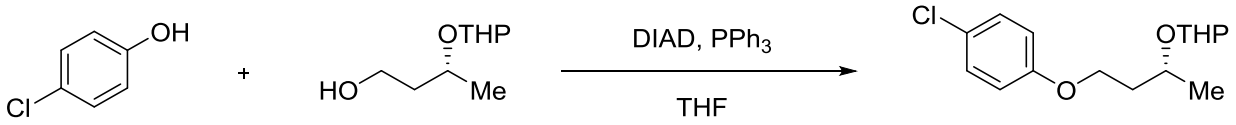


Peak#	Ret. Time	Area	Area%
1	33.997	29721952	98.83
2	36.957	352008	1.17
Total		30073960	100

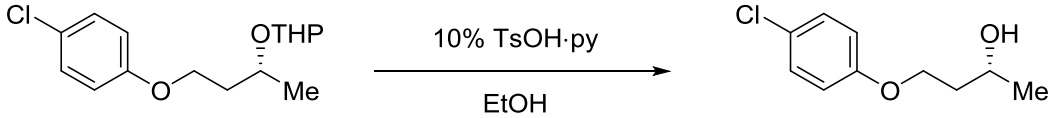


Methyl (*R*)-4-(3-(tosyloxy)butoxy)benzoate (SI-13) was synthesized by tosylating **SI-12** (1 g, 4.5 mmol) according to General Procedure A. The crude product was purified

via flash chromatography in 25% EtOAc/hexanes to provide 1.4 g (82%) of tosylate **SI-13** as a white solid. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.98 – 7.94 (m, 2H), 7.76 – 7.72 (m, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 6.72 – 6.67 (m, 2H), 4.87 (dqd, *J* = 12.6, 6.3, 3.8 Hz, 1H), 3.92 (s, 3H), 3.88 (dt, *J* = 9.6, 4.9 Hz, 1H), 3.72 (td, *J* = 9.1, 4.5 Hz, 1H), 2.25 (s, 3H), 2.09 – 1.97 (m, 2H), 1.48 (d, *J* = 6.3 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 166.8, 162.1, 144.7, 133.7, 131.4, 129.7, 127.6, 122.6, 113.8, 76.9, 63.2, 52.0, 36.0, 21.7, 21.6. **HRMS** (ESI) calculated for $[C_{20}H_{21}NO_5S+Na]^+$ 410.1038, found 410.1041.

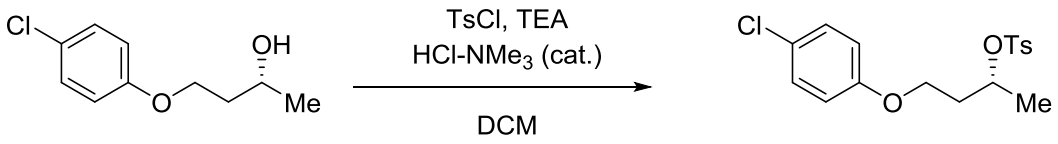


2-(((*R*)-4-(4-chlorophenoxy)butan-2-yl)oxy)tetrahydro-2H-pyran (SI-14) was synthesized by subjecting **SI-7** (2 g, 11.5 mmol) and 4-chlorophenol (1.5 g, 11.5 mmol) to General Procedure B. The crude product was purified via flash chromatography in 5% EtOAc/hexanes to provide 1.4 g (43%) of **SI-14** as a clear oil in a 1:1 mixture of diastereomers. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.28 – 7.21 (m, 4H), 6.89 – 6.81 (m, 4H), 4.75 (dd, *J* = 4.4, 2.9 Hz, 1H), 4.63 (dd, *J* = 5.0, 2.8 Hz, 1H), 4.21 – 3.91 (m, 7H), 3.78 (ddd, *J* = 11.5, 8.2, 3.3 Hz, 1H), 3.55 – 3.41 (m, 2H), 2.06 – 1.89 (m, 4H), 1.86 – 1.77 (m, 2H), 1.77 – 1.64 (m, 2H), 1.63 – 1.42 (m, 8H), 1.33 (d, *J* = 6.3 Hz, 3H), 1.21 (d, *J* = 6.2 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 157.6, 157.5, 129.3, 129.2, 125.4, 125.2, 115.8, 115.7, 99.5, 95.5, 71.4, 67.8, 65.1, 64.7, 63.0, 62.4, 36.8, 36.3, 31.15, 31.12, 25.5, 25.4, 22.1, 20.1, 19.7, 19.4. **HRMS** (ESI) calculated for $[C_{15}H_{21}ClO_3+Na]^+$ 307.1077, found 307.1070.

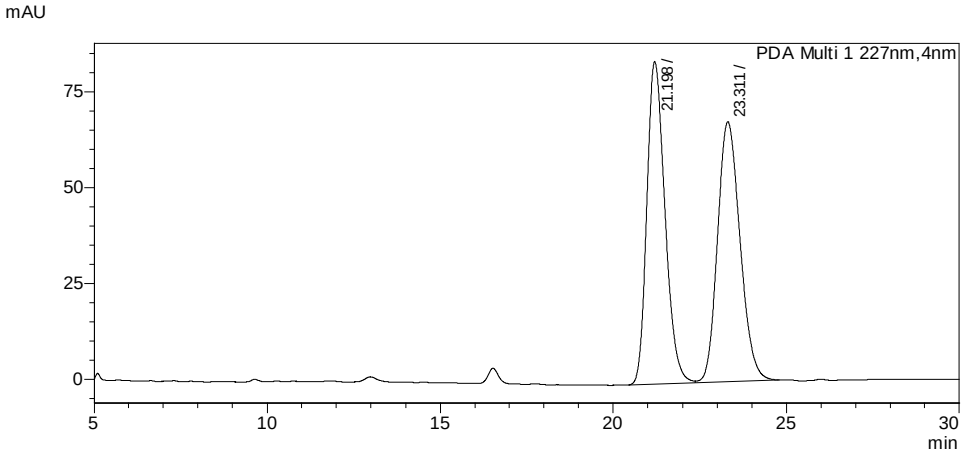


(*R*)-4-(4-chlorophenoxy)butan-2-ol (SI-15) was synthesized by subjecting **SI-14** (1.4 g, 4.9 mmol) to General Procedure C. The crude product was purified via flash chromatography in 25% EtOAc/hexanes to provide 0.8 g (81%) of **SI-15** as a clear oil. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.28 – 7.22 (m, 2H), 6.90 – 6.81 (m, 2H), 4.22 – 4.01 (m, 3H), 2.00 – 1.87 (m, 3H), 1.30 (d, *J* = 6.2 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$)

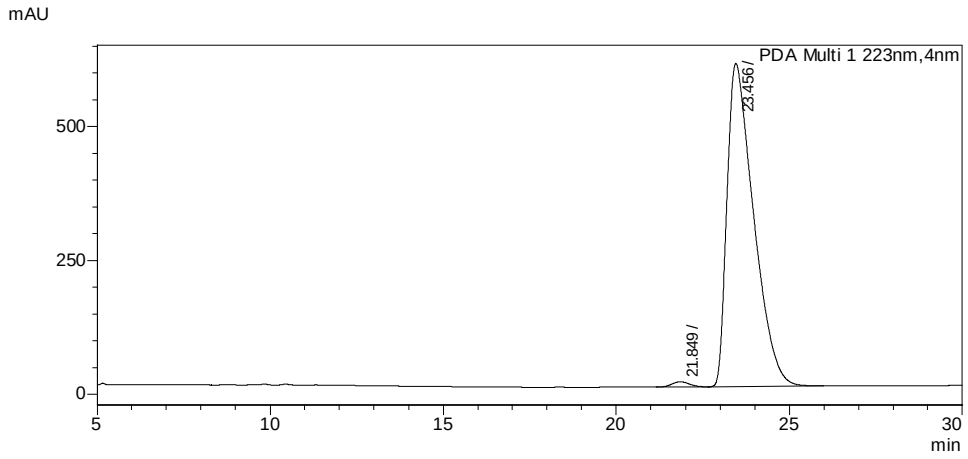
δ 157.3, 129.4, 125.7, 115.7, 66.08, 66.07, 38.0, 23.8. **HRMS** (ESI) calculated for $[C_{10}H_{13}ClNaO_2+Na]^+$ 223.0502, found 223.0497.



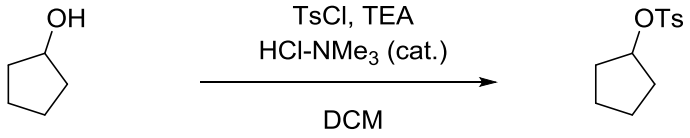
(*R*)-4-(4-chlorophenoxy)butan-2-yl 4-methylbenzenesulfonate (SI-16) was synthesized by tosylating **SI-15** (0.8 g, 4 mmol) according to General Procedure A. The crude product was purified via flash chromatography in 20% EtOAc/hexanes to provide 1.2 g (85%) of tosylate **SI-16** as a white solid. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.74 (d, *J* = 8.3 Hz, 2H), 7.22 – 7.17 (m, 2H), 7.16 – 7.13 (m, 2H), 6.63 – 6.58 (m, 2H), 4.87 (dqd, *J* = 8.8, 6.3, 3.8 Hz, 1H), 3.79 (dt, *J* = 9.5, 4.9 Hz, 1H), 3.63 (td, *J* = 9.1, 4.5 Hz, 1H), 2.30 (s, 3H), 2.07 – 1.93 (m, 2H), 1.46 (d, *J* = 6.3 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 157.0, 144.6, 133.7, 129.7, 129.1, 127.5, 125.5, 115.4, 63.2, 36.1, 21.7, 21.6. **HRMS** (ESI) calculated for $[C_{17}H_{19}ClO_4S+Na]^+$ 377.0590, found 377.0583. **Chiral HPLC**: (Daicel OJ-H, 80:20 hexanes:isopropanol): *ee* = 98%.



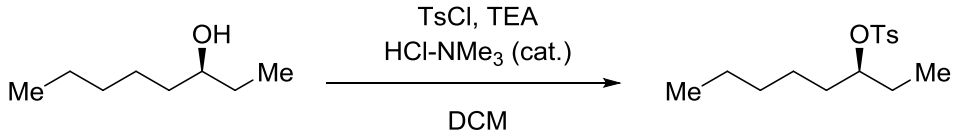
Peak#	Ret. Time	Area	Area%
1	21.198	3060132	49.985
2	23.311	3061943	50.015
Total		6122074	100



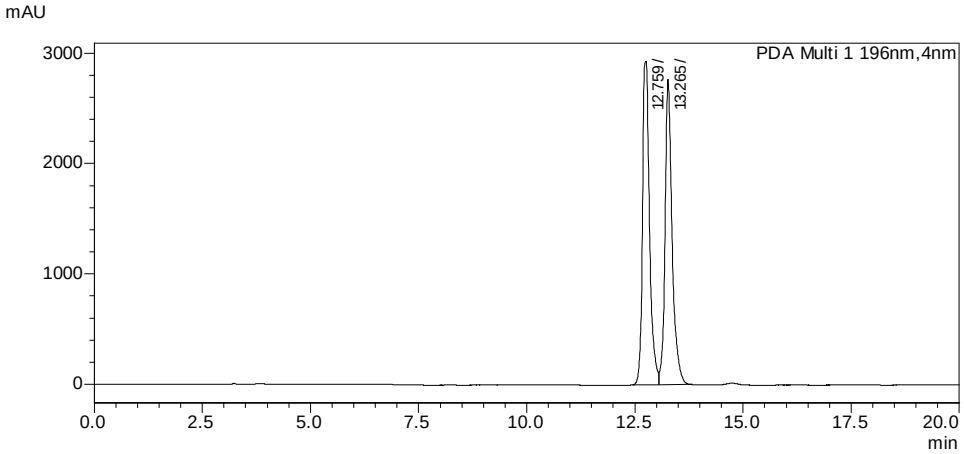
Peak#	Ret. Time	Area	Area%
1	21.849	340129	1.045
2	23.456	32205214	98.955
Total		32545344	100



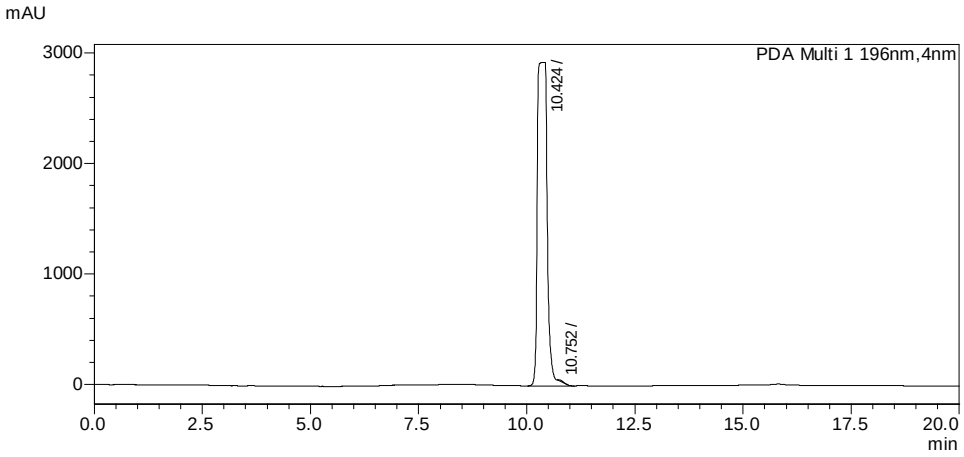
Cyclopentyl 4-methylbenzenesulfonate (SI-17) was synthesized by tosylating cyclopentanol (1 g, 11.6 mmol) according to General Procedure A. The crude product was purified via flash chromatography in 10% EtOAc/hexanes to provide 2.7 g (95%) of tosylate **SI-17** as a white solid. Physical and spectral data were in accordance with literature data.[6]



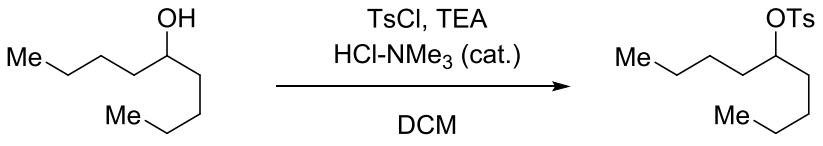
(S)-octan-3-yl 4-methylbenzenesulfonate (SI-18) was synthesized by tosylating (*S*)-3-octanol (1 mL, 6.3 mmol) according to General Procedure A. The crude product was purified via flash chromatography in 5% EtOAc/hexanes to provide 1.5 g (84%) of tosylate **SI-18** as a colorless oil. Physical and spectral data were in accordance with literature data.[2,7] **Chiral HPLC**: Chiralpak IF, 99:1 hexanes:isopropanol): *ee* = 99%.



Peak#	Ret. Time	Area	Area%
1	12.759	33274686	51.421
2	13.265	31435748	48.579
Total		64710435	100

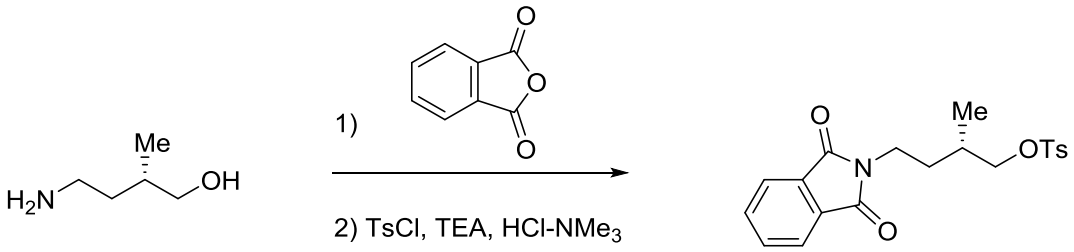


Peak#	Ret. Time	Area	Area%
1	10.327	13262788	99.632
2	10.763	48935	0.368
Total		13311723	100

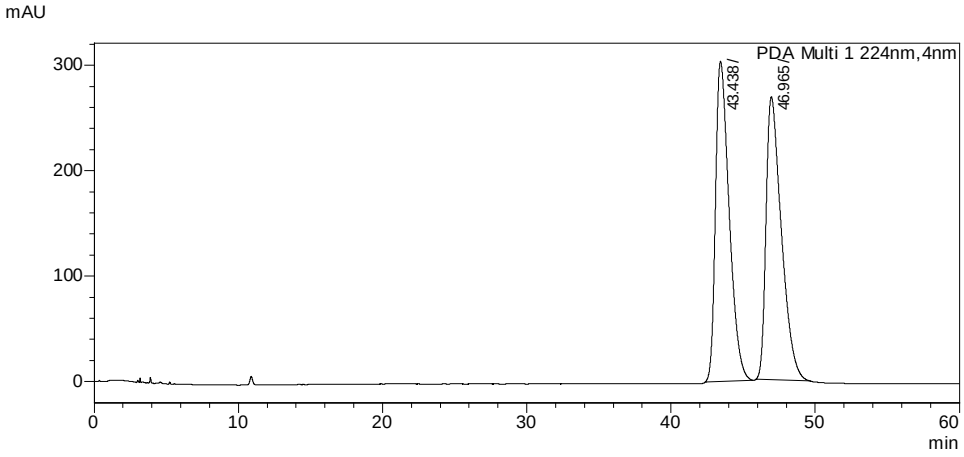


Nonan-5-yl 4-methylbenzenesulfonate (SI-19) was synthesized by tosylating nonan-5-ol (1.2 g, 6.9 mmol) according to General Procedure A. The crude product was

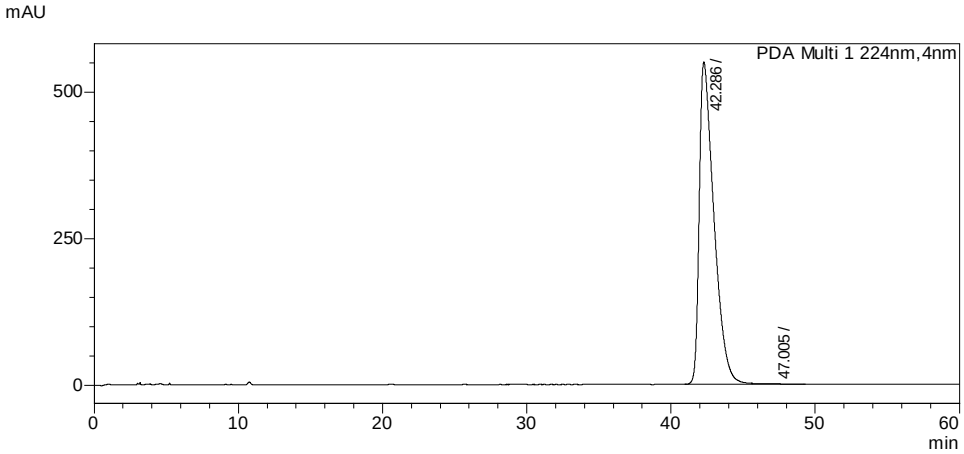
purified via flash chromatography in 5% EtOAc/hexanes to provide 2.3 g (90%) of **SI-19** as a colorless liquid. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.83 – 7.78 (m, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 4.55 (p, *J* = 6.0 Hz, 1H), 2.45 (s, 3H), 1.61 – 1.52 (m, 4H), 1.28 – 1.10 (m, 8H), 0.82 (t, *J* = 7.0 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 144.4, 134.7, 129.6, 127.7, 84.6, 33.8, 26.8, 22.4, 21.6, 13.9. **HRMS** (ESI) calculated for $[C_{16}H_{26}O_3S+Na]^+$ 321.1500, found 321.1496.



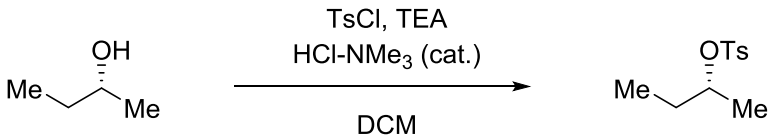
(*S*)-4-(1,3-dioxoisoindolin-2-yl)-2-methylbutyl 4-methylbenzenesulfonate (SI-20) was synthesized by refluxing (*S*)-4-amino-2-methylbutan-1-ol (1 g, 9.7 mmol) and succinic anhydride (1.3 g, 8.8 mmol) at 165 ºC for 2 hours. The reaction was cooled to room temperature and brought up in DCM. The organic layer was washed three times with water, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude alcohol (1 g, 4.3 mmol) was tosylated according to General Procedure A. The crude product was purified via flash chromatography in 40% EtOAc/hexanes to provide 1.2 g (72%) of tosylate **SI-20** as a pale yellow, viscous oil. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.83 – 7.78 (m, 2H), 7.78 – 7.69 (m, 2H), 7.41 – 7.33 (m, 2H), 3.95 (dd, *J* = 9.5, 5.9 Hz, 1H), 3.90 (dd, *J* = 9.5, 5.5 Hz, 1H), 3.68 (t, *J* = 7.3 Hz, 2H), 2.46 (s, 3H), 1.90 – 1.82 (m, 1H), 1.78 (dtd, *J* = 13.1, 7.4, 5.5 Hz, 1H), 1.49 (ddt, *J* = 13.8, 8.3, 7.0 Hz, 1H), 1.02 (d, *J* = 6.7 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 168.3, 144.8, 134.0, 132.8, 132.0, 129.9, 128.0, 123.3, 74.3, 35.5, 31.6, 30.7, 21.7, 16.2. **HRMS** (ESI) calculated for $[C_{19}H_{22}O_6S+Na]^+$ 401.1035, found 401.1024. **Chiral HPLC**: (Chiralpak IF, 80:20 hexanes:isopropanol): *ee* = 99%



Peak#	Ret. Time	Area	Area%
1	43.438	19958455	50.121
2	46.965	19862318	49.879
Total		39820773	100

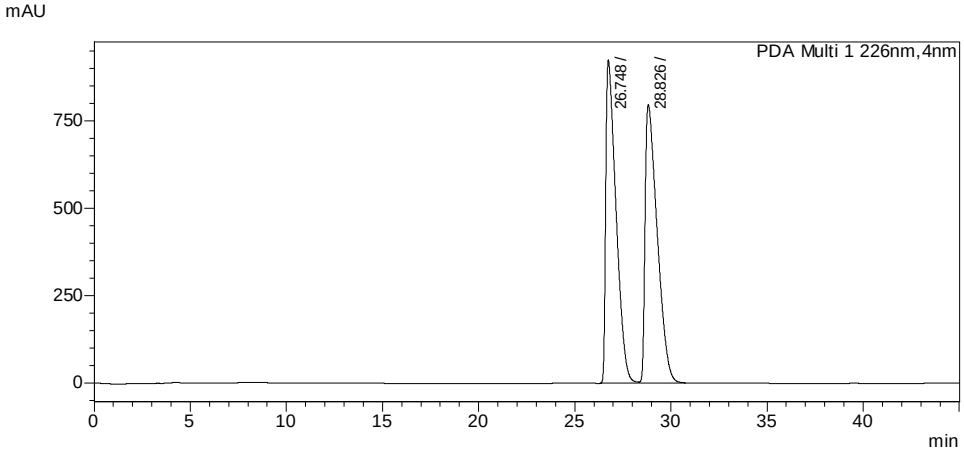


Peak#	Ret. Time	Area	Area%
1	42.286	41559070	99.587
2	47.005	172327	0.413
Total		41731397	100

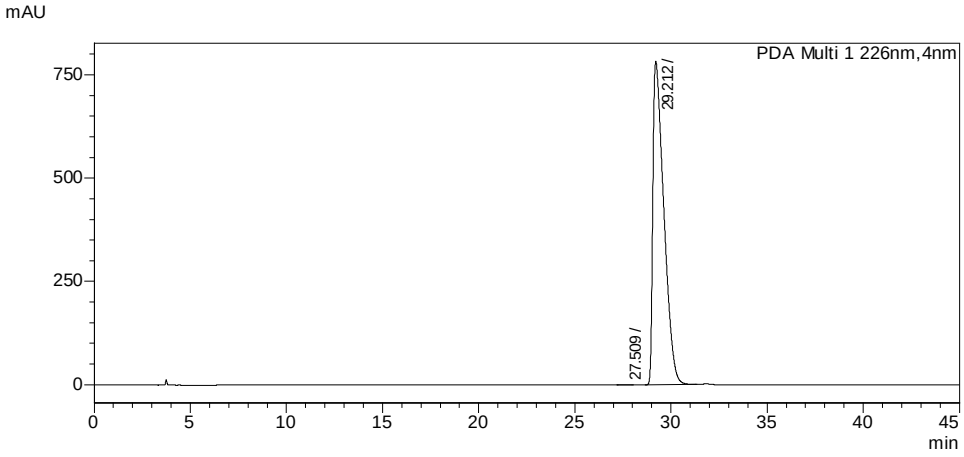


(*R*)-*sec*-butyl 4-methylbenzenesulfonate (21) was synthesized by tosylating (*R*)-*sec*-butanol (0.5 g, 6.7 mmol) according to General Procedure A. The crude product was

purified via flash chromatography in 10% EtOAc/hexanes to provide 1.5 g (98%) of tosylate **21** as a colorless oil. Physical and spectral data were in accordance with literature data.[8,9] **Chiral HPLC**: (Chiralpak IE, 99:1 hexanes:isopropanol): *ee* = >99%.

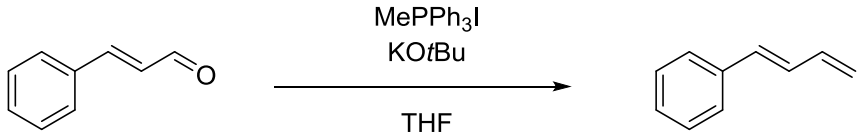


Peak#	Ret. Time	Area	Area%
1	26.748	34382243	49.627
2	28.826	34899591	50.373
Total		69281833	100

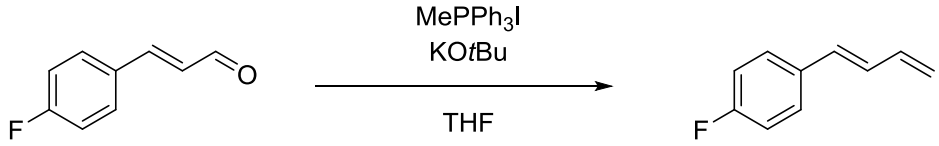


Peak#	Ret. Time	Area	Area%
1	27.497	19265	0.059
2	29.212	32464325	99.941
Total		32483589	100

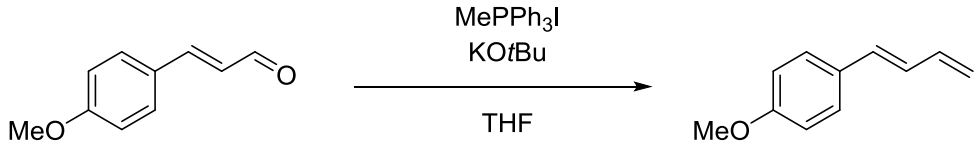
Dienes:



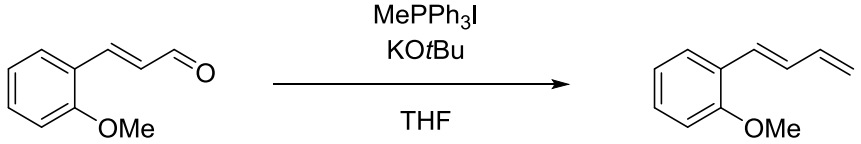
(*E*)-buta-1,3-dien-1-ylbenzene (SI-21) was synthesized by olefinating *trans*-cinnamaldehyde (52.9 g, .4 mol) according to General Procedure D. The crude product was distilled (1 torr, 75 ºC) to provide 21.5 g (41%) of diene **SI-21** as a pale yellow liquid. Physical and spectral data were in accordance with literature data.[10]



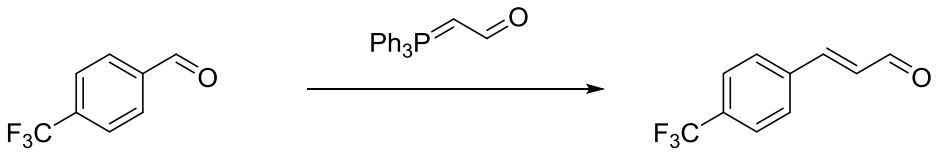
(*E*)-buta-1,3-dien-1-ylbenzene (SI-22) was synthesized by olefinating *trans-p*-fluorocinnamaldehyde (2.5 g, 16.7 mmol) according to General Procedure D. The crude product was purified via flash chromatography in hexanes to provide 1.2 g (50%) of diene **SI-22** as a colorless liquid. Physical and spectral data were in accordance with literature data.[11]



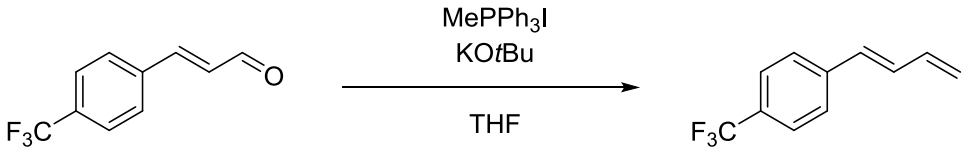
(*E*)-1-(buta-1,3-dien-1-yl)-4-methoxybenzene (SI-23) was synthesized by olefinating *trans-p*-methoxycinnamaldehyde (2 g, 12.3 mmol) according to General Procedure D. The crude product was purified via flash chromatography in 5% EtOAc/hexanes to provide 1.44 g (74%) of diene **SI-23** as a slightly yellow liquid. Physical and spectral data were in accordance with literature data.[12]



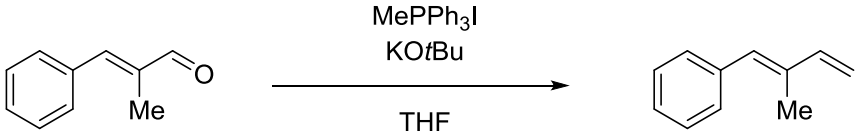
(*E*)-1-(buta-1,3-dien-1-yl)-2-methoxybenzene (SI-24) was synthesized by olefinating *trans*-*o*-methoxycinnamaldehyde (5.0 g, 30.8 mmol) according to General Procedure D. The crude product was purified via flash chromatography in hexanes to provide 3.5 g (71%) of diene **SI-24** as a pale yellow liquid. Physical and spectral data were in accordance with literature data.[12]



(*E*)-3-(4-(trifluoromethyl)phenyl)acrylaldehyde (SI-25) was synthesized by olefinating 4-(trifluoromethyl)benzaldehyde (4.1 g, 23 mmol) according to General Procedure D. The crude product was purified via flash chromatography in 5% EtOAc/hexanes to provide 1.6 g (34%) of cinnamaldehyde derivative **SI-25** as a white solid. Physical and spectral data were in accordance with literature data.[17]

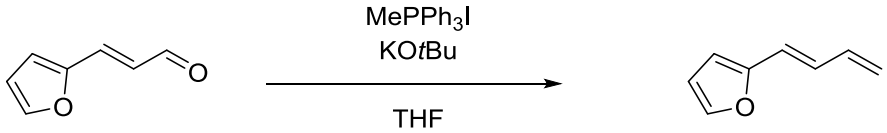


(*E*)-1-(buta-1,3-dien-1-yl)-4-(trifluoromethyl)benzene (SI-26) was synthesized by olefinating **SI-25** (1.6 g, 8 mmol) according to General Procedure D. The crude product was purified via flash chromatography in 1% EtOAc/hexanes to provide 1 g (63%) of diene **SI-26** as a colorless, refractive liquid. Physical and spectral data were in accordance with literature data.[18]

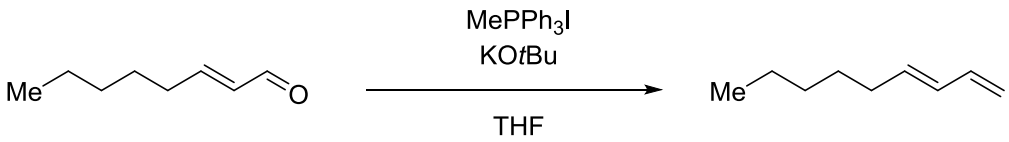


(*E*)-(2-methylbuta-1,3-dien-1-yl)benzene (SI-27) was synthesized by olefinating α-methyl-*trans*-cinnamaldehyde (9.5 mL, 68 mmol) according to General Procedure D. The crude product was purified via flash chromatography in hexanes to provide 8 g

(81%) of diene **SI-27** as a colorless liquid. Physical and spectral data were in accordance with literature data.[13]



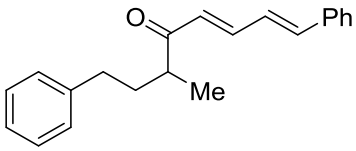
(*E*)-2-(buta-1,3-dien-1-yl)furan (SI-28) was synthesized by olefinating *trans*-3-(2-furyl)acrolein (3.0 g, 24.6 mmol) according to General Procedure D. The crude product was purified via flash chromatography in hexanes to provide 1.5 g (50%) of diene **SI-28** as a yellow liquid. Physical and spectral data were in accordance with literature data.[14]



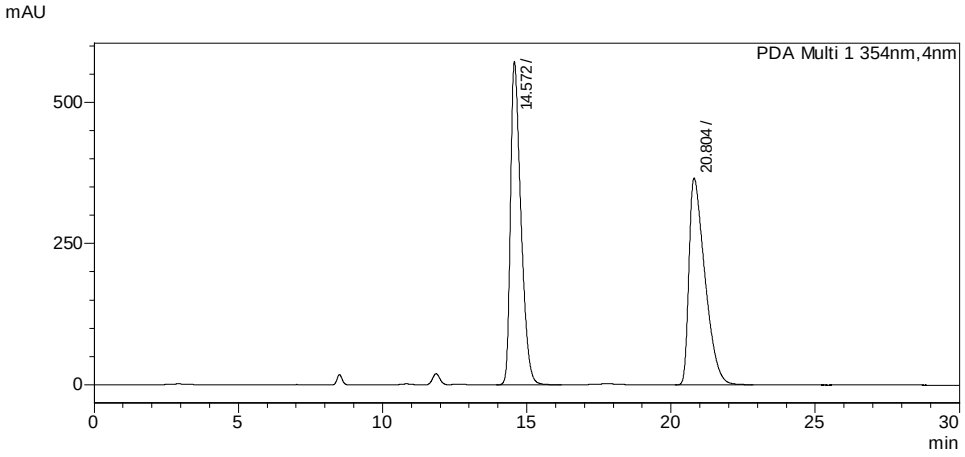
(*E*)-nona-1,3-diene (SI-29) was synthesized by olefinating *trans*-2-octenal (3.4 g, 27 mmol) according to General Procedure D. The crude product was purified via flash chromatography in hexanes to provide 1.2 g (36%) of diene **SI-29** as a colorless liquid. Physical and spectral data were in accordance with literature data.[15,16]

Cobalt-Catalyzed Reactions

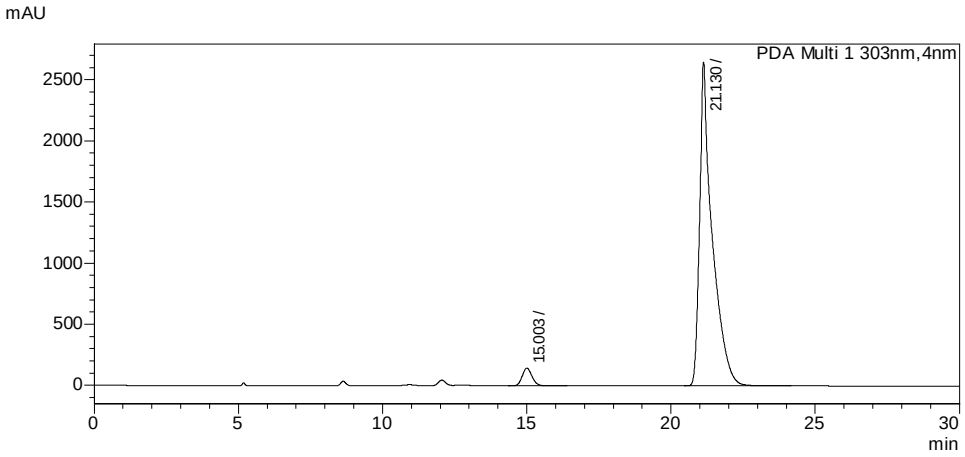
General Carbonylation Procedure A: In a glovebox under an argon atmosphere, alkyl tosylate (0.6 mmol) was combined with $K[Co(CO)_4]$ (10.5 mg, 0.05 mmol), TMP (93 μL, 0.55 mmol), diene (0.5 mmol), *t*-amyl alcohol (1 mL) in an Ace Glass pressure tube. The tube was sealed with a Swagelok gas quick-connect adapter and removed from the glovebox. Subsequently, the tube was purged 3 times with 5 atm CO and then the pressure was set to 2 atm CO. The reaction was stirred for 24 hours at 70 ºC. The reaction mixture was cooled to room temperature, depressurized, allowed to stir open for two hours to decompose the cobalt complex, and then 3 mL HCl (1 M) and 3 mL Et_2O were added. The organic layer was separated and the aqueous layer was extracted twice with Et_2O. The combined organic layers were filtered through a plug of SiO_2, eluting with Et_2O, and concentrated under reduced pressure. The crude product was purified by flash chromatography.



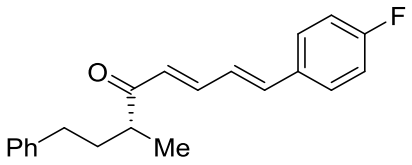
(5*E*,7*E*)-3-methyl-1,8-diphenylocta-5,7-dien-4-one (3) was obtained from General Carbonylation Procedure A and the crude product (84% NMR yield) was flashed in 5% EtOAc/hexanes yielding **3** as a colorless oil (110 mg, 76%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.53 – 7.48 (m, 2H), 7.43 – 7.29 (m, 6H), 7.26 – 7.17 (m, 3H), 6.99 – 6.87 (m, 2H), 6.35 (d, *J* = 15.3 Hz, 1H), 2.83 (h, *J* = 6.9 Hz, 1H), 2.65 (t, *J* = 7.8 Hz, 2H), 2.15 – 2.05 (m, 1H), 1.77 – 1.68 (m, 1H), 1.20 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 203.7, 142.8, 141.9, 141.4, 136.1, 129.2, 128.9, 128.5, 128.4, 128.4, 127.2, 126.8, 125.9, 43.5, 34.9, 33.5, 16.8. **HRMS** (ESI) calculated for $[C_{21}H_{22}O+H]^+$ 291.1749, found 291.1747. **Chiral HPLC**: (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 92%, *es* = 93%.



Peak#	Ret. Time	Area	Area%
1	14.572	14430038	50.622
2	20.804	14075465	49.378
Total		28505502	100

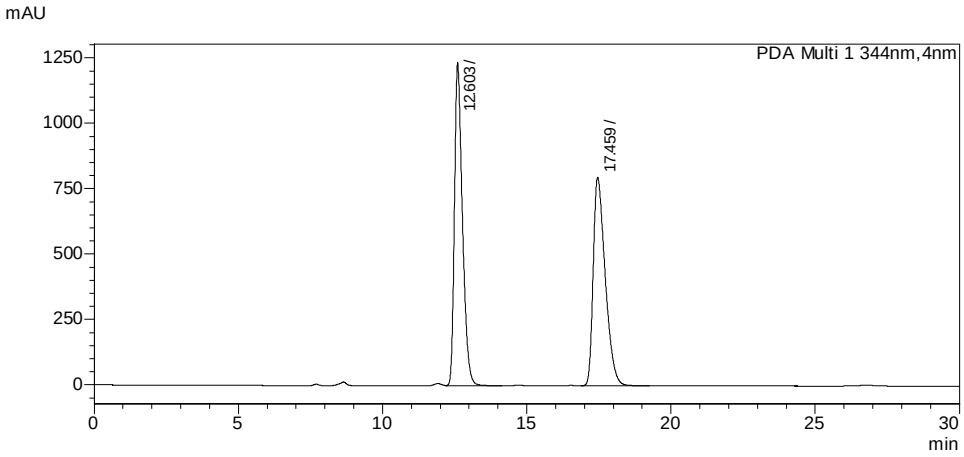


Peak#	Ret. Time	Area	Area%
1	15.003	3474294	4.15
2	21.13	80238800	95.85
Total		83713094	100

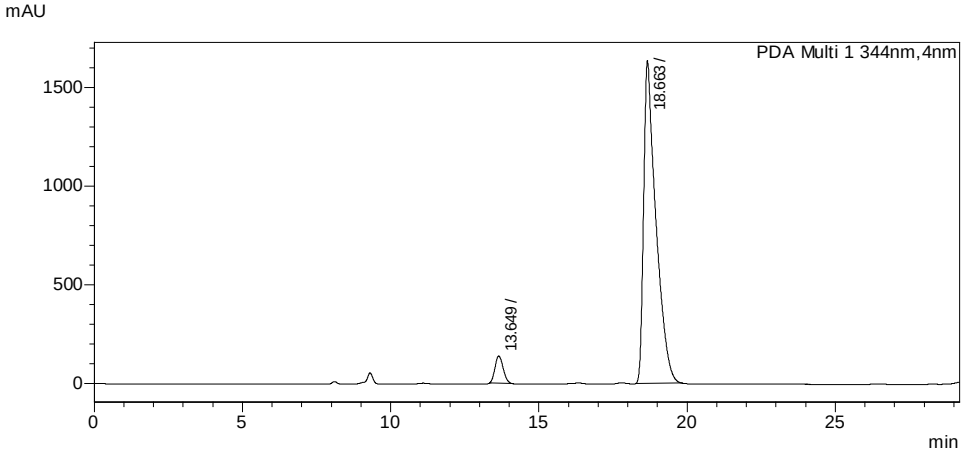


(*R*,5*E*,7*E*)-8-(4-fluorophenyl)-3-methyl-1-phenylocta-5,7-dien-4-one (4) was obtained from General Carbonylation Procedure A and the crude product was flashed in 5%

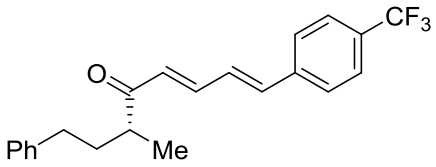
EtOAc/hexanes yielding **4** as a lemon yellow solid (110 mg, 71%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.51 – 7.42 (m, 2H), 7.38 – 7.26 (m, 3H), 7.28 – 7.18 (m, 3H), 7.09 (t, *J* = 8.6 Hz, 2H), 6.91 (d, *J* = 15.5 Hz, 1H), 6.83 (dd, *J* = 15.5, 10.7 Hz, 1H), 6.35 (d, *J* = 15.3 Hz, 1H), 2.83 (h, *J* = 6.9 Hz, 1H), 2.66 (t, *J* = 7.8 Hz, 2H), 2.15 – 2.06 (m, 1H), 1.78 – 1.69 (m, 1H), 1.21 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 203.6, 163.16 (d, *J* = 250.2 Hz), 142.5, 141.9, 140.0, 132.34 (d, *J* = 3.5 Hz), 128.93 (d, *J* = 8.2 Hz), 128.5, 128.43, 128.37, 126.52 (d, *J* = 2.6 Hz), 125.9, 115.96 (d, *J* = 21.7 Hz), 43.6, 34.9, 33.5, 16.8. **HRMS** (ESI) calculated for $[C_{21}H_{21}FO+Na]^+$ 331.1474, found 331.1475. **Chiral HPLC** (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 89%, *es* = 90%.



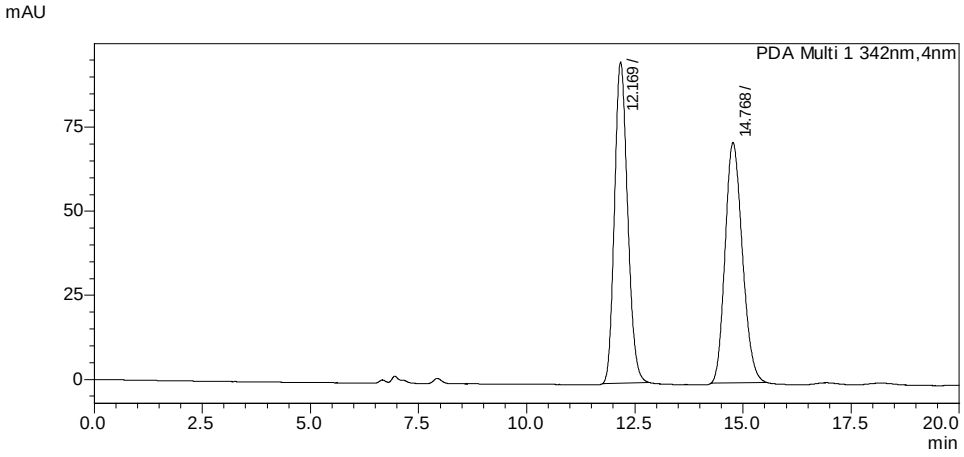
Peak#	Ret. Time	Area	Area%
1	12.603	23815809	50.805
2	17.459	23060992	49.195
Total		46876801	100



Peak#	Ret. Time	Area	Area%
1	13.649	2650631	5.343
2	18.663	46962888	94.657
Total		49613519	100

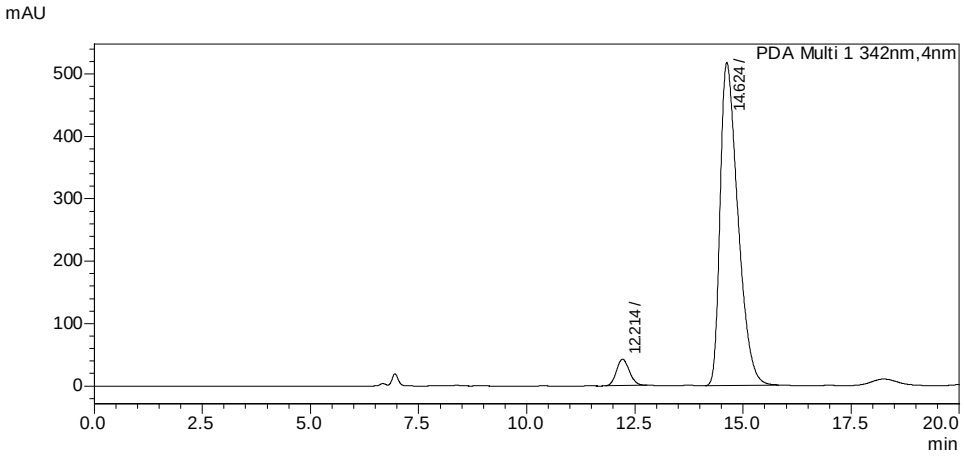


(*R*,5*E*,7*E*)-3-methyl-1-phenyl-8-(4-(trifluoromethyl)phenyl)octa-5,7-dien-4-one (5) was obtained from General Carbonylation Procedure A and the crude product was flashed in 5% EtOAc/hexanes yielding **5** as a lemon chiffon yellow solid (123 mg, 69%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.64 (d, *J* = 8.2 Hz, 2H), 7.59 (d, *J* = 8.2 Hz, 2H), 7.38 – 7.26 (m, 3H), 7.29 – 7.15 (m, 3H), 7.02 – 6.85 (m, 2H), 6.40 (d, *J* = 15.2 Hz, 1H), 2.82 (h, *J* = 6.9 Hz, 1H), 2.65 (t, *J* = 7.8 Hz, 2H), 2.14 – 2.04 (m, 1H), 1.78 – 1.67 (m, 1H), 1.20 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 203.5, 141.81, 141.78, 139.4, 139.3, 130.5 (q, *J* = 32.5 Hz), 129.6, 129.1, 128.50, 128.45, 127.3, 126.0, 125.8 (q, *J* = 3.8 Hz), 124.0 (q, *J* = 272.1 Hz), 43.7, 34.8, 33.4, 16.7. **HRMS** (ESI) calculated for $[C_{22}H_{21}F_3O+Na]^+$ 381.1442, found 381.1442. **Chiral HPLC** (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 89%, *es* = 90%.

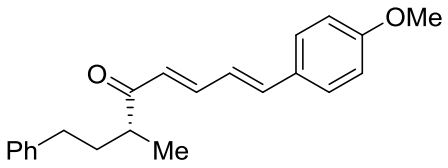


Peak#	Ret. Time	Area	Area%
1	12.169	2012953	50.302

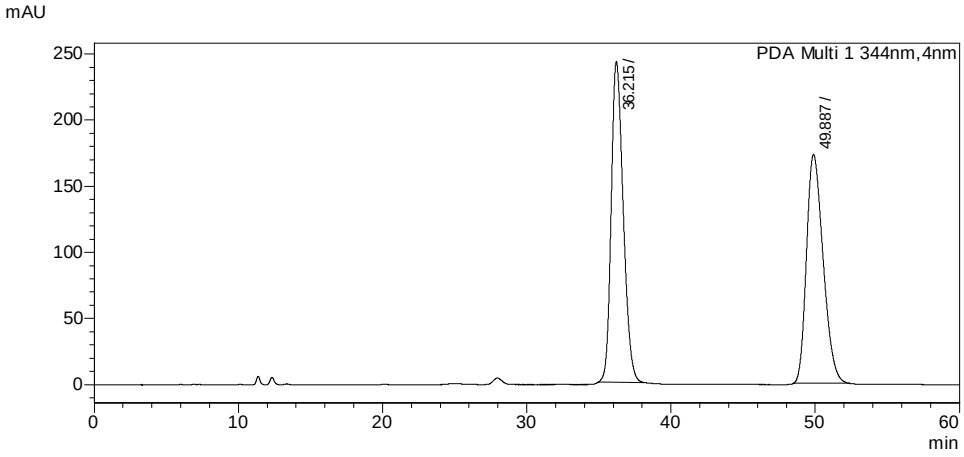
2	14.768	1988751	49.698
Total		4001703	100



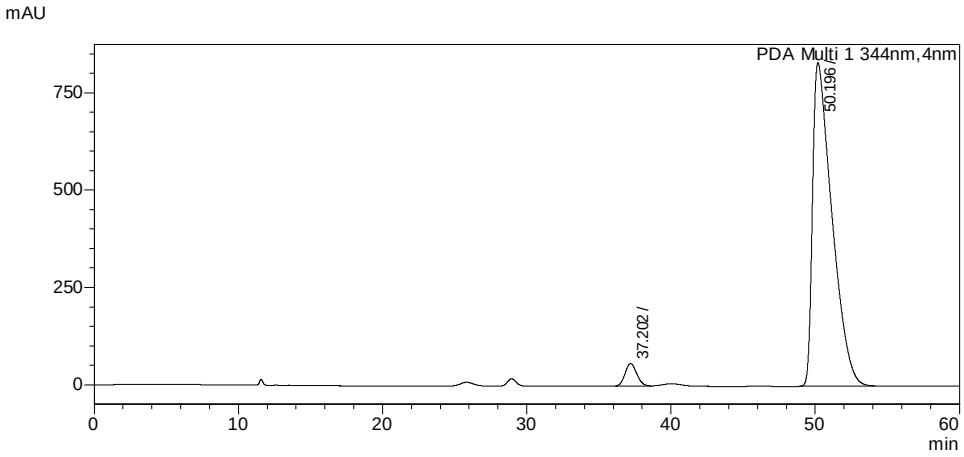
Peak#	Ret. Time	Area	Area%
1	12.214	868313	5.559
2	14.624	14750751	94.441
Total		15619064	100



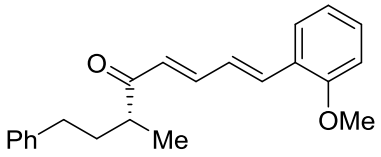
(*R*,5*E*,7*E*)-8-(4-methoxyphenyl)-3-methyl-1-phenylocta-5,7-dien-4-one (6) was obtained from General Carbonylation Procedure A and the crude product was flashed in 10% EtOAc/hexanes yielding **6** as a yellow solid (124 mg, 77%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.49 – 7.43 (m, 2H), 7.39 – 7.29 (m, 3H), 7.30 – 7.20 (m, 3H), 6.96 – 6.88 (m, 3H), 6.80 (dd, *J* = 15.5, 10.9 Hz, 1H), 6.32 (d, *J* = 15.2 Hz, 1H), 3.86 (s, 3H), 2.84 (h, *J* = 6.9 Hz, 1H), 2.67 (dt, *J* = 9.4, 7.3 Hz, 2H), 2.11 (ddt, *J* = 14.0, 8.6, 7.2 Hz, 1H), 1.74 (ddt, *J* = 13.5, 8.3, 6.7 Hz, 1H), 1.21 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 203.7, 160.5, 143.3, 142.0, 141.3, 128.9, 128.8, 128.5, 128.4, 127.3, 125.9, 124.7, 114.3, 55.4, 43.5, 35.0, 33.5, 16.9. **HRMS** (ESI) calculated for $[C_{22}H_{24}O_2+Na]^+$ 343.1674, found 343.1675. **Chiral HPLC** (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 92%, *es* = 93%.



Peak#	Ret. Time	Area	Area%
1	36.215	14045644	50.283
2	49.887	13887450	49.717
Total		27933094	100

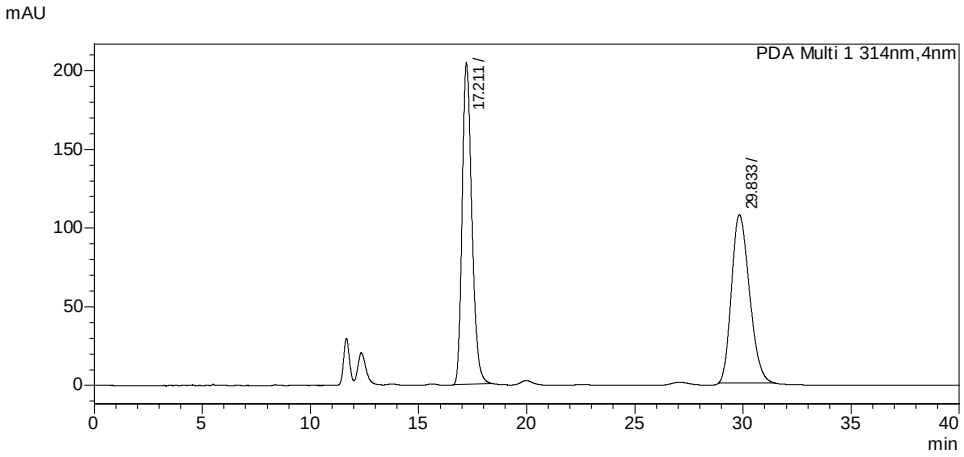


Peak#	Ret. Time	Area	Area%
1	37.202	3200494	3.962
2	50.196	77577343	96.038
Total		80777837	100

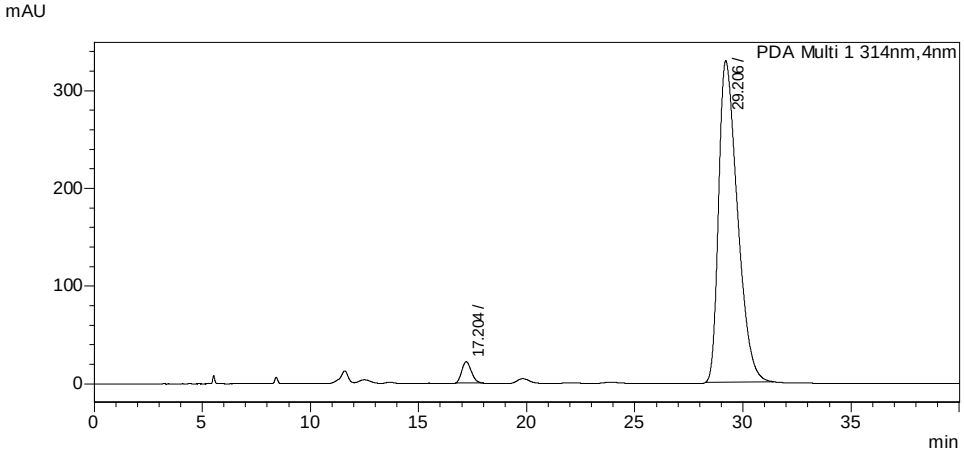


(*R*,5*E*,7*E*)-8-(2-methoxyphenyl)-3-methyl-1-phenylocta-5,7-dien-4-one (7) was obtained from General Carbonylation Procedure A and the crude product was flashed in

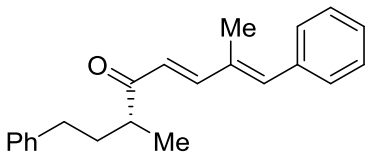
10% EtOAc/hexanes yielding **7** as a yellow oil (121 mg, 76%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.54 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.38 – 7.30 (m, 5H), 7.23 – 7.19 (m, 3H), 7.03 – 6.95 (m, 2H), 6.95 – 6.91 (m, 1H), 6.31 (d, *J* = 15.4 Hz, 1H), 3.92 (s, 3H), 2.89 – 2.81 (m, 1H), 2.64 (t, *J* = 7.8 Hz, 2H), 2.15 – 2.04 (m, 1H), 1.72 (ddt, *J* = 13.7, 8.3, 7.0 Hz, 1H), 1.19 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 203.9, 157.5, 143.9, 141.9, 136.7, 130.3, 128.5, 128.4, 127.9, 127.4, 127.3, 125.9, 125.0, 120.8, 111.1, 55.5, 43.2, 35.0, 33.5, 16.9. **HRMS** (ESI) calculated for $[C_{22}H_{24}O_2+Na]^+$ 343.1674, found 343.1676. **Chiral HPLC** (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 94%, *es* = 95%.



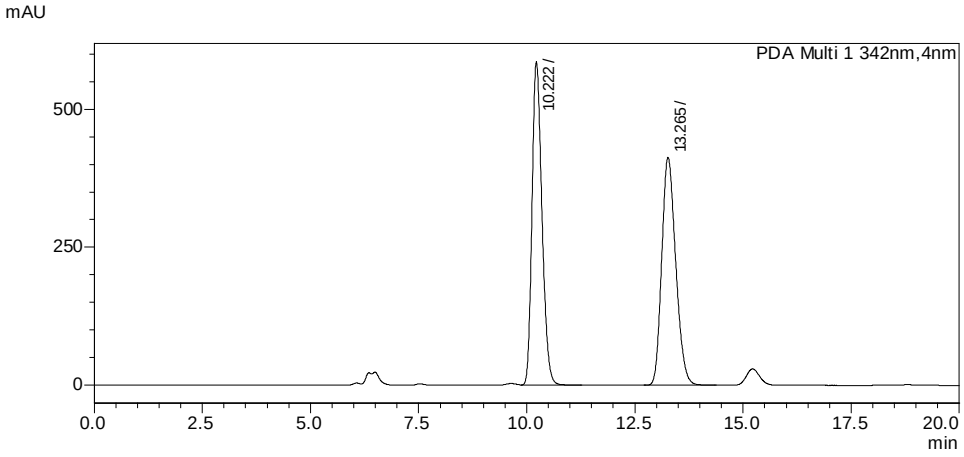
Peak#	Ret. Time	Area	Area%
1	17.211	6355211	50.715
2	29.833	6176018	49.285
Total		12531228	100



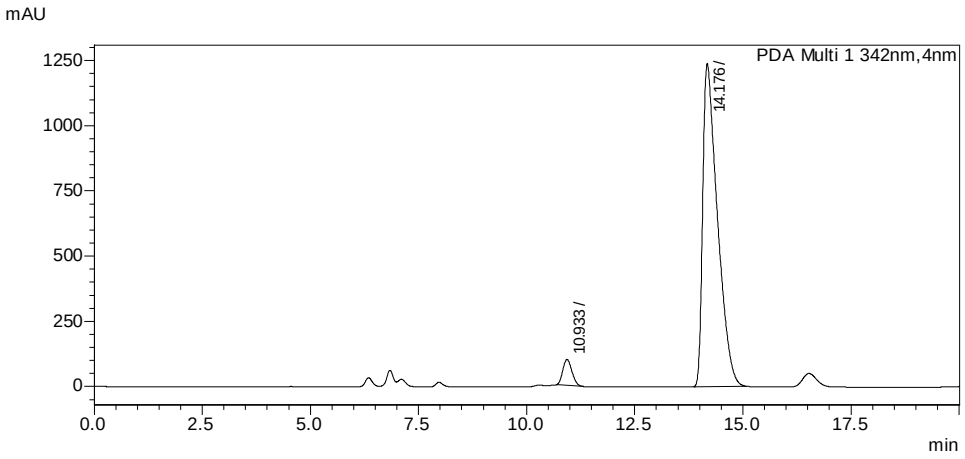
Peak#	Ret. Time	Area	Area%
1	17.204	663840	3.233
2	29.206	19870757	96.767
Total		20534597	100



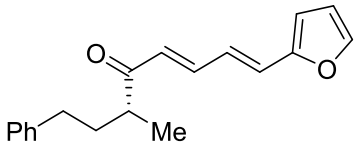
(*R*,5*E*,7*E*)-3,7-dimethyl-1,8-diphenylocta-5,7-dien-4-one (8) was obtained from General Carbonylation Procedure A and the crude product was flashed in 5% EtOAc/hexanes yielding **8** as a colorless oil (93 mg, 61%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.46 – 7.37 (m, 5H), 7.37 – 7.31 (m, 3H), 7.33 – 7.22 (m, 3H), 6.90 (s, 1H), 6.34 (d, *J* = 15.7 Hz, 1H), 2.91 (h, *J* = 6.9 Hz, 1H), 2.68 (t, *J* = 7.8 Hz, 2H), 2.19 – 2.08 (m, 4H), 1.81 – 1.72 (m, 1H), 1.24 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 203.9, 148.1, 142.0, 140.0, 136.7, 134.5, 129.5, 128.5, 128.44, 128.41, 127.9, 126.0, 124.6, 43.4, 35.0, 33.5, 16.9, 13.9. **HRMS** (ESI) calculated for $[C_{22}H_{24}O+Na]^+$ 327.1725, found 327.1730. **Chiral HPLC** (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 91%, *es* = 92%.



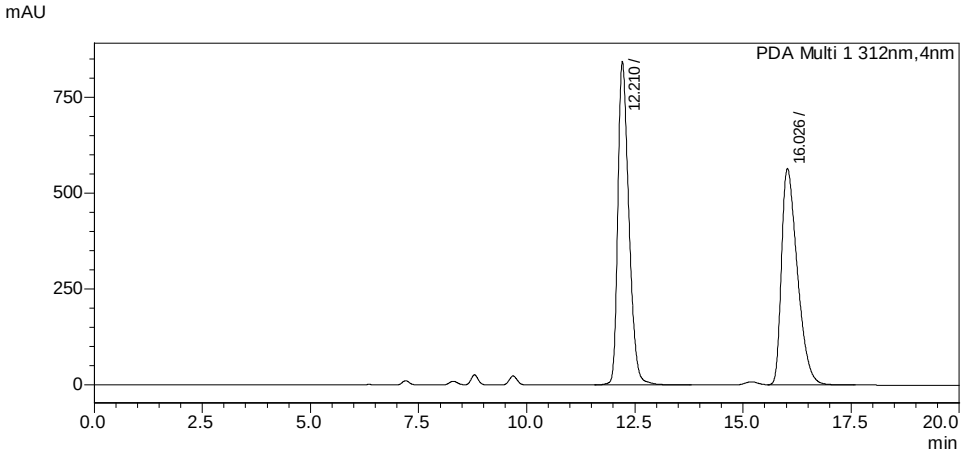
Peak#	Ret. Time	Area	Area%
1	10.222	9377357	50.183
2	13.265	9308938	49.817
Total		18686295	100



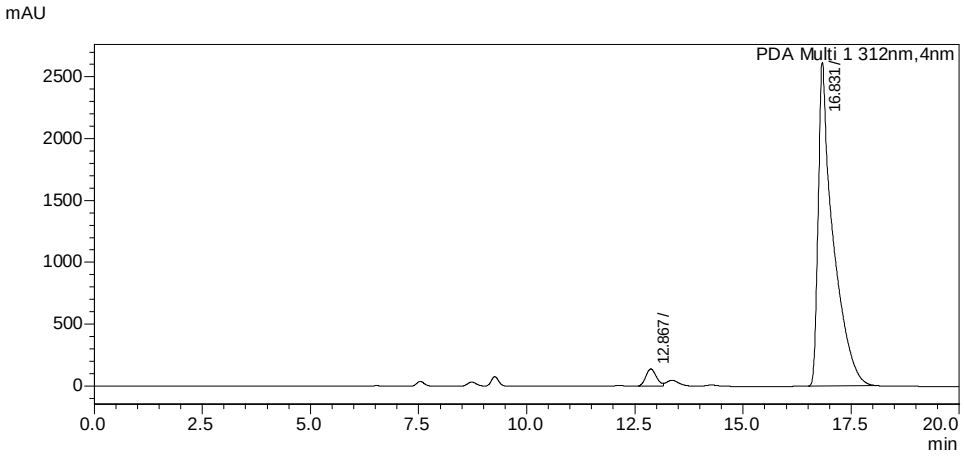
Peak#	Ret. Time	Area	Area%
1	10.933	1453848	4.738
2	14.176	29228244	95.262
Total		30682092	100



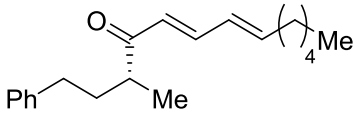
(*R*,5*E*,7*E*)-8-(furan-2-yl)-3-methyl-1-phenylocta-5,7-dien-4-one (9) was obtained from General Carbonylation Procedure A and the crude product was flashed in 10% EtOAc/hexanes yielding **9** as a dark yellow liquid (115 mg, 82%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.47 (d, *J* = 1.7 Hz, 1H), 7.32 (t, *J* = 7.3 Hz, 2H), 7.31 – 7.25 (m, 1H), 7.21 (t, *J* = 7.4 Hz, 3H), 6.81 (dd, *J* = 15.4, 11.2 Hz, 1H), 6.71 (d, *J* = 15.4 Hz, 1H), 6.52 – 6.45 (m, 2H), 6.34 (d, *J* = 15.2 Hz, 1H), 2.80 (h, *J* = 6.9 Hz, 1H), 2.64 (t, *J* = 7.9 Hz, 2H), 2.14 – 2.05 (m, 1H), 1.77 – 1.67 (m, 1H), 1.19 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 203.6, 152.3, 143.8, 142.3, 141.9, 128.5, 128.4, 128.2, 127.8, 125.9, 125.1, 112.3, 112.1, 43.7, 34.9, 33.5, 16.8. **HRMS** (ESI) calculated for $[C_{19}H_{20}O_2+Na]^+$ 303.1361, found 303.1365. **Chiral HPLC**: (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 93%, *es* = 94%.



Peak#	Ret. Time	Area	Area%
1	12.21	14973673	50.898
2	16.026	14445528	49.102
Total		29419201	100

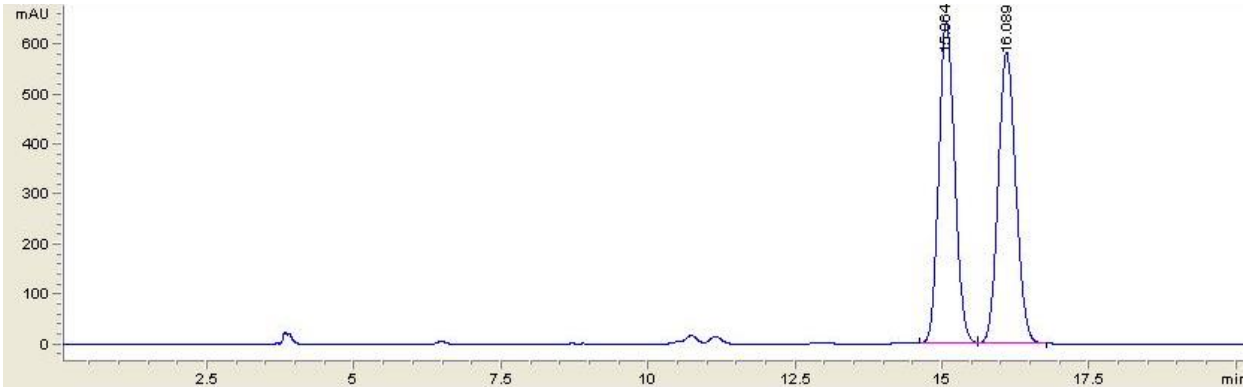


Peak#	Ret. Time	Area	Area%
1	12.867	2282133	3.518
2	16.831	62586511	96.482
Total		64868644	100

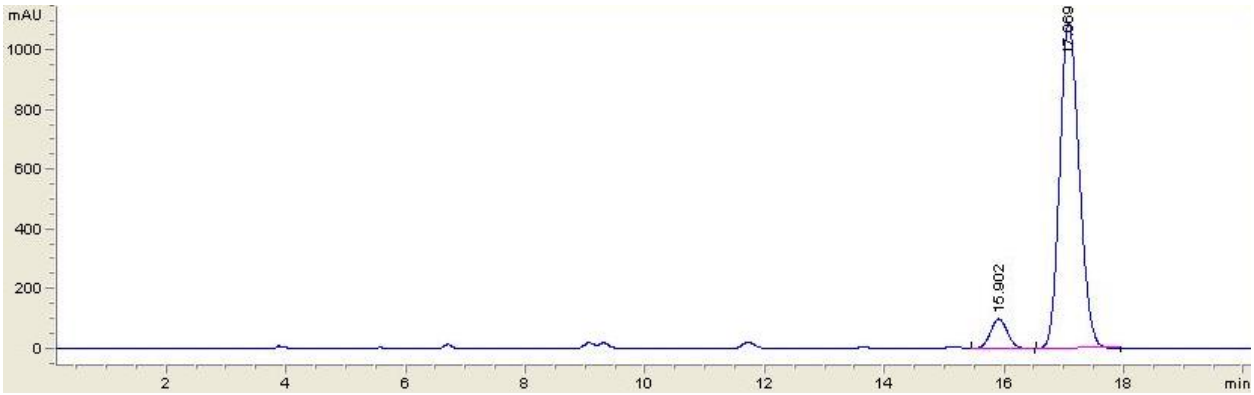


(*R*,5*E*,7*E*)-3-methyl-1-phenyltrideca-5,7-dien-4-one (10) was obtained from General Carbonylation Procedure A and the crude product was flashed in 2.5% EtOAc/hexanes yielding **10** as a yellow oil (74 mg, 52%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.31 (t, *J* =

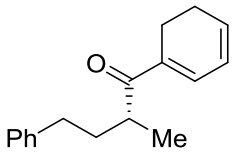
7.5 Hz, 2H), 7.24 – 7.12 (m, 4H), 6.22 – 6.12 (m, 3H), 2.80 (h, *J* = 6.9 Hz, 1H), 2.67 – 2.58 (m, 2H), 2.21 (td, *J* = 7.3, 5.2 Hz, 2H), 2.12 – 2.02 (m, 1H), 1.70 (ddt, *J* = 13.6, 8.5, 6.7 Hz, 1H), 1.47 (p, *J* = 7.4 Hz, 2H), 1.33 (ttd, *J* = 15.9, 8.4, 4.5 Hz, 4H), 1.17 (d, *J* = 6.9 Hz, 3H), 0.93 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 204.0, 146.1, 143.3, 141.9, 128.9, 128.5, 128.4, 126.5, 125.9, 43.3, 34.9, 33.5, 33.2, 31.4, 28.4, 22.5, 16.8, 14.1. **HRMS** (ESI) calculated for $[C_{20}H_{28}O+Na]^+$ 307.2038, found 307.2045. **Chiral HPLC** (ChiralPak IC, 99:1 hexanes:isopropanol): *ee* = 85%, *es* = 86%.



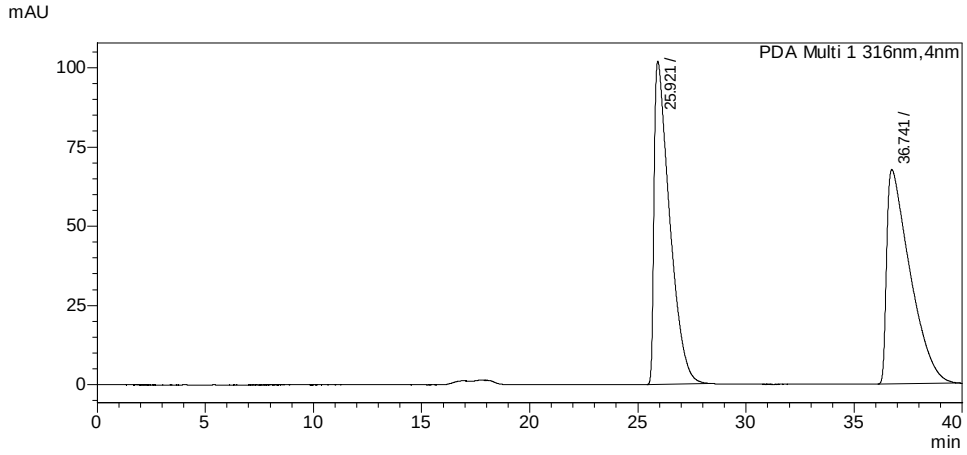
Peak#	Ret. Time	Area	Area%
1	15.064	12158	49.933
2	16.089	12191	50.067
Total		24349	100



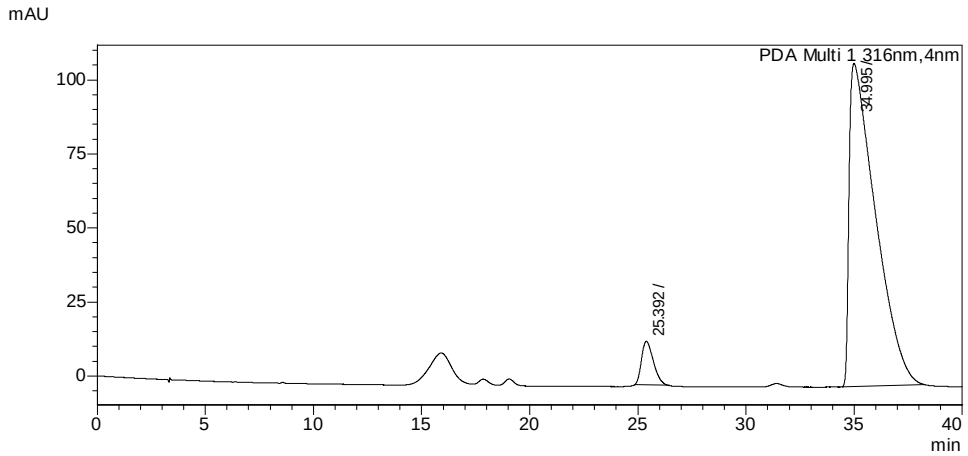
Peak#	Ret. Time	Area	Area%
1	15.902	1956	7.315
2	17.069	24792	92.686
Total		24349	100



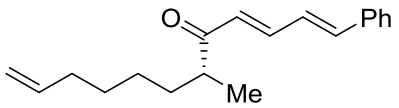
(*R*)-1-(cyclohexa-1,3-dien-1-yl)-2-methyl-4-phenylbutan-1-one (11) was obtained from General Carbonylation Procedure A (60% NMR yield) and the crude product was flashed in 5% EtOAc/hexanes yielding **11** as a colorless oil (49 mg, 41%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.30 (d, *J* = 7.6 Hz, 2H), 7.25 – 7.16 (m, 3H), 6.79 (d, *J* = 5.5 Hz, 1H), 6.29 – 6.22 (m, 1H), 6.10 (ddt, *J* = 9.4, 5.5, 2.0 Hz, 1H), 3.23 (h, *J* = 6.9 Hz, 1H), 2.60 (tt, *J* = 8.2, 6.8 Hz, 2H), 2.50 – 2.41 (m, 2H), 2.28 (tdd, *J* = 9.8, 4.4, 1.7 Hz, 2H), 2.08 (ddt, *J* = 13.5, 8.7, 7.3 Hz, 1H), 1.74 – 1.65 (m, 1H), 1.15 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 204.7, 142.0, 135.2, 135.1, 132.8, 128.5, 128.3, 125.9, 124.1, 38.2, 35.7, 33.6, 23.0, 19.8, 18.0. **HRMS** (ESI) calculated for $[C_{17}H_{20}O+Na]^+$ 263.1412, found 263.1415. **Chiral HPLC**: (Daicel OJ-H, 100:0 hexanes:isopropanol): *ee* = 88%, *es* = 89%.



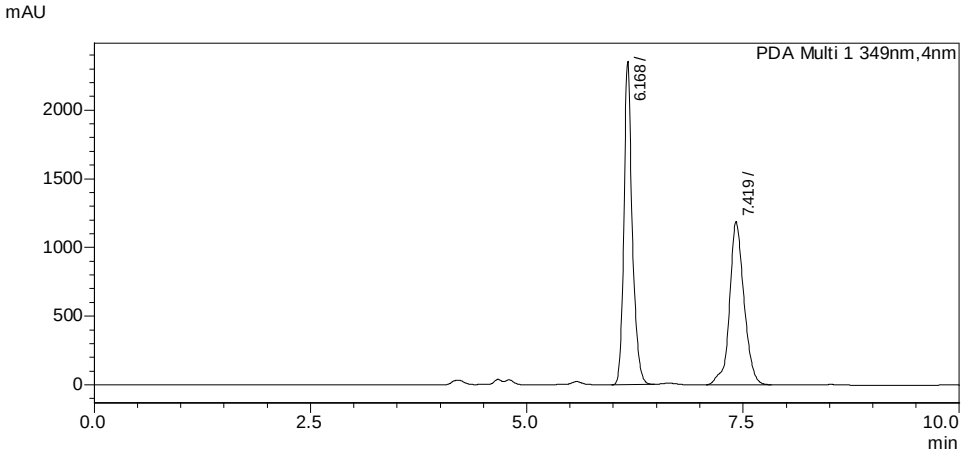
Peak#	Ret. Time	Area	Area%
1	25.921	5087005	50.077
2	36.741	5071352	49.923
Total		10158358	100



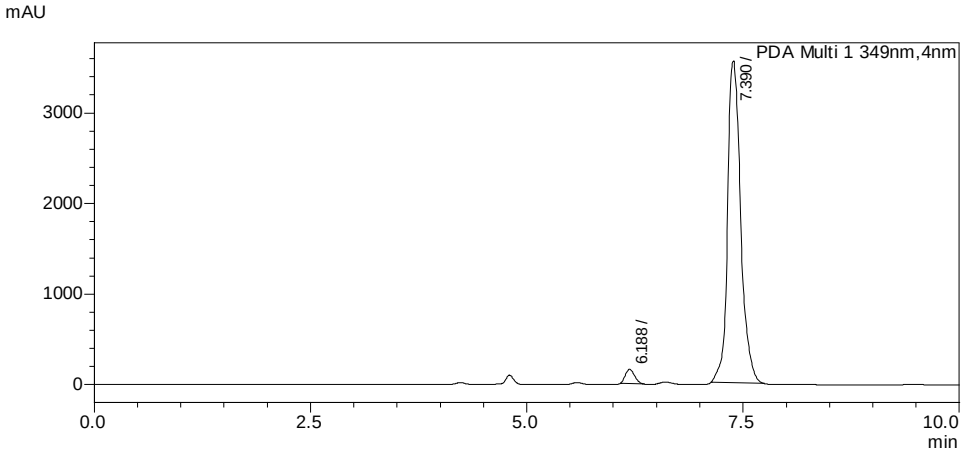
Peak#	Ret. Time	Area	Area%
1	25.392	563762	5.767
2	34.995	9211383	94.233
Total		9775145	100



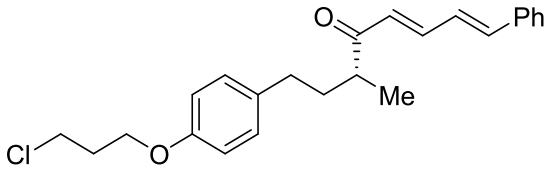
(*R*,1*E*,3*E*)-6-methyl-1-phenyldodeca-1,3,11-trien-5-one (12) was obtained from General Carbonylation Procedure A and the crude product was flashed in 5% EtOAc/hexanes yielding **12** as a pale yellow liquid (83 mg, 62%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.52 – 7.48 (m, 2H), 7.45 – 7.31 (m, 4H), 6.98 (d, *J* = 15.6 Hz, 1H), 6.92 (dd, *J* = 15.5, 10.4 Hz, 1H), 6.38 (d, *J* = 15.2 Hz, 1H), 5.82 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.05 – 4.98 (m, 1H), 4.96 (dd, *J* = 10.1, 2.0 Hz, 1H), 2.79 (h, *J* = 6.9 Hz, 1H), 2.07 (q, *J* = 7.2 Hz, 2H), 1.81 – 1.69 (m, 1H), 1.45 – 1.39 (m, 3H), 1.42 – 1.30 (m, 2H), 1.15 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 204.0, 142.5, 141.3, 138.9, 136.1, 129.1, 128.9, 128.3, 127.2, 126.8, 114.4, 44.6, 33.7, 33.2, 29.0, 26.9, 16.7. **HRMS** (ESI) calculated for $[C_{19}H_{24}O+Na]^+$ 291.1725, found 291.1726. **Chiral HPLC** (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 94%, *es* = 97%.



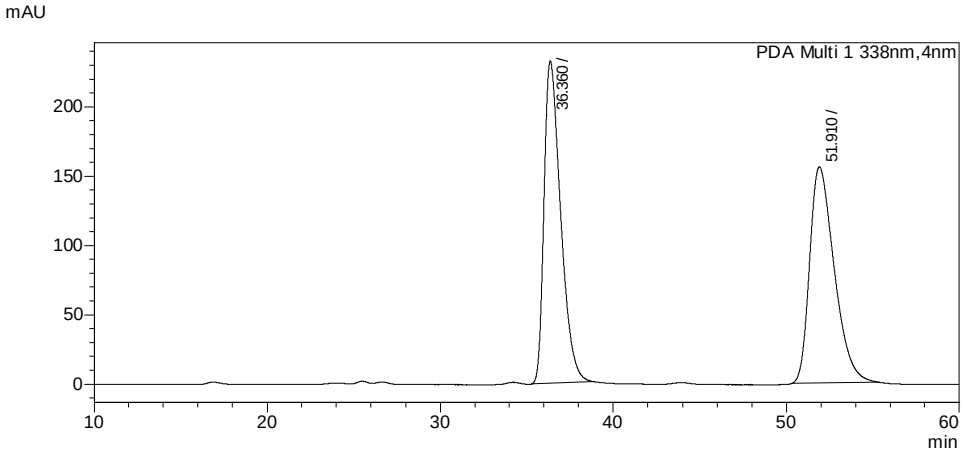
Peak#	Ret. Time	Area	Area%
1	6.168	15822020	53.466
2	7.419	13770424	46.534
Total		29592443	100



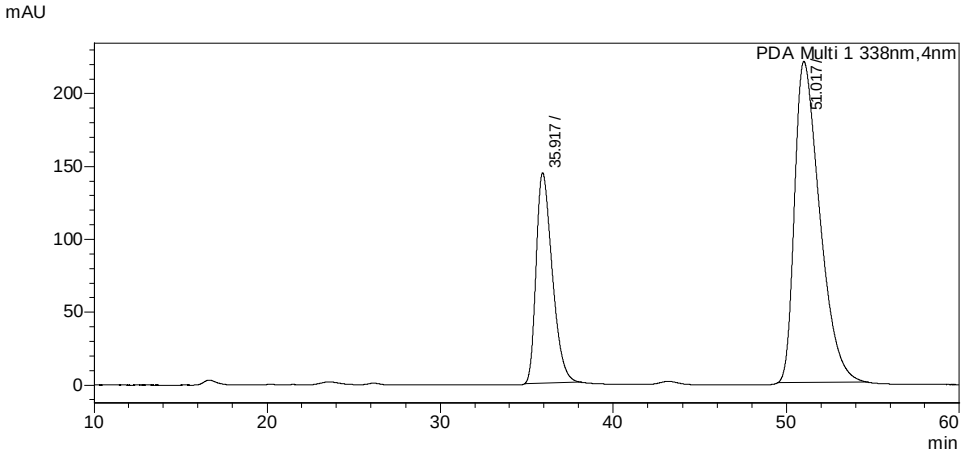
Peak#	Ret. Time	Area	Area%
1	6.188	1133130	2.93
2	7.39	37538924	97.07
Total		38672053	100



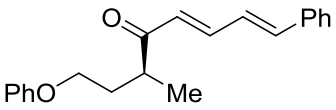
(*R*,5*E*,7*E*)-1-(4-(3-chloropropoxy)phenyl)-3-methyl-8-phenylocta-5,7-dien-4-one (13) was obtained from General Carbonylation Procedure A and the crude product was flashed in 10% EtOAc/hexanes yielding **13** as a yellow oil (103 mg, 54%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.51 (d, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.5 Hz, 2H), 7.38 – 7.30 (m, 2H), 7.13 (d, *J* = 8.2 Hz, 2H), 6.98 – 6.89 (m, 2H), 6.88 (t, *J* = 8.1 Hz, 2H), 6.35 (d, *J* = 15.3 Hz, 1H), 4.12 (t, *J* = 5.8 Hz, 2H), 3.77 (t, *J* = 6.3 Hz, 2H), 2.83 (h, *J* = 7.0 Hz, 1H), 2.60 (t, *J* = 7.8 Hz, 2H), 2.25 (p, *J* = 6.1 Hz, 2H), 2.07 (dq, *J* = 14.9, 7.6 Hz, 1H), 1.70 (dq, *J* = 14.3, 7.4 Hz, 1H), 1.20 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 203.8, 157.0, 142.7, 141.4, 136.1, 134.2, 129.5, 129.2, 128.9, 128.4, 127.3, 126.8, 114.4, 64.3, 43.4, 41.7, 35.2, 32.6, 32.3, 16.8. **HRMS** (ESI) calculated for $[C_{24}H_{27}ClO_2+Na]^+$ 405.1597, found 405.1599. **Chiral HPLC** (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 41%, *es* = 93%.



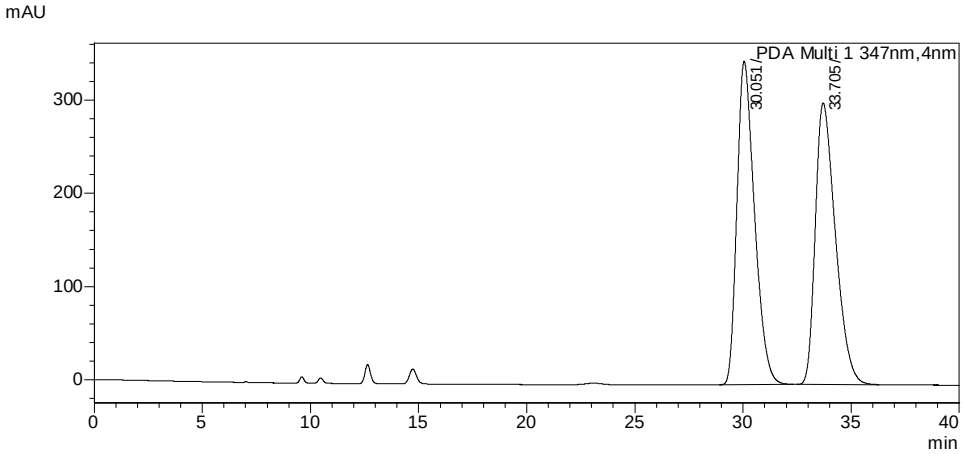
Peak#	Ret. Time	Area	Area%
1	36.36	15502264	50.261
2	51.91	15341104	49.739
Total		30843367	100



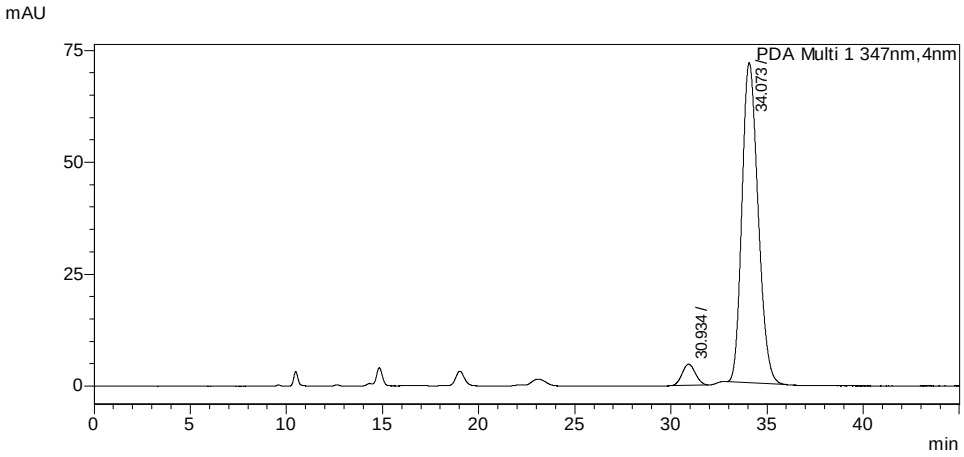
Peak#	Ret. Time	Area	Area%
1	35.917	9364415	29.56
2	51.017	22314972	70.44
Total		31679387	100



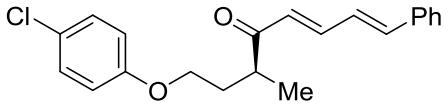
(*S*,5*E*,7*E*)-3-methyl-1-phenoxy-8-phenylocta-5,7-dien-4-one (14) was obtained from General Carbonylation Procedure A and the crude product was flashed using a gradient of 5-10% EtOAc/hexanes yielding **14** as a yellow, viscous oil (89 mg, 58%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.53 – 7.46 (m, 2H), 7.47 – 7.28 (m, 6H), 7.01 – 6.86 (m, 5H), 6.39 (d, *J* = 15.3 Hz, 1H), 4.04 (ddd, *J* = 9.5, 6.6, 5.4 Hz, 1H), 3.99 (ddd, *J* = 9.5, 7.0, 5.3 Hz, 1H), 3.24 – 3.15 (m, 1H), 2.28 (dddd, *J* = 14.2, 7.7, 6.7, 5.3 Hz, 1H), 1.89 (ddt, *J* = 14.1, 7.0, 5.7 Hz, 1H), 1.25 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 203.4, 158.8, 143.0, 141.5, 136.1, 129.5, 129.2, 128.9, 128.7, 127.3, 126.7, 120.7, 114.5, 65.4, 40.6, 32.7, 17.1. **HRMS** (ESI) calculated for $[C_{21}H_{22}O_2+Na]^+$ 329.1517, found 329.1519. **Chiral HPLC** (Daicel OJ-H, 70:30 hexanes:isopropanol): *ee* = 90%, *es* = 94%.



Peak#	Ret. Time	Area	Area%
1	30.051	19619700	50.091
2	33.705	19548166	49.909
Total		39167867	100

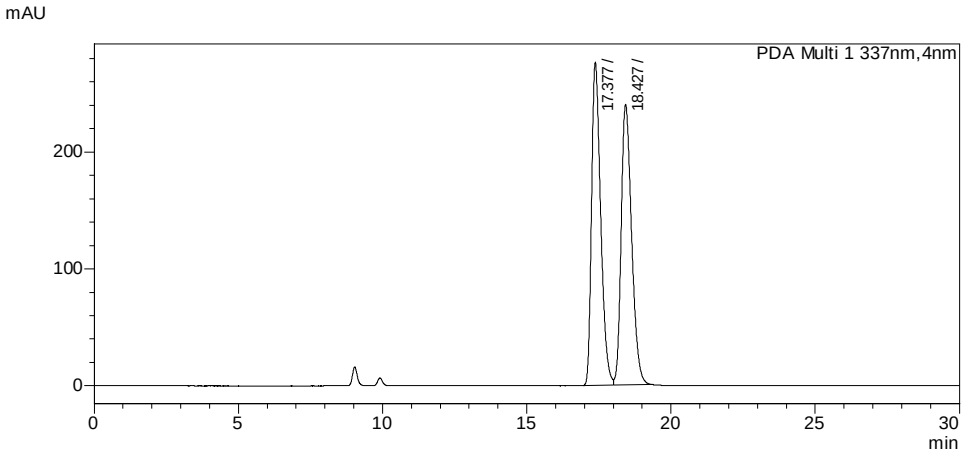


Peak#	Ret. Time	Area	Area%
1	30.917	528020	5.163
2	34.072	9699449	94.837
Total		10227469	100

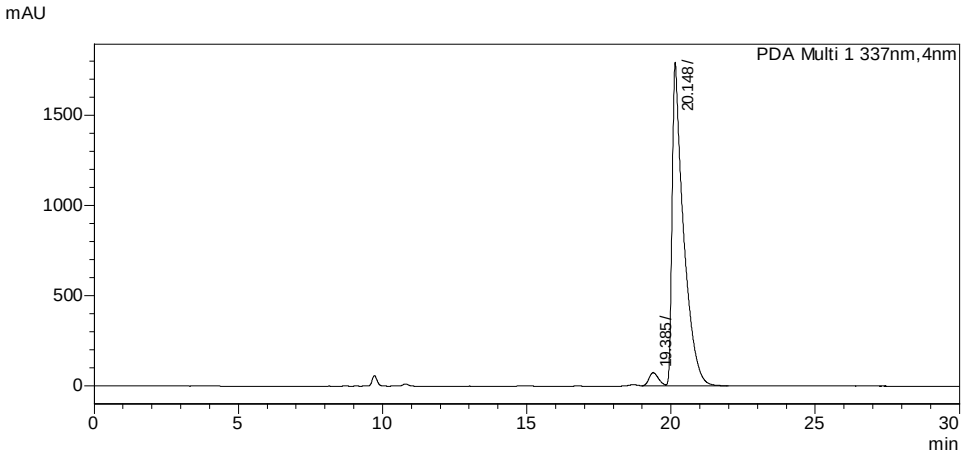


(*S*,5*E*,7*E*)-1-(4-chlorophenoxy)-3-methyl-8-phenylocta-5,7-dien-4-one (15) was obtained from General Carbonylation Procedure A, stopping the reaction after 16 hours, and the crude product was flashed using a gradient of 5-10% EtOAc/hexanes yielding

15 as a pale yellow oil (73 mg, 43%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.51 – 7.46 (m, 2H), 7.46 – 7.36 (m, 3H), 7.38 – 7.31 (m, 1H), 7.27 – 7.20 (m, 2H), 6.97 – 6.86 (m, 2H), 6.86 – 6.80 (m, 2H), 6.38 (d, *J* = 15.3 Hz, 1H), 4.03 – 3.91 (m, 2H), 3.15 (h, *J* = 7.1 Hz, 1H), 2.26 (ddt, *J* = 14.0, 7.8, 6.1 Hz, 1H), 1.91 – 1.83 (m, 1H), 1.24 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 203.2, 157.4, 143.0, 141.6, 136.0, 129.3, 129.2, 128.9, 128.5, 127.3, 126.7, 125.5, 115.7, 65.9, 40.7, 32.5, 17.2. **HRMS** (ESI) calculated for $[C_{21}H_{21}ClO_2+Na]^+$ 363.1128, found 363.1137. **Chiral HPLC** (Chiralpak IE, 95:5 hexanes:isopropanol): *ee* = 93%, *es* = 95%.

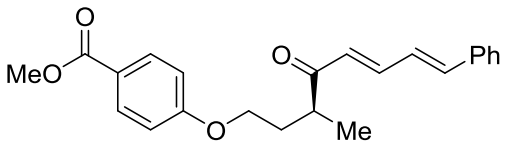


Peak#	Ret. Time	Area	Area%
1	17.377	6069750	50.072
2	18.427	6052304	49.928
Total		12122054	100

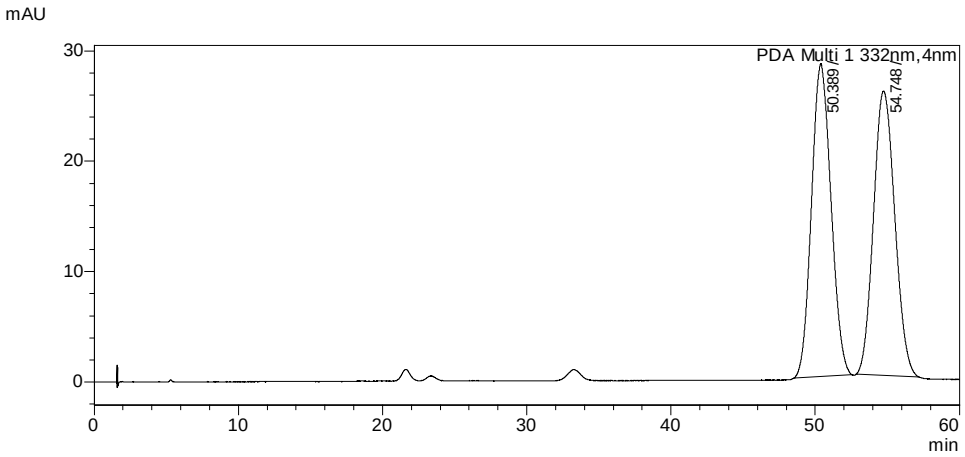


Peak#	Ret. Time	Area	Area%

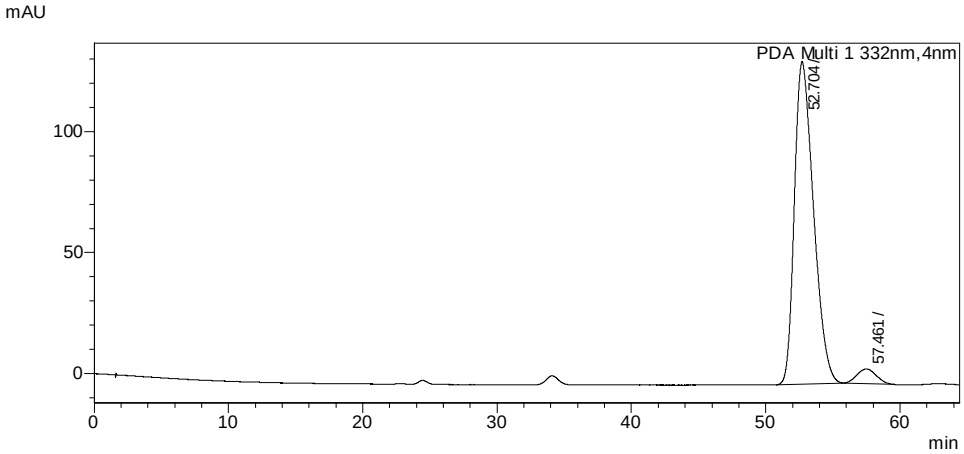
1	19.385	872620	3.484
2	20.148	24170590	96.516
Total		25043210	100



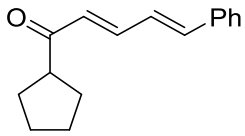
Methyl 4-(((*S*,5*E*,7*E*)-3-methyl-4-oxo-8-phenylocta-5,7-dien-1-yl)oxy)benzoate (16) was obtained from General Carbonylation Procedure A and the crude product was flashed using a gradient of 5-10% EtOAc/hexanes yielding **16** as a colorless oil (85 mg, 47%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 8.03 – 7.94 (m, 2H), 7.50 – 7.45 (m, 2H), 7.45 – 7.30 (m, 4H), 6.96 – 6.82 (m, 4H), 6.38 (d, *J* = 15.3 Hz, 1H), 4.11 – 3.98 (m, 2H), 3.89 (s, 3H), 3.20 – 3.10 (m, 1H), 2.33 – 2.24 (m, 1H), 1.90 (ddt, *J* = 14.2, 7.1, 5.7 Hz, 1H), 1.24 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 203.1, 166.8, 162.6, 143.1, 141.7, 136.0, 131.6, 129.2, 128.9, 128.4, 127.3, 126.6, 122.6, 114.1, 65.8, 51.9, 40.7, 32.4, 17.2. **HRMS** (ESI) calculated for $[C_{23}H_{24}O_4+Na]^+$ 387.1572, found 387.1575. **Chiral HPLC** (Chiralpak IG, 95:5 hexanes:isopropanol, 2 mL/min): *ee* = 91%, *es* = 93%.



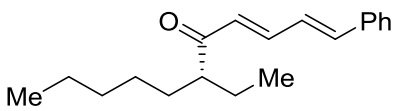
Peak#	Ret. Time	Area	Area%
1	50.386	2112095	50.122
2	54.748	2101852	49.878
Total		4213946	100



Peak#	Ret. Time	Area	Area%
1	52.704	12705069	95.636
2	57.461	579746	4.364
Total		13284815	100

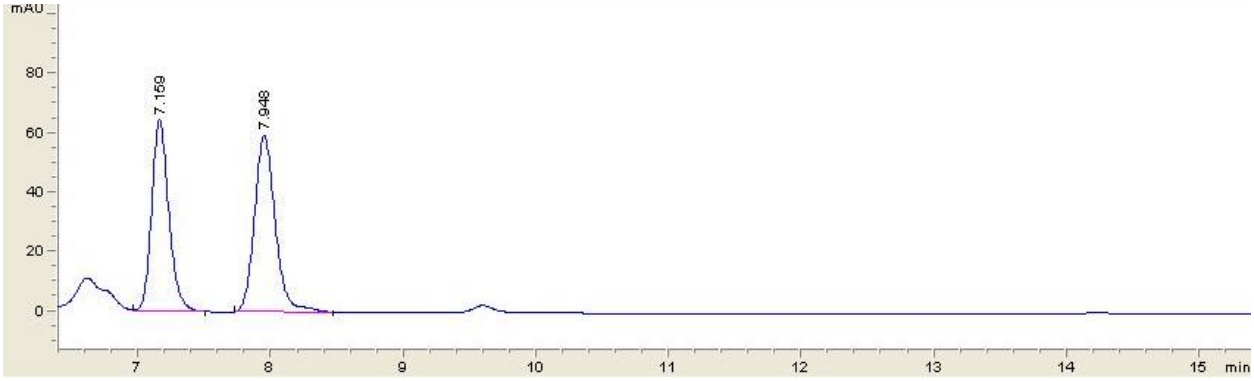


(2E,4E)-1-cyclopentyl-5-phenylpenta-2,4-dien-1-one (17) was obtained from General Carbonylation Procedure A and the crude product was flashed in 5% EtOAc/hexanes yielding **17** as a pale yellow liquid (54 mg, 48%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.50 (d, *J* = 7.3 Hz, 2H), 7.45 – 7.36 (m, 3H), 7.33 (t, *J* = 7.3 Hz, 1H), 7.04 – 6.84 (m, 2H), 6.37 (d, *J* = 15.3 Hz, 1H), 3.15 (p, *J* = 8.0 Hz, 1H), 1.94 – 1.78 (m, 4H), 1.78 – 1.68 (m, 2H), 1.64 (tdd, *J* = 11.9, 7.2, 4.0 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 202.7, 142.3, 141.0, 136.1, 129.13, 129.09, 128.8, 127.2, 126.9, 49.6, 29.3, 26.2. **HRMS** (ESI) calculated for $[C_{16}H_{18}O+H]^+$ 227.1436, found 227.1433.

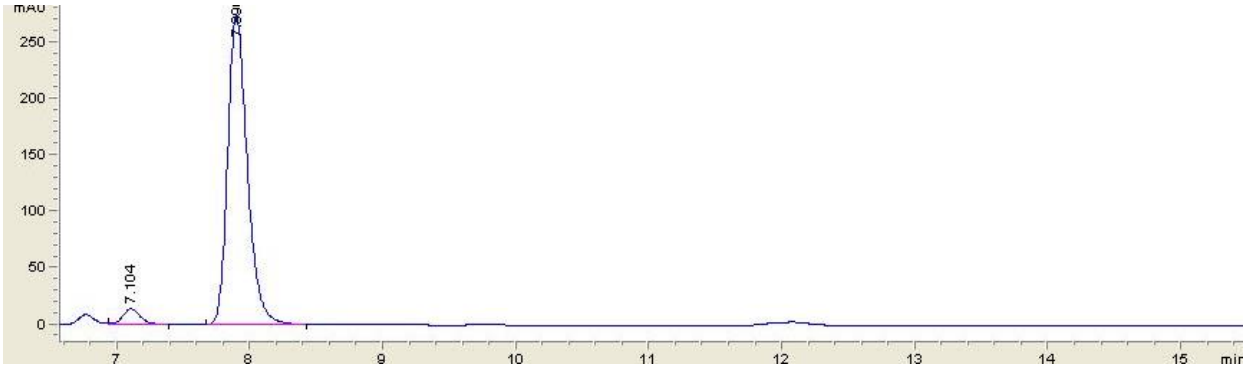


(*R*,1*E*,3*E*)-6-ethyl-1-phenylundeca-1,3-dien-5-one (18) was obtained from General Carbonylation Procedure A (38% NMR yield) and the crude product was flashed in 5% EtOAc/hexanes yielding **18** as a yellow oil (41 mg, 30%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.52 – 7.47 (m, 2H), 7.45 – 7.30 (m, 4H), 7.02 – 6.87 (m, 2H), 6.40 (d, *J*

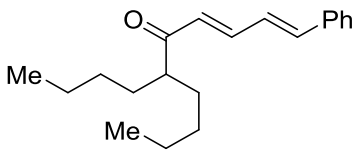
= 15.2 Hz, 1H), 2.65 (tt, *J* = 8.4, 5.5 Hz, 1H), 1.75 – 1.62 (m, 2H), 1.59 – 1.42 (m, 2H), 1.34 – 1.25 (m, 6H), 0.89 (td, *J* = 7.2, 5.3 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 204.2, 142.3, 141.3, 136.1, 129.1, 129.0, 128.8, 127.2, 126.9, 52.2, 32.0, 31.8, 27.2, 25.1, 22.5, 14.1, 12.0. **HRMS** (ESI) calculated for $[C_{19}H_{26}O+H]^+$ 271.2062, found 271.2068. **Chiral HPLC**: (Chiralpak IC, 95:5 hexanes:isopropanol): *ee* = 91%, *es* = 92%.



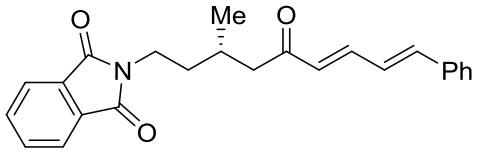
Peak#	Ret. Time	Area	Area%
1	7.159	580	47.066
2	7.948	652	52.934
Total		13721	100



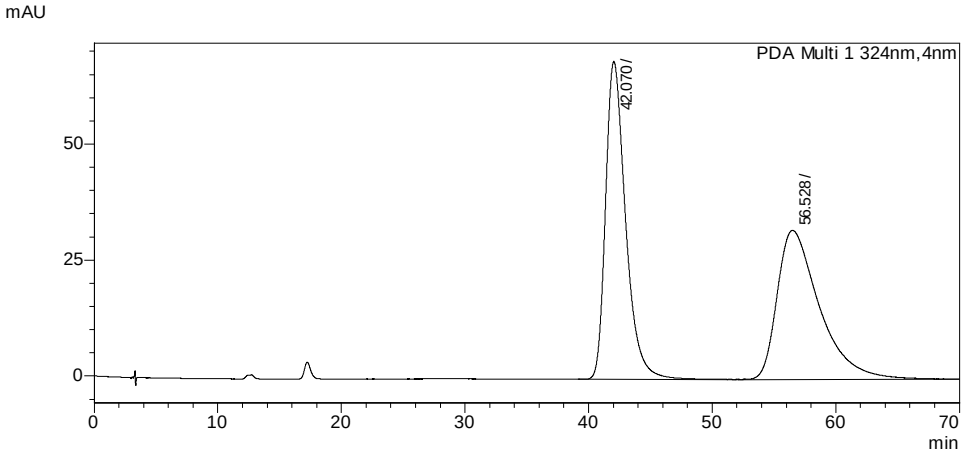
Peak#	Ret. Time	Area	Area%
1	7.104	133	4.486
2	7.896	275	95.514
Total		408	100



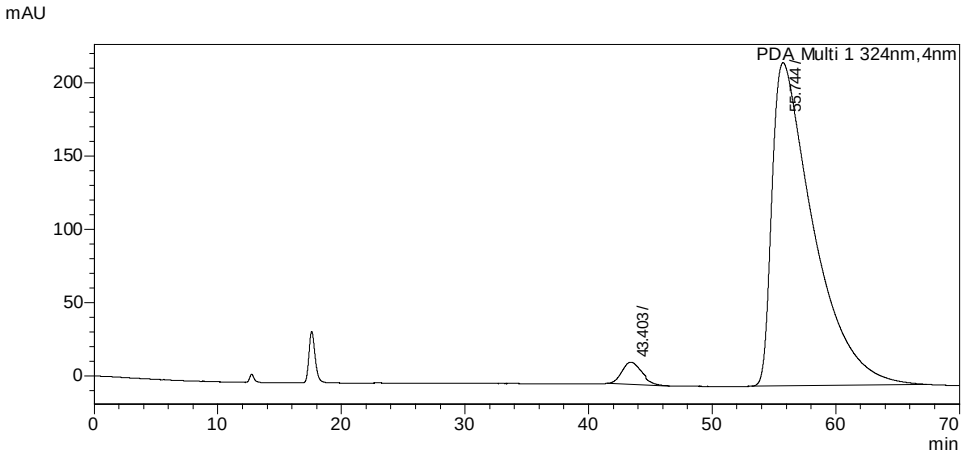
(1*E*,3*E*)-6-butyl-1-phenyldeca-1,3-dien-5-one (19) was obtained from General Carbonylation Procedure A and the crude product (47% NMR yield) was flashed in 5% EtOAc/hexanes yielding **19** as a colorless oil (56 mg, 40%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.53 – 7.48 (m, 2H), 7.46 – 7.31 (m, 4H), 6.99 (d, *J* = 15.6 Hz, 1H), 6.96 – 6.90 (m, 1H), 6.39 (d, *J* = 15.2 Hz, 1H), 2.70 (tt, *J* = 8.4, 5.4 Hz, 1H), 1.72 – 1.62 (m, 2H), 1.53 – 1.44 (m, 2H), 1.36 – 1.20 (m, 8H), 0.90 (t, *J* = 7.2 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 204.4, 142.4, 141.3, 136.1, 129.1, 128.9, 128.8, 127.2, 126.9, 50.6, 32.0, 29.8, 22.9, 14.0. **HRMS** (ESI) calculated for $[C_{20}H_{28}O+Na]^+$ 307.0238, found 307.0239.



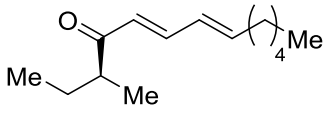
2-((*S*,6*E*,8*E*)-3-methyl-5-oxo-9-phenylnona-6,8-dien-1-yl)isoindoline-1,3-dione (20) was obtained from General Carbonylation Procedure A, increasing reaction temperature to 90 ºC and pressure to 10 atm CO, and the crude product was flashed in 25% EtOAc/hexanes yielding **20** as a yellow oil (85 mg, 46%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.85 (dt, *J* = 7.4, 3.7 Hz, 2H), 7.71 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.50 – 7.46 (m, 2H), 7.41 – 7.30 (m, 4H), 6.96 (d, *J* = 15.6 Hz, 1H), 6.86 (dd, *J* = 15.5, 10.8 Hz, 1H), 6.29 (d, *J* = 15.4 Hz, 1H), 3.80 – 3.70 (m, 2H), 2.71 (dd, *J* = 15.5, 5.4 Hz, 1H), 2.47 (dd, *J* = 15.5, 8.4 Hz, 1H), 2.19 – 2.10 (m, 1H), 1.84 – 1.72 (m, 1H), 1.62 (dq, *J* = 14.4, 7.4 Hz, 1H), 1.05 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 199.8, 168.4, 142.7, 141.4, 136.0, 133.9, 132.1, 129.8, 129.2, 128.8, 127.2, 126.7, 123.2, 47.6, 36.0, 35.5, 27.5, 19.7. **HRMS** (ESI) calculated for $[C_{24}H_{23}NO_3+H]^+$ 374.1756, found 374.1776. **Chiral HPLC**: (Daicel OJ-H, 50:50 hexanes:isopropanol): *ee* = 94%.



Peak#	Ret. Time	Area	Area%
1	42.07	7724703	50.33
2	56.528	7623398	49.67
Total		15348101	100

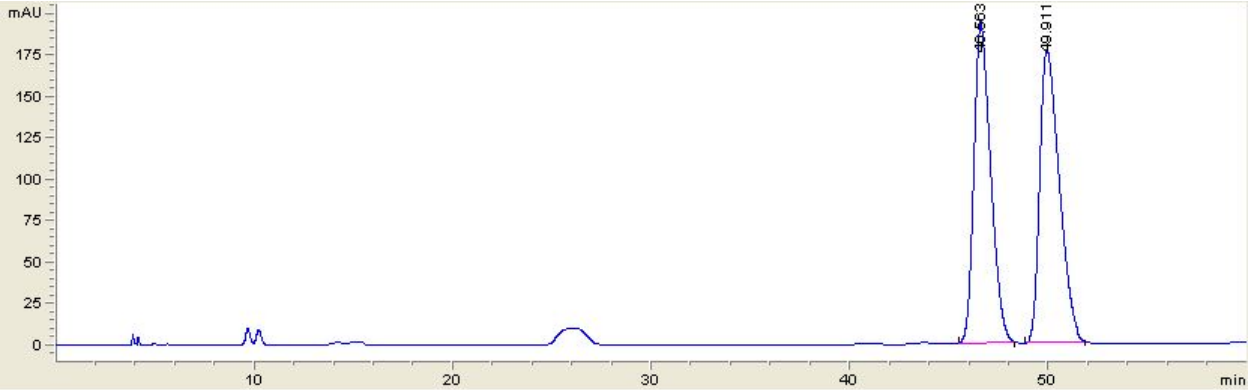


Peak#	Ret. Time	Area	Area%
1	43.403	1687262	3.181
2	55.744	51362640	96.819
Total		53049902	100

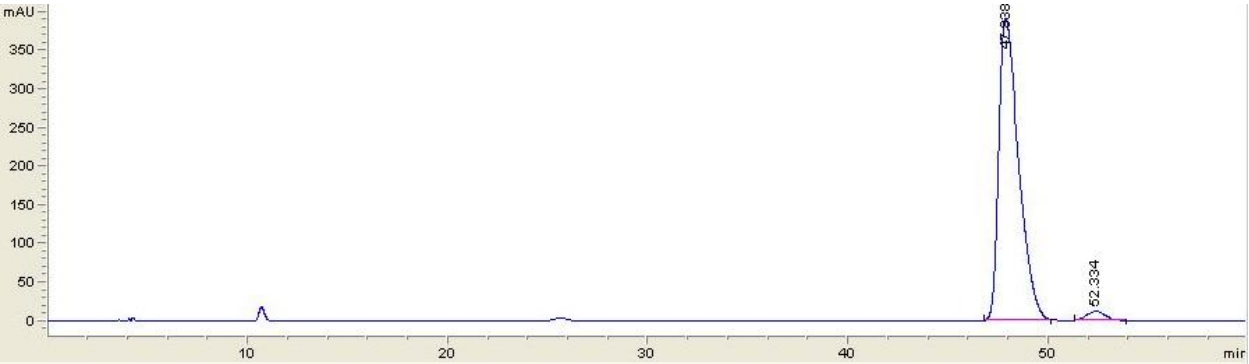


(*S*,5*E*,7*E*)-3-methyltrideca-5,7-dien-4-one (SI-30) was obtained from General Carbonylation Procedure A, decreasing reaction time to 8 hours, and the crude product was flashed in 2.5% EtOAc/hexanes yielding **SI-30** as a pale yellow oil (56 mg, 54%).

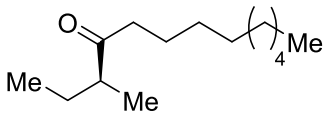
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.24 – 7.15 (m, 1H), 6.24 – 6.13 (m, 3H), 2.67 (h, *J* = 6.9 Hz, 1H), 2.23 – 2.15 (m, 2H), 1.71 (dp, *J* = 14.6, 7.4 Hz, 1H), 1.43 (dq, *J* = 13.8, 7.1 Hz, 3H), 1.38 – 1.25 (m, 4H), 1.10 (d, *J* = 6.9 Hz, 3H), 0.96 – 0.84 (m, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 204.4, 145.8, 143.0, 128.9, 126.5, 45.8, 33.1, 31.4, 28.4, 26.3, 22.5, 16.3, 14.0, 11.8. **HRMS** (ESI) calculated for $[C_{21}H_{22}O+H]^+$ 231.1725, found 231.1726. **Chiral HPLC**: (Chiralpak IC, 99.9:0.1 hexanes:isopropanol): *ee* = 94%, *es* = 94%.



Peak#	Ret. Time	Area	Area%
1	46.563	11904	50.174
2	49.911	11821	49.826
Total		23725	100



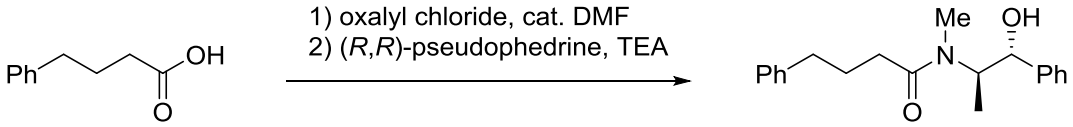
Peak#	Ret. Time	Area	Area%
1	47.838	26938	97.327
2	52.334	740	2.673
Total		27678	100



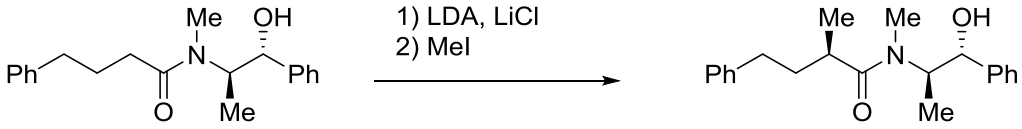
(*S*)-3-methyldecan-4-one (22) was obtained by hydrogenating **SI-30** (56 mg, 0.27 mmol) according to General Procedure F. **22** was obtained as a colorless oil without further purification (57 mg, 100%). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 2.50 – 2.37 (m, 3H), 1.73 – 1.63 (m, 1H), 1.56 (q, *J* = 7.1 Hz, 2H), 1.43 – 1.33 (m, 1H), 1.34 – 1.25 (m, 12H), 1.06 (d, *J* = 6.9 Hz, 3H), 0.94 – 0.85 (m, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 215.2, 47.8, 41.2, 31.9, 29.49, 29.47, 29.34, 29.30, 26.0, 23.7, 22.7, 16.0, 14.1, 11.7. **HRMS** (ESI) calculated for $[C_{14}H_{28}O+Na]^+$ 235.2038, found 235.2036.

Mechanistic Experiments

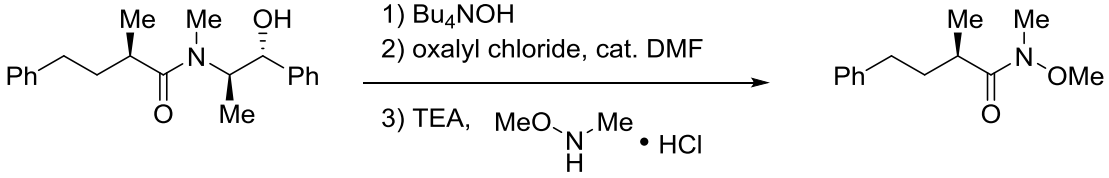
Absolute Stereochemistry Determination: An independent HPLC standard of reduced dienone product was synthesized, using the chiral auxiliary route shown below, to prove that the carbonylation reaction proceeds via inversion. This suggests an S_N2 oxidative addition is operative in the reaction mechanism.



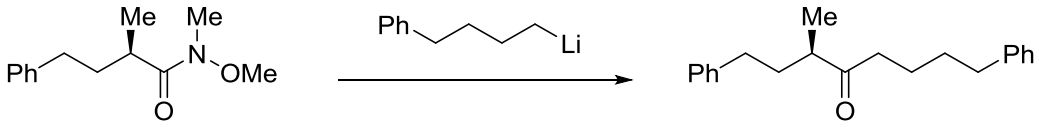
***N*-((1*R*,2*R*)-1-hydroxy-1-phenylpropan-2-yl)-*N*-methyl-4-phenylbutanamide (SI-31)** was synthesized by dissolving 4-phenylbutanoic acid (2.2 g, 13.4 mmol) in DCM (50 mL) with a few drops of DMF at 0 ºC. To this stirred solution was added oxalyl chloride (2.3 mL, 26.8 mmol) dropwise. The reaction was stirred at 0 ºC for 15 minutes and warmed to room temperature for an hour. Solvent and excess oxalyl chloride were removed under reduced pressure and the mixture was brought up in DCM (5 mL). This solution was added dropwise to a stirred solution of (*R*,*R*)-pseudoephedrine (2 g, 12.1 mmol) and TEA (2.2 mL, 16.1 mmol) in DCM (24 mL) at 0 ºC for 10 minutes. The reaction was then warmed to room temperature for an hour and quenched with water. Brine was added and the layers were separated. The organic layer was washed twice with brine, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified via flash chromatography in 50% EtOAc/hexanes to provide 2.5 g (66%) of **SI-31** as a white solid in a 3:1 ratio of rotamers. **^{1}H NMR** (600 MHz, Chloroform-*d,* minor rotamer, when resolved, indicated with *) δ 7.42 – 7.26 (m, 14H), 7.26 – 7.15 (m, 6H), 4.61 (d, *J* = 7.8 Hz, 1H), 4.55* (d, *J* = 8.7 Hz, 1H), 4.53 – 4.40* (m, 1H), 3.92 (dq, *J* = 8.7, 6.8 Hz, 1H), 2.94* (s, 3H), 2.77 (s, 3H), 2.75 – 2.62 (m, 5H), 2.49* (ddd, *J* = 15.3, 9.0, 6.3 Hz, 1H), 2.41* (ddd, *J* = 15.3, 8.8, 6.3 Hz, 1H), 2.37 – 2.22 (m, 2H), 2.08 – 1.91 (m, 5H), 1.12 (d, *J* = 7.0 Hz, 3H), 0.97* (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 175.2, 174.0, 142.5, 141.9, 141.7, 141.2, 128.7, 128.6, 128.52, 128.47, 128.43, 128.40, 128.38, 127.7, 126.9, 126.4, 125.9, 125.9, 76.6, 75.5, 58.6 (br), 58.3, 35.5, 35.2, 35.1, 33.4, 32.9, 26.9, 26.8, 26.4, 15.4, 14.5. **HRMS** (ESI) calculated for $[C_{20}H_{25}NO_2+H]^+$ 312.1964, found 312.1964.



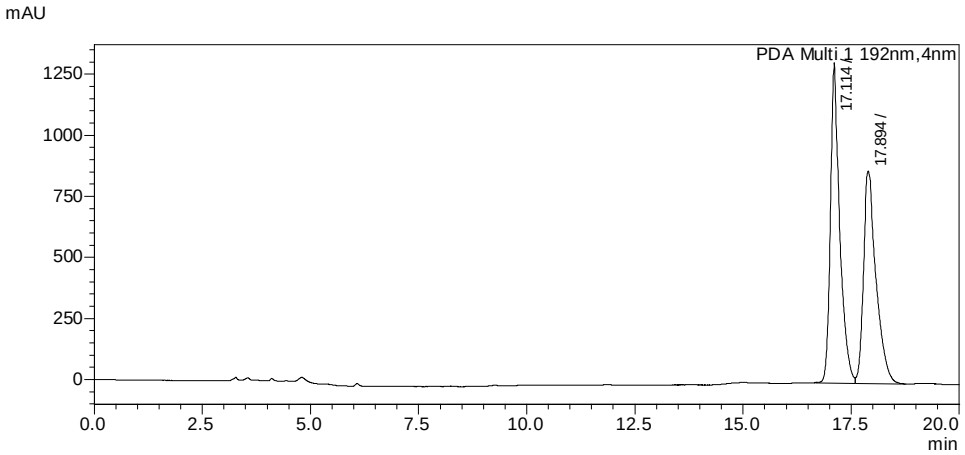
(*R*)-*N*-((1*R*,2*R*)-1-hydroxy-1-phenylpropan-2-yl)-*N*,2-dimethyl-4-phenylbutanamide (SI-32) was synthesized via diastereoselective alkylation described by Myers and co-workers.[19] *n*-BuLi (6.2 mL, 2.15 M in hexanes) was added dropwise to a stirred suspension of diisopropylamine (2 mL, 14.4 mmol) and LiCl (1.6 g, 38.5 mmol) in THF (30 mL) at -78 ºC. The mixture was warmed to 0 ºC for 10 minutes and then cooled back down to -78 ºC. To this mixture was added a solution of **SI-31** (2 g, 6.4 mmol) in THF (20 mL) dropwise. The reaction was stirred at -78 ºC for 1 hour, warmed to 0 ºC for 15 minutes, room temperature for 5 minutes, and then returned to 0 ºC. MeI (0.6 mL, 9.6 mmol) was added and the reaction was stirred at 0 ºC for 30 minutes and quenched with a saturated aqueous solution of NH_4Cl. The solution was extracted 3 times with EtOAc, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified via flash chromatography in 50% EtOAc/hexanes to provide 2.1 g (100%) of **SI-32** as a viscous yellow oil in a 3:1 ratio of rotamers. **^{1}H NMR** (600 MHz, Chloroform-*d,* minor rotamer, when resolved, indicated with *) δ 7.45 – 7.25 (m, 14H), 7.25 – 7.13 (m, 6H), 4.67 (t, *J* = 7.3 Hz, 1H), 4.52* (dd, *J* = 8.5, 2.1 Hz, 1H), 4.41 – 4.25 (m, 1H), 3.91 – 3.83* (m, 1H), 2.95* (s, 3H), 2.76* (dd, *J* = 13.0, 6.3 Hz, 1H), 2.72 – 2.61 (m, 6H), 2.61 – 2.48 (m, 2H), 2.14 – 2.03 (m, 1H), 2.03 – 1.93* (m, 1H), 1.79 – 1.66 (m, 4H), 1.22 (dd, *J* = 15.2, 6.8 Hz, 6H), 1.05 (d, *J* = 6.8 Hz, 3H), 0.96* (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 178.6, 177.6, 171.2, 142.6, 141.8, 141.7, 140.9, 128.7, 128.5, 128.43, 128.40, 128.38, 128.34, 128.31, 127.5, 126.9, 126.2, 126.0, 125.9, 76.6, 75.5, 60.4, 57.6, 35.9, 35.5, 35.4, 34.5, 33.4, 33.2, 27.1, 21.1, 17.4, 17.2, 15.5, 14.6, 14.2. **HRMS** (ESI) calculated for $[C_{21}H_{27}NO_2+H]^+$ 326.2120, found 326.2118.



(*R*)-*N*-methoxy-*N*,2-dimethyl-4-phenylbutanamide (SI-33) was synthesized by first removing the chiral auxiliary from **SI-32** as described by Myers and co-workers.[19] **SI-23** (0.5 g, 1.5 mmol) and 40% tetrabutylammonium hydroxide in water (5g, 7.7 mmol) were refluxed in water (15 mL) and *t*-BuOH (5 mL) overnight. The reaction mixture was then cooled to room temperature, 0.5 M aqueous NaOH solution (100 mL) was added, and extracted 3 times with Et_2O. The aqueous layer was acidified to pH 1 with 3 M HCl and extracted 3 times with Et_2O, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude acid was plugged through celite with Et_2O, concentrated under reduced pressure, and used without further purification. To synthesize the Weinreb amide **SI-33**, the crude acid (0.24 g, 1.3 mmol) was dissolved in DCM (5 mL) with a few drops of DMF at 0 ºC. To this stirred solution was added oxalyl chloride (0.23 mL, 2.7 mmol) dropwise. The reaction was stirred at 0 ºC for 15 minutes and warmed to room temperature for an hour. Solvent and excess oxalyl chloride were removed under reduced pressure and the mixture was brought up in DCM (2 mL). This solution was added dropwise to a stirred solution of *N*,*O*-dimethylhydroxylamine hydrochloride (0.13 g, 1.3 mmol) and TEA (0.4 mL, 2.9 mmol) in DCM (3 mL) at 0 ºC for 10 minutes. The reaction was then warmed to room temperature for an hour and quenched with a saturated solution of $NaHCO_3$. The organic layer was separated and washed with 1 M HCl, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified via flash chromatography in 20% EtOAc/hexanes. This provided a mixture of product and unreacted starting material, which was removed by washing three times with 1 M NaOH, dried over $MgSO_4$, filtered, and concentrated under reduced pressure to provide 78 mg (23% over 2 steps) of **SI-33** as a colorless oil. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.30 (dd, *J* = 8.1, 7.0 Hz, 2H), 7.24 – 7.17 (m, 3H), 3.59 (s, 3H), 3.21 (s, 3H), 2.93 – 2.84 (m, 1H), 2.64 (t, *J* = 7.8 Hz, 2H), 2.11 – 2.02 (m, 1H), 1.76 – 1.67 (m, 1H), 1.17 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 142.0, 128.5, 128.3, 125.8, 61.3, 35.2, 34.5, 33.6, 32.2, 31.0, 17.4. **HRMS** (ESI) calculated for $[C_{13}H_{19}NO_2+H]^+$ 222.1494, found 222.1495.



(*R*)-3-methyl-1,8-diphenyloctan-4-one (SI-34) was synthesized by dropwise addition of (4-phenylbutyl)lithium in Et_2O (0.7 mmol, previously made by combining equal amounts of lithium and (4-bromobutyl)benzene in 1 mL Et_2O at room temperature for 30 minutes) to a stirred solution of **SI-33** (42 mg, 0.2 mmol) in Et_2O (2 mL) at 0 ºC. The reaction was stirred and allowed to warm to room temperature overnight. The reaction was quenched with a saturated aqueous solution of NH_4Cl and extracted 3 times with Et_2O. The combined organic extracts were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified via flash chromatography using a gradient of 0-5% EtOAc/hexanes to provide 5 mg (8%) of **SI-34** as a colorless liquid. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.40 – 7.30 (m, 4H), 7.30 – 7.18 (m, 6H), 2.74 – 2.56 (m, 5H), 2.56 – 2.43 (m, 2H), 2.07 (ddt, *J* = 13.9, 8.9, 7.0 Hz, 1H), 1.74 – 1.62 (m, 5H), 1.17 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 214.4, 142.3, 141.8, 128.48, 128.47, 128.44, 128.38, 126.0, 125.8, 45.7, 41.1, 35.9, 34.5, 33.5, 31.2, 23.4, 16.6. **HRMS** (ESI) calculated for $[C_{21}H_{26}O+H]^+$ 295.2062, found 295.2060. **Chiral HPLC**: (Daicel OJ-H, 95:5 hexanes:isopropanol): *ee* = 95%.



Peak#	Ret. Time	Area	Area%
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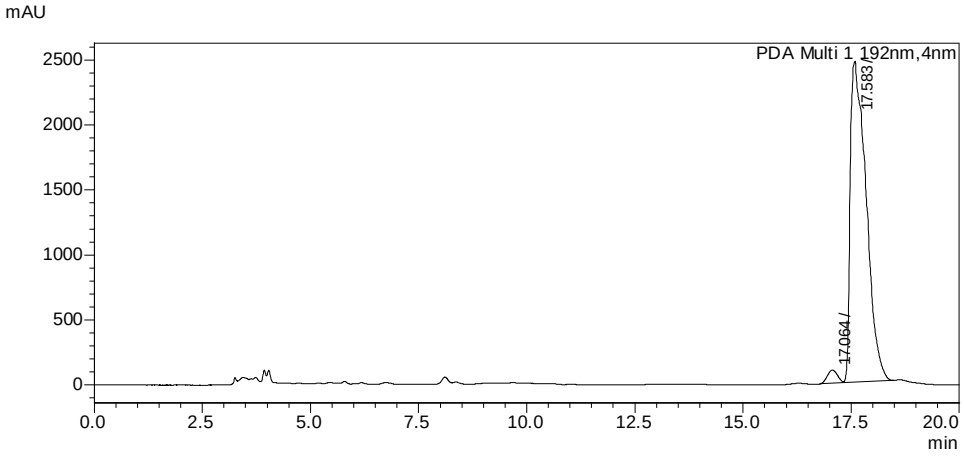
2	17.894	17468419	47.179
Total		37025845	100



Peak#	Ret. Time	Area	Area%
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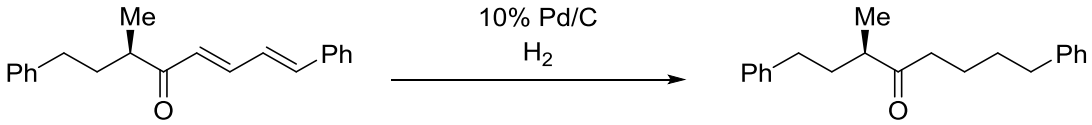
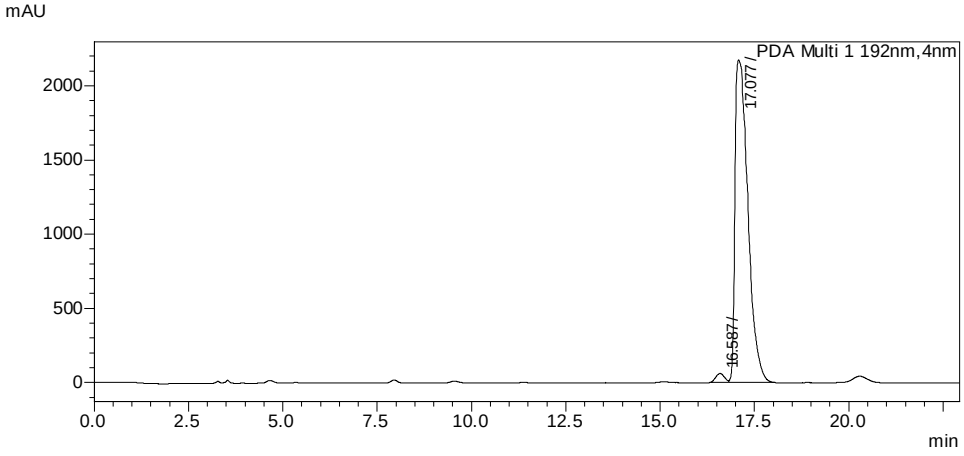
2	17.583	63016886	97.424
Total		64683452	100



(*R*)-3-methyl-1,8-diphenyloctan-4-one (SI-34) was obtained by hydrogenating **3** (98 mg, 0.34 mmol) according to General Procedure F. **SI-34** was obtained as a colorless liquid without further purification (100 mg, 100%). **Chiral HPLC**: (Daicel OJ-H, 95:5 hexanes:isopropanol): *ee* = 96%.



Peak#	Ret. Time	Area	Area%
1	16.587	881645	1.81

2	17.077	47836122	98.19
Total		48717768	100

References:

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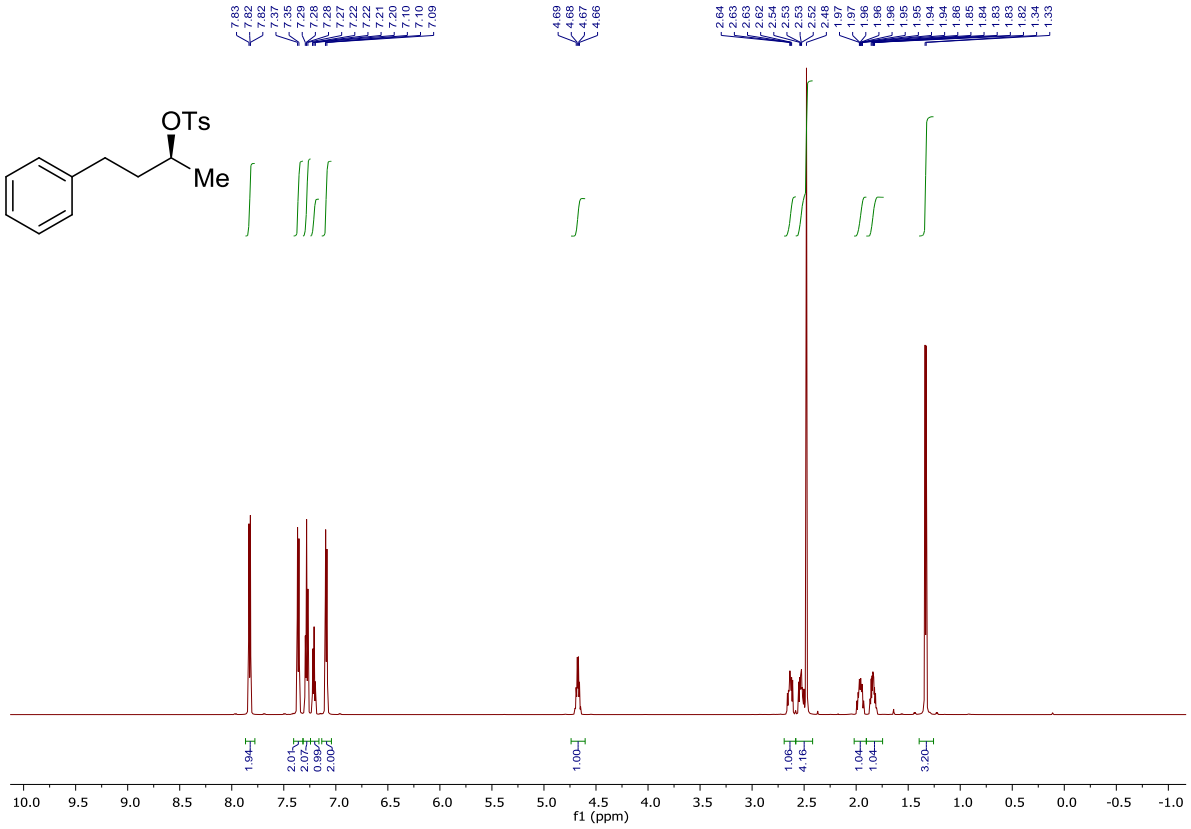
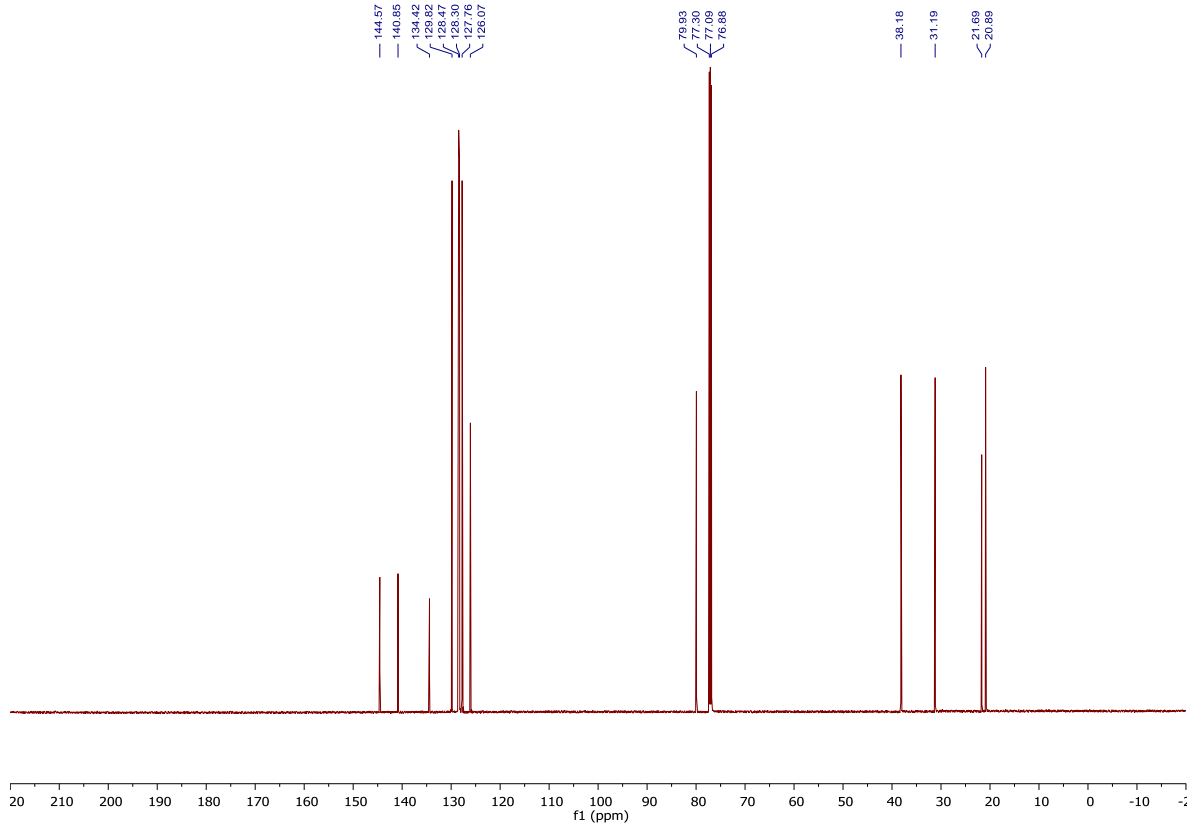
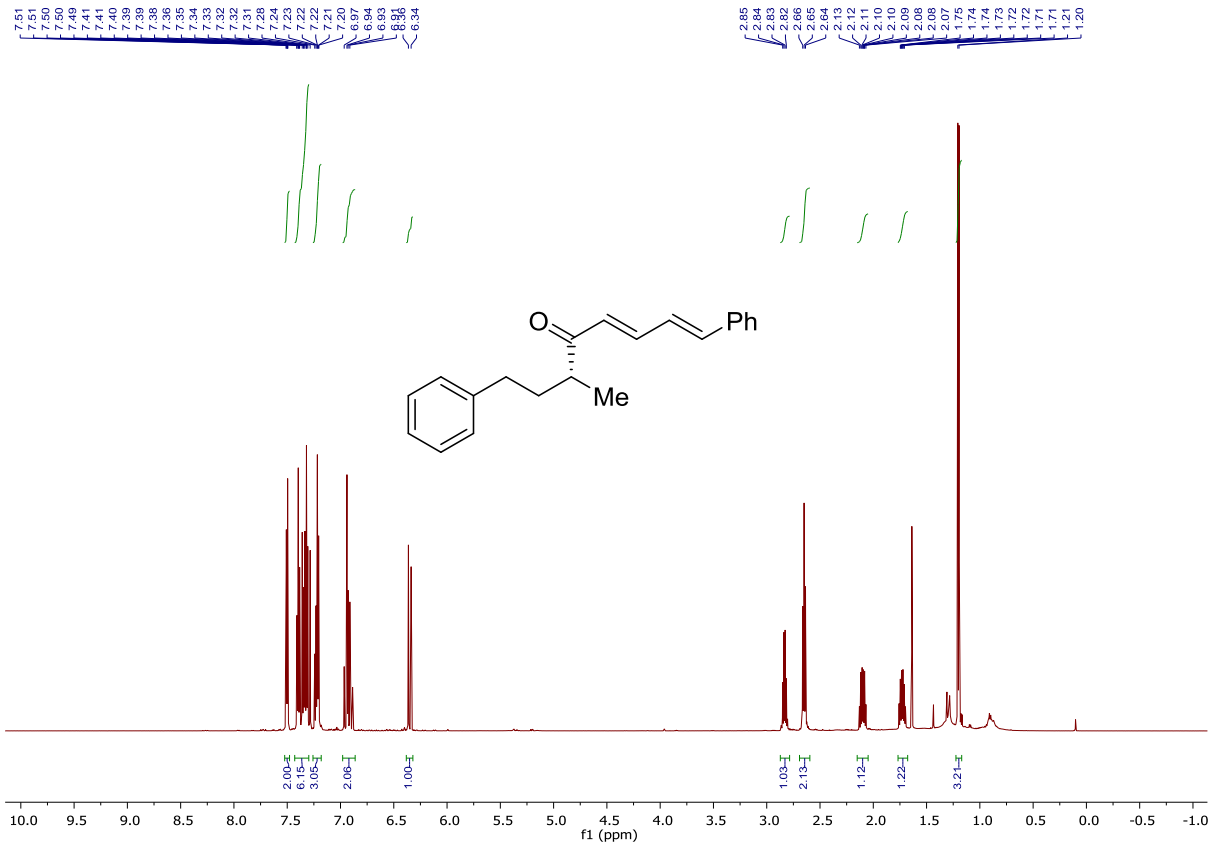
^{1}H and ^{13}C NMR Spectra



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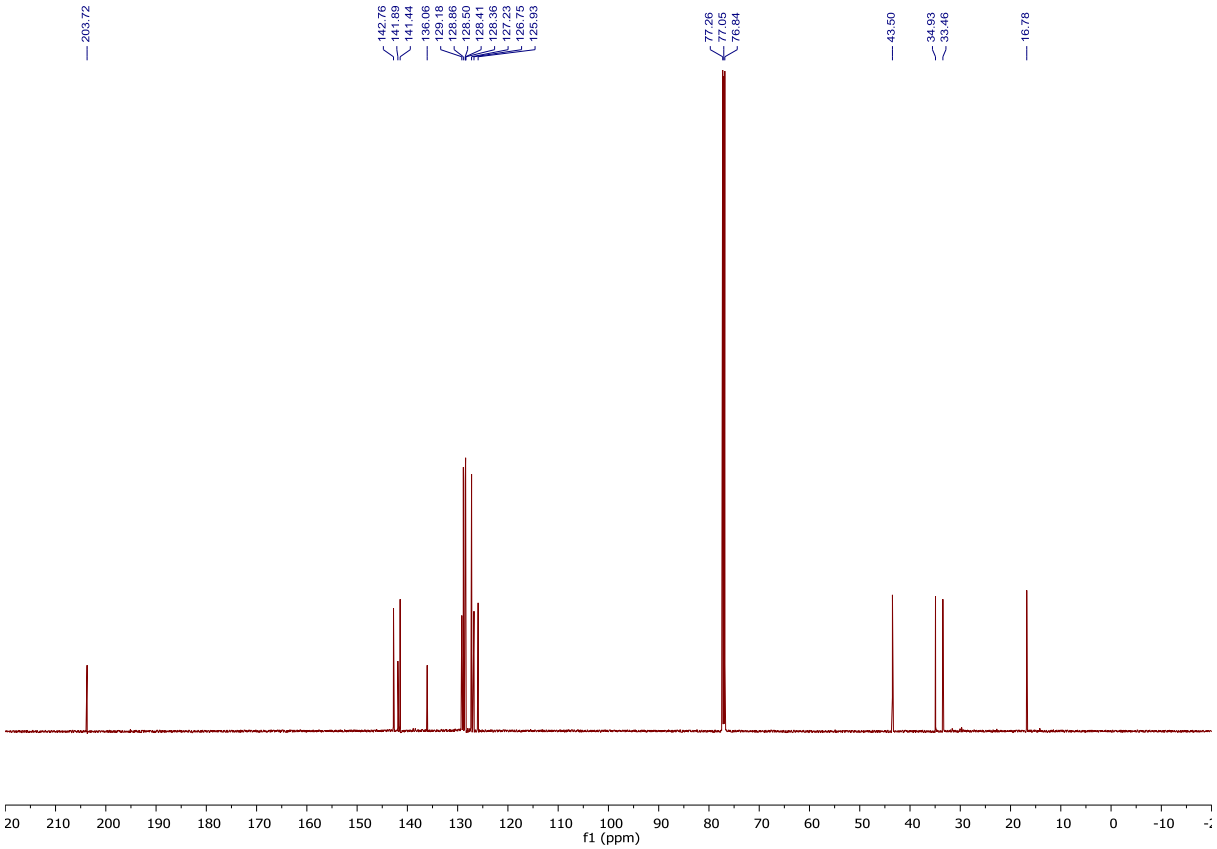
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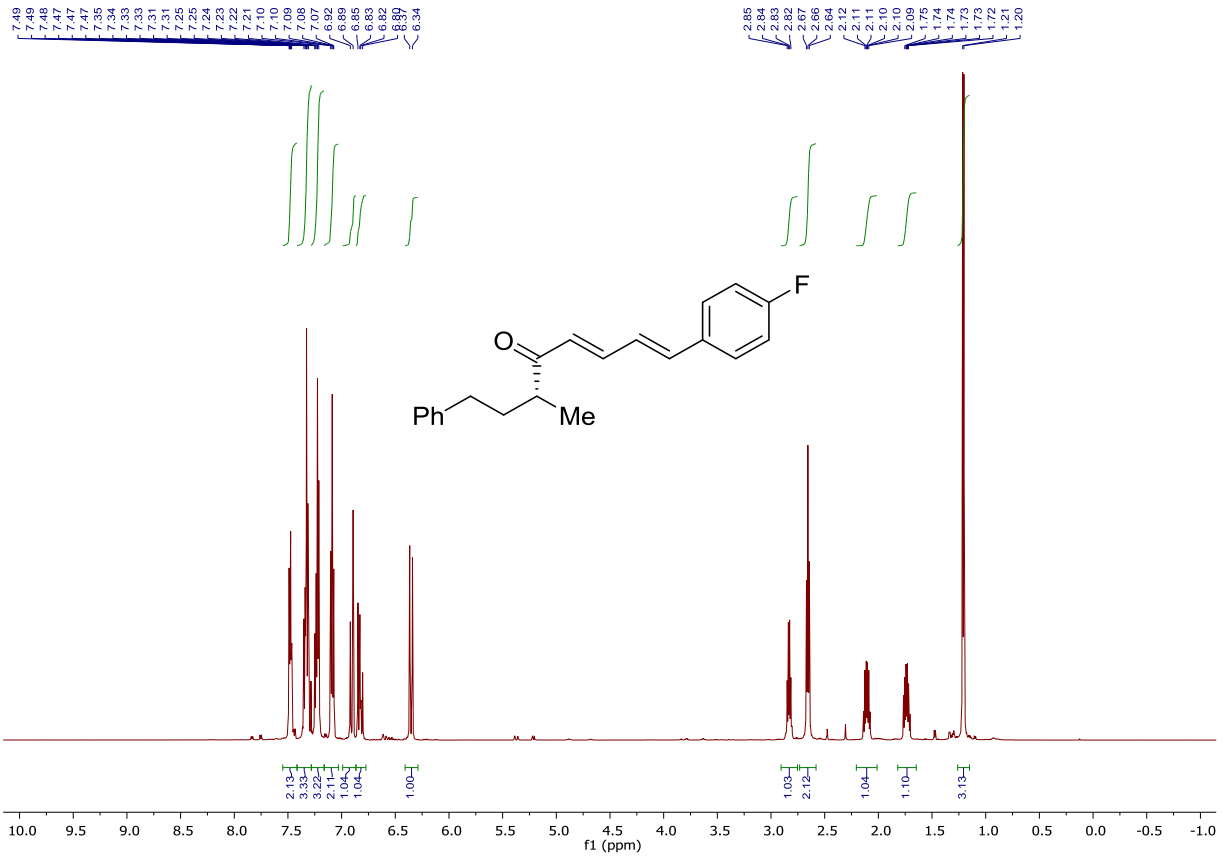
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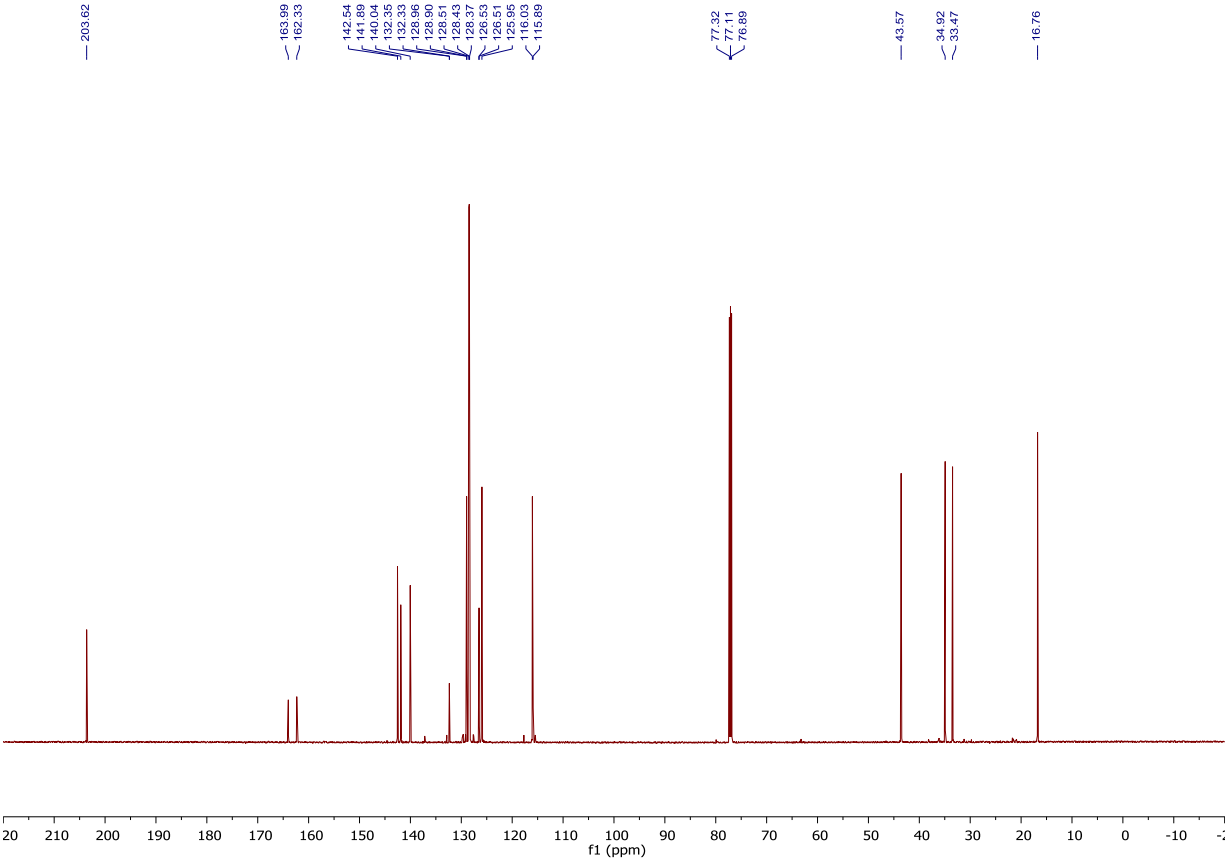
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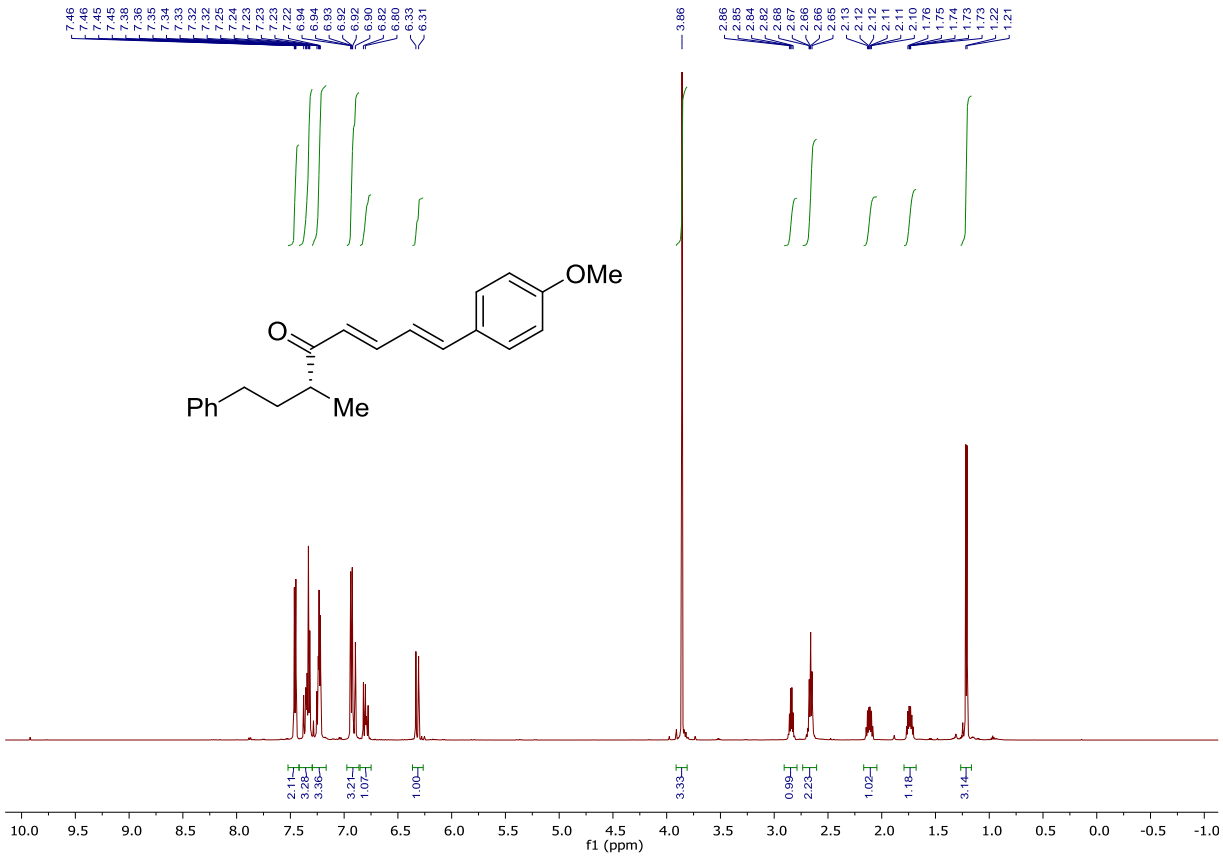
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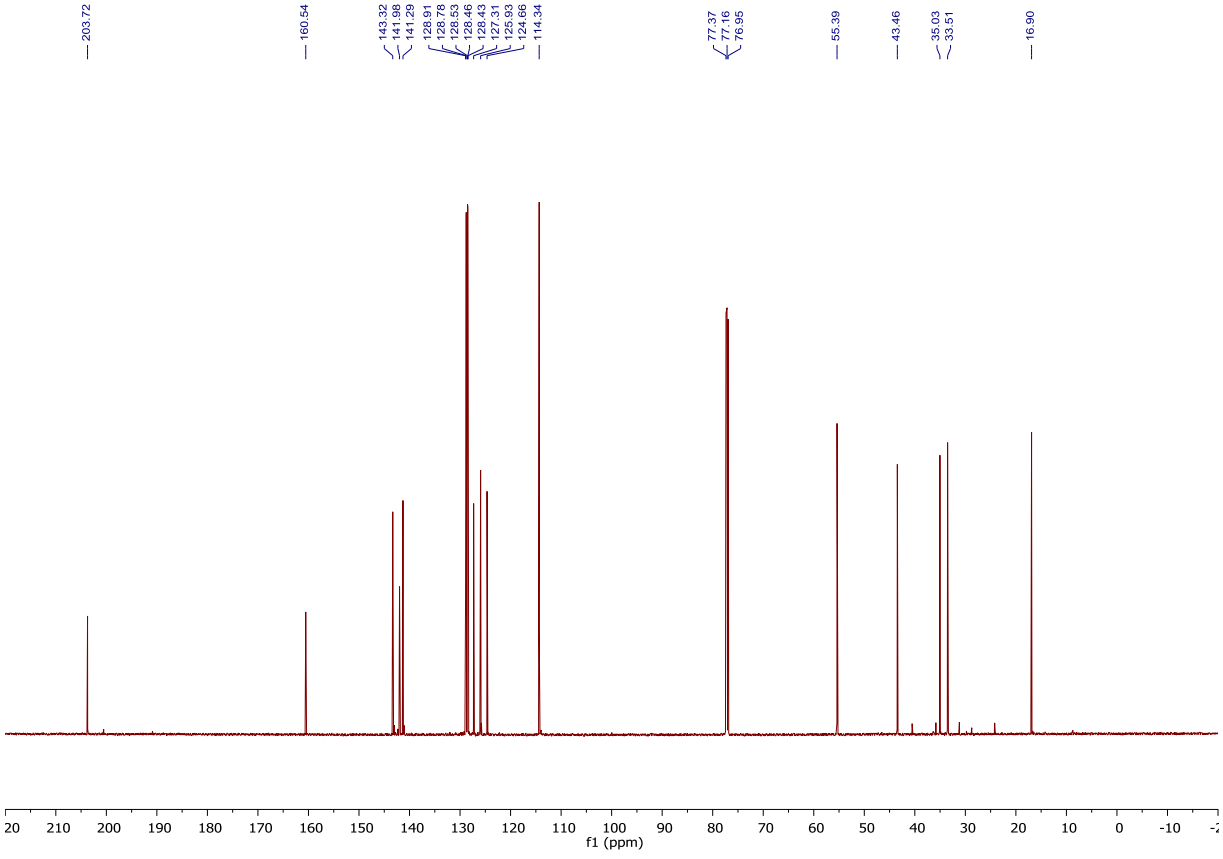
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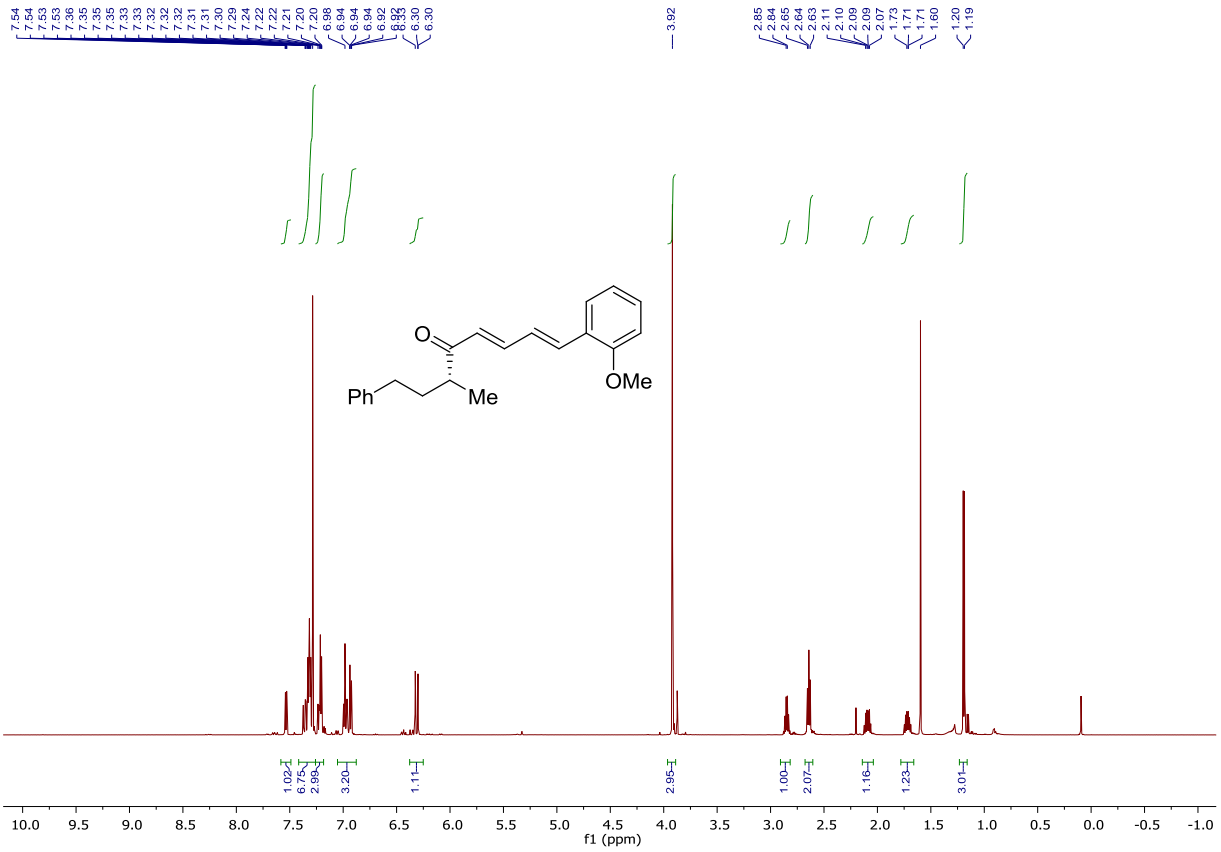
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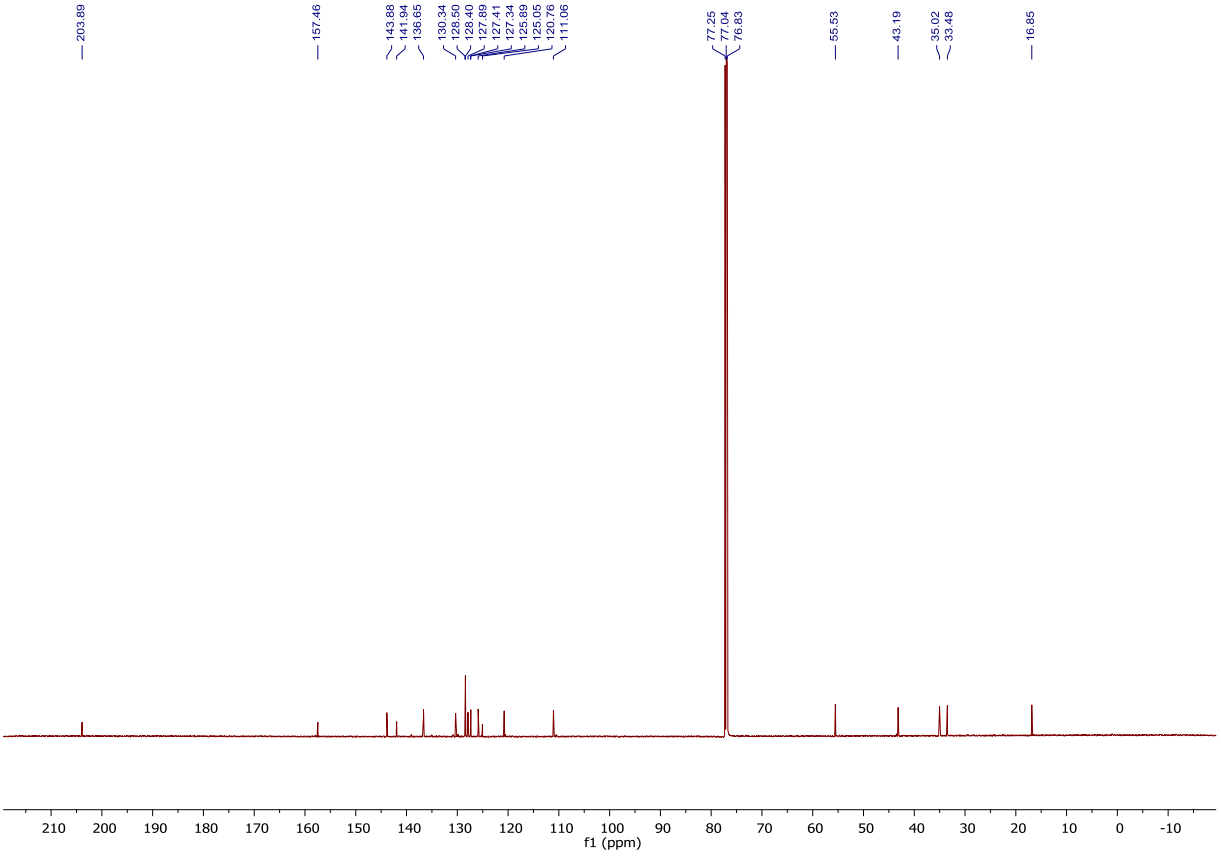
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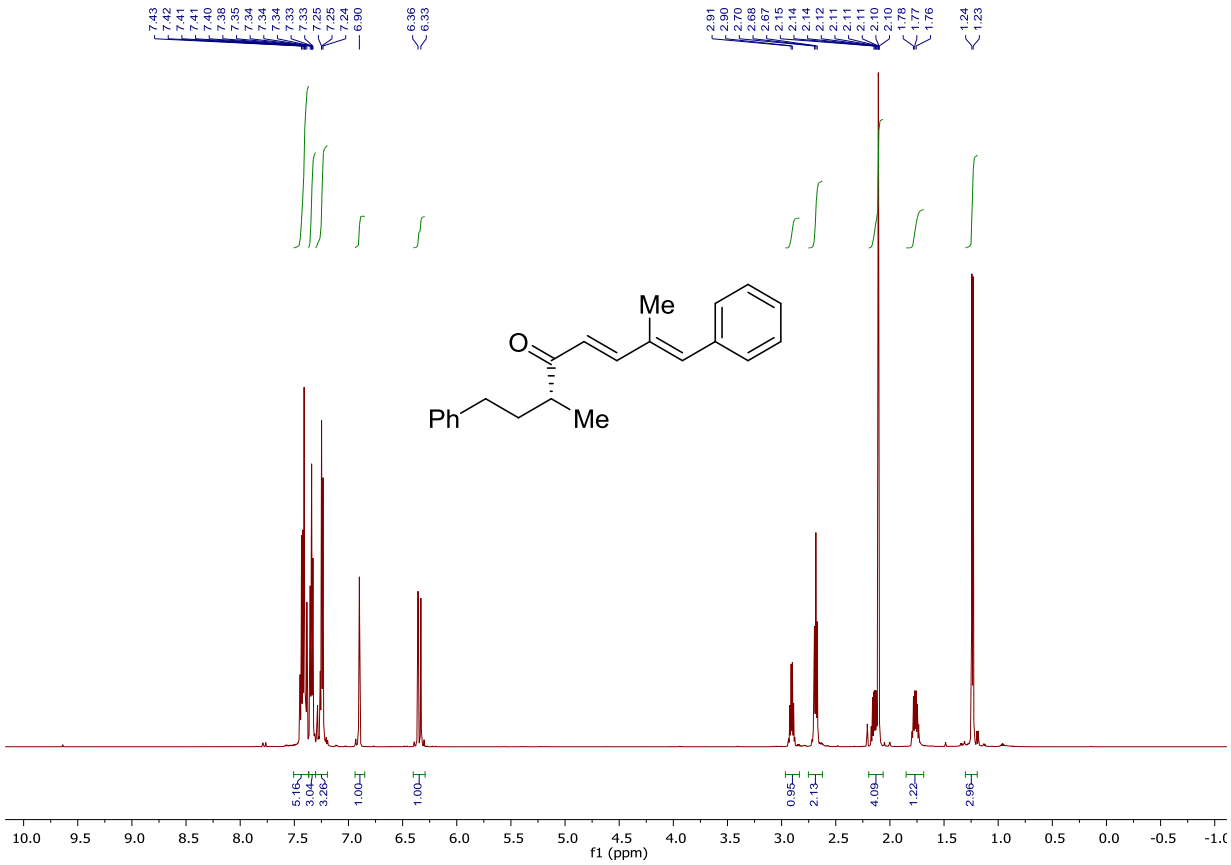
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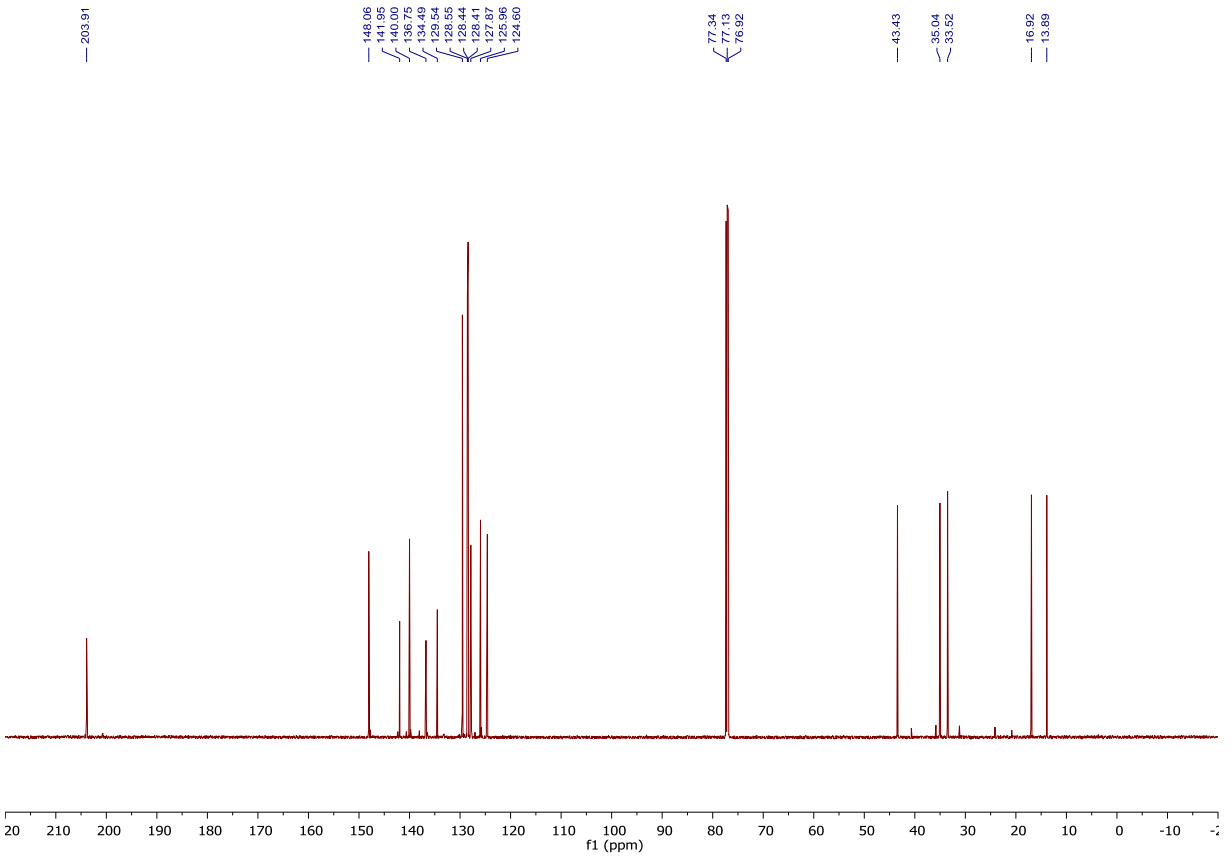
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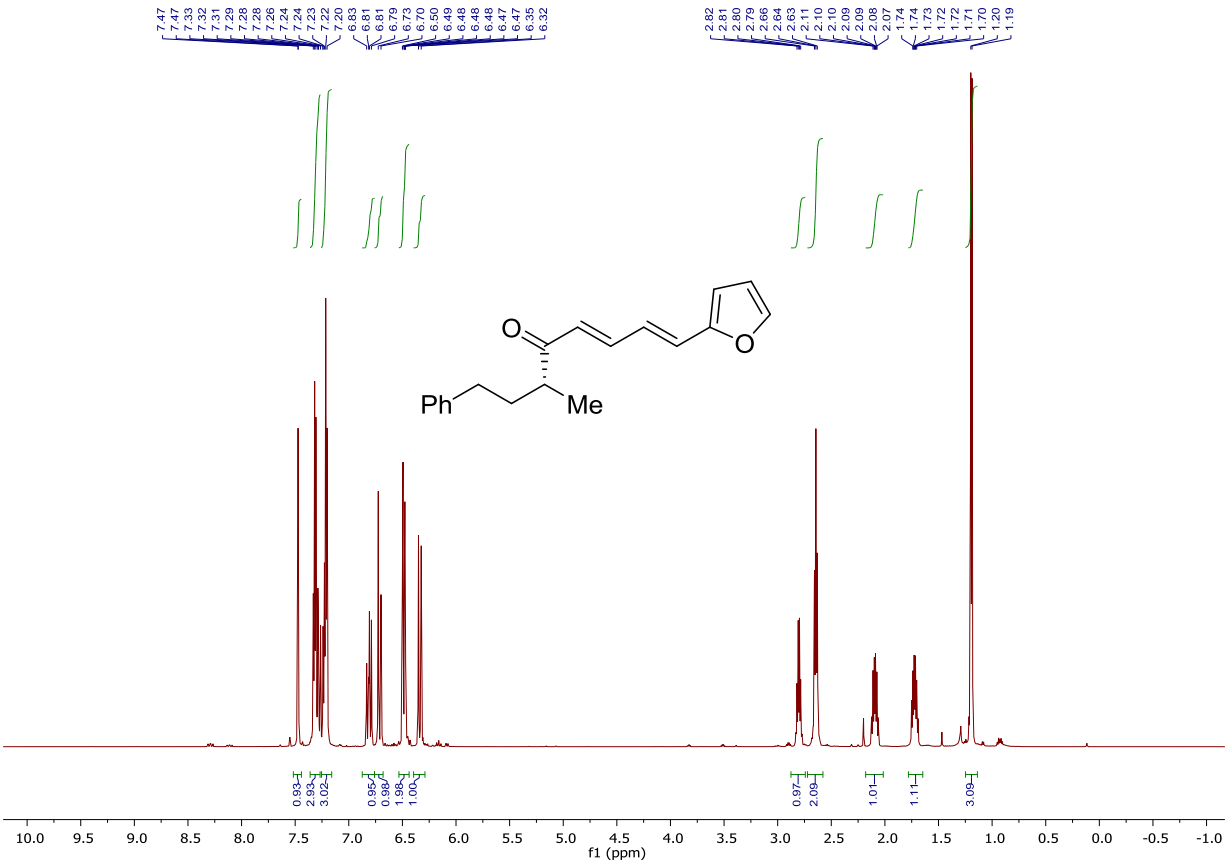
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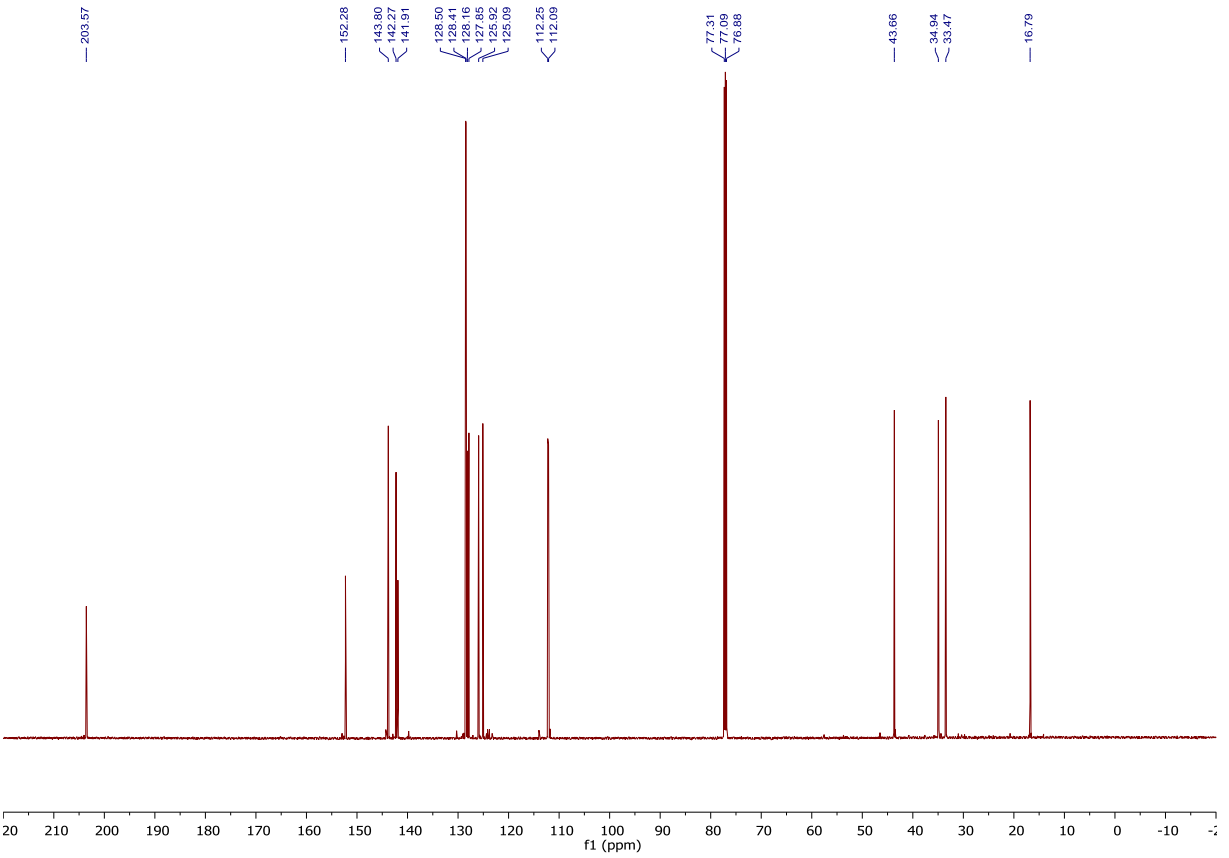
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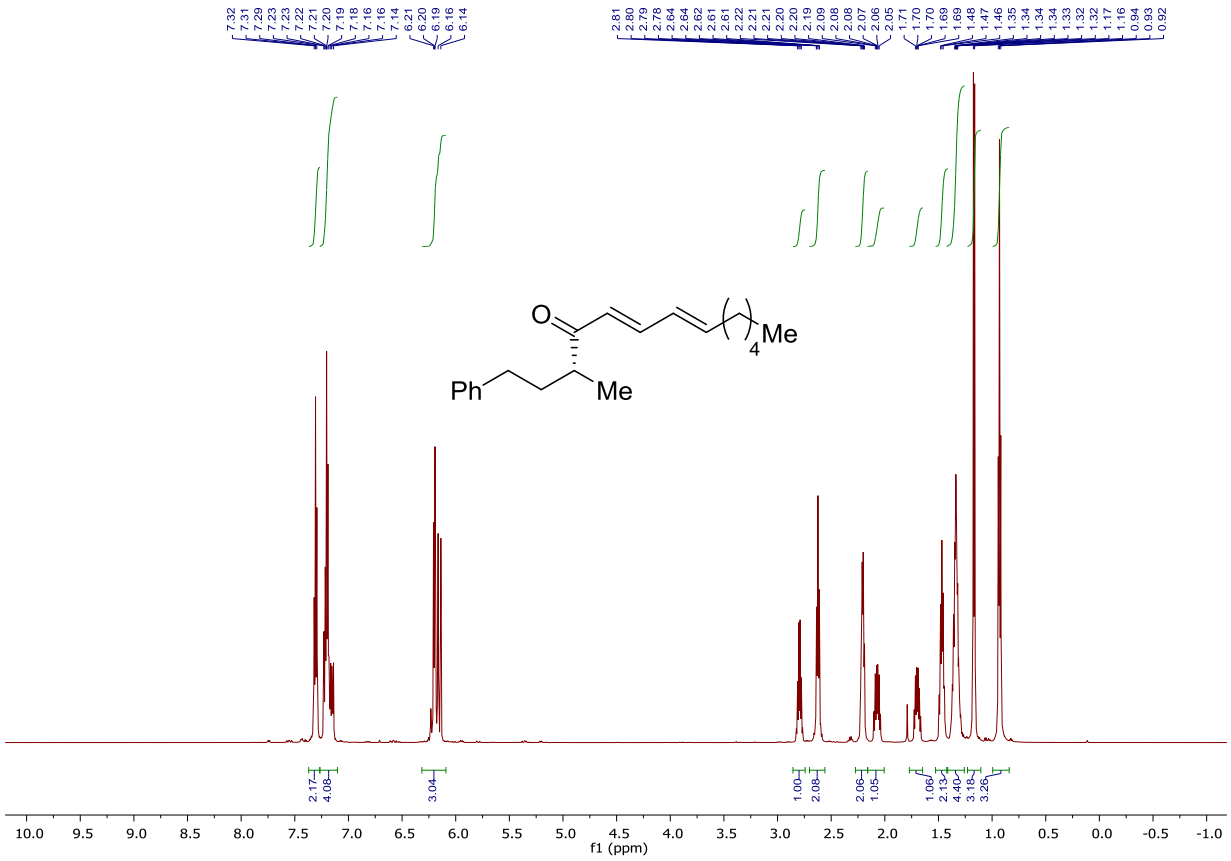
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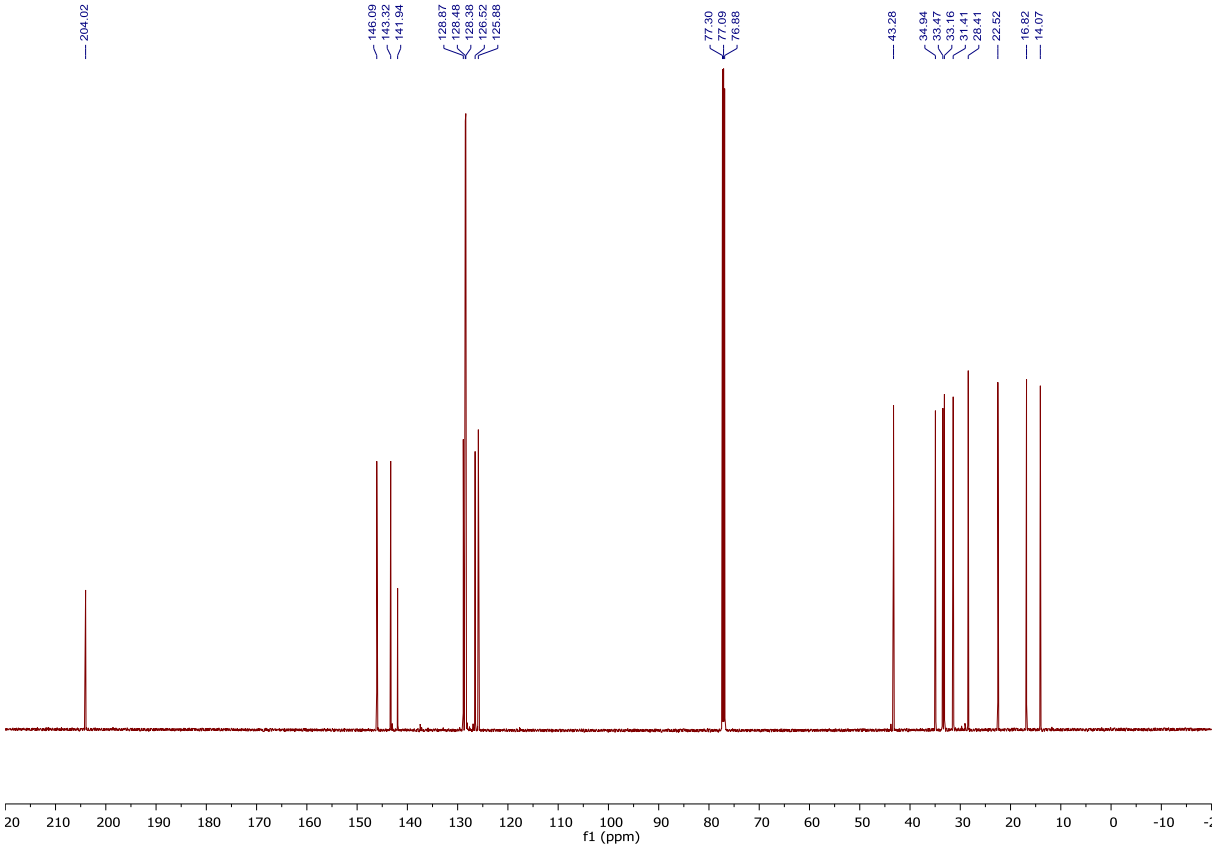
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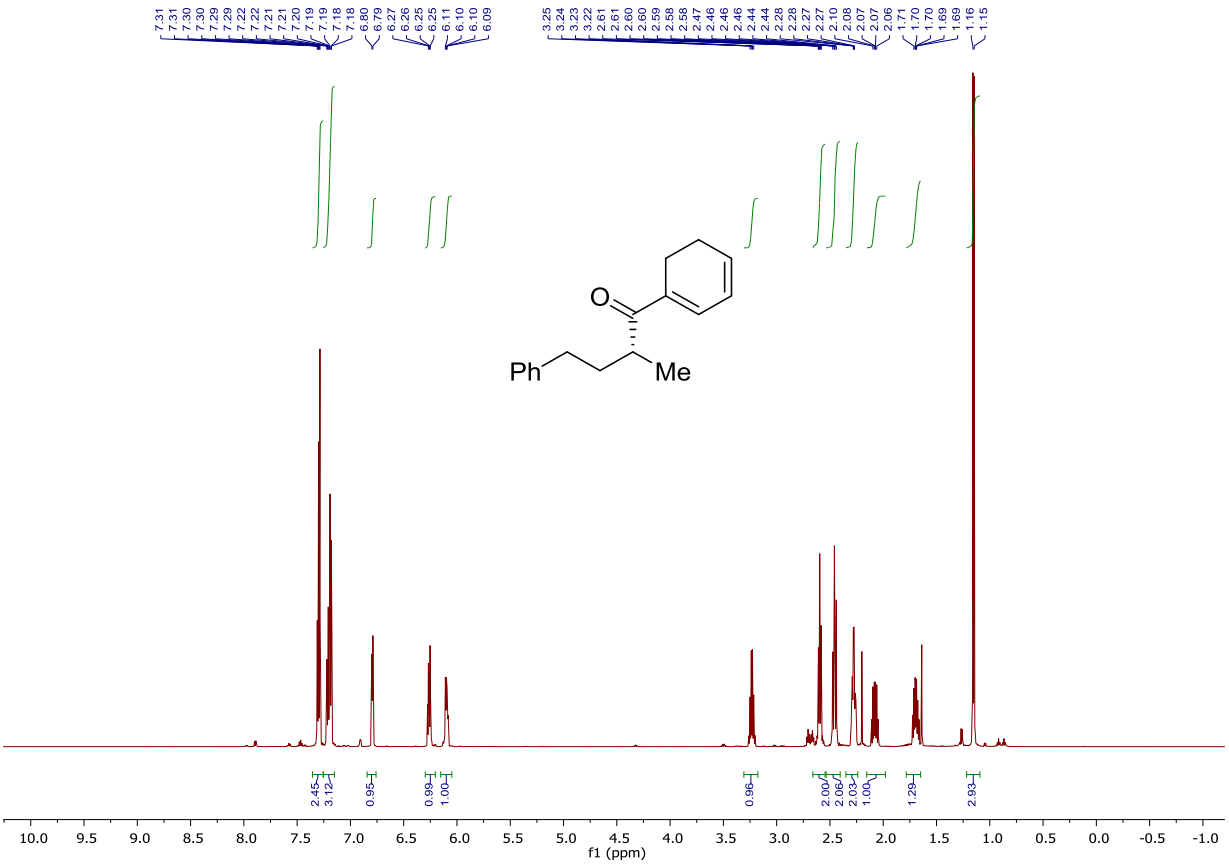
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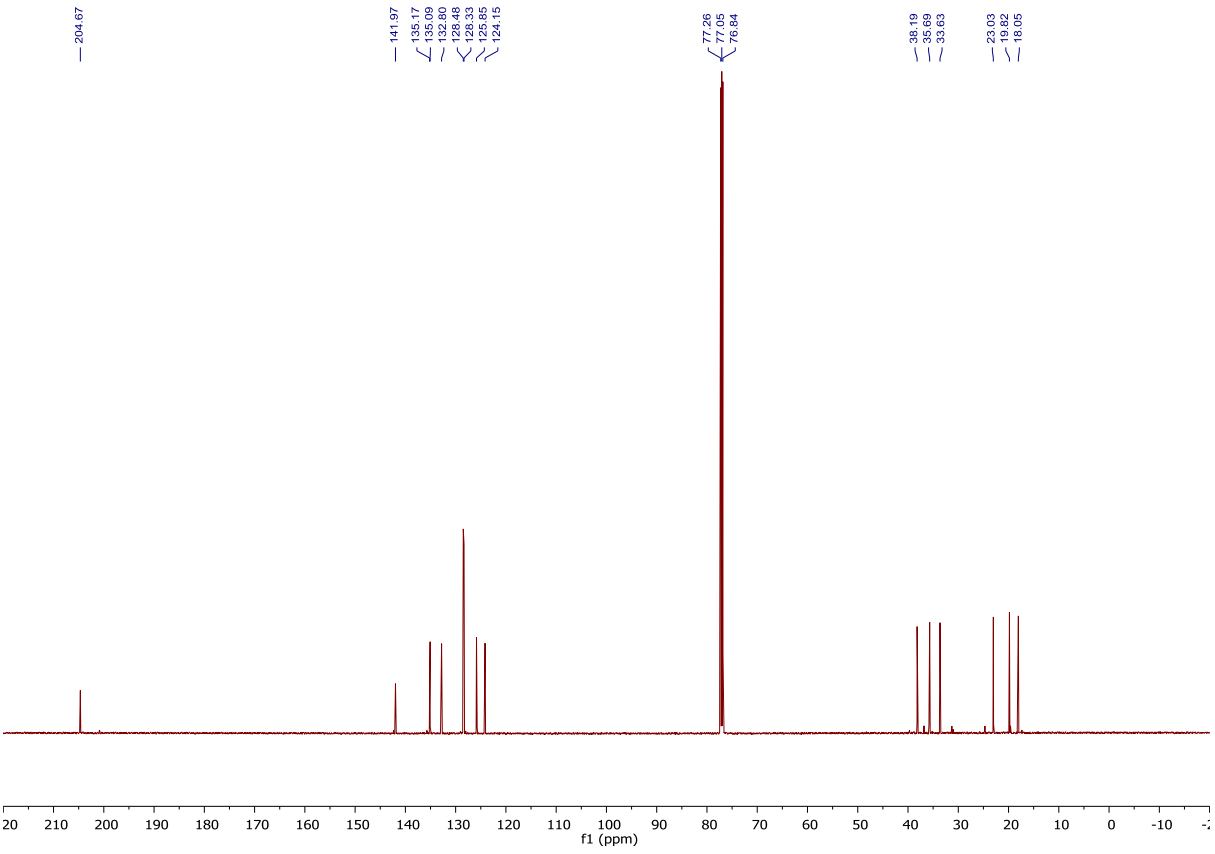
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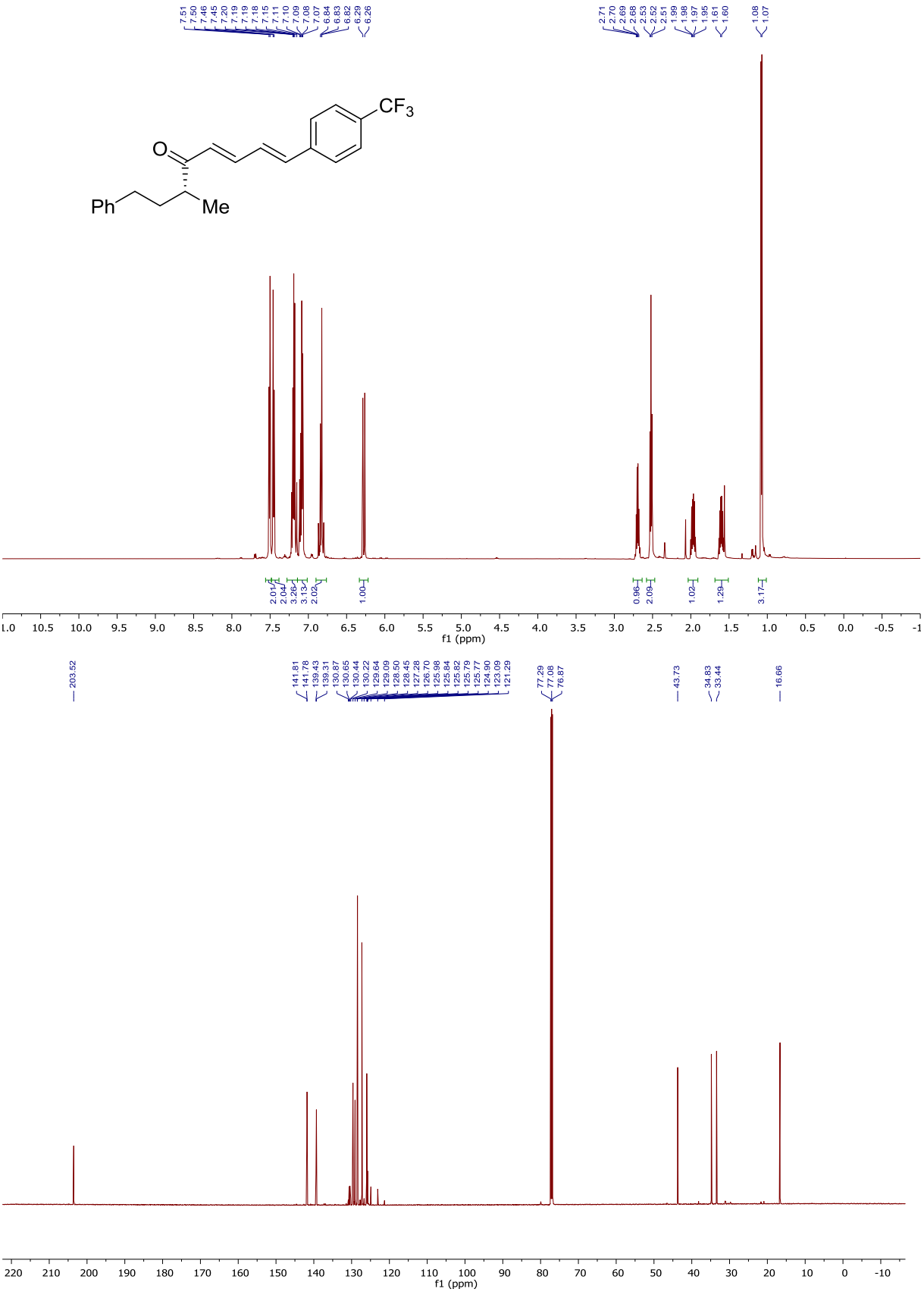
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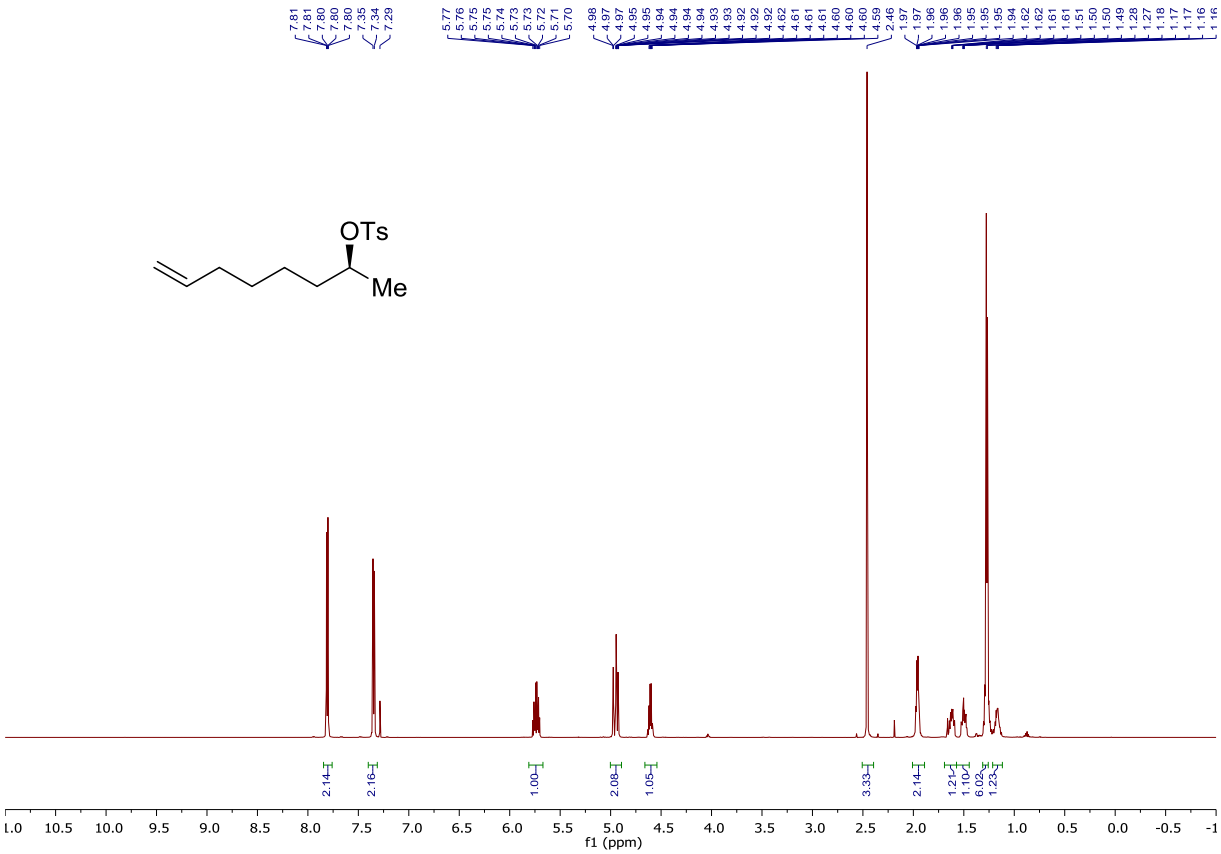
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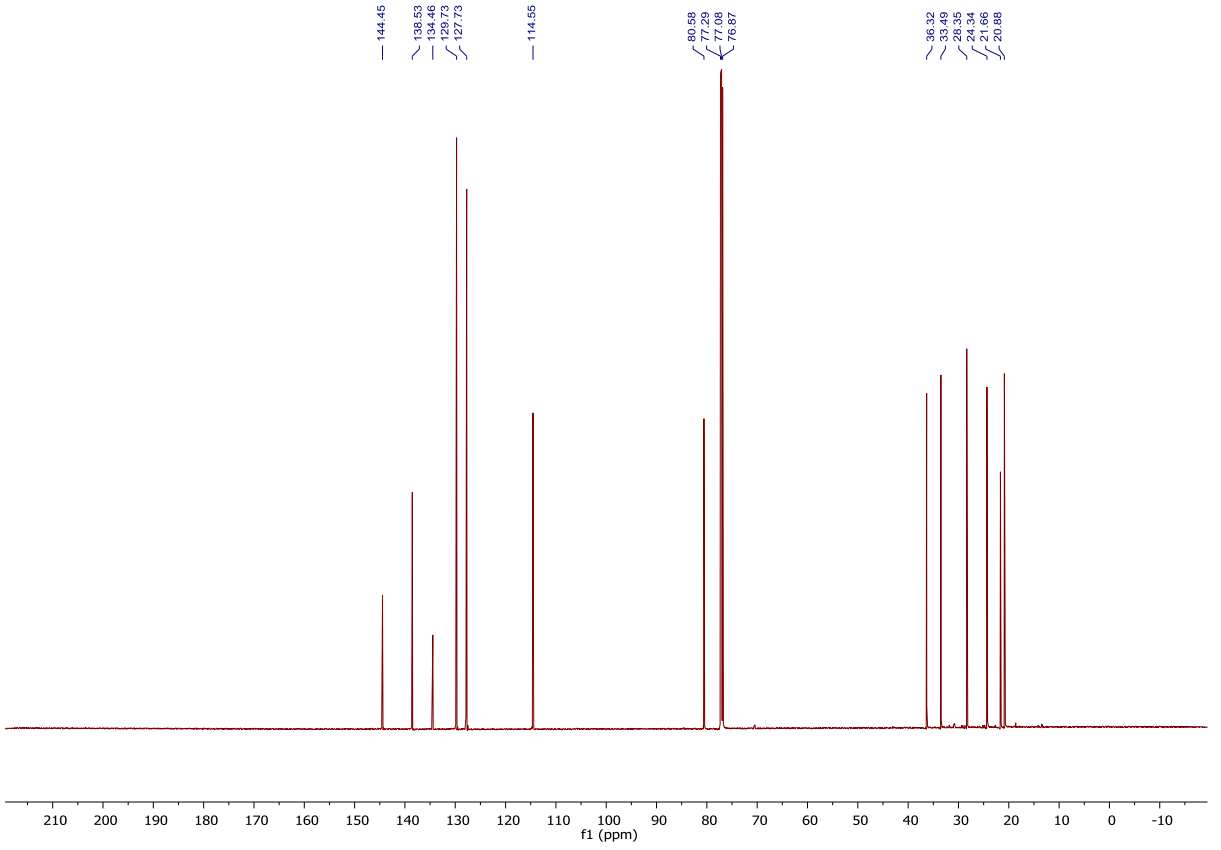
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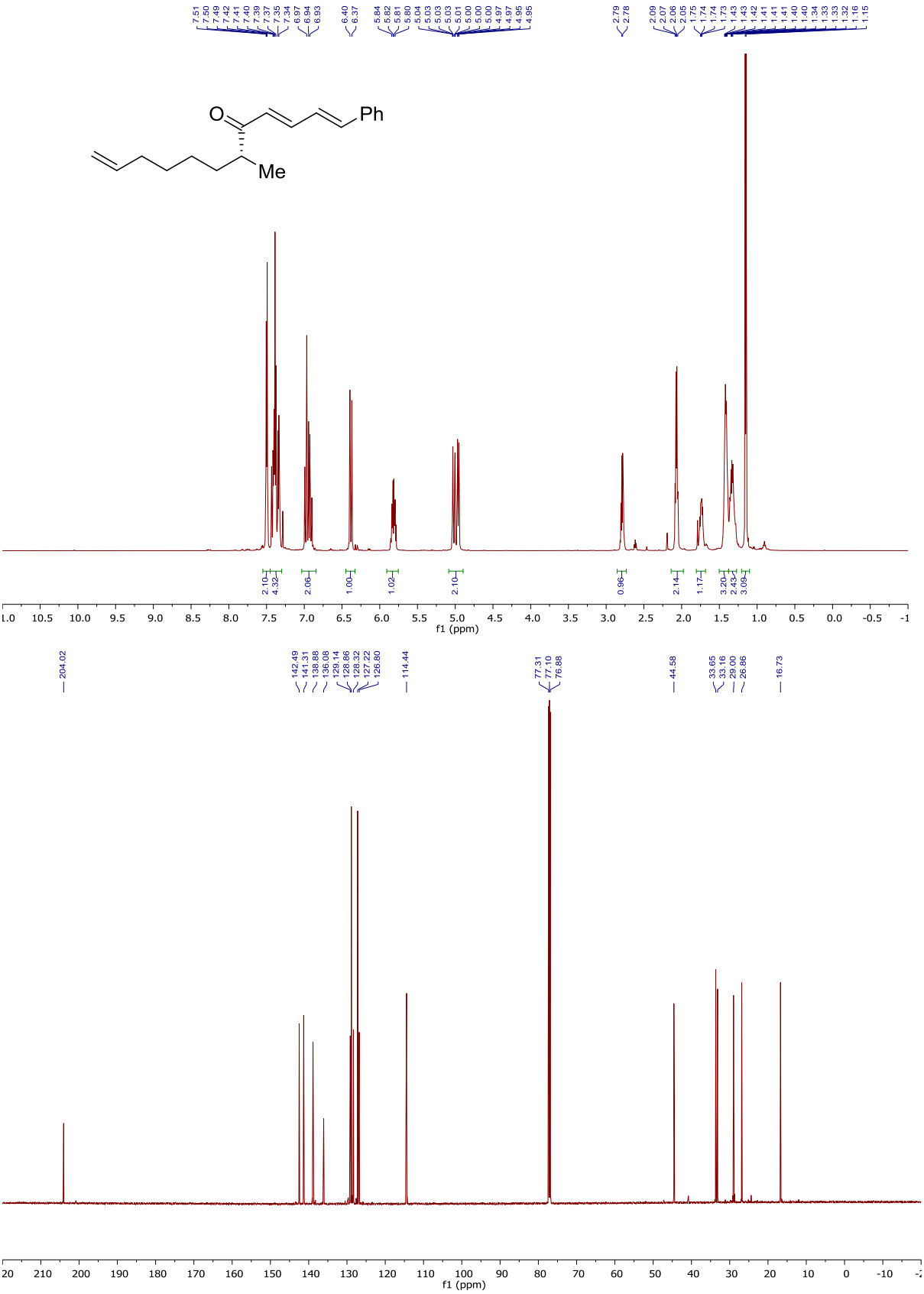
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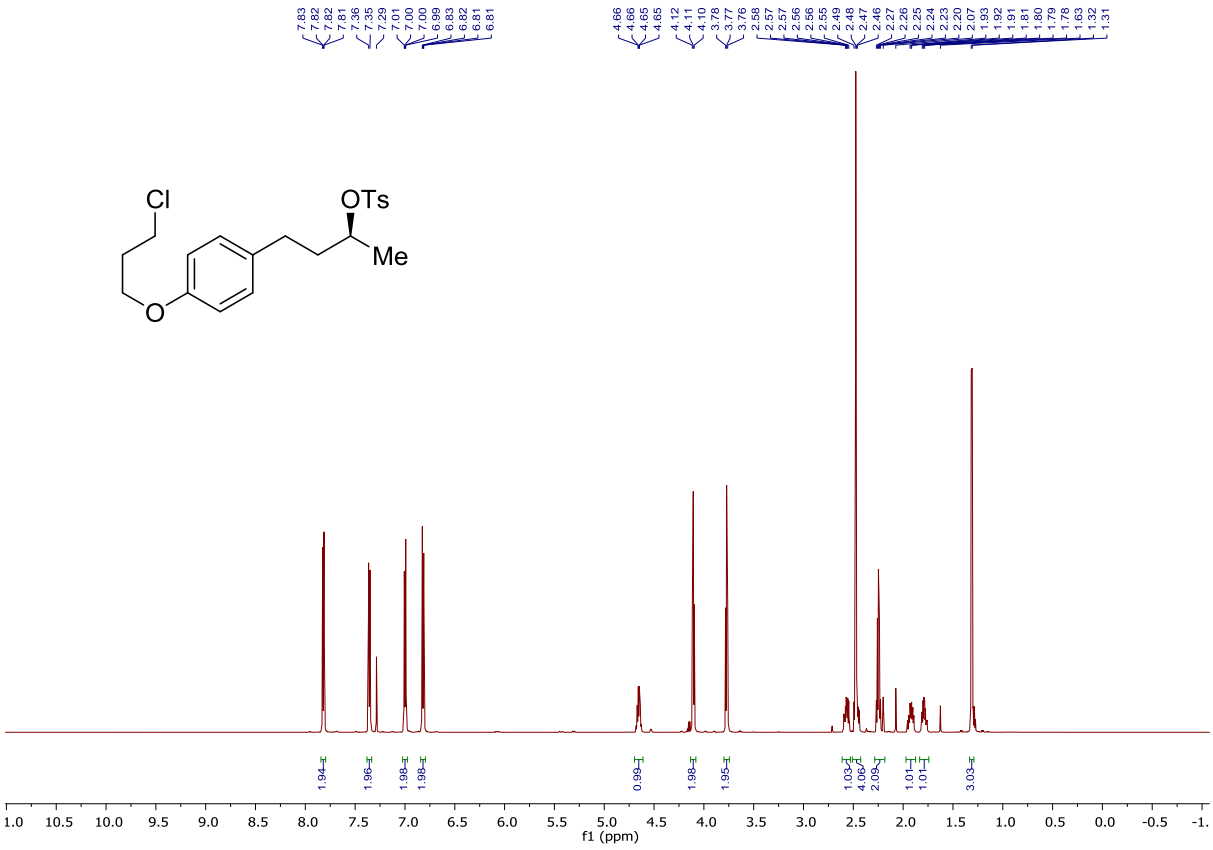
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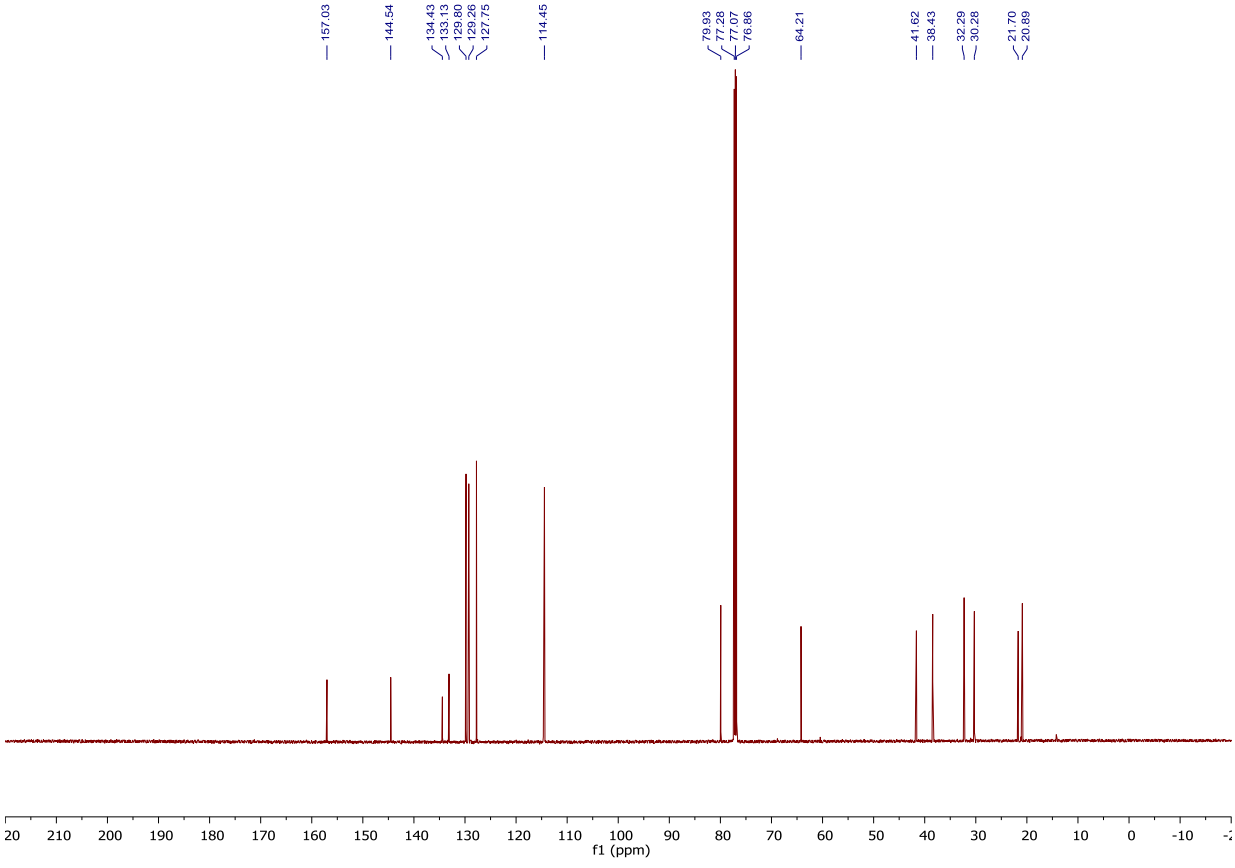
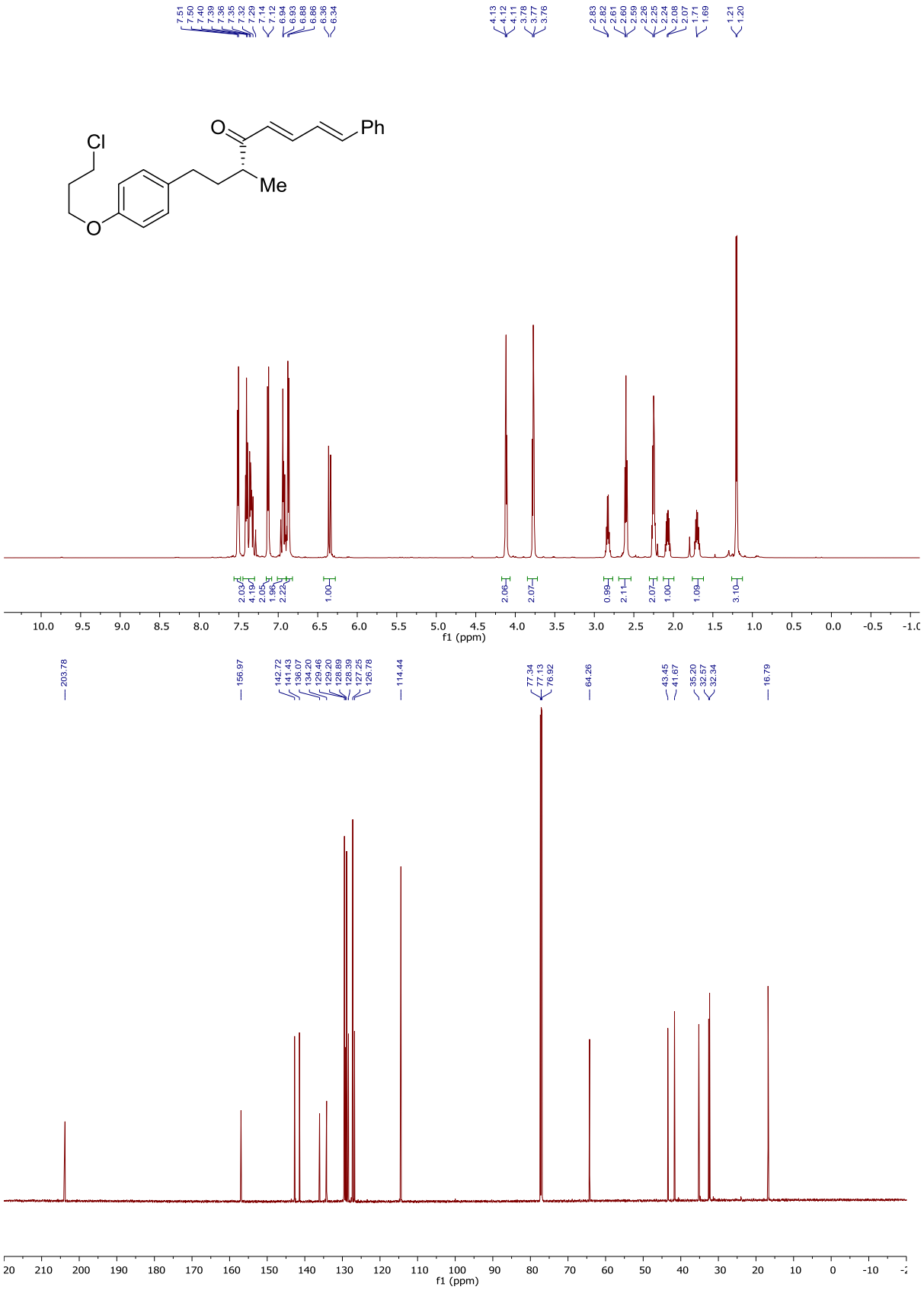
O
f1 (ppm)


f1 (ppm)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7.32
7.20
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7.12
6.94
6.93
6.88
6.86
6.36
6.34
4.13
4.12
4.11
3.78
3.77
3.76
2.83
2.82
2.61
2.60
2.59
2.26
2.25
2.24
2.08
2.07
1.71
1.69
1.21
1.20
Cl
O
Ph
Me
O
2.03
2.19
2.05
1.96
2.22
1.00
2.06
2.07
0.99
2.11
2.07
1.00
1.09
3.10
f1 (ppm)
203.78
156.97
142.72
141.43
136.07
134.20
129.46
129.20
128.89
128.39
127.25
126.78
114.44
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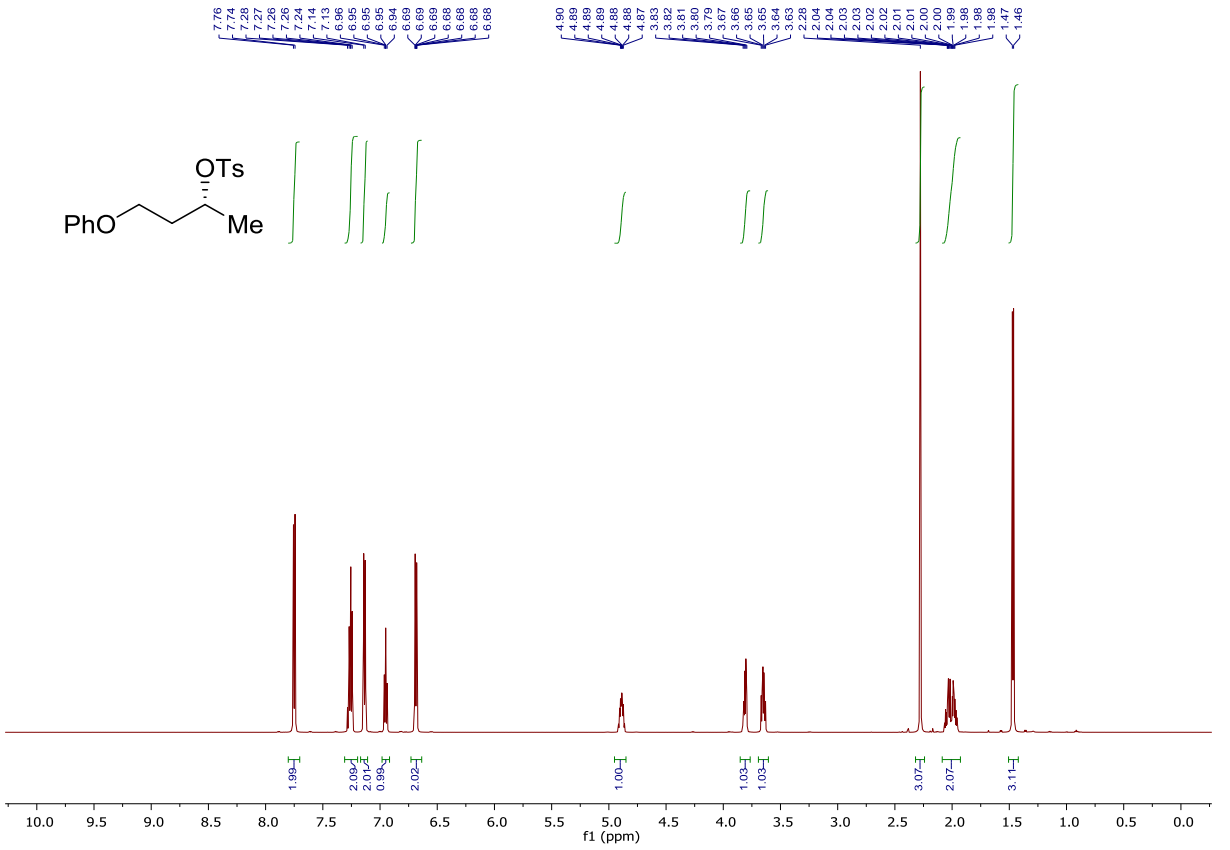
76.92
64.26
43.45
41.67
35.20
32.57
32.34
16.79
f1 (ppm)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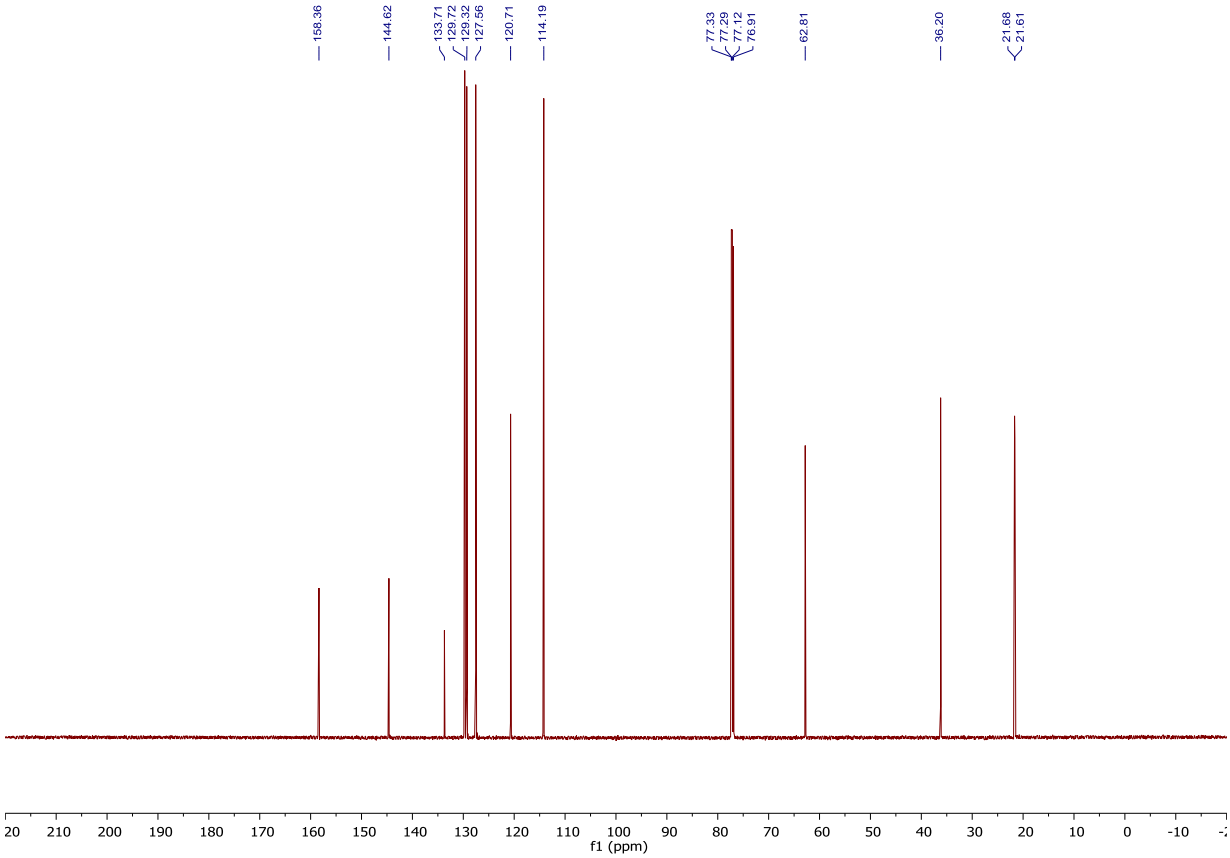
PhO
Me
f1 (ppm)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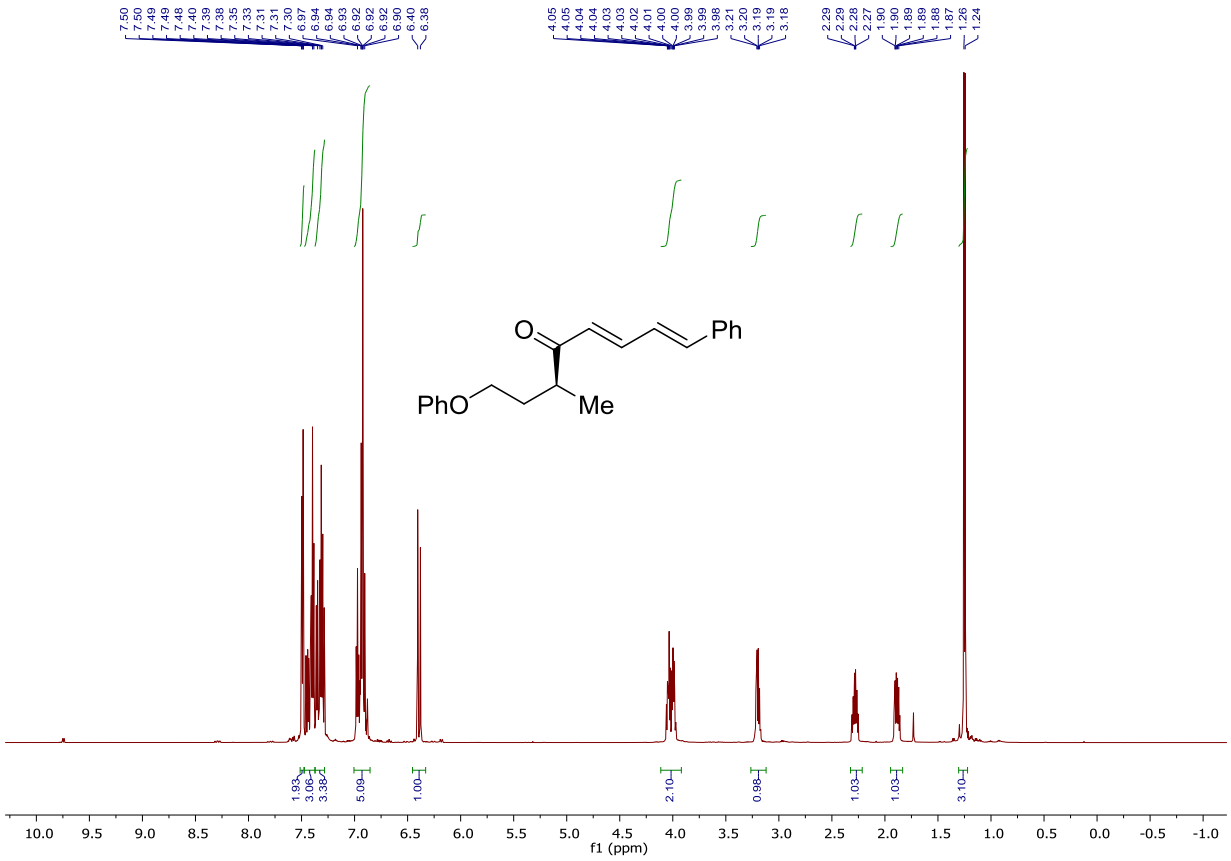
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6.92
6.92
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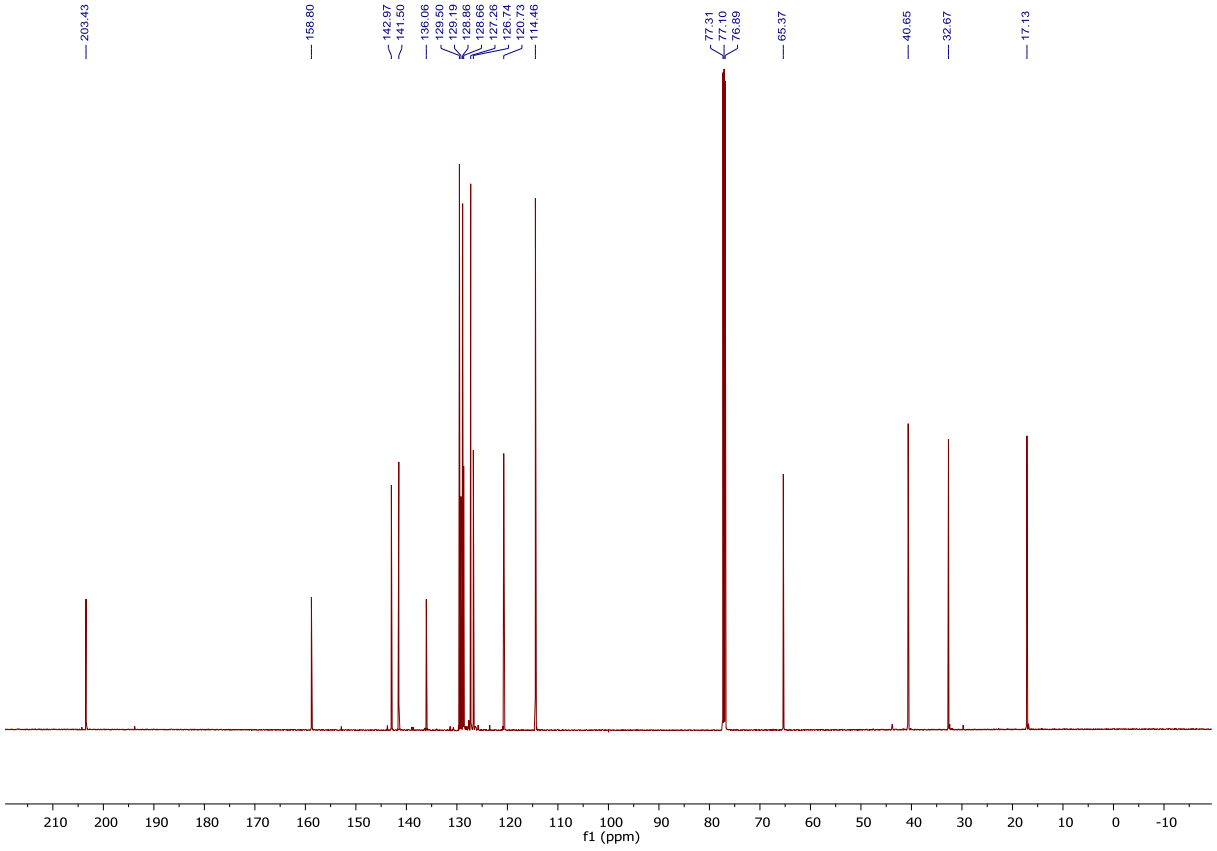
6.40
6.38
4.05
4.05
4.04
4.04
4.03
4.03
4.02
4.01
4.00
4.00
3.99
3.99
3.98
3.21
3.20
3.19
3.19
3.18
2.29
2.29
2.28
2.27
1.90
1.90
1.89
1.89
1.88
1.87
1.26
1.24
O
Ph
PhO
Me
1.93
3.06
3.38
5.09
1.00
2.10
0.98
1.03
1.03
3.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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128.66
127.26
126.74
120.73
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77.31
77.10
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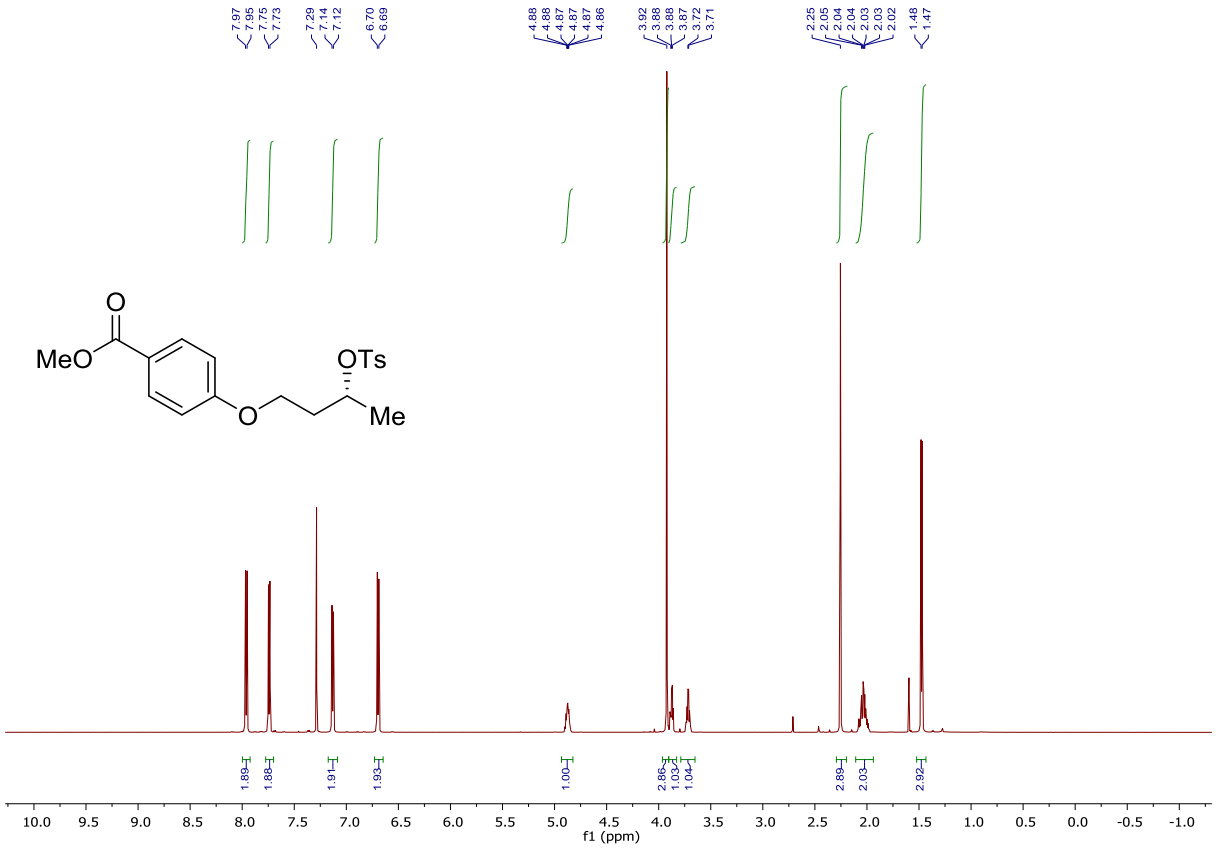
32.67
17.13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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6.69
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4.88
4.87
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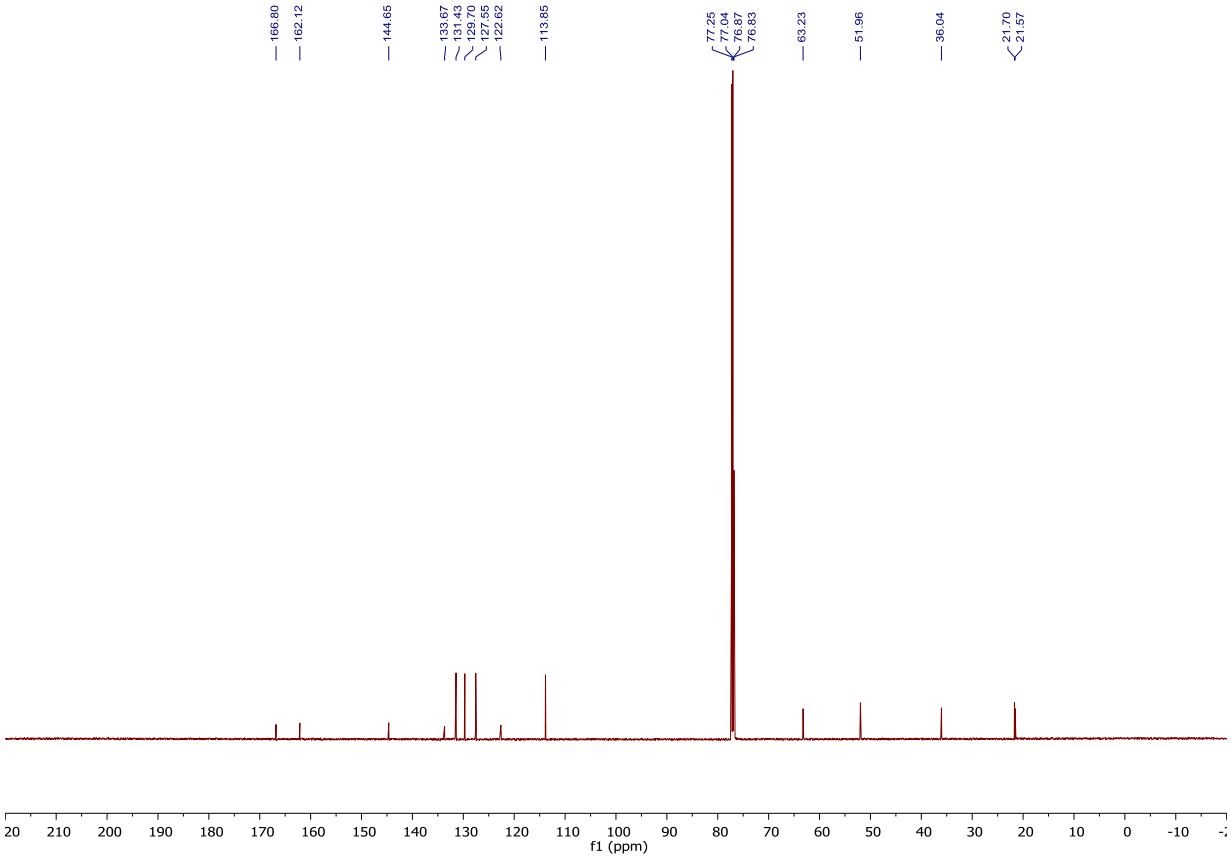
4.87
4.86
3.92
3.88
3.88
3.87
3.72
3.71
2.25
2.05
2.04
2.04
2.03
2.03
2.02
1.48
1.47
O
MeO
OTs
O
Me
1.89
1.88
1.91
1.93
1.00
2.86
1.03
1.04
2.89
2.03
2.92
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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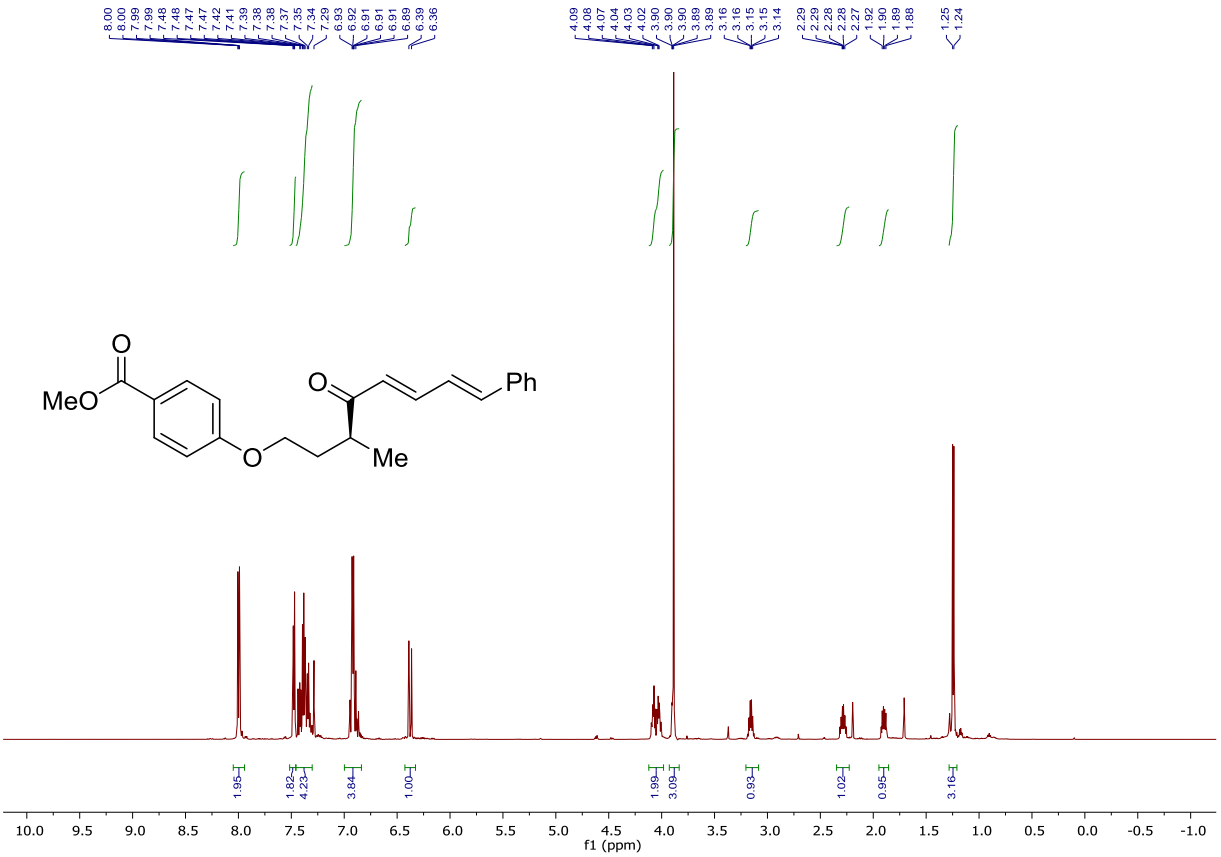
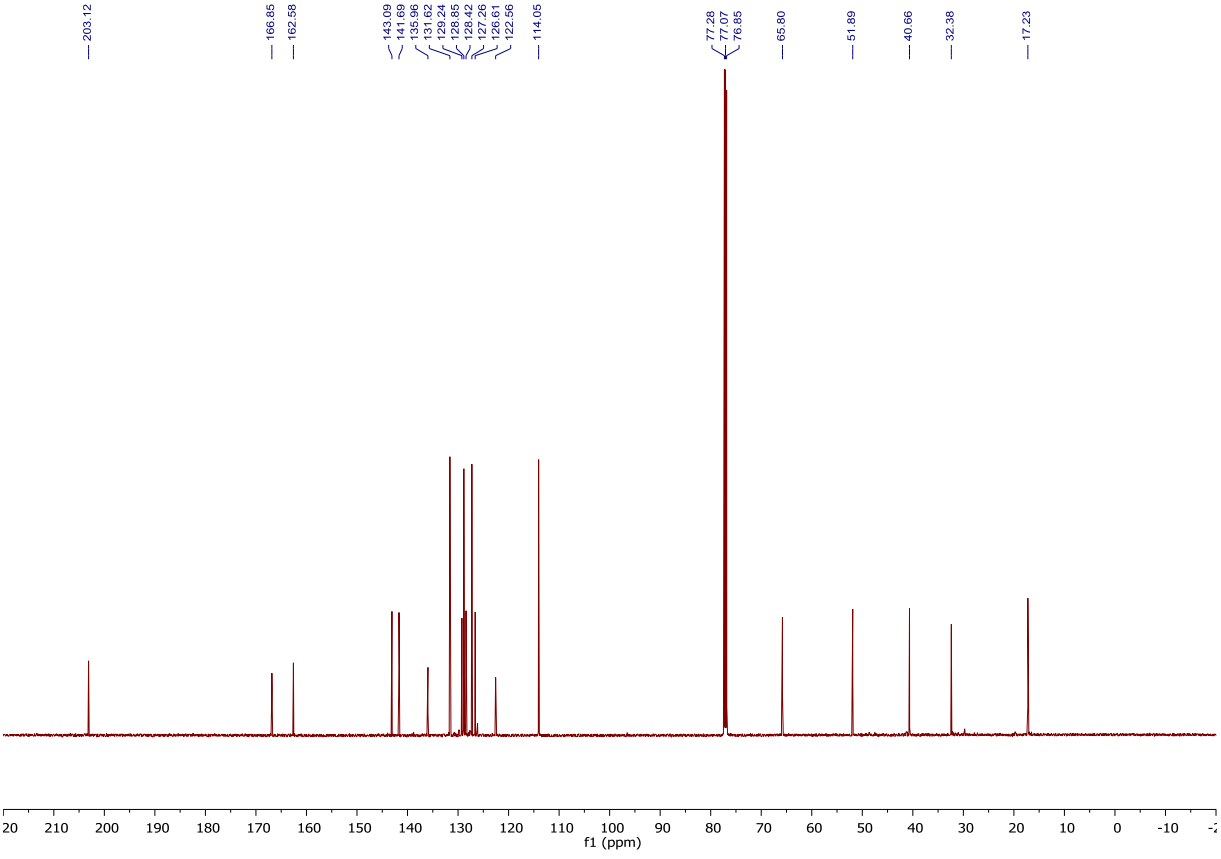
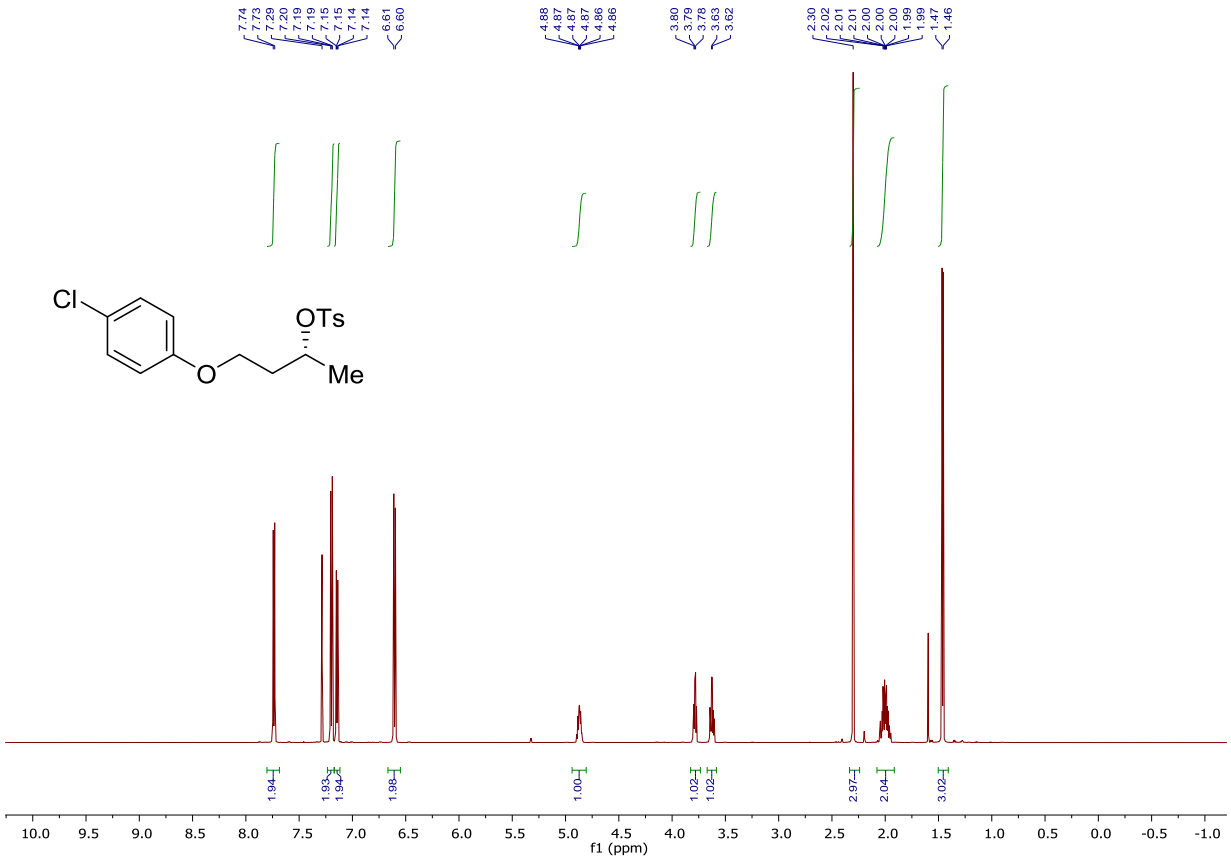
129.70
127.55
122.62
113.85
77.25
77.04
76.87
76.83
63.23
51.96
36.04
21.70
21.57
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.00 8.00 7.99 7.99 7.48 7.48 7.47 7.47 7.42 7.41 7.39 7.38 7.38 7.37 7.35 7.34 7.29 6.93 6.92 6.91 6.91 6.91 6.89 6.39 6.36
4.09 4.08 4.07 4.04 4.03 4.02 3.90 3.90 3.90 3.89 3.89 3.16 3.16 3.15 3.15 3.14 2.29 2.29 2.28 2.28 2.27 1.92 1.90 1.89 1.88 1.25 1.24
O
MeO
O
O
Ph
Me
1.95 1.82 4.23 3.84 1.00 1.99 3.09 0.93 1.02 0.95 3.16
10.0 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 -0.5 -1.0
f1 (ppm)


203.12 166.85 162.58 143.09 141.69 135.96 131.62 129.24 128.85 128.42 127.26 126.61 122.56 114.05 77.28 77.07 76.85 65.80 51.89 40.66 32.38 17.23
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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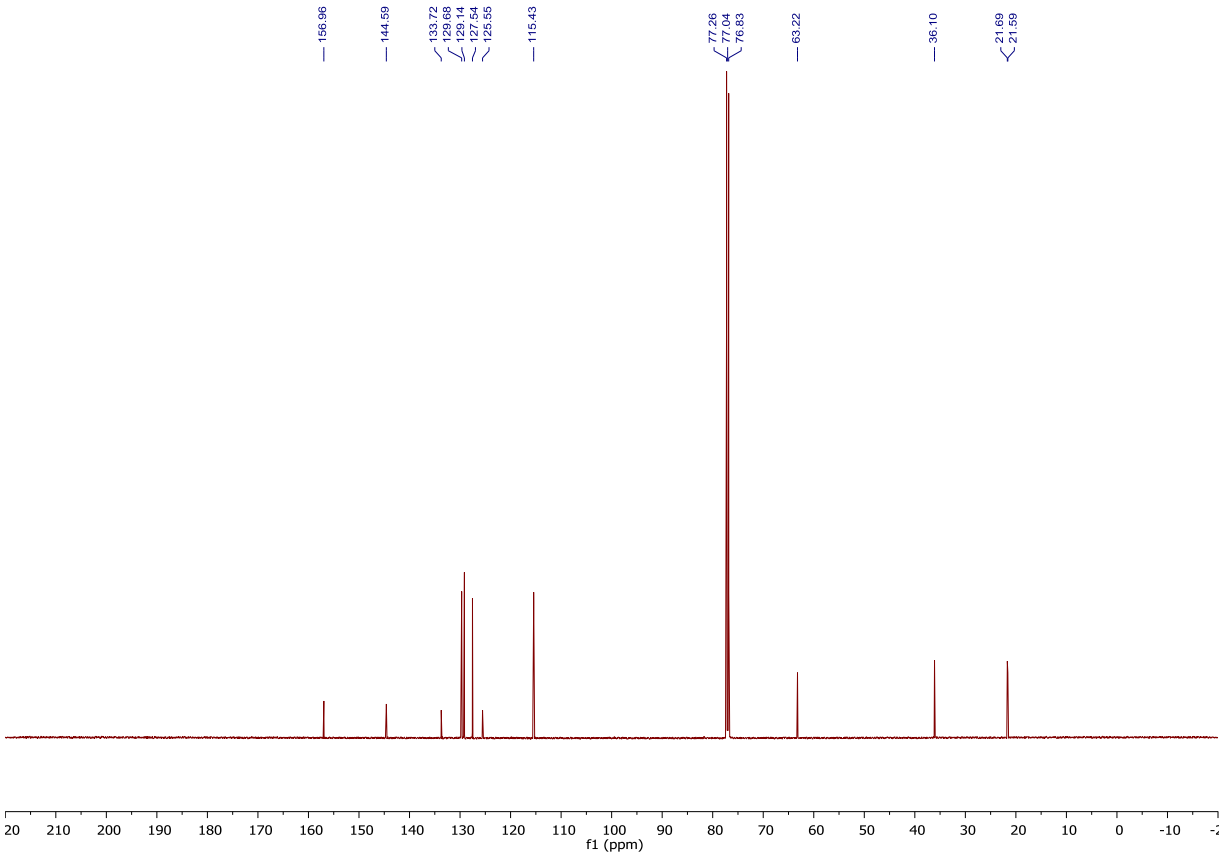
4.88
4.87
4.87
4.87
4.86
4.86
3.80
3.79
3.78
3.63
3.62
2.30
2.02
2.01
2.01
2.00
2.00
2.00
1.99
1.99
1.47
1.46
Cl
OTs
O
Me
1.94
1.93
1.94
1.98
1.00
1.02
1.02
2.97
2.04
3.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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127.54
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115.43
77.26
77.04
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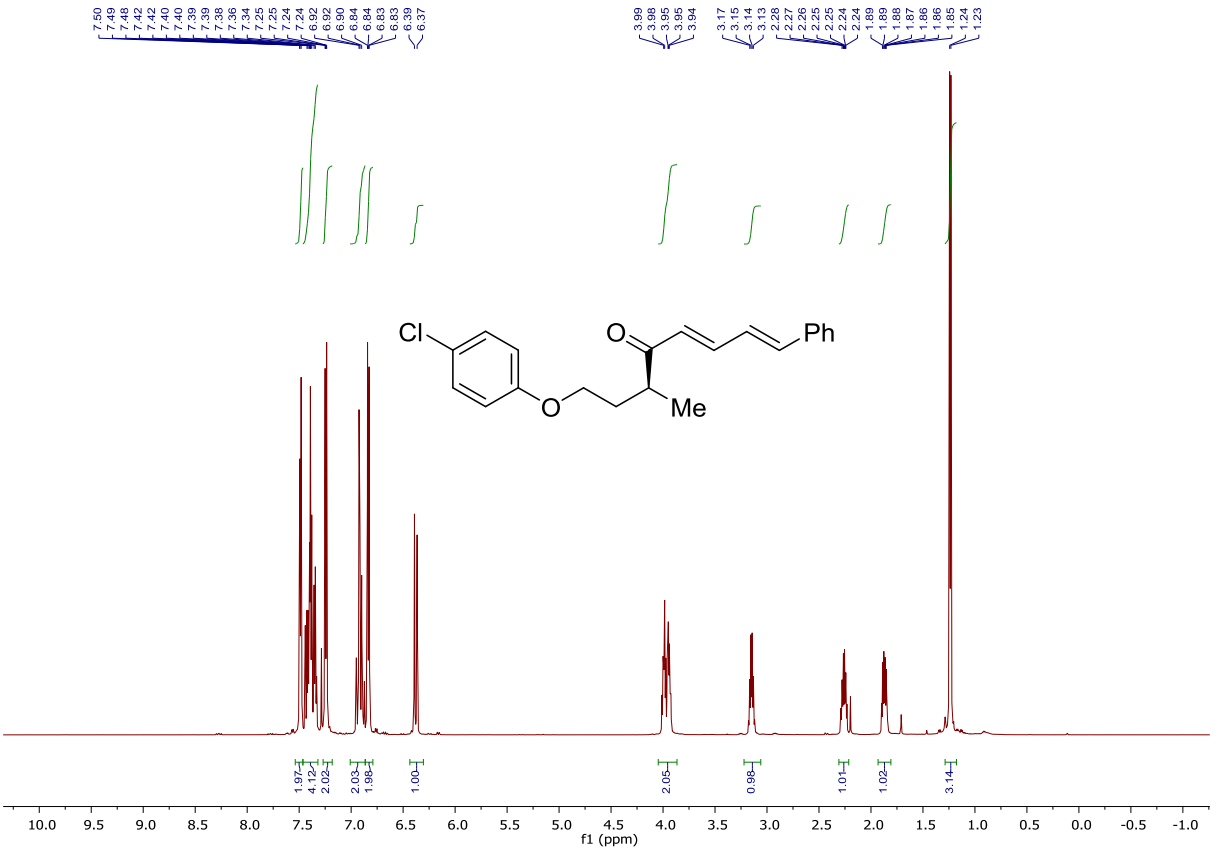
36.10
21.69
21.59
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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O
Ph
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Me
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1.02
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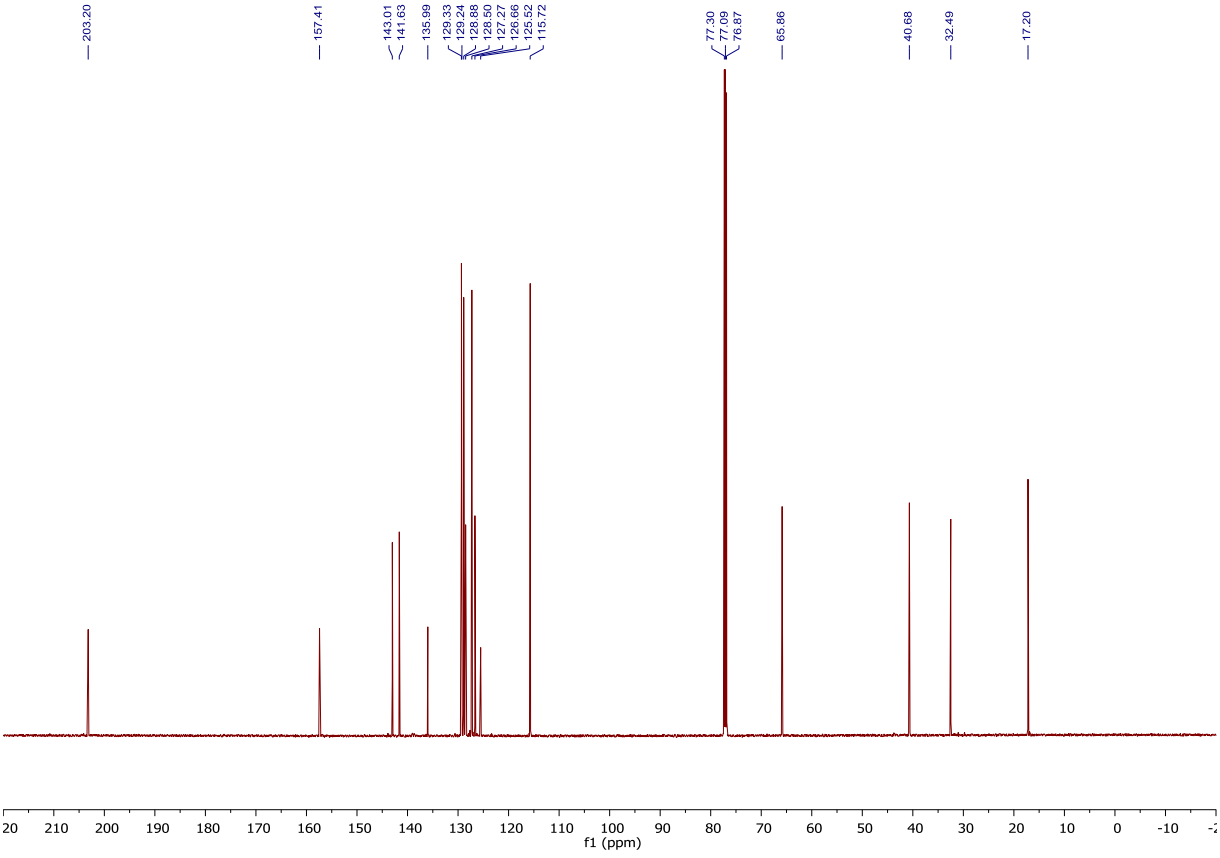
f1 (ppm)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126.66
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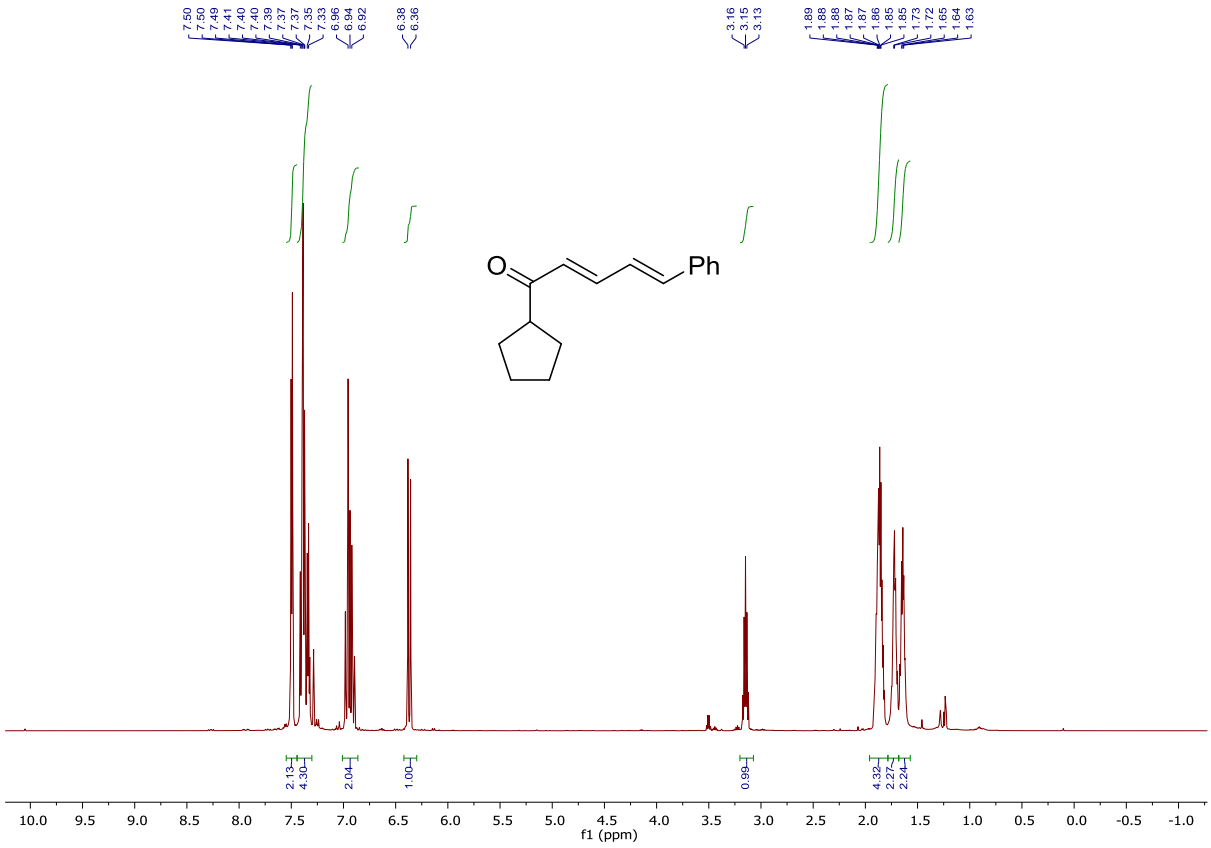
77.09
76.87
65.86
40.68
32.49
17.20
f1 (ppm)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7.40
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7.37
7.35
7.33
6.96
6.94
6.92
6.38
6.36
3.16
3.15
3.13
1.89
1.88
1.88
1.88
1.87
1.86
1.85
1.85
1.73
1.72
1.65
1.64
1.63
O
Ph
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4.30
2.04
1.00
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4.32
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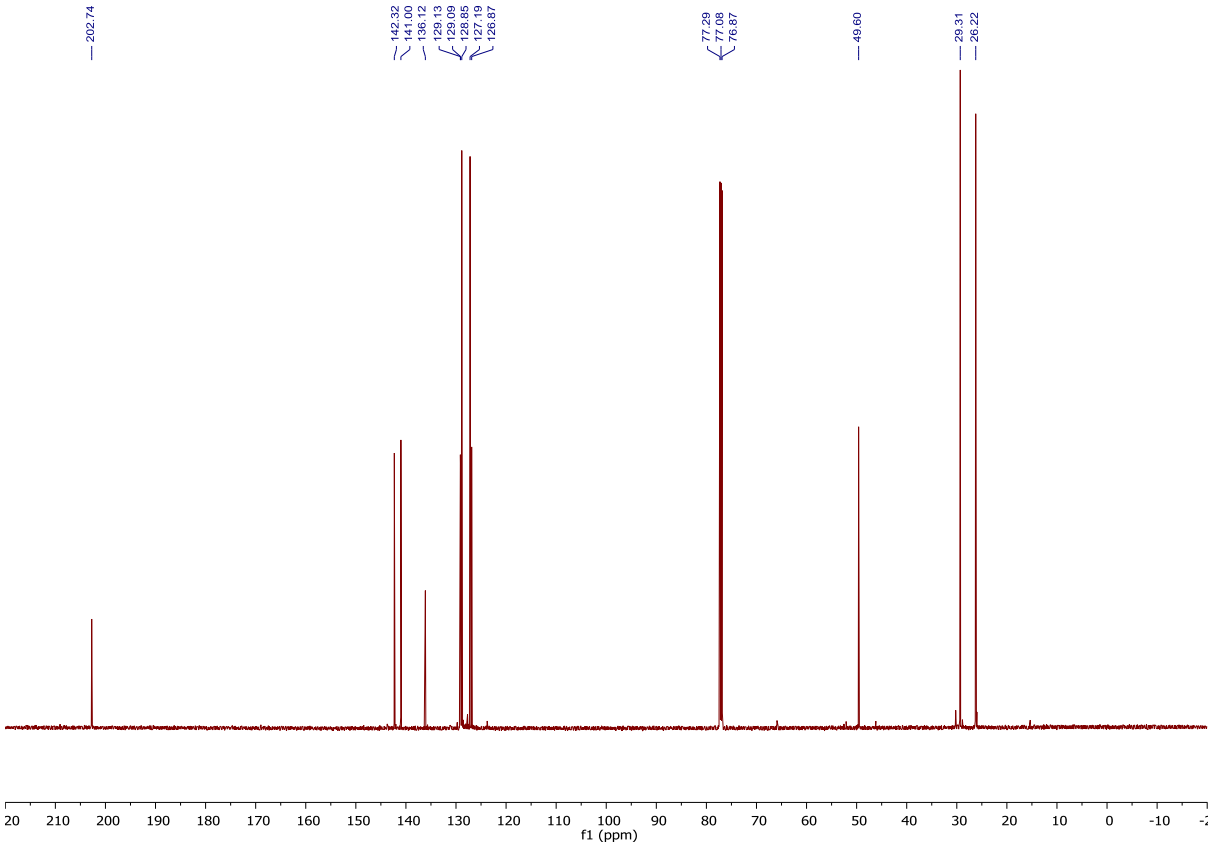
2.24
f1 (ppm)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49.60
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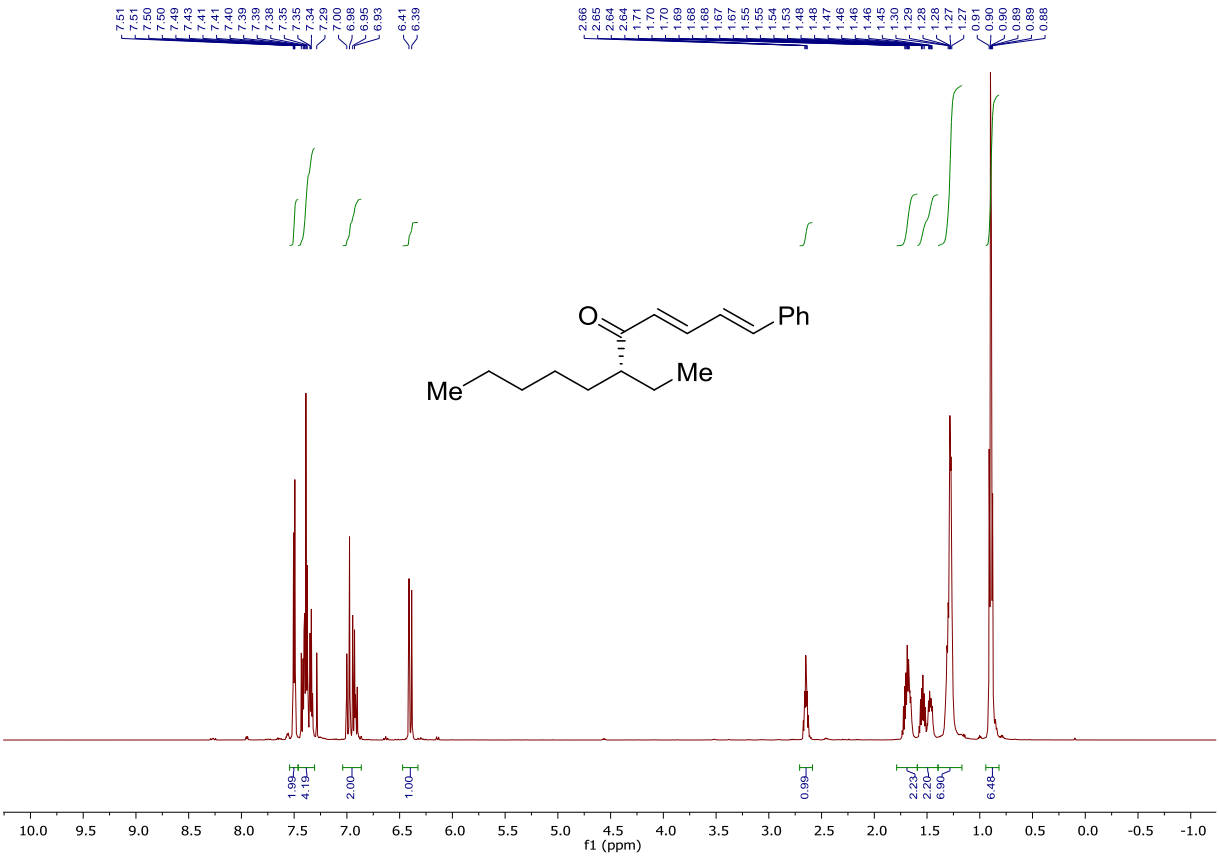
26.22
f1 (ppm)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1.45
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1.28
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1.27
1.27
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0.90
0.90
0.89
0.89
0.88
O
Ph
Me
Me
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2.00
1.00
0.99
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4.0
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1.0
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0.0
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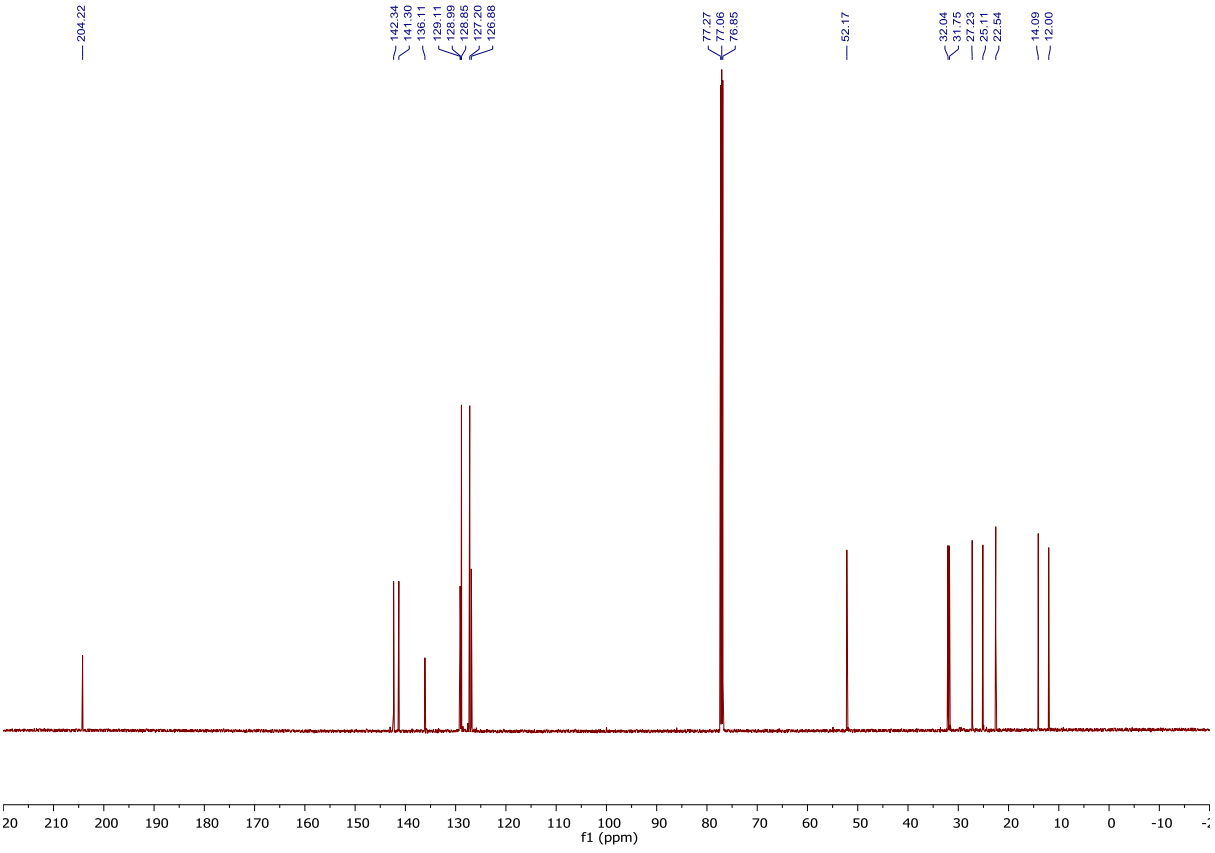
-1.0
f1 (ppm)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31.75
27.23
25.11
22.54
14.09
12.00
20
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200
190
180
170
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140
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120
110
100
90
80
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60
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40
30
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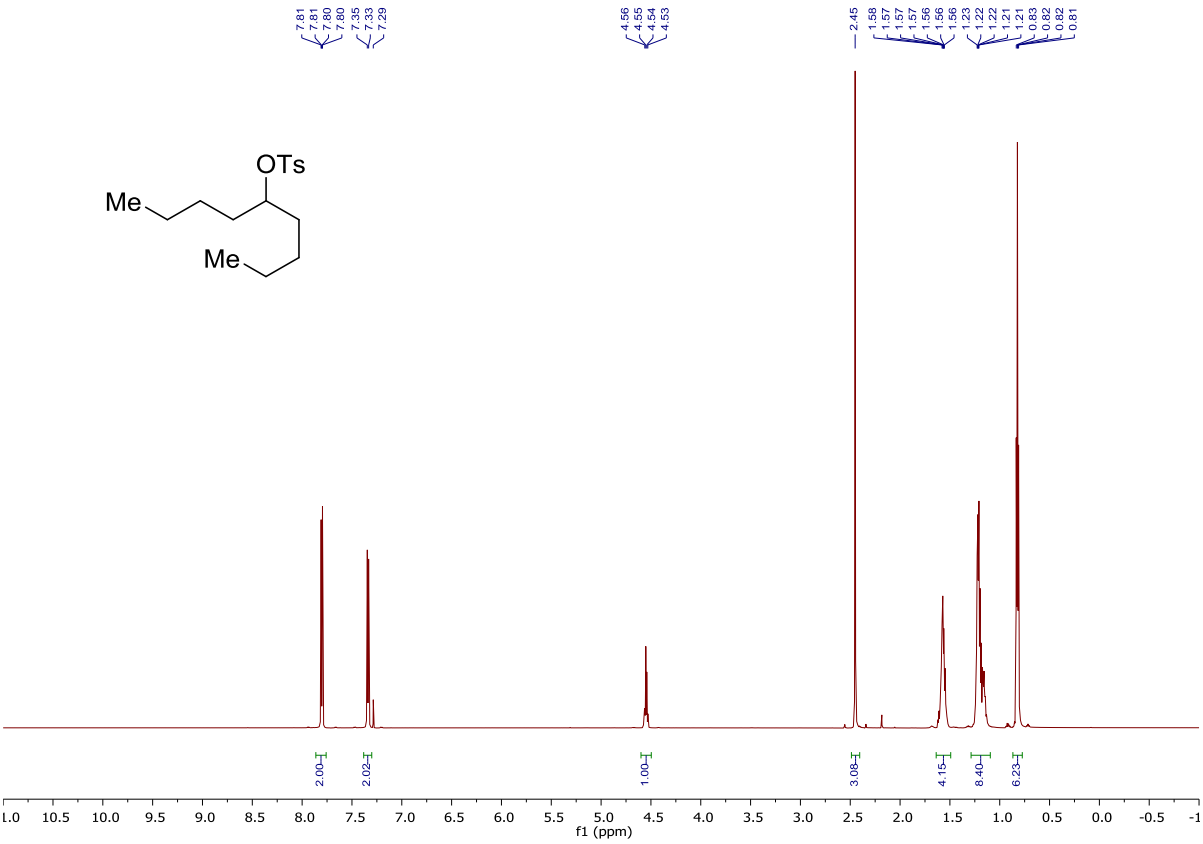
0
-10
f1 (ppm)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1.22
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0.83
0.82
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Me
Me
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1.00
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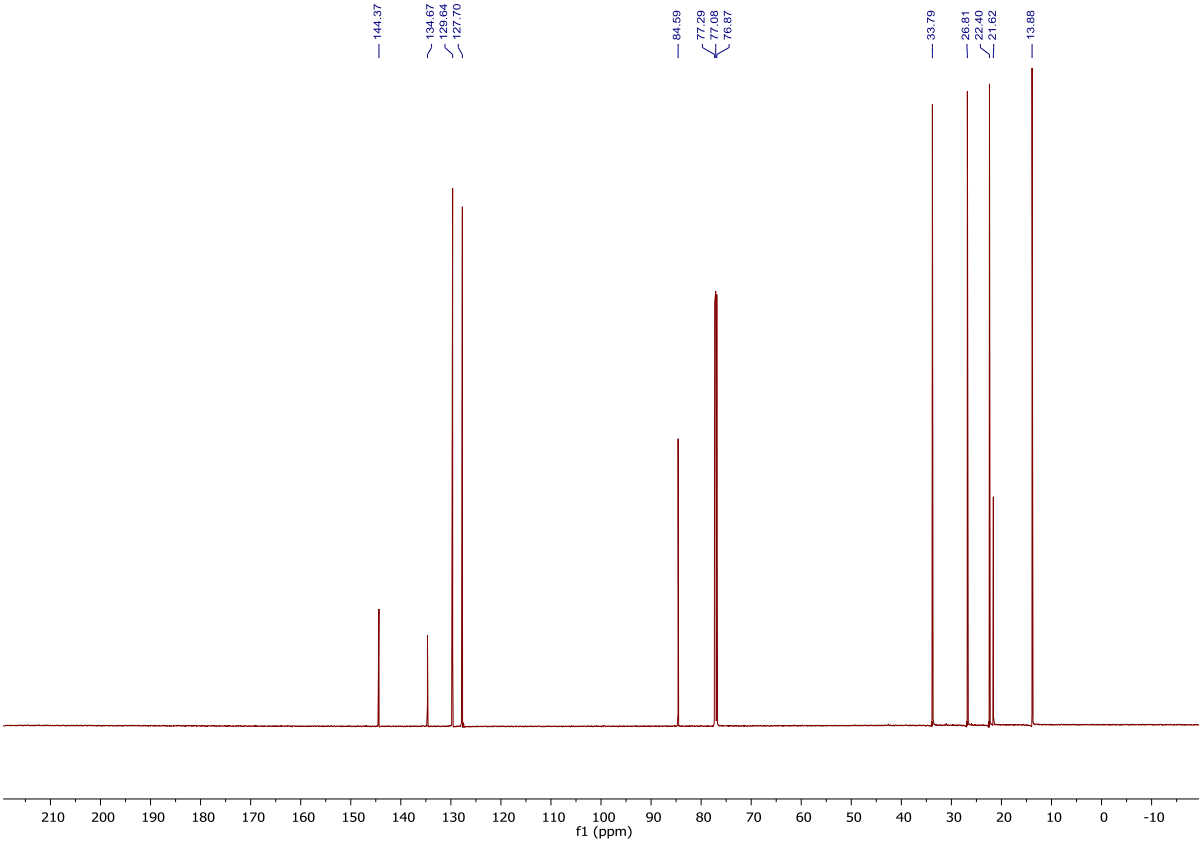
6.23
f1 (ppm)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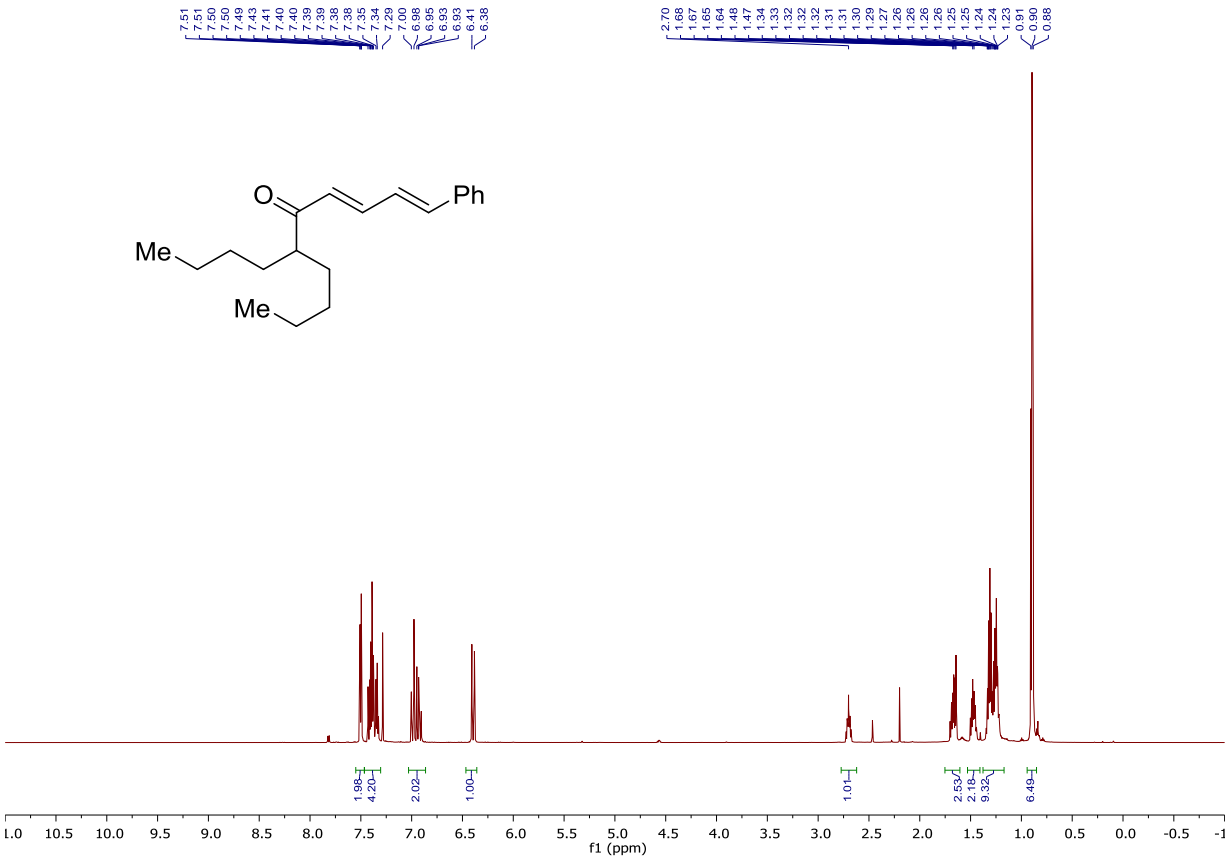
13.88
f1 (ppm)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1.23
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0.90
0.88
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Me
Me
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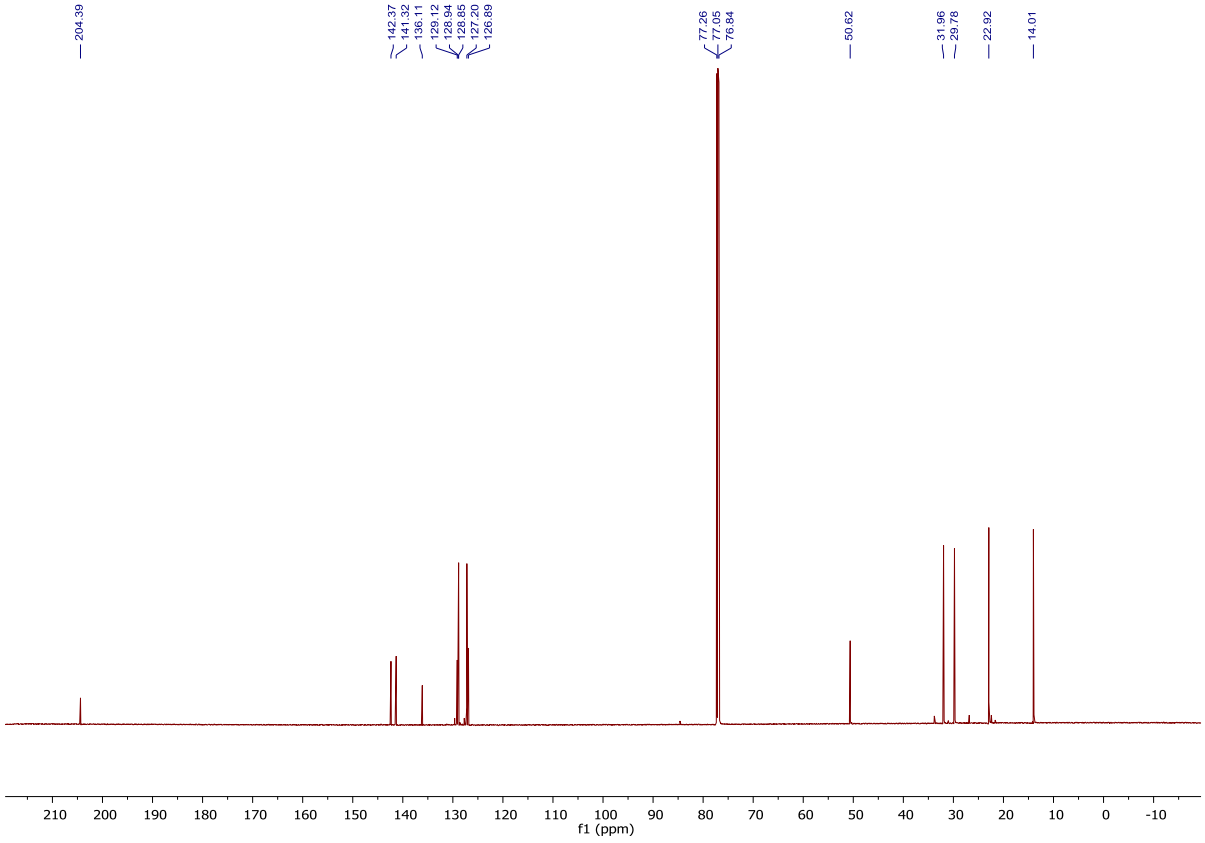
1.01
2.53
2.18
9.32
6.49
f1 (ppm)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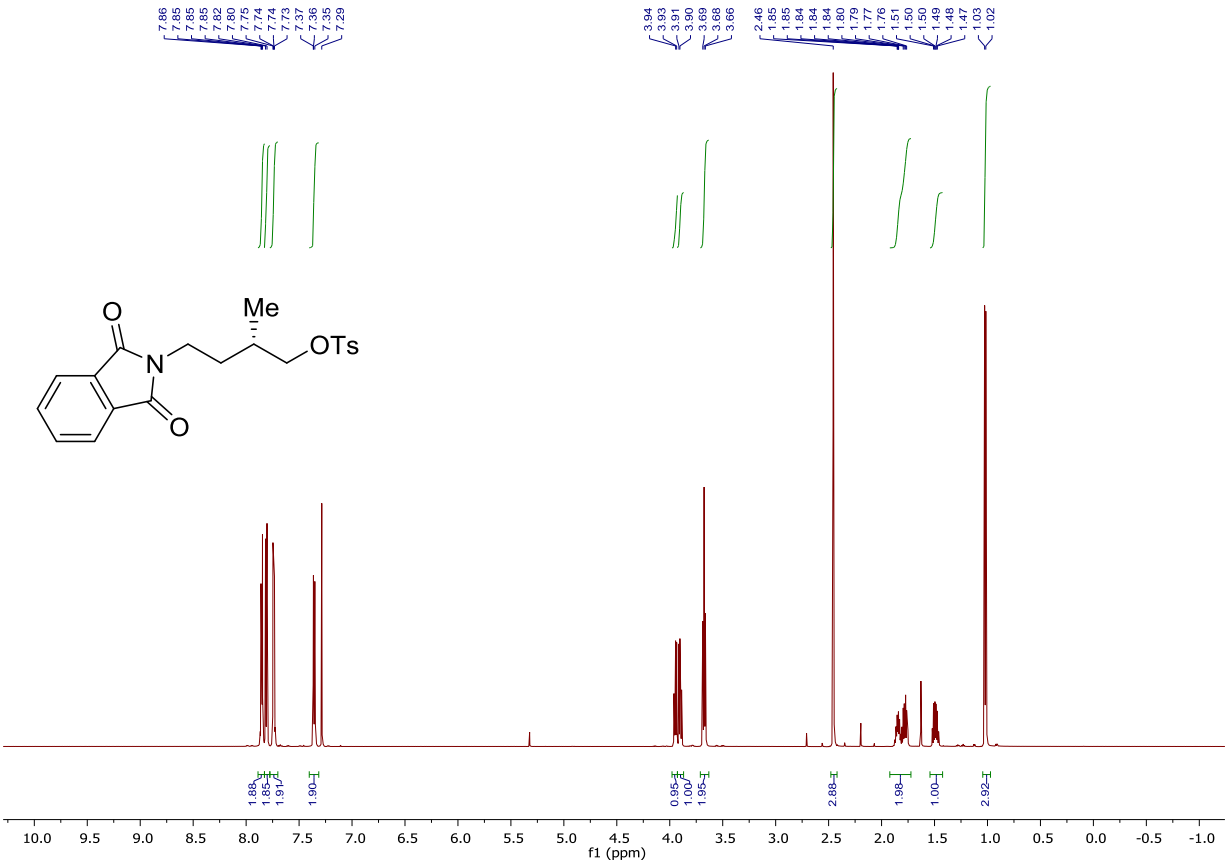
77.05
76.84
50.62
31.96
29.78
22.92
14.01
f1 (ppm)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7.29
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3.90
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3.68
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1.49
1.48
1.47
1.03
1.02
O
N
O
Me
OTs
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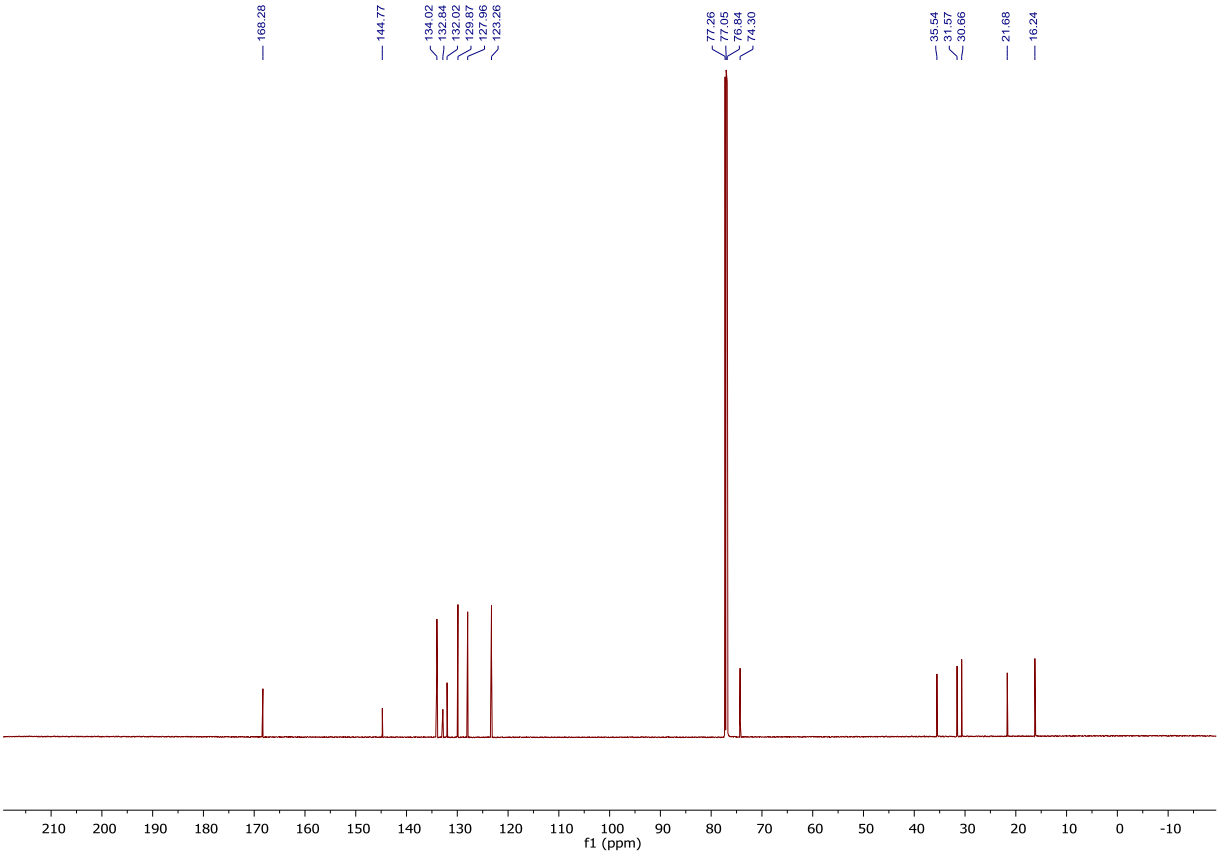
1.85
1.91
1.90
0.95
1.00
1.95
2.88
1.98
1.00
2.92
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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127.96
123.26
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77.05
76.84
74.30
35.54
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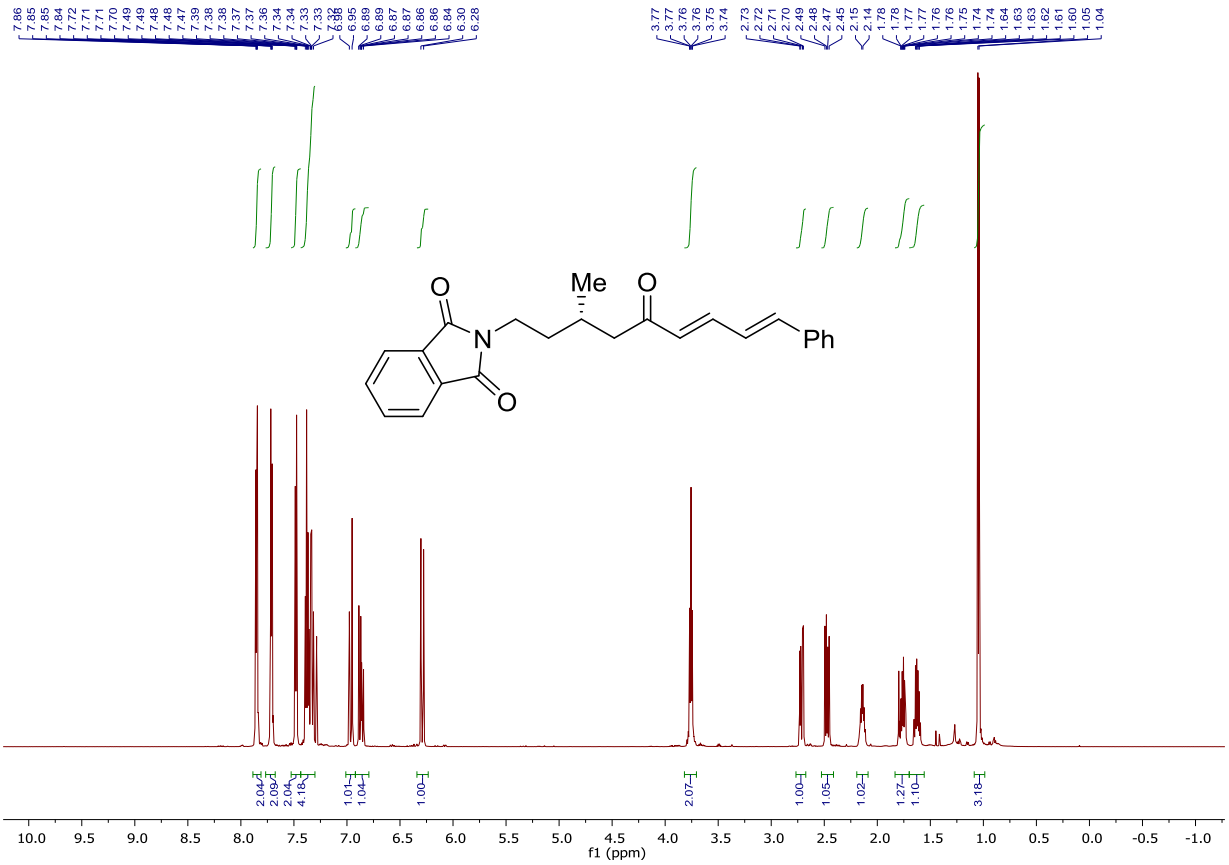
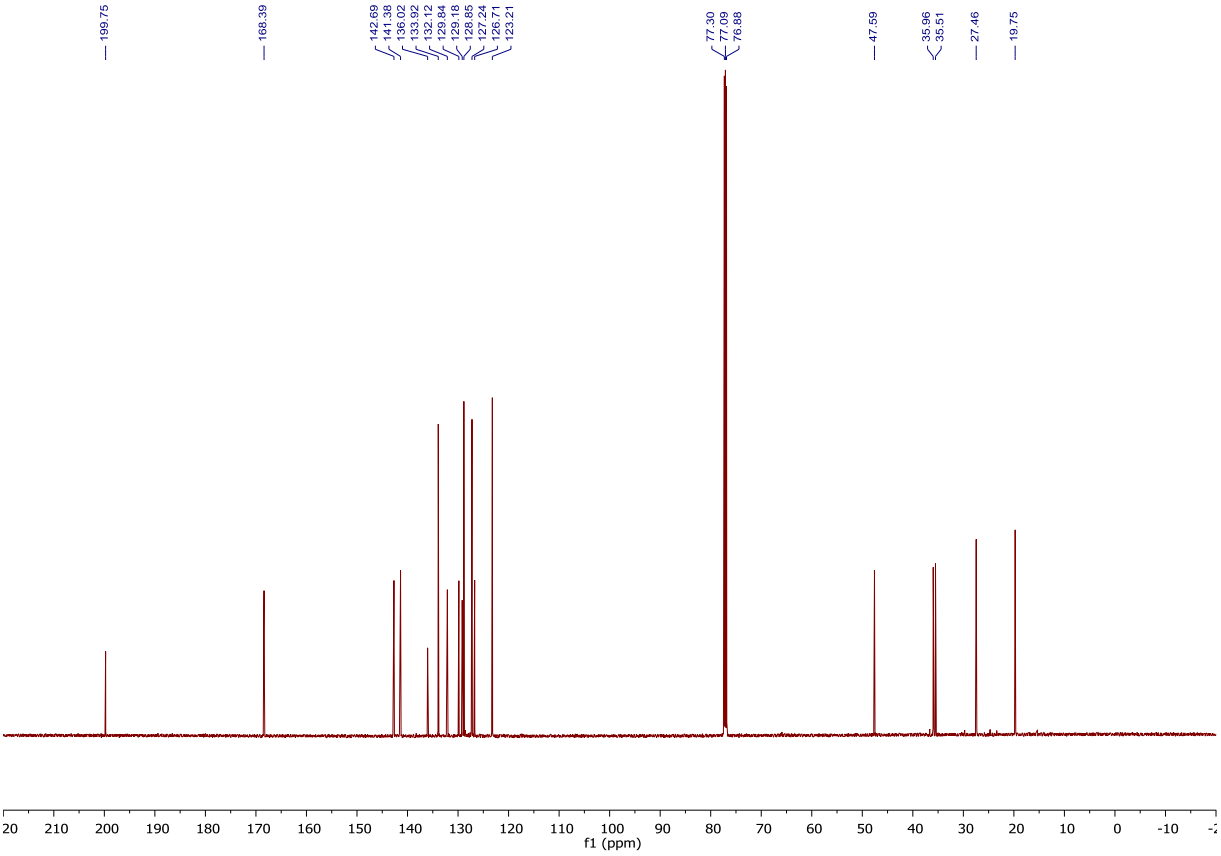
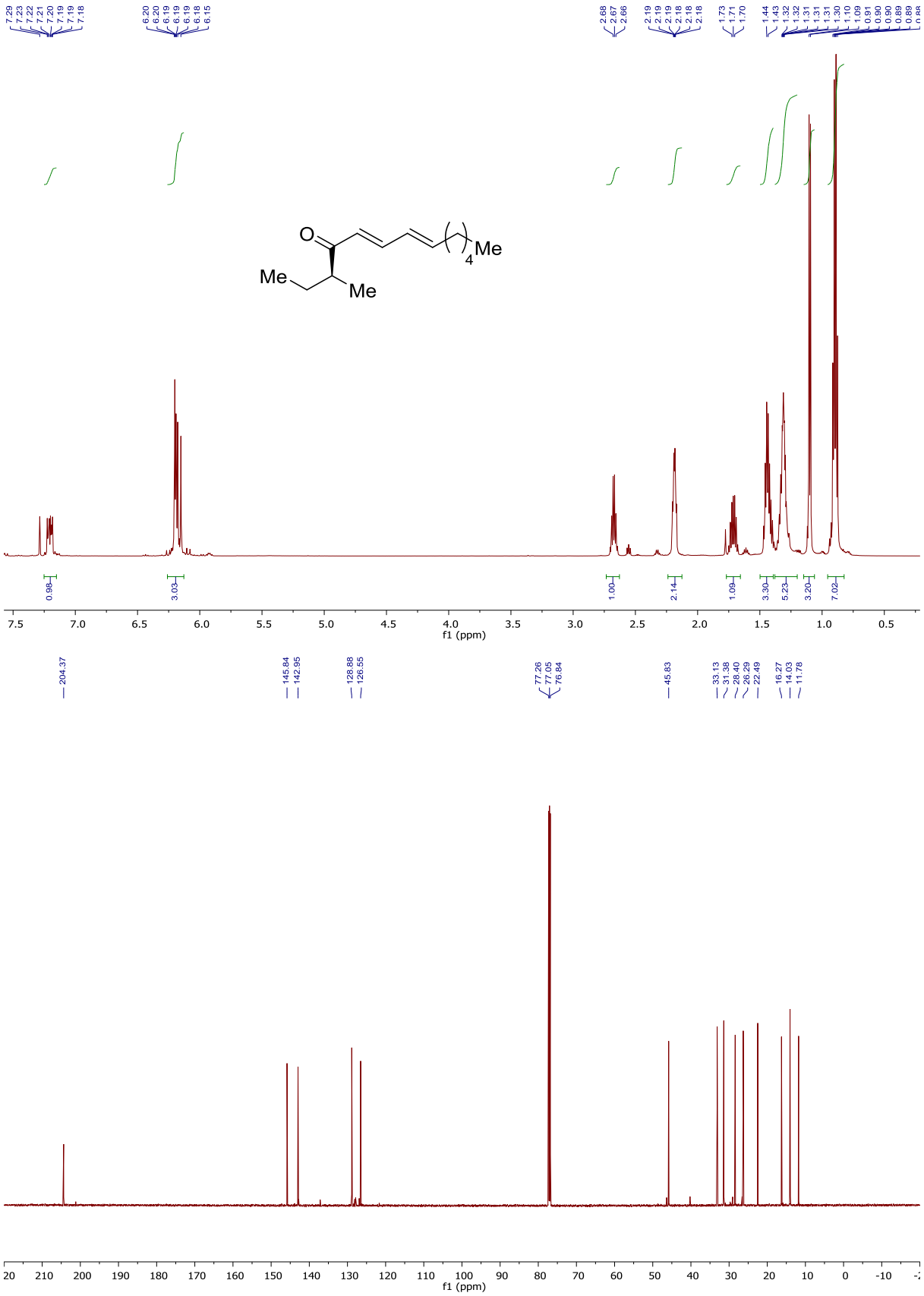
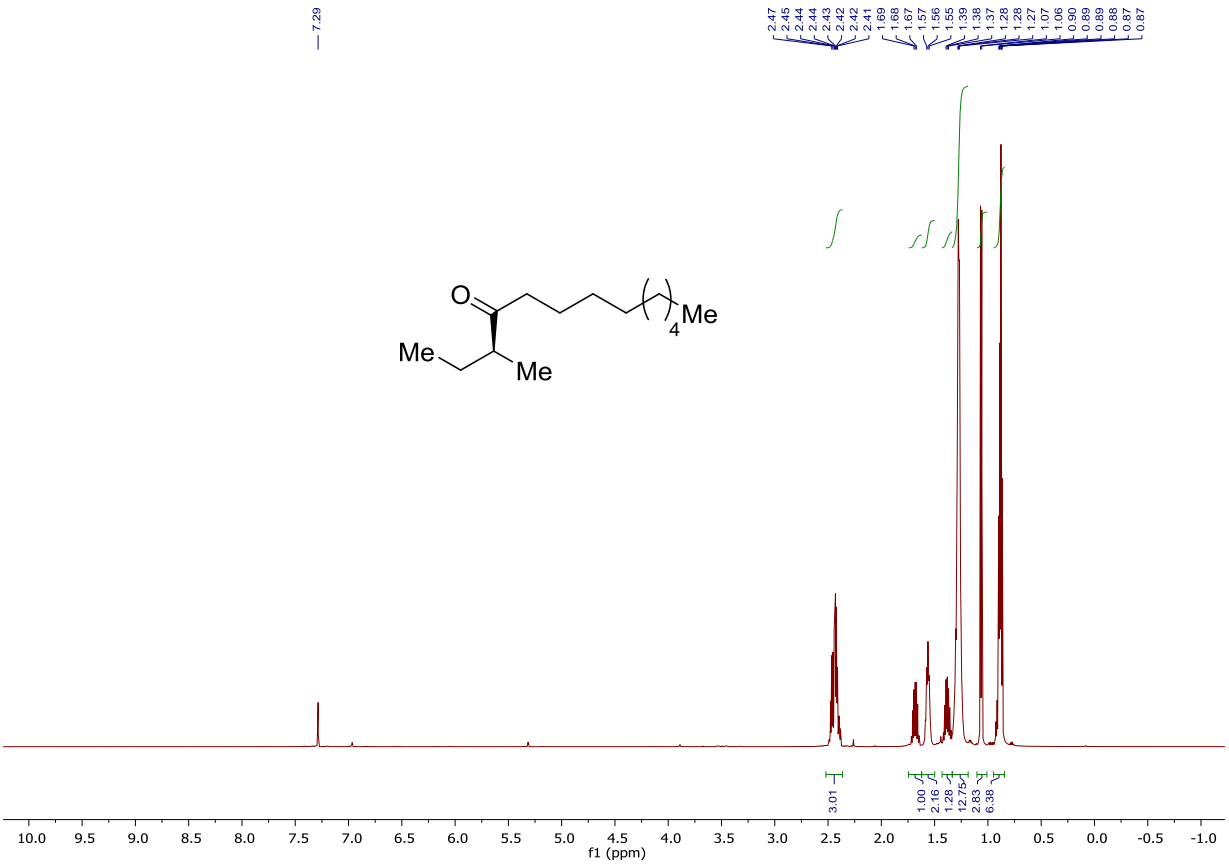
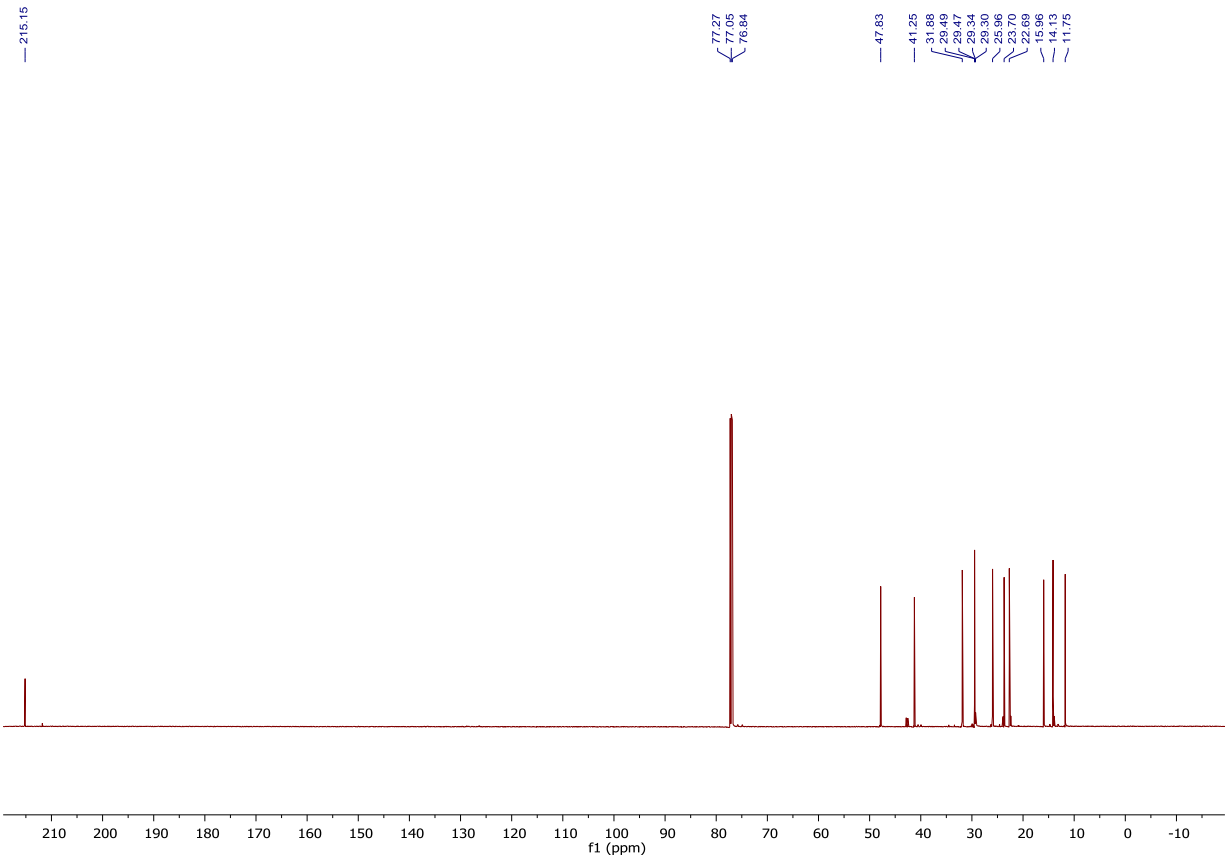
30.66
21.68
16.24
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.86 7.85 7.85 7.84 7.72 7.71 7.70 7.49 7.49 7.48 7.48 7.47 7.39 7.38 7.38 7.37 7.37 7.36 7.34 7.34 7.33 7.33 6.95 6.89 6.89 6.87 6.87 6.86 6.86 6.84 6.30 6.28
3.77 3.77 3.76 3.76 3.75 3.74 2.73 2.72 2.71 2.70 2.49 2.48 2.47 2.45 2.15 2.14 1.78 1.78 1.77 1.77 1.76 1.76 1.75 1.74 1.74 1.64 1.63 1.63 1.62 1.61 1.60 1.05 1.04
O
N
O
Me
O
Ph
2.04 2.09 2.04 4.18 1.01 1.04 1.00 2.07 1.00 1.05 1.02 1.27 1.10 3.18
10.0 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 -0.5 -1.0
f1 (ppm)


199.75 168.39 142.69 141.38 136.02 133.92 132.12 129.84 129.18 128.85 127.24 126.71 123.21 77.30 77.09 76.88 47.59 35.96 35.51 27.46 19.75
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


O
Me
Me
4
Me
f1 (ppm)
f1 (ppm)


7.29
2.47
2.45
2.44
2.44
2.43
2.42
2.42
2.41
1.69
1.68
1.67
1.57
1.56
1.55
1.39
1.38
1.37
1.28
1.28
1.27
1.07
1.06
0.90
0.89
0.88
0.88
0.87
0.87
O
Me
4
Me
Me
3.01
1.00
2.16
1.28
12.75
2.83
6.38
10.0
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
-0.5
-1.0
f1 (ppm)


215.15
77.27
77.05
76.84
47.83
41.25
31.88
29.49
29.47
29.34
29.30
25.96
23.70
22.69
15.96
14.13
11.75
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)