Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Iodine/water-mediated deprotective oxidation of allylic ethers to access α,β-unsaturated ketones and aldehydes

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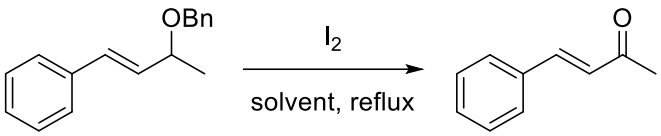
1. General information

Oxygen- and moisture-sensitive reactions were carried out under an argon atmosphere. Solvents were purified and dried by standard methods prior to use. All commercially available reagents were used without further purification, unless otherwise noted. Column chromatography was carried out on silica gel (200-300 mesh). Analytical thin layer chromatography (TLC) was performed using pre-coated silica gel of 60 F254 (0.25 mm) plates and was visualized by UV (254 nm) and/or permanganate stain.

High resolution mass spectra (HRMS) were recorded on a Bruker microQTOF-QII. NMR spectra were recorded with TMS as an internal standard on a Bruker AV-400 MHZ instrument (400 MHz for ^{1}H NMR, 101 MHz for ^{13}C NMR). Chemical shifts were reported in ppm on the δ scale relative to $CDCl_3$ (δ = 7.26 for ^{1}H NMR, δ = 77.2 for ^{13}C NMR). Multiplicities are indicated as: s (singlet), d (doublet), t (triplet), dd (doublet of doublet), or m (multiplet). Coupling constants (J) are reported in Hertz (Hz). GC-MS analyses were performed with Agilent 5977A/7890B GC-MS system. Most of allyl benzyl ethers[1,2], allyl silyl ethers[3], allyl esters[4] were known and prepared according to the previous reports.

2. Optimization studies

Table S1 Screening of solvents[a]

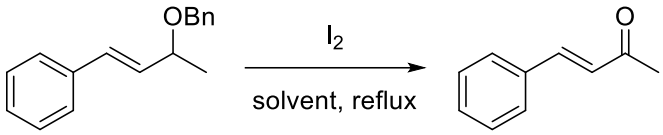


1a **2a**

Entry	Temperature (℃)	Solvent (ratio)	Yield (%)[b]
1	115	1,4-Dioxane/H_2O (5:1)	78
2	115	1,4-Dioxane	0
3	105	H_2O	35
4	90	Toluene/H_2O (5:1)	18
5	70	THF/H_2O (5:1)	40
6	82	MeOH/H_2O (5:1)	15
7	150	DMF/H_2O (5:1)	26
8	150	DMSO/H_2O (5:1)	51
9	60	DCM/H_2O (5:1)	12

[a] General conditions: **1a** (0.1 mmol), I_2 (0.16 mmol, 1.6 eq.), solvent (3.6 mL, solvent ratios are by volume), at refluxing temperature (60-150 ℃), 2 h, under air. [b] Isolated yields.

Table S2 Screening of other conditions[a]

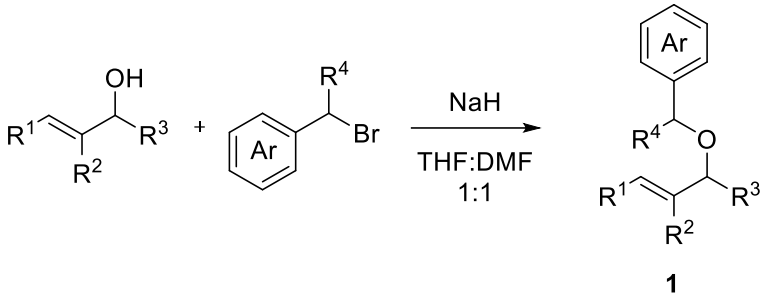


1a **2a**

Entry	I_2	Temperature (℃)	Solvent (ratio)	Time (h)	Yield (%)[b]
1	1.6eq	25	1,4-Dioxane/H_2O (5:1)	2	0
2[c]	1.6eq	115	1,4-Dioxane/H_2O (5:1)	2	63
3	1.6eq	115	1,4-Dioxane/H_2O (5:1)	24	91
4	0.5eq	115	1,4-Dioxane/H_2O (5:1)	24	72
5	2.0eq	115	1,4-Dioxane/H_2O (5:1)	24	79
6	0eq	115	1,4-Dioxane/H_2O (5:1)	24	0
7	1.6eq	115	1,4-Dioxane/H_2O (1:1)	24	63
8	1.6eq	115	1,4-Dioxane/H_2O (2:1)	24	72
9	1.6eq	115	1,4-Dioxane/H_2O (10:1)	24	90

[a] General conditions: **1a** (0.1 mmol), I_2 (0-2.0 eq.), solvent (3.6 mL, solvent ratios are by volume), at 25 or 115℃, 2 or 24 h, under air. [b] Isolated yields. [c] Under Ar.

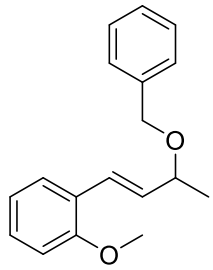
3. General procedure for the synthesis of new substrates 1



1

To a solution of the corresponding allyl alcohol (3.0 mmol, 1.0 eq.) in anhydrous THF (10 mL) and DMF (10 mL) was added NaH (6.0 mmol, 2.0 eq.) and benzyl bromide (6.0 mmol, 2.0 eq.) at 0 ℃ under Ar. The reaction mixture was allowed to stir at room temperature for 5 h and then quenched with H_2O (10 mL). The aqueous layer was extracted with EtOAc (5 mL × 3) and the organic layer was washed with H_2O and brine, dried over anhydrous $MgSO_4$ and evaporated in vacuo. The crude residue was purified by column chromatography (EtOAc/hexane = 1/30) to afford **1** as colorless or yellow oil.

(*E*)-1-(3-(benzyloxy)but-1-en-1-yl)-2-methoxybenzene (1h)



Chemical Formula: $C_{18}H_{20}O_2$
Exact Mass: 268.1463

General procedure was followed on 534.7 mg (*E*)-4-(2-methoxyphenyl)but-3-en-2-ol to afford 732.0 mg colorless oil, yield 91%.

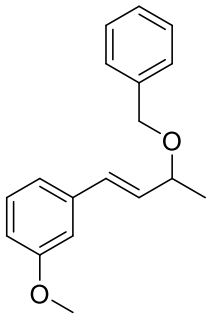
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.30 – 7.24 (m, 4H), 7.20 – 7.13 (m, 2H), 6.87 – 6.81 (m, 2H), 6.79-7.78 (m, 1H), 6.09 (dd, *J* = 16.1, 8.0 Hz, 1H), 4.54 (d, *J* = 12.0 Hz, 1H), 4.36 (d, *J* = 12.0 Hz, 1H), 4.07 – 4.00 (m, 1H), 3.77 (s, 3H), 1.31 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 156.7, 138.9, 132.1, 128.8, 128.4, 127.8, 127.4, 126.9, 126.4, 125.6, 120.7, 110.9, 76.5, 69.9, 55.5, 21.9.

HRMS (ESI) calcd. for $C_{18}H_{20}O_2Na$ [M+Na^+]: 291.1356, found: 291.1369.

(*E*)-1-(3-(benzyloxy)but-1-en-1-yl)-3-methoxybenzene (1i)

General procedure was followed on 534.7 mg (*E*)-4-(3-methoxyphenyl)but-3-en-2-ol to afford 732.0 mg colorless oil, yield 91%.



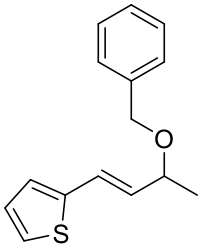
Chemical Formula: $C_{18}H_{20}O_2$
Exact Mass: 268.1463

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.24 (m, 4H), 7.21 – 7.18 (m, 1H), 7.16 – 7.14 (m, 1H), 6.92 (d, *J* = 7.6 Hz, 1H), 6.87 – 6.85 (m, 1H), 6.74 – 6.72 (m, 1H), 6.43 (d, *J* = 15.9 Hz, 1H), 6.08 (dd, *J* = 15.9, 7.7 Hz, 1H), 4.53 (d, *J* = 12.0 Hz, 1H), 4.36 (d, *J* = 12.0 Hz, 1H), 4.04–4.00 (m, 1H), 3.74 (s, 3H), 1.30 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.9, 138.8, 138.1, 132.0, 131.4, 129.6, 128.4, 127.7, 127.5, 119.2, 113.5, 111.7, 75.8, 70.1, 55.3, 21.8.

HRMS (ESI) calcd. for $C_{18}H_{20}O_2Na$ [M+Na $^+$]: 291.1356, found: 291.1369.

(*E*)-2-(3-(benzyloxy)but-1-en-1-yl)thiophene (1k)



Chemical Formula: $C_{15}H_{16}OS$
Exact Mass: 244.0922

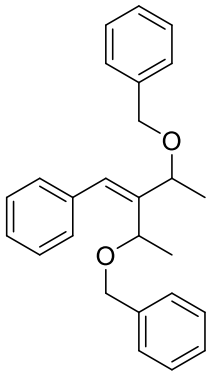
General procedure was followed on 462.6 mg (*E*)-4-(thiophen-2-yl)but-3-en-2-ol to afford 527.2 mg colorless oil, yield 72%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.27 (m, 5H), 7.17 – 7.16 (m, 1H), 6.97 (d, *J* = 3.4 Hz, 2H), 6.67 (d, *J* = 15.8 Hz, 1H), 6.00 (dd, *J* = 15.8, 7.6 Hz, 1H), 4.62 (d, *J* = 12.0 Hz, 1H), 4.43 (d, *J* = 12.0 Hz, 1H), 4.07 – 4.04 (m, 1H), 1.36 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.8, 138.7, 131.3, 128.4, 127.7, 127.5, 127.4, 125.8, 124.5, 124.4, 75.5, 70.1, 21.7.

HRMS (ESI) calcd. for $C_{15}H_{16}OSNa$ [M+Na $^+$]: 267.0814, found: 267.0806.

(((3-benzylidenepentane-2,4-diyl)bis(oxy))bis(methylene))dibenzene (1l)



Chemical Formula: $C_{26}H_{28}O_2$
Exact Mass: 372.2089

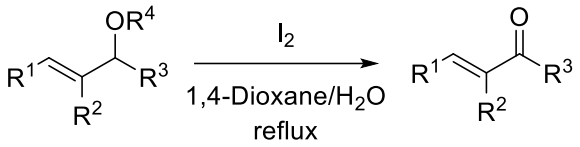
General procedure was followed on 576.7 mg 3-benzylidenepentane-2,4-diol to afford 993.7 mg colorless oil, yield 89%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.22 (m, 6H), 7.20 – 7.16 (m, 2H), 7.14 – 7.11 (m, 5H), 7.04 – 7.02 (m, 1H), 6.99 - 6.97 (m, 1H), 6.95 (d, *J* = 4.7 Hz, 1H), 4.58 – 4.48 (m, 2H), 4.39 – 4.28 (m, 3H), 4.06 (dd, *J* = 11.8, 9.2 Hz, 1H), 1.40 (dd, *J* = 17.3, 6.3 Hz, 3H), 1.31 (dd, *J* = 19.8, 6.7 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.6, 144.4, 139.0, 138.8, 138.5, 138.4, 137.0, 136.9, 129.1, 128.8, 128.4, 128.4, 128.3, 128.2, 128.2, 128.1, 127.7, 127.7, 127.6, 127.5, 127.4, 127.4, 127.3, 126.8, 126.8, 73.3, 72.7, 71.6, 71.1, 70.4, 70.3, 70.2, 70.1, 24.4, 23.6, 20.6, 20.1.

HRMS (ESI) calcd. for $C_{26}H_{28}O_2Na$ [M+Na $^+$]: 395.1982, found: 395.1995.

4. General procedure for the oxidation of 1

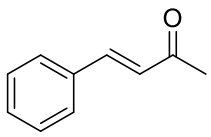


1 **2**

To a solution of **1** (0.1 mmol, 1.0 eq.) in 1,4-dioxane (3.0 mL) and water (0.6 mL) was added I_2 (40.6 mg, 0.16 mmol, 1.6 eq.). The mixture was refluxed for 24 h, then cooled to room temperature. The reaction was quenched with aq. $Na_2S_2O_3$ and extracted with EtOAc (5 mL × 3). The combined organic layer was washed with H_2O and brine, dried over anhydrous $MgSO_4$ and concentrated in vacuo. The crude residue was purified by column chromatography (EtOAc/hexane = 1:50 – 1:20) to afford **2**.

(*E*)-4-phenylbut-3-en-2-one (2a)

General procedures was followed on **1t** (26.2 mg) to afford **2a** as colorless oil (13.9 mg, yield 95%).



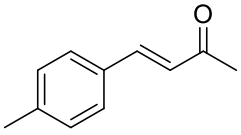
Chemical Formula: $C_{10}H_{10}O$
Exact Mass: 146.0732

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.42 (m, 3H), 7.33 – 7.31 (m, 3H), 6.64 (d, *J* = 16.3 Hz, 1H), 2.31 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 198.5, 143.5, 134.2, 130.5, 129.0, 128.2, 127.1, 27.5.

(*E*)-4-(p-tolyl)but-3-en-2-one (2f)

General procedures was followed on **1f** (25.2 mg) to afford **2f** as colorless oil (14.7 mg, yield 92%).



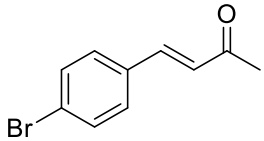
Chemical Formula: $C_{11}H_{12}O$
Exact Mass: 160.2160

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (m, 3H), 7.12 (d, *J* = 7.9 Hz, 2H), 6.60 (d, *J* = 16.3 Hz, 1H), 2.29 (d, *J* = 1.7 Hz, 6H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6, 143.6, 141.0, 131.6, 129.7, 128.2, 126.2, 27.4, 21.5.

(*E*)-4-(4-bromophenyl)but-3-en-2-one (2g)

General procedures was followed on **1g** (31.7 mg) to afford **2g** as colorless oil (20.5 mg, yield 91%).



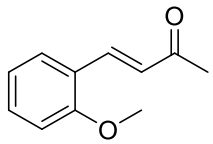
Chemical Formula: $C_{10}H_9BrO$
Exact Mass: 223.9837

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.4 Hz, 2H), 7.38 – 7.31 (m, 3H), 6.62 (d, *J* = 16.3 Hz, 1H), 2.30 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 198.1, 141.9, 133.3, 132.2, 129.6, 127.5, 124.8, 27.7.

(*E*)-4-(2-methoxyphenyl)but-3-en-2-one (2h)

General procedures was followed on **1h** (26.8 mg) to afford **2h** as colorless oil (15.5 mg, yield 88%).



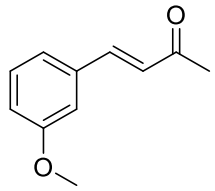
Chemical Formula: $C_{11}H_{12}O_2$
Exact Mass: 176.0837

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 16.5 Hz, 1 H), 7.53 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.38 – 7.34 (m, 1H), 6.98 – 6.90 (m, 2H), 6.74 (d, *J* = 16.5 Hz, 1H), 3.88 (s, 3H), 2.38 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.2, 158.3, 138.8, 131. 8, 128.3, 127.7, 123.3, 120.8, 111.2, 55.5, 27.2.

(*E*)-4-(3-methoxyphenyl)but-3-en-2-one (2i)

General procedures was followed on **1i** (26.8 mg) to afford **2i** as colorless oil (15.6 mg, yield 89%).



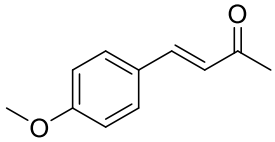
Chemical Formula: $C_{11}H_{12}O_2$
Exact Mass: 176.0837

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 16.3 Hz, 1H), 7.30 (dd, *J* = 14.7, 6.8 Hz, 1H), 7.13 (d, *J* = 7.7 Hz, 1H), 7.06 – 7.05 (m, 1H), 6.96 – 6.93 (m, 1H), 6.69 (d, *J* = 16.3 Hz, 1H), 3.82 (s, 3H), 2.37 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 198.5, 159.9, 143.4, 135.8, 130.0, 127.4, 121.0, 116.4, 113.0, 55.3, 27.5.

(*E*)-4-(4-methoxyphenyl)but-3-en-2-one (2j)

General procedures was followed on **1j** (26.8 mg) to afford **2j** as colorless oil (15.8 mg, yield 90%).



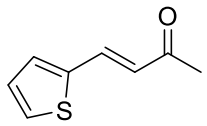
Chemical Formula: $C_{11}H_{12}O_2$
Exact Mass: 176.0837

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.45 (m, 3H), 6.91 (d, *J* = 8.7 Hz, 2H), 6.61 (d, *J* = 16.2 Hz, 1H), 3.84 (s, 3H), 2.36 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 203.4, 198.3, 163.6, 161.6, 143.2, 129.9, 127.0, 125.0, 114.4, 55.3, 27.4.

(*E*)-4-(thiophen-2-yl)but-3-en-2-one (2k)

General procedures was followed on **1k** (24.4 mg) to afford **2k** as colorless oil (7.7 mg, yield 51%).



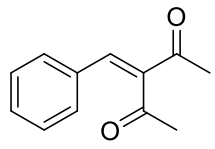
Chemical Formula: C_8H_8OS
Exact Mass: 152.0296

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 15.9 Hz, 1H), 7.40 (d, *J* = 5.1 Hz, 1H), 7.29 (d, *J* = 3.6 Hz, 1H), 7.06 (dd, *J* = 5.1, 3.7 Hz, 1H), 6.52 (d, *J* = 15.9 Hz, 1H), 2.33 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 197.8, 139.7, 135.8, 131.6, 128.9, 128.3, 125.8, 27.6.

3-benzylidenepentane-2,4-dione (2l)

General procedures was followed on **1l** (37.2 mg) to afford **2l** as colorless oil (16.7 mg, yield 89%).



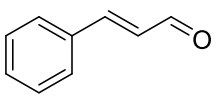
Chemical Formula: $C_{12}H_{12}O_2$
Exact Mass: 188.0837

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (s, 1H), 7.42 – 7.39 (m, 5H), 2.43 (s, 3H), 2.29 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 205.6, 196.5, 142.8, 139.8, 132.9, 130.7, 129.7, 129.1, 31.7, 26.5.

Cinnamaldehyde (2m)

General procedures was followed on **1m** (22.4 mg) to afford **2m** as colorless oil (12.1 mg, yield 92%).

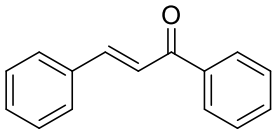


Chemical Formula: C_9H_8O
Exact Mass: 132.0575

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.70 (d, *J* = 7.7 Hz, 1H), 7.57 – 7.55 (m, 2H), 7.49 – 7.42 (m, 4H), 6.72 (dd, *J* = 15.9, 7.7 Hz, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 193.7, 152.8, 134.0, 131.3, 129.1, 128.6, 128.5.

(*E*)-chalcone (2p)

General procedures was followed on **1q** (30.0 mg) to afford **2p** as colorless oil (18.5 mg, yield 89%).



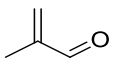
Chemical Formula: $C_{15}H_{12}O$
Exact Mass: 208.0888

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 – 7.94 (m, 2H), 7.74 (d, *J* = 15.7 Hz, 1H), 7.58 - 7.56 (m, 2H), 7.53 – 7.48 (m, 2H), 7.45 – 7.41 (m, 2H), 7.35 – 7.34 (m, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 190.6, 144.9, 138.3, 134.9, 132.8, 130.6, 129.0, 128.6, 128.5, 128.5, 122.1.

Methacrylaldehyde (2r)

General procedures was followed on **1r** (16.2 mg) to afford **2r** as yellow oil (6.37 mg, yield 91%).



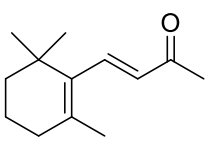
Chemical Formula: C_4H_6O
Exact Mass: 70.0419

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.56 (s, 1H), 6.31 – 6.30 (m, 1H), 6.00 (s, 1H), 1.85 – 1.84 (m, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 194.8, 145.9, 134.5, 13.9.

(*E*)-4-(2,6,6-trimethylcyclohex-1-en-1-yl)but-3-en-2-one (2s)

General procedures was followed on **1s** (28.4 mg) to afford **2s** as colorless oil (16.7 mg, yield 87%).



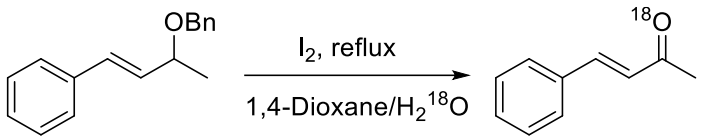
Chemical Formula: $C_{13}H_{20}O$
Exact Mass: 192.1514

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 17.6 Hz, 1H), 6.12 (d, *J* = 16.4 Hz, 1H), 2.30 (d, *J* = 1.2 Hz, 3H), 2.07 (t, *J* = 6.1 Hz, 2H), 1.76 (s, 3H), 1.64 – 1.60 (m, 2H), 1.48 (dd, *J* = 5.8, 3.1 Hz, 2H), 1.07 (d, *J* = 1.0 Hz, 6H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 198.7, 143.1, 136.0, 135.9, 131.6, 39.7, 34.0, 33.5, 28.8, 27.1, 21.7, 18.8.

5. Preliminary mechanistic studies

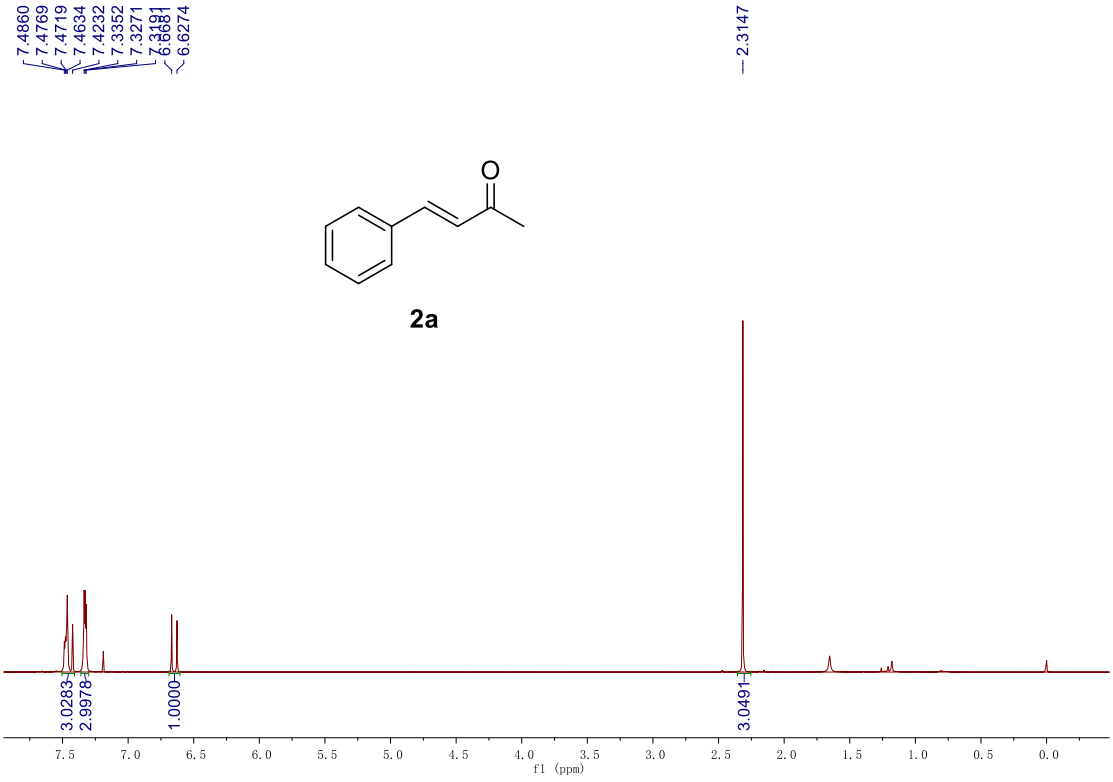
^{18}O-labeling experiment

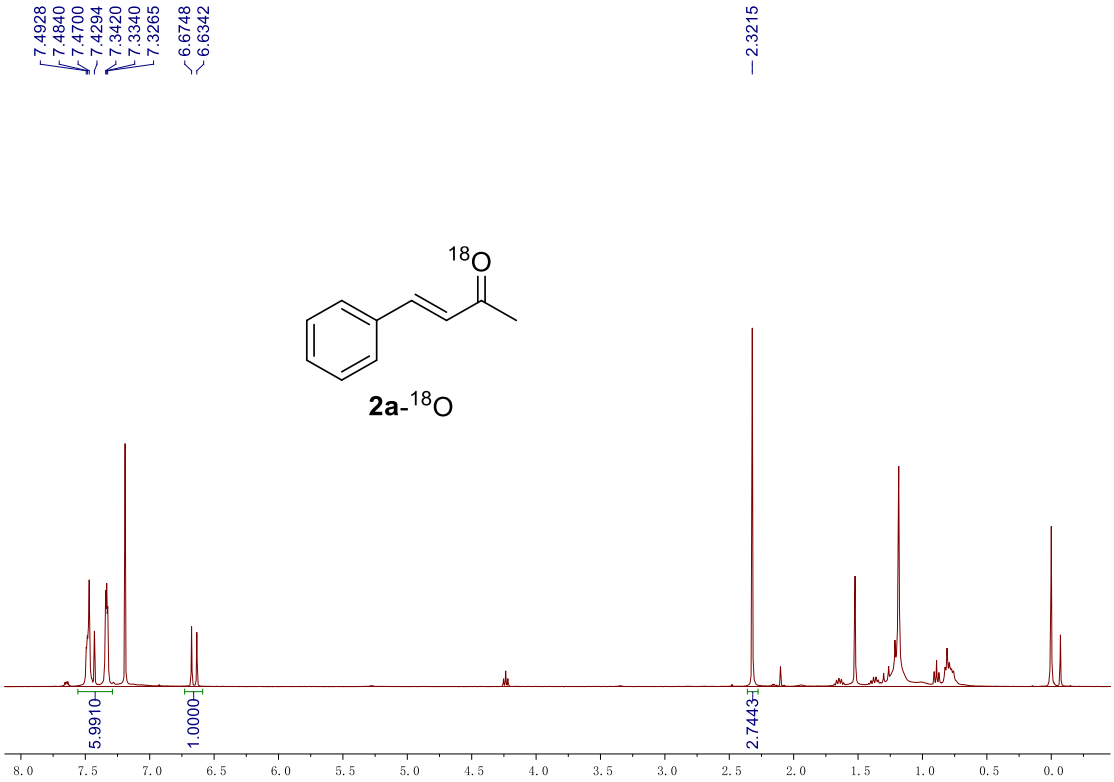


1a **2a-^{18}O**

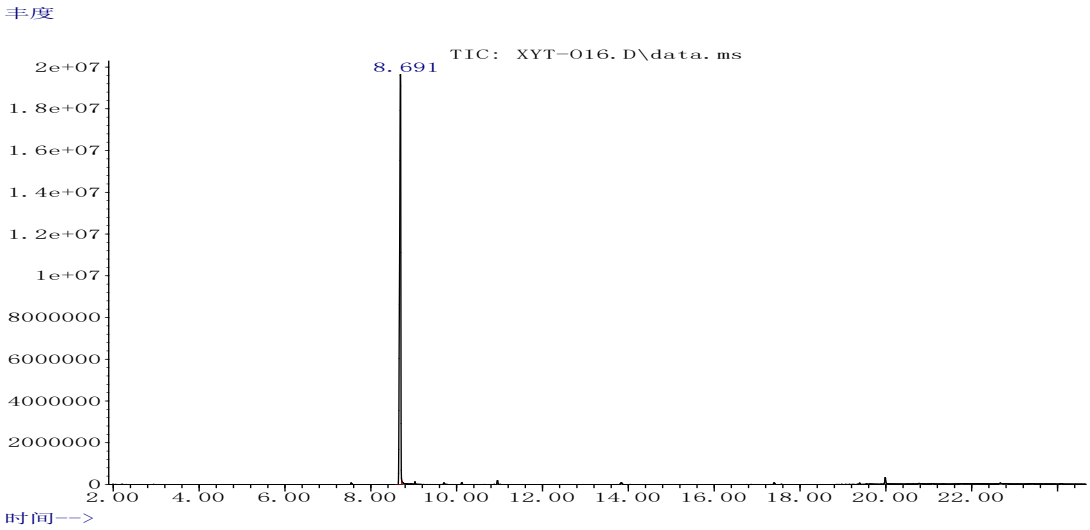
To a reaction tube was added I_2 (51.0 mg, 0.2 mmol, 1.6 eq.), **1a** (30.0 mg, 0.13 mmol, 1.0 eq.), 1,4-dioxane (3.0 mL) and $H_2{}^{18}O$ (0.6 mL). The mixture was refluxed for 24 h, then cooled to room temperature. The reaction was quenched with aq. $Na_2S_2O_3$ and extracted with EtOAc (5 mL × 3). The organic layer was washed with H_2O and brine, dried over anhydrous $MgSO_4$ and concentrated in vacuo. The crude residue was purified by column chromatography (EtOAc/hexane = 1:30) to afford **2a**-^{18}O, which was determined by NMR and GC-MS, as a colorless oil (17.4 mg, 92% yield).

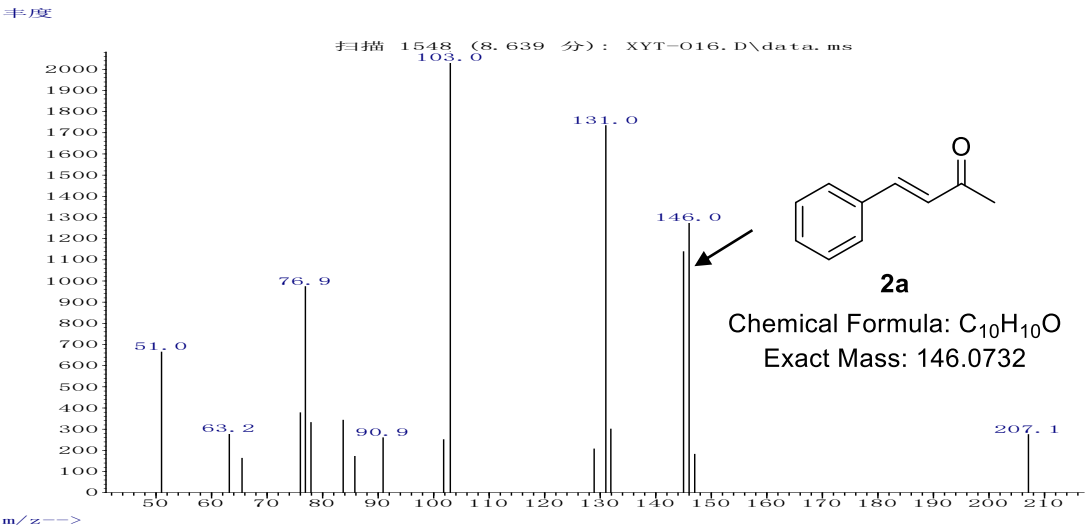
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.32 (m, 6H), 6.65 (d, *J* = 16.2 Hz, 1H), 2.32 (s, 3H).





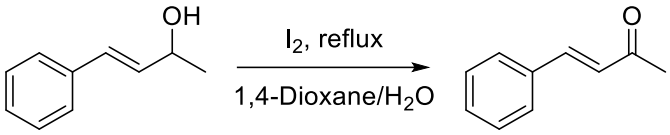
GC-MS plot of **2a**





GC-MS plot of **2a**-^{18}O

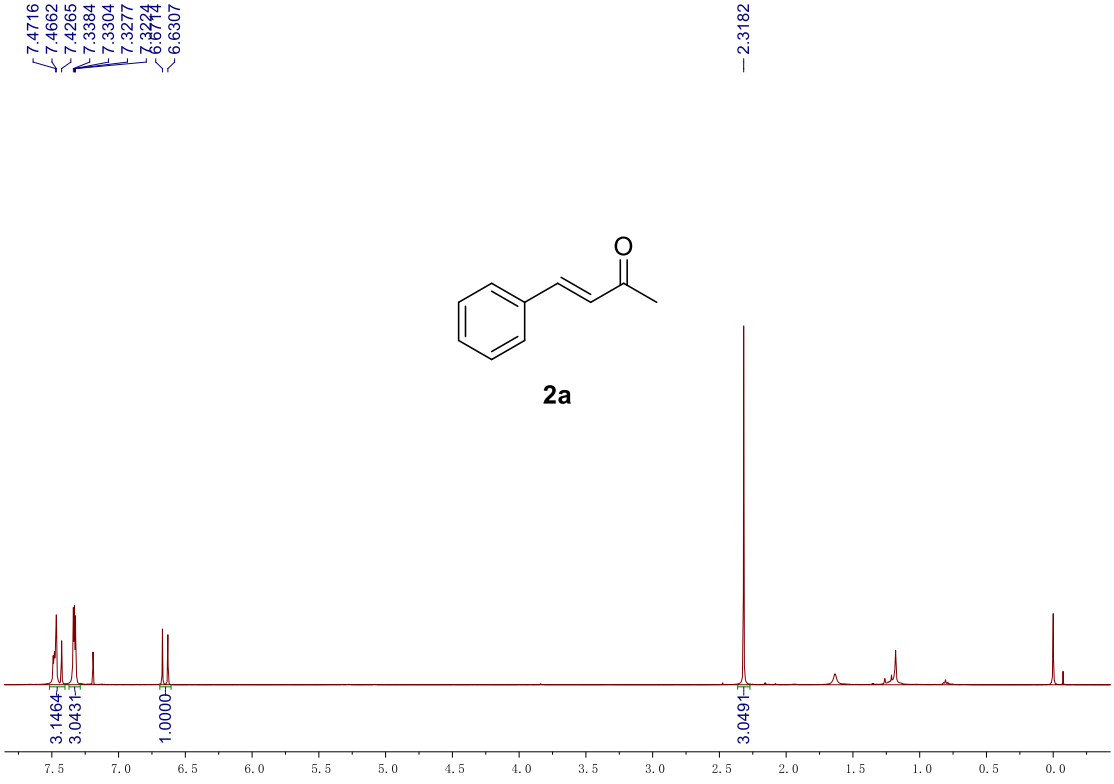
The oxidation of allylic alcohol 3



3 **2a**

To a reaction tube was added I_2 (40.6 mg, 0.16 mmol, 1.6 eq.), **3** (14.8 mg, 0.1 mmol, 1.0 eq.), 1,4-dioxane (2.0 mL) and H_2O (0.4 mL). The mixture was refluxed for 24 h, then cooled to room teperature. The reaction was quenched with aq. $Na_2S_2O_3$ and extracted with EtOAc (5 mL × 3). The organic layer was washed with H_2O and brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by column chromatography (EtOAc/hexane = 1:30) to afford **2a** (12.3 mg, 84% yield) as colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.42 (m, 3H), 7.34 – 7.32 (m, 3H), 6.65 (d, *J* = 16.3 Hz, 1H), 2.32 (s, 3H).



6. References

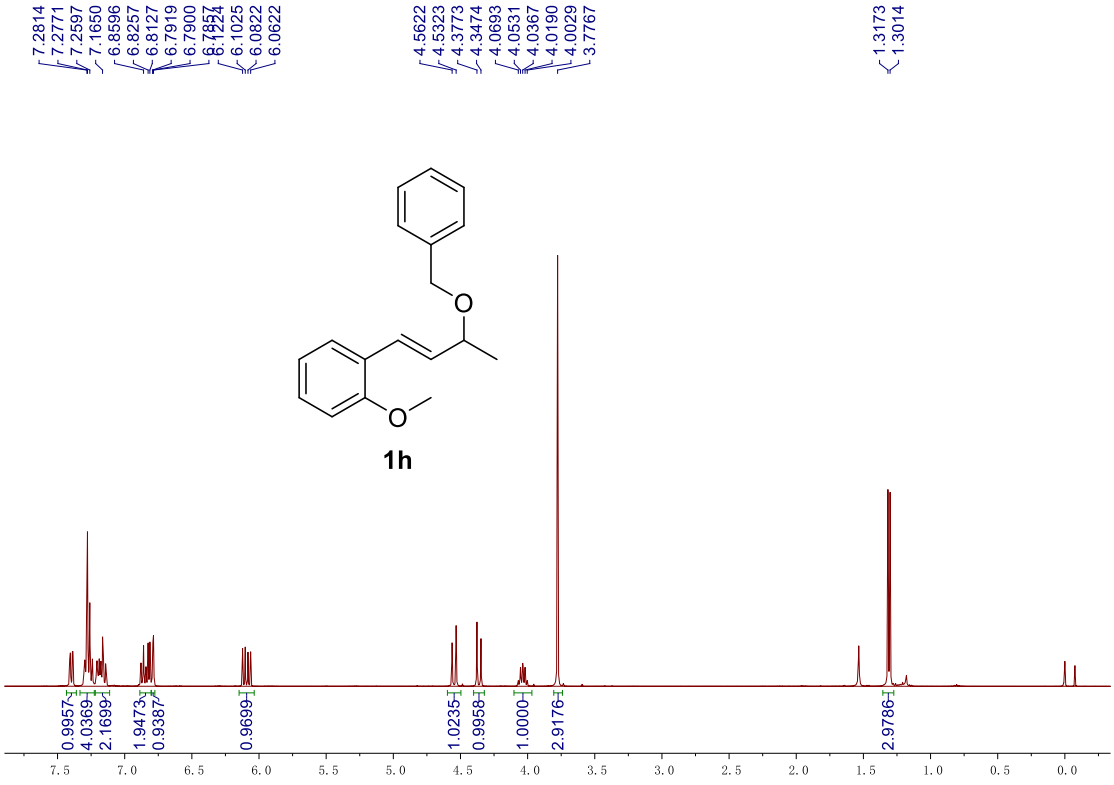
1 W. Gladkowski, A. Skrobiszewski, M. Mazur, M. Siepka, A. Pawlak, B. Obminska-Mrukowicz, A. Bialonska, D. Poradowski, A. Drynda, M. Urbaniak, *Tetrahedron*, 2013, **69**, 10414-10423.

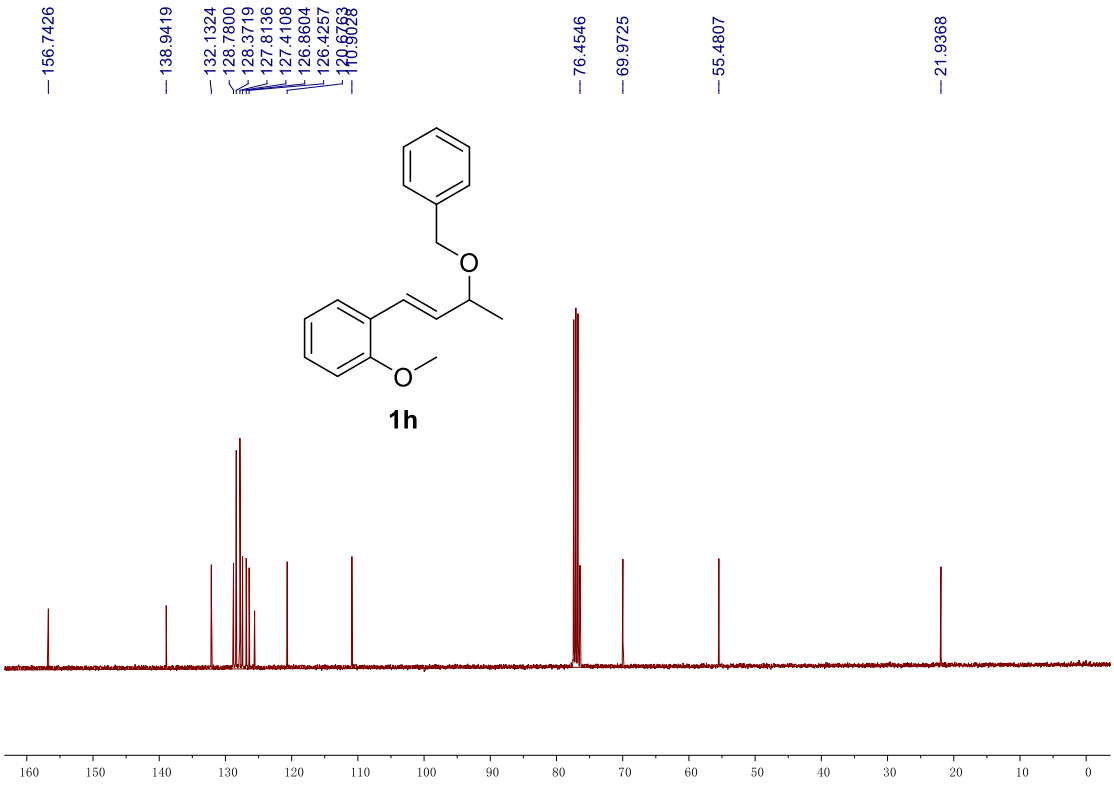
2 J. Waser, B. Gaspar, H. Nambu, E. M. Carreira, *J. Am. Chem. Soc.*, 2006, **128**, 11693-11712.

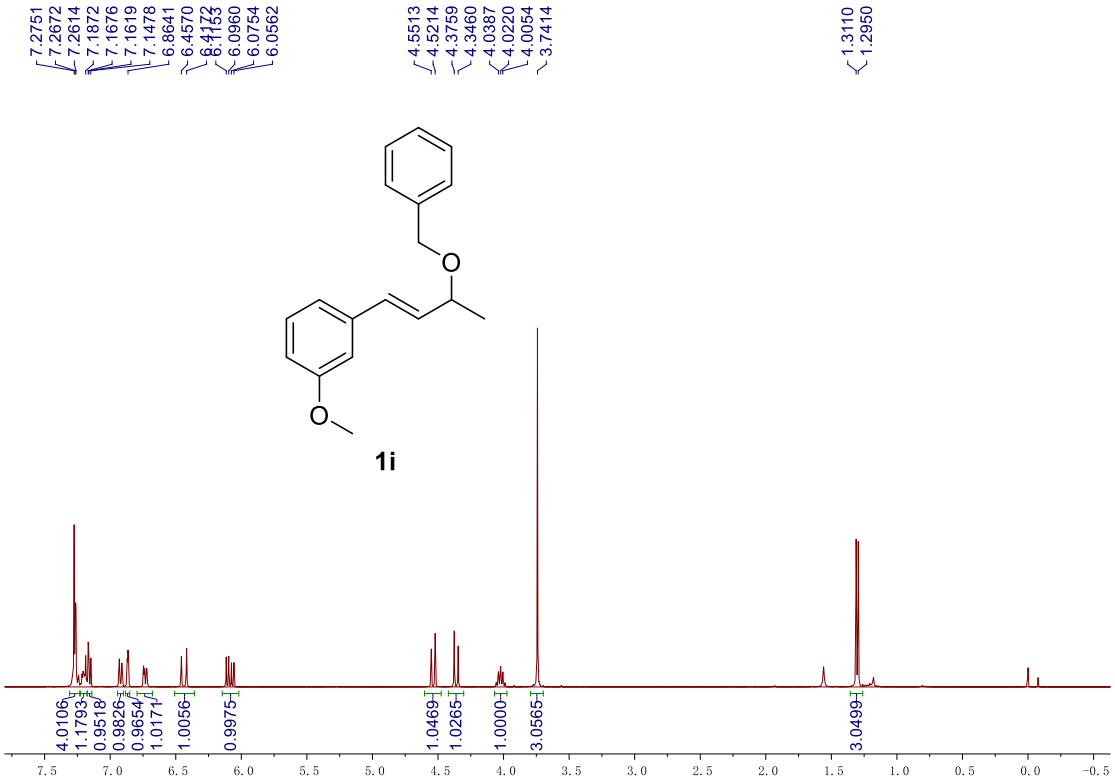
3 A. B. Charette, M.-C. Lacasse, *Org Lett.*, 2002, **4**, 3351-3353.

4 F. Chen, Y. Zhang, L. Yu, S. Zhu, *Angew. Chem., Int., Ed.*, 2017, **56**, 2022-2025.

7. NMR spectra



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6.0754
6.0562
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4.3460
4.0387
4.0220
4.0054
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1.2950
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O
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1.0265
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3.0499
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7.0
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6.0
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4.5
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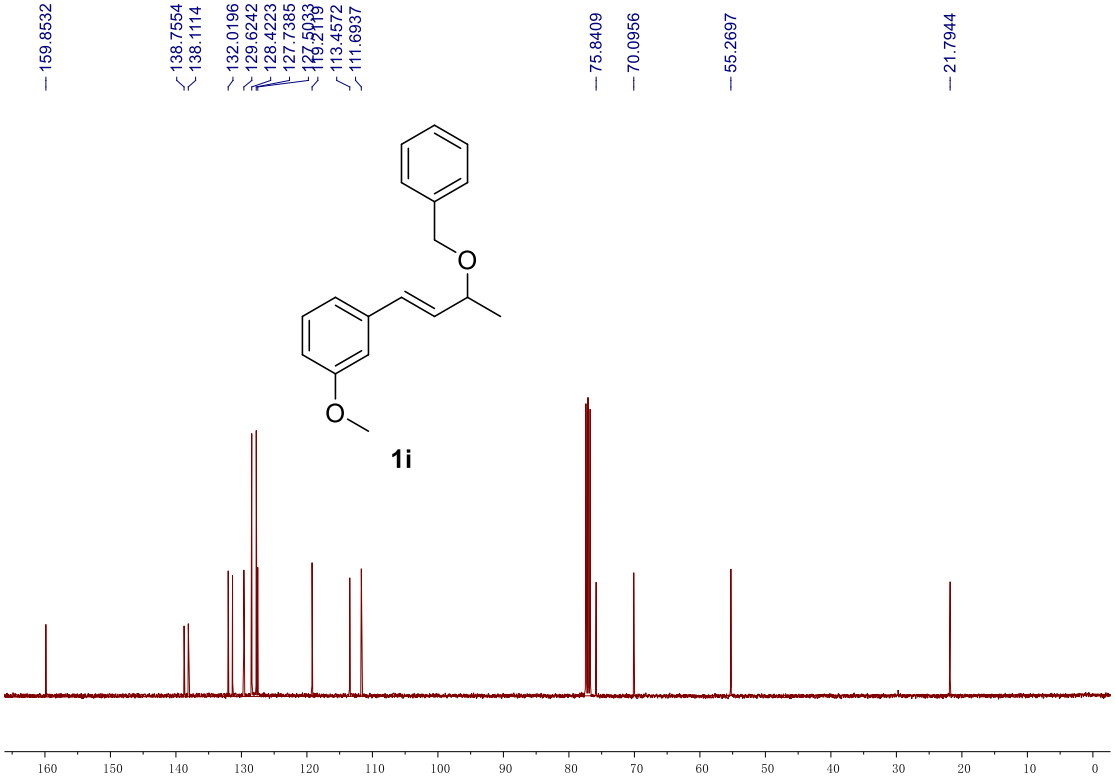
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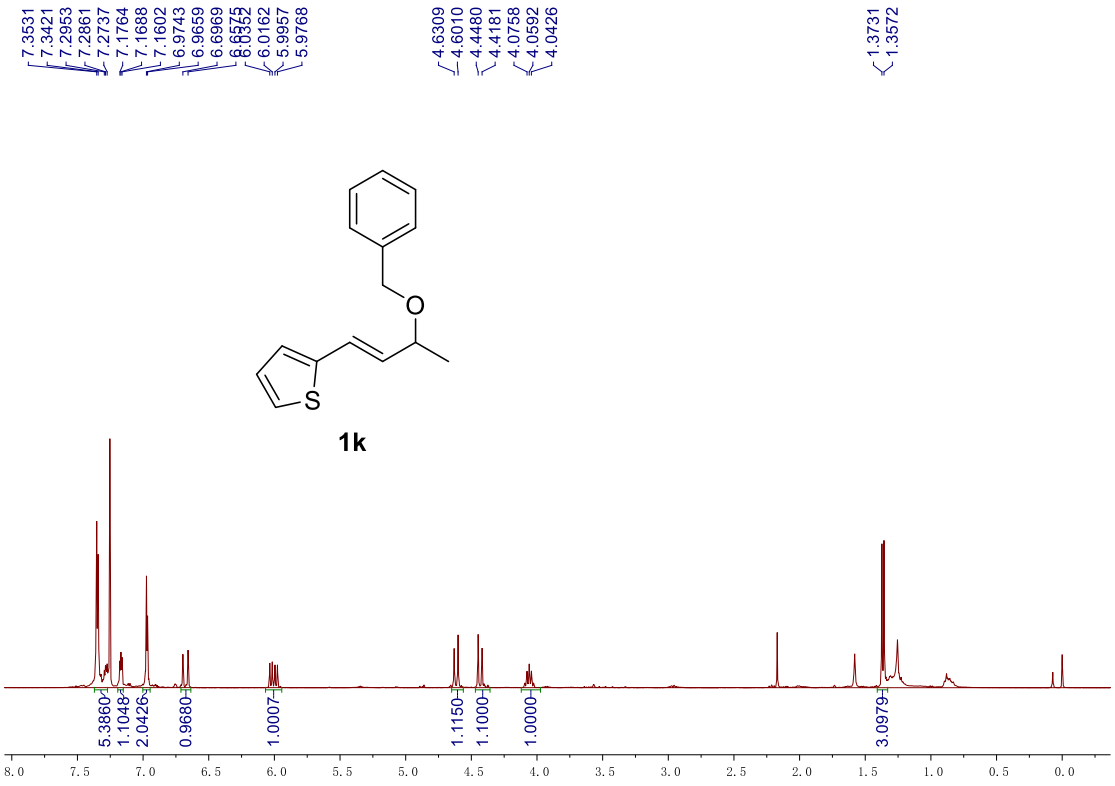
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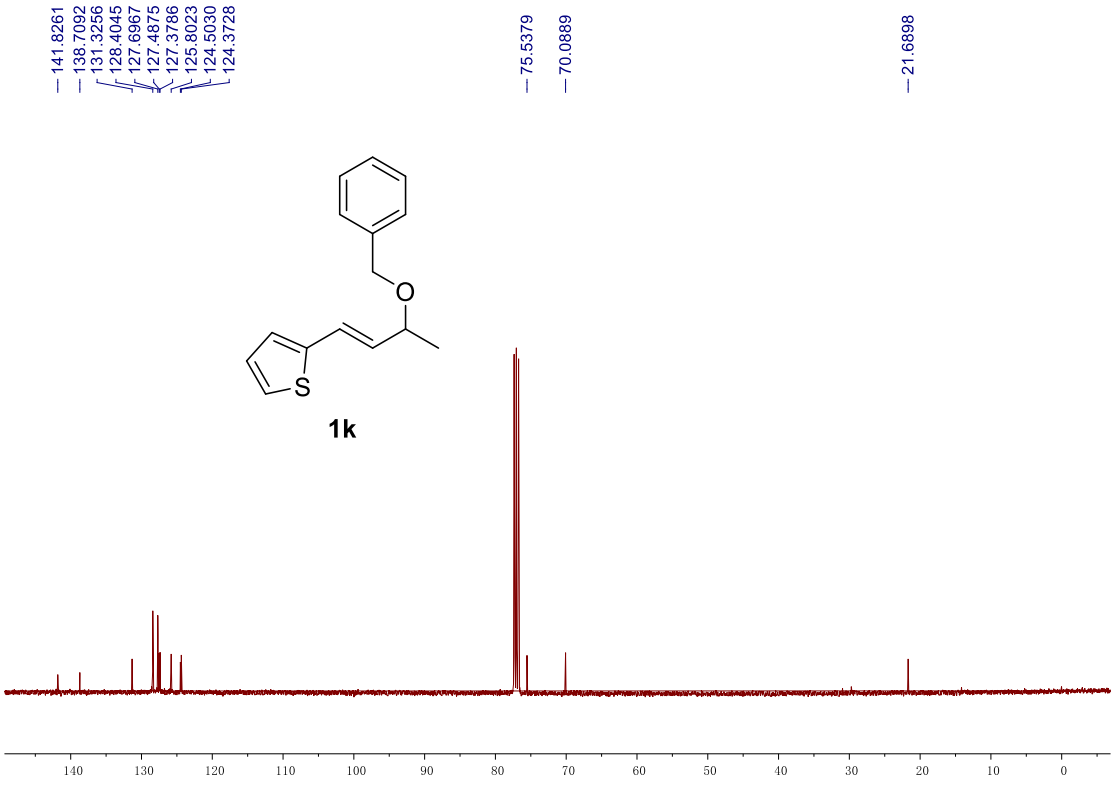
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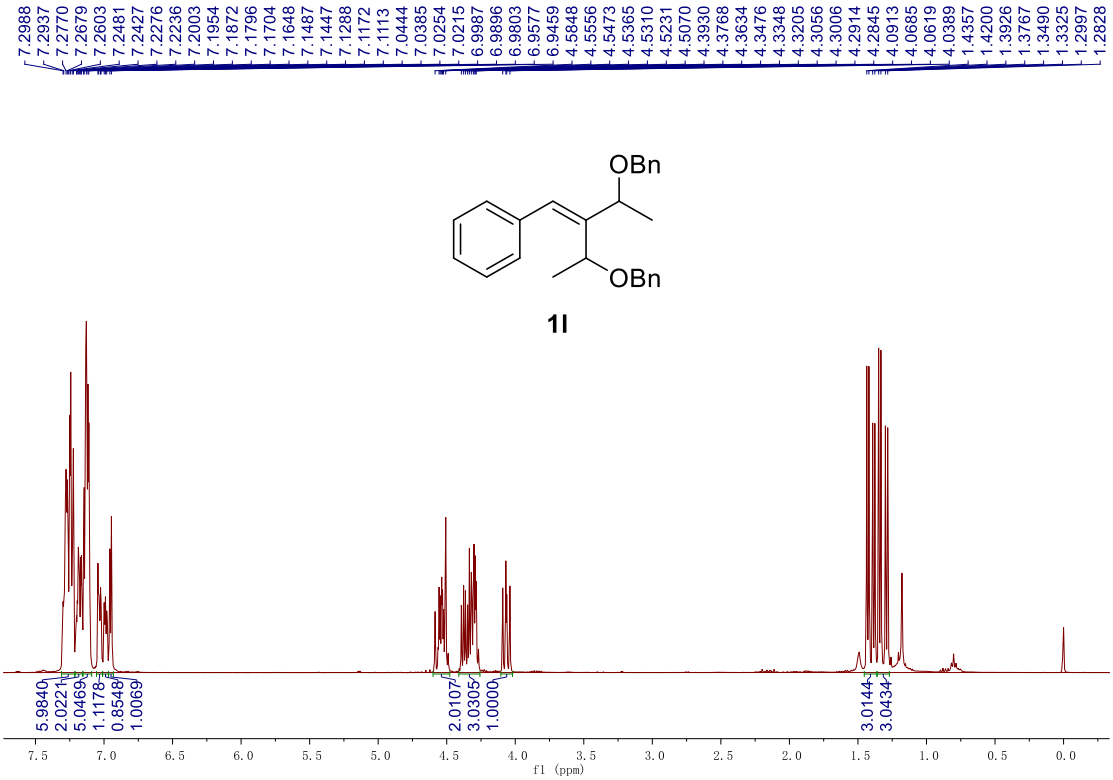
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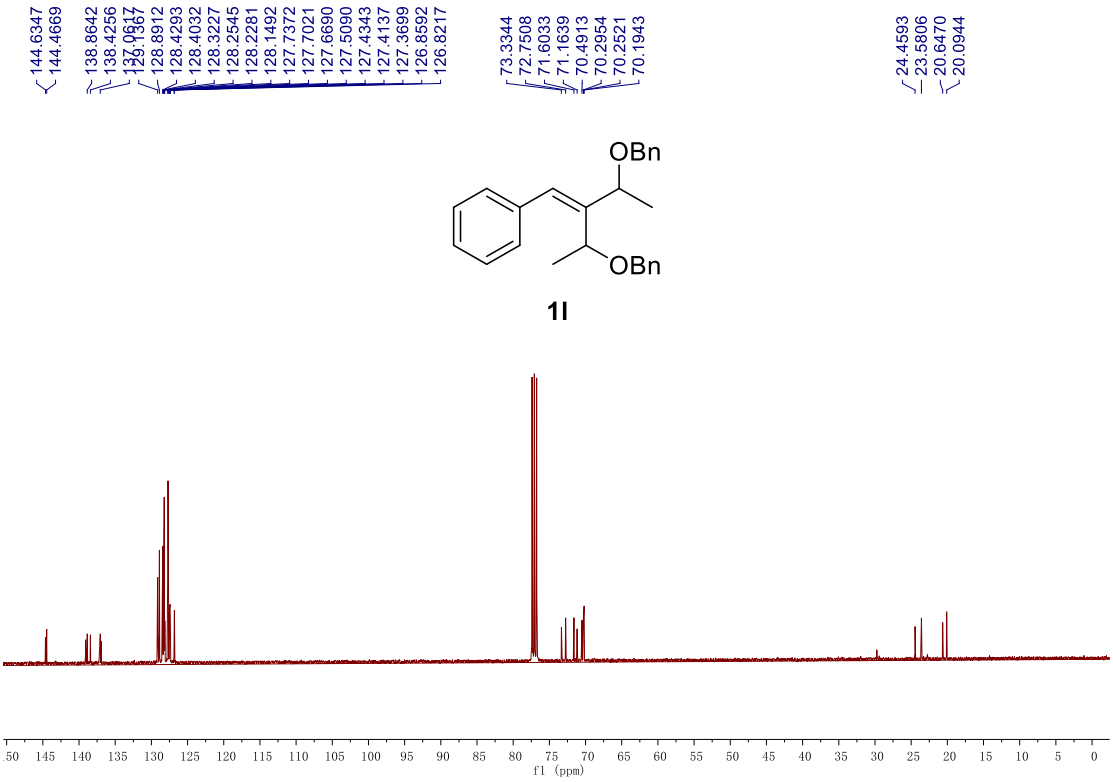
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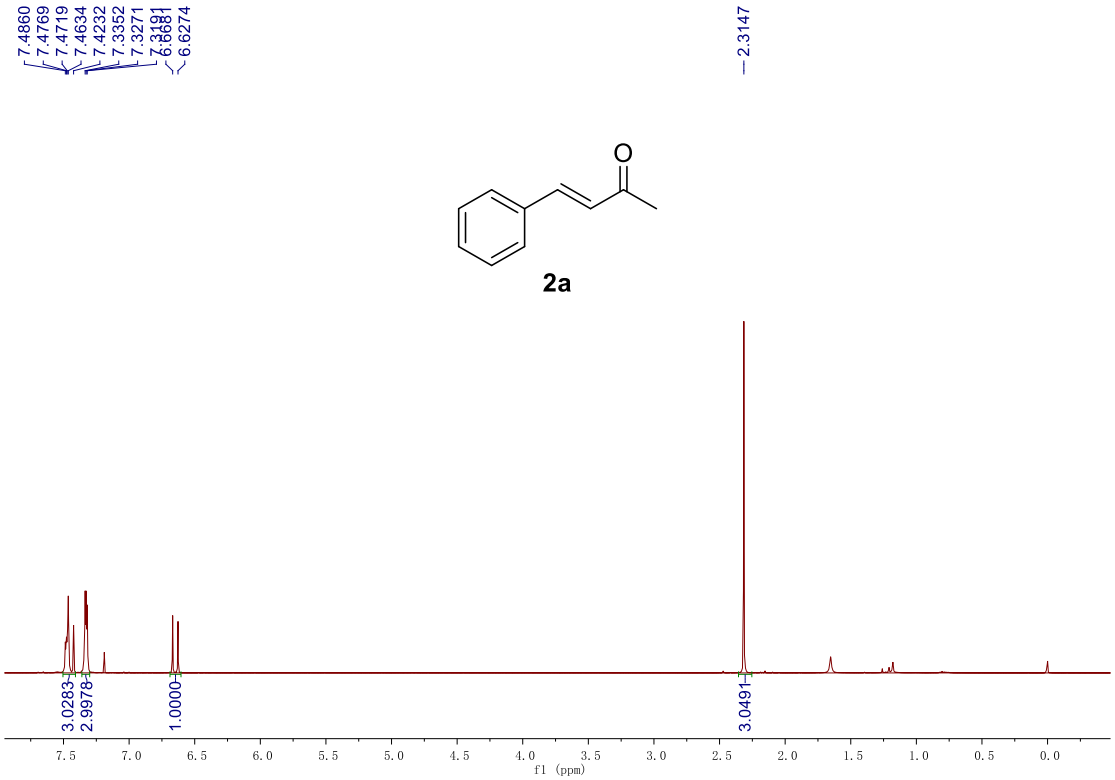
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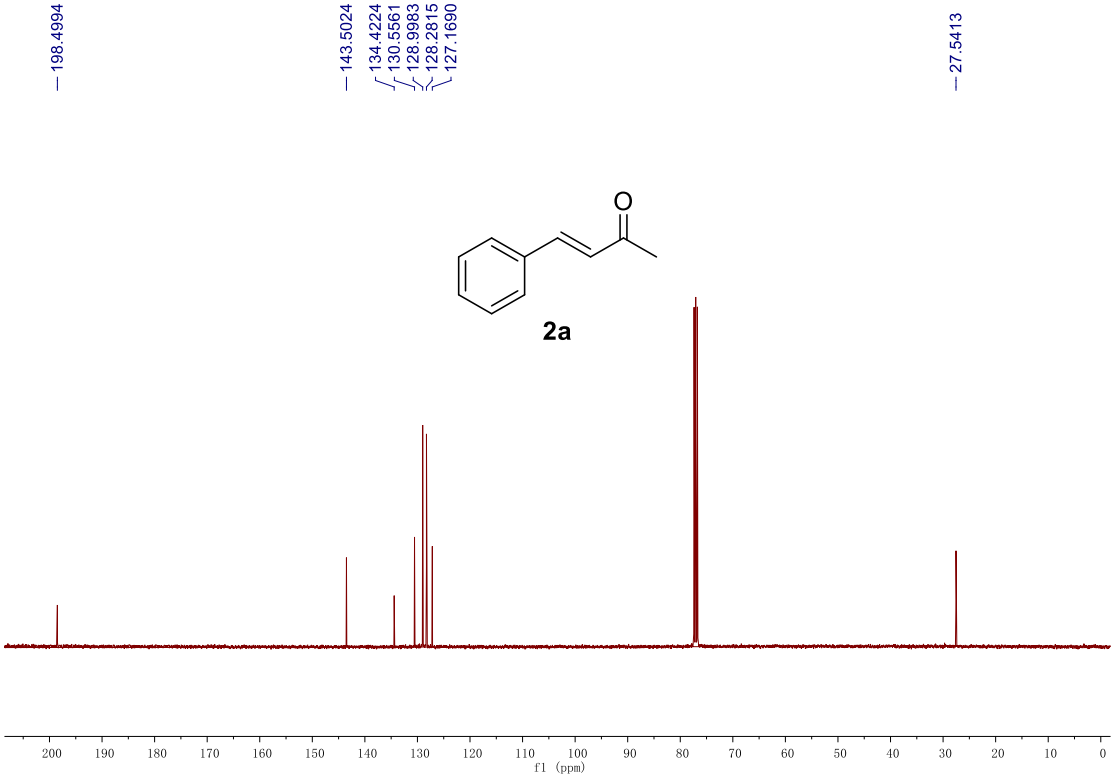
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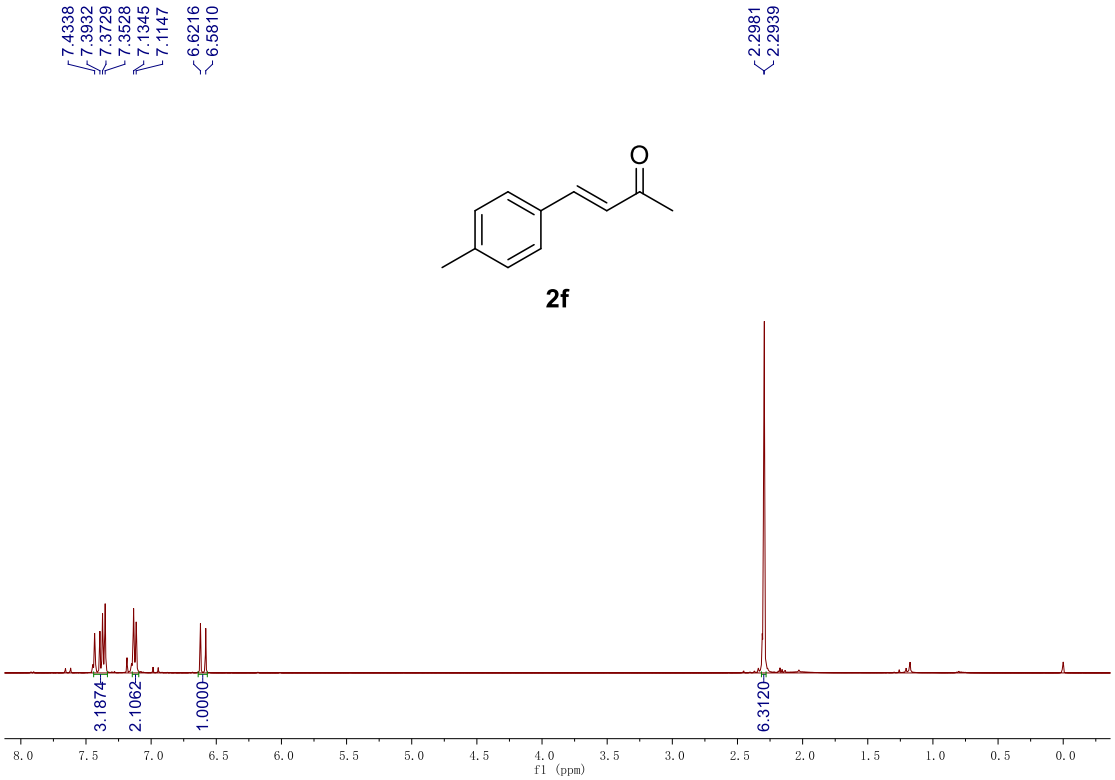
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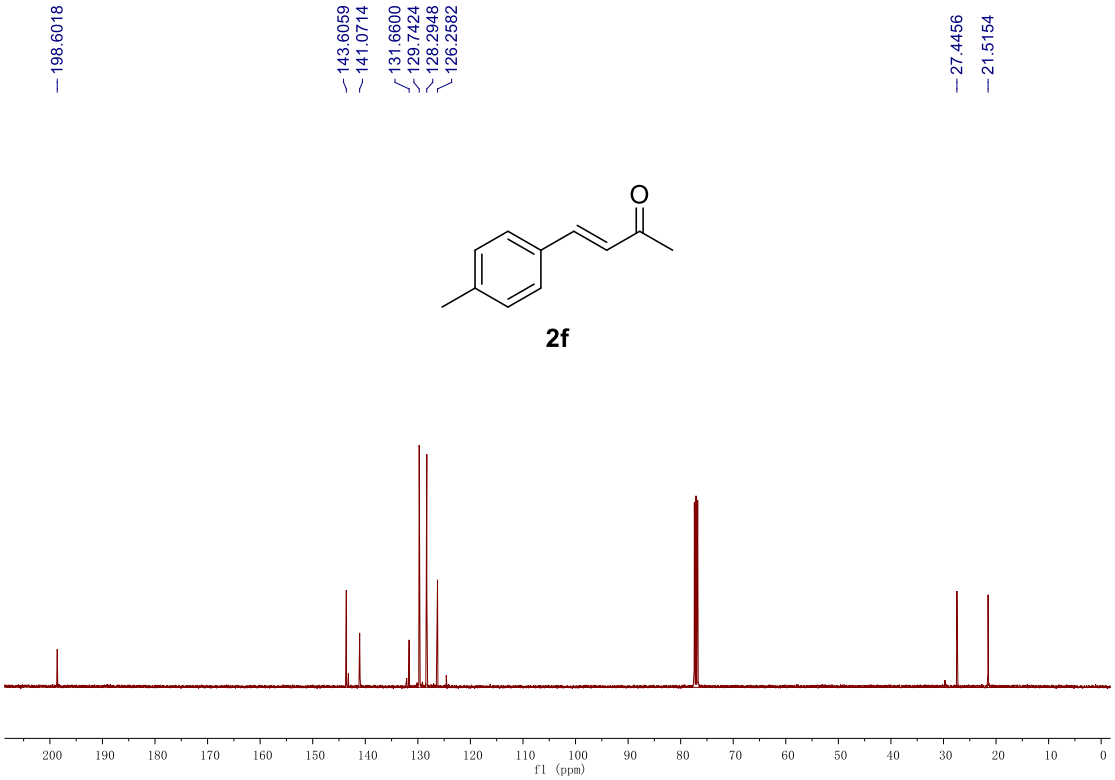
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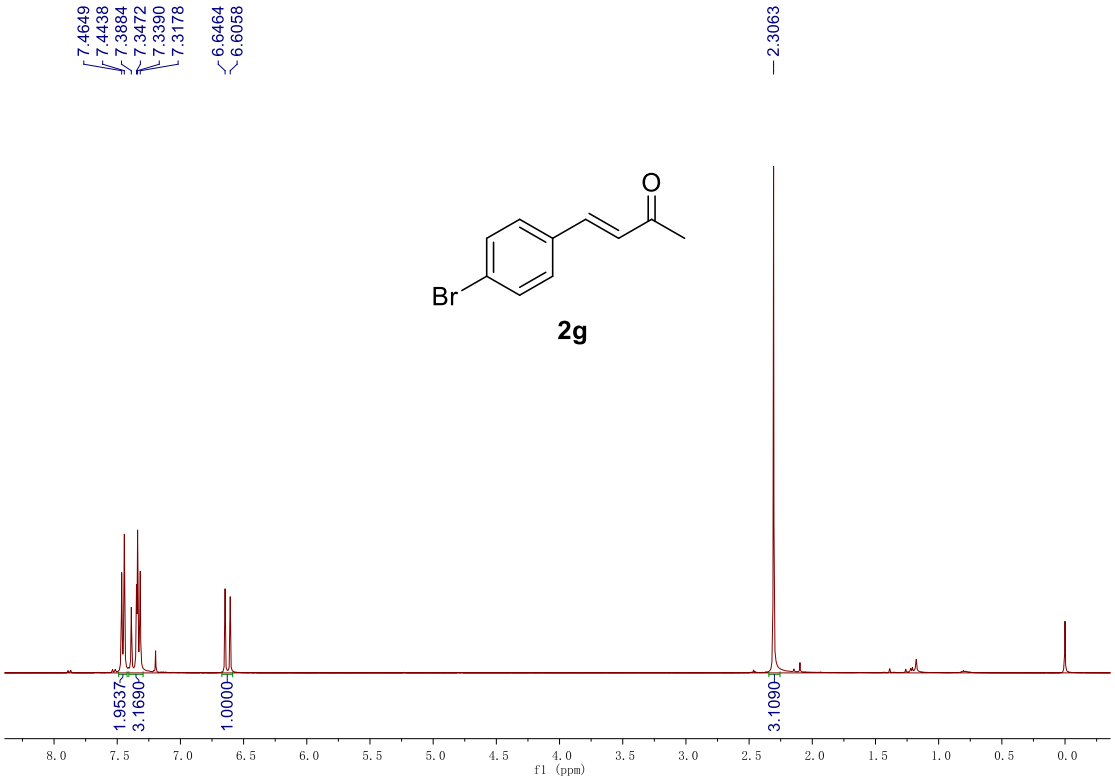
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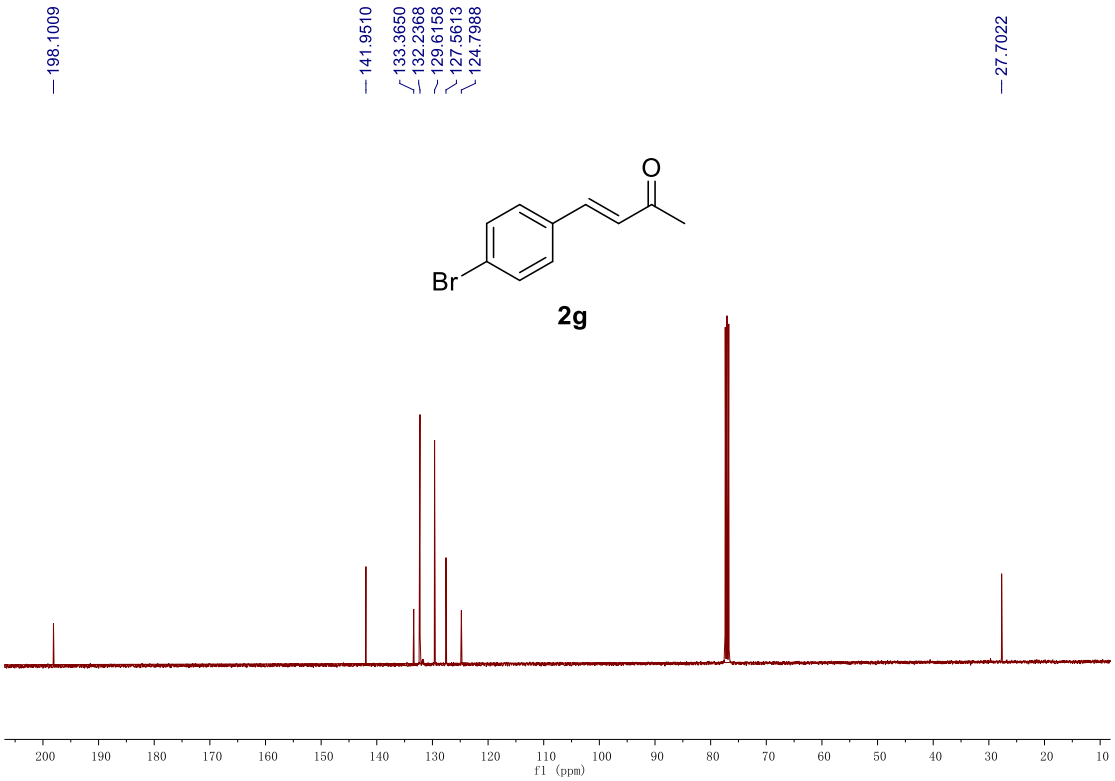
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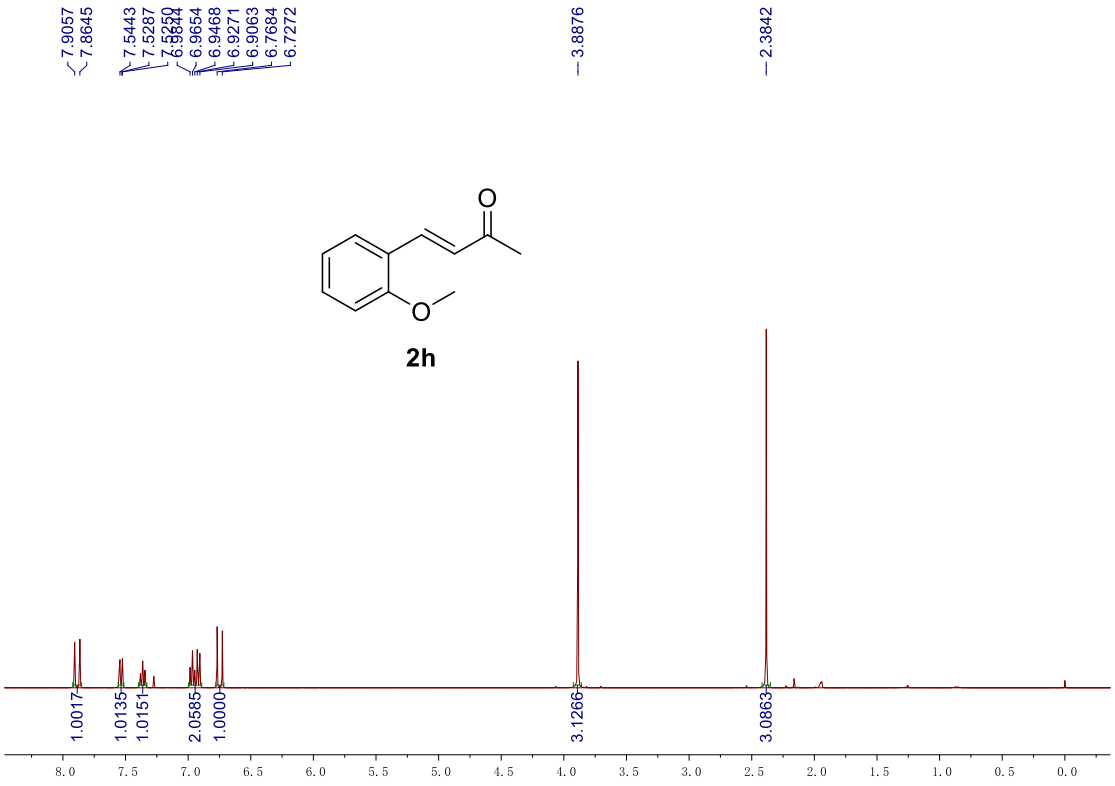
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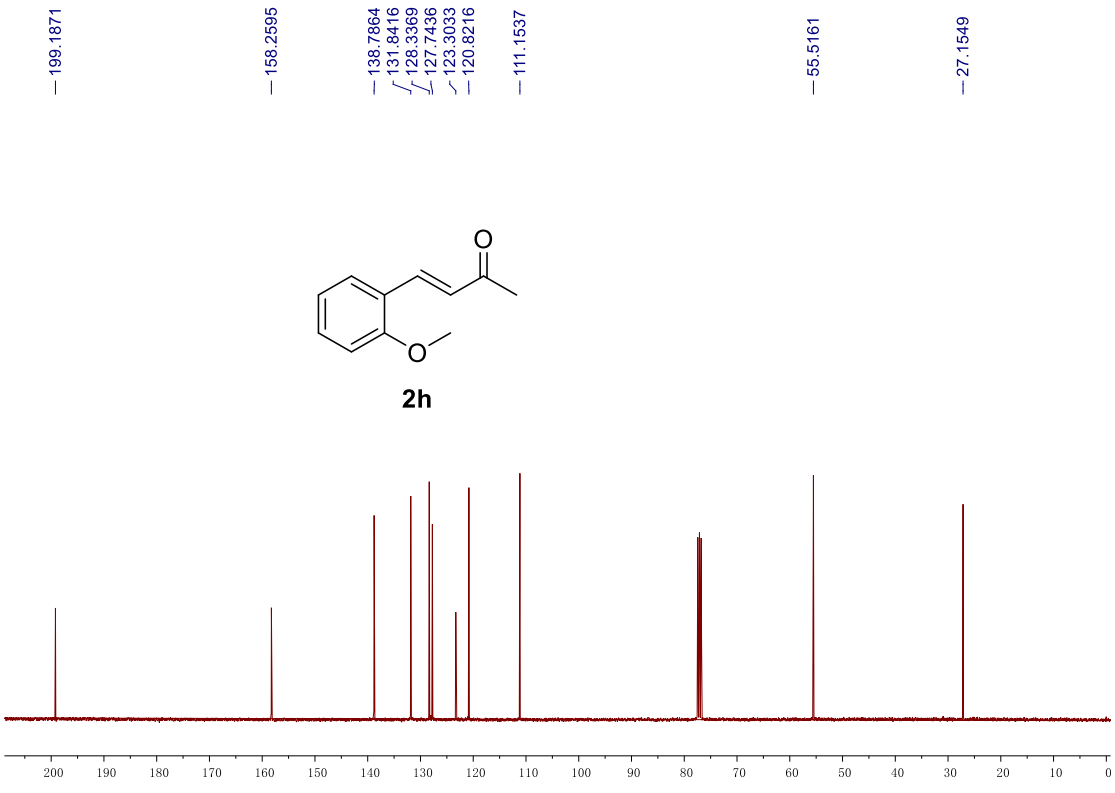
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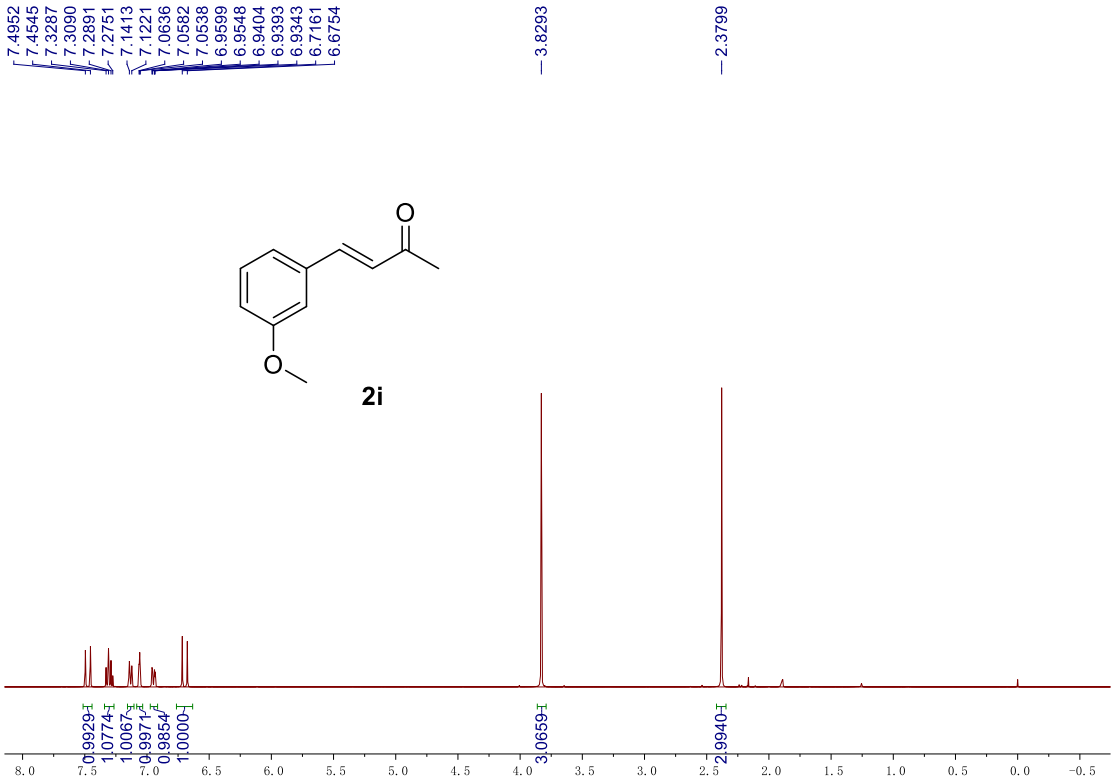
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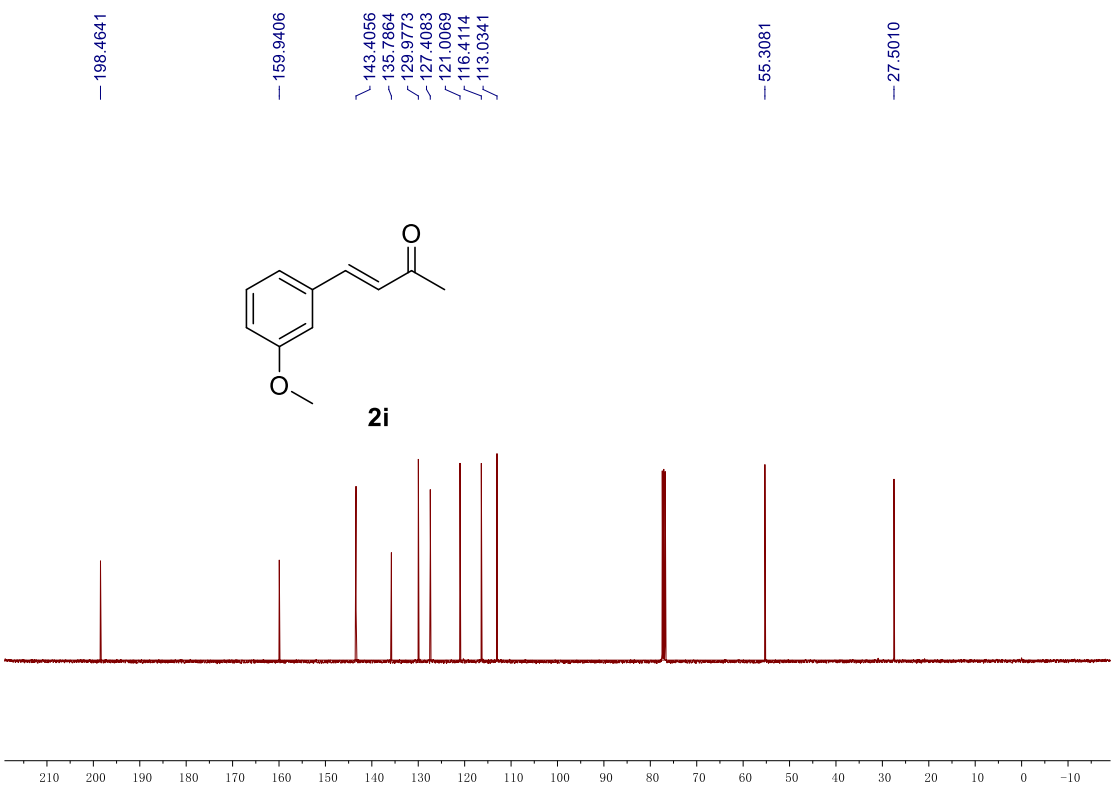
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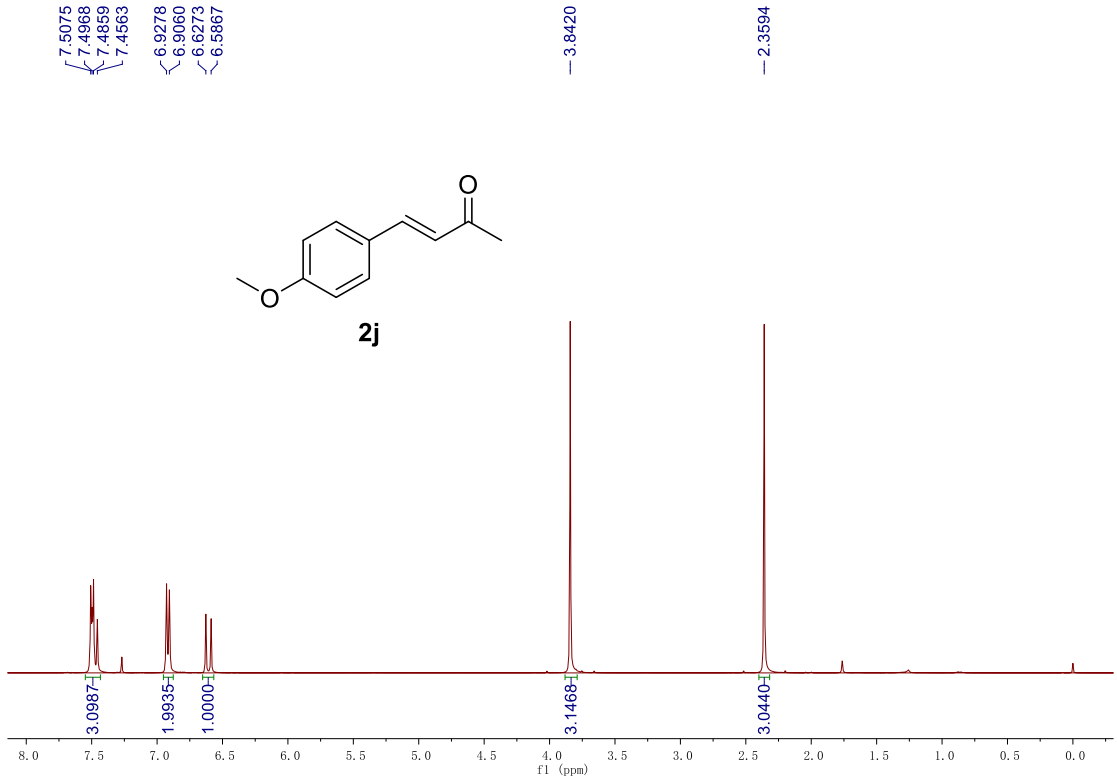
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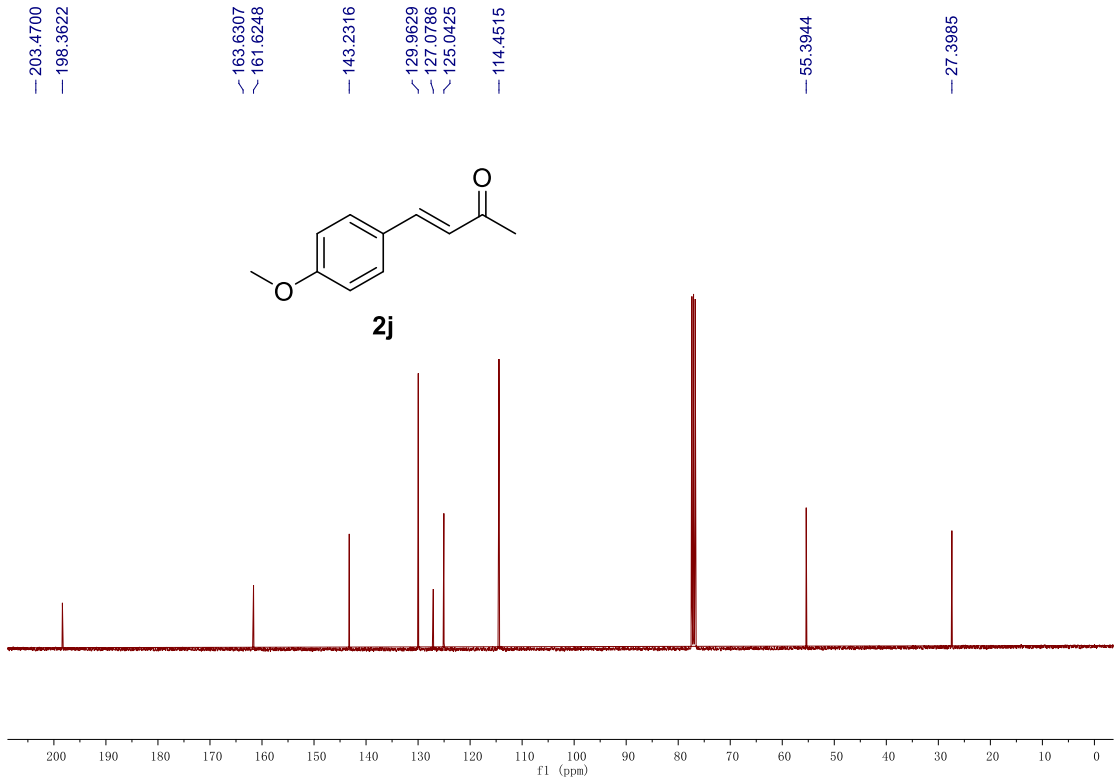
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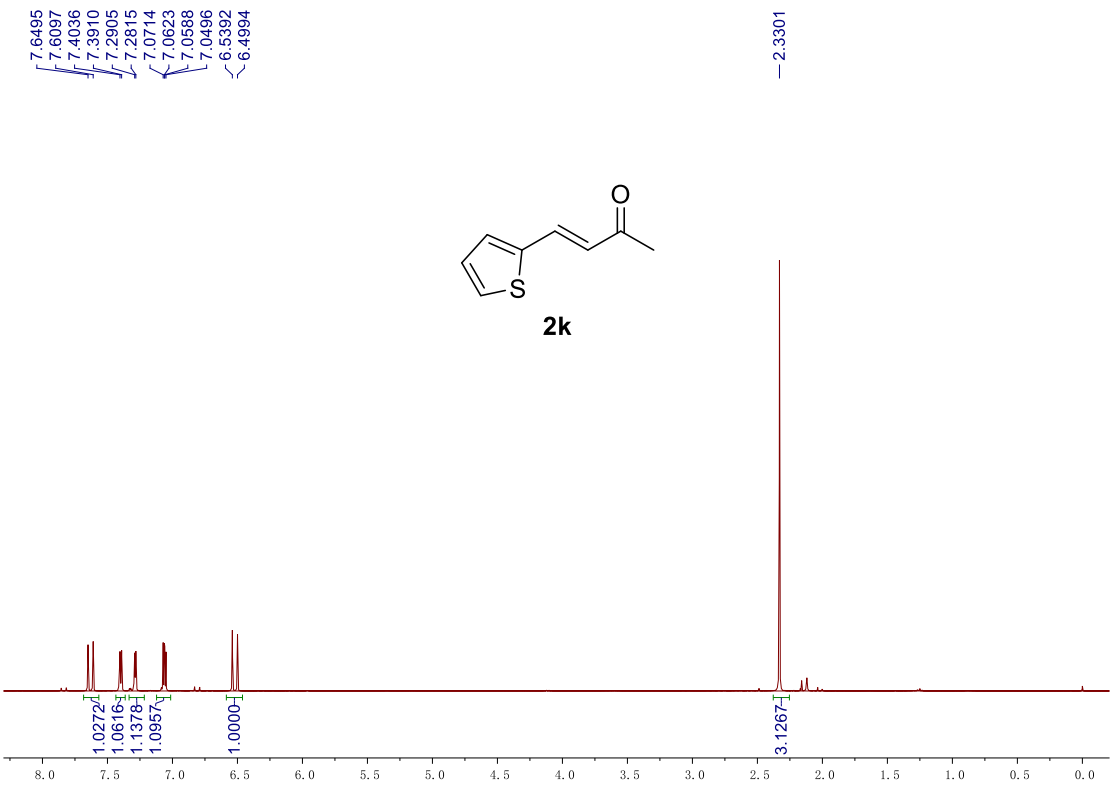
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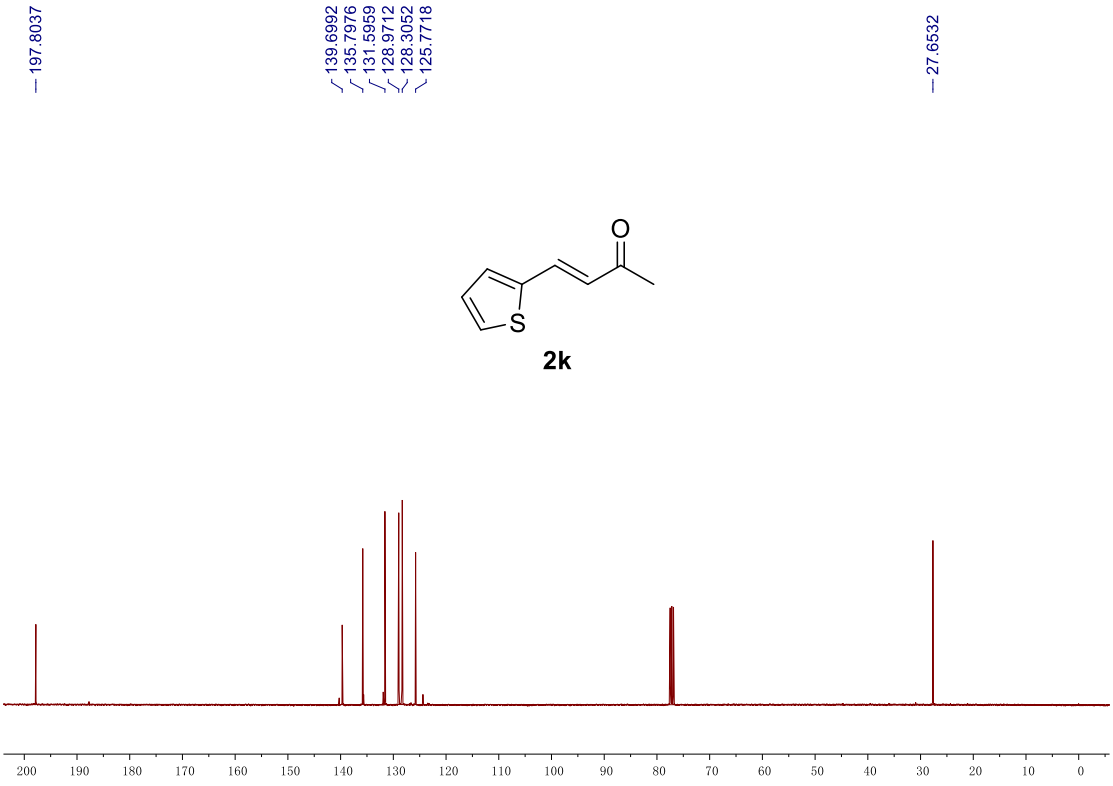
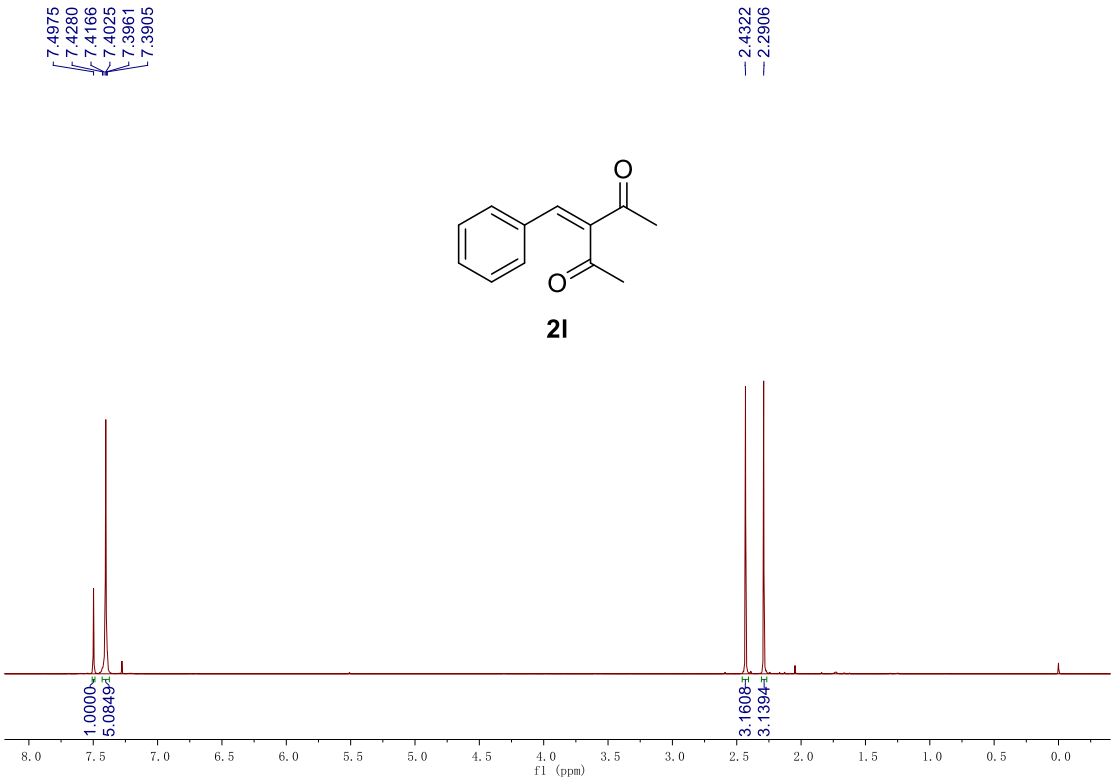
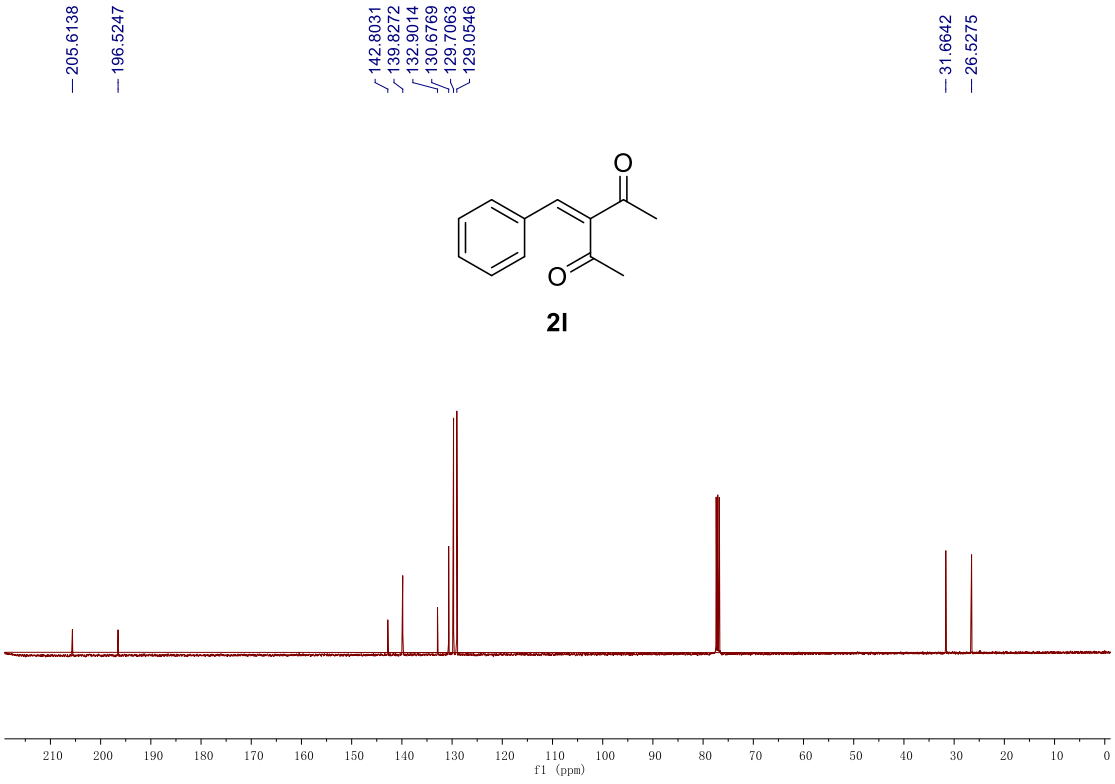
0.5
0.0

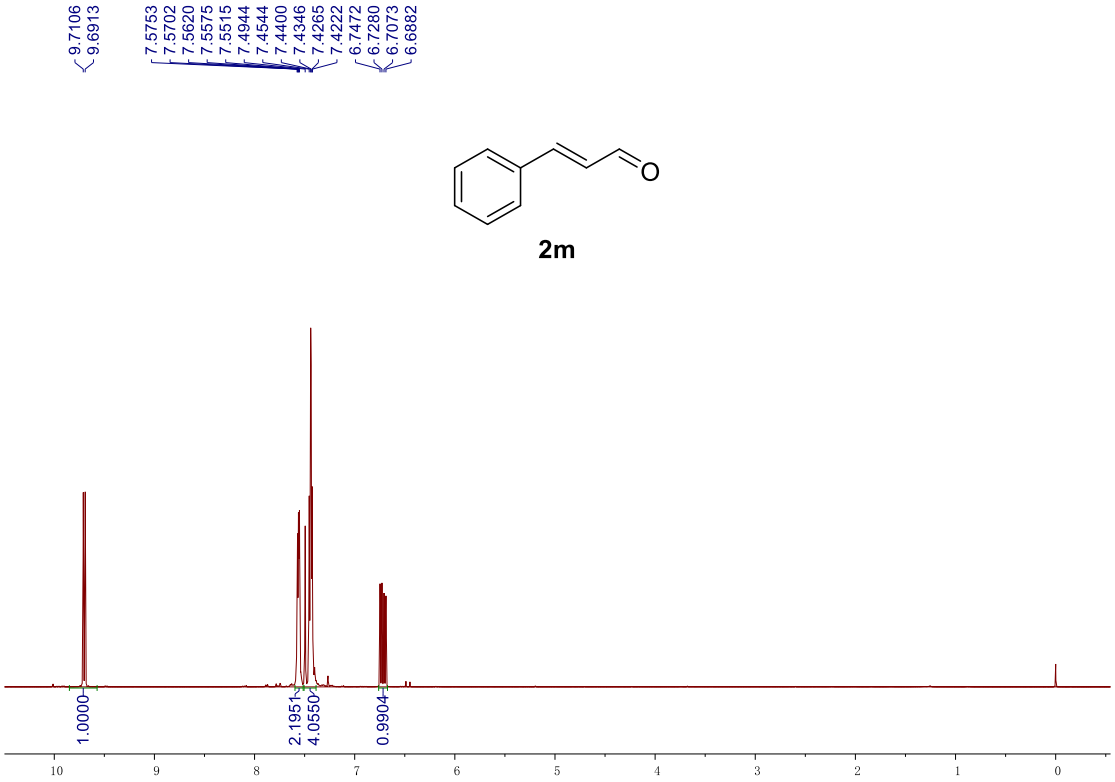

197.8037
139.6992
135.7976
131.5959
128.9712
128.3052
125.7718
27.6532
O
S
2k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

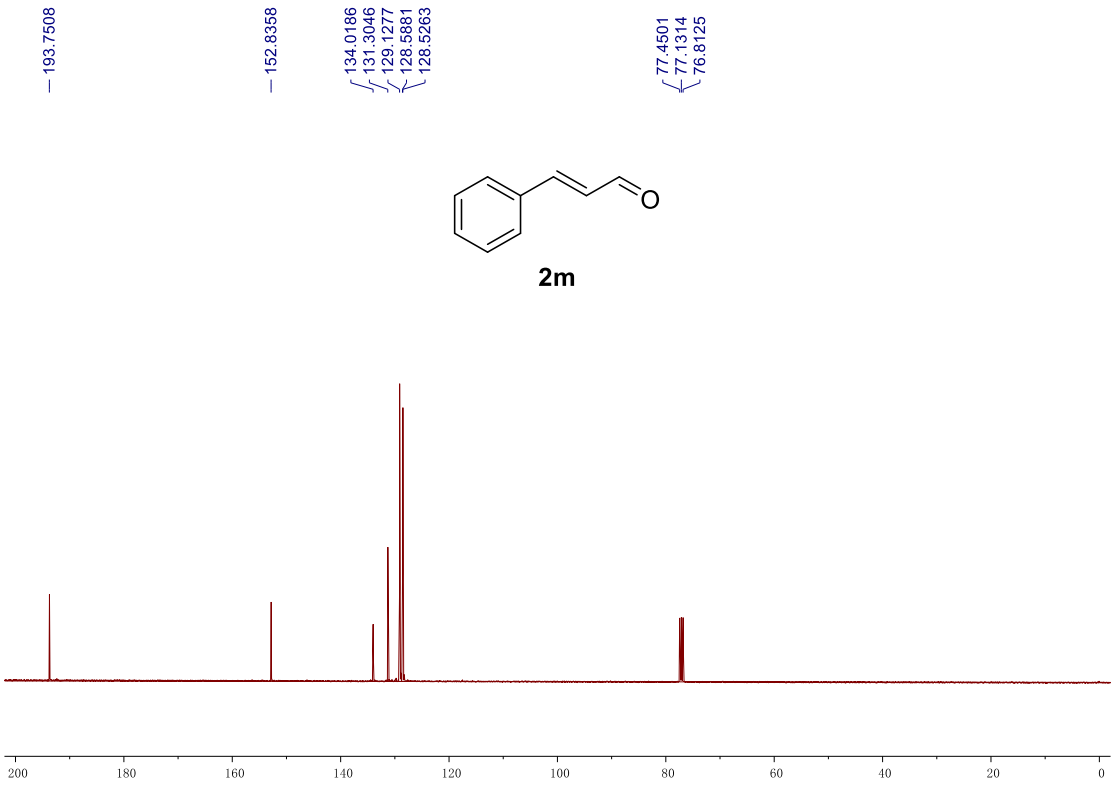

7.4975
7.4280
7.4166
7.4025
7.3961
7.3905
2.4322
2.2906
O
O
1.0000
5.0849
3.1608
3.1394
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

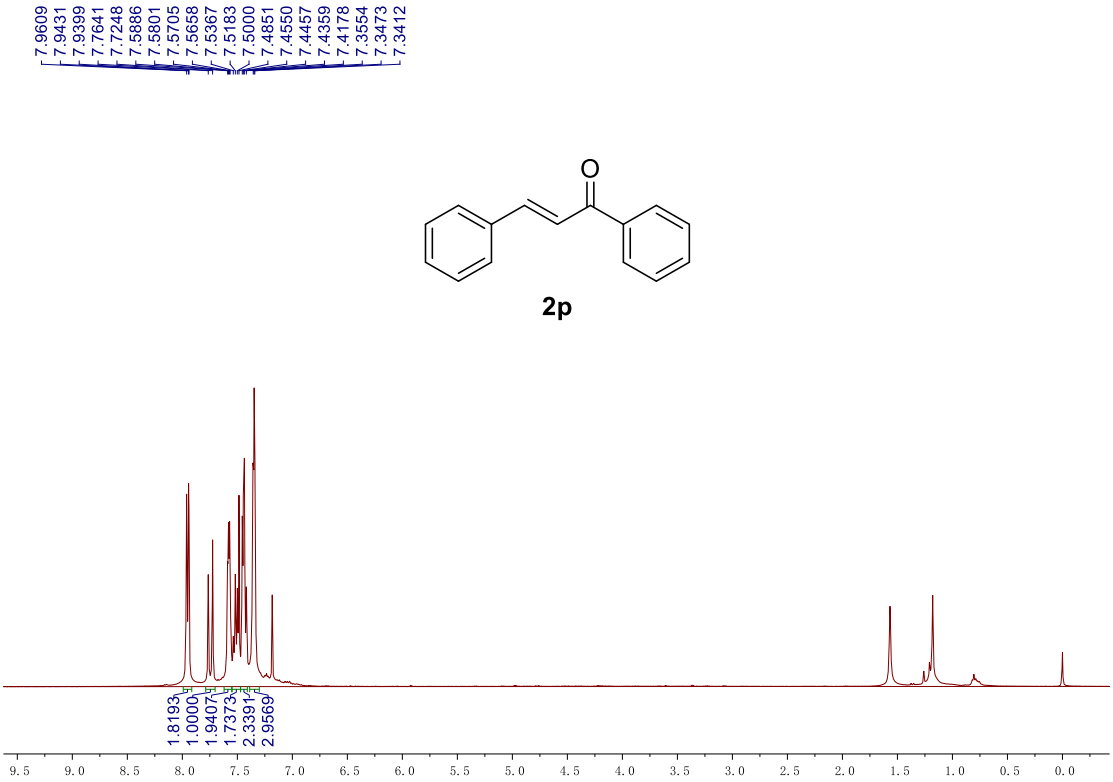
2l


205.6138
196.5247
142.8031
139.8272
132.9014
130.6769
129.7063
129.0546
31.6642
26.5275
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

2l


9.7106
9.6913
7.5753
7.5702
7.5620
7.5575
7.5515
7.4944
7.4544
7.4400
7.4346
7.4265
7.4222
6.7472
6.7280
6.7073
6.6882
O
2m
1.0000
2.1951
4.0550
0.9904
10
9
8
7
6
5
4
3
2
1
0


193.7508
152.8358
134.0186
131.3046
129.1277
128.5881
128.5263
77.4501
77.1314
76.8125
O
2m
200
180
160
140
120
100
80
60
40
20
0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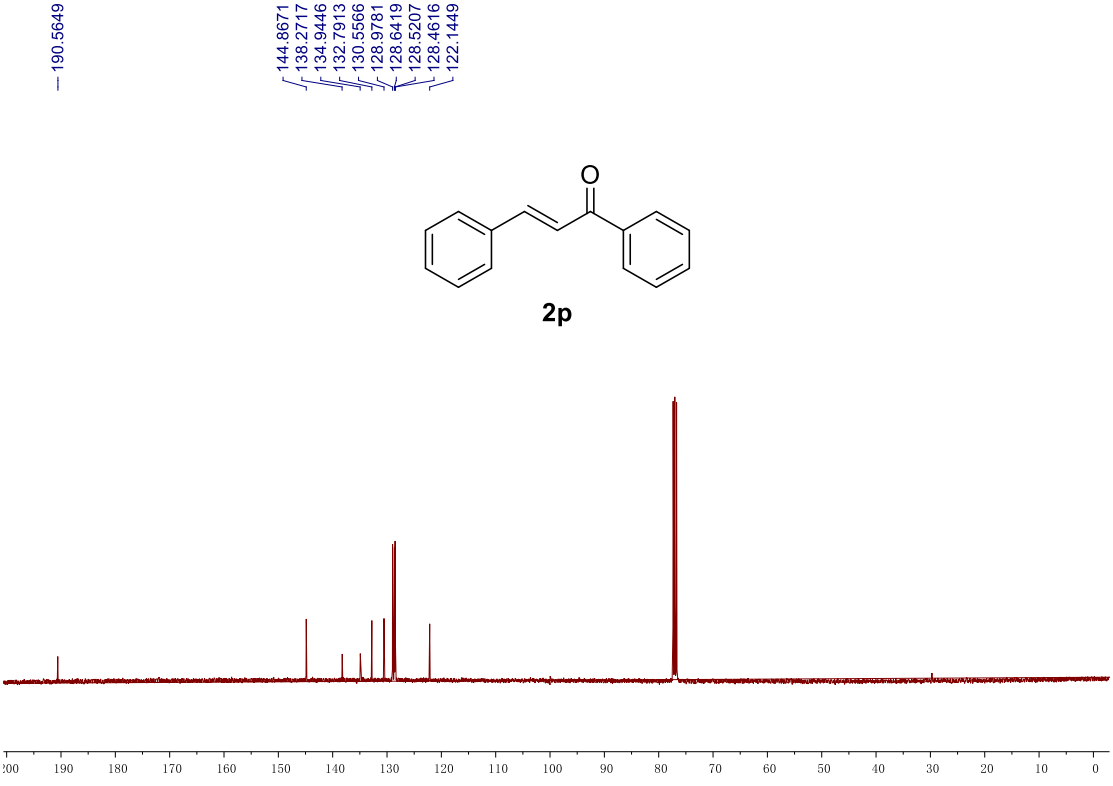
7.9431
7.9399
7.7641
7.7248
7.5886
7.5801
7.5705
7.5658
7.5367
7.5183
7.5000
7.4851
7.4550
7.4457
7.4359
7.4178
7.3554
7.3473
7.3412
O
2p
1.8193
1.0000
1.9407
1.7373
2.3391
2.9569
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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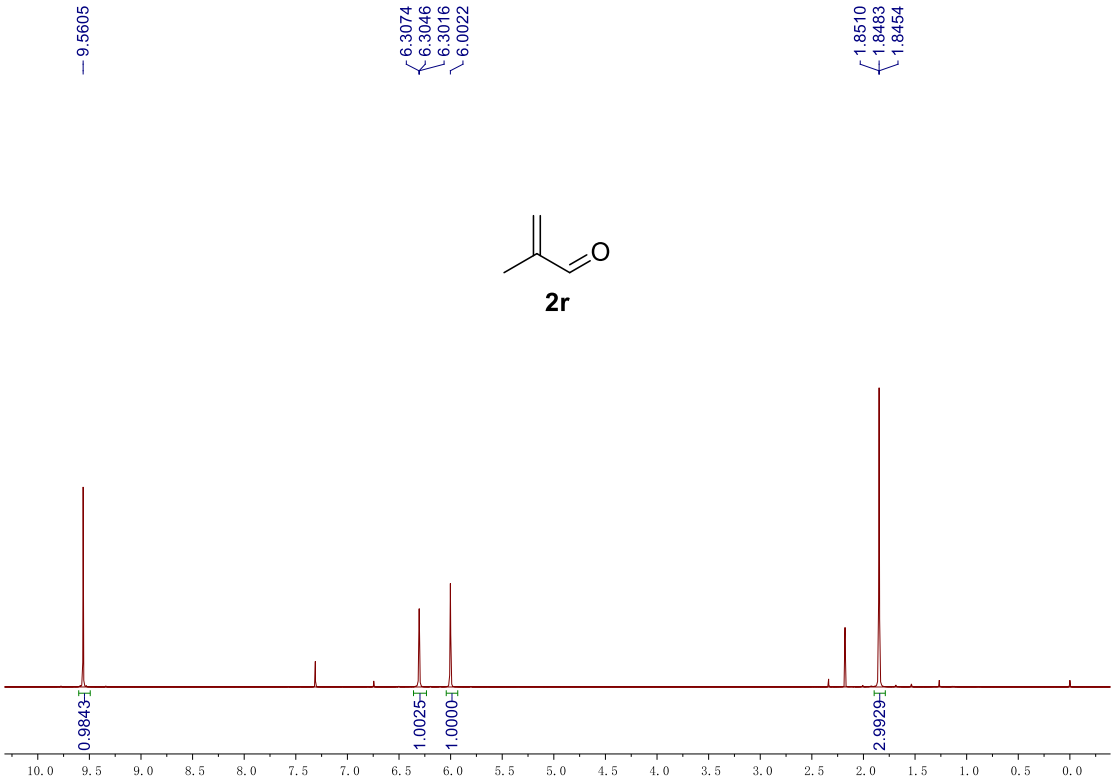
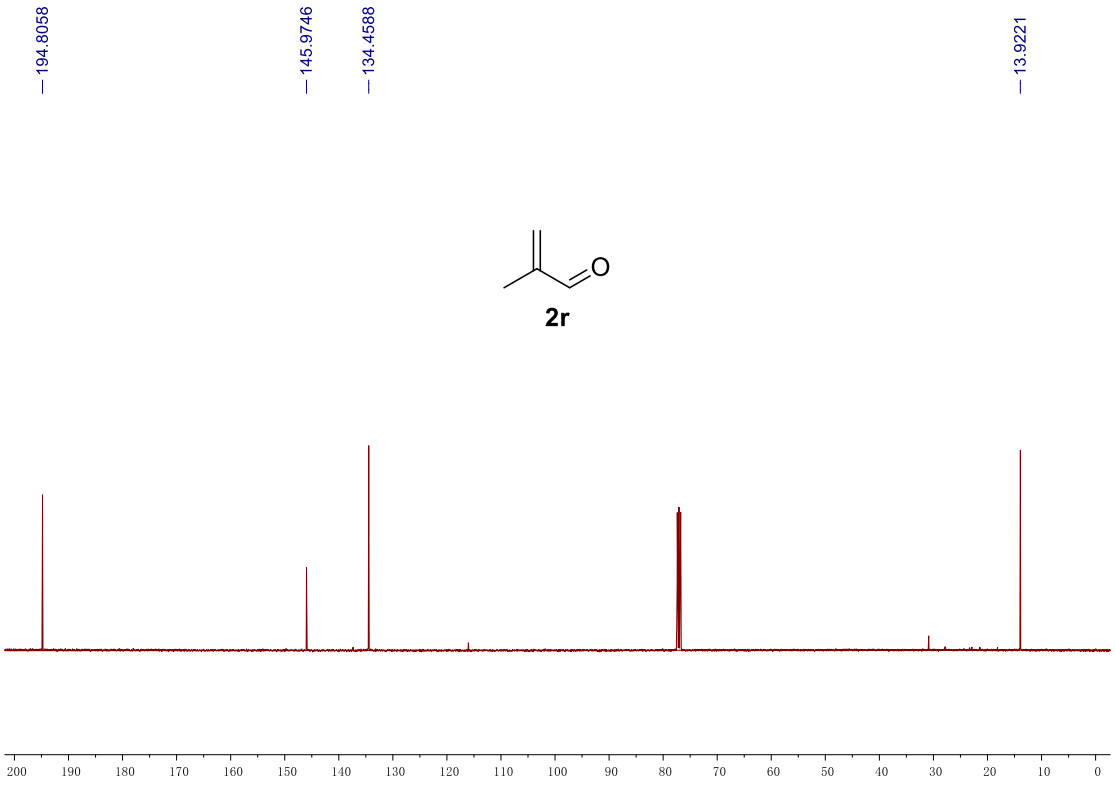
144.8671
138.2717
134.9446
132.7913
130.5566
128.9781
128.6419
128.5207
128.4616
122.1449
O
2p
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


9.5605
6.3074
6.3046
6.3016
6.0022
1.8510
1.8483
1.8454
O
2r
0.9843
1.0025
1.0000
2.9929
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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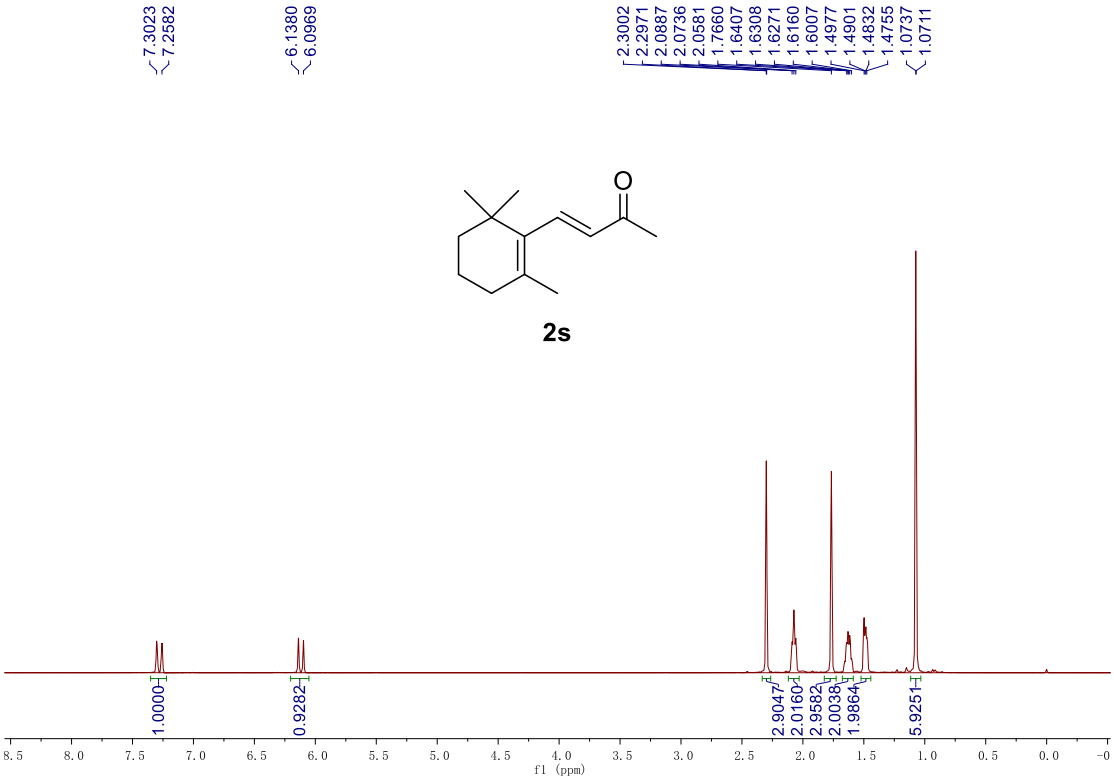
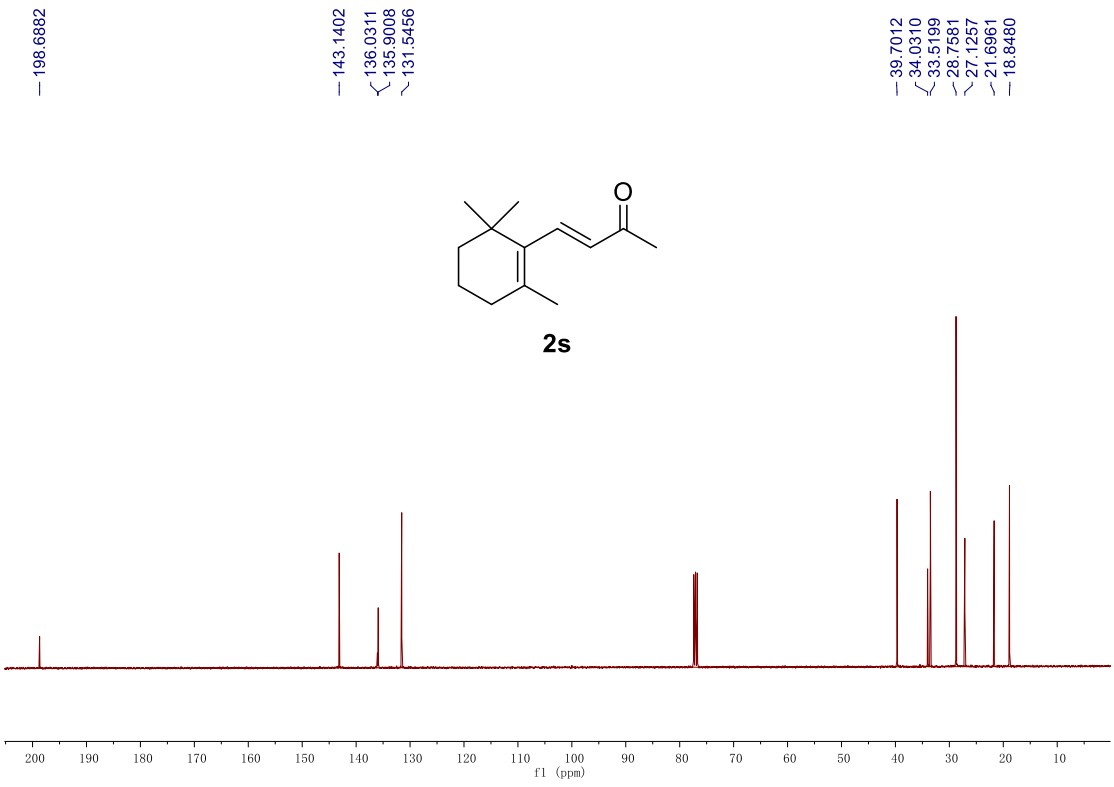
145.9746
134.4588
13.9221
O
2r
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


7.3023
7.2582
6.1380
6.0969
2.3002
2.2971
2.0887
2.0736
2.0581
1.7660
1.6407
1.6308
1.6271
1.6160
1.6007
1.4977
1.4901
1.4832
1.4755
1.0737
1.0711
O
2s
1.0000
0.9282
2.9047
2.0160
2.9582
2.0038
1.9864
5.9251
f1 (ppm)


198.6882
143.1402
136.0311
135.9008
131.5456
39.7012
34.0310
33.5199
28.7581
27.1257
21.6961
18.8480
O
2s
f1 (ppm)