Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Iridium-catalysed branched-selective hydroacylation of 1,3-dienes with salicylaldehydes

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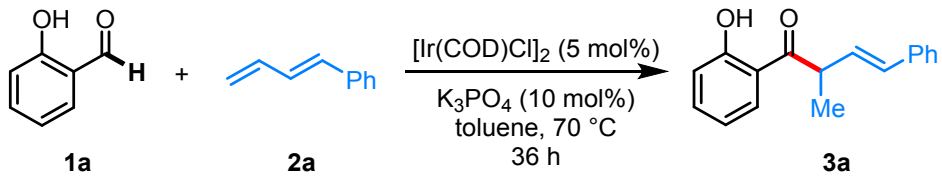
1. General Information

Unless noted otherwise, all ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on Brucker spectrometers in $CDCl_3$. Tetramethylsilane (TMS) served n internal standard (δ = 0) for ^{1}H NMR, and $CDCl_3$ was used as internal standard (δ = 77.0) for ^{13}C NMR. Chemical shifts are reported in parts per million as follows: chemical shift, multiplicity (s =singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad). Infrared (IR) spectra were obtained using a Bruker tensor 27 infrared spectrometer. High-resolution mass spectrometry (HRMS) was performed on IonSpec FT-ICR or Waters Micromass Q-TOF micro Synapt High-Definition Mass Spectrometer. Unless otherwise noted, solvents used for the key reactions were freshly distilled over calcium hydride or sodium. Toluene (Extra Dry, stabilized) used for the key reactions was purchased from Energy Chemical and degassed with nitrogen before use. All the key reactions were carried out under nitrogen atmosphere with a stir bar in a sealed vial and heated in a pie-block. Reaction temperatures were reported as the temperatures of the bather surrounding the vials. Sensitive ligands and metal catalysts and solvents were transferred under nitrogen into a nitrogen-filled glove box with standard techniques. $[Ir(COD)Cl]_2$ and ligands were purchased from commercial source. Salicylaldehydes used for the key reactions was purified by vacuum distillation, column isolation (liquid aldehyde) or recrystallization (solid aldehyde). 1-aryl 1,3-dienes were prepared by following literature procedures.[1] Athe deuterium-labelled salicylaldehyde *d*-**1a** was prepared by following literature procedures.[2] All other materials were obtained from commercial sources and were used as received.

2. Experimental Procedures

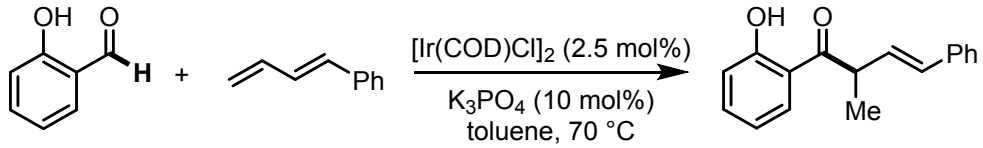
2.1 General procedure A: reaction between salicylaldehyde 1a and 1-phenyl-1,3-butadiene 2a.



1a **2a** **3a**

In glove box, a 4-mL vial charged with a stir bar was added $[Ir(COD)Cl]_2$ (0.01 mmol, 6.7 mg) and 0.2 mL of toluene, the mixture was stirred for 5 minutes before **1a** (0.2 mmol, 20.9 μL), K_3PO_4 (0.02 mmol, 4.2 mg), **2a** (0.3 mmol, 42 μL) were subsequently added. The vial was tightly capped, removed from glove box and heated at 70 ℃ for 36 h. After completion, the reaction mixture was cooled to room temperature, subjected to flash column chromatography (eluent: PE/Et_2O = 150:1) to get the pure product **3a**.

2.2 Experimental procedure for the gram-scale synthesis of 3a.



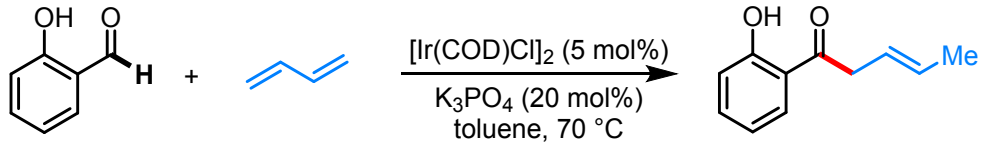
1a
1.0 g

2a

3a, 74%, rr 16:1
1.52 g

In glove box, a 75-mL pressure vessel with a stir bar was added $[Ir(COD)Cl]_2$ (0.205 mmol, 138 mg) and 8 mL of toluene, the mixture was stirred for 5 minutes before **1a** (8.2 mmol, 1.0 g), K_3PO_4 (0.82 mmol, 174 mg), **2a** (12.3 mmol, 1.6 g) and 8 mL of toluene were subsequently added. The vessel was tightly capped, removed from glove box and heated at 70 ℃ with stirring for 45 h. After completion, the reaction mixture was cooled to room temperature, directly subjected to flash column chromatography (eluent: PE/Et_2O = 150:1) to get the pure product **3a**.

2.3 General procedure B: reaction between salicylaldehyde 1a and 1,3-butadiene



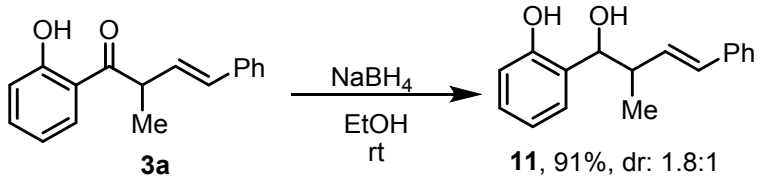
1a **5**

6, 87%, rr > 20:1

In glove box, a 4-mL vial charged with a stir bar was added $[Ir(COD)Cl]_2$ (0.01 mmol, 6.7 mg) and 0.2 mL of toluene, the mixture was stirred at room temperature for 5

minutes before **1a** (0.2 mmol, 20.9 μL), K_3PO_4 (0.04 mmol, 8.5 mg), 1, 3-butadiene (1.0 mmol, 2.0 M in THF, 5 equiv) and 0.2 mL of toluene. The vial was tightly capped, removed from glove box and heated at 70 ℃ for 6 h. After completion, the reaction mixture was cooled to room temperature, subjected to flash column chromatography (eluent: PE/Et_2O = 150:1) to get the pure product **6**.

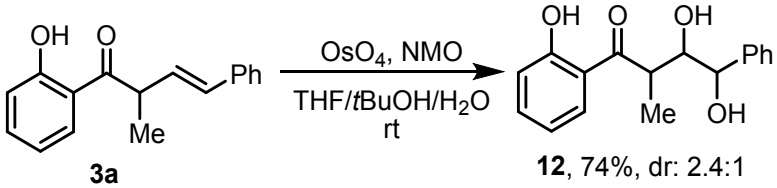
2.4 Procedure for the synthesis of compound 11.



3a **11**, 91%, dr: 1.8:1

A 25 mL three-necked flask with a stirrer was charged with **3a** (0.2 mmol, 40 mg, 1.0 equiv) and absolute EtOH (0.01M). The mixture was stirred at room temperature under N_2 atmosphere to result a clear solution, after which $NaBH_4$ (0.22 mmol, 6.6 mg, 1.1 equiv) was added in portions, the reaction was monitored by TLC. After stirring for 5 h the reaction mixture was concentrated in vacuo and the residue was dissolved in CH_2Cl_2. The organic layer was subsequently washed with distilled water for three times, the combined organic phase was dried over $MgSO_4$ and concentrated in vacuo. Purification of the crude product by column chromatography gave compound **11** in 91% isolated yield with a 1.8:1 dr.

2.5 Procedure for the synthesis of compound 12.

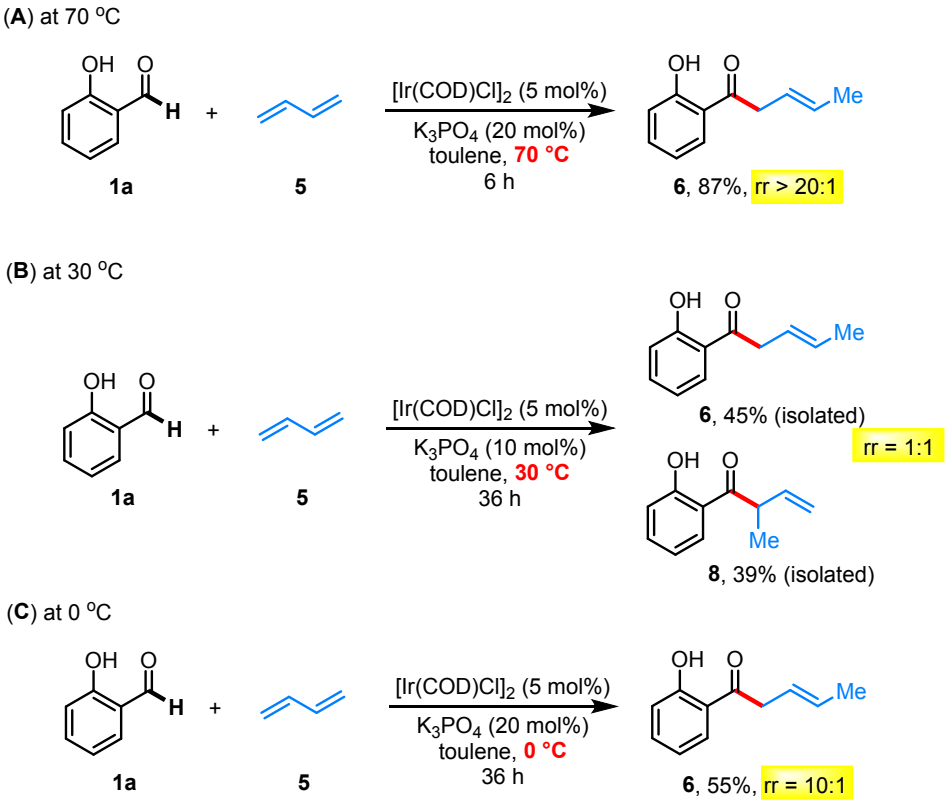


3a **12**, 74%, dr: 2.4:1

A 10 mL reaction tube charged with a stirrer, **3a** (0.2 mmol, 50.4 mg, 1.0 equiv) and NMO (0.22 mmol, 25.7 mg, 1.1 equiv) were added 0.25 mL of the mixed solvent (THF: *t*-BuOH: H_2O = 5:1:1). OsO_4 (0.002 mmol, 20 μL, 1 mol%, 2.5% in *t*-BuOH) was then added and the reaction mixture was stirred at room temperature for 12 h. The reaction was monitored by TLC. The final mixture was quench with 10% aqueous Na_2SO_3 and

H_2O, extracted with EtOAc (3×10 mL). The combined organic phase was washed with brine, dried over anhydrous Na_2SO_4, and concentrated in vacuum to give an oily crude product. Purification of the crude product by column chromatography (PE: EA=20:1) gave compound **12** as a 3:1 diastereoselective mixture in 74% yield.

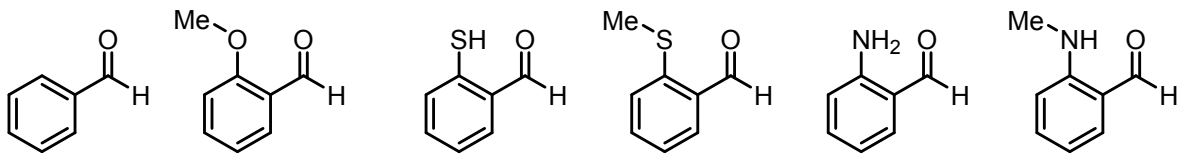
2.6 The reaction between 1a and butadiene 5 at different temperatures



2.6 Unsuccessful substrates for this transformation

Under standard reaction conditions, the following aldehyde substrates or substituted 1,3-dienes failed to give the desired hydroacylation products.

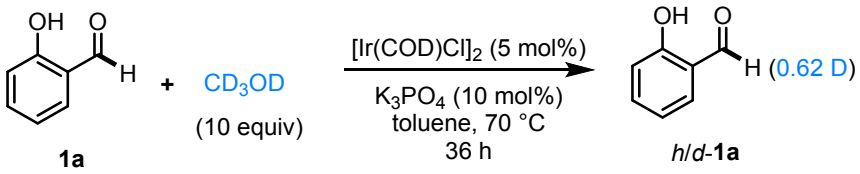
Unreactive aldehydes:



Unreactive substituted 1,3-dienes

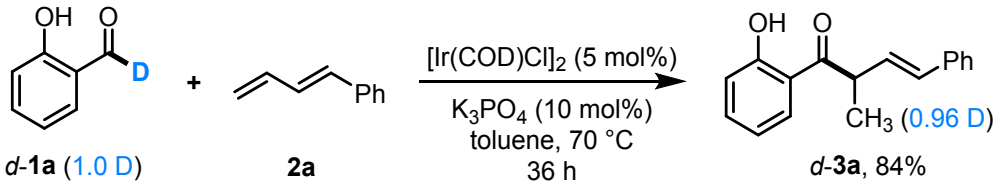
3. Deuterium Experiments and KIE analysis

a) deuterium scrambling experiment with CD_3OD

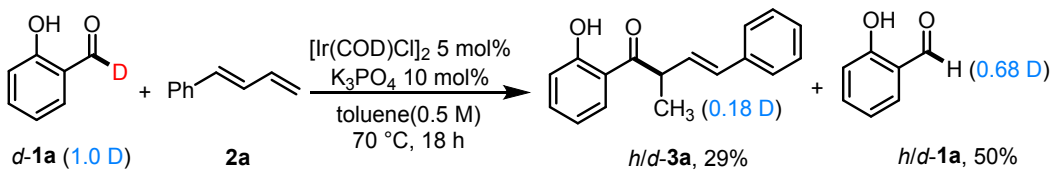


Under N_2 atmosphere, salicylaldehyde (244 mg, 2 mmol, 1.0 equiv.), $[Ir(COD)Cl]_2$ (67 mg, 0.1 mmol, 5 mol%) and K_3PO_4 (42 mg, 0.2 mmol, 10 mol%) were combined in toluene (0.5 mL) and methanol-d_4 (0.5 mL). The mixture was stirred at 70 °C for 36 hours and passed through a plug of silica (elution with DCM). Purification by flash column chromatography furnished *h*/*d*-**1a** as a yellow oil (42%). The deuterium content for the aldehydic C–H was determined by ^{1}HNMR to be 62%.

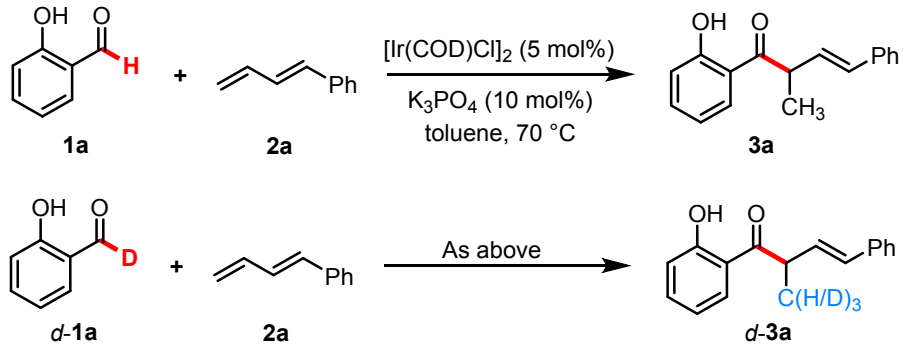
b) deuterium exchange experiment between *d*-1a and 2a (full conversion)



c) deuterium exchange experiment between *d*-1a and 2a (partial conversion)



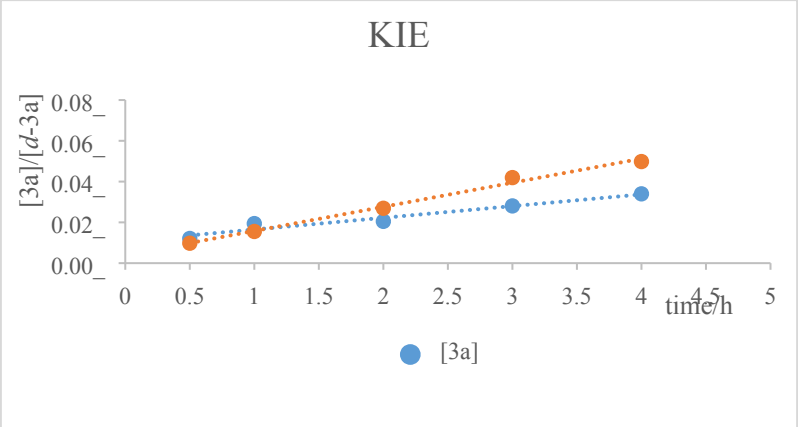
d) Side-by-side experiment of 1a and *d*-1a for KIE analysis



1a and *d*-**1a** was used for the side-by-side experiments under standard reaction conditions, dodecane was used as the internal standard. For each set, 5 samples were picked from 0.5 h to 4 h after the start of the reaction. The data related to the concentration of the product was monitored and measured by GC (Table S1).

Table S1: concentration of 3a and *d*-3a at each time spot from 0.5 h to 4 h

time/h	[3a]	[*d*-3a]
0.5	0.0121	0.0098
1	0.0194	0.0155
2	0.0205	0.0269
3	0.0281	0.0420
4	0.0340	0.0498

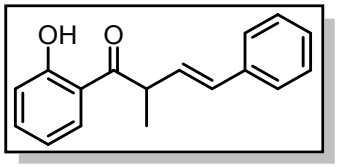


Conversion of the substrate at each time spot was also monitored and measured by GC by using dodecane as the internal standard (Table S2).

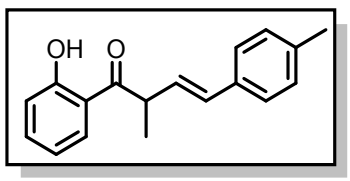
Table S2: conversions of 1a and *d*-1a at each time spot from 0.5 h to 4 h

time/h	**1a** (%)	***d*-1a** (%)
0.5	1.2%	1.2%
1	3.1%	5.5%
2	3.9%	7.0%
3	6.7%	9.3%
4	7.8%	11.4%

4. Characterization Data

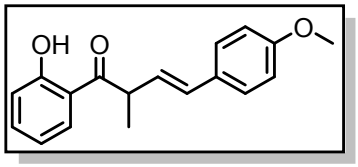


(*E*)-1-(2-hydroxyphenyl)-2-methyl-4-phenylbut-3-en-1-one (3a): Synthesized from **1a** and **2a** by following general procedure A on a 0.2 mmol scale. 94% isolate yield. Light yellow oil. R_f= 0.3 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.39 (s, 1H), 7.88 (d, J = 8.0 Hz, 1H), 7.46 (t, J = 8.0 Hz, 1H), 7.35 (s, 2H), 7.29 (t, J = 4.0 Hz, 2H), 7.25 – 7.15 (m, 1H), 6.99 (d, J = 4.0 Hz, 1H), 6.90 (t, J = 8.0 Hz, 1H), 6.54 (d, J = 16.0 Hz, 1H), 6.35 (dd, J = 12.0, 8.0 Hz, 1H), 4.35 (p, J = 7.4 Hz, 1H), 1.48 – 1.38 (d, J = 8.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.3, 163.2, 136.7, 136.5, 132.0, 130.1, 129.1, 128.6, 127.7, 126.3, 119.0, 118.8, 118.5, 44.3, 17.8. **IR (v/cm^{-1}):** 3650, 2179, 2016, 1795, 1264, 731, 702. **HRMS (EI)** calcd. $C_{17}H_{16}O_2$ $[M]^+$: 252.1150. Found: 252.1146.



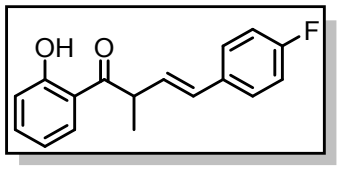
(*E*)-1-(2-hydroxyphenyl)-2-methyl-4-(p-tolyl) but-3-en-1-one (3b): Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl)-4-methylbenzene by following general procedure A on a 0.2 mmol scale. 93% isolate yield. Yellow liquid. R_f = 0.5 (PE: EA = 40:1). ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.88 (s, 1H). 8.00 (d, J = 8.0 Hz, 1H), 7.52

(t, J = 8.0 Hz, 1H), 7.28 (d, J = 8.2 Hz, 2H), 7.11 (d, J = 8.3 Hz, 2H), 6.98 (d, J = 8.0 Hz, 2H), 6.55 (d, J = 4.0 Hz, 1H), 6.32 (dd, J = 16.0, 8.0 Hz, 1H). 4.57 (q, J = 7.2 Hz, 1H), 2.26 (s, 1H), 1.32 (d, J = 6.9 Hz, 1H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 206.8, 160.9, 136.8, 136.0, 133.7, 131.1, 130.8, 129.1, 128.5, 126.0, 119.7, 119.2, 117.8, 44.2, 20.7, 17.1. **IR (v/cm^{-1}):** 3354, 2948, 2829, 1448, 1421, 1021, 684. **HRMS (EI)** calcd. $C_{18}H_{18}O_2$ [M] $^+$: 266.1307. Found: 266.1304.



(*E*)-1-(2-hydroxyphenyl)-4-(4-methoxyphenyl)-2-methylbut-3-en-1-one (3c):

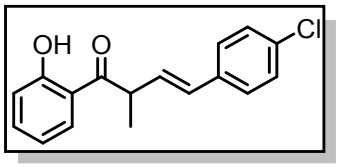
Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl)-4-methoxybenzene by following general procedure A on a 0.2 mmol scale. 58% isolate yield. Light yellow oil. R_f = 0.5 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.41 (s, 1H), 7.89 (d, J = 8.0 Hz, 1H), 7.46 (t, J = 7.6 Hz, 1H), 7.28 (d, J = 8.0 Hz, 2H), 6.99 (d, J = 8.4 Hz, 1H), 6.90 (t, J = 7.6 Hz, 1H), 6.82 (d, J = 7.6 Hz, 2H), 6.48 (d, J = 16.0 Hz, 1H), 6.20 (dd, J = 8.0, 8.0, 1H), 4.33 (m, 1H), 3.79 (s, 3H), 1.43 (d, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.4, 163.2, 159.3, 136.4, 131.4, 130.1, 129.5, 127.5, 126.9, 118.9, 118.7, 118.52, 114.0, 55.3, 44.2, 17.8. **IR (v/cm^{-1}):** 3324, 2948, 2829, 1688, 1448, 1412, 1020, 971. **HRMS (EI)** calcd. $C_{18}H_{18}O_3$ [M] $^+$: 282.1256. Found: 282.1254.



(*E*)-4-(4-fluorophenyl)-1-(2-hydroxyphenyl)-2-methylbut-3-en-1-one (3d):

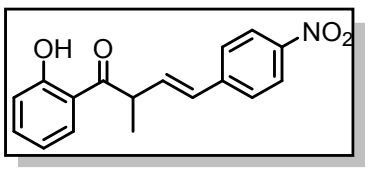
Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl)-4-fluorobenzene by following general procedure A on a 0.2 mmol scale. 94% isolate yield. Light yellow oil. R_f = 0.4 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.38 (s, 1H), 7.87 (d, J = 8.0 Hz, 1H), 7.47 (t, J = 8.0 Hz, 1H), 7.37 – 7.27 (m, 2H), 7.04 – 6.95 (m, 3H), 6.91 (t, J = 7.8 Hz,

1H), 6.50 (d, J = 16.0 Hz, 1H), 6.28 (dd, J = 8.4, 8.0 Hz, 1H), 4.35 (m, 1H), 1.44 (d, J = 5.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.2, 163.5 (d, $^1J_{C\text{-}F}$ = 247.5 Hz), 136.5,132.8 (d, $^4J_{C\text{-}F}$ = 3.0 Hz) 130.8, 130.0, 128.8 (d, $^3J_{C\text{-}F}$ = 3.0 Hz), 127.9, 127.8 (d, $^3J_{C\text{-}F}$ = 7.1 Hz), 119.0, 118.8 (d, $^2J_{C\text{-}F}$ = 14.1 Hz), 118.4, 115.6, 115.4 (d, $^2J_{C\text{-}F}$ = 22.2 Hz), 44.1, 17.8. ^{19}F NMR (376 MHz, $CDCl_3$) δ -114.2. **IR (ν/cm^{-1}):** 3199, 2944, 2830, 1673, 1449, 1026, 762, 722, 700. **HRMS (EI)** calcd. $C_{17}H_{15}FO_2$ [M] $^+$: 270.1056. Found: 270.1054.



(*E*)-4-(4-chlorophenyl)-1-(2-hydroxyphenyl)-2-methylbut-3-en-1-one (3e):

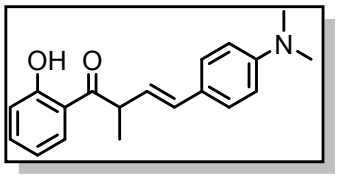
Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl)-4-chlorobenzene by following general procedure A on a 0.2 mmol scale. 82% isolate yield. Colorless oil. R_f = 0.3 (PE: EA= 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.34 (s, 1H), 7.87 (d, J = 8.4 Hz, 1H), 7.48 (t, J = 8.0 Hz, 1H), 7.00 (d, J = 8.4 Hz, 1H), 6.91 (t, J = 8.0 Hz, 1H), 6.49 (d, J = 15.6 Hz, 1H), 6.35 (dd, J =7.6, 8.4 Hz, 1H), 4.35 (m, 1H), 1.44 (d, J = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.1, 163.3, 136.6, 135.2, 133.3, 130.7, 130.0, 129.8, 128.7, 127.5, 119.0, 118.8, 118.4, 44.1, 17.8. **IR (ν/cm^{-1}):** 3414, 2329, 2164, 1689, 1419, 1264, 895, 735, 703. **HRMS (ESI)** calcd. $C_{17}H_{15}ClO_2$ [M] $^+$: 286.0761, 288.0731. Found: 286.0762, 288.0734.



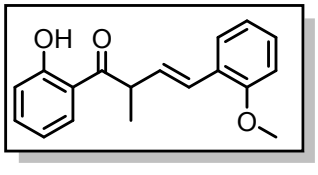
(*E*)-1-(2-hydroxyphenyl)-2-methyl-4-(4-nitrophenyl) but-3-en-1-one (3f):

Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl)-4-nitrobenzene by following general procedure A on a 0.2 mmol scale. 86% isolate yield. Yellow oil. R_f = 0.1 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.30 (s, 1H), 8.15 (d, J = 8.4, 2.6 Hz, 2H),

7.87 (d, J = 8.0 Hz, 1H), 7.49 (d, J = 8.0 Hz, 3H), 7.02 (d, J = 8.2 Hz, 1H), 6.94 (t, J = 8.0 Hz, 1H), 6.61 (t, J = 3.2 Hz, 2H), 4.43 (m, 1H), 1.49 (d, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.6, 163.3, 147.0, 143.1, 136.8, 134.1, 129.9, 129.9, 126.9, 124.0, 119.1, 118.9, 118.3, 44.2, 17.9. **IR (ν/cm^{-1}):** 3203, 2941, 2834, 1663, 1449, 1413, 1114, 1018, 700. **HRMS (EI)** calcd. $C_{17}H_{15}NO_4$ [M]$^+$: 297.1001. Found: 297.1004.

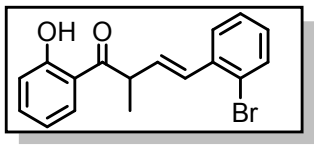


(*E*)-4-(4-(dimethylamino) phenyl)-1-(2-hydroxyphenyl)-2-methylbut-3-en-1-one (3g): Synthesized from **1a** and (*E*)-4-(buta-1, 3-dien-1-yl)-N, N-dimethylaniline following general procedure A on a 0.2 mmol scale. 23% isolate yield. Light yellow oil. R_f = 0.3 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.44 (s, 1H), 7.90 (d, J = 8.4 Hz, 1H), 7.45 (t, J = 8.0 Hz, 1H), 7.35 (d, J = 8.0 Hz 2H), 6.98 (d, J = 8.4 Hz, 1H), 6.89 (t, J = 7.6 Hz, 1H), 6.64 (d, J = 8.0 Hz, 2H), 6.45 (d, J = 16.0 Hz, 1H), 6.10 (dd, J = 8.0, 7.6 Hz, 1H), 4.35-4.27 (m, 1H), 2.94 (s, 6H), 1.41 (d, J = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.6, 163.2, 150.2, 136.3, 131.9, 130.2, 127.2, 125.2, 124.7, 118.8, 118.7, 118.6, 112.4, 44.4, 40.5, 17.7. **IR (ν/cm^{-1}):** 3404, 2941, 2833, 1641, 1533, 1437, 1089, 1015, 708. **HRMS (EI)** calcd. $C_{19}H_{21}NO_2$ [M]$^+$: 295.1572. Found: 295.1570.



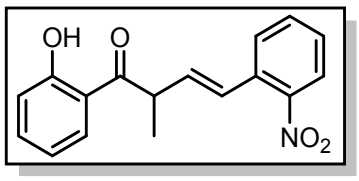
(*E*)-1-(2-hydroxyphenyl)-4-(2-methoxyphenyl)-2-methylbut-3-en-1-one (3h):
Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl)-2-methylbenzene by following general procedure A on a 0.2 mmol scale. 84% isolate yield. Colorless oil. R_f = 0.5 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.42 (d, J = 2.5 Hz, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.46 (t, J = 8.0 Hz, 1H), 7.40 (d, J = 7.6 Hz, 1H), 7.20 (t, J = 7.6 Hz, 1H), 6.99

(d, J = 8.4 Hz, 1H), 6.95 – 6.79 (m, 4H), 6.35 (dd, J = 8.4, 8.0 Hz, 1H), 4.38 (m, 1H), 3.84 (s, 3H), 1.45 (d, J = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.5, 163.2, 156.6, 136.3, 130.2, 129.6, 128.8, 126.9, 126.7, 125.7, 120.7, 118.9, 118.7, 118.5, 110.9, 77.3, 77.0, 76.7, 55.5, 44.9, 17.9. **IR (ν/cm^{-1}):** 3242, 1633, 1607, 1511.50, 1264, 1175, 1158, 1033, 975, 820, 732. **HRMS (EI)** calcd. $C_{18}H_{18}O_3$ $[M]^+$: 282.1256. Found: 282.1259.



(*E*)-4-(2-bromophenyl)-1-(2-hydroxyphenyl)-2-methylbut-3-en-1-one (3i):

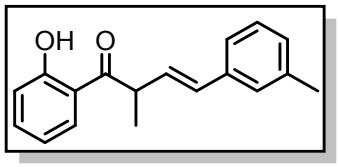
Synthesized from **1a** and (*E*)-1-bromo-2-(buta-1, 3-dien-1-yl)-benzene by following general procedure A on a 0.2 mmol scale. 78% isolate yield. Gray solid. R_f = 0.4 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.35 (s, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.53-7.48 (m, 3H), 7.23 (d, J = 7.6 Hz, 1H), 7.08 (t, J = 8.0 Hz, 1H), 7.00 (d, J = 8.8 Hz, 1H), 6.96 – 6.85 (m, 2H), 6.29 (dd, J = 8.0, 8.0 Hz, 1H), 4.43 (m, 1H), 1.47 (d, J = 6.8 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 207.0, 163.2, 136.6, 132.9, 132.2, 131.0, 130.2, 129.0, 127.5, 127.1, 123.5, 119.0, 118.8, 118.4, 44.6, 17.9. **IR (ν/cm^{-1}):** 3456, 2984, 2212, 2014, 1988, 1635, 1487, 1264, 1157, 1056, 895, 734, 703. **HRMS (ESI)** calcd. $C_{17}H_{15}BrO_2$ $[M-H]^+$: 329.0183, 331.0126. Found: 329.0190, 331.0168.



(*E*)-1-(2-hydroxyphenyl)-2-methyl-4-(2-nitrophenyl) but-3-en-1-one (3j):

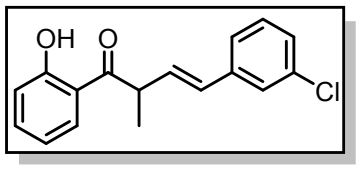
Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl)-2-nitrobenzene by following general procedure A on a 0.2 mmol scale. Contains 15% of the non-isolated linear-selective product. 41% isolate yield after correlation. Light yellow oil. R_f = 0.2 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.30 (s, 1H), 7.93 (t, J = 8.8 Hz, 2H), 7.56 (d, J = 8.0 Hz, 2H), 7.50 (t, J = 8.8 Hz, 1H), 7.39 (t, J = 8.0 Hz, 1H), 7.10 (d, J = 15.6 Hz,

1H), 7.01 (d, J = 8.8 Hz, 1H), 6.95 (t, J = 8.4 Hz, 1H), 6.33 (q, J = 9.2 Hz, 1H), 4.45 (m, 1H), 1.46 (d, J = 6.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.7, 163.2, 136.7, 134.5, 133.2, 130.1, 128.8, 128.3, 127.7, 127.6, 124.6, 119.1, 118.8, 44.7, 18.0. **IR (ν/cm^{-1}):** 3153, 2978, 2834, 1653, 1449, 1413, 1113, 1018, 700. **HRMS (EI)** calcd. $C_{17}H_{15}NO_4$ $[M]^+$: 297.1001. Found: 297.0999.



(*E*)-1-(2-hydroxyphenyl)-2-methyl-4-(*m*-tolyl) but-3-en-1-one (3k):

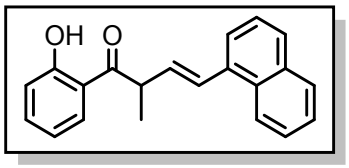
Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl)-3-methylbenzene by following general procedure A on a 0.2 mmol scale. 80% isolate yield. Colorless oil. R_f = 0.5 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.38 (s, 1H), 7.87 (d, J = 8.4 Hz, 1H), 7.45 (t, J = 9.6 Hz, 1H), 7.11-7.18 (m, 3H), 7.00 (dd, J = 7.2, 8.8 Hz, 2H), 6.89 (t, J = 8.4 Hz, 1H), 4.34 (q, J = 7.2 Hz, 1H), 2.30 (s, 3H), 1.42 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.3, 163.2, 138.2, 136.6, 136.4, 132.1, 130.1, 128.9, 128.5, 128.5, 127.0, 123.5, 118.9, 118.8, 118.5, 44.3, 21.4, 17.8. **IR (ν/cm^{-1}):** 3265, 2947, 1649, 1448, 1112, 949, 735, 700. **HRMS (EI)** calcd. $C_{18}H_{18}O_2$ $[M]^+$: 266.1307. Found: 266.1304.



(*E*)-4-(3-chlorophenyl)-1-(2-hydroxyphenyl)-2-methylbut-3-en-1-one (3l):

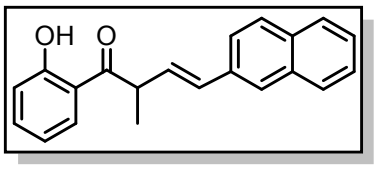
Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl)-3-chlorobenzene by following general procedure A on a 0.2 mmol scale. 90% isolate yield. Yellow solid. R_f= 0.3 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.34 (s, 1H), 7.86 (d, J = 8.4 Hz, 1H), 7.48 (t, J = 8.0 Hz, 1H), 7.34 (s, 1H), 7.22 (m, 3H), 7.01 (d, J = 8.4 Hz, 1H), 6.93 (t, J = 8.0 Hz, 1H), 6.48 (d, J = 5.6 Hz, 1H), 6.39 (dd, J = 7.6, 8.4 Hz, 1H), 4.36 (m, 1H), 1.45 (d,

J = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.9, 163.3, 138.5, 136.6, 134.5, 130.7, 130.6, 130.0, 129.8, 127.6, 126.3, 124.5, 119.0, 118.9, 118.4, 44.1, 17.8. **IR (ν/cm^{-1}):** 3344, 2978, 2211, 2165, 1981, 1264, 736, 704. **HRMS (ESI)** calcd. $C_{17}H_{15}ClO_2$ [M]$^+$: 286.0761, 288.0731. Found: 286.0757, 288.0734.



(*E*)-1-(2-hydroxyphenyl)-2-methyl-4-(naphthalen-1-yl)but-3-en-1-one (3m):

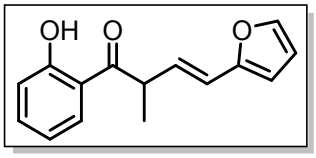
Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl) naphthalene by following general procedure A on a 0.2 mmol scale. 55% isolate yield. Colorless oil. R_f = 0.2 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.42 (s, 1H), 8.06 – 7.95 (m, 2H), 7.87 – 7.81 (m, 1H), 7.77 (d, J = 8.0 Hz, 1H), 7.55 (d, J = 6.8, 1.1 Hz, 1H), 7.53 – 7.45 (m, 3H), 7.42 (t, J = 7.6 Hz, 1H), 7.30 (d, J = 15.6 Hz, 1H), 7.03 (dd, J=1.6, 6.8 Hz, 1H), 6.95 (dt, J = 1.2, 6.8 Hz 1H), 6.40 (dd, J = 7.6, 8.0 Hz, 1H), 4.57 – 4.45 (m, 1H), 1.53 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.2, 163.3, 136.5, 133.6, 132.7, 132.4, 130.2, 129.4, 128.6, 128.1, 126.1, 125.8, 125.6, 124.0, 123.6, 119.0, 118.8, 118.5, 44.7, 17.8. **IR (ν/cm^{-1}):** 3277, 2947, 2831, 1657, 1447, 1410, 1113, 1020, 730. **HRMS (EI)** calcd. $C_{21}H_{18}O_2$ [M]$^+$: 302.1307. Found: 302.1305.



(*E*)-1-(2-hydroxyphenyl)-2-methyl-4-(naphthalen-2-yl)but-3-en-1-one (3n):

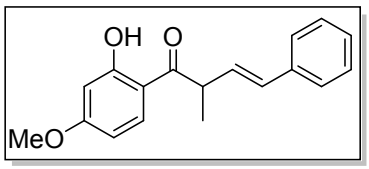
Synthesized from **1a** and (*E*)-2-(buta-1, 3-dien-1-yl) naphthalene by following general procedure A on a 0.2 mmol scale. 77% isolate yield. Colorless oil. R_f = 0.3 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.41 (s, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.77 (d, J = 7.2 Hz, 3H), 7.70 (s, 1H), 7.56 (d, J = 8.6 Hz, 1H), 7.52 – 7.37 (m, 3H), 7.01 (d, J = 8.4 Hz, 1H), 6.92 (t, J = 7.6 Hz, 1H), 6.70 (d, J = 16.0 Hz, 1H), 6.48 (dd, J = 8.0, 8.0

Hz, 1H), 4.41 (m, 1H), 1.48 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.3, 163.3, 136.5, 134.1, 133.5, 133.1, 132.1, 130.1, 129.5, 128.2, 128.0, 127.7, 126.3, 125.9, 123.4, 119.0, 118.8, 118.5, 44.3, 17.9. **IR (ν/cm^{-1}):** 3244, 2947, 2831, 1685, 1447, 1410, 1123, 1020, 703. **HRMS (EI)** calcd. $C_{21}H_{18}O_2$ [M] $^+$: 302.1307. Found: 302.1304.



(*E*)-4-(furan-2-yl)-1-(2-hydroxyphenyl)-2-methylbut-3-en-1-one (3o):

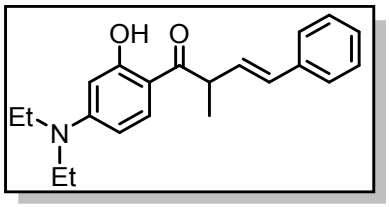
Synthesized from **1a** and (*E*)-2-(buta-1, 3-dien-1-yl)furanby following general procedure A on a 0.2 mmol scale. 70% isolate yield. Yellow solid. R_f=0.4 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.36 (s, 1H), 7.85 (dd, J = 2.0, 6.0 Hz, 1H), 7.47 (dt, J = 2.0, 5.2, 1.6 Hz, 1H), 7.32 (d, J = 2.0 Hz, 1H), 6.99 (d, J = 8.4 Hz, 1H), 6.93–6.88 (m, 1H), 6.35–6.31 (m, 3H), 6.21 (d, J = 3.6 Hz, 1H), 4.28-4.35 (m, 1H), 1.43 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.86, 163.21, 152.18, 142.01, 136.45, 130.05, 127.63, 120.31, 118.94, 118.78, 118.46, 111.28, 108.00, 43.74, 17.35. **IR (ν/cm^{-1}):** 3283, 2943, 2831, 1658, 1452, 1406, 1120, 1020, 706. **HRMS (EI)** calcd. $C_{15}H_{14}O_3$ [M] $^+$: 242.0943. Found: 242.0942.



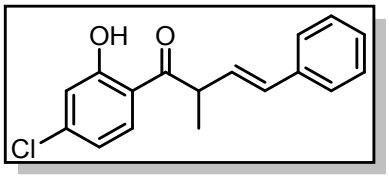
(*E*)-1-(2-hydroxy-4-methoxyphenyl)-2-methyl-4-phenylbut-3-en-1-one (4a):

Synthesized from **1a** and (*E*)-1-(buta-1, 3-dien-1-yl)-3-methylbenzene by following general procedure A on a 0.2 mmol scale. 80% isolate yield. Colorless oil. R_f = 0.5 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.38 (s, 1H), 7.87 (d, J = 8.4 Hz, 1H), 7.45 (t, J = 9.6 Hz, 1H), 7.11-7.18 (m, 3H), 7.00 (dd, J = 7.2, 8.8 Hz, 2H), 6.89 (t, J = 8.4 Hz, 1H), 4.34 (q, J = 7.2 Hz, 1H), 2.30 (s, 3H), 1.42 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.3, 163.2, 138.2, 136.6, 136.4, 132.1, 130.1, 128.9, 128.5,

128.5, 127.0, 123.5, 118.9, 118.8, 118.5, 44.3, 21.4, 17.8. **IR (ν/cm^{-1}):** 3265, 2947, 1649, 1448, 1112, 949, 735, 700. **HRMS (EI)** calcd. $C_{18}H_{18}O_2$ [M] $^+$: 266.1307. Found: 266.1304.

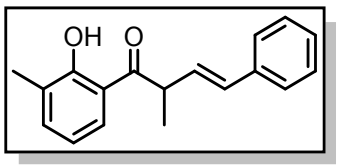


(*E*)-1-(4-(dimethylamino)-2-hydroxyphenyl)-2-methyl-4-phenylbut-3-en-1-one (4b): Synthesized from 4-(diethylamino)-2-hydroxybenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 90% isolate yield. Light yellow oil. R_f = 0.2 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.06 (s, 1H), 7.67 (d, J = 11.2 Hz, 1H), 7.42-7.15 (m, 5H), 6.52 (d, J =16.4 Hz, 1H), 6.37 (dd, J = 8.0, 8.0 Hz, 1H), 6.19 (d, J = 9.2 Hz, 1H), 6.09 (s, 1H), 4.26-4.13 (m, 1H), 3.39 (q, J = 9.2 Hz, 4H), 1.40 (d, J = 5.2 Hz, 3H), 1.20 (t, J = 8.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.0, 165.9, 153.7, 137.0, 131.9, 131.0, 130.5, 128.5, 127.4, 126.3, 108.5, 103.7, 97.4, 44.6, 43.4, 17.9, 12.7. **IR (ν/cm^{-1}):** 3235, 2988, 1626, 1523, 1355, 1264, 1237, 1141, 987, 735. **HRMS (EI)** calcd. $C_{19}H_{21}NO_2$ [M] $^+$: 323.1885. Found: 323.1885.



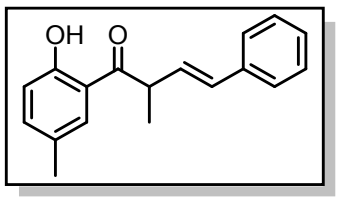
(*E*)-1-(4-chloro-2-hydroxyphenyl)-2-methyl-4-phenylbut-3-en-1-one (4c):
Synthesized from 4-chloro-2-hydroxybenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 73% isolate yield. Light yellow oil. R_f = 0.3 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.51 (s, 1H), 7.80 (d, J = 8.4 Hz, 1H), 7.33 (d, J = 8.4 Hz, 4H), 7.40-7.14 (m, 5H), 7.01 (s, 1H), 6.88 (d, J = 8.0 Hz, 1H), 6.53 (d, J = 16.0 Hz, 1H), 6.32 (dd, J = 8.4, 8.4 Hz, 1H), 4.35-4.20 (m, 1H), 1.42 (d, J = 4.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.5, 163.9, 142.2, 136.5, 132.3, 131.1, 128.7,

128.6, 127.9, 126.3, 119.6, 118.8, 117.1, 44.5, 17.7. **IR (ν/cm^{-1}):** 3295, 2988, 1633, 1608, 1488, 1264, 1189, 1081, 975, 736. **HRMS (EI)** calcd. $C_{17}H_{15}ClO_2$ [M] $^+$: 286.0761, 288.0731. Found: 286.0758, 288.0733.



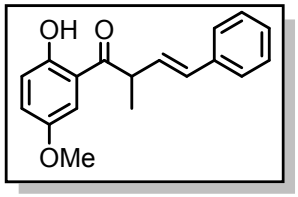
(*E*)-1-(2-hydroxy-3-methylphenyl)-2-methyl-4-phenylbut-3-en-1-one (4d):

Synthesized from 2-hydroxy-3-methylbenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 76% isolate yield. Colorless oil. R_f = 0.4 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.71 (s, 1H), 7.74 (d, J = 8.4 Hz, 1H), 7.34-7.19 (m, 6H), 6.80 (t, J = 4.4, 1H), 6.53 (d, J = 16.0 Hz, 1H), 6.36 (dd, J =8.0, 8.0 Hz, 1H), 4.39-4.32 (m, 1H), 2.26 (s, 3H), 1.43 (d, J = 2.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.5, 161.7, 137.2,136.7 131.8, 129.3, 128.6, 127.8, 127.7, 126.3, 118.3,117.7 44.2, 17.8, 15.6. **IR (ν/cm^{-1}):** 3335, 2985, 1629, 1453, 1264, 1047, 985, 734. **HRMS (EI)** calcd. $C_{18}H_{18}O_2$ [M] $^+$: 266.1307. Found: 266.1308.



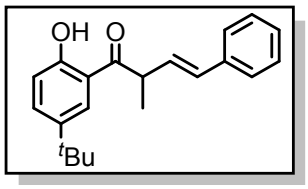
(*E*)-1-(2-hydroxy-5-methylphenyl)-2-methyl-4-phenylbut-3-en-1-one (4e):

Synthesized from 2-hydroxy-5-methylbenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 77% isolate yield. Light yellow oil. R_f = 0.5 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.23 (s, 1H), 7.65 (s, 1H), 7.36-7.18 (m, 3H), 6.90 (d, J = 8.8 Hz 1H), 6.53 (d, J = 16.8, 1H), 6.37 (dd, J = 8.0, 7.6 Hz, 1H), 4.32-4.46 (m, 1H), 2.32 (s, 3H), 1.44 (d, J = 5.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.2, 161.2, 137.6, 136.8, 131.8, 129.7, 129.3, 128.6, 128.0, 127.7, 126.3, 118.5, 118.1, 44.0, 20.7, 17.9. **IR (ν/cm^{-1}):** 3364, 1636, 1483, 1288, 1247, 1177, 979, 736. **HRMS (EI)** calcd. $C_{18}H_{18}O_2$ [M] $^+$: 266.1307. Found: 266.1305.



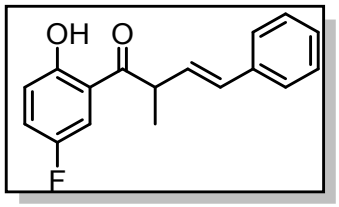
(*E*)-1-(2-hydroxy-5-methoxyphenyl)-2-methyl-4-phenylbut-3-en-1-one (4f):

Synthesized from 2-hydroxy-5-methoxybenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 95% isolate yield. Yellow oil. R_f = 0.3 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.95 (s, 1H), 7.35-7.15 (m, 5H), 7.08 (d, J = 9.2 Hz, 1H), 6.91 (d, J = 8.8 Hz, 1H), 6.52 (d, J = 16.0 Hz, 1H), 6.32 (dd, J = 8.0, 8.0 Hz, 1H), 4.33-4.22 (m, 1H), 3.76 (s, 3H), 1.41 (d, J = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.7, 157.6, 151.7, 136.6, 132.1, 129.1, 128.6, 127.8, 126.3, 124.1, 119.5, 118.0, 113.1, 56.0, 44.5, 17.7. **IR (ν/cm^{-1}):** 3342, 2986, 1638, 1614, 1486, 1264, 1224, 1175, 1038, 967, 833, 734. **HRMS (EI)** calcd. $C_{18}H_{18}O_3$ [M] $^+$: 282.1256. Found: 282.1253.



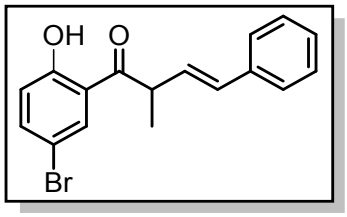
(*E*)-1-(5-(tert-butyl)-2-hydroxyphenyl)-2-methyl-4-phenylbut-3-en-1-one (4g):

Synthesized from 5-(tert-butyl)-2-hydroxybenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 71% isolate yield. Yellow liquid. R_f = 0.4 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.20 (s, 1H), 7.90 (d, J = 2.8 Hz, 1H), 7.53 (dd, J = 6.4, 2.4 Hz, 1H), 7.39 – 7.19 (m, 5H), 6.94 (d, J = 8.8 Hz, 1H), 6.59 (d, J = 16.0 Hz, 1H), 6.33 (dd, J = 16.0, 8.4 Hz, 1H), 4.43-4.33 (m, 1H), 1.45 (d, J = 6.4 Hz, 2H), 1.32 (s, 7H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.0, 161.0, 141.4, 136.7, 134.1, 132.1, 129.5, 128.6, 127.7, 126.3, 126.1, 118.3, 117.8, 44.6, 34.2, 31.3, 17.6. **IR (ν/cm^{-1}):** 3319, 2984, 2333, 1637, 1489, 1422, 1264, 1185, 895, 793. **HRMS (EI)** calcd. $C_{21}H_{24}O_2$ [M] $^+$: 308.1776. Found: 308.1772.



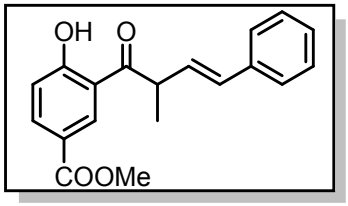
(*E*)-1-(5-fluoro-2-hydroxyphenyl)-2-methyl-4-phenylbut-3-en-1-one (4h):

Synthesized from 5-fluoro-2-hydroxybenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 51% isolate yield. Light yellow oil. R_f = 0.3 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.11 (s, 1H), 7.54 (d, J = 9.2, 1H), 7.38-7.15 (m, 5H), 6.97 (q, J =4.8 Hz, 1H), 6.55 (d, J = 16.0 Hz, 1H), 6.33 (dd, J = 7.6, 8.4 Hz, 1H), 4.28-4.21 (m, 1H), 1.44 (d, J = 6.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.3, 159.4, 154.8 (d, $^1J_{C\text{-}F}$ = 239.3 Hz), 136.5, 132.4, 128.6, 128.5 (d, $^3J_{C\text{-}F}$ = 16.2 Hz), 127.9, 126.3, 124.2 (d, $^2J_{C\text{-}F}$ = 24.2 Hz), 124.0, 120.1(d, $^3J_{C\text{-}F}$ = 7.1 Hz), 117.9 (d, $^4J_{C\text{-}F}$ = 5.1 Hz), 115.9 (d, $^2J_{C\text{-}F}$ =23.2 Hz), 114.8, 44.5, 17.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ -123.82. **IR (v/cm^{-1}):** 3054, 2987, 1662, 1481, 1421, 1264, 1192, 1168, 991, 895, 731. **HRMS (EI)** calcd. $C_{17}H_{15}FO_2$ [M] $^+$: 270.1056. Found: 270.1053.



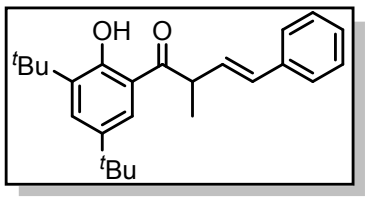
(*E*)-1-(3-bromophenyl)-2-methyl-4-phenylbut-3-en-1-one (4i):

Synthesized from 5-bromo-2-hydroxybenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 68% isolate yield. Gray solid. R_f = 0.4 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.29 (s, 1H), 7.97 (s, 1H), 7.54 (d, J = 8.8 Hz, 1H), 7.40-7.18 (m, 5H), 6.90 (d, J = 8.8 Hz, 1H), 6.54 (d, J = 16.4 Hz, 1H), 6.32 (dd, J = 8.0, 8.0 Hz, 1H), 4.35-4.20 (m, 1H), 1.44 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.4, 162.1, 139.1, 136.5, 132.4, 132.3, 128.6, 128.5, 127.9, 126.4, 120.8, 119.7, 110.5, 44.3, 17.8. **IR (v/cm^{-1}):** 3303, 2987, 1639, 1607, 1469, 1264, 1180, 979, 736. **HRMS (ESI)** calcd. $C_{17}H_{15}BrO_2$ [M-H] $^+$: 329.0183, 331.0126. Found: 329.0167, 331.0152.



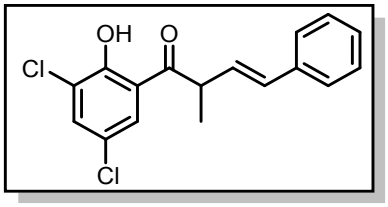
Methyl-(*E*)-4-hydroxy-3-(2-methyl-4-phenylbut-3-enoyl)-benzoate (4j):

Synthesized from methyl 3-formyl-4-hydroxybenzoate and **2a** following general procedure A on a 0.2 mmol scale. 38% isolate yield. Colorless liquid. R_f= 0.3 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.78 (s, 1H), 8.66 (d, J = 2.4 Hz, 1H), 8.12 (dd, J = 6.8, 2.0 Hz, 1H), 7.38 – 7.33 (m, 2H),7.32-7.27 (m, 2H), 7.25-7.20 (m, 1H) 7.03 (d, J = 8.8 Hz, 1H), 6.60 (d, J = 16.0 Hz, 1H), 6.34 (dd, J = 8.3, 7.6 Hz, 1H), 4.51-4.43 (m, 1H), 3.93 (s, 3H), 1.47 (d, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.3, 166.8, 166.0, 137.1, 136.6, 132.7, 132.5, 128.6, 128.5, 127.8, 126.4, 121.1, 119.0, 117.9, 52.2, 44.2, 17.7. **IR (ν/cm^{-1}):** 3262, 1717, 1637, 1438, 135, 1264, 1225, 1183, 1111, 969, 731. **HRMS (EI)** calcd. $C_{19}H_{18}O_4$ [M] $^+$: 310.1205. Found: 310.1208.



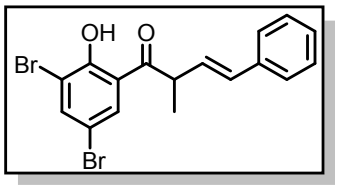
(*E*)-1-(3, 5-di-tert-butyl-2-hydroxyphenyl)-2-methyl-4-phenylbut-3-en-1-one (4k):

Synthesized from 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 88% isolate yield. Colorless liquid. R_f= 0.4 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.09 (s, 1H), 7.78 (d, J = 2.4 Hz, 1H), 7.55 (d, J = 2.5 Hz, 1H), 7.37 – 7.18 (m, 5H), 6.59 (d, J = 15.6 Hz, 1H), 6.34 (dd, J = 7.2, 8.4 Hz, 1H), 4.43-4.36 (m, 1H), 1.44 (d, J = 6.8 Hz, 3H), 1.42 (s, 9H), 1.32 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 213.7, 93.3, 131.9, 131.4, 129.8, 128.6, 127.6, 126.3, 124.0, 44.8, 35.2, 34.3, 31.4, 29.4, 17.8. **IR (ν/cm^{-1}):** 3219, 2879, 1664, 1435, 1264, 1167, 895, 735. **HRMS (EI)** calcd. $C_{25}H_{32}O_2$ [M] $^+$: 364.2402. Found: 364.2400.



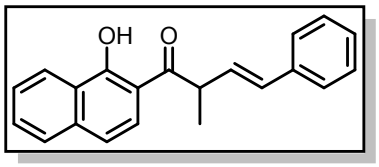
(*E*)-1-(3, 5-dichloro-2-hydroxyphenyl)-2-methyl-4-phenylbut-3-en-1-one (4l):

Synthesized from 3, 5-dichloro-2-hydroxybenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 63% isolate yield. White solid. R_f = 0.3 (PE: EA= 40:1). ^{1}H NMR (400 MHz, $CDCl_3$). δ12.84 (s, 1H), 7.76 (d, J = 2.4 Hz, 1H), 7.55 (d, J = 2.4 Hz, 1H), 7.38 – 7.20 (m, 5H), 6.53 (d, J = 16.0 Hz, 1H), 6.29 (dd, J = 8.0, 8.0 Hz, 1H), 4.35-4.20 (m, 1H), 1.43 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.4, 157.6, 136.3, 135.9, 132.8, 128.6, 128.0, 128.0, 127.9, 126.4, 123.4, 119.5, 44.6, 17.8. **IR (ν/cm^{-1}):** 3342, 2260, 1646, 1439, 1264, 1217, 1171, 968, 895, 736. **HRMS (EI)** calcd. $C_{17}H_{14}Cl_2O_2$ [M] $^+$: 320.0371, 322.0341. Found: 320.0376, 322.0313.



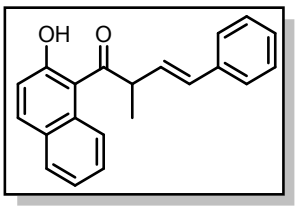
(*E*)-1-(3, 5-dibromo-2-hydroxyphenyl)-2-methyl-4-phenylbut-3-en-1-one (4m):

Synthesized from 3, 5-dibromo-2-hydroxybenzaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 44% isolate yield. Yellow solid. R_f = 0.2 (PE: EA = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.01 (s, 1H), 7.97 (s, 1H), 7.86 (s, 1H), 7.40 – 7.285 (m, 5H), 6.55 (d, J = 16.4 Hz, 1H), 6.30 (dd, J = 8.4, 8.0 Hz, 1H), 4.35-4.23 (m, 1H), 1.45 (m, J = 5.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.3, 158.8, 141.5, 136.3, 132.8, 131.6, 128.7, 128.0, 128.0, 126.4, 120.0, 113.6, 110.5, 44.5, 17.8. **IR (ν/cm^{-1}):** 3549, 2974, 1682, 1453, 1264, 896, 736. **HRMS (ESI)** calcd. $C_{17}H_{15}BrO_2$ [M-H] $^+$: 406.9288, 408.9267. Found: 406.9258, 408.9242.



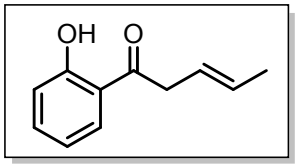
(*E*)-1-(1-hydroxynaphthalen-2-yl)-2-methyl-4-phenylbut-3-en-1-one (4n):

Synthesized from 1-hydroxy-2-naphthaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 83% isolate yield. Yellow oil. R_f= 0.4 (PE: EA = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.17 (s, 1H), 8.47 (d, J = 8.0 Hz, 1H), 7.79 (d, J = 8.8 Hz, 1H), 7.75 (d, J = 8.0 Hz, 1H), 7.62 (t, J = 6.8 Hz, 1H), 7.52 (t, J = 7.6 Hz, 1H), 7.40-7.15 (m, 5H), 6.58 (d, J = 16.0 Hz, 1H), 6.42 (dd, J = 8.4, 7.6 Hz, 1H), 4.46-4.43 (m, 1H), 1.50 (d, J = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.9, 163.5, 137.3, 136.8, 131.9, 130.2, 129.2, 128.6, 127.7, 127.4, 126.3, 126.0, 125.5, 124.5, 124.3, 118.4, 112.1, 76.8, 44.4, 17.7. **IR (v/cm^{-1}):** 3186, 1629, 1521, 1418, 1264, 1029, 895, 810, 731. **HRMS (EI)** calcd. $C_{21}H_{18}O_2$ [M]$^+$: 302.1307. Found: 302.1304.

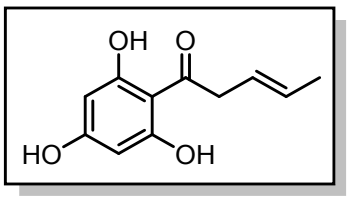


(*E*)-1-(2-hydroxynaphthalen-1-yl)-2-methyl-4-phenylbut-3-en-1-one (4o):

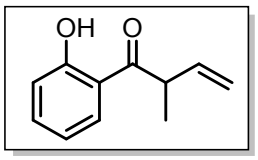
Synthesized from 2-hydroxy-1-naphthaldehyde and **2a** following general procedure A on a 0.2 mmol scale. 36% isolate yield. Light yellow oil. R_f= 0.4 (PE: EA = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.46 (d, J = 8.8 Hz, 1H), 7.92 (d, J = 8.4 Hz, 1H), 7.76 (d, J = 8.4 Hz, 2H), 7.47-7.23 (m, 6H), 7.10 (d, J = 9.2 Hz, 1H), 4.83 – 4.73 (m, 1H), 4.78 (s, 1H), 3.26 (t, J = 10.4 Hz, 1H), 2.99 (d, J = 14.0 Hz, 1H), 2.78-2.67 (m, 1H), 1.31 (d, J = 5.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.5, 162.9, 137.2, 136.8, 131.9, 129.5, 129.3, 129.2, 128.7, 128.4, 126.8, 125.8, 124.7, 118.7, 111.3, 81.1, 44.7, 36.7, 9.8. **IR (v/cm^{-1}):** 3124, 1667, 1597, 1513, 1461, 1436, 1264, 1235, 1128, 1090, 827, 732. **HRMS (EI)** calcd. $C_{21}H_{18}O_2$ [M]$^+$: 302.1307. Found: 302.1309.



(*E*)-1-(2-hydroxyphenyl) pent-3-en-1-one (6): Synthesized from **1a** and 1, 3-butdiene by following general procedure B on a 0.2 mmol scale, stirred for 6 hours at 70 ℃. 87% isolate yield. Colorless oil. R_f = 0.4 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.37 (s, 1H), 7.84 (d, J = 8.4 Hz, 1H), 7.47 (t, J = 7.2 Hz, 1H), 6.99 (d, 1H), 6.90 (t, J = 7.2 Hz, 1H) 6.08 – 5.93 (m, 1H), 5.26 – 5.14 (m, 2H), 4.27-4.13 (m, 1H), 1.36 (d, J = 8.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.2, 137.6, 136.4, 130.1, 118.8, 118.7, 118.5, 116.9, 44.9, 17.2. **IR (ν/cm^{-1}):** 3397, 1645, 1611, 1447, 1307, 1264, 1205, 1158, 854, 735. **HRMS (EI)** calcd. $C_{11}H_{12}O_2$ $[M]^+$: 176.0837. Found: 176.0839.

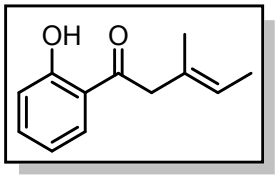


(*E*)-1-(2, 4, 6-trihydroxyphenyl) pent-3-en-1-one (7): Synthesized from 2,4,6-trihydroxybenzaldehyde and 1, 3-butdienes by following general procedure B on a 0.2 mmol scale, stirred for 36 hours at 50 ℃. 84% isolate yield. Light yellow oil. R_f=0.2 (PE: EA = 10:1). ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.20 (s, 1H), 10.37 (s, 1H), 5.80 (s, 1H), 5.70 – 5.09 (m, 1H), 3.73 (dd, J = 32.6, 6.8 Hz, 1H), 1.90 – 0.91 (m, 1H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 208.2, 169.9,169.4, 132.7, 130.3, 108.6, 99.8, 51.9, 23.1. **IR (ν/cm^{-1}):** 3153, 2932, 1659, 1589, 1463, 1391, 1288, 810, 732. **HRMS (EI)** calcd. $C_{11}H_{12}O_4$ $[M]^+$: 208.0736, Found: 208.0735.

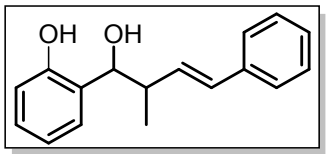


1-(2-hydroxyphenyl)-2-methylbut-3-en-1-one (8): Synthesized from **1a** and 1, 3-butdiene by following general procedure B on a 0.2 mmol scale, stirred for 36 hours at

30 ℃. 39% isolate yield, colorless oil. R_f=0.4 (PE: EA = 40:1). ^{1}HNMR (400 MHz, $CDCl_3$) δ 12.37 (s, 1H), 7.83 (d, J = 8.4 Hz, 1H), 7.47 (t, J = 7.2 Hz, 1H), 6.99 (d, J = 8.8 Hz, 1H), 6.90 (t, J = 7.2 Hz, 1H), 6.47 – 5.64 (m, 1H), 5.30 – 5.05 (m, 2H), 4.10-4.28 (m, 1H), 1.36 (d, J = 6.8 Hz 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.4, 163.2, 137.6, 136.4, 130.1, 118.8, 118.7, 118.5, 116.9, 44.9, 17.1. **IR (ν/cm^{-1}):** 3356, 2342, 1639, 1451, 1151, 734. **HRMS (EI)** calcd. $C_{11}H_{12}O_2$ [M] $^+$: 176.0837. Found: 176.0838.

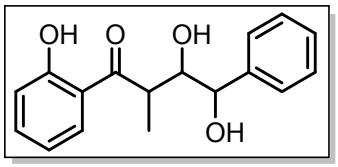


(*E*)-1-(2-hydroxyphenyl)-3-methylpent-3-en-1-one (10): Synthesized from **1a** and isoprene by following general procedure B on a 0.2 mmol scale, stirred for 6 hours at 70 ℃. 70% isolate yield. Colorless liquid. R_f=0.4 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.32 (s, 1H), 7.80 (d, J = 8.0 Hz, 1H), 7.46 (t, J = 8.0 Hz, 1H), 6.98 (d, J = 8.0 Hz, 1H), 6.88 (t, J = 8.0 Hz, 1H), 5.40 (q, J = 4.0, 8.0 Hz, 1H), 3.66 (s, 2H), 1.70 (s, 3H), 1.65 (d, J = 4.0 Hz, 3H). 13C NMR (101 MHz, $CDCl_3$) δ 205.3, 162.7, 136.4, 130.4, 129.6, 124.2, 119.4, 118.8, 118.5, 48.9, 16.3, 13.7. **IR (ν/cm^{-1}):** 3384, 1685, 1510, 1475. 1307, 730. **HRMS (EI)** calcd. $C_{12}H_{14}O$ [M] $^+$: 190.0994. Found:190.0995.



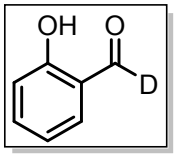
(*E*)-2-methyl-1, 4-diphenylbut-3-en-1-ol (11): Synthesized by following experimental procedure shown in section 2.4. 91% isolate yield, isolated as a diastereoselective mixture with a 1.8:1 dr. Colorless liquid. R_f=0.2 (PE: EA = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14(s, 0.37H), 8.02(s, 0.61H), 7.65 – 6.72 (m, 9H), 6.61 (d, J = 16.0 Hz, 0.64H), 6.43 (d, J = 16.0 Hz, 0.37H), 6.20 – 6.08 (m, 1H), 4.86 (d, J = 4.0 Hz, 0.36H), 4.54 (d, J = 8.0 Hz, 0.65H), 2.96 – 2.75 (m, 1H), 1.20 (d, J = 8.0 Hz, 1.1H), 1.00 (d, J = 8.0 Hz, 1.9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 155.9, 155.7, 136.7, 134.5, 133.1, 131.6, 131.4, 131.1, 129.8, 129.5, 129.2, 129.0, 128.9, 128.8, 128.7,

128.5, 128.1, 127.7, 127.3, 126.3, 126.2, 124.6, 119.4, 117.4, 117.2, 44.2, 44.0, 17.3, 14.4. **IR (ν/cm^{-1}):** 3324, 3229, 2835, 1456, 1378, 1044, 966, 893, 735, 703. **HRMS (ESI)** calcd. $C_{17}H_{17}O_2$ [M-H$^+$]: 253.1229. Found: 253.1226.



3, 4-dihydroxy-1-(2-hydroxyphenyl)-2-methyl-4-phenylbutan-1-one (12):

Synthesized by following experimental procedure shown in section 2.5. 74% isolate yield, isolated as a diastereoselective mixture with a 3:1 dr. Off-white foam. R_f=0.3-0.35 (PE: EA = 10:1). The spectra for one of the two diastereomers were obtained. ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.15 (s, 1H), 7.55 – 6.48 (m, 2H), 7.35-7.22 (m, 4H), 7.02 (d, J = 8.8 Hz, 1H), 6.87 (t, J = 7.6 Hz, 1H), 4.66 (d, J = 6.4 Hz, 1H), 3.88 (t, J = 5.2 Hz, 1H), 3.68-2.89 (m, 2H), 3.18 (s, 1H), 1.62 (s, 2H), 1.37 (d, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 211.7, 163.4, 140.5, 137.3, 130.2, 128.6, 128.2, 126.6, 119.2, 118.9, 118.6, 79.4, 75.2, 40.0, 16.5. **IR (ν/cm^{-1}):** 3511, 3425, 1635, 1493, 1258, 977, 713, 553. **HRMS (EI)**: calcd. $C_{17}H_{18}O_4$ [M] $^+$: 286.1205. Found: 286.1204.



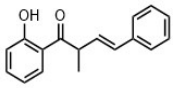
d-**Salicylaldehyde (*d*-1a):** Synthesis method following the literature procedures.[2]. 31% isolate yield, yellow solid. R_f=0.2 (PE: EA = 40:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.98 (s, 1H), 7.78 – 7.36 (m, 2H), 7.05-6.95 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.2 (t, J = 27.3 Hz), 160.7, 136.0, 132.7, 119.5, 118.8, 116.6.

5. Reference:

[1] (*a*) H. Kinuta, M. Tobisu and N. Chatani, *J. Am. Chem. Soc.*, 2015, **137**, 1593-1600. (*b*) A. Tortajada, R. Ninokata and R. Martin, *J. Am. Chem. Soc.*, 2018, **140**, 2050-2053.

[2] (*a*) M. von Delius, C. M. Le and V. M. Dong, *J. Am. Chem. Soc.*, 2012, **134**, 15022-15032. (*b*) H. S. Li, Y. Xiong and G. Z. Zhang, *Adv. Synth. Catal.*, 2018, **360**, 4246-4251.

6. NMR Spectrum



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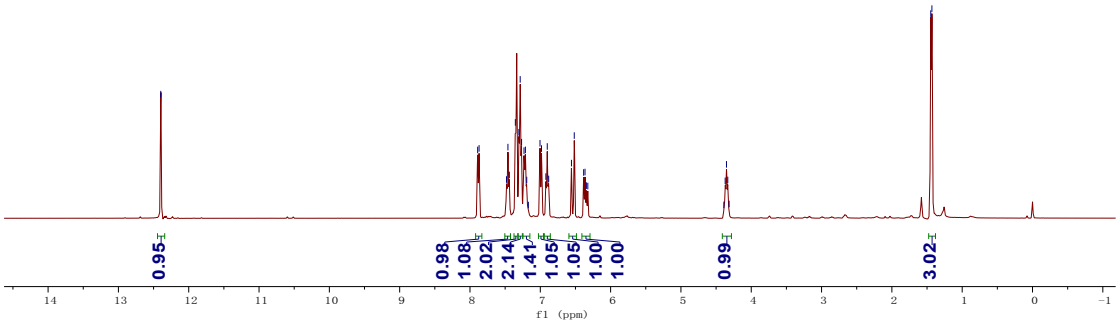

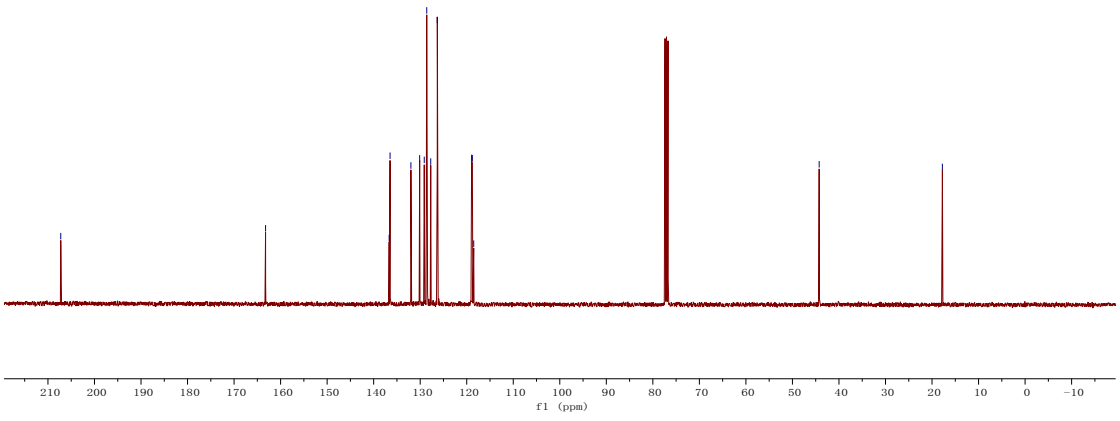

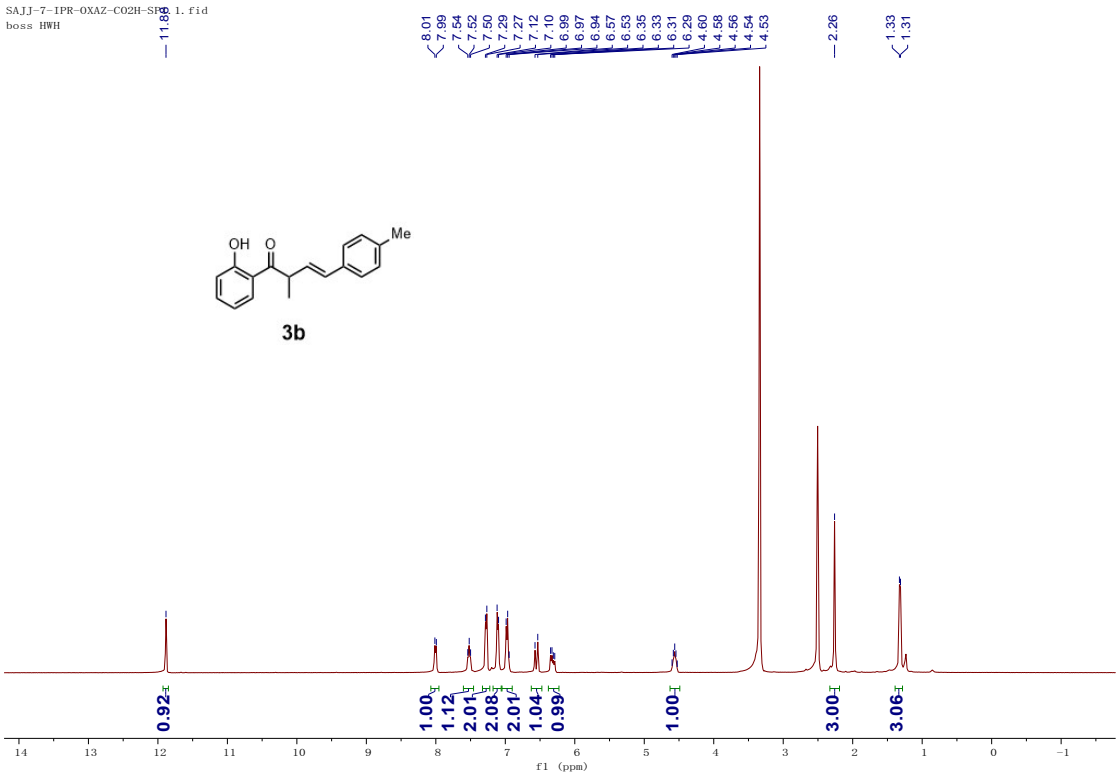
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7.52
7.50
7.29
7.27
7.12
7.10
6.99
6.97
6.94
6.57
6.53
6.35
6.33
6.31
6.29
4.60
4.58
4.56
4.54
4.53
2.26
1.33
1.31
OH
O
Me
3b
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2.01
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3.00
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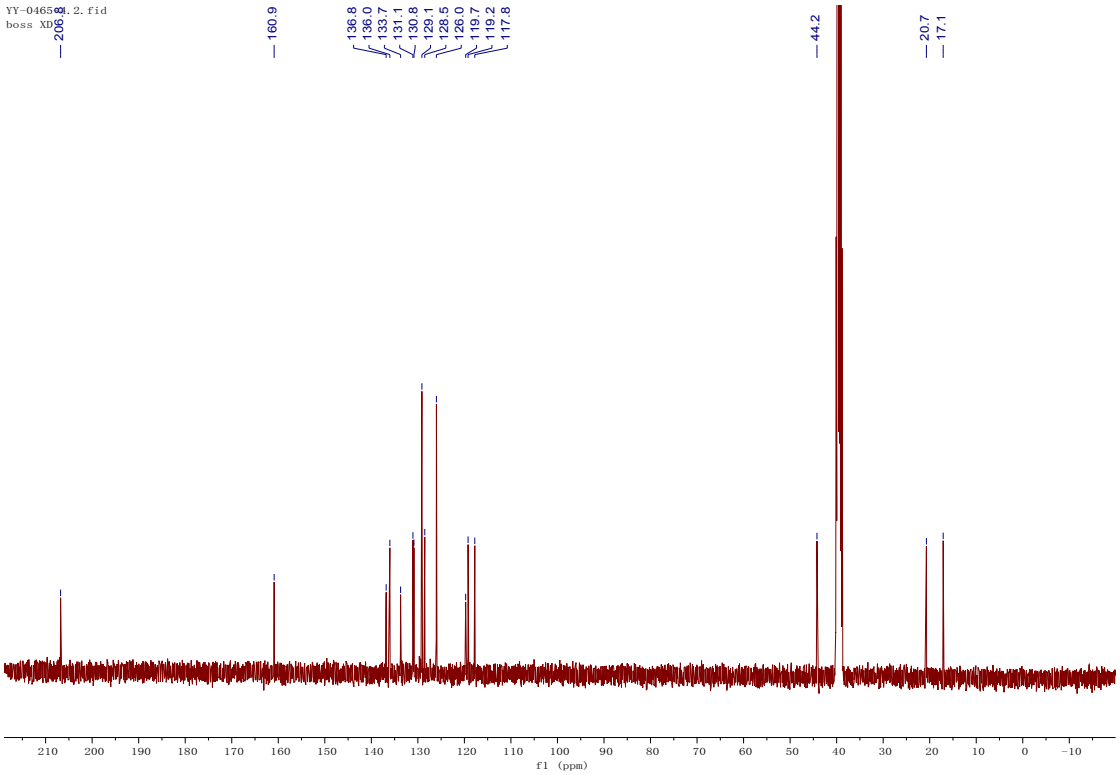
f1 (ppm)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129.1
128.5
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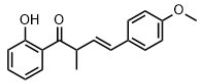
119.7
119.2
117.8
44.2
20.7
17.1
f1 (ppm)

YY-03109-A.1.fid
boss XD



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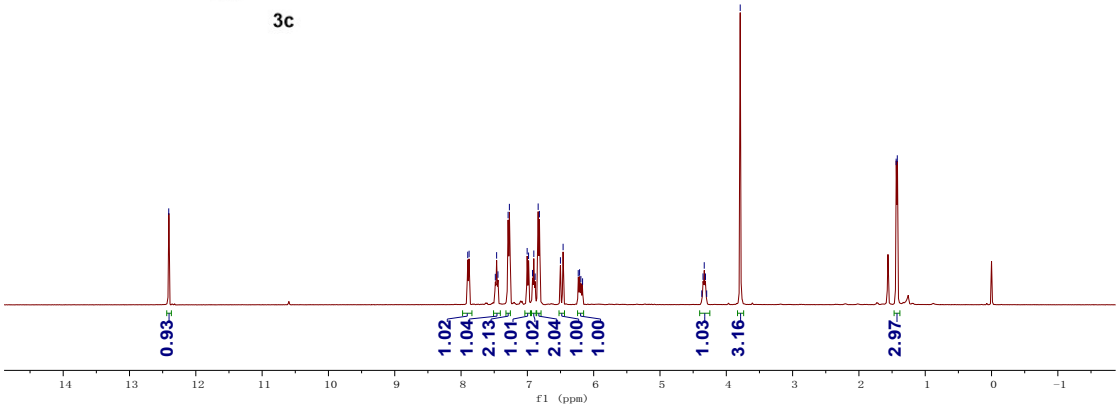

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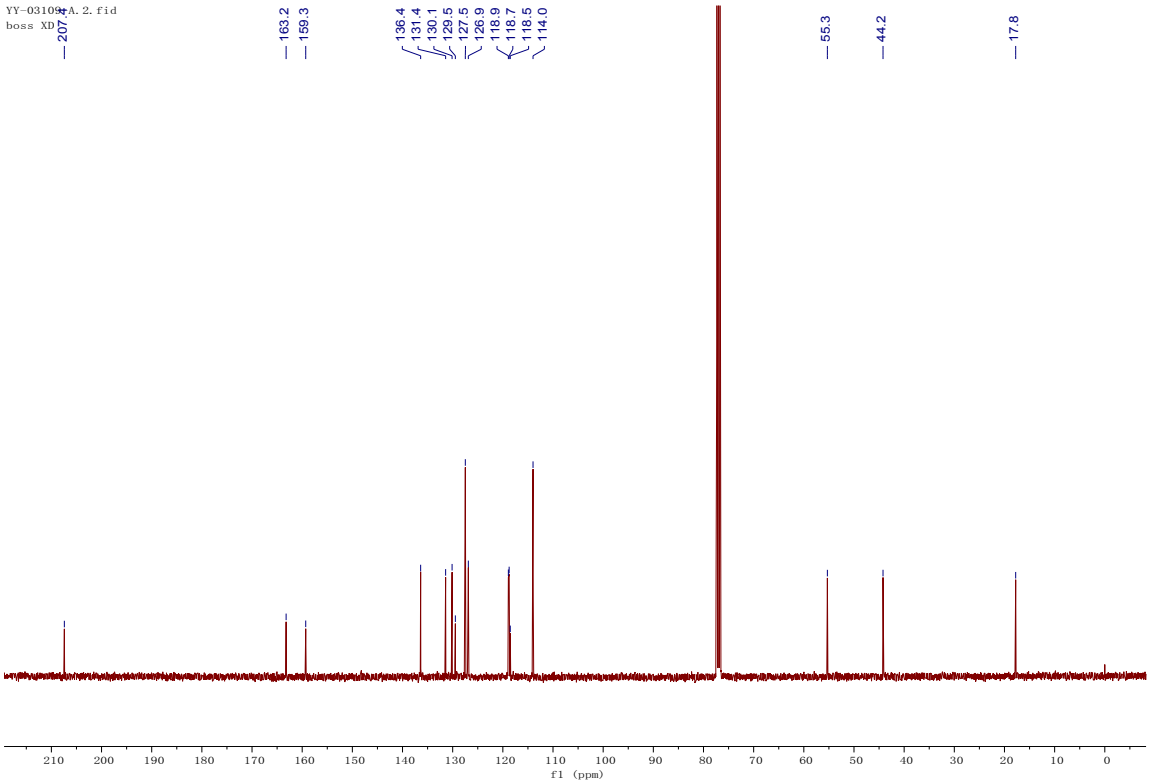
boss XD

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7.00
6.98
6.98
6.96
6.95
6.93
6.91
6.89
6.52
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4.31
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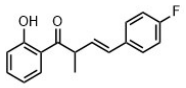
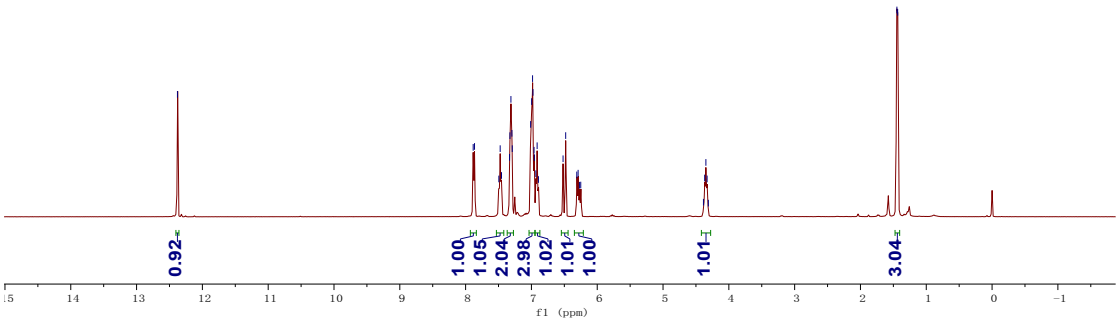
1.43


OH
O
F

3d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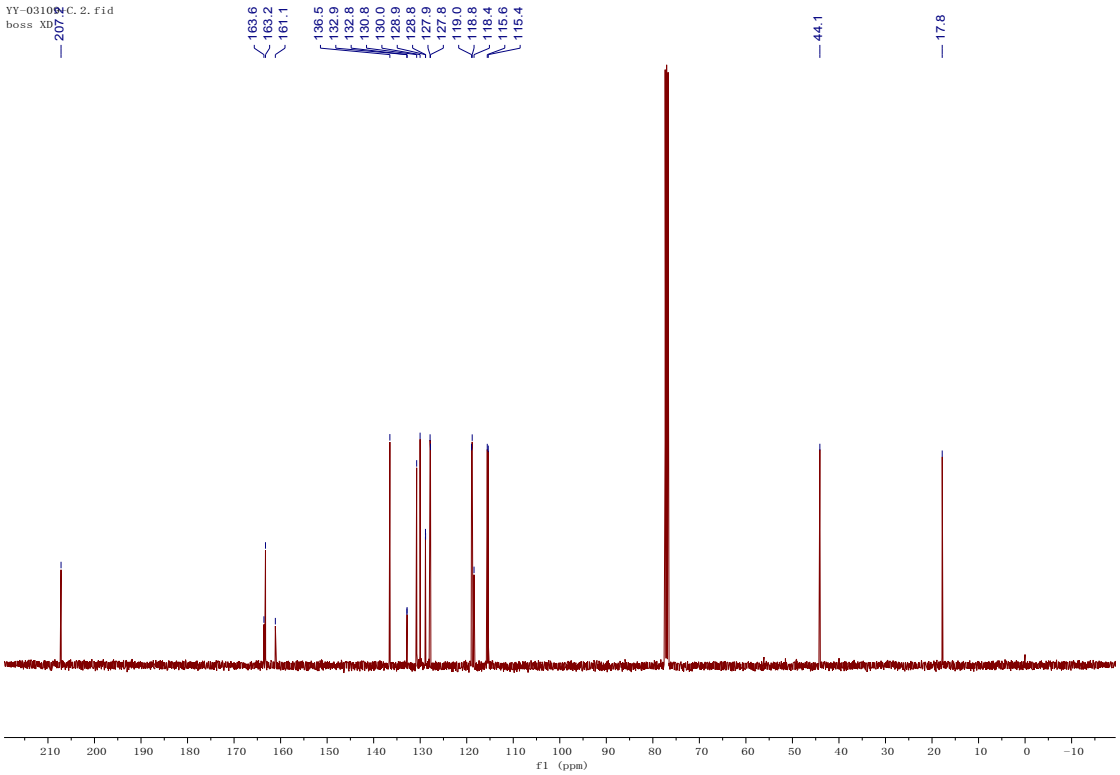
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130.0
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128.8
127.9
127.8
119.0
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118.4
115.6
115.4
44.1
17.8
f1 (ppm)

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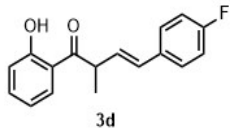
-114.2



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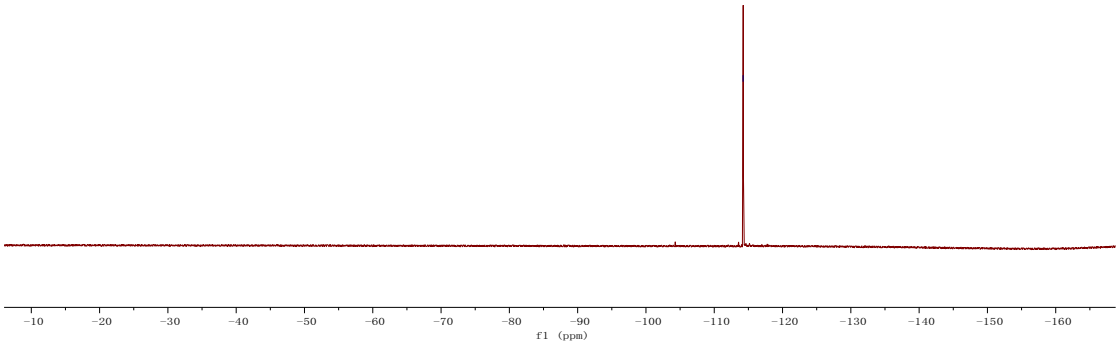

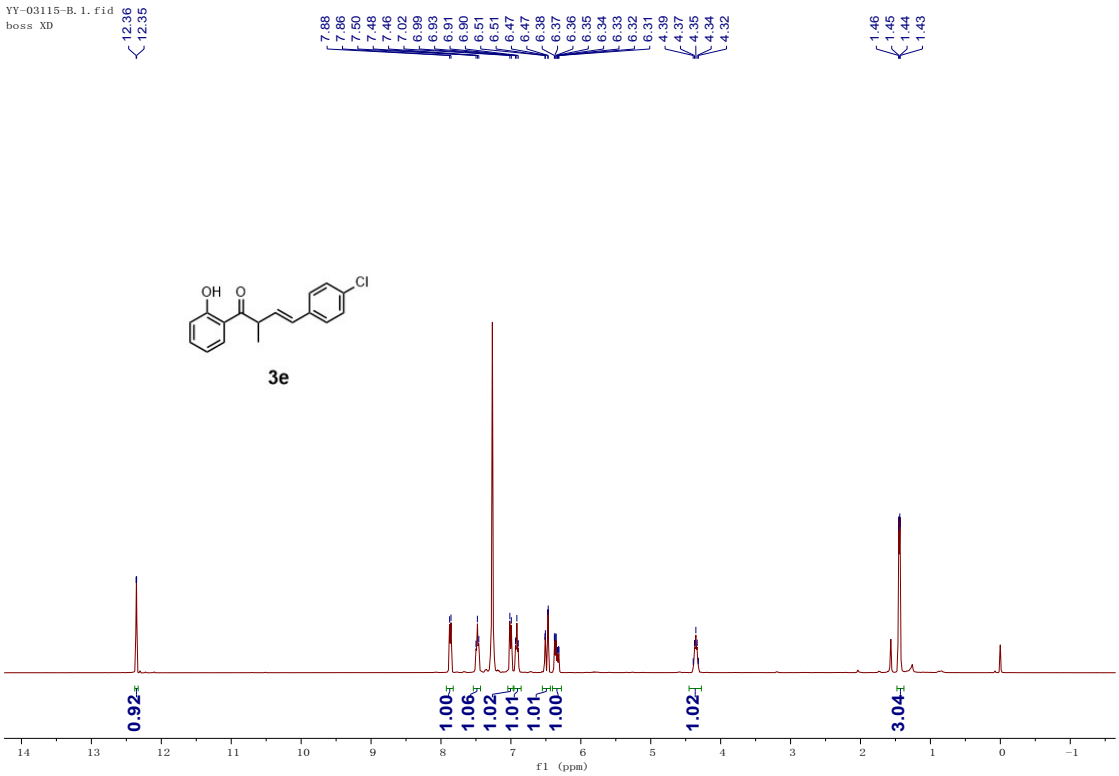
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6.90
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6.51
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6.47
6.38
6.37
6.36
6.35
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6.33
6.32
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4.34
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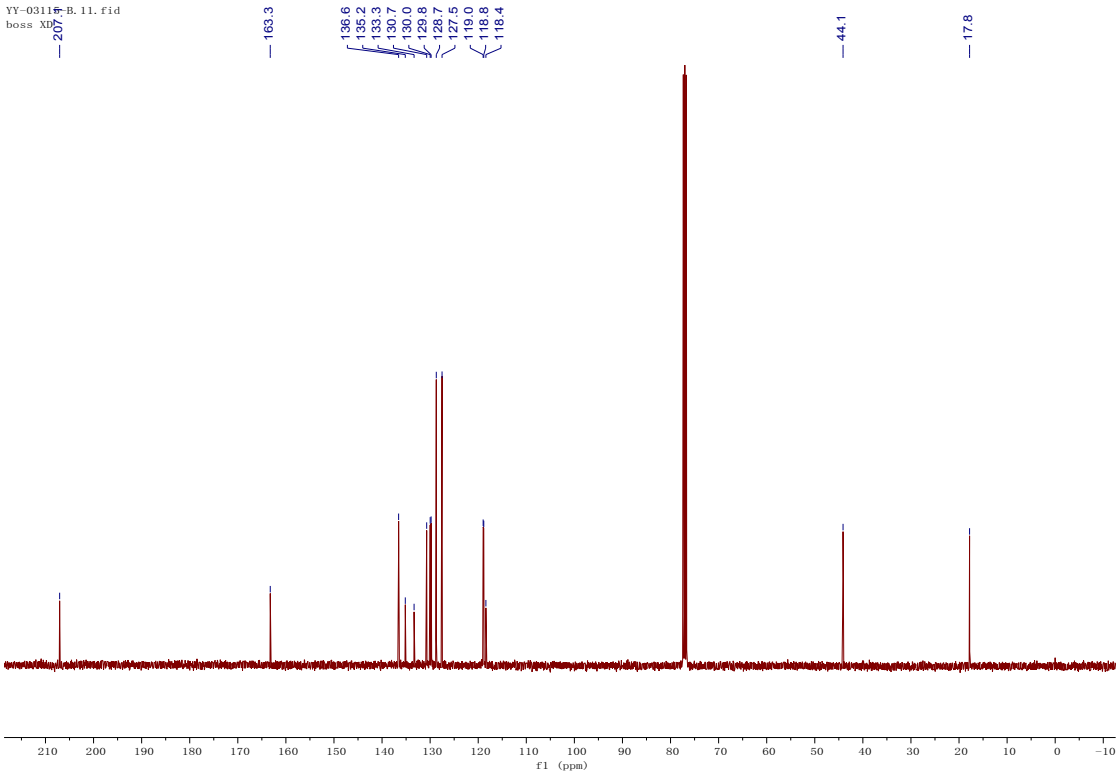
f1 (ppm)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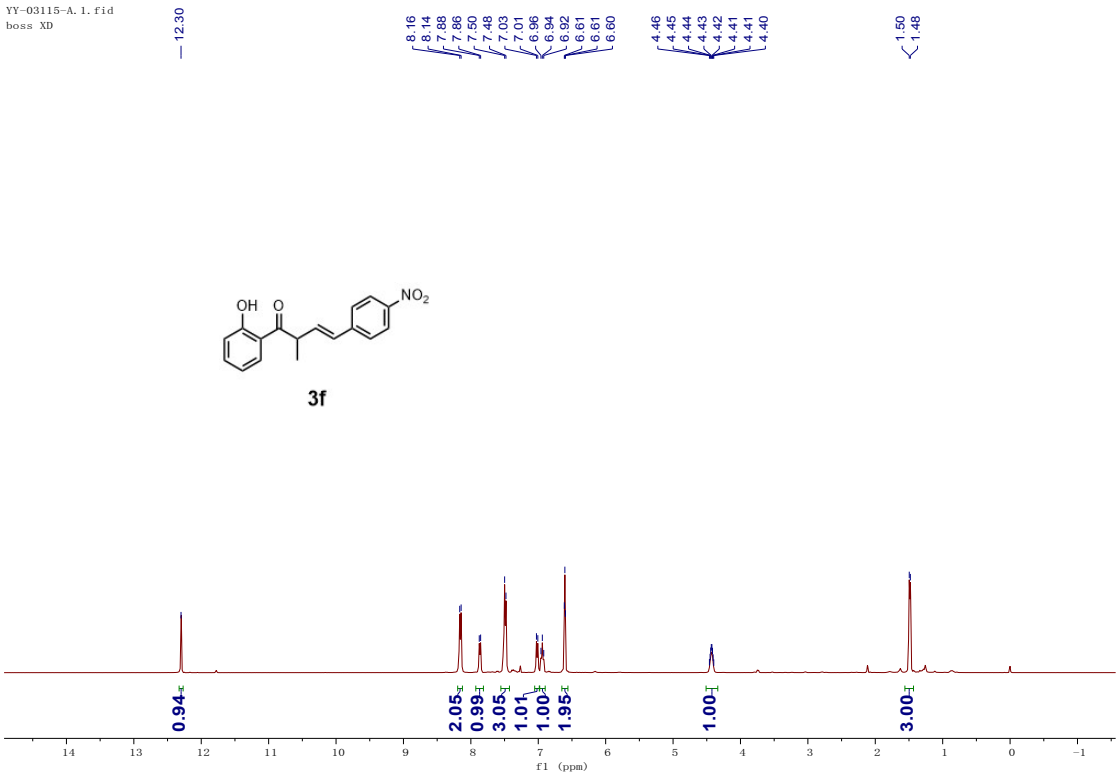
118.4
44.1
17.8
f1 (ppm)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6.61
6.61
6.60
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4.41
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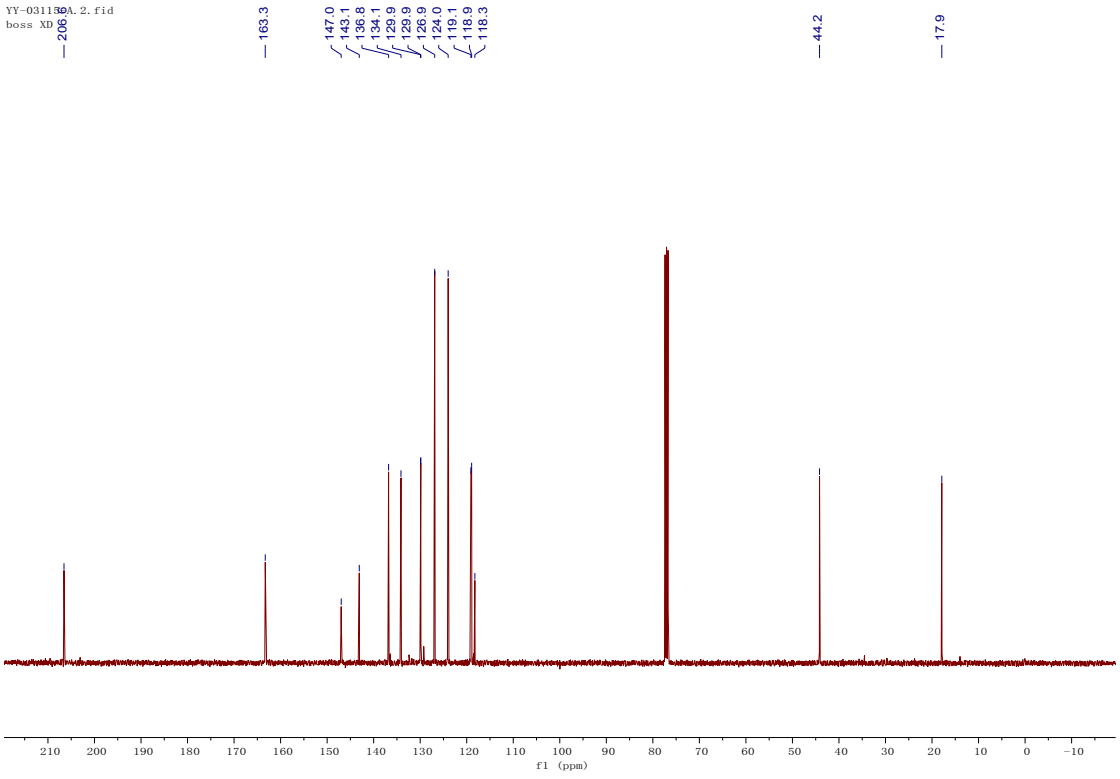
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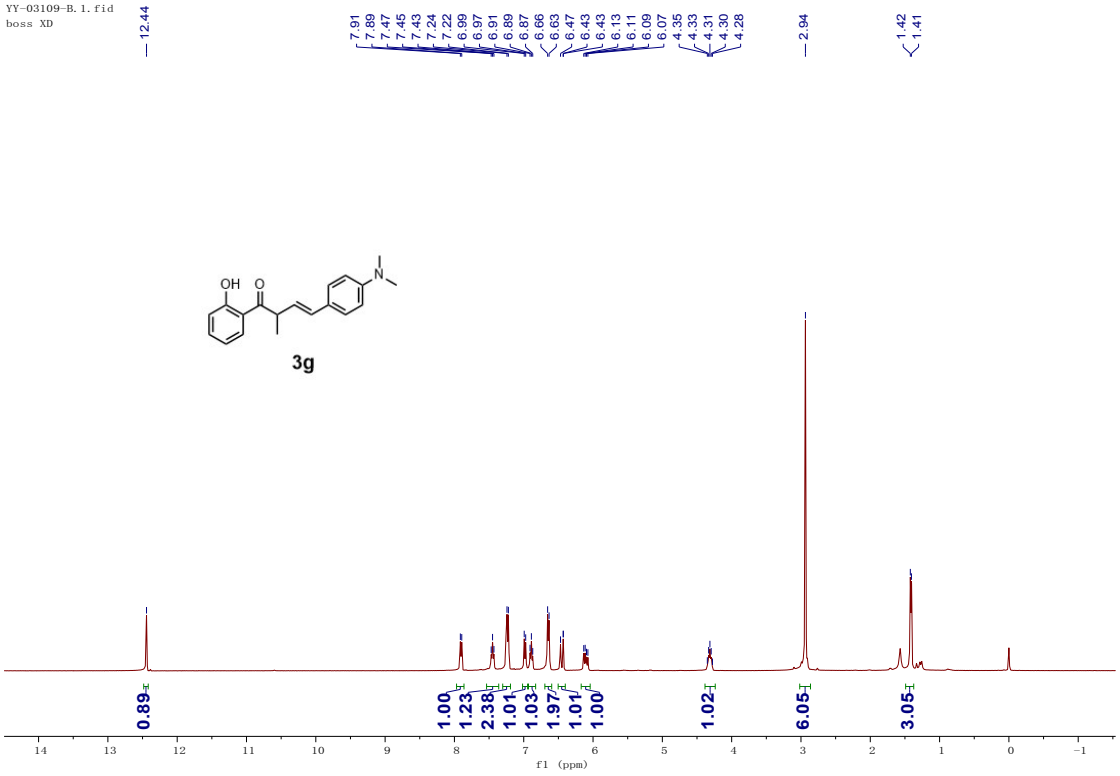
f1 (ppm)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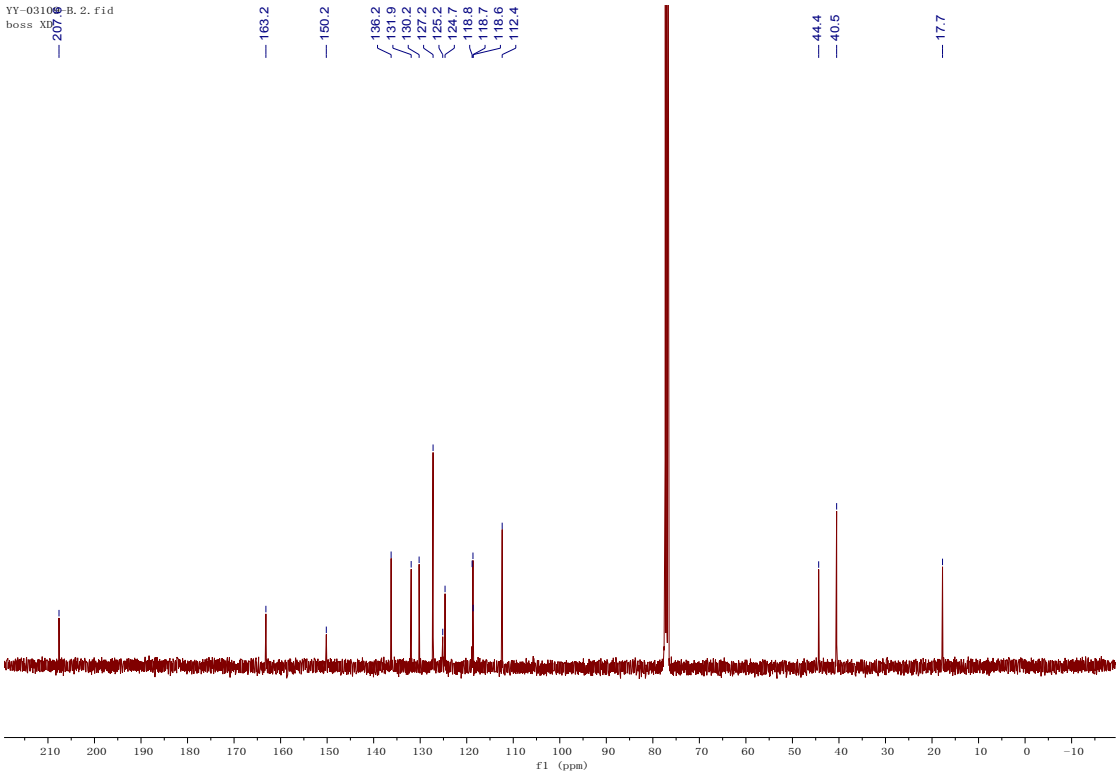
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f1 (ppm)


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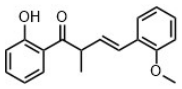
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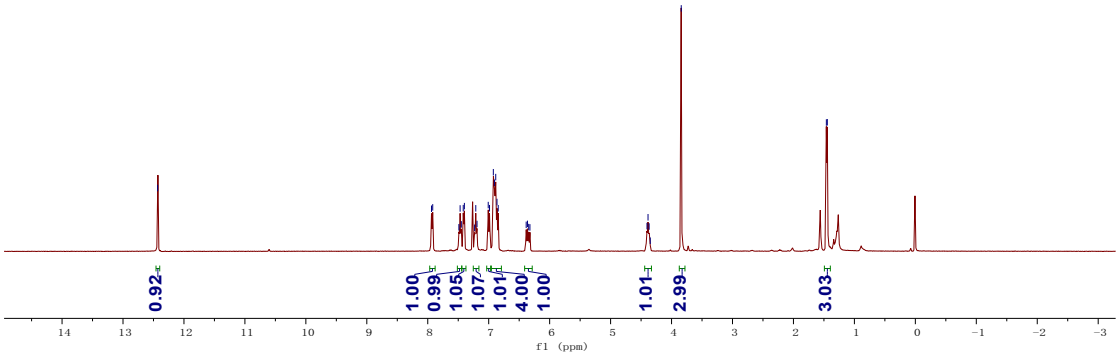
O

3h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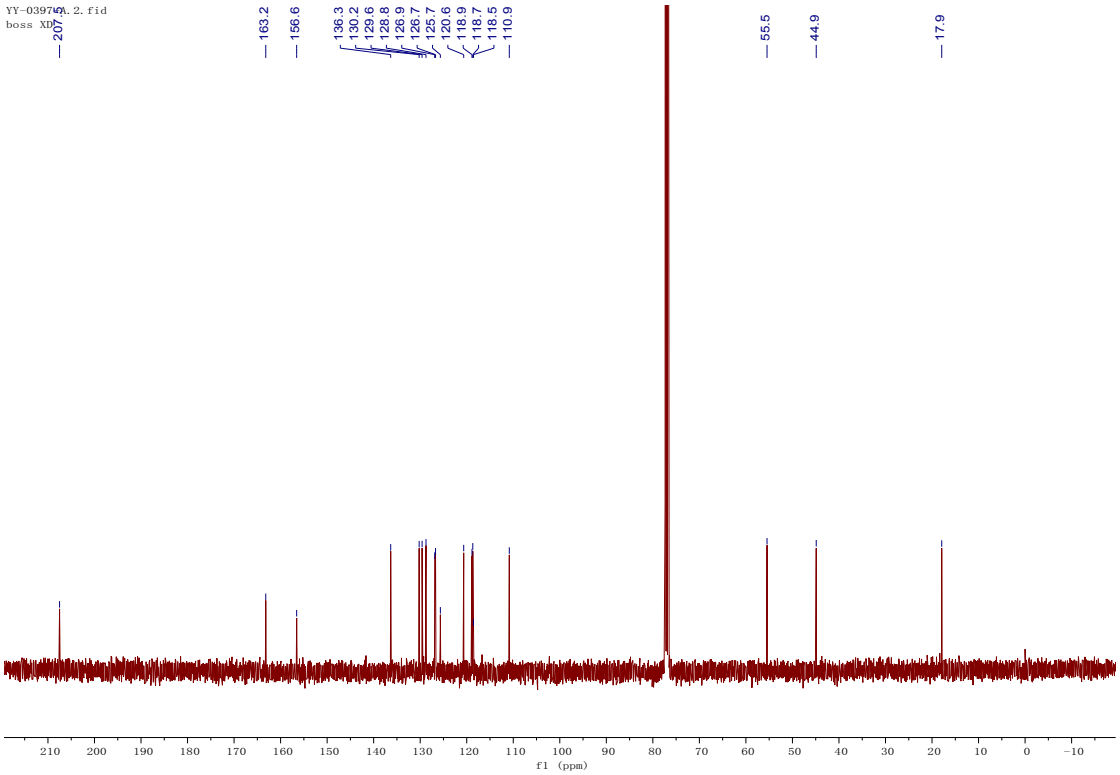
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118.5
110.9
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17.9
f1 (ppm)


YY-0397-B.20.fid
boss XD
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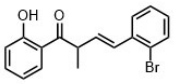
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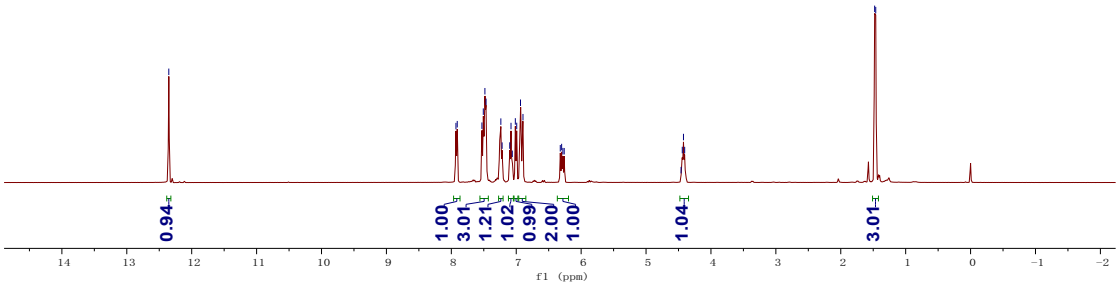
Br

3i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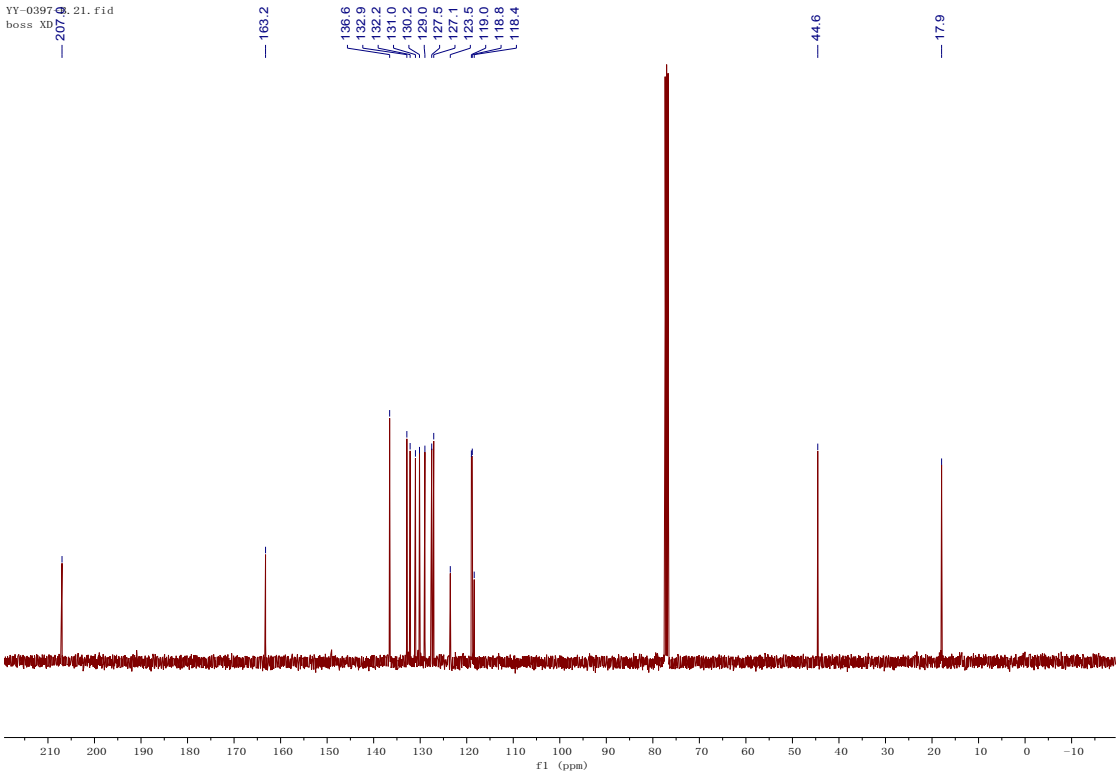
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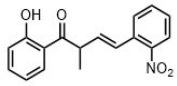
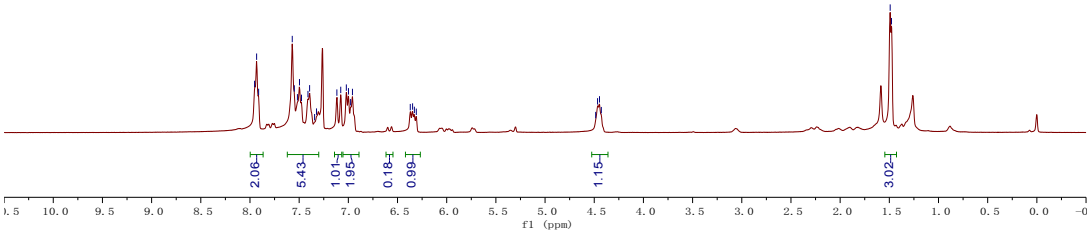
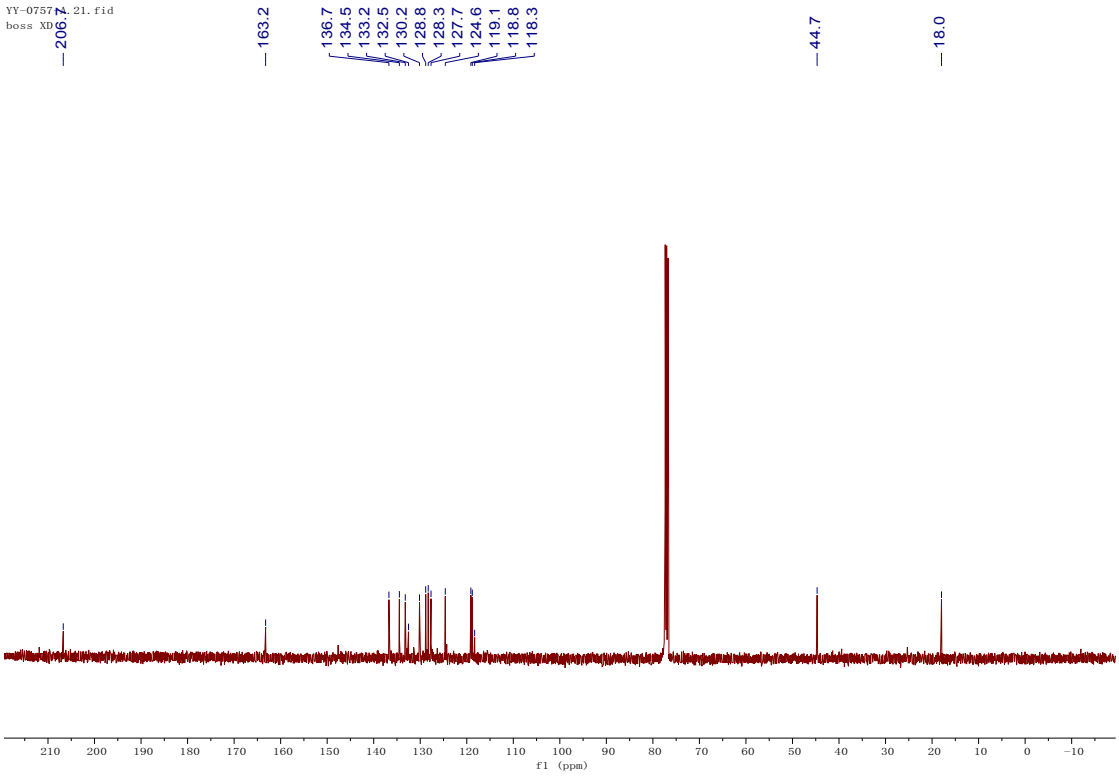
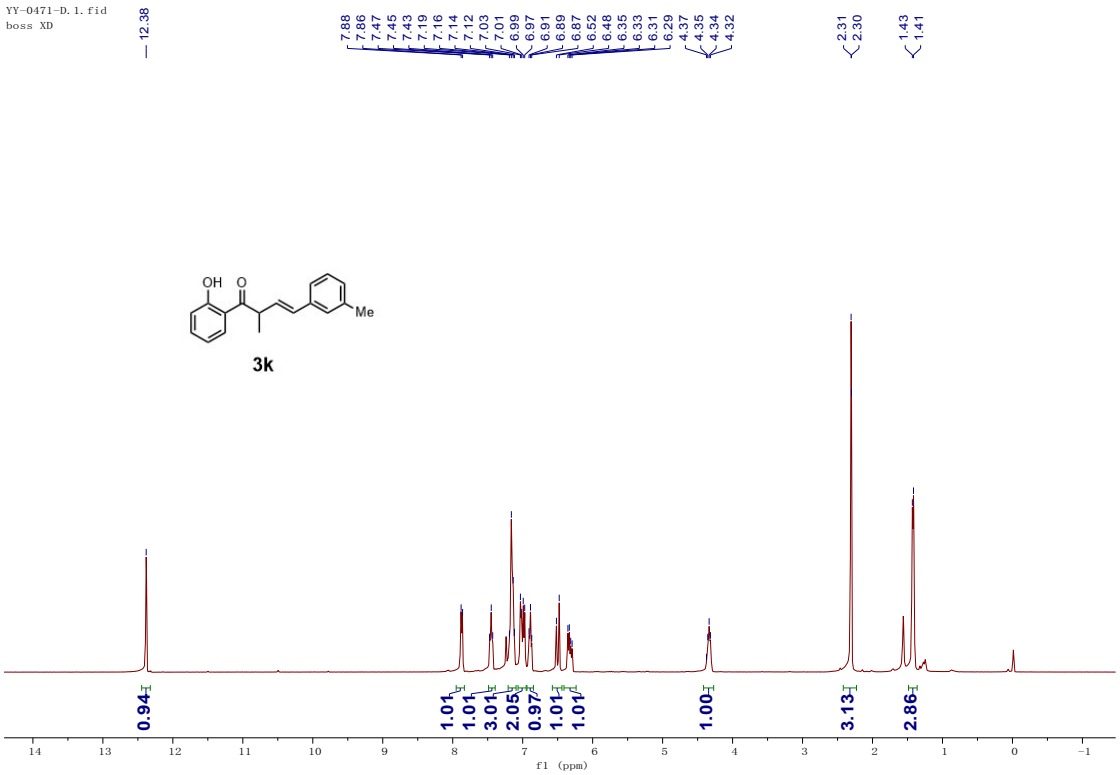


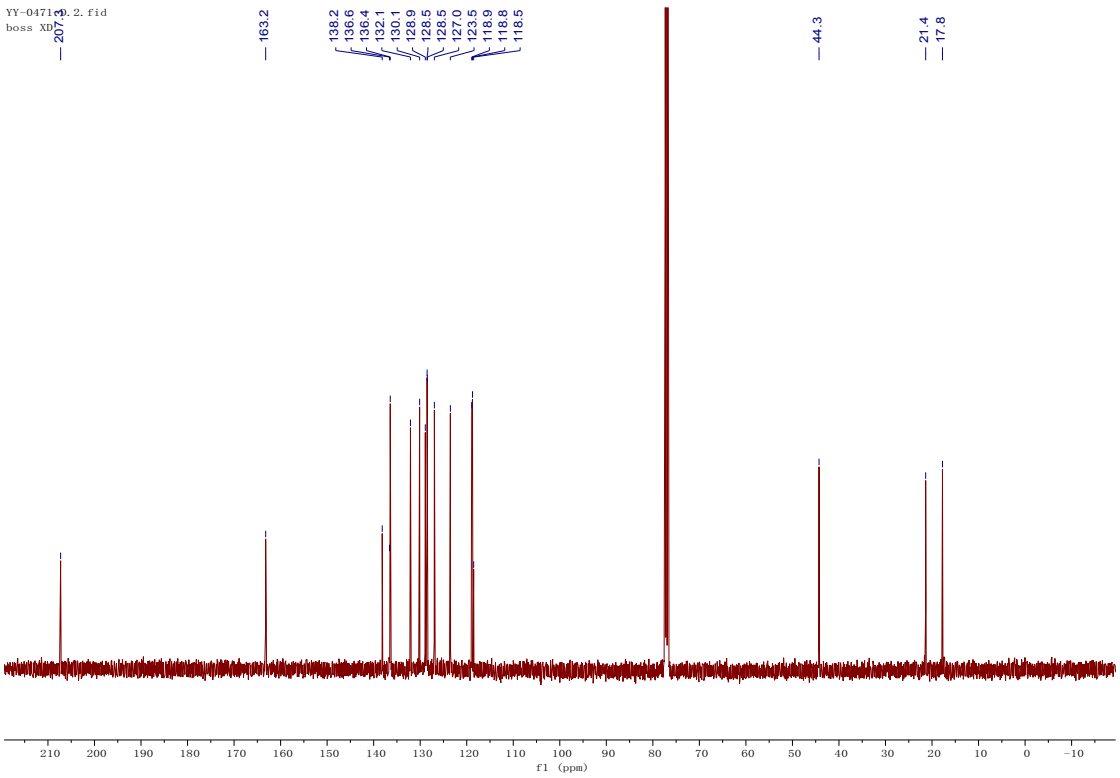
3j

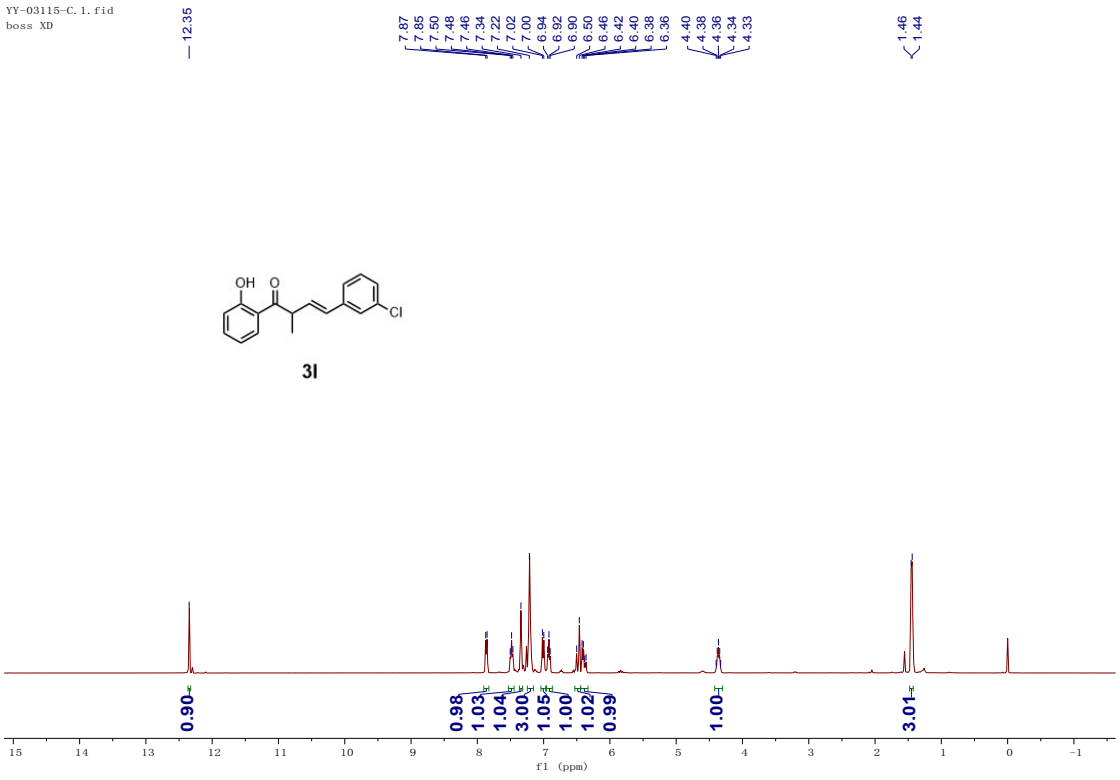
contains 15% of the linear-selective product



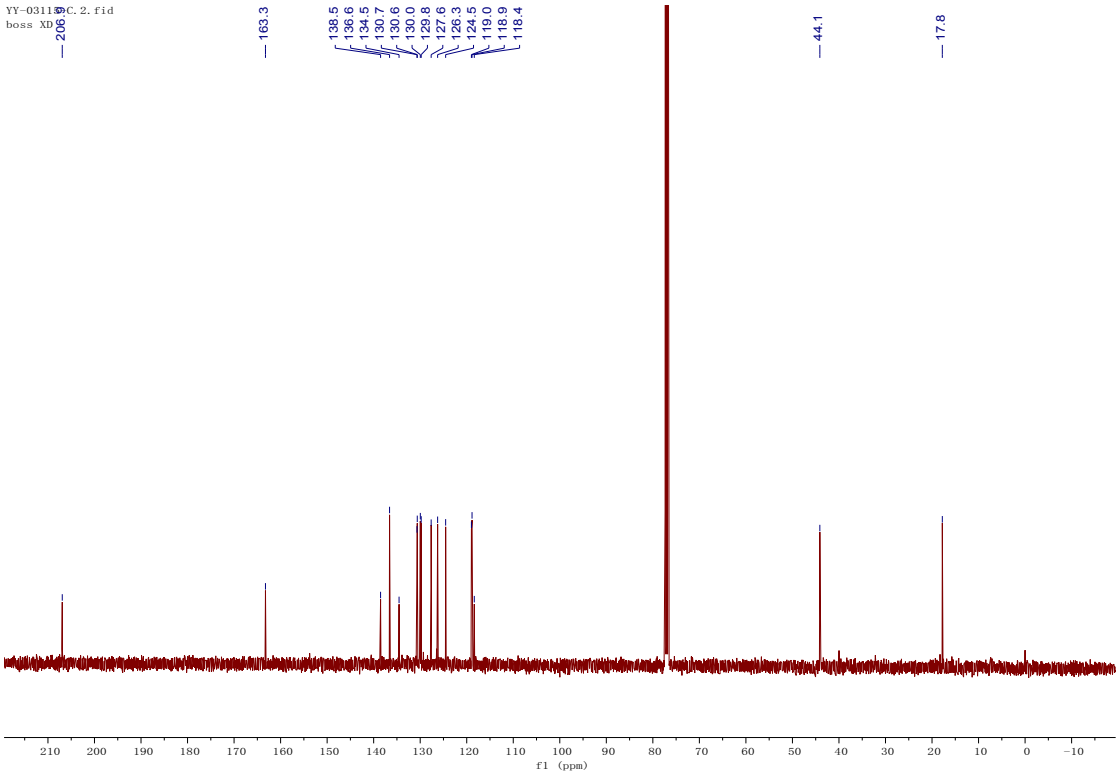


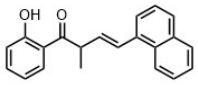

YY-0471-D.1.fid
boss XD
12.38
7.88
7.86
7.47
7.45
7.43
7.19
7.16
7.14
7.12
7.03
7.01
6.99
6.97
6.91
6.89
6.87
6.52
6.48
6.35
6.33
6.31
6.29
4.37
4.35
4.34
4.32
2.31
2.30
1.43
1.41
OH
O
Me
3k
0.94
1.01
1.01
3.01
2.05
0.97
1.01
1.01
1.00
3.13
2.86
f1 (ppm)


YY-0471-D.2.fid
boss XD
207.7
163.2
138.2
136.6
136.4
132.1
130.1
128.9
128.5
128.5
127.0
123.5
118.9
118.8
118.5
44.3
21.4
17.8
f1 (ppm)

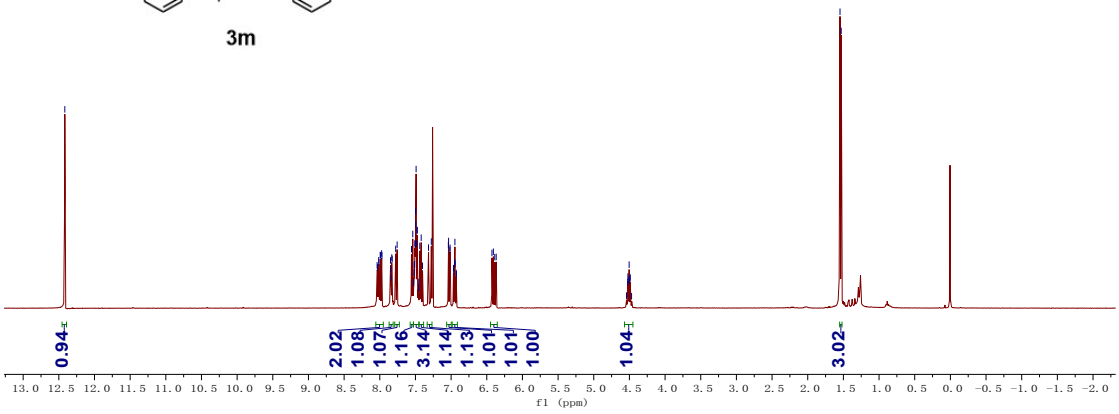

YY-03115-C.1.fid
boss XD
12.35
7.87
7.85
7.50
7.48
7.46
7.34
7.22
7.02
7.00
6.94
6.92
6.90
6.50
6.46
6.42
6.40
6.38
6.36
4.40
4.38
4.36
4.34
4.33
1.46
1.44
OH
O
Cl
0.90
0.98
1.03
1.04
3.00
1.05
1.00
1.02
0.99
1.00
3.01
f1 (ppm)

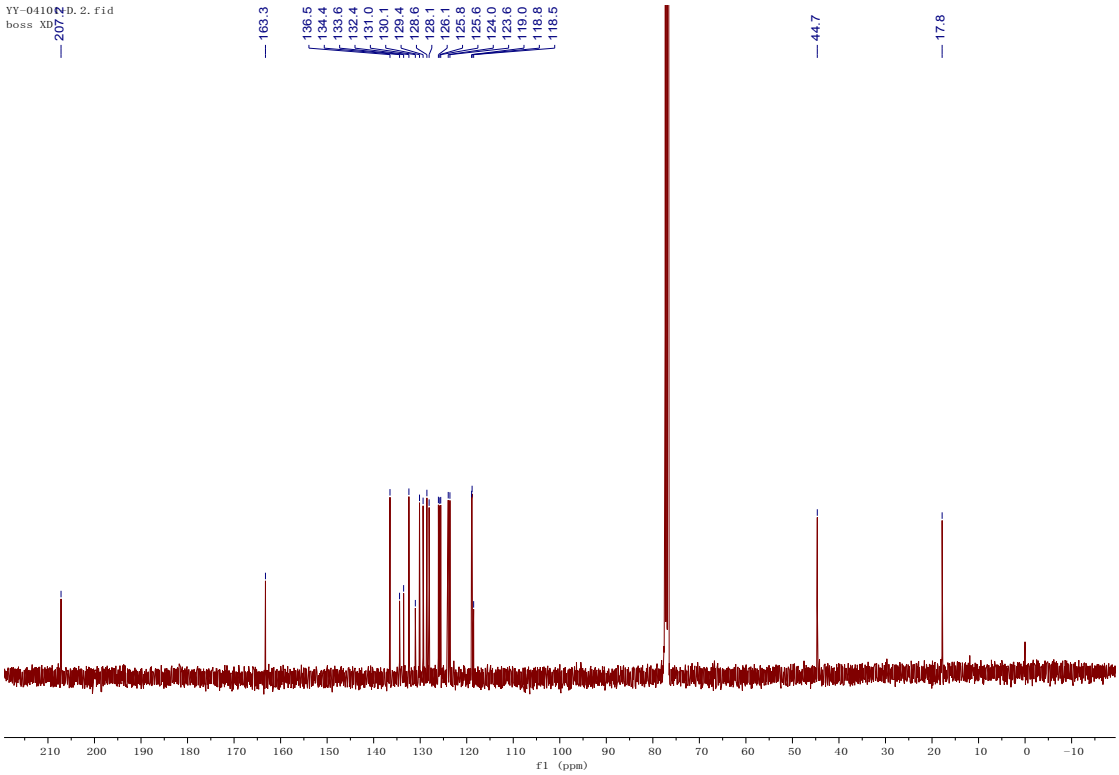
3l


YY-03115-C.2.fid
boss XD
206.9
163.3
138.5
136.6
134.5
130.7
130.6
130.0
129.8
127.6
126.3
124.5
119.0
118.9
118.4
44.1
17.8
f1 (ppm)

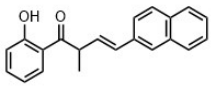

OH
O

3m

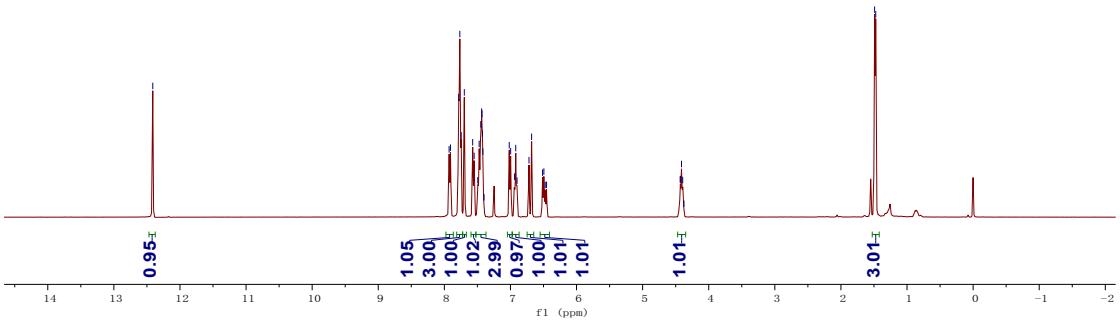

0.94
2.02
1.08
1.07
1.16
3.14
1.14
1.13
1.01
1.01
1.00
1.04
3.02
f1 (ppm)

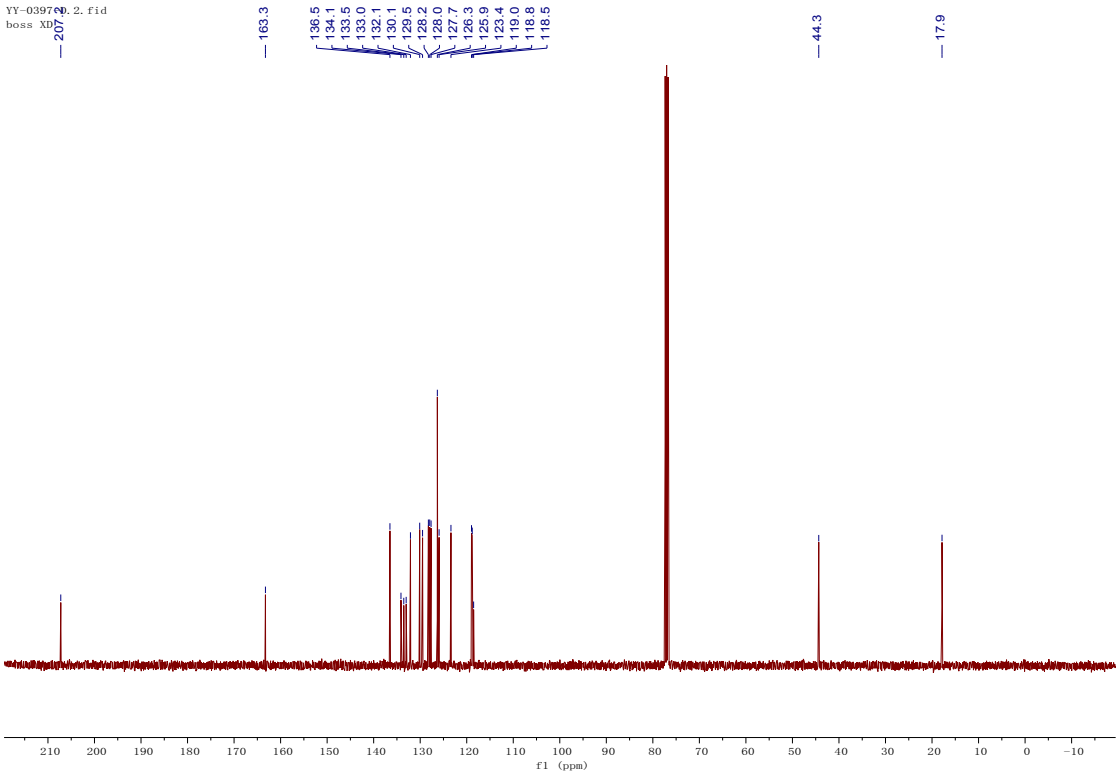

207.4
163.3
136.5
134.4
133.6
132.4
131.0
130.1
129.4
128.6
128.1
126.1
125.8
125.6
124.0
123.6
119.0
118.8
118.5
44.7
17.8
f1 (ppm)

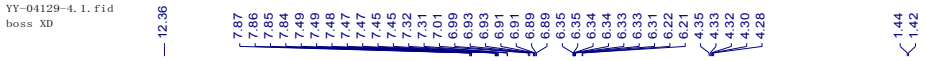

YY-0397-D.1.fid
boss XD
12.41
7.93
7.91
7.78
7.77
7.75
7.70
7.57
7.55
7.49
7.47
7.45
7.44
7.43
7.42
7.42
7.40
7.02
7.00
6.94
6.92
6.90
6.72
6.68
6.51
6.49
6.47
6.45
4.43
4.43
4.41
4.39
4.38
1.49
1.47

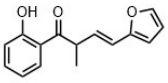

OH
O

3n

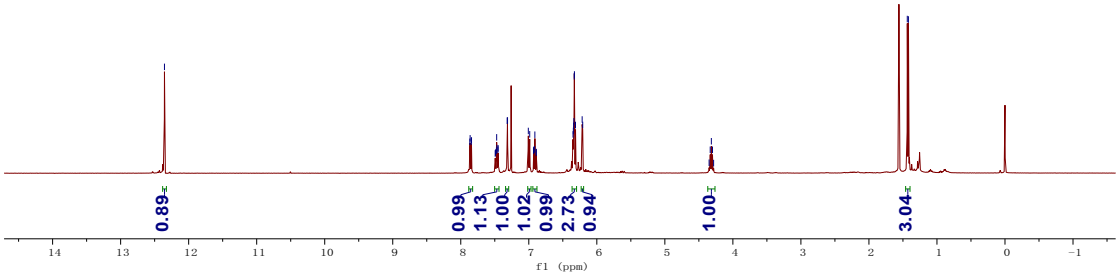

0.95
1.05
3.00
1.00
1.02
2.99
0.97
1.00
1.01
1.01
1.01
3.01
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
f1 (ppm)

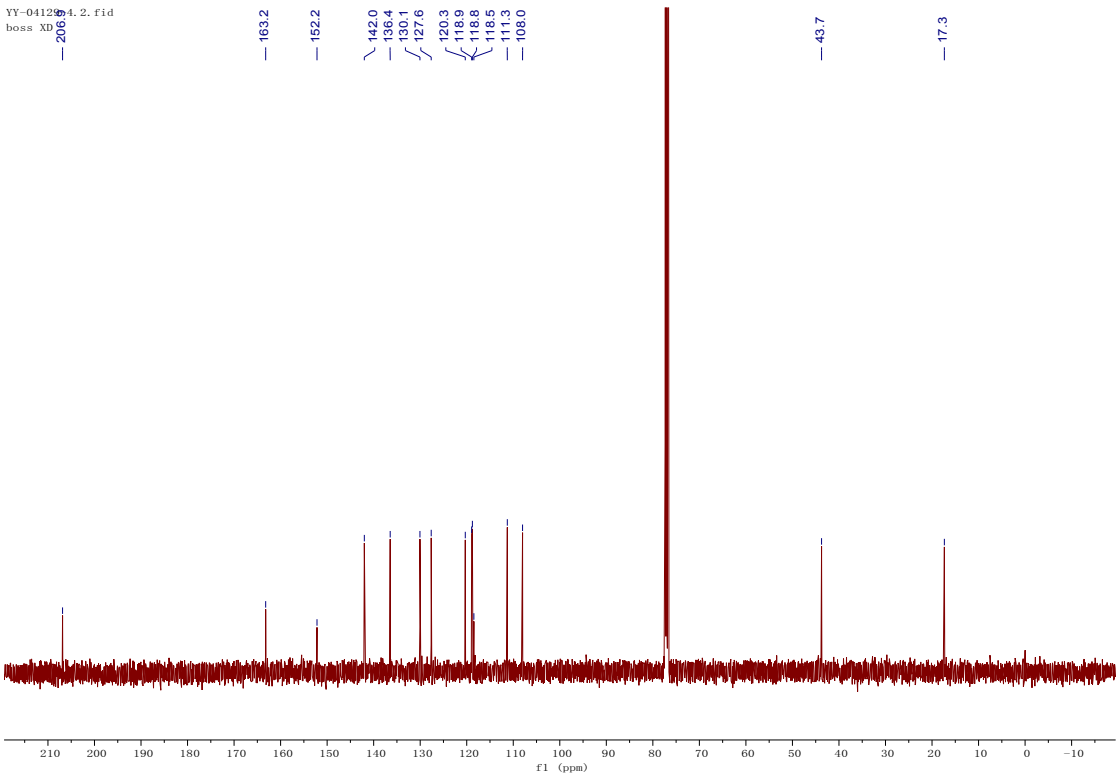

YY-0397-D.2.fid
boss XD
207.7
163.3
136.5
134.1
133.5
133.0
132.1
130.1
129.5
128.2
128.0
127.7
126.3
125.9
123.4
119.0
118.8
118.5
44.3
17.9
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


YY-04129-4.1.fid
boss XD
12.36
7.87
7.86
7.85
7.84
7.49
7.49
7.48
7.47
7.47
7.45
7.45
7.32
7.31
7.01
6.99
6.93
6.93
6.91
6.91
6.89
6.89
6.35
6.35
6.34
6.34
6.33
6.33
6.31
6.22
6.21
4.35
4.33
4.32
4.30
4.28
1.44
1.42


OH
O
O

3o


0.89
0.99
1.13
1.00
1.02
0.99
2.73
0.94
1.00
3.04
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
f1 (ppm)

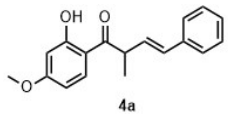

YY-04129-4.2.fid
boss XD
206.9
163.2
152.2
142.0
136.4
130.1
127.6
120.3
118.9
118.8
118.5
111.3
108.0
43.7
17.3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

YY-0757-B.30.fid
boss XD

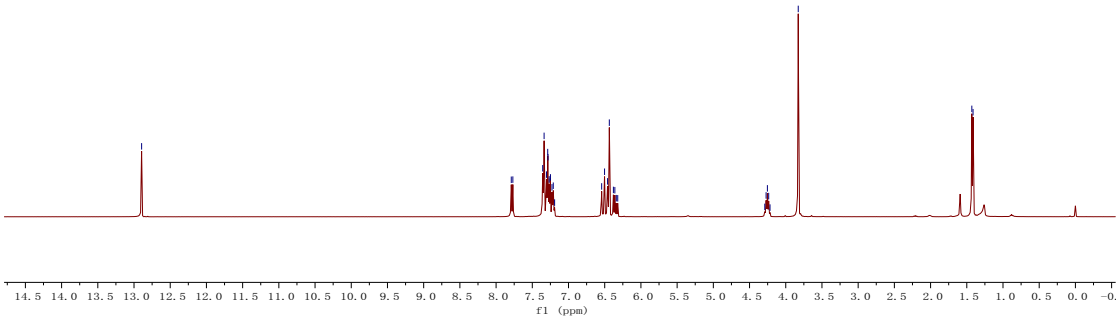
12.9

7.8 7.8 7.4 7.3 7.3 7.3 7.3 7.3 7.2 7.2 7.2 7.2 6.5 6.5 6.5 6.4 6.4 6.4 6.3 6.3 4.3 4.3 4.3 4.2 4.2 3.8

1.4 1.4



4a



YY-03139-2.fid
boss XD

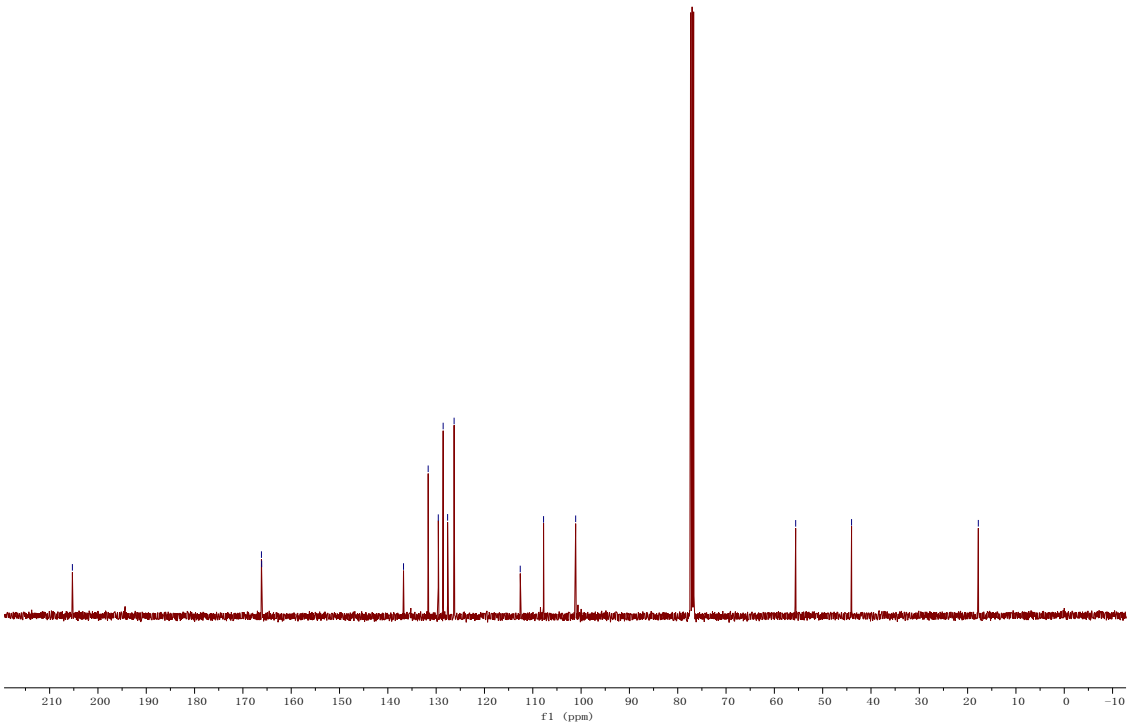
205.8

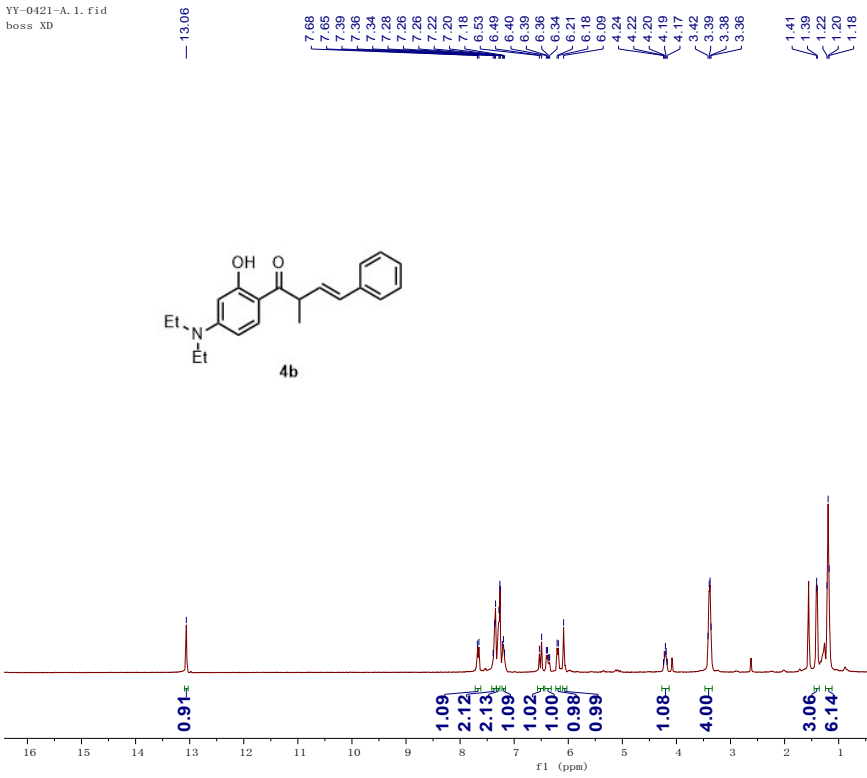
166.2 166.1

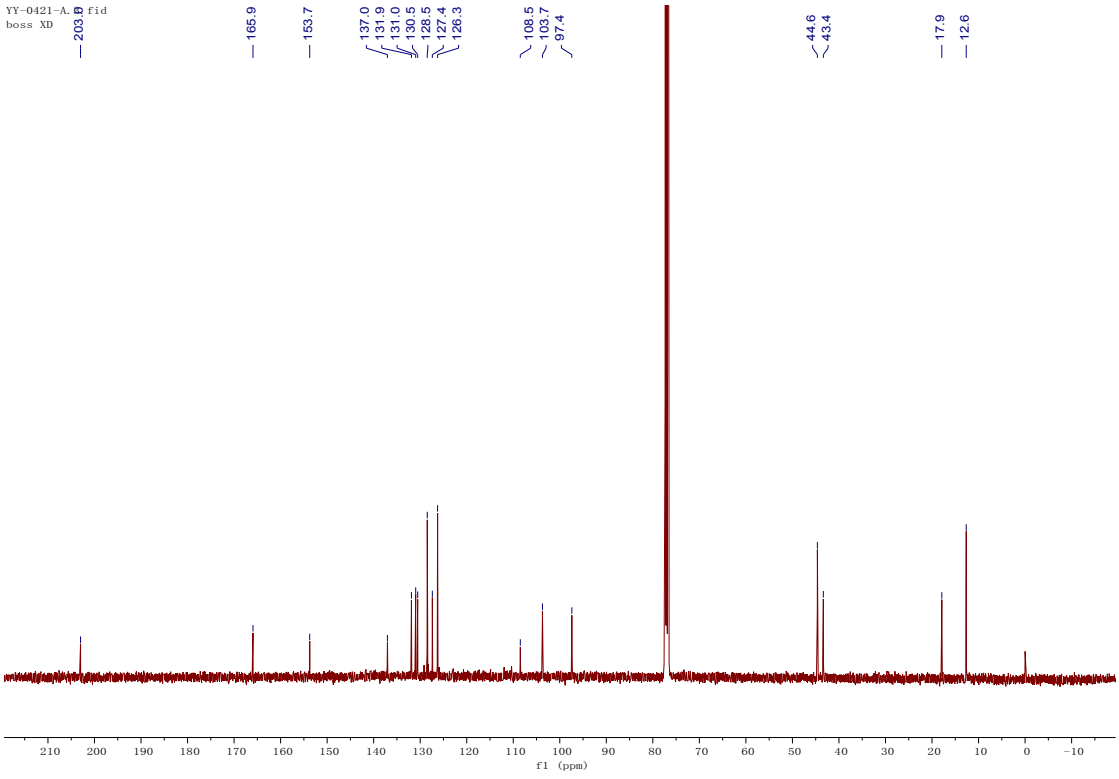
136.8 131.7 129.6 128.6 127.6 126.3

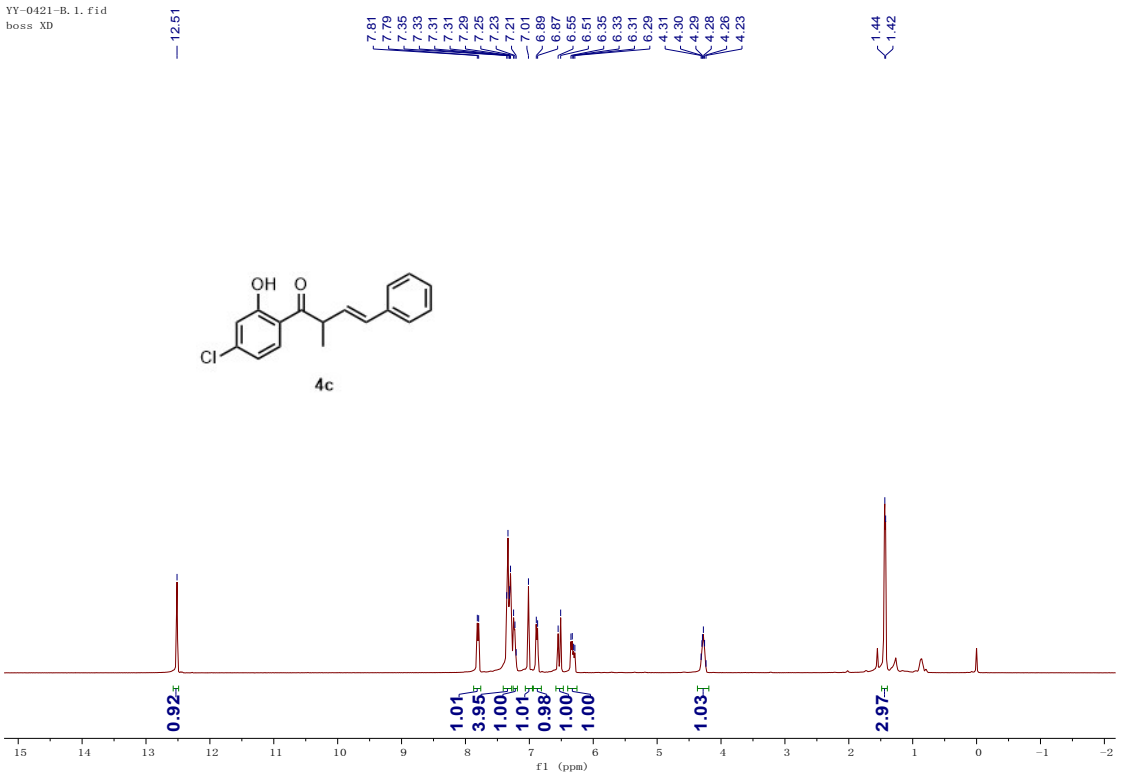
112.6 107.8 101.1

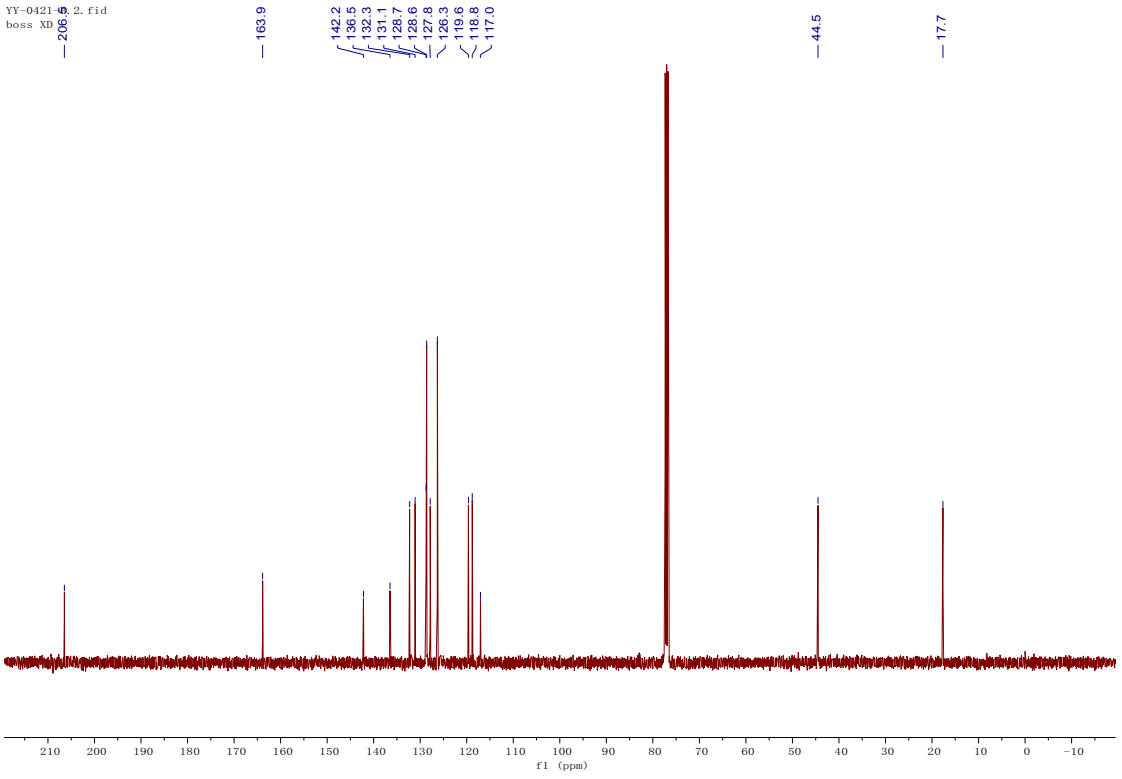
55.6 44.0 17.8

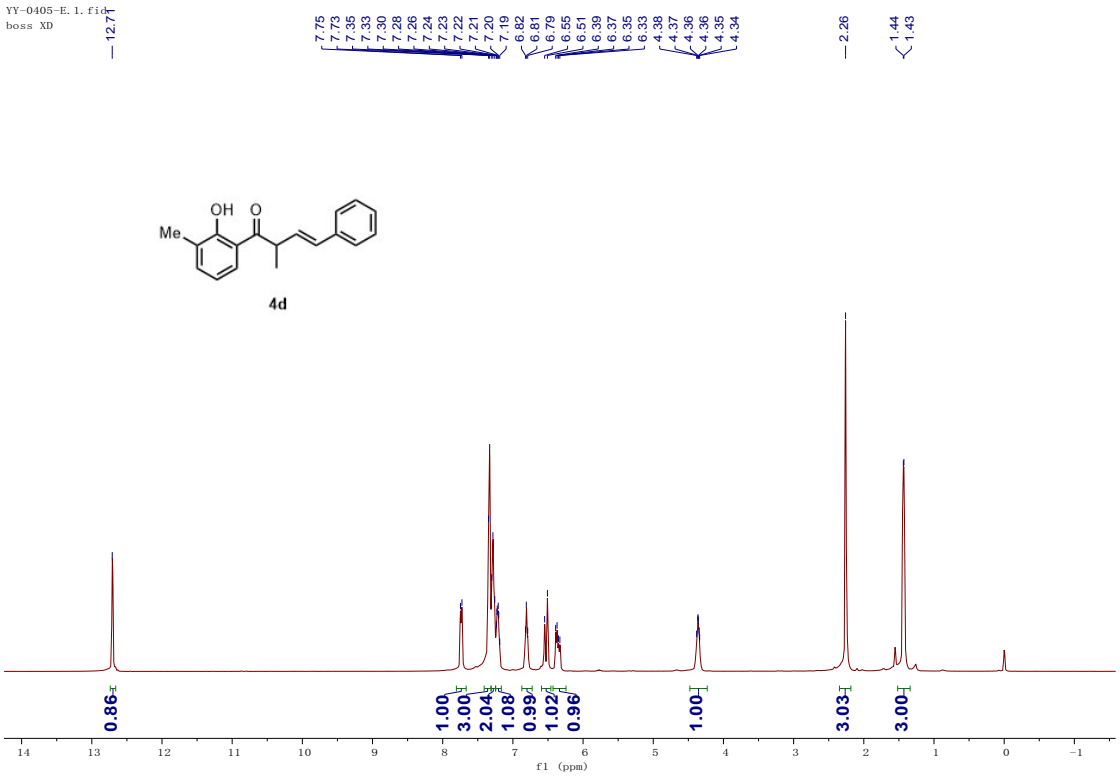


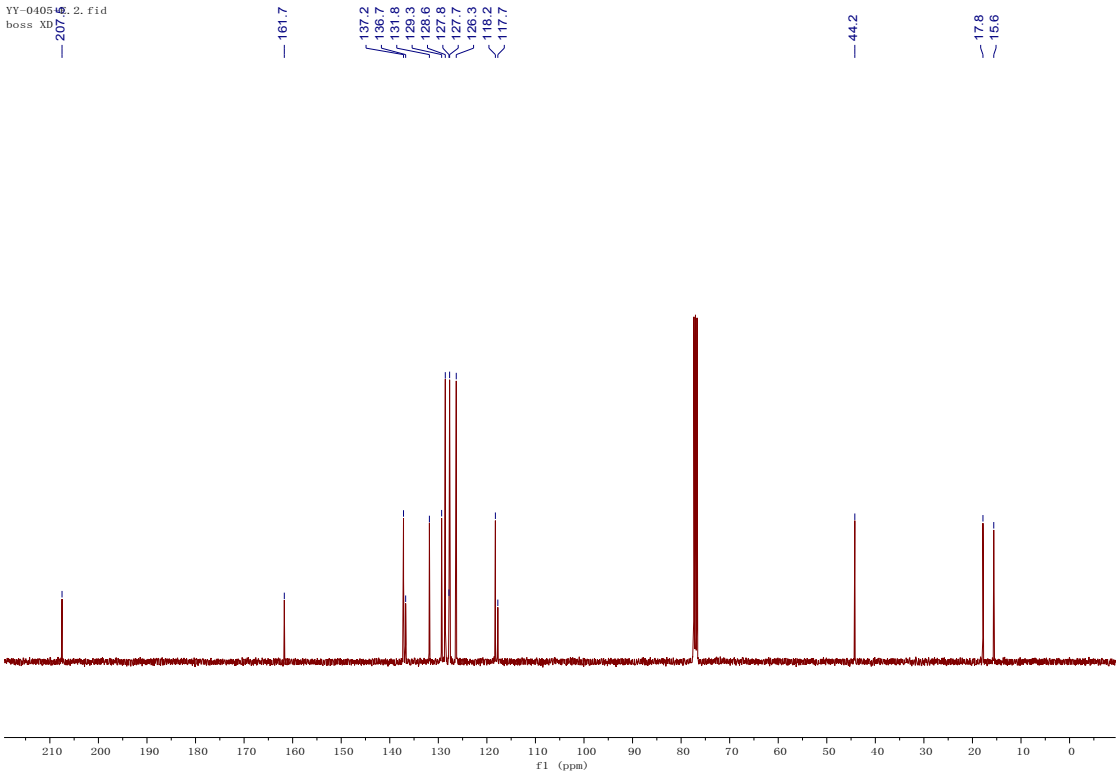

YY-0421-A.1.fid
boss XD
13.06
7.68
7.65
7.39
7.36
7.34
7.28
7.26
7.26
7.22
7.20
7.18
6.53
6.49
6.40
6.39
6.36
6.34
6.21
6.18
6.09
4.24
4.22
4.20
4.19
4.17
3.42
3.39
3.38
3.36
1.41
1.39
1.22
1.20
1.18
OH
O
Et
N
Et
4b
0.91
1.09
2.12
2.13
1.09
1.02
1.00
0.98
0.99
1.08
4.00
3.06
6.14
f1 (ppm)

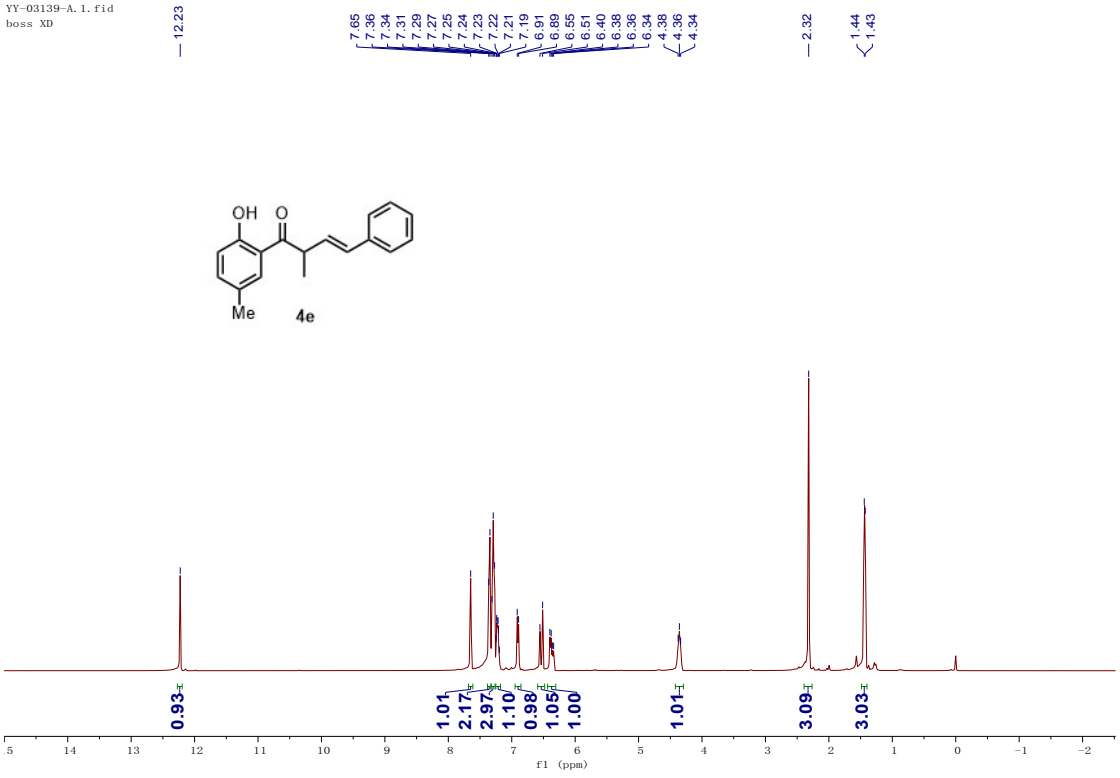

YY-0421-A.fid
boss XD
203.8
165.9
153.7
137.0
131.9
131.0
130.5
128.5
127.4
126.3
108.5
103.7
97.4
44.6
43.4
17.9
12.6
f1 (ppm)

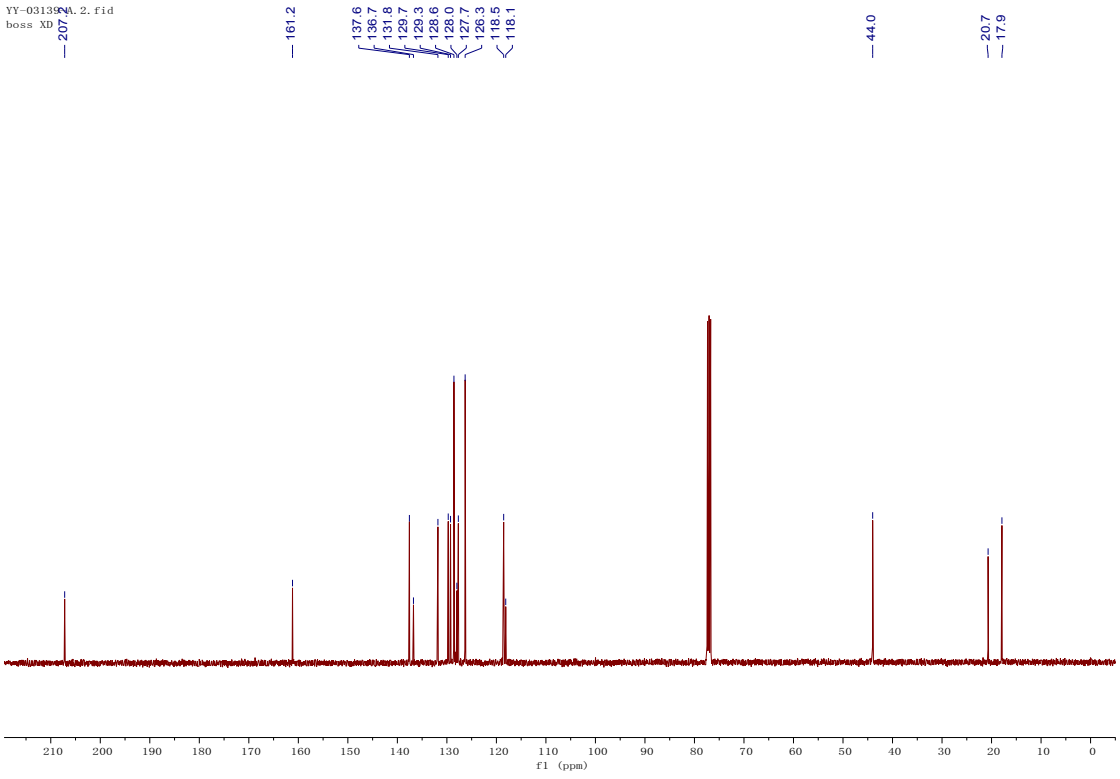

YY-0421-B.1.fid
boss XD
12.51
7.81
7.79
7.35
7.33
7.31
7.31
7.29
7.25
7.23
7.21
7.01
6.89
6.87
6.55
6.51
6.35
6.33
6.31
6.29
4.31
4.30
4.29
4.28
4.26
4.23
1.44
1.42
OH
O
Cl
4c
0.92
1.01
3.95
1.00
1.01
0.98
1.00
1.00
1.03
2.97
f1 (ppm)


YY-0421-B.2.fid
boss XD
206.5
163.9
142.2
136.5
132.3
131.1
128.7
128.6
127.8
126.3
119.6
118.8
117.0
44.5
17.7
f1 (ppm)

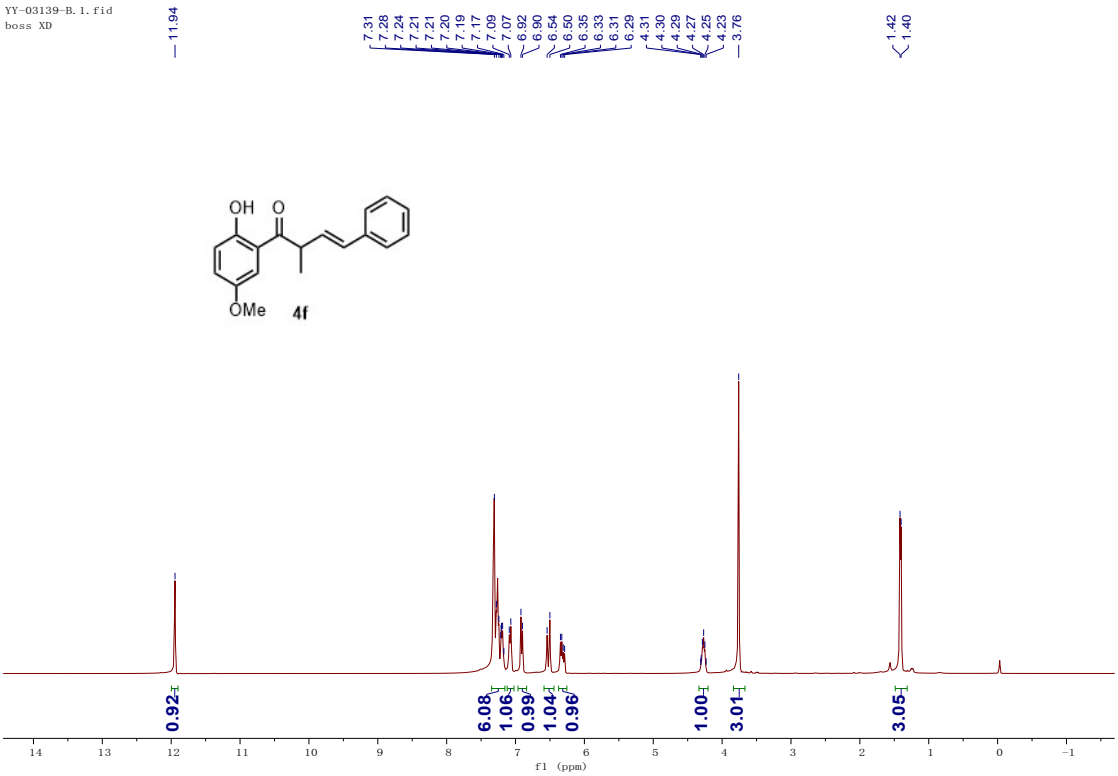

YY-0405-E.1.fid
boss XD
12.71
7.75
7.73
7.35
7.33
7.30
7.28
7.26
7.24
7.23
7.22
7.21
7.20
7.19
6.82
6.81
6.79
6.55
6.51
6.39
6.37
6.35
6.33
4.38
4.37
4.36
4.36
4.35
4.34
2.26
1.44
1.43
OH
O
Me
4d
0.86
1.00
3.00
2.04
1.08
0.99
1.02
0.96
1.00
3.03
3.00
f1 (ppm)


YY-0405-E.2.fid
boss XD
207.8
161.7
137.2
136.7
131.8
129.3
128.6
127.8
127.7
126.3
118.2
117.7
44.2
17.8
15.6
f1 (ppm)

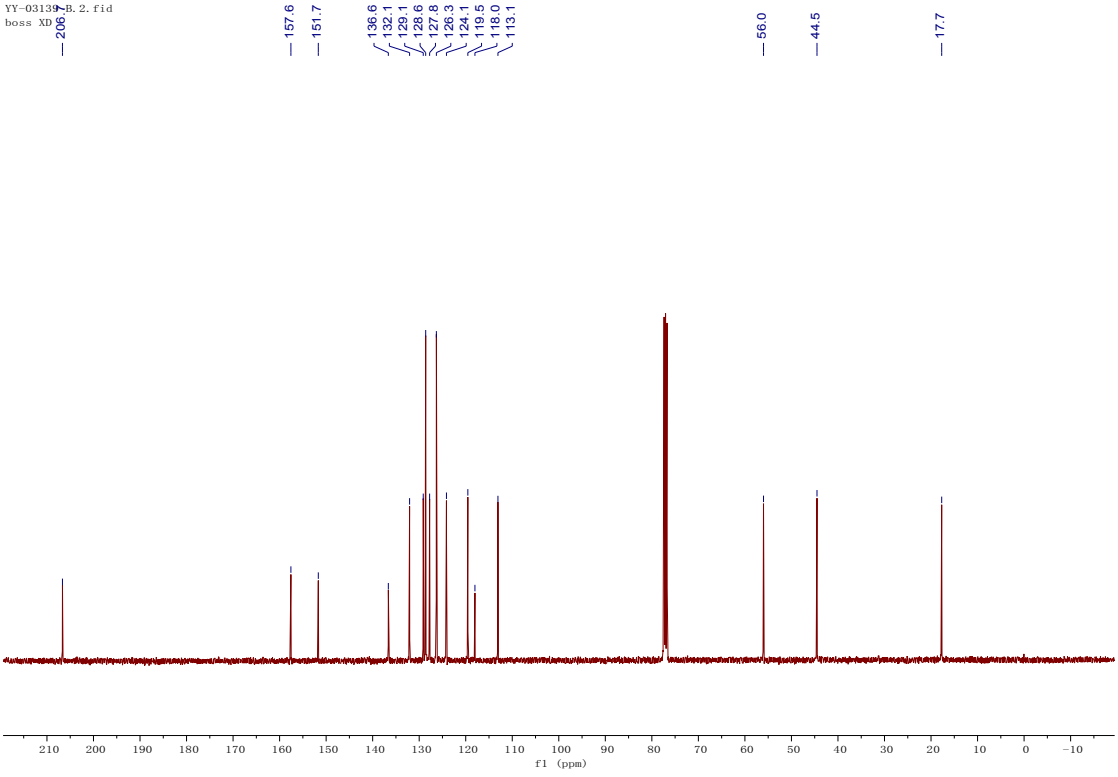

YY-03139-A.1.fid
boss XD
12.23
7.65
7.36
7.34
7.31
7.29
7.27
7.25
7.24
7.23
7.22
7.21
7.19
6.91
6.89
6.55
6.51
6.40
6.38
6.36
6.34
4.38
4.36
4.34
2.32
1.44
1.43
OH
O
Me
4e
0.93
1.01
2.17
2.97
1.10
0.98
1.05
1.00
1.01
3.09
3.03
f1 (ppm)

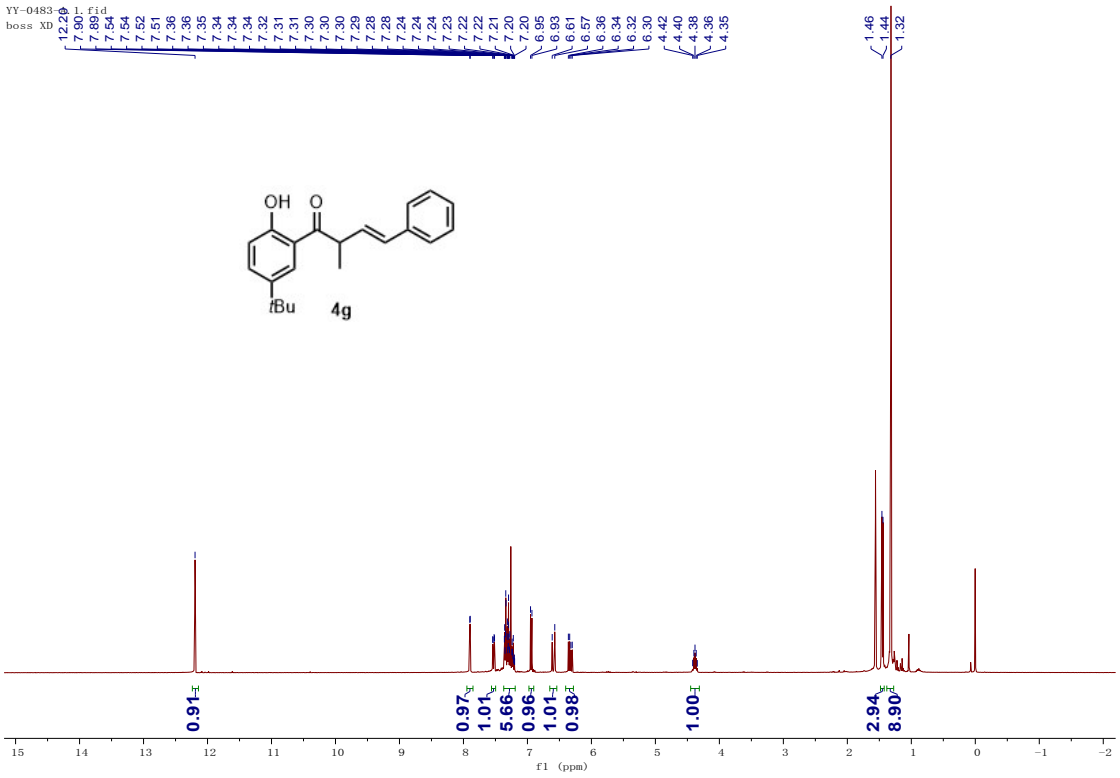

YY-03139-A.2.fid
boss XD
207.4
161.2
137.6
136.7
131.8
129.7
129.3
128.6
128.0
127.7
126.3
118.5
118.1
44.0
20.7
17.9
f1 (ppm)

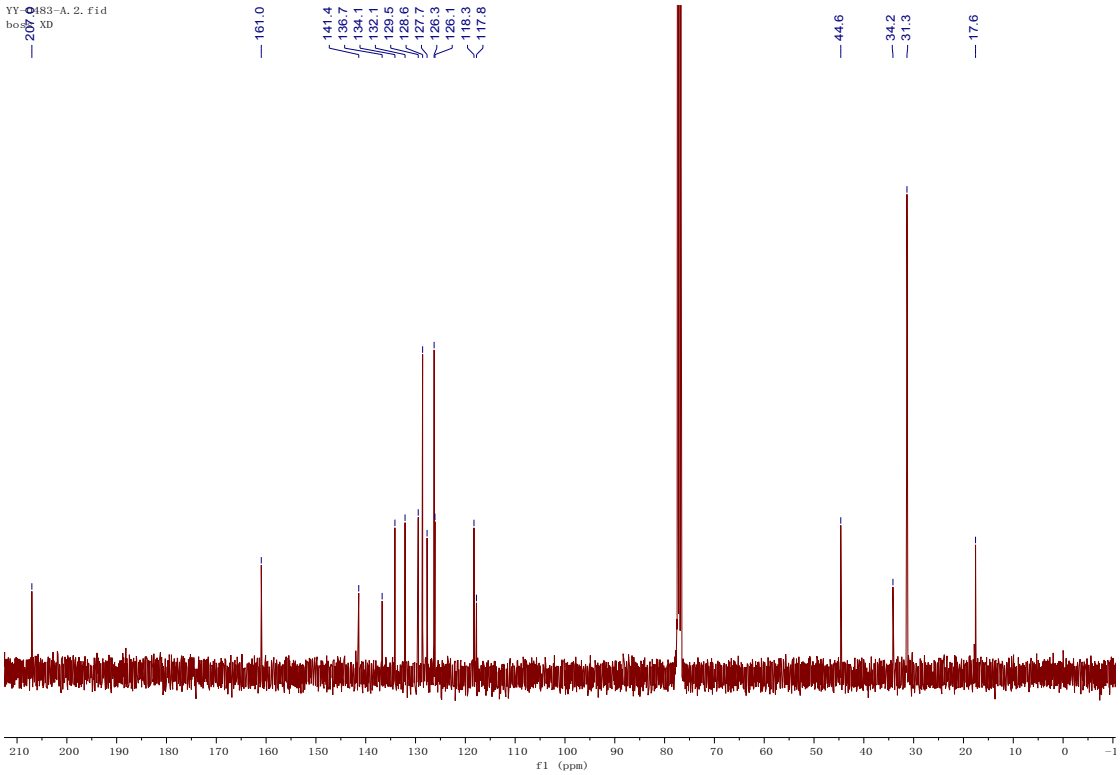
YY-03139-B.1.fid
boss XD

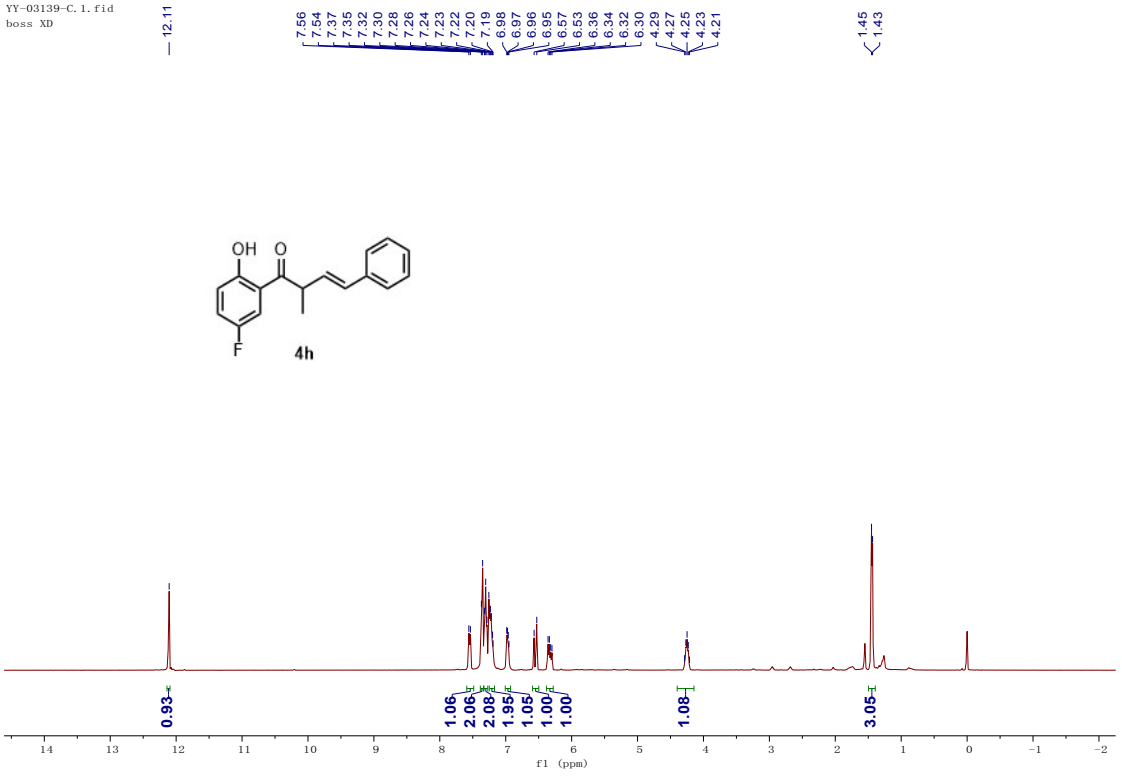


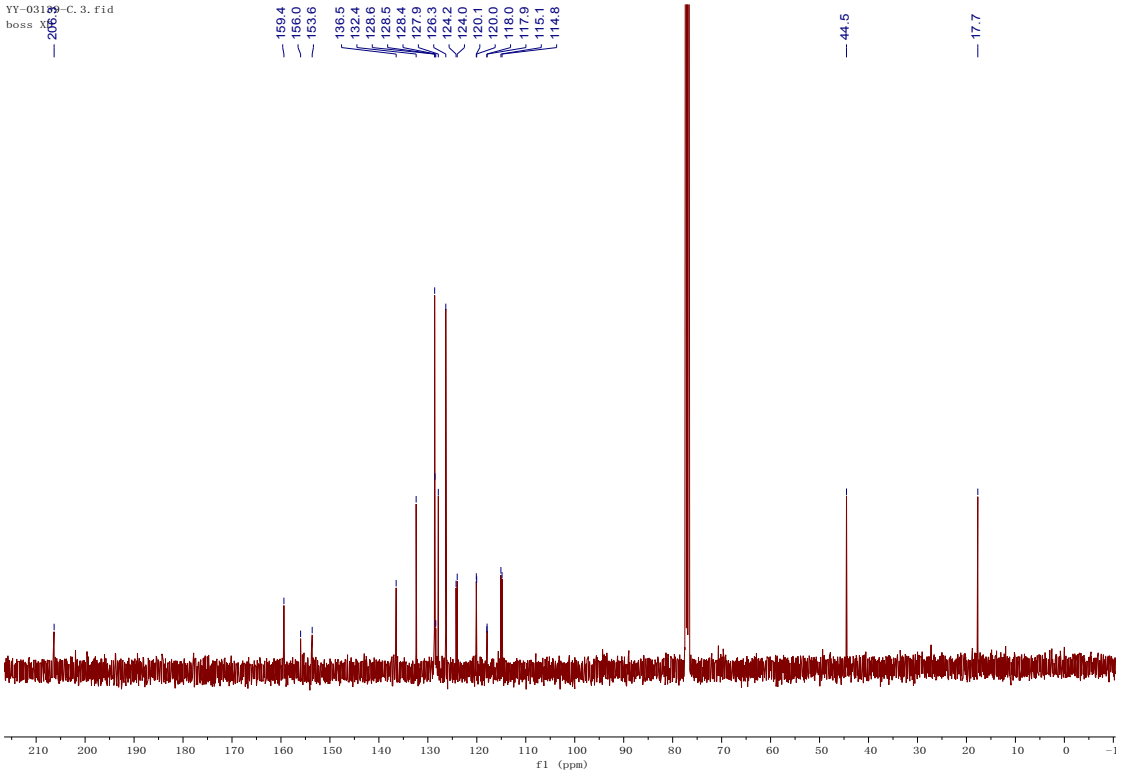
YY-03139-B.2.fid
boss XD



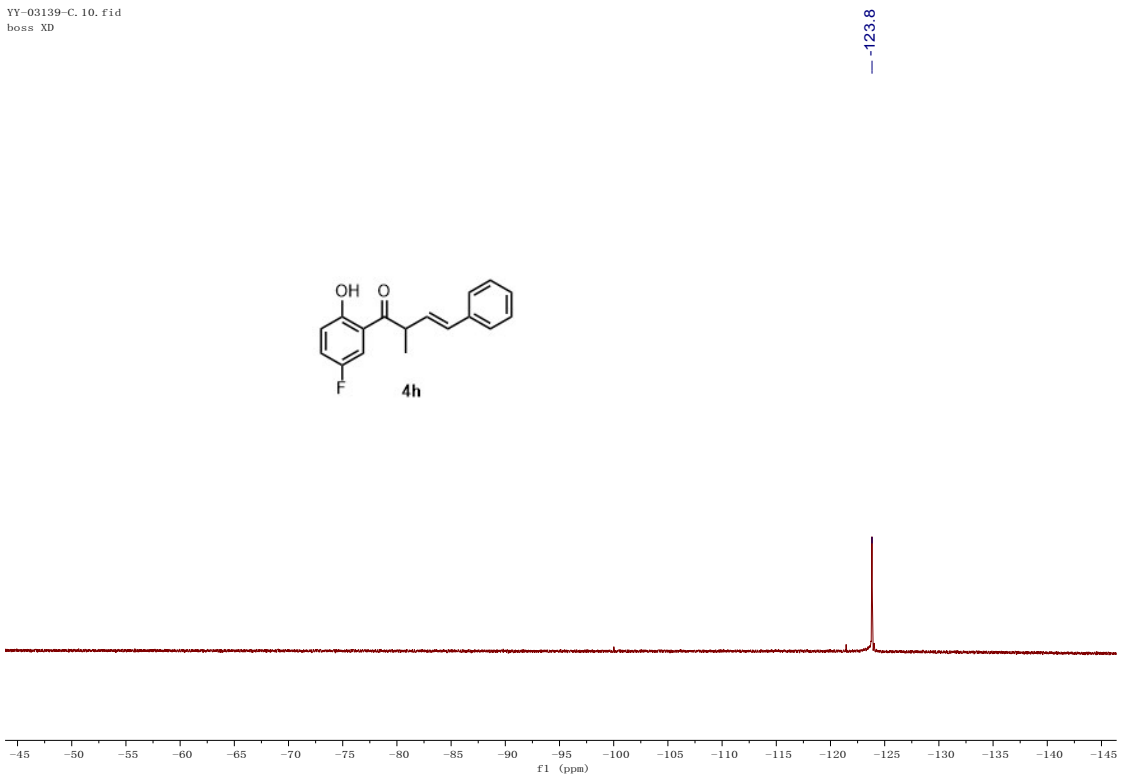

YY-0483-A.1.fid
boss XD
12.20
7.90
7.89
7.54
7.54
7.52
7.51
7.36
7.36
7.35
7.34
7.34
7.34
7.32
7.31
7.31
7.30
7.30
7.30
7.29
7.28
7.28
7.24
7.24
7.24
7.23
7.22
7.22
7.21
7.20
7.20
6.95
6.93
6.61
6.57
6.36
6.34
6.32
6.30
4.42
4.40
4.38
4.36
4.35
1.46
1.44
1.32
OH
O
tBu
4g
0.91
0.97
1.01
5.66
0.96
1.01
0.98
1.00
2.94
8.90
f1 (ppm)

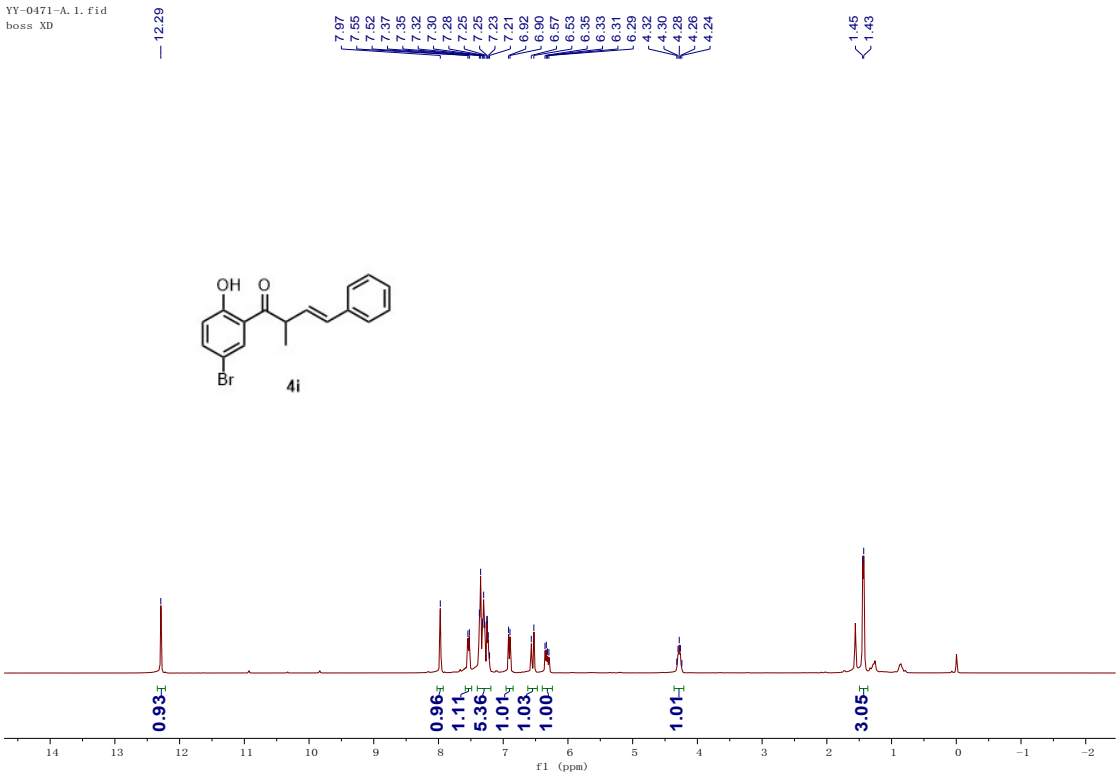

YY-0483-A.2.fid
boss XD
207.0
161.0
141.4
136.7
134.1
132.1
129.5
128.6
127.7
126.3
126.1
118.3
117.8
44.6
34.2
31.3
17.6
f1 (ppm)

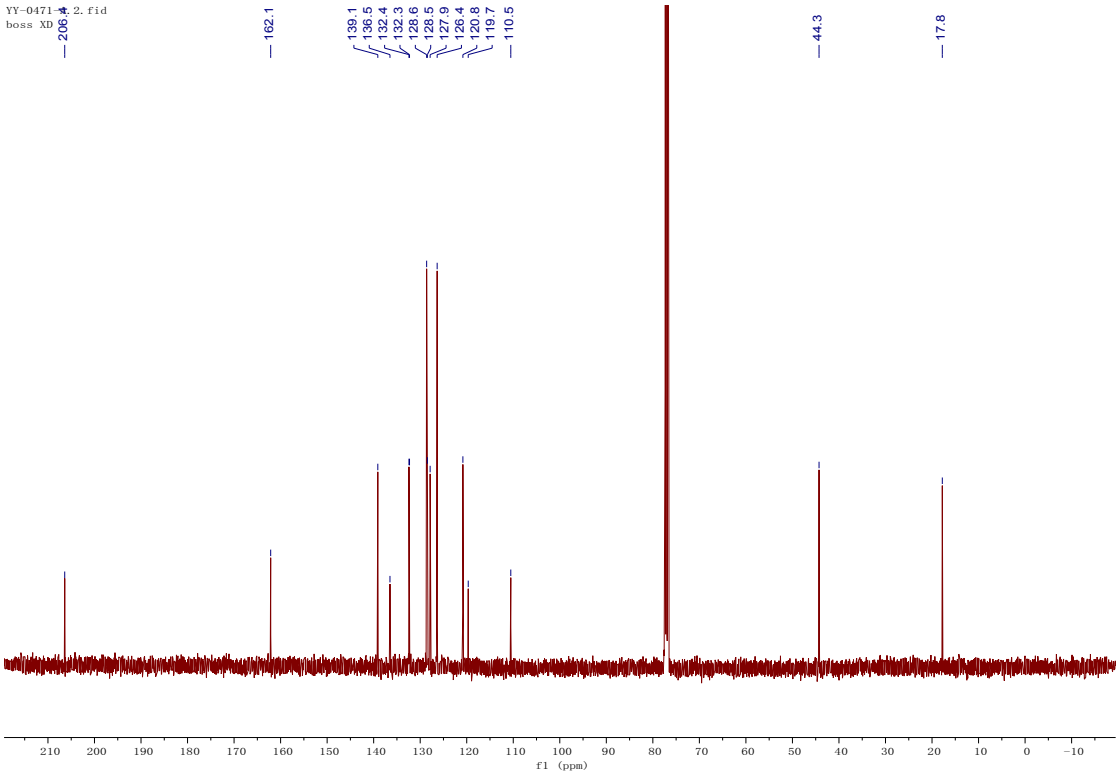

YY-03139-C.1.fid
boss XD
12.11
7.56
7.54
7.37
7.35
7.32
7.30
7.28
7.26
7.24
7.23
7.22
7.20
7.19
6.98
6.97
6.96
6.95
6.57
6.53
6.36
6.34
6.32
6.30
4.29
4.27
4.25
4.23
4.21
1.45
1.43
OH
O
F
4h
0.93
1.06
2.06
2.08
1.95
1.05
1.00
1.00
1.08
3.05
f1 (ppm)

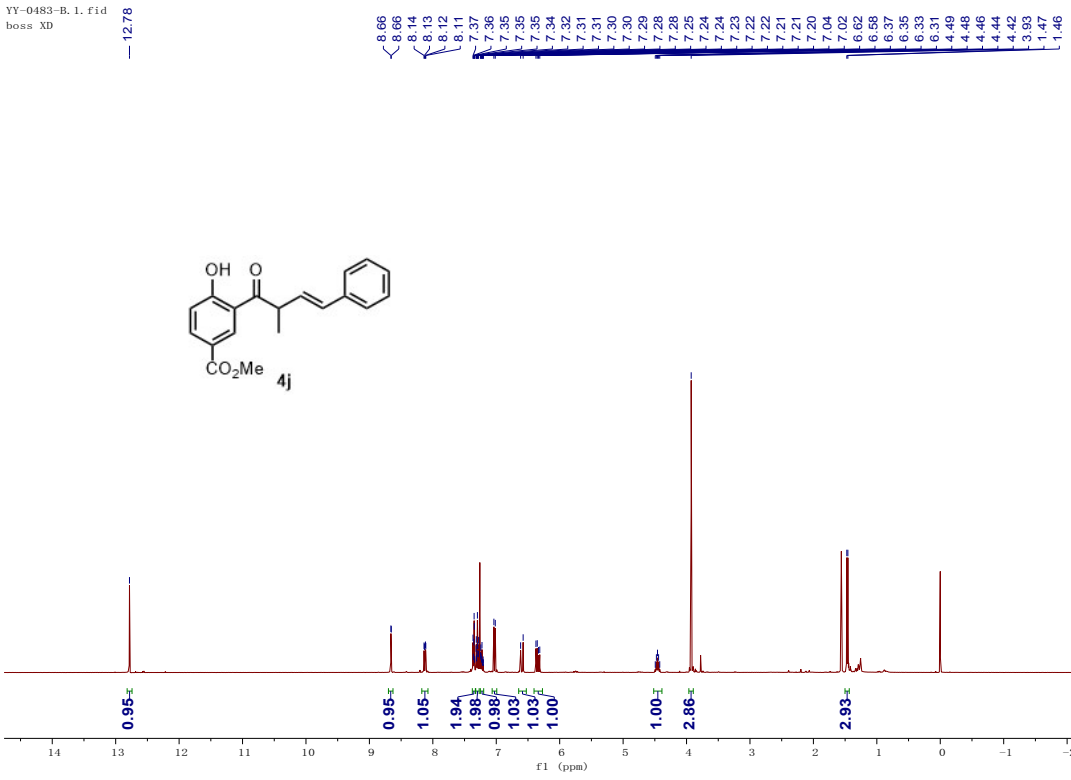

YY-03139-C.3.fid
boss XD
206.9
159.4
156.0
153.6
136.5
132.4
128.6
128.5
128.4
127.9
126.3
124.2
124.0
120.1
120.0
118.0
117.9
115.1
114.8
44.5
17.7
f1 (ppm)

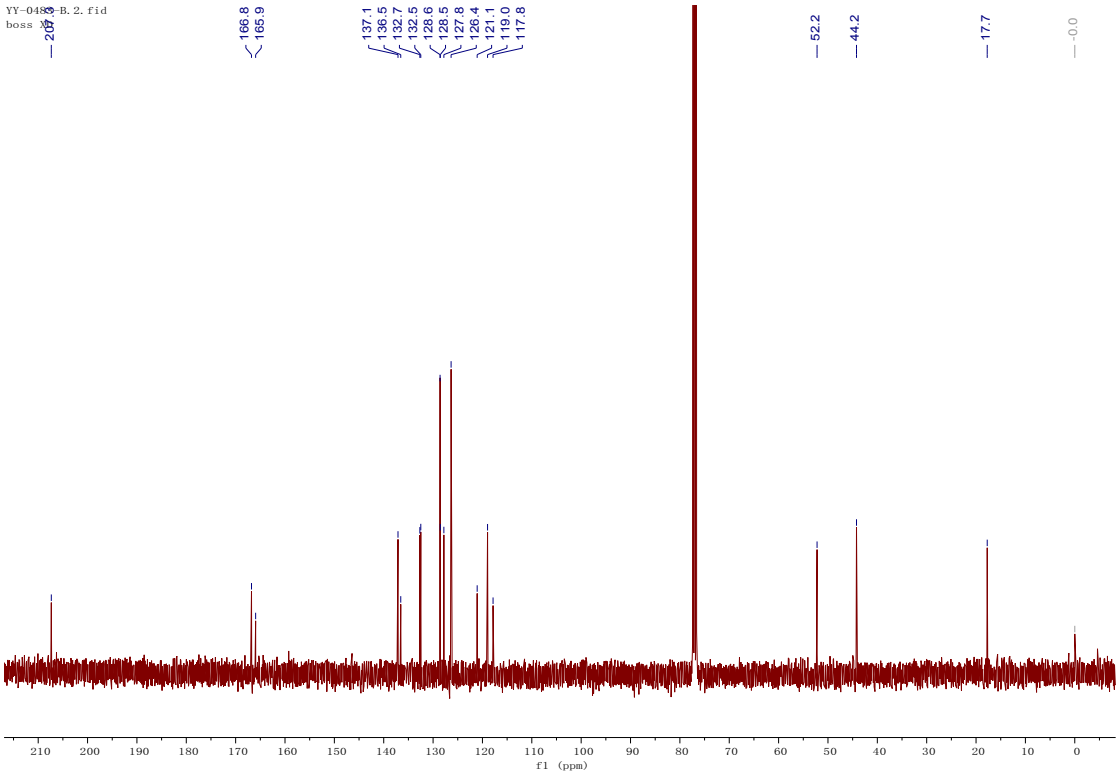
YY-03139-C.10.fid
boss XD

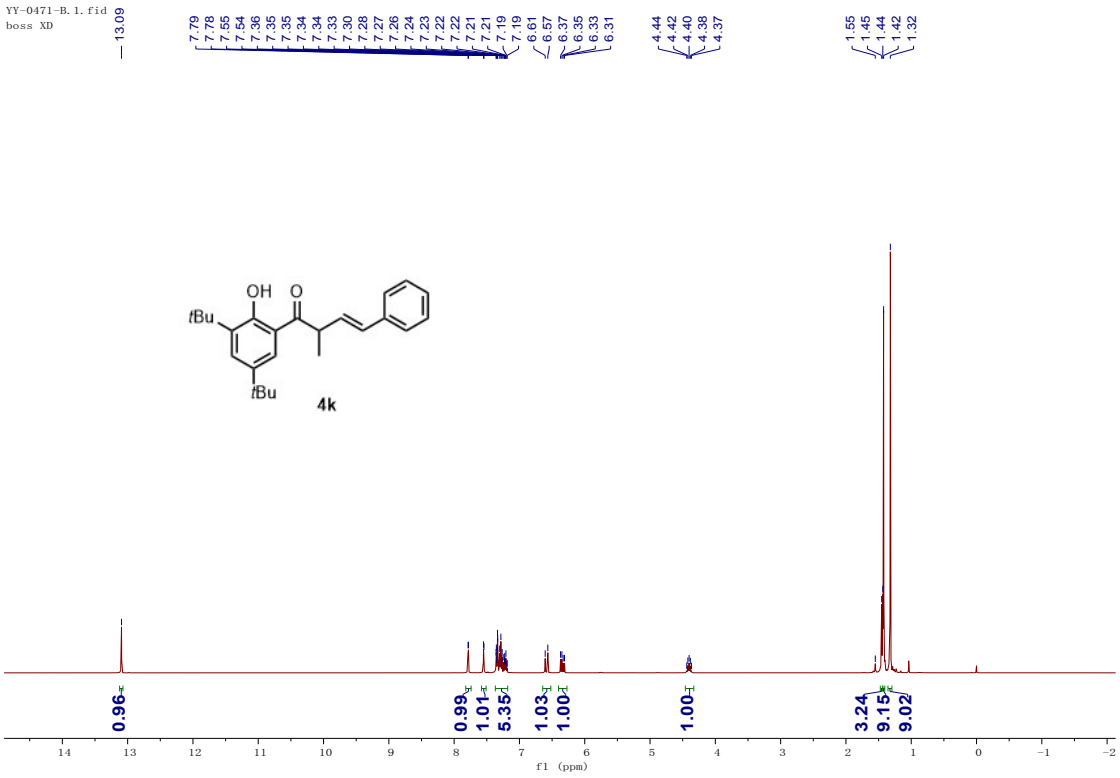


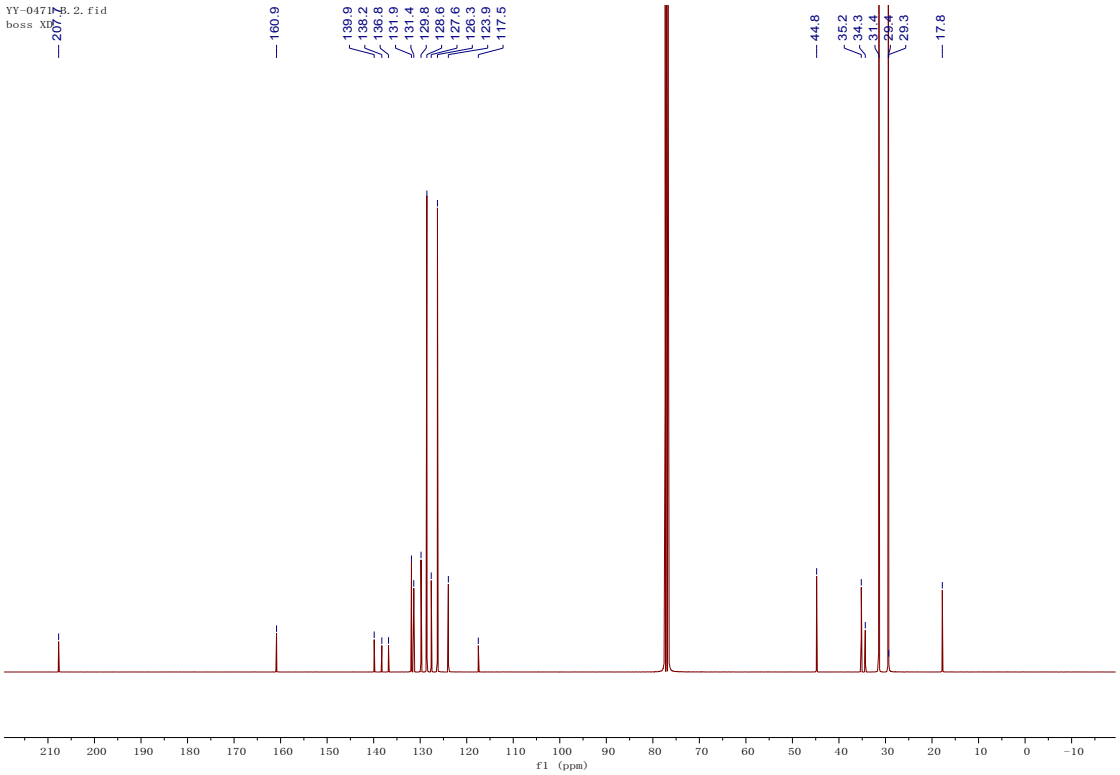

YY-0471-A.1.fid
boss XD
12.29
7.97
7.55
7.52
7.37
7.35
7.32
7.30
7.28
7.25
7.25
7.23
7.21
6.92
6.90
6.57
6.53
6.35
6.33
6.31
6.29
4.32
4.30
4.28
4.26
4.24
1.45
1.43
OH
O
Br
4i
0.93
0.96
1.11
5.36
1.01
1.03
1.00
1.01
3.05
f1 (ppm)

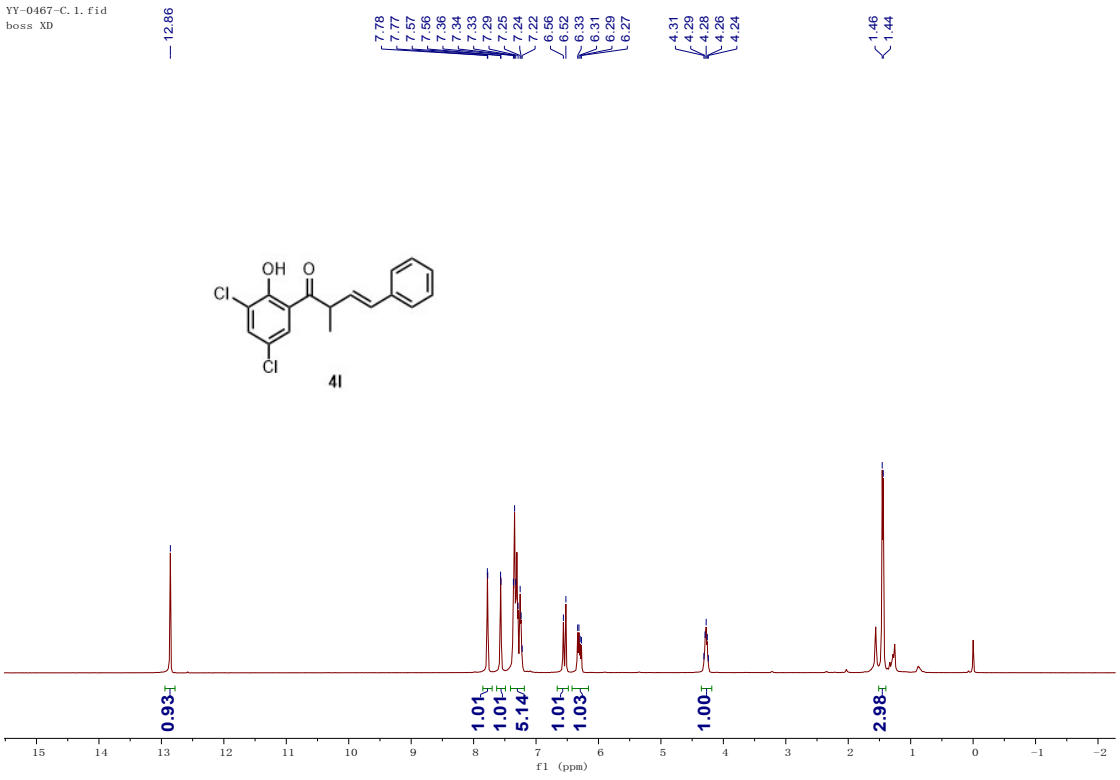

YY-0471-A.2.fid
boss XD
206.4
162.1
139.1
136.5
132.4
132.3
128.6
128.5
127.9
126.4
120.8
119.7
110.5
44.3
17.8
f1 (ppm)

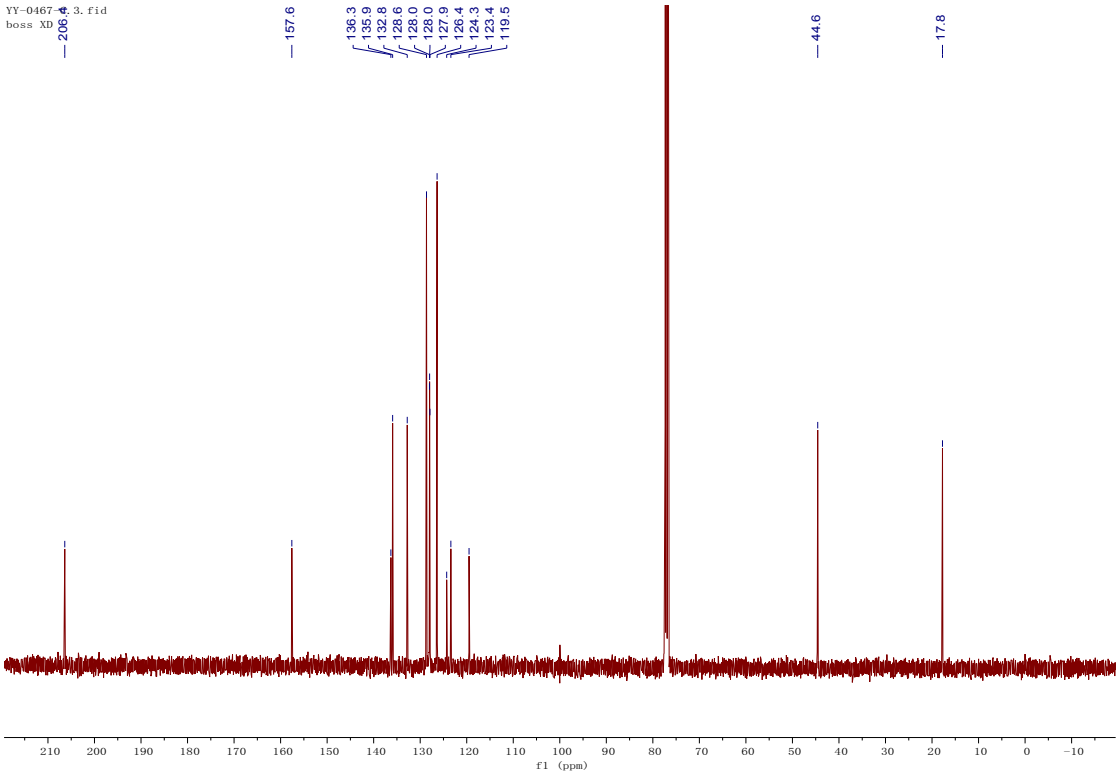

YY-0483-B.1.fid
boss XD
12.78
8.66
8.66
8.14
8.13
8.12
8.11
7.37
7.36
7.35
7.35
7.35
7.34
7.32
7.31
7.31
7.30
7.30
7.29
7.28
7.28
7.25
7.24
7.24
7.23
7.22
7.22
7.21
7.21
7.20
7.04
7.02
6.62
6.58
6.37
6.35
6.33
6.31
4.49
4.48
4.46
4.44
4.42
3.93
1.47
1.46
OH
O
CO2Me
4j
0.95
0.95
1.05
1.94
1.98
0.98
1.03
1.03
1.00
1.00
2.86
2.93
f1 (ppm)


YY-0483-B.2.fid
boss XD
166.8
165.9
137.1
136.5
132.7
132.5
128.6
128.5
127.8
126.4
121.1
119.0
117.8
52.2
44.2
17.7
f1 (ppm)

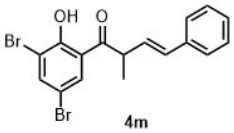

YY-0471-B.1.fid
boss XD
13.09
7.79 7.78 7.55 7.54 7.36 7.35 7.35 7.34 7.34 7.33 7.30 7.28 7.27 7.26 7.24 7.23 7.22 7.22 7.21 7.21 7.19 7.19 6.61 6.57 6.37 6.35 6.33 6.31
4.44 4.42 4.40 4.38 4.37
1.55 1.45 1.44 1.42 1.32
OH
O
tBu
tBu
4k
0.96 0.99 1.01 5.35 1.03 1.00 1.00 3.24 9.15 9.02
f1 (ppm)


YY-0471-B.2.fid
boss XD
207.1
160.9
139.9 138.2 136.8 131.9 131.4 129.8 128.6 127.6 126.3 123.9 117.5
44.8
35.2 34.3 31.4 29.4 29.3
17.8
f1 (ppm)

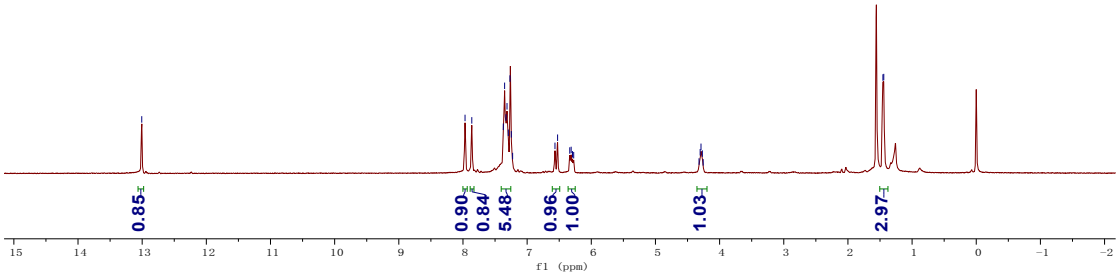

YY-0467-C.1.fid
boss XD
12.86
7.78
7.77
7.57
7.56
7.36
7.34
7.33
7.29
7.25
7.24
7.22
6.56
6.52
6.33
6.31
6.29
6.27
4.31
4.29
4.28
4.26
4.24
1.46
1.44
OH
O
Cl
Cl
4l
0.93
1.01
1.01
5.14
1.01
1.03
1.00
2.98
f1 (ppm)

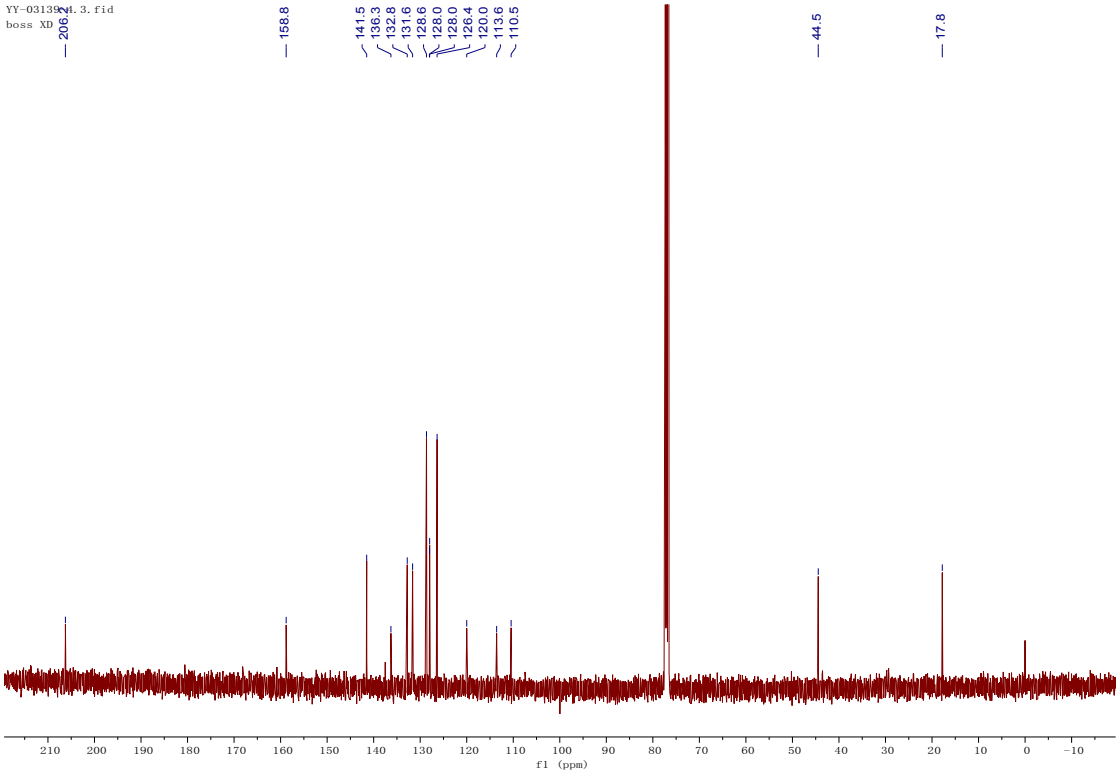

YY-0467-C.3.fid
boss XD
206.4
157.6
136.3
135.9
132.8
128.6
128.0
128.0
127.9
126.4
124.3
123.4
119.5
44.6
17.8
f1 (ppm)

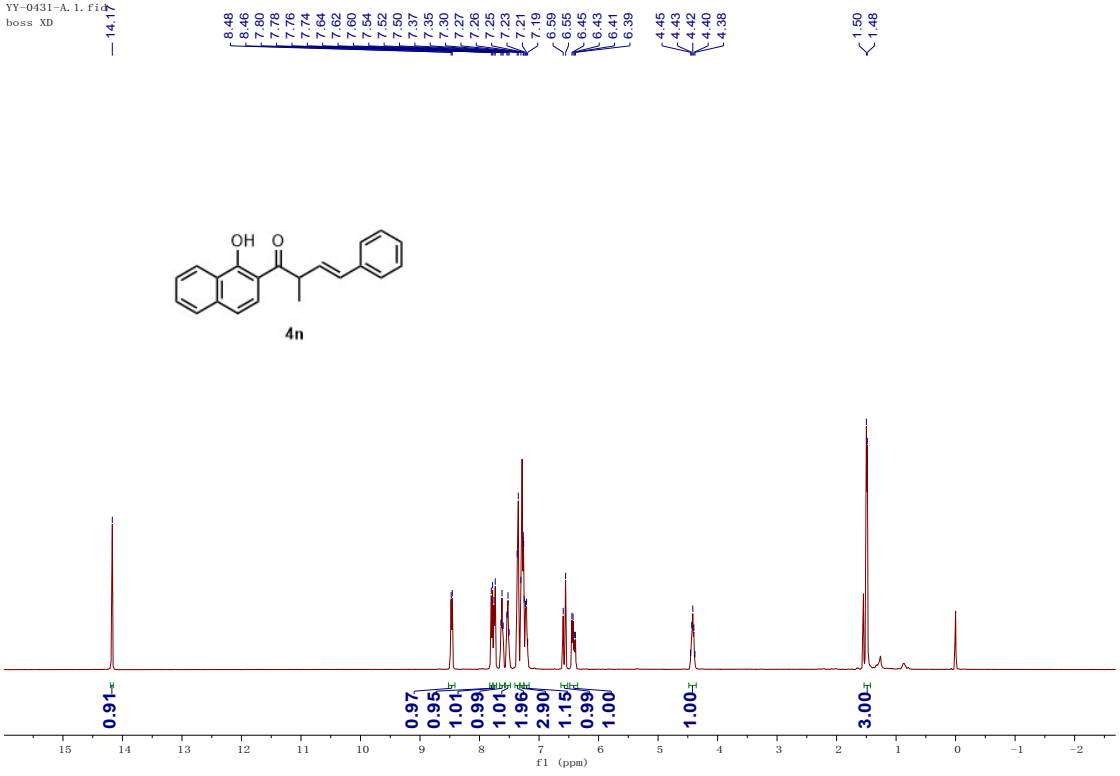

YY-03139-4.1.fid
boss XD
13.01
7.97
7.86
7.37
7.35
7.33
7.31
7.29
7.27
7.25
7.23
6.57
6.53
6.34
6.31
6.29
6.27
4.32
4.30
4.29
4.27
4.26
1.45
1.44

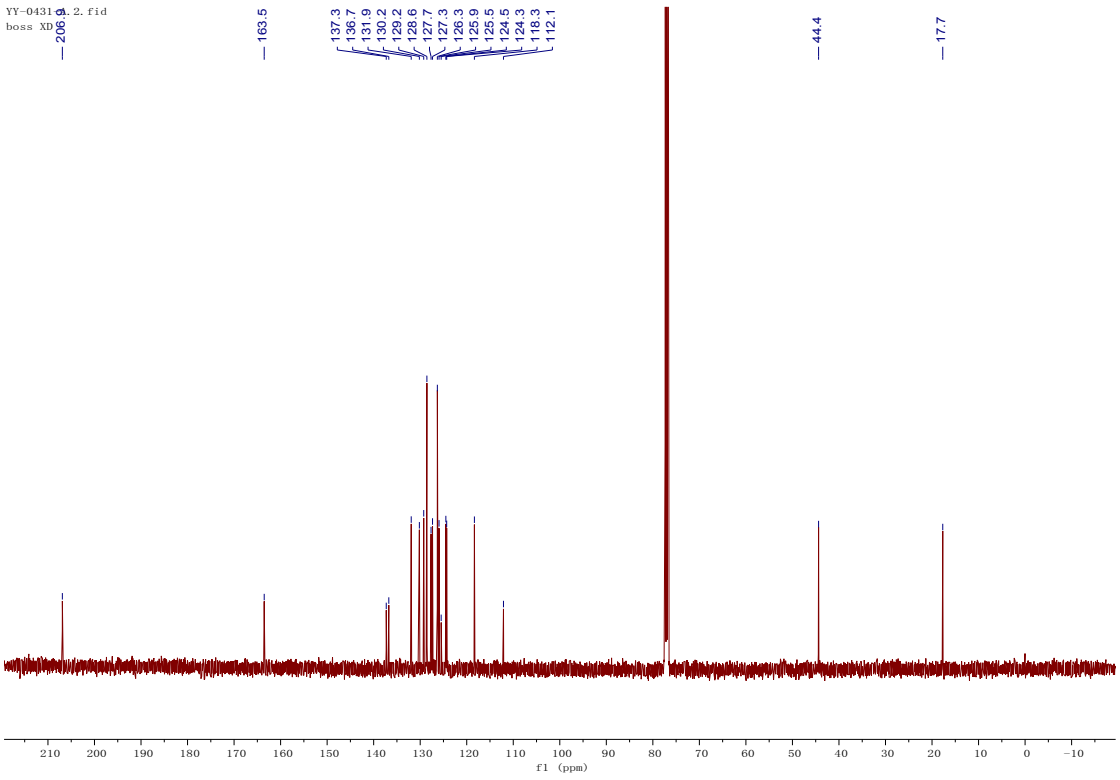

OH
O
Br
Br

4m


0.85
0.90
0.84
5.48
0.96
1.00
1.03
2.97
f1 (ppm)

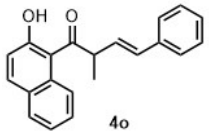

YY-03139-4.3.fid
boss XD
206.4
158.8
141.5
136.3
132.8
131.6
128.6
128.0
128.0
126.4
120.0
113.6
110.5
44.5
17.8
f1 (ppm)


YY-0431-A.1.fid
boss XD
14.17
8.48
8.46
7.80
7.78
7.76
7.74
7.64
7.62
7.60
7.54
7.52
7.50
7.37
7.35
7.30
7.27
7.26
7.25
7.23
7.21
7.19
6.59
6.55
6.45
6.43
6.41
6.39
4.45
4.43
4.42
4.40
4.38
1.50
1.48
OH
O
4n
0.91
0.97
0.95
1.01
0.99
1.01
1.96
2.90
1.15
0.99
1.00
1.00
3.00
f1 (ppm)

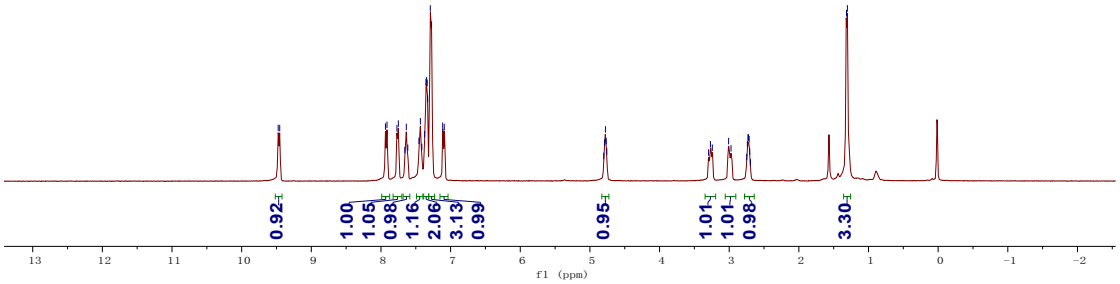

YY-0431-A.2.fid
boss XD
206.1
163.5
137.3
136.7
131.9
130.2
129.2
128.6
127.7
127.3
126.3
125.9
125.5
124.5
124.3
118.3
112.1
44.4
17.7
f1 (ppm)

YY-0421-C(X).1.fid
boss XD

9.48 9.45 7.94 7.91 7.77 7.75 7.66 7.64 7.61 7.45 7.43 7.41 7.37 7.35 7.34 7.34 7.33 7.29 7.27 7.11 7.09 4.80 4.79 4.78 4.77 4.76 3.30 3.27 3.24 3.01 2.97 2.75 2.74 2.73 2.72 2.71 2.71 2.69 1.32 1.30

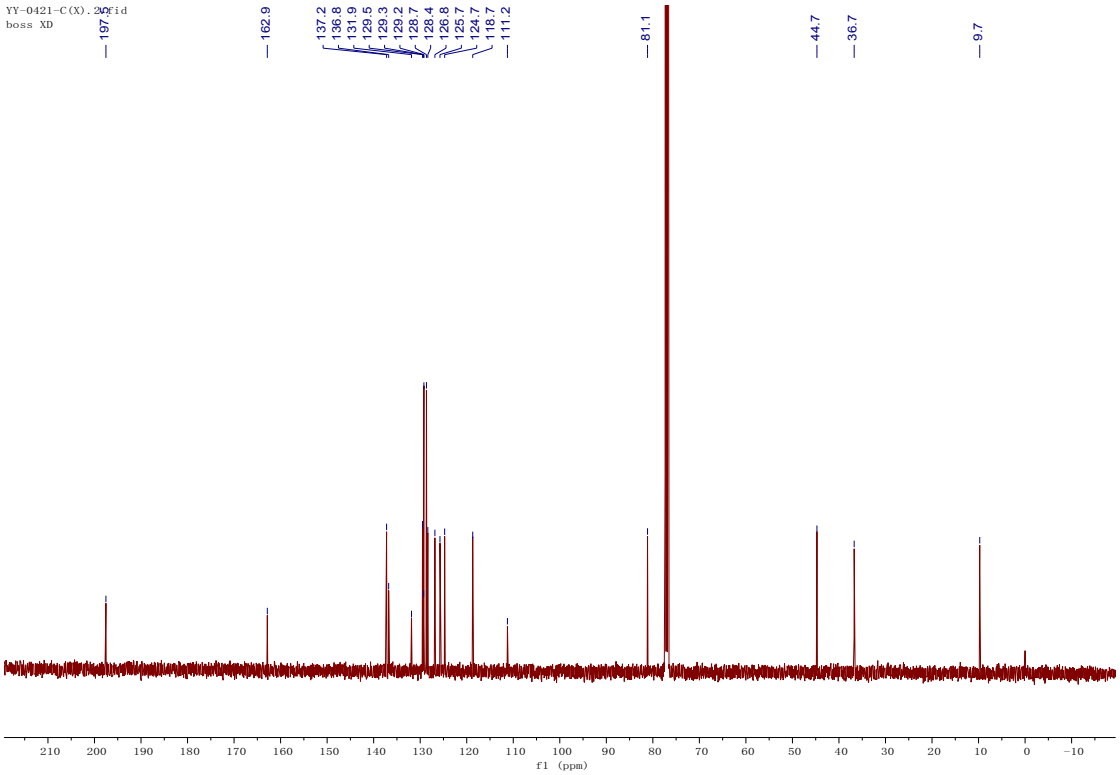


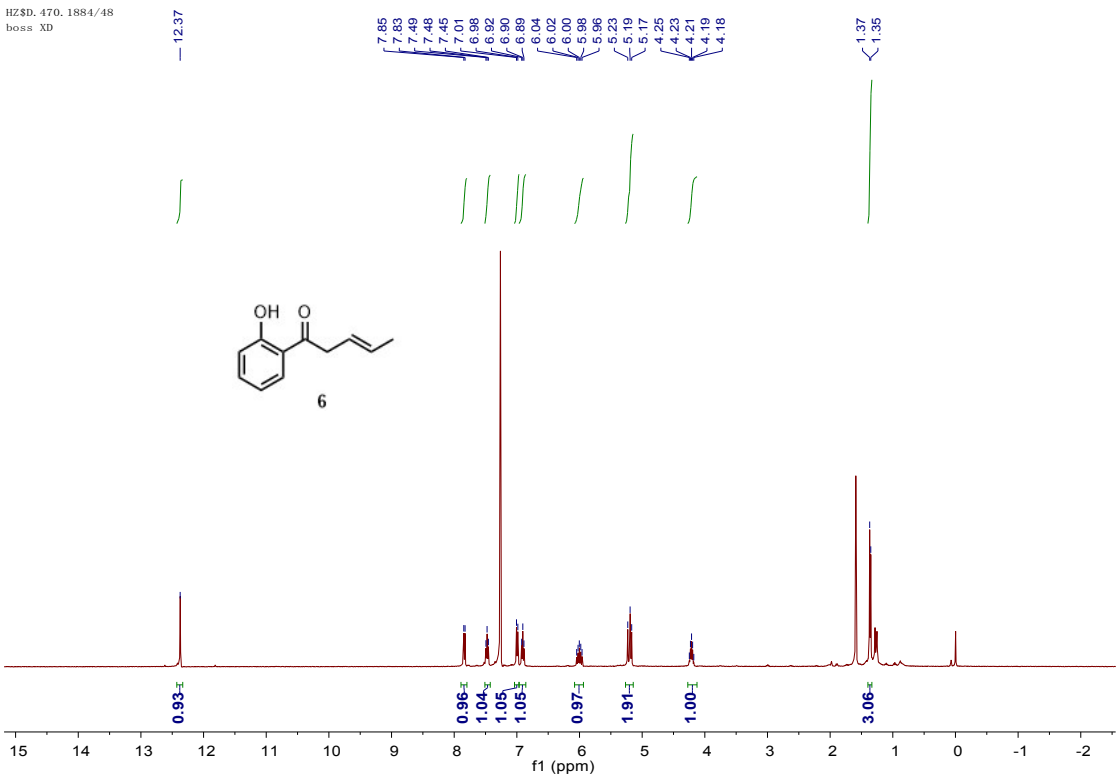
4o

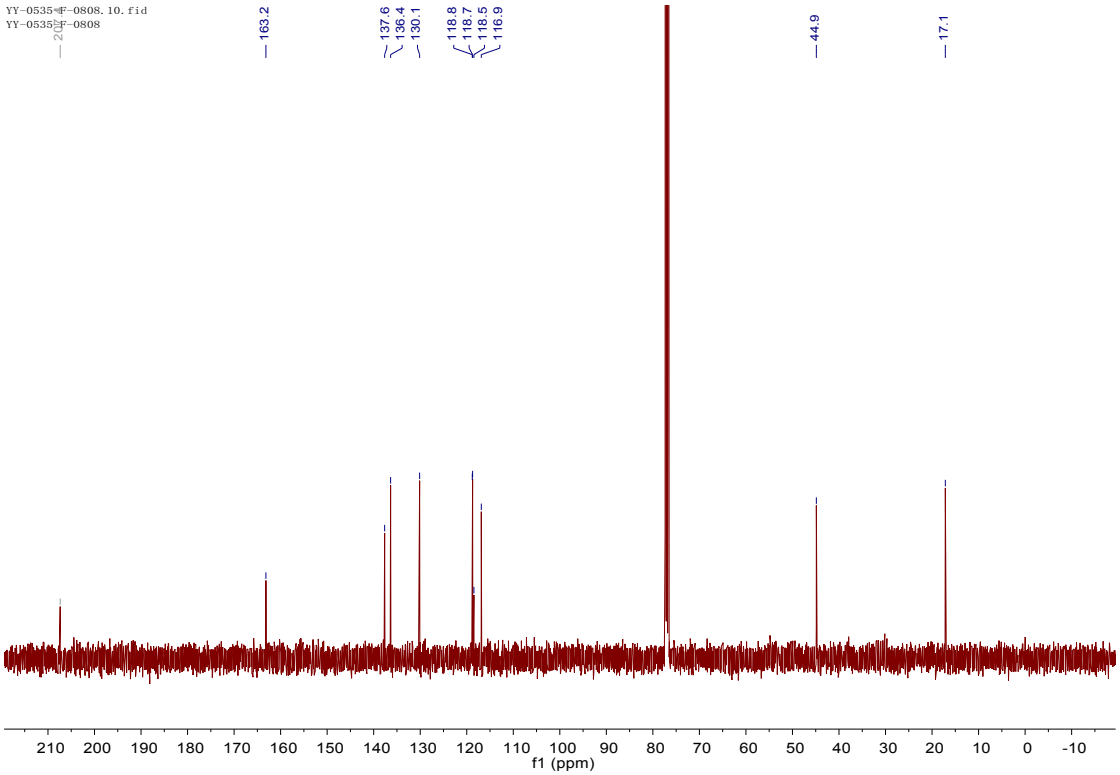


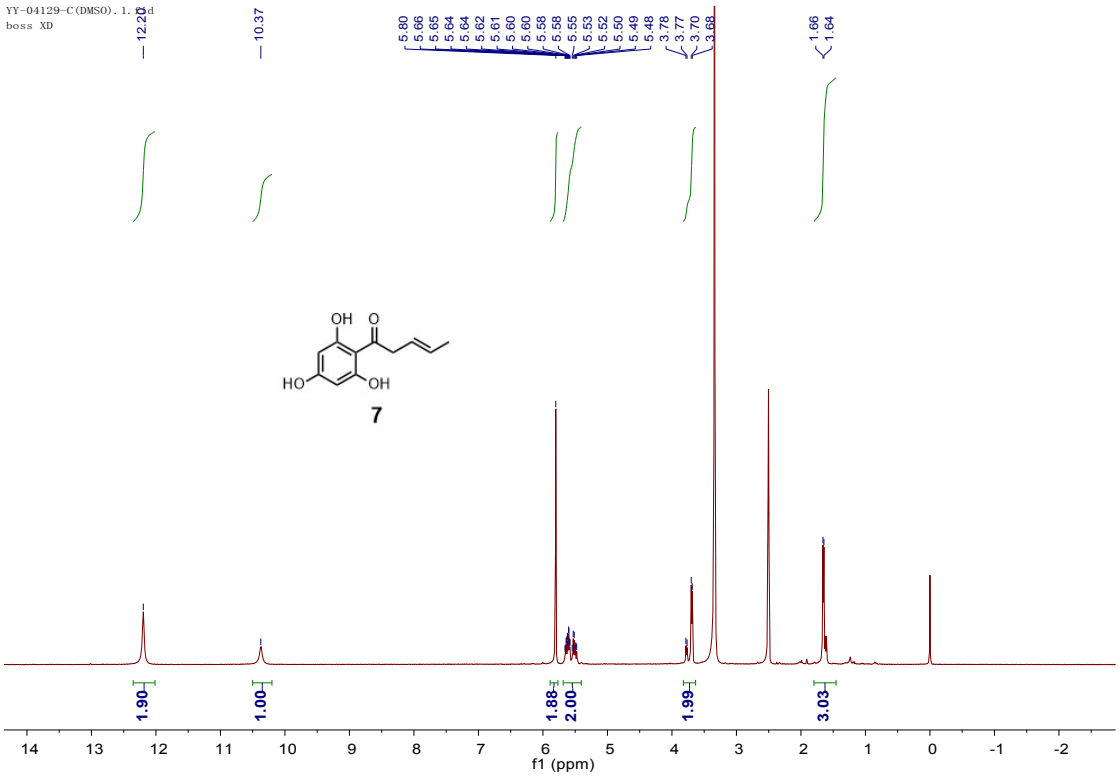
YY-0421-C(X).2.fid
boss XD

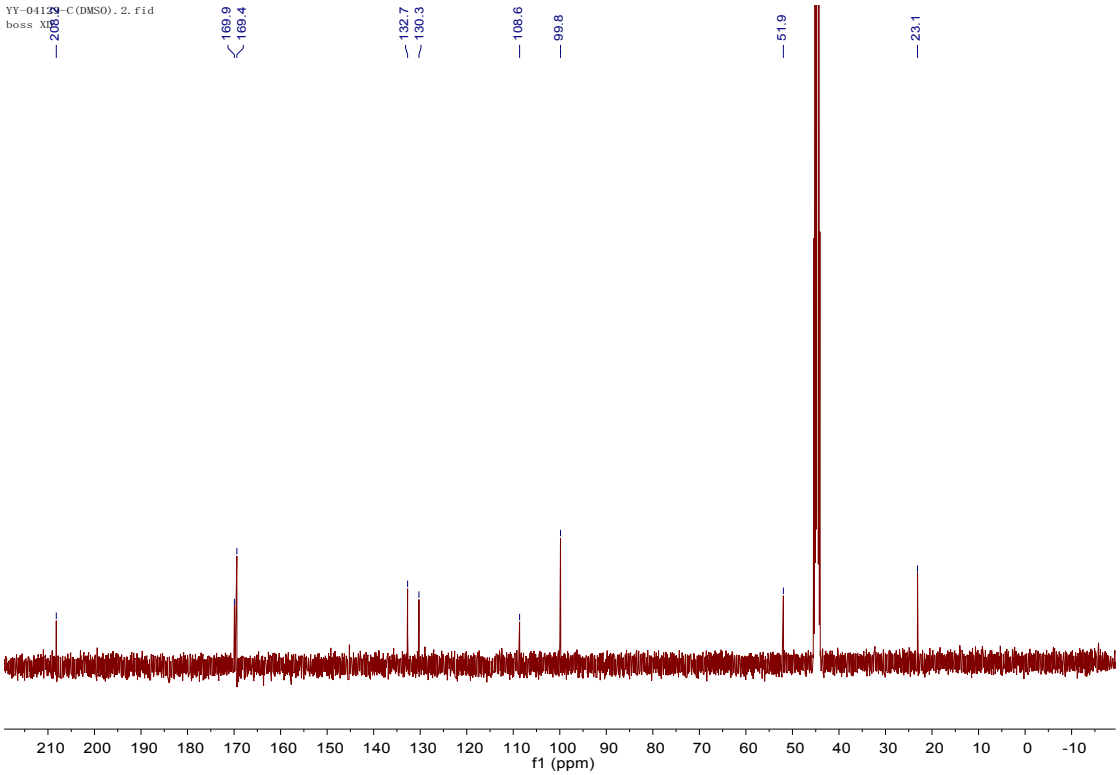
197.5 162.9 137.2 136.8 131.9 129.5 129.3 129.2 128.7 128.4 126.8 125.7 124.7 118.7 111.2 81.1 44.7 36.7 9.7

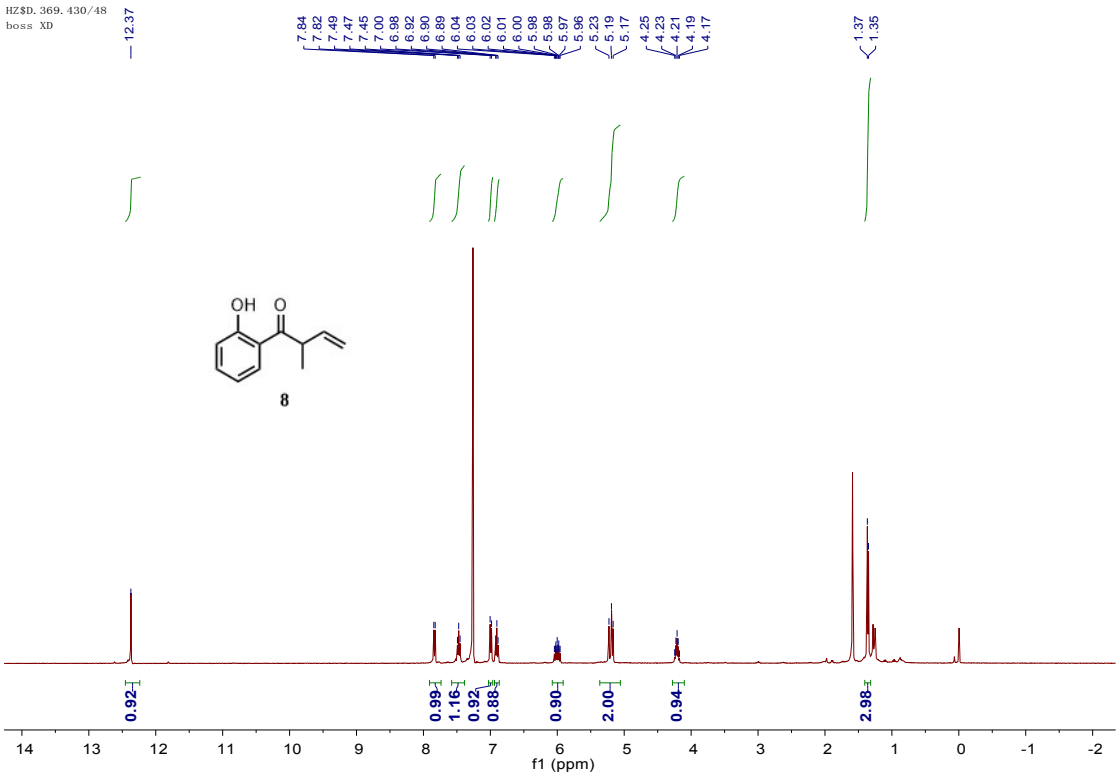


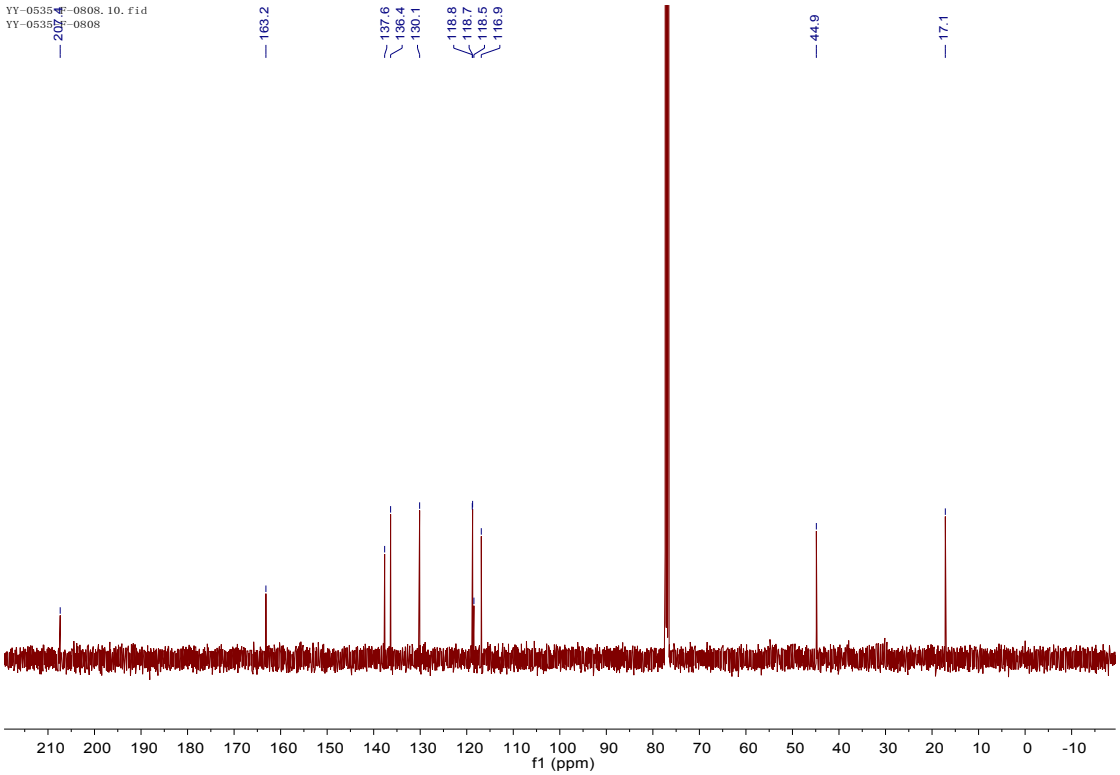

HZ$D.470.1884/48
boss XD
12.37
7.85
7.83
7.49
7.48
7.45
7.01
6.98
6.92
6.90
6.89
6.04
6.02
6.00
5.98
5.96
5.23
5.19
5.17
4.25
4.23
4.21
4.19
4.18
1.37
1.35
OH
O
6
0.93
0.96
1.04
1.05
1.05
0.97
1.91
1.00
3.06
f1 (ppm)


YY-0535-F-0808.10.fid
YY-0535-F-0808
163.2
137.6
136.4
130.1
118.8
118.7
118.5
116.9
44.9
17.1
f1 (ppm)

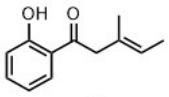

YY-04129-C(DMSO).1.fid
boss XD
12.20
10.37
5.80
5.66
5.65
5.64
5.64
5.62
5.61
5.60
5.60
5.58
5.58
5.55
5.53
5.52
5.50
5.49
5.48
3.78
3.77
3.70
3.68
1.66
1.64
OH
O
HO
OH
7
1.90
1.00
1.88
2.00
1.99
3.03
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
f1 (ppm)


YY-04129-C(DMSO).2.fid
boss XD
205.2
169.9
169.4
132.7
130.3
108.6
99.8
51.9
23.1
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)

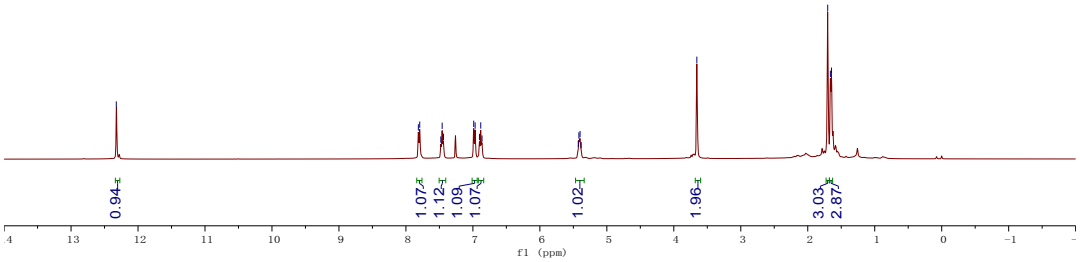

HZ$D.369.430/48
boss XD
12.37
7.84
7.82
7.49
7.47
7.45
7.00
6.98
6.92
6.90
6.89
6.04
6.03
6.02
6.01
6.00
5.98
5.98
5.97
5.96
5.23
5.19
5.17
4.25
4.23
4.21
4.19
4.17
1.37
1.35
OH
O
8
0.92
0.99
1.16
0.92
0.88
0.90
2.00
0.94
2.98
f1 (ppm)


YY-0535-F-0808.10.fid
YY-0535-F-0808
204.4
163.2
137.6
136.4
130.1
118.8
118.7
118.5
116.9
44.9
17.1
f1 (ppm)

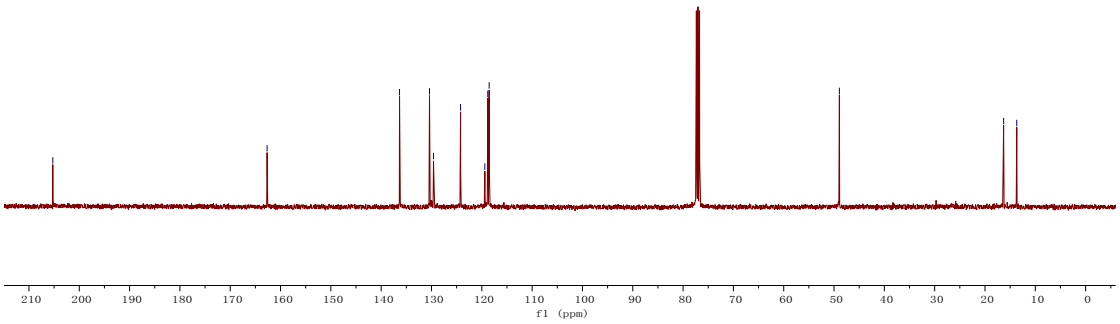

YY-0764-A. 10. fid
boss XD
12.3
7.8
7.8
7.5
7.5
7.4
7.0
7.0
6.9
6.9
6.9
5.4
5.4
5.4
5.4
3.7
1.7
1.7
1.6

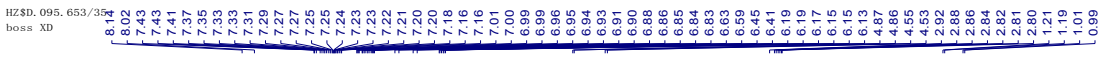

OH
O

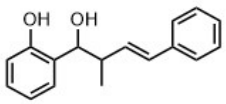
10


0.94
1.07
1.12
1.09
1.07
1.02
1.96
3.03
2.87
f1 (ppm)


YY-0764-A. 11. fid
boss XD
205.3
162.7
136.4
130.4
129.6
124.2
119.4
118.8
118.5
48.9
16.3
13.7

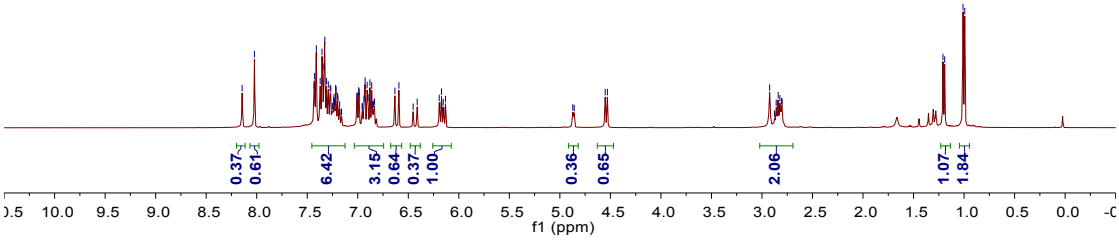

f1 (ppm)

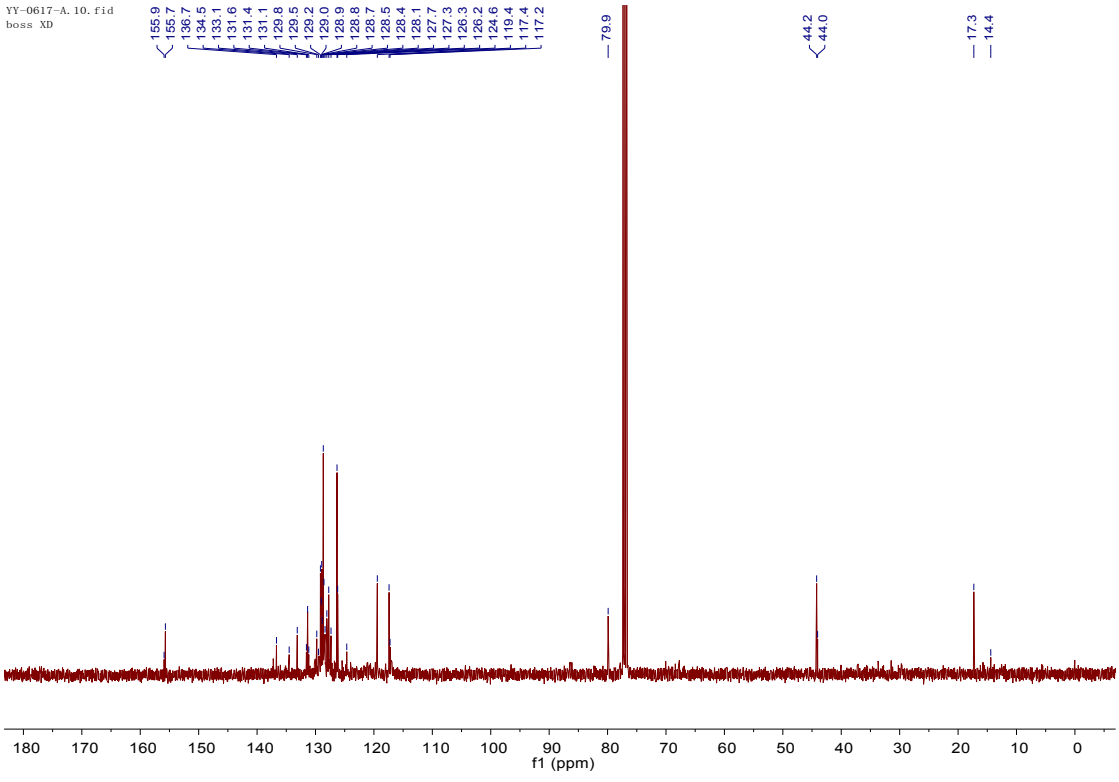


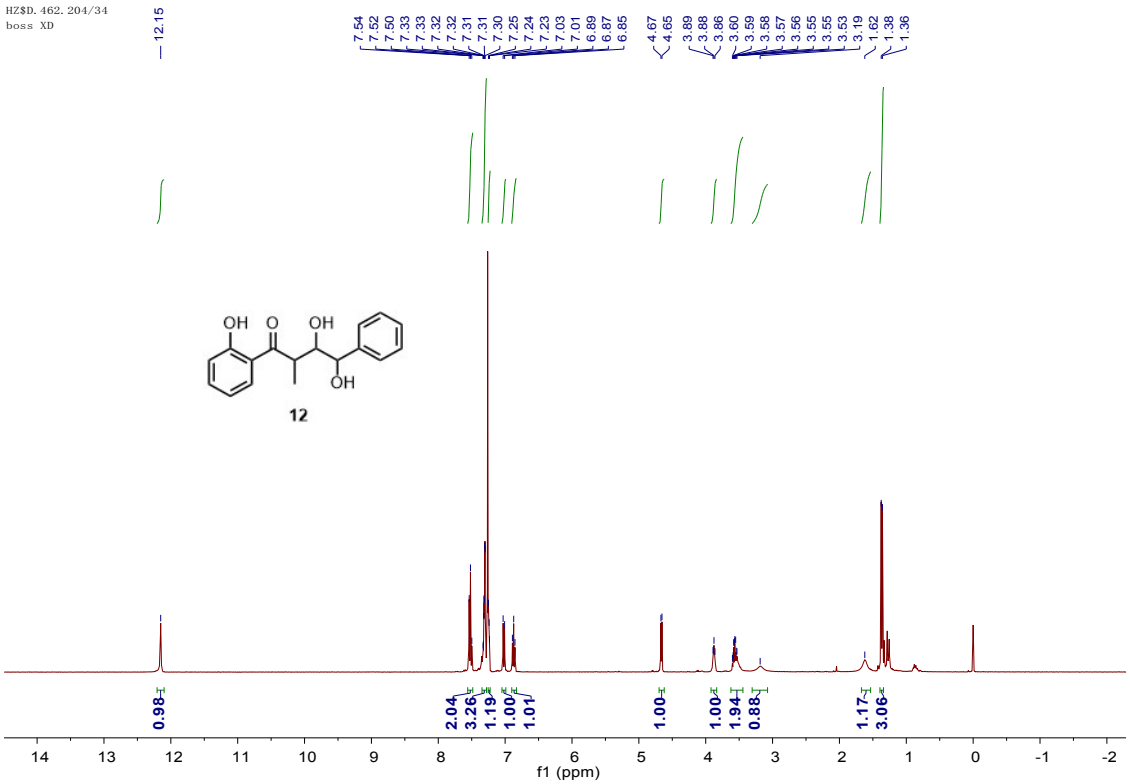


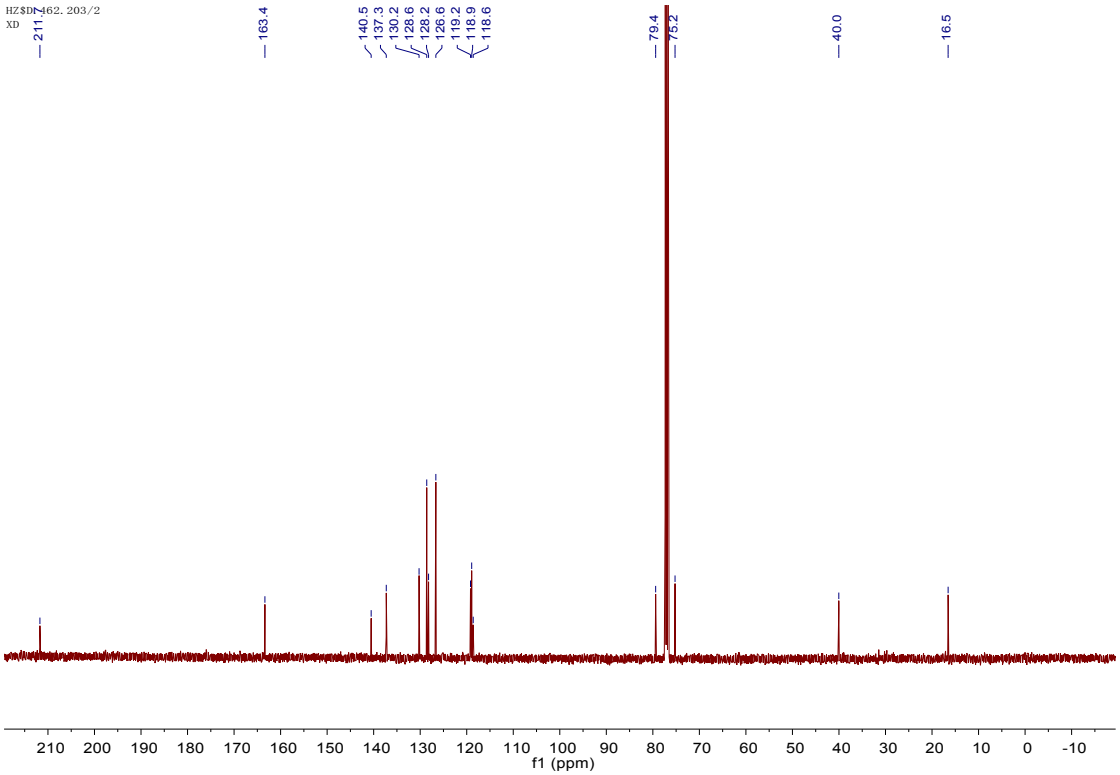
11

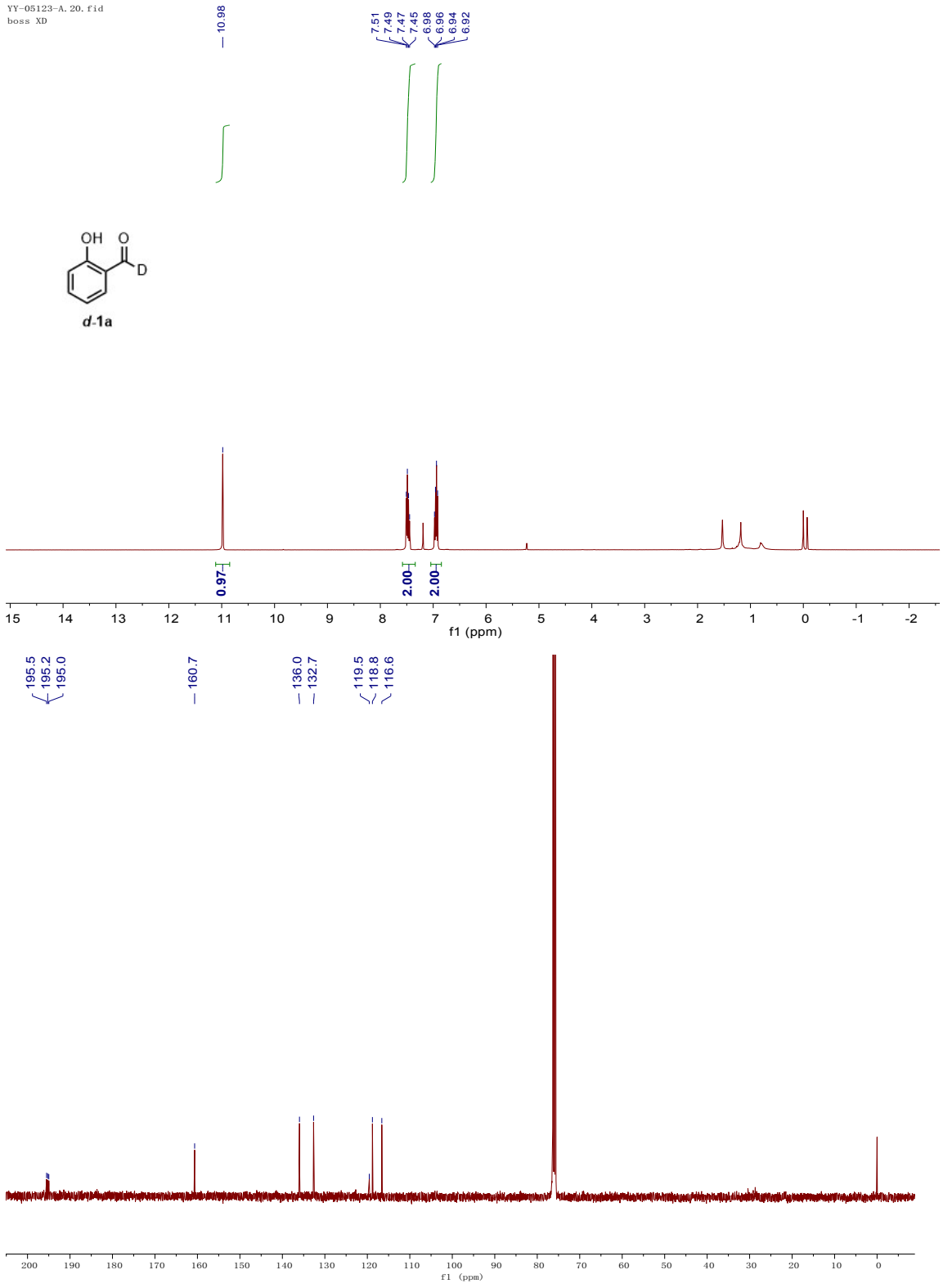
dr: 1.8:1






HZ$D. 462. 204/34
boss XD
12.15
7.54
7.52
7.50
7.33
7.33
7.32
7.32
7.31
7.31
7.30
7.25
7.24
7.23
7.03
7.01
6.89
6.87
6.85
4.67
4.65
3.89
3.88
3.86
3.60
3.59
3.58
3.57
3.56
3.55
3.55
3.53
3.19
1.62
1.38
1.36
OH
O
OH
OH
12
0.98
2.04
3.26
1.19
1.00
1.01
1.00
1.00
1.94
0.88
1.17
3.06
f1 (ppm)


HZ$D7. 462. 203/2
XD
211.7
163.4
140.5
137.3
130.2
128.6
128.2
126.6
119.2
118.9
118.6
79.4
75.2
40.0
16.5
f1 (ppm)


YY-05123-A.20.fid
boss XD
10.98
7.51
7.49
7.47
7.45
6.98
6.96
6.94
6.92
OH
O
D
d-1a
0.97
2.00
2.00
f1 (ppm)
195.5
195.2
195.0
160.7
136.0
132.7
119.5
118.8
116.6
f1 (ppm)