Electronic Supplementary Material (ESI) for Chemical Science.

Asymmetric Synthesis of Isochromanone Derivatives via Trapping Carboxylic Oxonium Ylides and Aldol Cascade

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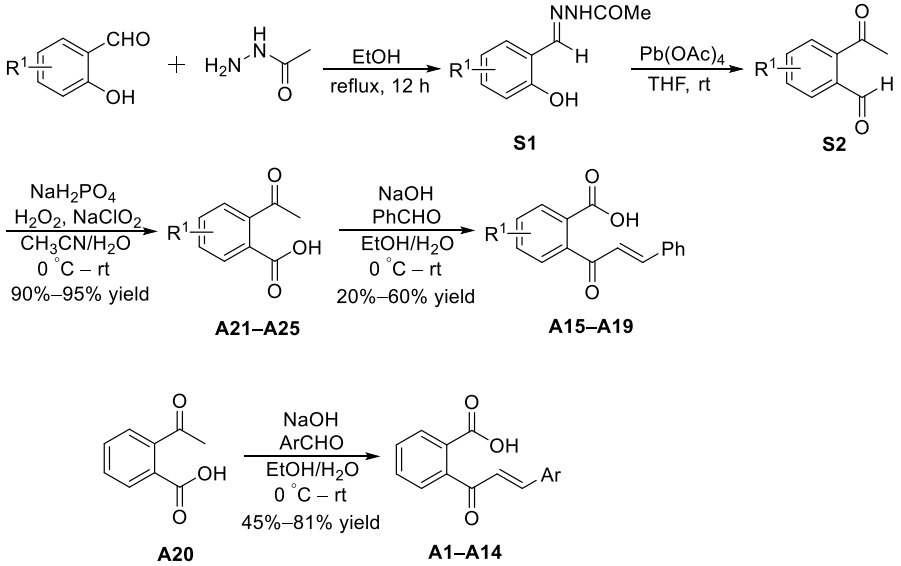
(A) General information

^{1}H NMR spectra were recorded on commercial instruments (400 MHz or 600 MHz). Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$, δ = 7.26, DMSO-d_6, δ = 2.50). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets), coupling constants (Hz), integration and assignment.
^{13}C{^{1}H} NMR spectra were collected on commercial instruments (101 MHz or 151MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$, δ = 77.0, DMSO-d_6, δ = 39.5).
^{19}F{^{1}H} NMR spectra were collected at 376 MHz with complete proton decoupling.
Enantiomeric excesses (ee) were determined by HPLC analysis using the corresponding commercial chiralpak column as stated in the experimental procedures at 25 °C.
Optical rotations were measured with a Perkin-Elmer model 241 polarimeter and reported as follows:
$[\alpha]_D^T$ (*c*: g/100 mL, in $CHCl_3$, unless otherwise noted, λ = 589 nm).
HRMS was recorded on a commercial apparatus (ESI Source).
IR spectra were recorded on SHIMADZU IR Tracer-100 FT-IR spectrophotometer.
Chromatography: Qingdao Haiyang silica gel, HG/T2354-92, HCP.
Solvents: Et_2O (diethyl ether), THF (tetrahydrofuran), and toluene were freshly distilled from sodium metal prior to use. EtOAc (ethyl acetate), $CHCl_3$, CH_2Cl_2, CH_2ClCH_2Cl, $CHCl_2CH_2Cl$ and $CHCl_2CHCl_2$ were freshly distilled from CaH_2 prior to use.
The preparation of acids[1], diazoketones[2] and ligands[3] followed the literature.

(B) Preparation of the substrates

General procedure for the synthesis of 2-acetylbenzoic acids A21-A25[1a] and 2-cinnamoylbenzoic acids A1-A19[1b].



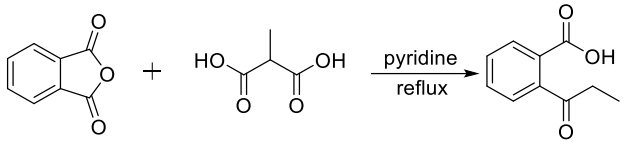
General procedure 1: To a stirred solution of salicylaldehyde derivative (30 mmol) in EtOH (150 mL) at room temperature was added acethydrazide (2.22 g, 30.0 mmol), and the reaction was heated to reflux for 12 h. Upon completion, the mixture was cooled to room temperature, concentrated and washed with PE. The crude **S1** was directly used in the next step without purification.

Product **S1** (30 mmol) was dissolved in 150 mL THF, then $Pb(OAc)_4$ (14.64 g, 33 mmol) was added slowly in portions while stirring. The reaction was stirred at room temperature for 2−3 h. After completion, the mixture was filtered and then the solvent was removed under reduced pressure. The residue was dissolved in 400 mL of EtOAc and washed with water (3x70 mL). The combined organic phases were successively washed with saturated aqueous $NaHCO_3$ and brine. The combined organic layers were dried over anhydrous Na_2SO_4, filtered and evaporated. The crude was purified by column chromatography on silica gel (PE/EtOAc = 15/1) to give product **S2**.

To a stirred solution of **S2** (18 mmol) in MeCN (180 mL) at 0 °C was successively added NaH_2PO_4 (0.96 g, 6.18 mmol), H_2O_2 (35% in H_2O, 5.2 mL) and $NaClO_2$ (6.48 g, 72 mmol). The reaction mixture was vigorously stirred at 0 °C for 1 h and at room temperature for 2 h. After completion, the reaction mixture were added EtOAc (120 mL) and water (75 mL). The organic layer was separated, and the aqueous layer was extracted by EtOAc (3×50 mL). The combined organic layer was washed with water and brine, dried over anhydrous Na_2SO_4, filtered and evaporated. The resulting acid substrates **A21-A25** are generally pure enough to use without further purification unless other notification.

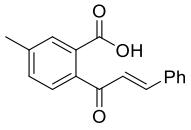
Substrates **A15-A19** were implemented following reported methods with modification[1b]. To to a solution of compounds **A21-A25** (15 mmol) and corresponding aldehyde (18 mmol) in ethanol (15 mL) was added an aqueous solution of NaOH (4.0 M, 2.0 equiv) at 0 °C. The ice-bath was removed, and the reaction stirred at room temperature until complete consumption of starting material. The reaction was then acidified with 6 M HCl aqueous solution at 0 °C. Then the EtOH was removed completely by rotoevaporation and the remaining aqueous layer was extracted with EtOAc, the combined organic layers were dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude solids were further purified by trituration using dichloromethane, ethanol and petroleum ether to provide pure ketoacids **A15-A19**.

Substrates **A1-A14** were synthesized as the same condition with those of **A15-A19**.



A26

General procedure 2[1c]: A mixture of phthalic anhydride (12 mmol), malonic acid (10 mmol) and pyridine (0.79 g, 10 mmol) was refluxed for 3 h to 5 h. Upon completion, the resulting mixture was cooled to room temperature, and then water (9 mL) was added and the mixture was stirred for 30 min. The solution was treated with concentrated HCl to pH 3-4, extracted with ethyl acetate (2×10 mL). The organic layers were combined, dried over Na_2SO_4 and evaporated under reduced pressure. The crude compound were further purified by trituration using dichloromethane, ethanol and petroleum ether to provide pure acid **A26**.



A15

2-cinnamoyl-5-methylbenzoic acid (A15)

Compound **A15** was obtained according to the general procedure 1 as a pale green solid (20% yield).

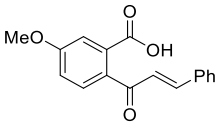
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.62 (s, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 7.49 – 7.47 (m, 2H), 7.37 – 7.32 (m, 4H), 7.18 – 7.13 (m, 2H), 7.00 (d, *J* = 16.4 Hz, 1H), 2.44 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 197.4, 171.0, 145.6, 144.3, 142.8, 134.3, 131.2, 130.5, 130.2, 128.8, 128.4, 128.1, 127.7, 124.9, 21.6 ppm;

m.p. 150.2 – 154.3 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{17}H_{14}O_3Na$: 289.0835, found: 289.0837;

IR (neat): 3028, 2921, 2653, 1689, 1648, 1600, 1495, 1447, 1408, 1287, 1193, 1126, 1059, 973, 866, 839, 787, 716, 604, 481, 456 cm^{-1}.



A16

2-cinnamoyl-5-methoxybenzoic acid (A16)

Compound **A16** was obtained according to the general procedure 1 as a pale yellow solid (36% yield).

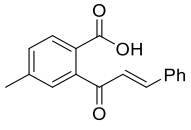
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.45 (s, 1H), 8.05 (d, *J* = 8.8 Hz, 1H), 7.49 – 7.46 (m, 2H), 7.37 – 7.33 (m, 3H), 7.14 (d, *J* = 16.4 Hz, 1H), 7.01 – 6.96 (m, 2H), 6.82 (d, *J* = 2.4 Hz, 1H), 3.88 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 196.9, 170.5, 163.4, 145.6, 145.2, 134.3, 133.4, 130.6, 128.8, 128.4, 127.6, 119.4, 114.6, 112.9, 55.7 ppm;

m.p. 155.1 – 158.7 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{17}H_{14}O_4Na$: 305.0784, found: 305.0784;

IR (neat): 3054, 2934, 2655, 2361, 1676, 1596, 1572, 1406, 1332, 1283, 1237, 1176, 1025, 841, 762, 690, 597, 556, 485 cm^{-1}.



A17

2-cinnamoyl-4-methylbenzoic acid (A17)

Compound **A17** was obtained according to the general procedure 1 as a white solid (34% yield).

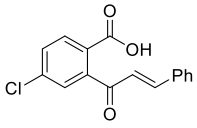
^{1}H NMR (600 MHz, DMSO-d_6) δ 13.12 (s, 1H), 7.70 – 7.68 (m, 3H), 7.49 (d, *J* = 7.8 Hz, 1H), 7.43 – 7.39 (m, 4H), 7.22 – 7.16 (m, 2H), 2.42 (s, 3H);

^{13}C{^{1}H} NMR (151 MHz, DMSO-d_6) δ 195.2, 167.9, 143.7, 140.0, 138.4, 134.3, 132.2, 130.9, 130.6, 130.0, 129.0, 128.5, 127.8, 127.3, 20.7 ppm;

m.p. 190.1 – 194.2 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{17}H_{14}O_3Na$: 289.0835, found: 289.0839;

IR (neat): 2986, 2816, 2661, 2577, 2361, 1681, 1651, 1496, 1448, 1306, 1280, 1209, 1056, 970, 832, 766, 584, 457 cm^{-1}.



A18

4-chloro-2-cinnamoylbenzoic acid (A18)

Compound **A18** was obtained according to the general procedure 1 as a white solid (32% yield).

^{1}H NMR (600 MHz, DMSO-d_6) δ 7.89 (d, *J* = 2.4 Hz, 1H), 7.76 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.71 – 7.70 (m, 2H), 7.55 (d, *J* = 8.4 Hz, 1H), 7.44 – 7.39 (m, 3H), 7.25 – 7.18 (m, 2H);

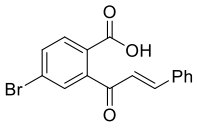
^{13}C{^{1}H} NMR (151 MHz, DMSO-d_6) δ 194.6, 166.4, 144.7, 139.9, 134.5, 134.2, 132.6, 131.8, 130.7, 129.6, 129.3, 129.0, 128.6, 127.0 ppm;

m.p. 185.9 – 190.7 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{16}H_{11}{}^{34.9689}ClO_3Na$: 309.0289, found:309.0290;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{16}H_{11}{}^{36.9659}ClO_3Na$: 311.0260, found: 311.0255;

IR (neat): 3008, 2633, 2361, 1703, 1645, 1623, 1593, 1449, 1277, 1261, 1211, 1104, 1029, 833, 797, 763, 688, 574 cm^{-1}.



A19

4-bromo-2-cinnamoylbenzoic acid (A19)

Compound **A19** was obtained according to the general procedure 1 as a white solid (20% yield).

^{1}H NMR (600 MHz, DMSO-d_6) δ 8.03 (d, J = 2.4 Hz, 1H), 7.90 (dd, J = 8.4, 2.4 Hz, 1H), 7.73 – 7.68 (m, 2H), 7.48 (d, J = 8.4 Hz, 1H), 7.44 – 7.39 (m, 3H), 7.25 – 7.17 (m, 2H);

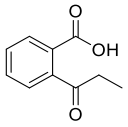
^{13}C{^{1}H} NMR (151 MHz, DMSO-d_6) δ 194.7, 166.2, 144.7, 140.2, 134.7, 134.2, 132.6, 132.1, 130.7, 129.8, 129.0, 128.7, 127.0, 123.0 ppm;

m.p. 209.8 – 212.1 °C;

HRMS (ESI) m/z [M+Na]$^+$ calcd for $C_{16}H_{11}{}^{78.9183}BrO_3Na$: 352.9784, found: 352.9787;

HRMS (ESI) m/z [M+Na]$^+$ calcd for $C_{16}H_{11}{}^{80.9163}BrO_3Na$: 354.9763, found: 354.9765;

IR (neat): 3307, 2988, 2937, 2361, 2338, 1719, 1611, 1424, 1372, 1334, 1276, 1198, 1142, 1035, 964, 883, 751, 695, 570, 471 cm^{-1}.



A26

2-propionylbenzoic acid (A26)

Compound **A26** was obtained according to the general procedure 2 as a white solid (78% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, J = 7.6 Hz, 1H), 7.67 (td, J = 7.6, 1.2 Hz, 1H), 7.55 – 7.49 (m, 2H), 4.89 (s, 1H), 2.21 (s, 2H), 0.88 (t, J = 7.2 Hz, 3H);

^{13}C{^{1}H} NMR (151 MHz, DMSO-d_6) δ 168.1, 149.2, 134.7, 130.3, 126.7, 124.5, 122.8, 108.7, 31.5, 7.8 ppm;

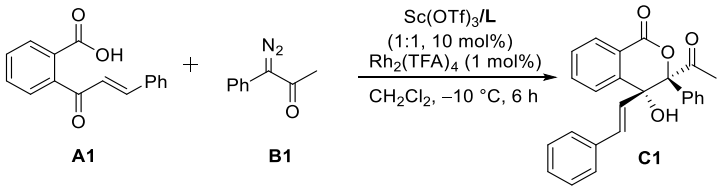
m.p. 93.5 – 98.0 °C;

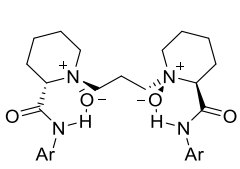
HRMS (ESI) m/z [M]$^-$ calcd for $C_{10}H_9O_3$: 177.0546, found: 177.0549;

IR (neat): 3361, 2980, 2941, 2361, 2337, 1740, 1461, 1416, 1380, 1344, 1276, 1262, 1135, 1061, 897, 761, 697 cm^{-1}.

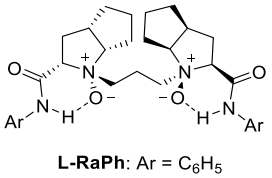
(C) Optimization of reaction conditions

Table S1. The screening of ligands.

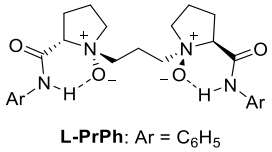




L-PiPh: Ar = C_6H_5
L-PiMe$_2$: Ar = 2,6-$Me_2C_6H_3$
L-PiEt$_2$: Ar = 2,6-$Et_2C_6H_3$
L-PiPr$_2$: Ar = 2,6-*i*$Pr_2C_6H_3$
L-PiBn: Ar = $CH_2C_6H_5$
L-PiC$_2$H$_4$Ph: Ar = $C_2H_4C_6H_5$



L-RaPh: Ar = C_6H_5



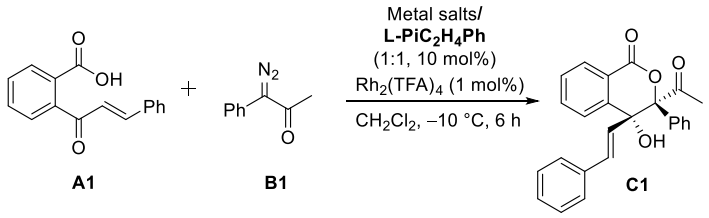
L-PrPh: Ar = C_6H_5

entry[a]	**ligand**	yield (%)[b]	ee (%)[c]
1	**L-PiPh**	40	66
2[d]	**L-RaPh**	20	2
3	**L-PrPh**	34	6
4	**L-PiPr$_2$**	26	0
5	**L-PiEt$_2$**	24	0
6	**L-PiMe$_2$**	25	8
7	**L-PiBn**	66	94

8	**L-PiC$_2$H$_4$Ph**	75	97

[a] Unless otherwise noted, the reactions were performed with **A1** (0.10 mmol), **B1** (2.0 equiv), $Sc(OTf)_3$ (10 mol%), **Ligand** (10 mol%), $Rh_2(TFA)_4$ (1 mol%), in CH_2Cl_2 (2.0 mL) at −10 °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC.

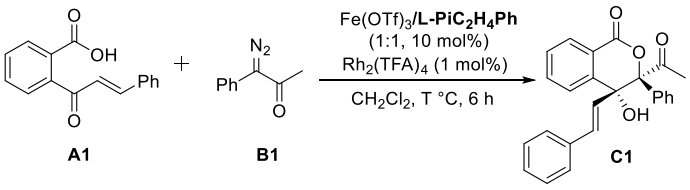
Table S2. The screening of metal salts.



entry[a]	metal salts	yield (%)[b]	ee (%)[c]
1	$Sc(OTf)_3$	75	97
2	$Ni(OTf)_2$	29	60
3	$Mg(OTf)_2$	22	49
4	$Cu(OTf)_2$	26	6
5	$Zn(OTf)_2$	37	20
6	$Fe(OTf)_3$	84	97
7	$Co(OTf)_2$	57	80
8	$Fe(OTf)_2$	67	0

[a] Unless otherwise noted, the reactions were performed with **A1** (0.10 mmol), **B1** (2.0 equiv), metal salts (10 mol%), **L-PiC$_2$H$_4$Ph** (10 mol%), $Rh_2(TFA)_4$ (1 mol%), in CH_2Cl_2 (2.0 mL) at −10 °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC.

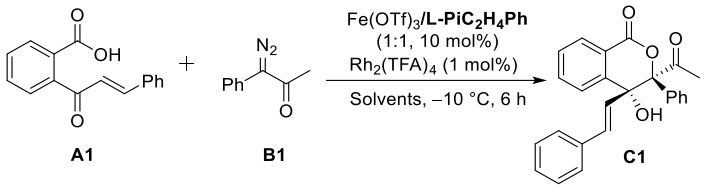
Table S3. The screening of temperature.



entry[a]	T (°C)	yield (%)[b]	ee (%)[c]
1	10	88	92
2	0	83	97
3	−10	84	97
4	−20	84	97

[a] Unless otherwise noted, the reactions were performed with **A1** (0.10 mmol), **B1** (2.0 equiv), $Fe(OTf)_3$ (10 mol%), **L-PiC$_2$H$_4$Ph** (10 mol%), $Rh_2(TFA)_4$ (1 mol%), in CH_2Cl_2 (2.0 mL) at T °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC.

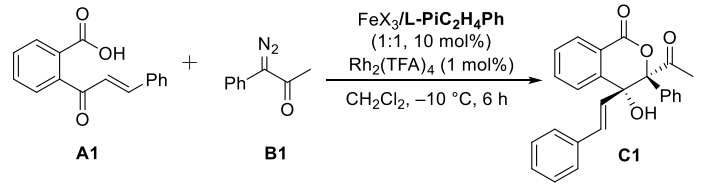
Table S4. The screening of solvents.



entry[a]	solvents	yield (%)[b]	ee (%)[c]
1	THF	10	4
2	EtOAc	32	0
3	Toluene	11	6
4	Et_2O	54	95
5	CH_2Cl_2	84	97
6	$CHCl_3$	80	91
7	CH_2ClCH_2Cl	78	97
8	$CHCl_2CH_2Cl$	31	5
9	$CHCl_2CHCl_2$	82	96

[a] Unless otherwise noted, the reactions were performed with **A1** (0.10 mmol), **B1** (2.0 equiv), $Fe(OTf)_3$ (10 mol%), **L-PiC$_2$H$_4$Ph** (10 mol%), $Rh_2(TFA)_4$ (1 mol%), in Solvents (2.0 mL) at −10 °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC.

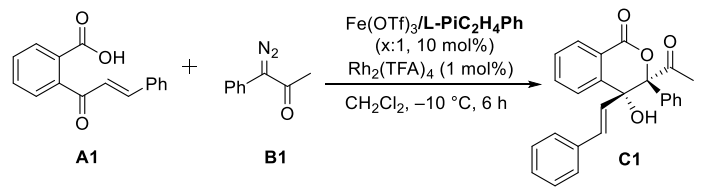
Table S5. The screening of counter ion of Fe(III) salt.



entry[a]	FeX_3	yield (%)[b]	ee (%)[c]
1	$FeCl_3$	85	–8
2	$Fe(NTf)_3$	78	88
3	$Fe(OTf)_3$	84	97

[a] Unless otherwise noted, the reactions were performed with **A1** (0.10 mmol), **B1** (2.0 equiv), FeX_3 (10 mol%), **L-PiC$_2$H$_4$Ph** (10 mol%), $Rh_2(TFA)_4$ (1 mol%), in CH_2Cl_2 (2.0 mL) at –10 °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC.

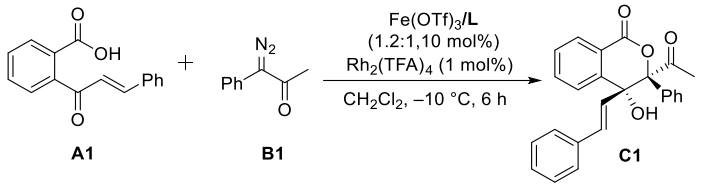
Table S6. The screening of ratio between $Fe(OTf)_3$ and **L-PiC$_2$H$_4$Ph**.



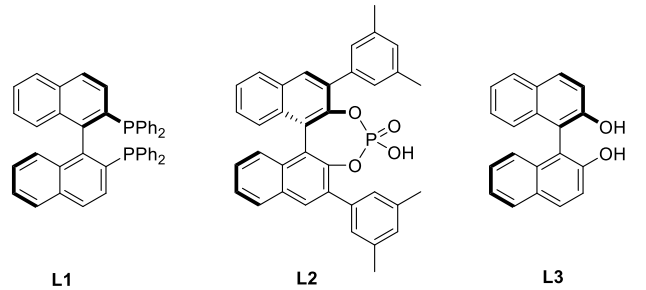
entry[a]	x:1	yield (%)[b]	ee (%)[c]
1	0.8:1	82	96
2	1:1	84	97
3	1.2:1	91	97
4	1.5:1	85	95

[a] Unless otherwise noted, the reactions were performed with **A1** (0.10 mmol), **B1** (2.0 equiv), $Fe(OTf)_3$ (10x mol%), **L-PiC$_2$H$_4$Ph** (10 mol%), $Rh_2(TFA)_4$ (1 mol%), in CH_2Cl_2 (2.0 mL) at –10 °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC.

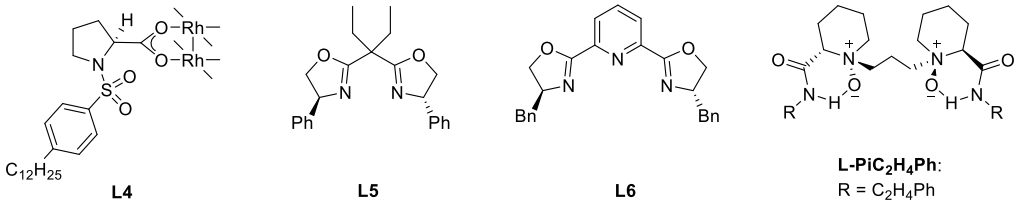
Table S8. The screening of other ligands



A1 **B1** **C1**



L1 **L2** **L3**

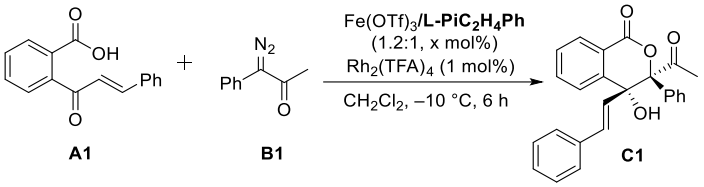


L4 **L5** **L6** **L-PiC$_2$H$_4$Ph**: R = C_2H_4Ph

entry[a]	**L**	yield (%)[b]	ee (%)[c]
1	**L1**	20	5
2	**L2**	Trace	–
3	**L3**	26	0
4[d]	**L4**	23	0
5	**L5**	24	0
6	**L6**	21	0
7	**L-PiC$_2$H$_4$Ph**	91	97

[a] Unless otherwise noted, the reactions were performed with **A1** (0.10 mmol), **B1** (2.0 equiv), $Fe(OTf)_3$ (12 mol%), **L** (10 mol%), $Rh_2(TFA)_4$ (1 mol%), in CH_2Cl_2 (2.0 mL) at –10 °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC. [d] Without using of $Fe(OTf)_3$ and $Rh_2(TFA)_4$.

Table S7. The screening of amount of $Fe(OTf)_3$ and **L-PiC$_2$H$_4$Ph**.



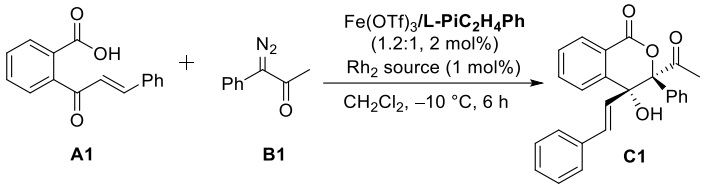
A1 **B1** **C1**

entry[a]	x	yield (%)[b]	ee (%)[c]
1	10	91	97
2	5	91	97
3	2	91	97

4	1	88	96

[a] Unless otherwise noted, the reactions were performed with **A1** (0.10 mmol), **B1** (2.0 equiv), $Fe(OTf)_3$ (1.2x mol%), **L-PiC$_2$H$_4$Ph** (x mol%), $Rh_2(TFA)_4$ (1 mol%), in CH_2Cl_2 (2.0 mL) at –10 °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC.

Table S9. The screening of rhodium source.

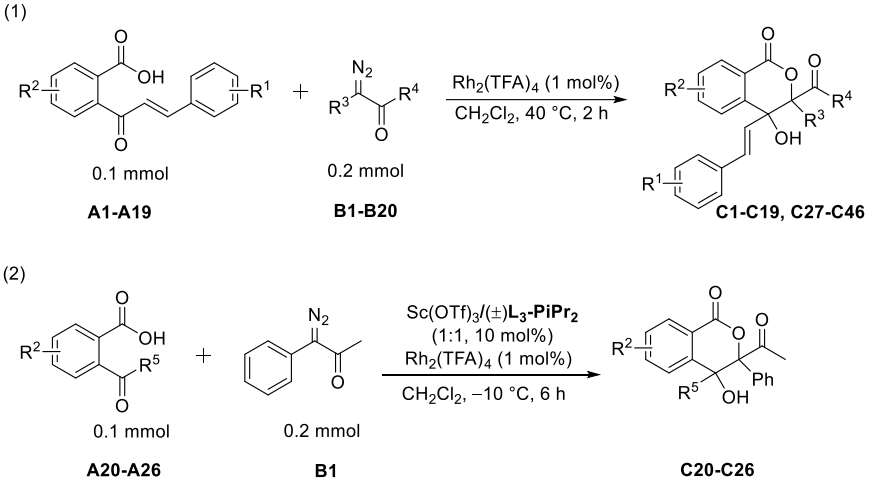


entry[a]	Rh_2 source	yield (%)[b]	ee (%)[c]
1	$Rh_2(TFA)_4$	91	97
2	$Rh_2(Oct)_4$	59	93
3	$Rh_2(OAc)_4$	76	94
4	$Rh_2(esp)_4$	64	93
5	$Rh_2(piv)_4$	68	94

[a] Unless otherwise noted, the reactions were performed with **A1** (0.10 mmol), **B1** (2.0 equiv), $Fe(OTf)_3$ (2.4 mol%), **L-PiC$_2$H$_4$Ph** (2 mol%), Rh_2 source (1 mol%), in CH_2Cl_2 (2.0 mL) at –10 °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC.

(D) Experimental procedures

General procedure for the preparation of the racemic products.

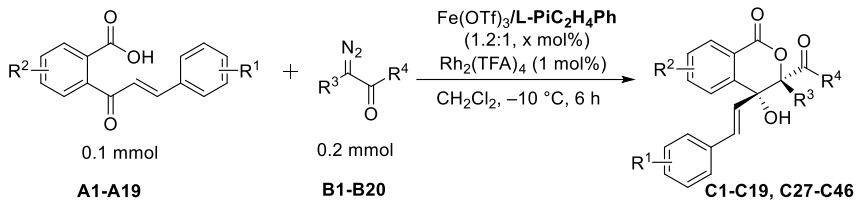


General produce for (1): To a test tube, $Rh_2(TFA)_4$ (1 mol%) and the substrates **A1-A19** (0.10 mmol) were added under an N_2 atmosphere. Then CH_2Cl_2 was added and the mixture was stirred at 40 °C, and a solution of the corresponding α-diazoketones **B1-B20** (0.20 mmol in 1.0 mL CH_2Cl_2) was added. After the addition was completed, the reaction was stirred at 40 °C for 2 h, The crude reaction mixture was purified using column chromatography on silica gel (PE/CH_2Cl_2 = 1:1) to afford racemic products.

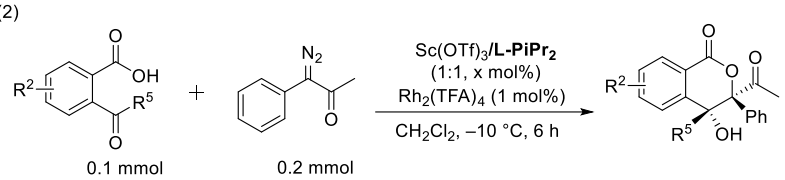
General produce for (2): To an oven-dried reaction tube under nitrogen atmosphere were added the $Sc(OTf)_3$ (4.9 mg, 10 mol %), (±)**L$_3$-PiPr$_2$** (6.5 mg, 10 mol %), and CH_2Cl_2 (1.0 mL). The mixture was stirred at 35 °C for 30 min. Then the solution was evaporated in vacuo and nitrogen was introduced. After that, CH_2Cl_2 (1.0 mL), $Rh_2(TFA)_4$ (0.7 mg, 1 mol %), acids **A20-A26** (0.10 mmol) were added subsequently. The reaction mixture was stirred at 35 °C for 20 min. After the solution was cooled to –10 °C, α-diazoketone **B1** (0.20 mmol in 1.0 mL CH_2Cl_2) was added, and the reaction mixture was stirred at –10 °C for 6 h. The reaction system was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1:1.5) to afford the desired products.

Representative experimental procedure for the catalytic asymmetric reactions

(1)



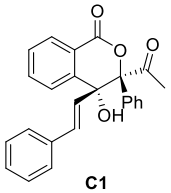
(2)



General produce for (1): In a glovebox, ketoacids **A1-A19** (0.1 mmol), $Rh_2(TFA)_4$ (0.70 mg, 0.001 mmol, 1 mol%) and $Fe(OTf)_3$/**L-PiC$_2$H$_4$Ph** (1.2:1, x mol%) were weighted into a dried test tube. Anhydrous CH_2Cl_2 (1.0 mL) was added and the solution was stirred at 35 °C for 0.5 h. After the solution was cooled to –10 °C, α-diazoketones **B1-B20** (0.20 mmol in 1.0 mL CH_2Cl_2) was added, the reaction mixture was stirred at –10 °C for 6 h. The reaction system was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1:1) to afford the desired product .

General produce for (2): The corresponding products **C20-C26** were obtained by using $Sc(OTf)_3$/**L-PiPr$_2$** (1:1, x mol%) as the catalyst in the same reaction conditions.

(E) The analytical and spectral characterization data of the products



C1

(3*S*,4*R*)-3-acetyl-4-hydroxy-3-phenyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C1**; 0.1 mmol scale reaction; 34.9 mg, white solid, 91% yield, 97% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 7.08 min, t_2 = 8.72 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.12 (m, 1H), 7.82 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.75 – 7.67 (m, 3H), 7.49 (td, *J* = 7.6, 1.2 Hz, 1H), 7.46 – 7.40 (m, 3H), 7.22 – 7.14 (m, 3H), 7.10 – 7.07 (m, 2H), 6.11 (d, *J* = 15.6 Hz, 1H), 5.99 (d, *J* = 15.6 Hz, 1H), 5.26 (s, 1H), 2.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.1, 163.0, 146.2, 135.9, 135.7, 132.1, 131.3, 130.2, 129.2, 128.6, 128.4, 128.3, 127.9, 127.7, 126.6, 126.5, 125.1, 122.0, 90.6, 76.8, 27.4 ppm;

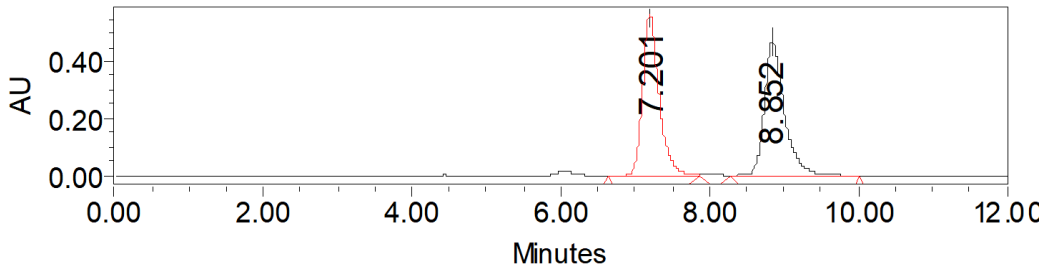
m.p. 136.6 – 139.0 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{20}O_4Na$: 407.1254, found: 407.1255;

[α]$_D^{21}$ = +480.3 (*c* = 0.38 in $CHCl_3$);

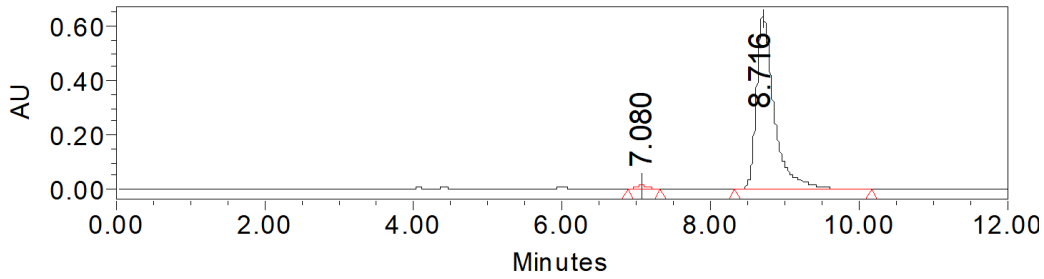
IR (neat): 3465, 3062, 3027, 2924, 1740, 1706, 1602, 1495, 1451, 1358, 1287, 1248, 1081, 969, 770, 751, 717, 694 cm^{-1}.

HPLC spectrum of **C1**

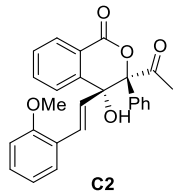


	Retention Time	Area	% Area
1	7.201	8657338	49.78

2	8.852	8732431	50.22



	Retention Time	Area	% Area
1	7.080	173259	1.58
2	8.716	10823763	98.42



C2

(3*S*,4*R*)-3-acetyl-4-hydroxy-4-((*E*)-2-methoxystyryl)-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/EtOAc = 10/1) to afford the desired product **C2**; 0.1 mmol scale reaction; 35.6 mg, white solid, 86% yield, 95% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 7.59 min, t_2 = 9.92 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 – 8.10 (m, 1H), 7.83 (d, *J* = 7.6 Hz, 1H), 7.74 – 7.69 (m, 3H), 7.50 – 7.37 (m, 4H), 7.15 – 7.11 (m, 1H), 7.09 – 7.07 (m, 1H), 6.79 (t, *J* = 7.6 Hz, 1H), 6.73 (d, *J* = 8.4 Hz, 1H), 6.40 (d, *J* = 16.4 Hz, 1H), 6.04 (d, *J* = 16.0 Hz, 1H), 5.22 (s, 1H), 3.65 (s, 3H), 2.13 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.2, 163.2, 156.9, 146.5, 135.7, 131.5, 130.1, 129.1, 129.0, 128.4, 128.2, 127.4, 127.2, 126.6, 125.2, 125.1, 122.0, 120.4, 110.8, 90.7, 55.3, 27.5 ppm;

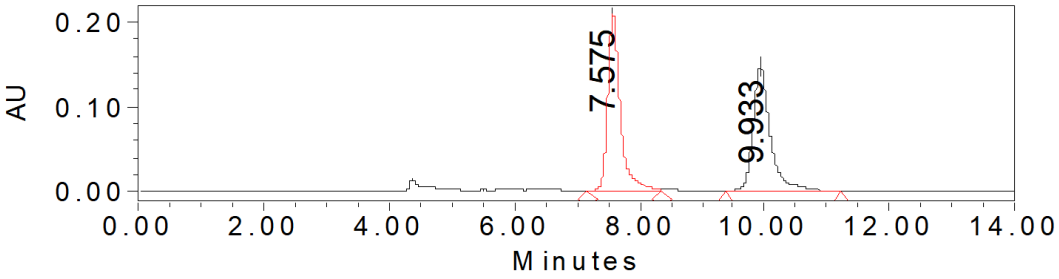
m.p. 84.0 – 87.0 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{26}H_{22}O_5Na$: 437.1359, found: 437.1355;

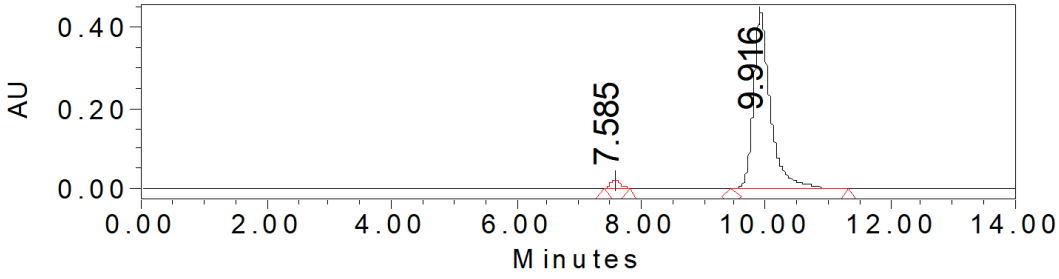
$[\alpha]_D^{21}$ = +508.3 (*c* = 0.40 in $CHCl_3$);

IR (neat): 3465, 3068, 3014, 2938, 2837, 1738, 1706, 1600, 1490, 1459, 1357, 1288, 1247, 1084, 1031, 753, 712 cm^{-1}.

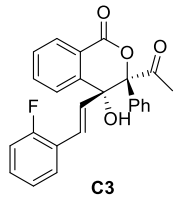
HPLC spectrum of **C2**



	Retention Time	Area	% Area
1	7.575	2912842	50.44
2	9.933	2861923	49.56



	Retention Time	Area	% Area
1	7.585	221101	2.55
2	9.916	8464312	97.45



C3

(3*S*,4*R*)-3-acetyl-4-((*E*)-2-fluorostyryl)-4-hydroxy-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C3**; 0.1 mmol scale reaction; 32.1 mg, white solid, 80% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 6.84 min, t_2 = 8.12 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.83 – 7.81 (m, 1H), 7.75 – 7.68 (m, 3H), 7.49 (td, *J* = 7.6, 1.2 Hz, 1H), 7.45 – 7.40 (m, 3H), 7.15 – 7.10 (m, 2H), 6.98 – 6.94 (m, 1H), 6.92 – 6.87 (m, 1H), 6.32 (d, *J* = 16.0 Hz, 1H), 6.12 (dd, *J* = 16.0, 1.6 Hz, 1H), 5.26 (d, *J* = 1.6 Hz, 1H), 2.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.0, 163.0, 161.5 (d, J_{C-F} = 251.5 Hz), 146.2, 135.8, 131.3, 130.3, 130.1 (d, J_{C-F} = 5.1 Hz), 129.3, 129.2, 128.6, 128.3, 127.6 (d, J_{C-F} = 4.0 Hz), 126.5, 125.1, 124.6 (d, J_{C-F} = 3.0 Hz), 123.9, 123.9 (d, J_{C-F} = 11.1 Hz), 121.9, 115.6 (d, J_{C-F} = 21.2 Hz), 90.5, 76.8, 27.4 ppm;

^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -117.17 ppm;

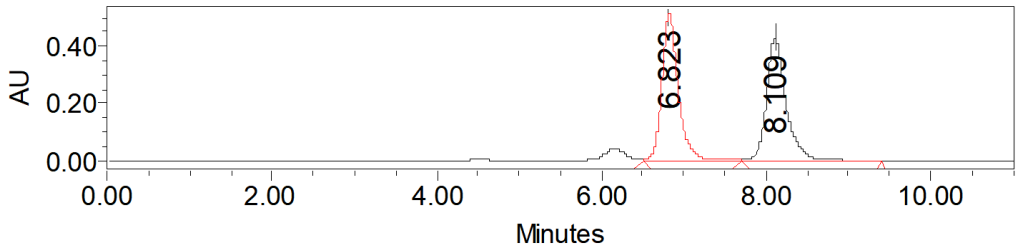
m.p. 145.2 – 147.1 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}FO_4Na$: 425.1159, found: 425.1158;

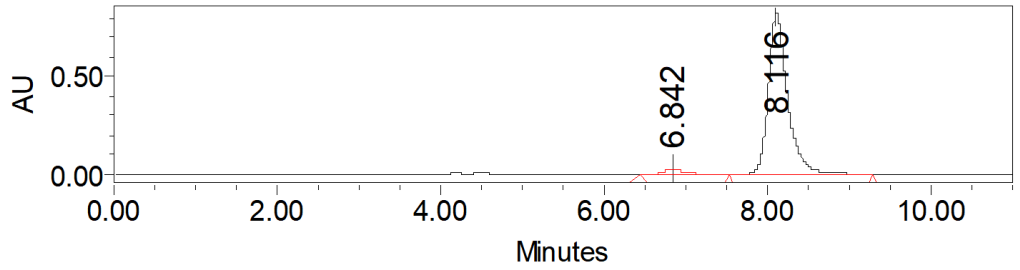
$[\alpha]_D^{21}$ = +522.9 (*c* = 0.31 in $CHCl_3$);

IR (neat): 3465, 3067, 2924, 1740, 1708, 1603, 1489, 1453, 1358, 1287, 1248, 1083, 755, 706, 573 cm^{-1}.

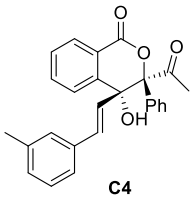
HPLC spectrum of **C3**



	Retention Time	Area	% Area
1	6.823	7205129	50.16
2	8.109	7159026	49.84



	Retention Time	Area	% Area
1	6.842	387357	2.81
2	8.116	13407445	97.19



C4

(3*S*,4*R*)-3-acetyl-4-hydroxy-4-((*E*)-3-methylstyryl)-3-phenylisochroman-1-one

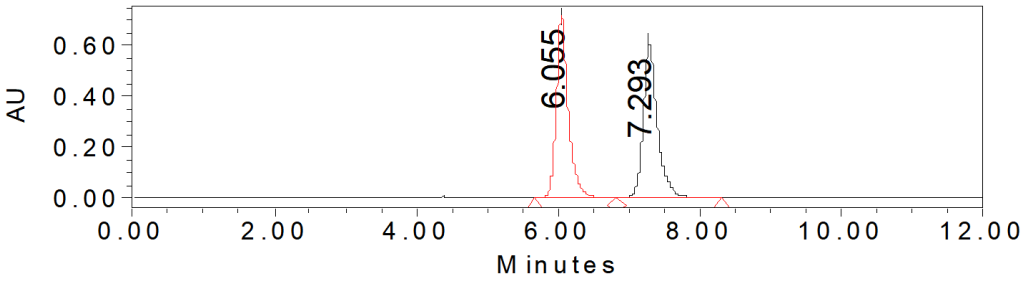
The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C4**; 0.1 mmol scale reaction; 36.2 mg, white solid, 91% yield, 92% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 6.07 min, t_2 = 7.31 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 7.6 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.75 – 7.68 (m, 3H), 7.51 – 7.39 (m, 4H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.98 (d, *J* = 7.6 Hz, 1H), 6.91 – 6.89 (m, 2H), 6.08 (d, *J* = 16.0 Hz, 1H), 5.97 (d, *J* = 16.0 Hz, 1H), 5.25 (s, 1H), 2.24 (s, 3H), 2.14 (s, 3H);

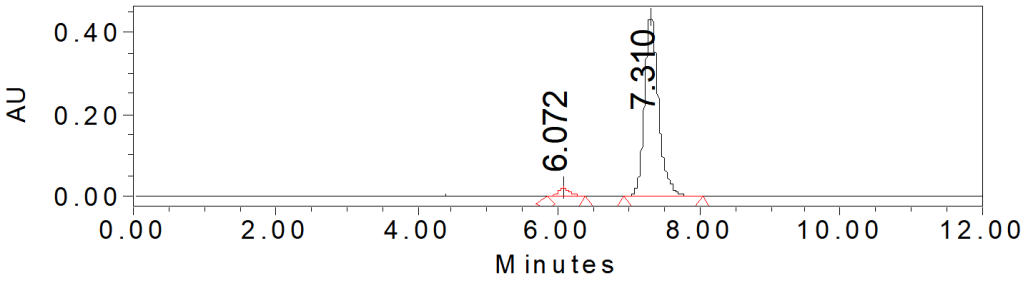
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.2, 163.1, 146.3, 137.9, 135.9, 135.7, 132.2, 131.3, 130.2, 129.2, 128.7, 128.5, 128.3, 128.3, 127.4, 127.4, 126.6, 125.1, 123.7, 122.0, 90.6, 76.8, 27.5, 21.2 ppm;

m.p. 71.0 – 73.3 °C;

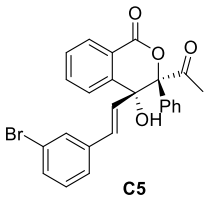
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{26}H_{22}O_4Na$: 421.1410, found: 421.1409;
$[\alpha]_D^{20}$ = +491.7 (*c* = 1.07 in $CHCl_3$);
IR (neat): 3463, 3019, 2855, 1737, 1705, 1602, 1492, 1451, 1357, 1286, 1247, 1215, 1080, 1037, 969, 751, 712, 693, 564 cm^{-1}.
HPLC spectrum of **C4**



	Retention Time	Area	% Area
1	6.055	8467393	49.78
2	7.293	8542637	50.22

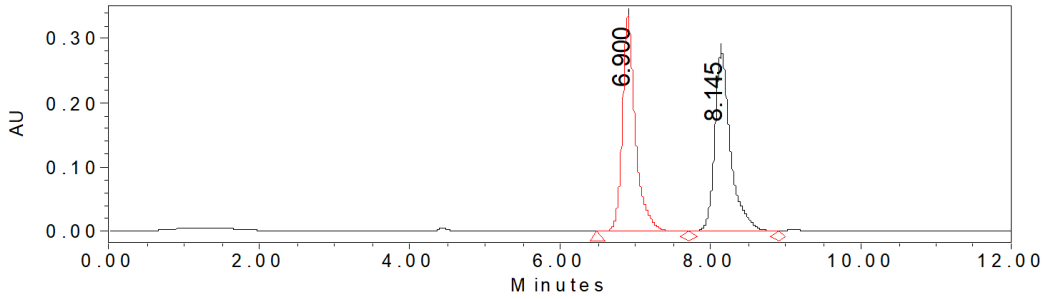


	Retention Time	Area	% Area
1	6.072	249309	3.95
2	7.310	6059396	96.05

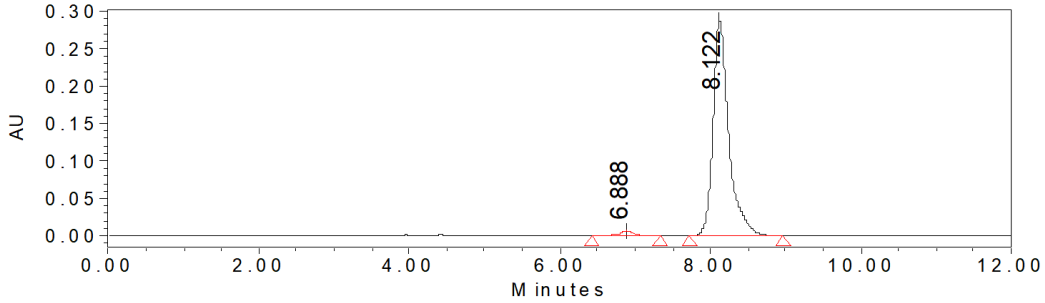


C5

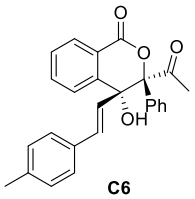
(3*S*,4*R*)-3-acetyl-4-((*E*)-3-bromostyryl)-4-hydroxy-3-phenylisochroman-1-one
The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C5**; 0.1 mmol scale reaction; 23.2 mg, white solid, 50% yield, 95% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 6.89 min, t_2 = 8.12 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 7.6 Hz, 1H), 7.80 – 7.78 (m, 1H), 7.73 (t, *J* = 7.6 Hz, 1H), 7.67 – 7.65 (m, 2H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.45 – 7.42 (m, 3H), 7.29 – 7.26 (m, 1H), 7.21 (s, 1H), 7.07 – 7.03 (m, 1H), 7.00 – 6.98 (m, 1H), 6.07 (d, *J* = 16.0 Hz, 1H), 5.99 (d, *J* = 16.0 Hz, 1H), 5.26 (s, 1H), 2.13 (s, 3H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.0, 162.9, 146.0, 138.1, 135.8, 131.2, 130.8, 130.5, 130.4, 129.9, 129.5 129.3, 129.1, 128.7, 128.3, 126.5, 125.2, 125.1, 122.5, 121.9, 90.5, 76.6, 27.4 ppm;
m.p. 69.8 – 74.2 °C;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{78.9183}BrO_4Na$: 485.0359, found: 485.0362;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{80.9163}BrO_4Na$: 487.0338, found: 487.0341;
$[\alpha]_D^{20}$ = +426.1 (*c* = 0.64 in $CHCl_3$);
IR (neat): 3464, 3021, 2925, 1738, 1706, 1600, 1563, 1452, 1357, 1287, 1248, 1210, 1080, 969, 757, 690, 560 cm^{-1}.
HPLC spectrum of **C5**



	Retention Time	Area	% Area
1	6.900	4093288	49.84
2	8.145	4119141	50.16



	Retention Time	Area	% Area
1	6.888	104683	2.45
2	8.122	4172597	97.55



C6

(3*S*,4*R*)-3-acetyl-4-hydroxy-4-((*E*)-4-methylstyryl)-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C6**; 0.1 mmol scale reaction; 32.3 mg, white solid, 81% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 9.52 min, t_2 = 11.00 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.82 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.75 – 7.68 (m, 3H), 7.49 (td, *J* = 7.6, 1.6 Hz, 1H), 7.46 – 7.40 (m, 3H), 7.02 – 6.97 (m, 4H), 6.06 (d, *J* = 16.0 Hz, 1H), 5.94 (dd, *J* = 16.0, 1.2 Hz, 1H), 5.25 (d, *J* = 1.2 Hz, 1H), 2.26 (s, 3H), 2.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.1, 163.0, 146.3, 137.8, 135.7, 133.1, 132.1, 131.3, 130.1, 129.1, 129.0, 128.5, 128.2, 126.7, 126.6, 126.5, 125.1, 122.0, 90.6, 76.8, 27.4, 21.1 ppm;

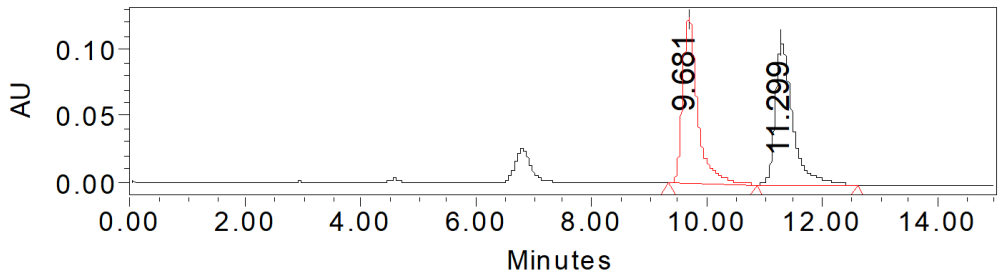
m.p. 72.8 – 76.3 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{26}H_{22}O_4Na$: 421.1410, found: 421.1397;

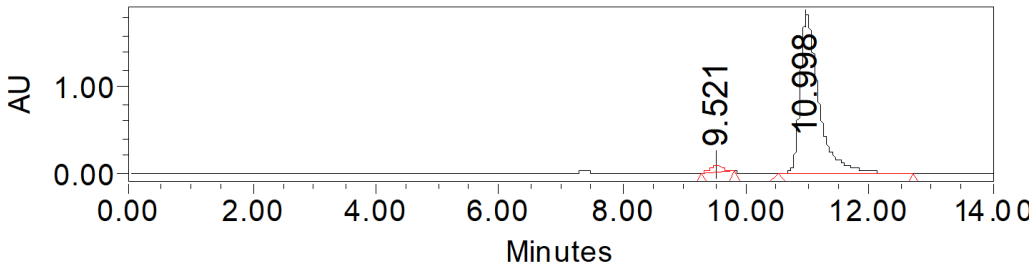
$[\alpha]_D^{20}$ = +494.1 (*c* = 1.32 in $CHCl_3$);

IR (neat): 3463, 3023, 2921, 1737, 1705, 1603, 1451, 1357, 1286, 1247, 1211, 1081, 1040, 971, 754, 712, 583 cm^{-1}.

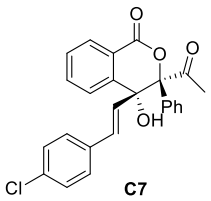
HPLC spectrum of **C6**



	Retention Time	Area	% Area
1	9.681	2295327	50.50
2	11.299	2249855	49.50



	Retention Time	Area	% Area
1	9.521	1206459	3.01
2	10.998	38874867	96.99



C7

(3*S*,4*R*)-3-acetyl-4-((*E*)-4-chlorostyryl)-4-hydroxy-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C7**; 0.1 mmol scale reaction; 38.5 mg, white solid, 92% yield, 95% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 10.51 min, t_2 = 11.57 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 – 8.12 (m, 1H), 7.81 – 7.79 (m, 1H), 7.75 – 7.71 (m, 1H), 7.67 – 7.64 (m, 2H), 7.50 (td, *J* = 7.6, 1.2 Hz, 1H), 7.45 – 7.40 (m, 3H), 7.17 – 7.14 (m, 2H), 7.02 – 6.98 (m, 2H), 6.06 (d, *J* = 16.0 Hz, 1H), 5.95 (dd, *J* = 16.0, 1.2 Hz, 1H), 5.25 (d, *J* = 1.2 Hz, 1H), 2.13 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.1, 163.0, 146.1, 135.8, 134.4, 133.6, 131.3, 130.9, 130.3, 129.3, 128.7, 128.6, 128.3, 128.3, 127.8, 126.5, 125.1, 122.0, 90.6, 76.7, 27.4 ppm;

m.p. 120.6 – 123.0 °C;

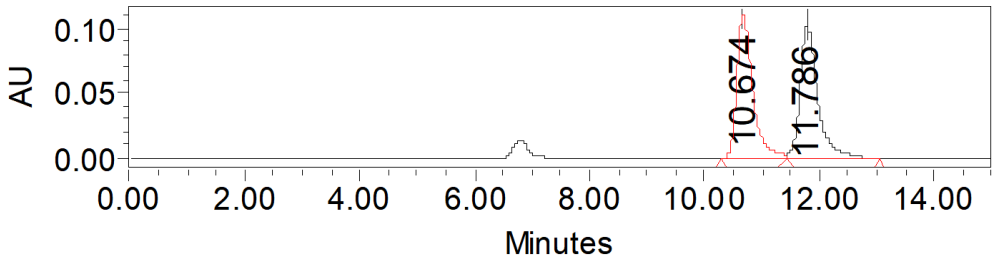
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}^{34.9689}ClO_4Na$: 441.0864, found: 441.0866;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}^{36.9659}ClO_4Na$: 443.0835, found: 443.0831;

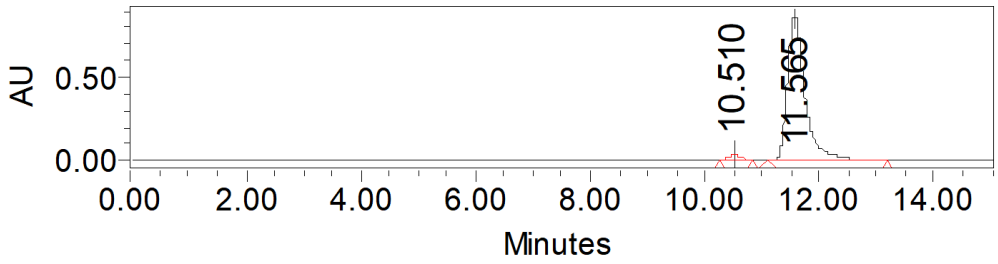
$[\alpha]_D^{20}$ = +388.0 (*c* = 0.61 in $CHCl_3$);

IR (neat): 3464, 3026, 1738, 1706, 1492, 1452, 1357, 1288, 1248, 1211, 1085, 1042, 970, 755, 727, 579 cm^{-1}.

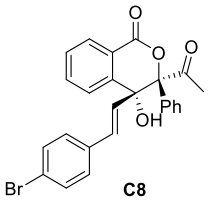
HPLC spectrum of **C7**



	Retention Time	Area	% Area
1	10.674	2150903	49.06
2	11.786	2233024	50.94



	Retention Time	Area	% Area
1	10.510	495033	2.56
2	11.565	18846445	97.44



C8

(3*S*,4*R*)-3-acetyl-4-((*E*)-4-bromostyryl)-4-hydroxy-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C8**; 0.1 mmol scale reaction; 30.1 mg, white solid, 65% yield, 98% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 8.54 min, t_2 = 9.79 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.80 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.73 (td, *J* = 7.2, 1.2 Hz, 1H), 7.67 – 7.65 (m, 2H), 7.50 (td, *J* = 7.6, 1.2 Hz, 1H), 7.45 – 7.40 (m, 3H), 7.32 – 7.29 (m, 2H), 6.95 – 6.92 (m, 2H), 6.05 (d, *J* = 16.0 Hz, 1H), 5.97 (d, *J* = 16.0 Hz, 1H), 5.26 (s, 1H), 2.13 (s, 3H).;

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.0, 163.0, 146.0, 135.8, 134.9, 131.5, 131.2, 130.9, 130.3, 129.3, 128.7, 128.4, 128.3, 128.1, 126.5, 125.1, 121.9, 121.8, 90.5, 76.7, 27.4 ppm;

m.p. 110.3 – 112.5 °C;

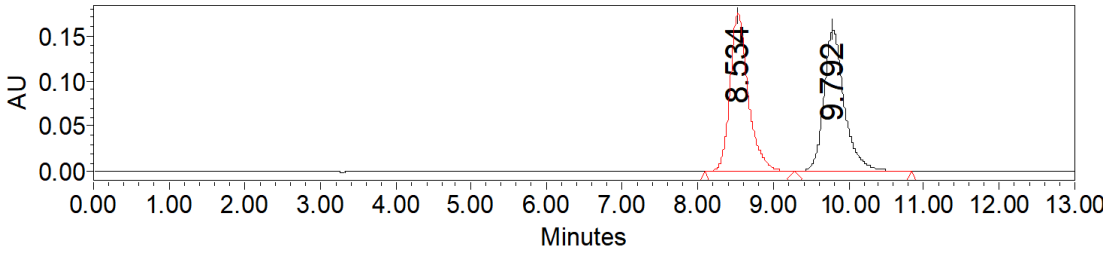
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{78.9183}BrO_4Na$: 485.0359, found: 485.0369;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{80.9163}BrO_4Na$: 487.0338, found: 487.0348;

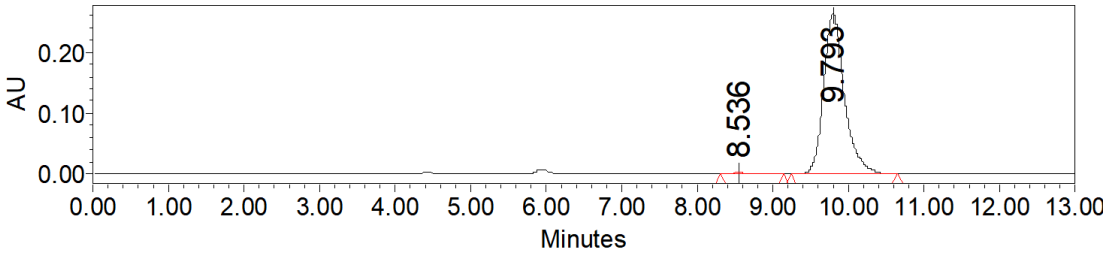
$[\alpha]_D^{20}$ = +433.1 (*c* = 0.82 in $CHCl_3$);

IR (neat): 3465, 3066, 2927, 1738, 1706, 1602, 1488, 1452, 1358, 1287, 1247, 1078, 970, 756, 723, 575 cm^{-1}.

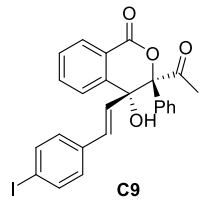
HPLC spectrum of **C8**



	Retention Time	Area	% Area
1	8.534	2979293	49.98
2	9.792	2981247	50.02



	Retention Time	Area	% Area
1	8.536	53136	1.05
2	9.793	5018478	98.95



C9

(3*S*,4*R*)-3-acetyl-4-hydroxy-4-((*E*)-4-iodostyryl)-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C9**; 0.1 mmol scale reaction; 29.6 mg, white solid, 58% yield, 96% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 11.93 min, t_2 = 13.06 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.81 – 7.78 (m, 1H), 7.73 (td, *J* = 7.6, 1.2 Hz, 1H), 7.66 – 7.64 (m, 2H), 7.52 – 7.47 (m, 3H), 7.45 – 7.39 (m, 3H), 6.82 – 6.80 (m, 2H), 6.04 (d, *J* = 16.0 Hz, 1H), 5.98 (d, *J* = 16.0 Hz, 1H), 5.25 (s, 1H), 2.13 (s, 3H);

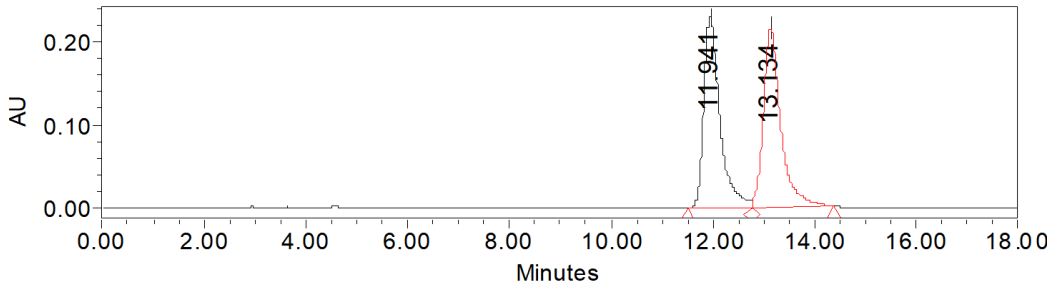
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.0, 163.0, 146.0, 137.5, 135.8, 135.4, 131.2, 131.0, 130.3, 129.2, 128.7, 128.5, 128.3, 128.3, 126.5, 125.1, 121.9, 93.4, 90.5, 27.4 ppm;

m.p. 73.2 – 75.4 °C;

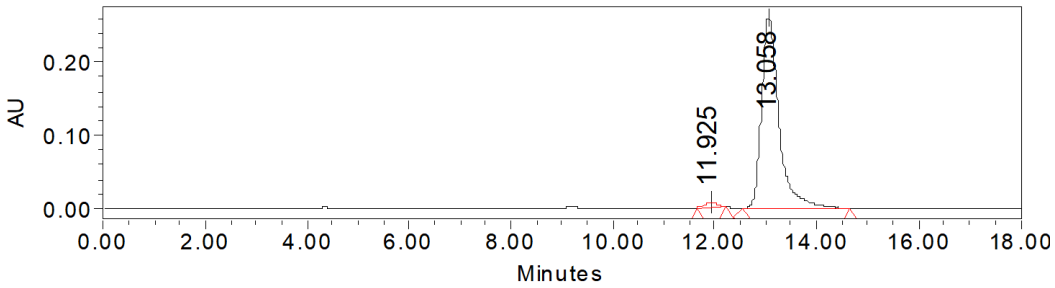
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}IO_4Na$: 533.0220, found: 533.0229;

$[\alpha]_D^{21}$ = +416.3 (*c* = 0.71 in $CHCl_3$);

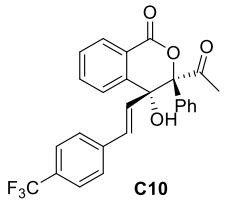
IR (neat): 3464, 3064, 2924, 1737, 1705, 1602, 1484,1452, 1357, 1287, 1247, 1212, 1082, 970, 755, 721, 572 cm^{-1}.
HPLC spectrum of **C9**



	Retention Time	Area	% Area
1	11.941	5068533	49.41
2	13.134	5189784	50.59

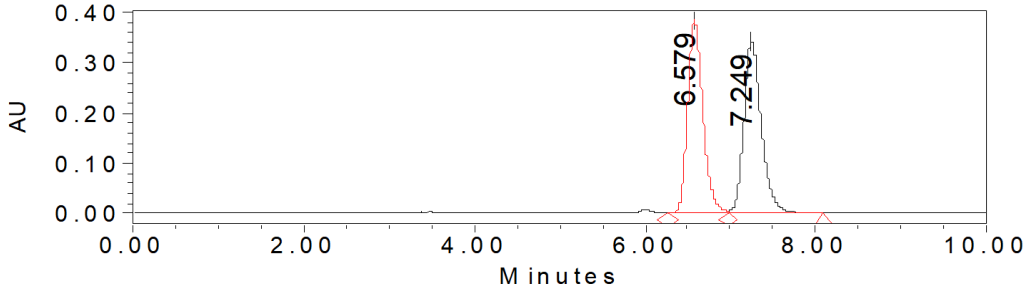


	Retention Time	Area	% Area
1	11.925	125552	1.97
2	13.058	6245403	98.03

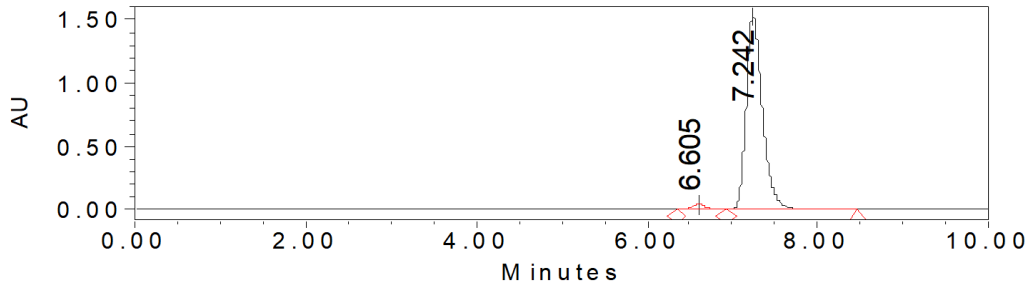


C10

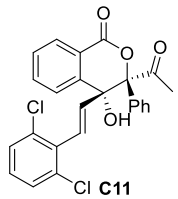
(3*S*,4*R*)-3-acetyl-4-hydroxy-3-phenyl-4-((*E*)-4-(trifluoromethyl)styryl)isochroman-1-one
The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C10**; 0.1 mmol scale reaction; 29.4 mg, white solid, 65% yield, 95% ee; determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t_1 = 6.60 min, t_2 = 7.24 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 7.6 Hz, 1H), 7.81 (d, *J* = 7.6 Hz, 1H), 7.74 (t, *J* = 7.6 Hz, 1H), 7.68 – 7.66 (m, 2H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.45 – 7.42 (m, 5H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.19 (d, *J* = 15.6 Hz, 1H), 6.10 (d, *J* = 16.0 Hz, 1H), 5.30 (s, 1H), 2.14 (s, 3H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.0, 162.9, 145.9, 139.4, 135.9, 131.2, 130.5, 130.4, 130.2, 129.8 (q, J_{C-F} = 32.3 Hz), 129.3, 128.8, 128.3, 126.8, 126.4, 125.3 (q, J_{C-F} = 272.7 Hz), 125.4 (q, J_{C-F} = 4.0 Hz), 125.1, 121.9, 90.5, 76.6, 27.4 ppm;
^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -62.59 ppm;
m.p. 76.7 – 80.7 °C;
HRMS (ESI) *m*/*z* $[M+Na]^+$ calcd for $C_{26}H_{19}F_3O_4Na$: 475.1128, found: 475.1135;
$[\alpha]_D^{21}$ = +452.4 (*c* = 1.09 in $CHCl_3$);
IR (neat): 3465, 3069, 2926, 1740, 1707, 1608, 1452, 1358, 1324, 1288, 1248, 1164, 1122, 1070, 755, 715, 575 cm^{-1}.
HPLC spectrum of **C10**



	Retention Time	Area	% Area
1	6.579	4860281	49.75
2	7.249	4908214	50.25



	Retention Time	Area	% Area
1	6.605	504812	2.41
2	7.242	20413363	97.59



C11

(3*S*,4*R*)-3-acetyl-4-((*E*)-2,6-dichlorostyryl)-4-hydroxy-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C11**; 0.1 mmol scale reaction; 33.5 mg, white solid, 74% yield, 98% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 6.85 min, t_2 = 8.48 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.79 – 7.73 (m, 3H), 7.48 (td, *J* = 7.6, 1.2 Hz, 1H), 7.45 – 7.40 (m, 3H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 8.0 Hz, 1H), 6.26 (d, *J* = 16.0 Hz, 1H), 6.05 (dd, *J* = 16.0, 1.2 Hz, 1H), 5.34 (d, *J* = 1.6 Hz, 1H), 2.13 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.0, 162.8, 146.0, 135.8, 135.4, 134.2, 133.6, 131.3, 130.2, 129.1, 128.7, 128.4, 128.3, 128.0, 126.7, 126.2, 125.2, 121.9, 90.1, 77.1, 27.2 ppm;

m.p. 148.4 – 151.7 °C;

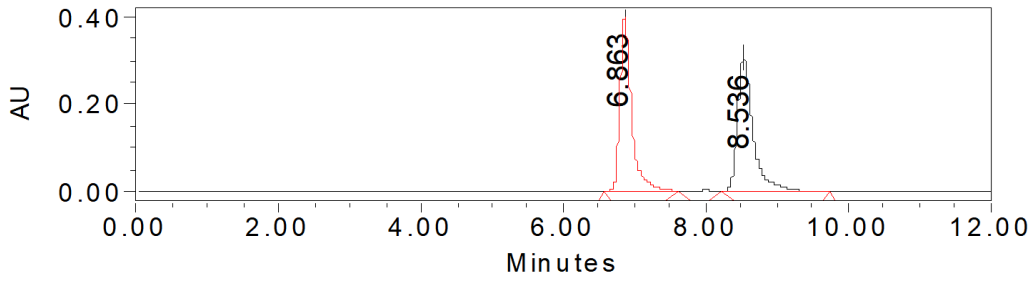
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{18}^{34.9689}Cl_2O_4Na$: 475.0474, found: 475.0485;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{18}^{36.9659}Cl_2O_4Na$: 477.0445, found: 477.0455;

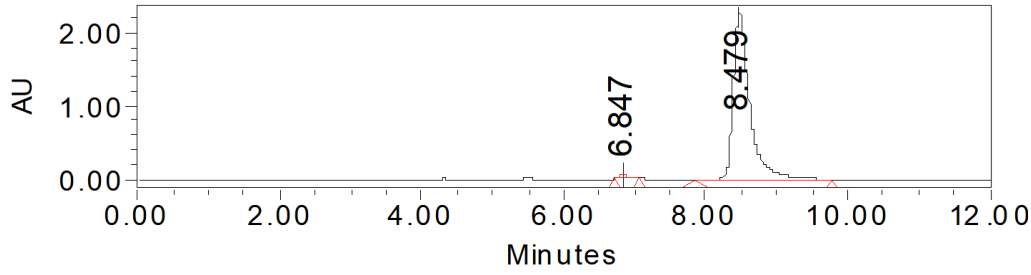
$[\alpha]_D^{20}$ = +393.9 (*c* = 0.93 in $CHCl_3$);

IR (neat): 3462, 3021, 2922, 1738, 1705, 1602, 1495, 1428, 1357, 1286, 1247, 1209, 1082, 1042, 967, 755, 729, 705, 573 cm^{-1}.

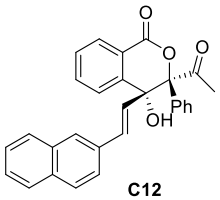
HPLC spectrum of **C11**



	Retention Time	Area	% Area
1	6.863	4668688	49.88
2	8.536	4690519	50.12



	Retention Time	Area	% Area
1	6.847	380137	1.04
2	8.479	36058456	98.96



C12

(3*S*,4*R*)-3-acetyl-4-hydroxy-4-((*E*)-2-(naphthalen-2-yl)vinyl)-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C12**; 0.1 mmol scale reaction; 26.5 mg, white solid, 61% yield, 95% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 12.16 min, t_2 = 14.14 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 – 8.16 (m, 1H), 7.86 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.77 – 7.65 (m, 6H), 7.51 (td, *J* = 7.6, 1.2 Hz, 1H), 7.48 – 7.39 (m, 6H), 7.30 – 7.27 (m, 1H), 6.29 (d, *J* = 15.6 Hz, 1H), 6.13 (dd, *J* = 16.0, 1.2 Hz, 1H), 5.31 (d, *J* = 0.8 Hz, 1H), 2.16 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.1, 163.1, 146.3, 135.8, 133.4, 133.3, 133.0, 132.2, 131.4, 130.3, 129.2, 128.6, 128.3, 128.0, 128.0, 127.9, 127.5, 127.0, 126.6, 126.2, 126.0, 125.2, 123.4, 122.0, 90.7, 76.9, 27.4 ppm;

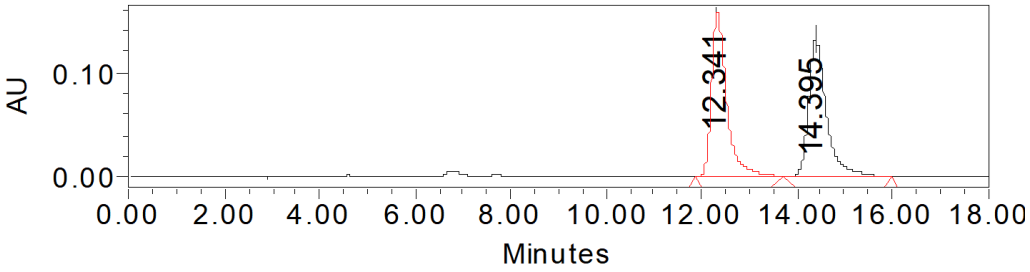
m.p. 128.1 – 130.8 °C;

HRMS (ESI) m/z [M+Na]$^+$ calcd for $C_{29}H_{22}O_4Na$: 457.1410, found: 457.1403;

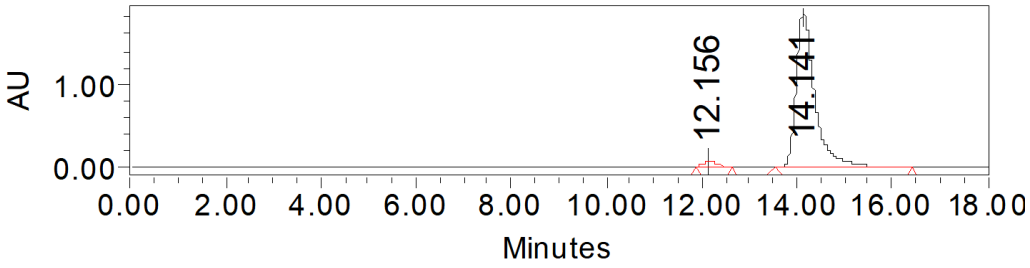
[α]$_D^{22}$ = +532.3 (*c* = 0.83 in $CHCl_3$);

IR (neat): 3464, 3020, 1738, 1706, 1602, 1452, 1356, 1287, 1248, 1082, 751, 710, 583 cm^{-1}.

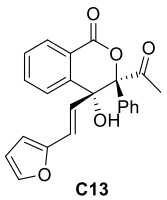
HPLC spectrum of **C12**



	Retention Time	Area	% Area
1	12.341	3591537	50.09
2	14.395	3578357	49.91



	Retention Time	Area	% Area
1	12.156	1222421	2.36
2	14.141	50546689	97.64



C13

(3*S*,4*R*)-3-acetyl-4-((*E*)-2-(furan-2-yl)vinyl)-4-hydroxy-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C13**; 0.1 mmol scale reaction; 26.2 mg, white solid, 70% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 7.26 min, t_2 = 9.78 min];

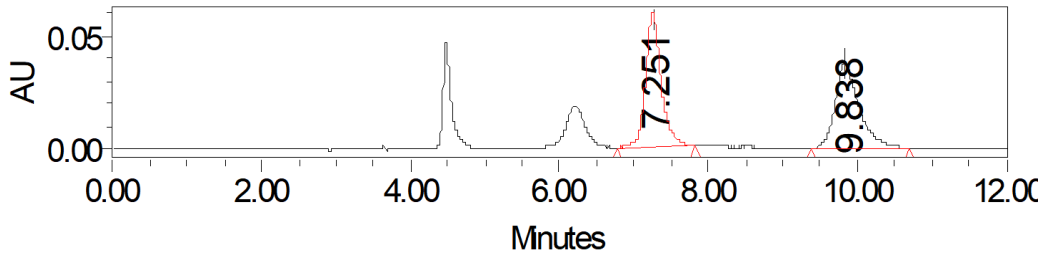
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.77 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.73 – 7.65 (m, 3H), 7.47 (td, *J* = 7.6, 1.2 Hz, 1H), 7.44 – 7.38 (m, 3H), 7.21 (d, *J* = 2.0 Hz, 1H), 6.24 (dd, *J* = 3.2, 1.6 Hz, 1H), 6.05 – 6.03 (m, 1H), 6.00 – 5.99 (m, 2H), 5.22 (d, *J* = 0.8 Hz, 1H), 2.12 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.1, 163.0, 151.7, 146.4, 142.2, 135.8, 131.2, 130.3, 129.1, 128.5, 128.3, 126.6, 125.8, 124.9, 121.8, 119.9, 111.2, 109.3, 90.6, 76.6, 27.4 ppm;

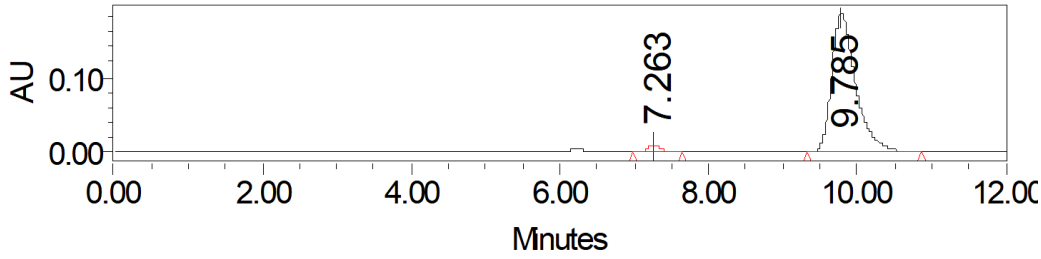
m.p. 75.9 – 78.2 °C;

HRMS (ESI) *m/z* [M+Na]$^+$ calcd for $C_{23}H_{18}O_5Na$: 397.1046, found: 397.1037;

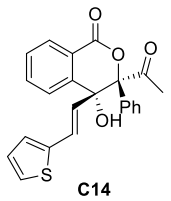
[α]$_D^{21}$ = +496.8 (*c* = 0.34 in $CHCl_3$);
IR (neat): 3464, 3025, 2924, 2338, 1739, 1706, 1602, 1491, 1452, 1357, 1287, 1248, 1081, 963, 750, 708, 576 cm^{-1}.
HPLC spectrum of **C13**



	Retention Time	Area	% Area
1	7.251	852548	50.47
2	9.838	836522	49.53

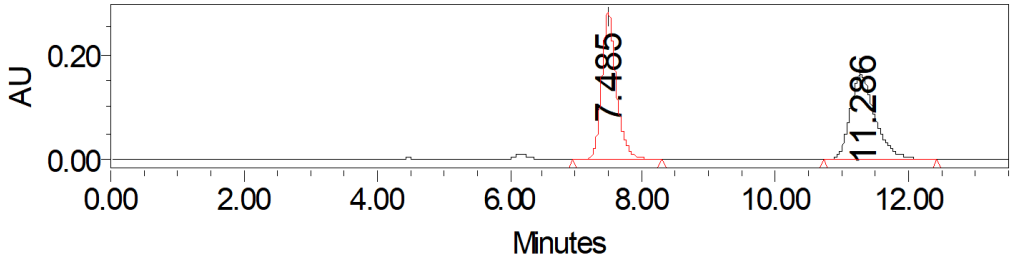


	Retention Time	Area	% Area
1	7.263	130677	3.05
2	9.785	4156254	96.95



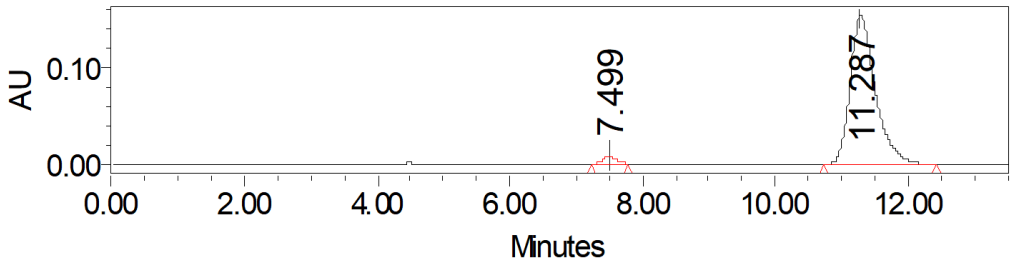
C14

(3*S*,4*R*)-3-acetyl-4-hydroxy-3-phenyl-4-((*E*)-2-(thiophen-2-yl)vinyl)isochroman-1-one
The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C14**; 0.1 mmol scale reaction; 27.7 mg, white solid, 71% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 7.50 min, t_2 = 11.29 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 – 8.11 (m, 1H), 7.80 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.73 (td, *J* = 7.2, 1.2 Hz, 1H), 7.67 – 7.64 (m, 2H), 7.49 (td, *J* = 7.6, 1.2 Hz, 1H), 7.46 – 7.38 (m, 3H), 7.08 – 7.06 (m, 1H), 6.84 (dd, *J* = 4.8, 3.6 Hz, 1H), 6.74 – 6.72 (m, 1H), 6.20 (d, *J* = 16.0 Hz, 1H), 5.82 (dd, *J* = 15.6, 1.2 Hz, 1H), 5.23 (d, *J* = 1.2 Hz, 1H), 2.13 (s, 3H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.1, 163.0, 146.0, 140.9, 135.8, 131.2, 130.3, 129.2, 128.6, 128.3, 127.2, 127.0, 126.7, 126.5, 125.2, 125.1, 124.9, 121.9, 90.6, 76.6, 27.5 ppm;
m.p. 70.7 – 73.3 °C;
HRMS (ESI) m/z $[M+Na]^+$ calcd for $C_{23}H_{18}O_4SNa$: 413.0818, found: 413.0825;
[α]$_D^{21}$ = +481.3 (*c* = 0.26 in $CHCl_3$);
IR (neat): 3463, 3069, 2924, 2361, 1739, 1706, 1602, 1452, 1356, 1287, 1248, 1210, 1082, 959, 753, 700, 564 cm^{-1}.
HPLC spectrum of **C14**

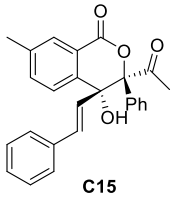


	Retention Time	Area	% Area
1	7.485	4364926	50.15

2	11.286	4338922	49.85



	Retention Time	Area	% Area
1	7.499	124095	2.94
2	11.287	4102791	97.06



C15

(3*S*,4*R*)-3-acetyl-4-hydroxy-7-methyl-3-phenyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C15**; 0.1 mmol scale reaction; 30.2 mg, white solid, 76% yield, 95% ee; determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 5.56 min, t_2 = 6.61 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.0 Hz, 1H), 7.69 – 7.66 (m, 2H), 7.60 (s, 1H), 7.45 – 7.38 (m, 3H), 7.29 – 7.27 (m, 1H), 7.22 – 7.14 (m, 3H), 7.11 – 7.08 (m, 2H), 6.11 (d, *J* = 16.0 Hz, 1H), 5.98 (d, *J* = 16.0 Hz, 1H), 5.22 (s, 1H), 2.47 (s, 3H), 2.13 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.4, 163.2, 147.2, 146.2, 136.0, 131.9, 131.4, 130.4, 129.5, 129.1, 128.4, 128.3, 127.9, 127.8, 126.6, 126.6, 125.5, 119.3, 90.6, 76.8, 27.5, 22.2 ppm;

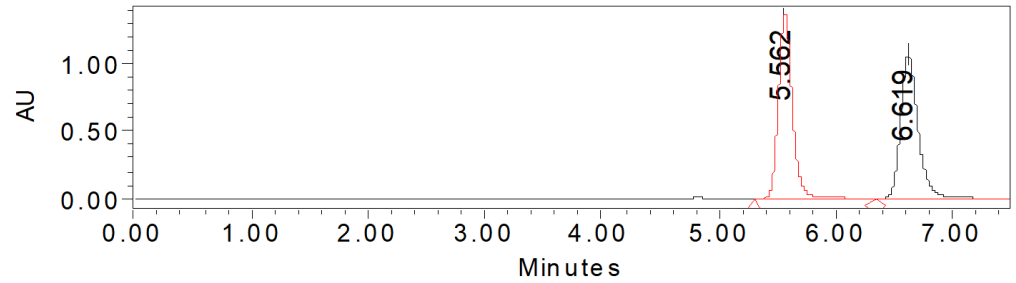
m.p. 128.7 – 131.6 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{26}H_{22}O_4Na$: 421.1410, found: 421.1408;

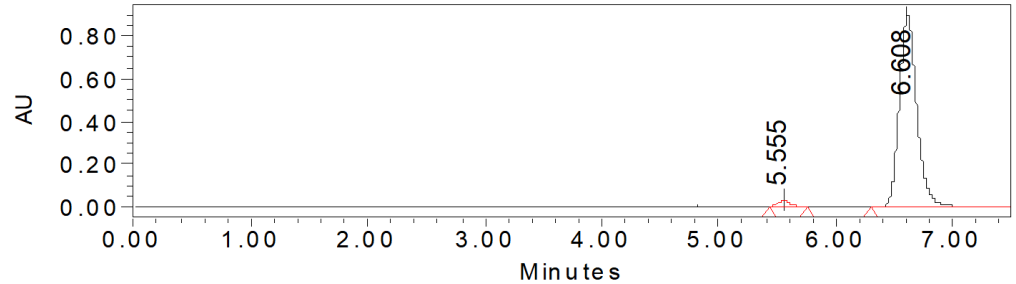
$[\alpha]_D^{21}$ = +407.1 (*c* = 0.96 in $CHCl_3$);

IR (neat): 3461, 3024, 2922, 2361, 1737, 1704, 1607, 1495, 1448, 1357, 1283, 1256, 1214, 1071, 770, 748, 693, 591, 550 cm^{-1}.

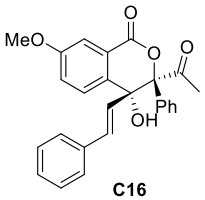
HPLC spectrum of **C15**



	Retention Time	Area	% Area
1	5.562	11101522	49.86
2	6.619	11162483	50.14



	Retention Time	Area	% Area
1	5.555	238836	2.50
2	6.608	9302608	97.50



C16

(3*S*,4*R*)-3-acetyl-4-hydroxy-7-methoxy-3-phenyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C16**; 0.1 mmol scale reaction; 31.5 mg, white solid, 76% yield, 92% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 12.05 min, t_2 = 13.84 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (dd, *J* = 8.8, 1.2 Hz, 1H), 7.69 – 7.66 (m, 2H), 7.45 – 7.38 (m, 3H), 7.26 – 7.25 (m, 1H), 7.22 – 7.14 (m, 3H), 7.11 – 7.09 (m, 2H), 6.98 – 6.95 (m, 1H), 6.13 (d, *J* = 16.0 Hz, 1H), 5.98 (d, *J* = 16.0 Hz, 1H), 5.29 (s, 1H), 3.91 (s, 3H), 2.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.6, 165.6, 162.9, 149.1, 135.9, 132.8, 132.0, 131.4, 129.1, 128.4, 128.2, 127.9, 127.5, 126.6, 115.3, 114.3, 109.1, 90.5, 76.9, 55.8, 27.4 ppm;

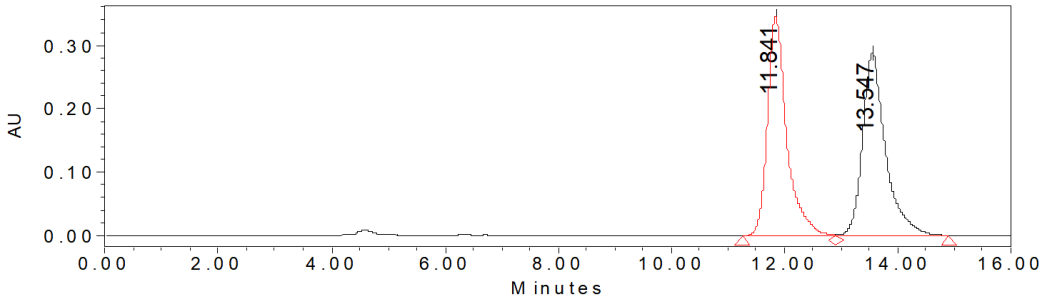
m.p. 87.5 – 91.6 °C;

HRMS (ESI) m/z $[M+Na]^+$ calcd for $C_{26}H_{22}O_5Na$: 437.1360, found: 437.1360;

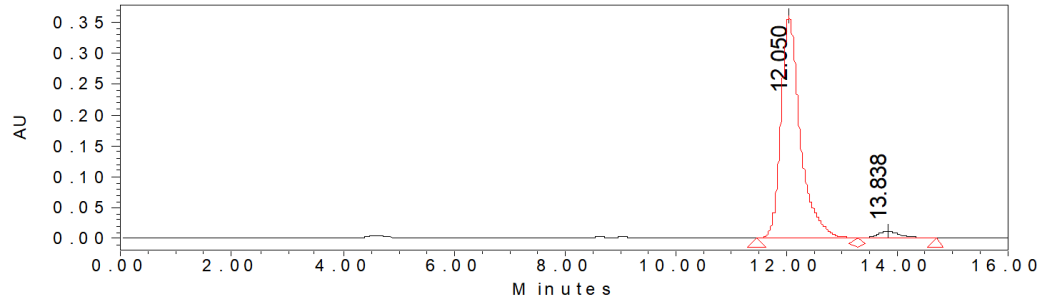
$[\alpha]_D^{21}$ = +312.4 (*c* = 0.44 in $CHCl_3$);

IR (neat): 3462, 3061, 2941, 2361, 1732, 1707, 1603, 1492, 1448, 1264, 1214, 1077, 771, 751, 695, 590, 552 cm^{-1}.

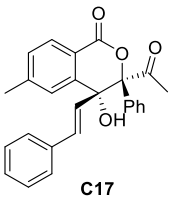
HPLC spectrum of **C16**



	Retention Time	Area	% Area
1	11.841	8116330	49.92
2	13.547	8142419	50.08



	Retention Time	Area	% Area
1	12.050	8767417	96.30
2	13.838	337137	3.70



C17

(3*S*,4*R*)-3-acetyl-4-hydroxy-6-methyl-3-phenyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C17**; 0.1 mmol scale reaction; 28.3 mg, white solid, 71% yield, 91% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 7.31 min, t_2 = 8.91 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.92 (m, 1H), 7.70 – 7.67 (m, 3H), 7.55 – 7.52 (m, 1H), 7.46 – 7.38 (m, 3H), 7.21 – 7.13 (m, 3H), 7.09 – 7.06 (m, 2H), 6.07 (d, *J* = 16.0 Hz, 1H), 5.96 (dd, *J* = 16.0, 1.2 Hz, 1H), 5.21 (d, *J* = 1.2 Hz, 1H), 2.44 (s, 3H), 2.13 (s, 3H);

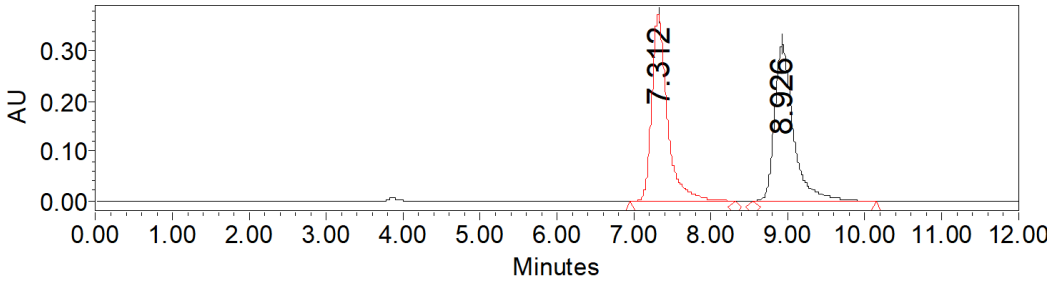
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.2, 163.6, 143.3, 138.7, 136.7, 136.0, 132.0, 131.4, 130.4, 129.1, 128.4, 128.3, 127.9, 127.9, 126.6, 126.6, 125.1, 121.8, 90.6, 76.8, 27.5, 21.0 ppm;

m.p. 66.6 – 69.8 °C;

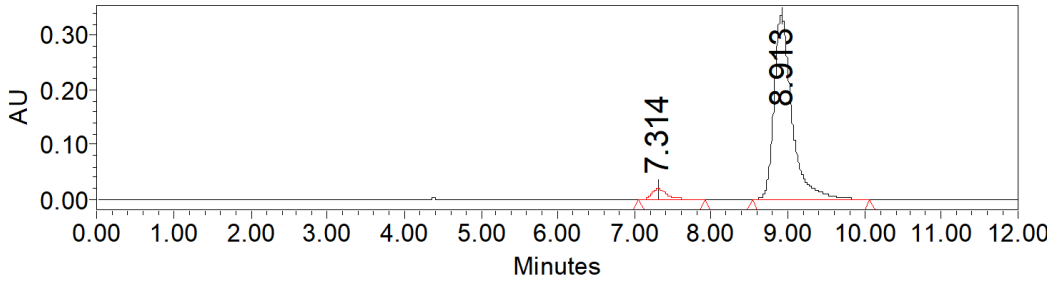
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{26}H_{22}O_4Na$: 421.1410, found: 421.1407;

$[\alpha]_D^{21}$ = +472.0 (*c* = 0.49 in $CHCl_3$);

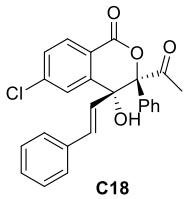
IR (neat): 3464, 3060, 3026, 2924, 1736, 1705, 1613, 1494, 1357, 1283, 1251, 1210, 1181, 1079, 1044, 969, 749, 695, 593, 561 cm^{-1}.
HPLC spectrum of **C17**



	Retention Time	Area	% Area
1	7.312	5329922	50.02
2	8.926	5324603	49.98

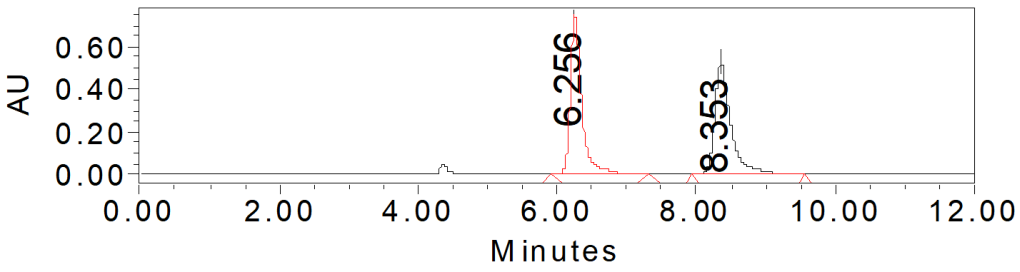


	Retention Time	Area	% Area
1	7.314	254877	4.42
2	8.913	5507908	95.58

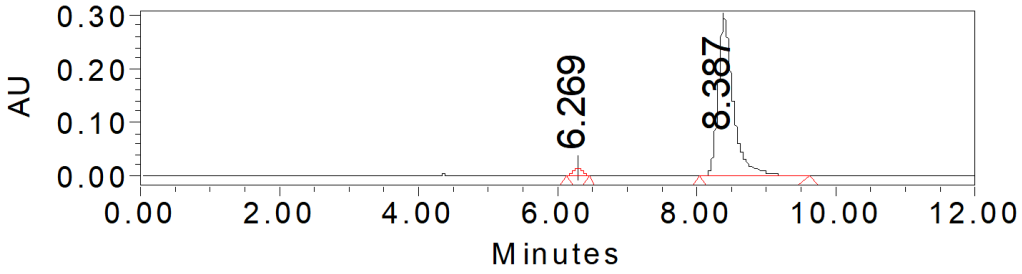


C18

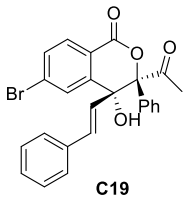
(3*S*,4*R*)-3-acetyl-6-chloro-4-hydroxy-3-phenyl-4-((*E*)-styryl)isochroman-1-one
The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C18**; 0.1 mmol scale reaction; 30.1 mg, white solid, 72% yield, 95% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 6.27 min, t_2 = 8.39 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 2.0 Hz, 1H), 7.77 (d, *J* = 8.4 Hz, 1H), 7.70 – 7.63 (m, 3H), 7.45 – 7.41 (m, 3H), 7.22 – 7.15 (m, 3H), 7.09 – 7.06 (m, 2H), 6.09 (d, *J* = 16.0 Hz, 1H), 5.93 (dd, *J* = 16.0, 0.8 Hz, 1H), 5.26 (d, *J* = 0.8 Hz, 1H), 2.14 (s, 3H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.9, 161.9, 144.7, 135.8, 135.7, 134.9, 132.5, 131.0, 129.8, 129.4, 128.4, 128.4, 128.1, 127.3, 127.1, 126.7, 126.5, 123.5, 90.7, 76.7, 27.4 ppm;
m.p. 58.7 – 63.4 °C;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{34.9689}ClO_4Na$: 441.0864, found: 441.0858;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{36.9659}ClO_4Na$: 443.0835, found: 443.0829;
$[\alpha]_D^{22}$ = +410.1 (*c* = 0.50 in $CHCl_3$);
IR (neat): 3464, 3063, 2925, 1742, 1707, 1598, 1449, 1358, 1299, 1243, 1078, 969, 752, 695, 593, 559 cm^{-1}.
HPLC spectrum of **C18**



	Retention Time	Area	% Area
1	6.256	8582517	50.01
2	8.353	8580724	49.99



	Retention Time	Area	% Area
1	6.269	121592	2.57
2	8.387	4610064	97.43



C19

(3*S*,4*R*)-3-acetyl-6-bromo-4-hydroxy-3-phenyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C19**; 0.1 mmol scale reaction; 35.7 mg, white solid, 77% yield, 95% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 6.94 min, t_2 = 9.82 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 – 8.24 (m, 1H), 7.85 – 7.83 (m, 1H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.66 – 7.64 (m, 2H), 7.46 – 7.39 (m, 3H), 7.23 – 7.15 (m, 3H), 7.09 – 7.07 (m, 2H), 6.09 (d, *J* = 16.0 Hz, 1H), 5.93 (d, *J* = 16.0 Hz, 1H), 5.26 (s, 1H), 2.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.9, 161.8, 145.2, 138.7, 135.7, 132.8, 132.5, 131.0, 129.4, 128.4, 128.4, 128.1, 127.2, 127.1, 126.7, 126.5, 123.6, 122.6, 90.6, 76.7, 27.5 ppm;

m.p. 70.9 – 75.0 °C;

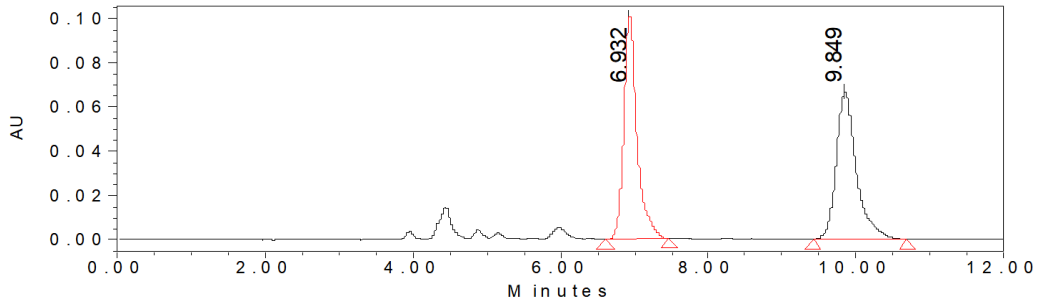
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}^{78.9183}BrO_4Na$: 485.0359, found: 485.0359;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}^{80.9163}BrO_4Na$: 487.0339, found: 487.0338;

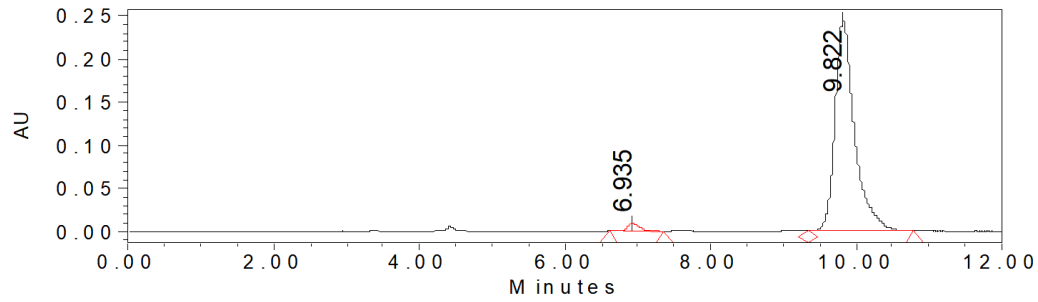
$[\alpha]_D^{21}$ = +422.9 (*c* = 0.40 in $CHCl_3$);

IR (neat): 3463, 3063, 3027, 2361, 2337, 1742, 1706, 1594, 1450, 1358, 1241, 1215, 1083, 969, 769, 754, 695, 593, 557 cm^{-1}.

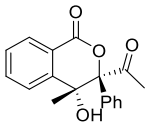
HPLC spectrum of **C19**



	Retention Time	Area	% Area
1	6.932	1252127	49.93
2	9.849	1255614	50.07



	Retention Time	Area	% Area
1	6.935	115818	2.46
2	9.822	4590776	97.54



C20

(3*S*,4*R*)-3-acetyl-4-hydroxy-4-methyl-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1.5) to afford the desired product **C20**; 0.1 mmol scale reaction; 26.7 mg, white solid, 90% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 230 nm, t_1 = 5.61 min, t_2 = 6.48 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.0 Hz, 1H), 7.82 (d, *J* = 7.6 Hz, 1H), 7.76 – 7.68 (m, 3H), 7.50 – 7.42 (m, 4H), 4.86 (s, 1H), 2.08 (s, 3H), 1.16 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.2, 163.0, 149.2, 135.7, 131.6, 130.3, 129.2, 128.5, 128.2, 126.5, 124.1, 121.3, 90. 7, 74.5, 27.3, 25.8 ppm;

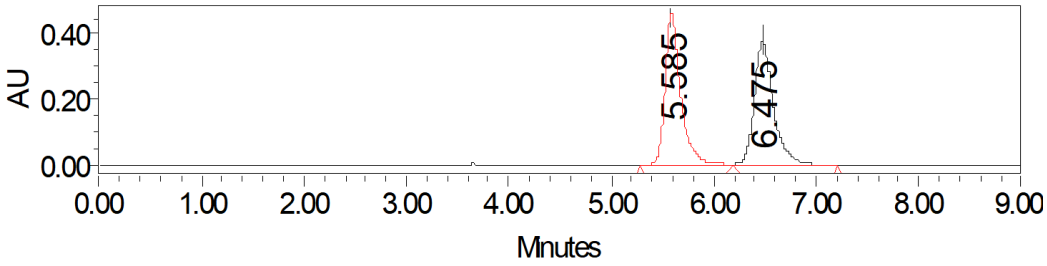
m.p. 100.2 – 102.8°C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{18}H_{16}O_4Na$: 319.0941, found: 319.0940;

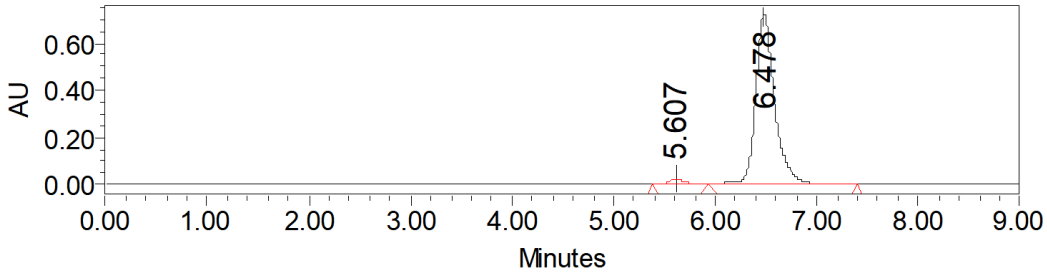
$[\alpha]_D^{21}$ = +421.7 (*c* = 0.95 in $CHCl_3$);

IR (neat): 3476, 3067, 2933, 1738, 1706, 1603, 1452, 1357, 1290, 1257, 1212, 1099, 1052, 755, 705, 602, 579, 559 cm^{-1}.

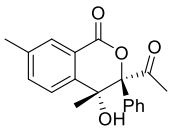
HPLC spectrum of **C20**



	Retention Time	Area	% Area
1	5.585	4893032	50.02
2	6.475	4889725	49.98



	Retention Time	Area	% Area
1	5.607	281685	2.90
2	6.478	9417063	97.10



C21

(3*S*,4*R*)-3-acetyl-4-hydroxy-4,7-dimethyl-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1.5) to afford the desired product **C21**; 0.1 mmol scale reaction; 14.9 mg, white solid, 48% yield, 91% ee; determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 230 nm, t_1 = 5.97 min, t_2 = 7.88 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.0 Hz, 1H), 7.75 – 7.72 (m, 2H), 7.60 (s, 1H), 7.49 – 7.44 (m, 3H), 7.23 (dd, *J* = 8.0, 1.6 Hz, 1H), 4.83 (s, 1H), 2.47 (s, 3H), 2.08 (s, 3H), 1.15 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.5, 163.1, 149.2, 147.1, 131.6, 130.4, 129.2, 129.1, 128.5, 126.6, 124.5, 118.6, 90.6, 74.5, 27.3, 25.8, 22.2 ppm;

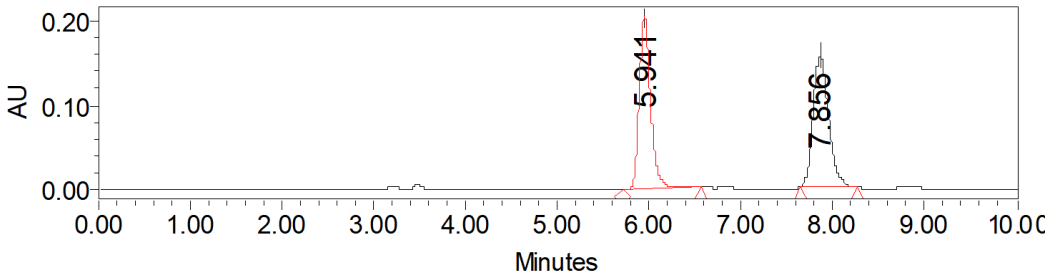
m.p. 164.7 – 169.3 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{19}H_{18}O_4Na$: 333.1097, found: 333.1094;

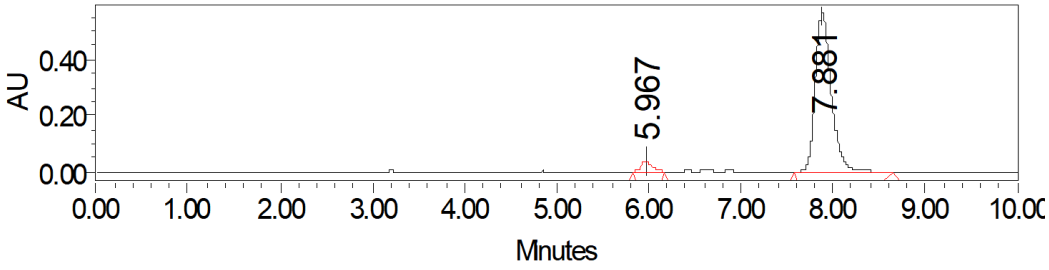
$[\alpha]_D^{21}$ = +301.7 (*c* = 0.29 in $CHCl_3$);

IR (neat): 3475, 2922, 2361, 1740, 1706, 1610, 1449, 1359, 1269, 1218, 1105, 1054, 773, 701, 588 cm^{-1}.

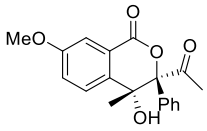
HPLC spectrum of **C21**



	Retention Time	Area	% Area
1	5.941	1737080	48.64
2	7.856	1834056	51.36



	Retention Time	Area	% Area
1	5.967	311761	4.41
2	7.881	6763155	95.59



C22

(3*S*,4*R*)-3-acetyl-4-hydroxy-7-methoxy-4-methyl-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1.5) to afford the desired product **C22**; 0.1 mmol scale reaction; 16.0 mg, white solid, 49% yield, 96% ee; determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 230 nm, t_1 = 8.78 min, t_2 = 12.07 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.8 Hz, 1H), 7.74 – 7.71 (m, 2H), 7.49 – 7.41 (m, 3H), 7.27 – 7.26 (m, 1H), 6.91 (dd, *J* = 8.4, 2.4 Hz, 1H), 4.89 (s, 1H), 3.92 (s, 3H), 2.08 (s, 3H), 1.15 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.6, 165.6, 162.8, 152.0, 132.9, 131.6, 129.1, 128.4, 126.6, 114.8, 113.6, 108.4, 90.5, 74.6, 55.8, 27.2, 25.6 ppm;

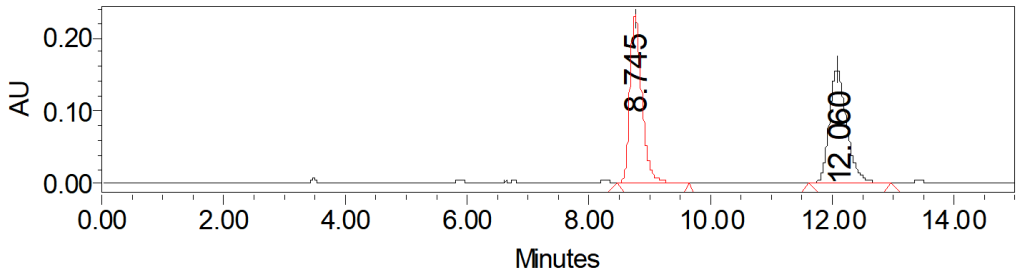
m.p. 127.2 – 129.8 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{19}H_{18}O_5Na$: 349.1046, found: 349.1041;

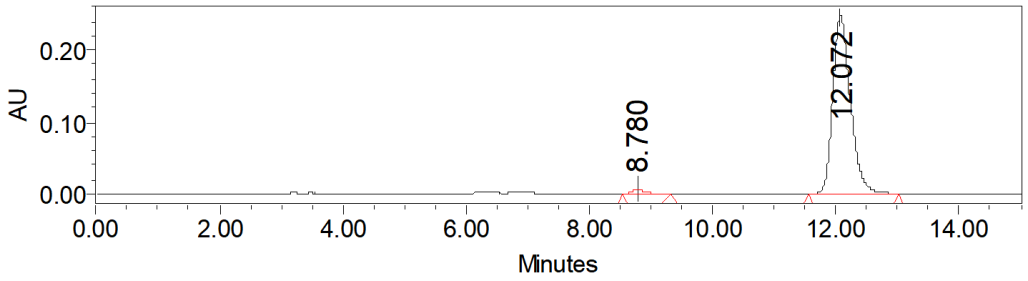
$[\alpha]_D^{28}$ = +372.5 (*c* = 0.49 in $CHCl_3$);

IR (neat): 3476, 2933, 2846, 2332, 1732, 1605, 1492, 1449, 1358, 1329, 1274, 1093, 1049, 773, 733, 702, 597 cm^{-1}.

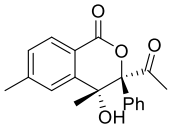
HPLC spectrum of **C22**



	Retention Time	Area	% Area
1	8.745	3198032	50.29
2	12.060	3160710	49.71



	Retention Time	Area	% Area
1	8.780	100233	1.99
2	12.072	4925776	98.01



C23

(3*S*,4*R*)-3-acetyl-4-hydroxy-4,6-dimethyl-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1.5) to afford the desired product **C23**; 0.1 mmol scale reaction; 25.8 mg, white solid, 83% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 230 nm, t_1 = 5.40 min, t_2 = 6.46 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (s, 1H), 7.75 – 7.73 (m, 2H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.52 – 7.43 (m, 4H), 4.83 (s, 1H), 2.41 (s, 3H), 2.07 (s, 3H), 1.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.3, 163.2, 146.4, 138.3, 136.6, 131.6, 130.4, 129.1, 128.5, 126.5, 124.1, 121.1, 90.7, 74.5, 27.3, 25.8, 20.9 ppm;

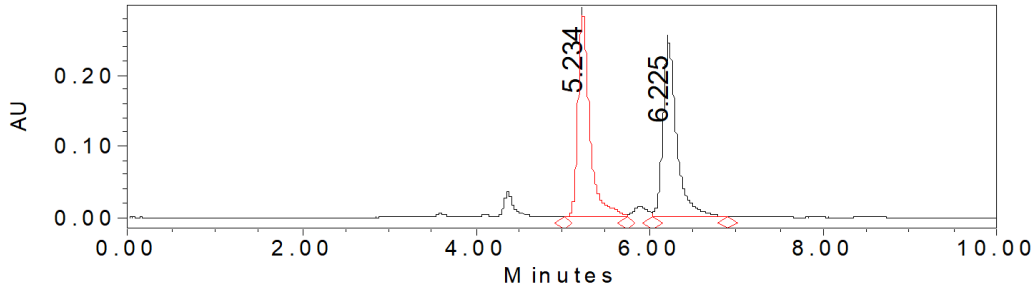
m.p. 116.6 – 118.1 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{19}H_{18}O_4Na$: 333.1097, found: 333.1093;

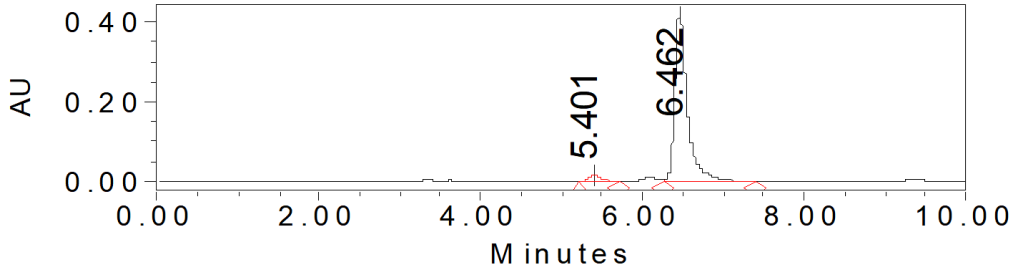
$[\alpha]_D^{21}$ = +419.4 (*c* = 0.79 in $CHCl_3$);

IR (neat): 3477, 2987, 2929, 1737, 1707, 1495, 1447, 1358, 1262, 1212, 1181, 1110, 1051, 780, 737, 698, 563 cm^{-1}.

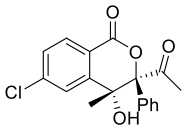
HPLC spectrum of **C23**



	Retention Time	Area	% Area
1	5.234	2742574	50.09
2	6.225	2732882	49.91



	Retention Time	Area	% Area
1	5.401	142897	3.01
2	6.462	4605005	96.99



C24

(3*S*,4*R*)-3-acetyl-6-chloro-4-hydroxy-4-methyl-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1.5) to afford the desired product **C24**; 0.1 mmol scale reaction; 13.9 mg, white solid, 42% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 230 nm, t_1 = 5.37 min, t_2 = 6.69 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 2.4 Hz, 1H), 7.77 (d, *J* = 8.4 Hz, 1H), 7.73 – 7.69 (m, 2H), 7.66 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.50 – 7.43 (m, 3H), 4.88 (s, 1H), 2.08 (s, 3H), 1.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.9, 161.8, 147.6, 135.7, 134.5, 131.2, 129.9, 129.3, 128.6, 126.4, 126.0, 122.8, 90.7, 74.3, 27.2, 25.8 ppm;

m.p. 110.7 – 114.9 °C;

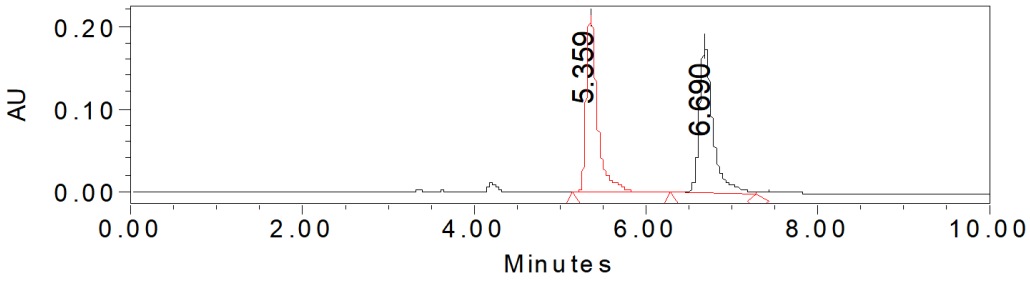
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{18}H_{15}^{34.9689}ClO_4Na$: 353.0551, found: 353.0551;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{18}H_{15}^{36.9659}ClO_4Na$: 355.0522, found: 355.0518;

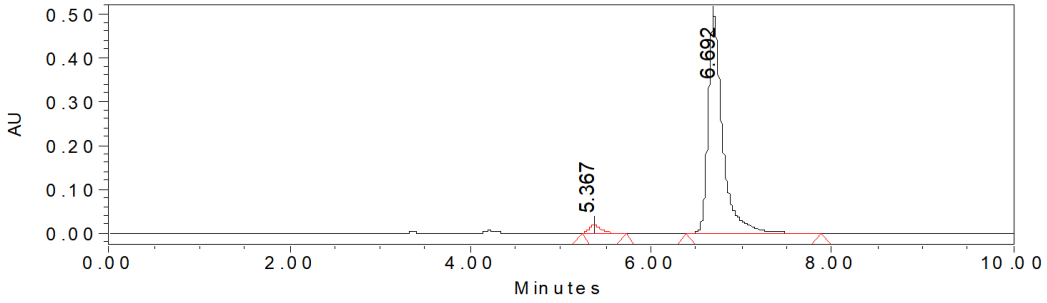
$[\alpha]_D^{20}$ = +387.4 (*c* = 0.52 in $CHCl_3$);

IR (neat): 3474, 3068, 2856, 1740, 1707, 1458, 1409, 1358, 1248, 1213, 1109, 1049, 840, 766, 719, 699, 604, 558 cm^{-1}.

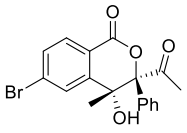
HPLC spectrum of **C24**



	Retention Time	Area	% Area
1	5.359	1987684	50.16
2	6.690	1974887	49.84



	Retention Time	Area	% Area
1	5.367	161919	2.79
2	6.692	5634501	97.21



C25

(3*S*,4*R*)-3-acetyl-6-bromo-4-hydroxy-4-methyl-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1.5) to afford the desired product **C25**; 0.1 mmol scale reaction; 30.8 mg, white solid, 82% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 230 nm, t_1 = 5.49 min, t_2 = 7.01 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 2.0 Hz, 1H), 7.81 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.73 – 7.69 (m, 3H), 7.50 – 7.45 (m, 3H), 4.87 (s, 1H), 2.08 (s, 3H), 1.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.9, 161.7, 148.1, 138.6, 132.9, 131.2, 129.3, 128.6, 126.4, 126.2, 123.0, 122.2, 90.7, 74.4, 27.2, 25.8 ppm;

m.p. 110.4 – 113.6 °C;

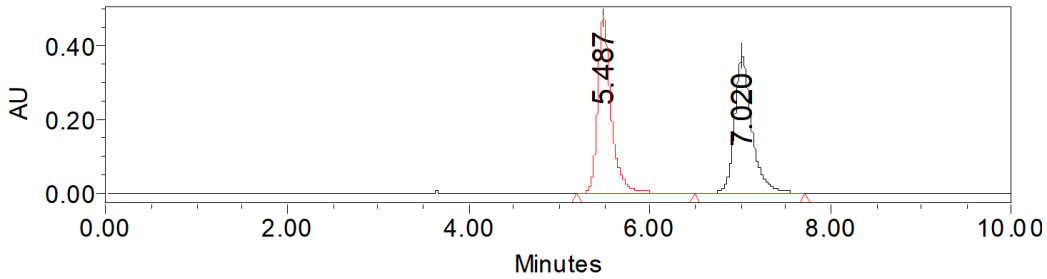
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{18}H_{15}^{78.9183}BrO_4Na$: 397.0046, found: 397.0044;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{18}H_{15}^{80.9163}BrO_4Na$: 399.0025, found: 399.0023;

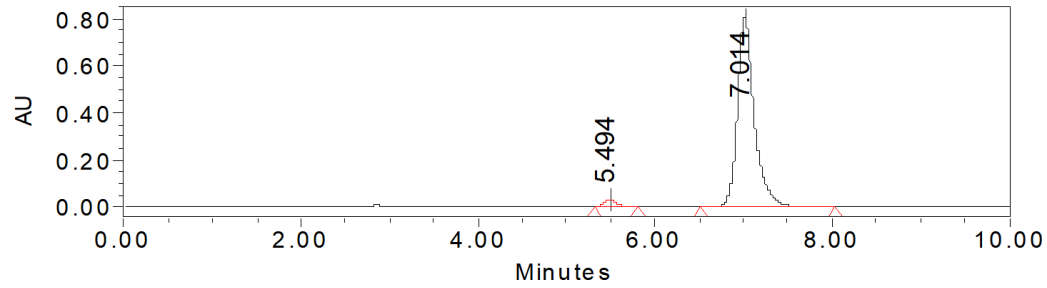
$[\alpha]_D^{21}$ = +332.6 (*c* = 0.63 in $CHCl_3$);

IR (neat): 3474, 3067, 2932, 2361, 1743, 1707, 1449, 1404, 1358, 1245, 1213, 1108, 1050, 762, 713, 700, 609, 556 cm^{-1}.

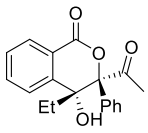
HPLC spectrum of **C25**



	Retention Time	Area	% Area
1	5.487	4845550	50.14
2	7.020	4818639	49.86



	Retention Time	Area	% Area
1	5.494	309841	2.78
2	7.014	10826655	97.22



C26

(3*S*,4*R*)-3-acetyl-4-ethyl-4-hydroxy-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1.5) to afford the desired product **C26**; 0.1 mmol scale reaction; 19.6 mg, colorless oil, 63% yield, 96% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 230 nm, t_1 = 5.05 min, t_2 = 6.05 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 7.6 Hz, 1H), 7.76 – 7.67 (m, 4H), 7.49 – 7.42 (m, 4H), 4.83 (d, *J* = 2.4 Hz, 1H), 2.05 (s, 3H), 1.66 – 1.58 (m, 2H), 1.39 – 1.30 (m, 1H), 0.58 (t, *J* = 7.2 Hz, 3H);

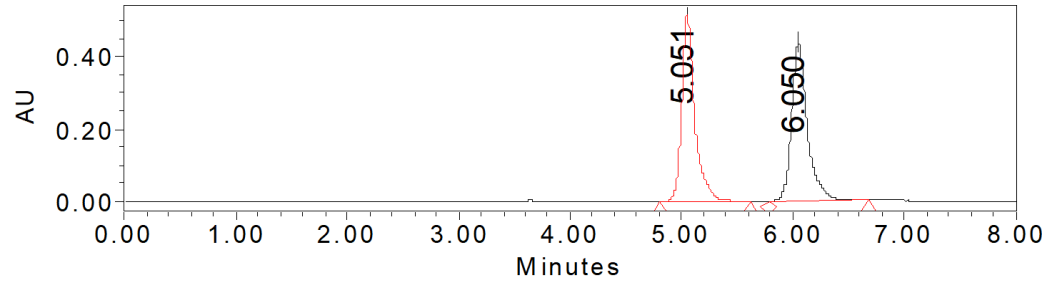
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.4, 163.1, 146.6, 134.7, 131.5, 130.3, 129.1, 128.5, 128.3, 126.7, 126.0, 121.8, 91.1, 76.5, 29.4, 27.2, 6.5 ppm;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{19}H_{18}O_4Na$: 333.1097, found: 333.1093;

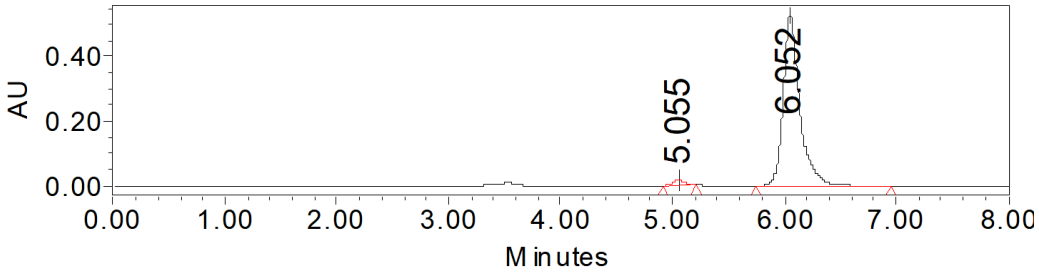
$[\alpha]_D^{21}$ = +374.7 (*c* = 0.44 in $CHCl_3$);

IR (neat): 3478, 2974, 2937, 2361, 1741, 1707, 1603, 1452, 1358, 1289, 1249, 1102, 1068, 772, 752, 710, 594 cm^{-1}.

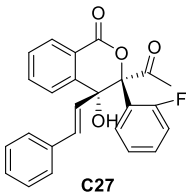
HPLC spectrum of **C26**



	Retention Time	Area	% Area
1	5.051	4250009	49.59
2	6.050	4320069	50.41



	Retention Time	Area	% Area
1	5.055	110384	1.99
2	6.052	5436258	98.01



C27

(3*S*,4*R*)-3-acetyl-3-(2-fluorophenyl)-4-hydroxy-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C27**; 0.1 mmol scale reaction; 29.7 mg, white solid, 74% yield, 82% ee; determined by HPLC analysis [Daicel chiralpak ID, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t_1 = 7.95 min, t_2 = 15.79 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.6 Hz, 1H), 7.79 – 7.70 (m, 3H), 7.46 (td, *J* = 7.6, 1.6 Hz, 1H), 7.42 – 7.37 (m, 1H), 7.24 – 7.18 (m, 4H), 7.14 – 7.10 (m, 3H), 6.15 (d, *J* = 16.0 Hz, 1H), 6.01 (d, *J* = 16.0 Hz, 1H), 4.71 (s, 1H), 2.23 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 206.9, 162.6, 159.6 (d, $J_{C\text{-}F}$ = 249.5 Hz), 146.2, 135.9, 135.8, 131.7, 131.3 (d, $J_{C\text{-}F}$ = 8.1 Hz), 130.2, 129.9 (d, $J_{C\text{-}F}$ = 3.0 Hz), 128.4, 128.2, 128.0, 127.9, 126.6, 124.8, 124.1 (d, $J_{C\text{-}F}$ = 3.0 Hz), 121.5, 120.9 (d, $J_{C\text{-}F}$ = 11.1 Hz), 116.3 (d, $J_{C\text{-}F}$ = 23.2 Hz), 88.3 (d, $J_{C\text{-}F}$ = 5.1 Hz), 76.5, 26.4 ppm;

^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -109.72 ppm;

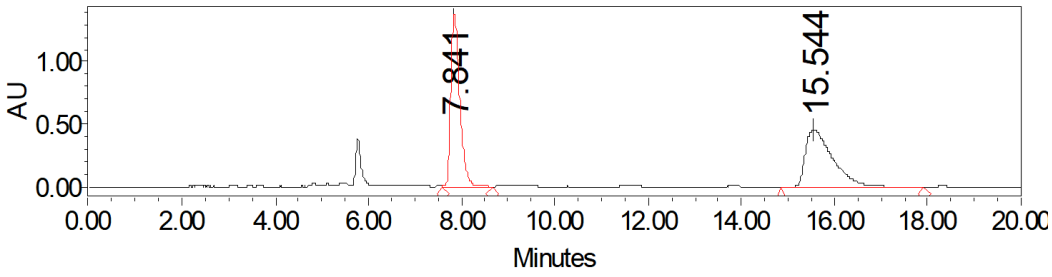
m.p. 135.5 – 138.4 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}FO_4Na$: 425.1160, found: 425.1158;

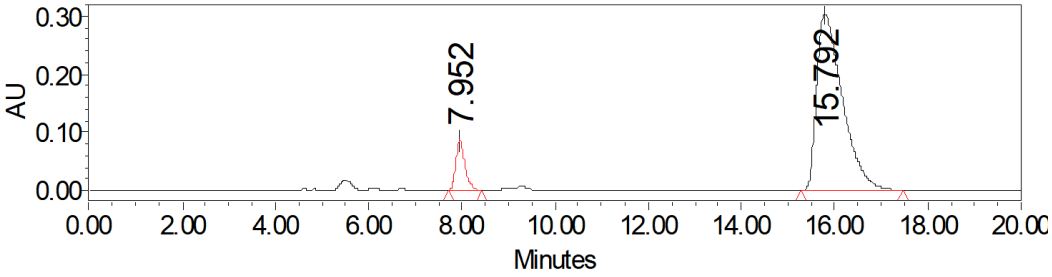
$[\alpha]_D^{27}$ = +438.1 (*c* = 0.58 in $CHCl_3$);

IR (neat): 3500, 3067, 2924, 2360, 1740, 1490, 1453, 1359, 1284, 1246, 1077, 1035, 768, 694 cm^{-1}.

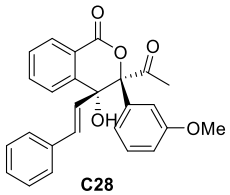
HPLC spectrum of **C27**



	Retention Time	Area	% Area
1	7.841	18064852	49.71
2	15.544	18275148	50.29



	Retention Time	Area	% Area
1	7.952	1153454	8.93
2	15.792	11761161	91.07



C28

(3*S*,4*R*)-3-acetyl-4-hydroxy-3-(3-methoxyphenyl)-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/2) to afford the desired product **C28**; 0.1 mmol scale reaction; 38.1 mg, white solid, 92% yield, 96% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 9.91 min, t_2 = 11.16 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 7.2 Hz, 1H), 7.81 (d, *J* = 7.6 Hz, 1H), 7.73 (td, *J* = 7.6, 1.6 Hz, 1H), 7.49 (td, *J* = 7.6, 1.2 Hz, 1H), 7.34 (t, *J* = 8.0 Hz, 1H), 7.26 – 7.16 (m, 5H), 7.11 – 7.09 (m, 2H), 6.96 – 6.93 (m, 1H), 6.11 (d, *J* = 16.0 Hz, 1H), 6.00 (d, *J* = 16.0 Hz, 1H), 5.21 (s, 1H), 3.79 (s, 3H), 2.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.0, 163.0, 159.5, 146.3, 136.0, 135.8, 132.8, 132.1, 130.2, 129.3, 128.6, 128.4, 128.0, 127.8, 126.6, 125.2, 122.0, 118.9, 114.9, 112.3, 90.5, 76.8, 55.4, 27.4 ppm;

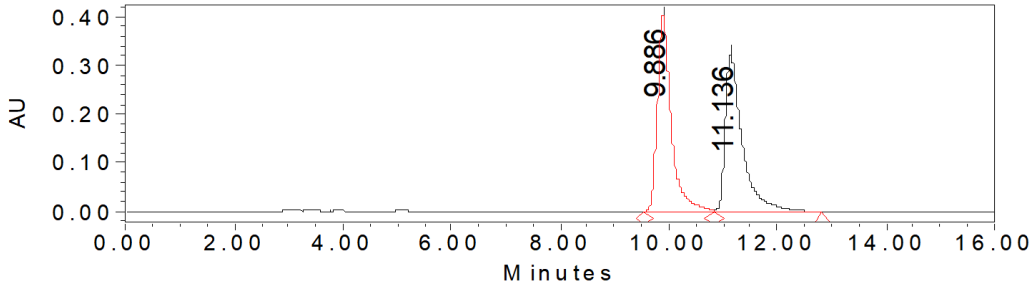
m.p. 99.8 – 104.2 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{26}H_{22}O_5Na$: 437.1359, found: 437.1359;

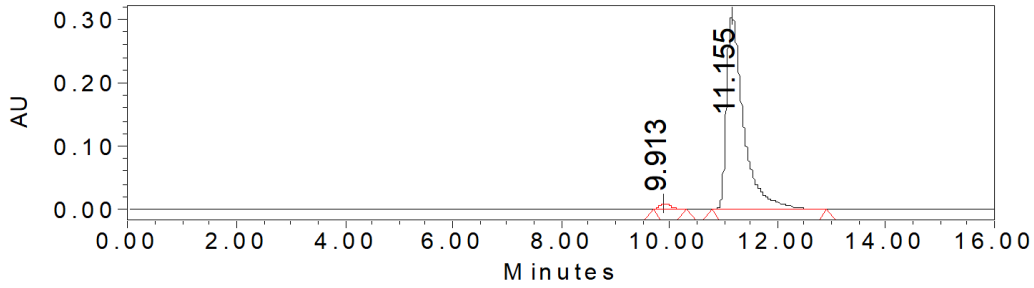
$[\alpha]_D^{21}$ = +488.7 (*c* = 0.99 in $CHCl_3$);

IR (neat): 3464, 3023, 2924, 1737, 1706, 1602, 1490, 1454, 1357, 1287, 1247, 1078, 1040, 969, 749, 721, 691, 558 cm^{-1}.

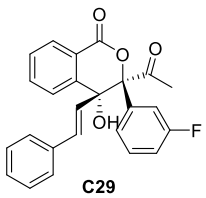
HPLC spectrum of **C28**



	Retention Time	Area	% Area
1	9.886	7221578	50.17
2	11.136	7172165	49.83



	Retention Time	Area	% Area
1	9.913	129424	1.87
2	11.155	6781096	98.13



C29

(3*S*,4*R*)-3-acetyl-3-(3-fluorophenyl)-4-hydroxy-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C29**; 0.1 mmol scale reaction; 35.0 mg, white solid, 87% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 8.00 min, t_2 = 9.23 min];

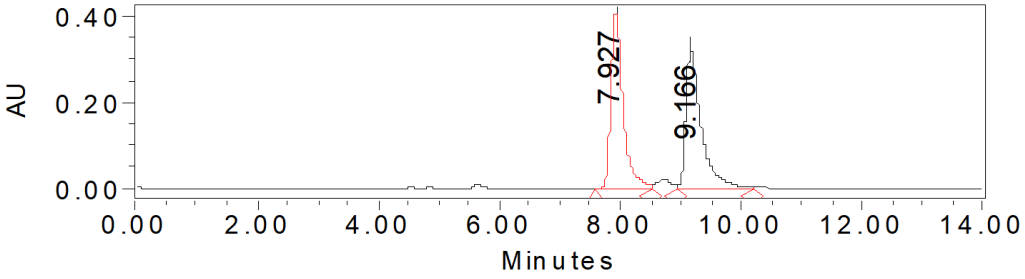
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 – 8.11 (m, 1H), 7.81 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.74 (td, *J* = 7.6, 1.6 Hz, 1H), 7.52 – 7.44 (m, 3H), 7.43 – 7.37 (m, 1H), 7.23 – 7.17 (m, 3H), 7.13 – 7.08 (m, 3H), 6.17 (d, *J* = 15.6 Hz, 1H), 5.98 (dd, *J* = 15.6, 1.2 Hz, 1H), 5.22 (d, *J* = 1.2 Hz, 1H), 2.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.8, 162.6, 162.6 (d, J_{C-F} = 247.4 Hz), 146.1, 135.9, 135.8, 134.0 (d, J_{C-F} = 8.1 Hz), 132.3, 130.3, 129.9 (d, J_{C-F} = 8.1 Hz), 128.7, 128.4, 128.1, 127.2, 126.6, 125.1, 122.3 (d, J_{C-F} = 4.0 Hz), 121.8, 116.3 (d, J_{C-F} = 21.2 Hz), 114.1 (d, J_{C-F} = 25.3 Hz), 89.9, 76.8, 27.4 ppm;

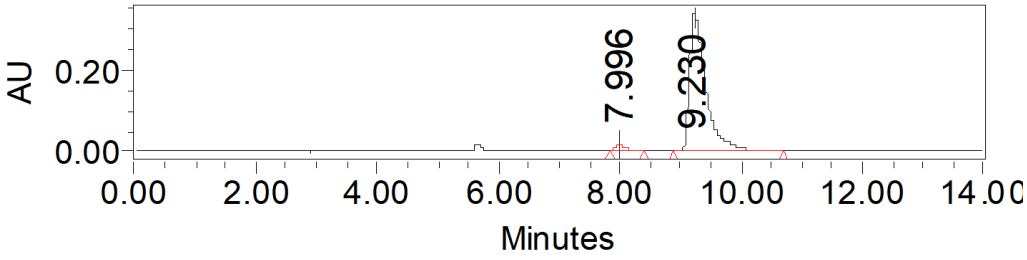
^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -111.59 ppm;

m.p. 64.6 – 67.0 °C;

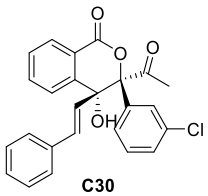
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}FO_4Na$: 425.1159, found: 425.1151;
$[\alpha]_D^{21}$ = +448.5 (*c* = 1.02 in $CHCl_3$);
IR (neat): 3466, 3078, 3026, 1739, 1707, 1589, 1487, 1442, 1358, 1288, 1248, 1210, 1079, 970, 786, 750, 721, 689, 558 cm^{-1}.
HPLC spectrum of **C29**



	Retention Time	Area	% Area
1	7.927	5734876	49.68
2	9.166	5809593	50.32

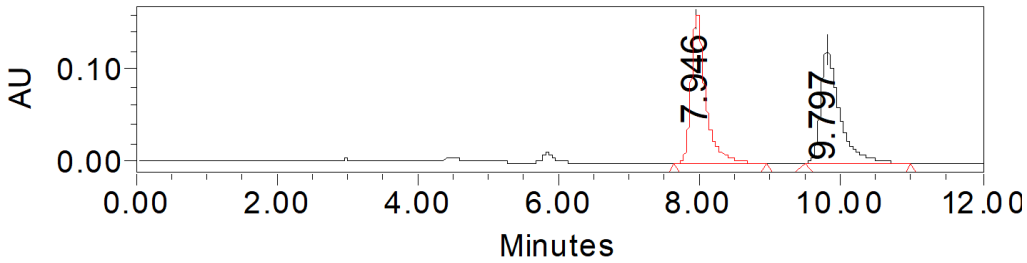


	Retention Time	Area	% Area
1	7.996	190711	2.88
2	9.230	6435069	97.12

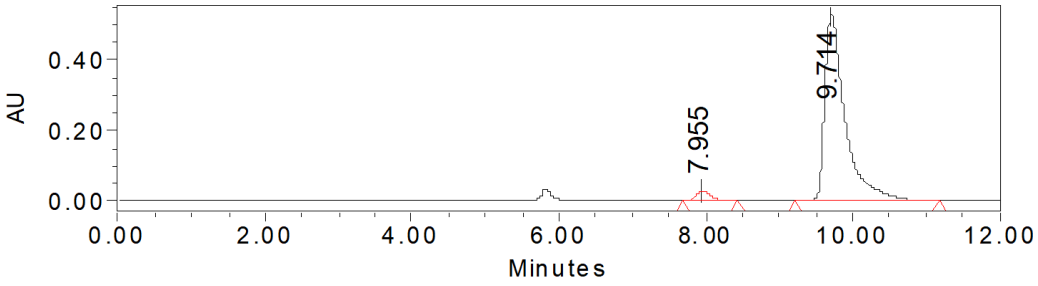


C30

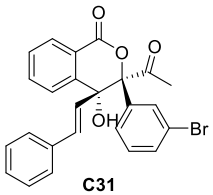
(3*S*,4*R*)-3-acetyl-3-(3-chlorophenyl)-4-hydroxy-4-((*E*)-styryl)isochroman-1-one
The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C30**; 0.1 mmol scale reaction; 33.9 mg, white solid, 81% yield, 93% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 7.95 min, t_2 = 9.71 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 – 8.11 (m, 1H), 7.82 – 7.79 (m, 1H), 7.75 – 7.71 (m, 2H), 7.60 (dt, *J* = 7.2, 1.2 Hz, 1H), 7.50 (td, *J* = 7.2, 1.2 Hz, 1H), 7.39 – 7.34 (m, 2H), 7.23 – 7.17 (m, 3H), 7.11 – 7.09 (m, 2H), 6.17 (d, *J* = 16.0 Hz, 1H), 5.97 (dd, *J* = 16.0, 1.2 Hz, 1H), 5.20 (d, *J* = 1.2 Hz, 1H), 2.14 (s, 3H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.7, 162.6, 146.0, 135.9, 135.8, 134.5, 133.5, 132.4, 130.3, 129.6, 129.4, 128.7, 128.4, 128.1, 127.2, 126.8, 126.7, 125.1, 124.8, 121.8, 89.9, 76.8, 27.5 ppm;
m.p. 60.2 – 64.4 °C;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{34.9689}ClO_4Na$: 441.0864, found: 441.0866;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{36.9659}ClO_4Na$: 443.0835, found: 443.0836;
$[\alpha]_D^{20}$= +462.4 (*c* = 0.73 in $CHCl_3$);
IR (neat): 3067, 2924, 1741, 1707, 1600, 1477, 1454, 1358, 1287, 1247, 1213, 1080, 970, 772, 750, 690, 560 cm^{-1}.
HPLC spectrum of **C30**



	Retention Time	Area	% Area
1	7.946	2348109	49.90
2	9.797	2357440	50.10



	Retention Time	Area	% Area
1	7.955	378559	3.57
2	9.714	10234872	96.43



C31

(3*S*,4*R*)-3-acetyl-3-(3-bromophenyl)-4-hydroxy-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C31**; 0.1 mmol scale reaction; 40.3 mg, white solid, 87% yield, 93% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 8.13 min, t_2 = 10.12 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 7.6 Hz, 1H), 7.89 – 7.88 (m, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.76 – 7.72 (m, 1H), 7.65 – 7.62 (m, 1H), 7.56 – 7.53 (m, 1H), 7.52 – 7.48 (m, 1H), 7.30 (t, *J* = 8.0 Hz, 1H), 7.23 – 7.17 (m, 3H), 7.11 – 7.09 (m, 2H), 6.16 (d, *J* = 16.0 Hz, 1H), 5.95 (d, *J* = 16.0 Hz, 1H), 5.18 (s, 1H), 2.14 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.8, 162.6, 146.0, 135.9, 135.8, 133.6, 132.5, 132.4, 130.3, 129.8, 129.6, 128.7, 128.4, 128.1, 127.2, 126.7, 125.3, 125.1, 122.6, 121.8, 89.8, 76.8, 27.5 ppm;

m.p. 109.5 – 111.7 °C;

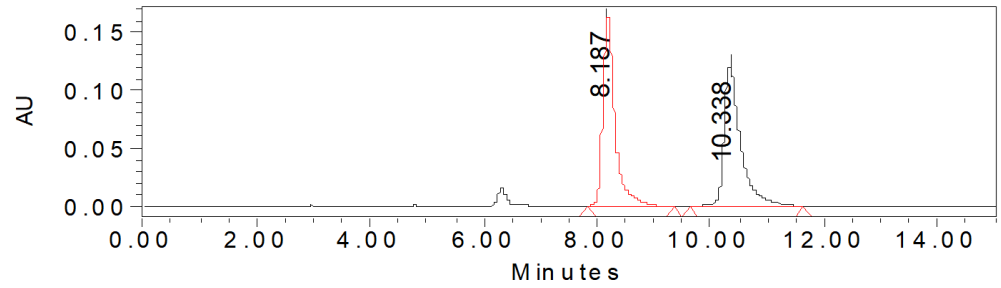
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{78.9183}BrO_4Na$: 485.0359, found: 485.0362;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{80.9163}BrO_4Na$: 487.0338, found: 487.0341;

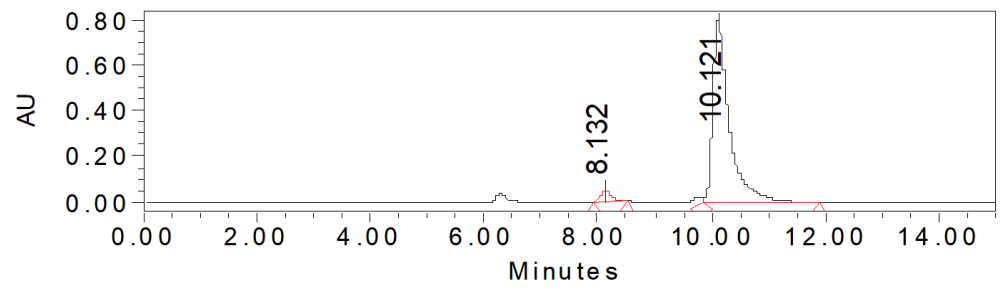
$[\alpha]_D^{20}$ = +394.8 (*c* = 0.53 in $CHCl_3$);

IR (neat): 3464, 3024, 2920, 2361, 1741, 1707, 1600, 1566, 1454, 1357, 1287, 1246, 1210, 1078, 969, 748, 718, 692, 558 cm^{-1}.

HPLC spectrum of **C31**

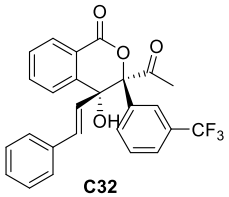


	Retention Time	Area	% Area
1	8.187	2433017	49.61
2	10.338	2471409	50.39



	Retention Time	Area	% Area

1	8.132	576315	3.43
2	10.121	16203249	96.57



C32

(3*S*,4*R*)-3-acetyl-4-hydroxy-4-((*E*)-styryl)-3-(3-(trifluoromethyl)phenyl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C32**; 0.1 mmol scale reaction; 38.4 mg, white solid, 85% yield, 92% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 6.38 min, t_2 = 7.94 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, *J* = 8.0, 1.2 Hz, 1H), 8.00 (s, 1H), 7.89 – 7.86 (m, 1H), 7.81 – 7.79 (m, 1H), 7.75 (td, *J* = 7.2, 1.2 Hz, 1H), 7.69 – 7.67 (m, 1H), 7.58 – 7.49 (m, 2H), 7.22 – 7.14 (m, 3H), 7.08 – 7.05 (m, 2H), 6.14 (d, *J* = 16.0 Hz, 1H), 5.92 (dd, *J* = 15.6, 1.2 Hz, 1H), 5.21 (d, *J* = 1.2 Hz, 1H), 2.16 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.8, 162.6, 145.9, 136.0, 135.7, 132.7, 132.5, 130.9 (q, $J_{C\text{-}F}$ = 32.3 Hz), 130.4, 129.9, 129.9, 128.8, 128.4, 128.1, 127.0, 126.6, 126.5 (d, $J_{C\text{-}F}$ = 273.7 Hz), 126.1 (d, $J_{C\text{-}F}$ = 4.0 Hz), 125.1, 123.7 (d, $J_{C\text{-}F}$ = 4.0 Hz), 121.7, 90.0, 76.8, 27.6 ppm;

^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -62.49 ppm;

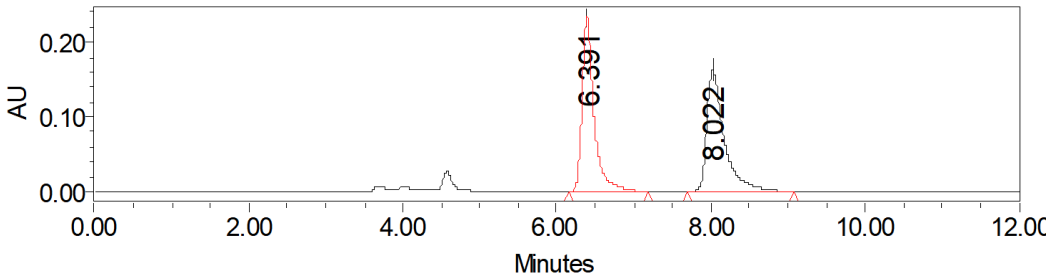
m.p. 50.5 – 52.5 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{26}H_{19}F_3O_4Na$: 475.1128, found: 475.1129;

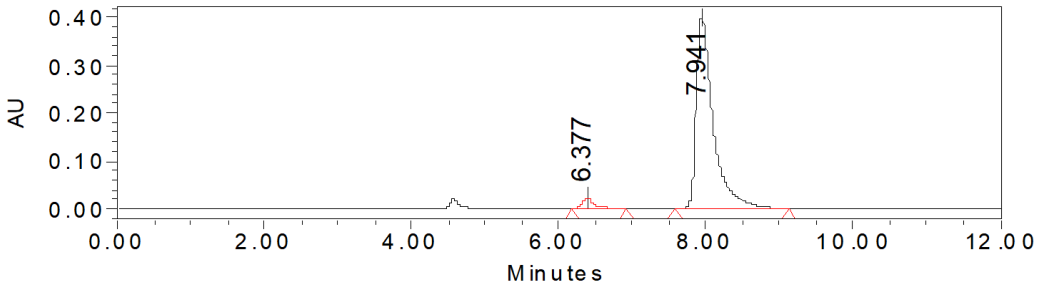
$[\alpha]_D^{21}$ = +410.2 (*c* = 0.54 in $CHCl_3$);

IR (neat): 3468, 2922, 2360, 1743, 1709, 1603, 1450, 1329, 1247, 1168, 1128, 1078, 768, 749, 696, 556 cm^{-1}.

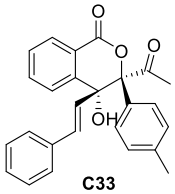
HPLC spectrum of **C32**



	Retention Time	Area	% Area
1	6.391	2560894	49.64
2	8.022	2597578	50.36



	Retention Time	Area	% Area
1	6.377	245154	3.64
2	7.941	6490950	96.36

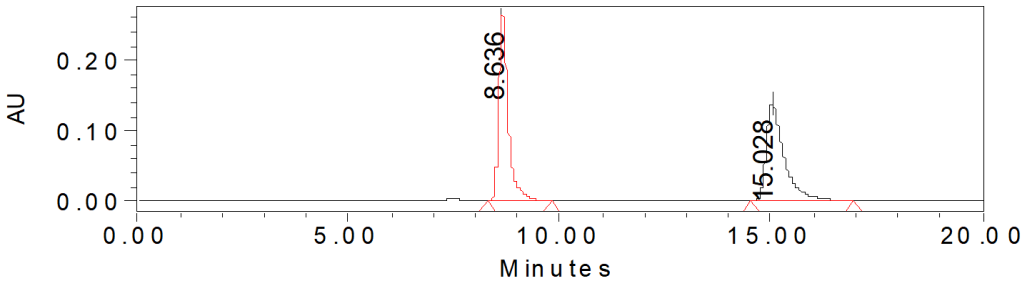


C33

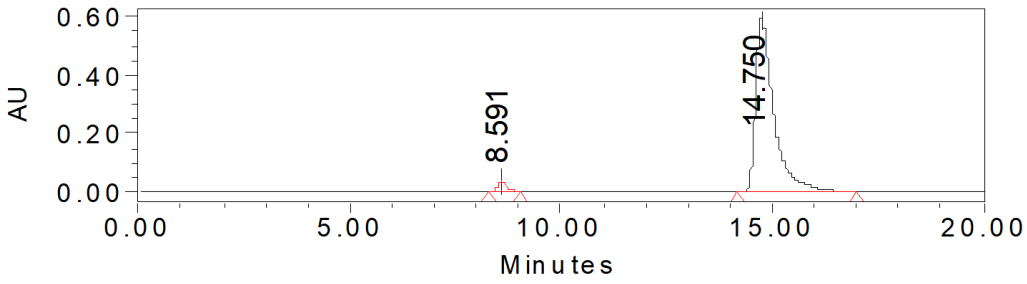
(3*S*,4*R*)-3-acetyl-4-hydroxy-4-((*E*)-styryl)-3-(p-tolyl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C33**; 0.1 mmol scale reaction; 36.6 mg, white solid, 92% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 8.59 min, t_2 = 14.75 min];

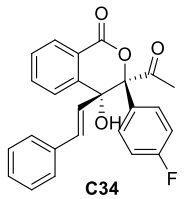
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 7.6 Hz, 1H), 7.82 – 7.80 (m, 1H), 7.72 (td, *J* = 7.6, 1.2 Hz, 1H), 7.56 (d, *J* = 8.4 Hz, 2H), 7.48 (td, *J* = 7.6, 1.2 Hz, 1H), 7.24 – 7.16 (m, 5H), 7.12 – 7.09 (m, 2H), 6.12 (d, *J* = 16.0 Hz, 1H), 6.01 (d, *J* = 16.0 Hz, 1H), 5.24 (s, 1H), 2.37 (s, 3H), 2.12 (s, 3H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.2, 163.2, 146.4, 139.1, 136.0, 135.7, 132.0, 130.2, 129.0, 128.5, 128.4, 127.9, 127.9, 127.8, 126.7, 126.5, 125.1, 122.1, 90.7, 76.8, 27.3, 21.1 ppm;
m.p. 62.8 – 67.0 °C;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{26}H_{22}O_4Na$: 421.1410, found: 421.1413;
$[\alpha]_D^{20}$ = +509.1 (*c* = 0.58 in $CHCl_3$);
IR (neat): 3463, 3029, 2920, 2361, 1740, 1706, 1603, 1453, 1358, 1289, 1248, 1081, 969, 769, 725, 693, 559 cm^{-1}.
HPLC spectrum of **C33**



	Retention Time	Area	% Area
1	8.636	4200436	50.14
2	15.028	4176196	49.86

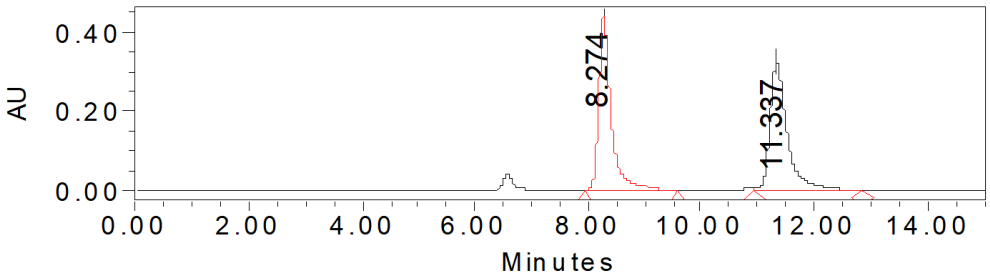


	Retention Time	Area	% Area
1	8.591	540084	2.92
2	14.750	17960208	97.08

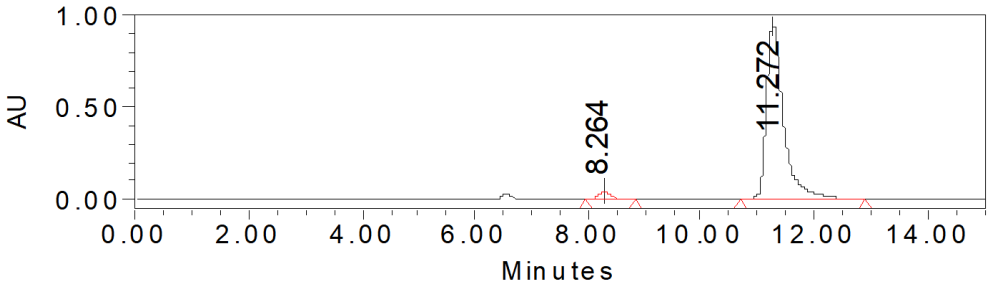


C34

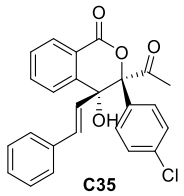
(3*S*,4*R*)-3-acetyl-3-(4-fluorophenyl)-4-hydroxy-4-((*E*)-styryl)isochroman-1-one
The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C34**; 0.1 mmol scale reaction; 37.4 mg, white solid, 93% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 8.26 min, t_2 = 11.27 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.80 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.73 (td, *J* = 7.6, 1.6 Hz, 1H), 7.70 – 7.66 (m, 2H), 7.50 (td, *J* = 7.6, 1.6 Hz, 1H), 7.23 – 7.16 (m, 3H), 7.15 – 7.08 (m, 4H), 6.13 (d, *J* = 16.0 Hz, 1H), 5.96 (dd, *J* = 16.0, 1.2 Hz, 1H), 5.22 (d, *J* = 1.2 Hz, 1H), 2.13 (s, 3H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.1, 163.2 (d, $J_{C\text{-}F}$ = 250.5 Hz), 162.8, 146.1, 135.9, 135.8, 132.3, 130.3, 128.7, 128.6 (d, $J_{C\text{-}F}$ = 12.1 Hz), 128.4, 128.1, 127.3, 127.2 (d, $J_{C\text{-}F}$ = 3.0 Hz), 126.6, 125.1, 121.9, 115.4 (d, $J_{C\text{-}F}$ = 21.2 Hz), 90.2, 76.8, 27.4 ppm;
^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -112.16 ppm;
m.p. 63.3– 64.7 °C;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}FO_4Na$: 425.1160, found: 425.1165;
$[\alpha]_D^{21}$ = +444.7 (*c* = 0.60 in $CHCl_3$);
IR (neat) 3466, 3027, 2341, 1740, 1706, 1603, 1509, 1358, 1288, 1243, 1164, 1080, 970, 840, 727, 693, 559 cm^{-1}.
HPLC spectrum of **C34**



	Retention Time	Area	% Area
1	8.274	6837263	50.14
2	11.337	6799990	49.86



	Retention Time	Area	% Area
1	8.264	606208	2.94
2	11.272	19991680	97.06



C35

(3*S*,4*R*)-3-acetyl-3-(4-chlorophenyl)-4-hydroxy-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C35**; 0.1 mmol scale reaction; 37.3 mg, white solid, 89% yield, 92% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 7.81 min, t_2 = 13.00 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 7.6 Hz, 1H), 7.80 (d, *J* = 7.6 Hz, 1H), 7.73 (td, *J* = 7.6, 1.2 Hz, 1H), 7.66 – 7.62 (m, 2H), 7.50 (td, *J* = 7.6, 1.2 Hz, 1H), 7.42 – 7.39 (m, 2H), 7.23 – 7.16 (m, 3H), 7.11 – 7.09 (m, 2H), 6.14 (d, *J* = 16.0 Hz, 1H), 5.96 (d, *J* = 16.0 Hz, 1H), 5.20 (s, 1H), 2.13 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.0, 162.7, 146.1, 135.9, 135.7, 135.5, 132.4, 130.3, 130.0, 128.7, 128.6, 128.5, 128.1, 128.0, 127.2, 126.7, 125.1, 121.8, 90.1, 76.8, 27.4 ppm;

m.p. 71.9 – 74.7 °C;

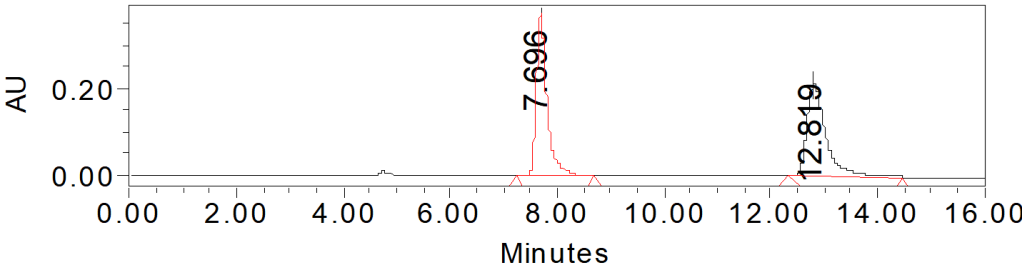
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}^{34.9689}ClO_4Na$: 441.0864, found: 441.0862;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}^{36.9659}ClO_4Na$: 443.0835, found: 443.0833;

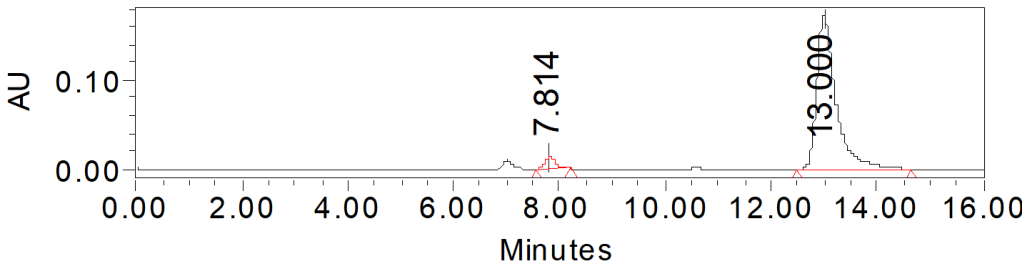
$[\alpha]_D^{20}$ = +411.7 (*c* = 1.12 in $CHCl_3$);

IR (neat): 3466, 3026, 1739, 1706, 1600, 1492, 1454, 1357, 1287, 1247, 1210, 1080, 1013, 969, 804, 750, 693, 591, 557 cm^{-1}.

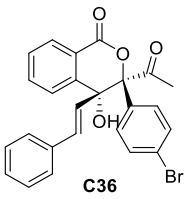
HPLC spectrum of **C35**



	Retention Time	Area	% Area
1	7.696	5295053	50.06
2	12.819	5281477	49.94



	Retention Time	Area	% Area
1	7.814	173841	3.82
2	13.000	4377555	96.18



C36

(3*S*,4*R*)-3-acetyl-3-(4-bromophenyl)-4-hydroxy-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1/1) to afford the desired product **C36**; 0.1 mmol scale reaction; 42.5 mg, white solid, 92% yield, 95% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 8.64 min, t_2 = 14.68 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 7.6, 0.8 Hz, 1H), 7.81 – 7.79 (m, 1H), 7.73 (td, *J* = 7.6, 1.2 Hz, 1H), 7.60 – 7.54 (m, 4H), 7.51 – 7.47 (m, 1H), 7.23 – 7.15 (m, 3H), 7.11 – 7.08 (m, 2H), 6.15 (d, *J* = 16.0 Hz, 1H), 5.97 (dd, *J* = 16.0, 1.2 Hz, 1H), 5.19 (d, *J* = 1.2 Hz, 1H), 2.13 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.8, 162.7, 146.0, 135.9, 135.7, 132.4, 131.5, 130.5, 130.3, 128.7, 128.4, 128.3, 128.1, 127.2, 126.6, 125.0, 123.8, 121.8, 90.1, 76.8, 27.4 ppm;

m.p. 75.0 – 76.2 °C;

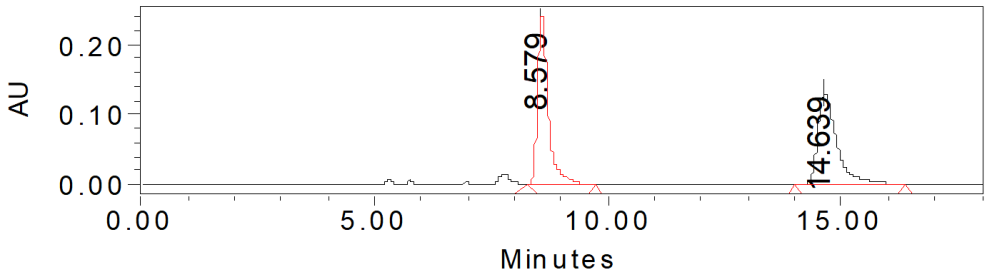
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{78.9183}BrO_4Na$: 485.0359, found: 485.0367;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{19}{}^{80.9163}BrO_4Na$: 487.0338, found: 487.0348;

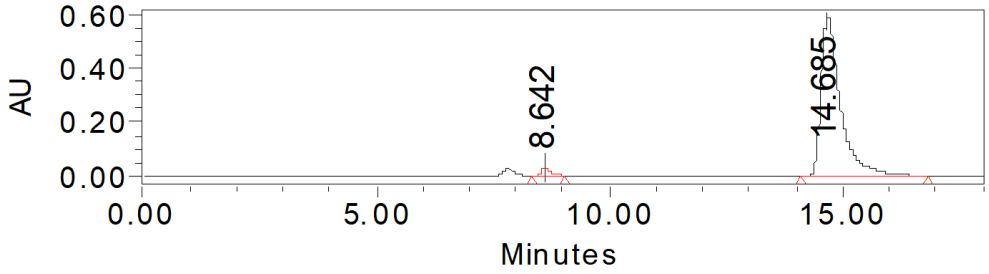
$[\alpha]_D^{21}$ = +458.8 (*c* = 1.12 in $CHCl_3$);

IR (neat): 3465, 3026, 2360, 1739, 1706, 1602, 1489, 1454, 1358, 1287, 1247, 1211, 1076, 1009, 969, 751, 694, 591, 556 cm^{-1}.

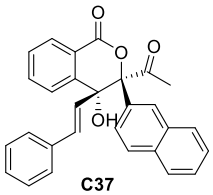
HPLC spectrum of **C36**



	Retention Time	Area	% Area
1	8.579	3803496	50.29
2	14.639	3760034	49.71



	Retention Time	Area	% Area
1	8.642	410260	2.34
2	14.685	17143417	97.66



C37

(3*S*,4*R*)-3-acetyl-4-hydroxy-3-(naphthalen-2-yl)-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1.5/1) to afford the desired product **C37**; 0.1 mmol scale reaction; 38.6 mg, white solid, 89% yield, 94% ee; determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 5.74 min, t_2 = 6.59 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 2.0 Hz, 1H), 8.16 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.93 – 7.90 (m, 2H), 7.88 – 7.81 (m, 3H), 7.76 (td, *J* = 7.6, 1.2 Hz, 1H), 7.57 – 7.50 (m, 3H), 7.17 – 7.10 (m, 3H), 7.05 – 7.03 (m, 2H), 6.11 (d, *J* = 16.0 Hz, 1H), 6.03 (d, *J* = 16.0 Hz, 1H), 5.35 (s, 1H), 2.16 (s, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.1, 163.1, 146.3, 135.9, 135.8, 133.3, 132.7, 132.3, 130.3, 128.8, 128.7, 128.3, 128.1, 127.9, 127.8, 127.5, 127.0, 126.6, 126.6, 126.5, 125.2, 123.7, 122.1, 90.7, 77.1, 27.5 ppm;

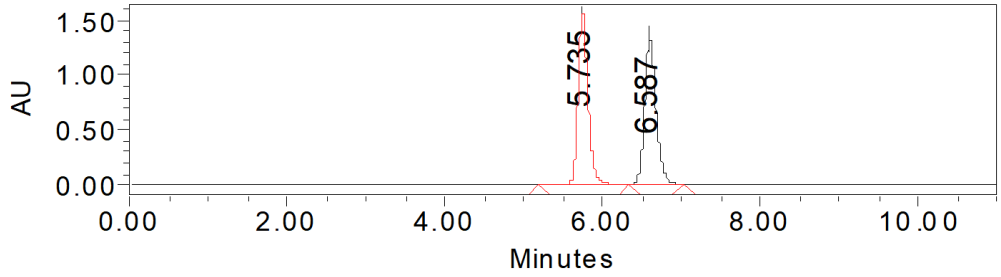
m.p. 85.9 – 89.9 °C;

HRMS (ESI) m/z [M+Na]$^+$ calcd for $C_{29}H_{22}O_4Na$: 457.1410, found: 457.1406;

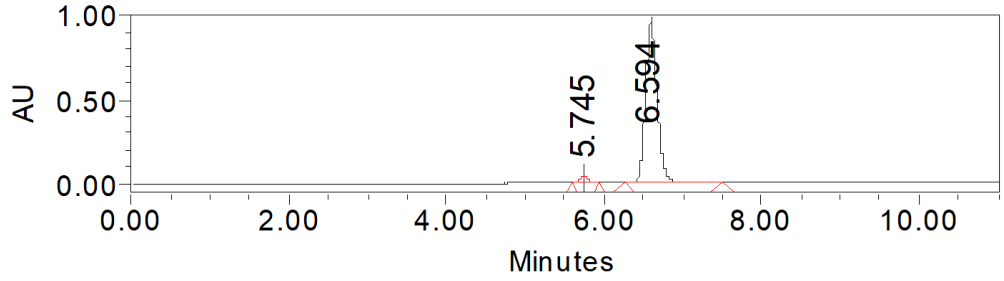
[α]$_D^{21}$ = +510.6 (*c* = 0.66 in $CHCl_3$);

IR (neat): 3464, 3060, 3024, 2361, 1738, 1705, 1601, 1454, 1357, 1285, 1247, 1217, 1079, 969, 814, 768, 748, 693, 558, 477 cm^{-1}.

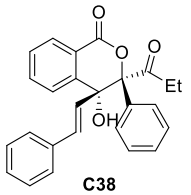
HPLC spectrum of **C37**



	Retention Time	Area	% Area
1	5.735	14129712	50.29
2	6.587	13967910	49.71



	Retention Time	Area	% Area
1	5.745	289571	2.83
2	6.594	9956580	97.17



C38

(3*S*,4*R*)-4-hydroxy-3-phenyl-3-propionyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1.5/1) to afford the desired product **C38**; 0.1 mmol scale reaction; 37.4 mg, colorless oil, 94% yield, 89% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 8.56 min, t_2 = 10.11 min];

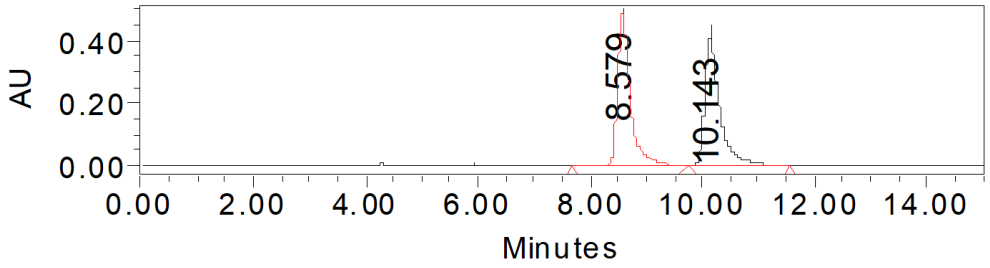
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 7.6 Hz, 1H), 7.82 (d, *J* = 7.6 Hz, 1H), 7.73 (t, *J* = 7.6 Hz, 1H), 7.66 – 7.64 (m, 2H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.44 – 7.37 (m, 3H), 7.21 – 7.13 (m, 3H), 7.09 – 7.07 (m, 2H), 6.09 (d, *J* = 16.0 Hz, 1H), 5.98 (d, *J* = 16.0 Hz, 1H), 5.38 (s, 1H), 2.64 – 2.54 (m, 1H), 2.46 – 2.36 (m, 1H), 0.85 (t, *J* = 7.2 Hz, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 214.1, 163.2, 146.3, 136.0, 135.7, 132.1, 131.7, 130.2, 129.1, 128.6, 128.4, 128.3, 127.9, 127.8, 126.6, 126.5, 125.2, 122.1, 90.7, 76.8, 32.9, 7.6 ppm;

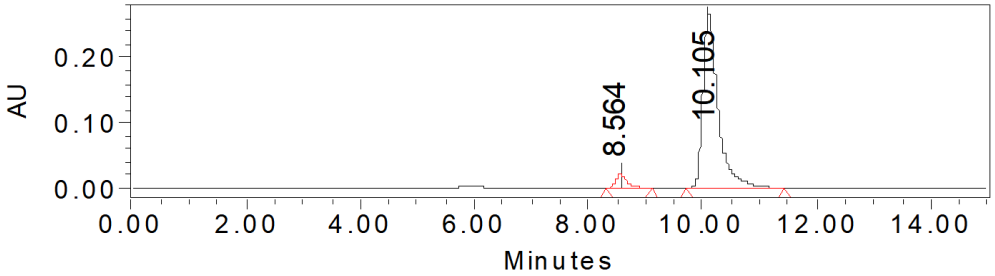
HRMS (ESI) m/z [M+Na]$^+$ calcd for $C_{26}H_{22}O_4Na$: 421.1410, found: 421.1416;

[α]$_D^{21}$ = +474.4 (*c* = 0.36 in $CHCl_3$);

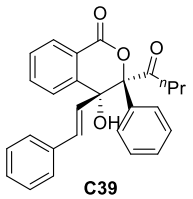
IR (neat): 3463, 2925, 1739, 1705, 1602, 1452, 1287, 1248, 1081, 1044, 969, 750, 707, 694 cm^{-1}.
HPLC spectrum of **C38**



	Retention Time	Area	% Area
1	8.579	7730654	50.05
2	10.143	7716495	49.95

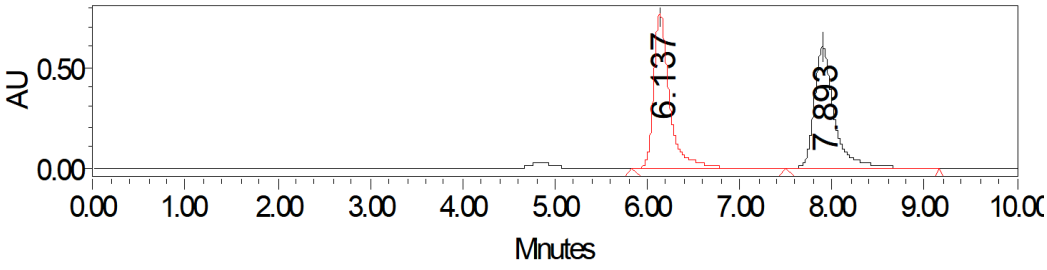


	Retention Time	Area	% Area
1	8.564	286227	5.40
2	10.105	5012049	94.60

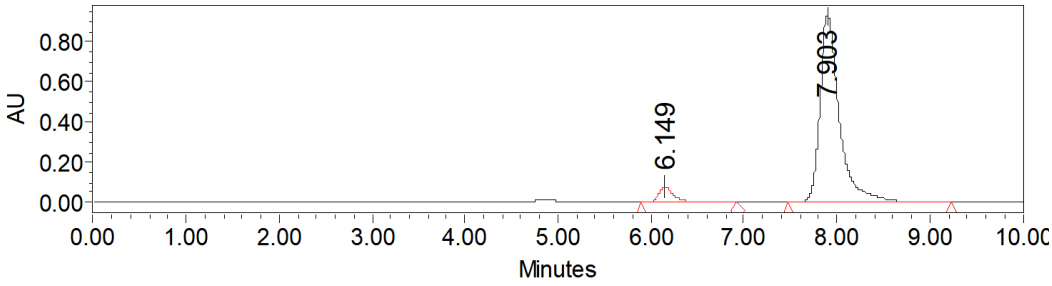


C39

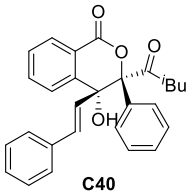
(3*S*,4*R*)-3-butyryl-4-hydroxy-3-phenyl-4-((*E*)-styryl)isochroman-1-one
The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1.5/1) to afford the desired product **C39**; 0.1 mmol scale reaction; 37.9 mg, colorless oil, 92% yield, 87% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 6.15 min, t_2 = 7.90 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.82 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.75 – 7.70 (m, 1H), 7.68 – 7.65 (m, 2H), 7.49 (td, *J* = 7.6, 1.2 Hz, 1H), 7.45 – 7.37 (m, 3H), 7.21 – 7.13 (m, 3H), 7.10 – 7.06 (m, 2H), 6.10 (d, *J* = 16.0 Hz, 1H), 5.99 (d, *J* = 15.6 Hz, 1H), 5.39 (s, 1H), 2.56 – 2.47 (m, 1H), 2.43 – 2.35 (m, 1H), 1.45 – 1.31 (m, 2H), 0.65 (t, *J* = 7.2 Hz, 3H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 213.4, 163.2, 146.4, 136.0, 135.8, 132.2, 131.5, 130.2, 129.1, 128.6, 128.4, 128.3, 128.0, 127.9, 126.7, 126.6, 125.2, 122.2, 90.6, 76.9, 41.2, 16.7, 13.2 ppm;
HRMS (ESI) m/z $[M+Na]^+$ calcd for $C_{27}H_{24}O_4Na$: 435.1567, found: 435.1563;
$[\alpha]_D^{20}$ = +414.1 (*c* = 1.34 in $CHCl_3$);
IR (neat): 3463, 2966, 1738, 1703, 1603, 1495, 1452, 1362, 1286, 1247, 1081, 1049, 969, 749, 695, 560 cm^{-1}.
HPLC spectrum of **C39**



	Retention Time	Area	% Area
1	6.137	9112916	50.10
2	7.893	9075824	49.90



	Retention Time	Area	% Area
1	6.149	953327	6.35
2	7.903	14051612	93.65



C40

(3*S*,4*R*)-4-hydroxy-3-(3-methylbutanoyl)-3-phenyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1.5/1) to afford the desired product **C40**; 0.1 mmol scale reaction; 42.2 mg, colorless oil, 99% yield, 82% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 6.60 min, t_2 = 8.98 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.83 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.75 – 7.67 (m, 3H), 7.49 (td, *J* = 7.6, 1.2 Hz, 1H), 7.45 – 7.38 (m, 3H), 7.21 – 7.13 (m, 3H), 7.09 – 7.06 (m, 2H), 6.11 (d, *J* = 15.6 Hz, 1H), 5.99 (d, *J* = 16.0 Hz, 1H), 5.41 (s, 1H), 2.42 – 2.30 (m, 2H), 2.01 – 1.91 (m, 1H), 0.66 (d, *J* = 6.8 Hz, 3H), 0.61 (d, *J* = 6.4 Hz, 3H);

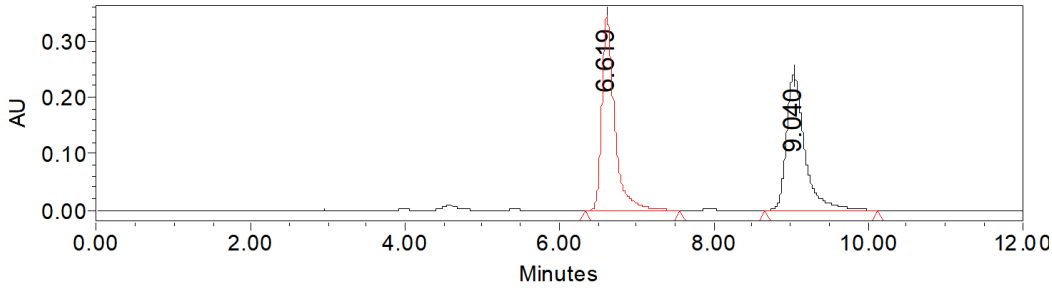
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 212.8, 163.1, 146.3, 135.9, 135.7, 132.1, 131.3, 130.1, 129.1, 128.5, 128.4, 128.3, 128.2, 127.9, 127.8, 126.6, 125.1, 122.2, 90.4, 76.8, 47.8, 23.4, 22.2, 21.6 ppm;

HRMS (ESI) m/z $[M+Na]^+$ calcd for $C_{28}H_{26}O_4Na$: 449.1723, found: 449.1724;

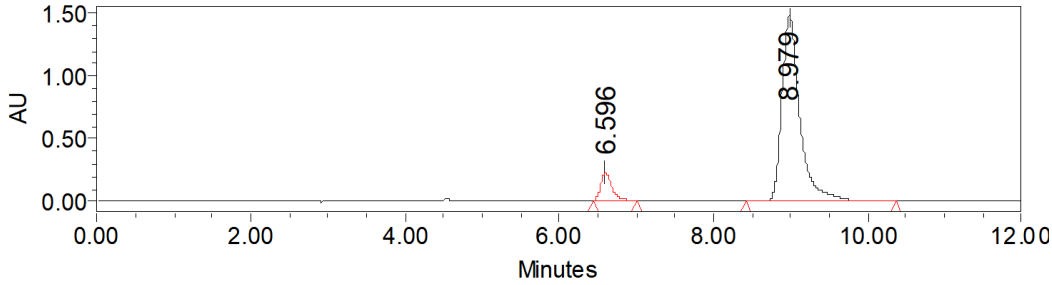
$[\alpha]_D^{21}$ = +352.3 (*c* = 1.10 in $CHCl_3$);

IR (neat): 3464, 3062, 2960, 2360, 1739, 1702, 1602, 1495, 1452, 1364, 1287, 1247, 1159, 1081, 1050, 1035, 969, 748, 700, 560 cm^{-1}.

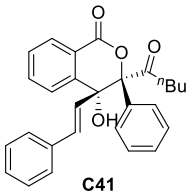
HPLC spectrum of **C40**



	Retention Time	Area	% Area
1	6.619	3892206	50.08
2	9.040	3879362	49.92



	Retention Time	Area	% Area
1	6.596	2397820	9.00
2	8.979	24256870	91.00



C41

(3*S*,4*R*)-4-hydroxy-3-pentanoyl-3-phenyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1.5/1) to afford the desired product **C41**; 0.1 mmol scale reaction; 42.2 mg, colorless oil, 99% yield, 85% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 7.01 min, t_2 = 8.94 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.84 – 7.82 (m, 1H), 7.73 (td, *J* = 7.6, 1.2 Hz, 1H), 7.69 – 7.66 (m, 2H), 7.49 (td, *J* = 7.6, 1.2 Hz, 1H), 7.45 – 7.39 (m, 3H), 7.21 – 7.13 (m, 3H), 7.10 – 7.06 (m, 2H), 6.11 (d, *J* = 16.0 Hz, 1H), 6.00 (d, *J* = 15.6 Hz, 1H), 5.40 (s, 1H), 2.59 – 2.51 (m, 1H), 2.44 – 2.36 (m, 1H), 1.45 – 1.24 (m, 2H), 1.12 – 0.89 (m, 3H), 0.71 (t, *J* = 7.6 Hz, 3H);

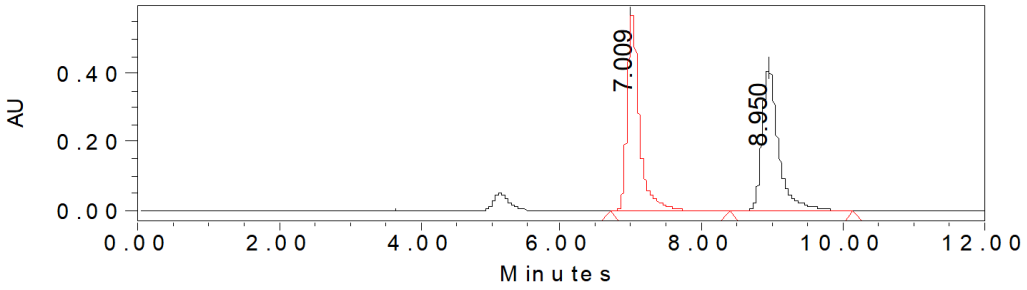
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 213.5, 163.1, 146.3, 136.0, 135.7, 132.1, 131.5, 130.1, 129.1, 128.5, 128.3, 128.2, 127.9, 127.8, 126.6, 126.5, 125.1, 122.1, 90.6, 76.8, 39.0, 25.2, 21.6, 13.5 ppm;

HRMS (ESI) m/z [M+Na]$^+$ calcd for $C_{28}H_{26}O_4Na$: 449.1723, found: 449.1720;

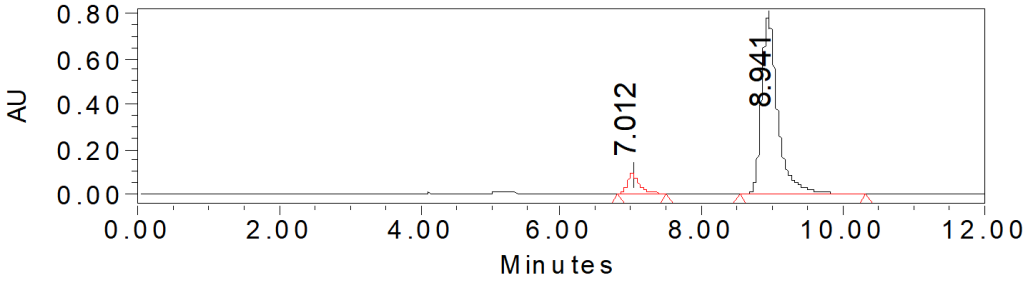
[α]$_D^{20}$ = +405.7 (*c* = 1.36 in $CHCl_3$);

IR (neat): 3463, 3062, 2959, 2960, 2932, 1738, 1703, 1602, 1495, 1451, 1373, 1286, 1247, 1081, 1050, 969, 770, 749, 694, 560 cm^{-1}.

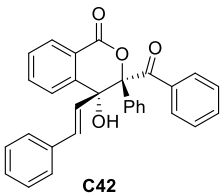
HPLC spectrum of **C41**



	Retention Time	Area	% Area
1	7.009	6787094	50.12
2	8.950	6754122	49.88



	Retention Time	Area	% Area
1	7.012	1039951	7.57
2	8.941	12702235	92.43



C42

(3*S*,4*R*)-3-benzoyl-4-hydroxy-3-phenyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1.5/1) to afford the desired product **C42**; 0.1 mmol scale reaction; 44.2 mg, white solid, 99% yield, 87% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 85/15, 1.0 mL/min, λ = 254 nm, t_1 = 7.30 min, t_2 = 11.52 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.84 (m, 2H), 7.73 – 7.66 (m, 5H), 7.46 – 7.37 (m, 5H), 7.28 – 7.16 (m, 5H), 7.15 – 7.11 (m, 2H), 6.16 – 6.08 (m, 2H), 5.53 (s, 1H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 202.2, 162.9, 146.6, 136.1, 135.5, 134.0, 134.0, 132.7, 132.4, 130.7, 130.0, 129.1, 128.4, 128.3, 128.3, 128.3, 127.9, 127.7, 126.6, 126.4, 125.0, 122.3, 90.8, 77.6 ppm;

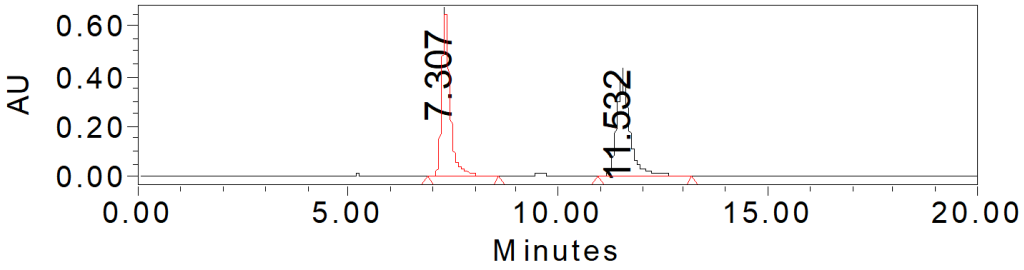
m.p. 87.2 – 88.9 °C;

HRMS (ESI) m/z [M+Na]$^+$ calcd for $C_{30}H_{22}O_4Na$: 469.1410, found: 469.1413;

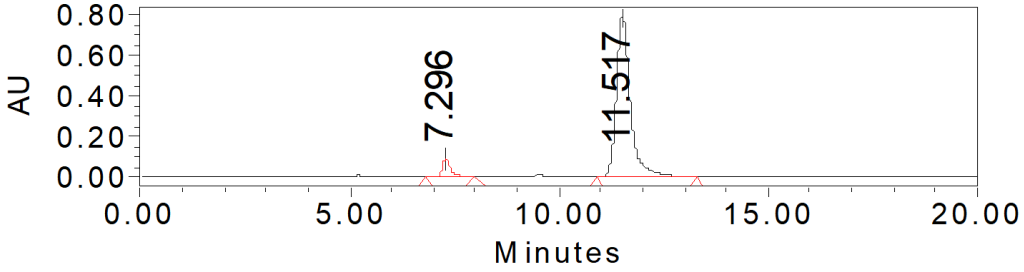
[α]$_D^{21}$ = +504.2 (*c* = 0.81 in $CHCl_3$);

IR (neat): 3465, 3062, 3027, 2361, 1741, 1665, 1599, 1450, 1370, 1286, 1245, 1076, 772, 709, 691 cm^{-1}.

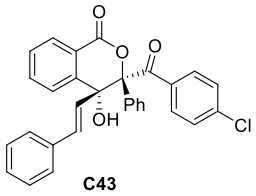
HPLC spectrum of **C42**



	Retention Time	Area	% Area
1	7.307	8882979	50.13
2	11.532	8836635	49.87



	Retention Time	Area	% Area
1	7.296	1294355	6.53
2	11.517	18539573	93.47



C43

(3*S*,4*R*)-3-(4-chlorobenzoyl)-4-hydroxy-3-phenyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1.5/1) to afford the desired product **C43**; 0.1 mmol scale reaction; 47.6 mg, white solid, 99% yield, 72% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 85/15, 1.0 mL/min, λ = 254 nm, t_1 = 7.75 min, t_2 = 16.88 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.6 Hz, 1H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.73 – 7.65 (m, 5H), 7.44 – 7.37 (m, 4H), 7.24 – 7.11 (m, 7H), 6.15 – 6.06 (m, 2H), 5.40 (s, 1H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 200.6, 162.7, 146.6, 140.7, 136.0, 135.6, 132.5, 132.5, 132.2, 132.1, 130.1, 129.2, 128.8, 128.4, 128.4, 128.0, 127.9, 127.6, 126.6, 126.4, 125.0, 122.2, 90.8, 77.5 ppm;

m.p. 200.8 – 205.6 °C;

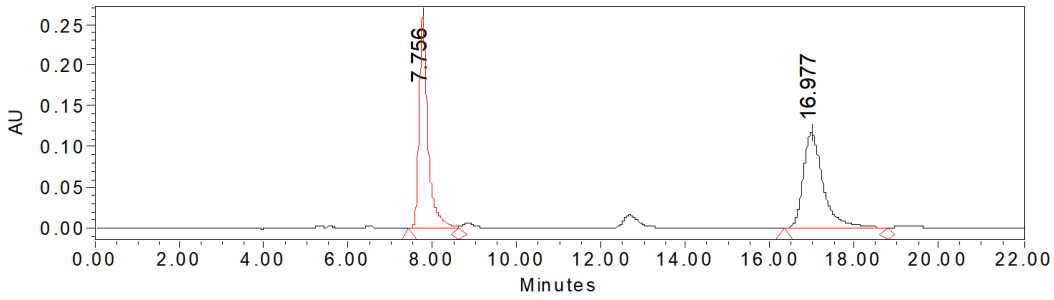
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{30}H_{21}{}^{34.9689}ClO_4Na$: 503.1021, found: 503.1020;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{30}H_{21}{}^{36.9659}ClO_4Na$: 505.0991, found: 505.0995;

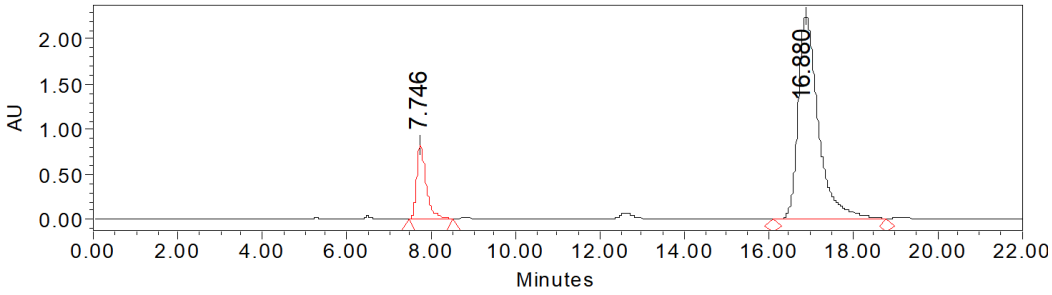
$[\alpha]_D^{20}$ = +385.4 (*c* = 1.57 in $CHCl_3$);

IR (neat): 3469, 3063, 3027, 2361, 1740, 1667, 1585, 1492, 1451, 1366, 1285, 1244, 1077, 844, 752, 669, 587 cm^{-1}.

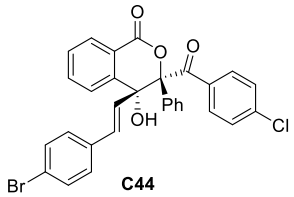
HPLC spectrum of **C43**



	Retention Time	Area	% Area
1	7.756	3805376	50.16
2	16.977	3781716	49.84



	Retention Time	Area	% Area
1	7.746	12319879	13.72
2	16.880	77448401	86.28



C44

(3*S*,4*R*)-4-((*E*)-4-bromostyryl)-3-(4-chlorobenzoyl)-4-hydroxy-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/EtOAc = 15/1) to afford the desired product **C44**; 0.1 mmol scale reaction; 42.0 mg, white solid, 75% yield, 84% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t_1 = 7.13 min, t_2 = 14.83 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.6 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.73 (t, *J* = 7.6 Hz, 1H), 7.67 – 7.65 (m, 4H), 7.45 – 7.41 (m, 4H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.26 – 7.24 (m, 2H), 6.98 (d, *J* = 8.4 Hz, 2H), 6.11 – 6.03 (m, 2H), 5.41 (s, 1H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 200.5, 162.7, 146.4, 140.8, 135.7, 134.9, 132.4, 132.2, 132.0, 131.6, 131.3, 130.2, 129.3, 128.8, 128.5, 128.4, 128.3, 128.2, 126.3, 125.0, 122.1, 121.9, 90.7, 77.4 ppm;

m.p. 100.8 – 105.4 °C;

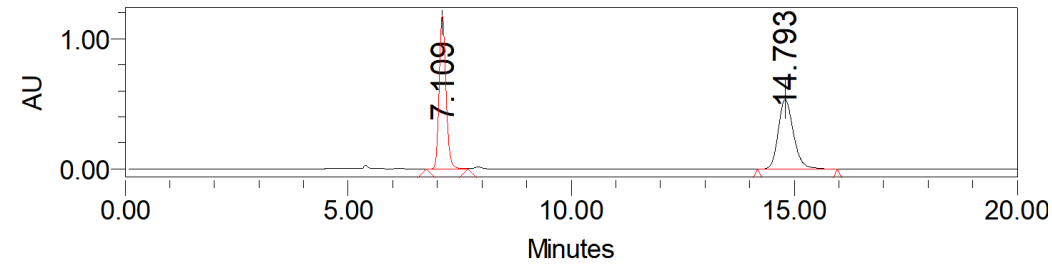
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{30}H_{20}{}^{78.9183}Br{}^{34.9689}ClO_4Na$: 581.0126, found: 581.0135;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{30}H_{20}{}^{80.9163}Br{}^{36.9659}ClO_4Na$: 583.0105, found: 583.0115;

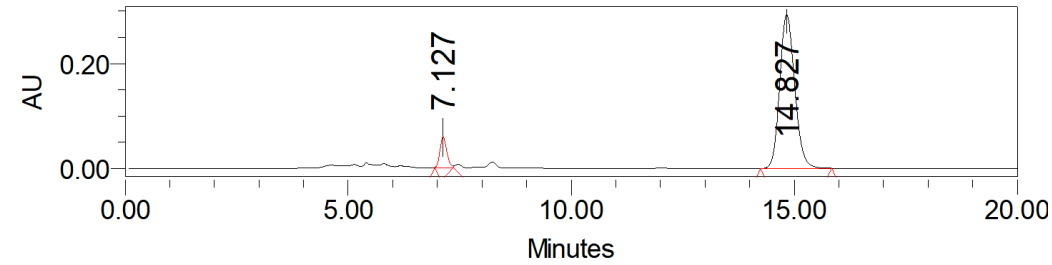
$[\alpha]_D^{20}$ = +349.7 (*c* = 0.93 in $CHCl_3$);

IR (neat): 3468, 3066, 3029, 2926, 1739, 1666, 1586, 1487, 1451, 1284, 1244, 1076, 1011, 828, 769, 757, 590 cm^{-1}.

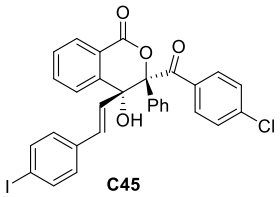
HPLC spectrum of **C44**



	Retention Time	Area	% Area
1	7.109	12432245	49.63
2	14.793	12617477	50.37



	Retention Time	Area	% Area
1	7.127	613886	8.22
2	14.827	6854498	91.78



C45

(3*S*,4*R*)-3-(4-chlorobenzoyl)-4-hydroxy-4-((*E*)-4-iodostyryl)-3-phenylisochroman-1-one

The residue was purified by column chromatography on silica gel (PE/EtOAc = 15/1) to afford the desired product **C45**; 0.1 mmol scale reaction; 37.6 mg, white solid, 62% yield, 86% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t_1 = 7.47 min, t_2 = 15.86 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.0 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.73 (t, *J* = 7.6 Hz, 1H), 7.67 – 7.64 (m, 4H), 7.54 (d, *J* = 8.0 Hz, 2H), 7.45 – 7.41 (m, 4H), 7.26 – 7.24 (m, 2H), 6.85 (d, *J* = 8.0 Hz, 2H), 6.12 – 6.02 (m, 2H), 5.41 (s, 1H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 200.5, 162.7, 146.3, 140.9, 137.5, 135.8, 135.5, 132.4, 132.2, 132.0, 131.4, 130.3, 129.3, 128.8, 128.5, 128.5, 128.4, 126.3, 125.0, 122.1, 93.5, 90.7, 77.4 ppm;

m.p. 101.6 – 106.5 °C;

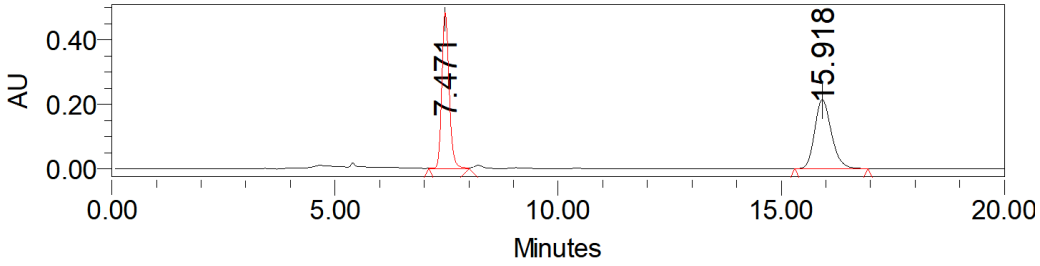
HRMS (ESI) *m/z* $[M+K]^+$ calcd for $C_{30}H_{20}{}^{34.9689}ClIO_4K$: 644.9726, found: 644.9726;

HRMS (ESI) *m/z* $[M+K]^+$ calcd for $C_{30}H_{20}{}^{36.9659}ClIO_4K$: 646.9697, found: 646.9703;

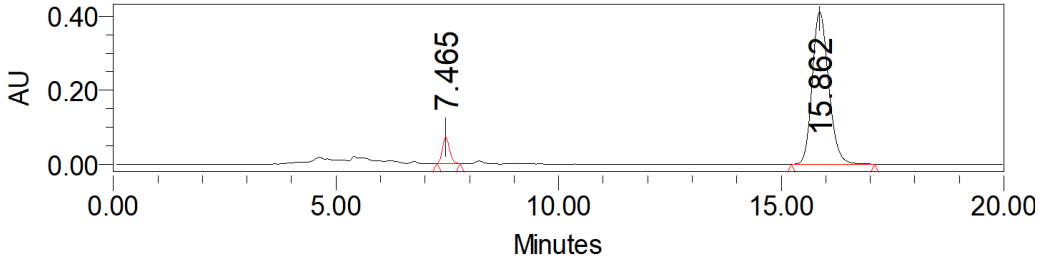
$[\alpha]_D^{20}$ = +280.8 (*c* = 0.95 in $CHCl_3$);

IR (neat): 3467, 3064, 3031, 2925, 1739, 1666, 1586, 1487, 1451, 1283, 1246, 1220, 1089, 973, 837, 771, 534 cm^{-1}.

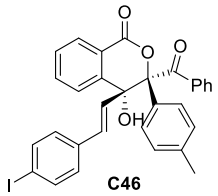
HPLC spectrum of **C45**



	Retention Time	Area	% Area
1	7.471	5495027	49.68
2	15.918	5566895	50.32



	Retention Time	Area	% Area
1	7.465	784920	6.94
2	15.862	10528162	93.06



C46

(3*S*,4*R*)-3-benzoyl-4-hydroxy-4-((*E*)-4-iodostyryl)-3-(p-tolyl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/EtOAc = 15/1) to afford the desired product **C46**; 0.1 mmol scale reaction; 37.5 mg, white solid, 64% yield, 93% ee; determined by HPLC analysis [Daicel chiralpak IC, *n*-hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t_1 = 8.84 min, t_2 = 9.90 min];

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 – 7.83 (m, 2H), 7.72 (td, J = 7.2, 1.2 Hz, 1H), 7.67 – 7.66 (m, 2H), 7.57 – 7.54 (m, 4H), 7.47 – 7.39 (m, 2H), 7.29 – 7.26 (m, 2H), 7.23 (d, J = 7.8 Hz, 2H), 6.89 – 6.88 (m, 2H), 6.14 (d, J = 15.6 Hz, 1H), 6.07 (d, J = 15.6 Hz, 1H), 5.52 (s, 1H), 2.36 (s, 3H);

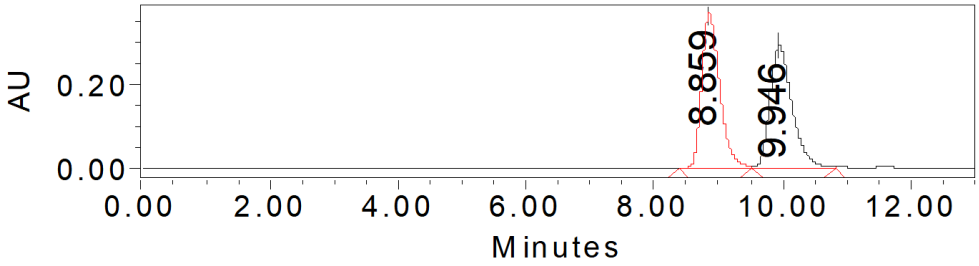
^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 202.3, 163.0, 146.5, 139.1, 137.5, 135.6, 135.6, 134.0, 131.2, 130.7, 130.1, 129.6, 129.1, 128.7, 128.4, 128.4, 126.3, 125.0, 122.4, 93.4, 90.8, 77.6, 21.2 ppm;

m.p. 102.2 – 106.8 °C;

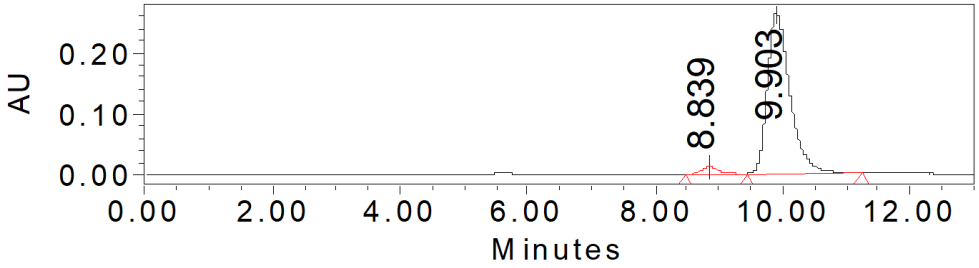
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{31}H_{23}IO_4Na$: 609.0533, found: 609.0535;

$[\alpha]_D^{22}$ = +288.1 (*c* = 0.81 in $CHCl_3$);

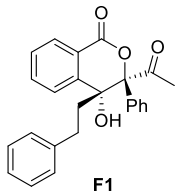
IR (neat): 3463, 3064, 3030, 2922, 1741, 1666, 1660, 1483, 1450, 1286, 1244, 1079, 1005, 971, 769, 693, 588 cm^{-1}.
HPLC spectrum of **C46**



	Retention Time	Area	% Area
1	8.859	7180977	50.05
2	9.946	7166952	49.95

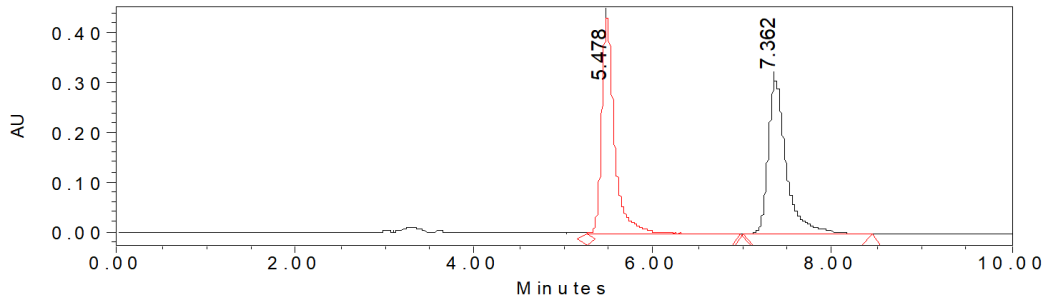


	Retention Time	Area	% Area
1	8.839	240582	3.56
2	9.903	6511716	96.44

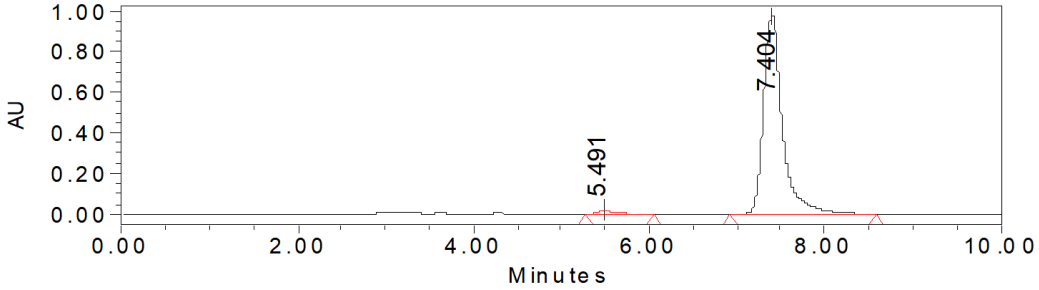


F1

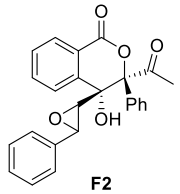
(3*S*,4*R*)-3-acetyl-4-hydroxy-4-phenethyl-3-phenylisochroman-1-one
The residue was purified by column chromatography on silica gel (PE/EtOAc = 5/1) to afford the desired product **F1**; 0.1 mmol scale reaction; 36.9 mg, colorless oil, 96% yield, 97% ee; determined by HPLC analysis [Daicel chiralpak IA, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 230 nm, t_1 = 5.49 min, t_2 = 7.40 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 – 8.10 (m, 1H), 7.85 – 7.82 (m, 1H), 7.78 – 7.72 (m, 3H), 7.51 – 7.41 (m, 4H), 7.15 – 7.05 (m, 3H), 6.89 – 6.86 (m, 2H), 4.96 (d, *J* = 2.8 Hz, 1H), 2.60 (td, *J* = 13.2, 4.4 Hz, 1H), 2.11 – 2.03 (m, 4H), 1.96 – 1.87 (m, 1H), 1.63 (td, *J* = 13.6, 4.4 Hz, 1H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 211.3, 162.9, 146.7, 141.2, 134.9, 131.2, 130.5, 129.2, 128.6, 128.5, 128.2, 126.6, 125.8, 125.8, 121.7, 91.0, 76.2, 38.1, 28.4, 27.2 ppm;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{22}O_4Na$: 409.1410, found: 409.1415;
$[\alpha]_D^{28}$ = +405.2 (*c* = 0.71 in $CHCl_3$);
IR (neat): 3473, 3065, 3027, 2930, 1739, 1707, 1602, 1495, 1452, 1355, 1284, 1246, 1210, 1089, 1040, 756, 707, 587 cm^{-1}.
HPLC spectrum of **F1**



	Retention Time	Area	% Area
1	5.478	4352013	49.97
2	7.362	4357269	50.03



	Retention Time	Area	% Area
1	5.491	195444	1.30
2	7.404	14807457	98.70



F2

(3*S*,4*S*)-3-acetyl-4-hydroxy-3-phenyl-4-((2*S*,3*R*)-3-phenyloxiran-2-yl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/EtOAc = 5/1) to afford the desired product **F2**; 0.1 mmol scale reaction; 35.2 mg, colorless oil, 88% yield, 87:13 dr, >99% ee/>99% ee; determined by HPLC analysis [Daicel chiralpak IE, *n*-hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 230 nm, t_1 = 25.49 min, t_2 = 42.18 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.12 (m, 1H), 7.86 – 7.84 (m, 1H), 7.78 (td, *J* = 7.6, 1.2 Hz, 1H), 7.73 – 7.69 (m, 2H), 7.54 (td, *J* = 7.6, 1.2 Hz, 1H), 7.46 – 7.39 (m, 3H), 7.19 – 7.14 (m, 3H), 6.73 – 6.70 (m, 2H), 4.98 (d, *J* = 1.2 Hz, 1H), 3.31 (d, *J* = 2.0 Hz, 1H), 3.00 – 2.99 (m, 1H), 2.10 (s, 3H);

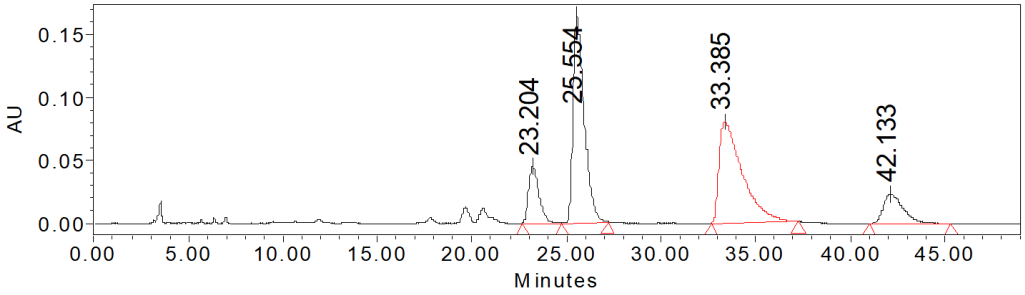
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.5, 162.4, 143.0, 135.6, 135.4, 131.1, 130.3, 129.3, 129.1, 128.8, 128.0, 128.0, 126.4, 125.7, 125.3, 122.4, 89.7. 74.1, 63.3, 55.5, 26.9 ppm;

HRMS (ESI) *m/z* $[M+K]^+$ calcd for $C_{25}H_{20}O_5K$: 439.0942, found: 439.0944;

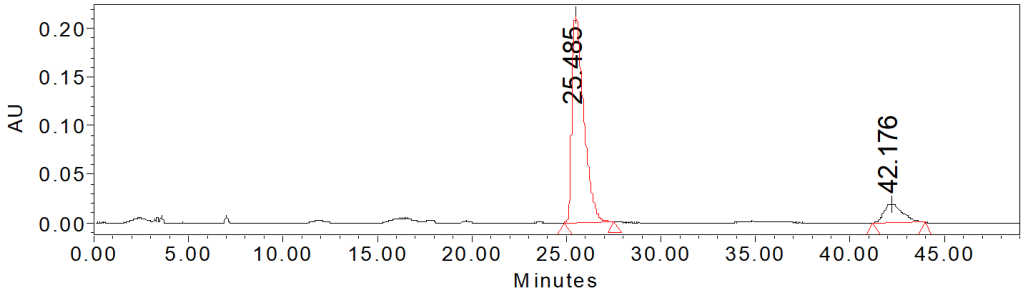
$[\alpha]_D^{28}$ = +226.7 (*c* = 0.76 in $CHCl_3$);

IR (neat): 3464, 3021, 2926, 2334, 1738, 1710, 1602, 1493, 1454, 1358, 1288, 1249, 1212, 1078, 1038, 906, 752, 721, 695, 580 cm^{-1}.

HPLC spectrum of **F2**

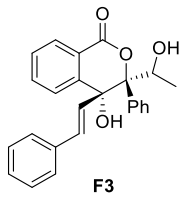


	Retention Time	Area	% Area
1	23.204	1714503	9.99
2	25.554	6876961	40.08
3	33.385	6840723	39.87
4	42.133	1725535	10.06



	Retention Time	Area	% Area
1	25.485	9323401	87.32

2	42.176	1353773	12.68



F3

(3*R*,4*R*)-4-hydroxy-3-((*R*)-1-hydroxyethyl)-3-phenyl-4-((*E*)-styryl)isochroman-1-one

The residue was purified by column chromatography on silica gel (PE/EtOAc = 4/1) to afford the desired product **F3**; 0.1 mmol scale reaction; 35.1 mg, white solid, 91% yield, 97% ee; determined by HPLC analysis [Daicel chiralpak ID, *n*-hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t_1 = 4.99 min, t_2 = 5.66 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.97 – 7.94 (m, 2H), 7.88 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.73 (td, *J* = 7.6, 1.4 Hz, 1H), 7.50 (td, *J* = 7.6, 1.2 Hz, 1H), 7.42 – 7.33 (m, 3H), 7.17 – 7.09 (m, 3H), 7.01 – 6.99 (m, 2H), 6.12 (d, *J* = 16.0 Hz, 1H), 6.01 (d, *J* = 16.0 Hz, 1H), 5.27 (s, 1H), 4.47 – 4.41 (m, 1H), 2.88 (d, *J* = 5.2 Hz, 1H), 1.03 (d, *J* = 6.4 Hz, 3H);

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 163.1, 143.9, 136.1, 135.2, 134.9, 131.0, 130.4, 129.3, 128.3, 128.3, 128.1, 127.8, 127.7, 127.3, 126.5, 125.4, 123.2, 86.1, 77.7, 72.4, 18.9 ppm;

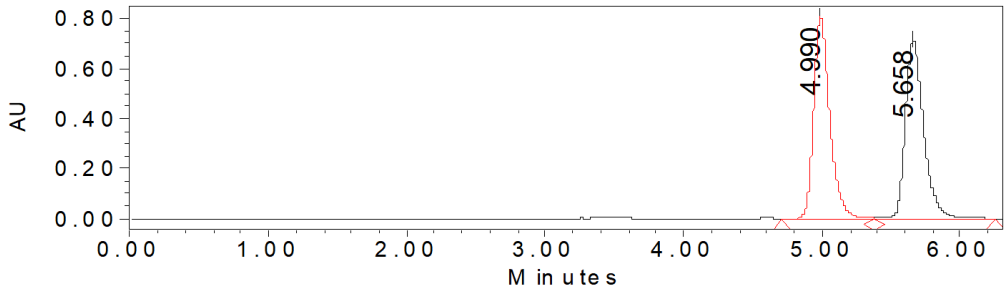
m.p. 149.1 – 152.0 °C;

HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{22}O_4Na$: 409.1410, found: 409.1412;

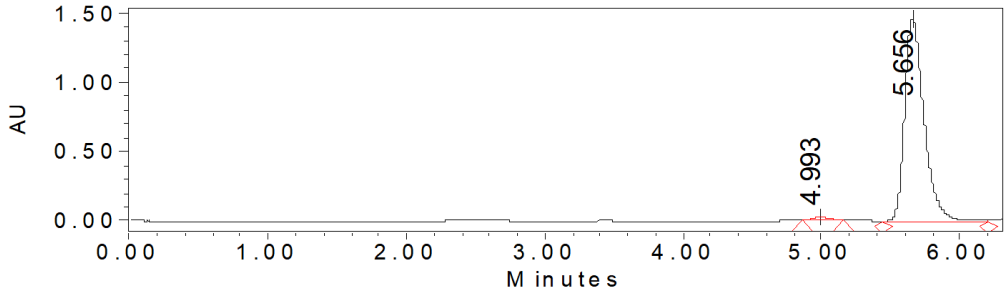
$[\alpha]_D^{28}$ = +302.0 (*c* = 0.75 in $CHCl_3$);

IR (neat): 3350, 3022, 2925, 2856, 1706, 1602, 1493, 1451, 1415, 1375, 1294, 1250, 1218, 1093, 1024, 965, 751, 695, 560 cm^{-1}.

HPLC spectrum of **F3**



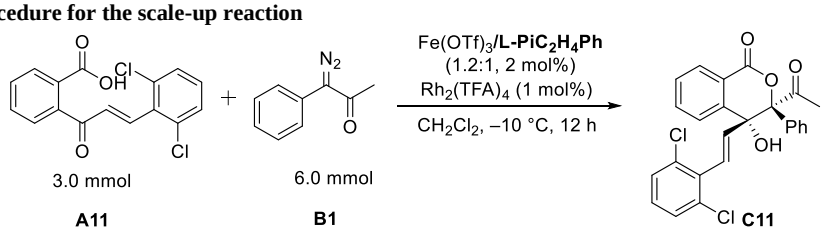
	Retention Time	Area	% Area
1	4.990	6511575	50.01
2	5.658	6508858	49.99



	Retention Time	Area	% Area
1	4.993	191598	1.35
2	5.656	13992313	98.65

(F) Large scale experiment

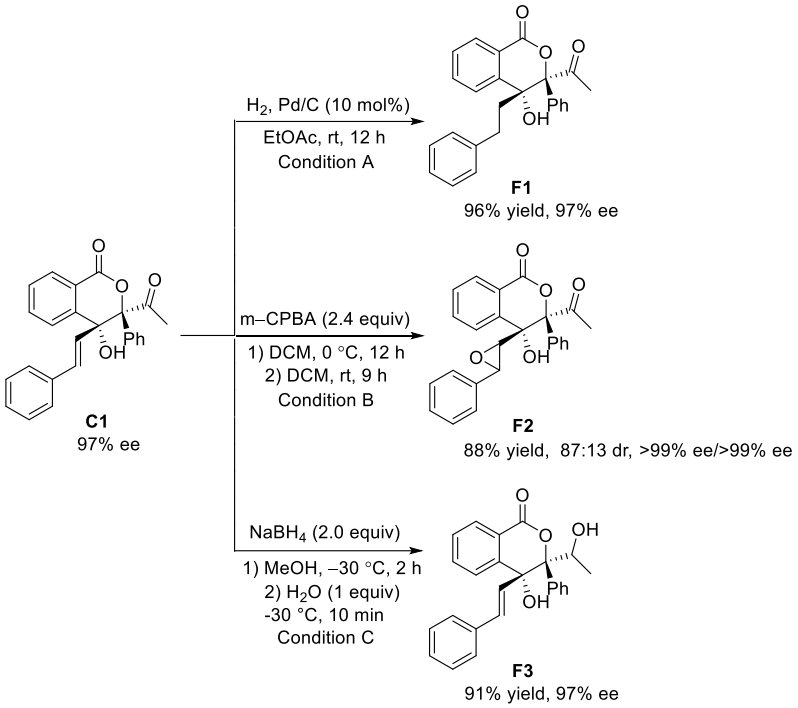
General experimental procedure for the scale-up reaction



Ketoacid **A11** (3.0 mmol, 0.96 g), $Rh_2(TFA)_4$ (21.0 mg, 0.001 mmol, 1 mol%) were weighted into a oven-dried reaction tube. Then, in a glove box, prepared in advance $Fe(OTf)_3$/**L-PiC$_2$H$_4$Ph** catalyst (72.0 mg, 0.02 mmol, 2 mol%) was added to the tube, anhydrous CH_2Cl_2(35.0 mL) was added and the resulting solution was stirred at 35 °C for 2–3 h. After the solution was cooled to –10 °C for 20 min, α-diazoketones **B1** solution (6.0 mmol,

0.96 g, 25 mL CH_2Cl_2) was slowly added, the reaction mixture was stirred at –10 °C for 12 h. The reaction system was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1.5:1) to afford the desired product **C11** in 71% yield (0.963g) with 97% ee.

(G) The transformations of the product



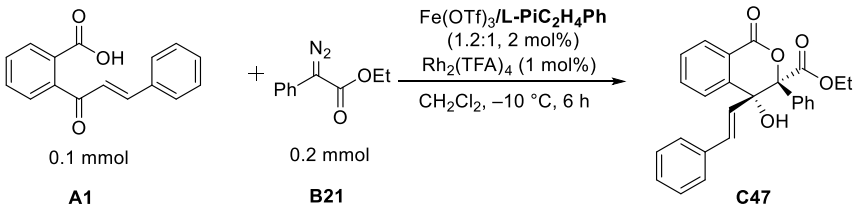
Condition A: The compound **C1** (38.4 mg, 0.10 mmol, 97% ee) was dissolved in EtOAc (2 mL), and 10% Pd/C (3.84 mg) was added to the mixture. Under H_2 atmosphere using balloon, the mixture was stirred at rt for 12 h. After completion of the reaction, the reaction mixture was filtered through a pad of celite, and the solvent was removed under reduced pressure and the obtained crude compound was purified by column chromatography on silica gel (PE/EtOAc = 5/1) to give the product **F1** in 96% yield with 97% ee.

Condition B: A reaction tube was charged with **C1** (38.4 mg, 0.10 mmol, 97% ee), *m*-CPBA (2.0 equiv, 48.8 mg) and anhydrous CH_2Cl_2 (1.0 mL).The resulting solution was stirred at 0 °C for 12 h, then continued to react for 9 h at room temperature. The reaction would be quenched by $NaHCO_3$ (aq). The resulting solution was extracted with CH_2Cl_2 (3×10 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by column chromatography on silica gel (PE/EtOAc = 5/1) to afford the desired product **F2** in 88% yield with 87:13 dr and >99% ee/>99% ee.

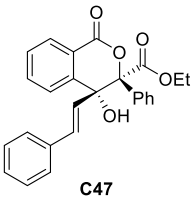
Condition C: An oven-dried test tube was charged with **C1** (38.4 mg, 0.10 mmol, 97% ee) and $NaBH_4$ (2.0 equiv, 7.6 mg) under N_2 atmosphere. After MeOH (2.0 mL) was added under –30 °C, the mixture was stirred at –30 °C for 2 h. Then the reaction was quenched by H_2O (15 μL) at –30 °C. The reaction system was purified by column chromatography on silica gel (PE/EtOAc = 4/1) to afford the desired product **F3** in 91% yield with 97% ee.

(H) Control experiments

Control experiment 1

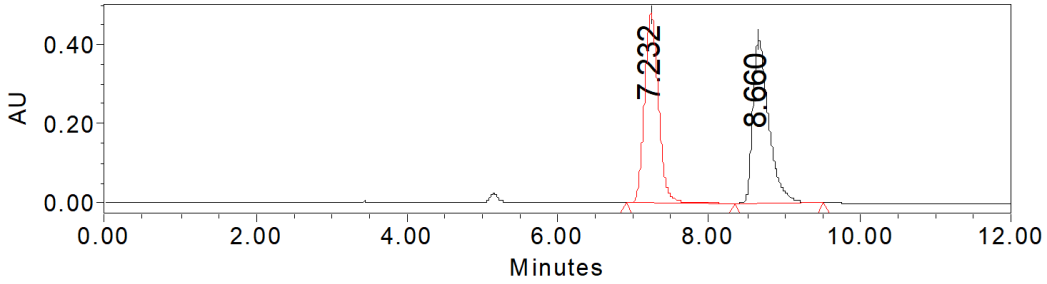


In a glovebox, ketoacid **A1** (0.1 mmol), $Rh_2(TFA)_4$ (0.70 mg, 0.001 mmol, 1 mol%), and $Fe(OTf)_3$/**L-PiC$_2$H$_4$Ph** (2.0 mol%, 1.2:1) were weighted into a dried test tube. Anhydrous CH_2Cl_2 (1.0 mL) was added and the solution was stirred at 35 °C for 0.5 h. After the solution was cooled to –10 °C, ethyl 2-diazo-2-phenylacetate **B21** (0.20 mmol in 1.0 mL CH_2Cl_2) was added, and the reaction mixture was stirred at –10 °C for 6 h. The reaction system was purified by column chromatography on silica gel (PE/EtOAc = 10:1) to afford the desired product **C47** in 99% yield without enantioselectivity.



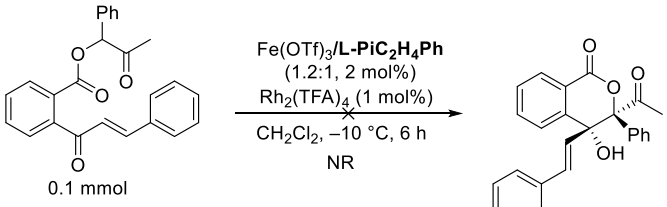
C47

C47: HPLC analysis [Daicel chiralpak IA, n-hexane/i-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t_1 = 7.23 min, t_2 = 8.66 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.83 – 7.79 (m, 3H), 7.73 (td, *J* = 7.6, 1.6 Hz, 1H), 7.53 – 7.49 (m, 1H), 7.44 – 7.38 (m, 3H), 7.22 – 7.17 (m, 3H), 7.13 – 7.11 (m, 2H), 6.20 (d, *J* = 15.6 Hz, 1H), 6.08 (dd, *J* = 15.6, 1.2 Hz, 1H), 5.25 (d, *J* = 1.2 Hz, 1H), 4.19 – 4.11 (m, 1H), 4.06 – 3.97 (m, 1H), 1.01 (t, *J* = 7.2 Hz, 3H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 172.2, 163.2, 145.2, 135.9, 135.3, 132.8, 132.0, 129.8, 129.1, 128.8, 128.4, 128.0, 128.0, 126.9, 126.7, 126.6, 125.1, 122.9, 87.1, 76.6, 63.1, 13.5 ppm;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{26}H_{22}O_5Na$: 437.1359, found: 437.1358;
HPLC spectrum of **C47**



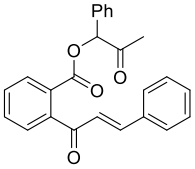
	Retention Time	Area	% Area
1	7.232	6122554	50.34
2	8.660	6040087	49.66

Control experiment 2



D1 **C1**

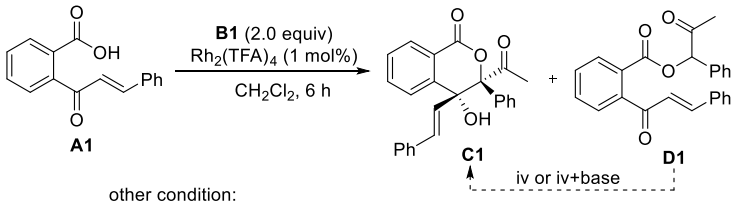
In a glovebox, intermediate **D1** (0.1 mmol), $Rh_2(TFA)_4$ (0.70 mg, 0.001 mmol, 1 mol%) and $Fe(OTf)_3$/**L-PiC_2H_4Ph** (2.0 mol%, 1.2:1) were weighted into a dried test tube. Anhydrous CH_2Cl_2 (1.0 mL) was added and the solution was stirred at 35 °C for 0.5 h. After the solution was cooled to –10 °C, the reaction mixture was stirred at –10 °C for 6 h. No reaction happened in this condition.



D1

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.66 – 7.62 (m, 1H), 7.59 – 7.55 (m, 1H), 7.46 – 7.44 (m, 3H), 7.41 – 7.33 (m, 8H), 7.21 (d, *J* = 16.4 Hz, 1H), 6.93 (d, *J* = 16.4 Hz, 1H), 6.10 (s, 1H), 2.11 (s, 3H);
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 201.2, 196.0, 165.5, 145.5, 142.2, 134.3, 132.7, 132.6, 130.6, 130.6, 129.7, 129.3, 128.9, 128.8, 128.4, 128.2, 128.1, 127.6, 127.1, 81.9, 26.1 ppm;
HRMS (ESI) *m/z* $[M+Na]^+$ calcd for $C_{25}H_{20}O_4Na$: 407.1254, found: 407.1251;

Control experiment 3



other condition:		
i. 40 °C	74% yield	24% yield
ii. -10 °C	30% yield	69% yield
iii. $Fe(OTf)_3$ (4.8 mol%), -10 °C	40% yield	54% yield
iv. $Fe(OTf)_3$ (4.8 mol%), **L*** (4 mol%)	91% yield	trace

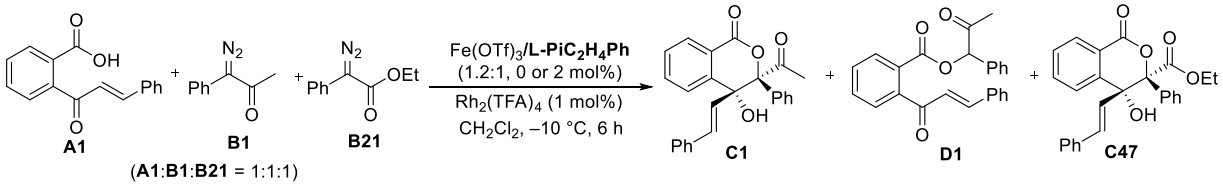
Condition **i**: In a glovebox, ketoacid **A1** (0.1 mmol) and $Rh_2(TFA)_4$ (0.70 mg, 0.001 mmol, 1 mol%) were weighted into a dried test tube. Anhydrous CH_2Cl_2 (1.0 mL) was added and the solution was stirred at 40 °C. α-diazoketone **B1** (0.20 mmol in 1.0 mL CH_2Cl_2) was added, the reaction mixture was stirred at 40 °C for 6 h. The reaction system was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1:1) to afford the racemic product **C1** in 74% yield and racemic intermediate **D1** in 24% yield.

Condition **ii**: In a glovebox, ketoacid **A1** (0.1 mmol) and $Rh_2(TFA)_4$ (0.70 mg, 0.001 mmol, 1 mol%) were weighted into a dried test tube. Anhydrous CH_2Cl_2 (1.0 mL) was added and the solution was stirred at −10 °C. Then, α-diazoketone **B1** (0.20 mmol in 1.0 mL CH_2Cl_2) was added at −10 °C, and the reaction mixture was stirred at −10 °C for 6 h. The reaction system was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1:1) to afford the racemic product **C1** in 30% yield and racemic intermediate **D1** in 69% yield.

Condition **iii**: In a glovebox, ketoacid **A1** (0.1 mmol), $Rh_2(TFA)_4$ (0.70 mg, 0.001 mmol, 1 mol%) and $Fe(OTf)_3$ (4.8 mol%) were weighted into a dried test tube. Anhydrous CH_2Cl_2 (1.0 mL) was added and the solution was stirred at 35 °C for 0.5 h. After the solution was cooled to −10 °C, α-diazoketone **B1** (0.20 mmol in 1.0 mL CH_2Cl_2) was added, and the reaction mixture was stirred at −10 °C for 6 h. The reaction system was purified by column chromatography on silica gel (PE/CH_2Cl_2 = 1:1) to afford the racemic product **C1** in 40% yield and racemic intermediate **D1** in 54% yield.

Condition **iv**: In a glovebox, ketoacid **A1** (0.1 mmol), $Rh_2(TFA)_4$ (0.70 mg, 0.001 mmol, 1 mol%) and $Fe(OTf)_3$/**L$_3$-PiC_2H_4Ph** (4 mol%, 1.2:1) were weighted into a dried test tube. Anhydrous CH_2Cl_2 (1.0 mL) was added and the solution was stirred at 35 °C for 0.5 h. After the solution was cooled to −10 °C, α-diazoketone **B1** (0.20 mmol in 1.0 mL CH_2Cl_2) was added, and the reaction mixture was stirred at −10 °C for 6 h. The reaction system was purified by column chromatography on silica gel (PE/EtOAc = 10:1) to afford the desired product **C1** in 91% yield with 97% ee and racemic intermediate **D1** in trace yield.

Control experiment 4

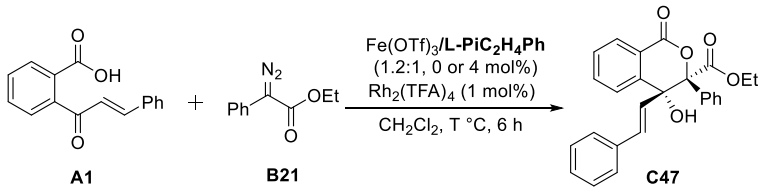


1) with Fe-L: **C1** (45% yield, 95% ee); **C47** (51% yield, 0 % ee)
2) without Fe-L: **C1** (18% yield); **D1** (32% yield); **C47** (54% yield)

Condition 1: In a glovebox, ketoacid **A1** (0.1 mmol), $Rh_2(TFA)_4$ (0.70 mg, 0.001 mmol, 1 mol%) and $Fe(OTf)_3$/**L-PiC_2H_4Ph** (2.0 mol%, 1.2:1) were weighted into a dried test tube. Anhydrous CH_2Cl_2 (1.0 mL) was added and the solution was stirred at 35 °C for 0.5 h. After the solution was cooled to −10 °C, α-diazoketone **B1** (0.10 mmol) and α-diazoester **B27** (0.10 mmol) in 1.0 mL CH_2Cl_2 were added, and the reaction mixture was stirred at −10 °C for 6 h. The reaction system was purified by column chromatography on silica gel (PE/EtOAc = 10:1) to afford the desired product **C1** in 45% yield with 95% ee and **C47** in 51% yield with 0% ee.

Condition 1: In a glovebox, ketoacid **A1** (0.1 mmol), $Rh_2(TFA)_4$ (0.70 mg, 0.001 mmol, 1 mol%) weighted into a dried test tube. Anhydrous CH_2Cl_2 (1.0 mL) was added. After the solution was cooled to −10 °C, α-diazoketone **B1** (0.10 mmol) and α-diazoester **B27** (0.10 mmol) in 1.0 mL CH_2Cl_2 were added, and the reaction mixture was stirred at −10 °C for 6 h. The reaction system was purified by column chromatography on silica gel (PE/EtOAc = 10:1) to afford the desired product **C1** in 18% yield, **D1** in 32% yield and **C47** in 54% yield.

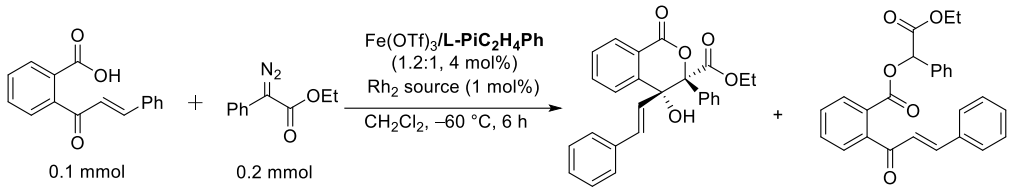
Control experiment 5



Entry[a]	T	Background reaction: yield (%)[b]	Cat: yield (%)[b]	ee (%)[c]
1	-30	91	92	0
2	-40	90	90	0
3	-50	87	89	0
4	-60	85	82	0

[a] Unless otherwise noted, all reactions were carried out with **A1** (0.10 mmol), **B21** (2.0 equiv), $Fe(OTf)_3$/**L-PiC_2H_4Ph** (1:1, 0 or 4 mol%), $Rh_2(TFA)_4$(1 mol%), in CH_2Cl_2 (2.0 mL) at T °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC analysis on a chiral stationary phase. All dr value were up to >19:1 detected by ^1H NMR.

Control experiment 6

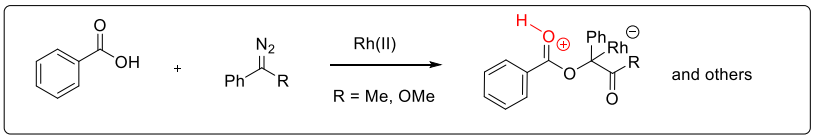


A1 **B21** **C47** **D2**

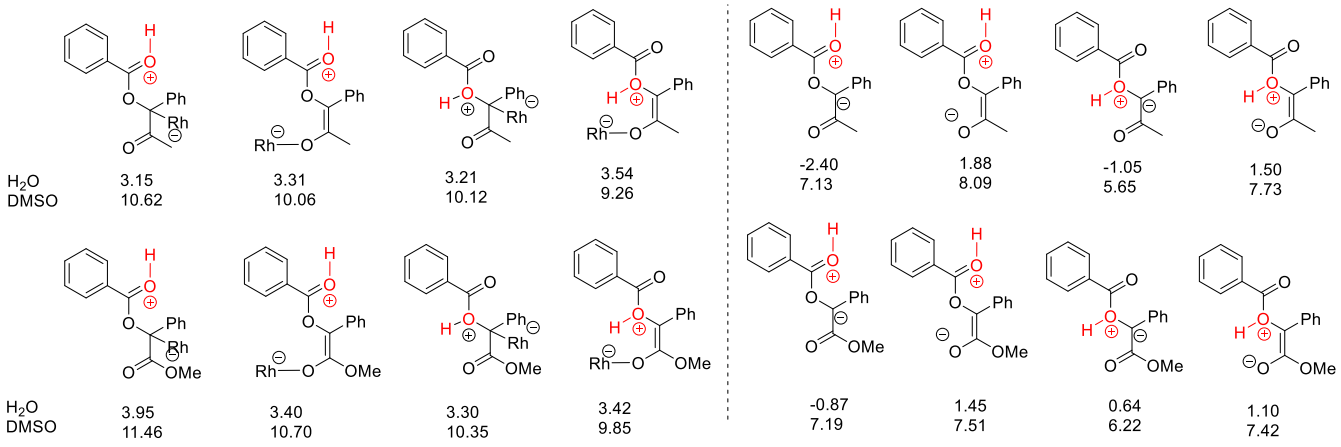
Entry[a]	Rh_2 source	Conv. (%)	**C47/D2**[b]	**C47** yield (%)[b]	**C47** ee (%)[c]
1	$Rh_2(TFA)_4$	>99	85/15	85	0
2	$Rh_2(OAc)_4$	>99	88/12	88	0
3	$Rh_2(esp)_4$	>99	70/30	70	0
4	$Rh_2(Oct)_4$	>99	80/20	80	0
5	$Rh_2(piv)_4$	>99	35/65	35	0

[a] Unless otherwise noted, the reactions were performed with **A1** (0.10 mmol), **B21** (0.20 mmol), $Fe(OTf)_3$ (4.8 mol%), **L-PiC$_2$H$_4$Ph** (4 mol%), Rh_2 source (1 mol%), in CH_2Cl_2 (2.0 mL) at –60 °C for 6 h. [b] Isolated yield. [c] Determined by chiral HPLC.

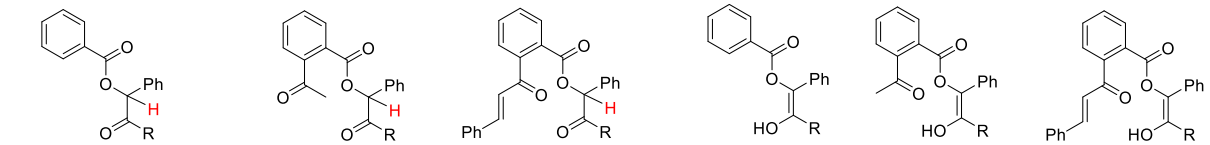
(I) pK_{aH} prediction via XGBoost



pK_{aH} prediction via XGBoost (Agenw. Chem. Int. Ed. 2020, 59, 19282)



pK_a prediction via XGBoost (Agenw. Chem. Int. Ed. 2020, 59, 19282)

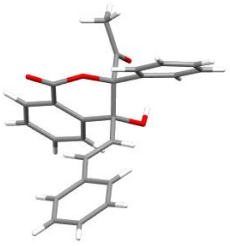


R	H_2O	DMSO	H_2O	DMSO	H_2O	DMSO	H_2O	DMSO	H_2O	DMSO	H_2O	DMSO
Me	7.82	16.17	7.51	13.06	7.69	13.36	8.04	13.37	6.77	12.77	6.50	12.01
OMe	**10.30**	**17.43**	**7.23**	**13.43**	7.60	13.92	7.52	12.16	6.04	11.52	6.03	10.92
Ph	8.56	16.53	7.77	13.55	**7.31**	**11.64**	8.01	12.53	6.75	12.15	6.11	9.70

website: http://isyn.luoszgroup.com/prediction

(J) X-ray crystal structure of the product C1 and F3

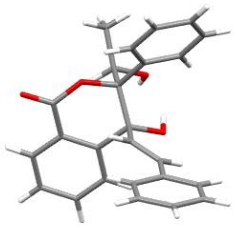
Single crystal of **C1** was obtained by recrystallization in hexane, CH_2Cl_2 and ^{i}PrOH; CCDC (**C1**: 2004207) contains the supplementary crystallographic data which can be obtained free of charge from The Cambrige Crystallographic Data Centere.



Formula	$C_{25}H_{20}O_4$
Formula mass (amu)	384.41
Space group	P 61
a (Å)	17.6742 (3)
b (Å)	17.6742 (3)
c (Å)	11.6231 (3)
α (deg)	90
β (deg)	90
γ (deg)	120
V (Å^3)	3144.36 (15)
Z	6
λ (Å)	1.54178
T (K)	170
ρ_{calcd} (g cm^{-3})	1.218
μ (mm^{-1})	0.663
Transmission factors	0.797, 0.975
θ_{max} (deg)	80.488
No. of unique data, including $F_o^2 < 0$	4224
No. of unique data, with $F_o^2 > 2\sigma(F_o^2)$	4032
No. of variables	268
$R(F)$ for $F_o^2 > 2\sigma(F_o^2)$ [a]	0.0383
$R_w(F_o^2)$ [b]	0.1046
Goodness of fit	1.089

[a] $R(F) = \sum||F_o| - |F_c|| / \sum|F_o|$.
[b] $R_w(F_o^2) = [\sum[w(F_o^2 - F_c^2)^2] / \sum wF_o^4]^{1/2}$; $w^{-1} = [\sigma^2(F_o^2) + (Ap)^2 + Bp]$, where $p = [\max(F_o^2,0) + 2F_c^2] / 3$

Single crystal of **F3** was obtained by recrystallization in hexane, CH_2Cl_2 and ^{i}PrOH; CCDC (**F3**: 2085693) contains the supplementary crystallographic data which can be obtained free of charge from The Cambrige Crystallographic Data Centere.

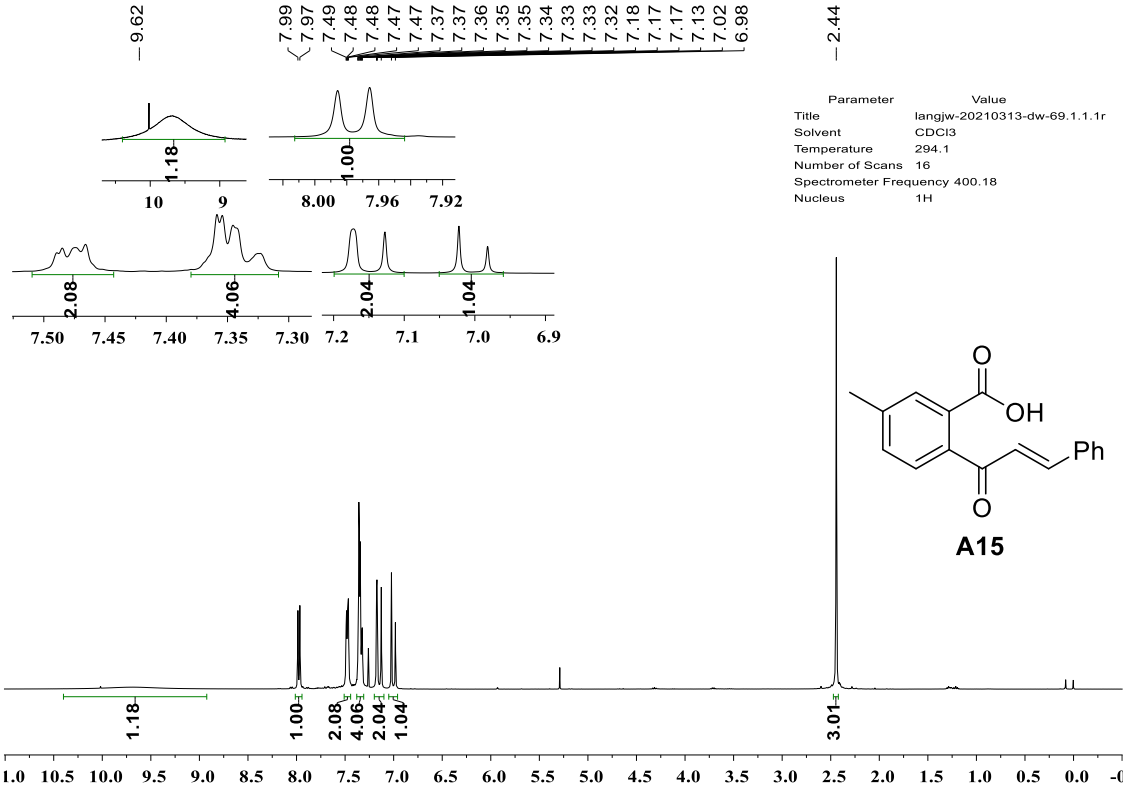


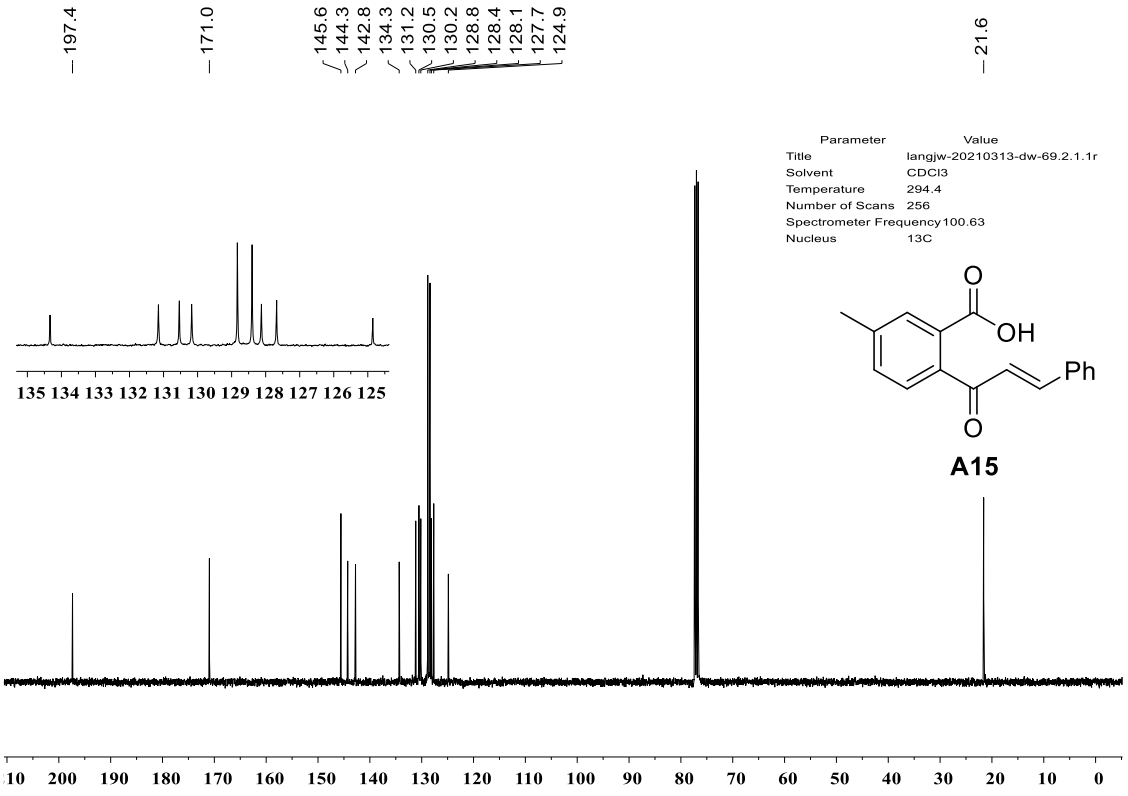
Formula	$C_{25}H_{22}O_4$
Formula mass (amu)	386.42
Space group	P 21 21 21
a (Å)	6.5263 (1)
b (Å)	13.9032 (3)
c (Å)	21.5712 (4)
α (deg)	90
β (deg)	90
γ (deg)	90
V (Å^3)	1957.29 (6)
Z	4
λ (Å)	1.54178
T (K)	173
ρ_{calcd} (g cm^{-3})	1.311

μ (mm^{-1})	0.711
Transmission factors	0.794, 0.940
θ_{max} (deg)	68.254
No. of unique data, including $F_o^2 < 0$	3454
No. of unique data, with $F_o^2 > 2\sigma(F_o^2)$	3307
No. of variables	271
$R(F)$ for $F_o^2 > 2\sigma(F_o^2)$ [a]	0.0282
$R_w(F_o^2)$ [b]	0.0752
Goodness of fit	1.127

[a] $R(F) = \sum||F_o| - |F_c|| / \sum|F_o|$.
[b] $R_w(F_o^2) = [\sum[w(F_o^2 - F_c^2)^2] / \sum wF_o^4]^{1/2}$; $w^{-1} = [\sigma^2(F_o^2) + (Ap)^2 + Bp]$, where $p = [\max(F_o^2,0) + 2F_c^2] / 3$

(K) Copy of ^{1}H, ^{13}C{^{1}H}, and ^{19}F{^{1}H} NMR Spectra.

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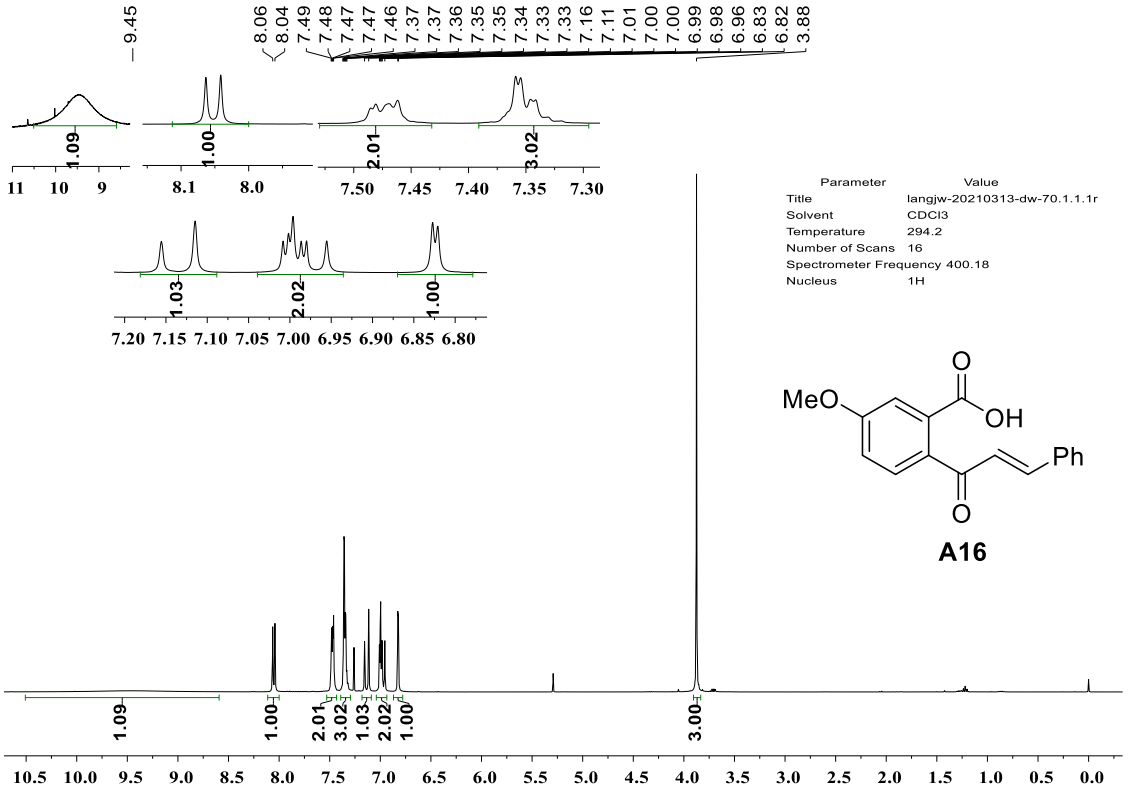
145.6
144.3
142.8
134.3
131.2
130.5
130.2
128.8
128.4
128.1
127.7
124.9
21.6
Parameter Value
Title langjw-20210313-dw-69.2.1.1r
Solvent CDCl3
Temperature 294.4
Number of Scans 256
Spectrometer Frequency 100.63
Nucleus 13C
O
OH
Ph
O
A15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8.06
8.04
7.49
7.48
7.47
7.47
7.46
7.37
7.37
7.36
7.35
7.35
7.34
7.33
7.33
7.16
7.11
7.01
7.00
7.00
6.99
6.98
6.96
6.83
6.82
3.88
1.09
1.00
2.01
3.02
1.03
2.02
1.00
Parameter Value
Title langjw-20210313-dw-70.1.1.1r
Solvent CDCl3
Temperature 294.2
Number of Scans 16
Spectrometer Frequency 400.18
Nucleus 1H
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O
OH
Ph
O
A16
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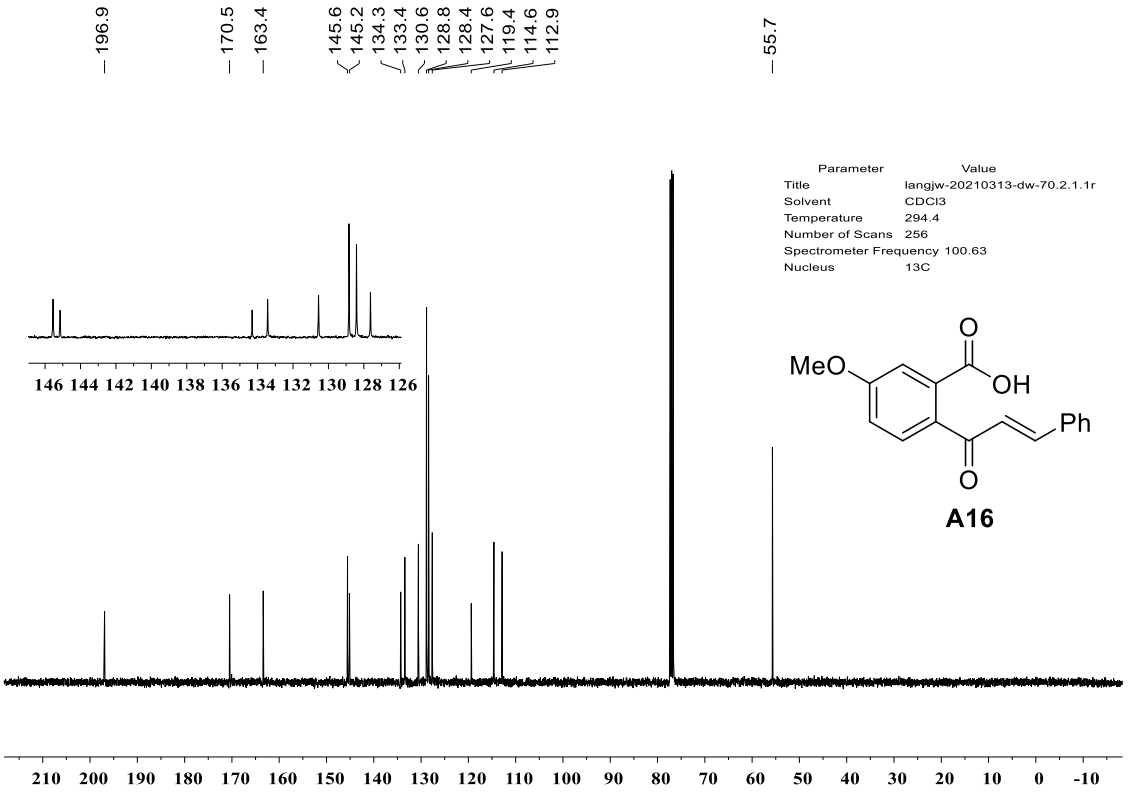
1.00
2.01
3.02
1.03
2.02
1.00
3.00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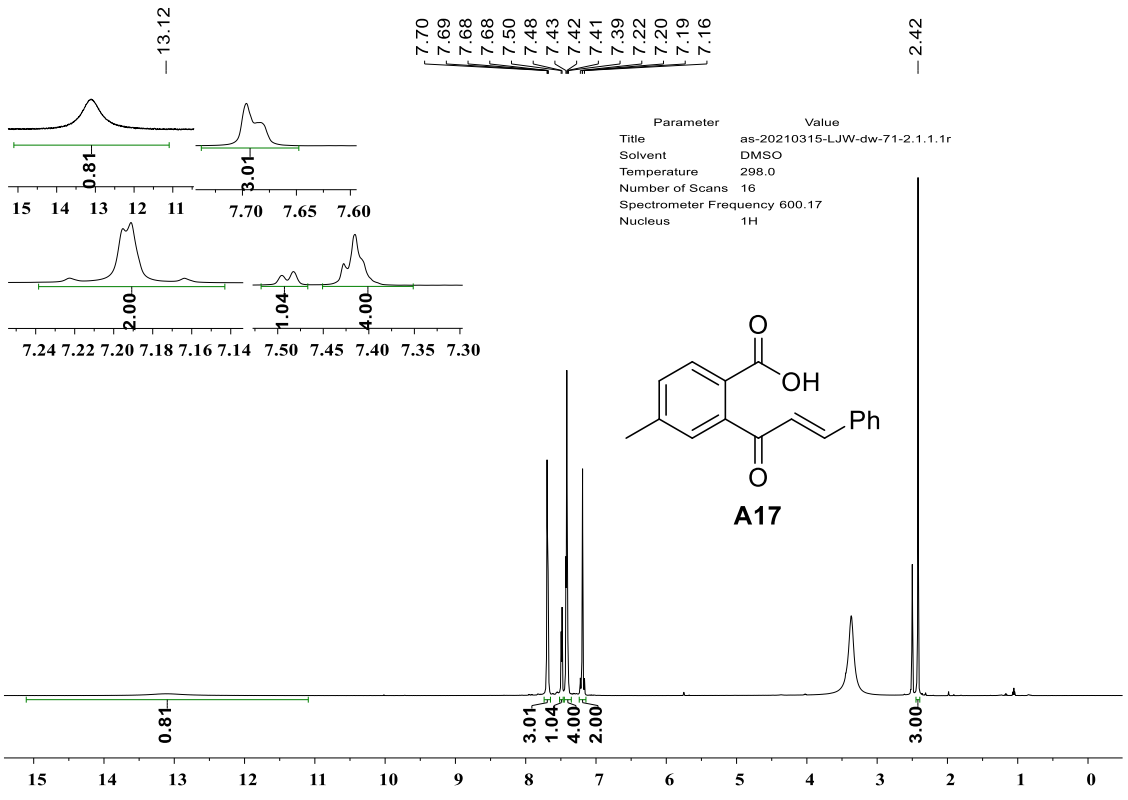
170.5
163.4
145.6
145.2
134.3
133.4
130.6
128.8
128.4
127.6
119.4
114.6
112.9
55.7
Parameter Value
Title langjw-20210313-dw-70.2.1.1r
Solvent CDCl3
Temperature 294.4
Number of Scans 256
Spectrometer Frequency 100.63
Nucleus 13C
MeO
OH
Ph
O
A16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7.69
7.68
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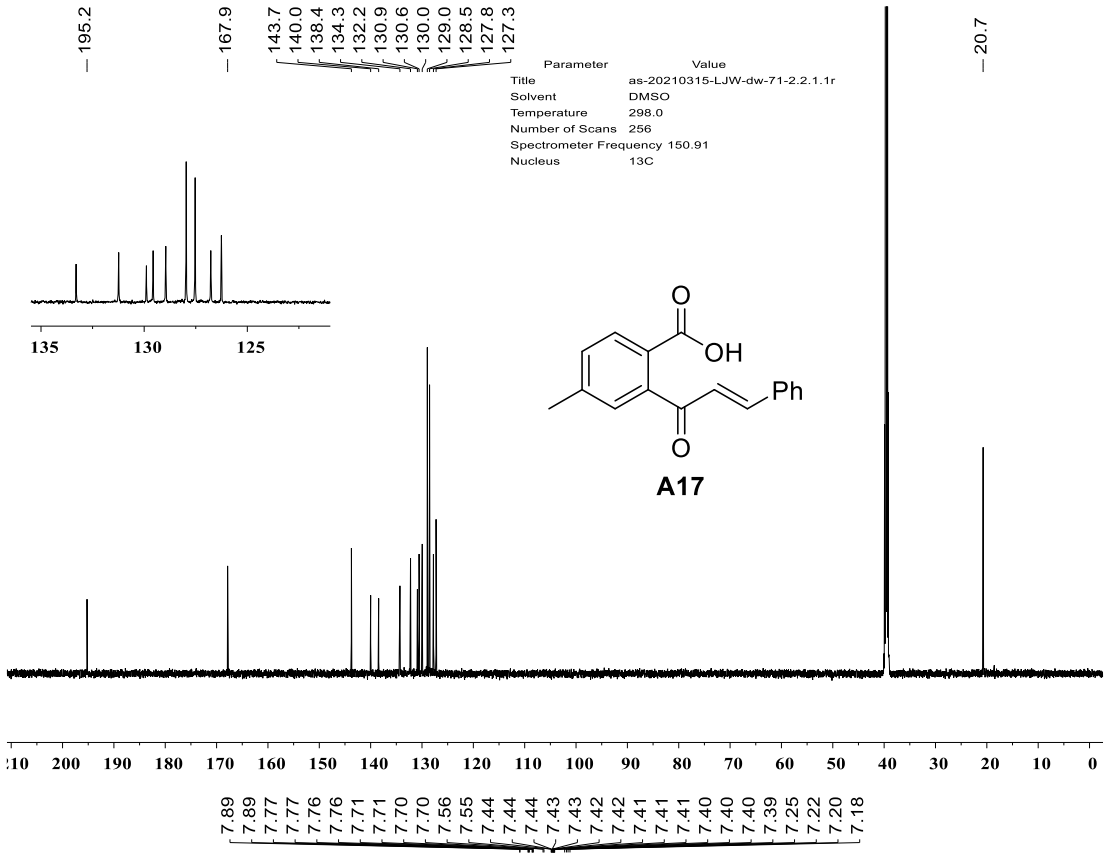
7.50
7.48
7.43
7.42
7.41
7.39
7.22
7.20
7.19
7.16
2.42
Parameter Value
Title as-20210315-LJW-dw-71-2.1.1.1r
Solvent DMSO
Temperature 298.0
Number of Scans 16
Spectrometer Frequency 600.17
Nucleus 1H
OH
Ph
A17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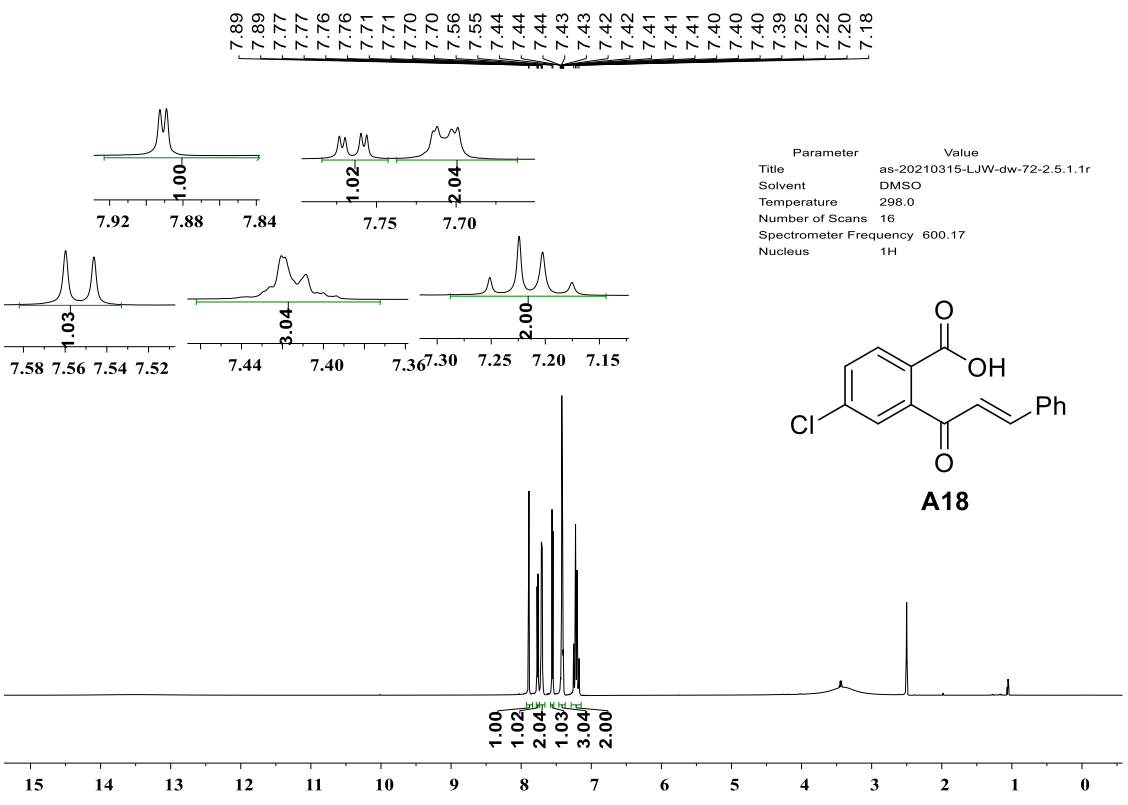
167.9
143.7
140.0
138.4
134.3
132.2
130.9
130.6
130.0
129.0
128.5
127.8
127.3
20.7
Parameter Value
Title as-20210315-LJW-dw-71-2.2.1.1r
Solvent DMSO
Temperature 298.0
Number of Scans 256
Spectrometer Frequency 150.91
Nucleus 13C
135 130 125
O
OH
Ph
O
A17
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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7.89
7.77
7.77
7.76
7.76
7.71
7.70
7.70
7.56
7.55
7.44
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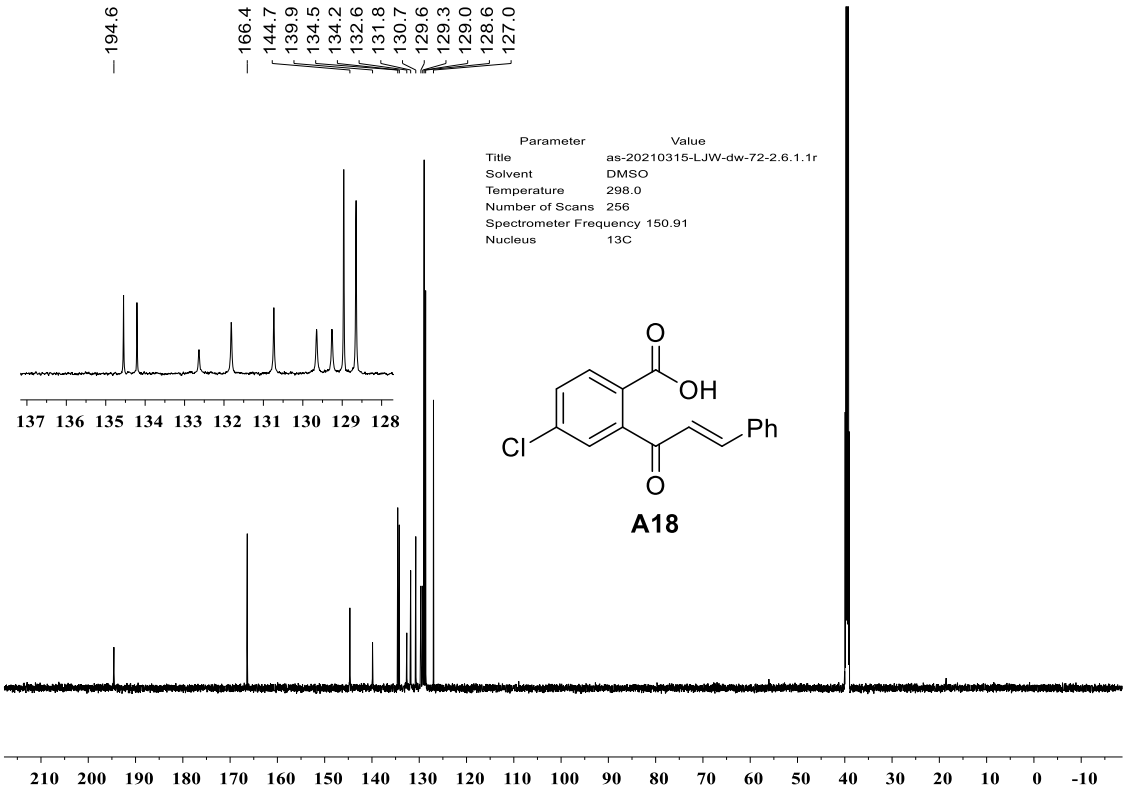
7.44
7.43
7.43
7.42
7.42
7.41
7.41
7.41
7.40
7.40
7.40
7.39
7.25
7.22
7.20
7.18
1.00
1.02
2.04
1.03
3.04
2.00
7.92 7.88 7.84
7.75 7.70
7.58 7.56 7.54 7.52
7.44 7.40 7.36
7.30 7.25 7.20 7.15
Parameter Value
Title as-20210315-LJW-dw-72-2.5.1.1r
Solvent DMSO
Temperature 298.0
Number of Scans 16
Spectrometer Frequency 600.17
Nucleus 1H
O
OH
Cl
Ph
O
A18
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0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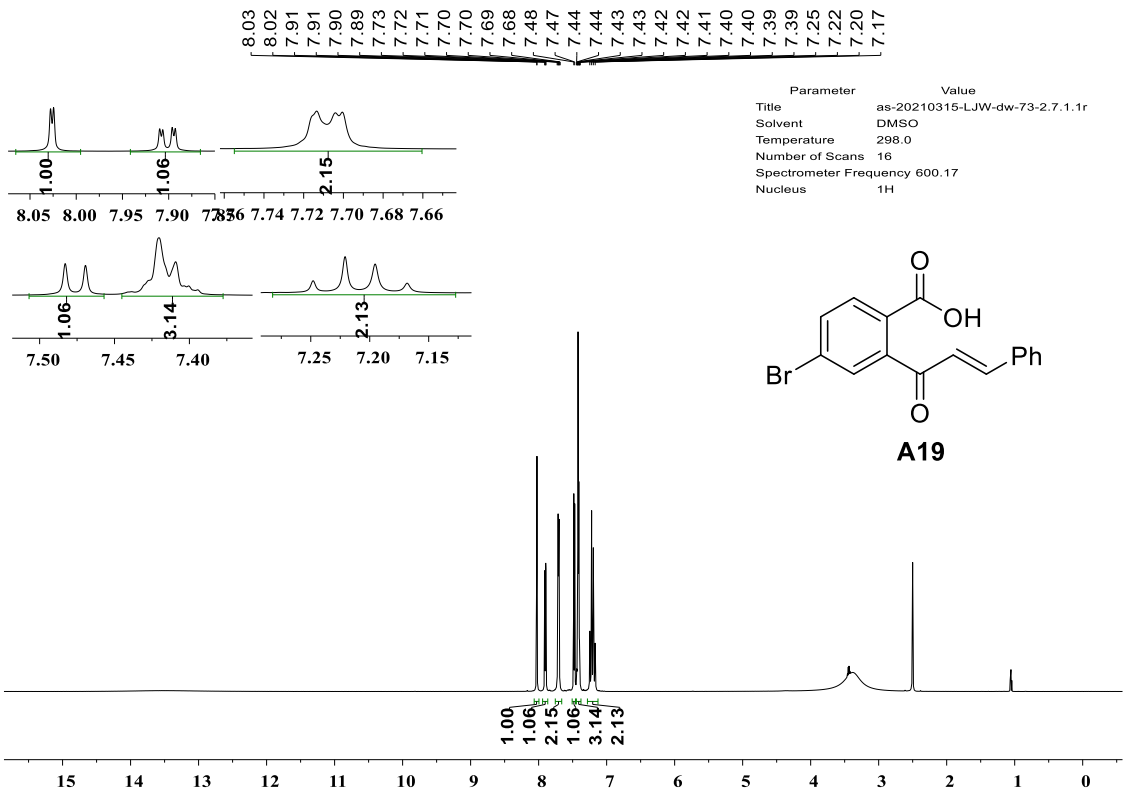
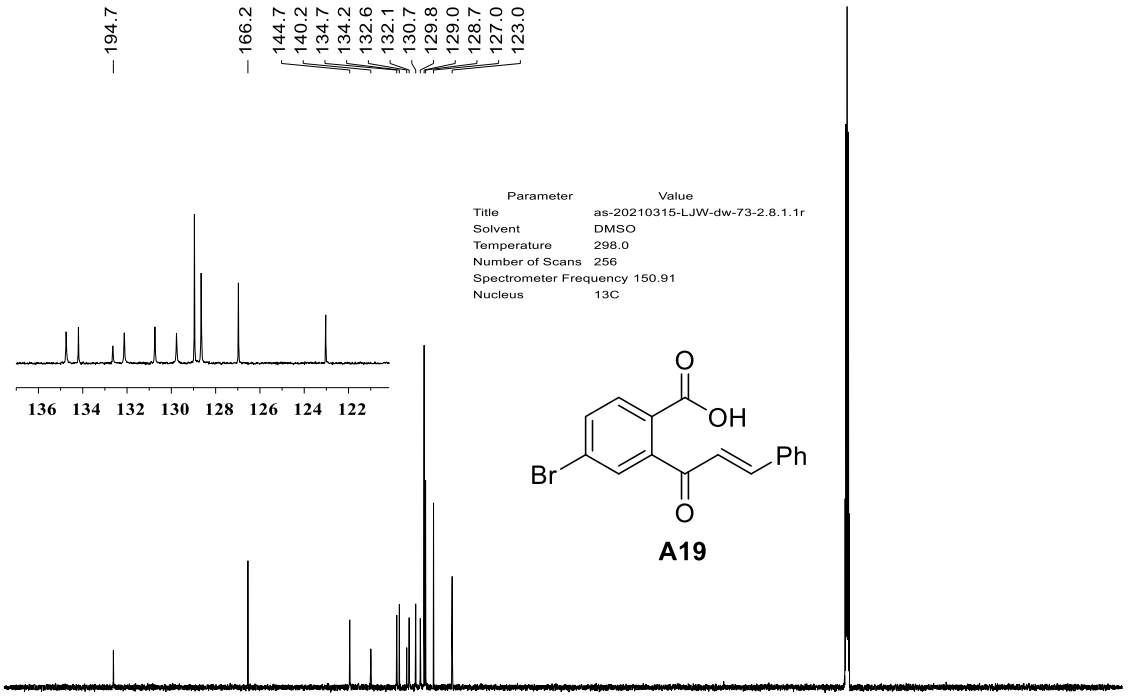
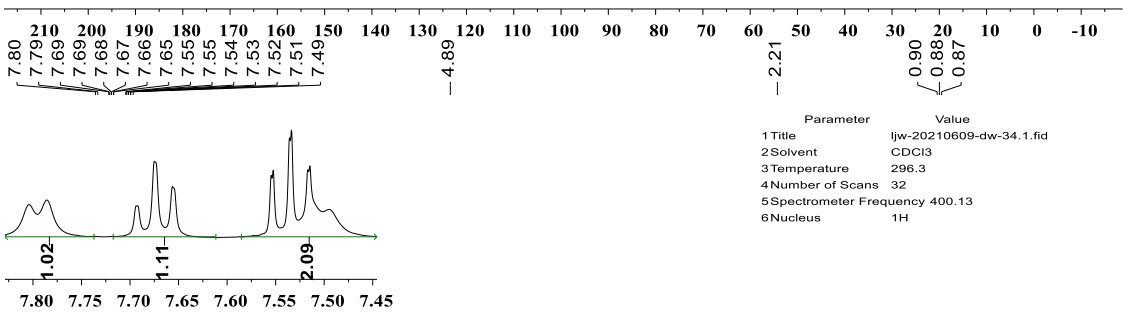
166.4
144.7
139.9
134.5
134.2
132.6
131.8
130.7
129.6
129.3
129.0
128.6
127.0
Parameter Value
Title as-20210315-LJW-dw-72-2.6.1.1r
Solvent DMSO
Temperature 298.0
Number of Scans 256
Spectrometer Frequency 150.91
Nucleus 13C
137 136 135 134 133 132 131 130 129 128
O
OH
Cl
Ph
O
A18
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10


8.03
8.02
7.91
7.91
7.90
7.89
7.73
7.72
7.71
7.70
7.70
7.69
7.68
7.48
7.47
7.44
7.44
7.43
7.43
7.42
7.42
7.41
7.40
7.40
7.39
7.39
7.25
7.22
7.20
7.17
Parameter Value
Title as-20210315-LJW-dw-73-2.7.1.1r
Solvent DMSO
Temperature 298.0
Number of Scans 16
Spectrometer Frequency 600.17
Nucleus 1H
1.00
1.06
2.15
8.05 8.00 7.95 7.90 7.74 7.72 7.70 7.68 7.66
1.06
3.14
2.13
7.50 7.45 7.40 7.25 7.20 7.15
O
OH
Br
Ph
O
A19
1.00
1.06
2.15
1.06
3.14
2.13
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0


194.7
166.2
144.7
140.2
134.7
134.2
132.6
132.1
130.7
129.8
129.0
128.7
127.0
123.0
Parameter Value
Title as-20210315-LJW-dw-73-2.8.1.1r
Solvent DMSO
Temperature 298.0
Number of Scans 256
Spectrometer Frequency 150.91
Nucleus 13C
136 134 132 130 128 126 124 122
O
OH
Br
Ph
O
A19
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10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7.79
7.69
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7.68
7.67
7.66
7.65
7.55
7.55
7.54
7.53
7.52
7.51
7.49
4.89
2.21
0.90
0.88
0.87
Parameter Value
1 Title ljw-20210609-dw-34.1.fid
2 Solvent CDCl3
3 Temperature 296.3
4 Number of Scans 32
5 Spectrometer Frequency 400.13
6 Nucleus 1H
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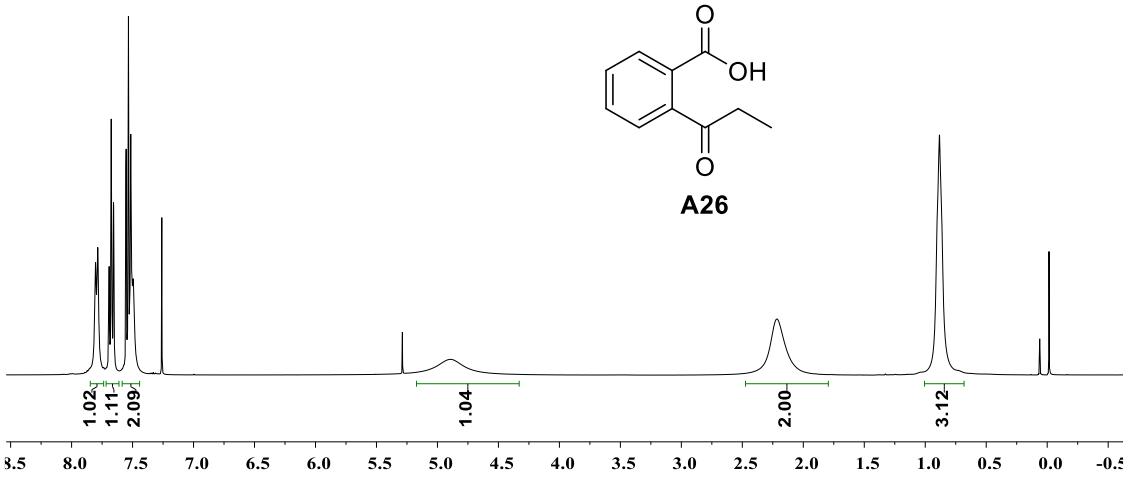
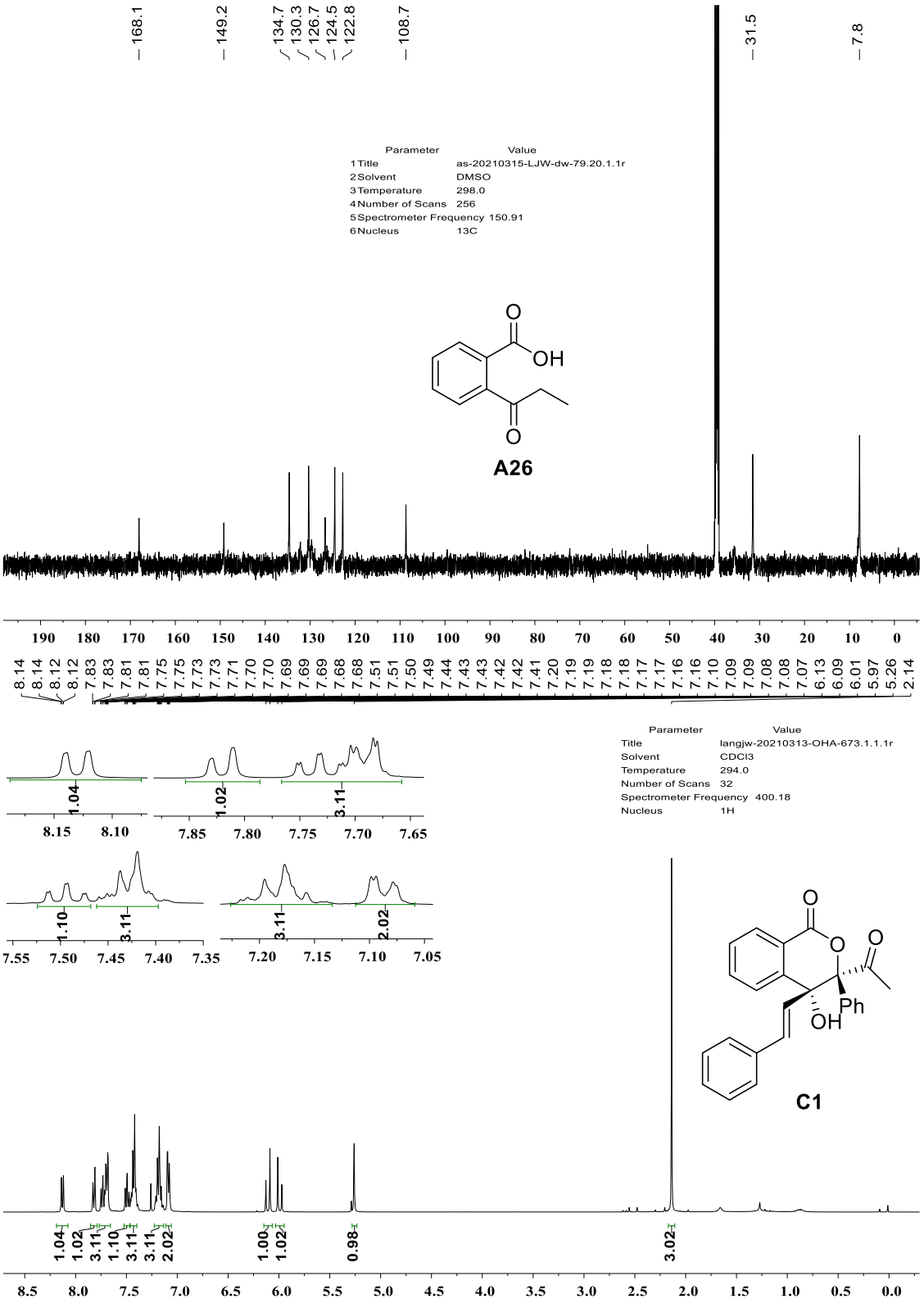
1.11
2.09
7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45


O
OH
O
A26
1.02
1.11
2.09
1.04
2.00
3.12
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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130.3
126.7
124.5
122.8
108.7
31.5
7.8
Parameter Value
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2 Solvent DMSO
3 Temperature 298.0
4 Number of Scans 256
5 Spectrometer Frequency 150.91
6 Nucleus 13C
O
OH
O
A26
Parameter Value
Title langjw-20210313-OHA-673.1.1.1r
Solvent CDCl3
Temperature 294.0
Number of Scans 32
Spectrometer Frequency 400.18
Nucleus 1H
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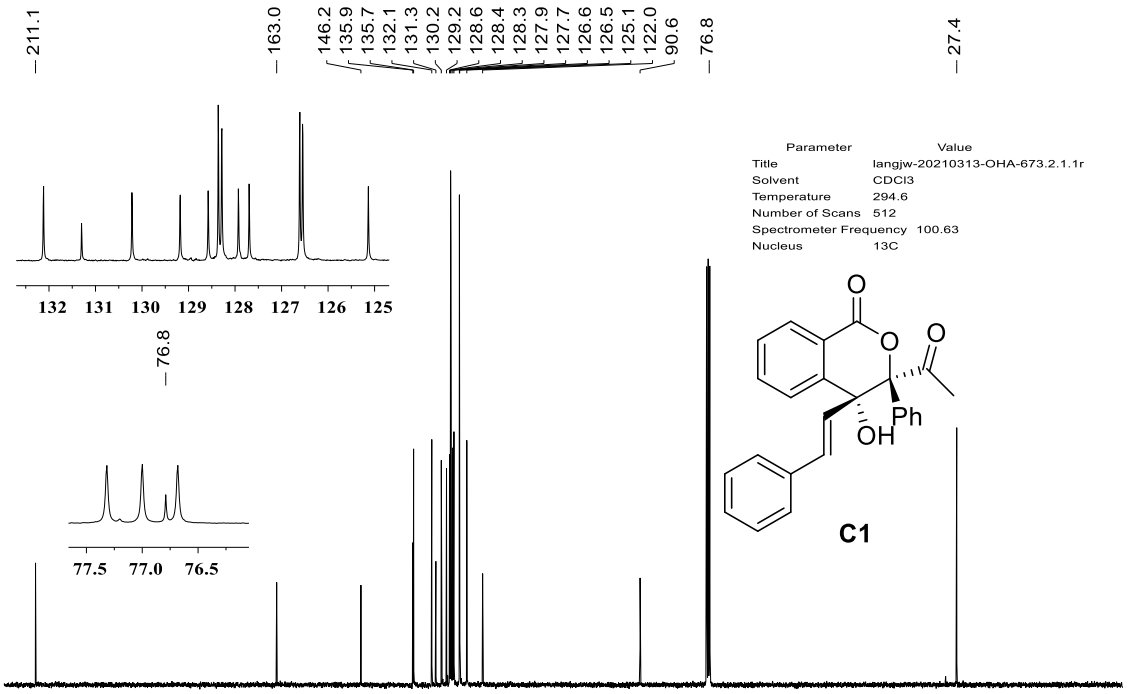
O
O
Ph
OH
C1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146.2
135.9
135.7
132.1
131.3
130.2
129.2
128.6
128.4
128.3
127.9
127.7
126.6
126.5
125.1
122.0
90.6
76.8
27.4
Parameter Value
Title langjw-20210313-OHA-673.2.1.1r
Solvent CDCl3
Temperature 294.6
Number of Scans 512
Spectrometer Frequency 100.63
Nucleus 13C
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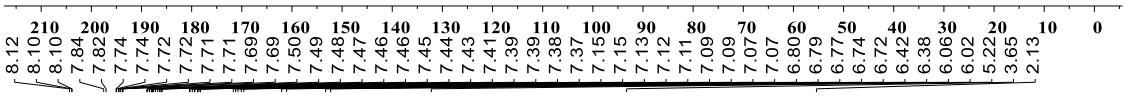
O
O
OH
Ph
C1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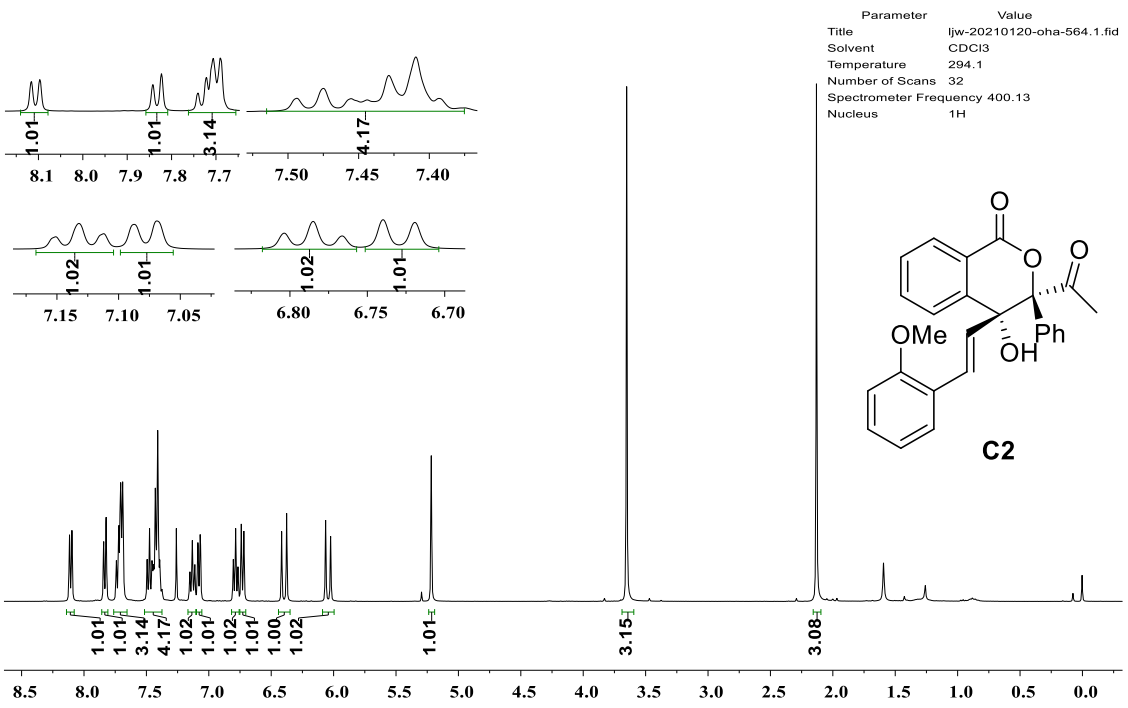
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Temperature 294.1
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
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O
O
OMe
OH
Ph
C2
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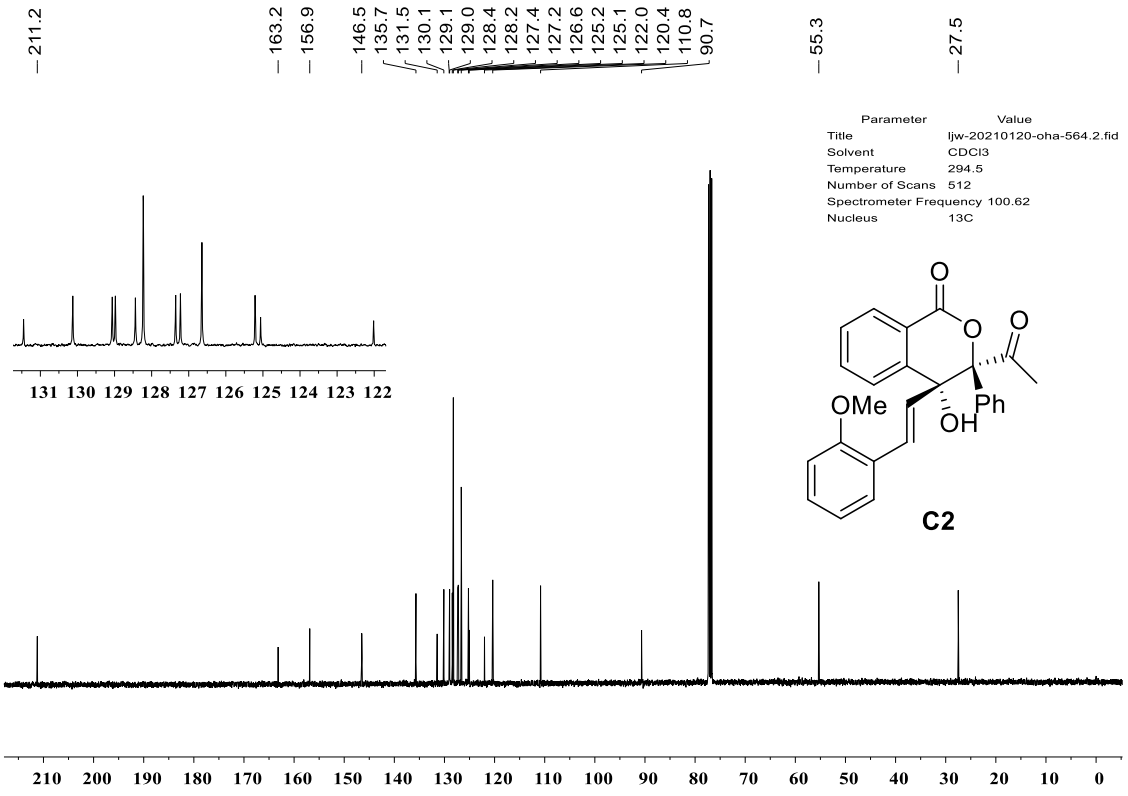
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131.5
130.1
129.1
129.0
128.4
128.2
127.4
127.2
126.6
125.2
125.1
122.0
120.4
110.8
90.7
55.3
27.5
Parameter Value
Title ljw-20210120-oha-564.2.fid
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Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
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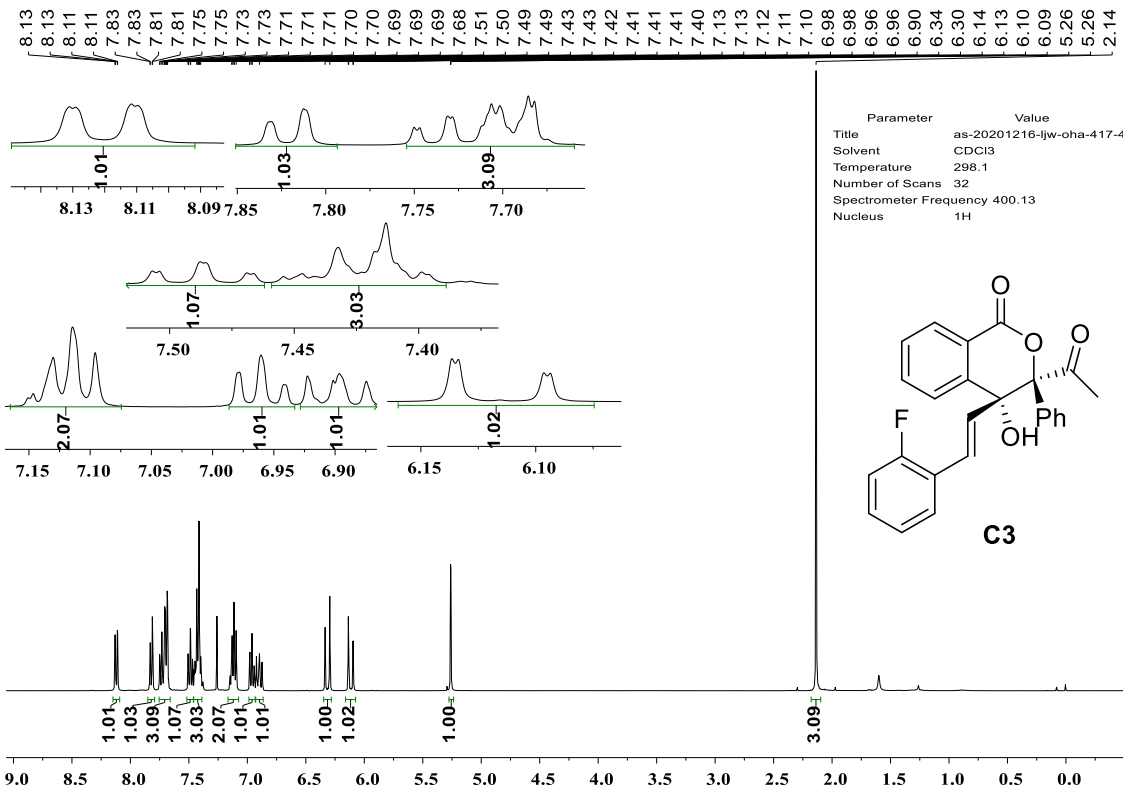
O
O
OMe
Ph
OH
C2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6.98
6.96
6.96
6.90
6.34
6.30
6.14
6.13
6.10
6.09
5.26
2.14
Parameter Value
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Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
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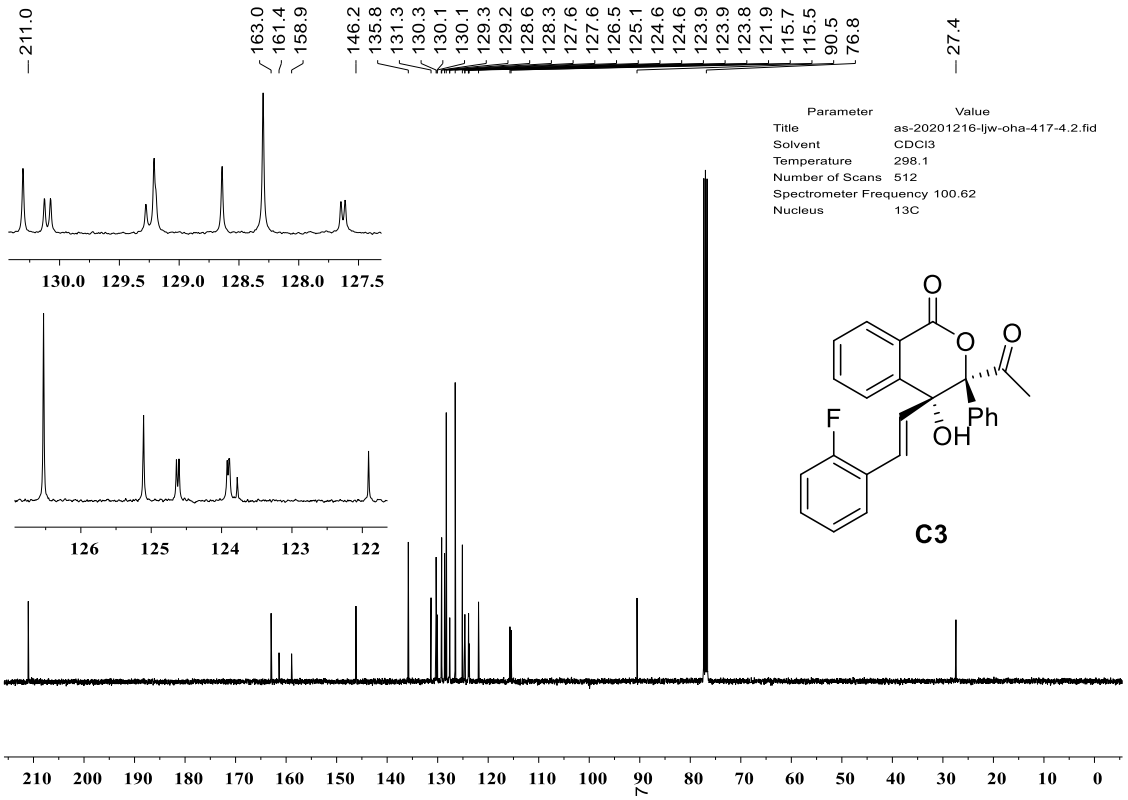
O
F
Ph
OH
C3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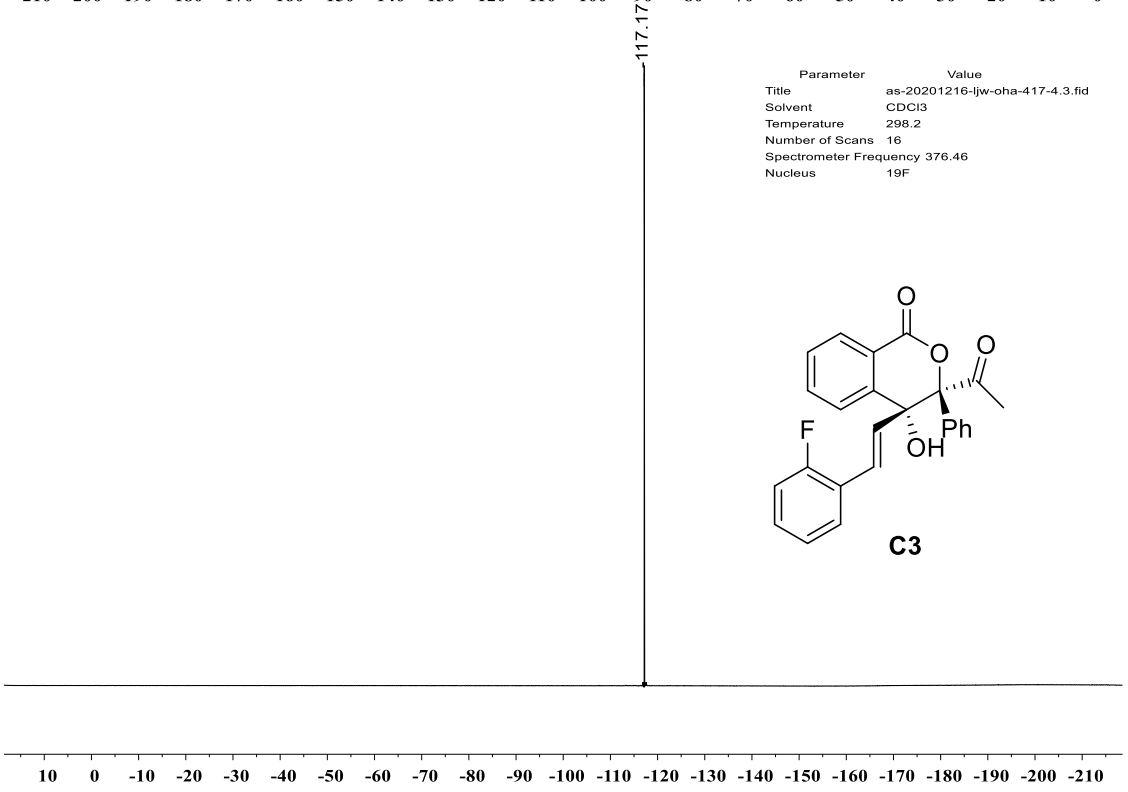
125.1
124.6
124.6
123.9
123.9
123.8
121.9
115.7
115.5
90.5
76.8
27.4
Parameter Value
Title as-20201216-ljw-oha-417-4.2.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
130.0 129.5 129.0 128.5 128.0 127.5
126 125 124 123 122
O
O
O
F
Ph
OH
C3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


117.17
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Temperature 298.2
Number of Scans 16
Spectrometer Frequency 376.46
Nucleus 19F
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O
O
F
Ph
OH
C3
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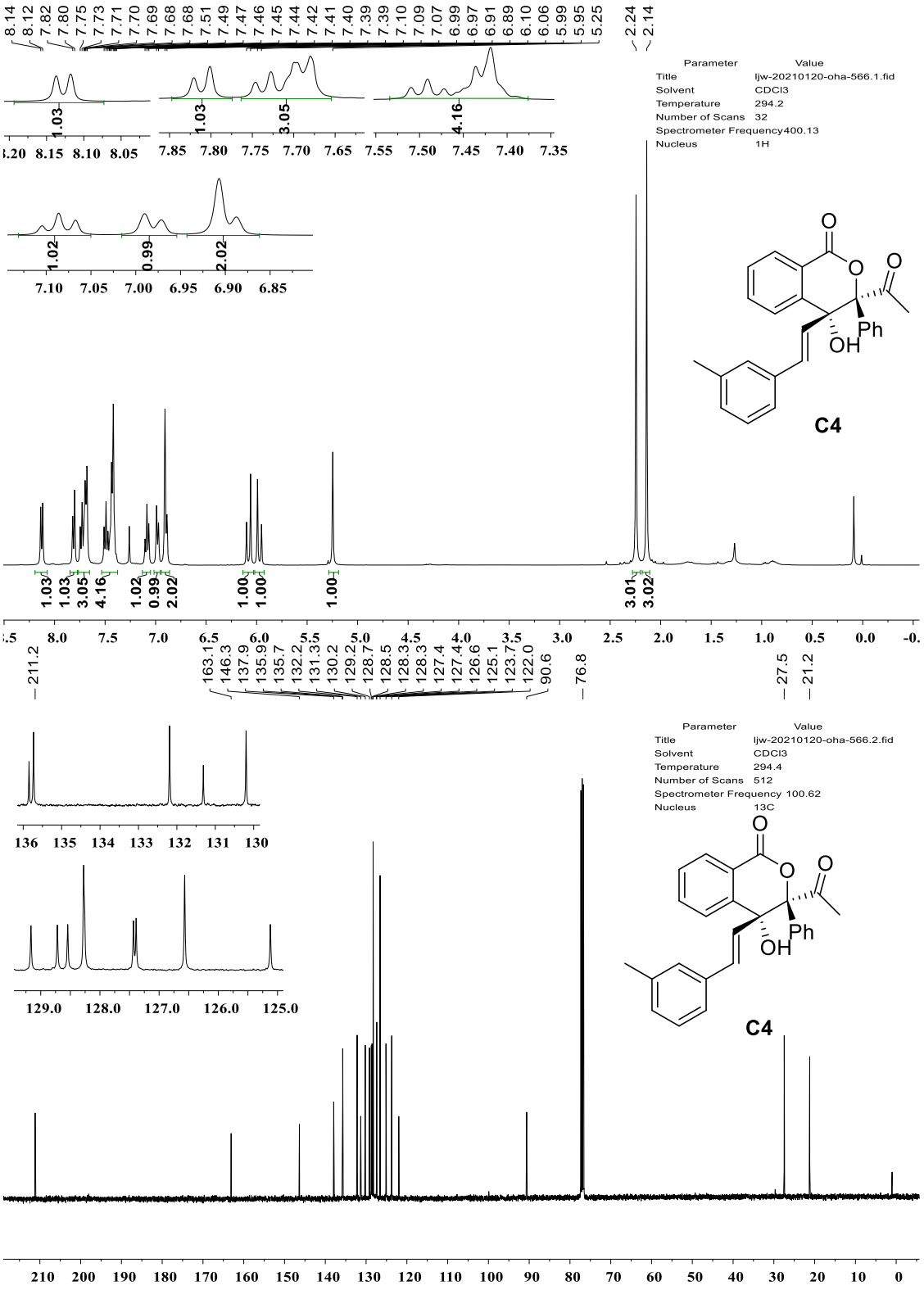
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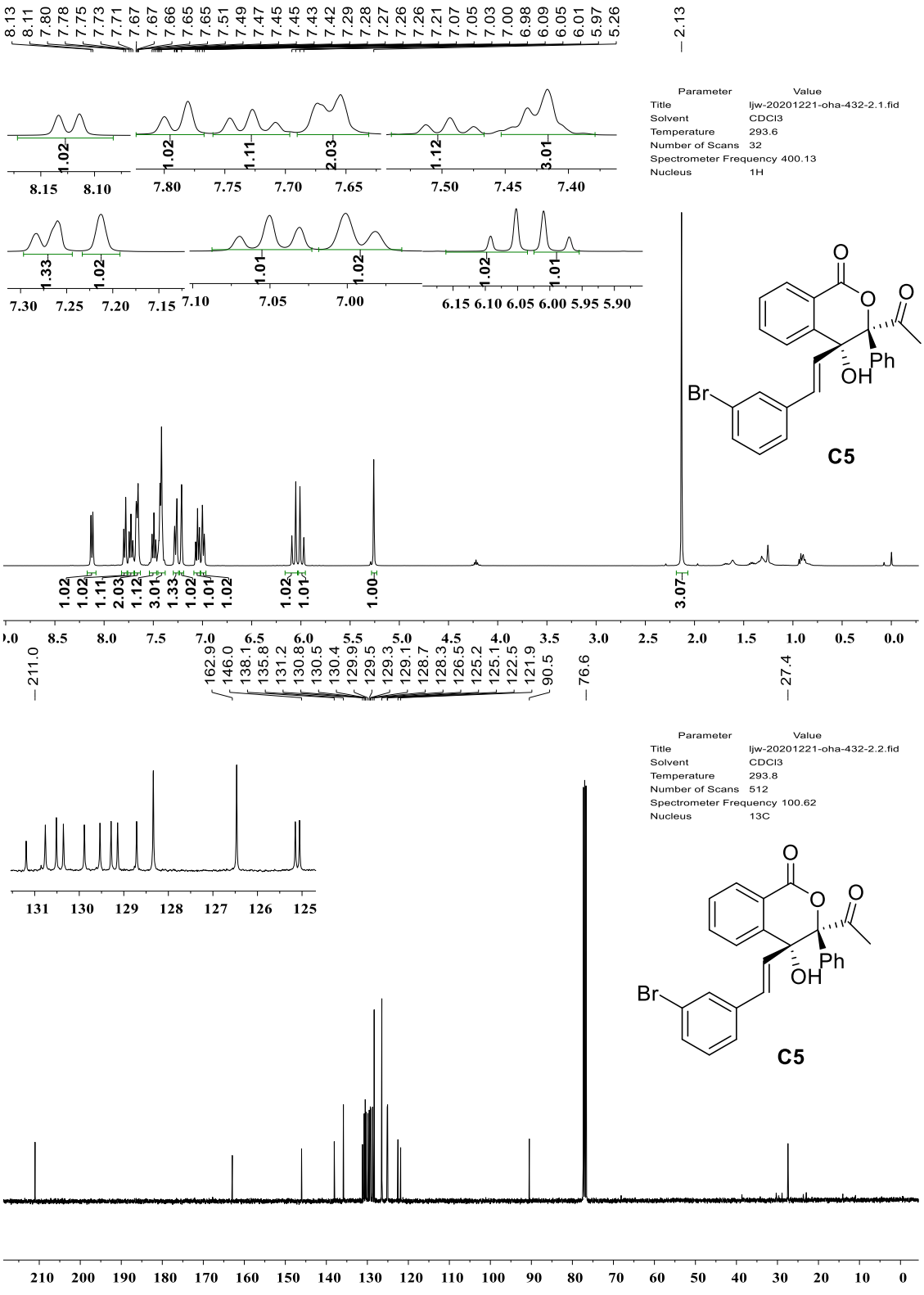
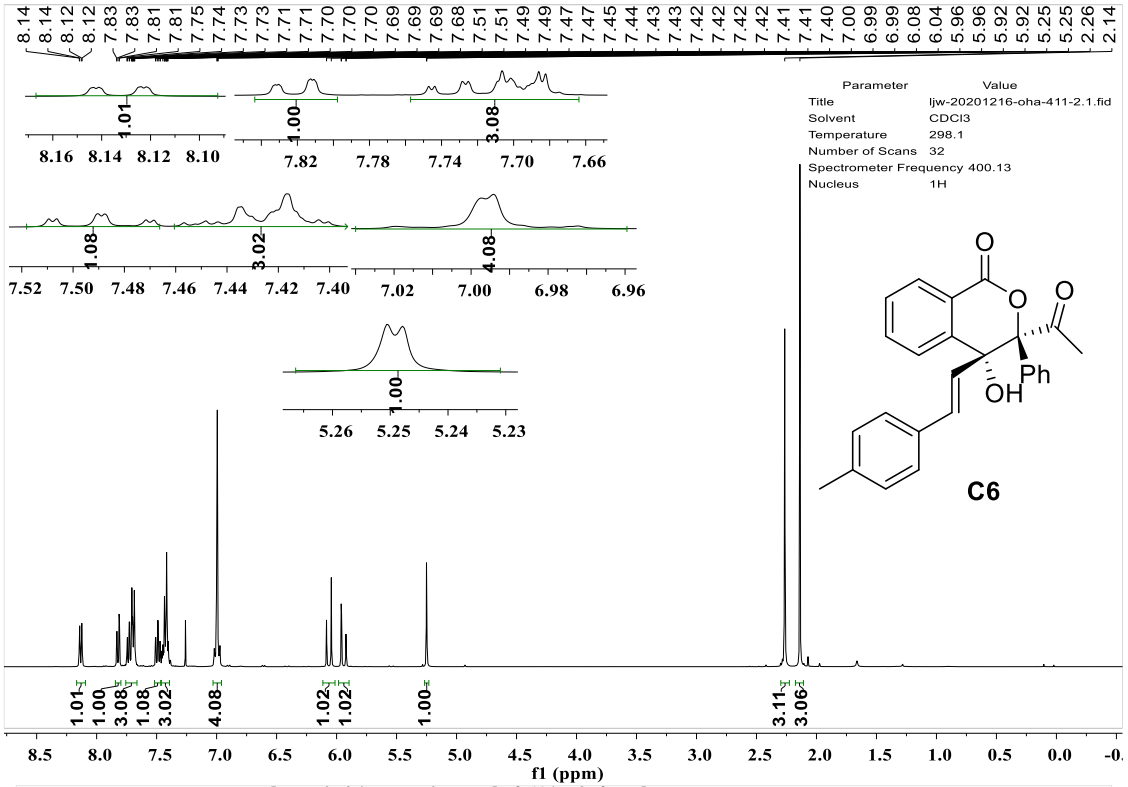
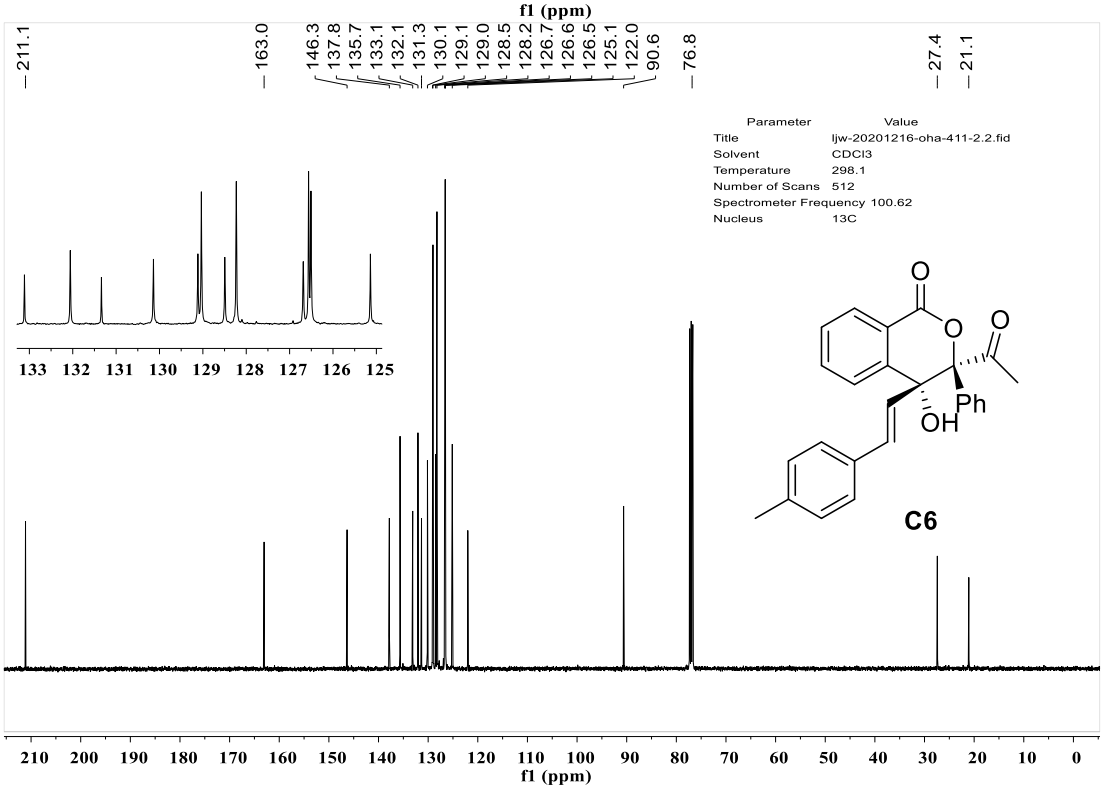
Temperature 294.2
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C4
Parameter Value
Title ljw-20210120-oha-566.2.fid
Solvent CDCl3
Temperature 294.4
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
C4


Parameter Value
Title ljw-20201221-oha-432-2.1.fid
Solvent CDCl3
Temperature 293.6
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C5
Parameter Value
Title ljw-20201221-oha-432-2.2.fid
Solvent CDCl3
Temperature 293.8
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
C5


Parameter Value
Title ljw-20201216-oha-411-2.1.fid
Solvent CDCl3
Temperature 298.1
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C6
f1 (ppm)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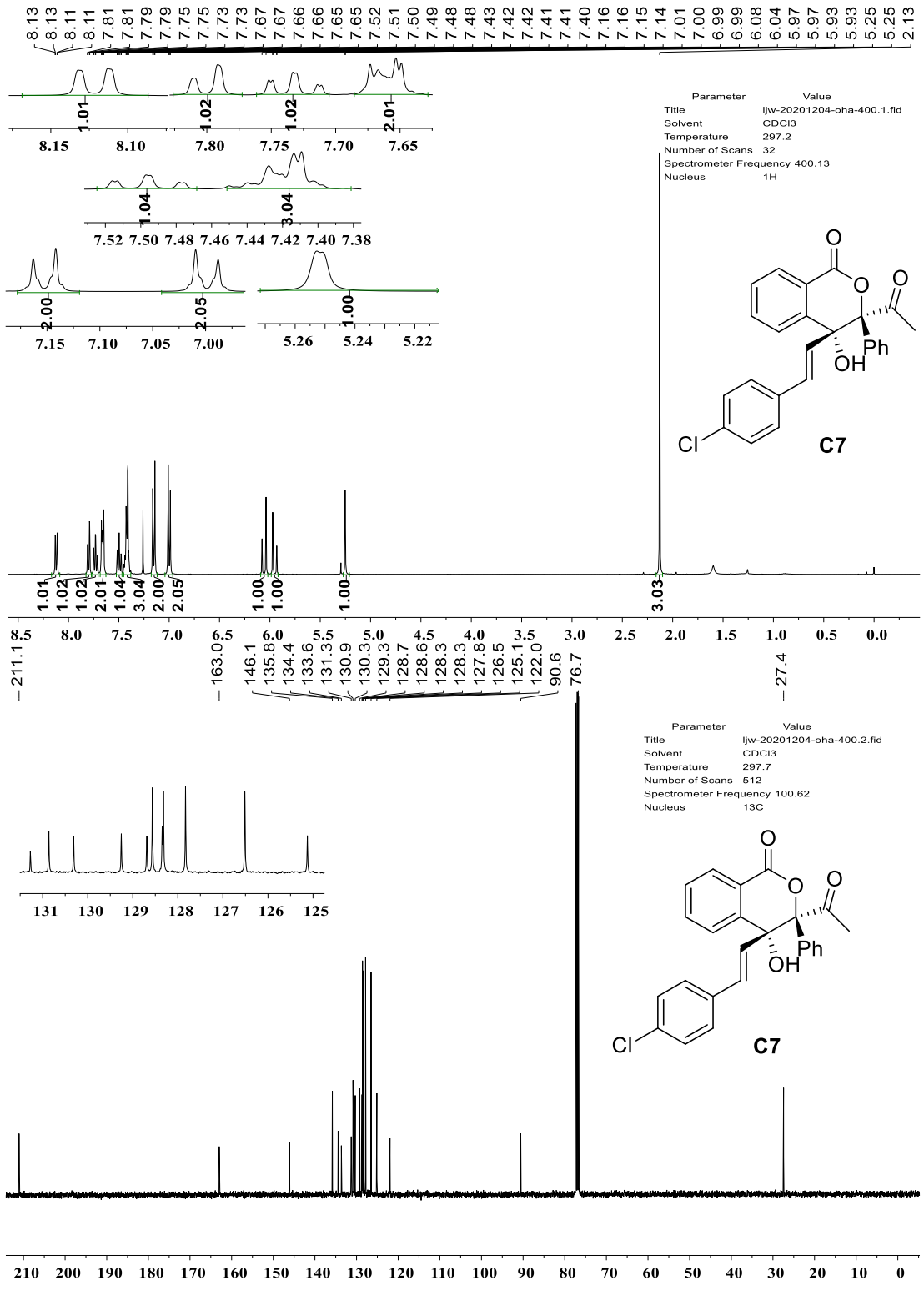
Spectrometer Frequency 100.62
Nucleus 13C
C6
f1 (ppm)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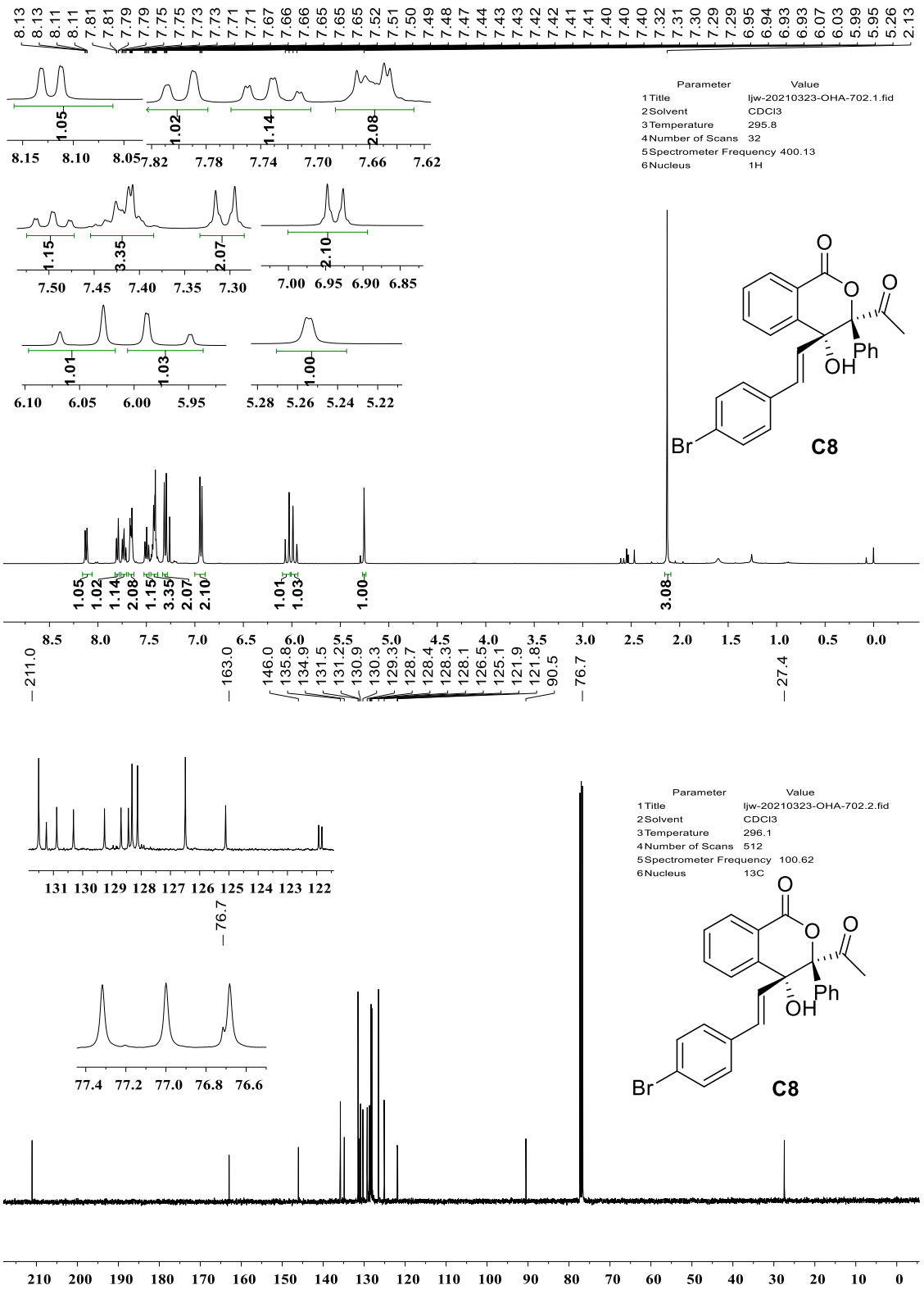
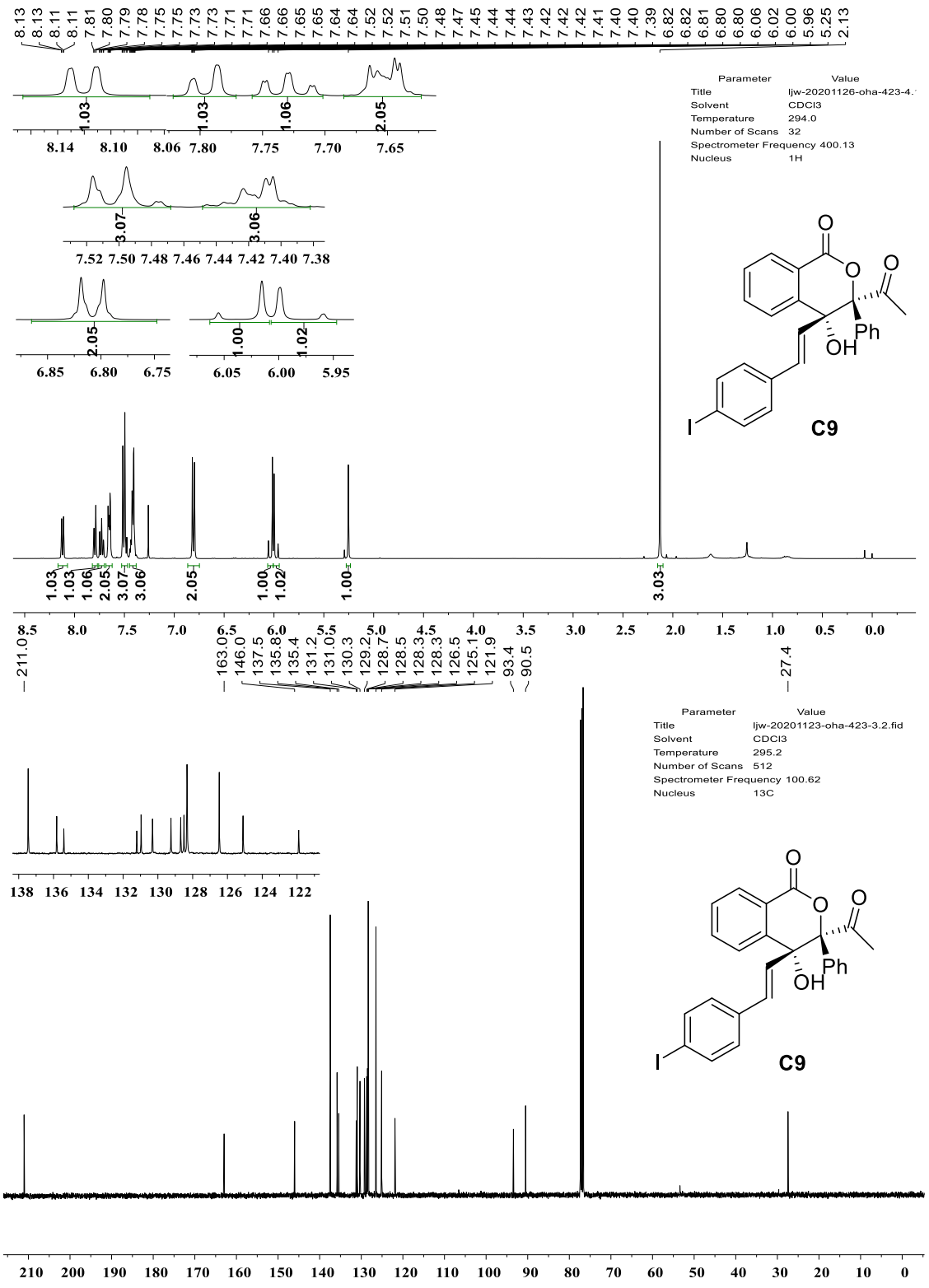
Title ljw-20201204-oha-400.1.fid
Solvent CDCl3
Temperature 297.2
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C7
Parameter Value
Title ljw-20201204-oha-400.2.fid
Solvent CDCl3
Temperature 297.7
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
C7


Parameter Value
1 Title ljw-20210323-OHA-702.1.fid
2 Solvent CDCl3
3 Temperature 295.8
4 Number of Scans 32
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Br
OH
Ph
C8
Parameter Value
1 Title ljw-20210323-OHA-702.2.fid
2 Solvent CDCl3
3 Temperature 296.1
4 Number of Scans 512
5 Spectrometer Frequency 100.62
6 Nucleus 13C
Br
OH
Ph
C8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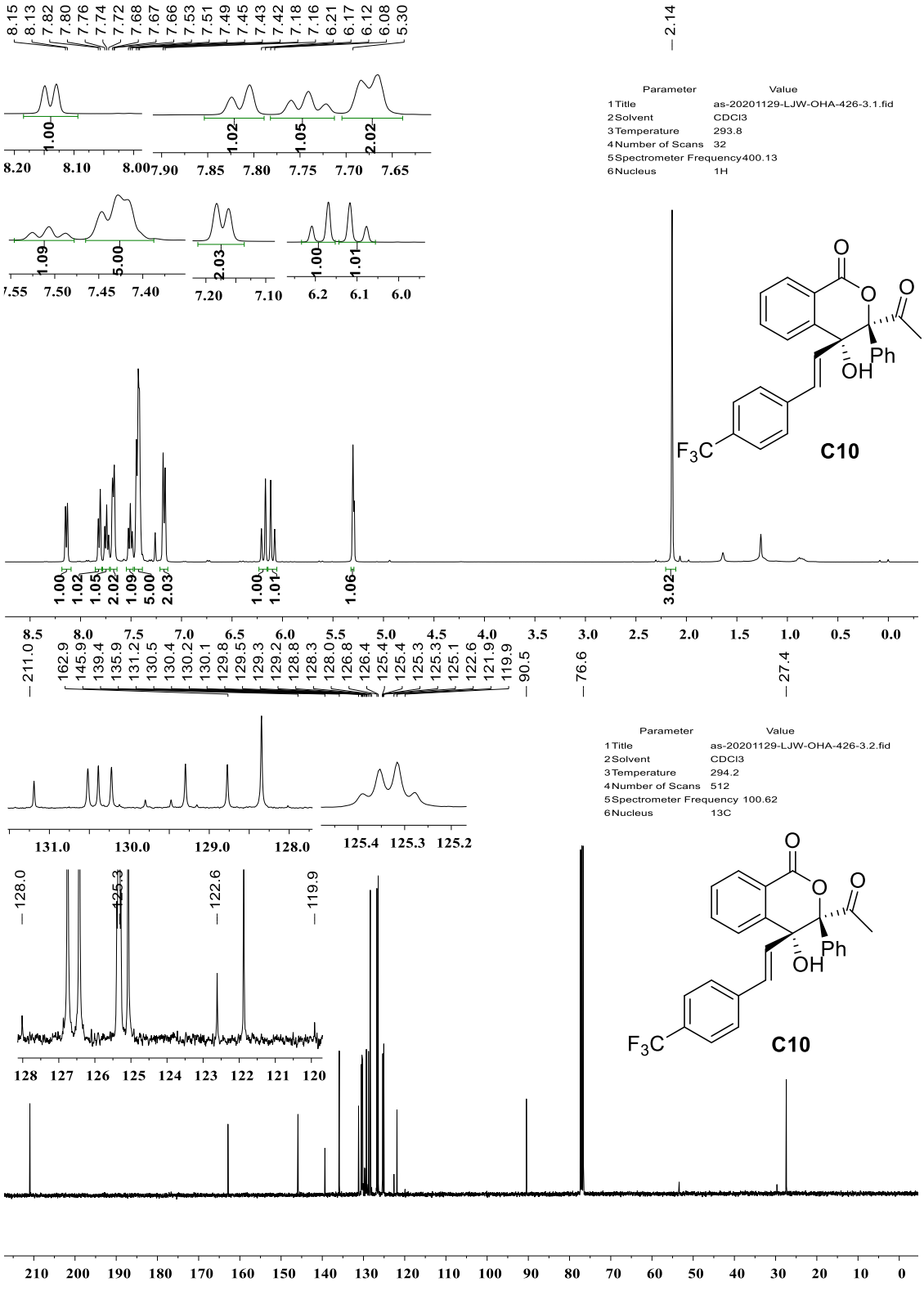
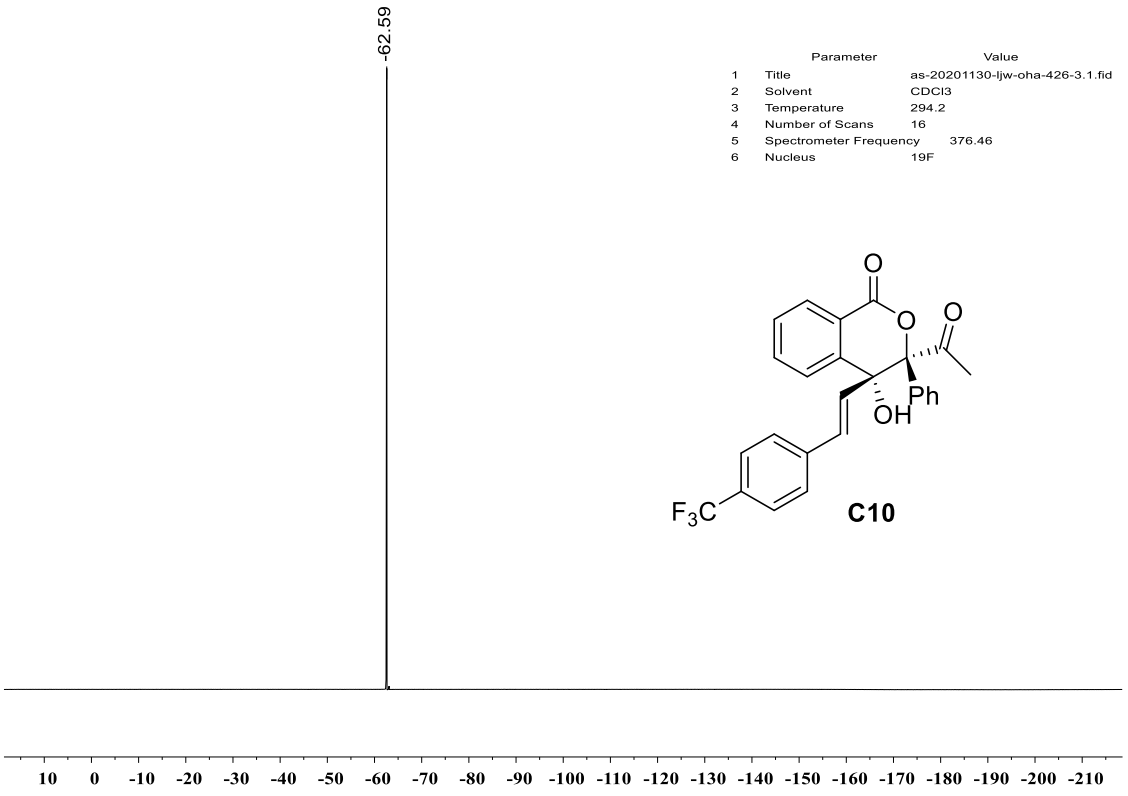
Title ljw-20201126-oha-423-4.
Solvent CDCl3
Temperature 294.0
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C9
Parameter Value
Title ljw-20201123-oha-423-3.2.fid
Solvent CDCl3
Temperature 295.2
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
C9


Parameter Value
1 Title as-20201129-LJW-OHA-426-3.1.fid
2 Solvent CDCl3
3 Temperature 293.8
4 Number of Scans 32
5 Spectrometer Frequency 400.13
6 Nucleus 1H
C10
Parameter Value
1 Title as-20201129-LJW-OHA-426-3.2.fid
2 Solvent CDCl3
3 Temperature 294.2
4 Number of Scans 512
5 Spectrometer Frequency 100.62
6 Nucleus 13C
C10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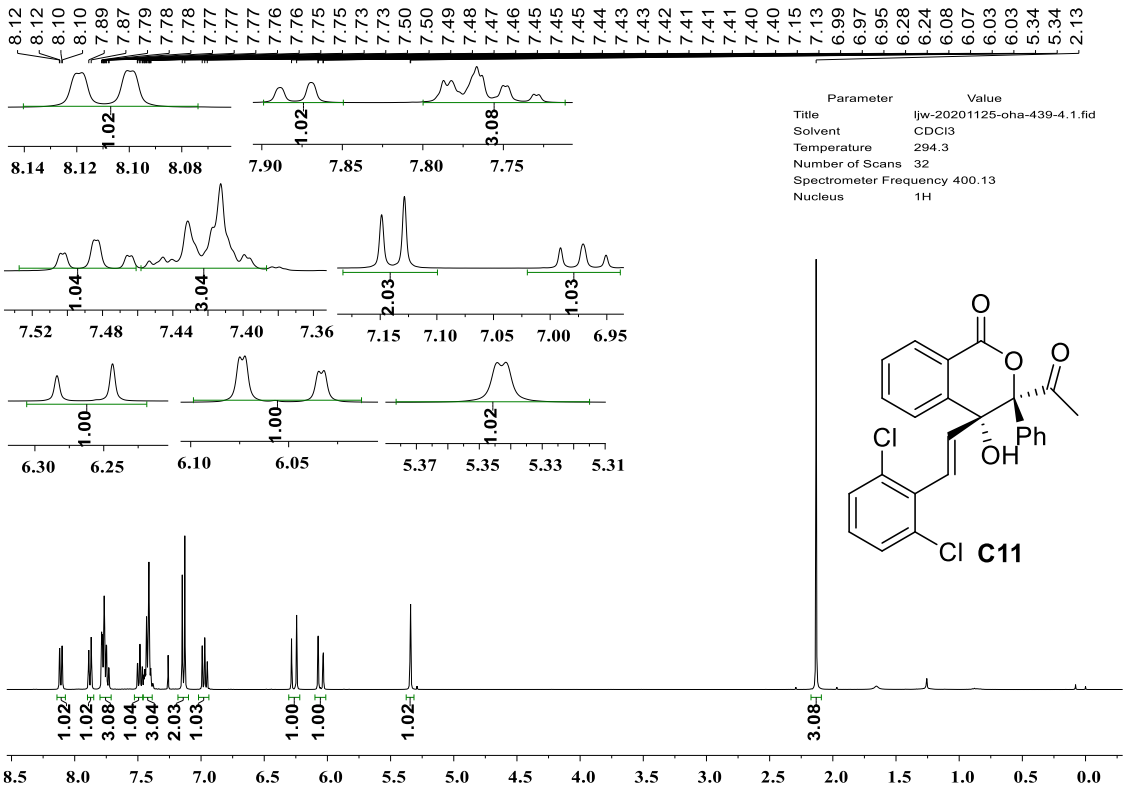
1 Title as-20201130-ljw-oha-426-3.1.fid
2 Solvent CDCl3
3 Temperature 294.2
4 Number of Scans 16
5 Spectrometer Frequency 376.46
6 Nucleus 19F
O
O
O
Ph
OH
F3C
C10
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210


Parameter Value
Title ljw-20201125-oha-439-4.1.fid
Solvent CDCl3
Temperature 294.3
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
O
O
O
Cl
Ph
OH
Cl C11
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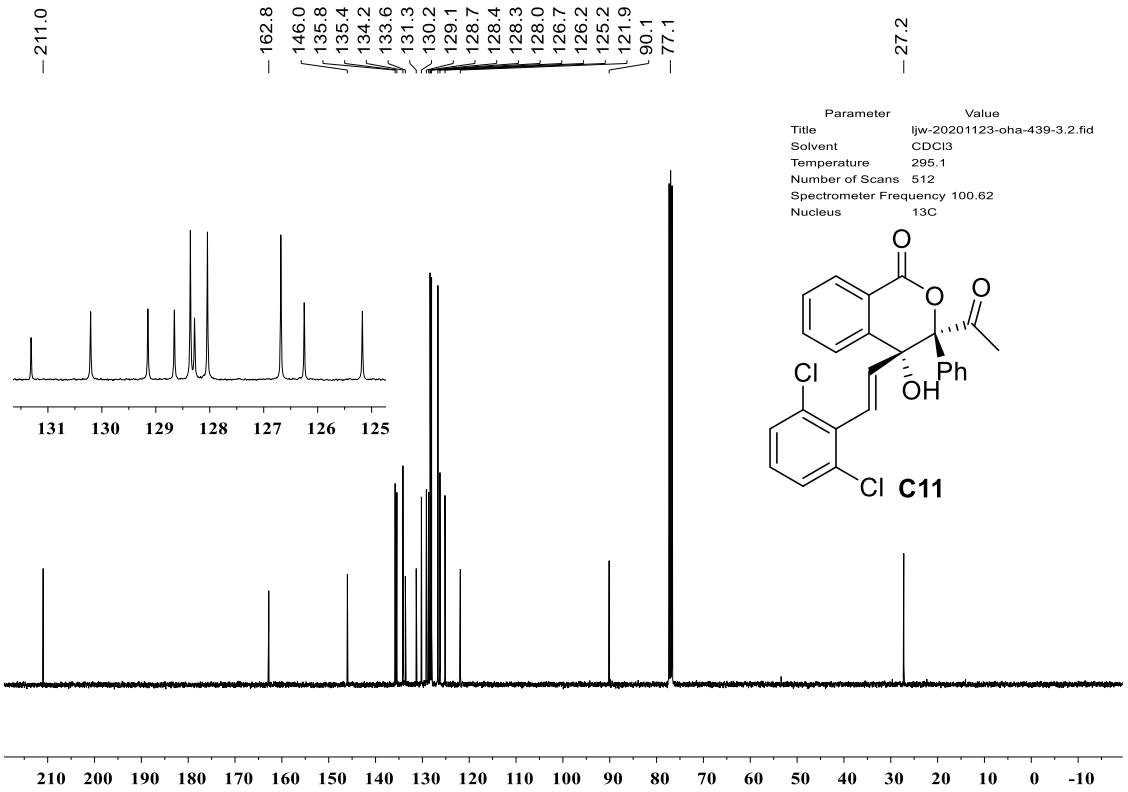
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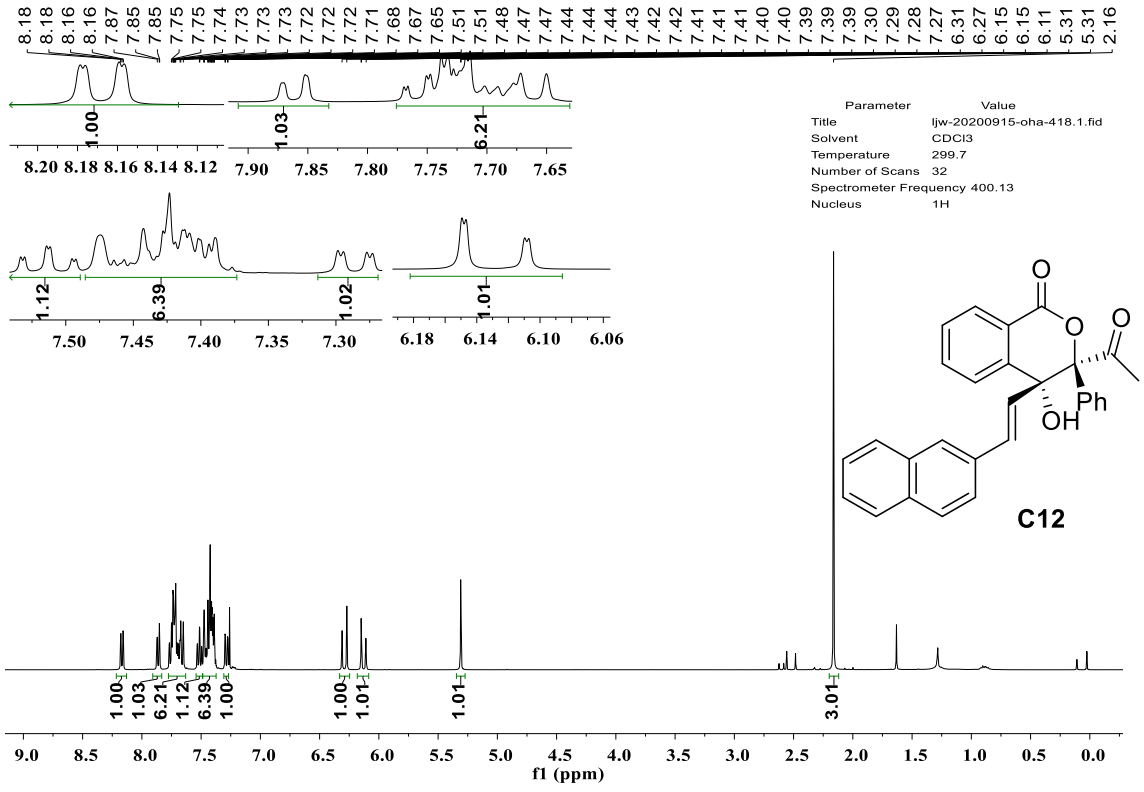
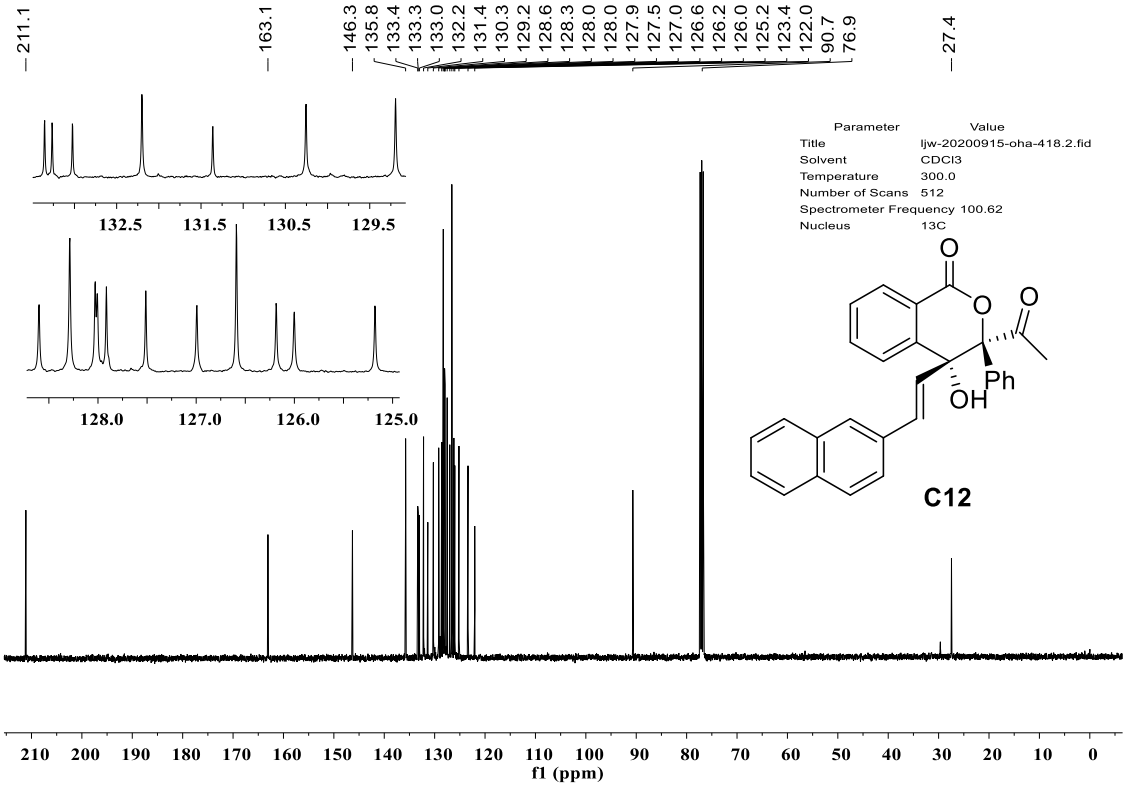
134.2
133.6
131.3
130.2
129.1
128.7
128.4
128.3
128.0
126.7
126.2
125.2
121.9
90.1
77.1
27.2
Parameter Value
Title ljw-20201123-oha-439-3.2.fid
Solvent CDCl3
Temperature 295.1
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
C11


Parameter Value
Title ljw-20200915-oha-418.1.fid
Solvent CDCl3
Temperature 299.7
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C12
f1 (ppm)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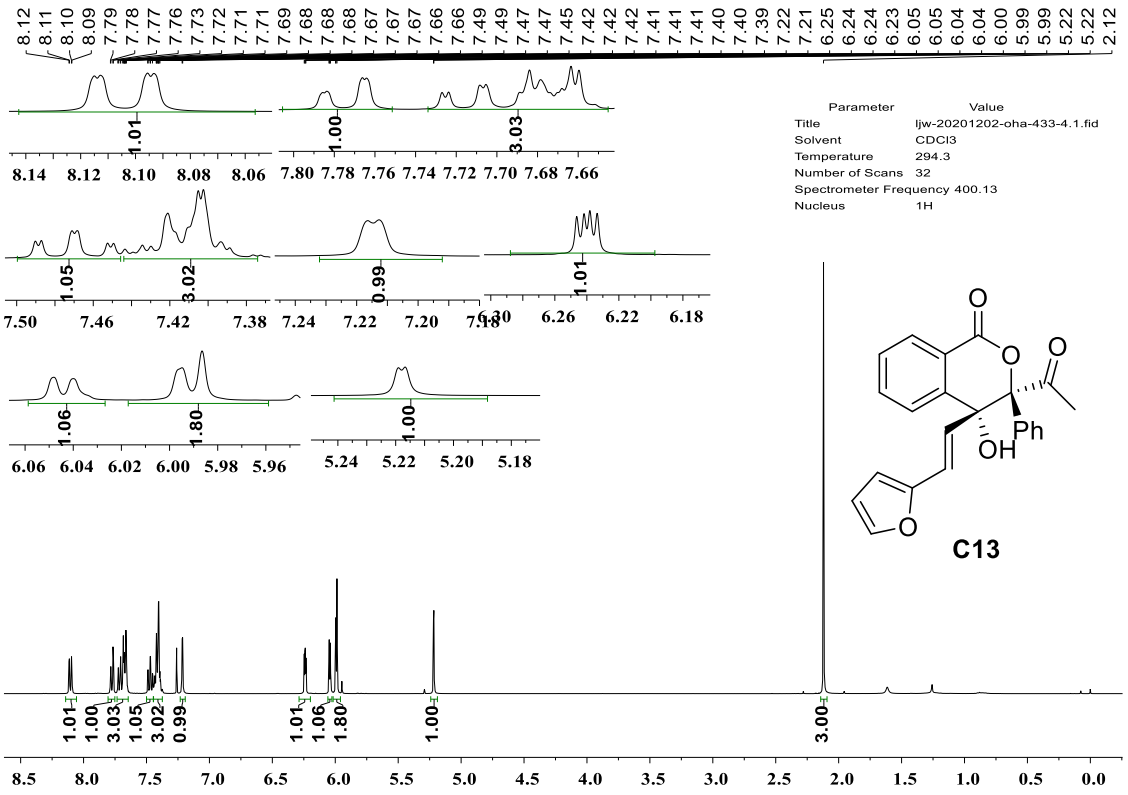
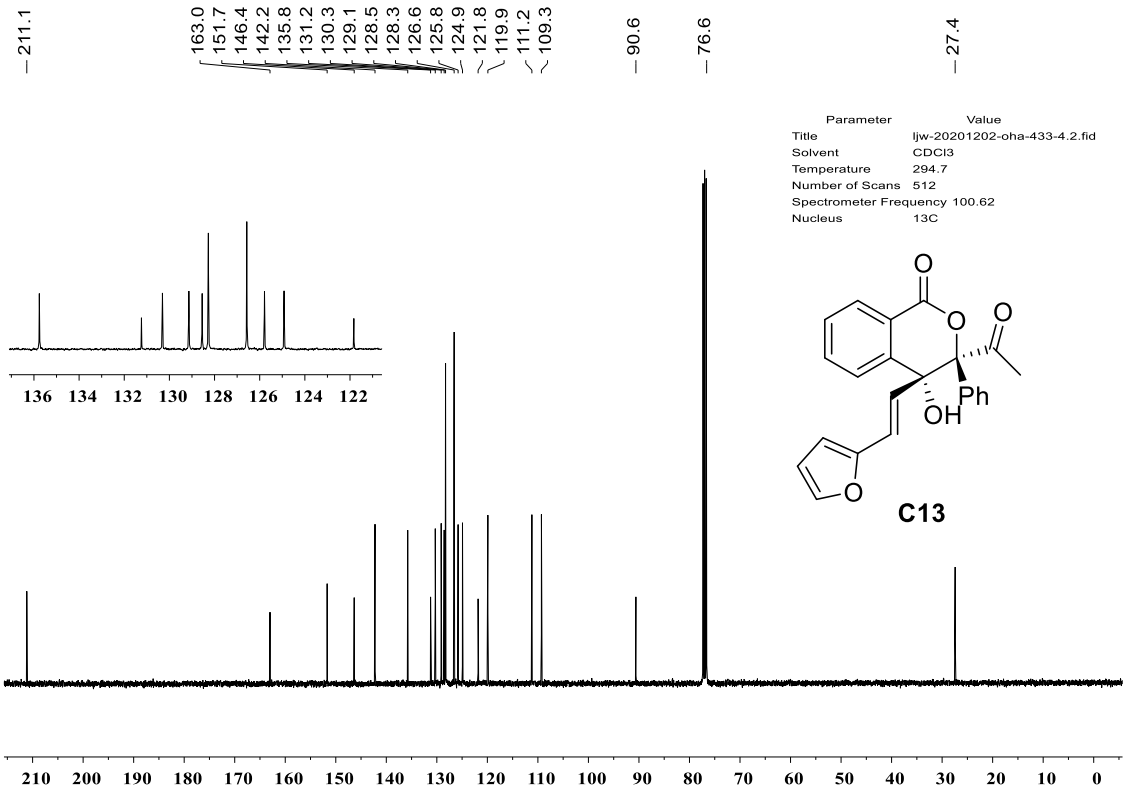
135.8
133.4
133.3
133.0
132.2
131.4
130.3
129.2
128.6
128.3
128.0
128.0
127.9
127.5
127.0
126.6
126.2
126.0
125.2
123.4
122.0
90.7
76.9
27.4
Parameter Value
Title ljw-20200915-oha-418.2.fid
Solvent CDCl3
Temperature 300.0
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
Ph
OH
C12
f1 (ppm)


Parameter Value
Title ljw-20201202-oha-433-4.1.fid
Solvent CDCl3
Temperature 294.3
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
O
O
O
Ph
OH
O
C13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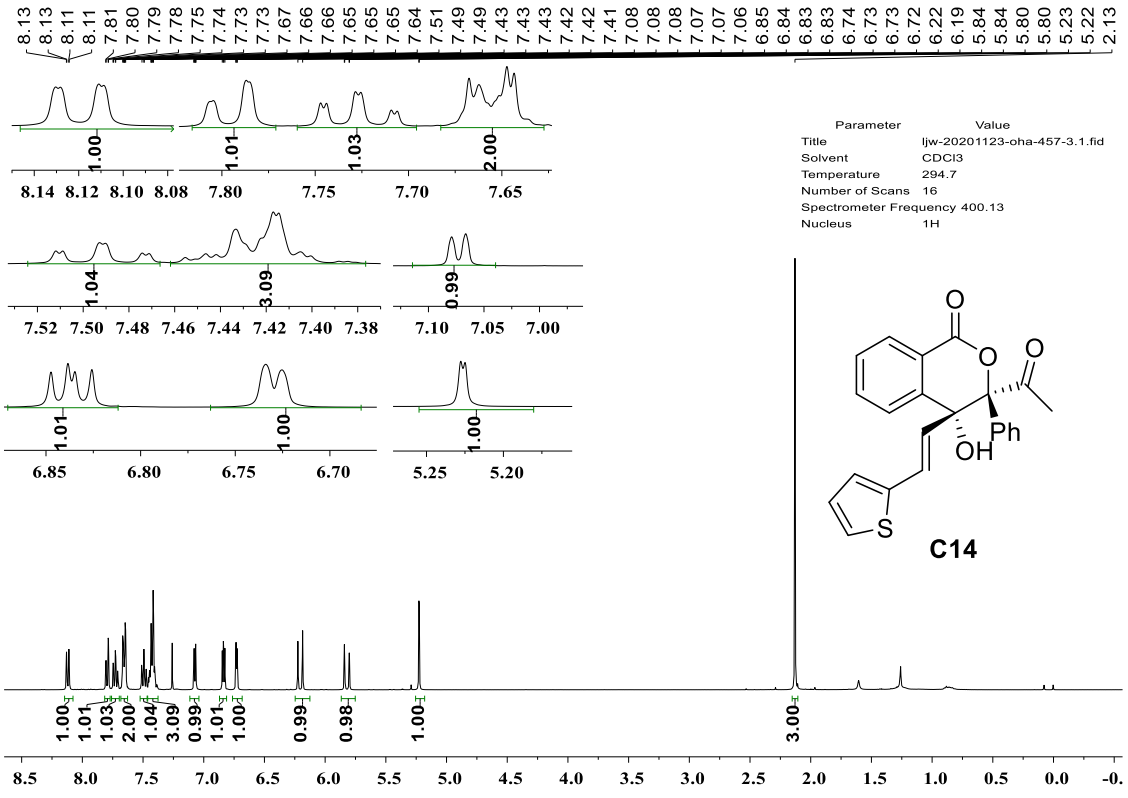
131.2
130.3
129.1
128.5
128.3
126.6
125.8
124.9
121.8
119.9
111.2
109.3
90.6
76.6
27.4
Parameter Value
Title ljw-20201202-oha-433-4.2.fid
Solvent CDCl3
Temperature 294.7
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
C13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7.66
7.65
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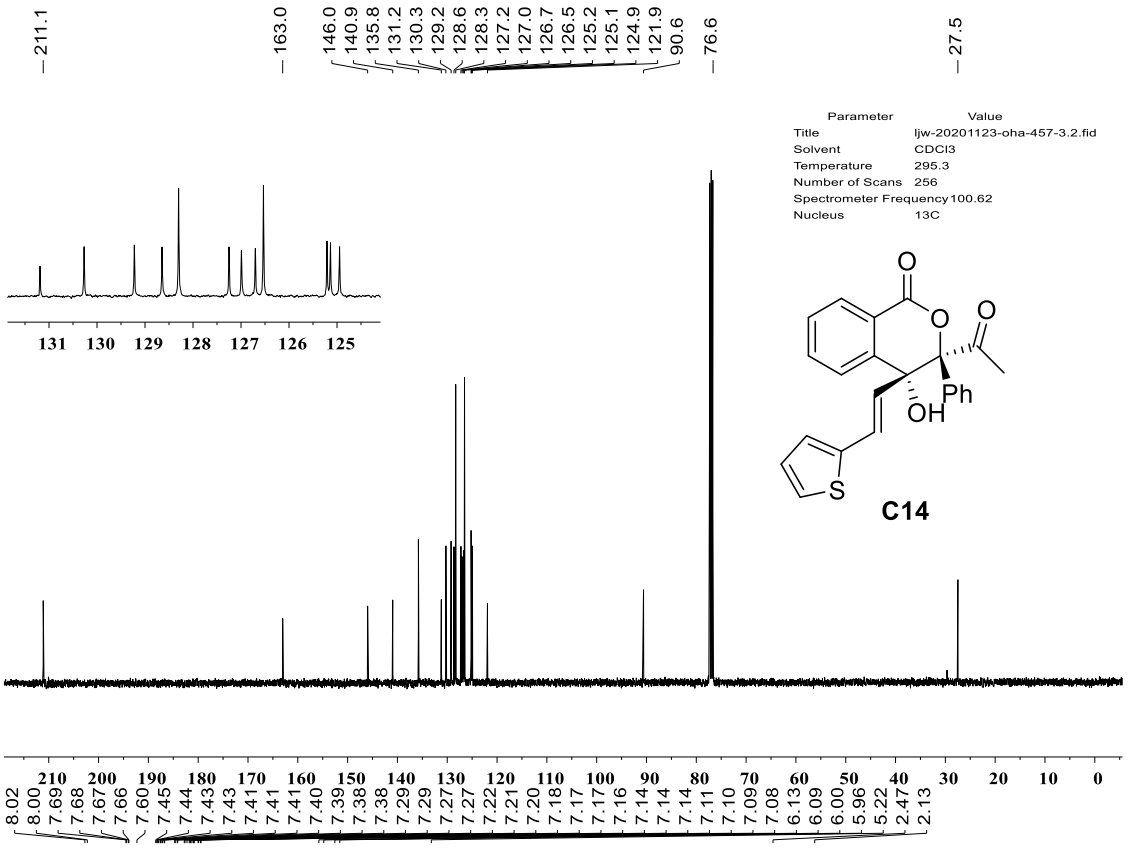
7.64
7.51
7.49
7.49
7.43
7.43
7.43
7.42
7.42
7.41
7.08
7.08
7.08
7.07
7.07
7.06
6.85
6.84
6.83
6.83
6.74
6.73
6.73
6.72
6.22
6.19
5.84
5.84
5.80
5.80
5.23
5.22
2.13
Parameter Value
Title ljw-20201123-oha-457-3.1.fid
Solvent CDCl3
Temperature 294.7
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
C14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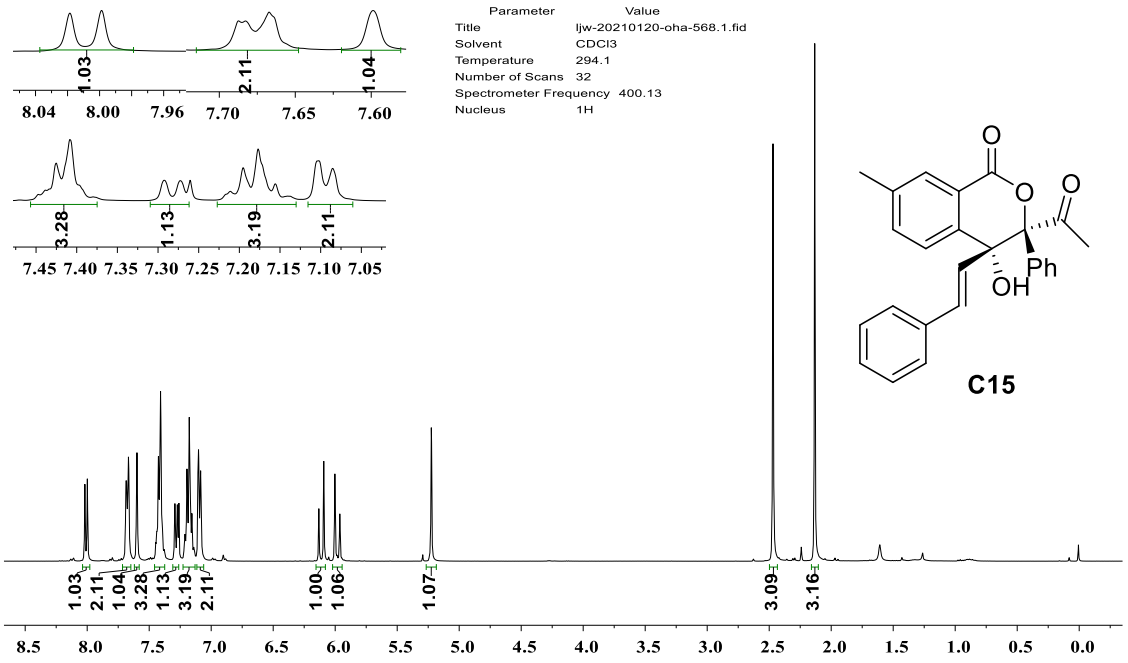
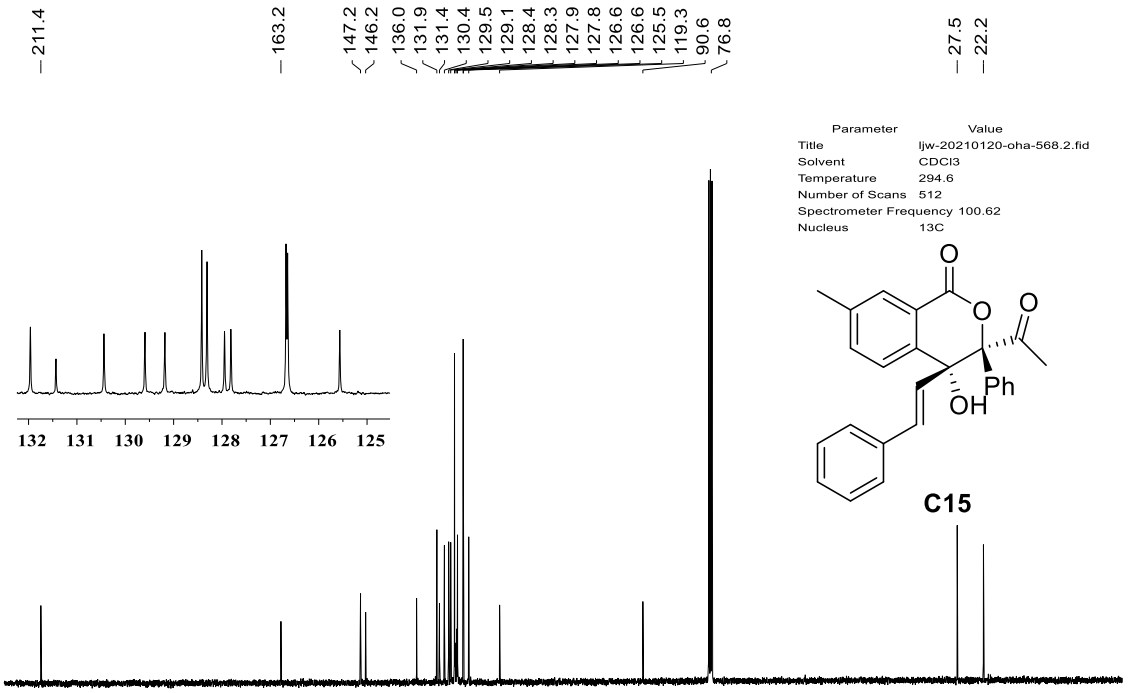
146.0
140.9
135.8
131.2
130.3
129.2
128.6
128.3
127.2
127.0
126.7
126.5
125.2
125.1
124.9
121.9
90.6
76.6
27.5
Parameter Value
Title ljw-20201123-oha-457-3.2.fid
Solvent CDCl3
Temperature 295.3
Number of Scans 256
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
Ph
OH
S
C14


Parameter Value
Title ljw-20210120-oha-568.1.fid
Solvent CDCl3
Temperature 294.1
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
O
O
O
Ph
OH
C15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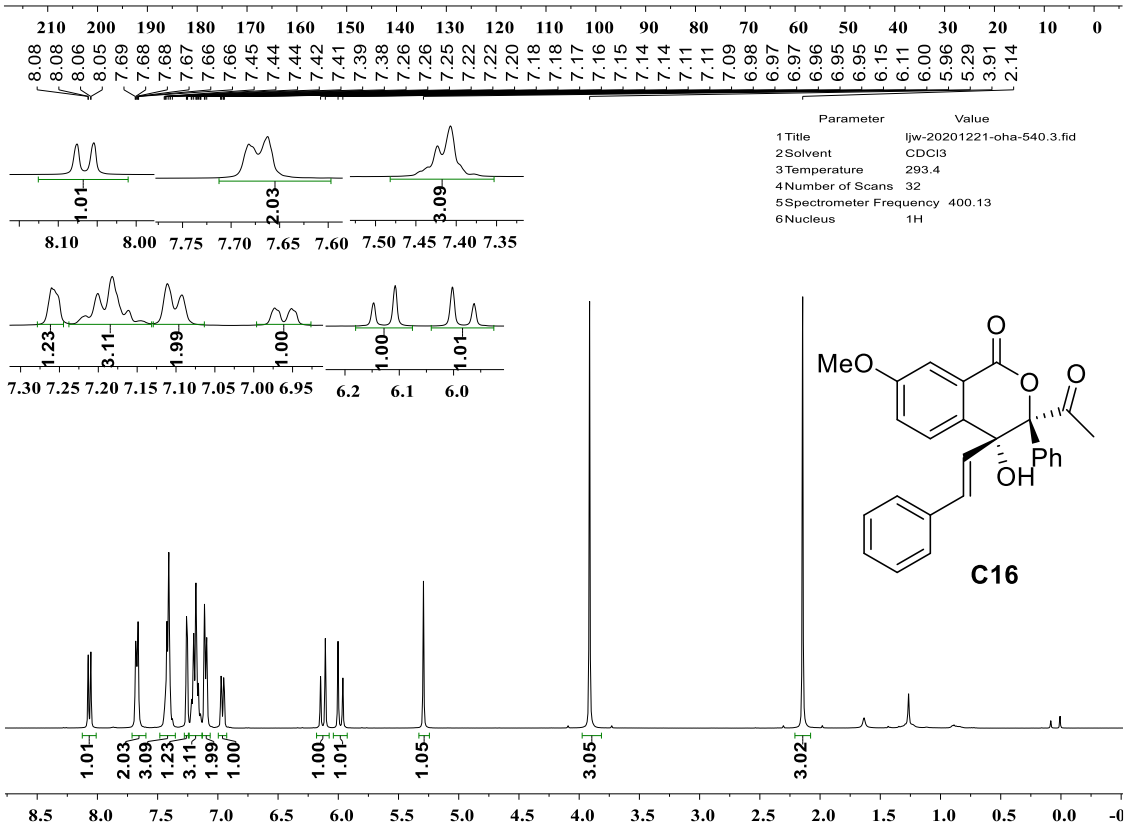
147.2
146.2
136.0
131.9
131.4
130.4
129.5
129.1
128.4
128.3
127.9
127.8
126.6
126.6
125.5
119.3
90.6
76.8
27.5
22.2
Parameter Value
Title ljw-20210120-oha-568.2.fid
Solvent CDCl3
Temperature 294.6
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
Ph
OH
C15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7.66
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7.42
7.41
7.39
7.38
7.26
7.26
7.25
7.22
7.22
7.20
7.18
7.18
7.17
7.16
7.15
7.14
7.14
7.11
7.11
7.09
6.98
6.97
6.97
6.96
6.95
6.95
6.15
6.11
6.00
5.96
5.29
3.91
2.14
Parameter Value
1 Title ljw-20201221-oha-540.3.fid
2 Solvent CDCl3
3 Temperature 293.4
4 Number of Scans 32
5 Spectrometer Frequency 400.13
6 Nucleus 1H
MeO
O
O
O
Ph
OH
C16
1.01
2.03
3.09
1.23
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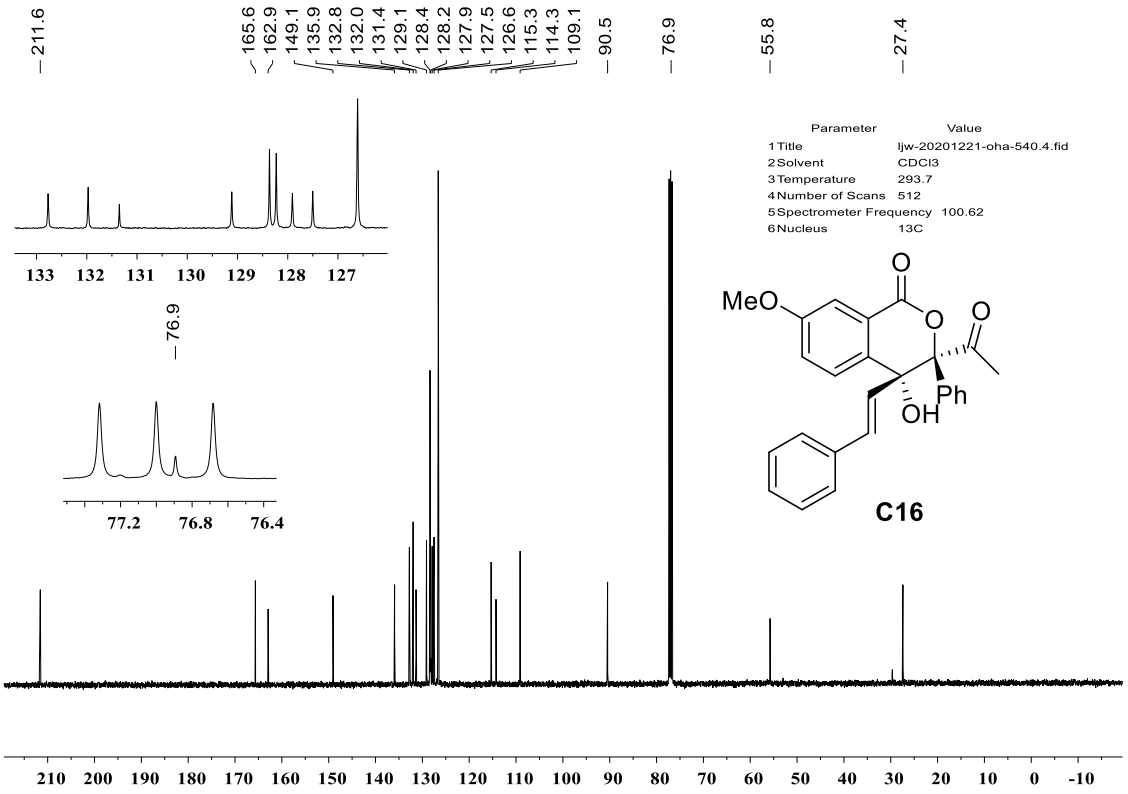
1.00
1.00
1.01
1.05
3.05
3.02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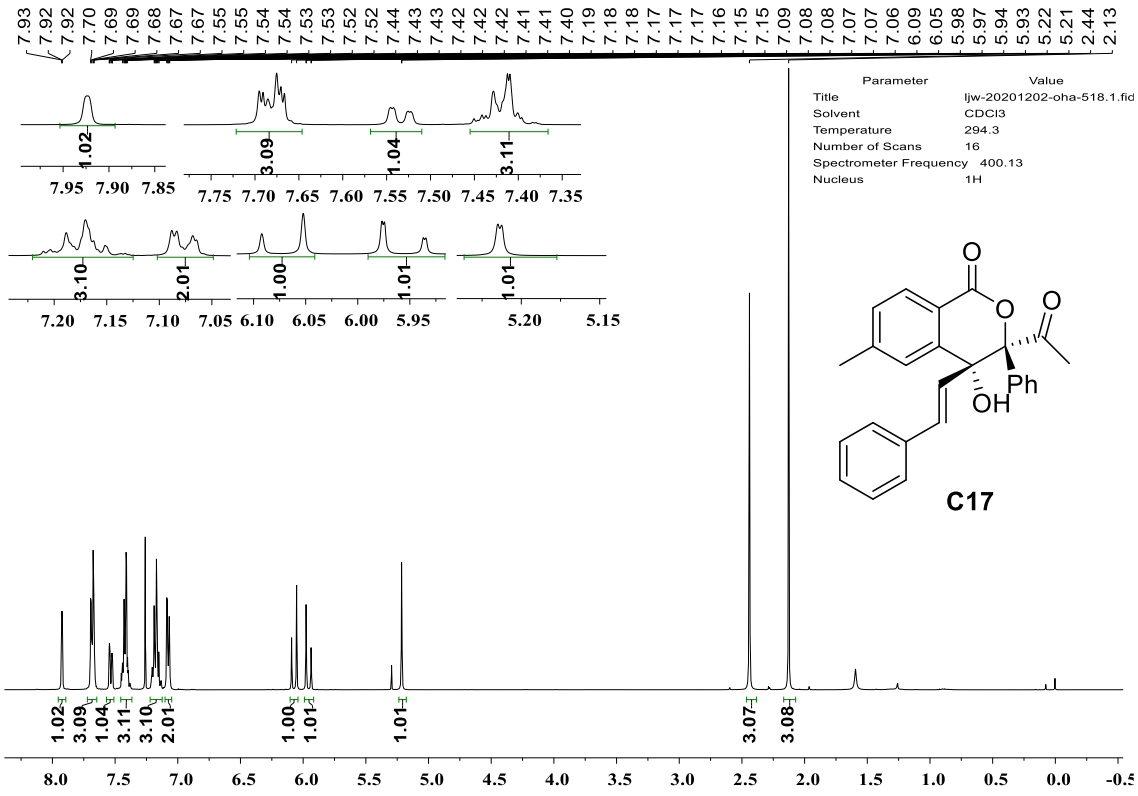
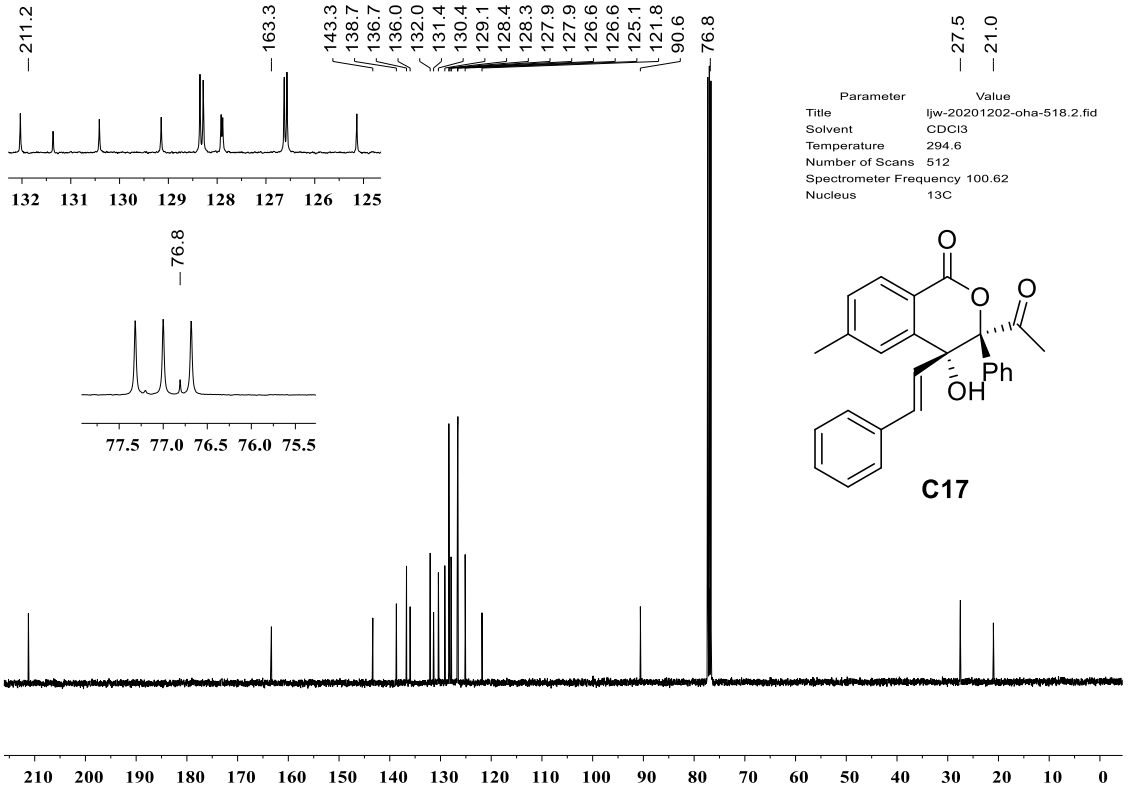
132.8
132.0
131.4
129.1
128.4
128.2
127.9
127.5
126.6
115.3
114.3
109.1
90.5
76.9
55.8
27.4
Parameter Value
1 Title ljw-20201221-oha-540.4.fid
2 Solvent CDCl3
3 Temperature 293.7
4 Number of Scans 512
5 Spectrometer Frequency 100.62
6 Nucleus 13C
MeO
O
Ph
OH
C16
133 132 131 130 129 128 127
77.2 76.8 76.4
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10


Parameter Value
Title ljw-20201202-oha-518.1.fid
Solvent CDCl3
Temperature 294.3
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
O
Ph
OH
C17
1.02 3.09 1.04 3.11 3.10 2.01 1.00 1.01 1.01 3.07 3.08
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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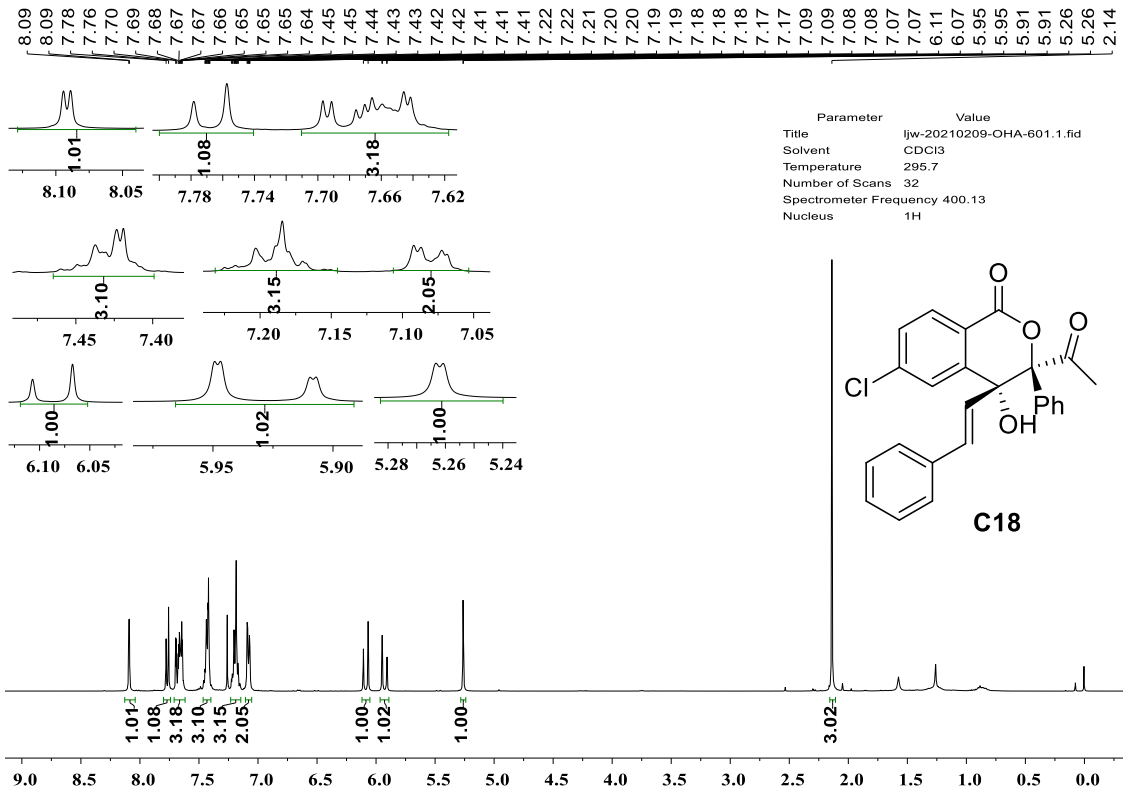
143.3
138.7
136.7
136.0
132.0
131.4
130.4
129.1
128.4
128.3
127.9
127.9
126.6
126.6
125.1
121.8
90.6
76.8
27.5
21.0
Parameter Value
Title ljw-20201202-oha-518.2.fid
Solvent CDCl3
Temperature 294.6
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
Ph
OH
C17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7.64
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7.44
7.43
7.42
7.41
7.22
7.21
7.20
7.19
7.18
7.17
7.09
7.08
7.07
6.11
6.07
5.95
5.91
5.26
5.14
Parameter Value
Title ljw-20210209-OHA-601.1.fid
Solvent CDCl3
Temperature 295.7
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
Cl
O
O
Ph
OH
C18
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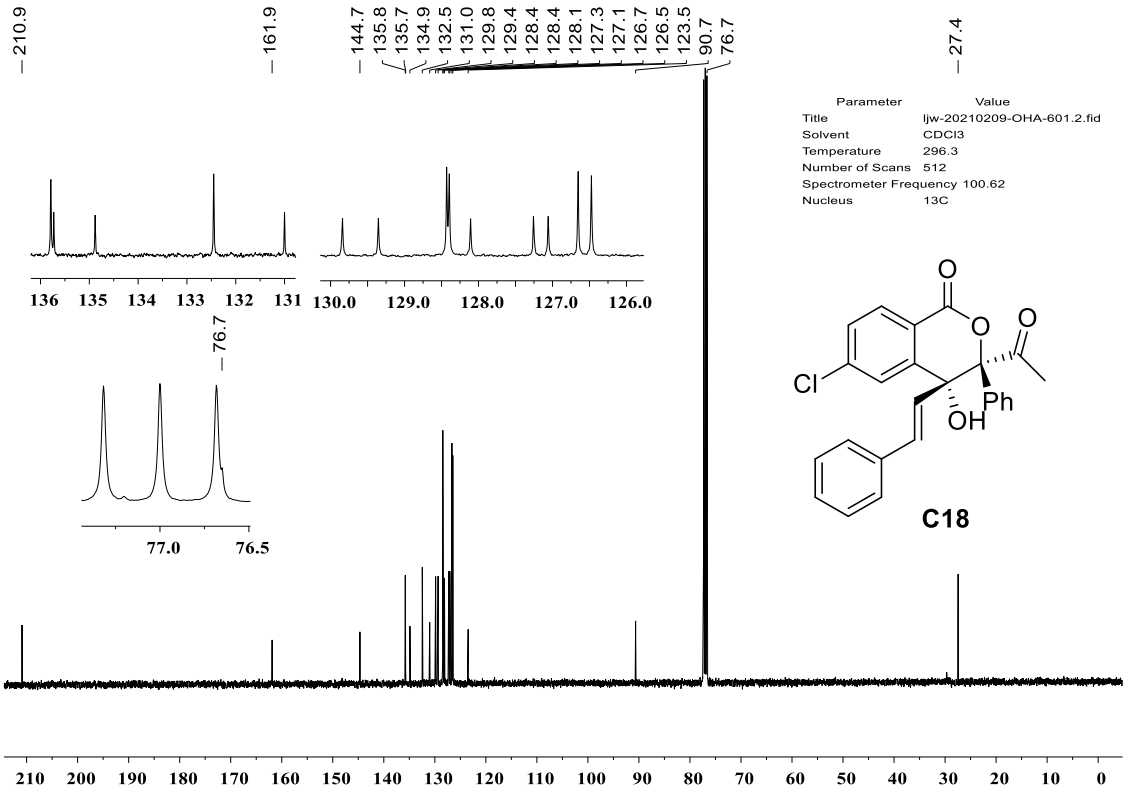
1.08
3.18
3.10
3.15
2.05
1.00
1.02
1.00
3.02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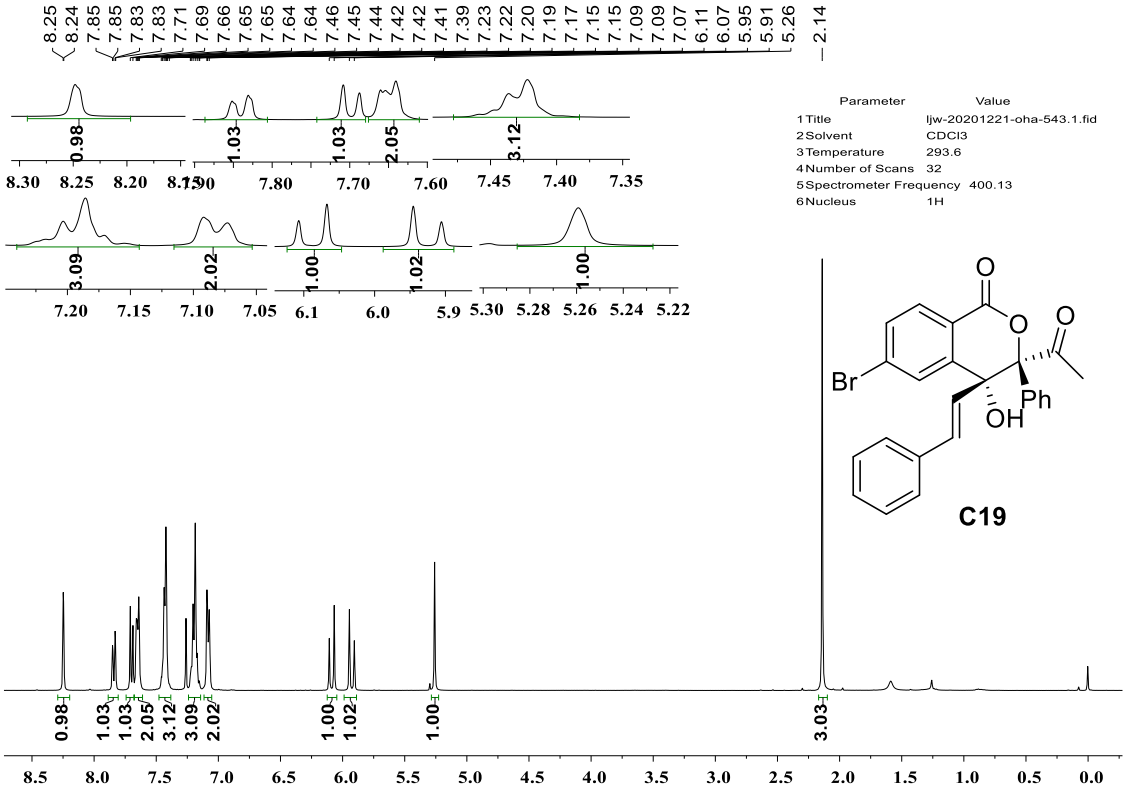
135.7
134.9
132.5
131.0
129.8
129.4
128.4
128.4
128.1
127.3
127.1
126.7
126.5
123.5
90.7
76.7
27.4
Parameter Value
Title ljw-20210209-OHA-601.2.fid
Solvent CDCl3
Temperature 296.3
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
Cl
O
Ph
OH
C18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7.85
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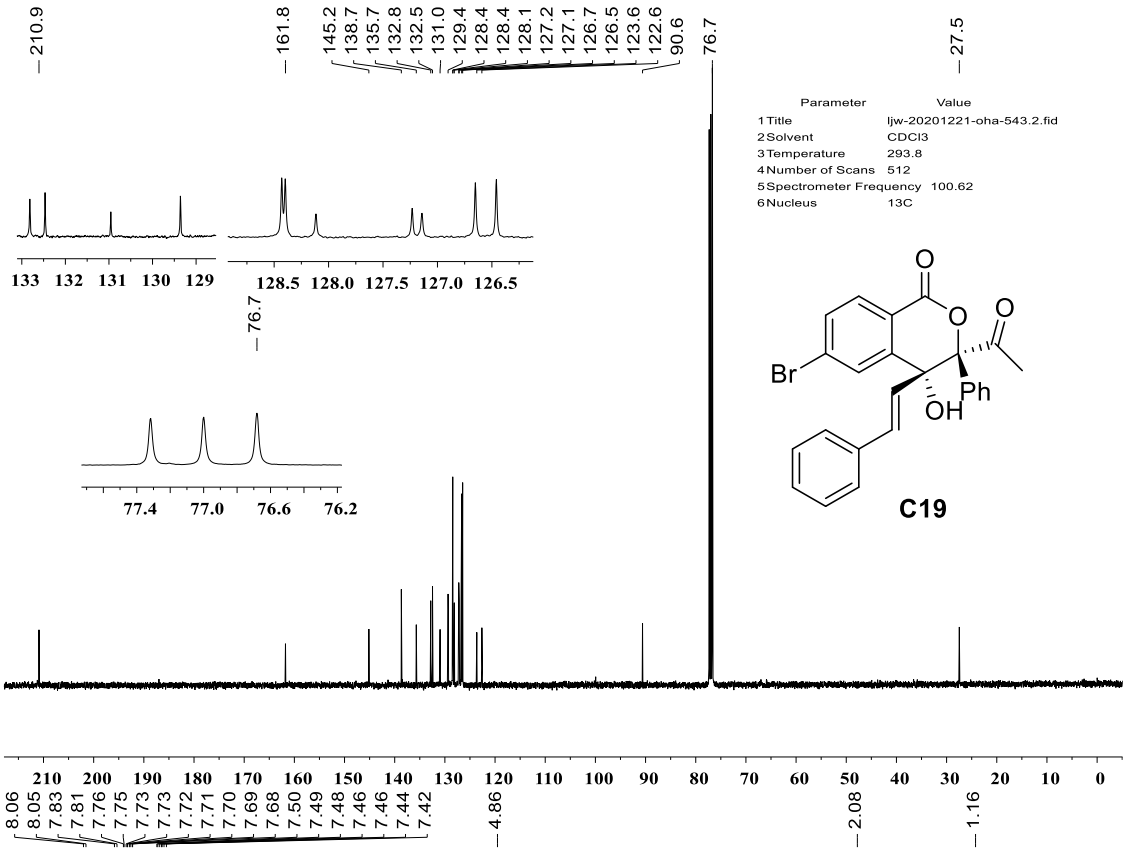
7.83
7.71
7.69
7.66
7.65
7.65
7.64
7.64
7.46
7.45
7.44
7.42
7.42
7.41
7.39
7.23
7.22
7.20
7.19
7.17
7.15
7.15
7.09
7.09
7.07
6.11
6.07
5.95
5.91
5.26
2.14
Parameter Value
1 Title ljw-20201221-oha-543.1.fid
2 Solvent CDCl3
3 Temperature 293.6
4 Number of Scans 32
5 Spectrometer Frequency 400.13
6 Nucleus 1H
Br
O
Ph
OH
C19


210.9
161.8
145.2
138.7
135.7
132.8
132.5
131.0
129.4
128.4
128.4
128.1
127.2
127.1
126.7
126.5
123.6
122.6
90.6
76.7
27.5
Parameter Value
1 Title ljw-20201221-oha-543.2.fid
2 Solvent CDCl3
3 Temperature 293.8
4 Number of Scans 512
5 Spectrometer Frequency 100.62
6 Nucleus 13C
133 132 131 130 129
128.5 128.0 127.5 127.0 126.5
76.7
77.4 77.0 76.6 76.2
Br
Ph
OH
C19
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
8.06 8.05 7.83 7.81 7.76 7.75 7.73 7.73 7.72 7.71 7.70 7.69 7.68 7.50 7.49 7.48 7.46 7.46 7.44 7.42
4.86
2.08
1.16
1.00
1.01
3.06
8.08 8.06 8.04
7.80 7.75 7.70
4.11
1.00
7.50 7.48 7.46 7.44 7.42
4.88 4.86 4.84
Parameter Value
Title ljw-20201218-oha-537.1.fid
Solvent CDCl3
Temperature 295.5
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
Ph
OH
C20
1.00
1.01
3.06
4.11
1.00
3.01
3.06
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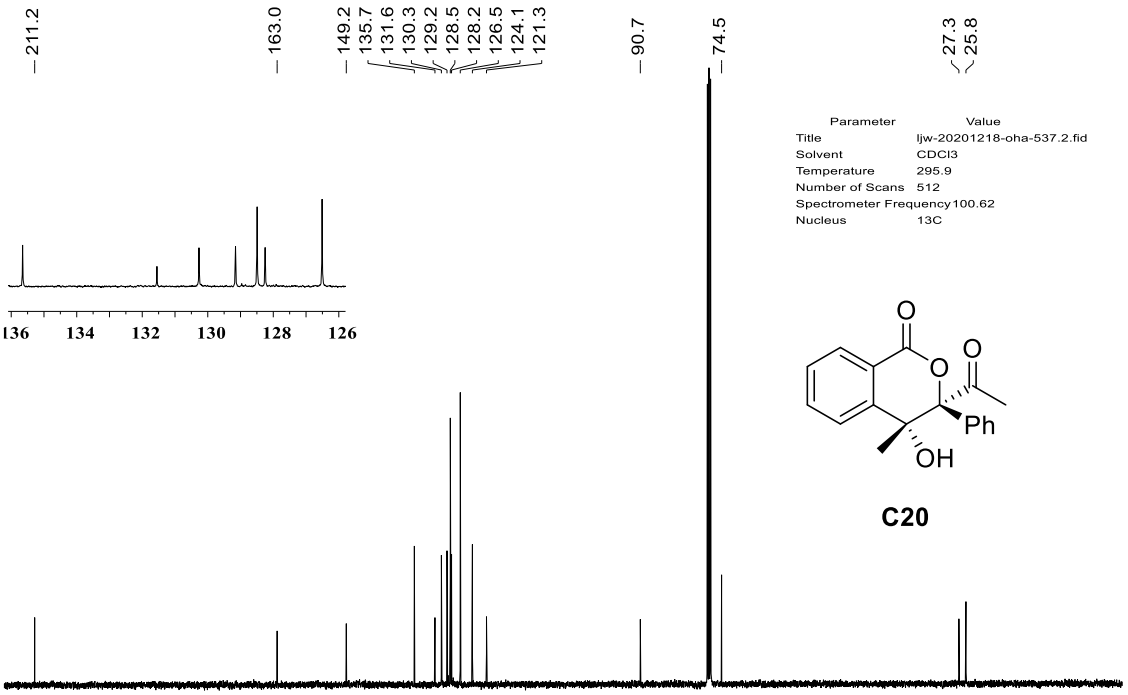
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124.1
121.3
90.7
74.5
27.3
25.8
Parameter Value
Title ljw-20201218-oha-537.2.fid
Solvent CDCl3
Temperature 295.9
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
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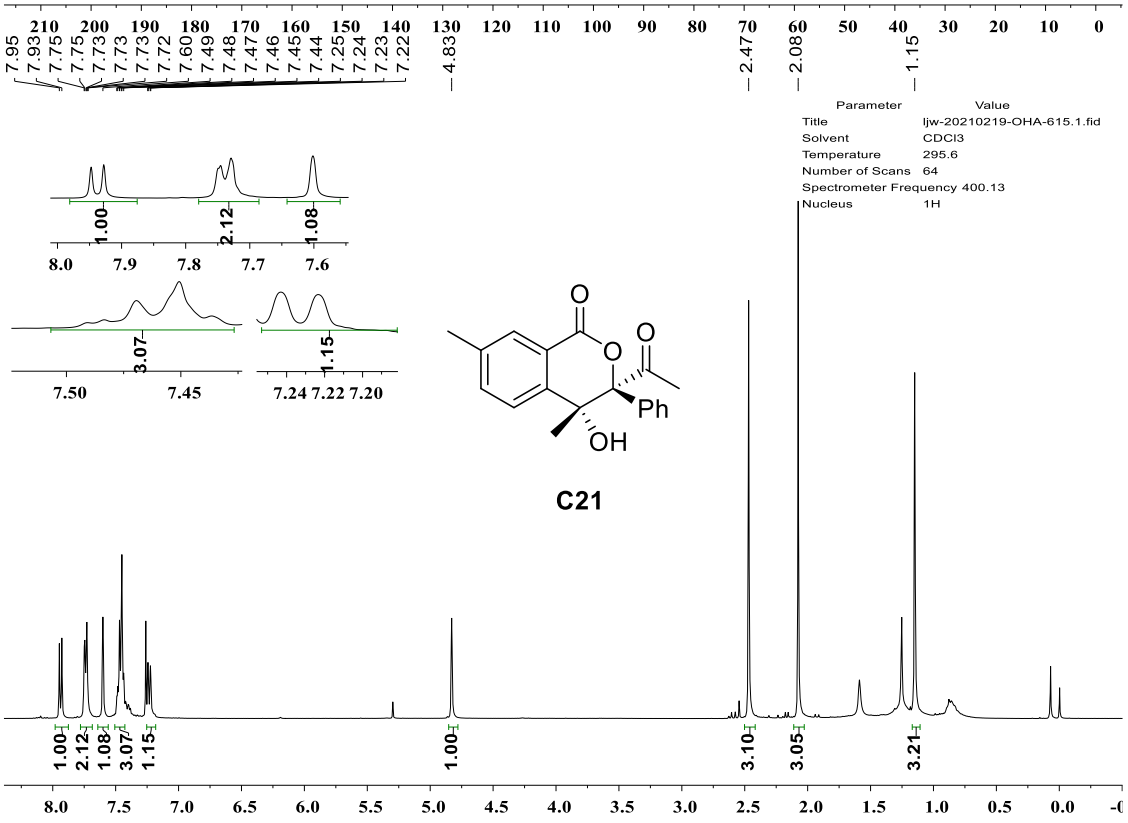
O
O
Ph
OH

C20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7.46
7.45
7.44
7.25
7.24
7.23
7.22
4.83
2.47
2.08
1.15
Parameter Value
Title ljw-20210219-OHA-615.1.fid
Solvent CDCl3
Temperature 295.6
Number of Scans 64
Spectrometer Frequency 400.13
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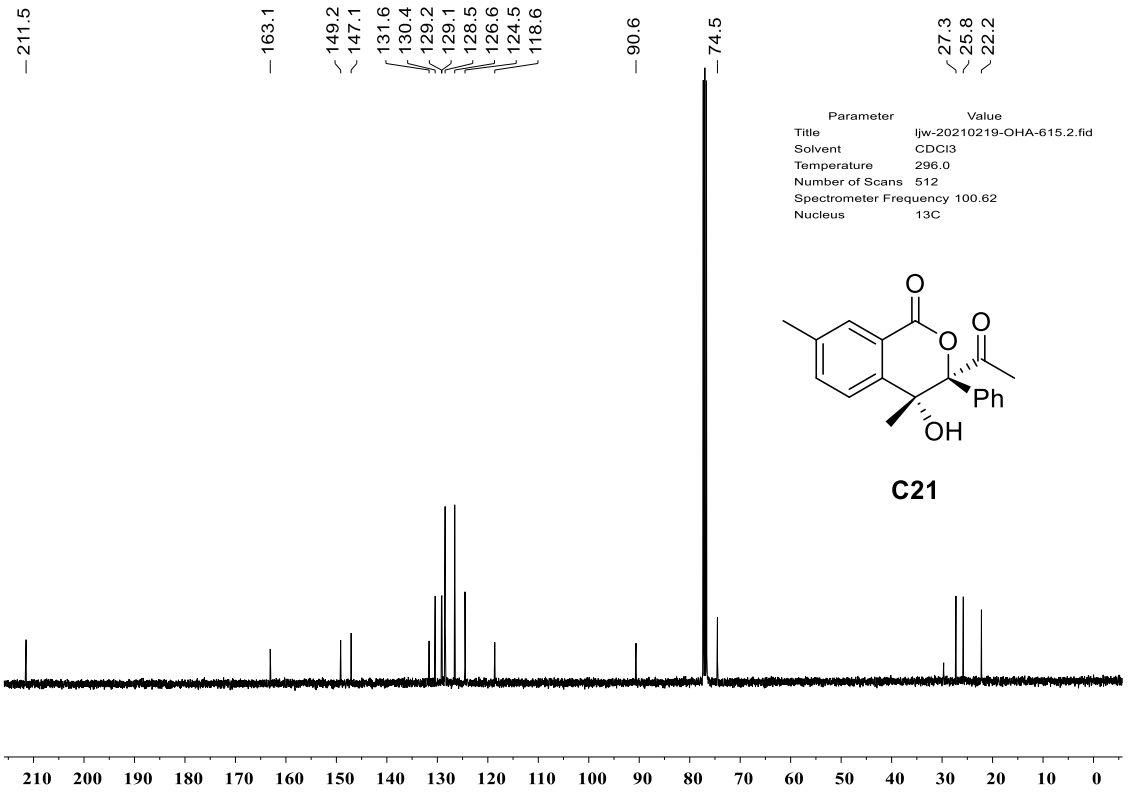
2.12
1.08
3.07
1.15
O
O
O
Ph
OH

C21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129.1
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124.5
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90.6
74.5
27.3
25.8
22.2
Parameter Value
Title ljw-20210219-OHA-615.2.fid
Solvent CDCl3
Temperature 296.0
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
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O
O
Ph
OH
C21
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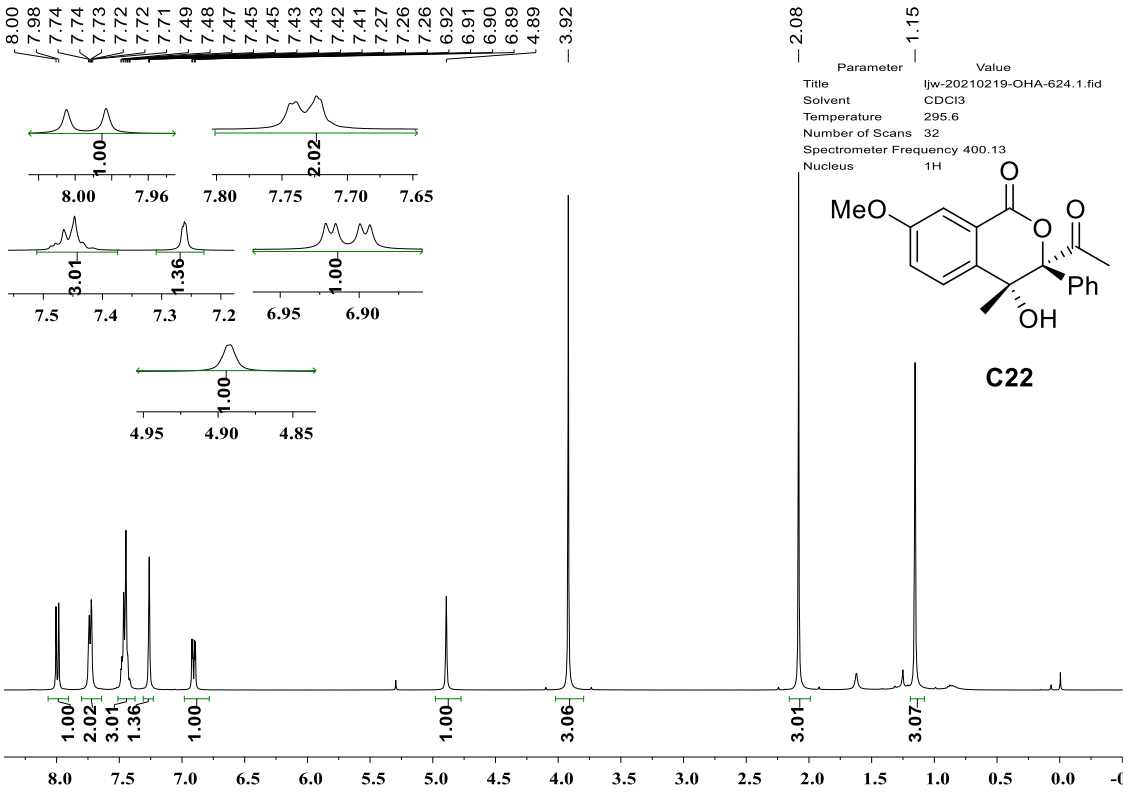
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7.47
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7.45
7.43
7.43
7.42
7.41
7.27
7.26
7.26
6.92
6.91
6.90
6.89
4.89
3.92
2.08
1.15
Parameter Value
Title ljw-20210219-OHA-624.1.fid
Solvent CDCl3
Temperature 295.6
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
MeO
O
O
O
Ph
OH
C22
1.00
2.02
3.01
1.36
1.00
1.00
3.06
3.01
3.07
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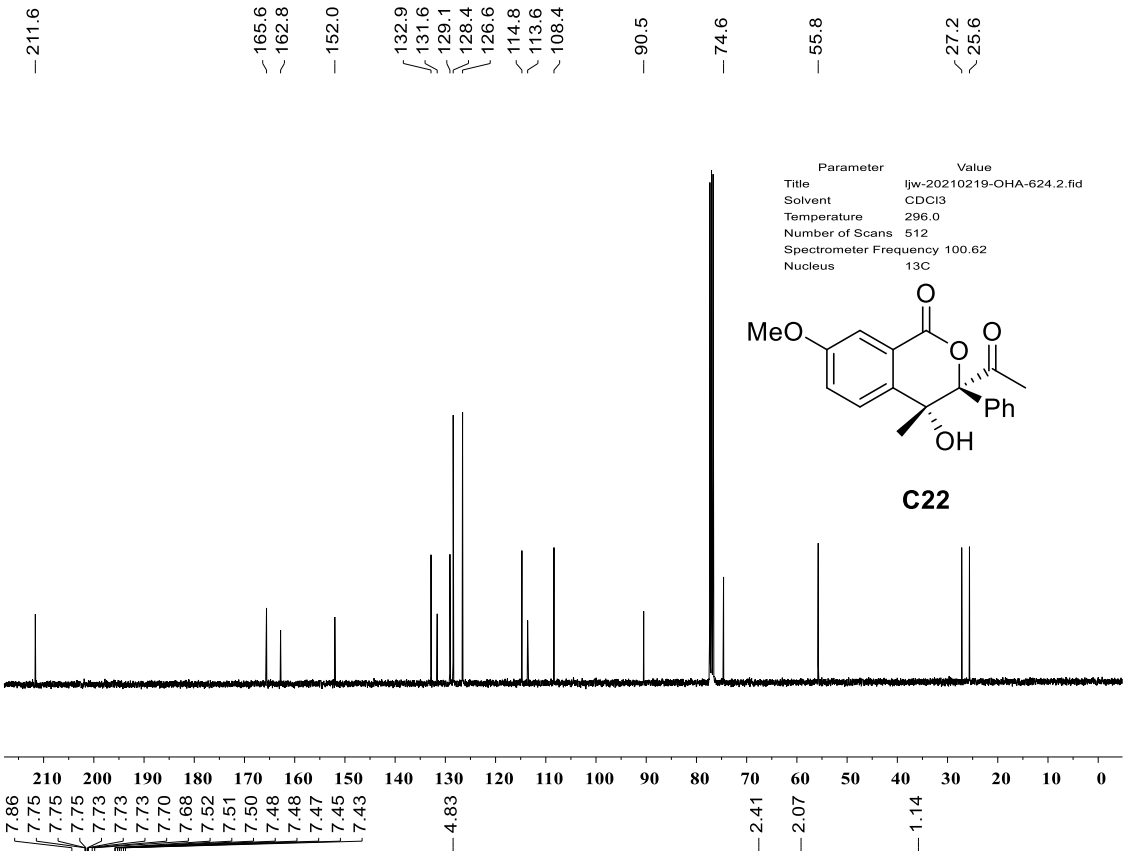
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129.1
128.4
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114.8
113.6
108.4
90.5
74.6
55.8
27.2
25.6
Parameter Value
Title ljw-20210219-OHA-624.2.fid
Solvent CDCl3
Temperature 296.0
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
MeO
O
O
O
Ph
OH
C22
7.86
7.75
7.75
7.75
7.73
7.73
7.73
7.70
7.68
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7.43
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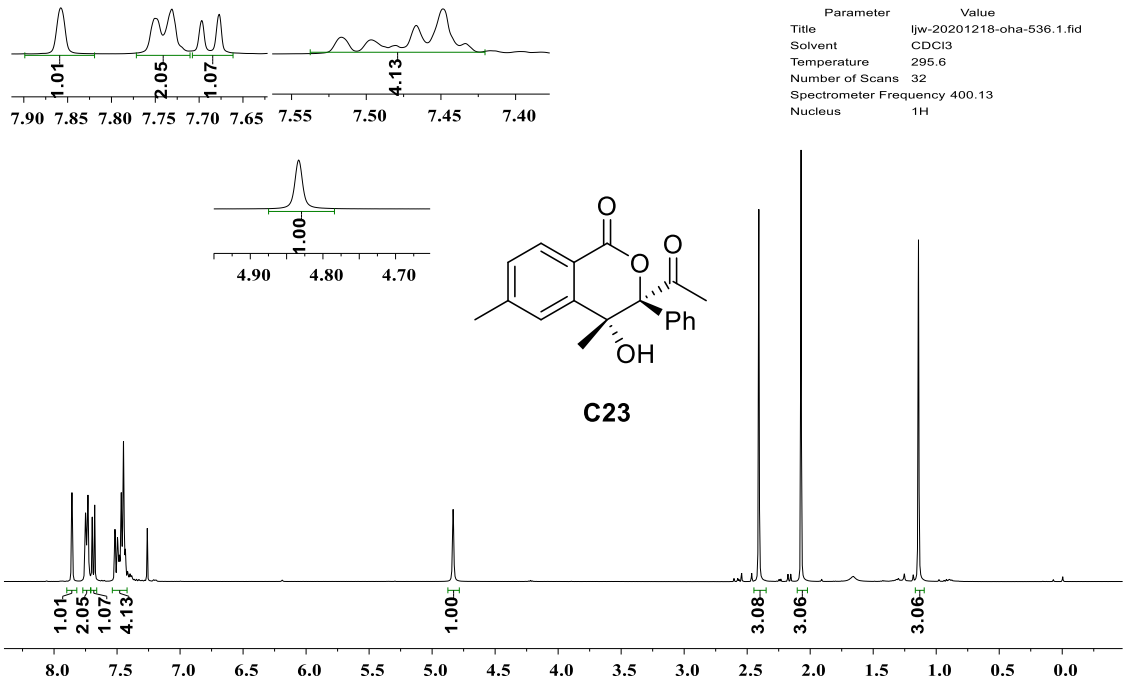
2.07
1.14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Temperature 295.6
Number of Scans 32
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1.07
4.13
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O
Ph
OH
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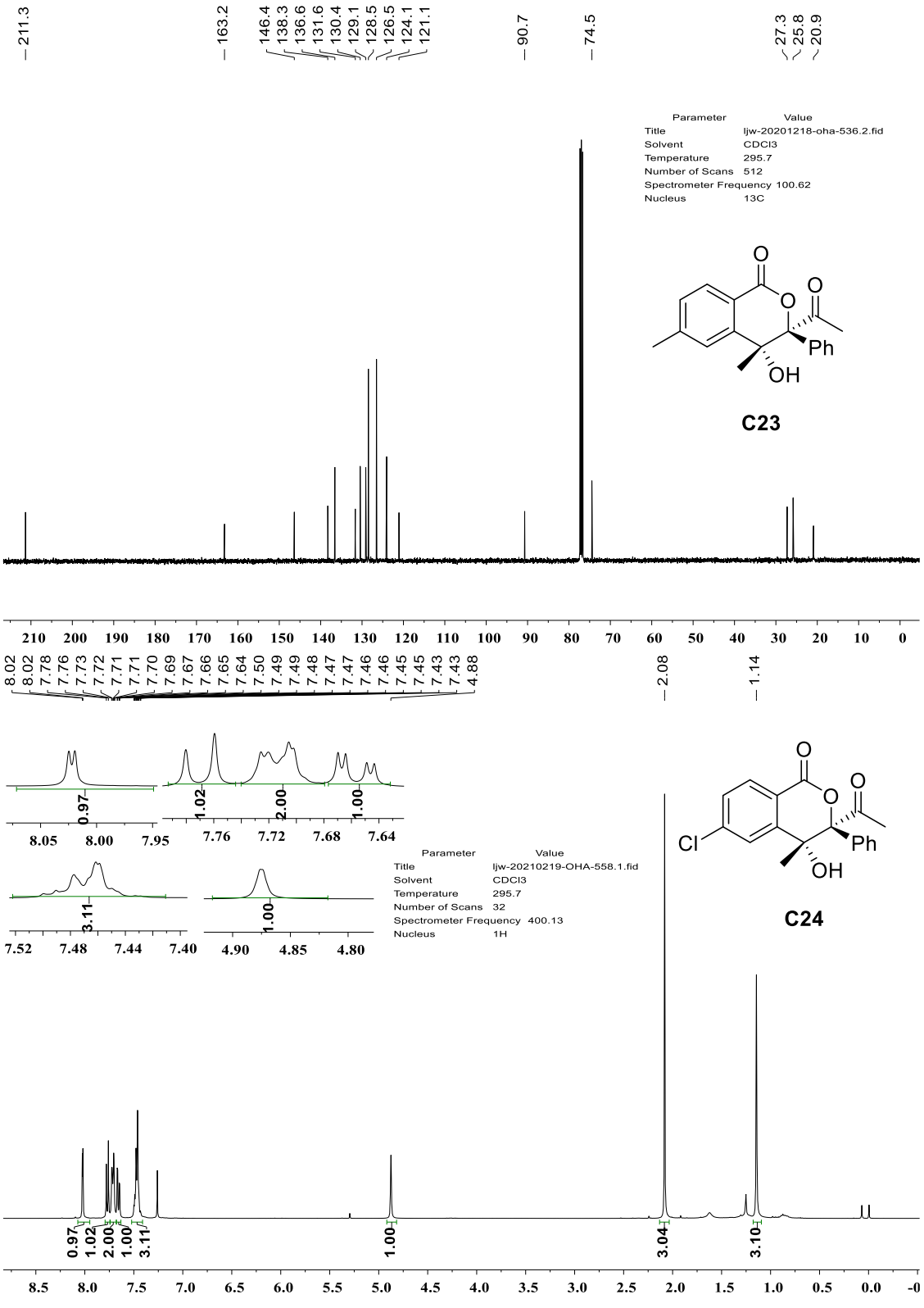
3.06
3.06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130.4
129.1
128.5
126.5
124.1
121.1
90.7
74.5
27.3
25.8
20.9
Parameter Value
Title ljw-20201218-oha-536.2.fid
Solvent CDCl3
Temperature 295.7
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
Ph
OH
C23
8.02
8.02
7.78
7.76
7.73
7.72
7.71
7.71
7.70
7.69
7.67
7.66
7.65
7.64
7.50
7.49
7.49
7.48
7.47
7.47
7.46
7.46
7.45
7.45
7.43
7.43
4.88
2.08
1.14
0.97
1.02
2.00
1.00
3.11
1.00
Parameter Value
Title ljw-20210219-OHA-558.1.fid
Solvent CDCl3
Temperature 295.7
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
Cl
O
O
O
Ph
OH
C24
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2.00
1.00
3.11
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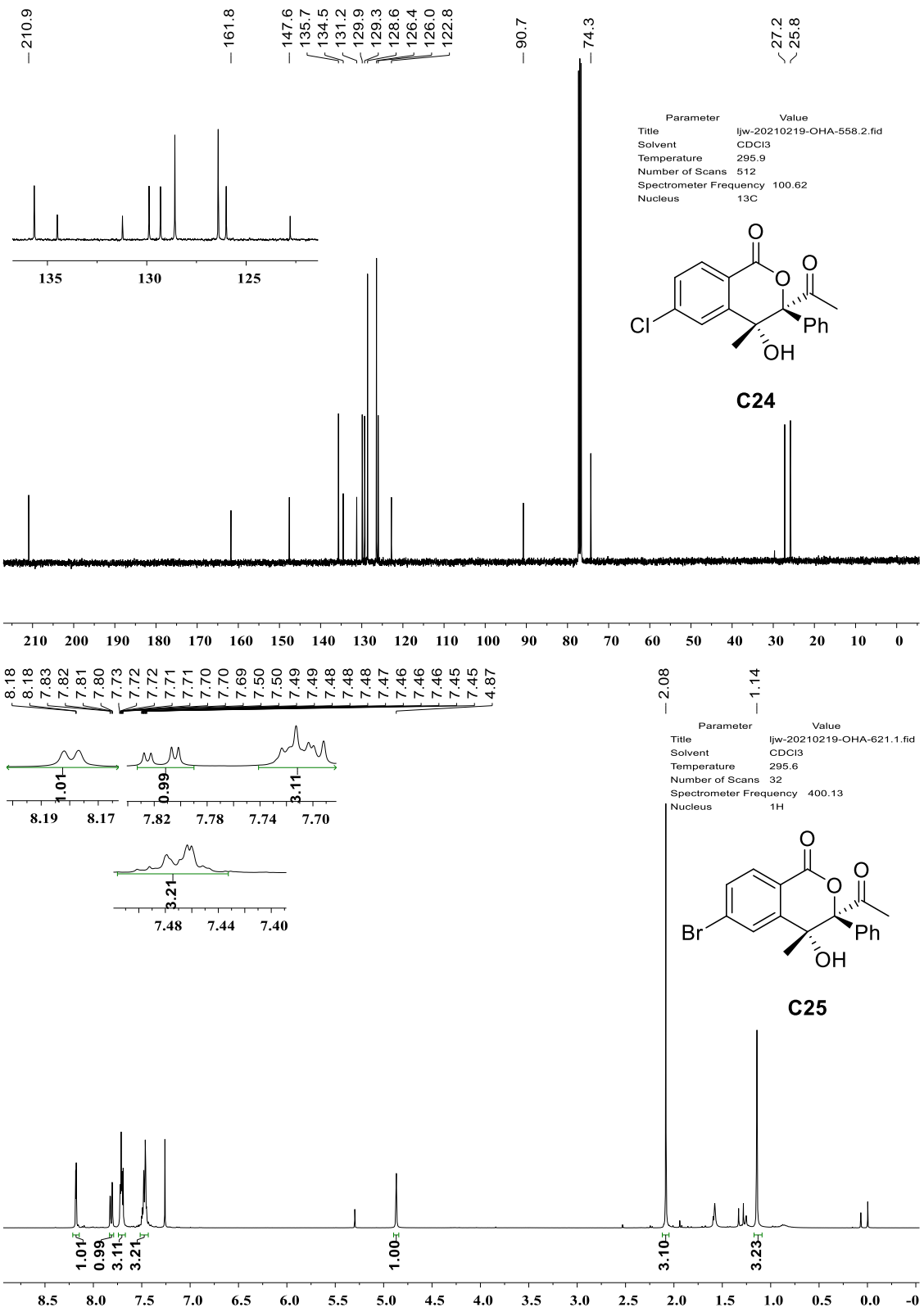
3.04
3.10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129.3
128.6
126.4
126.0
122.8
90.7
74.3
27.2
25.8
Parameter Value
Title ljw-20210219-OHA-558.2.fid
Solvent CDCl3
Temperature 295.9
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
Cl
Ph
OH
C24
8.18
8.18
7.83
7.82
7.81
7.80
7.73
7.72
7.72
7.71
7.71
7.70
7.70
7.69
7.50
7.50
7.49
7.49
7.48
7.48
7.48
7.47
7.46
7.46
7.46
7.45
7.45
4.87
2.08
1.14
Parameter Value
Title ljw-20210219-OHA-621.1.fid
Solvent CDCl3
Temperature 295.6
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
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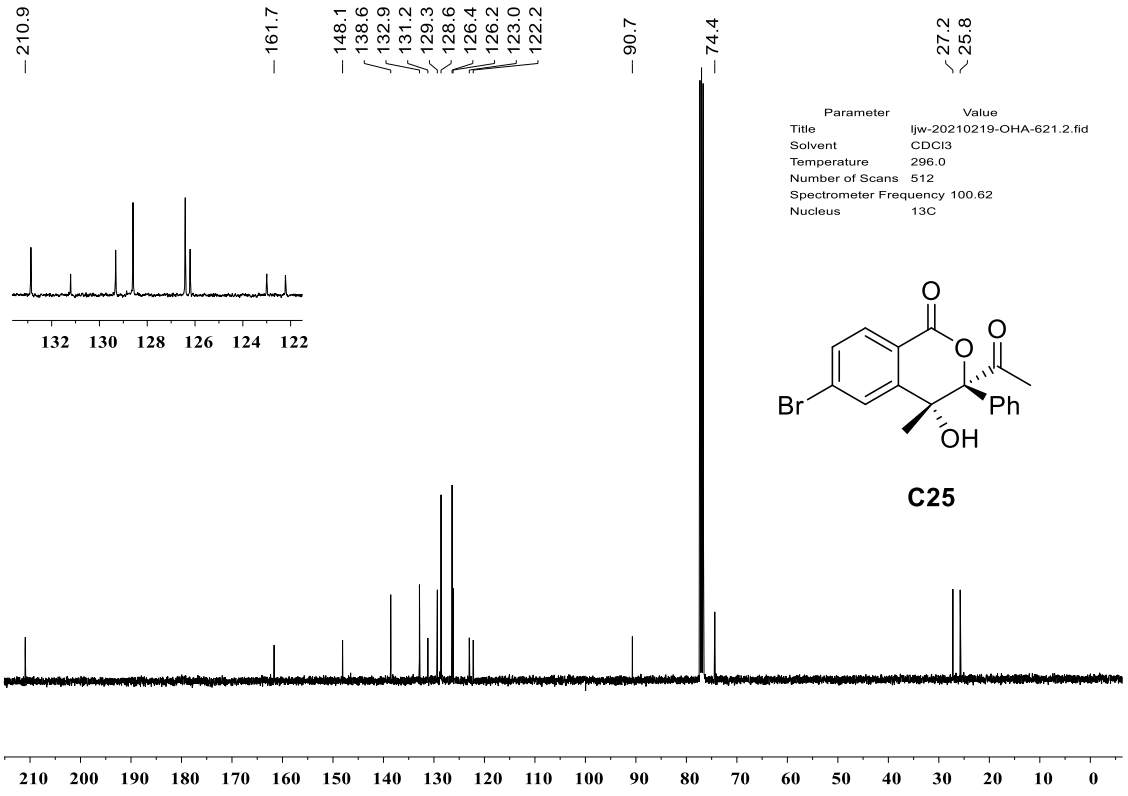
3.11
3.21
1.00
3.10
3.23
O
O
O
Br
Ph
OH
C25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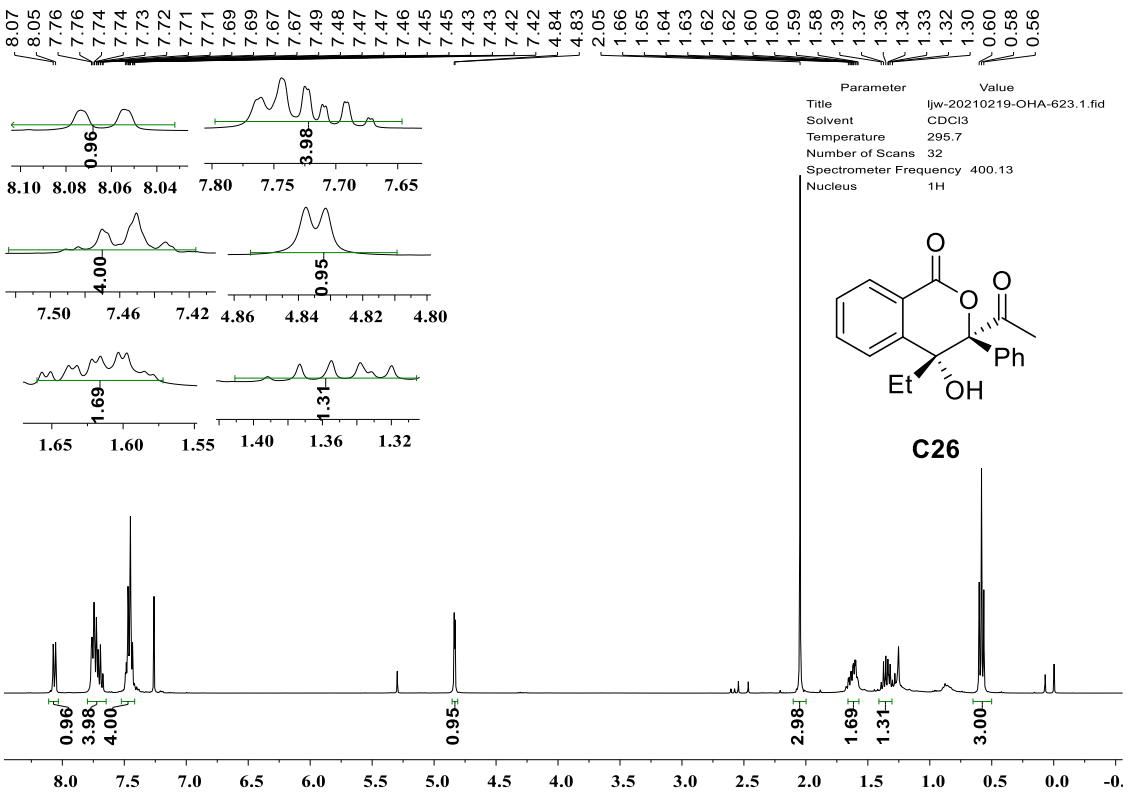
148.1
138.6
132.9
131.2
129.3
128.6
126.4
126.2
123.0
122.2
90.7
74.4
27.2
25.8
Parameter Value
Title ljw-20210219-OHA-621.2.fid
Solvent CDCl3
Temperature 296.0
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
Br
Ph
OH
C25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7.71
7.69
7.69
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7.67
7.49
7.48
7.47
7.47
7.46
7.45
7.45
7.43
7.43
7.42
7.42
4.84
4.83
2.05
1.66
1.65
1.64
1.63
1.62
1.62
1.60
1.60
1.59
1.58
1.39
1.37
1.36
1.34
1.33
1.32
1.30
0.60
0.58
0.56
Parameter Value
Title ljw-20210219-OHA-623.1.fid
Solvent CDCl3
Temperature 295.7
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
0.96
3.98
4.00
0.95
1.69
1.31
O
O
O
Ph
Et
OH
C26
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3.98
4.00
0.95
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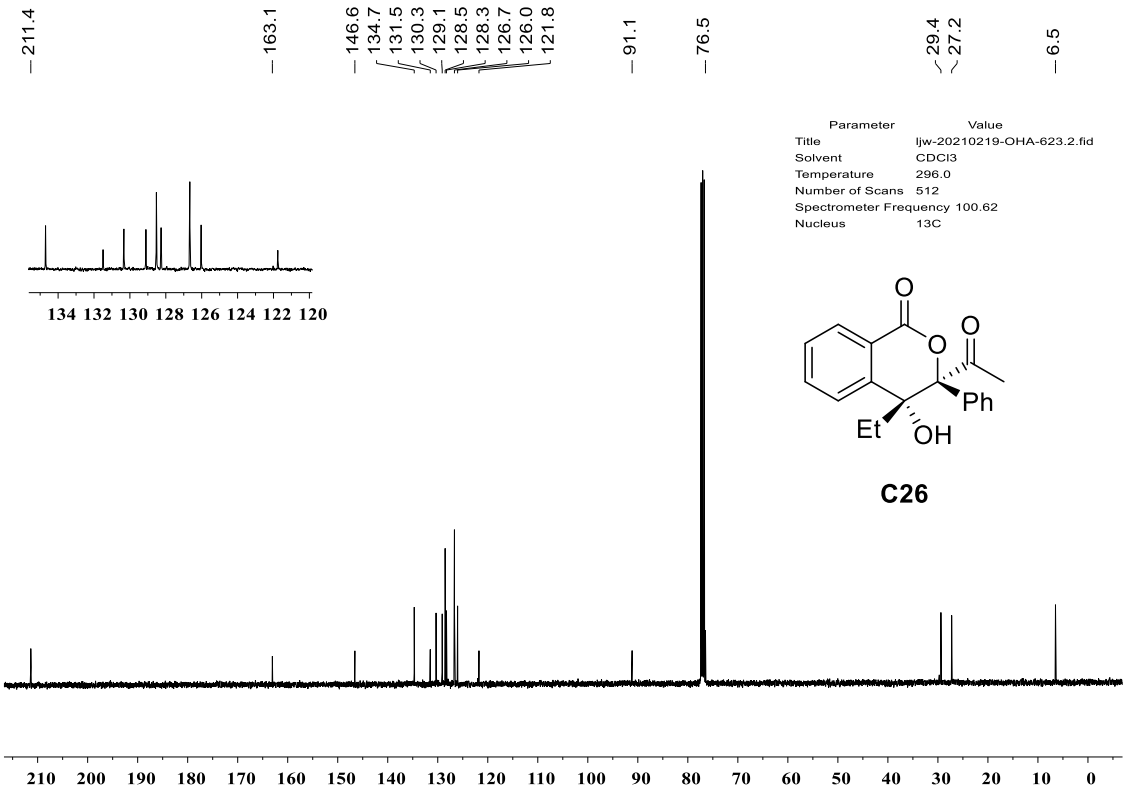
1.69
1.31
3.00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130.3
129.1
128.5
128.3
126.7
126.0
121.8
91.1
76.5
29.4
27.2
6.5
Parameter Value
Title ljw-20210219-OHA-623.2.fid
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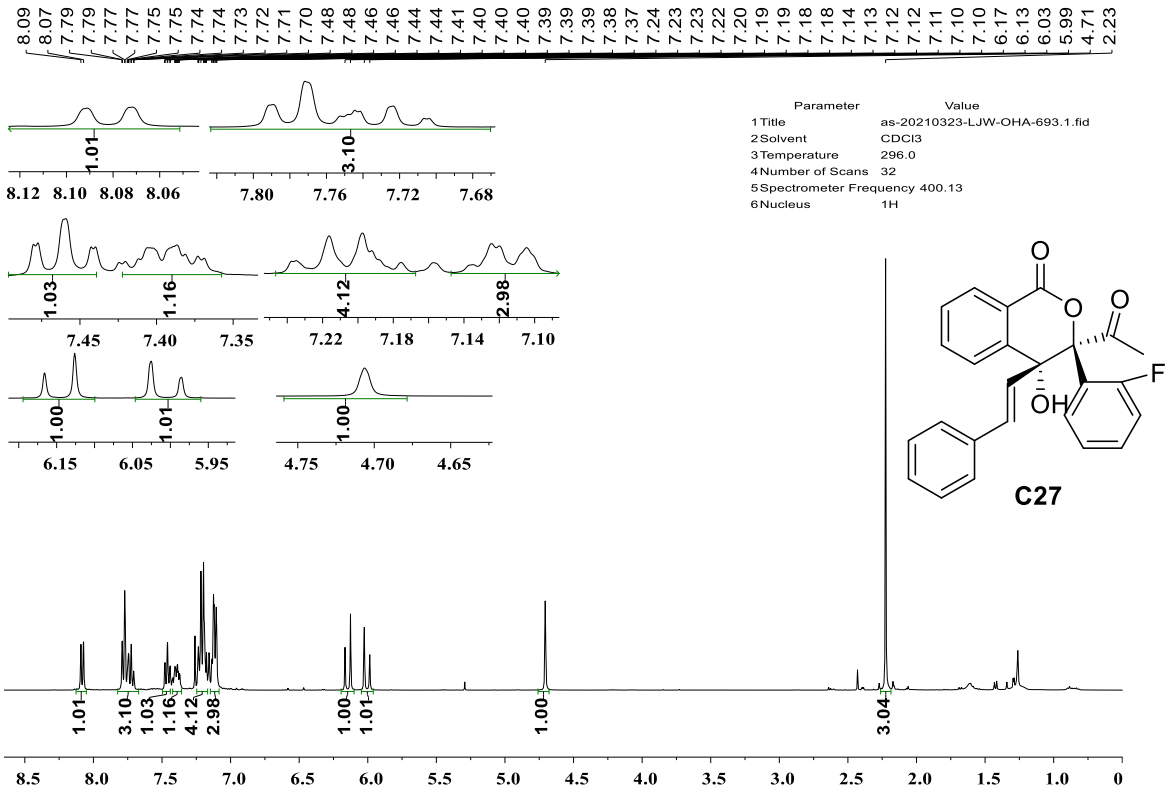
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
Ph
Et
OH
C26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Parameter Value
1 Title as-20210323-LJW-OHA-693.1.fid
2 Solvent CDCl3
3 Temperature 296.0
4 Number of Scans 32
5 Spectrometer Frequency 400.13
6 Nucleus 1H
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O
O
F
OH
C27
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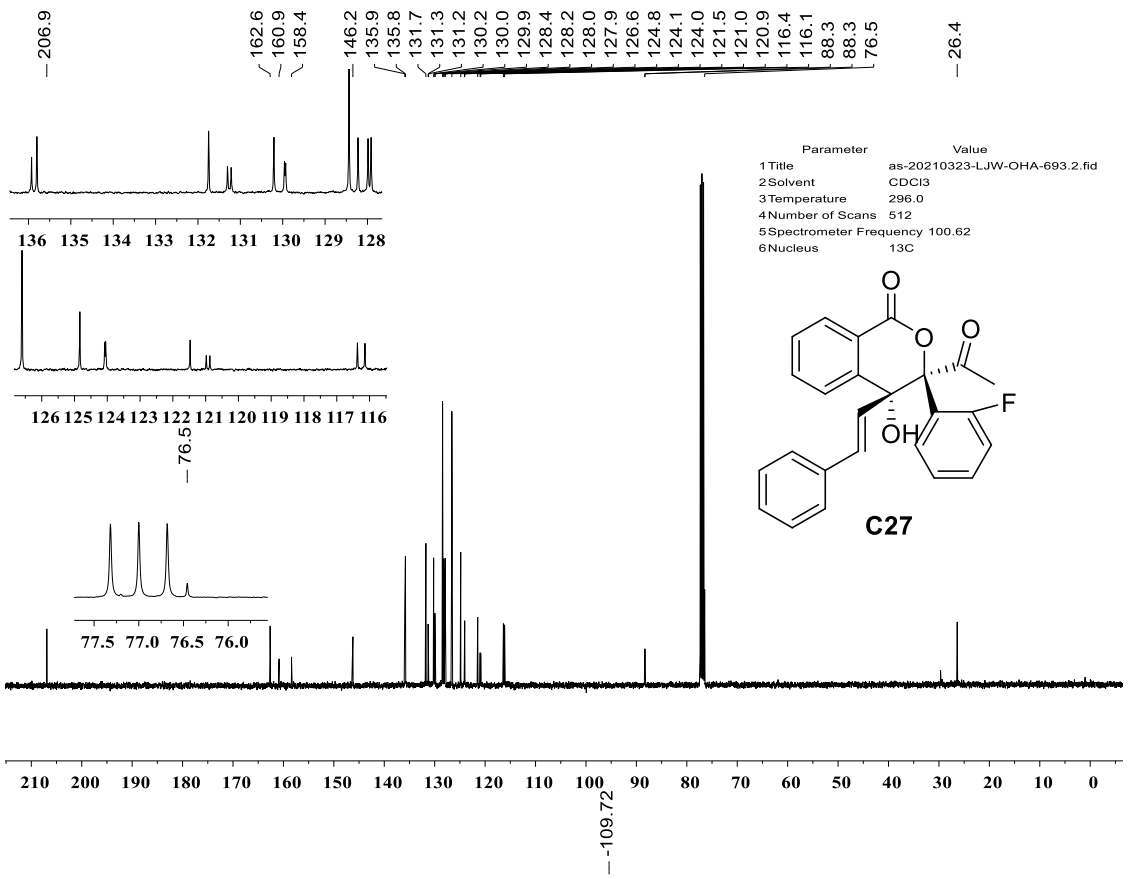
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135.8
131.7
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131.2
130.2
130.0
129.9
128.4
128.2
128.0
127.9
126.6
124.8
124.1
124.0
121.5
121.0
120.9
116.4
116.1
88.3
88.3
76.5
26.4
136 135 134 133 132 131 130 129 128
126 125 124 123 122 121 120 119 118 117 116
76.5
77.5 77.0 76.5 76.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
Parameter Value
1 Title as-20210323-LJW-OHA-693.2.fid
2 Solvent CDCl3
3 Temperature 296.0
4 Number of Scans 512
5 Spectrometer Frequency 100.62
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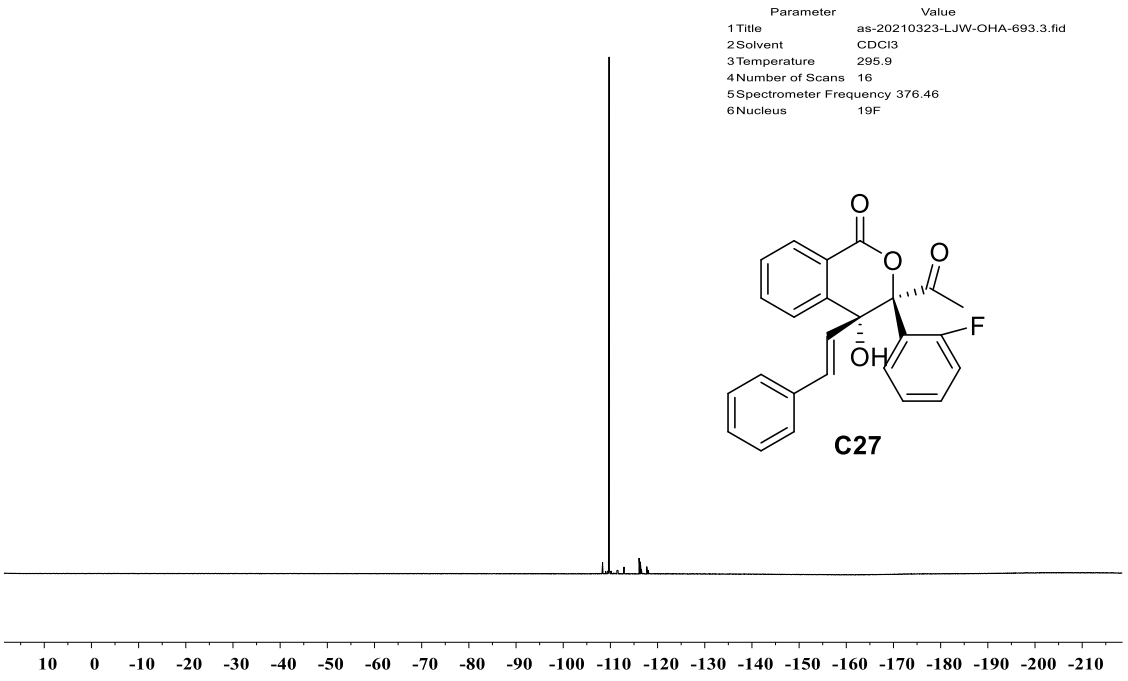
O
O
F
OH
C27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2 Solvent CDCl3
3 Temperature 295.9
4 Number of Scans 16
5 Spectrometer Frequency 376.46
6 Nucleus 19F
O
O
O
F
OH
C27
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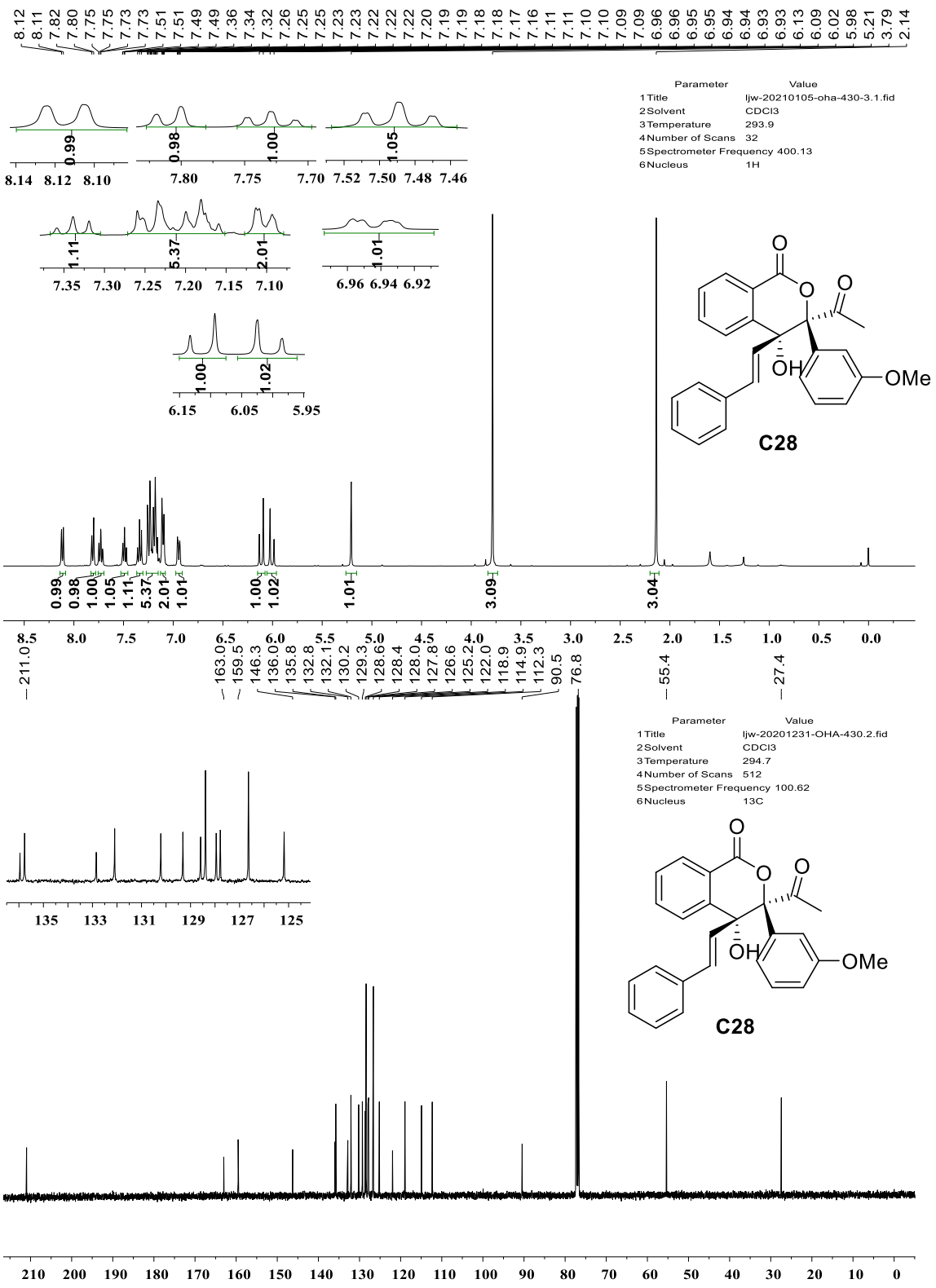
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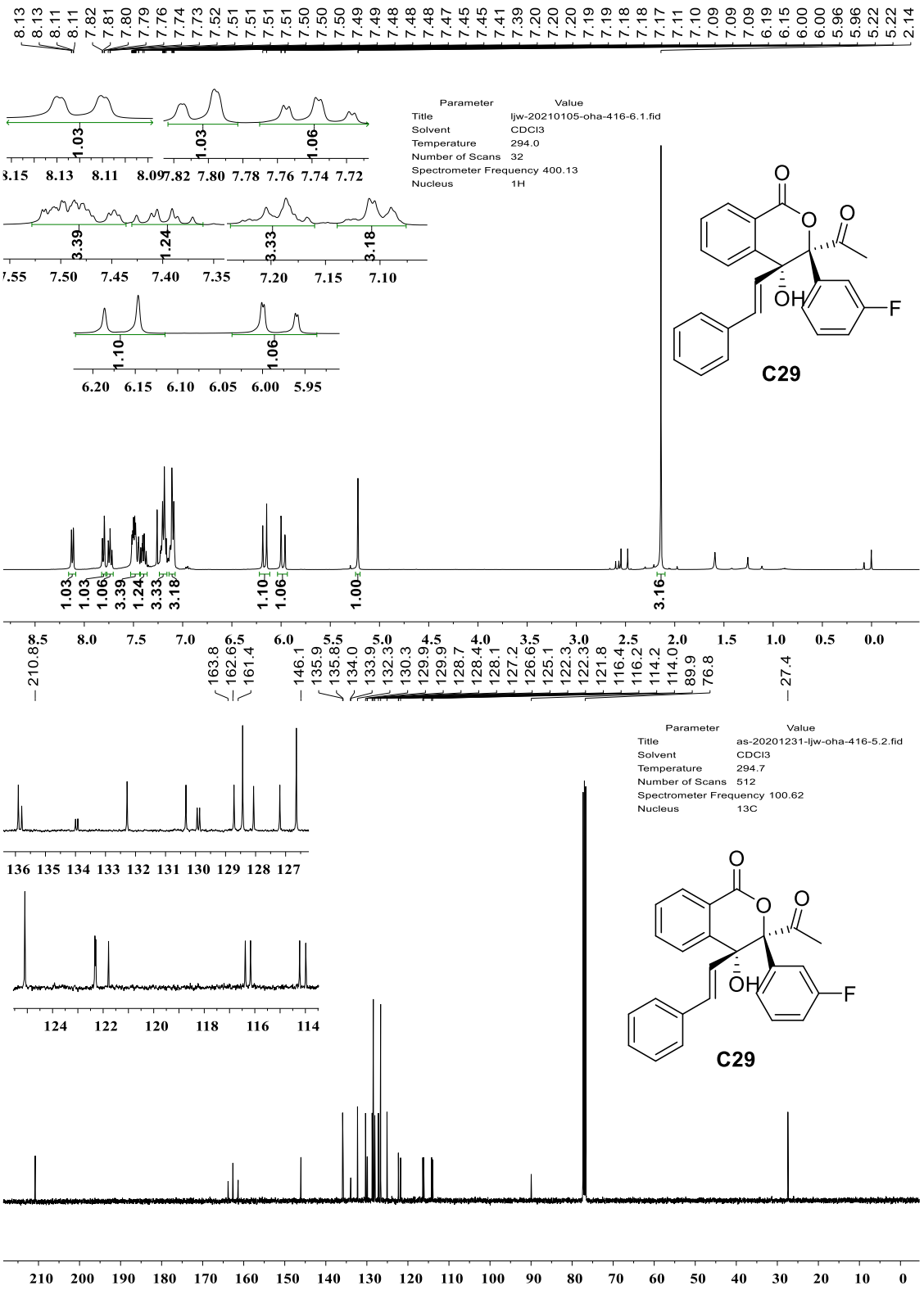
2 Solvent CDCl3
3 Temperature 293.9
4 Number of Scans 32
5 Spectrometer Frequency 400.13
6 Nucleus 1H
C28
Parameter Value
1 Title ljw-20201231-OHA-430.2.fid
2 Solvent CDCl3
3 Temperature 294.7
4 Number of Scans 512
5 Spectrometer Frequency 100.62
6 Nucleus 13C
C28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Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C29
Parameter Value
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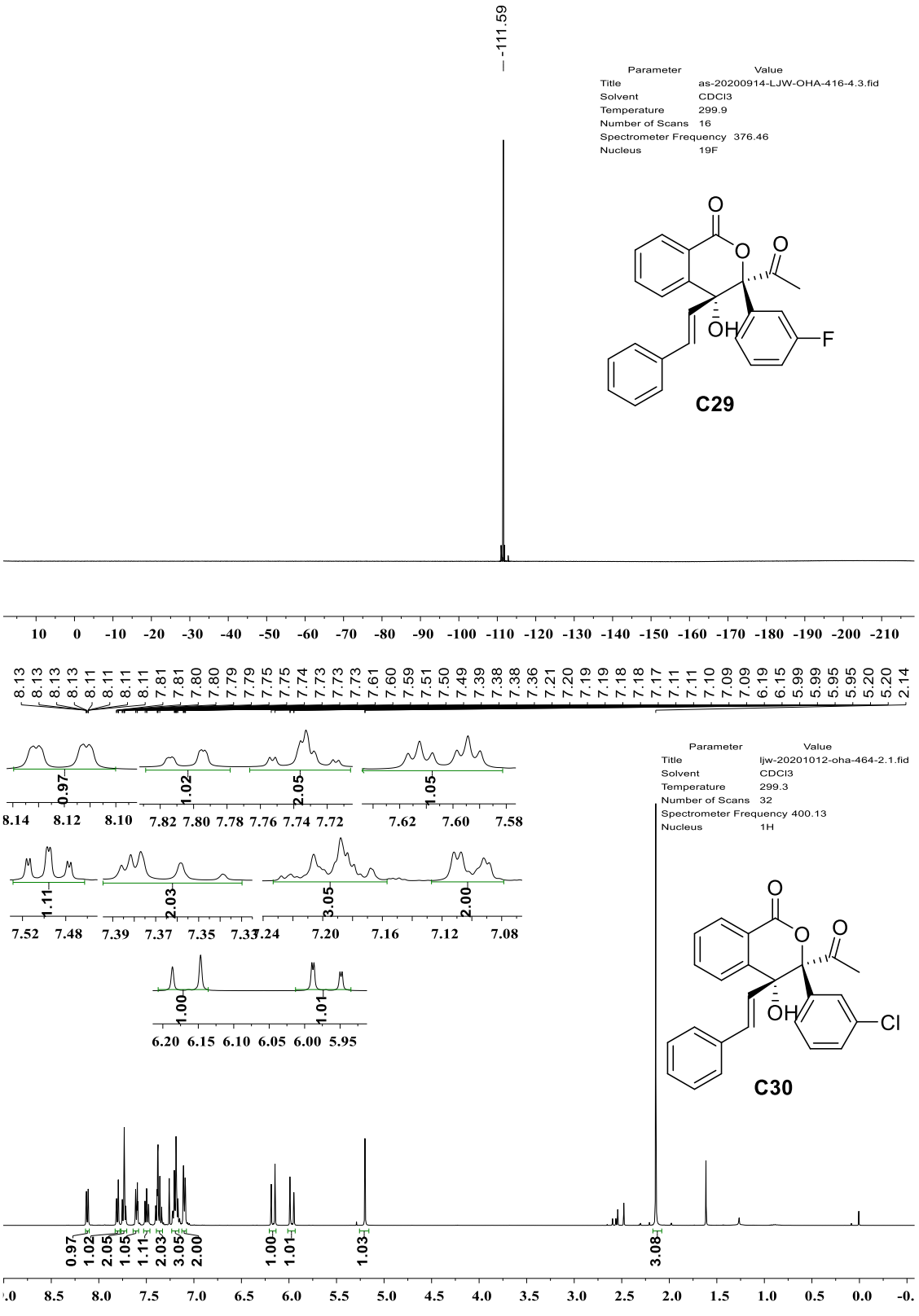
Solvent CDCl3
Temperature 294.7
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
C29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Number of Scans 16
Spectrometer Frequency 376.46
Nucleus 19F
C29
Parameter Value
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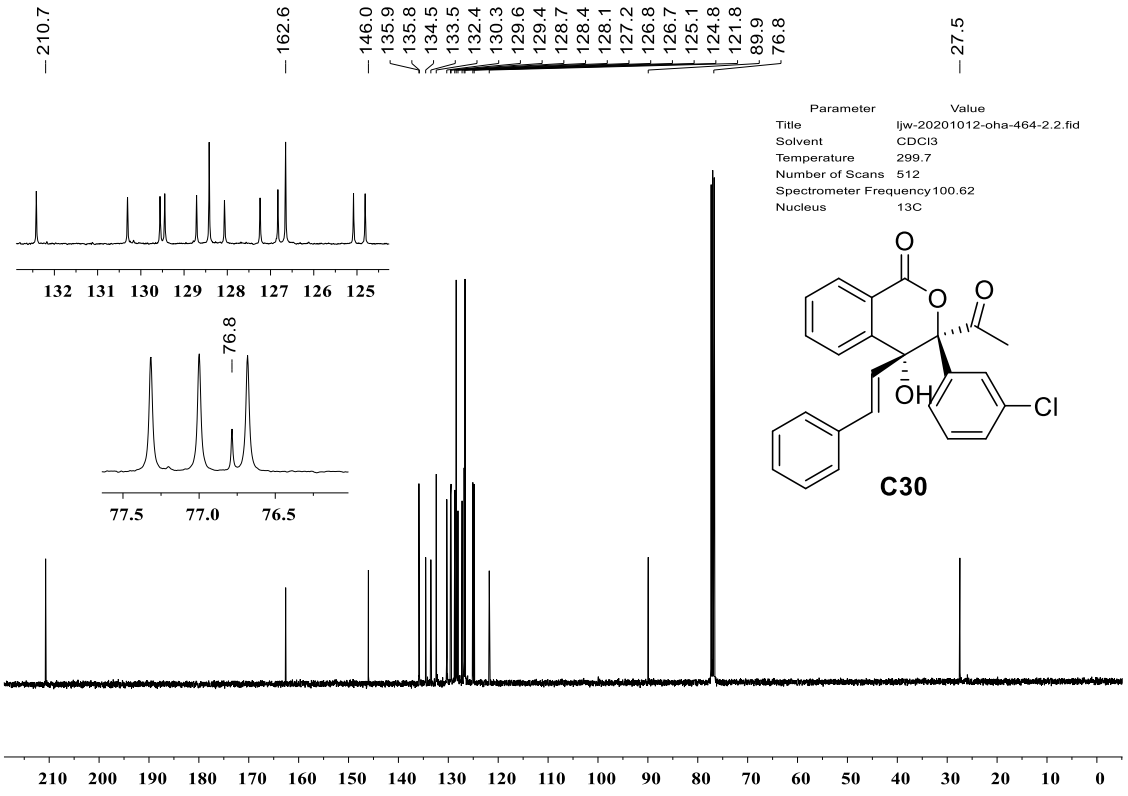
Solvent CDCl3
Temperature 299.3
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C30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133.5
132.4
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129.4
128.7
128.4
128.1
127.2
126.8
126.7
125.1
124.8
121.8
89.9
76.8
27.5
Parameter Value
Title ljw-20201012-oha-464-2.2.fid
Solvent CDCl3
Temperature 299.7
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
132 131 130 129 128 127 126 125
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O
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O
OH
Cl
C30
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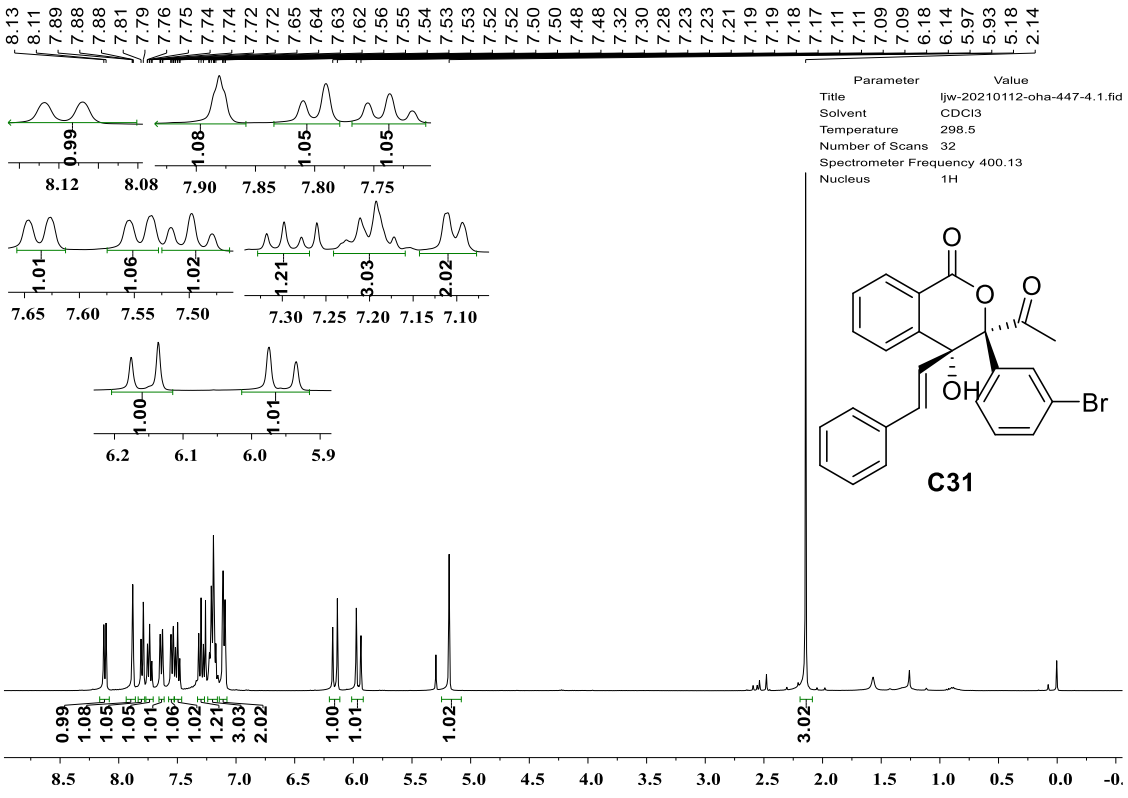
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7.53
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7.30
7.28
7.23
7.23
7.21
7.19
7.19
7.18
7.17
7.11
7.11
7.09
7.09
6.18
6.14
5.97
5.93
5.18
2.14
Parameter Value
Title ljw-20210112-oha-447-4.1.fid
Solvent CDCl3
Temperature 298.5
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
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8.12 8.08 7.90 7.85 7.80 7.75
1.01 1.06 1.02 1.21 3.03 2.02
7.65 7.60 7.55 7.50 7.30 7.25 7.20 7.15 7.10
1.00 1.01
6.2 6.1 6.0 5.9
O
O
O
OH
Br
C31
0.99 1.08 1.05 1.05 1.01 1.06 1.02 1.21 3.03 2.02 1.00 1.01 1.02 3.02
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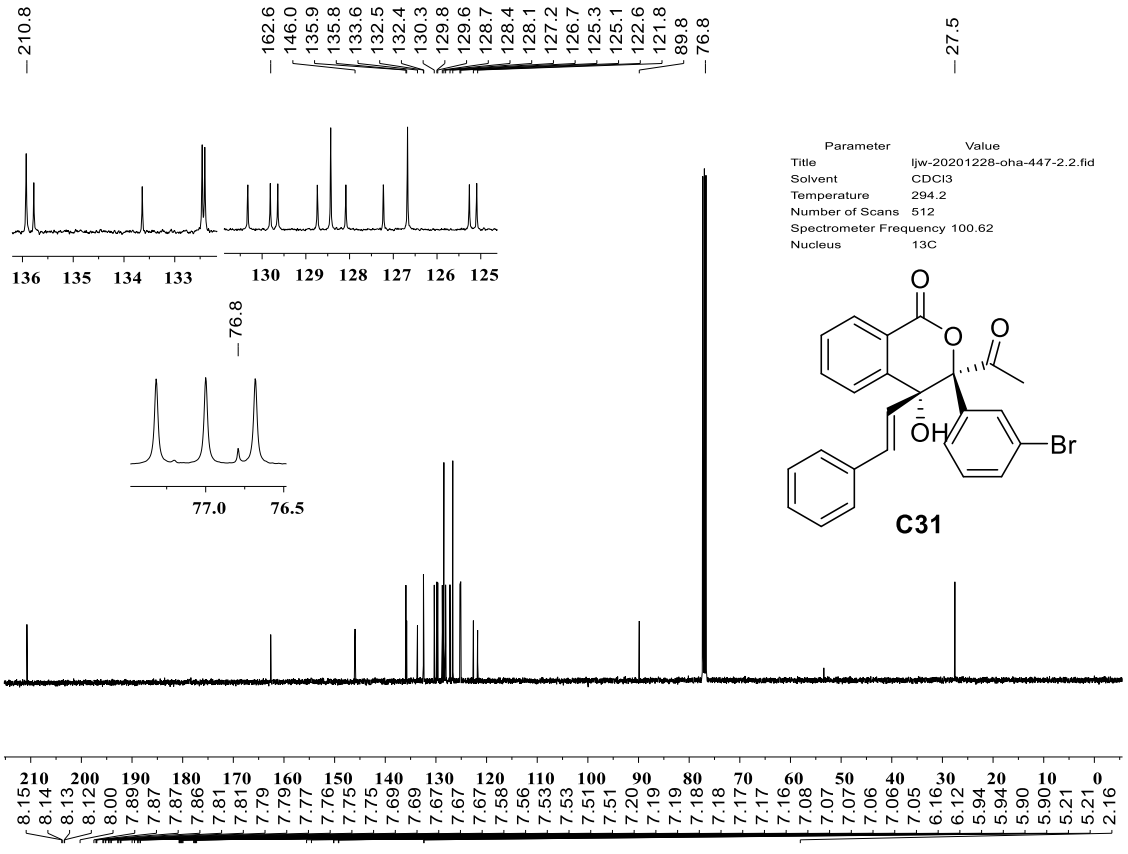
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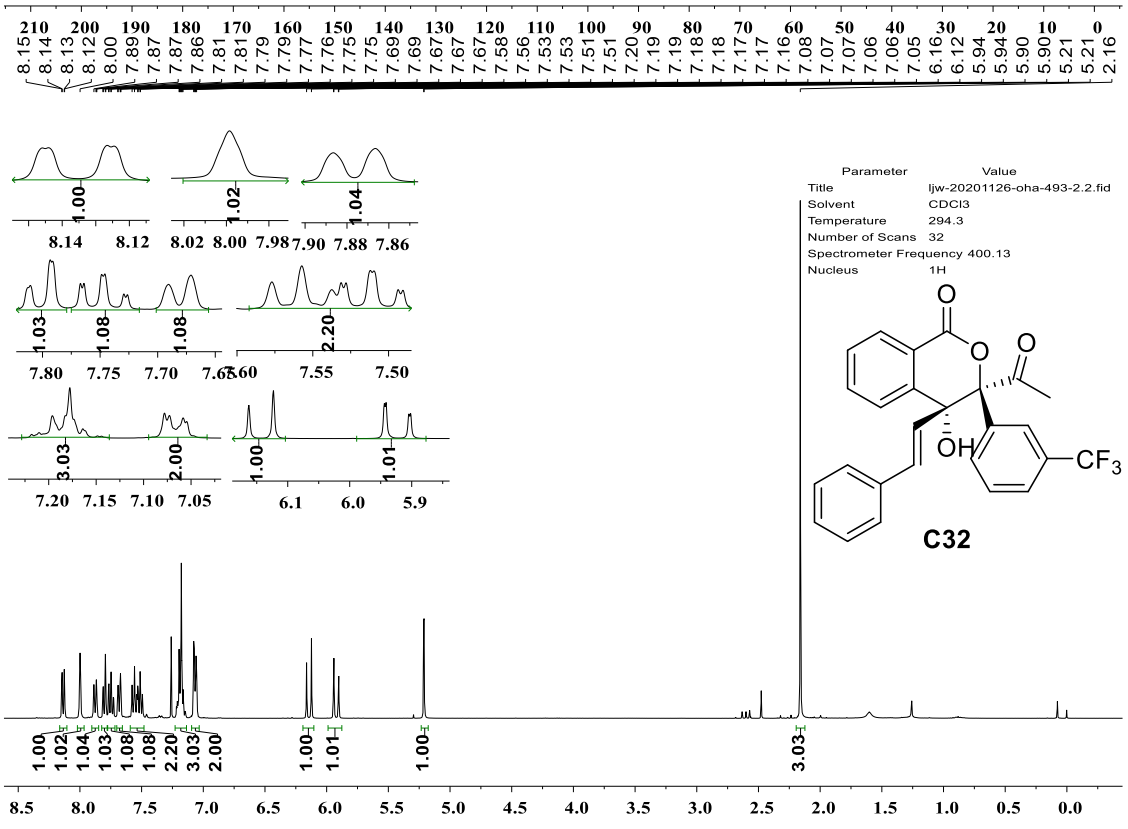
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
C31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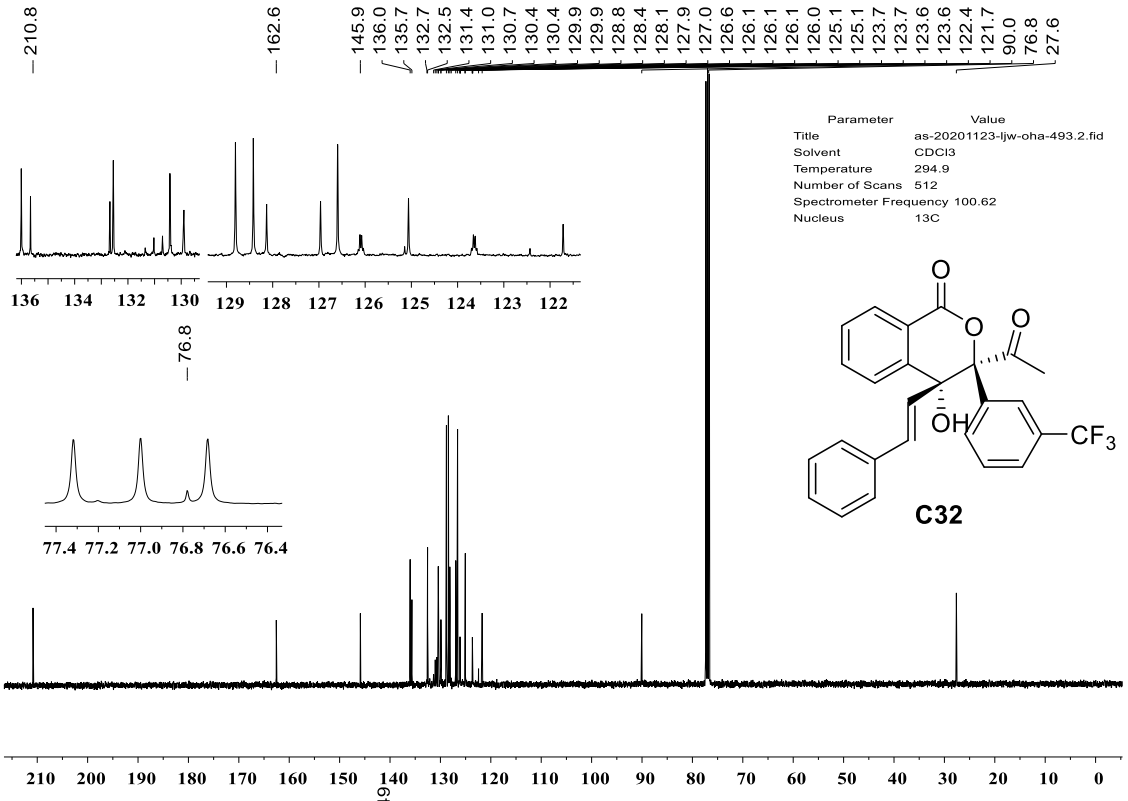
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C32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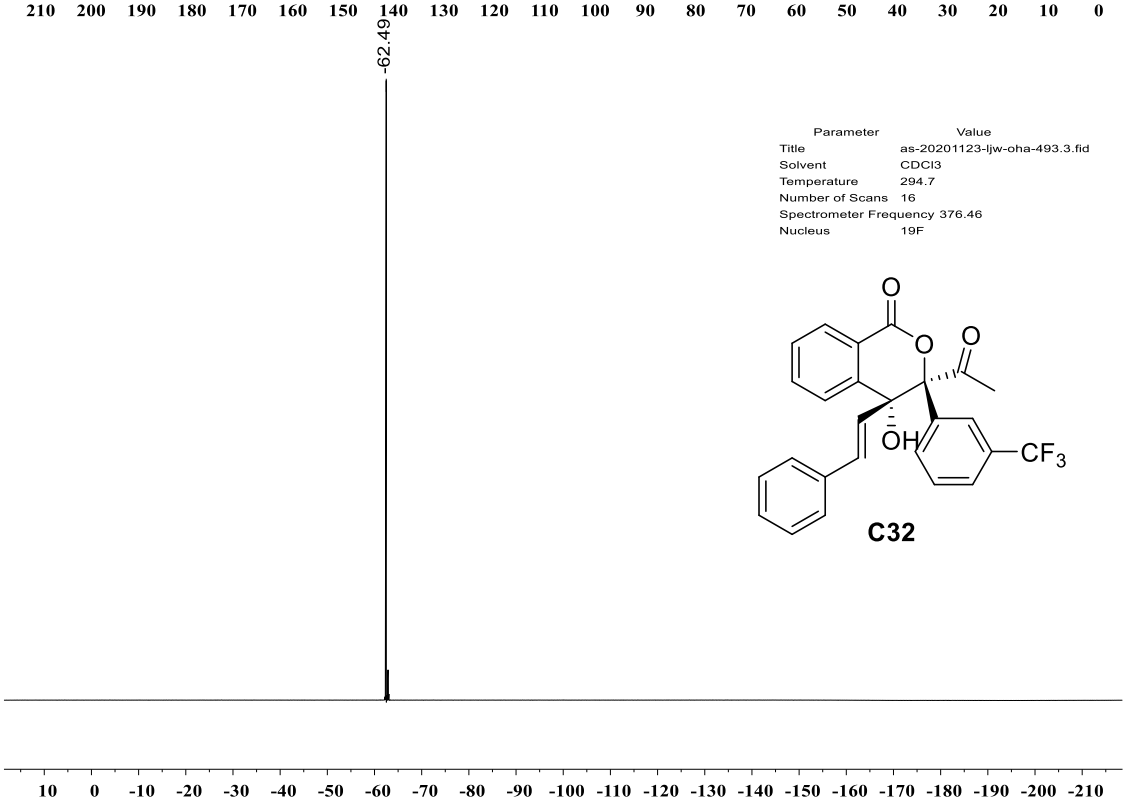
129.9
129.9
128.8
128.4
128.1
127.9
127.0
126.6
126.1
126.1
126.1
126.0
125.1
125.1
123.7
123.7
123.6
123.6
122.4
121.7
90.0
76.8
27.6
Parameter Value
Title as-20201123-ljw-oha-493.2.fid
Solvent CDCl3
Temperature 294.9
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
136 134 132 130
129 128 127 126 125 124 123 122
76.8
77.4 77.2 77.0 76.8 76.6 76.4
O
O
O
OH
CF3
C32
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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Number of Scans 16
Spectrometer Frequency 376.46
Nucleus 19F
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C32
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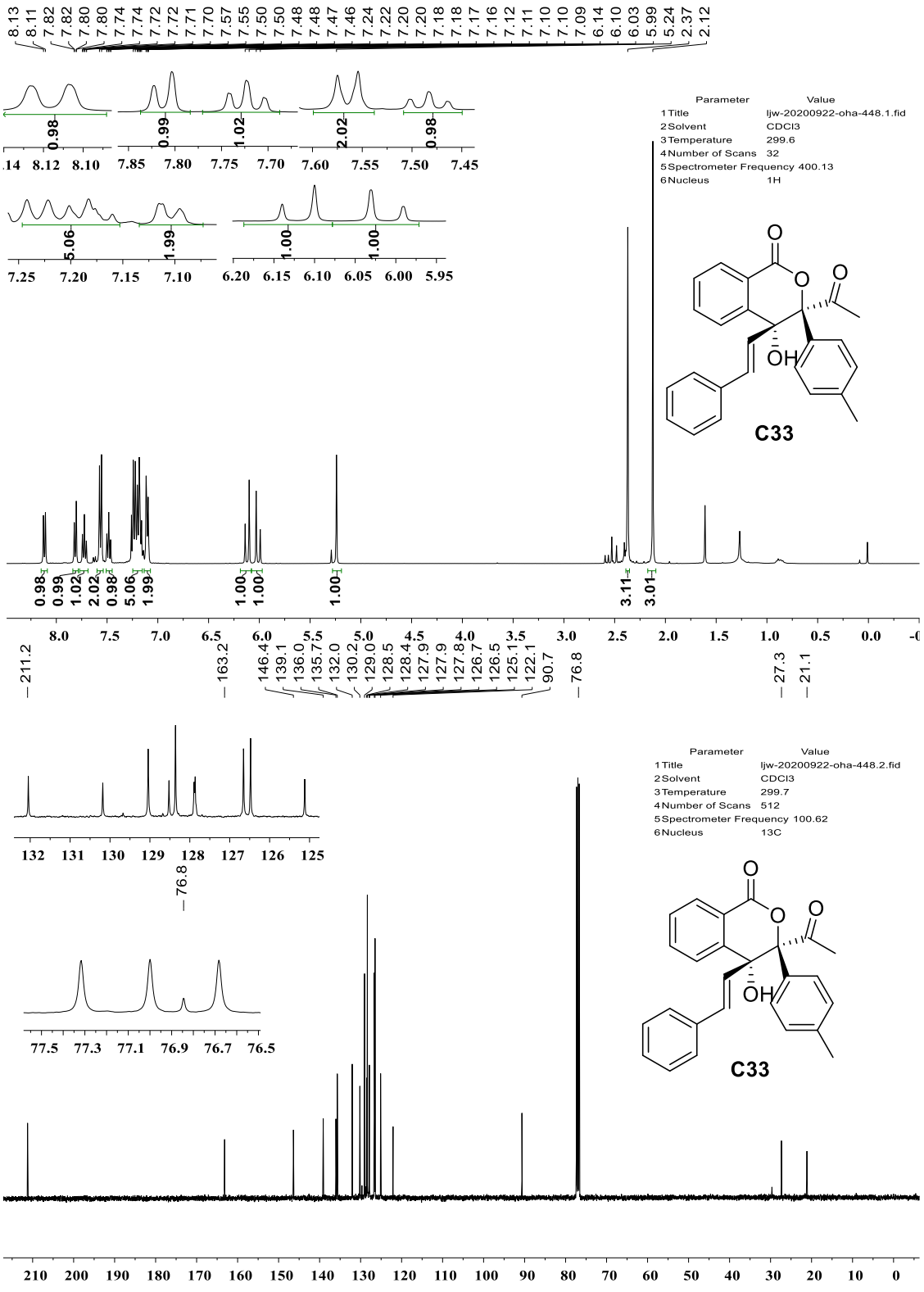
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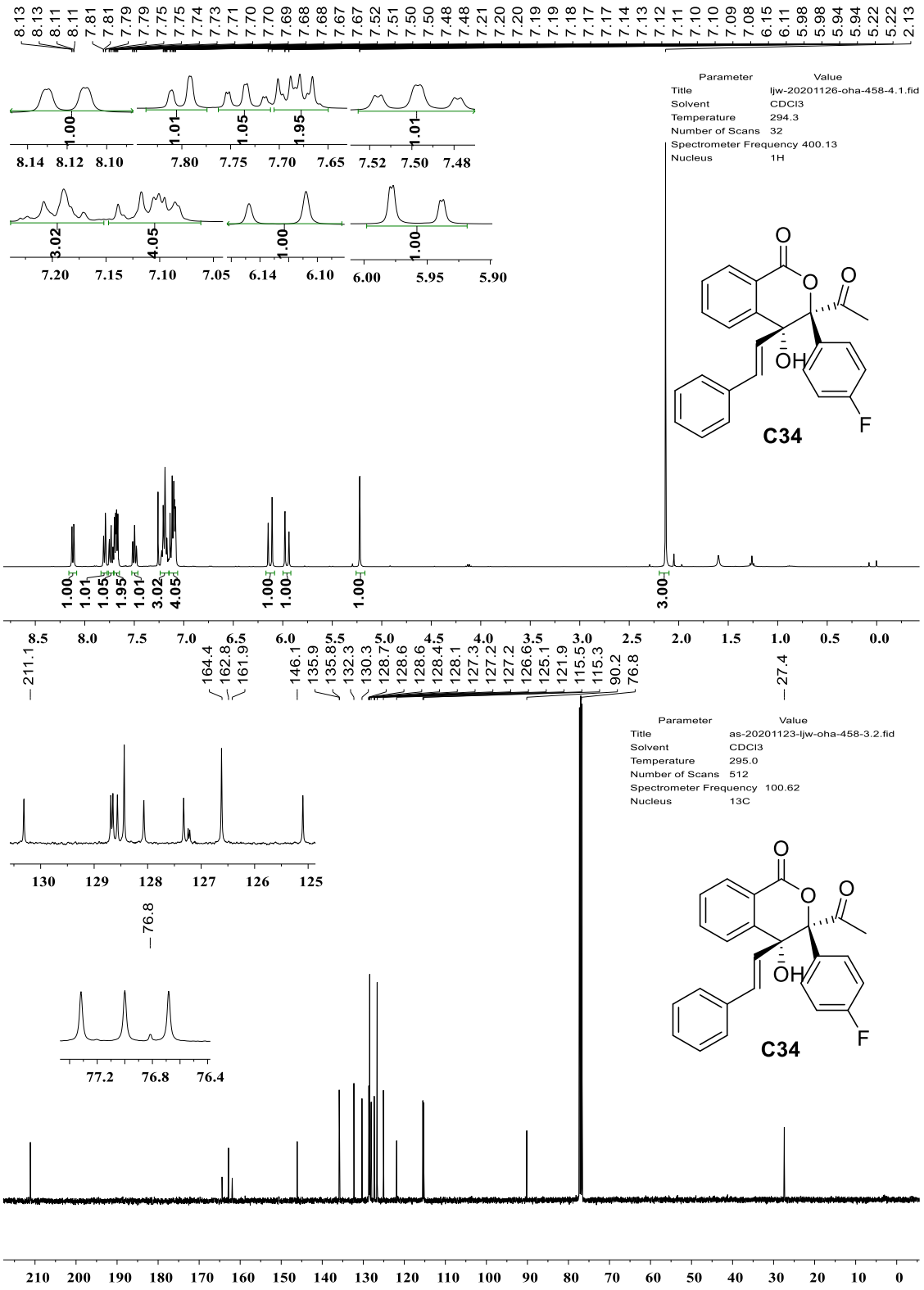
1 Title ljw-20200922-oha-448.1.fid
2 Solvent CDCl3
3 Temperature 299.6
4 Number of Scans 32
5 Spectrometer Frequency 400.13
6 Nucleus 1H
C33
Parameter Value
1 Title ljw-20200922-oha-448.2.fid
2 Solvent CDCl3
3 Temperature 299.7
4 Number of Scans 512
5 Spectrometer Frequency 100.62
6 Nucleus 13C
C33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Temperature 294.3
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C34
Parameter Value
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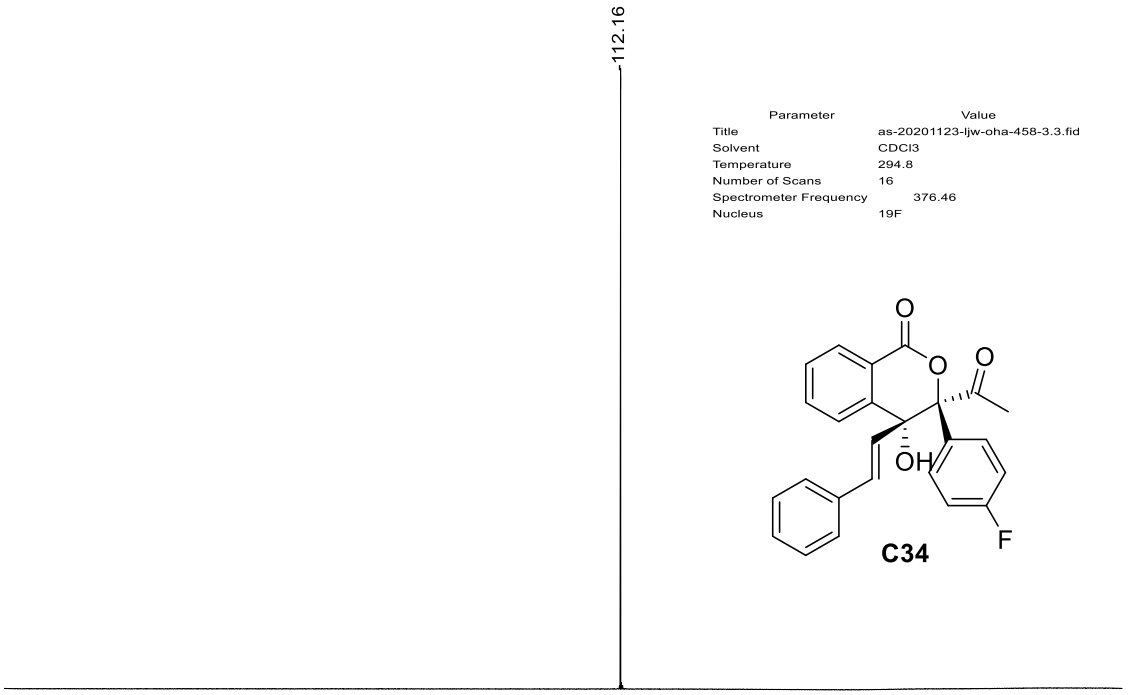
Solvent CDCl3
Temperature 295.0
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
C34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Temperature 294.8
Number of Scans 16
Spectrometer Frequency 376.46
Nucleus 19F
O
O
O
OH
F
C34
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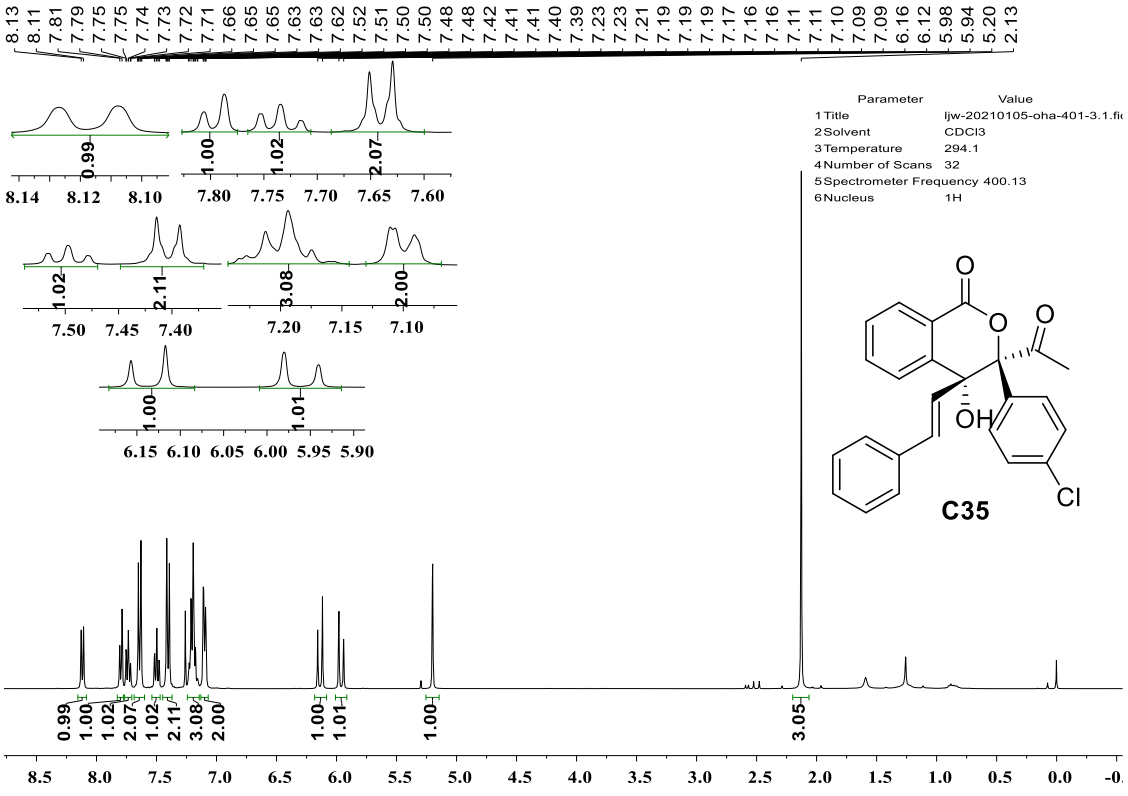
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0.99 1.00 1.02 2.07
8.14 8.12 8.10 7.80 7.75 7.70 7.65 7.60
1.02 2.11 3.08 2.00
7.50 7.45 7.40 7.20 7.15 7.10
1.00 1.01
6.15 6.10 6.05 6.00 5.95 5.90
Parameter Value
1 Title ljw-20210105-oha-401-3.1.fi
2 Solvent CDCl3
3 Temperature 294.1
4 Number of Scans 32
5 Spectrometer Frequency 400.13
6 Nucleus 1H
O
O
O
OH
Cl
C35
0.99 1.00 1.02 2.07 1.02 2.11 3.08 2.00 1.00 1.01 1.00 3.05
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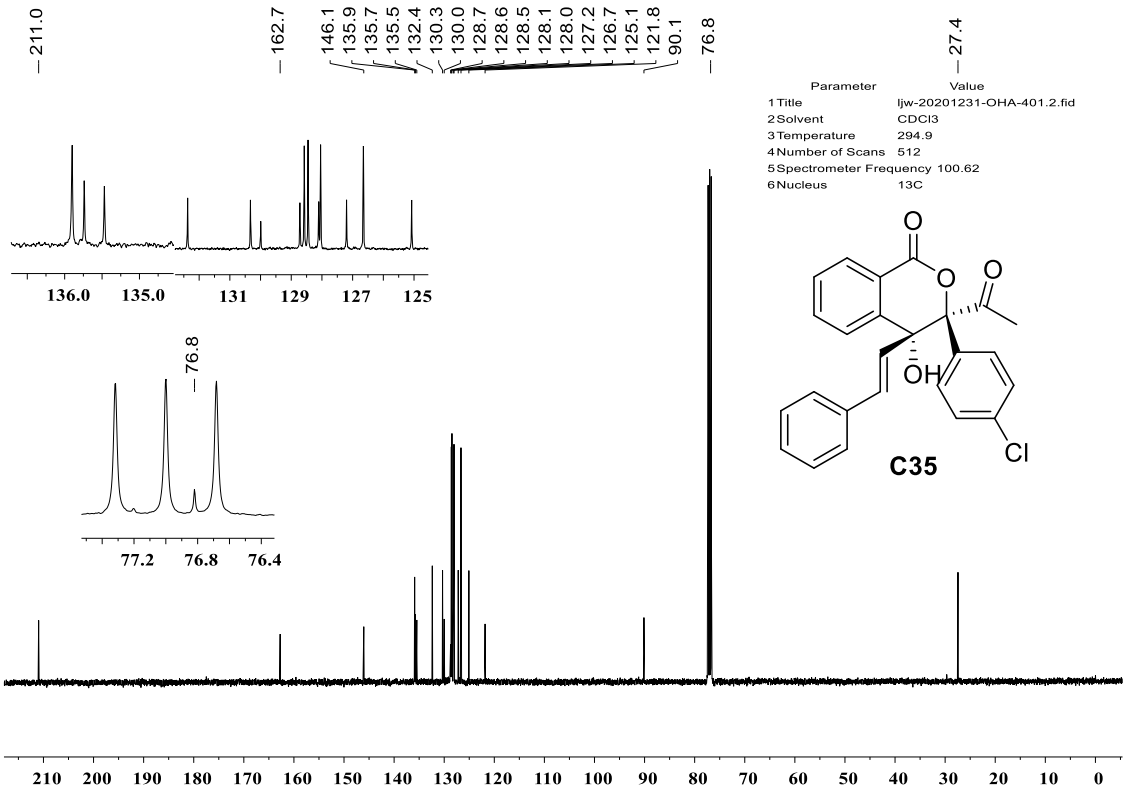
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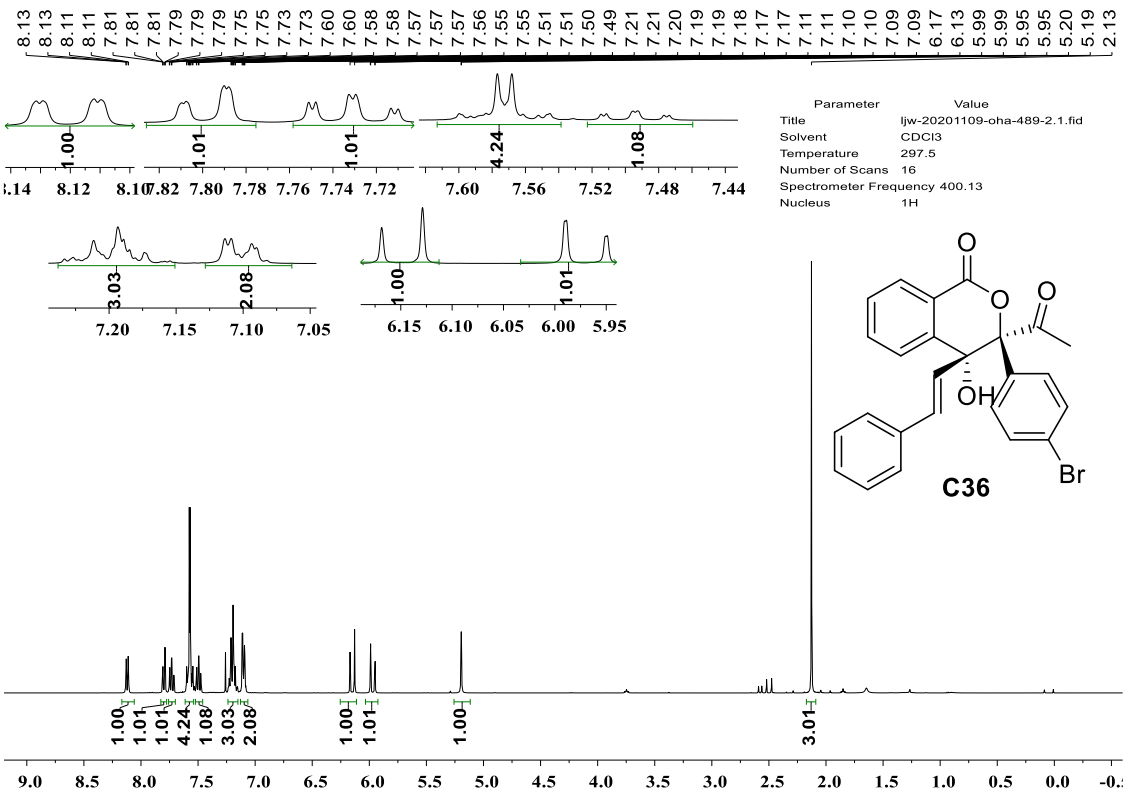
135.5
132.4
130.3
130.0
128.7
128.6
128.5
128.1
128.0
127.2
126.7
125.1
121.8
90.1
76.8
27.4
Parameter Value
1 Title ljw-20201231-OHA-401.2.fid
2 Solvent CDCl3
3 Temperature 294.9
4 Number of Scans 512
5 Spectrometer Frequency 100.62
6 Nucleus 13C
O
O
O
OH
Cl
C35
136.0 135.0 131 129 127 125
77.2 76.8 76.4
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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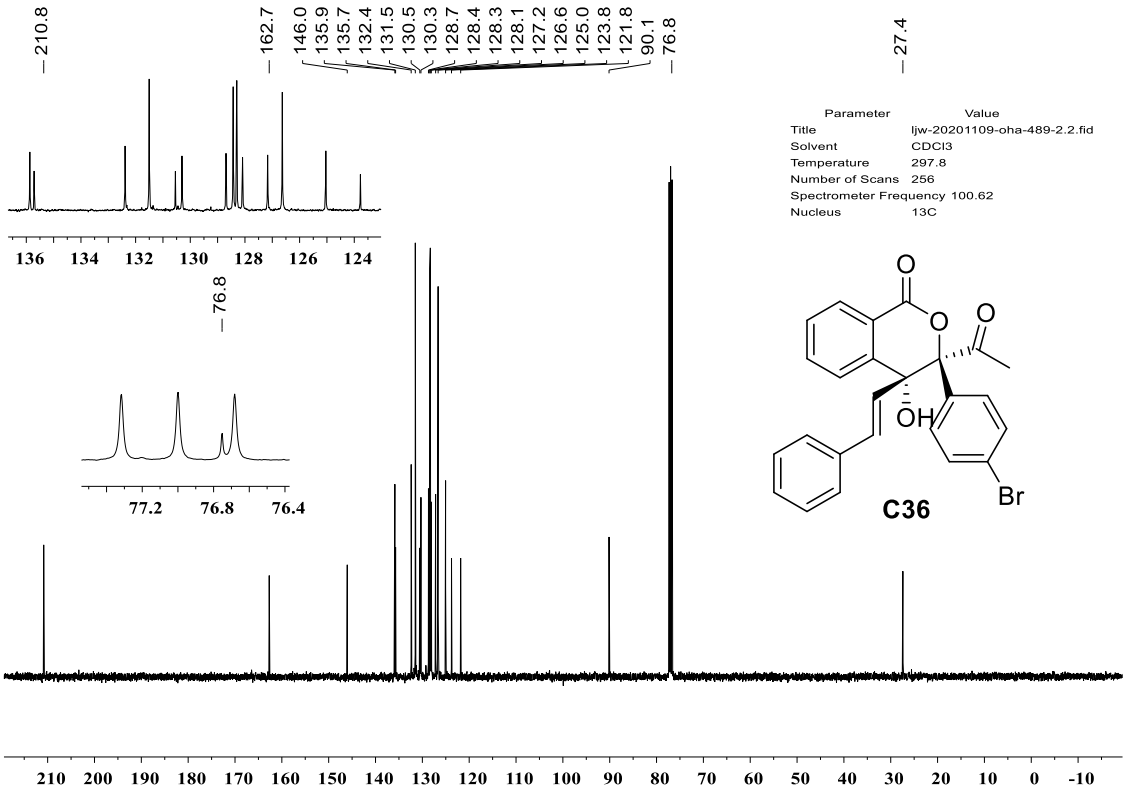
Parameter Value
Title ljw-20201109-oha-489-2.1.fid
Solvent CDCl3
Temperature 297.5
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
1.00 1.01 1.01 4.24 1.08
7.60 7.56 7.52 7.48 7.44
3.03 2.08
7.20 7.15 7.10 7.05
1.00 1.01
6.15 6.10 6.05 6.00 5.95
O
O
O
OH
Br
C36
1.00 1.01 1.01 4.24 1.08 3.03 2.08 1.00 1.01 1.00 3.01
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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135.9
135.7
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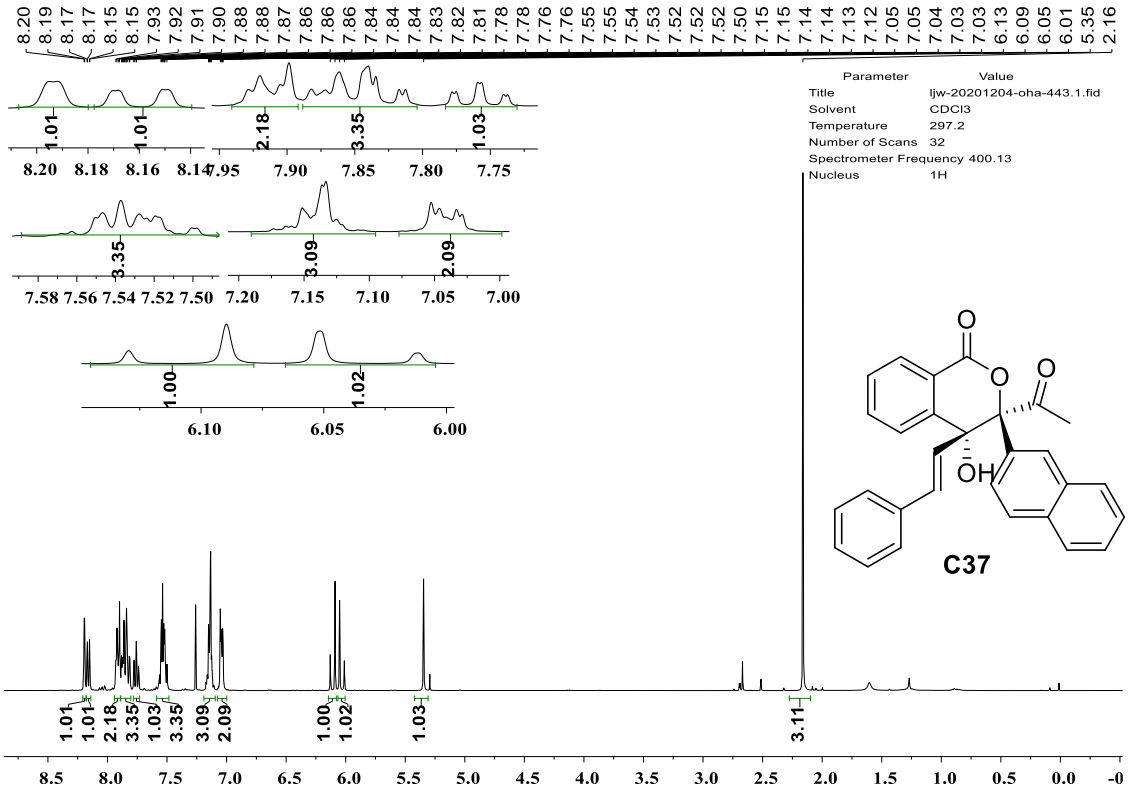
131.5
130.5
130.3
128.7
128.4
128.3
128.1
127.2
126.6
125.0
123.8
121.8
90.1
76.8
27.4
Parameter Value
Title ljw-20201109-oha-489-2.2.fid
Solvent CDCl3
Temperature 297.8
Number of Scans 256
Spectrometer Frequency 100.62
Nucleus 13C
C36
Br
OH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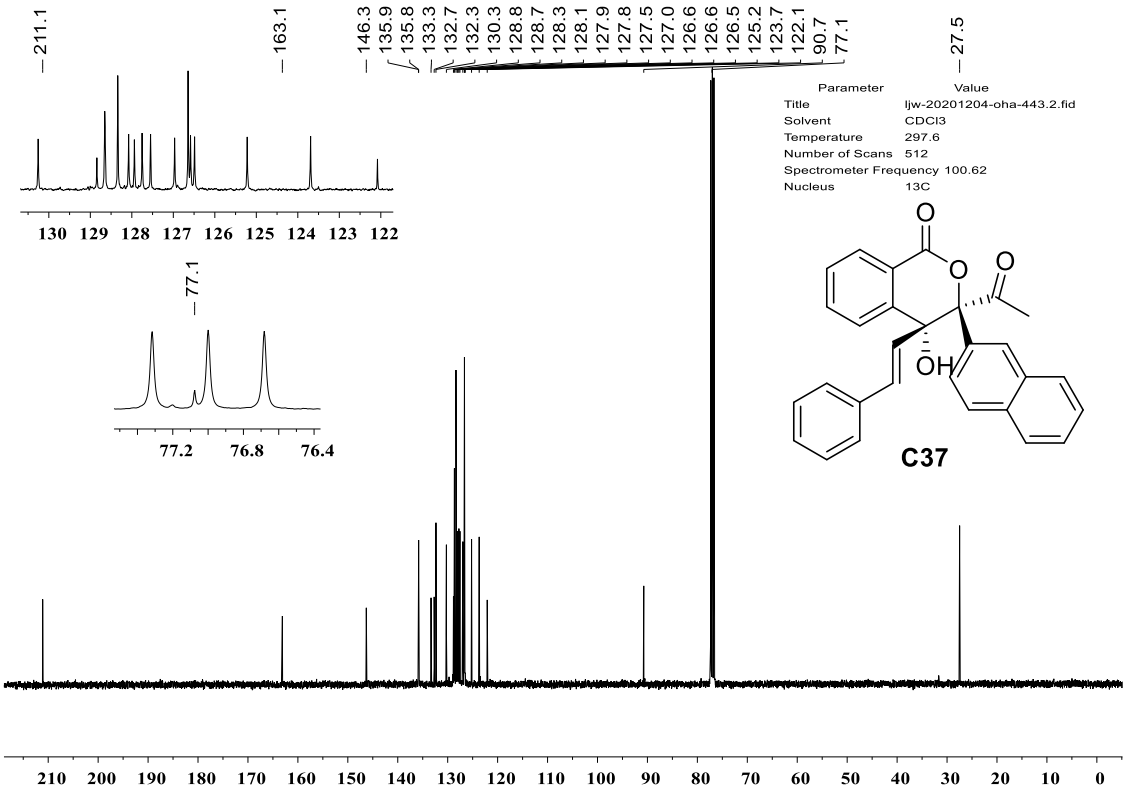
Temperature 297.2
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C37
OH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132.7
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128.8
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128.3
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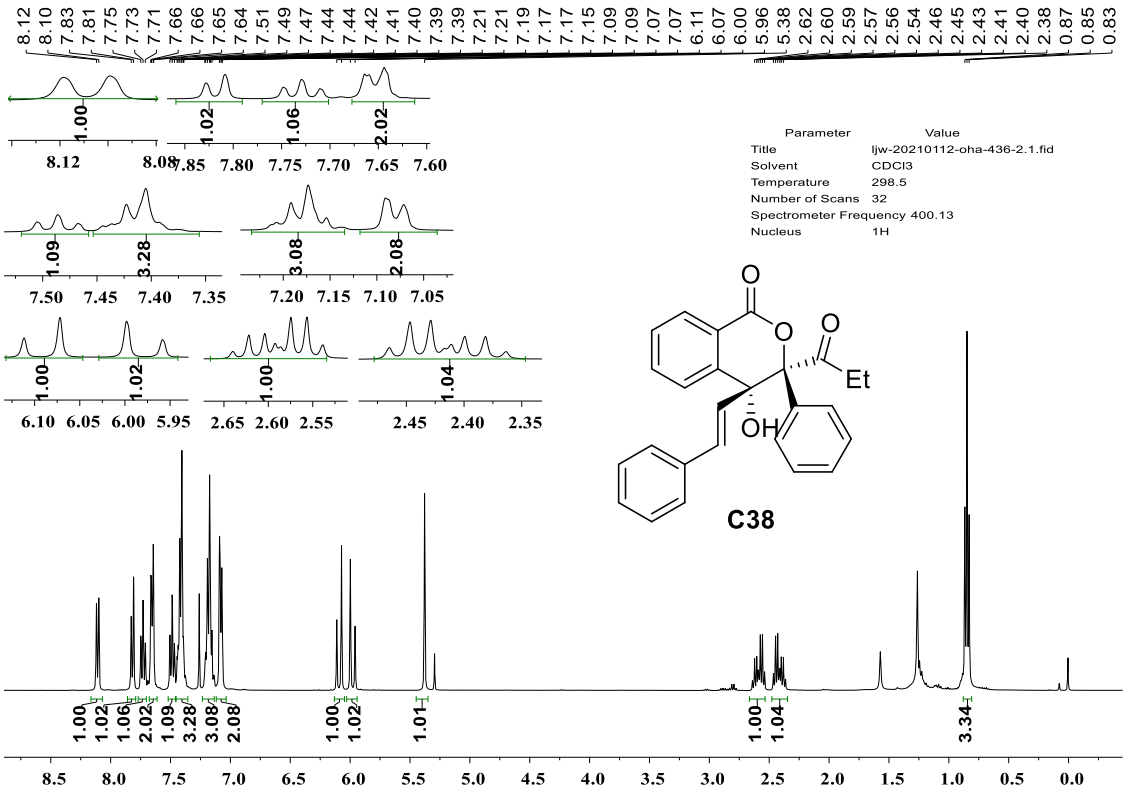
127.9
127.8
127.5
127.0
126.6
126.6
126.5
125.2
123.7
122.1
90.7
77.1
27.5
Parameter Value
Title ljw-20201204-oha-443.2.fid
Solvent CDCl3
Temperature 297.6
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
OH
C37
77.1
77.2 76.8 76.4
130 129 128 127 126 125 124 123 122
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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7.64
7.51
7.49
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7.44
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7.42
7.41
7.40
7.39
7.39
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7.19
7.17
7.17
7.15
7.09
7.09
7.07
7.07
6.11
6.07
6.00
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2.62
2.60
2.59
2.57
2.56
2.54
2.46
2.45
2.43
2.41
2.40
2.38
0.87
0.85
0.83
Parameter Value
Title ljw-20210112-oha-436-2.1.fid
Solvent CDCl3
Temperature 298.5
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
O
O
O
Et
OH
C38
1.00 1.02 1.06 2.02 1.09 3.28 3.08 2.08 1.00 1.02 1.01 1.00 1.04 3.34
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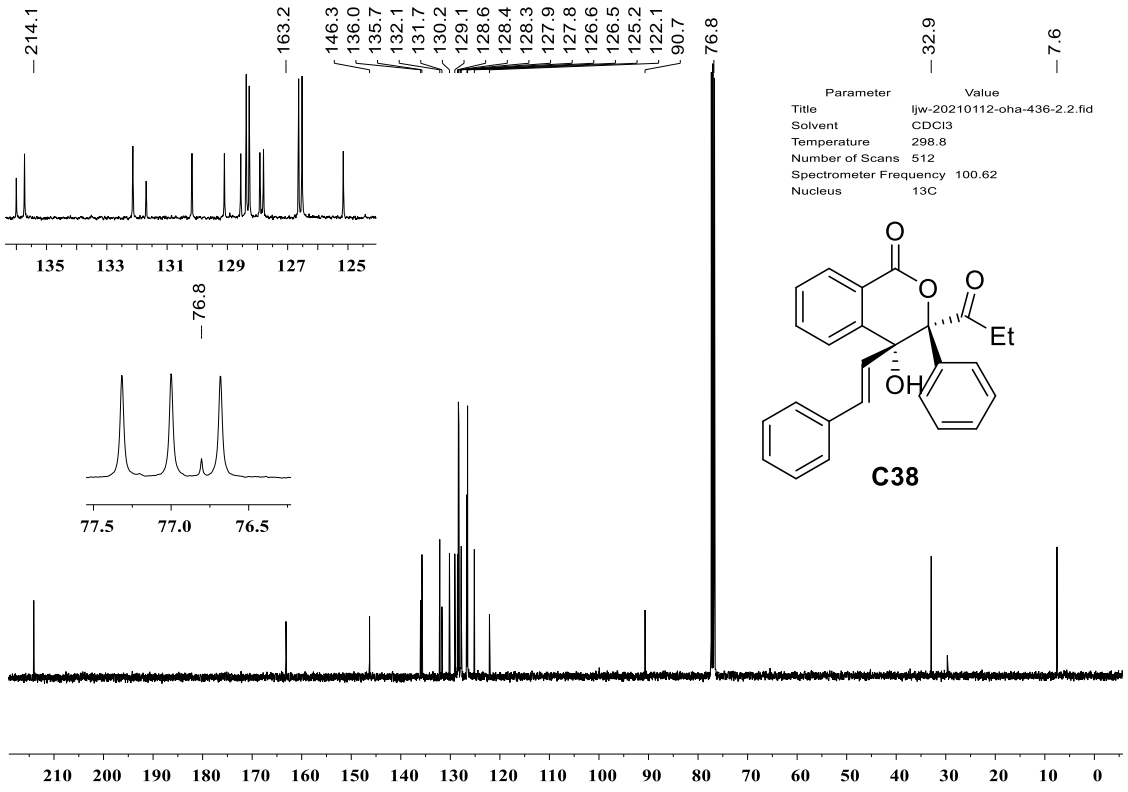
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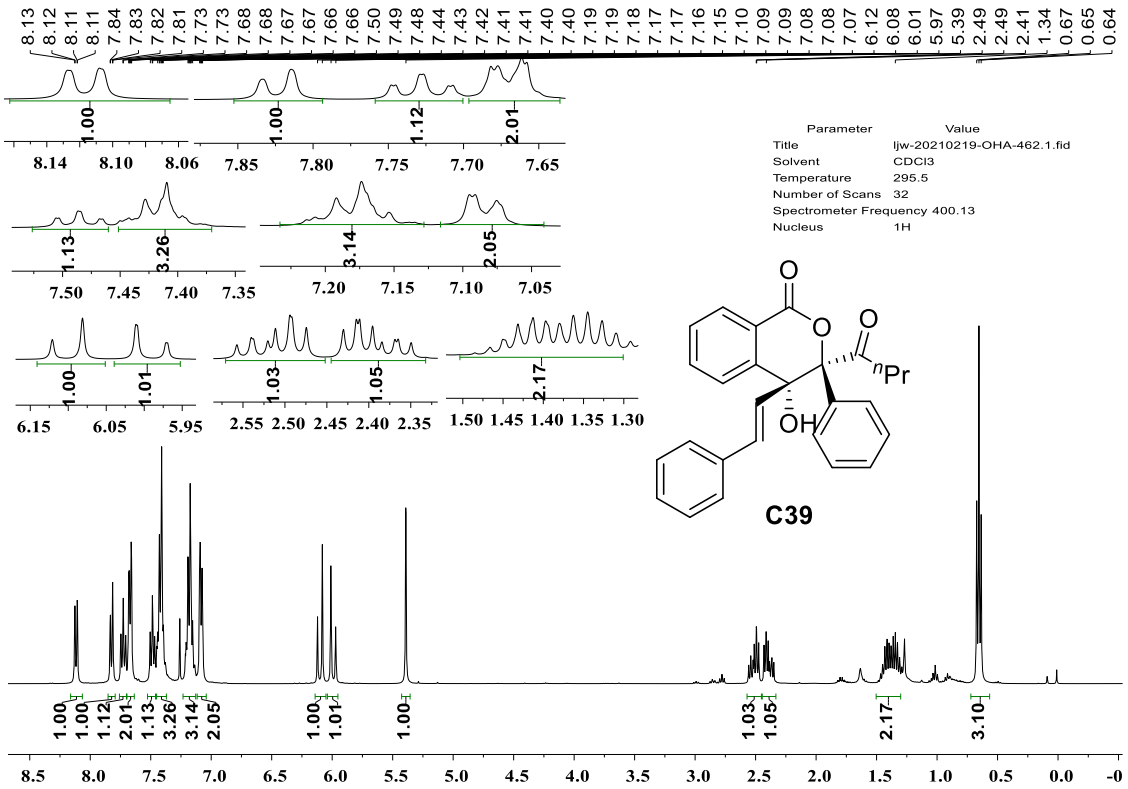
128.6
128.4
128.3
127.9
127.8
126.6
126.5
125.2
122.1
90.7
76.8
32.9
7.6
Parameter Value
Title ljw-20210112-oha-436-2.2.fid
Solvent CDCl3
Temperature 298.8
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
Et
OH
C38


8.13 8.12 8.11 8.11 7.84 7.83 7.82 7.81 7.73 7.73 7.68 7.68 7.67 7.67 7.66 7.66 7.50 7.49 7.48 7.44 7.43 7.42 7.41 7.41 7.40 7.40 7.19 7.19 7.18 7.17 7.17 7.16 7.15 7.10 7.09 7.09 7.08 7.08 7.07 6.12 6.08 6.01 5.97 5.39 2.49 2.49 2.41 1.34 0.67 0.65 0.64
Parameter Value
Title ljw-20210219-OHA-462.1.fid
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Temperature 295.5
Number of Scans 32
Spectrometer Frequency 400.13
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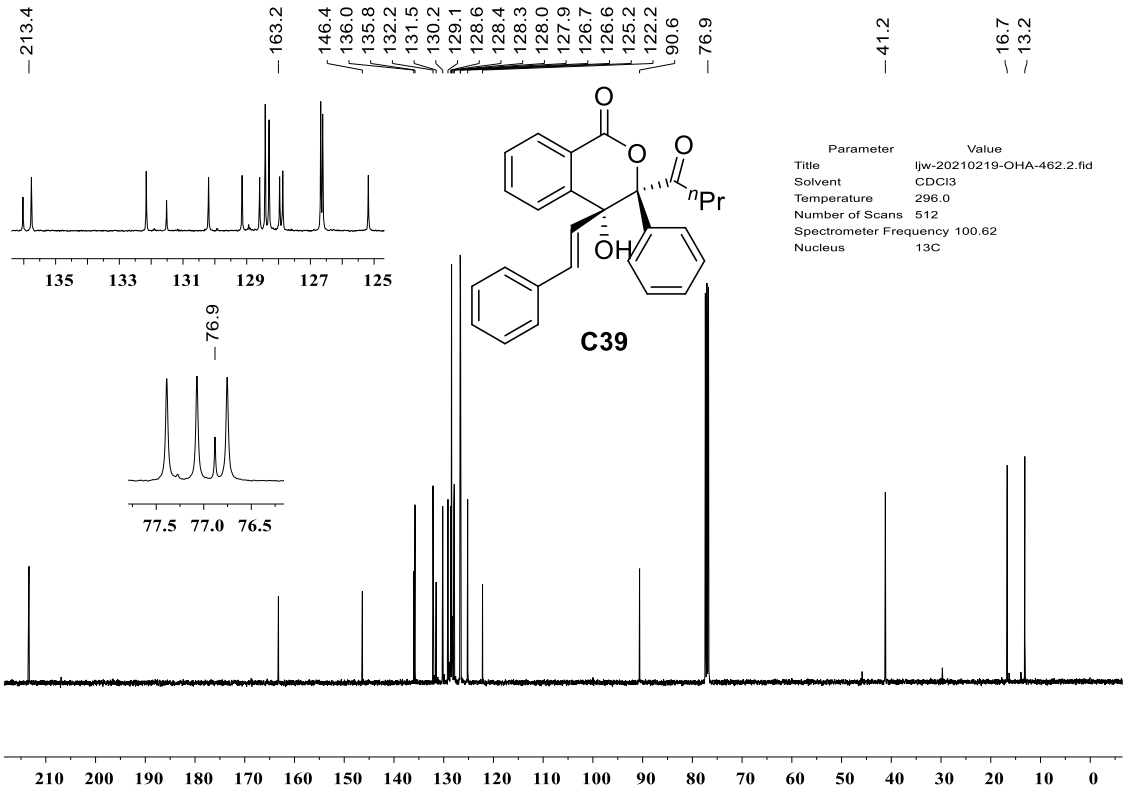
O
nPr
OH
C39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128.6
128.4
128.3
128.0
127.9
126.7
126.6
125.2
122.2
90.6
76.9
41.2
16.7
13.2
Parameter Value
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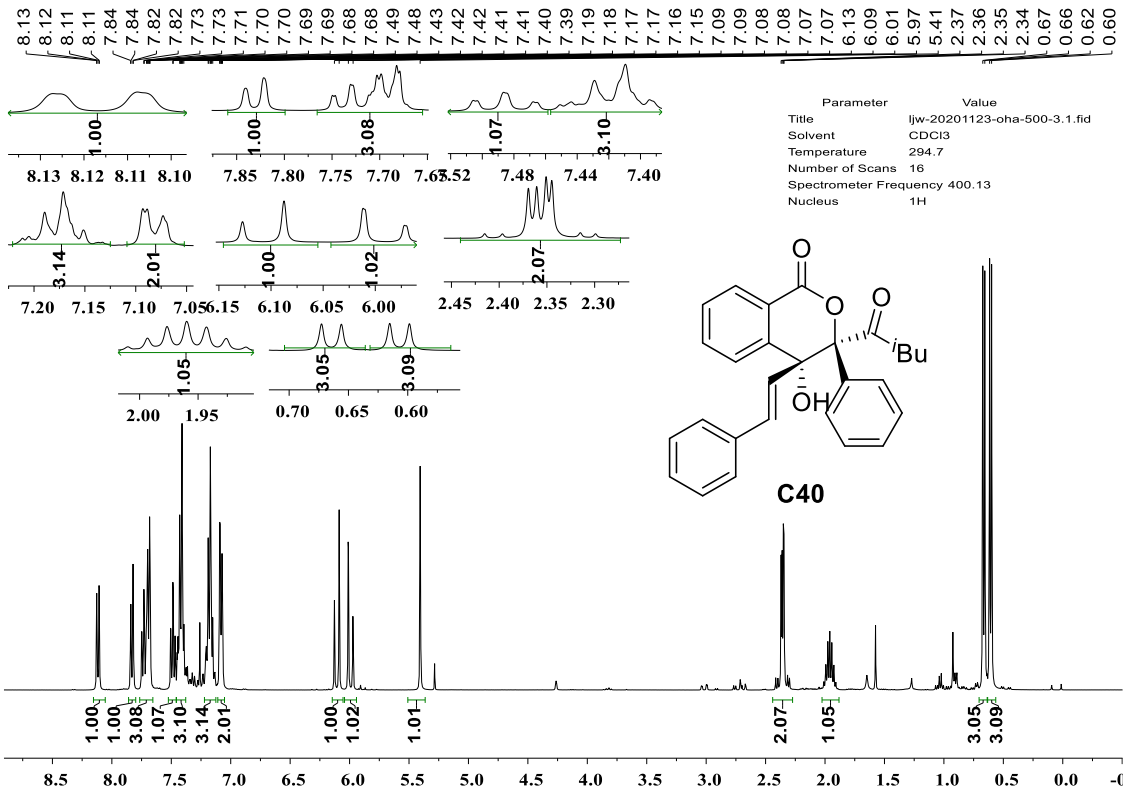
Temperature 296.0
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
C39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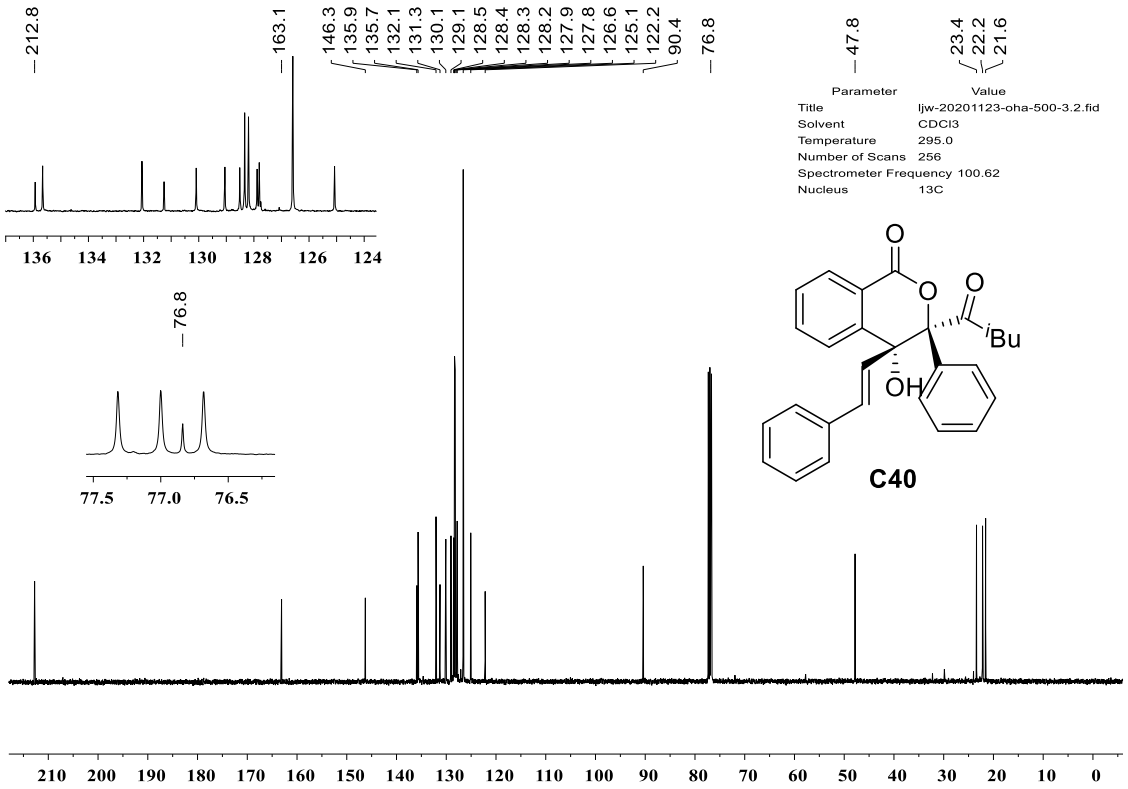
Solvent CDCl3
Temperature 294.7
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
C40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128.5
128.4
128.3
128.2
127.9
127.8
126.6
125.1
122.2
90.4
76.8
47.8
23.4
22.2
21.6
Parameter Value
Title ljw-20201123-oha-500-3.2.fid
Solvent CDCl3
Temperature 295.0
Number of Scans 256
Spectrometer Frequency 100.62
Nucleus 13C
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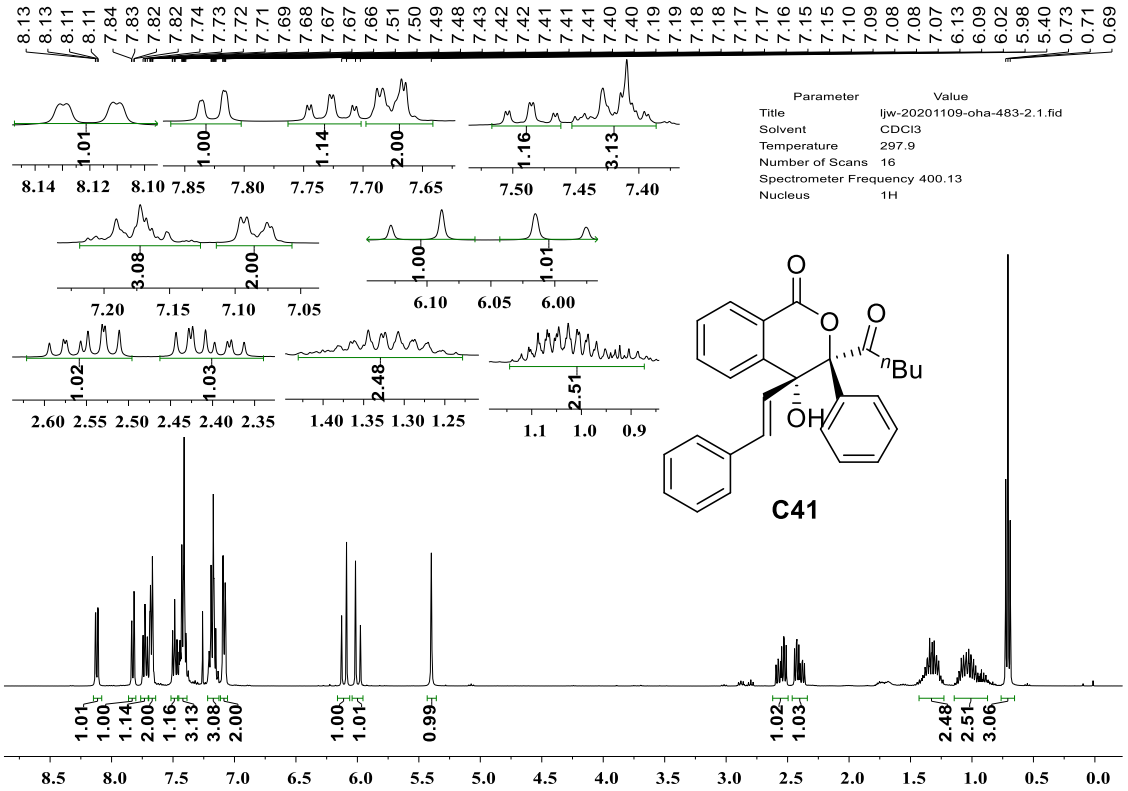
O
O
iBu
OH
C40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7.67
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7.66
7.51
7.50
7.49
7.48
7.43
7.42
7.42
7.41
7.41
7.41
7.40
7.40
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7.18
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7.10
7.09
7.08
7.08
7.07
6.13
6.09
6.02
5.98
5.40
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0.71
0.69
Parameter Value
Title ljw-20201109-oha-483-2.1.fid
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Temperature 297.9
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
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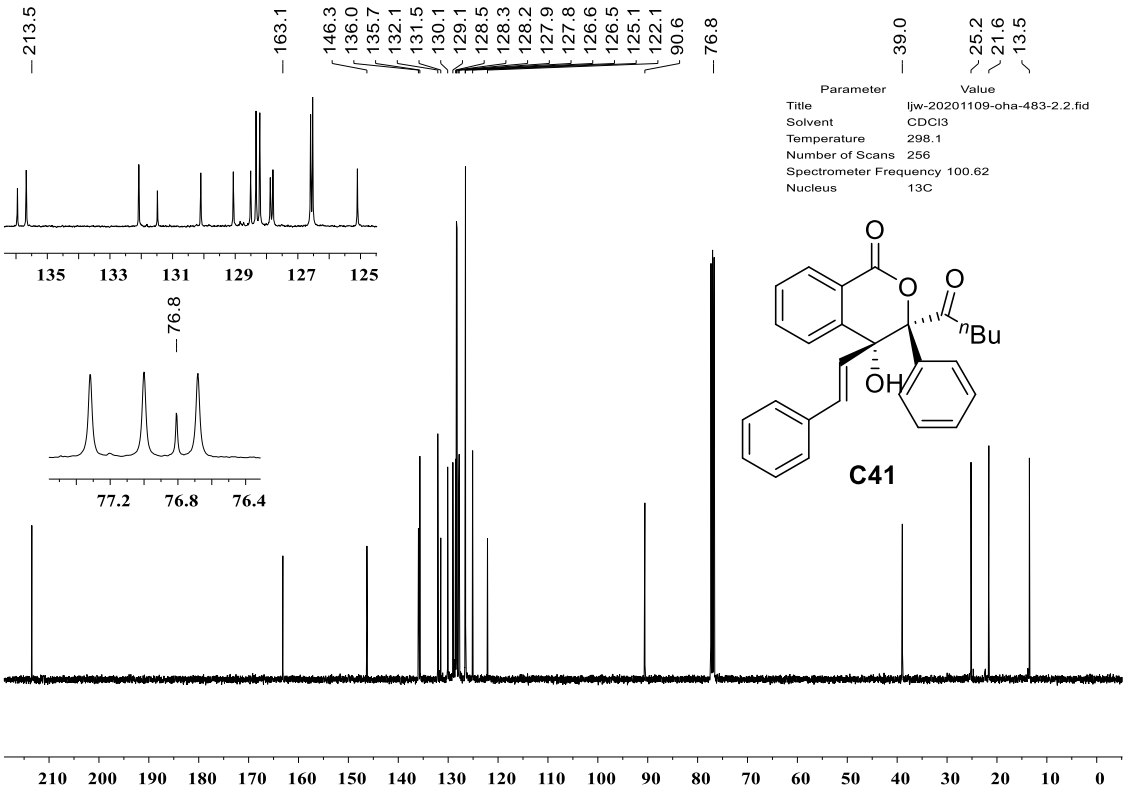
O
O
nBu
OH
C41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129.1
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128.3
128.2
127.9
127.8
126.6
126.5
125.1
122.1
90.6
76.8
39.0
25.2
21.6
13.5
Parameter Value
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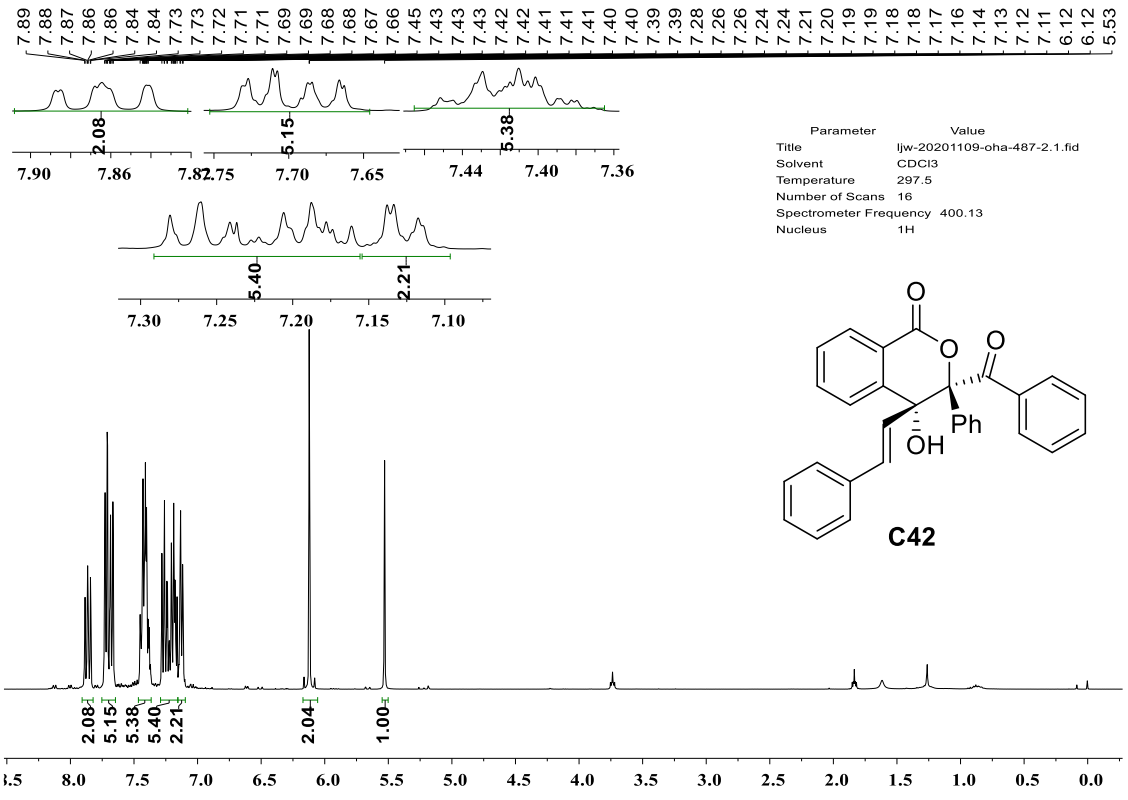
Solvent CDCl3
Temperature 298.1
Number of Scans 256
Spectrometer Frequency 100.62
Nucleus 13C
C41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7.41
7.41
7.40
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7.39
7.39
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7.24
7.24
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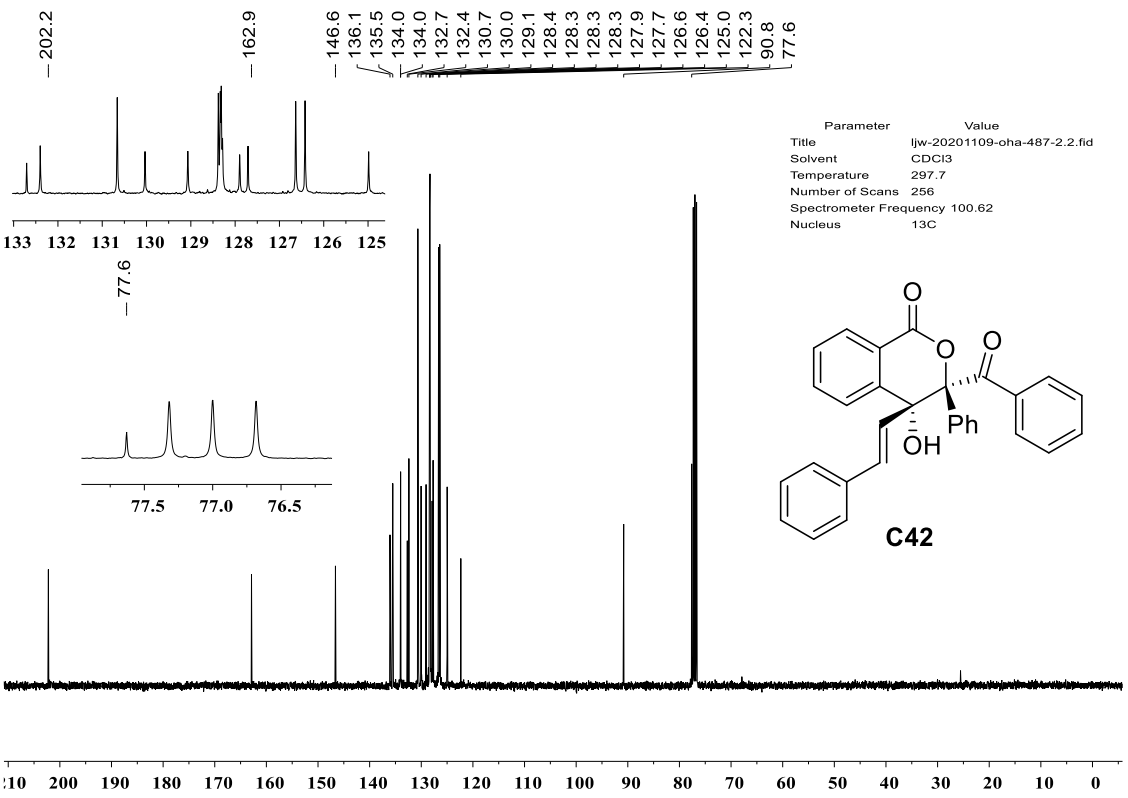
7.19
7.19
7.18
7.18
7.17
7.16
7.14
7.13
7.12
7.11
6.12
6.12
5.53
2.08
5.15
5.38
5.40
2.21
2.04
1.00
Parameter Value
Title ljw-20201109-oha-487-2.1.fid
Solvent CDCl3
Temperature 297.5
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
C42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134.0
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130.7
130.0
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128.4
128.3
128.3
128.3
127.9
127.7
126.6
126.4
125.0
122.3
90.8
77.6
Parameter Value
Title ljw-20201109-oha-487-2.2.fid
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Temperature 297.7
Number of Scans 256
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Nucleus 13C
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O
O
Ph
OH
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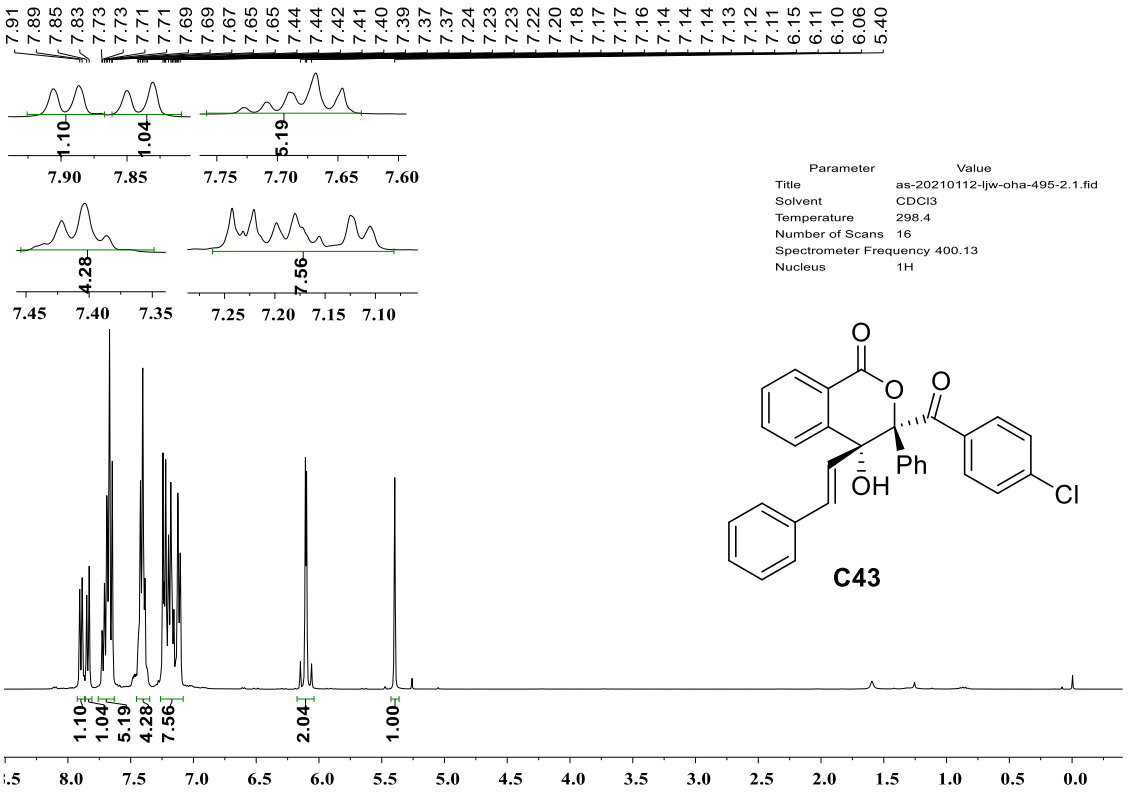
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7.44
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7.24
7.23
7.23
7.22
7.20
7.18
7.17
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7.16
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7.14
7.14
7.13
7.12
7.11
6.15
6.11
6.10
6.06
5.40
1.10
1.04
5.19
4.28
7.56
2.04
1.00
Parameter Value
Title as-20210112-ljw-oha-495-2.1.fid
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Temperature 298.4
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
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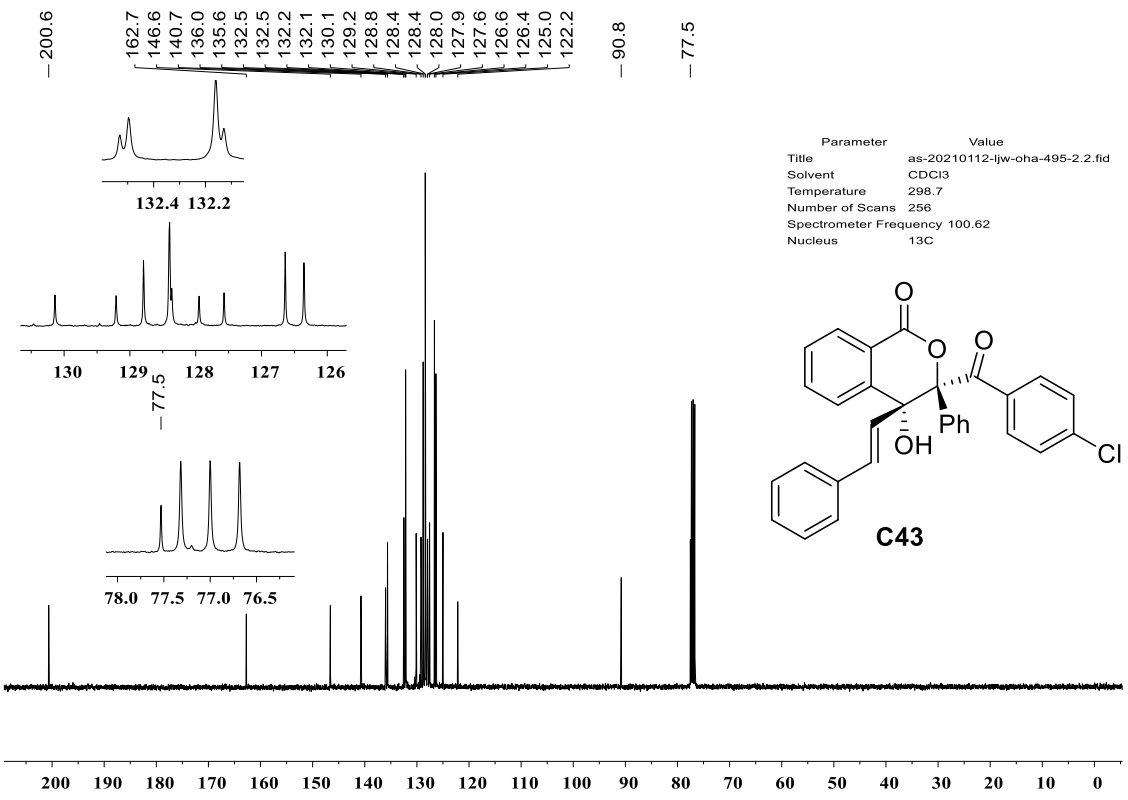
O
O
Ph
OH
Cl
C43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128.8
128.4
128.4
128.0
127.9
127.6
126.6
126.4
125.0
122.2
90.8
77.5
132.4 132.2
130 129 128 127 126
77.5
78.0 77.5 77.0 76.5
Parameter Value
Title as-20210112-ljw-oha-495-2.2.fid
Solvent CDCl3
Temperature 298.7
Number of Scans 256
Spectrometer Frequency 100.62
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O
O
O
Ph
OH
Cl
C43
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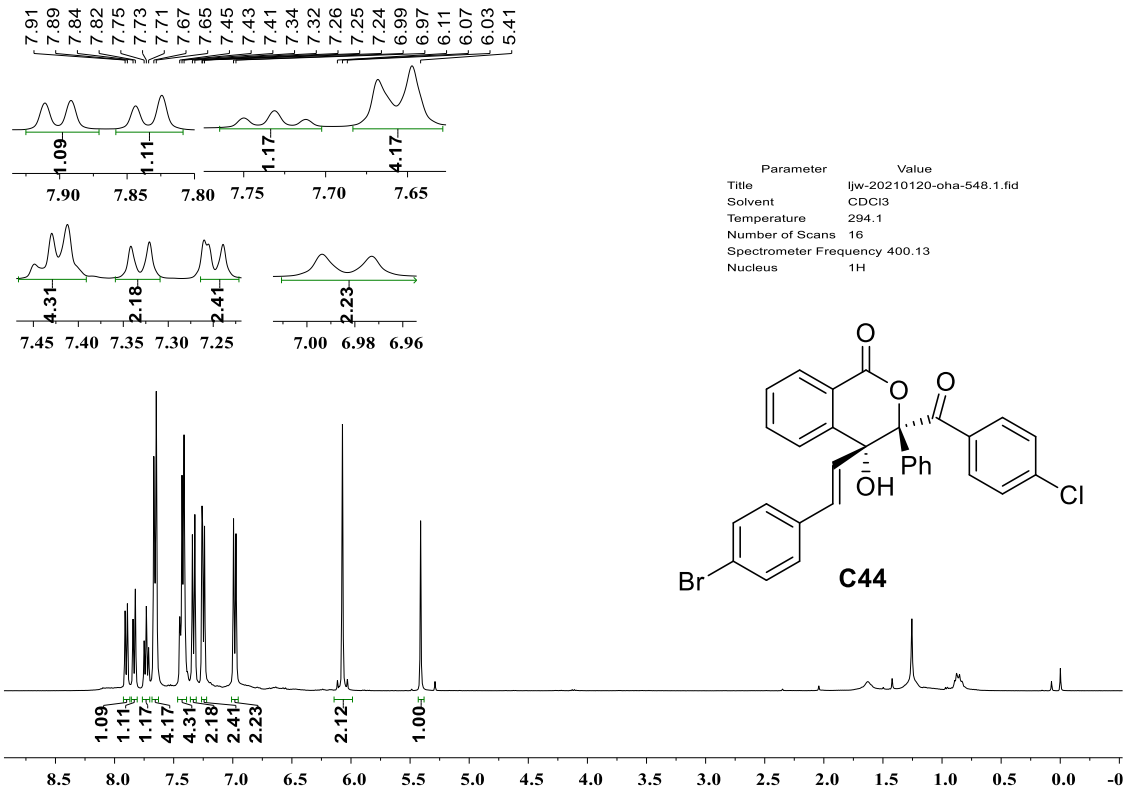
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7.34
7.32
7.26
7.25
7.24
6.99
6.97
6.11
6.07
6.03
5.41
1.09
1.11
1.17
4.17
7.90 7.85 7.80 7.75 7.70 7.65
4.31
2.18
2.41
2.23
7.45 7.40 7.35 7.30 7.25
7.00 6.98 6.96
Parameter Value
Title ljw-20210120-oha-548.1.fid
Solvent CDCl3
Temperature 294.1
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
O
O
O
Ph
OH
Cl
Br
C44
1.09
1.11
1.17
4.17
4.31
2.18
2.41
2.23
2.12
1.00
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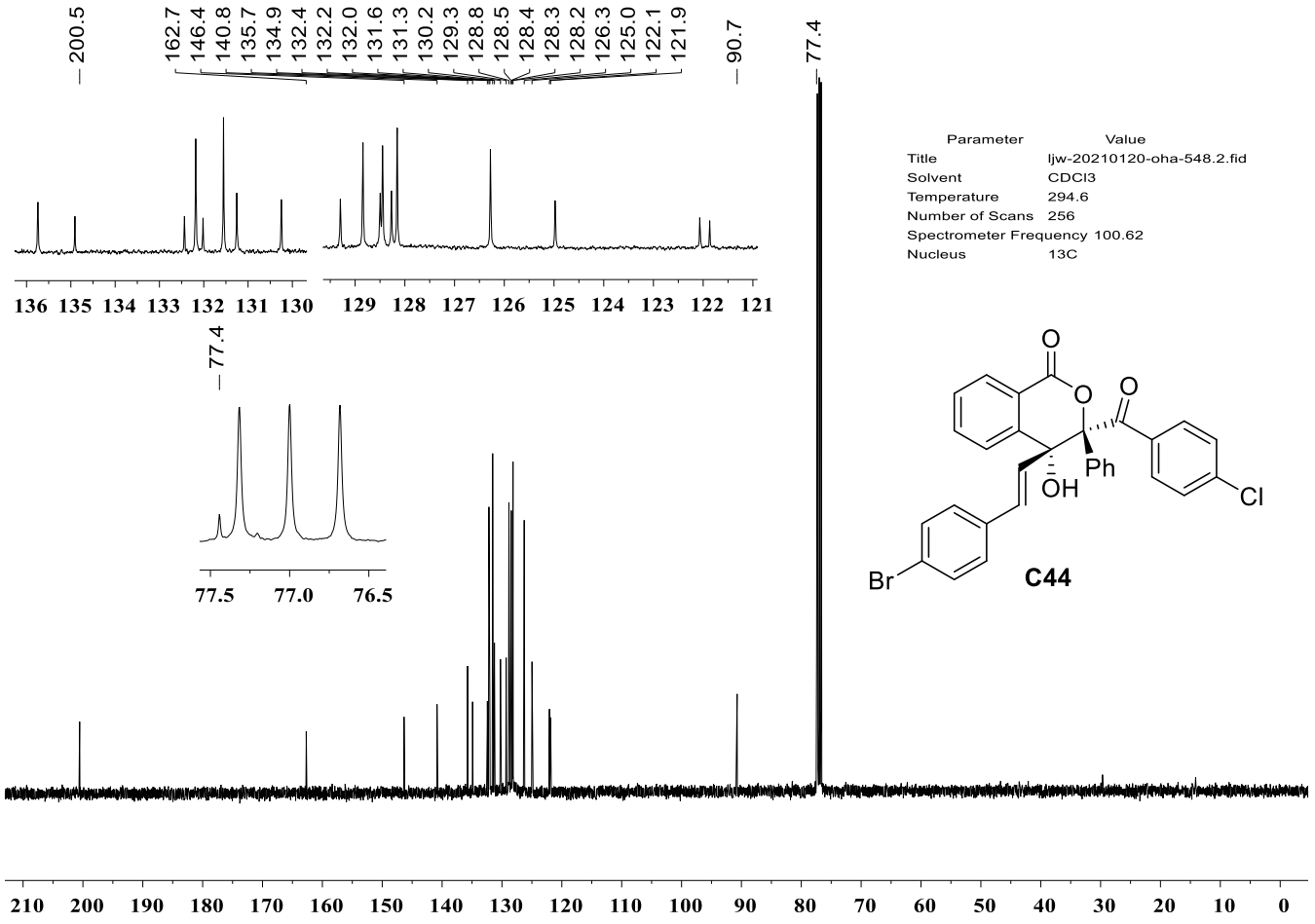
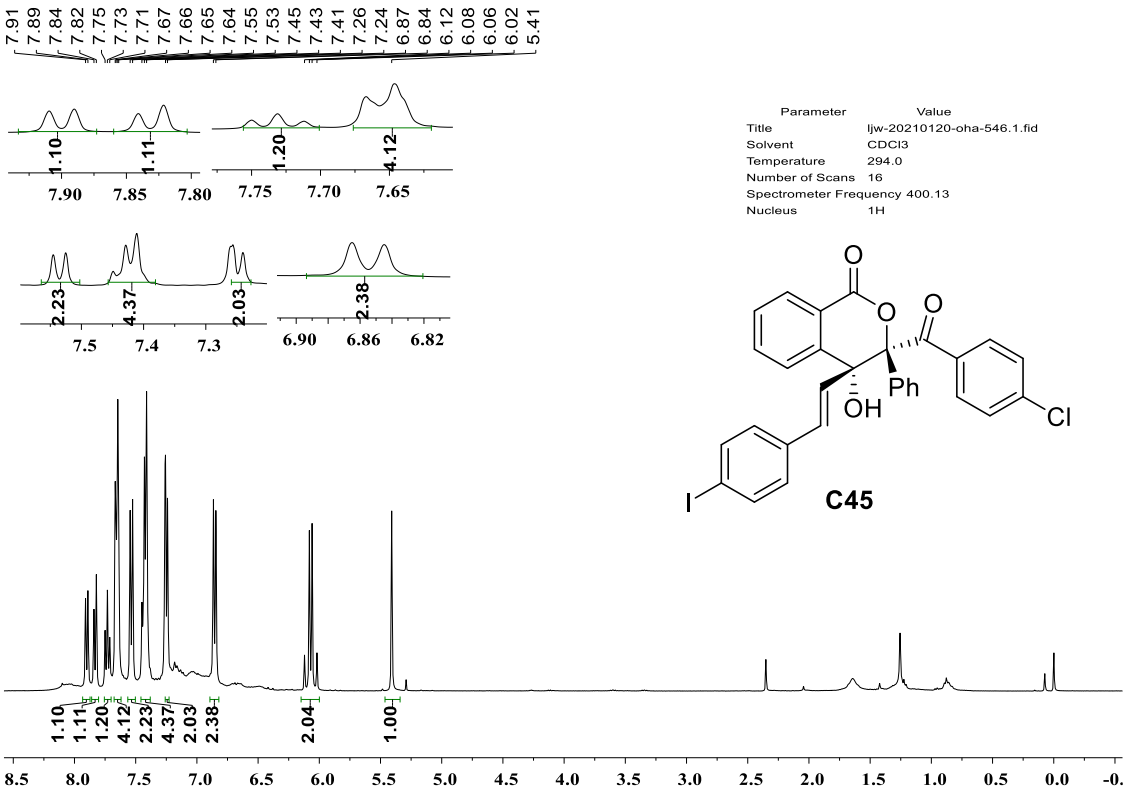


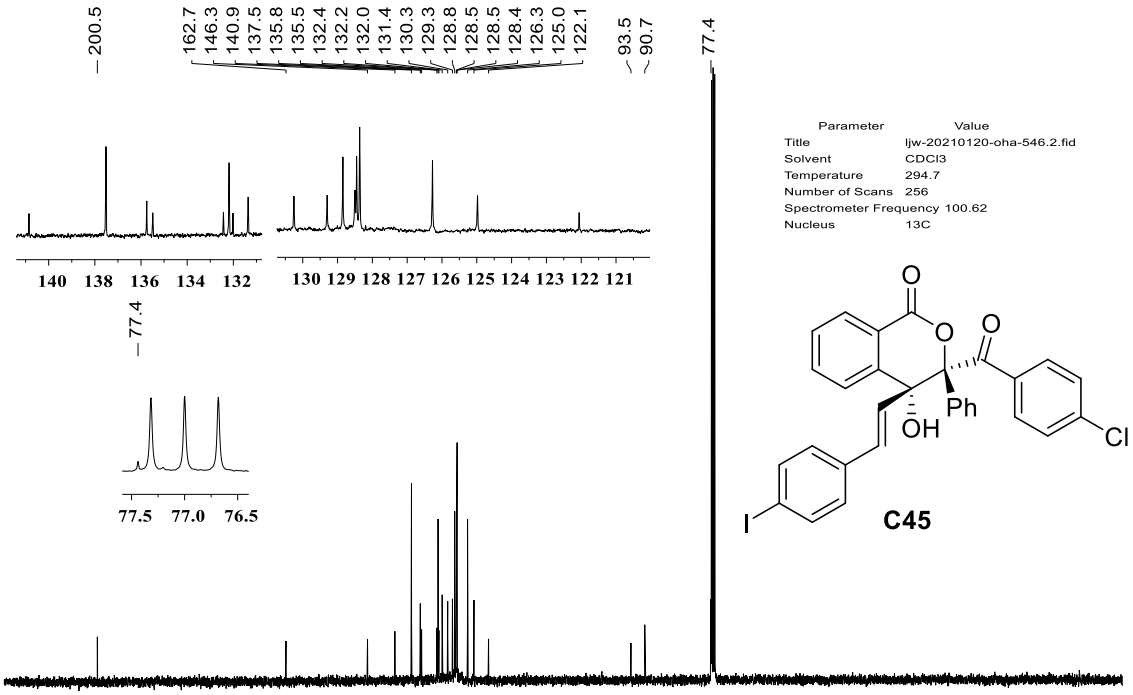
Parameter	Value
Title	ljw-20210120-oha-548.2.fid
Solvent	CDCl3
Temperature	294.6
Number of Scans	256
Spectrometer Frequency	100.62
Nucleus	13C

C44



Parameter	Value
Title	ljw-20210120-oha-546.1.fid
Solvent	CDCl3
Temperature	294.0
Number of Scans	16
Spectrometer Frequency	400.13
Nucleus	1H

C45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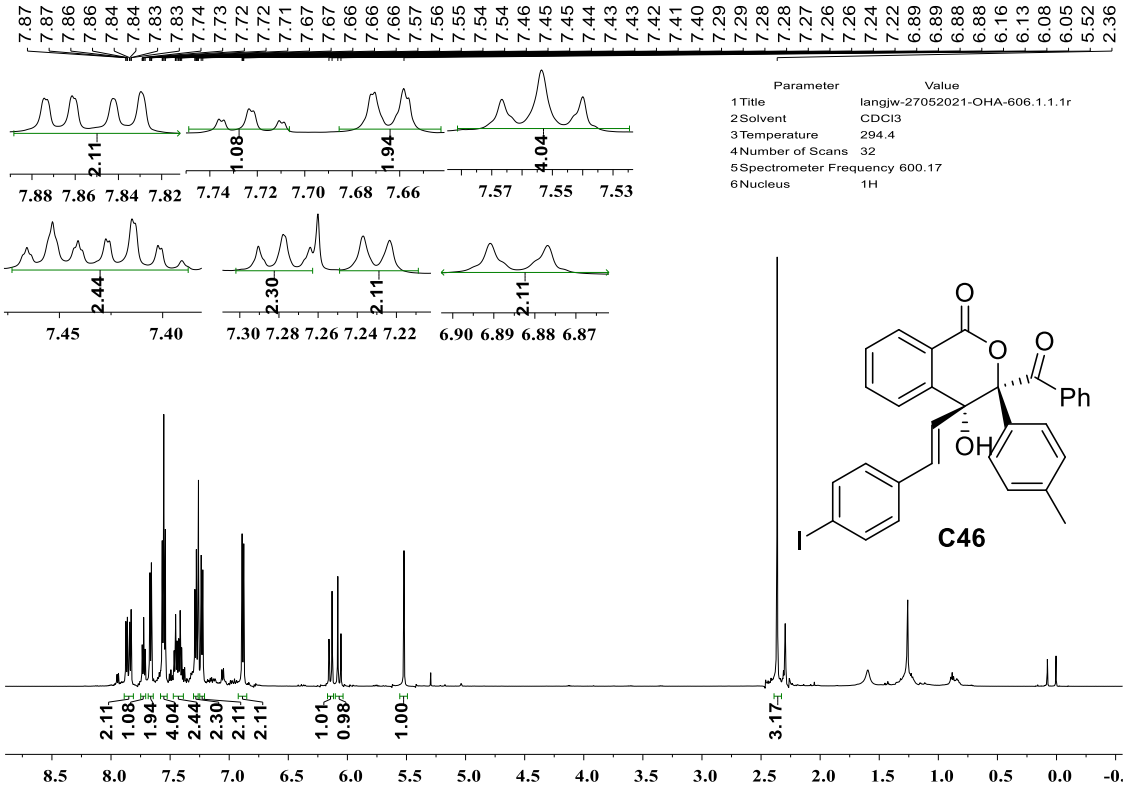
137.5
135.8
135.5
132.4
132.2
132.0
131.4
130.3
129.3
128.8
128.5
128.5
128.4
126.3
125.0
122.1
93.5
90.7
77.4
Parameter Value
Title ljw-20210120-oha-546.2.fid
Solvent CDCl3
Temperature 294.7
Number of Scans 256
Spectrometer Frequency 100.62
Nucleus 13C
O
O
O
Ph
OH
Cl
I
C45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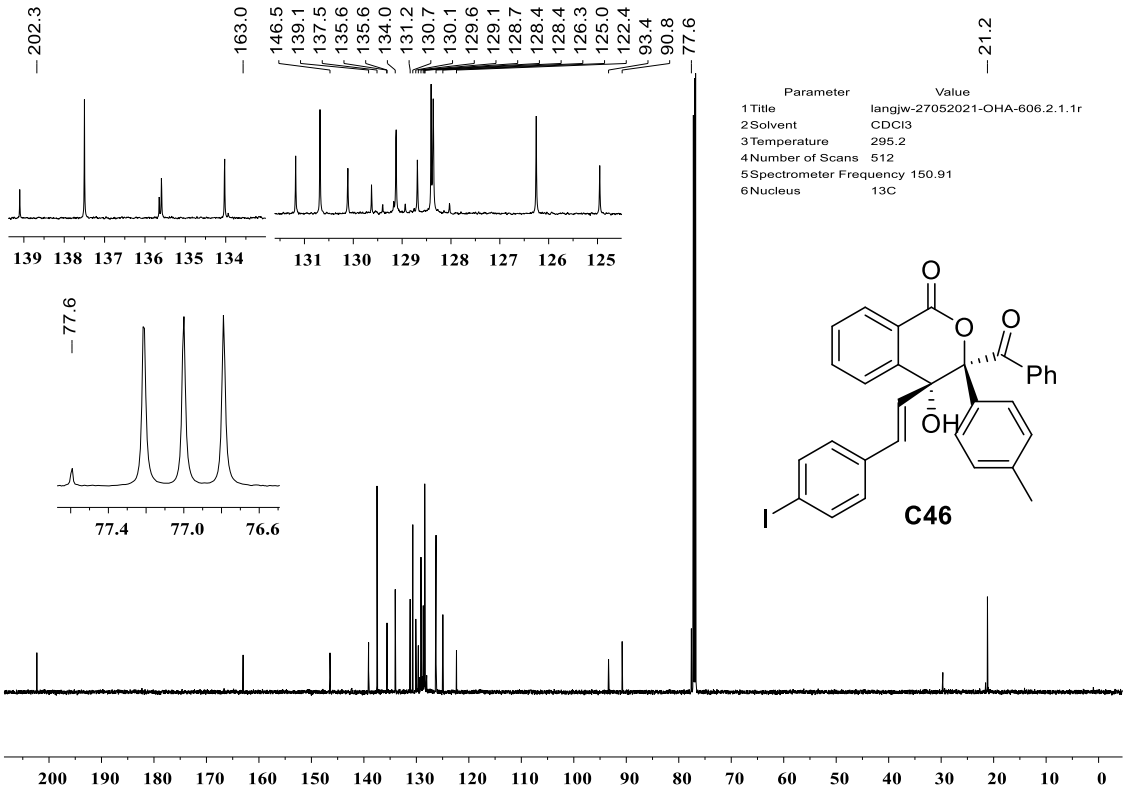
7.72
7.71
7.67
7.66
7.57
7.56
7.55
7.54
7.46
7.45
7.44
7.43
7.42
7.41
7.40
7.29
7.28
7.27
7.26
7.24
7.22
6.89
6.88
6.16
6.13
6.08
6.05
5.52
2.36
Parameter Value
1 Title langjw-27052021-OHA-606.1.1.1r
2 Solvent CDCl3
3 Temperature 294.4
4 Number of Scans 32
5 Spectrometer Frequency 600.17
6 Nucleus 1H
O
O
O
Ph
OH
I
C46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137.5
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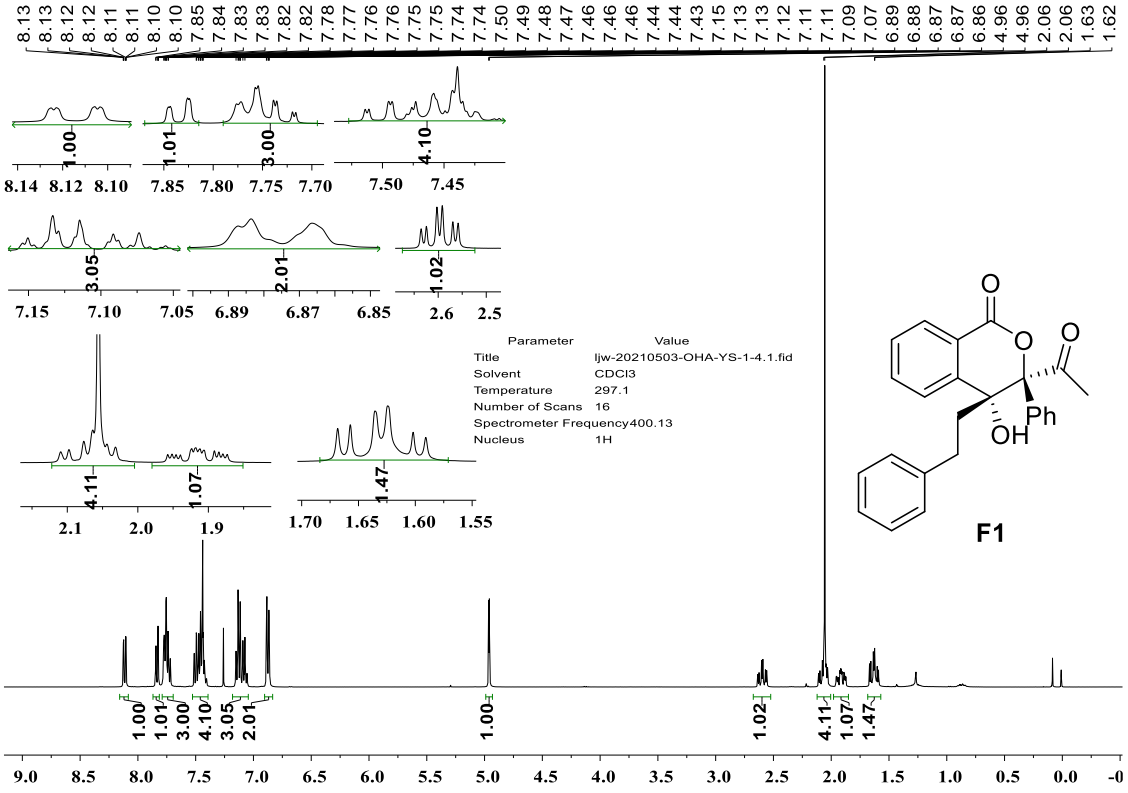
135.6
134.0
131.2
130.7
130.1
129.6
129.1
128.7
128.4
128.4
126.3
125.0
122.4
93.4
90.8
77.6
21.2
Parameter Value
1 Title langjw-27052021-OHA-606.2.1.1r
2 Solvent CDCl3
3 Temperature 295.2
4 Number of Scans 512
5 Spectrometer Frequency 150.91
6 Nucleus 13C
O
O
O
Ph
OH
I
C46
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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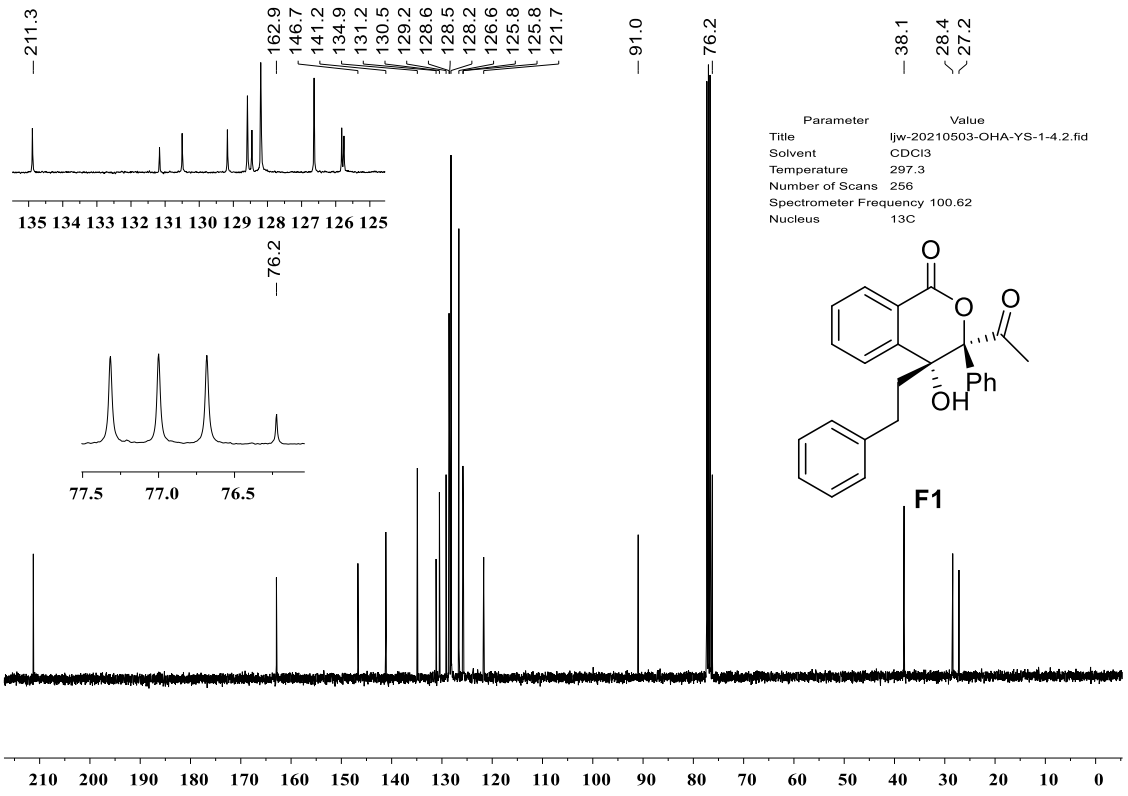
Parameter Value
Title ljw-20210503-OHA-YS-1-4.1.fid
Solvent CDCl3
Temperature 297.1
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
O
O
O
Ph
OH
F1
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0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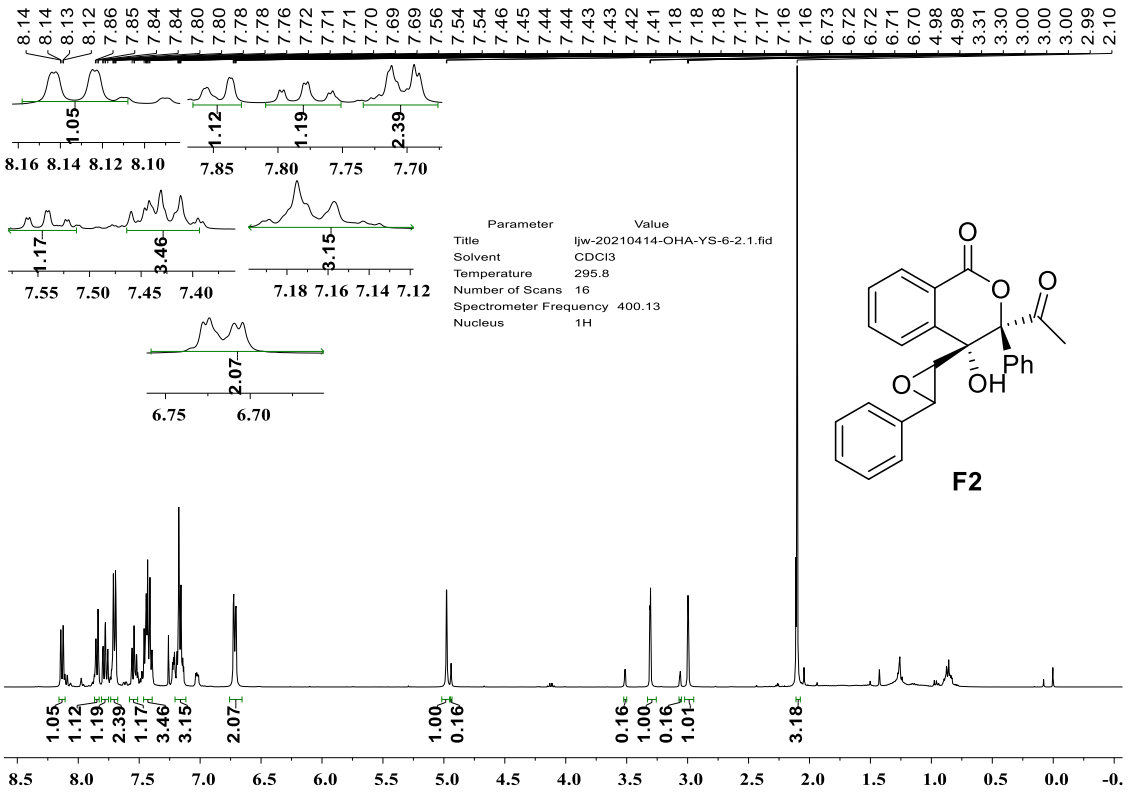
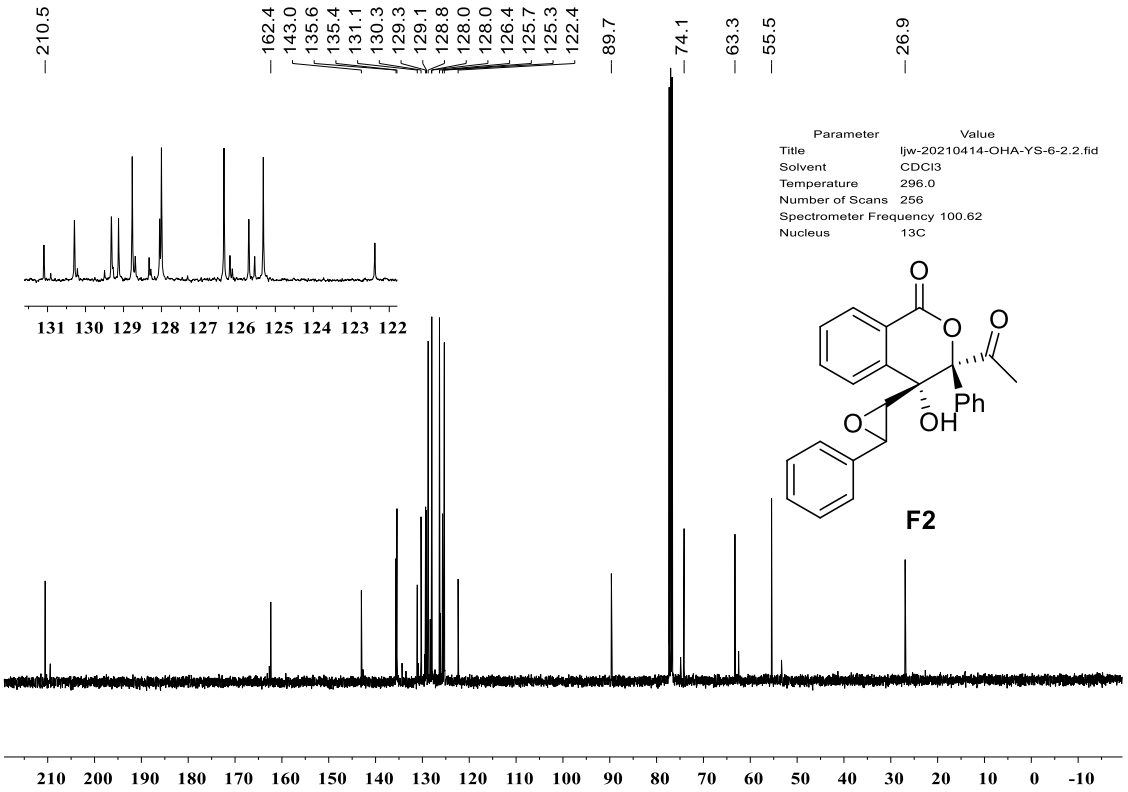
141.2
134.9
131.2
130.5
129.2
128.6
128.5
128.2
126.6
125.8
125.8
121.7
91.0
76.2
38.1
28.4
27.2
Parameter Value
Title ljw-20210503-OHA-YS-1-4.2.fid
Solvent CDCl3
Temperature 297.3
Number of Scans 256
Spectrometer Frequency 100.62
Nucleus 13C
F1


Parameter Value
Title ljw-20210414-OHA-YS-6-2.1.fid
Solvent CDCl3
Temperature 295.8
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
F2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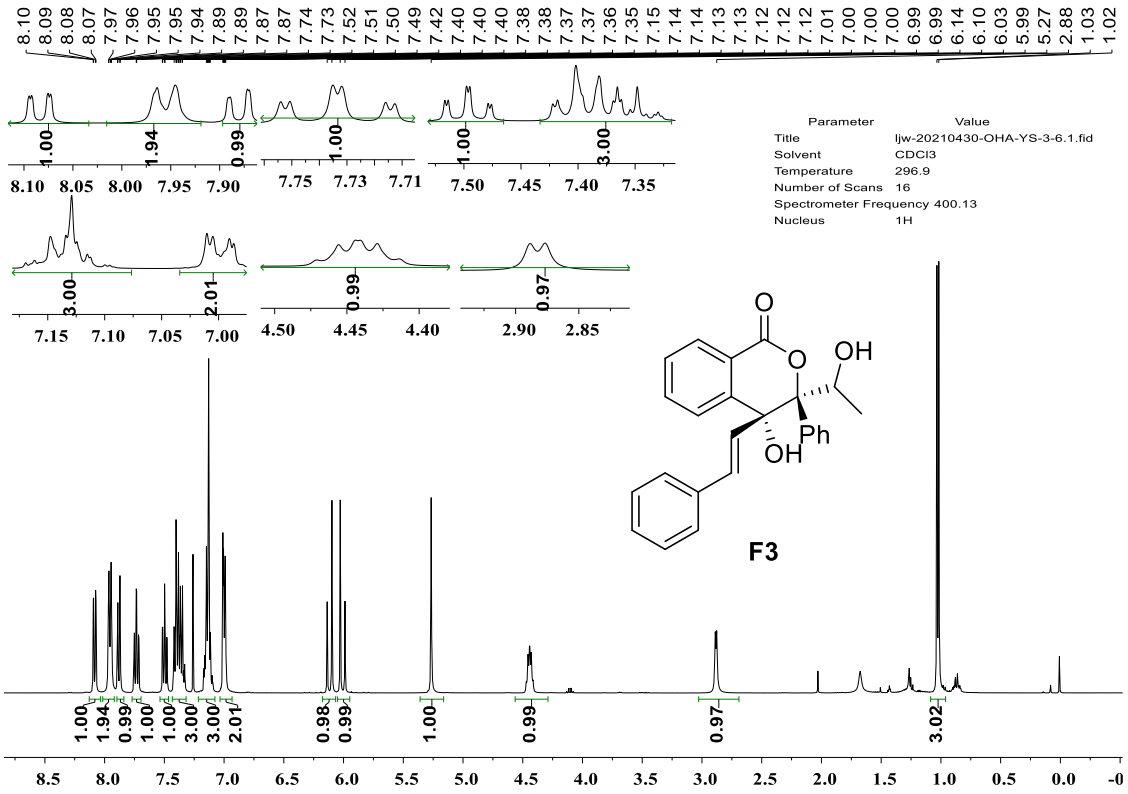
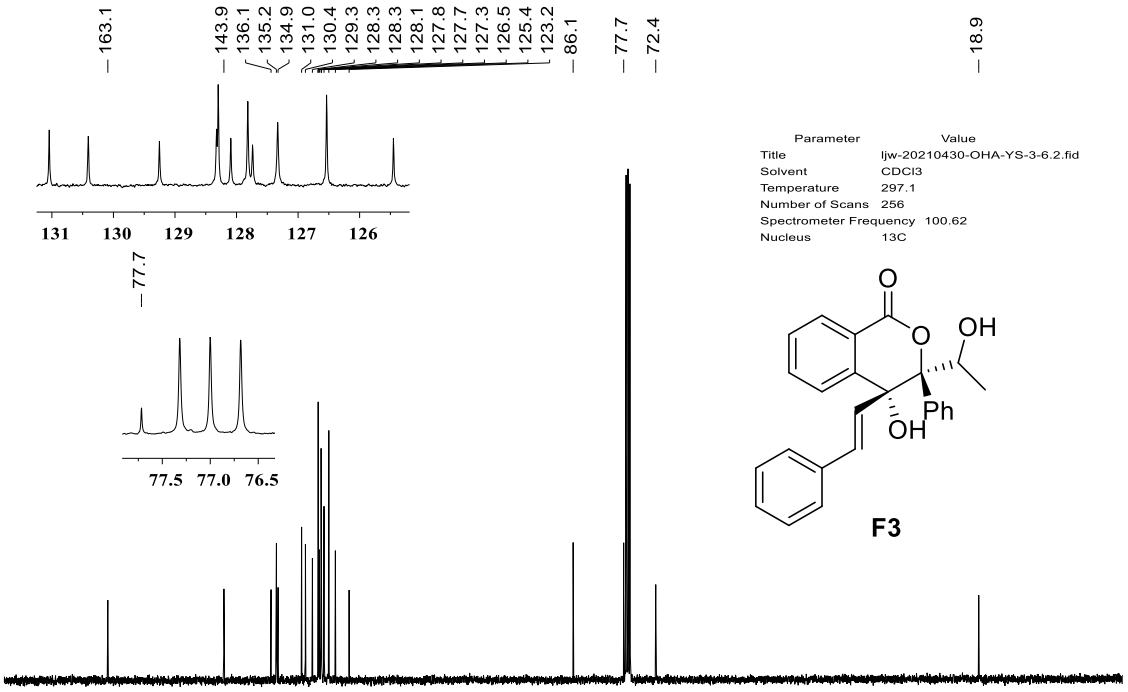
143.0
135.6
135.4
131.1
130.3
129.3
129.1
128.8
128.0
128.0
126.4
125.7
125.3
122.4
89.7
74.1
63.3
55.5
26.9
Parameter Value
Title ljw-20210414-OHA-YS-6-2.2.fid
Solvent CDCl3
Temperature 296.0
Number of Scans 256
Spectrometer Frequency 100.62
Nucleus 13C
F2


Parameter Value
Title ljw-20210430-OHA-YS-3-6.1.fid
Solvent CDCl3
Temperature 296.9
Number of Scans 16
Spectrometer Frequency 400.13
Nucleus 1H
F3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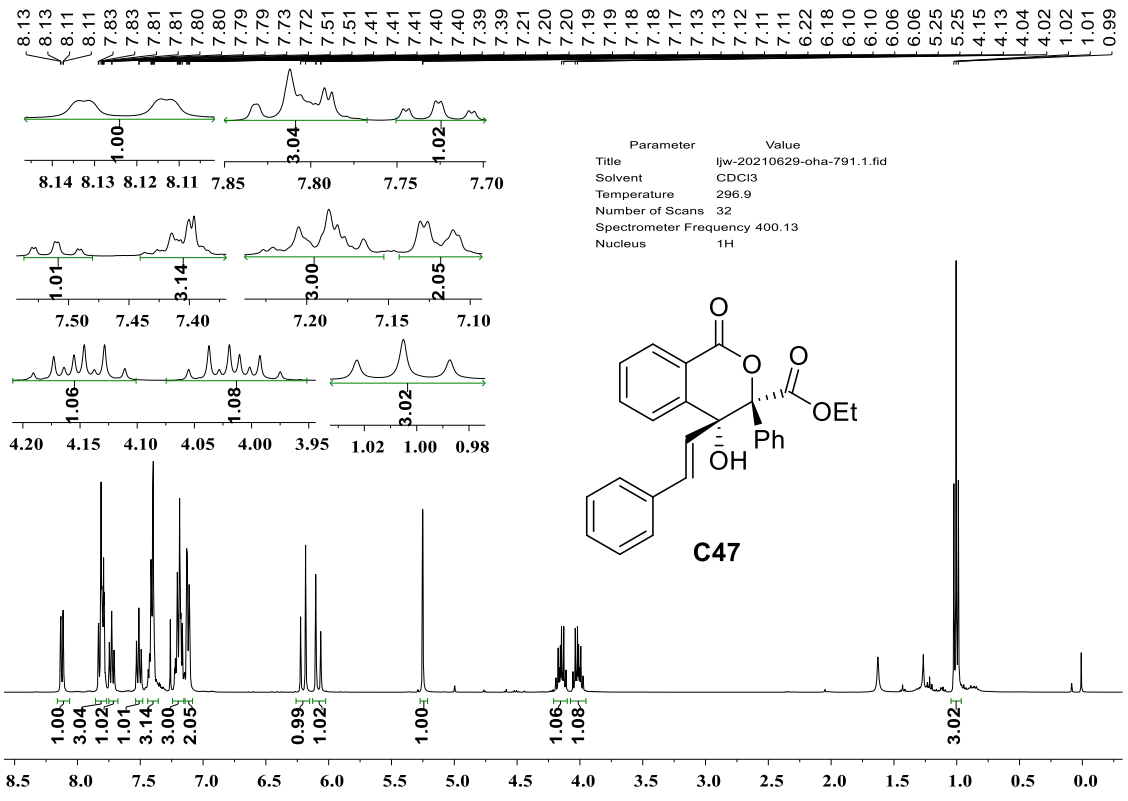
136.1
135.2
134.9
131.0
130.4
129.3
128.3
128.3
128.1
127.8
127.7
127.3
126.5
125.4
123.2
86.1
77.7
72.4
18.9
Parameter Value
Title ljw-20210430-OHA-YS-3-6.2.fid
Solvent CDCl3
Temperature 297.1
Number of Scans 256
Spectrometer Frequency 100.62
Nucleus 13C
F3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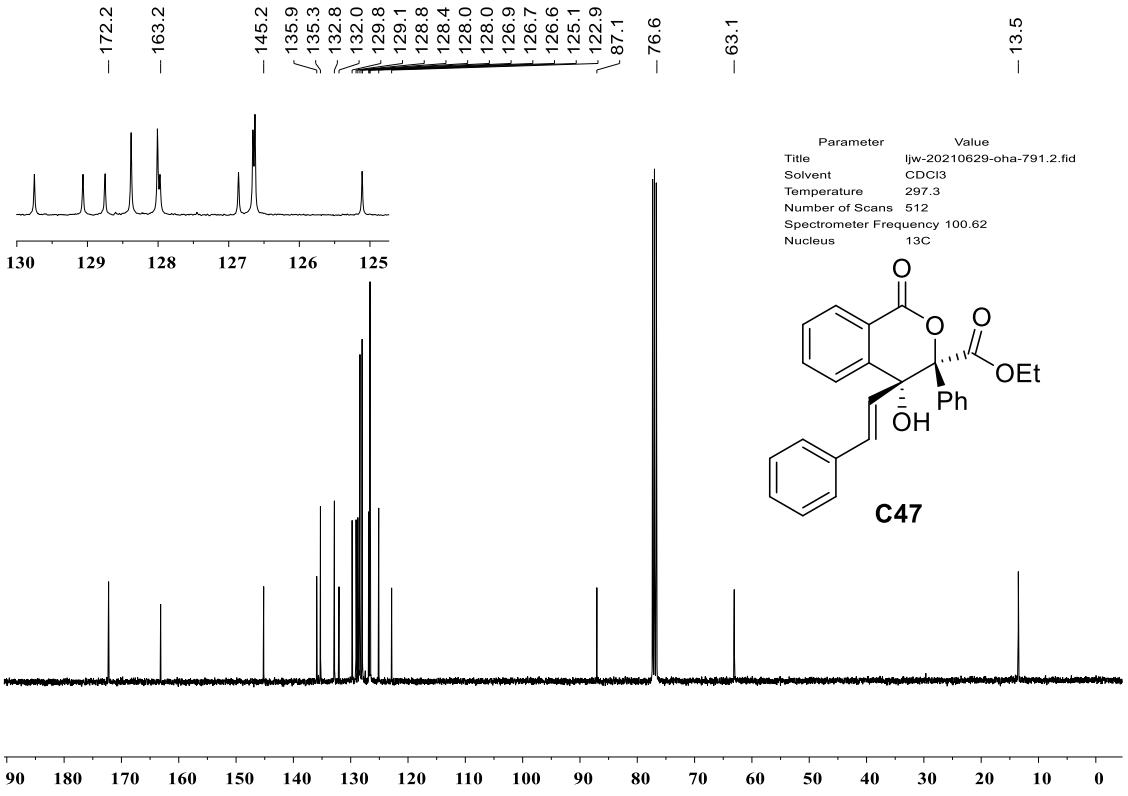
7.39
7.21
7.20
7.19
7.18
7.17
7.13
7.12
7.11
6.22
6.18
6.10
6.06
5.25
4.15
4.13
4.04
4.02
1.02
1.01
0.99
Parameter Value
Title ljw-20210629-oha-791.1.fid
Solvent CDCl3
Temperature 296.9
Number of Scans 32
Spectrometer Frequency 400.13
Nucleus 1H
C47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163.2
145.2
135.9
135.3
132.8
132.0
129.8
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128.8
128.4
128.0
128.0
126.9
126.7
126.6
125.1
122.9
87.1
76.6
63.1
13.5
Parameter Value
Title ljw-20210629-oha-791.2.fid
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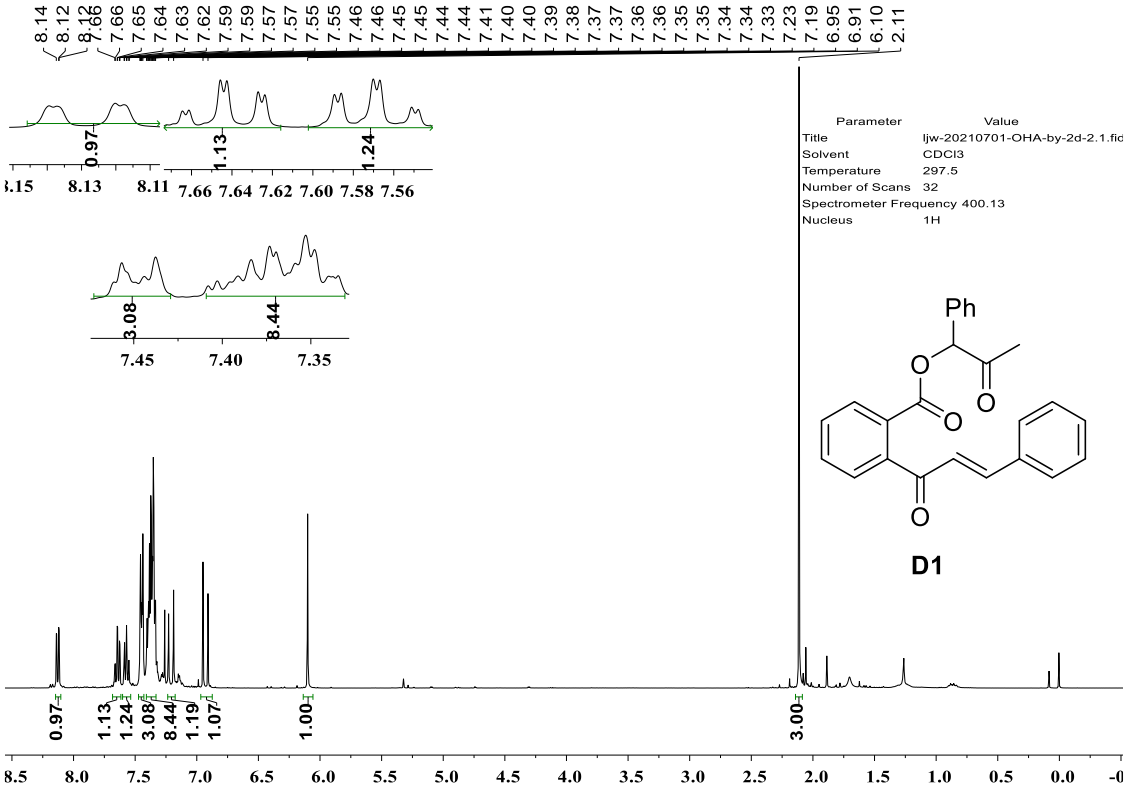
Temperature 297.3
Number of Scans 512
Spectrometer Frequency 100.62
Nucleus 13C
OEt
Ph
OH
C47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7.36
7.36
7.35
7.35
7.34
7.34
7.33
7.23
7.19
6.95
6.91
6.10
2.11
Parameter Value
Title ljw-20210701-OHA-by-2d-2.1.fid
Solvent CDCl3
Temperature 297.5
Number of Scans 32
Spectrometer Frequency 400.13
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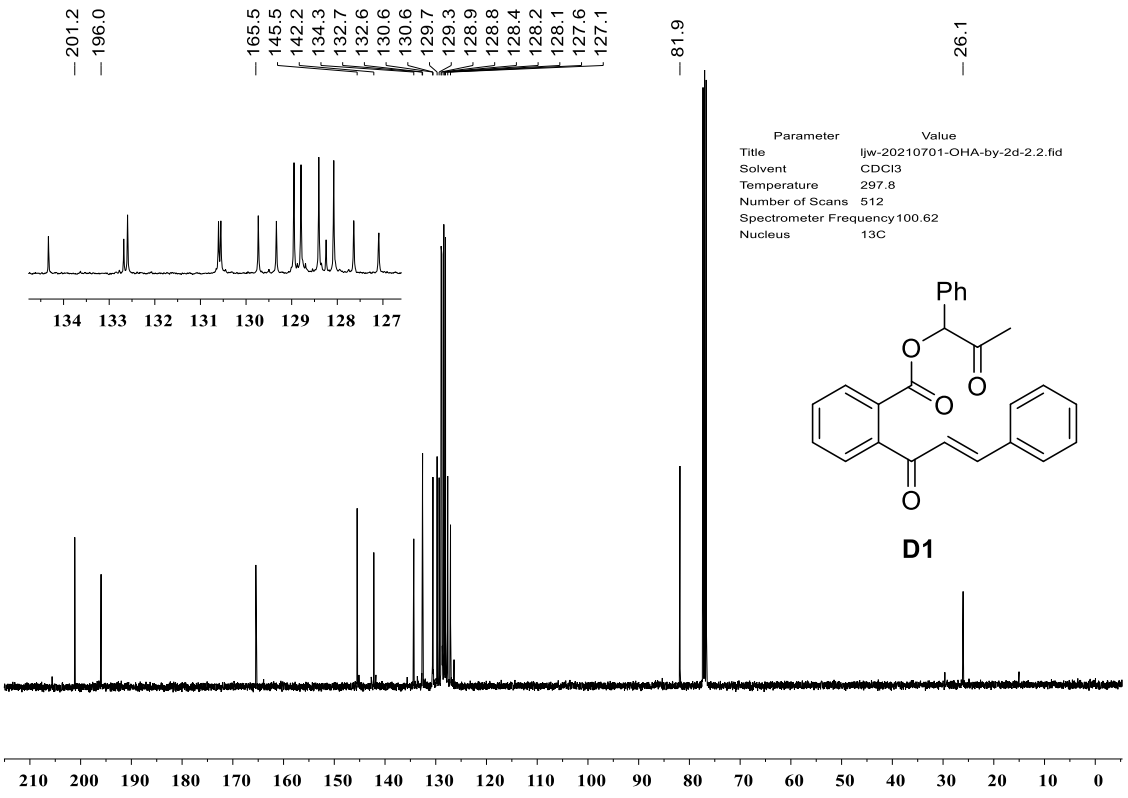
1.00
3.00



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3 a) X. H. Liu, L. L. Lin and X. M. Feng, *Acc. Chem. Res.*, 2011, **44**, 574; (b) X. H. Liu, L. L. Lin and X. M. Feng, *Org. Chem. Front.*, 2014, **1**, 298.