Chiral Chemistry
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Supplementary information

CuH-catalyzed asymmetric reduction of vinyl-substituted 1,2-diketones

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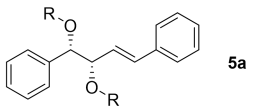
I. Materials and methods

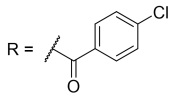
Commercially available materials were used as received, unless otherwise noted, all reactions and manipulations involving air- or moisture-sensitive compounds were performed using standard Schlenk technique. All solvents were purified and dried using typical procedures. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a Bruker AVANCE III HD400 (400 MHz) spectrometer, a JEOL ECZ600S (600 MHz) and a JEOL ECZ400S (400 MHz). Chemical shifts were recorded in parts per million (ppm, δ) relative to tetramethylsilane (δ = 0.00 ppm) , chloroform (δ = 7.26 ppm). ^{1}H NMR splitting patterns are designated as singlet (s), doublet (d), triplet (t), quartet (q), dd (doublet of doublets), m (multiplet), etc. All first-order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted are designated as multiplet (m) or broad (br). Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on a Bruker AVANCE III HD400 (101 MHz) spectrometer, JEOL ECZ600S (151 MHz) and a JEOL ECZ400S (101 MHz). High resolution mass spectral analysis (HRMS) was performed on Thermo Fisher Scientific Q Exactive Plus Hybrid Quadrupole-Orbitrap Mass Spectrometer. X-ray crystallography analysis was performed on Agilent Super Nova X-ray diffractionmeter. Analytical thin-layer chromatography (TLC) was carried out on WFH-203 F254 pre-coated silica gel plate (0.2 mm thickness). Visualization was performed using a UV lamp or 2,4-Dinitrophenylhydrazine or potassium

permanganate stain or phosphomolybdic acid.

II. X-ray crystallographic analysis

Method for single crystals cultivation: a pure solid sample (10–20 mg) was dissolved in isopropanol (1 mL) in a vial at room temperature, and petroleum hexane (1 mL) was added into the above solution slowly while keeping the sample completely dissolved. The vial was properly sealed with parafilm and kept at room temperature to allow the slow evaporation of the solvents until a single crystal was obtained.





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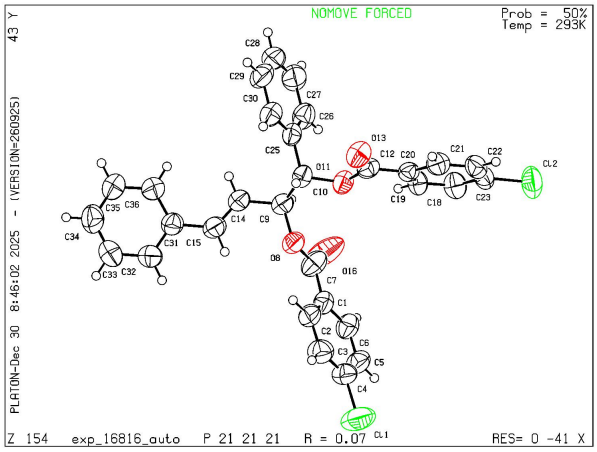

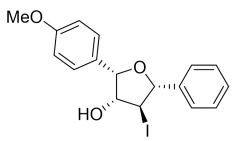

3b'

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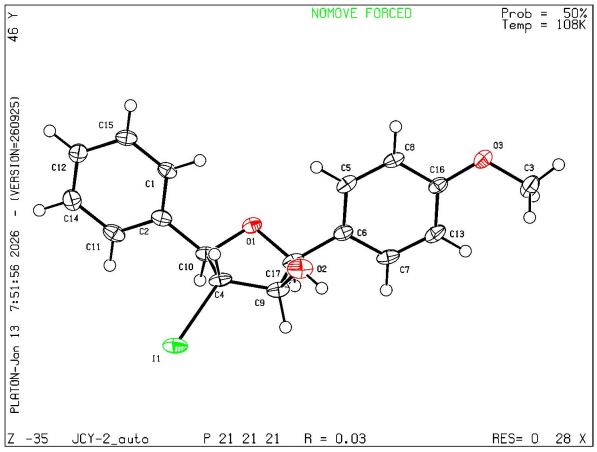

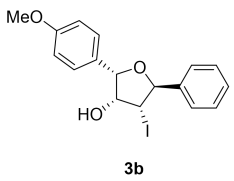

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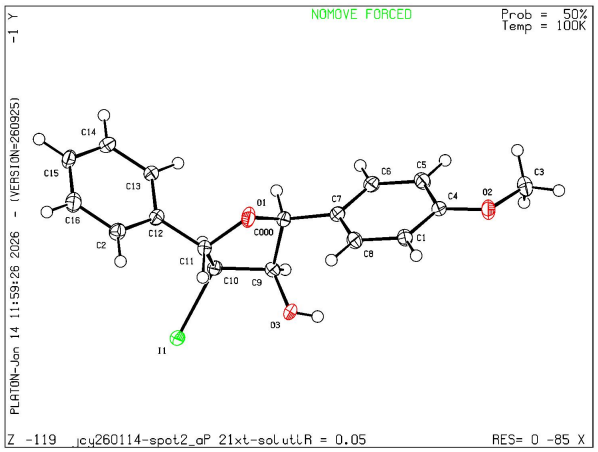


Table S1 Crystal data and structure refinement for 3a

Identification code	**5a**
Empirical formula	$C_{30}H_{22}O_4SCl_2$
Formula weight	517.37
Temperature/K	293 (2)
Crystal system	orthorhombic
Space group	P212121
a/Å	6.3459 (2)
b/Å	10.7079 (3)
c/Å	38.2260 (14)
α/°	90
β/°	90
γ/°	90
Volume/Å3	2597.52 (15)
Z	4
ρcalcg/cm3	1.323
μ/mm-1	2.527
F(000)	1072.0
Crystal size/mm3	0.1 × 0.1 × 0.1
Radiation	CuKα (λ = 1.54184)

2Θ range for data collection/°	4.624 to 152.854
Index ranges	-8 ⩽ h ⩽ 5, -13 ⩽ k ⩽ 13, -38 ⩽ l ⩽ 48
Reflections collected	9593
Independent reflections	4763 [Rint = 0.0535, Rsigma = 0.0585]
Data/restraints/parameters	4763/0/326
Goodness-of-fit on F2	1.155
Final R indexes [I>=2σ (I)]	R1 = 0.0731, wR2 = 0.2269
Final R indexes [all data]	R1 = 0.0996, wR2 = 0.2335
Largest diff. peak/hole / e Å-3	0.30/−0.25
Flack parameter	0.012 (17)

Table S2 Crystal data and structure refinement for 3b'

Identification code	**3b'**
Empirical formula	$C_{2.34}H_{2.34}O_{0.41}I_{0.14}$
Formula weight	54.65
Temperature/K	108 (2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	6.01880 (10)
b/Å	9.43060 (10)
c/Å	27.1072 (3)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1538.63 (3)
Z	29
ρcalcg/cm^3	1.710
μ/mm^{-1}	11.261

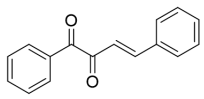
F (000)	784.0
Crystal size/mm^3	0.1 × 0.1 × 0.1
Radiation	GaKα (λ = 1.3405)
2Θ range for data collection/°	5.67 to 109.728
Index ranges	-7 ⩽ h ⩽ 7, -11 ⩽ k ⩽ 11, -32 ⩽ l ⩽ 32
Reflections collected	54517
Independent reflections	2916 [Rint = 0.1681, Rsigma = 0.0392]
Data/restraints/parameters	2916/0/195
Goodness-of-fit on F^2	1.098
Final R indexes [I>=2σ (I)]	R1 = 0.0299, wR2 = 0.0760
Final R indexes [all data]	R1 = 0.0304, wR2 = 0.0763
Largest diff. peak/hole / e Å^{-3}	0.65/−1.03
Flack parameter	−0.016 (6)

Table S3 Crystal data and structure refinement for 3b

Identification code	**3b**
Empirical formula	$C_{17}H_{17}O_3I$
Formula weight	396.21
Temperature/K	100 (2)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	8.90430 (10)
b/Å	5.38250 (10)
c/Å	16.6120 (2)
α/°	90
β/°	101.3530 (10)
γ/°	90
Volume/Å3	780.59 (2)

Z	2
ρcalcg/cm^3	1.686
μ/mm^{-1}	11.099
F (000)	392.0
Crystal size/mm^3	0.1 × 0.1 × 0.1
Radiation	GaKα (λ = 1.3405)
2Θ range for data collection/°	4.718 to 110.486
Index ranges	-10 ⩽ h ⩽ 10, -6 ⩽ k ⩽ 6, -19 ⩽ l ⩽ 20
Reflections collected	27112
Independent reflections	2995 [Rint = 0.1040, Rsigma = 0.0387]
Data/restraints/parameters	2995/1/193
Goodness-of-fit on F^2	1.208
Final R indexes [I>=2σ (I)]	R1 = 0.0488, wR2 = 0.1310
Final R indexes [all data]	R1 = 0.0493, wR2 = 0.1315
Largest diff. peak/hole / e Å^{-3}	0.51/−1.02
Flack parameter	−0.033 (19)

III. Typical procedures for the preparation of substrates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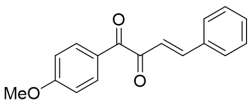
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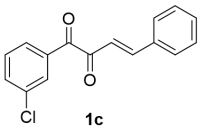
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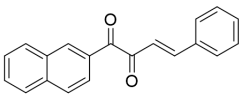
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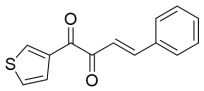
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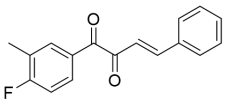
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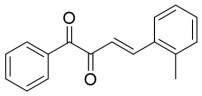
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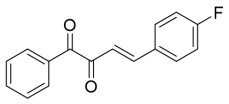
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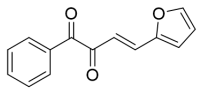
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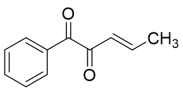
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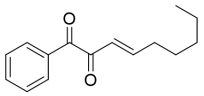
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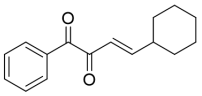
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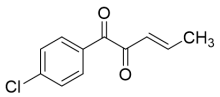
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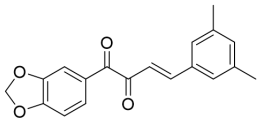
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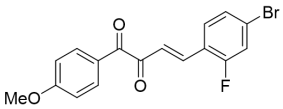
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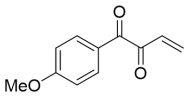
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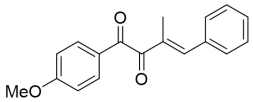
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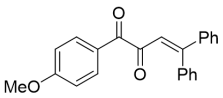
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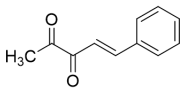
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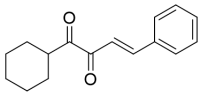
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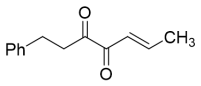
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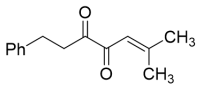
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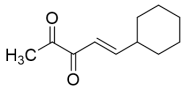
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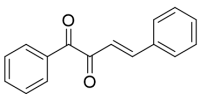
1. Substrates **1a–1d**, **1g–1m**, **1p**, **1s** are known compounds and were prepared according to the literature report.[1]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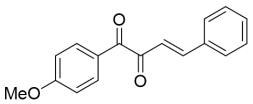
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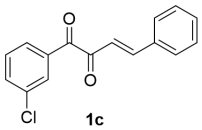
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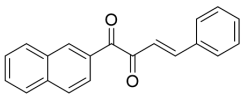
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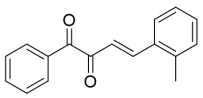
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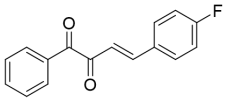
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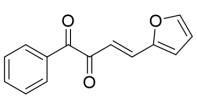
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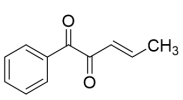
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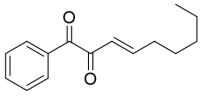
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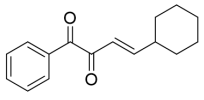
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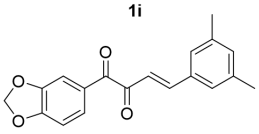
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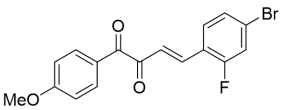
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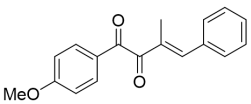
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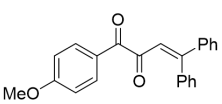
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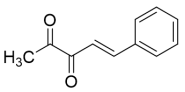
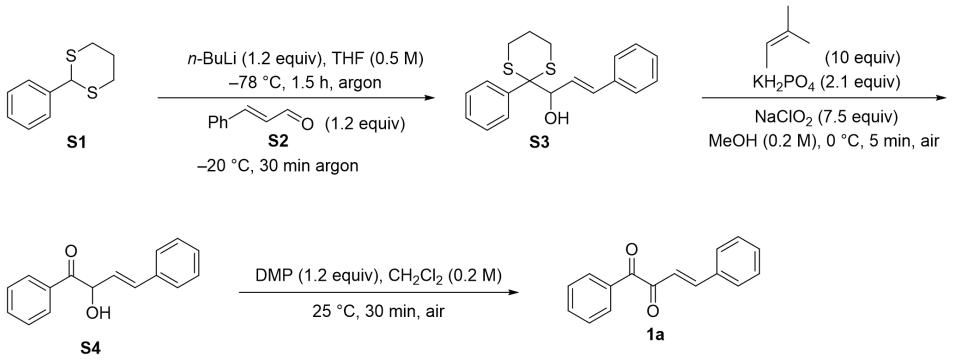

1s

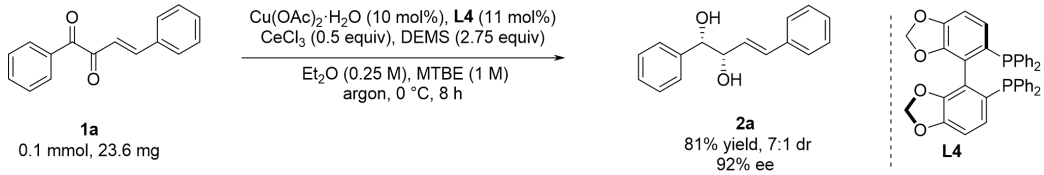
2. General procedures for the preparation of substrates **1e**, **1f**, **1n**, **1o**, **1q**, **1r**, **1t–1w**.



Typically, a dried 50 mL of two-necked flask containing THF (20 mL) at −78 ˚C under argon atmosphere was added 2-phenyl-1,3-dithiane **S1** (1.96 g, 10.0 mmol). To this mixture, *n*-BuLi (4.8 mL, 12.0 mmol, 2.5 M in hexanes) was added at −78 ºC. The mixture was stirred at the same temperature for 1.5 h. Then, cinnamaldehyde **S2** (yellow oil, 1.59 g, 12.0 mmol) was added dropwise via a syringe. The reaction was stirred for additional 30 min at −20 ºC. The reaction was quenched with saturated aqueous NH_4Cl (15 mL) at 0 ºC and warmed to room temperature. The reaction mixture was extracted with ethyl acetate (3 × 100 mL) and saturated brine (3 × 100 mL). The combined organic phase was dried with anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by chromatography (petroleum ether/ethyl acetate, v:v = 10:1) to afford product **S3** (yellow oil, 2.95 g, 90% yield). To the solution of **S3** (2.95 g, 9.0 mmol) in MeOH (45 mL) were added the mixture of 2-methyl-2-butene (6.3 g, 90 mmol), $NaClO_2$ (6.1 g, 67.5 mmol) and KH_2PO_4 (2.57 g, 18.9 mmol) at room temperature. The mixture was stirred at room temperature for 5 min. After completion, saturated aqueous $Na_2S_2O_3$ solution (10 mL) was added. Then the reaction mixture was extracted with CH_2Cl_2 (3 × 30 mL) and saturated brine (3 × 20 mL), and then dried over anhydrous Na_2SO_4. The residue was purified by chromatography (petroleum ether/ethyl acetate, v:v = 5:1) to afford product **S4** (1.76 g, 82% yield). To a dry 250 mL round bottom flask was added a mixture of **S4** (yellow oil, 1.76 g, 7.38 mmol), DMP (3.76 g, 8.86 mmol) in CH_2Cl_2 (74 mL) at 0 °C. The mixture was stirred for 0.5 h. After completion, saturated aqueous $Na_2S_2O_3$ solution (10 mL) was added. Then

the reaction mixture was extracted with CH_2Cl_2 (3 × 15 mL) and saturated brine (3 × 20 mL). The combined organic phase was dried with anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by chromatography (petroleum ether/ethyl acetate, v:v = 20:1) to afford product **1a** (yellow solid, 1.39 g, 80% yield).

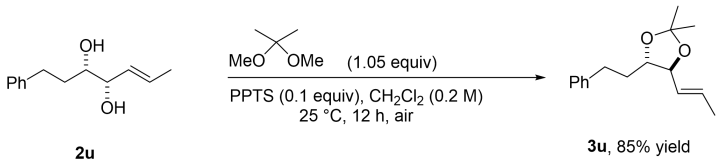
IV. Procedures for Copper-Catalyzed Asymmetric Hydrosilylation



An oven-dried screw-cap reaction tube equipped with a magnetic stir bar was charged with $Cu(OAc)_2•H_2O$ (2.0 mg, 0.01 mmol) and ligand **L4** (6.7 mg, 0.011 mmol) in a glove box. Anhydrous Et_2O (0.2 mL) and anhydrous MTBE (0.05 mL) were added via syringe. The mixture was stirred at room temperature for 15 min, and $(EtO)_2MeSiH$ (44 μL, 0.275 mmol) was added via syringe. The mixture was stirred at room temperature for 10 min until the color changed from blue to green. Meanwhile a second oven-dried screw-cap reaction tube equipped with a magnetic stir bar was charged with substrate **1a** (23.6 mg, 0.1 mmol), $CeCl_3$ (12.3 mg, 0.05 mmol), anhydrous Et_2O (0.2 mL), and anhydrous MTBE (0.05 mL). The mixture was stirred at room temperature for 30 min. After this period, the catalyst solution from the first reaction tube was added slowly to the stirred mixture via syringe, and the resulting mixture was stirred at 0 °C for 8 h. Then, a saturated solution of NH_4F in MeOH (1 mL) was added and the mixture was stirred at room temperature for 10 min until the reaction was complete. The reaction mixture was then extracted with CH_2Cl_2 (3 × 15 mL). The combined organic extracts were washed with saturated brine (3 × 20 mL), dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash chromatography (petroleum ether/ethyl acetate, v:v = 3:1) to afford the product **2a** (colorless oil, 19.7 mg, 82% yield, 7:1 dr, 92% ee).

V. Determination of the Absolute Configurations of 2u

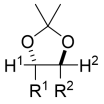
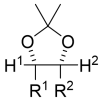
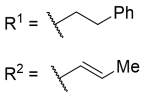
Typical Procedure for the Synthesis of 3u and 3u'.

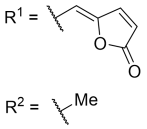
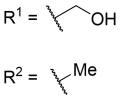
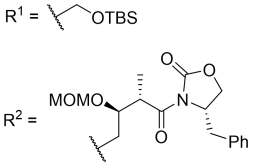
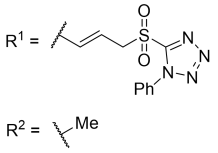


To a 10 mL reaction tube was charged with **2u** (20.6 mg，0.1 mmol) and PPTS (2.5 mg, 0.01 mmol) at room temperature. CH_2Cl_2 (0.5 mL) was added via a syringe. Then, 2,2-dimethoxypropane (10.9 mg, 0.105 mmol) was added to this tube. The reaction was stirred for 12 h at room temperature. Upon completion, the reaction was quenched with saturated aqueous H_2O (1 mL) at room temperature. The reaction mixture was extracted with ethyl acetate (3 × 10 mL) and saturated brine (3 × 10 mL). The combined organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by chromatography (petroleum ether/ethyl acetate, v:v = 10:1) to afford product **3u** (colorless oil, 20.9 mg, 85% yield).

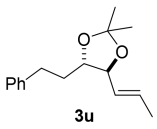
Determine the relative configuration of 2u.

According to the comparison of literature data, the coupling constant between H^1 and H^2 in the trans five-membered ring is approximately 8.5 Hz, while for the cis five-membered ring product, the coupling constant between H^1 and H^2 is around 6.5 Hz.[2-4] Thus, it can be deduced that the relative configuration of the hydroxyl group in product **2u** is *cis*.

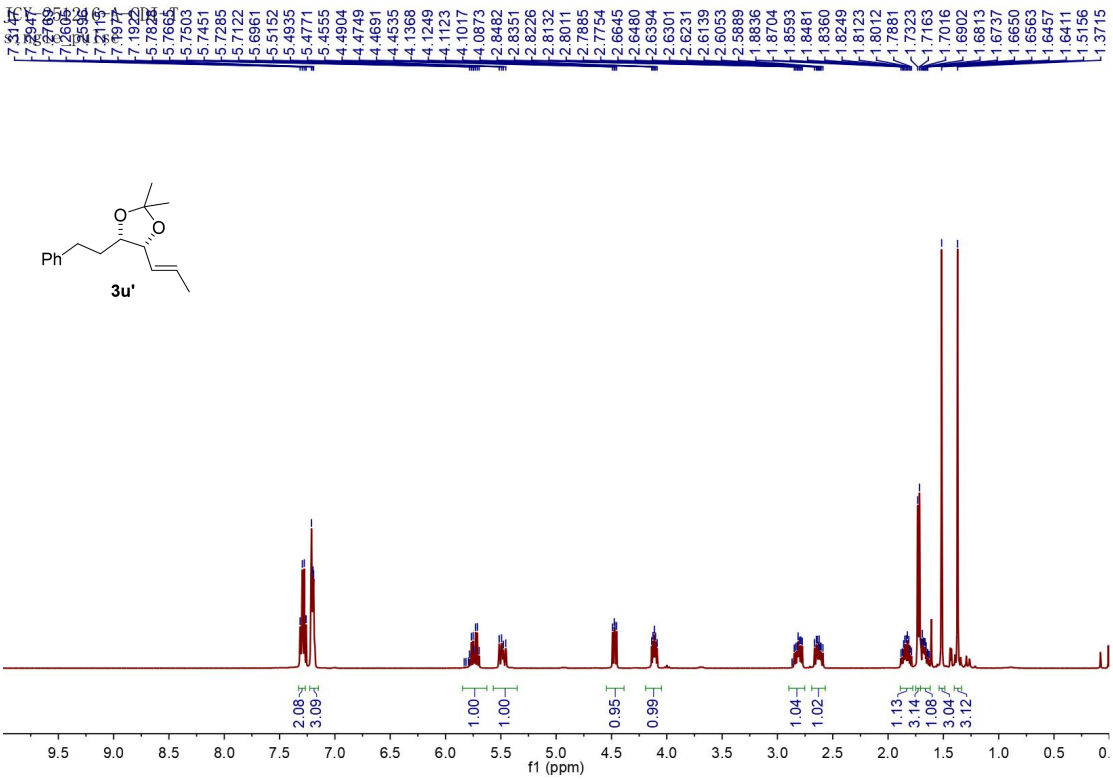
Literature comparison			
entry 1		J_{12} = 8.3 Hz	J_{12} = 6.2 Hz

entry 2[2]		J_{12} = 8.5 Hz	J_{12} = 6.3 Hz
entry 3[2]		J_{12} = 8.3 Hz	J_{12} = 4.8 Hz
entry 4[3]		J_{12} = 8.5 Hz	J_{12} = 5.9 Hz
entry 5[4]		J_{12} = 8.3 Hz	J_{12} = 6.7 Hz

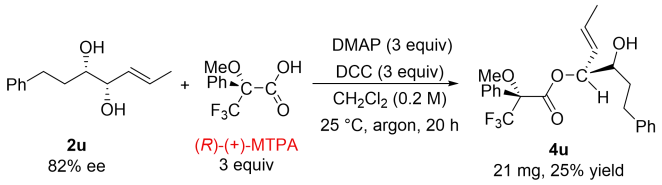
JCY-251203-Z-PPPPPP



3u

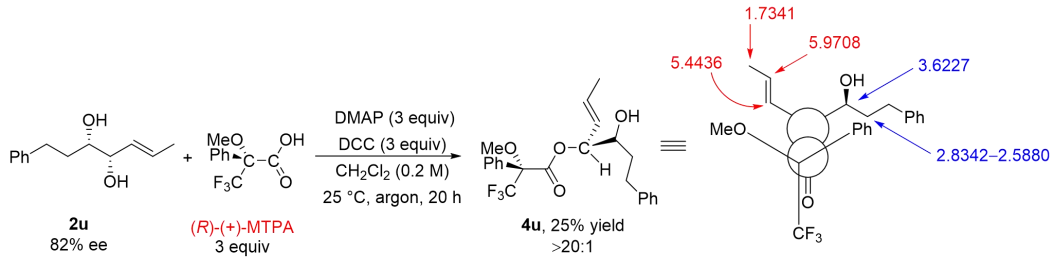


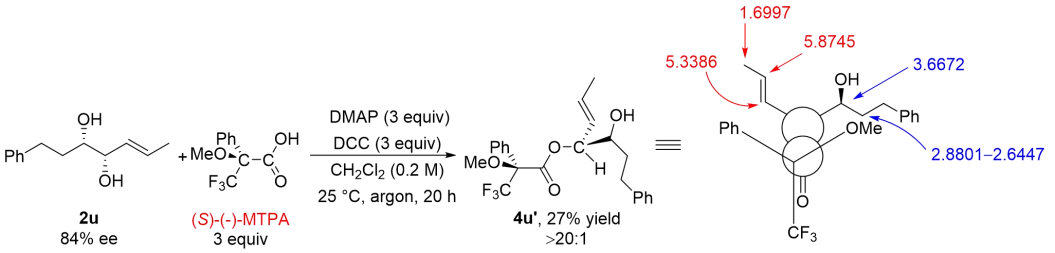
Typical procedure for the synthesis of the corresponding Mosher esters:

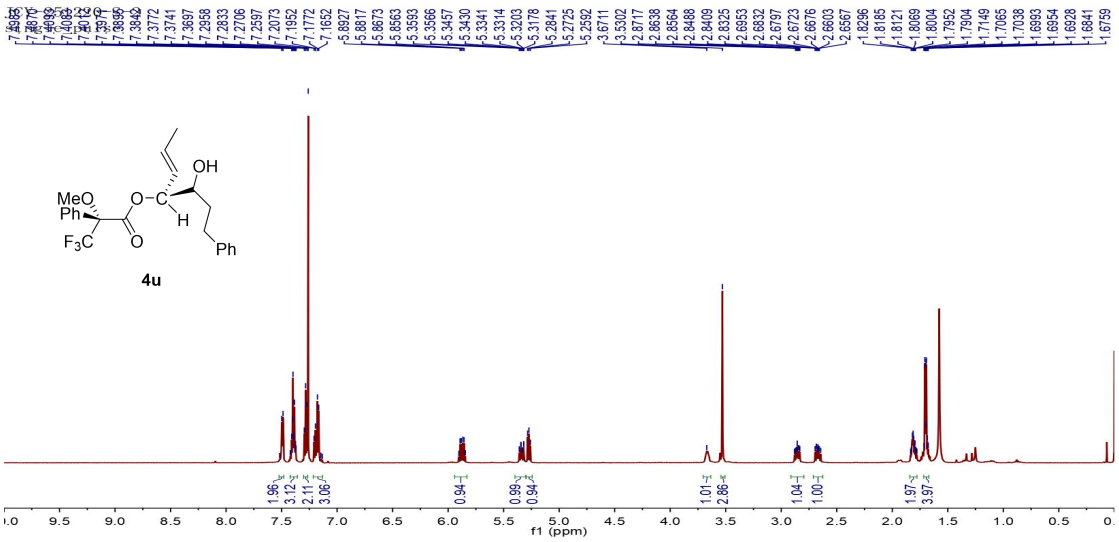


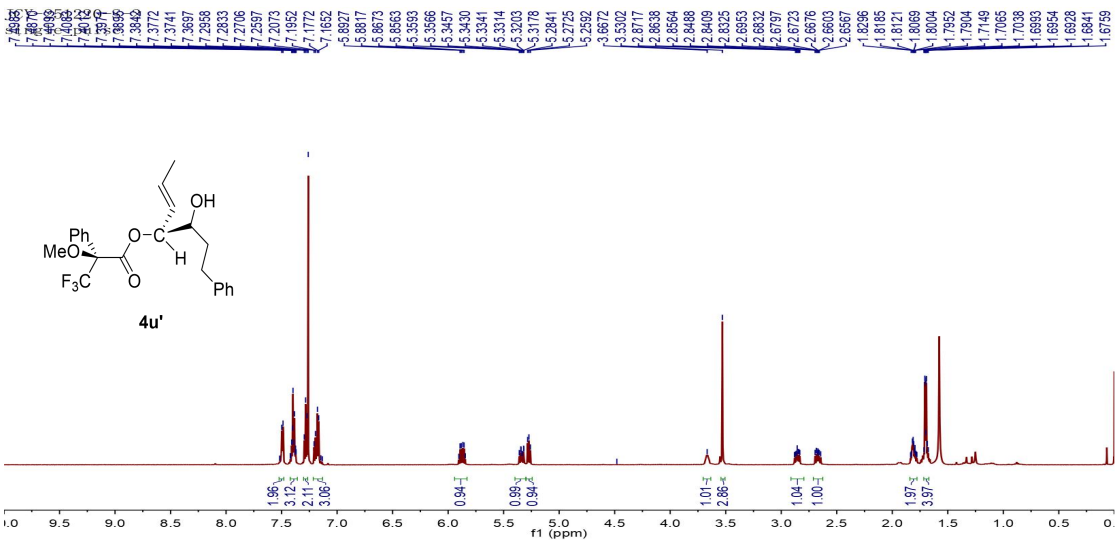
A solution of **2u** (20.6 mg, 0.1 mmol, 82% ee) and (*R*)-(+)-MTPA (70.3 mg, 0.3 mmol, 3 equiv) in dry CH_2Cl_2 (0.5 mL) was added DCC (61.9 mg, 0.3 mmol, 3 equiv) and DMAP (36.7 mg, 0.3 mmol, 3 equiv). The resulting solution was stirred at room temperature for 20 h. After the reaction is complete, the white precipitate was removed through a cotton plug. The reaction solution was concentrated on a rotary evaporator and purified by column chromatography on silica gel (petroleum ether/ethyl acetate, v:v = 10:1) to give the colorless oil of **4u** (colorless oil, 21 mg, 25% yield, >20:1 dr). **4u'**、**5u** and **5u'** was synthesized through the same method.

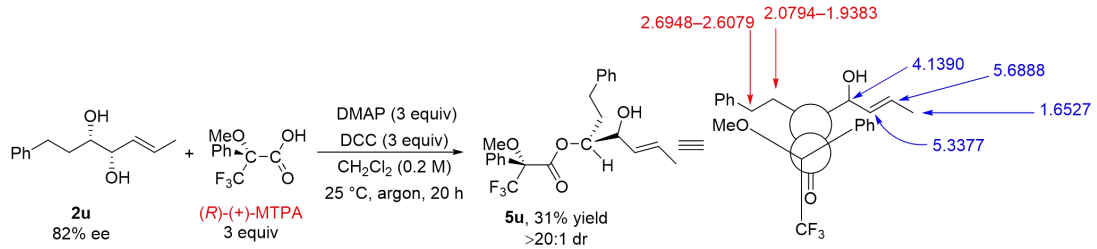
Determination of the absolute configuration of 2u

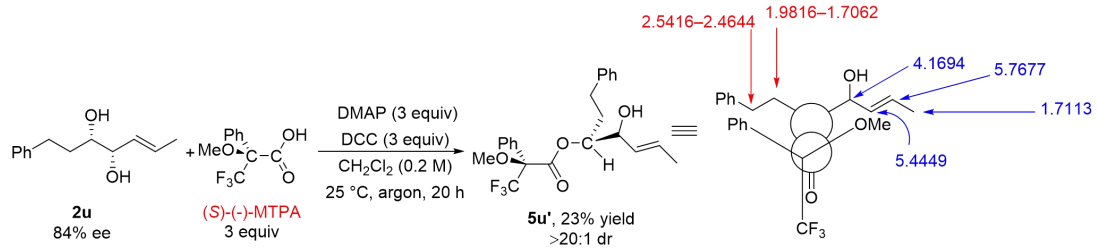

DMAP (3 equiv)
DCC (3 equiv)
CH2Cl2 (0.2 M)
25 °C, argon, 20 h
2u
82% ee
(R)-(+)-MTPA
3 equiv
4u, 25% yield
>20:1
1.7341
5.9708
5.4436
3.6227
2.8342–2.5880

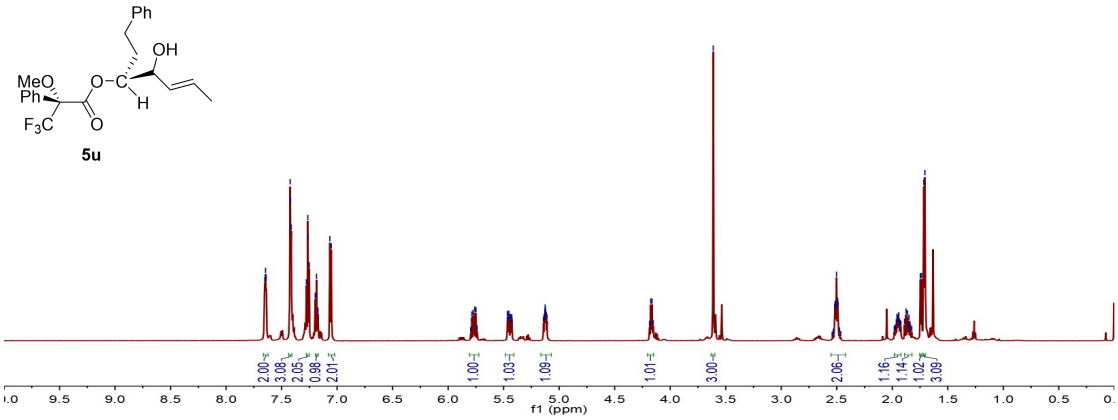

DMAP (3 equiv)
DCC (3 equiv)
CH2Cl2 (0.2 M)
25 °C, argon, 20 h
2u
84% ee
(S)-(-)-MTPA
3 equiv
4u', 27% yield
>20:1
1.6997
5.8745
5.3386
3.6672
2.8801–2.6447

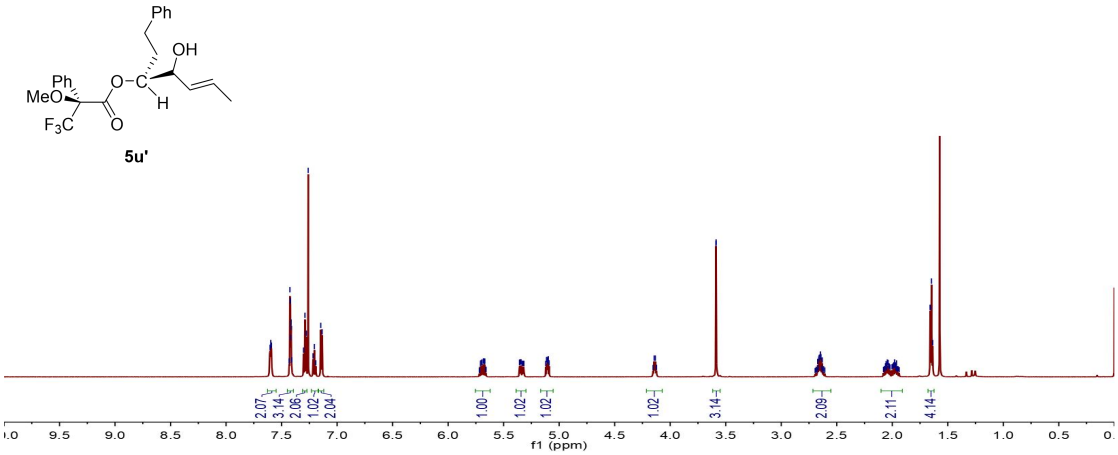

4u
f1 (ppm)


4u'
f1 (ppm)

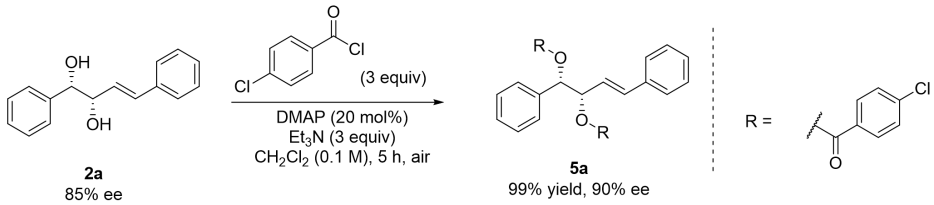

2.6948–2.6079
2.0794–1.9383
4.1390
5.6888
1.6527
5.3377
DMAP (3 equiv)
DCC (3 equiv)
CH2Cl2 (0.2 M)
25 °C, argon, 20 h
2u
82% ee
(R)-(+)-MTPA
3 equiv
5u, 31% yield
>20:1 dr


2.5416–2.4644
1.9816–1.7062
4.1694
5.7677
1.7113
5.4449
DMAP (3 equiv)
DCC (3 equiv)
CH2Cl2 (0.2 M)
25 °C, argon, 20 h
2u
84% ee
(S)-(-)-MTPA
3 equiv
5u', 23% yield
>20:1 dr

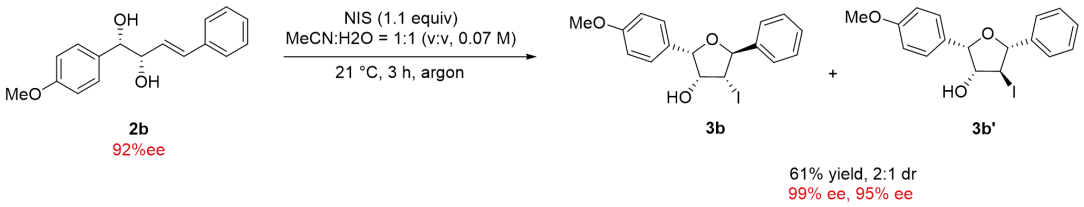

5u
f1 (ppm)


5u'
f1 (ppm)

VI. Procedures for the derivatizations of 2b

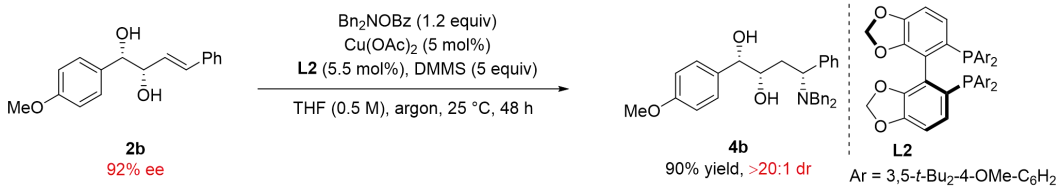


A 10 mL reaction tube was charged with **2a** (24 mg, 0.1 mmol, 85% ee) and DMAP (2.4 mg, 0.02 mmol) at room temperature. CH_2Cl_2 (1 mL) was added via a syringe. Then, Et_3N (42 μL, 0.3 mmol) and 4-chlorobenzoyl chloride (38.5 μL, 0.3 mmol) were added to this tube. The reaction was stirred for 5 h at room temperature. Upon completion, the reaction was quenched with saturated aqueous H_2O (1 mL) at room temperature. The reaction mixture was extracted with ethyl acetate (3 × 10 mL) and saturated brine (3 × 10 mL). The combined organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by chromatography (petroleum ether/ethyl acetate, v:v = 2:1) to afford product **3a** (white solid, 51.2 mg, 99% yield, 90% ee).



A 10 mL reaction tube was charged with **2b** (27 mg, 0.1 mmol, 92% ee) and NIS (24.7 mg, 0.11 mmol) at room temperature under argon atmosphere. MeCN (0.7 mL) and H_2O (0.7 mL) were added via a syringe. The reaction was stirred for 3 h at room temperature. Upon completion, the reaction was quenched with saturated aqueous $Na_2S_2O_3$ (1 mL) at room temperature. The reaction mixture was extracted with ethyl acetate (3 × 10 mL) and saturated brine (3 × 10 mL). The combined organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by chromatography (petroleum ether/ethyl acetate, v:v = 2:1) to afford product **3b and 3b'** (white solid, 24.2 mg,

61% yield, 2:1 dr).



An oven-dried screw-cap reaction tube equipped with a magnetic stir bar was charged with $Cu(OAc)_2$ (1.8 mg, 0.005 mmol) and ligand **L2** (6.5 mg, 0.0055 mmol) in a glove box. Anhydrous THF (0.2 mL) was added via syringe. The mixture was stirred at room temperature for 15 min, and DMMS (61.5 μL, 1 mmol) was added via syringe. The mixture was stirred at room temperature for 10 min until the color changed from blue to yellow. Then, **2b** (27 mg, 0.1 mmol, 92% ee) was added to the reation. The mixture was stirred at room temperature for 48 h. Then, a saturated solution of NH_4F in MeOH (1 mL) was added and the mixture was stirred at room temperature for 10 min until the reaction was complete. The reaction mixture was then extracted with CH_2Cl_2 (3 × 15 mL).The combined organic extracts were washed with saturated brine (3 × 20 mL), dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash chromatography (petroleum ether/ethyl acetate, v:v = 2:1) to afford the product **4b** (colorless oil, 42 mg, 90% yield, >20:1 dr). The absolute configuration of **4b** was determined based on the literature.[5-9]

References:

[1] Fang, X. and co-workers. *J. Am. Chem. Soc.* **2024**, *146*, 33543−33560.

[2] Zhu, W. and co-workers. *Tetrahedron Lett.* **2013**, *54*, 6729−6731.

[3] Ichikawa, S. and co-workers. *Org. Lett.* **2020**, *22*, 2697−2701.

[4] Jayathirtha rao , V. and co-workers. *RSC Adv.* **2014**, *4*, 8365−8375.

[5] Buchwald, S.-L. and co-workers. *J. Am. Chem. Soc.* **2013**, *135*, 15746−15749.

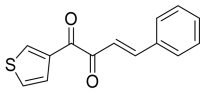
[6] Buchwald, S.-L. and co-workers. *nature* **2016**, *532*, 353−356.

[7] Wang, J. and co-workers. *CCS Chem.* **2022**, *4*, 1901–1911.

[8] Buchwald, S.-L. and co-workers. *Angew. Chem. Int. Ed.* **2018**, *57*, 8714–8718.

[9] Buchwald, S.-L. and co-workers. *J. Am. Chem. Soc.* **2015**, *137*, 9716–9721.

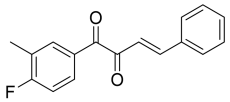
VII. Characterizations of new compounds



1e

(*E*)-4-Phenyl-1-(thiophen-3-yl)but-3-ene-1,2-dione (1e):

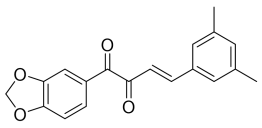
Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v/v = 20:1), amorphous yellow solid mp 54–56 °C, 1.33 g, 74% yield. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.52–8.50 (m, 1H), 7.82 (d, *J* = 16.2 Hz, 1H), 7.72 (d, *J* = 5.2 Hz, 1H), 7.63 (d, *J* = 7.3 Hz, 2H), 7.46–7.41 (m, 3H), 7.38–7.37 (m, 1H), 7.34 (d, *J* = 16.3 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.4, 185.1, 148.3, 137.7, 137.4, 134.3, 131.6, 129.2, 129.1, 128.0, 126.7, 121.1. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{14}H_{10}O_2NaS$ 265.0294; Found 265.0295.; IR (KBr thin film, cm^{-1}): ν 2840, 1652, 1593, 1572, 1261, 1163, 1081,1023, 974, 752, 690, 603.



1f

(*E*)-1-(4-Fluoro-3-methylphenyl)-4-phenylbut-3-ene-1,2-dione (1f):

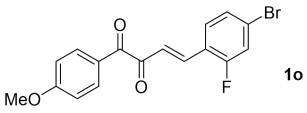
Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v:v = 20:1), amorphous yellow solid, mp 80–81 ºC, 0.93 g, 47% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93–7.88 (m, 2H), 7.71 (d, *J* = 16.4 Hz, 1H), 7.61–7.59 (m, 2H), 7.47–7.40 (m, 3H), 7.16–7.10 (m, 2H), 2.33 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.7, 192.0, 165.5 (d, $^1J_{C-F}$ = 256.6 Hz), 149.1, 134.2 (d, $^3J_{C-F}$ = 7.0 Hz), 134.1, 131.8, 130.6 (d, $^3J_{C-F}$ = 9.7 Hz), 129.3, 129.0, 126.2 (d, $^2J_{C-F}$ = 18.1 Hz), 122.4, 115.9 (d, $^2J_{C-F}$ = 23.4 Hz), 14.7. ^{19}F NMR (376 MHz, CDCl3) δ −105.7. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{13}O_2FNa$ 291.0792; Found 291.0791. IR (KBr thin film, cm^{-1}): ν 3051, 1674, 1637, 1494, 1239, 1109, 975, 835, 765, 606.



1n

(*E*)-1-(Benzo[d][1,3]dioxol-5-yl)-4-(3,5-dimethylphenyl)but-3-ene-1,2-dione (1n):

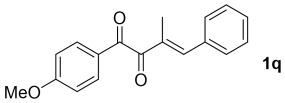
Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v:v = 7:1), amorphous yellow solid, mp 95–97 ºC, 1.21 g, 53% yield. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63–7.59 (m, 2H), 7.51 (d, *J* = 1.4 Hz, 1H), 7.20 (s, 2H), 7.07–7.05 (m, 2H), 6.88 (d, *J* = 8.2 Hz, 1H), 6.08 (s, 2H), 2.33 (s, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 193.3, 191.9, 153.4, 149.4, 148.6, 138.8, 134.1, 133.6, 128.1, 127.7, 126.9, 122.4, 108.9, 108.5, 102.3, 21.3. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{19}H_{16}O_4Na$ 331.0941; Found 331.0945. IR (KBr thin film, cm^{-1}): ν 2911, 1648, 1590, 1487, 1444, 1263, 1037, 971, 835, 764, 605.



1o

(*E*)-4-(4-Bromo-2-fluorophenyl)-1-(4-methoxyphenyl)but-3-ene-1,2-dione (1o):

Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v:v = 5:1), amorphous yellow solid, mp 93–95 ºC, 1.17 g, 44% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04–8.00 (m, 2H), 7.78–7.73 (m, 2H), 7.51–7.47 (m, 1H), 7.22 (d, *J* = 16.5 Hz, 1H), 7.03–6.96 (m, 3H), 3.89 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.2, 191.1, 165.1, 160.7 (d, $^{1}J_{C\text{-}F}$ = 255.9 Hz), 138.7 (d, $^{4}J_{C\text{-}F}$ = 2.2 Hz), 135.5 (d, $^{3}J_{C\text{-}F}$ = 8.6 Hz), 132.9, 131.8, 125.6, 124.4 (d, $^{2}J_{C\text{-}F}$ = 12.8 Hz), 118.3 (d, $^{2}J_{C\text{-}F}$ = 23.6 Hz), 117.3 (d, $^{3}J_{C\text{-}F}$ = 3.0 Hz), 114.4, 55.8. ^{19}F NMR (376 MHz, $CDCl_3$) δ –115.4. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{12}O_3BrFNa$ 384.9846; Found 384.9844. IR (KBr thin film, cm^{-1}): ν 3006, 2156, 1657, 1596, 1510, 1478, 1262, 1159, 764, 750, 635.

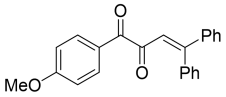


1q

(*E*)-1-(4-Methoxyphenyl)-3-methyl-4-phenylbut-3-ene-1,2-dione (1q):

Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v:v = 10:1), amorphous yellow solid, mp 47–49 ºC, 1.42 g, 69% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.6 Hz, 2H), 7.45–7.36 (m, 6H), 6.98 (d, *J* = 8.6 Hz, 2H), 3.89 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.5, 194.7, 164.9, 147.4, 135.0, 134.5, 132.3,

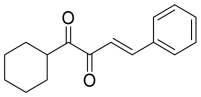
130.4, 129.8, 128.7, 126.7, 114.4, 55.8, 12.3. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{16}O_3Na$ 303.0992; Found 303.0993. IR (KBr thin film, cm^{-1}): ν 3004, 1600, 1574, 1422, 1258, 1185, 1025, 877, 755, 697.



1r

1-(4-Methoxyphenyl)-4,4-diphenylbut-3-ene-1,2-dione (1r):

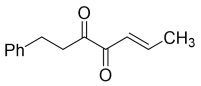
Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v:v = 10:1), amorphous yellow solid, mp 121–123 ºC, 0.96 g, 38% yield. ^1H NMR (600 MHz, $CDCl_3$) δ 7.73–7.71 (m, 2H), 7.42–7.39 (m, 1H), 7.38–7.34 (m, 4H), 7.22–7.18 (m, 1H), 7.12 (d, *J* = 4.4 Hz, 4H), 6.93 (s, 1H), 6.85–6.83 (m, 2H), 3.86 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 194.4, 190.8, 164.3, 159.8, 140.0, 138.0, 132.3, 130.50, 130.45, 129.2, 128.9, 128.6, 128.1, 126.0, 123.6, 113.7, 55.6. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{23}H_{18}O_3Na$ 365.1148; Found 365.1146. IR (KBr thin film, cm^{-1}): ν 3006, 1660, 1595, 1568, 1509, 1260, 1161, 1114, 766, 697.



1t

(*E*)-1-Cyclohexyl-4-phenylbut-3-ene-1,2-dione (1t):

Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v:v = 10:1), amorphous yellow oil, 0.82 g, 46% yield. ^1H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 16.2 Hz, 1H), 7.63–7.61 (m, 2H), 7.45–7.38 (m, 3H), 7.35–7.28 (m, 1H), 3.27–3.21 (m, 1H), 1.91–1.69 (m, 5H), 1.42–1.18 (m, 5H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.2, 189.0, 147.7, 134.5, 131.5, 129.2, 129.0, 119.8, 44.0, 27.8, 25.9, 25.5. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + H]^+$ Calcd for $C_{16}H_{19}O_2$ 243.1380; Found 243.1379. IR (KBr thin film, cm^{-1}): ν 2932, 2856, 1706, 1679, 1597, 1575, 1449, 966, 732, 564.

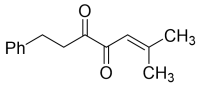


1u

(*E*)-1-Phenylhept-5-ene-3,4-dione (1u):

Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v:v = 20:1), amorphous yellow oil, 0.57 g, 38% yield. ^1H NMR (400 MHz, $CDCl_3$) δ 7.30–7.26

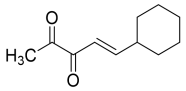
(m, 2H), 7.21–7.19 (m, 3H), 7.16–7.07 (m, 1H), 6.70 (d, *J* = 15.8 Hz, 1H), 3.14 (t, *J* = 7.6 Hz, 2H), 2.94 (t, *J* = 7.5 Hz, 2H), 1.97 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 200.7, 187.6, 149.7, 140.6, 128.6, 128.5, 126.4, 124.7, 38.6, 29.1, 19.3. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{13}H_{14}O_2Na$ 225.0886; Found 225.0885. IR (KBr thin film, cm^{-1}): ν 3028, 1710, 1605, 1453, 1375, 1275, 1260, 1143, 1077, 960, 764, 698.



1v

6-Methyl-1-phenylhept-5-ene-3,4-dione (1v):

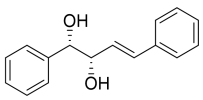
Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v:v = 25:1), amorphous colorless oil, 0.34 g, 21% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.26 (m, 2H), 7.21–7.17 (m, 3H), 6.75 (s, 1H), 3.14 (t, *J* = 7.4 Hz, 2H), 2.92 (t, *J* = 7.6 Hz, 2H), 2.24 (s, 3H), 2.01 (s, 3H) ^{13}C NMR (151 MHz, $CDCl_3$) δ 200.9, 187.1, 164.3, 140.8, 128.6, 128.5, 126.3, 116.8, 37.9, 29.3, 28.8, 21.8. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + H]^+$ Calcd for $C_{14}H_{17}O_2$ 217.1223; Found 217.1224. IR (KBr thin film, cm^{-1}): ν 3028, 1710, 1605, 1497, 1453, 1275, 1260, 1077, 764, 750, 698.



1w

(*E*)-5-cyclohexylpent-4-ene-2,3-dione (1w):

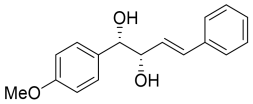
Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v:v = 25:1), amorphous colorless oil, 0.25 g, 19% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (dd, *J* = 16.0, 6.8 Hz, 1H), 6.68 (d, *J* = 16.0 Hz, 1H), 2.37 (s, 3H), 2.26–2.16 (m, 1H), 1.80–1.74 (m, 4H), 1.68 (d, *J* = 12.3 Hz, 1H), 1.35–1.25 (m, 2H), 1.22–1.11 (m, 3H) ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.5, 187.7, 159.1, 120.0, 41.5, 31.6, 26.0, 25.8, 24.7. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + H]^+$ Calcd for $C_{11}H_{17}O_2$ 181.1223; Found 181.1221. IR (KBr thin film, cm^{-1}): ν 2927, 2853, 1694, 1650, 1449, 1275, 1262 1176, 985, 764.



2a

(1*S*,2*S*,*E*)-1,4-Diphenylbut-3-ene-1,2-diol (2a):

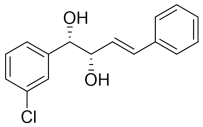
Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v/v = 3:1), amorphous colorless oil, 19.7 mg, 82% yield, 7:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.34 (m, 4H), 7.33–7.27 (m, 5H), 7.25–7.20 (m, 1H), 6.57 (dd, *J* = 16.0, 1.3 Hz, 1H), 6.07 (dd, *J* = 16.0, 5.9 Hz, 1H), 4.60 (d, *J* = 7.0 Hz, 1H), 4.40 (ddd, *J* = 7.1, 6.0, 1.4 Hz, 1H), 2.69 (br, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.3, 136.6, 132.3, 128.7, 128.6, 128.3, 127.9, 127.6, 127.1, 126.6, 78.0, 76.9. HRMS (ESI-Quadrupole-Orbitrap) m/z: [M + Na]$^+$ Calcd for $C_{16}H_{16}O_2Na$ 263.1043; Found 263.1044. $[\alpha]_D^{20}$: −53.0 (c 0.3, $CHCl_3$); HPLC analysis: 92% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 32.2 min, R_t (minor) = 37.9 min. IR (KBr thin film, cm^{-1}): ν 3368, 3053, 1494, 1450, 1265, 1195, 1051, 968, 735, 701, 546.



2b

(1*S*,2*S*,*E*)-1-(4-Methoxyphenyl)-4-phenylbut-3-ene-1,2-diol (2b):

Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v/v = 2:1), amorphous white solid, m.p. 105–107 °C, 24.5 mg, 91% yield, 11:1 dr. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31–7.27 (m, 6H), 7.24–7.20 (m, 1H), 6.89–6.87 (m, 2H), 6.57 (dd, *J* = 15.9, 1.3 Hz, 1H), 6.05 (dd, *J* = 16.0, 5.9 Hz, 1H), 4.54 (d, *J* = 7.2 Hz, 1H), 4.37 (dd, *J* = 6.4, 6.4 Hz, 1H), 3.80 (s, 3H), 2.77 (br, 1H), 2.66 (br, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 159.6, 136.6, 132.4, 132.1, 128.7, 128.3, 127.9, 127.6, 126.6, 114.0, 77.6, 55.4. HRMS (ESI-Quadrupole-Orbitrap) m/z: [M + Na]$^+$ Calcd for $C_{17}H_{18}O_3Na$ 293.1148; Found 293.1149. $[\alpha]_D^{20}$: −106.1 (c 0.4, $CHCl_3$); HPLC analysis: 96% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 54.2 min, R_t (minor) = 60.7 min. IR (KBr thin film, cm^{-1}): ν 3372, 3005, 2926, 1613 1515, 1491 1446, 1275, 1257, 1178, 1015, 824, 750, 570.

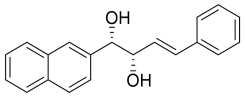


2c

(1*S*, 2*S*, *E*)-1-(3-Chlorophenyl)-4-phenylbut-3-ene-1,2-diol (2c):

Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v/v = 2:1), amorphous colorless oil, 22.5 mg, 82% yield, 5:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ

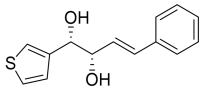
7.38 (s, 1H), 7.30–7.21 (m, 7H), 7.18–7.17 (m, 1H), 6.52 (dd, *J* = 16.0, 0.7 Hz, 1H), 6.03 (dd, *J* = 16.0, 6.2 Hz, 1H), 4.54 (d, *J* = 6.7 Hz, 1H), 4.30 (dd, *J* = 6.0, 6.0 Hz, 1H), 3.25 (br, 1H), 2.91 (br, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.4, 136.3, 134.5, 132.8, 129.7, 128.7, 128.4, 128.1, 127.13, 127.11, 126.6, 125.4. HRMS (ESI-Quadrupole-Orbitrap) m/z: [M + Na]$^+$ Calcd for $C_{16}H_{15}O_2ClNa$ 297.0653; Found 297.0652. $[\alpha]_D^{20}$: −28.1 (c 0.8, $CHCl_3$); HPLC analysis: 87% ee (Chiralpak IA, 2:98 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 57.7 min, R_t (minor) = 64.0 min. IR (KBr thin film, cm^{-1}): ν 3353, 3026, 2891, 1597, 1574, 1448, 1434, 1274, 1263, 1055, 967, 690, 531.



2d

(1*S*, 2*S*, *E*)-1-(Naphthalen-2-yl)-4-phenylbut-3-ene-1,2-diol (2d):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 2:1), amorphous white solid, m.p. 112–115 °C, 25.3 mg, 87% yield, 8:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86–7.82 (m, 4H), 7.49–7.47 (m, 3H), 7.26–7.22 (m, 5H), 6.61 (d, *J* = 15.9 Hz, 1H), 6.11 (dd, *J* = 15.8, 5.6 Hz, 1H), 4.78 (d, *J* = 6.8 Hz, 1H), 4.51 (dd, *J* = 6.2, 6.2 Hz, 1H), 2.92 (br, 1H), 2.65 (br, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.7, 136.5, 133.4, 133.3, 132.3, 128.7, 128.4, 128.2, 127.9, 127.8, 127.5, 126.7, 126.4, 126.3, 126.2, 124.9, 78.0, 76.8. HRMS (ESI-Quadrupole-Orbitrap) m/z: [M + Na]$^+$ Calcd for $C_{20}H_{18}O_2Na$ 313.1199; Found 313.1203. $[\alpha]_D^{20}$: −59.5 (c 0.4, $CHCl_3$); HPLC analysis: 90% ee (Chiralpak IA, 2:98 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 68.2 min, R_t (minor) = 77.6 min. IR (KBr thin film, cm^{-1}): ν 3374, 3054, 1600, 1437, 1264, 1111, 1056, 968, 733, 701.

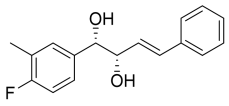


2e

(1*S*,2*S*,*E*)-4-Phenyl-1-(thiophen-3-yl)but-3-ene-1,2-diol (2e):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 3:1), amorphous colorless oil, 19.7 mg, 80% yield, 7:1 dr. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33–7.29 (m, 6H), 7.25–7.22 (m, 1H), 7.11 (dd, *J* = 5.0, 1.3 Hz, 1H), 6.61 (dd, *J* = 15.9, 1.0 Hz, 1H), 6.13 (dd, *J* = 15.9, 6.0 Hz, 1H), 4.72 (dd, *J* = 6.6, 2.6 Hz, 1H), 4.42 (ddd, *J* = 6.4, 6.4, 1.4 Hz, 1H), 2.82 (br, 1H), 2.63 (br, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.7,

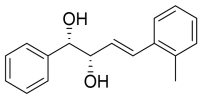
136.5, 132.5, 128.7, 128.0, 127.6, 126.7, 126.4, 126.2, 122.7, 76.4, 74.1. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{14}H_{14}O_2NaS$ 269.0607; Found 269.0607. $[\alpha]_D^{20}$: −10.1 (c 0.3, $CHCl_3$); HPLC analysis: 93% ee (Chiralcel OJ-H, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 76.1 min, R_t (minor) = 67.4 min. IR (KBr thin film, cm^{-1}): ν 3006, 2999, 2156, 2025, 1447, 1275, 1261, 764, 750, 456.



2f

(1*S*,2*S*,*E*)-1-(4-Fluoro-3-methylphenyl)-4-phenylbut-3-ene-1,2-diol (2f):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 2:1), amorphous colorless oil, 22.6 mg, 83% yield, 7:1 dr. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31–7.28 (m, 6H), 7.25–7.20 (m, 1H), 7.14–7.12 (m, 1H), 6.96 (dd, *J* = 9.0, 9.0 Hz, 1H), 6.56 (d, *J* = 16.0 Hz, 1H), 6.05 (dd, *J* = 16.0, 6.0 Hz, 1H), 4.54 (d, *J* = 7.0 Hz, 1H), 4.42 (dd, *J* = 6.2, 6.2 Hz, 1H), 2.92 (br, 1H), 2.67 (br, 1H), 2.26 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 161.2 (d, $^1J_{C-F}$ = 245.1 Hz), 136.5, 135.7 (d, $^4J_{C-F}$ = 3.4 Hz), 132.4, 130.1 (d, $^3J_{C-F}$ = 5.3 Hz), 128.7, 128.0, 127.5, 126.6, 126.0 (d, $^3J_{C-F}$ = 8.2 Hz), 125.0 (d, $^2J_{C-F}$ = 17.5 Hz), 115.0 (d, $^2J_{C-F}$ = 22.3 Hz), 14.8 (d, $^3J_{C-F}$ = 3.1 Hz). ^{19}F NMR (376 MHz, $CDCl_3$) δ −118.3. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{17}O_2FNa$ 295.1105; Found 295.1110. $[\alpha]_D^{20}$: −20.8 (c 1.0, $CHCl_3$); HPLC analysis: 96% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 26.1 min, R_t (minor) = 30.8 min. IR (KBr thin film, cm^{-1}): ν 3376, 3027, 2927, 1598, 1503, 1449, 1275, 1264, 1116, 1057, 968, 820, 684.

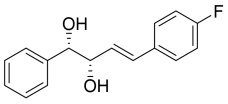


2g

(1S, 2S, *E*)-1-Phenyl-4-(o-tolyl)but-3-ene-1,2-diol (2g) :

Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v/v = 3:1), amorphous colorless oil, 18.2 mg, 73% yield, 7:1 dr. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39–7.34 (m, 4H), 7.33–7.29 (m, 2H), 7.16–7.09 (m, 3H), 6.69 (dd, *J* = 15.8, 1.1 Hz, 1H), 5.93 (dd, *J* = 15.9, 6.2 Hz, 1H), 4.60 (dd, *J* = 7.3, 1.6 Hz, 1H), 4.40 (dd, *J* = 6.6, 6.6 Hz, 1H), 2.88 (d, *J* = 2.6 Hz, 1H), 2.72 (d, *J* = 2.8 Hz, 1H), 2.19 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.3, 135.8, 135.7, 130.6, 130.3, 128.9, 128.6, 128.3, 127.8, 127.2, 126.2,

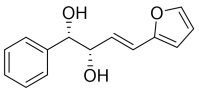
125.9, 78.1, 77.4, 19.8. HRMS (ESI-Quadrupole-Orbitrap) m/z: [M + Na]$^+$ Calcd for $C_{17}H_{18}O_2Na$ 277.1199; Found 277.1201. $[\alpha]_D^{20}$: −4.8 (c 1.5, $CHCl_3$); HPLC analysis: 92% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 26.0 min, R_t (minor) = 31.5 min. IR (KBr thin film, cm^{-1}): ν 3055, 3004, 2987, 1507, 1453, 1264, 1057, 896, 731, 701.



2h

(1*S*,2*S*,*E*)-4-(4-Fluorophenyl)-1-phenylbut-3-ene-1,2-diol (2h):

Purified by chromatography on silica gel (petroleum ether/ethyl acetate, v/v = 3:1), amorphous colorless oil, 23.2 mg, 90% yield, 7:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.28 (m, 5H), 7.26–7.21 (m, 2H), 6.96 (t, *J* = 8.6 Hz, 2H), 6.51 (d, *J* = 16.0 Hz, 1H), 5.96 (dd, *J* = 16.0, 5.9 Hz, 1H), 4.58 (d, *J* = 6.9 Hz, 1H), 4.37 (dd, *J* = 6.2, 6.2 Hz, 1H), 2.95 (br, 1H), 2.80 (br, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.5 (d, $^1J_{C-F}$ = 247.1 Hz), 140.3, 132.8 (d, $^3J_{C-F}$ = 3.3 Hz), 131.1, 128.6, 128.4, 128.1 (d, $^3J_{C-F}$ = 8.0 Hz), 127.3 (d, $^5J_{C-F}$ = 2.1 Hz), 127.0, 115.6 (d, $^2J_{C-F}$ = 21.6 Hz), 78.0, 76.8. ^{19}F NMR (565 MHz, $CDCl_3$) δ −114.02. HRMS (ESI-Quadrupole-Orbitrap) m/z: [M + Na]$^+$ Calcd for $C_{16}H_{15}O_2FNa$ 281.0948; Found 281.0947. $[\alpha]_D^{20}$: − 43.5 (c 0.8, $CHCl_3$); HPLC analysis: 91% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 34.9 min, R_t (minor) = 43.1 min. IR (KBr thin film, cm^{-1}): ν 3377, 3005, 1601, 1509, 1275, 1262, 1229, 970, 764, 750, 702.

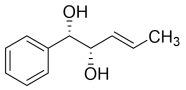


2i

(1*S*,2*S*,*E*)-4-(Furan-2-yl)-1-phenylbut-3-ene-1,2-diol (2i):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 3:1), amorphous colorless oil, 16.3 mg, 71% yield, 7:1 dr. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38–7.34 (m, 4H), 7.32–7.29 (m, 2H), 6.42 (dd, *J* = 15.8, 1.3 Hz, 1H), 6.34 (dd, *J* = 3.3, 1.8 Hz, 1H), 6.19 (d, *J* = 3.2 Hz, 1H), 6.02 (dd, *J* = 15.8, 5.6 Hz, 1H), 4.57 (d, J = 6.8 Hz, 1H), 4.37 (dd, *J* = 6.1, 6.1 Hz, 1H), 2.83 (br, 1H), 2.65 (br, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 152.3, 142.2, 140.2, 128.7, 128.3, 127.1, 126.0, 120.4, 111.4, 108.6, 77.8, 76.5. HRMS (ESI-Quadrupole-Orbitrap) m/z: [M + Na]$^+$ Calcd for $C_{14}H_{14}O_3Na$ 253.0835;

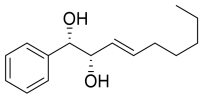
Found 253.0836. $[\alpha]_D^{20}$: −11.1 (c 0.7, $CHCl_3$); HPLC analysis: 90% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 32.8 min, R_t (minor) = 39.4 min. IR (KBr thin film, cm^{-1}): ν 3399, 3061, 2996, 2158, 1669, 1453, 1264, 1013, 896, 731, 701, 595.



2j

(1*S*,2*S*,*E*)-1-Phenylpent-3-ene-1,2-diol (2j):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 3:1), amorphous colorless oil, 14.5 mg, 81% yield, 10:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37–7.29 (m, 5H), 5.68–5.59 (m, 1H), 5.41 (dd, *J* = 15.4, 6.5 Hz, 1H), 4.50 (d, *J* = 6.7 Hz, 1H), 4.16 (dd, *J* = 6.2, 6.2 Hz, 1H), 2.75 (br, 1H), 2.35 (br, 1H), 1.63 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.6, 129.5, 129.3, 128.4, 128.1, 127.1, 77.8, 77.0, 17.9. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + H]^+$ Calcd for $C_{11}H_{15}O_2$ 179.1067; Found 179.1068. $[\alpha]_D^{20}$: −15.8 (c 0.5, $CHCl_3$); HPLC analysis: 97% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 17.7 min, R_t (minor) = 19.4 min. IR (KBr thin film, cm^{-1}): ν 3345, 2973, 2923, 2851, 1668, 1448, 1369, 1274, 1263, 969, 736, 576.

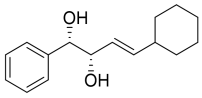


2k

(1*S*,2*S*,*E*)-1-Phenylnon-3-ene-1,2-diol (2k):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 3:1), amorphous colorless oil, 19.9 mg, 85% yield, 20:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.31 (m, 4H), 7.30–7.26 (m, 1H), 5.57 (dtd, *J* = 15.5, 6.9, 1.1 Hz, 1H), 5.35 (ddt, *J* = 15.4, 6.6, 1.4 Hz, 1H), 4.48 (d, *J* = 7.1 Hz, 1H), 4.15 (dd, *J* = 7.0, 7.0 Hz, 1H), 2.87 (br, 1H), 2.48 (br, 1H), 1.99–1.87 (m, 2H), 1.29–1.19 (m, 4H), 1.17–1.09 (m, 2H), 0.85 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.5, 135.0, 128.4, 128.1, 127.9, 127.2, 78.0, 77.2, 32.3, 31.3, 28.7, 22.6, 14.1. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + H]^+$ Calcd for $C_{15}H_{23}O_2$ 235.1693; Found 235.1692. $[\alpha]_D^{20}$: −15.2 (c 1.4, $CHCl_3$); HPLC analysis: 94% ee (Chiralpak IA, 2:98 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 20.1 min, R_t (minor) = 22.5 min. IR (KBr thin film, cm^{-1}): ν 3329, 3003, 2927, 1531,

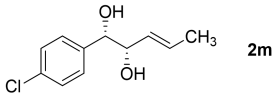
1375, 1261, 764, 701, 502.



2l

(1*S*,2*S*,*E*)-4-Cyclohexyl-1-phenylbut-3-ene-1,2-diol (2l):

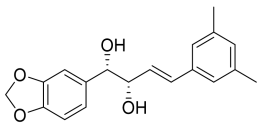
Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 3:1), amorphous colorless oil, 20.5 mg, 83% yield, >20:1 dr. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34–7.30 (m, 4H), 7.29–7.27 (m, 1H), 5.48 (dd, *J* = 15.6, 6.7 Hz, 1H), 5.29 (ddd, *J* = 15.6, 6.6, 0.9 Hz, 1H), 4.47 (d, *J* = 7.1 Hz, 1H), 4.12 (dd, *J* = 7.0, 7.0 Hz, 1H), 2.86 (br, 1H), 2.50 (br, 1H), 1.88–1.83 (m, 1H), 1.66–1.59 (m, 4H), 1.55–1.52 (m, 1H), 1.24–1.16 (m, 2H), 1.13–1.06 (m, 1H), 0.97–0.89 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.50, 140.45, 128.3, 128.0, 127.2, 125.5, 78.0, 77.4, 40.4, 32.7, 32.6, 26.2, 26.0. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{16}H_{22}O_2Na$ 269.1512; Found 269.1513. $[\alpha]_D^{20}$: −22.6 (c 0.6, $CHCl_3$); HPLC analysis: 97% ee (Chiralpak IC, 1:99 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 69.5 min, R_t (minor) = 76.8 min. IR (KBr thin film, cm^{-1}): ν 3372, 2923, 2850, 1668, 1449, 1275, 1264, 1050, 967, 735, 699.



2m

(1*S*,2*S*,*E*)-1-(4-Chlorophenyl)pent-3-ene-1,2-diol (2m):

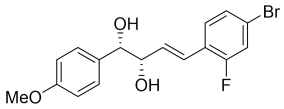
Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 2:1), amorphous colorless oil, 14.9 mg, 70% yield, 7:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.27 (m, 2H), 7.24–7.22 (m, 2H), 5.61–5.52 (m, 1H), 5.36–5.30 (m, 1H), 4.42 (d, *J* = 7.1 Hz, 1H), 4.04 (dd, *J* = 7.0, 7.0 Hz, 1H), 3.22 (br, 1H), 2.70 (br, 1H), 1.62–1.60 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 135.0, 133.7, 131.6, 129.1, 128.9, 127.9, 77.7, 71.1, 19.2. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{11}H_{13}O_2ClNa$ 235.0496; Found 235.0501. $[\alpha]_D^{20}$: −1.8 (c 4.0, $CHCl_3$); HPLC analysis: 83% ee (Chiralcel OJ-H, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 24.2 min, R_t (minor) = 21.8 min. IR (KBr thin film, cm^{-1}): ν 3354, 2979, 2928, 1714, 1592, 1490, 1274, 1263, 1089, 1012, 807, 734, 518.



2n

(1*S*,2*S*,*E*)-1-(Benzo[d][1,3]dioxol-5-yl)-4-(3,5-dimethylphenyl)but-3-ene-1,2-diol (2n):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 2:1), amorphous colorless oil, 24.3 mg, 78% yield, 5:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.92–6.88 (m, 4H), 6.81–6.75 (m, 2H), 6.49 (d, *J* = 16.0 Hz, 1H), 6.03 (dd, *J* = 16.0, 6.2 Hz, 1H), 5.95 (s, 2H), 4.49 (d, *J* = 7.1 Hz, 1H), 4.30 (dd, *J* = 6.4, 6.4 Hz, 1H), 2.96 (br, 1H), 2.70 (br, 1H), 2.28 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.9, 147.5, 138.1, 136.5, 134.3, 132.6, 129.7, 127.1, 124.5, 120.7, 108.2, 107.4, 101.2, 77.7, 77.0, 21.4. HRMS (ESI-Quadrupole-Orbitrap) m/z: [M + Na]$^+$ Calcd for $C_{19}H_{20}O_4Na$ 335.1254; Found 335.1255. $[\alpha]_D^{20}$: −28.1 (c 0.9, $CHCl_3$); HPLC analysis: 91% ee (Chiralcel OJ-H, 5:95 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 42.2 min, R_t (minor) = 36.6 min. IR (KBr thin film, cm^{-1}): ν 3378, 3008, 2893, 1682, 1601, 1487, 1439, 1275, 1243, 1035, 969, 931, 764, 695.

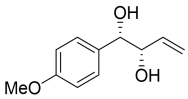


2o

(1*S*,2*S*,*E*)-4-(4-Bromo-2-fluorophenyl)-1-(4-methoxyphenyl)but-3-ene-1,2-diol (2o):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 1:1), amorphous colorless oil, 23.9 mg, 65% yield, 6:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (dd, *J* = 6.6, 2.2 Hz, 1H), 7.29–7.26 (m, 3H), 6.90–6.86 (m, 3H), 6.64 (d, *J* = 16.1 Hz, 1H), 6.12 (dd, *J* = 16.1, 5.6 Hz, 1H), 4.53 (d, *J* = 7.2 Hz, 1H), 4.37 (dd, *J* = 6.0, 6.0 Hz, 1H), 3.80 (s, 3H), 2.83 (br, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 159.7, 159.3 (d, $^1J_{C\text{-}F}$ = 250.3 Hz), 132.2, 131.9 (d, $^3J_{C\text{-}F}$ = 4.6 Hz), 131.7 (d, $^3J_{C\text{-}F}$ = 8.5 Hz), 130.3 (d, $^4J_{C\text{-}F}$ = 3.4 Hz), 128.2, 126.6 (d, $^2J_{C\text{-}F}$ = 13.6 Hz), 123.2 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 117.6 (d, $^2J_{C\text{-}F}$ = 23.9 Hz), 116.8 (d, $^3J_{C\text{-}F}$ = 3.2 Hz), 114.1, 77.5, 76.8, 55.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ −119.8. HRMS (ESI-Quadrupole-Orbitrap) m/z: [M + Na]$^+$ Calcd for $C_{17}H_{16}O_3BrFNa$ 389.0159; Found 389.0155. $[\alpha]_D^{20}$: −2.6 (c 2.0, $CHCl_3$); HPLC analysis: 89% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 44.6 min, R_t (minor) = 55.1 min. IR

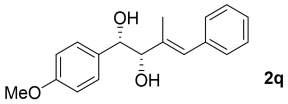
(KBr thin film, cm^{-1}): ν 3388, 3005, 1800, 1612, 1514, 1479, 1264, 1175, 1032, 813, 731, 623.



2p

(1*S*,2*S*)-1-(4-Methoxyphenyl)but-3-ene-1,2-diol (2p):

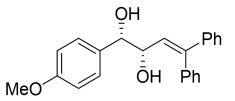
Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 5:1), amorphous colorless oil, 14 mg, 72% yield, 5:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.7 Hz, 2H), 6.88 (d, *J* = 8.6 Hz, 2H), 5.75–5.67 (m, 1H), 5.25 (d, *J* = 17.2 Hz, 1H), 5.14 (d, *J* = 10.7 Hz, 1H), 4.45 (d, *J* = 7.2 Hz, 1H), 4.21 (dd, *J* = 5.7, 5.7 Hz, 1H), 3.80 (s, 3H), 2.68 (br, 1H), 2.48 (br, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 159.6, 136.5, 132.4, 128.3, 117.1, 114.0, 55.4. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{11}H_{14}O_3Na$ 217.0835; Found 217.0831. $[\alpha]_D^{20}$: −3.0 (c 2.5, $CHCl_3$); HPLC analysis: 86% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 27.9 min, R_t (minor) = 32.3 min. IR (KBr thin film, cm^{-1}): ν 3005, 2987, 2164, 1974, 1683, 1601, 1515, 1422, 1264, 896, 732, 703.



2q

(1*S*,2*S*,*E*)-1-(4-Methoxyphenyl)-3-methyl-4-phenylbut-3-ene-1,2-diol (2q):

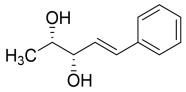
Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 2:1), amorphous colorless oil, 15.3 mg, 53% yield, 4:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.26 (m, 4H), 7.18 (dd, *J* = 7.2, 7.2 Hz, 1H), 7.10 (d, *J* = 7.5 Hz, 2H), 6.86 (d, *J* = 8.5 Hz, 2H), 6.33 (s, 1H), 4.67 (d, *J* = 7.2 Hz, 1H), 4.20 (d, *J* = 7.2 Hz, 1H), 3.79 (s, 3H), 2.82 (br, 1H), 2.75 (br, 1H), 1.75 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.5, 137.3, 136.3, 132.7, 129.0, 128.6, 128.2, 128.0, 126.7, 113.9, 82.3, 76.2, 55.4, 14.9. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{20}O_3Na$ 307.1305; Found 307.1309. $[\alpha]_D^{20}$: −8.4 (c 1.5, $CHCl_3$); HPLC analysis: 97% ee (Chiralcel OJ-H, 5:95 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 42.3 min, R_t (minor) = 38.7 min. IR (KBr thin film, cm^{-1}): ν 3399, 2907, 2836, 1749, 1681, 1512, 1440, 1174, 1031, 829, 750, 698.



2r

(1*S*,2*S*)-1-(4-Methoxyphenyl)-4,4-diphenylbut-3-ene-1,2-diol (2r):

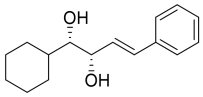
Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 1:1), amorphous colorless oil, 24.5 mg, 71% yield, >20:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27–7.21 (m, 6H), 7.10 (d, *J* = 8.6 Hz, 4H), 6.79 (d, *J* = 8.5 Hz, 2H), 6.74–6.72 (m, 2H), 5.99 (d, *J* = 9.5 Hz, 1H), 4.56 (d, *J* = 7.4 Hz, 1H), 4.16 (d, *J* = 8.5, 8.5 Hz, 1H), 3.77 (s, 3H), 2.96 (br, 1H), 2.67 (br, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 159.5, 146.3, 141.6, 139.0, 132.5, 129.6, 128.3, 128.23, 128.15, 127.9, 127.5, 127.4, 126.3, 113.8, 77.4, 74.3, 55.4. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{23}H_{22}O_3Na$ 369.1461; Found 369.1460. $[\alpha]_D^{20}$: − 77.7 (c 0.4, $CHCl_3$); HPLC analysis: 99% ee (Chiralpak IC, 5:95 ^{i}PrOH/hexanes, 1 mL/min, 220 nm), R_t (major) = 23.5 min, R_t (minor) = 37.7 min. IR (KBr thin film, cm^{-1}): ν 3362, 3054, 2835, 1611, 1585, 1512, 1275, 1246, 1173, 1031, 828, 765, 697.



2s

(2*S*,3*S*,*E*)-5-Phenylpent-4-ene-2,3-diol (2s):

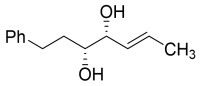
Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 1:1), amorphous colorless oil, 7.8 mg, 44% yield, 8:1 dr. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.3 Hz, 2H), 7.33–7.31 (m, 2H), 7.26 (dd, *J* = 7.2, 5.5 Hz, 1H), 6.67 (d, *J* = 15.9 Hz, 1H), 6.18 (dd, *J* = 16.0, 7.0 Hz, 1H), 4.02 (dd, *J* = 6.9, 6.9 Hz, 1H), 3.76–3.72 (m, 1H), 2.66 (br, 1H), 2.65 (br, 1H), 1.22 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.4, 132.9, 128.8, 128.4, 128.1, 126.7, 77.9, 71.1, 19.1. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + H]^+$ Calcd for $C_{11}H_{15}O_2$ 179.1067; Found 179.1069. $[\alpha]_D^{20}$: −14.8 (c 0.6, $CHCl_3$); HPLC analysis: 91% ee (Chiralcel OJ-H, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 220 nm), R_t (major) = 45.8 min, R_t (minor) = 41.5 min. IR (KBr thin film, cm^{-1}): ν 3377, 2972, 2922, 2851, 1669, 1448, 1369, 1275, 1262, 1020, 969, 870, 749, 577.



2t

(1*S*,2*S*,*E*)-1- Cyclohexyl-4-phenylbut-3-ene-1,2-diol (2t):

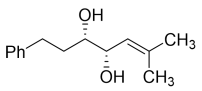
Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 3:1), amorphous colorless oil, 10.1 mg, 41% yield, 3:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.3 Hz, 2H), 7.34–7.30 (m, 2H), 7.27–7.23 (m, 1H), 6.66 (d, *J* = 15.9 Hz, 1H), 6.22 (dd, *J* = 16.0, 6.8 Hz, 1H), 4.31 (dd, *J* = 6.1, 6.1 Hz, 1H), 3.33 (dd, *J* = 5.2, 5.2 Hz, 1H), 2.58 (br, 1H), 2.40 (br, 1H), 1.82–1.74 (m, 3H), 1.66–1.60 (m, 2H), 1.60–1.47 (m, 1H), 1.27–1.08 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.6, 132.3, 129.3, 128.7, 128.0, 126.7, 78.9, 73.2, 39.7, 30.1, 27.3, 26.5, 26.4, 26.2. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{16}H_{22}O_2Na$ 269.1512; Found 269.1511. $[\alpha]_D^{20}$: −5.5 (c 2.1, $CHCl_3$); HPLC analysis: 85% ee (Chiralpak IC, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 220 nm), R_t (major) = 15.7 min, R_t (minor) = 17.5 min. IR (KBr thin film, cm^{-1}): ν 3006, 2928, 1275, 1281, 1031, 764, 750, 531, 440.



2u

(3*S*,4*S*,*E*)-1-Phenylhept-5-ene-3,4-diol (2u):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 3:1), amorphous colorless oil, 11.1 mg, 54% yield, 7:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.26 (m, 2H), 7.21–7.16 (m, 3H), 5.75 (dq, *J* = 13.2, 6.5 Hz, 1H), 5.44 (dd, *J* = 15.3, 7.4 Hz, 1H), 3.87 (dd, *J* = 7.0, 7.0 Hz, 1H), 3.48–3.44 (m, 1H), 2.89–2.82 (m, 1H), 2.71–2.64 (m, 1H), 2.53 (br, 1H), 2.33 (br, 1H), 1.85–1.73 (m, 2H), 1.70 (d, *J* = 6.6 Hz, 3H) ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.2, 130.4, 130.1, 128.6, 128.5, 126.0, 76.6, 74.1, 34.8, 32.1, 18.0. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{13}H_{18}O_2Na$ 229.1199; Found 229.1199. $[\alpha]_D^{20}$: −2.2 (c 3.1, $CHCl_3$); The ee value of the product was measured following derivatization. HPLC analysis: 91% ee (Chiralpak IA, 10:90 ^{i}PrOH/hexanes, 1 mL/min, 220 nm), R_t (major) = 30.1 min, R_t (minor) = 21.5 min. IR (KBr thin film, cm^{-1}): ν 3354, 3026, 2915, 1496, 1453, 1275, 1262, 1072, 1029, 967, 921, 699.

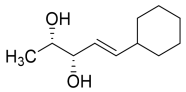


2v

(3*S*,4*S*)-6-Methyl-1-phenylhept-5-ene-3,4-diol (2v):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v =

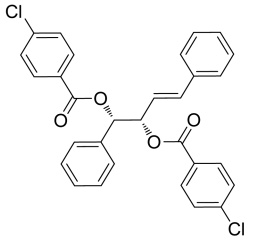
3:1), amorphous colorless oil, 10 mg, 45% yield, 7:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.26 (m, 2H), 7.20–7.16 (m, 3H), 5.13 (d, *J* = 9.0 Hz, 1H), 4.14 (dd, *J* = 8.3, 8.3 Hz, 1H), 3.46–3.42 (m, 1H), 2.90–2.82 (m, 1H), 2.74–2.64 (m, 2H), 2.27 (br, 1H), 1.80–1.73 (m, 4H), 1.69–1.60 (m, 4H) ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.2, 138.4, 128.6, 128.5, 125.9, 124.0, 74.8, 72.4, 34.6, 32.2, 26.1, 18.7. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{14}H_{20}O_2Na$ 243.1356; Found 243.1352. $[\alpha]_D^{20}$: −9.9 (c 0.8, $CHCl_3$); HPLC analysis: 95% ee (Chiralcel OD-H, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 220 nm), R_t (major) = 23.7 min, R_t (minor) = 29.0 min. IR (KBr thin film, cm^{-1}): ν 3371, 3026, 2926, 1697, 1603, 1496, 1453, 1275, 1261, 1029, 841, 764, 699.



2w

(2*S*,3*S*,*E*)-5-Cyclohexylpent-4-ene-2,3-diol (2w):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 4:1), amorphous colorless oil, 12.3 mg, 67% yield, 7:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) 5.70 (dd, *J* = 15.6, 6.7 Hz, 1H), 5.36 (ddd, *J* = 15.6, 7.4, 1.2 Hz, 1H), 3.76 (dd, *J* = 7.3, 7.3 Hz, 1H), 3.63–3.56 (m, 1H), 2.44 (br, 2H), 2.00–1.91 (m, 1H), 1.73–1.61 (m, 5H), 1.32–1.15 (m, 3H), 1.13 (d, *J* = 6.3 Hz, 3H), 1.10–1.01 (m, 2H) ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.0, 126.6, 78.2, 71.1, 40.5, 32.9, 32.8, 26.2, 26.1, 19.0. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{11}H_{20}O_2Na$ 207.1356; Found 207.1361. $[\alpha]_D^{20}$: −5.5 (c 1.1, $CHCl_3$); The ee value of the product was measured following derivatization. HPLC analysis: 91% ee (Chiralpak IC, 10:90 ^{i}PrOH/hexanes, 1 mL/min, 220 nm), R_t (major) = 42.0 min, R_t (minor) = 38.7 min. IR (KBr thin film, cm^{-1}): ν 3369, 2922, 2851, 1767, 1667, 1448, 1397, 1291, 1239, 1179, 1096, 1021, 968, 768, 746.

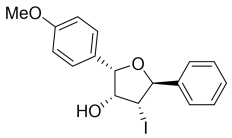


5a

(1*S*,2*S*,*E*)-1,4-Diphenylbut-3-ene-1,2-diyl bis(4-chlorobenzoate) (3a):

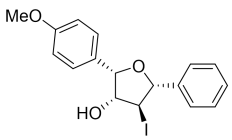
Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v =

4:1), amorphous white solid, mp 113–115 °C, 51 mg, 99% yield. ^{1}H NMR (600 MHz, $CDCl_3$) 7.96 (dd, *J* = 8.6, 2.4 Hz, 4H), 7.50 (d, *J* = 7.3 Hz, 2H), 7.39 – 7.36 (m, 6H), 7.34–7.32 (m, 1H), 7.28–7.25 (m, 4H), 7.24–7.21 (m, 1H), 6.64 (d, *J* = 15.8 Hz, 1H), 6.29 (d, *J* = 7.8 Hz, 1H), 6.17 (dd, *J* = 7.2, 7.2 Hz, 1H), 6.07 (dd, *J* = 15.9, 7.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 164.7, 139.9, 136.0, 135.9, 135.4, 131.2, 129.1, 128.9, 128.8, 128.7, 128.5, 128.33, 128.27, 127.8, 126.8, 122.6, 77.3, 76.3. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{30}H_{22}O_4Cl_2Na$ 539.0787; Found 539.0782. $[\alpha]_D^{20}$: −120.0 (c 0.05, $CHCl_3$); HPLC analysis: 90% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 26.9 min, R_t (minor) = 31.9 min. IR (KBr thin film, cm^{-1}): ν 3368, 2961, 2924, 2312, 1721, 1594, 1488, 1455, 1400, 1258, 1090, 1014, 797, 756, 697.



3b

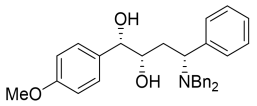
(2*S*,3*R*,4*R*,5*S*)-4-Iodo-2-(4-methoxyphenyl)-5-phenyltetrahydrofuran-3-ol (3b): Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 2:1), amorphous white solid, mp 102–104 °C, 24.2 mg, 61% yield, 2:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) 7.57–7.50 (m, 2H), 7.43–7.33 (m, 5H), 6.98–6.90 (m, 2H), 5.53 (d, *J* = 3.0 Hz, 1H), 5.47 (d, *J* = 10.2 Hz, 1H), 4.40–4.35 (m, 1H), 4.26 (dd, *J* = 10.2, 3.4 Hz, 1H), 3.82 (s, 3H), 1.86 (d, *J* = 1.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.8, 139.1, 128.69, 128.65, 128.5, 128.1, 126.8, 114.1, 86.8, 83.0, 75.6, 55.4, 36.1. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{17}O_3INa$ 419.0115; Found 419.0114. $[\alpha]_D^{20}$: −78.3 (c 0.08, $CHCl_3$); HPLC analysis: 99% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), R_t (major) = 12.2 min, R_t (minor) = 16.6 min. IR (KBr thin film, cm^{-1}): ν 3458, 2957, 2923, 2853, 1613, 1514, 1456, 1259, 1240, 1071, 1014, 843, 795, 703.



3b'

(2*S*,3*R*,4*S*,5*R*)-4-Iodo-2-(4-methoxyphenyl)-5-phenyltetrahydrofuran-3-ol (3b'): Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v =

2:1), amorphous white solid, mp 101–103 °C, 8.3 mg, 21% yield. NMR (400 MHz, $CDCl_3$) 7.63–7.52 (m, 2H), 7.49–7.30 (m, 5H), 6.97 (d, *J* = 8.7 Hz, 2H), 5.38 (d, *J* = 6.7 Hz, 1H), 5.30 (d, *J* = 4.0 Hz, 1H), 4.68 (d, *J* = 2.4 Hz, 1H), 4.08 (dd, *J* = 6.7, 2.3 Hz, 1H), 3.83 (s, 3H), 1.59 (d, *J* = 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.9, 139.2, 128.8, 128.7, 128.5, 127.1, 126.7, 114.3, 89.7, 83.4, 82.8, 55.57, 33.7. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{17}O_3INa$ 419.0115; Found 419.0111. [α]D 20: −134.7 (c 0.05, $CHCl_3$); HPLC analysis: 95% ee (Chiralpak IA, 3:97 ^{i}PrOH/hexanes, 1 mL/min, 254 nm), Rt (major) = 25.0 min, Rt (minor) = 30.0 min. IR (KBr thin film, cm^{-1}): ν 3369, 2922, 2851, 2031, 1970, 1614, 1514, 1456, 1248, 1173, 1026, 794, 759, 698.



4b

(1*S*,2*S*,4*R*)-4-(Dibenzylamino)-1-(4-methoxyphenyl)-4-phenylbutane-1,2-diol (4b):

Purified by chromatogrzaphy on silica gel (petroleum ether/ethyl acetate, v/v = 2:1), amorphous colorless oil, 42 mg, 90% yield. ^1H NMR (600 MHz, $CDCl_3$) 7.43–7.34 (m, 11H), 7.30–7.28 (m, 2H), 7.24 (d, *J* = 8.3 Hz, 2H), 7.17 (d, *J* = 7.0 Hz, 2H), 6.81 (d, *J* = 8.5 Hz, 2H), 4.44 (d, *J* = 6.7 Hz, 1H), 4.02 (d, *J* = 13.0 Hz, 3H), 3.82–3.72 (m, 4H), 3.32 (s, 1H), 2.94 (d, *J* = 13.0 Hz, 2H), 2.49–2.31 (m, 1H), 1.37 (d, *J* = 14.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.3, 138.2, 135.8, 133.0, 129.5, 129.4, 128.9, 128.4, 128.3, 127.9, 127.6, 113.9, 78.1, 77.4, 62.4, 55.3, 54.0, 32.8. HRMS (ESI-Quadrupole-Orbitrap) m/z: $[M + H]^+$ Calcd for $C_{31}H_{34}O_3N$ 468.2533; Found 468.2530. $[\alpha]_D^{20}$: +22.3 (c 0.4, $CHCl_3$). IR (KBr thin film, cm^{-1}): ν 3304, 3061, 3026, 2835, 1970, 1610, 1512, 1494, 1452, 1247, 1105, 1028, 831, 746, 697.

VIII. ^{1}H NMR and ^{13}C NMR spectra of new compounds

jcy250320-sf-s3
single_pulse

8.5123
8.5109
8.5078
7.8340
7.8070
7.7264
7.7178
7.6393
7.6272
7.4594
7.4478
7.4340
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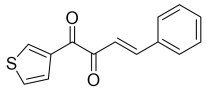
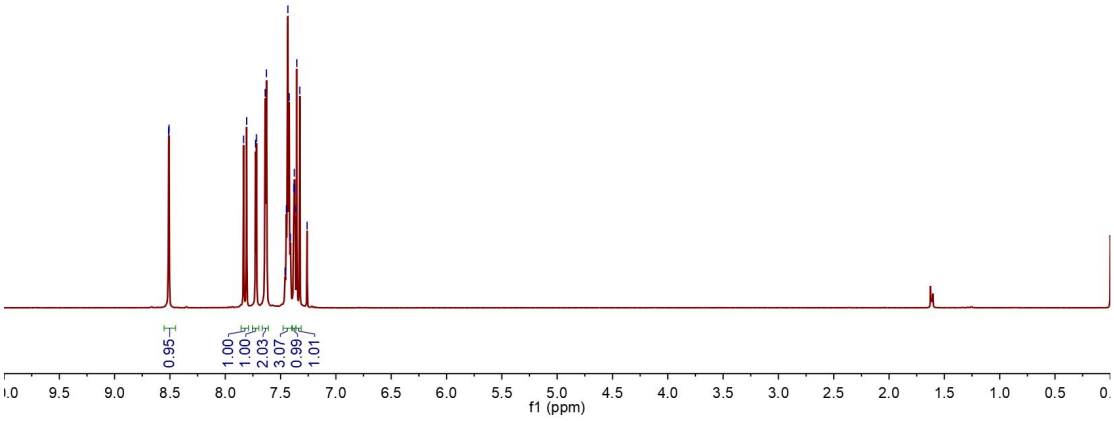
7.3720
7.3672
7.3533
7.3262
7.2603

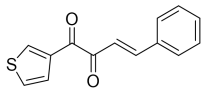


1e
600 MHz, $CDCl_3$



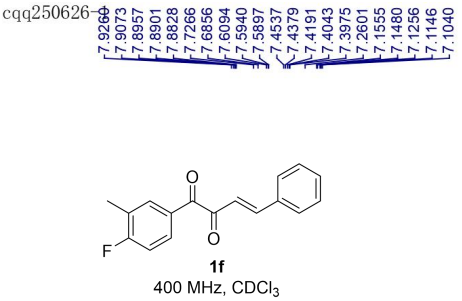
JCY-250320-SF-S2
single pulse decoupled gated NOE

190.34
185.11
148.33
137.74
137.43
134.30
131.61
129.20
129.07
127.98
126.70
121.09
77.37
77.16
76.95



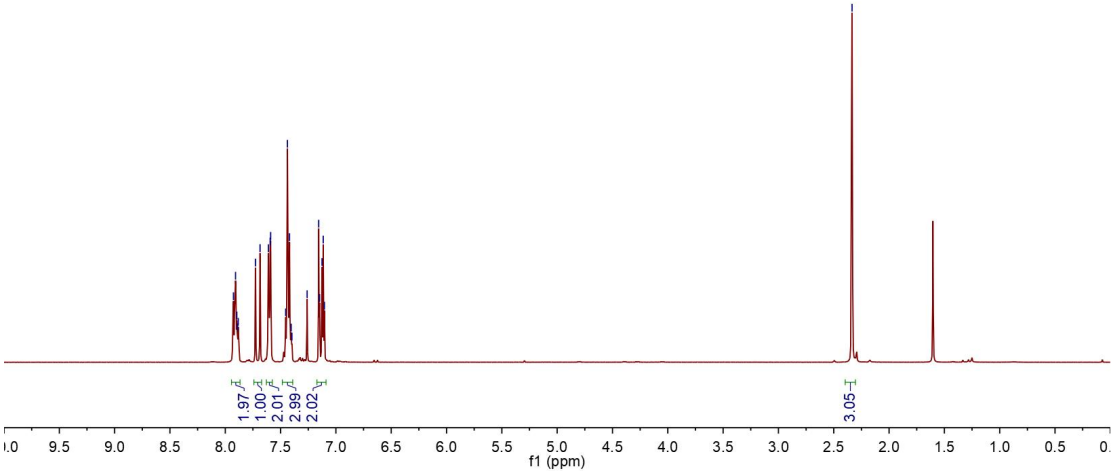
1e
151 MHz, $CDCl_3$

cqq250626



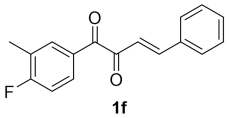
1f

400 MHz, $CDCl_3$



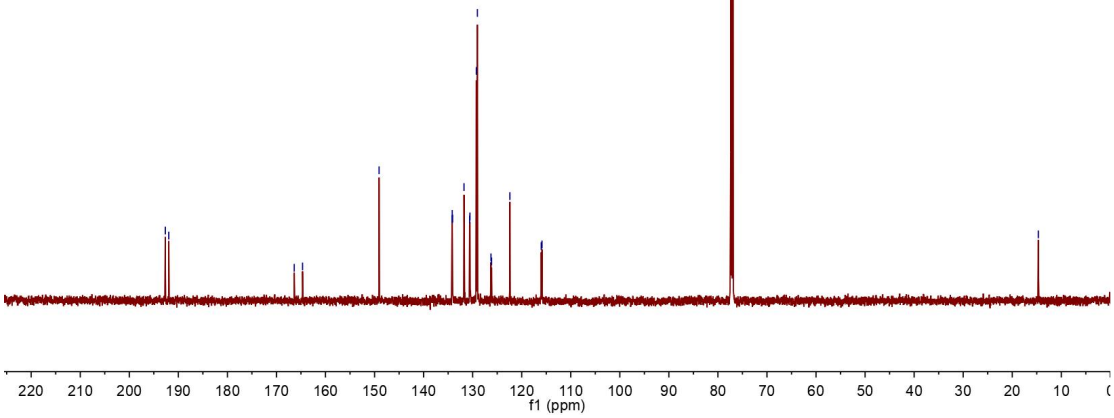
CQQ250625-2

single pulse decoupled gated C13

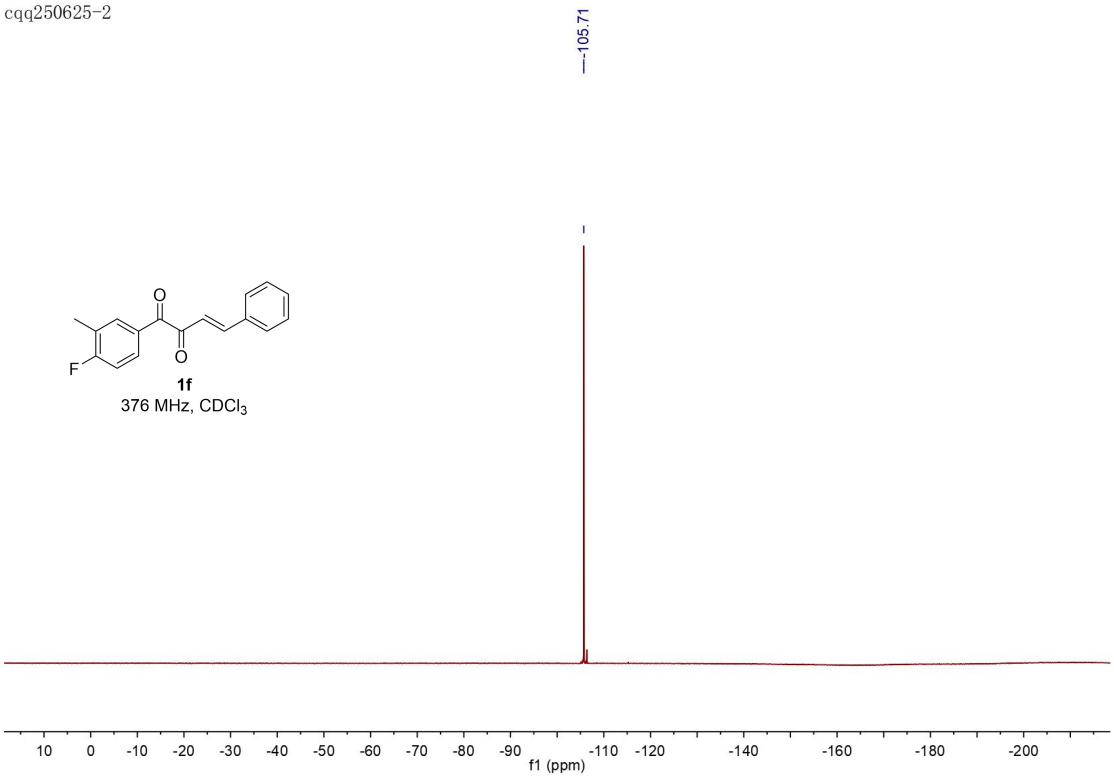


1f

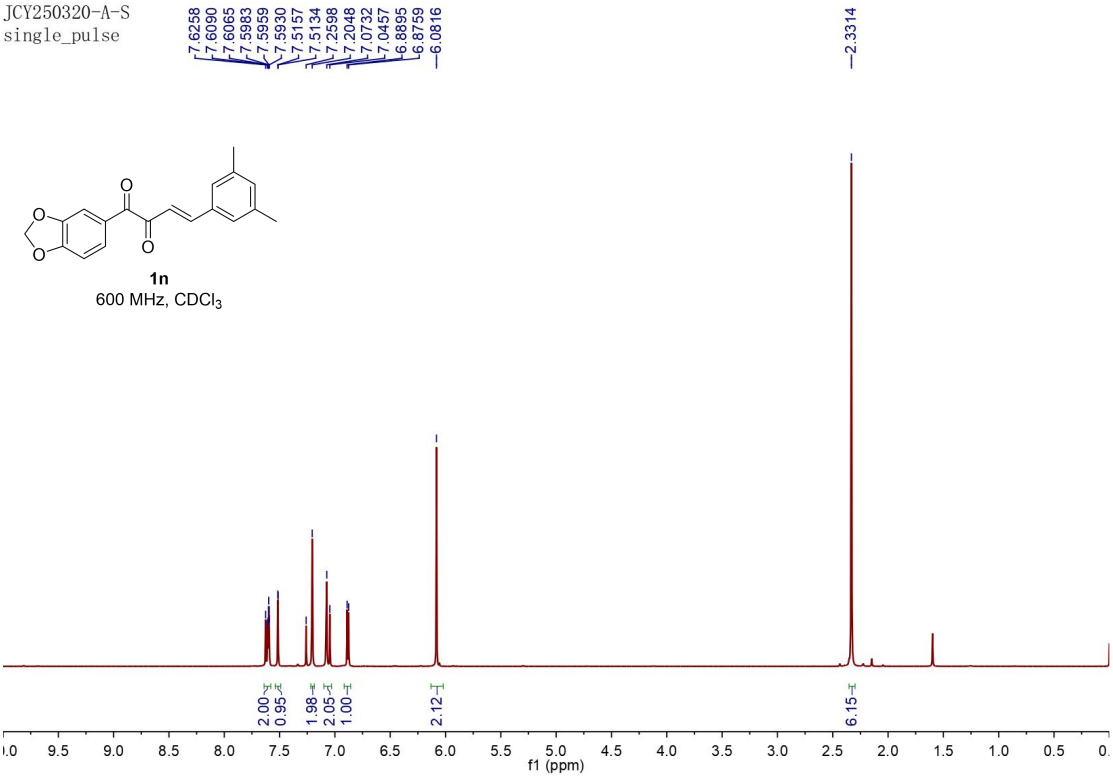
151 MHz, $CDCl_3$



cqq250625-2

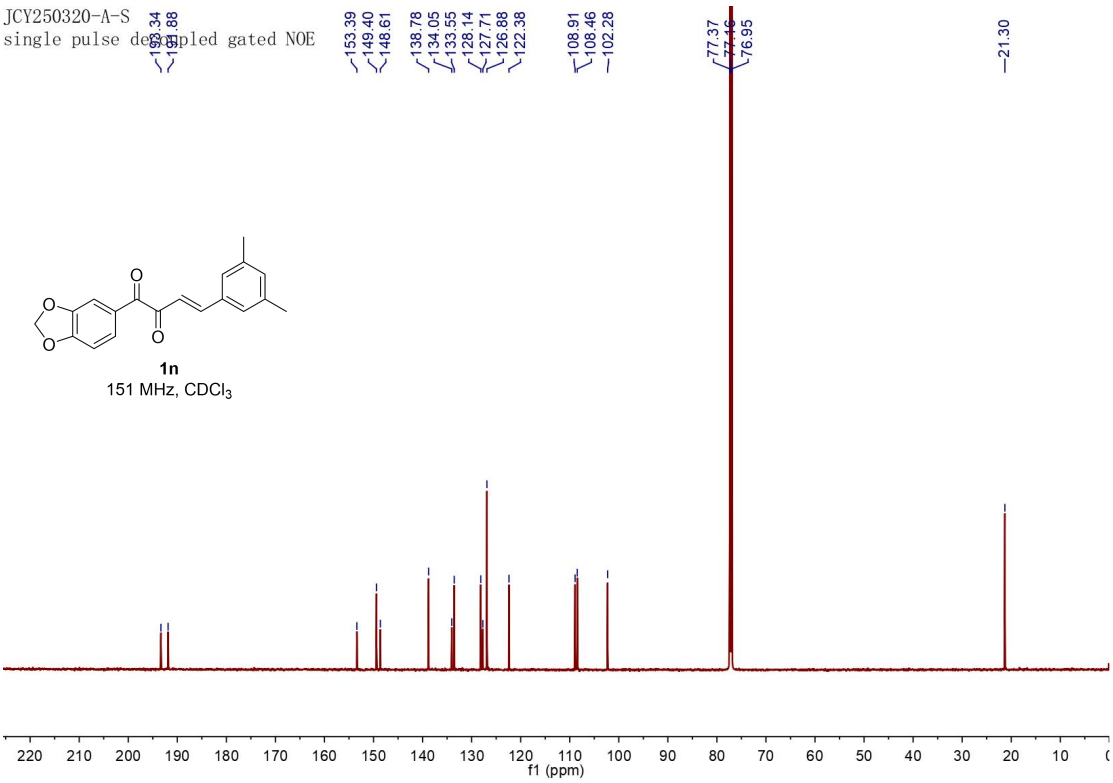

-105.71
O
O
F
1f
376 MHz, CDCl3
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-120
-140
-160
-180
-200
f1 (ppm)

JCY250320-A-S
single_pulse

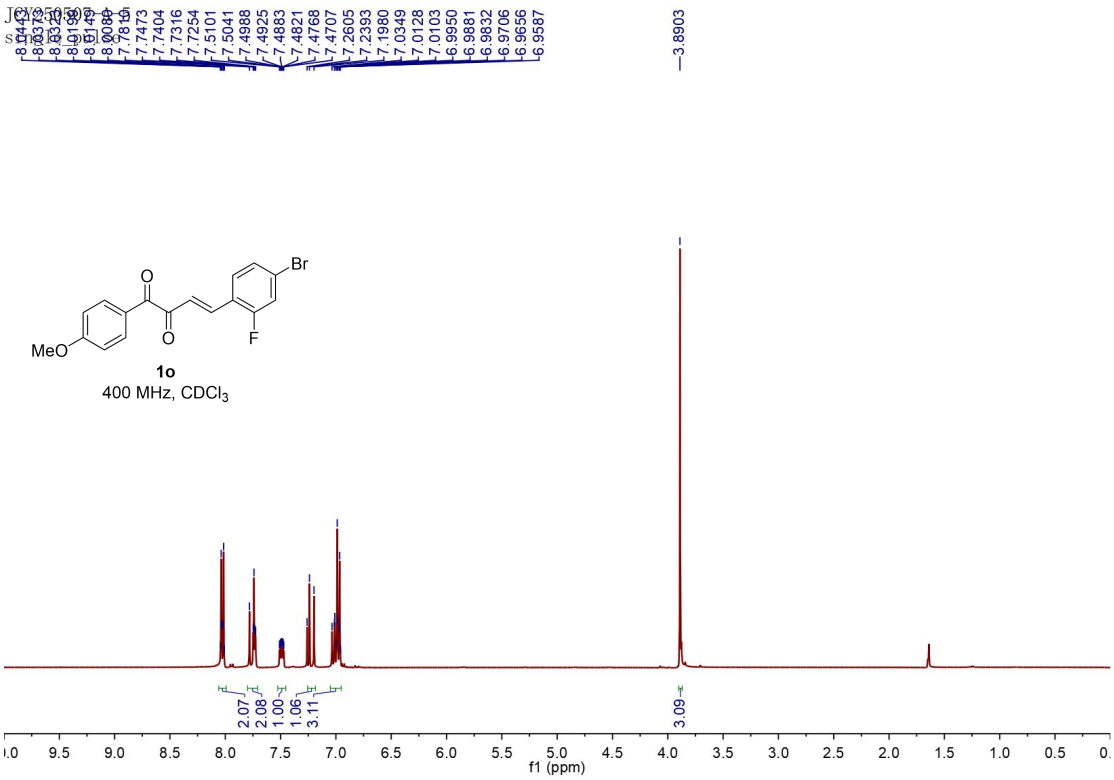


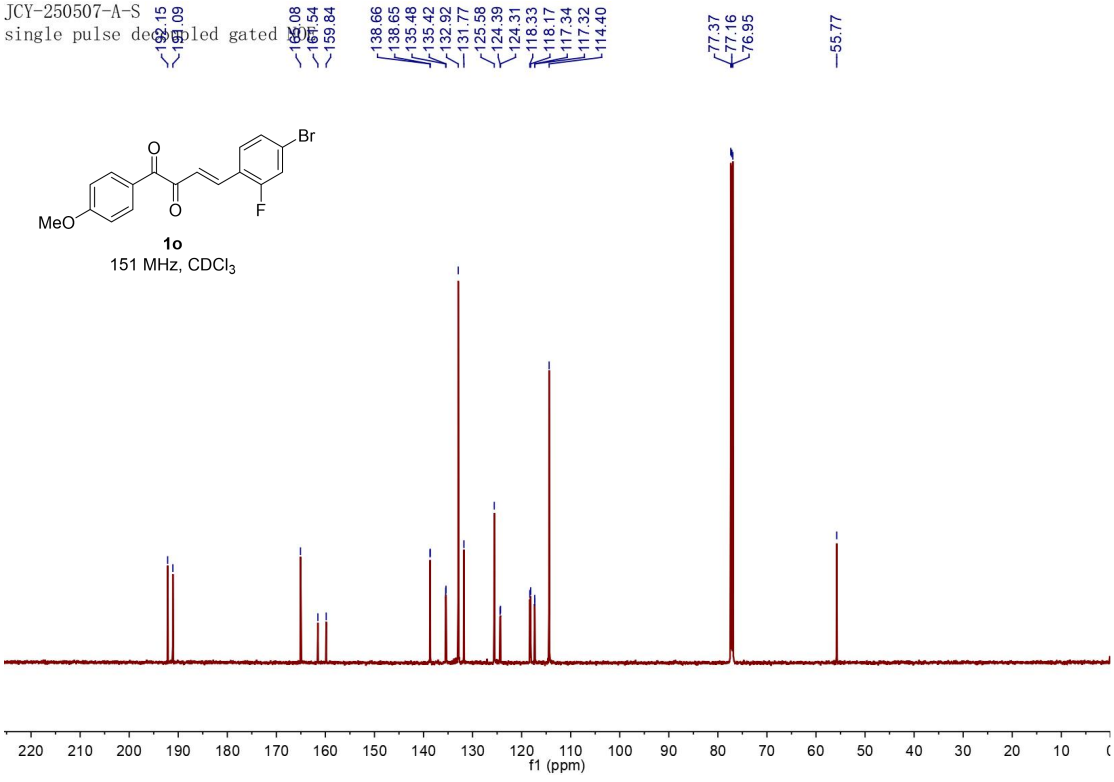
1n
600 MHz, $CDCl_3$

JCY250320-A-S
single pulse decoupled gated NOE

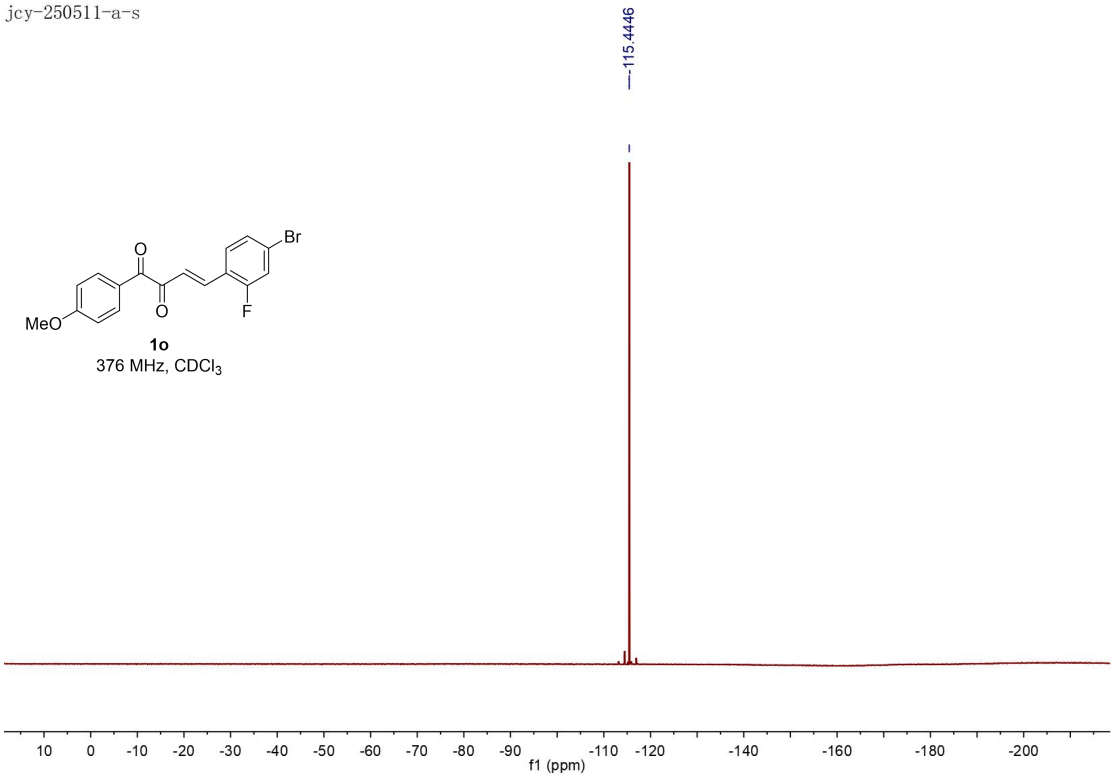


1n
151 MHz, $CDCl_3$


3.8903
Br
O
O
F
MeO
1o
400 MHz, CDCl3
2.07
2.08
1.00
1.06
3.11
3.09
f1 (ppm)


77.37
77.16
76.95
55.77
Br
O
O
F
MeO
1o
151 MHz, CDCl3
f1 (ppm)

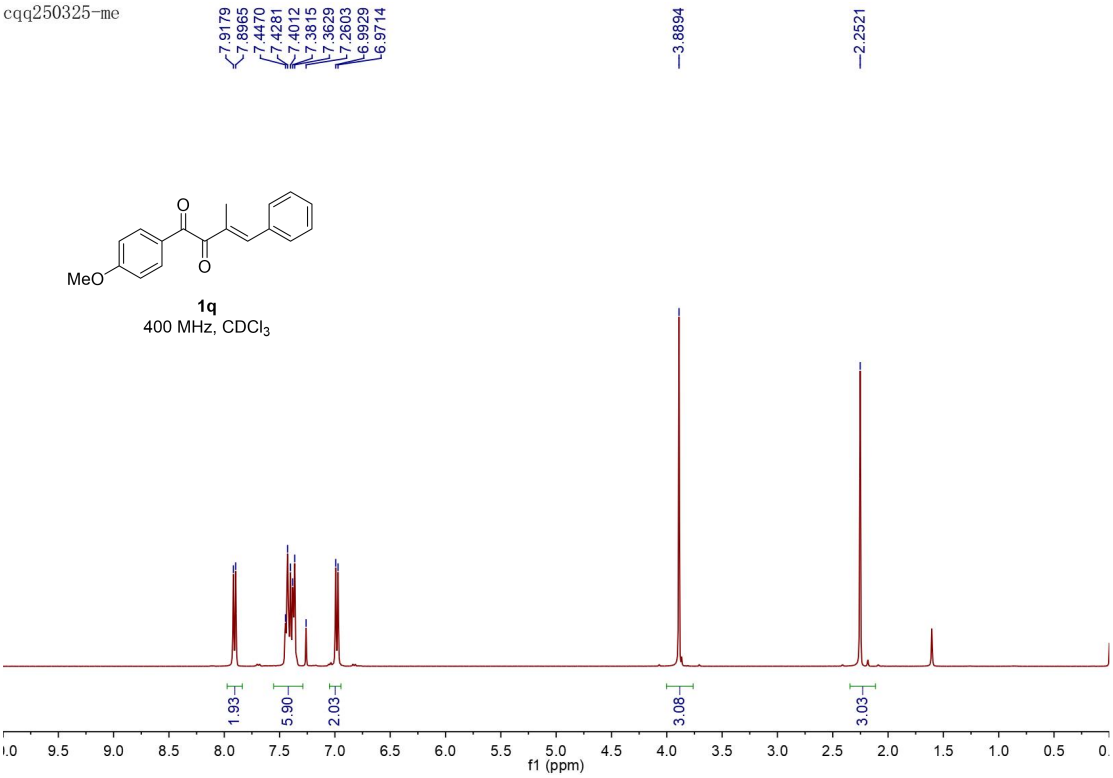
jcy-250511-a-s



1o

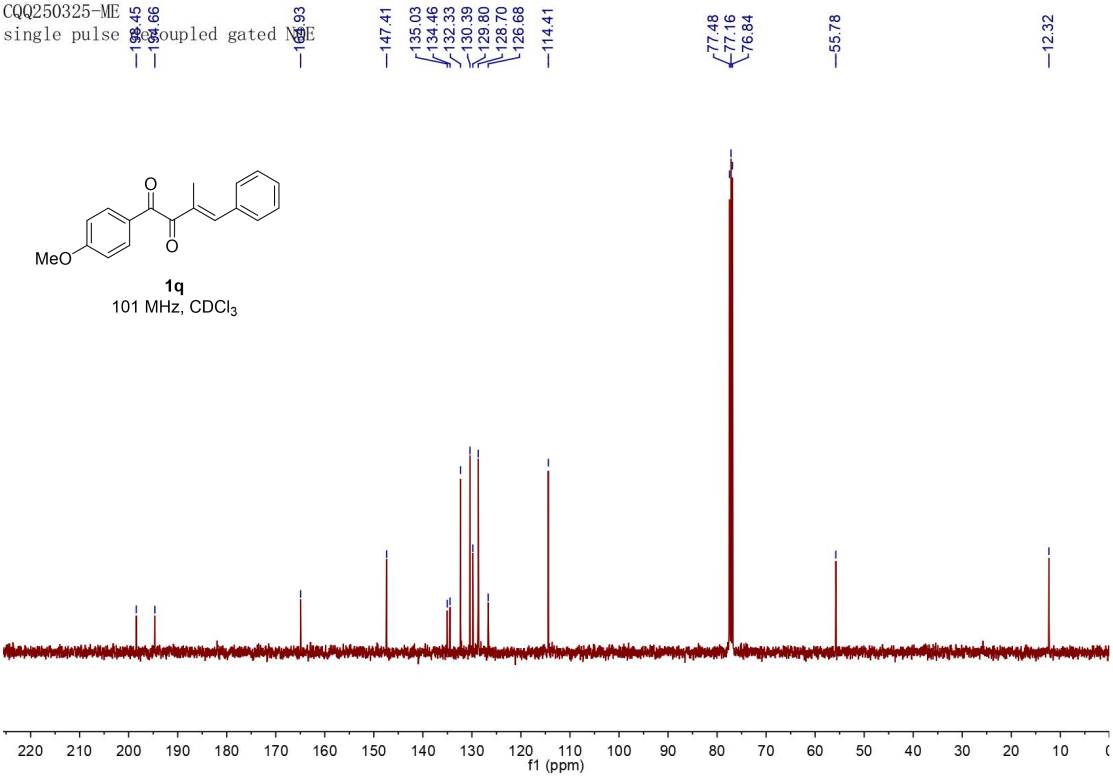
376 MHz, $CDCl_3$

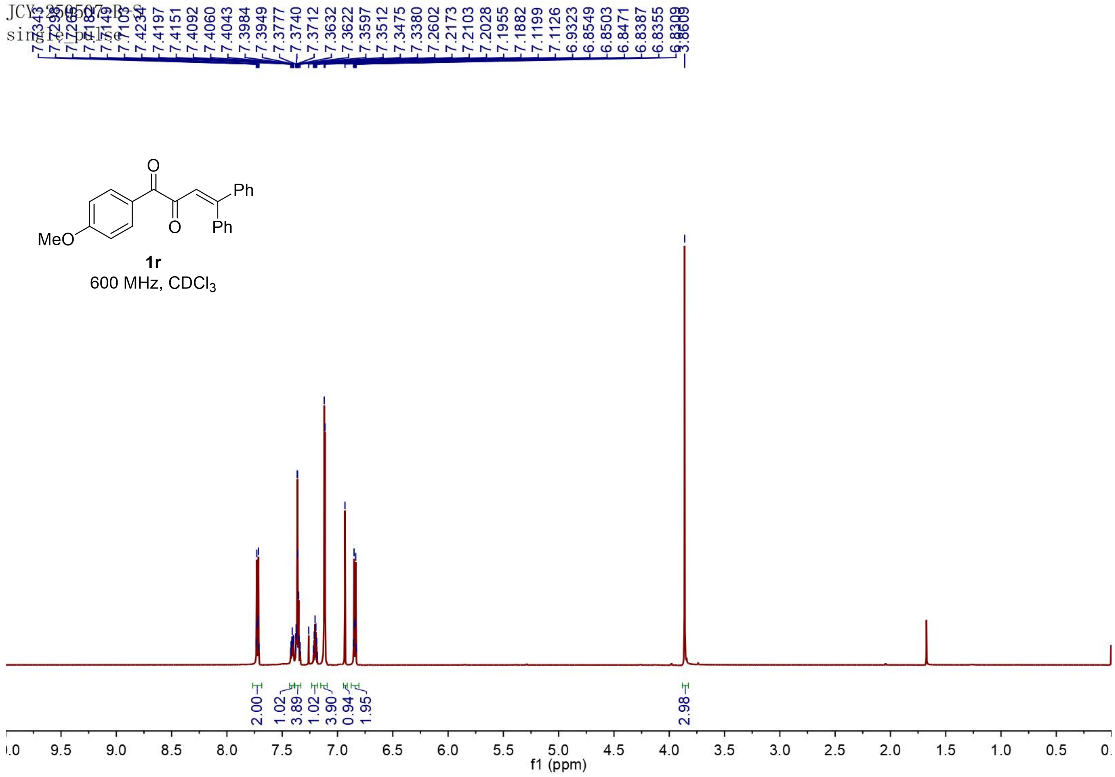
cqq250325-me

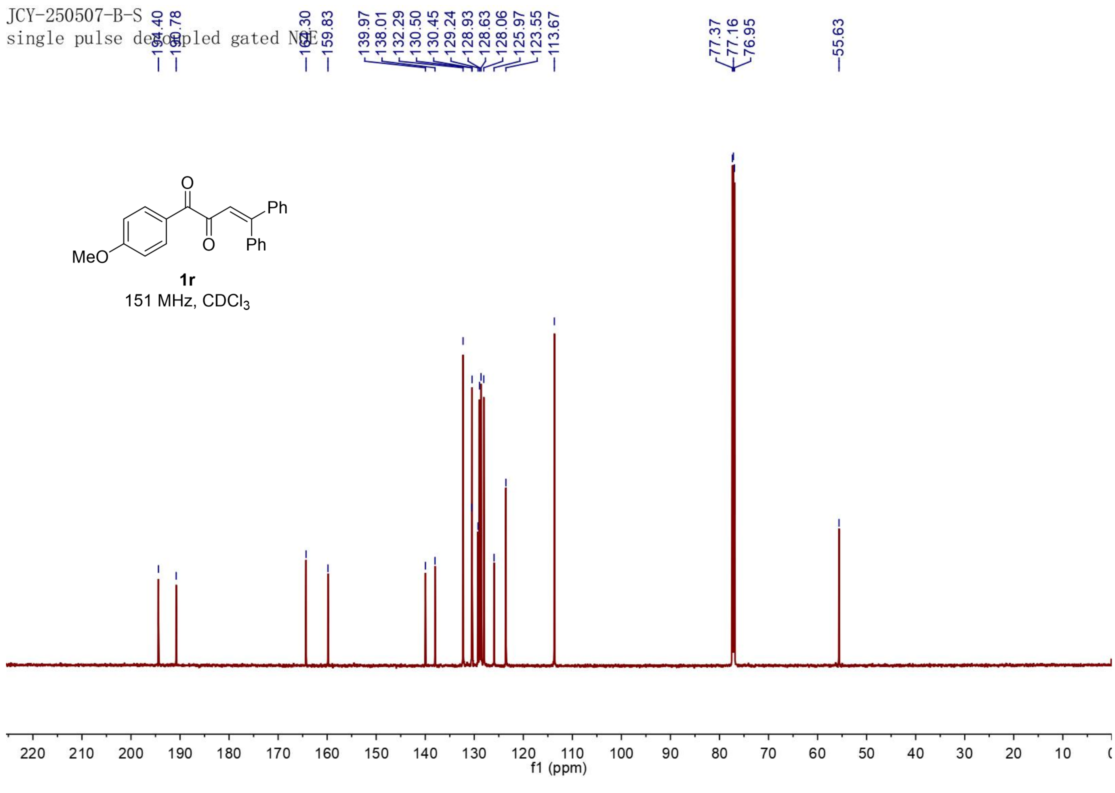


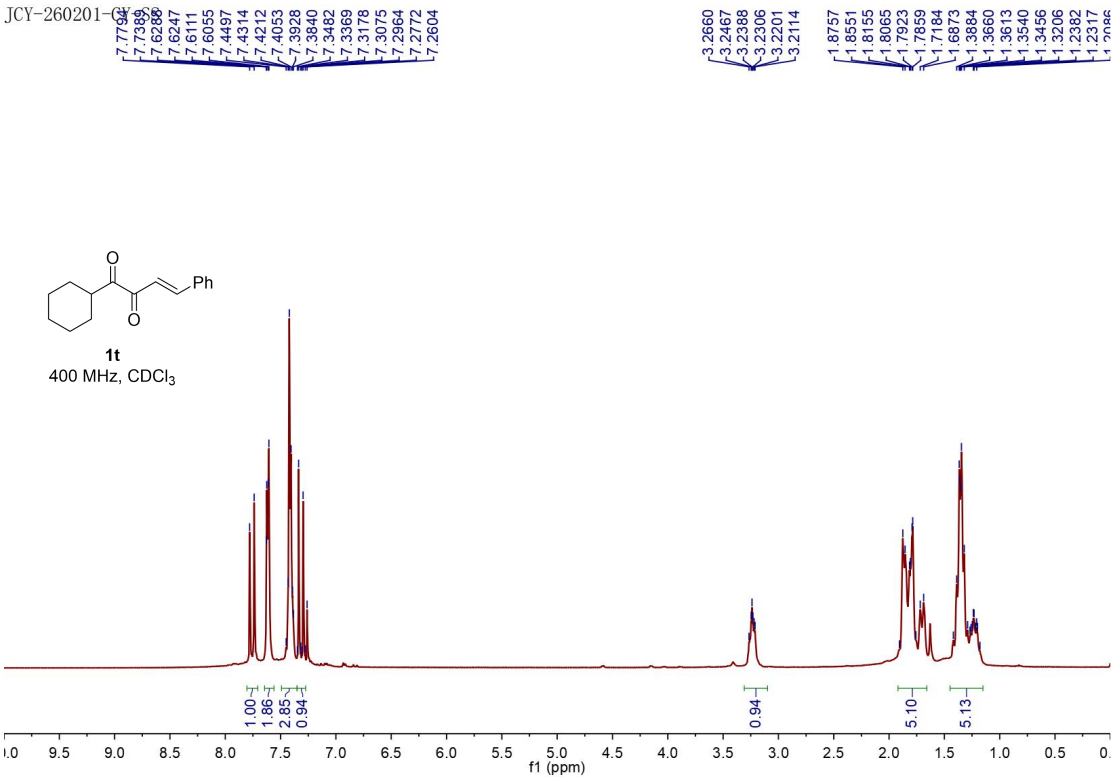
CQQ250325-ME

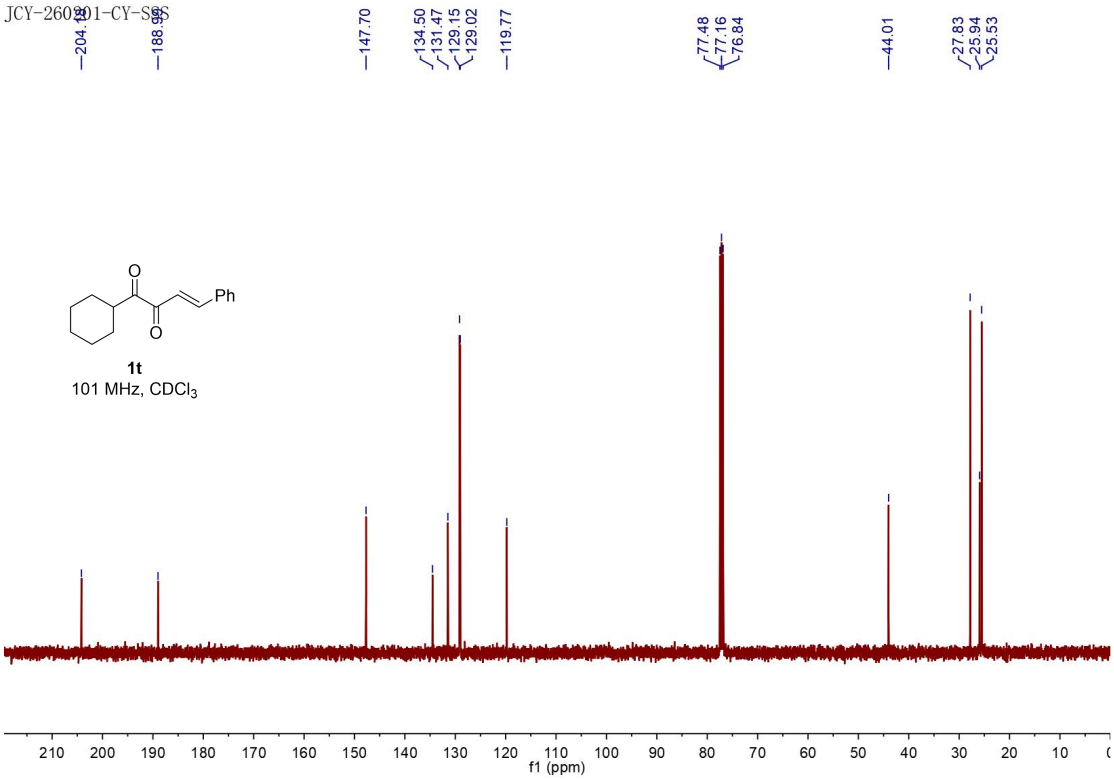
single pulse decoupled gated NOE



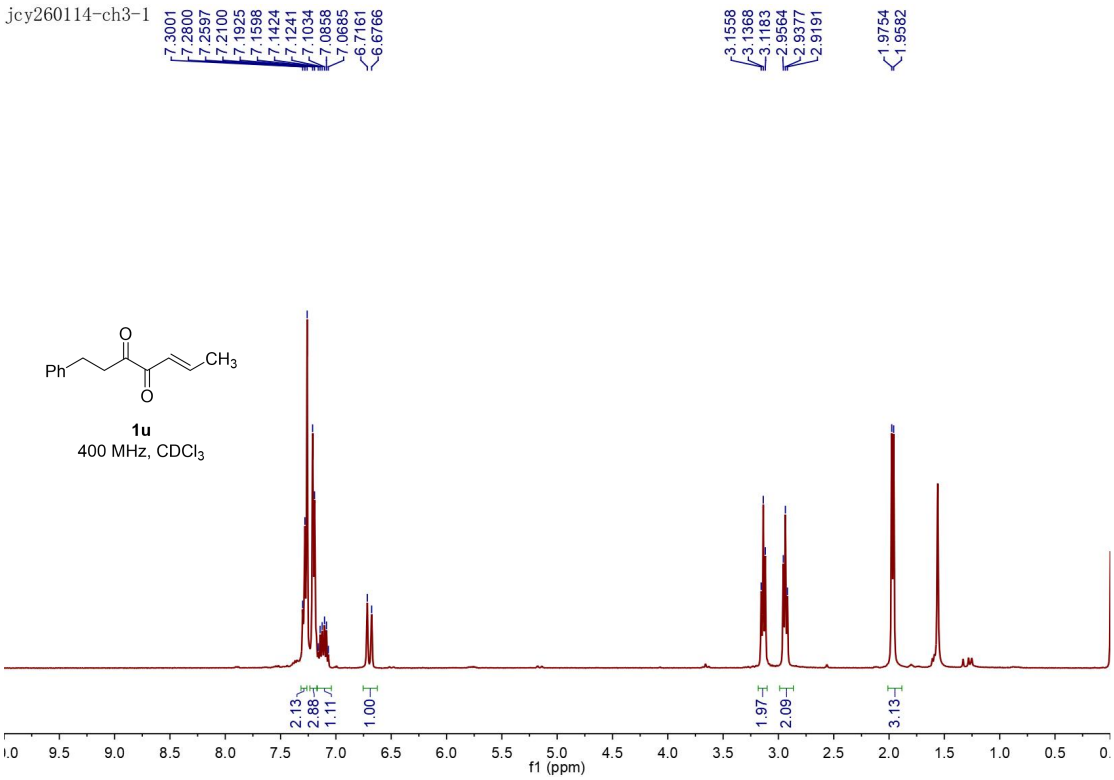

JCY-250507-B-S
single pulse
1r
600 MHz, CDCl$_3$
MeO
O
Ph
2.00
1.02
3.89
1.02
3.90
0.94
1.95
2.98
f1 (ppm)


JCY-250507-B-S
single pulse decoupled gated NOE
159.83
139.97
138.01
132.29
130.50
130.45
129.24
128.93
128.63
128.06
125.97
123.55
113.67
77.37
77.16
76.95
55.63
1r
151 MHz, CDCl$_3$
MeO
O
Ph
f1 (ppm)

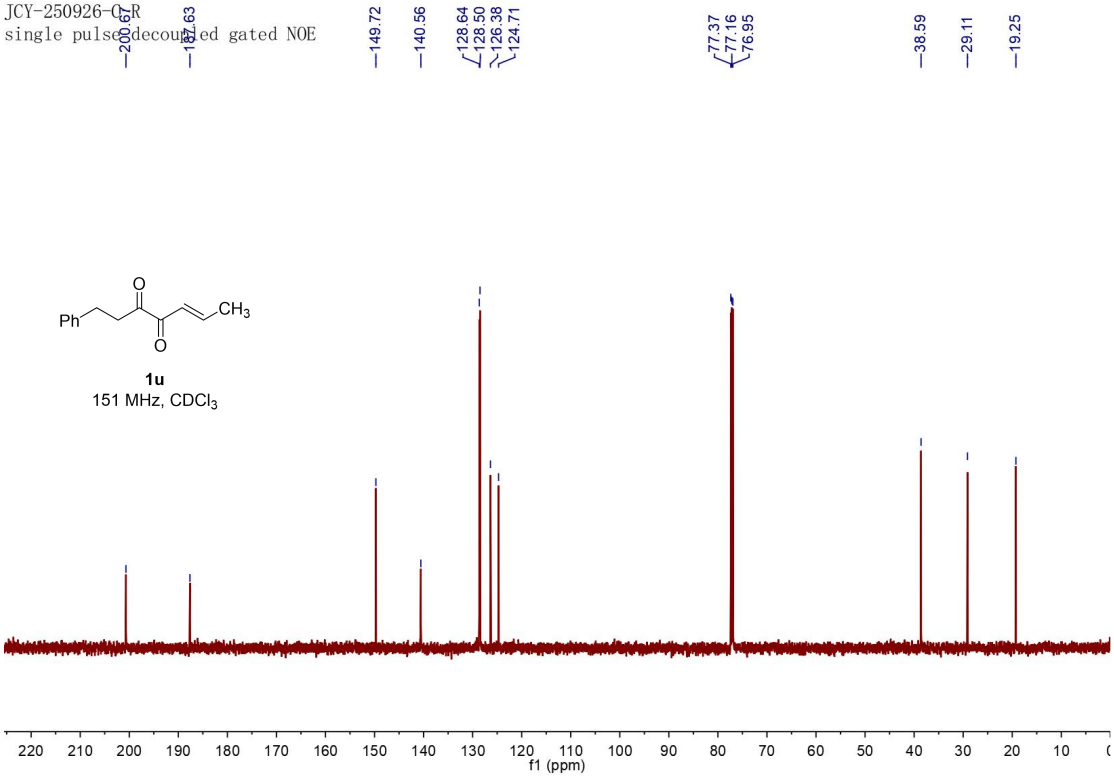

JCY-260201-CY-S2S
7.7794
7.7389
7.6288
7.6247
7.6111
7.6055
7.4497
7.4314
7.4212
7.4053
7.3928
7.3840
7.3482
7.3369
7.3178
7.3075
7.2964
7.2772
7.2604
3.2660
3.2467
3.2388
3.2306
3.2201
3.2114
1.8757
1.8551
1.8155
1.8065
1.7923
1.7859
1.7184
1.6873
1.3884
1.3660
1.3613
1.3540
1.3456
1.3206
1.2382
1.2317
O
O
Ph
1t
400 MHz, CDCl3
1.00
1.86
2.85
0.94
0.94
5.10
5.13
f1 (ppm)


JCY-260201-CY-S3S
204.12
188.95
147.70
134.50
131.47
129.15
129.02
119.77
77.48
77.16
76.84
44.01
27.83
25.94
25.53
O
O
Ph
1t
101 MHz, CDCl3
f1 (ppm)

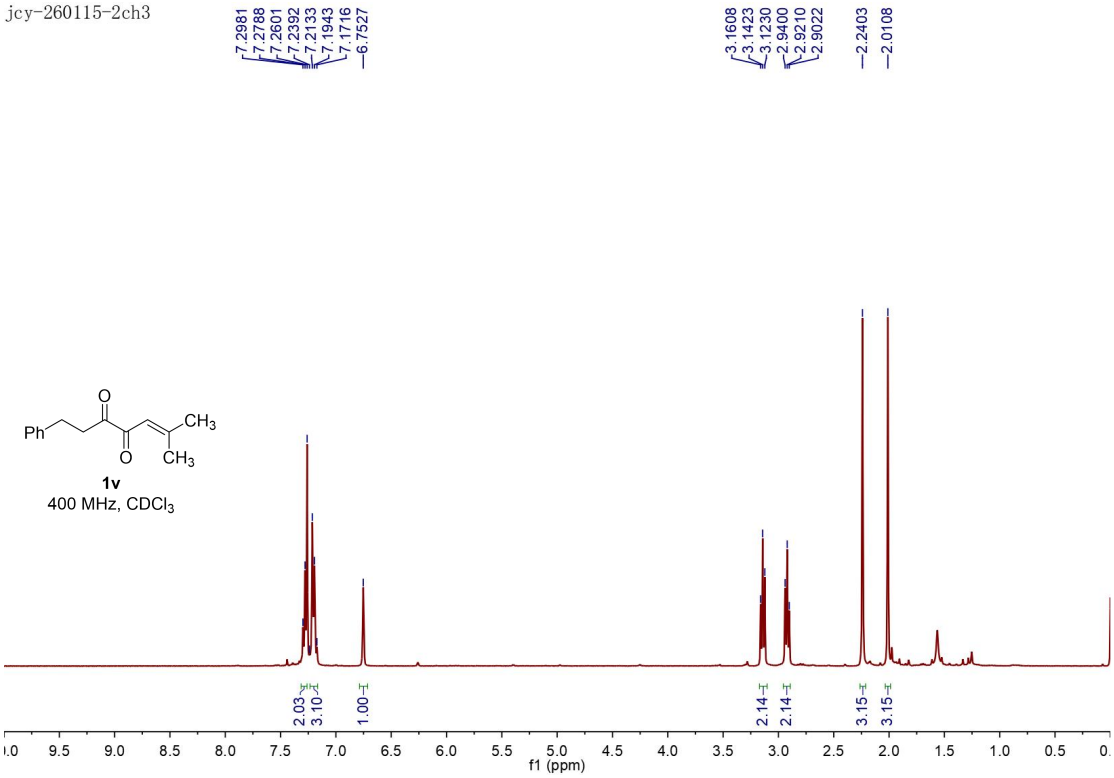
jcy260114-ch3-1



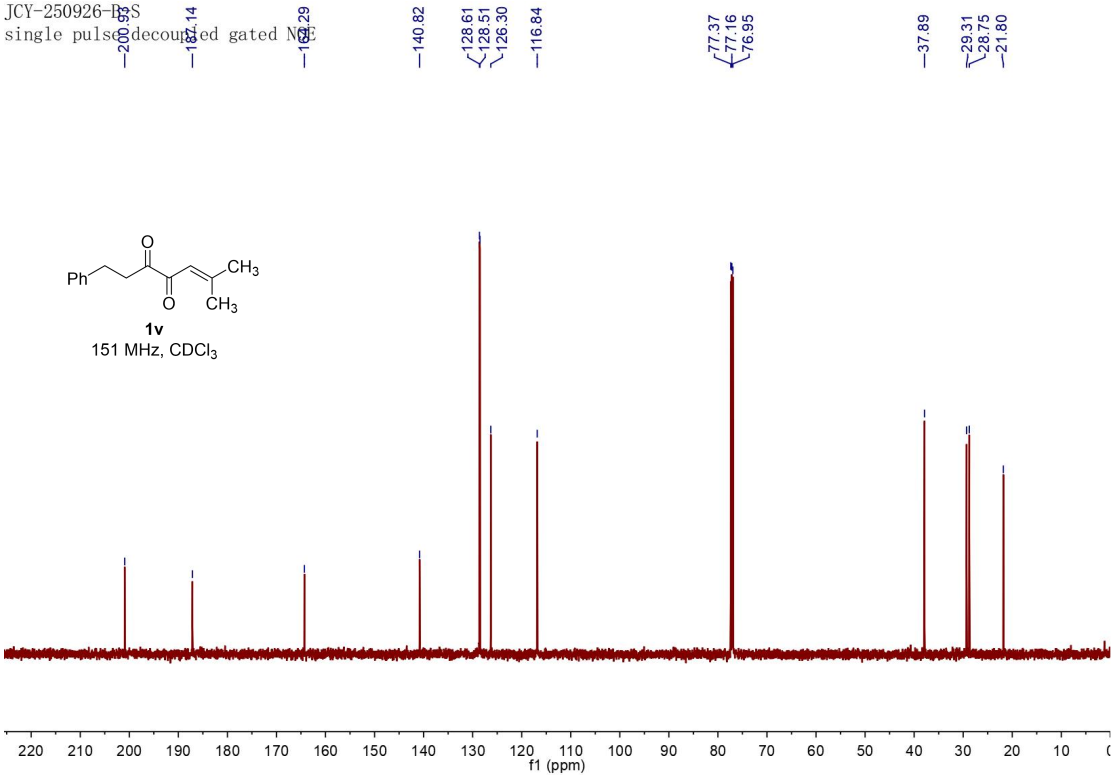
JCY-250926-CAR
single pulse decoupled gated NOE



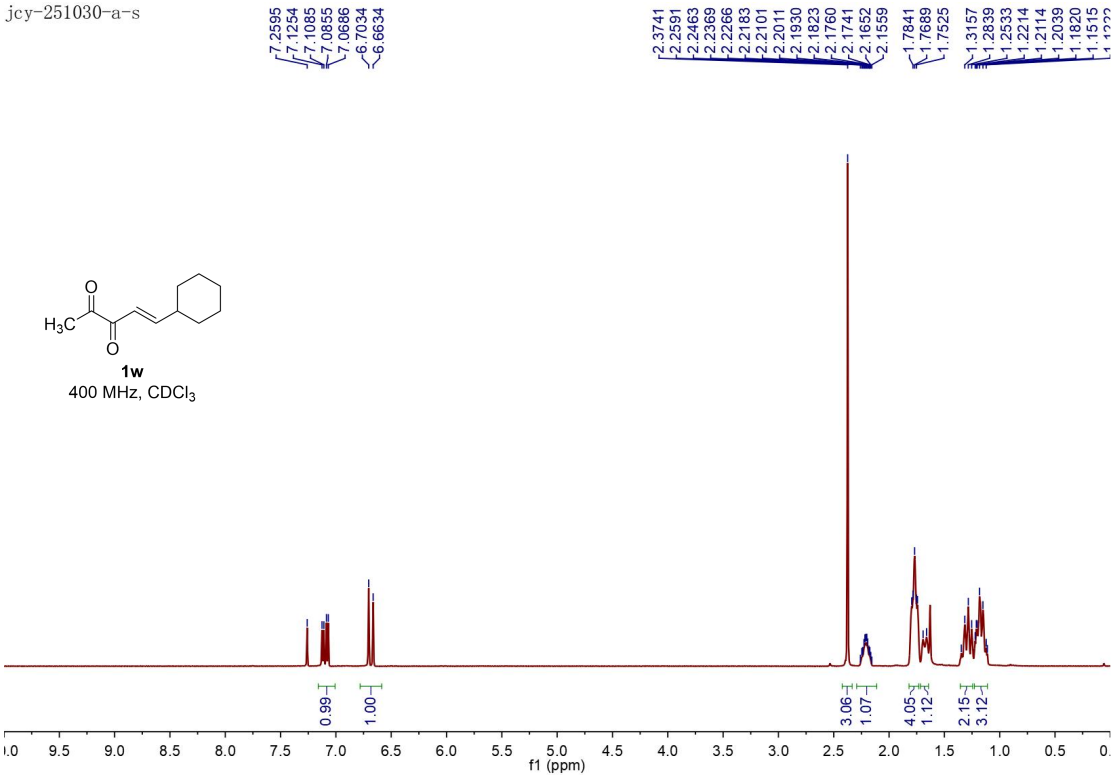
jcy-260115-2ch3



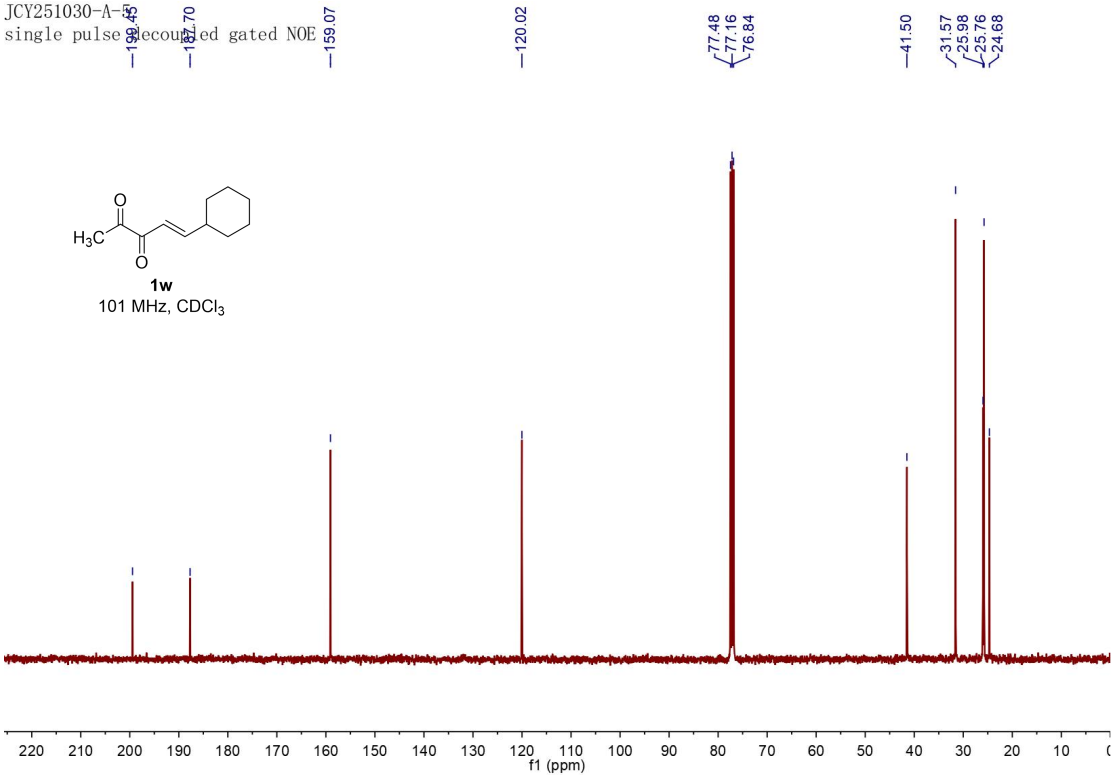
JCY-250926-BS
single pulse decoupled gated NOE

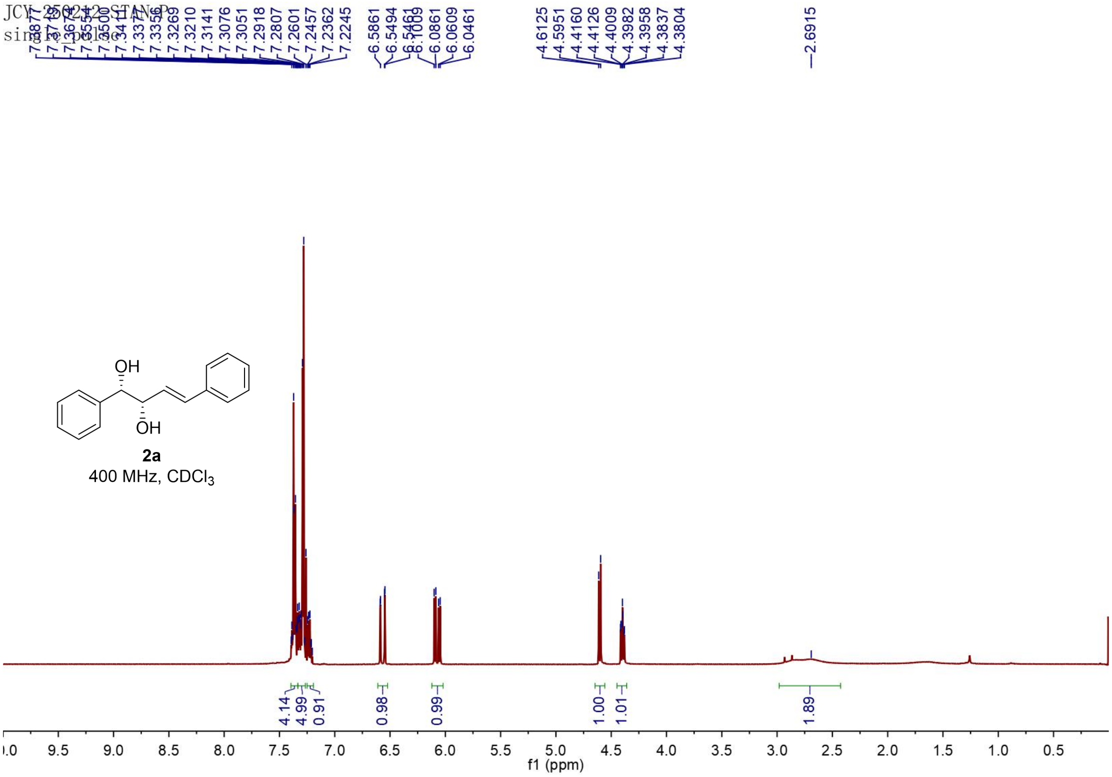


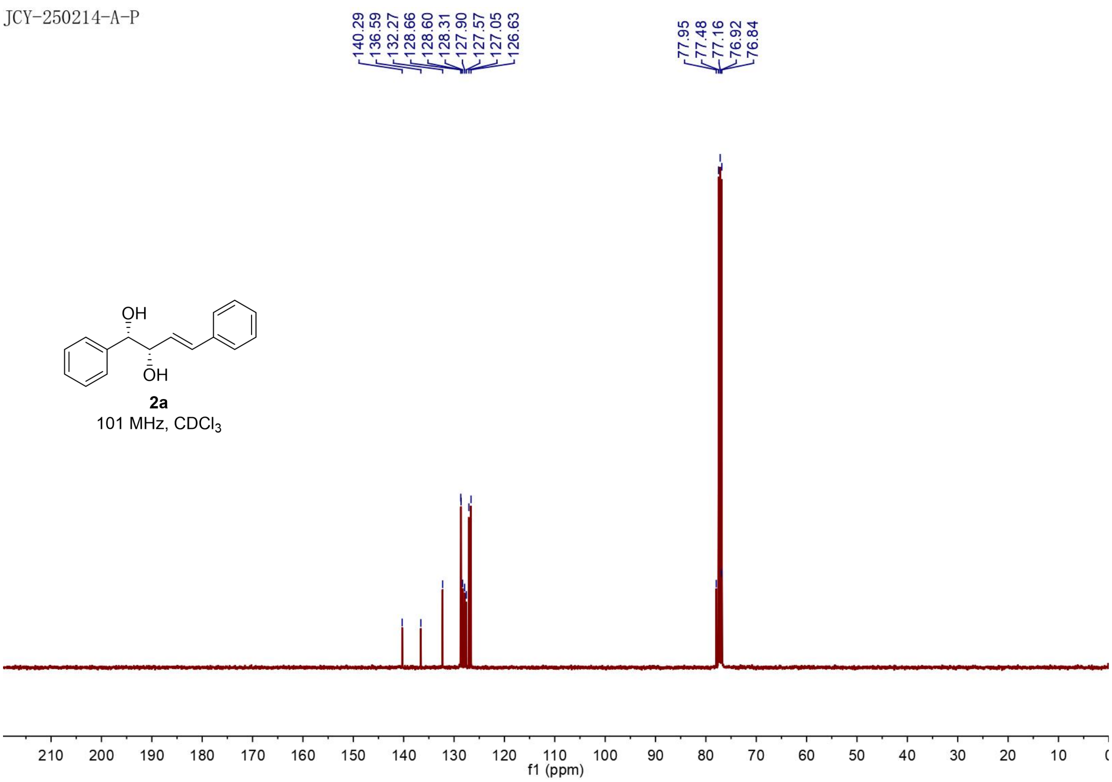
jcy-251030-a-s

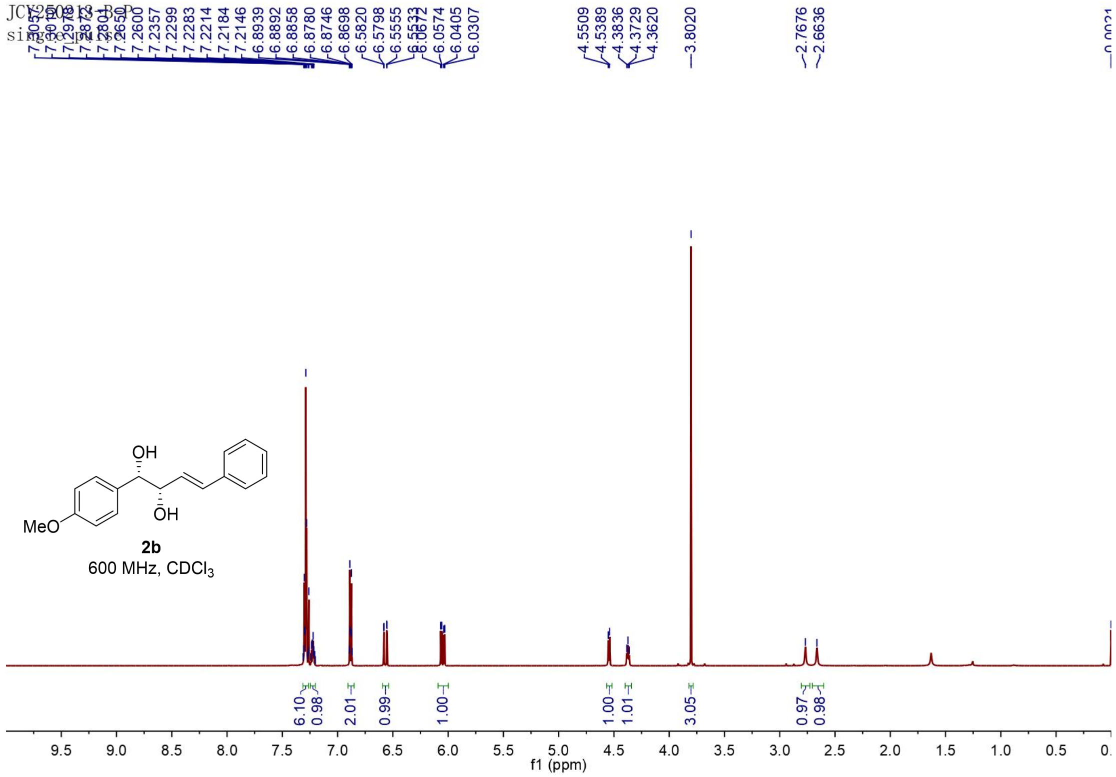


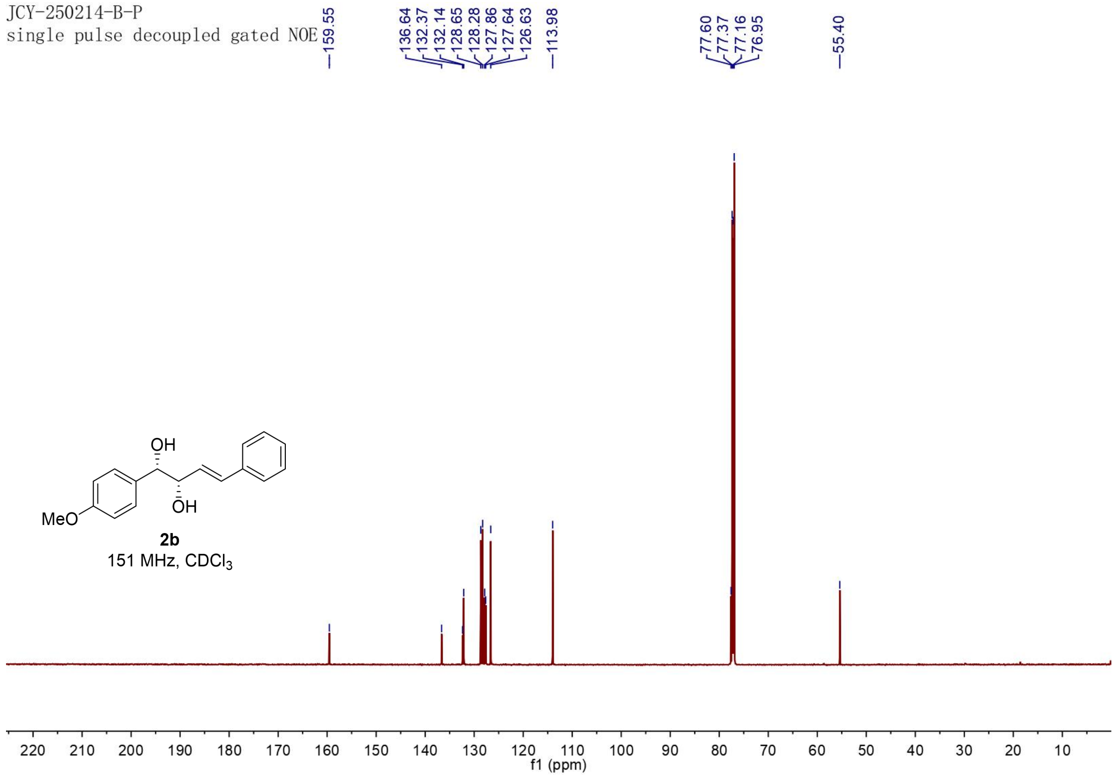
JCY251030-A-5
single pulse decoupled gated NOE



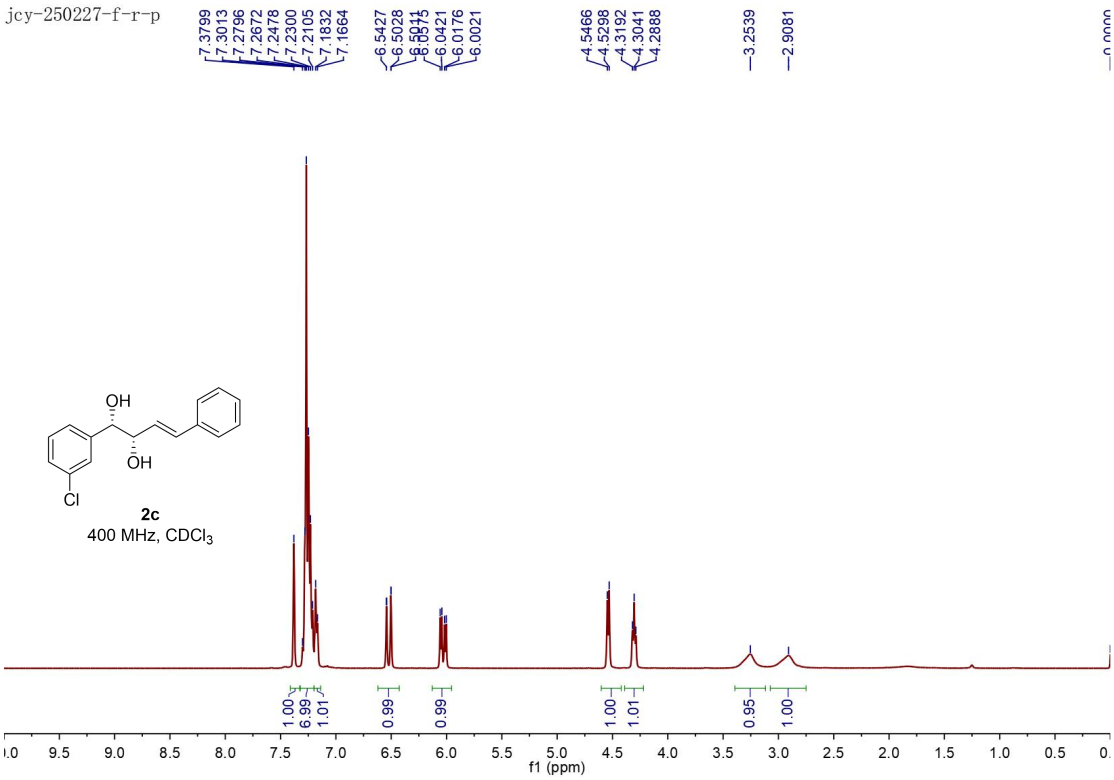

7.3877
7.3710
7.3674
7.3554
7.3509
7.3417
7.3377
7.3356
7.3269
7.3210
7.3141
7.3076
7.3051
7.2918
7.2807
7.2601
7.2457
7.2362
7.2245
6.5861
6.5494
6.1003
6.0861
6.0609
6.0461
4.6125
4.5951
4.4160
4.4126
4.4009
4.3982
4.3958
4.3837
4.3804
2.6915
OH
OH
2a
400 MHz, CDCl3
4.14
4.99
0.91
0.98
0.99
1.00
1.01
1.89
f1 (ppm)


JCY-250214-A-P
140.29
136.59
132.27
128.66
128.60
128.31
127.90
127.57
127.05
126.63
77.95
77.48
77.16
76.92
76.84
OH
OH
2a
101 MHz, CDCl3
f1 (ppm)

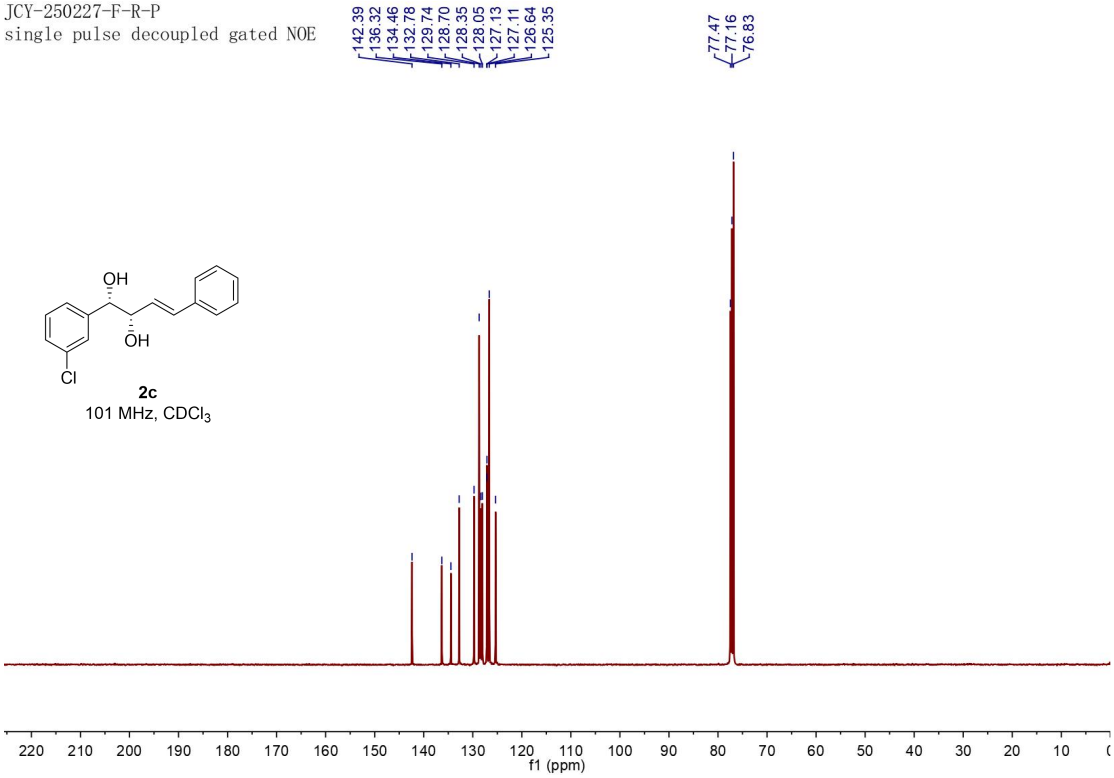

JCY-250213-B-P
single pulse
600 MHz, CDCl3
f1 (ppm)


JCY-250214-B-P
single pulse decoupled gated NOE
151 MHz, CDCl3
f1 (ppm)

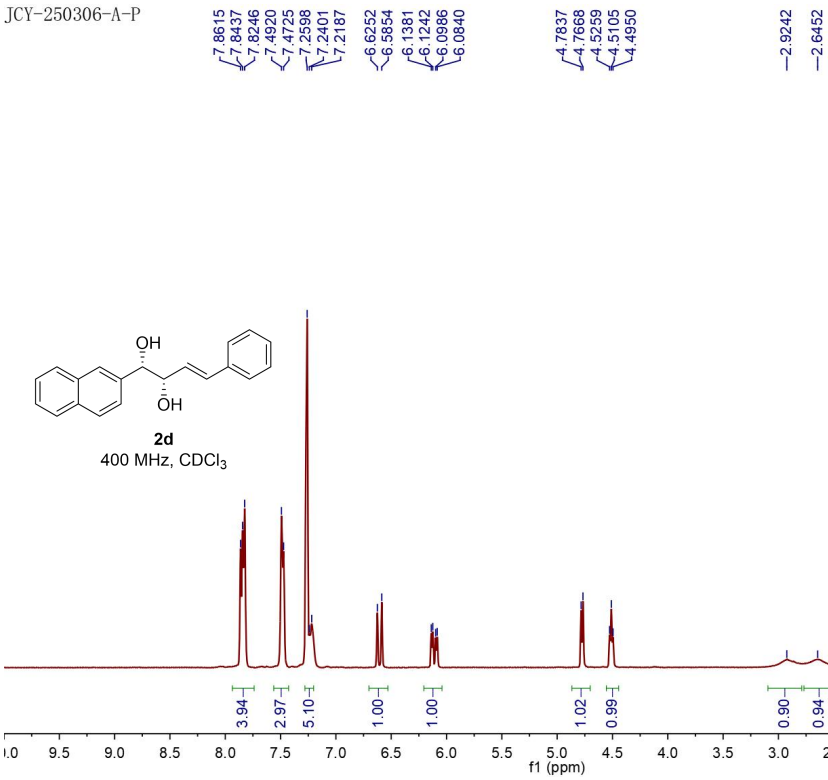
jcy-250227-f-r-p



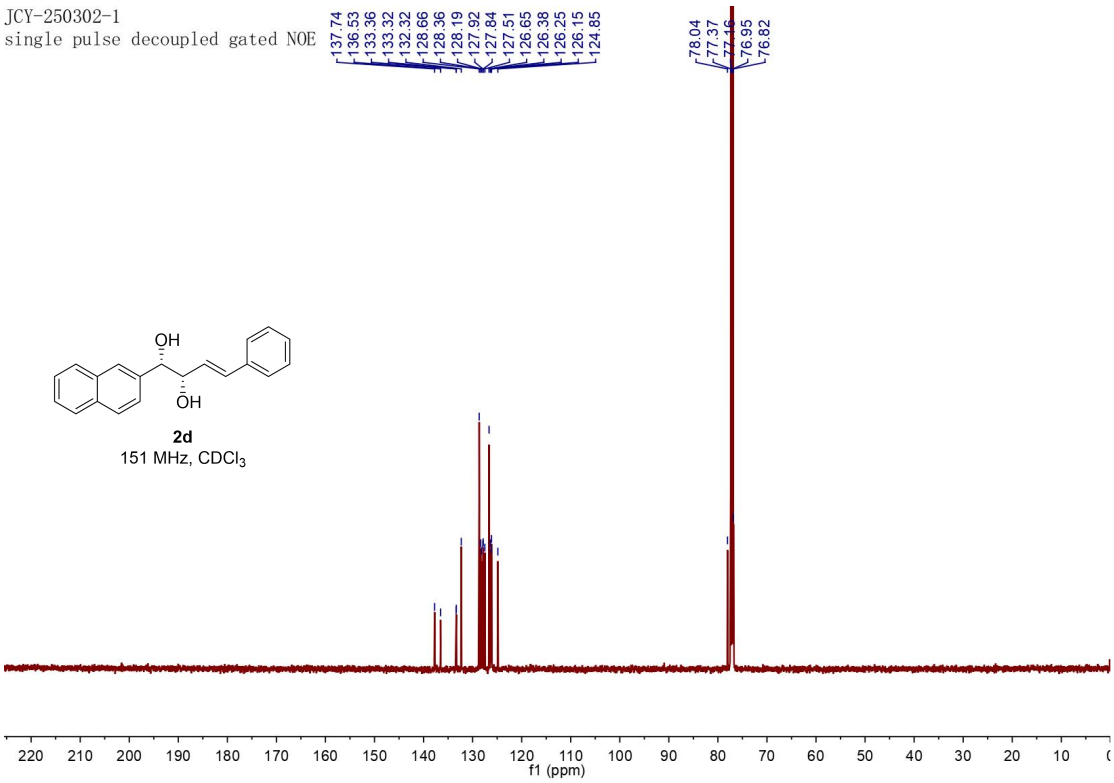
JCY-250227-F-R-P
single pulse decoupled gated NOE

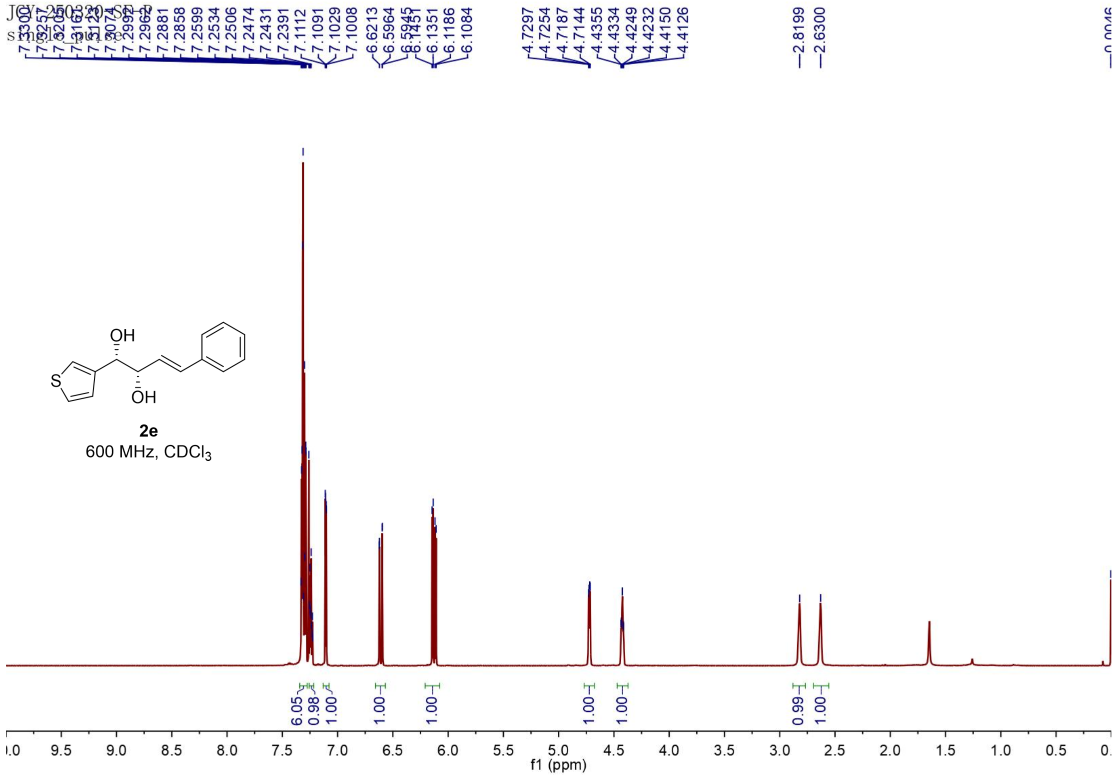


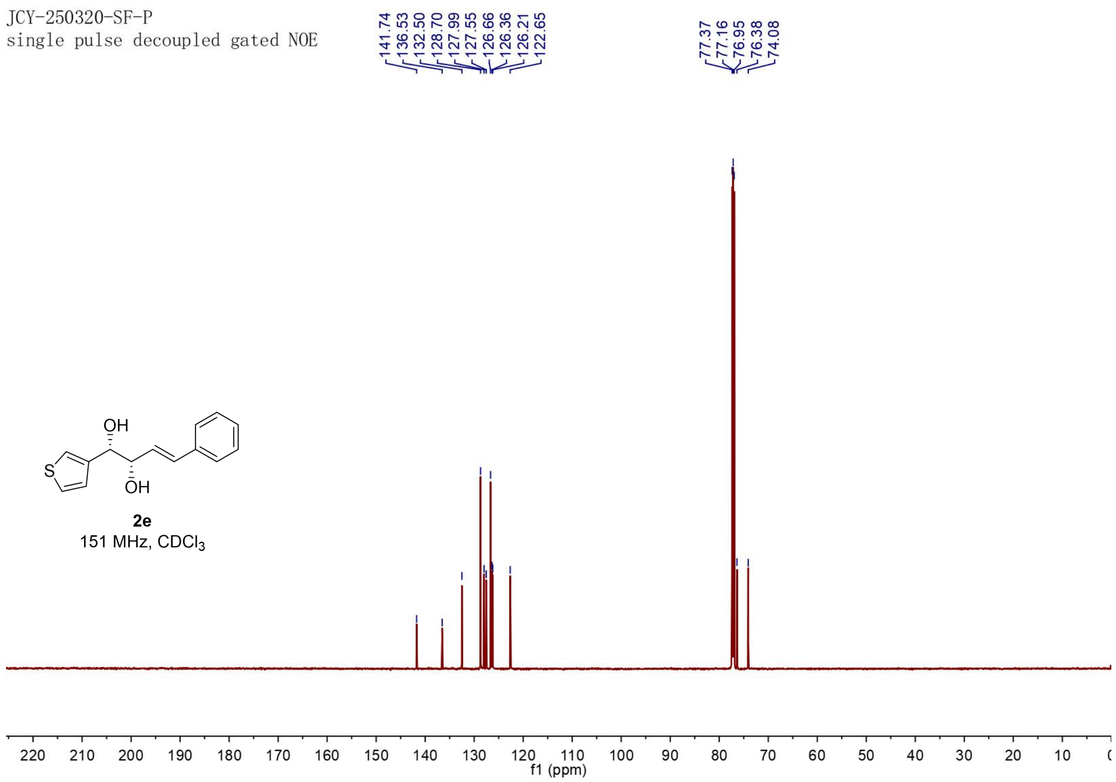
JCY-250306-A-P



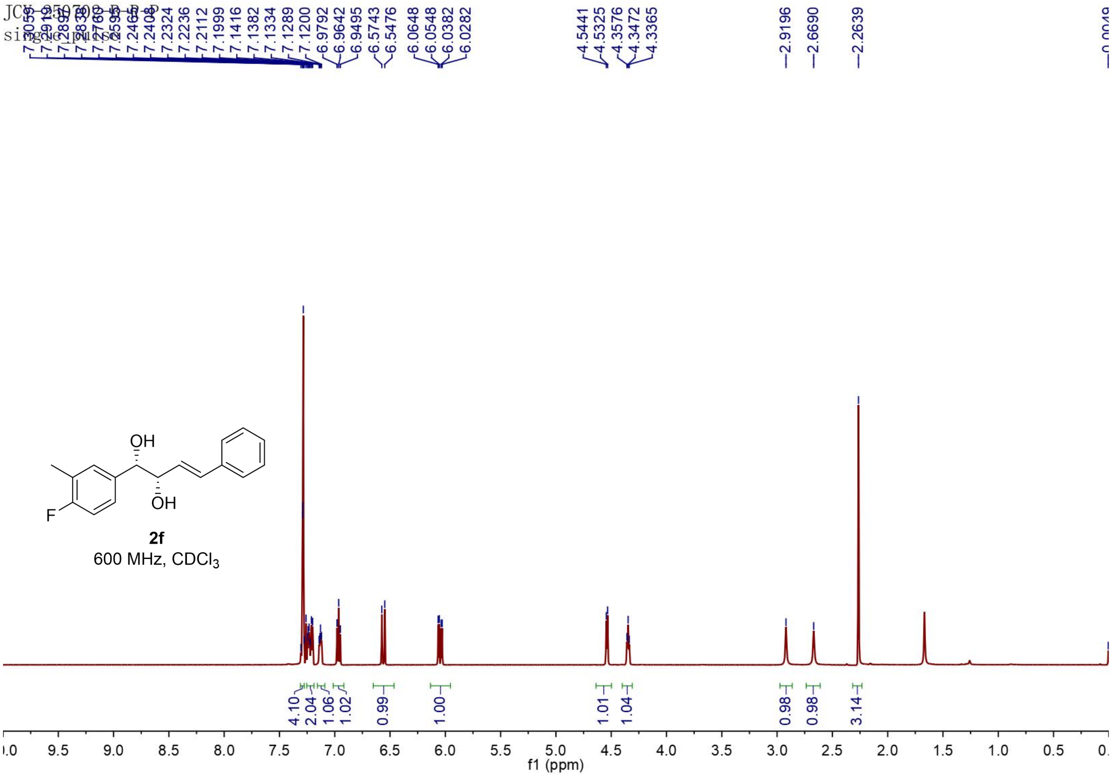
JCY-250302-1
single pulse decoupled gated NOE



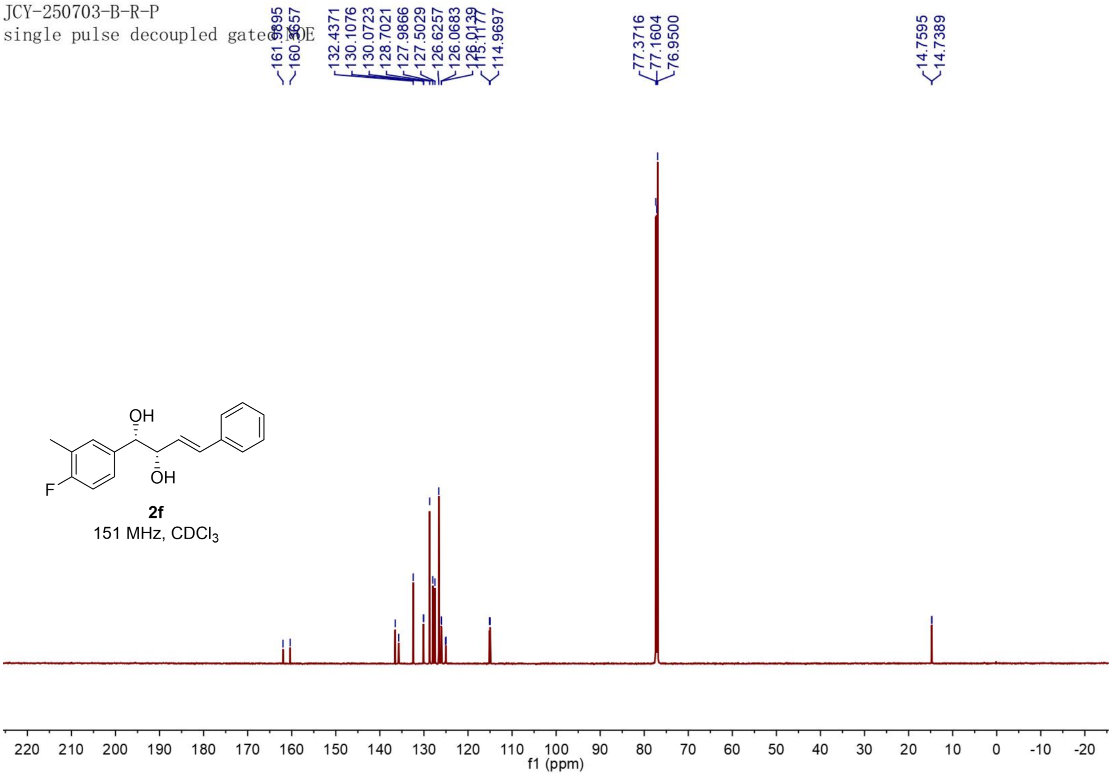

JCY-250320-SF-P
7.2881 7.2858 7.2599 7.2534 7.2506 7.2474 7.2431 7.2391 7.1112 7.1091 7.1029 7.1008 6.6213 6.5964 6.1351 6.1186 6.1084
4.7297 4.7254 4.7187 4.7144 4.4355 4.4334 4.4249 4.4232 4.4150 4.4126
2.8199 2.6300
OH
S
OH
2e
600 MHz, CDCl3
6.05 0.98 1.00 1.00 1.00 1.00 1.00 0.99 1.00
f1 (ppm)


JCY-250320-SF-P
single pulse decoupled gated NOE
141.74 136.53 132.50 128.70 127.99 127.55 126.66 126.36 126.21 122.65
77.37 77.16 76.95 76.38 74.08
OH
S
OH
2e
151 MHz, CDCl3
f1 (ppm)

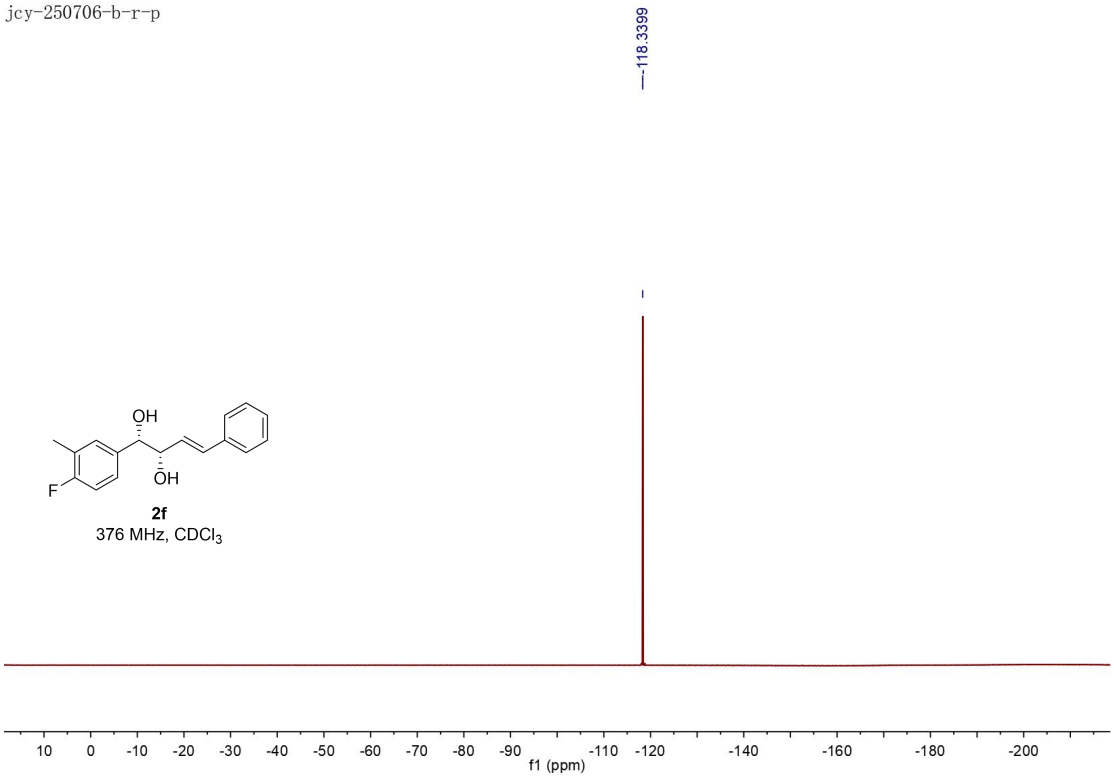
JCY-250703-B-R-P
single pulse

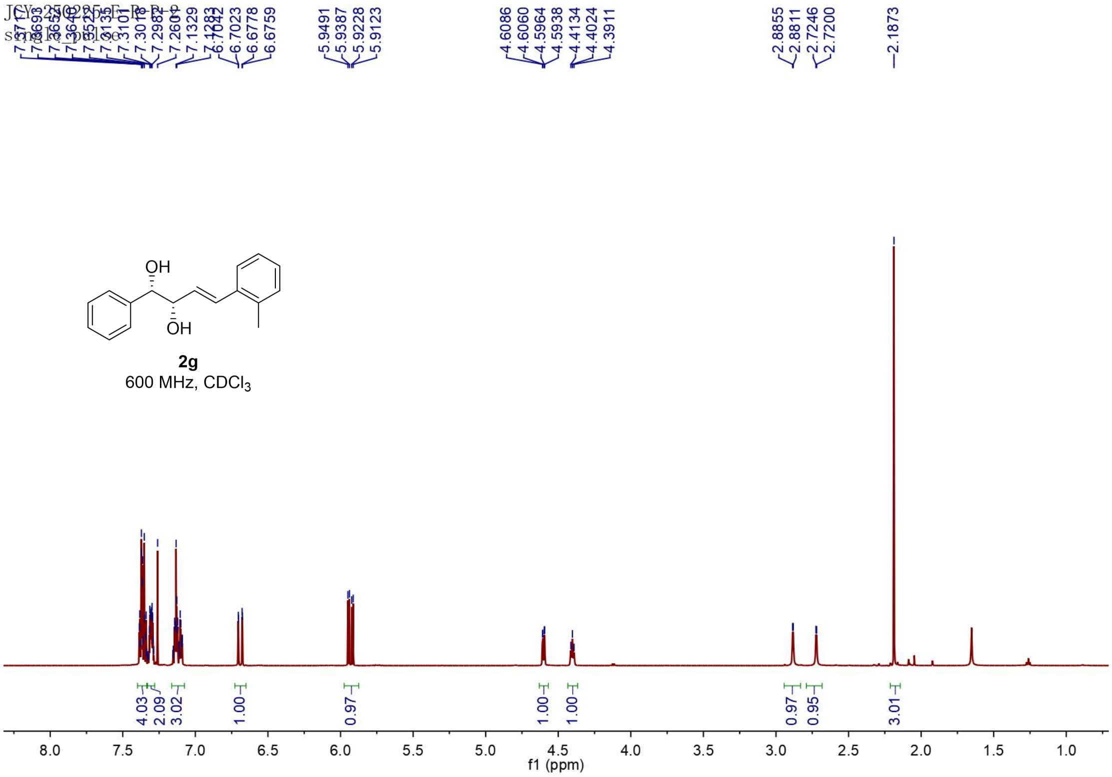


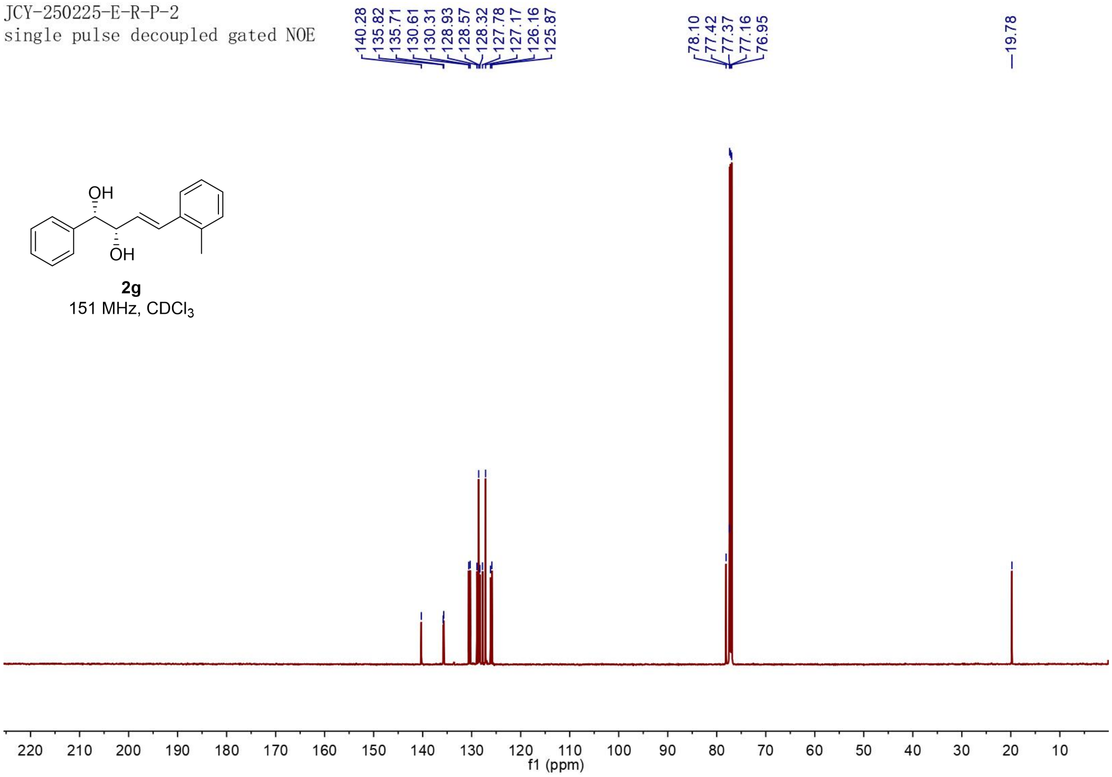
JCY-250703-B-R-P
single pulse decoupled gated NOE

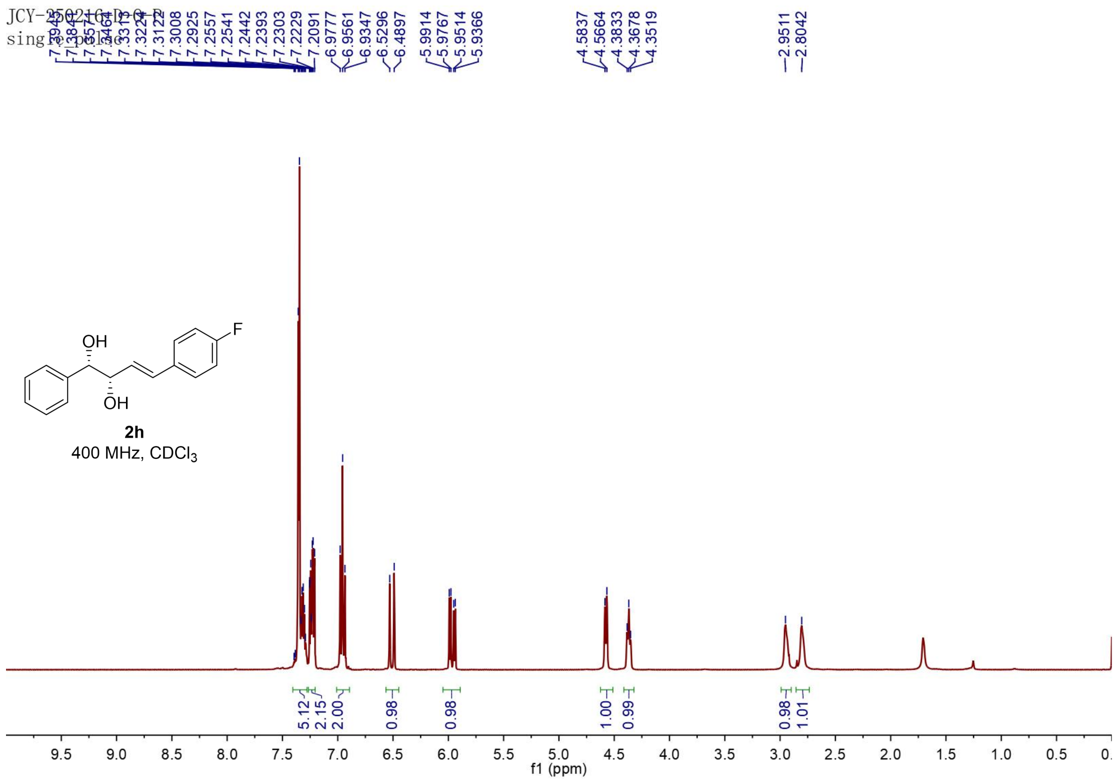


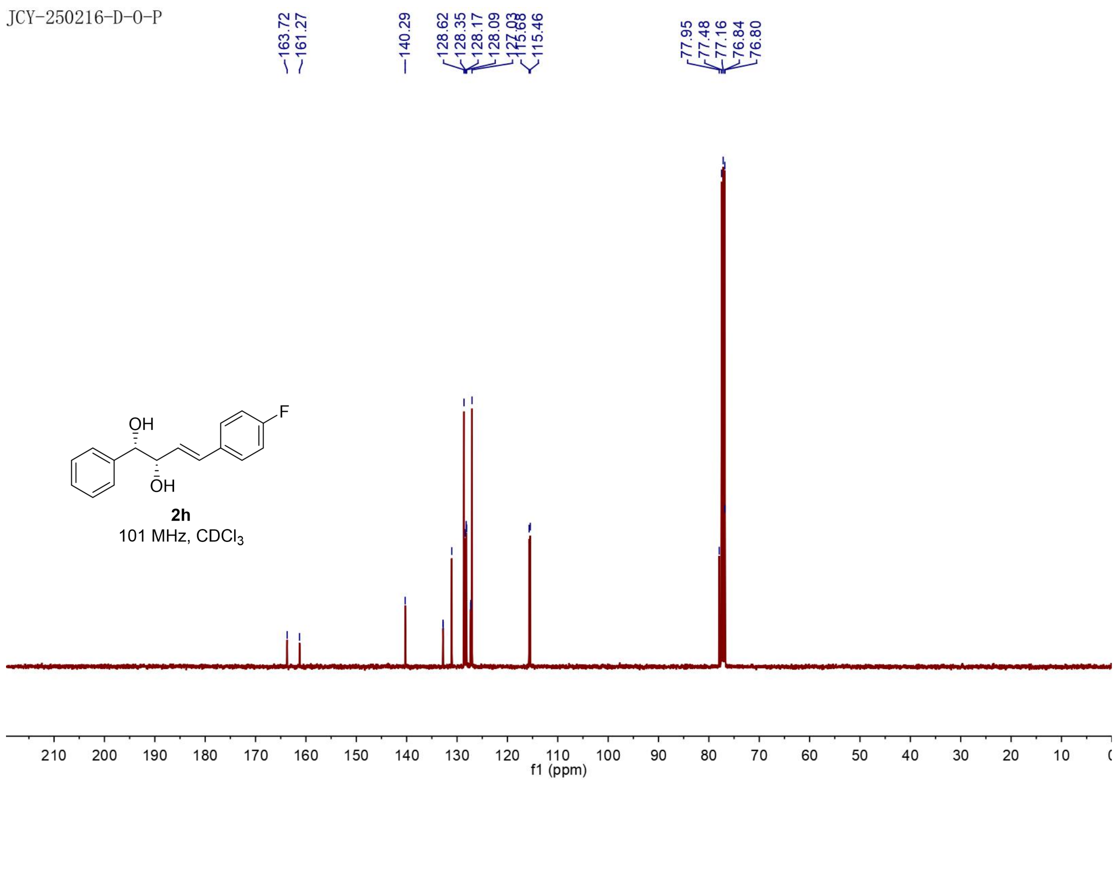
jcy-250706-b-r-p



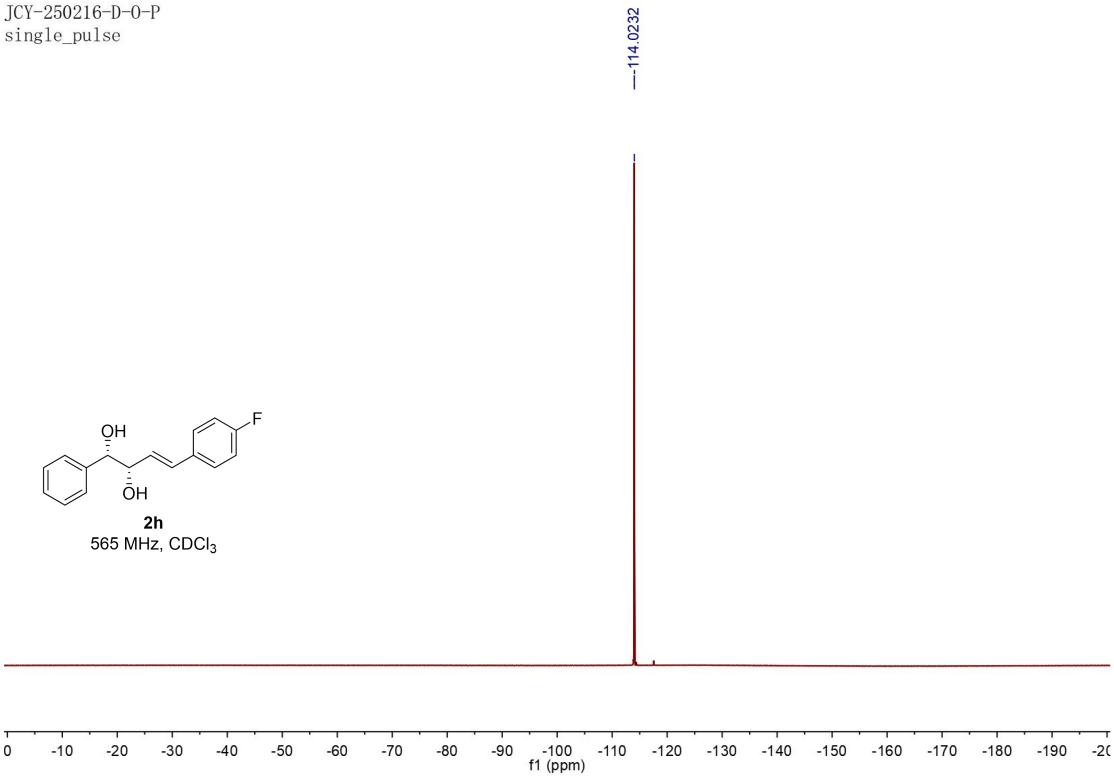

7.2601
7.1329
6.7023
6.6778
6.6759
5.9491
5.9387
5.9228
5.9123
4.6086
4.6060
4.5964
4.5938
4.4134
4.4024
4.3911
2.8855
2.8811
2.7246
2.7200
2.1873
OH
OH
2g
600 MHz, CDCl3
4.03
2.09
3.02
1.00
0.97
1.00
1.00
0.97
0.95
3.01
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)

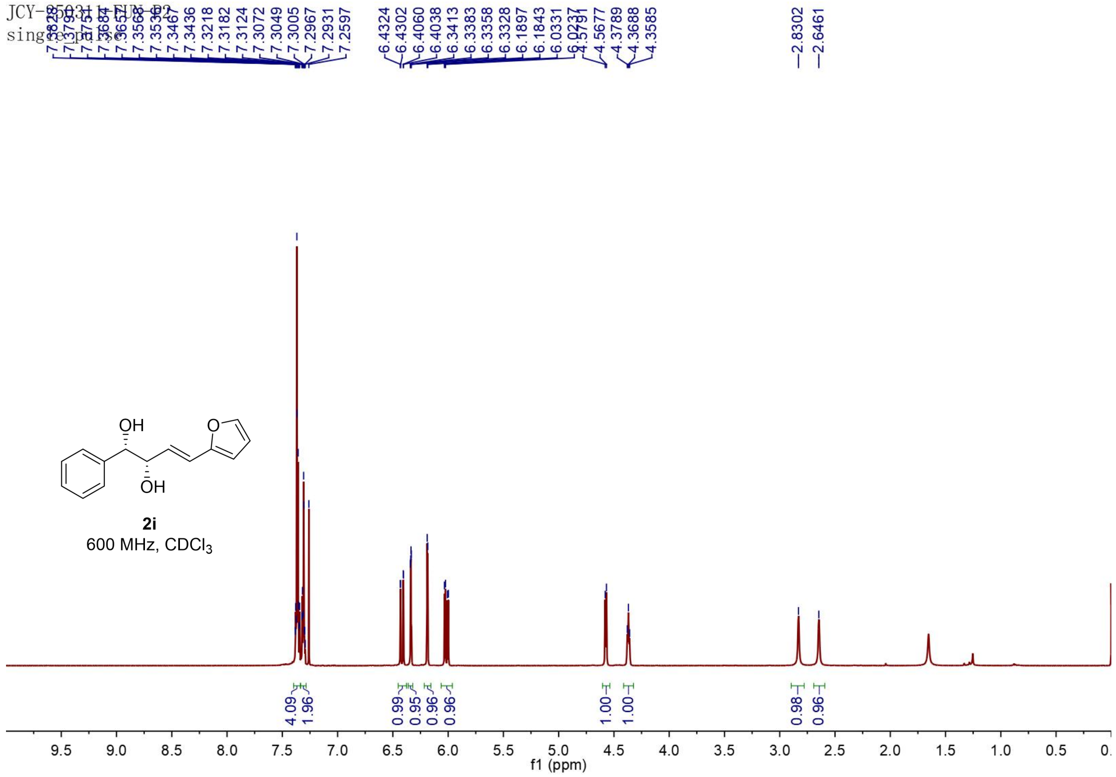

JCY-250225-E-R-P-2
single pulse decoupled gated NOE
140.28
135.82
135.71
130.61
130.31
128.93
128.57
128.32
127.78
127.17
126.16
125.87
78.10
77.42
77.37
77.16
76.95
19.78
OH
OH
2g
151 MHz, CDCl3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

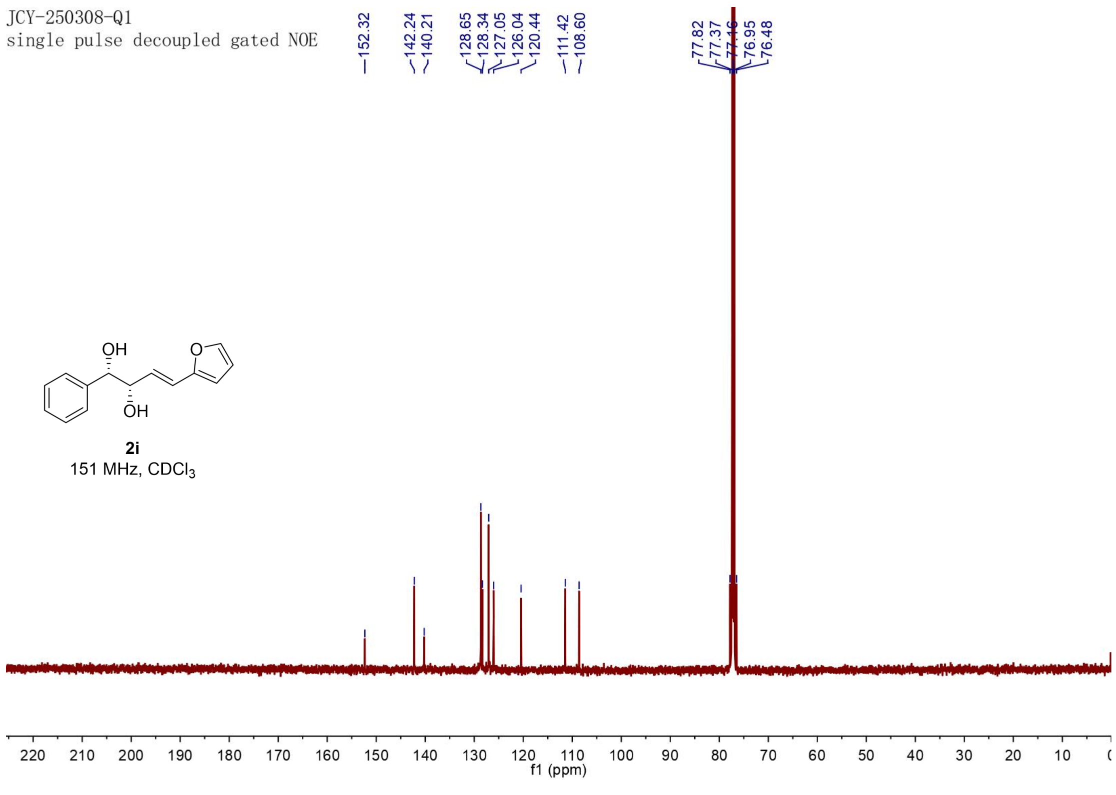

7.3224
7.3122
7.3008
7.2925
7.2557
7.2541
7.2442
7.2393
7.2303
7.2229
7.2091
6.9777
6.9561
6.9347
6.5296
6.4897
5.9914
5.9767
5.9514
5.9366
4.5837
4.5664
4.3833
4.3678
4.3519
2.9511
2.8042
OH
OH
F
2h
400 MHz, CDCl3
5.12
2.15
2.00
0.98
0.98
1.00
0.99
0.98
1.01
f1 (ppm)


JCY-250216-D-O-P
163.72
161.27
140.29
128.62
128.35
128.17
128.09
115.68
115.46
77.95
77.48
77.16
76.84
76.80
OH
OH
F
2h
101 MHz, CDCl3
f1 (ppm)

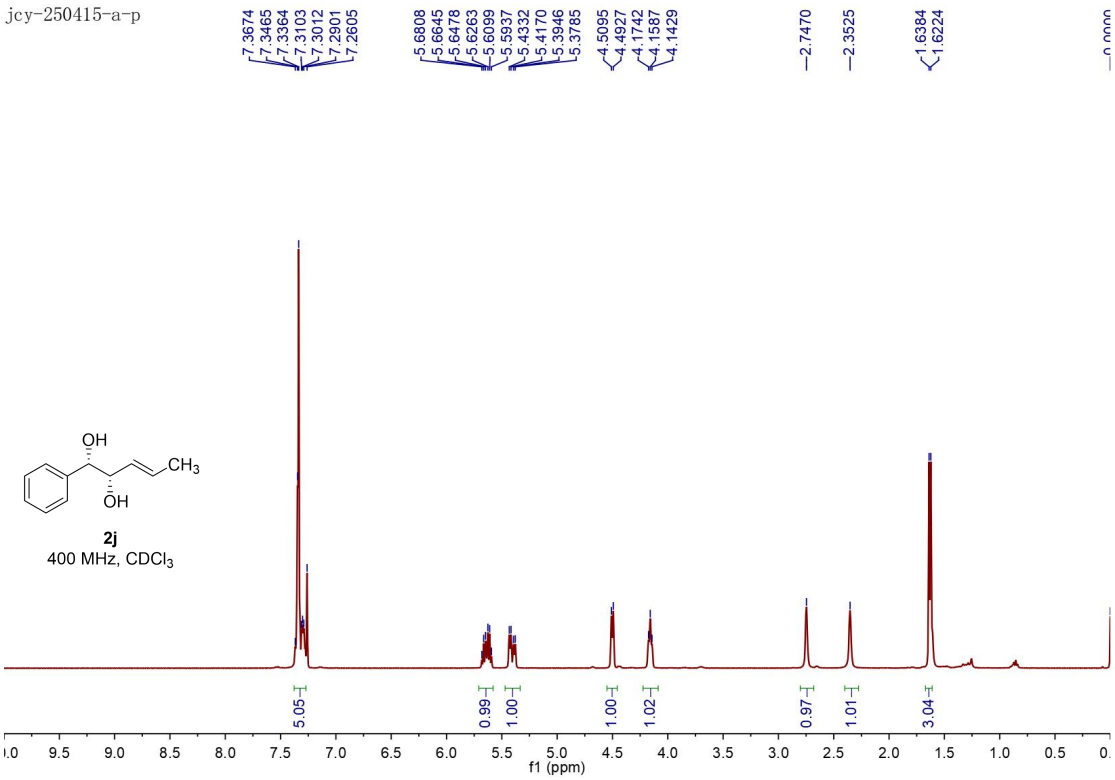
JCY-250216-D-O-P
single_pulse



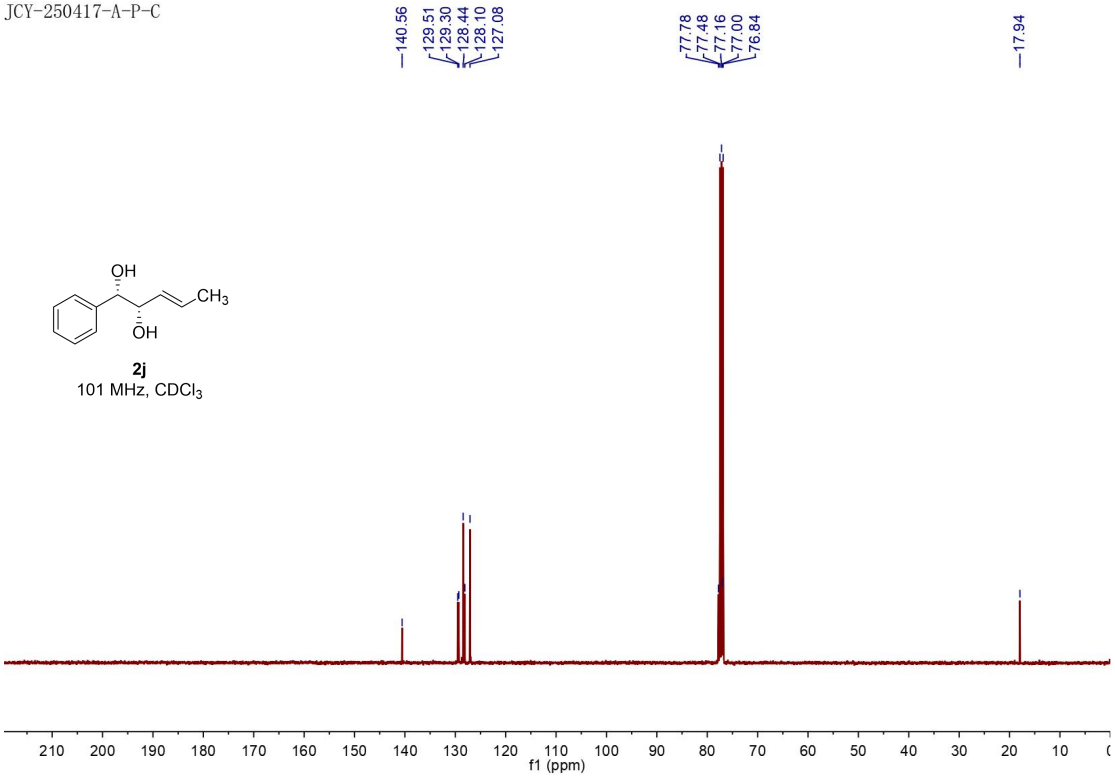

7.3823 7.3791 7.3751 7.3684 7.3652 7.3568 7.3536 7.3467 7.3436 7.3218 7.3182 7.3124 7.3072 7.3049 7.3005 7.2967 7.2931 7.2597
6.4324 6.4302 6.4060 6.4038 6.3413 6.3383 6.3358 6.3328 6.1897 6.1843 6.0331 6.0237
4.5791 4.5677 4.3789 4.3688 4.3585
2.8302 2.6461
OH
O
OH
2i
600 MHz, $CDCl_3$
4.09 1.96 0.99 0.95 0.96 0.96 1.00 1.00 0.98 0.96
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

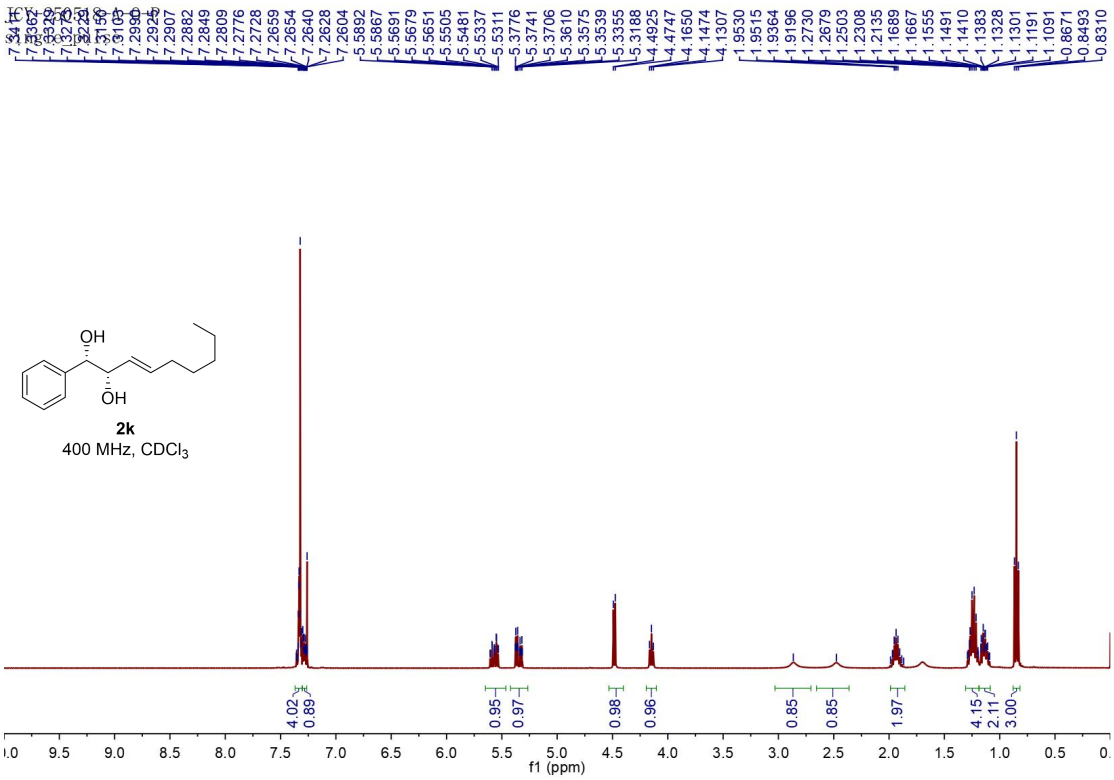

JCY-250308-Q1
single pulse decoupled gated NOE
152.32
142.24 140.21
128.65 128.34 127.05 126.04 120.44
111.42 108.60
77.82 77.37 77.16 76.95 76.48
OH
O
OH
2i
151 MHz, $CDCl_3$
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

jcy-250415-a-p

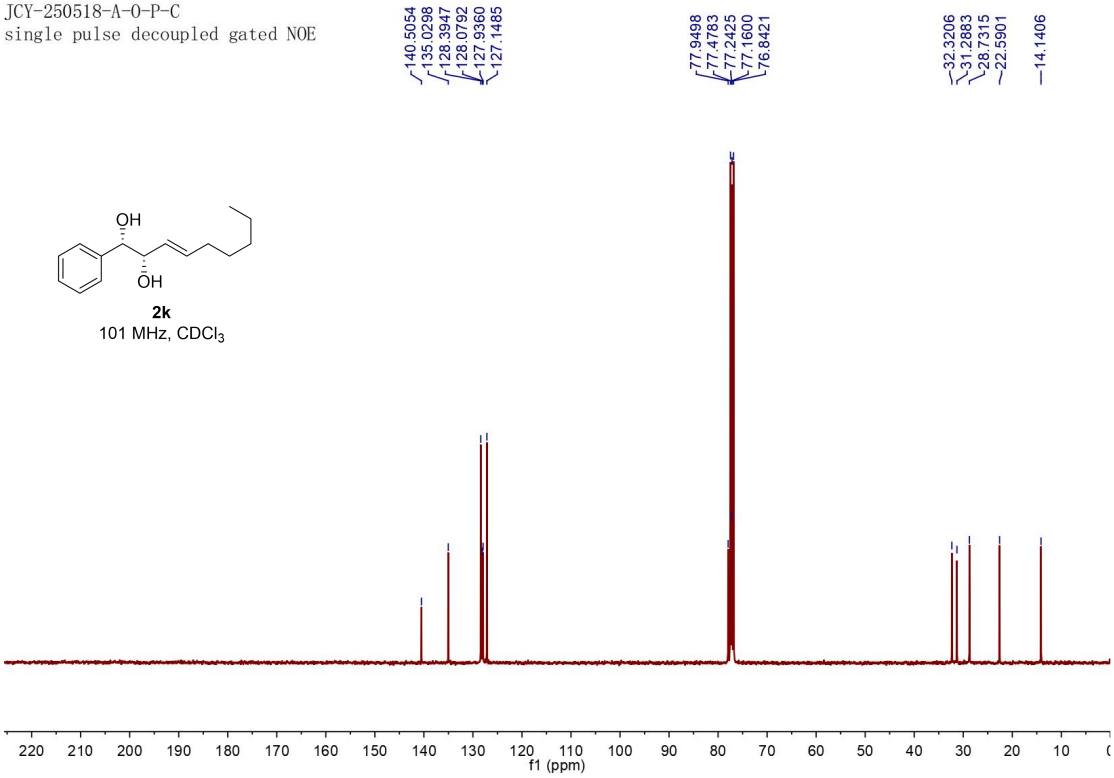


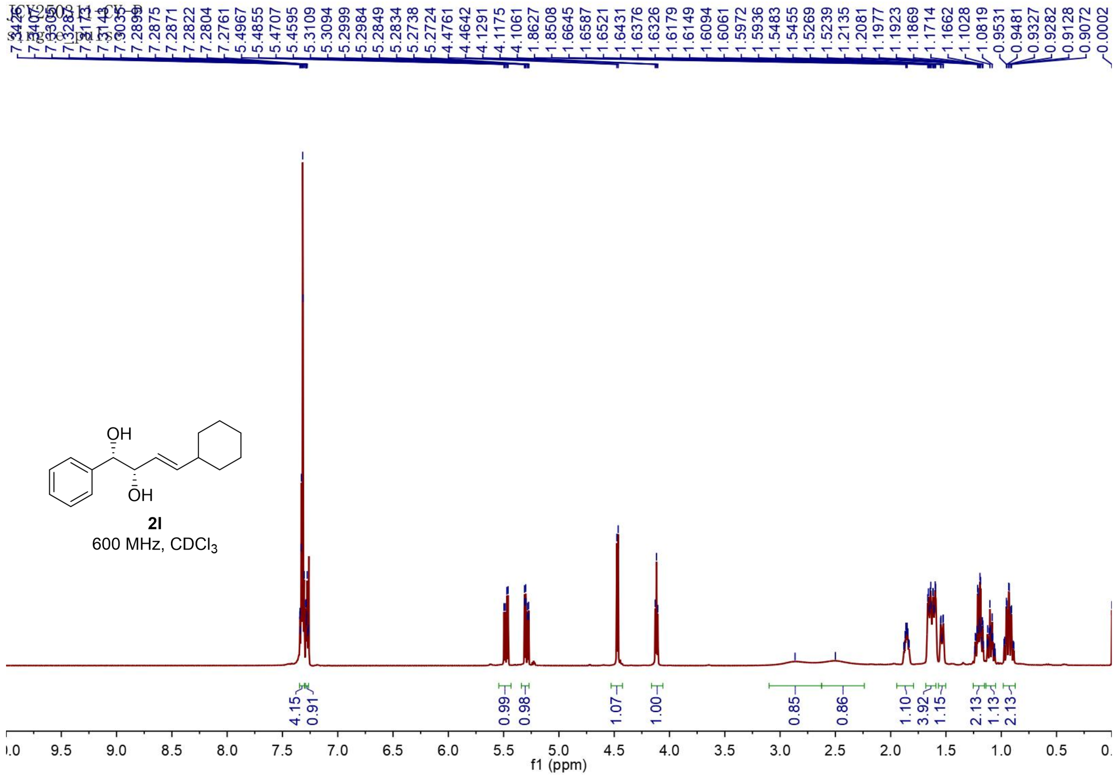
JCY-250417-A-P-C

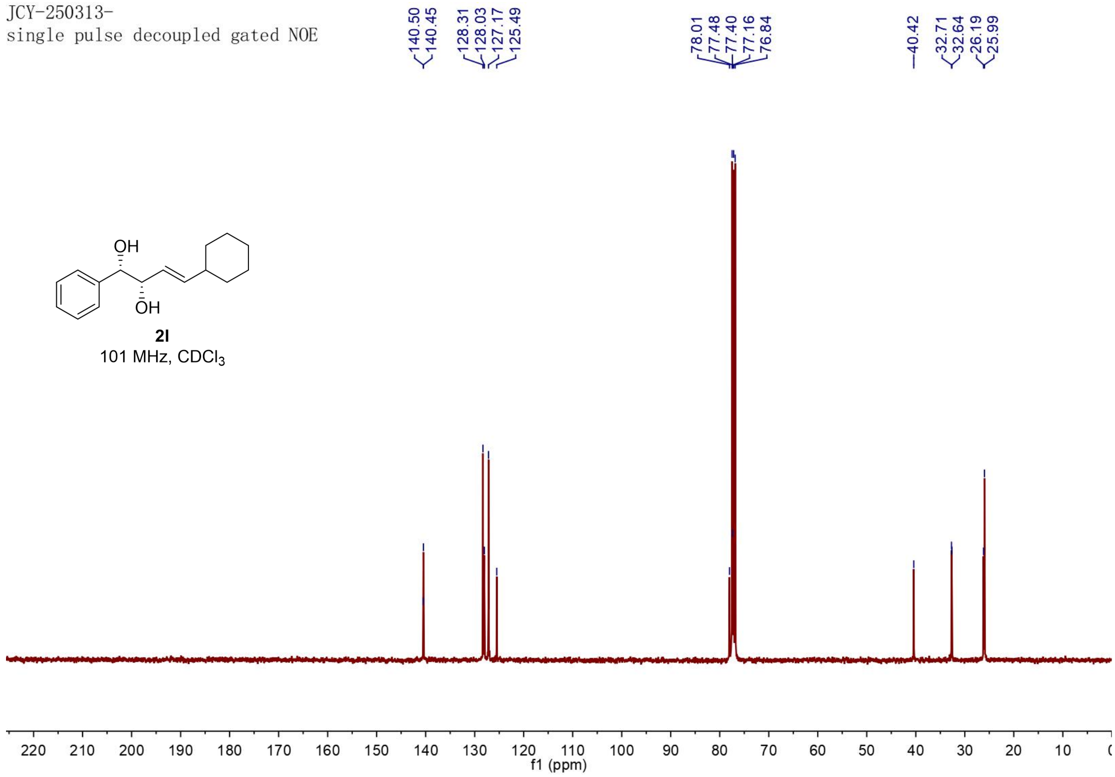




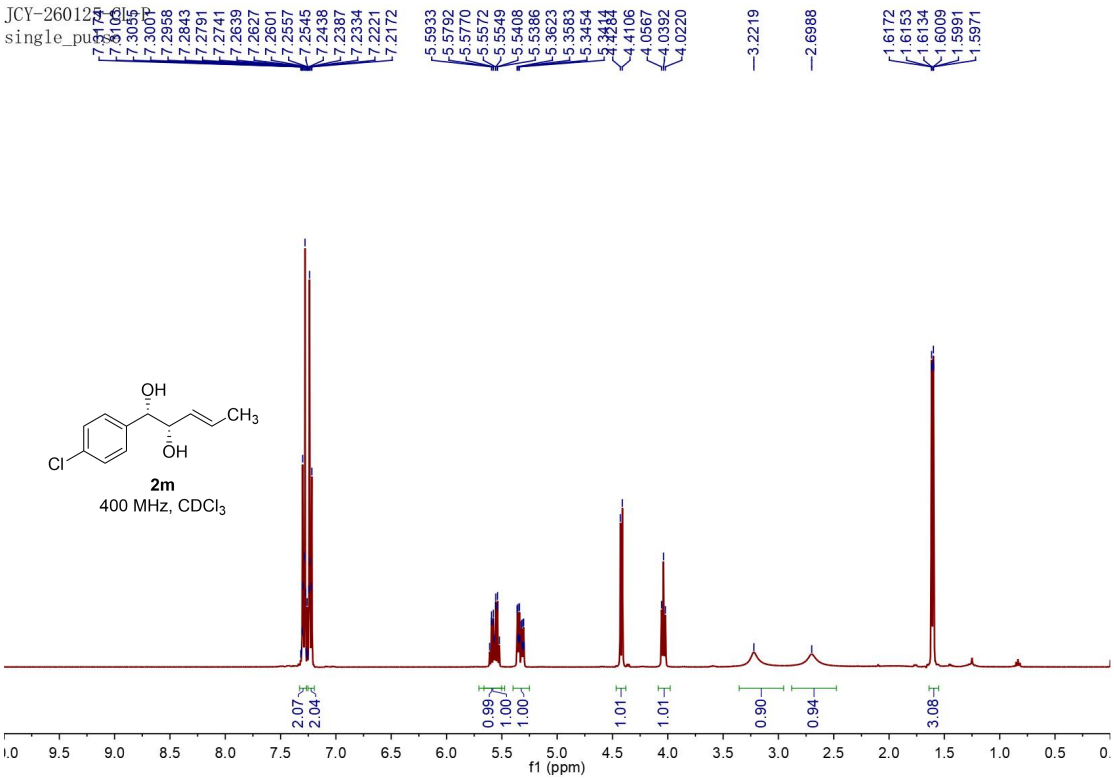
JCY-250518-A-O-P-C
single pulse decoupled gated NOE



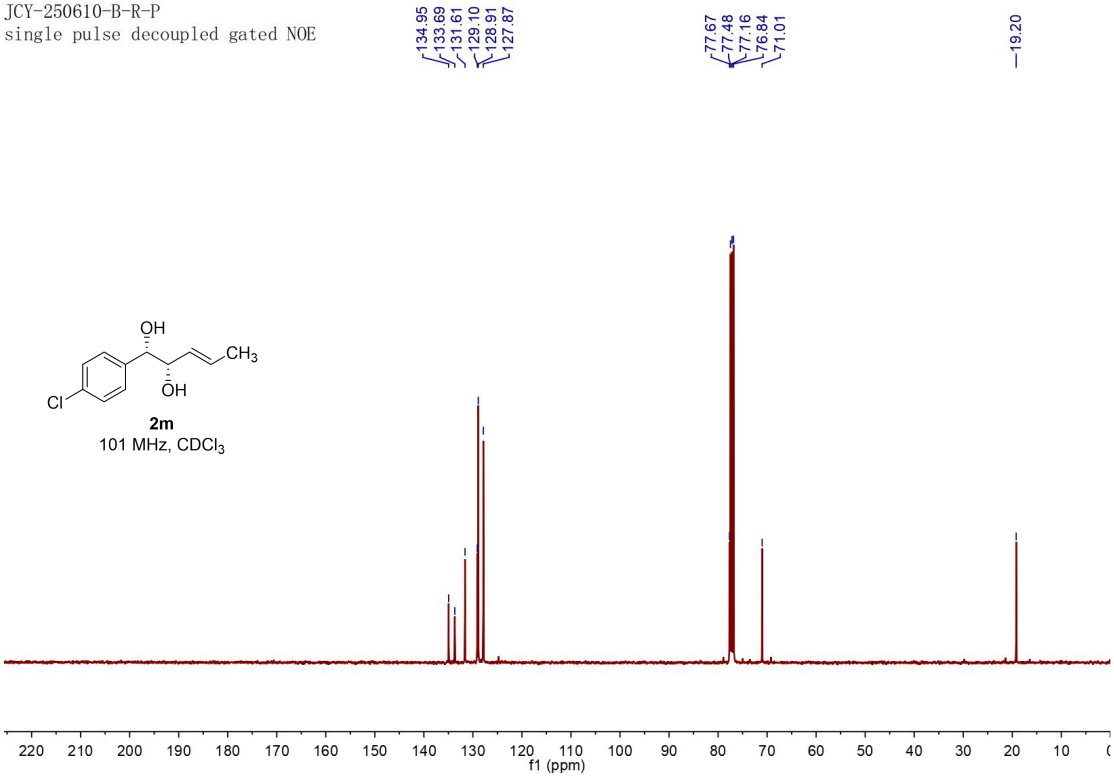

7.2871
7.2822
7.2804
7.2761
5.4967
5.4855
5.4707
5.4595
5.3109
5.3094
5.2999
5.2984
5.2849
5.2834
5.2738
5.2724
4.4761
4.4642
4.1291
4.1175
4.1061
1.8627
1.8508
1.6645
1.6587
1.6521
1.6431
1.6376
1.6326
1.6179
1.6149
1.6094
1.6061
1.5972
1.5936
1.5483
1.5455
1.5269
1.5239
1.2135
1.2081
1.1977
1.1923
1.1869
1.1714
1.1662
1.1028
1.0819
0.9531
0.9481
0.9327
0.9282
0.9128
0.9072
0.0002
OH
OH
2l
600 MHz, CDCl3
4.15
0.91
0.99
0.98
1.07
1.00
0.85
0.86
1.10
3.92
1.15
2.13
1.13
2.13
f1 (ppm)


JCY-250313-
single pulse decoupled gated NOE
140.50
140.45
128.31
128.03
127.17
125.49
78.01
77.48
77.40
77.16
76.84
40.42
32.71
32.64
26.19
25.99
OH
OH
2l
101 MHz, CDCl3
f1 (ppm)

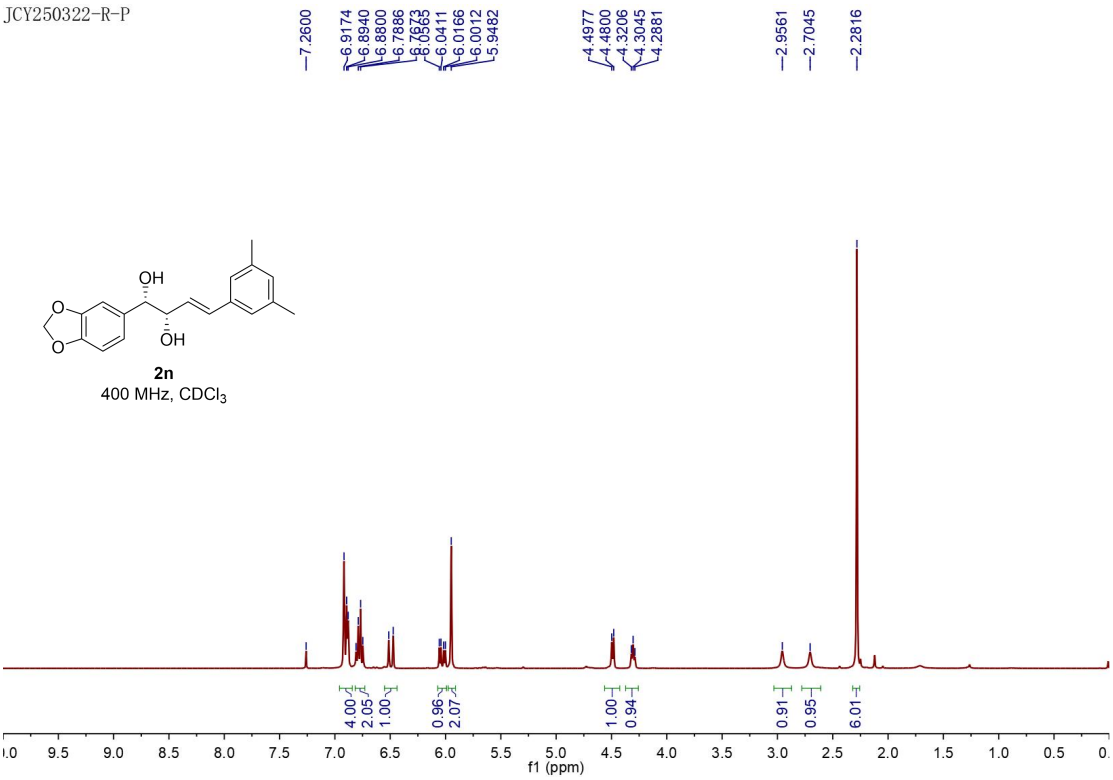
JCY-260125-C-P
single_pulse



JCY-250610-B-R-P
single pulse decoupled gated NOE

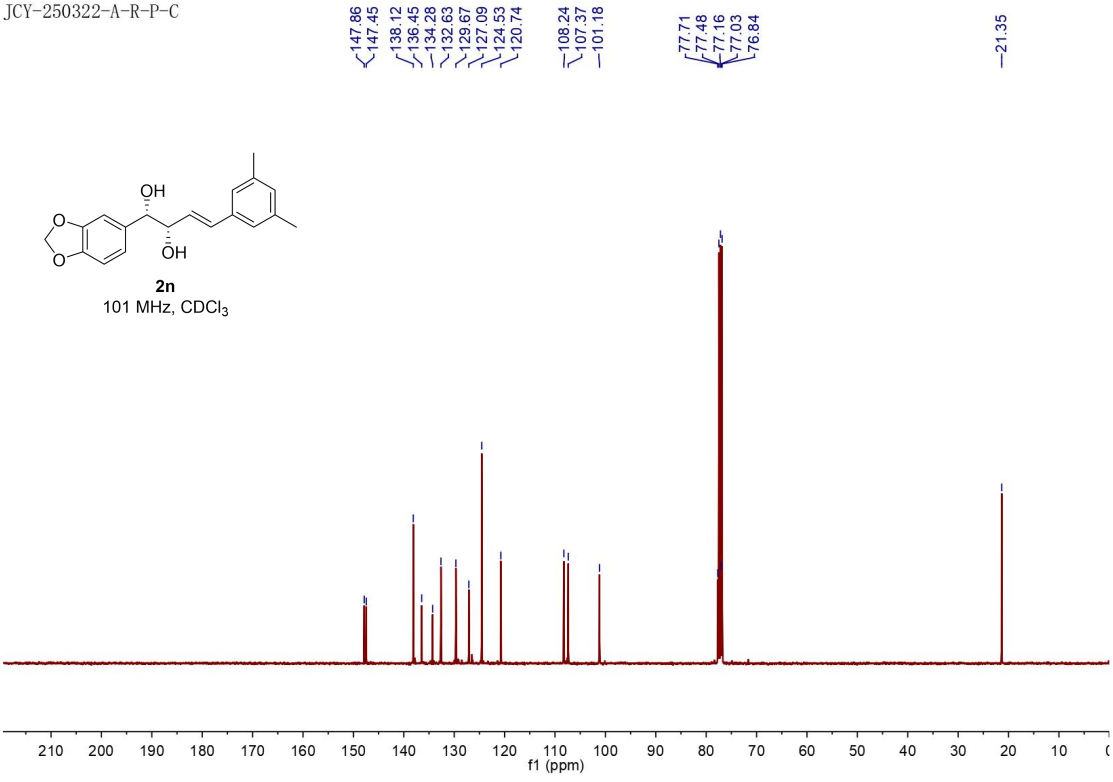


JCY250322-R-P



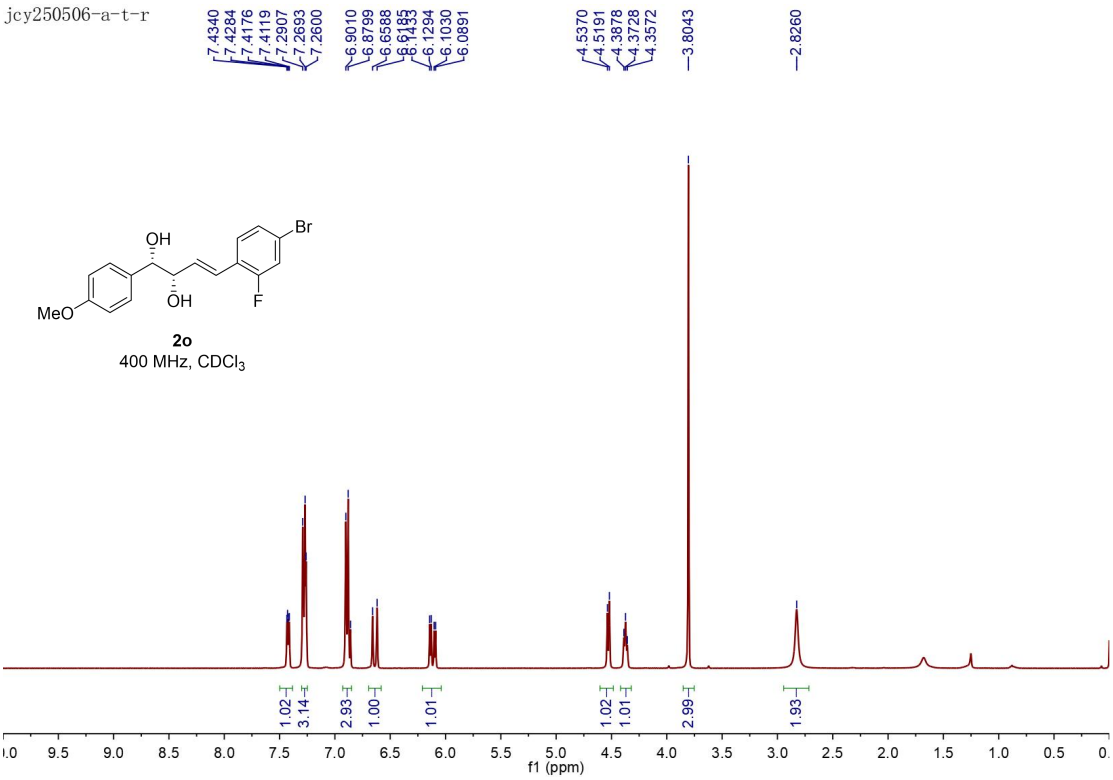
2n
400 MHz, $CDCl_3$

JCY-250322-A-R-P-C



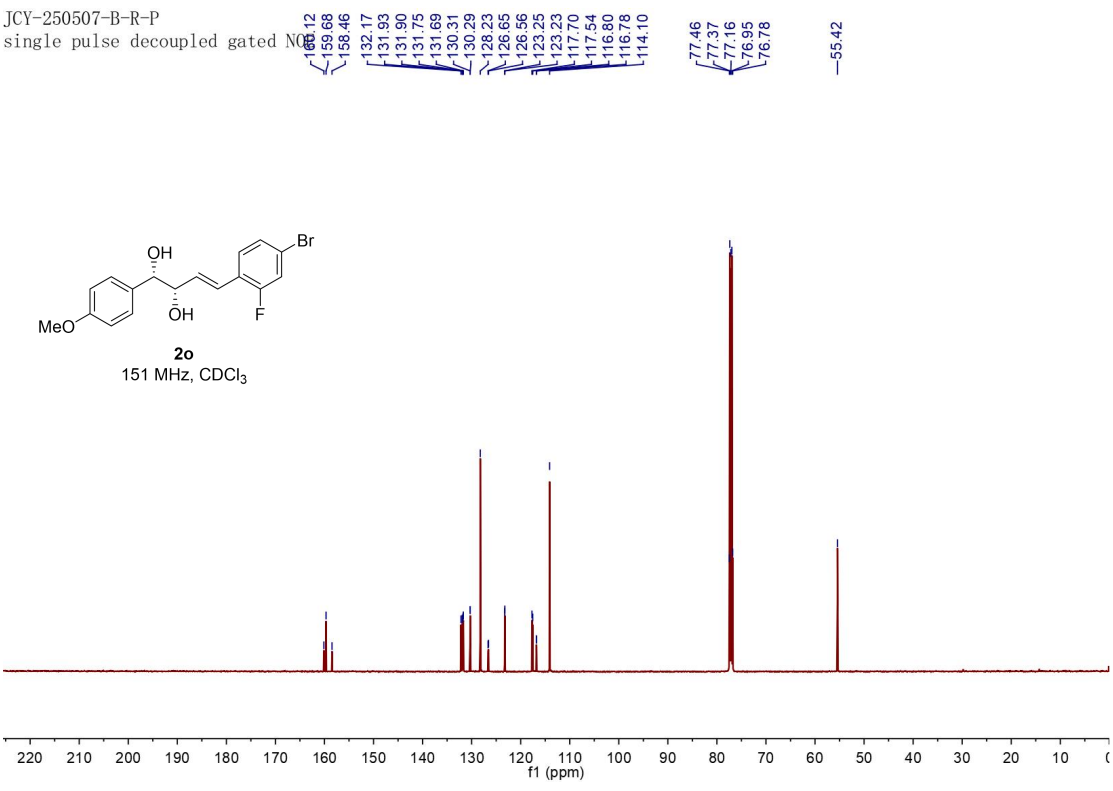
2n
101 MHz, $CDCl_3$

jcy250506-a-t-r



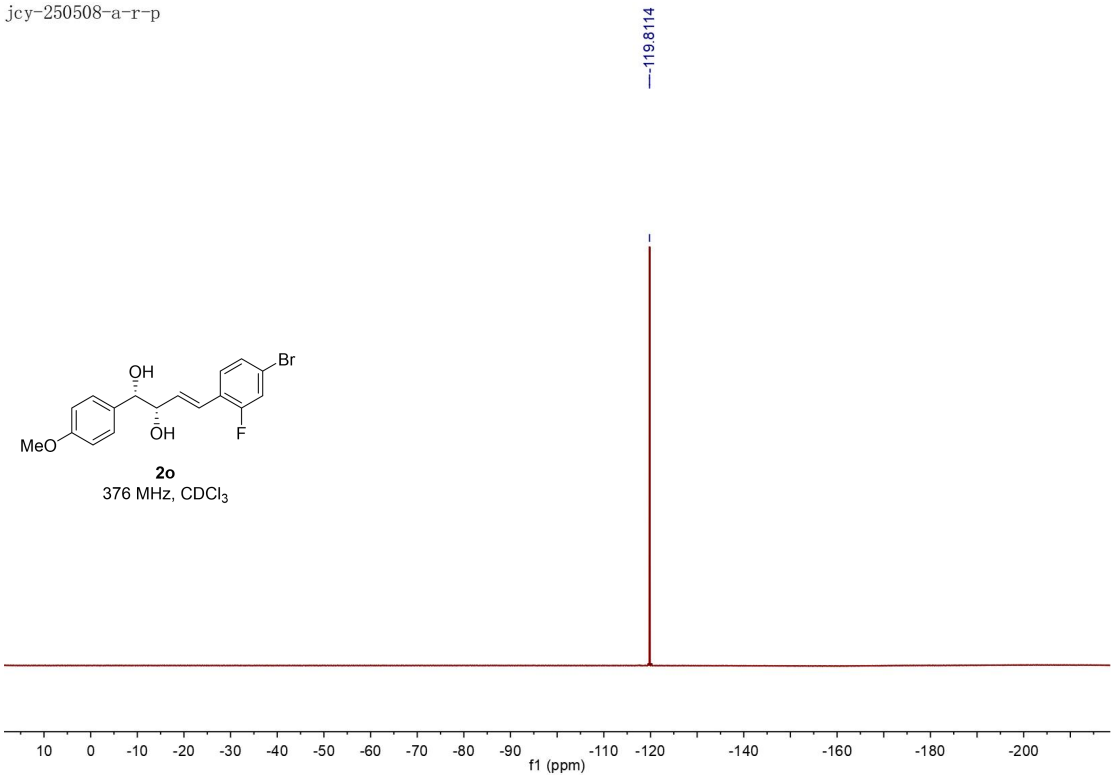
2o
400 MHz, $CDCl_3$

JCY-250507-B-R-P
single pulse decoupled gated NOE



2o
151 MHz, $CDCl_3$

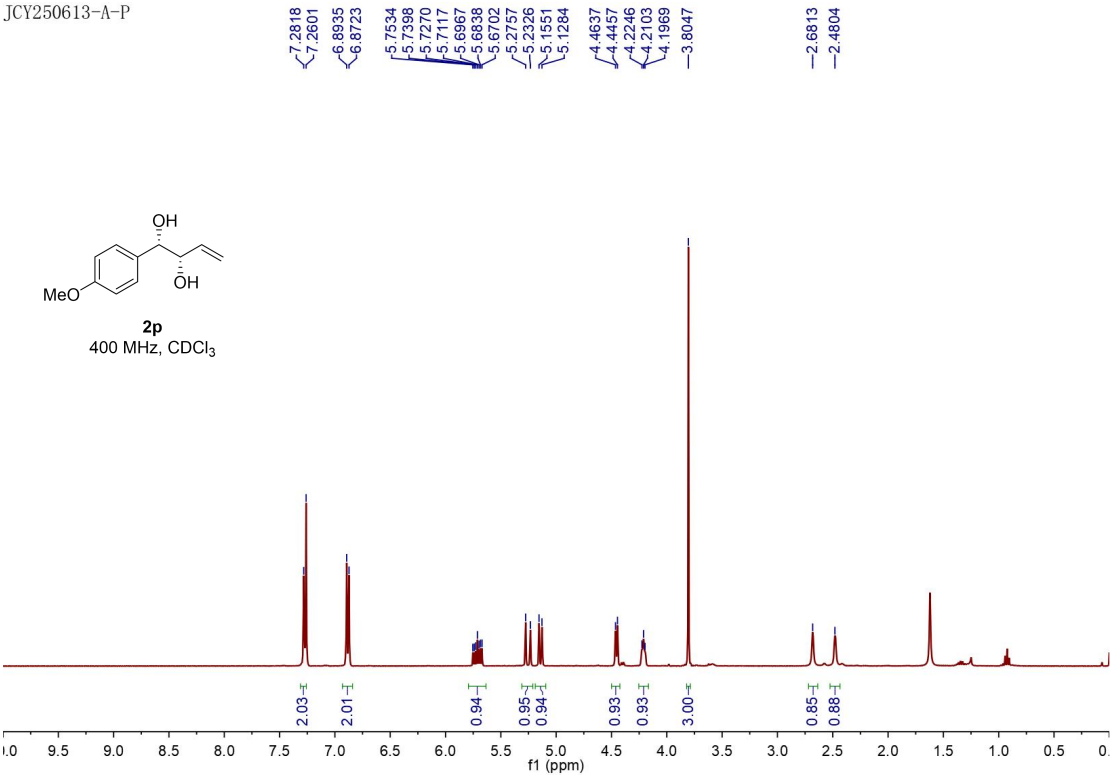
jcy-250508-a-r-p



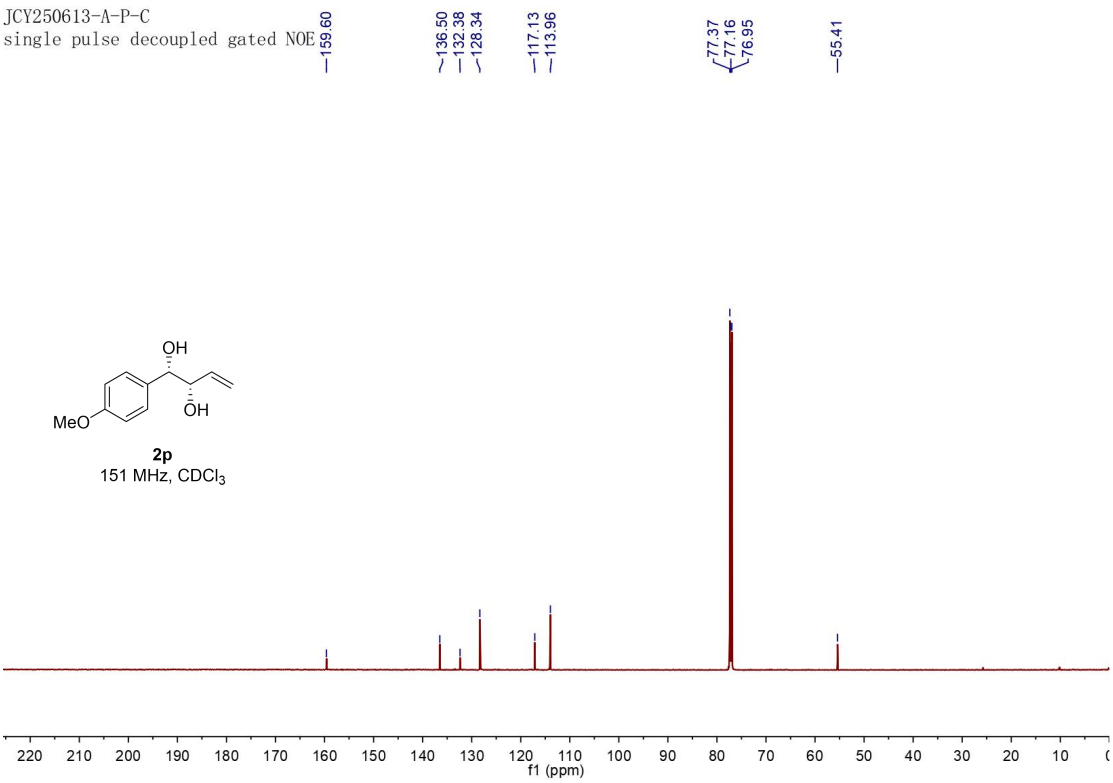
2o

376 MHz, $CDCl_3$

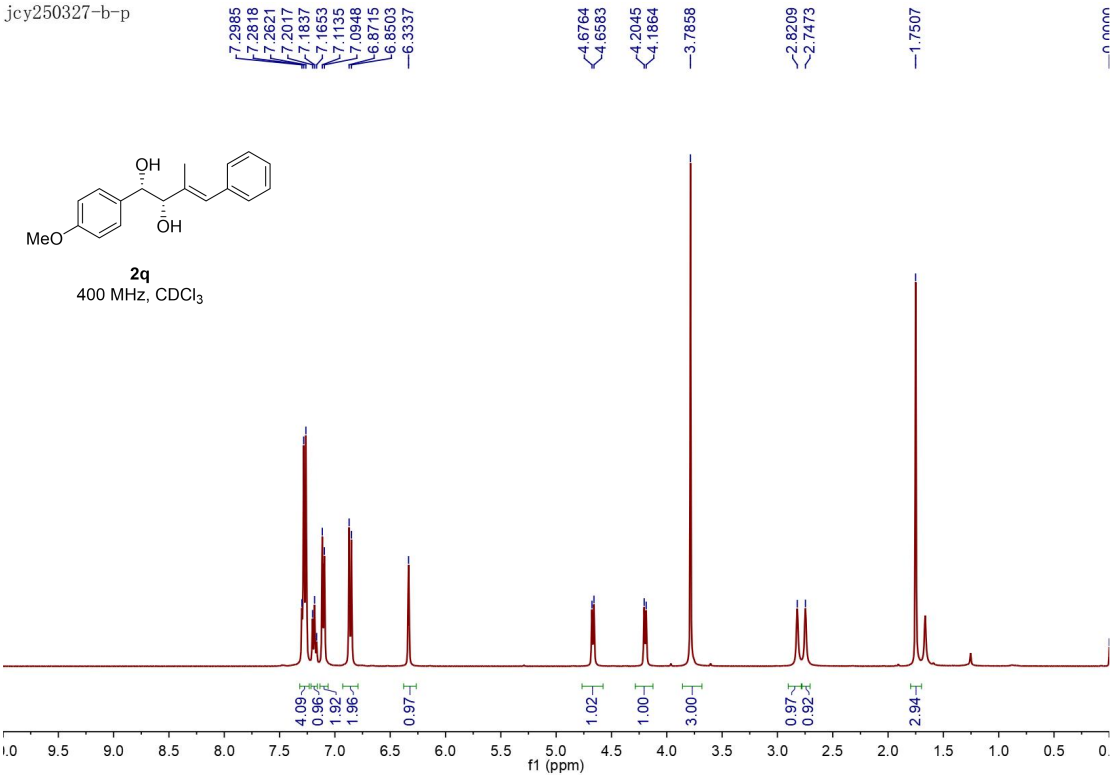
JCY250613-A-P



JCY250613-A-P-C
single pulse decoupled gated NOE

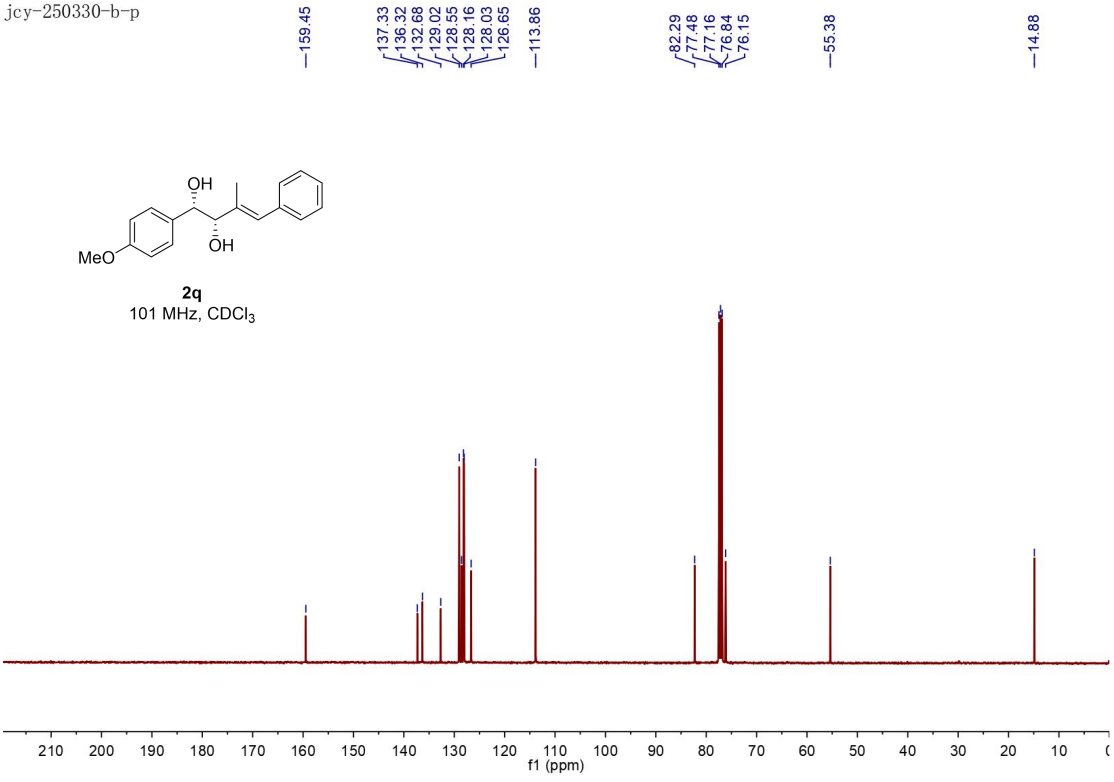


jcy250327-b-p



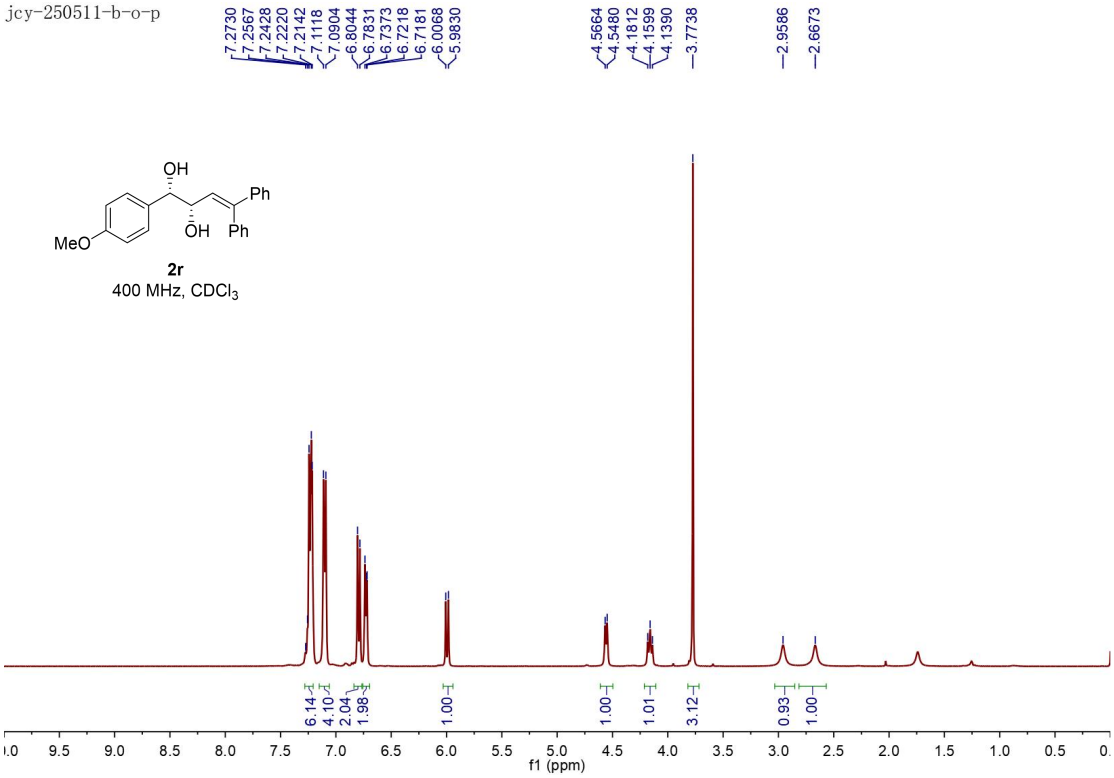
2q
400 MHz, $CDCl_3$

jcy-250330-b-p

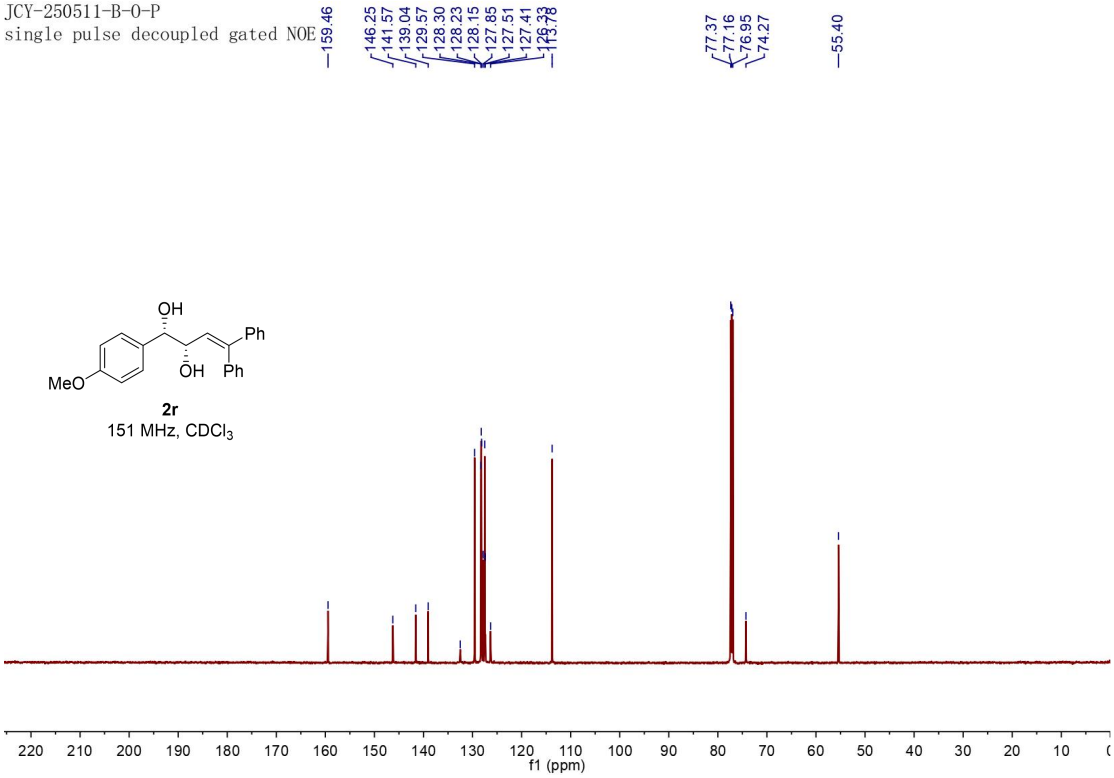


2q
101 MHz, $CDCl_3$

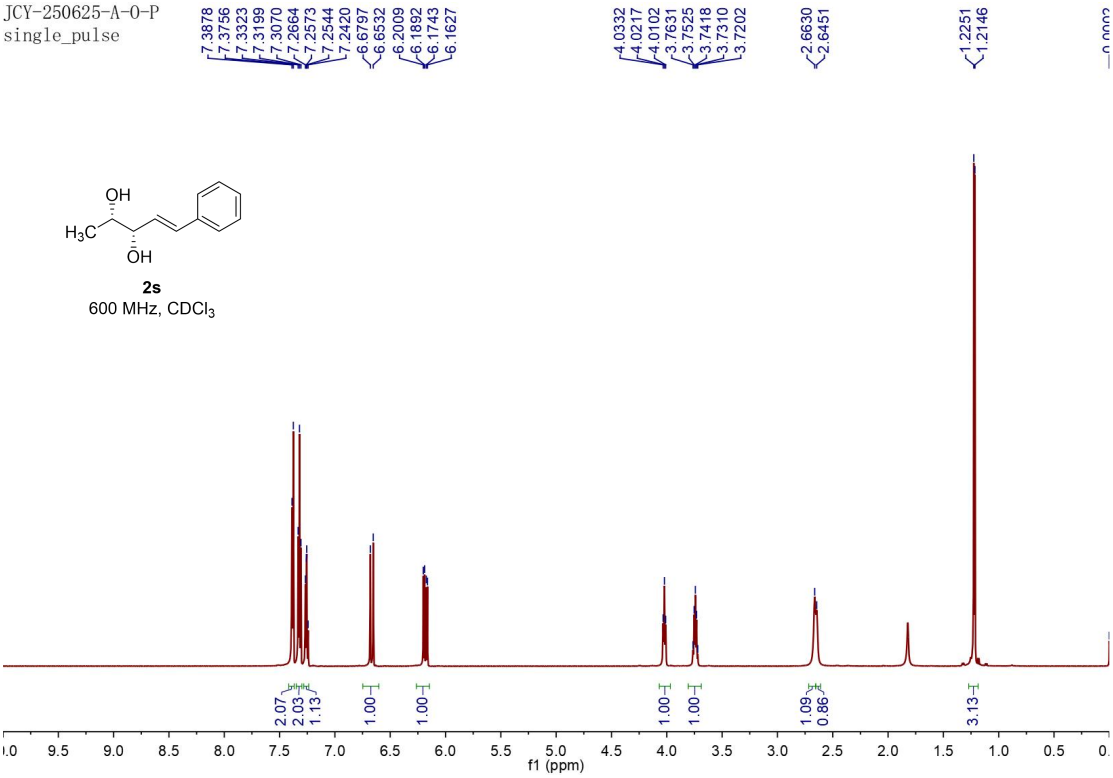
jcy-250511-b-o-p



JCY-250511-B-O-P
single pulse decoupled gated NOE

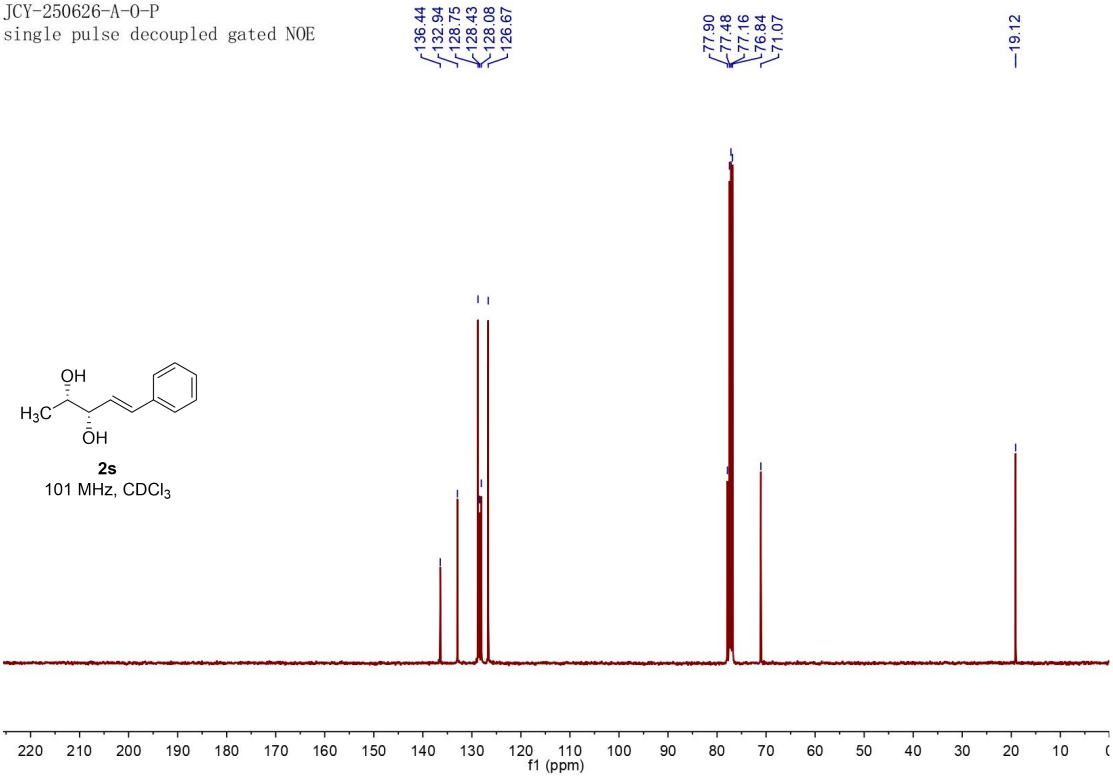


JCY-250625-A-O-P
single_pulse



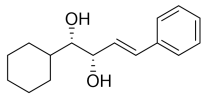
2s
600 MHz, $CDCl_3$

JCY-250626-A-O-P
single pulse decoupled gated NOE

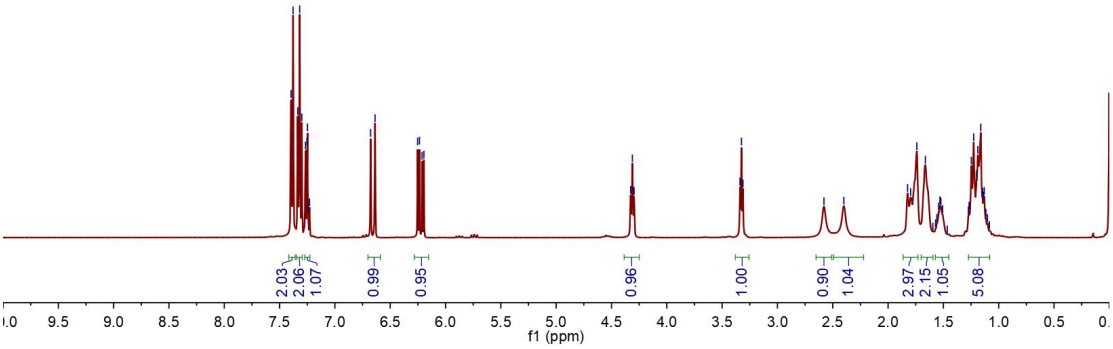


2s
101 MHz, $CDCl_3$

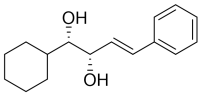
JCY-250902-CY-1-R
single_pulse



2t
400 MHz, $CDCl_3$

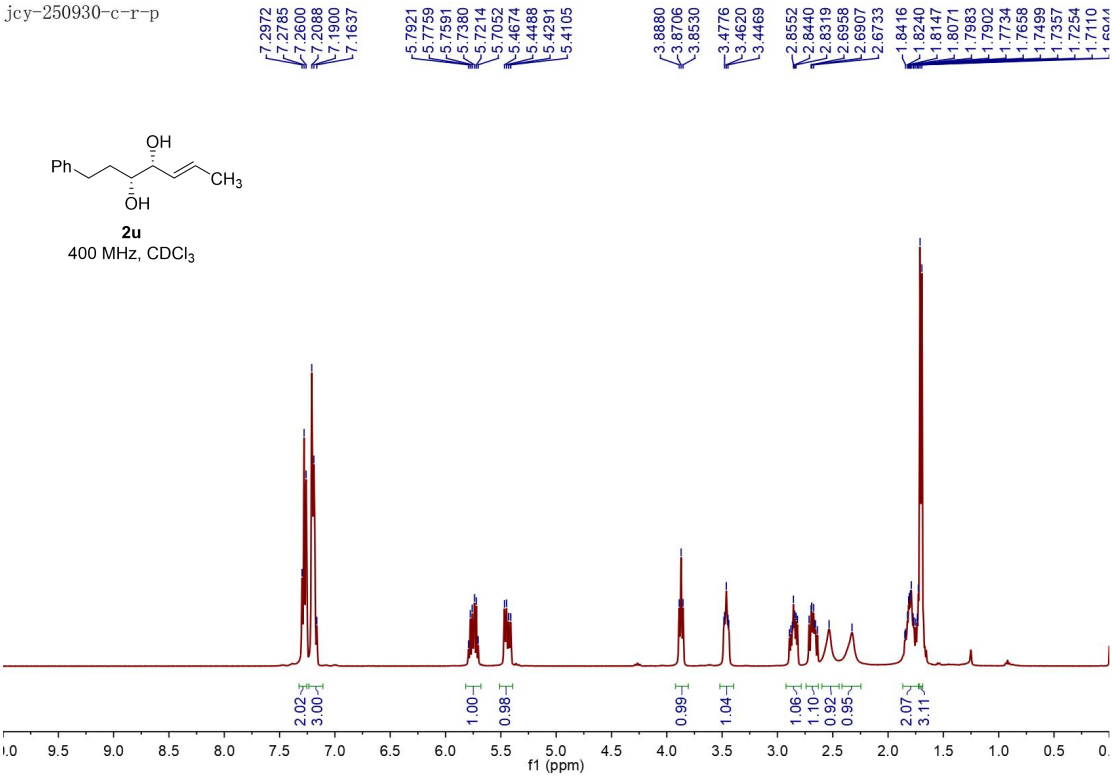


JCY-250902-CY-1-R
single pulse decoupled gated NOE



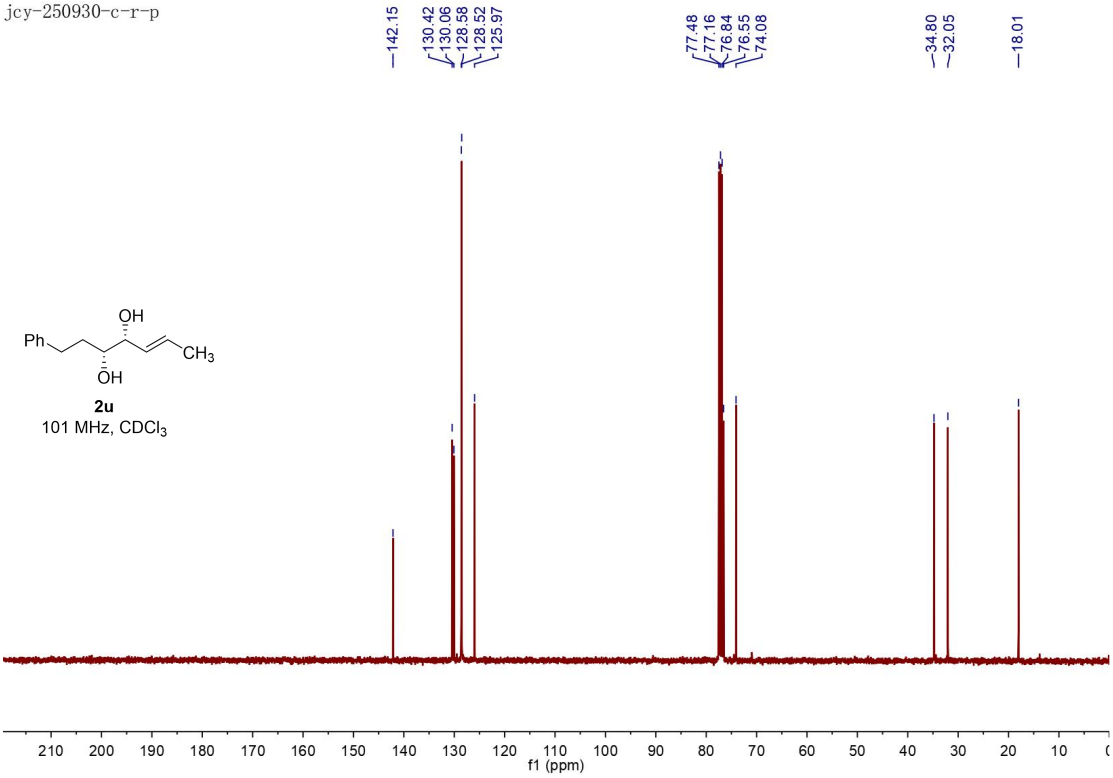
2t
101 MHz, $CDCl_3$

jcy-250930-c-r-p



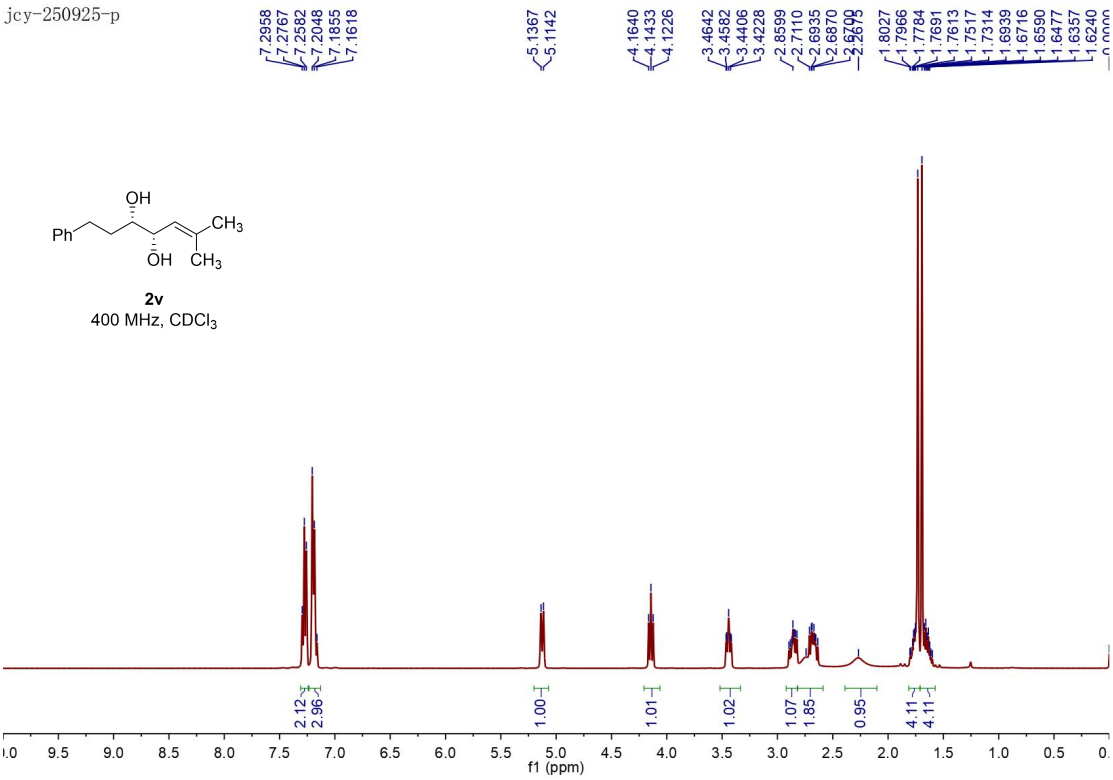
2u
400 MHz, $CDCl_3$

jcy-250930-c-r-p

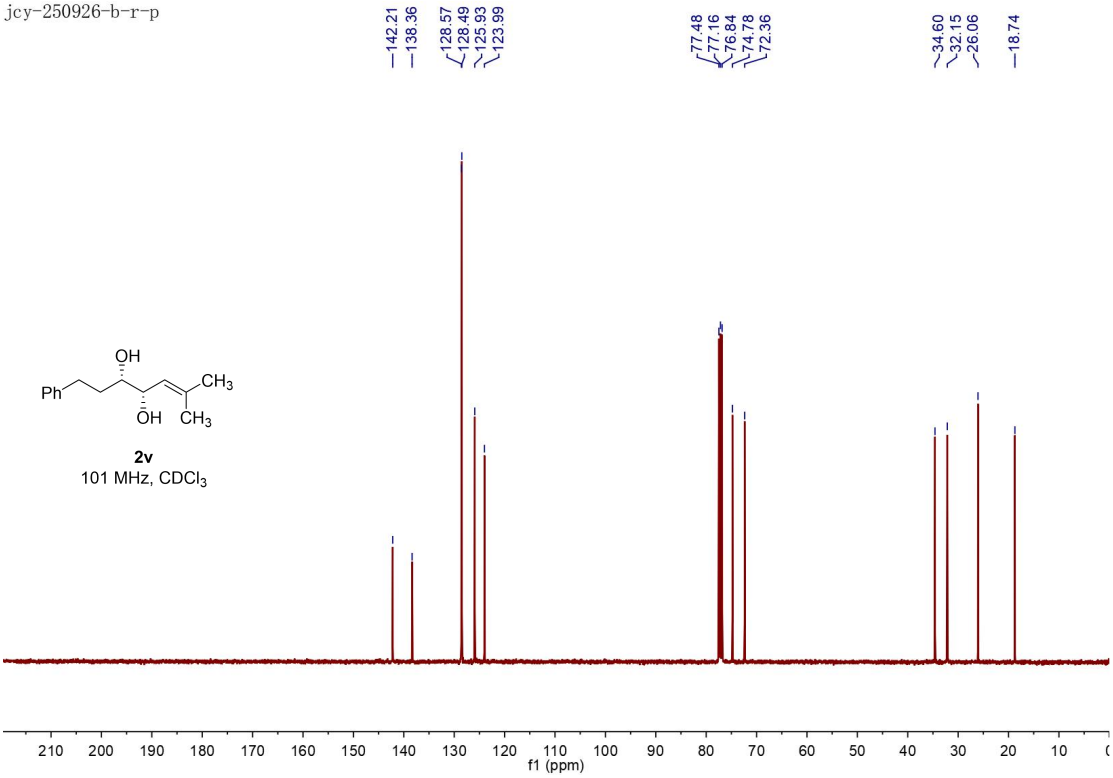


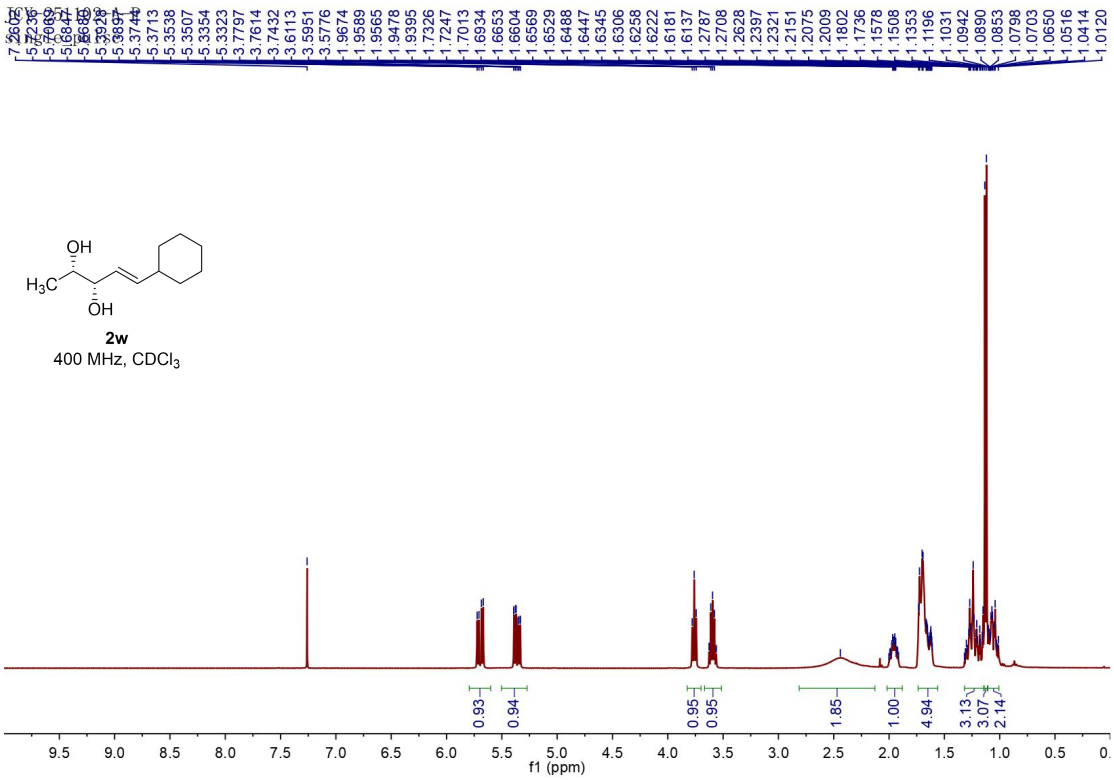
2u
101 MHz, $CDCl_3$

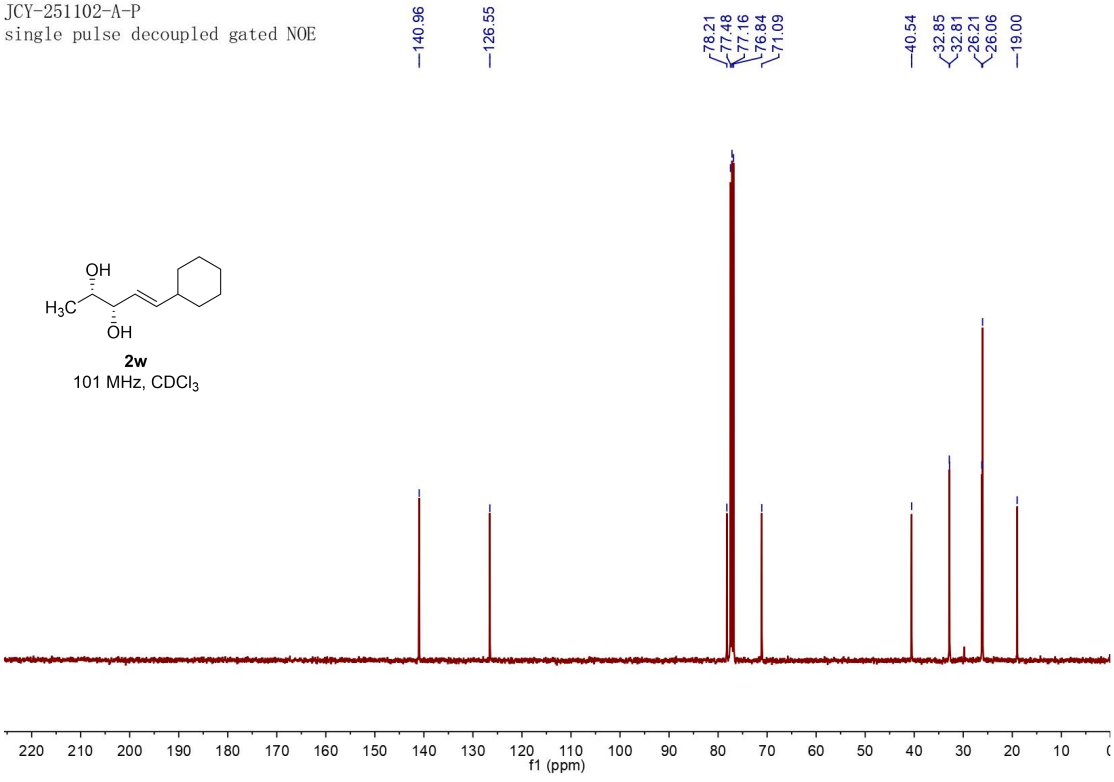
jcy-250925-p



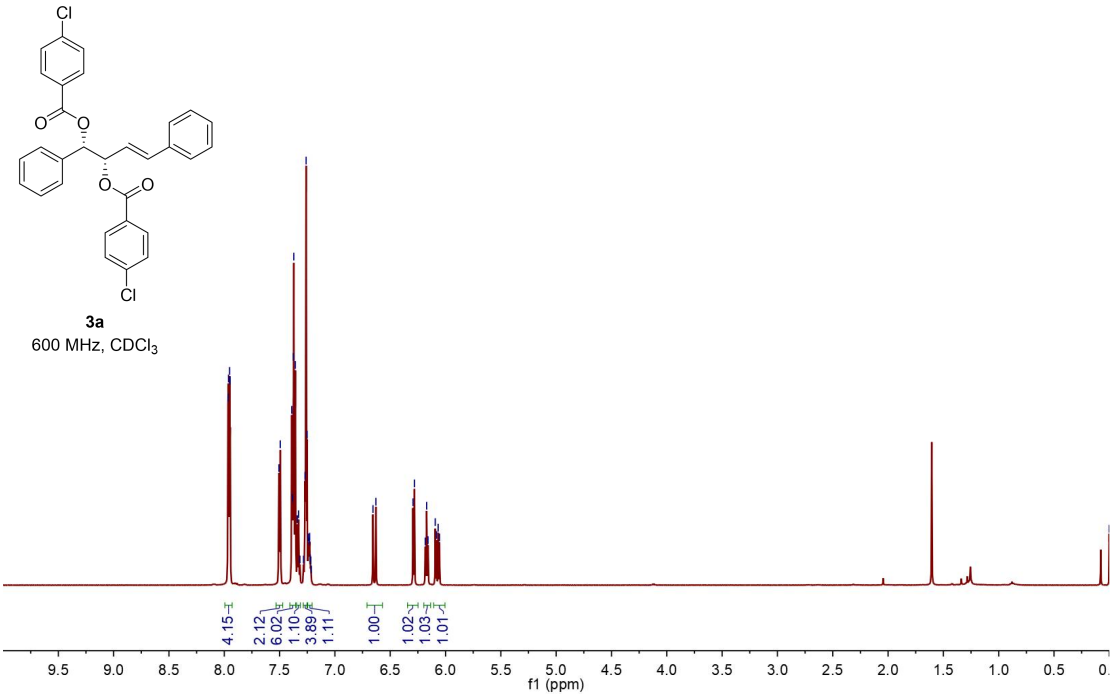
jcy-250926-b-r-p




OH
H3C
OH
2w
400 MHz, CDCl3
0.93
0.94
0.95
0.95
1.85
1.00
4.94
3.13
3.07
2.14
f1 (ppm)

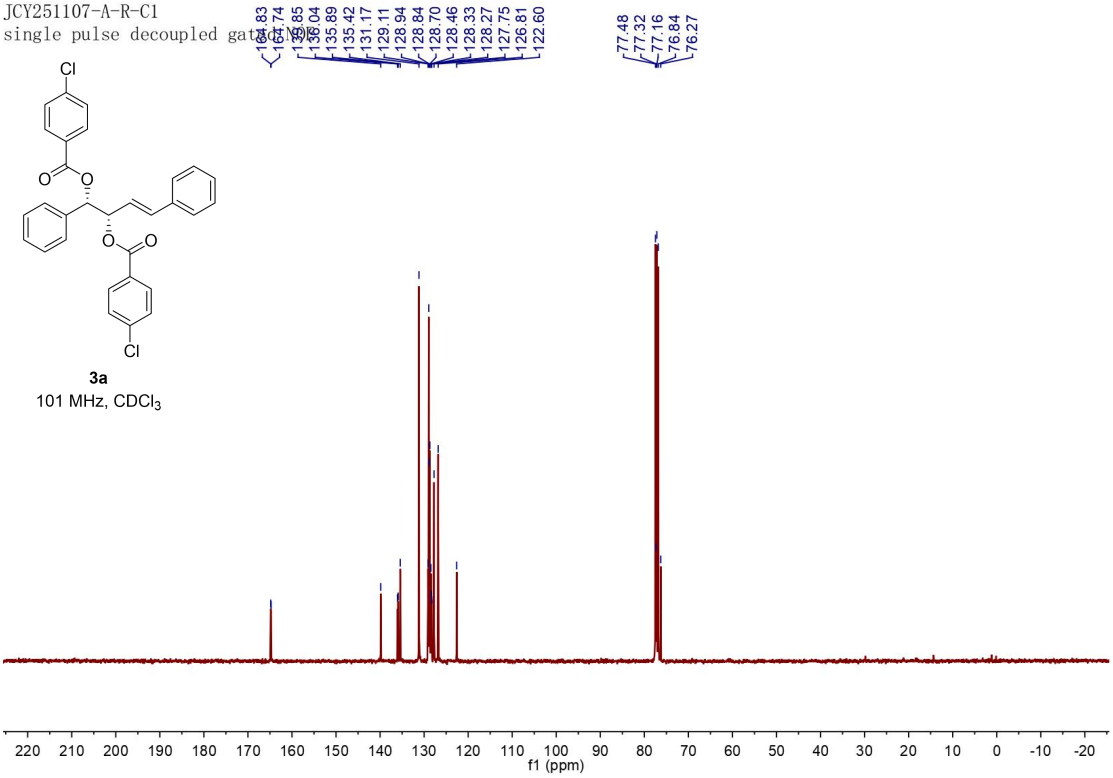

JCY-251102-A-P
single pulse decoupled gated NOE
140.96
126.55
78.21
77.48
77.16
76.84
71.09
40.54
32.85
32.81
26.21
26.06
19.00
OH
H3C
OH
2w
101 MHz, CDCl3
f1 (ppm)

JCY-251108-A-R-CL
single_pulse

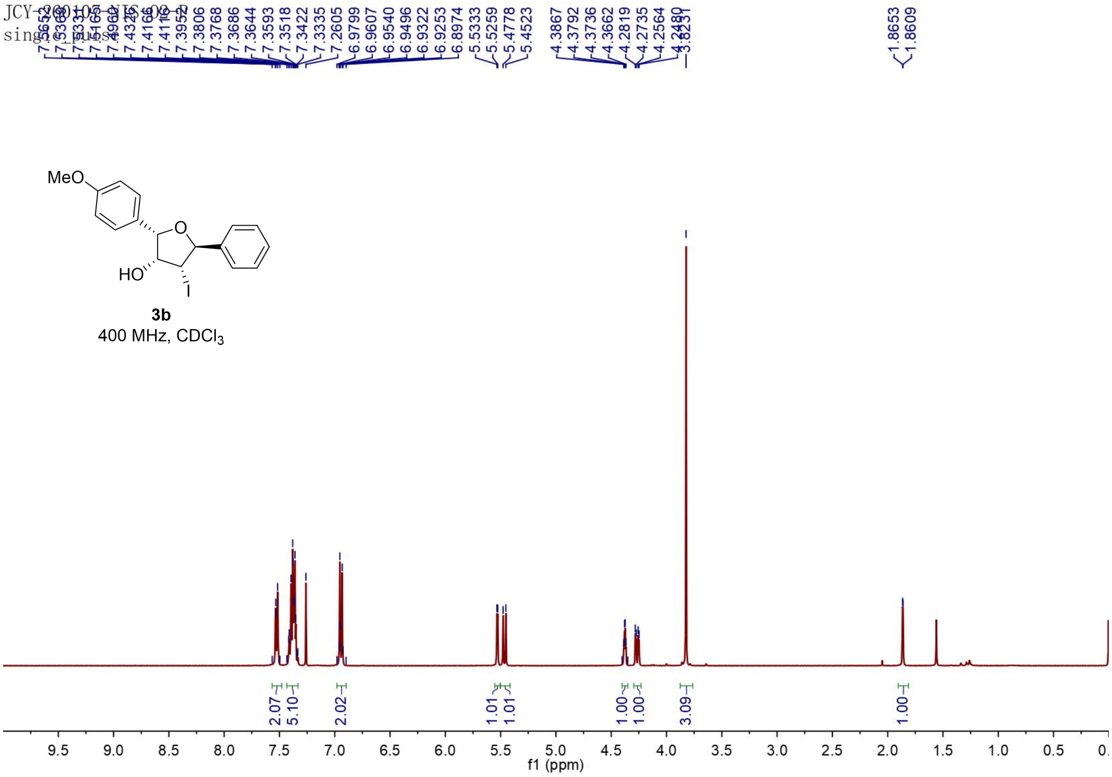


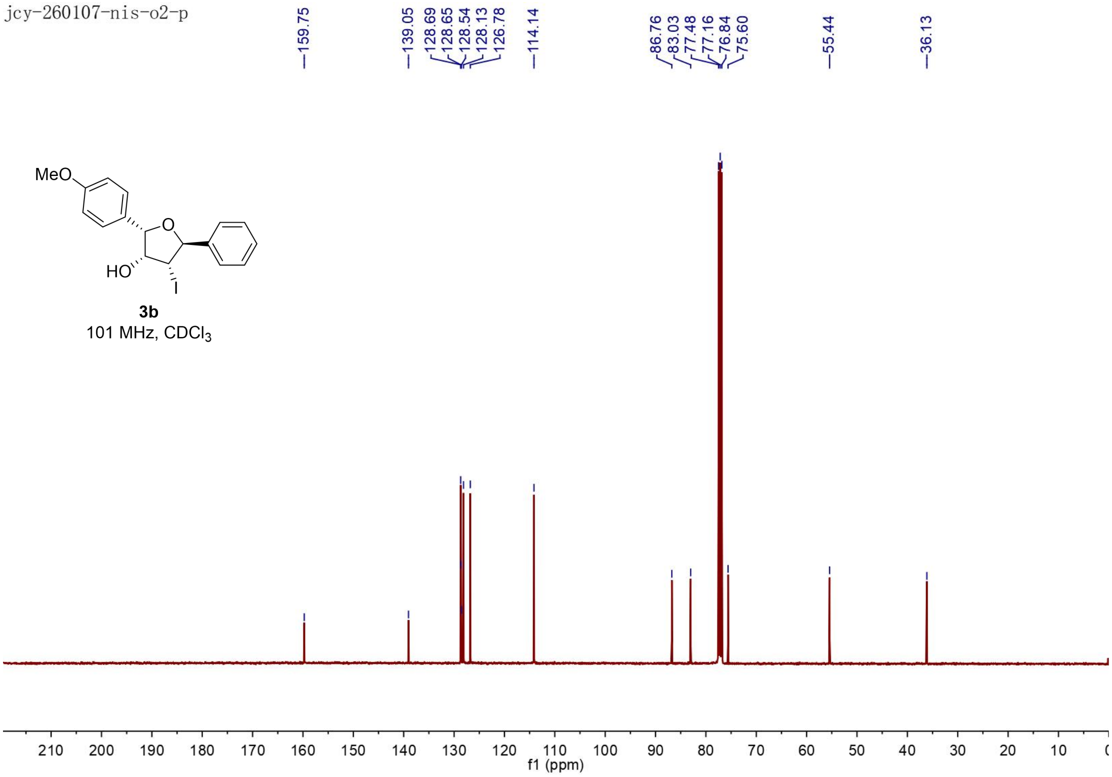
3a
600 MHz, $CDCl_3$

JCY251107-A-R-Cl
single pulse decoupled gated NOE

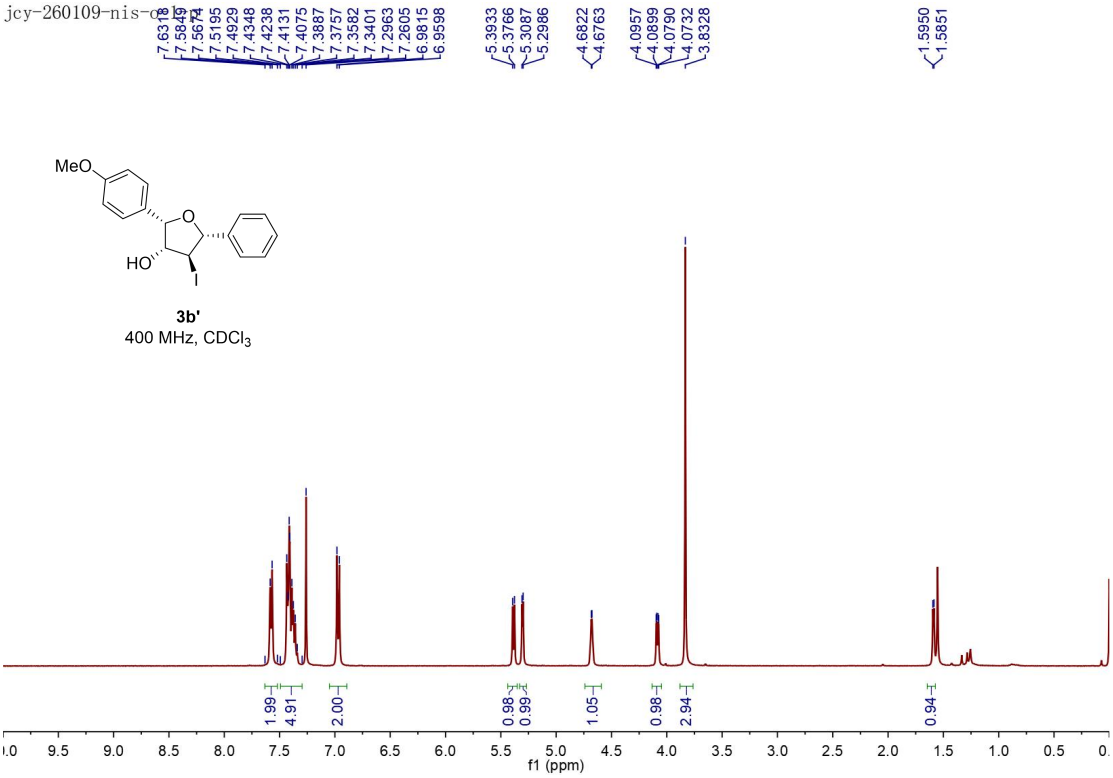


3a
101 MHz, $CDCl_3$


single pulse
7.5652 7.5360 7.5331 7.5165 7.4960 7.4326 7.4165 7.4116 7.3952 7.3806 7.3768 7.3686 7.3644 7.3593 7.3518 7.3422 7.3335 7.2605 6.9799 6.9607 6.9540 6.9496 6.9322 6.9253 6.8974 5.5333 5.5259 5.4778 5.4523 4.3867 4.3792 4.3736 4.3662 4.2819 4.2735 4.2564 4.2480 3.8231 1.8653 1.8609
MeO
O
HO
I
3b
400 MHz, CDCl3
2.07 5.10 2.02 1.01 1.01 1.00 1.00 3.09 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

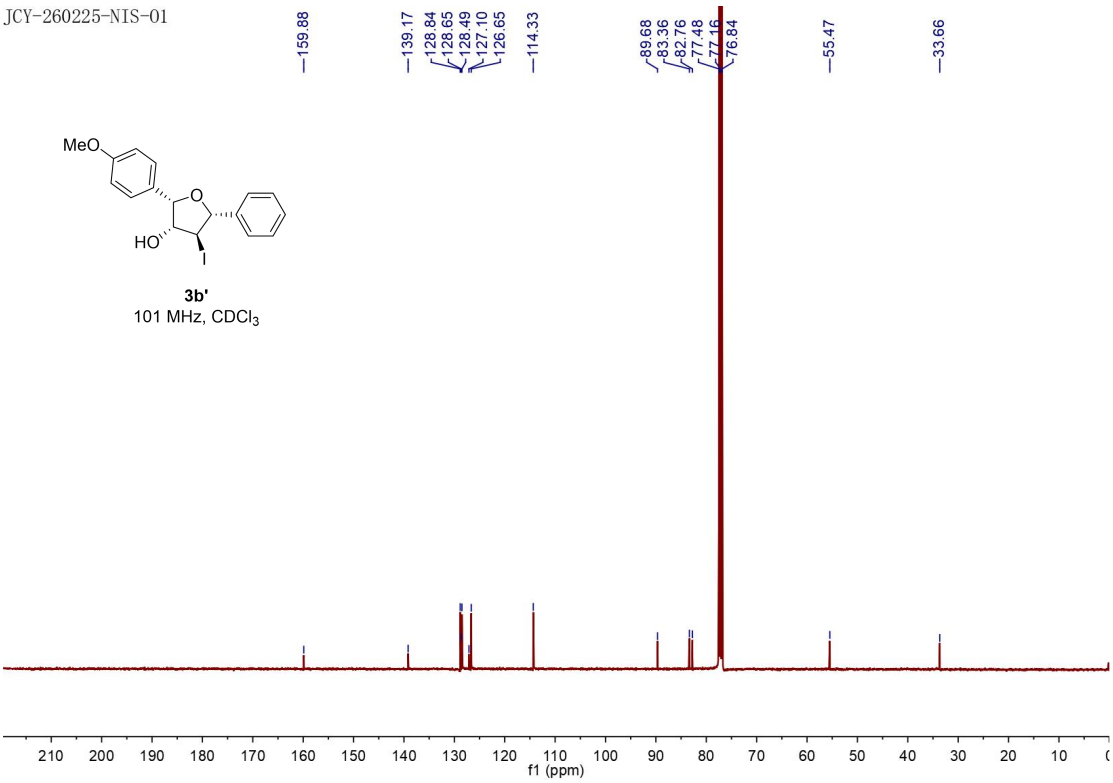

jcy-260107-nis-o2-p
159.75 139.05 128.69 128.65 128.54 128.13 126.78 114.14 86.76 83.03 77.48 77.16 76.84 75.60 55.44 36.13
MeO
O
HO
I
3b
101 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

jcy-260109-nis-c

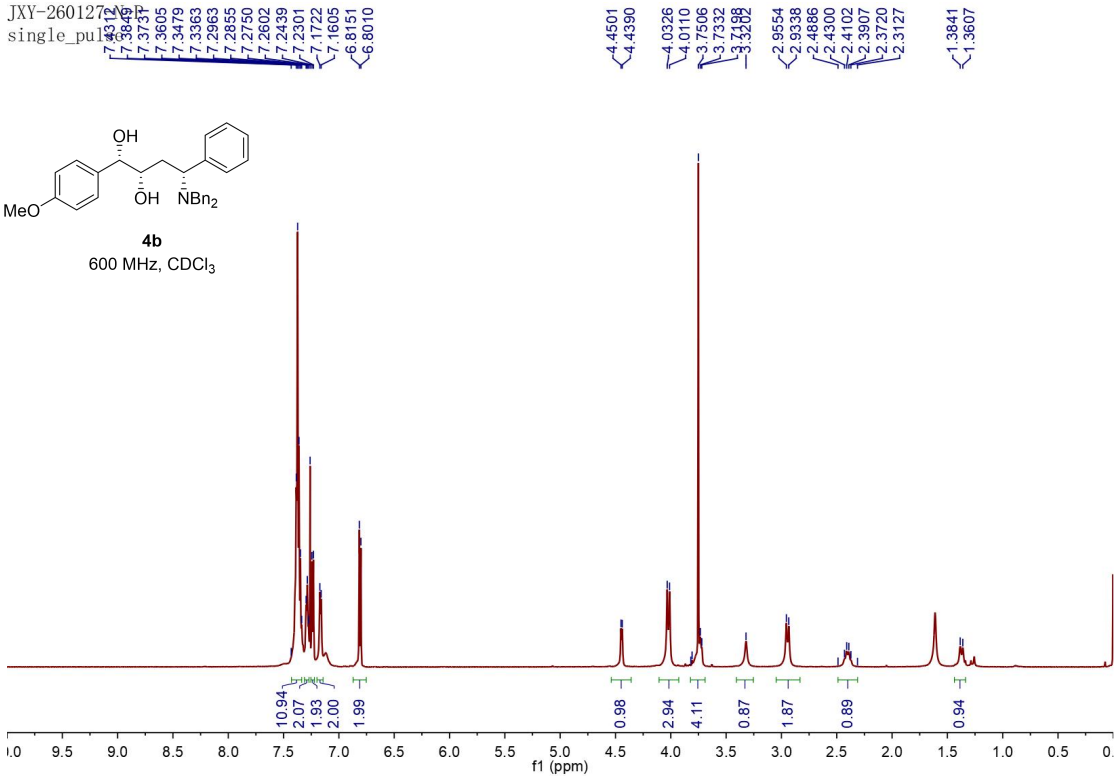


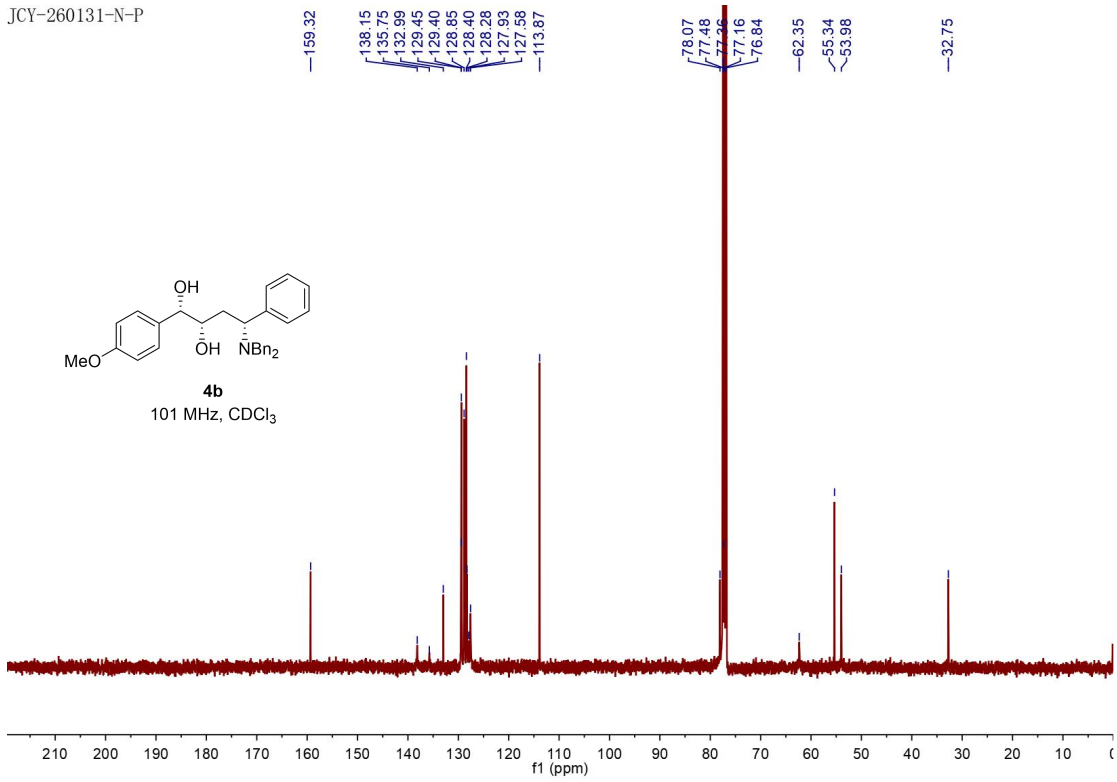
3b'
400 MHz, $CDCl_3$

JCY-260225-NIS-01

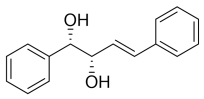


3b'
101 MHz, $CDCl_3$


JXY-260127-N-P
single_pul
7.4312
7.3845
7.3731
7.3605
7.3479
7.3363
7.2963
7.2855
7.2750
7.2602
7.2439
7.2301
7.1722
7.1605
6.8151
6.8010
4.4501
4.4390
4.0326
4.0110
3.7506
3.7332
3.3202
2.9554
2.9338
2.4886
2.4300
2.4102
2.3907
2.3720
2.3127
1.3841
1.3607
OH
OH
NBn2
MeO
4b
600 MHz, CDCl3
10.94
2.07
1.93
2.00
1.99
0.98
2.94
4.11
0.87
1.87
0.89
0.94
f1 (ppm)


JCY-260131-N-P
159.32
138.15
135.75
132.99
129.45
129.40
128.85
128.40
128.28
127.93
127.58
113.87
78.07
77.48
77.16
76.84
62.35
55.34
53.98
32.75
OH
OH
NBn2
MeO
4b
101 MHz, CDCl3
f1 (ppm)

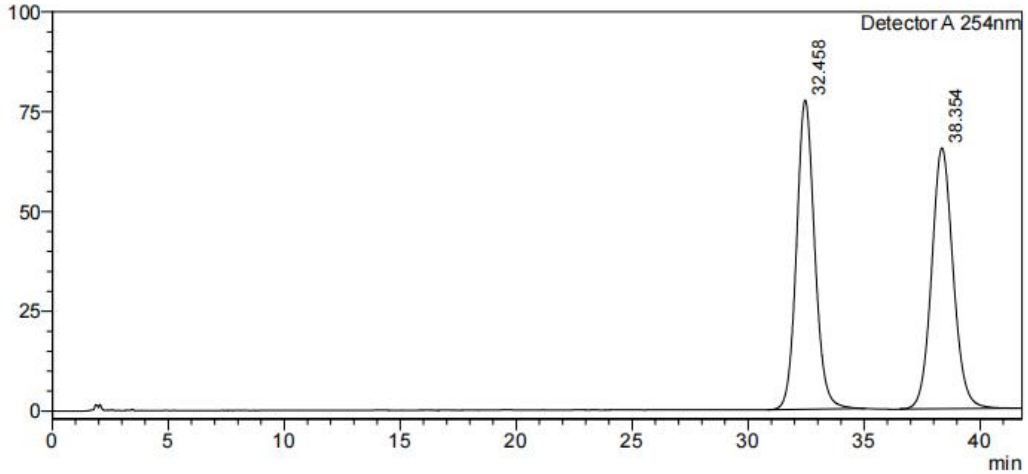
IX. HPLC spectra of the products



2a

<Chromatogram>

mV



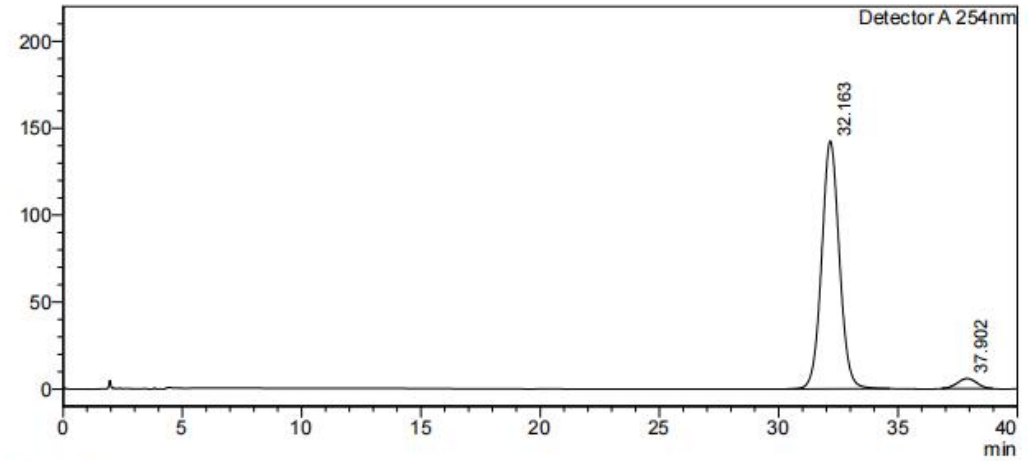
<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	32.458	4224168	77481	50.012		M	
2	38.354	4222059	65343	49.988		M	
Total		8446227	142824				

<Chromatogram>

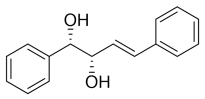
mV



<Peak Table>

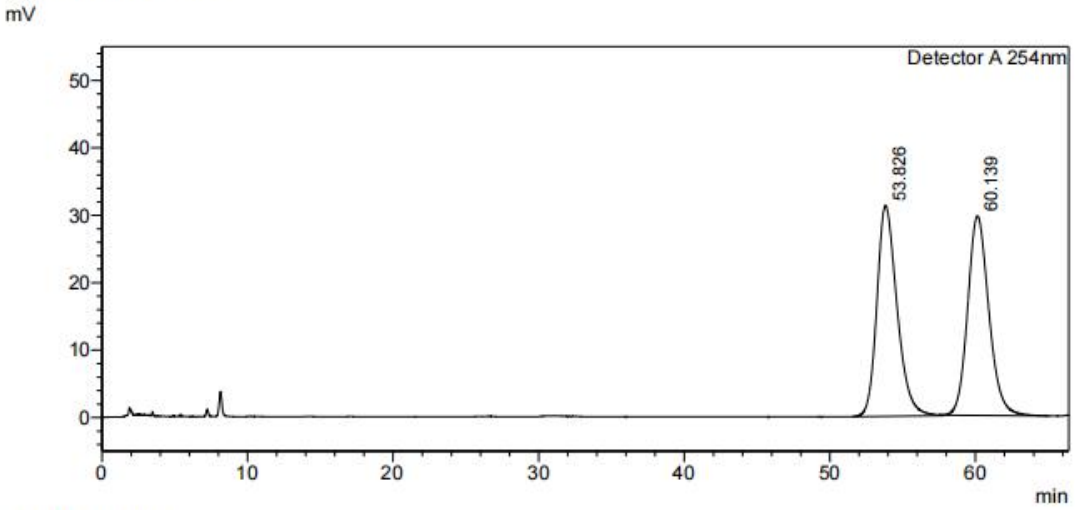
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	32.163	7370625	142603	95.814		M	
2	37.902	321994	5735	4.186		M	
Total		7692620	148338				



2b

<Chromatogram>

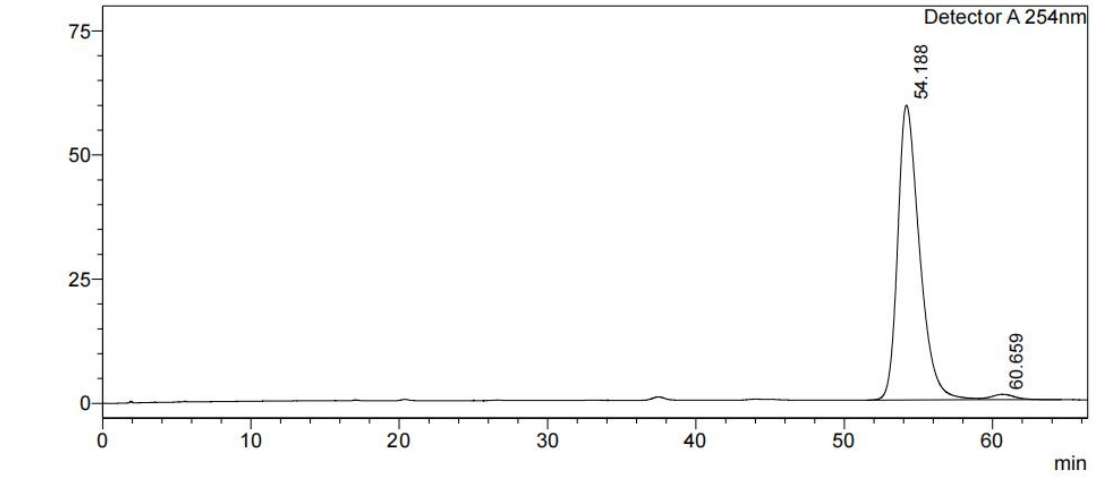


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	53.826	3014957	31231	50.139		M	
2	60.139	2998236	29568	49.861		M	
Total		6013193	60800				

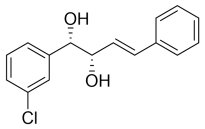
<Chromatogram>



<Peak Table>

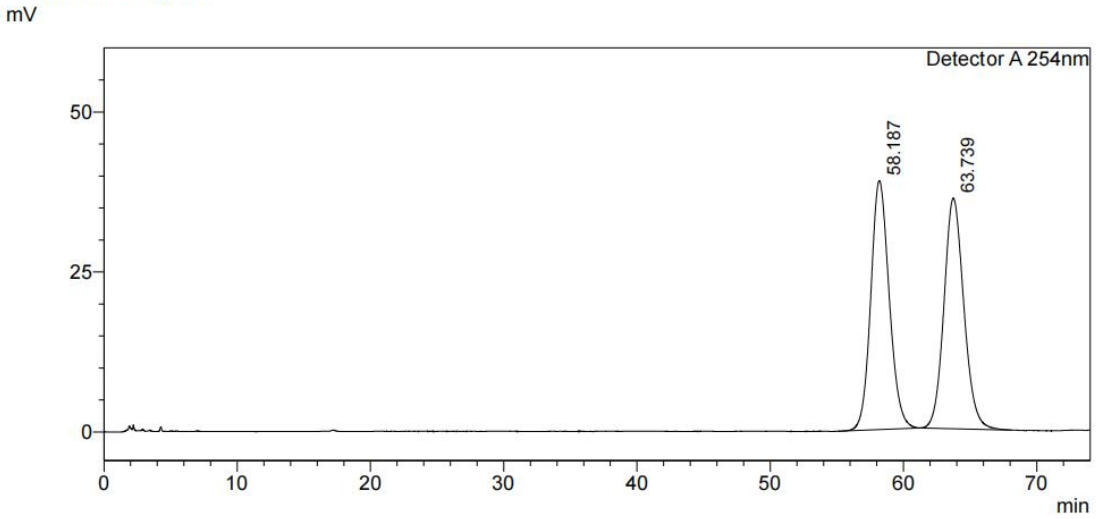
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	54.188	6079991	59370	97.999		M	
2	60.659	124116	1085	2.001		M	
Total		6204107	60455				



2c

<Chromatogram>

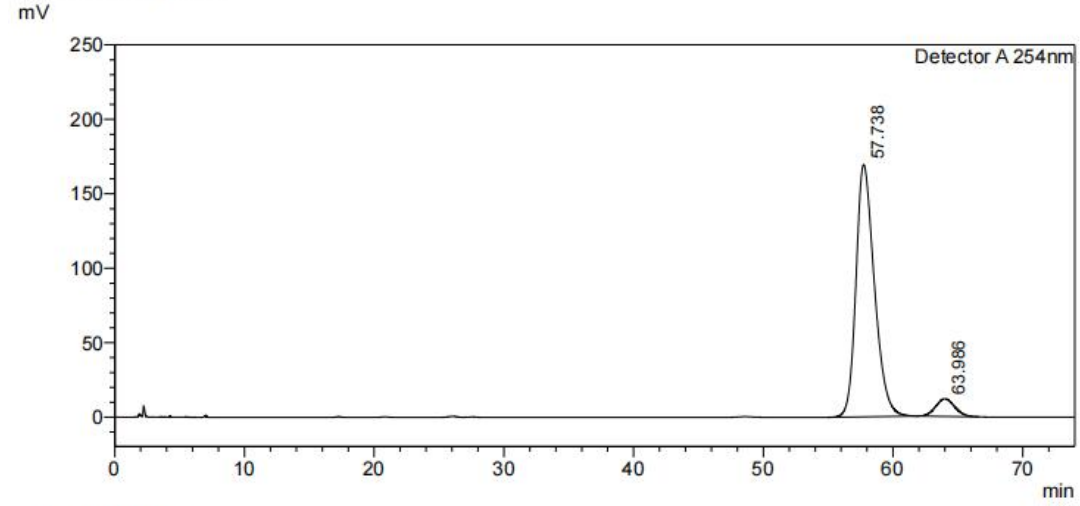


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	58.187	3743651	38908	49.955		M	
2	63.739	3750436	36017	50.045		M	
Total		7494087	74925				

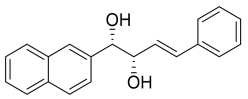
<Chromatogram>



<Peak Table>

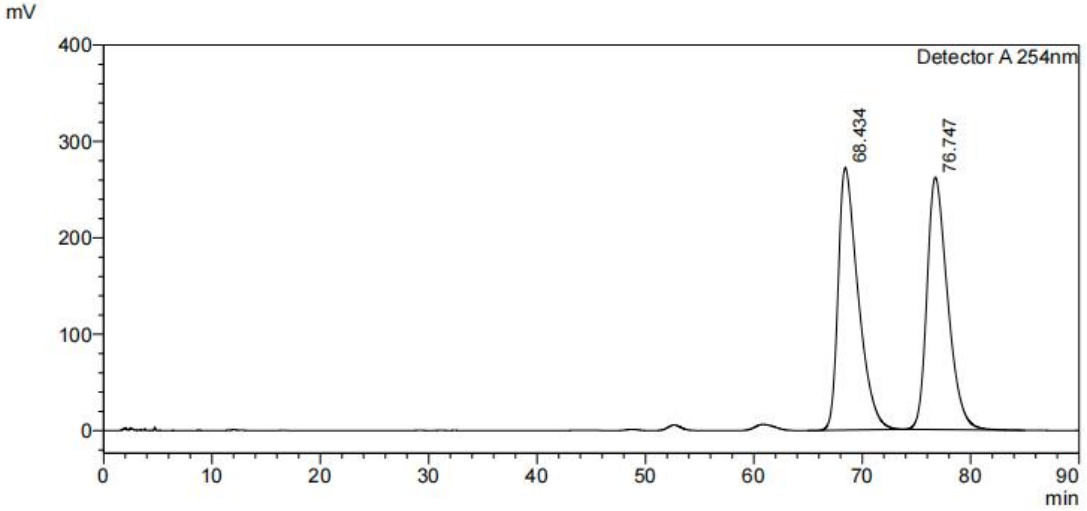
Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	57.738	16706590	169321	93.251		M	
2	63.986	1209103	11841	6.749		M	
Total		17915692	181162				



2d

<Chromatogram>

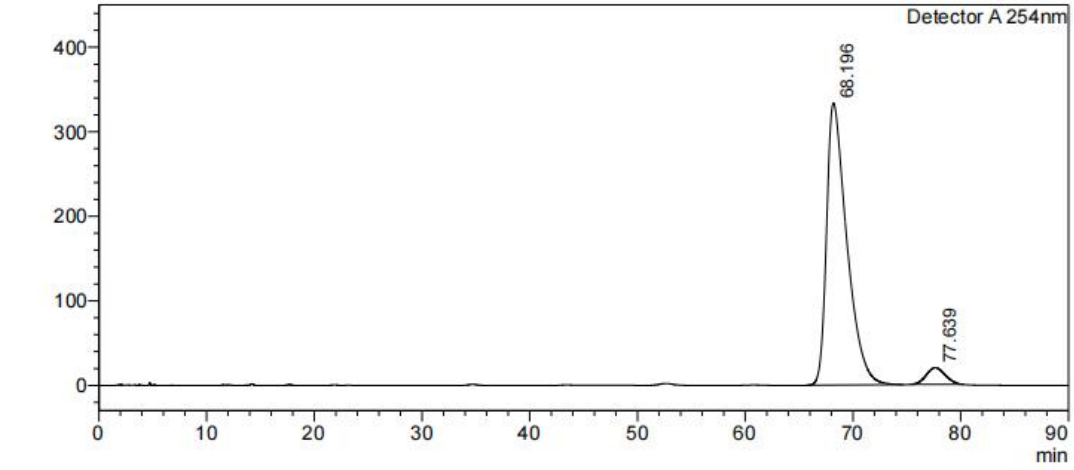


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	68.434	34945243	272011	50.185		M	
2	76.747	34687407	261441	49.815		M	
Total		69632650	533453				

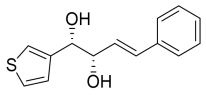
<Chromatogram>



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	68.196	43620391	333325	94.780		M	
2	77.639	2402437	19658	5.220		M	
Total		46022828	352983				



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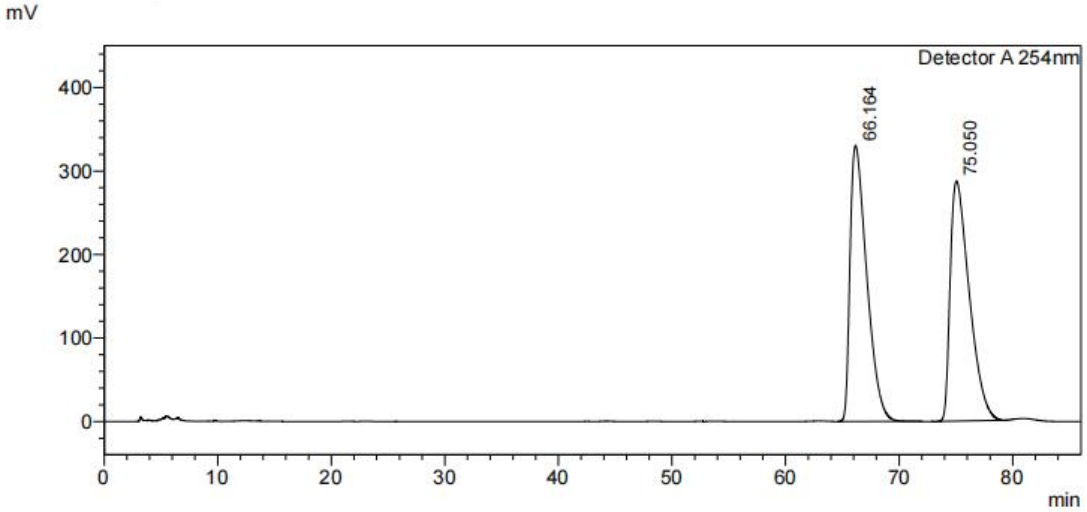
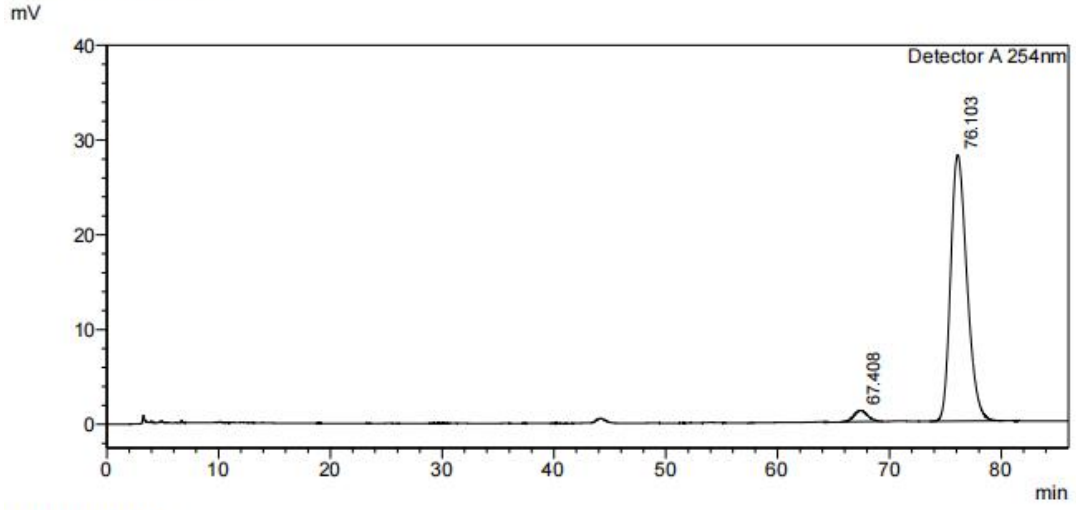


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	66.164	33778011	330501	50.208		M	
2	75.050	33498604	287498	49.792		M	
Total		67276615	617998				

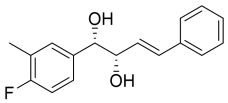
<Chromatogram>



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	67.408	100700	1182	3.411		M	
2	76.103	2851846	28113	96.589		M	
Total		2952545	29296				



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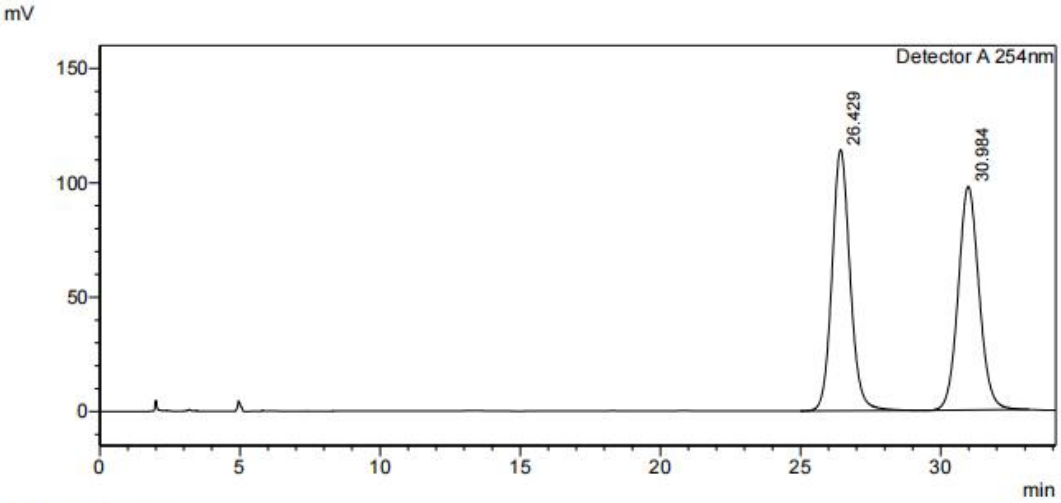
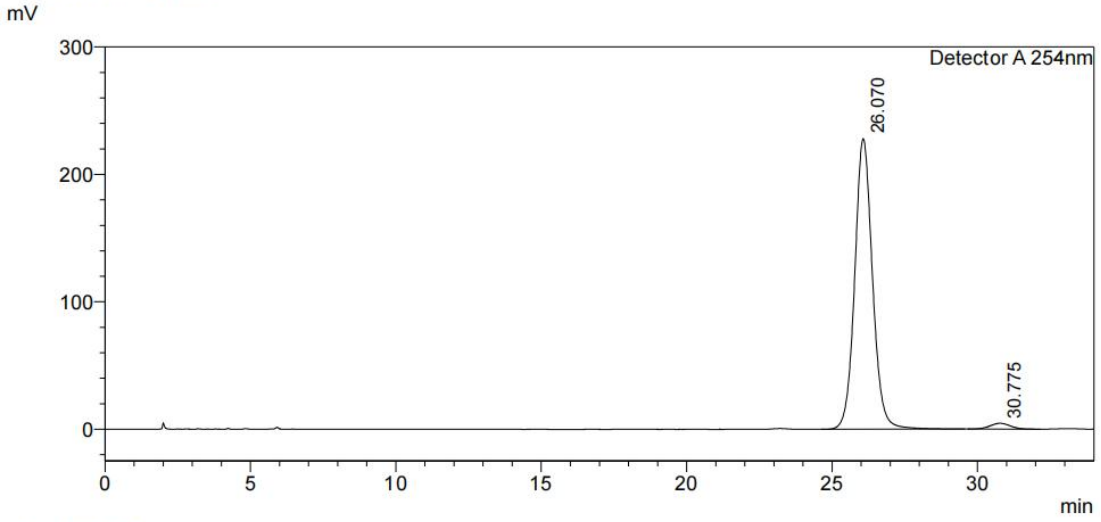


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	26.429	5118205	114156	50.269			
2	30.984	5063475	97763	49.731		M	
Total		10181680	211920				

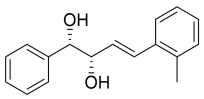
<Chromatogram>



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	26.070	9698772	228059	97.772			
2	30.775	221036	4558	2.228			
Total		9919808	232617				



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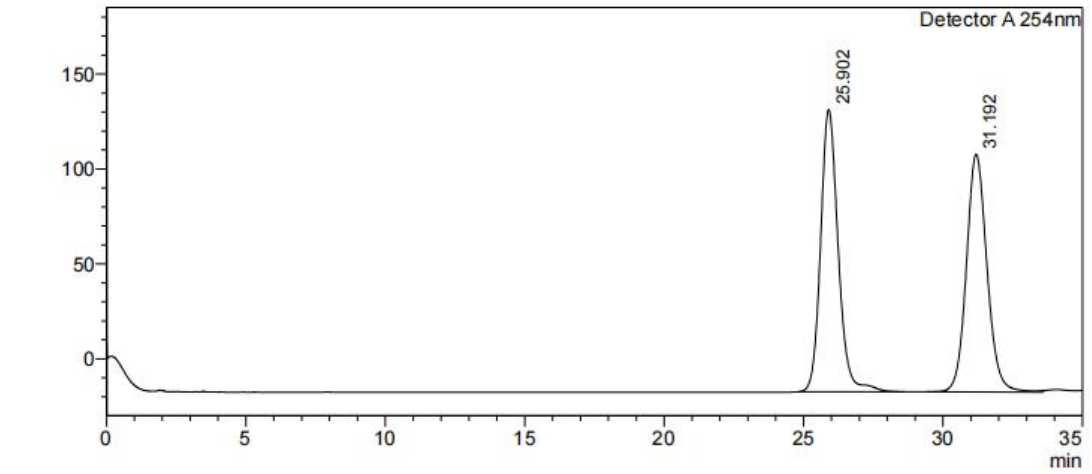
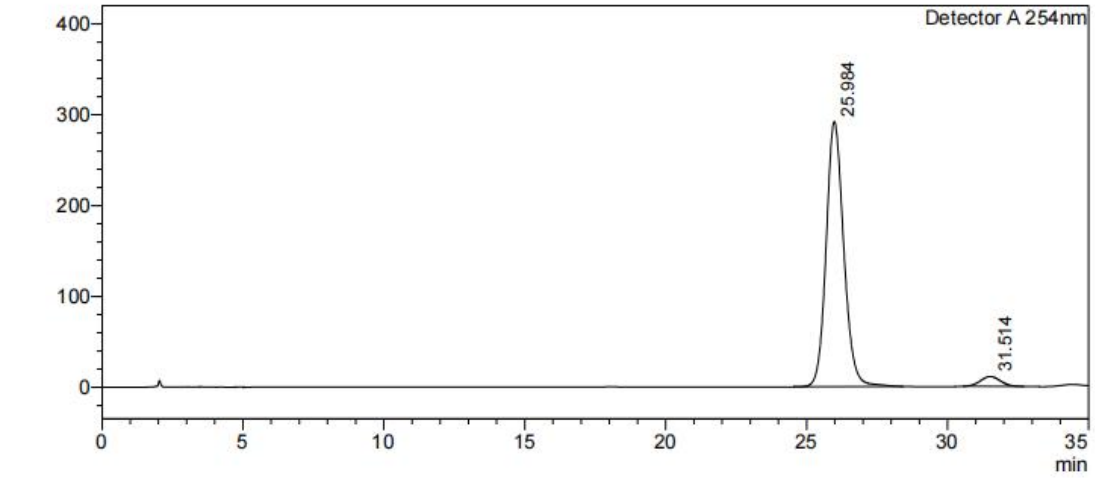


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	25.902	6514839	148654	50.271		M	
2	31.192	6444483	125240	49.729		M	
Total		12959322	273894				

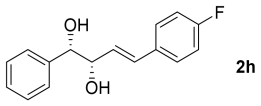
<Chromatogram>



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	25.984	12690003	292035	95.976		M	
2	31.514	532063	10796	4.024		M	
Total		13222065	302831				



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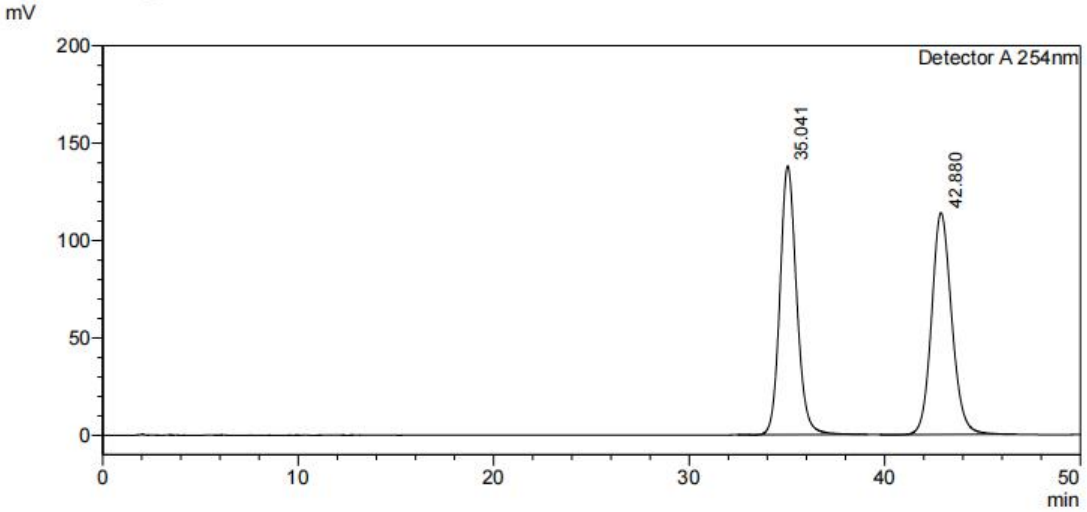
<Chromatogram>

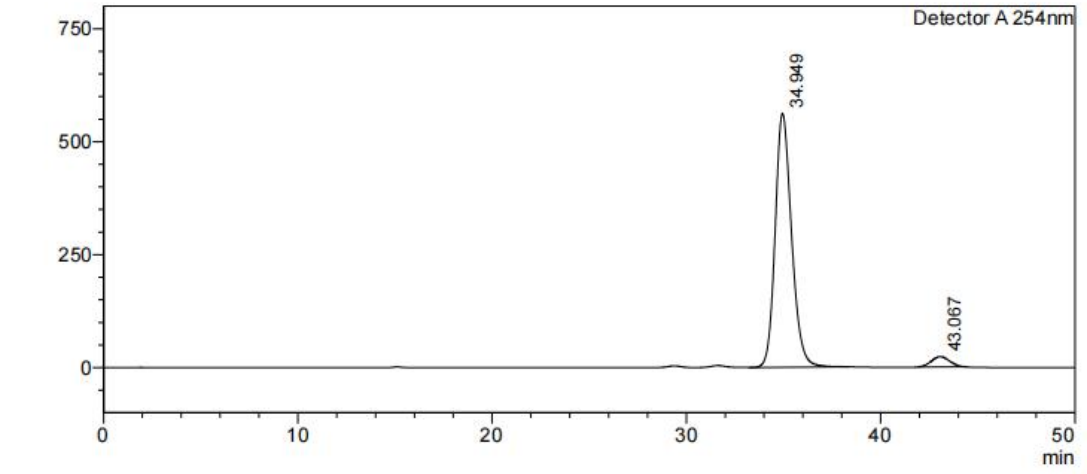


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	35.041	8155928	137890	50.025		M	
2	42.880	8147717	113846	49.975		M	
Total		16303644	251736				

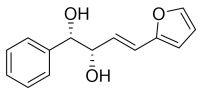
<Chromatogram>



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	34.949	32998823	561837	95.655		M	
2	43.067	1498780	22794	4.345		M	
Total		34497602	584631				



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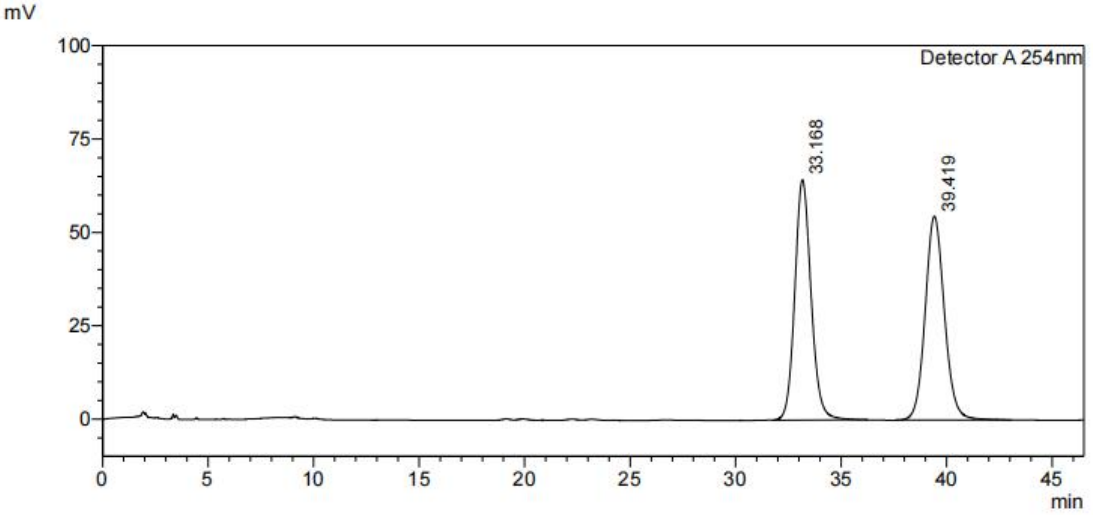
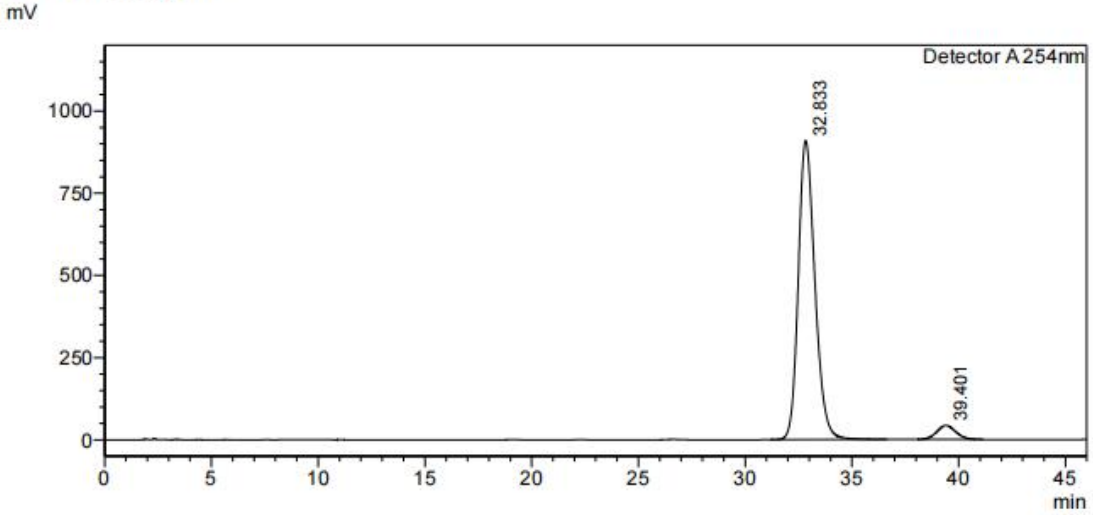


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	33.168	3460477	64356	49.948		V	
2	39.419	3467681	54607	50.052		S	
Total		6928158	118963				

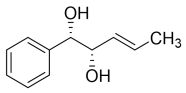
<Chromatogram>



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	32.833	49192456	909725	94.921		M	
2	39.401	2632386	42488	5.079		M	
Total		51824842	952213				



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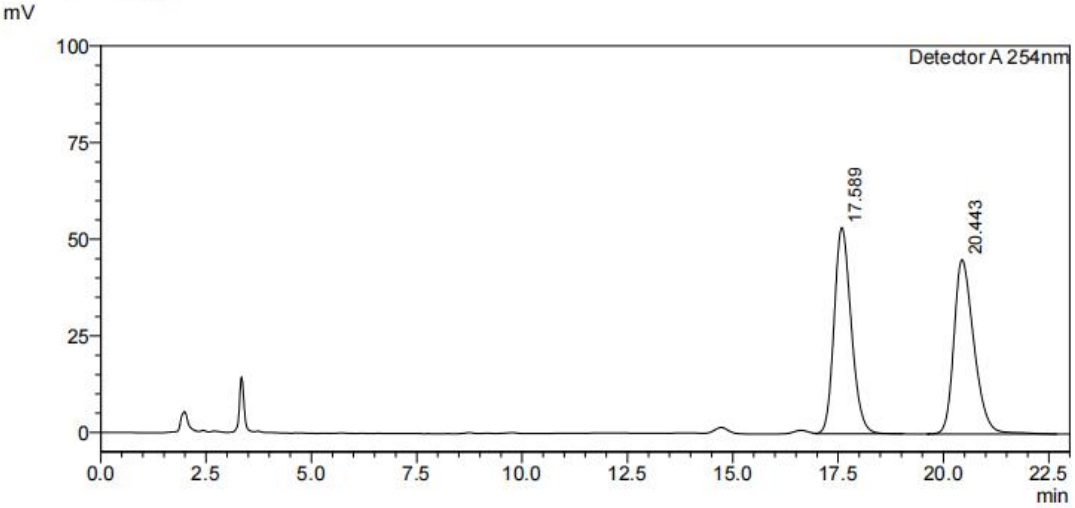
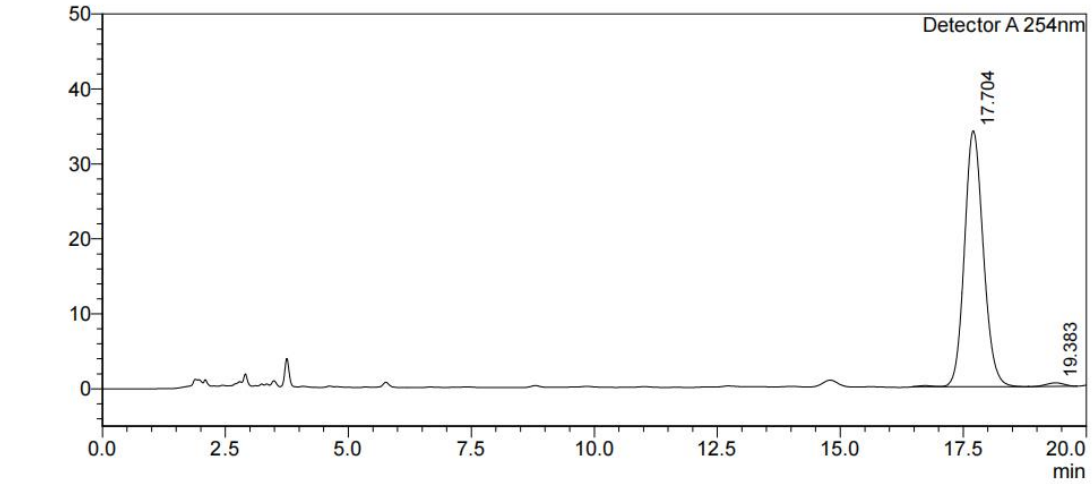


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.589	1511188	53410	50.250			
2	20.443	1496179	45102	49.750			
Total		3007367	98512				

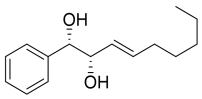
<Chromatogram>



<Peak Table>

Detector A 254nm

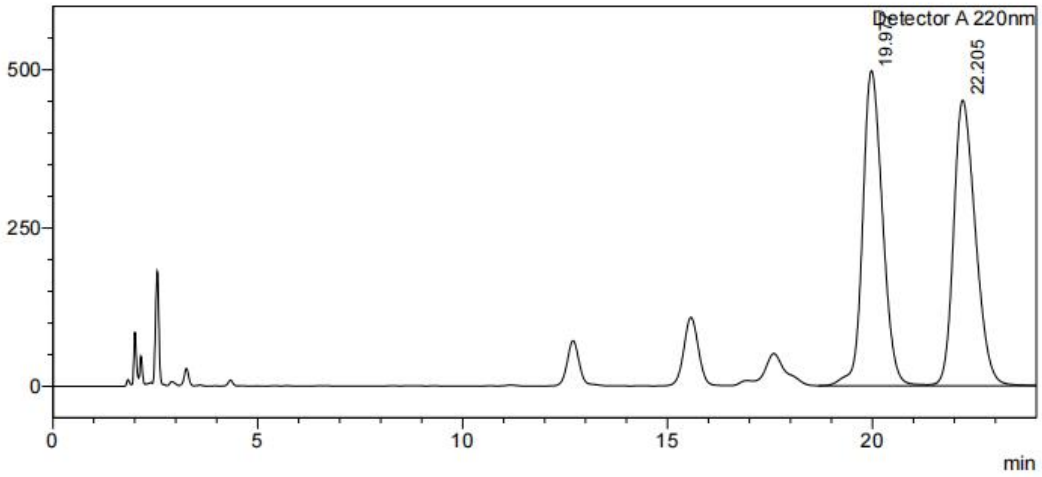
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	17.704	914185	34139	98.675		M	
2	19.383	12275	481	1.325		M	
Total		926460	34620				



2k

<Chromatogram>

mV



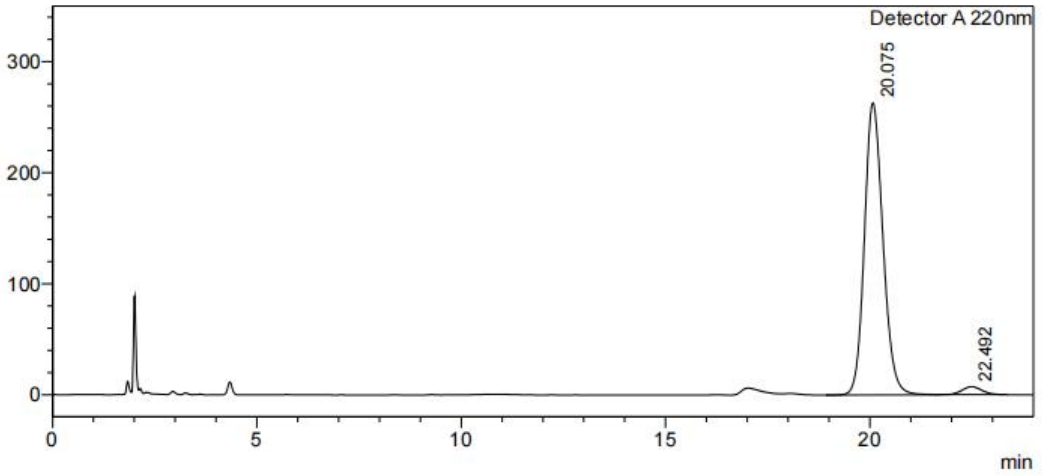
<Peak Table>

Detector A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	19.977	16812932	498019	50.067			
2	22.205	16767660	451361	49.933		V	
Total		33580592	949380				

<Chromatogram>

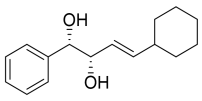
mV



<Peak Table>

Detector A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	20.075	8331425	263262	97.155			
2	22.492	243981	7219	2.845		M	
Total		8575407	270482				



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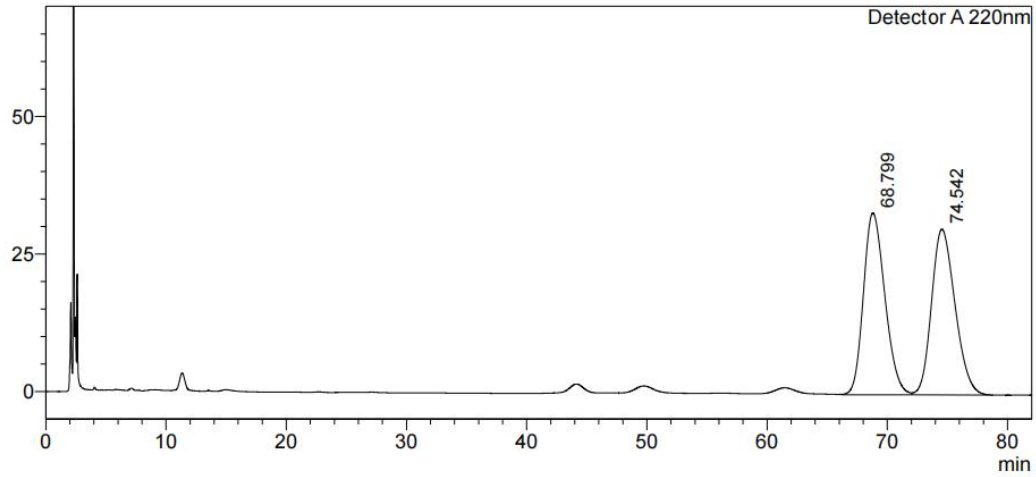
mV



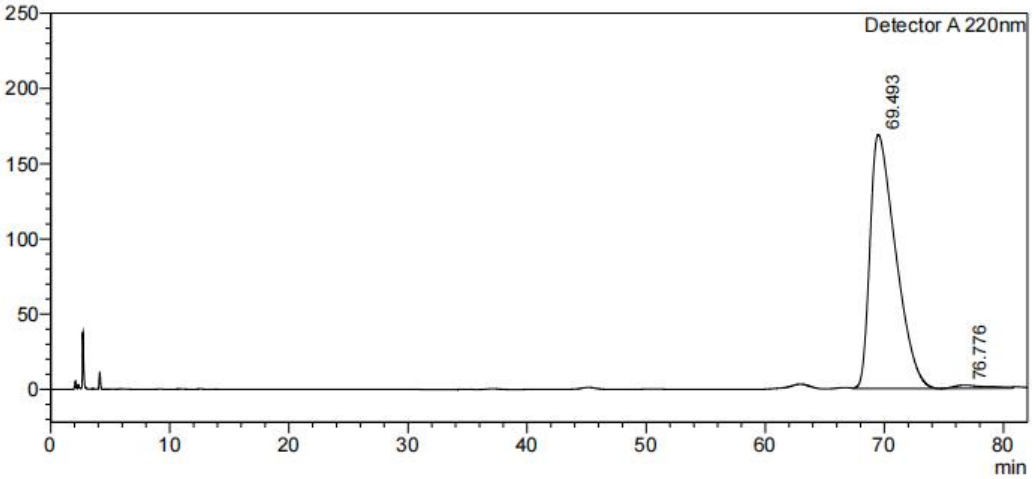
<Peak Table>

Detector A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	68.799	4105110	33056	50.068		M	
2	74.542	4094006	30129	49.932		V	
Total		8199116	63185				

<Chromatogram>

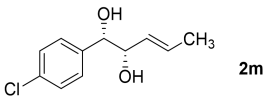
mV



<Peak Table>

Detector A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	69.493	25260868	168858	98.272		M	
2	76.776	444205	2194	1.728		M	
Total		25705073	171051				



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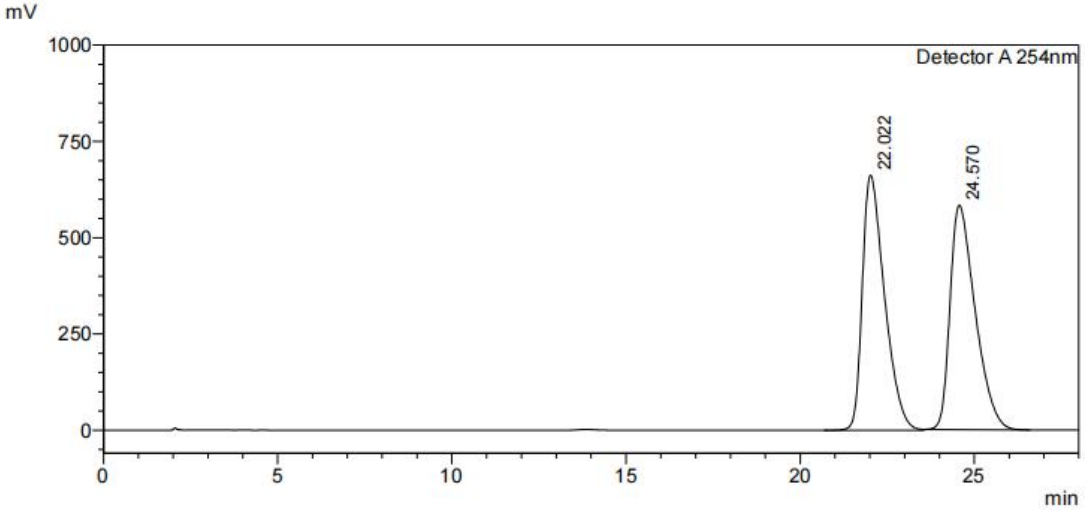

<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	22.022	29383262	662035	50.101			
2	24.570	29264460	582503	49.899		M	
Total		58647722	1244539				

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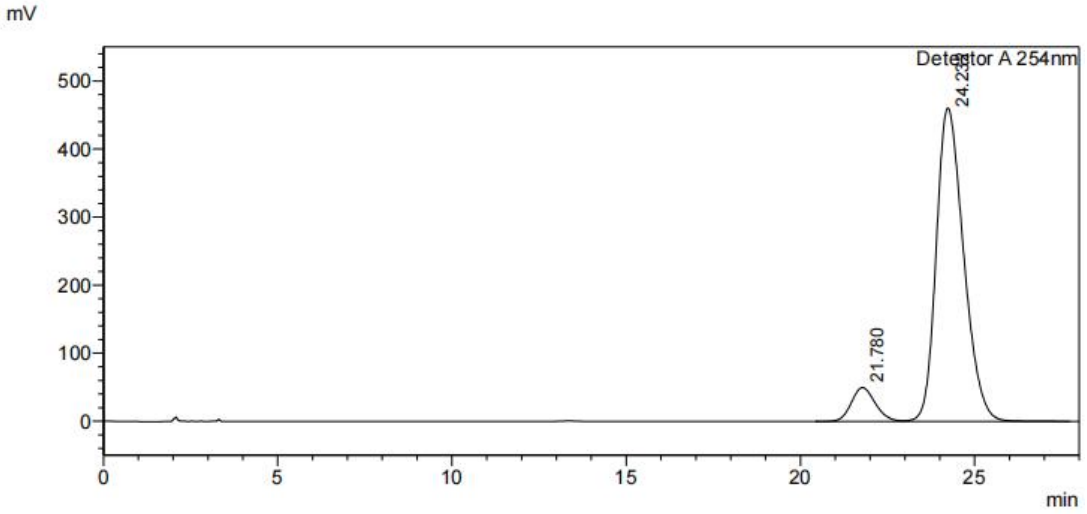
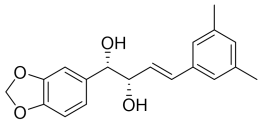


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	21.780	2388983	49765	8.671			
2	24.232	25163076	460843	91.329		V	
Total		27552059	510608				



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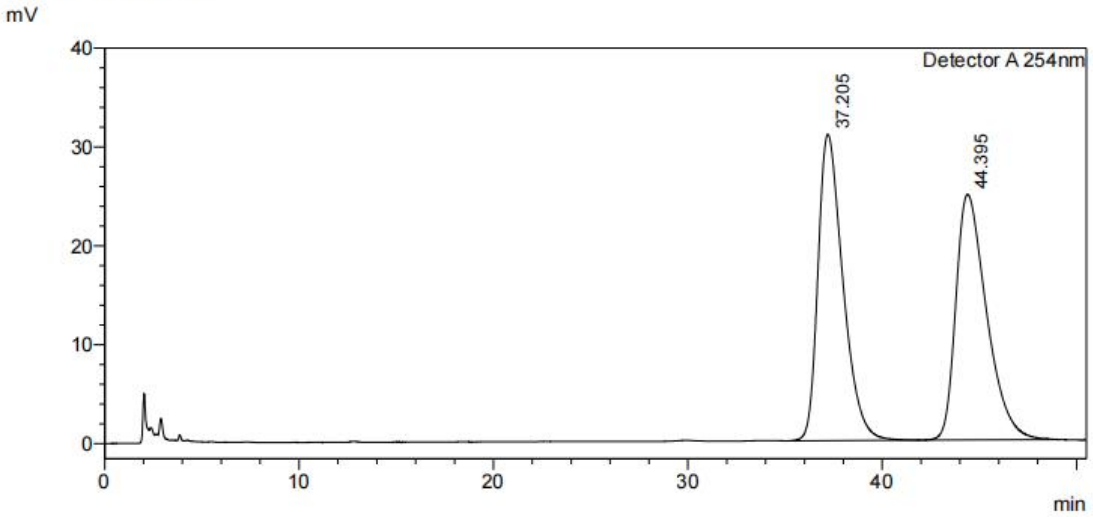
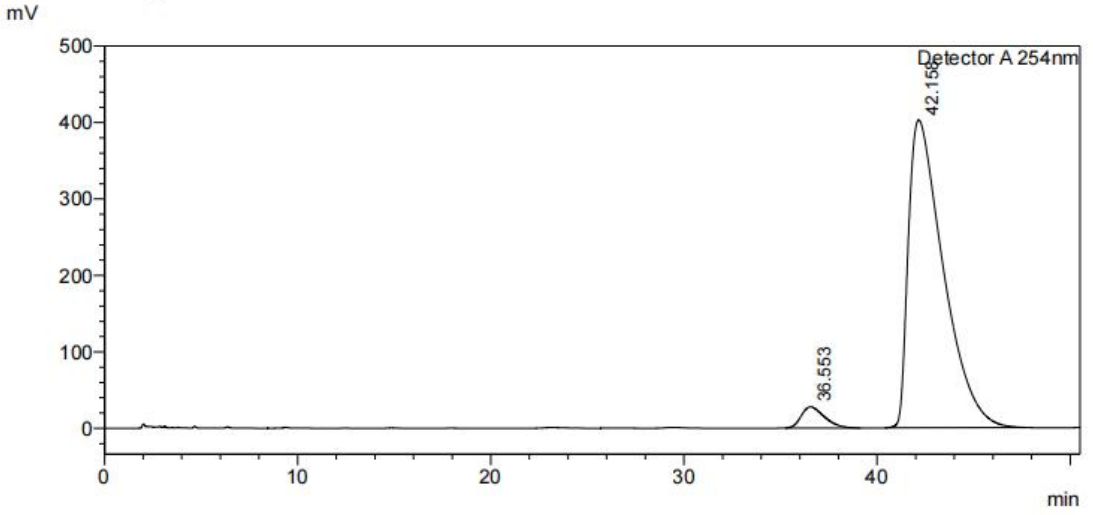


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	37.205	2766505	30980	50.303		M	
2	44.395	2733212	24843	49.697		M	
Total		5499718	55823				

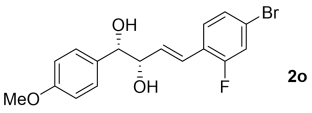
<Chromatogram>



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	36.553	2296773	27606	4.334		M	
2	42.158	50691666	402770	95.666		M	
Total		52988439	430376				



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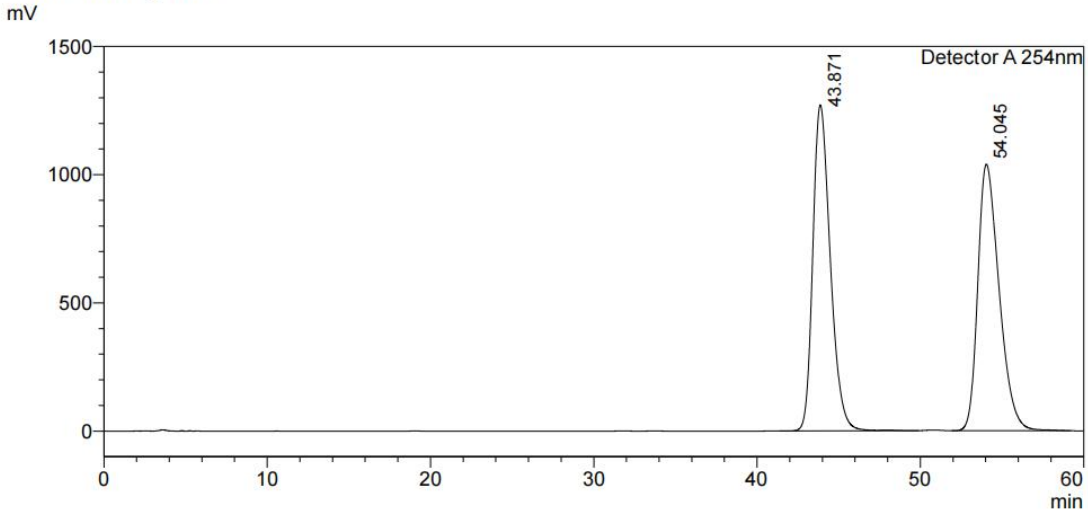

<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	43.871	94500221	1271045	50.000		M	
2	54.045	94500152	1038961	50.000		M	
Total		189000373	2310006				

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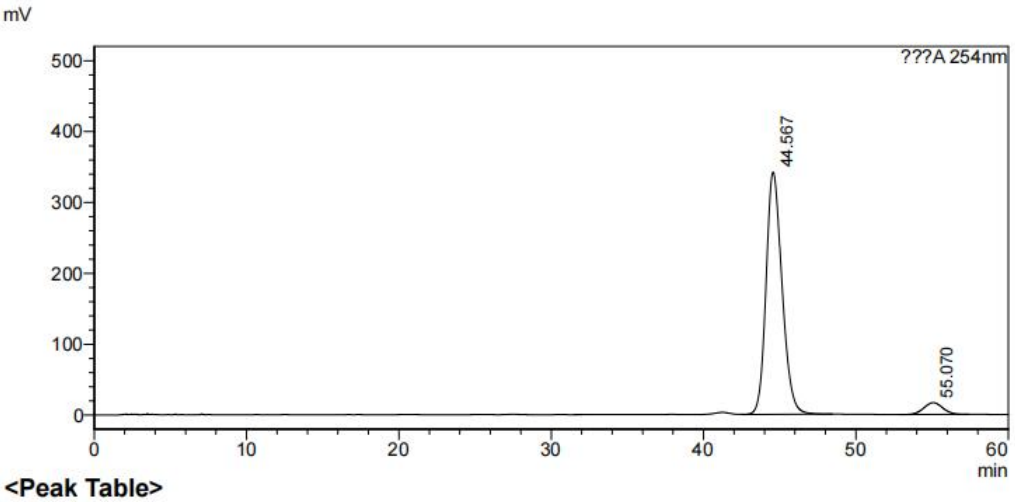
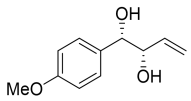


<Peak Table>

???A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	44.567	24922798	341776	94.523			
2	55.070	1444045	16702	5.477			
Total		26366842	358478				



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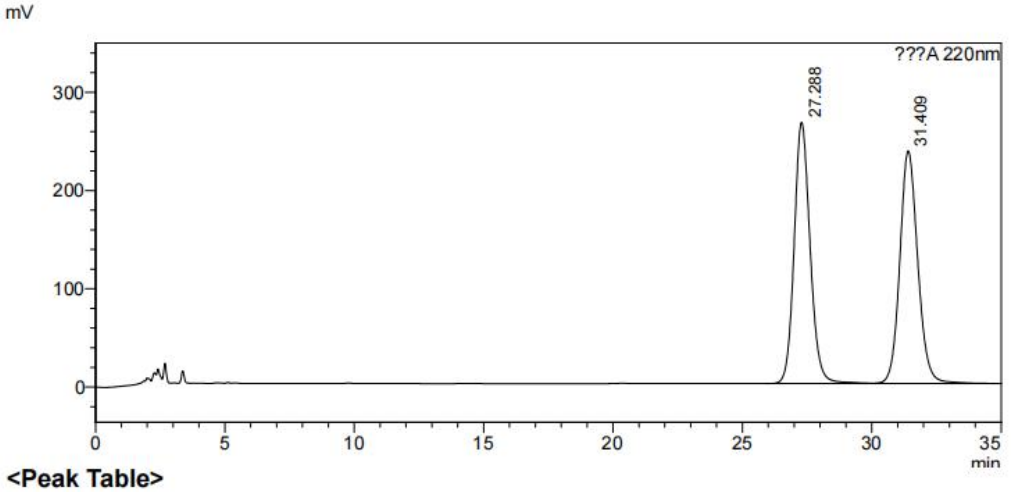
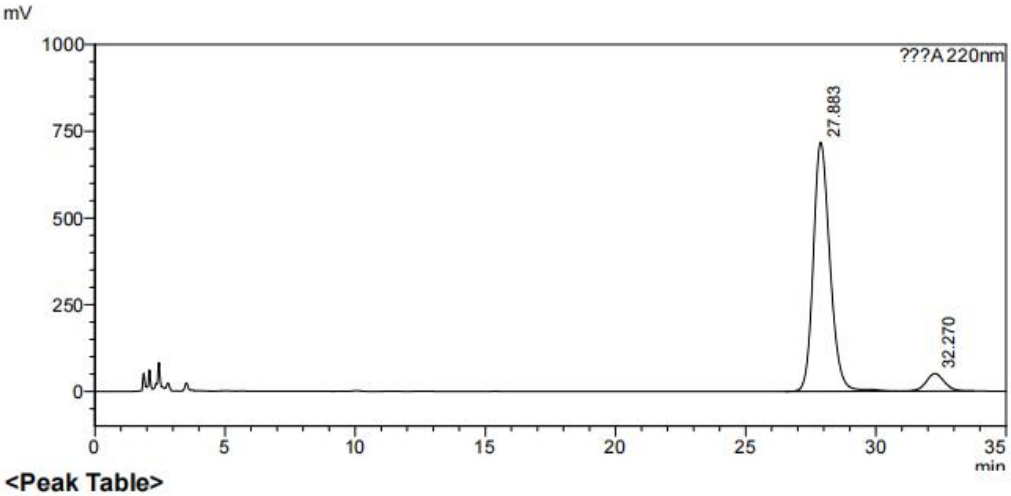


<Peak Table>

???A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	27.288	11414997	265863	49.948			
2	31.409	11438571	236767	50.052		V	
Total		22853568	502629				

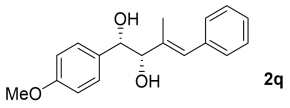
<Chromatogram>



<Peak Table>

???A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	27.883	31928833	718260	92.935		M	
2	32.270	2427221	50294	7.065			
Total		34356054	768554				



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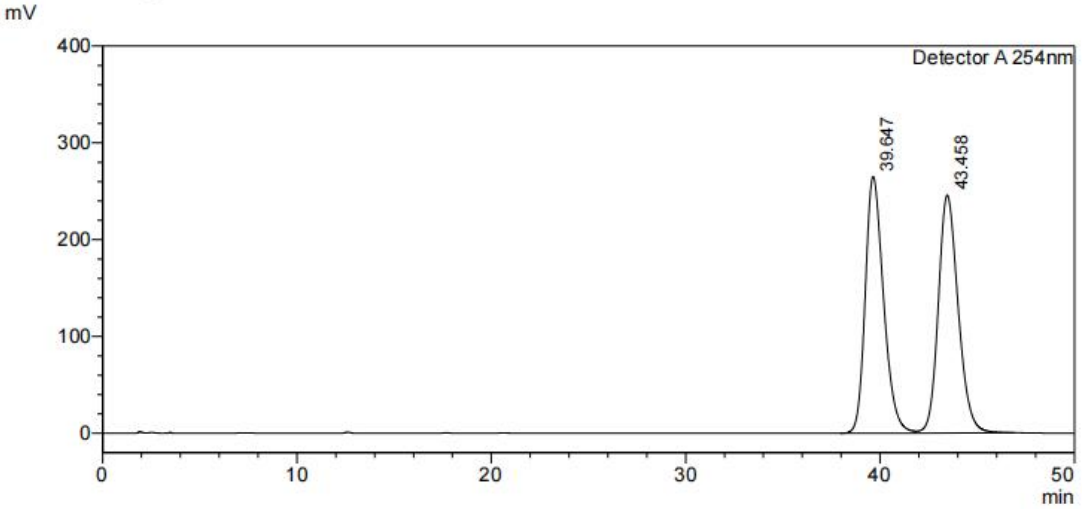
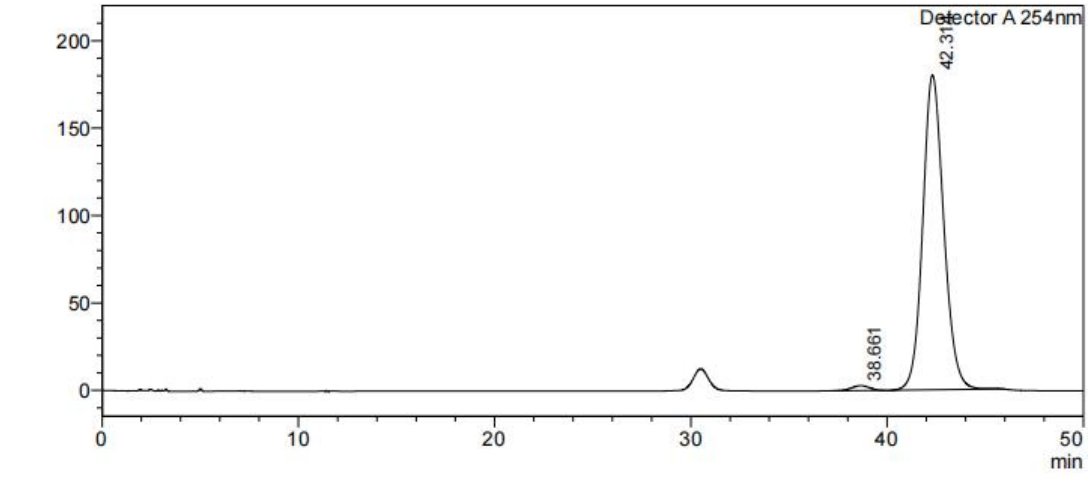


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	39.647	17396830	265288	49.846		M	
2	43.458	17504292	245889	50.154		M	
Total		34901122	511177				

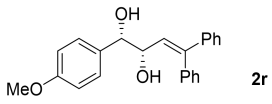
<Chromatogram>



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	38.661	182674	2852	1.343		M	
2	42.314	13421118	180089	98.657		M	
Total		13603792	182941				



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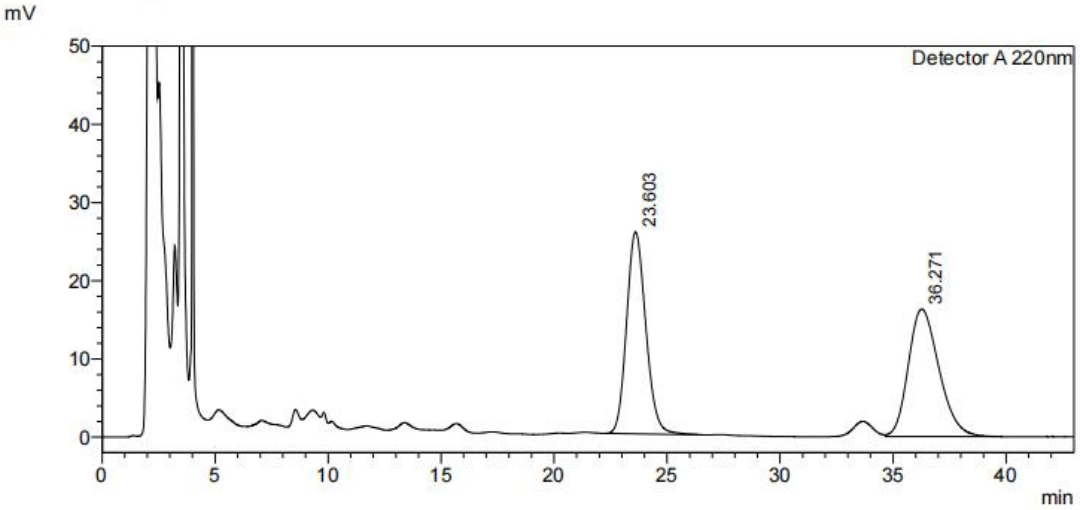
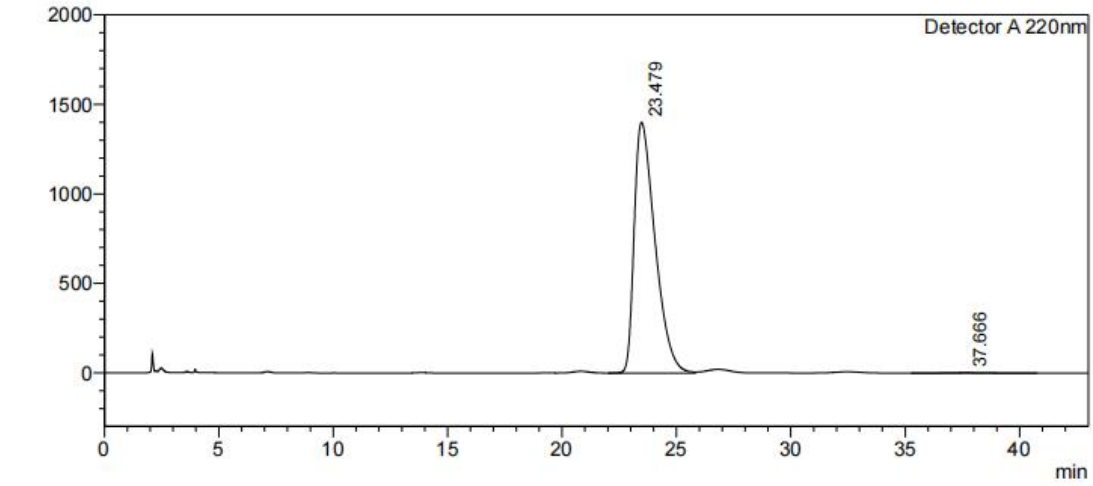


<Peak Table>

Detector A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	23.603	1531088	25811	49.856			
2	36.271	1539906	16308	50.144			
Total		3070994	42119				

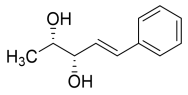
<Chromatogram>



<Peak Table>

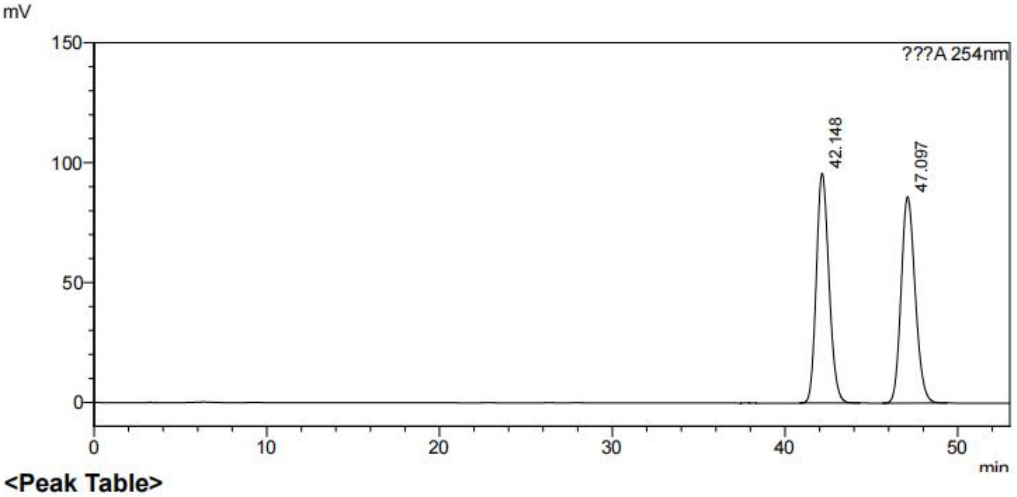
Detector A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	23.479	88674195	1401170	99.751			
2	37.666	221789	2182	0.249		M	
Total		88895984	1403351				



2s

<Chromatogram>

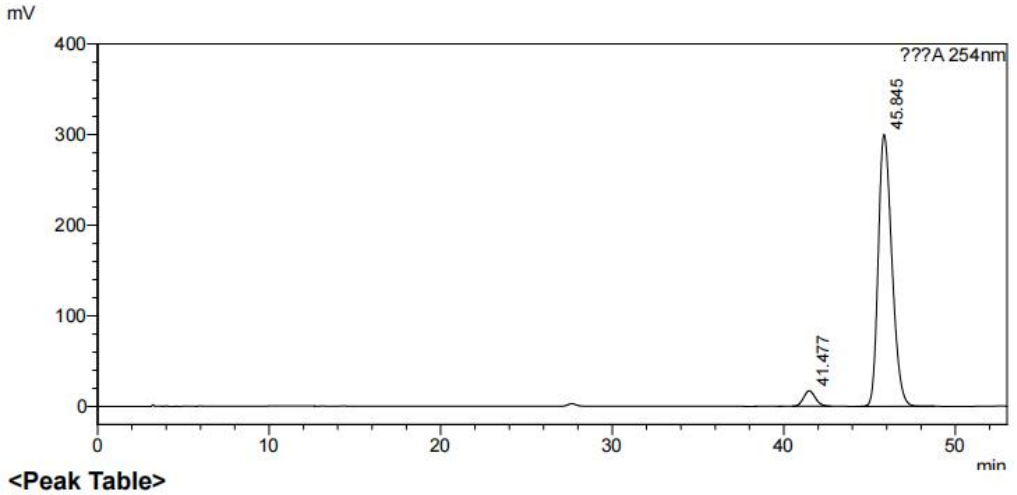


<Peak Table>

???A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	42.148	4880661	95886	50.012			
2	47.097	4878280	86088	49.988			
Total		9758941	181973				

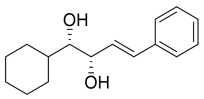
<Chromatogram>



<Peak Table>

???A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	41.477	807008	16943	4.643		M	
2	45.845	16574869	299913	95.357		M	
Total		17381877	316856				



2t

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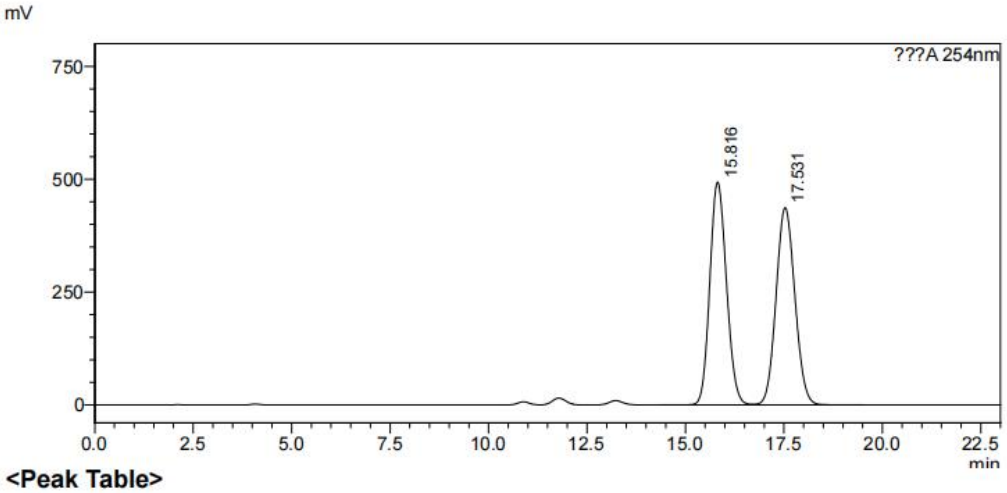
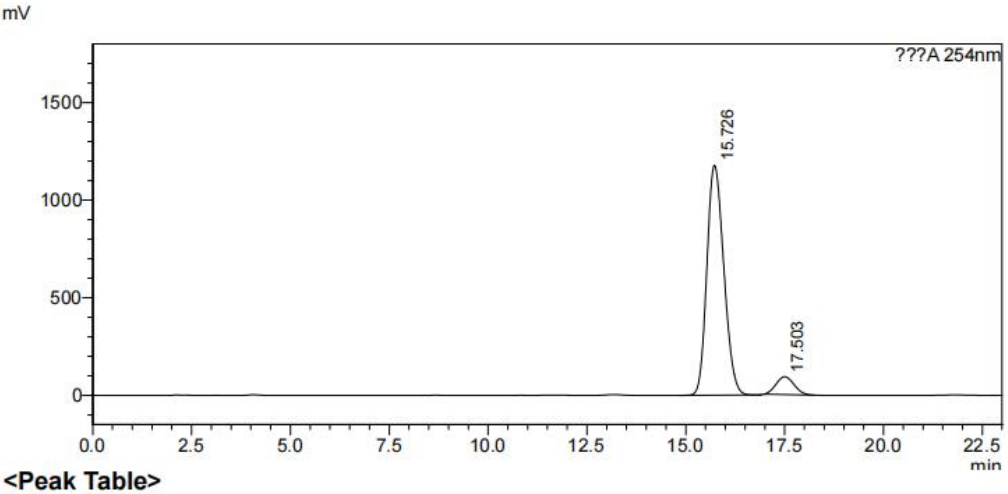


<Peak Table>

???A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	15.816	14512042	493613	49.931			
2	17.531	14552415	436979	50.069		V	
Total		29064458	930592				

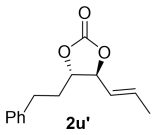
<Chromatogram>



<Peak Table>

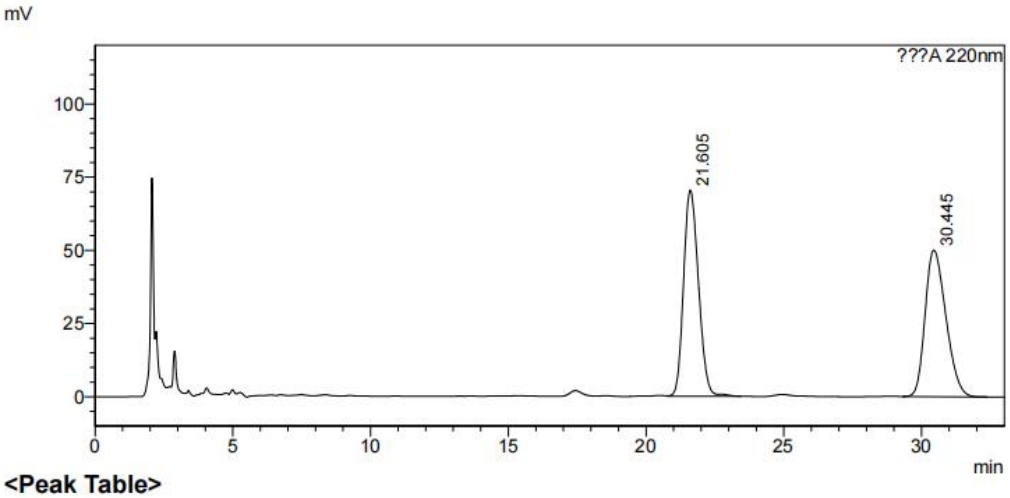
???A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	15.726	35071811	1177992	92.315		M	
2	17.503	2919826	91394	7.685		M	
Total		37991636	1269386				



2u'

<Chromatogram>

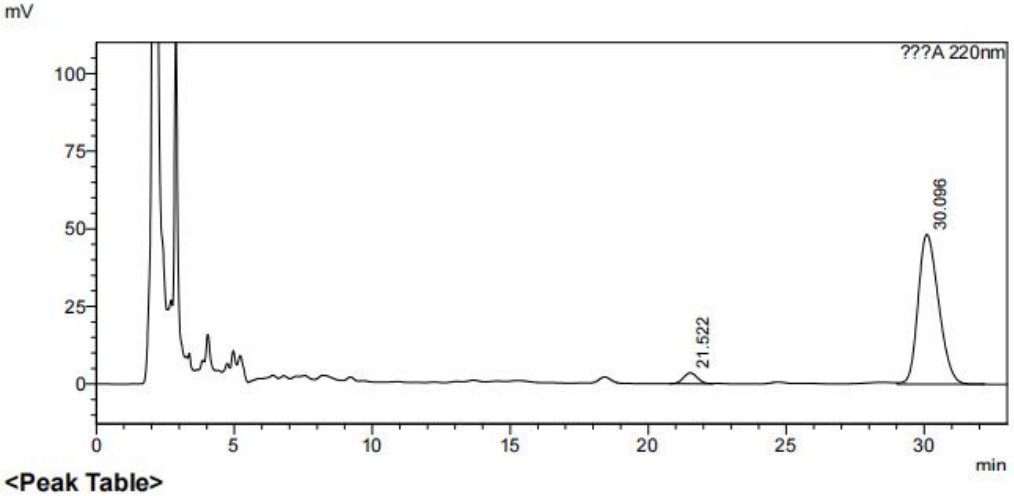


<Peak Table>

???A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	21.605	2646028	70337	50.206			
2	30.445	2624311	50066	49.794			
Total		5270339	120403				

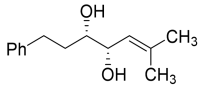
<Chromatogram>



<Peak Table>

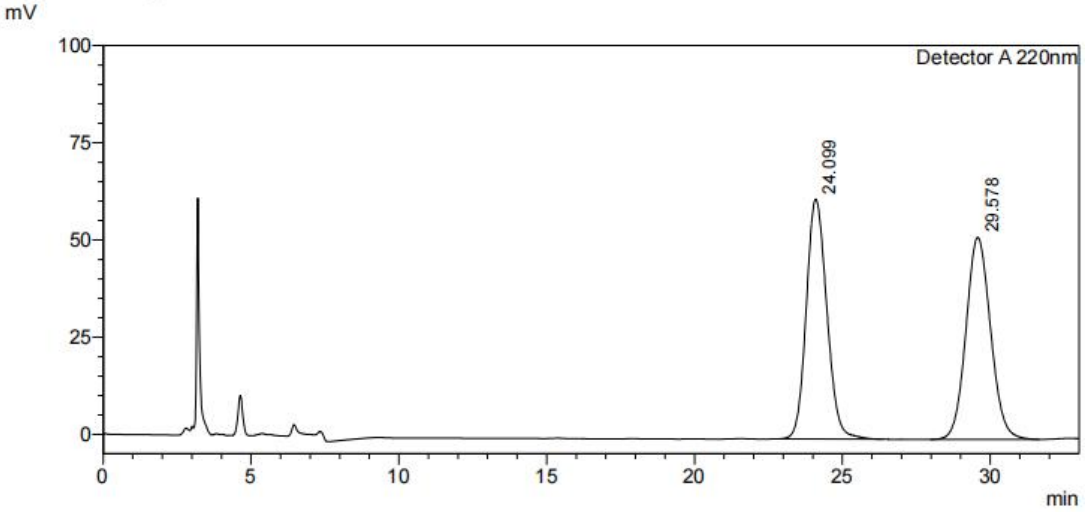
???A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	21.522	122606	3500	4.689			
2	30.096	2492177	48234	95.311			
Total		2614783	51734				



2v

<Chromatogram>

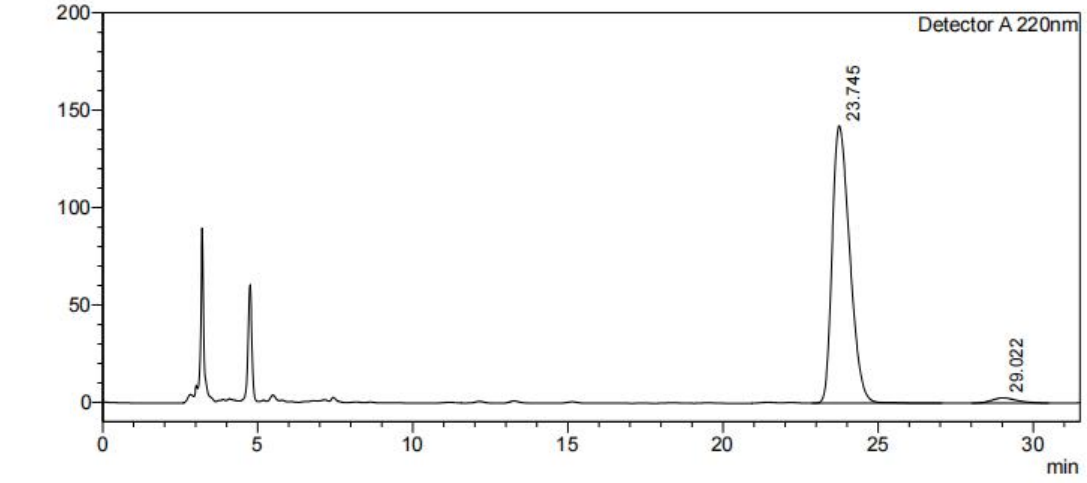


<Peak Table>

Detector A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	24.099	2992241	61664	49.847			
2	29.578	3010568	51981	50.153			
Total		6002809	113645				

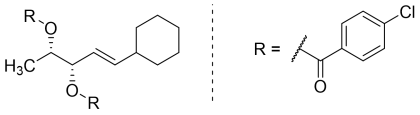
<Chromatogram>



<Peak Table>

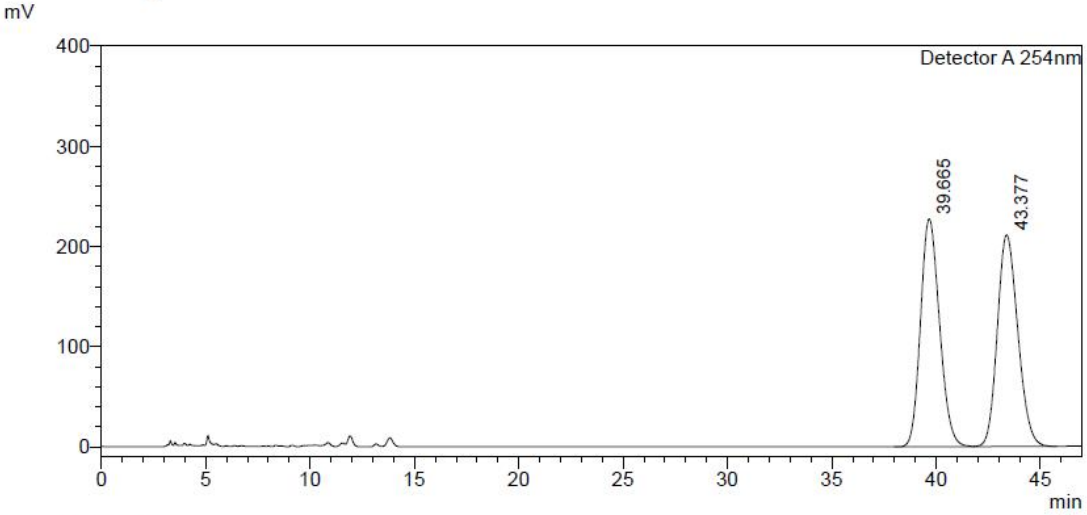
Detector A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	23.745	5606988	142473	97.359		M	
2	29.022	152091	2723	2.641		M	
Total		5759079	145196				



2w'

<Chromatogram>

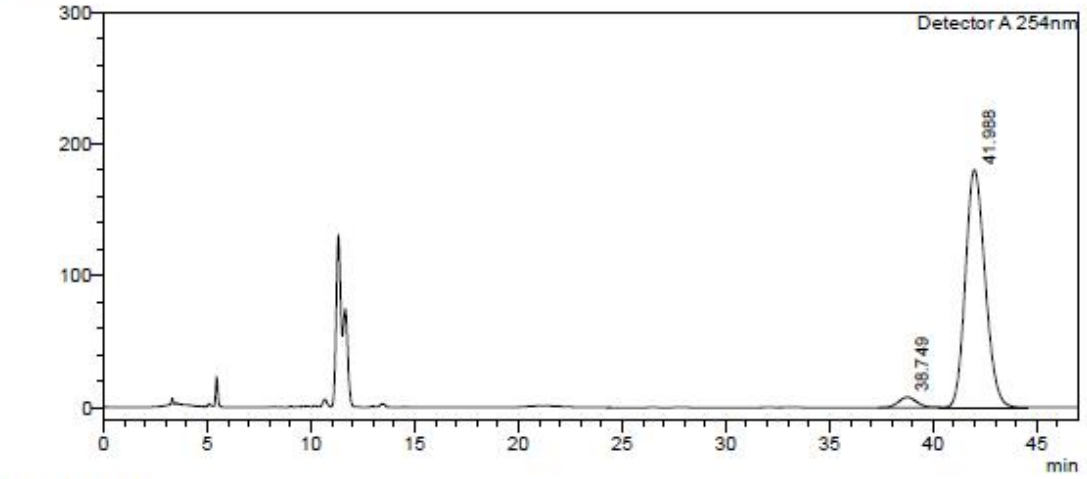


<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	39.665	14409068	227544	49.915			
2	43.377	14458234	211398	50.085		V	
Total		28867303	438942				

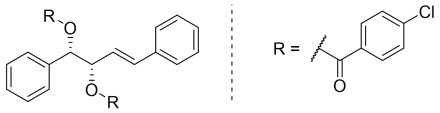
<Chromatogram>



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	38.749	482917	7904	3.896			
2	41.988	11913476	180649	96.104			
Total		12396393	188553				



5a

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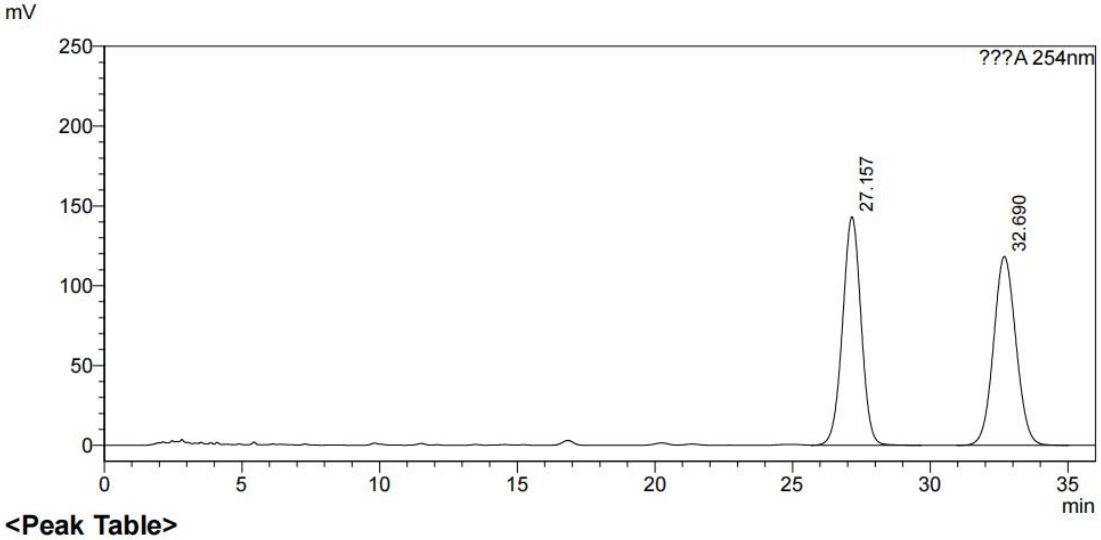
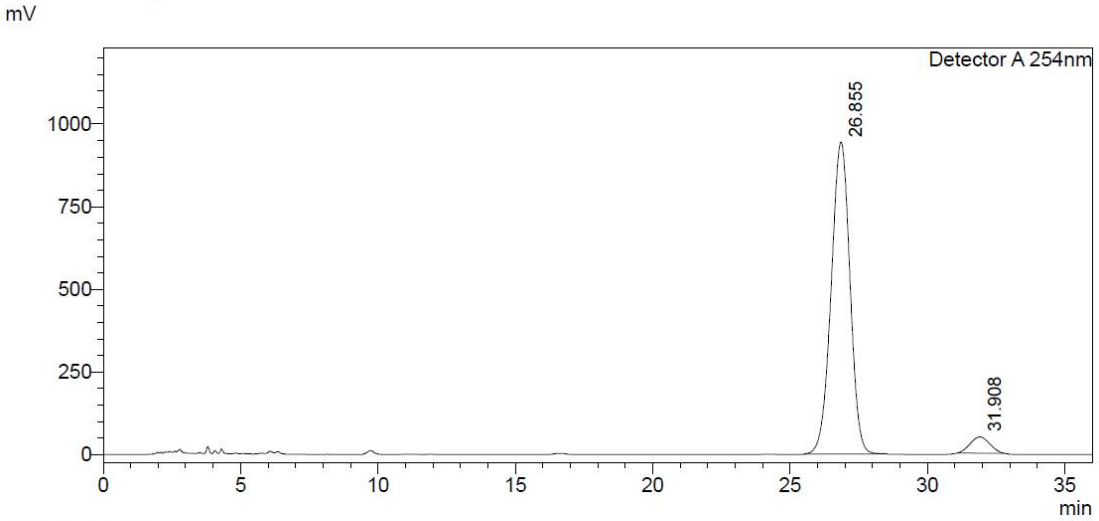


<Peak Table>

???A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	27.157	6712131	143189	50.034			
2	32.690	6702920	118370	49.966			
Total		13415051	261559				

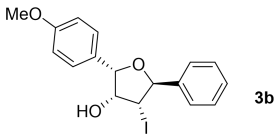
<Chromatogram>



<Peak Table>

Detector A 254nm

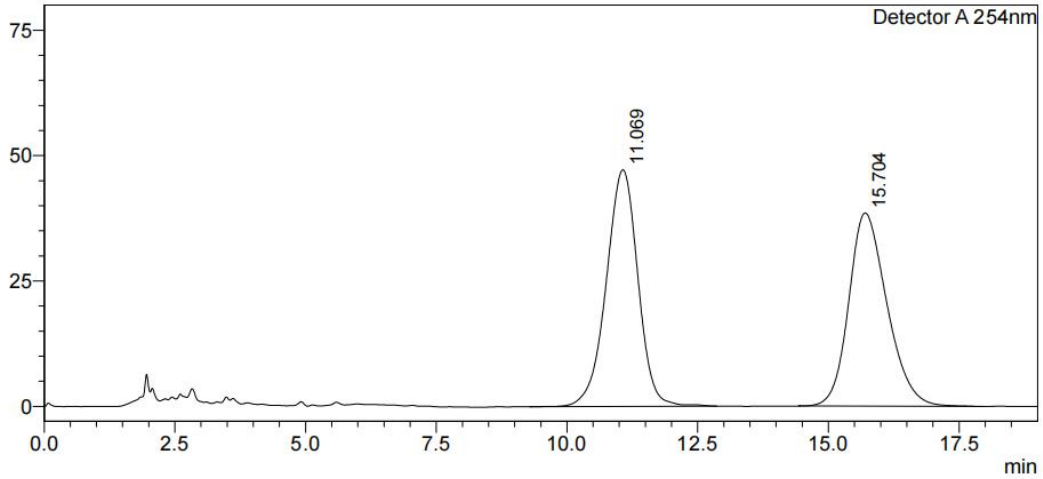
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	26.855	44836918	943877	94.820		M	
2	31.908	2449467	49182	5.180		M	
Total		47286385	993059				



3b

<Chromatogram>

mV



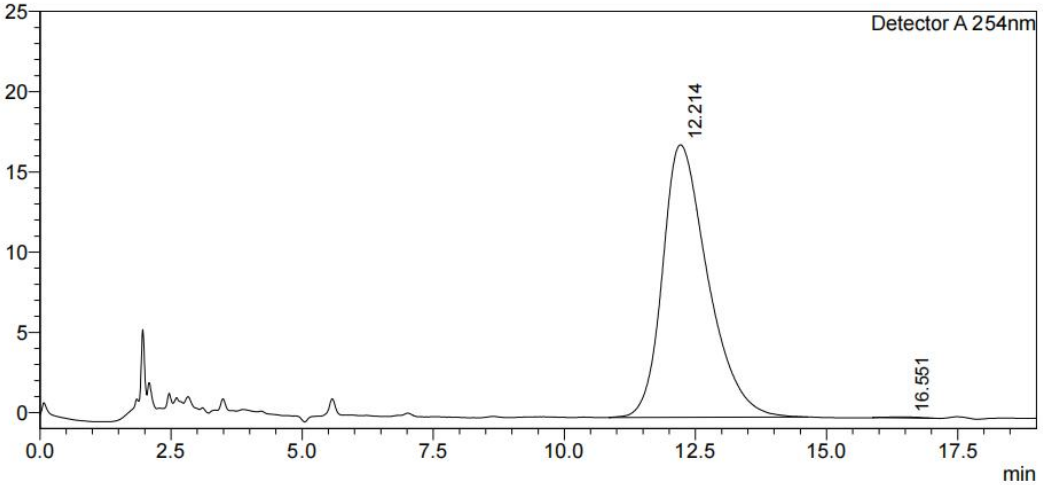
<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	11.069	1990165	47166	50.371		M	
2	15.704	1960812	38539	49.629		M	
Total		3950977	85705				

<Chromatogram>

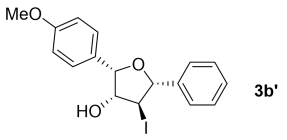
mV



<Peak Table>

Detector A 254nm

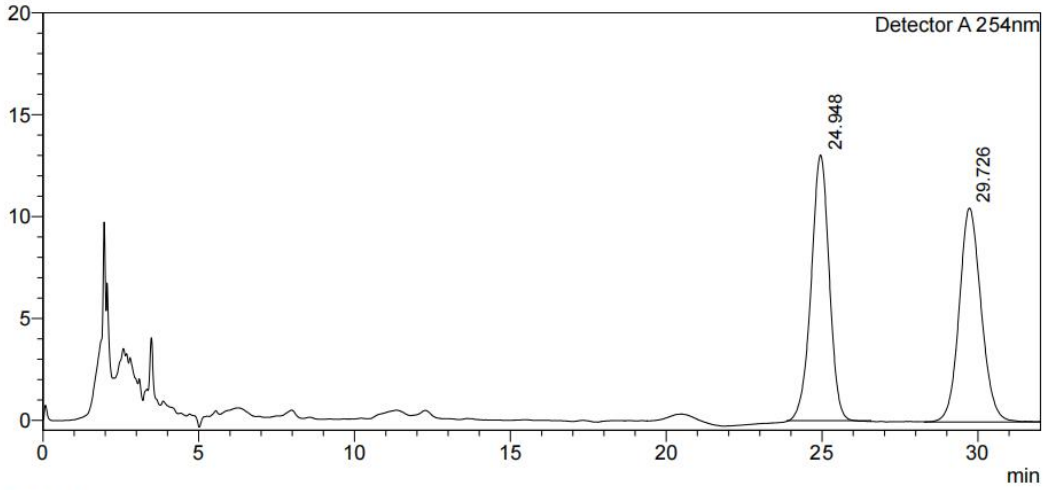
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	12.214	1022775	16981	99.709			
2	16.551	2986	74	0.291		M	
Total		1025761	17055				



3b'

<Chromatogram>

mV



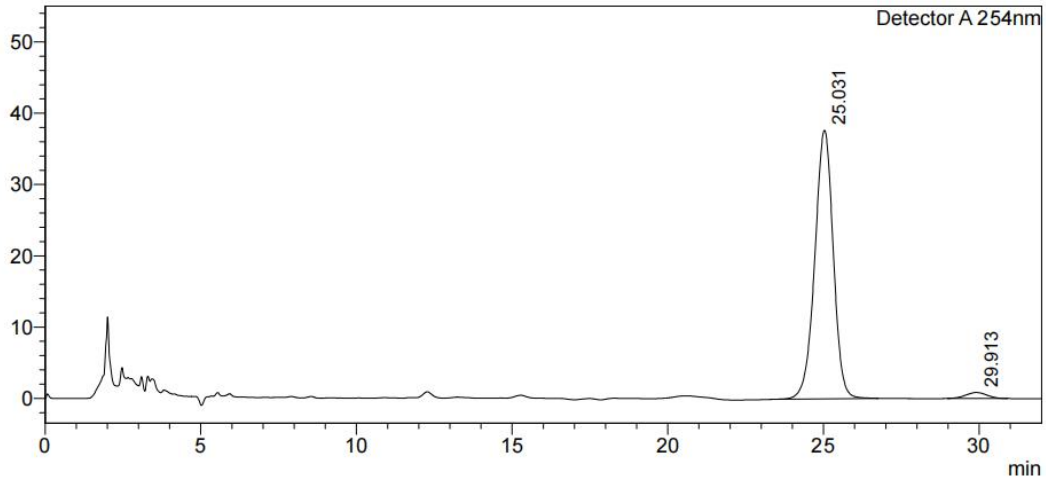
<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	24.948	525139	13053	50.488		M	
2	29.726	514983	10497	49.512		M	
Total		1040122	23550				

<Chromatogram>

mV



<Peak Table>

Detector A 254nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	25.031	1554973	37661	97.505		M	
2	29.913	39785	843	2.495		M	
Total		1594759	38503				