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Supporting Information

Copper-Catalyzed Enantioconvergent Cross-Coupling of Racemic Alkyl Bromides with Azole C(sp^2)–H Bonds

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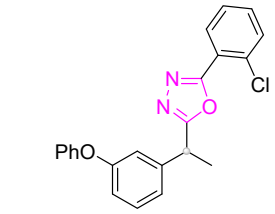
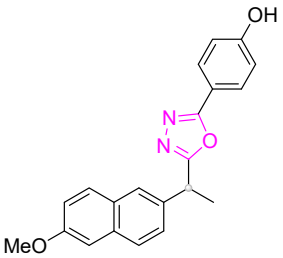


secretory phospholipase A_2 inhibitor



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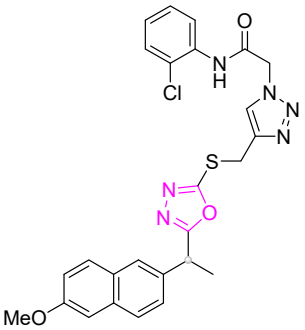

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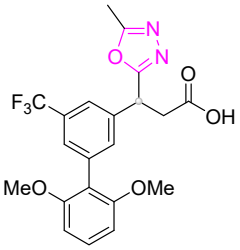
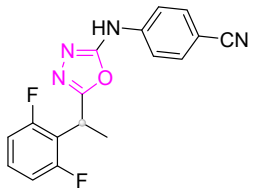
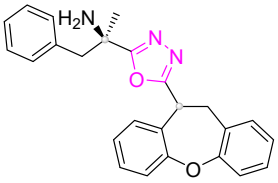


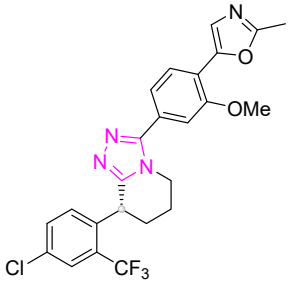
cathepsin A inhibitor



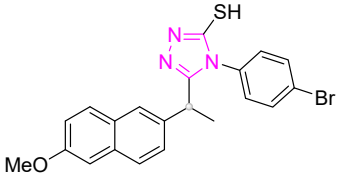
human immunodeficiency virus reverse transcriptase inhibitor



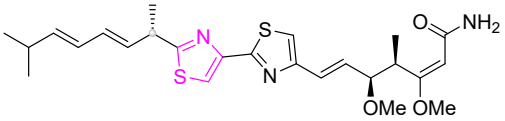
β-secretase inhibitor



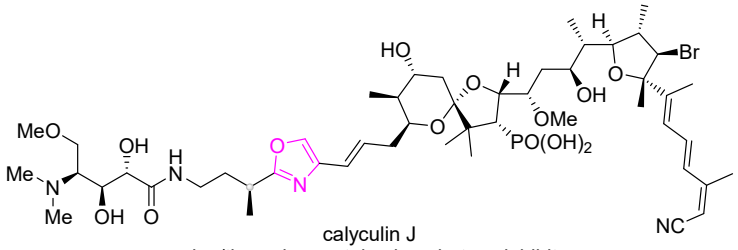
γ-secretase modulator



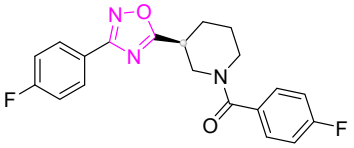
anti-inflammatory



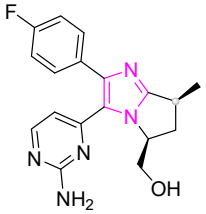
myxothiazol
mitochondrial cytochrome bc1 complex inhibitor



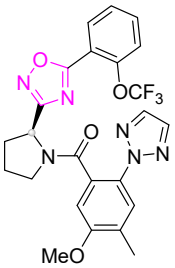
calyculin J
serine/threonine protein phosphatase inhibitor



ADX-47273
mGluR5 modulator

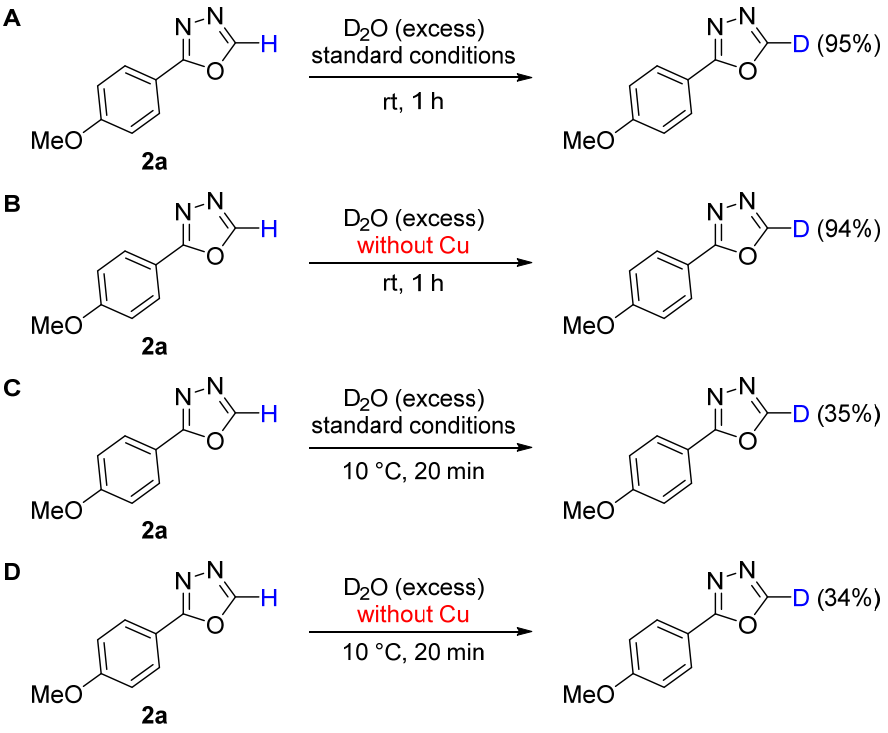


c-jun N-terminal kinase 3 (JNK3) inhibitor



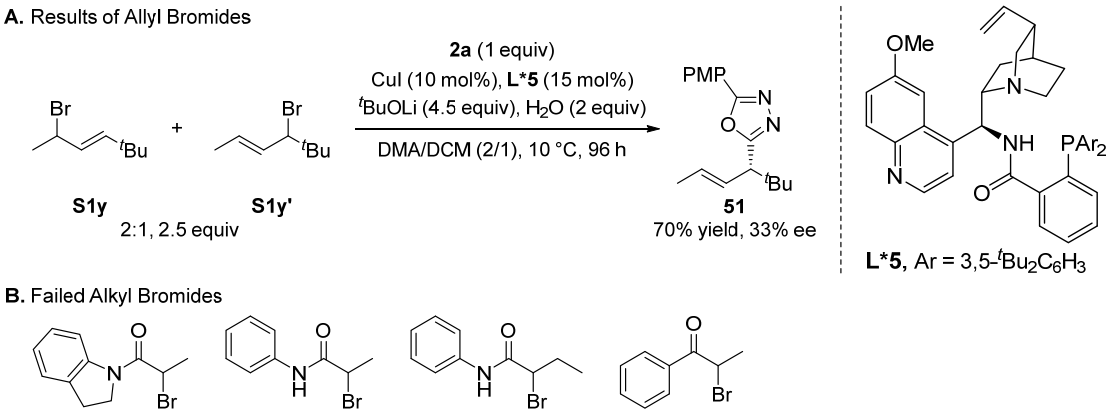
dual orexin receptor antagonist

Scheme S1. Selected bioactive α-chiral alkyl azoles.

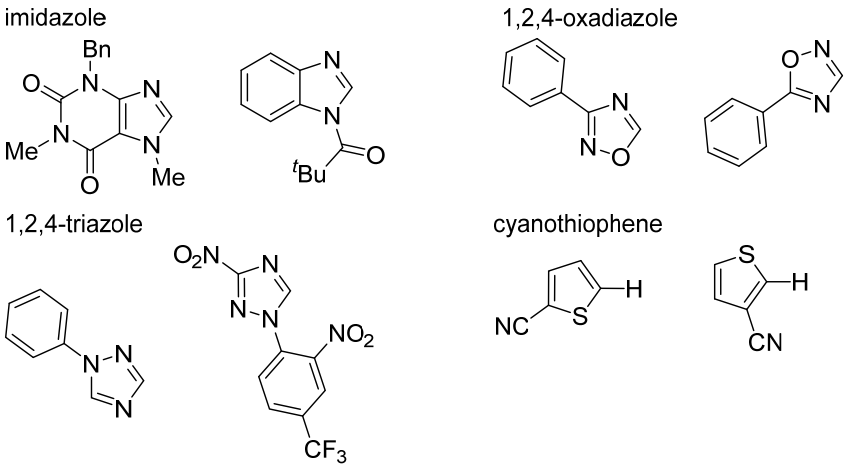


[a] Reaction conditions: **2a** (0.050 mmol), $CuBH_4(PPh_3)_2$ (0 or 10 mol%), **L*3** (12 mol%), D_2O (1 mL), and LiO^{t}Bu (4.5 equiv) in DMA/DCM (2/1, 0.60 mL) under argon. Deuterium incorporation percentage of **2a** was based on ^{1}H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard.

Scheme S2. H/D exchange experiments.[a]

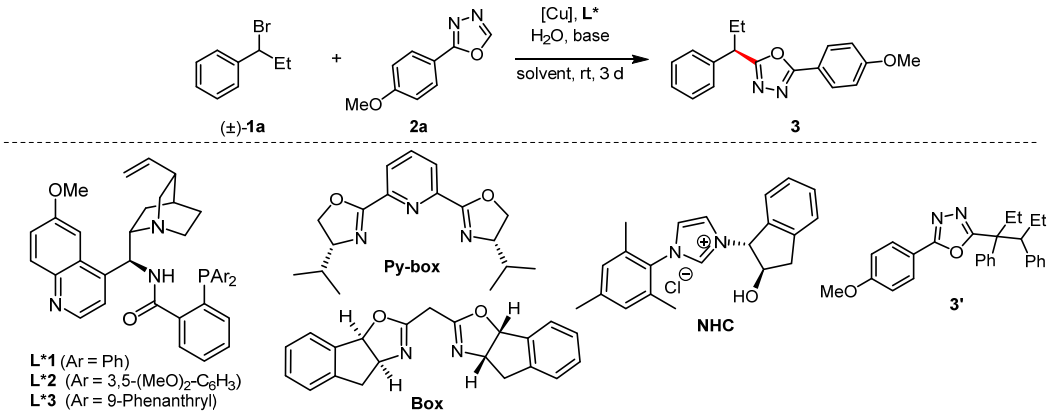


Scheme S3. Results of alkyl bromides other than benzylic ones.



Scheme S4. Failed heteroaromatic substrates.

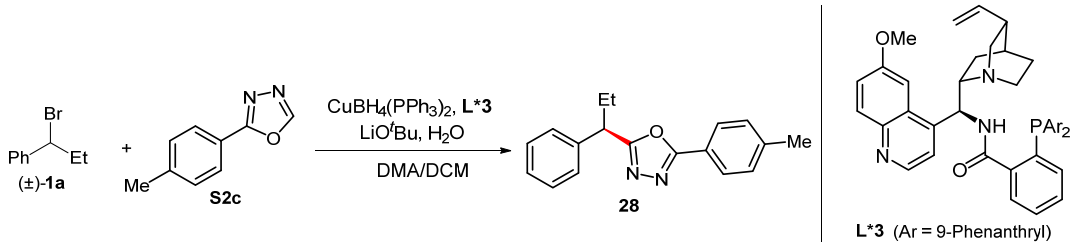
Table S1: Screening of reaction conditions.[a]



entry	solvent (ratio)	[Cu]	L*	base (equiv)	conversion (%)[c]	yield (%)[c]	ee (%)[d]
1[b]	DMA	$CuBH_4(PPh_3)_2$	**L*1**	LiO^{t}Bu (3)	72	24	49
2[b]	DCM	$CuBH_4(PPh_3)_2$	**L*1**	LiO^{t}Bu (3)	40	27	46
3[b]	$PhCF_3$	$CuBH_4(PPh_3)_2$	**L*1**	LiO^{t}Bu (3)	13	9	48
4	DMA	$CuBH_4(PPh_3)_2$	**L*1**	LiO^{t}Bu (3)	81	58	49
5	DMA:$PhCF_3$ (1:2)	$CuBH_4(PPh_3)_2$	**L*1**	LiO^{t}Bu (3)	40	29	49
6	DMA:DCM (1:2)	$CuBH_4(PPh_3)_2$	**L*1**	LiO^{t}Bu (3)	47	36	51
7	DMA:DCM (1:1)	$CuBH_4(PPh_3)_2$	**L*1**	LiO^{t}Bu (3)	58	57	50
8	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*1**	LiO^{t}Bu (3)	88	79	50
9	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*2**	LiO^{t}Bu (3)	85	74	75
10	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*3**	LiO^{t}Bu (3)	75	68	89
11	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*3**	LiO^{t}Bu (4.5)	84	71	89
12[b]	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*3**	LiO^{t}Bu (4.5)	85	55	89
13	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*3**	Cs_2CO_3 (4.5)	10	8	80
14	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*3**	K_3PO_4 (4.5)	2	ND	NA
15	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*3**	NaO^{t}Bu (4.5)	92	ND	NA
16	DMA:DCM (2:1)	$Cu(acac)_2$	**L*3**	LiO^{t}Bu (4.5)	75	65	88
17	DMA:DCM (2:1)	$Cu(PPh_3)_3CF_3$	**L*3**	LiO^{t}Bu (4.5)	76	67	88
18	DMA:DCM (2:1)	CuI	**L*3**	LiO^{t}Bu (4.5)	78	70	88
19[e]	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*3**	LiO^{t}Bu (4.5)	90	90	90
20[f]	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*3**	LiO^{t}Bu (4.5)	80	61	83
21[g]	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**L*3**	LiO^{t}Bu (4.5)	73	22	66
22[e]	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**Py-box**	LiO^{t}Bu (4.5)	25	10	5
23[e]	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**Box**	LiO^{t}Bu (4.5)	25	11	56
24[e]	DMA:DCM (2:1)	$CuBH_4(PPh_3)_2$	**NHC**	LiO^{t}Bu (4.5)	28	7	5

[a] Reaction conditions: (±)-**1a** (2.0 equiv), **2a** (0.050 mmol), $CuBH_4(PPh_3)_2$ (10 mol%), **L*** (12 mol%), H_2O (0 or 2.0 equiv) and base (3.0 or 4.5 equiv) in solvent (0.60 mL) at room temperature for 3 d under argon. [b] Without H_2O. [c] Conversion of **2a** and yield of **3** were based on ^{1}H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. [d] Ee value was based on HPLC analysis. [e] At 10 °C. [f] At 40 °C. Side product **3′** was detected in 18% yield based on ^{1}H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. [g] At 50 °C. Side product **3′** was detected in 28% yield based on ^{1}H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. ND = not detected; NA = not applicable.

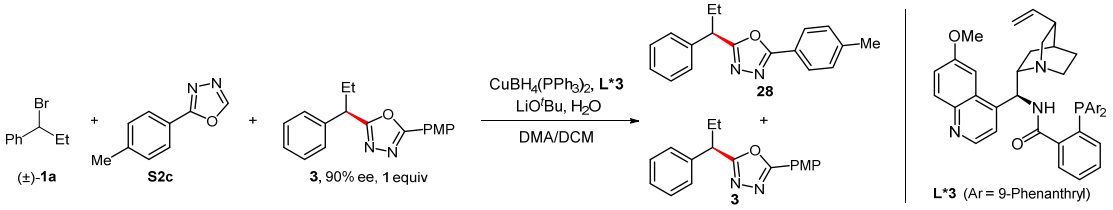
Table S2: Temperature effect on product enantioselectivity.[a]



entry	temperature	time	yield (%)[b]	ee (%)[c]
1	10 °C	72 h	61	90
2	30 °C	12 h	50	87
3	40 °C	12 h	51	85
4	60 °C	12 h	18	74

[a] Reaction conditions: (±)-**1a** (2.0 equiv), **S2c** (0.050 mmol), $CuBH_4(PPh_3)_2$ (10 mol%), **L*3** (12 mol%), H_2O (2.0 equiv), and LiO^tBu (4.5 equiv) in DMA/DCM (2/1, 0.60 mL) under argon. [b] Yield of **28** was based on ^1H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. [c] Ee value was based on HPLC analysis.

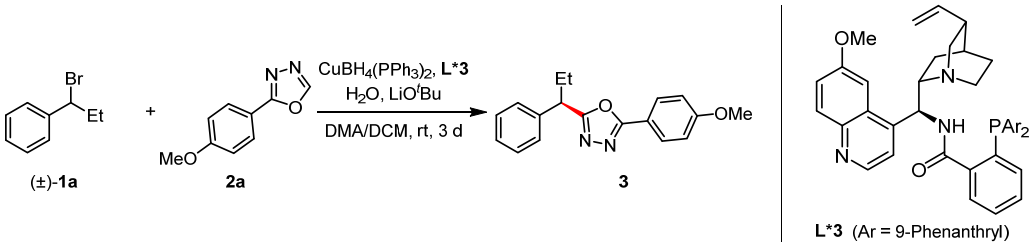
Table S3: Temperature effect on product racemization.[a]



entry	temperature	time	yield of **28** (%)[b]	ee of **28**(%)[c]	recovered yield of **3** (%)[b]	ee of **3** (%)[c]
1	10 °C	72 h	54	90	98	89
2	30 °C	12 h	38	87	98	89
3	40 °C	12 h	38	85	98	89
4	60 °C	12 h	11	49	66	59

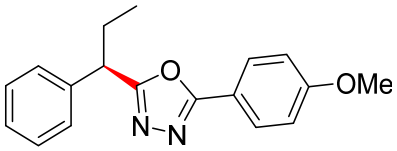
[a] Reaction conditions: (±)-**1a** (2.0 equiv), **S2c** (0.050 mmol), $CuBH_4(PPh_3)_2$ (10 mol%), **L*3** (12 mol%), H_2O (2.0 equiv) and LiO^tBu (4.5 equiv) in DMA/DCM (2/1, 0.60 mL) under argon. [b] Yield of **28** and the recovered yield of **3** were based on ^1H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. [c] Ee value was based on HPLC analysis.

Table S4: Control experiments.[a]



entry	$CuBH_4(PPh_3)_2$	**L*3**	LiO^{t}Bu	yield (%)[b]
1	10 mol%	0 mol%	4.5 equiv	ND
2	0 mol%	12 mol%	4.5 equiv	ND
3	0 mol%	0 mol%	4.5 equiv	ND
4	10 mol%	12 mol%	0 equiv	ND

[a] Reactions were performed according to general procedure as described for the synthesis of **3** unless otherwise specified in Table **S4**. [b] Yield of **3** was based on ^{1}H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. ND = not detected.



3

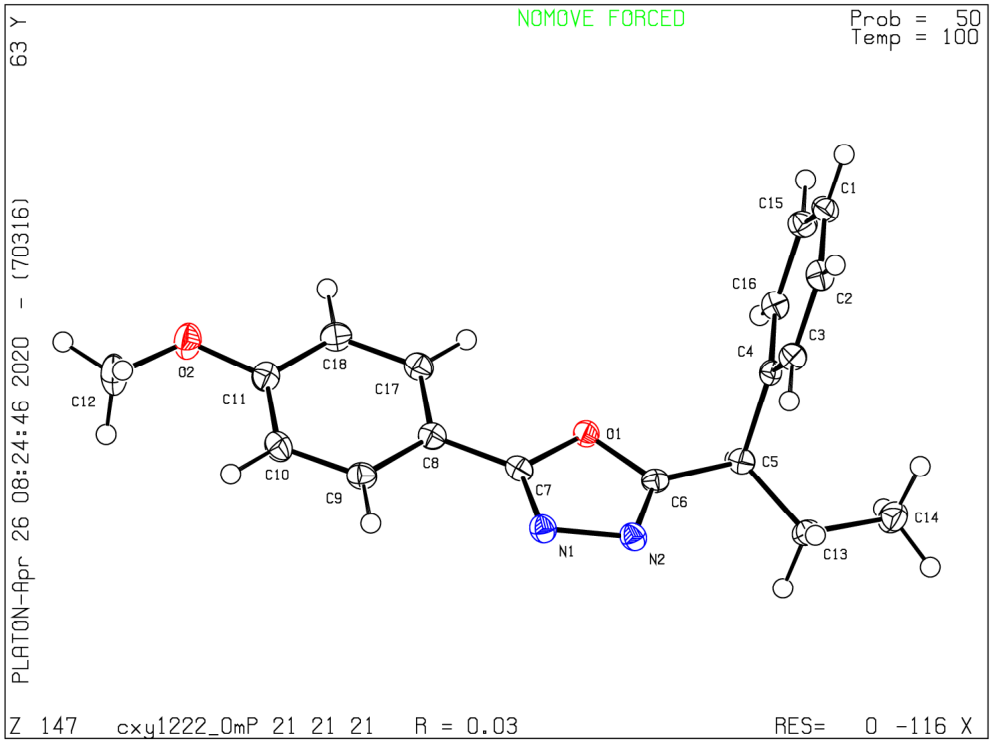


Figure S1. The X ray structure of **3**.

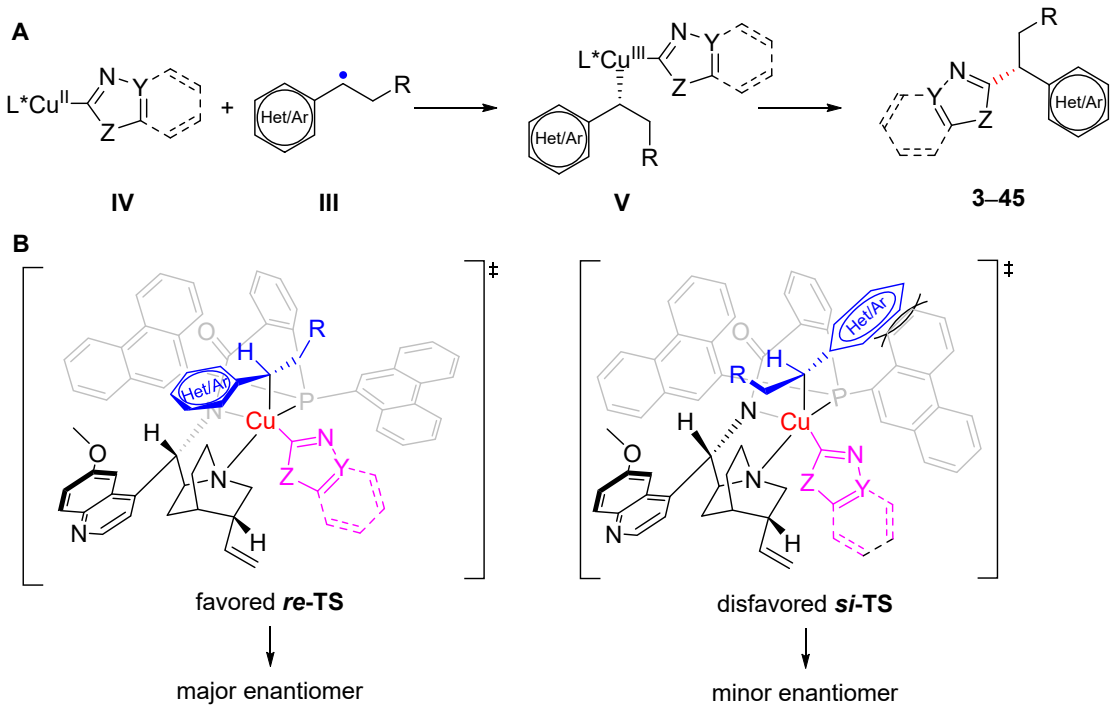


Figure S2. The stereodiscriminative model.

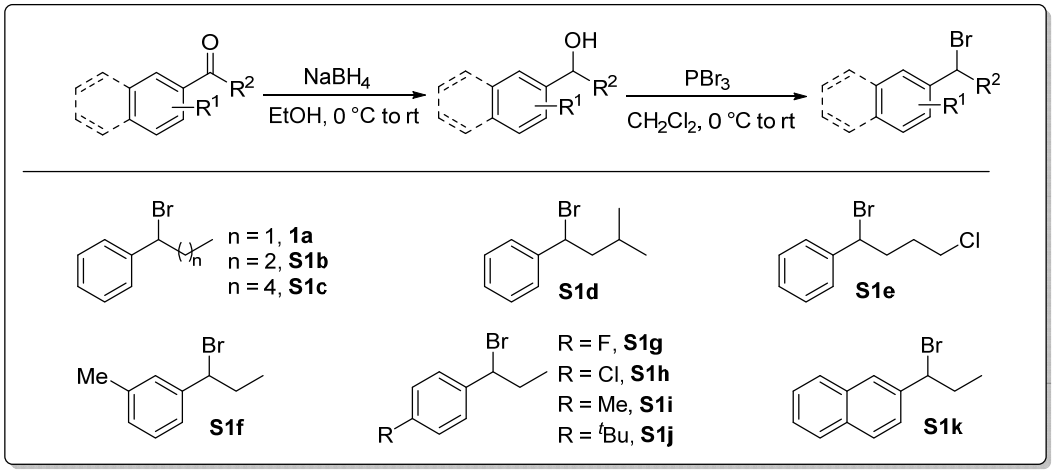
General information

All reactions were carried out under argon atmosphere using Schlenk techniques. Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. *N,N*-Dimethylacetamide (DMA) was purchased from Aladdin, which was redistilled with calcium hydride under argon atmosphere. Anhydrous dichloromethane (DCM) and (trifluoromethyl)benzene were purchased from JK and transferred under an argon atmosphere. LiO^{t}Bu (98%) was purchased from JK. $CuBH_4(PPh_3)_2$, $Cu(^i PrCO_2)_2$, CuI and $Cu(acac)_2$ were purchased from TCL. Analytical thin layer chromatography (TLC) was performed on precoated silica gel 60 GF254 plates. Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040 – 0.063 mm). Visualization on TLC was achieved by use of UV light (254 nm) or iodine. NMR spectra were recorded on Bruker DPX-400 spectrometer at 400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR, 376 MHz for ^{19}F NMR, respectively, in $CDCl_3$ with tetramethylsilane (TMS) as internal standard. The chemical shifts are expressed in ppm and coupling constants are given in Hz. Data for ^{1}H NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quarter; p, pentet; m, multiplet; br, broad), coupling constant (Hz), integration. Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm). Mass spectrometric data were obtained using Bruker Apex IV RTMS. Enantiomeric excess (ee) was determined using SHIMADZU LC-20AD with SPD-20AV detector (at an appropriate wavelength). Column conditions are reported in the experimental section below.

Preparation of substrates

The alkyl bromides **1a** and **S1b**–**S1x** were synthesized According to the literature procedures.[1–3] All the characterization data are consistent with those in the reported literature.

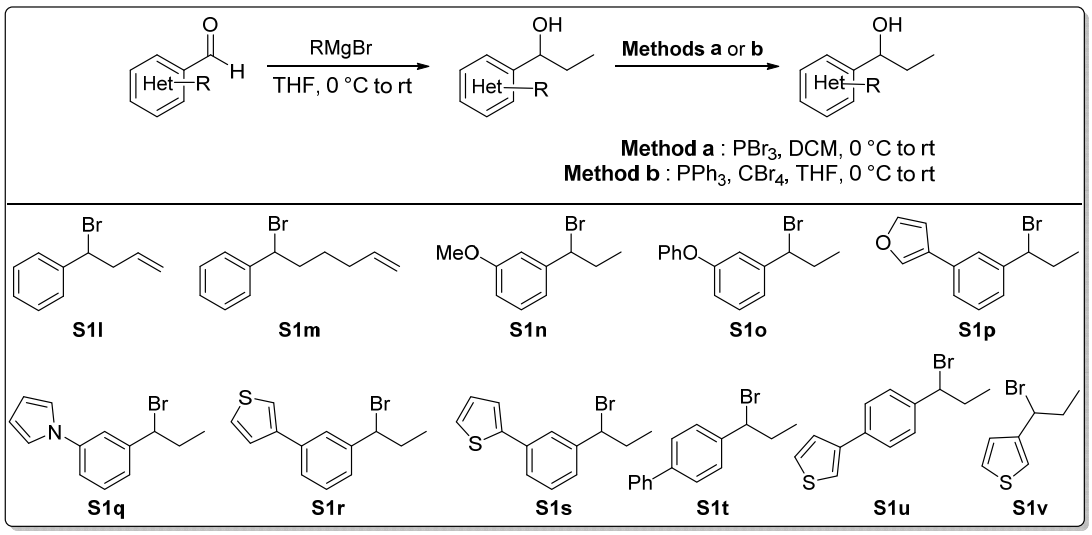
General procedure for the synthesis of alkyl bromides 1a and S1b–S1k:



To a solution of ketone (3.0 mmol, 1.0 equiv) in EtOH (9.0 mL) was added $NaBH_4$ (136.2 mg, 3.6 mmol, 1.2 equiv) at 0 °C and the reaction mixture was stirred at room temperature for 0.5–2 h. After completion of reaction (monitored by TLC), the reaction was quenched by water, diluted with CH_2Cl_2, and extracted with CH_2Cl_2 three times. The combined organic layer was washed by brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure to afford the corresponding alcohol, which was directly used in the next step without further purification.

To a solution of the residue obtained above in CH_2Cl_2 (9.0 mL) was added PBr_3 (0.20 mL, 2.1 mmol, 0.70 equiv) under argon atmosphere at 0 °C and the resulting reaction mixture was stirred at room temperature. After completion of reaction (monitored by TLC), the mixture was quenched by water and extracted with CH_2Cl_2 three times. The combined organic phase was washed by brine, filtered by a pad of silica gel, and concentrated under reduced pressure to afford the corresponding crude alkyl bromide, which was directly used in the next step without further purification or stored in refrigerator (The product readily decomposed in air or on silica gel).

General procedure for the synthesis of alkyl bromides S1l–S1v:

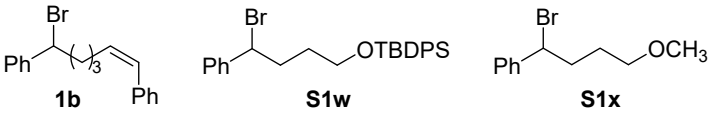


To a solution of aldehyde in anhydrous THF (2.0 mL/mmol aldehyde) was slowly added alkyl-magnesium bromide (1.0 M in THF, 1.2 equiv) at 0 °C under argon atmosphere. The reaction mixture was stirred for another 2 h. And then, the reaction mixture was warmed up to room temperature and stirred until the aldehyde was completely consumed (monitored by TLC). The reaction was quenched by saturated aqueous NH_4Cl, and extracted with ethyl acetate three times. The combined organic phase was dried over Na_2SO_4, filtered, concentrated under reduced pressure to afford the desired alcohol, which was directly used in the next step without further purification.

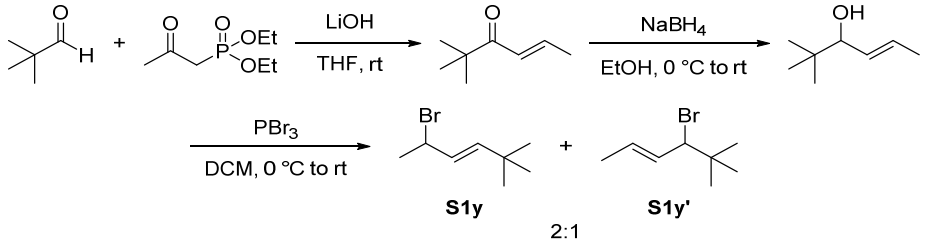
Method a: To a solution of the crude alcohol obtained above in CH_2Cl_2 (2.0 mL/mmol alcohol) was added PBr_3 (0.70 equiv) with vigorous stirring at 0 °C and the resulting reaction mixture was stirred at room temperature. After completion of reaction (monitored by TLC), the mixture was quenched by water and extracted with CH_2Cl_2 three times. The combined organic phase was washed by brine, dried over Na_2SO_4, filtered through a silica gel pad, and concentrated under reduced pressure to afford the corresponding crude alkyl bromide product, which was directly used in the next step without further purification or stored in a refrigerator unless otherwise noted. (The product usually readily decomposed in air and on silica gel.)

Method b: To a solution of the crude alcohol obtained above and triphenylphosphine (1.8 equiv) in THF (2.0 mL/mmol alcohol) was added carbon tetrabromide (1.5 equiv) in one portion at 0 °C under argon atmosphere and the resulting reaction mixture was stirred at 0 °C for 5 min. Then, the mixture was allowed to warm up to room temperature and stirred for another 2 h. The precipitate was filtered off through a pad of celite and washed by cold THF. The solution was concentrated under reduced pressure and the residues was purified by column chromatography on silica gel to provide the corresponding bromo compound.

Alkyl bromides **1b, S1w** and **S1x** were prepared according to literature procedures.[1]



Synthesis of **S1y** and **S1y'**



To a solution of pivalaldehyde (861.3 mg, 10.0 mmol, 1.0 equiv) in THF (20 mL) were sequentially added lithium hydroxide (239.5 mg, 12.0 mmol, 1.2 equiv) and diethyl (2-oxopropyl)phosphonate (2.04 g, 10.5 mmol, 1.05 equiv). The reaction was stirred for 15 h, and then concentrated in vacuo. The residue was dissolved in EtOAc, and washed twice with 1 M aqueous hydrochloric acid and twice with saturated aqueous sodium bicarbonate solution. The combined organic phases were dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The (*E*)-2,2-dimethylhex-4-en-3-one was isolated after flash column chromatography.

To a solution of (*E*)-2,2-dimethylhex-4-en-3-one (378.6 mg, 3.0 mmol, 1.0 equiv) in EtOH (9.0 mL) was added $NaBH_4$ (136.2 mg, 3.6 mmol, 1.2 equiv) at 0 °C and the reaction mixture was stirred at room temperature for 0.5–2 h. After completion of reaction (monitored by TLC), the reaction was quenched by water, diluted with CH_2Cl_2, and extracted with CH_2Cl_2 three times. The combined organic layer was washed by brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure to afford the corresponding alcohol, which was directly used in the next step without further purification.

To a solution of the residue obtained above in CH_2Cl_2 (9.0 mL) was added PBr_3 (0.20 mL, 2.1 mmol, 0.70 equiv) under argon atmosphere at 0 °C and the resulting reaction mixture was stirred at room temperature. After completion of reaction (monitored by TLC), the mixture was quenched by water and extracted with CH_2Cl_2 three times. The combined organic phase was washed by brine, filtered by a pad of silica gel, and concentrated under reduced pressure to afford the corresponding crude alkyl bromide **S1y** and **S1y'** (2:1, 328 mg, 57% crude yield), which was directly used in the next step without further purification.

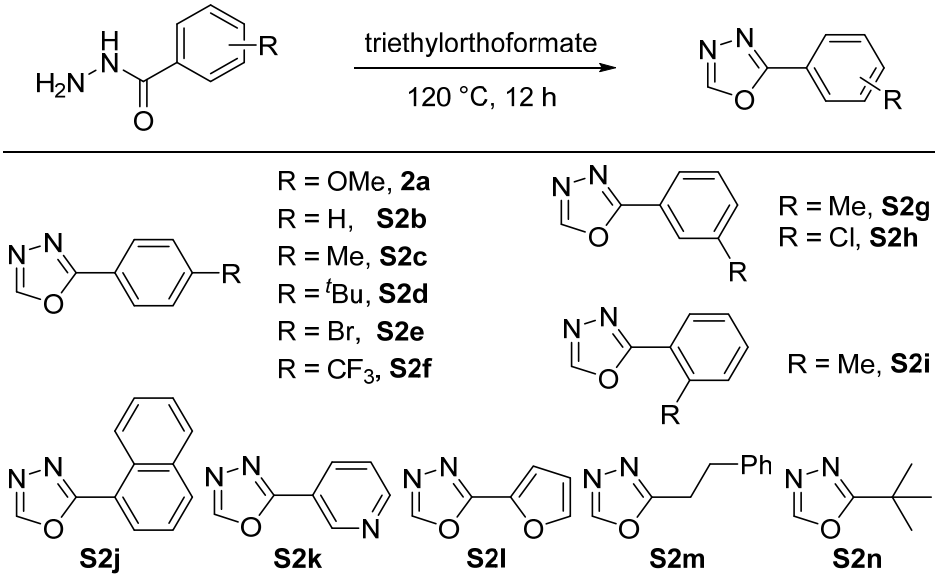
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.80 – 5.60 (m, 2H(major)+1H(minor)), 4.72 (dq, *J* = 7.9, 6.7 Hz, 1H) (major), 4.42 (d, *J* = 10.1 Hz, 0.5H) (minor), 1.79 (d, *J* = 6.7 Hz, 3H) (major), 1.74 (dd, *J* = 6.3, 1.4 Hz, 1.5H) (minor), 1.05 (s, 4.5H) (minor), 1.03 (s, 9H) (major).

^{13}C NMR (100 MHz, $CDCl_3$) δ 142.9 (major), 130.4 (minor), 128.8 (minor), 128.2 (major), 70.6 (minor), 51.1 (major), 35.8 (minor), 32.7 (major), 29.3 (major), 27.3 (major), 26.5 (minor), 17.0 (minor).

HRMS (ESI) *m/z* calcd. for C_8H_{15} $[M - Br]^+$ 111.1168, found 111.1169.

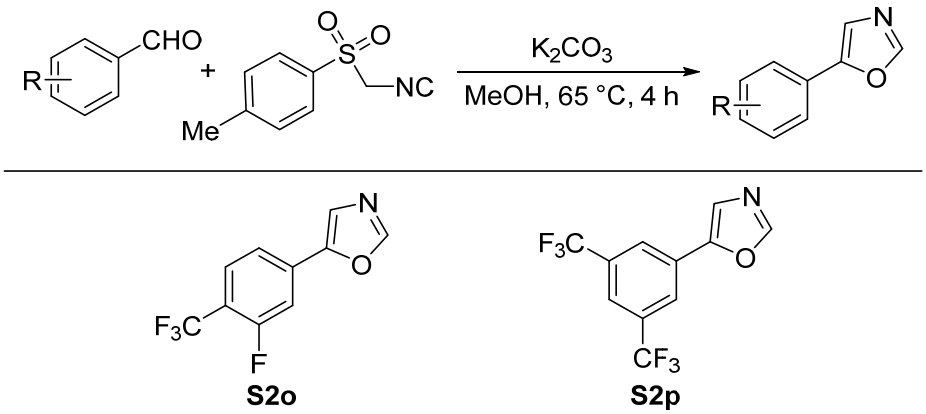
Azoles **2a** and **S2b**–**S2r** were synthesized According to the literature procedures.[4–8]All the characterization data are consistent with those in the reported literature.

General procedure for the synthesis of azoles 2a and S2b–S2n:



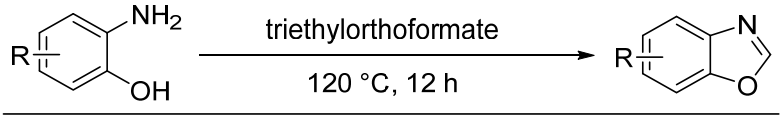
An appropriate benzhydrazide (7.3 mmol) was dissolved in triethyl orthoformate (6.7 mL, 40 mmol, 5.5 equiv) and the mixture was vigorously stirred at 120 °C overnight. The excess triethyl orthoformate was removed by evaporation under reduced pressure. The resulting oil was purified by column chromatography (petroleum ether/EtOAc = 4/1) to afford the desired product.

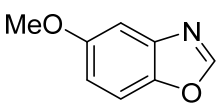
General procedure for the synthesis of azoles S2o and S2p:



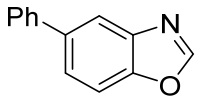
A mixture of an appropriate benzaldehyde (5.0 mmol), TosMIC (*p*-toluenesulfonylmethyl isocyanide) (1.46 g, 7.5 mmol, 1.5 equiv), and K_2CO_3 (1.38 g, 10 mmol, 2.0 equiv) in MeOH (7.5 mL) was heated at 65 °C. After completion of reaction (monitored by TLC), the mixture was cooled, quenched with water, and extracted with EtOAc. The solvent was removed under reduced pressure and the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 20/1) to yield the desired product.

General procedure for the synthesis of azoles S2q and S2r:





S2q

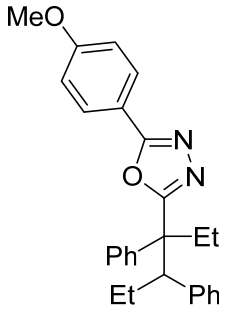


S2r

An appropriate 2-aminophenol (5.0 mmol, 1.0 equiv) was dissolved in triethyl orthoformate (5.0 mL, 30 mmol, 6.0 equiv) and the reaction mixture was carefully heated to 120 °C overnight. The excess triethyl orthoformate was removed by evaporation under reduced pressure. The resulting oil was purified by column chromatography (petroleum ether/EtOAc = 20/1) to yield the desired product.

General procedures for reaction condition optimization (*Table S1: Screening of reaction conditions*)

To a flame-dried Schlenk tube equipped with a magnetic stir bar were added copper (0.005 mmol, 10 mol%), ligand (0.006 mmol, 12 mol%), LiO^{t}Bu (18 mg, 0.22 mmol, 4.5 equiv), and an appropriate azole (0.05 mmol, 1.0 equiv). The tube was evacuated and backfilled with argon for three times, and then, an appropriate alkyl bromide (0.10 mmol, 2.0 equiv), water (1.8 μL, 0.10 mmol, 2.0 equiv), and anhydrous *N,N*-dimethylacetamide (0.40 mL) and dichloromethane (0.20 mL) were sequentially added. The reaction mixture was stirred at room temperature for 30 minutes and then stirred at rt or at 10 °C for 3–5 d. The resulting reaction mixture was diluted with 10 mL ethyl acetate and washed with brine (10 mL × 3). The organic layer was dried over anhydrous Na_2SO_4 and filtered through a pad of celite. The organic solvent was removed under vacuum and the residue was purified by column chromatography on silica gel to afford the desired product. Specially, when the reaction performed at 40 °C and 50 °C, the byproduct **3'** was obtained in 18% and 28% yield, respectively (dr : 1:1).



3'

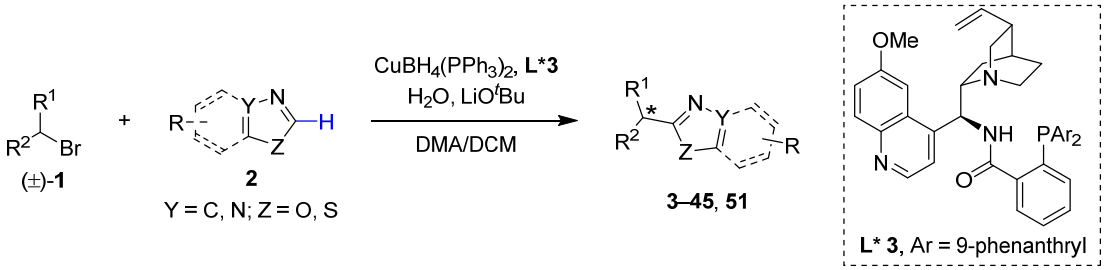
2-(3,4-diphenylhexan-3-yl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.91 (m, 2H), 7.37 – 7.27 (m, 3H), 7.22 – 7.15 (m, 4H), 7.10 – 7.07 (m, 1H), 7.02 – 6.95 (m, 2H), 6.76 – 6.70 (m, 2H), 3.88 (s, 1.5H), *3.86 (s, 2.7 Hz, 1.5H)*, 3.64 (dd, *J* = 11.9, 2.7 Hz, 0.5H), *3.37 (dd, J = 11.6, 2.7 Hz, 0.5H)*, 2.20 – 1.82 (m, 4H), 0.75 – 0.64 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, ***168.2***, 164.6, ***164.5***, 162.3, ***162.2***, 141.3, ***139.8***, 139.3, ***137.7***, 130.4, ***129.8***, 128.7(0), ***128.6(6)***, 128.3, ***128.0***, 127.5*(1)*, ***127.4(9)***, 127.3, ***127.2***, 127.0, ***126.8***, 126.7, 116.6(4), ***116.6(2)***, 114.5, ***114.4***, 57.0, ***55.8***, 55.5, ***54.2***, 54.0, ***53.5***, 32.3, ***31.0***, 25.1, ***24.4***, 13.0, ***12.6***, 9.4, ***9.1***.

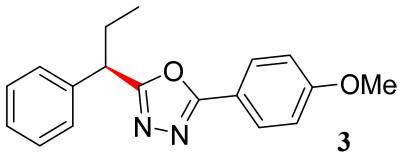
HRMS (ESI) *m/z* calcd. for $C_{27}H_{28}N_2O$ $[M + H]^+$ 413.2224, found 413.2219.

General procedures for enantioconvergent cross-coupling of racemic alkyl bromides with azole C(sp^2)–H bonds



To a flame-dried Schlenk tube equipped with a magnetic stir bar were added $CuBH_4(PPh_3)_2$ (6.0 mg, 0.010 mmol, 10 mol%), **L*3** (9.7 mg, 0.012 mmol, 12 mol%), LiO^tBu (36 mg, 0.45 mmol, 4.5 equiv), and an appropriate azole (0.10 mmol, 1.0 equiv). The tube was evacuated and backfilled with argon for three times, and then, an appropriate alkyl bromide (0.20 mmol, 2.0 equiv), water (3.6 μL, 0.20 mmol, 2.0 equiv), and anhydrous *N,N*-dimethylacetamide (0.70 mL) and dichloromethane (0.35 mL) were sequentially added. The reaction mixture was stirred at room temperature for 30 minutes and then stirred at 10 °C for 3–5 d. The resulting reaction mixture was diluted with 10 mL ethyl acetate and washed with brine (10 mL × 3). The organic layer was dried over anhydrous Na_2SO_4 and filtered through a pad of celite. The organic solvent was removed under vacuum and the residue was purified by column chromatography on silica gel to afford the desired product.

Characterization data of products 3–45 and 51



3

(*R*)-2-(4-Methoxyphenyl)-5-(1-phenylpropyl)-1,3,4-oxadiazole

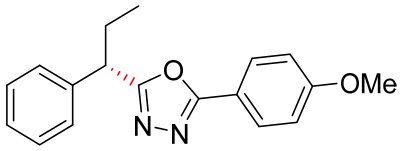
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **1a** (39.8 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **3** (24.3 mg) in 83% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.38 – 7.32 (m, 4H), 7.31 – 7.25 (m, 1H), 6.98 – 6.95 (m, 2H), 4.14 (t, *J* = 7.8 Hz, 1H), 3.85 (s, 3H), 2.40 – 2.29 (m, 1H), 2.18 – 2.07 (m, 1H), 0.98 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 164.7, 162.1, 139.0, 128.8, 128.5, 127.9, 127.4, 116.5, 114.3, 55.4, 45.0, 27.5, 12.1.

HRMS (ESI) *m*/*z* calcd. for $C_{18}H_{19}N_2O_2$ $[M + H]^+$ 295.1441, found 295.1438.

HPLC conditions: Chiralcel IF, *n*-hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 11.3 min, t(major) = 14.4 min, 90% ee (95.2:4.8 er).

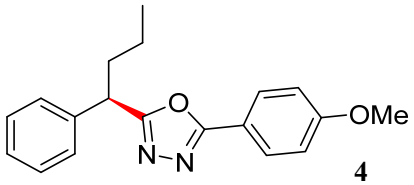


***ent*-3**

(*S*)-2-(4-methoxyphenyl)-5-(1-phenylpropyl)-1,3,4-oxadiazole

According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) alkyl bromide **1a** (39.8 mg, 0.20 mmol, 2.0 equiv) and with the the ligand **L*4** for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product ***ent*-3** (21.4 mg) in 78% isolated yield.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 82/20, flow rate 1.0 mL/min. λ = 254 nm, t(major) = 10.9 min, t(minor) = 13.7 min, 89% ee (94.6:5.4 er).



4

(*R*)-2-(4-Methoxyphenyl)-5-(1-phenylbutyl)-1,3,4-oxadiazole

According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1b** (42.6 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **4** (26.5 mg) in 85% isolated yield.

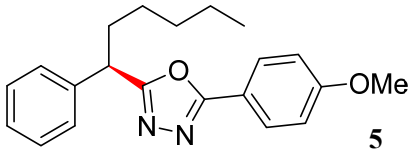
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.94 (m, 2H), 7.41 – 7.34 (m, 4H), 7.31 – 7.29 (m, 1H), 7.01 – 6.97 (m, 2H), 4.27 (t, *J* = 8.0 Hz, 1H), 3.88 (s, 3H), 2.35 – 2.26 (m, 1H), 2.15 – 2.05 (m, 1H), 1.44 – 1.34 (m, 2H), 0.97 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.7, 164.8, 162.2, 139.2, 128.8, 128.6, 127.9, 127.5,

116.6, 114.4, 55.4, 43.1, 36.3, 20.6, 13.7.

HRMS (ESI) *m/z* calcd. for $C_{19}H_{21}N_2O_2$ $[M + H]^+$ 309.1598, found 309.1594.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 14.2 min, t(major) = 18.3 min, 90% ee (94.8:5.2 er).



5

(*R*)-2-(4-Methoxyphenyl)-5-(1-phenylhexyl)-1,3,4-oxadiazole

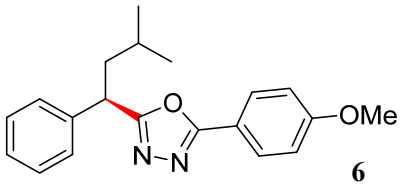
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1c** (48.2 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **5** (24.2 mg) in 72% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.38 – 7.32 (m, 4H), 7.29 – 7.27 (m, 1H), 6.98 – 6.95 (m, 2H), 4.23 (t, *J* = 8.0 Hz, 1H), 3.85 (s, 3H), 2.32 – 2.25 (m, 1H), 2.11 – 2.06 (m, 1H), 1.35 – 1.26 (m, 6H), 0.85 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.7, 164.8, 162.2, 139.3, 128.8, 128.6, 127.9, 127.5, 116.6, 114.4, 55.4, 43.4, 34.2, 31.4, 27.1, 22.4, 14.0.

HRMS (ESI) *m/z* calcd. for $C_{21}H_{25}N_2O_2$ $[M + H]^+$ 337.1911, found 337.1905.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 13.8 min, t(major) = 17.5 min, 89% ee (94.5:5.5 er).



6

(*R*)-2-(4-Methoxyphenyl)-5-(3-methyl-1-phenylbutyl)-1,3,4-oxadiazole

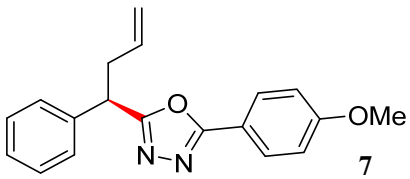
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1d** (45.4 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **6** (24.2 mg) in 75% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.39 – 7.32 (m, 4H), 7.29 – 7.24 (m, 1H), 6.98 – 6.95 (m, 2H), 4.35 (t, *J* = 8.0 Hz, 1H), 3.85 (s, 3H), 2.21 – 2.14 (m, 1H), 2.04 – 1.97 (m, 1H), 1.58 – 1.48 (m, 1H), 0.95 (d, *J* = 6.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8, 164.8, 162.2, 139.3, 128.9, 128.6, 127.9, 127.5, 116.6, 114.4, 55.4, 43.2, 41.2, 25.6, 22.6, 22.1.

HRMS (ESI) *m/z* calcd. for $C_{20}H_{23}N_2O_2$ $[M + H]^+$ 323.1754, found 323.1751.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 13.1 min, t(major) = 17.0 min, 88% ee (93.8:6.2 er).



7

(*R*)-2-(4-Methoxyphenyl)-5-(1-phenylbut-3-en-1-yl)-1,3,4-oxadiazole

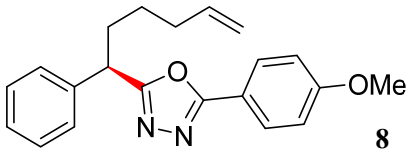
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1l** (42.2 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **7** (13.0 mg) in 42% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.38 – 7.33 (m, 4H), 7.30 – 7.27 (m, 1H), 6.99 – 6.95 (m, 2H), 5.83 – 5.73 (m, 1H), 5.14 – 5.01 (m, 2H), 4.33 (t, *J* = 8.0 Hz, 1H), 3.86 (s, 3H), 3.11 – 3.03 (m, 1H), 2.89 – 2.81 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 164.9, 162.2, 138.6, 134.6, 128.9, 128.6, 127.9, 127.6, 117.9, 116.5, 114.4, 55.5, 43.4, 38.4.

HRMS (ESI) *m*/*z* calcd. for $C_{19}H_{19}N_2O_2$ $[M + H]^+$ 307.1441, found 307.1438.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 14.9 min, t(major) = 18.3 min, 82% ee (91.2:8.8 er).



8

(*R*)-2-(4-Methoxyphenyl)-5-(1-phenylhex-5-en-1-yl)-1,3,4-oxadiazole

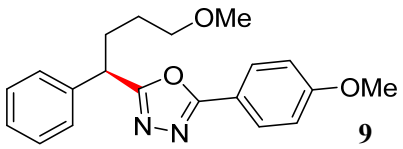
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1m** (47.8 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **8** (19.0 mg) in 56% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.94 (m, 2H), 7.39 – 7.35 (m, 4H), 7.32 – 7.29 (m, 1H), 7.05 – 6.97 (m, 2H), 5.83 – 5.73 (m, 1H), 5.05 – 4.95 (m, 2H), 4.25 (t, *J* = 7.8 Hz, 1H), 3.88 (s, 3H), 2.39 – 2.30 (m, 1H), 2.18 – 2.09 (m, 3H), 1.51 – 1.43 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 164.8, 162.2, 139.1, 138.1, 128.9, 128.6, 127.9, 127.5, 116.5, 115.0, 114.4, 55.5, 43.3, 33.7, 33.3, 26.7.

HRMS (ESI) *m*/*z* calcd. for $C_{21}H_{23}N_2O_2$ $[M + H]^+$ 335.1754, found 335.1749.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 14.7 min, t(major) = 18.7 min, 87% ee (93.5:6.5 er).



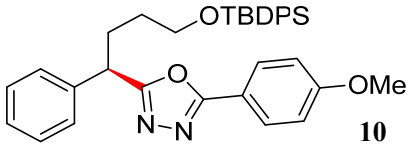
9

(*R*)-2-(4-Methoxy-1-phenylbutyl)-5-(4-Methoxyphenyl)-1,3,4-oxadiazole

According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1x** (67.8 mg, 0.25 mmol, 2.5 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **9** (22.0 mg) in 65% isolated yield.

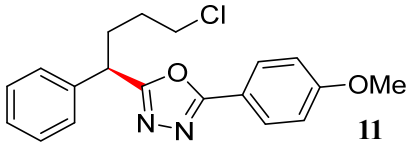
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.38 – 7.32 (m, 4H), 7.29 – 7.25 (m, 1H), 6.98 – 6.95 (m, 2H), 4.26 (t, *J* = 8.0 Hz, 1H), 3.86 (s, 3H), 3.42 – 3.38 (m,

2H), 3.30 (s, 3H), 2.42 – 2.33 (m, 1H), 2.24 – 2.14 (m, 1H), 1.69 – 1.58 (m, 2H)
^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 164.8, 162.2, 139.0, 128.9, 128.6, 127.9, 127.6, 116.5, 114.4, 72.1, 58.6, 55.4, 43.2, 31.0, 27.5.
HRMS (ESI) *m*/*z* calcd. for $C_{20}H_{23}N_2O_3$ $[M + H]^+$ 339.1703, found 339.1700.
HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 22.6 min, t(major) = 26.6 min, 87% ee (93.4:6.6 er).



10

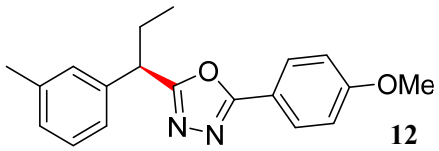
(*R*)-2-(4-((*tert*-Butyldiphenylsilyl)oxy)-1-phenylbutyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1w** (93.5 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **10** (22.5 mg) in 40% isolated yield.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.91 (m, 2H), 7.65 – 7.61 (m, 4H), 7.42 – 7.33 (m, 10H), 7.29 – 7.25 (m, 1H), 6.98 – 6.94 (m, 2H), 4.24 (t, *J* = 7.8 Hz, 1H), 3.85 (s, 3H), 3.71 – 3.67 (m, 2H), 2.43 – 2.36 (m, 1H), 2.26 – 2.16 (m, 1H), 1.62 – 1.55 (m, 2H), 1.03 (s, 9H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2, 139.1, 135.6, 133.8, 129.6, 128.9, 128.6, 127.9, 127.7, 127.5, 116.6, 114.4, 63.3, 55.5, 43.0, 30.6, 30.2, 26.9, 19.2.
HRMS (ESI) *m*/*z* calcd. for $C_{35}H_{39}N_2O_3Si$ $[M + H]^+$ 563.2724, found 563.2722.
HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 12.0 min, t(major) = 16.9 min, 90% ee (95:5 er).



11

(*R*)-2-(4-Chloro-1-phenylbutyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **S1e** (74.3 mg, 0.30 mmol, 3.0 equiv), LiO^{t}Bu (28.0 mg, 0.35 mmol, 3.5 equiv), and H_2O (5.4 μL, 0.30 mmol, 3.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **11** (13.7 mg) in 40% isolated yield.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.36 – 7.33 (m, 4H), 7.32 – 7.26 (m, 1H), 6.99 – 6.95 (m, 2H), 4.25 (t, *J* = 7.8 Hz, 1H), 3.86 (s, 3H), 3.59 – 3.53 (m, 2H), 2.51 – 2.42 (m, 1H), 2.33 – 2.24 (m, 1H), 1.93 – 1.78 (m, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 164.9, 162.3, 138.6, 129.0, 128.6, 127.8, 127.8, 116.4, 114.4, 55.5, 44.4, 42.8, 31.5, 30.3.
HRMS (ESI) *m*/*z* calcd. for $C_{19}H_{20}{}^{35}ClN_2O_2$ $[M + H]^+$ 343.1208, found 343.1207; for $C_{19}H_{20}{}^{37}ClN_2O_2$ $[M + H]^+$ 345.1178, found 345.1176.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 20.0 min, t(major) = 23.2 min, 78% ee (89:11 er).



12

(*R*)-2-(4-Methoxyphenyl)-5-(1-(*m*-tolyl)propyl)-1,3,4-oxadiazole

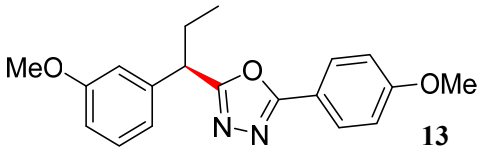
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1f** (42.6 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **12** (15.5 mg) in 50% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.92 (m, 2H), 7.23 (t, *J* = 8.0 Hz, 1H), 7.16 – 7.14 (m, 2H), 7.08 (d, *J* = 7.2 Hz, 1H), 6.98 – 6.95 (m, 2H), 4.09 (t, *J* = 8.0 Hz, 1H), 3.85 (s, 3H), 2.34 (s, 3H), 2.37 – 2.28 (m, 1H), 2.15 – 2.08 (m, 1H), 0.98 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.7, 164.8, 162.2, 139.0, 138.5, 128.7, 128.6(2), 128.6(0), 128.3, 124.9, 116.6, 114.4, 55.4, 45.1, 27.5, 21.5, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{19}H_{21}N_2O_2$ $[M + H]^+$ 309.1598, found 309.1593.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 11.8 min, t(major) = 14.0 min, 83% ee (91.7:8.3 er).



13

(*R*)-2-(4-Methoxyphenyl)-5-(1-(3-methoxyphenyl)propyl)-1,3,4-oxadiazole

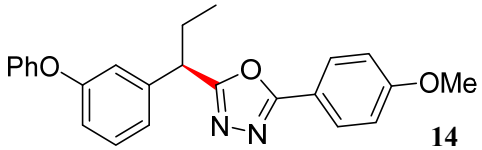
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1n** (57.3 mg, 0.25 mmol, 2.5 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **13** (26.0 mg) in 80% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.92 (m, 2H), 7.26 (t, *J* = 8.0 Hz, 1H), 6.99 – 6.93 (m, 3H), 6.91 (t, *J* = 2.0 Hz, 1H), 6.81 (ddd, *J* = 8.0, 2.8, 1.2 Hz, 1H), 4.11 (t, *J* = 8.0 Hz, 1H), 3.86 (s, 3H), 3.80 (s, 3H), 2.37 – 2.29 (m, 1H), 2.16 – 2.09 (m, 1H), 0.99 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 164.8, 162.2, 159.9, 140.6, 129.8, 128.6, 120.3, 116.6, 114.4, 113.8, 112.7, 55.4, 55.3, 45.1, 27.4, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{19}H_{21}N_2O_3$ $[M + H]^+$ 325.1547, found 325.1543.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 19.2 min, t(major) = 22.7 min, 90% ee (95:5 er).



14

(*R*)-2-(4-Methoxyphenyl)-5-(1-(3-phenoxyphenyl)propyl)-1,3,4-oxadiazole

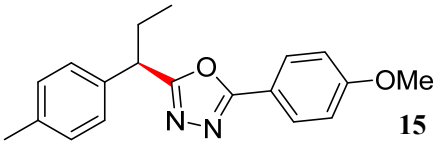
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1o** (58.2 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **14** (22.5 mg) in 58% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.91 (m, 2H), 7.35 – 7.26 (m, 3H), 7.16 – 7.08 (m, 2H), 7.06 – 6.96 (m, 5H), 6.90 (dd, *J* = 8.4, 1.2 Hz, 1H), 4.12 (t, *J* = 7.8 Hz, 1H), 3.87 (s, 3H), 2.36 – 2.25 (m, 1H), 2.16 – 2.05 (m, 1H), 0.98 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 164.9, 162.2, 157.7, 156.9, 141.0, 130.1, 129.8, 128.6, 123.5, 122.7, 119.0, 118.3, 117.7, 116.5, 114.4, 55.5, 44.9, 27.5, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{24}H_{23}N_2O_3$ $[M + H]^+$ 387.1703, found 387.1699.

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min. λ = 254 nm, t(major) = 24.7 min, t(minor) = 28.6 min, 84% ee (92:8 er).



15

(*R*)-2-(4-Methoxyphenyl)-5-(1-(*p*-tolyl)propyl)-1,3,4-oxadiazole

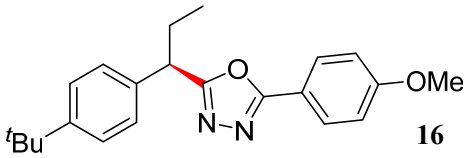
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1i** (42.6 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **15** (21.0 mg) in 68% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 – 7.94 (m, 2H), 7.29 – 7.26 (m, 2H), 7.17 (d, *J* = 7.6 Hz, 2H), 7.00 – 6.98 (m, 2H), 4.12 (t, *J* = 7.6 Hz, 1H), 3.88 (s, 3H), 2.38 – 2.31 (m, 1H), 2.38 (s, 3H), 2.16 – 2.09 (m, 1H), 1.00 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8, 164.8, 162.2, 137.2, 136.1, 129.5, 128.6, 127.8, 116.6, 114.4, 55.4, 44.7, 27.5, 21.1, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{19}H_{21}N_2O_2$ $[M + H]^+$ 309.1598, found 309.1593.

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min. λ = 254 nm, t(major) = 13.9 min, t(minor) = 16.6 min, 90% ee (95:5 er).



16

(*R*)-2-(1-(4-(*tert*-Butyl)phenyl)propyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole

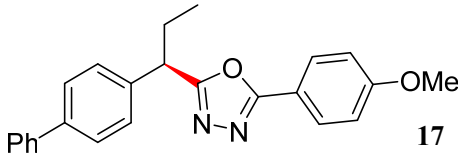
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1j** (63.8 mg, 0.25 mmol, 2.5 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **16** (17.5 mg) in 50% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.95 (m, 2H), 7.35 (dd, *J* = 6.0, 2.0 Hz, 2H), 7.28 (d, *J* = 6.0, 2.0 Hz, 2H), 7.00 – 6.98 (m, 2H), 4.12 (t, *J* = 7.8 Hz, 1H), 3.86 (s, 3H), 2.38 – 2.29 (m, 1H), 2.19 – 2.07 (m, 1H), 1.30 (s, 9H), 0.98 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.7, 164.7, 162.1, 150.4, 136.0, 128.6, 127.5, 125.7, 116.7, 114.4, 55.4, 44.6, 34.5, 31.3, 27.6, 12.3.

HRMS (ESI) *m*/*z* calcd. for $C_{22}H_{27}N_2O_2$ $[M + H]^+$ 351.2067, found 351.2066.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.6 mL/min. λ = 254 nm, t(minor) = 20.4 min, t(major) = 22.3 min, 90% ee (95:5 er).



17

(*R*)-2-(1-([1,1'-Biphenyl]-4-yl)propyl)-5-(4-Methoxyphenyl)-1,3,4-oxadiazole

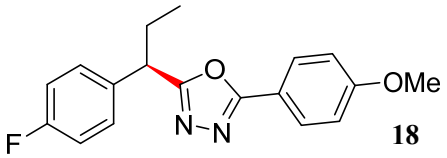
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1t** (68.8 mg, 0.25 mmol, 2.5 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **17** (24.1 mg) in 65% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 – 7.93 (m, 2H), 7.59 – 7.56 (m, 4H), 7.45 – 7.41 (m, 4H), 7.36 – 7.32 (m, 1H), 6.99 – 6.95 (m, 2H), 4.19 (t, *J* = 7.7 Hz, 1H), 3.86 (s, 3H), 2.41 – 2.32 (m, 1H), 2.22 – 2.11 (m, 1H), 1.02 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 164.8, 162.2, 140.6, 140.5, 138.1, 128.8, 128.6, 128.4, 127.6, 127.4, 127.1, 116.6, 114.4, 55.5, 44.8, 27.6, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{24}H_{23}N_2O_3$ $[M + H]^+$ 371.1754, found 371.1750.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 20.2 min, t(major) = 26.8 min, 90% ee (95.1:4.9 er).



18

(*R*)-2-(1-(4-Fluorophenyl)propyl)-5-(4-Methoxyphenyl)-1,3,4-oxadiazole

According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1g** (65.1 mg, 0.30 mmol, 3.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **18** (20.6 mg) in 66% isolated yield.

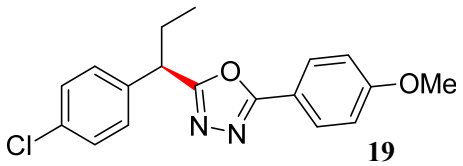
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.92 (m, 2H), 7.35 – 7.32(m, 2H), 7.06 – 7.01 (m, 2H), 7.00 – 6.96 (m, 2H), 4.13 (t, *J* = 7.8 Hz, 1H), 3.86 (s, 3H), 2.36 – 2.29 (m, 1H), 2.13 – 2.06 (m, 1H), 0.98 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 166.1 (d, $J_{C\text{-}F}$ = 241.9 Hz), 163.4, 162.2, 160.9, 134.8 (d, $J_{C\text{-}F}$ = 3.0 Hz), 129.5 (d, $J_{C\text{-}F}$ = 8.0 Hz), 128.6, 116.5, 115.7 (d, $J_{C\text{-}F}$ = 21.5 Hz), 114.4, 55.5, 44.4, 27.6, 12.1.

^{19}F NMR (376 MHz, $CDCl_3$): δ −115.0.

HRMS (ESI) *m*/*z* calcd. for $C_{18}H_{18}FN_2O_3$ $[M + H]^+$ 313.1347, found 313.1341.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 13.3 min, t(major) = 17.6 min, 87% ee (93.6:6.4 er).



19

(*R*)-2-(1-(4-Chlorophenyl)propyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole

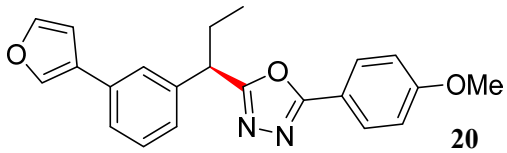
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1h** (70.1 mg, 0.30 mmol, 3.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **19** (12.5 mg) in 38% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.34 – 7.26 (m, 4H), 6.99 – 6.95 (m, 2H), 4.12 (t, *J* = 8.0 Hz, 1H), 3.86 (s, 3H), 2.36 – 2.29 (m, 1H), 2.13 – 2.06 (m, 1H), 0.98 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 164.9, 162.3, 137.5, 133.4, 129.3, 129.0, 128.6, 116.4, 114.4, 55.5, 44.5, 27.5, 12.1.

HRMS (ESI) *m*/*z* calcd. for $C_{18}H_{18}{}^{35}ClN_2O_2$ $[M + H]^+$ 329.1051, found 329.1047. for $C_{18}H_{18}{}^{37}ClN_2O_2$ $[M + H]^+$ 331.1022, found 331.1017.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 14.0 min, t(major) = 20.2 min, 85% ee (92.5:7.5 er).



20

(*R*)-2-(1-(3-(Furan-3-yl)phenyl)propyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole

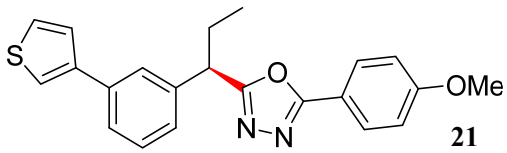
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1p** (66.3 mg, 0.25 mmol, 2.5 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **20** (28.0 mg) in 78% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.92 (m, 2H), 7.73 (t, *J* = 1.2 Hz, 1H), 7.47 (q, *J* = 2.0 Hz, 2H), 7.40 (dt, *J* = 8.0, 1.6 Hz, 1H), 7.35 (t, *J* = 7.2 Hz, 1H), 7.28 – 7.25 (m, 1H), 6.98 – 6.95 (m, 2H), 6.70 (dd, *J* = 2.0, 0.8 Hz, 1H), 4.16 (t, *J* = 7.8 Hz, 1H), 3.85 (s, 3H), 2.43 – 2.32 (m, 1H), 2.21 – 2.09 (m, 1H), 1.01 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 164.8, 162.2, 143.8, 139.7, 138.7, 133.0, 129.3, 128.6, 126.5, 126.2, 125.5, 125.1, 116.5, 114.4, 108.8, 55.5, 45.1, 27.5, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{22}H_{21}N_2O_3$ $[M + H]^+$ 361.1547, found 361.1543.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 18.8 min, t(major) = 23.3 min, 93% ee (96.4:3.6 er).



21

(*R*)-2-(4-Methoxyphenyl)-5-(1-(3-(thiophen-3-yl)phenyl)propyl)-1,3,4-oxadiazole

According to the general procedure with azole **2a** (35.2 mg, 0.20 mmol, 1.0 equiv), alkyl bromide **S1r** (140.6 mg, 0.50 mmol, 2.5 equiv), and LiO^tBu (60.8 mg, 0.76 mmol, 3.8 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **21** (60.0 mg) in 80% isolated yield.

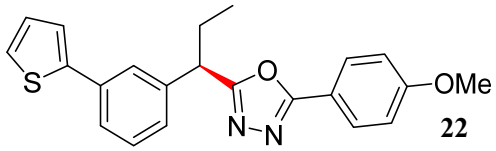
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.92 (m, 2H), 7.58 (t, *J* = 1.2 Hz, 1H), 7.51 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.46 (t, *J* = 2.4 Hz, 1H), 7.40 – 7.36 (m, 3H), 7.29 (dt, *J* = 7.6,

1.6 Hz, 1H), 6.98 – 6.95 (m, 2H), 4.18 (t, *J* = 7.8 Hz, 1H), 3.85 (s, 3H), 2.42 – 2.33 (m, 1H), 2.22 – 2.13 (m, 1H), 1.01 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 164.8, 162.2, 141.9, 139.7, 136.4, 129.3, 128.6, 126.6, 126.4, 126.3, 126.1, 125.7, 120.7, 116.5, 114.4, 55.5, 45.2, 27.5, 12.3.

HRMS (ESI) *m*/*z* calcd. for $C_{22}H_{21}N_2O_2S$ $[M + H]^+$ 377.1318, found 377.1313.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 19.8 min, t(major) = 21.2 min, 93% ee (96.3:3.7 er).



22

(*R*)-2-(4-Methoxyphenyl)-5-(1-(3-(thiophen-2-yl)phenyl)propyl)-1,3,4-oxadiazole

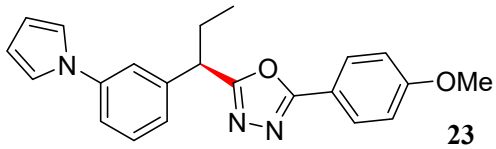
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1s** (56.2 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **22** (22.0 mg) in 55% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.93 (m, 2H), 7.60 (t, *J* = 1.2 Hz, 1H), 7.52 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.36 (t, *J* = 7.6 Hz, 1H), 7.32 (dd, *J* = 3.6, 1.2 Hz, 1H), 7.29 – 7.27 (m, 2H), 7.08 (dd, *J* = 4.8, 3.6 Hz, 1H), 6.98 – 6.95 (m, 2H), 4.17 (t, *J* = 7.8 Hz, 1H), 3.85 (s, 3H), 2.43 – 2.32 (m, 1H), 2.22 – 2.11 (m, 1H), 1.01 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.4, 164.9, 162.2, 143.9, 139.8, 135.0, 129.4, 128.6, 128.1, 126.9, 125.6, 125.2, 125.1, 123.4, 116.5, 114.4, 55.5, 45.1, 27.5, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{22}H_{21}N_2O_2S$ $[M + H]^+$ 377.1318, found 377.1313.

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min. λ = 254 nm, t(major) = 25.3 min, t(minor) = 30.9 min, 85% ee (92.6:7.4 er).



23

(*R*)-2-(1-(3-(1*H*-Pyrrol-1-yl)phenyl)propyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole

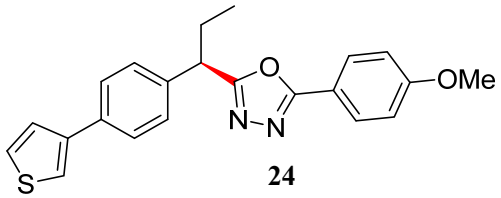
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **S1q** (79.2 mg, 0.30 mmol, 3.0 equiv), LiO^tBu (28.0 mg, 0.35 mmol, 3.5 equiv), and H_2O (5.4 μL, 0.30 mmol, 3.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **23** (12.6 mg) in 35% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.92 (m, 2H), 7.42 – 7.38 (m, 2H), 7.32 – 7.30 (m, 1H), 7.26 – 7.23 (m, 1H), 7.08 (t, *J* = 2.0 Hz, 2H), 6.98 – 6.96 (m, 2H), 6.35 (t, *J* = 2.0 Hz, 2H), 4.18 (t, *J* = 8.0 Hz, 1H), 3.86 (s, 3H), 2.43 – 2.32 (m, 1H), 2.22 – 2.11 (m, 1H), 1.01 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.3, 141.2, 140.7, 130.0, 128.6, 125.1, 120.1, 119.6, 119.3, 116.4, 114.4, 110.6, 55.5, 45.0, 27.5, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{22}H_{22}N_3O_2$ $[M + H]^+$ 360.1707, found 360.1703.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 19.1 min, t(major) = 20.4 min, 87% ee (93.6:6.4 er).



24

(*R*)-2-(4-Methoxyphenyl)-5-(1-(4-(thiophen-3-yl)phenyl)propyl)-1,3,4-oxadiazole

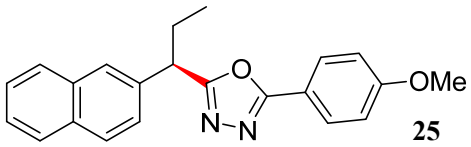
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1u** (70.3 mg, 0.25 mmol, 2.5 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **24** (22.6 mg) in 60% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 – 7.92 (m, 2H), 7.58 – 7.56 (m, 2H), 7.43 (dd, *J* = 2.4, 1.6 Hz, 1H), 7.40 – 7.36 (m, 4H), 6.98 – 6.96 (m, 2H), 4.16 (t, *J* = 8.0 Hz, 1H), 3.85 (s, 3H), 2.42 – 2.31 (m, 1H), 2.20 – 2.09 (m, 1H), 1.01 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2, 141.8, 137.9, 135.2, 128.6, 128.4, 126.9, 126.3, 126.3, 120.4, 116.6, 114.4, 55.5, 44.8, 27.5, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{22}H_{21}N_2O_2S$ $[M + H]^+$ 377.1318, found 377.1313.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 28.7 min, t(major) = 38.4 min, 89% ee (94.6:5.4 er).



25

(*R*)-2-(4-Methoxyphenyl)-5-(1-(naphthalen-2-yl)propyl)-1,3,4-oxadiazole

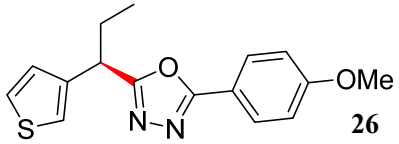
According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1k** (74.7 mg, 0.30 mmol, 3.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **25** (18.5 mg) in 53% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.90 (m, 2H), 7.84 – 7.80 (m, 4H), 7.50 – 7.44 (m, 3H), 6.96 – 6.94 (m, 2H), 4.30 (t, *J* = 7.6 Hz, 1H), 3.84 (s, 3H), 2.48 – 2.39 (m, 1H), 2.29 – 2.20 (m, 1H), 1.02 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 164.9, 162.2, 136.4, 133.4, 132.7, 128.7, 128.6, 127.8, 127.7, 126.9, 126.4, 126.1, 125.7, 116.5, 114.4, 55.4, 45.2, 27.4, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{22}H_{21}N_2O_3$ $[M + H]^+$ 345.1598, found 345.1593.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 17.2 min, t(major) = 22.3 min, 77% ee (88.7:11.3 er).



26

(*R*)-2-(4-Methoxyphenyl)-5-(1-(thiophen-3-yl)propyl)-1,3,4-oxadiazole

According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **S1v** (61.5 mg, 0.30 mmol, 3.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding

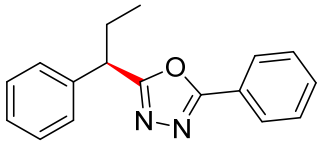
product **26** (13.8 mg) in 46% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.93 (m, 2H), 7.31 (dd, *J* = 4.8, 2.8 Hz, 1H), 7.21 (dd, *J* = 3.2, 1.6 Hz, 1H), 7.11 (dd, *J* = 5.2, 1.2 Hz, 1H), 7.00 – 6.96 (m, 2H), 4.32 (t, *J* = 7.7 Hz, 1H), 3.87 (s, 3H), 2.31 – 2.22 (m, 1H), 2.19 – 2.10 (m, 1H), 0.99 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2, 139.3, 128.6, 127.0, 126.2, 122.2, 116.5, 114.4, 55.5, 40.3, 27.5, 12.1.

HRMS (ESI) *m*/*z* calcd. for $C_{16}H_{17}N_2O_2S$ $[M + H]^+$ 301.1005, found 301.1002.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 18.5 min, t(major) = 23.2 min, 86% ee (93:7 er).



27

(*R*)-2-Phenyl-5-(1-phenylpropyl)-1,3,4-oxadiazole

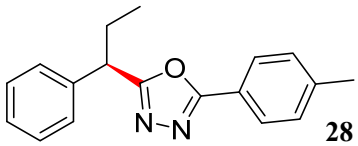
According to the general procedure with azole **S2b** (14.6 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv), and LiO^tBu (20.0 mg, 0.25 mmol, 2.5 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **27** (17.6 mg) in 67% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 8.01 (m, 2H), 7.55 – 7.47 (m, 3H), 7.41 – 7.35 (m, 4H), 7.32 – 7.28 (m, 1H), 4.19 (t, *J* = 7.8 Hz, 1H), 2.44 – 2.33 (m, 1H), 2.22 – 2.11 (m, 1H), 1.02 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 168.1, 164.9, 138.9, 131.6, 129.0, 128.9, 127.9, 127.6, 126.9, 124.0, 45.1, 27.5, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{17}H_{17}N_2O$ $[M + H]^+$ 265.1335, found 265.1331.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min. λ = 254 nm, t(minor) =10.5 min, t(major) = 12.6 min, 89% ee (94.3:5.7 er).



28

(*R*)-2-(1-Phenylpropyl)-5-(*p*-tolyl)-1,3,4-oxadiazole

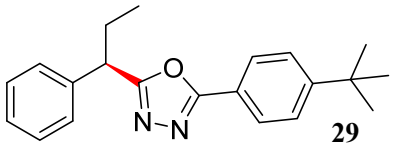
According to the general procedure with azole **S2c** (16.0 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv), and LiO^tBu (20.0 mg, 0.25 mmol, 2.5 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **28** (20.0 mg) in 72% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.87 (m, 2H), 7.38 – 7.32 (m, 4H), 7.29 – 7.25 (m, 3H), 4.15 (t, *J* = 7.7 Hz, 1H), 2.40 – 2.31 (m, 4H), 2.19 – 2.08 (m, 1H), 0.98 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8, 165.0, 142.0, 139.0, 129.6, 128.8, 127.9, 127.5, 126.8, 121.2, 45.1, 27.5, 21.6, 12.1.

HRMS (ESI) *m*/*z* calcd. for $C_{18}H_{19}N_2O$ $[M + H]^+$ 279.1492, found 279.1487.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 8.8 min, t(major) = 11.0 min, 89% ee (94.5:5.5 er).



29

(*R*)-2-(4-(*tert*-Butyl)phenyl)-5-(1-phenylpropyl)-1,3,4-oxadiazole

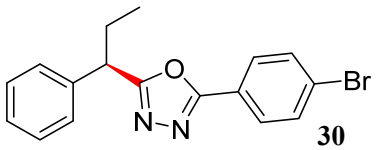
According to the general procedure with azole **S2d** (20.2 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv), and LiO^{t}Bu (20.0 mg, 0.25 mmol, 2.5 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **29** (22.1 mg) in 69% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.91 (m, 2H), 7.50 – 7.47 (m, 2H), 7.38 – 7.31 (m, 4H), 7.29 – 7.25 (m, 1H), 4.16 (t, *J* = 7.8 Hz, 1H), 2.40 – 2.29 (m, 1H), 2.19 – 2.08 (m, 1H), 1.33 (s, 9H), 0.98 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8, 164.9, 155.1, 139.0, 128.8, 127.9, 127.5, 126.6, 125.9, 121.2, 45.1, 35.0, 31.1, 27.5, 12.1.

HRMS (ESI) *m*/*z* calcd. for $C_{21}H_{25}N_2O$ $[M + H]^+$ 321.1961, found 321.1956.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 7.1 min, t(major) = 8.8 min, 90% ee (95.2:4.8 er).



30

(*R*)-2-(4-Bromophenyl)-5-(1-phenylpropyl)-1,3,4-oxadiazole

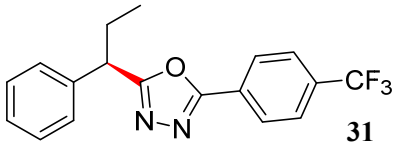
According to the general procedure with azole **S2e** (45.0 mg, 0.20 mmol, 1.0 equiv) and alkyl bromide **1a** (119.5 mg, 0.60 mmol, 3.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **30** (51.3 mg) in 75% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.85 (m, 2H), 7.63 – 7.59 (m, 2H), 7.37 – 7.32 (m, 4H), 7.31 – 7.27 (m, 1H), 4.15 (t, *J* = 7.8 Hz, 1H), 2.41 – 2.30 (m, 1H), 2.19 – 2.08 (m, 1H), 0.99 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 168.3, 164.1, 138.7, 132.2, 128.9, 128.2, 127.9, 127.6, 126.2, 122.9, 45.1, 27.4, 12.1.

HRMS (ESI) *m*/*z* calcd. for $C_{17}H_{16}{}^{79}BrN_2O$ $[M + H]^+$ 343.0441, found 343.0436, $C_{17}H_{16}{}^{81}BrN_2O$ $[M + H]^+$ 345.0420 found 345.0415.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 9.3 min, t(major) = 11.3 min, 87% ee (93.3:6.7 er).

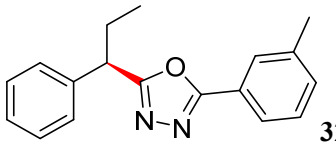


31

(*R*)-2-(1-Phenylpropyl)-5-(4-(trifluoromethyl)phenyl)-1,3,4-oxadiazole

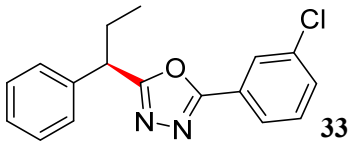
According to the general procedure with azole **S2f** (21.4 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv) at room temperature for 3 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **31** (15.7 mg) in 47% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.12 (m, 2H), 7.75 – 7.73 (m, 2H), 7.39 – 7.34 (m, 4H), 7.32 – 7.28 (m, 1H), 4.18 (t, *J* = 7.8 Hz, 1H), 2.43 – 2.32 (m, 1H), 2.22 – 2.11 (m, 1H), 1.00 (t, *J* = 7.4 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 168.8, 163.7, 138.6, 133.2 (q, *J* = 32.7 Hz), 128.9, 127.9, 127.7, 127.2(0), 127.2(6), 126.0 (q, *J* = 4.0 Hz), 123.5 (q, *J* = 271.0 Hz), 45.1, 27.5, 12.1.
^{19}F NMR (376 MHz, $CDCl_3$) δ −63.10.
HRMS (ESI) *m/z* calcd. for $C_{18}H_{16}F_3N_2O$ $[M + H]^+$ 333.1209, found 333.1204.
HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min. λ = 254 nm, t(minor) =7.3 min, t(major) = 8.3 min, 78% ee (89.2:10.8 er).



32

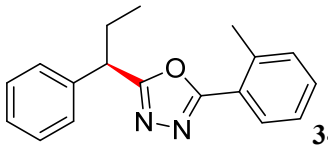
(*R*)-2-(1-Phenylpropyl)-5-(*m*-tolyl)-1,3,4-oxadiazole
According to the general procedure with azole **S2g** (16.0 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **1a** (39.8 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **32** (13.5 mg) in 49% isolated yield.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.77 (m, 2H), 7.38 – 7.26 (m, 7H), 4.16 (t, *J* = 7.8 Hz, 1H), 2.40 – 2.30 (m, 4H), 2.19 – 2.08 (m, 1H), 0.99 (t, *J* = 7.4 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 168.0, 165.0, 139.0, 138.8, 132.3, 128.8, 127.9, 127.5, 127.3, 124.0, 123.8, 45.1, 27.5, 21.3, 12.2.
HRMS (ESI) *m/z* calcd. for $C_{18}H_{19}N_2O$ $[M + H]^+$ 279.1492, found 279.1487.
HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 6.9 min, t(major) = 7.4 min, 85% ee (92.4:7.6 er).



33

(*R*)-2-(3-Chlorophenyl)-5-(1-phenylpropyl)-1,3,4-oxadiazole
According to the general procedure with azole **S2h** (18.1 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv) and H_2O (5.4 μL, 0.30 mmol, 3.0 equiv) in anhydrous *N,N*-dimethylacetamide (0.35 mL) and $PhCF_3$ (0.70 mL) at room temperature for 3 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **33** (10.2 mg) in 34% isolated yield (57% yield based on recovered starting material).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.97 (m, 1H), 7.92 – 7.89 (m, 1H), 7.49 – 7.46 (m, 1H), 7.43 – 7.41 (m, 1H), 7.36 – 7.34 (m, 4H), 7.32 – 7.27 (m, 1H), 4.16 (t, *J* = 7.8 Hz, 1H), 2.41 – 2.30 (m, 1H), 2.20 – 2.09 (m, 1H), 0.99 (t, *J* = 7.4 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 168.4, 163.7, 138.7, 135.0, 131.6, 130.3, 128.9, 127.9, 127.6, 126.8, 125.6, 124.9, 45.1, 27.4, 12.1.
HRMS (ESI) *m/z* calcd. for $C_{17}H_{16}{}^{35}ClN_2O$ $[M + H]^+$ 299.0946, found 299.0943; for $C_{17}H_{15}{}^{37}ClN_2O$ $[M + H]^+$ 301.0916 found 301.0913.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.8 mL/min. λ = 254 nm, t(minor) =14.3 min, t(major) = 15.7 min, 79% ee (89.5:10.5 er).



34

(*R*)-2-(1-Phenylpropyl)-5-(*o*-tolyl)-1,3,4-oxadiazole

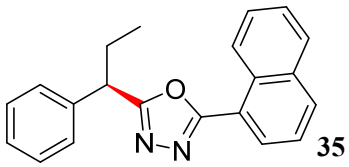
According to the general procedure with azole **S2i** (16.0 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **1a** (39.8 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **34** (14.1 mg) in 51% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.85 (m, 1H), 7.40 – 7.26 (m, 8H), 4.18 (t, *J* = 7.8 Hz, 1H), 2.65 (s, 3H), 2.41 – 2.30 (m, 1H), 2.20 – 2.09 (m, 1H), 0.99 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 165.1, 138.9, 138.2, 131.6, 131.0, 128.9, 128.8, 127.9, 127.5, 126.0, 123.1, 45.0, 27.4, 22.0, 12.1.

HRMS (ESI) *m*/*z* calcd. for $C_{18}H_{19}N_2O$ $[M + H]^+$ 279.1492, found 279.1488.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 11.6 min, t(major) = 12.9 min, 85% ee (92.3:7.7 er).



35

(*R*)-2-(Naphthalen-1-yl)-5-(1-phenylpropyl)-1,3,4-oxadiazole

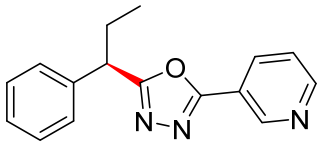
According to the general procedure with azole **S2j** (19.6 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv) and H_2O (5.4 μL, 0.30 mmol, 3.0 equiv) in anhydrous *N,N*-dimethylacetamide (0.35 mL) and $PhCF_3$ (0.70 mL) at room temperature for 3 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **35** (12.6 mg) in 40% isolated yield (73% yield based on recovered starting material).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.20 (dd, *J* = 8.7, 1.1 Hz, 1H), 8.12 (dd, *J* = 7.3, 1.3 Hz, 1H), 8.04 – 8.01 (m, 1H), 7.94 – 7.92 (m, 1H), 7.70 – 7.66 (m, 1H), 7.62 – 7.54 (m, 2H), 7.46 – 7.37 (m, 4H), 7.34 – 7.30 (m, 1H), 4.27 (t, *J* = 7.8 Hz, 1H), 2.50 – 2.39 (m, 1H), 2.28 – 2.17 (m, 1H), 1.06 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.7, 164.8, 138.9, 133.8, 132.4, 130.0, 128.9, 128.6, 128.2, 128.0, 127.9, 127.5, 126.6, 126.2, 124.7, 120.6, 45.0, 27.5, 12.2.

HRMS (ESI) *m*/*z* calcd. for $C_{21}H_{19}N_2O$ $[M + H]^+$ 315.1492, found 315.1487.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.8 mL/min. λ = 254 nm, t(minor) =20.2 min, t(major) = 21.5 min, 85% ee (92.6:7.4 er).



36

(*R*)-2-(1-Phenylpropyl)-5-(pyridin-3-yl)-1,3,4-oxadiazole

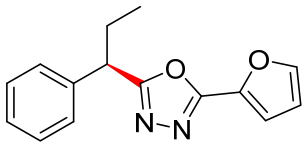
According to the general procedure with azole **S2k** (14.7 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv) and LiO^{t}Bu (20.0 mg, 0.25 mmol, 2.5 equiv) in anhydrous *N,N*-dimethylacetamide (0.35 mL) and $PhCF_3$ (0.70 mL) at room temperature for 3 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **36** (6.7 mg) in 25% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.21 (s, 1H), 8.75 – 8.73 (m, 1H), 8.30 (dt, *J* = 8.0, 1.9 Hz, 1H), 7.45 – 7.41 (m, 1H), 7.38 – 7.34 (m, 4H), 7.32 – 7.28 (m, 1H), 4.18 (t, *J* = 7.8 Hz, 1H), 2.43 – 2.32 (m, 1H), 2.22 – 2.11 (m, 1H), 1.00 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 162.8, 152.2, 147.8, 138.6, 134.1, 128.9, 127.9, 127.7, 123.7, 120.5, 45.1, 27.4, 12.1.

HRMS (ESI) *m/z* calcd. for $C_{16}H_{16}N_3O$ $[M + H]^+$ 266.1288, found 266.1284.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min. λ = 254 nm, t(minor) =21.2 min, t(major) = 23.7 min, 75% ee (87.3:12.7 er).



37

(*R*)-2-(Furan-2-yl)-5-(1-phenylpropyl)-1,3,4-oxadiazole

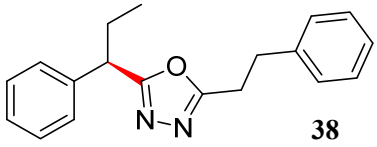
According to the general procedure with azole **S2l** (13.6 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv) and LiO^{t}Bu (20.0 mg, 0.25 mmol, 2.5 equiv) in anhydrous *N,N*-dimethylacetamide (0.35 mL) and $PhCF_3$ (0.70 mL) at room temperature for 3 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **37** (8.7 mg) in 34% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (dd, *J* = 1.9, 0.8 Hz, 1H), 7.37 – 7.32 (m, 4H), 7.31 – 7.25 (m, 1H), 7.10 (dd, *J* = 3.6, 0.7 Hz, 1H), 6.55 (dd, *J* = 3.5, 1.8 Hz, 1H), 4.14 (t, *J* = 7.8 Hz, 1H), 2.40 – 2.29 (m, 1H), 2.18 – 2.08 (m, 1H), 0.98 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.4, 157.7, 145.4, 139.5, 138.7, 128.8, 127.9, 127.6, 113.8, 112.0, 45.0, 27.4, 12.1.

HRMS (ESI) *m/z* calcd. for $C_{15}H_{15}N_2O_2$ $[M + H]^+$ 255.1128, found 255.1124.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. λ = 254 nm, t(minor) =18.5 min, t(major) = 22.2 min, 82% ee (91:9 er).



38

(*R*)-2-Phenethyl-5-(1-phenylpropyl)-1,3,4-oxadiazole

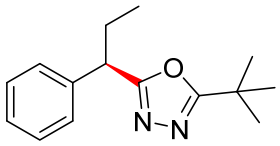
According to the general procedure with azole **S2m** (17.4 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **1a** (39.8 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **38** (16.1 mg) in 55% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.17 (m, 8H), 7.14 – 7.11 (m, 2H), 4.03 (t, *J* = 7.8 Hz, 1H), 3.12 – 3.02 (m, 4H), 2.30 – 2.19 (m, 1H), 2.10 – 1.99 (m, 1H), 0.92 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 168.1, 166.3, 139.4, 138.9, 128.8, 128.6, 128.2, 127.8, 127.4, 126.5, 44.9, 32.5, 27.2(4), 27.2(3), 12.1.

HRMS (ESI) *m*/*z* calcd. for $C_{19}H_{21}N_2O$ $[M + H]^+$ 293.1648, found 293.1643.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. λ = 214 nm, t(minor) = 17.0 min, t(major) = 17.9 min, 81% ee (90.5:9.5 er).



39

(*R*)-2-(*tert*-Butyl)-5-(1-phenylpropyl)-1,3,4-oxadiazole

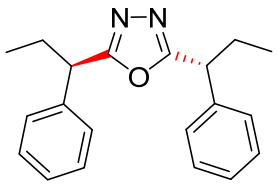
According to the general procedure with azole **S2n** (12.6 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv), and LiO^tBu (24.0 mg, 0.30 mmol, 3.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **39** (11.5 mg) in 47% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.25 (m, 5H), 4.09 (t, *J* = 7.8 Hz, 1H), 2.32 – 2.21 (m, 1H), 2.13 – 2.04 (m, 1H), 1.39 (s, 9H), 0.94 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 167.9, 139.1, 128.7, 127.8, 127.4, 45.0, 32.3, 28.1, 27.7, 12.1.

HRMS (ESI) *m*/*z* calcd. For $C_{16}H_{21}NO$ $[M + H]^+$ 245.1648, found 245.1646.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 99/1, flow rate 0.8 mL/min. λ = 214 nm, t(minor) = 31.1 min, t(major) = 32.3 min, 93% ee (96.5:3.5 er).



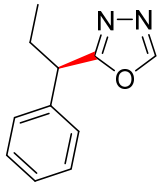
40

2,5-bis(1-phenylpropyl)-1,3,4-oxadiazole

According to the general procedure with 1,3,4-oxadiazole (7.0 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (199 mg, 1.0 mmol, 10.0 equiv), and LiO^tBu (36.0 mg, 0.45 mmol, 4.5 equiv) for 6 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **40** (13.2 mg) in 43% isolated yield, the *dr* value is 6:1, and give the mono-alkylated product **40′** (6.4 mg) in 34% isolated yield.

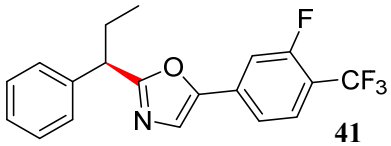
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.24 (m, 10H), 4.08 (t, *J* = 7.8 Hz, 1H), 4.03 (t, *J* = 7.8 Hz, 1H), 2.31 – 2.17 (m, 2H), 2.11 – 1.99 (m, 2H), 0.94 – 0.89 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 168.3, 139.1, ***139.0***, 128.7(2), ***128.7(1)***, 127.8, 127.4(0), ***127.3(9)***, 44.9(8), ***44.9(6)***, 27.6, ***27.5***, 12.0.
HRMS (ESI) *m/z* calcd. for $C_{20}H_{23}N_2O$ $[M + H]^+$ 307.1805, found 307.1800.
HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 99/1, flow rate 1.0 mL/min. λ = 214 nm, t(major) = 16.1 min, t(minor) = 23.7 min, 97% ee (98.5:1.5 er).



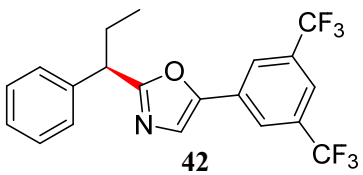
40′

2-(1-phenylpropyl)-1,3,4-oxadiazole
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (s, 1H), 7.36 – 7.25 (m, 5H), 4.13 (t, *J* = 7.8 Hz, 1H), 2.37 – 2.26 (m, 1H), 2.17 – 2.06 (m, 1H), 0.95 (t, *J* = 7.4 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 168.3, 153.0, 138.6, 128.9, 127.9, 127.7, 44.9, 27.3, 12.1.
HRMS (ESI) *m/z* calcd. for $C_{11}H_{13}N_2O$ $[M + H]^+$ 189.1022, found 189.1021.
HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 98/2, flow rate 1.0 mL/min. λ = 214 nm, t(major) = 19.1 min, t(minor) = 21.1 min, 77% ee (88.8:11.2 er).



41

(*R*)-5-(3-Fluoro-4-(trifluoromethyl)phenyl)-2-(1-phenylpropyl)oxazole
According to the general procedure with azole **S2o** (23.1 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv), and $Cu(^{i}PrCO_2)_2$ (2.38 mg, 0.010 mmol, 10 mol%) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **41** (10.5 mg) in 30% isolated yield (51% yield based on recovered starting material).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (t, *J* = 7.7 Hz, 1H), 7.47 – 7.40 (m, 3H), 7.37 (d, *J* = 4.3 Hz, 4H), 7.31 – 7.28 (m, 1H), 4.08 (t, *J* = 7.8 Hz, 1H), 2.40 – 2.29 (m, 1H), 2.17 – 2.06 (m, 1H), 0.99 (t, *J* = 7.3 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 160.1 (dd, *J* = 256.2, 2.3 Hz), 148.6 (d, *J* = 2.7 Hz), 139.9, 133.7 (d, *J* = 9.0 Hz), 128.8, 127.9, 127.3, 124.7, 122.4 (q, *J* = 272.4 Hz), 119.3 (d, *J* = 3.7 Hz), 117.5 (q, *J* = 33.2 Hz), 117.3 (q, *J* = 33.1 Hz), 112.1 (d, *J* = 23.0 Hz), 47.5, 28.0, 12.3.
^{19}F NMR (376 MHz, $CDCl_3$) δ –61.26 (d, *J* = 12.6 Hz), –113.44 (q, *J* = 12.6 Hz).
HRMS (ESI) *m/z* calcd. for $C_{19}H_{16}F_4NO$ $[M + H]^+$ 350.1163, found 350.1159.
HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. λ = 254 nm, t(major) = 7.9 min, t(minor) = 9.6 min, 95% ee (97.4:2.6 er).



42

(*R*)-5-(3,5-Bis(trifluoromethyl)phenyl)-2-(1-phenylpropyl)oxazole

According to the general procedure with azole **S2p** (28.1 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (59.7 mg, 0.30 mmol, 3.0 equiv), and CuI (1.90 mg, 0.010 mmol, 10 mol%) at room temperature for 3 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **42** (14 mg) in 35% isolated yield.

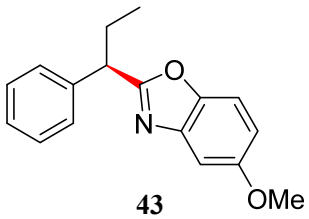
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.0 (d, *J* = 1.5 Hz, 2H), 7.80 (s, 1H), 7.49 (s, 1H), 7.39 (s, 2H), 7.38 (d, *J* = 1.9 Hz, 2H), 7.32 – 7.29 (m, 1H), 4.11 (t, *J* = 7.8 Hz, 1H), 2.42 – 2.31 (m, 1H), 2.18 – 2.06 (m, 1H), 1.00 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 148.3, 139.8, 132.4 (q, *J* = 33.6 Hz), 130.1, 128.8, 127.9, 127.3, 124.6, 123.8 (q, *J* = 4.3 Hz), 123.0 (q, *J* = 272.4 Hz), 121.4 – 121.3 (m), 47.5, 28.0, 12.3.

^{19}F NMR (376 MHz, $CDCl_3$) δ –63.11.

HRMS (ESI) *m*/*z* calcd. for $C_{20}H_{16}F_6NO$ $[M + H]^+$ 400.1131, found 400.1128.

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 99/1, flow rate 1.0 mL/min. λ = 254 nm, t(major) = 12.2 min, t(minor) = 14.3 min, 85% ee (92.6:7.4 er).



43

(*R*)-5-Methoxy-2-(1-phenylpropyl)benzo[*d*]oxazole

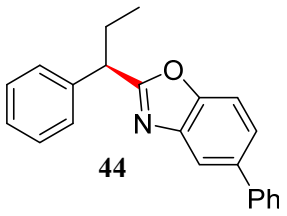
According to the general procedure with azole **S2q** (14.9 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (49.8 mg, 0.25 mmol, 2.5 equiv), and $Cu(acac)_2$ (2.62 mg, 0.010 mmol, 10 mol%) for 4 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **43** (8.0 mg) in 30% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.39 (m, 2H), 7.37 – 7.35 (m, 2H), 7.33 (d, *J* = 2.1 Hz, 1H), 7.30 – 7.27 (m, 1H), 7.23 (d, *J* = 2.6 Hz, 1H), 6.90 (dd, *J* = 8.9, 2.6 Hz, 1H), 4.12 (t, *J* = 7.8 Hz, 1H), 3.86 (s, 3H), 2.47 – 2.36 (m, 1H), 2.21 – 2.10 (m, 1H), 1.00 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 157.0, 142.0, 139.9, 128.7, 128.0, 127.3, 113.0, 110.6, 103.0, 56.0, 48.0, 27.6, 12.3.

HRMS (ESI) *m*/*z* calcd. for $C_{17}H_{18}NO_2$ $[M + H]^+$ 268.1332, found 268.1330.

HPLC condition: Chiralcel OJ, *n*-hexane/*i*-PrOH = 93/7, flow rate 0.5 mL/min. λ = 254 nm, t(minor) = 17.7 min, t(major) = 23.1 min, 91% ee (95.4:4.6 er).



44

(*R*)-5-Phenyl-2-(1-phenylpropyl)benzo[*d*]oxazole

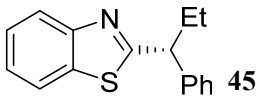
According to the general procedure with azole **S2r** (19.5 mg, 0.10 mmol, 1.0 equiv) and alkyl bromide **1a** (39.8 mg, 0.20 mmol, 2.0 equiv) for 5 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **44** (11.0 mg) in 35% isolated yield (63% yield based on recovered starting material).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (t, *J* = 1.2 Hz, 1H), 7.63 – 7.61 (m, 2H), 7.53 (brs, 2H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.44 – 7.42 (m, 2H), 7.40 – 7.35 (m, 3H), 7.31 – 7.27 (m, 1H), 4.18 (t, *J* = 7.7 Hz, 1H), 2.52 – 2.41 (m, 1H), 2.25 – 2.13 (m, 1H), 1.03 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 168.8, 150.4, 141.9, 141.2, 139.8, 138.1, 128.9, 128.8, 128.0, 127.5, 127.3, 127.2, 124.2, 118.4, 110.5, 48.0, 27.6, 12.3.

HRMS (ESI) *m*/*z* calcd. for $C_{22}H_{20}NO$ $[M + H]^+$ 314.1539, found 314.1537.

HPLC condition: Chiralcel OJ, *n*-hexane /*i*-PrOH = 93/7, flow rate 0.5 mL/min. λ = 254 nm, t(minor) = 22.3 min, t(major) = 33.7 min, 93% ee (96.5:3.5 er).



45

(*R*)-2-(1-Phenylpropyl)benzo[*d*]thiazole

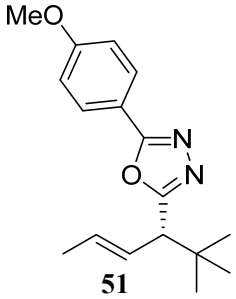
According to the general procedure with benzo[*d*]thiazole (13.5 mg, 0.10 mmol, 1.0 equiv), alkyl bromide **1a** (49.8 mg, 0.25 mmol, 2.5 equiv), and CuI (1.90 mg, 0.010 mmol, 10 mol%) at room temperature for 4 d, the reaction mixture was purified by column chromatography on silica gel to give the corresponding product **45** (3.0 mg) in 12% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (dt, *J* = 8.2, 0.8 Hz, 1H), 7.79 (d, 1H), 7.46 – 7.39 (m, 3H), 7.36 – 7.29 (m, 3H), 7.28 – 7.24 (m, 1H), 4.29 (t, *J* = 7.7 Hz, 1H), 2.50 – 2.39 (m, 1H), 2.26 – 2.15 (m, 1H), 0.99 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.4, 153.1, 141.7, 135.2, 128.7, 128.1, 127.3, 125.8, 124.7, 122.9, 121.5, 52.8, 28.9, 12.5.

HRMS (ESI) *m*/*z* calcd. for $C_{17}H_{16}NS$ $[M + H]^+$ 254.0998, found 254.0997.

HPLC condition: Chiralcel OJ, *n*-hexane/*i*-PrOH = 98/2, flow rate 1.0 mL/min. λ = 254 nm, t(major) = 13.8 min, t(mnior) = 16.0 min, 87% ee (93.7:6.3 er).



51

(*R*,*E*)-2-(2,2-Dimethylhex-4-en-3-yl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole

According to the general procedure with azole **2a** (17.6 mg, 0.10 mmol, 1.0 equiv), alkyl bromides **S1y** and **S1y'** (2:1, 47.5 mg, 0.25 mmol, 2.5 equiv), CuI (1.90 mg, 0.010 mmol, 10 mol%), and **L*5** (12.5 mg, 0.015 mmol, 15 mol%) for 4 d, the

reaction mixture was purified by column chromatography on silica gel to give the corresponding product **51** (20.0 mg) in 70% isolated yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.9 Hz, 2H), 7.01 (d, *J* = 9.0 Hz, 2H), 5.80 – 5.73 (m, 1H), 5.68 – 5.59 (m, 1H), 3.89 (s, 3H), 3.51 (d, *J* = 9.3 Hz, 1H), 1.74 (dd, *J* = 6.3, 1.5 Hz, 3H), 1.03 (s, 9H).

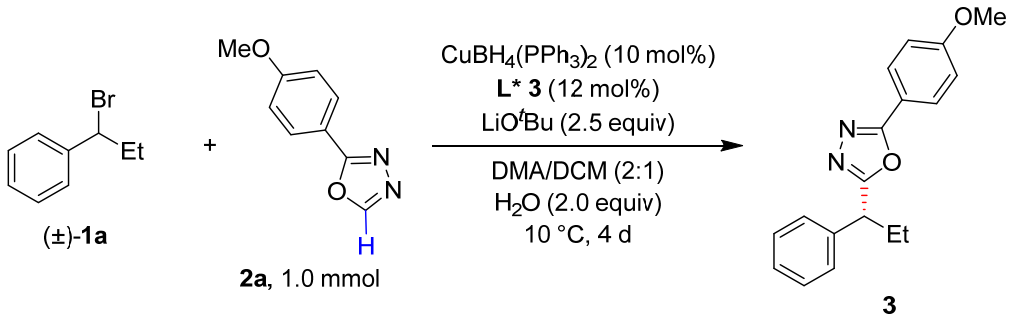
^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 164.3, 162.1, 130.3, 128.5, 125.9, 116.7, 114.4, 55.5, 51.6, 34.4, 27.7, 18.0.

HRMS (ESI) *m*/*z* calcd. for $C_{17}H_{23}N_2O_2$ $[M + H]^+$ 287.1754, found 295.1753.

HPLC condition: Chiralcel IA, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 15.2 min, t(major) = 18.7 min, 33% ee (66.6:33.4 er).

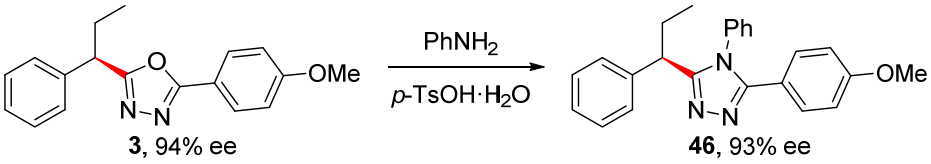
Preparative scale reaction and transformation

Preparative scale reaction

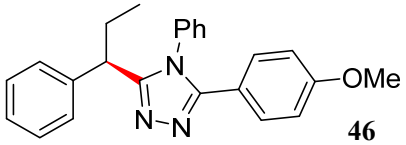


To a flame-dried Schlenk tube equipped with a magnetic stir bar were added $CuBH_4(PPh_3)_2$ (60.3 mg, 0.10 mmol, 10 mol%), **L*3** (97.4 mg, 0.12 mmol, 12 mol%), LiO^tBu (200 mg, 2.5 mmol, 2.5 equiv), and azole **2a** (176 mg, 1.0 mmol, 1.0 equiv). The tube was evacuated and backfilled with argon for three times and then, alkyl bromide **1a** (498 mg, 2.5 mmol, 2.5 equiv), water (36 μL, 2.0 mmol, 2.0 equiv), and anhydrous *N,N*-dimethylacetamide (7.0 mL) and dichloromethane (3.0 mL) were sequentially added. The reaction mixture was stirred at room temperature for 30 min and then stirred at 10 °C for 4 d. The resulting reaction mixture was diluted with 50 mL ethyl acetate and washed with brine (30 mL × 3). The organic layer was dried over anhydrous Na_2SO_4 and filtered through a pad of celite. The organic solvent was removed under vacuum and the residue was purified by column chromatography on silica gel to afford the desired product **3** (224 mg, 76% yield, 94% ee).

Transformation



To a flame-dried Schlenk tube equipped with a magnetic stir bar were charged with **3** (30 mg, 0.10 mmol, 1.0 equiv), aniline (0.10 mL, 1.1 mmol, 11 equiv), and *p*-toluenesulfonic acid monohydrate (7.6 mg, 0.04 mmol, 0.40 equiv). The resulting reaction mixture was stirred at 80 °C for 4 d. After completion, the reaction mixture was purified by column chromatography on silica gel to afford **46** (33 mg, 90% yield).



(*R*)-3-(4-Methoxyphenyl)-4-phenyl-5-(1-phenylpropyl)-4*H*-1,2,4-triazole

46, 33 mg, 90% yield.

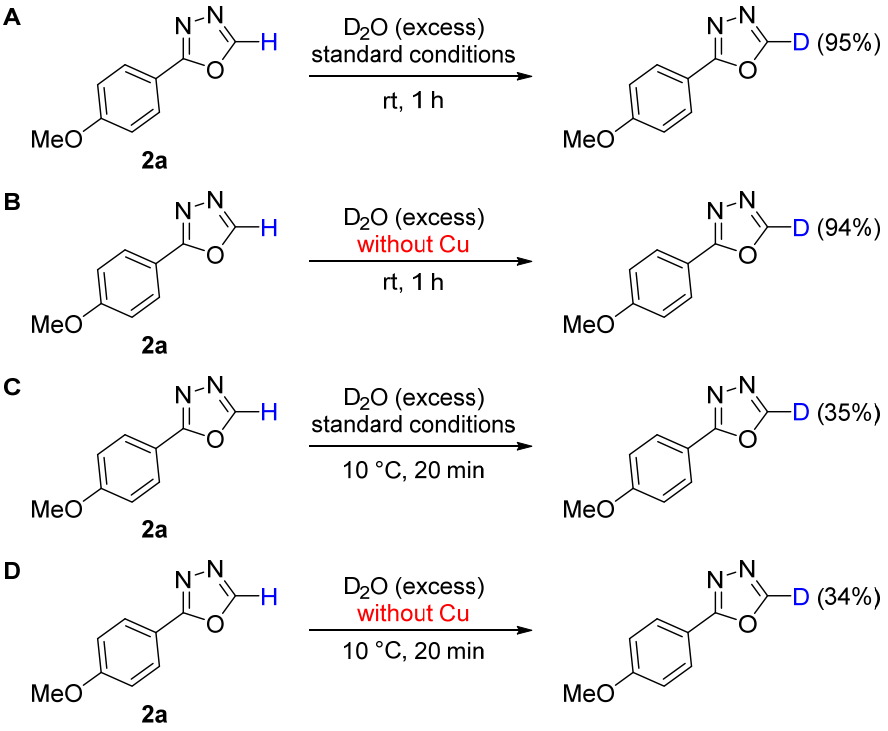
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.42 (m, 2H), 7.32 – 7.27 (m, 3H), 7.20 – 7.14 (m, 4H), 7.02 – 6.99 (m, 2H), 6.74 – 6.72 (m, 2H), 6.52 (br s, 1H), 3.73 (s, 3H), 3.57 (t, *J* = 7.8 Hz, 1H), 2.50 – 2.39 (m, 1H), 2.16 – 2.04 (m, 1H), 0.90 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 160.4, 157.2, 153.8, 140.9, 134.9, 129.7, 129.6, 129.5,

128.4, 128.0, 126.8, 119.5, 113.8, 55.2, 44.7, 28.6, 12.5.
HRMS (ESI) *m*/*z* calcd. for $C_{24}H_{24}N_3O$ $[M + H]^+$ 370.1914, found 370.1913.
HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 75/25, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 22.3 min, t(major) = 26.2 min, 93% ee (96.5:3.5 er).

Mechanistic studies

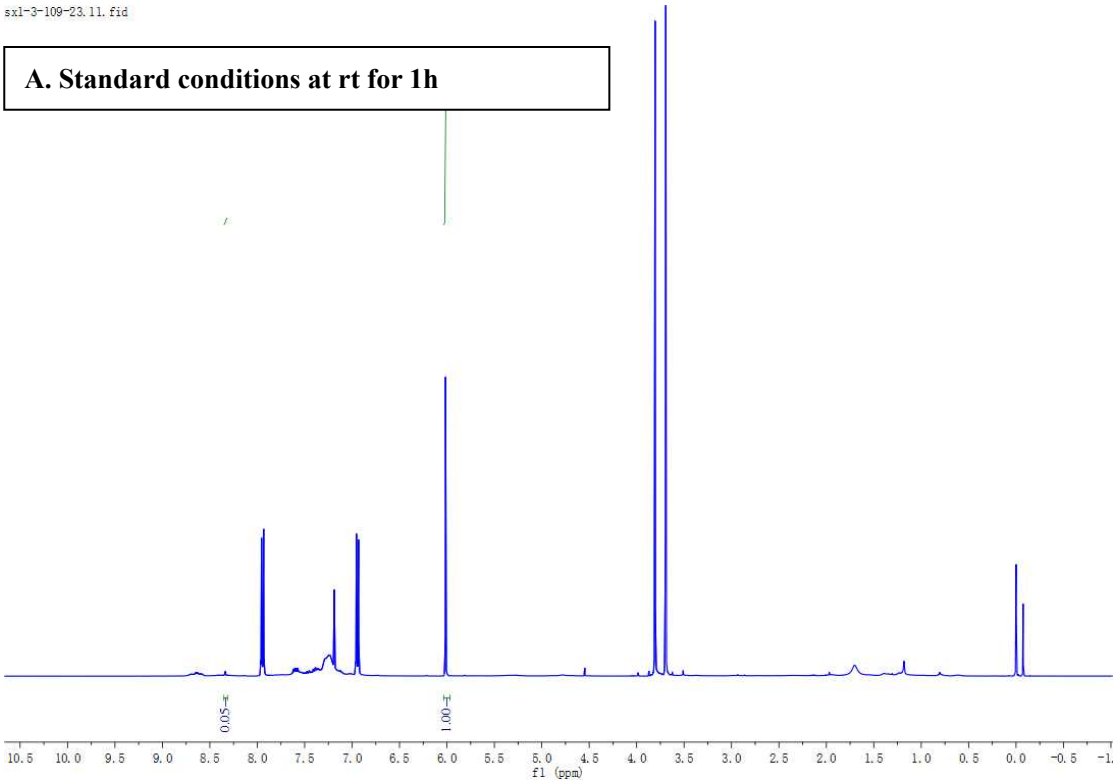
H/D exchange experiments



To a flame-dried Schlenk tube equipped with a magnetic stir bar were charged with **2a** (8.8 mg, 0.050 mmol, 1.0 equiv), $CuBH_4(PPh_3)_2$ (3.0 mg, 0.010 mmol, 10 mol% or 0 mmol), **L*3** (4.87 mg, 0.012 mmol, 12 mol%), and LiO^tBu (18 mg, 0.22 mmol, 4.5 equiv). The tube was evacuated and backfilled with argon for three times and then, D_2O (1.0 mL) and anhydrous *N,N*-dimethylacetamide (0.40 mL) and dichloromethane (0.20 mL) were sequentially added. The resulting reaction mixture was stirred at room temperature for 1 h or at 10 °C for 20 min. The resulting reaction mixture was diluted with 10 mL ethyl acetate, and washed with water (10 mL × 3). The organic layer was dried over anhydrous $NaSO_4$ and filtered through a pad of celite. The organic solvent was removed under vacuum. The ^1H NMR analysis of the residue showed 95%, 94%, 35% and 34% incorporation of deuterium at the C5–H of **2a**.

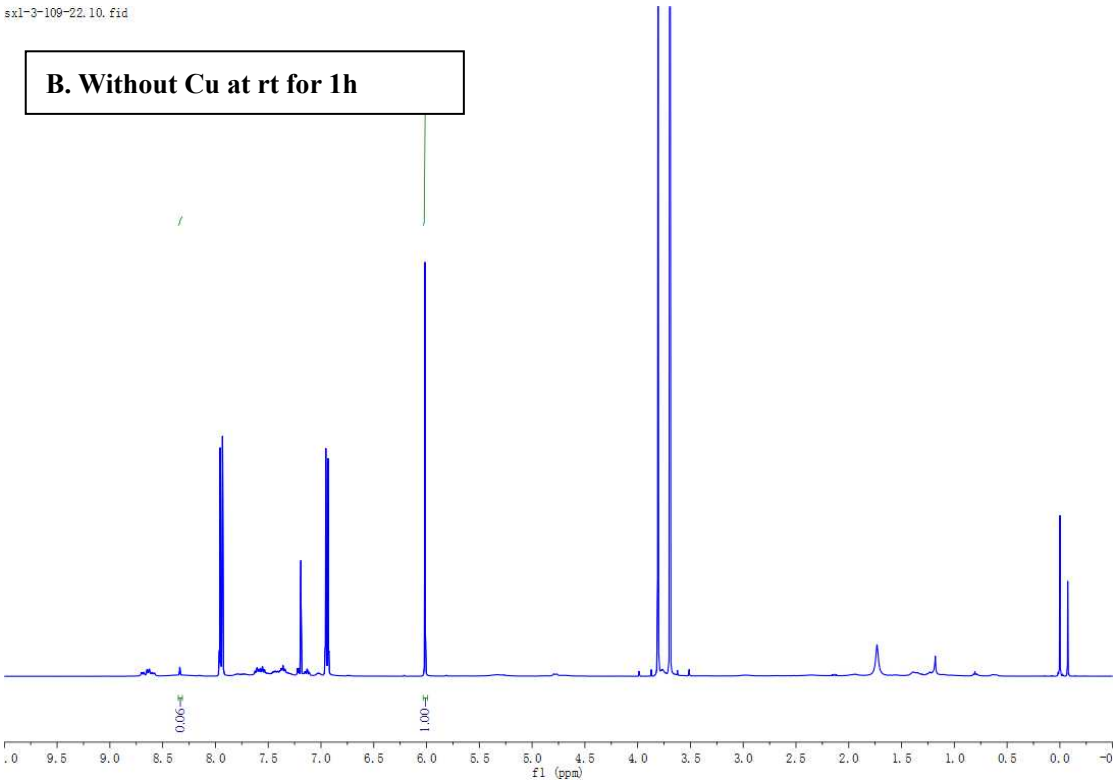
sxl-3-109-23.11.fid

A. Standard conditions at rt for 1h



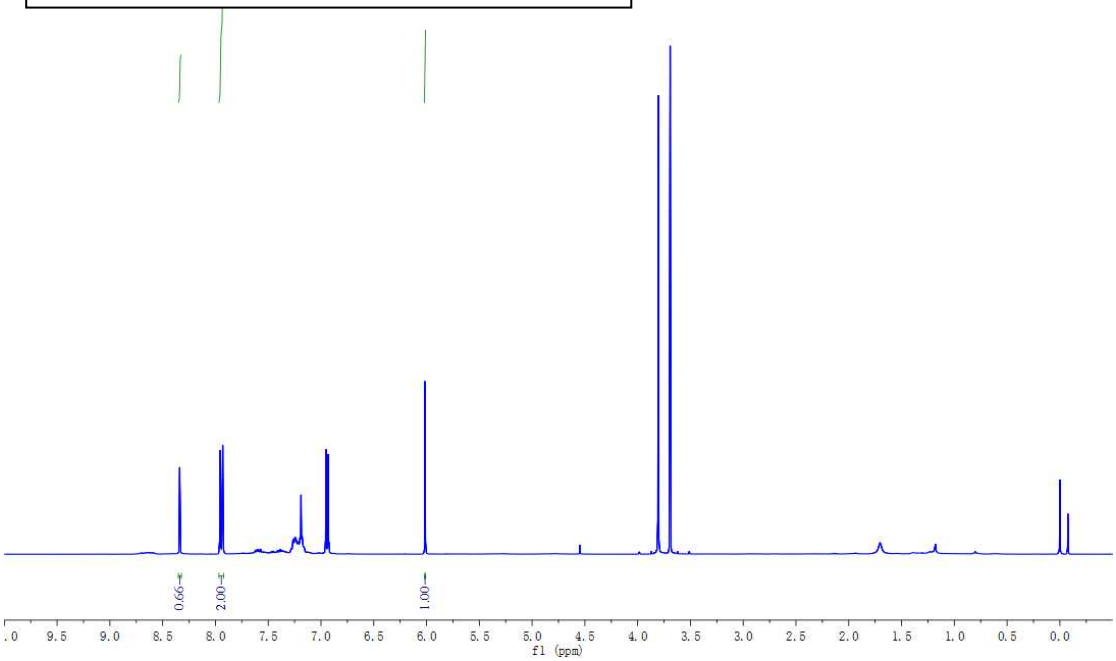
sxl-3-109-22.10.fid

B. Without Cu at rt for 1h



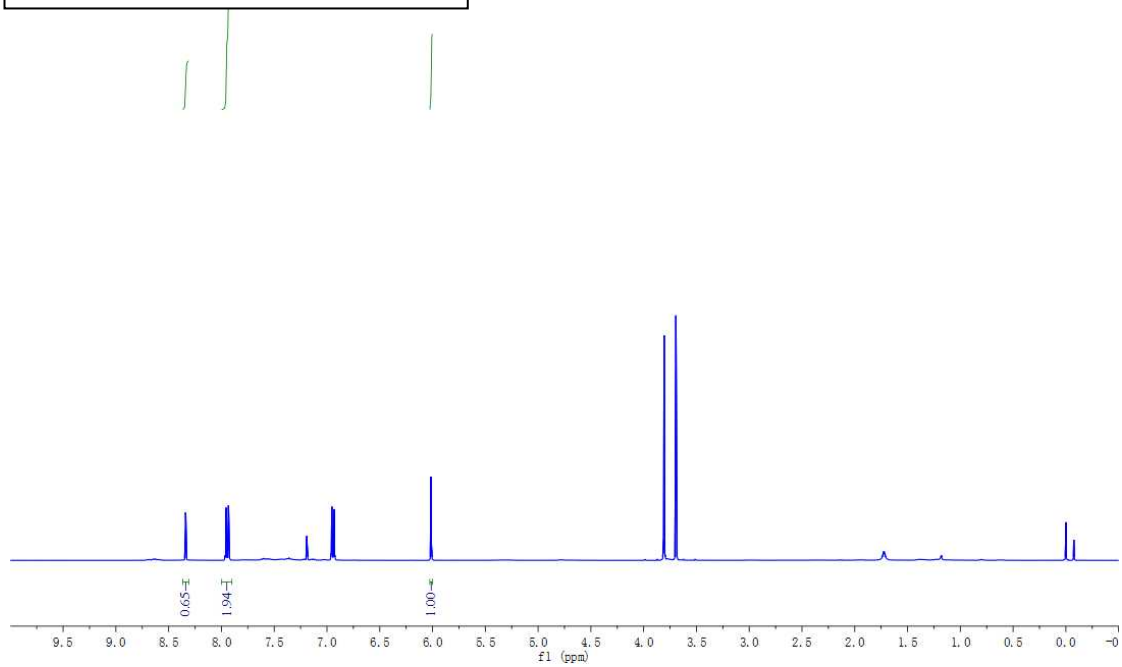
sxl-3-110-6.11.fid

C. Standard conditions at 10 °C for 20 min

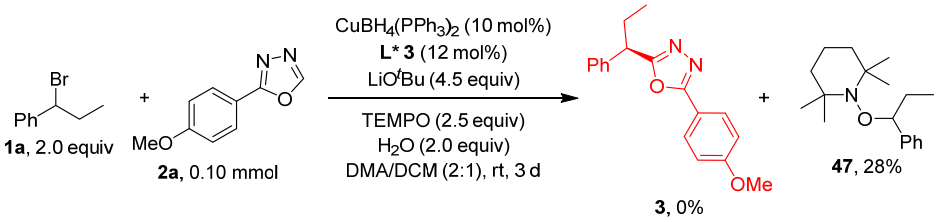


sxl-3-110-2.10.fid

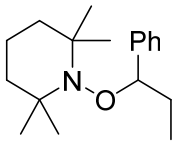
D. Without Cu at 10 °C for 20 min



Control experiment with TEMPO



To a flame-dried Schlenk tube equipped with a magnetic stir bar were charged with **2a** (17.6 mg, 0.10 mmol, 1.0 equiv), $CuBH_4(PPh_3)_2$ (6.03 mg, 0.01 mmol, 10 mol%), **L*3** (9.74 mg, 0.012 mmol, 12 mol%), LiO^{t}Bu (36 mg, 0.45 mmol, 4.5 equiv), and (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO) (39.1 mg, 0.25 mmol, 2.5 equiv). The tube was evacuated and backfilled with argon for three times and then, **1a** (39.8 mg, 0.20 mmol, 2.0 equiv), water (3.6 μL, 0.20 mmol, 2.0 equiv), and anhydrous *N,N*-dimethylacetamide (0.70 mL) and dichloromethane (0.35 mL) were sequentially added. The resulting reaction mixture was stirred at room temperature for 3 d. The resulting reaction mixture was diluted with 10 mL ethyl acetate and washed with water (10 mL × 3). The organic layer was dried over anhydrous $NaSO_4$ and filtered through a pad of celite. The organic solvent was removed under vacuum and the residue was purified by column chromatography on neutral alumina to afford **47** (7.7 mg, 28% yield).[1] The desired product **3** was not observed.



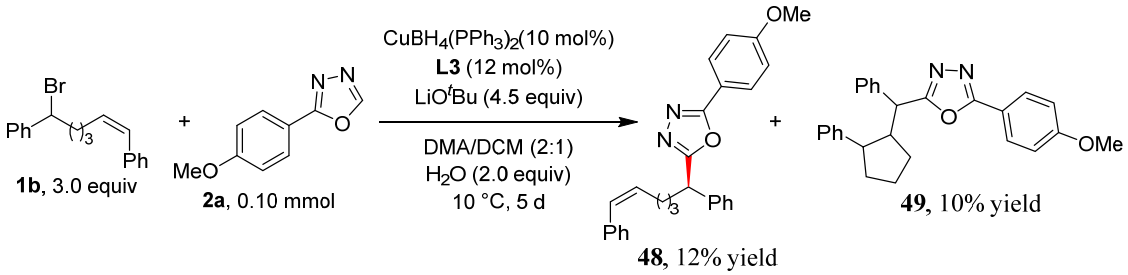
47

2,2,6,6-Tetramethyl-1-(1-phenylpropoxy)piperidine

47, 7.7 mg, 28% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38 – 7.33 (m, 4H), 7.29 – 7.24 (m, 1H), 4.57 (dd, J = 9.5, 3.9 Hz, 1H), 2.18 – 2.08 (m, 1H), 1.89 – 1.78 (m, 1H), 1.56 – 1.46 (m, 3H), 1.40 – 1.29 (m, 6H), 1.21 (br s, 3H), 1.04 (br s, 3H), 0.70 (t, J = 7.5 Hz, 3H), 0.62 (br s, 3H).

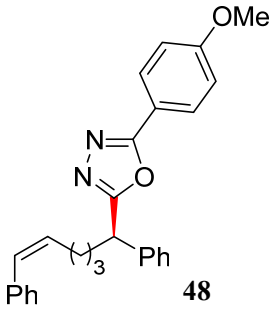
HRMS (ESI) *m/z* calcd. for $C_{18}H_{30}NO$ $[M + H]^+$ 276.2321, found 276.2318.

Radical clock experiment



To a flame-dried Schlenk tube equipped with a magnetic stir bar were charged with **2a** (17.6 mg, 0.10 mmol, 1.0 equiv), $CuBH_4(PPh_3)_2$ (6.03 mg, 0.010 mmol, 10 mol%), **L*3** (9.74 mg, 0.012 mmol, 12 mol%), and LiO^{t}Bu (36 mg, 0.45 mmol, 4.5 equiv). The tube was evacuated and backfilled with argon for three times and then, **1b** (94.6 mg, 0.30 mmol, 3.0 equiv), water (3.6 μL, 0.20 mmol, 2.0 equiv), and anhydrous

N,N-dimethylacetamide (0.70 mL) and dichloromethane (0.35 mL) were sequentially added. The resulting reaction mixture was stirred 10 °C for 5 d. The resulting reaction mixture was diluted with 10 mL ethyl acetae and washed with water (10 mL × 3). The organic layer was dried over anhydrous $NaSO_4$ and filtered through a pad of celite. The organic solvent was removed under vacuum and the residue was purified by column chromatography on silica gel to afford **48** (5.0 mg, 12% yield) and the desired product **49** (4.0 mg, 10% yield).



48

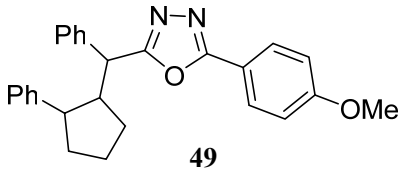
(*R,Z*)-2-(1,6-Diphenylhex-5-en-1-yl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.9 Hz, 2H), 7.36 – 7.34 (m, 5H), 7.33 – 7.29 (m, 2H), 7.26 – 7.22 (m, 3H), 7.00 (d, *J* = 8.9 Hz, 2H), 6.46 – 6.42 (m, 1H), 5.63 (dt, *J* = 11.7, 7.2 Hz, 1H), 4.21 (t, *J* = 7.8 Hz, 1H), 3.88 (s, 3H), 2.44 – 2.31 (m, 3H), 2.20 – 2.10 (m, 1H), 1.57 – 1.49 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 164.8, 162.2, 139.0, 137.5, 132.0, 129.5, 128.9, 128.7, 128.6, 128.2, 127.9, 127.5, 126.6, 116.5, 114.4, 55.4, 43.2, 33.7, 28.1, 27.6.

HRMS (ESI) *m/z* calcd. for $C_{27}H_{27}N_2O_2$ $[M + H]^+$ 411.2067, found 411.2063.

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min. λ = 254 nm, t (minor) =15.8 min, t (major) = 19.4 min, 88% ee (94:6 er).



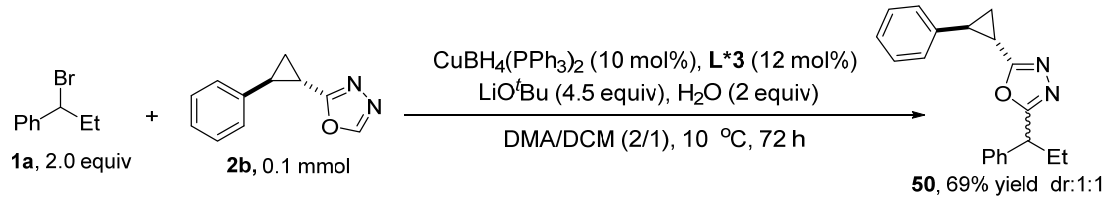
49

2-(4-Methoxyphenyl)-5-(phenyl(2-phenylcyclopentyl)methyl)-1,3,4-oxadiazole

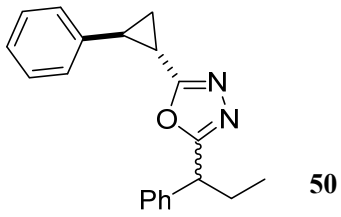
^{1}H NMR (400 MHz, $CDCl_3$) (the major diastereomer) δ 7.95 (d, *J* = 8.8 Hz, 2H), 7.27 – 7.24 (m, 2H), 7.19 – 7.14 (m, 5H), 7.11 – 7.07 (m, 1H), 7.00 (d, *J* = 8.9 Hz, 2H), 6.94 (d, *J* = 8.9 Hz, 2H), 4.23 (d, *J* = 8.7 Hz, 1H), 3.89 (s, 3H), 2.97 – 2.89 (m, 1H), 2.79 (q, *J* = 8.1 Hz, 1H), 2.17 – 2.11 (m, 1H), 2.08 – 2.00 (m, 1H), 1.89 – 1.72 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9, 164.6, 162.2, 145.6, 137.9, 128.6(0), 128.6(9), 128.4, 128.2, 127.3, 125.6, 116.5, 114.4, 55.5, 51.8, 50.5, 47.7, 36.2, 30.7, 24.8.

HRMS (ESI) *m/z* calcd. for $C_{27}H_{27}N_2O_2$ $[M + H]^+$ 411.2067, found 411.2062.



To a flame-dried Schlenk tube equipped with a magnetic stir bar were charged with **2b** (18.6 mg, 0.10 mmol, 1.0 equiv), $CuBH_4(PPh_3)_2$ (6.03 mg, 0.010 mmol, 10 mol%), **L*3** (9.74 mg, 0.012 mmol, 12 mol%), and LiO^{t}Bu (36 mg, 0.45 mmol, 4.5 equiv). The tube was evacuated and backfilled with argon for three times and then, **1a** (39.8 mg, 0.20 mmol, 2.0 equiv), water (3.6 μL, 0.20 mmol, 2.0 equiv), and anhydrous *N,N*-dimethylacetamide (0.70 mL) and dichloromethane (0.35 mL) were sequentially added. The resulting reaction mixture was stirred 10 °C for 3 d. The resulting reaction mixture was diluted with 10 mL ethyl acetae and washed with water (10 mL × 3). The organic layer was dried over anhydrous $NaSO_4$ and filtered through a pad of celite. The organic solvent was removed under vacuum and the residue was purified by column chromatography on silica gel to afford **50** (21.0 mg, 69% yield, dr: 1:1)



50

2-Phenylcyclopropyl)-5-(1-phenylpropyl)-1,3,4-oxadiazole

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.28 (m, 7H), 7.24 (t, *J* = 6.8 Hz, 1H), 7.15 (d, *J* = 7.3 Hz, 2H), 4.06 (t, *J* = 7.8 Hz, 1H), 2.65 – 2.57 (m, 1H), 2.39 – 2.36 (m, 1H), 2.34 – 2.27 (m, 1H), 2.15 – 2.04 (m, 1H), 1.77 – 1.70 (m, 1H), 1.59 – 1.53 (m, 1H), 0.98 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5(4), ***167.4(9)***, 139.5, 138.9(7), ***138.9(5)***, 128.8, 128.6, 127.9, 127.5, 126.7, 126.1, 45.1, 27.4(2), ***27.3(9)***, 26.6, ***26.5***, 17.1(1), ***17.0(6)***, 17.0(4), ***17.0(0)***, 12.1.

HRMS (ESI) *m/z* calcd. for $C_{20}H_{21}N_2O$ $[M + H]^+$ 305.1648, found 305.1645.

References

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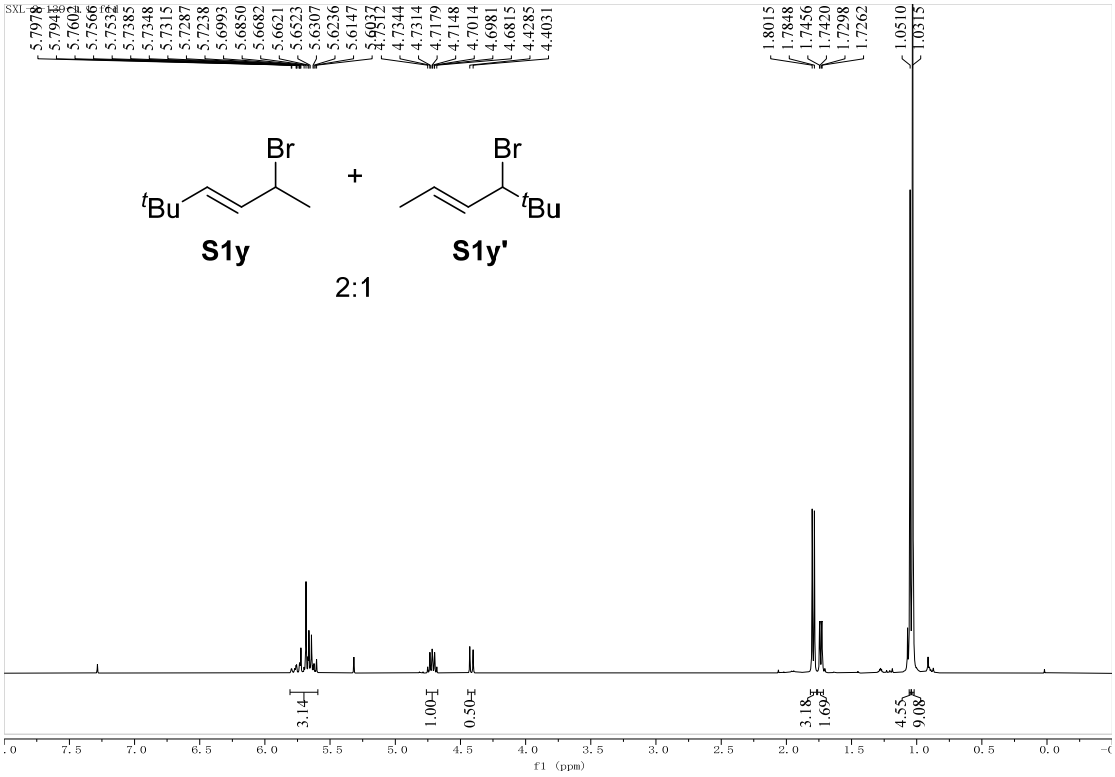
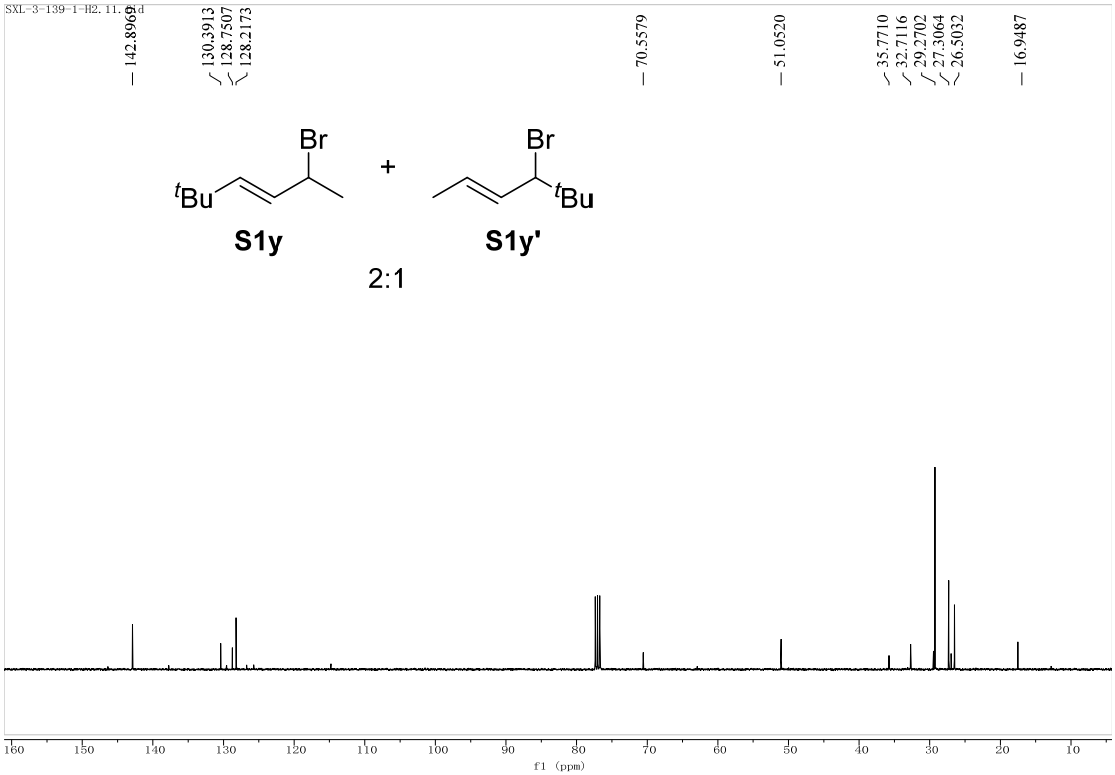
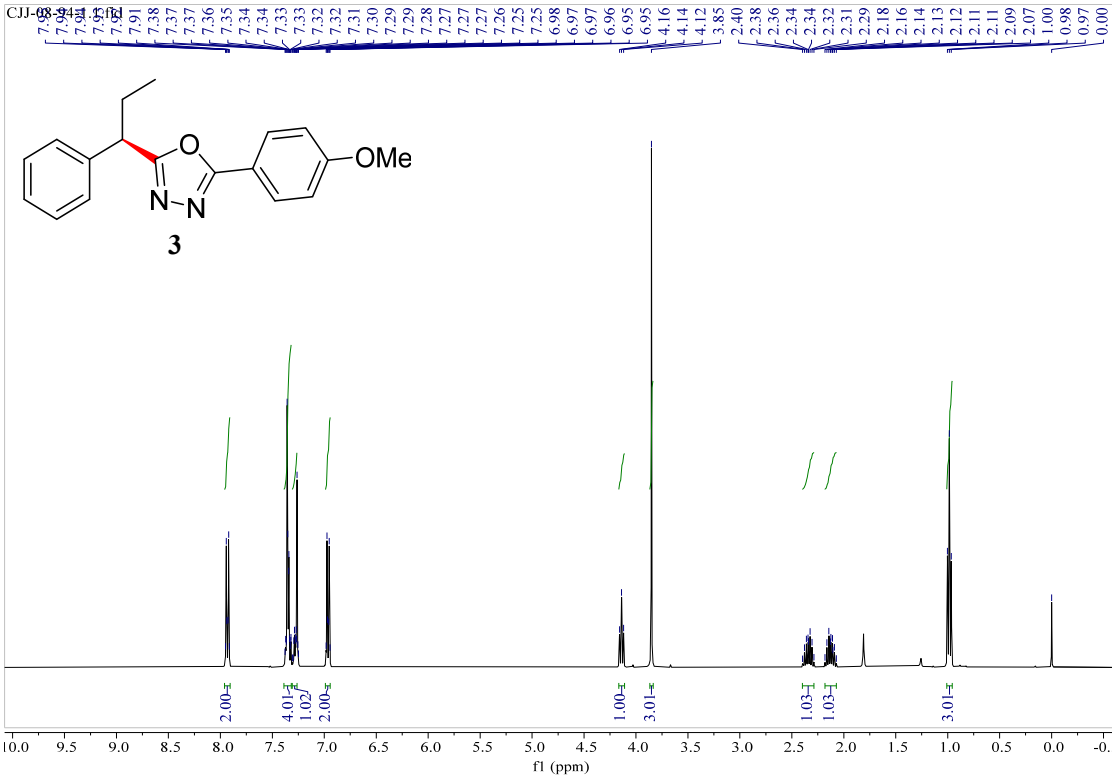
NMR spectra



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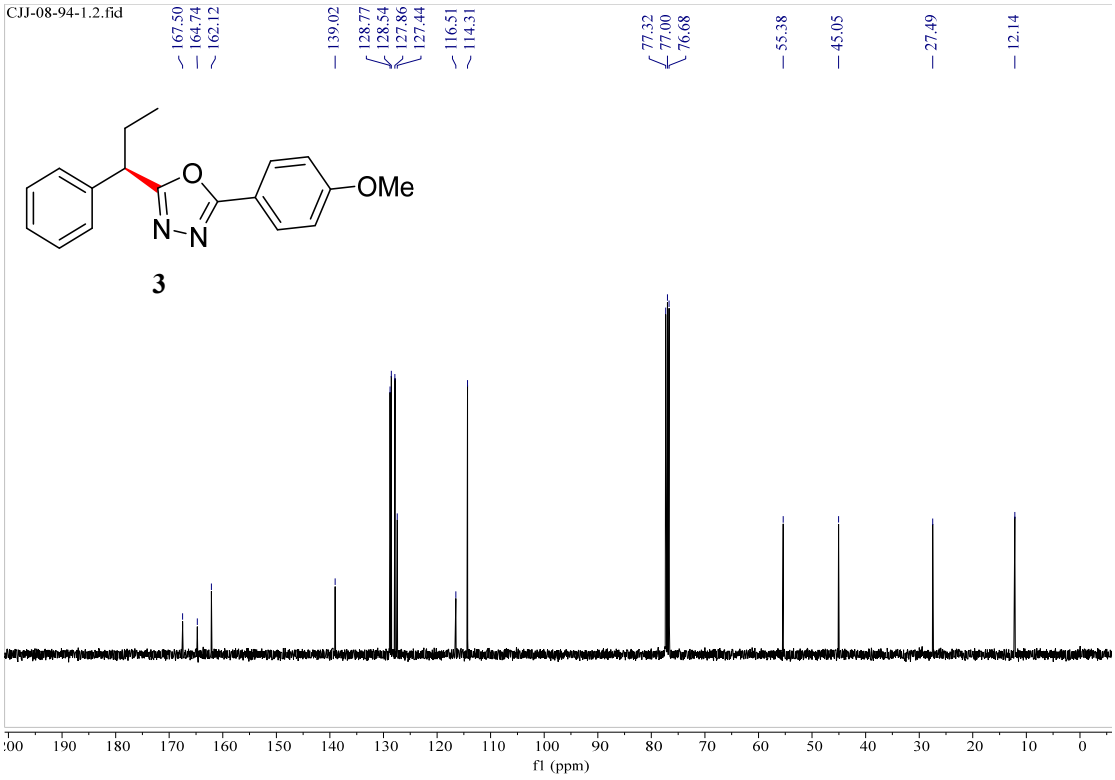
fl (ppm)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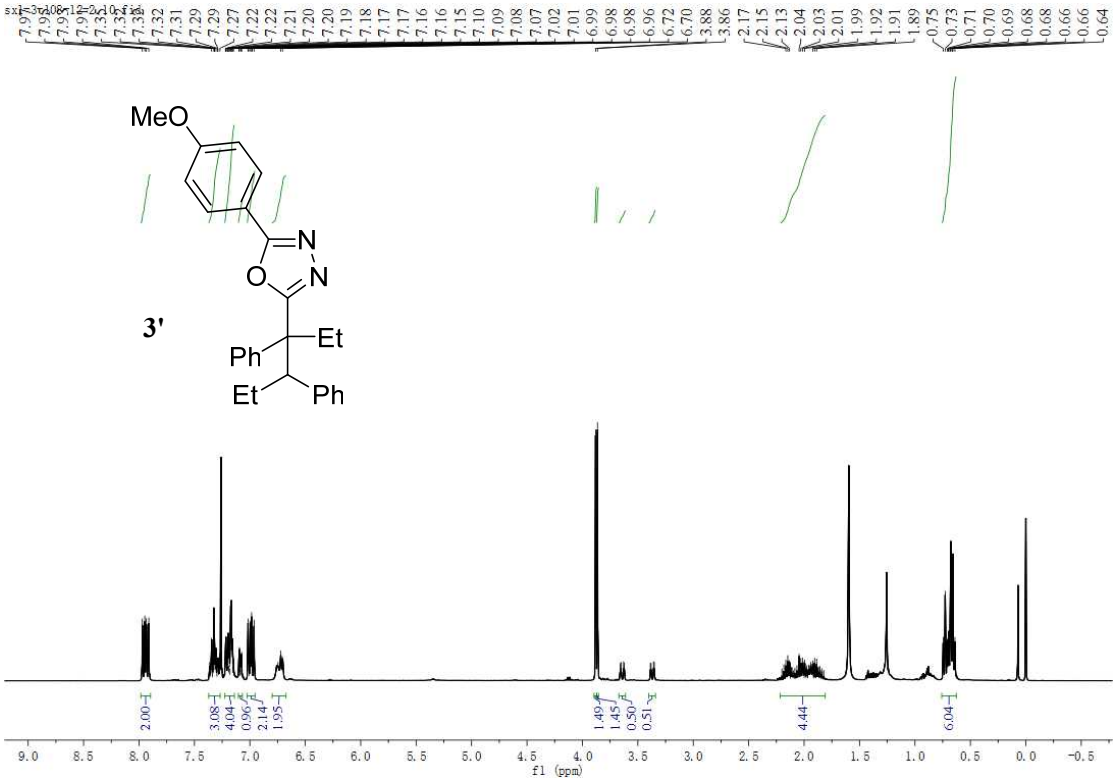
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Et
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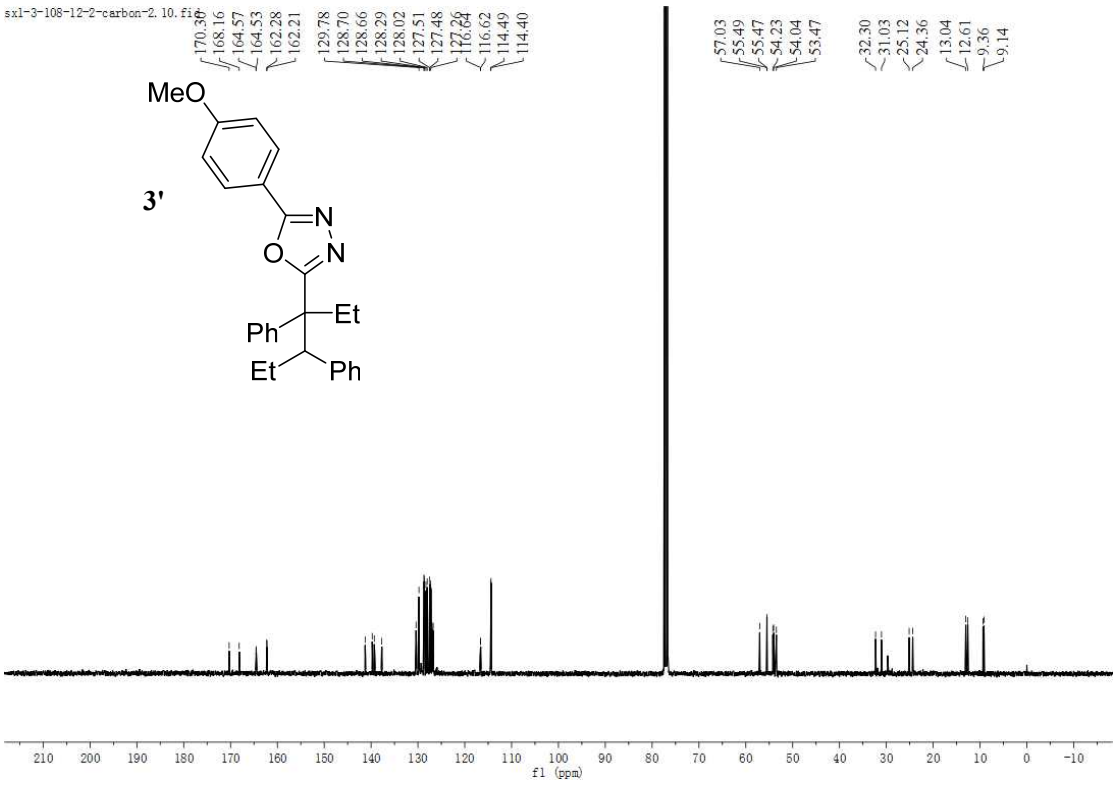
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3'
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Ph
Et
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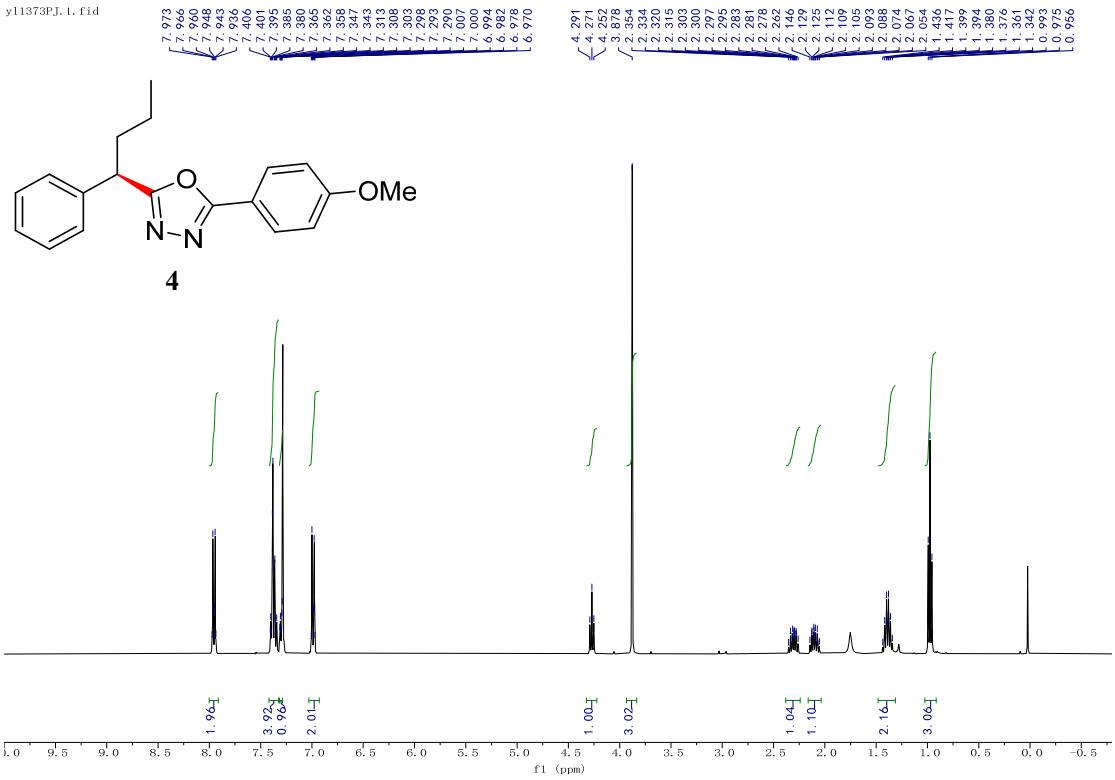
f1 (ppm)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OMe
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N
O
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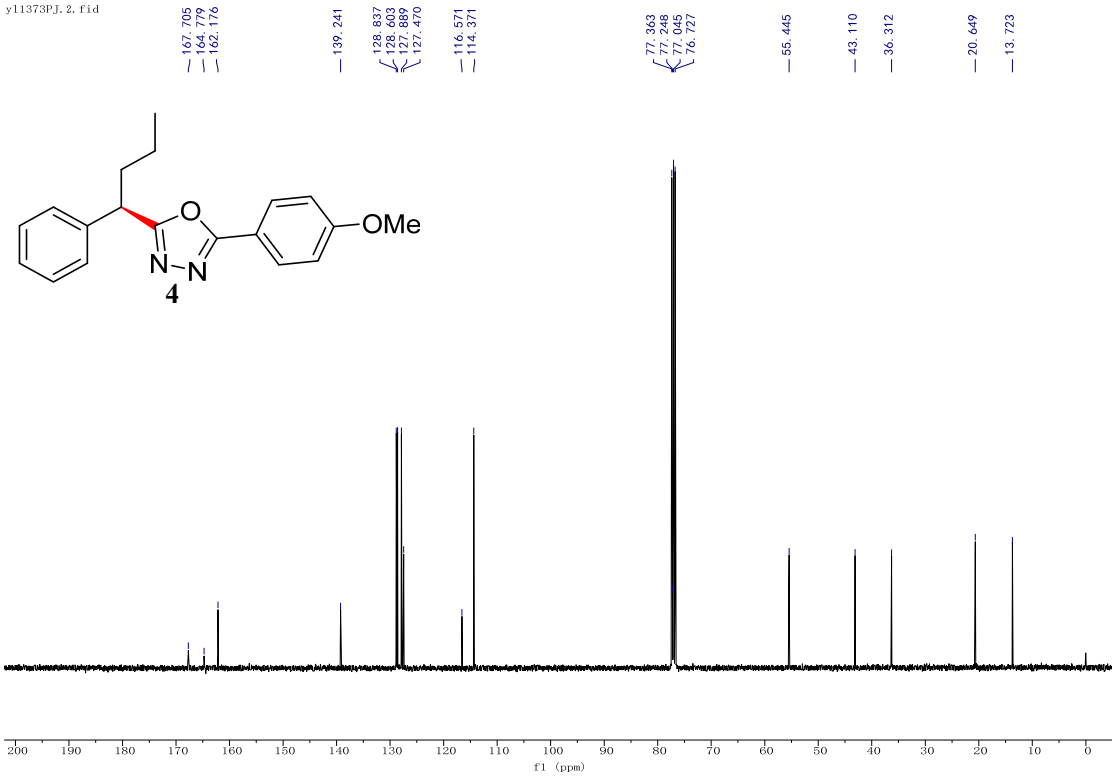
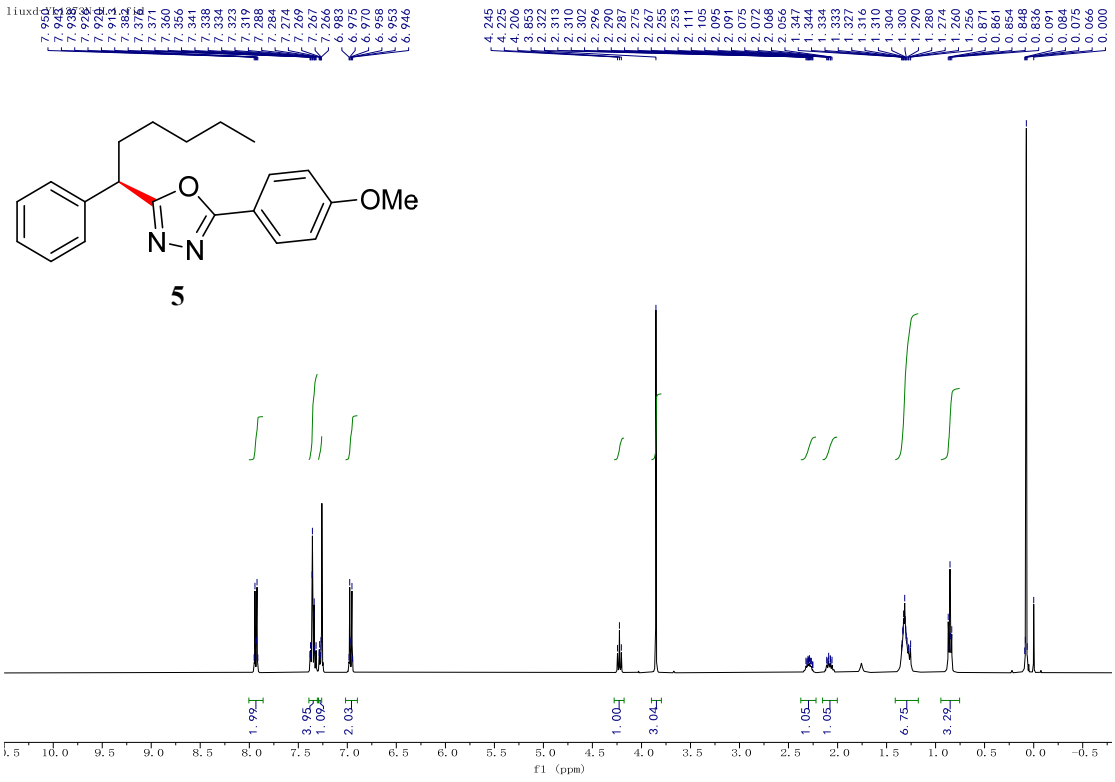
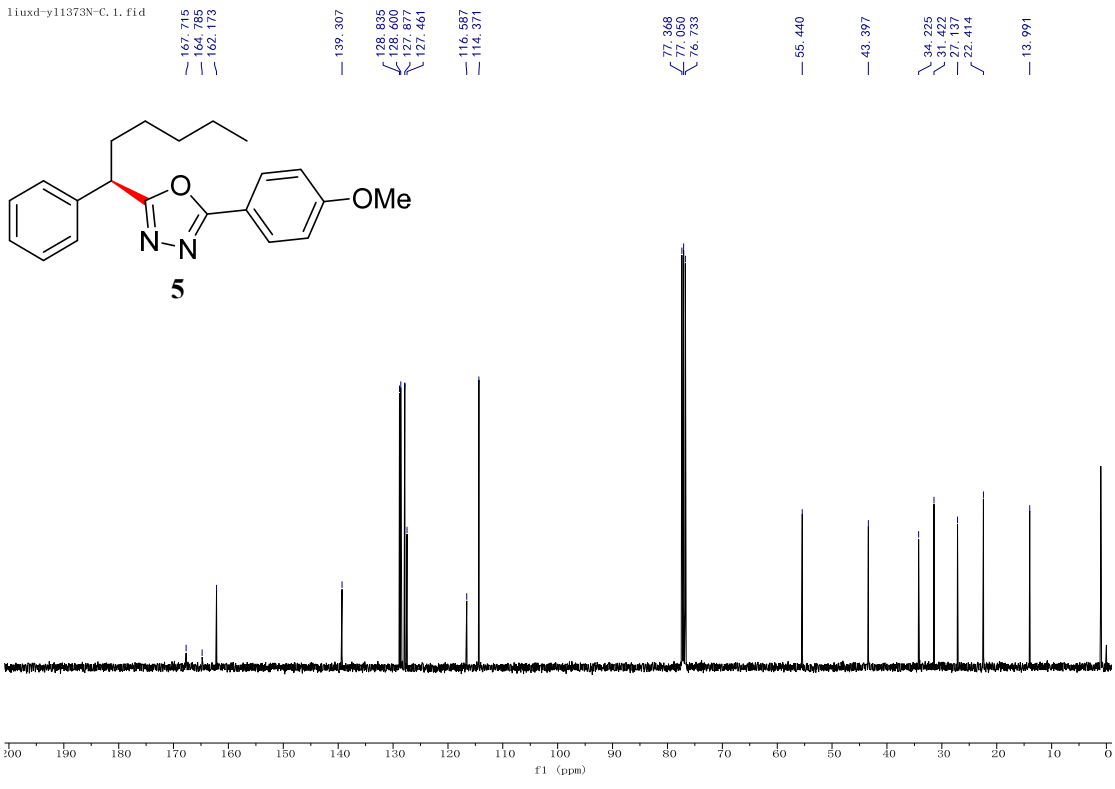
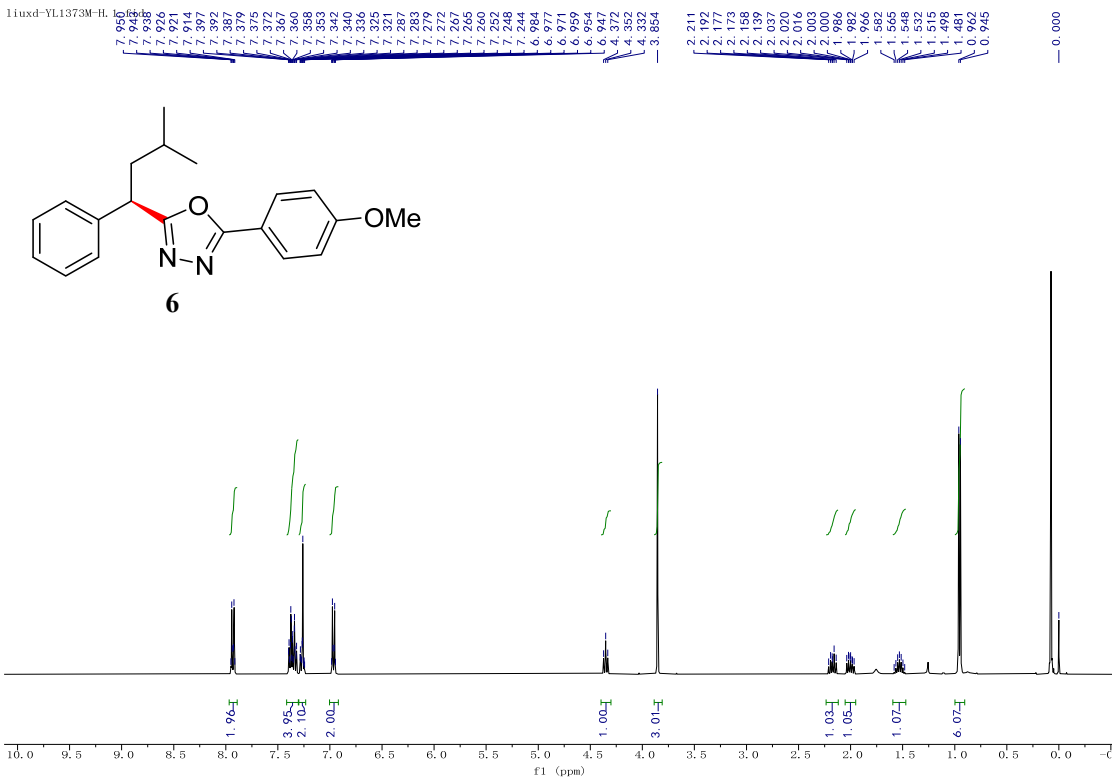
1.00
3.02
1.04
1.10
2.16
3.06
f1 (ppm)

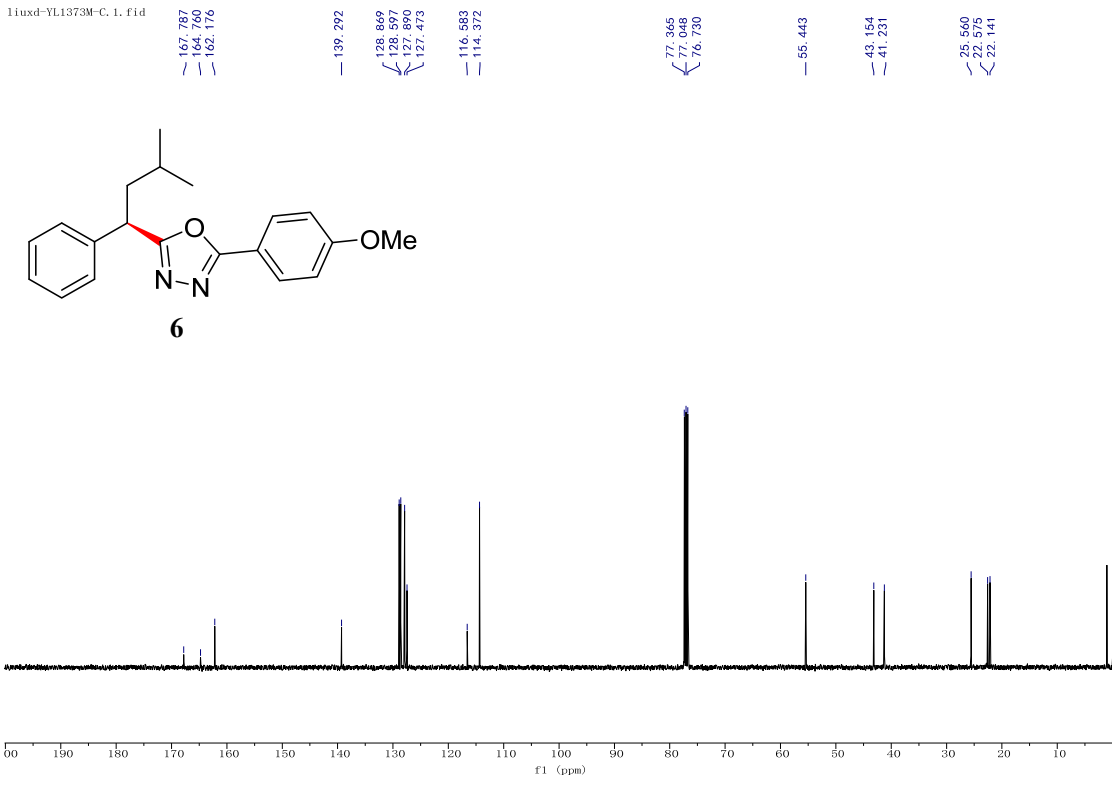

yl1373PJ.2.fid
167.705
164.779
162.176
139.241
128.837
128.603
127.889
127.470
116.571
114.371
77.363
77.248
77.045
76.727
55.445
43.110
36.312
20.649
13.723
OMe
N
N
O
4
f1 (ppm)


OMe
O
N
N
5
1.99
3.95
1.09
2.03
1.00
3.04
1.05
1.05
6.75
3.29
f1 (ppm)

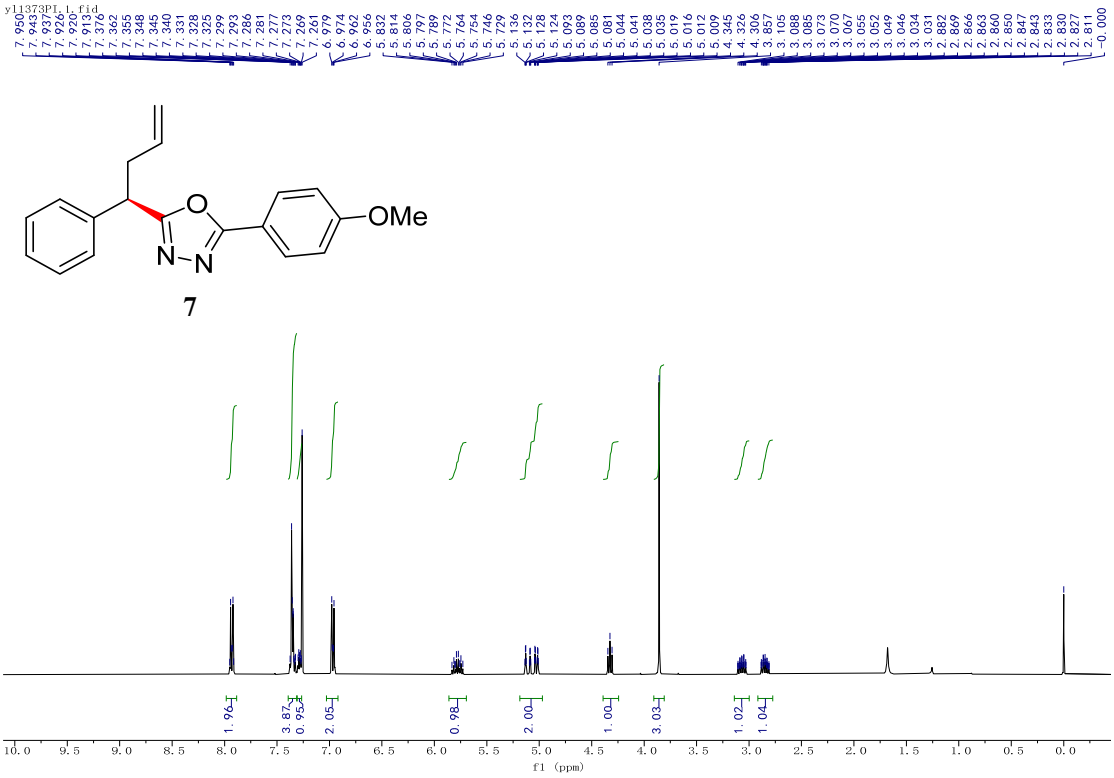

OMe
O
N
N
5
f1 (ppm)

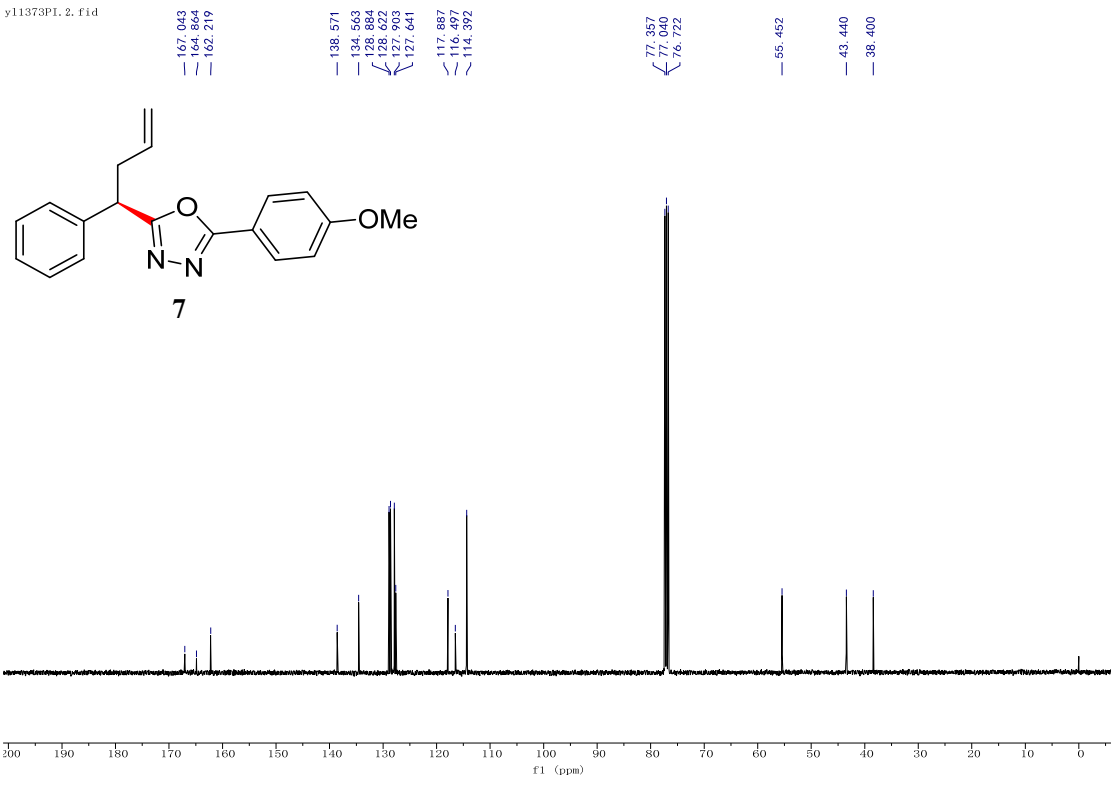

liuxd-YL1373M-H.1.fid
OMe
1.96
3.95
2.10
2.00
1.00
3.01
1.03
1.05
1.07
6.07
f1 (ppm)

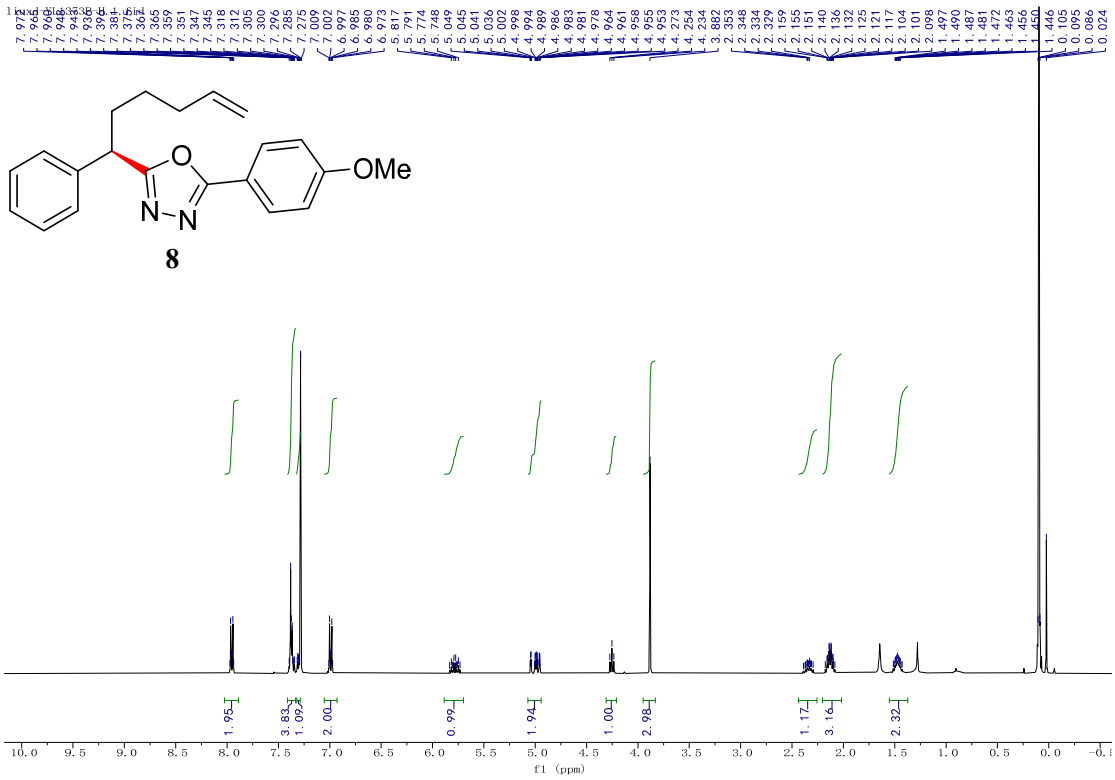
6

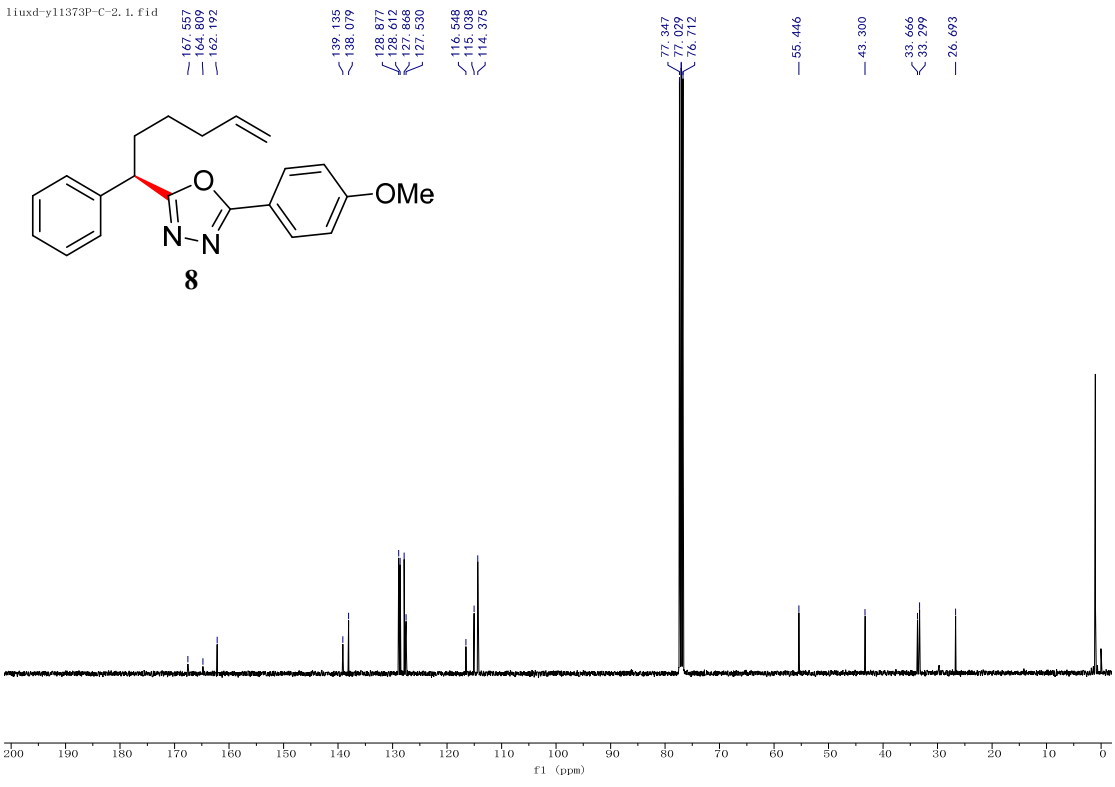

liuxd-YL1373M-C.1.fid
OMe
f1 (ppm)

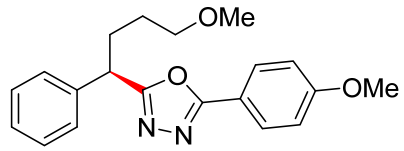
6


y11373PI.1.fid
OMe
7
1.96
3.87
0.95
2.05
0.98
2.00
1.00
3.03
1.02
1.04
f1 (ppm)

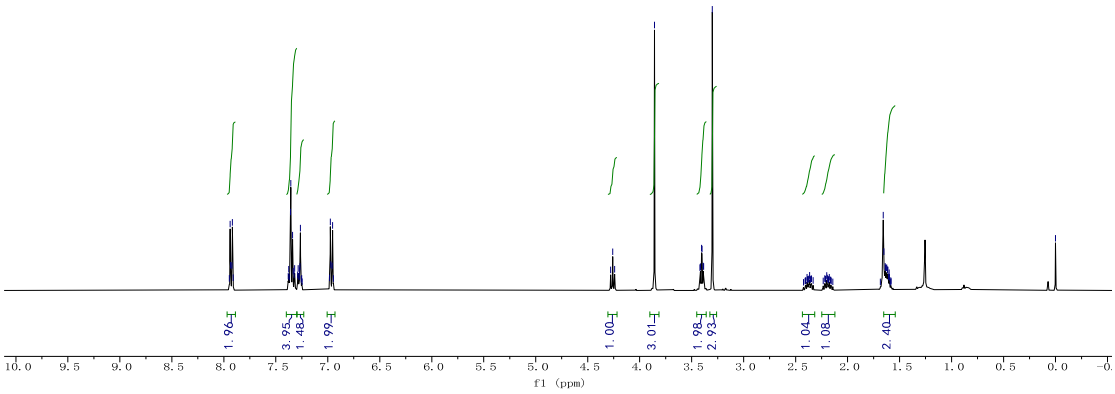

y11373PI.2.fid
167.043
164.864
162.219
138.571
134.563
128.884
128.622
127.903
127.641
117.887
116.497
114.392
77.357
77.040
76.722
55.452
43.440
38.400
OMe
7
f1 (ppm)

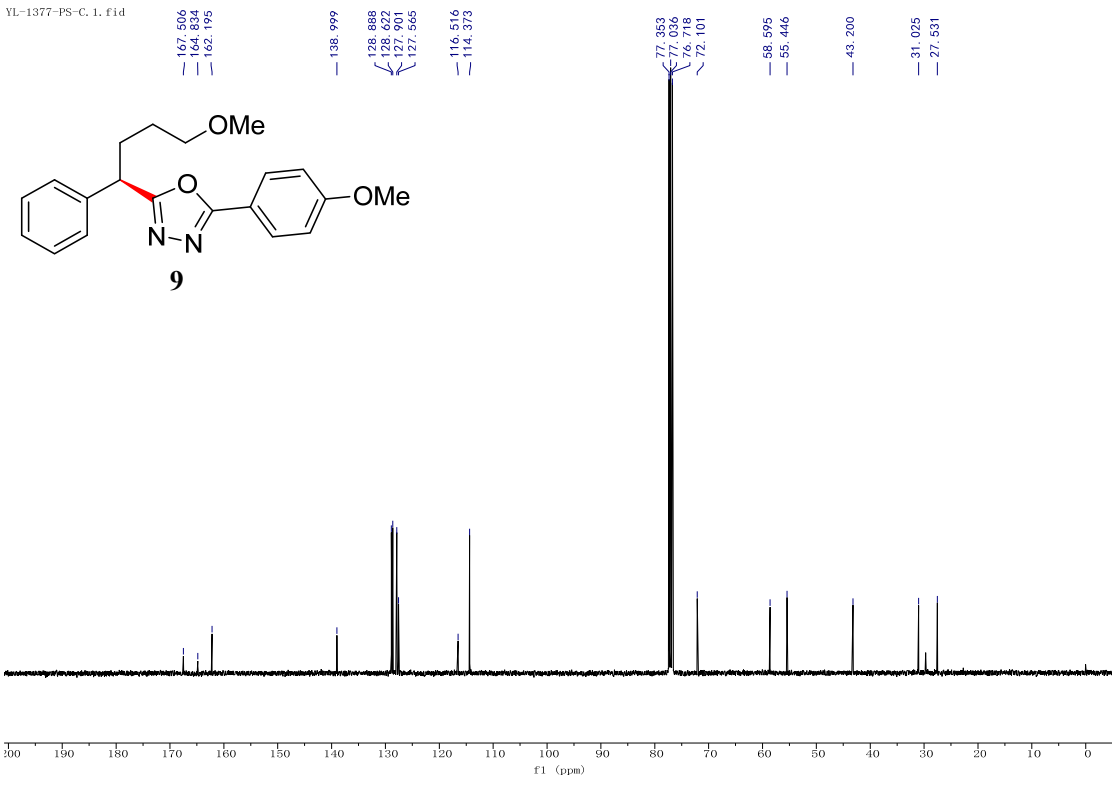

OMe
N
N
O
8
1.95
3.83
1.09
2.00
0.99
1.94
1.00
2.98
1.17
3.16
2.32
f1 (ppm)


liuxd-yl1373P-C-2.1.fid
167.557
164.809
162.192
139.135
138.079
128.877
128.612
127.868
127.530
116.548
115.038
114.375
77.347
77.029
76.712
55.446
43.300
33.666
33.299
26.693
OMe
N
N
O
8
f1 (ppm)


OMe
O
N
N
OMe

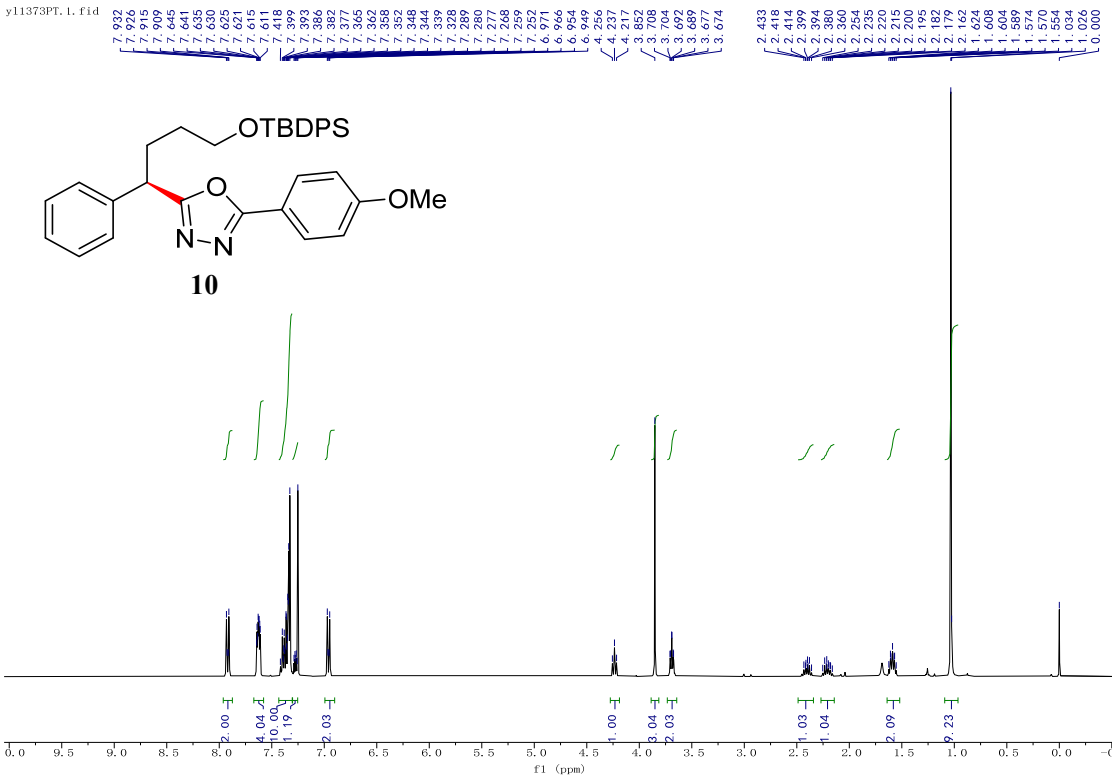
9

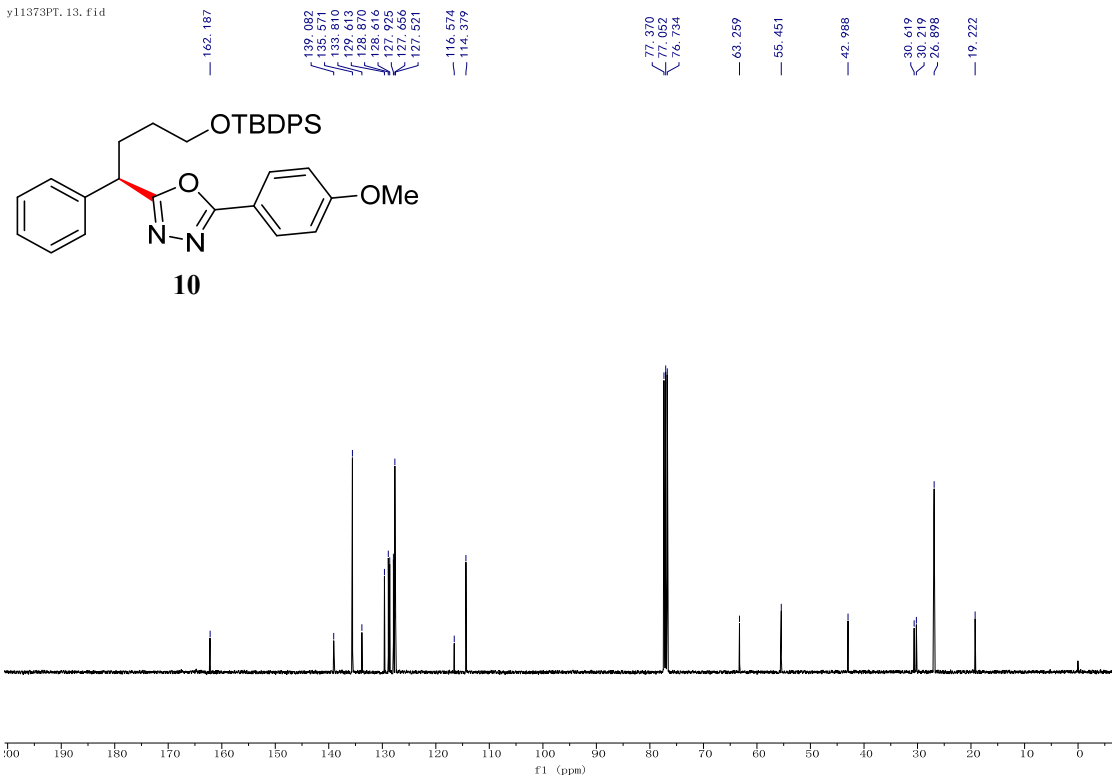

1.96
3.95
1.48
1.99
1.00
3.01
1.98
2.93
1.04
1.08
2.40
f1 (ppm)

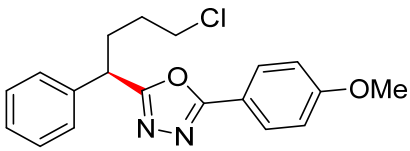

YL-1377-PS-C.1.fid
167.506
164.834
162.195
138.999
128.888
128.622
127.901
127.565
116.516
114.373
77.353
77.036
76.718
72.101
58.595
55.446
43.200
31.025
27.531
f1 (ppm)

OMe
O
N
N
OMe

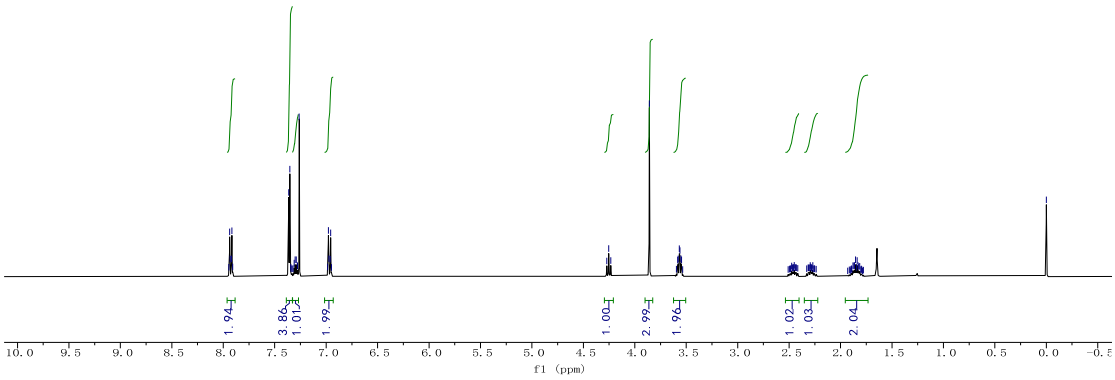
9


y11373PT.1.fid
OTBDPS
OMe
10
f1 (ppm)

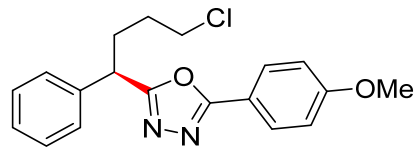

y11373PT.13.fid
OTBDPS
OMe
10
f1 (ppm)


Cl
O
N
N
OMe

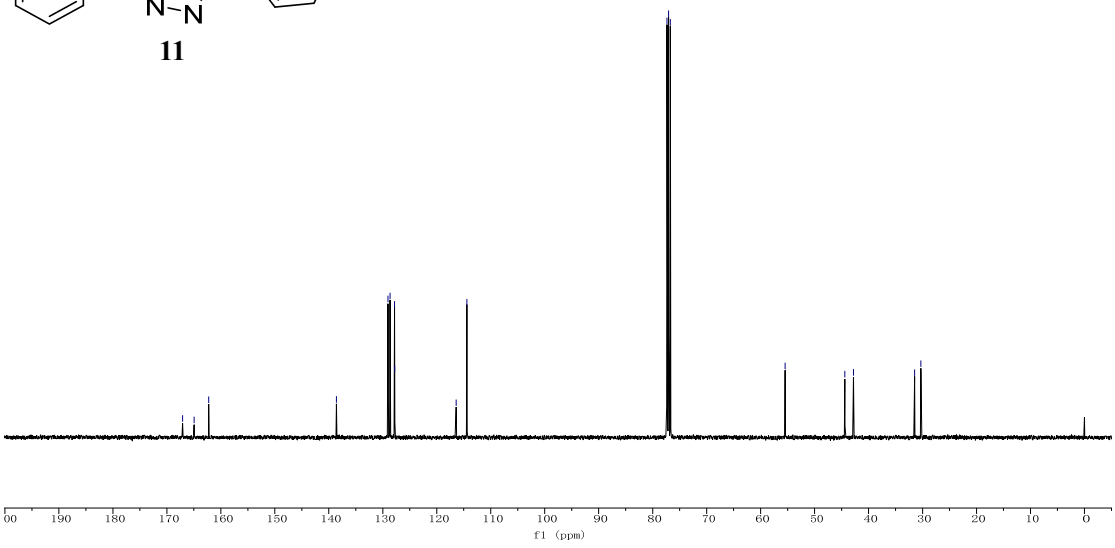
11

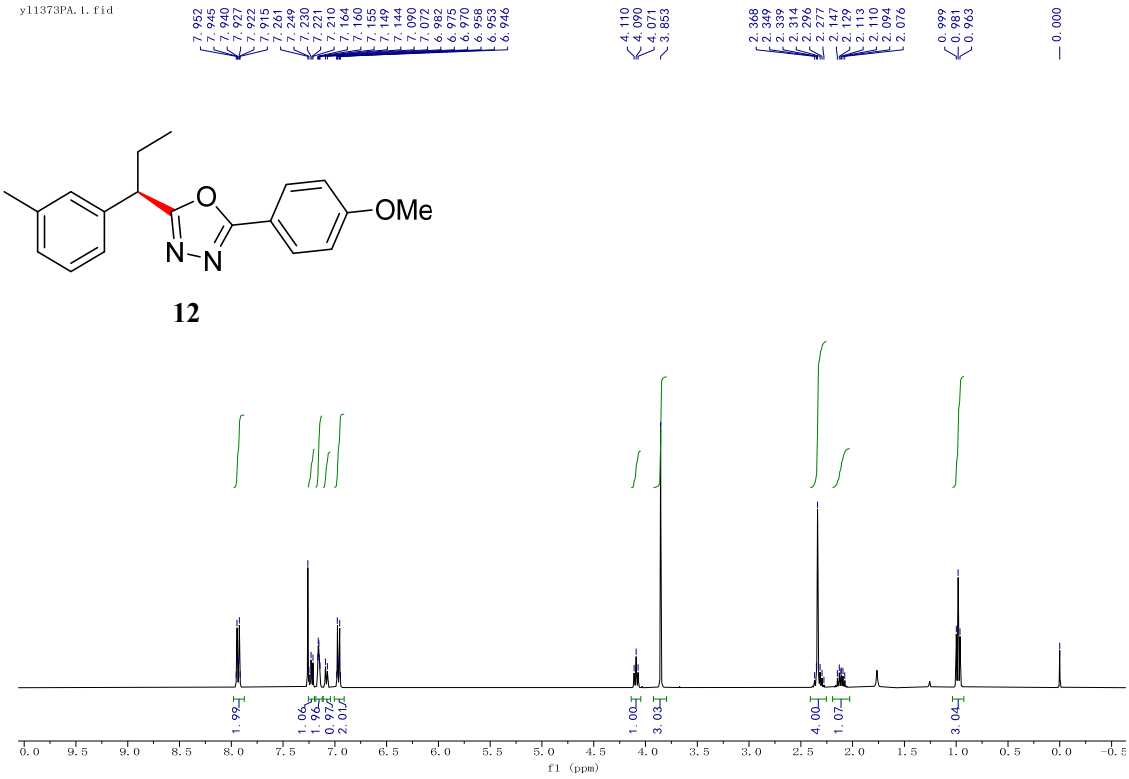

1.94
3.86
1.01
1.99
1.00
2.99
1.96
1.02
1.03
2.04
f1 (ppm)

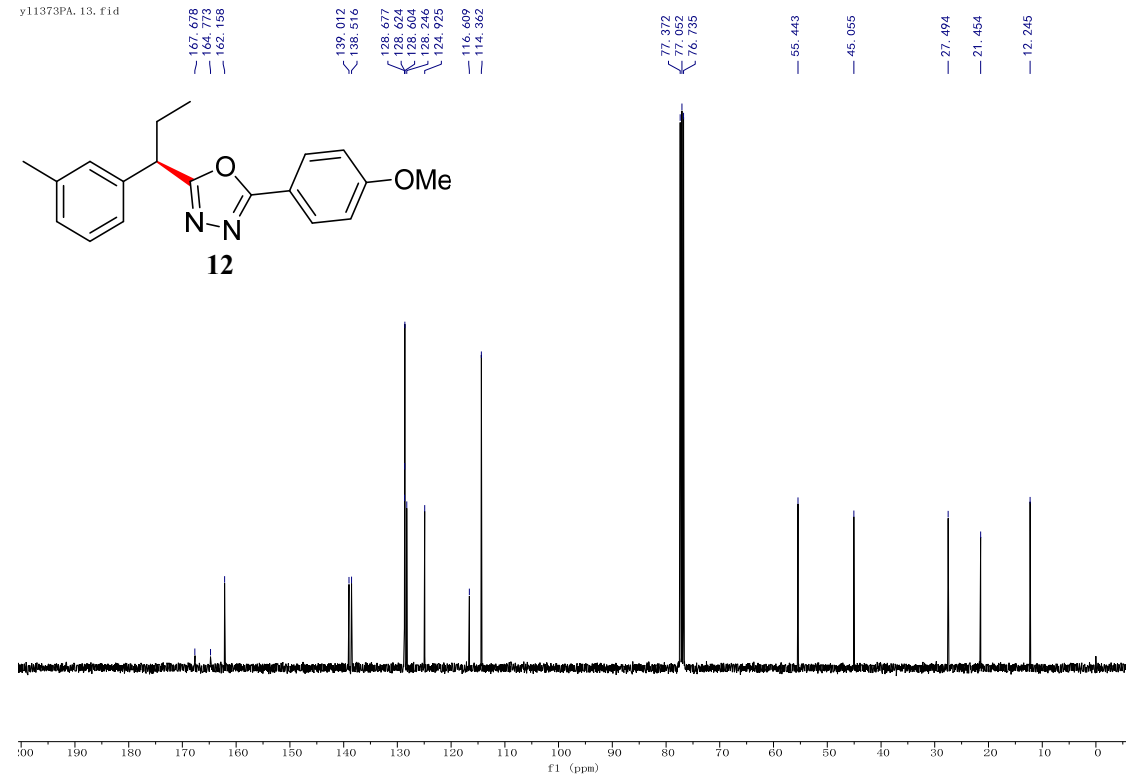

y11388PC.13.fid
167.098
164.959
162.263
138.581
129.027
128.644
127.825
127.782
116.405
114.404
77.357
77.039
76.722
55.457
44.397
42.787
31.476
30.324

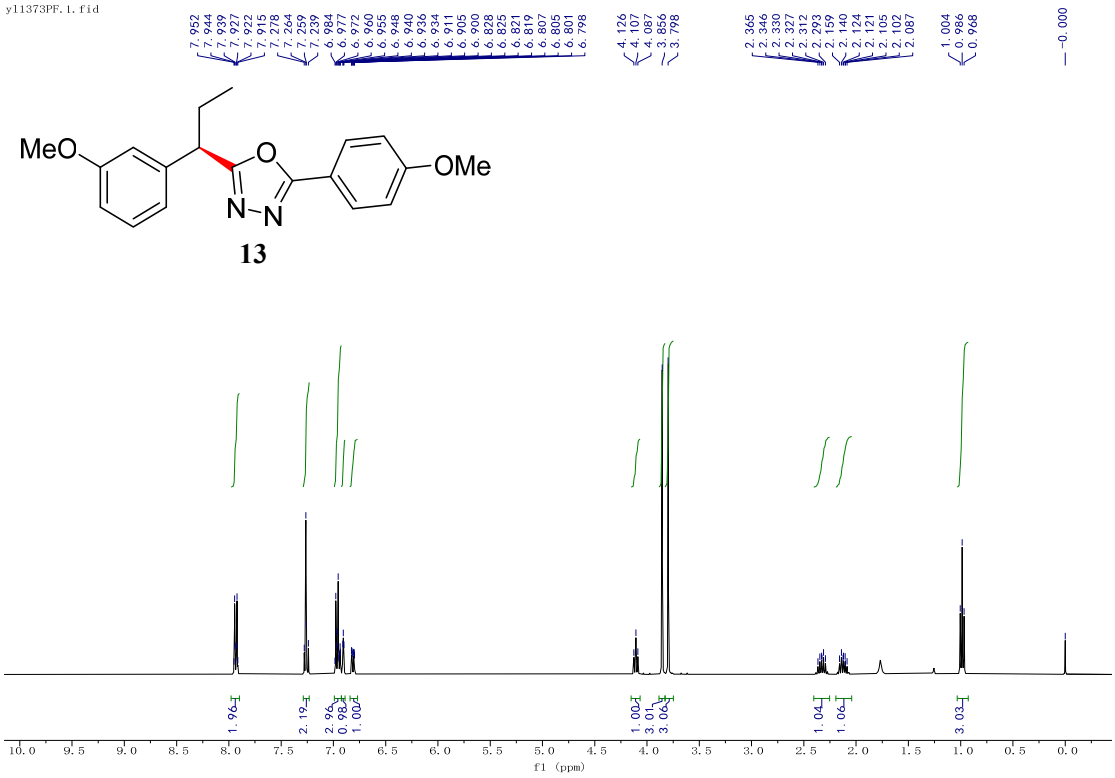

Cl
O
N
N
OMe

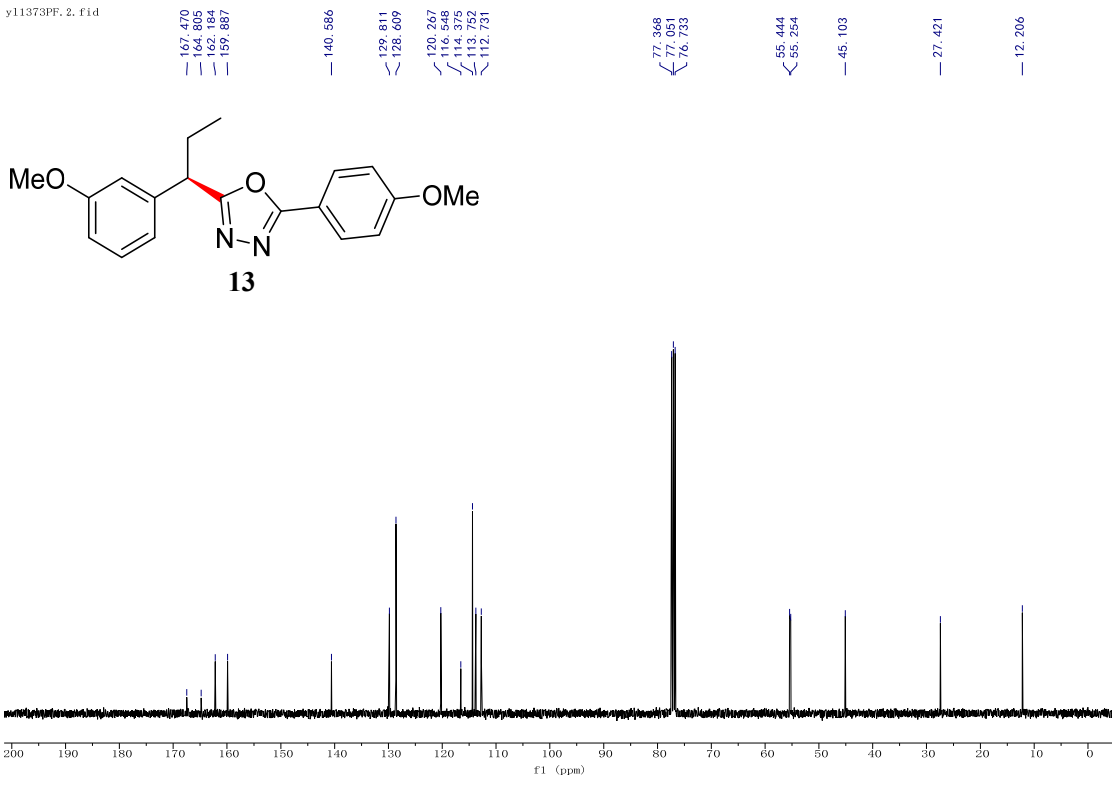
11

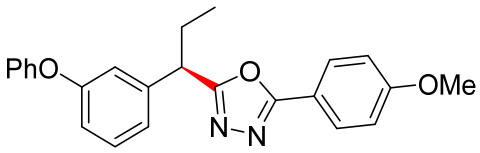

f1 (ppm)


y11373PA.1.fid
7.952
7.945
7.940
7.927
7.922
7.915
7.261
7.249
7.230
7.221
7.210
7.164
7.160
7.155
7.149
7.144
7.090
7.072
6.982
6.975
6.970
6.958
6.953
6.946
4.110
4.090
4.071
3.853
2.368
2.349
2.339
2.314
2.296
2.277
2.147
2.129
2.113
2.110
2.094
2.076
0.999
0.981
0.963
0.000
OMe
12
1.99
1.06
1.96
0.97
2.01
1.00
3.03
4.00
1.07
3.04
f1 (ppm)

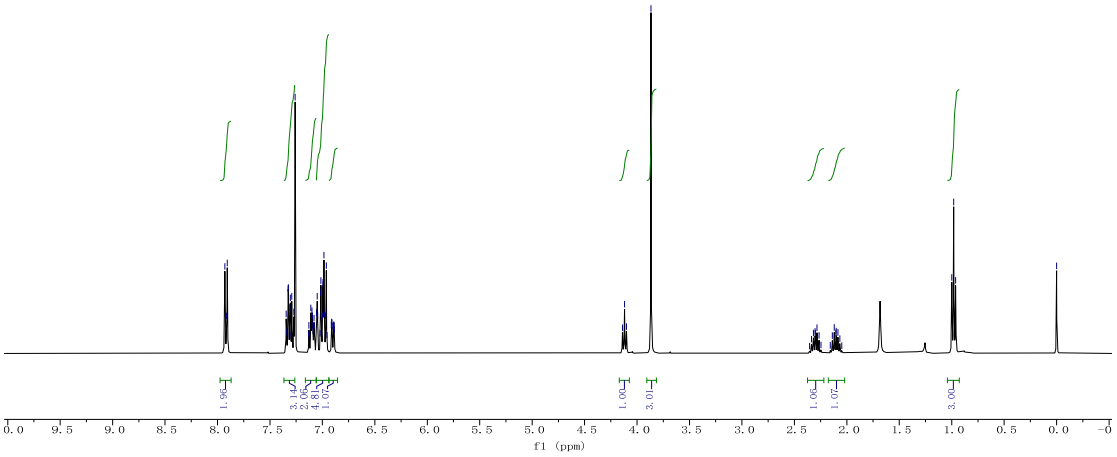

y11373PA.13.fid
167.678
164.773
162.158
139.012
138.516
128.677
128.624
128.604
128.246
124.925
116.609
114.362
77.372
77.052
76.735
55.443
45.055
27.494
21.454
12.245
OMe
12
f1 (ppm)

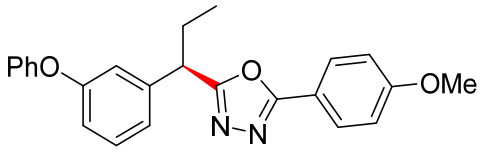

yl1373PF.1.fid
7.952
7.944
7.939
7.927
7.922
7.915
7.278
7.264
7.259
7.239
6.984
6.977
6.972
6.960
6.955
6.948
6.940
6.936
6.934
6.911
6.905
6.900
6.828
6.825
6.821
6.819
6.807
6.805
6.801
6.798
4.126
4.107
4.087
3.856
3.798
2.365
2.346
2.330
2.327
2.312
2.293
2.159
2.140
2.124
2.121
2.105
2.102
2.087
1.004
0.986
0.968
-0.000
MeO
OMe
O
N
N
13
1.96
2.19
2.96
0.98
1.00
1.00
3.01
3.06
1.04
1.06
3.03
f1 (ppm)


yl1373PF.2.fid
167.470
164.805
162.184
159.887
140.586
129.811
128.609
120.267
116.548
114.375
113.752
112.731
77.368
77.051
76.733
55.444
55.254
45.103
27.421
12.206
MeO
OMe
O
N
N
13
f1 (ppm)

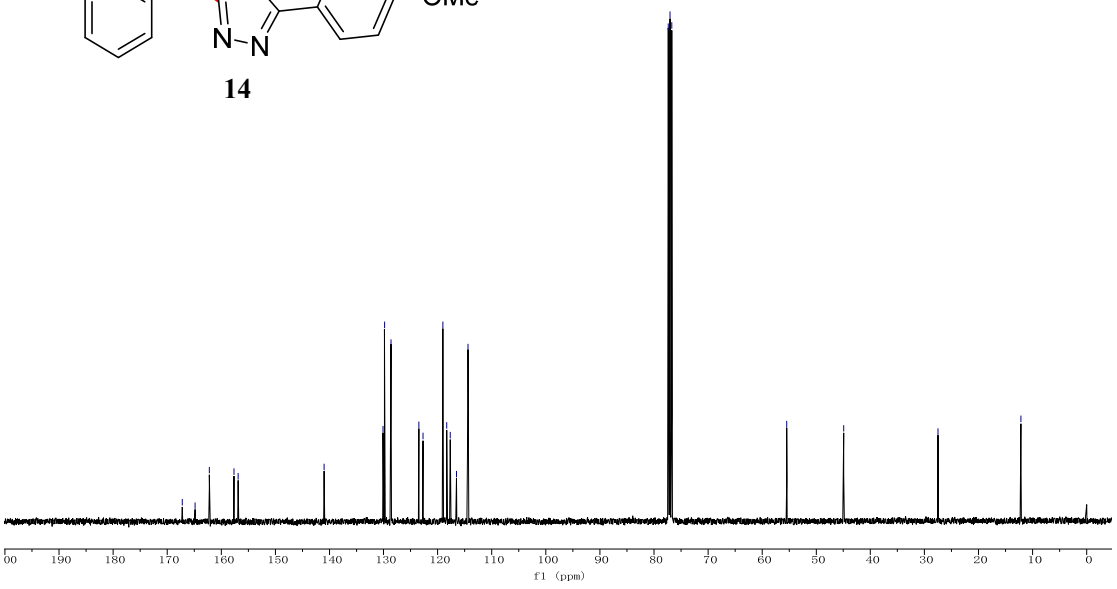

PhO
O
N
N
OMe

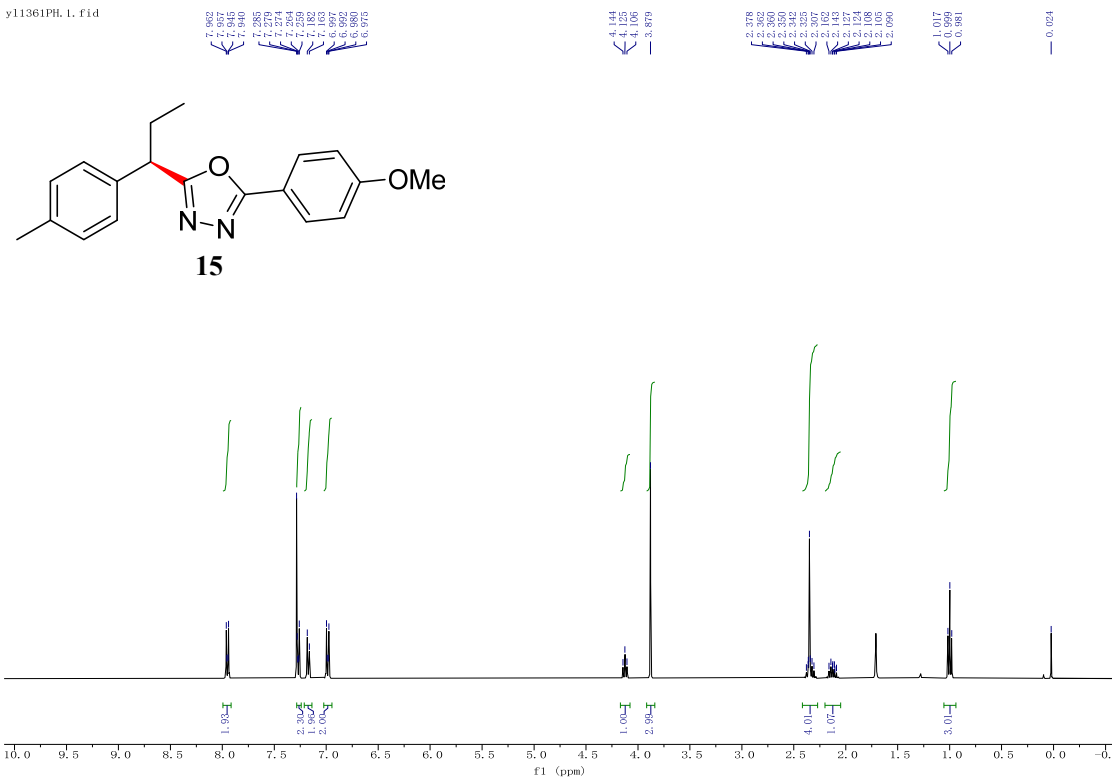
14

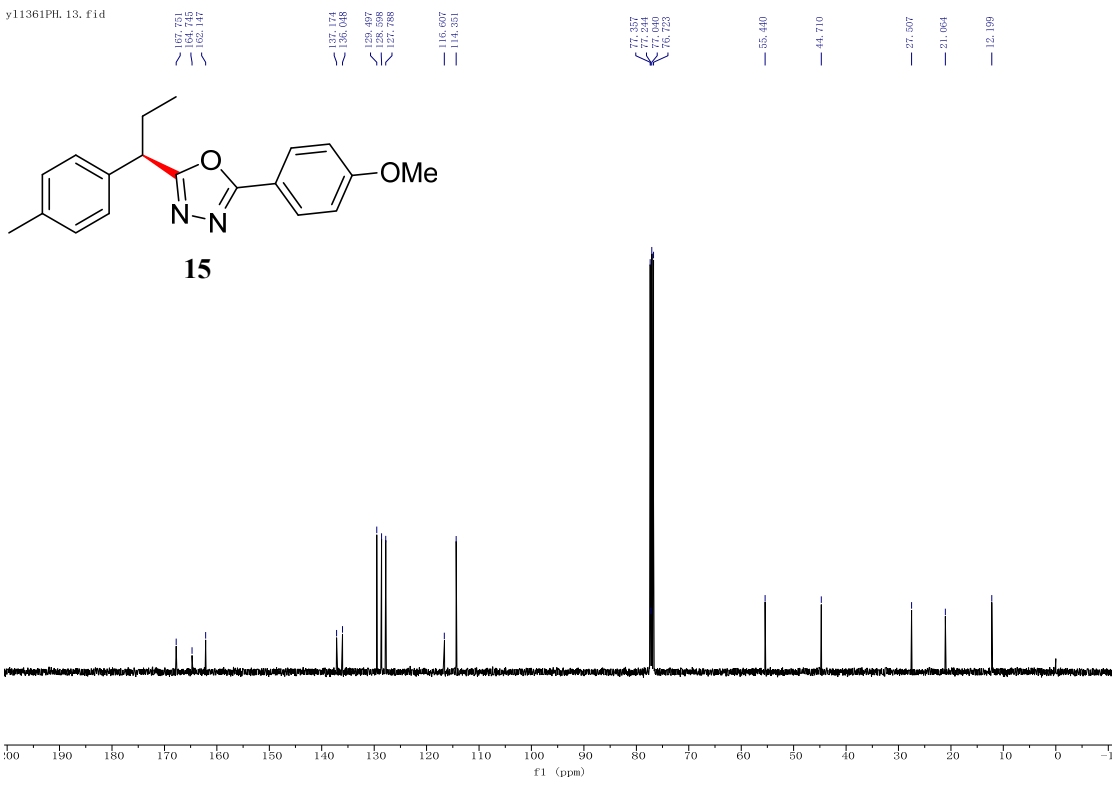

1.96
3.14
2.06
4.81
1.07
1.00
3.01
1.06
1.07
3.00
f1 (ppm)

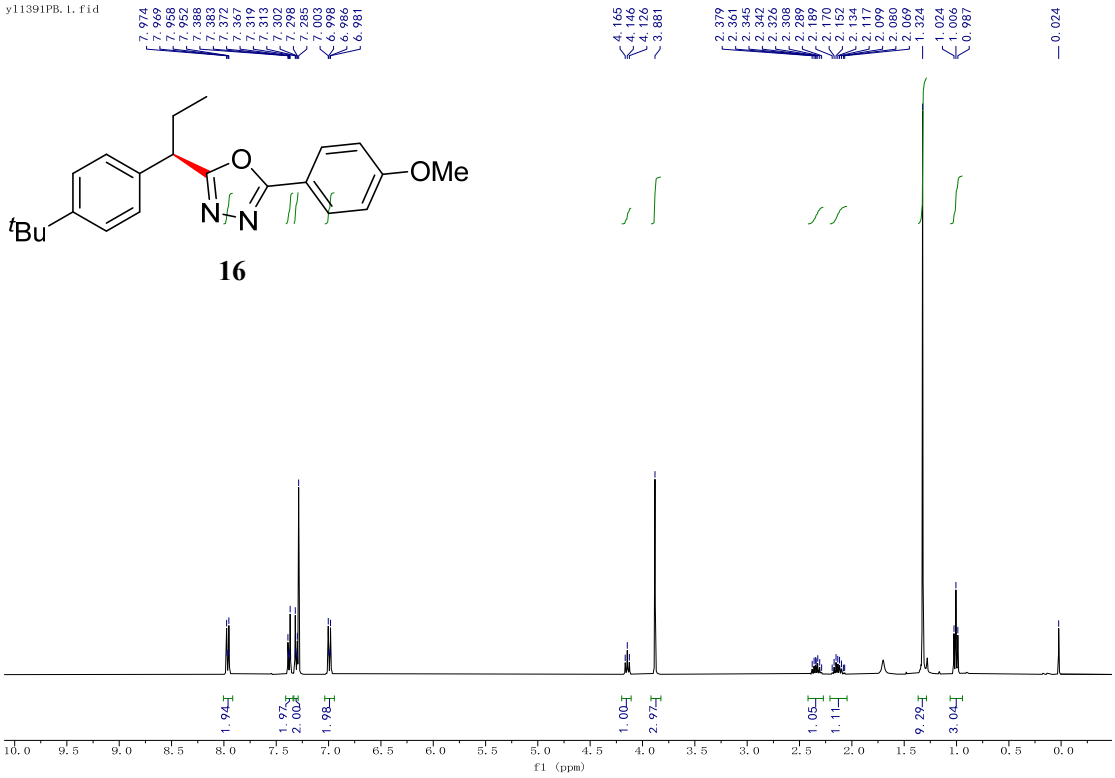

PhO
O
N
N
OMe

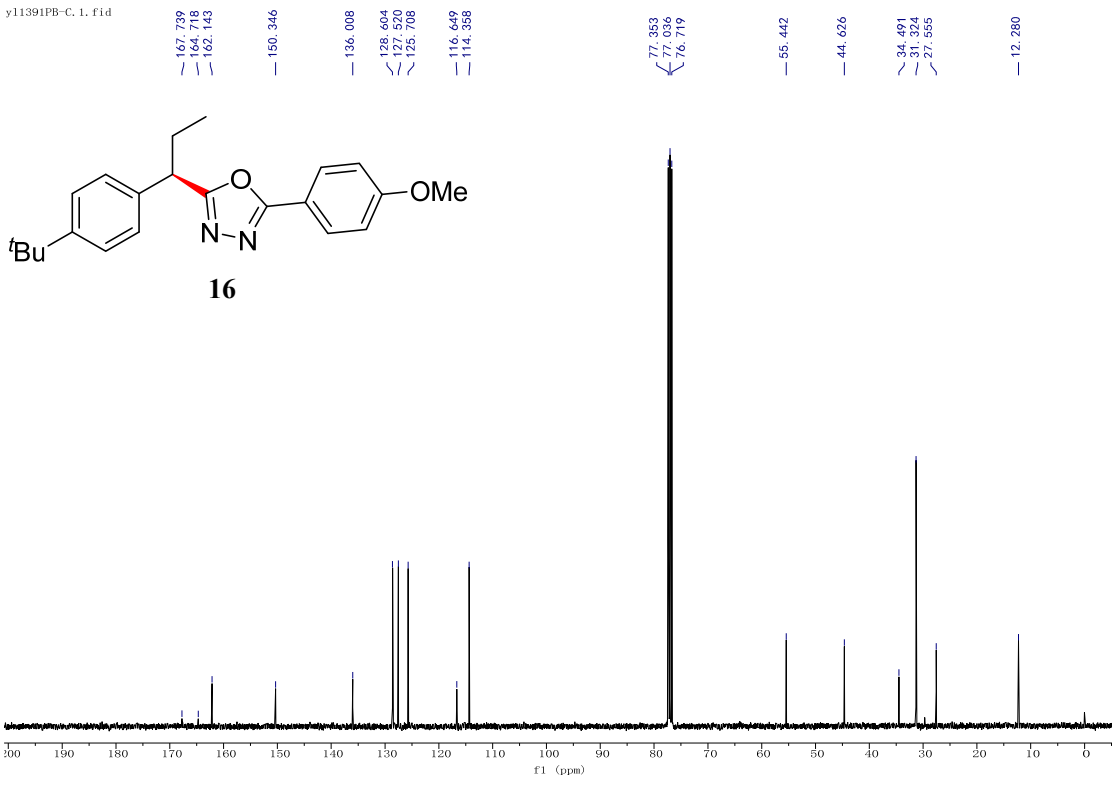
14

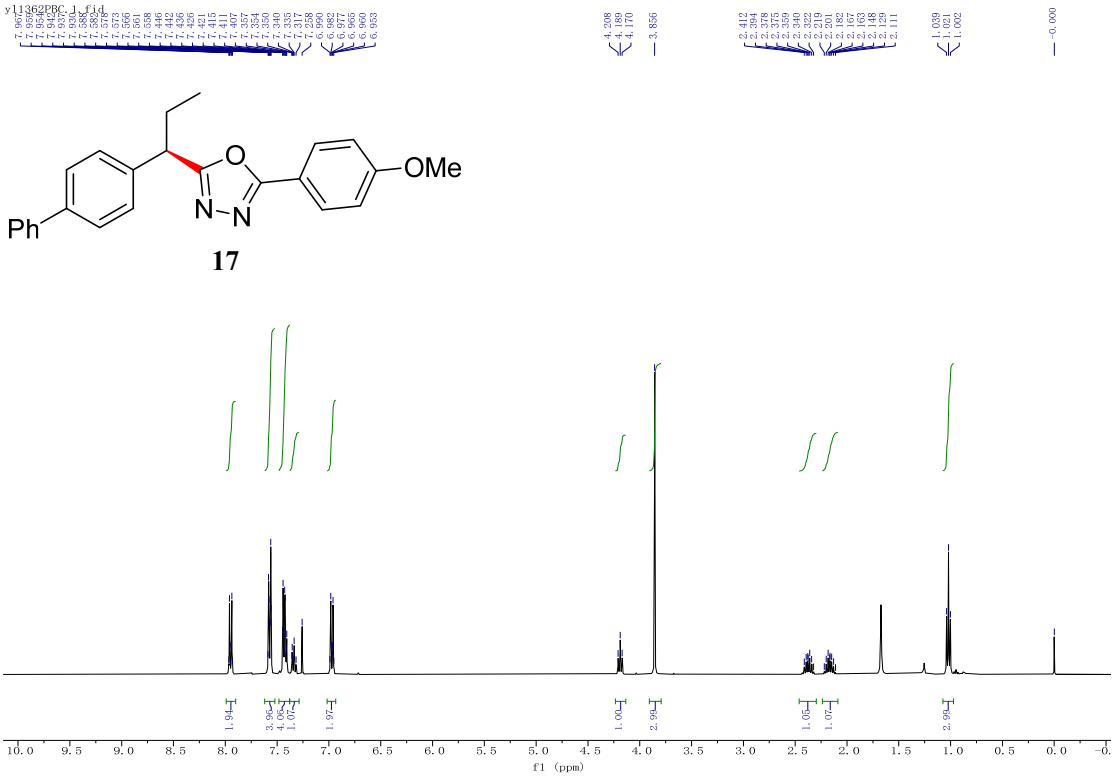

f1 (ppm)

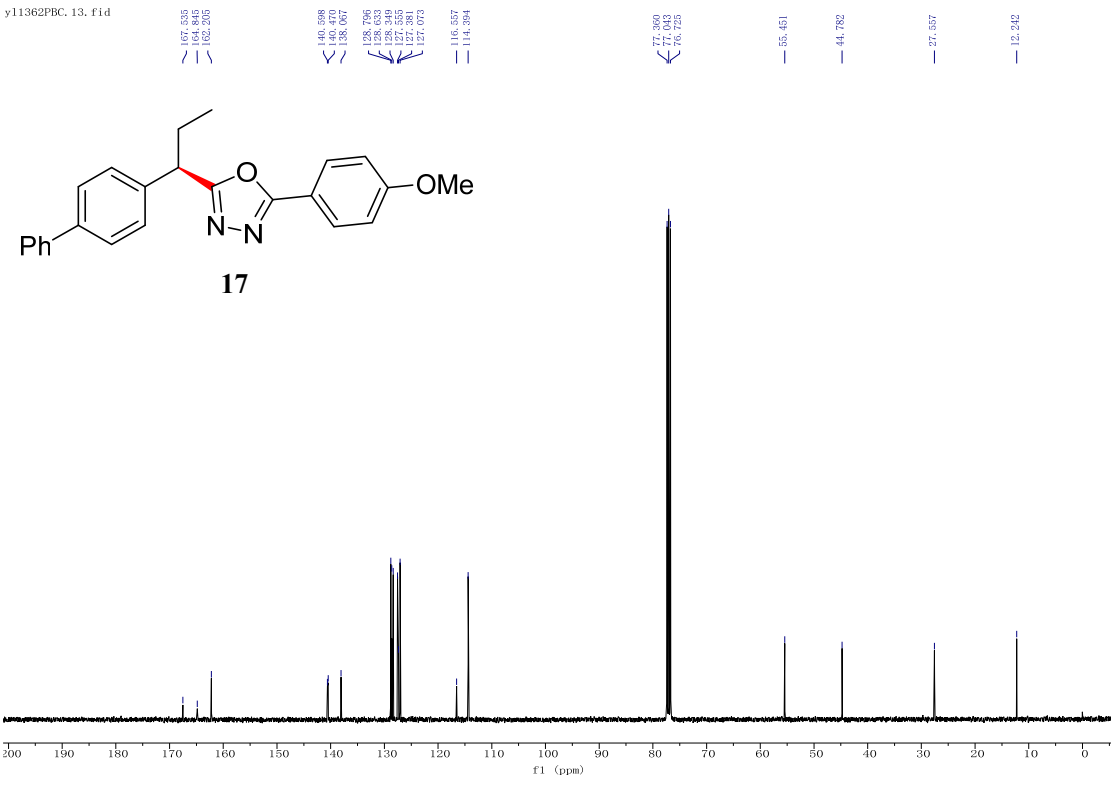

y11361PH.1.fid
7.962
7.957
7.945
7.940
7.285
7.273
7.274
7.264
7.259
7.182
7.165
6.997
6.992
6.980
6.976
4.144
4.125
4.106
3.879
2.378
2.362
2.356
2.350
2.342
2.335
2.307
2.165
2.143
2.127
2.124
2.108
2.103
2.090
1.017
0.999
0.981
0.024
OMe
N
N
O
15
1.93
2.30
1.96
2.00
1.06
2.99
4.01
1.07
3.01
f1 (ppm)

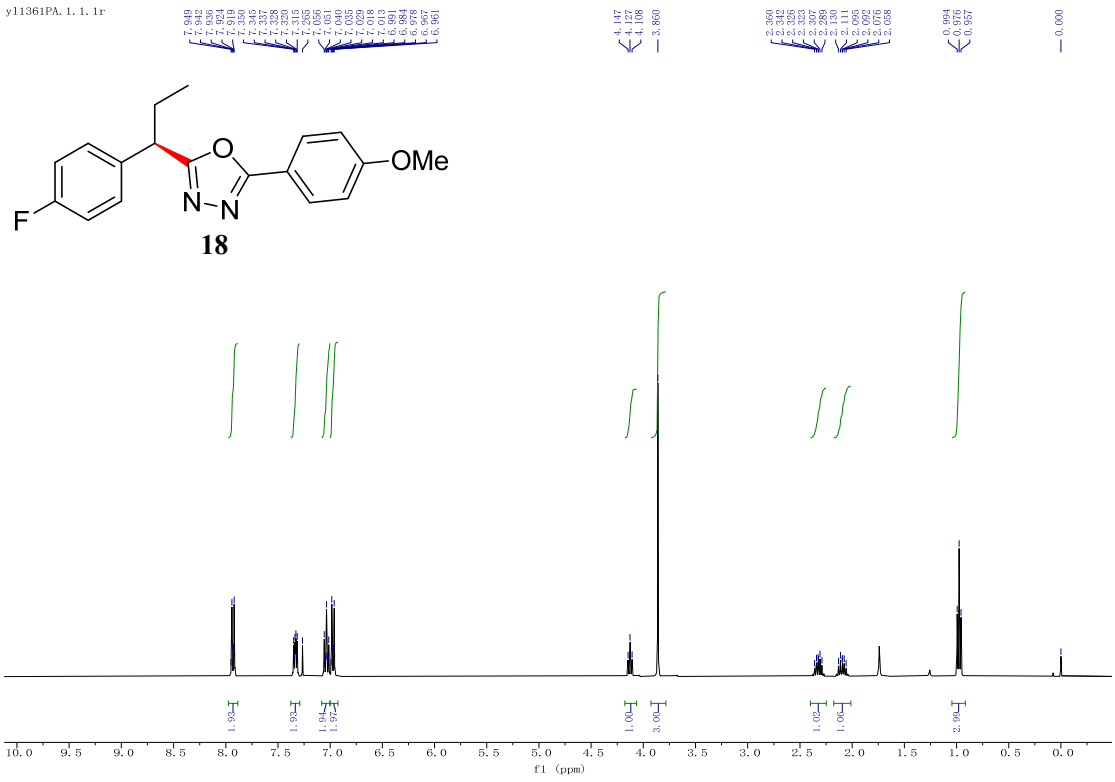

y11361PH.13.fid
167.751
164.745
162.147
137.174
136.048
129.497
129.088
127.788
116.607
114.351
77.357
77.044
77.040
76.723
55.440
44.710
27.507
21.064
12.199
OMe
N
N
O
15
f1 (ppm)


yl1391PB.1.fid
7.974
7.969
7.958
7.952
7.388
7.383
7.372
7.367
7.319
7.313
7.302
7.298
7.285
7.003
6.998
6.986
6.981
4.165
4.146
4.126
3.881
2.379
2.361
2.345
2.342
2.326
2.308
2.289
2.189
2.170
2.152
2.134
2.117
2.099
2.080
2.069
1.324
1.024
1.006
0.987
0.024
OMe
tBu
N
N
O
16
1.94
1.97
2.00
1.98
1.00
2.97
1.05
1.11
9.29
3.04
f1 (ppm)


yl1391PB-C.1.fid
167.739
164.718
162.143
150.346
136.008
128.604
127.520
125.708
116.649
114.358
77.353
77.036
76.719
55.442
44.626
34.491
31.324
27.555
12.280
OMe
tBu
N
N
O
16
f1 (ppm)


y11362PBC.1.fid
OMe
Ph
N
N
O
17
f1 (ppm)


y11362PBC.13.fid
OMe
Ph
N
N
O
17
f1 (ppm)


y11361PA. 1. 1. 1r
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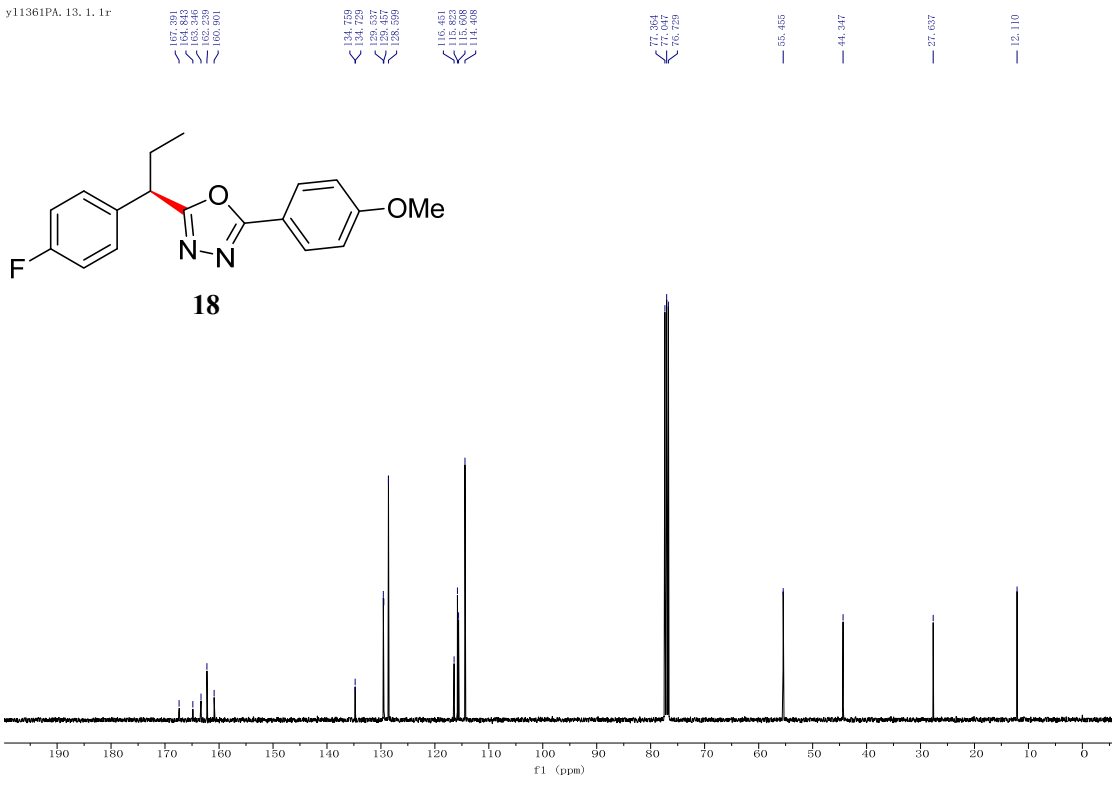
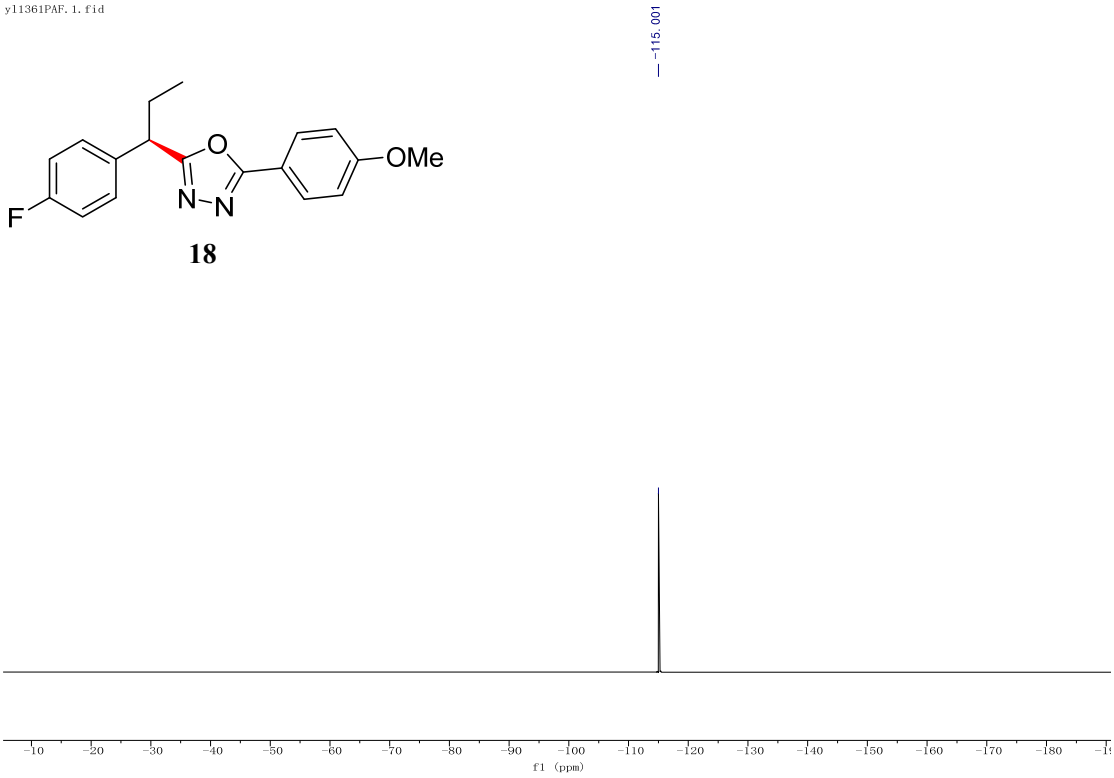
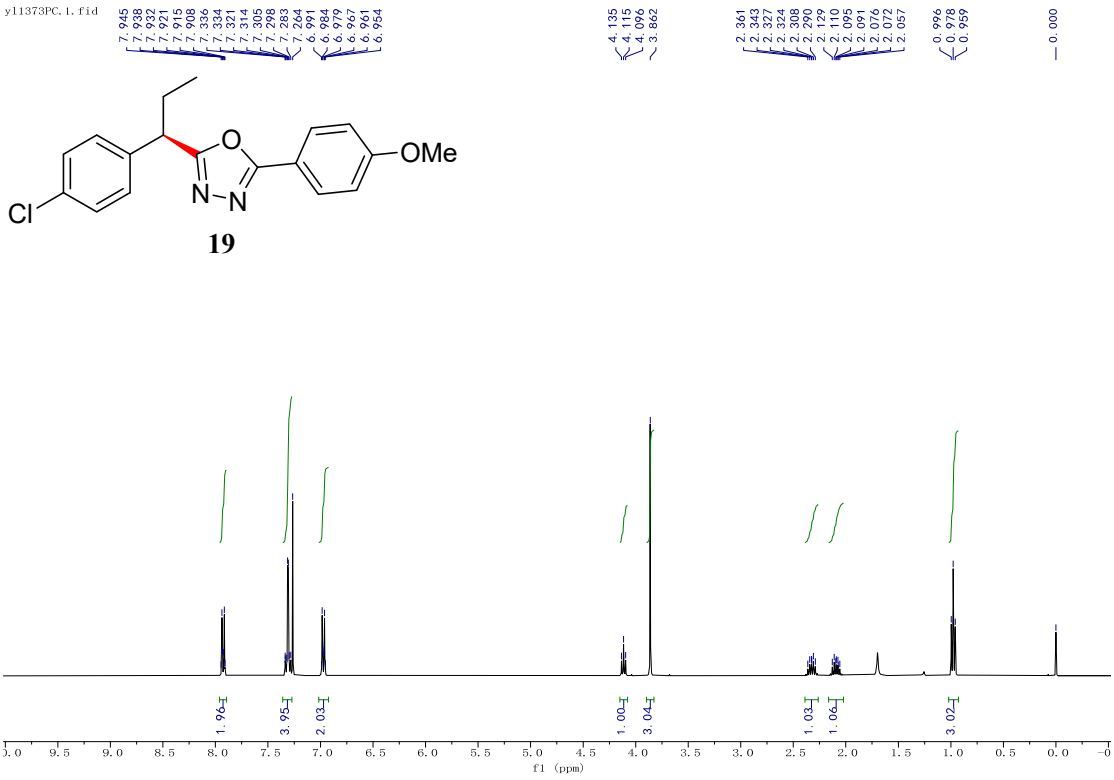
7.942
7.936
7.924
7.919
7.350
7.345
7.337
7.328
7.320
7.315
7.255
7.056
7.051
7.040
7.035
7.029
7.018
7.013
6.991
6.984
6.978
6.967
6.961
4.147
4.127
4.108
3.860
2.360
2.342
2.326
2.323
2.307
2.289
2.130
2.111
2.095
2.092
2.076
2.058
0.994
0.976
0.957
0.000
OMe
F
O
N
N
18
1.93
1.93
1.94
1.97
1.00
3.00
1.02
1.06
2.99
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

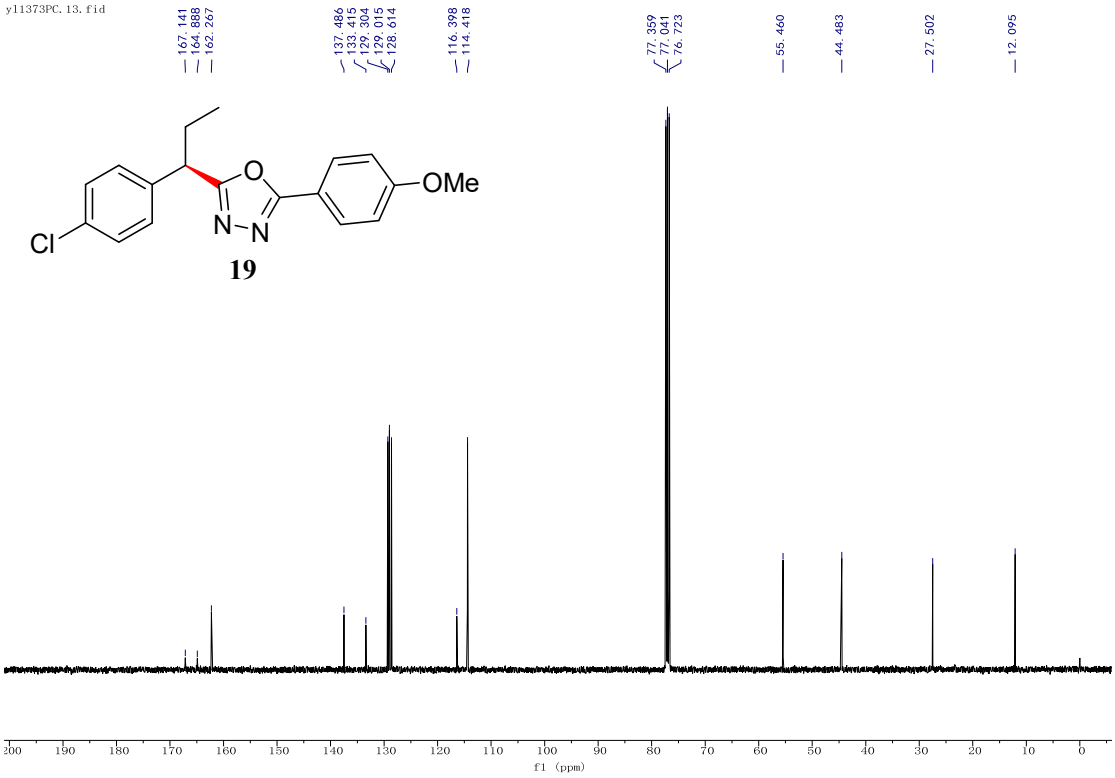

y11361PA. 13. 1. 1r
167.391
164.453
163.946
162.239
160.901
134.739
134.729
129.537
129.457
128.399
116.461
115.823
115.588
114.408
77.364
77.047
76.729
55.455
44.347
27.637
12.110
OMe
F
O
N
N
18
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


y11361PAF.1.fid
-115.001
OMe
F
f1 (ppm)

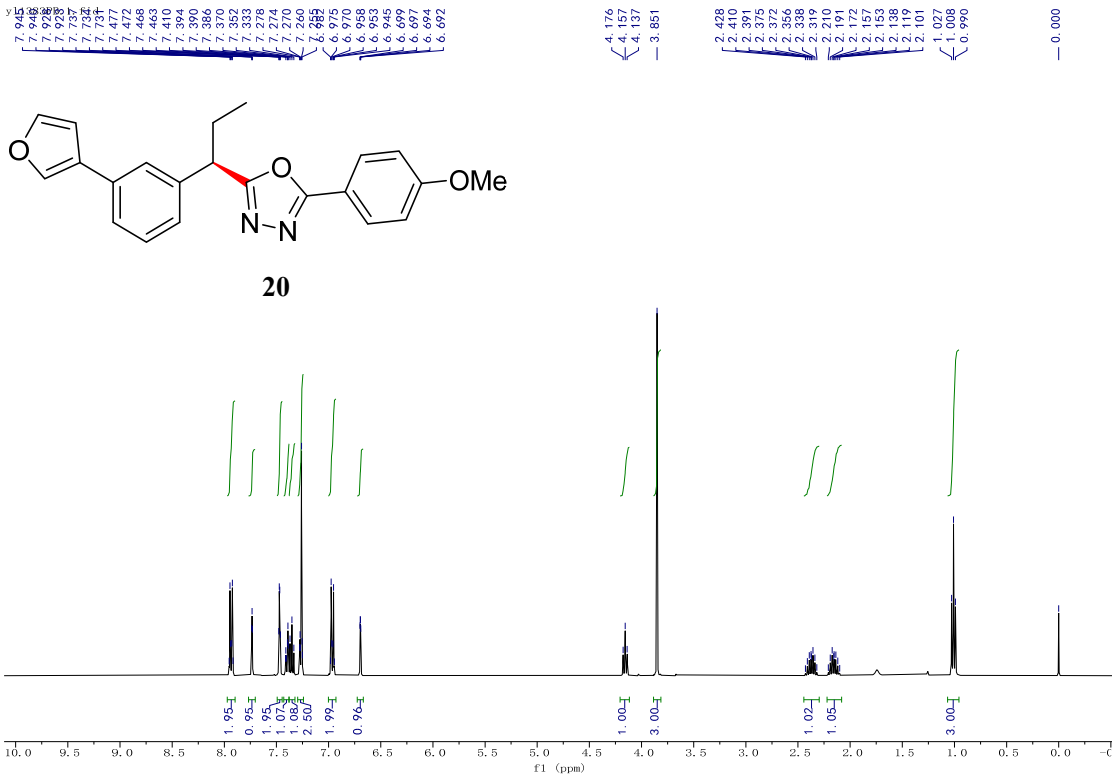
18


y11373PC.1.fid
7.945
7.938
7.932
7.921
7.915
7.908
7.336
7.334
7.321
7.314
7.305
7.298
7.283
7.264
6.991
6.984
6.979
6.967
6.961
6.954
4.135
4.115
4.096
3.862
2.361
2.343
2.327
2.324
2.308
2.290
2.129
2.110
2.095
2.091
2.076
2.072
2.057
0.996
0.978
0.959
0.000
OMe
Cl
1.96
3.95
2.03
1.00
3.04
1.03
1.04
3.02
f1 (ppm)

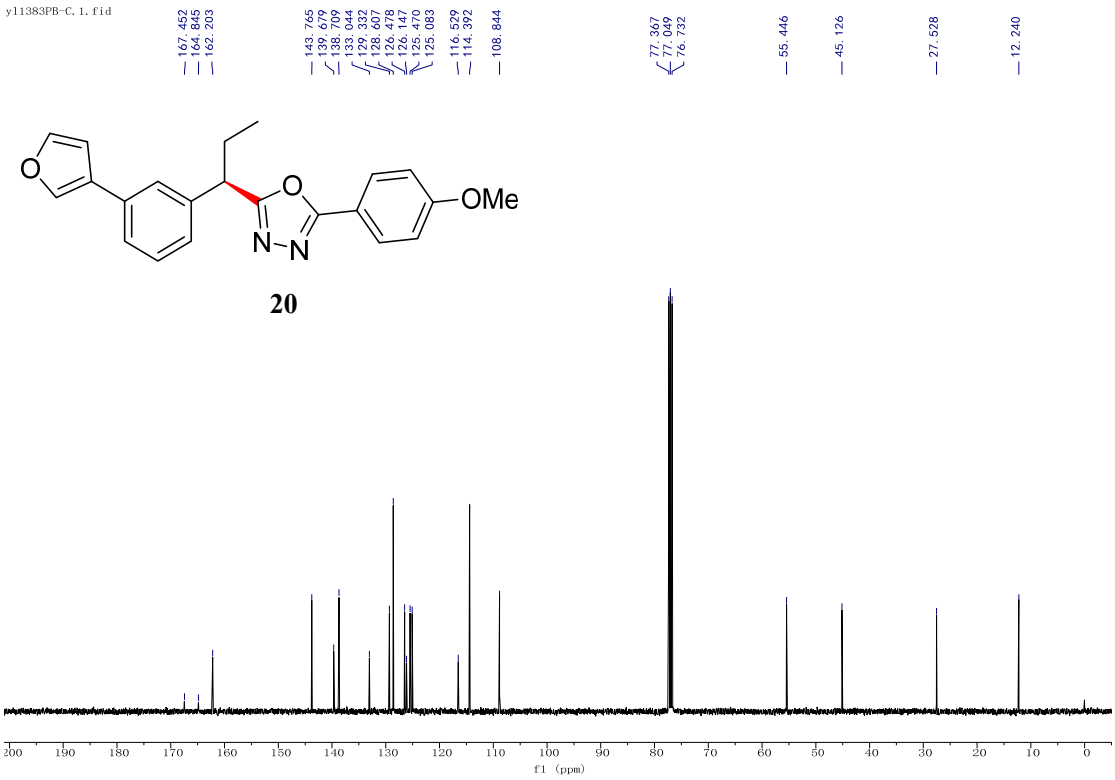
19


y11373PC.13.fid
167.141
164.888
162.267
137.486
133.415
129.304
129.015
128.614
116.398
114.418
77.359
77.041
76.723
55.460
44.483
27.502
12.095
Cl
O
N
N
OMe
f1 (ppm)

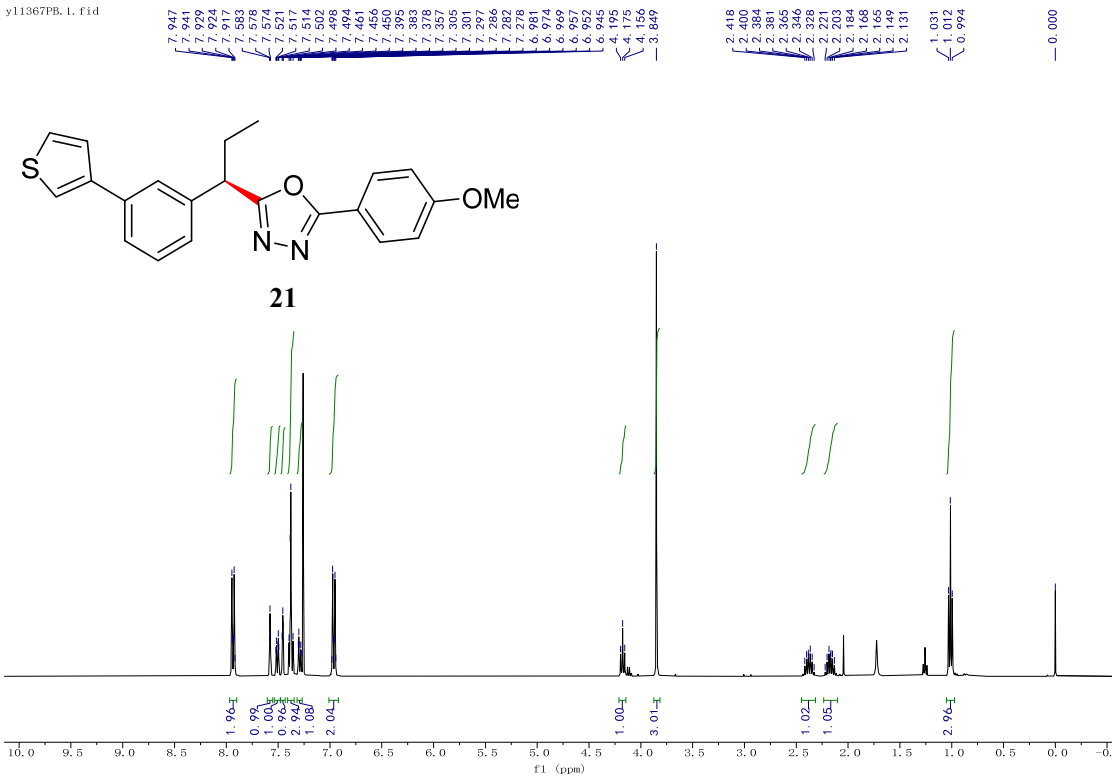
19


7.945
7.926
7.737
7.734
7.477
7.472
7.468
7.463
7.410
7.394
7.390
7.386
7.370
7.352
7.333
7.278
7.274
7.270
7.260
6.975
6.970
6.958
6.953
6.945
6.699
6.697
6.694
6.692
4.176
4.157
4.137
3.851
2.428
2.410
2.391
2.375
2.372
2.356
2.338
2.319
2.210
2.191
2.172
2.157
2.153
2.138
2.119
2.101
1.027
1.008
0.990
0.000
O
O
N
N
OMe
1.95
0.95
1.95
1.07
1.08
2.50
1.92
0.96
1.00
3.00
1.02
1.05
3.00
f1 (ppm)

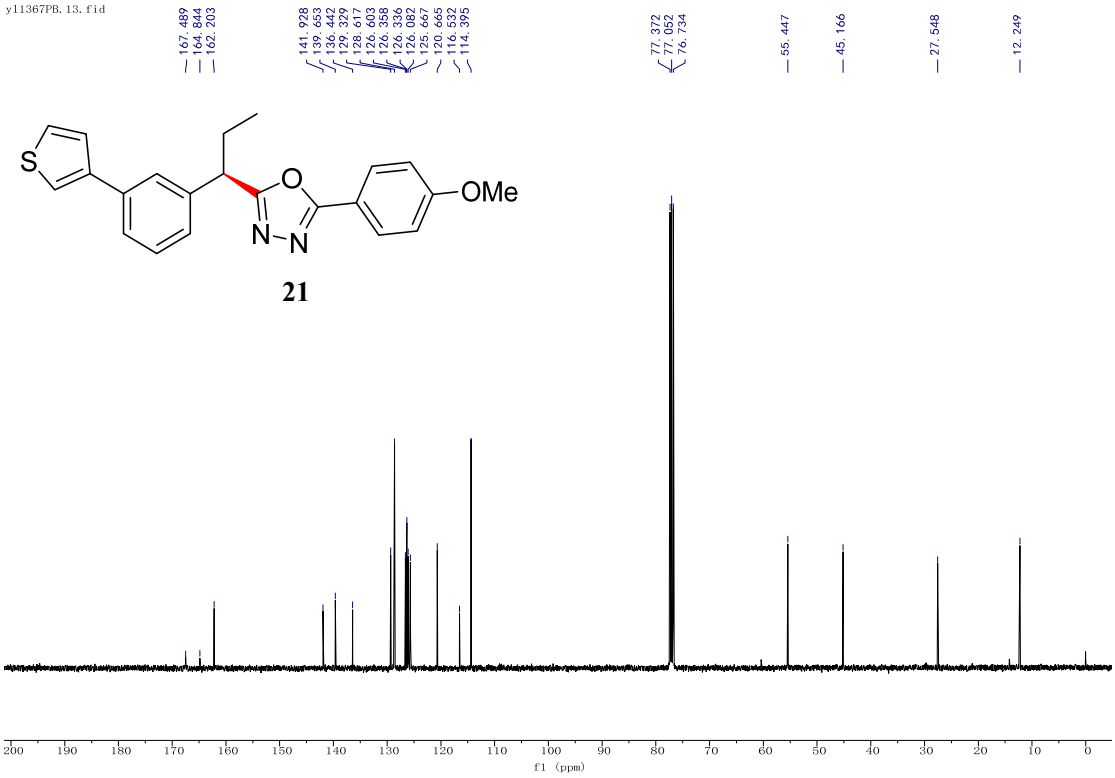
20


yl1383PB-C.1.fid
167.452
164.845
162.203
143.765
139.679
138.709
133.044
129.332
128.607
126.478
126.147
125.470
125.083
116.529
114.392
108.844
77.367
77.049
76.732
55.446
45.126
27.528
12.240
O
O
N
N
OMe
f1 (ppm)

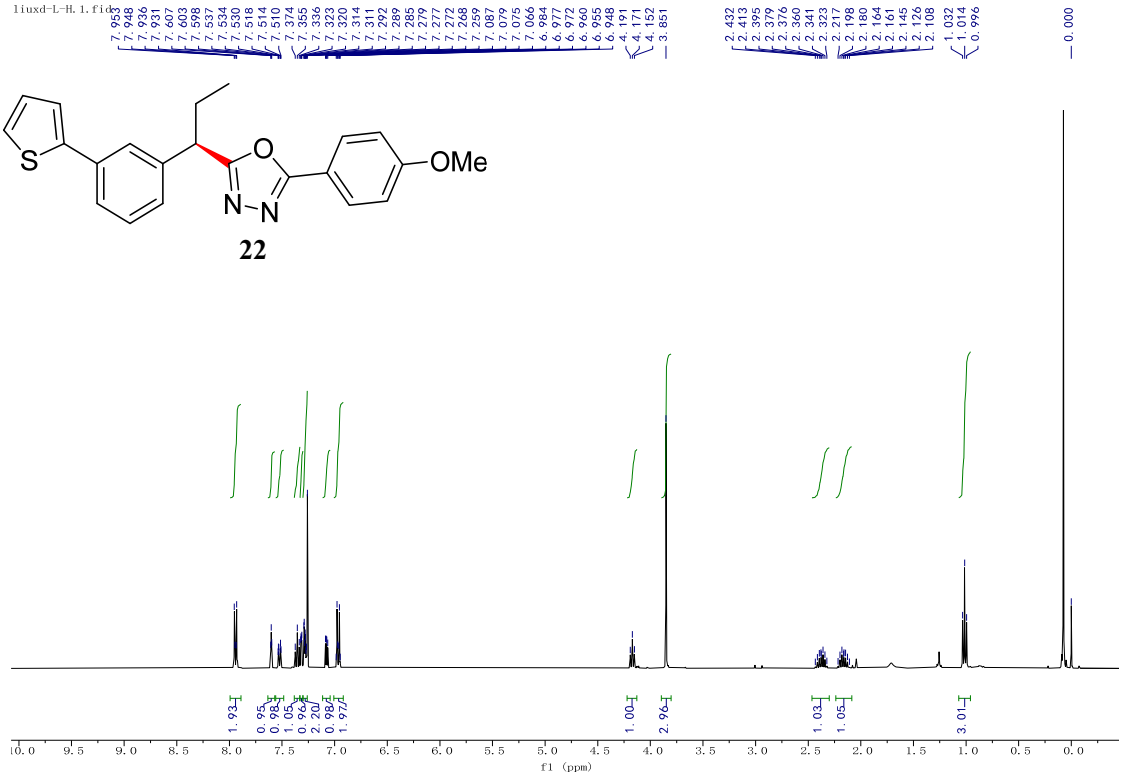
20


yl1367PB.1.fid
7.947
7.941
7.929
7.924
7.597
7.583
7.578
7.574
7.521
7.517
7.514
7.502
7.498
7.494
7.461
7.456
7.450
7.395
7.383
7.378
7.357
7.305
7.301
7.297
7.286
7.282
7.278
6.991
6.974
6.969
6.957
6.952
6.945
4.195
4.175
4.156
3.849
2.418
2.400
2.384
2.381
2.365
2.346
2.328
2.221
2.203
2.184
2.168
2.165
2.149
2.131
1.031
1.012
0.994
0.000
S
O
N
N
OMe
1.96
0.99
1.00
0.94
2.94
1.08
2.04
1.00
3.01
1.02
1.05
2.96
f1 (ppm)

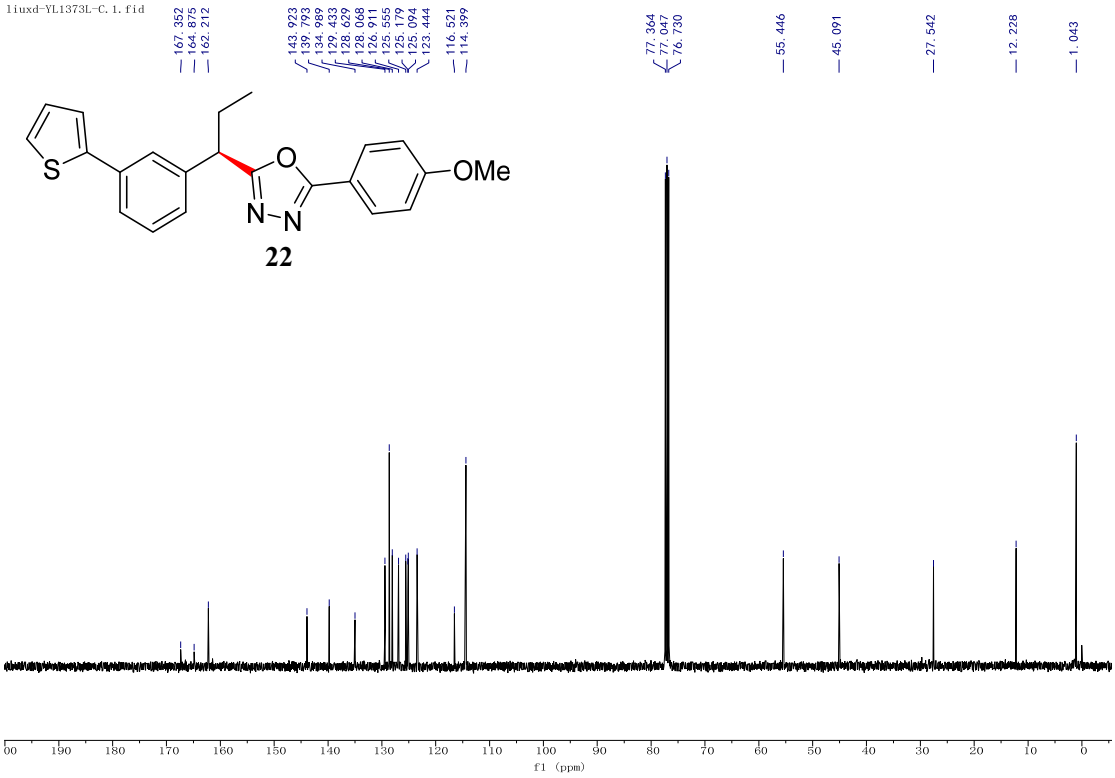
21


y11367PB.13.fid
167.489
164.844
162.203
141.928
139.653
136.442
129.329
128.617
126.603
126.358
126.336
126.082
125.667
120.665
116.532
114.395
77.372
77.052
76.734
55.447
45.166
27.548
12.249
S
O
N
N
OMe
f1 (ppm)

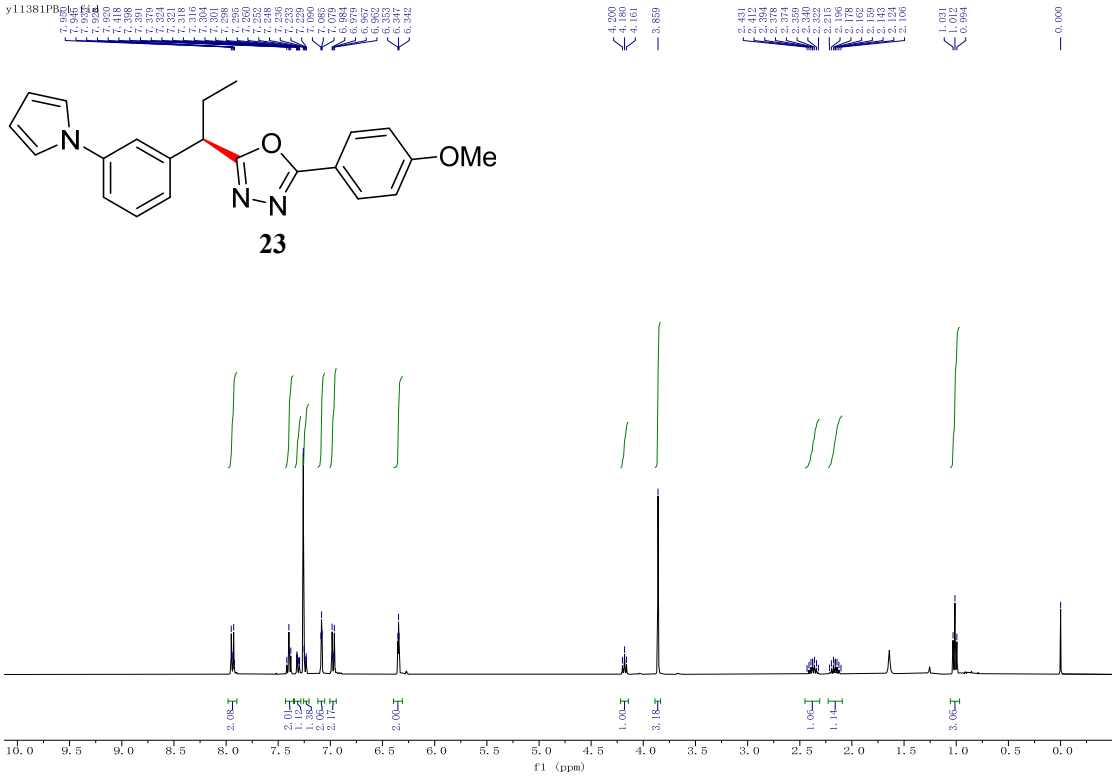
21


liuxd-L-H.1.fid
7.953
7.948
7.936
7.931
7.607
7.603
7.598
7.537
7.534
7.530
7.518
7.514
7.510
7.374
7.355
7.336
7.323
7.320
7.314
7.311
7.292
7.289
7.285
7.279
7.277
7.272
7.268
7.259
7.087
7.079
7.075
7.066
6.984
6.977
6.972
6.966
6.955
6.948
4.191
4.171
4.152
3.851
2.432
2.413
2.395
2.379
2.376
2.360
2.341
2.323
2.217
2.198
2.180
2.164
2.161
2.145
2.126
2.108
1.032
1.014
0.996
0.000
1.93
0.95
0.98
1.05
1.96
2.20
0.98
1.97
1.00
2.96
1.03
1.05
3.01
S
O
N
N
OMe
f1 (ppm)

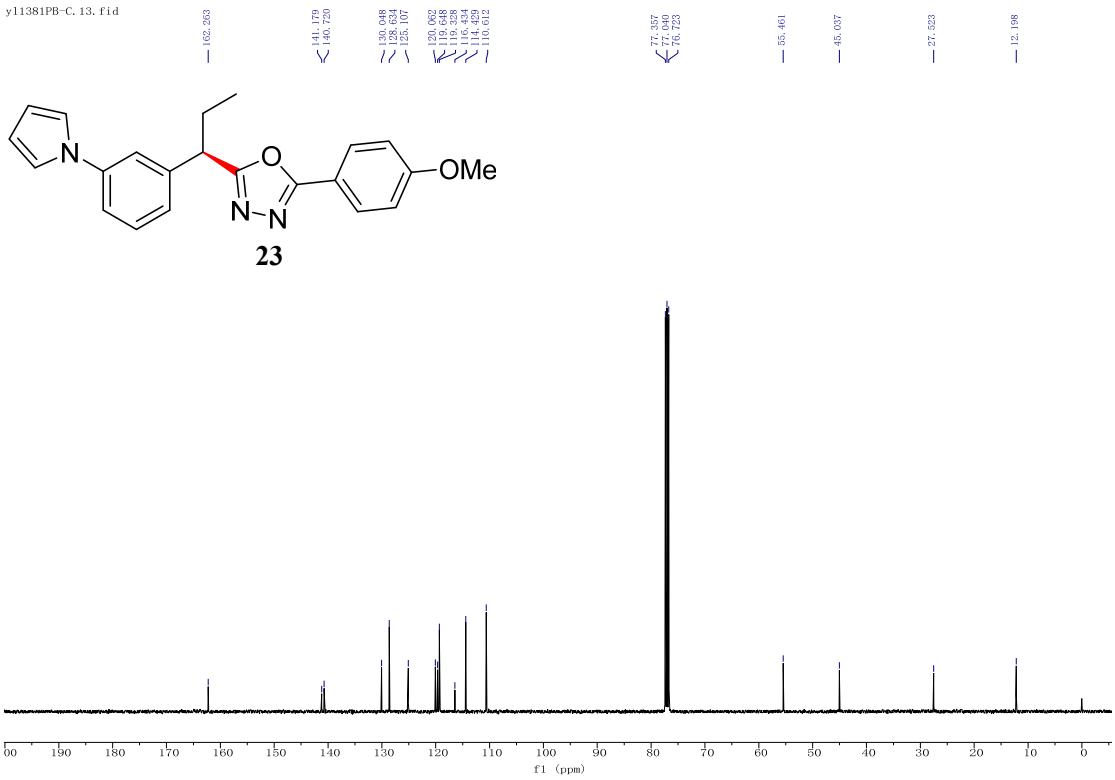
22


liuxd-YL1373L-C.1.fid
167.352
164.875
162.212
143.923
139.793
134.989
129.433
128.629
128.068
126.911
125.555
125.179
125.094
123.444
116.521
114.399
77.364
77.047
76.730
55.446
45.091
27.542
12.228
1.043
S
O
N
N
OMe
f1 (ppm)

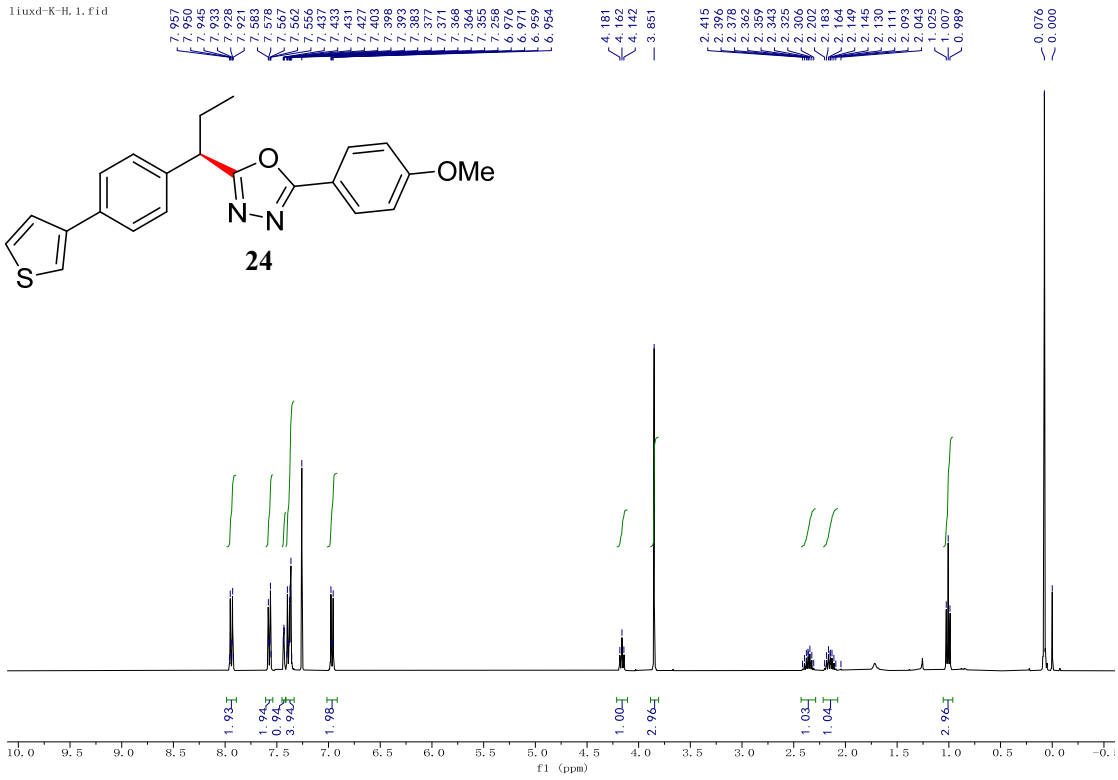
22


y11381PB
4.200
4.180
4.161
3.859
2.431
2.412
2.394
2.378
2.359
2.340
2.322
2.215
2.196
2.178
2.162
2.143
2.124
2.106
1.031
1.012
0.994
0.000
N
O
N
N
OMe
2.08
2.01
1.12
1.06
2.17
2.00
1.00
3.18
1.06
1.14
3.06
f1 (ppm)

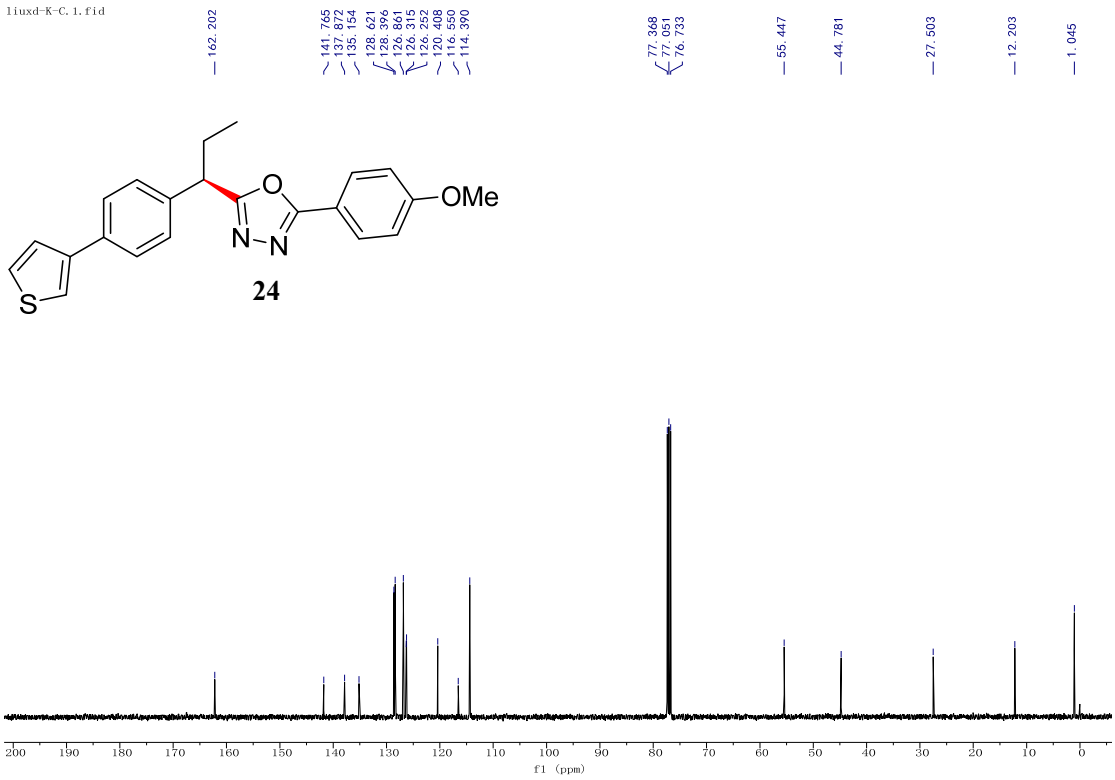
23

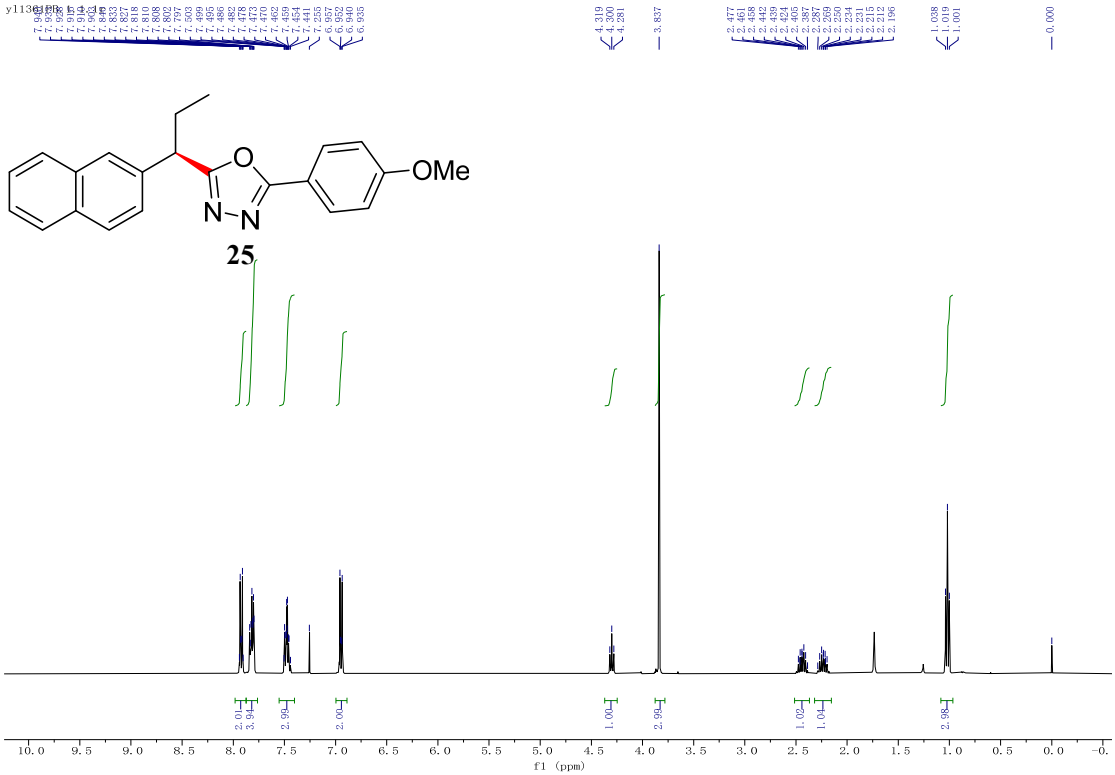

yl1381PB-C.13.fid
162.263
141.179
140.720
130.048
128.634
125.107
120.062
119.648
119.328
116.434
114.429
110.612
77.357
77.040
76.723
55.461
45.037
27.523
12.198
N
O
N
N
OMe
f1 (ppm)

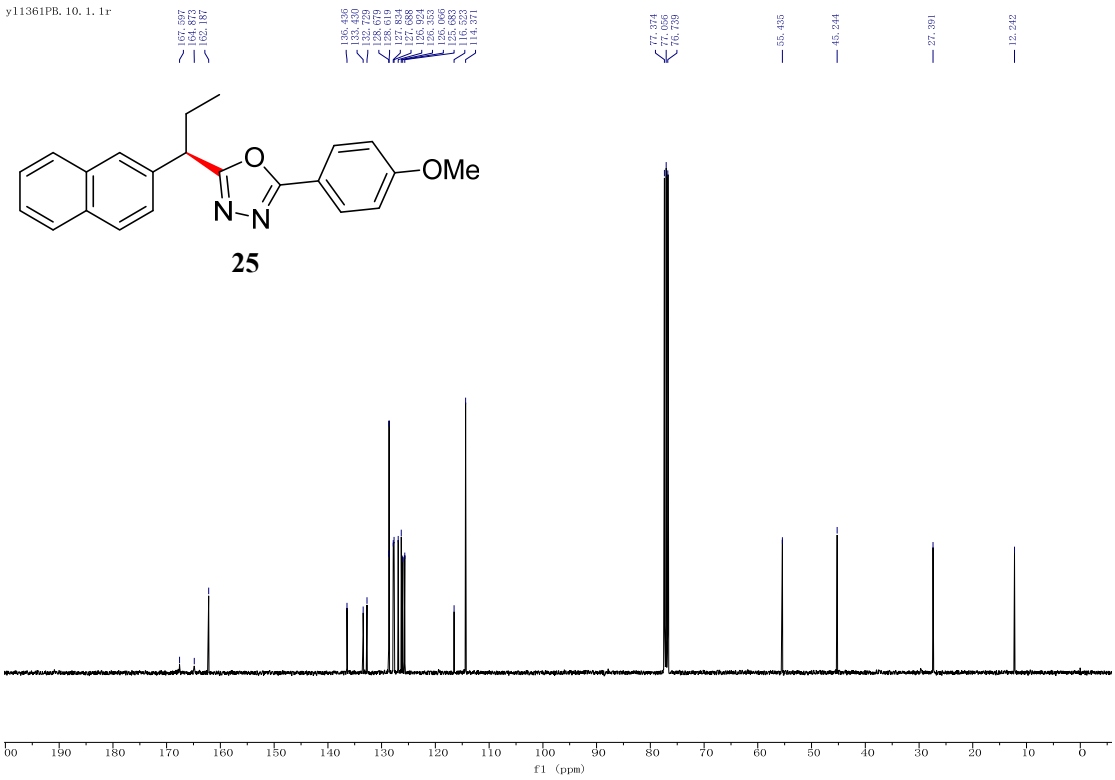
23


liuxd-K-H.1.fid
7.957
7.950
7.945
7.933
7.928
7.921
7.583
7.578
7.567
7.562
7.556
7.437
7.433
7.431
7.427
7.403
7.398
7.393
7.383
7.377
7.371
7.368
7.364
7.355
7.258
6.976
6.971
6.959
6.954
4.181
4.162
4.142
3.851
2.415
2.396
2.378
2.362
2.359
2.343
2.325
2.306
2.202
2.183
2.164
2.149
2.145
2.130
2.111
2.093
2.043
1.025
1.007
0.989
0.076
0.000
1.93
1.94
0.92
3.94
1.98
1.00
2.96
1.03
1.04
2.96
f1 (ppm)

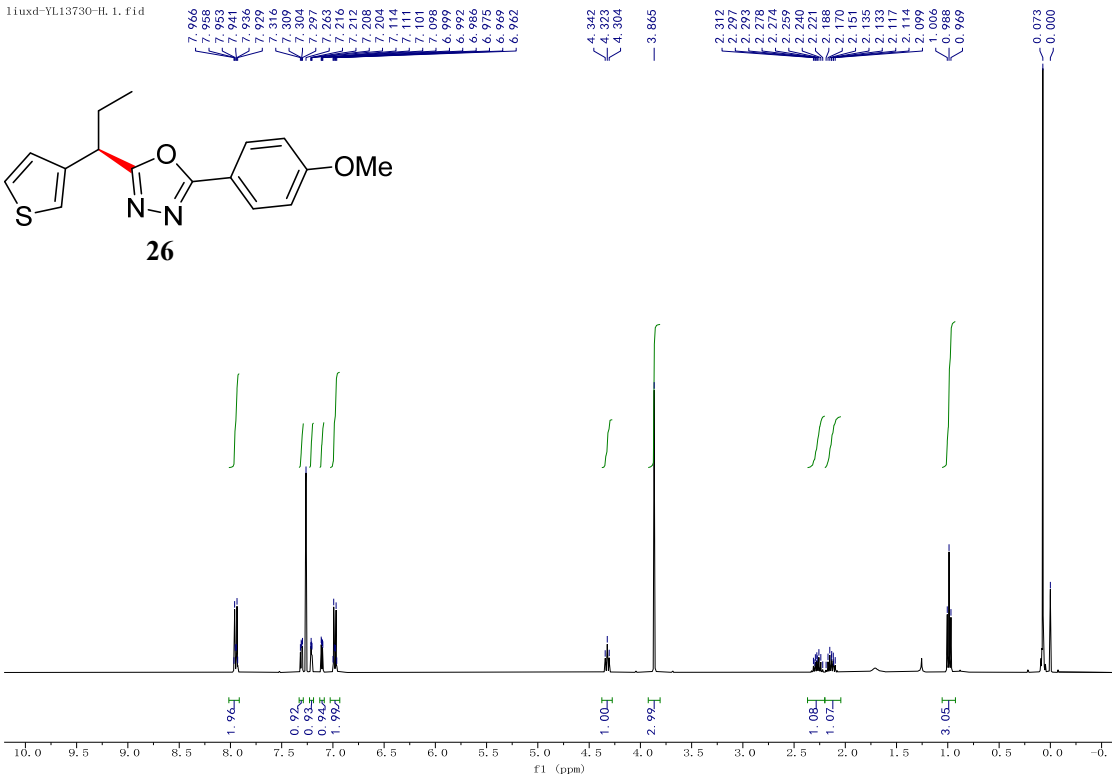
24


liuxd-K-C.1.fid
162.202
141.765
137.872
135.154
128.621
128.396
126.861
126.315
126.252
120.408
116.550
114.390
77.368
77.051
76.733
55.447
44.781
27.503
12.203
1.045
OMe
24
f1 (ppm)

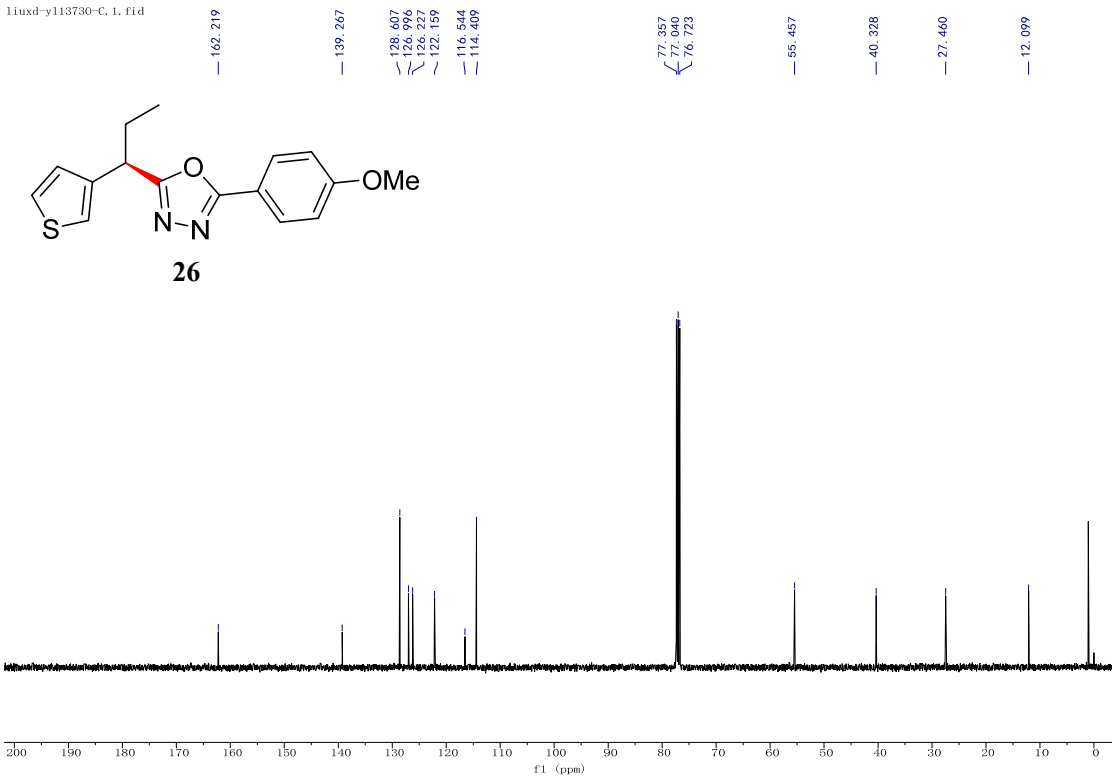

4.319
4.300
4.281
3.837
2.477
2.461
2.458
2.442
2.439
2.421
2.405
2.387
2.269
2.250
2.234
2.231
2.215
2.212
2.196
1.038
1.019
1.001
0.000
OMe
25
2.01
3.94
2.99
2.04
1.04
2.94
1.02
1.04
2.98
f1 (ppm)

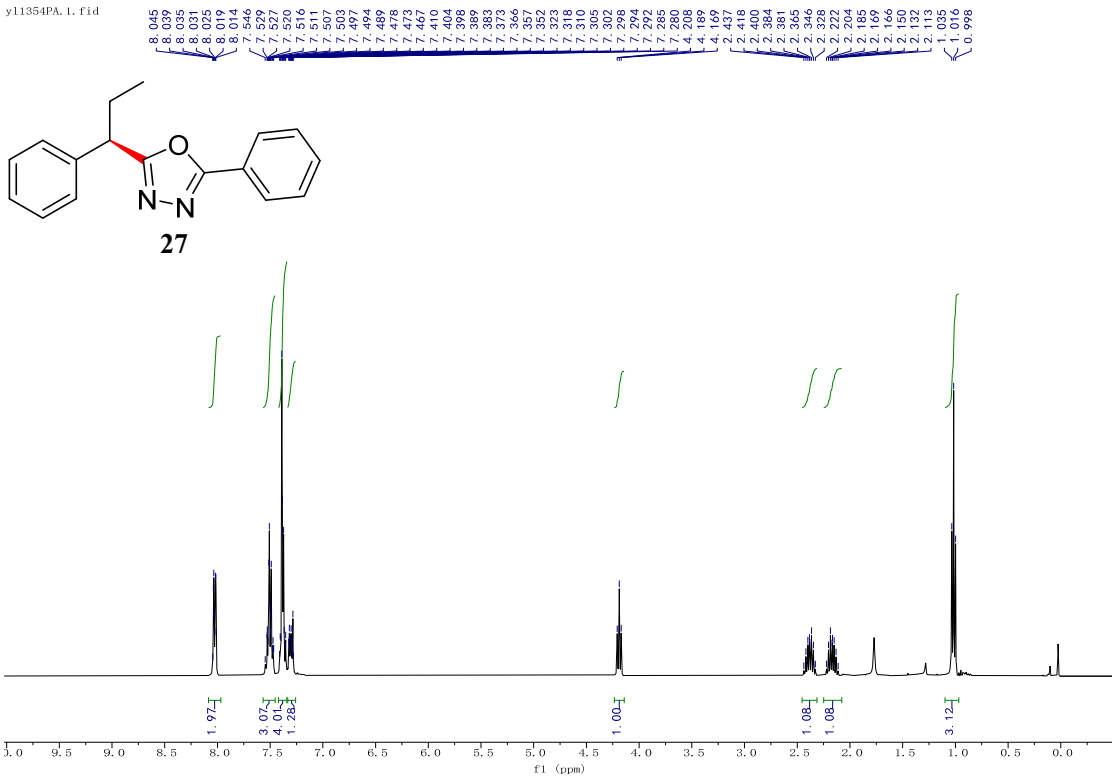

yl1361PB.10.1.1r
167.597
164.873
162.187
136.436
133.430
132.729
128.679
128.619
127.834
127.698
126.924
126.353
125.066
125.083
115.323
114.371
77.374
77.056
76.739
55.435
45.244
27.391
12.242
OMe
O
N
N
f1 (ppm)

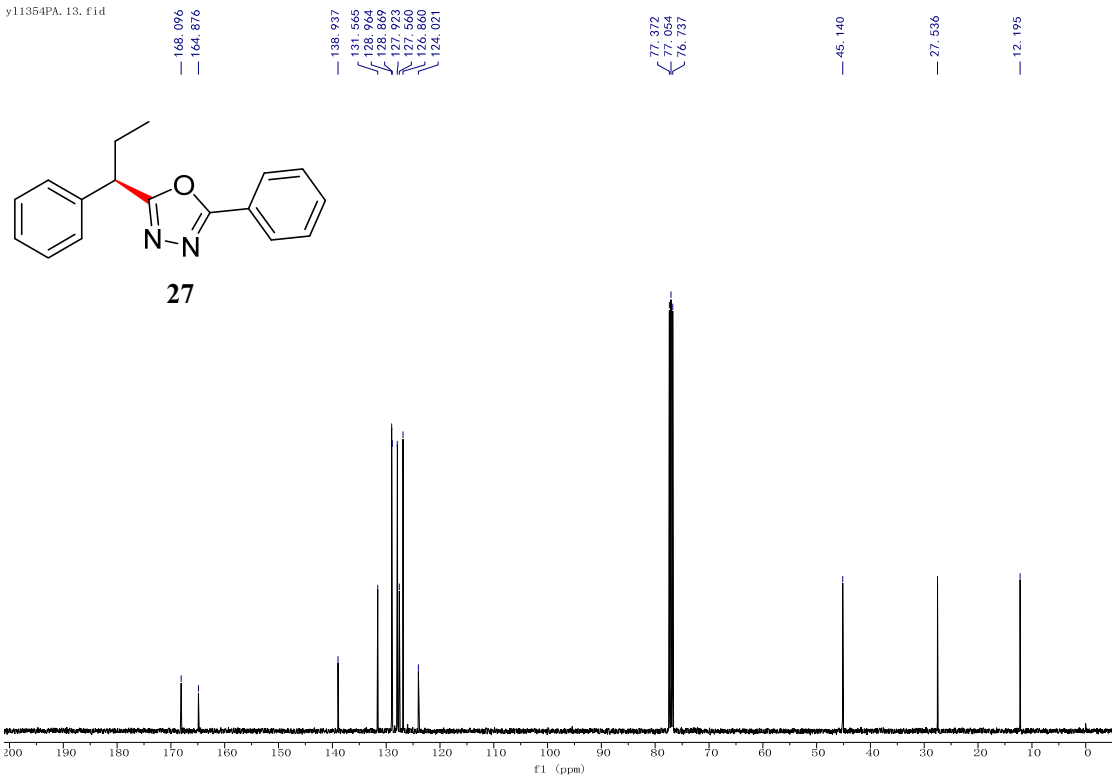
25

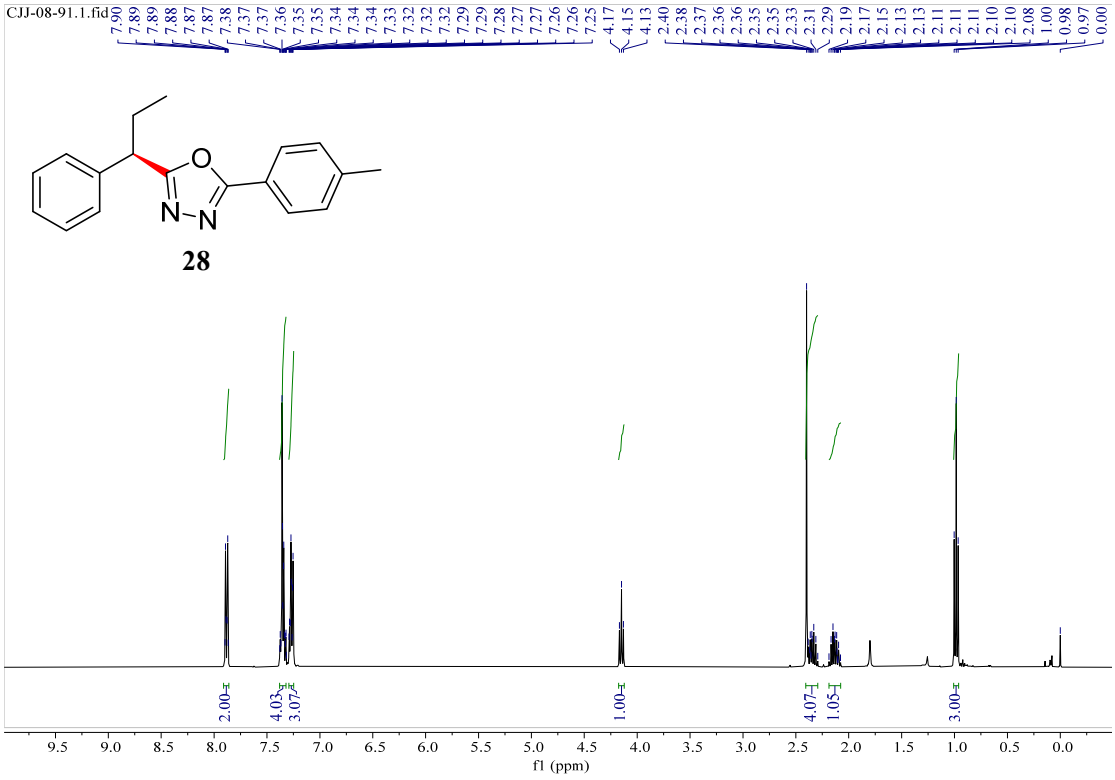

liuxd-YL13730-H.1.fid
7.966
7.958
7.953
7.941
7.936
7.929
7.316
7.309
7.304
7.297
7.263
7.216
7.212
7.208
7.204
7.114
7.111
7.101
7.098
6.999
6.992
6.986
6.975
6.969
6.962
4.342
4.323
4.304
3.865
2.312
2.297
2.293
2.278
2.274
2.259
2.240
2.221
2.188
2.170
2.151
2.135
2.133
2.117
2.114
2.099
1.006
0.988
0.969
0.073
0.000
1.94
0.92
0.93
0.94
1.99
1.00
2.99
1.08
1.07
3.05
OMe
O
N
N
S
f1 (ppm)

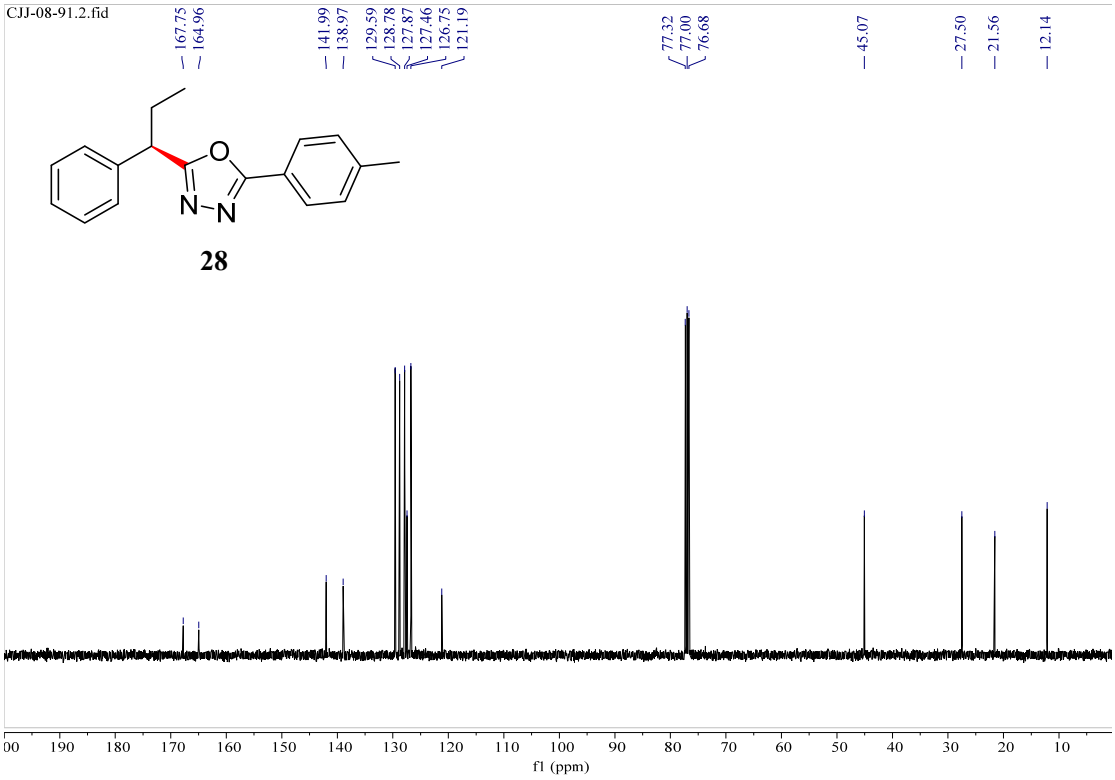
26

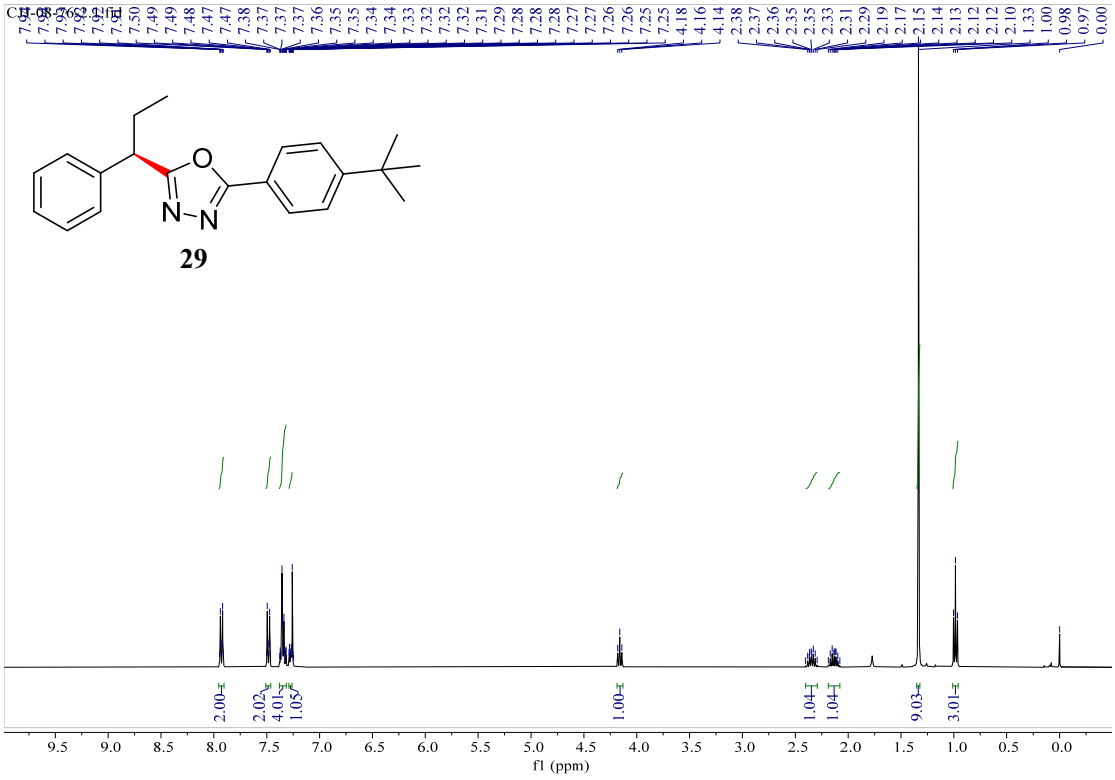

liuxd-yl13730-C.1.fid
162.219
139.267
128.607
126.996
126.227
122.159
116.544
114.409
77.357
77.040
76.723
55.457
40.328
27.460
12.099
OMe
O
N
N
S
26
f1 (ppm)

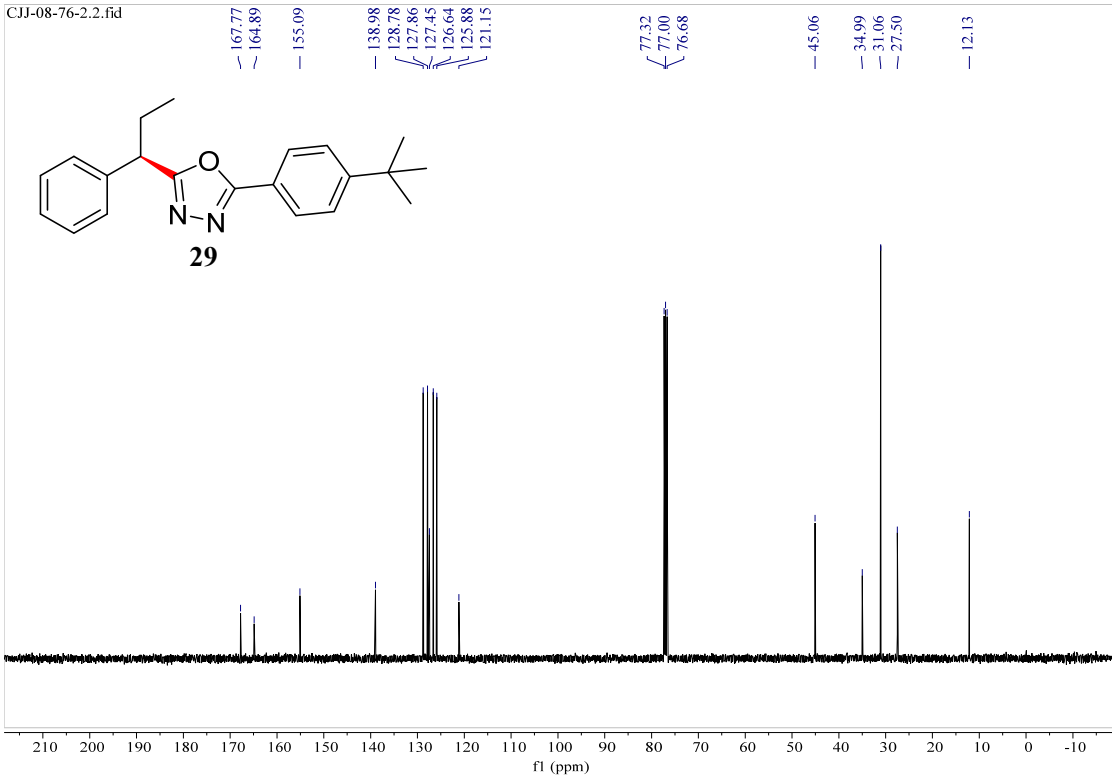

yl1354PA.1.fid
8.045
8.039
8.035
8.031
8.025
8.019
8.014
7.546
7.529
7.527
7.520
7.516
7.511
7.507
7.503
7.497
7.494
7.489
7.478
7.473
7.467
7.410
7.404
7.398
7.389
7.383
7.373
7.366
7.357
7.352
7.323
7.318
7.310
7.305
7.302
7.298
7.294
7.292
7.285
7.280
7.208
4.189
4.169
2.437
2.418
2.400
2.384
2.381
2.365
2.346
2.328
2.222
2.204
2.185
2.169
2.166
2.150
2.132
2.113
1.035
1.016
0.998
O
N
N
27
1.97
3.07
4.01
1.28
1.00
1.08
1.08
3.12
f1 (ppm)

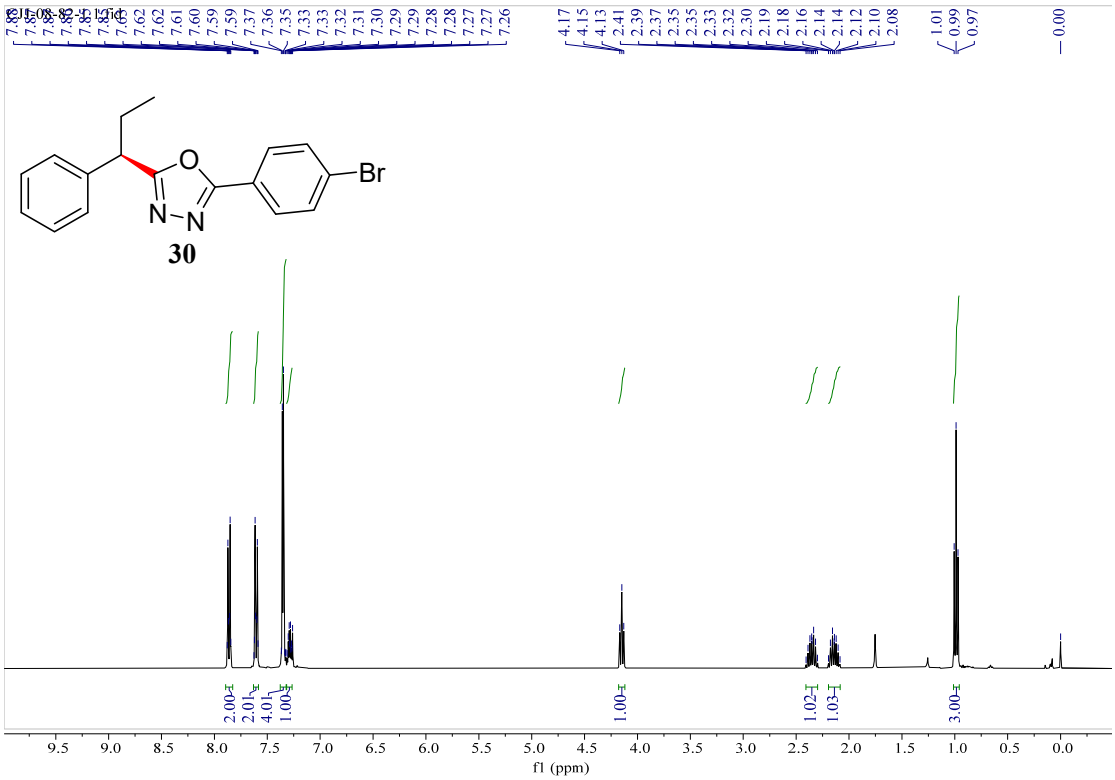

y11354PA.13.fid
168.096
164.876
138.937
131.565
128.964
128.869
127.923
127.860
126.860
124.021
77.372
77.054
76.737
45.140
27.536
12.195
O
N
N
27
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

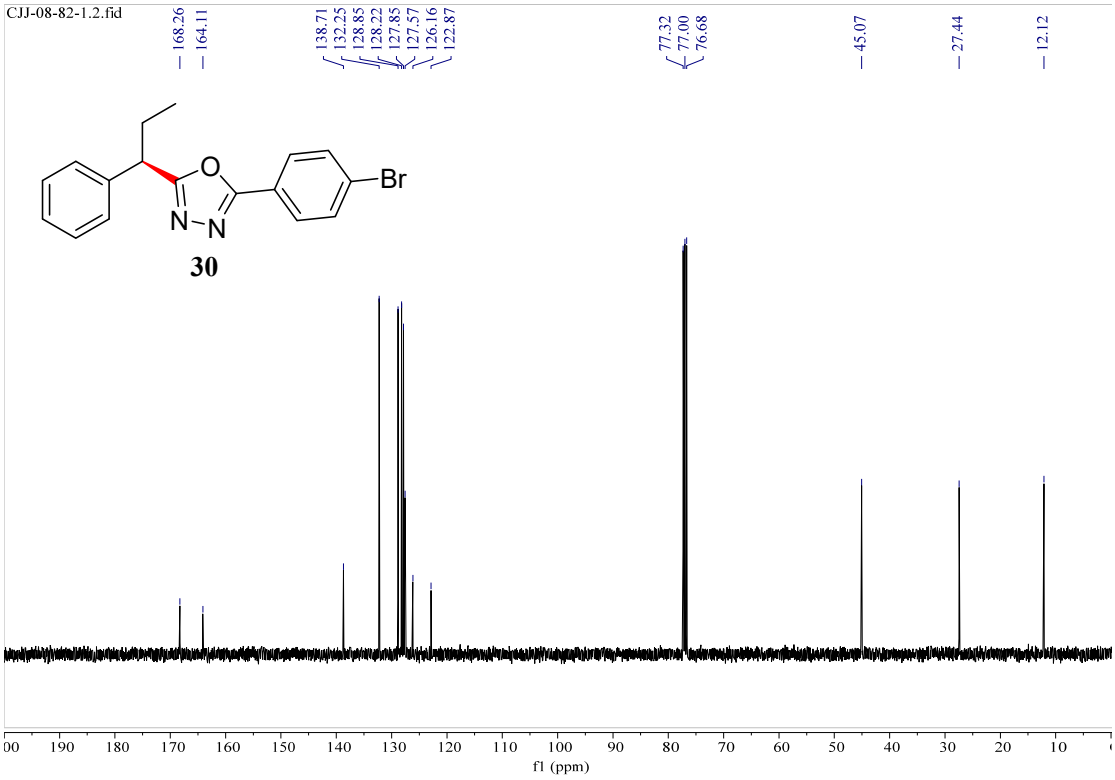

CJJ-08-91.1.fid
7.90
7.89
7.89
7.88
7.87
7.87
7.38
7.37
7.37
7.36
7.35
7.35
7.34
7.34
7.33
7.32
7.32
7.32
7.29
7.29
7.28
7.27
7.27
7.26
7.26
7.25
4.17
4.15
4.13
2.40
2.38
2.37
2.36
2.36
2.35
2.35
2.33
2.31
2.29
2.19
2.17
2.15
2.13
2.13
2.11
2.11
2.11
2.10
2.10
2.08
1.00
0.98
0.97
0.00
O
N
N
28
2.00
4.03
3.07
1.00
4.07
1.05
3.00
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
0.0
f1 (ppm)

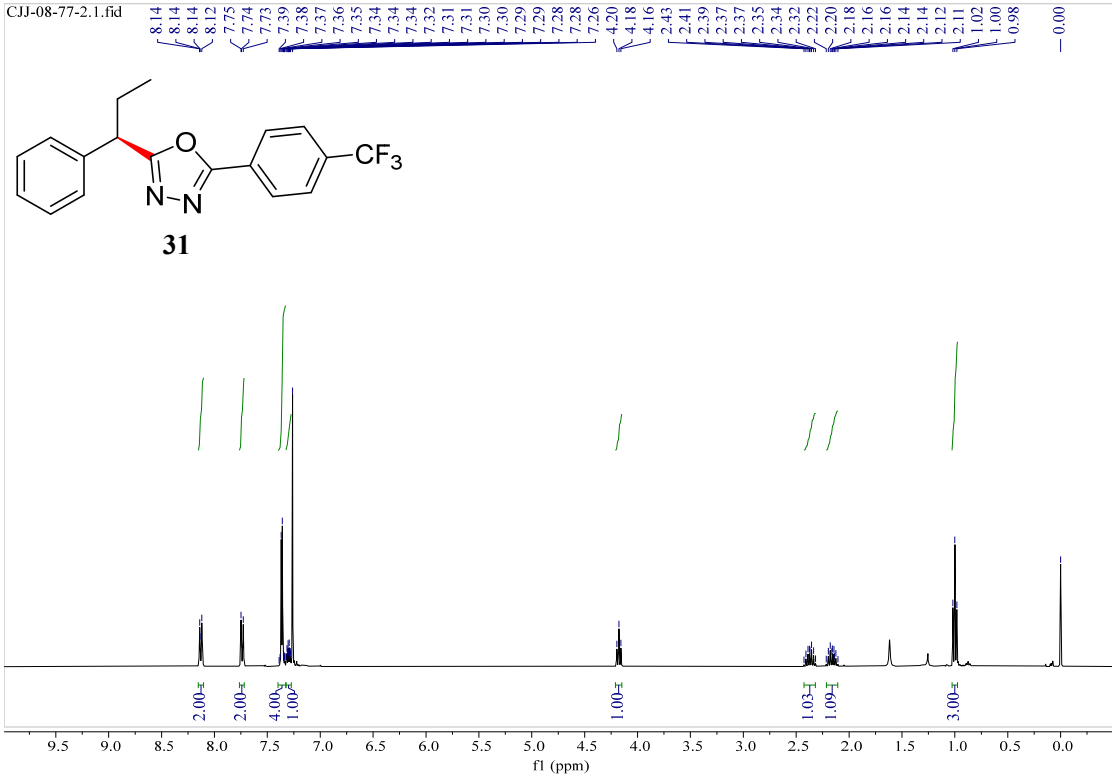

CJJ-08-91.2.fid
167.75
164.96
141.99
138.97
129.59
128.78
127.87
127.46
126.75
121.19
77.32
77.00
76.68
45.07
27.50
21.56
12.14
O
N
N
28
f1 (ppm)

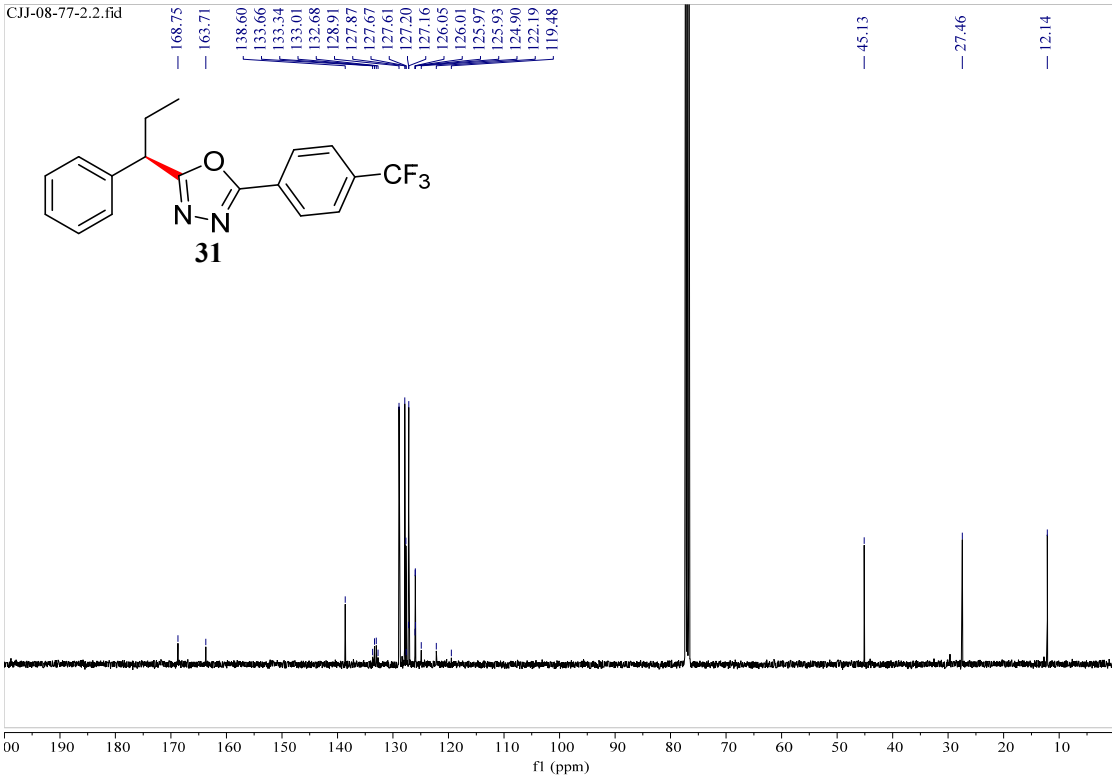

CJJ-08-76.2.fid
O
N
N
29
2.00
2.02
4.01
1.05
1.00
1.04
1.04
9.03
3.01
f1 (ppm)

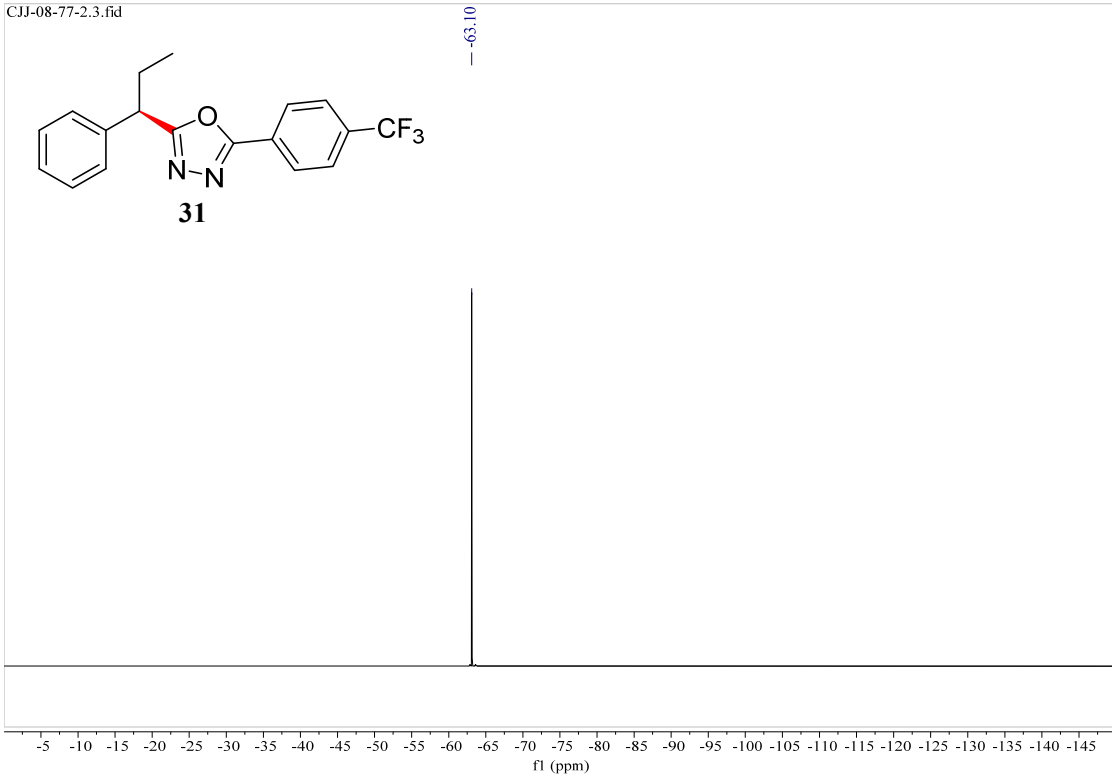

CJJ-08-76-2.2.fid
167.77
164.89
155.09
138.98
128.78
127.86
127.45
126.64
125.88
121.15
77.32
77.00
76.68
45.06
34.99
31.06
27.50
12.13
29
f1 (ppm)

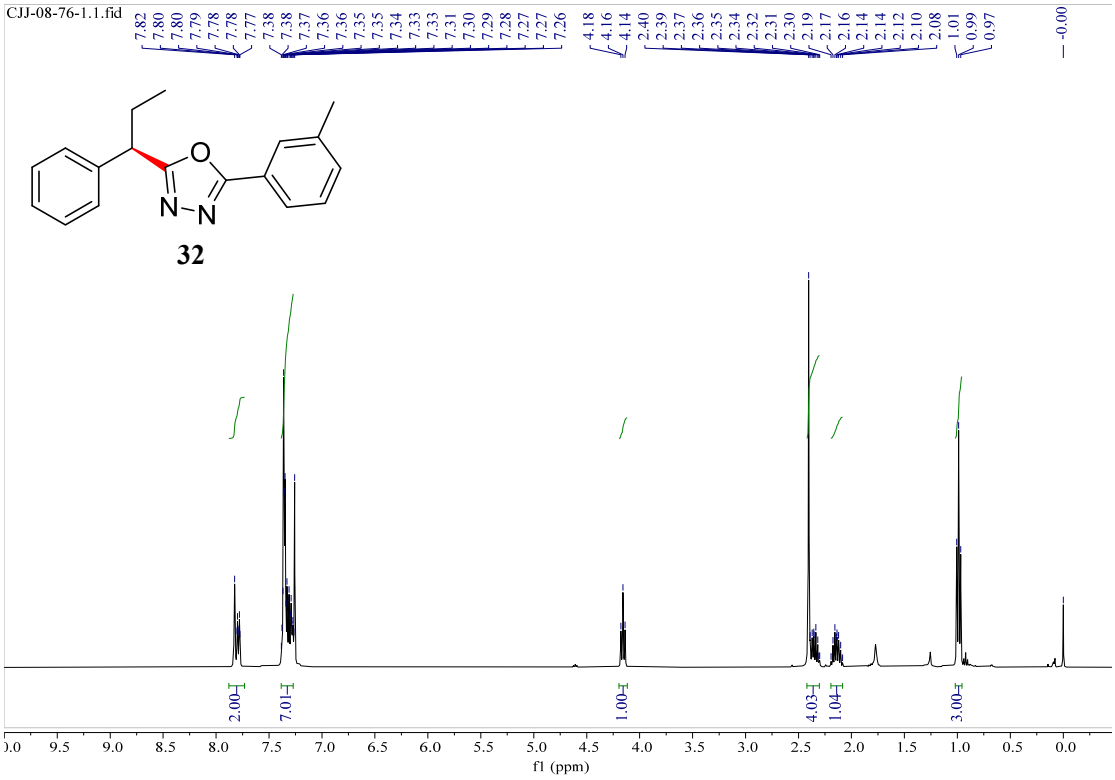

CJJ-08-82-1
4.17
4.15
4.13
2.41
2.39
2.37
2.35
2.33
2.32
2.30
2.19
2.18
2.16
2.14
2.14
2.12
2.10
2.08
1.01
0.99
0.97
0.00
Br
30
2.00
2.01
4.01
1.00
1.00
1.02
1.03
3.00
f1 (ppm)

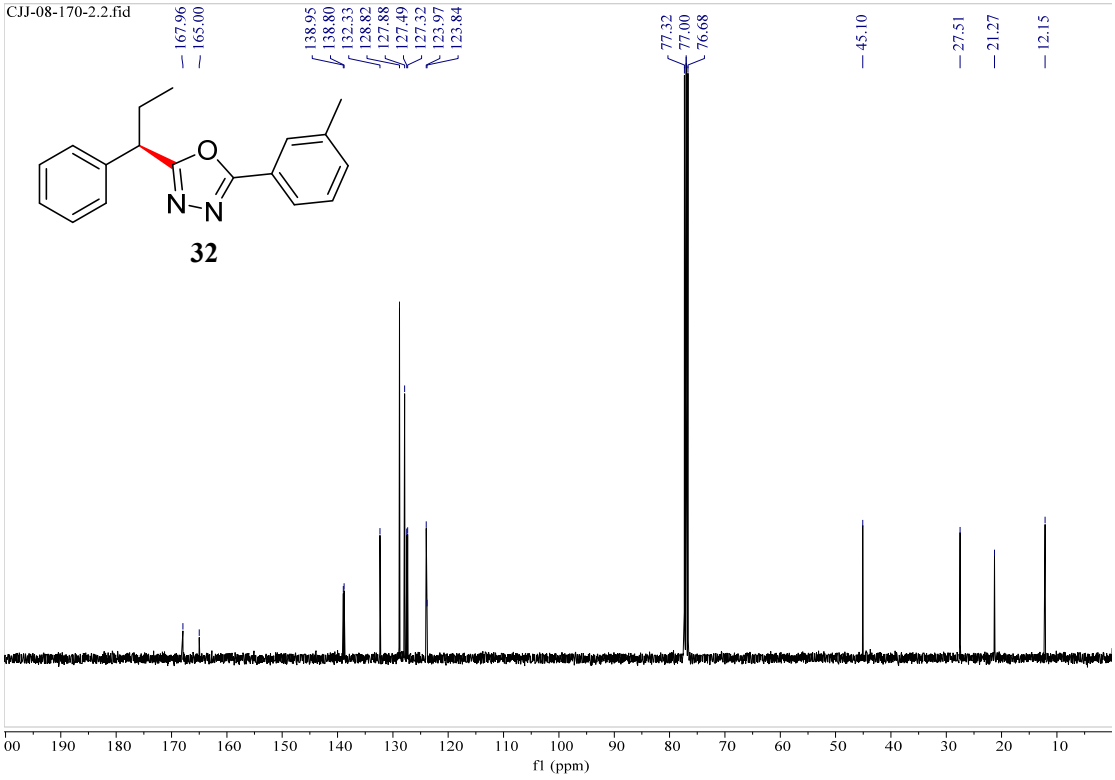

CJJ-08-82-1.2.fid
168.26
164.11
138.71
132.25
128.85
128.22
127.85
127.57
126.16
122.87
77.32
77.00
76.68
45.07
27.44
12.12
Br
O
N
N
30
f1 (ppm)

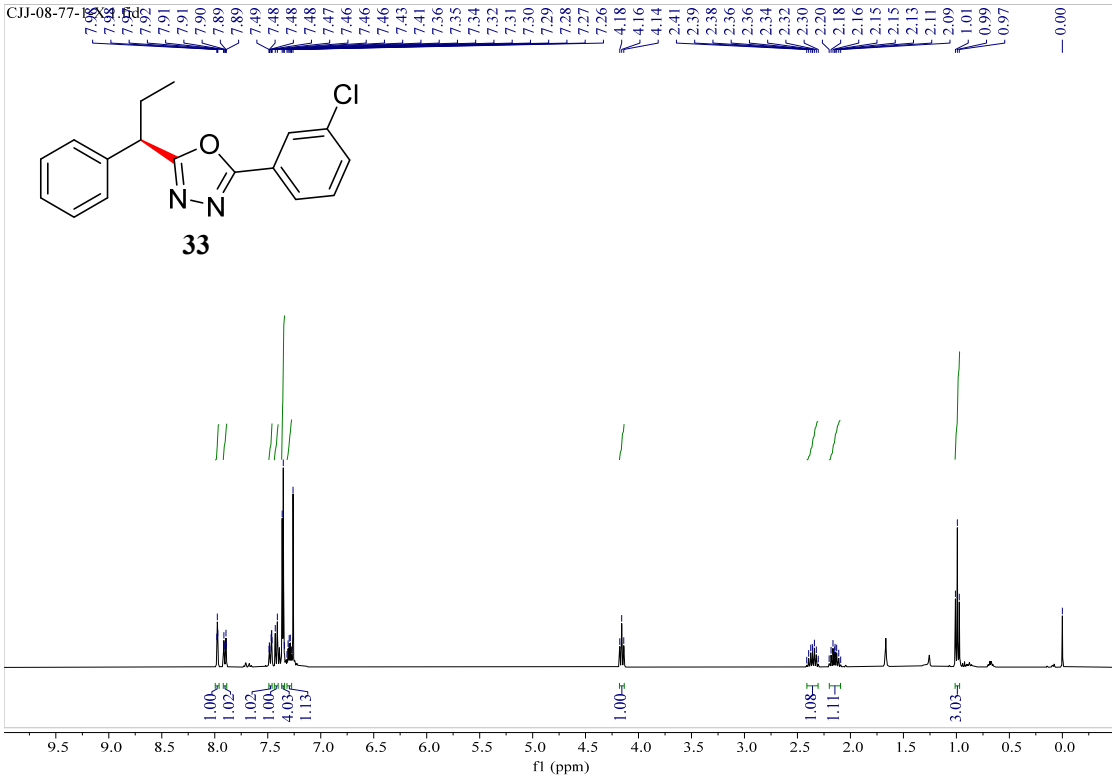

CJJ-08-77-2.1.fid
8.14
8.14
8.14
8.12
7.75
7.74
7.73
7.39
7.38
7.37
7.36
7.35
7.34
7.34
7.34
7.32
7.31
7.31
7.30
7.30
7.29
7.29
7.28
7.28
7.26
4.20
4.18
4.16
2.43
2.41
2.39
2.37
2.37
2.35
2.34
2.32
2.22
2.20
2.18
2.16
2.14
2.14
2.12
2.11
1.02
1.00
0.98
0.00
CF3
O
N
N
31
2.00
2.00
4.00
1.00
1.00
1.03
1.09
3.00
f1 (ppm)

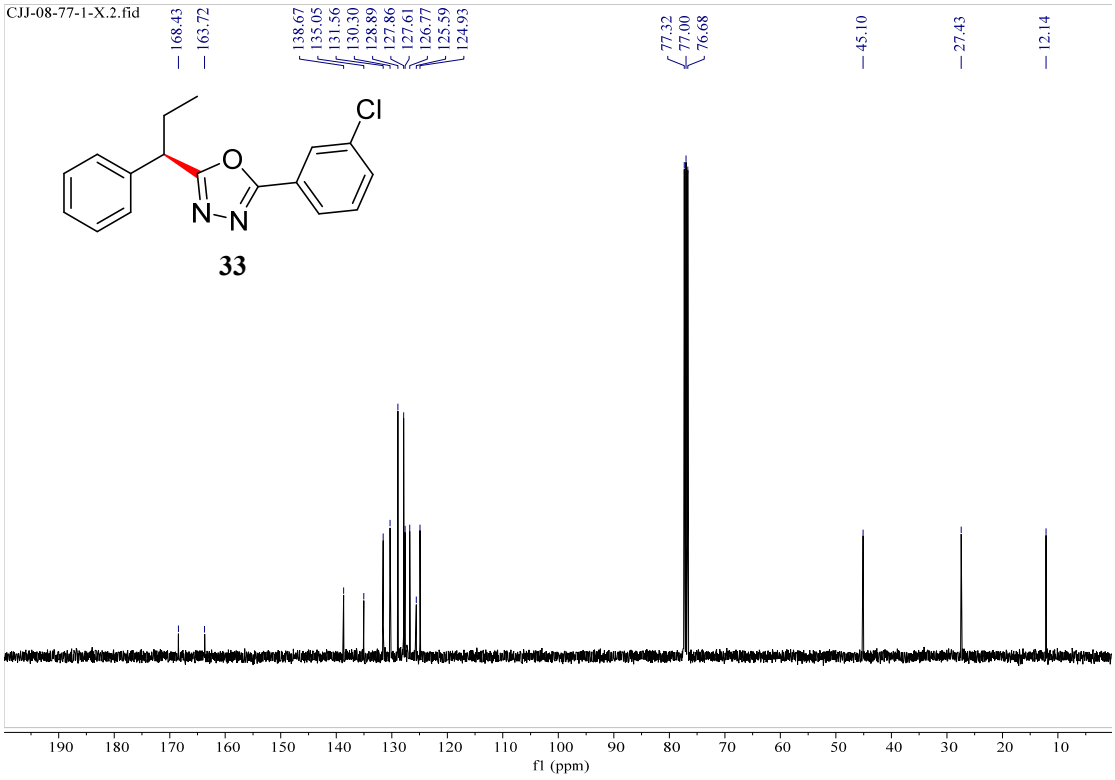

CJJ-08-77-2.2.fid
168.75
163.71
138.60
133.66
133.34
133.01
132.68
128.91
127.87
127.67
127.61
127.20
127.16
126.05
126.01
125.97
125.93
124.90
122.19
119.48
45.13
27.46
12.14
O
N
N
CF3
31
f1 (ppm)

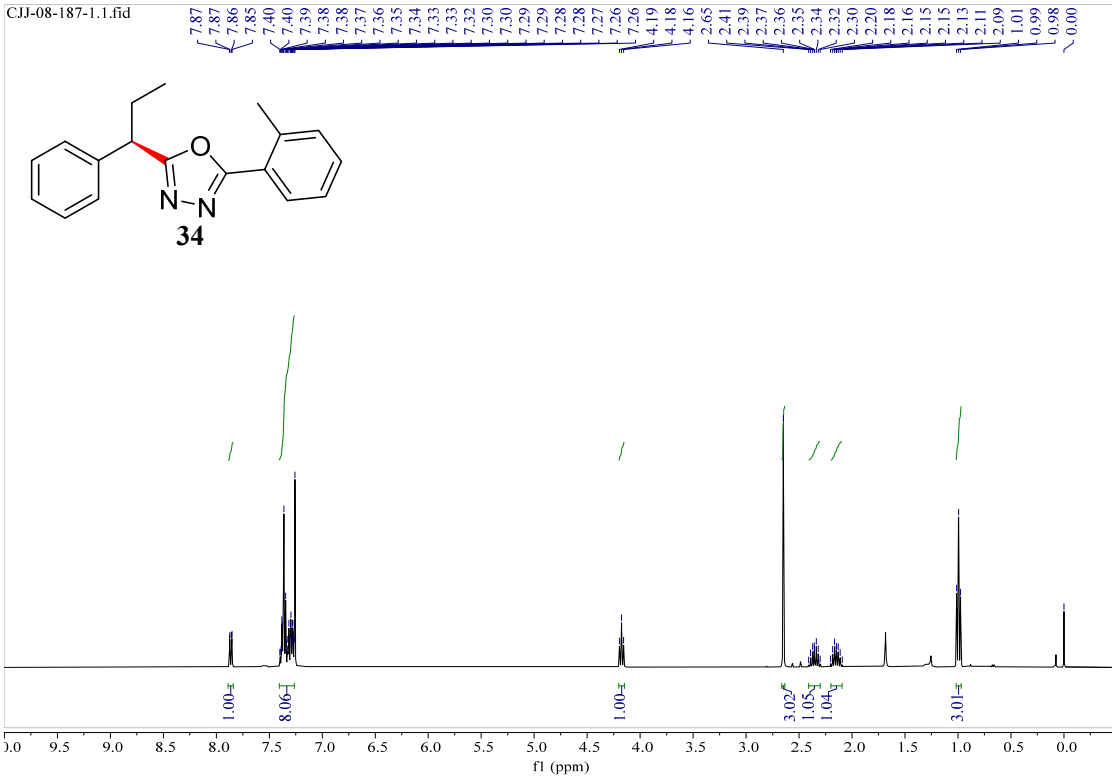

CJJ-08-77-2.3.fid
-63.10
O
N
N
CF3
31
f1 (ppm)

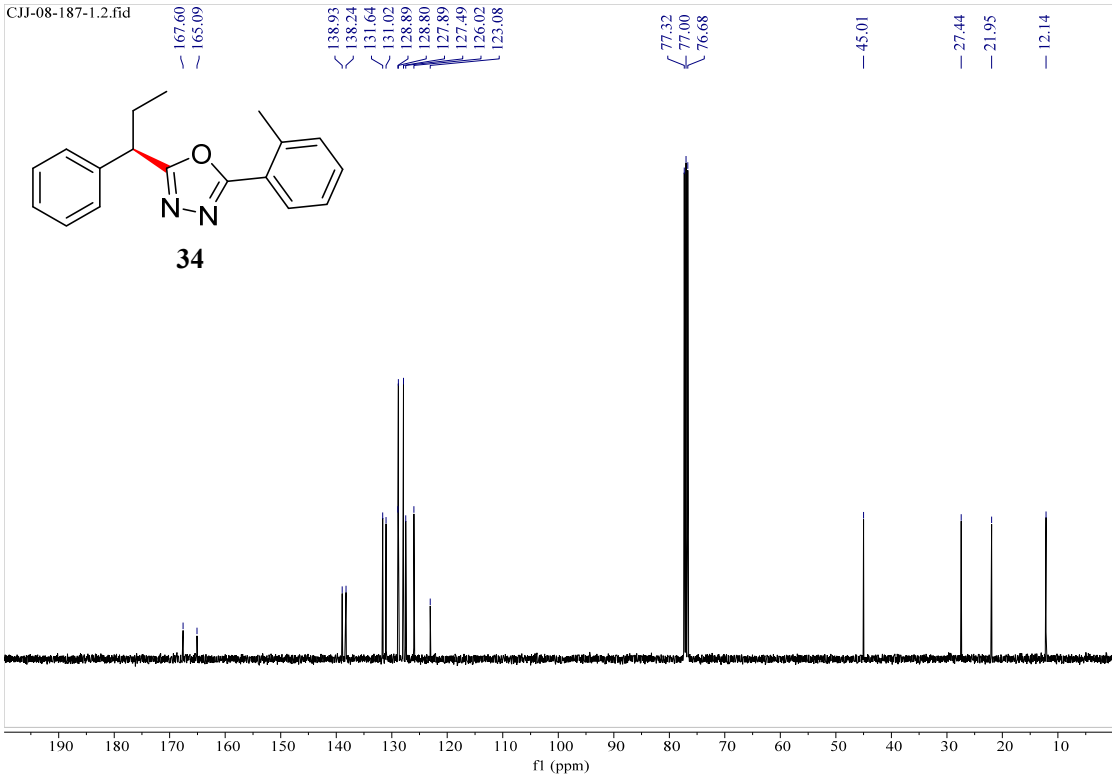

CJJ-08-76-1.1.fid
7.82 7.80 7.80 7.79 7.78 7.78 7.77 7.38 7.38 7.37 7.36 7.36 7.35 7.35 7.34 7.33 7.33 7.31 7.30 7.29 7.28 7.27 7.27 7.26 4.18 4.16 4.14 2.40 2.39 2.37 2.36 2.35 2.34 2.32 2.31 2.30 2.19 2.17 2.16 2.14 2.14 2.12 2.10 2.08 1.01 0.99 0.97 -0.00
O
N
N
32
2.00 7.01 1.00 4.03 1.04 3.00
f1 (ppm)

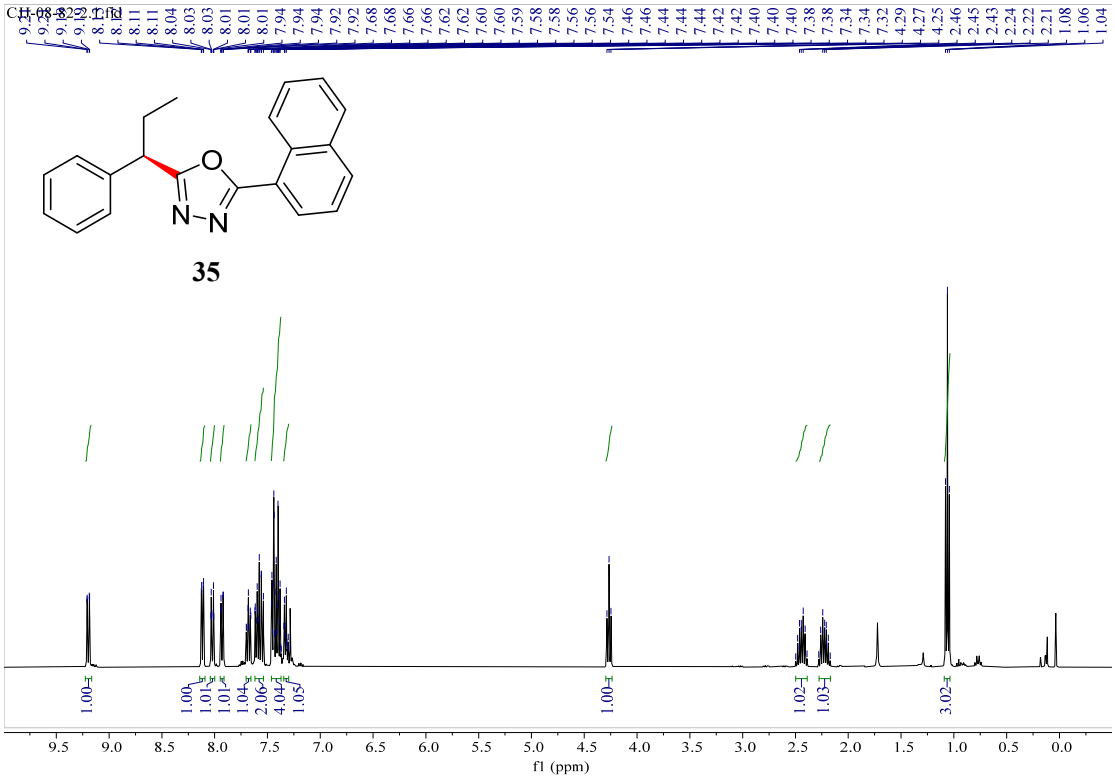

CJJ-08-170-2.2.fid
167.96 165.00 138.95 138.80 132.33 128.82 127.88 127.49 127.32 123.97 123.84 77.32 77.00 76.68 45.10 27.51 21.27 12.15
O
N
N
32
f1 (ppm)

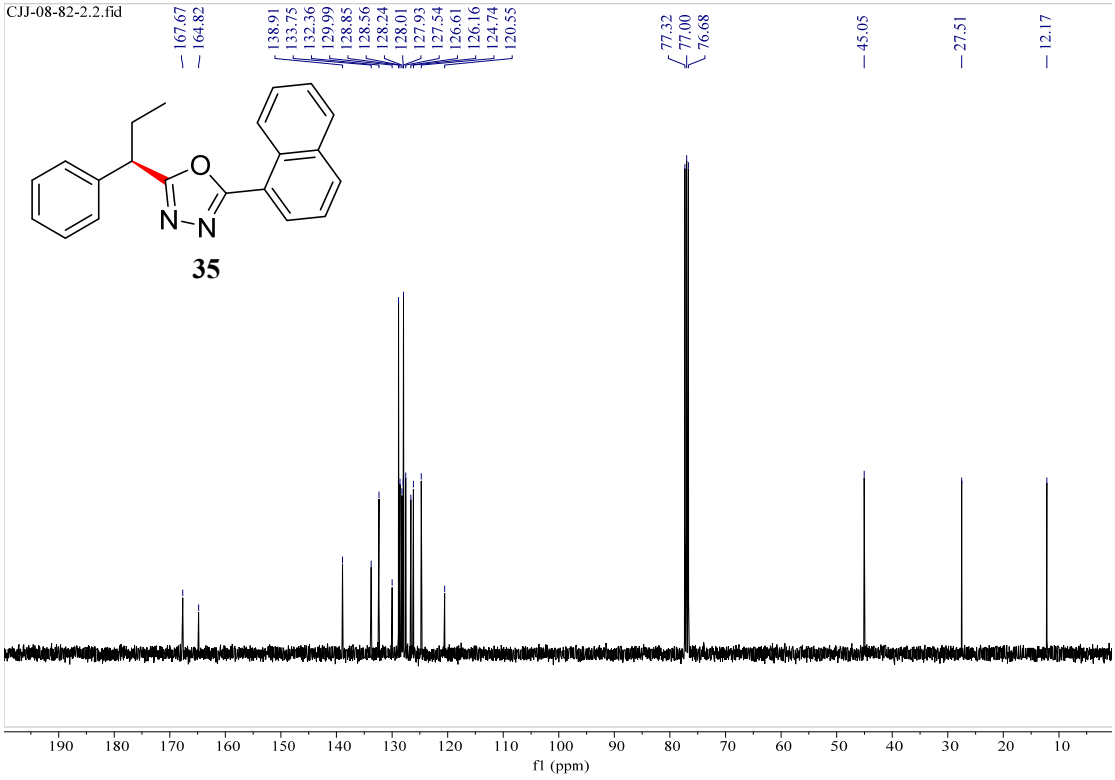

CJJ-08-77-1-X.1.fid
Cl
O
N
N
33
1.00
1.02
1.02
1.00
4.03
1.13
1.00
1.08
1.11
3.03
fl (ppm)

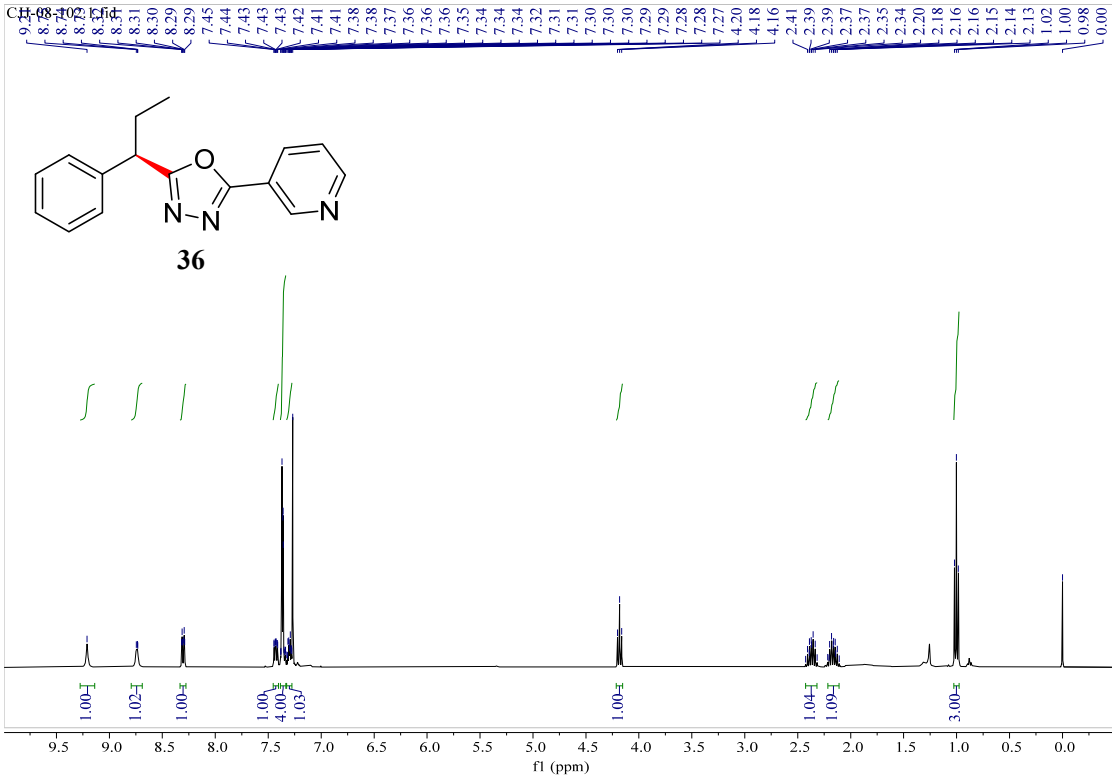

CJJ-08-77-1-X.2.fid
168.43
163.72
138.67
135.05
131.56
130.30
128.89
127.86
127.61
126.77
125.59
124.93
77.32
77.00
76.68
45.10
27.43
12.14
Cl
O
N
N
33
fl (ppm)

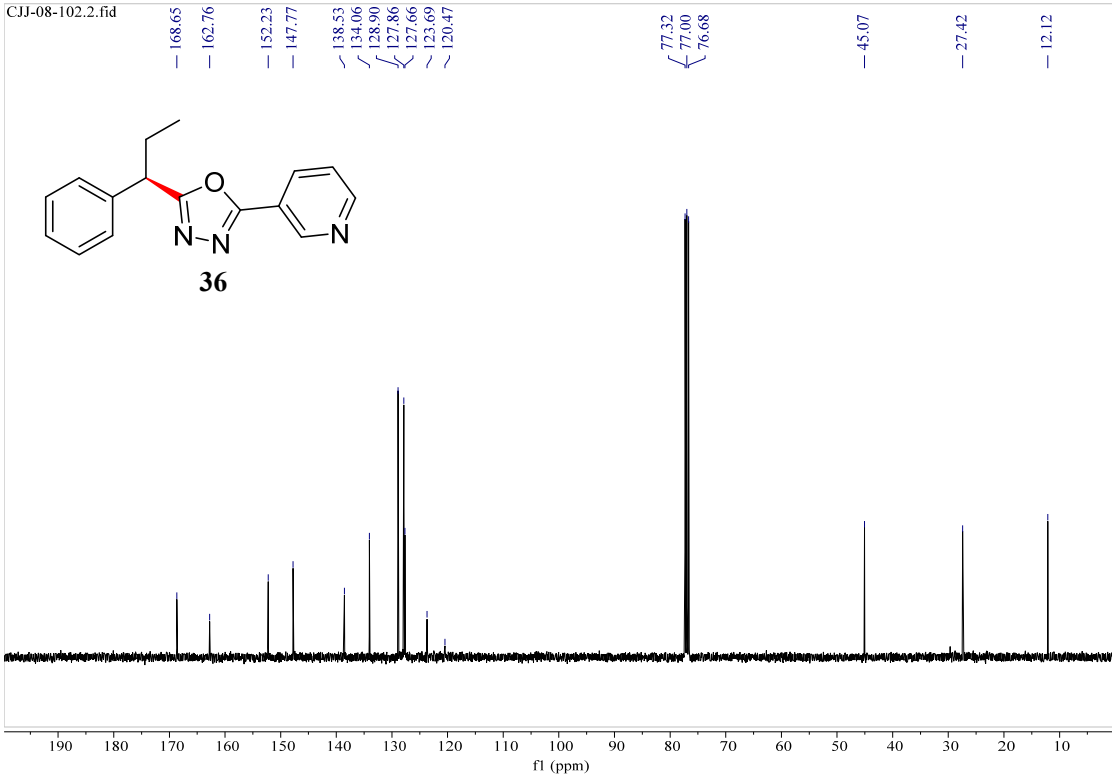

CJJ-08-187-1.1.fid
34
1.00
8.06
1.00
3.02
1.05
1.04
3.01
f1 (ppm)

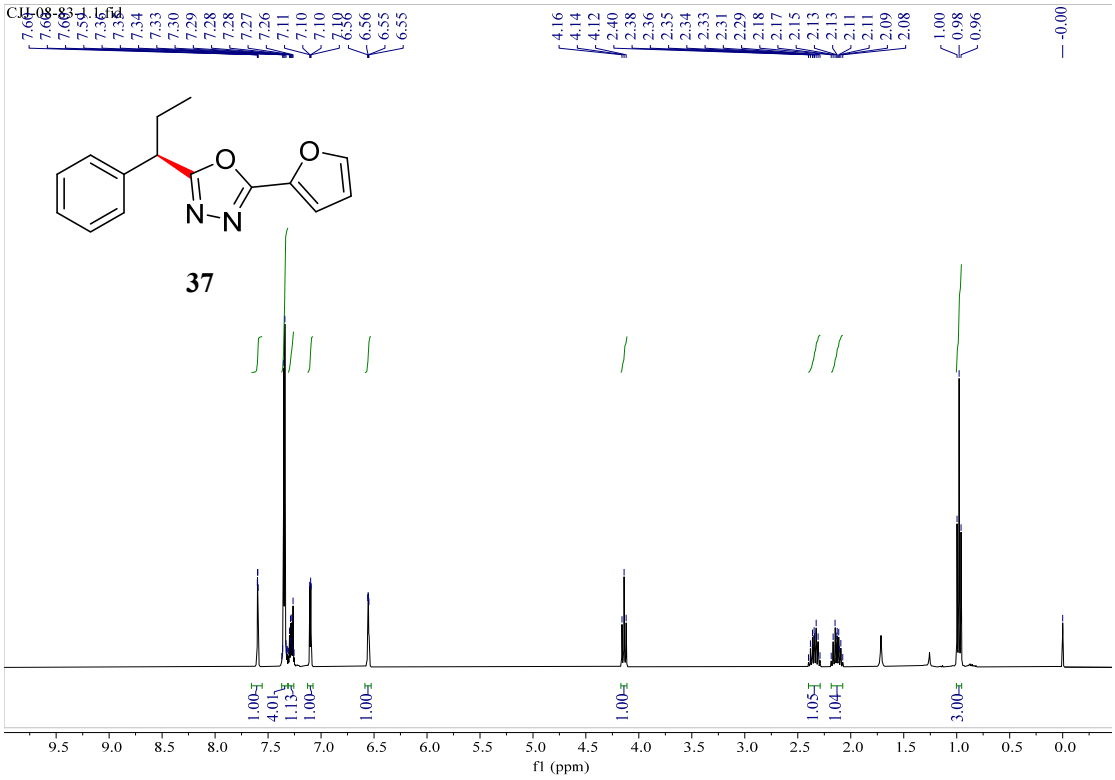

CJJ-08-187-1.2.fid
167.60
165.09
138.93
138.24
131.64
131.02
128.89
128.80
127.89
127.49
126.02
123.08
77.32
77.00
76.68
45.01
27.44
21.95
12.14
34
f1 (ppm)

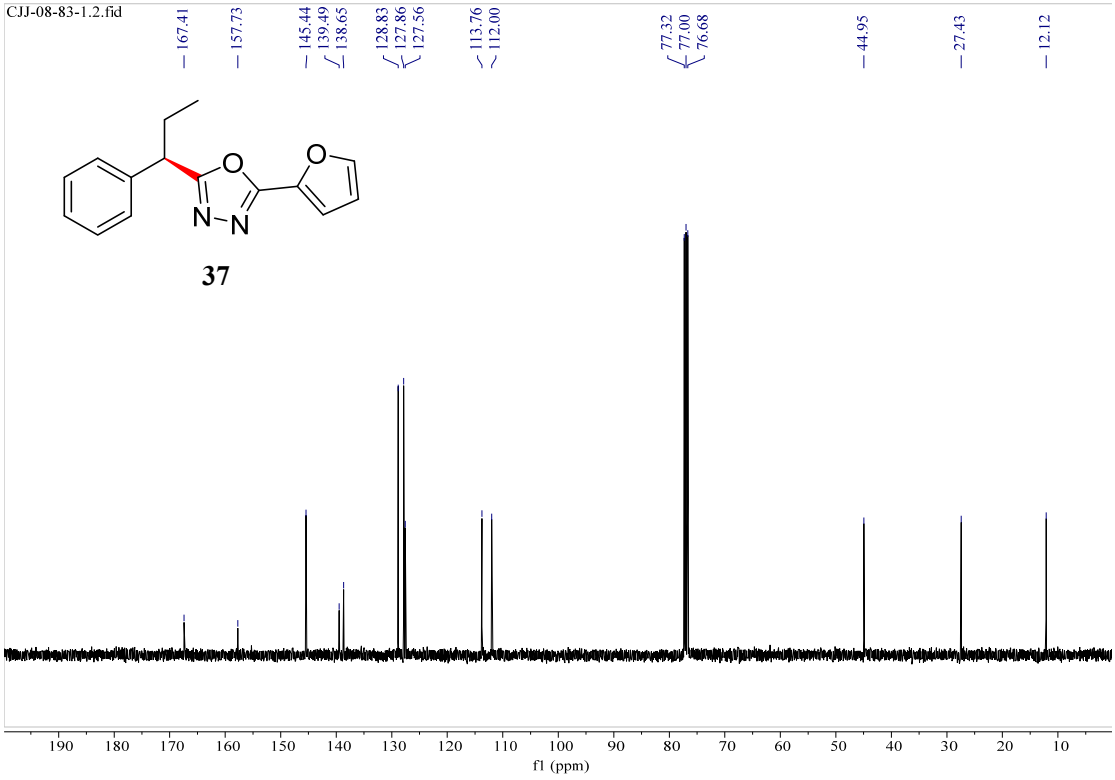

CJJ-08-82-1.1.fid
35
1.00
1.00
1.01
1.01
1.04
2.06
4.04
1.05
1.00
1.02
1.03
3.02
f1 (ppm)

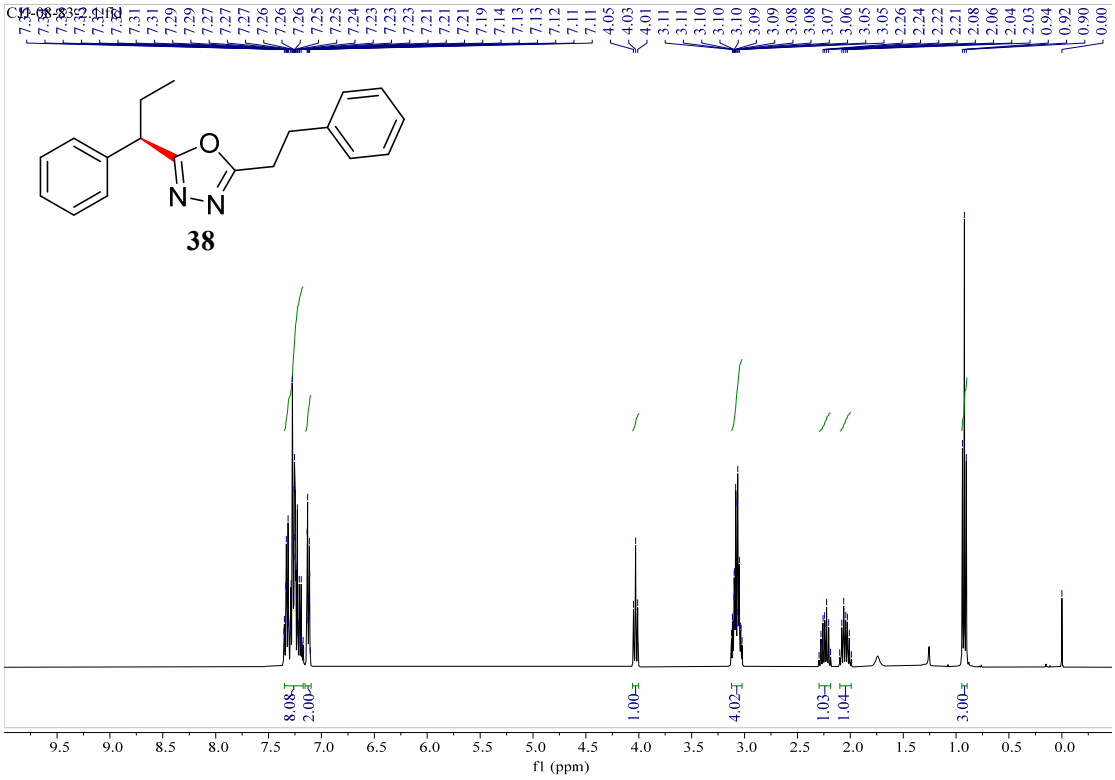

CJJ-08-82-2.2.fid
167.67
164.82
138.91
133.75
132.36
129.99
128.85
128.56
128.24
128.01
127.93
127.54
126.61
126.16
124.74
120.55
77.32
77.00
76.68
45.05
27.51
12.17
35
f1 (ppm)

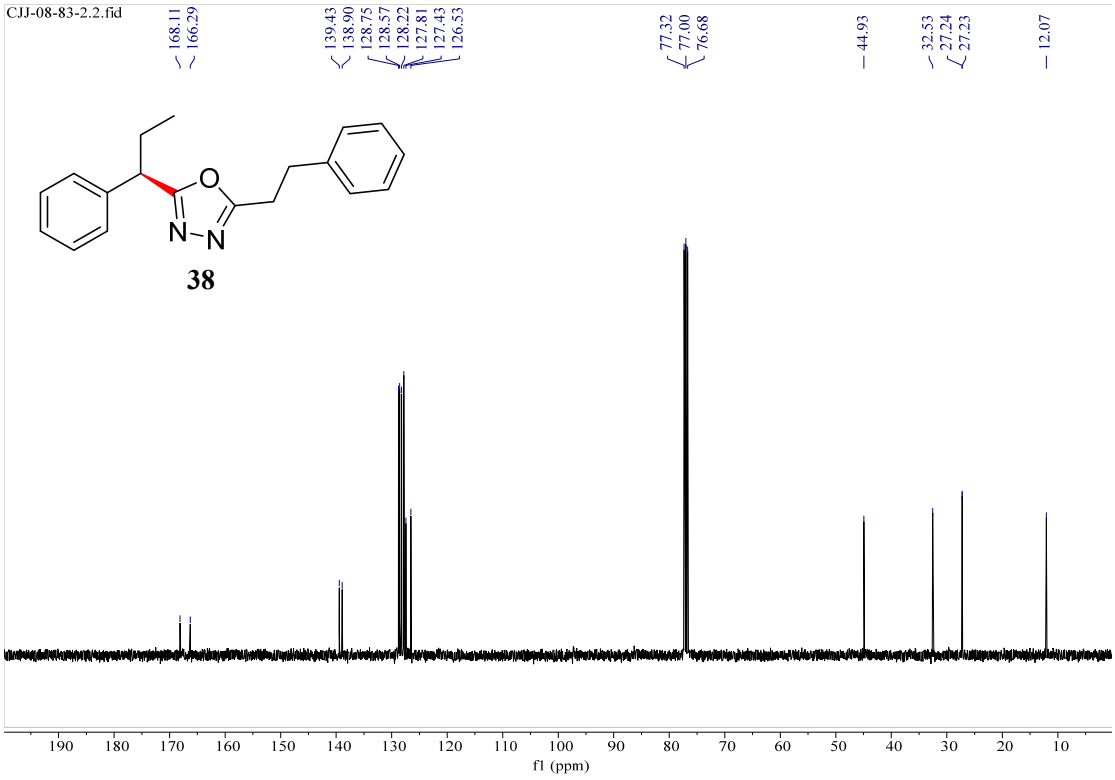

CJJ-08-102.1.fid
36
f1 (ppm)

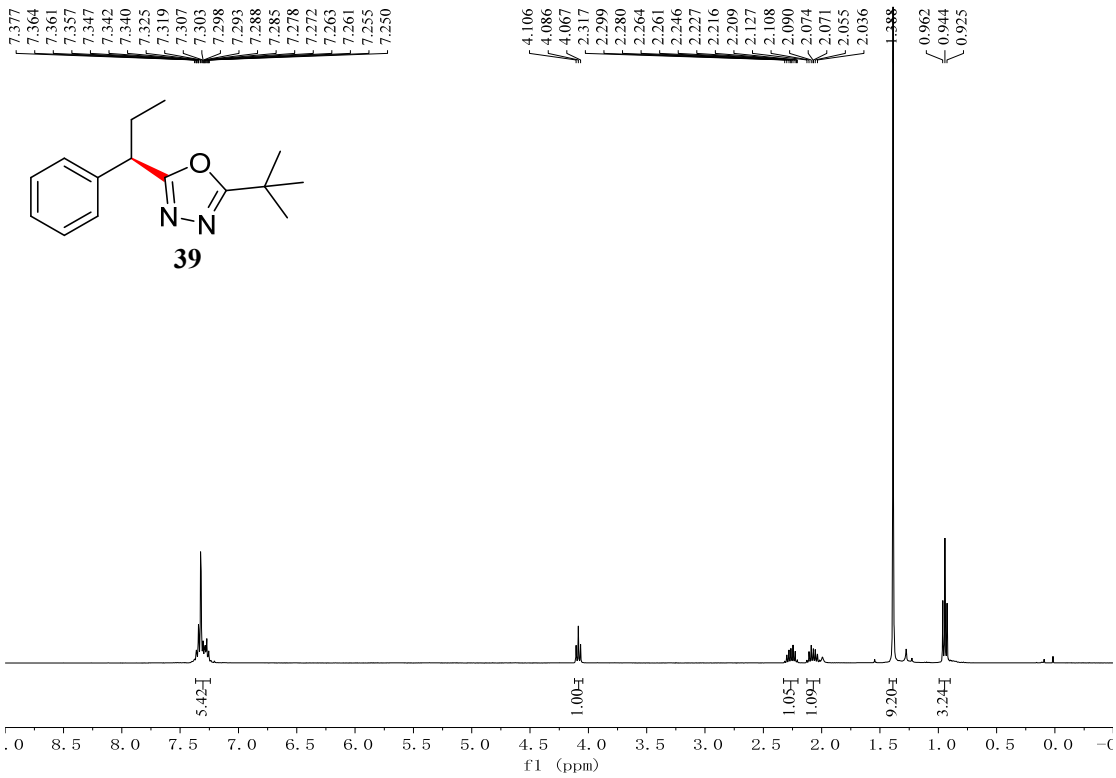

CJJ-08-102.2.fid
36
f1 (ppm)

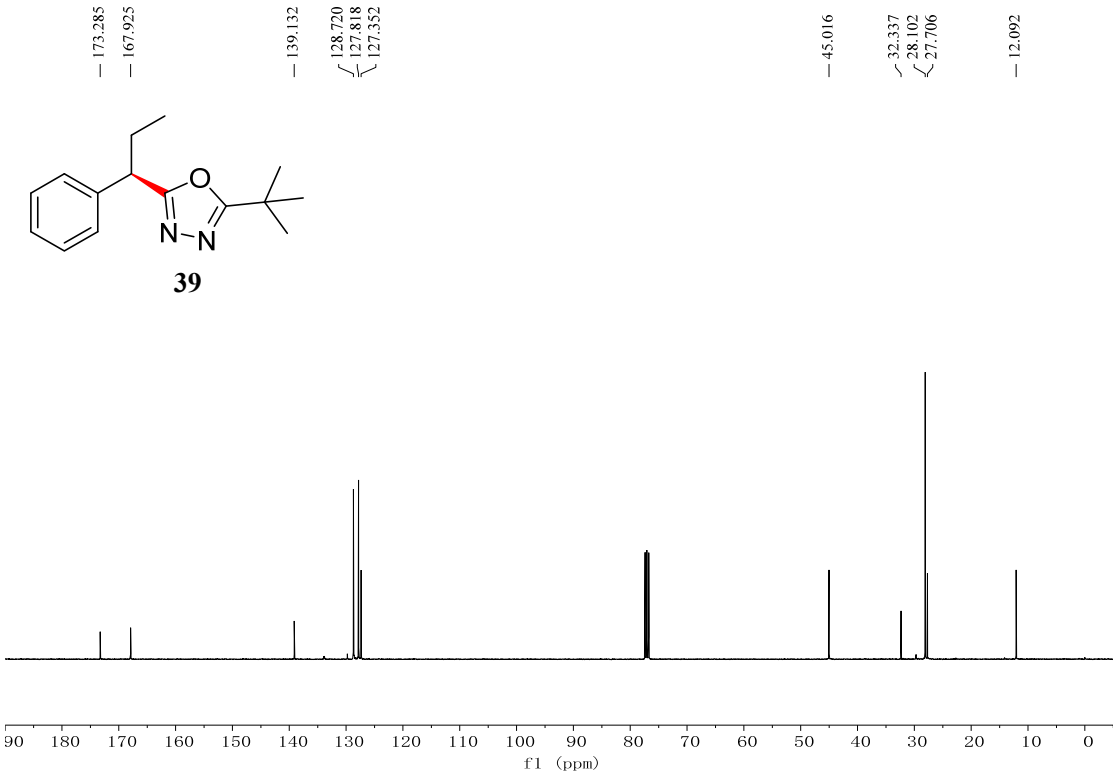

CJJ-08-83-1.1.fid
37
f1 (ppm)

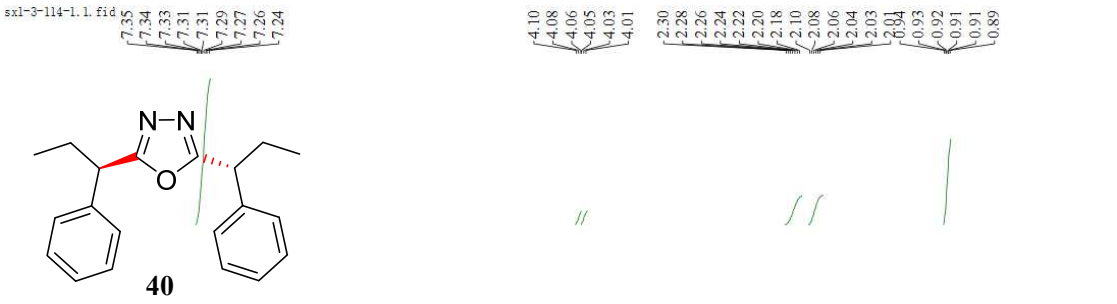

CJJ-08-83-1.2.fid
37
f1 (ppm)


CJJ-08-83-1.1.fid
38
8.08
2.00
1.00
4.02
1.03
1.04
3.00
f1 (ppm)

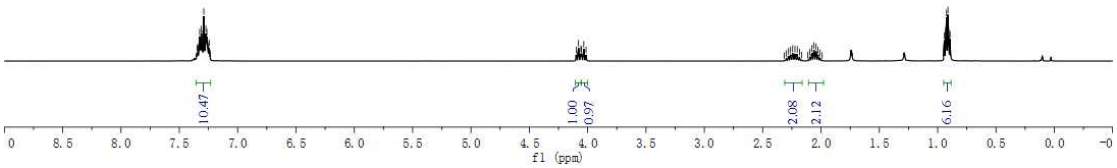

CJJ-08-83-2.2.fid
168.11
166.29
139.43
138.90
128.75
128.57
128.22
127.81
127.43
126.53
77.32
77.00
76.68
44.93
32.53
27.24
27.23
12.07
38
f1 (ppm)

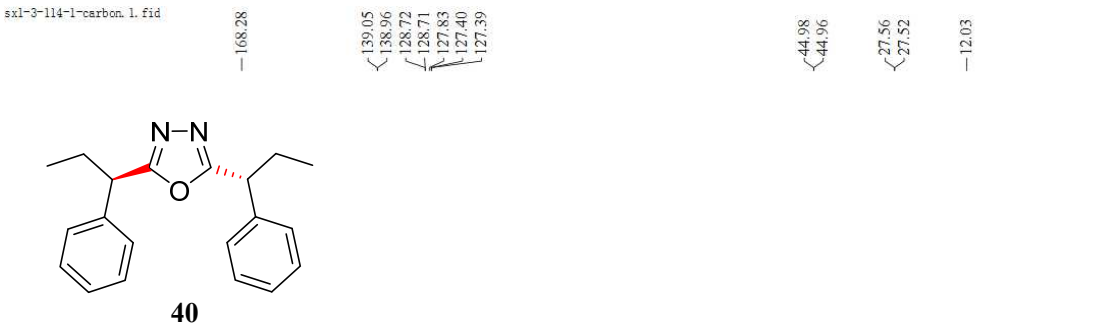

7.377
7.364
7.361
7.357
7.347
7.342
7.340
7.325
7.319
7.307
7.303
7.298
7.293
7.288
7.285
7.278
7.272
7.263
7.261
7.255
7.250
4.106
4.086
4.067
2.317
2.299
2.280
2.264
2.261
2.246
2.227
2.216
2.209
2.127
2.108
2.090
2.074
2.071
2.055
2.036
1.388
0.962
0.944
0.925
O
N
N
39
5.42
1.00
1.05
1.09
9.20
3.24
f1 (ppm)


173.285
167.925
139.132
128.720
127.818
127.352
45.016
32.337
28.102
27.706
12.092
O
N
N
39
f1 (ppm)

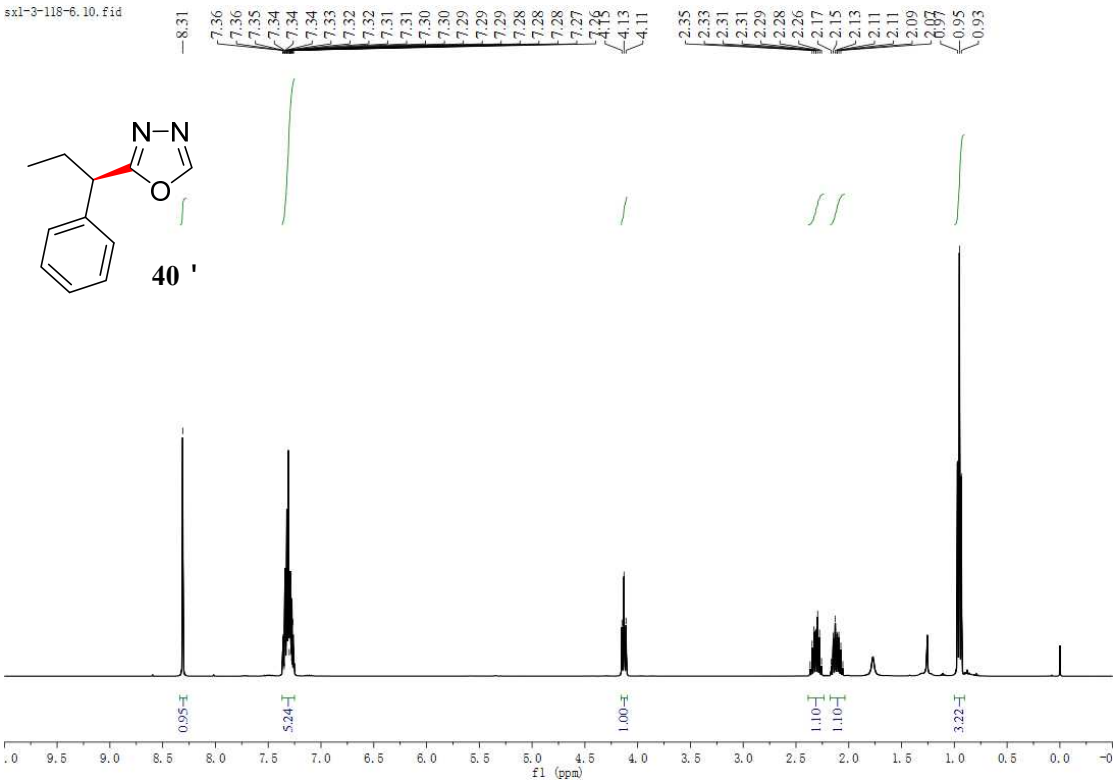

sxl-3-114-1.1.fid
7.35
7.34
7.33
7.31
7.31
7.29
7.27
7.26
7.24
4.10
4.08
4.06
4.05
4.03
4.01
2.30
2.28
2.26
2.24
2.22
2.20
2.18
2.10
2.08
2.06
2.04
2.03
2.01
0.94
0.93
0.92
0.91
0.91
0.89
N–N
O

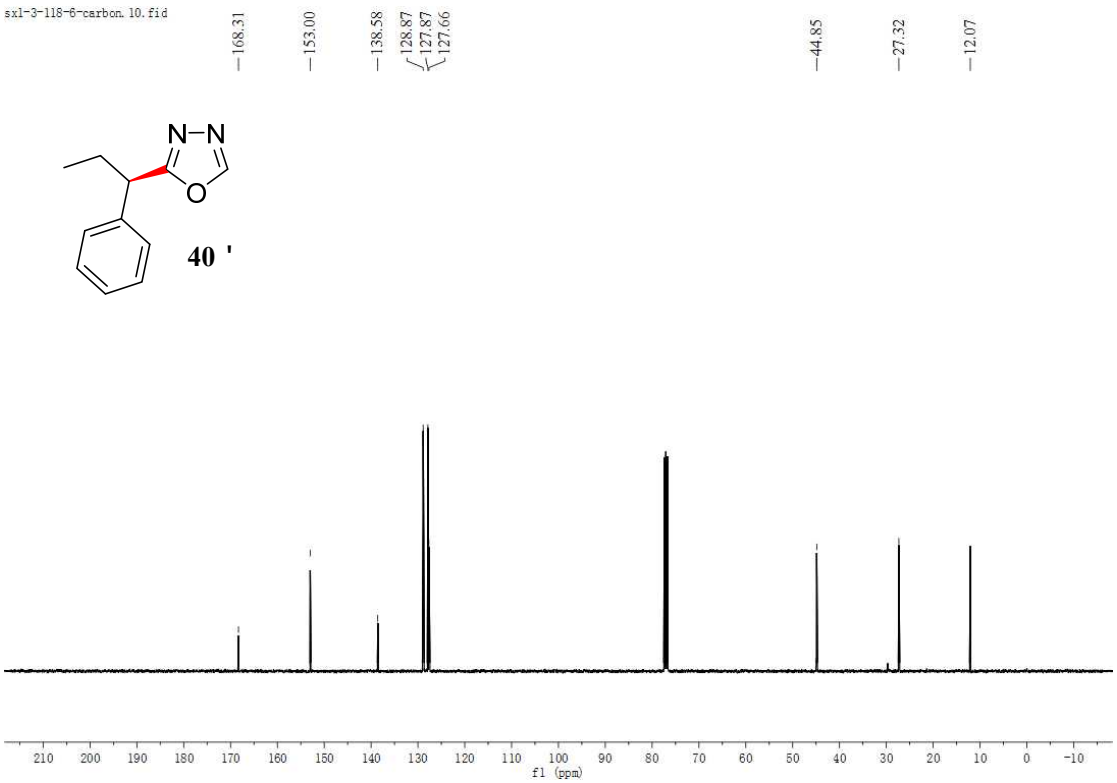
40

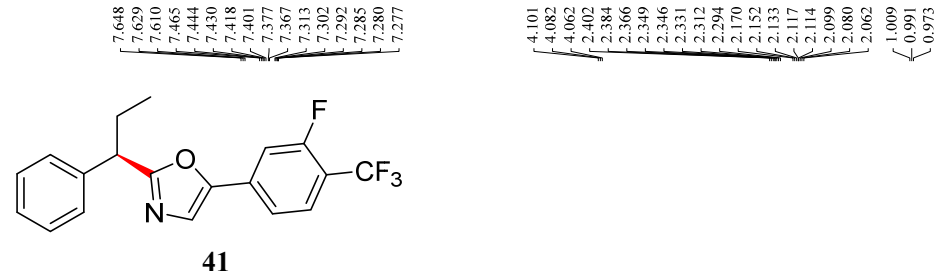

10.47
1.00
0.97
2.08
2.12
6.16
f1 (ppm)


sxl-3-114-1-carbon.1.fid
168.28
139.05
138.96
128.72
128.71
127.83
127.40
127.39
44.98
44.96
27.56
27.52
12.03
N–N
O
f1 (ppm)

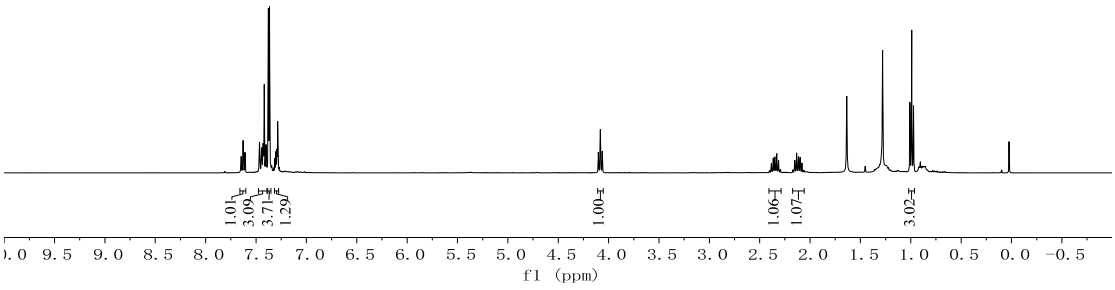
40

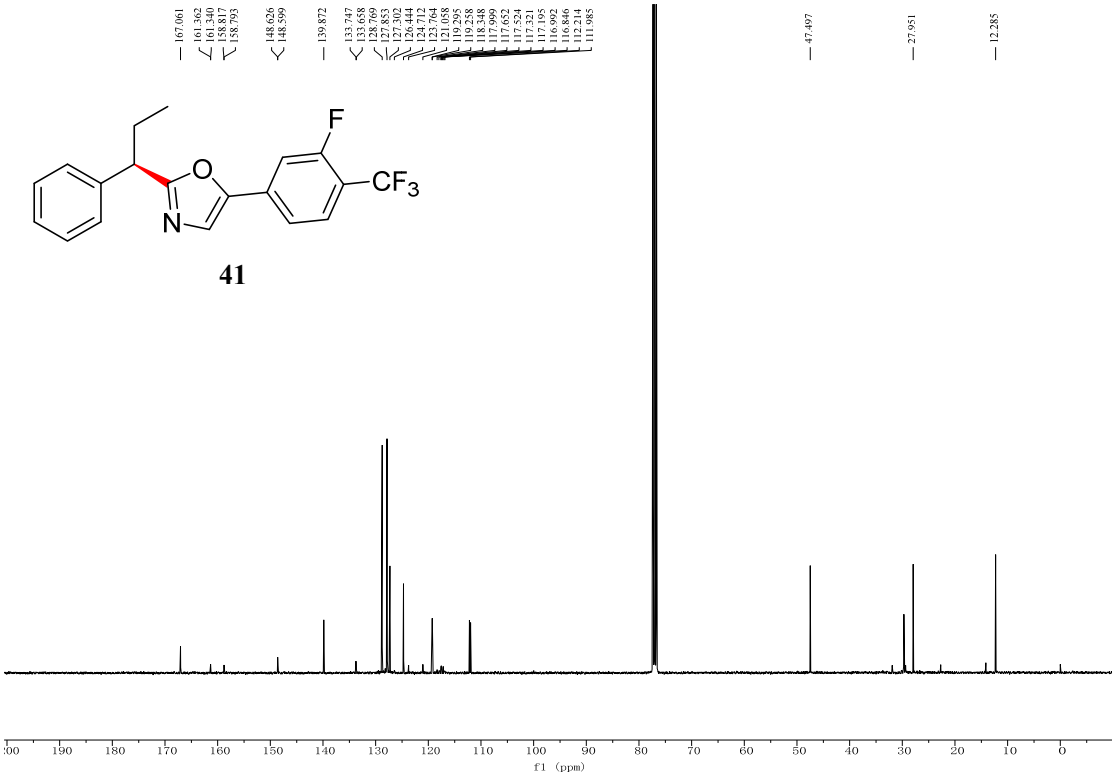

sxl-3-118-6.10.fid
8.31
7.36
7.36
7.35
7.34
7.34
7.34
7.33
7.32
7.32
7.31
7.31
7.30
7.30
7.29
7.29
7.29
7.28
7.28
7.28
7.27
7.26
4.15
4.13
4.11
2.35
2.33
2.31
2.31
2.29
2.28
2.26
2.17
2.15
2.13
2.11
2.11
2.09
0.97
0.95
0.93
N–N
O
40 '
0.95
5.24
1.00
1.10
1.10
3.22
f1 (ppm)


sxl-3-118-6-carbon.10.fid
168.31
153.00
138.58
128.87
127.87
127.66
44.85
27.32
12.07
N–N
O
40 '
f1 (ppm)

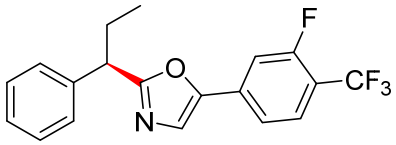

7.648
7.629
7.610
7.465
7.444
7.430
7.418
7.401
7.377
7.367
7.313
7.302
7.292
7.285
7.280
7.277
4.101
4.082
4.062
2.402
2.384
2.366
2.349
2.346
2.331
2.312
2.294
2.170
2.152
2.133
2.117
2.114
2.099
2.080
2.062
1.009
0.991
0.973
F
O
N
CF3

41


1.01
3.09
3.71
1.29
1.00
1.06
1.07
3.02
f1 (ppm)

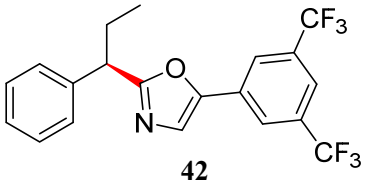

167.061
161.362
161.340
158.817
158.793
148.626
148.599
139.872
133.747
133.658
128.769
127.853
127.302
126.444
125.772
123.764
121.058
119.295
119.258
118.348
117.999
117.652
117.324
117.321
117.195
116.992
116.846
112.214
111.985
47.497
27.951
12.285
F
O
N
CF3
f1 (ppm)

41


F
O
N
CF3

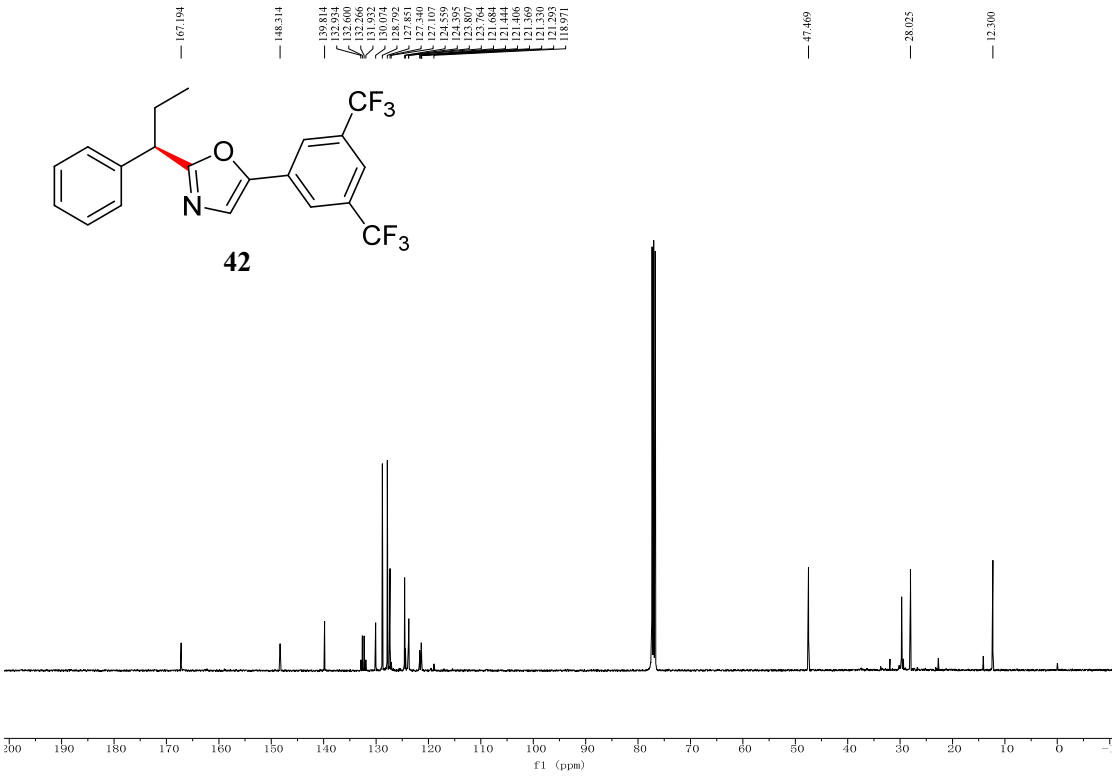
41

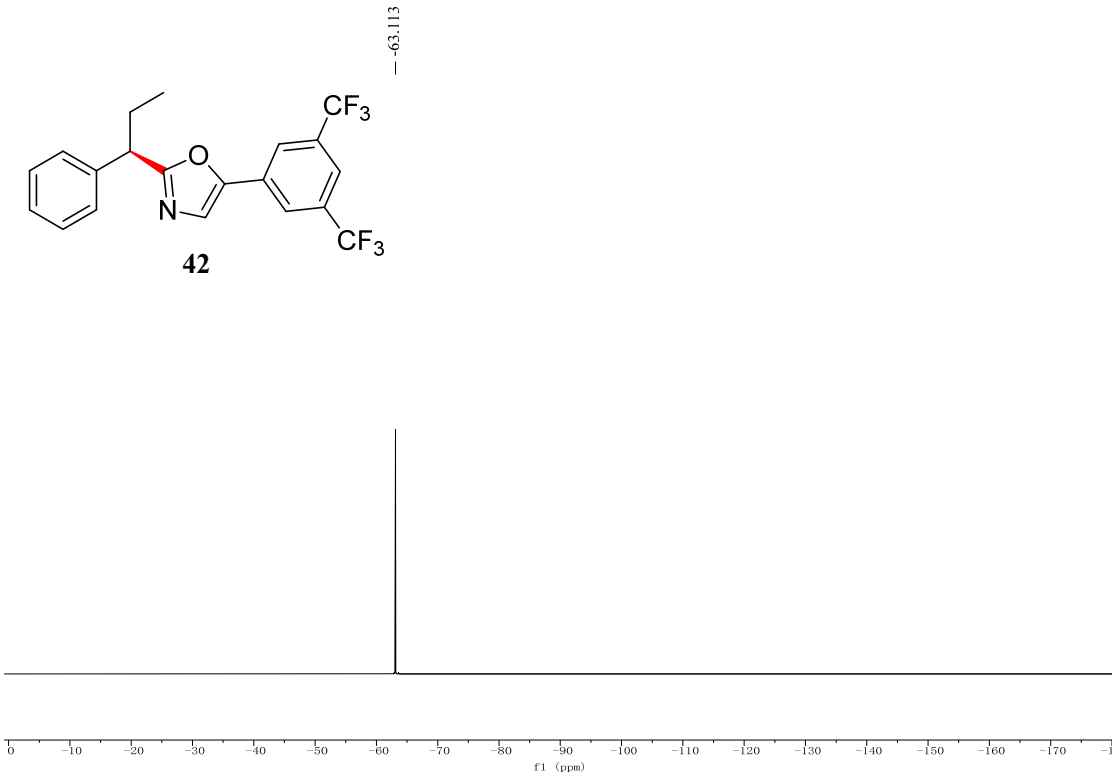
-61.242
-61.275
-113.391
-113.424
-113.456
-113.490
f1 (ppm)

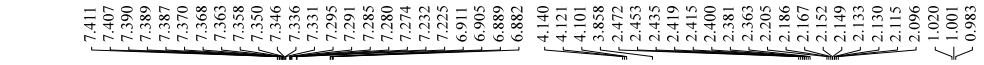

CF3
O
N
CF3

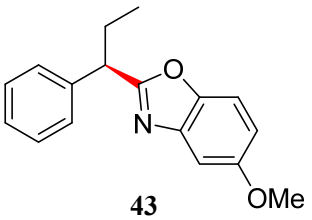
42

8.001
7.998
7.801
7.485
7.388
7.379
7.375
7.318
7.311
7.306
7.303
7.297
7.286
4.125
4.106
4.087
2.418
2.400
2.381
2.365
2.362
2.347
2.328
2.309
2.184
2.166
2.147
2.131
2.128
2.112
2.094
2.076
2.055
1.019
1.001
0.982
2.00
1.01
1.00
1.86
1.94
1.13
1.00
1.05
1.07
3.07
f1 (ppm)

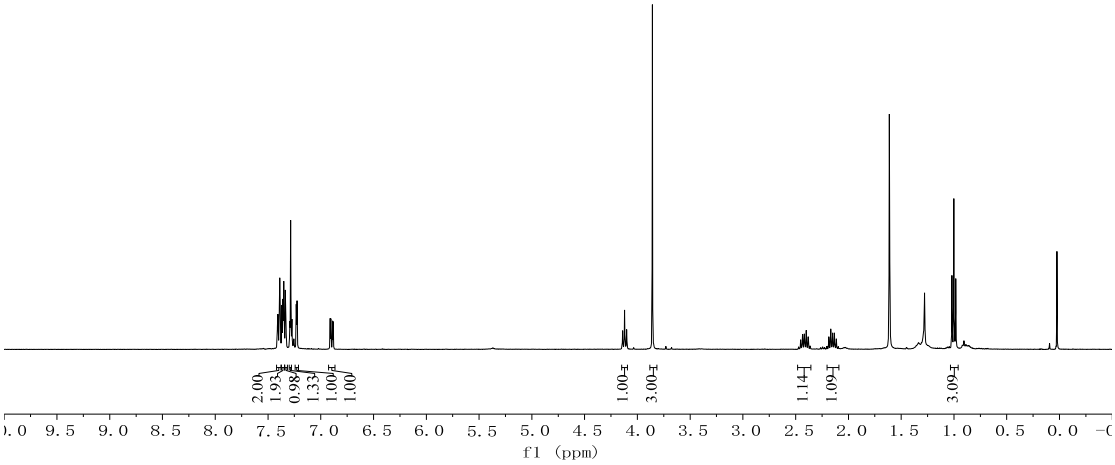

167.194
148.314
139.814
132.934
132.600
132.266
131.932
130.074
128.792
127.951
127.340
127.107
124.559
124.395
123.807
123.764
121.684
121.444
121.406
121.369
121.330
121.293
118.971
47.469
28.025
12.300
CF3
CF3
O
N
42
f1 (ppm)

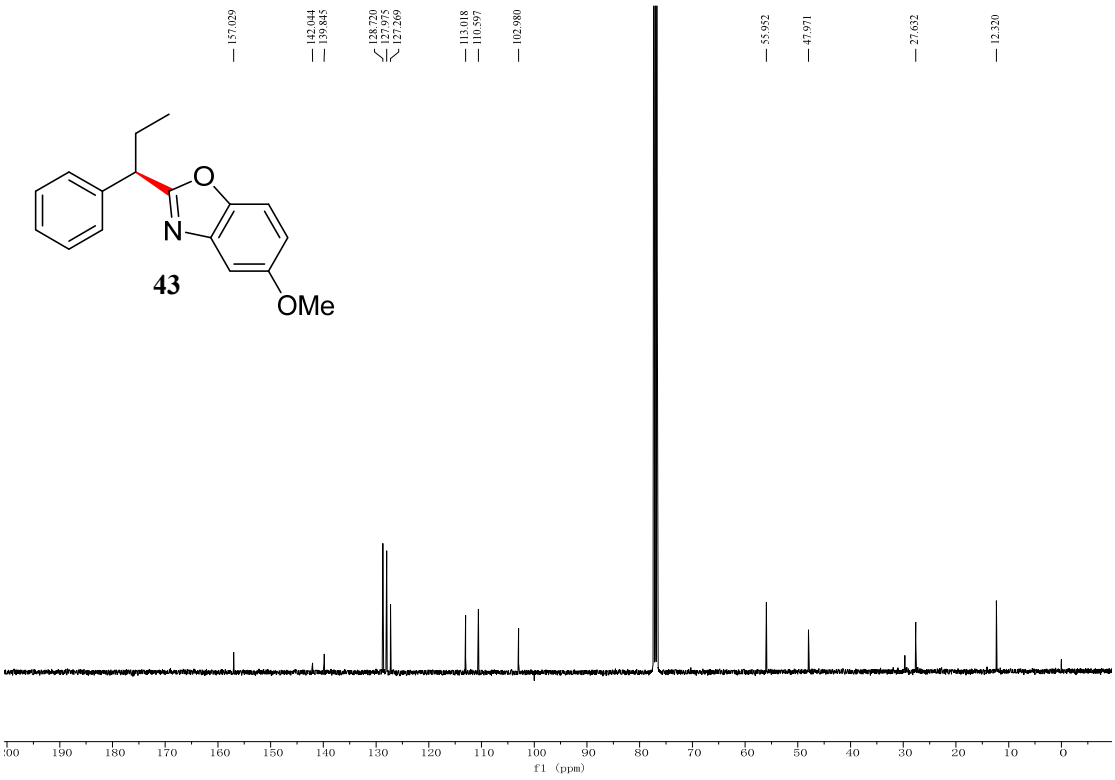

-63.113
CF3
CF3
O
N
42
f1 (ppm)


7.411
7.407
7.390
7.389
7.387
7.370
7.368
7.363
7.358
7.350
7.346
7.336
7.331
7.295
7.291
7.285
7.280
7.274
7.232
7.225
6.911
6.905
6.889
6.882
4.140
4.121
4.101
3.858
2.472
2.453
2.435
2.419
2.415
2.400
2.381
2.363
2.205
2.186
2.167
2.152
2.149
2.133
2.130
2.115
2.096
1.020
1.001
0.983


O
N
OMe

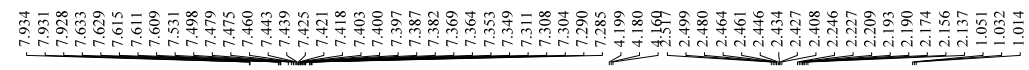
43

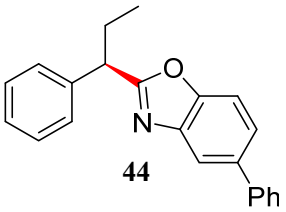

2.00
1.93
0.98
1.33
1.00
1.00
1.00
3.00
1.14
1.09
3.09
f1 (ppm)


157.029
142.044
139.845
128.720
127.975
127.269
113.018
110.597
102.980
55.952
47.971
27.632
12.320
f1 (ppm)

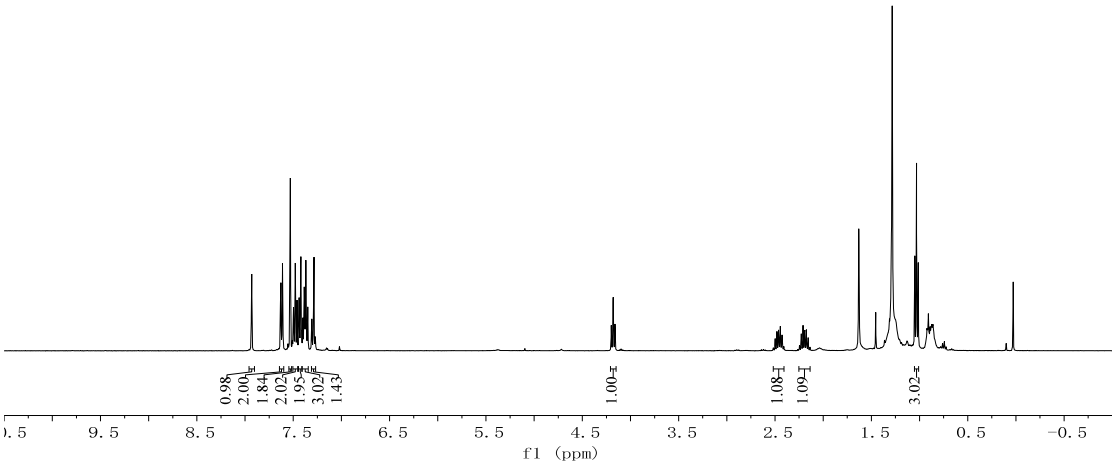
O
N
OMe

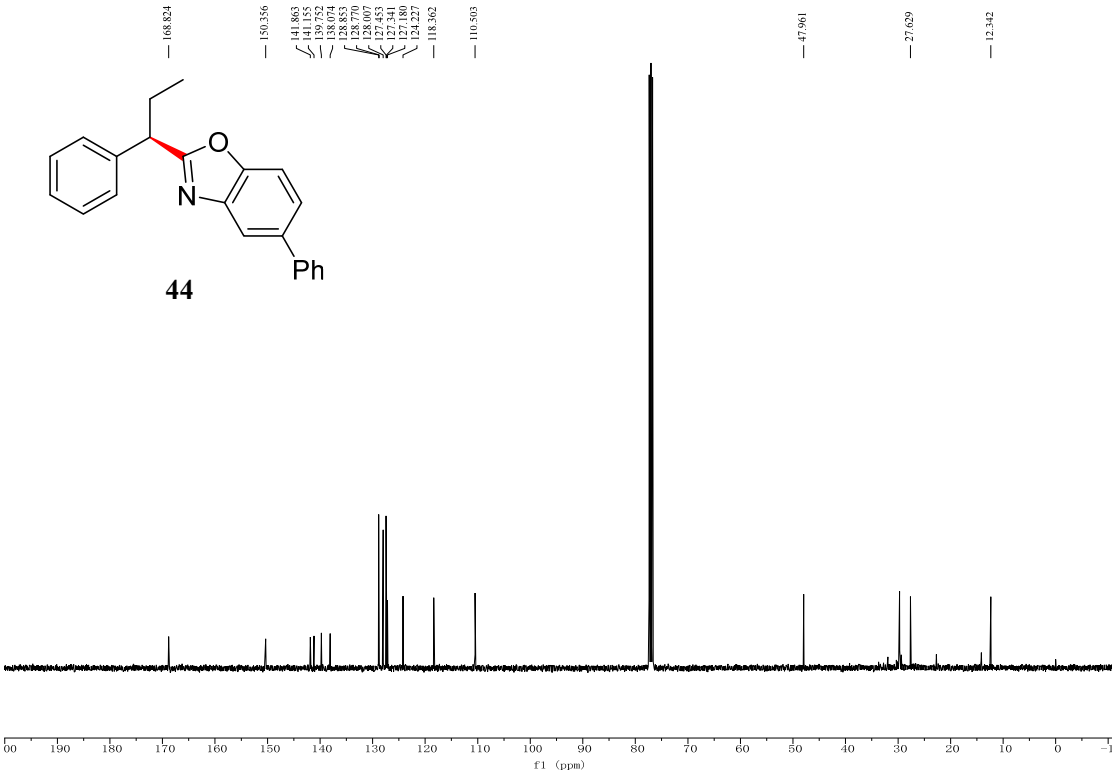
43


7.934
7.931
7.928
7.633
7.629
7.615
7.611
7.609
7.531
7.498
7.479
7.475
7.460
7.443
7.439
7.425
7.421
7.418
7.403
7.400
7.397
7.387
7.382
7.369
7.364
7.353
7.349
7.311
7.308
7.304
7.290
7.285
4.199
4.180
4.160
3.517
2.499
2.480
2.464
2.461
2.446
2.434
2.427
2.408
2.246
2.227
2.209
2.193
2.190
2.174
2.156
2.137
1.051
1.032
1.014


O
N
Ph

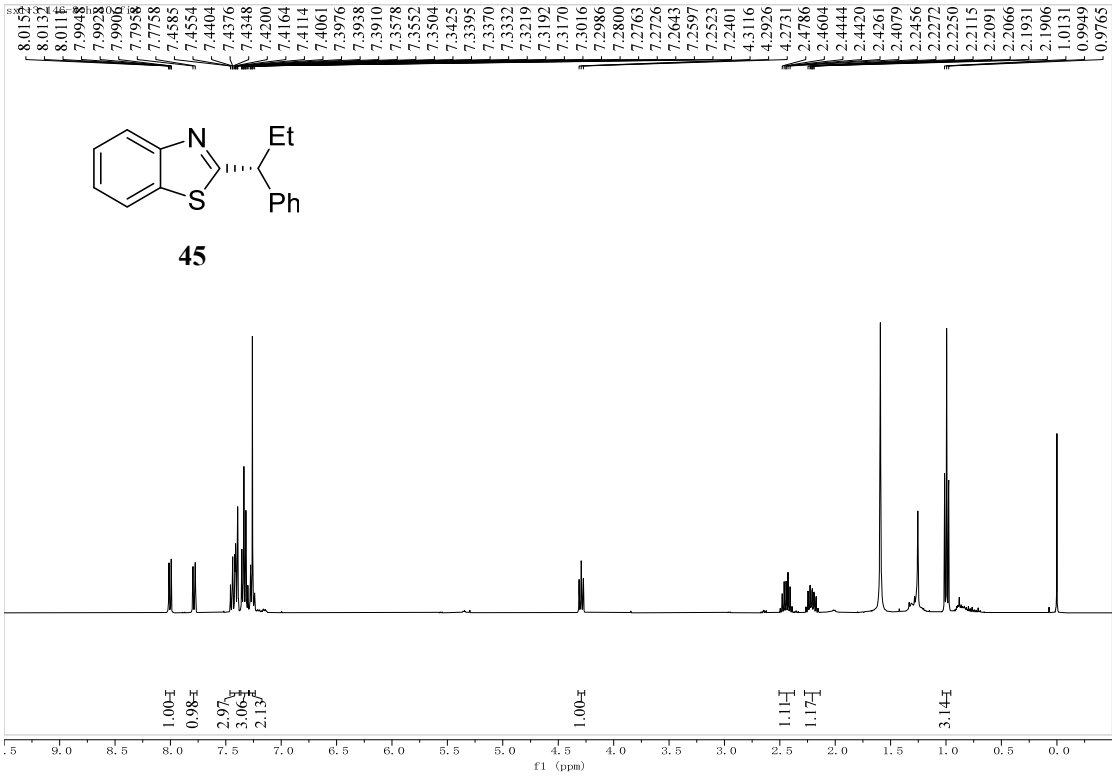
44

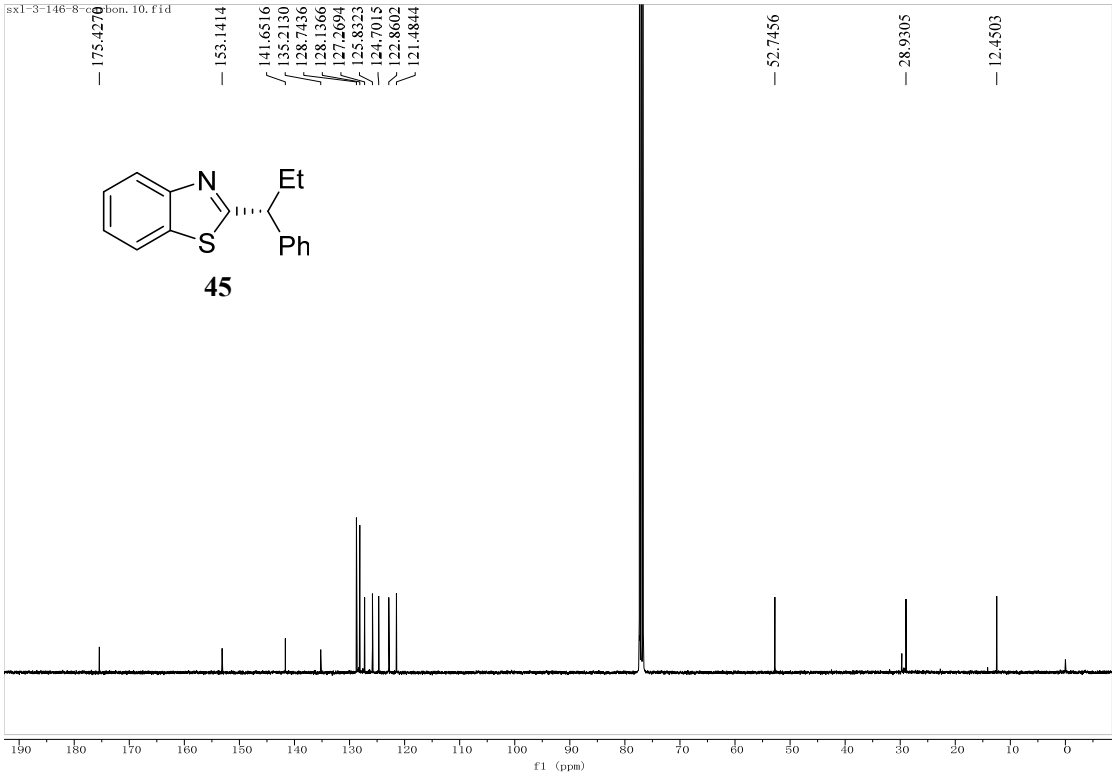

0.98
2.00
1.84
2.02
1.95
3.02
1.43
1.00
1.08
1.09
3.02
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
−0.5
f1 (ppm)

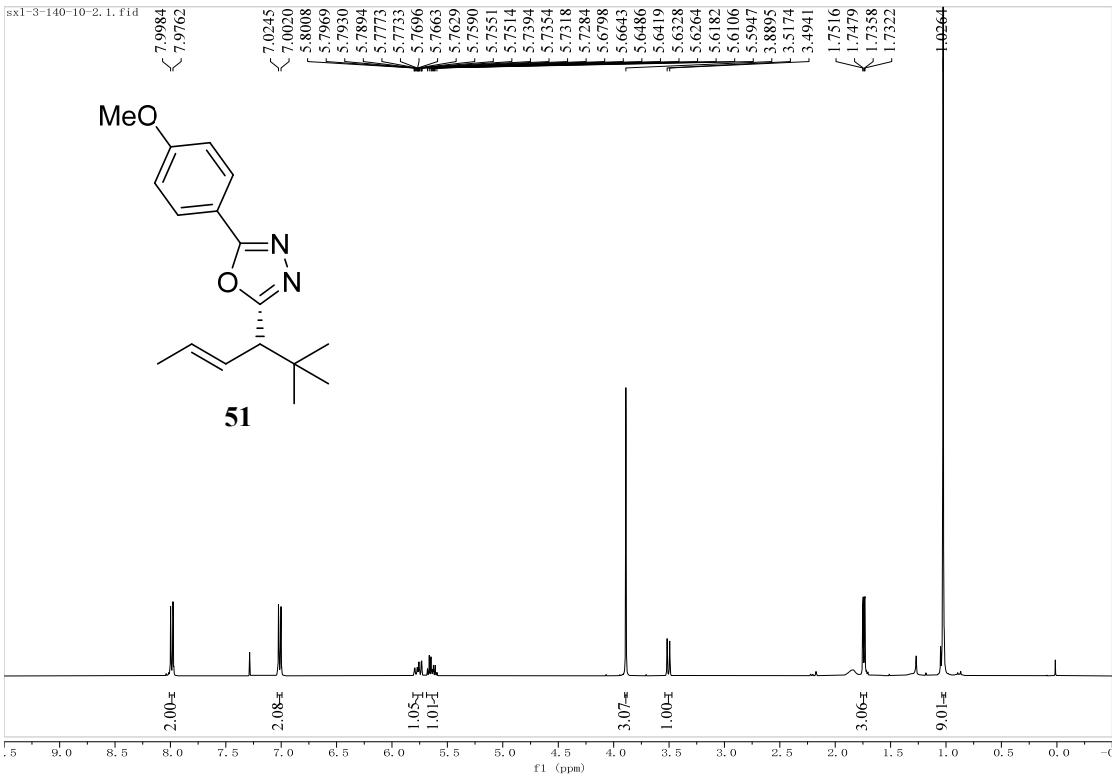

168.824
150.356
141.863
141.155
139.752
138.074
128.853
128.770
128.697
128.455
127.341
127.180
124.227
118.362
110.503
47.961
27.629
12.342
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

O
N
Ph

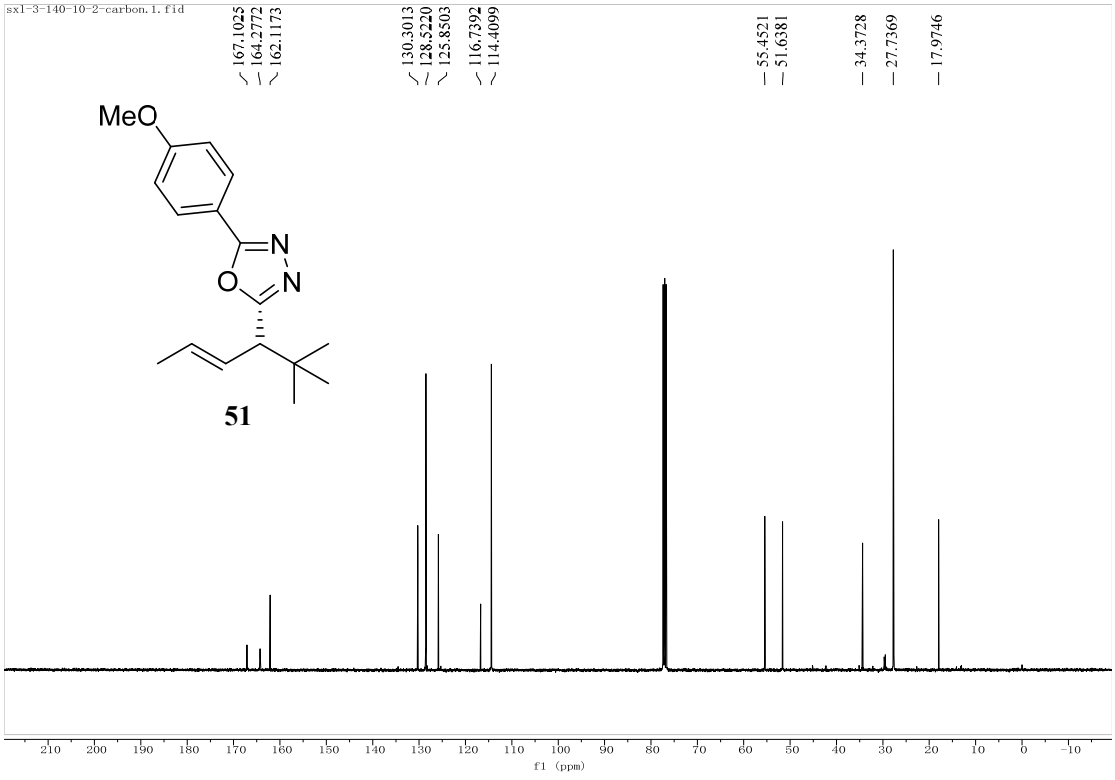
44


8.0153
8.0137
8.0116
7.9948
7.9923
7.9906
7.7958
7.7758
7.4585
7.4554
7.4404
7.4376
7.4348
7.4200
7.4164
7.4114
7.4061
7.3976
7.3938
7.3910
7.3578
7.3552
7.3504
7.3425
7.3395
7.3370
7.3332
7.3219
7.3192
7.3170
7.3016
7.2986
7.2800
7.2763
7.2726
7.2643
7.2597
7.2523
7.2401
4.3116
4.2926
4.2731
2.4786
2.4604
2.4444
2.4420
2.4261
2.4079
2.2456
2.2272
2.2250
2.2115
2.2091
2.2066
2.1931
2.1906
1.0131
0.9949
0.9765
N
Et
S
Ph
45
1.00
0.98
2.97
3.06
2.13
1.00
1.11
1.17
3.14
f1 (ppm)

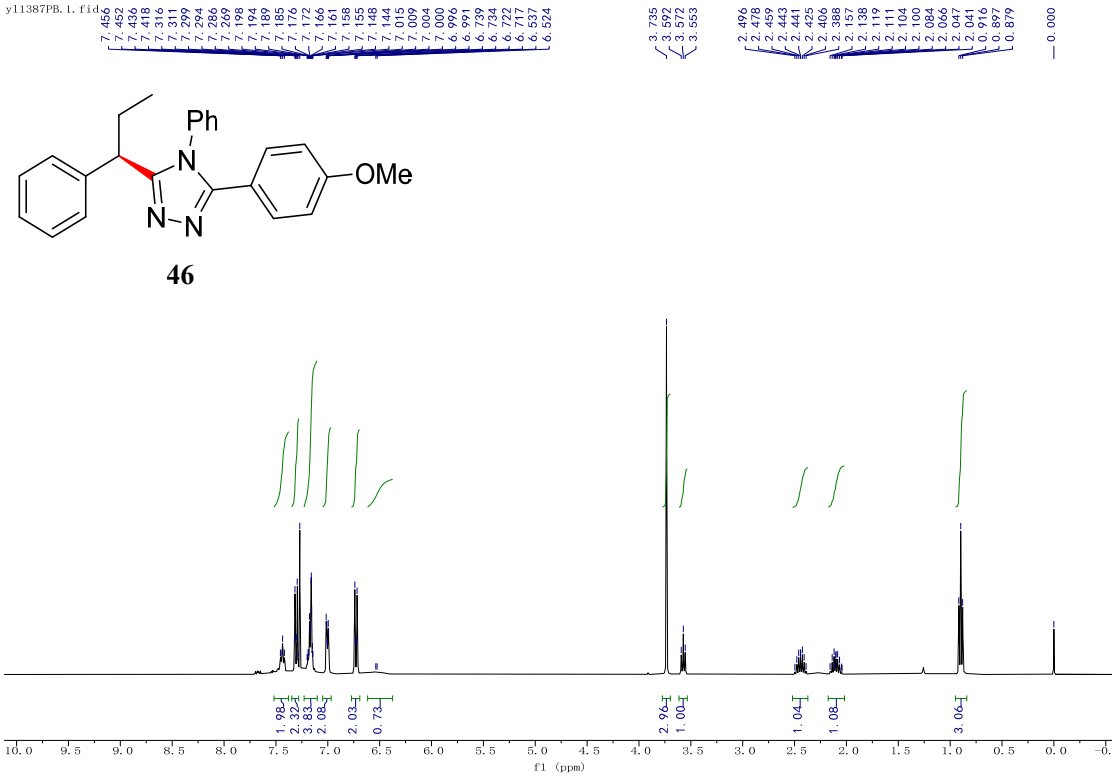

175.4270
153.1414
141.6516
135.2130
128.7436
128.1366
127.2694
125.8323
124.7015
122.8602
121.4844
52.7456
28.9305
12.4503
N
Et
S
Ph
45
f1 (ppm)


sx1-3-140-10-2.1.fid
7.9984
7.9762
7.0245
7.0020
5.8008
5.7969
5.7930
5.7894
5.7773
5.7733
5.7696
5.7663
5.7629
5.7590
5.7551
5.7514
5.7394
5.7354
5.7318
5.7284
5.6798
5.6643
5.6486
5.6419
5.6328
5.6264
5.6182
5.6106
5.5947
3.8895
3.5174
3.4941
1.7516
1.7479
1.7358
1.7322
1.0264
MeO
N
O
N
2.00
2.08
1.05
1.01
3.07
1.00
3.06
9.01
f1 (ppm)

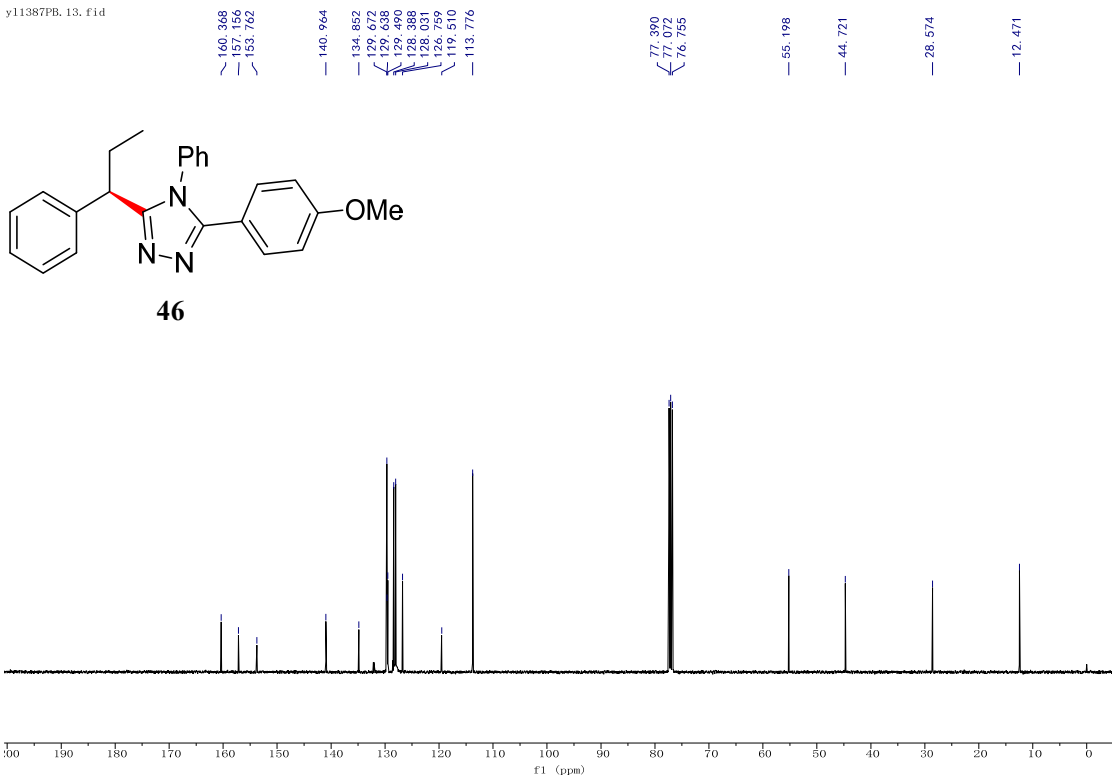
51


sx1-3-140-10-2-carbon.1.fid
167.1025
164.2772
162.1173
130.3013
128.5220
125.8503
116.7392
114.4099
55.4521
51.6381
34.3728
27.7369
17.9746
MeO
N
O
N
f1 (ppm)

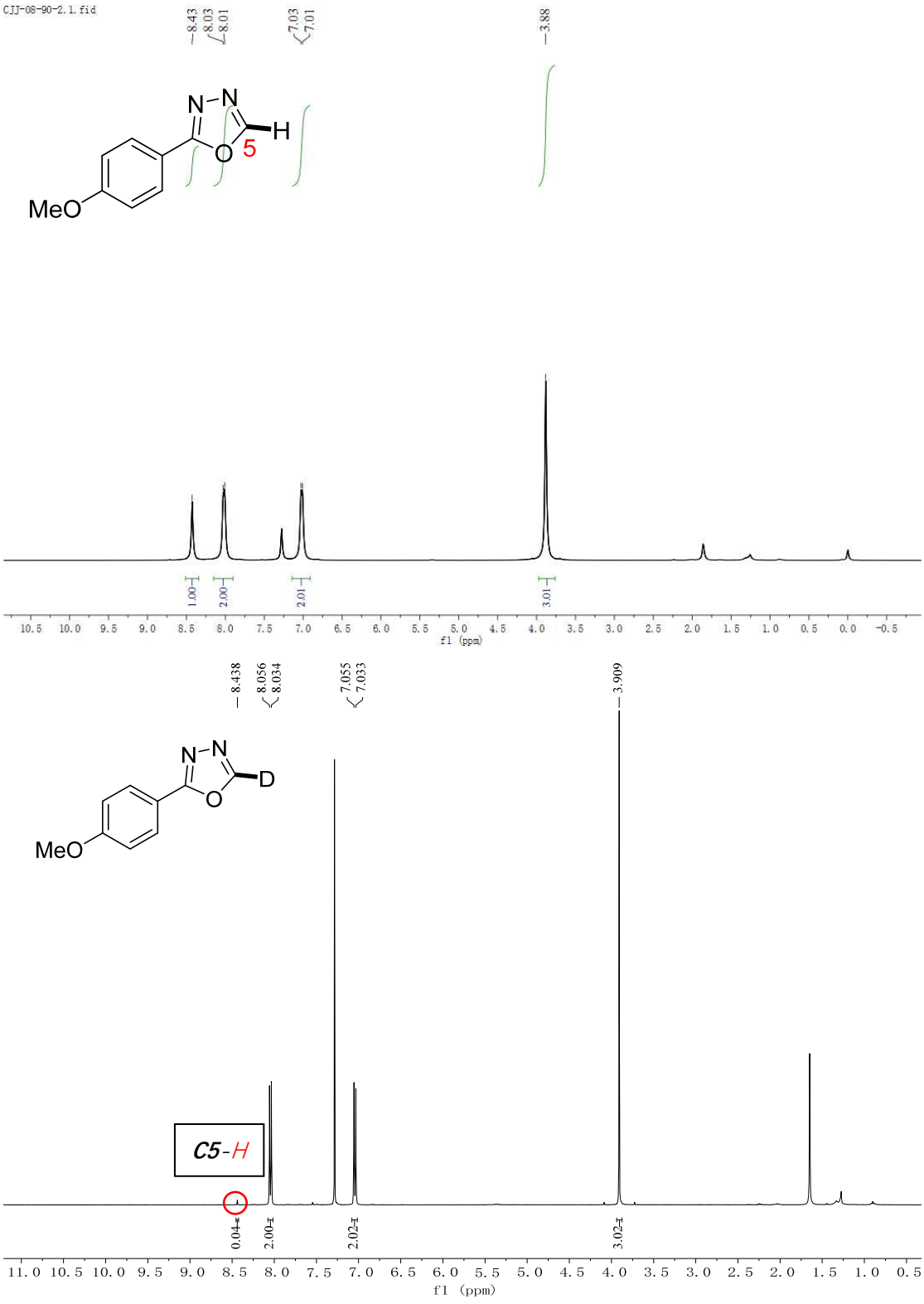
51

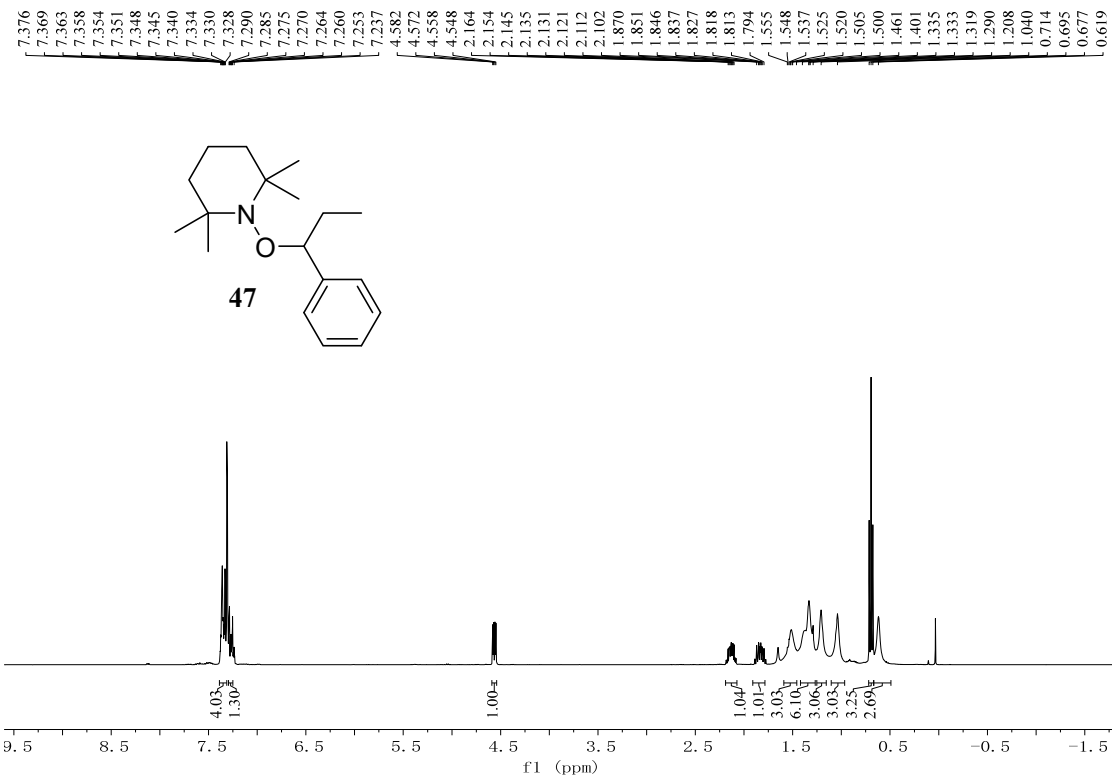

y11387PB.1.fid
7.456
7.452
7.436
7.418
7.316
7.311
7.299
7.294
7.286
7.269
7.198
7.194
7.189
7.185
7.176
7.172
7.166
7.161
7.158
7.155
7.148
7.144
7.015
7.009
7.004
7.000
6.996
6.991
6.739
6.734
6.722
6.717
6.537
6.524
3.735
3.592
3.572
3.553
2.496
2.478
2.459
2.443
2.441
2.425
2.406
2.388
2.157
2.138
2.119
2.111
2.104
2.100
2.084
2.066
2.047
2.041
0.916
0.897
0.879
0.000
Ph
N
N
N
OMe
1.98
2.32
3.83
2.08
2.03
0.73
2.96
1.00
1.04
1.08
3.06
f1 (ppm)

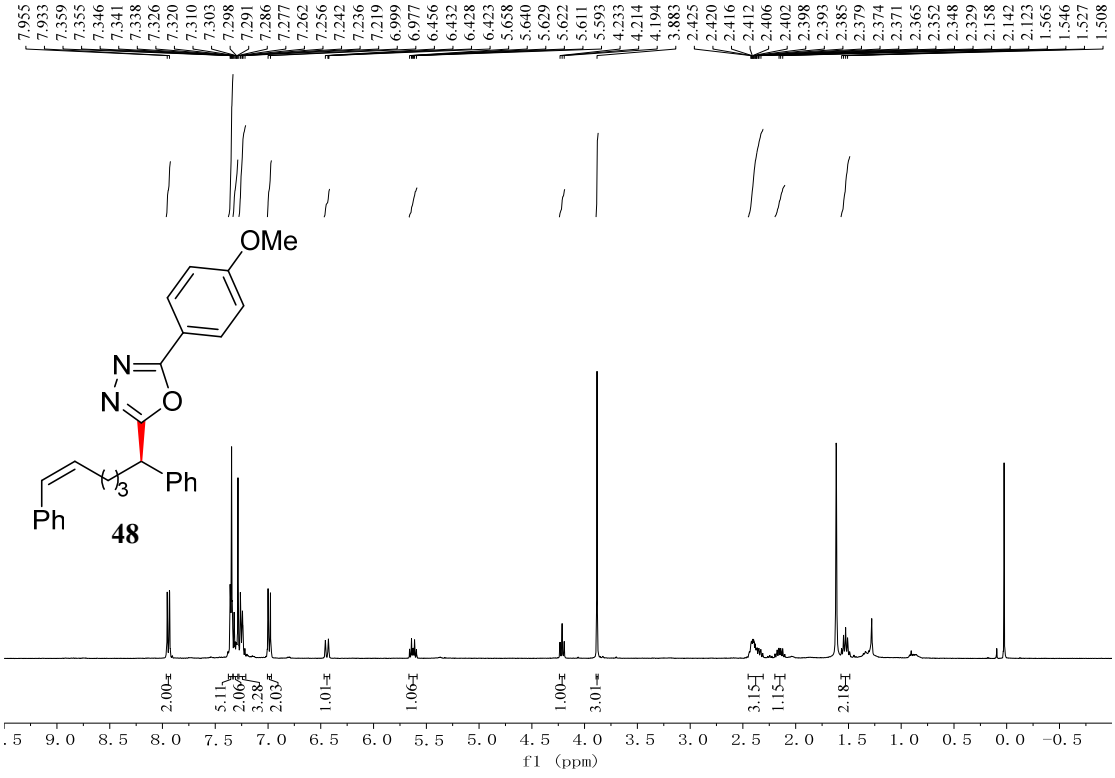
46

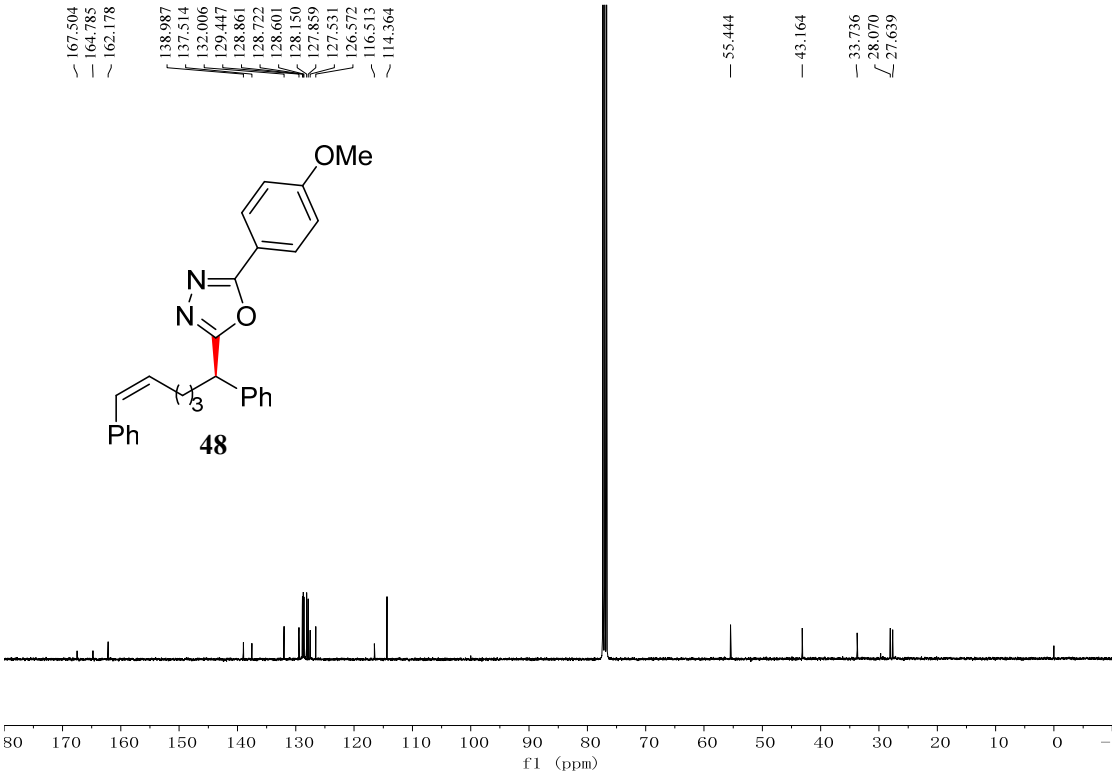

y11387PB.13.fid
160.368
157.156
153.762
140.964
134.852
129.672
129.638
129.490
128.388
128.031
126.759
119.510
113.776
77.390
77.072
76.755
55.198
44.721
28.574
12.471
Ph
N
N
N
OMe
f1 (ppm)

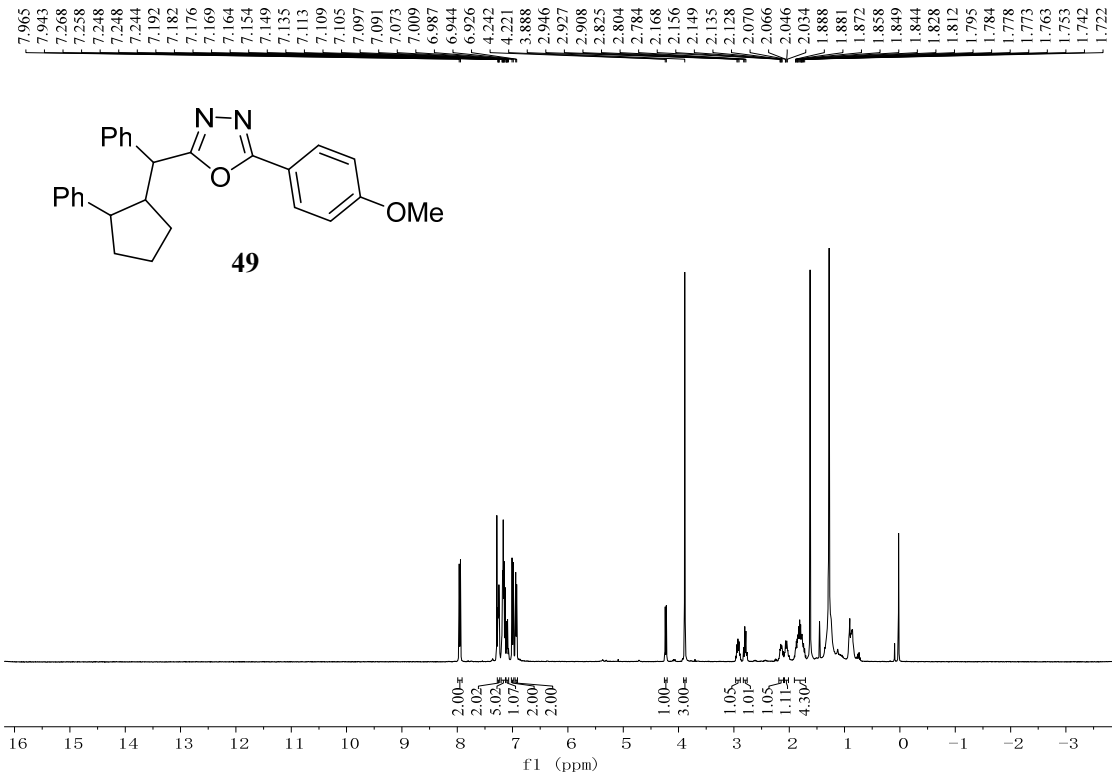
46

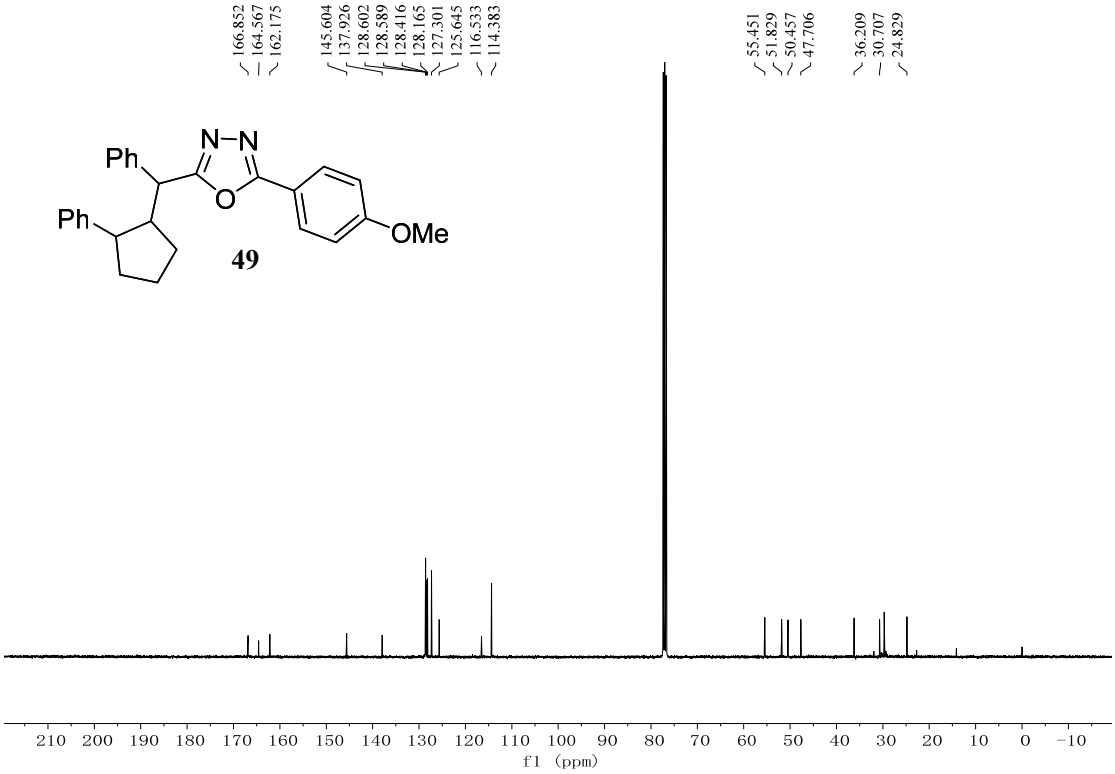

CJJ-08-90-2.1.fid
8.43
8.03
8.01
7.03
7.01
3.88
N
N
H
O
5
MeO
1.00
2.00
2.01
3.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
8.438
8.056
8.034
7.055
7.033
3.909
N
N
D
O
MeO
C5-H
0.04
2.00
2.02
3.02
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

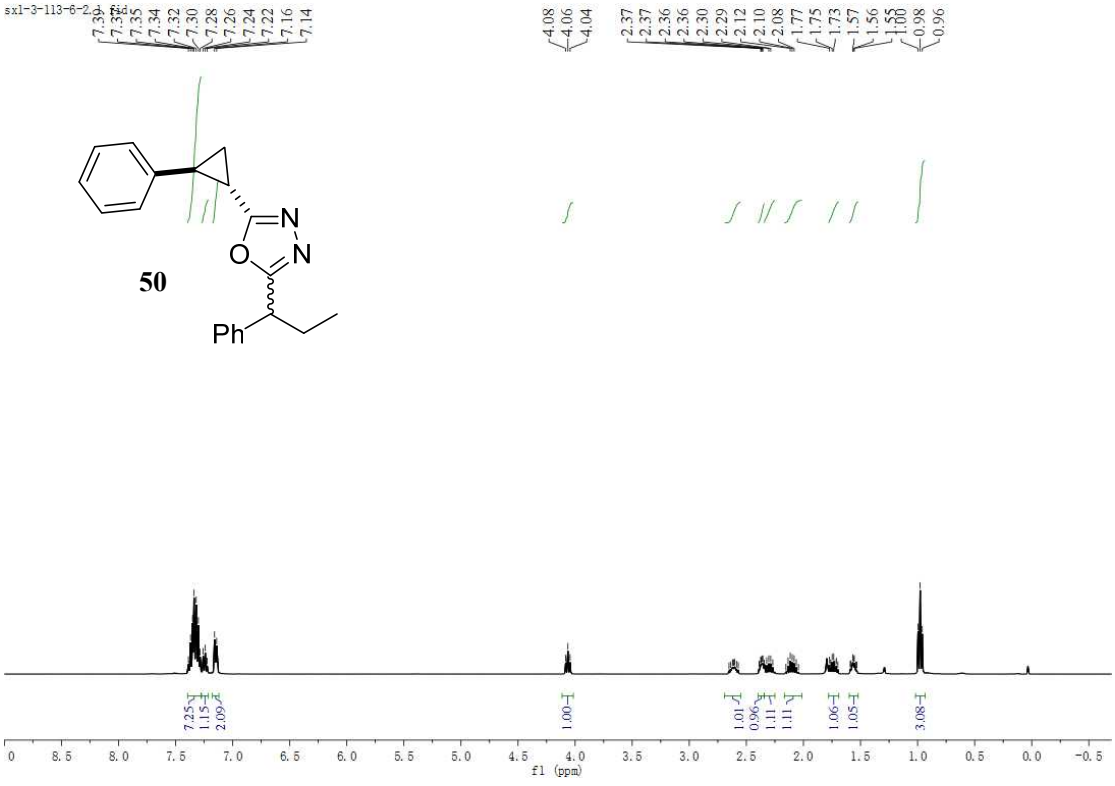

7.376
7.369
7.363
7.358
7.354
7.351
7.348
7.345
7.340
7.334
7.330
7.328
7.290
7.285
7.275
7.270
7.264
7.260
7.253
7.237
4.582
4.572
4.558
4.548
2.164
2.154
2.145
2.135
2.131
2.121
2.112
2.102
1.870
1.851
1.846
1.837
1.827
1.818
1.813
1.794
1.555
1.548
1.537
1.525
1.520
1.505
1.500
1.461
1.401
1.335
1.333
1.319
1.290
1.208
1.040
0.714
0.695
0.677
0.619
N
O
47
4.03
1.30
1.00
1.04
1.01
3.03
6.10
3.06
3.03
3.25
2.69
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
−0.5
−1.5
f1 (ppm)

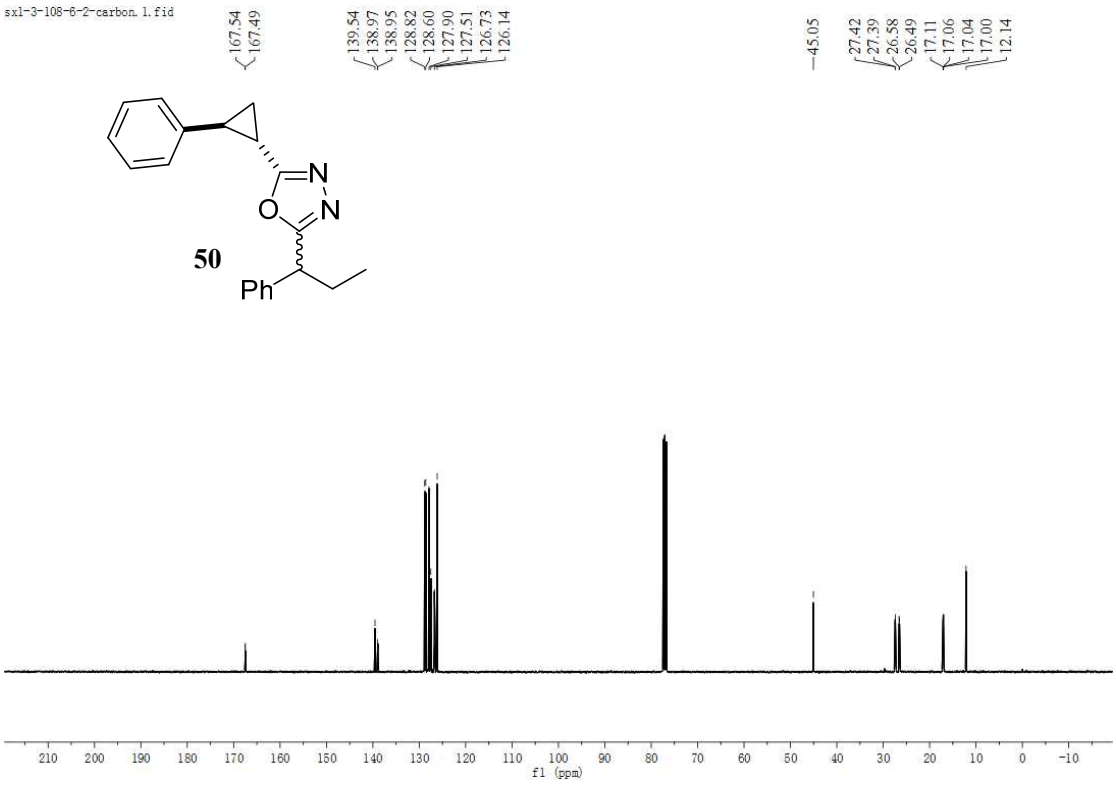

7.955
7.933
7.359
7.355
7.346
7.341
7.338
7.326
7.320
7.310
7.303
7.298
7.291
7.286
7.277
7.262
7.256
7.242
7.236
7.219
6.999
6.977
6.456
6.432
6.428
6.423
5.658
5.640
5.629
5.622
5.611
5.593
4.233
4.214
4.194
3.883
2.425
2.420
2.416
2.412
2.406
2.402
2.398
2.393
2.385
2.379
2.374
2.371
2.365
2.352
2.348
2.329
2.158
2.142
2.123
1.565
1.546
1.527
1.508
OMe
N
N
O
Ph
Ph
48
2.00
5.11
2.06
3.28
2.03
1.01
1.06
1.00
3.01
3.15
1.15
2.18
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)


167.504
164.785
162.178
138.987
137.514
132.006
129.447
128.861
128.722
128.601
128.150
127.859
127.531
126.572
116.513
114.364
55.444
43.164
33.736
28.070
27.639
OMe
N
N
O
Ph
Ph
3
48
f1 (ppm)

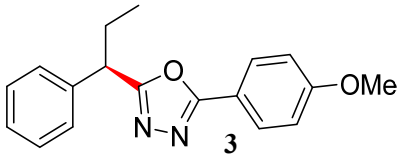

7.965
7.943
7.268
7.258
7.248
7.248
7.244
7.192
7.182
7.176
7.169
7.164
7.154
7.149
7.135
7.113
7.109
7.105
7.097
7.091
7.073
7.009
6.987
6.944
6.926
4.242
4.221
3.888
2.946
2.927
2.908
2.825
2.804
2.784
2.168
2.156
2.149
2.135
2.128
2.070
2.066
2.046
2.034
1.888
1.881
1.872
1.858
1.849
1.844
1.828
1.812
1.795
1.784
1.778
1.773
1.763
1.753
1.742
1.722
N–N
Ph
Ph
O
OMe
49
2.00
2.02
5.02
1.07
2.00
2.00
1.00
3.00
1.05
1.01
1.05
1.11
4.30
f1 (ppm)


166.852
164.567
162.175
145.604
137.926
128.602
128.589
128.416
128.165
127.301
125.645
116.533
114.383
55.451
51.829
50.457
47.706
36.209
30.707
24.829
Ph
Ph
N–N
O
OMe
49
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


sxl-3-113-6-2.1.fid
7.39
7.37
7.35
7.34
7.32
7.30
7.28
7.26
7.24
7.22
7.16
7.14
4.08
4.06
4.04
2.37
2.37
2.36
2.36
2.30
2.29
2.12
2.10
2.08
1.77
1.75
1.73
1.57
1.56
1.55
1.00
0.98
0.96
N
O
N
Ph
50
7.25
1.15
2.09
1.00
1.01
0.96
1.11
1.11
1.06
1.05
3.08
0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)

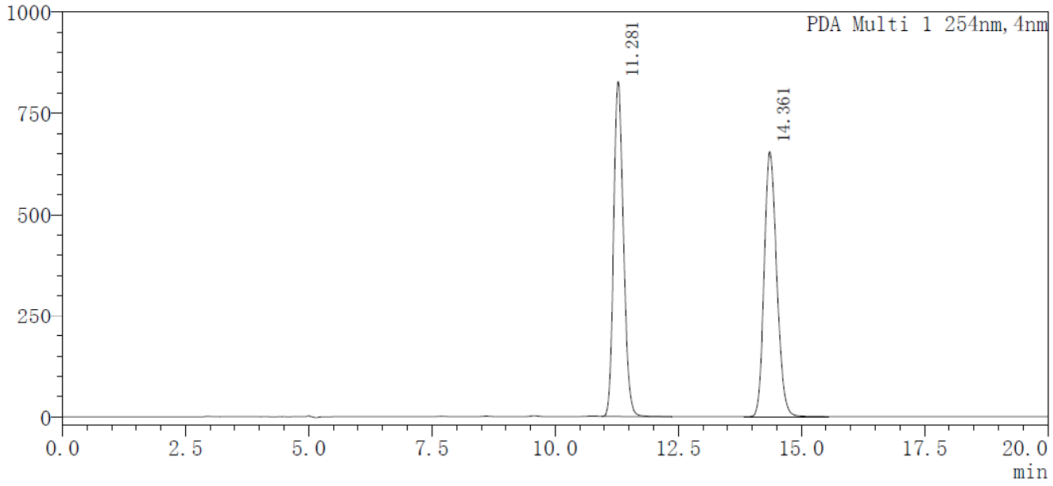

sx1-3-108-6-2-carbon.1.fid
167.54
167.49
139.54
138.97
138.95
128.82
128.60
127.90
127.51
126.73
126.14
45.05
27.42
27.39
26.58
26.49
17.11
17.06
17.04
17.00
12.14
N
O
N
Ph
50
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

HPLC spectra



3

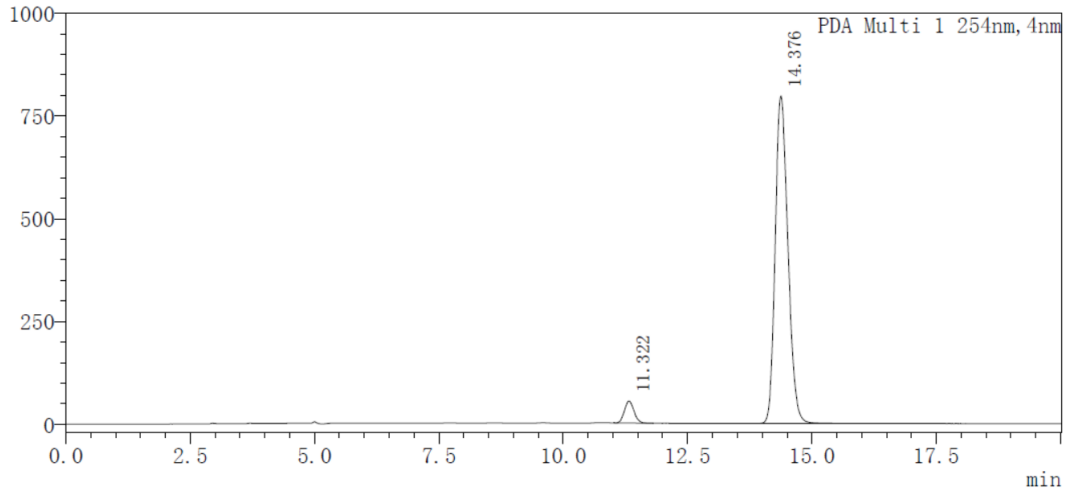
HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 11.3 min, t(major) = 14.4 min, 90% ee (95.2:4.8 er).



Peak Table

PDA Ch1 254nm

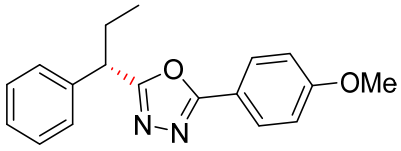
Peak#	Ret. Time	Area	Area%
1	11.281	11483401	49.727
2	14.361	11609339	50.273



Peak Table

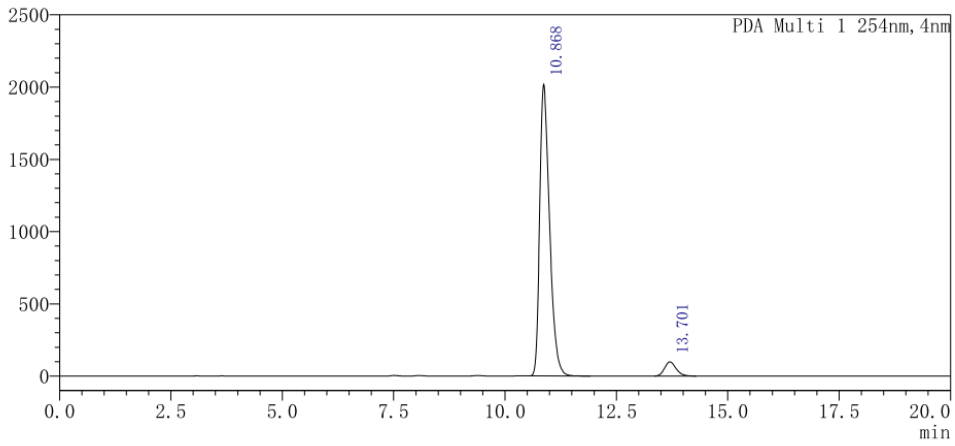
PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	11.322	723400	4.840
2	14.376	14221390	95.160



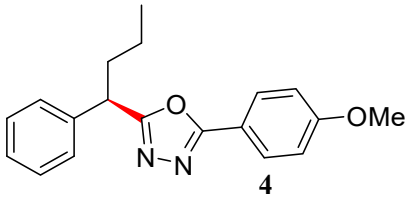
***ent*-3**

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 82/20, flow rate 1.0 mL/min. λ = 254 nm, t(major) = 10.9 min, t(minor) = 13.7 min, 89% ee (94.6:5.4 er).



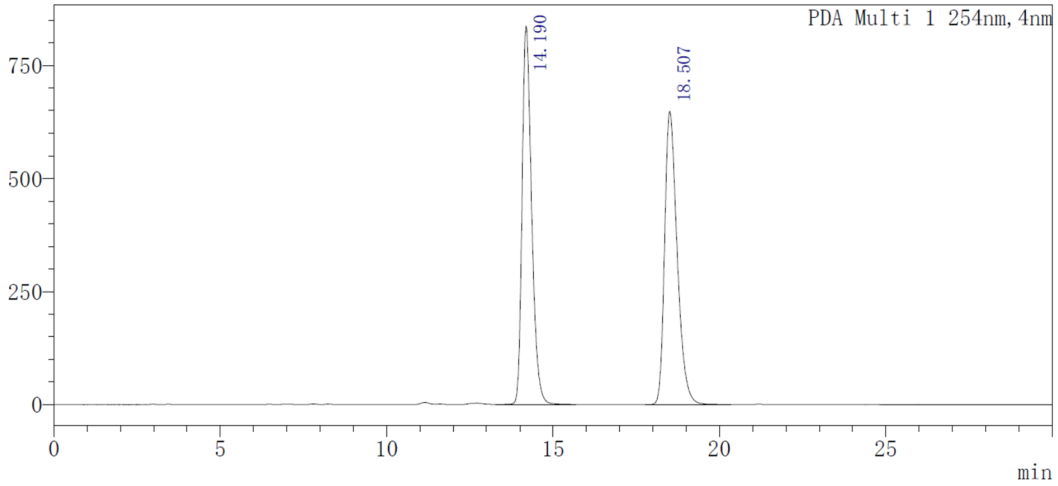
PDA Ch1 254nm

T	Hight	Area	Area%
10.868	2052944	31468200	94.621
13.701	99003	1788880	5.379



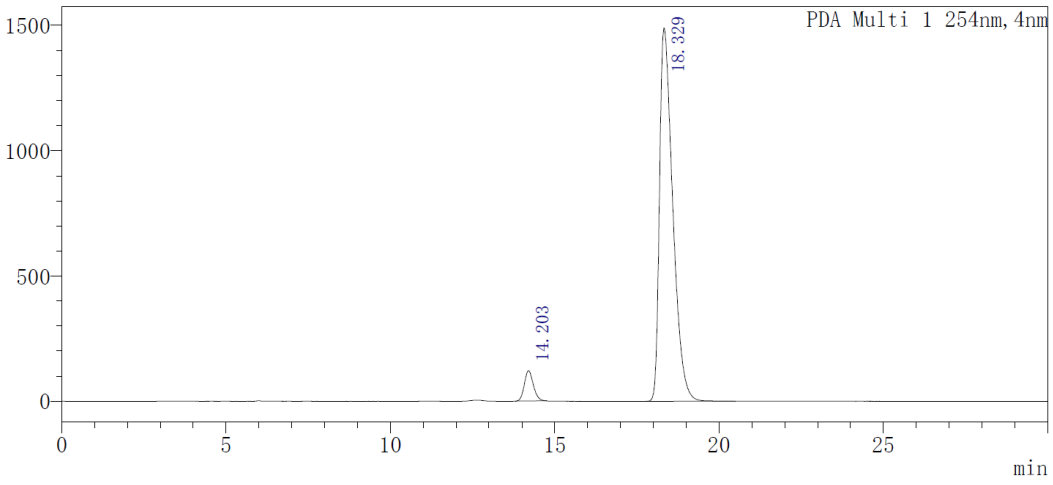
4

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 14.2 min, t(major) = 18.3 min, 90% ee (94.8:5.2 er).



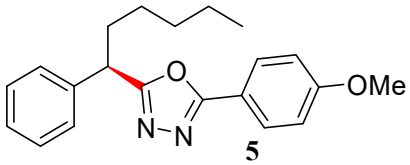
PDA Ch1 254nm

T	Hight	Area	Area%
14.190	836292	16698464	49.590
18.507	647991	16974729	50.410



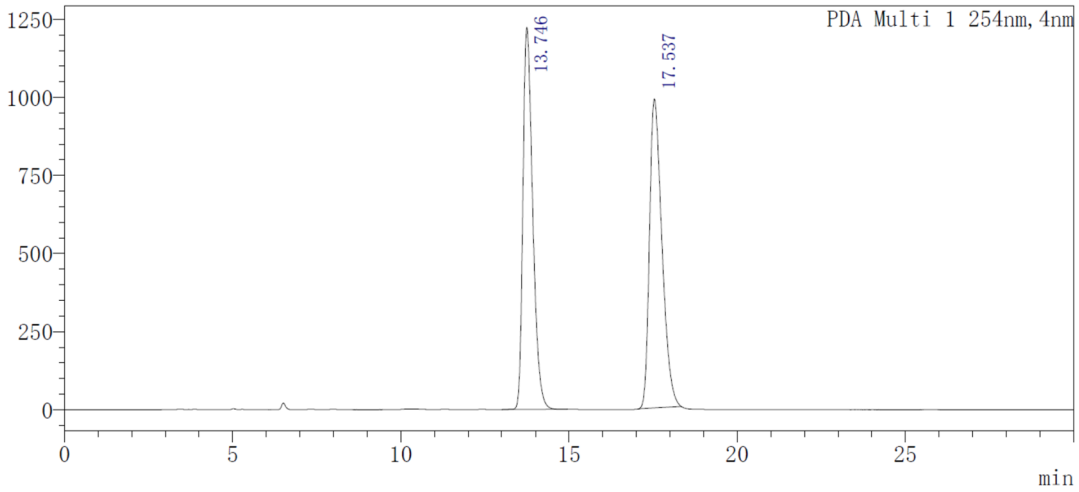
PDA Ch1 254nm

T	Hight	Area	Area%
14.203	120496	2302653	5.227
18.329	1488823	41747084	94.773



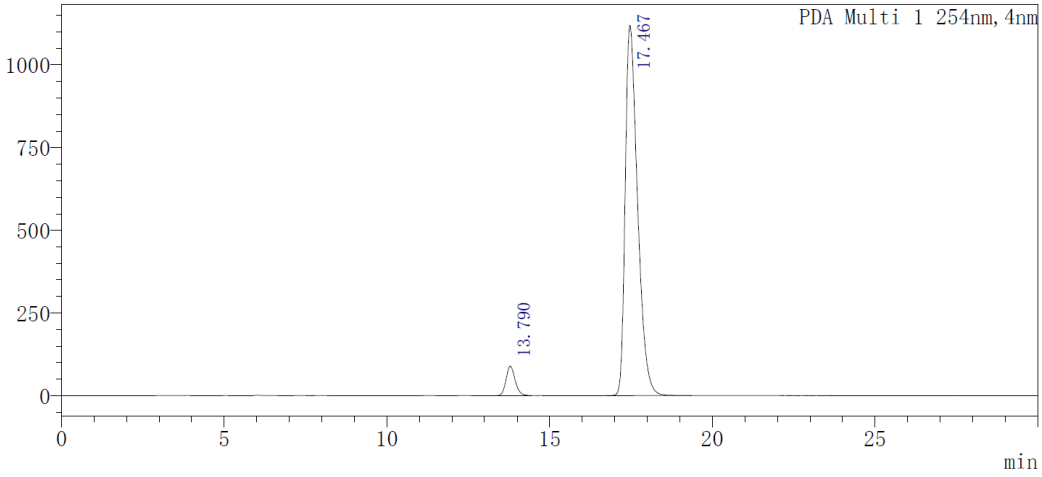
5

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 13.8 min, t(major) = 17.5 min, 89% ee (94.5:5.5 er).



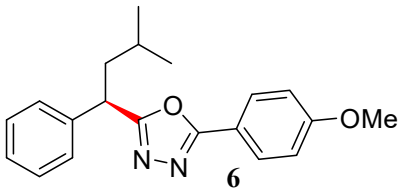
PDA Ch1 254nm

T	Hight	Area	Area%
13.746	1222649	24764164	49.291
17.537	988590	25476168	50.709



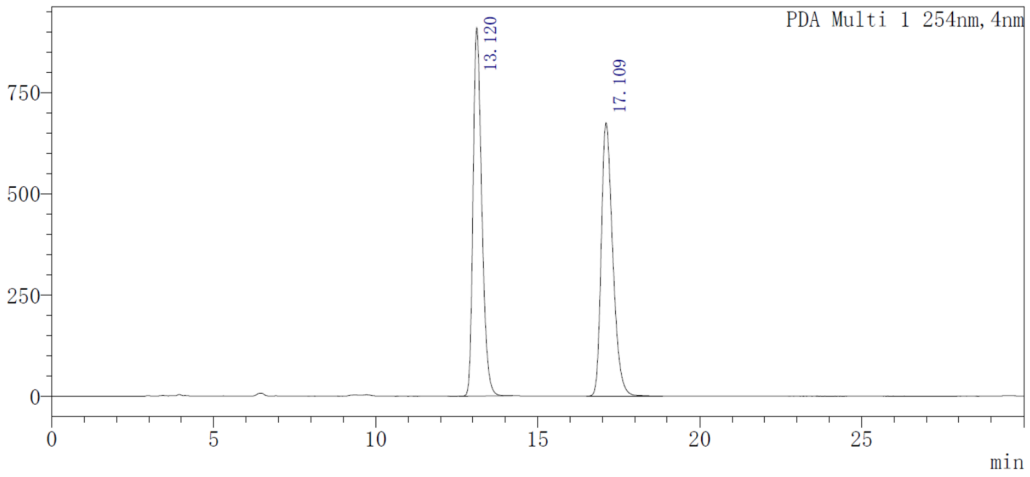
PDA Ch1 254nm

T	Hight	Area	Area%
13.790	89086	1719017	5.505
17.467	1119786	29505767	94.495



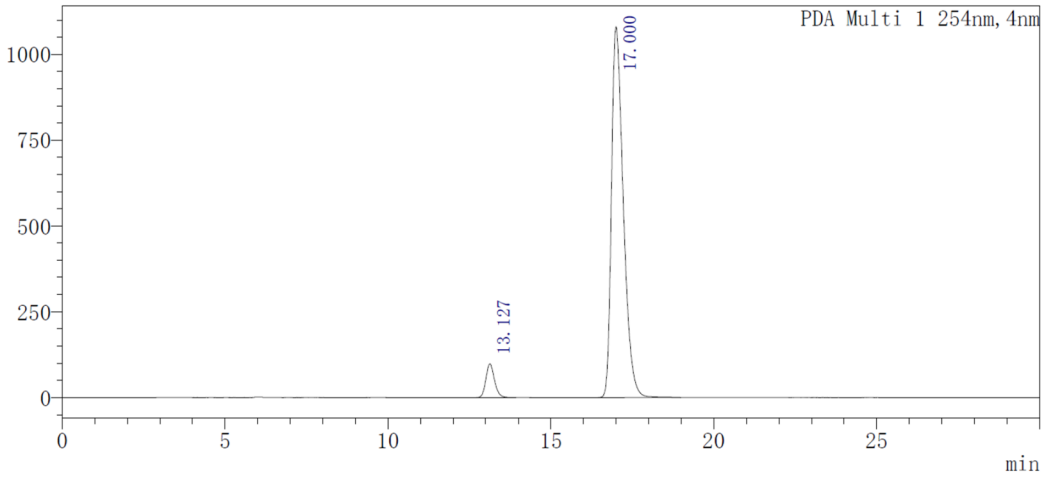
6

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 13.1 min, t(major) = 17.0 min, 88% ee (93.8:6.2 er).



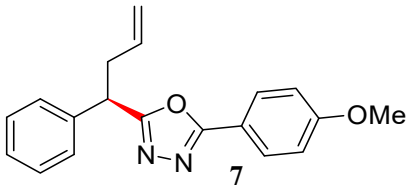
PDA Ch1 254nm

T	Hight	Area	Area%
13.120	909677	17018153	50.528
17.109	675539	16662490	49.472



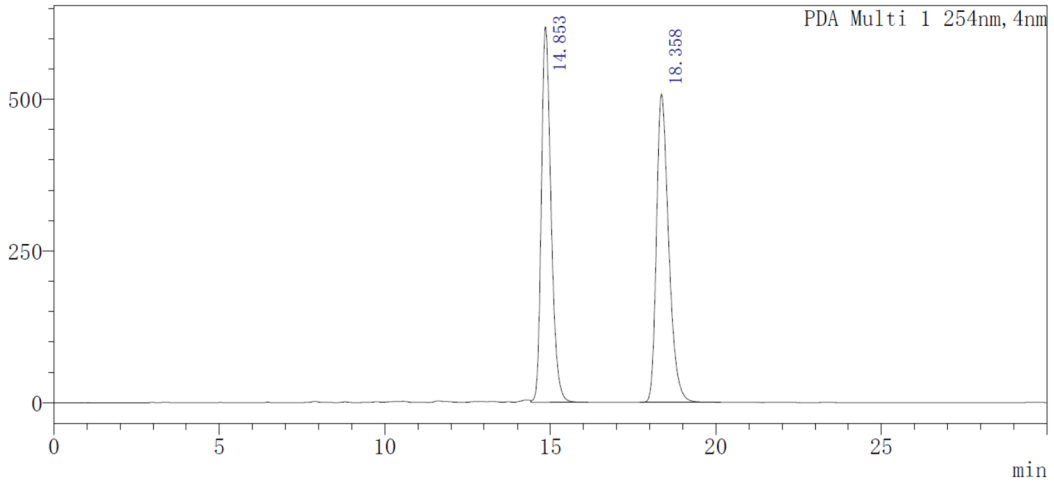
PDA Ch1 254nm

T	Hight	Area	Area%
13.127	98269	1808771	6.205
17.000	1079859	27341414	93.795



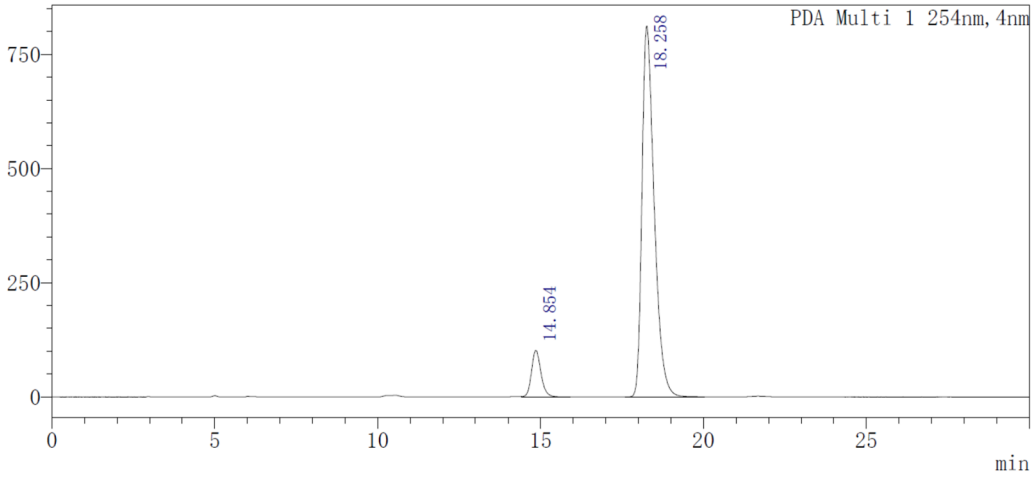
7

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 14.9 min, t(major) = 18.3 min, 82% ee (91.2:8.8 er).



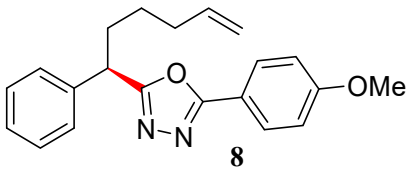
PDA Ch1 254nm

T	Hight	Area	Area%
14.853	618454	12606077	49.058
18.358	507816	13090277	50.942



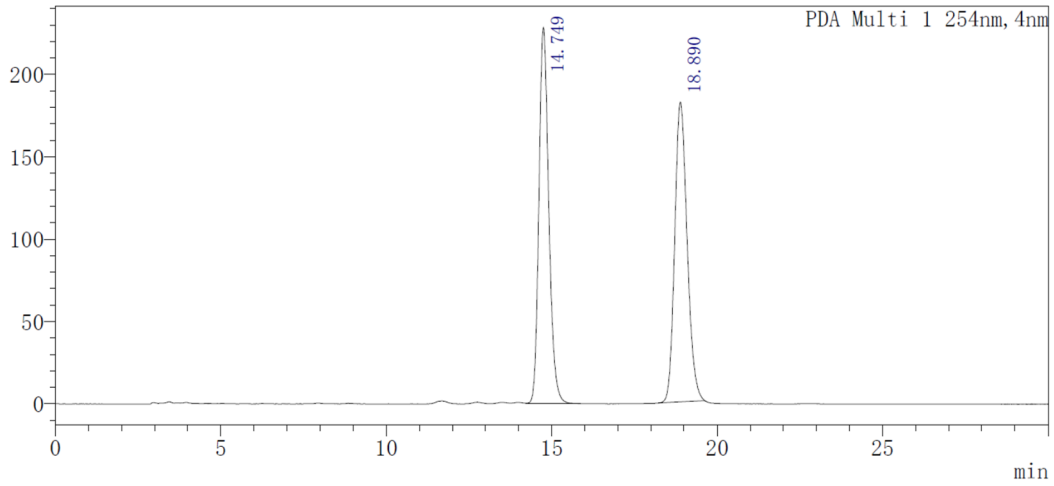
PDA Ch1 254nm

T	Hight	Area	Area%
14.854	102013	2052181	8.843
18.258	811142	21155421	91.157



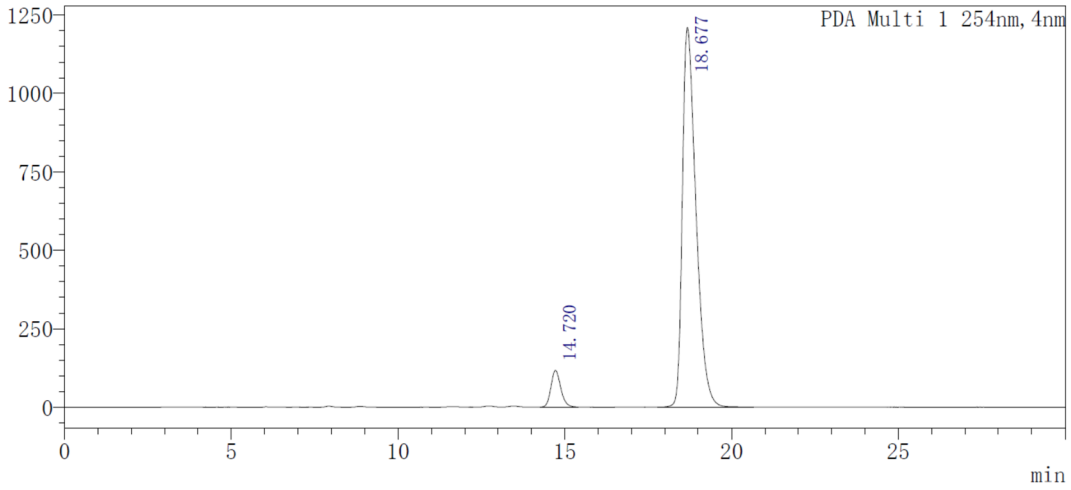
8

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 14.7 min, t(major) = 18.7 min, 87% ee (93.5:6.5 er).



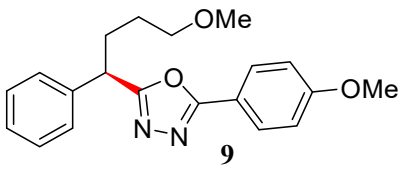
PDA Ch1 254nm

T	Hight	Area	Area%
14.749	228429	4741414	49.961
18.890	182078	4748741	50.039



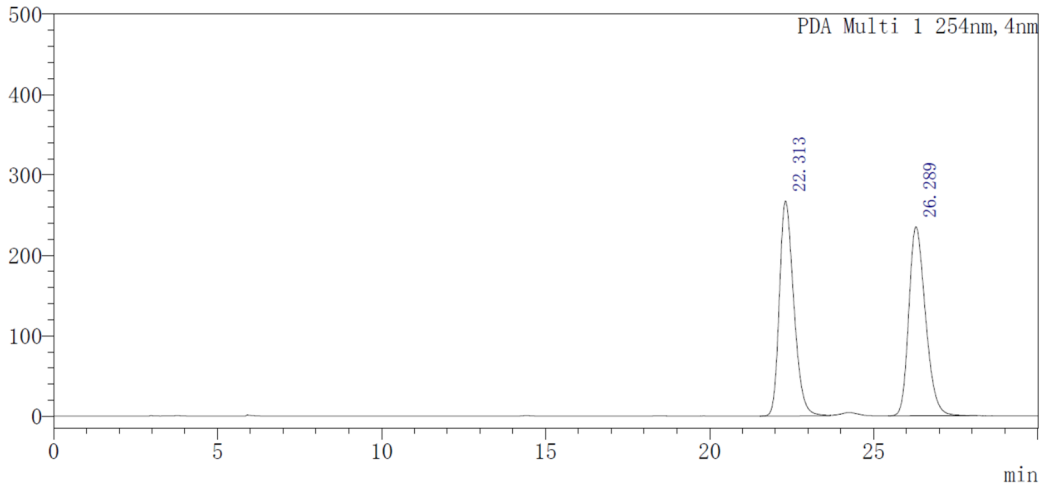
PDA Ch1 254nm

T	Hight	Area	Area%
14.720	116724	2375534	6.481
18.677	1209989	34281072	93.519



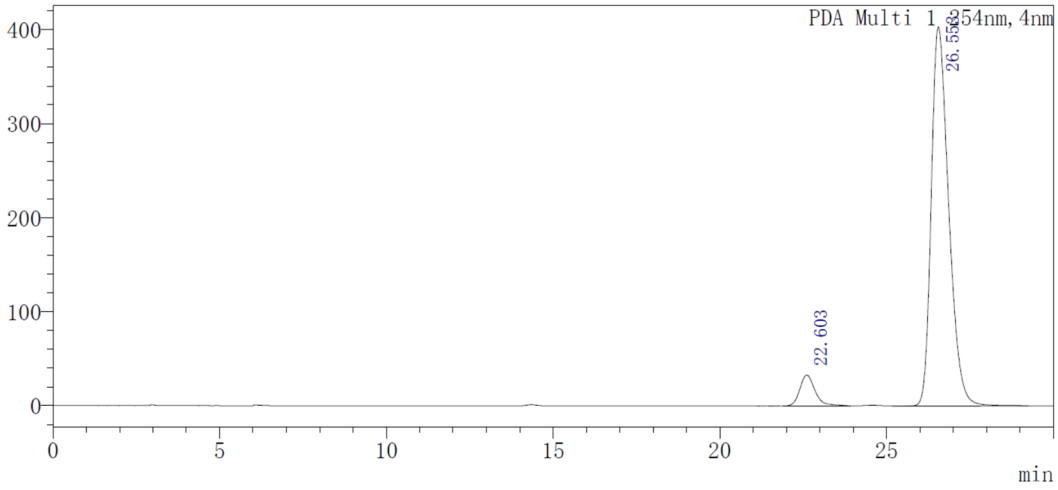
9

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 22.6 min, t(major) = 26.6 min, 87% ee (93.4:6.6 er).



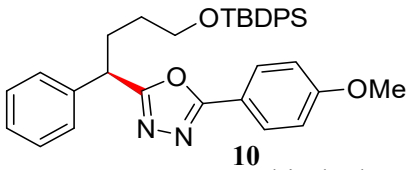
PDA Ch1 254nm

T	Hight	Area	Area%
22.313	267317	8183531	49.568
26.289	235104	8326013	50.432



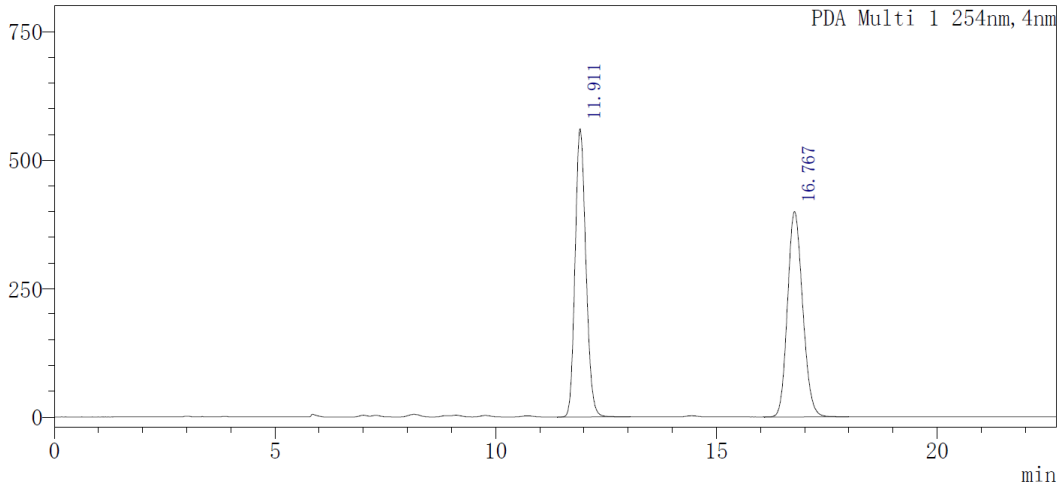
PDA Ch1 254nm

T	Hight	Area	Area%
22.603	32903	1042527	6.639
26.553	403509	14660080	93.361



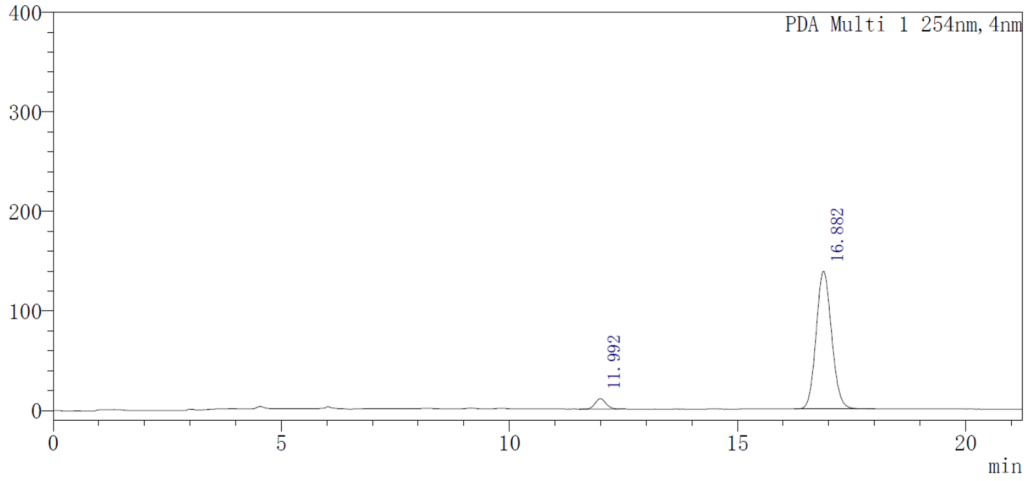
10

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 12.0 min, t(major) = 16.9 min, 90% ee (95:5 er).



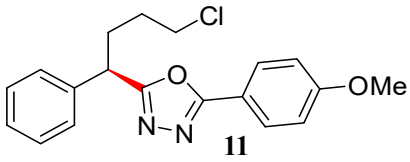
PDA Ch1 254nm

T	Hight	Area	Area%
11.911	567715	9649584	49.962
16.767	404957	9664420	50.038



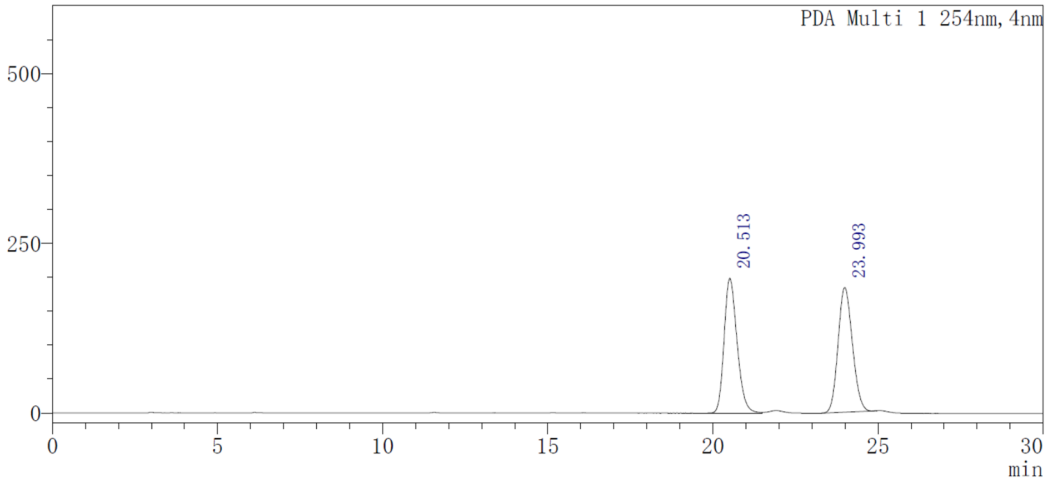
PDA Ch1 254nm

T	Hight	Area	Area%
11.992	10516	174956	5.023
16.882	139931	3307987	94.977



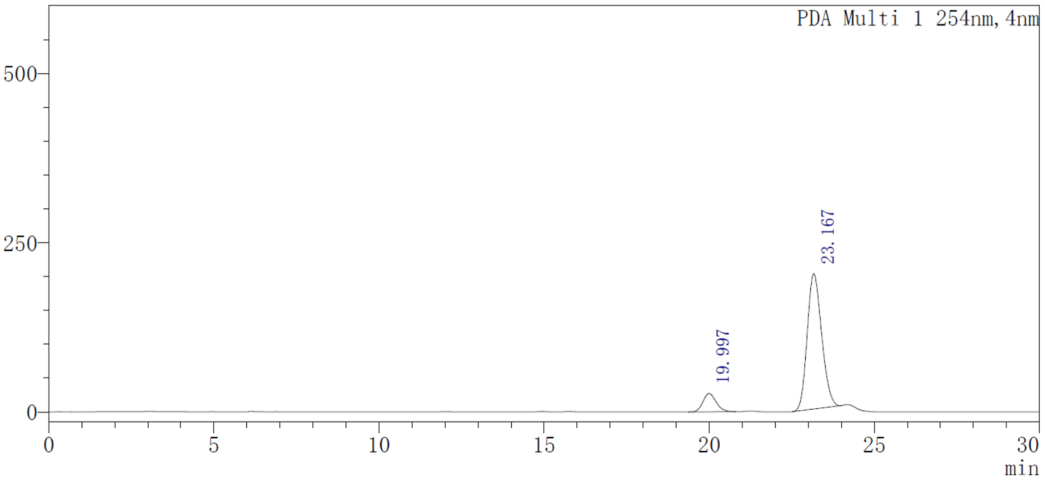
11

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 20.0 min, t(major) = 23.2 min, 78% ee (89:11 er).



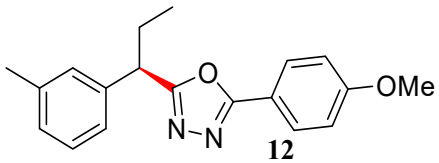
PDA Ch1 254nm

T	Hight	Area	Area%
20.513	198884	5446290	49.550
23.993	183772	5545314	50.450



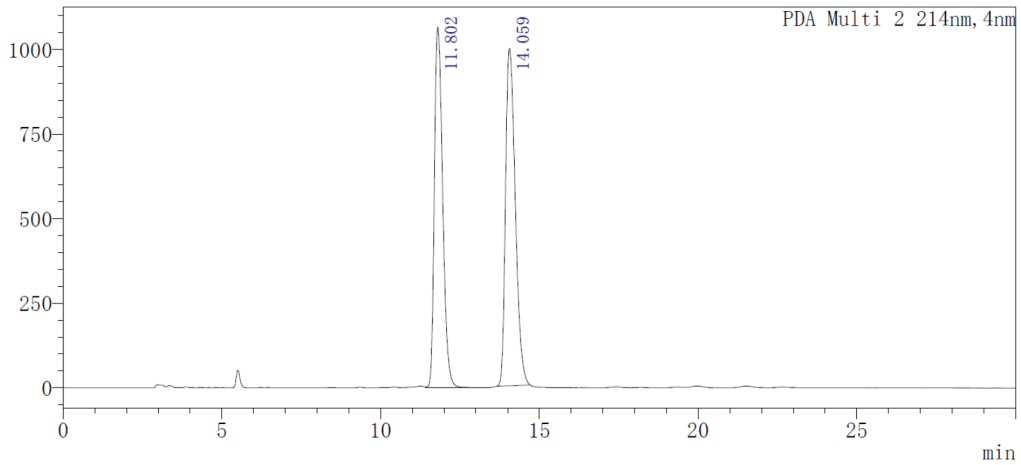
PDA Ch1 254nm

T	Hight	Area	Area%
19.997	27341	758222	11.053
23.167	199975	6101507	88.947



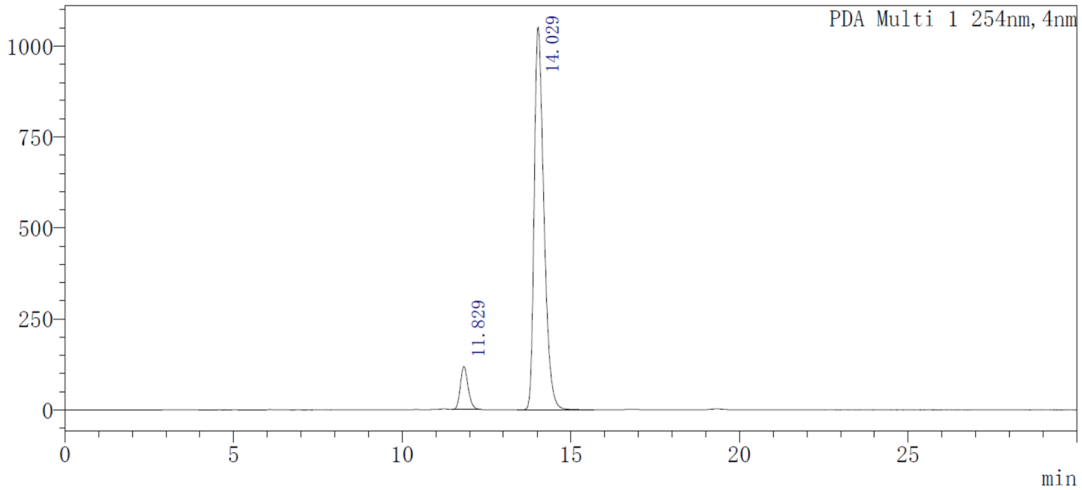
12

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 11.8 min, t(major) = 14.0 min, 83% ee (91.7:8.3 er).



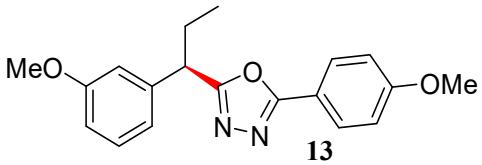
PDA Ch1 254nm

T	Hight	Area	Area%
11.803	1005807	16692172	48.900
14.060	904998	17443267	51.100



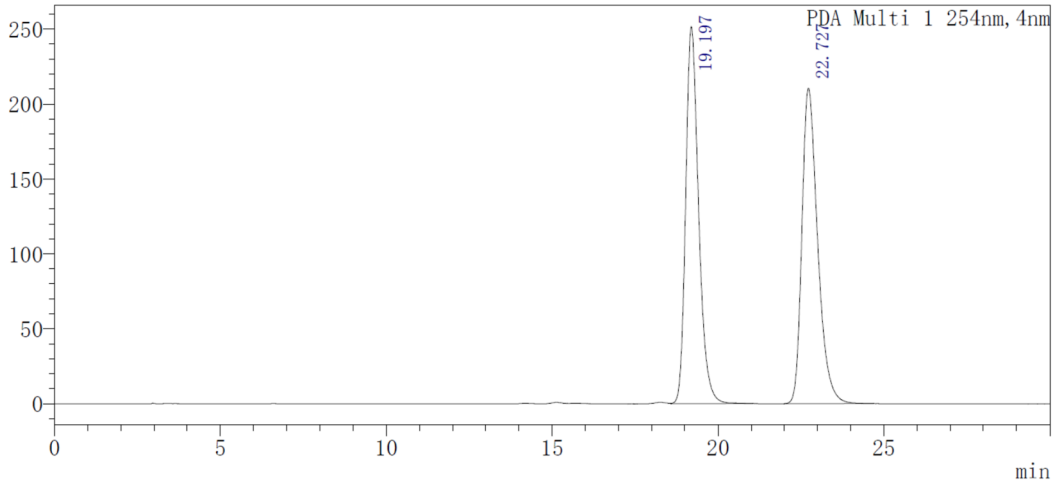
PDA Ch1 254nm

T	Hight	Area	Area%
11.829	118062	1897784	8.256
14.029	1050943	21090206	91.744



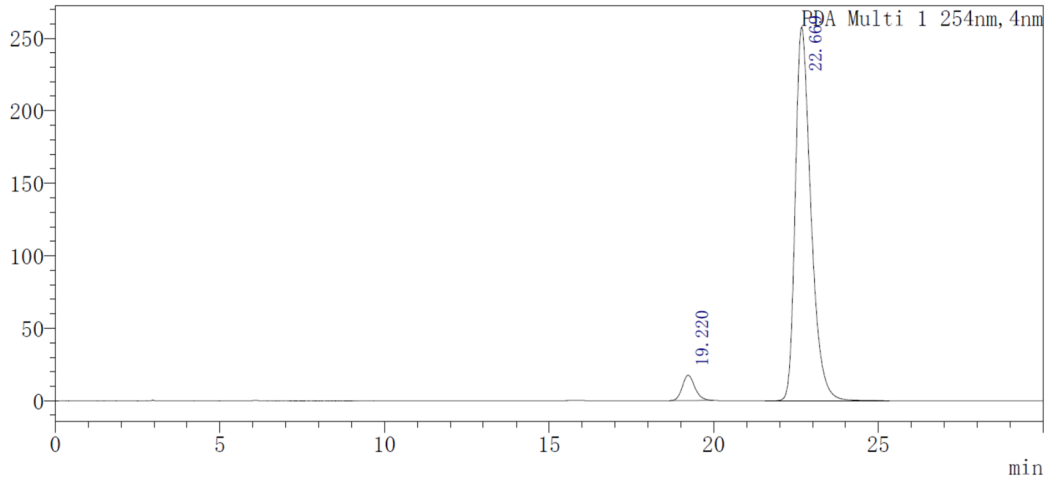
13

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 19.2 min, t(major) = 22.7 min, 90% ee (95:5 er).



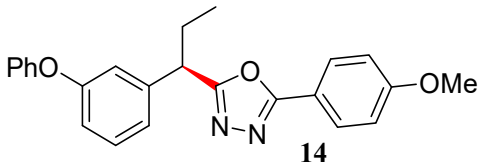
PDA Ch1 254nm

T	Hight	Area	Area%
19.197	251643	6823767	49.515
22.727	210572	6957458	50.485



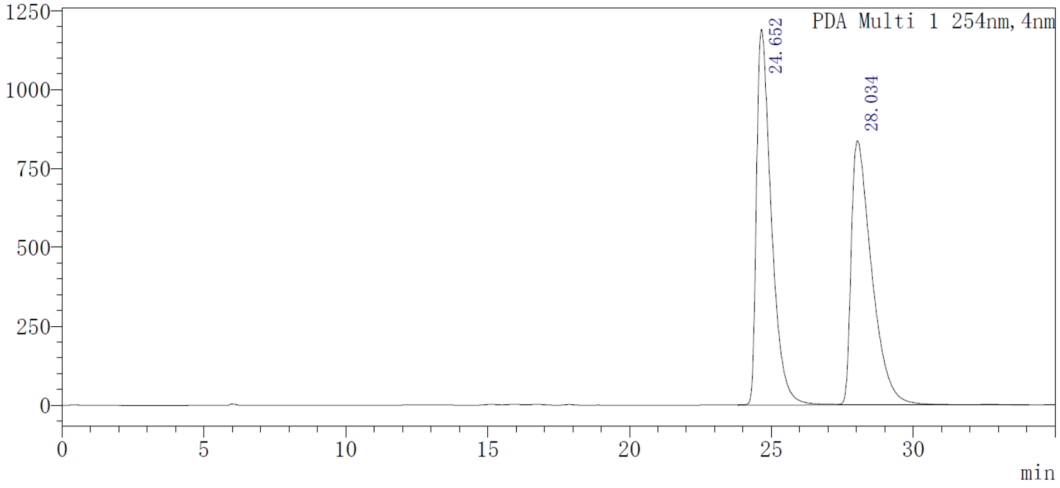
PDA Ch1 254nm

T	Hight	Area	Area%
19.220	17387	458124	5.101
22.669	257703	8522205	94.899



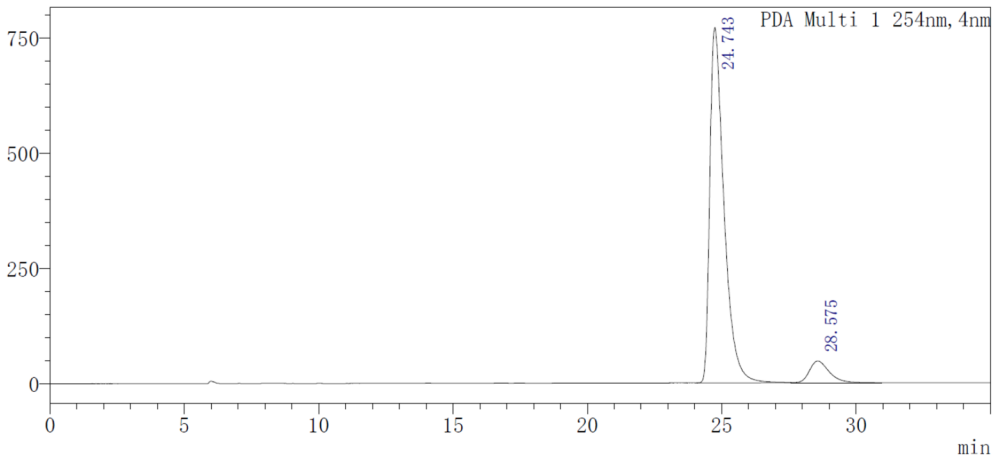
14

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min. λ = 254 nm, t(major) = 24.7 min, t(minor) = 28.6 min, 84% ee (92:8 er).



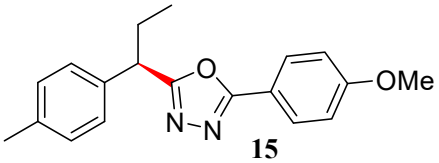
PDA Ch1 254nm

T	Hight	Area	Area%
24.652	1191311	43513420	50.661
28.034	838044	42378706	49.339



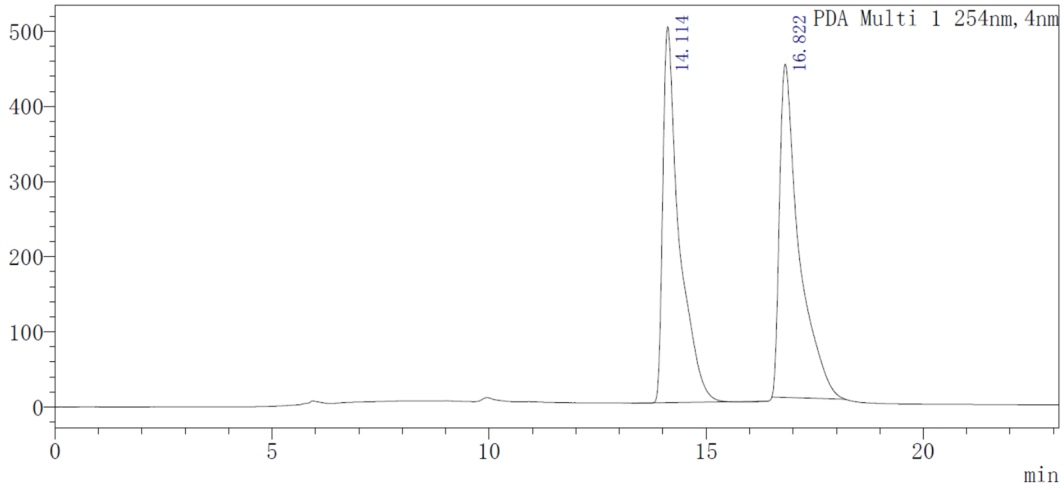
PDA Ch1 254nm

T	Hight	Area	Area%
24.743	771263	28349428	92.130
28.575	47175	2421632	7.870



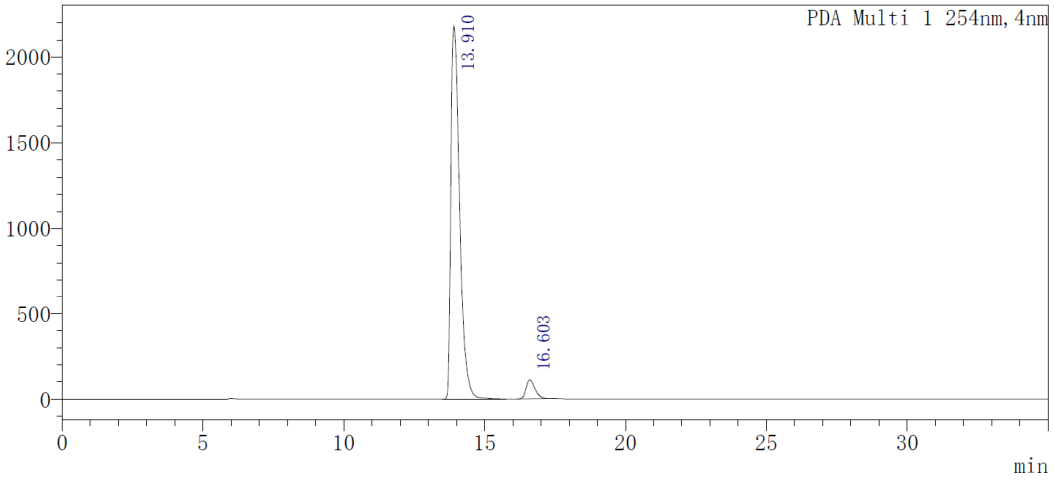
15

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min. λ = 254 nm, t(major) = 13.9 min, t(minor) = 16.6 min, 90% ee (95:5 er).



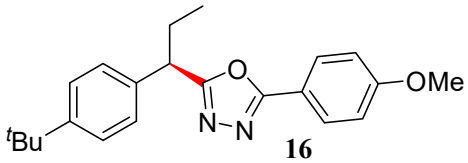
PDA Ch1 254nm

T	Hight	Area	Area%
14.114	500145	13090804	48.467
16.822	443823	13918788	51.533



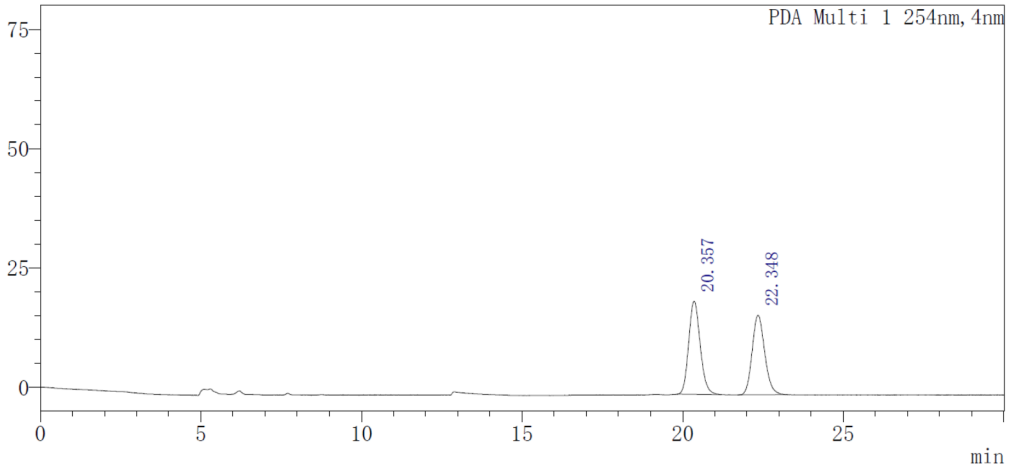
PDA Ch1 254nm

T	Hight	Area	Area%
13.910	2178093	47233419	95.095
16.603	110794	2436266	4.905



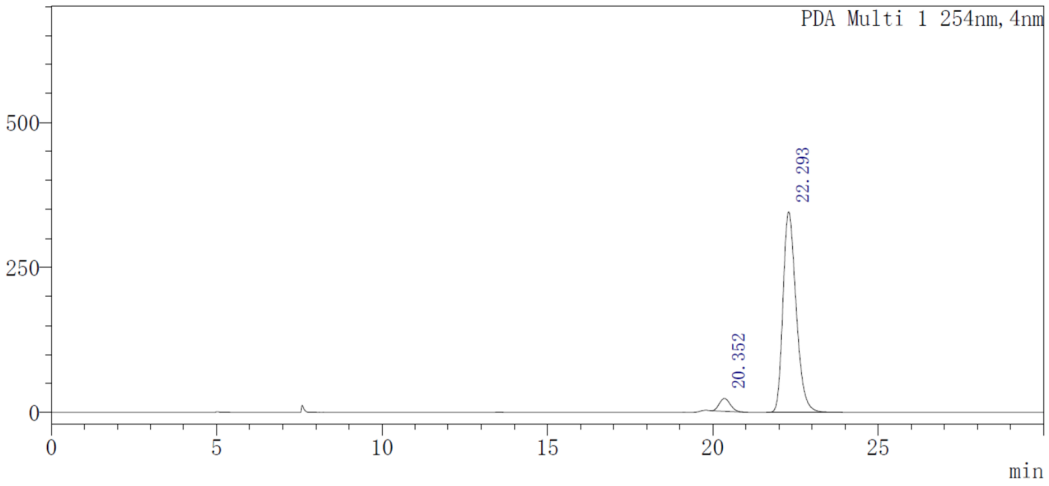
16

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.6 mL/min. λ = 254 nm, t(minor) = 20.4 min, t(major) = 22.3 min, 90% ee (95:5 er).



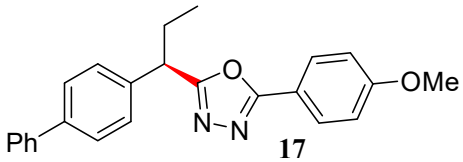
PDA Ch1 254nm

T	Hight	Area	Area%
20.357	19727	492157	51.715
22.348	16868	459507	48.285



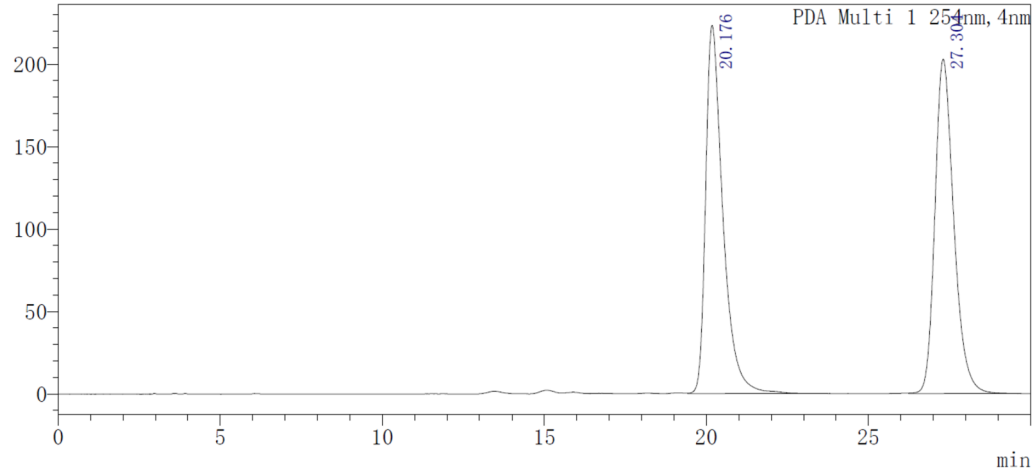
PDA Ch1 254nm

T	Hight	Area	Area%
20.352	22481	518415	5.090
22.293	349261	9666733	94.910



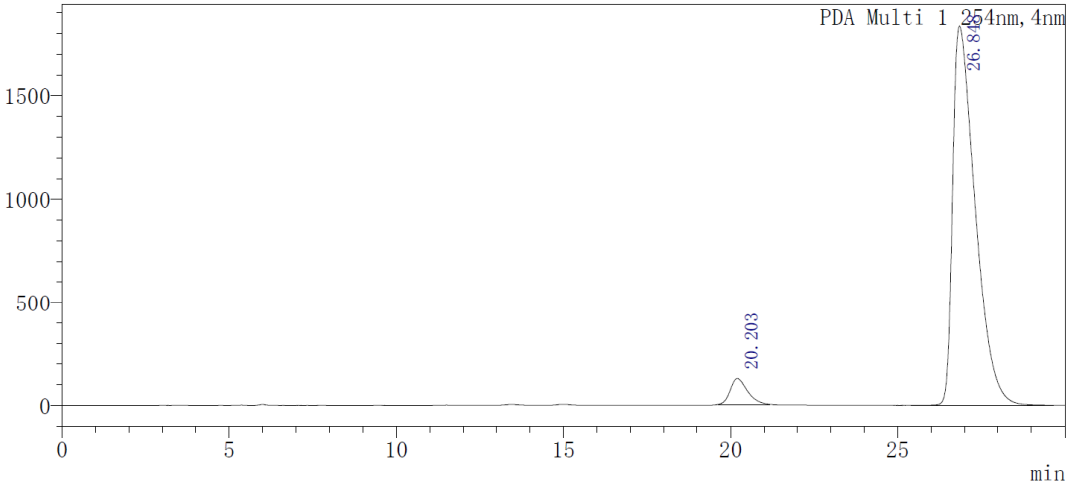
17

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 20.2 min, t(major) = 26.8 min, 90% ee (95.1:4.9 er).



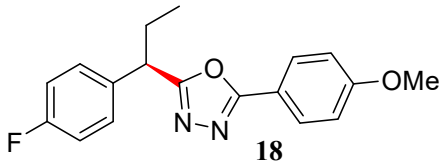
PDA Ch1 254nm

T	Hight	Area	Area%
20.176	223309	8260687	50.078
27.304	202824	8235015	49.922



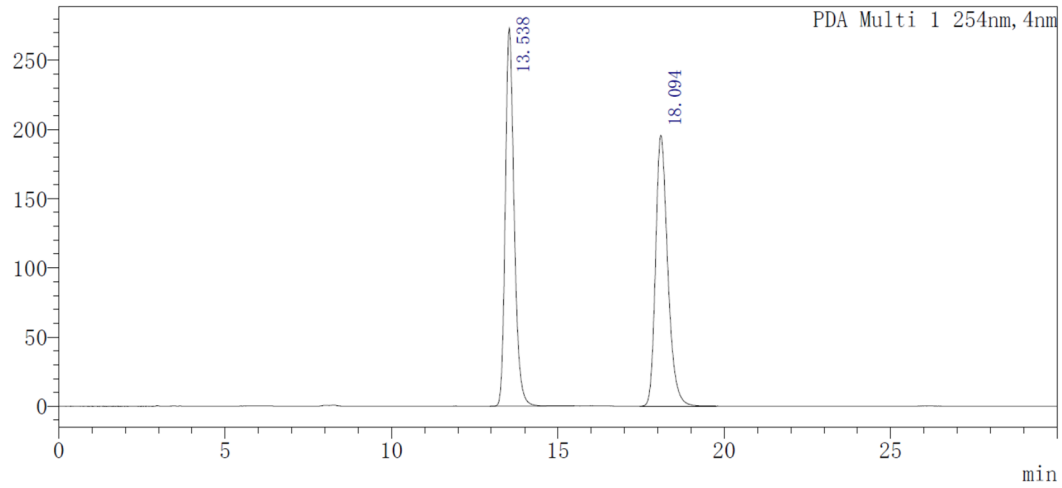
PDA Ch1 254nm

T	Hight	Area	Area%
20.203	124815	4423818	4.887
26.848	1836097	86105590	95.113



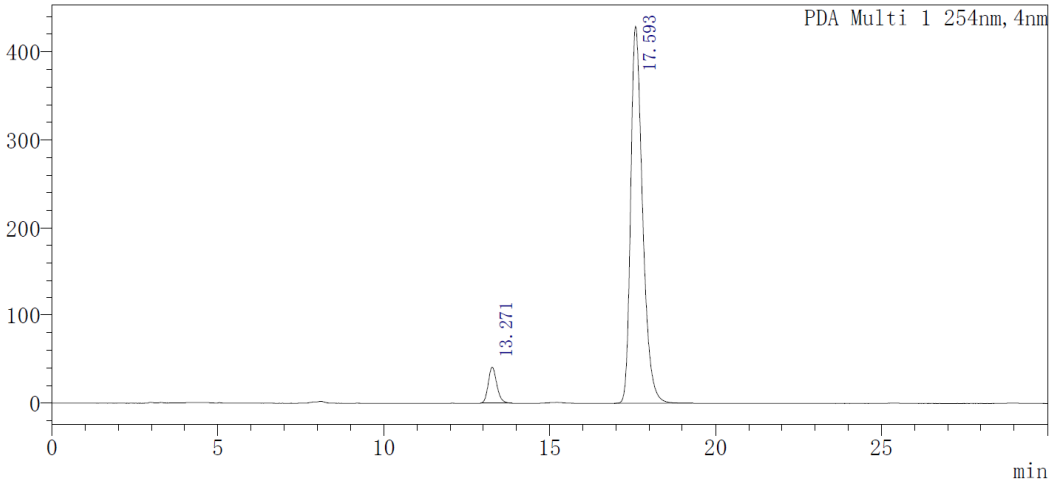
18

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 13.3 min, t(major) = 17.6 min, 87% ee (93.6:6.4 er).



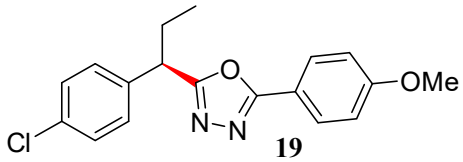
PDA Ch1 254nm

T	Hight	Area	Area%
13.538	273165	5057328	50.340
18.094	195761	4989020	49.660



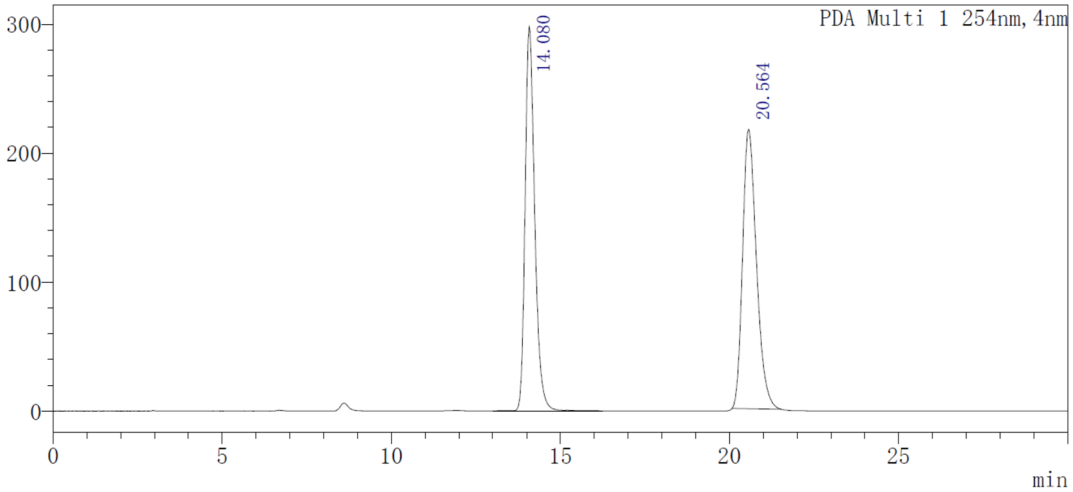
PDA Ch1 254nm

T	Hight	Area	Area%
13.271	40938	733804	6.404
17.593	428976	10723833	93.596



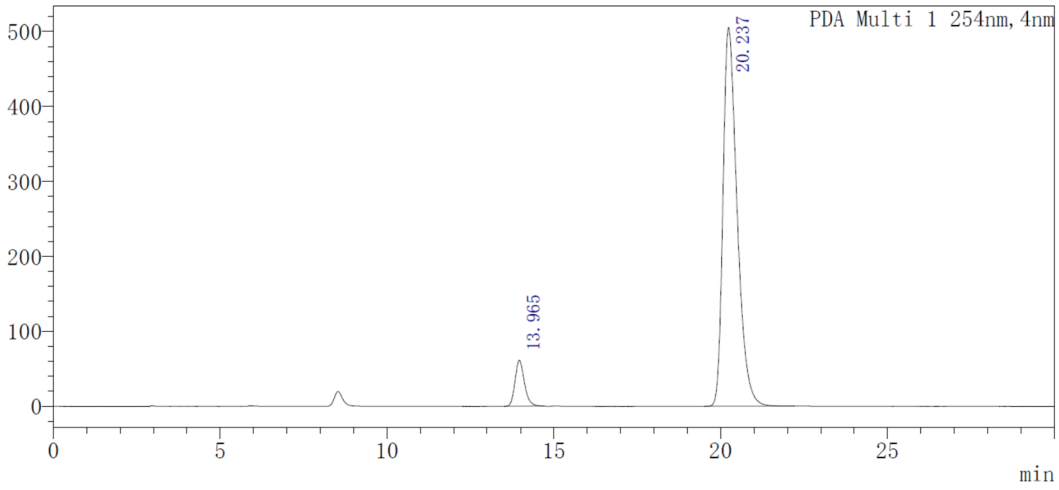
19

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 14.0 min, t(major) = 20.2 min, 85% ee (92.5:7.5 er).



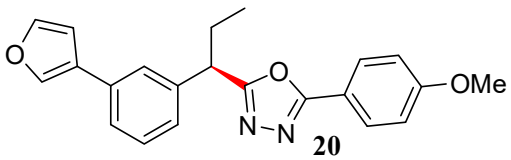
PDA Ch1 254nm

T	Hight	Area	Area%
14.080	297756	5887598	48.844
20.564	216536	6166216	51.156



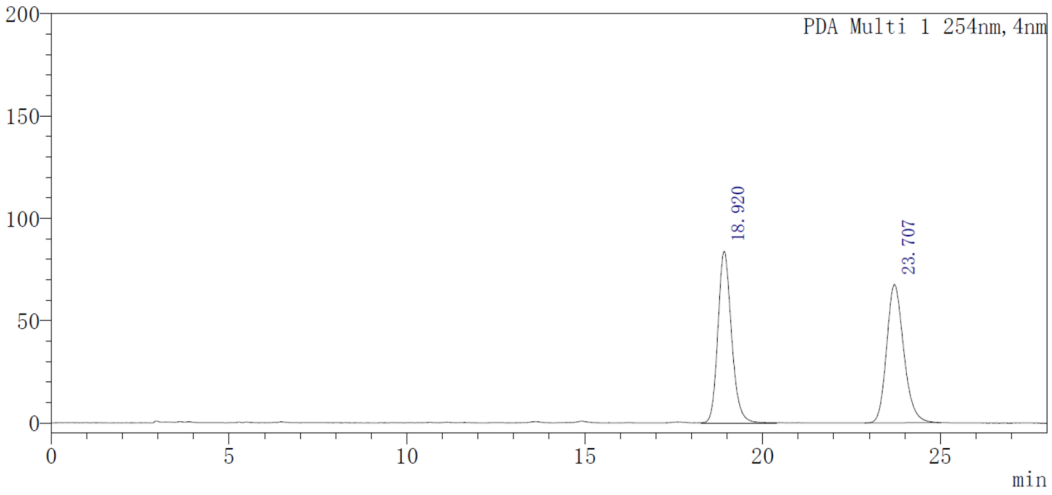
PDA Ch1 254nm

T	Hight	Area	Area%
13.965	61284	1205091	7.489
20.237	505124	14885670	92.511



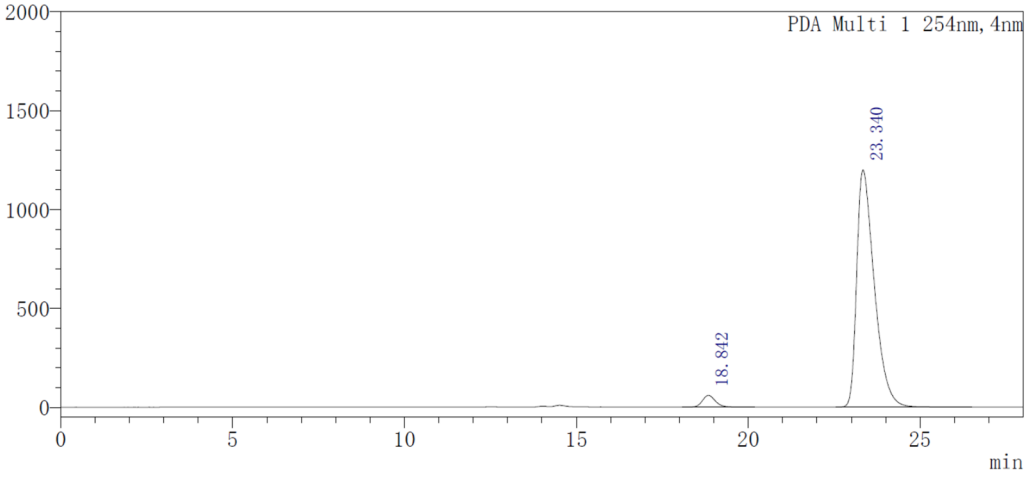
20

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 18.8 min, t(major) = 23.3 min, 93% ee (96.4:3.6 er).



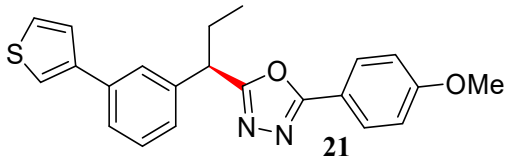
PDA Ch1 254nm

T	Hight	Area	Area%
18.920	83885	2230766	49.568
23.707	67666	2269621	50.432



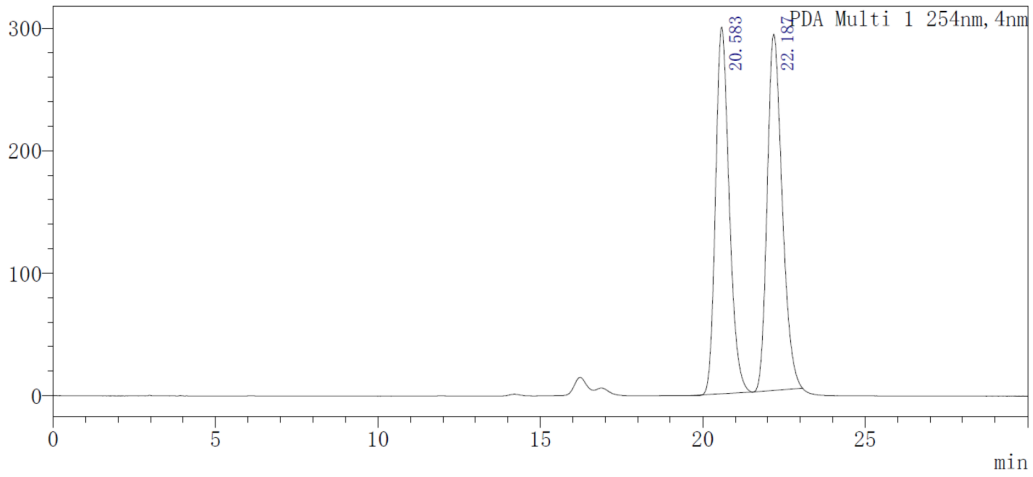
PDA Ch1 254nm

T	Hight	Area	Area%
18.842	60290	1597919	3.598
23.340	1200852	42807561	96.402



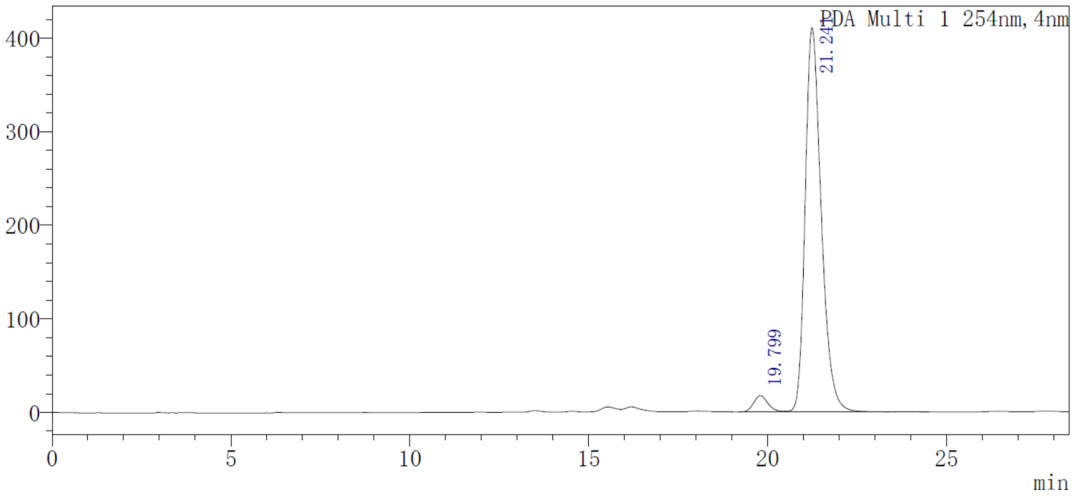
21

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 19.8 min, t(major) = 21.2 min, 93% ee (96.3:3.7 er).



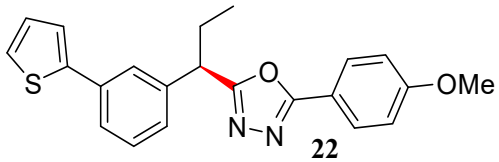
PDA Ch1 254nm

T	Hight	Area	Area%
20.583	299466	8769107	48.401
22.187	291022	9348559	51.599



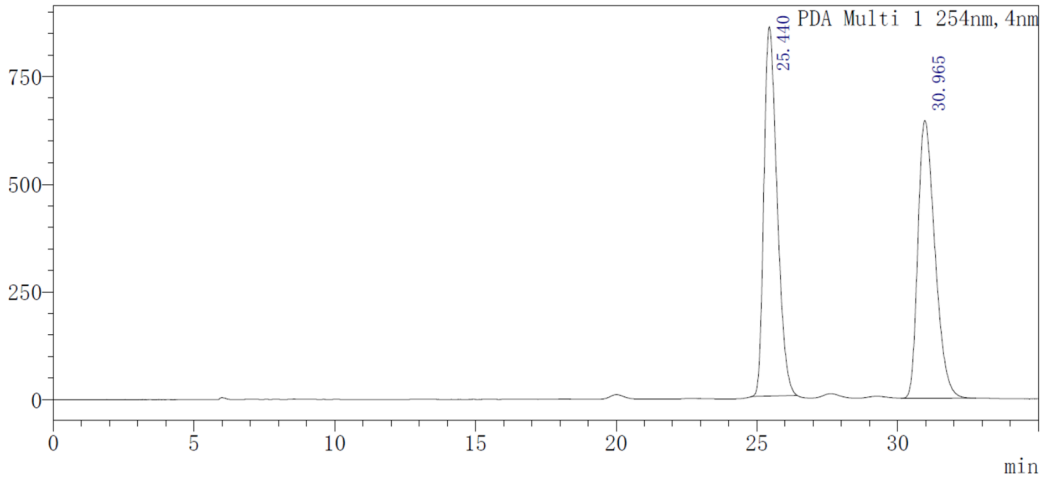
PDA Ch1 254nm

T	Hight	Area	Area%
19.799	17469	489874	3.671
21.241	410390	12856034	96.329



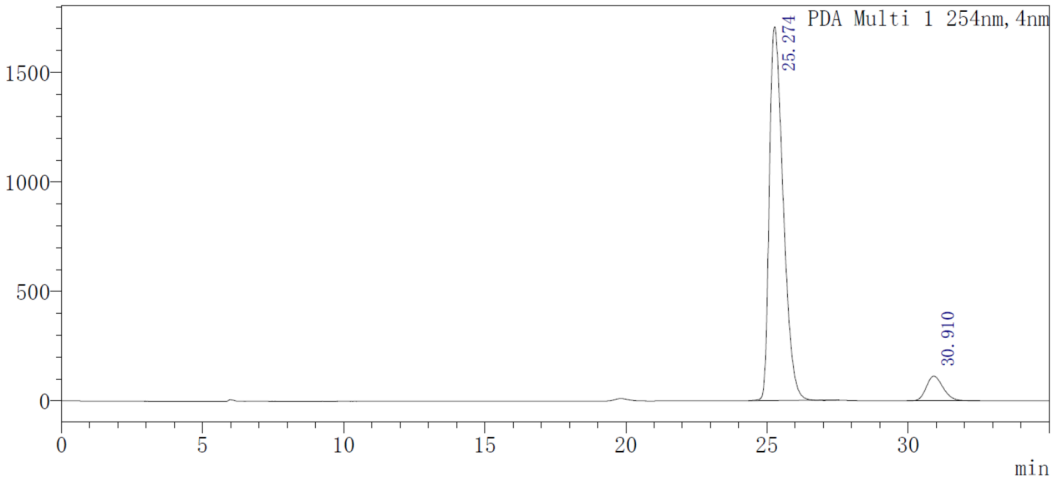
22

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min. λ = 254 nm, t(major) = 25.3 min, t(minor) = 30.9 min, 85% ee (92.5:7.5 er).



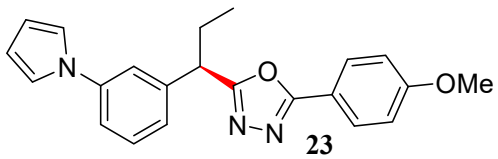
PDA Ch1 254nm

T	Hight	Area	Area%
25.440	857567	27973949	50.698
30.965	645251	27204111	49.302



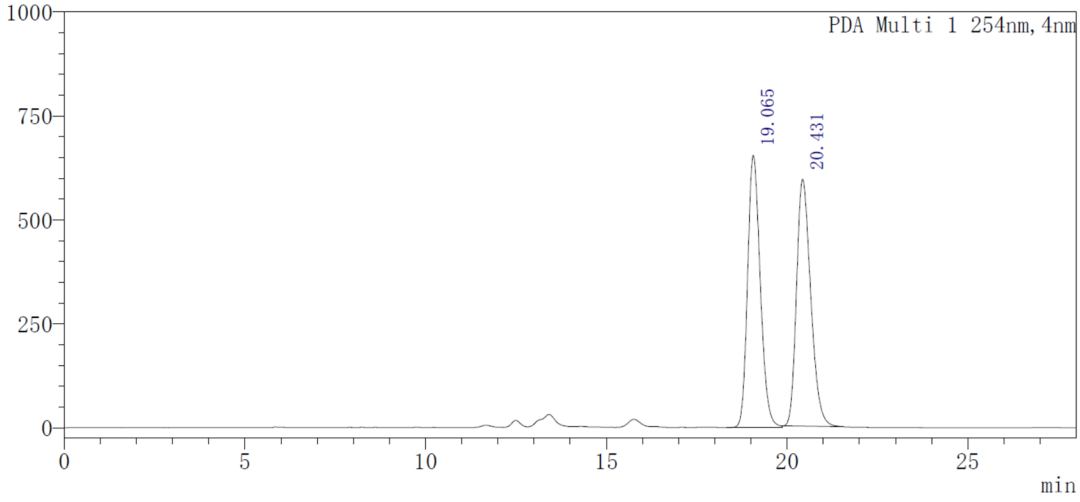
PDA Ch1 254nm

T	Hight	Area	Area%
25.274	1707297	58340610	92.582
30.910	112810	4674250	7.418



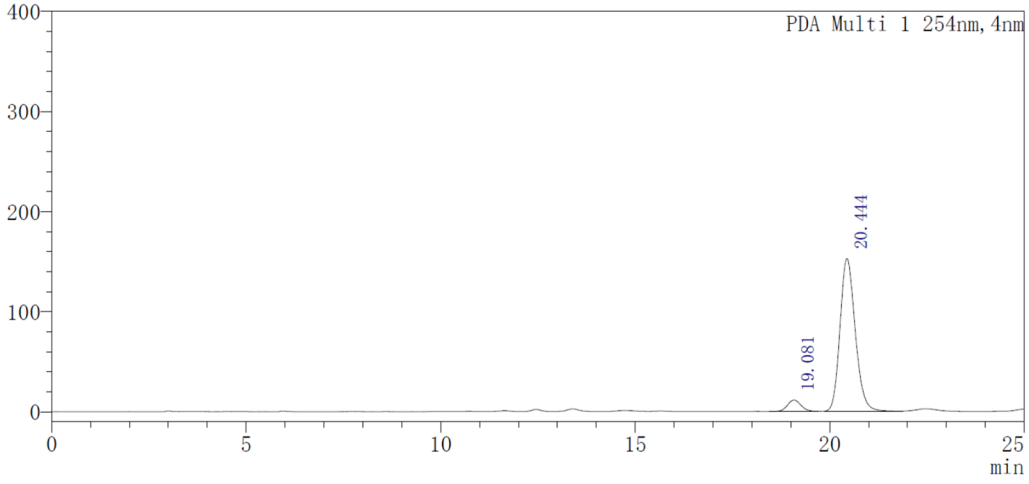
23

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 19.1 min, t(major) = 20.4 min, 87% ee (93.6:6.4 er).



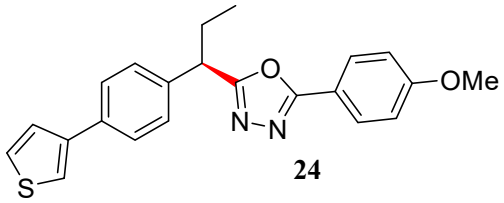
PDA Ch1 254nm

T	Hight	Area	Area%
19.065	655025	16016089	49.704
20.431	593734	16207106	50.296



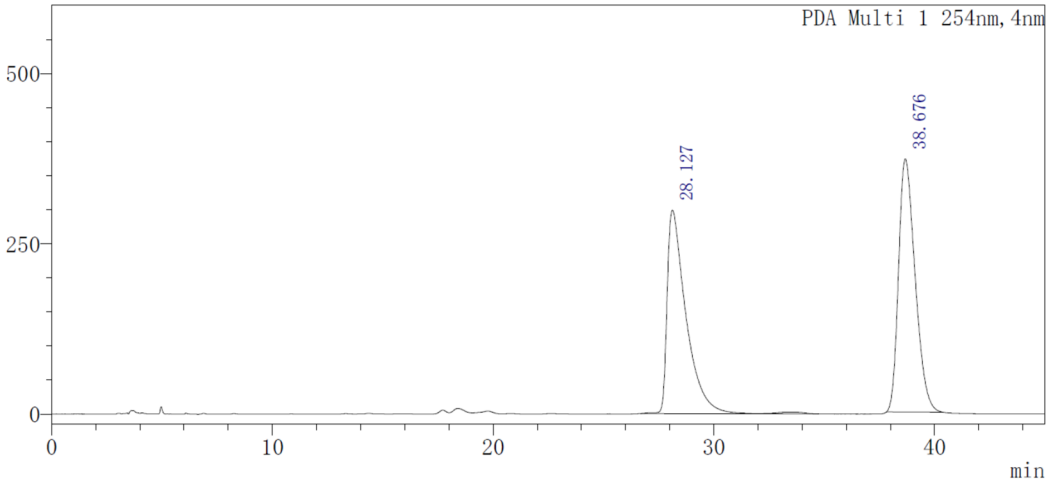
PDA Ch1 254nm

T	Hight	Area	Area%
19.081	11591	282797	6.376
20.444	152727	4152314	93.624



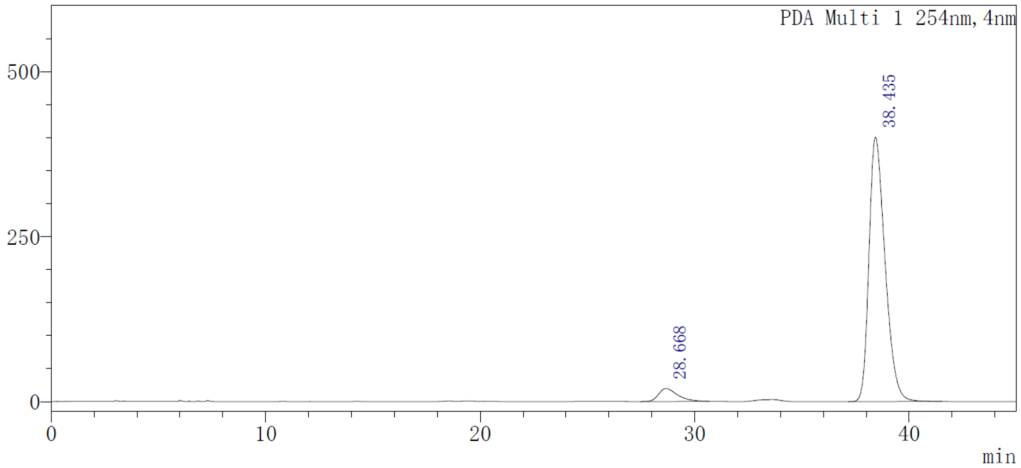
24

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 28.7 min, t(major) = 38.4 min, 89% ee (94.5:5.5 er).



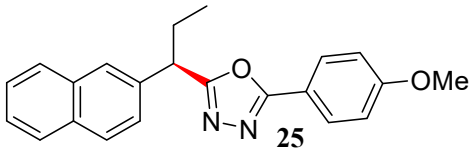
PDA Ch1 254nm

T	Hight	Area	Area%
28.127	298934	17881878	47.999
38.676	371803	19372489	52.001



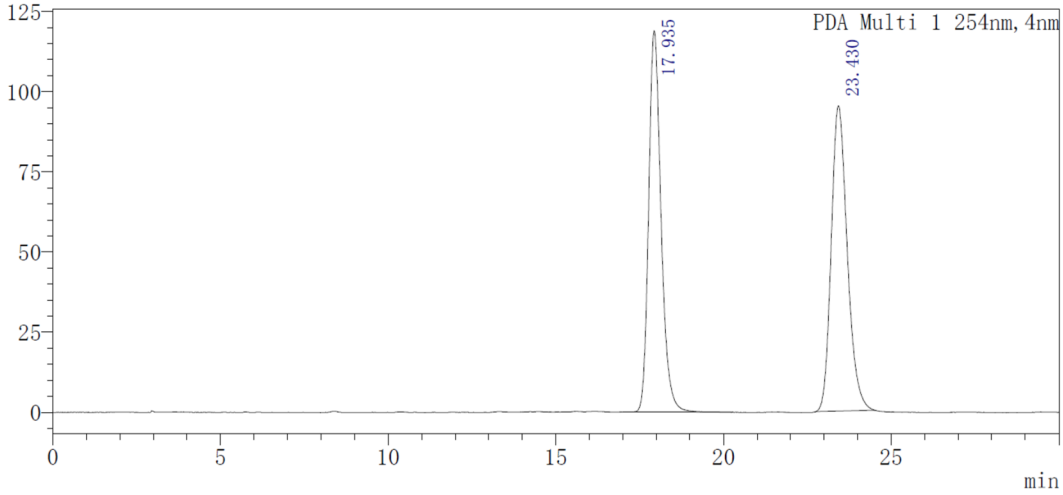
PDA Ch1 254nm

T	Hight	Area	Area%
28.668	19737	1214728	5.449
38.435	404044	21079883	94.551



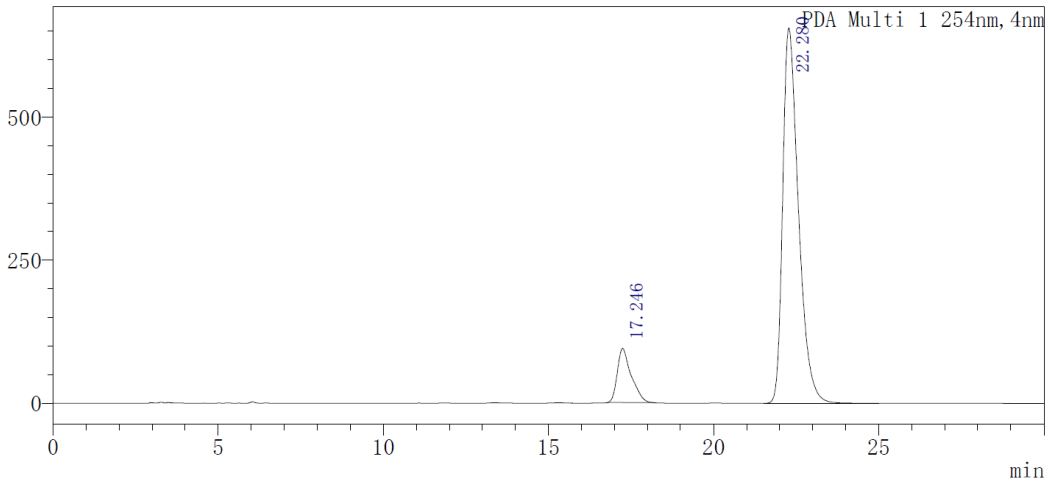
25

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 17.2 min, t(major) = 22.3 min, 77% ee (88.7:11.3 er).



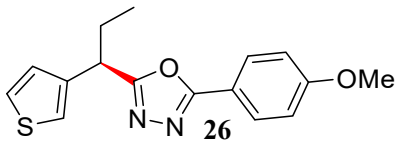
PDA Ch1 254nm

T	Hight	Area	Area%
17.935	118908	3064242	49.177
23.430	95209	3166763	50.823



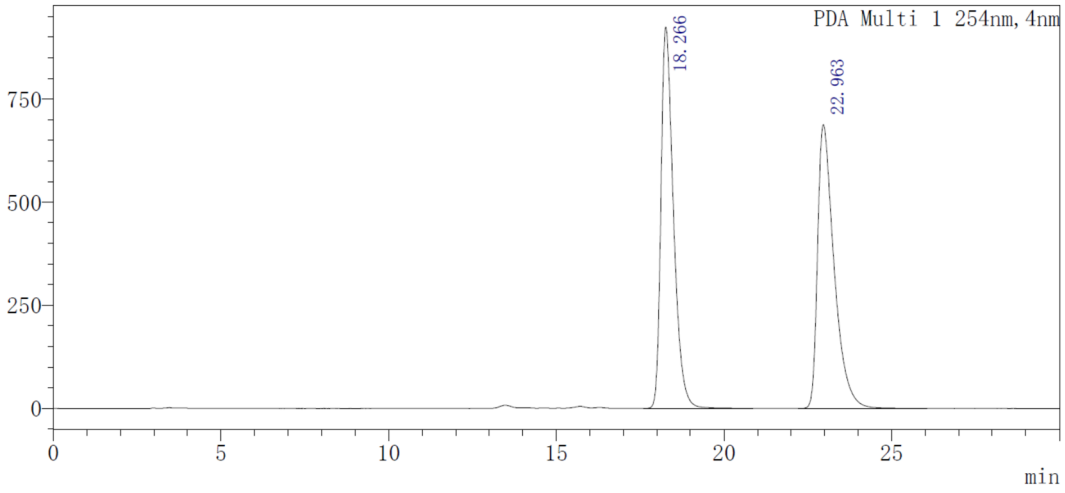
PDA Ch1 254nm

T	Hight	Area	Area%
17.246	94705	2763340	11.262
22.280	655987	21772619	88.738



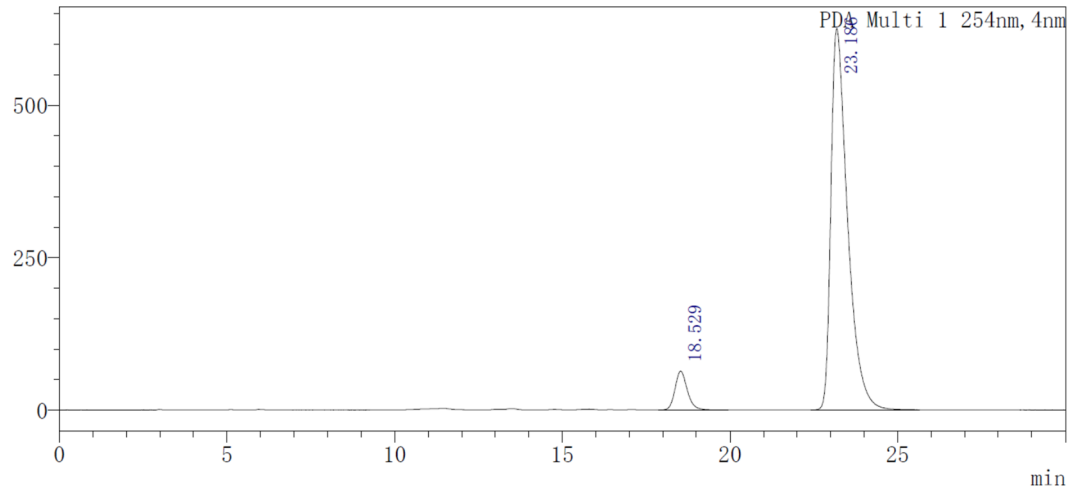
26

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 18.5 min, t(major) = 23.2 min, 86% ee (93:7 er).



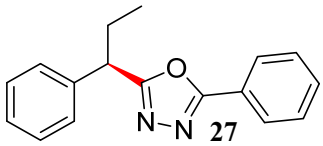
PDA Ch1 254nm

T	Hight	Area	Area%
18.266	924033	23980570	50.423
22.963	687766	23578505	49.577

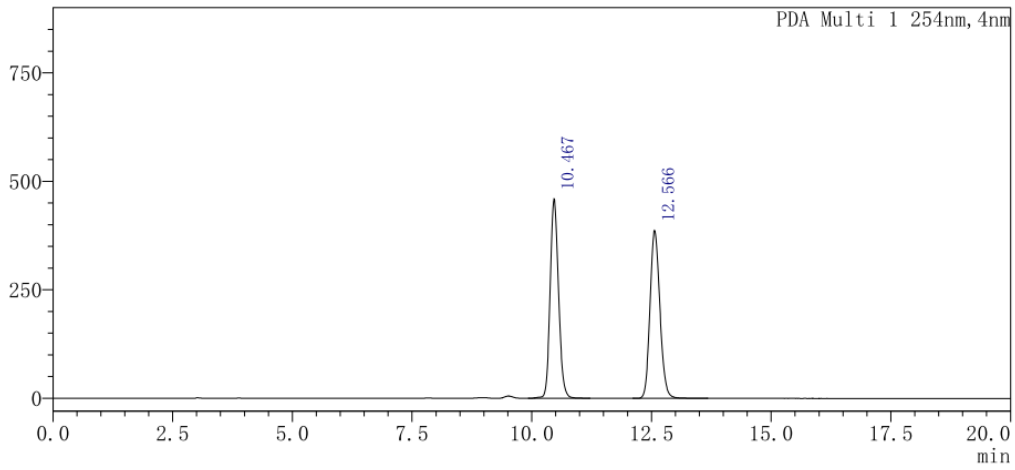


PDA Ch1 254nm

T	Hight	Area	Area%
18.529	63642	1612869	6.932
23.186	626006	21655262	93.068

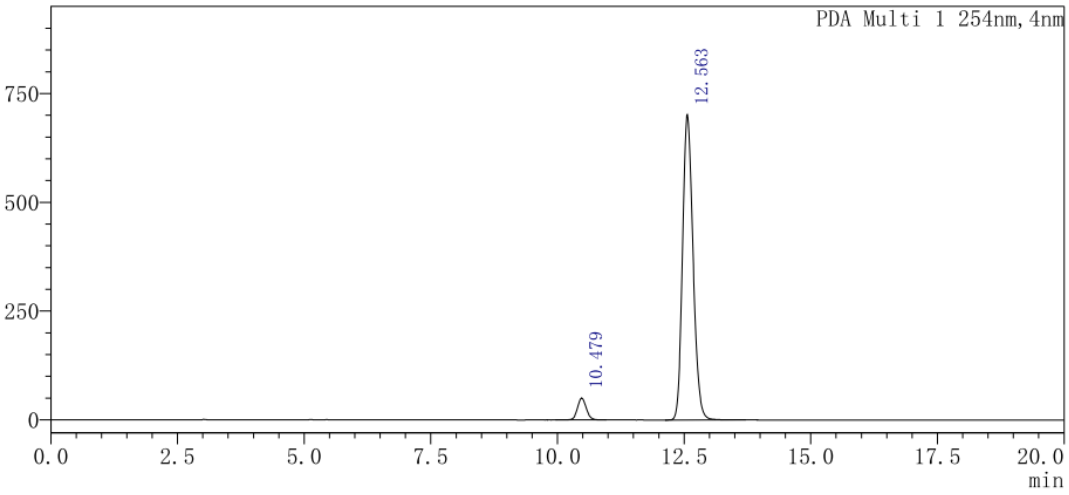


HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min. λ = 254 nm, t(minor) =10.5 min, t(major) = 12.6 min, 89% ee (94.3:5.7 er).



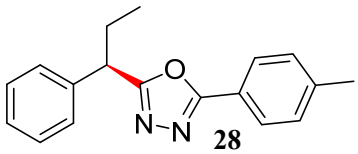
PDA Ch1 254nm

T	Hight	Area	Area%
10.467	457598	5593940	50.187
12.566	385859	5552291	49.813



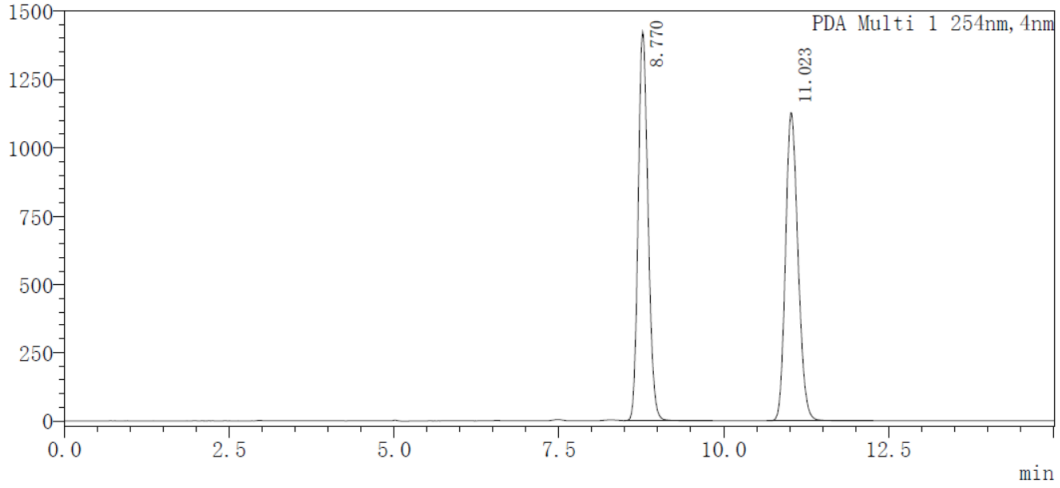
PDA Ch1 254nm

T	Hight	Area	Area%
10.479	50271	611948	5.699
12.563	698615	10126359	94.301



28

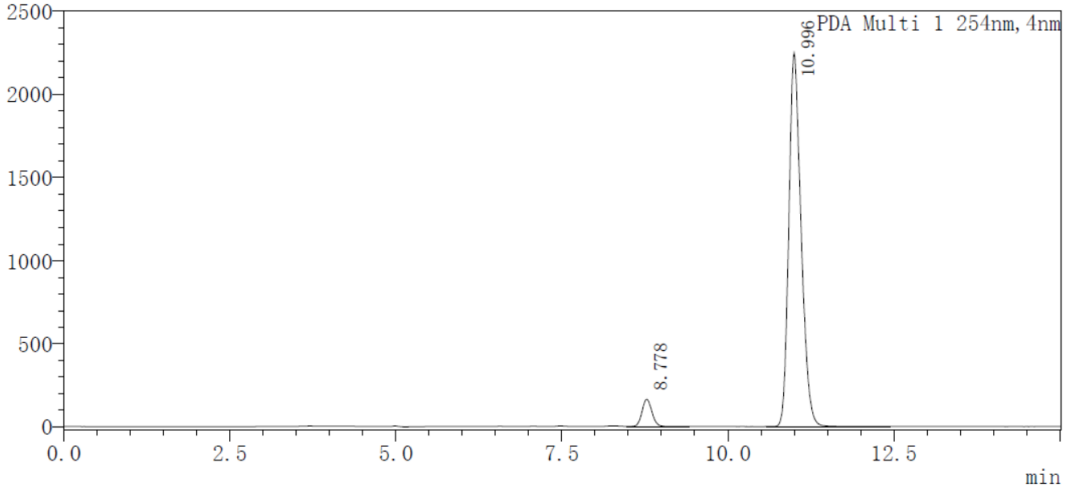
HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 8.8 min, t(major) = 11.0 min, 89% ee (94.5:5.5 er).



Peak Table

PDA Ch1 254nm

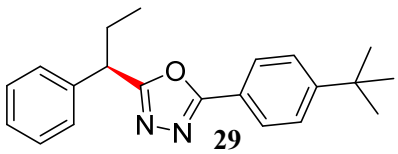
Peak#	Ret. Time	Area	Area%
1	8.770	14851058	49.885
2	11.023	14919306	50.115



Peak Table

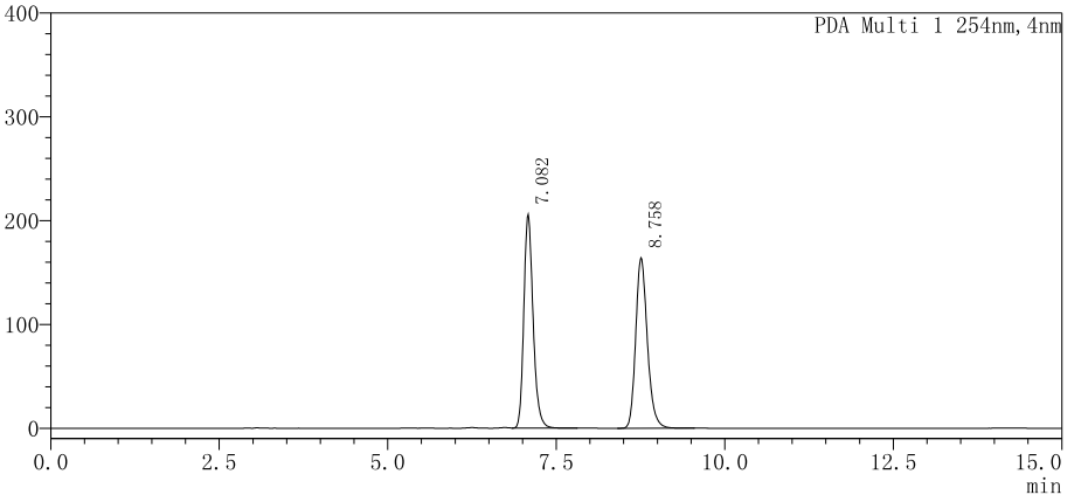
PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	8.778	1690817	5.520
2	10.996	28938449	94.480



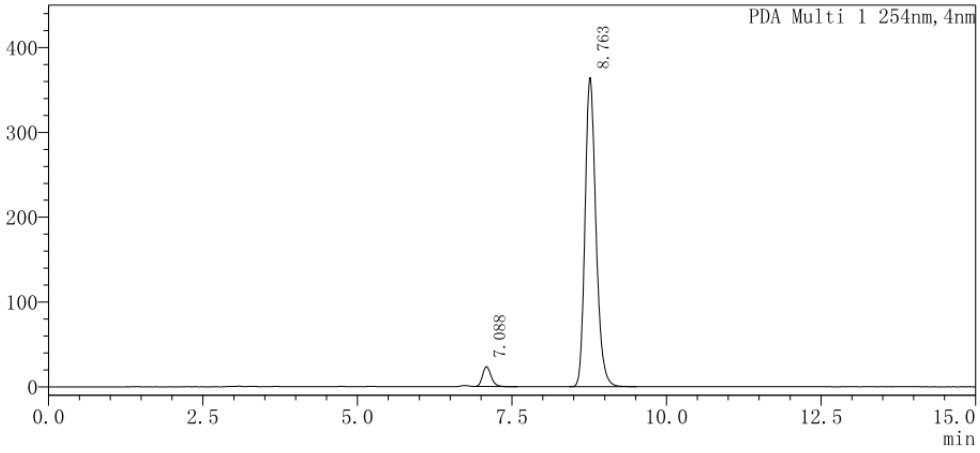
29

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 7.1 min, t(major) = 8.8 min, 90% ee (95.2:4.8 er).



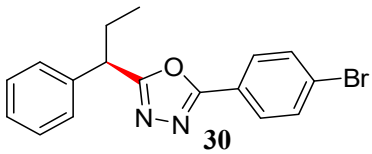
PDA Ch1 254nm

T	Hight	Area	Area%
7.082	206076	1970031	49.942
8.758	164324	1974611	50.058



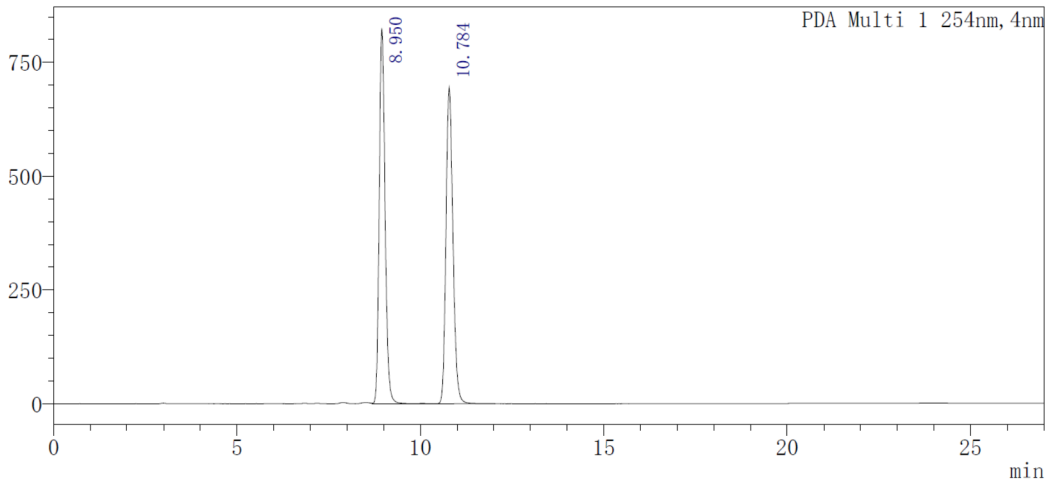
PDA Ch1 254nm

T	Hight	Area	Area%
7.088	23468	228590	4.847
8.763	364535	4487475	95.153



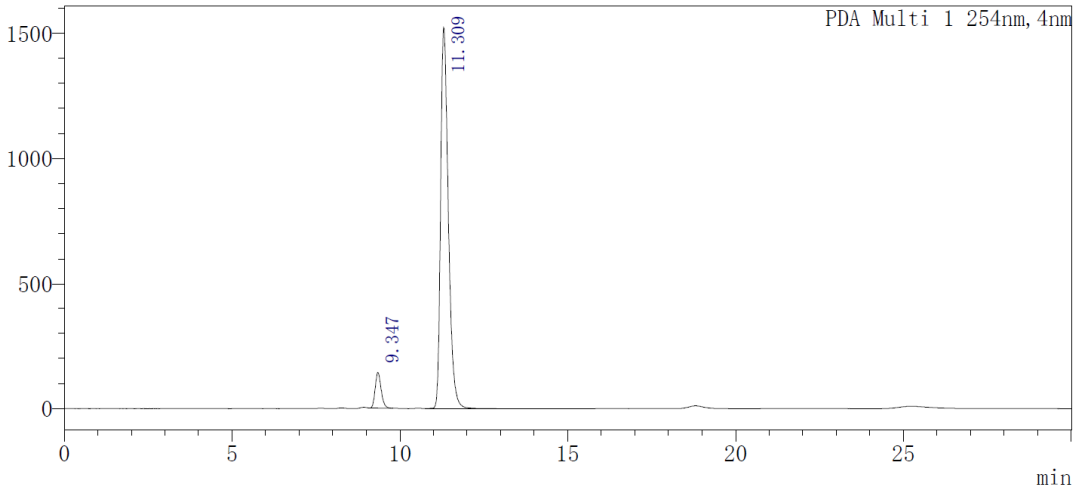
30

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 85/15, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 9.3 min, t(major) = 11.3 min, 87% ee (93.3:6.7 er).



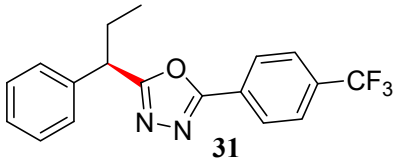
PDA Ch1 254nm

T	Hight	Area	Area%
8.950	824763	9162717	49.960
10.784	694124	9177449	50.040



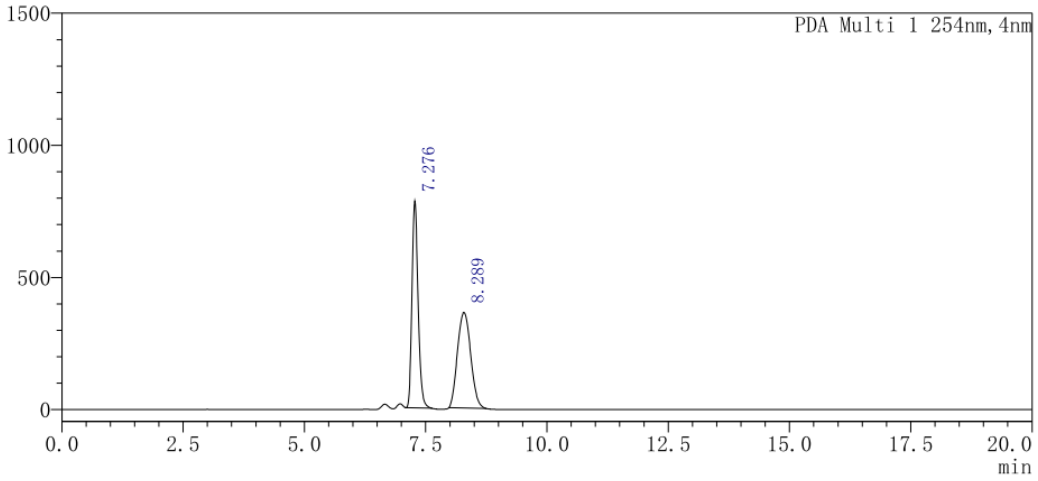
PDA Ch1 254nm

T	Hight	Area	Area%
9.347	141991	1744624	6.673
11.309	1522642	24398382	93.327



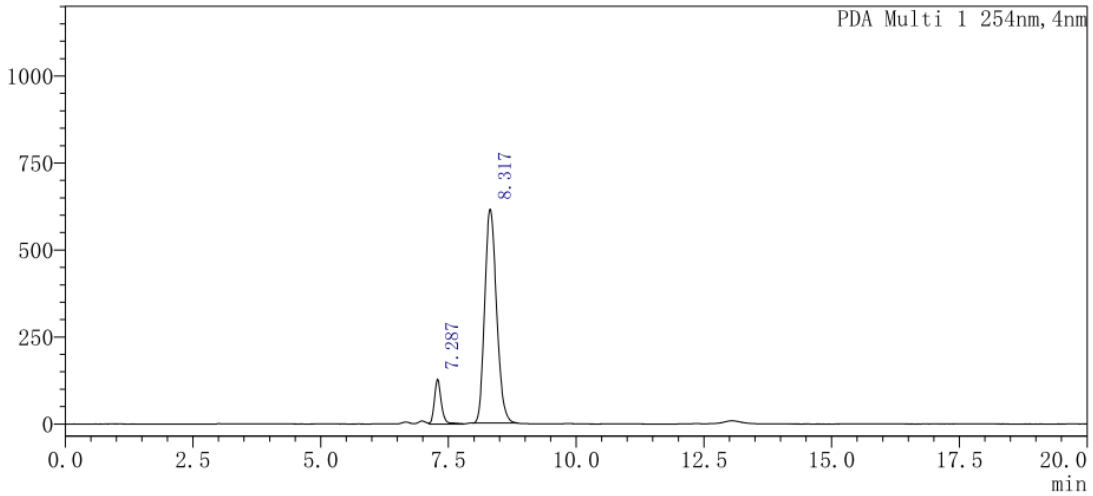
31

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min. λ = 254 nm, t(minor) =7.3 min, t(major) = 8.3 min, 78% ee (89.2:10.8 er).



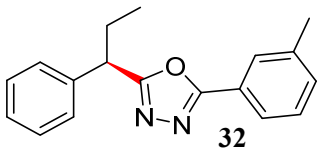
PDA Ch1 254nm

T	Hight	Area	Area%
7.276	782678	6887061	50.118
8.289	361982	6854605	49.882



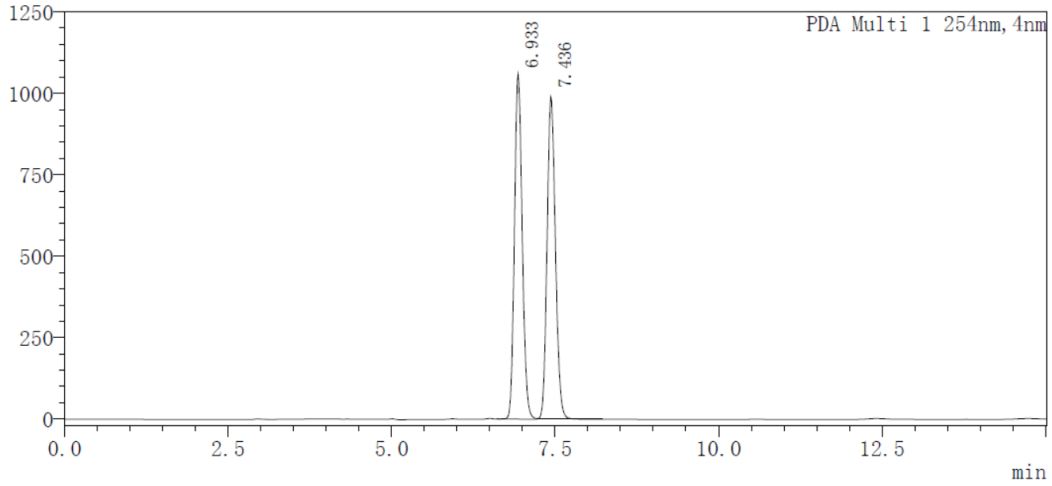
PDA Ch1 254nm

T	Hight	Area	Area%
7.287	127974	1202504	10.781
8.317	614776	9951647	89.219



32

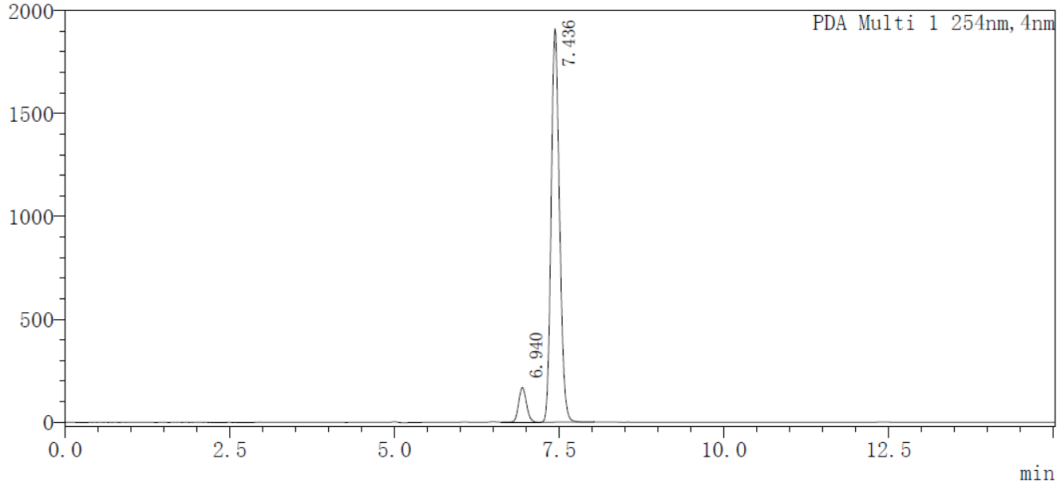
HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 6.9 min, t(major) = 7.4 min, 85% ee (92.4:7.6 er).



Peak Table

PDA Ch1 254nm

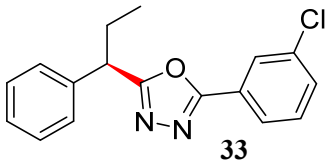
Peak#	Ret. Time	Area	Area%
1	6.933	8779525	49.903
2	7.436	8813510	50.097



Peak Table

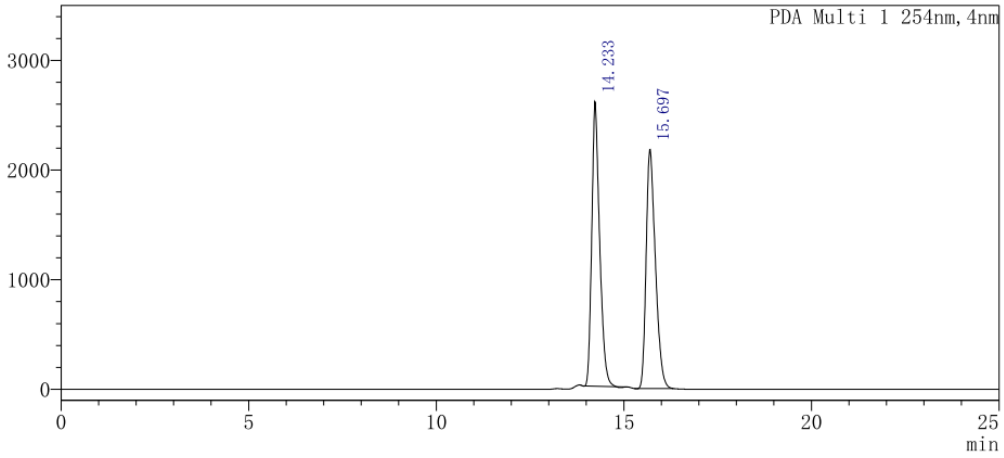
PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	6.940	1385938	7.584
2	7.436	16887749	92.416



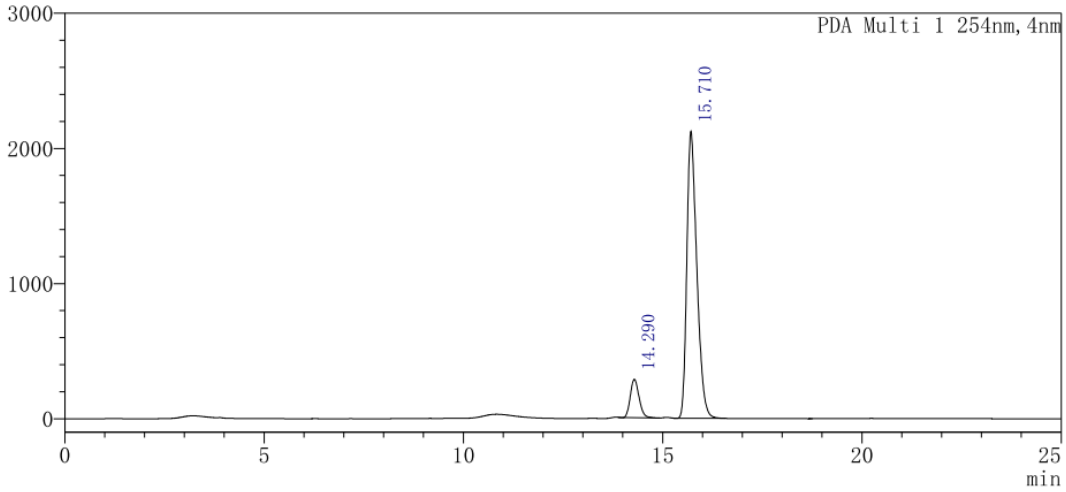
33

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.8 mL/min. λ = 254 nm, t(minor) =14.3 min, t(major) = 15.7 min, 79% ee (89.5:10.5 er).



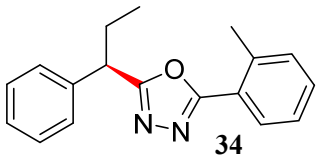
PDA Ch1 254nm

T	Hight	Area	Area%
14.233	2595656	37616078	50.156
15.697	2181228	37382696	49.844



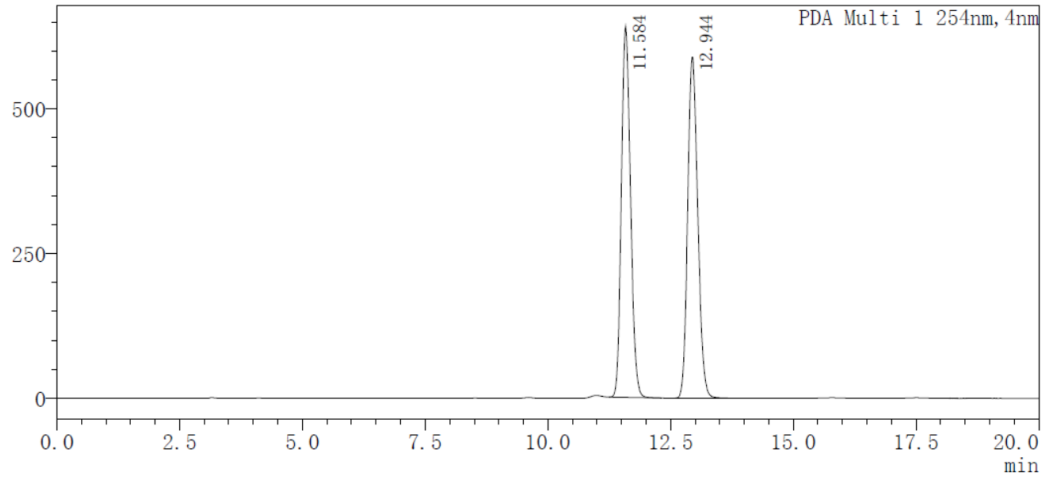
PDA Ch1 254nm

T	Hight	Area	Area%
14.290	284115	4338178	10.515
15.710	2122672	36917848	89.485



34

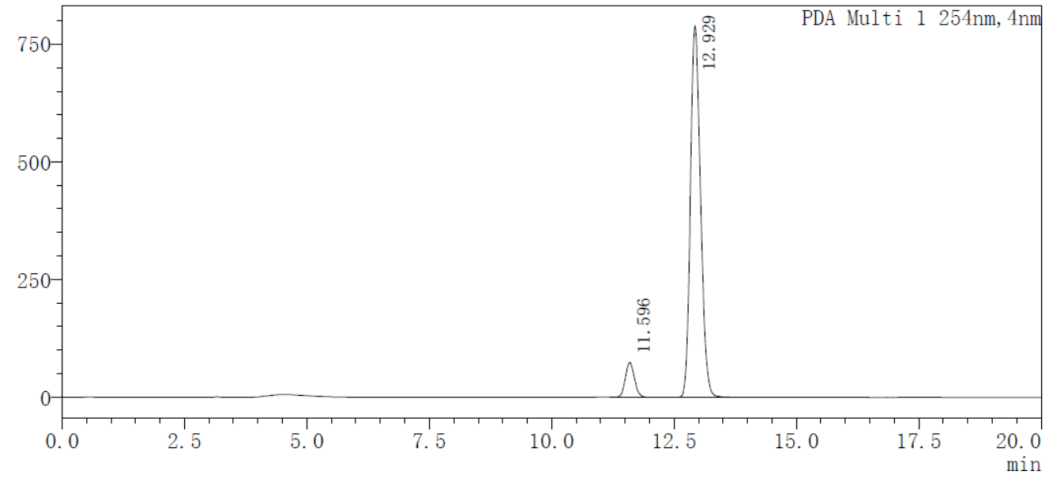
HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 11.6 min, t(major) = 12.9 min, 85% ee (92.3:7.7 er).



Peak Table

PDA Ch1 254nm

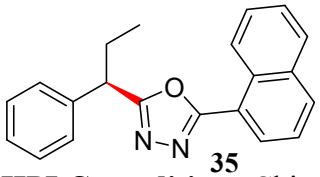
Peak#	Ret. Time	Area	Area%
1	11.584	8324156	49.637
2	12.944	8445759	50.363



Peak Table

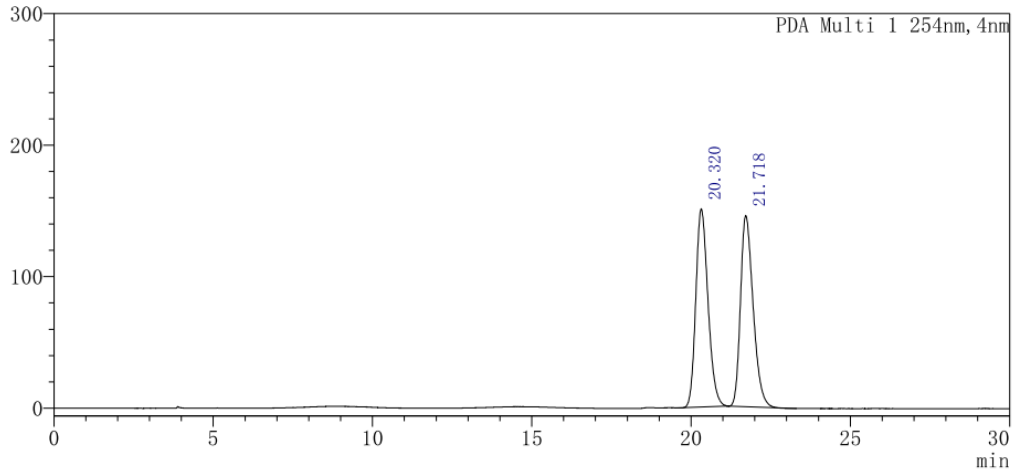
PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	11.596	948664	7.712
2	12.929	11353012	92.288



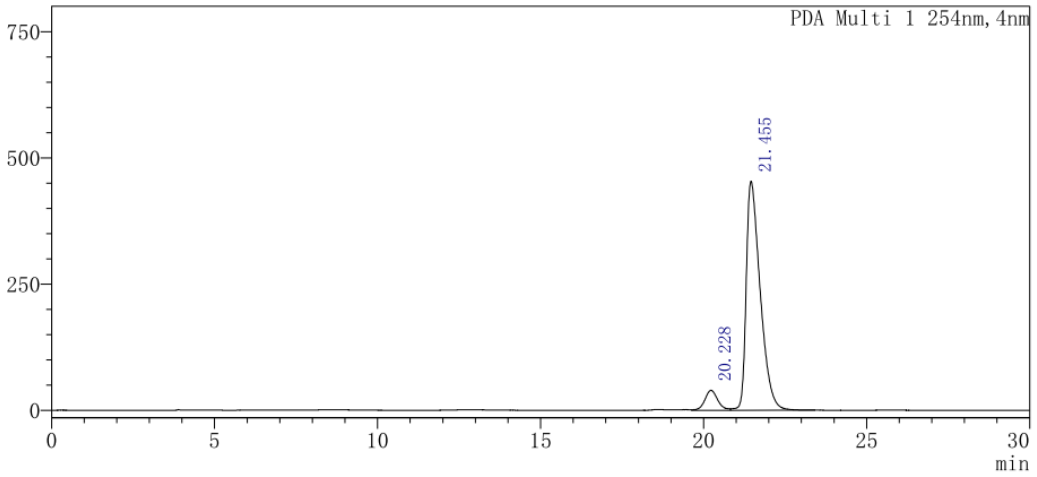
35

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 95/5, flow rate 0.8 mL/min. λ = 254 nm, t(minor) =20.2 min, t(major) = 21.5 min, 85% ee (92.6:7.4 er).



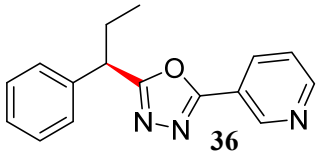
PDA Ch1 254nm

T	Hight	Area	Area%
20.320	150562	4001354	49.962
21.718	145412	4007389	50.038



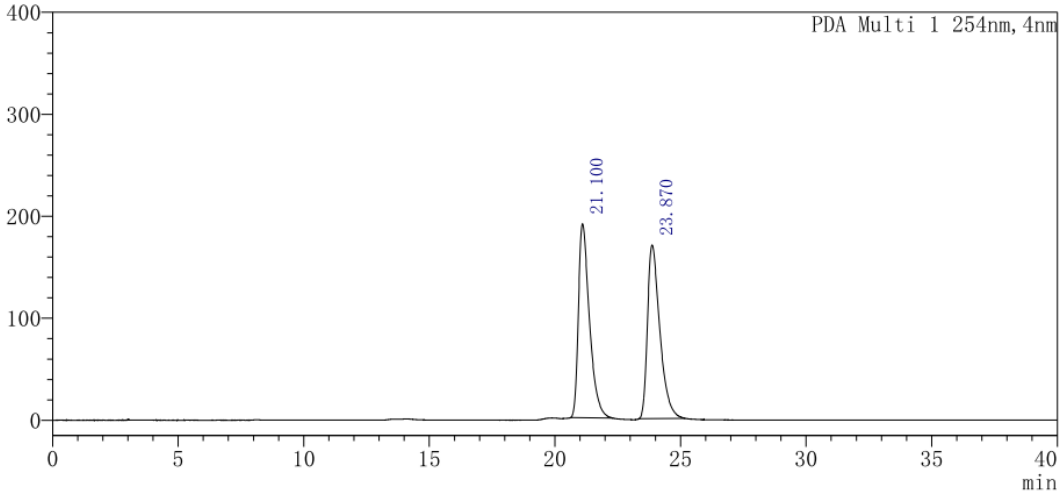
PDA Ch1 254nm

T	Hight	Area	Area%
20.228	39446	1080809	7.398
21.455	453471	13528951	92.602



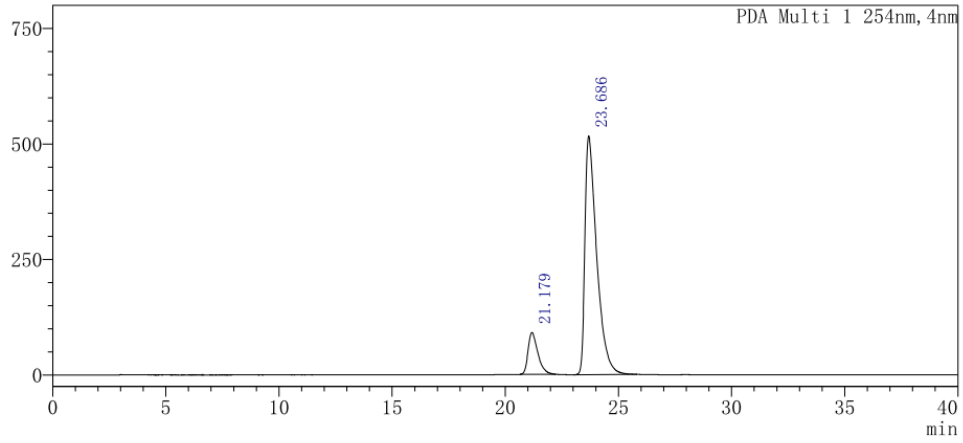
36

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min. λ = 254 nm, t(minor) =21.2 min, t(major) = 23.7 min, 75% ee (87.3:12.7 er).



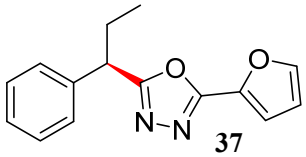
PDA Ch1 254nm

T	Hight	Area	Area%
21.100	190096	5799767	49.944
23.870	170187	5812743	50.056



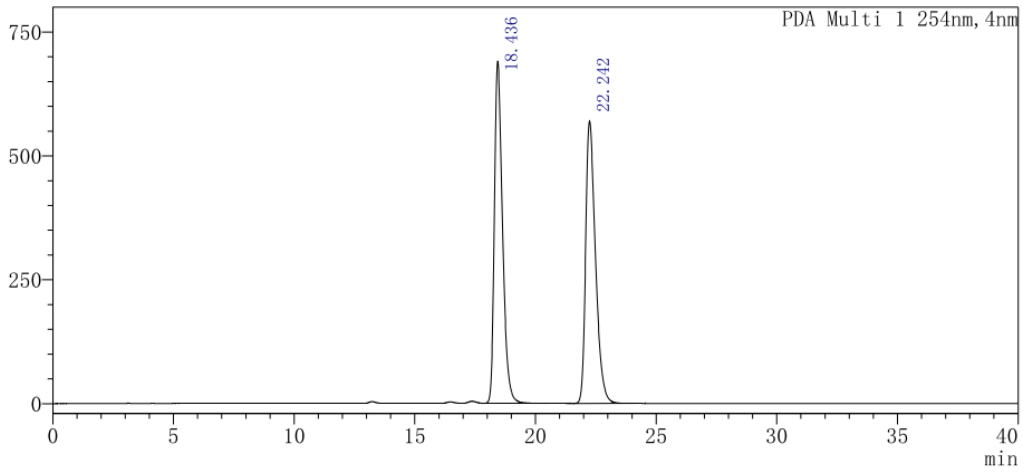
PDA Ch1 254nm

T	Hight	Area	Area%
21.179	90624	2722082	12.717
23.686	516608	18683799	87.283



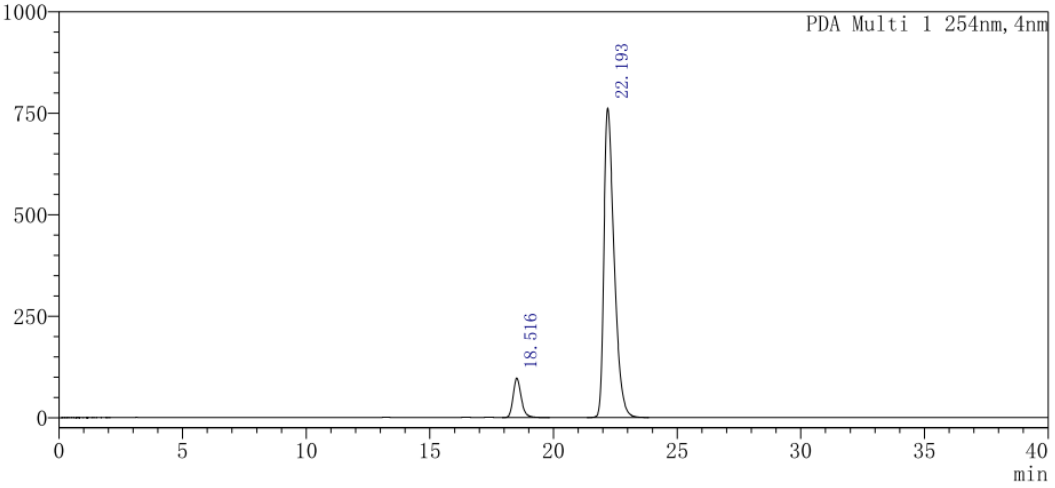
37

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. λ = 254 nm, t(minor) =18.5 min, t(major) = 22.2 min, 82% ee (90.8:9.2 er).



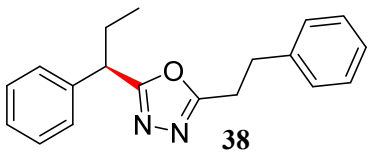
PDA Ch1 254nm

T	Hight	Area	Area%
18.436	690169	16178374	50.069
22.242	569653	16133566	49.931



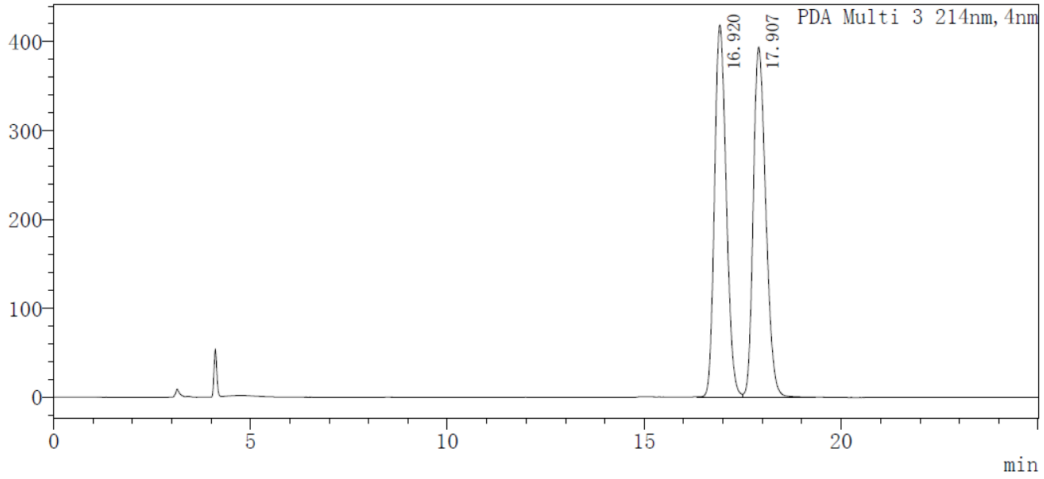
PDA Ch1 254nm

T	Hight	Area	Area%
18.516	97295	2211530	9.163
22.193	762329	21924217	90.837



38

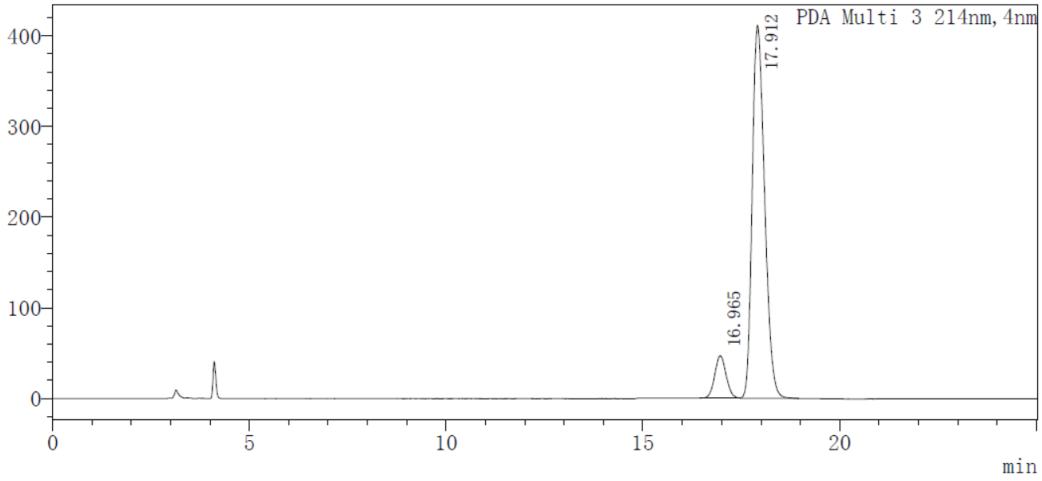
HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. λ = 214 nm, t(minor) = 17.0 min, t(major) = 17.9 min, 81% ee (90.5:9.5 er).



Peak Table

PDA Ch3 214nm

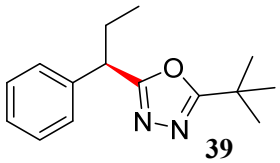
Peak#	Ret. Time	Area	Area%
1	16.920	8684482	49.919
2	17.907	8712652	50.081



Peak Table

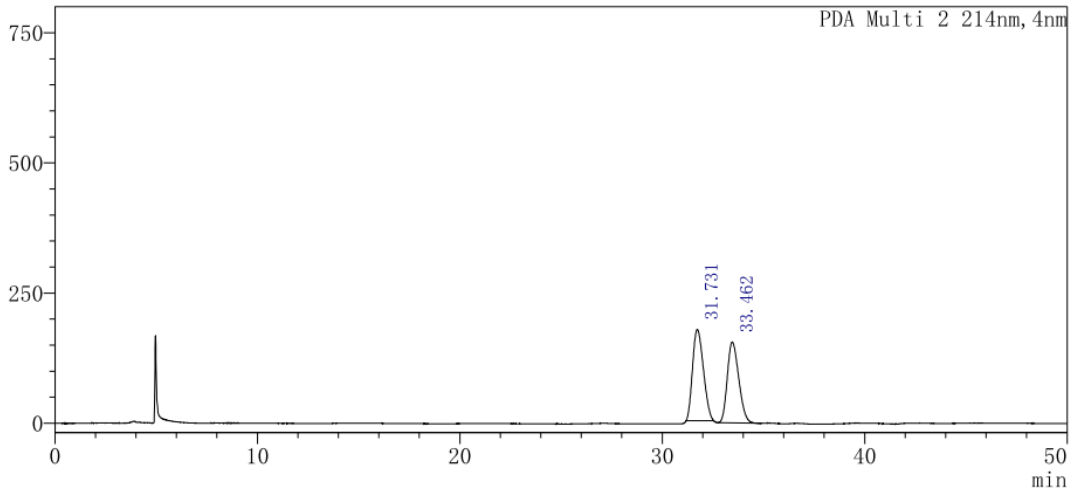
PDA Ch3 214nm

Peak#	Ret. Time	Area	Area%
1	16.965	941641	9.458
2	17.912	9014839	90.542



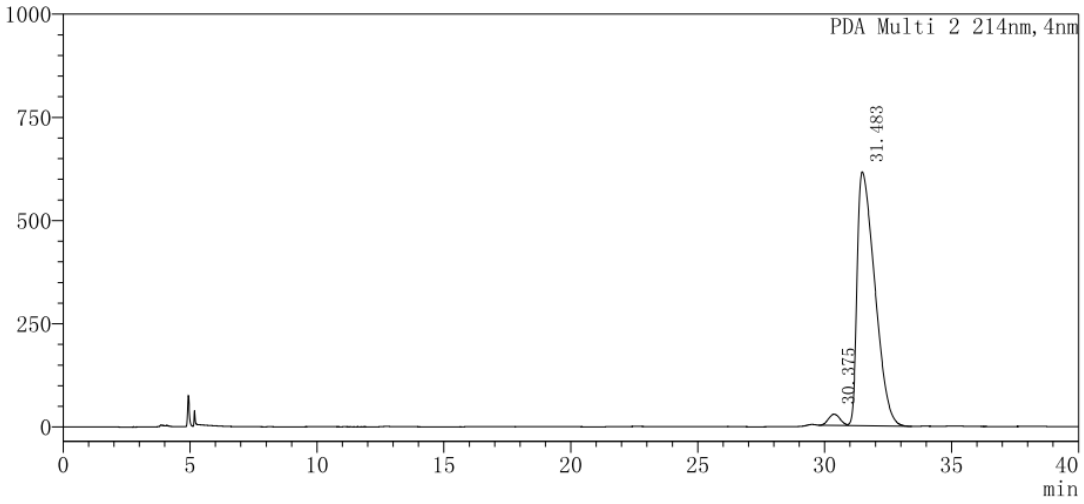
39

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 99/1, flow rate 0.8 mL/min. λ = 214 nm, t(minor) =31.1 min, t(major) = 32.3 min, 93% ee (96.5:3.5 er).



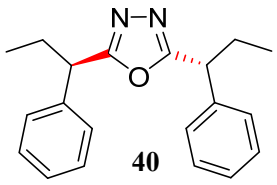
PDA Ch2 214nm

T	Hight	Area	Area%
31.731	175557	6534983	51.647
33.462	155307	6118306	48.353



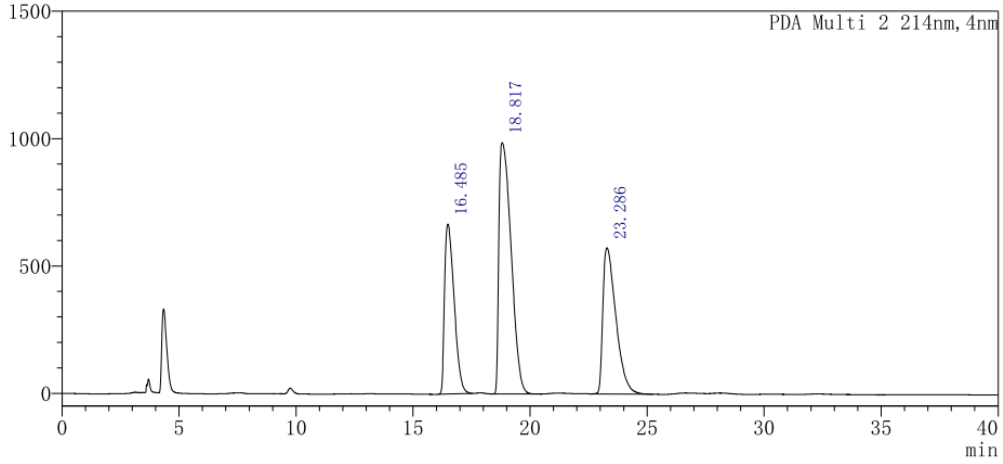
PDA Ch2 214nm

T	Hight	Area	Area%
31.084	41014	1409272	3.499
32.277	774652	38869853	96.501



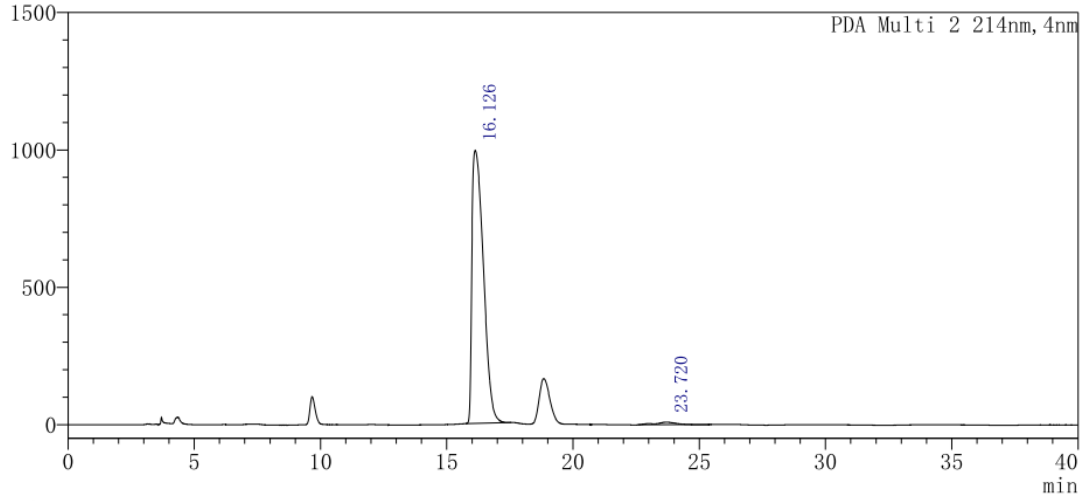
40

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 99/1, flow rate 1.0 mL/min. λ = 214 nm, t(major) = 16.1 min, t(minor) = 23.7 min, 97% ee (98.5:1.5 er).



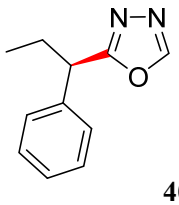
PDA Ch2 214nm

T	Hight	Area	Area%
16.485	666824	18927268	24.578
18.817	986269	35867089	46.575
23.286	573730	22214559	28.847



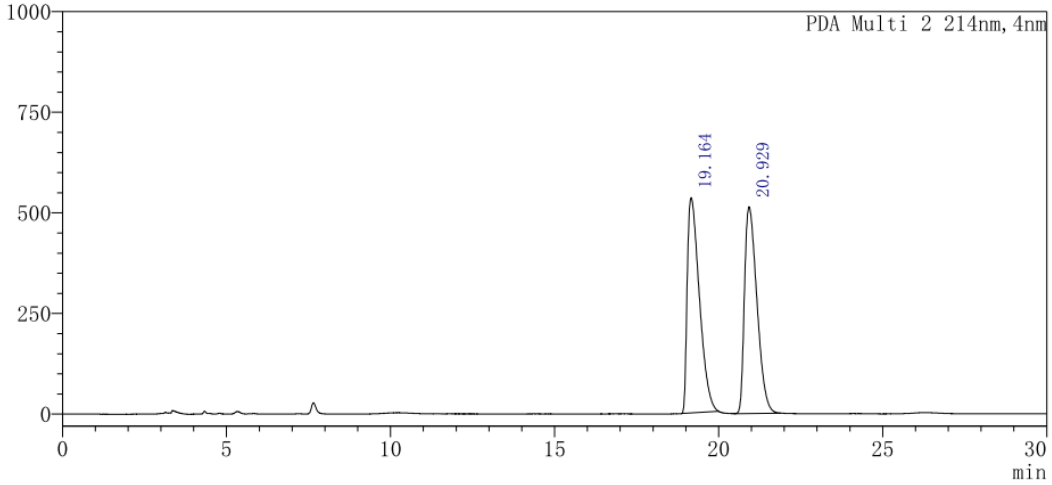
PDA Ch2 214nm

T	Hight	Area	Area%
16.126	993378	31454103	98.457
23.720	9230	492926	1.543



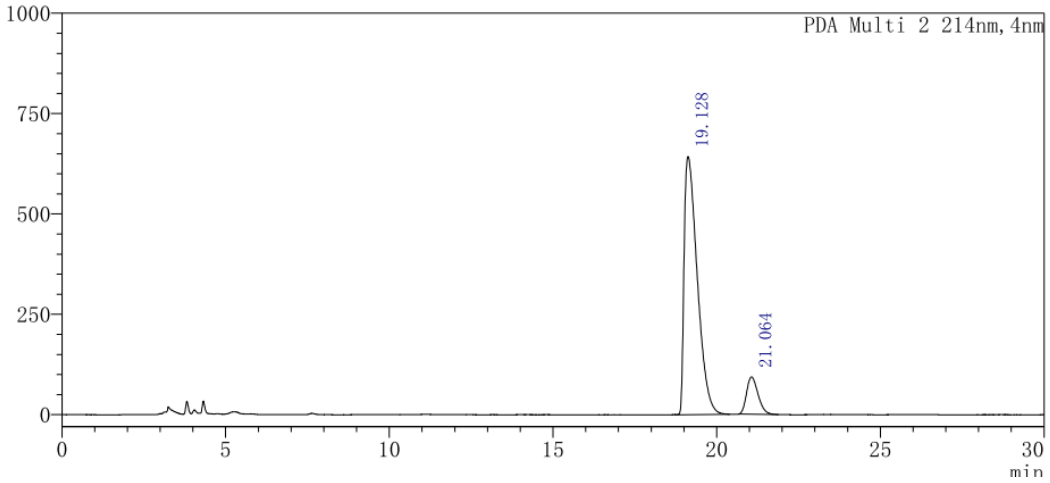
40 '

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 98/2, flow rate 1.0 mL/min. λ = 214 nm, t(major) = 19.1 min, t(minor) = 21.1 min, 77% ee (88.8:11.2 er).



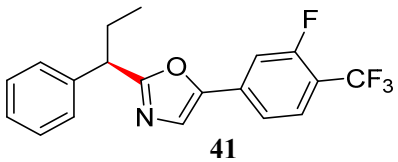
PDA Ch2 214nm

T	Hight	Area	Area%
19.164	534927	13780987	50.382
20.929	513488	13572156	49.618



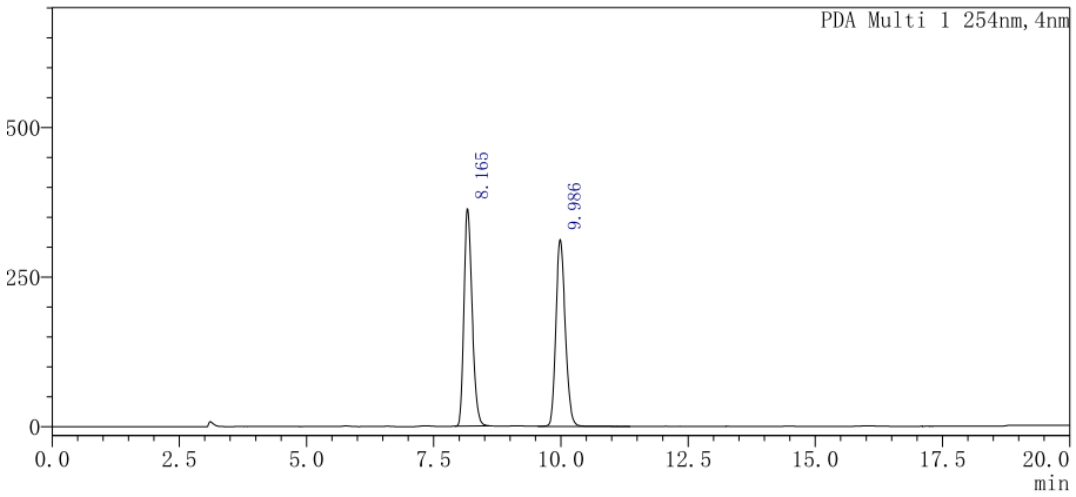
PDA Ch2 214nm

T	Hight	Area	Area%
19.128	642579	17963934	88.846
21.064	92727	2255345	11.154



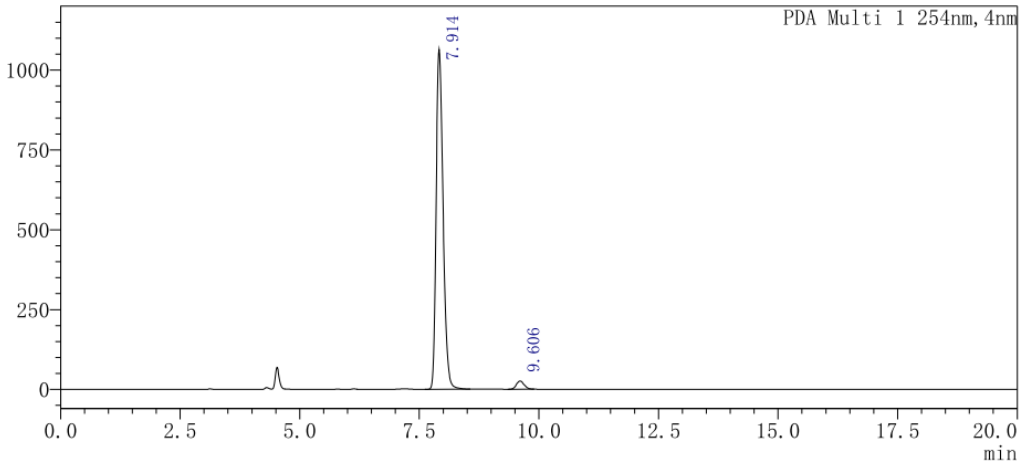
41

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. λ = 254 nm, t(major) = 7.9 min, t(minor) = 9.6 min, 95% ee (97.4:2.6 er).



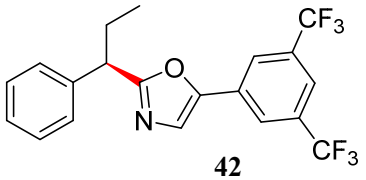
PDA Ch1 254nm

T	Hight	Area	Area%
8.165	362875	4064504	50.332
9.986	312214	4010828	49.668



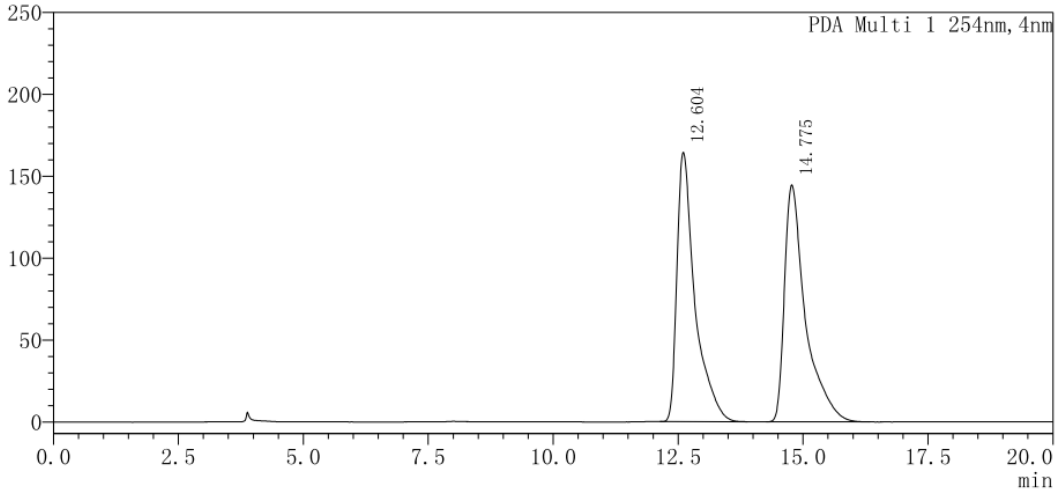
PDA Ch1 254nm

T	Hight	Area	Area%
7.914	1064419	10761943	97.361
9.606	25461	291738	2.639



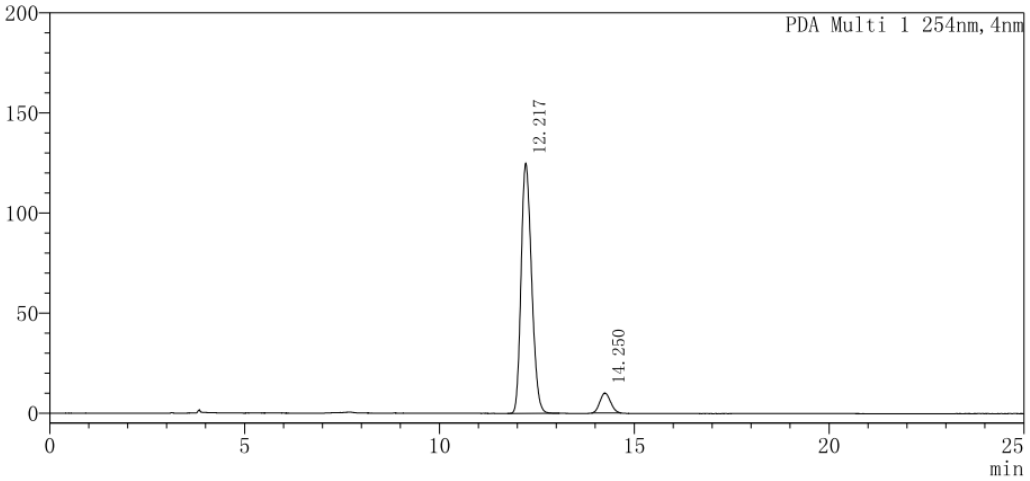
42

HPLC condition: Chiralcel OD-3, *n*-hexane/*i*-PrOH = 99/1, flow rate 1.0 mL/min. λ = 254 nm, t(major) = 12.2 min, t(minor) = 14.3 min, 85% ee (92.6:7.4 er).



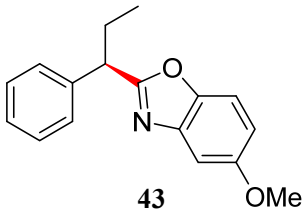
PDA Ch1 254nm

T	Hight	Area	Area%
12.604	164305	4109352	49.340
14.775	144600	4219278	50.660



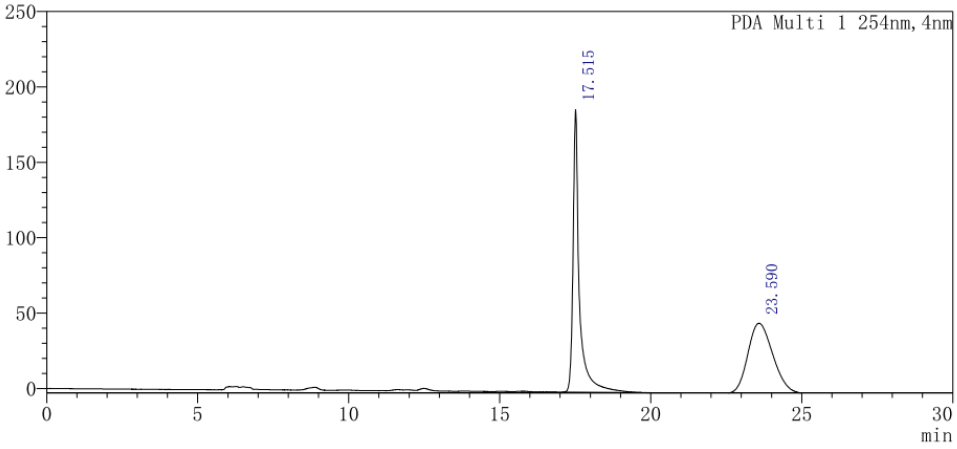
PDA Ch1 254nm

T	Hight	Area	Area%
12.217	124980	2343061	92.619
14.250	9916	186733	7.381



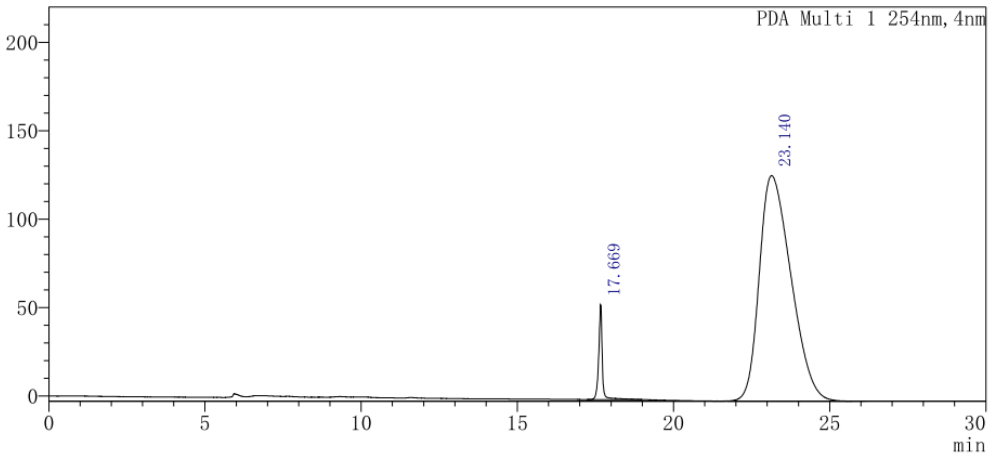
43

HPLC condition: Chiralcel OJ, *n*-hexane/*i*-PrOH = 93/7, flow rate 0.5 mL/min. λ = 254 nm, t(minor) = 17.7 min, t(major) = 23.1 min, 91% ee (95.4:4.6 er).



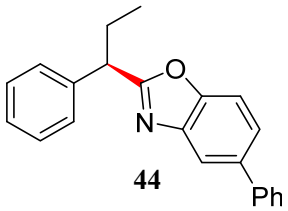
PDA Ch1 254nm

T	Hight	Area	Area%
17.515	187490	2748200	50.236
23.590	46798	2722390	49.764



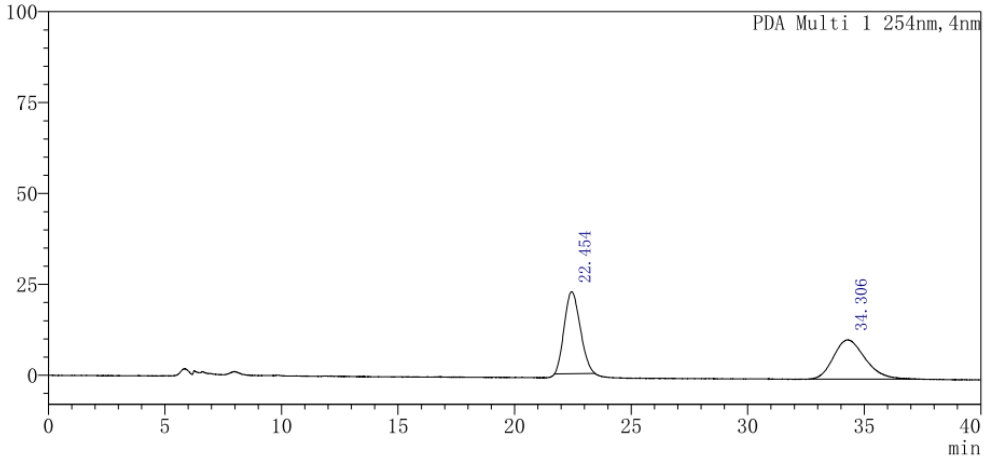
PDA Ch1 254nm

T	Hight	Area	Area%
17.669	53612	431137	4.557
23.140	127647	9029023	95.443



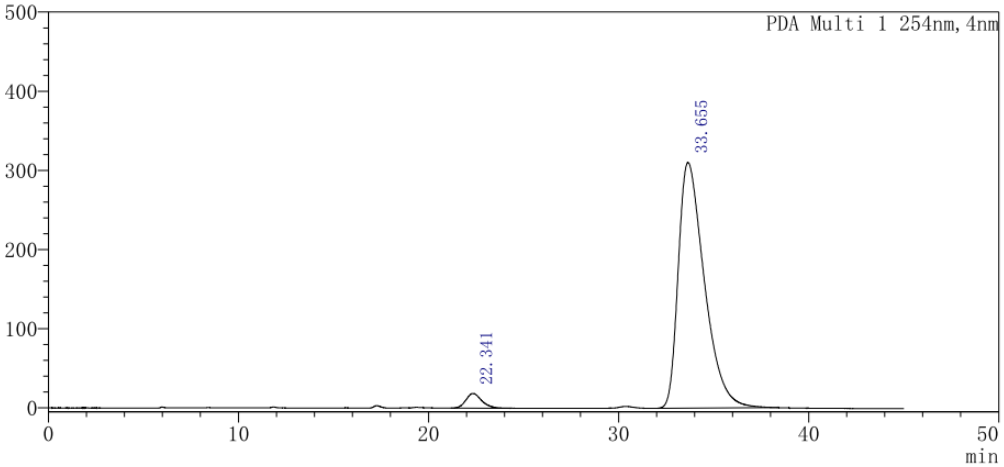
44

HPLC condition: Chiralcel OJ, *n*-hexane/*i*-PrOH = 93/7, flow rate 0.5 mL/min. λ = 254 nm, t(minor) = 22.3 min, t (major) = 33.7 min, 93% ee (96.5:3.5 er).



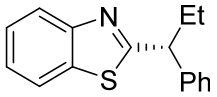
PDA Ch1 254nm

T	Hight	Area	Area%
22.454	22577	1077123	51.382
34.306	10792	1019198	48.618



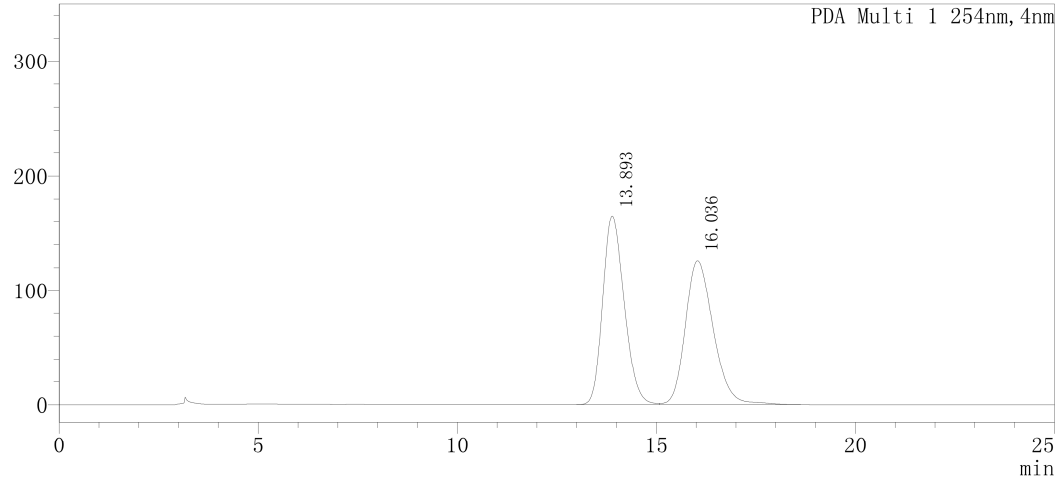
PDA Ch1 254nm

T	Hight	Area	Area%
22.341	18588	1076736	3.509
33.655	310310	29610644	96.491



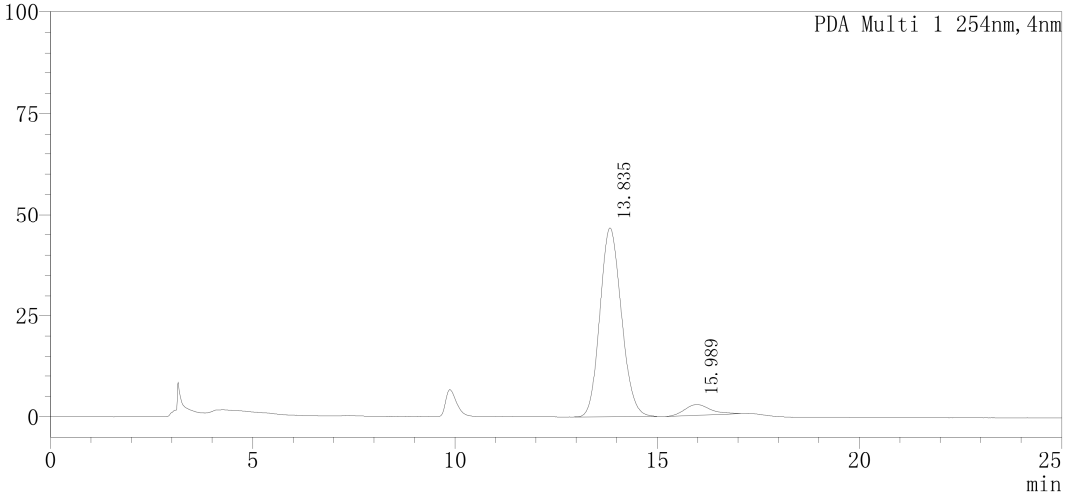
45

HPLC conditions: Chiralcel OJ, *n*-hexane/*i*-PrOH = 98/2, flow rate 1.0 mL/min. λ = 254 nm, t(major) = 13.8 min, t(mnior) = 16.0 min, 87% ee (93.7:6.3 er).



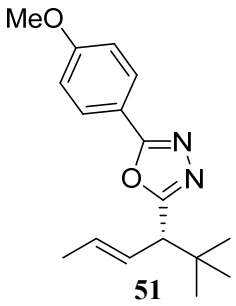
PDA Ch1 254nm

T	Hight	Area	Area%
13.893	164044	6183770	50.198
16.036	125813	6134955	49.802



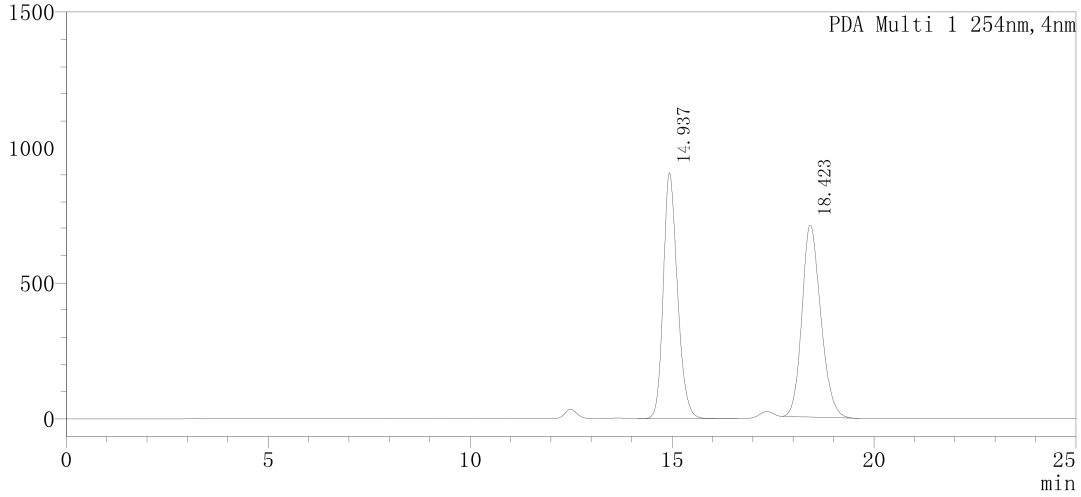
PDA Ch1 254nm

T	Hight	Area	Area%
13.835	46571	1723690	93.717
15.989	2615	115555	6.283



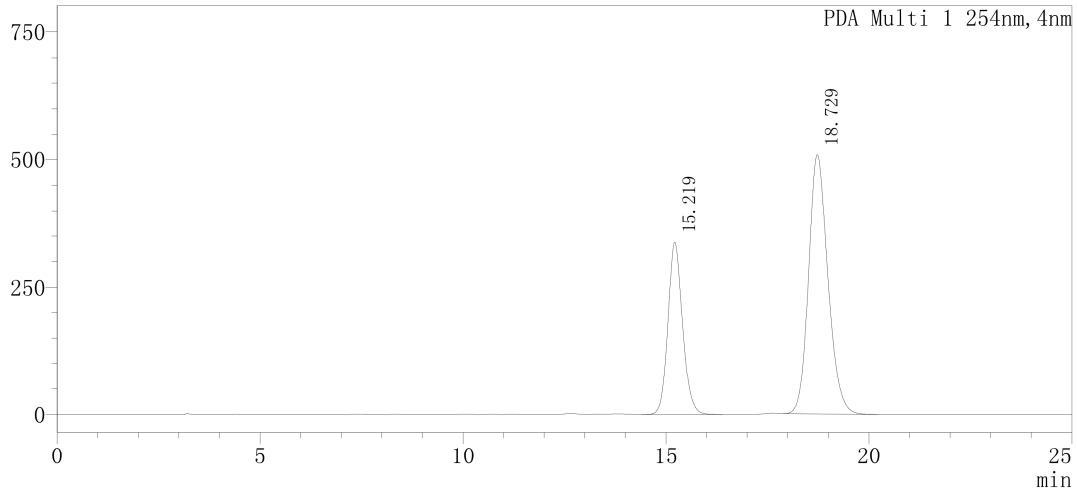
51

HPLC conditions: Chiralcel IA, *n*-hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 15.2 min, t(major) = 18.7 min, 33% ee (66.6:33.4 er).



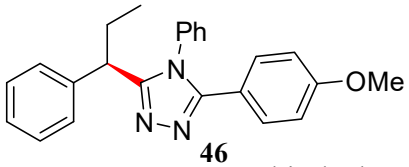
PDA Ch1 254nm

T	Hight	Area	Area%
14.937	907141	22165112	49.074
18.423	707483	23001538	50.926



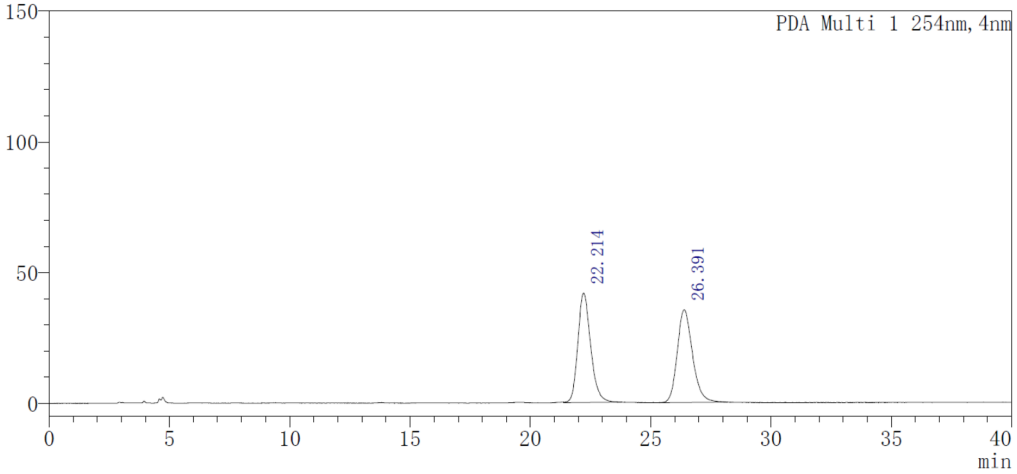
PDA Ch1 254nm

T	Hight	Area	Area%
15.219	337782	8442954	33.359
18.729	508446	16866153	66.641



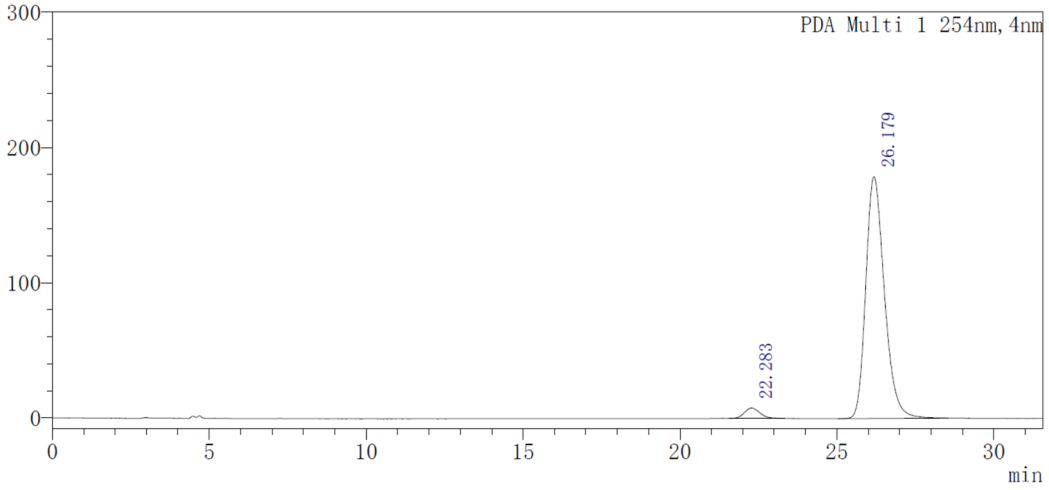
46

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 75/25, flow rate 1.0 mL/min. λ = 254 nm, t(minor) = 22.3 min, t(major) = 26.2 min, 93% ee (96.5:3.5 er).



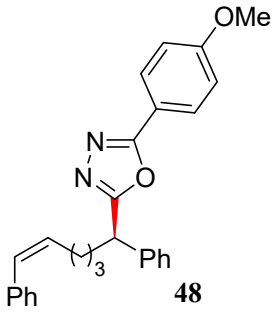
PDA Ch1 254nm

T	Hight	Area	Area%
22.214	41856	1555495	49.880
26.391	35443	1562982	50.120



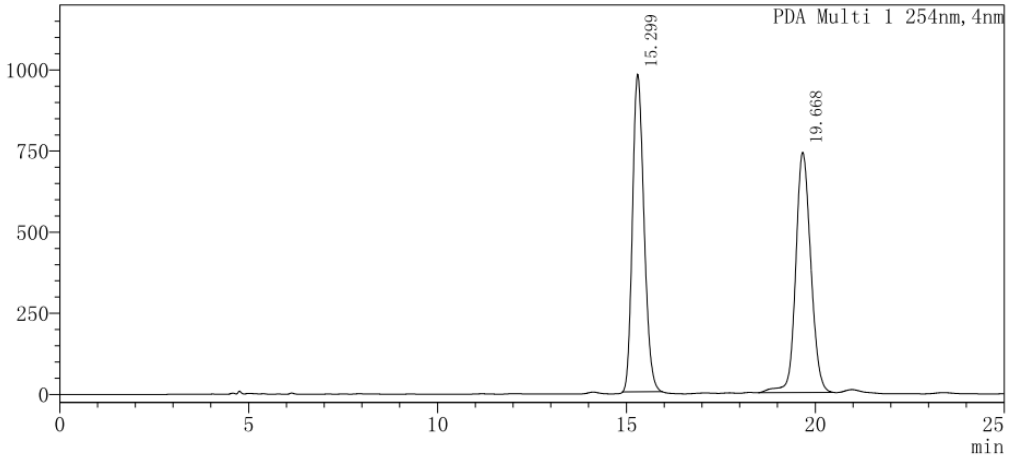
PDA Ch1 254nm

T	Hight	Area	Area%
22.283	7787	270888	3.532
26.179	178923	7398029	96.468



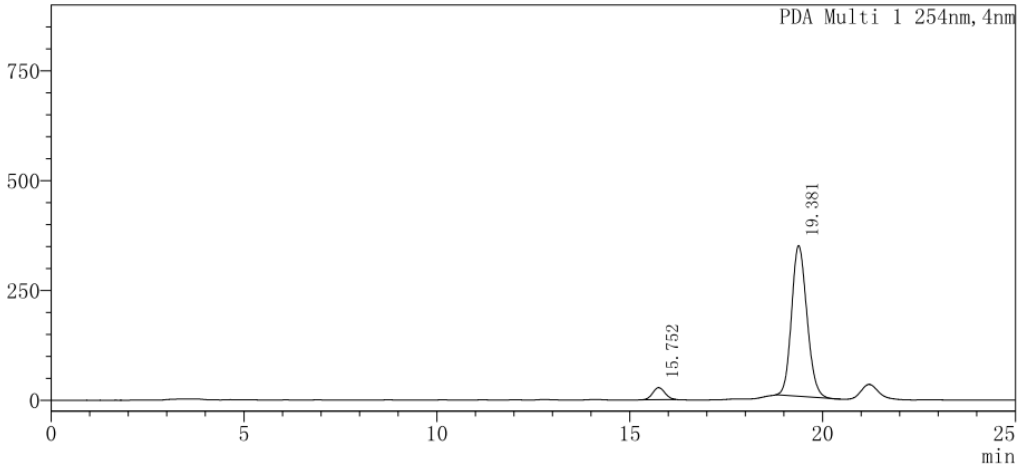
48

HPLC condition: Chiralcel IF, *n*-hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min. λ = 254 nm, t (minor) =15.8 min, t (major) = 19.4 min, 88% ee (94:6 er).



PDA Ch1 254nm

T	Hight	Area	Area%
15.299	979177	20800815	50.203
19.668	740828	20633003	49.797



PDA Ch1 254nm

T	Hight	Area	Area%
15.752	27456	605669	5.976
19.381	343686	9529948	94.024