Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Synthesis of Chiral Pyridine-oxazolines *via* Catalytic Asymmetric Heine Reaction of (*meso*)-N-(2-picolinoyl)-aziridines

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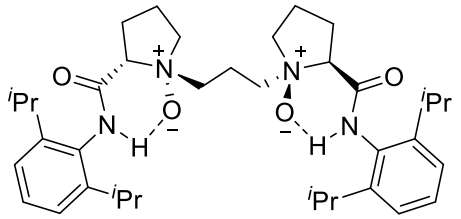
1. General information

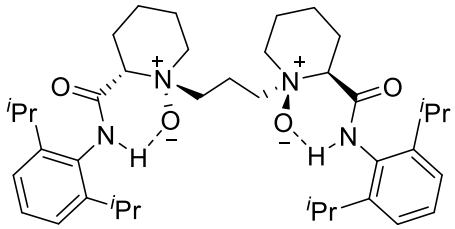
Unless otherwise noted, all commercially available compounds were used as provided without further purification. Enantiomeric excess (e.e.) were determined by HPLC or UPC^2 analysis using the corresponding commercial chiral column as stated in the experimental procedures at 35 ºC. Data for ^{1}H NMR are reported as follows: chemical shift in reference to residual $CHCl_3$ at 7.26 ppm (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, m = multiplet), coupling constants (*J*) are in Hertz (Hz), and integration. Data for ^{13}C{1H} NMR are reported in terms of chemical shift in reference to the $CDCl_3$ solvent signal (77.1 ppm) and are reported relative to the solvent residual peaks. ^{19}F{^{1}H} NMR spectra were collected on Bruker AMX-400 (376 MHz) with complete proton decoupling. HRMS was recorded on Thermo Scientific Q Exactive hybrid quadrupole-Orbitrap mass spectrometer (ESI Source). The chiral HPLC or UPC^2 methods were calibrated with the corresponding racemic mixtures. Optical rotations were measured on a Rudolph Autopol V automatic polarimeter and are reported as follows: $[\alpha]_D^T$ (*c* g/100 mL, in solvent). IR spectra were recorded on Bruker TENSOR II IR spectrophotometer. All catalytic reactions were run in dried glassware. THF and toluene were distilled from sodium benzophenone ketyl. $CHCl_3$, CH_2Cl_2, or CH_2ClCH_2Cl was distilled over CaH_2.

2. General procedure for *N,N'*-dioxide preparation.

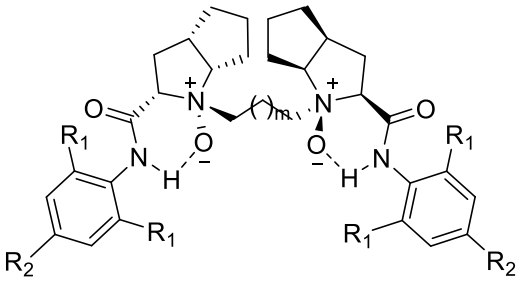
The following *N,N'*-dioxide ligands were synthesized by the same procedure in the literature.[1]



L_3-PrPr$_2$

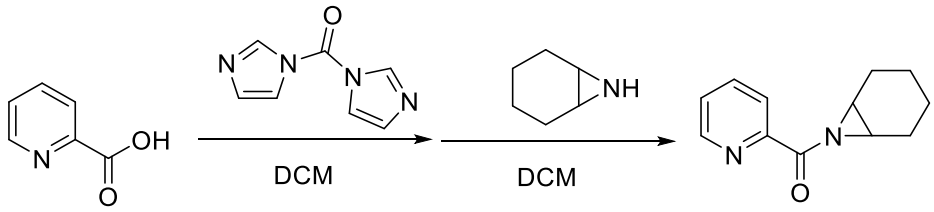


L_3-PiPr$_2$



L_3-RaPr$_2$: R_1=^{i}Pr, R_2=H, m=1
L_3-RaEt$_2$: R_1=Et, R_2=H, m=1
L_3-RaMe$_2$: R_1=Me, R_2=H, m=1
L_3-RaEt$_2$Me: R_1=Et, R_2=Me, m=1
L_2-RaPr$_2$: R_1=^{i}Pr, R_2=H, m=0

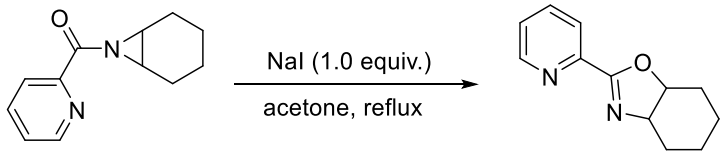
3.1 General procedure for the preparation of aziridine 1a:[2]



Acid (10 mmol) and di(1H-imidazol-1-yl)methanone (CDI) (1.62 g, 10 mmol) were dissolved in CH_2Cl_2 (40 mL), and the mixture was refluxed for 3 h. After cooling to 0 °C, 7-azabicyclo[4.1.0]heptane (0.97 g, 10 mmol) in CH_2Cl_2 (5 mL) was added slowly, then the reaction was stirred for 3 h at room temperature. Solvent was removed under reduced pressure, and the crude product was purified by flash column chromatography.

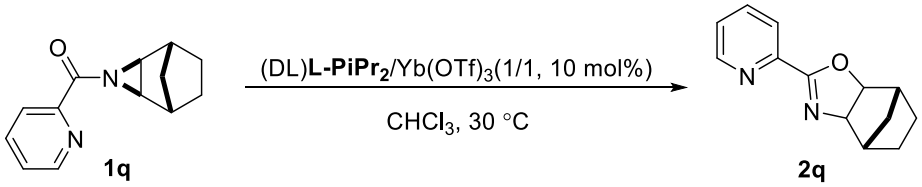
4. General procedure for the catalytic Henei reaction.

4.1 Preparation of the racemic products 2:



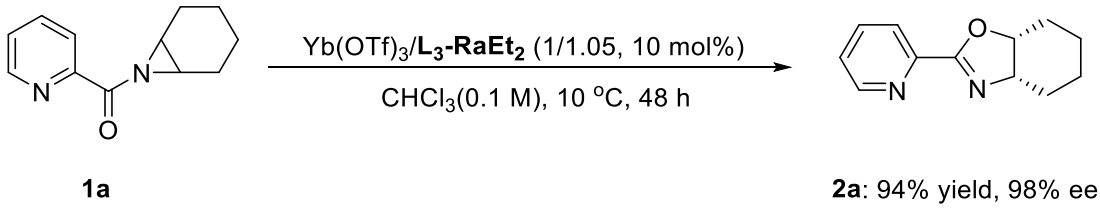
1a **2a**

An oven-dried test tube was charged with NaI (0.1 mmol), **1a** (0.10 mmol), acetone (1.0 mL). The flask was equipped with a reflux condenser and heated to 70 ℃. After stirring for 24 h, the reaction mixture was cooled to room temperature. The reaction mixture was subjected to flash column chromatography on silica gel and eluted with ethyl acetate to afford the corresponding product **2a** (colourless oil, 20.1 mg, 99% yield).



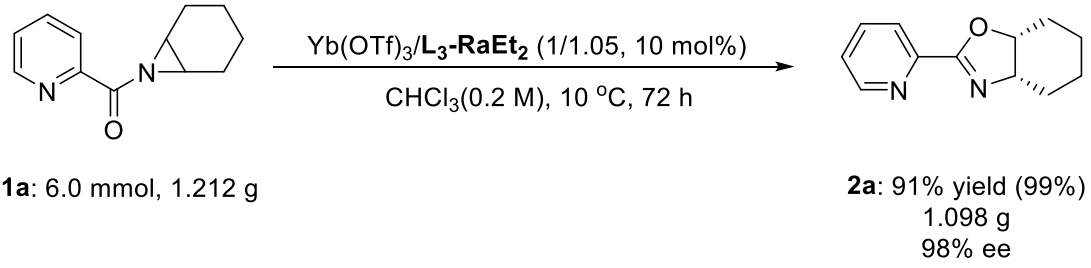
An oven-dried test tube was charged with the catalyst racemic **L-PrPr**$_2$/Yb(OTf)$_3$ (1:1, 10 mol %), **1q** (0.10 mmol), $CHCl_3$ (1.0 mL) under N_2 atmosphere. The resulted solution was stirred at 30 °C for 48 h. The reaction mixture was subjected to flash column chromatography on silica gel and eluted with ethyl acetate to afford the corresponding product **2q** (white solide, 19.3 mg, 90% yield).

4.2 Preparation of enantiomeric enriched product 2a:



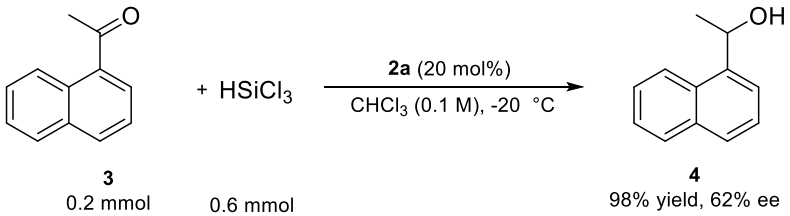
An oven-dried test tube was charged with the catalyst **L-RaEt**$_2$/Yb(OTf)$_3$ (1.05:1, 10 mol %), **1a** (0.10 mmol), $CHCl_3$ (1.0 mL) under N_2 atmosphere. The resulted solution was stirred at 10 °C for 72 h. The reaction mixture was subjected to flash column chromatography on silica gel and eluted with ethyl acetate to afford the corresponding product **2a** (colourless oil, 19.0 mg, 94% yield, 98% ee).

5. Experimental procedure for the gram-scale synthesis of 2a.



An oven-dried round-bottom flask was charged with the catalyst **L-RaEt**$_2$/Yb(OTf)$_3$ (1:1.05, 10 mol %), **1a** (6.0 mmol, 1.212 g), $CHCl_3$ (30 mL) under N_2 atmosphere. The resulted solution was stirred at 10 °C for 72 h. The reaction mixture concentrated under reduced pressure and the crude product was subjected to flash column chromatography on silica gel and eluted with ethyl acetate to afford the corresponding product **2a** (1.098 g, 91% yield, 98% ee) and recovery **1a** (104 mg, 9% yield).

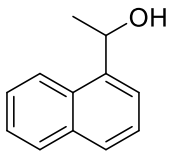
6. The hydrosilyation of 1-(naphthalen-2-yl)ethenone .



Trichlorosilane (60 μL, 0.6 mmol, 3.0 equiv.) was slowly added dropwise to a solution of **2a** (8.1 mg, 0.04 mmol) and the ketone **3** (0.20 mmol, 1.0 equiv.) in $CHCl_3$ (2.0 mL) at -20 °C. The reaction mixture was stirred at -20 °C for 24 h, then saturated aqueous $NaHCO_3$ (1 mL) was added to quench the reaction. The mixture was extracted with CH_2Cl_2 (3 × 10 mL) and the combined organic extracts were dried over $MgSO_4$. Concentration in vacuo followed by flash chromatography on silica gel with CH_2Cl_2 afforded white solid **4**， The data of known compound **4** were consistent with those reported in literatures.[3]

1-(naphthalen-2-yl)ethan-1-ol

1-(naphthalen-1-yl)ethan-1-ol



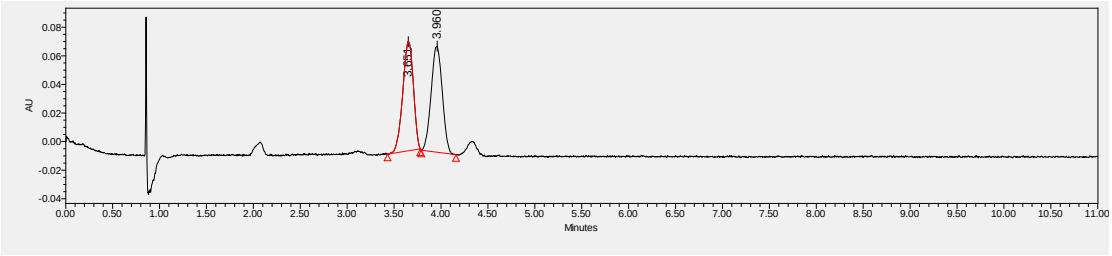
4

Following the typical procedure, white solid **4** was isolated in 98% yield (33.2 mg) and 62% ee.

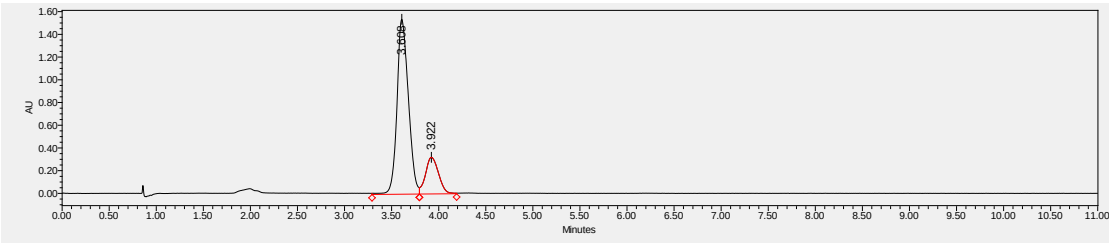
UPC² (chiral IB-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) =3.9 min, t (major) = 3.6 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 7.90 – 7.68 (m, 4H), 7.46 (ddt, *J* = 10.7, 6.9, 3.3 Hz, 3H), 5.02 (q, *J* = 6.4 Hz, 1H), 1.55 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$). δ143.2, 133.3, 132.9, 128.3, 127.9, 127.7, 126.2, 125.8, 123.8, 123.8, 25.2.

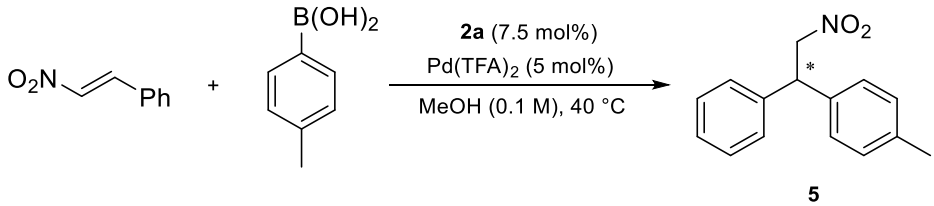


	Retention Time	Area	% Area
1	3.651	601357	49.52
2	3.960	613048	50.48



	Retention Time	Area	% Area
1	3.608	12977719	80.69
2	3.922	3106040	19.31

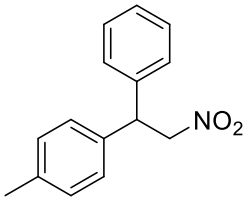
7. The conjugation addition reaction of nitroalken and boronic acid.



0.2 mmol 0.3 mmol **5** 81% yield, 75% ee

$Pd(TFA)_2$ (1.7 mg, 0.01 mmol, 5 mol%) and **2a** (1.5 mg, 0.015 mmol, 7.5 mol%) were weighted in air and placed in an oven-dried test tube. MeOH (0.5 mL, without special treatment) was added and the solution was stirred at room temperature for 0.5 h to afford the catalyst solution. Nitroalken (0.20 mmol, 1.0 equiv) and arylboronic acid (0.3 mmol, 1.5 equiv.) were added to the catalyst solution. The wall of the flask was rinsed with MeOH (0.5 mL). The tube was equipped with a reflux condenser and heated to 40 °C. After stirring for 24 h, the reaction mixture was cooled to room temperature, and the solvent was removed by rotary evaporation. The residue was purified by preparative TLC on silica gel (petroleum ether/EtOAc = 20/1) to give the product. The racemic products were prepared using 2,2'-bispyridine as ligand. The data of the known compound **5** were consistent with those reported in literatures.[4]

1-methyl-4-(2-nitro-1-phenylethyl)benzene



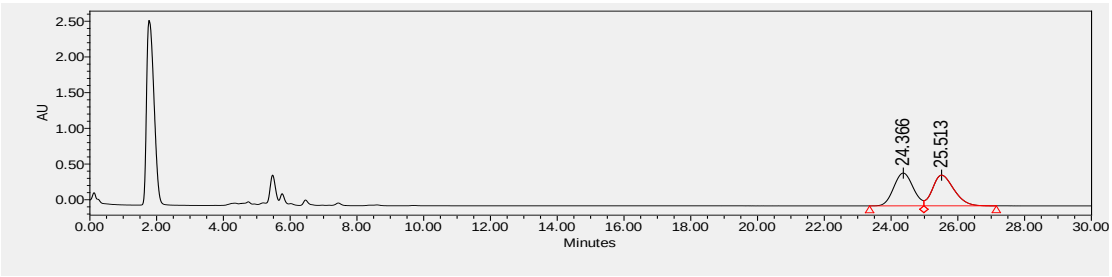
8

Following the typical procedure, **6** was isolated as oil in 81% yield (40.0 mg) and 75% ee.

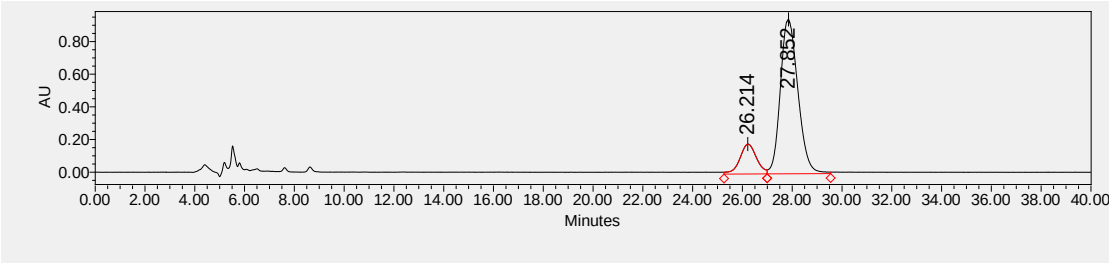
HPLC (chiral ODH column), hexane/*i*-PrOH = 60/40, flow rate 0.7 mL/min, λ = 210 nm, retention time: t (minor) = 26.2 min, t (major) = 27.8 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 7.35 – 7.28 (m, 2H), 7.23 (t, *J* = 8.4 Hz, 3H), 7.12 (s, 4H), 4.95 (d, *J* = 8.7 Hz, 2H), 4.90 – 4.81 (m, 1H), 2.30 (s, 3H).[4]

^{13}C NMR (100 MHz, $CDCl_3$). δ 139.4, 137.3, 136.2, 129.7, 129.0, 127.6, 79.3, 48.6, 21.0.[4]



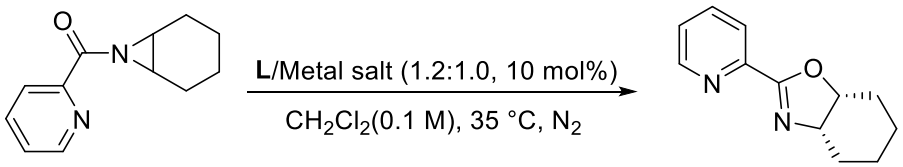
	Retention Time	Area	% Area
1	24.366	18516092	49.27
2	25.513	19065778	50.73



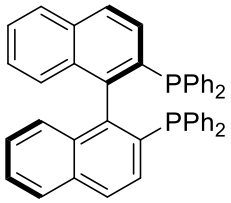
	Retention Time	Area	% Area
1	26.214	6223373	12.43
2	27.852	43853686	87.57

8. Optimization of the reaction conditions

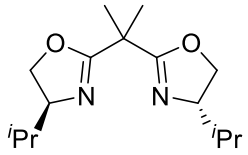
Table S1. Screening of the classical Lewis acid catalysts.



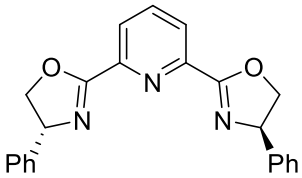
1a **2a**



L1'



L2'

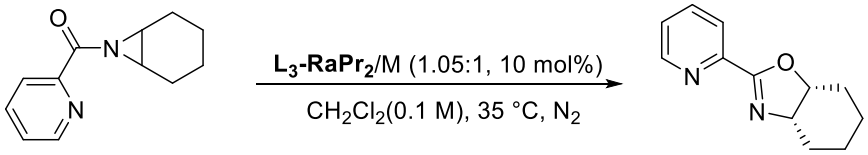


L3'

Entry[a]	**L**	Metal salt	Yield (%)	Ee (%)[b]
1	**L1**'	$Pd(OTf)_2$	Messy	-
2	**L2**'	$Cu(OTf)_2$	50	0
3	**L3**'	$Cu(OTf)_2$	67	0
4	**L3**'	$Yb(OTf)_3$	69	7

[a]Unless otherwise noted, the reactions were carried out with **1a** (0.1 mmol), metal salt/L (1:1.2, 10 mol%) in CH_2Cl_2 (0.1 M) at 35 ℃ under a N_2 atmosphere for 36 h. [b]Ee values were determined by UPC[2].

Table S2. Screening of metal salts.

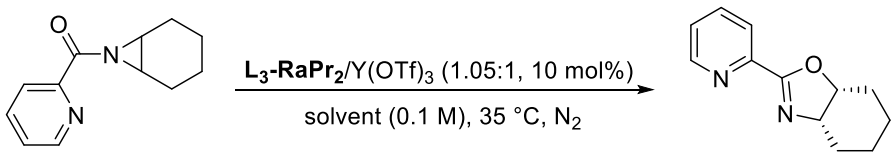


1a **2a**

Entry[a]	Metal salt	Yield (%)	Ee (%)[b]
1	$Y(OTf)_3$	98	76
2	$La(OTf)_3$	60	33
3	$Yb(OTf)_3$	93	48
4	$Mg(OTf)_2$	16	-37
5	$Fe(OTf)_2$	73	-6
6	$Co(OTf)_2$	messy	-
7	$Ni(OTf)_2$	messy	-
8	$Cu(OTf)_2$	messy	-
9	$Sc(OTf)_3$	54	-36

[a]Unless otherwise noted, the reactions were carried out with **1a** (0.1 mmol), metal salt/**L3-RaPr2** (1:1.05, 10 mol%) in CH_2Cl_2 (0.1 M) at 35 ℃ under a N_2 atmosphere for 36 h. [b]Ee values were determined by UPC[2].

Table S3. Screening of solvents.

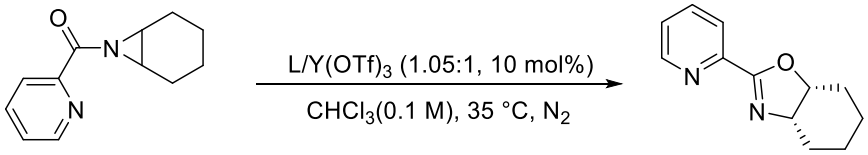


1a **2a**

Entry[a]	Solvent	Yield (%)	Ee (%)[b]
1	THF	55	60
2	PhMe	59	60
3	DCE	72	72
4	$CHCl_3$	80	88

[a]Unless otherwise noted, the reactions were carried out with **1a** (0.1 mmol), $Y(OTf)_3$/**L3-RaPr2** (1:1.05, 10 mol%) in solvent (0.1 M) at 35 ℃ for 36 h. [b]Ee values were determined by UPC[2].

Table S4. Screening of chiral ligand.

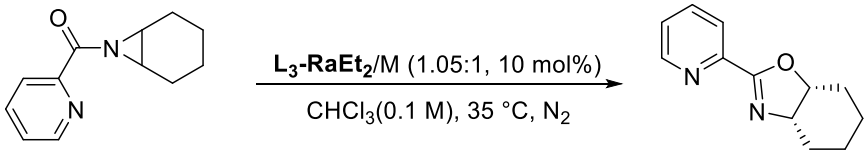


1a **2a**

Entry[a]	Ligand	Yield (%)	Ee (%)[b]
1	**L_3-PrPr$_2$**	84	89
2	**L_3-PiPr$_2$**	85	88
3	**L_3-RaPr$_2$**	80	88
4	**L_2-RaPr$_2$**	73	69
6	**L_3-RaMe$_2$**	85	90
7	**L_3-RaEt$_2$**	80	94
8	**L_3-RaEt$_2$Me**	80	87

[a]Unless otherwise noted, the reactions were carried out with **1a** (0.1 mmol), Y(OTf)$_3$/ligand (1:1.05, 10 mol%) in $CHCl_3$ (0.1 M) at 35 ℃ under a N_2 atmosphere for 36 h. [b]Ee values were determined by UPC^2.

Table S5. Rescreening of metal salts.

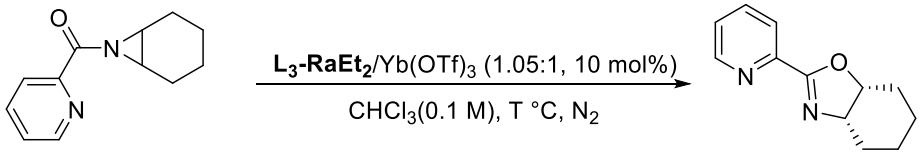


1a **2a**

Entry[a]	Metal salt	Yield (%)	Ee (%)[b]
1	$Y(OTf)_3$	85	94
2	$La(OTf)_3$	37	19
3	$Ce(OTf)_3$	49	68
4	$Pr(OTf)_3$	87	85
5	$Nd(OTf)_3$	95	92
6	$Sm(OTf)_3$	97	85
7	$Eu(OTf)_3$	97	92
8	$Gd(OTf)_3$	97	93
9	$Tb(OTf)_3$	97	94
10	$Dy(OTf)_3$	95	93
11	$Ho(OTf)_3$	94	94
12	$Er(OTf)_3$	95	95
13	$Tm(OTf)_3$	98	95
14	$Yb(OTf)_3$	98	96
15	$Lu(OTf)_3$	97	94

[a]Unless otherwise noted, the reactions were carried out with **1a** (0.1 mmol), metal salt/**L_3-RaEt2** (1:1.05, 10 mol%) in $CHCl_3$ (0.1 M) at 35 ℃ under a N_2 atmosphere for 36 h. [b]Ee values were determined by UPC[2].

Table S6. Screening of temperature.

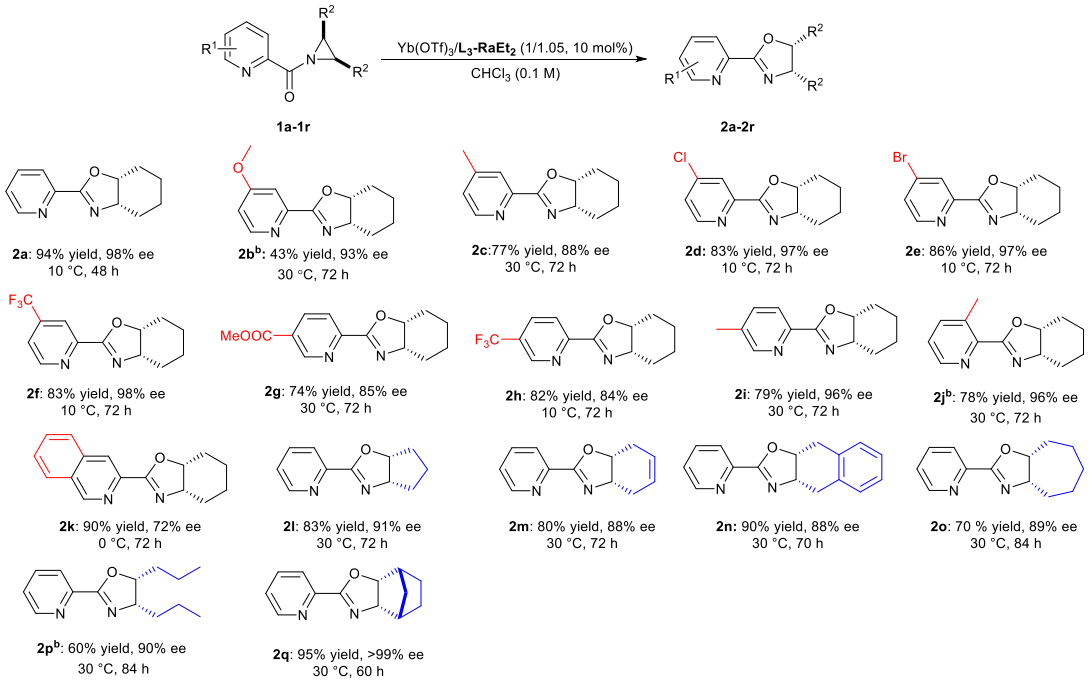


1a **2a**

Entry[a]	T (°C)	Yield (%)	Ee (%)[b]
1	35	98	96
2	30	97	96
3	20	95	96
4	**10**	**94**	**98**
5	0	94	97

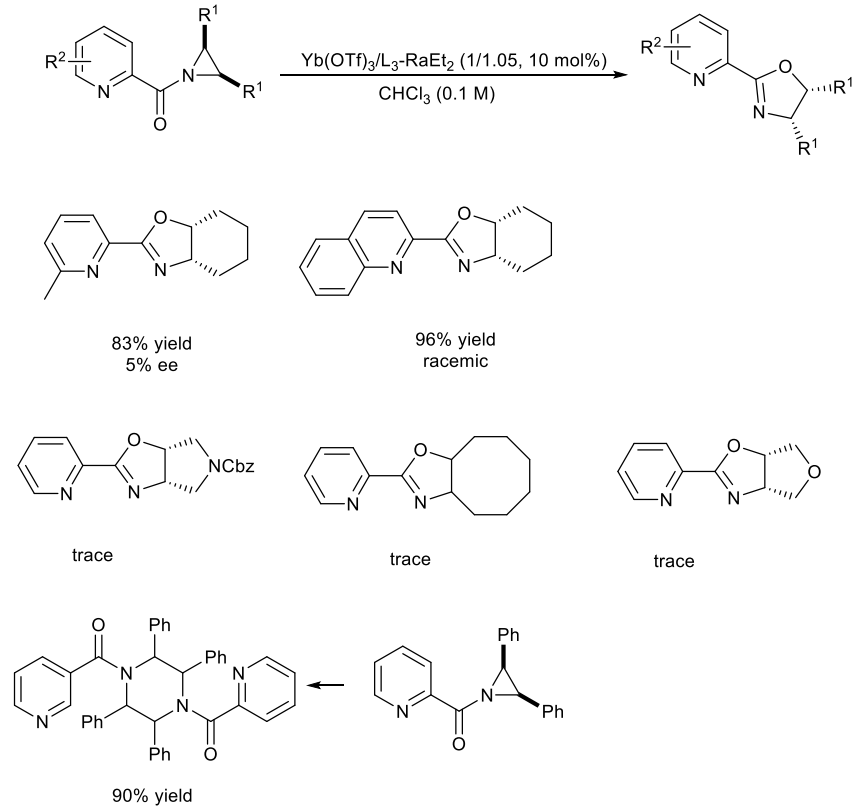
[a]Unless otherwise noted, the reactions were carried out with **1a** (0.1 mmol), $Yb(OTf)_3$/**L_3-RaEt$_2$** (1:1.05, 10 mol%) in $CHCl_3$ (0.1 M) at T °C under a N_2 atmosphere for 48 h. [b]Ee values were determined by UPC[2].

9. The list of substrates scope.



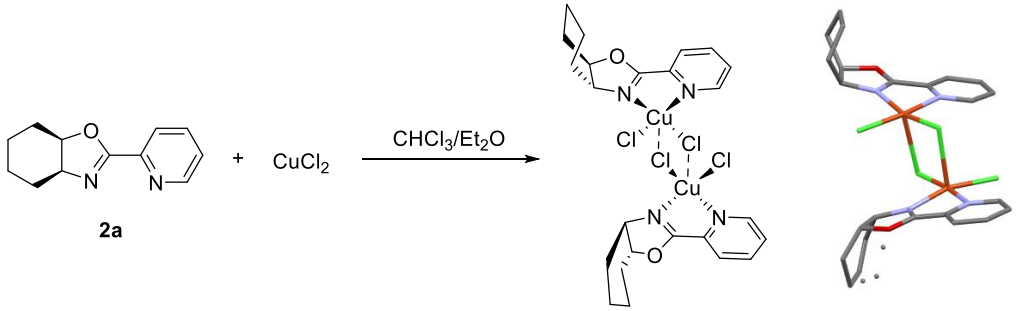
b: **L3-PrEt2** was used.

10. Unsuccessful substrate scope.

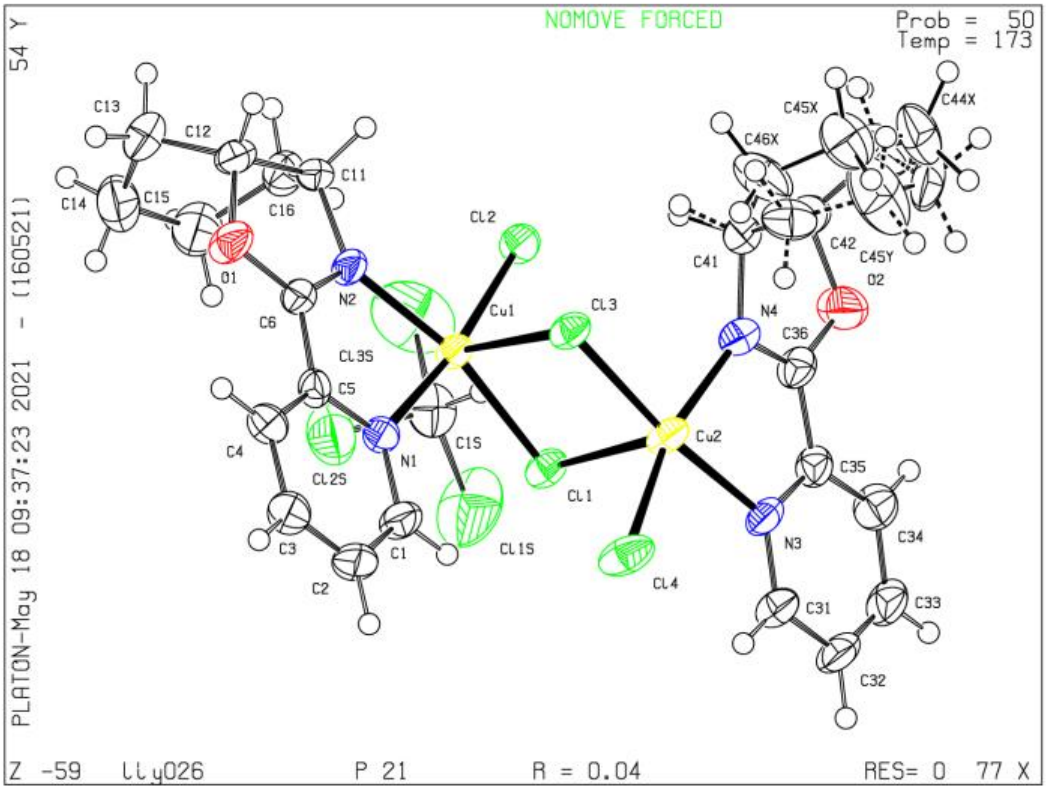


11. Determination of absolute configurations of the compounds.

Crystals suitable for the X-ray crystal structure analysis were obtained from a solution of compound **2a** and $CuCl_2$ in $CHCl_3$ (ca. 0.1 mL) and Et_2O (0.3 mL) at r.t. The green crystal in block-shape, with approximate dimensions of 0.062 × 0.087 × 0.336 mm^3, was selected and mounted for the single-crystal X-ray diffraction. The data set was collected by Bruker D8 Venture Photon II diffractometer at 173(2)K equipped with micro-focus Mo radiation source (K_α = 0.71073Å). Applied with face-indexed numerical absorption correction, the structure solution was solved and refinement was processed by SHELXTL (version 6.14) and OLEX 2.3 program package, The structure was analyzed by ADDSYM routine implemented in PLATON suite and no higher symmetry was suggested. CCDC 2084388 contains the supplementary crystallographic data which can be obtained free of charge from The Cambrige Crystallographic Data Centere via www.ccdc.cam.ac.uk/data_request/cif



CCDC 2084388



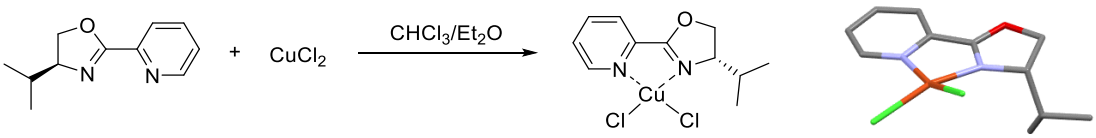
Crystallographic Data for $C_{25}H_{29}Cl_7Cu_2N_4O_2$

Formula	$C_{25}H_{29}Cl_7Cu_2N_4O_2$
Formula mass (amu)	792.75
Space group	P 2_1
a (Å)	11.4615(5)
b (Å)	9.9184(4)
c (Å)	14.1599(7)
α (deg)	90
β (deg)	104.423(2)
γ (deg)	90
V (Å^3)	1558.96(12)
Z	2
λ (Å)	0.71073
T (K)	173(2) K
ρ_{calcd} (g cm^{-3})	1.689
μ (mm^{-1})	1.996
Transmission factors	0.731,0.957
$2\theta_{max}$ (deg)	28.342
No. of unique data, including $F_o^2 < 0$	7585
No. of unique data, with $F_o^2 > 2\sigma(F_o^2)$	6103
No. of variables	389
$R(F)$ for $F_o^2 > 2\sigma(F_o^2)$ [a]	0.0358
$R_w(F_o^2)$ [b]	0.0847
Goodness of fit	1.117

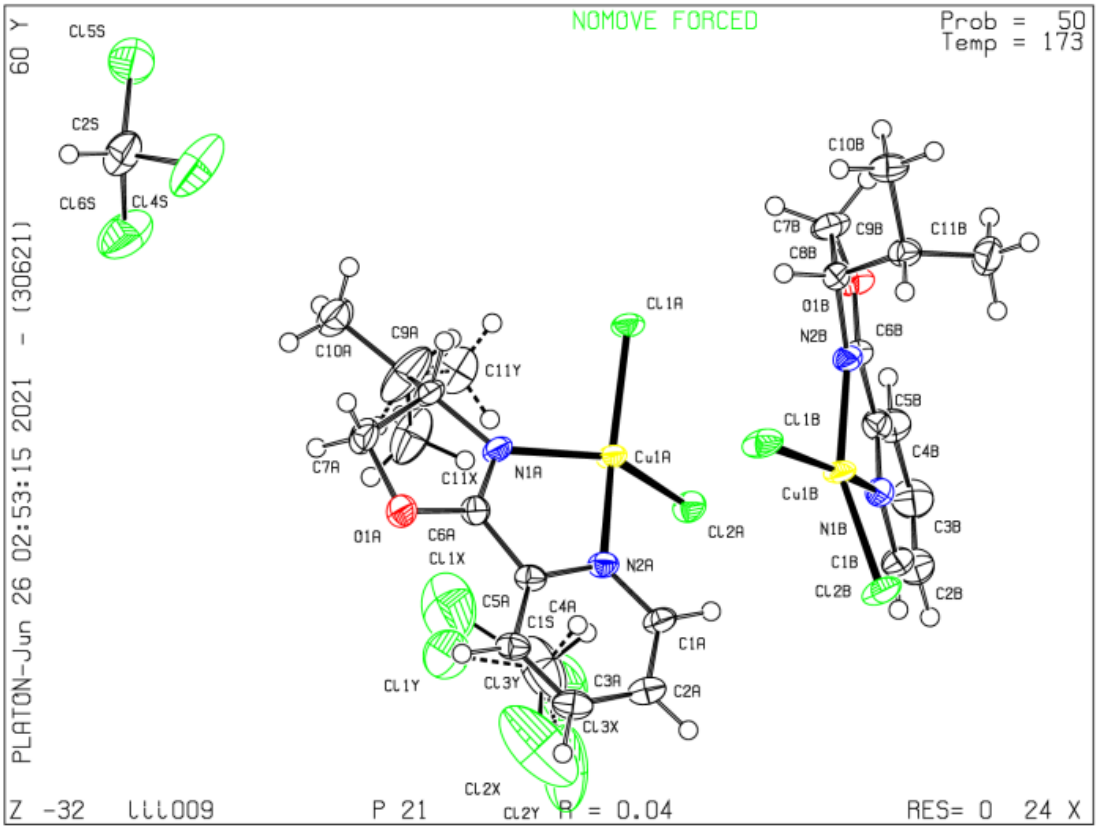
[a] $R(F) = \sum||F_o| - |F_c|| / \sum|F_o|$.

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

Crystals suitable for the X-ray crystal structure analysis were obtained from a solution of optical pure (*S*)-***i*PrProx** and $CuCl_2$ in $CHCl_3$ (ca. 0.1 mL) and Et_2O (0.3 mL) at r.t. The green crystal in flake-shape, with approximate dimensions of 0.110 × 0.307 × 0.538 mm^3, was selected and mounted for the single-crystal X-ray diffraction. The data set was collected by Bruker D8 Venture Photon II diffractometer at 173(2)K equipped with micro-focus Cu radiation source (K_α = 1.54178Å). Applied with face-indexed numerical absorption correction, the structure solution was solved and refinement was processed by SHELXTL (version 6.14) and OLEX 2.3 program package, The value observed herein is indicative of racemic twinning and was accommodated during the refinement (using the SHELXL TWIN instruction). In this case, the relatively large standard uncertainty indicates that the structural data alone should not be used to confirm absolute stereochemistry, but should be used in conjunction with the established stereochemistry of the precursor compound. The structure was analyzed by ADDSYM routine implemented in PLATON suite and no higher symmetry was suggested. CCDC 2094394 contains the supplementary crystallographic data which can be obtained free of charge from The Cambrige Crystallographic Data Centere via www.ccdc.cam.ac.uk/data_request/cif



CCDC 2094394



Crystallographic Data for $C_{24}H_{30}Cl_{10}Cu_2N_4O_2$.

Formula	$C_{24}H_{30}Cl_{10}Cu_2N_4O_2$.
Formula mass (amu)	888.10
Space group	P 2_1
a (Å)	13.8291(4)
b (Å)	9.7078(3)
c (Å)	14.4138(4)
α (deg)	90
β (deg)	116.083(1)
γ (deg)	90
V (Å^3)	1737.98(9)
Z	2
λ (Å)	1.54178
T (K)	173(2) K
ρ_{calcd} (g cm^{-3})	1.697
μ (mm^{-1})	8.840
Transmission factors	0.080,0.581
$2\theta_{max}$ (deg)	74.533
No. of unique data, including $F_o^2 < 0$	6833
No. of unique data, with $F_o^2 > 2\sigma(F_o^2)$	6661
No. of variables	423
$R(F)$ for $F_o^2 > 2\sigma(F_o^2)$ [a]	0.0402
$R_w(F_o^2)$ [b]	0.1037
Goodness of fit	1.063

[a] $R(F) = \sum||F_o| - |F_c|| / \sum|F_o|$.

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

12. References.

1): (a) Wen, Y. H.; Huang, X.; Huang, J. L.; Xiong, Y.; Qin, B.; Feng, X. M. *Synlett* **2005**, 2445. (b) Yu, Z. P.; Liu, X. H.; Dong, Z. H.; Xie, M. S.; Feng, X. M. *Angew. Chem. Int. Ed***. 2008**, *47*, 1308. (c) Zheng, K.; Qin, B.; Liu, X. H.; Feng, X. M. *J. Org. Chem***. 2007**, *72*, 8478. (d) Zhang, X.; Chen, D. H.; Liu, X. H.; Feng, X. M. *J. Org. Chem.* **2007**, *72*, 5227. (e) Zhou, X.; Shang, D. J.; Zhang, Q.; Lin, L. L.; Liu, X. H.; Feng, X. M. *Org. Lett.* **2009**, *11*, 1401.

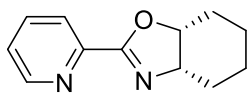
2): Li, D.; Wang, L.-Q.; Zhu, H.-Y.; Bai, L.-T.; Yang, Y.-L.; Zhang, M.-M.; Yang, D.-X.; Wang, R. *Org. Lett.*, **2019**, *21*, 4717.

3): Malkov, A. V.; Liddon, A. J. P. S.; Ramírez-López, P.; Bendov, L.; D. Haigh, Kočovský, P. *Angew. Chem. Int. Ed.* **2006**, *45*, 1432.

4): He, Q.; Xie, F.; Fu, G.-H.; Quan, M.; Shen, C.-Ren; Yang, G.-Q.; Gridnev, I. D.; Zhang, W.-B. *Org. Lett.*, **2015**, *17*, 2250.

13. Characterization of the products.

2a: (3aS,7aR)-2-(pyridin-2-yl)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazole



2a

Following the typical procedure, colourless oil **2a** was isolated in 94% yield (19.0 mg) and 98% ee. $[\alpha]^{25}_{D}$ = –322.5 (*c* = 0.4 in CH_2Cl_2).

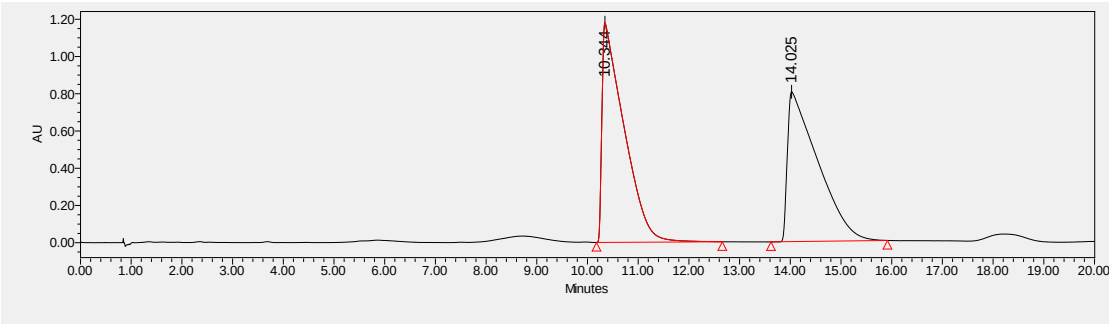
UPC² (chiral OX-3 column), CO_2/MeOH = 93/7, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 12.2 min, t (major) = 13.6 min.

^{1}H NMR (400 MHz, Chloroform-*d*). 8.65 (d, *J* = 4.7 Hz, 1H), 8.00 (d, *J* = 7.9 Hz, 1H), 7.71 (td, *J* = 7.8, 1.8 Hz, 1H), 7.35 – 7.28 (m, 1H), 4.72 (dt, *J* = 8.3, 5.2 Hz, 1H), 4.18 – 4.07 (m, 1H), 2.03 – 1.74 (m, 4H), 1.57 – 1.45 (m, 3H), 1.43 – 1.31 (m, 2H).

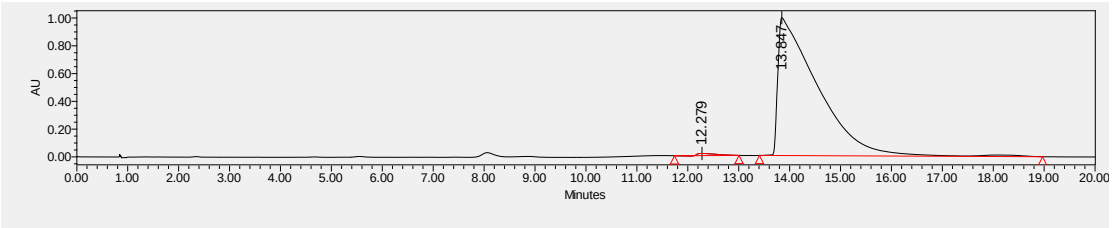
^{13}C NMR (100 MHz, $CDCl_3$). δ 162.5, 148.7, 146.0, 135.5, 124.4, 122.7, 78.7, 62.7, 26.6, 25.0, 18.8, 18.0.

ESI-HRMS: calcd for $C_{12}H_{15}N_2O^+$ ($[M + H]^+$) = 203.1179, found 203.1171.

IR (neat): 2933, 2858, 1661, 1596, 1567, 1525, 1452, 1308, 1263, 1225, 1126, 1083, 1033 cm^{-1}.

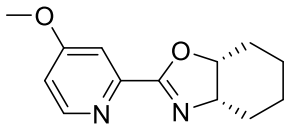


	Retention Time	Area	% Area
1	10.344	35468993	51.50
2	14.025	33403934	48.50



	Retention Time	Area	% Area
1	12.279	443383	0.81
2	13.847	54039714	99.19

2b: (3aS,7aR)-2-(4-methoxypyridin-2-yl)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazole



2b

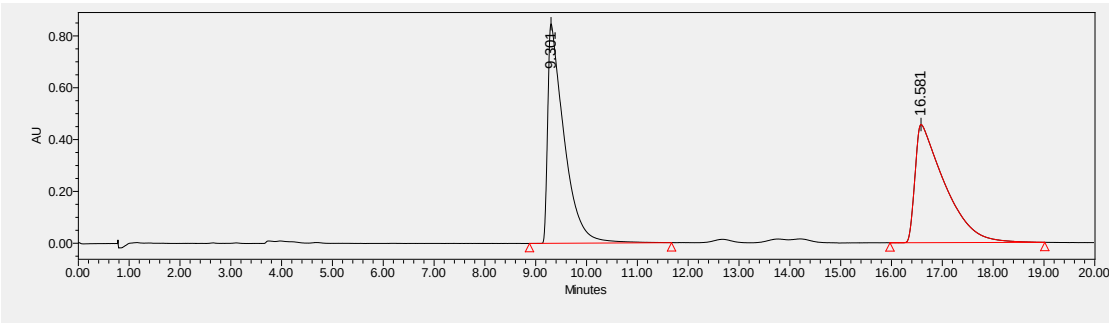
Following the typical procedure, colourless oil **2b** was isolated in 43% yield (10.0 mg) and 93% ee. $[\alpha]^{25}_{D}$ = –68.7 (*c* = 0.29 in CH_2Cl_2). **UPC²** (chiral OX-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 9.5 min, t (major) = 16.2 min.

¹H NMR (400 MHz, Chloroform-*d*). δ 8.51 (d, *J* = 5.7 Hz, 1H), 7.60 (d, *J* = 2.4 Hz, 1H), 6.91 (dd, *J* = 5.6, 2.5 Hz, 1H), 4.81 – 4.74 (m, 1H), 4.21 – 4.14 (m, 1H), 3.91 (s, 3H), 2.13 – 1.85 (m, 4H), 1.66 – 1.33 (m, 6H).

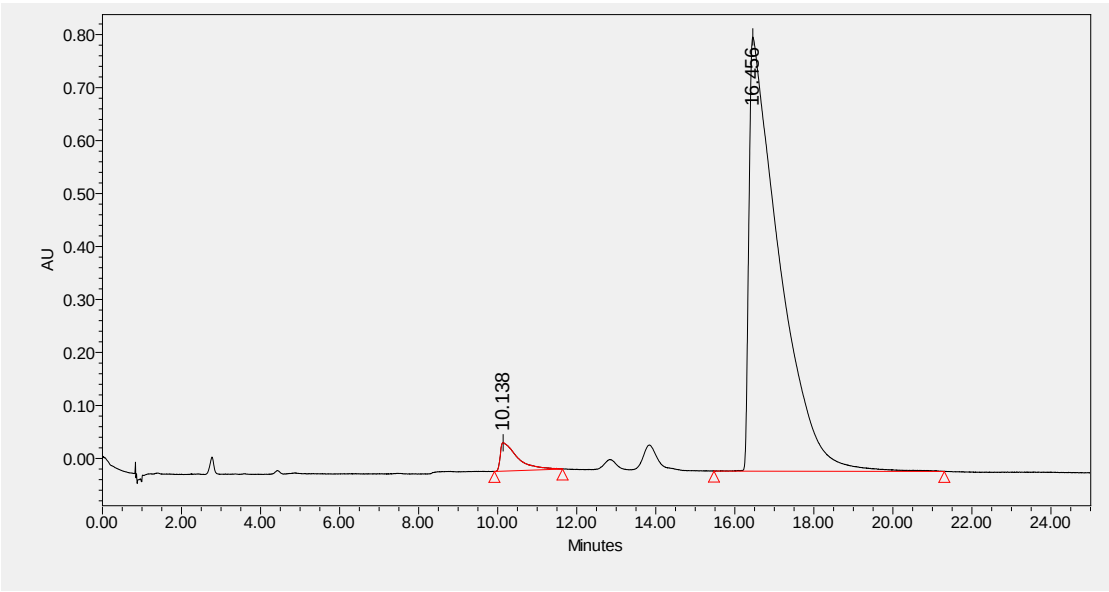
¹³C NMR (100 MHz, $CDCl_3$). δ 166.1, 163.6, 150.8, 148.7, 112.5, 109.0, 79.8, 63.6, 55.4, 27.7, 25.9, 19.8, 19.0.

ESI-HRMS: calcd for $C_{13}H_{17}N_2O_2^+$ ([M + H]⁺) = 233.1285, found 233.1285.

IR (neat): 2933, 2858, 1668, 1554, 1519, 1457, 1417, 1334, 1262, 1134, 1089, 978, 774, 690 cm^{-1}.

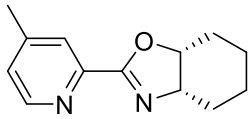


	Retention Time	Area	% Area
1	9.301	18951478	50.69
2	16.581	18437454	49.31



	Retention Time	Area	% Area
1	10.138	1629035	3.61
2	16.456	43461425	96.39

2c: (3aS,7aR)-2-(4-methylpyridin-2-yl)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazole



2c

Following the typical procedure, colourless oil **2c** was isolated in 77% yield (16.5 mg) and 88% ee. $[\alpha]^{25}_{D}$ = –142.1 (*c* = 0.204 in CH_2Cl_2).

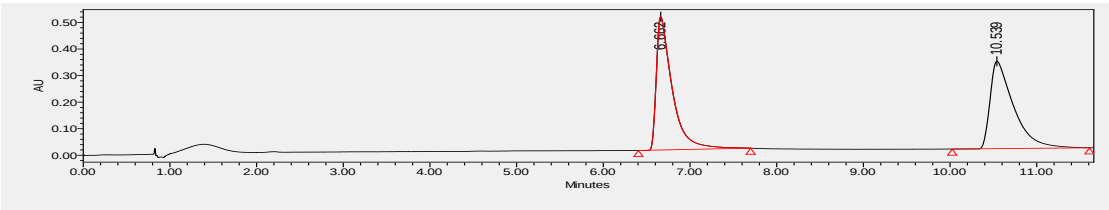
UPC² (chiral OX-3 column), CO_2/MeOH = 93/7, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 6.8 min, t (major) = 10.3 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 8.49 (d, *J* = 5.0 Hz, 1H), 7.83 (s, 1H), 7.14 (d, *J* = 5.8 Hz, 1H), 4.71 (dt, *J* = 8.2, 5.1 Hz, 1H), 4.19 – 3.99 (m, 1H), 2.33 (s, 3H), 2.03 – 1.69 (m, 4H), 1.58 – 1.24 (m, 4H).

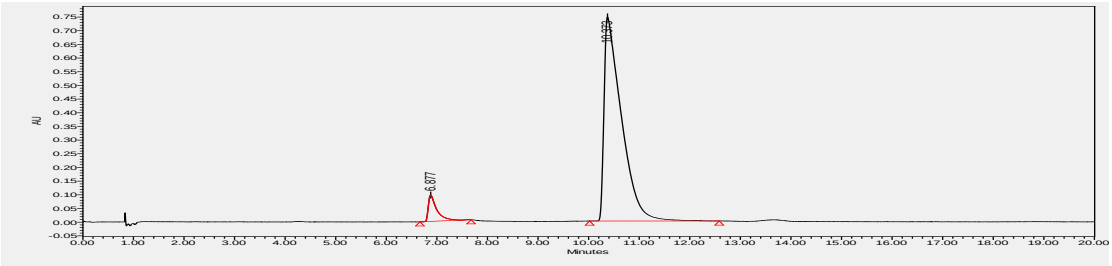
^{13}C NMR (100 MHz, $CDCl_3$). δ 163.9, 149.5, 148.1, 146.7, 126.5, 124.5, 79.8, 63.5, 27.6, 26.0, 21.0, 19.8, 19.0.

ESI-HRMS: calcd for $C_{13}H_{17}N_2O^+$ ($[M + H]^+$) = 217.1335, found 217.1331.

IR (neat): 2936, 1862, 1725, 1660, 1601, 1524, 1448, 1282, 1204, 1156, 1127, 1032 cm^{-1}.

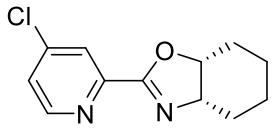


	Retention Time	Area	% Area
1	6.662	6376477	50.74
2	10.539	6190121	49.26



	Retention Time	Area	% Area
1	6.877	1117065	5.90
2	10.373	17816493	94.10

2d: (3aS,7aR)-2-(4-chloropyridin-2-yl)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazole



2d

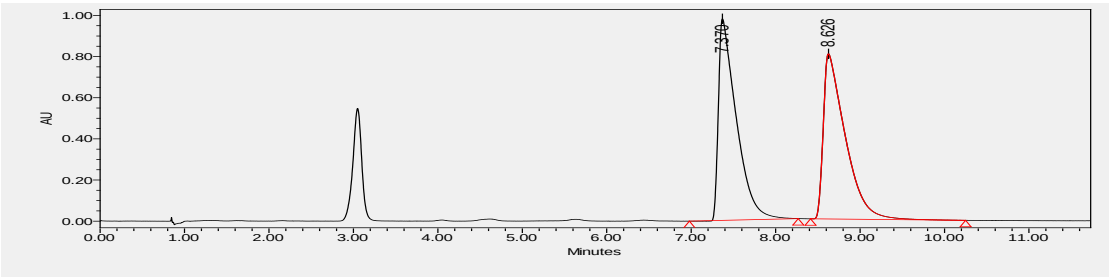
Following the typical procedure, colourless oil **2d** was isolated in 83% yield (19.5 mg) and 97% ee. $[\alpha]^{25}_{D}$ = –163.1 (*c* = 0.178 in CH_2Cl_2). **UPC²** (chiral OX-3 column), CO_2/MeOH = 93/7, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 7.3 min, t (major) = 8.2 min.

¹H NMR (400 MHz, Chloroform-*d*). δ 8.45 (d, *J* = 5.2 Hz, 1H), 8.19 (d, *J* = 1.6 Hz, 1H), 7.53 – 7.46 (m, 1H), 4.78 – 4.67 (m, 1H), 4.19 – 4.08 (m, 1H), 2.01 – 1.74 (m, 3H), 1.62 – 1.45 (m, 3H), 1.44 – 1.29 (m, 2H).

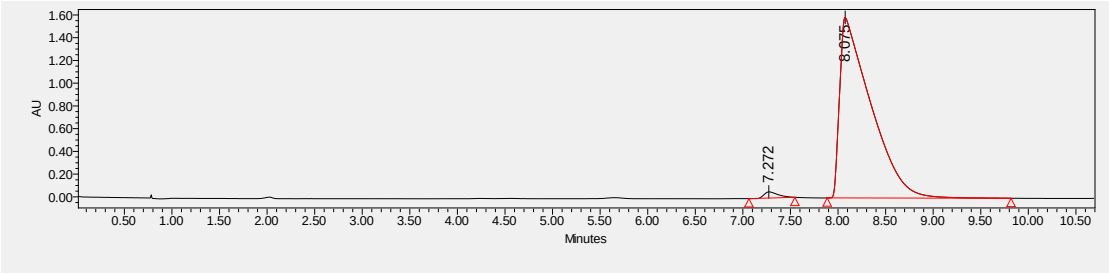
¹³C NMR (100 MHz, $CDCl_3$). δ 162.4, 150.3, 148.3, 133.3, 128.6, 127.0, 80.0, 63.8, 27.6, 26.0, 19.8, 18.9.

ESI-HRMS: calcd for $C_{12}H_{14}ClN_2O^+$ ($[M + H]^+$) = 237.0789 and 239.0760, found 237.0781 and 239.0750.

IR (neat): 2933, 2858, 1668, 1554, 1519, 1457, 1417, 1334, 1262, 1134, 1089, 978 cm^{-1}.

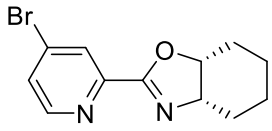


	Retention Time	% Area	Height
1	7.370	50.02	979976
2	8.626	49.98	803007



	Retention Time	Area	% Area
1	7.272	551750	1.51
2	8.075	36014687	98.49

2e: (3aS,7aR)-2-(4-bromopyridin-2-yl)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazole



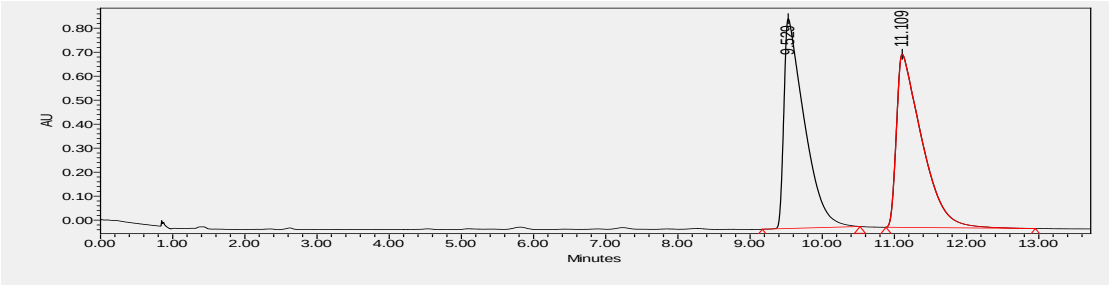
2e

Following the typical procedure, colourless oil **2e** was isolated in 86% yield (24.4 mg) and 97% ee. $[\alpha]^{25}_{D}$ = –137.2 (*c* = 0.182 in CH_2Cl_2). **UPC²** (chiral OX-3 column), CO_2/MeOH = 93/7, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 9.5 min, t (major) = 10.4 min. **^{1}H NMR** (400 MHz, Chloroform-*d*). δ 8.54 (d, *J* = 5.3 Hz, 1H), 8.02 (d, *J* = 2.0 Hz, 1H), 7.33 (dd, *J* = 5.3, 2.0 Hz, 1H), 4.73 (dt, *J* = 8.3, 5.2 Hz, 1H), 4.25 – 4.08 (m, 1H), 2.02 – 1.75 (m, 3H), 1.62 – 1.45 (m, 3H), 1.44 – 1.30 (m, 2H).

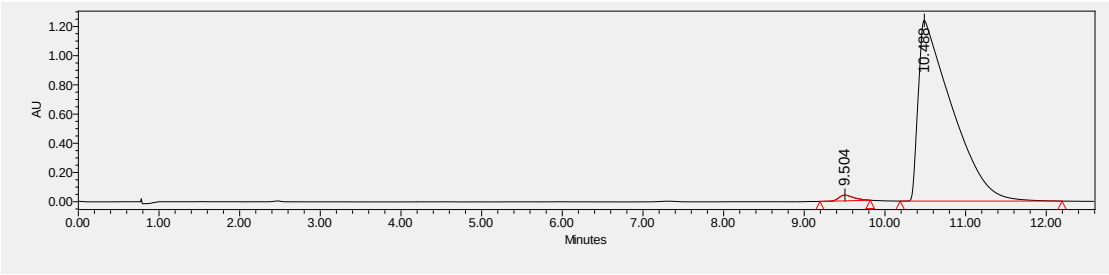
^{13}C NMR (100 MHz, $CDCl_3$). δ 162.6, 150.5, 148.5, 144.8, 125.6, 124.1, 80.0, 63.8, 27.5, 26.0, 19.8, 18.9.

ESI-HRMS: calcd for $C_{12}H_{14}BrN_2O^+$ ($[M + H]^+$) = 281.0284 and 283.0264, found 281.0274 and 283.0251.

IR (neat): 2932, 2857, 1659, 1570, 1551, 1518, 1459, 1412, 1132, 1085, 985, 972, 749, 721, 679 cm^{-1}.

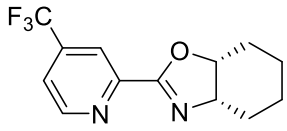


	Retention Time	Area	% Area
1	9.529	17156592	49.43
2	11.109	17553648	50.57



	Retention Time	Area	% Area
1	9.504	505145	1.39
2	10.488	35919549	98.61

2f: (3aS,7aR)-2-(4-(trifluoromethyl)pyridin-2-yl)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazole



2f

Following the typical procedure, colourless oil **2f** was isolated in 83% yield (22.4 mg) and 98% ee. $[\alpha]^{25}_{D}$ = –89.8 (*c* = 0.364 in CH_2Cl_2). **UPC²** (chiral OX-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 5.1 min, t (major) = 6.1 min.

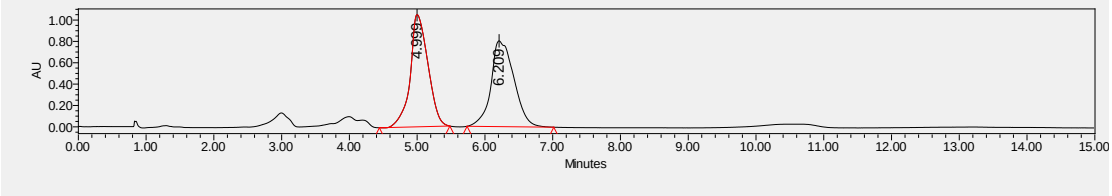
¹H NMR (400 MHz, Chloroform-*d*). 8.83 (d, *J* = 5.0 Hz, 1H), 8.24 (s, 1H), 7.54 (d, *J* = 6.0 Hz, 1H), 4.76 (dt, *J* = 8.3, 5.2 Hz, 1H), 4.23 – 4.12 (m, 1H), 2.09 – 1.74 (m, 3H), 1.64 – 1.47 (m, 3H), 1.45 – 126 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$). δ 162.5, 150.7, 139.3(d, *J* = 34.3 Hz), 123.8(d, *J* = 269.6 Hz), 120.9(d, *J* = 3.5 Hz), 119.6(d, *J* = 3.5 Hz), 80.2, 63.9, 27.5, 26.0, 19.8, 18.9.

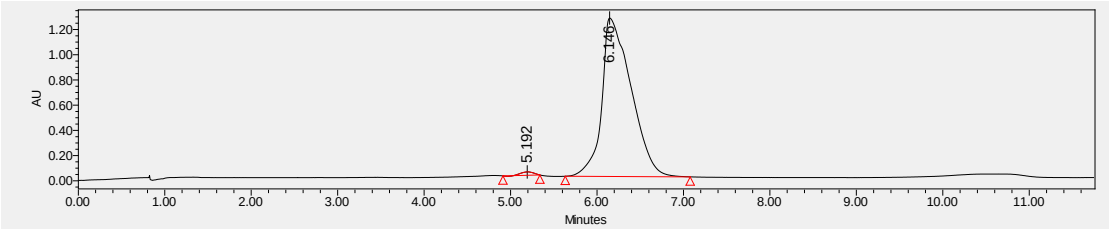
^{19}F NMR (376 MHz, Chloroform-*d*) δ -64.81.

ESI-HRMS: calcd for $C_{13}H_{14}F_3N_2O^+$ ($[M + H]^+$) = 271.1053, found 271.1042.

IR (neat): 2938, 2861, 1677, 1528, 1408, 1331, 1318, 1140, 971, 854 cm^{-1}.

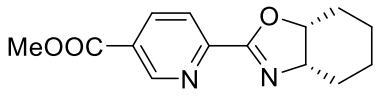


	Retention Time	Area	% Area
1	4.999	19128546	50.24
2	6.209	18947877	49.76



	Retention Time	Area	% Area
1	5.192	303635	1.01
2	6.146	29684831	98.99

2g: methyl 6-((3aS,7aR)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazol-2-yl)nicotinate



2g

Following the typical procedure, white solid **2g** was isolated in 74% yield (19.3 mg) and 85% ee. $[\alpha]^{25}_{D}$ = –126.3 (*c* = 0.340 in CH_2Cl_2). M.p.: 103 – 105 °C.

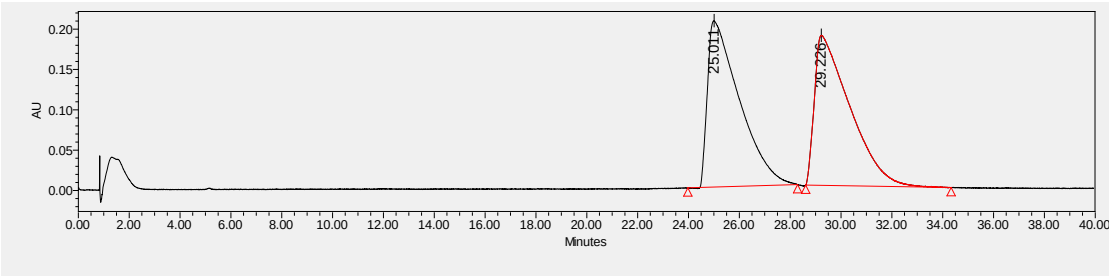
UPC² (chiral OX-3 column), CO_2/MeOH = 96/4, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 26.9 min, t (major) = 29.2 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 9.28 (s, 1H), 8.38 (d, *J* = 8.2 Hz, 1H), 8.15 (d, *J* = 8.2 Hz, 1H), 4.90 – 4.73 (m, 1H), 4.23 (m, 1H), 3.98 (s, 3H), 2.10 – 1.95 (m, 2H), 1.72 – 1.33 (m, 6H).

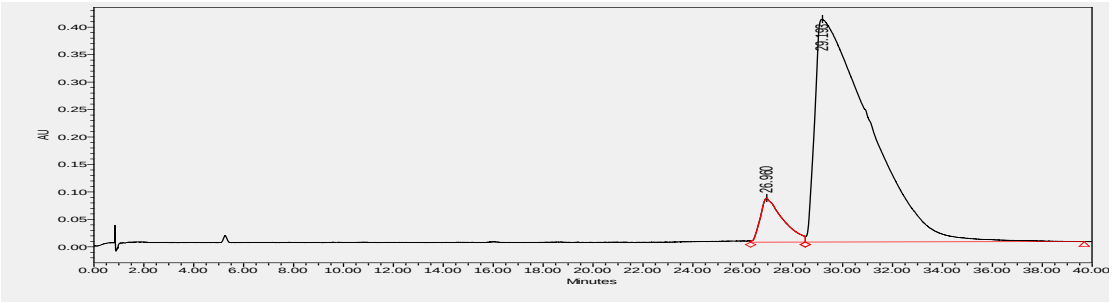
^{13}C NMR (100 MHz, $CDCl_3$). δ 165.2, 162.9, 150.7, 150.3, 137.7, 127.2, 123.2, 80.0, 63.9, 52.6, 27.6, 26.0, 19.8, 19.0.

ESI-HRMS: calcd for $C_{14}H_{17}N_2O_3^+$ ([M + H]$^+$) = 261.1234, found 261.1226.

IR (neat): 2935, 2857, 1720, 1675, 1636, 1595, 1523, 1437, 1391, 1279, 1194, 1116, 1083, 1019 cm^{-1}.

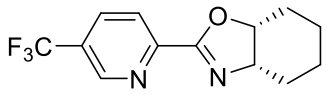


	Retention Time	Area	% Area
1	25.011	17971727	50.05
2	29.226	17935448	49.95



	Retention Time	Area	% Area
1	26.960	5035789	7.55
2	29.193	61643822	92.45

2h: (3aS,7aR)-2-(5-(trifluoromethyl)pyridin-2-yl)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazole



2h

Following the typical procedure, colourless oil **2h** was isolated in 82% yield (22.0 mg) and 84% ee. $[\alpha]_D^{25}$ = –97.4 (*c* = 0.530 in CH_2Cl_2).

UPC² (chiral AD-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 2.0 min, t (major) = 2.5 min.

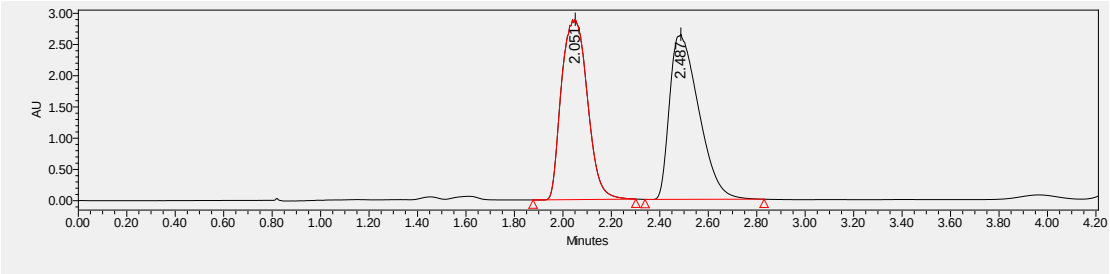
^{1}H NMR (400 MHz, Chloroform-*d*). δ 9.01 – 8.89 (m, 1H), 8.21 (d, *J* = 8.2 Hz, 1H), 8.03 (dd, *J* = 8.3, 1.8 Hz, 1H), 4.83 (dt, *J* = 8.3, 5.2 Hz, 1H), 4.31 – 4.18 (m, 1H), 4.28 – 4.20 (m, 3H), 2.05 – 1.85 (m, 3H), 1.71 – 1.54 (m, 3H), 1.52 – 1.38 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$).δ 162.5, 150.3, 146.6, 133.9 (q, *J* = 3.1 Hz) , 128.1 (q, *J* = 34.1 Hz), 123.4, 121.8 (q, *J* = 271.3 Hz), 80.1, 64.0, 26.0, 19.7, 18.9.

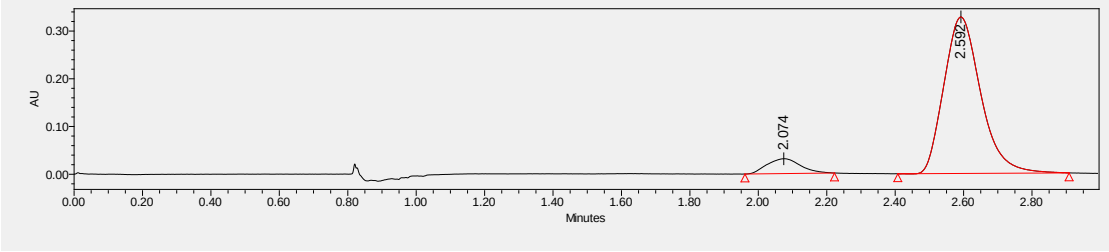
^{19}F NMR (376 MHz, Chloroform-*d*) δ -64.81.

ESI-HRMS: calcd for $C_{13}H_{14}F_3N_2O^+$ ($[M + H]^+$) = 271.1053, found 271.1043.

IR (neat): 2938, 2863, 1673, 1571, 1529, 1395, 1331, 1266, 1164, 1131, 1099, 1017,871, 803 cm^{-1}.

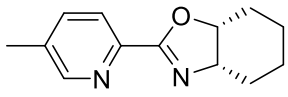


	Retention Time	Area	% Area
1	2.051	21456234	49.31
2	2.487	22052819	50.69



	Retention Time	Area	% Area
1	2.074	212580	7.98
2	2.592	2450221	92.02

2i: (3aS,7aR)-2-(5-methylpyridin-2-yl)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazole



2i

Following the typical procedure, colourless oil **2i** was isolated in 79% yield (16.9 mg) and 98% ee. $[\alpha]^{25}_{D}$ = –97.4 (*c* = 0.280 in CH_2Cl_2).

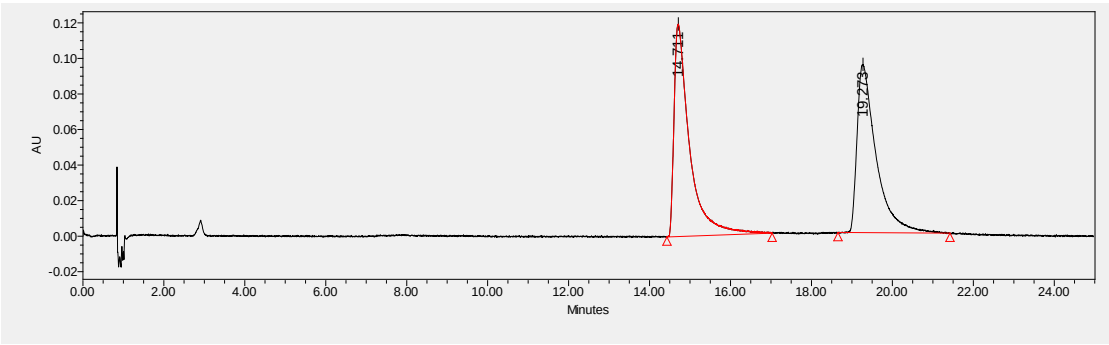
UPC² (chiral OX-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 8.5 min, t (major) = 10.3 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 8.54 (s, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.57 (d, *J* = 9.6 Hz, 1H), 4.76 (dt, *J* = 8.2, 5.2 Hz, 1H), 4.24 – 4.06 (m, 1H), 2.39 (s, 3H), 2.08– 1.78 (m, 4H), 1.69 – 1.58 (m, 2H), 1.52 – 1.30 (m, 2H).

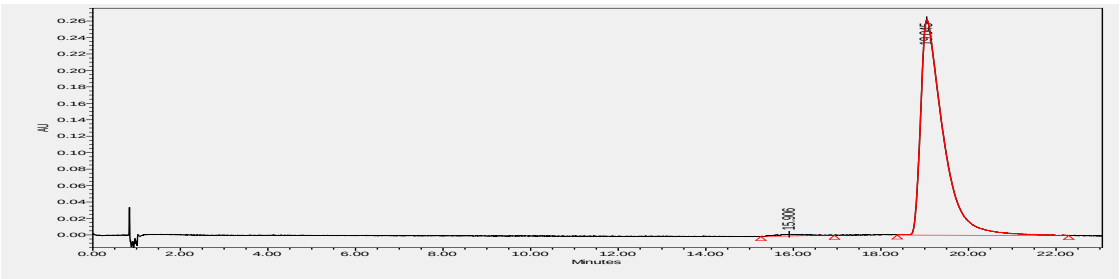
^{13}C NMR (100 MHz, $CDCl_3$). δ 163.6, 150.1, 144.6, 136.9, 135.5, 123.2, 79.6, 63.6, 27.7, 26.1, 19.9, 19.0, 18.5.

ESI-HRMS: calcd for $C_{13}H_{17}N_2O^+$ ($[M + H]^+$) = 217.1335, found 217.1329.

IR (neat): 2927, 2851, 1613, 1517, 1493, 1312, 1147, 891, 876 cm^{-1}.

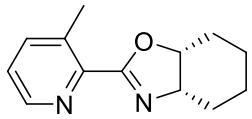


	Retention Time	Area	% Area
1	14.711	3186789	50.82
2	19.273	3084287	49.18



	Retention Time	Area	% Area
1	15.906	97478	1.04
2	19.045	9271138	98.96

2j: (3aS,7aR)-2-(3-methylpyridin-2-yl)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazole



2j

Following the typical procedure, colourless oil **2j** was isolated in 78% yield (16.9 mg) and 96% ee. $[\alpha]^{19}_{D}$ = - 9.02 (*c* =0.964 in CH_2Cl_2).

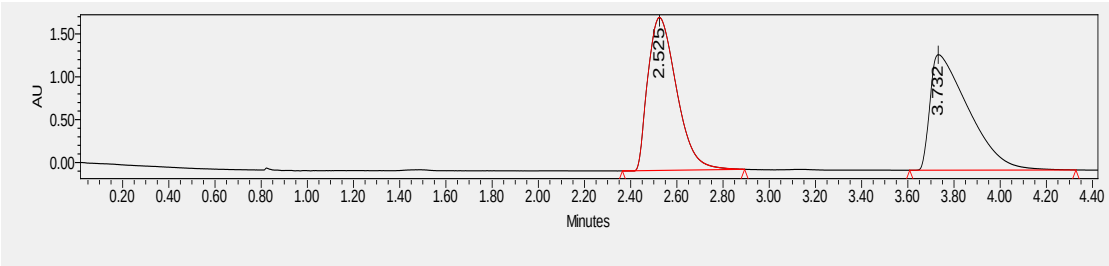
UPC² (chiral AD-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 2.5 min, t (major) = 3.7 min.

^{1}H NMR (600 MHz, Chloroform-*d*). δ 8.49 (d, *J* = 5.0 Hz, 1H), 7.83 (s, 1H), 7.14 (d, *J* = 5.8 Hz, 1H), 4.71 (dt, *J* = 8.2, 5.1 Hz, 1H), 4.19 – 3.99 (m, 1H), 2.33 (s, 3H), 2.03 – 1.69 (m, 4H), 1.64 – 1.56 (m, 2H), 1.53 – 1.33 (m, 2H).

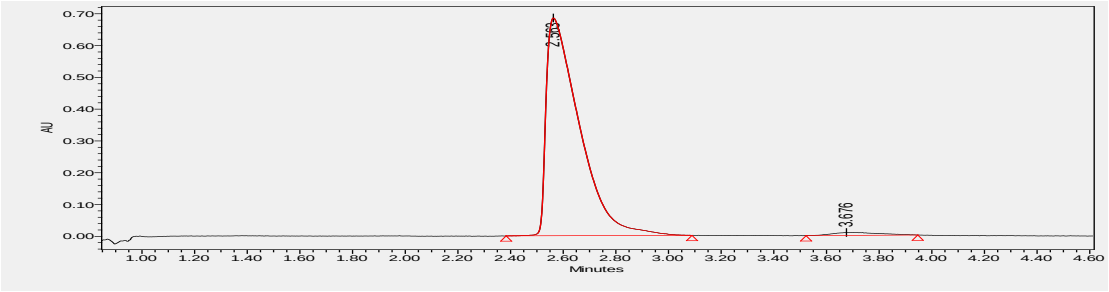
^{13}C NMR (151 MHz, Chloroform-*d*) δ 162.3, 145.9, 145.1, 138.2, 133.7, 123.6, 77.6, 63.1, 26.9, 24.9, 19.4, 18.9, 18.0.

ESI-HRMS: calcd for $C_{13}H_{17}N_2O^+$ ($[M + H]^+$) = 217.1335, found 217.1328.

IR (neat): 2931, 2859, 1725, 1662, 1512, 1449, 1269, 1098 cm^{-1}.

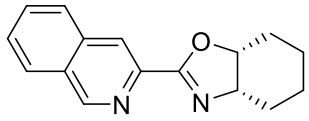


	Retention Time	Area	% Area
1	2.525	15375898	49.66
2	3.732	15587417	50.34



	Retention Time	Area	% Area
1	2.563	5987932	98.11
2	3.676	115145	1.89

2k: (3aS,7aR)-2-(isoquinolin-3-yl)-3a,4,5,6,7,7a-hexahydrobenzo[d]oxazole



2k

Following the typical procedure, white solid **2k** was isolated in 90% yield (22.6 mg) and 72% ee. $[\alpha]^{25}_{D}$ = 120.9 (*c* = 0.372 in CH_2Cl_2). M.p.: 69 – 72 ℃.

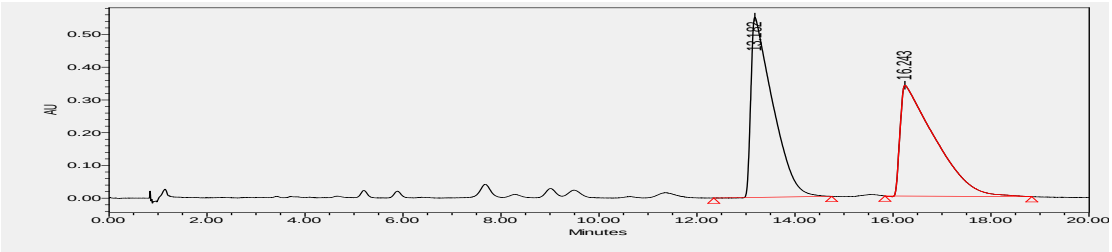
UPC² (chiral OX-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (major) = 12.8 min, t (minor) = 16.5 min.

^{1}H NMR (400 MHz, Chloroform-*d*).δ 9.25 (d, *J* = 8.4 Hz, 1H), 8.66 (d, *J* = 5.5 Hz, 1H), 7.86 (d, *J* = 7.5 Hz, 1H), 7.81 – 7.60 (m, 3H), 4.92 – 4.77 (m, 1H), 4.48 – 4.27 (m, 1H), 2.23 – 2.00 (m, 2H), 1.99 – 1.76 (m, 2H), 1.72 – 1.59 (m, 2H), 1.56 – 1.36 (m, 2H).

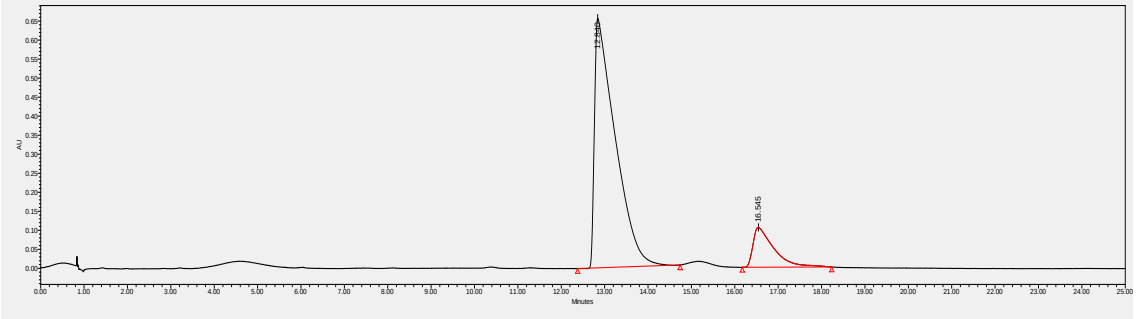
^{13}C NMR (100 MHz, $CDCl_3$). δ 146.9, 141.8, 136.7, 130.3, 128.4, 127.4, 127.3, 127.0, 123.1, 78.72, 64.6, 27.8, 26.0, 19.9, 19.0.

ESI-HRMS: calcd for $C_{16}H_{17}N_2O^+$ ($[M + H]^+$) = 253.1355, found 253.1329.

IR (neat): 2934, 2859, 1665, 1582, 1561, 1552, 1447, 1421, 1157, 1136, 989, 819, 764, 747 cm^{-1}.

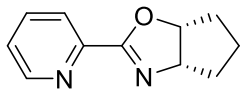


	Retention Time	Area	% Area
1	13.182	16989430	50.45
2	16.243	16683290	49.55



	Retention Time	Area	% Area
1	12.840	21730038	86.24
2	16.545	3465707	13.76

2l: (3aS,6aR)-2-(pyridin-2-yl)-3a,5,6,6a-tetrahydro-4H-cyclopenta[d]oxazole



2l

Following the typical procedure, colourless oil **2l** was isolated in 83% yield (16.0 mg) and 91% ee. $[\alpha]^{25}_{D}$ = –97.4 (*c* = 0.530 in CH_2Cl_2).

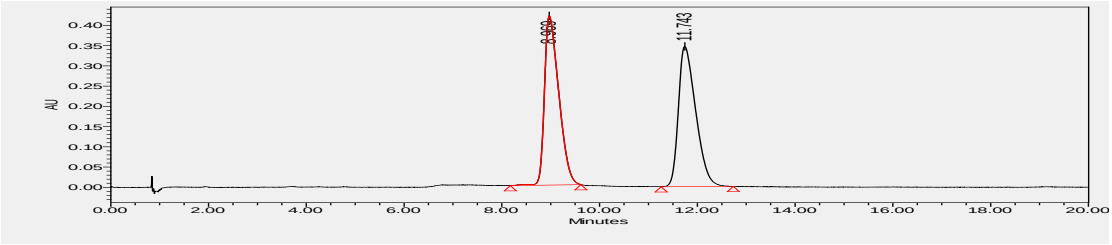
UPC² (chiral OX-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 8.9 min, t (major) = 10.8 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 8.78 – 8.65 (m, 1H), 8.03 (m, 1H), 7.77 (m, 1H), 7.38 (m, 1H), 5.25 – 5.16 (m, 1H), 4.82 – 4.73 (m, 1H), 2.20 (dd, *J* = 13.9, 5.7 Hz, 1H), 2.05 (dd, *J* = 13.9, 5.7 Hz, 1H), 1.90 – 1.60 (m, 4H), 1.63 – 1.44 (m, 1H).

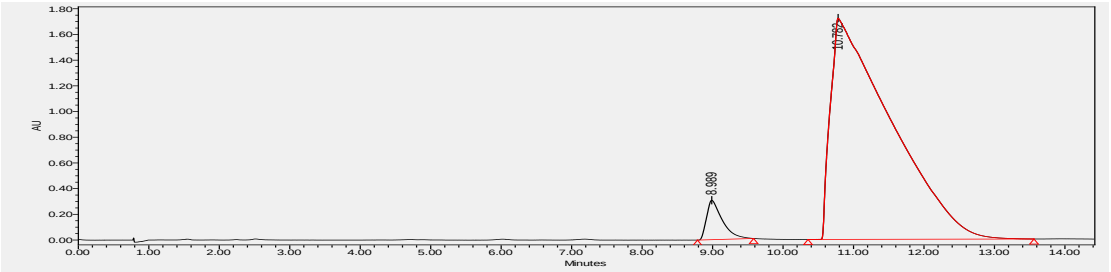
^{13}C NMR (100 MHz, $CDCl_3$). δ 163.0, 149.7, 146.7, 136.5, 125.3, 123.8, 85.6, 72.1, 34.6, 33.8, 22.2.

ESI-HRMS: calcd for $C_{11}H_{13}N_2O^+$ ($[M + H]^+$) = 189.1022, found 189.1017.

IR (neat): 2961, 2854, 1659, 1587, 1521, 1360, 1263, 1100, 1023, 748, 685 cm^{-1}.

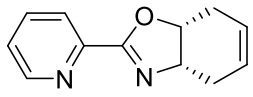


	Retention Time	Area	% Area
1	8.969	8500981	49.91
2	11.743	8532681	50.09



	Retention Time	Area	% Area
1	8.989	4729858	4.41
2	10.782	102447810	95.59

2m: (3aS,7aR)-2-(pyridin-2-yl)-3a,4,7,7a-tetrahydrobenzo[d]oxazole



2m

Following the typical procedure, colourless oil **2m** was isolated in 80% yield (15.9 mg) and 89% ee. $[\alpha]^{25}_{D} = -211.4$ (c = 0.112 in CH_2Cl_2).

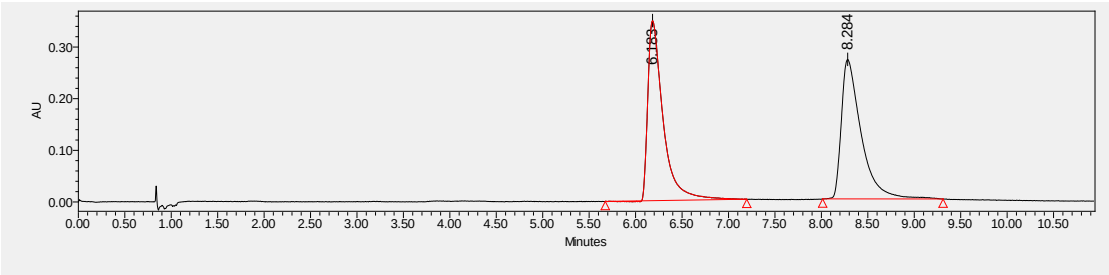
UPC² (chiral OX-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 9.7 min, t (major) = 12.5 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 8.63 (m, 1H), 7.93 (m, 1H), 7.68 (m, 1H), 7.30 (ddd, *J* = m, 1H), 5.94 – 5.73 (m, 2H), 5.10 – 5.00 (m, 1H), 4.79 – 4.53 (m, 1H), 2.64 – 2.50 (m, 1H), 2.56 – 2.34 (m, 1H), 2.31 – 2.17 (m, 2H).

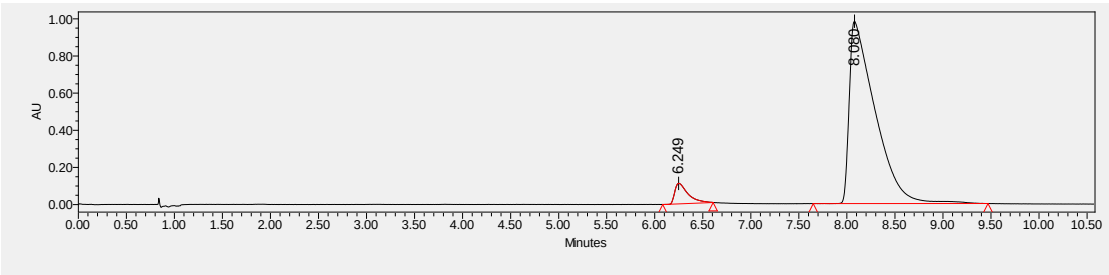
^{13}C NMR (100 MHz, $CDCl_3$). δ 163.2, 149.7, 146.7, 136.5, 128.4, 125.9, 125.4, 123.8, 79.4, 65.4, 27.8, 27.7.

ESI-HRMS: calcd for $C_{12}H_{13}N_2O^+$ ($[M + H]^+$) = 201.1022, found 201.1015.

IR (neat): 2921, 2853, 1665, 1552, 1466, 1434, 1262, 1093, 799, 732, 702 cm^{-1}.

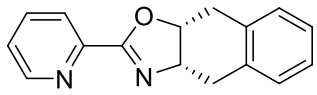


	Retention Time	Area	% Area
1	6.183	4011469	50.11
2	8.284	3993387	49.89



	Retention Time	Area	% Area
1	6.249	1055538	5.46
2	8.080	18277256	94.54

2n: (3aS,9aR)-2-(pyridin-2-yl)-3a,4,9,9a-tetrahydronaphtho[2,3-d]oxazole



2n

Following the typical procedure, white solid **2n** was isolated in 90% yield (21.8 mg) and 91% ee. $[\alpha]^{25}_{D}$ = –232.1 (*c* = 0.056 in CH_2Cl_2). M.p.: 67 – 69 °C.

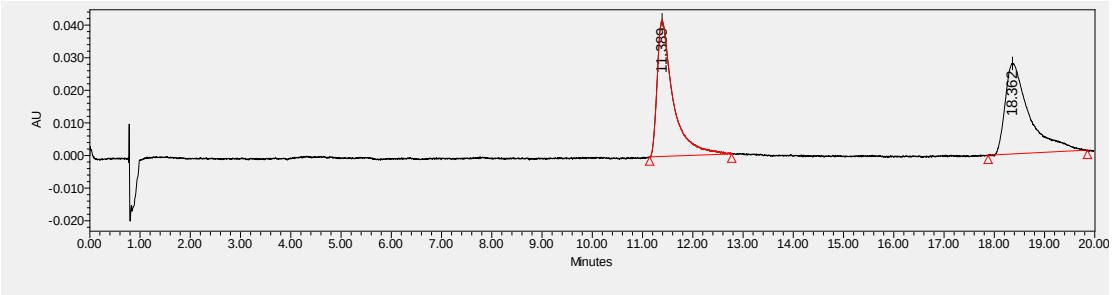
UPC² (chiral OX-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 11.1 min, t (major) = 16.4 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 8.56 (m, 1H), 7.82 (m, 1H), 7.61 (m, 1H), 7.31 – 7.21 (m, 1H), 7.17 – 7.01 (m, 4H), 5.18 (dt, J = 9.6, 4.6 Hz, 1H), 4.75 (dt, J = 10.3, 5.3 Hz, 1H), 3.25 – 3.05 (m, 1H), 3.05 – 2.87 (m, 3H).

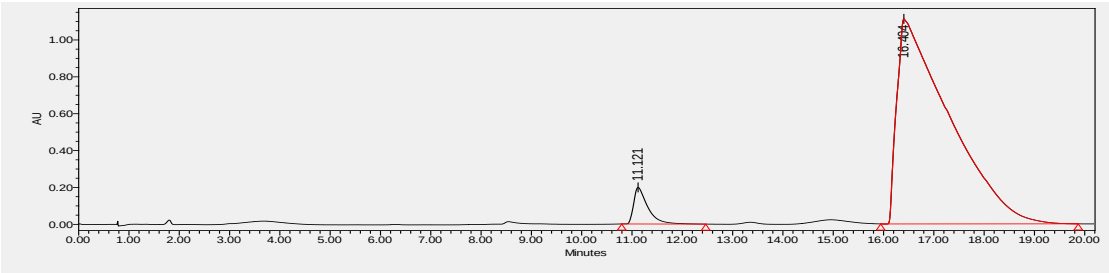
^{13}C NMR (100 MHz, $CDCl_3$). δ 163.0, 149.6, 146.5, 136.4, 136.0, 134.2, 128.4, 128.3, 126.9, 126.7, 125.4, 123.8, 79.4, 66.1, 33.7, 33.6.

ESI-HRMS: calcd for $C_{16}H_{15}N_2O^+$ ($[M + H]^+$) = 251.1179, found 251.1172.

IR (neat): 2941, 2873, 1648, 2578, 1519, 1257, 1041, 859, 801, 753, 687, 621 cm^{-1}.

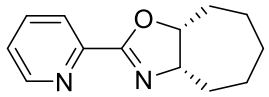


	Retention Time	Area	% Area
1	11.389	918156	49.76
2	18.362	926920	50.24



	Retention Time	Area	% Area
1	11.121	3836733	4.55
2	16.404	80554327	95.45

2o: (3aS,8aR)-2-(pyridin-2-yl)-3a,5,6,7,8,8a-hexahydro-4H-cyclohepta[d]oxazole



2o

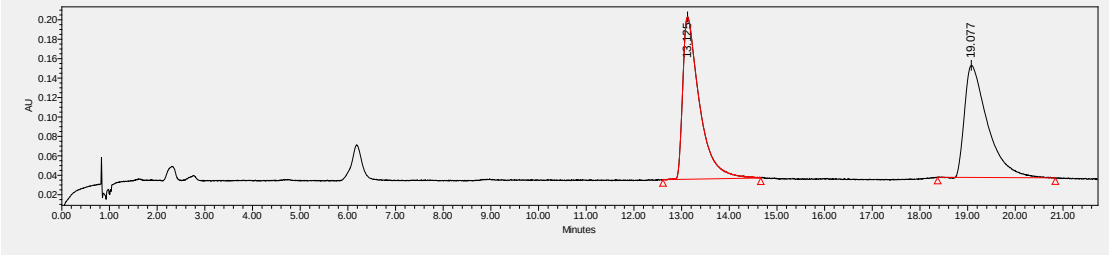
Following the typical procedure, colourless oil **2o** was isolated in 70% yield (14.9 mg) and 89% ee. $[\alpha]^{25}_{D}$ = –211.4 (*c* = 0.336 in CH_2Cl_2). **UPC²** (chiral OX-3 column), CO_2/MeOH = 91/9, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 12.8 min, t (major) = 17.9 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 8.77 – 8.56 (m, 1H), 7.97 (d, *J* = 7.9 Hz, 1H), 7.69 (td, *J* = 7.8, 1.7 Hz, 1H), 7.35 – 7.26 (m, 1H), 4.87 (td, *J* = 10.0, 3.6 Hz, 1H), 4.40 (td, *J* = 10.0, 3.6 Hz, 1H), 2.01 – 1.81 (m, 3H), 1.78 – 1.60 (m, 3H), 1.45 – 1.21 (m, 4H).

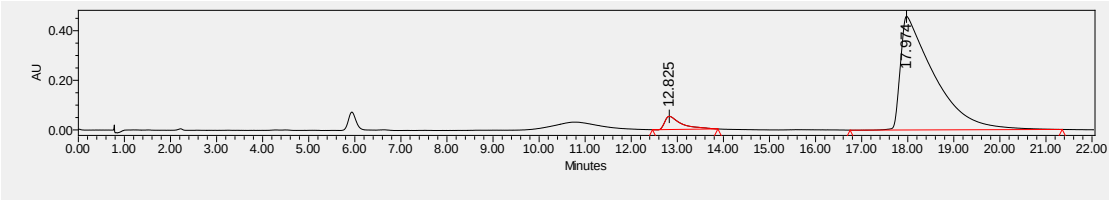
^{13}C NMR (100 MHz, $CDCl_3$). δ 161.4, 149.6, 147.0, 136.5, 123.7, 83.9, 70.0, 31.4, 30.8, 30.7, 26.2, 24.3.

ESI-HRMS: calcd for $C_{13}H_{17}N_2O^+$ ($[M + H]^+$) = 217.1335, found 217.1329.

IR (neat): 2927, 2855, 1658, 1586, 1518, 1366, 1102, 748, 679 cm^{-1}.

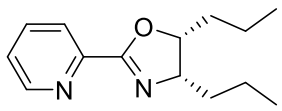


	Retention Time	Area	% Area
1	13.125	4078854	51.23
2	19.077	3883373	48.77



	Retention Time	Area	% Area
1	12.825	1342774	5.45
2	17.974	23298162	94.55

2p: (4S,5R)-4,5-dipropyl-2-(pyridin-2-yl)-4,5-dihydrooxazole



2p

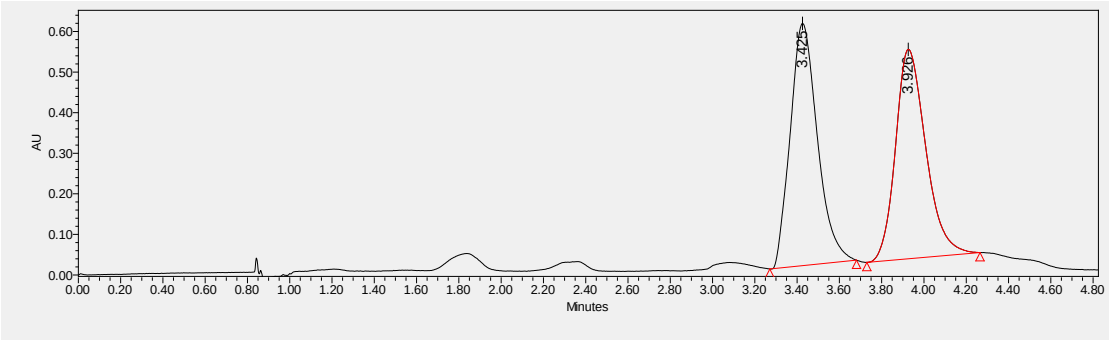
Following the typical procedure, colourless oil **2p** was isolated in 60% yield (13.8 mg) and 90% ee. $[\alpha]^{25}_{D}$ = –144.1 (*c* = 0.287 in CH_2Cl_2). **UPC²** (chiral OX-3 column), CO_2/MeOH = 93/7, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 3.6 min, t (major) = 4.3 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 8.65 (d, *J* = 4.8 Hz, 1H), 7.94 (d, *J* = 7.9 Hz, 1H), 7.74 – 7.60 (m, 1H), 7.33 – 7.27 (m, 1H), 4.69 (td, *J* = 9.5, 3.6 Hz, 1H), 4.26 – 4.11 (m, 1H), 1.60 – 1.35 (m, 8H), 1.06 – 0.83 (m, 6H).

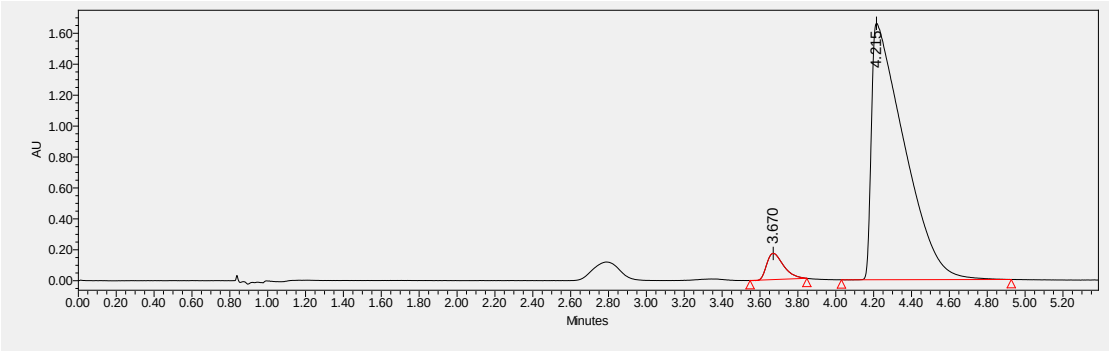
^{13}C NMR (100 MHz, $CDCl_3$). δ 162.0, 149.8, 147.3, 136.5, 125.2, 123.7, 83.5, 68.2, 32.5, 31.5, 20.5, 20.0, 14.2, 14.0.

ESI-HRMS: calcd for $C_{14}H_{21}N_2O^+$ ($[M + H]^+$) = 233.1648, found 233.1642.

IR (neat): 2957, 2932, 2871, 1658, 1569 1522, 1433, 1030, 977, 747, 638, 620 cm^{-1}.

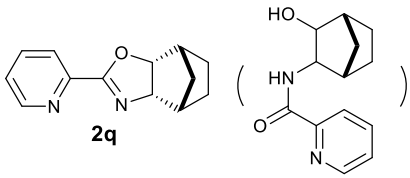


	Retention Time	Area	% Area
1	3.425	5328989	50.96
2	3.926	5128042	49.04



	Retention Time	Area	% Area
1	3.670	1052269	5.02
2	4.215	19920928	94.98

2q: (3aS,4S,7R,7aR)-2-(pyridin-2-yl)-3a,4,5,6,7,7a-hexahydro-4,7-methanobenzo[d]oxazole



2q

Following the typical procedure, white solid **2q** was isolated in 95% yield (20.4 mg) and >99% ee. (hydrolysis byproducts include). $[\alpha]^{16}_{D}$ = +51.0 (*c* = 0.200 in CH_2Cl_2). M. p. :71-73 ^{o}C

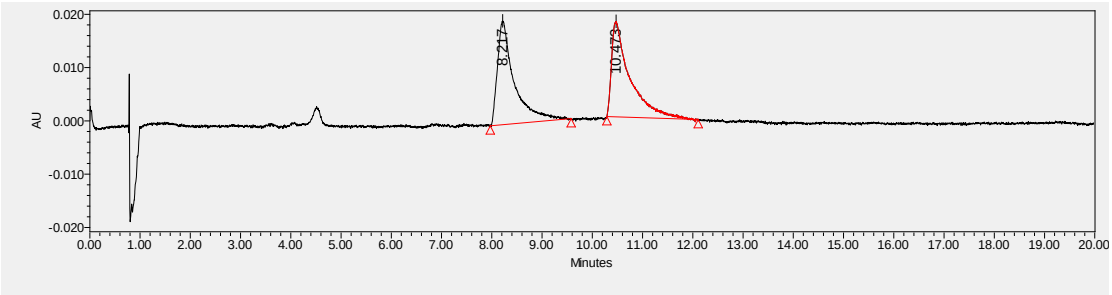
UPC² (chiral OX-3 column), CO_2/MeOH = 90/10, flow rate 1.5 mL/min, λ = 254 nm, retention time: t (minor) = 8.0 min, t (major) = 10.5 min.

^{1}H NMR (400 MHz, Chloroform-*d*). δ 8.71 (d, *J* = 2.3 Hz, 1H), 8.03 (d, *J* = 7.9 Hz, 1H), 7.77 (td, *J* = 7.8, 1.7 Hz, 1H), 7.41 – 7.34 (m, 1H), 4.62 (d, *J* = 7.0 Hz, 1H), 4.20 (d, *J* = 7.0 Hz, 1H), 2.63 – 2.40 (m, 2H), 1.59 – 1.55 (m, 2H), 1.29 – 1.17 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$). δ 164.2, 149.7, 146.6, 136.6, 125.5, 123.9, 86.4, 75.2, 41.2, 41.2, 31.4, 25.7, 23.5.

ESI-HRMS: calcd for $C_{13}H_{15}N_2O^+$ ($[M + H]^+$) = 215.1179, found 215.1177.

IR (neat): 2901, 1636, 1585, 1589, 1470, 1440, 1358, 1327, 1289, 1112, 982, 891, 746, 679 cm^{-1}.



	Retention Time	Area	% Area
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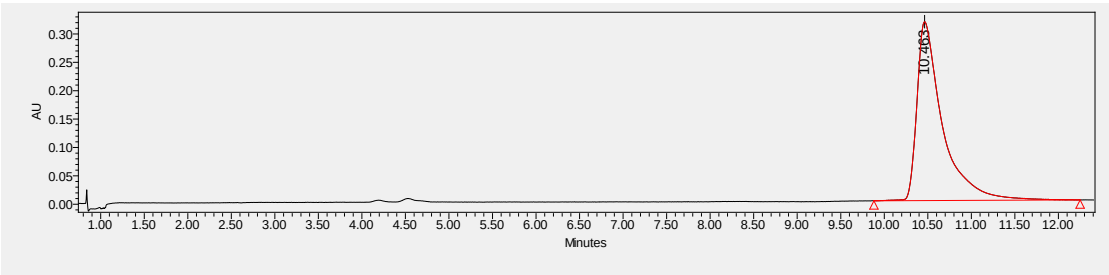
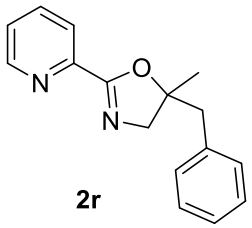
2	10.473	451623	50.05



	Retention Time	Area	% Area
1	10.547	4985322	100.00

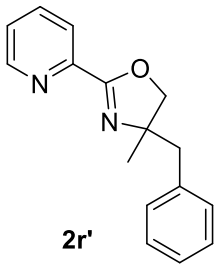
2r: 5-benzyl-5-methyl-2-(pyridin-2-yl)-4,5-dihydrooxazole



2r

^{1}H NMR (400 MHz, Chloroform-*d*). δ 8.65 (d, *J* = 4.8 Hz, 1H), 7.94 (d, *J* = 7.9 Hz, 1H), 7.74 – 7.60 (m, 1H), 7.33 – 7.27 (m, 1H), 4.69 (td, *J* = 9.5, 3.6 Hz, 1H), 4.26 – 4.11 (m, 1H), 1.60 – 1.35 (m, 8H), 1.06 – 0.83 (m, 6H).
^{13}C NMR (100 MHz, $CDCl_3$). δ 162.0, 149.8, 147.3, 136.5, 125.2, 123.7, 83.5, 68.2, 32.5, 31.5, 20.5, 20.0, 14.2, 14.0.

2r': 4-benzyl-4-methyl-2-(pyridin-2-yl)-4,5-dihydrooxazole

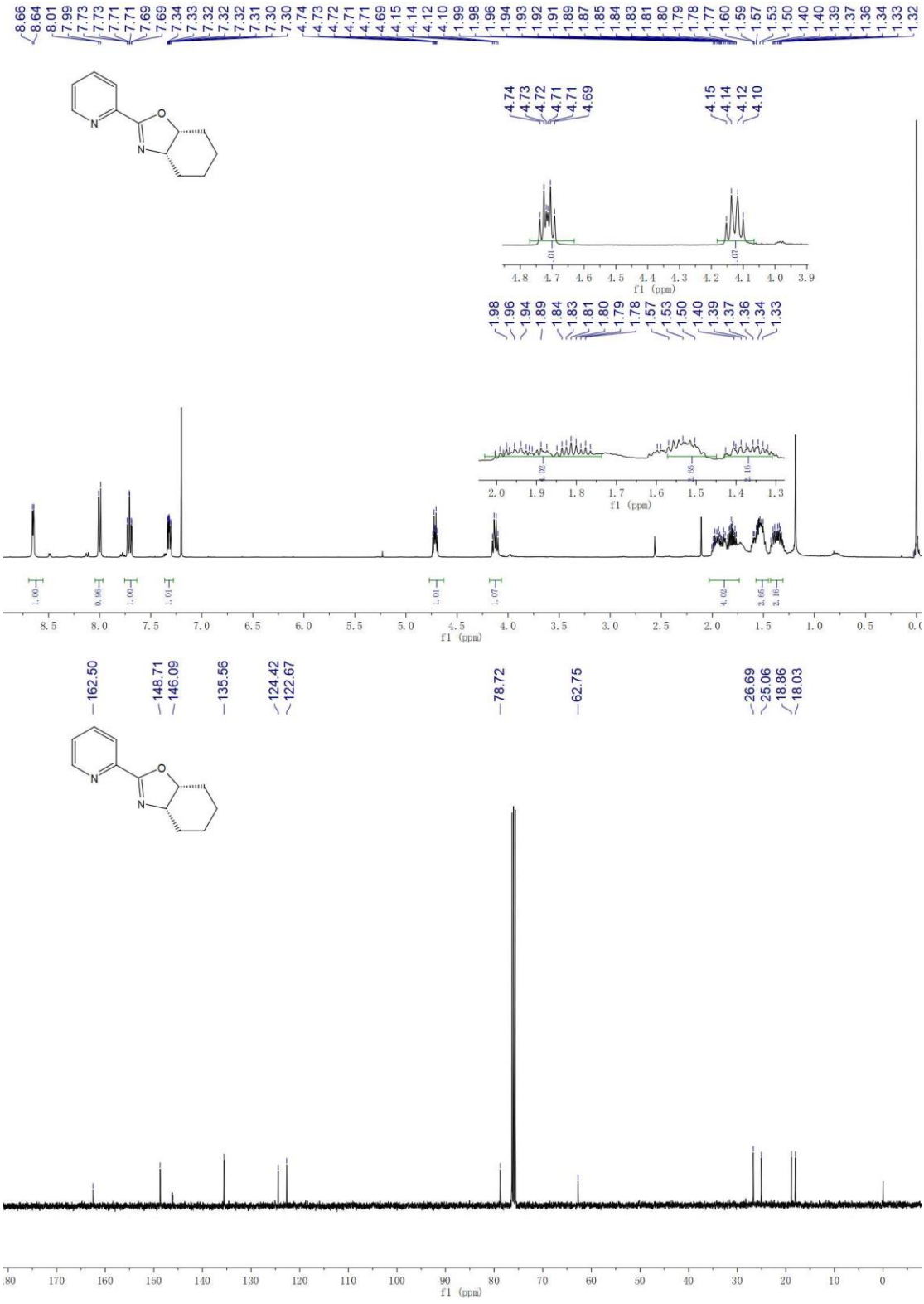


2r'

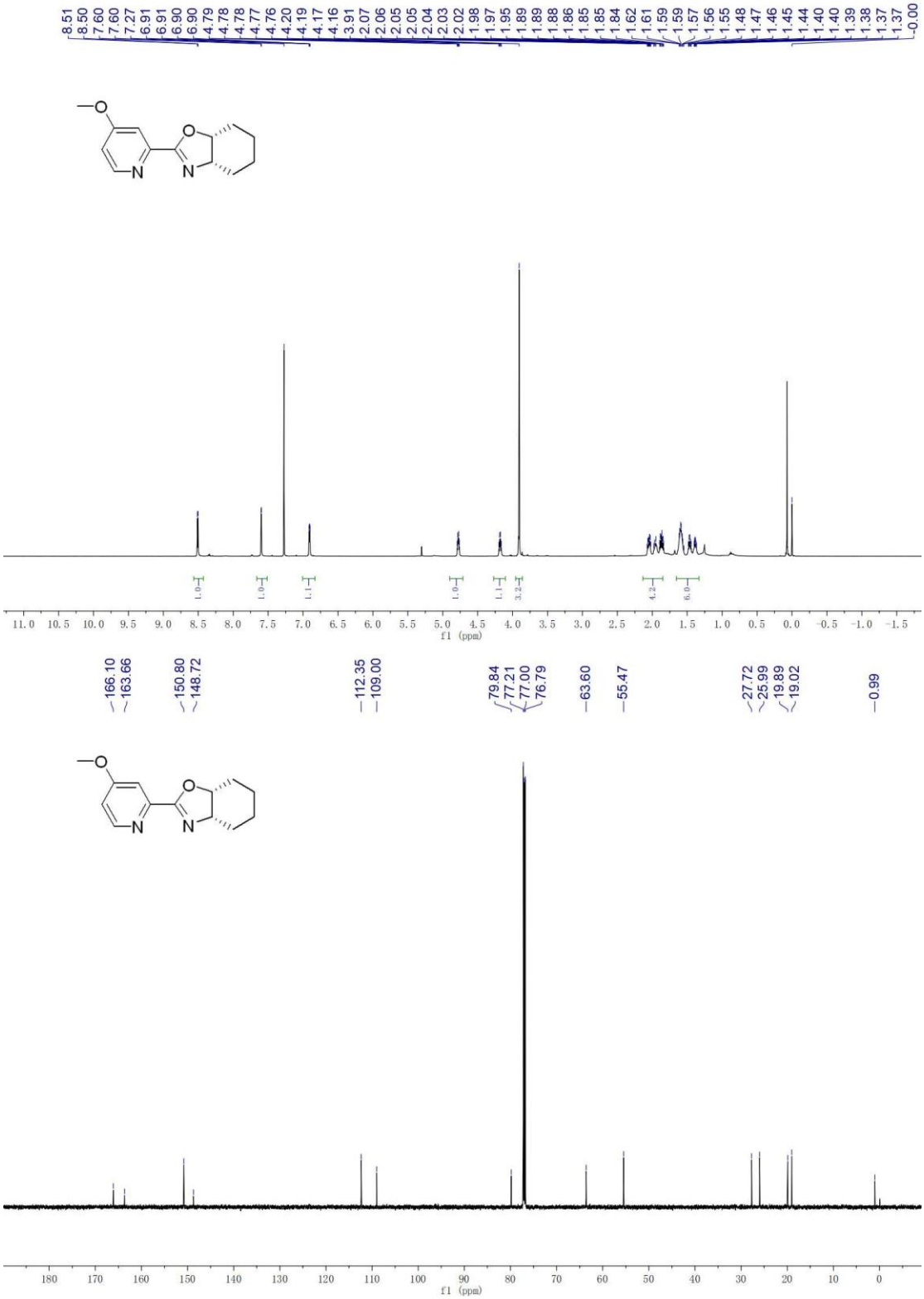
^{1}H NMR (400 MHz, Chloroform-*d*). δ 8.65 (d, *J* = 4.8 Hz, 1H), 7.94 (d, *J* = 7.9 Hz, 1H), 7.74 – 7.60 (m, 1H), 7.33 – 7.27 (m, 1H), 4.69 (td, *J* = 9.5, 3.6 Hz, 1H), 4.26 – 4.11 (m, 1H), 1.60 – 1.35 (m, 8H), 1.06 – 0.83 (m, 6H).
^{13}C NMR (100 MHz, $CDCl_3$). δ 162.0, 149.8, 147.3, 136.5, 125.2, 123.7, 83.5, 68.2, 32.5, 31.5, 20.5, 20.0, 14.2, 14.0.
(Z. Q. Li, Y. Fu, R. Deng, V. T. Tran, Y. Gao, P. Liu, K. M. Engle, *Angew. Chem. Int. Ed.* 2020, 59, 23306)

14. NMR spectrum.

2a



2b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4.76
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4.19
4.17
4.16
3.91
2.07
2.06
2.05
2.05
2.04
2.03
2.02
1.98
1.97
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1.45
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1.38
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N
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1.1
1.0
1.1
3.2
4.2
6.0
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 0.0 -0.5 -1.0 -1.5
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112.35
109.00
79.84
77.21
77.00
76.79
63.60
55.47
27.72
25.99
19.89
19.02
0.99
O
N
O
N
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

2c


8.50 8.48 7.83 7.20 7.15 7.13 4.73 4.72 4.71 4.71 4.70 4.69 4.14 4.12 4.10 4.08 2.33 2.00 1.98 1.97 1.96 1.96 1.95 1.93 1.92 1.89 1.88 1.86 1.85 1.84 1.83 1.81 1.80 1.77 1.57 1.56 1.55 1.54 1.53 1.52 1.51 1.50 1.49 1.47 1.46 1.40 1.38 1.37 1.36 1.35 1.34 1.32 1.31 1.30 1.29 0.00
1.97 1.95 1.93 1.85 1.84 1.83 1.81 1.80 1.54 1.53 1.52 1.51 1.50 1.49 1.47 1.40 1.38 1.32 1.31
4.73 4.72 4.71 4.71 4.70 4.69
4.14 4.12 4.10 4.08
f1 (ppm)
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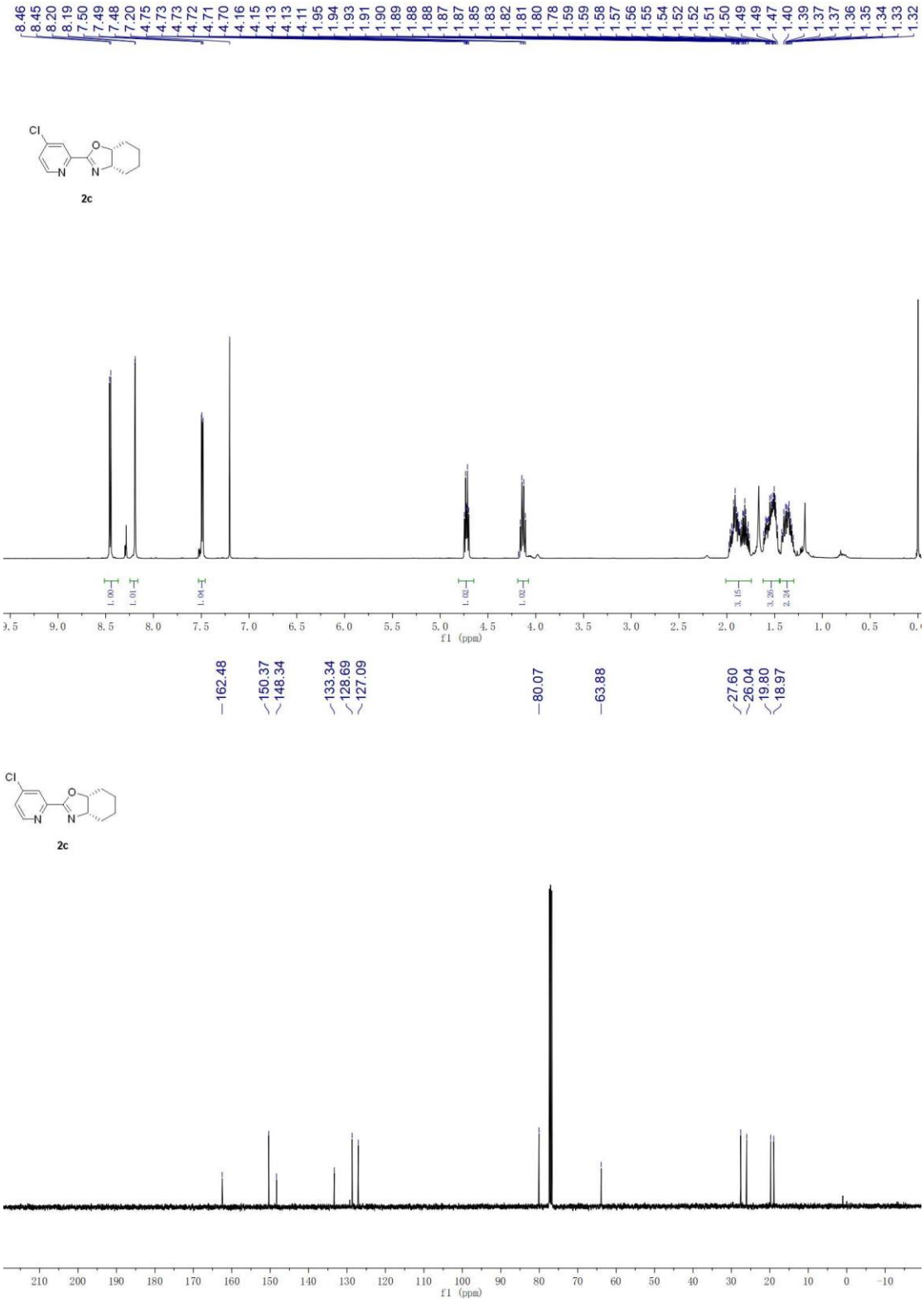
f1 (ppm)

2d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4.13
4.11
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1.89
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1.87
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1.85
1.83
1.82
1.81
1.80
1.78
1.59
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2c
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1.02
1.02
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2.24
f1 (ppm)
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150.37
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133.34
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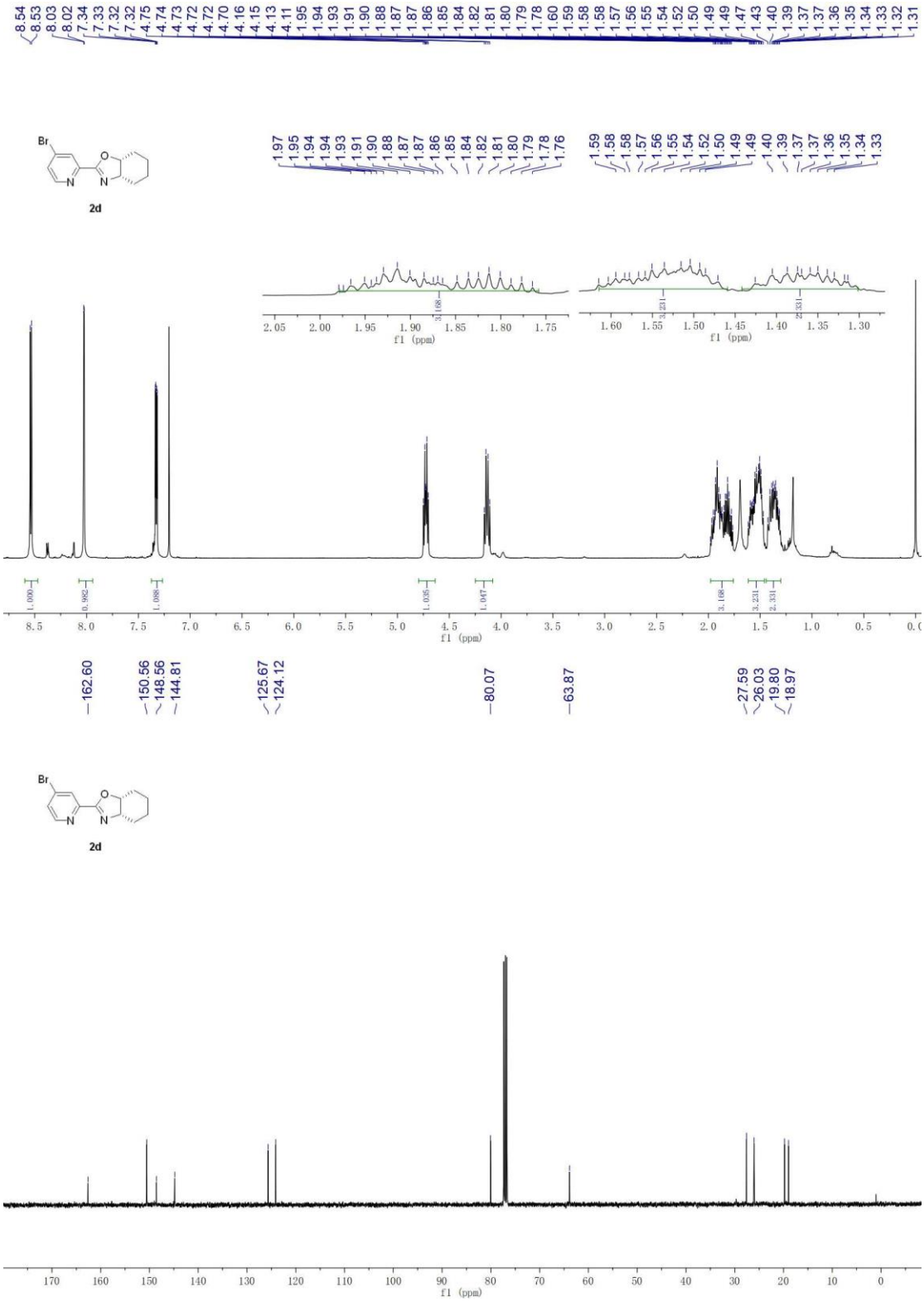
19.80
18.97
Cl
O
N
N
2c
f1 (ppm)

2e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Br
O
N
N
2d
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1.59 1.58 1.58 1.57 1.56 1.55 1.54 1.52 1.50 1.49 1.49 1.40 1.39 1.37 1.37 1.36 1.35 1.34 1.33
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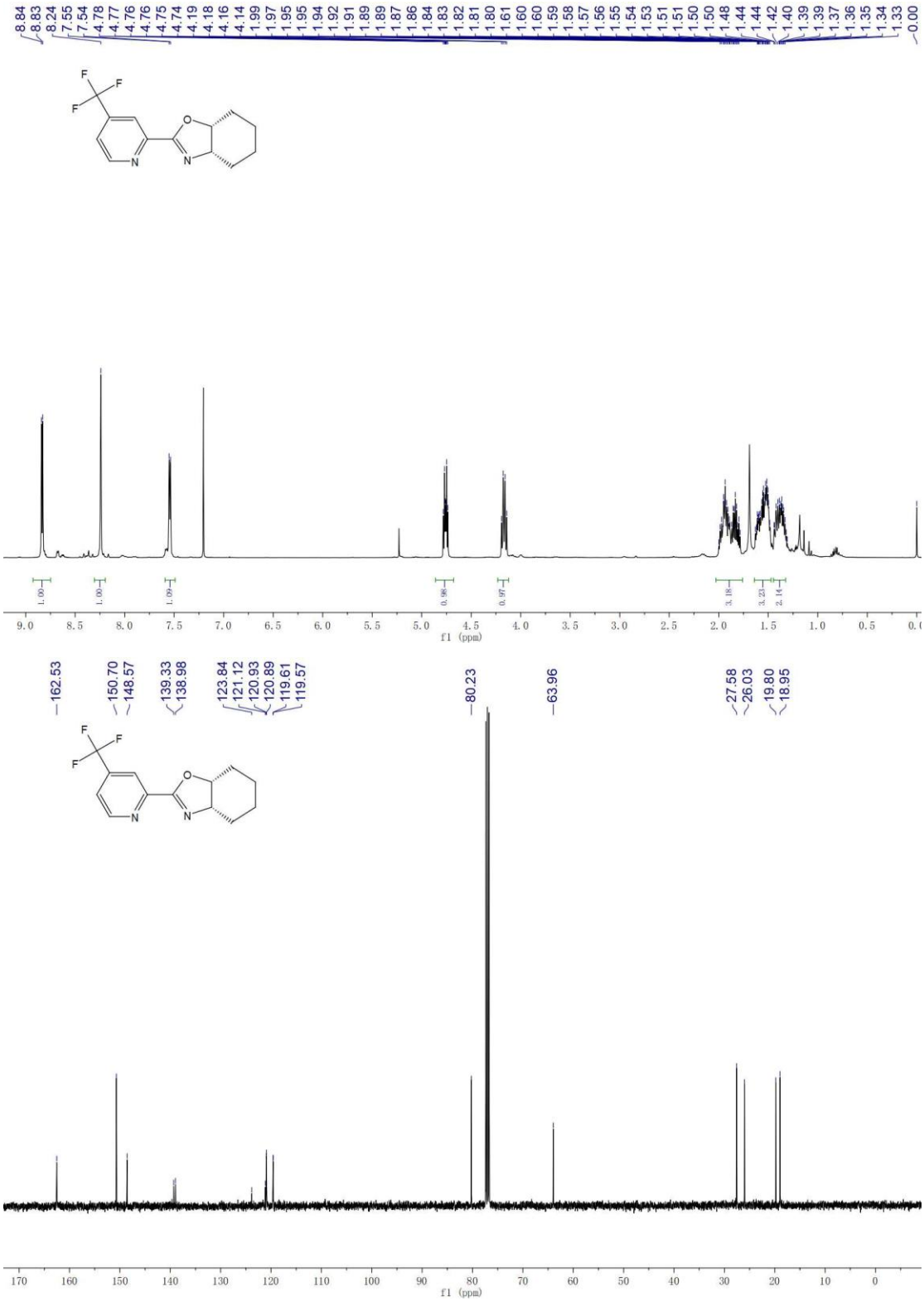
2.231
2.331
f1 (ppm)
1.000 0.982 1.088 1.035 1.047 3.168 3.231 2.331
f1 (ppm)
162.60 150.56 148.56 144.81 125.67 124.12 80.07 63.87 27.59 26.03 19.80 18.97
Br
O
N
N
2d
f1 (ppm)

2f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2.0
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f1 (ppm)
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150.70
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120.89
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119.57
80.23
63.96
27.58
26.03
19.80
18.95
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O
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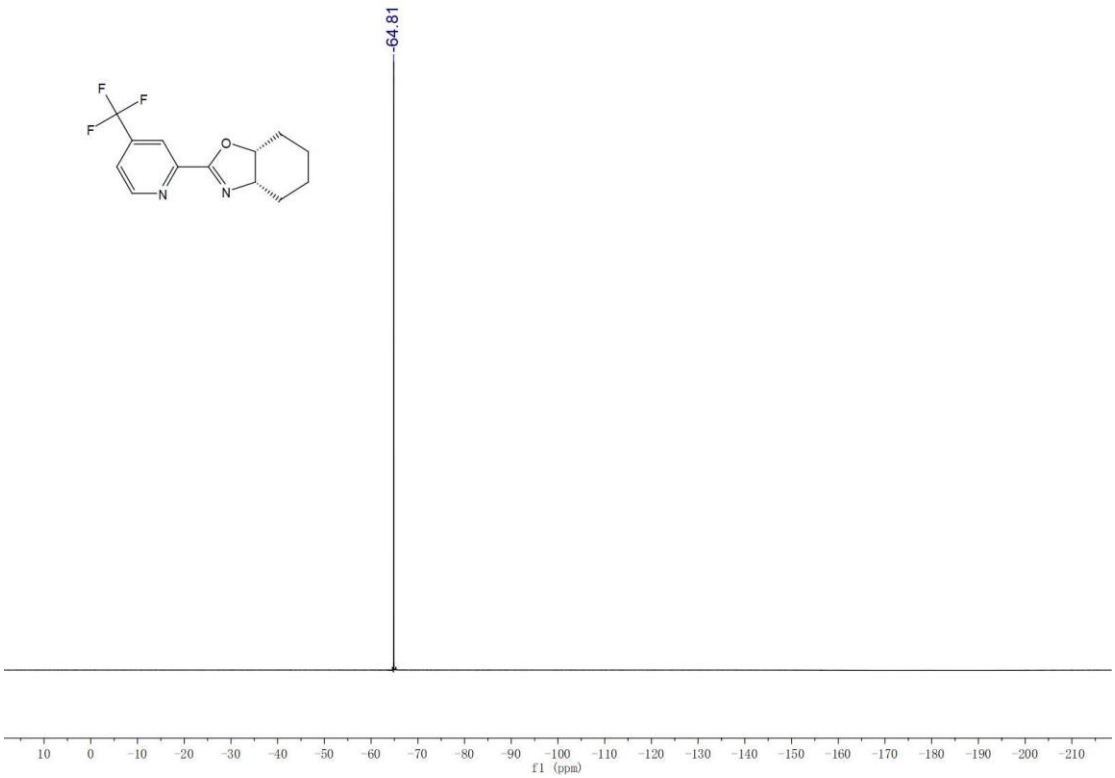
f1 (ppm)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-140
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-190
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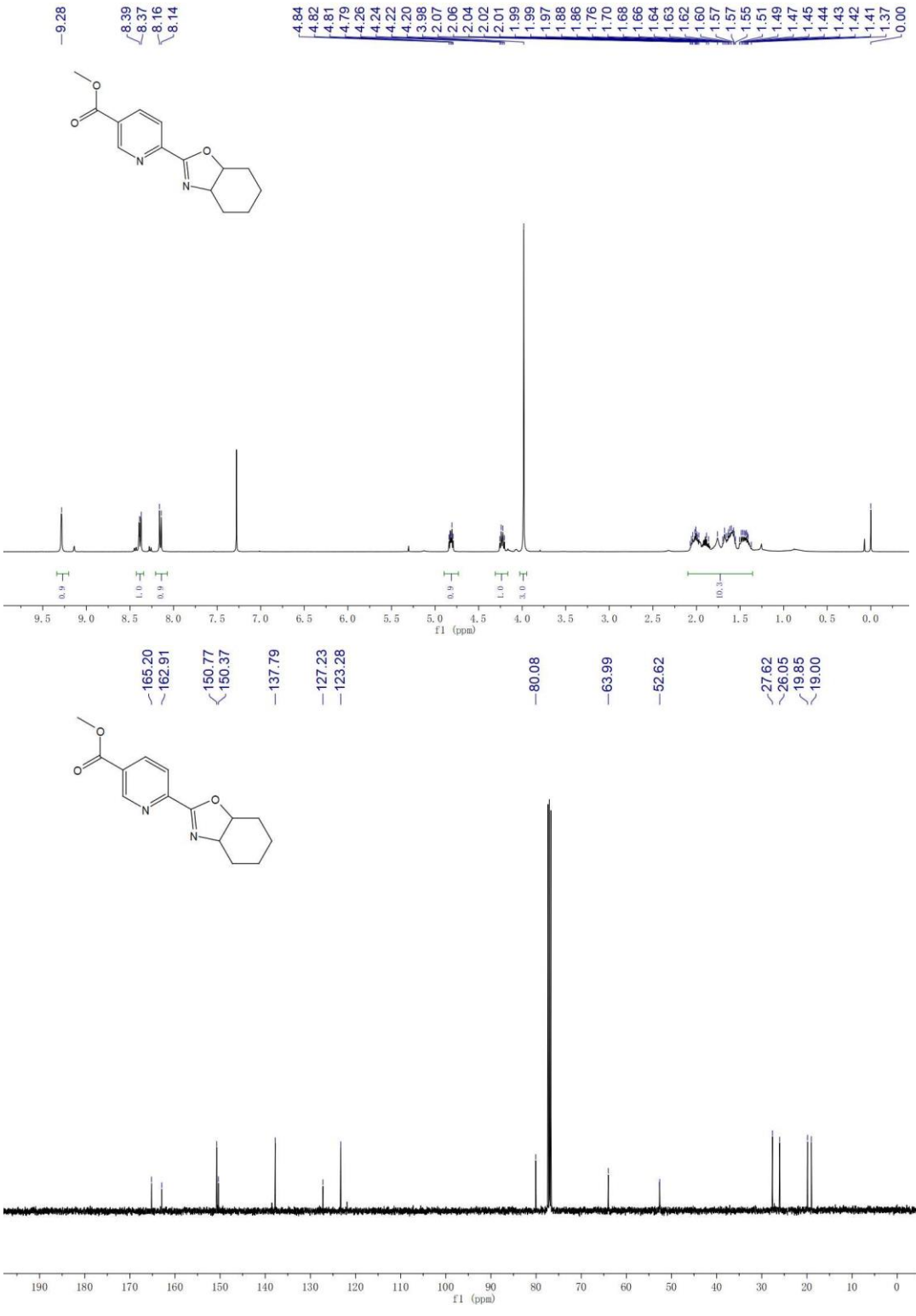
-210
f1 (ppm)

2g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2.07
2.06
2.04
2.02
2.01
1.99
1.99
1.97
1.88
1.86
1.76
1.70
1.68
1.66
1.64
1.63
1.62
1.60
1.57
1.57
1.55
1.51
1.49
1.47
1.45
1.44
1.43
1.42
1.41
1.37
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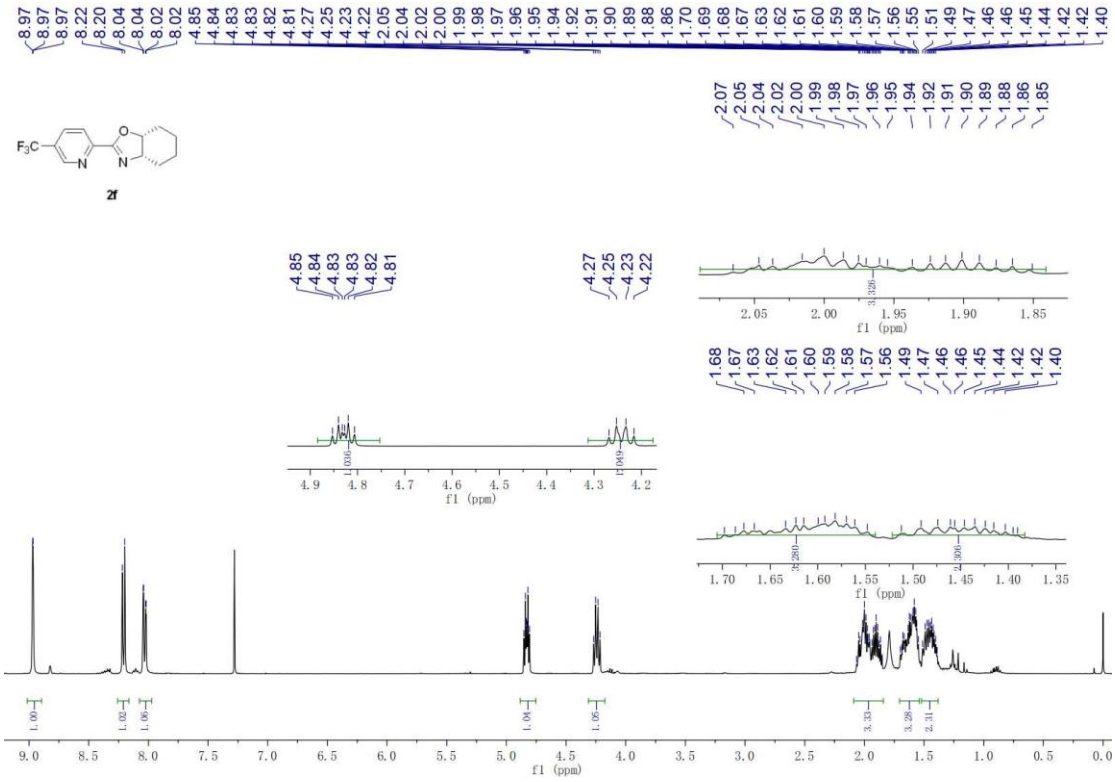
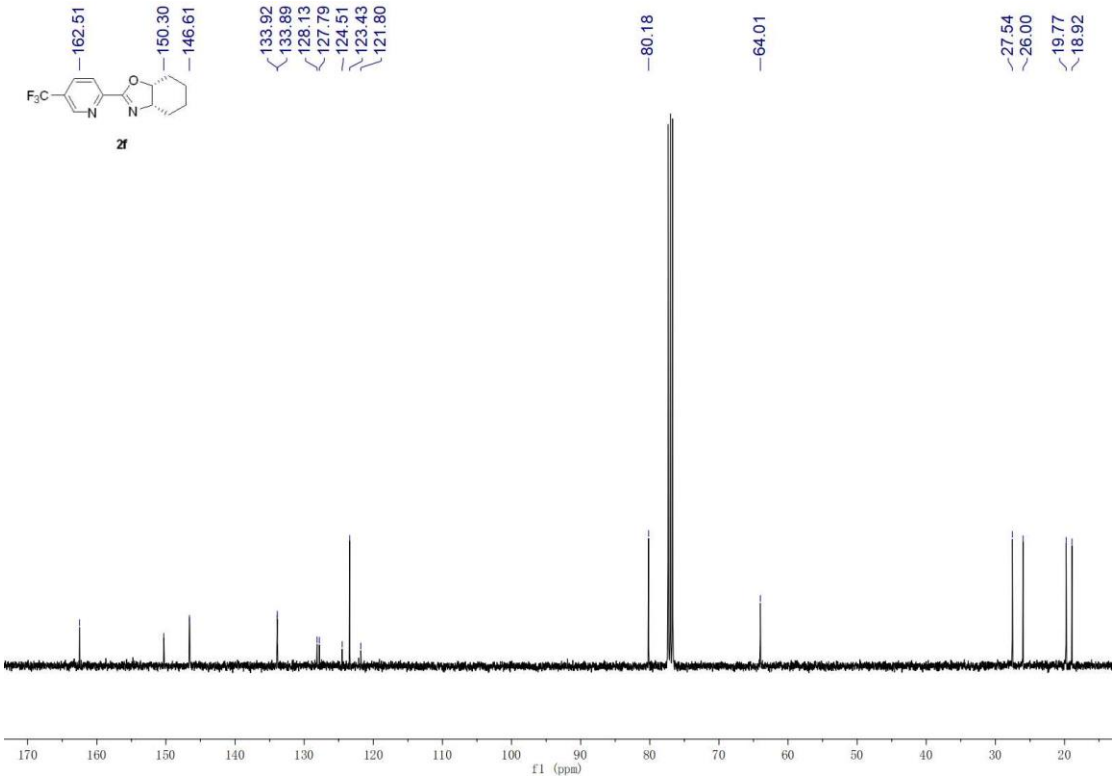
f1 (ppm)
165.20
162.91
150.77
150.37
137.79
127.23
123.28
80.08
63.99
52.62
27.62
26.05
19.85
19.00
f1 (ppm)

2h

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80.18
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26.00
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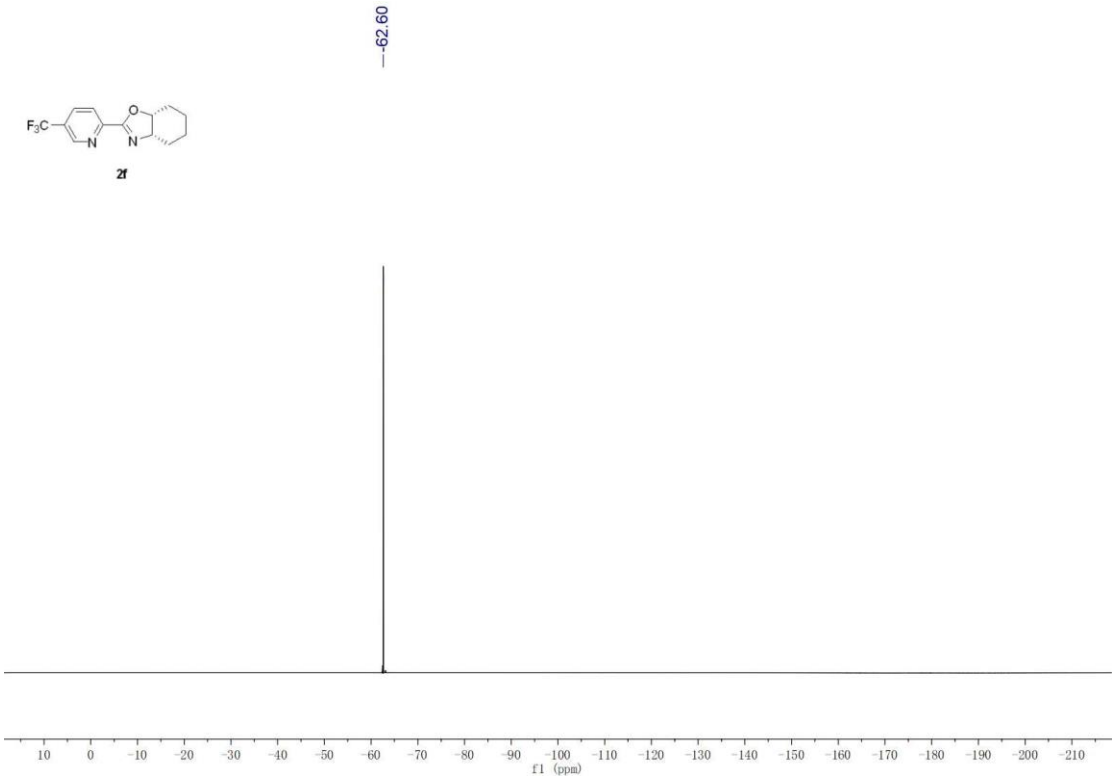
18.92
F3C
N
O
N
2f
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)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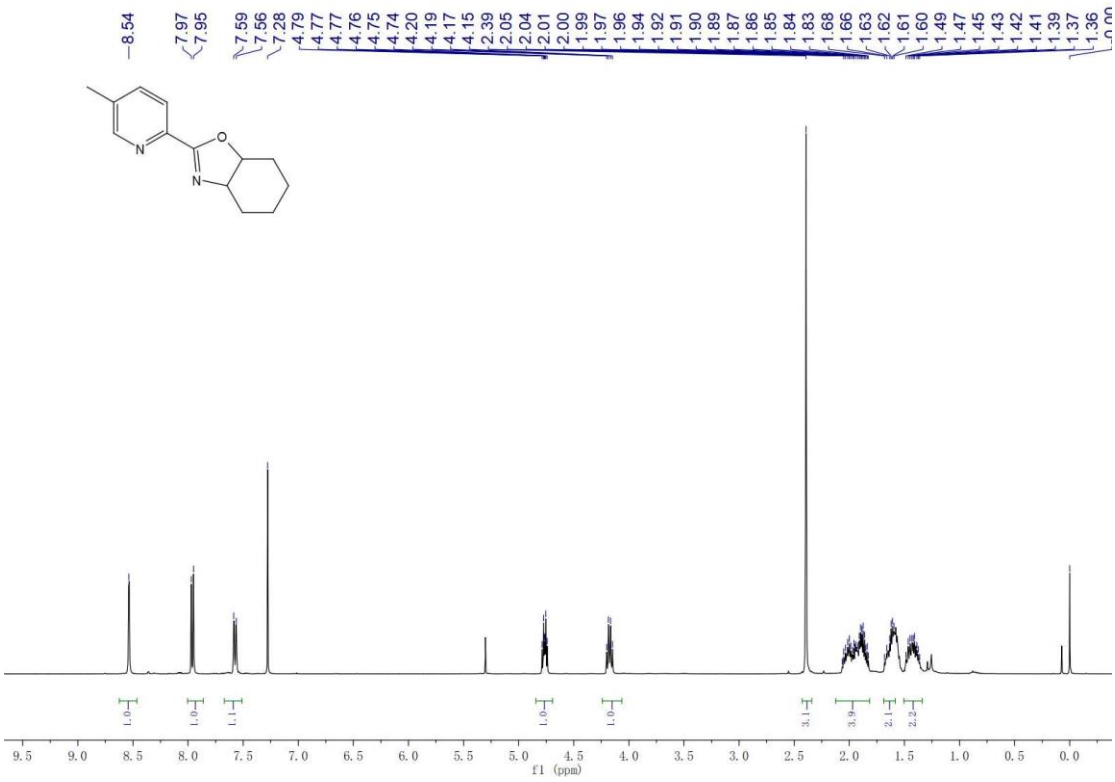
F3C
N
O
N
2f
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

2i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4.76
4.75
4.74
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4.19
4.17
4.15
2.39
2.05
2.04
2.01
2.00
1.99
1.97
1.96
1.94
1.92
1.91
1.90
1.89
1.87
1.86
1.85
1.84
1.83
1.68
1.66
1.63
1.62
1.61
1.60
1.49
1.47
1.45
1.43
1.42
1.41
1.39
1.37
1.36
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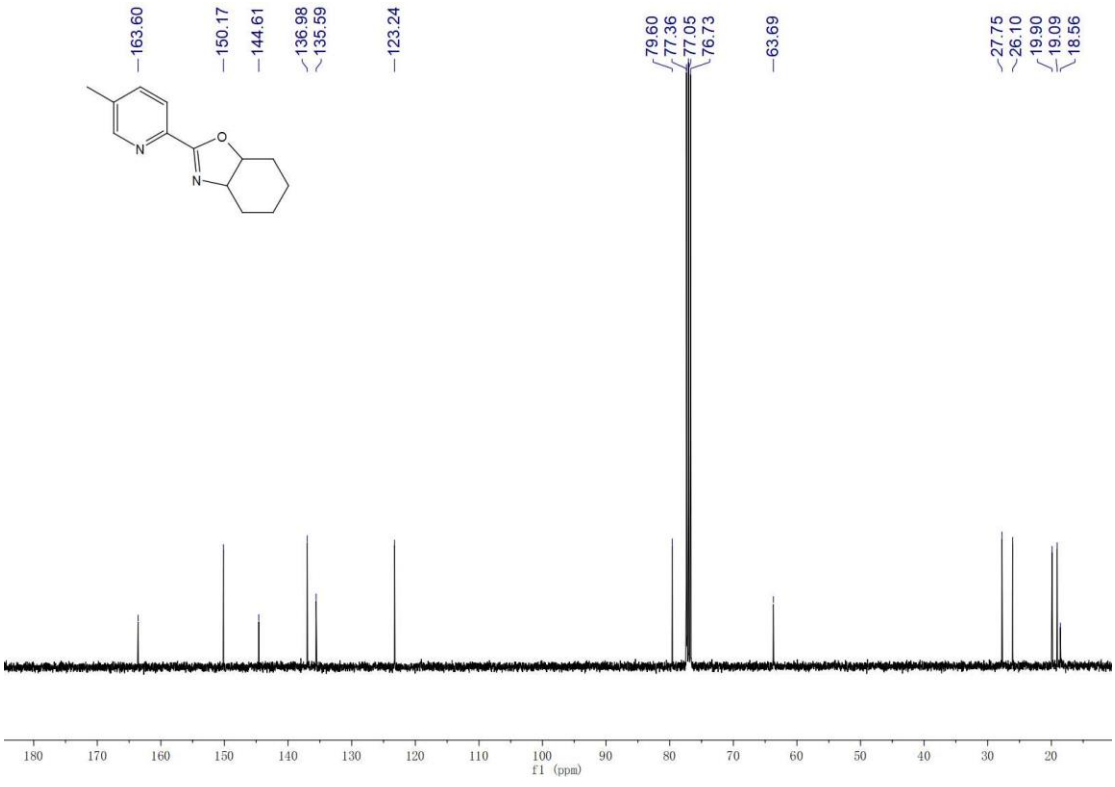
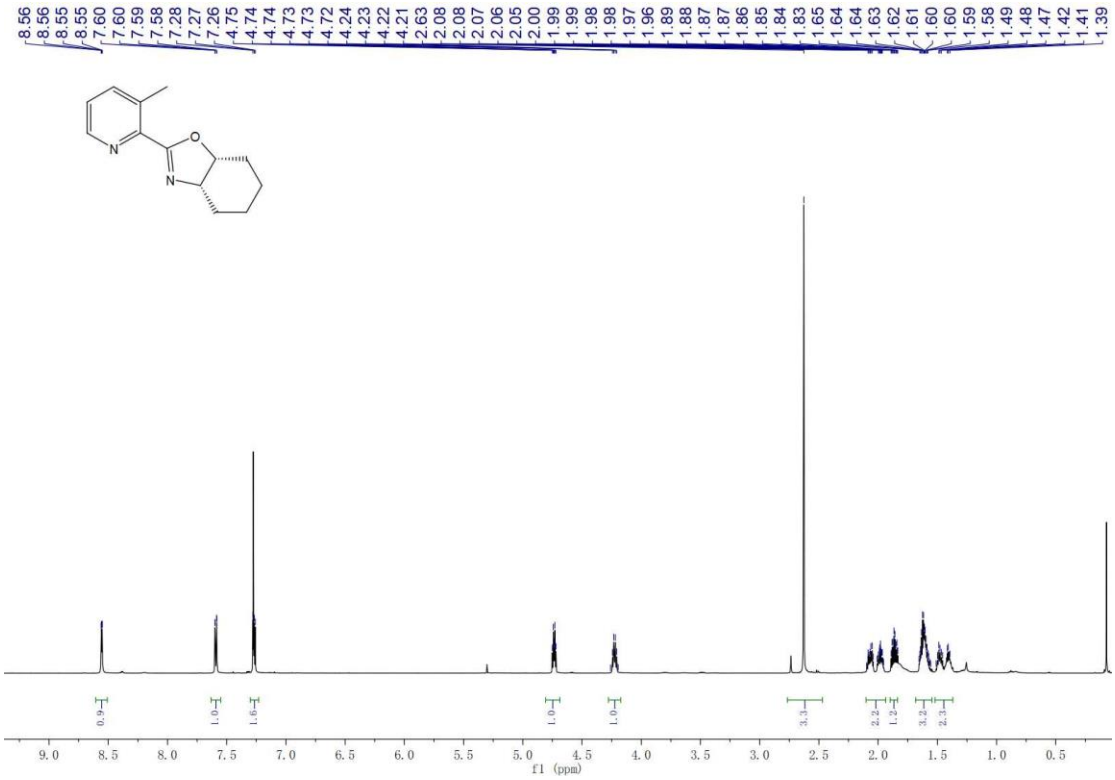
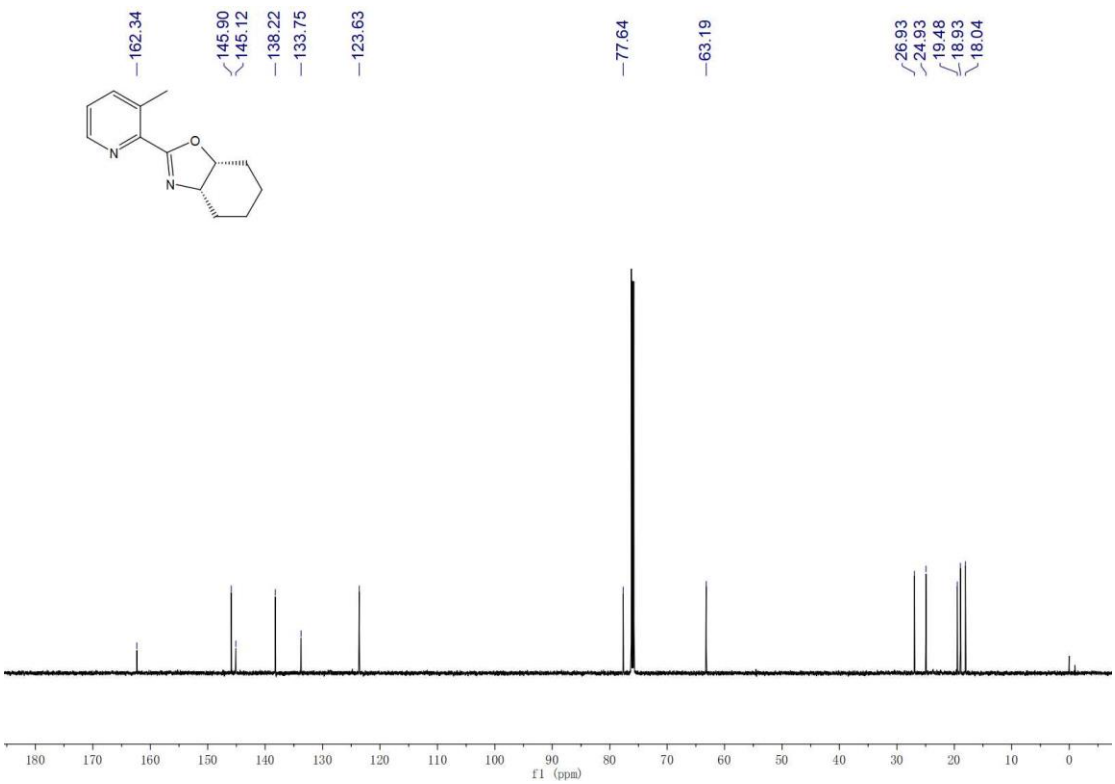
1.0
1.1
1.0
1.0
3.1
3.9
2.1
2.2
f1 (ppm)

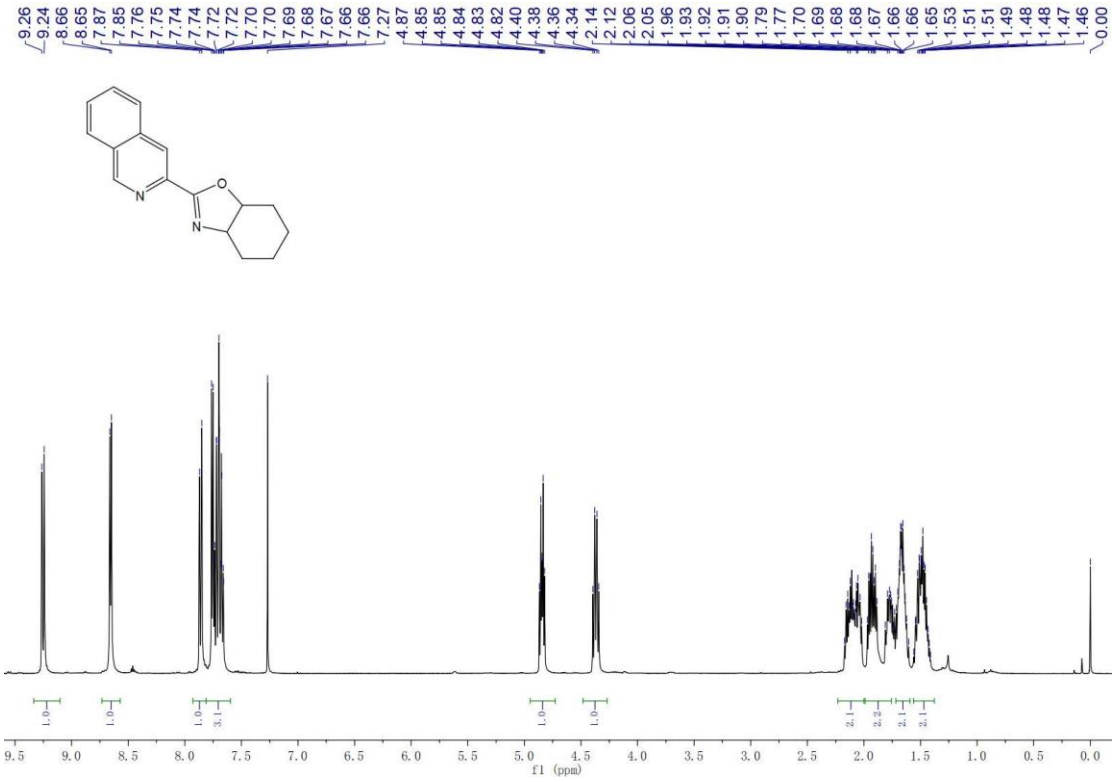

163.60
150.17
144.61
136.98
135.59
123.24
79.60
77.36
77.05
76.73
63.69
27.75
26.10
19.90
19.09
18.56
f1 (ppm)

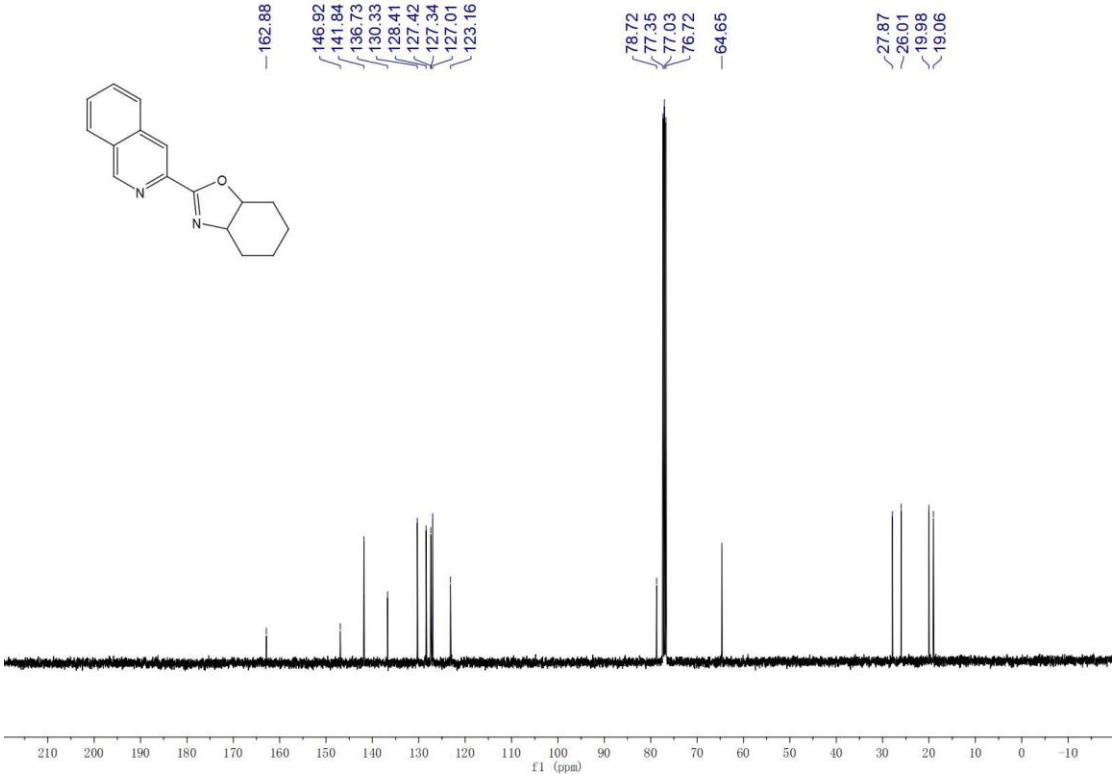
2j


8.56 8.56 8.55 8.55 7.60 7.60 7.59 7.58 7.28 7.27 7.26 4.75 4.74 4.74 4.73 4.73 4.72 4.24 4.23 4.22 4.21 2.63 2.08 2.08 2.07 2.06 2.05 2.00 1.99 1.99 1.98 1.98 1.97 1.96 1.89 1.88 1.87 1.87 1.86 1.85 1.84 1.83 1.65 1.64 1.64 1.63 1.62 1.61 1.60 1.60 1.59 1.58 1.49 1.48 1.47 1.42 1.41 1.39
0.9 1.0 1.6 1.0 1.0 3.3 2.2 1.2 3.2 2.3
f1 (ppm)

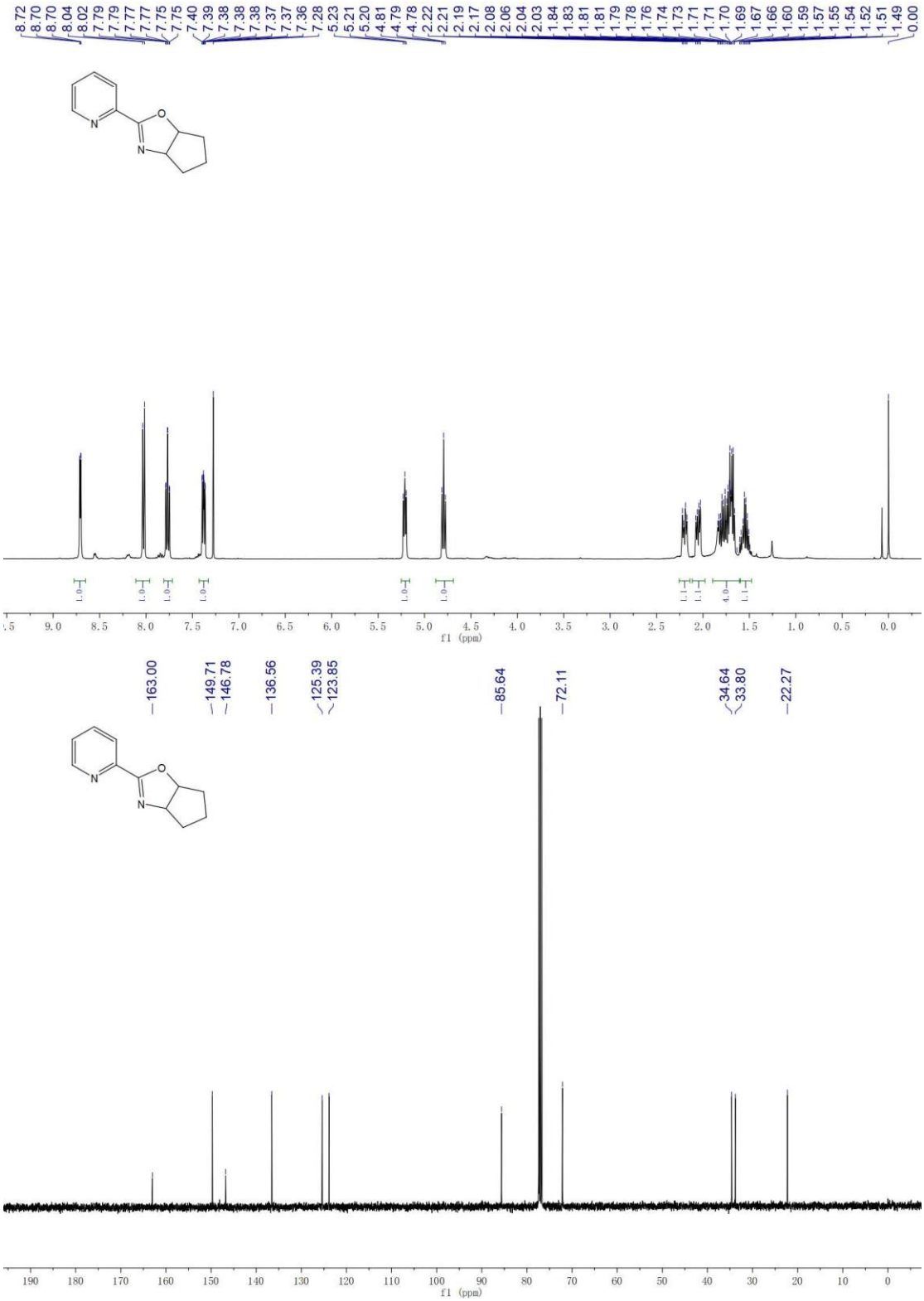

162.34
145.90
145.12
138.22
133.75
123.63
77.64
63.19
26.93
24.93
19.48
18.93
18.04
f1 (ppm)

2k

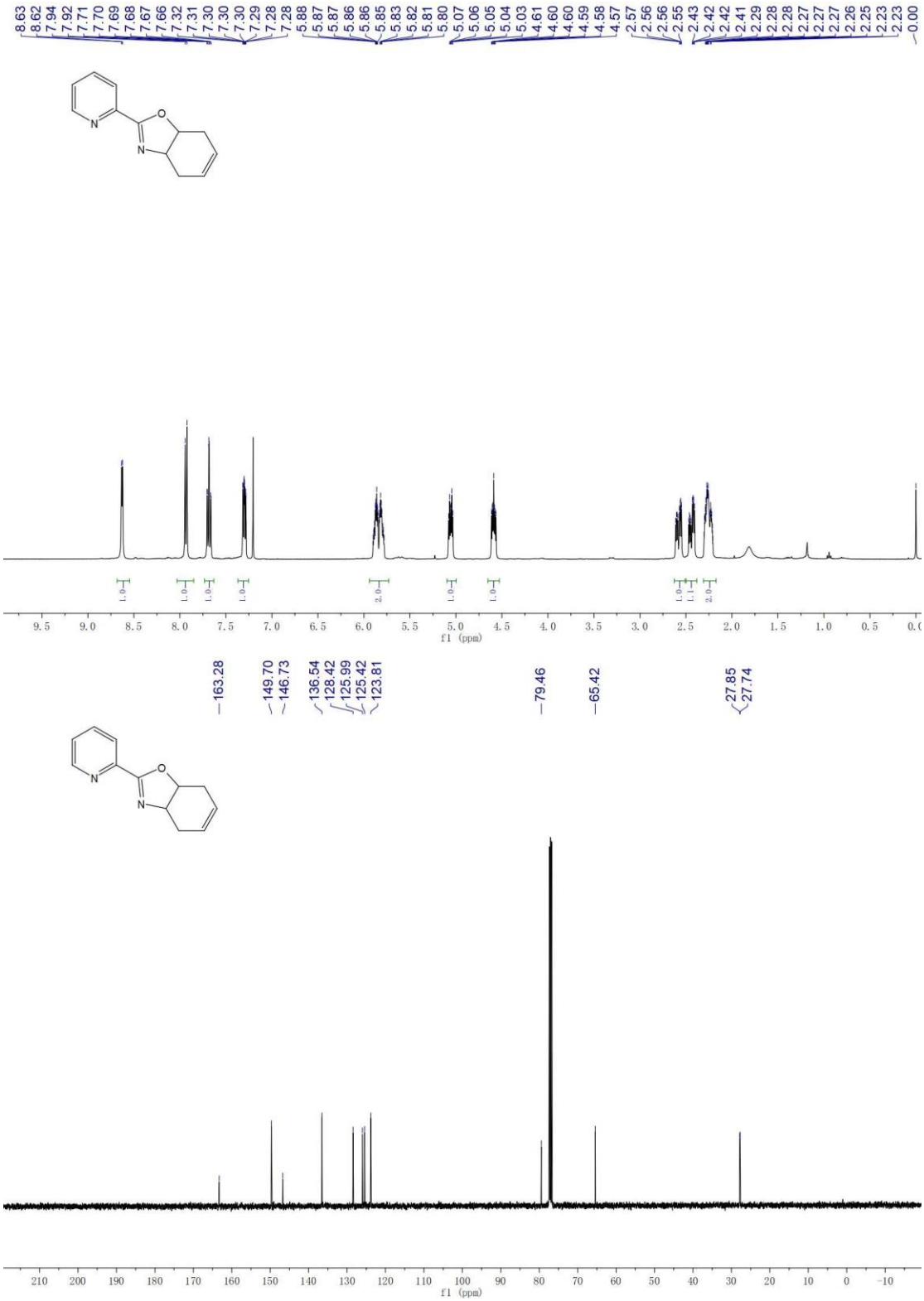

9.26
9.24
8.66
8.65
8.87
7.85
7.76
7.75
7.74
7.74
7.72
7.72
7.70
7.70
7.69
7.68
7.67
7.66
7.66
7.27
4.87
4.85
4.85
4.84
4.83
4.82
4.40
4.38
4.36
4.34
2.14
2.12
2.06
2.05
1.96
1.93
1.92
1.91
1.90
1.79
1.77
1.70
1.69
1.68
1.68
1.67
1.66
1.66
1.65
1.53
1.51
1.51
1.49
1.48
1.48
1.47
1.46
0.00
N
O
N
1.0
1.0
1.0
3.1
1.0
1.0
2.1
2.2
2.1
2.1
f1 (ppm)


162.88
146.92
141.84
136.73
130.33
128.41
127.42
127.34
127.01
123.16
78.72
77.35
77.03
76.72
64.65
27.87
26.01
19.98
19.06
N
O
N
f1 (ppm)

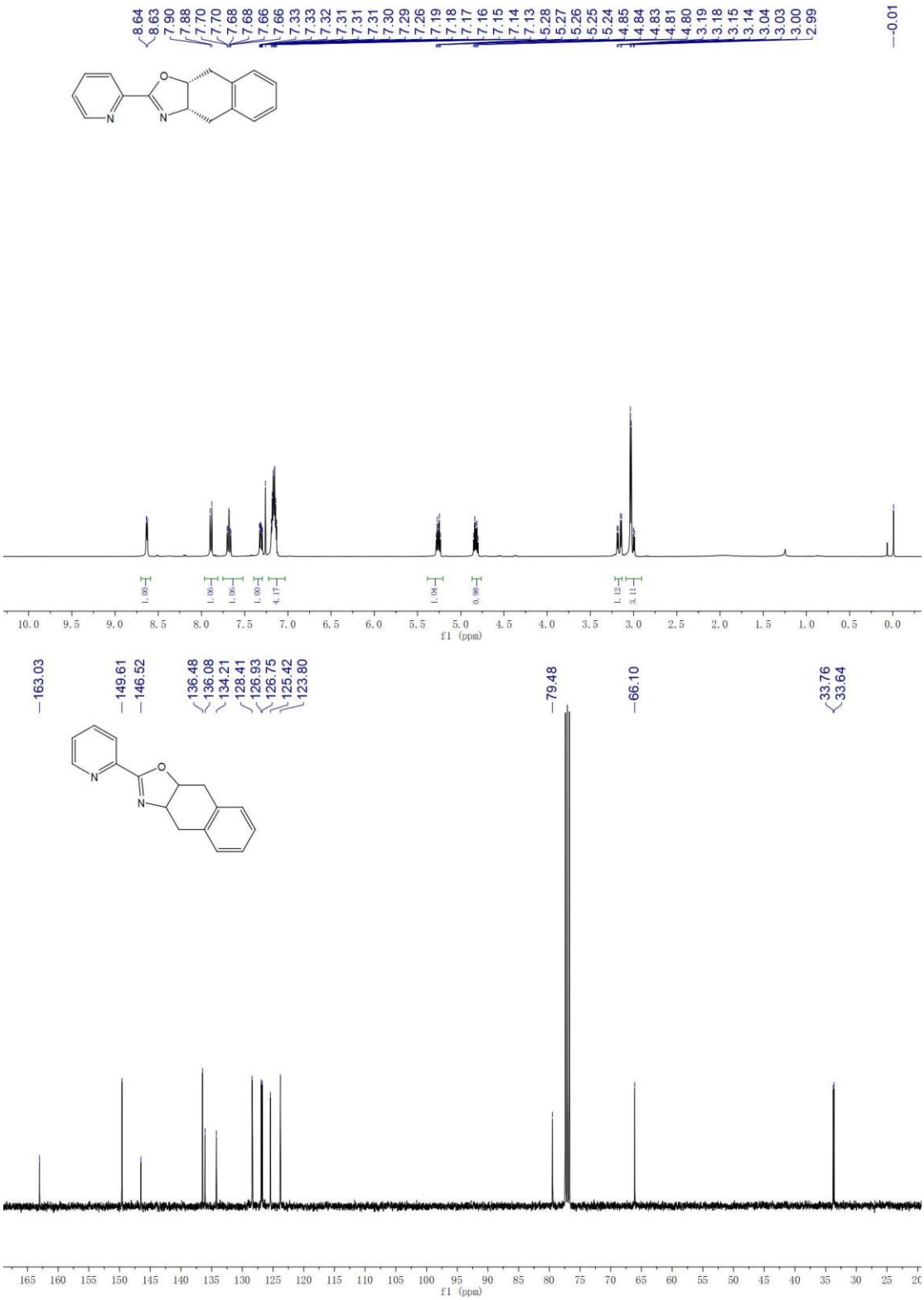
2l


8.72
8.70
8.70
8.04
8.02
7.79
7.79
7.77
7.77
7.75
7.75
7.40
7.39
7.38
7.38
7.38
7.37
7.37
7.36
7.28
5.23
5.21
5.20
4.81
4.79
4.78
2.22
2.21
2.19
2.17
2.08
2.06
2.04
2.03
1.84
1.83
1.81
1.81
1.79
1.78
1.76
1.74
1.73
1.71
1.71
1.70
1.69
1.67
1.66
1.60
1.59
1.57
1.55
1.54
1.52
1.51
1.49
0.00
f1 (ppm)
163.00
149.71
146.78
136.56
125.39
123.85
85.64
72.11
34.64
33.80
22.27
f1 (ppm)

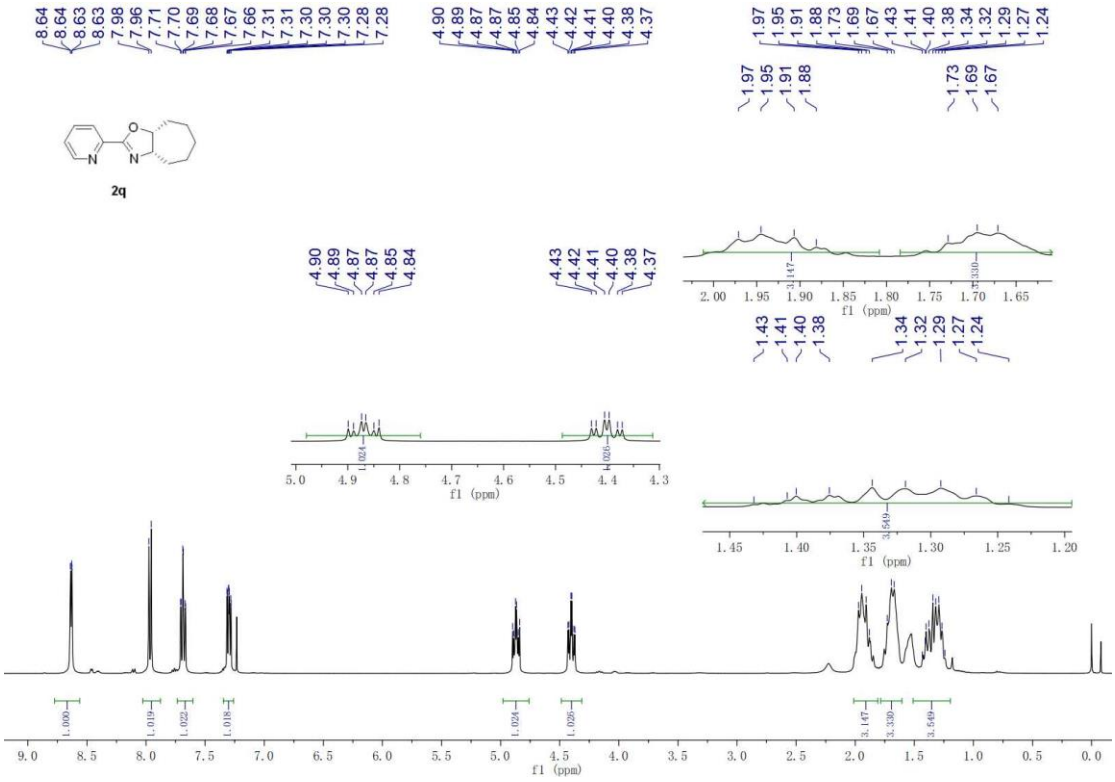
2m

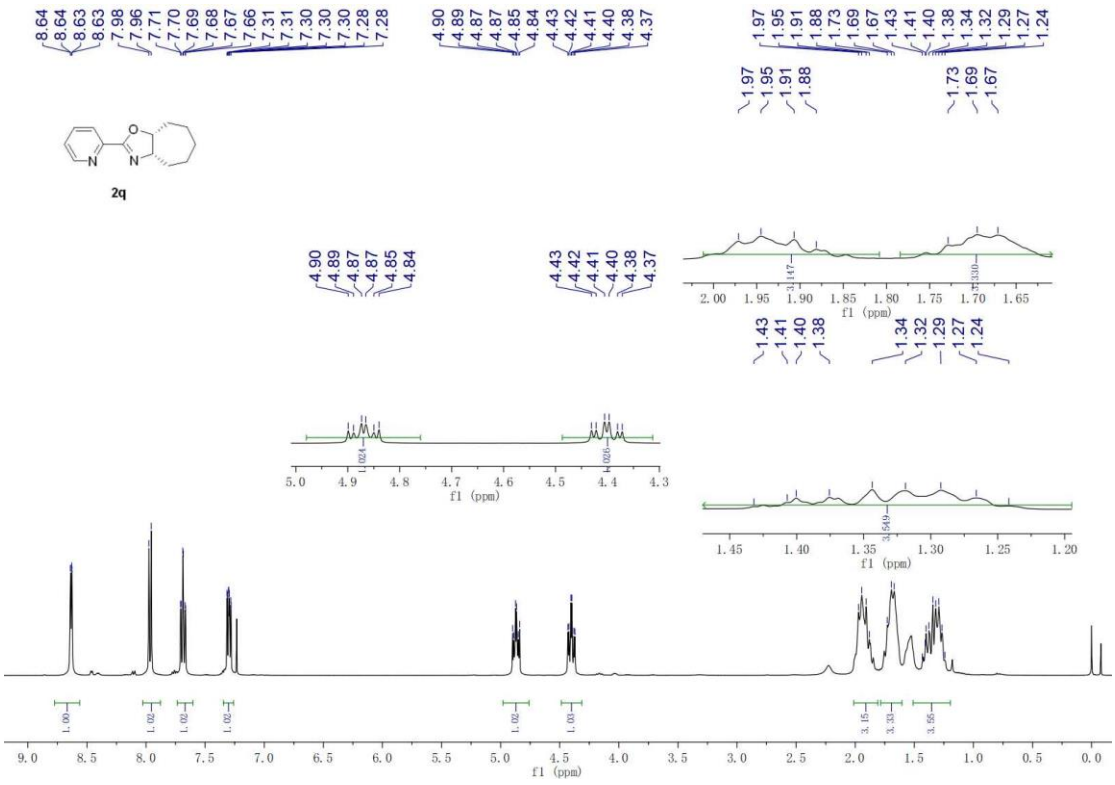

8.63
8.62
7.94
7.92
7.71
7.70
7.69
7.68
7.67
7.66
7.32
7.31
7.30
7.30
7.30
7.29
7.28
7.28
5.88
5.87
5.87
5.86
5.86
5.85
5.83
5.82
5.81
5.80
5.07
5.06
5.05
5.04
5.03
4.61
4.60
4.60
4.59
4.58
4.57
2.57
2.56
2.56
2.55
2.43
2.42
2.42
2.41
2.29
2.28
2.28
2.27
2.27
2.27
2.26
2.25
2.23
2.23
0.00
f1 (ppm)
163.28
149.70
146.73
136.54
128.42
125.99
125.42
123.81
79.46
65.42
27.85
27.74
f1 (ppm)

2n

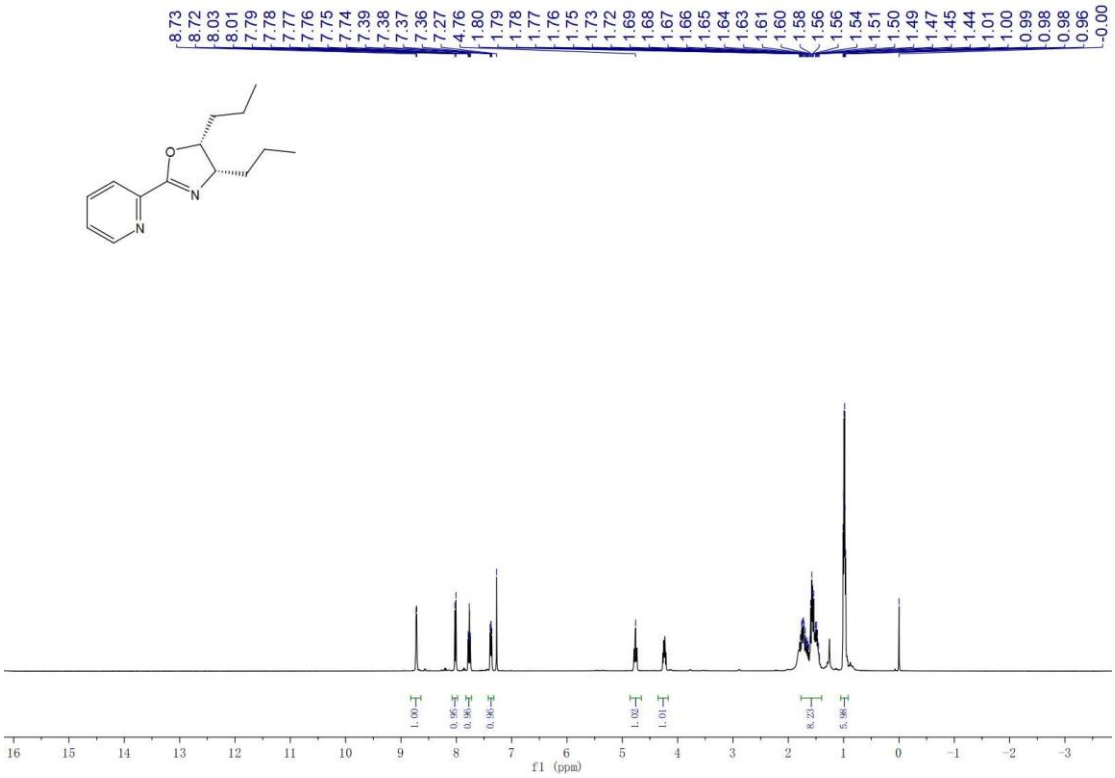

8.64
8.63
7.90
7.88
7.70
7.70
7.68
7.68
7.66
7.66
7.33
7.33
7.32
7.31
7.31
7.31
7.30
7.29
7.26
7.19
7.18
7.17
7.16
7.15
7.14
7.13
5.28
5.27
5.26
5.25
5.24
4.85
4.84
4.83
4.81
4.80
3.19
3.18
3.15
3.14
3.04
3.03
3.00
2.99
-0.01
1.00
1.06
1.06
1.00
4.17
1.04
0.98
1.12
3.11
f1 (ppm)
163.03
149.61
146.52
136.48
136.08
134.21
128.41
126.93
126.75
125.42
123.80
79.48
66.10
33.76
33.64
f1 (ppm)

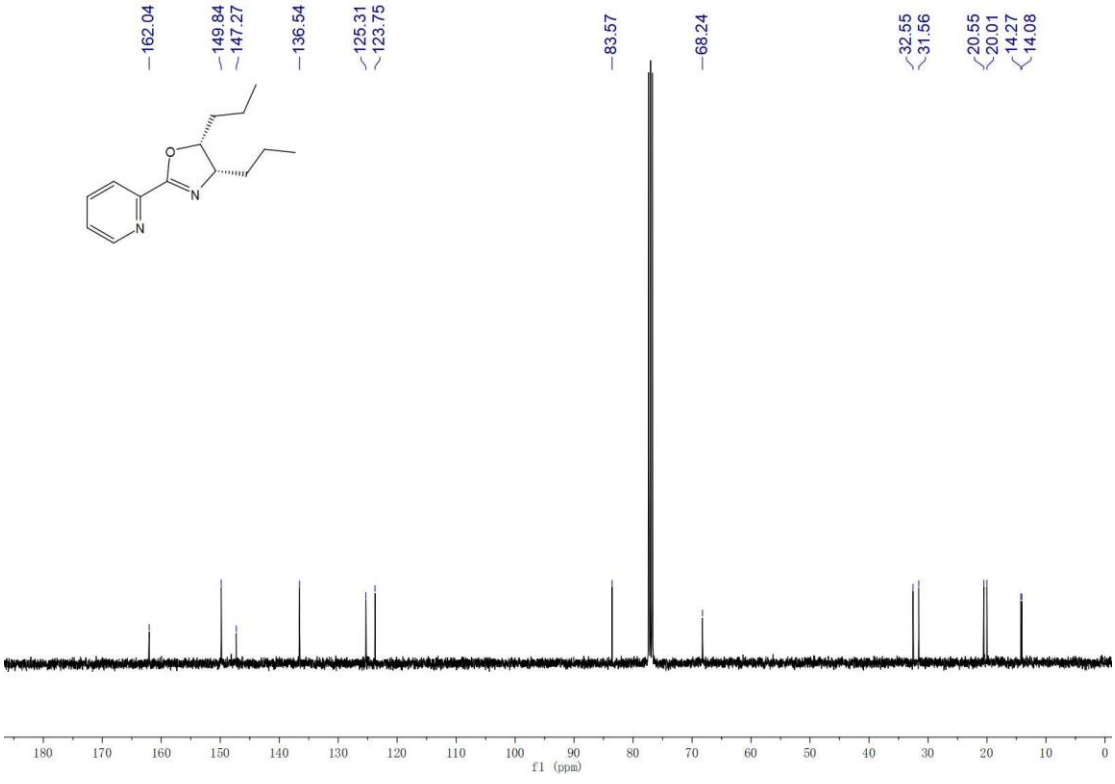
2o


8.64 8.64 8.63 8.63 7.98 7.96 7.71 7.70 7.69 7.68 7.67 7.66 7.31 7.31 7.30 7.30 7.30 7.28 7.28
4.90 4.89 4.87 4.87 4.85 4.84 4.43 4.42 4.41 4.40 4.38 4.37
1.97 1.95 1.91 1.88 1.73 1.69 1.67 1.43 1.41 1.40 1.38 1.34 1.32 1.29 1.27 1.24
2q
f1 (ppm)
1.000 1.019 1.022 1.018 1.024 1.029 3.147 3.330 3.549

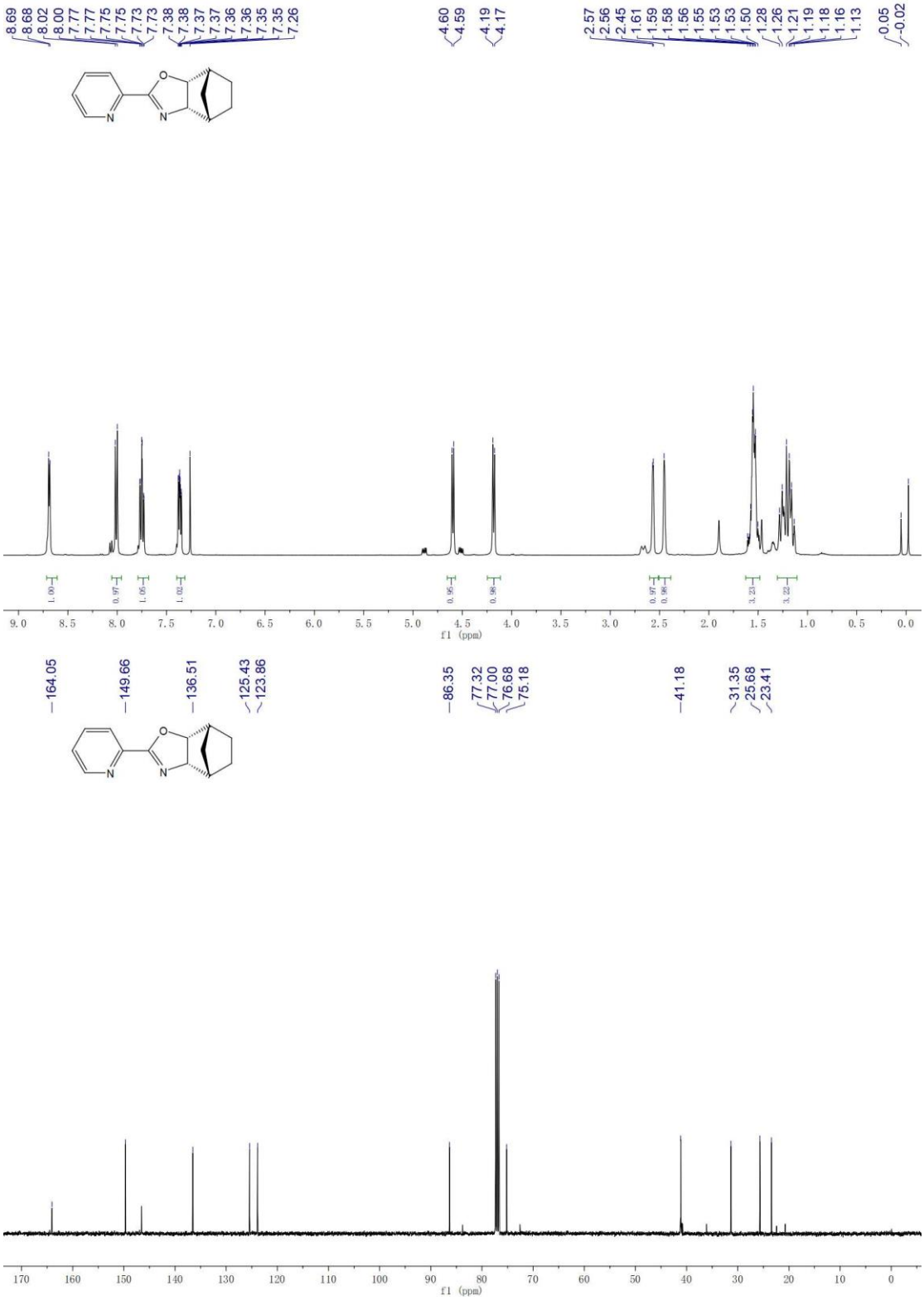

8.64 8.64 8.63 8.63 7.98 7.96 7.71 7.70 7.69 7.68 7.67 7.66 7.31 7.31 7.30 7.30 7.30 7.28 7.28
4.90 4.89 4.87 4.87 4.85 4.84 4.43 4.42 4.41 4.40 4.38 4.37
1.97 1.95 1.91 1.88 1.73 1.69 1.67 1.43 1.41 1.40 1.38 1.34 1.32 1.29 1.27 1.24
2q
f1 (ppm)
1.00 1.02 1.02 1.02 1.02 1.03 3.15 3.33 3.65

2p

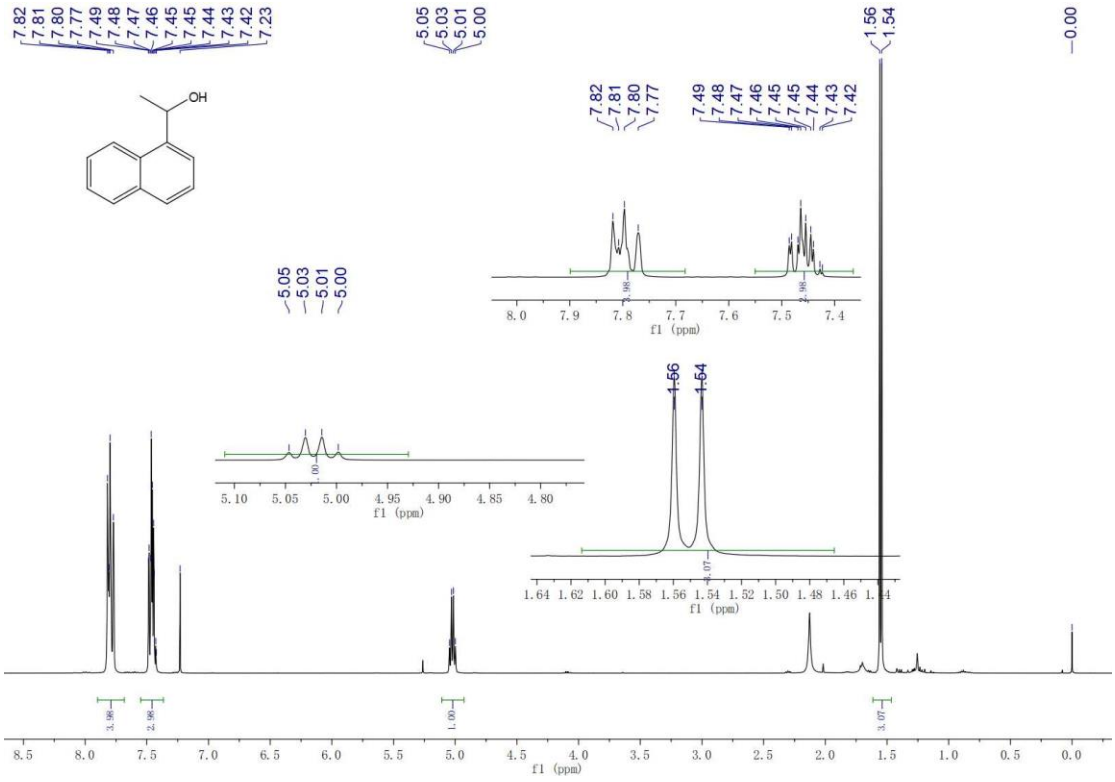

8.73
8.72
8.03
8.01
7.79
7.78
7.77
7.76
7.75
7.74
7.39
7.38
7.37
7.36
7.27
4.76
1.80
1.79
1.78
1.77
1.76
1.75
1.73
1.72
1.69
1.68
1.67
1.66
1.65
1.64
1.63
1.61
1.60
1.58
1.56
1.56
1.54
1.51
1.50
1.49
1.47
1.45
1.44
1.01
1.00
0.99
0.98
0.98
0.96
0.00
1.00
0.95
0.96
0.96
1.02
1.01
8.23
5.98
f1 (ppm)

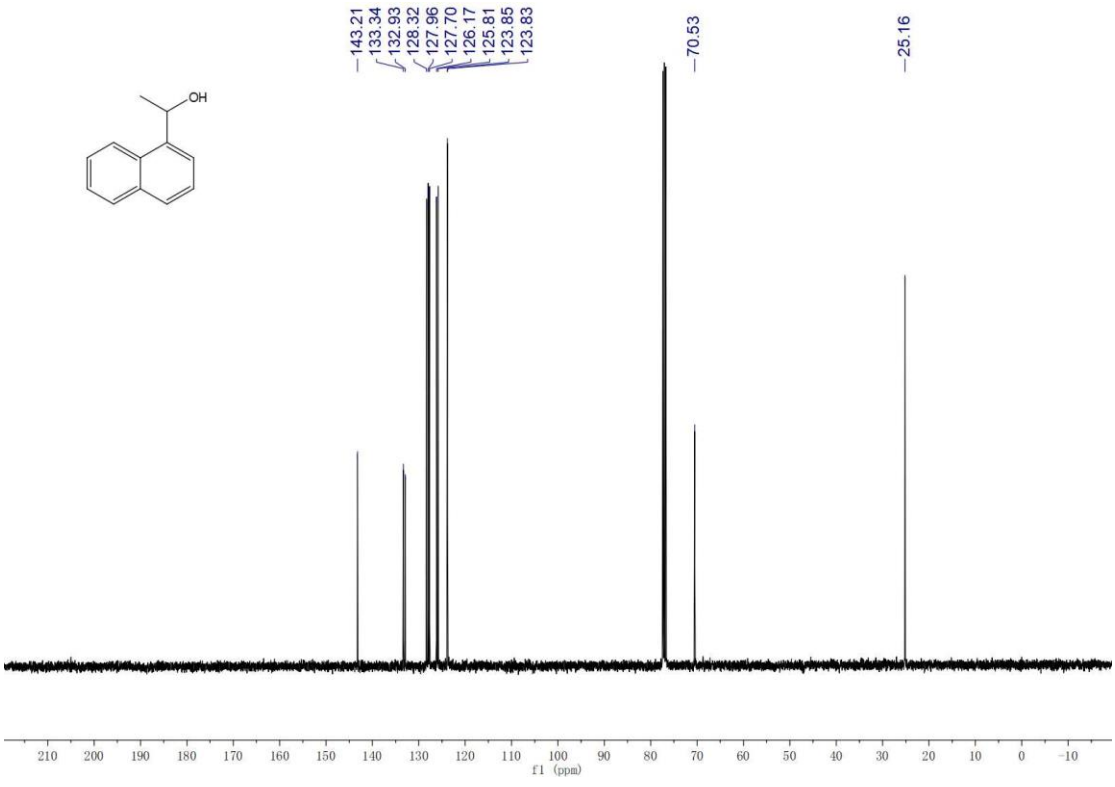

162.04
149.84
147.27
136.54
125.31
123.75
83.57
68.24
32.55
31.56
20.55
20.01
14.27
14.08
f1 (ppm)

2q

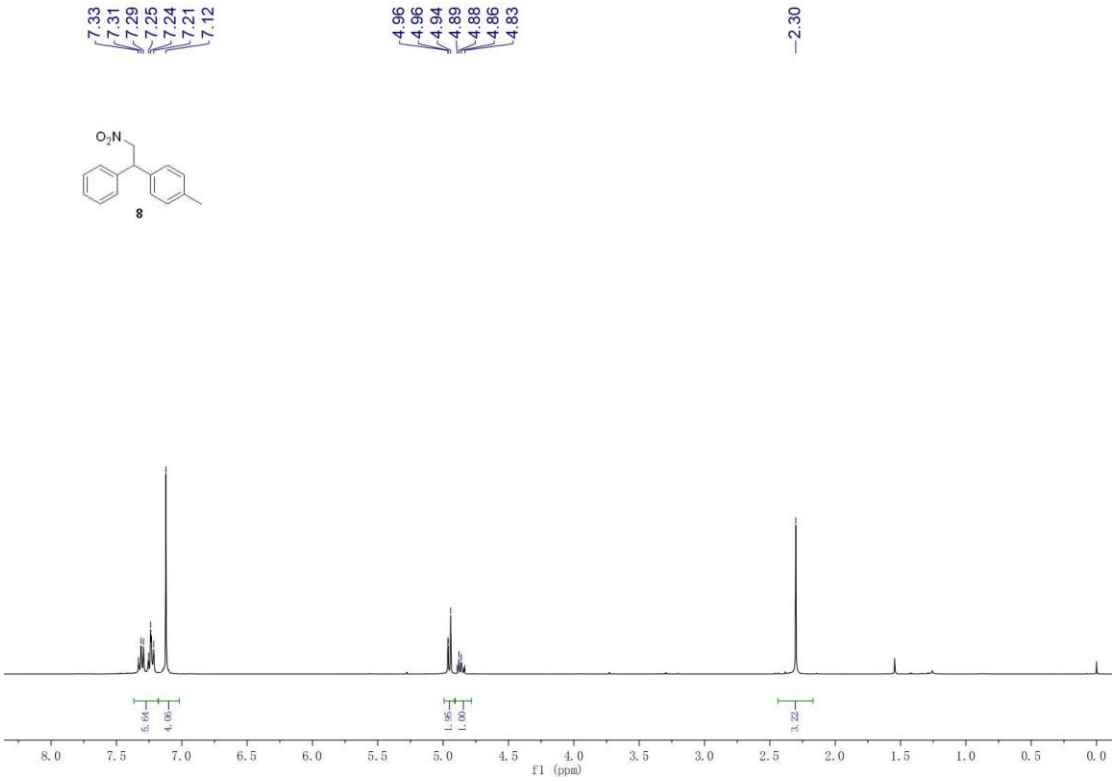

8.69
8.68
8.02
8.00
7.77
7.77
7.75
7.75
7.73
7.73
7.38
7.38
7.37
7.37
7.36
7.36
7.35
7.35
7.26
4.60
4.59
4.19
4.17
2.57
2.56
2.45
1.61
1.59
1.58
1.56
1.55
1.53
1.53
1.50
1.28
1.26
1.21
1.19
1.18
1.16
1.13
0.05
-0.02
O
N
N
1.00
0.97
1.05
1.02
0.95
0.98
0.97
0.96
3.23
3.22
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
164.05
149.66
136.51
125.43
123.86
86.35
77.32
77.00
76.68
75.18
41.18
31.35
25.68
23.41
O
N
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

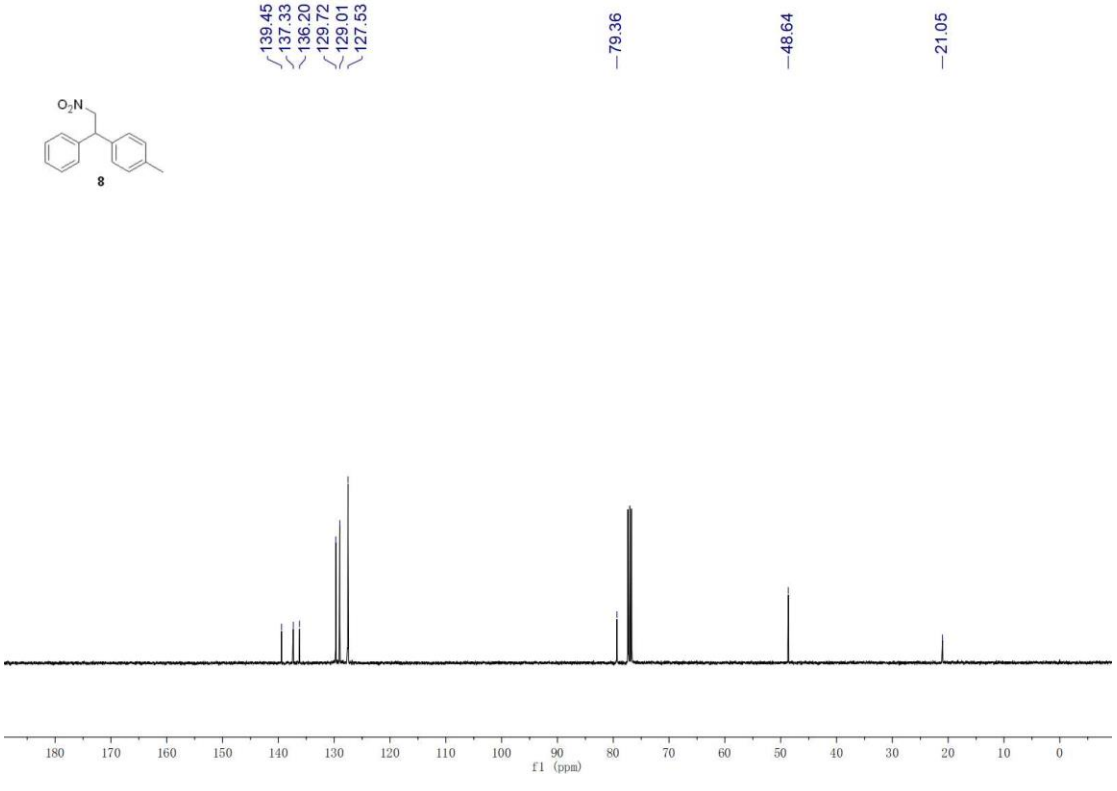
4


7.82
7.81
7.80
7.77
7.49
7.48
7.47
7.46
7.45
7.44
7.43
7.42
7.23
5.05
5.03
5.01
5.00
1.56
1.54
0.00
OH
f1 (ppm)

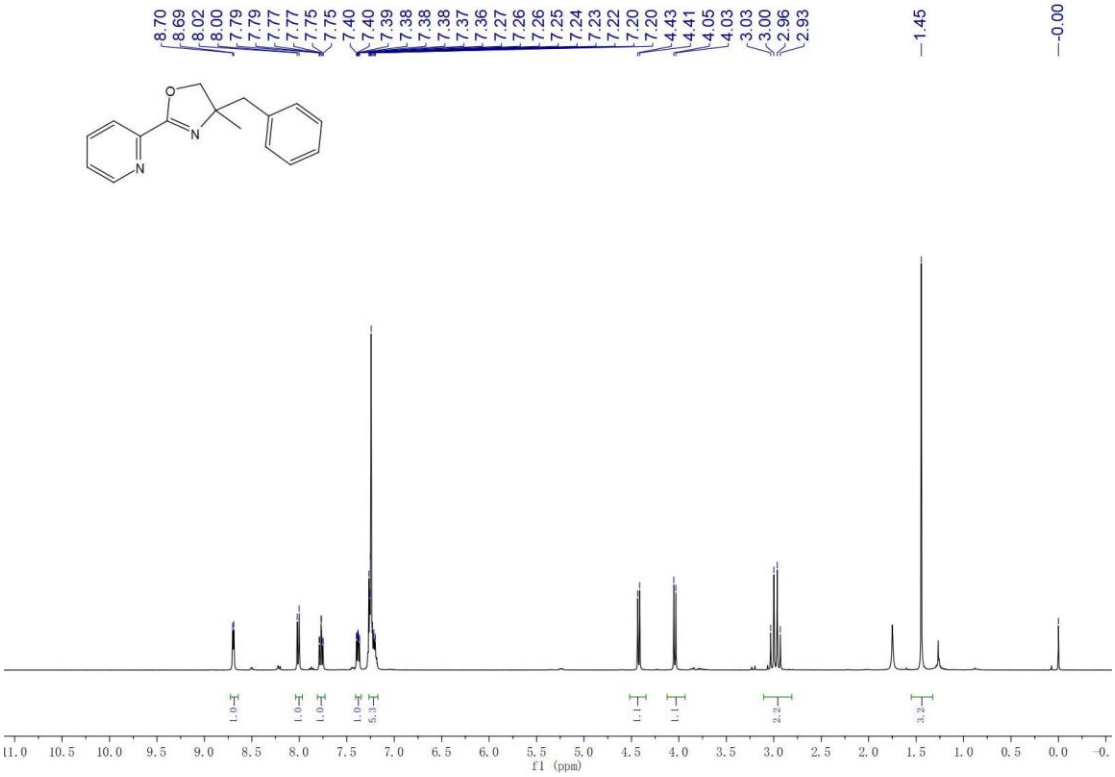

143.21
133.34
132.93
128.32
127.96
127.70
126.17
125.81
123.85
123.83
70.53
25.16
OH
f1 (ppm)

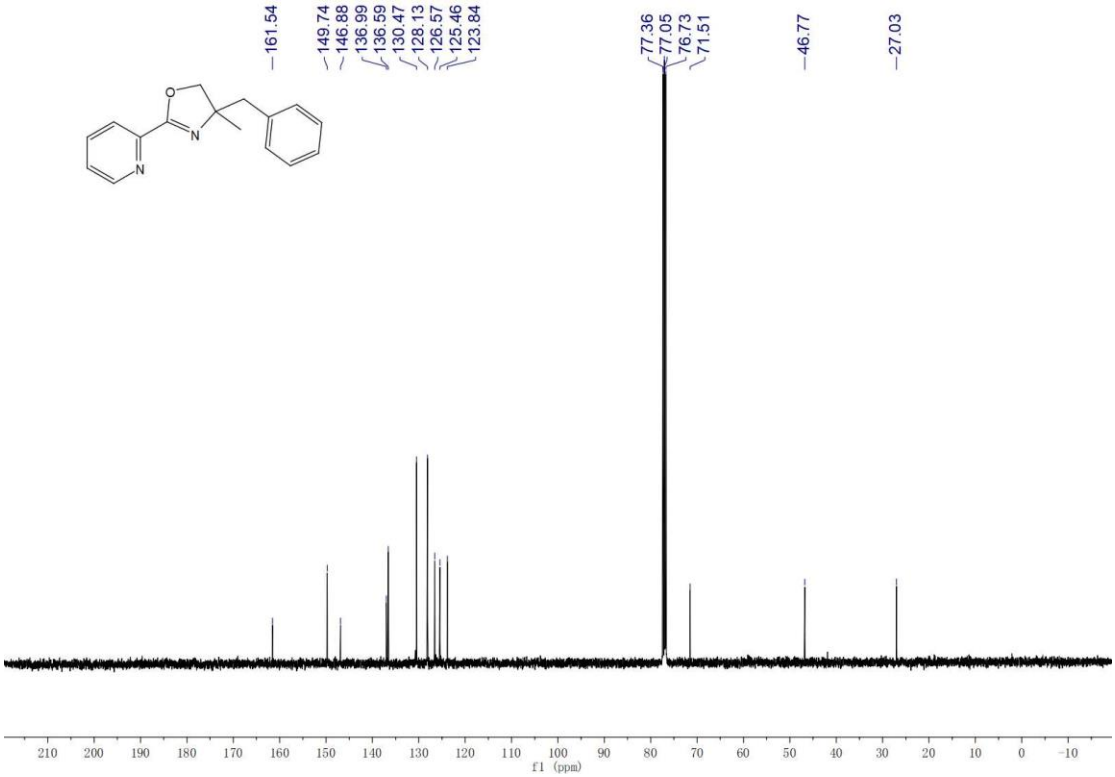
5


7.33
7.31
7.29
7.25
7.24
7.21
7.12
4.96
4.96
4.94
4.89
4.88
4.86
4.83
2.30
O2N
8
5.64
4.00
1.95
1.00
3.22
f1 (ppm)

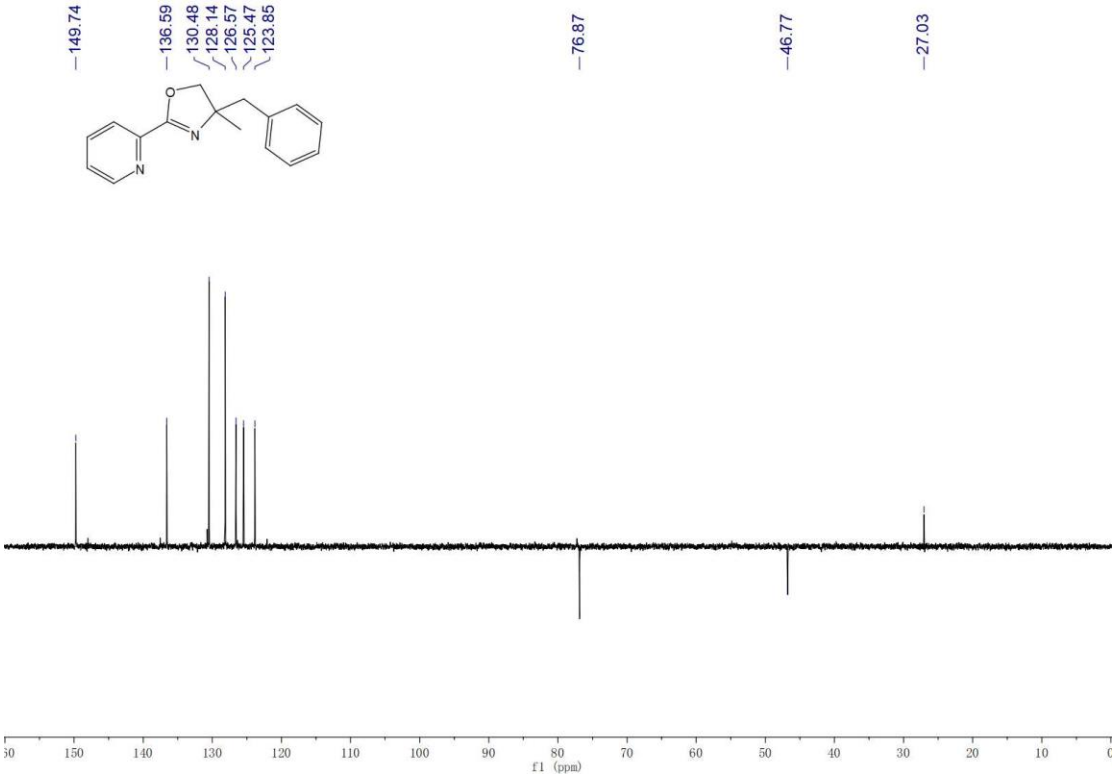

139.45
137.33
136.20
129.72
129.01
127.53
79.36
48.64
21.05
O2N
8
f1 (ppm)

2r’

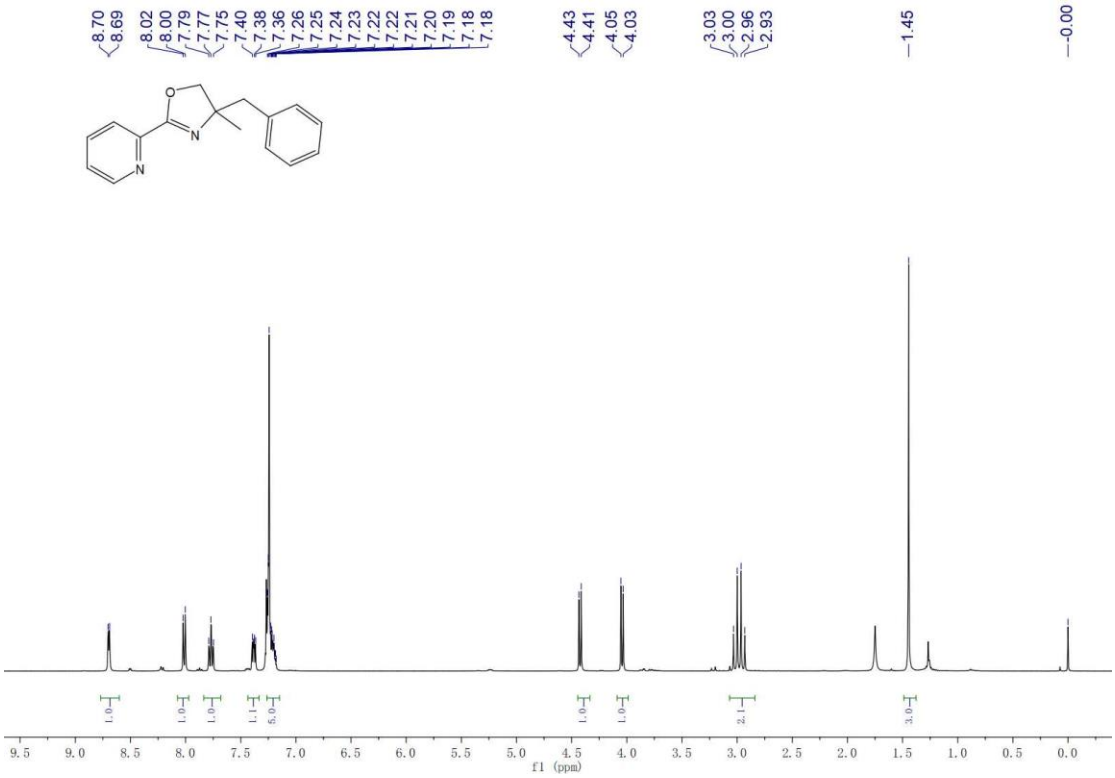

8.70
8.69
8.02
8.00
7.79
7.79
7.77
7.77
7.75
7.75
7.40
7.40
7.39
7.38
7.38
7.38
7.37
7.36
7.27
7.26
7.26
7.25
7.24
7.23
7.22
7.20
7.20
4.43
4.41
4.05
4.03
3.03
3.00
2.96
2.93
1.45
0.00
f1 (ppm)

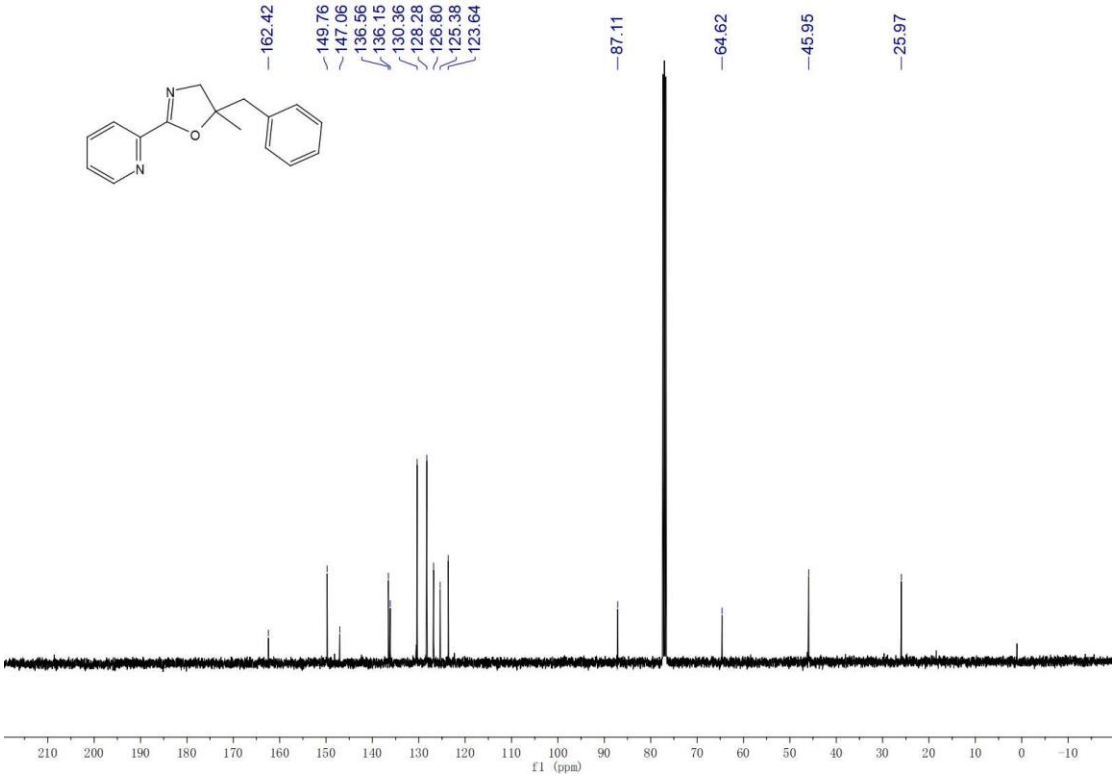

161.54
149.74
146.88
136.99
136.59
130.47
128.13
126.57
125.46
123.84
77.36
77.05
76.73
71.51
46.77
27.03
f1 (ppm)

dept 135

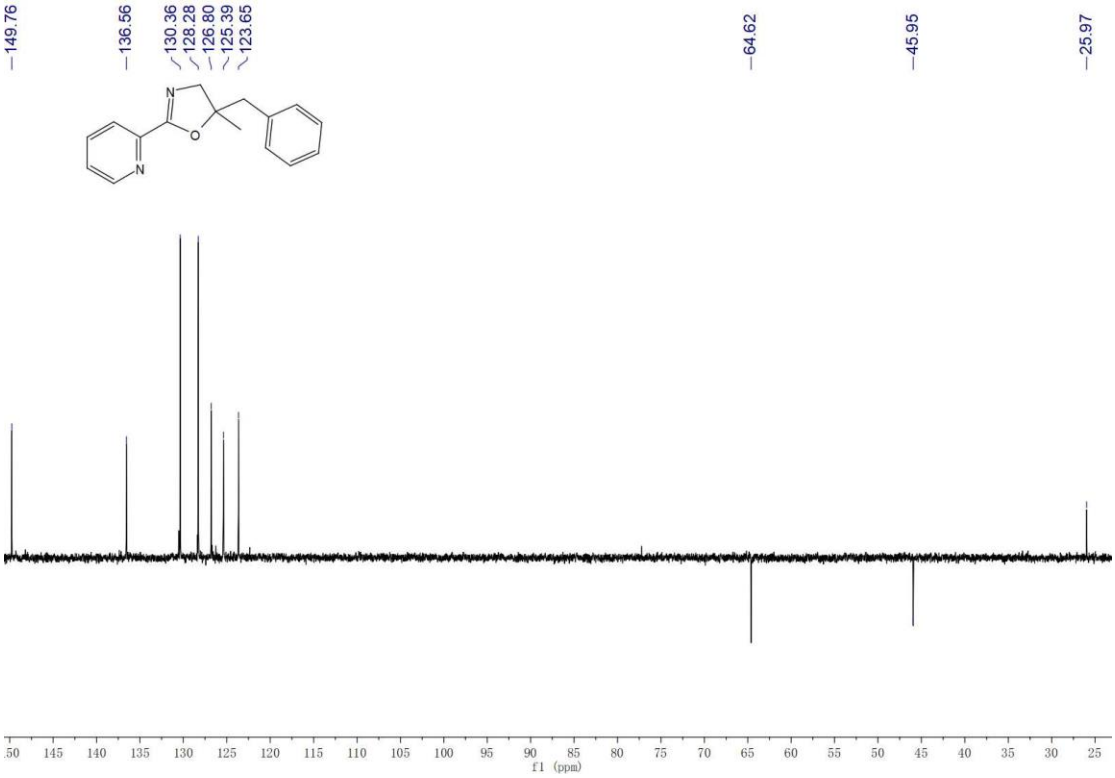

149.74
136.59
130.48
128.14
126.57
125.47
123.85
76.87
46.77
27.03
O
N
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

2r


8.70
8.69
8.02
8.00
7.79
7.77
7.75
7.40
7.38
7.36
7.26
7.25
7.24
7.23
7.22
7.22
7.21
7.20
7.19
7.18
7.18
4.43
4.41
4.05
4.03
3.03
3.00
2.96
2.93
1.45
0.00
f1 (ppm)


162.42
149.76
147.06
136.56
136.15
130.36
128.28
126.80
125.38
123.64
87.11
64.62
45.95
25.97
f1 (ppm)

Dept 135


149.76
136.56
130.36
128.28
126.80
125.39
123.65
64.62
45.95
25.97
N
O
N
50 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25
f1 (ppm)