Supporting Information

Photoredox-Catalysed Formal [3+2] Cycloaddition of *N*-Aryl *α*-Amino Acids with Isoquinoline *N*-Oxides

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1. General information

General procedures and methods

Experiments involving moisture and/or air sensitive components were performed under a positive pressure of argon in oven-dried glassware equipped with a rubber septum inlet. Dried solvents and liquid reagents were transferred by oven-dried syringes or hypodermic syringe cooled to ambient temperature in a desiccator. Reactions mixtures were stirred in 10 mL sample vial with Teflon-coated magnetic stirring bars unless otherwise stated. Moisture in non-volatile reagents/compounds was removed in high *vacuo* by means of an oil pump and subsequent purging with nitrogen. Solvents were removed *in vacuo* under ~30 mmHg and heated with a water bath at 30–35 ^{o}C using rotary evaporator with aspirator. The condenser was cooled with running water at 0 ^{o}C.

All experiments were monitored by analytical thin layer chromatography (TLC). TLC was performed on pre-coated plates, 60 F_{254}. After elution, plate was visualized under UV illumination at 254 nm for UV active material. Further visualization was achieved by staining $Ce(SO_4)_2$ and phosphomolybdic acid solution. For those using the aqueous stains, the TLC plates were heated on a hot plate.

Columns for flash chromatography (FC) contained *silica gel* 200–300 mesh. Columns were packed as slurry of *silica gel* in petroleum ether and equilibrated solution using the appropriate solvent system. The elution was assisted by applying pressure of about 2 atm with an air pump.

Instrumentations

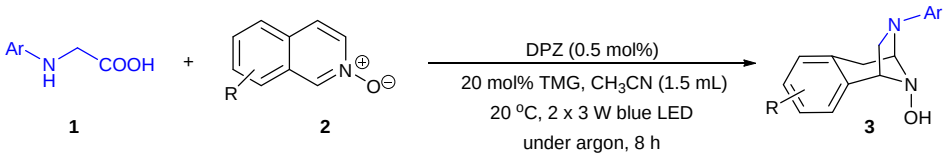
Proton nuclear magnetic resonance (^{1}H NMR) and carbon NMR (^{13}C NMR) were recorded in $CDCl_3$ otherwise stated. Chemical shifts are reported in parts per million (ppm), using the residual solvent signal as an internal standard: $CDCl_3$ (^{1}H NMR: δ 7.26, singlet; ^{13}C NMR: δ 77.0, triplet). Multiplicities were given as: *s* (singlet), *d* (doublet), *t* (triplet), *q* (quartet), *quintet*, *m* (multiplets), *dd* (doublet of doublets), *dt* (doublet of triplets), and *br* (broad). Coupling constants (*J*) were recorded in Hertz (Hz). The number of proton atoms (*n*) for a given resonance was indicated by *n*H. The number of carbon atoms (*n*) for a given resonance was indicated by *n*C. HRMS (Analyzer: TOF) was reported in units of mass of charge ratio (m/z). Mass samples were dissolved in CH_3CN (HPLC Grade) unless otherwise stated.

Optical rotations were recorded on a polarimeter with a sodium lamp of wavelength 589 nm and reported as follows; $[\alpha]_{\lambda}^{T^{\circ}C}$ (c = g/100 mL, solvent). Melting points were determined on a melting point apparatus.

Materials

All commercial reagents were purchased with the highest purity grade. They were used without further purification unless specified. All solvents used, mainly petroleum ether (PE) and ethyl acetate (EtOAc) were distilled. Anhydrous dichloromethane (DCM), CH_3CN and DMF were freshly distilled from CaH_2 and stored under N_2 atmosphere. THF, Et_2O, MTBE, 1,2-dimethoxyethane (DME), 1,4-dioxane, CPME and toluene were freshly distilled from sodium/benzophenone before used. All compounds synthesized were stored in 2~8 ^{o}C and light-sensitive compounds were protected with aluminium foil.

2. General experimental procedures for the synthesis of 3

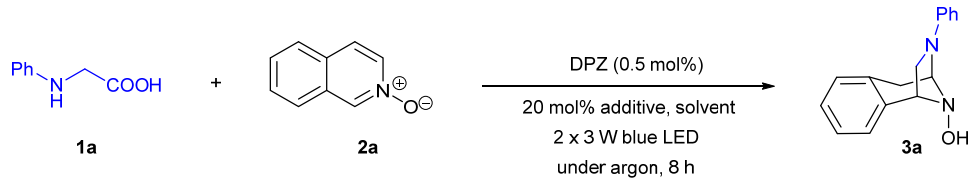


35.5 μL (0.0005 mmol, 0.005 equiv) of DPZ solution (1.0 mg of DPZ in 200 μL of toluene) was added into a 10 mL Schlenk tube, and then solvent was removed in *vacuo*. Subsequently, **1** (0.10 mmol, 1.0 equiv), **2** (0.15 mmol, 1.5 equiv), TMG (0.02 mmol, 0.2 equiv) and CH_3CN (1.5 mL) were sequentially added, degassed three times by freeze-pump-thaw method. The reaction mixture was stirred under an argon atmosphere at 20 ^oC (the temperature was maintained in an incubator) and irradiated by 2 x 3 W blue LED (λ = 450−455 nm) for 8 h. The reaction was monitored by TLC. After completion of the reaction, the reaction mixture was removing the solvent and purification by a short *silica gel* column, followed by gradient elution with petroleum ether/ethyl acetate (30/1–5/1 ratio). Removing the solvent in *vacuo*, afforded products **3a**−**3o**.

Photography of the reaction setup:

3. Optimization of reaction conditions

Table S1. Optimization of reaction conditions[a]



Entry	Additive (20 mol%)	Solvent (mL)	T (°C)	Conv. (%)[b]	Yield (%)[c]
1	--	DCM (0.5)	20	80	50
2	--	THF (0.5)	20	50	35
3	--	DME (0.5)	20	50	40
4	--	MeCN (0.5)	20	80	65
5	--	Toluene (0.5)	20	40	10
6	--	Et_2O (0.5)	20	45	25
7	--	1,4-Dioxane (0.5)	20	60	45
8	--	MTBE (0.5)	20	50	43
9	--	CPME (0.5)	20	50	48
10	--	DMF (0.5)	20	70	60
11	HCOOH	MeCN (0.5)	20	70	45
12	CH_3COOH	MeCN (0.5)	20	65	30
13	Diphenyl phosphate	MeCN (0.5)	20	70	55
14	TMG	MeCN (0.5)	20	95	80
15	$LiPF_6$	MeCN (0.5)	20	80	63
16	Na_2CO_3	MeCN (0.5)	20	75	58
17	K_2CO_3	MeCN (0.5)	20	85	67
18	NaOAc	MeCN (0.5)	20	90	70
19	TMG	MeCN (0.5)	30	100	75
20	TMG	MeCN (0.5)	10	90	78
21	TMG	MeCN (0.5)	0	80	71
22	TMG	MeCN (0.5)	−10	65	65
23	TMG	MeCN (0.75)	20	90	85
24	TMG	MeCN (1.0)	20	90	83
25	TMG	MeCN (1.25)	20	90	78
26	TMG	MeCN (1.5)	20	90	80

[a]Reaction conditions: **1a** (0.075 mmol), **2a** (0.05 mmol). [b]Determined by TLC anaylsis. [c]Isolated by flash column chromatography on *silica gel*.

4. Emission quenching experiments

Emission intensities were recorded on a spectrofluorometer. DPZ solution was excited at 448 nm and the emission intensity at 544 nm was observed. A solution of DPZ (5.0×10^{-5} M) in CH_3CN was added to the appropriate amount of quencher in 5.0 mL volumetric flask under N_2. The solution was transferred to a 1.5 mL quartz cell and the emission spectrum of the sample was collected.

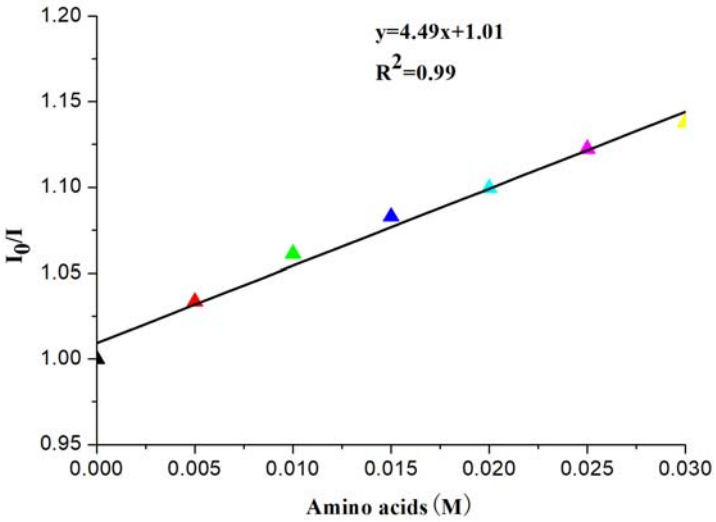


Fig. S1. Stern–Volmer quenching experiment of DPZ and $PhNHCH_2COOH$.

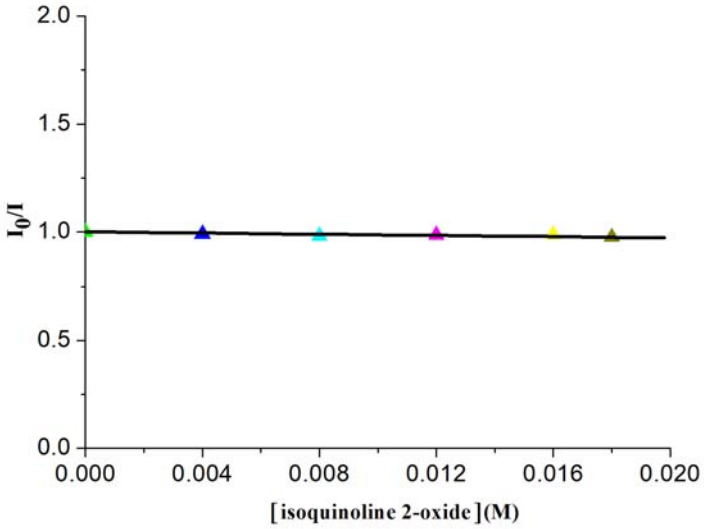


Fig. S2. Stern–Volmer quenching experiment of DPZ and **2a**. No quenching observed.

5. Cyclic voltammetry measurement

Electrochemical potentials were obtained with a standard set of conditions to main internal consistency. Cyclic voltammograms were collected with a potentiostat. Samples were prepared with 0.01 mmol of **2a**, $PhNHCH_2CO_2Na$ in 10 mL of 0.1 M tetrabutylammonium hexafluorophosphate in anhydrous acetonitrile. Measurements employed a radium glassy carbon working electrode, platinum wire counter electrode, saturated KCl silver-silver chloride reference electrode. The obtained value was referenced to Ag/AgCl.

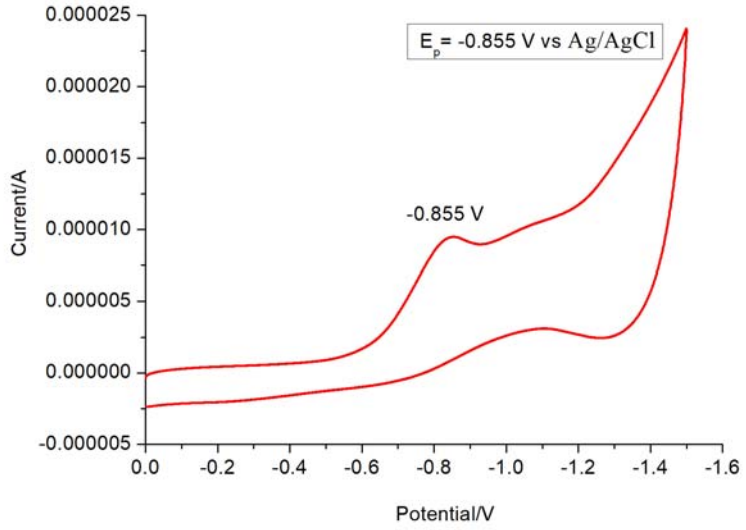


Fig. S3. Cyclic voltammogram of **2a** in MeCN.

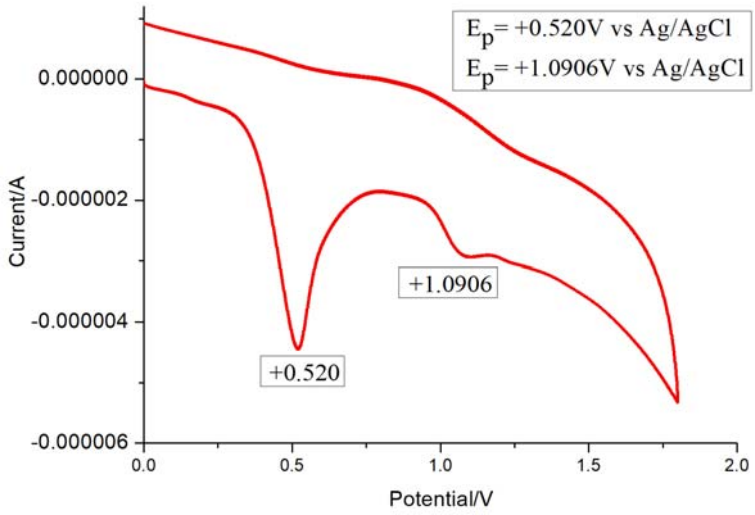


Fig. S4. Cyclic voltammogram of $PhNHCH_2CO_2Na$ in MeCN.

6. Single crystal X-ray diffraction to confirm the structure of products

The structure of these adducts was assigned based on the structure of the derivative of **3m**, which was prepared by **3m** reacting with di-*tert*-butyl dicarbonate, as solved by single crystal X-ray diffraction.

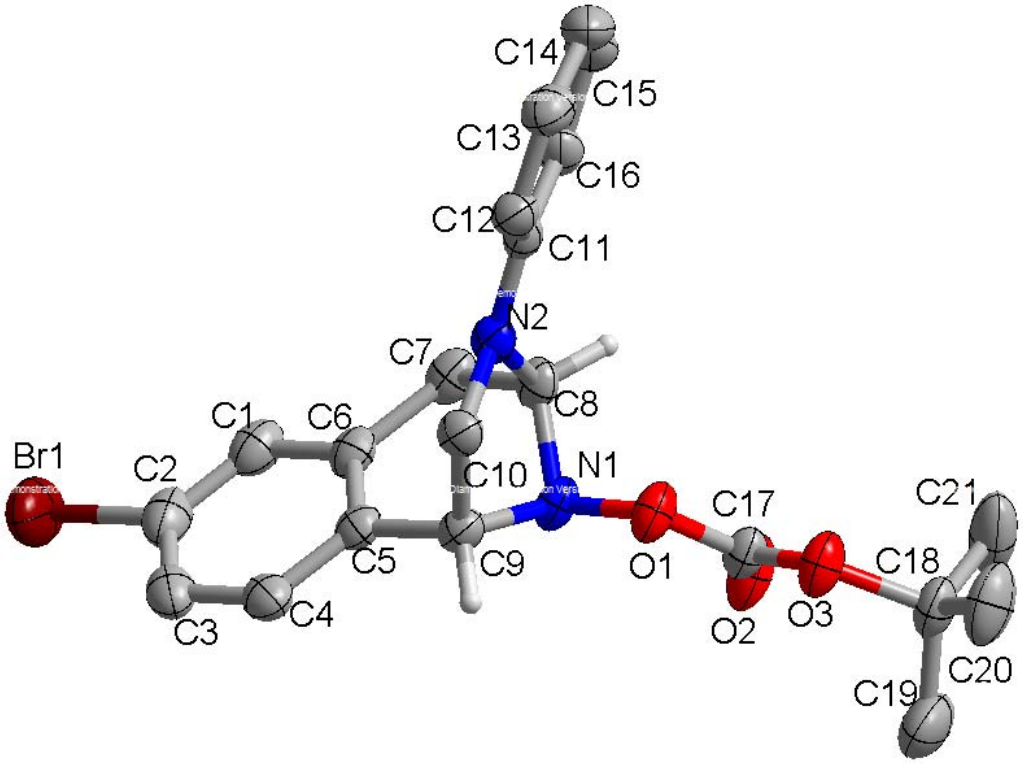


Fig. S5. Structure of **3m** derivative (CCDC 1946502)

Displacement ellipsoids are drawn at the 30% probability level.

(Solvent: ethyl acetate)

Table S2 Crystal data and structure refinement

Identification code	LVY2694HTAB
Empirical formula	$C_{21}H_{23}BrN_2O_3$
Formula weight	431.32
Temperature/K	293(2)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	15.1042(3)
b/Å	6.24720(17)
c/Å	22.4658(7)
α/°	90
β/°	91.320(3)

γ/°	90
Volume/Å^3	2119.30(10)
Z	4
ρ_{calc}g/cm^3	1.352
μ/mm^{-1}	2.824
F(000)	888.0
Crystal size/mm^3	0.19 × 0.15 × 0.1
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.128 to 134.144
Index ranges	-18 ≤ h ≤ 13, -4 ≤ k ≤ 7, -26 ≤ l ≤ 24
Reflections collected	7829
Independent reflections	3786 [R_{int} = 0.0252, R_{sigma} = 0.0347]
Data/restraints/parameters	3786/1/251
Goodness-of-fit on F^2	1.053
Final R indexes [I>=2σ (I)]	R_1 = 0.0507, wR_2 = 0.1388
Final R indexes [all data]	R_1 = 0.0628, wR_2 = 0.1531
Largest diff. peak/hole / e Å^{-3}	0.63/-0.42

Table S3 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for LVY2694HTAB. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Br1	-1889.9(5)	11407(3)	3570.4(9)	118.0(5)
Br1A	-1853(3)	10871(12)	3325(4)	118.0(5)
C1	-275(2)	9109(6)	3704.4(15)	74.0(8)
C2	-743(2)	10880(7)	3886.3(16)	81.3(10)
C3	-411(2)	12302(6)	4298.4(16)	76.9(9)
C4	429(2)	11934(5)	4536.0(13)	65.2(7)
C5	920.4(18)	10178(4)	4357.3(10)	54.6(6)
C6	572.6(18)	8755(4)	3938.1(12)	57.9(6)
C7	1111(2)	6855(4)	3743.1(13)	63.3(7)
C8	2059.9(19)	7033(4)	3983.4(12)	56.4(6)
C9	1850.6(18)	9840(4)	4598.0(11)	54.5(5)
C10	2534.9(18)	10612(4)	4154.2(11)	53.7(5)
C11	3147.0(17)	8652(4)	3292.9(10)	51.7(5)
C12	3791(2)	10203(5)	3212.3(11)	60.3(6)
C13	4432(2)	9937(6)	2783.7(14)	77.3(8)
C14	4423(3)	8139(7)	2423.5(14)	85.6(10)
C15	3764(3)	6647(6)	2487.3(14)	79.4(9)
C16	3131(2)	6869(5)	2911.3(12)	65.3(7)
C17	2952.8(19)	5525(4)	5233.1(12)	58.0(6)

C18	4106(2)	4004(5)	5890.2(15)	74.2(8)
C19	3557(4)	4021(10)	6440(2)	117.1(16)
C20	5024(3)	4818(8)	6028(2)	113.4(16)
C21	4114(4)	1863(7)	5604(3)	123(2)
N1	1985.2(15)	7495(3)	4620.3(10)	57.7(5)
N2	2524.9(15)	8863(3)	3726.2(9)	53.2(5)
O1	2879.4(13)	7278(3)	4880.9(9)	67.7(5)
O2	2398.1(18)	4219(4)	5301.8(12)	88.5(8)
O3	3756.7(15)	5643(3)	5471.0(10)	74.8(6)

Table S4 Anisotropic Displacement Parameters ($Å^2×10^3$) for LVY2694HTAB. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br1	64.0(3)	173.8(8)	115.6(10)	10.8(7)	-11.2(4)	23.4(3)
Br1A	64.0(3)	173.8(8)	115.6(10)	10.8(7)	-11.2(4)	23.4(3)
C1	65.4(16)	88(2)	68.8(16)	18.7(15)	-4.6(13)	-15.2(15)
C2	55.9(15)	106(3)	83(2)	36.6(19)	7.4(14)	3.2(16)
C3	70.4(17)	79.4(19)	81.8(19)	24.8(17)	22.0(15)	11.8(15)
C4	74.3(17)	60.7(15)	61.4(14)	12.1(12)	15.1(13)	0.6(13)
C5	63.9(14)	52.3(12)	47.8(11)	14.0(10)	4.3(10)	-4.0(11)
C6	59.9(14)	57.5(13)	56.2(13)	18.0(11)	-0.5(11)	-7.8(11)
C7	74.9(16)	50.9(13)	63.6(14)	6.4(11)	-12.2(12)	-16.9(12)
C8	69.1(15)	36.7(10)	62.8(14)	4.2(10)	-6.1(11)	-0.6(10)
C9	67.1(14)	50.5(12)	45.7(11)	2.7(9)	-3.0(10)	-3.7(11)
C10	64.4(14)	41.6(11)	55.0(12)	-1.8(9)	0.0(11)	-3.5(10)
C11	60.5(13)	49.0(12)	45.0(11)	1.7(9)	-12.1(9)	7.2(10)
C12	69.8(15)	59.9(14)	51.1(12)	2.0(11)	0.0(11)	1.6(12)
C13	77.3(19)	90(2)	64.6(16)	14.9(15)	9.0(14)	0.1(16)
C14	98(2)	105(3)	54.1(15)	3.6(16)	14.5(15)	32(2)
C15	98(2)	83(2)	56.8(15)	-14.8(14)	-3.9(15)	22.1(19)
C16	76.5(17)	59.8(15)	59.1(14)	-11.9(11)	-13.4(12)	10.6(13)
C17	68.9(15)	46.3(12)	58.0(13)	5.0(10)	-17.0(11)	-0.7(12)
C18	81.2(19)	67.7(17)	72.2(17)	11.8(14)	-29.5(15)	5.2(14)
C19	115(3)	153(4)	82(2)	30(3)	-22(2)	26(3)
C20	88(3)	115(3)	134(4)	32(3)	-53(3)	-3(2)
C21	150(4)	74(2)	143(4)	-7(2)	-70(4)	31(3)
N1	62.9(12)	52.1(11)	57.2(11)	14.7(9)	-14.1(9)	-4.2(9)
N2	64.6(12)	39.5(9)	55.3(10)	-1.0(8)	-2.5(9)	-3.2(8)
O1	63.9(11)	66.1(11)	72.3(11)	24.2(9)	-16.8(9)	-6.9(9)
O2	92.1(16)	60.8(12)	110.3(18)	27.4(12)	-44.2(14)	-15.5(12)

O3	71.8(12)	67.1(11)	84.1(13)	18.8(10)	-26.4(10)	-5.8(10)

Table S5 Bond Lengths for LVY2694HTAB.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br1	C2	1.886(3)	C11	C12	1.388(4)
Br1A	C2	2.076(6)	C11	C16	1.406(4)
C1	C2	1.379(5)	C11	N2	1.375(4)
C1	C6	1.390(4)	C12	C13	1.392(4)
C2	C3	1.370(6)	C13	C14	1.384(6)
C3	C4	1.383(5)	C14	C15	1.374(6)
C4	C5	1.389(4)	C15	C16	1.372(5)
C5	C6	1.389(4)	C17	O1	1.354(3)
C5	C9	1.509(4)	C17	O2	1.182(4)
C6	C7	1.510(4)	C17	O3	1.317(3)
C7	C8	1.523(4)	C18	C19	1.503(6)
C8	N1	1.466(3)	C18	C20	1.502(5)
C8	N2	1.468(3)	C18	C21	1.484(5)
C9	C10	1.531(4)	C18	O3	1.480(3)
C9	N1	1.480(3)	N1	O1	1.466(3)
C10	N2	1.455(3)			

Table S6 Bond Angles for LVY2694HTAB.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C1	C6	119.3(3)	N2	C11	C16	120.4(3)
C1	C2	Br1	120.1(3)	C11	C12	C13	120.5(3)
C1	C2	Br1A	103.3(4)	C14	C13	C12	120.2(3)
C3	C2	Br1	117.4(3)	C15	C14	C13	119.2(3)
C3	C2	Br1A	133.7(4)	C16	C15	C14	121.5(3)
C3	C2	C1	122.5(3)	C15	C16	C11	120.1(3)
C2	C3	C4	118.1(3)	O2	C17	O1	125.8(2)
C3	C4	C5	120.7(3)	O2	C17	O3	129.4(2)
C4	C5	C6	120.3(3)	O3	C17	O1	104.7(2)
C4	C5	C9	120.4(3)	C20	C18	C19	110.6(4)
C6	C5	C9	119.2(2)	C21	C18	C19	111.9(4)
C1	C6	C7	120.9(3)	C21	C18	C20	112.2(4)
C5	C6	C1	119.0(3)	O3	C18	C19	108.8(3)
C5	C6	C7	120.1(2)	O3	C18	C20	102.1(3)
C6	C7	C8	110.4(2)	O3	C18	C21	110.8(3)
N1	C8	C7	105.5(2)	C8	N1	C9	100.11(18)
N1	C8	N2	106.20(19)	O1	N1	C8	106.3(2)
N2	C8	C7	111.8(2)	O1	N1	C9	103.26(19)

C5	C9	C10	111.1(2)	C10	N2	C8	108.8(2)
N1	C9	C5	106.1(2)	C11	N2	C8	123.0(2)
N1	C9	C10	103.9(2)	C11	N2	C10	122.9(2)
N2	C10	C9	101.36(19)	C17	O1	N1	111.73(19)
C12	C11	C16	118.4(3)	C17	O3	C18	122.1(2)
N2	C11	C12	121.2(2)				

Table S7 Hydrogen Bonds for LVY2694HTAB.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
C9	H9	O2[1]	0.98	2.52	3.257(4)	132.1

[1]+X,1+Y,+Z

Table S8 Torsion Angles for LVY2694HTAB.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
Br1	C2	C3	C4	179.8(2)	C9	C5	C6	C7	2.0(3)
Br1A	C2	C3	C4	-170.6(3)	C9	C10	N2	C8	-13.6(3)
C1	C2	C3	C4	0.3(4)	C9	C10	N2	C11	-168.6(2)
C1	C6	C7	C8	170.6(2)	C9	N1	O1	C17	145.5(2)
C2	C1	C6	C5	1.1(4)	C10	C9	N1	C8	-44.3(2)
C2	C1	C6	C7	-179.0(3)	C10	C9	N1	O1	65.3(2)
C2	C3	C4	C5	0.4(4)	C11	C12	C13	C14	1.4(5)
C3	C4	C5	C6	-0.4(4)	C12	C11	C16	C15	2.6(4)
C3	C4	C5	C9	177.3(2)	C12	C11	N2	C8	-156.2(2)
C4	C5	C6	C1	-0.4(4)	C12	C11	N2	C10	-4.7(4)
C4	C5	C6	C7	179.7(2)	C12	C13	C14	C15	1.2(5)
C4	C5	C9	C10	-99.4(3)	C13	C14	C15	C16	-2.0(5)
C4	C5	C9	N1	148.4(2)	C14	C15	C16	C11	0.0(5)
C5	C6	C7	C8	-9.5(3)	C16	C11	C12	C13	-3.3(4)
C5	C9	C10	N2	-77.5(2)	C16	C11	N2	C8	25.2(4)
C5	C9	N1	C8	72.9(2)	C16	C11	N2	C10	176.7(2)
C5	C9	N1	O1	-177.58(18)	C19	C18	O3	C17	-63.5(4)
C6	C1	C2	Br1	179.5(2)	C20	C18	O3	C17	179.5(3)
C6	C1	C2	Br1A	172.2(3)	C21	C18	O3	C17	59.9(5)
C6	C1	C2	C3	-1.1(4)	N1	C8	N2	C10	-13.8(3)
C6	C5	C9	C10	78.3(3)	N1	C8	N2	C11	141.1(2)
C6	C5	C9	N1	-33.9(3)	N1	C9	C10	N2	36.1(2)
C6	C7	C8	N1	49.6(3)	N2	C8	N1	C9	35.8(2)
C6	C7	C8	N2	-65.4(3)	N2	C8	N1	O1	-71.4(2)
C7	C8	N1	C9	-83.1(2)	N2	C11	C12	C13	178.1(3)

C7	C8	N1	O1	169.75(18)	N2	C11	C16	C15	-178.8(3)
C7	C8	N2	C10	100.8(3)	O1	C17	O3	C18	-179.9(3)
C7	C8	N2	C11	-104.3(3)	O2	C17	O1	N1	4.7(4)
C8	N1	O1	C17	-109.7(2)	O2	C17	O3	C18	0.4(5)
C9	C5	C6	C1	-178.1(2)	O3	C17	O1	N1	-174.9(2)

Table S9 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for LVY2694HTAB.

Atom	*x*	*y*	*z*	U(eq)
H1	-524	8163	3428	89
H3	-740	13481	4415	92
H4	666	12872	4819	78
H7A	1112	6790	3312	76
H7B	846	5547	3888	76
H8	2386	5699	3920	68
H9	1940	10502	4990	65
H10A	2357	11953	3969	64
H10B	3115	10782	4343	64
H12	3794	11430	3447	72
H13	4869	10971	2739	93
H14	4857	7943	2142	103
H15	3746	5461	2237	95
H16	2691	5838	2946	78
H19A	3529	5452	6594	176
H19B	3822	3094	6734	176
H19C	2970	3529	6341	176
H20A	5388	4605	5689	170
H20B	5271	4053	6363	170
H20C	4998	6317	6120	170
H21A	3517	1377	5539	185
H21B	4427	871	5858	185
H21C	4404	1958	5229	185

Table S10 Atomic Occupancy for LVY2694HTAB.

Atom	*Occupancy*	Atom	*Occupancy*	Atom	*Occupancy*
Br1	0.823(5)	Br1A	0.177(5)		

Experimental

The crystal was kept at 293(2) K during data collection. Using Olex2, the structure was solved with the ShelXS structure solution program using Direct Methods and refined with the ShelXL refinement package using Least Squares minimisation.

Crystal structure determination

Crystal Data for $C_{21}H_{23}BrN_2O_3$ (M =431.32 g/mol): monoclinic, space group $P2_1/n$ (no. 14), a = 15.1042(3) Å, b = 6.24720(17) Å, c = 22.4658(7) Å, β = 91.320(3)°, V = 2119.30(10) Å^3, Z = 4, T = 293(2) K, μ(CuKα) = 2.824 mm^{-1}, $Dcalc$ = 1.352 g/cm^3, 7829 reflections measured (7.128° ≤ 2Θ ≤ 134.144°), 3786 unique (R_{int} = 0.0252, R_{sigma} = 0.0347) which were used in all calculations. The final R_1 was 0.0507 (I > 2σ(I)) and wR_2 was 0.1531 (all data).

Refinement model description

Number of restraints - 1, number of constraints - unknown.

Details:

1. Fixed Uiso
   At 1.2 times of:
   All C(H) groups, All C(H,H) groups
   At 1.5 times of:
   All C(H,H,H) groups
2. Restrained distances
   Br1-C2
   1.9 with sigma of 0.02
3. Uiso/Uaniso restraints and constraints

Uanis(Br1) = Uanis(Br1A)

4. Others
   Sof(Br1A)=1-FVAR(1)
   Sof(Br1)=FVAR(1)

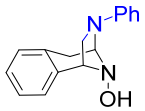
5.a Ternary CH refined with riding coordinates:
   C8(H8), C9(H9)

5.b Secondary CH2 refined with riding coordinates:
   C7(H7A,H7B), C10(H10A,H10B)

5.c Aromatic/amide H refined with riding coordinates:
   C1(H1), C3(H3), C4(H4), C12(H12), C13(H13), C14(H14), C15(H15), C16(H16)

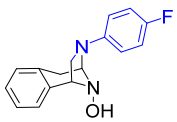
5.d Idealised Me refined as rotating group:
   C19(H19A,H19B,H19C), C20(H20A,H20B,H20C), C21(H21A,H21B,H21C)

7. Characterization data of products



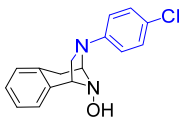
3a

Yellow solid; Mp 69.7−71.3 °C; 21.5 mg, 85.0% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24 – 7.13 (m, 4H), 7.12 – 7.04 (m, 2H), 6.69 (t, *J* = 7.3 Hz, 1H), 6.53 (d, *J* = 8.0 Hz, 2H), 5.06 (s, 1H), 4.51 (s, 1H), 4.00 – 3.90 (m, 1H), 3.40 – 3.13 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.6, 138.2, 131.4, 129.3, 128.8, 127.9, 126.1, 125.8, 116.1, 111.4, 80.4, 68.2, 52.8, 35.8. HRMS (ESI) m/z 275.1154 ($M+Na^+$), calc. for $C_{16}H_{16}N_2NaO$ 275.1155.



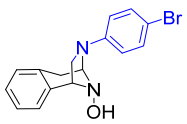
3b

White solid; Mp 84.3−85.9 °C; 25.0 mg, 93.0% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20 – 7.04 (m, 4H), 6.91 (d, *J* = 8.7 Hz, 2H), 6.49 – 6.38 (m, 2H), 5.00 (s, 1H), 4.50 (d, *J* = 4.4 Hz, 1H), 3.94 – 3.85 (m, 1H), 3.24 (dt, *J* = 37.2, 16.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.7, 153.6, 142.4, 138.1, 131.3, 128.8, 127.9, 126.1, 125.8, 115.9, 115.6, 111.8, 111.7, 81.1, 68.4, 53.2, 36.1. HRMS (ESI) m/z 271.1246 ($M+H^+$), calc. for $C_{16}H_{16}FN_2O$ 271.1241.



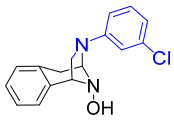
3c

Yellow solid; Mp 88.6−90.3 °C; 27.2 mg, 95.0% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.21 – 7.05 (m, 6H), 6.45 (d, *J* = 8.8 Hz, 2H), 5.04 (s, 1H), 4.52 (s, 1H), 3.95 – 3.88 (m, 1H), 3.25 (dt, *J* = 37.8, 16.3 Hz, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 144.3, 138.0, 131.1, 129.1, 128.8, 128.0, 126.2, 125.8, 120.9, 112.4, 80.7, 68.2, 53.0, 35.7. HRMS (ESI) m/z 309.0765 ($M+Na^+$), calc. for $C_{16}H_{15}ClN_2NaO$ 309.0765.



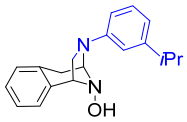
3d

Yellow solid; Mp 99.8−101.3 °C; 30.4 mg, 92.0% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (s, 2H), 7.22 – 7.13 (m, 2H), 7.12 – 7.03 (m, 2H), 6.39 (d, *J* = 8.7 Hz, 2H), 4.97 (s, 1H), 4.50 (d, *J* = 3.9 Hz, 1H), 3.92 – 3.83 (m, 1H), 3.23 (dt, *J* = 38.6, 16.4 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.7, 137.9, 131.9, 131.1, 128.8, 128.0, 126.2, 125.8, 113.0, 108.0, 80.7, 68.2, 52.9, 35.7. HRMS (ESI) m/z 353.0259 ($M+Na^+$), calc. for $C_{16}H_{15}BrN_2NaO$ 353.0260.



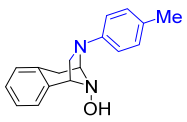
3e

Yellow solid; Mp 97.3−98.8 °C; 21.0 mg, 73.0% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.16 – 6.93 (m, 5H), 6.56 (d, *J* = 7.9 Hz, 1H), 6.43 (s, 1H), 6.33 (d, *J* = 8.3 Hz, 1H), 4.97 (s, 1H), 4.45 (d, *J* = 4.2 Hz, 1H), 3.91 – 3.82 (m, 1H), 3.19 (dt, *J* = 35.1, 16.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.7, 138.0, 135.1, 131.1, 130.3, 128.8, 128.0, 126.2, 125.8, 116.0, 111.4, 109.7, 80.6, 68.2, 52.9, 35.7. HRMS (ESI) m/z 309.0762 ($M+Na^+$), calc. for $C_{16}H_{15}ClN_2NaO$ 309.0765.



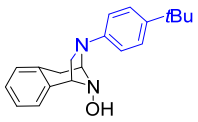
3f

Yellow oil; 25.6 mg, 87.0% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.18 – 7.03 (m, 5H), 6.57 (d, *J* = 7.5 Hz, 1H), 6.38 (d, *J* = 7.3 Hz, 2H), 5.12 (s, 1H), 4.52 (s, 1H), 3.97 (dd, *J* = 7.5, 4.7 Hz, 1H), 3.29 (dt, *J* = 29.6, 15.9 Hz, 3H), 2.83 (dt, *J* = 13.8, 6.9 Hz, 1H), 1.23 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.2, 145.6, 138.2, 131.4, 129.2, 128.9, 127.8, 126.0, 125.8, 114.3, 109.6, 109.0, 80.4, 68.2, 52.8, 35.9, 34.5, 24.0. HRMS (ESI) m/z 317.1621 ($M+Na^+$), calc. for $C_{19}H_{22}N_2NaO$ 317.1624.



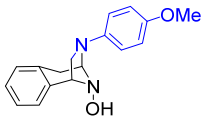
3g

Yellow solid; Mp 125.9−127.2 °C; 23.0 mg, 87.4% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.19 – 7.00 (m, 6H), 6.46 (d, *J* = 8.4 Hz, 2H), 5.02 (s, 1H), 4.49 (d, *J* = 3.8 Hz, 1H), 3.97 – 3.87 (m, 1H), 3.25 (dt, *J* = 32.1, 16.4 Hz, 3H), 2.24 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.6, 138.3, 131.5, 129.8, 128.8, 127.8, 126.0, 125.8, 125.1, 111.3, 80.7, 68.2, 52.9, 36.0, 20.2. HRMS (ESI) m/z 267.1490 ($M+H^+$), calc. for $C_{17}H_{19}N_2O$ 267.1492.



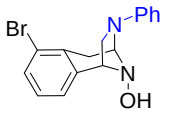
3h

Yellow solid; Mp 80.6−82.5 °C; 29.3 mg, 95.2% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25 (d, *J* = 7.2 Hz, 2H), 7.10 (m, 4H), 6.51 (d, *J* = 8.7 Hz, 2H), 5.11 (s, 1H), 4.51 (s, 1H), 3.95 (dd, *J* = 7.5, 4.7 Hz, 1H), 3.27 (dt, *J* = 28.9, 16.3 Hz, 3H), 1.27 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.3, 138.6, 138.2, 131.5, 128.8, 127.7, 126.1, 125.9, 125.7, 110.9, 80.4, 68.2, 52.8, 35.9, 33.7, 31.5. HRMS (ESI) m/z 309.1962 ($M+H^+$), calc. for $C_{20}H_{25}N_2O$ 309.1961.



3i

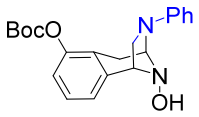
Yellow solid; Mp 126.8−128.3 °C; 26.9 mg, 95.3% yield; 1H NMR (300 MHz, $CDCl_3$) δ 7.16 (t, *J* = 8.2 Hz, 2H), 7.11 – 7.02 (m, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 6.48 (d, *J* = 8.9 Hz, 2H), 4.96 (s, 1H), 4.48 (s, 1H), 3.95 – 3.86 (m, 1H), 3.75 (s, 3H), 3.24 (dt, *J* = 36.4, 16.4 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.0, 140.6, 138.2, 131.5, 128.8, 127.8, 126.0, 125.8, 115.1, 112.0, 81.1, 68.3, 55.9, 53.2, 36.2. HRMS (ESI) m/z 283.1445 ($M+H^+$), calc. for $C_{17}H_{19}N_2O_2$ 283.1441.



3j

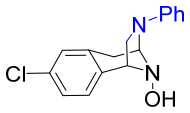
Yellow solid; Mp 128.4−130.1 °C; 28.2 mg, 85.0% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 (dd, *J* = 7.1, 1.9 Hz, 1H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.05 (q, *J* = 7.4 Hz, 2H), 6.71 (t, *J* = 7.3 Hz, 1H), 6.58 (d, *J* = 8.1 Hz, 2H), 5.18 (s, 1H), 4.53 (d, *J* = 4.4 Hz, 1H), 3.98 (dd, *J* = 7.6, 4.9 Hz, 1H), 3.30 (d, *J* = 7.9 Hz, 1H), 3.20 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.4, 140.2, 131.8, 131.8, 129.4, 127.7, 125.2, 125.0, 116.5, 111.4, 80.2, 68.0, 52.7, 38.1. HRMS (ESI) m/z 331.0448 ($M+H^+$), calc. for $C_{16}H_{16}BrN_2O$

331.0441.



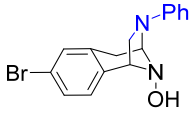
3k

Yellow solid; Mp 145.1−146.6 °C; 27.8 mg, 82.2% yield;^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20 (dd, *J* = 14.5, 7.3 Hz, 3H), 7.02 (dd, *J* = 15.5, 7.7 Hz, 2H), 6.68 (t, *J* = 7.3 Hz, 1H), 6.55 (d, *J* = 8.0 Hz, 2H), 5.14 (s, 1H), 4.54 (d, *J* = 3.8 Hz, 1H), 4.02 – 3.87 (m, 1H), 3.30 (d, *J* = 7.7 Hz, 1H), 3.14 (s, 2H), 1.52 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.1, 149.7, 145.6, 140.2, 129.3, 127.2, 124.2, 123.2, 121.0, 116.3, 111.4, 83.6, 79.9, 67.9, 52.6, 31.8, 27.6. HRMS (ESI) m/z 369.1800 ($M+H^+$), calc. for $C_{21}H_{25}N_2O_4$ 369.1809.



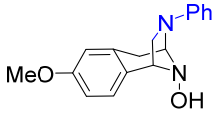
3l

White solid; Mp 124.9−125.5 °C; 26.8 mg, 93.4% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (t, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.3 Hz, 1H), 7.05 (d, *J* = 8.3 Hz, 2H), 6.69 (t, *J* = 7.3 Hz, 1H), 6.54 (d, *J* = 8.0 Hz, 2H), 5.12 (s, 1H), 4.52 (d, *J* = 4.1 Hz, 1H), 3.95 (dd, *J* = 7.8, 4.7 Hz, 1H), 3.37 – 3.11 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.4, 136.7, 133.5, 133.4, 129.4, 128.9, 127.1, 126.2, 116.4, 111.4, 80.0, 67.6, 52.7, 35.7. HRMS (ESI) m/z 309.0766 ($M+Na^+$), calc. for $C_{16}H_{15}ClN_2NaO$ 309.0765.



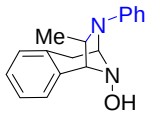
3m

White solid; Mp 132.5−133.7 °C; 25.5 mg, 77% yield; ^{1}H NMR (300 MHz, CD_2Cl_2) δ 7.24 (dt, *J* = 15.8, 8.5 Hz, 4H), 6.98 (d, *J* = 8.1 Hz, 1H), 6.69 (t, *J* = 7.3 Hz, 1H), 6.53 (d, *J* = 8.0 Hz, 2H), 5.11 (s, 1H), 4.49 (d, *J* = 4.4 Hz, 1H), 3.94 (dd, *J* = 7.7, 4.7 Hz, 1H), 3.39 – 3.09 (m, 3H). ^{13}C NMR (75 MHz, CD_2Cl_2) δ 145.30, 137.19, 133.71, 131.2, 128.8, 128.6, 127.0, 120.7, 115.8, 110.9, 79.9, 67.3, 35.4. HRMS (ESI) m/z 353.0256 ($M+Na^+$), calc. for $C_{16}H_{15}BrN_2NaO$ 353.0260.



3n

Yellow solid; Mp 75.5−76.8 °C; 20.0mg, 71% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.21 (t, *J* = 7.7 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 1H), 6.69 (dd, *J* = 13.7, 6.8 Hz, 2H), 6.59 (s, 1H), 6.51 (d, *J* = 8.0 Hz, 2H), 4.93 (s, 1H), 4.50 (s, 1H), 3.91 (t, *J* = 6.1 Hz, 1H), 3.74 (s, 3H), 3.19 (dd, *J* = 31.2, 12.5 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.2, 145.7, 132.7, 130.7, 129.3, 126.8, 116.1, 113.7, 112.0, 111.4, 80.2, 67.6, 55.2, 52.8, 36.0. HRMS (ESI) m/z 283.1445 ($M+H^+$), calc. for $C_{17}H_{19}N_2O_2$ 283.1441.

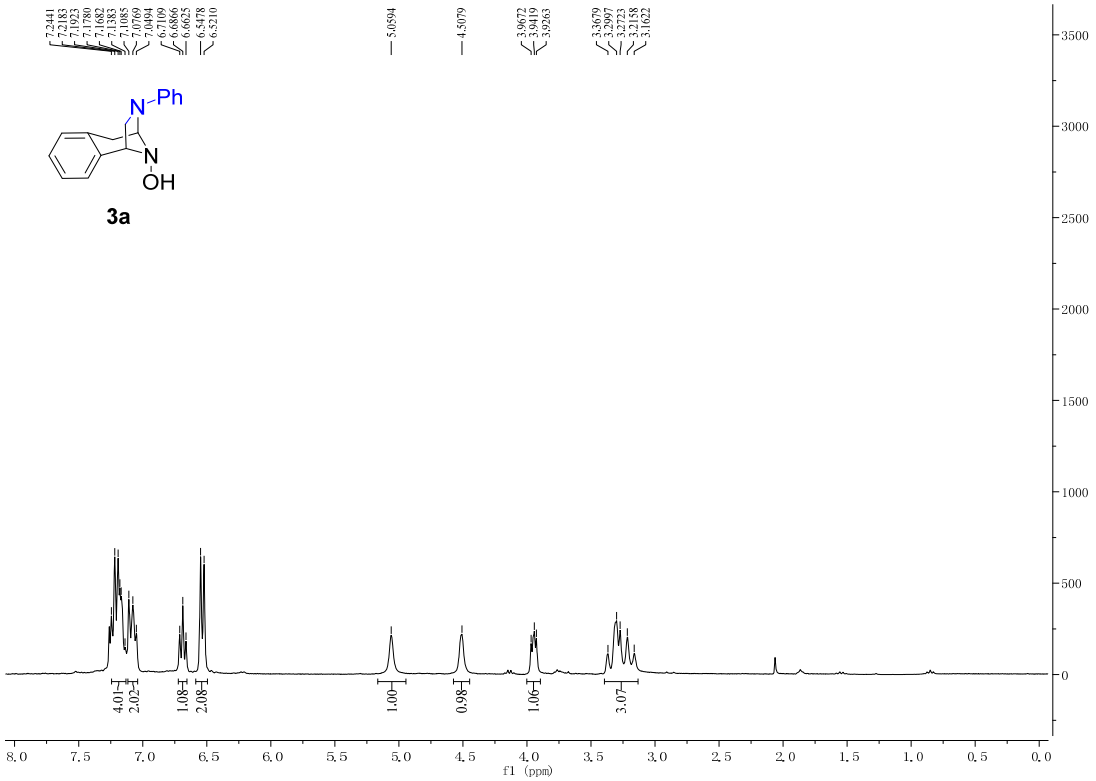


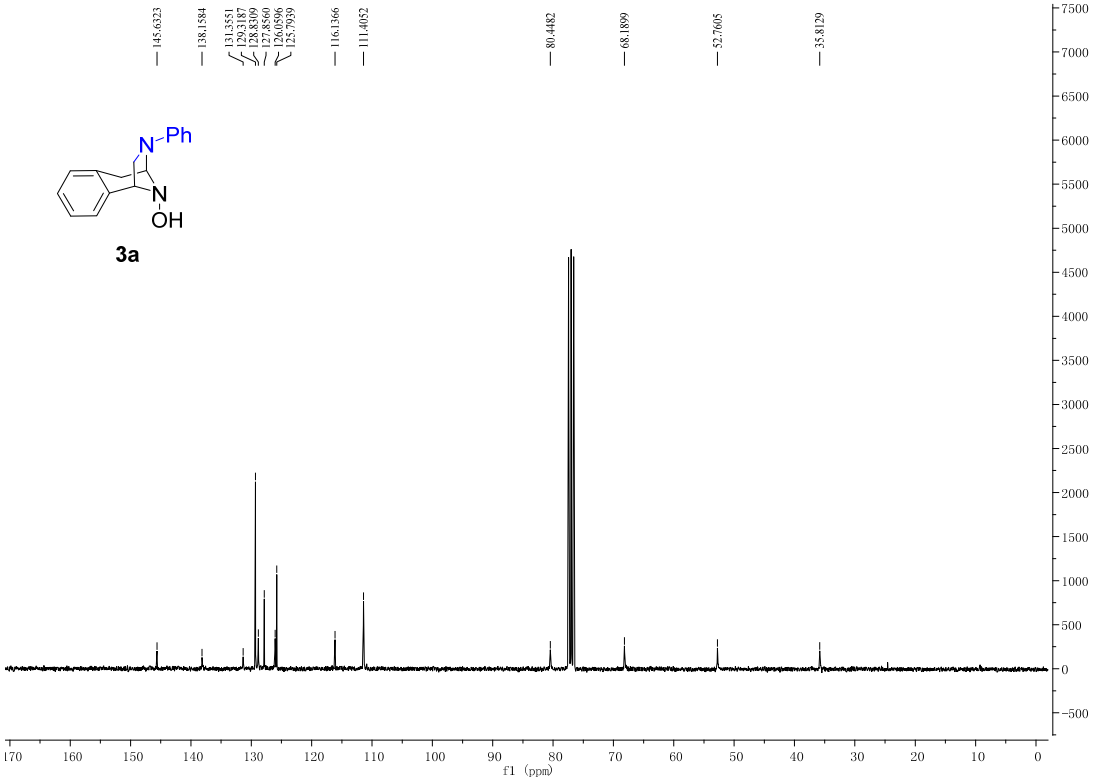
3o

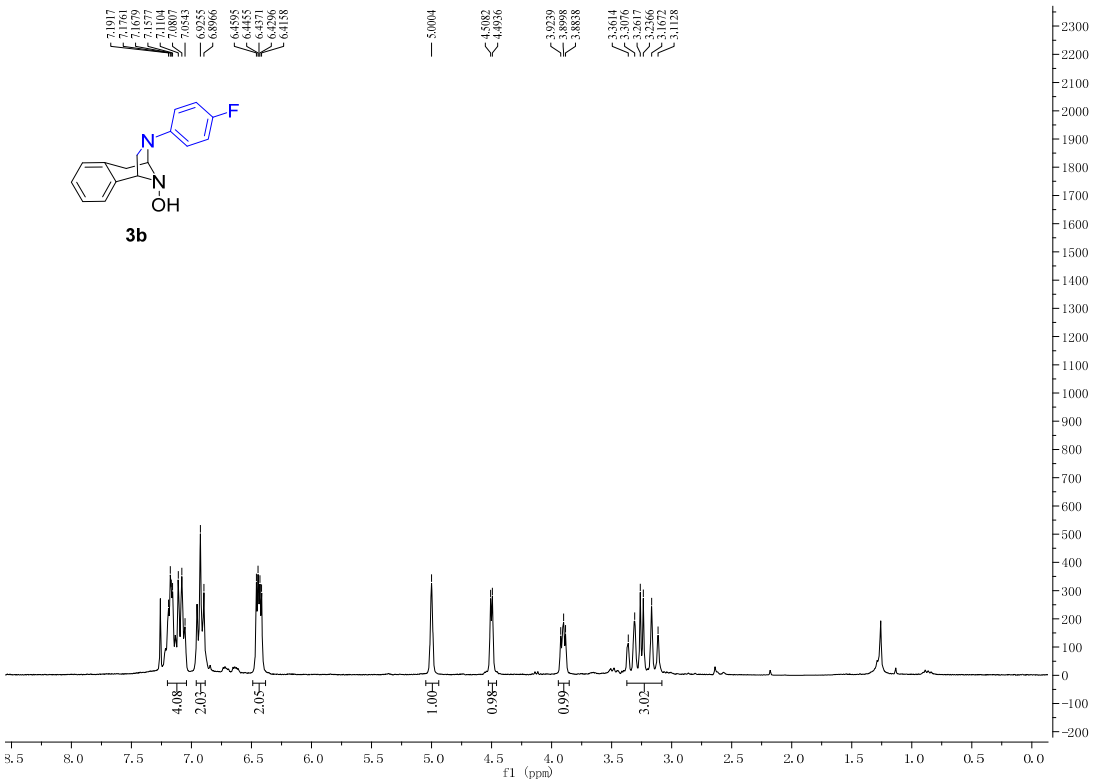
Yellow solid; Mp 86.5−87.6 °C; 16.0 mg, 60.0% yield; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 – 7.12 (m, 6H), 7.04 (d, *J* = 7.3 Hz, 1H), 6.74 (dd, *J* = 23.9, 7.8 Hz, 3H), 5.02 (s, 1H), 4.49 – 4.33 (m, 2H), 3.45 (dd, *J* = 16.4, 3.4 Hz, 1H), 3.16 (d, *J* = 16.3 Hz, 1H), 0.91 (d, *J* = 6.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.8, 135.2,

131.84, 129.2, 128.7, 127.9, 127.6, 125.5, 117.4, 112.9, 85.6, 73.3, 55.9, 38.6, 15.0. HRMS (ESI) m/z 267.1497 ($M+H^+$), calc. for $C_{17}H_{19}N_2O$ 267.1492.

8. ^{1}H and ^{13}C NMR spectra



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6.8966
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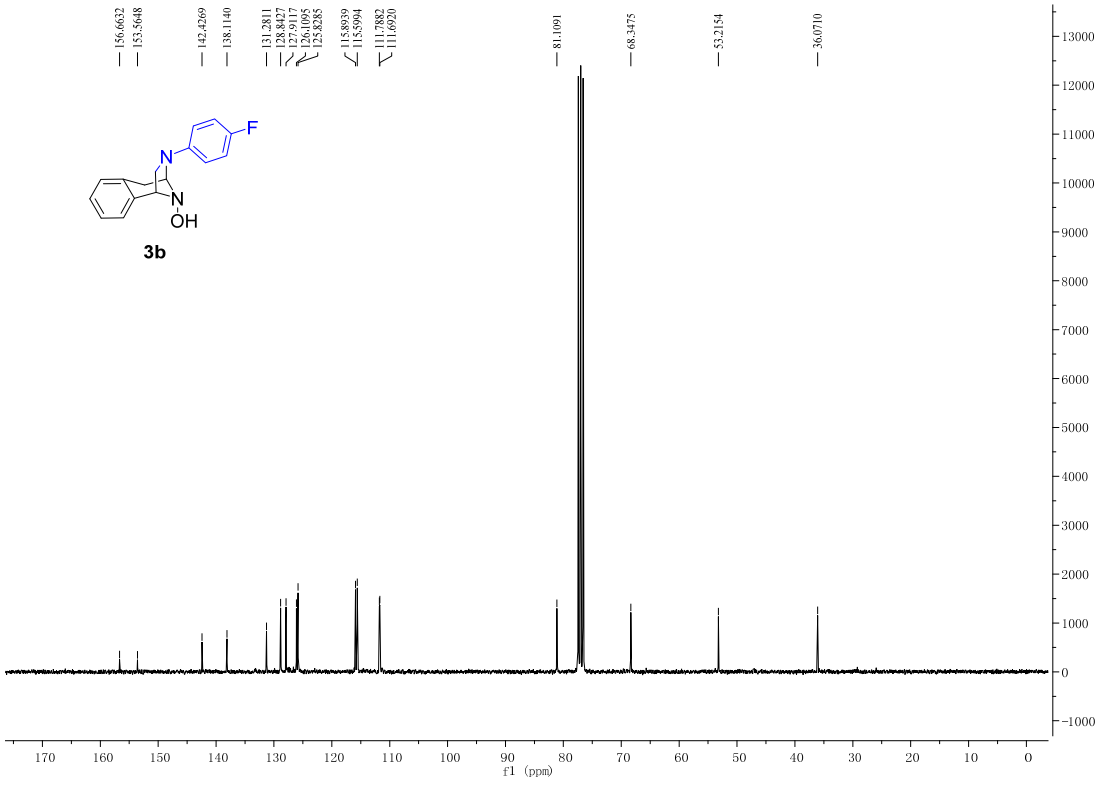
6.4371
6.4296
6.4158
5.0004
4.5082
4.4936
3.9239
3.8998
3.8838
3.3614
3.3076
3.2617
3.2366
3.1672
3.1128
F
N
N
OH
3b
4.08
2.03
2.05
1.00
0.98
0.99
3.02
f1 (ppm)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142.4269
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128.8427
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125.8285
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115.5994
111.7882
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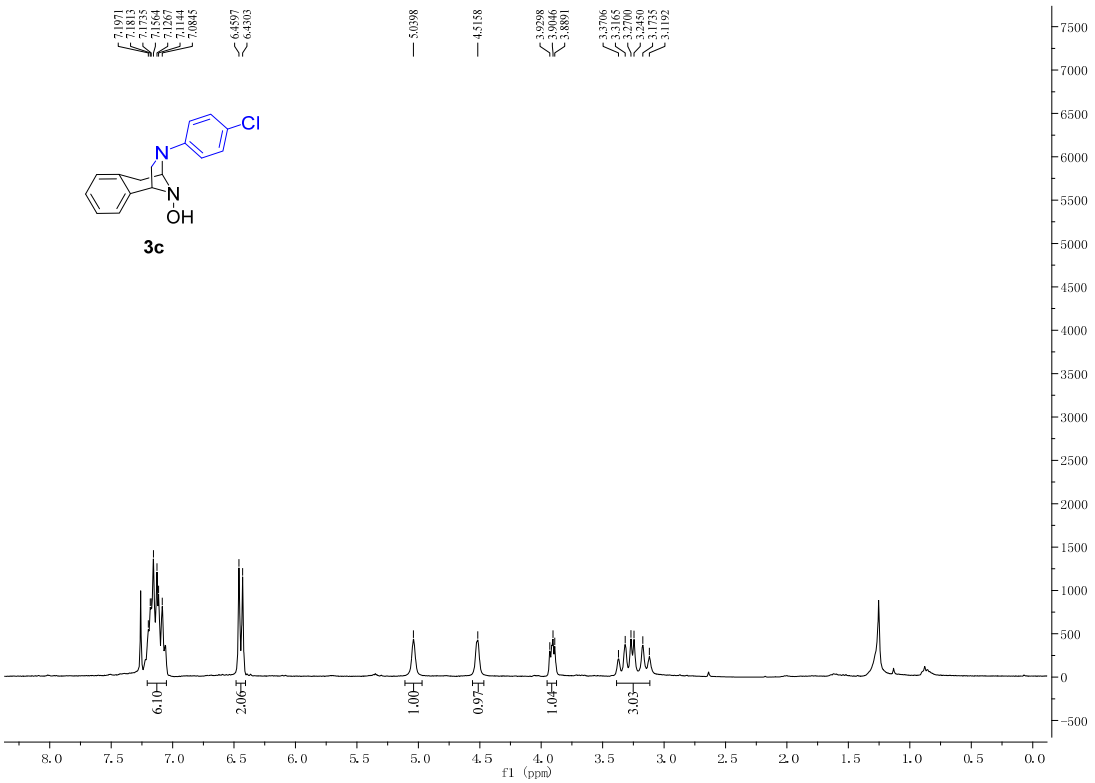
81.1091
68.3475
53.2154
36.0710
F
N
N
OH
3b
f1 (ppm)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7.0845
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5.0398
4.5158
3.9298
3.9046
3.8891
3.3706
3.3165
3.2700
3.2450
3.1735
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N
OH
3c
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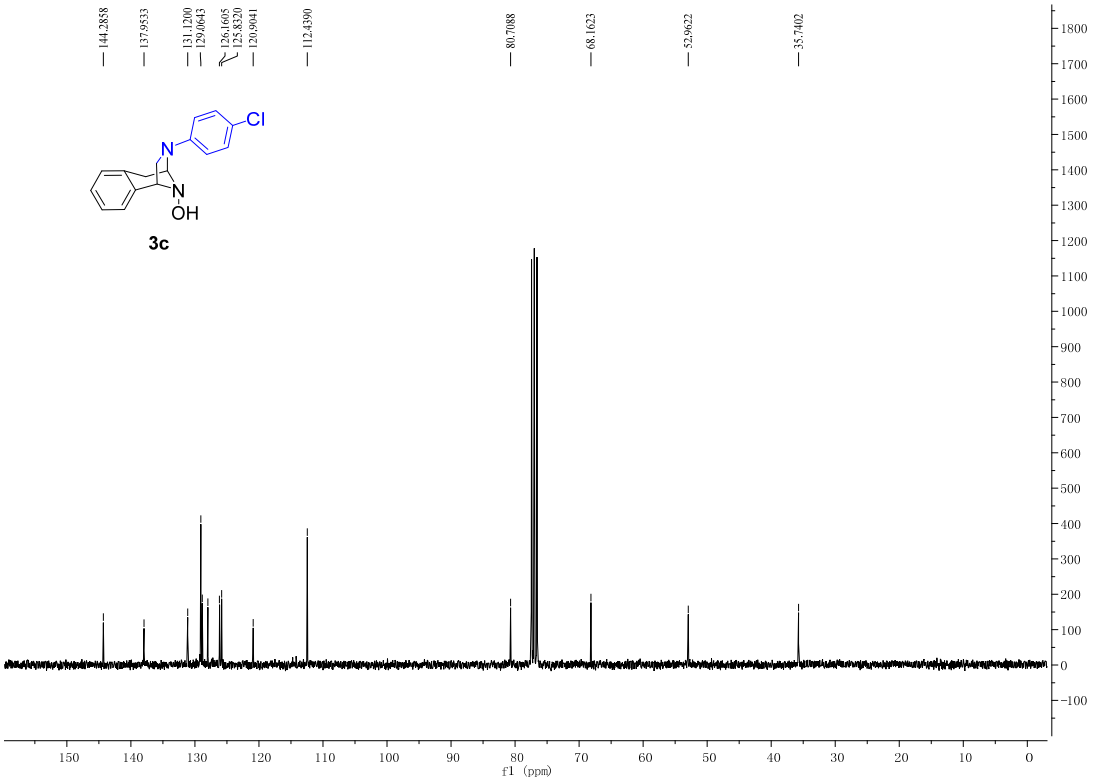
f1 (ppm)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Cl
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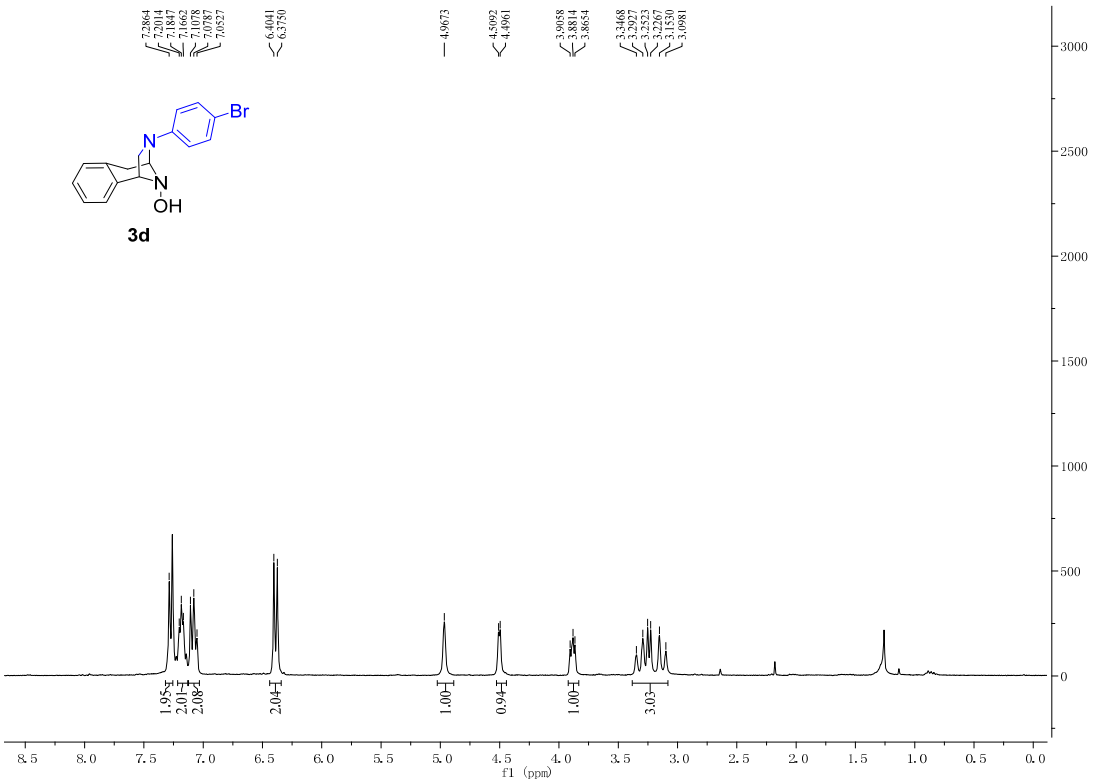
OH
3c
f1 (ppm)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3.8814
3.8654
3.3468
3.2927
3.2753
3.2267
3.1530
3.0981
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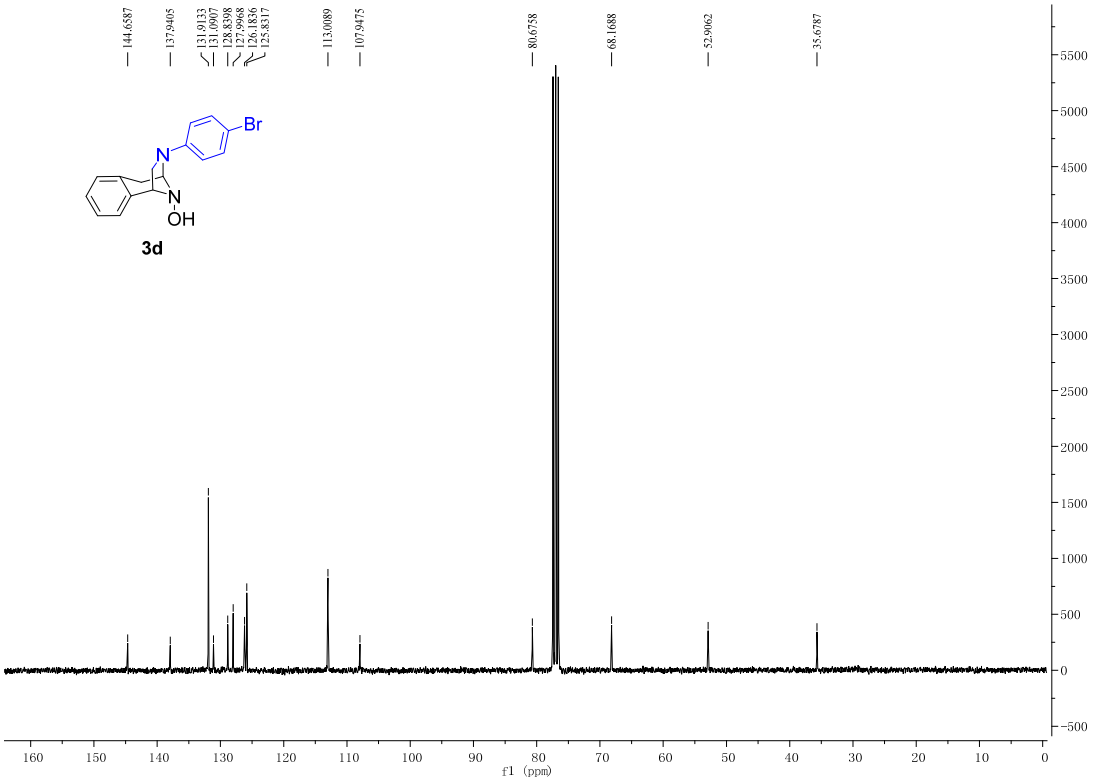
f1 (ppm)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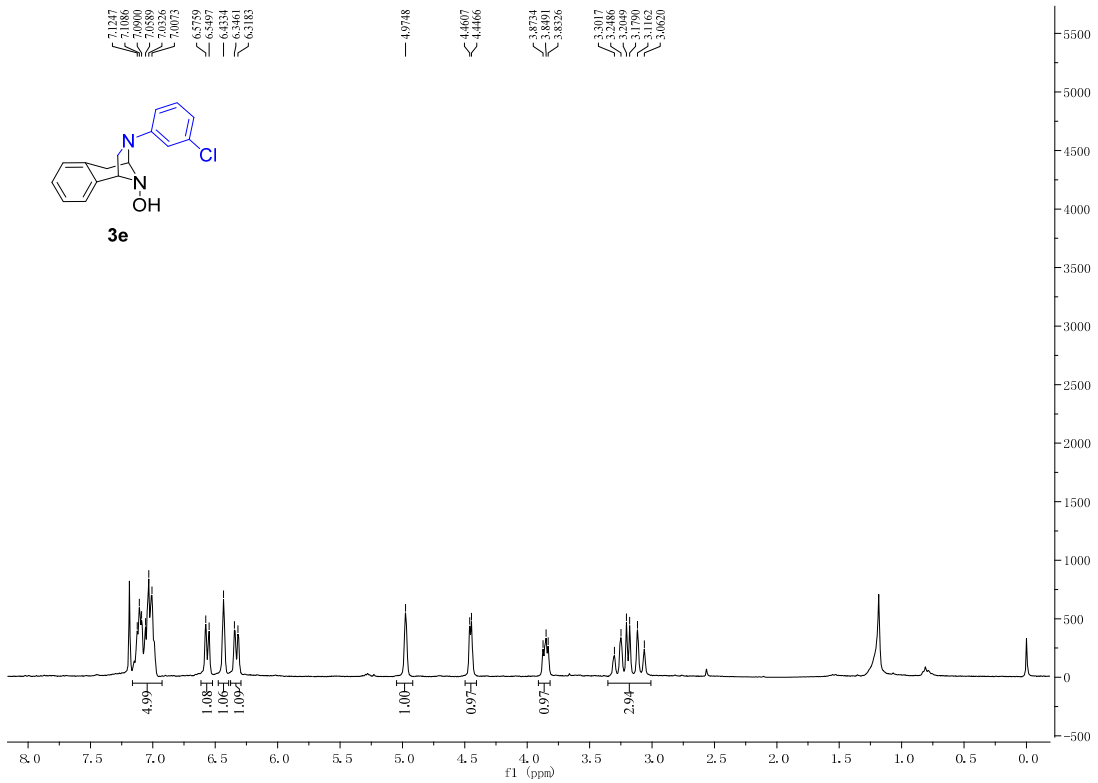
OH
3d
f1 (ppm)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4.9748
4.4607
4.4466
3.8734
3.8491
3.8326
3.3017
3.2486
3.2049
3.1790
3.1162
3.0620
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3e
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1.06
1.09
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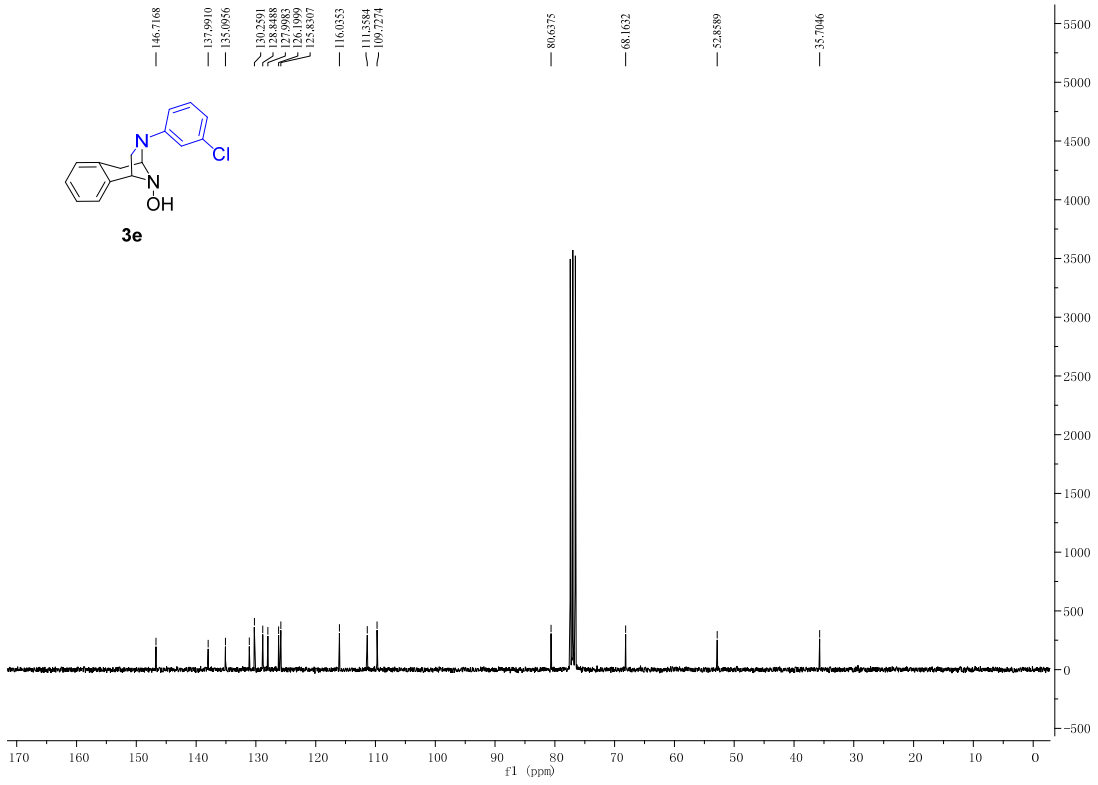
2.94
f1 (ppm)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Cl
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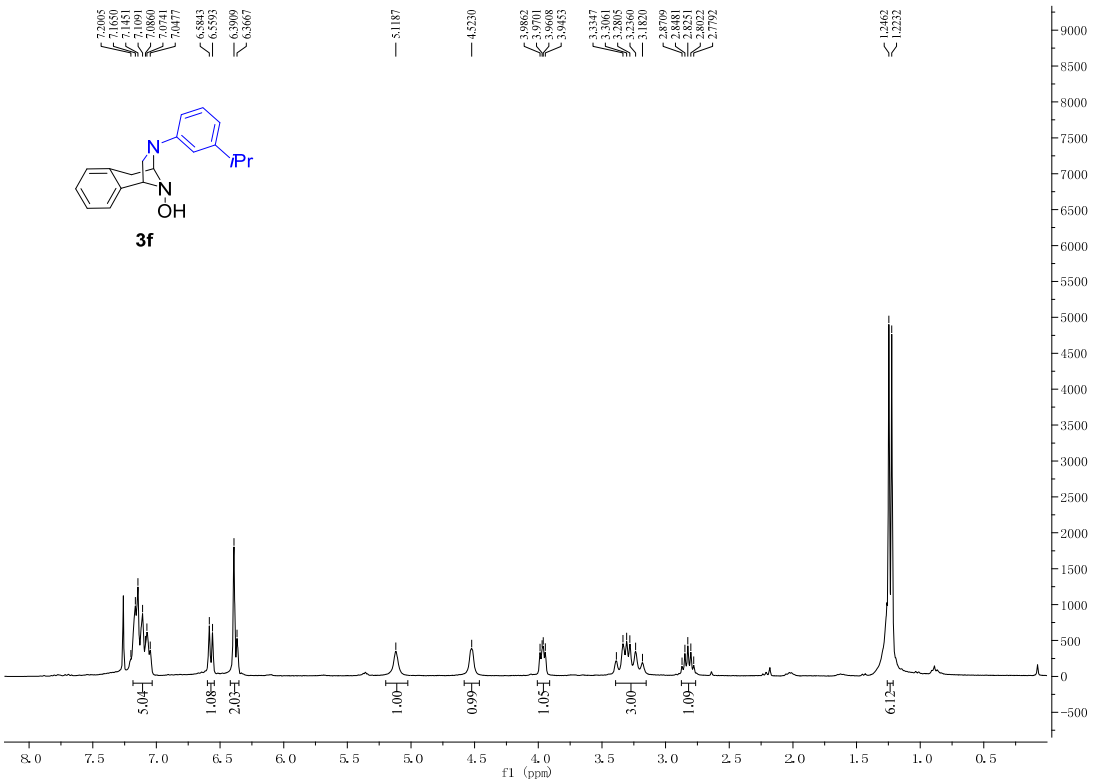
OH
3e
f1 (ppm)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6.3667
5.1187
4.5230
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3.3347
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3.2965
3.2366
3.1820
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2.8022
2.7792
1.2462
1.2232
iPr
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OH
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1.05
3.00
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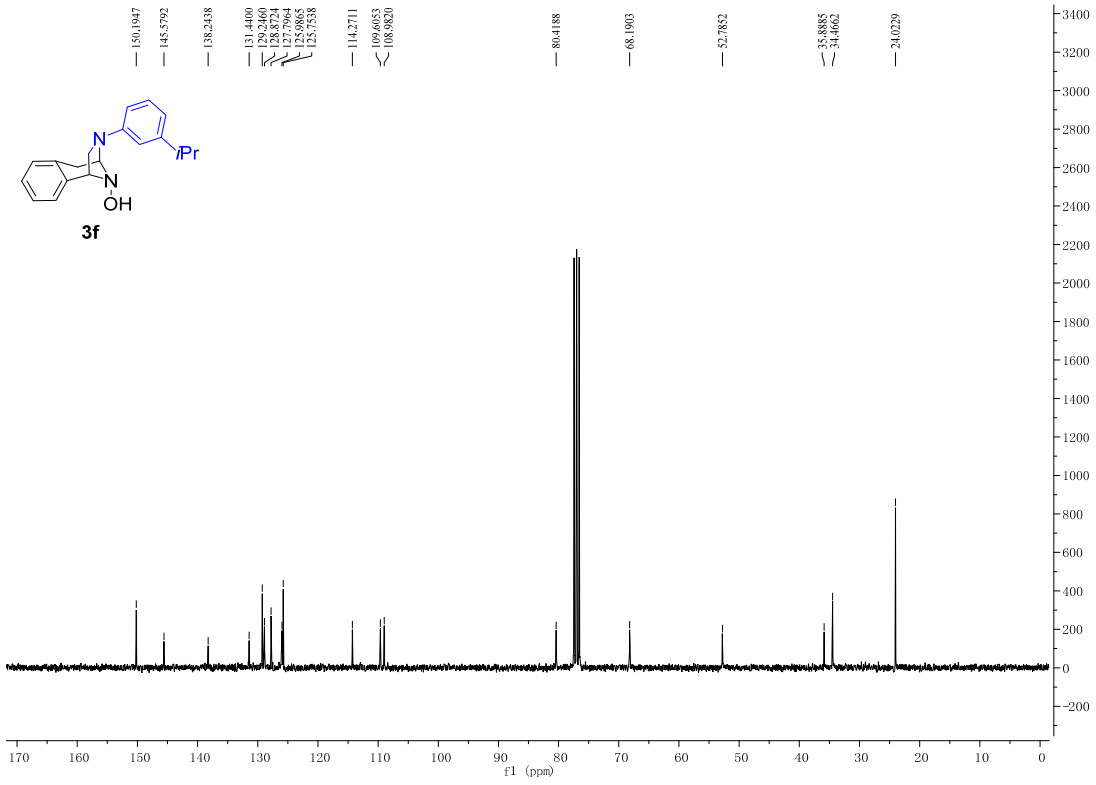
f1 (ppm)

3f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109.6053
108.9820
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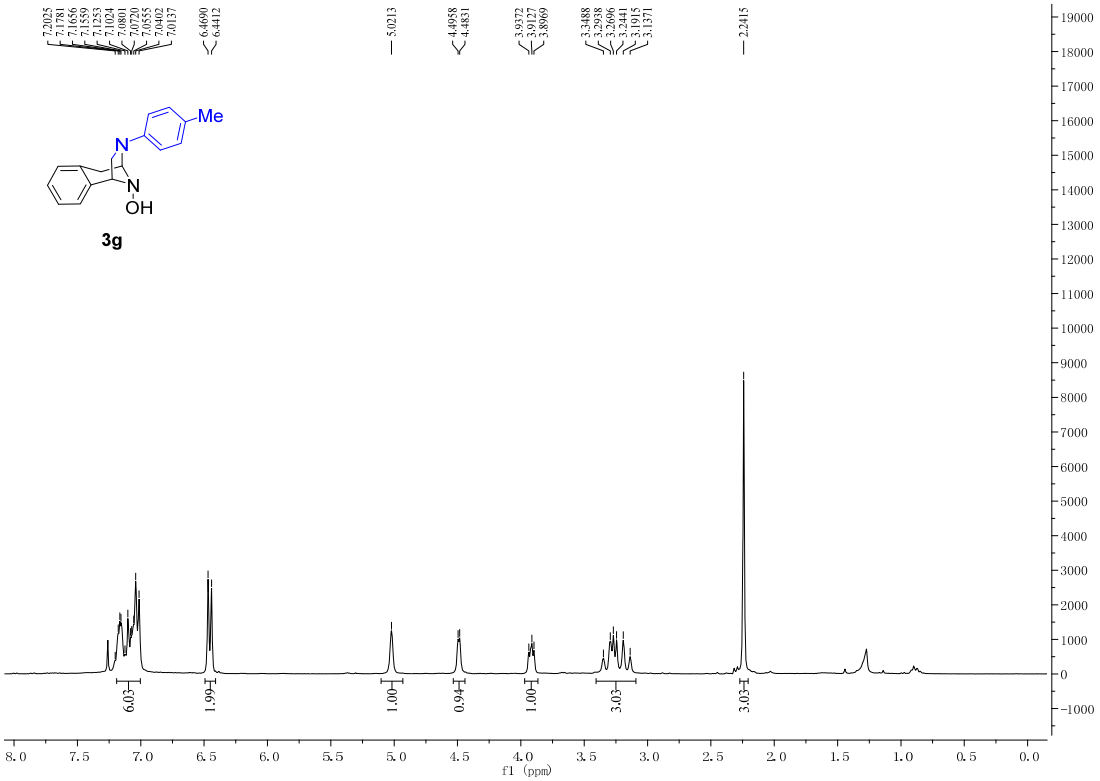
OH
f1 (ppm)

3f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7.0801
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6.4412
5.0213
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4.4831
3.9372
3.9127
3.8969
3.3488
3.2938
3.2696
3.2441
3.1915
3.1571
2.2415
Me
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OH
6.03
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1.00
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3.03
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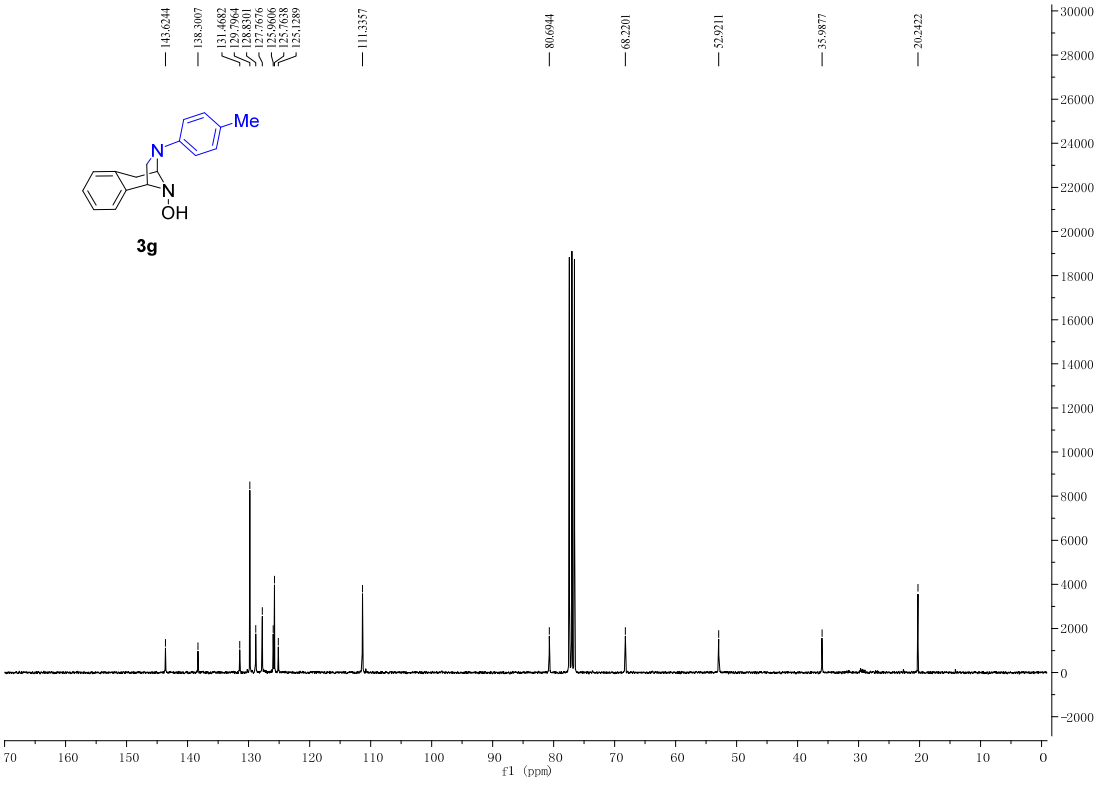
f1 (ppm)

3g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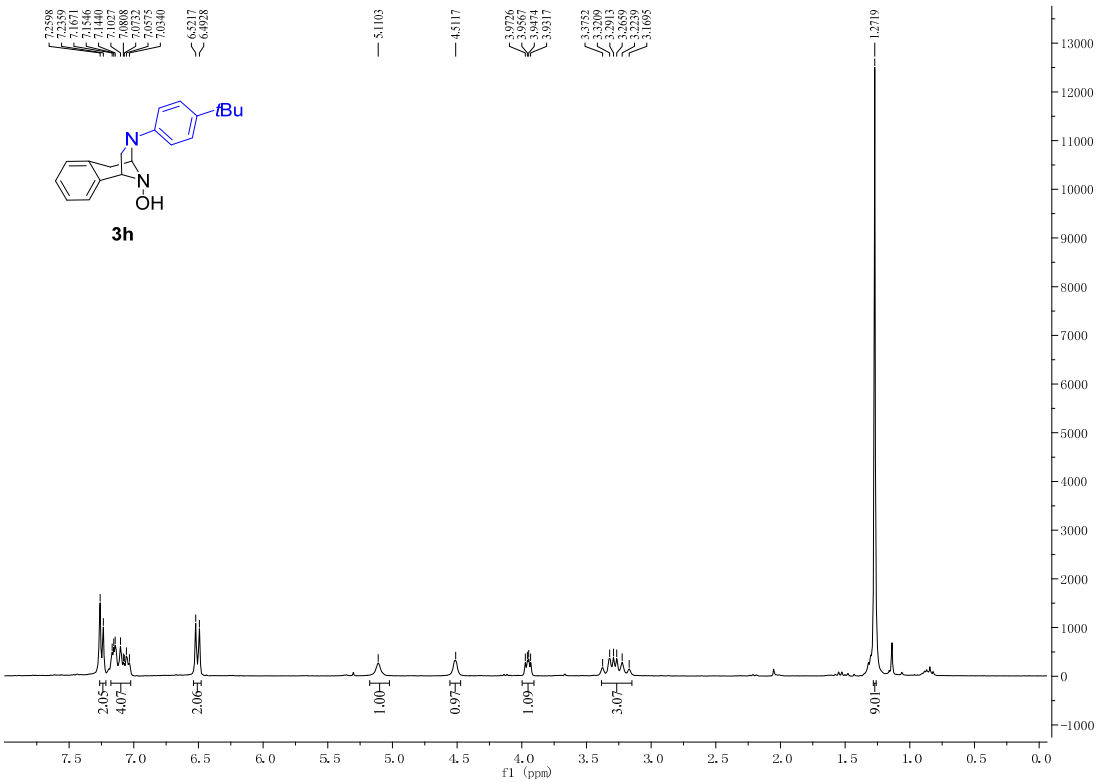
OH
f1 (ppm)

3g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6.4928
5.1103
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3.3209
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3.2659
3.2239
3.1695
1.2719
tBu
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2.06
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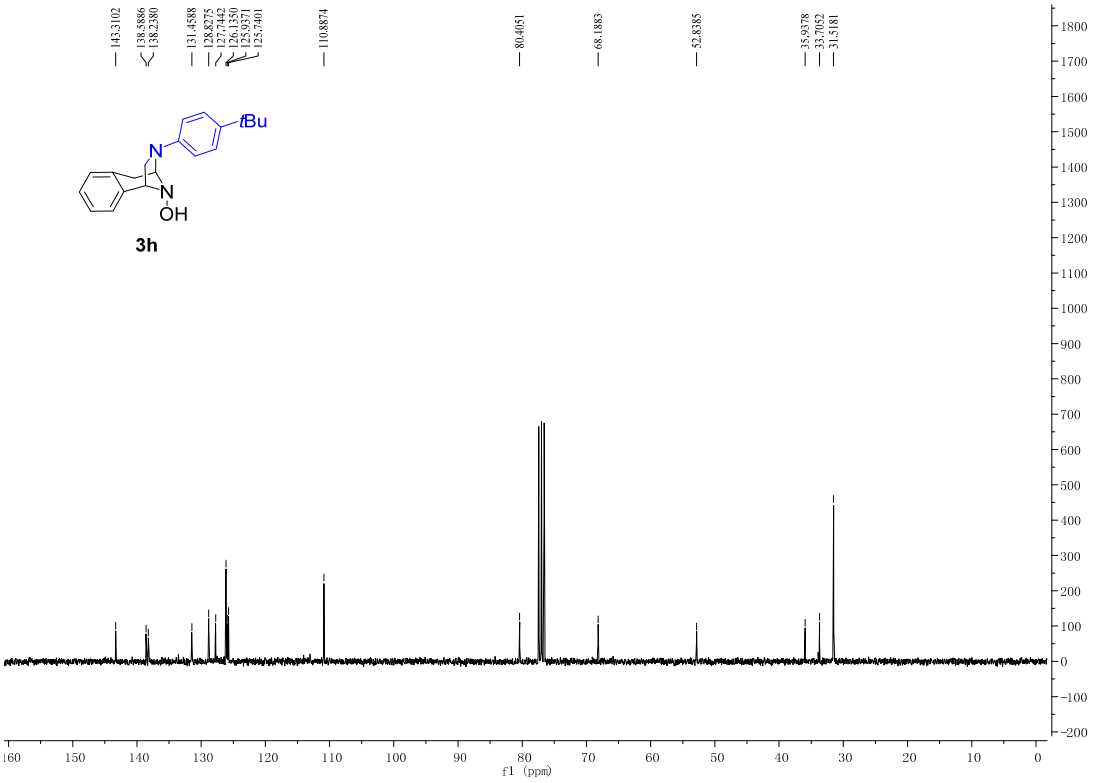
1.09
3.07
9.01
f1 (ppm)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tBu
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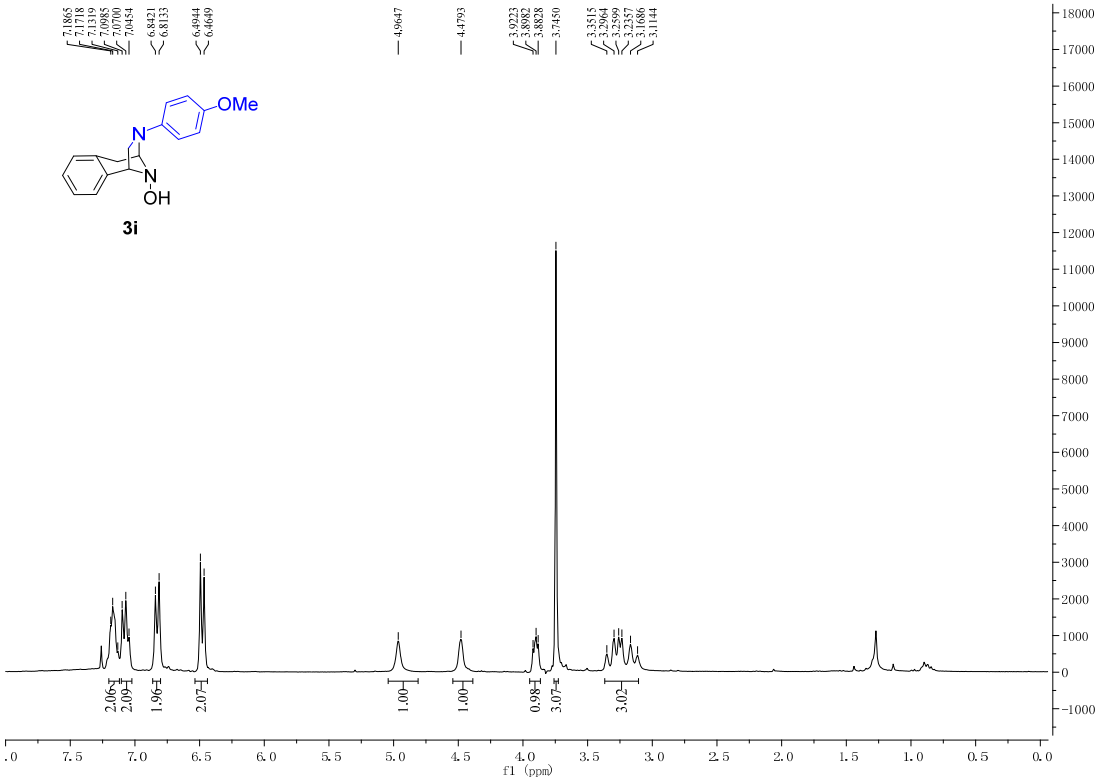
3h
f1 (ppm)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3.8982
3.8828
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3.3515
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OMe
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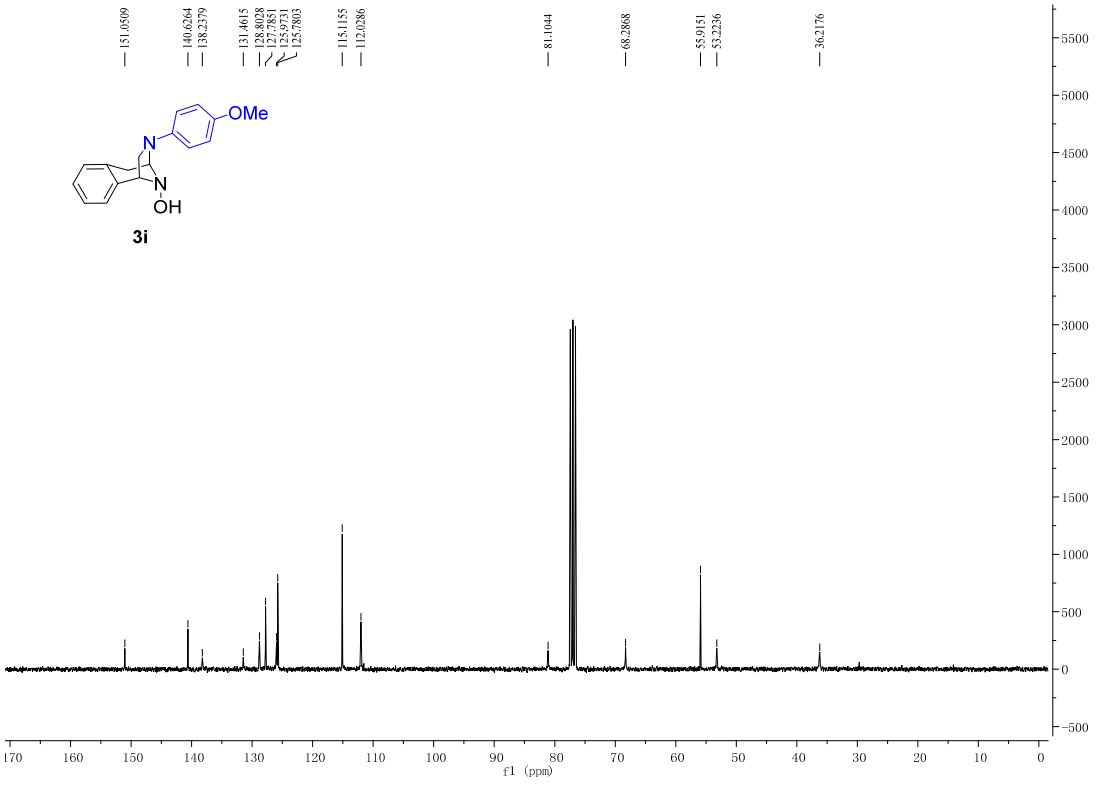
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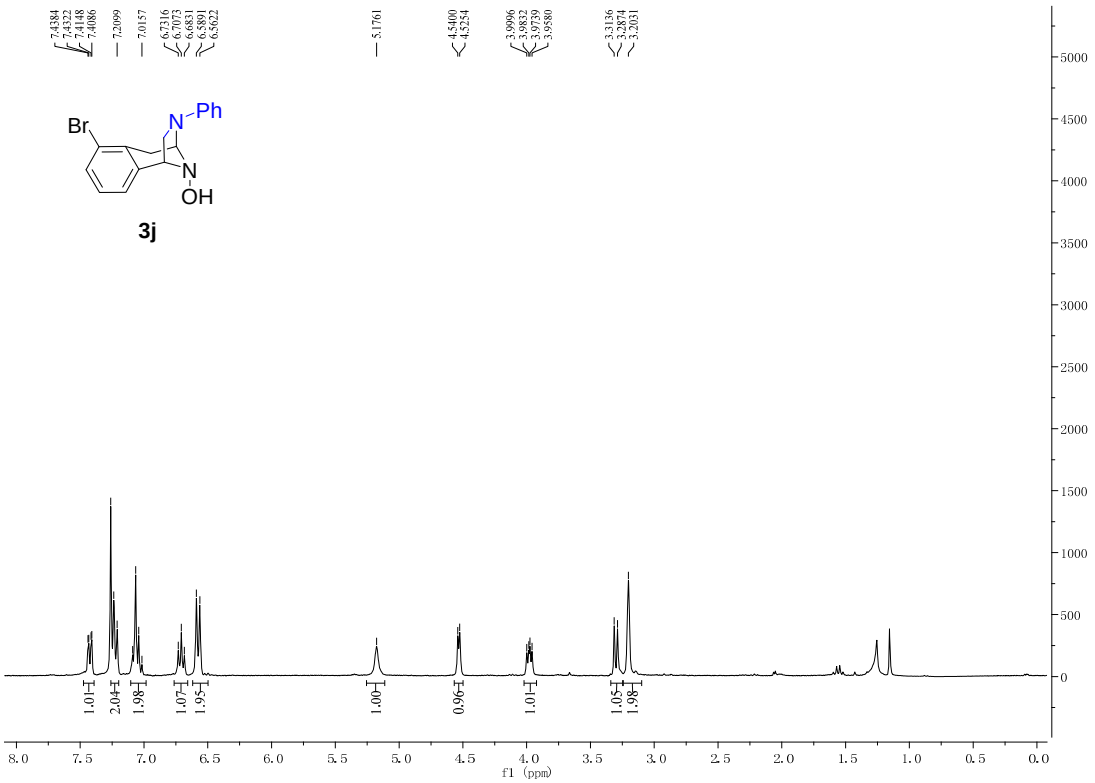
f1 (ppm)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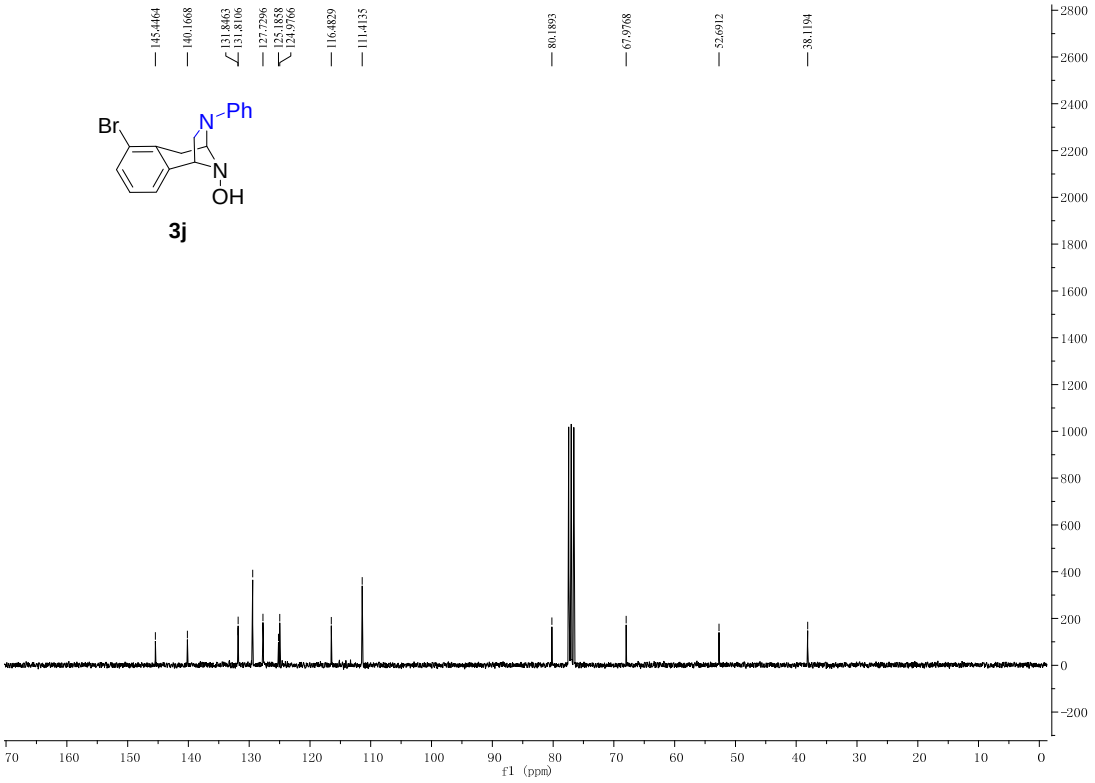
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Ph
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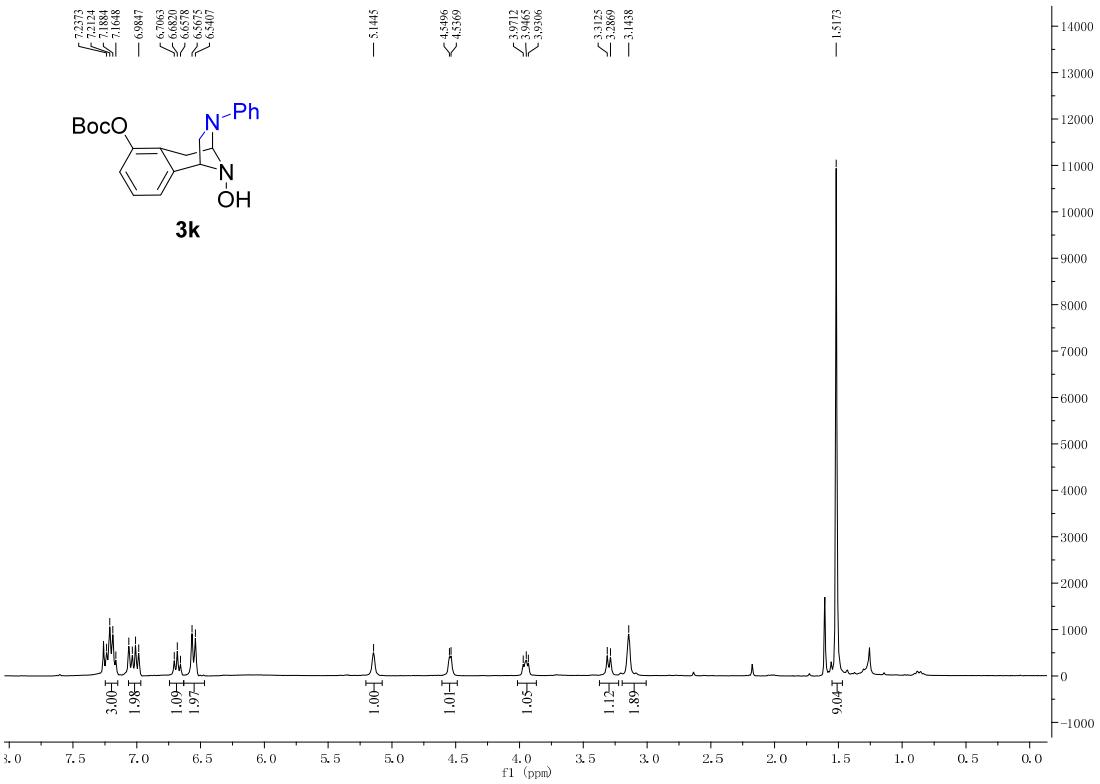
f1 (ppm)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4.5369
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BocO
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Ph
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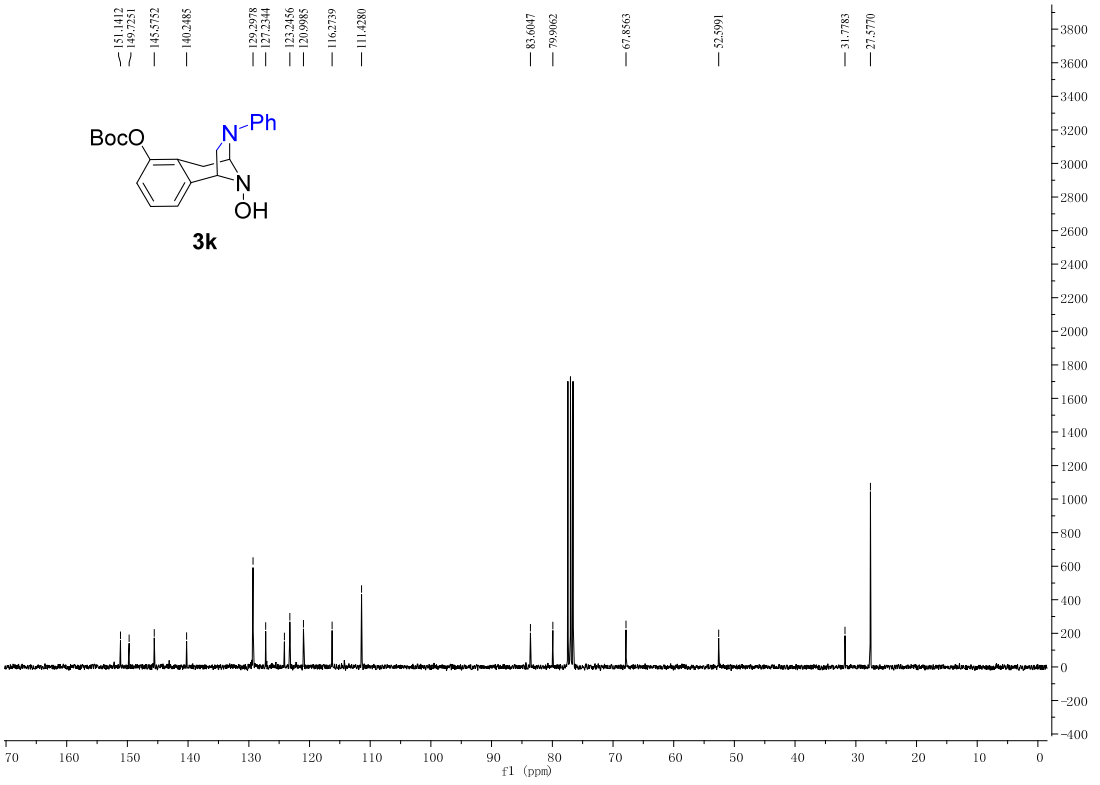
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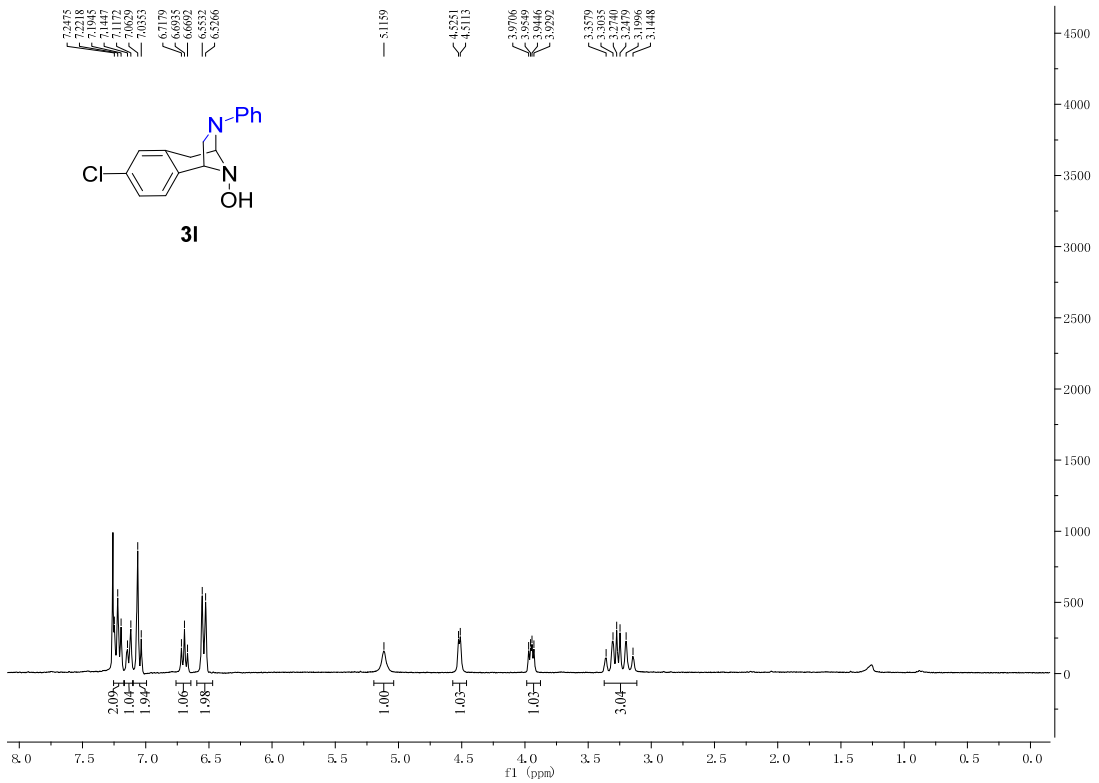
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3.9446
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3.3035
3.2740
3.2479
3.1996
3.1448
N
Ph
Cl
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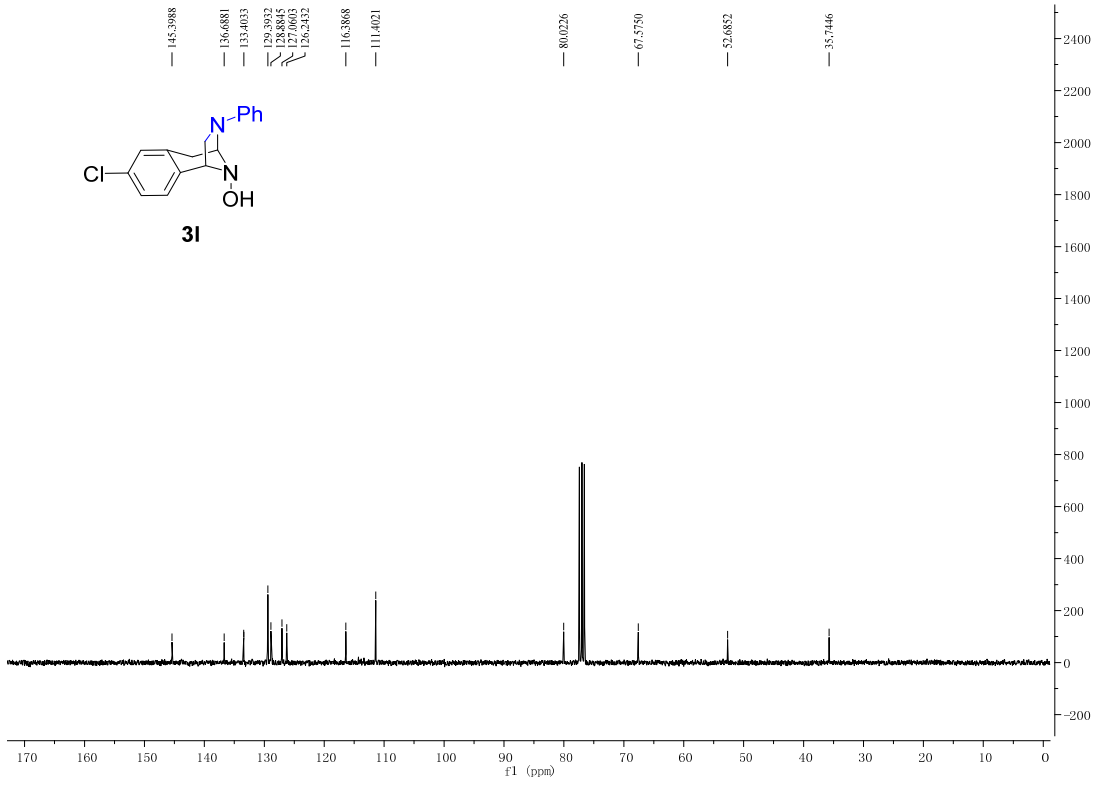
3.04
f1 (ppm)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Cl
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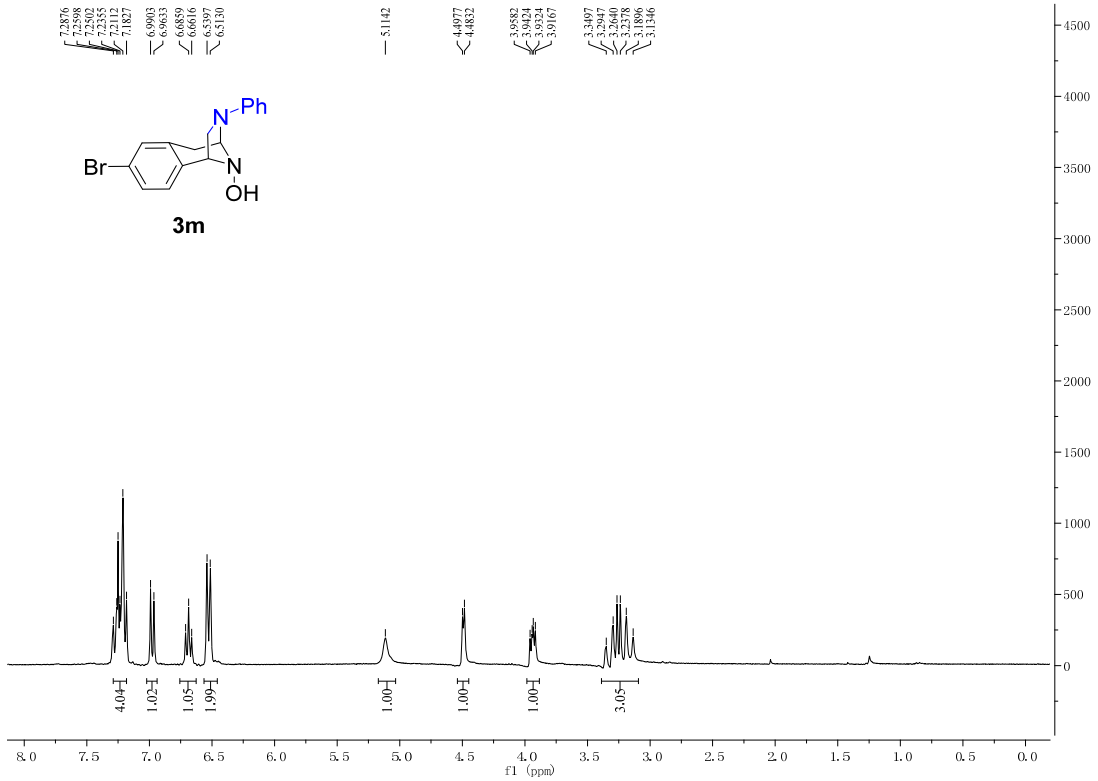
f1 (ppm)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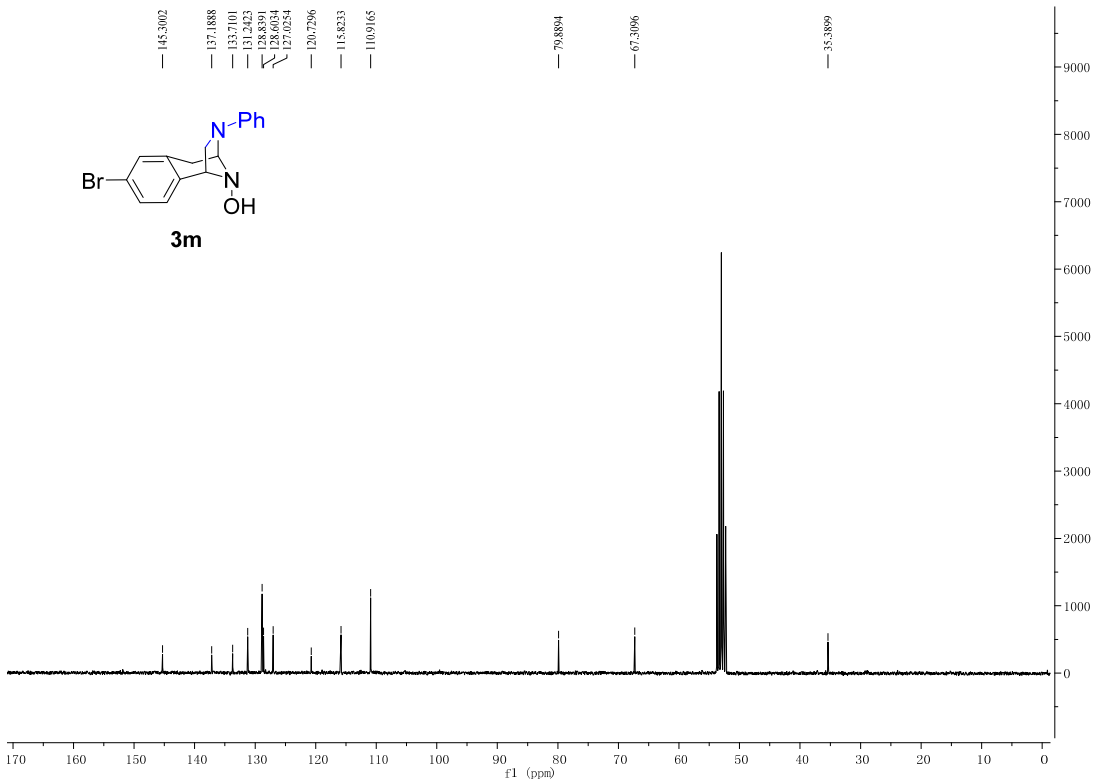
f1 (ppm)

3m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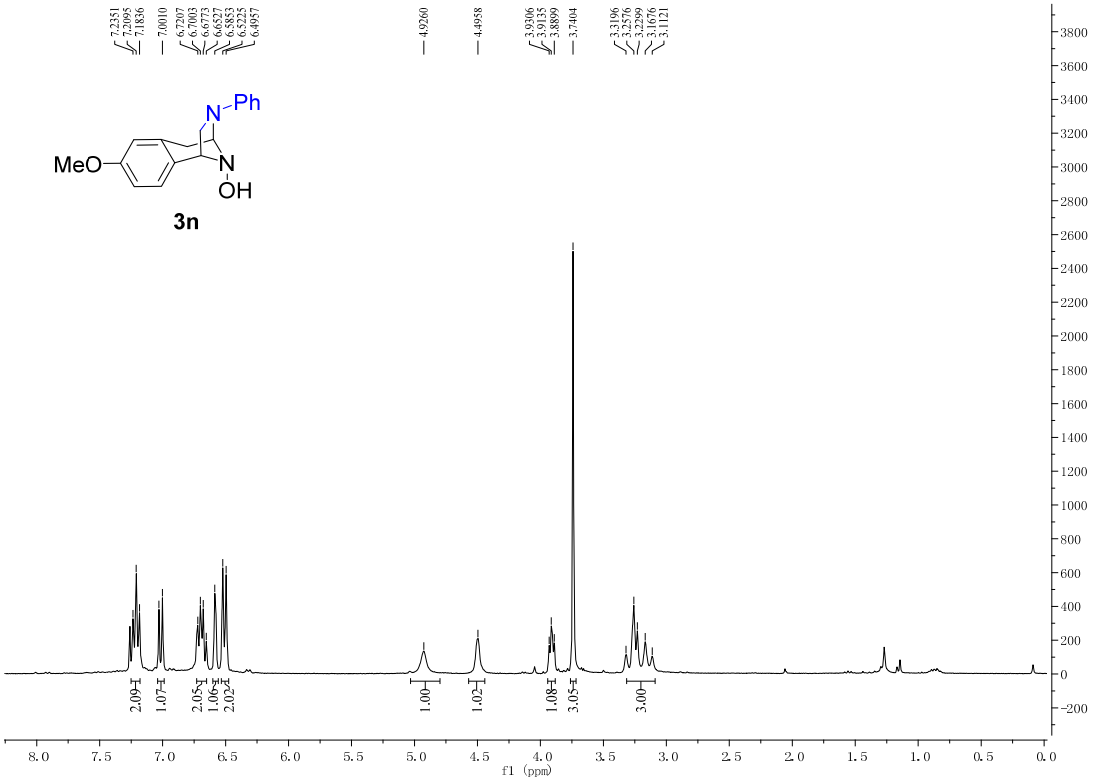
N-Ph
Br
N
OH
f1 (ppm)

3m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3.1121
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MeO
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OH
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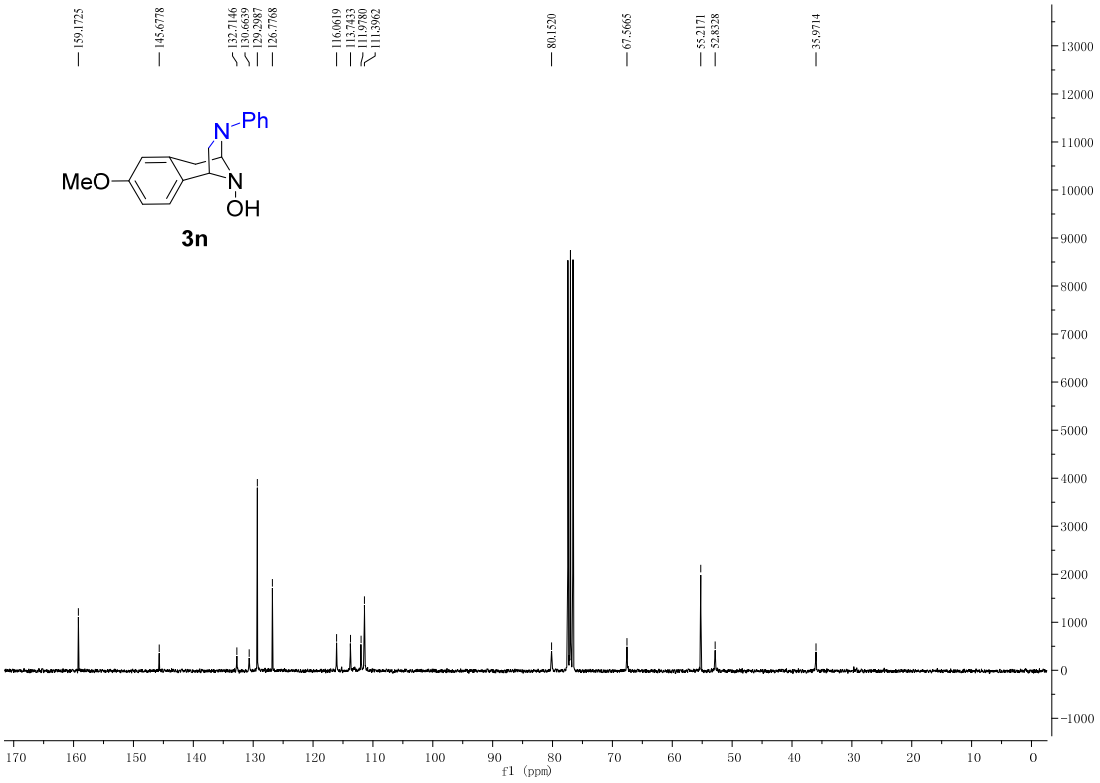
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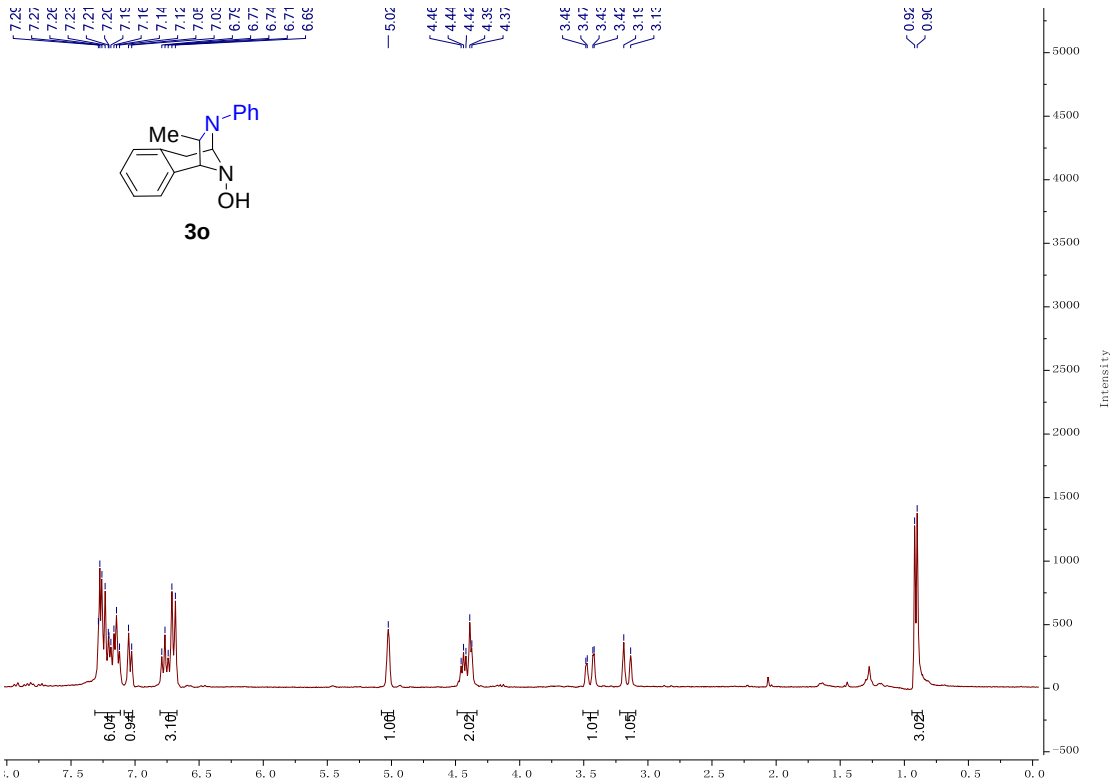
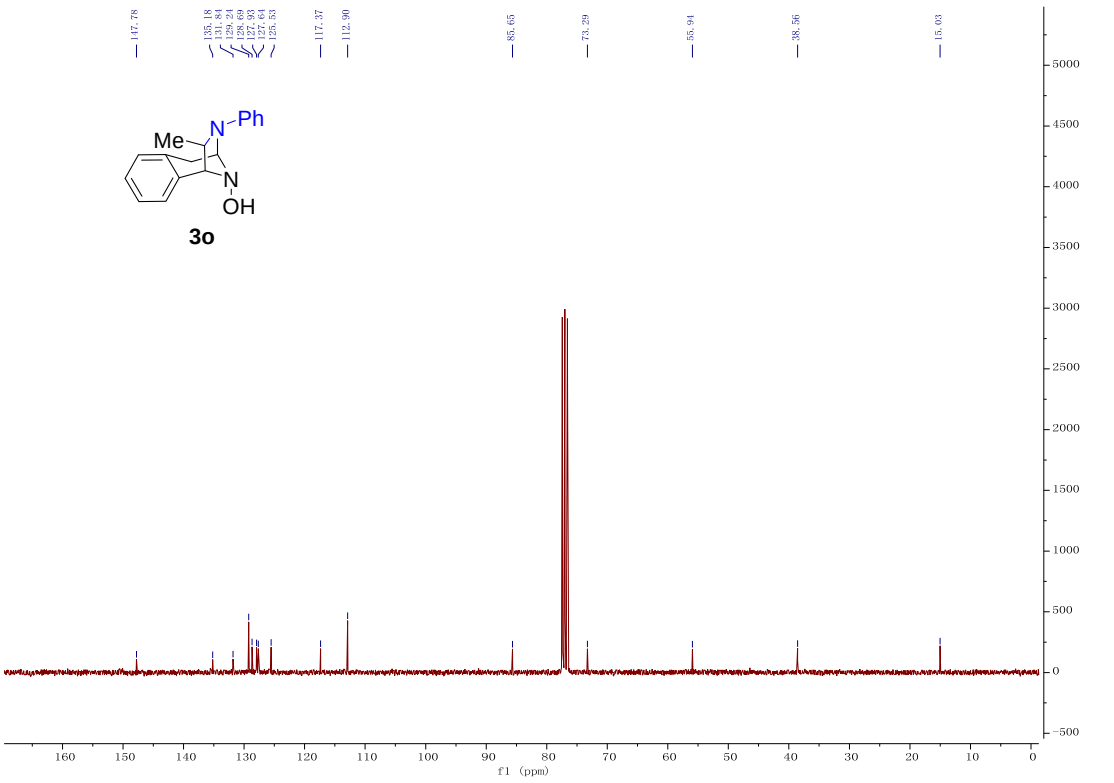
35.9714
N
Ph
MeO
N
OH
3n
f1 (ppm)


Me
N
Ph
N
OH
3o
Intensity
6.04
0.94
3.16
1.06
2.02
1.01
1.05
3.02
f1 (ppm)


Me
N
Ph
N
OH
3o
147.78
135.18
131.84
129.24
128.69
127.93
127.64
125.53
117.37
112.90
85.65
73.29
55.94
38.56
15.03
f1 (ppm)