Supporting Information

Direct *α*-C-H alkylation of alcohols *via* photoinduced hydrogen atom transfer

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1. General remark

Unless otherwise noted, all starting materials and reagents were commercially available and used directly without further purification. Anhydrous MeCN were purchased from Energy Chemical and used without purification. All of phenyl vinyl sulfone were prepared according to the literature procedures.[1] Reactions were examined by thin-layer chromatography (TLC) on Silica Gel (GF254) visualized under UV light (254 nm) or in an aqueous phosphomolybdic solution followed by heating. ^{1}H NMR and ^{13}C NMR spectra of materials and products were respectively recorded on 500MHz and 125MHz (BRUKER 500M) in $CDCl_3$. All chemical shifts were given as δ value (ppm) with reference to tetramethylsilane (TMS) as an internal standard. Products were purified by flash chromatography on 200-300 mesh silica gels. All melting points were determined using X-4 micro melting point apparatus without correction. HRMS were performed on Thermo Scientific LTQ-Obitrap-ETD HRMS-TOF with electron spray ionization (ESI). Single crystal X-ray diffraction were recorded on Agilent Supernova with CuKα (λ = 1.54184) radiation. All reactions were carried out under argon atmosphere in oven-dried glassware, unless otherwise noted. All reagents were purchased commercially and used as received unless otherwise noted.

2. Reaction setup

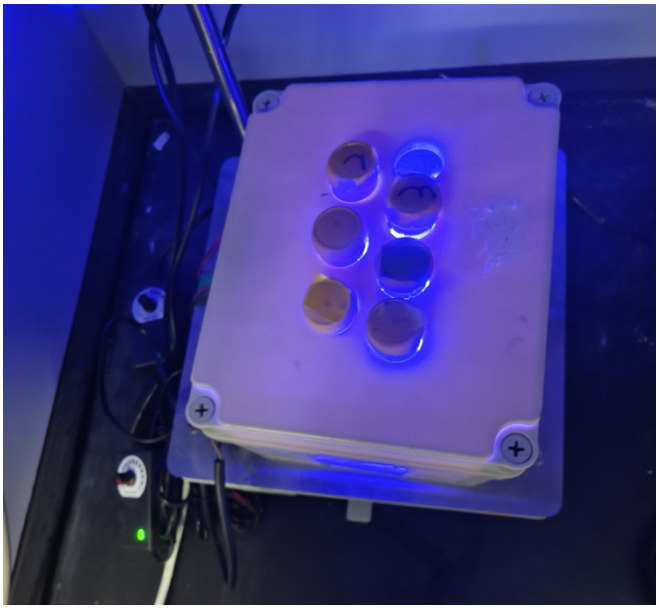
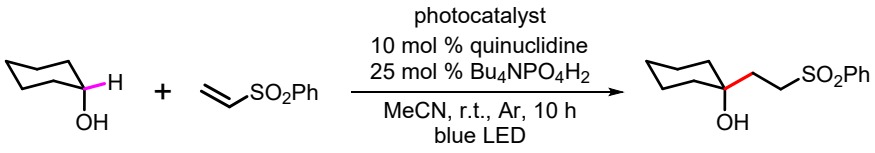


Figure S1 Photograph of the reaction set-up with a 20 W (450 nm) LED lamp, two mini fans and seven Schlenk tubes.

3. Experimental section

3.1 Process of optimizing reaction conditions

Table S1 Optimization of photocatalyst[a]



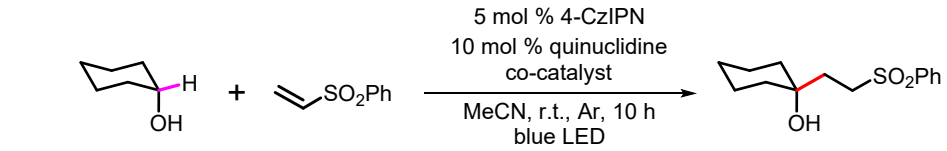
1a **2a** **3a**

entry	photocatalyst	yield (%)[b]
1	$Ir(ppy)_2(dtbbpy)PF_6$	43
2	$Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$	79
3	$Ru(bpy)_3(PF_6)_2$	-

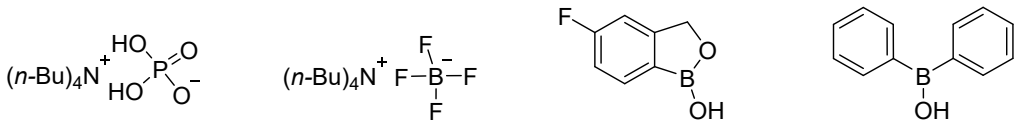
4	$Ru(bpy)_3Cl_2·6H_2O$	-
5	**4-CzIPN**	**94**
6	4-CzIPN (3 mol%)[c]	87
7	Eosin Y	23
8	$(Mes·Acr)ClO_4$	31

[a]Reaction conditions: substrates **1a** (0.6 mmol), **2a** (0.2 mmol), photocatalyst (5 mol%), quinuclidine (10 mol%), $(Bu)_4NPO_4H_2$ (25 mol%), MeCN (1.5 mL), Ar, the 20 W blue LED (450 nm), r.t., 10 h. [b]Isolated yield. [c]4-CzIPN with 3 mol% loading.

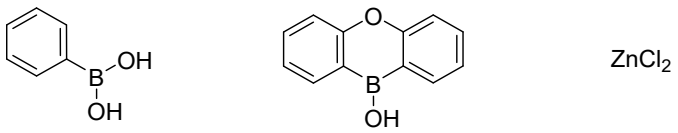
Table S2 Optimization of the co-catalyst[a]



1a **2a** **3a**



TBAP A1 A2 A3



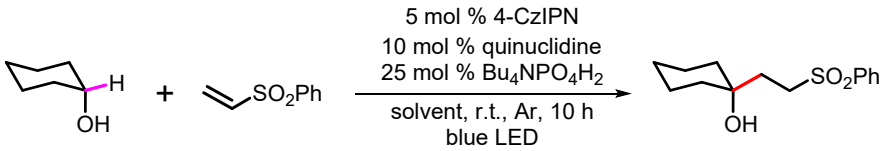
A4 A5 A6

entry	**co-catalyst**	yield (%)[b]
1	**TBAP**	**94**
2	A1	28
3	A2	57
4	A3	62

5	A4	45
6	A5	50
7	A6	23

[a]Reaction conditions: substrates **1a** (0.6 mmol), **2a** (0.2 mmol), photocatalyst (5 mol%), quinuclidine (10 mol%), co-catalyst (25 mol%), MeCN (1.5 mL), Ar, the 20 W blue LED (450 nm), r.t., 10 h. [b]Isolated yield.

Table S3 Optimization of solvent[a]

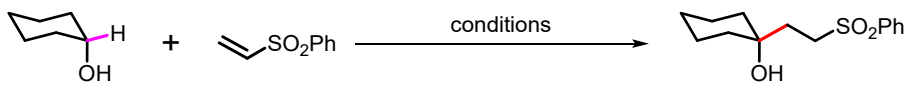


1a **2a** **3a**

entry	solvent	yield (%)[b]
1	**MeCN (0.13 M)**	**94**
2	MeCN (0.25 M)	90
3	DMSO (0.25 M)	65
4	DCE(0.25 M)	52
5	MeCN:DMSO=1:1（0.25 M）	81

[a]Reaction conditions: substrates **1a** (0.6 mmol), **2a** (0.2 mmol), photocatalyst (5 mol%), quinuclidine (10 mol%), TBAP (25 mol%), solvent, Ar, the 20 W blue LED (450 nm), r.t., 10 h. [b]Isolated yield.

Table S4 Control experiments[a]

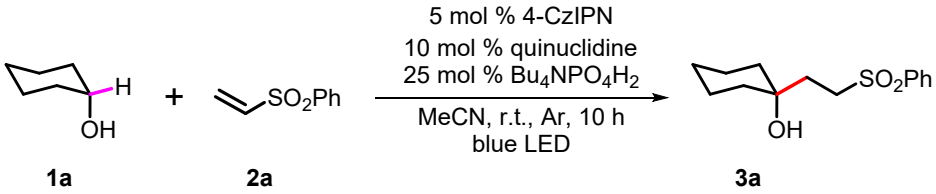


1a **2a** **3a**

Entry	deviations from standard conditions	Yield (**3a**)[b]
1	standard conditions	94
2	no blue LED	nd
3	no photocatalyst	nd
4	no $Bu_4NPO_4H_2$	nd
5	no quinuclidine	nd
6	under air	<5%

[a]Standard reaction conditions: substrates **1a** (0.6 mmol), **2a** (0.2 mmol), photocatalyst (5 mol%), quinuclidine (10 mol%), $(Bu)_4NPO_4H_2$ (25 mol%), MeCN (1.5 mL), Ar, the 20 W blue LED (450 nm), r.t., 10 h. [b]Isolated yield. r.t. = room temperature, nd = not detected

3.2 General procedures for the *α*-C-H alkylation of alcohols



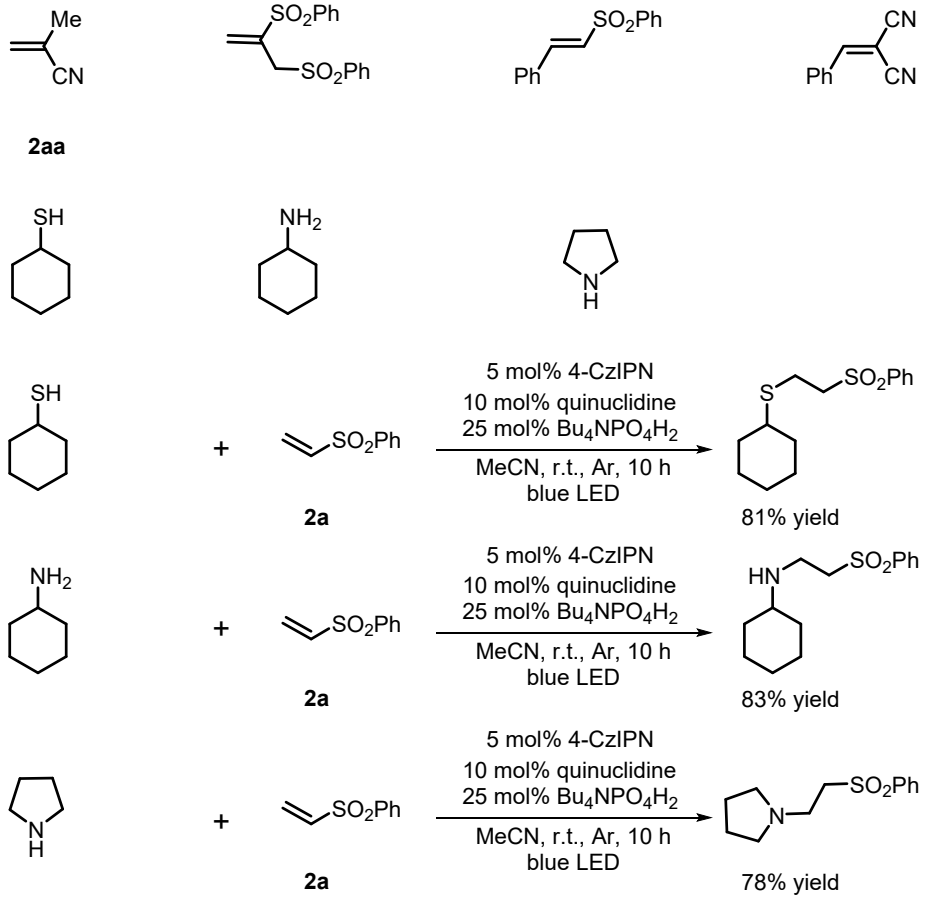
1a **2a** **3a**

Into a well-dried 10 mL seal tube, phenyl vinyl sulfone **2a** (33.6 mg, 0.2 mmol, 1.0 equiv.), 4-CzIPN (7.9 mg, 5 mol%), quinuclidine (1.2 mg, 10 mol%) and $(Bu)_4NPO_4H_2$ (17.0 mg, 25 mol%) were subsequently added, a 10 x 3 mm PTFE magnetic stir bar was added. the reaction mixture was degassed *via* freeze pump thaw (× 3 times) and refilled with Ar, then cyclohexanol **1a** (60 mg, 0.6 mmol, 3.0 equiv.) and MeCN (1.5 mL) was added *via* injection through the cap. The reaction tube was left stirring at room temperature under 20 W blue LED irradiation with two cooling fan. Then, the mixture was stirred for 10 h until the reaction was completed. The crude reaction mixture was concentrated under vacuum to furnish a wet residue, the residue was purified by flash column chromatography

(PE/EA) to afford the desired products.

Note 1: Unless otherwise noted, the reported yield is the isolated yield of two duplicate reactions.

3.3 Invalid substrate



4. The X-ray data of 3a (CCDC 2393054)

An amount of 20 mg **3a** were dissolved in acetonitrile (2 mL) on the brown small reagent bottle (5 mL), which acted as good solvent, and a layer of ether was injected on the surface of acetonitrile, and the cap is covered with a thin film, white crystals will be presented after four days. The data were collected at 293 K using a Bruker APEX-II CCD X-ray diffractometer equipped with a graphite monochromated Cu Kα radiation source (λ = 1.54184 Å) operation at 50 kV and 1.10 mA. Using Olex2[2], the structure was solved with the ShelXS[3]structure solution program using Direct

Methods and refined with the ShelXL[4]refinement package using Least Squares minimisation. Nonhydrogen atoms were refined with anisotropic displacement parameters during the final cycles. All hydrogen atoms were placed by geometrical considerations and were added to the structure factor calculations.

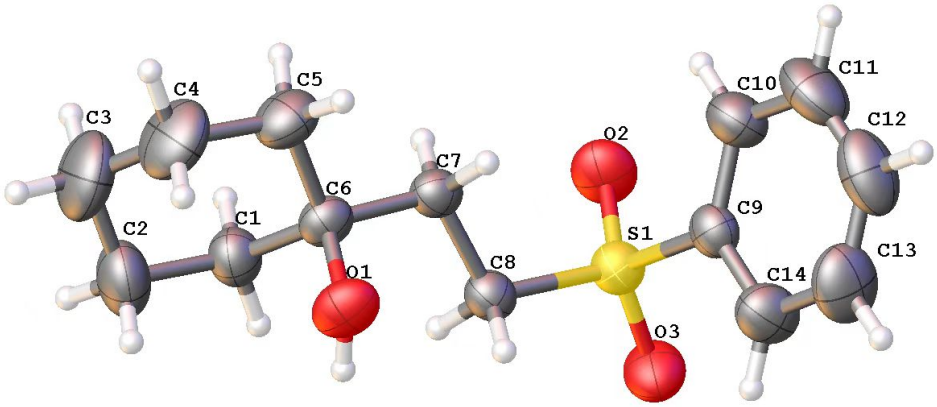


Figure S2 X-ray crystal structure of compound **3a**

Table S5 The crystal data and structure refinement for **3a**

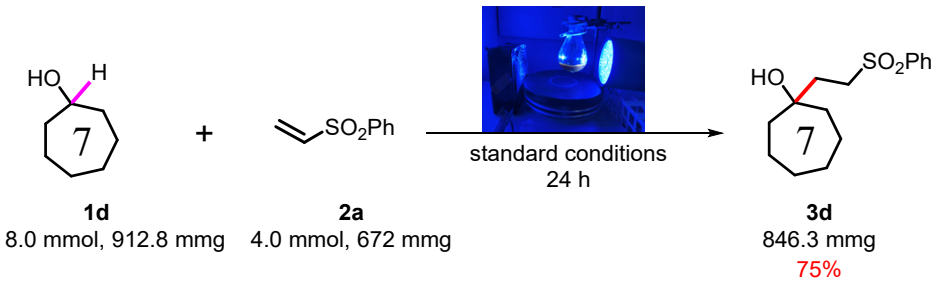
Identification code	**3a**
Empirical formula	$C_{14}H_{20}O_3S$
Formula weight	268.36
Temperature/K	293.00
Crystal system	monoclinic
Space group	C2/c
a/=C5	22.0552(3)
b/=C5	5.67162(7)
c/=C5	22.7509(3)
α/=B0	90
β/=B0	98.7926(12)
γ/=B0	90
Volume/=C53	2812.44(6)
Z	8
ρcalcg/cm3	1.268
μ/mm-1	2.035
F(000)	1152.0

Crystal size/mm3	0.06 =D7 0.05 =D7 0.04
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/=B0	7.864 to 135.928
Index ranges	-26 ≤ h ≤ 26, -6 ≤ k ≤ 5, -27 ≤ l I-= 4; 25
Reflections collected	7352
Independent reflections	2539 [Rint =3D 0.0262, Rsigma =3D 0.0279]
Data/restraints/parameters	2539/0/171
Goodness-of-fit on F2	1.142
Final R indexes [I>=3D2σ (I)]	R1 =3D 0.0453, wR2 =3D 0.1197
Final R indexes [all data]	R1 =3D 0.0478, wR2 =3D 0.1221
Largest diff. peak/hole / e =C5-3	0.24/-0.51

Datablock: mwj_auto

```
Bond precision:   C-C = 0.0030 A          Wavelength=1.54184

Cell:             a=22.0552(3)    b=5.67162(7)         c=22.7509(3)
                  alpha=90        beta=98.7926(12)     gamma=90
Temperature:      293 K

                  Calculated                  Reported
Volume            2812.44(6)                  2812.44(6)
Space group       C 2/c                       C 1 2/c 1
Hall group        -C 2yc                      -C 2yc
Moiety formula    C14 H20 O3 S                C14 H20 O3 S
Sum formula       C14 H20 O3 S                C14 H20 O3 S
Mr                268.36                      268.36
Dx,g cm-3         1.268                       1.268
Z                 8                           8
Mu (mm-1)         2.035                       2.035
F000              1152.0                      1152.0
F000'             1157.78
h,k,lmax          26,6,27                     26,6,27
Nref              2570                        2539
Tmin,Tmax         0.885,0.922                 0.415,1.000
Tmin'             0.885

Correction method= # Reported T Limits: Tmin=0.415 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.988           Theta(max)= 67.964

R(reflections)= 0.0453( 2297)                wR2(reflections)=
                                             0.1221( 2539)
S = 1.142                  Npar= 171
```

5. Reaction procedure for gram-scale reaction



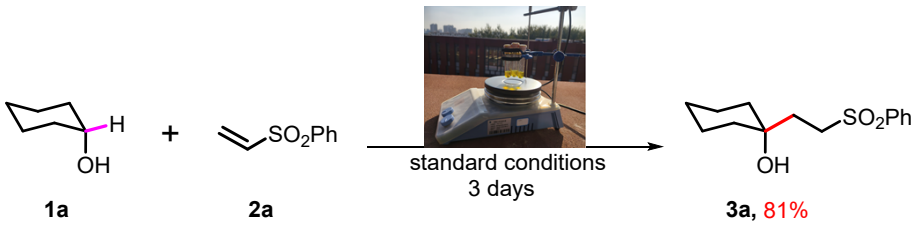
1d
8.0 mmol, 912.8 mmg

2a
4.0 mmol, 672 mmg

3d
846.3 mmg
75%

Into a well-dried 50-mL seal tube, phenyl vinyl sulfone **2a** (672 mg, 4.0 mmol, 1.0 equiv.) 4-CzIPN (157.6 mg, 5 mol%), quinuclidine (44.4 mg, 10 mol%), $(Bu)_4NPO_4H_2$ (340 mg, 25 mol%) were subsequently added, a 10 x 3 mm PTFE magnetic stir bar was added. The reaction mixture was degassed *via* freeze pump thaw (× 3 times) and refilled with Ar. Then cycloheptanol **1d** (912.8 mg, 8.0 mmol, 3.0 equiv.) and MeCN (30 mL) was added *via* injection through the cap.The reaction tube was left stirring at room temperature under 20 W blue LED irradiation with two cooling fan (**shown above**). The reaction was monitored by TLC. About 40 h, the crude reaction mixture was concentrated under vacuum to furnish a wet residue, The residue was purified by flash column chromatography (PE/EA) to afford the desired products **3d** (75%, 846.3 mg).

6. Sunlight experiment



1a

2a

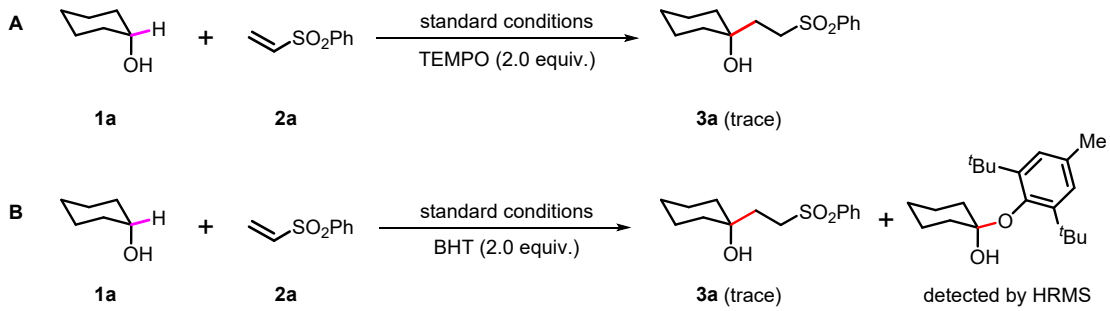
3a, 81%

Into a well-dried 10-mL seal tube, phenyl vinyl sulfone **2a** (33.6 mg, 0.2 mmol, 1.0 equiv.), 4-CzIPN (7.9 mg, 5 mol%), quinuclidine (1.2 mg, 10 mol%) and $(Bu)_4NPO_4H_2$ (17.0 mg, 25 mol%) were subsequently added, a 10 x 3 mm PTFE magnetic stir bar was added. the reaction mixture was degassed *via* freeze pump thaw (× 3 times) and refilled with Ar. Then cyclohexanol **1a** (60 mg, 0.6 mmol, 3.0 equiv.) and MeCN (1.5 mL) was

added *via* injection through the cap. The reaction tube was left stirring at room temperature under sunlight irradiation for 3 days until the reaction was completed, the crude reaction mixture was concentrated and purified by flash column chromatography to afford the desired product **3a** in 81% yield.

7. Mechanistic studies

7.1. The radical trappinmg experiments



Scheme S1 The radical quenching and trappinmg experiments

Into a well-dried 10-mL seal tube, phenyl vinyl sulfone **2a** (33.6 mg, 0.2 mmol, 1.0 equiv.), 4-CzIPN (7.9 mg, 5 mol%), quinuclidine (1.2 mg, 10 mol%), $(Bu)_4NPO_4H_2$ (17.0 mg, 25 mol%) and TEMPO (62.4 mg, 0.4 mmol, 2.0 equiv.) or BHT (88.0 mg, 0.4 mmol, 2.0 equiv.) were subsequently added, a 10 x 3 mm PTFE magnetic stir bar was added. the reaction mixture was degassed *via* freeze pump thaw (× 3 times) and refilled with Ar. Then cyclohexanol **1a** (60 mg, 0.6 mmol, 3.0 equiv.) and MeCN (1.5 mL) was added *via* injection through the cap. The reaction tube was left stirring at room temperature under 20 W blue LED irradiation with two cooling fan. Then, the mixture was stirred for 10 h until the reaction was completed, only trace amounts of the product were obtained. When the reaction has been carried out for 1 h, the reaction mixture was detected by HRMS. The results revealed that the reaction mixture contained intermediate **IV** (Figure S3).

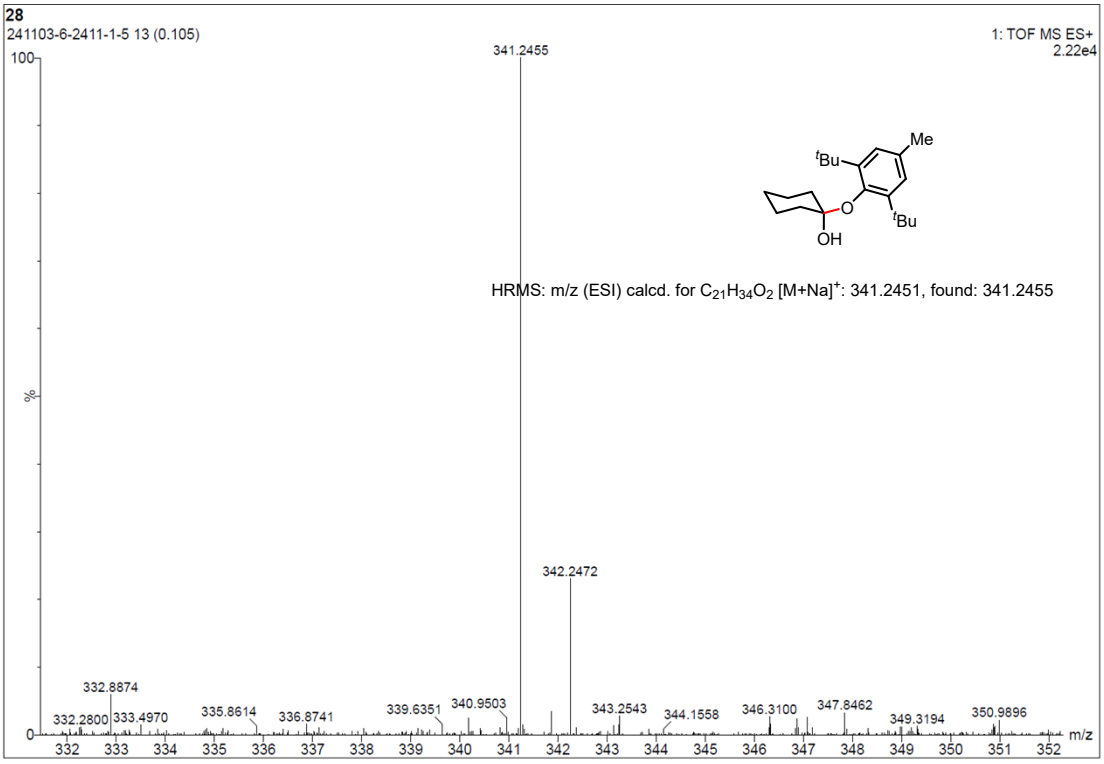
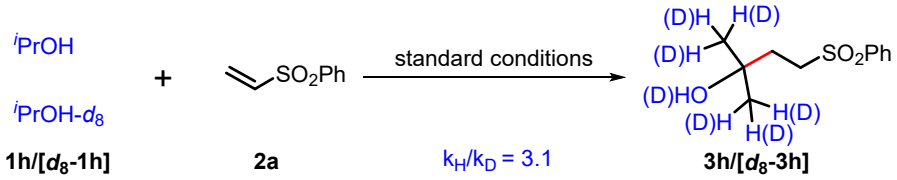


Figure S3 HRMS of the adduct of BHT

7.2 KIE Studies



Scheme S2 The kinetic isotopic effect determination from intermolecular competition reactions employing isopropanol and d_8-isopropanol as coupling partner.[a]

Into a well-dried 10-mL seal tube, phenyl vinyl sulfone **2a** (33.6 mg, 0.2 mmol, 1.0 equiv.), 4-CzIPN (7.9 mg, 5 mol%), quinuclidine (1.2 mg, 10 mol%), $(Bu)_4NPO_4H_2$ (17.0 mg, 25 mol%) were subsequently added, a 10 x 3 mm PTFE magnetic stir bar was added. the reaction mixture was degassed *via* freeze pump thaw (× 3 times) and refilled with Ar. Then ^{i}PrOH **1h** (0.3 mmol, 1.5 equiv.), d_8-^{i}PrOH [***d*$_8$-1h**] (0.3 mmol, 1.5 equiv.) and MeCN (1.5 mL) was added *via* injection through the cap. The reaction tube was left stirring at room temperature under 20 W blue LED irradiation with two cooling fan. Then, the mixture was stirred for 10 h until the

reaction was completed (monitored by TLC). the crude reaction mixture was concentrated under vacuum to furnish a wet residue, The residue was purified by flash column chromatography to afford the desired product **3h**/[***d*$_8$-1h**]. ^{1}H NMR analysis of the mixture showed a modest value of K_H/K_D = 3.1 (see the attached spectrum).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93-7.91 (m, 2H), 7.68-7.65 (m, 1H), 7.59-7.56 (dd, *J* = 8.5, 7.2 Hz, 2H), 3.27-3.23 (m, 2H), 1.89-1.86 (m, 2H), 1.21 (s, 4.5H).

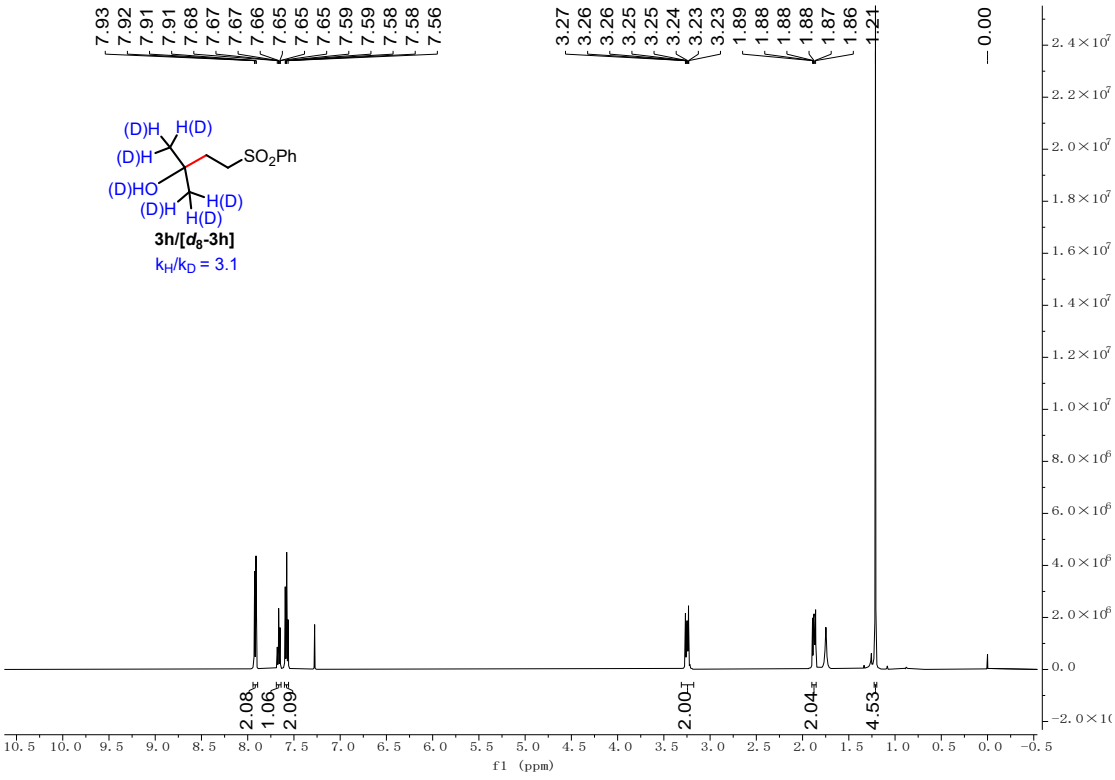
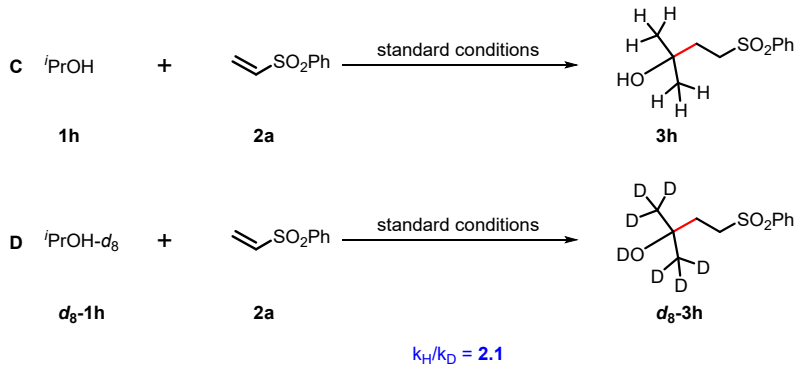


Figure S4 ^{1}H NMR analysis of the mixture **3h**/[***d*$_8$-3h**]



Scheme S3 The kinetic isotopic effect determination from two parallel reactions employing isopropanol and d_8-isopropanol as coupling partner[a]

[a]Conditions employed 20 W Blue Leds, isopropanol **1h** (or d_8-isopropanol ***d*$_8$-1h**) (0.6 mmol), **2a** (0.2 mmol), 4-CzIPN (0.5 mol%), Quinuclidine (10 mol%), TBAP (25 mol%), MeCN (1.5 mL), the reaction mixture was degassed *via* freeze pump thaw (× 3 times) and refilled with Ar, r.t.; [b]Analyzed by NMR spectroscopy for the formation of product with 1,3,5-trimethoxybenzene as an internal standard.

3h and [***d*$_8$-1h**] was obtained respectively according to the general reaction procedure above. ^{1}H NMR analysis of **3h** and [***d*$_8$-1h**] showed a modest value of K_H/K_D = 2.3. [***d*$_8$-1h**] was analyzed by ^{1}H NMR.

^{1}H NMR (500 MHz, $CDCl_3$):δ 7.92 (d, *J* = 7.7 Hz, 2H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.58 (t, *J* = 7.7 Hz, 2H), 3.26-3.22 (m, 2H), 1.89-1.86 (m, 2H).

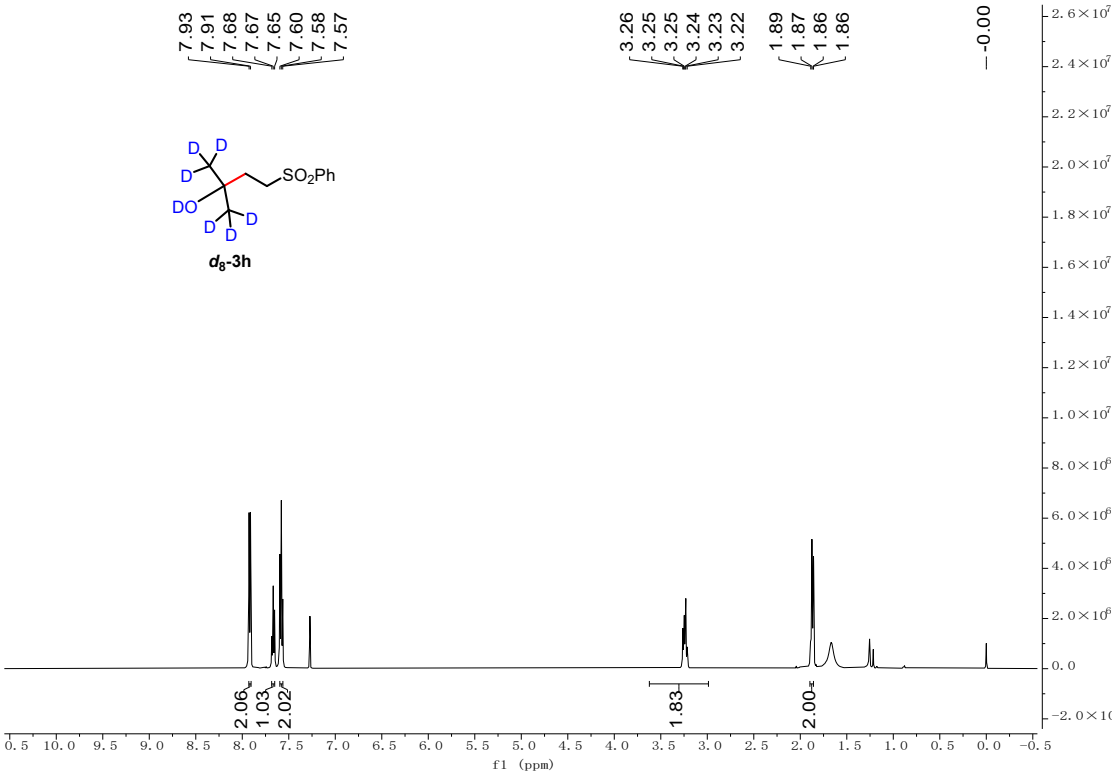


Figure S5 The kinetic isotopic effect determination from intermolecular competition reactions employing isopropanol and d_8-isopropanol as coupling partner[a]

7.3 Stern-Volmer fluorescence quenching experiments

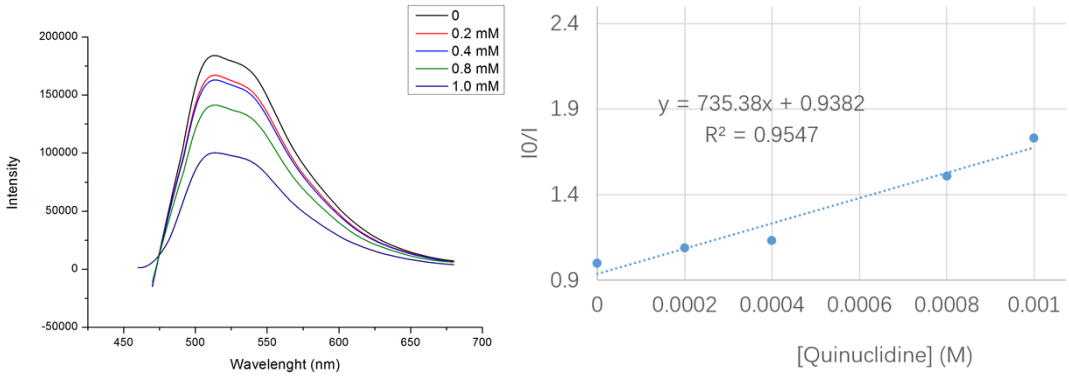


Figure S6 Stern-Volmer plot of 4-CzIPN with varying concentration of quinuclidine: 5.0 mL sample solution containing 0.1 mL of 4-CzIPN (0.001 M) together with 0.1- 0.5 mL of quinuclidine (0.01 M).

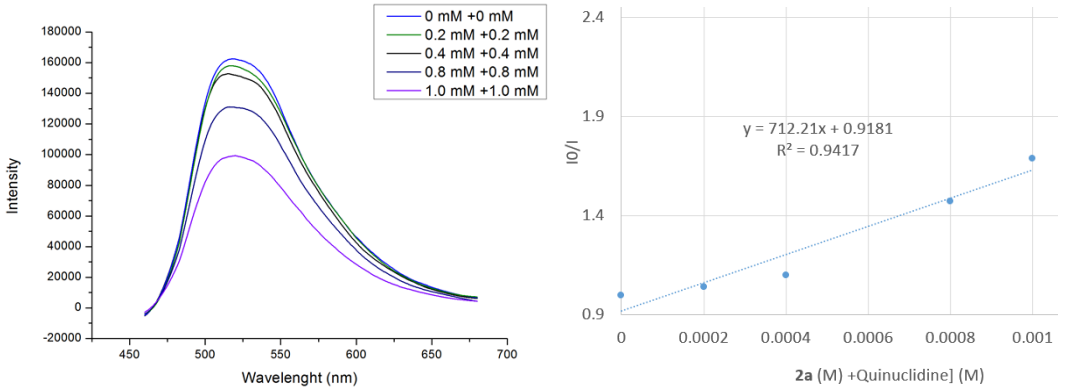


Figure S7 Stern-Volmer plot of 4-CzIPN with varying concentration of phenyl vinyl sulfone **2a** and quinuclidine: 5.0 mL sample solution containing 0.1 mL of 4-CzIPN (0.001 M) together with 0.1- 0.5 mL of **2a** (0.01 M) and quinuclidine (0.01M).

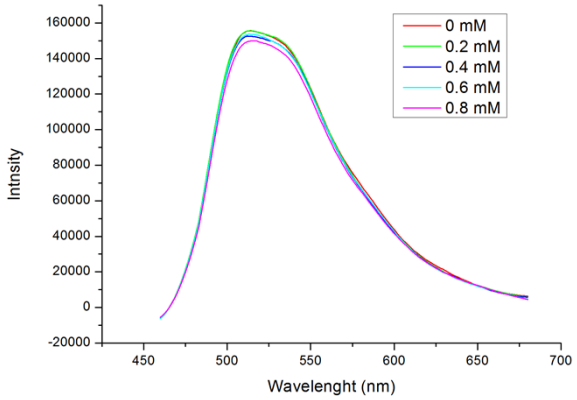


Figure S8 Stern-Volmer plot of 4-CzIPN with varying concentration of cyclohexanol: 5.0 mL sample solution containing 0.1 mL of 4-CzIPN

(0.001 M) together with 0.1- 0.5 mL of cyclohexanol (0.01 M).

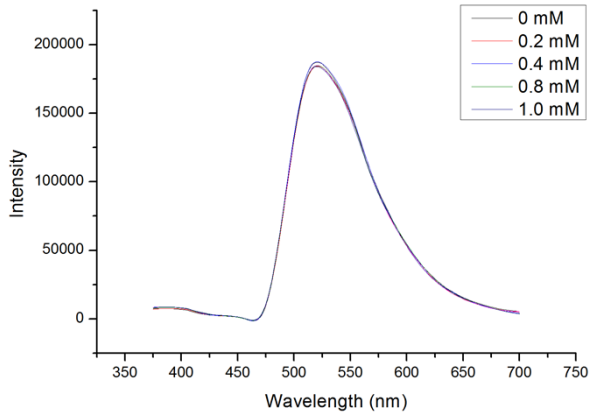
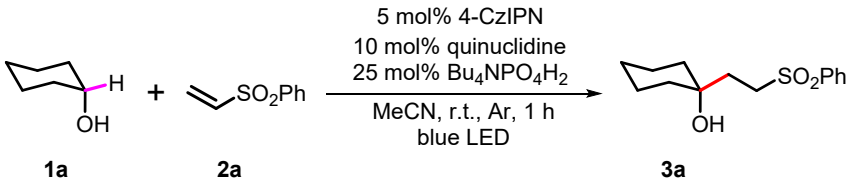


Figure S9 Stern-Volmer plot of 4-CzIPN with varying concentration of phenyl vinyl sulfone **2a**: 5.0 mL sample solution containing 0.1 mL of 4-CzIPN (0.001 M) together with 0.1- 0.5 mL of **2a** (0.01 M).

7.4 Quantum yield determination[5-9]

To further investigate the mechanism of the reactions, we employed the model reaction of **1a** with **2a** to **3a** measure the quantum yield.



Into a well-dried 10 mL seal tube, phenyl vinyl sulfone **2a** (33.6 mg, 0.2 mmol, 1.0 equiv.), 4-CzIPN (7.9 mg, 5 mol%), quinuclidine (1.2 mg, 10 mol%) and $(Bu)_4NPO_4H_2$ (17.0 mg, 25 mol%) were subsequently added, a 10 x 3 mm PTFE magnetic stir bar was added. the reaction mixture was degassed *via* freeze pump thaw (× 3 times) and refilled with Ar, then cyclohexanol **1a** (60 mg, 0.6 mmol, 3.0 equiv.) and MeCN (1.5 mL) was added *via* injection through the cap. The reaction tube was left stirring at room temperature under 20 W blue LED irradiation for 1 h with two cooling fan. The reaction mixture was concentrated in vacuo and analyzed by ^1H NMR spectrum using CH_2Br_2 as an internal standard. The quantum yield is calculated to be 0.21.

$$\phi = \frac{n_x}{n_p} = \frac{\frac{n_x}{\Delta E \times S \times t}}{\frac{}{N_A h v}} = \frac{n_x \times N_A \times h \times c}{\Delta E \times S \times t \times \lambda}$$

$$= \frac{0.04 \times 10^{-3} mol \times 6.022 \times 10^{23} \times 6.626 \times 10^{-34} J{\cdot}s \times 2.998 \times 10^{8} m{\cdot}s^{-1}}{(7.0 \times 10^{-3} W{\cdot}cm^{-2} \times 2cm^{-2}) \times 3600s \times 450 \times 10^{-9} m}$$

= 0.21

n_x is the amount of photochemical or photophysical events x occurred during irradiation, n_p is the number of photons absorbed by the reactant. E is the radiant power. S is the irradiated area: 2 cm^2; t is the irradiated time: 3600 s; N_A is the Avogadro constant: 6.022×10^{23}/mol; h is the Planck constant: 6.626×10^{-34} J·s; v is the frequency of incident light; c is velocity of light 2.998×10^{8} m/s. λ is the wavelength: 450 nm; n_x was analyzed by ^{1}H NMR, □E was measured by ILT1400 Portable Radiometer/Photometer. The quantum yield (Φ = 0.21) implies that the directed alkylation reactions is highly efficient, and the transformation proceeds through a photoredox catalytic pathway rather than radical chain propagation.

7.5 Time profile of the transformation with the light on/off over time

From the profile of the reaction with the light on/off over time, it was observed that the transformation progressed smoothly under light, but no further conversion was observed when the light is turned off. This result, together with a quantum yield ϕ = 0.21, suggest that the transformation proceeds through a photoredox catalytic pathway rather than radical chain propagation.

Ten standard reaction mixtures in 10 mL seal tubes were charged with phenyl vinyl sulfone **2a** (33.6 mg, 0.2 mmol, 1.0 equiv.), 4-CzIPN (7.9 mg, 5 mol%), quinuclidine (1.2 mg, 10 mol%) and $(Bu)_4NPO_4H_2$ (17.0 mg, 25 mol%). the reaction mixture was degassed *via* freeze pump thaw (× 3 times) and refilled with Ar, then cyclohexanol **1a** (60 mg, 0.6 mmol, 3.0 equiv.) and MeCN (1.5 mL) was added *via* injection through the cap. The reaction tubes was left stirring at room temperature under 20 W blue LED irradiation with two cooling fan. After 2 h, the light source was turned off, and one seal tube was removed from the irradiation setup for analysis. The remaining nine seal tubes were stirred in the absence of light for an

additional 2 h. Then, one seal tube was removed for analysis, and the light source was turned back on to irradiate the remaining eight reaction mixtures. After an additional 2 h of irradiation, the light source was turned off, and one seal tube was removed for analysis. The remaining seven seal tubess were stirred in the absence of light for an additional 2 h. Then, a seal tube was removed for analysis, and the light source was turned back on to irradiate the remaining six reaction mixtures. Repeat the above operation 6 times (The reaction mixtures were analyzed by GC against an internal standard).

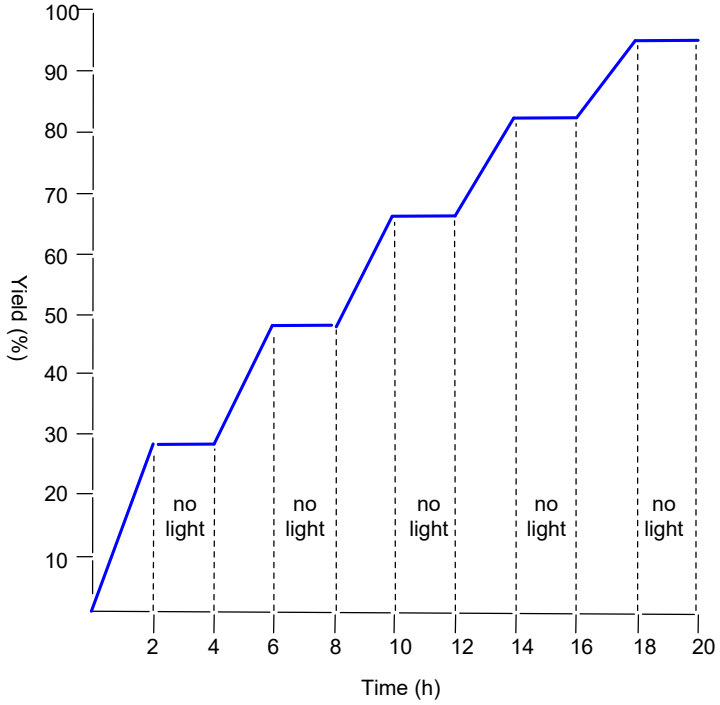


Figure S10 Time profile of the transformation with the light on/off over time

7.6 The NMR study for α-C-H alkylation reaction

General procedure: To a solution of TBAP (0.1 mmol, 1.0 equiv.) in $CDCl_3$ (0.6 mL), was added methanol (0.1-0.2 mmol, 100-200 % mol). The ^1H NMR data of the resulting solution were then collected accordingly.

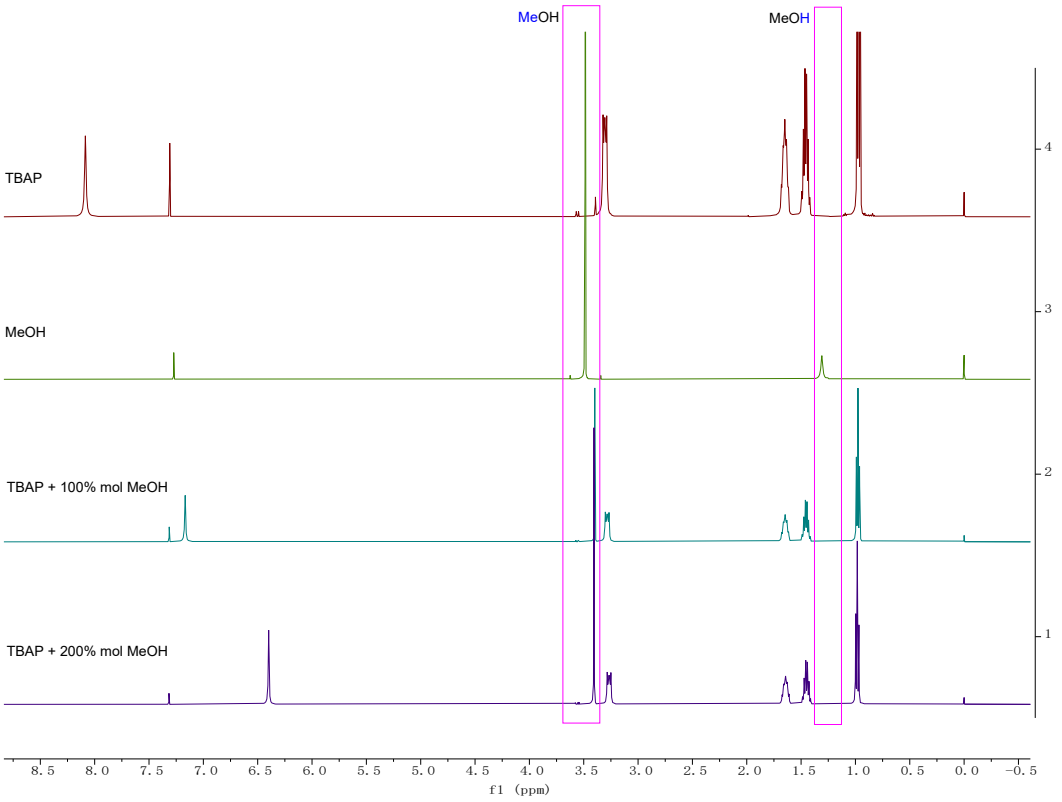


Figure S11 The ^{1}H NMR study of TBAP with methanol

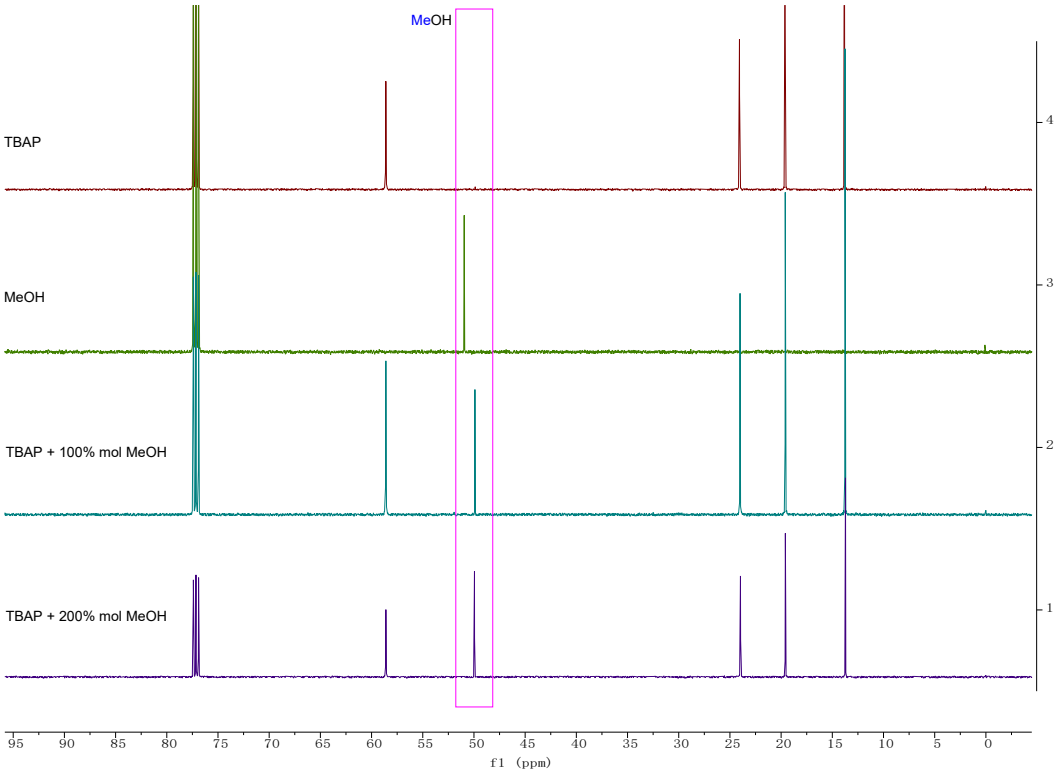
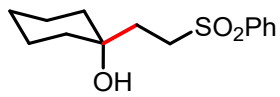


Figure S12 The $^{13}C\{^{1}H\}$ NMR study of TBAP with methanol

Note: $^{13}C\{^{1}H\}$ NMR signal of methanol 50.95 (singlet), and the mixture of

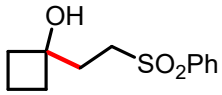
TBAP and methanol (1:1) change to 50.95-49.95 and According to previous studies, it has been shown that the strength of intermolecular hydrogen bonding between alcohols and acceptor molecules which is reflected in the $^{13}C\{^{1}H\}$ NMR chemical shift.[10-12]

8. Data of products



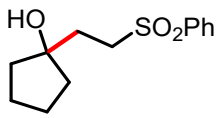
1-(2-(phenylsulfonyl)ethyl)cyclohexan-1-ol (3a)

Product **3a** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as white solid (50.3 mg, 94% yield), melting point: 69 - 71 °C.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 - 7.91 (m, 2H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 2H), 3.26 - 3.23 (m, 2H), 1.88 - 1.84 (m, 2H), 1.53 - 1.48 (m, 8H), 1.37 (d, *J* = 9.9 Hz, 2H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.3, 133.6, 129.3, 127.9, 70.3, 51.3, 37.44, 34.2, 25.5, 22.0.
HRMS: m/z (ESI) calcd. for $C_{14}H_{20}O_3SNa$ $[M+Na]^+$ 291.1025, found 291.1035.



1-(2-(phenylsulfonyl)ethyl)cyclobutan-1-ol (3b)

Product **3b** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as light yellow oil (42.7 mg, 89% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 - 7.92 (m, 2H), 7.65 (dd, *J* = 4.8, 3.7 Hz, 1H), 7.58 (t, *J* = 7.7 Hz, 2H), 3.22 - 3.19 (m, 2H), 2.08 (s, 1H), 2.02 (dd, *J* = 8.1, 3.3 Hz, 5H), 1.75 (s, 2H), 1.52 (dt, *J* = 11.4, 8.9 Hz, 1H).
^{13}C{1H} NMR (125 MHz, $CDCl_3$) δ 139.2, 133.7, 129.3, 128.0, 73.8, 51.9, 36.0, 31.7, 11.8.
HRMS: m/z (ESI) calcd. for $C_{12}H_{16}O_3SNa$ $[M+Na]^+$ 263.0712, found 263.0723.

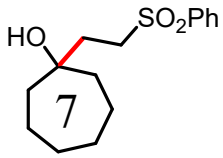


1-(2-(phenylsulfonyl)ethyl)cyclopentan-1-ol (3c)

Product **3c** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as yellow oil (48.8 mg, 96% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.68 - 7.65 (m, 1H), 7.59 - 7.56 (m, 2H), 3.31 - 3.28 (m, 2H), 2.01 - 1.97 (m, 2H), 1.80 - 1.76 (m, 2H), 1.62 (ddd, *J* = 8.6, 7.1, 3.2 Hz, 4H), 1.61 - 1.52 (m, 2H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.2, 133.7, 129.3, 128.0, 80.9, 52.8, 39.8, 33.8, 23.6.

HRMS: m/z (ESI) calcd. for $C_{13}H_{18}O_3SNa$ $[M+Na]^+$ 263.0712, found 263.0717.

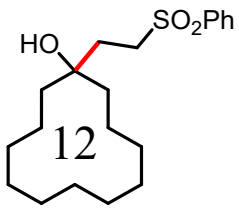


1-(2-(phenylsulfonyl)ethyl)cycloheptan-1-ol (3d)

Product **3d** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as white oil (50.8 mg, 90% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 - 7.91 (m, 2H), 7.66 (ddd, *J* = 6.8, 2.3, 1.1 Hz, 1H), 7.57 (dd, *J* = 10.6, 4.7 Hz, 2H), 3.26 - 3.23 (m, 2H), 1.90 - 1.86 (m, 2H), 1.62 - 1.57 (m, 7H), 1.49 (dd, *J* = 7.3, 3.3 Hz, 2H), 1.35 (s, 3H).**^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.3, 133.6, 129.3, 128.0, 74.4, 51.7, 41.2, 34.9, 29.5, 22.2.

HRMS: m/z (ESI) calcd. for $C_{15}H_{22}O_3SNa$ $[M+Na]^+$ 305.1182, found 305.1178.

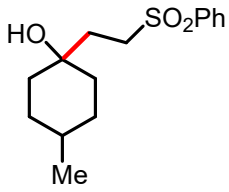


1-(2-(phenylsulfonyl)ethyl)cyclododecan-1-ol (3e)

Product **3e** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as white solid (57.0 mg, 81% yield), melting point: 60 - 62 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 - 7.84 (m, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.50 (t, *J* = 7.7 Hz, 2H), 3.19 - 3.16 (m, 2H), 1.76 - 1.72 (m, 2H), 1.45 - 1.40 (m, 2H), 1.25 - 1.18(m, 20H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.3, 133.6, 129.3, 128.0, 74.1, 51.6, 34.3, 32.8, 26.2, 25.9, 22.4, 21.9, 19.3.

HRMS: m/z (ESI) calcd. for $C_{20}H_{32}O_3SNa$ $[M+Na]^+$ 375.1964, found 397.1965.

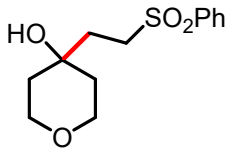


4-methyl-1-(2-(phenylsulfonyl)ethyl)cyclohexan-1-ol (3f)

Product **3f** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as white solid (44.6 mg, 79% yield, d.r. > 20:1), melting point: 70 - 72 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 - 7.92 (m, 2H), 7.68 - 7.64 (m, 1H), 7.59 - 7.56 (m, 2H), 3.22 - 3.19 (m, 2H), 1.96 - 1.93 (m, 2H), 1.66 - 1.61 (m, 5H), 1.42 - 1.39 (m, 4H), 0.88 (d, *J* = 6.6 Hz, 3H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.3, 133.7, 129.3, 128.0, 71.0, 51.4, 37.6, 31.3 (d, *J* = 5.4 Hz), 29.8, 21.0.

HRMS: m/z (ESI) calcd. for $C_{15}H_{22}O_3SNa$ $[M+Na]^+$ 305.1182, found 305.1189.



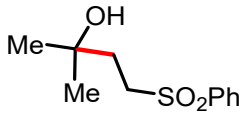
4-(2-(phenylsulfonyl)ethyl)tetrahydro-2H-pyran-4-ol (3g)

Product **3g** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as white solid (45.4 mg, 84% yield), melting point: 78 - 80 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (d, *J* = 7.5 Hz, 2H), 7.67 (d, *J* = 7.4 Hz, 1H), 7.59 (t, *J* = 7.7 Hz, 2H), 3.71 (dd, *J* = 8.0, 2.5 Hz, 4H), 3.28 -

3.25 (m, 2H), 1.93 - 1.89 (m, 2H), 1.64 - 1.60 (m, 2H), 1.47 (d, J = 12.0 Hz, 2H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.0, 133.9, 129.4, 128.0, 67.8, 63.5, 50.9, 37.5, 35.2.

HRMS: m/z (ESI) calcd. for $C_{13}H_{18}O_4SNa$ $[M+Na]^+$ 293.0818, found 293.0828.

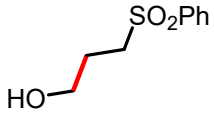


2-methyl-4-(phenylsulfonyl)butan-2-ol (3h)

Product **3h** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as colorless oil (42.0 mg, 92% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (dd, J = 7.8, 1.6 Hz, 2H), 7.68 - 7.65 (m, 1H), 7.58 (t, J = 7.6 Hz, 2H), 3.27 - 3.23 (m, 2H), 1.90 - 1.87 (m, 2H), 1.22 (s, 6H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.2, 133.7, 129.3, 128.0, 69.6, 52.2, 35.5, 29.4.

HRMS: m/z (ESI) calcd. for $C_{11}H_{16}O_3SNa$ $[M+Na]^+$ 251.0712, found 251.0717.

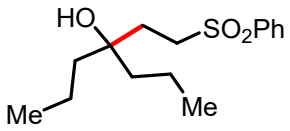


3-(phenylsulfonyl)propan-1-ol (3i)

Product **3i** was prepared according to the general procedure (MeOH (1.2 mmol)) and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 1/1) as light yellow oil (31.2 mg, 78% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 - 7.92 (m, 2H), 7.67 (t, J = 7.4 Hz, 1H), 7.58 (t, J = 7.7 Hz, 2H), 3.75 (t, J = 5.9 Hz, 2H), 3.27 - 3.24 (m, 2H), 2.01 - 1.98 (m, 2H), 1.78 (br s, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_{3)}$ δ 139.03, 133.81, 129.37, 128.05, 60.56, 53.34, 25.73.

HRMS: m/z (ESI) calcd. for $C_9H_{12}O_3SNa$ $[M+Na]^+$ 223.0399, found 223.0398.



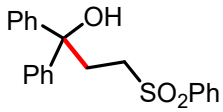
5-(2-(phenylsulfonyl)ethyl)heptan-4-ol (3j)

Product **3j** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as yellow oil (49.4 mg, 87% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 - 7.91 (m, 2H), 7.68 - 7.64 (m, 1H), 7.58 (t, *J* = 7.7 Hz, 2H), 3.19 (d, *J* = 16.6 Hz, 2H), 1.83 (d, *J* = 16.6 Hz, 2H), 1.39 - 1.35 (m, 4H), 1.26 - 1.21 (m, 4H), 0.89 (t, *J* = 7.2 Hz, 6H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ 139.2, 133.7, 129.3, 128.0, 73.3, 51.7, 41.4, 31.6, 16.7, 14.5.

HRMS: m/z (ESI) calcd. for $C_{15}H_{24}O_3SNa$ $[M+Na]^+$ 307.1338, found 307.1335.

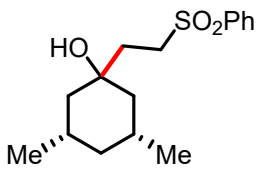


1,1-diphenyl-3-(phenylsulfonyl)propan-1-ol (3k)

Product **3k** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as white solid (50.0 mg, 71% yield), melting point: 86 - 88 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 - 7.84 (m, 2H), 7.64 (t, *J* = 7.5 Hz, 1H), 7.54 (t, *J* = 7.7 Hz, 2H), 7.33 - 7.29 (m, 8H), 7.29 - 7.23 (m, 2H), 3.12 - 3.09 (m, 2H), 2.73 - 2.70 (m, 2H), 2.34 (s, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 145.4, 139.2, 133.9, 129.3, 128.5, 128.0, 127.5, 125.8, 52.0, 34.4, 29.7.

HRMS: m/z (ESI) calcd. for $C_{21}H_{20}O_3SNa$ $[M+Na]^+$ 375.1025, found 375.1029.



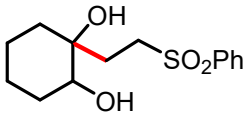
(3R,5S)-3,5-dimethyl-1-(2-(phenylsulfonyl)ethyl)cyclohexan-1-ol (3l)

Product **3l** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 2/1) as a light yellow solid (51.5 mg, 87% yield, d.r. = 1:1). melting point: 76 - 78 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.68 - 7.64 (m, 1H), 7.57 (dd, *J* = 8.4, 7.1 Hz, 2H), 3.29 - 3.25 (m, 2H), 1.84 - 1.81 (m, 2H), 1.72 - 1.68 (m, 3H), 1.56 (d, *J* = 2.2 Hz, 2H), 1.29 (d, *J* = 35.9 Hz, 2H), 0.86 (d, *J* = 6.4 Hz, 6H), 0.84 - 0.82 (m, 1H), 0.49 (q, *J* = 11.8 Hz, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.3, 133.7, 129.3, 128.0, 71.3, 51.4, 45.3, 43.3, 36.1, 27.7, 22.2.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 - 7.92 (m, 2H), 7.68 - 7.65 (m, 1H), 7.58 (dd, *J* = 8.4, 7.1 Hz, 2H), 3.23 - 2.19 (m, 2H), 1.95 - 1.92 (m, 2H), 1.65 - 1.61 (m, 1H), 1.42 (ddt, *J* = 9.1, 6.2, 3.0 Hz, 2H), 1.26 (d, *J* = 2.6 Hz, 2H), 0.94 (t, *J* = 12.7 Hz, 1H), 0.87 (d, *J* = 6.5 Hz, 6H), 0.49 (dt, *J* = 13.1, 11.8 Hz, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.2, 133.7, 129.3, 128.0, 71.9, 51.5, 47.0, 43.3, 30.1, 29.5, 22.3.

HRMS: m/z (ESI) calcd. for $C_{14}H_{24}O_3SNa$ $[M+Na]^+$ 319.1338, found 319.1342.



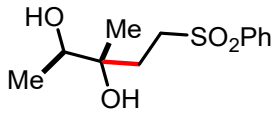
1-(2-(phenylsulfonyl)ethyl)cyclohexane-1,2-diol (3m)

Product **3m** was prepared according to the general procedure (From commercially available materials 1,2-cyclohexanediol (*cis*-and *trans*-mixture) and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as yellow solid (42.6 mg, 75% yield, d.r. > 20:1), melting point: 93-95 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (d, *J* = 7.5 Hz, 2H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 2H), 3.42 – 3.38 (m, 1H), 3.37 - 3.32 (m, 1H), 3.20 (ddd, *J* = 13.9, 11.9, 5.0 Hz, 1H), 2.36 (s, 1H), 2.27 - 2.17 (m, 2H), 1.82 - 1.75 (m, 2H), 1.71-1.69 (m, 3H), 1.59 - 1.56 (m, 1H), 1.49 - 1.17 (m, 2H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.2, 133.7, 129.3, 128.0, 73.9, 72.0, 51.6, 34.0, 31.5, 30.4, 23.2, 20.9.

HRMS: m/z (ESI) calcd. for $C_{14}H_{20}O_4SNa$ $[M+Na]^+$ 307.0975, found

307.0979.

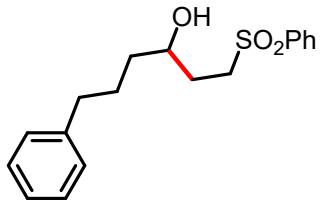


3-methyl-5-(phenylsulfonyl)pentane-2,3-diol (3n)

Product **3n** was prepared according to the general procedure (From commercially available materials 2,3-butanediol (d.r. = 2.5:1) and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as colorless oil (43.9 mg, 85% yield), (d.r. = 1.4:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 - 7.91 (m, 2H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.58 (t, *J* = 7.7 Hz, 2H), 3.65 – 3.58 (m, 1H), 3.42 - 3.37 (m, 2H), 3.21 (ddd, *J* = 13.9, 11.9, 5.0 Hz, 1H), 2.36 (s, 1H), 2.30 - 2.17 (m, 1H), 1.82 - 1.75 (m, 1H), 1.13 (dd, *J* = 6.5, 3.0 Hz, 3H), 1.08 (d, *J* = 18.3 Hz, 3H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.2, 139.1, 133.8, 133.8, 129.4, 129.3, 128.0, 74.3, 73.5, 73.5, 72.8, 51.8, 51.7, 31.1, 29.7, 27.8, 23.4, 20.6, 17.6.

HRMS: m/z (ESI) calcd. for $C_{12}H_{18}O_4SNa$ $[M+Na]^+$ 281.0818, found 281.0827.



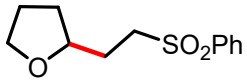
6-phenyl-1-(phenylsulfonyl)hexan-3-ol (3o)

Product **3o** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as colorless oil (35.0 mg, 55% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.64 (d, *J* = 7.5 Hz, 1H), 7.56 (dd, *J* = 10.9, 4.5 Hz, 2H), 7.28 - 7.25 (m, 2H), 7.19 - 7.13 (m, 3H), 3.72 - 3.69 (m, 1H), 3.23 (dddd, *J* = 29.9, 14.1, 10.0, 5.5 Hz, 2H), 2.60 (t, *J* = 7.4 Hz, 2H), 1.95 - 1.92 (m, 1H), 1.76 - 1.71 (m, 2H), 1.63 - 1.61 (m, 1H), 1.46 (dt, *J* = 8.2, 7.1 Hz, 2H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 141.9, 139.2, 133.7, 129.3, 128.4, 128.0, 125.9,

69.83, 53.0, 37.0, 35.6, 30.0, 27.2.

HRMS: m/z (ESI) calcd. for $C_{18}H_{22}O_3SNa$ $[M+Na]^+$ 341.1182, found 341.1191.

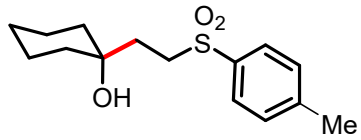


2-(2-(phenylsulfonyl)ethyl)tetrahydrofuran (3p)

Product **3p** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 1/1) as colorless oil (40.8 mg, 85% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.68 - 7.64 (m, 1H), 7.57 (dd, *J* = 8.4, 7.1 Hz, 2H), 3.87 - 3.82 (m, 1H), 3.79 (dt, *J* = 8.4, 6.8 Hz, 1H), 3.68 (dt, *J* = 8.3, 6.9 Hz, 1H), 3.30 (ddd, *J* = 13.9, 11.5, 4.8 Hz, 1H), 3.14 (ddd, *J* = 14.0, 11.4, 4.8 Hz, 1H), 2.00 - 1.93 (m, 2H), 1.89 - 1.83 (m, 3H), 1.50 - 1.47 (m, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 139.2, 133.7, 129.3, 128.0, 77.0, 67.9, 53.6, 31.2, 28.5, 25.6.

HRMS: m/z (ESI) calcd. for $C_{12}H_{16}O_3SNa$ $[M+Na]^+$: 263.0718, found: 263.0724.

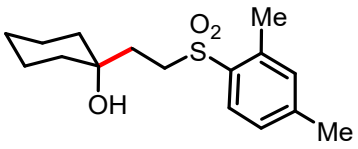


1-(2-tosylethyl)cyclohexan-1-ol (3q)

Product **3q** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as yellow oil (46.1 mg, 86% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 3.24 - 3.21 (m, 2H), 2.44 (s, 3H), 1.85 - 1.82 (m, 2H), 1.53 - 1.44 (m, 7H), 1.36 (dd, *J* = 13.4, 3.5 Hz, 2H), 1.24 (dd, *J* = 8.8, 5.2 Hz, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 144.6, 136.2, 129.9, 127.9, 70.3, 51.4, 37.4, 34.3, 25.5, 22.0, 21.6.

HRMS: m/z (ESI) calcd. for $C_{15}H_{22}O_3SNa$ $[M+Na]^+$ 305.1182, found 305.1190.

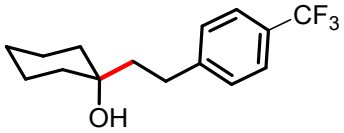


1-(2-((2,4-dimethylphenyl)sulfonyl)ethyl)cyclohexan-1-ol (3r)

Product **3r** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as yellow oil (49.7 mg, 84% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.0 Hz, 1H), 7.17 - 7.13 (m, 2H), 3.27 - 3.24 (m, 2H), 2.65 (s, 3H), 2.39 (s, 3H), 1.84 - 1.81K (m, 2H), 1.52 - 1.48 (m, 8H), 1.39 - 1.36 (m, 2H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 144.4, 137.7, 134.4, 133.5, 130.3, 127.2, 70.3, 50.4, 37.5, 34.1, 25.5, 22.0, 21.7, 20.4.

HRMS: m/z (ESI) calcd. for $C_{16}H_{24}O_3SNa$ $[M+Na]^+$ 319.1338, found 319.1336.



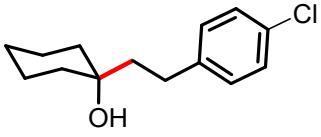
1-(2-((4-(trifluoromethyl)phenyl)sulfonyl)ethyl)cyclohexan-1-ol (3s)

Product **3s** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as white solid (50.4 mg, 75% yield), melting point: 83 - 85 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.2 Hz, 2H), 7.85 (d, *J* = 8.2 Hz, 2H), 3.29 - 3.26 (m, 2H), 1.88 - 1.85 (m, 2H), 1.51 (dd, *J* = 9.8, 6.3 Hz, 7H), 1.38 (dd, *J* = 21.1, 10.9 Hz, 3H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 142.8, 135.3 (q, *J* = 66.3 Hz), 128.7, 126.5 (d, *J* = 3.7 Hz), 123.2, (d, *J* = 273.0 Hz), 70.3, 51.3, 37.5, 34.1, 25.4, 22.0.

^{19}F NMR (471 MHz, $CDCl_3$) δ -63.17.

HRMS: m/z (ESI) calcd. for $C_{15}H_{19}O_3SNa$ $[M+Na]^+$ 359.0899, found 359.0895.

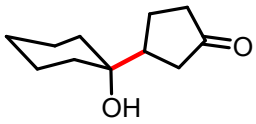


1-(2-((4-chlorophenyl)sulfonyl)ethyl)cyclohexan-1-ol (3t)

Product **3t** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as light yellow oil (43.5 mg, 72% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 - 7.84 (m, 2H), 7.56 - 7.54 (m, 2H), 3.26 - 3.22 (m, 2H), 1.86 - 1.83 (m, 2H), 1.52 - 1.48 (m, 7H), 1.41 - 1.36 (m, 2H), 1.27 - 1.26 (d, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 140.4, 137.7, 129.6, 129.5, 70.3, 51.4, 37.5, 34.2, 25.5, 22.0.

HRMS: m/z (ESI) calcd. for $C_{14}H_{19}ClO_3SNa$ $[M+Na]^+$ 325.0636, found 325.0640.

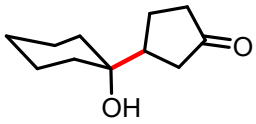


3-(1-hydroxycyclohexyl)cyclopentan-1-one (3u)

Product **3u** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as yellow oil (34.6 mg, 95% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 2.39 - 2.33 (m, 1H), 2.23 (d, J = 3.0 Hz, 3H), 2.18 - 2.12 (m, 1H), 2.05 - 2.01 (m, 1H), 1.81 (tdd, J = 12.0, 8.7, 3.9 Hz, 1H), 1.63 (dd, J = 27.0, 12.1 Hz, 2H), 1.57 - 1.53 (m, 5H), 1.46 - 1.36 (m, 3H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 219.6, 71.2, 46.7, 39.1, 38.8, 36.7, 35.6, 25.6, 22.7, 21.9, 21.8.

HRMS: m/z (ESI) calcd. for $C_{11}H_{18}O_2Na$ $[M+Na]^+$ 205.1199, found 205.1197.

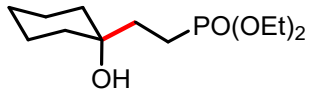


4-(1-hydroxycyclohexyl)dihydrofuran-2(*3H*)-one (3v)

Product **3v** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as colorless oil (33.1 mg, 90% yield).

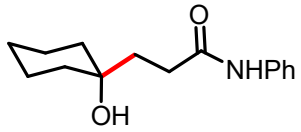
^{1}H NMR (500 MHz, $CDCl_3$) δ 4.32 (dd, J = 10.1, 8.2 Hz, 2H), 2.64 - 2.59 (m, 2H), 2.47 - 2.42 (m, 1H), 1.57 (ddd, J = 15.0, 13.7, 6.3 Hz, 8H), 1.38

(dt, J = 13.3, 8.0 Hz, 2H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 177.36, 70.3, 68.6, 36.1, 35.4, 28.8, 25.3, 21.6.
HRMS: m/z (ESI) calcd. for $C_{10}H_{16}O_3Na$ $[M+Na]^+$ 207.0992, found 207.0988.



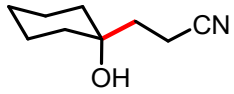
diethyl (2-(1-hydroxycyclohexyl)ethyl)phosphonate (3w)

Product **3w** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as colorless oil (44.9 mg, 85% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 4.10 (d, J = 7.6 Hz, 4H), 1.93 (d, J = 11.0 Hz, 2H), 1.88 - 1.81 (m, 2H), 1.76 - 1.70 (m, 2H), 1.57 (ddd, J = 12.6, 12.0, 3.1 Hz, 5H), 1.51 - 1.46 (m, 2H), 1.39 (dd, J = 13.0, 3.1 Hz, 2H), 1.33 (t, J = 7.1 Hz, 6H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 70.34, 61.6, 37.2, 25.8, 22.1, 19.9, 18.8, 16.5.
HRMS: m/z (ESI) calcd. for $C_{12}H_{25}O_4PNa$ $[M+Na]^+$ 287.1383, found 287.1387.



3-(1-hydroxycyclohexyl)-*N*-phenylpropanamide (3x)

Product **3x** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as white solid (40.0 mg, 81% yield), melting point: 51-53 °C.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (br, 1H), 7.50 (d, J = 7.9 Hz, 2H), 7.30 (t, J = 7.9 Hz, 2H), 7.09 (t, J = 7.4 Hz, 1H), 2.51 (t, J = 7.3 Hz, 2H), 2.10 (s, 1H), 1.89 (t, J = 7.3 Hz, 2H), 1.60 - 1.44 (m, 9H), 1.29 (d, J = 9.5 Hz, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 172.3, 138.0, 129.0, 124.2, 119.8, 71.0, 37.7, 36.8, 31.6, 25.7, 22.3.
HRMS: m/z (ESI) calcd. for $C_{15}H_{21}NO_2Na$ $[M+Na]^+$ 270.1465, found 270.1470.

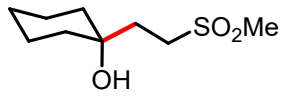


3-(1-hydroxycyclohexyl)propanenitrile (3y)

Product **3y** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as colorless oil (29.1 mg, 95% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 2.46 (dd, *J* = 8.3, 7.4 Hz, 2H), 1.84 - 1.81 (m, 2H), 1.55 (dt, *J* = 10.3, 7.0 Hz, 7H), 1.44 - 1.41 (m, 2H), 1.31 - 1.25 (m, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 120.7, 70.4, 37.5, 37.1, 25.5, 22.0, 11.1.

HRMS: m/z (ESI) calcd. for $C_{15}H_9NONa$ $[M+Na]^+$ 252.1359 found 252.1357.

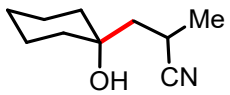


1-(2-(methylsulfonyl)ethyl)cyclohexan-1-ol (3z)

Product **3z** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as light yellow oil (33.8 mg, 82% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.20 - 3.16 (m, 2H), 2.93 (s, 3H), 1.98 - 1.95 (m, 2H), 1.73 (s, 1H), 1.58 - 1.54 (m, 7H), 1.45 (dd, *J* = 13.5, 4.2 Hz, 2H), 1.33 - 1.25 (m, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 70.3, 49.8, 40.6, 37.5, 33.9, 25.5, 22.0.

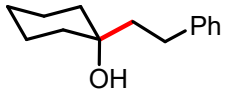
HRMS: m/z (ESI) calcd. for $C_9H_{18}O_3SNa$ $[M+Na]^+$ 229.0869 found 229.0879.



3-(1-hydroxycyclohexyl)-2-methylpropanenitrile (3aa)

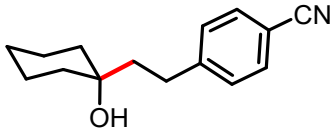
Product **3aa** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as colorless oil (23.7 mg, 71% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 2.89 - 2.85 (m, 1H), 1.96 (dd, *J* = 14.4, 9.8 Hz, 1H), 1.70 - 1.67 (m, 1H), 1.60 - 1.52 (m, 8H), 1.45 (s, 2H), 1.36 (d, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 124.3, 70.8, 46.4, 38.2, 37.1, 25.5, 22.1, 22.0, 20.0, 19.7.
HRMS: m/z (ESI) calcd. for $C_{10}H_{18}NO$ $[M+H]^+$ 168.1383 found 168.1384



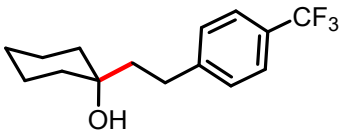
1-phenethylcyclohexan-1-ol (3ab)

Product **3ab** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as white solid (21.2 mg, 52% yield), melting point: 45 - 47 °C.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 - 7.25 (m, 2H), 7.21 - 7.16 (m, 3H), 2.72 - 2.69 (m, 2H), 1.78 - 1.75 (m, 2H), 1.61 - 1.58 (m, 4H), 1.51 (dd, *J* = 12.0, 7.4 Hz, 4H), 1.31 - 1.25 (m, 2H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 142.9, 128.4, 128.3, 125.7, 71.4, 44.4, 37.5, 29.4, 25.8, 22.3.
HRMS: m/z (ESI) calcd. for $C_{14}H_{20}ONa$ $[M+Na]^+$ 227.1406 found 227.1410.



4-(2-(1-hydroxycyclohexyl)ethyl)benzonitrile (3ac)

Product **3ac** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as light yellow oil (39 mg, 85% yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 2.79 - 2.75 (m, 2H), 1.76 - 1.72 (m, 2H), 1.60 (dd, *J* = 9.7, 6.6 Hz, 5H), 1.50 - 1.48 (m, 4H), 1.29 (s, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 148.8, 132.2, 129.2, 119.1, 109.6, 71.2, 37.5, 29.7, 25.7, 22.2.
HRMS: m/z (ESI) calcd. for $C_{15}H_{19}NONa$ $[M+Na]^+$ 252.1359 found 252.1357.



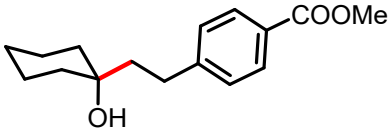
1-(4-(trifluoromethyl)phenethyl)cyclohexan-1-ol (3ad)

Product **3ad** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as white solid (44.6 mg, 82% yield), melting point: 45-47 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 2.79 - 2.75 (m, 2H), 1.76 - 1.72 (m, 2H), 1.61 - 1.58 (m, 5H), 1.50 - 1.48 (m, 4H), 1.31 - 1.25 (m, 1H). **^{13}C{^{1}H} NMR (**125 MHz, $CDCl_3$) δ 147.2, 12jk8.7, 127.3 (q, *J* = 32.3 Hz), 125.3 (q, *J* = 3.8 Hz), 124.4 (d, *J* = 271.7 Hz), 71.3, 44.0, 37.5, 29.3, 25.8, 22.2.

^{19}F NMR (471 MHz, $CDCl_3$) δ -62.25.

HRMS: m/z (ESI) calcd. for $C_{15}H_{19}F_3ONa$ $[M+Na]^+$ 295.1280 found 295.1282.

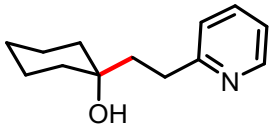


methyl 4-(2-(1-hydroxycyclohexyl)ethyl)benzoate (3ae)

Product **3ae** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as colorless oil (40.9 mg, 78% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 - 7.94 (m, 2H), 7.27 - 7.25 (m, 2H), 3.89 (s, 3H), 2.78 - 2.74 (m, 2H), 1.77 - 1.74 (m, 2H), 1.61 - 1.58 (m, 4H), 1.53 - 1.48 (m, 5H), 1.30 (dt, *J* = 15.6, 8.5 Hz, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 167.2, 148.6, 129.8, 128.4, 127.7, 71.3, 52.0, 43.9, 37.5, 29.5, 25.8, 22.3.

HRMS: m/z (ESI) calcd. for $C_{17}H_{22}O_3Na$ $[M+Na]^+$ 300.1696 found 300.1692.

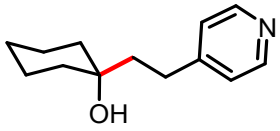


1-(2-(pyridin-2-yl)ethyl)cyclohexan-1-ol (3af)

Product **3af** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as colorless oil (29.5 mg, 72% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.49 - 8.48 (m, 1H), 7.59 (td, *J* = 7.7, 1.8 Hz, 1H), 7.18 (d, *J* = 7.8 Hz, 1H), 7.11 - 7.09 (m, 1H), 2.96 (t, *J* = 7.4 Hz, 2H), 1.92 - 1.89 (m, 2H), 1.67 - 1.62 (m, 4H), 1.50 - 1.44 (m, 4H), 1.30 - 1.25 (m, 2H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 162.4, 148.7, 136.6, 123.1, 121.0, 70.5, 41.0, 38.0, 31.7, 26.0, 22.4.

HRMS: m/z (ESI) calcd. for $C_{13}H_{19}NONa$ $[M+Na]^+$ 228.1359 found 228.1355.

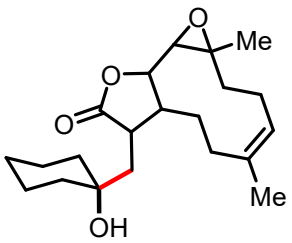


1-(2-(pyridin-4-yl)ethyl)cyclohexan-1-ol (3ag)

Product **3ag** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 3/1) as light yellow solid (27.9 mg, 68% yield), melting point: 52 - 54 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.47 (dd, *J* = 4.6, 1.4 Hz, 2H), 7.13 (d, *J* = 5.9 Hz, 2H), 2.73 - 2.70 (m, 2H), 1.77 - 1.73 (m, 2H), 1.63 - 1.59 (m, 5H), 1.54 - 1.48 (m, 4H), 1.31 - 1.25 (m, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 152.2, 149.6, 123.9, 71.1, 43.1, 37.5, 28.9, 25.8, 22.2.

HRMS: m/z (ESI) calcd. for $C_{13}H_{19}NONa$ $[M+Na]^+$ 228.1359 found 228.1362.



(Z)-8-((1-hydroxycyclohexyl)methyl)-1a,5-dimethyl 2,3,6,7,7a,8,10a,10b-octahydrooxireno[2',3':9,10]cyclodeca[1,2-b]furan-9(1aH)-one (3ai)

Product **3ai** was prepared according to the general procedure and obtained from flash column chromatography (eluent: petroleum ether/ethylacetate = 2/1) as a colourless oil (42.4 mg, 61% yield, d.r. > 20:1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.17 (dd, *J* = 12.9, 3.7 Hz, 1H), 3.92 (t, *J* = 9.1 Hz, 1H), 3.51 (s, 1H), 2.72 (d, *J* = 8.9 Hz, 1H), 2.59 (ddd, *J* = 12.0, 9.5, 2.1 Hz, 1H), 2.44 - 2.29 (m, 2H), 2.20 - 2.12 (m, 2H), 2.05 - 1.88 (m, 4H), 1.71 (s, 3H), 1.68 - 1.62 (m, 5H), 1.55-43 (m, 3H), 1.34-31 (m, 1H), 1.30 (s, 3H), 1.29 - 1.21 (m, 4H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ 179.5, 134.4, 125.2, 83.4, 69.8, 66.2, 61.6, 50.7, 43.5, 41.2, 40.5, 39.7, 36.7, 36.6, 29.7, 29.5, 25.8, 24.1, 22.3, 17.2, 16.9.

HRMS: m/z (ESI) calcd. for $C_{21}H_{32}O_4Na$ $[M+Na]^+$ 371.2193, found 371.2195.

9. References

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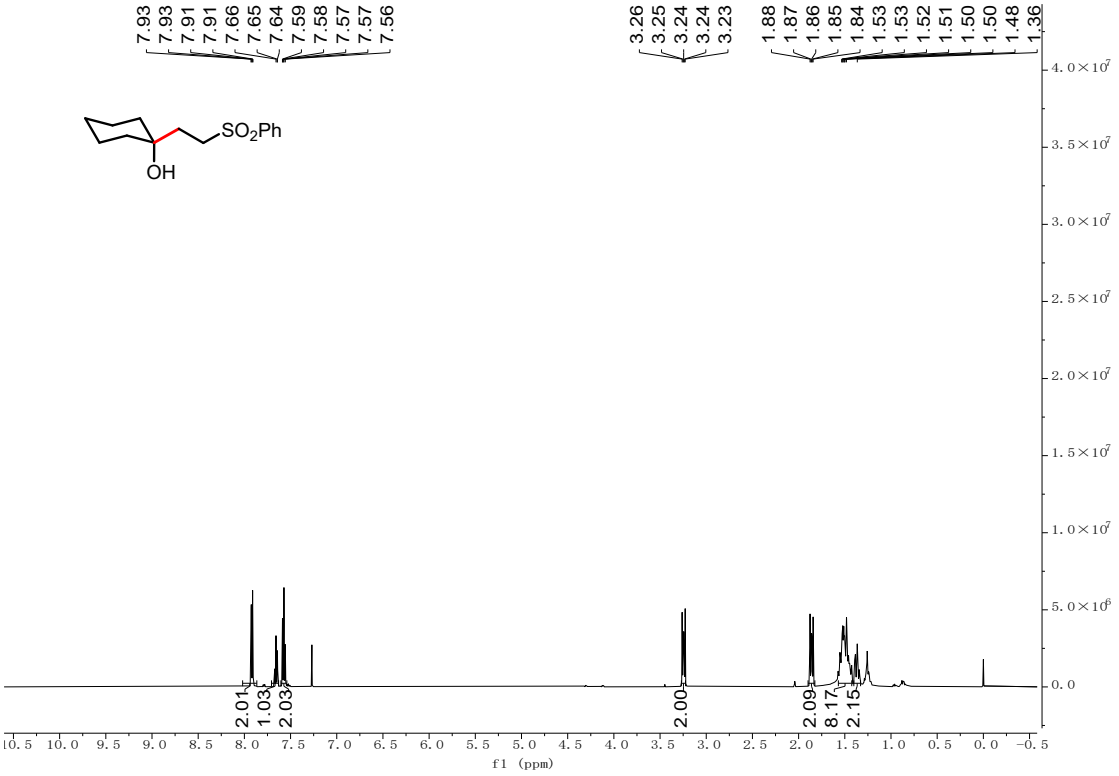
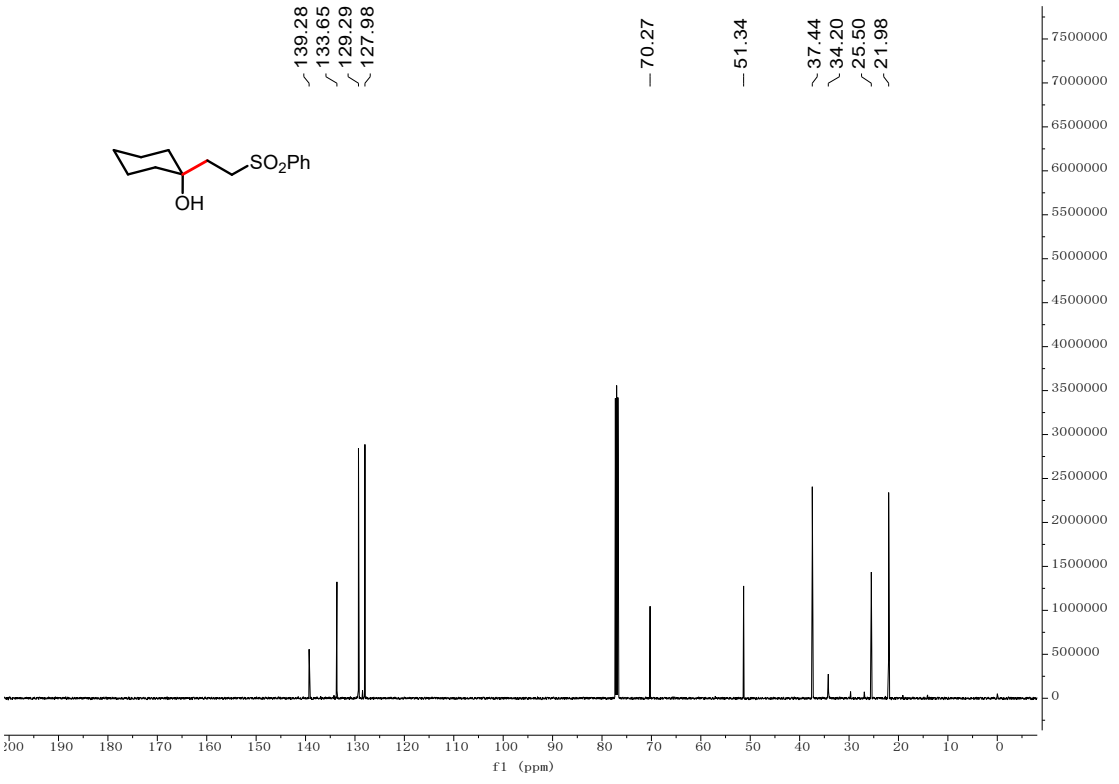
10. NMR spectra

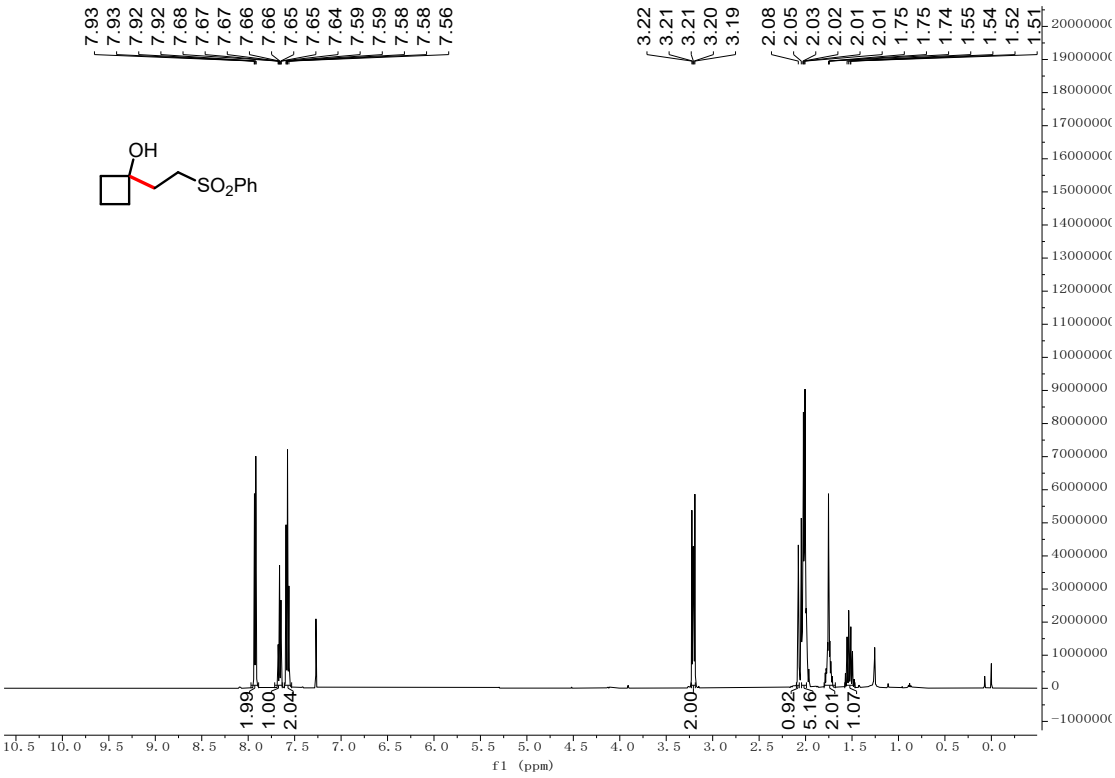
3a (^{1}H NMR, 500 MHz, $CDCl_3$)



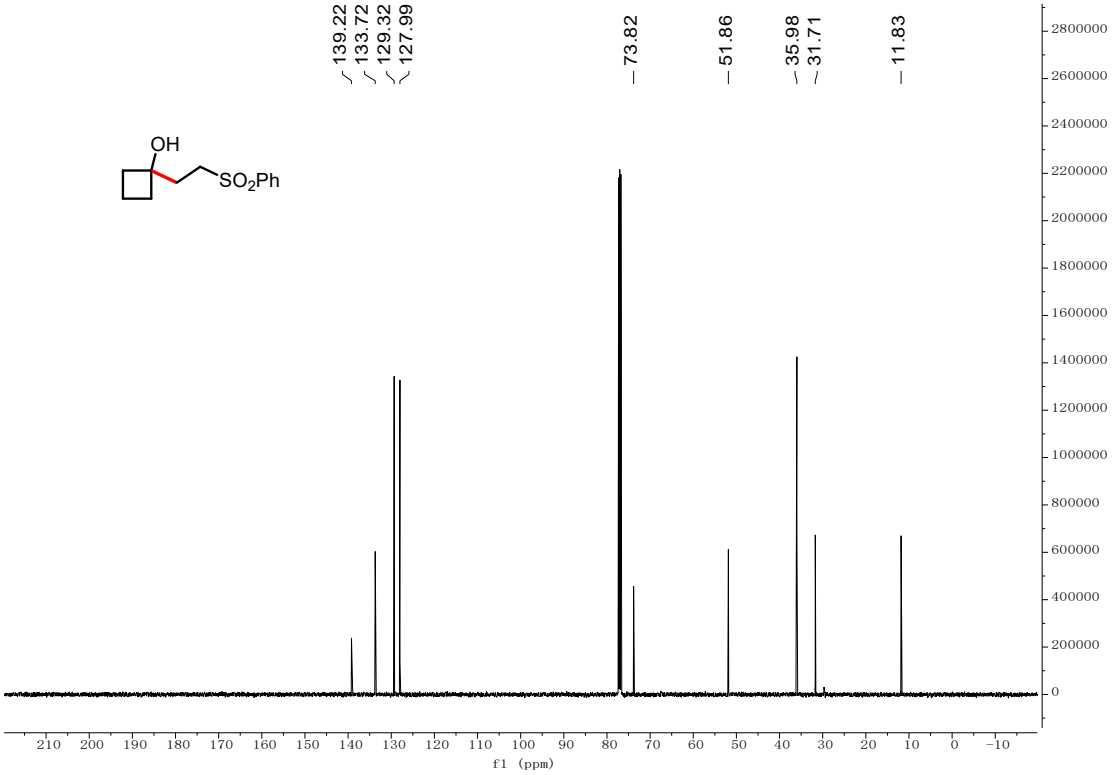
3a (^{13}C{^{1}H} NMR, 125 MHz, $CDCl_3$)



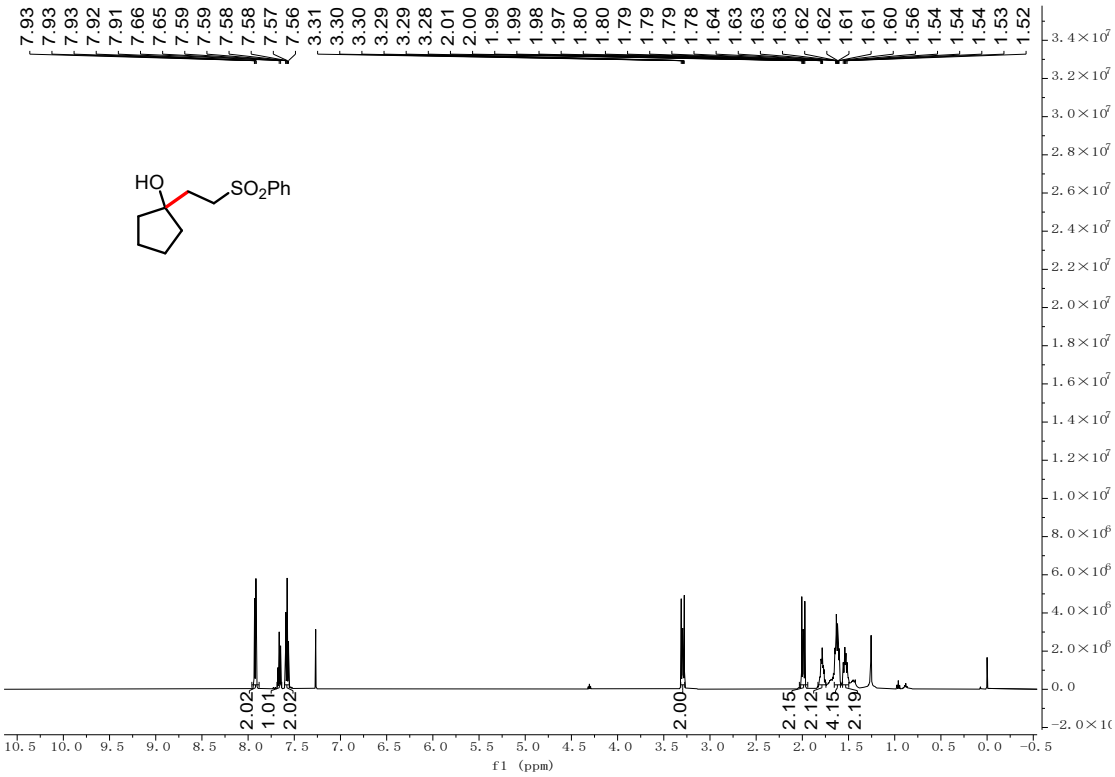
3b (^{1}H NMR, 500 MHz, $CDCl_3$)



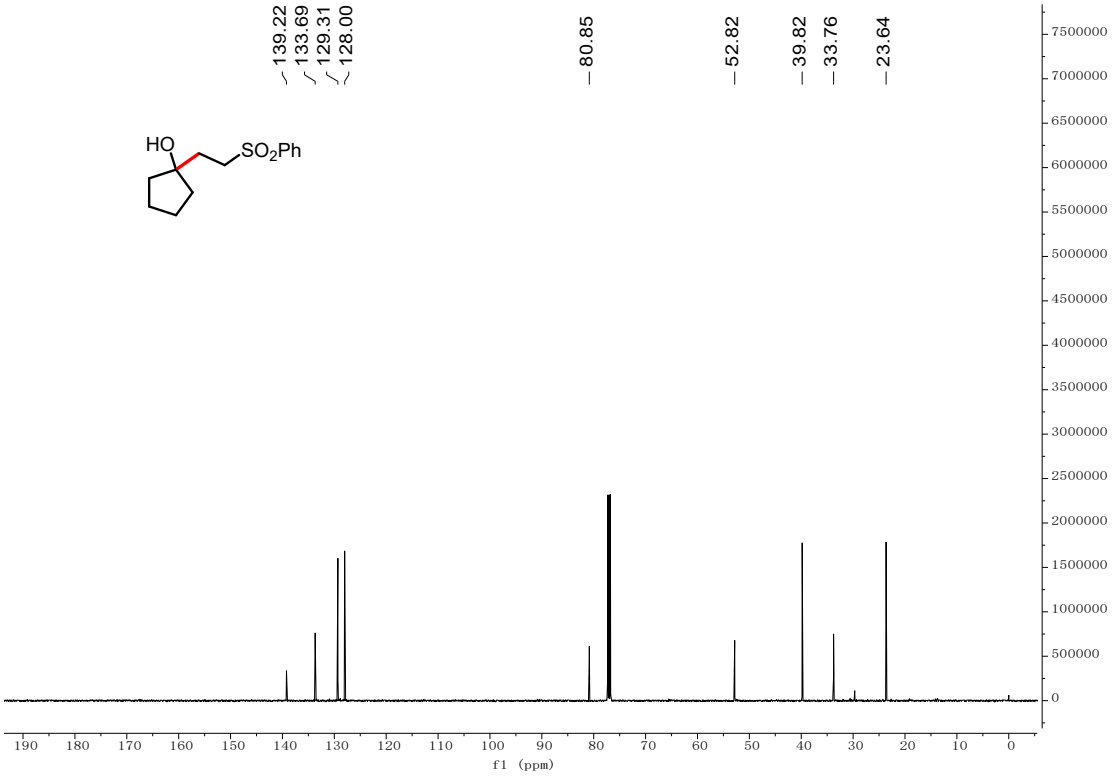
3b ($^{13}C\{^1H\}$ NMR, 125 MHz, $CDCl_3$)



3c (^{1}H NMR, 500 MHz, $CDCl_3$)



3c (^{13}C{^{1}H} NMR, 125 MHz, $CDCl_3$)



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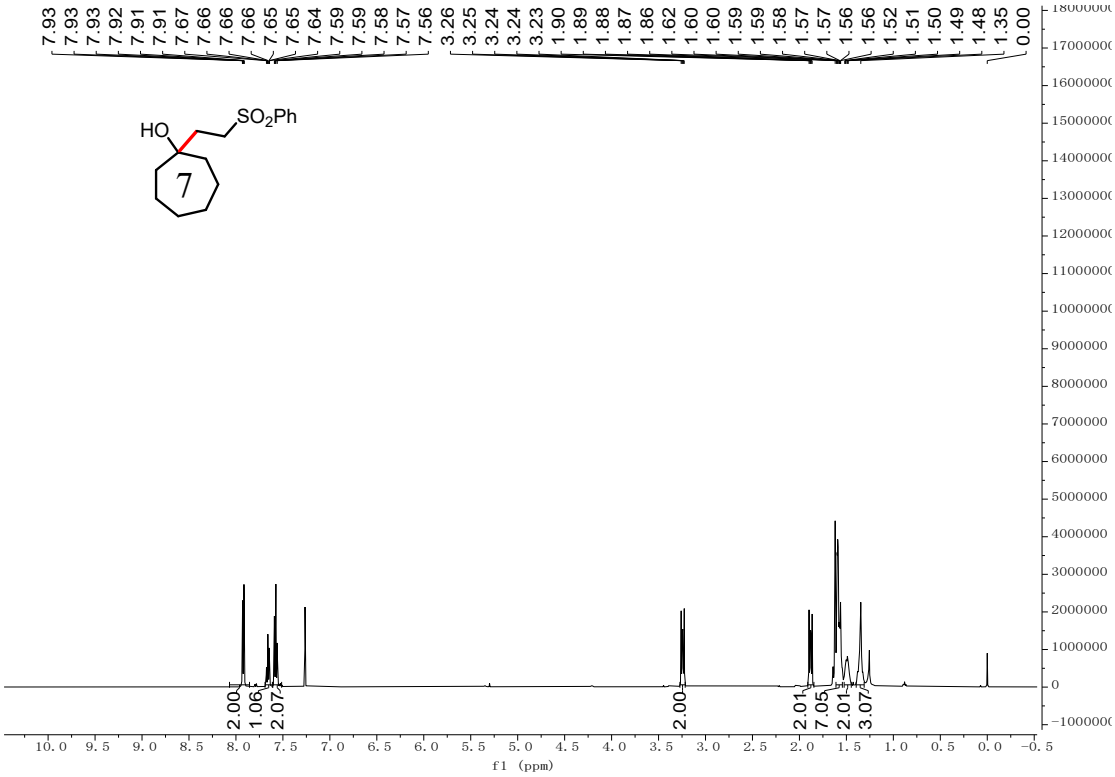
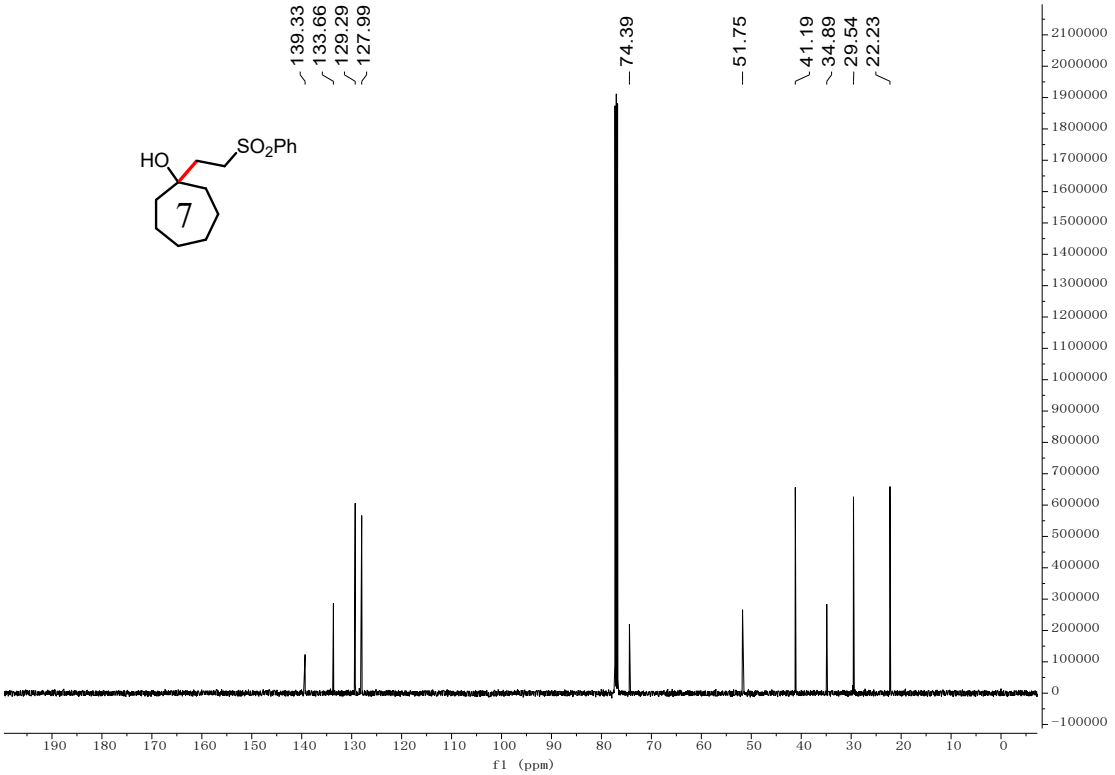
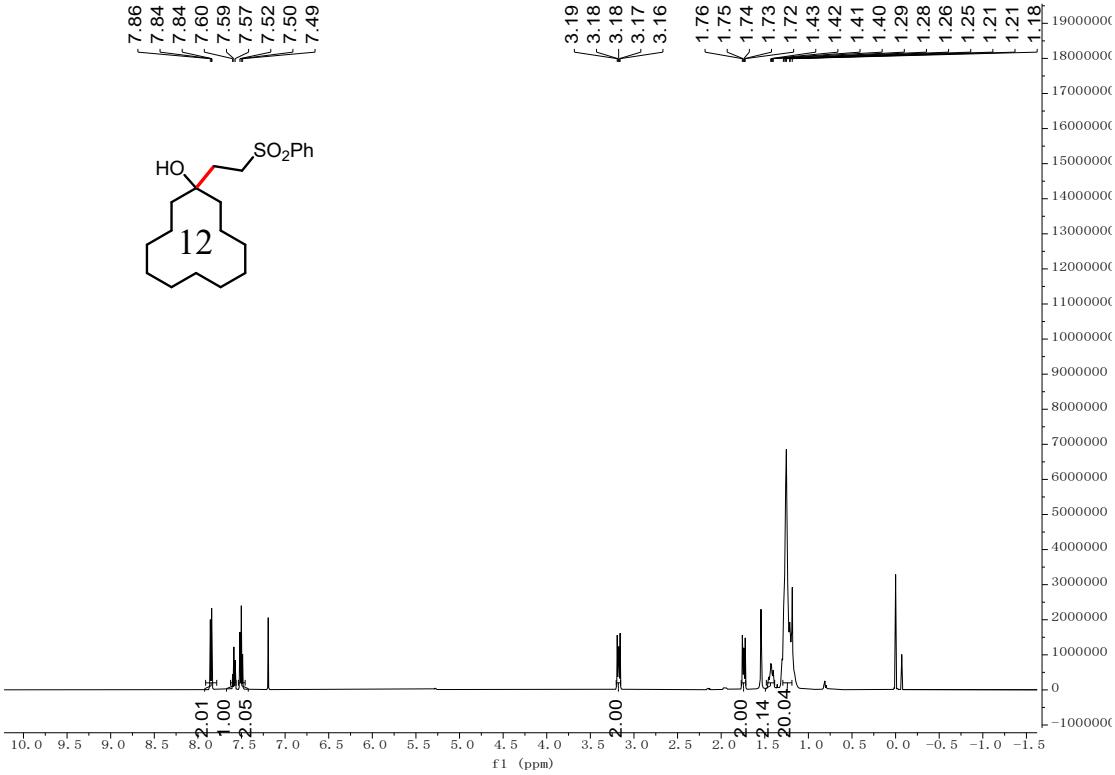


3d (^{13}C{^{1}H} NMR, 125 MHz, $CDCl_3$)



3e (^{1}H NMR, 500 MHz, $CDCl_3$)



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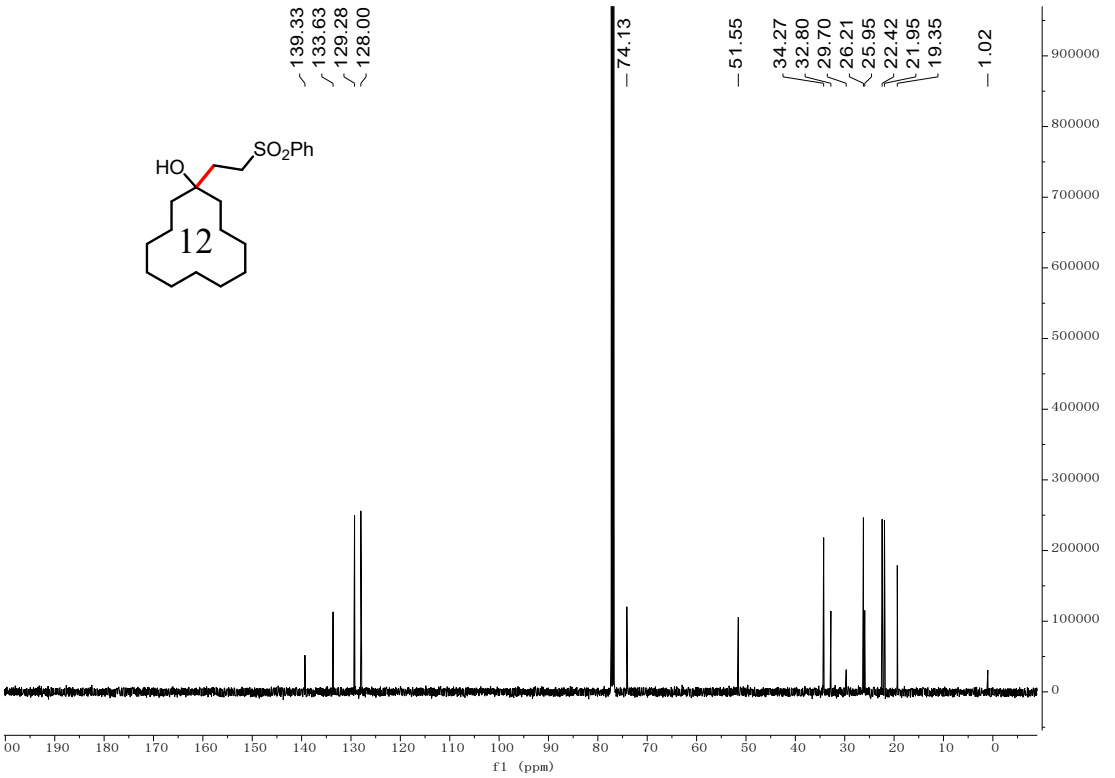
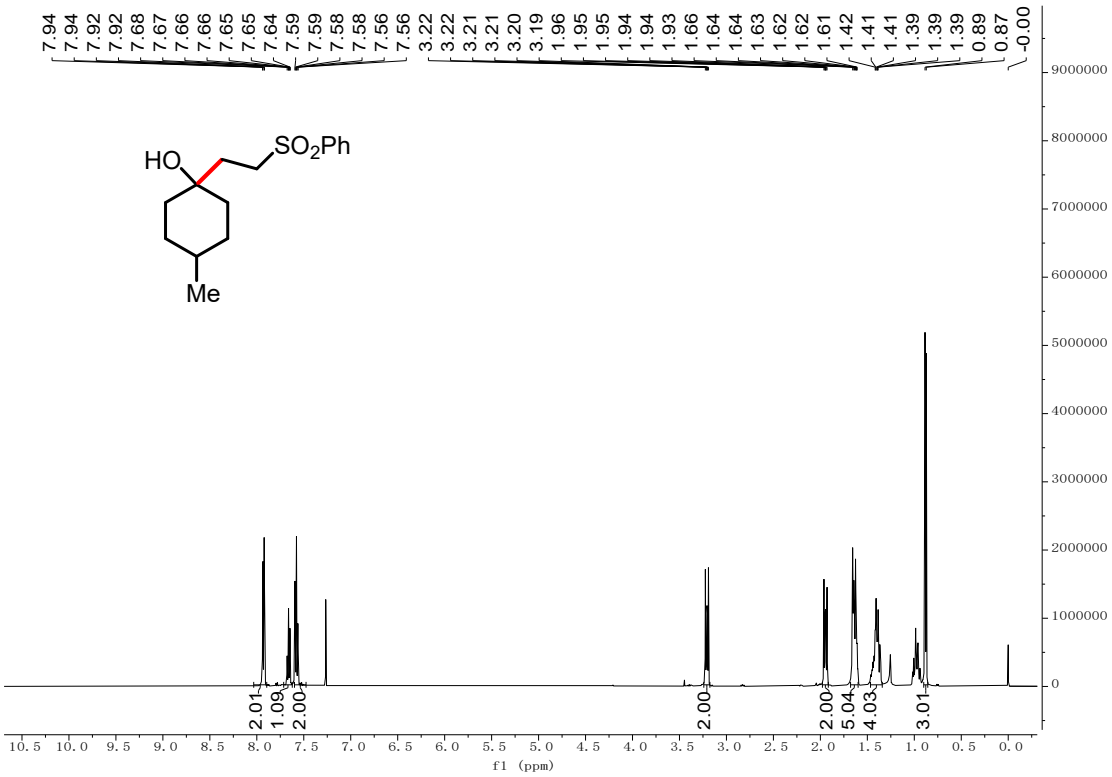
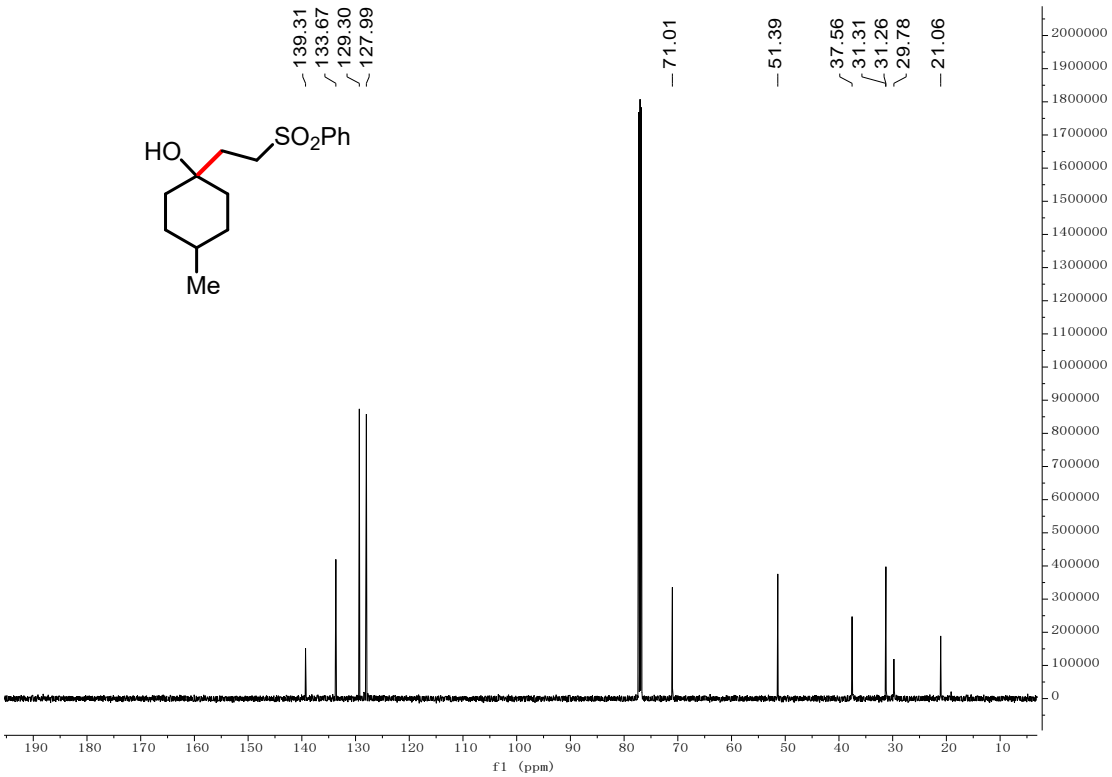


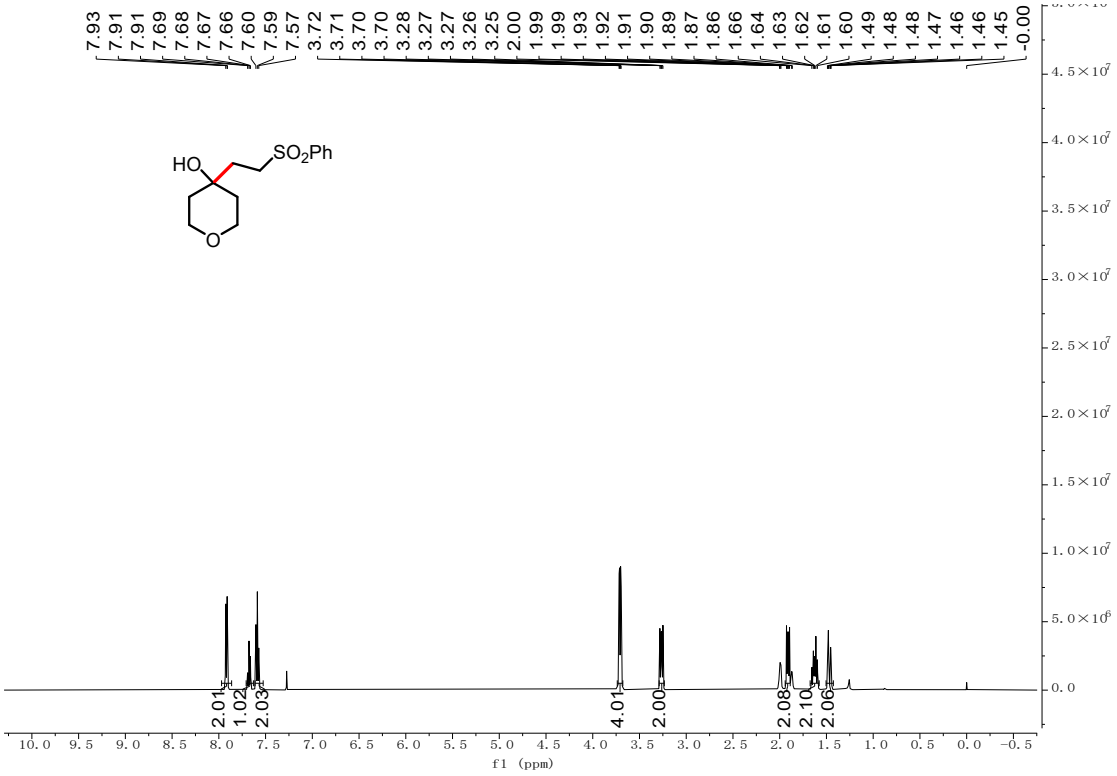
3f (^{1}H NMR, 500 MHz, $CDCl_3$)



3f (^{13}C{^{1}H} NMR, 125 MHz, $CDCl_3$)



3g (^{1}H NMR, 500 MHz, $CDCl_3$)



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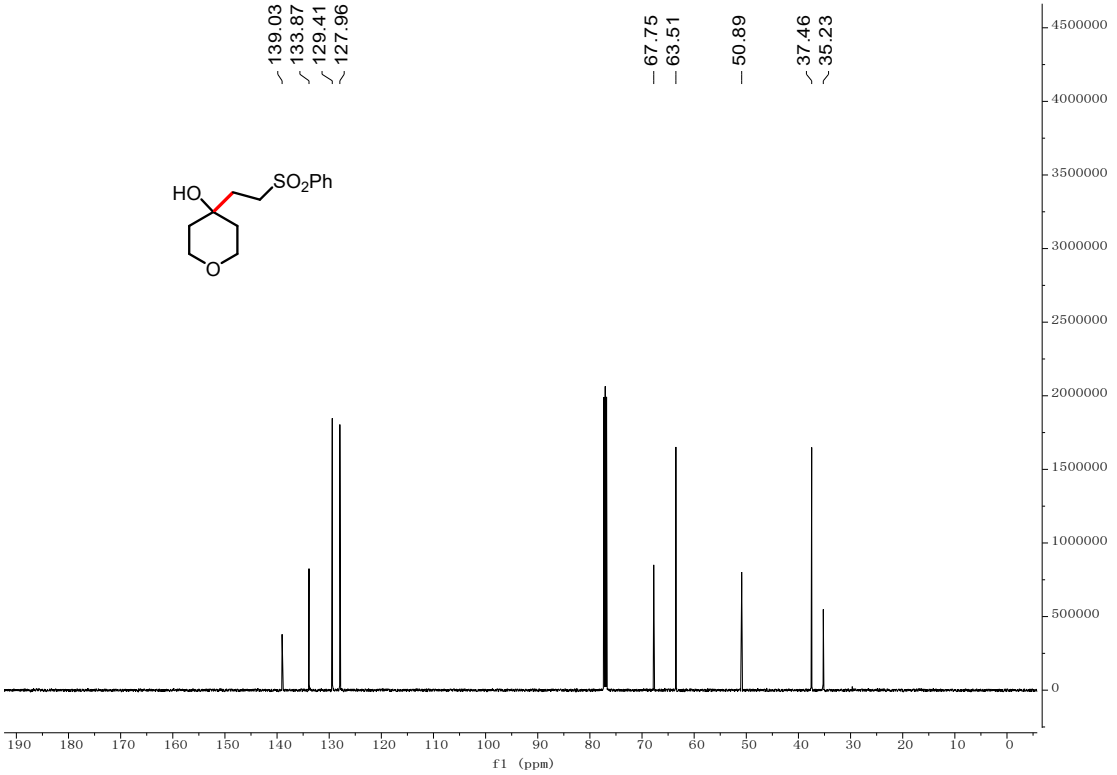
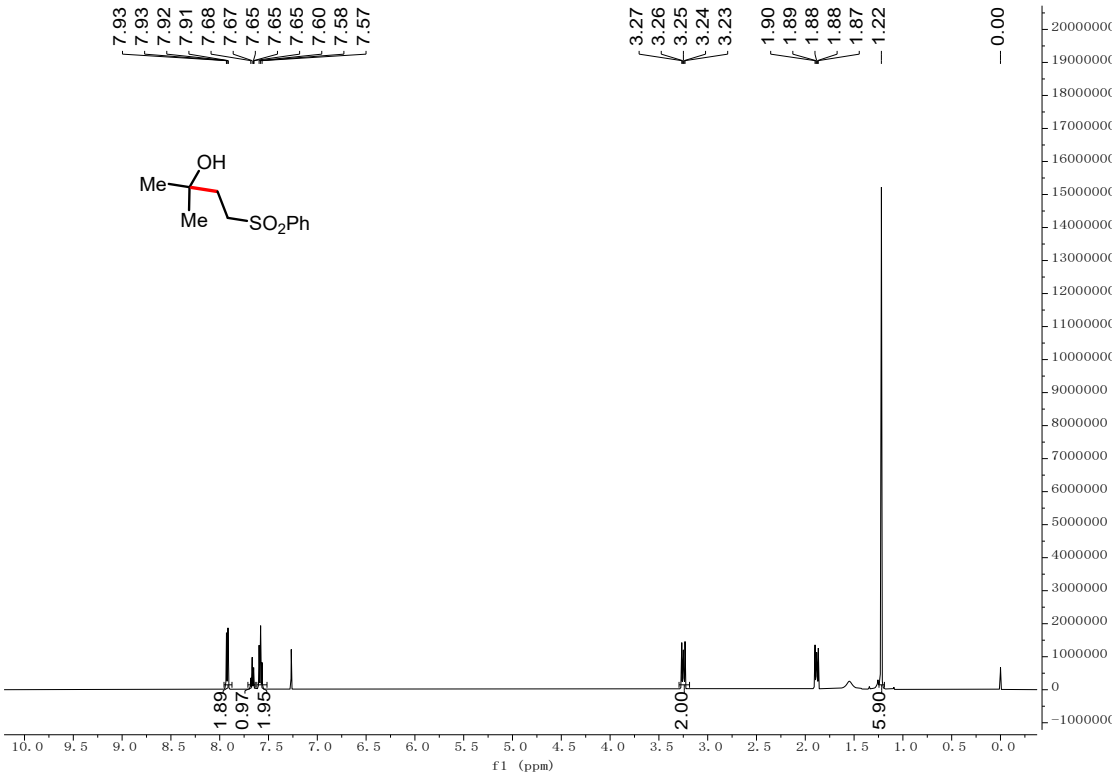


3h (^{1}H NMR, 500 MHz, $CDCl_3$)



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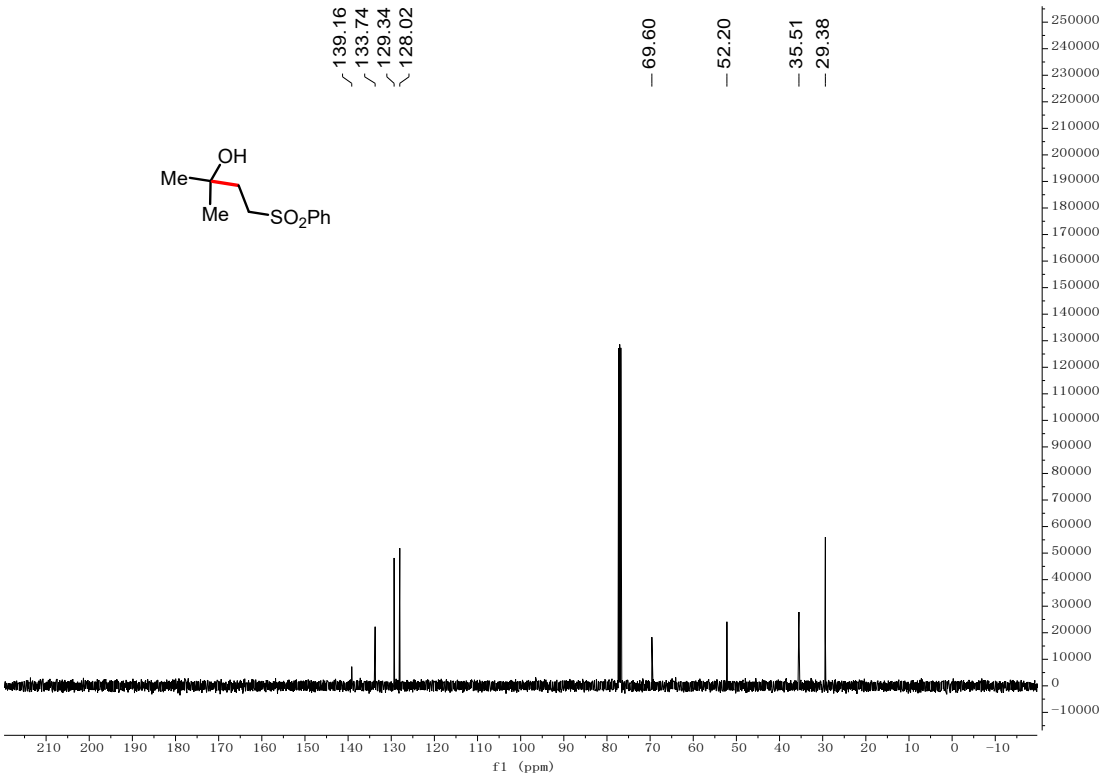

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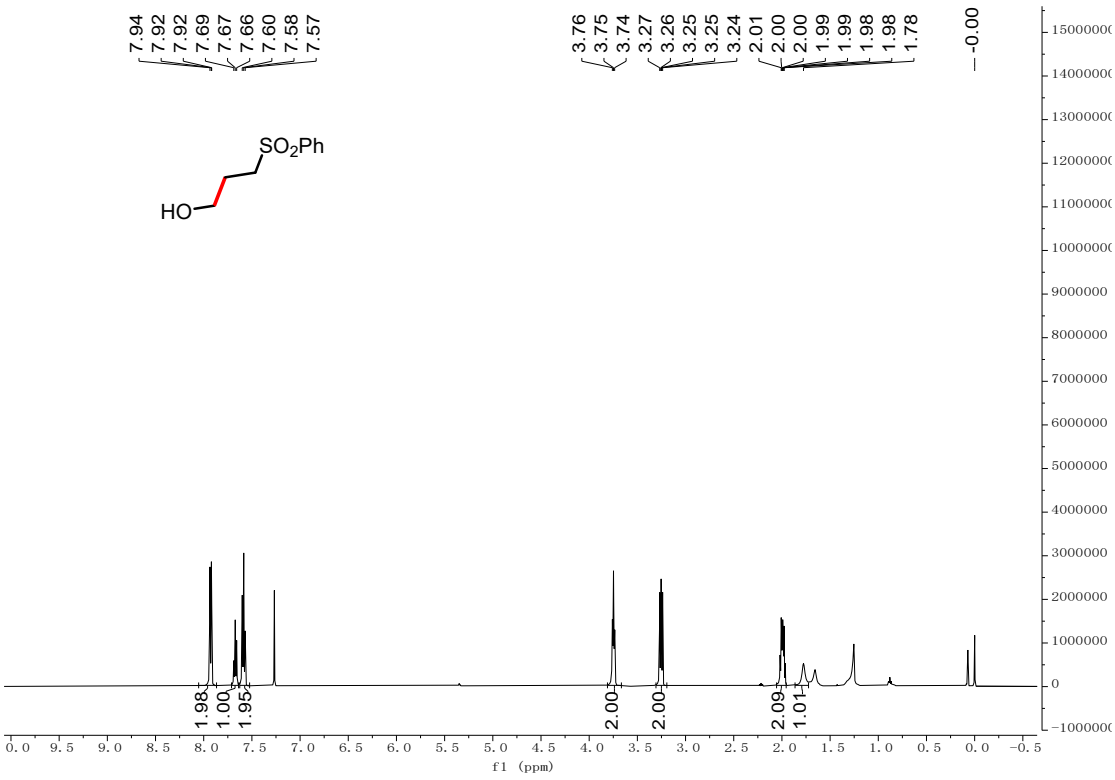

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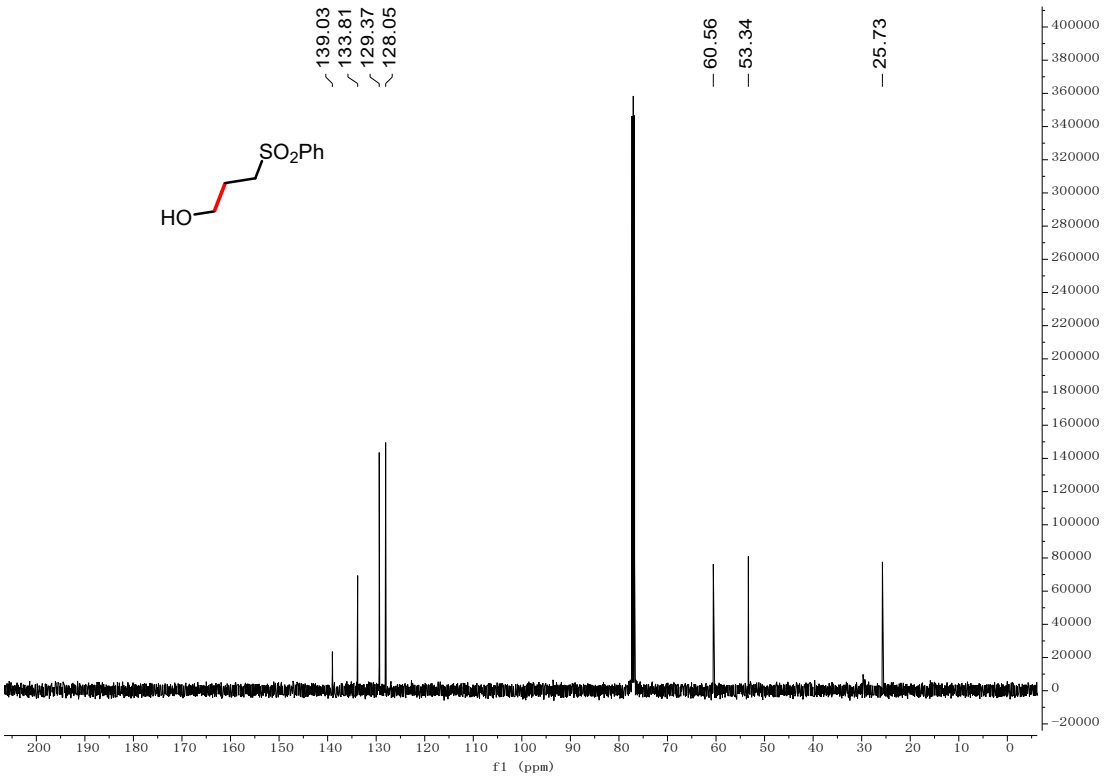
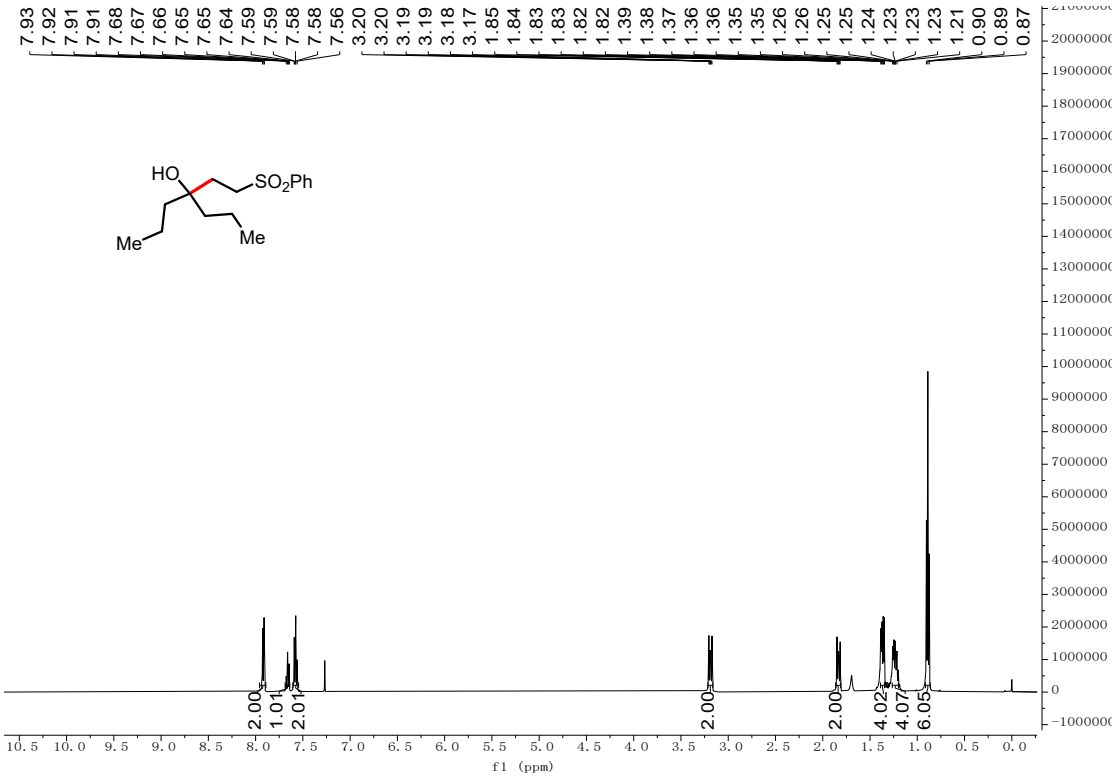


3j (^{1}H NMR, 500 MHz, $CDCl_3$)



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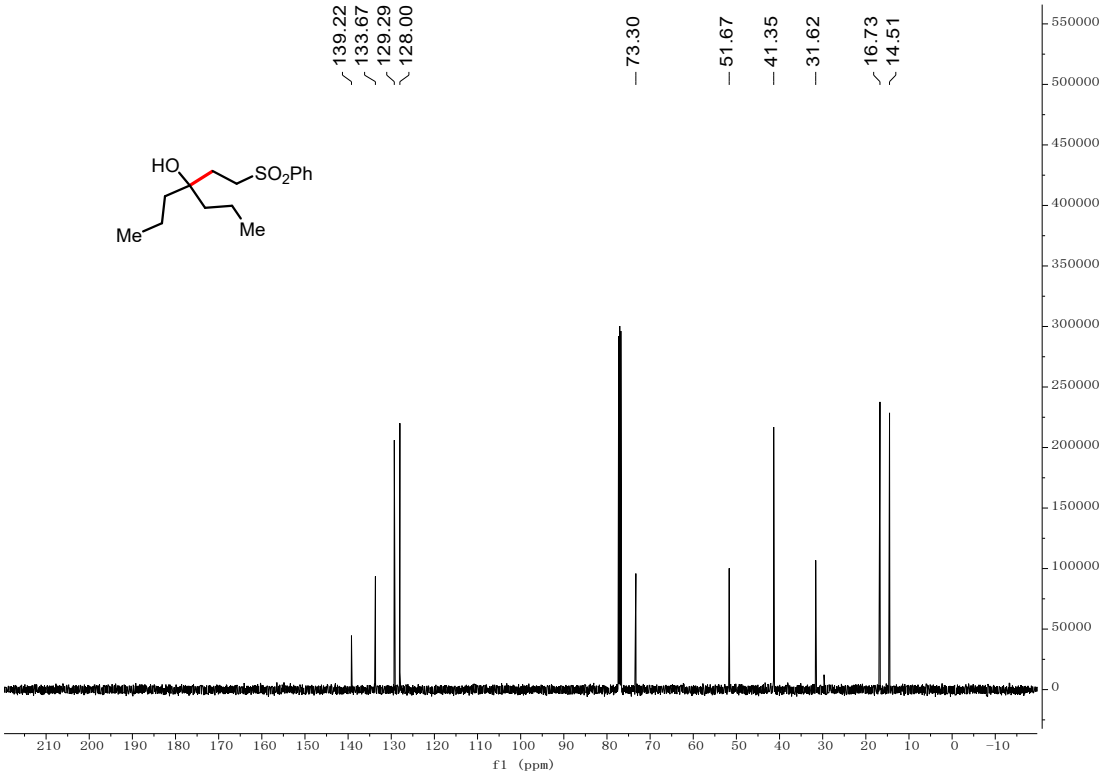

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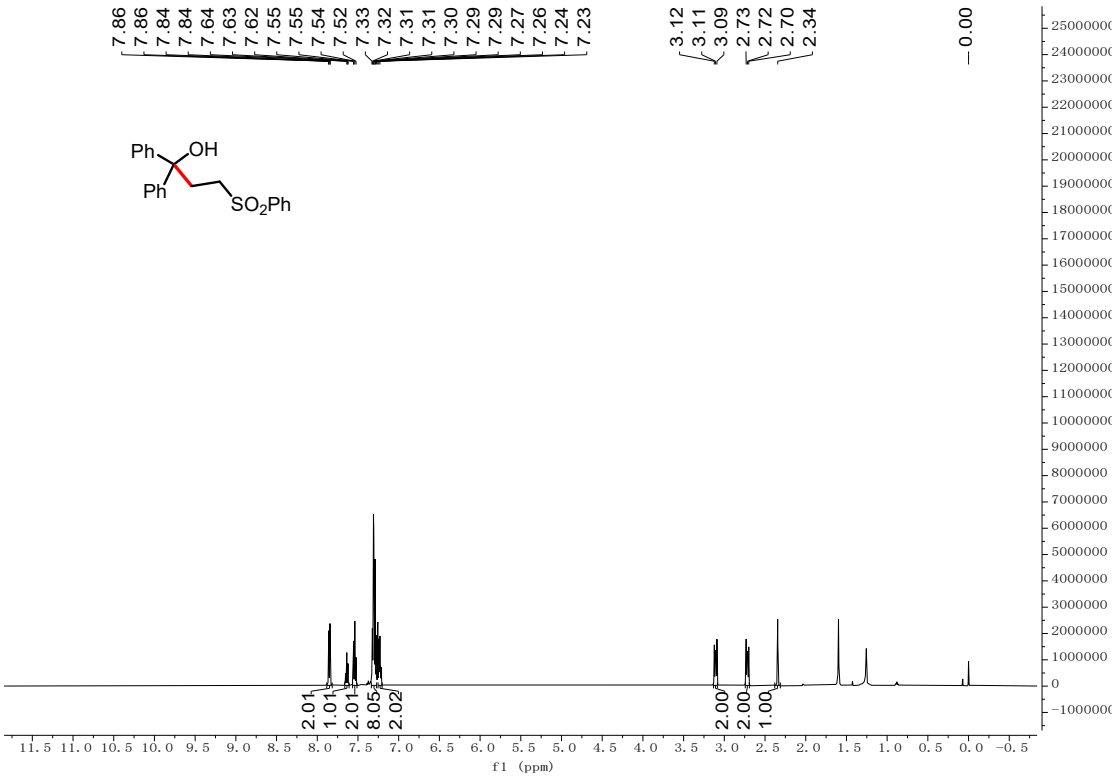
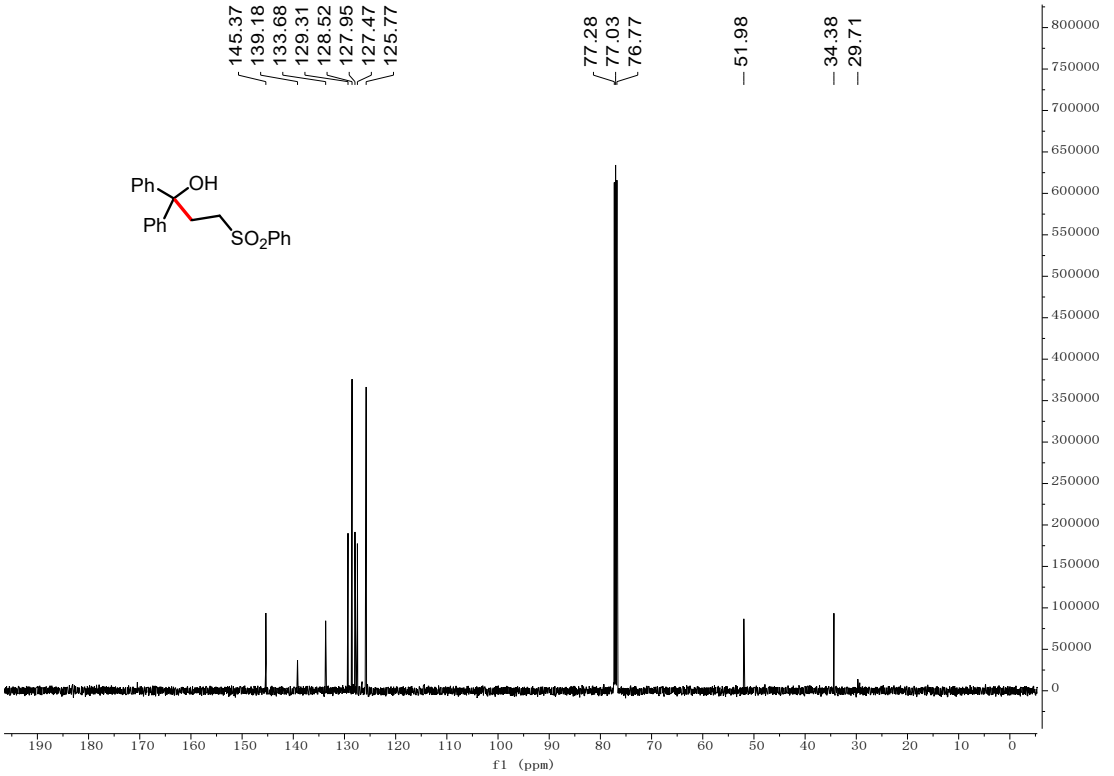


3k ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



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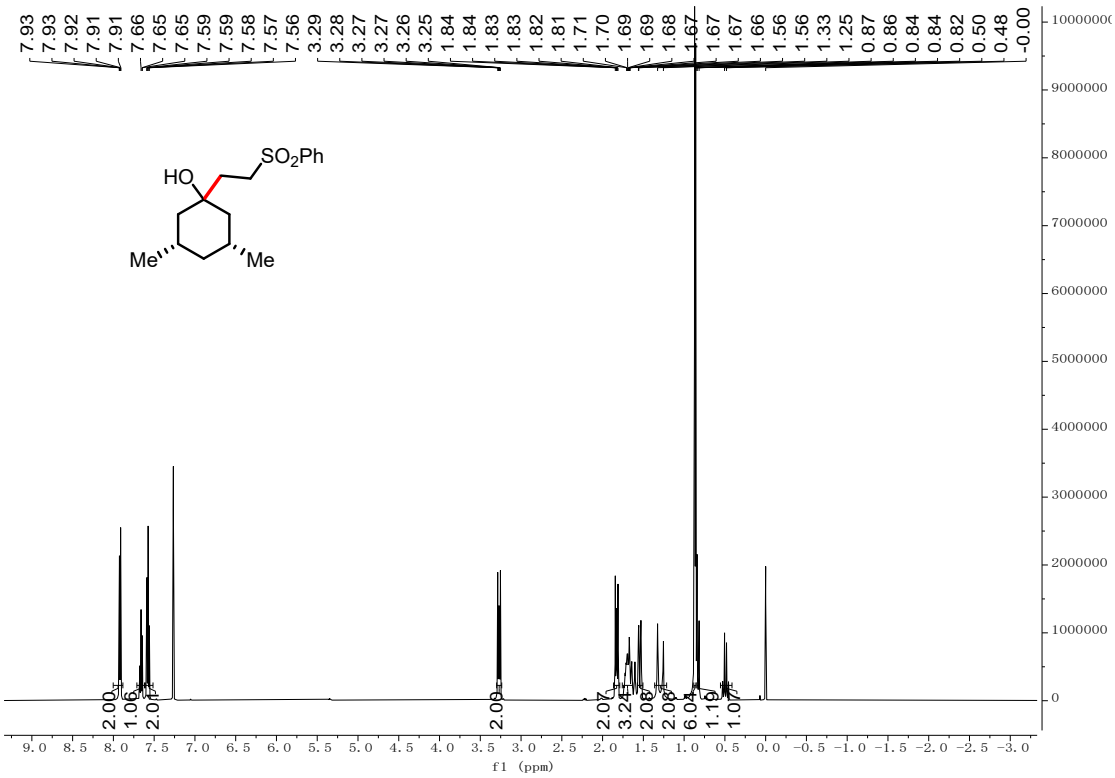

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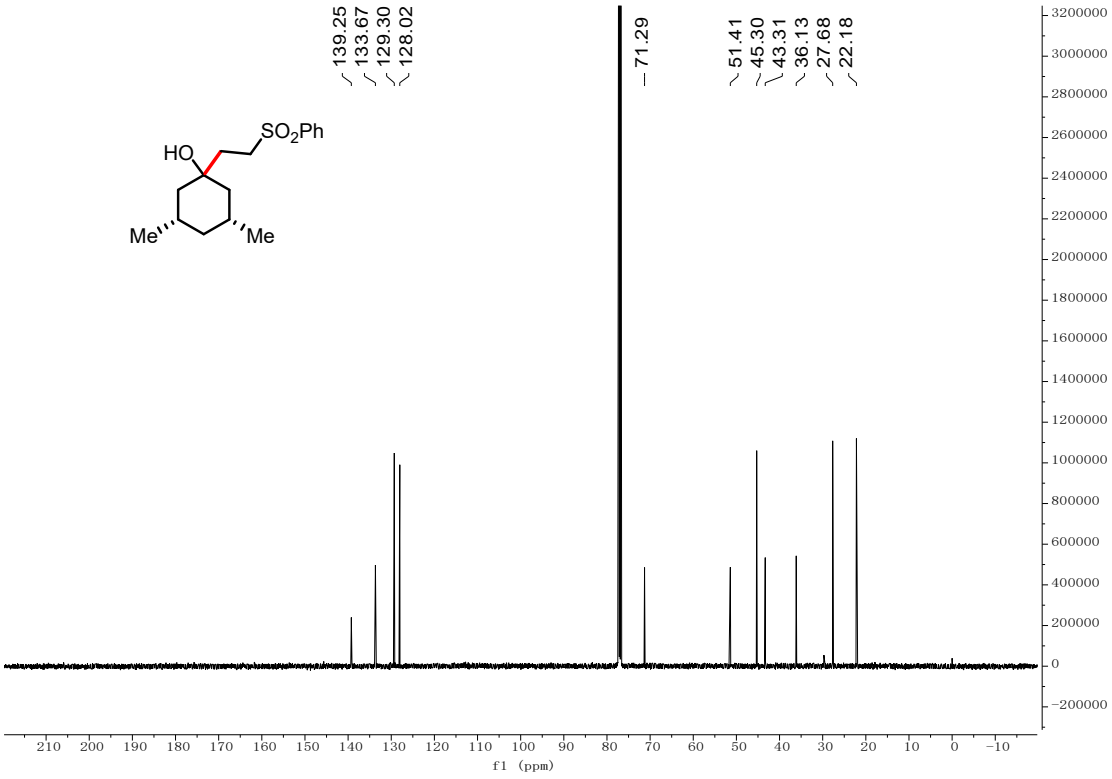

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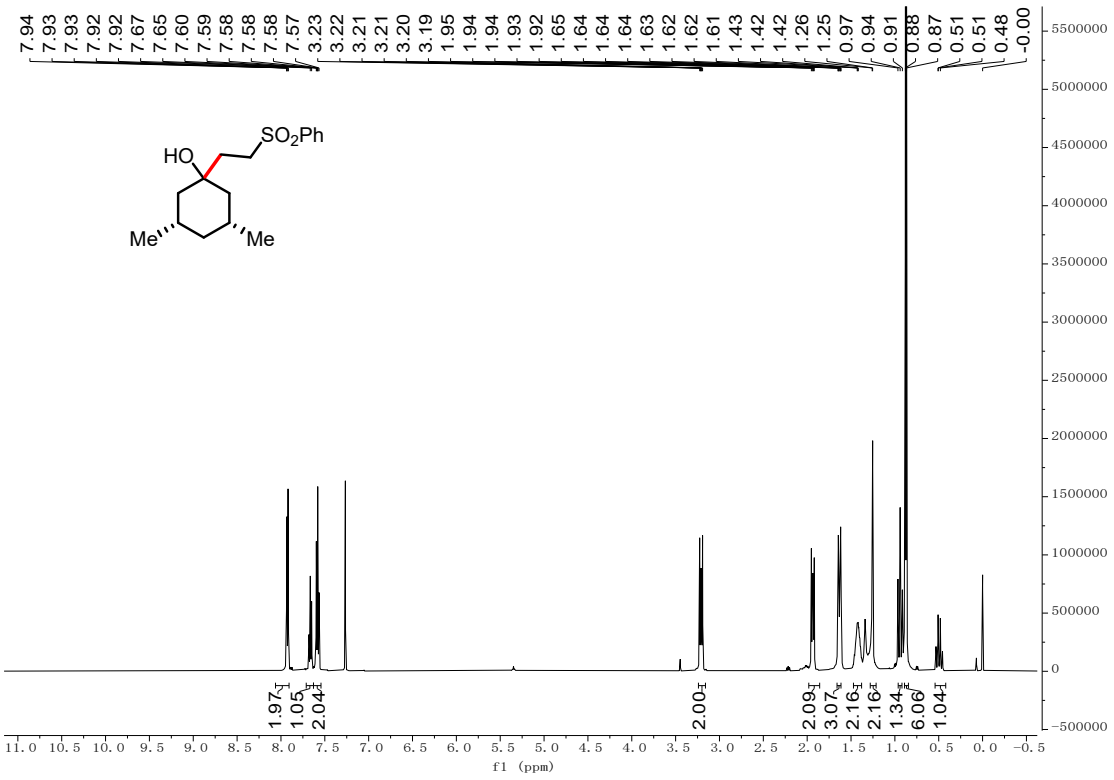

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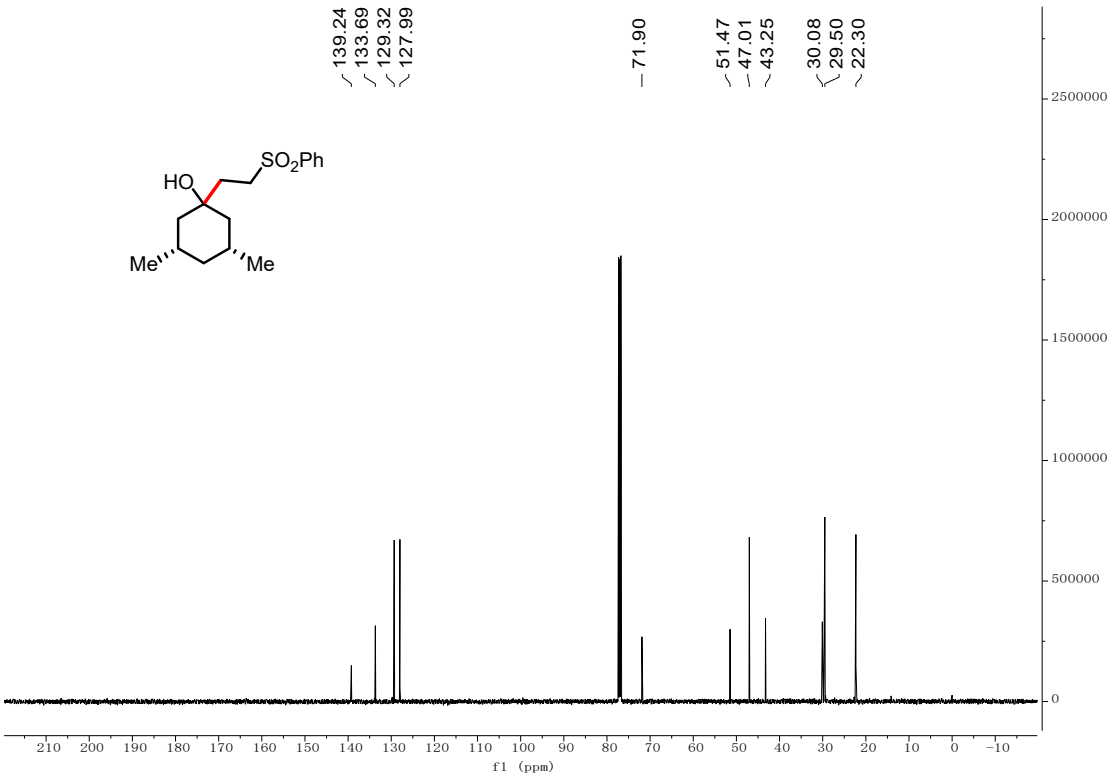
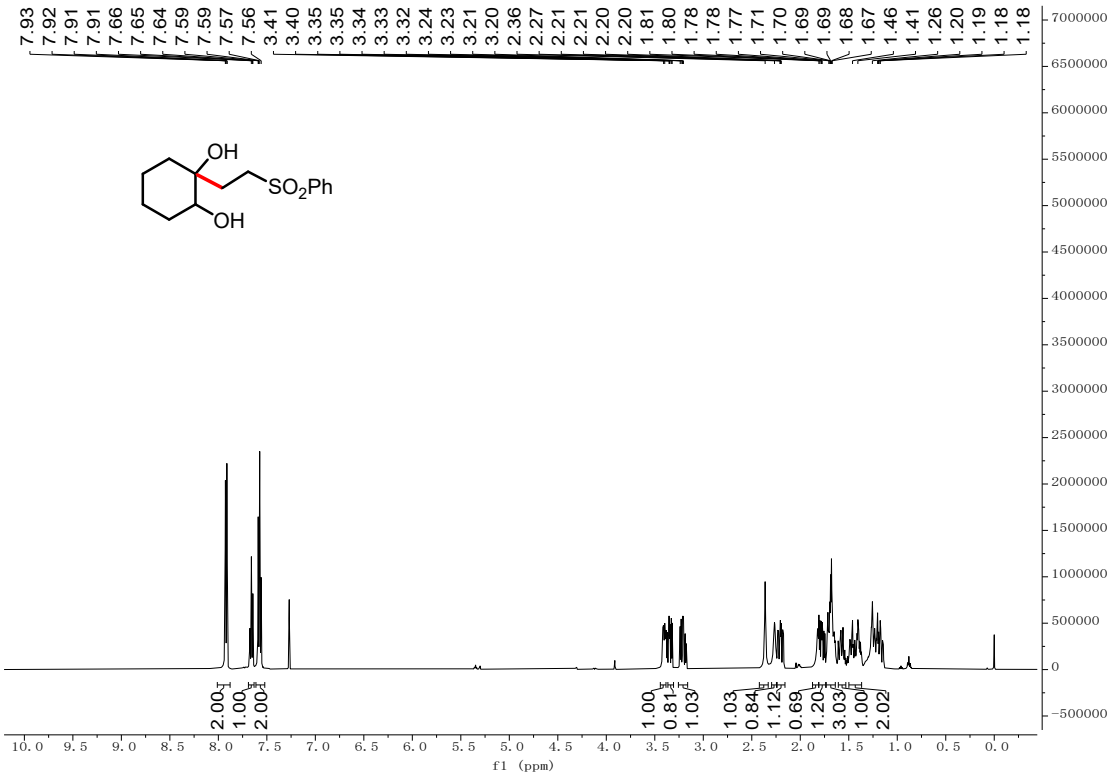


3m (^{1}H NMR, 500 MHz, $CDCl_3$)



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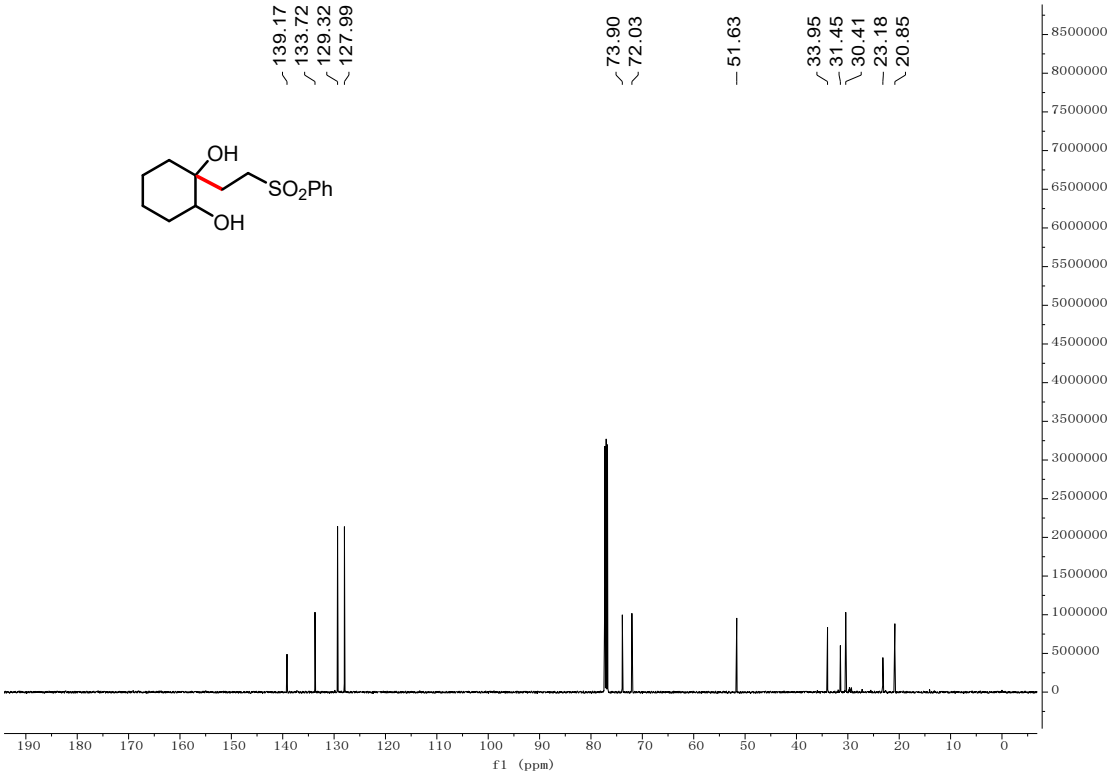

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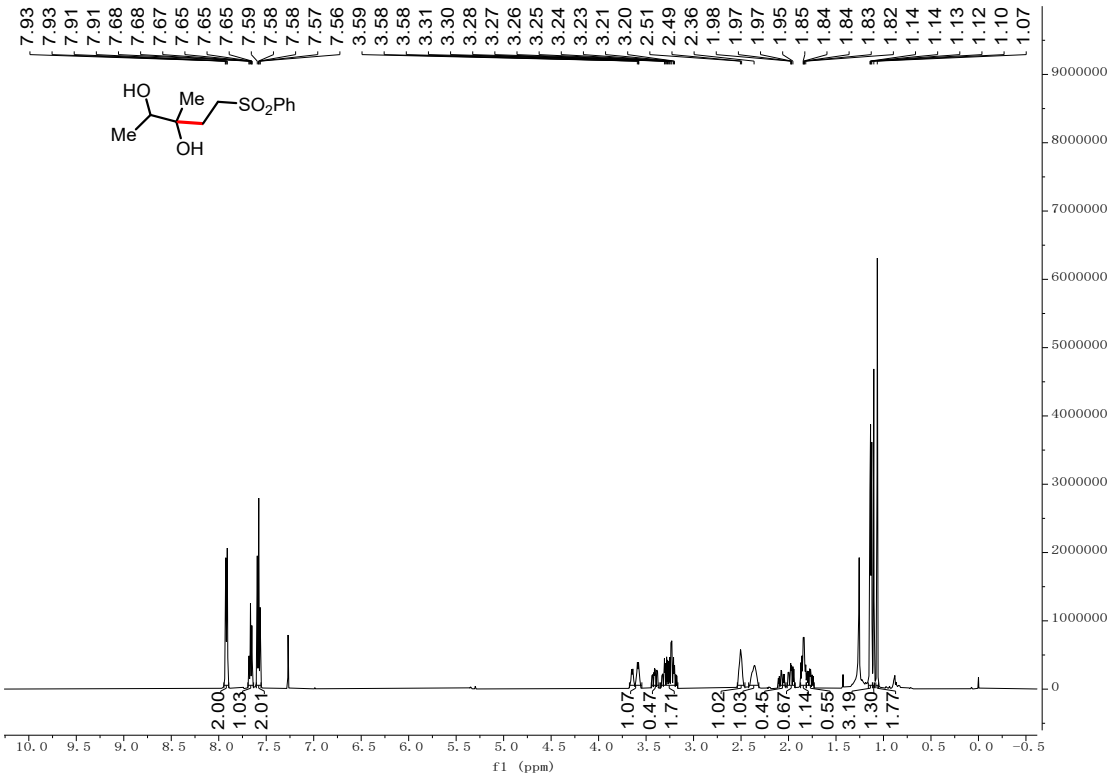
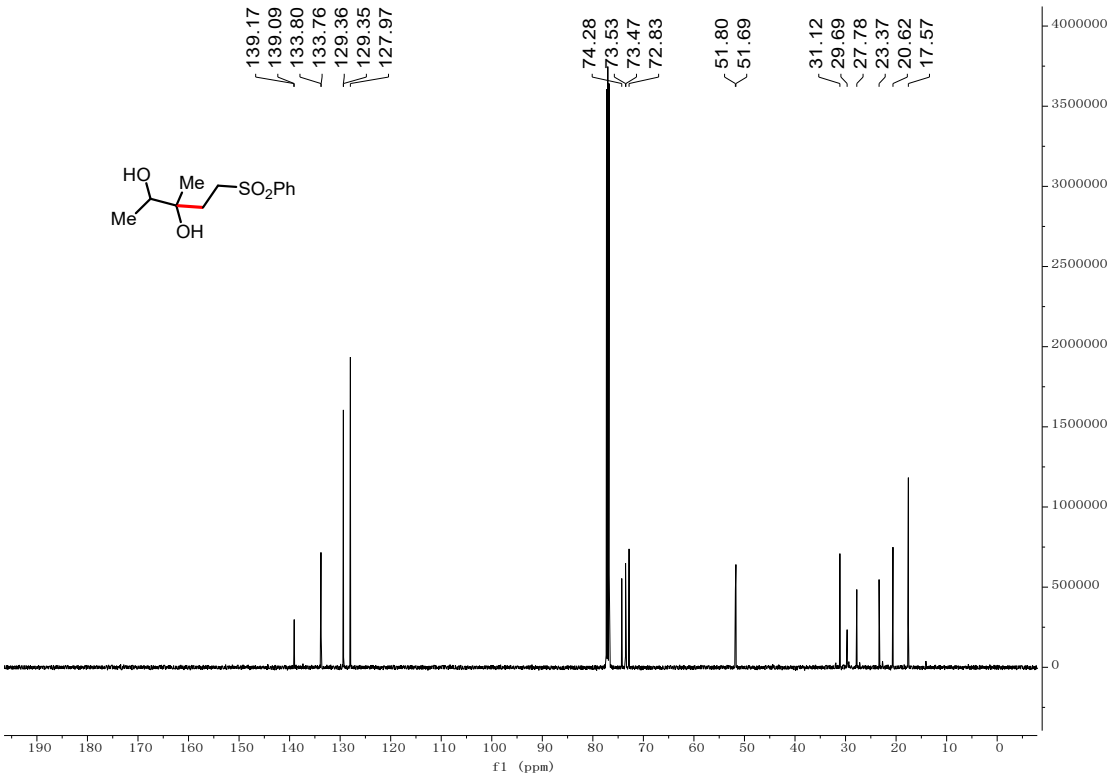
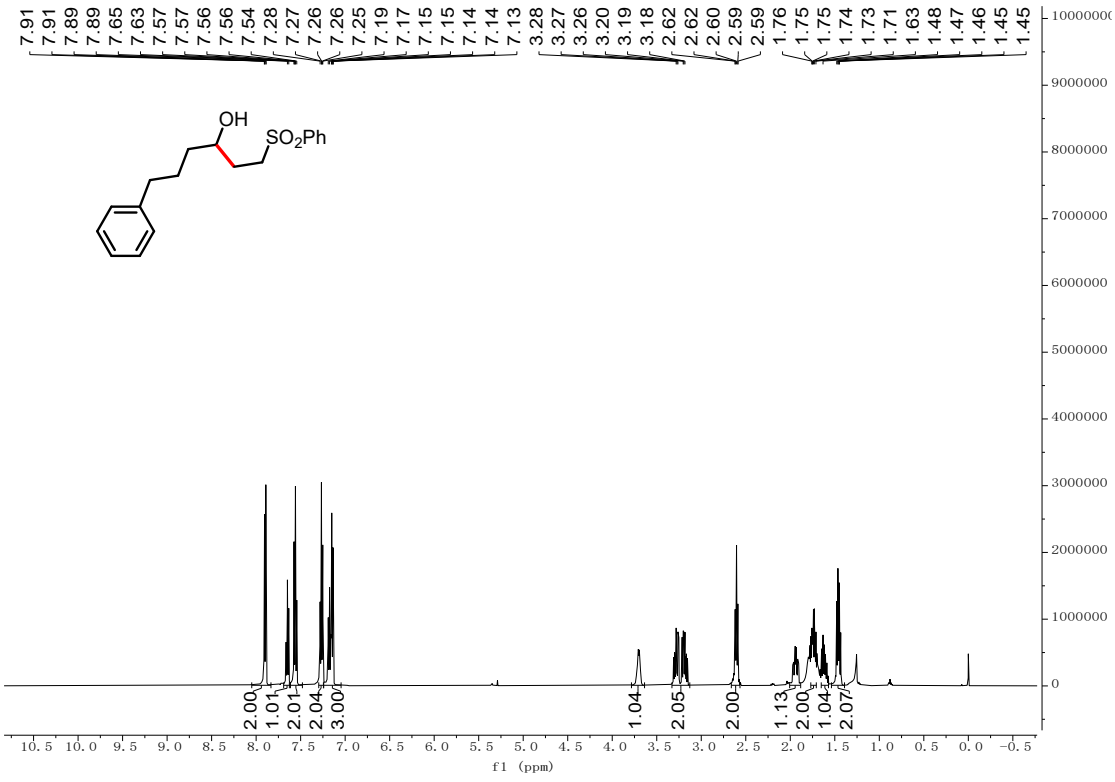


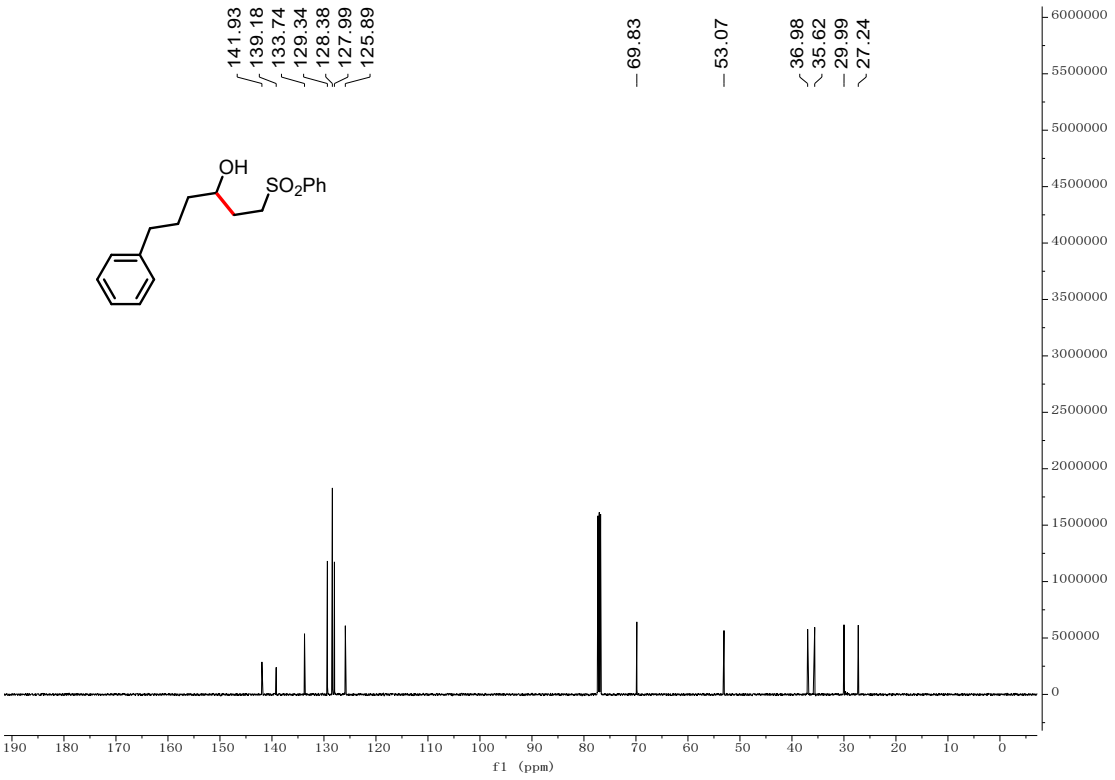
3n (^{13}C{^{1}H} NMR, 125 MHz, $CDCl_3$)



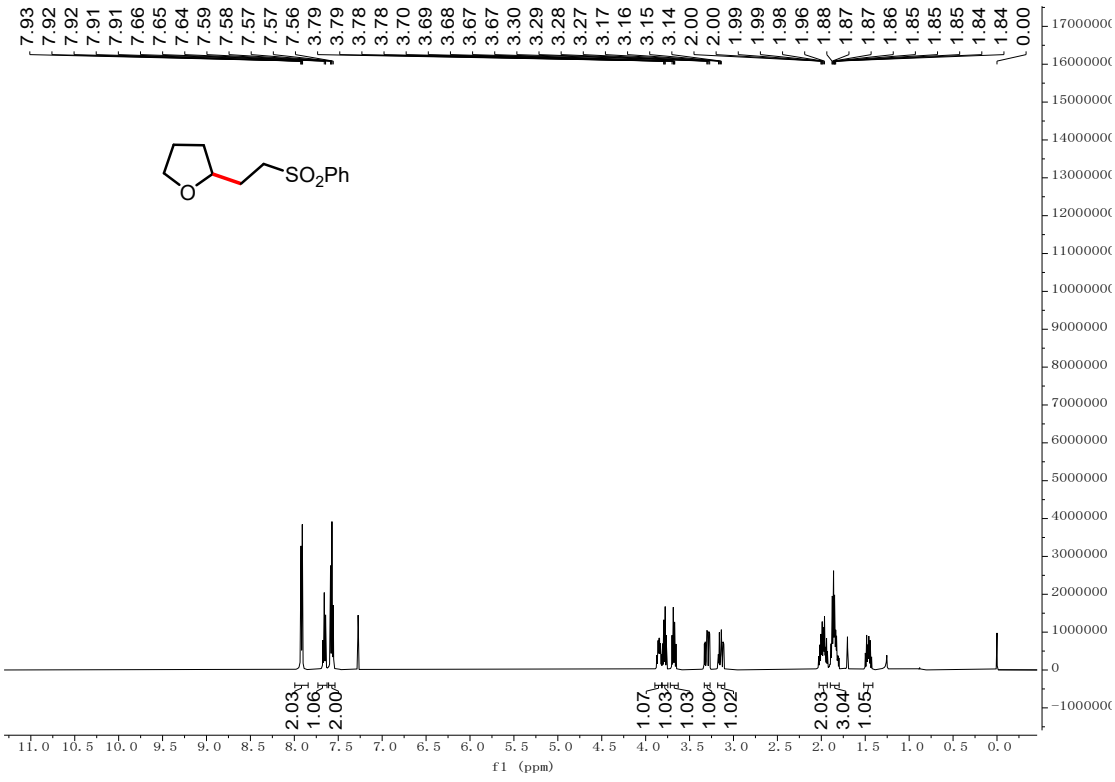
3o (^{1}H NMR, 500 MHz, $CDCl_3$)



3o (^{13}C{^{1}H} NMR, 125 MHz, $CDCl_3$)



3p (^{1}H NMR, 500 MHz, $CDCl_3$)



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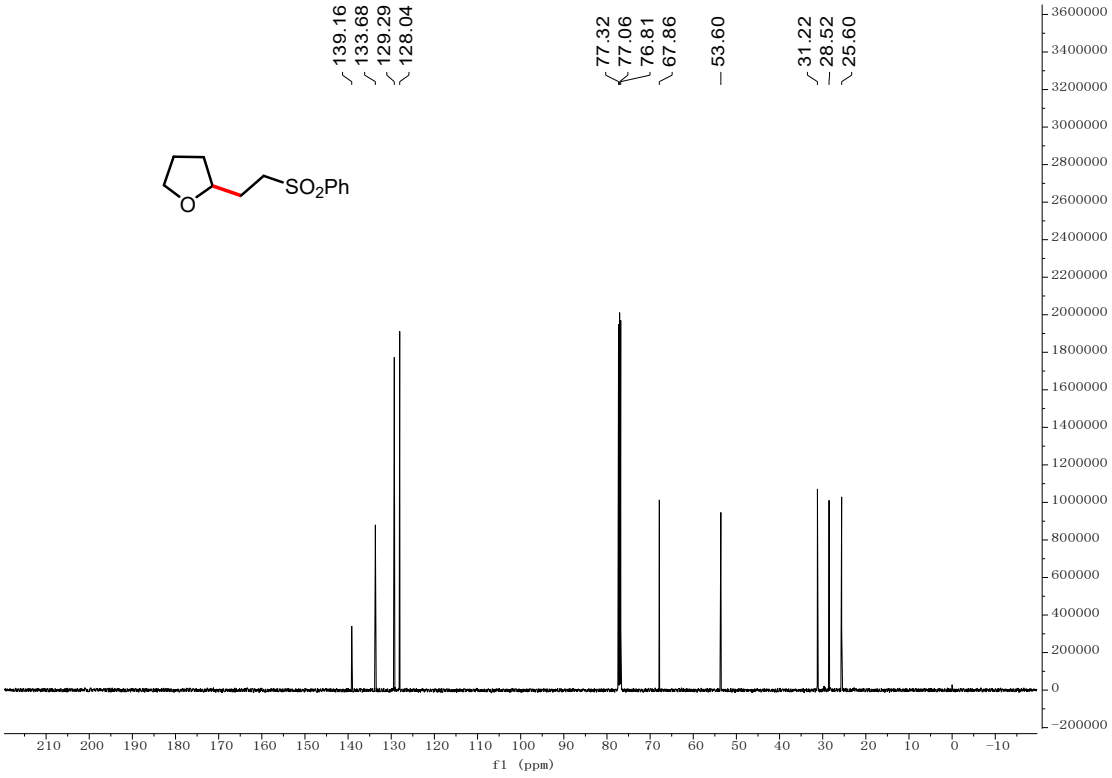
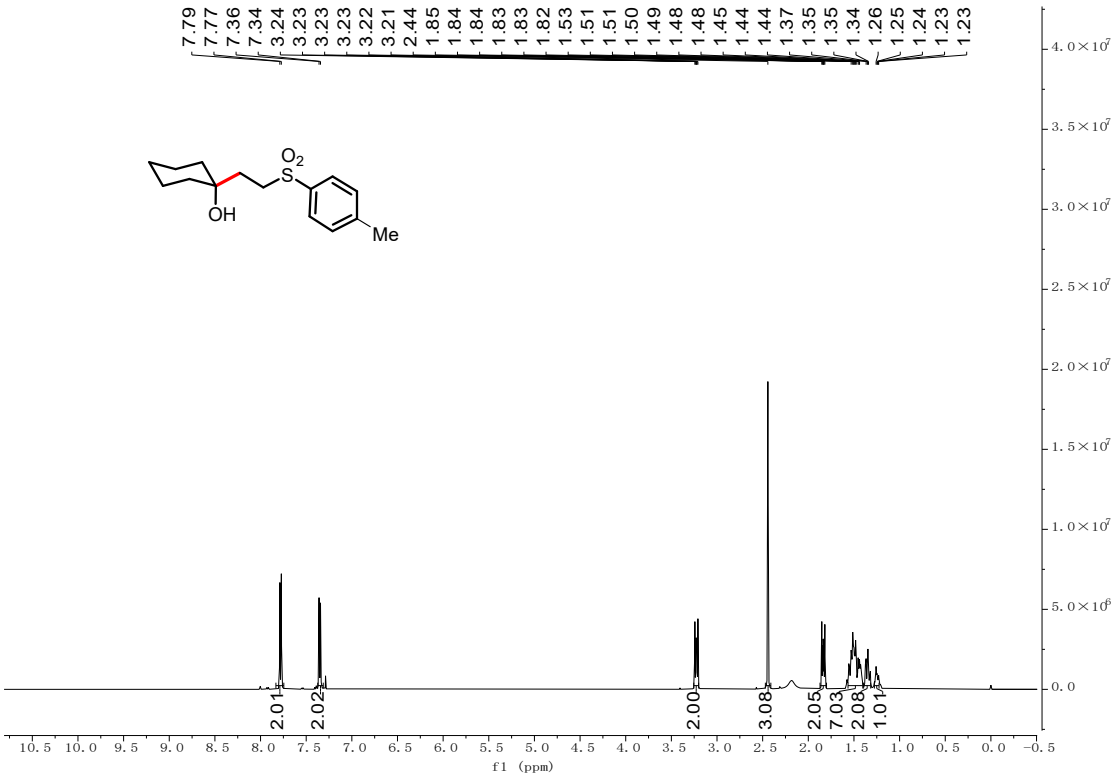
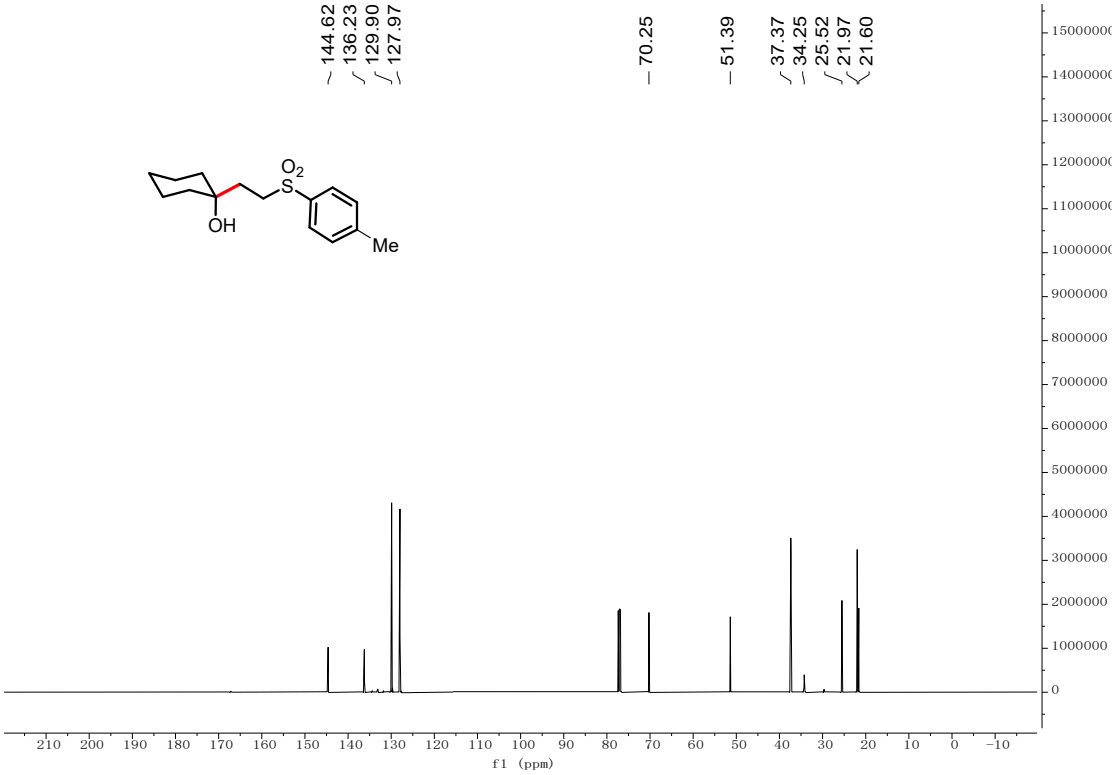


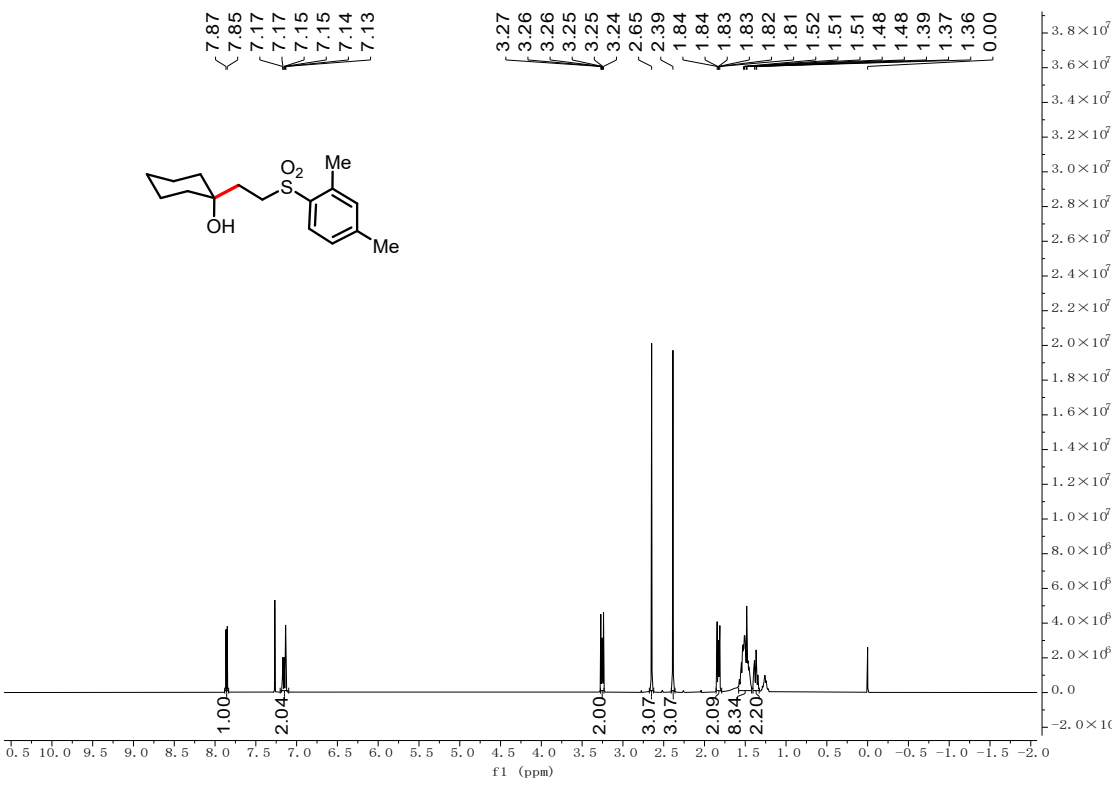
3q (^{1}H NMR, 500 MHz, $CDCl_3$)



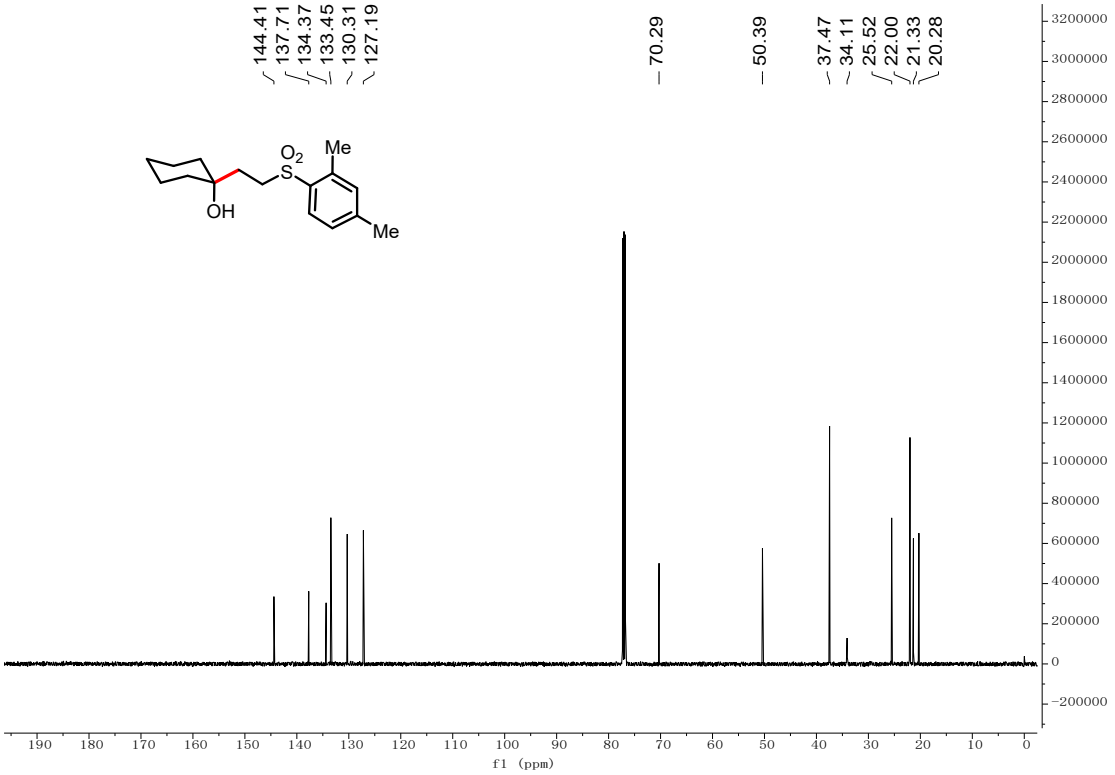
3q ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



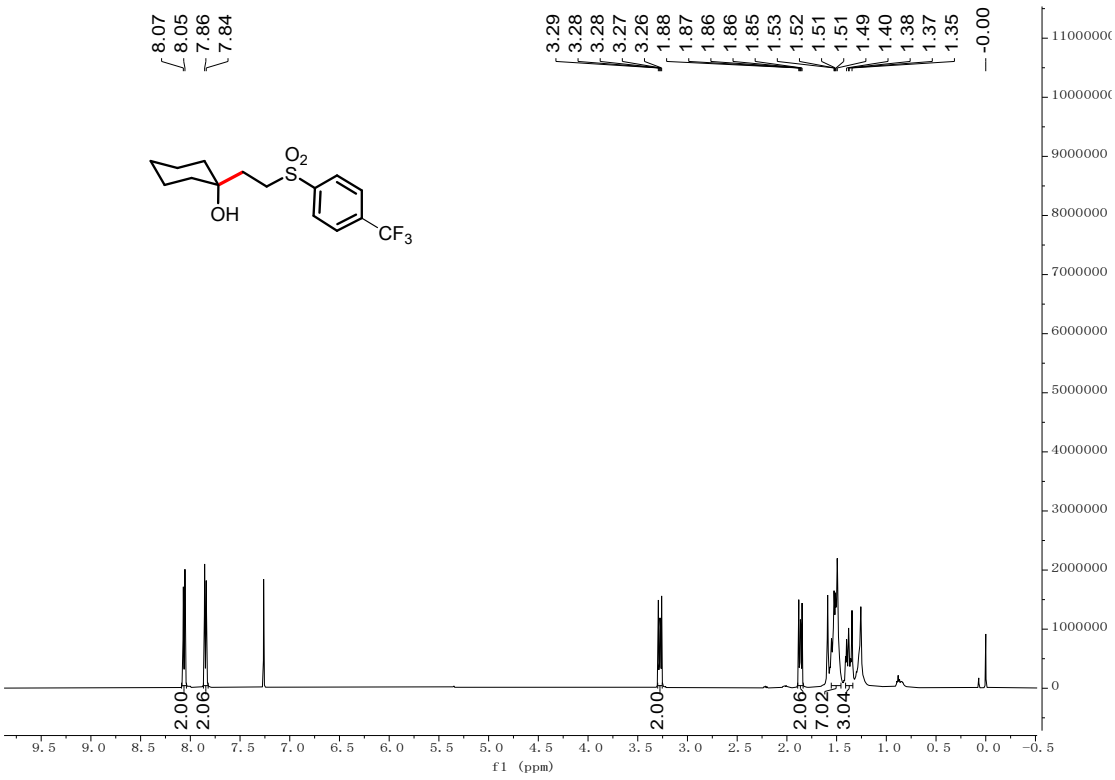
3r (^{1}H NMR, 500 MHz, $CDCl_3$)



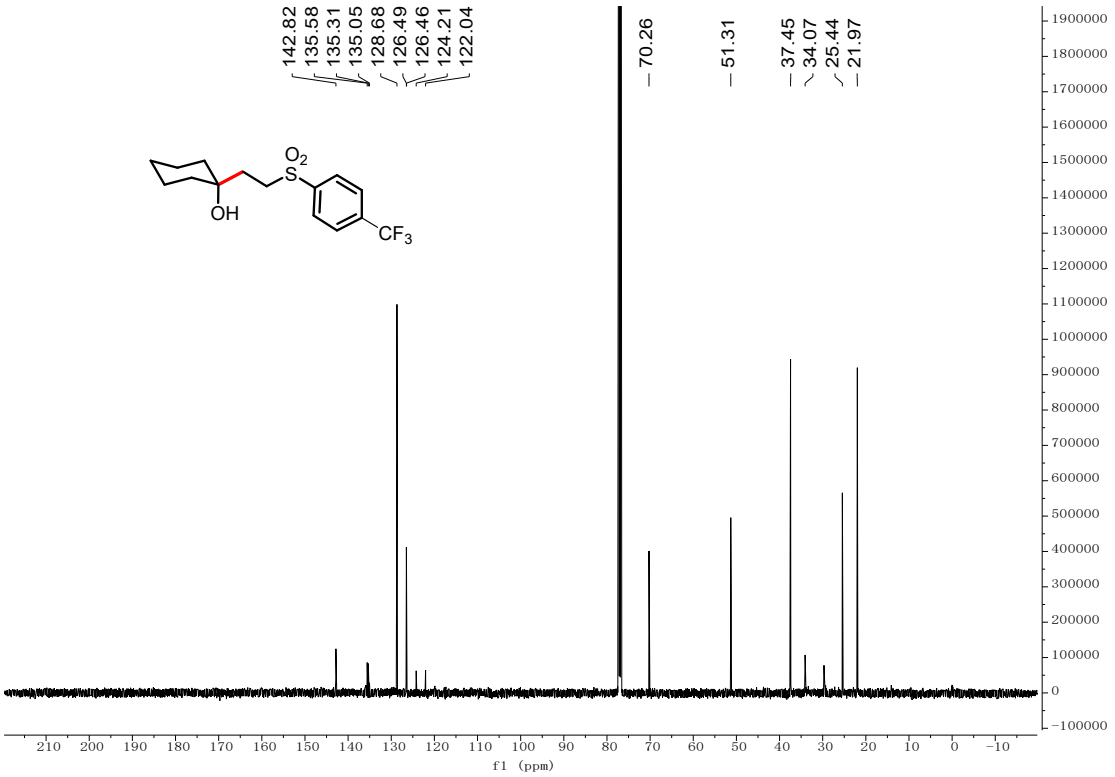
3r ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



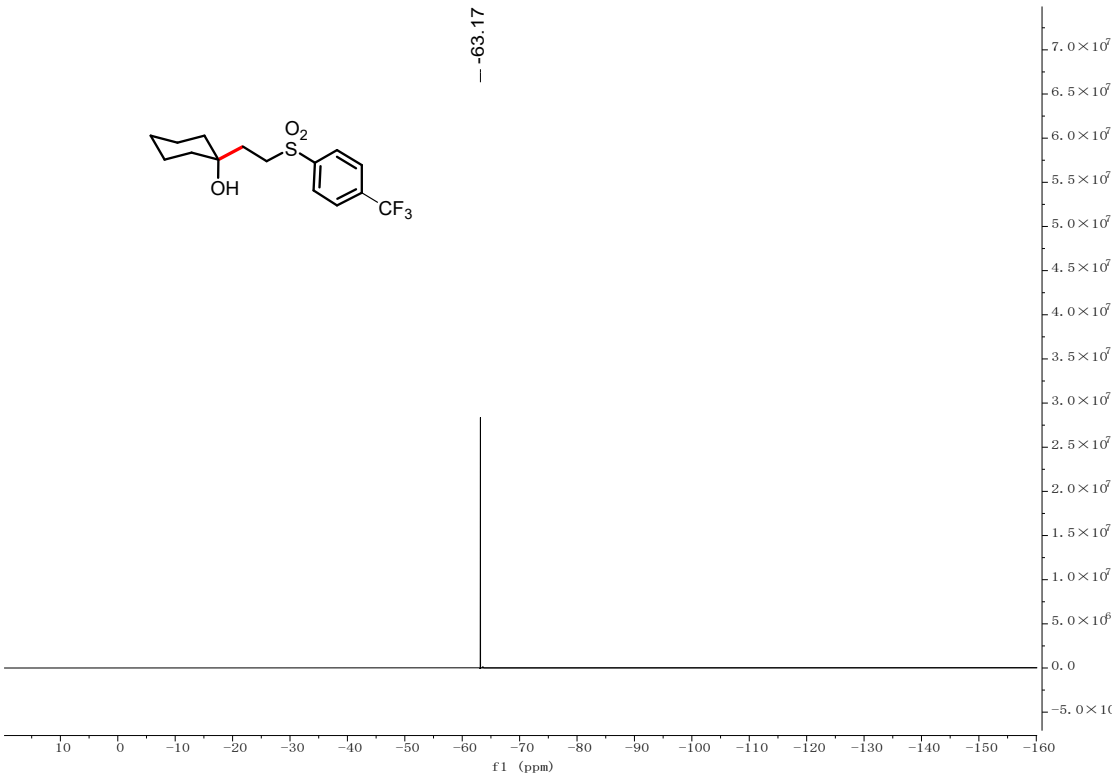
3s (^{1}H NMR, 500 MHz, $CDCl_3$)



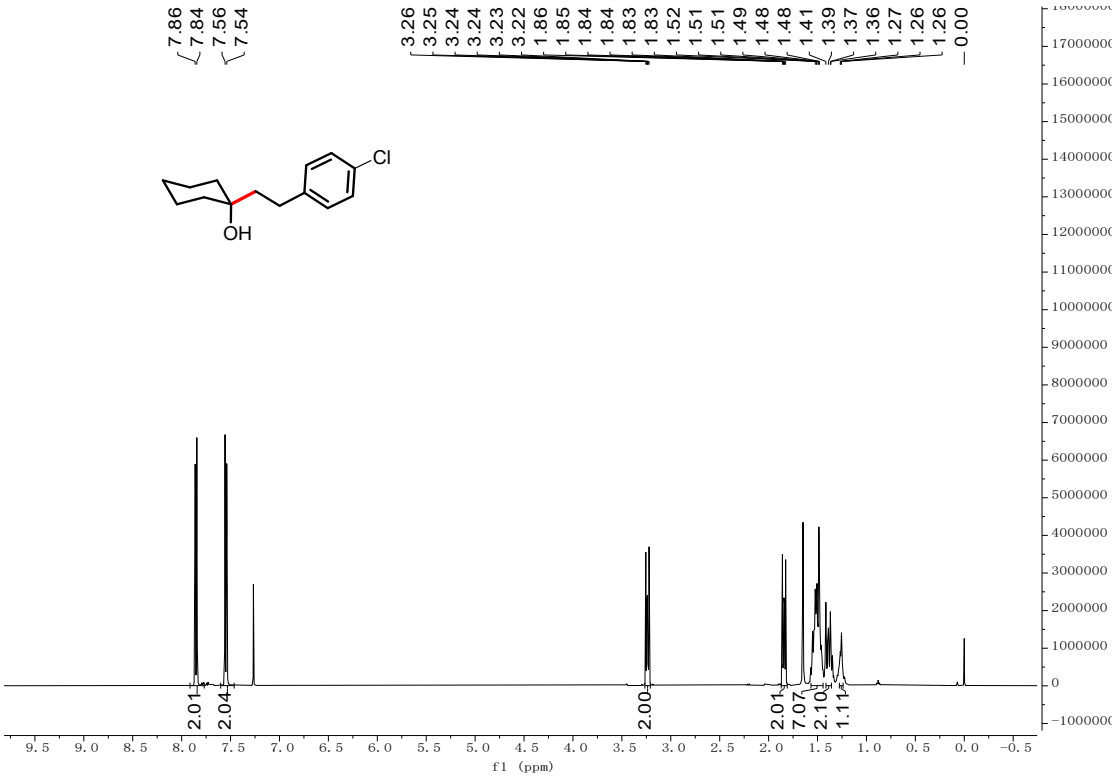
3s ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



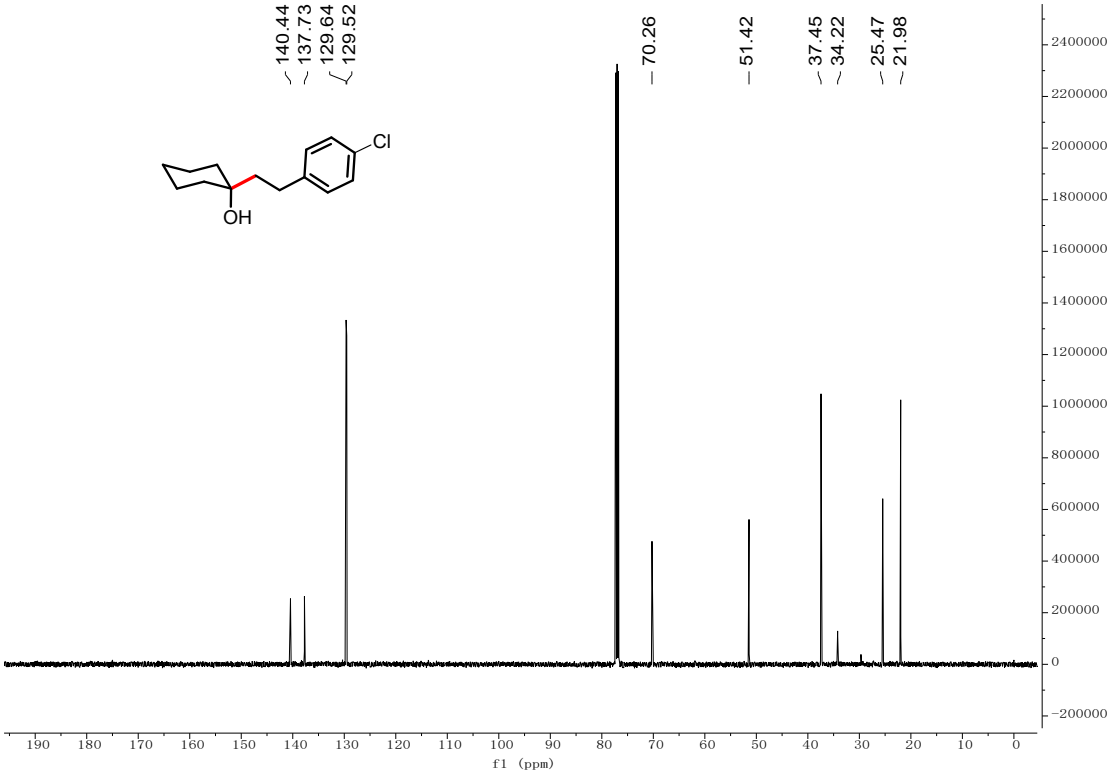
3s (^{19}F NMR 471 MHz, $CDCl_3$)



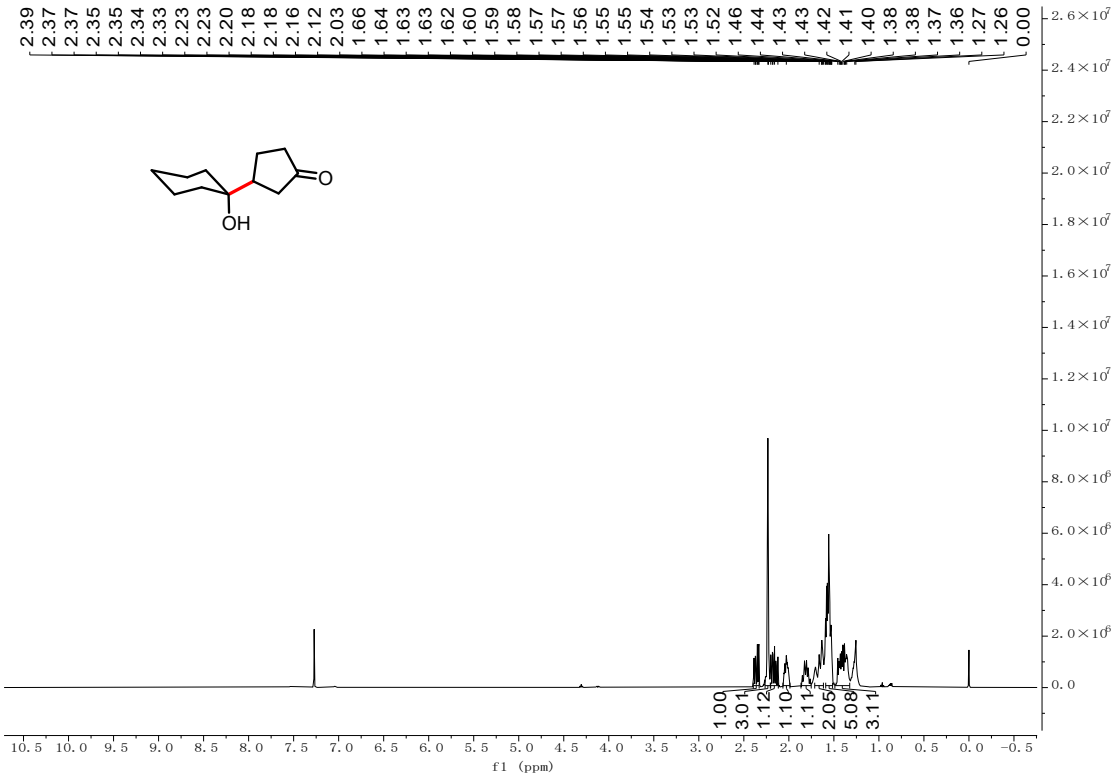
3t (^{1}H NMR, 500 MHz, $CDCl_3$)



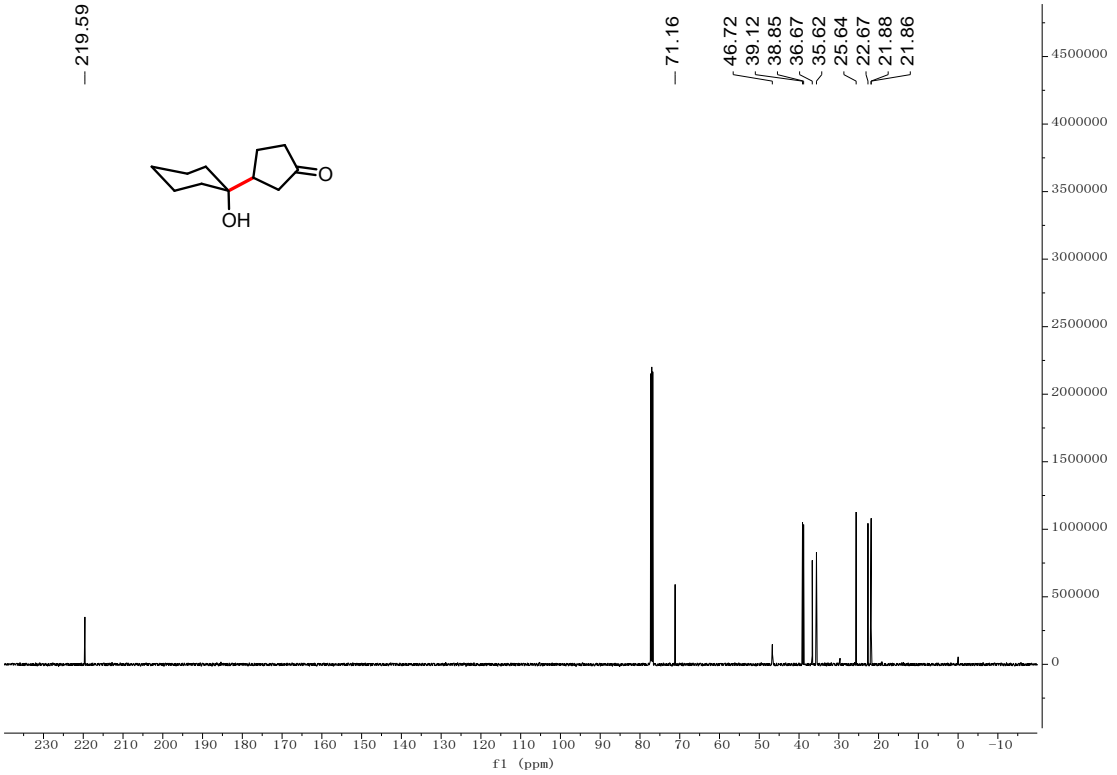
3t ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



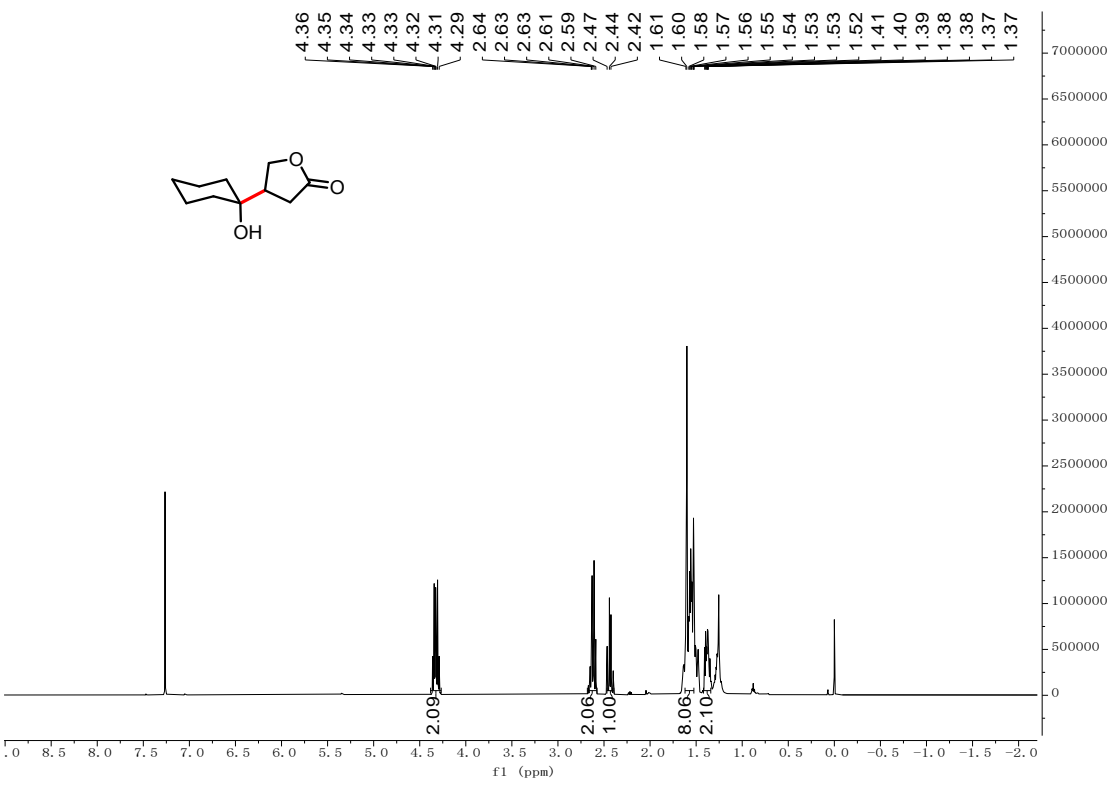
3u (^{1}H NMR, 500 MHz, $CDCl_3$)



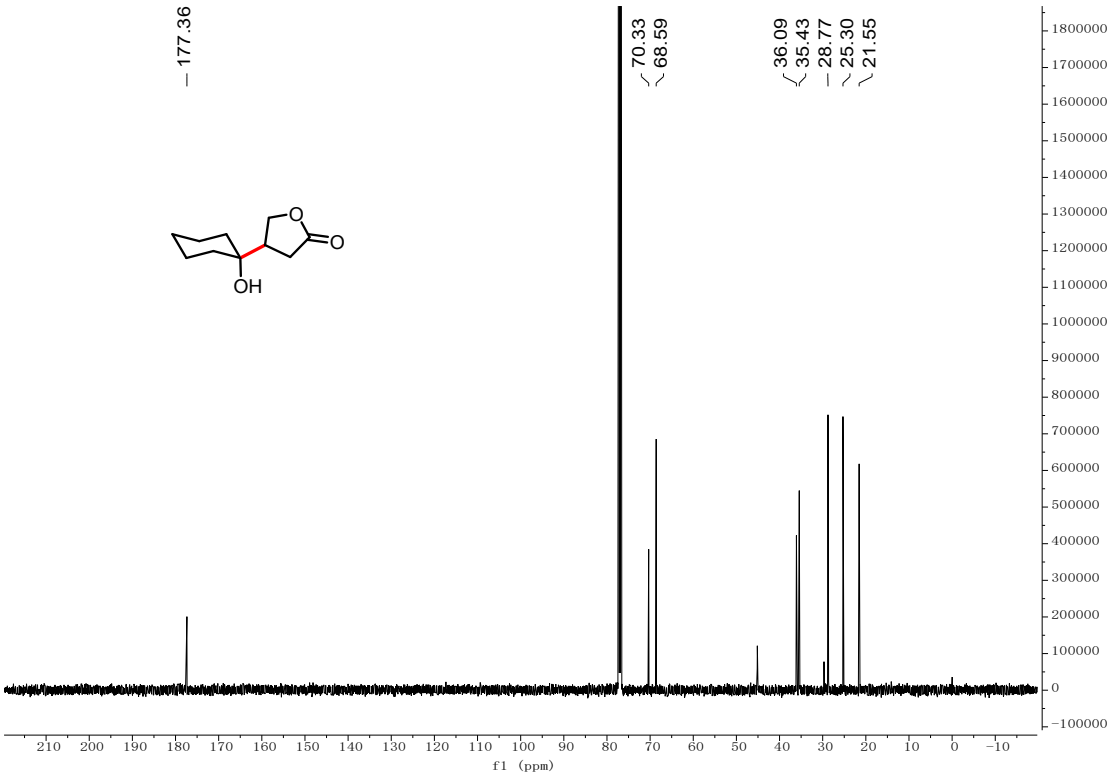
3u ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



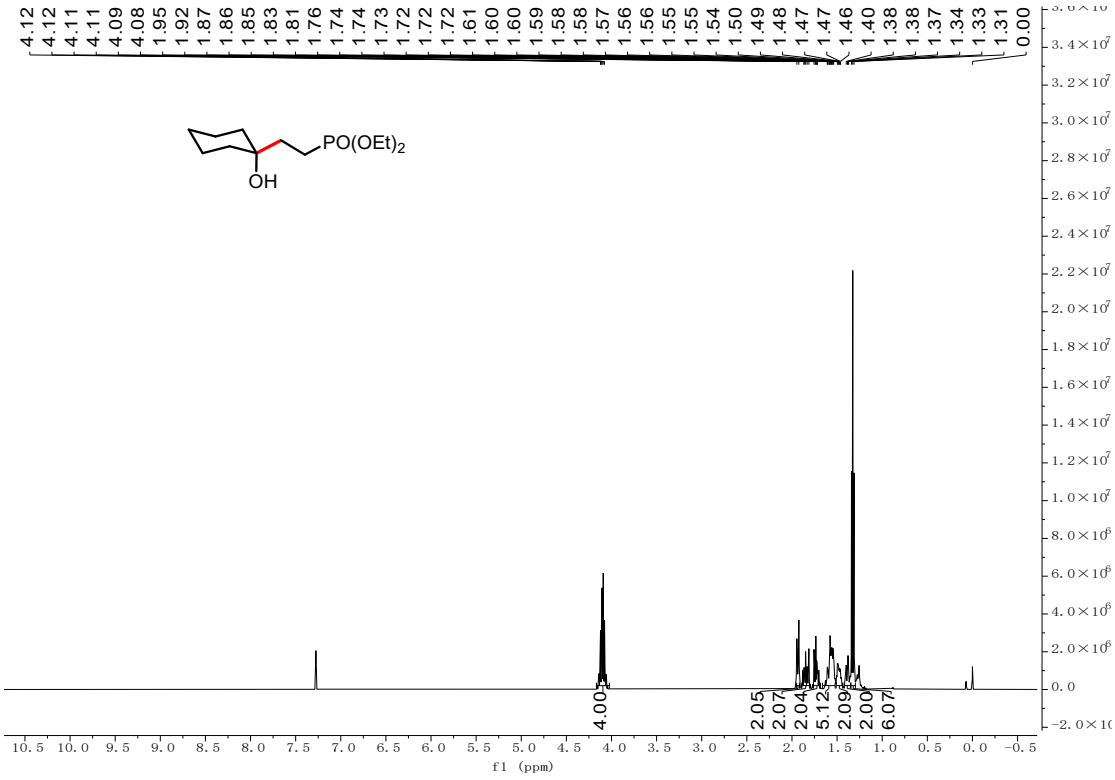
3v (^{1}H NMR, 500 MHz, $CDCl_3$)



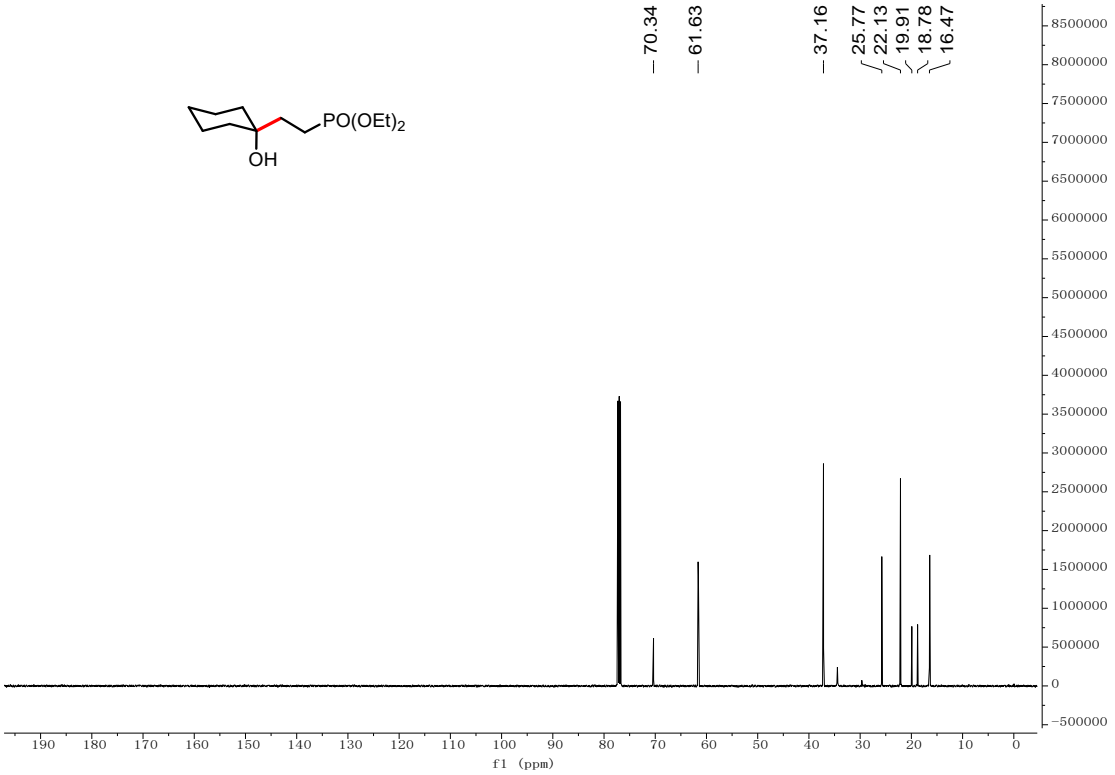
3v ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



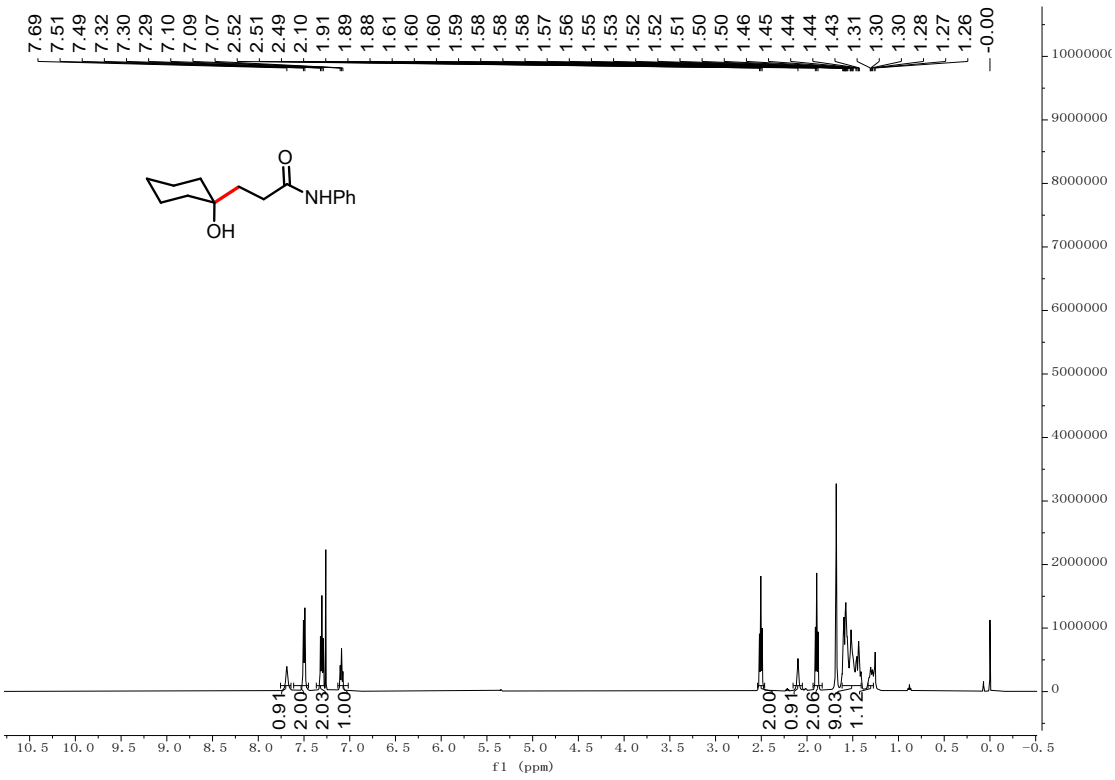
3w (^{1}H NMR, 500 MHz, $CDCl_3$)



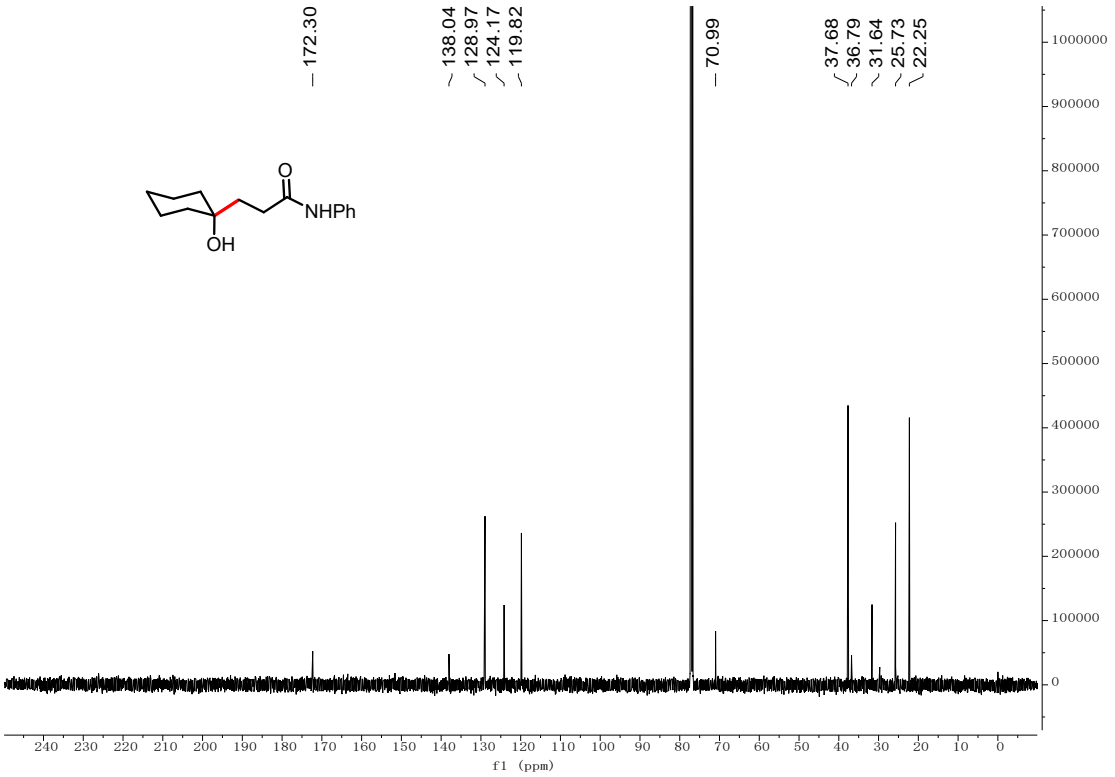
3w ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



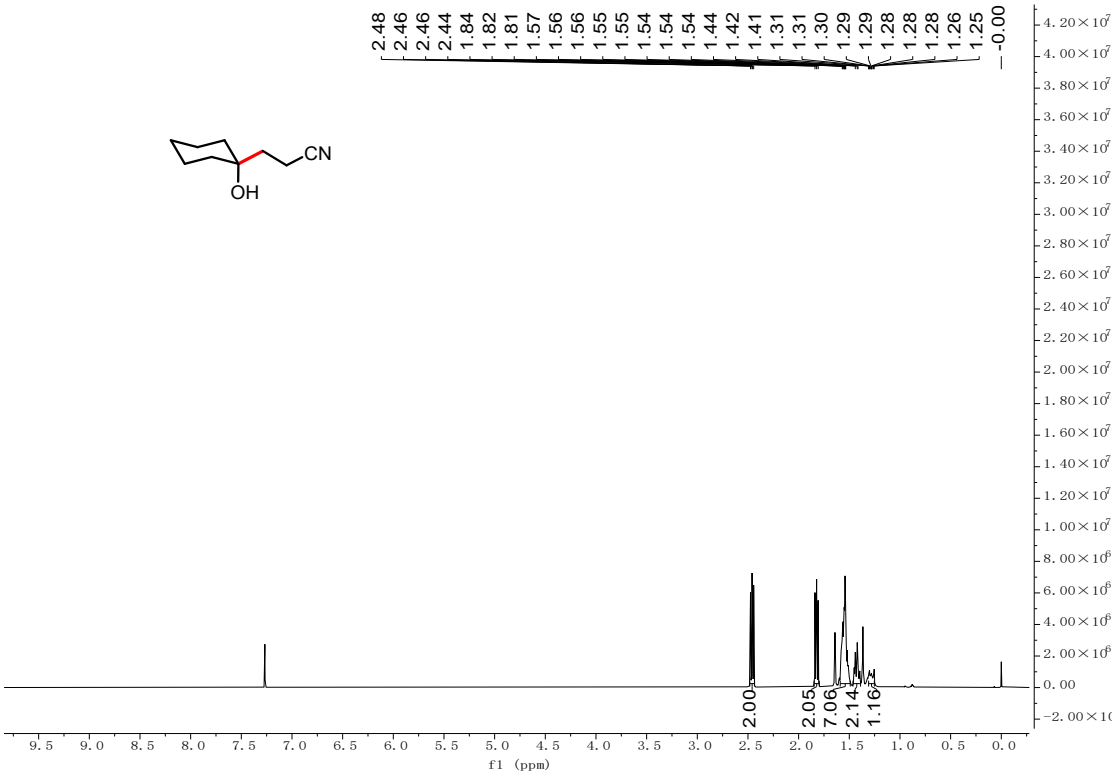
3x (^{1}H NMR, 500 MHz, $CDCl_3$)



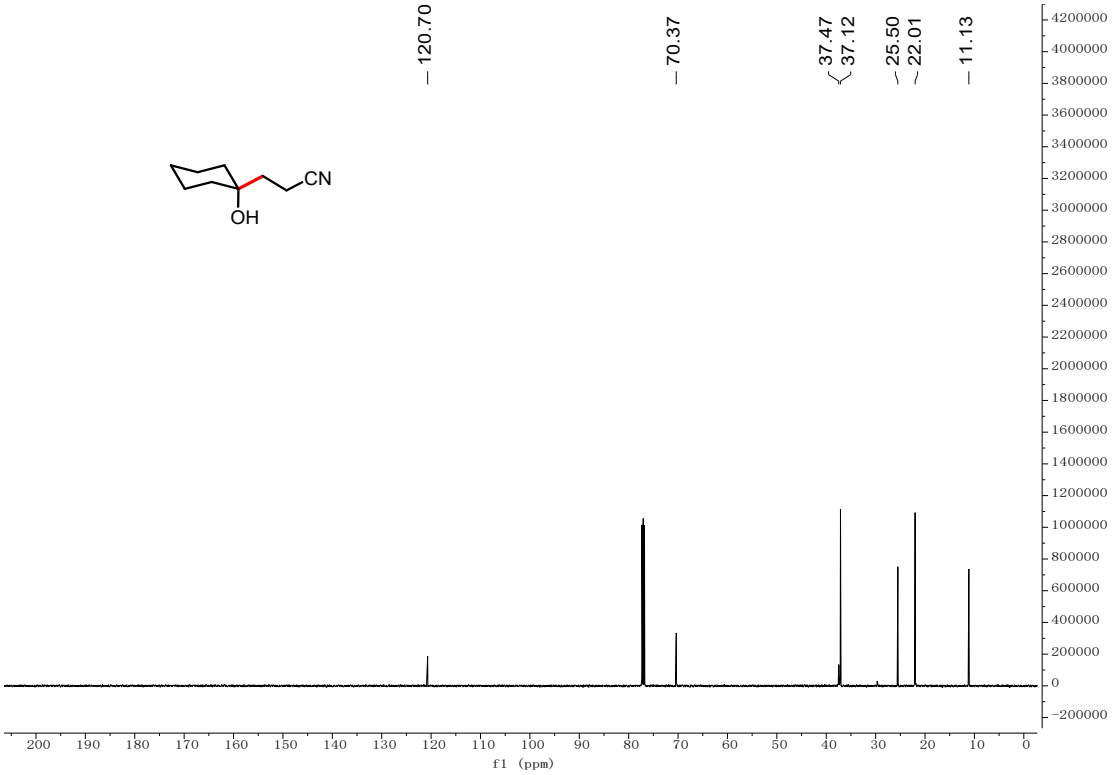
3x ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



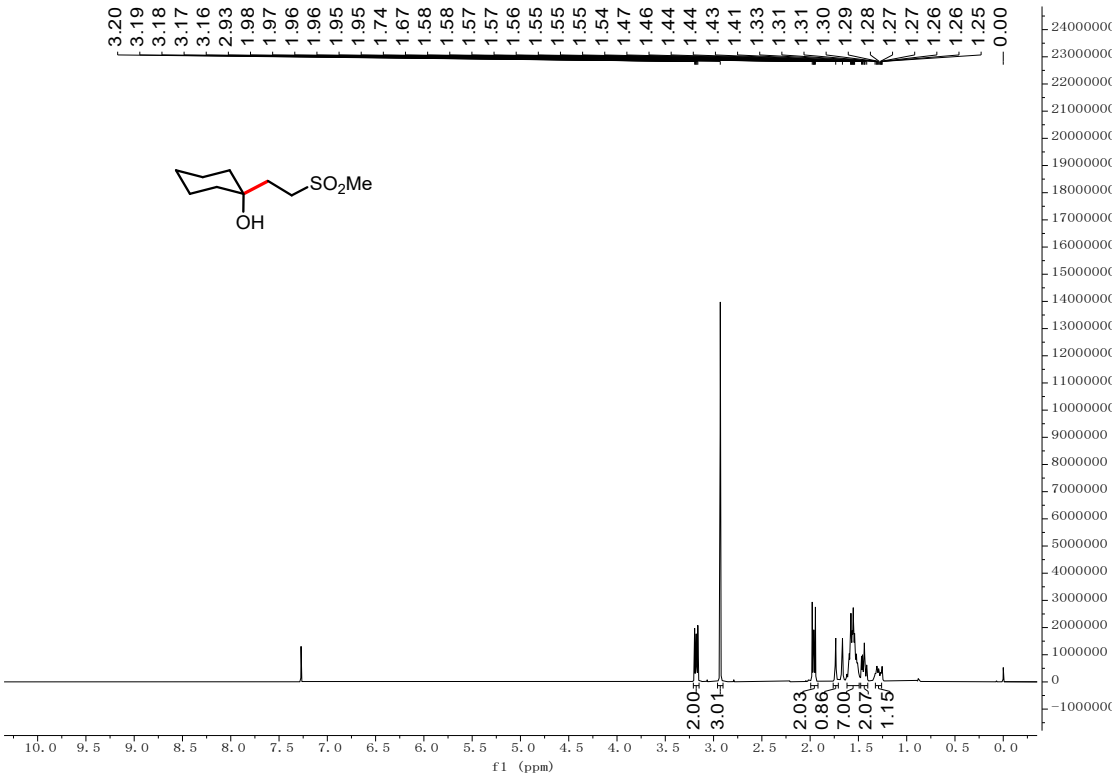
3y (^{1}H NMR, 500 MHz, $CDCl_3$)



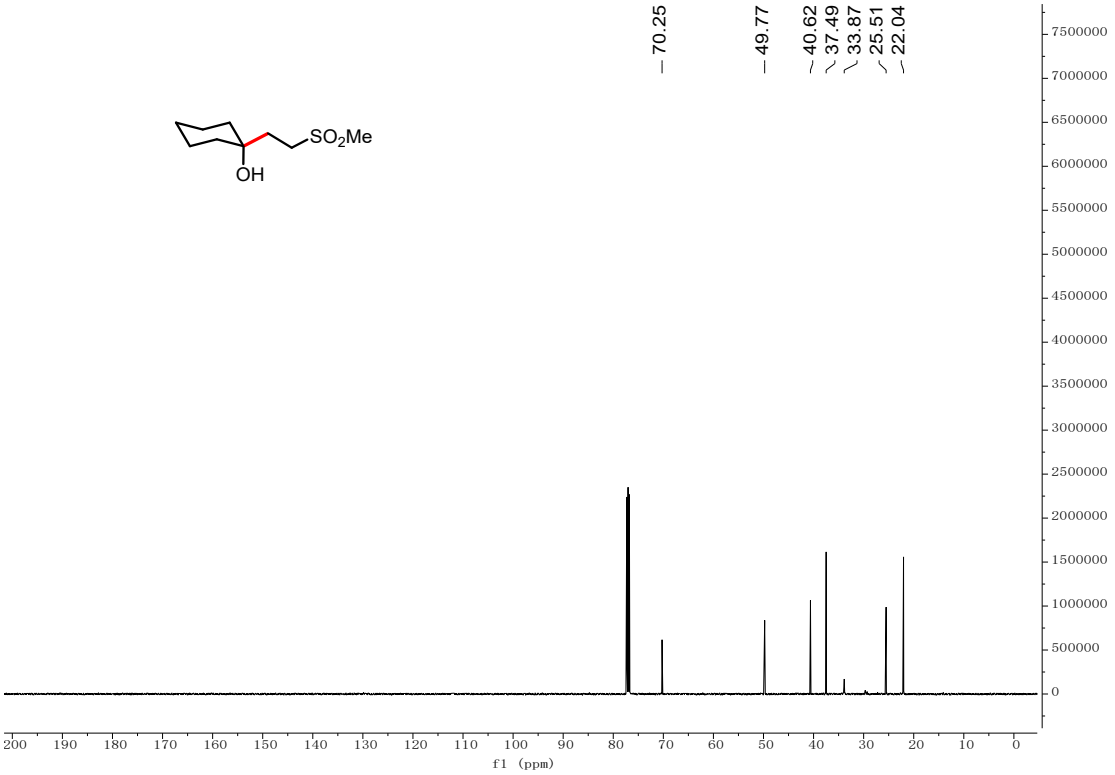
3y ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



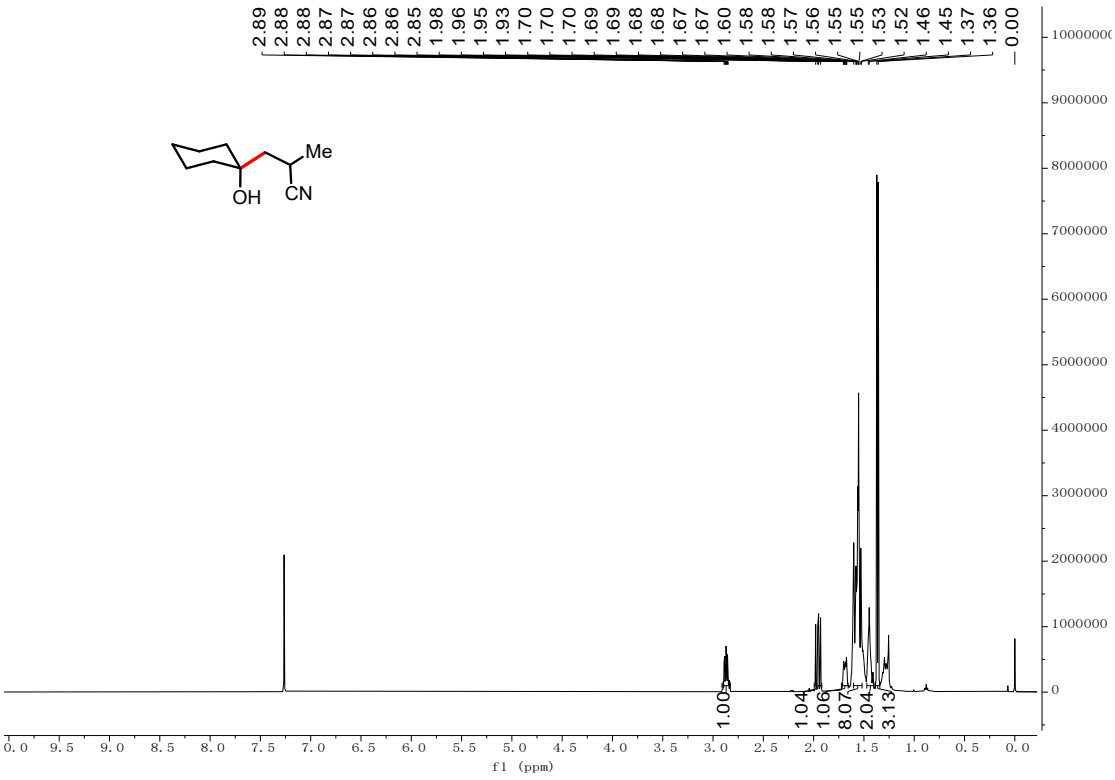
3z (^{1}H NMR, 500 MHz, $CDCl_3$)



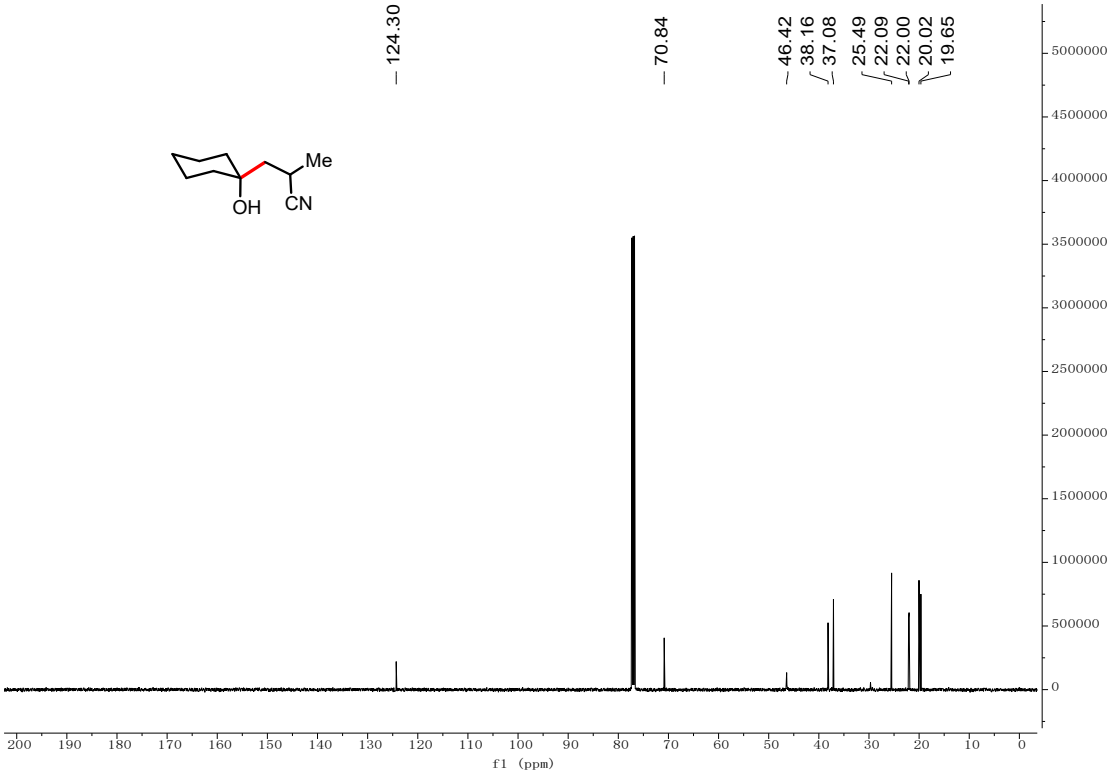
3z ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



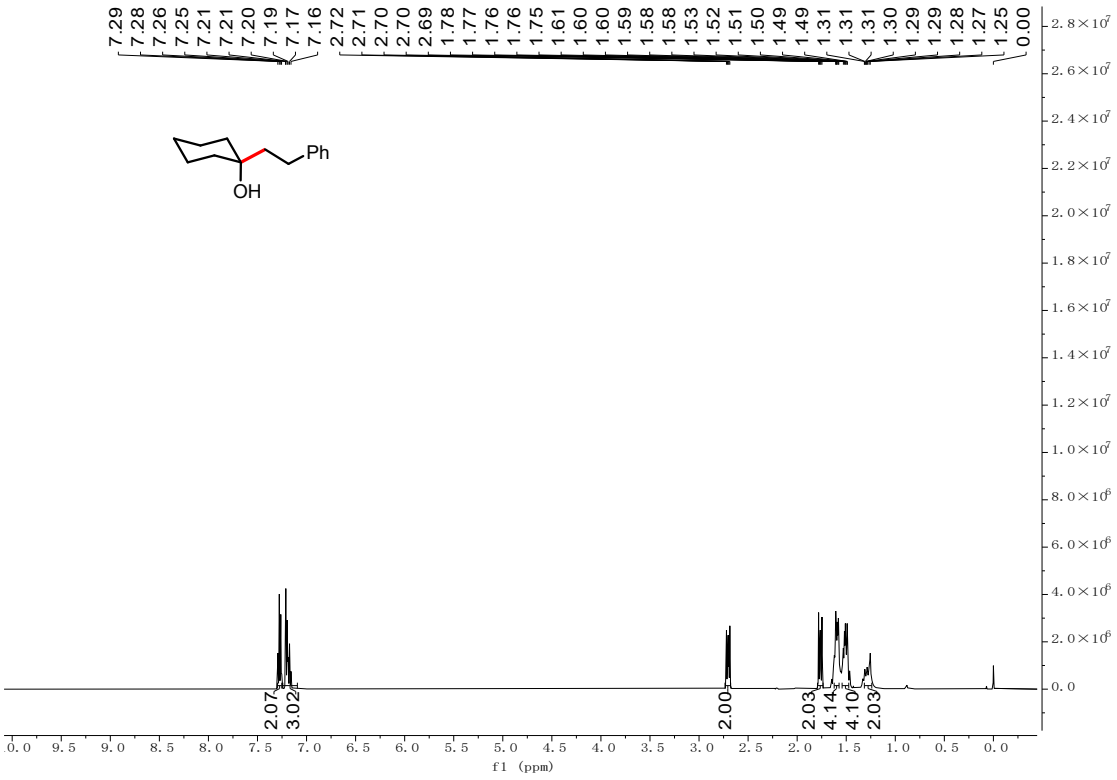
3aa (^{1}H NMR, 500 MHz, $CDCl_3$)



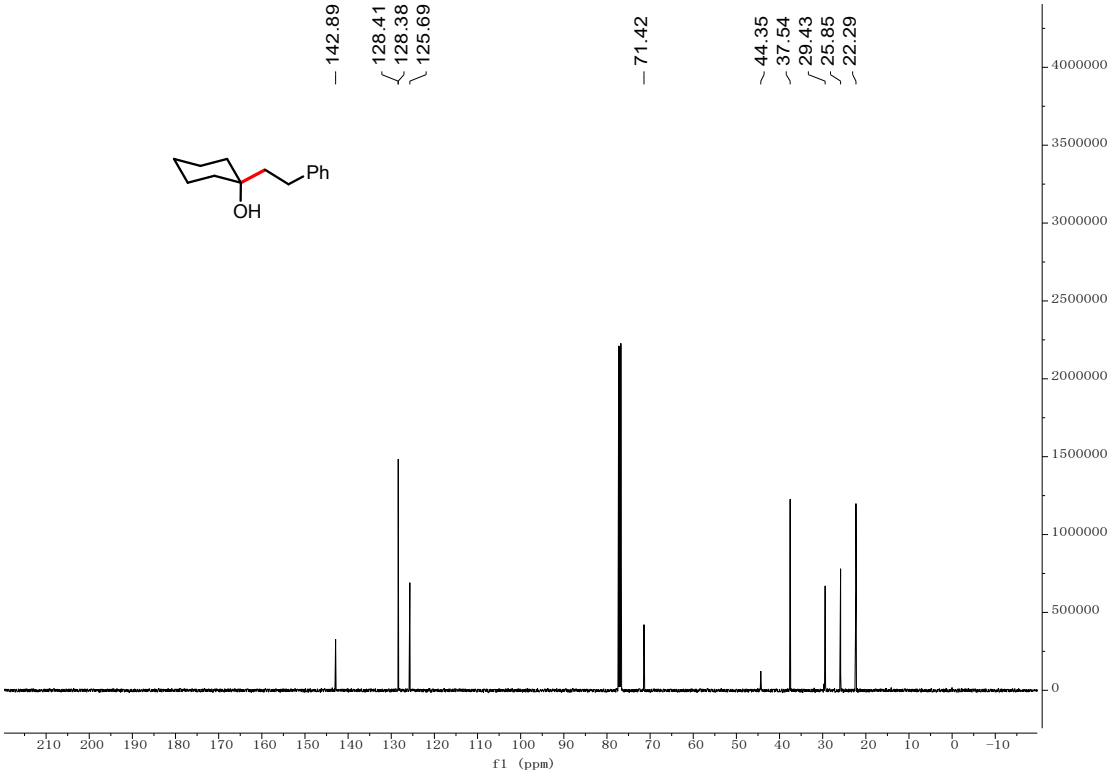
3aa ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



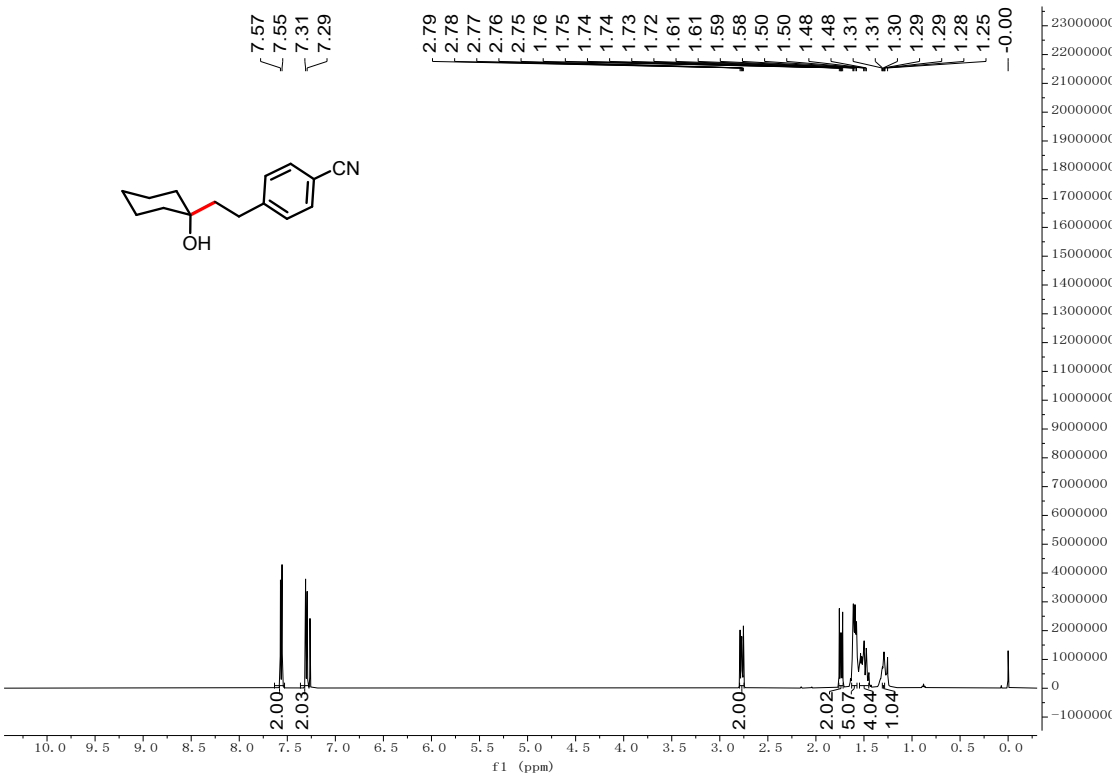
3ab (^{1}H NMR, 500 MHz, $CDCl_3$)



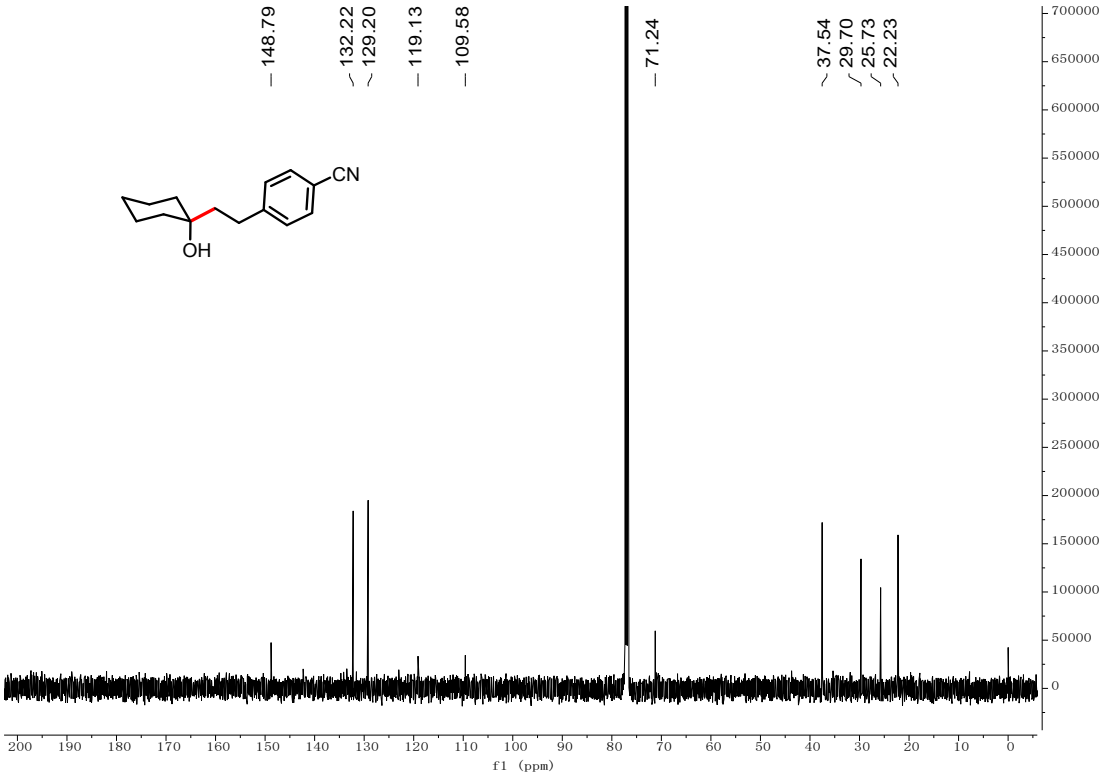
3ab ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



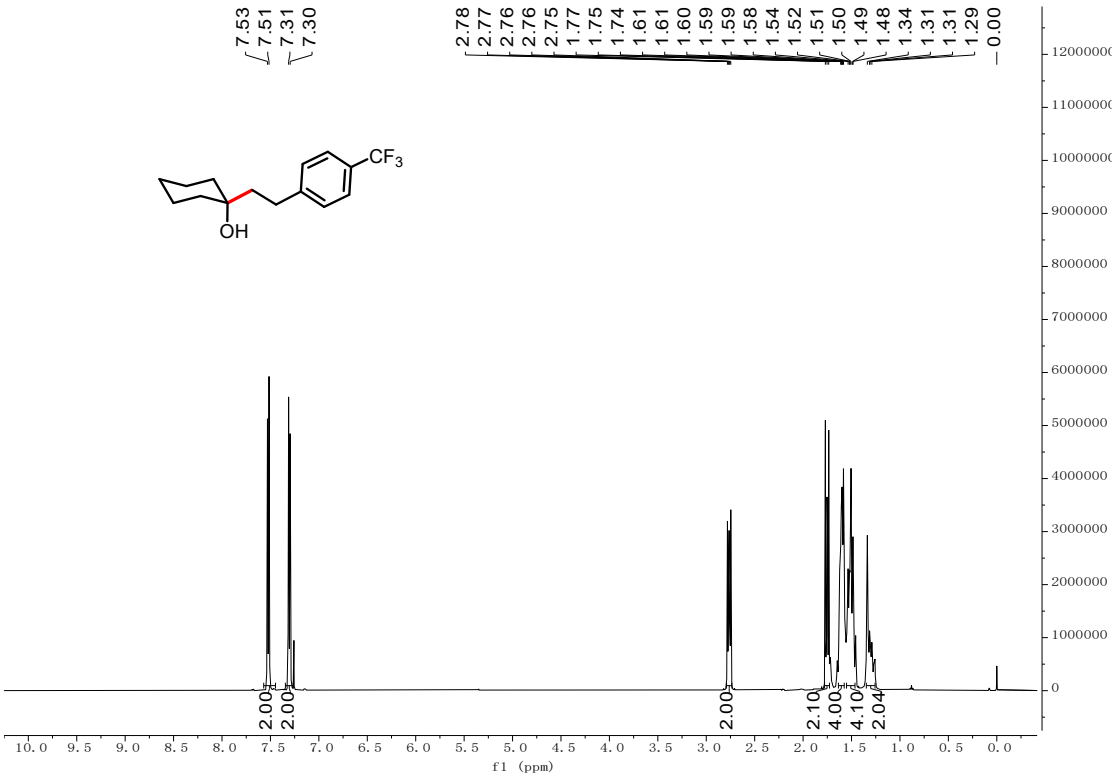
3ac (^{1}H NMR, 500 MHz, $CDCl_3$)



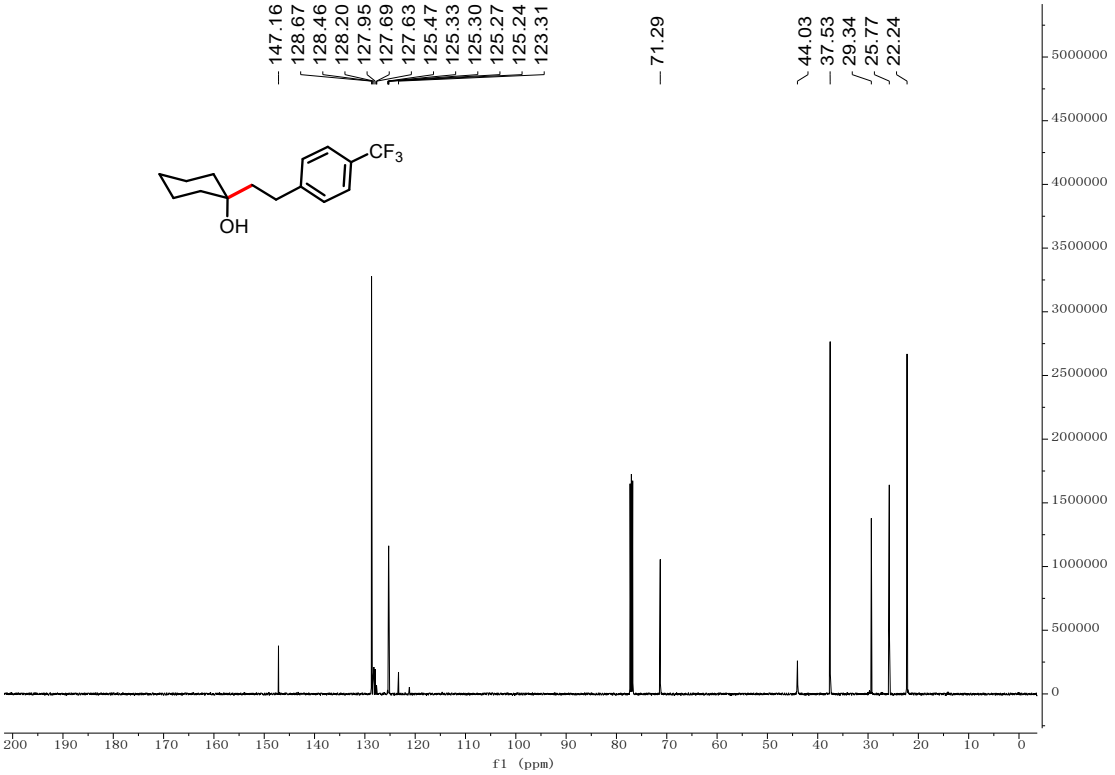
3ac ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



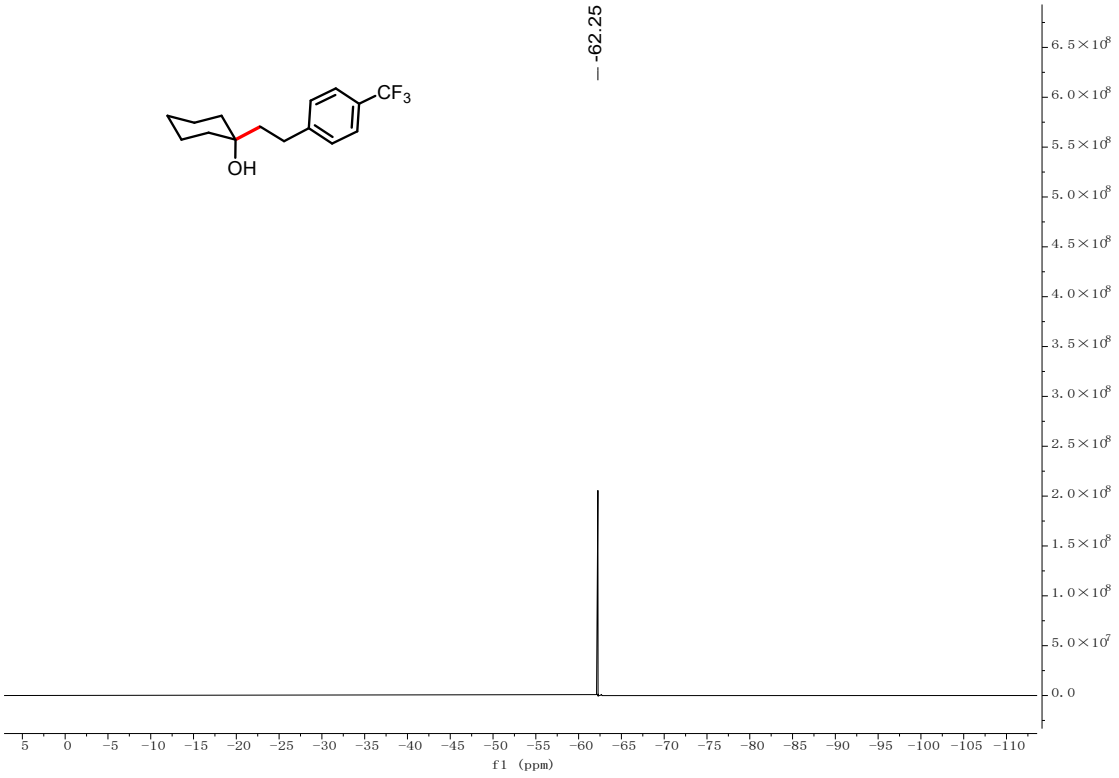
3ad (^{1}H NMR, 500 MHz, $CDCl_3$)



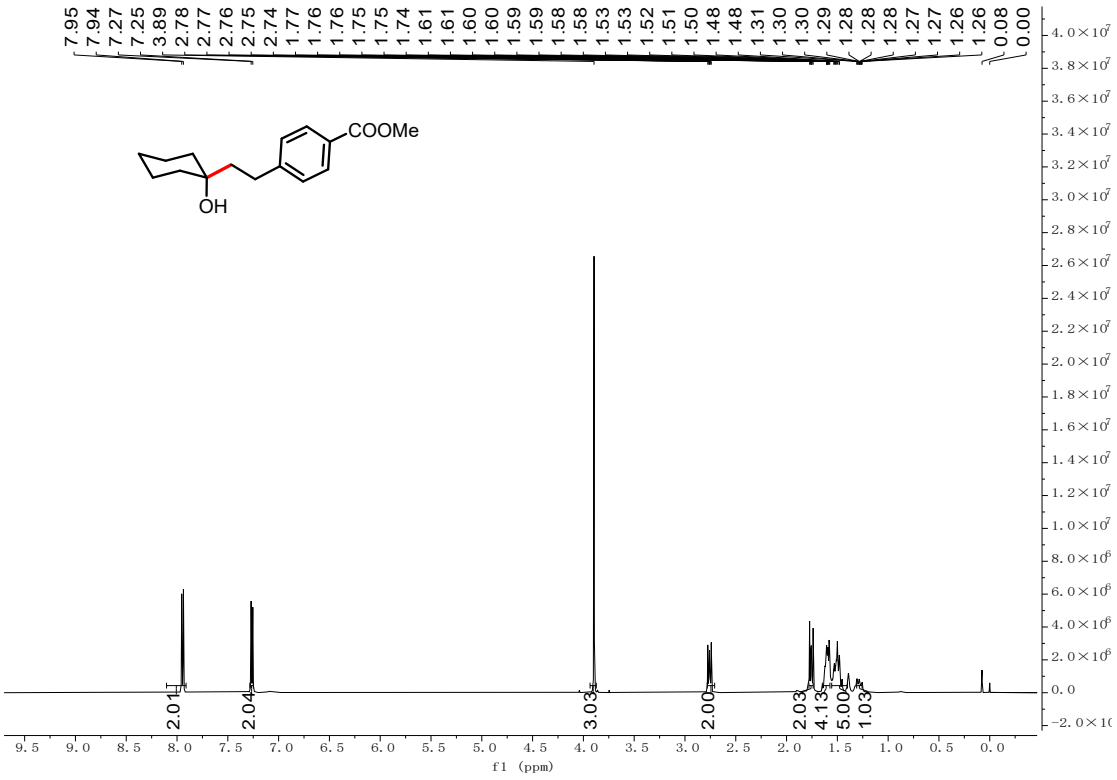
3ad ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



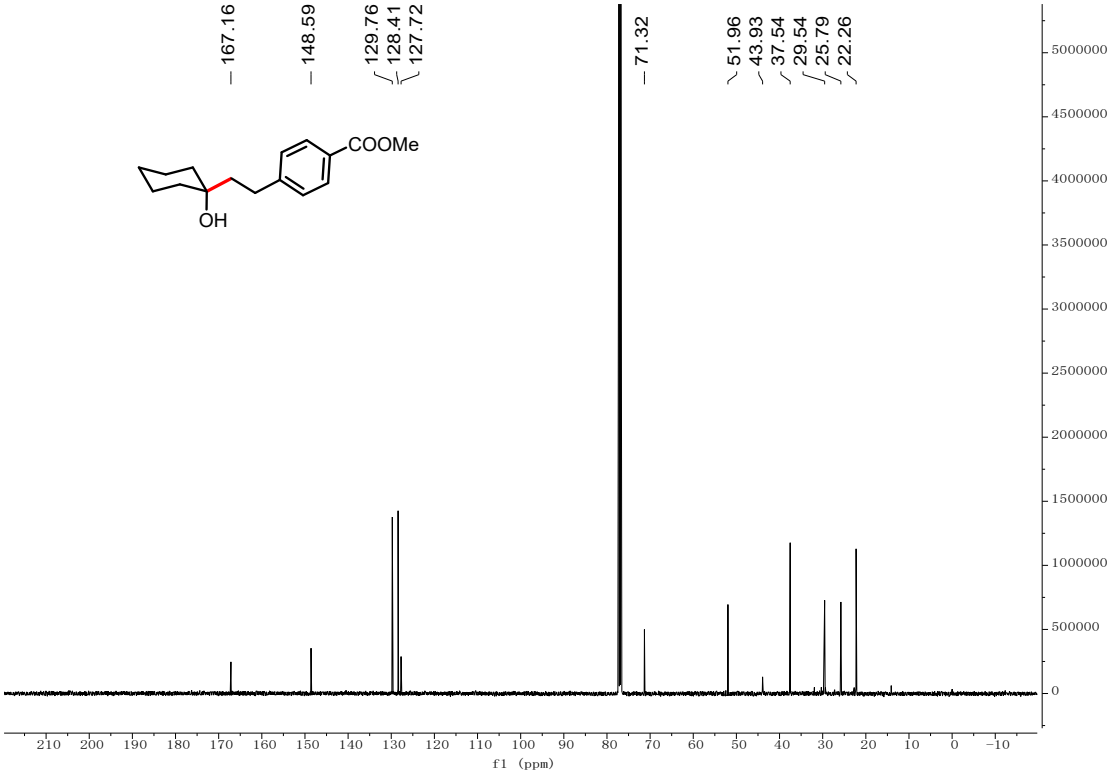
3ad (^{19}F NMR, 471 MHz, $CDCl_3$)



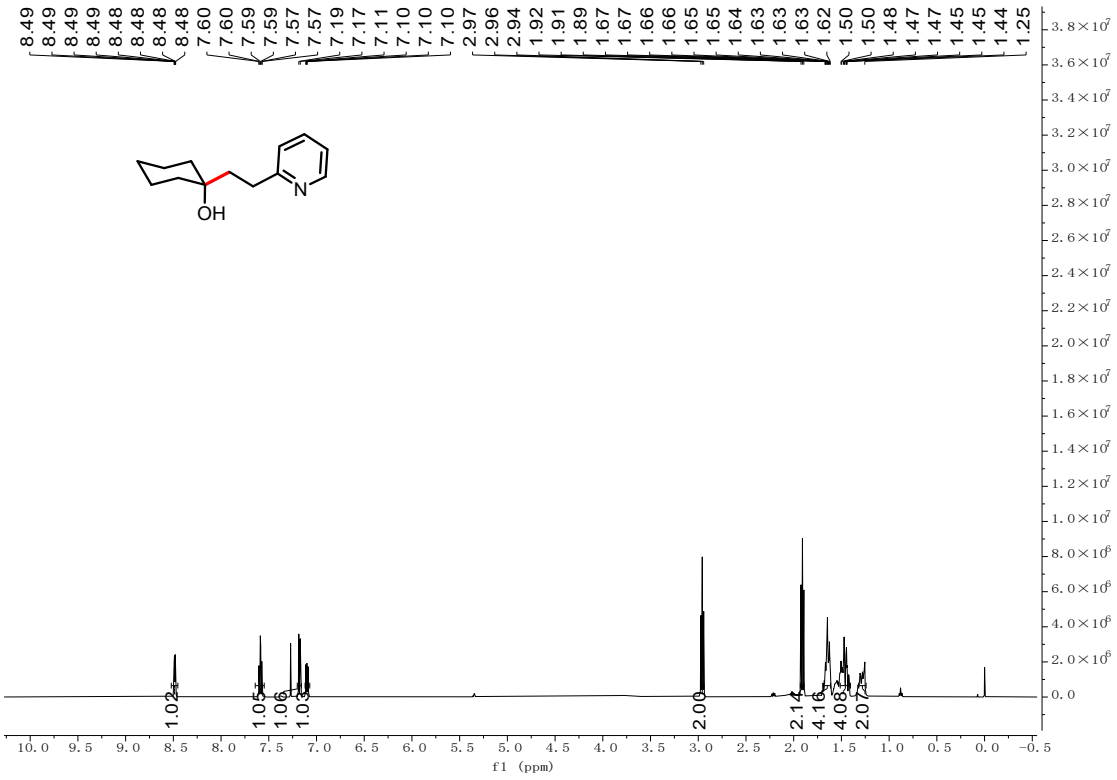
3ae (^{1}H NMR, 500 MHz, $CDCl_3$)



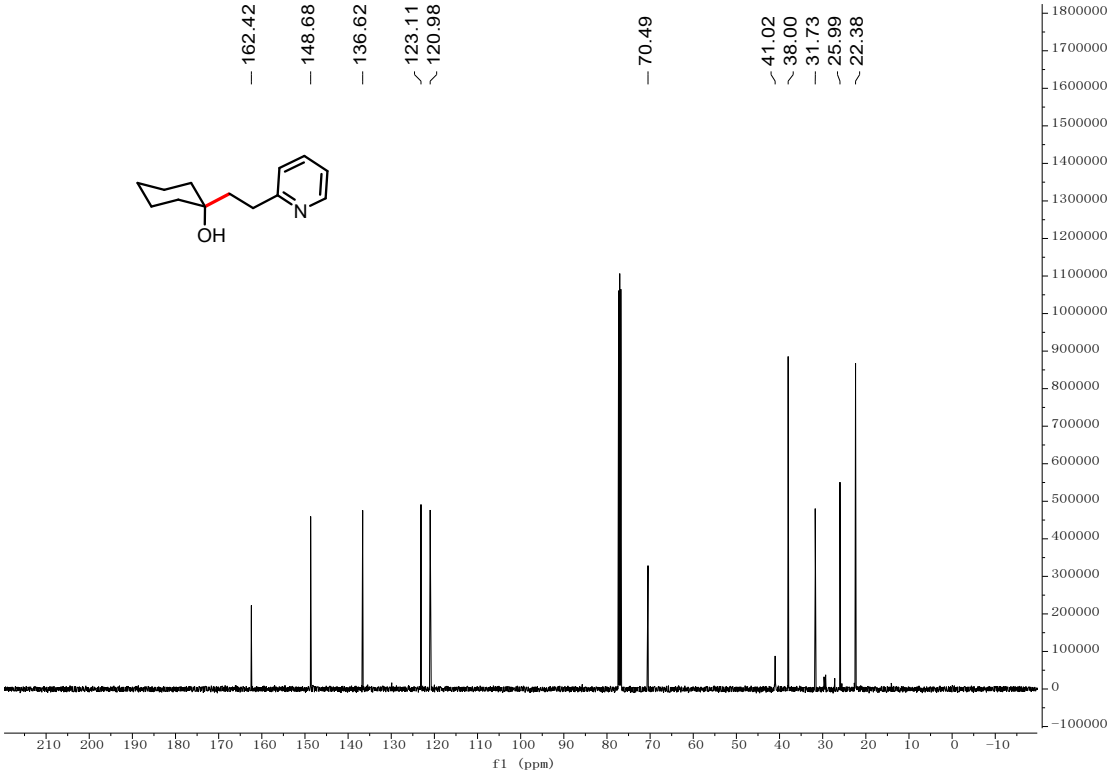
3ae ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



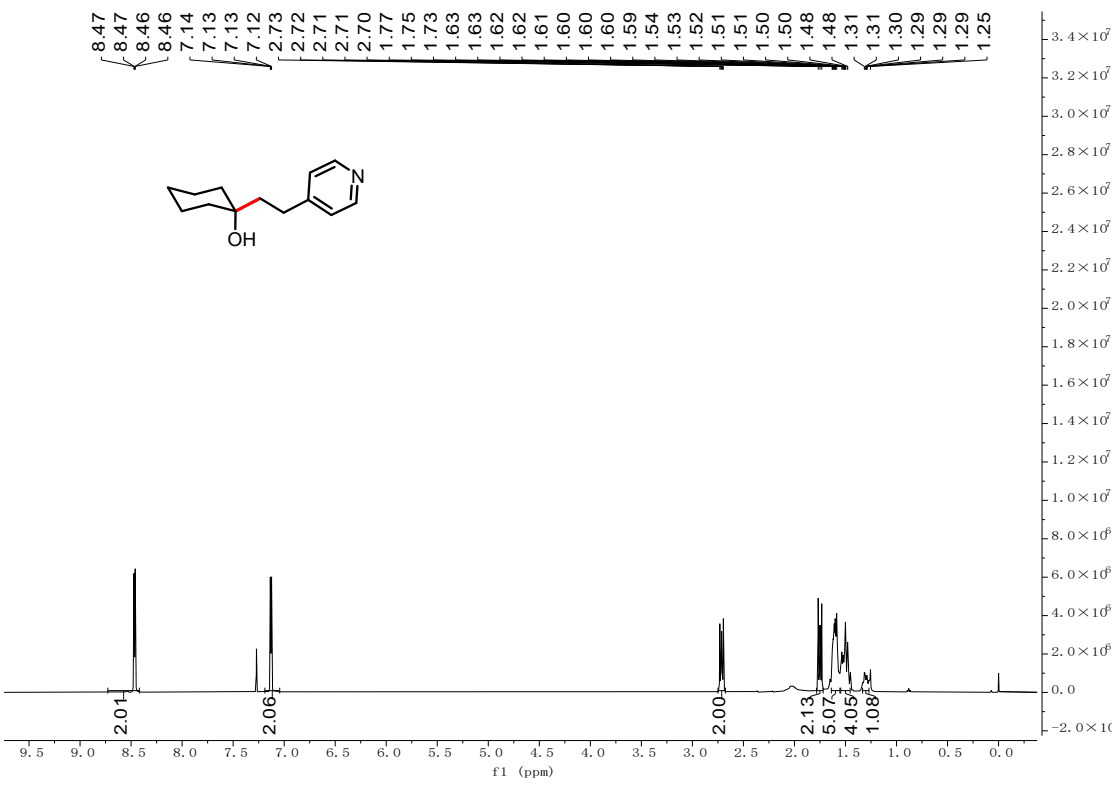
3af (^{1}H NMR, 500 MHz, $CDCl_3$)



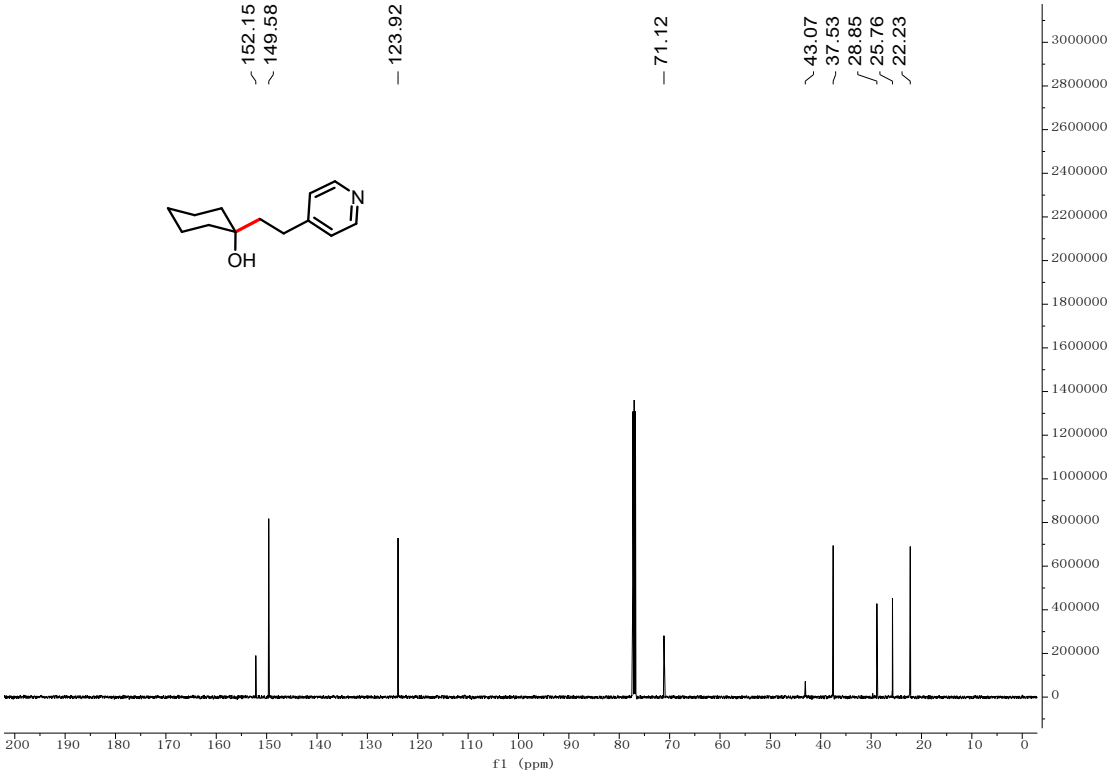
3af ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



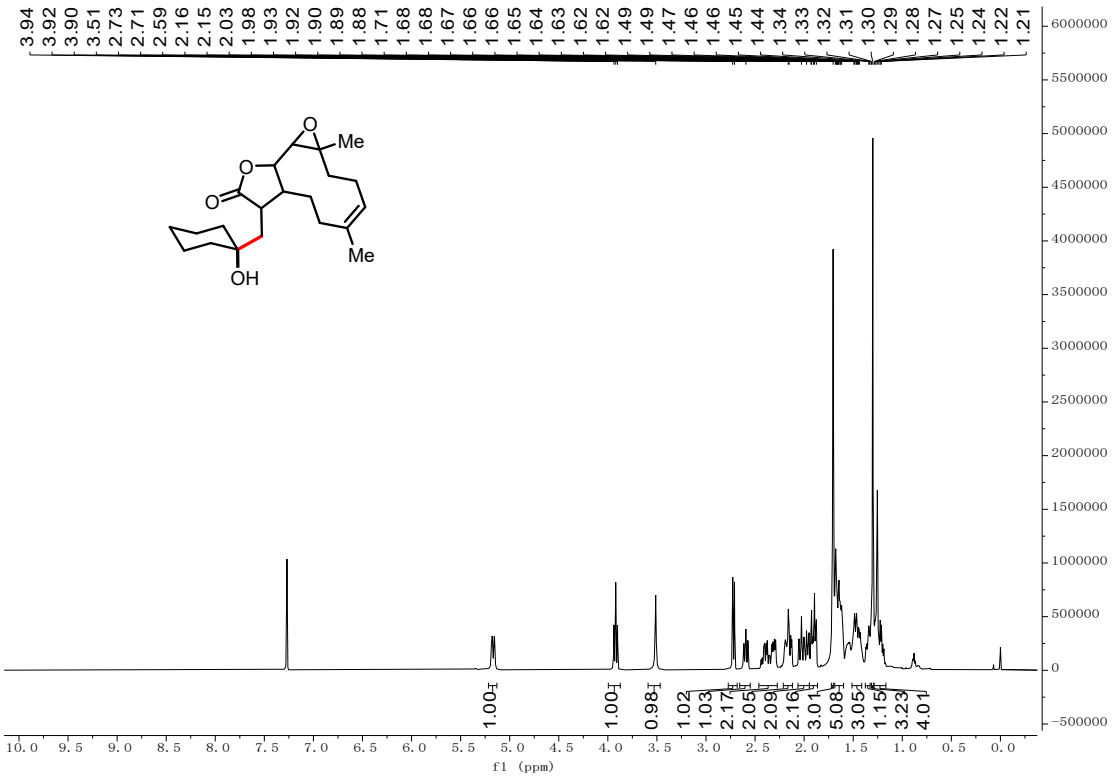
3ag (^{1}H NMR, 500 MHz, $CDCl_3$)



3ag ($^{13}C\{^{1}H\}$ NMR, 125 MHz, $CDCl_3$)



3ai (^{1}H NMR, 500 MHz, $CDCl_3$)



3ai (^{1}H NMR, 500 MHz, $CDCl_3$)