

EnT mediated alkoxy radical generation: the construction of 1,6-amino alcohols using bifunctional oxime esters

Zetian Sun, Xiaohua Du, Xiaoqing Li*, Xiangsheng Xu*

College of Chemical Engineering, Zhejiang University of Technology,
Hangzhou 310014, P. R. China.

*X. Li. E-mail: xqli@zjut.edu.cn. *X. Xu. E-mail: future@zjut.edu.cn

Supporting information

Table of contents

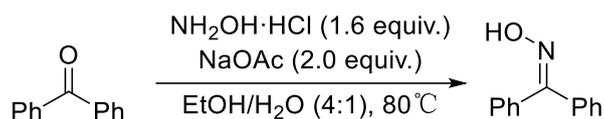
1. GENERAL INFORMATIONS.....	S2
2. METHODS FOR THE SYNTHESIS OF SUBSTRATES	S2
2.1 Preparation method of benzophenone oxime	S2
2.2 Preparation method of iminophenylacetic acids	S2
2.3 prepared method of bifunctional reagent.....	S4
2.4 Scope of bifunctional reagents	S5
3. EXPERIMENTAL SECTION.....	S13
3.1 Reaction set-up.....	S13
3.2 General produced of 1,6-oxyimination of alkenes	S13
3.3 Scale-up reaction.....	S13
3.4 Products derivatization.....	S14
4. MECHANISM STUDY.....	S16
4.1 Tempo trapping experiment	S16
4.2 Alkoxy radical cyclization experiment	S17
4.3 Blank experiment	S18
4.4 Direct excitation experiment	S19
4.5 Quantum yield calculations.....	S19
5. REFERENCE	S20
6. CHARACTERIZATION DATA.....	S21
7. NMR SPECTRUM	S34

1. General Informations

All commercial reagents were used without additional purification. Reactions were monitored by thin-layer chromatography (TLC) on commercial silica gel plates (GF 254) using UV light as a visualizing agent. Products were purified by flash chromatography on 200 – 300 mesh silica gels, SiO₂ was carried out with silica gel (200–300 mesh). ¹H NMR, ¹³C NMR and ¹⁹F NMR spectra were recorded with 400 MHz, 101 MHz and 377 MHz spectrometers in CDCl₃ by using tetramethylsilane (TMS) as the internal standard, respectively. High-resolution mass spectra (HRMS) were recorded using a positive-ion electrospray ionization (ESI⁺) source.

2. Methods for the synthesis of substrates

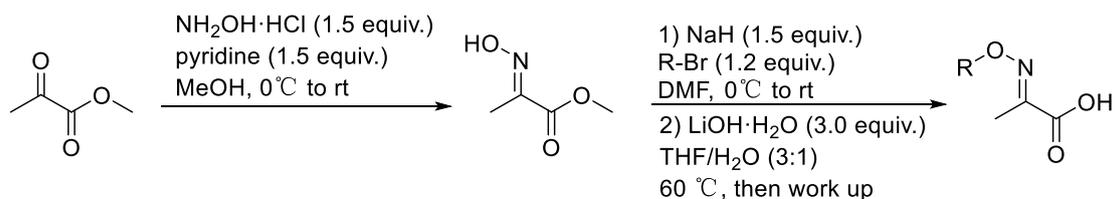
2.1 Preparation method of benzophenone oxime



In a 250 mL round bottom flask equipped with a condenser, aromatic ketones (50.0 mmol, 1.0 equiv.) were dissolved in the mixture of EtOH/H₂O (v/v, 4:1, 125 mL). Then, hydroxylamine hydrochloride (80.0 mmol, 1.6 equiv.) and NaOAc (100.0 mmol, 2.0 equiv.) were added in one portion. After the reaction mixture was refluxed in an oil bath at 80 °C overnight, the consumption of starting material was monitored by TLC. In order to remove as much ethanol as possible, the reaction was then cooled to room temperature and added 50 mL saturated NaHCO₃ carefully. Then extracted with ethyl acetate 3 times and then dried over anhydrous Na₂SO₄. Evaporation of the solvent afforded the product as a white solid in quantitative yield.

2.2 Preparation method of iminophenylacetic acids

General procedure A



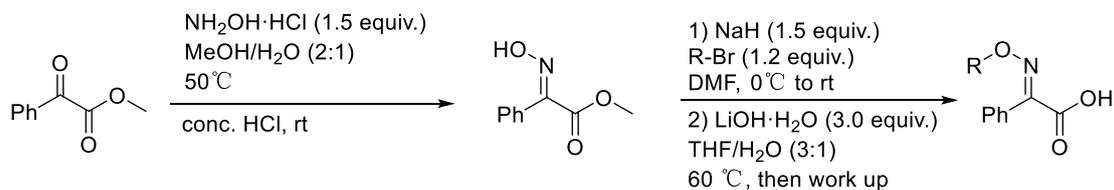
A flame-dried 100 mL round-bottom flask equipped with a magnetic stirring bar was charged with methyl pyruvate (20.0 mmol, 1.0 equiv.), hydroxylamine hydrochloride (30 mmol, 1.5 equiv.) and dehydrated MeOH (30 mL). After pyridine (30 mmol, 1.5 equiv.) was added to the flask at 0 °C,

the resulting mixture was stirred under nitrogen atmosphere at ambient temperature for overnight. After completion, the solvent was removed in vacuo with the aid of a rotary evaporator. The residue was transferred to a separatory funnel with DCM and 1N HCl solution was added. The phases were separated and aqueous layer was extracted with DCM. The organic phase was washed with brine, and then dried over Na₂SO₄, filtrated, concentrated in vacuo to afford the crude product which was further purified by recrystallization from DCM/PE to give the desired methyl (*E*)-2-(hydroxyimino)propanoate as a colourless solid.

The *E*-oxime ester (10 mmol, 1 equiv.) in 10 mL DMF was added to NaH (60% in oil, 15 mmol, 1.5 equiv.). The mixture was stirred at 0 °C for additional 30 min (insoluble yellow solid was formed). Then, haloalkanes (12 mmol, 1.2 equiv.) was slowly added and the mixture was stirred at room temperature overnight. The reaction was quenched with water. Then, the solvents were diluted with water and extracted with EtOAc, and the combined organic phases were washed with NaCl, dried over Na₂SO₄. After filtration and evaporation of the solvents in vacuo, the crude product was purified by column chromatography to afford the product.

Using THF and H₂O as cosolvent, LiOH·H₂O as base, stirred at 60 °C in an oil bath. The reaction was monitored by TLC. After acidification, the aqueous phase was extracted with EtOAc, and the combined organic phases were washed with NaCl and dried over Na₂SO₄. After filtration and evaporation of the solvents in vacuo to afford the target acids.

General procedure B

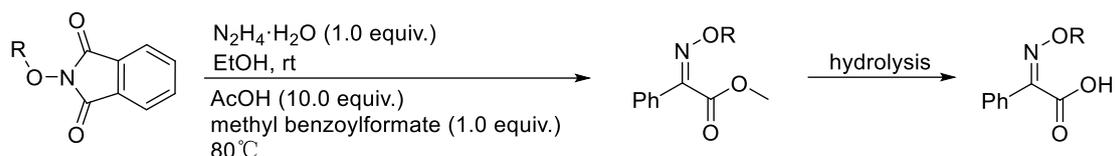


Following the literature procedure, to a stirred solution of methyl benzoylformate (14.8 g, 90.0 mmol) in methanol (45.0 mL) was added hydroxylamine hydrochloride (9.4 g, 135 mmol) in H₂O (22.5 mL) at 50 °C and the mixture was stirred for 3 h. Then, concentrated HCl (12N, 38.0 mL) was added dropwise at room temperature. After 8 h, resulting suspension was filtrated and collected precipitates were rinsed with cooled MeOH/H₂O (2/1). The precipitates were dried under reduced pressure to give as a white solid.

The *E*-oxime ester (10 mmol) in 10 mL DMF was added to NaH (60% in oil, 15 mmol). The mixture was stirred at 0 °C for additional 30 min (insoluble yellow solid was formed). Then, haloalkanes (12 mmol) was slowly added and the mixture was stirred at room temperature overnight. The reaction was quenched with water. Then, the solvents were diluted with water and extracted with EtOAc, and the combined organic phases were washed with NaCl, dried over Na₂SO₄. After filtration and evaporation of the solvents in vacuo, the crude product was purified by column chromatography to afford the product.

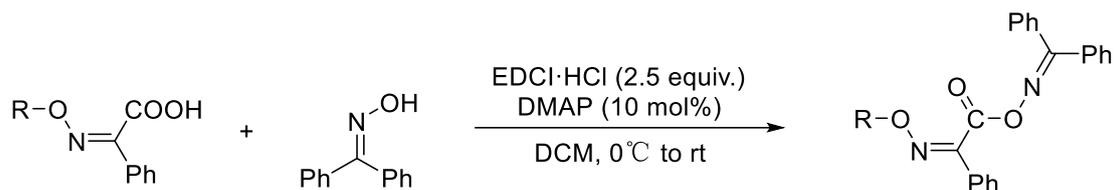
Using THF and H₂O as cosolvent, LiOH·H₂O as base, stirred at 60 °C in an oil bath. The reaction was monitored by TLC (in most cases for 3h). After acidification, the aqueous phase was extracted with EtOAc, and the combined organic phases were washed with NaCl and dried over Na₂SO₄. After filtration and evaporation of the solvents in vacuo to afford the target iminophenylacetic acids.

General procedure B



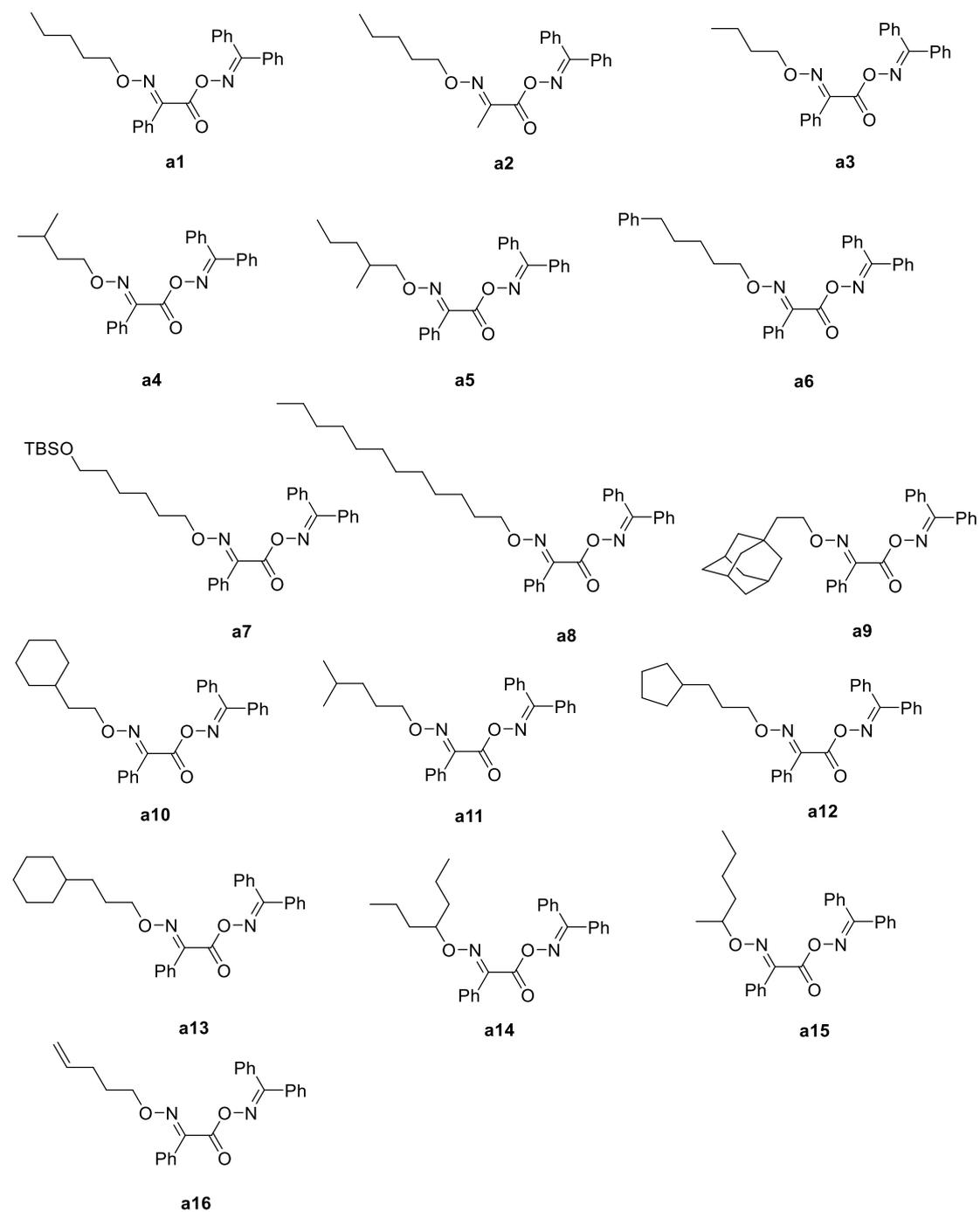
Following the literature procedure with slight modifications, the phthalimide derivative (1.00 equiv.) was dissolved in anhydrous ethanol (0.2 M) and hydrazine monohydrate (1.00 equiv.) was added in a single portion. The mixture was stirred at room temperature for 2 h with the formation of a white precipitate. The mixture was filtered and the filter pad was washed with ethanol. To the filtrate methyl benzoylformate (1.00 equiv.) and acetic acid (10.00 equiv.) were added. The mixture was stirred at 80°C overnight, quenched by saturated NaHCO₃ solution and concentrated by rotary evaporation. Then the mixture was extracted with EtOAc, and the combined organic phases were washed with NaCl, dried over Na₂SO₄. After evaporation of the solvents in vacuo, the crude product was purified by column chromatography to afford the product. The hydrolysis then afforded the iminophenylacetic acids.

2.3 prepared method of bifunctional reagent



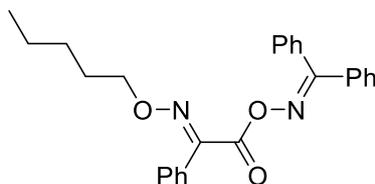
Benzophenone oxime (2.0 mmol) and aliphatic carboxylic acid (2.4 mmol) were dissolved in CH₂Cl₂ (15 mL). The reaction bath is lowered to 0 °C in a low-temperature stirring reaction bath. Then, EDCI·HCl (5 mmol) and DMAP (10 mol%, 0.2 mmol) was added sequentially. The mixture was stirred at room temperature under argon atmosphere until the reaction was complete as monitored by TLC analysis. The reaction mixture was diluted with distilled water (10 mL) and then the CH₂Cl₂ layer was separated. The resulting crude product was concentrated under reduced pressure and subsequently purified via column chromatography to yield the bifunctional reagents.

2.4 Scope of bifunctional reagents



Scheme S1 Scope of bifunctional reagents

Characterization data of substrates



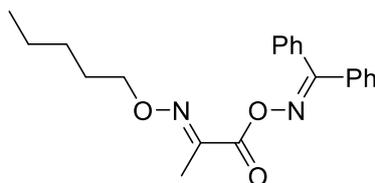
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-pentyl 1-oxime (a1)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((pentyloxy)imino)propanoic acid (prepared using General procedure B, 2.4 mmol) to afford as a white solid (597 mg, 72%).;

¹H NMR (400 MHz, CDCl₃) δ 7.76 – 7.59 (m, 2H), 7.55 – 7.20 (m, 13H), 4.21 (t, *J* = 6.8 Hz, 2H), 1.71 – 1.59 (m, 2H), 1.38 – 1.24 (m, 4H), 0.91 (t, *J* = 6.9 Hz, 3H).;

¹³C NMR (101 MHz, CDCl₃) δ 166.16, 161.05, 147.66, 134.60, 132.21, 131.05, 129.64, 129.37, 129.30, 129.23, 129.18, 129.07, 128.42, 127.99, 127.90, 76.46, 28.62, 27.87, 22.39, 14.02.;

HRMS (ESI) *m/z* calcd for C₂₆H₂₇N₂O₃⁺ (M+H)⁺ 415.2016, found 415.2014.



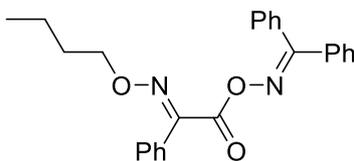
1-(((diphenylmethylene)amino)oxy)propane-1,2-dione O-pentyl 2-oxime (a2)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((pentyloxy)imino)propanoic acid (prepared using General procedure A, 2.4 mmol) to afford as a white solid (435 mg, 62%).

¹H NMR (400 MHz, CDCl₃) δ 7.69 – 7.58 (m, 2H), 7.57 – 7.36 (m, 8H), 4.18 (t, *J* = 6.7 Hz, 2H), 1.98 (s, 3H), 1.66 (t, *J* = 7.1 Hz, 2H), 1.35 – 1.31 (m, 3H), 0.92 (d, *J* = 7.0 Hz, 3H).;

¹³C NMR (101 MHz, CDCl₃) δ 166.02, 161.37, 147.19, 134.70, 132.35, 130.98, 129.78, 129.47, 129.31, 128.41, 127.98, 75.86, 28.71, 27.93, 22.45, 14.03, 11.17.

HRMS (ESI) *m/z* calcd for C₂₁H₂₅N₂O₃⁺ (M+H)⁺ 353.1860, found 353.1865.



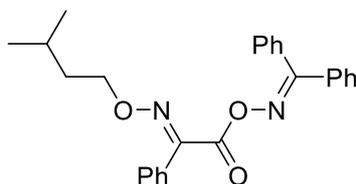
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-butyl 1-oxime (a3)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-(butoxyimino)-2-phenylacetic acid (prepared using General procedure B, 2.4 mmol) to afford as a white solid (638 mg, 80%).

¹H NMR (400 MHz, CDCl₃) δ 7.68 – 7.59 (m, 2H), 7.50 – 7.30 (m, 13H), 4.23 (t, *J* = 6.7 Hz, 2H), 1.66 (dq, *J* = 8.9, 6.8 Hz, 2H), 1.40 – 1.31 (m, 2H), 0.93 (t, *J* = 7.4 Hz, 3H).;

¹³C NMR (101 MHz, CDCl₃) δ 166.15, 161.06, 147.65, 134.61, 132.22, 131.05, 129.64, 129.38, 129.31, 129.23, 129.18, 129.08, 128.43, 127.99, 127.90, 76.18, 31.03, 18.99, 13.86.;

HRMS (ESI) *m/z* calcd for C₂₅H₂₅N₂O₃⁺ (M+H)⁺ 401.1860, found 401.1863.



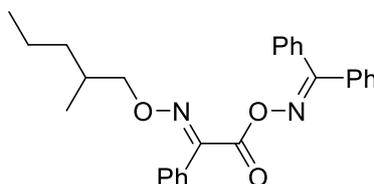
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-isopentyl 1-oxime (a4)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((isopentyl)oxy)imino)-2-phenylacetic acid (prepared using General procedure B, 2.4 mmol) to afford as a white solid (578 mg, 70%).

¹H NMR (400 MHz, CDCl₃) δ 7.63 (dd, *J* = 7.7, 2.1 Hz, 2H), 7.49 – 7.22 (m, 13H), 4.26 (t, *J* = 6.9 Hz, 2H), 1.70 – 1.51 (m, 3H), 0.91 (d, *J* = 6.5 Hz, 6H).;

¹³C NMR (101 MHz, CDCl₃) δ 166.14, 161.04, 147.66, 134.62, 132.22, 131.04, 129.65, 129.37, 129.30, 129.22, 129.19, 129.08, 128.42, 128.00, 127.89, 75.06, 37.62, 25.05, 22.60.;

HRMS (ESI) *m/z* calcd for C₂₆H₂₇N₂O₃⁺ (M+H)⁺ 415.2016, found 415.2012.



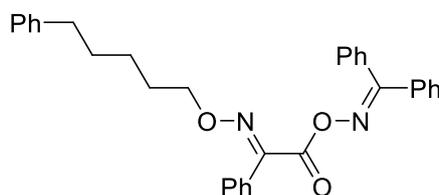
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-(2-methylpentyl) 1-oxime 9 (a5)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-(((2-methylpentyl)oxy)imino)-2-phenylacetic acid (prepared using General procedure C, 2.4 mmol) to afford as a white solid (508 mg, 59%).

¹H NMR (400 MHz, CDCl₃) δ 7.71 – 7.58 (m, 2H), 7.58 – 7.21 (m, 13H), 4.23 – 3.91 (m, 2H), 1.96 – 1.75 (m, 1H), 1.40 – 1.04 (m, 4H), 0.96 – 0.74 (m, 6H).;

¹³C NMR (101 MHz, CDCl₃) δ 166.15, 161.06, 147.65, 134.62, 132.22, 131.04, 129.63, 129.35, 129.30, 129.22, 129.03, 128.42, 127.99, 127.88, 81.66, 35.51, 32.56, 19.87, 16.72, 14.33.

HRMS (ESI) *m/z* calcd for C₂₇H₂₉N₂O₃⁺ (M+H)⁺ 429.2173, found 429.2168.



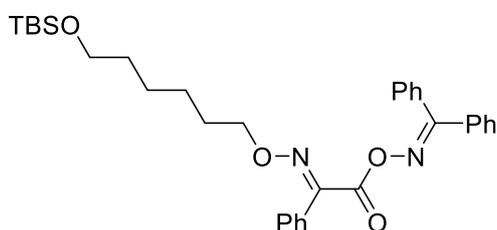
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-(5-phenylpentyl) 1-oxime (a6)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-phenyl-2-(((5-phenylpentyl)oxy)imino) acetic acid (prepared using General procedure B, 2.4 mmol) to afford as a white solid (726 mg, 74%).

¹H NMR (400 MHz, CDCl₃) δ 7.70 – 7.60 (m, 2H), 7.55 – 7.47 (m, 1H), 7.46 – 7.27 (m, 14H), 7.20 (dd, *J* = 7.0, 5.3 Hz, 3H), 4.22 (t, *J* = 6.7 Hz, 2H), 2.63 (t, *J* = 7.7 Hz, 2H), 1.68 (dp, *J* = 23.6, 7.7, 7.3 Hz, 4H), 1.44 – 1.30 (m, 2H).;

¹³C NMR (101 MHz, CDCl₃) δ 166.16, 161.01, 147.77, 142.45, 134.61, 132.22, 131.08, 129.66, 129.40, 129.32, 129.24, 129.16, 129.07, 128.45, 128.43, 128.32, 128.00, 127.93, 125.73, 76.29, 35.80, 31.11, 28.81, 25.36.;

HRMS (ESI) *m/z* calcd for C₃₂H₃₁N₂O₃⁺ (M+H)⁺ 491.2329, found 491.2326.



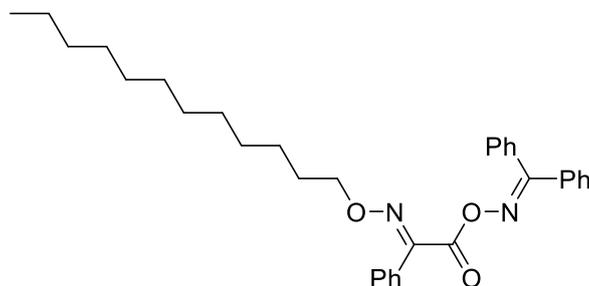
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-(6-((tert-butyldimethylsilyl)oxy)hexyl) 1-oxime (a7)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 12,12,13,13-tetramethyl-2-phenyl-4,11-dioxo-3-aza-12-silatetradec-2-enoic acid (prepared using General procedure B, 2.4 mmol) to afford as a colorless oil (717 mg, 64%).;

¹H NMR (400 MHz, CDCl₃) δ 7.69 – 7.60 (m, 2H), 7.50 – 7.34 (m, 11H), 7.30 (dq, *J* = 9.5, 2.1 Hz, 2H), 4.22 (t, *J* = 6.8 Hz, 2H), 3.62 (t, *J* = 6.5 Hz, 2H), 1.72 – 1.64 (m, 2H), 1.52 (q, *J* = 6.7, 5.5 Hz, 2H), 1.34 (dt, *J* = 7.1, 3.0 Hz, 4H), 0.92 (s, 9H), 0.08 (s, 6H).;

¹³C NMR (101 MHz, CDCl₃) δ 166.15, 161.01, 147.71, 134.60, 132.22, 131.04, 129.64, 129.38, 129.30, 129.22, 129.07, 128.42, 127.99, 127.90, 76.37, 63.13, 32.77, 28.95, 26.01, 25.58, 18.39, -5.23.;

HRMS (ESI) *m/z* calcd for C₃₃H₄₃N₂O₄Si⁺ (M+H)⁺ 559.2987, found 559.2990.



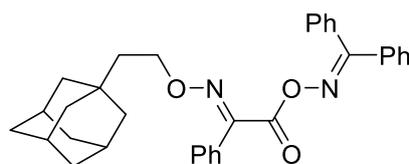
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-dodecyl 1-oxime (a8)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((dodecyloxy)imino)-2-phenylacetic acid (prepared using General procedure B, 2.4 mmol) to afford as a yellow oil (799 mg, 78%);

¹H NMR (400 MHz, CDCl₃) δ 7.68 – 7.57 (m, 2H), 7.50 – 7.26 (m, 13H), 4.21 (t, *J* = 6.8 Hz, 2H), 1.73 – 1.63 (m, 2H), 1.30 (d, *J* = 6.0 Hz, 18H), 0.90 (d, *J* = 7.0 Hz, 3H);

¹³C NMR (101 MHz, CDCl₃) δ 166.13, 161.03, 147.66, 134.62, 132.22, 131.04, 129.63, 129.37, 129.31, 129.25, 129.19, 129.08, 128.42, 127.98, 127.89, 76.48, 31.94, 29.68, 29.67, 29.60, 29.56, 29.38, 29.34, 28.93, 25.73, 22.72, 14.15.;

HRMS (ESI) *m/z* calcd for C₃₃H₄₁N₂O₃⁺ (*M*+*H*)⁺ 513.3112, found 513.3112.



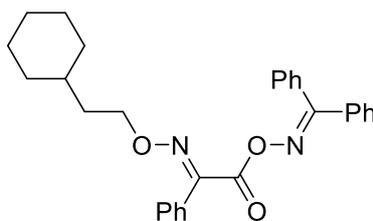
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-(2-((3r,5r,7r)-adamantan-1-yl)ethyl) 1-oxime (a9)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((2-((3r,5r,7r)-adamantan-1-yl)ethoxy)imino)-2-phenylacetic acid (prepared using General procedure B, 2.4 mmol) to afford as a white solid (564 mg, 56%);

¹H NMR (400 MHz, CDCl₃) δ 7.67 – 7.59 (m, 2H), 7.52 – 7.27 (m, 13H), 4.29 (t, *J* = 7.4 Hz, 2H), 1.94 (q, *J* = 3.3 Hz, 3H), 1.76 – 1.58 (m, 7H), 1.50 – 1.43 (m, 7H);

¹³C NMR (101 MHz, CDCl₃) δ 166.12, 161.03, 147.65, 134.62, 132.22, 131.03, 129.65, 129.30, 129.27, 129.21, 129.06, 128.42, 128.02, 127.86, 72.83, 42.60, 37.03, 31.71, 28.64.;

HRMS (ESI) *m/z* calcd for C₃₃H₃₅N₂O₃⁺ (*M*+*H*)⁺ 507.2642, found 507.2644.



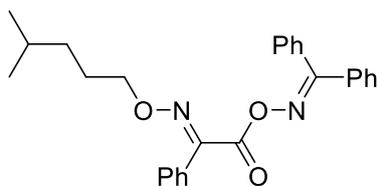
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-(2-cyclohexylethyl) 1-oxime (a10)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((2-cyclohexylethoxy)imino)-2-phenylacetic acid (prepared using General procedure B, 2.4 mmol) to afford as a white solid (636 mg, 70%).

¹H NMR (400 MHz, CDCl₃) δ 7.63 (dd, *J* = 7.1, 1.6 Hz, 2H), 7.52 – 7.28 (m, 13H), 4.26 (t, *J* = 7.0 Hz, 2H), 1.69 – 1.20 (m, 11H), 0.98 – 0.86 (m, 2H);

¹³C NMR (101 MHz, CDCl₃) δ 166.13, 161.03, 147.69, 134.62, 132.21, 131.04, 129.64, 129.35, 129.30, 129.22, 129.07, 128.42, 127.99, 127.88, 74.71, 36.19, 34.61, 33.24, 26.50, 26.25.

HRMS (ESI) *m/z* calcd for C₂₉H₃₁N₂O₃⁺ (*M*+*H*)⁺ 455.2329, found 455.2339.



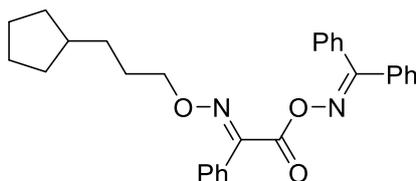
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-(4-methylpentyl) 1-oxime (a11)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-(((4-methylpentyl)oxy)imino)-2-phenylacetic acid (prepared using General procedure B, 2.4 mmol) to afford as a white solid (633 mg, 74%).

¹H NMR (400 MHz, CDCl₃) δ 7.62 (dd, *J* = 9.5, 7.9 Hz, 2H), 7.48 – 7.30 (m, 13H), 4.20 (t, *J* = 6.9 Hz, 2H), 1.72 – 1.61 (m, 2H), 1.61 – 1.49 (m, 1H), 1.20 (dd, *J* = 15.8, 7.0 Hz, 2H), 0.89 (d, *J* = 6.6 Hz, 6H);

¹³C NMR (101 MHz, CDCl₃) δ 166.15, 161.03, 147.69, 134.61, 132.20, 131.05, 129.65, 129.38, 129.31, 129.24, 129.08, 128.42, 127.99, 127.90, 76.71, 34.77, 27.76, 26.83, 22.55.;

HRMS (ESI) *m/z* calcd for C₂₇H₂₉N₂O₃⁺ (M+H)⁺ 429.2173, found 429.2170.



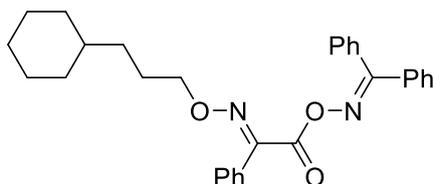
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-(3-cyclopentylpropyl) 1-oxime (a12)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((3-cyclopentylpropoxy)imino)-2-phenylacetic acid (prepared using General procedure B, 2.4 mmol) to afford as a white solid (537 mg, 59%).

¹H NMR (400 MHz, CDCl₃) δ 7.68 – 7.59 (m, 2H), 7.56 – 7.29 (m, 13H), 4.21 (t, *J* = 6.8 Hz, 2H), 1.84 – 1.46 (m, 10H), 1.39 – 1.25 (m, 2H), 1.08 (qd, *J* = 9.2, 8.2, 3.9 Hz, 2H);

¹³C NMR (101 MHz, CDCl₃) δ 166.14, 161.03, 147.66, 134.61, 132.20, 131.05, 129.64, 129.37, 129.31, 129.24, 129.19, 129.07, 128.42, 127.98, 127.90, 76.70, 39.83, 32.68, 32.09, 28.17, 25.17.

HRMS (ESI) *m/z* calcd for C₂₉H₃₁N₂O₃⁺ (M+H)⁺ 455.2329, found 455.2326.



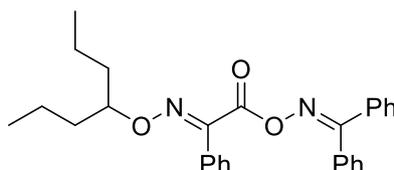
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-(3-cyclohexylpropyl) 1-oxime (a13)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((3-cyclohexylpropoxy)imino)-2-phenylacetic acid (prepared using General procedure B, 2.4 mmol) to afford as white solid (559 mg, 60%).

¹H NMR (400 MHz, CDCl₃) δ 7.69 – 7.31 (m, 15H), 4.29 (t, *J* = 6.9 Hz, 2H), 1.75 – 1.58 (m, 8H), 1.41 – 0.97 (m, 7H).;

¹³C NMR (101 MHz, CDCl₃) δ 165.83, 149.30, 134.80, 131.89, 131.15, 130.37, 130.17, 129.97, 129.19, 128.74, 128.45, 128.20, 126.24, 73.71, 36.51, 34.42, 33.30, 26.54, 26.22.;

HRMS (ESI) *m/z* calcd for C₃₀H₃₃N₂O₃⁺ (M+H)⁺ 469.2486, found 469.2488.



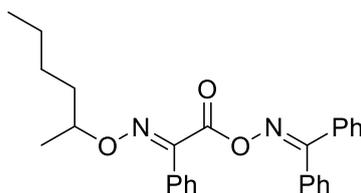
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-heptan-4-yl 1-oxime (a14)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((heptan-4-yloxy)imino)-2-phenylacetic acid (prepared using General procedure C, 2.4 mmol) to afford as a yellow oil (584 mg, 66%).;

¹H NMR (400 MHz, CDCl₃) δ 7.68 – 7.37 (m, 15H), 4.26 (dq, *J* = 7.5, 4.9 Hz, 1H), 1.72 – 1.64 (m, 2H), 1.56 – 1.33 (m, 6H), 0.88 (t, *J* = 7.3 Hz, 6H).;

¹³C NMR (101 MHz, CDCl₃) δ 165.35, 148.63, 134.51, 131.93, 131.06, 130.73, 129.95, 129.88, 129.13, 128.70, 128.41, 128.22, 126.15, 84.76, 36.10, 18.58, 14.02.;

HRMS (ESI) *m/z* calcd for C₂₈H₃₁N₂O₃⁺ (M+H)⁺ 443.2329, found 443.2324.



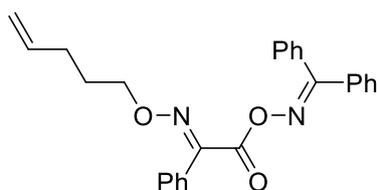
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-hexan-2-yl 1-oxime (a15)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((hexan-2-yloxy)imino)-2-phenylacetic acid (prepared using General procedure C, 2.4 mmol) to afford as a yellow oil (724 mg, 85%).;

¹H NMR (400 MHz, CDCl₃) δ 7.67 – 7.51 (m, 4H), 7.43 – 7.32 (m, 11H), 4.35 (h, *J* = 6.3 Hz, 1H), 1.66 – 1.24 (m, 9H), 0.86 (t, *J* = 6.9 Hz, 3H).;

¹³C NMR (101 MHz, CDCl₃) δ 165.58, 148.83, 137.26, 134.51, 133.86, 133.66, 131.93, 131.09, 130.66, 130.02, 129.91, 129.20, 129.17, 128.74, 128.70, 128.55, 128.48, 128.43, 128.20, 126.19, 81.27, 35.31, 27.56, 22.70, 19.84, 14.02.;

HRMS (ESI) *m/z* calcd for C₂₇H₂₉N₂O₃⁺ (M+H)⁺ 429.2173, found 429.2177.



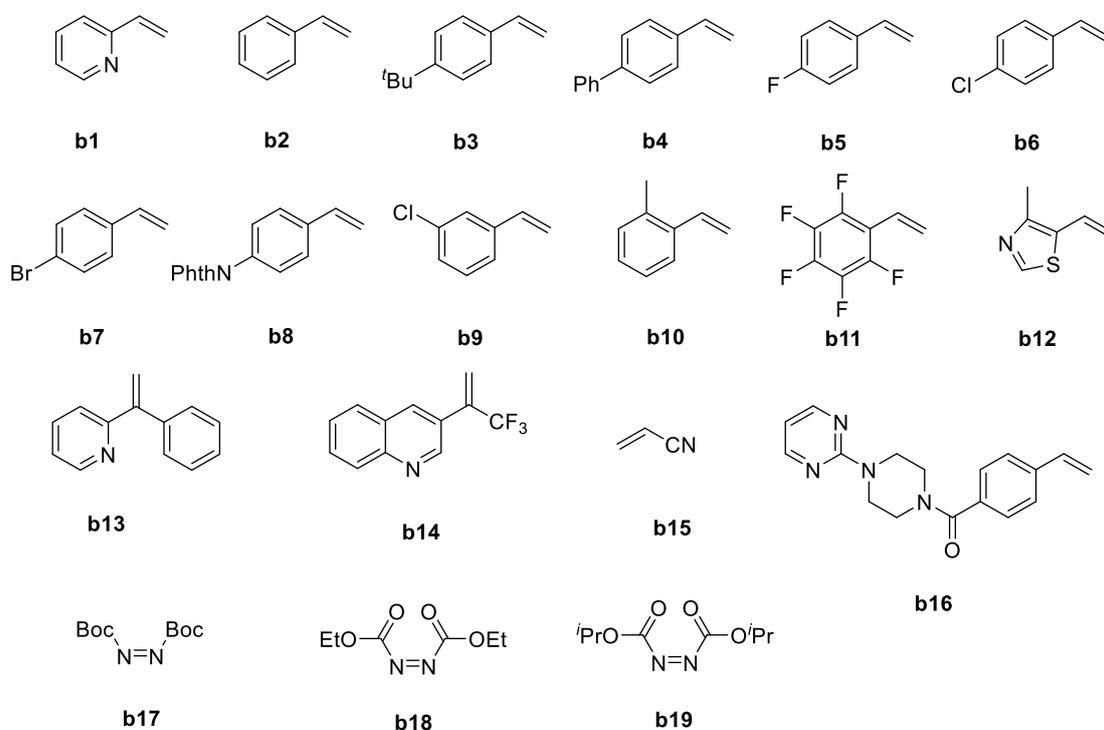
1-(((diphenylmethylene)amino)oxy)-2-phenylethane-1,2-dione O-pent-4-en-1-yl 1-oxime (**a16**)

Synthesized by following General Procedure using diphenylmethanone oxime (2.0 mmol) and 2-((pent-4-en-1-yloxy)imino)-2-phenylacetic acid (prepared using General procedure B, 2.4 mmol) to afford as a white solid (617 mg, 75%).;

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.68 – 7.59 (m, 2H), 7.53 – 7.28 (m, 13H), 5.87 – 5.70 (m, 1H), 5.08 – 4.93 (m, 2H), 4.23 (t, $J = 6.7$ Hz, 2H), 2.09 (qd, $J = 8.3, 1.5$ Hz, 2H), 1.84 – 1.74 (m, 2H).;

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 166.19, 160.98, 147.87, 137.71, 134.59, 132.21, 131.06, 129.65, 129.42, 129.30, 129.21, 129.13, 129.06, 128.43, 128.00, 127.91, 115.14, 75.61, 29.89, 28.14.;

HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{25}\text{N}_2\text{O}_3^+$ ($\text{M}+\text{H}$) $^+$ 413.1860, found 413.1860.



Scheme S2 Scope of alkenes. Alkenes **b1-b18** were commercially available and **b16** was prepared using reported method².

3. Experimental section

3.1 Reaction set-up

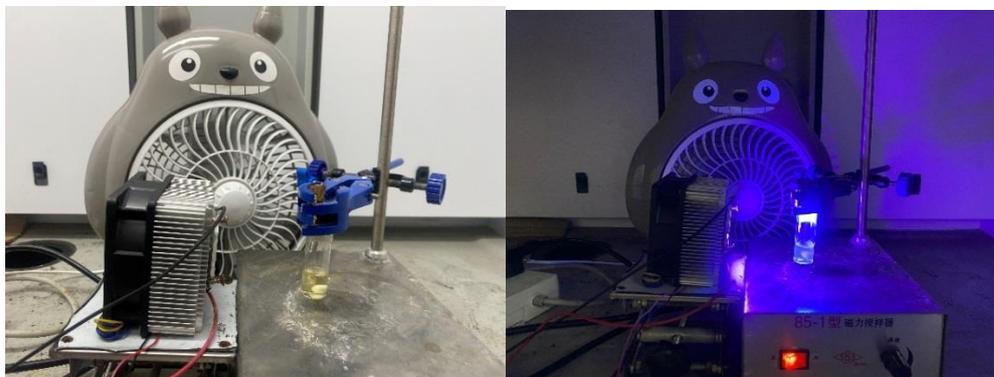
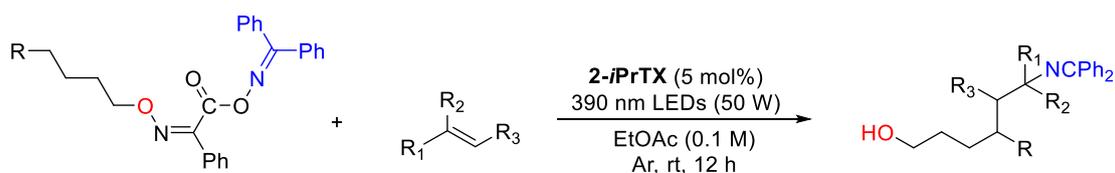


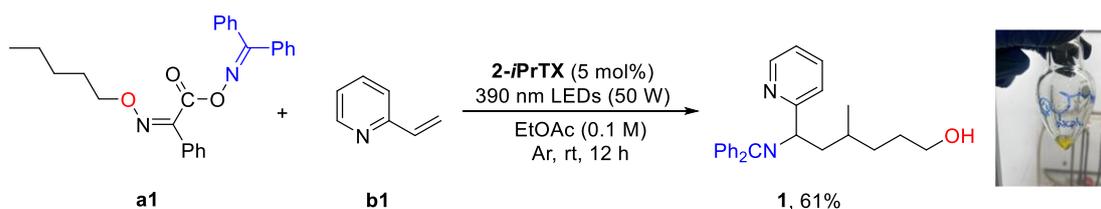
Figure 1 Reaction set-up

3.2 General product of 1,6-oxyimination of alkenes



An oven dried 8 mL reaction vial was charged with a stir bar, bifunctional reagent (0.3 mmol, 1.5 equiv.), and 2-*i*PrTX photosensitizer (2.5 mg, 5 mol %) were charged under air. The reaction vial was sealed, evacuated and backfilled three times with Ar. Then under Ar atmosphere, added of EtOAc (2.0 mL, 0.1 M) and alkenes (0.2 mmol, 1.0 equiv.). The reaction mixture was stirred and irradiated using a 50 W 395 nm LED lamp for 12 h until the reaction was completed. After irradiation, the resulting homogenous solution was transferred to a 25 mL round bottom flask with aid of EtOAc (2 x 3 mL). NEt₃ (approx. 0.5 mL) and SiO₂ were added to this solution and the volatiles were removed under reduced pressure, affording a powder which was loaded on column. Purification by flash column chromatography on SiO₂, pre-basified with NEt₃ using pentane: EtOAc mixtures afforded the corresponding products.

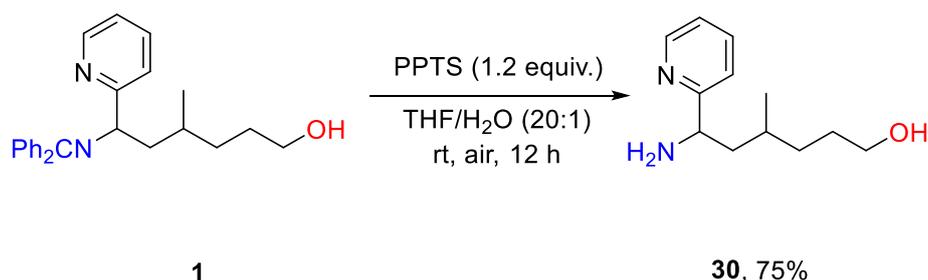
3.3 Scale-up reaction



An oven dried 50 mL Schlenk tube was charged with a stir bar, bifunctional reagent **a1** (3 mmol, 1.5 equiv.), and 2-*i*PrTX (25.4 mg, 5 mol %) were charged under air. The reaction vial was

sealed, evacuated and backfilled three times with Ar. Then under Ar atmosphere, added of EtOAc (20 mL, 0.1 M) and 2-Vinylpyridine **b1** (2 mmol, 1.0 equiv.). The reaction mixture was stirred and irradiated using a 50 W 395 nm LED lamp for 12 h until the reaction was completed. After irradiation, the resulting homogenous solution was transferred to a 50 mL round bottom flask with aid of EtOAc (2 x 5 mL). NEt₃ (approx. 3 mL) and SiO₂ were added to this solution and the volatiles were removed under reduced pressure, affording a powder which was loaded on column. Purification by flash column chromatography on SiO₂, pre-basified with NEt₃ using pentane: EtOAc mixtures afforded the corresponding product **1** as a yellow oil (452.1 mg, 61% yield).

3.4 Products derivatization

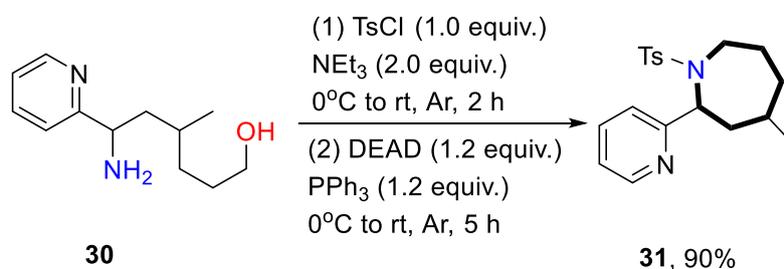


A 25 mL vial was charged with compound **1** (372.2 mg, 1.0 mmol), THF (18.5 mL) and H₂O (1.5 mL) were added. Pyridinium p-toluene sulfonate (1.2 mmol, 1.2 equiv.) was added to the vial. The reaction was stirred at room temperature for 12 h. The reaction was then diluted with EtOAc (20.0 mL) and H₂O (8.0 mL). The organic layer was separated, and the aqueous layer was extracted with EtOAc (10.0 mL x 2). The combined organic phases were dried (Na₂SO₄), filtered, and evaporated. Crude mixture was purified using column chromatography to give **30** as a colorless oil (156.5 mg, 75% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.45 (d, *J* = 4.6 Hz, 1H), 7.64 – 7.50 (m, 1H), 7.24 – 6.97 (m, 2H), 3.99 (q, *J* = 7.4, 6.1 Hz, 1H), 3.48 (dt, *J* = 11.6, 6.3 Hz, 2H), 2.93 (s, 3H), 1.66 – 1.27 (m, 6H), 1.21 – 1.08 (m, 1H), 0.85 (dd, *J* = 6.4, 3.1 Hz, 3H).;

¹³C NMR (101 MHz, CDCl₃) δ 164.81, 164.43, 149.21, 148.98, 137.13, 136.65, 122.01, 121.21, 120.92, 62.10, 62.04, 54.76, 45.87, 45.64, 33.00, 32.30, 29.77, 29.69, 29.20, 29.15, 19.98, 19.51.;

HRMS (ESI) *m/z* calcd for C₁₂H₂₁N₂O⁺ (M+H)⁺ 209.1648, found 209.1644.



An oven dried 8 mL reaction vial was charged with a stir bar, TsCl (38.1 mg, 0.2 mmol) was charged under air. The reaction vial was sealed, evacuated and backfilled three times with Ar. Then under Ar atmosphere, added of dry DCM (2 mL, 0.1 M) and NEt₃ (40.5 mg, 0.4 mmol). The reaction mixture was stirred at 0°C for 5 min, then compound **35** (41.6 mg, 0.2 mmol) was added. The

solution was left stirring for 2 h. The reaction mixture was washed with 1M HCl and saturated NaHCO₃ aqueous solution. The organic phase was dried (Na₂SO₄), filtered, and evaporated. An oven dried 8 mL reaction vial was charged with a stir bar, PPh₃ (62.9 mg, 0.24 mmol) and crude Ts protected product were charged under air. The reaction vial was sealed, evacuated and backfilled three times with Ar. The reaction mixture was stirred at 0°C for 5 min, then DEAD (41.8 mg, 0.24 mmol) was added under Ar. The solution was left stirring for 5 h. Reaction mixture was evaporated, crude mixture was purified using column chromatography to give **31** as a white solid (61.9 mg, 90% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.38 (ddd, *J* = 20.4, 5.1, 2.2 Hz, 1H), 7.61 – 7.41 (m, 2H), 7.41 – 7.25 (m, 2H), 7.13 – 7.01 (m, 3H), 5.29 – 4.94 (m, 1H), 3.98 – 3.55 (m, 2H), 2.35 (d, *J* = 16.4 Hz, 3H), 2.30 – 1.99 (m, 1H), 1.97 – 1.73 (m, 4H), 1.70 – 1.53 (m, 1H), 1.37 – 1.10 (m, 1H), 1.06 – 0.96 (m, 3H).;

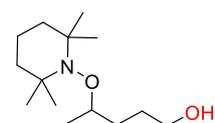
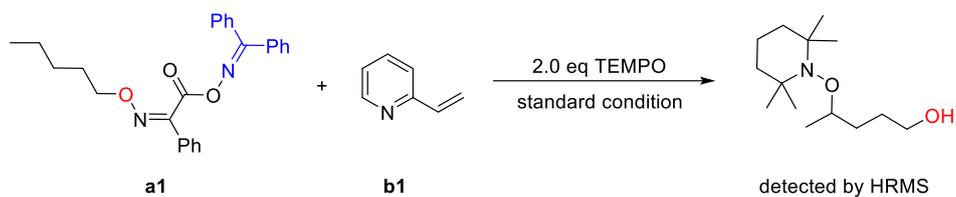
¹³C NMR (101 MHz, CDCl₃) δ 161.97, 161.83, 149.24, 149.01, 142.58, 142.33, 138.15, 137.31, 136.29, 136.24, 129.21, 129.03, 127.03, 121.89, 121.76, 121.68, 121.31, 61.31, 59.78, 46.18, 45.02, 43.83, 39.73, 38.36, 35.93, 32.23, 30.20, 29.96, 27.27, 24.63, 21.43, 21.38, 18.97.;

HRMS (ESI) *m/z* calcd for C₁₉H₂₅N₂O₂S⁺ (M+H)⁺ 345.1631, found 345.1640.

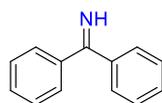
4. Mechanism Study

4.1 Tempo trapping experiment

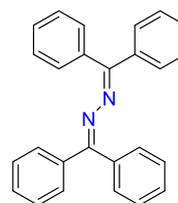
An oven dried 8 mL reaction vial was charged with a stir bar, bifunctional reagent **a1** (0.3 mmol, 1.5 equiv.), **b1** (0.2 mmol, 1.0 equiv.), TEMPO (0.4 mmol, 2.0 equiv.) and 2-*i*PrTX (25.4 mg, 5 mol %) were charged under air. The reaction vial was sealed, evacuated and backfilled three times with Ar. Then under Ar atmosphere, added of EtOAc (2.0 mL, 0.1 M). The reaction mixture was stirred and irradiated using a 50 W 395 nm LED lamp until the reaction was completed. Intermediates and products of the reaction process was detected by HRMS.



(M+H⁺) calc: 244.2271
Exact Mass: 244.2272

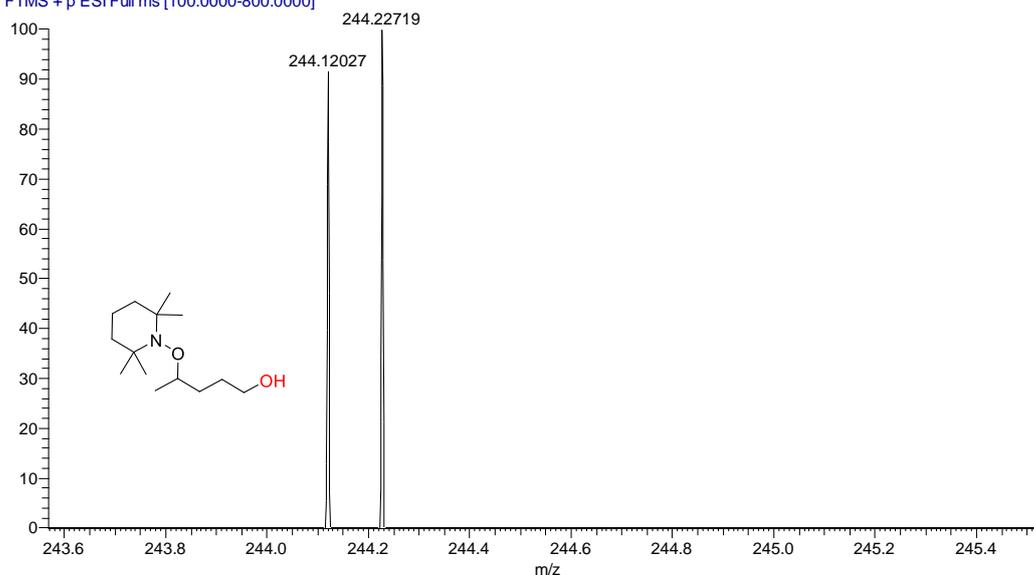


(M+H⁺) calc: 182.0964
Exact Mass: 182.0963

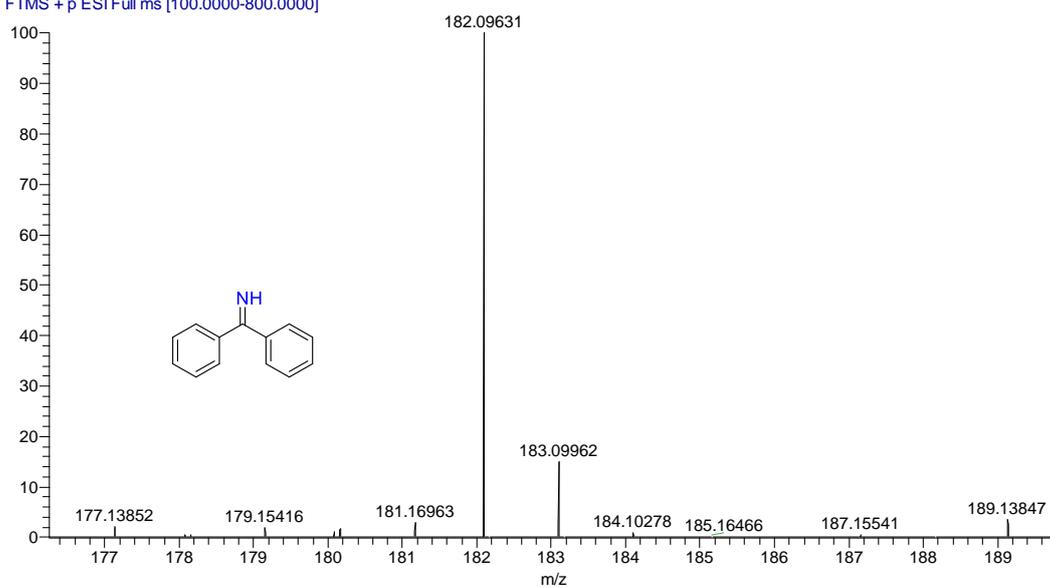


(M+H⁺) calc: 361.1999
Exact Mass: 361.1696

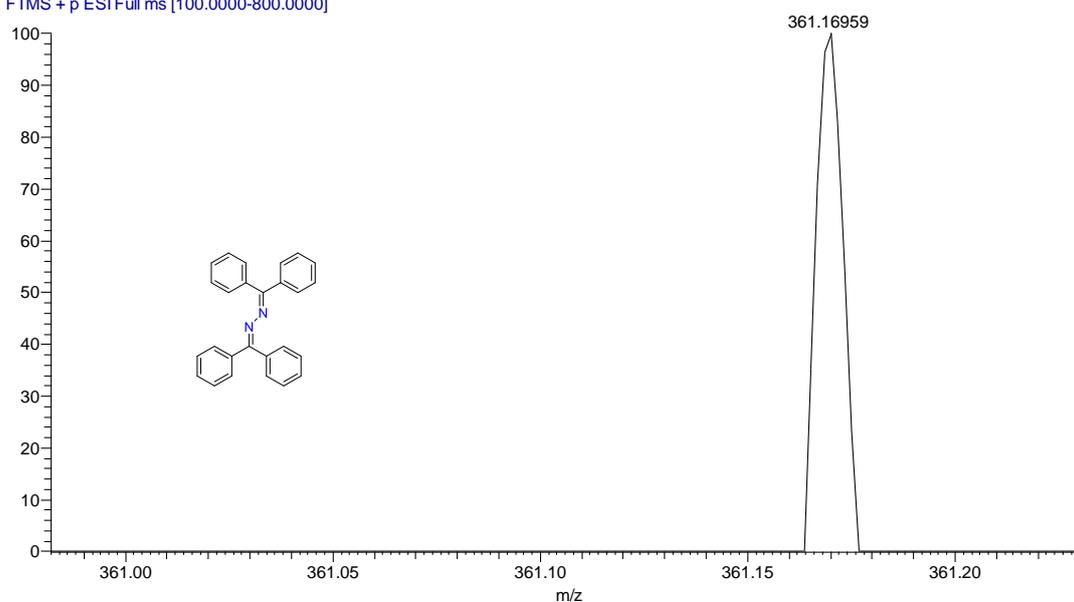
20240812_12_3 #42 RT: 0.21 AV: 1 NL: 1.66E6
T: FTMS + p ESI Full ms [100.0000-800.0000]



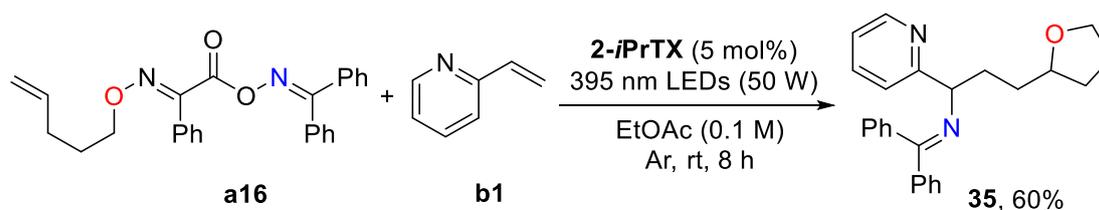
20240812_12_3 #61 RT: 0.31 AV: 1 NL: 1.51E8
T: FTMS + p ESI Full ms [100.0000-800.0000]



20240812_12_3 #32 RT: 0.16 AV: 1 NL: 5.93E5
T: FTMS + p ESI Full ms [100.0000-800.0000]



4.2 Alkoxy radical cyclization experiment



An oven dried 8 mL reaction vial was charged with a stir bar, bifunctional reagent **a16** (0.3 mmol, 1.5 equiv.), **b1** (0.2 mmol, 1.0 equiv.) and 2-*i*PrTX (25.4 mg, 5 mol %) were charged under air. The reaction vial was sealed, evacuated and backfilled three times with Ar. Then under Ar

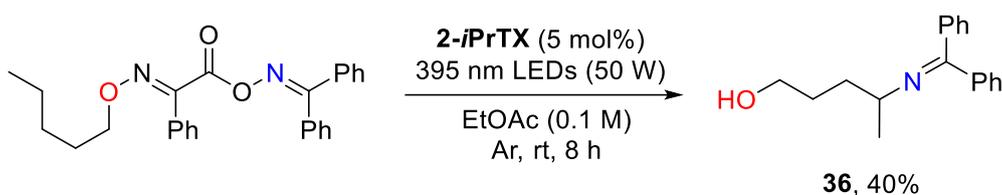
atmosphere, added of EtOAc (2.0 mL, 0.1 M). The reaction mixture was stirred and irradiated using a 50 W 395 nm LED lamp for 8 h until the reaction was complete. After irradiation, the resulting homogenous solution was transferred to a 25 mL round bottom flask with aid of EtOAc (2 x 3 mL). NEt₃ (approx. 0.5 mL) and SiO₂ were added to this solution and the volatiles were removed under reduced pressure, affording a powder which was loaded on column. Purification by flash column chromatography on SiO₂, pre-basified with NEt₃ using pentane: EtOAc mixtures afforded the corresponding product **35** as a yellow oil (44.4 mg, 59% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.49 (d, *J* = 4.9 Hz, 1H), 7.71 – 7.31 (m, 10H), 7.15 – 7.02 (m, 3H), 4.59 (dd, *J* = 8.0, 4.9 Hz, 1H), 3.83 – 3.76 (m, 1H), 3.67 (dq, *J* = 22.5, 7.7 Hz, 2H), 2.04 – 1.77 (m, 5H), 1.61 – 1.32 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 168.00, 167.94, 163.95, 163.89, 148.87, 139.95, 136.76, 136.48, 136.46, 130.02, 128.70, 128.68, 128.46, 128.42, 128.02, 127.80, 127.77, 121.84, 121.80, 121.75, 121.73, 79.36, 79.28, 67.92, 67.53, 34.94, 34.89, 32.20, 31.20, 31.17, 25.67, 25.65.

HRMS (ESI) *m/z* calcd for C₂₅H₂₇N₂O⁺ (M+H)⁺ 373.2118, found 373.2118.

4.3 Blank experiment



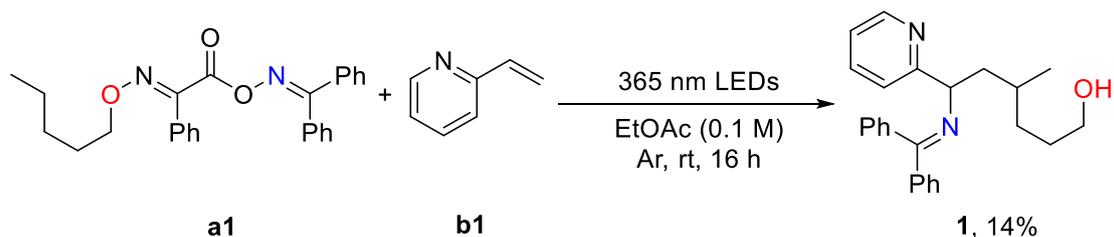
An oven dried 8 mL reaction vial was charged with a stir bar, bifunctional reagent **a1** (0.2 mmol, 1.0 equiv.), and 2-*i*PrTX (2.5 mg, 5 mol %) were charged under air. The reaction vial was sealed, evacuated and backfilled three times with Ar. Then under Ar atmosphere, added of EtOAc (2.0 mL, 0.1 M). The reaction mixture was stirred and irradiated using a 50 W 395 nm LED lamp for 12 h. After irradiation, the resulting homogenous solution was transferred to a 25 mL round bottom flask with aid of EtOAc (2 x 3 mL). NEt₃ (approx. 0.5 mL) and SiO₂ were added to this solution and the volatiles were removed under reduced pressure, affording a powder which was loaded on column. Purification by flash column chromatography on SiO₂, pre-basified with NEt₃ using pentane: EtOAc mixtures afforded the corresponding product **36** in 40% yield.

¹H NMR (400 MHz, CDCl₃) δ 7.62 – 7.55 (m, 2H), 7.51 – 7.31 (m, 6H), 7.21 – 7.14 (m, 2H), 3.69 – 3.46 (m, 3H), 3.33 (s, 1H), 1.75 – 1.52 (m, 4H), 1.18 (d, *J* = 6.3 Hz, 3H);

¹³C NMR (101 MHz, CDCl₃) δ 167.01, 139.72, 137.14, 130.02, 128.50, 128.47, 128.31, 128.16, 127.58, 63.02, 56.66, 35.00, 29.07, 21.48.

HRMS (ESI) *m/z* calcd for C₁₈H₂₂NO⁺ (M+H)⁺ 268.1696, found 268.1674.

4.4 Direct excitation experiment



An oven dried 8 mL reaction vial was charged with a stir bar, bifunctional reagent **a1** (0.3 mmol, 1.5 equiv.), and **b1** (0.2 mmol, 1.0 equiv.) were charged under air. The reaction vial was sealed, evacuated and backfilled three times with Ar. Then under Ar atmosphere, added of EtOAc (2.0 mL, 0.1 M). The reaction mixture was stirred and irradiated using a 30 W 365 nm LED lamp for 16 h. After irradiation, the resulting homogenous solution was transferred to a 25 mL round bottom flask with aid of EtOAc (2 x 3 mL). NEt_3 (approx. 0.5 mL) and SiO_2 were added to this solution and the volatiles were removed under reduced pressure, affording a powder which was loaded on column. Purification by flash column chromatography on SiO_2 , pre-basified with NEt_3 using pentane: EtOAc mixtures afforded the corresponding product **1** in 14% yield.

4.5 Quantum yield calculations

Determination of the photon flux

The photon flux was determined by ferrioxalate actinometry similar to a procedure by Xia,² the photon flux of the LED ($\lambda_{\text{max}} = 395 \text{ nm}$) was first determined by standard ferrioxalate actinometry. For this, a 10 mL 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate hydrate (0.737 g) in H_2SO_4 (10 mL of a 0.05 M solution). A 20 mL buffered solution of 1,10-phenanthroline was prepared by dissolving 1,10-phenanthroline (20 mg) and sodium acetate (4.5 g) in H_2SO_4 (20 mL of a 0.5 M solution). Both solutions were stored in the dark. To determine the photon flux of the spectrophotometer, 4.00 mL of the ferrioxalate solution was placed in a cuvette and irradiated for 90 seconds at 395 nm with excitation and emission slit width of 10 nm on the benchtop under air. After irradiation, 1.00 mL of this irradiated ferrioxalate solution, 0.20 mL of phenanthroline buffer solution and 2.00 mL of water were added to an 8 mL scintillation vial with a stir bar. stirred for 1 h to allow the ferrous ions to completely coordinate with the phenanthroline. The absorption of the solution was measured at 510 nm. A non-irradiated sample was also prepared identically and the absorption at 510 nm was also measured. Each sample preparation and measurements were repeated two more times. The average of the absorption of the irradiated and non-irradiated samples were determined and used for the calculation of photon flux.

Conversion was calculated using equation 1

$$\text{mol Fe}^{2+} = \frac{V \times \Delta A(510 \text{ nm})}{l \times \epsilon} \quad (1)$$

Where V is the total volume (0.0128 L) of the solution after addition of phenanthroline, ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, l is the path length (1.0 cm), and ϵ is the molar absorptivity of the ferrioxalate actinometer at 510 nm ($11100 \text{ L mol}^{-1} \text{ cm}^{-1}$).

The average value of the experiment was $1.41 \times 10^{-6} \text{ mol of Fe}^{2+}$.

The photon flux can be calculated using equation 2.

$$\text{Photon flux} = \frac{\text{mol}(\text{Fe}^{2+})}{\Phi \times t \times f} \quad (2)$$

Where Φ is the quantum yield for the ferrioxalate actinometer (1.13 at 395 nm), t is the irradiation time, and f is the fraction of light absorbed at $\lambda_{\text{max}} = 390$ nm by the ferrioxalate actinometer. This value is calculated using equation 3 where $A(395 \text{ nm})$ is the absorption of the ferrioxalate solution at 395 nm.

$$f = 1 - 10^{-A(395 \text{ nm})} \quad (3)$$

A measured absorbance value of >3 at 395 nm indicates the fraction of absorbed light (f) to be >0.999 .

The average photon flux was calculated to be 1.39×10^{-8} einsteins s^{-1}

Determination of the 1,6-oxyimination reaction quantum yield

An oven dried 8 mL reaction vial was charged with a stir bar, bifunctional reagent **a1** (0.3 mmol, 1.5 equiv.), and 2-*i*PrTX photosensitizer (2.5 mg, 5 mol %) were charged under air. The reaction vial was sealed, evacuated and backfilled three times with Ar. Then under Ar atmosphere, added of EtOAc (2.0 mL, 0.1 M) and **b1** (0.2 mmol, 1.0 equiv.). The reaction mixture was stirred and irradiated using a 50 W 395 nm LED lamp for 150s using the same setup as for the photon flux determination. Then, the NMR yield was determined (9%) using CH_2Br_2 as internal standard. The reaction quantum yield was determined using equation 4, where photon flux was determined as above described, t is the reaction time, f is determined by using equation 4.

$$\Phi = \frac{\text{mol of product formed}}{\text{photo flux} \times t \times f} \quad (4)$$

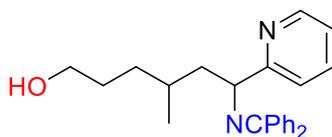
$$\Phi = \frac{0.0002 \times 0.09}{1.39 \times 10^{-8} \times 150 \times 0.978} = 8.52$$

The 1,6-oxyimination reaction quantum yield (Φ) was determined to be 8.52, indicating that a radical chain propagation might be operative in this reaction.

5. Reference

1. Z. Liu, Y. Pan, P. Zou, H. Huang, Y. Chen and Y. Chen, *Org. Lett.*, **2022**, *24*, 5951-5956.
2. X.-K. Qi, M.-J. Zheng, C. Yang, Y. Zhao, L. Guo and W. Xia, *J. Am. Chem. Soc.*, **2023**, *145*, 16630-16641.

6. Characterization Data



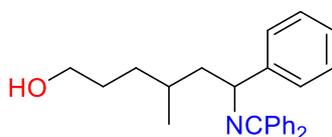
6-((diphenylmethylene)amino)-4-methyl-6-(pyridin-2-yl)hexan-1-ol (**1**)

Compound **1** was prepared following the general procedure as a yellow oil (48.4 mg, 65%, 1.1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 8.59 – 8.44 (m, 1H), 7.73 – 7.53 (m, 4H), 7.45 – 7.33 (m, 6H), 7.17 – 7.01 (m, 3H), 4.71 (ddd, *J* = 8.8, 5.5, 3.7 Hz, 1H), 3.59 – 3.40 (m, 2H), 1.99 – 1.73 (m, 2H), 1.57 – 1.50 (m, 1H), 1.44 – 1.35 (m, 2H), 1.26 – 0.94 (m, 2H), 0.83 (d, *J* = 6.7 Hz, 1.4H), 0.71 (d, *J* = 6.4 Hz, 1.6H).;

¹³C NMR (101 MHz, CDCl₃) δ 167.85, 167.66, 164.60, 164.36, 148.77, 148.54, 139.99, 136.79, 136.71, 136.63, 136.57, 130.05, 130.03, 128.68, 128.65, 128.57, 128.43, 128.42, 128.06, 128.05, 127.85, 122.00, 121.94, 121.81, 121.79, 66.33, 65.90, 63.14, 62.21, 46.01, 45.94, 32.39, 32.07, 29.99, 29.65, 29.56, 28.56, 20.27, 19.57.;

HRMS (ESI) *m/z* calcd for C₂₅H₂₉N₂O⁺ (M+H)⁺ 373.2274, found 373.2270.



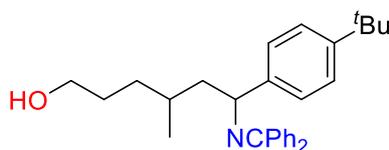
6-((diphenylmethylene)amino)-4-methyl-6-phenylhexan-1-ol (**2**)

Compound **2** was prepared following the general procedure as a yellow oil (41.6 mg, 56%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.70 (ddd, *J* = 8.4, 4.4, 1.7 Hz, 2H), 7.50 – 7.30 (m, 10H), 7.27 – 7.20 (m, 1H), 7.15 – 7.00 (m, 2H), 4.55 – 4.38 (m, 1H), 3.65 – 3.39 (m, 2H), 2.13 – 1.70 (m, 2H), 1.47 – 1.00 (m, 5H), 0.81 (d, *J* = 6.77 Hz, 1.5H), 0.72 (d, *J* = 6.44 Hz, 1.5H).;

¹³C NMR (101 MHz, CDCl₃) δ 167.10, 166.33, 145.81, 145.22, 140.04, 137.69, 137.11, 129.84, 128.56, 128.39, 128.35, 128.33, 128.30, 128.24, 128.02, 128.01, 127.94, 127.84, 127.26, 127.04, 126.63, 126.53, 64.77, 64.38, 63.28, 47.15, 46.99, 33.27, 32.48, 30.06, 29.98, 29.56, 29.48, 20.05, 19.47.;

HRMS (ESI) *m/z* calcd for C₂₆H₃₀NO⁺ (M+H)⁺ 372.2322, found 372.2321.



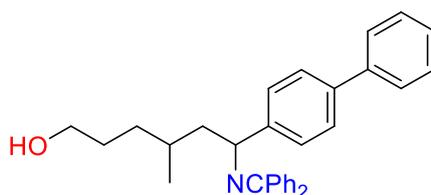
6-(4-(tert-butyl)phenyl)-6-((diphenylmethylene)amino)-4-methylhexan-1-ol (**3**)

Compound **3** was prepared following the general procedure as a yellow oil (42.7 mg, 50%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.74 – 7.64 (m, 2H), 7.50 – 7.32 (m, 8H), 7.29 – 7.25 (m, 2H), 7.16 – 7.07 (m, 2H), 4.48 (q, *J* = 6.0, 5.1 Hz, 1H), 3.64 – 3.43 (m, 2H), 2.15 – 1.74 (m, 2H), 1.34 (s, 14H), 0.80 (d, *J* = 6.86, 1.5H), 0.70 (d, *J* = 6.52, 1.5H);

¹³C NMR (101 MHz, CDCl₃) δ 166.30, 165.93, 149.33, 149.25, 142.57, 142.00, 140.13, 137.20, 137.14, 129.78, 129.75, 128.56, 128.34, 128.30, 128.27, 128.20, 128.07, 127.98, 127.94, 126.85, 126.65, 125.19, 64.41, 64.00, 63.30, 46.97, 46.91, 34.43, 33.26, 32.46, 31.45, 30.07, 29.97, 29.53, 29.43, 20.07, 19.50.;

HRMS (ESI) *m/z* calcd for C₃₀H₃₈NO⁺ (M+H)⁺ 428.2948, found 428.2944.



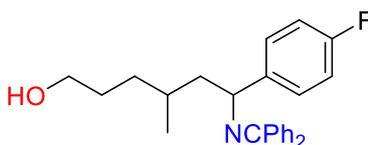
6-([1,1'-biphenyl]-4-yl)-6-((diphenylmethylene)amino)-4-methylhexan-1-ol (**4**)

Compound **4** was prepared following the general procedure as a yellow oil (44.7 mg, 50%, 1.1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.70 (ddt, *J* = 6.2, 4.2, 1.7 Hz, 2H), 7.62 – 7.52 (m, 4H), 7.50 – 7.30 (m, 11H), 7.11 (dq, *J* = 9.9, 2.8, 2.2 Hz, 2H), 4.61 – 4.39 (m, 1H), 3.61 – 3.38 (m, 2H), 2.17 – 1.81 (m, 2H), 1.54 – 1.20 (m, 5H), 0.82 (d, *J* = 6.87 Hz, 1.57H), 0.72 (d, *J* = 6.66 Hz, 1.43H);

¹³C NMR (101 MHz, CDCl₃) δ 166.87, 166.47, 144.94, 144.34, 141.12, 140.04, 139.51, 139.42, 137.16, 137.10, 129.93, 129.90, 128.74, 128.60, 128.46, 128.41, 128.35, 128.29, 128.06, 128.05, 127.98, 127.87, 127.66, 127.45, 127.10, 127.06, 64.47, 64.09, 63.29, 47.14, 47.00, 33.31, 32.49, 30.10, 30.01, 29.62, 29.52, 20.08, 19.49.;

HRMS (ESI) *m/z* calcd for C₃₂H₃₄NO⁺ (M+H)⁺ 448.2635, found 448.2635.



6-((diphenylmethylene)amino)-6-(4-fluorophenyl)-4-methylhexan-1-ol (**5**)

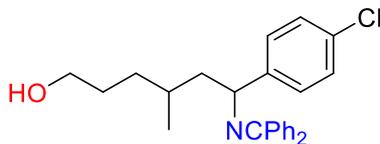
Compound **5** was prepared following the general procedure as a yellow oil (48.3 mg, 62%, 1.1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.68 (ddd, *J* = 6.0, 4.1, 2.1 Hz, 2H), 7.47 – 7.25 (m, 8H), 7.11 – 6.94 (m, 4H), 4.55 – 4.40 (m, 1H), 3.66 – 3.50 (m, 2H), 2.10 – 1.70 (m, 2H), 1.49 – 1.15 (m, 5H), 0.80 (d, *J* = 6.6 Hz, 1.43H), 0.71 (d, *J* = 6.4 Hz, 1.57H).;

¹³C NMR (101 MHz, CDCl₃) δ 166.90, 166.49, 162.84, 162.77, 160.41, 160.34, 141.52, 141.49, 140.91, 140.88, 139.89, 137.07, 137.02, 129.98, 129.96, 128.67, 128.59, 128.54, 128.46, 128.43, 128.38, 128.36, 128.30, 128.05, 127.83, 127.73, 115.18, 114.97, 63.99, 63.62, 63.24, 47.22, 47.04, 33.24, 32.51, 30.06, 29.96, 29.53, 29.45, 19.97, 19.46.;

¹⁹F NMR (377 MHz, CDCl₃) δ -116.39, -116.53.

HRMS (ESI) m/z calcd for C₂₆H₂₉FNO⁺ (M+H)⁺ 390.2228, found 390.2223.



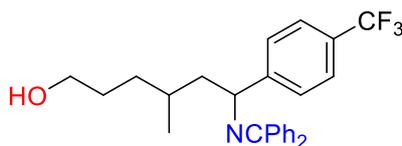
6-(4-chlorophenyl)-6-((diphenylmethylene)amino)-4-methylhexan-1-ol (6)

Compound **6** was prepared following the general procedure as a yellow oil (47.8 mg, 59%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.72 – 7.58 (m, 2H), 7.43 – 7.20 (m, 10H), 7.08 – 6.96 (m, 2H), 4.49 – 4.32 (m, 1H), 3.60 – 3.45 (m, 2H), 2.07 – 1.69 (m, 2H), 1.48 – 1.21 (m, 5H), 0.76 (d, *J* = 6.5 Hz, 1.5H), 0.67 (d, *J* = 6.4 Hz, 1.5H);

¹³C NMR (101 MHz, CDCl₃) δ 167.18, 166.76, 144.34, 143.74, 139.83, 137.01, 136.95, 132.18, 132.08, 130.03, 130.00, 128.58, 128.55, 128.51, 128.47, 128.37, 128.32, 128.06, 127.80, 127.70, 64.06, 63.70, 63.21, 47.15, 46.96, 33.26, 32.48, 30.05, 29.96, 29.55, 29.47, 19.98, 19.42.

HRMS (ESI) m/z calcd for C₂₆H₂₉ClNO⁺ (M+H)⁺ 406.1932, found 406.1933.



6-((diphenylmethylene)amino)-4-methyl-6-(4-(trifluoromethyl)phenyl)hexan-1-ol (7)

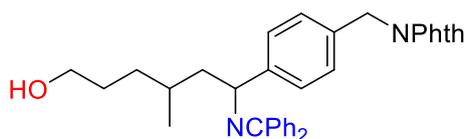
Compound **7** was prepared following the general procedure as a yellow oil (56.2 mg, 64%, 1.2:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.76 – 7.57 (m, 4H), 7.49 – 7.29 (m, 8H), 7.12 – 7.01 (m, 2H), 4.63 – 4.44 (m, 1H), 3.63 – 3.42 (m, 2H), 2.16 – 1.74 (m, 2H), 1.50 – 1.13 (m, 5H), 0.82 (d, *J* = 6.6 Hz, 1.36H), 0.72 (d, *J* = 6.4 Hz, 1.64H);

¹³C NMR (101 MHz, CDCl₃) δ 167.58, 167.18, 149.91, 149.34, 139.72, 136.92, 136.86, 130.14, 130.11, 128.58, 128.43, 128.37, 128.10, 127.78, 127.67, 127.53, 127.32, 125.35, 125.32, 125.28, 125.24, 64.37, 64.03, 63.19, 47.17, 47.02, 33.29, 32.43, 30.04, 29.97, 29.59, 29.50, 19.98, 19.39;

¹⁹F NMR (377 MHz, CDCl₃) δ -62.25, -62.26.

HRMS (ESI) m/z calcd for C₂₇H₂₉F₃NO⁺ (M+H)⁺ 440.2196, found 440.2195.



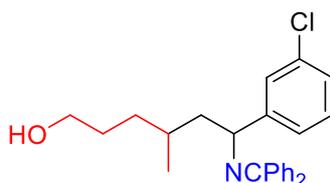
2-(4-(1-((diphenylmethylene)amino)-6-hydroxy-3-methylhexyl)benzyl)isoindoline-1,3-dione (8)

Compound **8** was prepared following the general procedure as a white solid (60.5 mg, 57%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.88 – 7.82 (m, 2H), 7.73 – 7.63 (m, 4H), 7.45 – 7.26 (m, 10H), 7.10 – 7.01 (m, 2H), 4.84 (d, *J* = 2.9 Hz, 2H), 4.53 – 4.35 (m, 1H), 3.59 – 3.41 (m, 2H), 2.03 – 1.65 (m, 2H), 1.46 – 1.13 (m, 5H), 0.77 (d, *J* = 6.6 Hz, 1.5H), 0.68 (d, *J* = 6.4 Hz, 1.5H).;

¹³C NMR (101 MHz, CDCl₃) δ 168.12, 166.68, 166.29, 145.42, 144.81, 139.94, 137.08, 137.01, 134.66, 134.57, 133.98, 132.15, 129.86, 129.84, 128.69, 128.67, 128.53, 128.41, 128.37, 128.32, 128.25, 127.99, 127.98, 127.92, 127.79, 127.59, 127.37, 123.34, 64.50, 64.09, 63.20, 47.06, 46.89, 41.40, 33.23, 32.51, 30.03, 29.94, 29.50, 29.42, 20.00, 19.47.;

HRMS (ESI) *m/z* calcd for C₃₅H₃₅N₂O₃⁺ (M+H)⁺ 531.2642, found 531.2647.



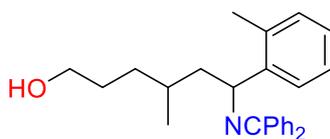
6-(3-chlorophenyl)-6-((diphenylmethylene)amino)-4-methylhexan-1-ol (9)

Compound **9** was prepared following the general procedure as a yellow oil (46.2 mg, 57%, 1.1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.80 – 7.67 (m, 2H), 7.53 – 7.32 (m, 7H), 7.25 – 6.99 (m, 5H), 4.53 – 4.39 (m, 1H), 3.61 – 3.44 (m, 2H), 2.14 – 1.72 (m, 2H), 1.52 – 1.24 (m, 5H), 0.81 (d, *J* = 6.7 Hz, 1.43H), 0.70 (d, *J* = 6.1 Hz, 1.57H).;

¹³C NMR (101 MHz, CDCl₃) δ 167.34, 166.94, 147.92, 147.36, 139.78, 136.94, 136.87, 134.10, 130.08, 130.05, 129.60, 128.60, 128.55, 128.51, 128.40, 128.34, 128.07, 127.83, 127.72, 127.37, 127.17, 126.82, 126.72, 125.38, 125.17, 64.26, 63.88, 63.21, 47.15, 47.03, 33.27, 32.42, 30.04, 29.96, 29.54, 29.44, 20.01, 19.41.;

HRMS (ESI) *m/z* calcd for C₂₆H₂₉ClNO⁺ (M+H)⁺ 406.1932, found 406.1933.



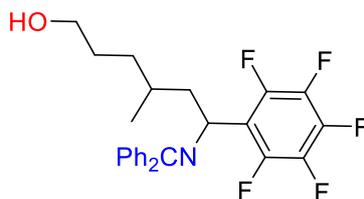
6-((diphenylmethylene)amino)-4-methyl-6-(o-tolyl)hexan-1-ol (10)

Compound **10** was prepared following the general procedure as a yellow oil (40.9 mg, 53%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.78 – 7.64 (m, 3H), 7.48 – 7.33 (m, 6H), 7.23 – 6.95 (m, 5H), 4.66 (ddd, *J* = 23.4, 9.4, 3.8 Hz, 1H), 3.59 (dt, *J* = 36.3, 6.6 Hz, 2H), 1.94 (d, *J* = 11.1 Hz, 3H), 1.73 – 1.22 (m, 7H), 0.89 (d, *J* = 6.6 Hz, 1.5H), 0.75 (d, *J* = 6.4 Hz, 1.5H).;

¹³C NMR (101 MHz, CDCl₃) δ 167.17, 166.61, 144.95, 144.43, 140.01, 139.97, 137.65, 133.97, 133.60, 130.07, 130.03, 129.88, 129.84, 128.55, 128.53, 128.35, 128.33, 128.31, 128.24, 128.05, 128.03, 127.65, 127.63, 127.59, 127.32, 126.24, 126.07, 126.00, 63.31, 60.48, 60.17, 46.65, 46.43, 33.82, 31.75, 30.22, 30.16, 29.94, 29.83, 20.53, 19.08, 18.99, 18.84.;

HRMS (ESI) m/z calcd for $C_{27}H_{32}NO^+$ (M+H)⁺ 386.2478, found 386.2478.



6-((diphenylmethylene)amino)-4-methyl-6-(perfluorophenyl)hexan-1-ol (**11**)

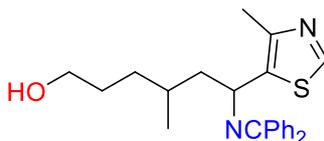
Compound **11** was prepared following the general procedure as a yellow oil (59.1 mg, 64%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.65 (ddd, $J = 7.2, 3.4, 1.9$ Hz, 2H), 7.58 – 7.30 (m, 6H), 7.14 – 7.01 (m, 2H), 5.07 – 4.88 (m, 1H), 3.84 – 3.67 (m, 1H), 3.61 – 3.42 (m, 2H), 2.29 – 1.97 (m, 1H), 1.86 – 1.56 (m, 2H), 1.41 – 1.12 (m, 4H), 0.74 (d, $J = 6.6$ Hz, 1.5H), 0.67 (d, $J = 6.2$ Hz, 1.5H);

¹³C NMR (101 MHz, CDCl₃) δ 169.55, 169.33, 139.28, 136.39, 136.29, 130.46, 128.73, 128.65, 128.13, 127.35, 127.28, 67.99, 63.11, 55.79, 55.40, 46.08, 42.56, 42.32, 33.07, 32.38, 30.14, 30.00, 29.71, 29.57, 25.62, 19.48, 19.27;

¹⁹F NMR (377 MHz, CDCl₃) δ -140.86 (ddd, $J = 63.5, 22.2, 7.3$ Hz), -156.70 (dt, $J = 75.9, 20.9$ Hz), -162.18 – -162.85 (m).

HRMS (ESI) m/z calcd for $C_{26}H_{25}F_5NO^+$ (M+H)⁺ 462.1851, found 462.1850.



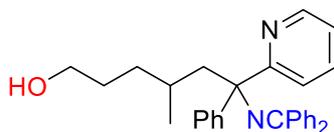
6-((diphenylmethylene)amino)-4-methyl-6-(4-methylthiazol-5-yl)hexan-1-ol (**12**)

Compound **12** was prepared following the general procedure as a yellow oil (27.5 mg, 35%, 1.9:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 8.62 (d, $J = 8.2$ Hz, 1H), 7.64 (d, $J = 6.6$ Hz, 2H), 7.46 – 7.29 (m, 6H), 7.10 – 6.99 (m, 2H), 4.84 – 4.71 (m, 1H), 3.54 (m, 2H), 2.07 (d, $J = 3.8$ Hz, 3H), 1.85 – 1.71 (m, 2H), 1.56 – 1.37 (m, 4H), 1.19 – 1.05 (m, 1H), 0.77 (d, $J = 6.5$ Hz, 1.97H), 0.71 (d, $J = 6.0$ Hz, 1.03H);

¹³C NMR (101 MHz, CDCl₃) δ 167.86, 167.35, 151.17, 150.94, 146.71, 145.99, 139.25, 136.68, 136.60, 136.43, 136.07, 130.31, 130.28, 128.72, 128.66, 128.62, 128.60, 128.55, 128.11, 127.55, 127.49, 63.18, 63.13, 57.83, 57.63, 47.47, 47.37, 33.21, 32.58, 30.05, 30.03, 29.51, 29.47, 19.98, 19.34, 15.26, 15.15;

HRMS (ESI) m/z calcd for $C_{24}H_{29}N_2OS^+$ (M+H)⁺ 393.1995, found 393.1992.



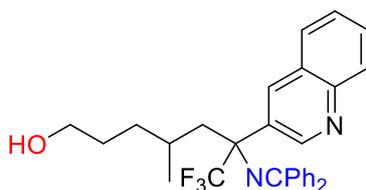
6-((diphenylmethylene)amino)-4-methyl-6-phenyl-6-(pyridin-2-yl)hexan-1-ol (**13**)

Compound **13** was prepared following the general procedure as a yellow oil (79.9 mg, 88%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 8.31 (dd, *J* = 25.6, 6.7 Hz, 1H), 7.77 – 7.69 (m, 2H), 7.66 – 7.55 (m, 1H), 7.50 – 7.37 (m, 4H), 7.30 – 6.95 (m, 9H), 6.55 (d, *J* = 7.5 Hz, 2H), 3.48 – 3.34 (m, 2H), 2.80 – 2.30 (m, 2H), 1.69 – 1.36 (m, 4H), 1.28 – 0.91 (m, 3H), 0.82 (d, *J* = 6.6 Hz, 1.5H), 0.78 (d, *J* = 6.8 Hz, 1.5H);

¹³C NMR (101 MHz, CDCl₃) δ 168.37, 168.30, 166.44, 166.40, 149.64, 149.31, 148.43, 147.96, 141.82, 138.62, 138.59, 135.87, 135.80, 129.94, 128.41, 128.36, 128.05, 128.04, 127.86, 127.81, 127.62, 127.58, 127.32, 127.28, 126.92, 126.84, 125.80, 125.74, 123.77, 123.25, 120.85, 120.81, 71.16, 63.05, 46.04, 34.10, 33.64, 29.87, 29.85, 28.89, 28.80, 21.44, 21.07.;

HRMS (ESI) *m/z* calcd for C₃₁H₃₃N₂O⁺ (M+H)⁺ 449.2587, found 449.2588.



6-((diphenylmethylene)amino)-7,7,7-trifluoro-4-methyl-6-(quinolin-3-yl)heptan-1-ol (**14**)

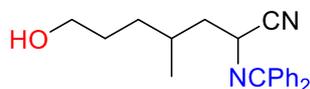
Compound **14** was prepared following the general procedure as a yellow oil (58.9 mg, 60%, 1.3:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 8.94 – 8.82 (m, 1H), 8.02 (d, *J* = 8.5 Hz, 1H), 7.90 – 7.79 (m, 1H), 7.74 – 7.55 (m, 4H), 7.53 – 7.28 (m, 4H), 6.83 (m, 3H), 6.59 (m, 2H), 3.70 – 3.42 (m, 2H), 2.58 (dd, *J* = 15.1, 3.4 Hz, 0.44H), 2.47 (dd, *J* = 14.4, 5.4 Hz, 0.56H), 2.22 – 2.03 (m, 3H), 1.69 – 1.30 (m, 4H), 1.06 (dd, *J* = 10.0, 6.5 Hz, 3H);

¹³C NMR (101 MHz, CDCl₃) δ 169.43, 169.37, 151.27, 151.06, 146.78, 140.59, 140.45, 137.00, 135.10, 134.87, 133.05, 132.77, 130.78, 130.72, 129.93, 128.75, 128.12, 128.10, 127.99, 127.97, 127.94, 127.81, 127.57, 127.18, 127.06, 126.96, 126.82, 69.42, 69.17, 68.98, 62.94, 62.91, 42.73, 41.26, 35.44, 33.97, 30.45, 30.20, 29.41, 28.97, 21.39, 21.13.;

¹⁹F NMR (377 MHz, CDCl₃) δ -72.57, -72.91.

HRMS (ESI) *m/z* calcd for C₃₀H₃₀F₃N₂O⁺ (M+H)⁺ 491.2305, found 491.2305.



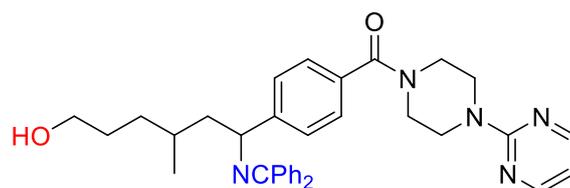
2-((diphenylmethylene)amino)-7-hydroxy-4-methylheptanenitrile (**15**)

Compound **15** was prepared following the general procedure as a colorless oil (37.2 mg, 58%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 7.73 – 7.61 (m, 2H), 7.61 – 7.34 (m, 6H), 7.24 (td, *J* = 6.8, 2.2 Hz, 2H), 4.47 – 4.23 (m, 1H), 3.66 – 3.36 (m, 2H), 2.05 (m, 1H), 1.77 – 1.52 (m, 4H), 1.39 – 1.15 (m, 2H), 0.78 (dd, *J* = 9.7, 6.5 Hz, 3H);

¹³C NMR (101 MHz, CDCl₃) δ 173.07, 172.72, 138.44, 138.42, 135.26, 135.19, 131.21, 131.19, 129.45, 129.03, 128.99, 128.97, 128.28, 127.48, 127.35, 62.83, 51.74, 51.12, 41.81, 41.67, 32.56, 29.85, 29.69, 29.23, 19.37, 19.07.;

HRMS (ESI) m/z calcd for $C_{21}H_{25}N_2O^+$ ($M+H$)⁺ 321.1961, found 321.1971.



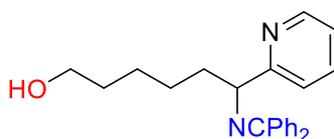
(4-(1-((diphenylmethylene)amino)-6-hydroxy-3-methylhexyl)phenyl)(4-(pyrimidin-2-yl)piperazin-1-yl)methanone (16)

Compound **16** was prepared following the general procedure as a yellow oil (51.6 mg, 46%, 1:1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 8.31 (d, J = 4.8 Hz, 2H), 7.70 – 7.63 (m, 2H), 7.50 – 7.28 (m, 10H), 7.17 – 6.87 (m, 2H), 6.52 (d, J = 4.8 Hz, 1H), 4.55 – 4.41 (m, 1H), 3.97 – 3.42 (m, 10H), 1.88 – 1.26 (m, 7H), 0.77 (d, J = 6.6 Hz, 1.43H), 0.67 (d, J = 6.6 Hz, 1.57H);

¹³C NMR (101 MHz, CDCl₃) δ 170.82, 167.25, 166.85, 161.56, 157.81, 147.87, 147.28, 139.83, 136.95, 136.88, 133.79, 133.68, 130.06, 130.03, 128.57, 128.54, 128.51, 128.40, 128.34, 128.08, 127.84, 127.72, 127.45, 127.33, 127.21, 110.51, 64.49, 64.08, 63.16, 47.08, 46.94, 33.27, 32.48, 30.03, 29.96, 29.52, 29.45, 20.00, 19.45.;

HRMS (ESI) m/z calcd for $C_{35}H_{40}N_5O_2^+$ ($M+H$)⁺ 562.3177, found 562.3170.



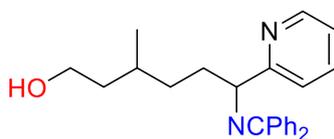
6-((diphenylmethylene)amino)-6-(pyridin-2-yl)hexan-1-ol (17)

Compound **17** was prepared following the general procedure as a yellow oil (43.7 mg, 61%);

¹H NMR (400 MHz, CDCl₃) δ 8.51 (dd, J = 5.1, 1.8 Hz, 1H), 7.78 – 7.33 (m, 10H), 7.19 – 7.01 (m, 3H), 4.61 (dd, J = 7.4, 5.6 Hz, 1H), 3.56 (t, J = 6.6 Hz, 2H), 2.02 – 1.89 (m, 2H), 1.49 (q, J = 6.8 Hz, 2H), 1.33 – 1.24 (m, 4H).;

¹³C NMR (101 MHz, CDCl₃) δ 167.82, 164.14, 148.75, 139.97, 136.76, 136.21, 130.03, 128.65, 128.46, 128.44, 128.06, 127.78, 121.82, 121.75, 67.87, 62.73, 38.25, 32.57, 25.85, 25.42.;

HRMS (ESI) m/z calcd for $C_{24}H_{27}N_2O^+$ ($M+H$)⁺ 359.2118, found 359.2111.



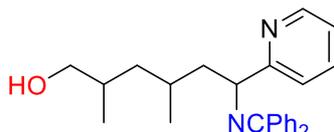
6-((diphenylmethylene)amino)-3-methyl-6-(pyridin-2-yl)hexan-1-ol (18)

Compound **18** was prepared following the general procedure as a yellow oil (34.3 mg, 46%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 8.48 (ddd, *J* = 10.8, 5.4, 1.5 Hz, 1H), 7.78 – 7.28 (m, 10H), 7.18 – 6.95 (m, 3H), 4.65 – 4.42 (m, 1H), 3.65 – 3.43 (m, 2H), 2.00 – 1.81 (m, 2H), 1.52 – 1.09 (m, 5H), 0.83 (d, *J* = 6.5 Hz, 1.5H), 0.79 (d, *J* = 6.5 Hz, 1.5H).;

¹³C NMR (101 MHz, CDCl₃) δ 167.87, 167.75, 164.16, 164.12, 148.81, 148.53, 139.96, 136.80, 136.73, 136.70, 136.54, 130.05, 128.66, 128.63, 128.51, 128.49, 128.47, 128.44, 128.07, 127.78, 127.72, 121.99, 121.84, 121.80, 121.75, 68.27, 67.68, 61.07, 60.45, 39.78, 39.71, 35.70, 35.64, 33.43, 32.45, 29.47, 28.47, 19.91, 19.63.;

HRMS (ESI) *m/z* calcd for C₂₅H₂₉N₂O⁺ (M+H)⁺ 373.2274, found 373.2277.



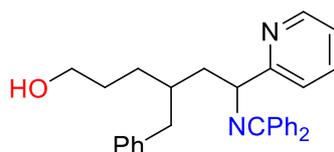
6-((diphenylmethylene)amino)-2,4-dimethyl-6-(pyridin-2-yl)hexan-1-ol (**19**)

Compound **19** was prepared following the general procedure as a yellow oil (55.6 mg, 72%, 1.3:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 8.56 – 8.43 (m, 1H), 7.77 – 7.31 (m, 10H), 7.21 – 6.98 (m, 3H), 4.82 – 4.64 (m, 1H), 3.52 – 3.30 (m, 2H), 2.11 – 1.97 (m, 1H), 1.75 – 1.16 (m, 5H), 0.91 – 0.79 (m, 3H), 0.70 (m, 3H).;

¹³C NMR (101 MHz, CDCl₃) δ 168.07, 167.67, 167.49, 164.61, 164.24, 148.76, 148.21, 140.02, 139.96, 136.98, 136.71, 136.62, 136.51, 130.05, 130.00, 128.67, 128.64, 128.60, 128.58, 128.55, 128.47, 128.44, 128.39, 128.07, 128.03, 127.89, 127.84, 127.77, 122.19, 122.02, 121.88, 121.83, 121.80, 121.72, 68.50, 68.11, 66.78, 66.25, 66.07, 65.59, 46.54, 46.40, 45.72, 41.07, 40.93, 39.48, 33.18, 33.00, 32.55, 27.25, 26.80, 26.44, 21.02, 20.71, 19.49, 18.09, 17.23, 16.73.;

HRMS (ESI) *m/z* calcd for C₂₆H₃₁N₂O⁺ (M+H)⁺ 387.2431, found 387.2431.



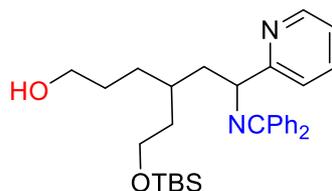
4-benzyl-6-((diphenylmethylene)amino)-6-(pyridin-2-yl)hexan-1-ol (**20**)

Compound **20** was prepared following the general procedure as a yellow oil (44.0 mg, 49%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 8.58 – 8.43 (m, 1H), 7.75 – 7.62 (m, 3H), 7.59 – 7.48 (m, 1H), 7.46 – 7.34 (m, 6H), 7.27 – 7.06 (m, 7H), 6.95 (d, *J* = 6.8 Hz, 1H), 4.77 (m, 1H), 3.52 – 3.34 (m, 2H), 2.57 – 2.42 (m, 2H), 2.01 – 1.82 (m, 2H), 1.74 – 1.58 (m, 1H), 1.55 – 1.39 (m, 2H), 1.29 – 1.11 (m, 2H).;

¹³C NMR (101 MHz, CDCl₃) δ 167.83, 167.80, 164.27, 164.23, 148.78, 148.34, 141.13, 140.86, 139.99, 139.94, 136.84, 136.65, 136.57, 136.54, 130.11, 130.08, 129.23, 129.08, 128.77, 128.65, 128.53, 128.46, 128.17, 128.09, 128.05, 127.93, 127.82, 125.68, 125.61, 122.18, 122.04, 121.92, 121.84, 66.21, 66.12, 63.01, 61.77, 42.62, 42.36, 40.50, 40.21, 36.30, 34.60, 29.35, 29.00, 28.64, 28.46.;

HRMS (ESI) m/z calcd for $C_{31}H_{33}N_2O^+$ ($M+Na$) $^+$ 449.2587, found 449.2585.



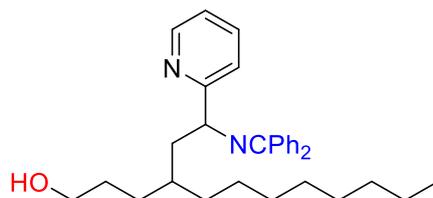
4-(2-((tert-butyldimethylsilyl)oxy)ethyl)-6-((diphenylmethylene)amino)-6-(pyridin-2-yl)hexan-1-ol
(**21**)

Compound **21** was prepared following the general procedure as a colorless oil (49.6 mg, 47%, 1.1:1 d.r.);

1H NMR (400 MHz, $CDCl_3$) δ 8.47 (dt, $J = 4.9, 1.4$ Hz, 1H), 7.77 – 7.67 (m, 4H), 7.45 – 7.33 (m, 6H), 7.19 – 7.03 (m, 3H), 4.81 – 4.68 (m, 1H), 3.56 – 3.42 (m, 4H), 1.98 – 1.70 (m, 2H), 1.49 – 1.26 (m, 7H), 0.81 (s, 9H), -0.05 (d, $J = 4.9$ Hz, 6H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.63, 164.22, 148.34, 139.91, 136.87, 136.63, 130.04, 128.60, 128.56, 128.49, 128.04, 127.75, 122.16, 121.91, 65.93, 61.48, 60.80, 43.13, 36.73, 29.27, 28.73, 28.37, 25.89, 18.17, -5.40, -5.41;

HRMS (ESI) m/z calcd for $C_{32}H_{45}N_2O_2Si^+$ ($M+H$) $^+$ 517.3245, found 517.3244.



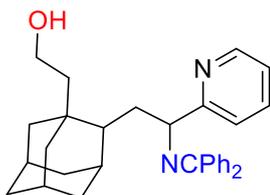
4-(2-((diphenylmethylene)amino)-2-(pyridin-2-yl)ethyl)dodecan-1-ol (**22**)

Compound **22** was prepared following the general procedure as a yellow oil (56.5 mg, 60%, 1:1 d.r.);

1H NMR (400 MHz, $CDCl_3$) δ 8.55 – 8.45 (m, 1H), 7.77 – 7.53 (m, 4H), 7.48 – 7.32 (m, 6H), 7.18 – 7.05 (m, 3H), 4.80 – 4.63 (m, 1H), 3.47 (m, 2H), 1.96 – 1.74 (m, 2H), 1.46 (q, $J = 6.7$ Hz, 2H), 1.27 – 0.99 (m, 15H), 0.94 – 0.85 (m, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.70, 167.56, 164.52, 164.47, 148.79, 148.42, 140.04, 139.99, 136.79, 136.68, 136.62, 136.60, 130.02, 128.70, 128.63, 128.57, 128.46, 128.41, 128.05, 128.03, 127.94, 127.82, 122.11, 121.97, 121.85, 121.75, 66.26, 66.10, 63.26, 62.05, 43.11, 42.83, 34.03, 33.77, 33.47, 32.89, 31.95, 31.90, 30.02, 29.90, 29.67, 29.59, 29.50, 29.41, 29.35, 29.08, 28.98, 28.84, 26.51, 26.32, 22.72, 22.69, 14.17, 14.15;

HRMS (ESI) m/z calcd for $C_{32}H_{43}N_2O^+$ ($M+H$) $^+$ 471.3370, found 471.3376.



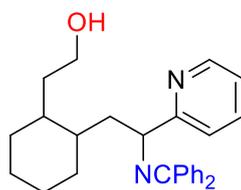
2-((1*r*,3*s*,5*R*,7*S*)-2-(2-((diphenylmethylene)amino)-2-(pyridin-2-yl)ethyl)adamantan-1-yl)ethan-1-ol (**23**)

Compound **23** was prepared following the general procedure as a yellow oil (50.2 mg, 54%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 8.50 (dd, *J* = 5.1, 1.8 Hz, 1H), 7.79 – 7.61 (m, 3H), 7.54 – 7.34 (m, 7H), 7.14 (dd, *J* = 7.4, 4.9 Hz, 1H), 7.04 – 6.93 (m, 2H), 4.59 (dd, *J* = 10.7, 3.0 Hz, 1H), 3.99 – 3.85 (m, 1H), 3.65 (dt, *J* = 11.3, 6.7 Hz, 1H), 2.35 – 2.25 (m, 1H), 1.99 – 1.79 (m, 4H), 1.72 – 1.53 (m, 7H), 1.47 – 1.33 (m, 3H), 1.29 – 1.17 (m, 3H);

¹³C NMR (101 MHz, CDCl₃) δ 169.15, 164.68, 148.80, 139.45, 136.68, 136.65, 130.29, 128.75, 128.56, 128.36, 128.22, 127.53, 121.82, 121.72, 65.66, 57.80, 44.17, 43.88, 42.06, 39.02, 38.67, 37.84, 35.79, 34.45, 31.34, 29.35, 28.60, 28.46;

HRMS (ESI) *m/z* calcd for C₃₂H₃₇N₂O⁺ (M+H)⁺ 465.2900, found 465.2901.



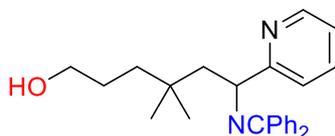
2-(2-(2-((diphenylmethylene)amino)-2-(pyridin-2-yl)ethyl)cyclohexyl)ethan-1-ol (**24**)

Compound **24** was prepared following the general procedure as a yellow oil (58.5 mg, 71%, *trans:cis* = 3.5:1);

¹H NMR (400 MHz, CDCl₃) δ 8.58 – 8.47 (m, 1H), 7.79 – 7.32 (m, 10H), 7.18 – 6.98 (m, 3H), 4.77 – 4.58 (m, 1H), 3.73 – 3.43 (m, 2H), 2.50 – 2.28 (m, 1H), 1.90 – 1.44 (m, 7H), 1.32 – 1.05 (m, 5H), 0.91 – 0.76 (m, 1H);

¹³C NMR (101 MHz, CDCl₃) δ 168.13, 168.01, 167.23, 164.73, 164.55, 163.80, 148.80, 148.68, 148.61, 139.93, 139.81, 136.82, 136.75, 136.65, 136.62, 136.59, 130.10, 130.00, 128.70, 128.66, 128.63, 128.58, 128.51, 128.50, 128.47, 128.41, 128.37, 128.11, 128.09, 128.03, 127.82, 127.79, 122.44, 121.91, 121.77, 121.74, 67.00, 65.94, 65.38, 61.48, 60.77, 60.38, 42.39, 42.35, 38.67, 38.32, 37.37, 36.93, 36.42, 32.34, 31.71, 31.65, 31.23, 28.81, 28.03, 25.74, 25.67, 25.50, 25.35;

HRMS (ESI) *m/z* calcd for C₂₈H₃₃N₂O⁺ (M+H)⁺ 413.2587, found 413.2589.



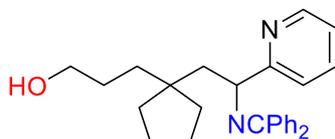
6-((diphenylmethylene)amino)-4,4-dimethyl-6-(pyridin-2-yl)hexan-1-ol (**25**)

Compound **25** was prepared following the general procedure as a colorless oil (42.5 mg, 55%);

¹H NMR (400 MHz, CDCl₃) δ 8.51 (dd, *J* = 4.9, 2.2 Hz, 1H), 7.73 – 7.60 (m, 3H), 7.47 – 7.32 (m, 7H), 7.16 – 7.10 (m, 1H), 7.06 – 6.97 (m, 2H), 4.74 (dd, *J* = 8.0, 4.3 Hz, 1H), 3.55 – 3.33 (m, 2H), 2.09 – 1.92 (m, 2H), 1.56 – 1.44 (m, 2H), 1.14 (tt, *J* = 11.1, 7.2 Hz, 2H), 0.80 (s, 6H);

^{13}C NMR (101 MHz, CDCl_3) δ 166.88, 165.38, 148.74, 139.98, 136.79, 136.61, 129.99, 128.69, 128.60, 128.35, 128.05, 127.78, 122.21, 121.68, 65.95, 63.53, 49.75, 38.08, 33.20, 28.18, 28.12, 27.44.;

HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{31}\text{N}_2\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 387.2431, found 387.2430.



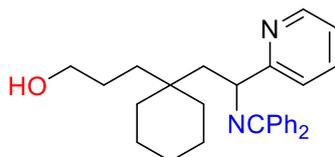
3-(1-(2-((diphenylmethylene)amino)-2-(pyridin-2-yl)ethyl)cyclopentyl)propan-1-ol (26)

Compound **26** was prepared following the general procedure as a colorless oil (48.7 mg, 59%).;

^1H NMR (400 MHz, CDCl_3) δ 8.49 (dd, $J = 5.0, 2.0$ Hz, 1H), 7.76 – 7.69 (m, 2H), 7.64 (td, $J = 7.6, 1.9$ Hz, 1H), 7.50 – 7.33 (m, 7H), 7.17 – 7.10 (m, 1H), 7.08 – 7.00 (m, 2H), 4.72 (dd, $J = 7.8, 4.6$ Hz, 1H), 3.44 – 3.27 (m, 2H), 2.14 – 2.03 (m, 2H), 1.62 – 1.39 (m, 8H), 1.29 – 1.16 (m, 4H).;

^{13}C NMR (101 MHz, CDCl_3) δ 166.86, 165.25, 148.70, 139.93, 136.72, 136.60, 130.01, 128.68, 128.58, 128.34, 128.07, 127.91, 122.22, 121.73, 66.28, 63.43, 46.67, 44.97, 38.64, 38.25, 33.72, 27.98, 24.32, 24.00.;

HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{33}\text{N}_2\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 413.2587, found 413.2588.



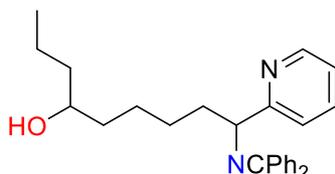
3-(1-(2-((diphenylmethylene)amino)-2-(pyridin-2-yl)ethyl)cyclohexyl)propan-1-ol (27)

Compound **27** was prepared following the general procedure as a yellow oil (52.9 mg, 62%).;

^1H NMR (400 MHz, CDCl_3) δ 8.49 – 8.43 (m, 1H), 7.70 – 7.57 (m, 3H), 7.47 – 7.30 (m, 7H), 7.18 – 7.06 (m, 1H), 6.99 (dd, $J = 6.5, 2.8$ Hz, 2H), 4.70 (dd, $J = 7.9, 3.9$ Hz, 1H), 3.48 – 3.22 (m, 2H), 2.26 (t, $J = 5.7$ Hz, 1H), 2.16 (dd, $J = 14.5, 7.9$ Hz, 1H), 1.93 (dd, $J = 14.5, 4.0$ Hz, 1H), 1.47 – 1.26 (m, 9H), 1.22 – 1.05 (m, 5H).;

^{13}C NMR (101 MHz, CDCl_3) δ 166.79, 165.62, 148.66, 139.98, 136.78, 136.65, 129.99, 128.68, 128.60, 128.33, 128.06, 127.83, 122.23, 121.68, 65.23, 63.45, 36.71, 36.65, 35.55, 26.44, 26.24, 21.68, 21.61.;

HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{35}\text{N}_2\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 427.2744, found 427.2749.



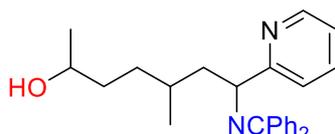
9-((diphenylmethylene)amino)-9-(pyridin-2-yl)nonan-4-ol (28)

Compound **28** was prepared following the general procedure as a yellow oil (41.6 mg, 52%).;

¹H NMR (400 MHz, CDCl₃) δ 8.51 (dt, *J* = 4.6, 2.2 Hz, 1H), 7.75 – 7.64 (m, 3H), 7.60 – 7.52 (m, 1H), 7.39 (m, 6H), 7.11 (m, 3H), 4.67 – 4.51 (m, 1H), 3.60 – 3.43 (m, 1H), 2.02 – 1.91 (m, 2H), 1.47 – 1.20 (m, 10H), 0.97 – 0.86 (m, 3H).;

¹³C NMR (101 MHz, CDCl₃) δ 167.77, 167.75, 164.17, 148.80, 148.73, 139.99, 136.78, 136.76, 136.55, 136.51, 130.02, 128.65, 128.46, 128.43, 128.05, 127.80, 127.78, 121.82, 121.79, 121.73, 121.72, 71.58, 71.29, 67.97, 67.84, 39.60, 39.55, 38.31, 37.37, 37.31, 26.29, 26.06, 25.51, 25.28, 18.84, 14.14.;

HRMS (ESI) *m/z* calcd for C₂₇H₃₃N₂O⁺ (M+H)⁺ 401.2587, found 401.2589.



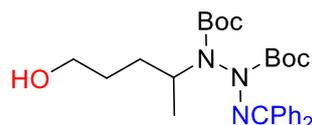
N-(2-((diphenylmethylene)amino)-2-(perfluorophenyl)ethyl)-*N*,4-dimethylbenzenesulfonamide (**29**)

Compound **29** was prepared following the general procedure as a yellow oil (57.9 mg, 75%, 1:1 d.r.);

¹H NMR (400 MHz, CDCl₃) δ 8.51 (dd, *J* = 10.6, 5.4 Hz, 1H), 7.69 – 7.36 (m, 10H), 7.15 – 7.05 (m, 3H), 4.77 – 4.65 (m, 1H), 3.77 – 3.52 (m, 1H), 2.11 – 1.68 (m, 3H), 1.39 – 1.09 (m, 7H), 0.85 – 0.66 (m, 3H).;

¹³C NMR (101 MHz, CDCl₃) δ 167.85, 167.76, 167.61, 164.62, 164.59, 164.38, 148.77, 148.49, 140.01, 136.80, 136.73, 136.65, 136.61, 136.58, 130.03, 130.01, 128.67, 128.64, 128.58, 128.55, 128.53, 128.45, 128.43, 128.38, 128.04, 127.93, 127.87, 127.86, 127.82, 121.99, 121.94, 121.83, 121.81, 121.76, 121.72, 68.45, 68.35, 66.84, 66.35, 66.03, 65.82, 46.15, 45.98, 45.94, 45.79, 36.54, 36.38, 36.21, 33.38, 32.23, 32.13, 29.78, 29.68, 28.55, 23.40, 23.36, 23.25, 20.31, 19.66, 19.43.

HRMS (ESI) *m/z* calcd for C₂₆H₃₁N₂O⁺ (M+H)⁺ 387.2431, found 387.2437.



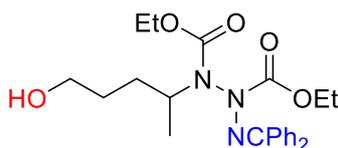
di-tert-butyl 3-(diphenylmethylene)-1-(5-hydroxypentan-2-yl)triazane-1,2-dicarboxylate (**32**)

Compound **32** was prepared following the general procedure as a colorless oil (69.6 mg, 70%);

¹H NMR (400 MHz, CDCl₃) δ 7.53 (d, *J* = 7.7 Hz, 2H), 7.44 – 7.21 (m, 8H), 4.24 (dq, *J* = 84.3, 6.1 Hz, 1H), 3.49 (dt, *J* = 20.2, 6.4 Hz, 2H), 2.37 (s, 1H), 1.50 (s, 13H), 1.27 – 1.02 (m, 12H).;

¹³C NMR (101 MHz, CDCl₃) δ 154.71, 153.89, 152.39, 138.08, 136.35, 130.40, 130.18, 128.99, 128.86, 128.47, 128.08, 127.98, 127.54, 81.36, 62.73, 62.45, 53.05, 46.16, 31.47, 29.88, 29.57, 28.39, 27.81, 27.75, 18.77, 11.33.;

HRMS (ESI) *m/z* calcd for C₂₈H₄₀N₃O₅⁺ (M+H)⁺ 498.2962, found 498.2965.



diethyl 3-(diphenylmethylene)-1-(5-hydroxypentan-2-yl)triazane-1,2-dicarboxylate (33)

Compound **33** was prepared following the general procedure as a colorless oil (59.2 mg, 67%);

¹H NMR (400 MHz, CDCl₃) δ 7.56 (d, *J* = 7.0 Hz, 2H), 7.47 – 7.28 (m, 8H), 4.31 – 3.82 (m, 5H), 3.53 (dt, *J* = 17.9, 6.3 Hz, 2H), 1.81 – 1.54 (m, 4H), 1.31 – 1.06 (m, 9H);

¹³C NMR (101 MHz, CDCl₃) δ 155.48, 137.84, 135.96, 130.66, 130.45, 129.09, 129.04, 128.84, 128.33, 128.27, 128.12, 128.06, 127.80, 127.75, 62.99, 62.69, 62.53, 62.33, 31.52, 29.80, 14.63, 14.19.;

HRMS (ESI) *m/z* calcd for C₂₄H₃₂N₃O₅⁺ (M+H)⁺ 442.2336, found 442.2337.



diisopropyl 3-(diphenylmethylene)-1-(5-hydroxypentan-2-yl) triazane-1,2-dicarboxylate (34)

Compound **34** was prepared following the general procedure as a colorless oil (54.4 mg, 58%);

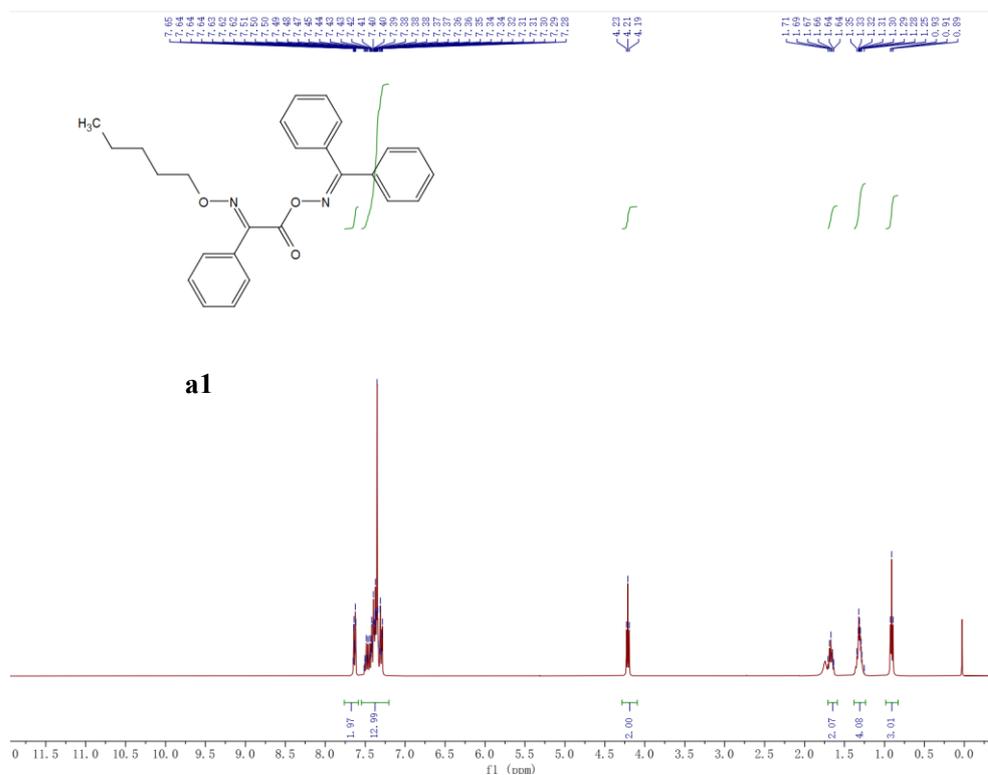
¹H NMR (400 MHz, CDCl₃) δ 7.54 (d, *J* = 7.7 Hz, 2H), 7.48 – 7.35 (m, 6H), 7.34 – 7.26 (m, 2H), 5.06 – 4.95 (m, 1H), 4.79 – 3.96 (m, 2H), 3.62 – 3.41 (m, 2H), 1.80 – 1.47 (m, 4H), 1.33 – 0.92 (m, 15H);

¹³C NMR (101 MHz, CDCl₃) δ 155.65, 136.16, 130.53, 130.31, 129.00, 128.72, 128.41, 128.09, 128.02, 127.68, 70.03, 69.99, 62.74, 62.53, 31.53, 29.85, 22.20, 22.12, 22.08, 21.65, 18.48.;

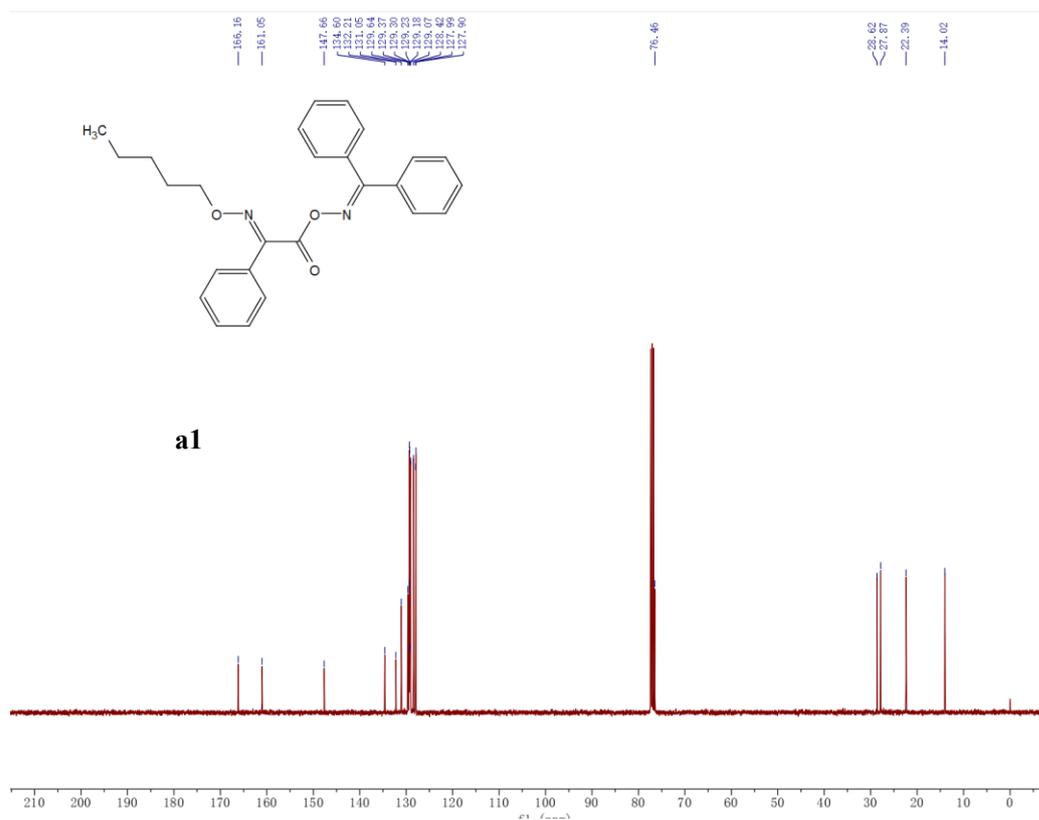
HRMS (ESI) *m/z* calcd for C₂₄H₃₂N₃O₅⁺ (M+H)⁺ 470.2649, found 470.2649.

7. NMR Spectrum

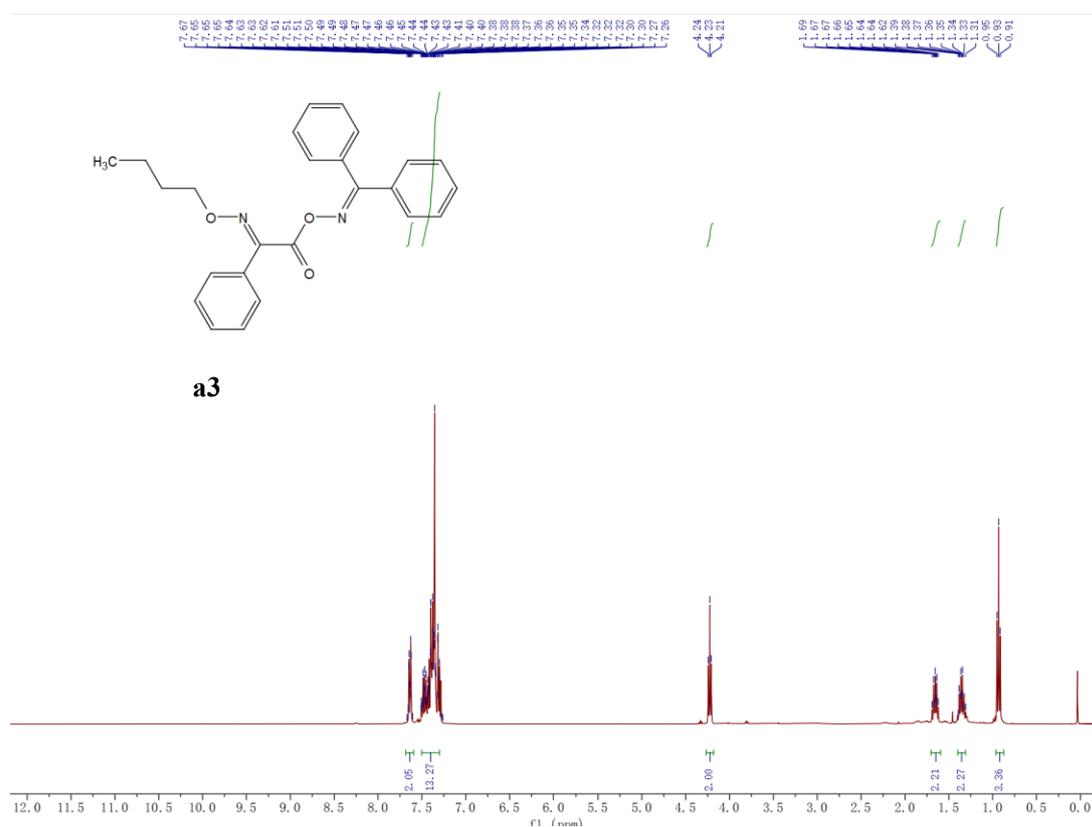
^1H NMR (400 MHz, CDCl_3)



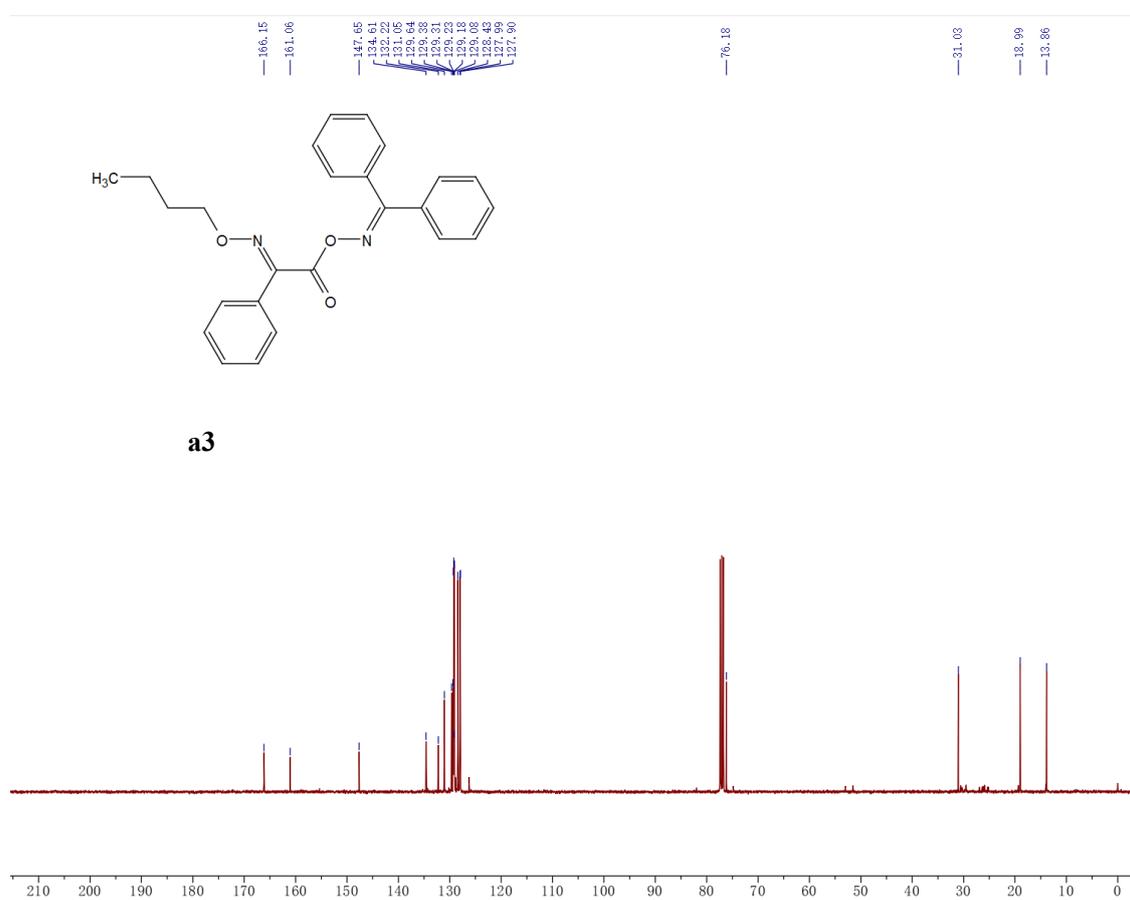
^{13}C NMR (101 MHz, CDCl_3)



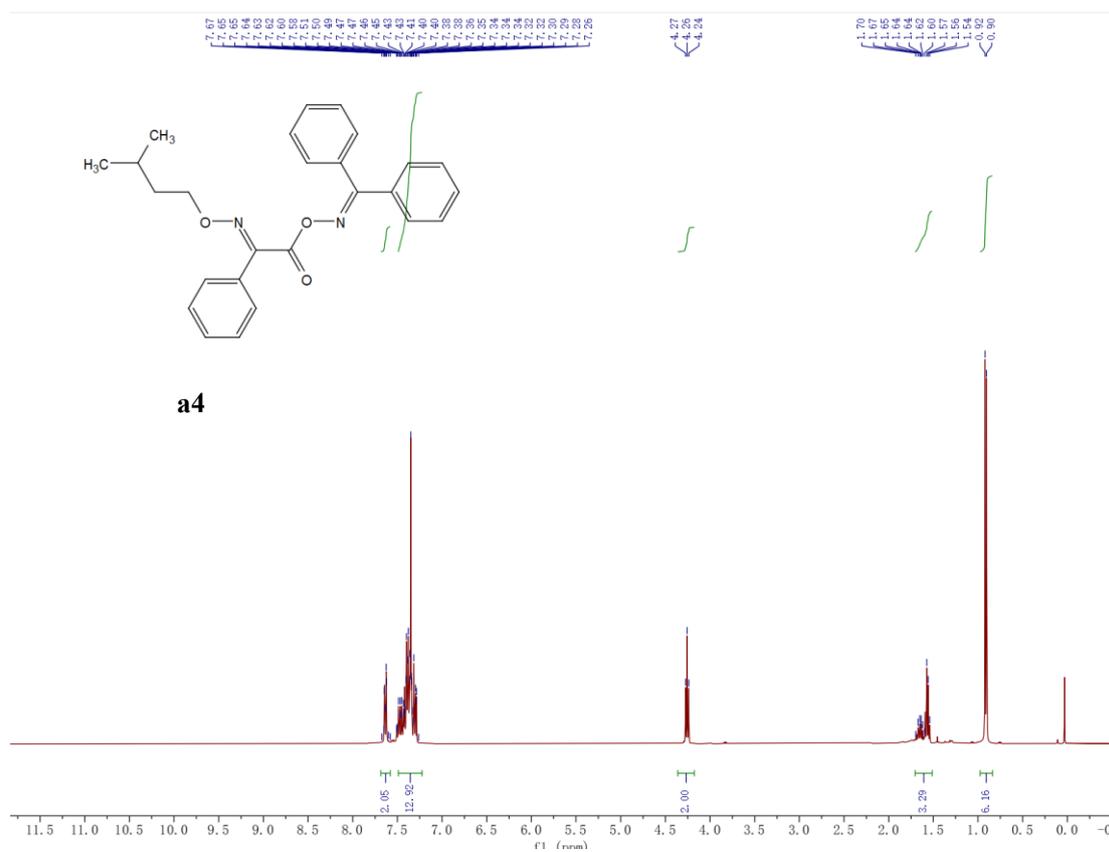
¹H NMR (400 MHz, CDCl₃)



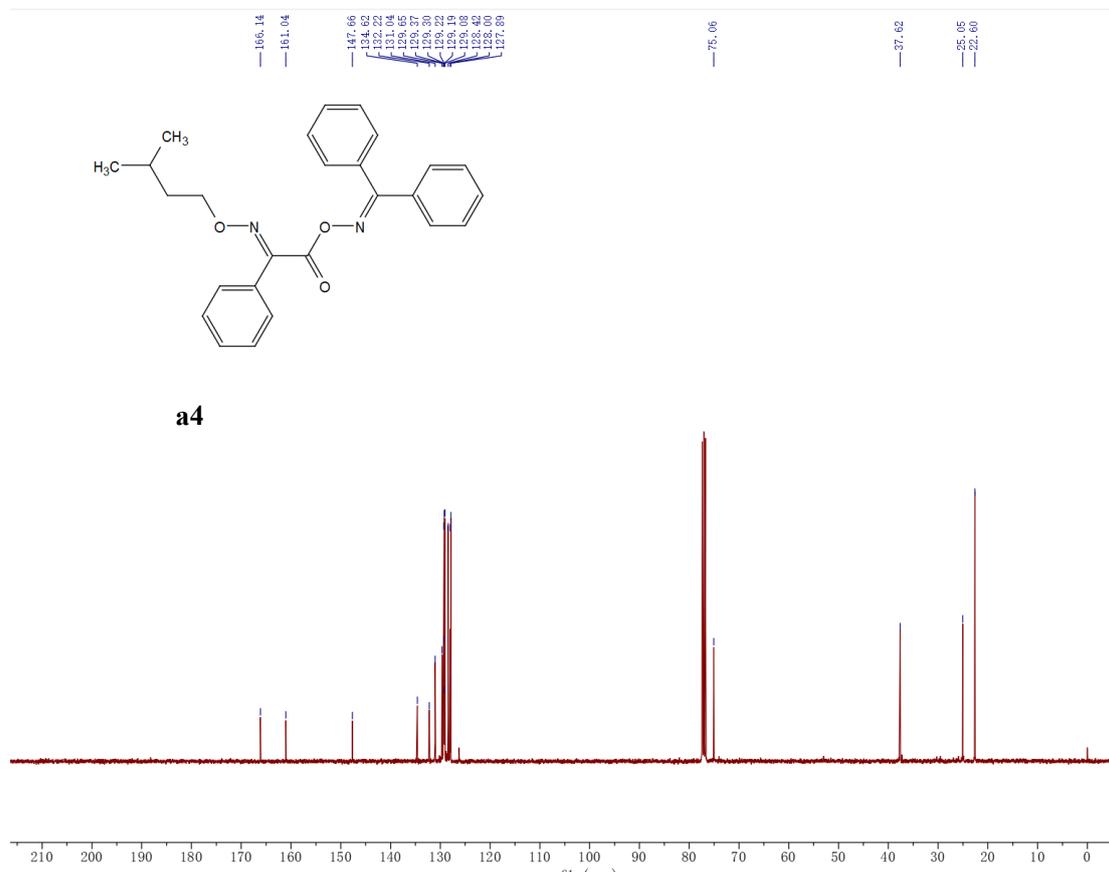
¹³C NMR (101 MHz, CDCl₃)



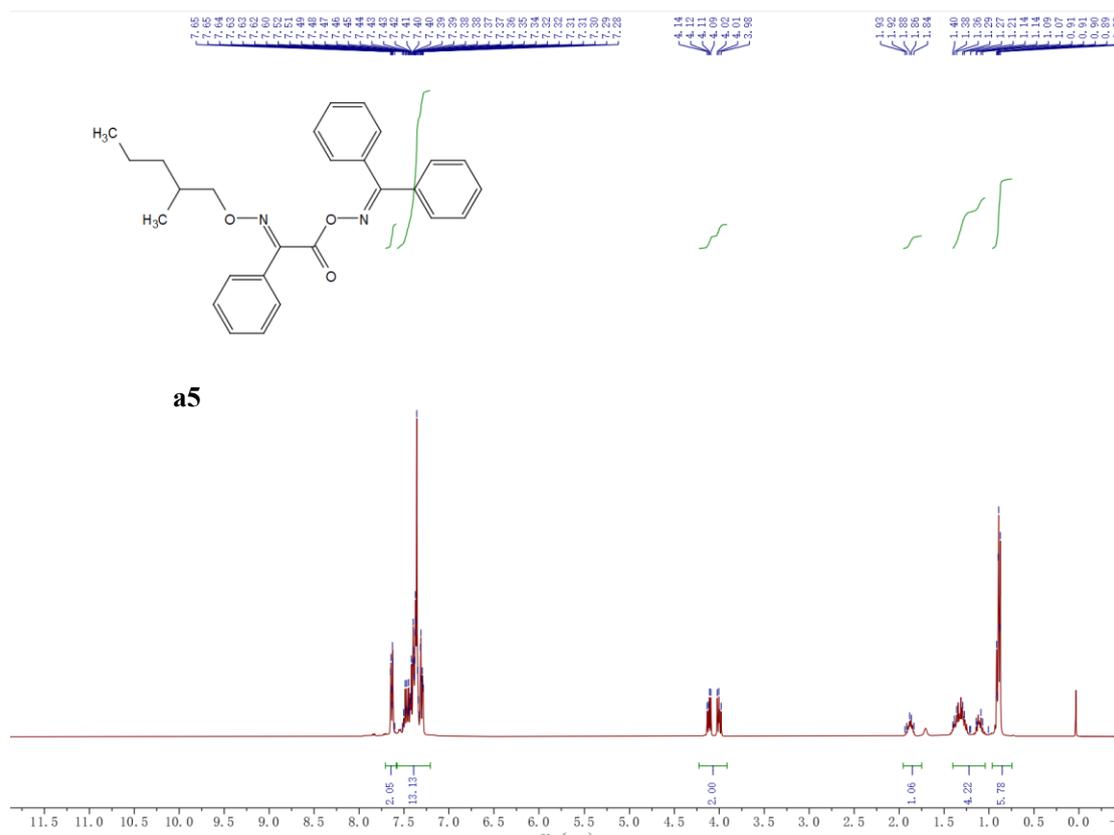
¹H NMR (400 MHz, CDCl₃)



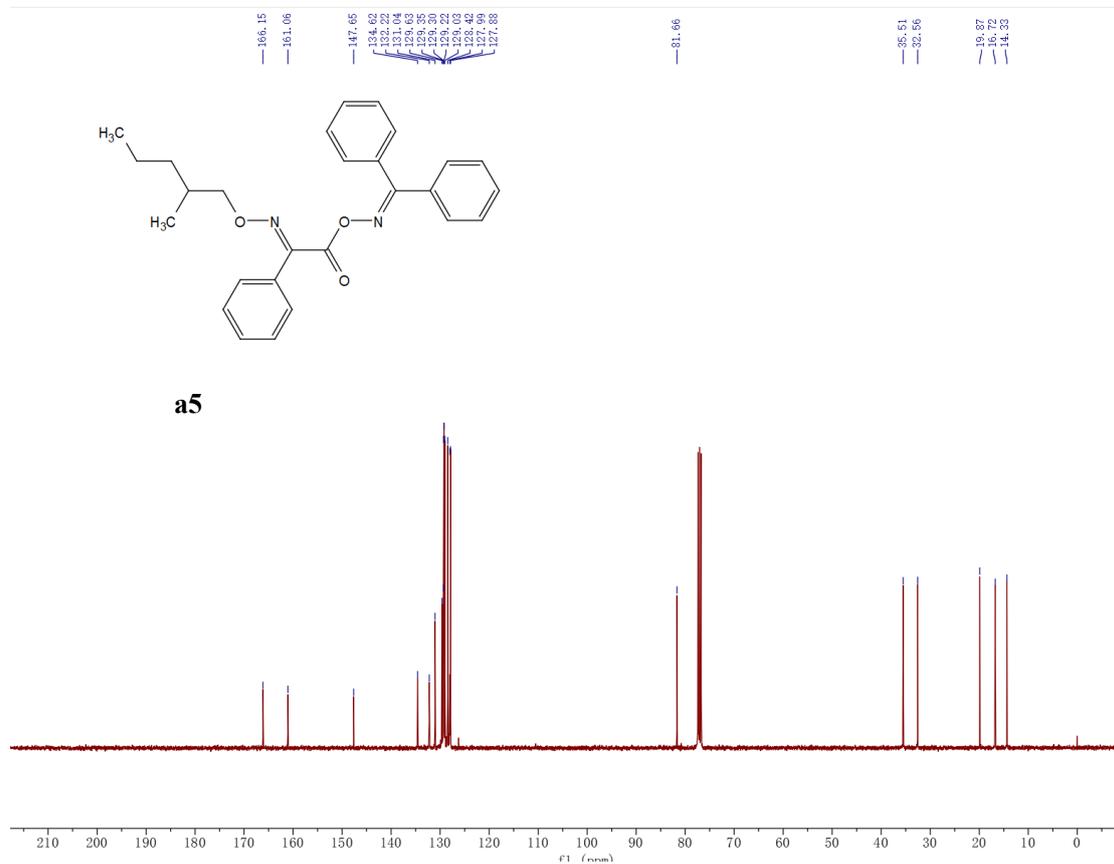
¹³C NMR (101 MHz, CDCl₃)



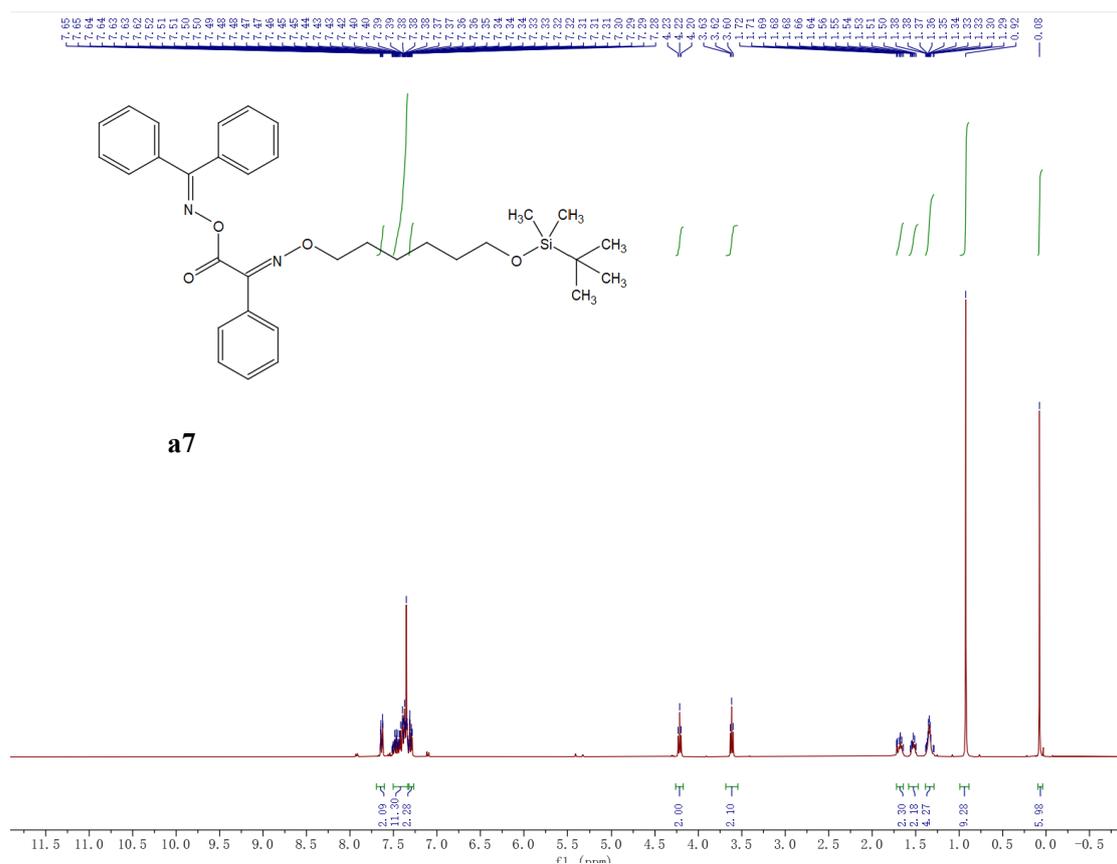
¹H NMR (400 MHz, CDCl₃)



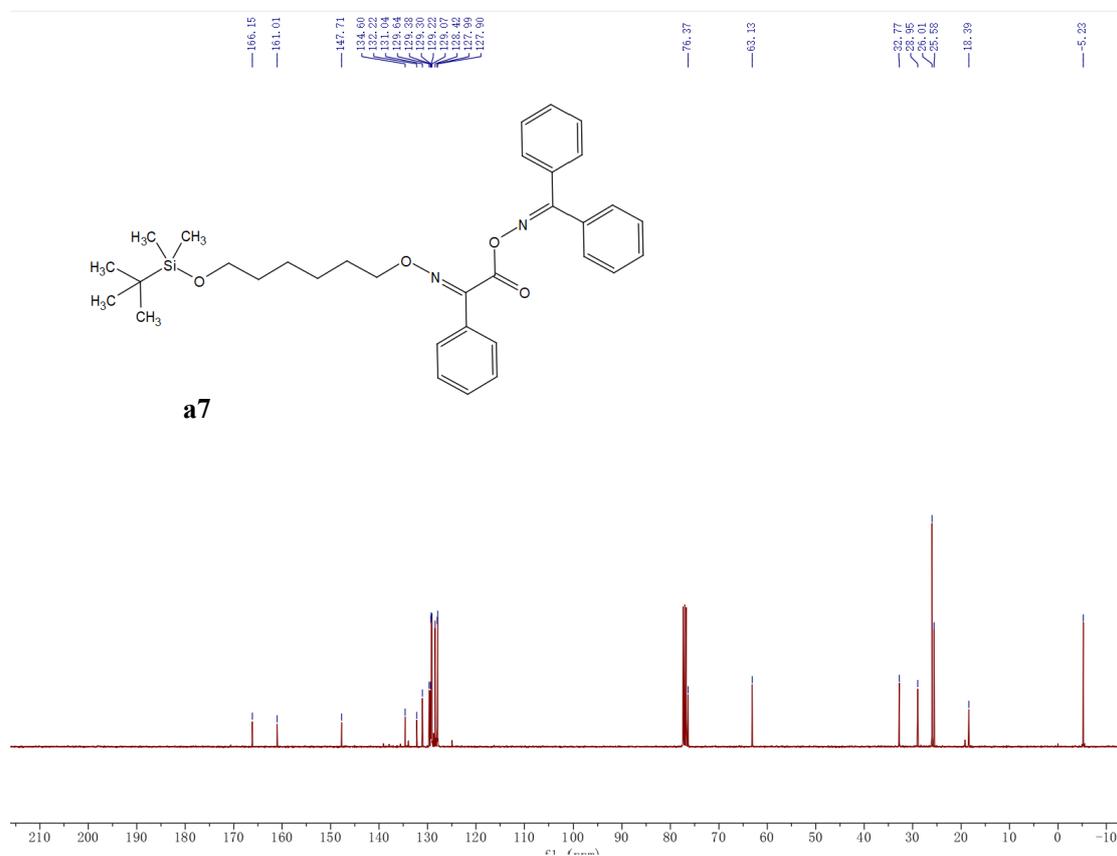
¹³C NMR (101 MHz, CDCl₃)



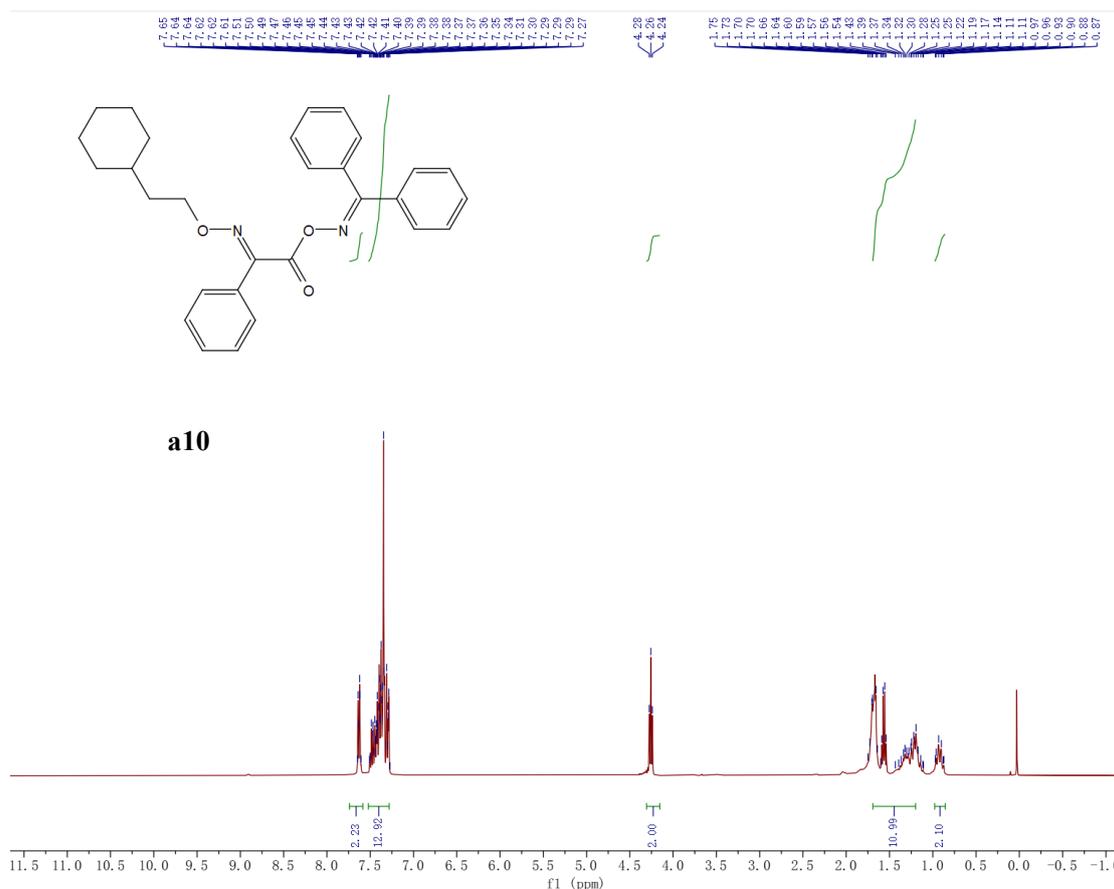
¹H NMR (400 MHz, CDCl₃)



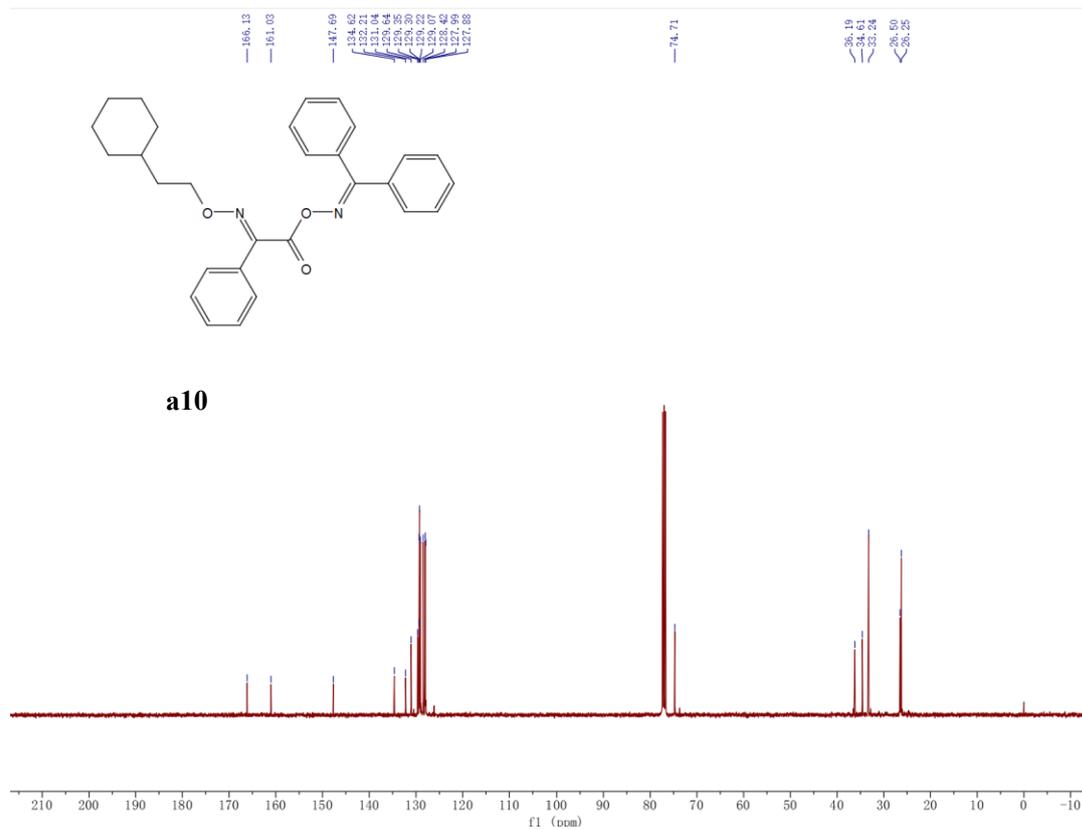
¹³C NMR (101 MHz, CDCl₃)



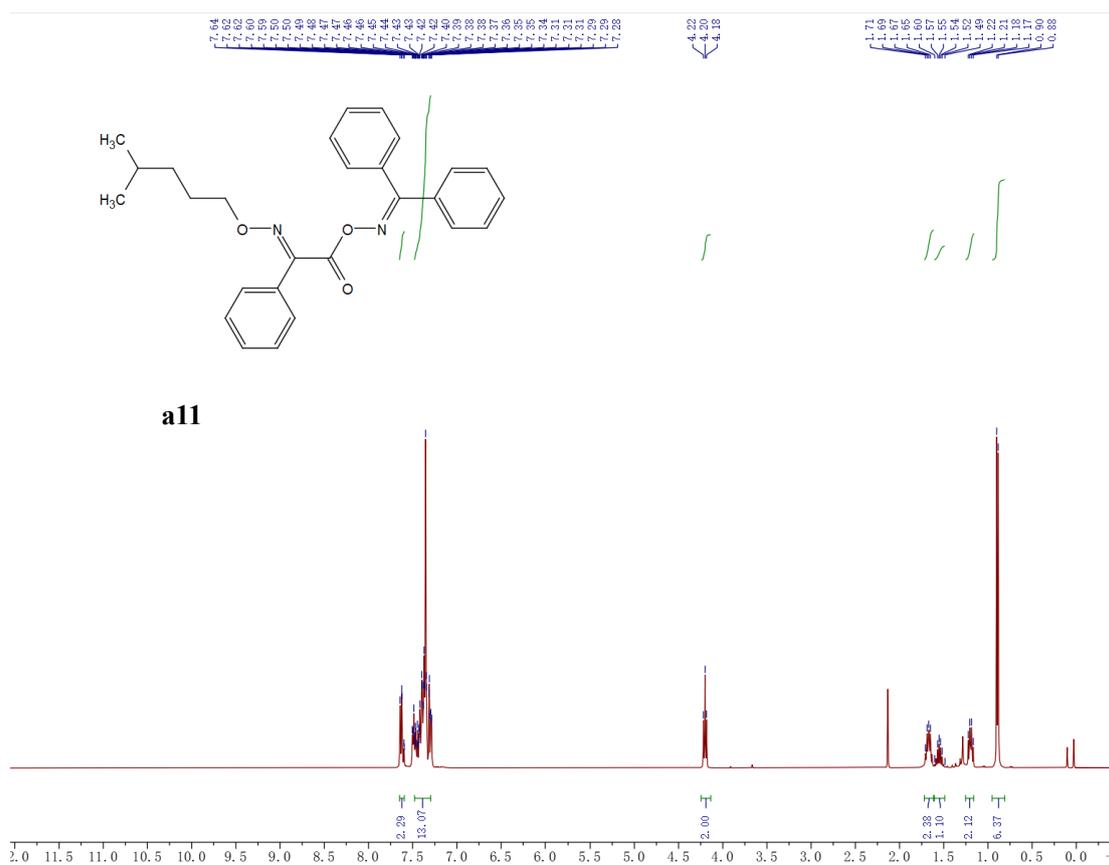
¹H NMR (400 MHz, CDCl₃)



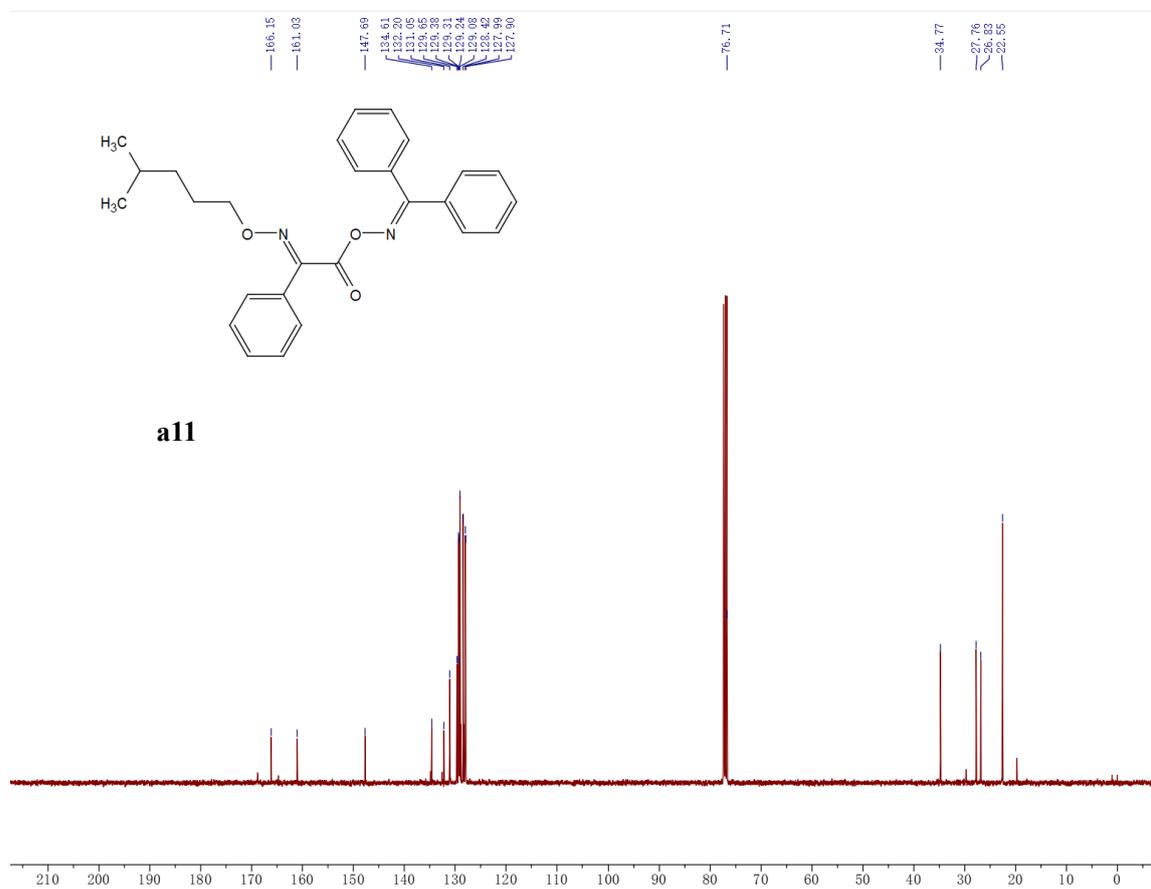
¹³C NMR (101 MHz, CDCl₃)



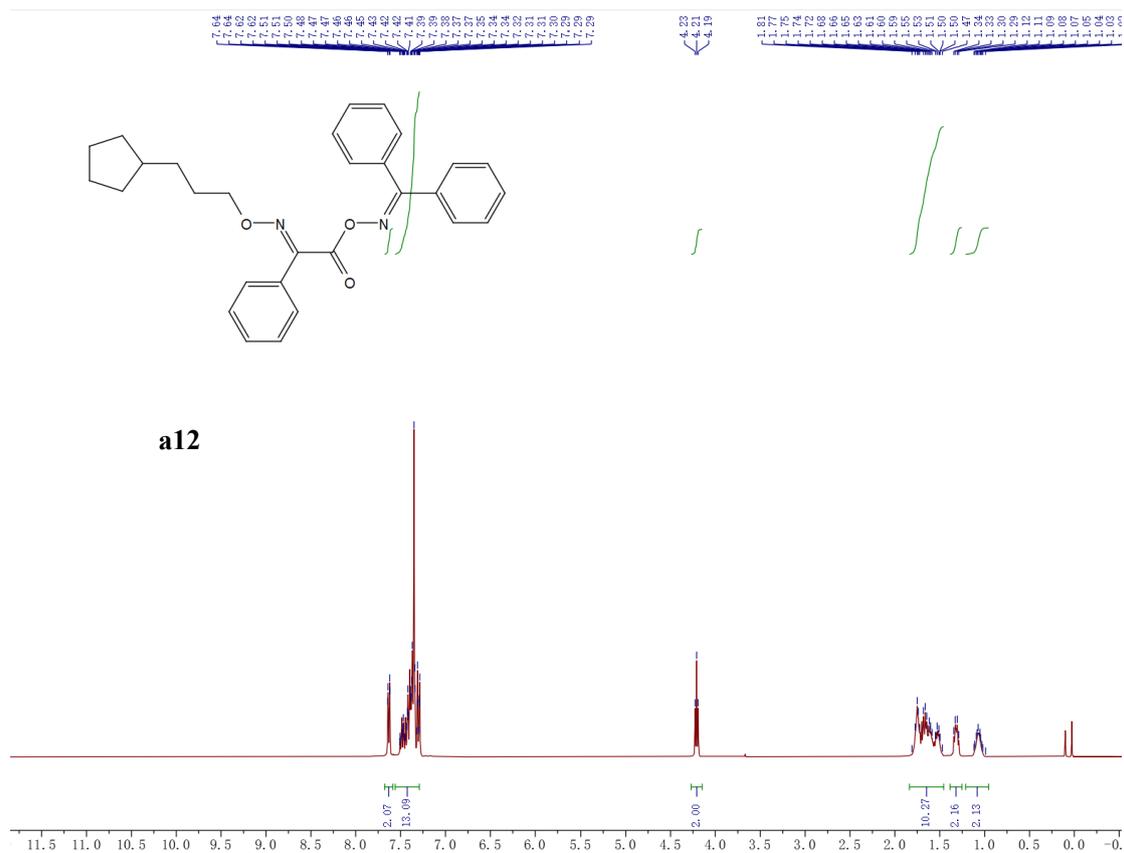
¹H NMR (400 MHz, CDCl₃)



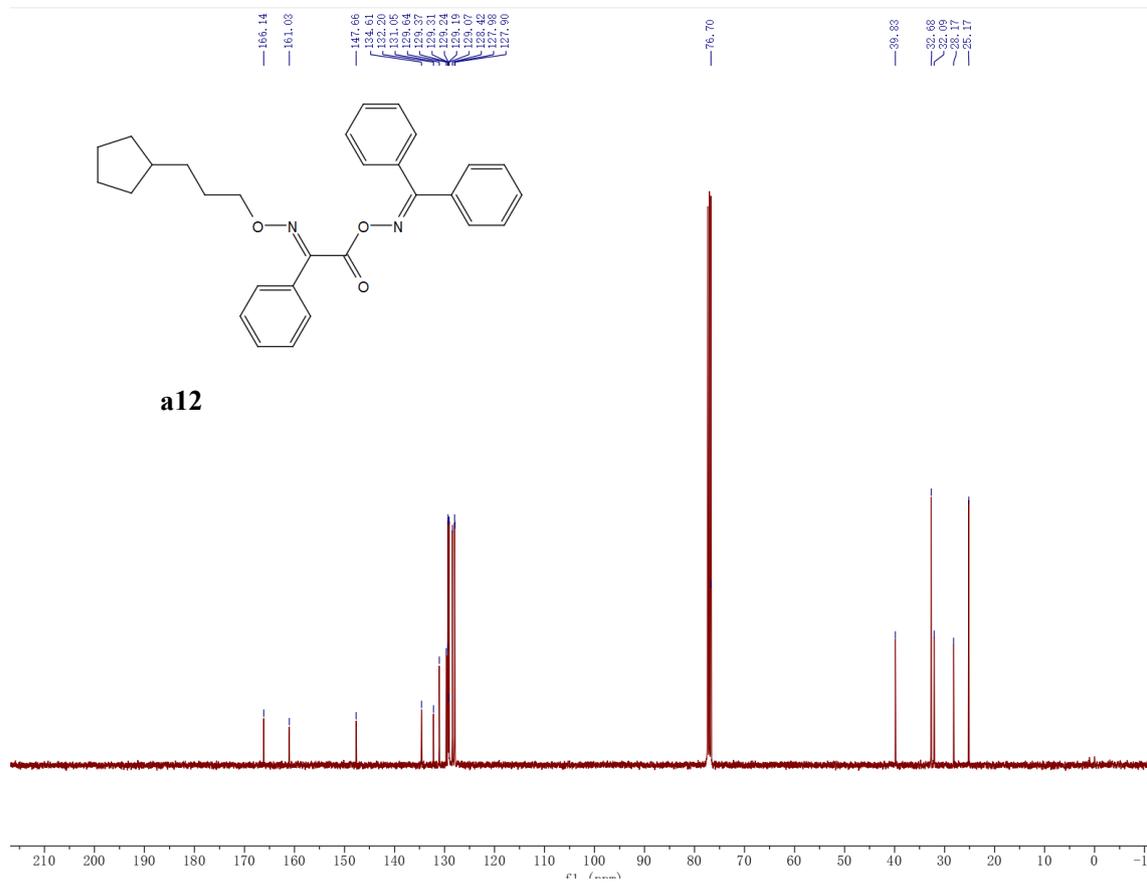
¹³C NMR (101 MHz, CDCl₃)



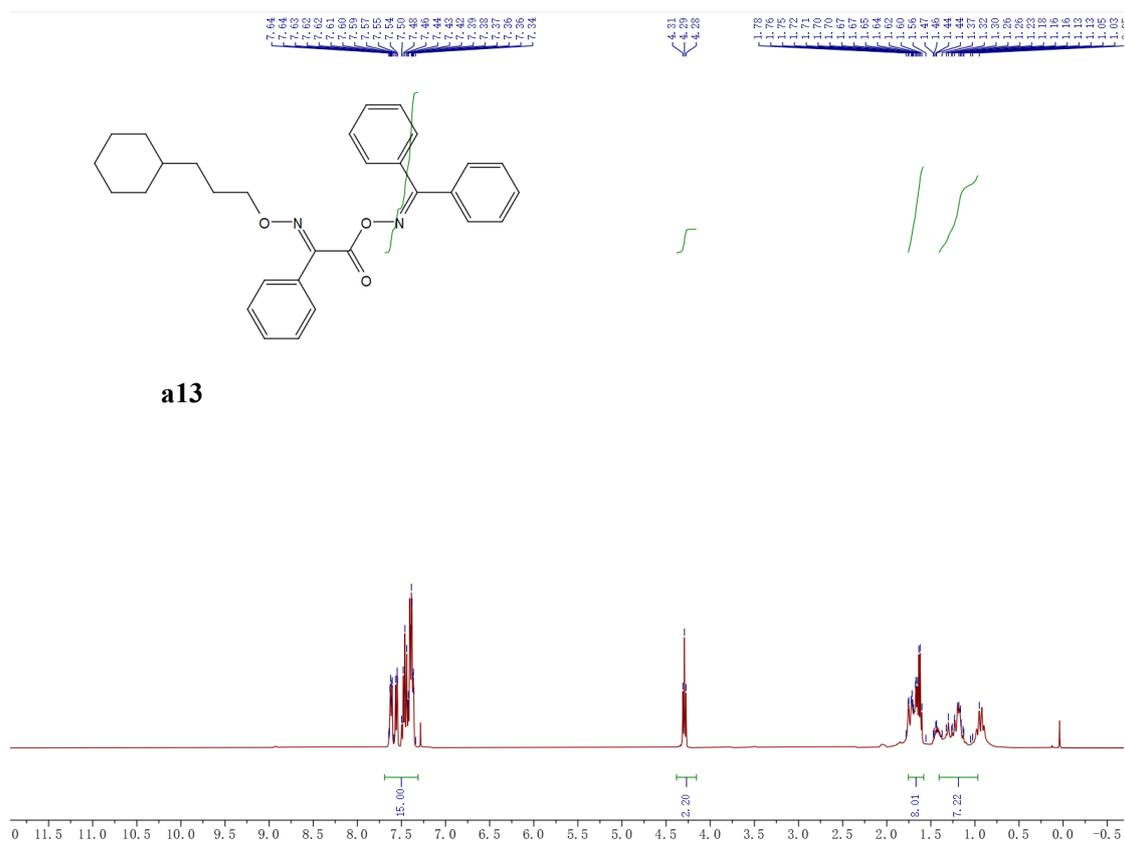
¹H NMR (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)

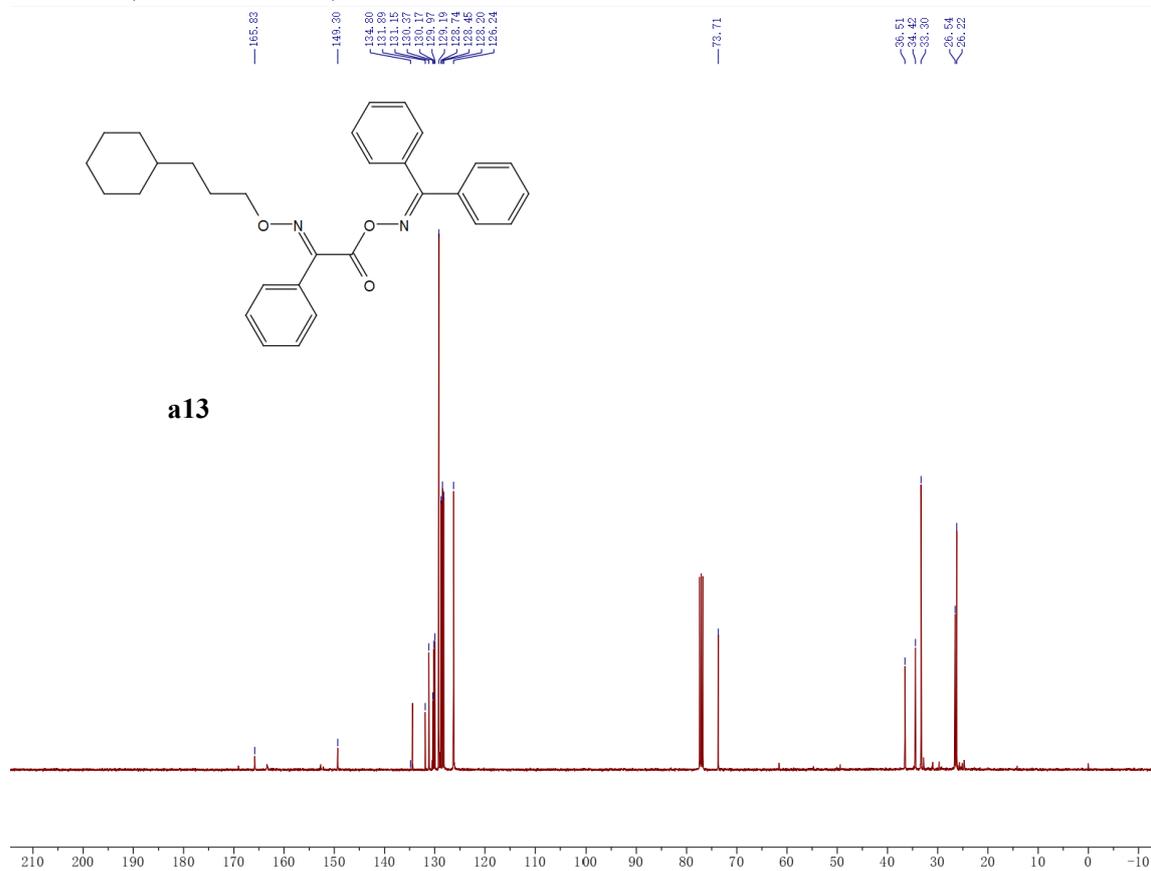


¹H NMR (400 MHz, CDCl₃)



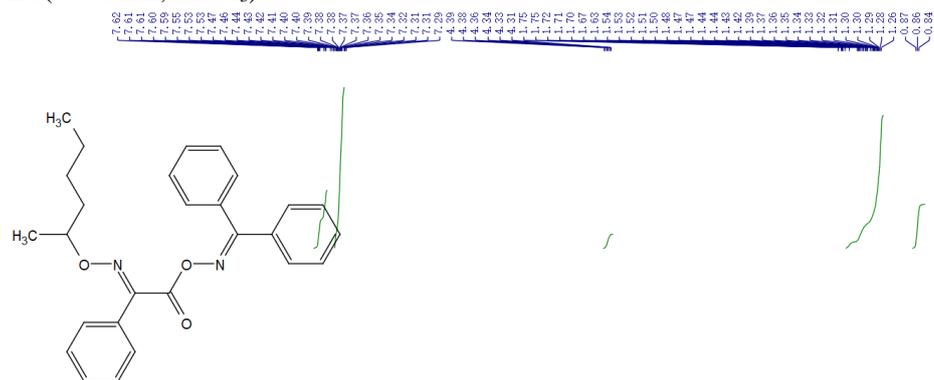
a13

¹³C NMR (101 MHz, CDCl₃)

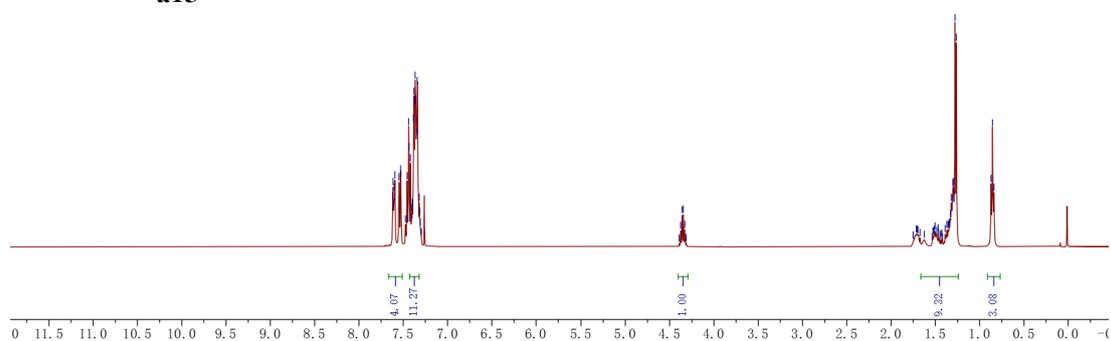


a13

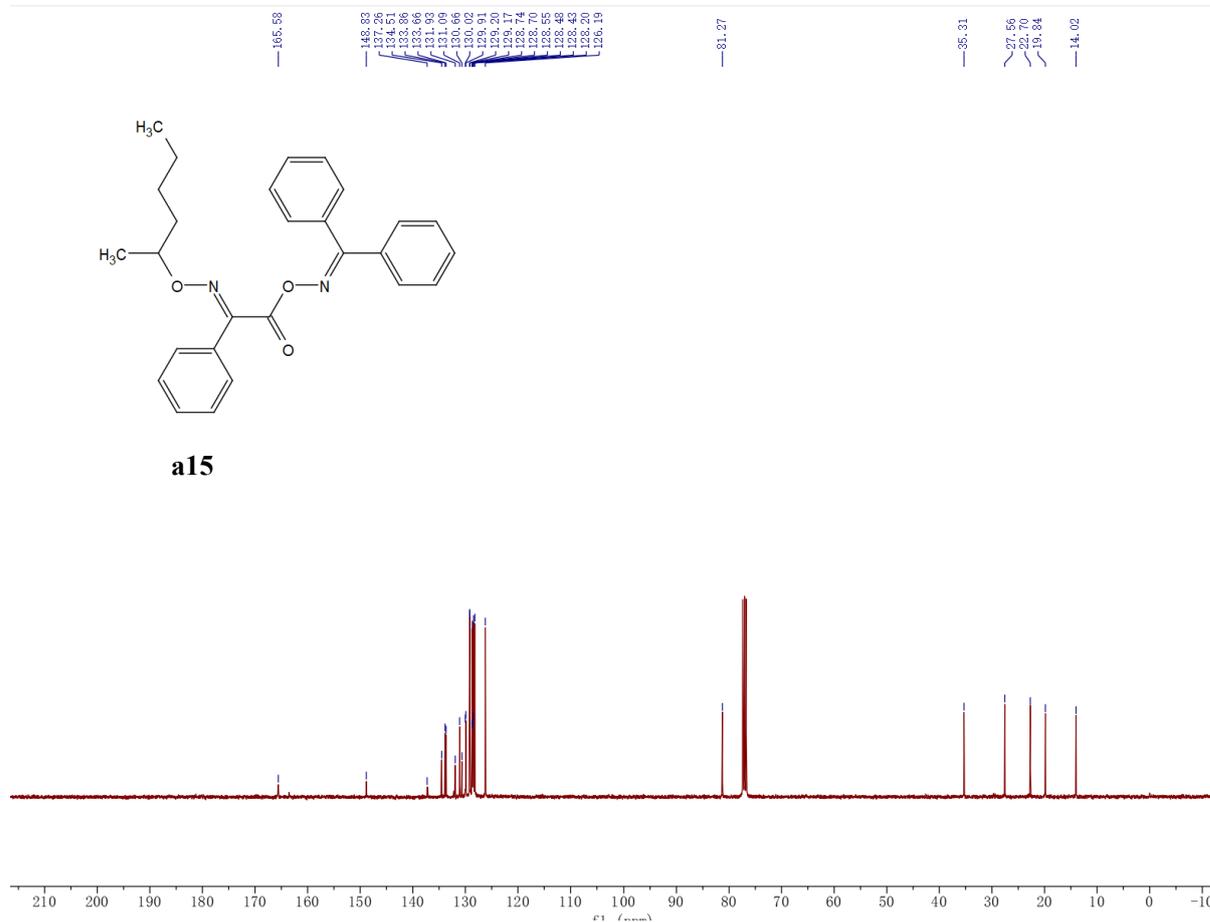
¹H NMR (400 MHz, CDCl₃)



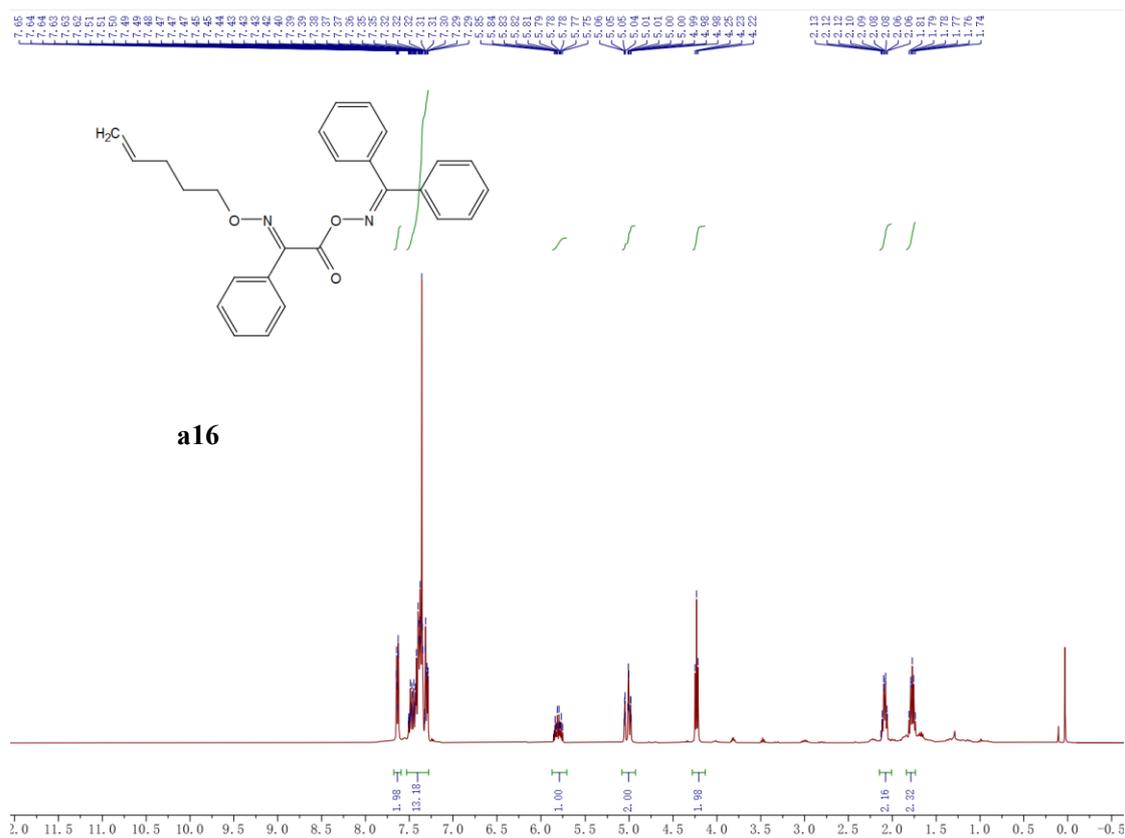
a15



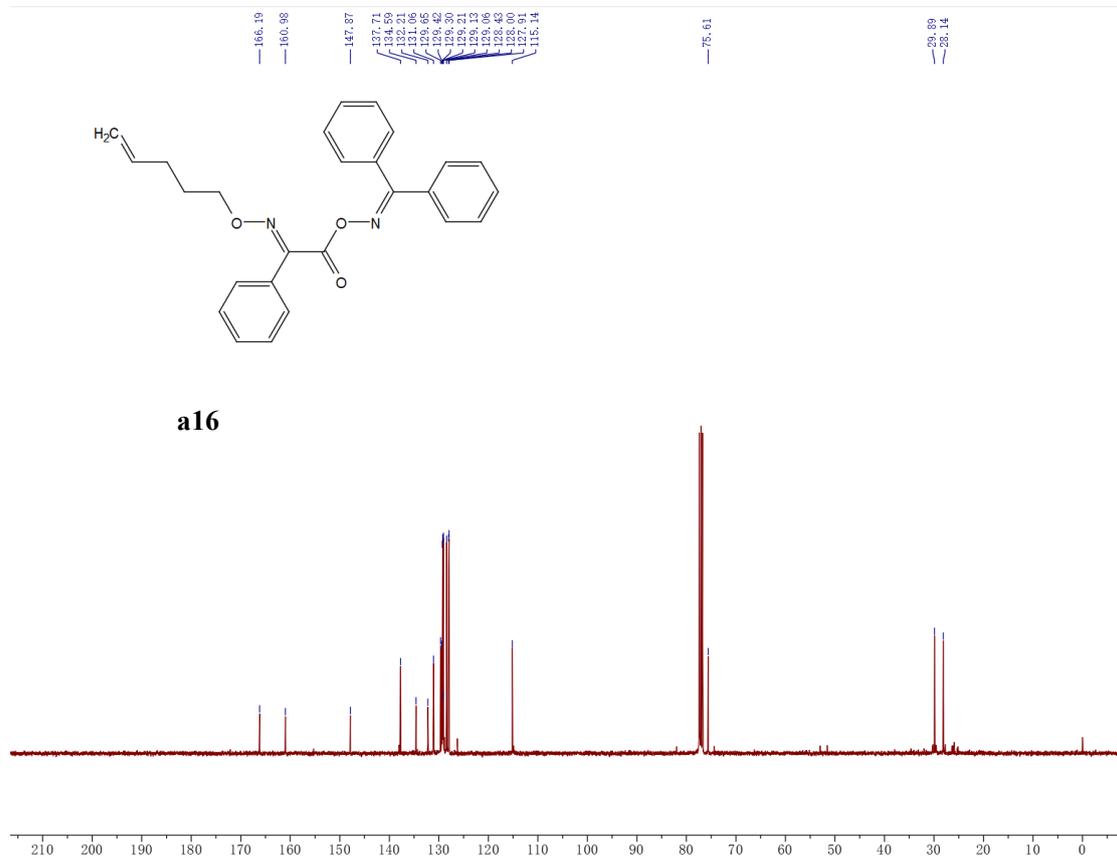
¹³C NMR (101 MHz, CDCl₃)



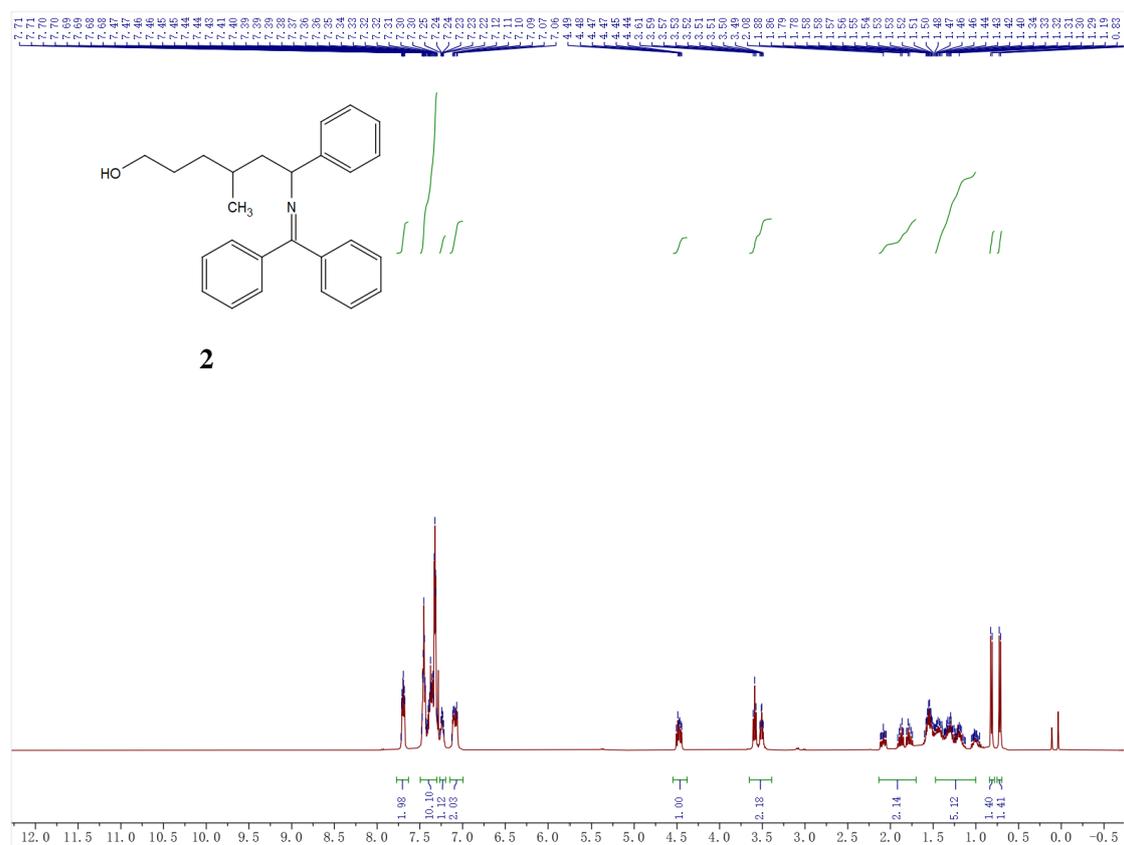
¹H NMR (400 MHz, CDCl₃)



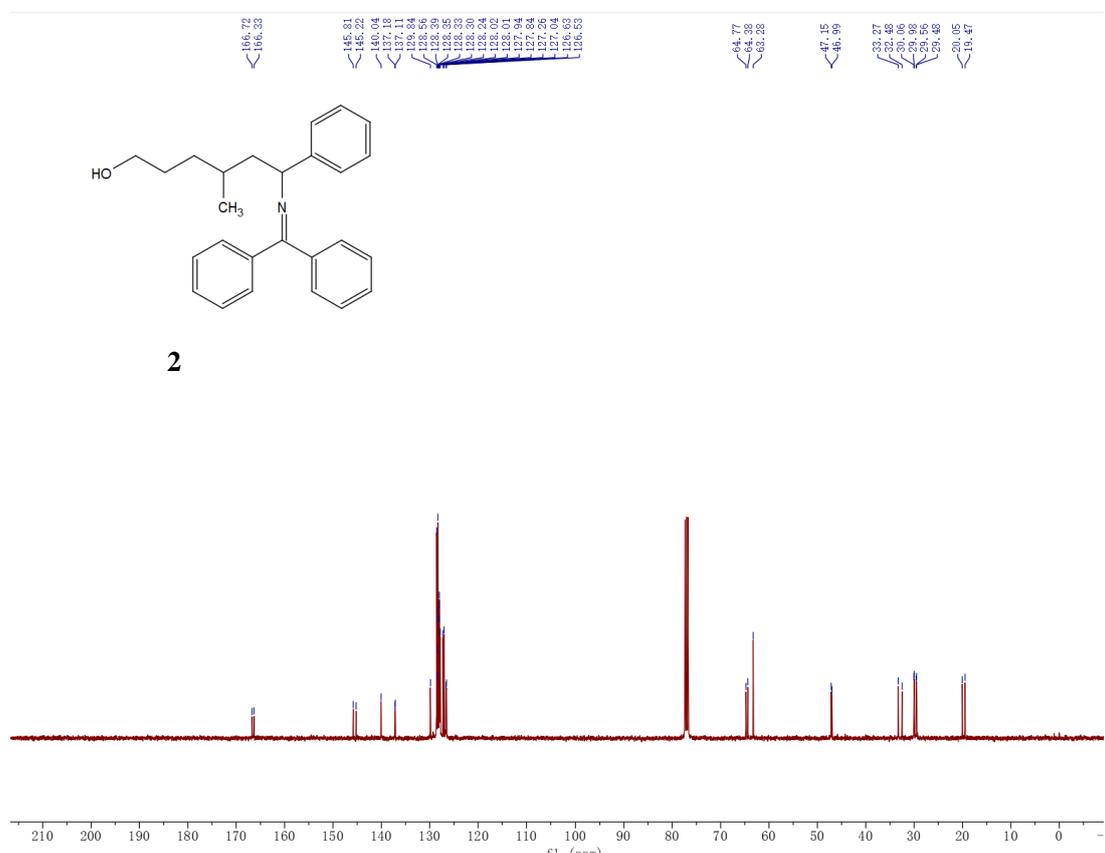
¹³C NMR (101 MHz, CDCl₃)



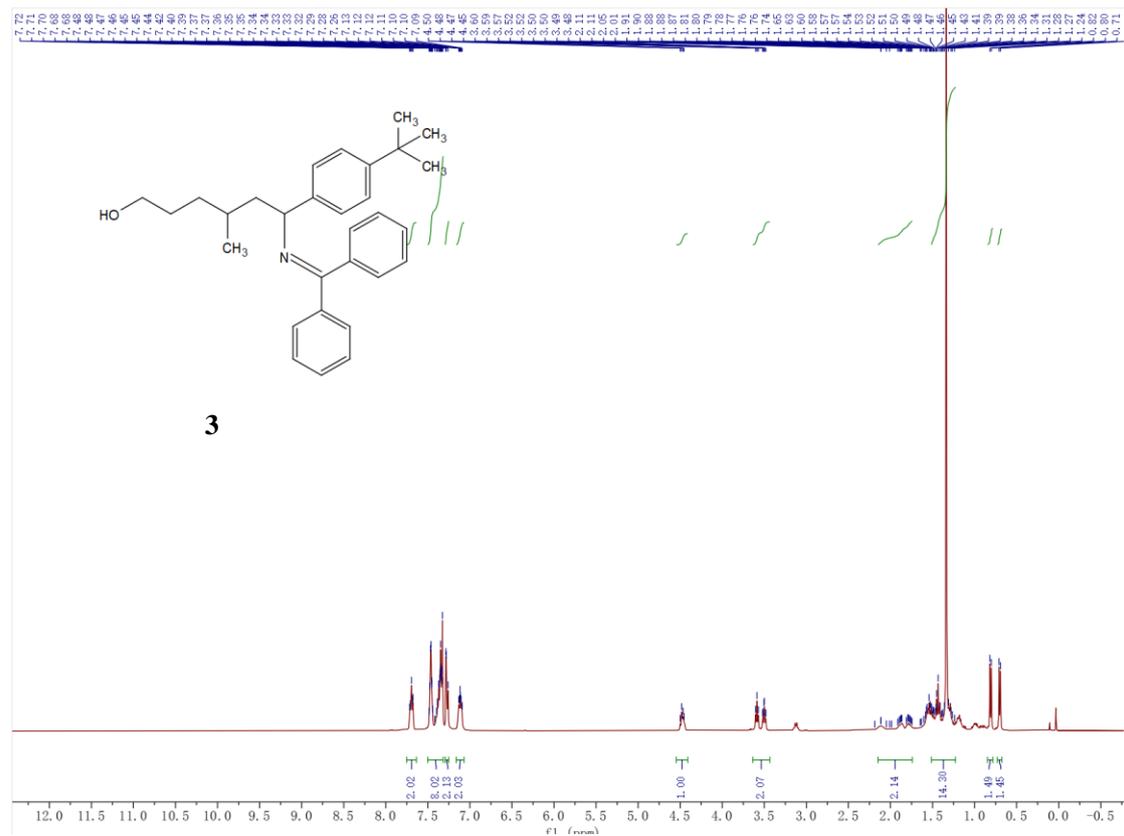
¹H NMR (400 MHz, CDCl₃)



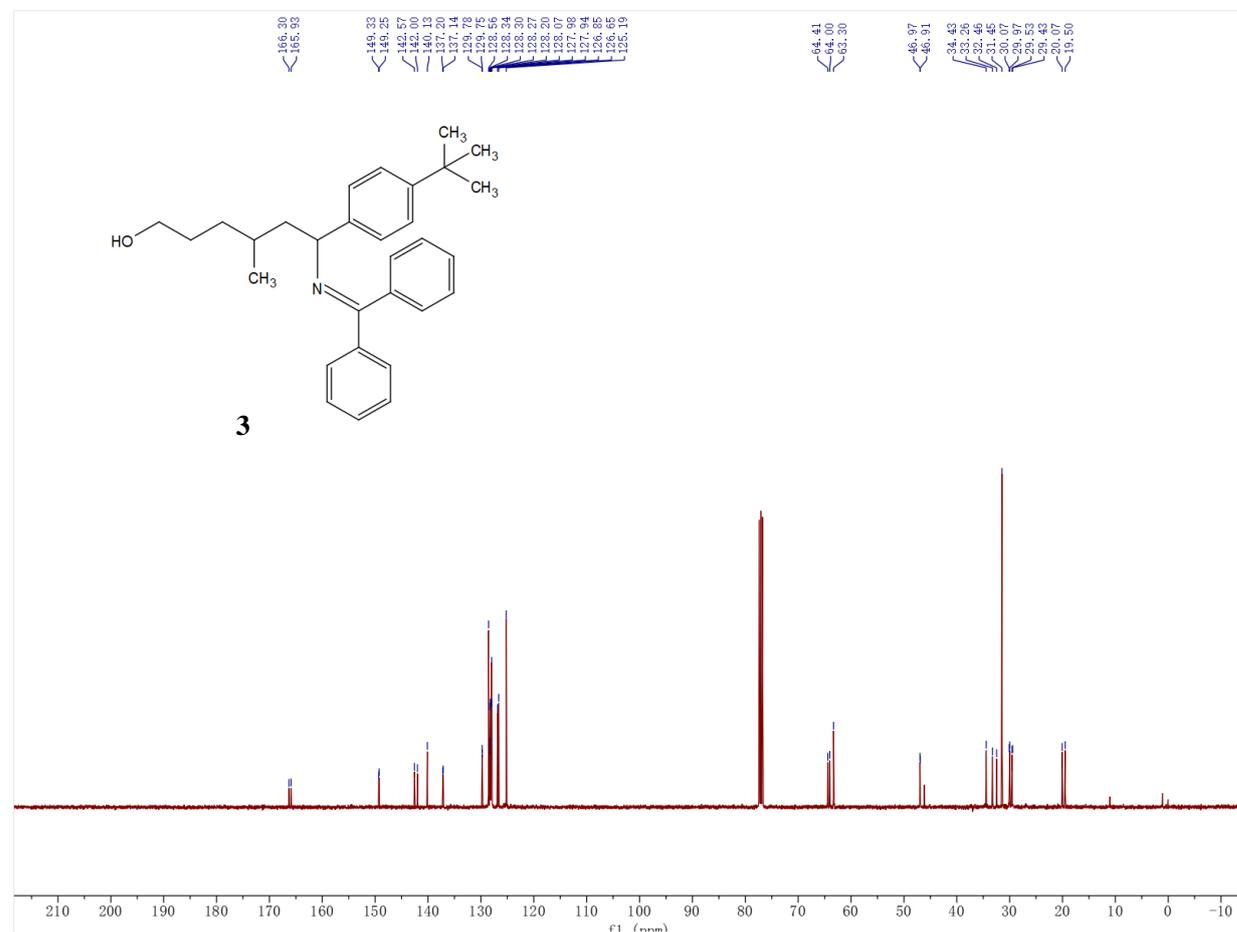
¹³C NMR (101 MHz, CDCl₃)



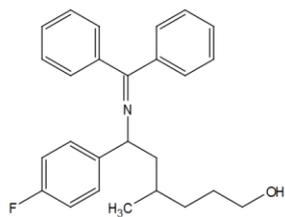
¹H NMR (400 MHz, CDCl₃)



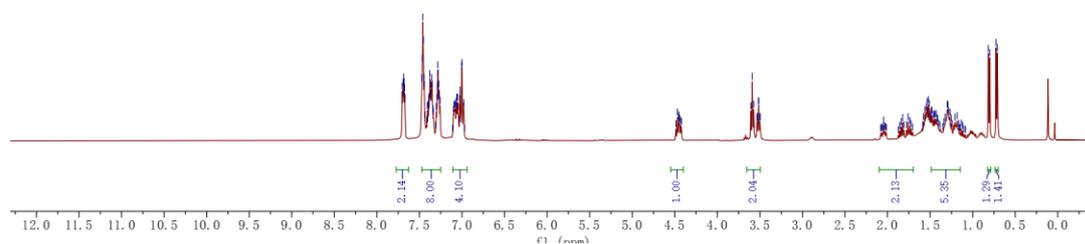
¹³C NMR (101 MHz, CDCl₃)



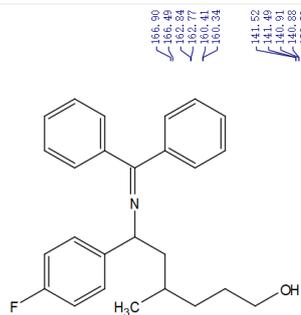
¹H NMR (400 MHz, CDCl₃)



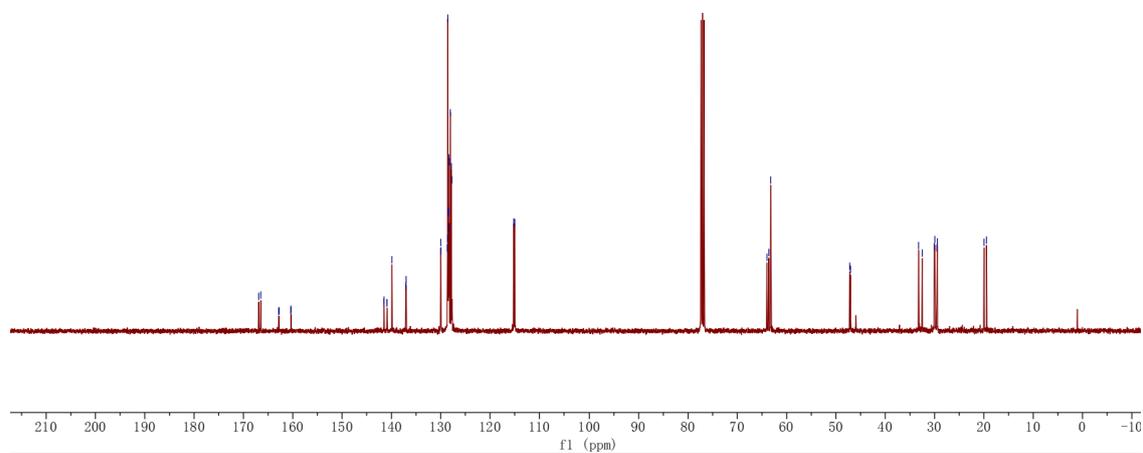
5



¹³C NMR (101 MHz, CDCl₃)

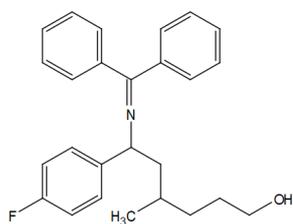


5

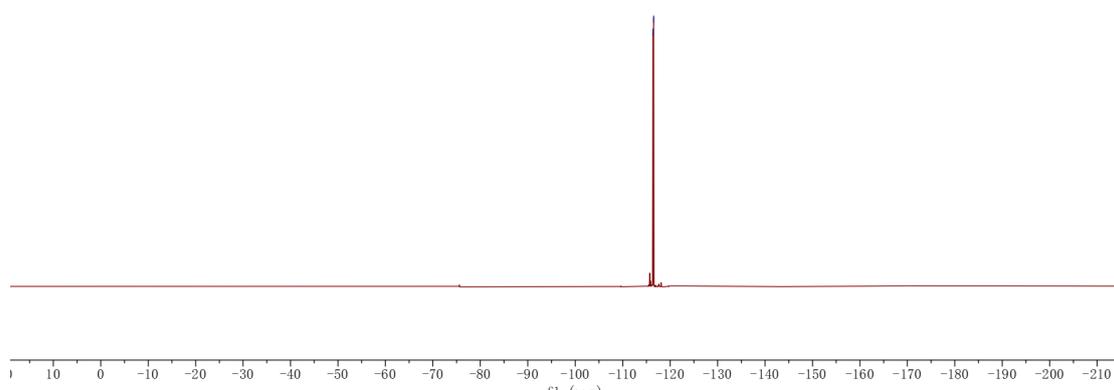


^1F NMR (377 MHz, CDCl_3)

^{19}F NMR (377 MHz, CDCl_3) δ -116.39, -116.53.

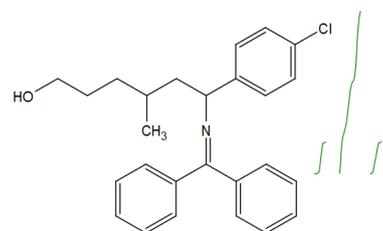


5

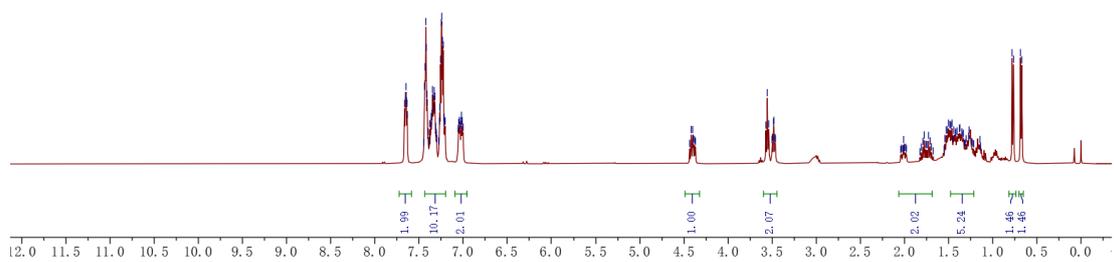


^1H NMR (400 MHz, CDCl_3)

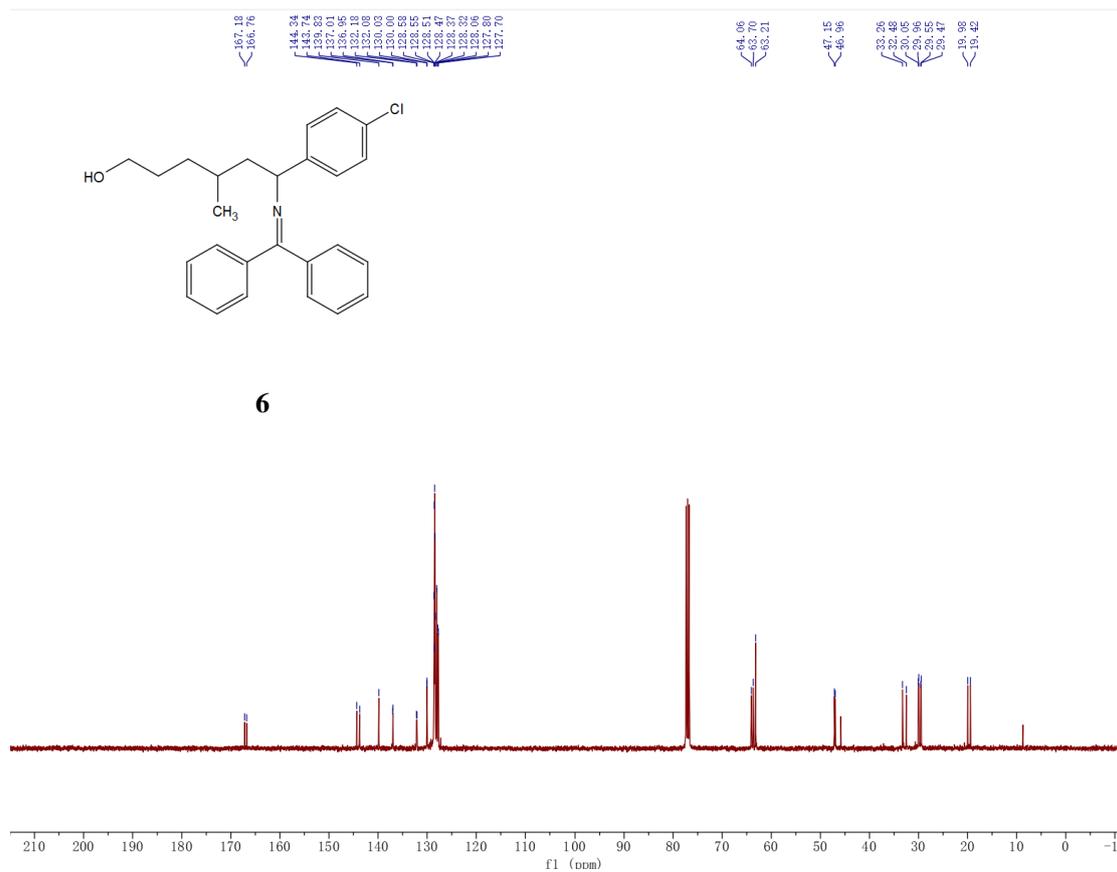
7.66, 7.65, 7.64, 7.63, 7.62, 7.48, 7.44, 7.43, 7.42, 7.41, 7.40, 7.39, 7.38, 7.37, 7.36, 7.35, 7.34, 7.33, 7.32, 7.31, 7.30, 7.29, 7.27, 7.26, 7.25, 7.24, 7.23, 7.22, 7.21, 7.20, 7.19, 7.18, 7.17, 7.16, 7.15, 7.14, 7.13, 7.12, 7.11, 7.10, 7.09, 7.08, 7.07, 7.06, 7.05, 7.04, 7.03, 7.02, 7.01, 7.00, 6.99, 6.98, 6.97, 6.96, 6.95, 6.94, 6.93, 6.92, 6.91, 6.90, 6.89, 6.88, 6.87, 6.86, 6.85, 6.84, 6.83, 6.82, 6.81, 6.80, 6.79, 6.78, 6.77, 6.76, 6.75, 6.74, 6.73, 6.72, 6.71, 6.70, 6.69, 6.68, 6.67, 6.66, 6.65, 6.64, 6.63, 6.62, 6.61, 6.60, 6.59, 6.58, 6.57, 6.56, 6.55, 6.54, 6.53, 6.52, 6.51, 6.50, 6.49, 6.48, 6.47, 6.46, 6.45, 6.44, 6.43, 6.42, 6.41, 6.40, 6.39, 6.38, 6.37, 6.36, 6.35, 6.34, 6.33, 6.32, 6.31, 6.30, 6.29, 6.28, 6.27, 6.26, 6.25, 6.24, 6.23, 6.22, 6.21, 6.20, 6.19, 6.18, 6.17, 6.16, 6.15, 6.14, 6.13, 6.12, 6.11, 6.10, 6.09, 6.08, 6.07, 6.06, 6.05, 6.04, 6.03, 6.02, 6.01, 6.00, 5.99, 5.98, 5.97, 5.96, 5.95, 5.94, 5.93, 5.92, 5.91, 5.90, 5.89, 5.88, 5.87, 5.86, 5.85, 5.84, 5.83, 5.82, 5.81, 5.80, 5.79, 5.78, 5.77, 5.76, 5.75, 5.74, 5.73, 5.72, 5.71, 5.70, 5.69, 5.68, 5.67, 5.66, 5.65, 5.64, 5.63, 5.62, 5.61, 5.60, 5.59, 5.58, 5.57, 5.56, 5.55, 5.54, 5.53, 5.52, 5.51, 5.50, 5.49, 5.48, 5.47, 5.46, 5.45, 5.44, 5.43, 5.42, 5.41, 5.40, 5.39, 5.38, 5.37, 5.36, 5.35, 5.34, 5.33, 5.32, 5.31, 5.30, 5.29, 5.28, 5.27, 5.26, 5.25, 5.24, 5.23, 5.22, 5.21, 5.20, 5.19, 5.18, 5.17, 5.16, 5.15, 5.14, 5.13, 5.12, 5.11, 5.10, 5.09, 5.08, 5.07, 5.06, 5.05, 5.04, 5.03, 5.02, 5.01, 5.00, 4.99, 4.98, 4.97, 4.96, 4.95, 4.94, 4.93, 4.92, 4.91, 4.90, 4.89, 4.88, 4.87, 4.86, 4.85, 4.84, 4.83, 4.82, 4.81, 4.80, 4.79, 4.78, 4.77, 4.76, 4.75, 4.74, 4.73, 4.72, 4.71, 4.70, 4.69, 4.68, 4.67, 4.66, 4.65, 4.64, 4.63, 4.62, 4.61, 4.60, 4.59, 4.58, 4.57, 4.56, 4.55, 4.54, 4.53, 4.52, 4.51, 4.50, 4.49, 4.48, 4.47, 4.46, 4.45, 4.44, 4.43, 4.42, 4.41, 4.40, 4.39, 4.38, 4.37, 4.36, 4.35, 4.34, 4.33, 4.32, 4.31, 4.30, 4.29, 4.28, 4.27, 4.26, 4.25, 4.24, 4.23, 4.22, 4.21, 4.20, 4.19, 4.18, 4.17, 4.16, 4.15, 4.14, 4.13, 4.12, 4.11, 4.10, 4.09, 4.08, 4.07, 4.06, 4.05, 4.04, 4.03, 4.02, 4.01, 4.00, 3.99, 3.98, 3.97, 3.96, 3.95, 3.94, 3.93, 3.92, 3.91, 3.90, 3.89, 3.88, 3.87, 3.86, 3.85, 3.84, 3.83, 3.82, 3.81, 3.80, 3.79, 3.78, 3.77, 3.76, 3.75, 3.74, 3.73, 3.72, 3.71, 3.70, 3.69, 3.68, 3.67, 3.66, 3.65, 3.64, 3.63, 3.62, 3.61, 3.60, 3.59, 3.58, 3.57, 3.56, 3.55, 3.54, 3.53, 3.52, 3.51, 3.50, 3.49, 3.48, 3.47, 3.46, 3.45, 3.44, 3.43, 3.42, 3.41, 3.40, 3.39, 3.38, 3.37, 3.36, 3.35, 3.34, 3.33, 3.32, 3.31, 3.30, 3.29, 3.28, 3.27, 3.26, 3.25, 3.24, 3.23, 3.22, 3.21, 3.20, 3.19, 3.18, 3.17, 3.16, 3.15, 3.14, 3.13, 3.12, 3.11, 3.10, 3.09, 3.08, 3.07, 3.06, 3.05, 3.04, 3.03, 3.02, 3.01, 3.00, 2.99, 2.98, 2.97, 2.96, 2.95, 2.94, 2.93, 2.92, 2.91, 2.90, 2.89, 2.88, 2.87, 2.86, 2.85, 2.84, 2.83, 2.82, 2.81, 2.80, 2.79, 2.78, 2.77, 2.76, 2.75, 2.74, 2.73, 2.72, 2.71, 2.70, 2.69, 2.68, 2.67, 2.66, 2.65, 2.64, 2.63, 2.62, 2.61, 2.60, 2.59, 2.58, 2.57, 2.56, 2.55, 2.54, 2.53, 2.52, 2.51, 2.50, 2.49, 2.48, 2.47, 2.46, 2.45, 2.44, 2.43, 2.42, 2.41, 2.40, 2.39, 2.38, 2.37, 2.36, 2.35, 2.34, 2.33, 2.32, 2.31, 2.30, 2.29, 2.28, 2.27, 2.26, 2.25, 2.24, 2.23, 2.22, 2.21, 2.20, 2.19, 2.18, 2.17, 2.16, 2.15, 2.14, 2.13, 2.12, 2.11, 2.10, 2.09, 2.08, 2.07, 2.06, 2.05, 2.04, 2.03, 2.02, 2.01, 2.00, 1.99, 1.98, 1.97, 1.96, 1.95, 1.94, 1.93, 1.92, 1.91, 1.90, 1.89, 1.88, 1.87, 1.86, 1.85, 1.84, 1.83, 1.82, 1.81, 1.80, 1.79, 1.78, 1.77, 1.76, 1.75, 1.74, 1.73, 1.72, 1.71, 1.70, 1.69, 1.68, 1.67, 1.66, 1.65, 1.64, 1.63, 1.62, 1.61, 1.60, 1.59, 1.58, 1.57, 1.56, 1.55, 1.54, 1.53, 1.52, 1.51, 1.50, 1.49, 1.48, 1.47, 1.46, 1.45, 1.44, 1.43, 1.42, 1.41, 1.40, 1.39, 1.38, 1.37, 1.36, 1.35, 1.34, 1.33, 1.32, 1.31, 1.30, 1.29, 1.28, 1.27, 1.26, 1.25, 1.24, 1.23, 1.22, 1.21, 1.20, 1.19, 1.18, 1.17, 1.16, 1.15, 1.14, 1.13, 1.12, 1.11, 1.10, 1.09, 1.08, 1.07, 1.06, 1.05, 1.04, 1.03, 1.02, 1.01, 1.00, 0.99, 0.98, 0.97, 0.96, 0.95, 0.94, 0.93, 0.92, 0.91, 0.90, 0.89, 0.88, 0.87, 0.86, 0.85, 0.84, 0.83, 0.82, 0.81, 0.80, 0.79, 0.78, 0.77, 0.76, 0.75, 0.74, 0.73, 0.72, 0.71, 0.70, 0.69, 0.68, 0.67, 0.66, 0.65, 0.64, 0.63, 0.62, 0.61, 0.60, 0.59, 0.58, 0.57, 0.56, 0.55, 0.54, 0.53, 0.52, 0.51, 0.50, 0.49, 0.48, 0.47, 0.46, 0.45, 0.44, 0.43, 0.42, 0.41, 0.40, 0.39, 0.38, 0.37, 0.36, 0.35, 0.34, 0.33, 0.32, 0.31, 0.30, 0.29, 0.28, 0.27, 0.26, 0.25, 0.24, 0.23, 0.22, 0.21, 0.20, 0.19, 0.18, 0.17, 0.16, 0.15, 0.14, 0.13, 0.12, 0.11, 0.10, 0.09, 0.08, 0.07, 0.06, 0.05, 0.04, 0.03, 0.02, 0.01, 0.00



6



¹³C NMR (101 MHz, CDCl₃)



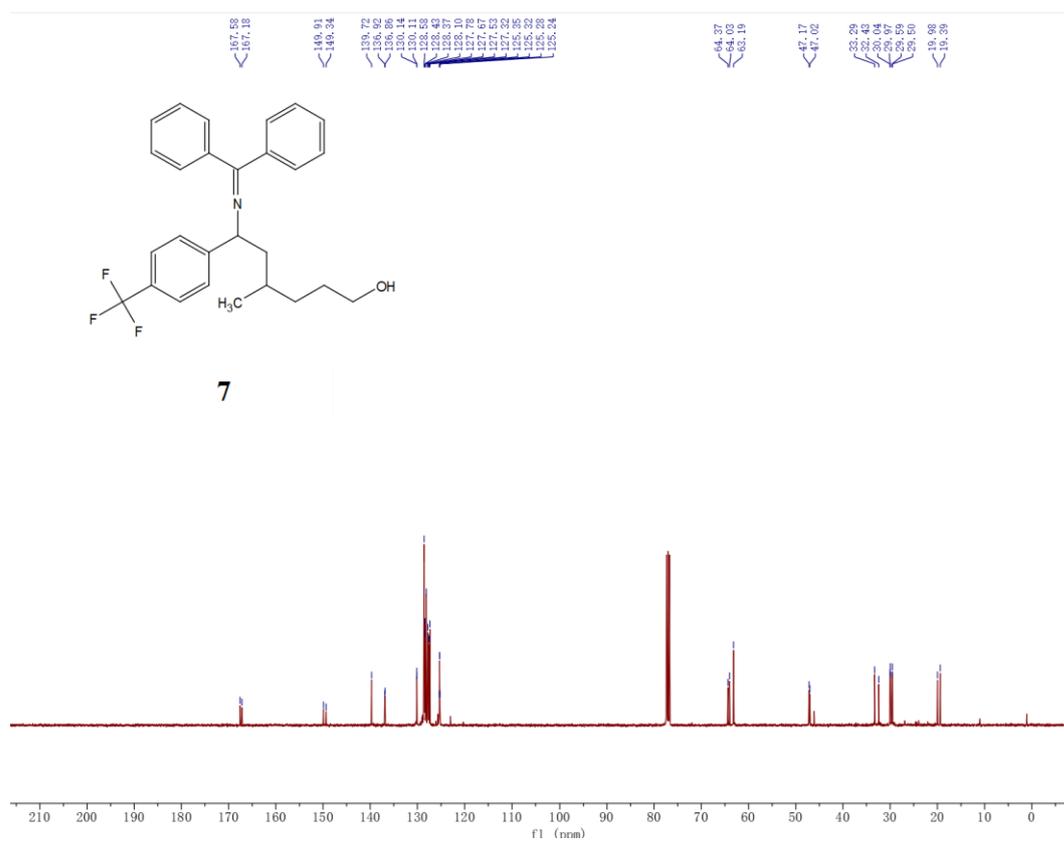
6

¹H NMR (400 MHz, CDCl₃)



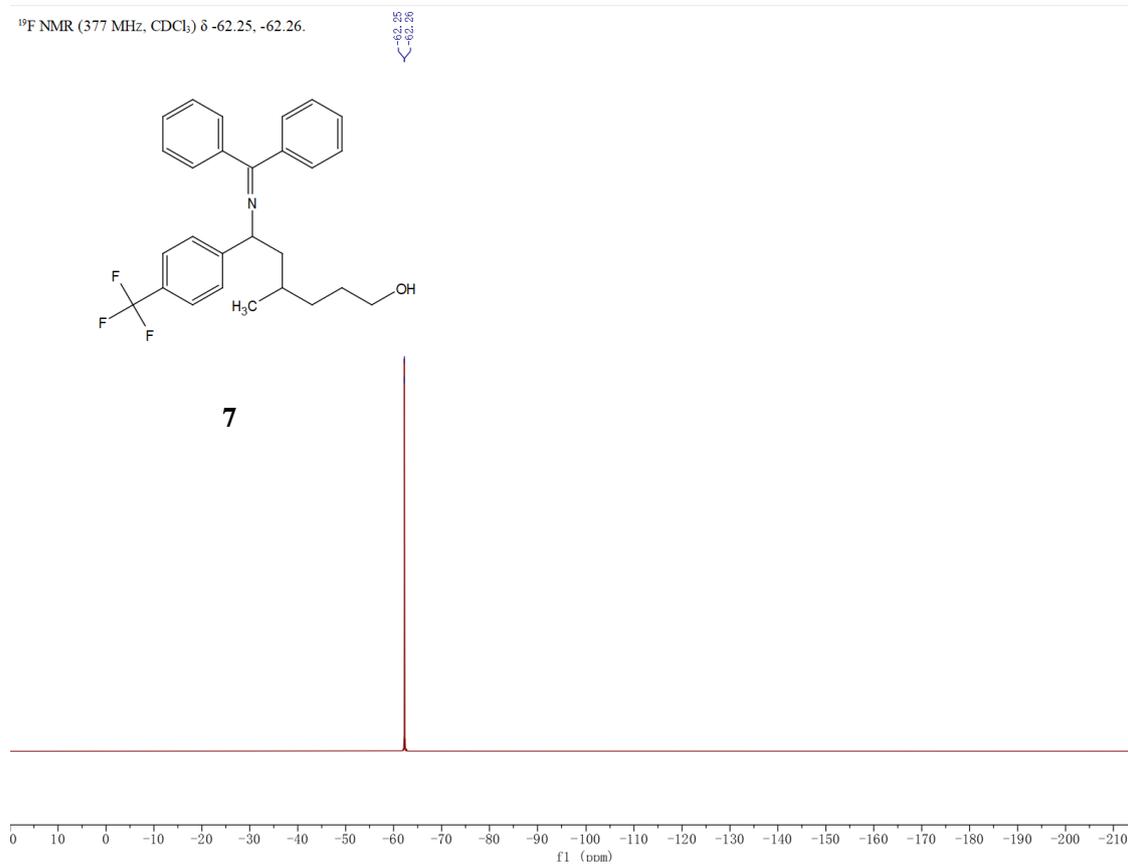
7

¹³C NMR (101 MHz, CDCl₃)

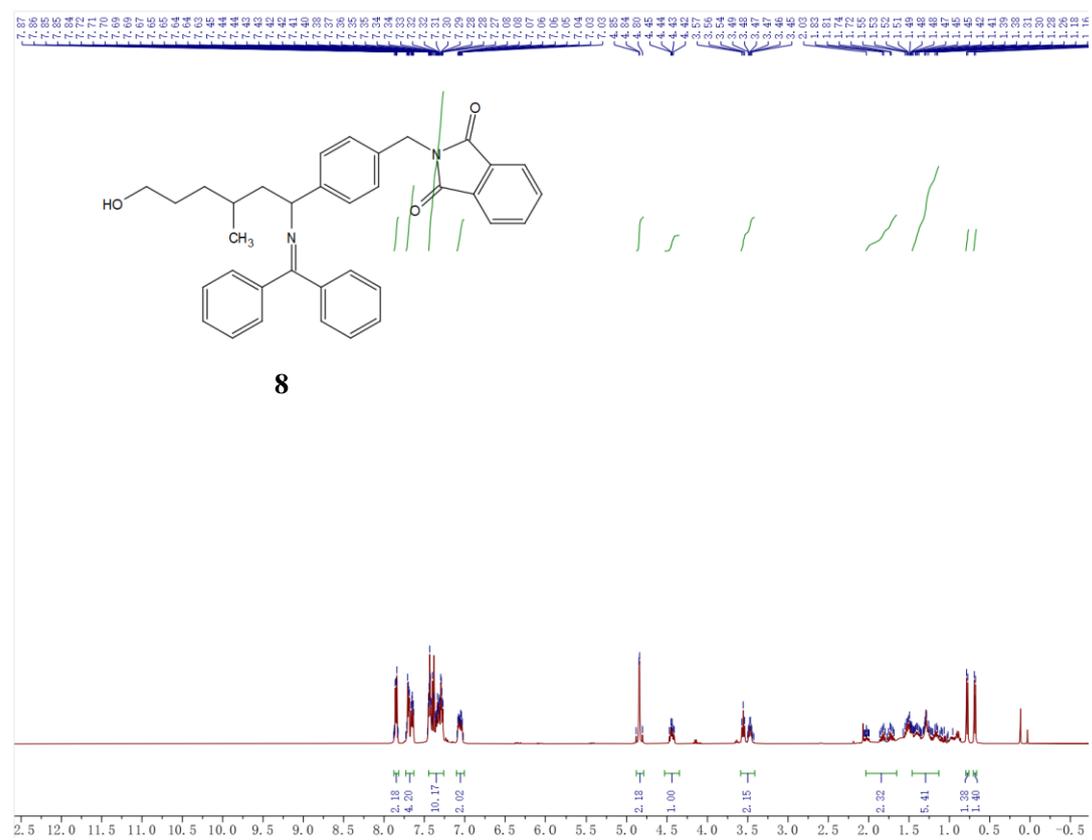


¹⁹F NMR (377 MHz, CDCl₃)

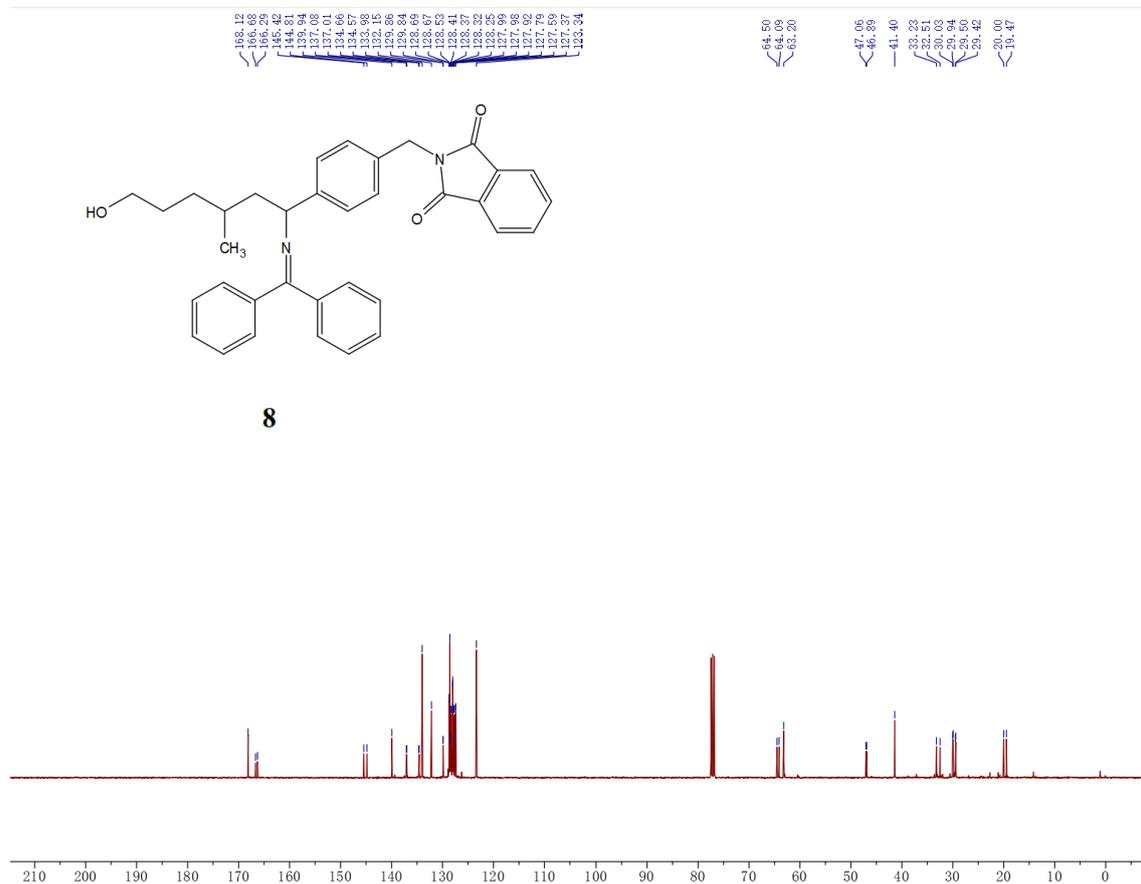
¹⁹F NMR (377 MHz, CDCl₃) δ -62.25, -62.26.



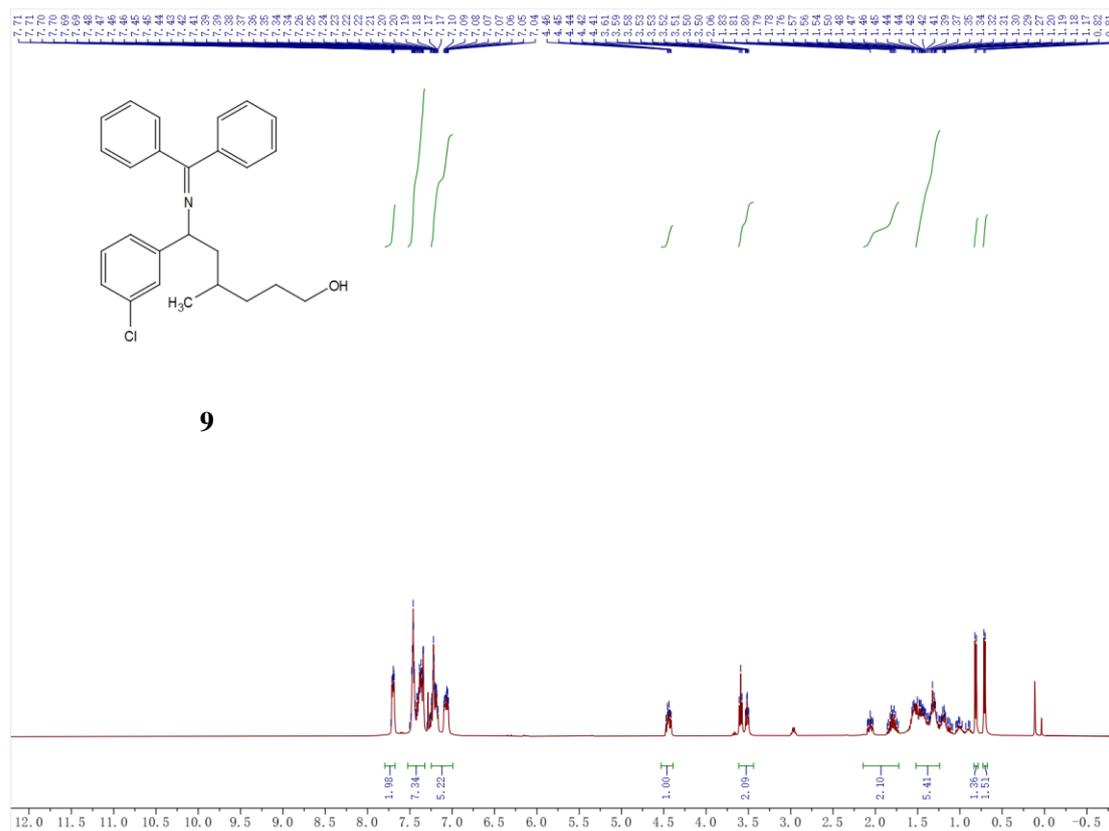
¹H NMR (400 MHz, CDCl₃)



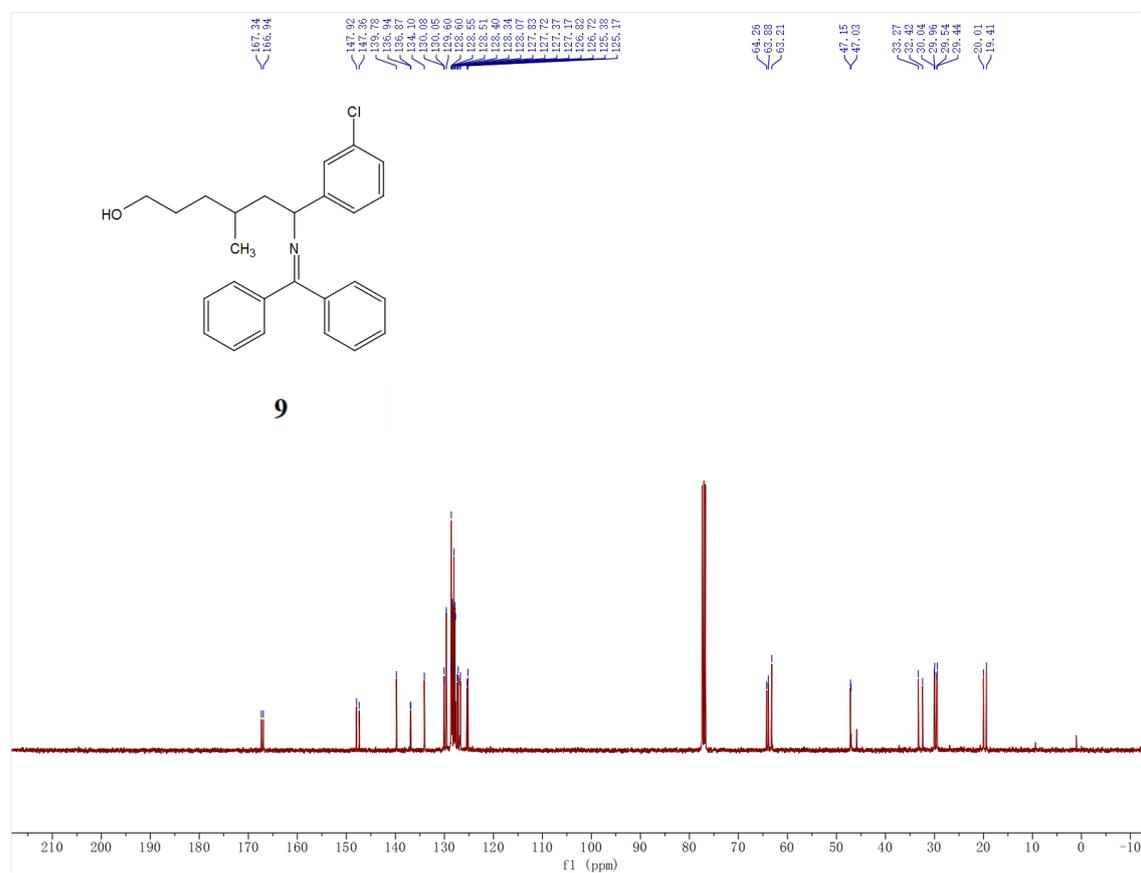
¹³C NMR (101 MHz, CDCl₃)



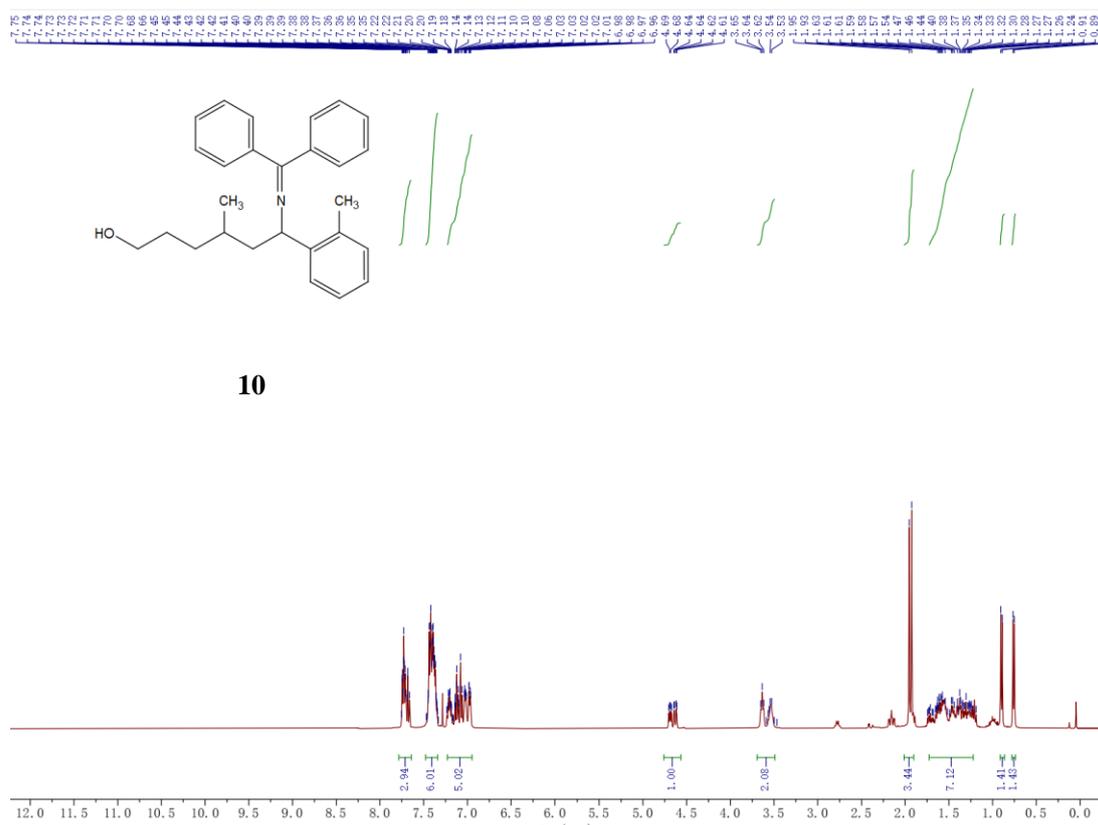
¹H NMR (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)

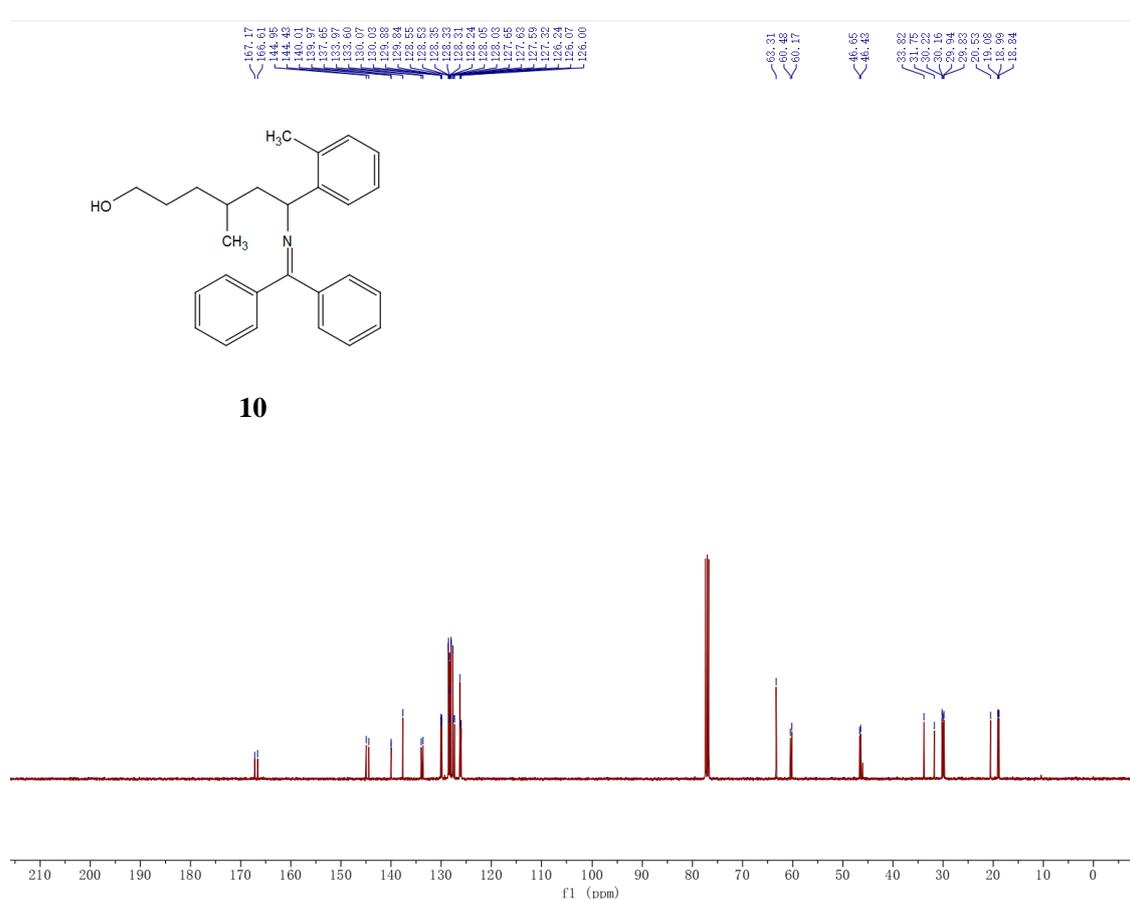


¹H NMR (400 MHz, CDCl₃)



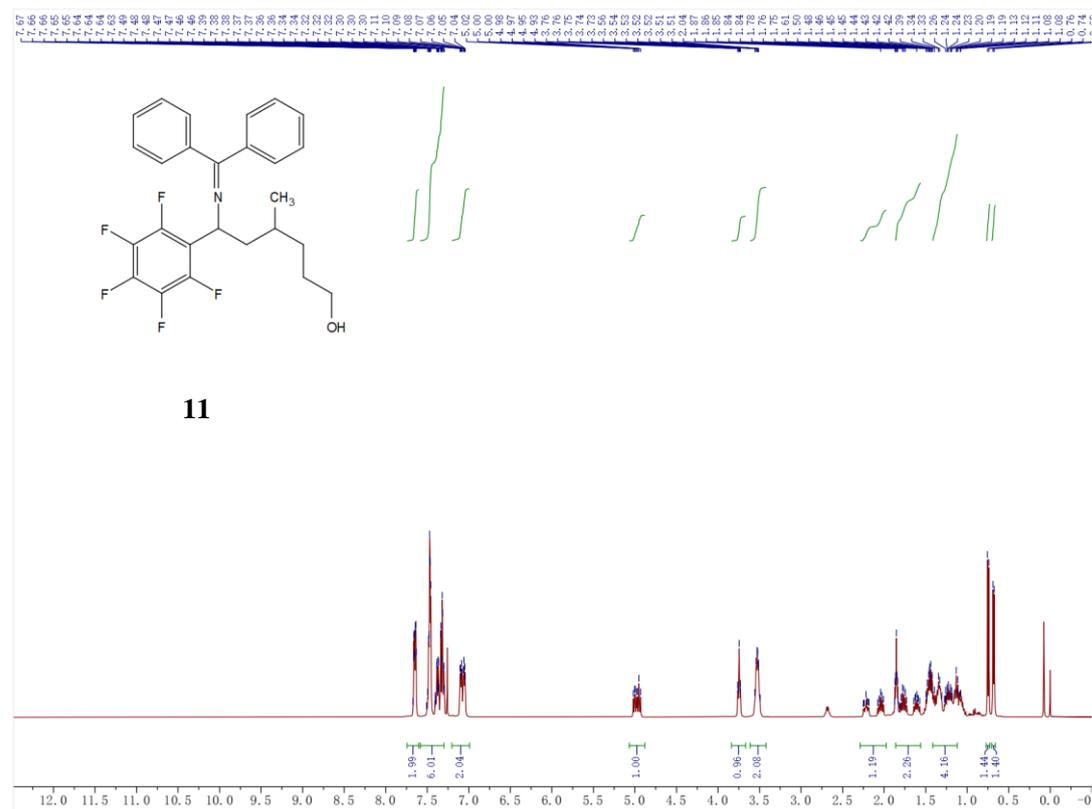
10

¹³C NMR (101 MHz, CDCl₃)

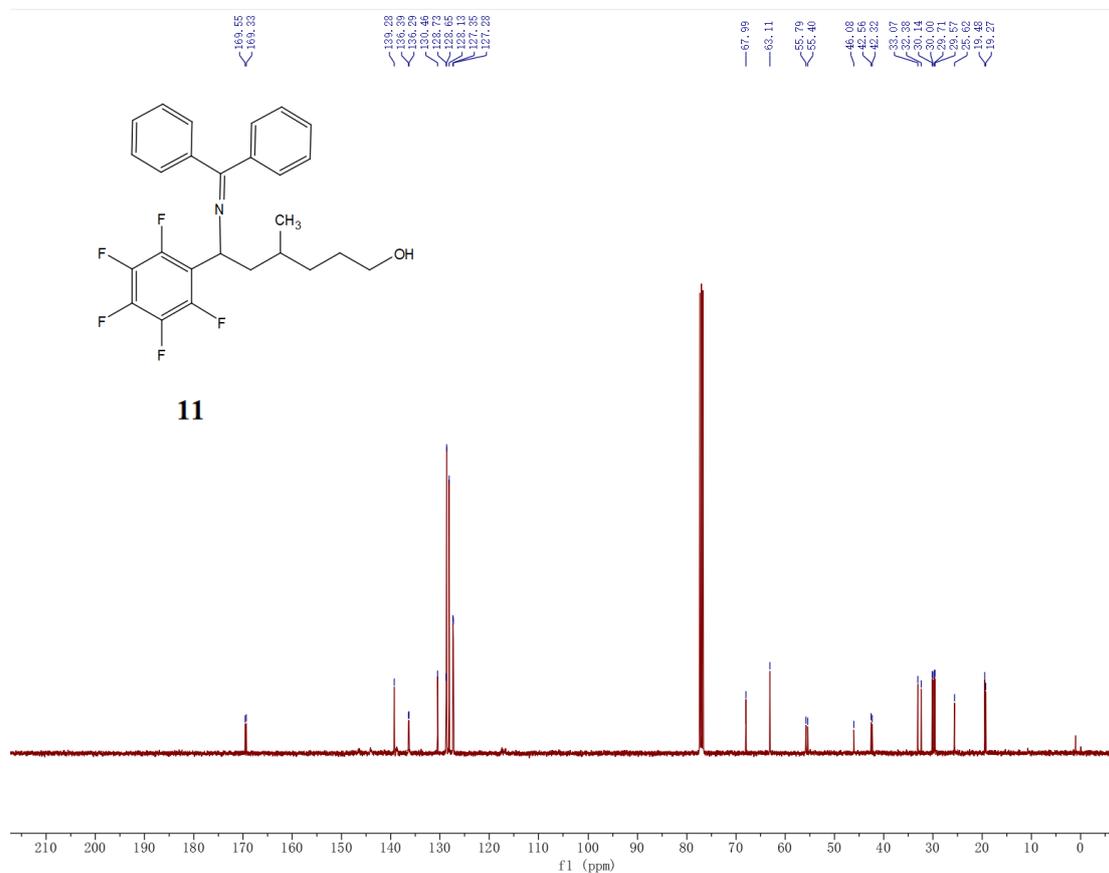


10

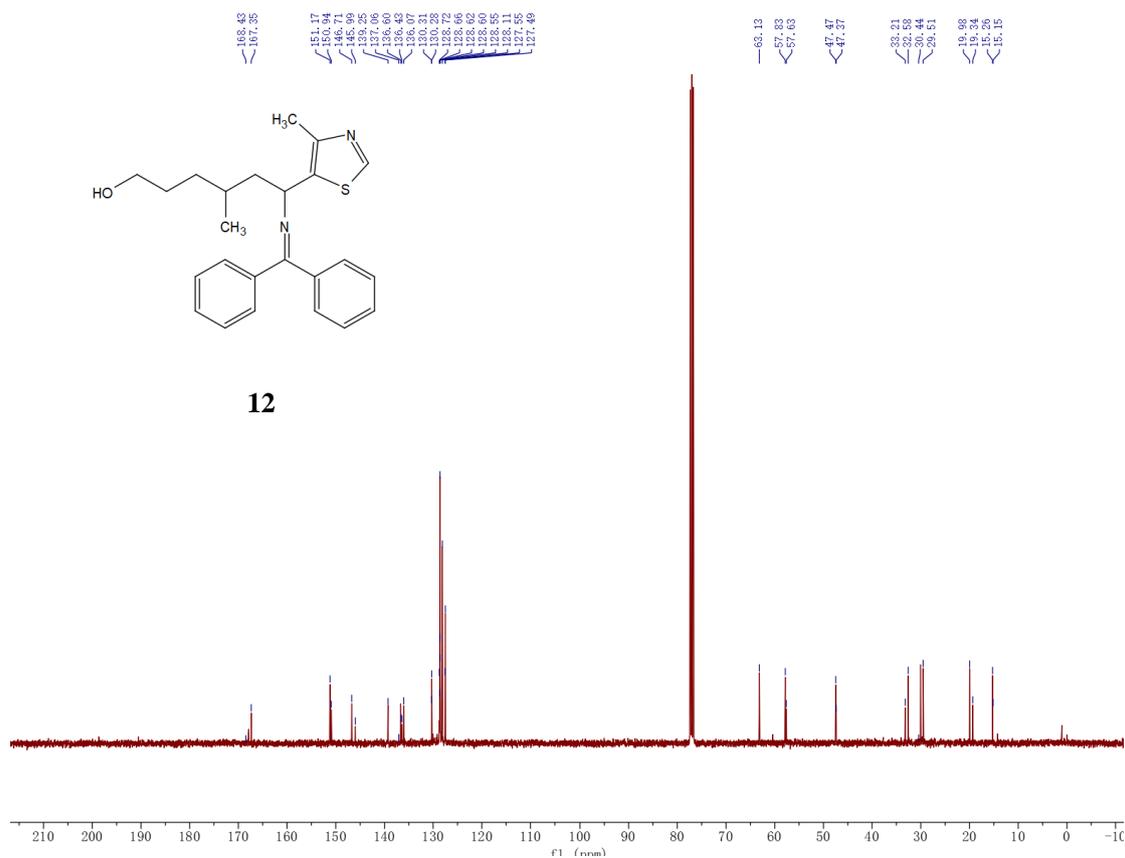
¹H NMR (400 MHz, CDCl₃)



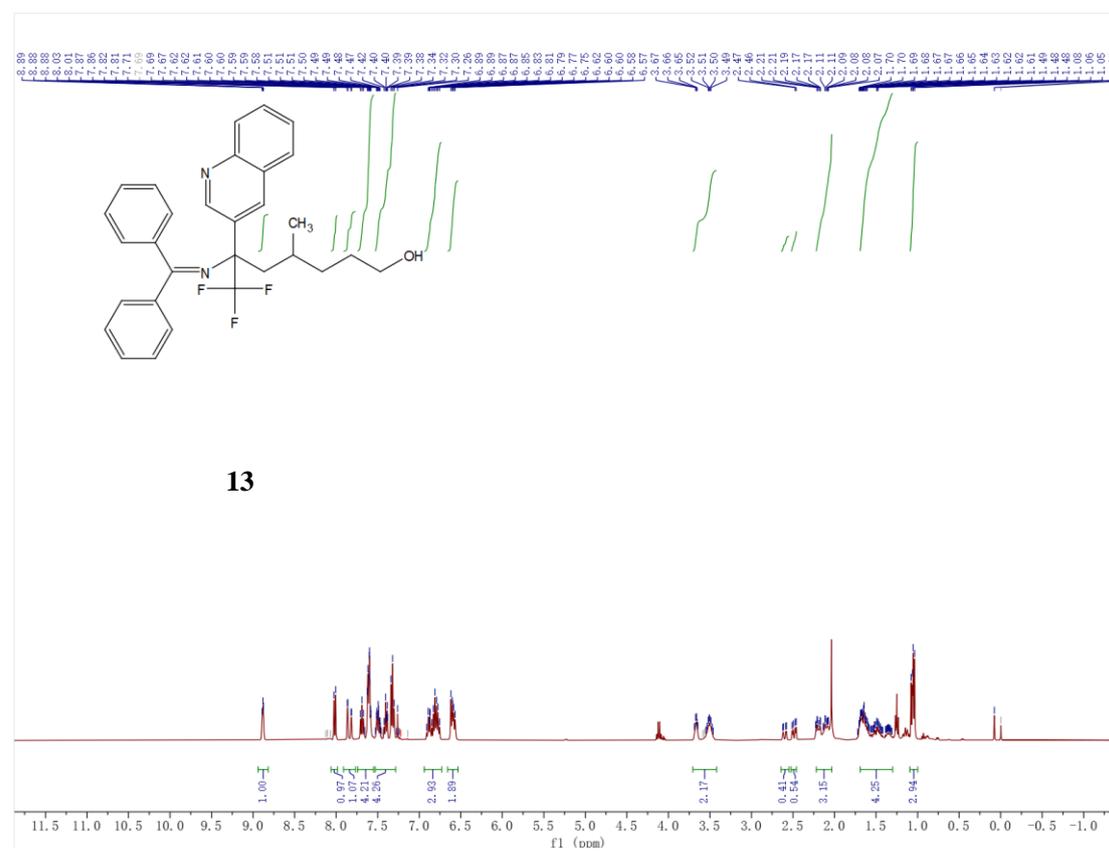
¹³C NMR (101 MHz, CDCl₃)



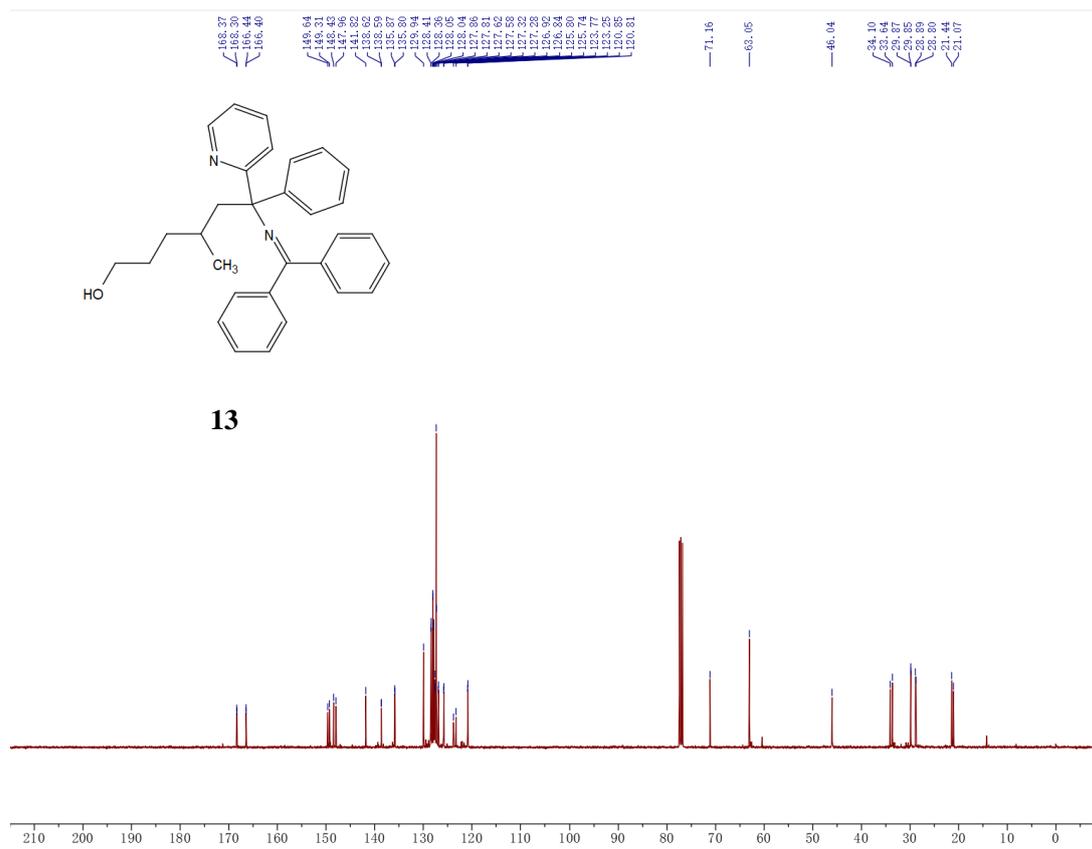
^{13}C NMR (101 MHz, CDCl_3)



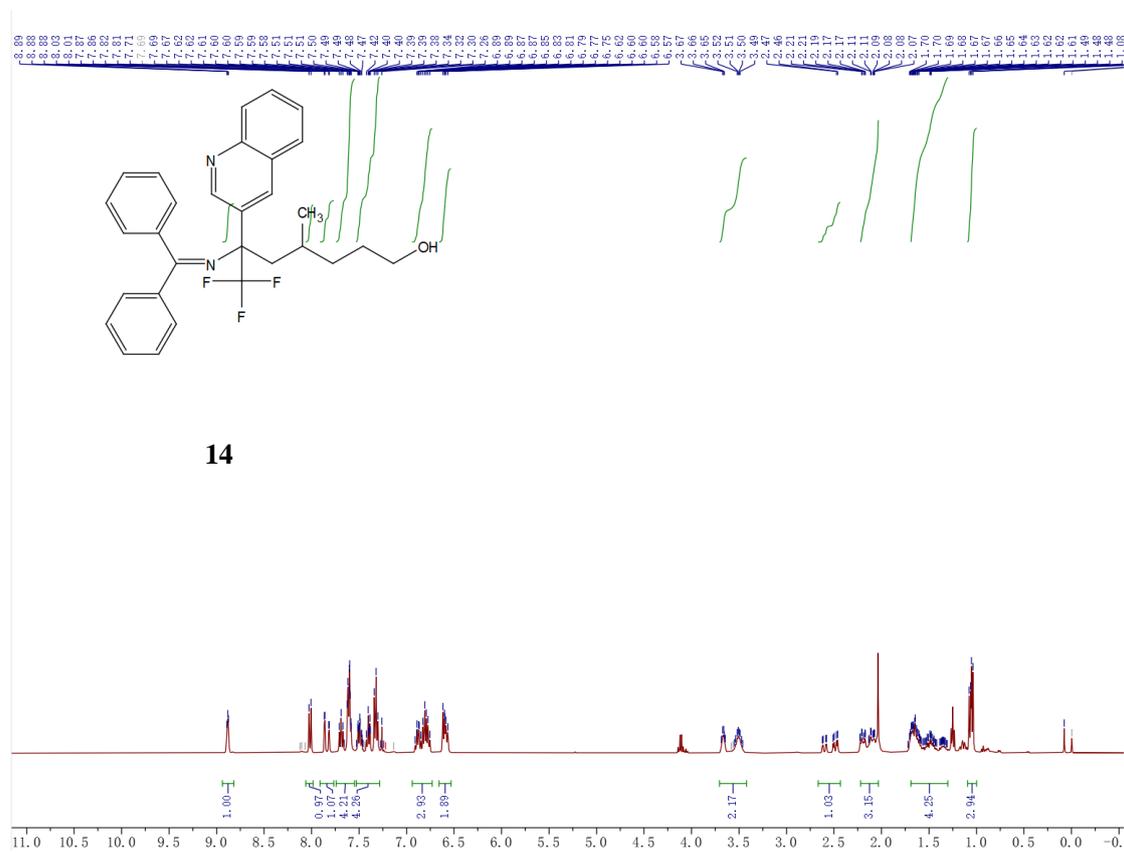
^1H NMR (400 MHz, CDCl_3)



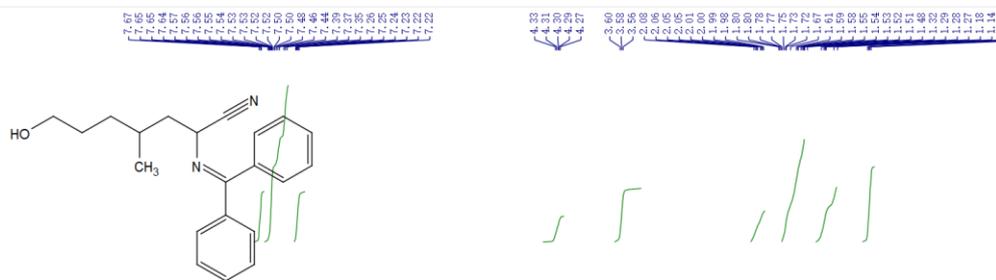
^{13}C NMR (101 MHz, CDCl_3)



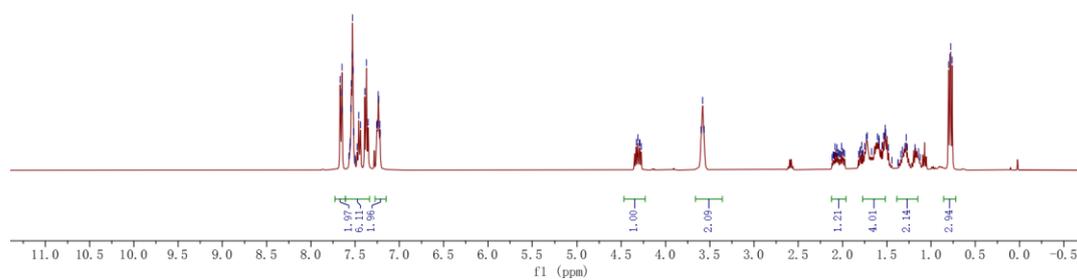
^1H NMR (400 MHz, CDCl_3)



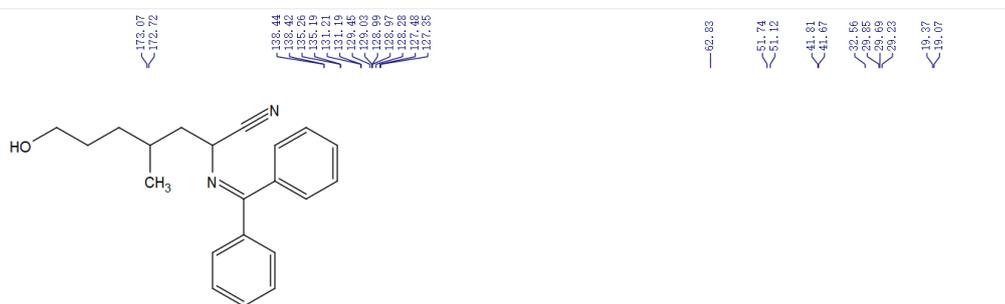
¹H NMR (400 MHz, CDCl₃)



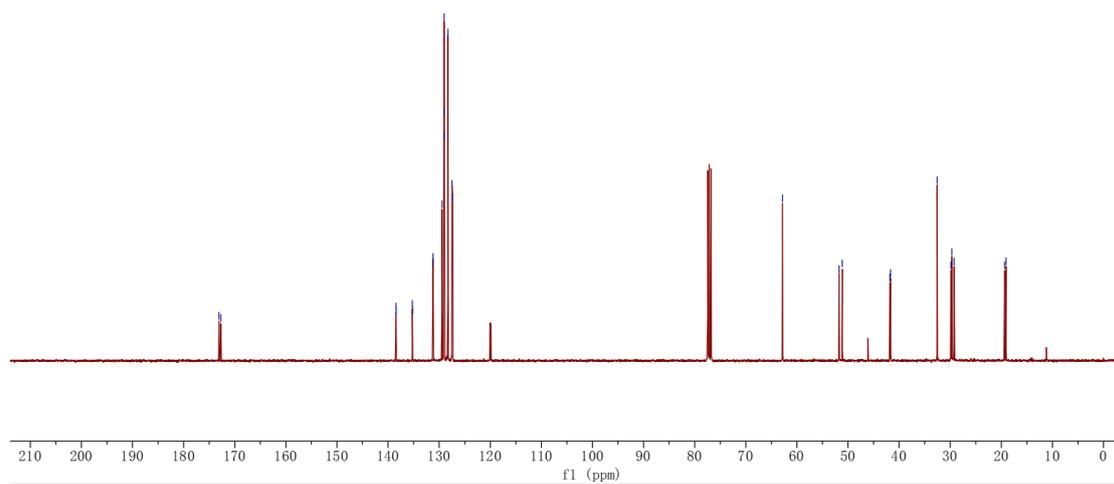
15



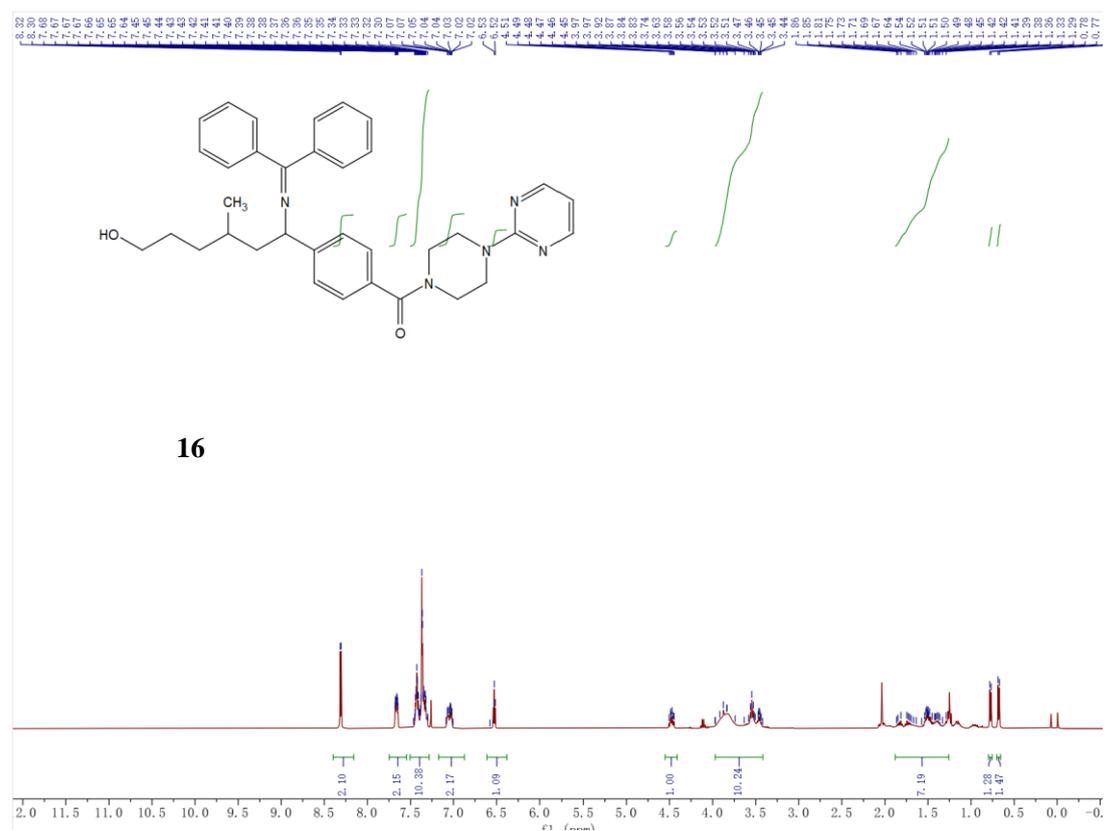
¹³C NMR (101 MHz, CDCl₃)



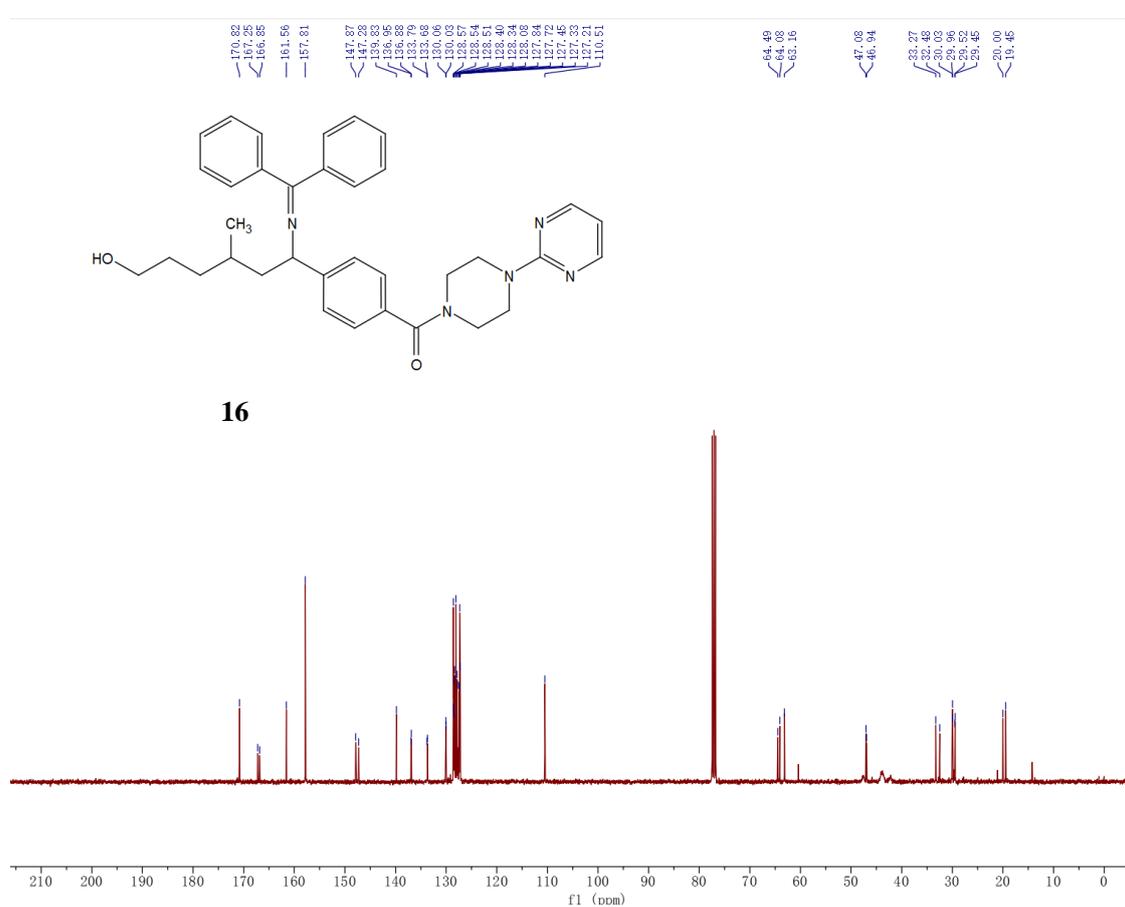
15



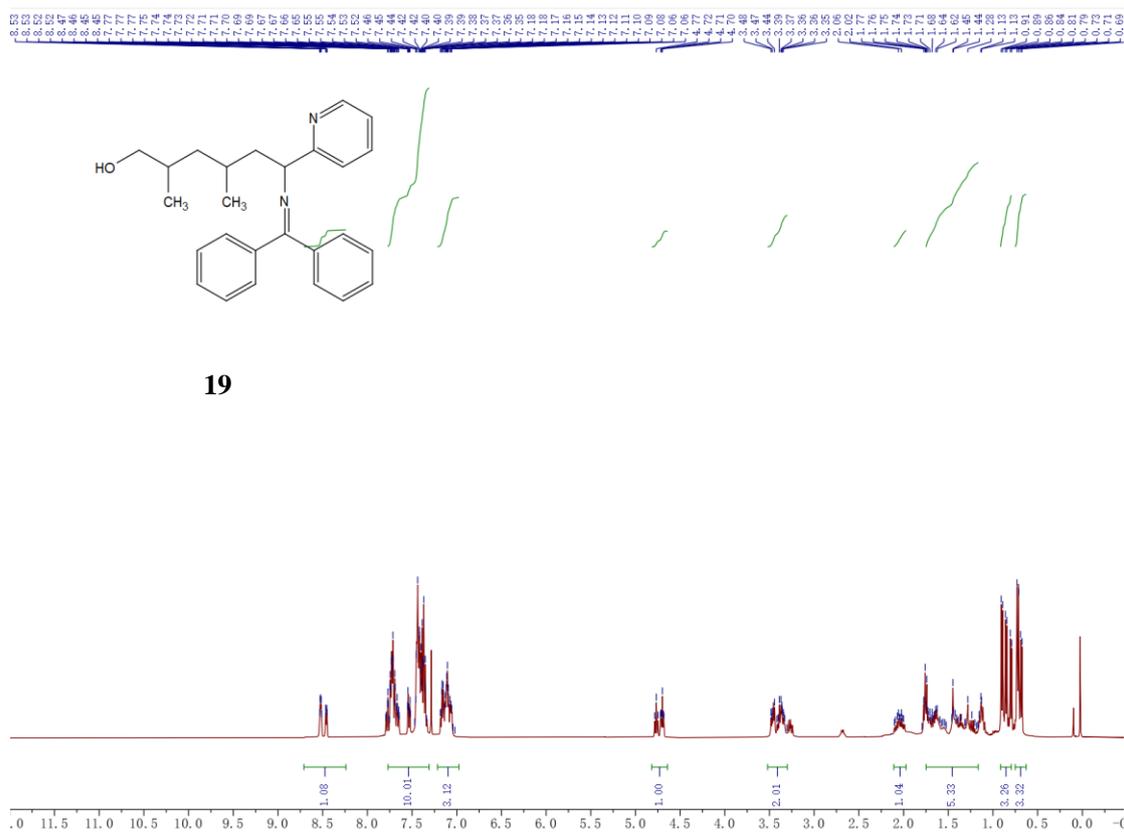
¹H NMR (400 MHz, CDCl₃)



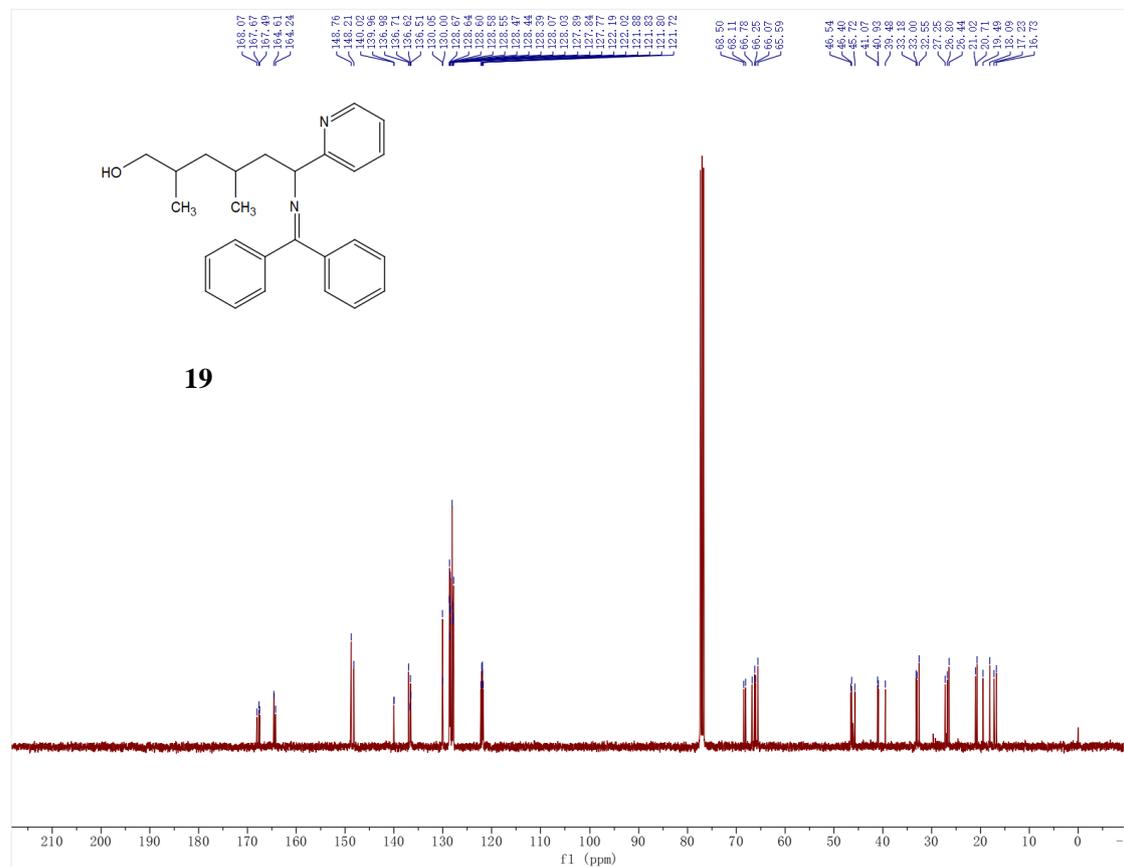
¹³C NMR (101 MHz, CDCl₃)



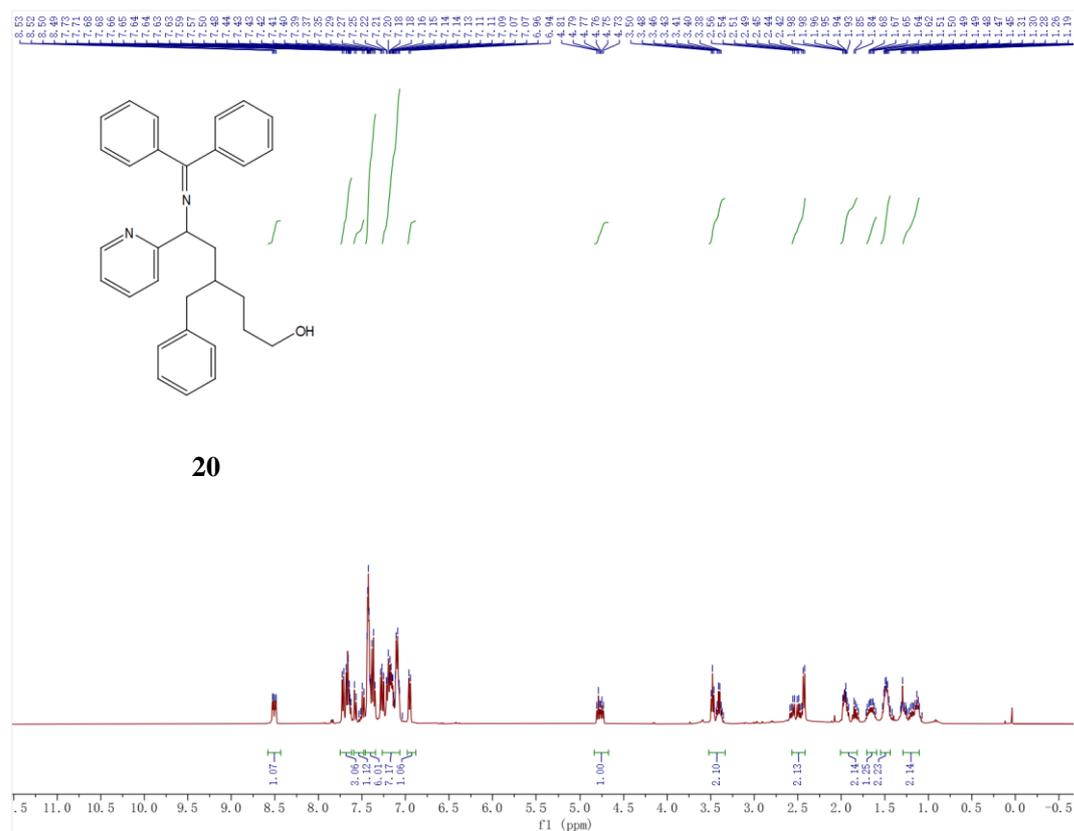
¹H NMR (400 MHz, CDCl₃)



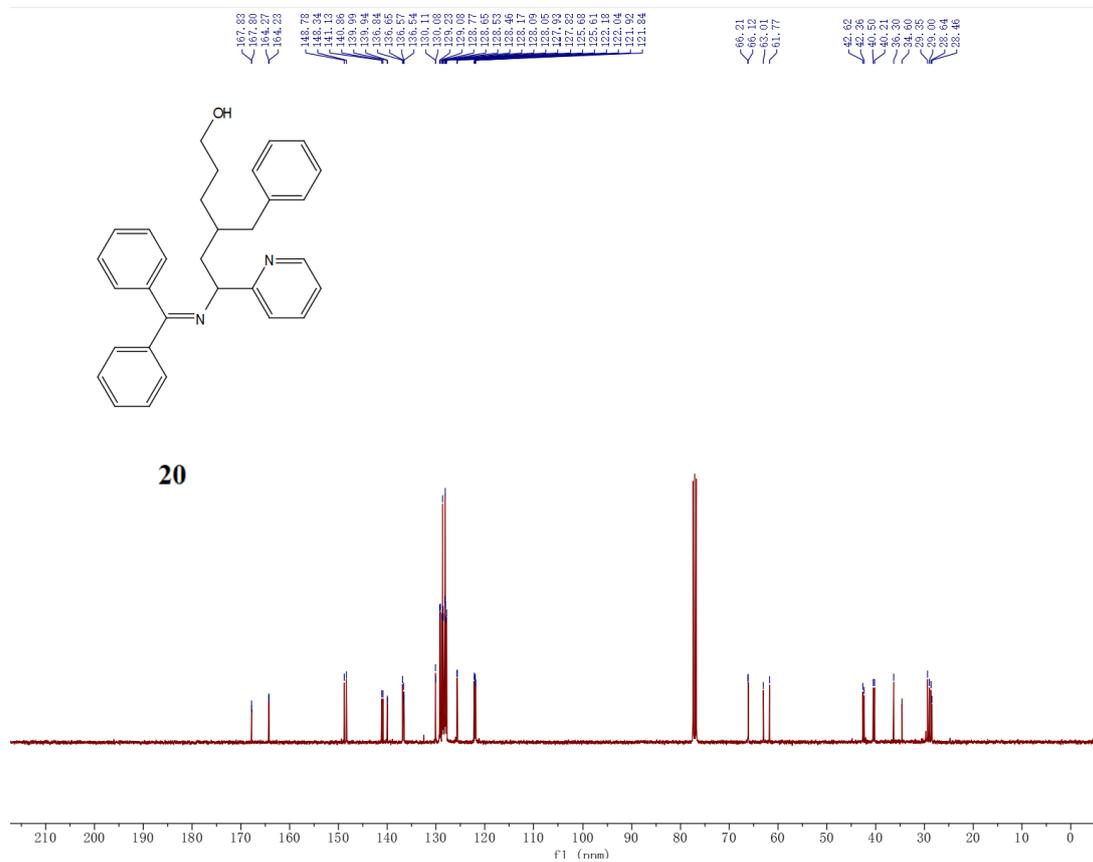
¹³C NMR (101 MHz, CDCl₃)



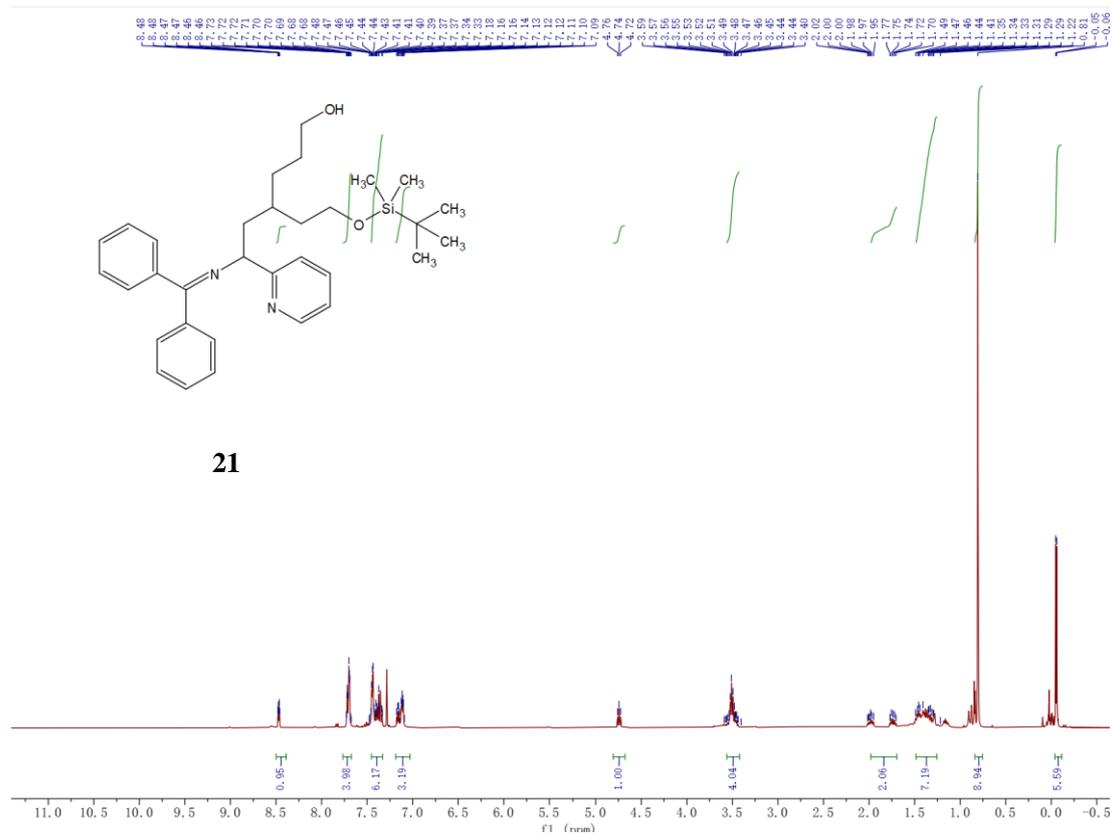
^1H NMR (400 MHz, CDCl_3)



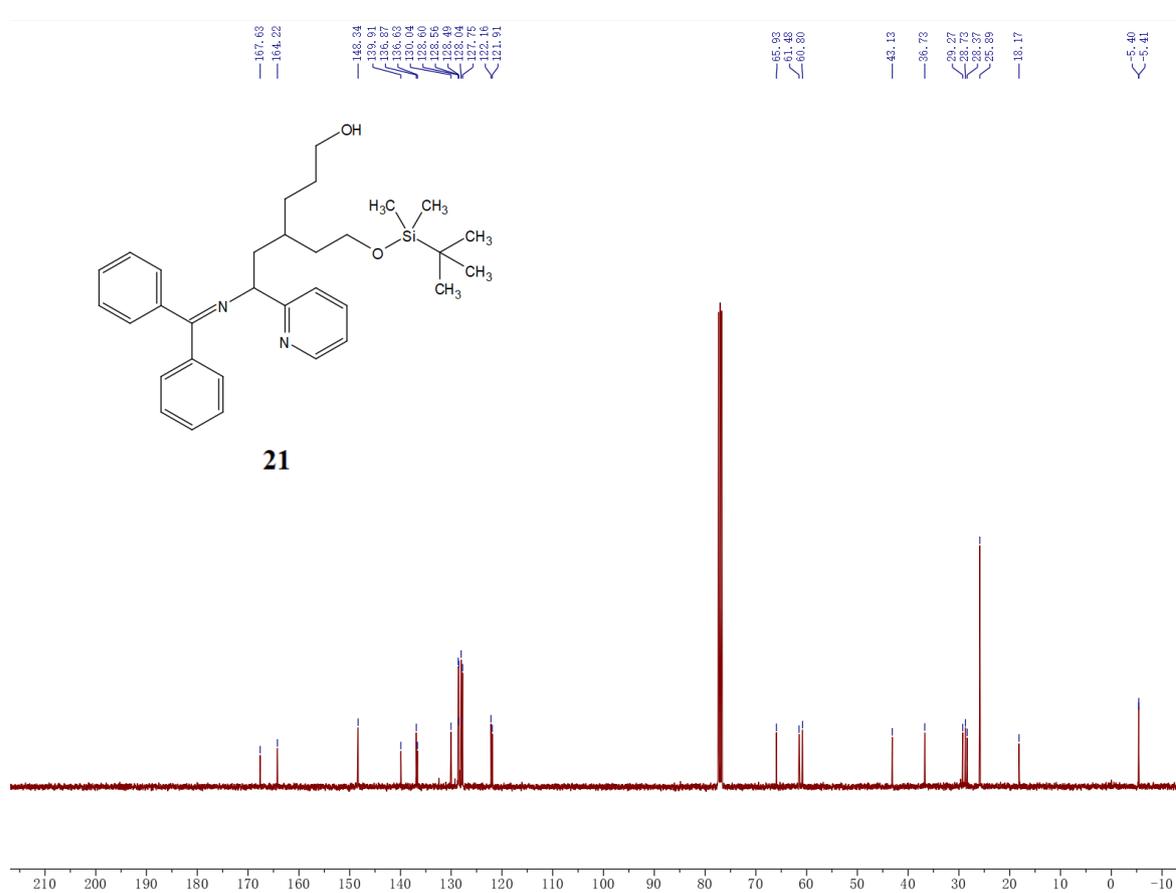
^{13}C NMR (101 MHz, CDCl_3)



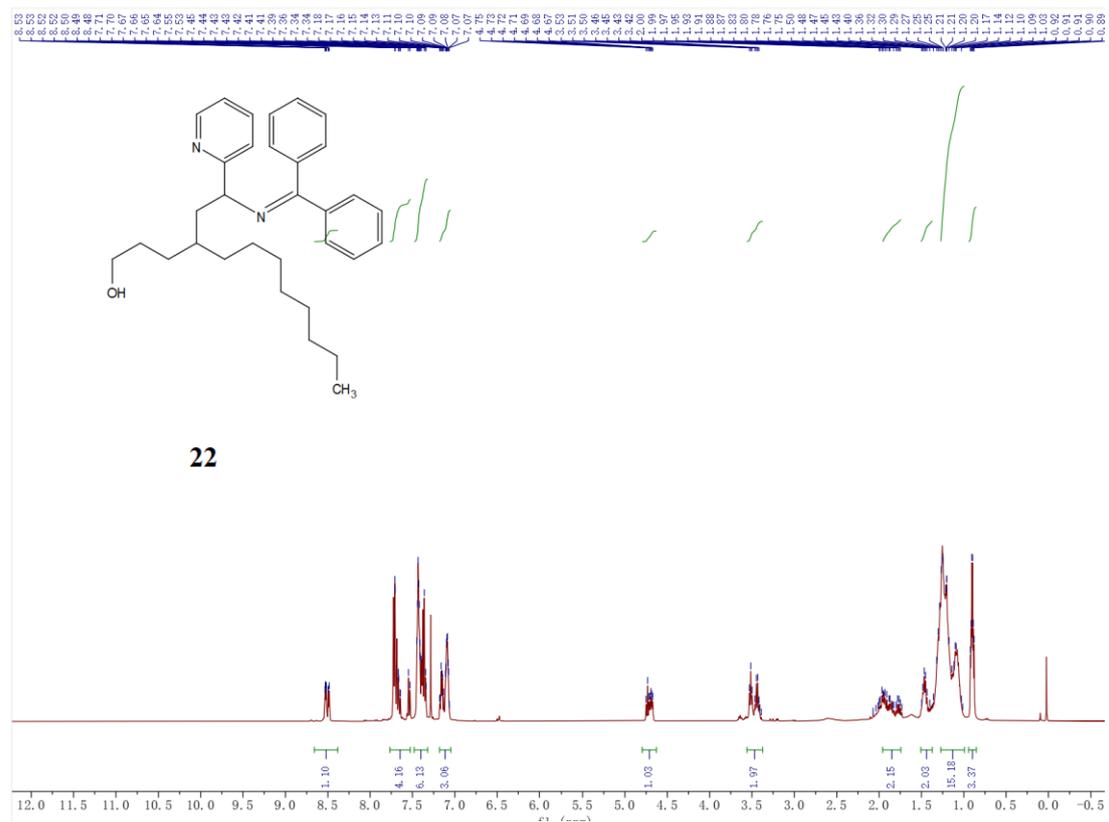
¹H NMR (400 MHz, CDCl₃)



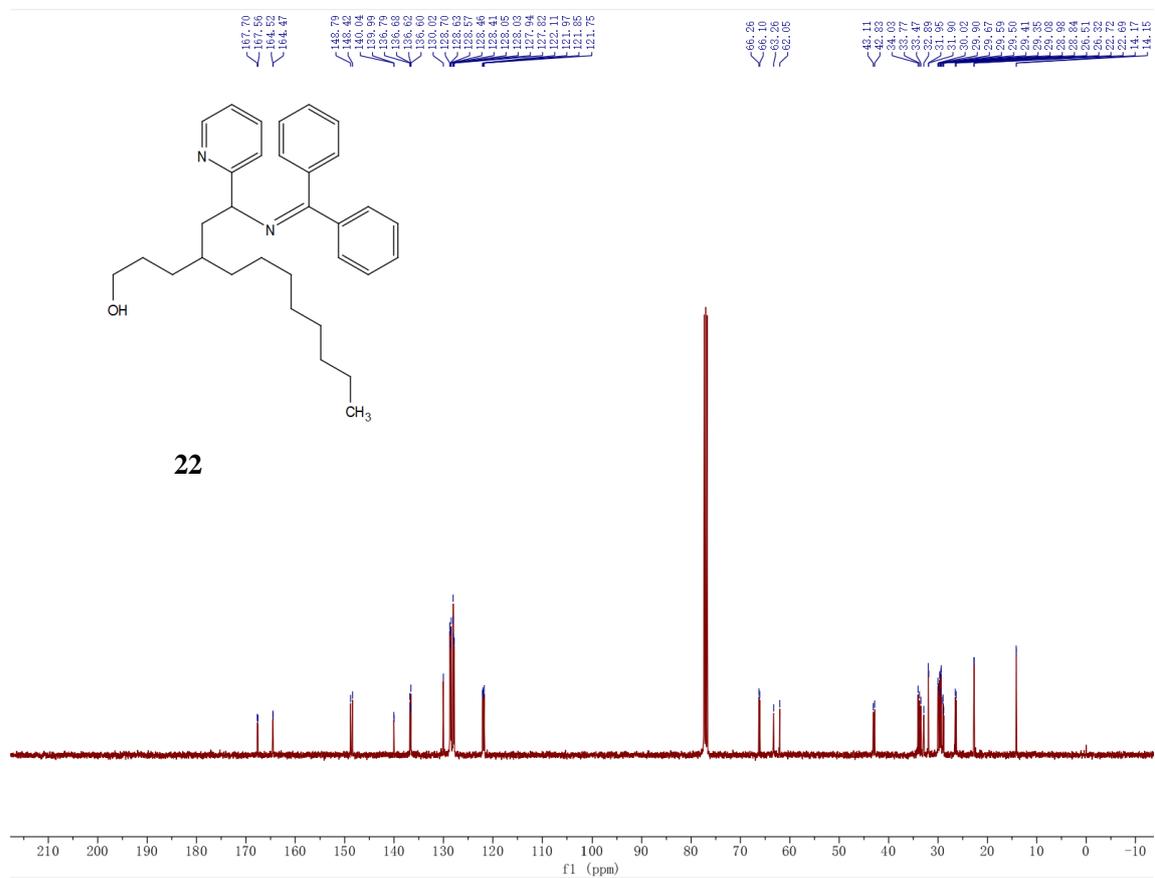
¹³C NMR (101 MHz, CDCl₃)



¹H NMR (400 MHz, CDCl₃)

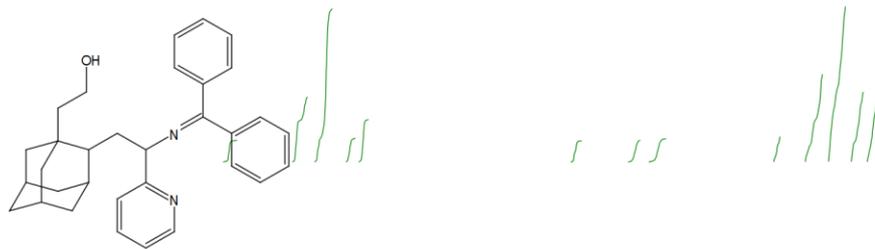


¹³C NMR (101 MHz, CDCl₃)

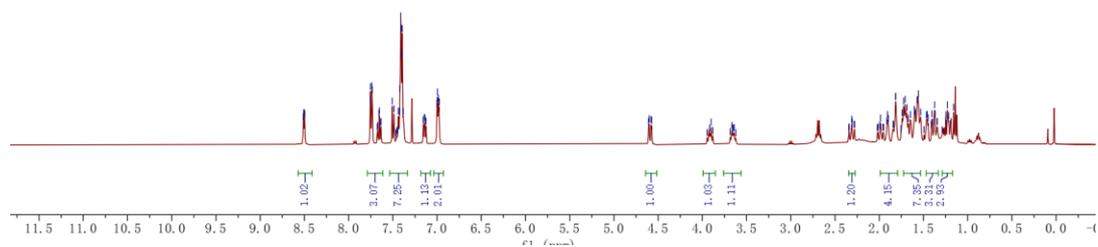


¹H NMR (400 MHz, CDCl₃)

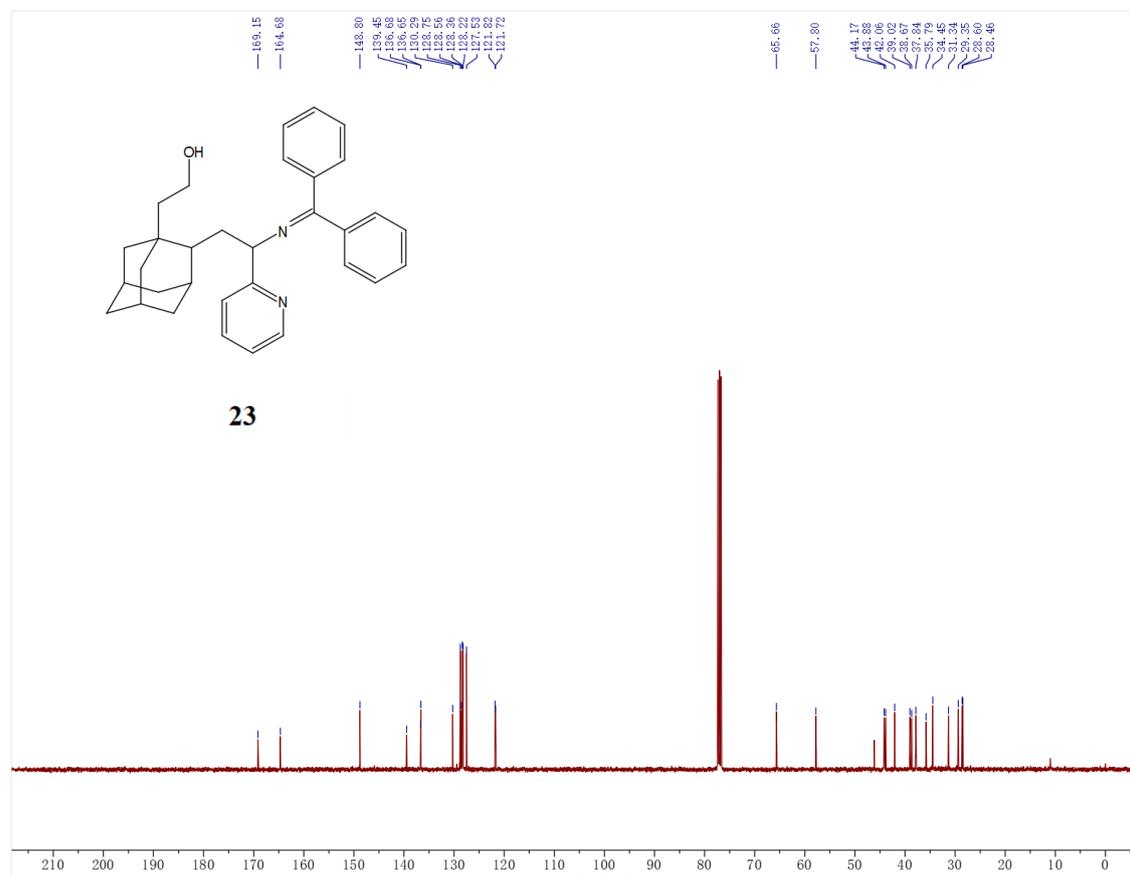
8.51, 8.50, 8.50, 7.76, 7.76, 7.72, 7.72, 7.73, 7.68, 7.68, 7.66, 7.66, 7.64, 7.64, 7.51, 7.49, 7.49, 7.44, 7.44, 7.43, 7.43, 7.41, 7.41, 7.39, 7.39, 7.38, 7.38, 7.16, 7.16, 7.14, 7.14, 7.00, 7.00, 6.99, 6.99, 6.98, 6.98, 6.91, 6.91, 4.60, 4.60, 4.59, 4.59, 3.92, 3.92, 3.87, 3.87, 3.66, 3.66, 3.65, 3.65, 2.95, 2.95, 2.34, 2.34, 2.31, 2.31, 2.28, 2.28, 2.01, 2.01, 1.99, 1.99, 1.99, 1.99, 1.95, 1.95, 1.91, 1.91, 1.90, 1.90, 1.84, 1.84, 1.81, 1.81, 1.75, 1.75, 1.72, 1.72, 1.69, 1.69, 1.65, 1.65, 1.60, 1.60, 1.57, 1.57, 1.56, 1.56, 1.52, 1.52, 1.49, 1.49, 1.46, 1.46, 1.41, 1.41, 1.40, 1.40, 1.37, 1.37, 1.35, 1.35, 1.29, 1.29, 1.25, 1.25, 1.23, 1.23, 1.21, 1.21, 1.19, 1.19



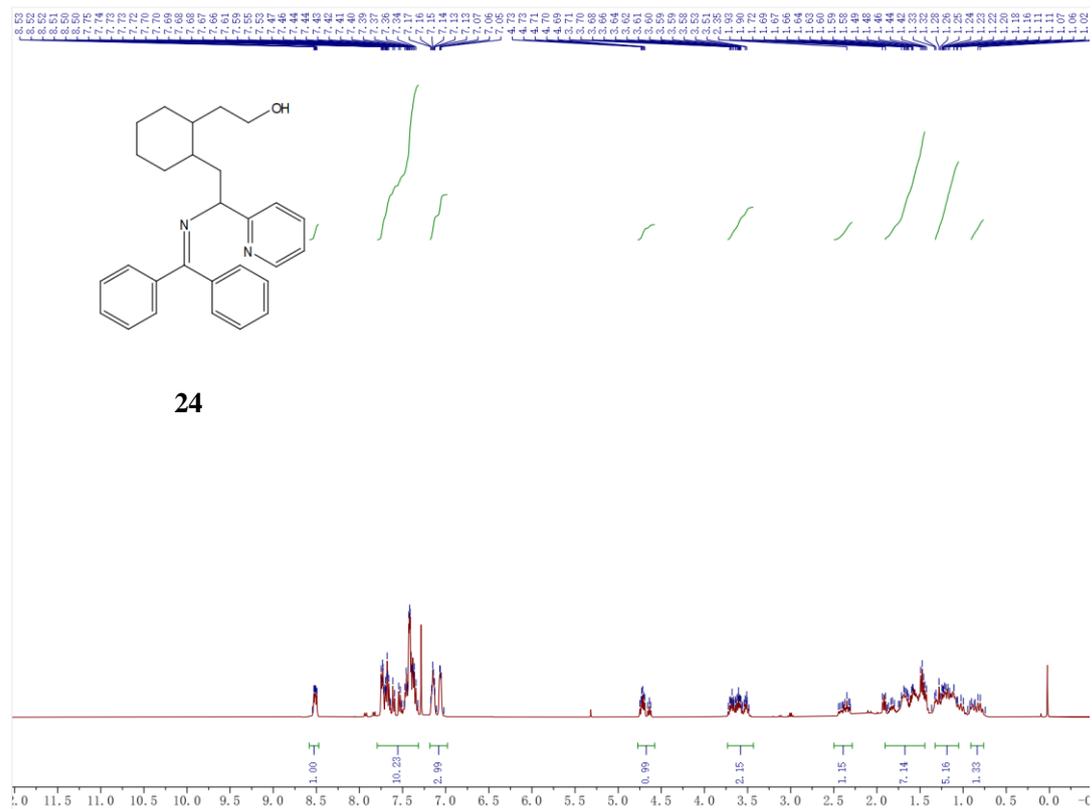
23



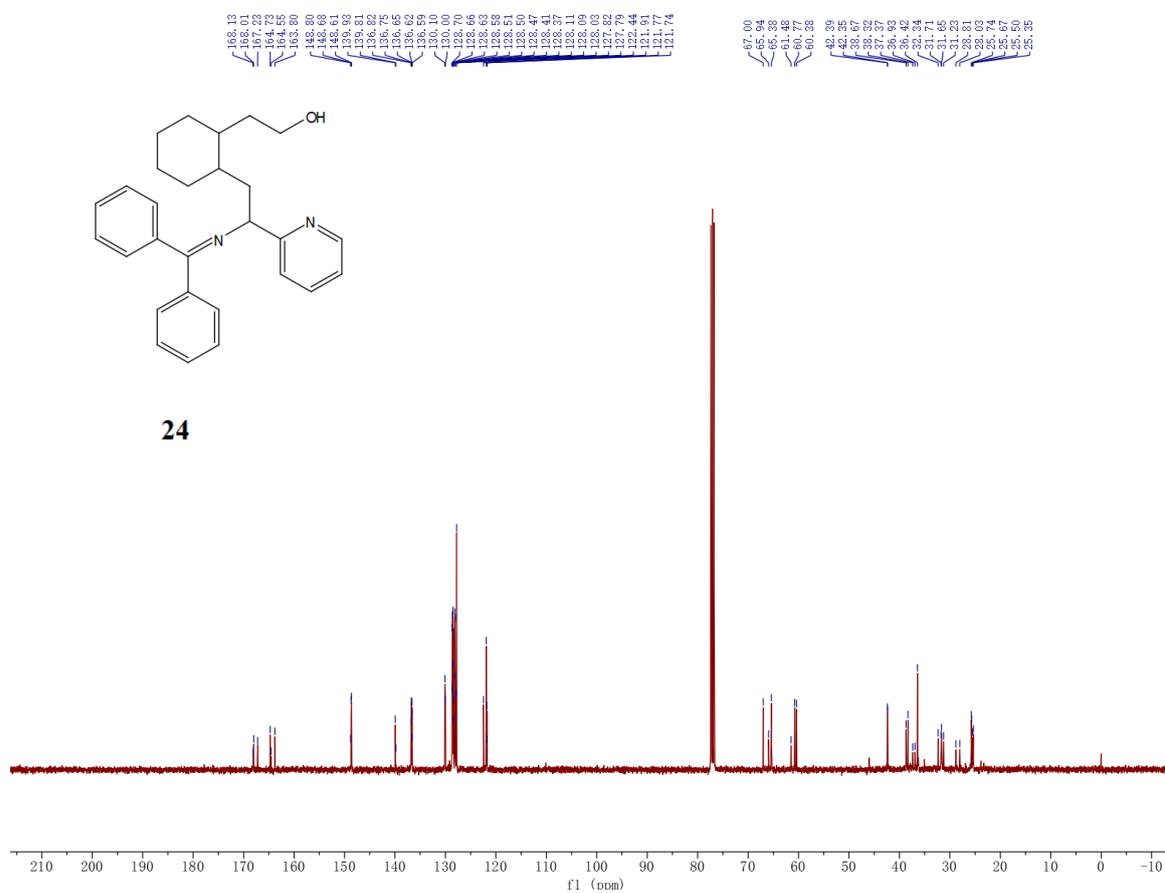
¹³C NMR (101 MHz, CDCl₃)



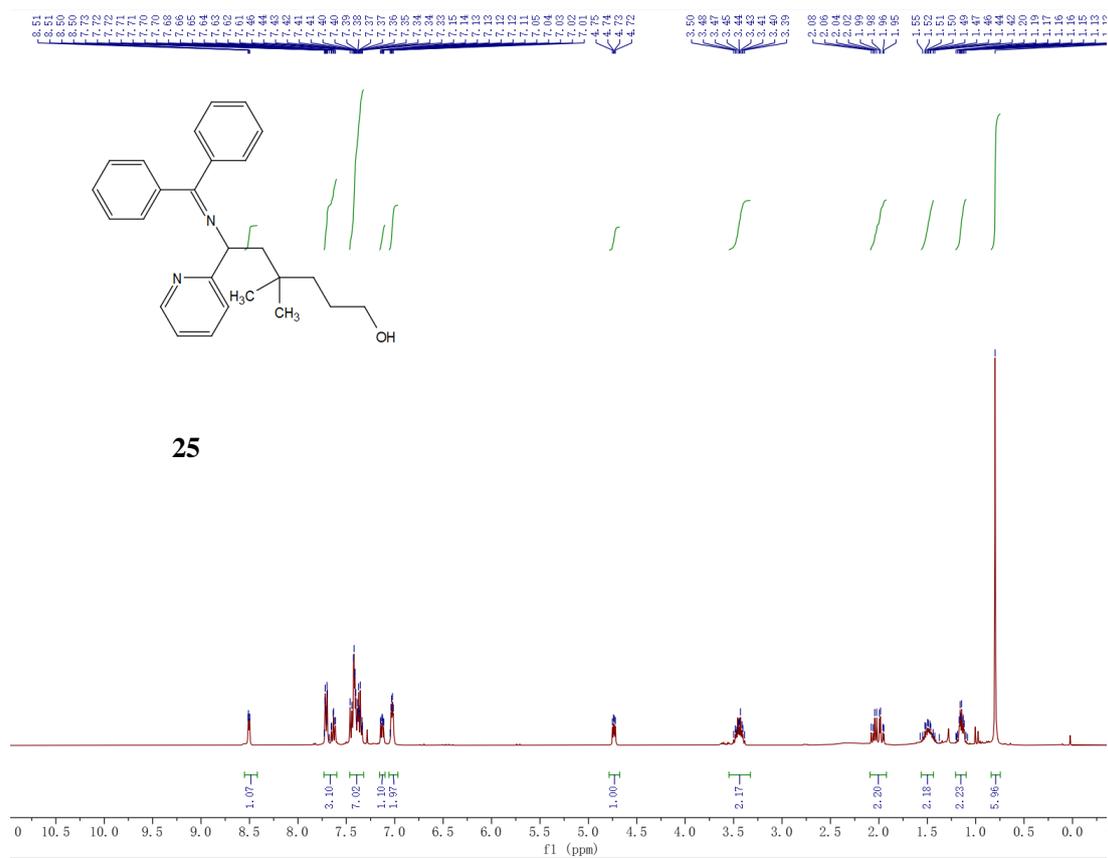
¹H NMR (400 MHz, CDCl₃)



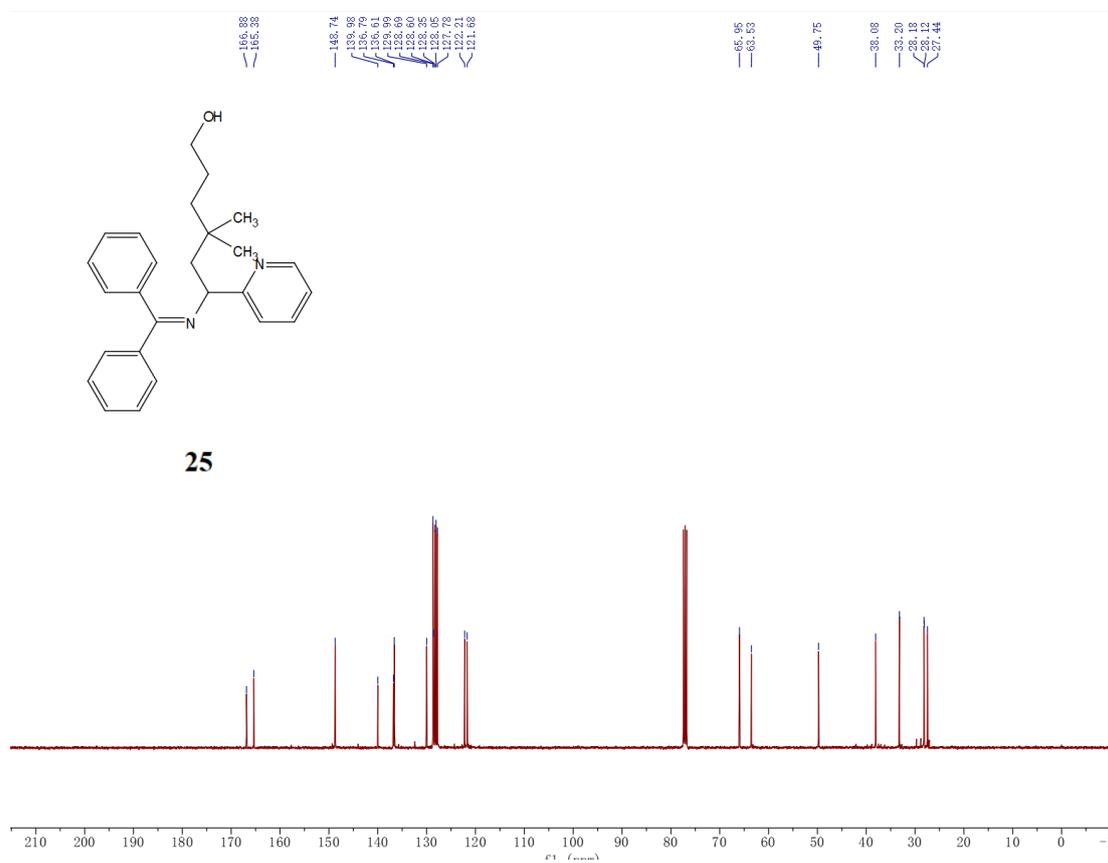
¹³C NMR (101 MHz, CDCl₃)



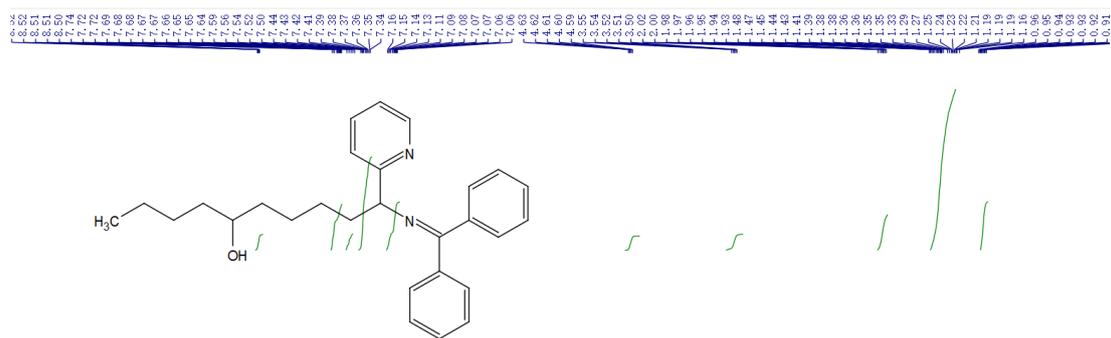
¹H NMR (400 MHz, CDCl₃)



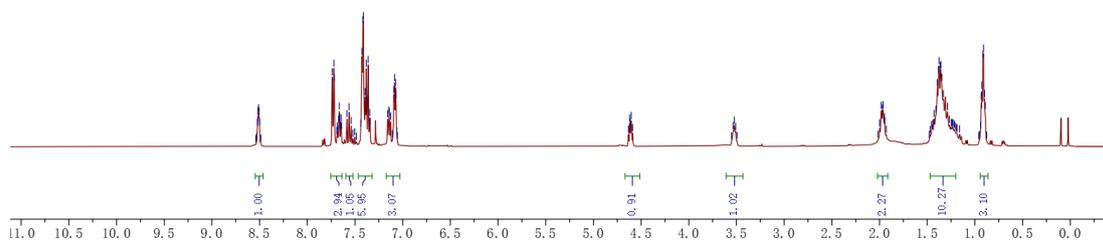
¹³C NMR (101 MHz, CDCl₃)



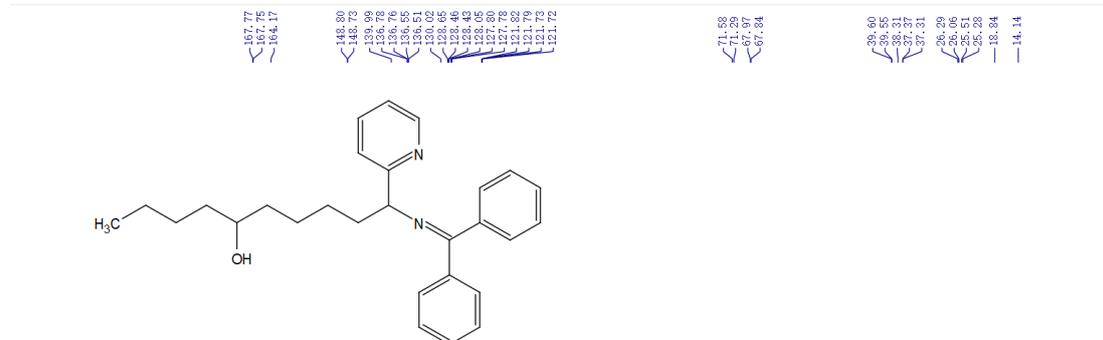
¹H NMR (400 MHz, CDCl₃)



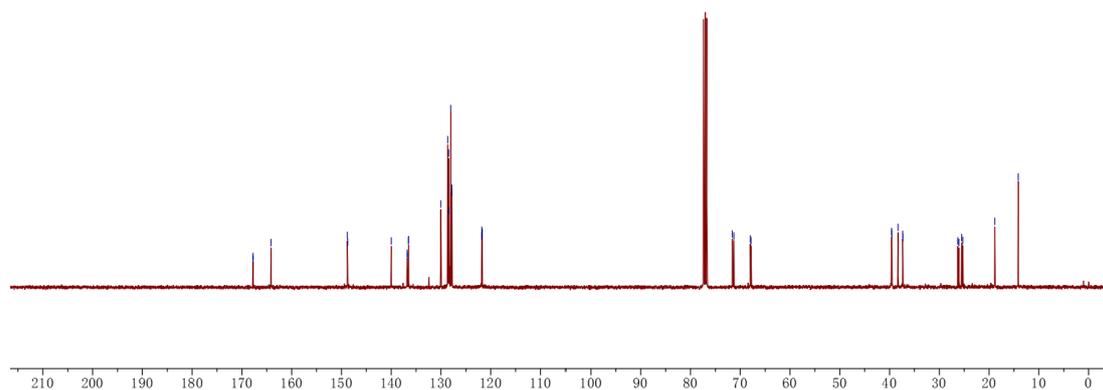
28



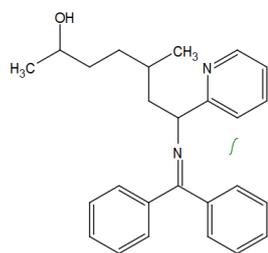
¹³C NMR (101 MHz, CDCl₃)



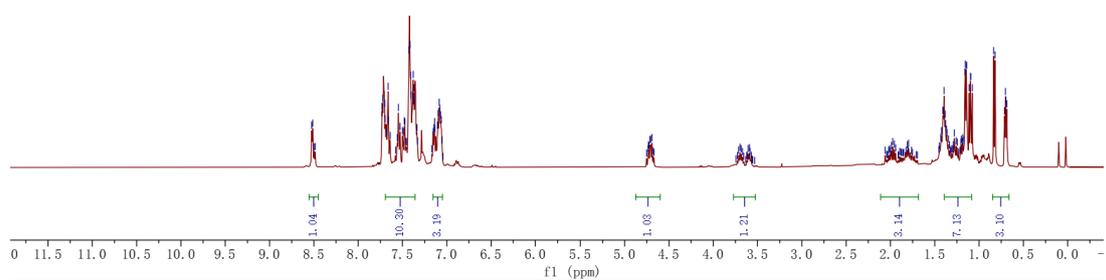
28



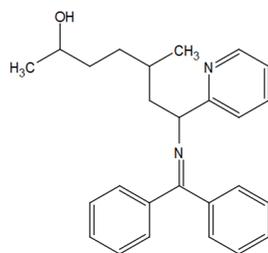
¹H NMR (400 MHz, CDCl₃)



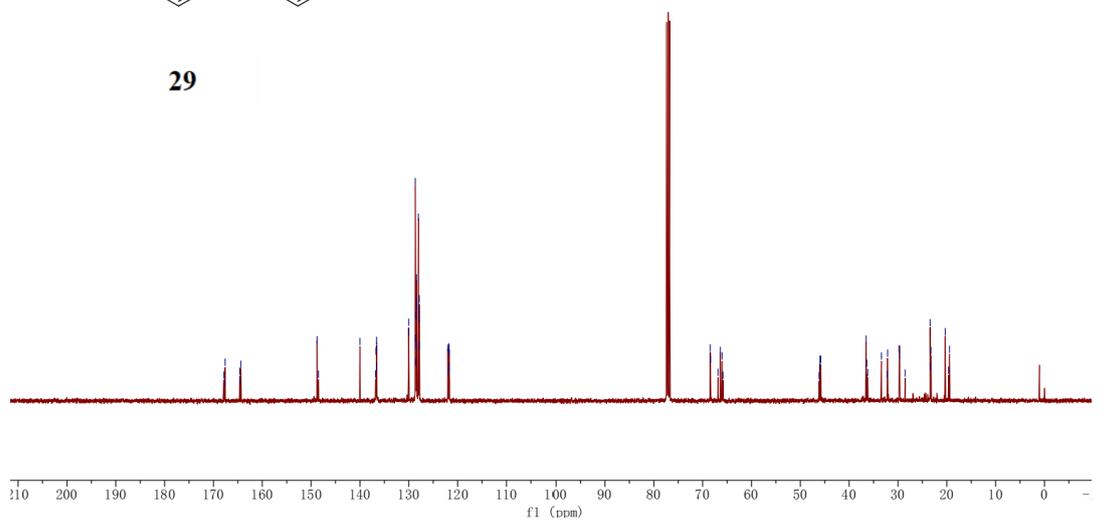
29



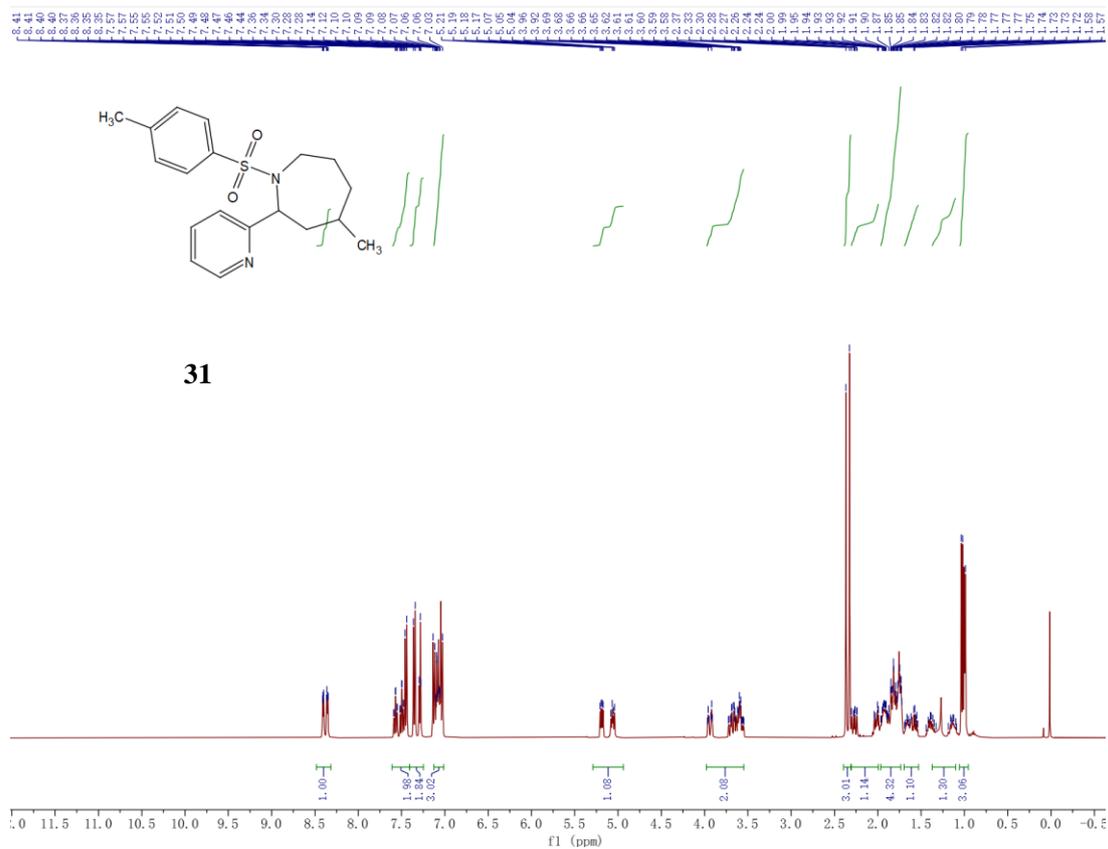
¹³C NMR (101 MHz, CDCl₃)



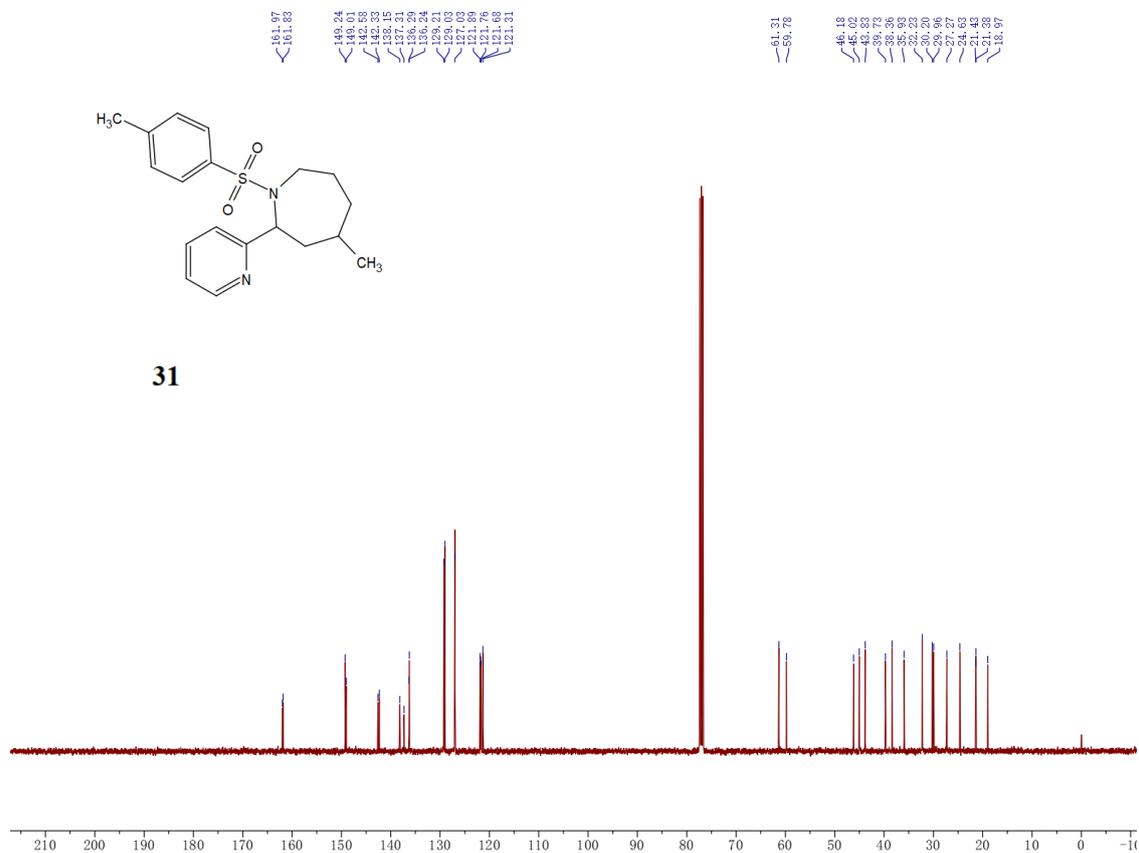
29



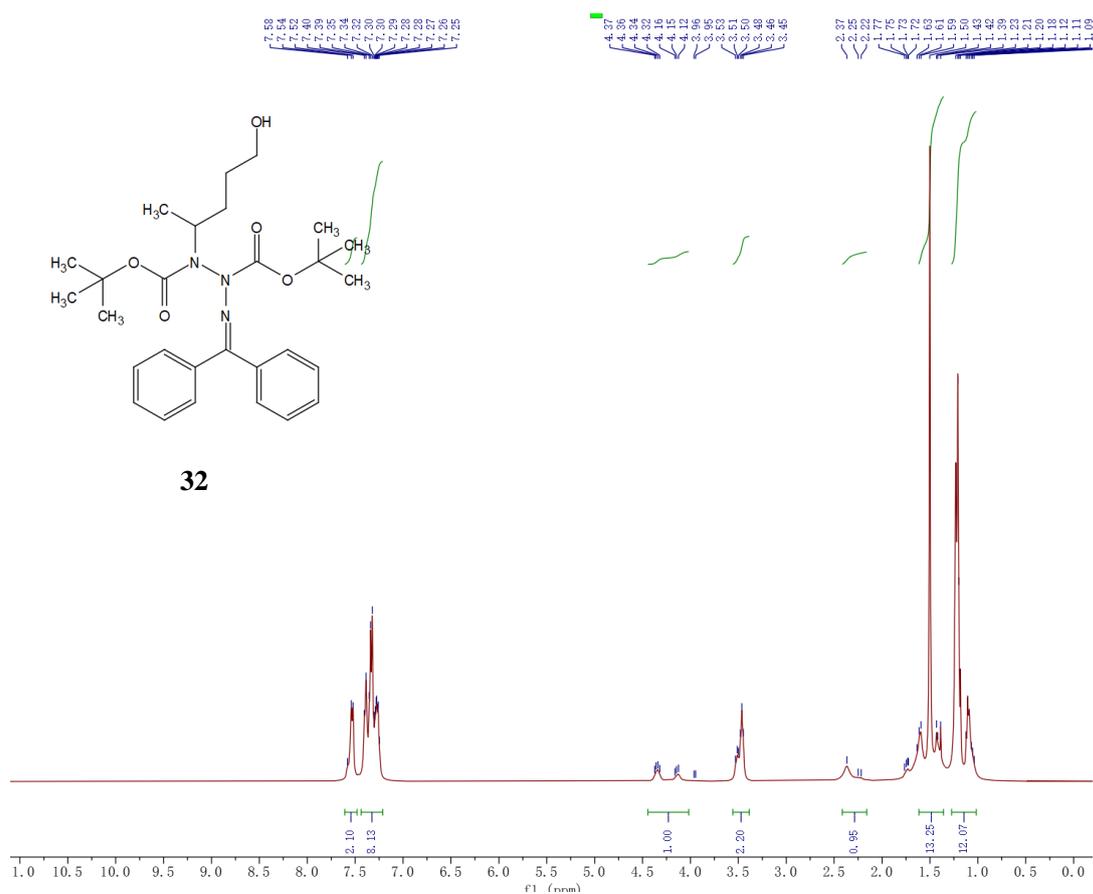
¹H NMR (400 MHz, CDCl₃)



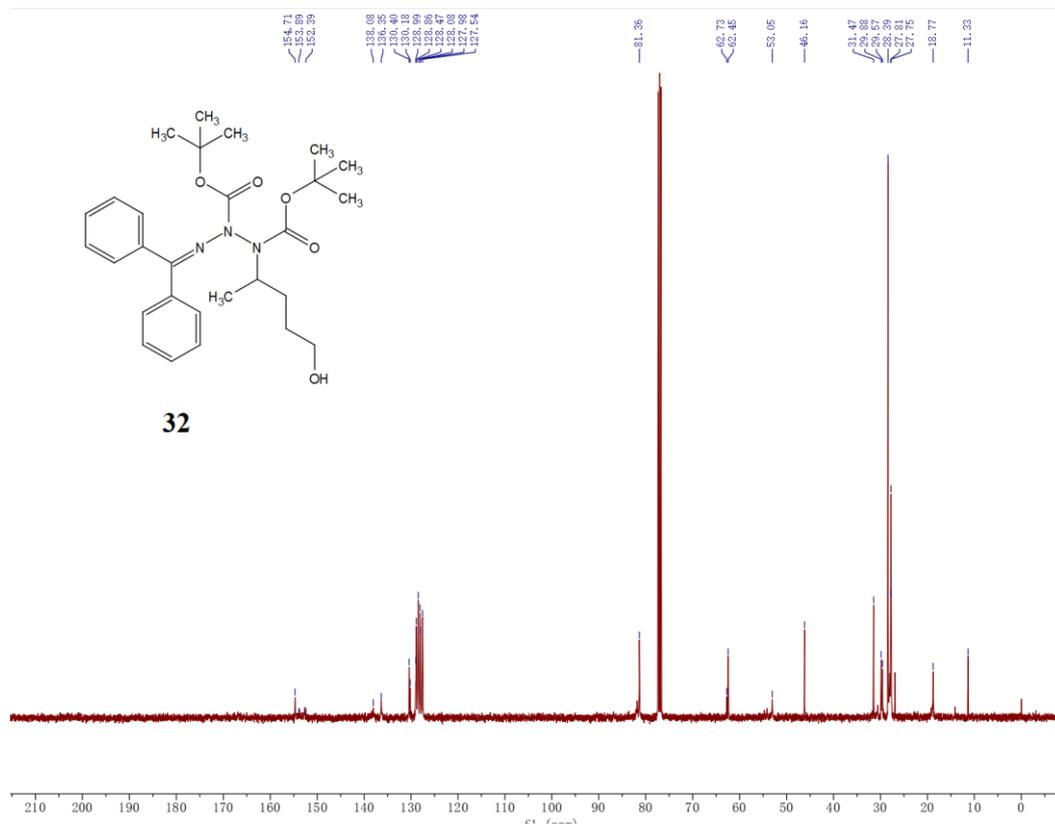
¹³C NMR (101 MHz, CDCl₃)



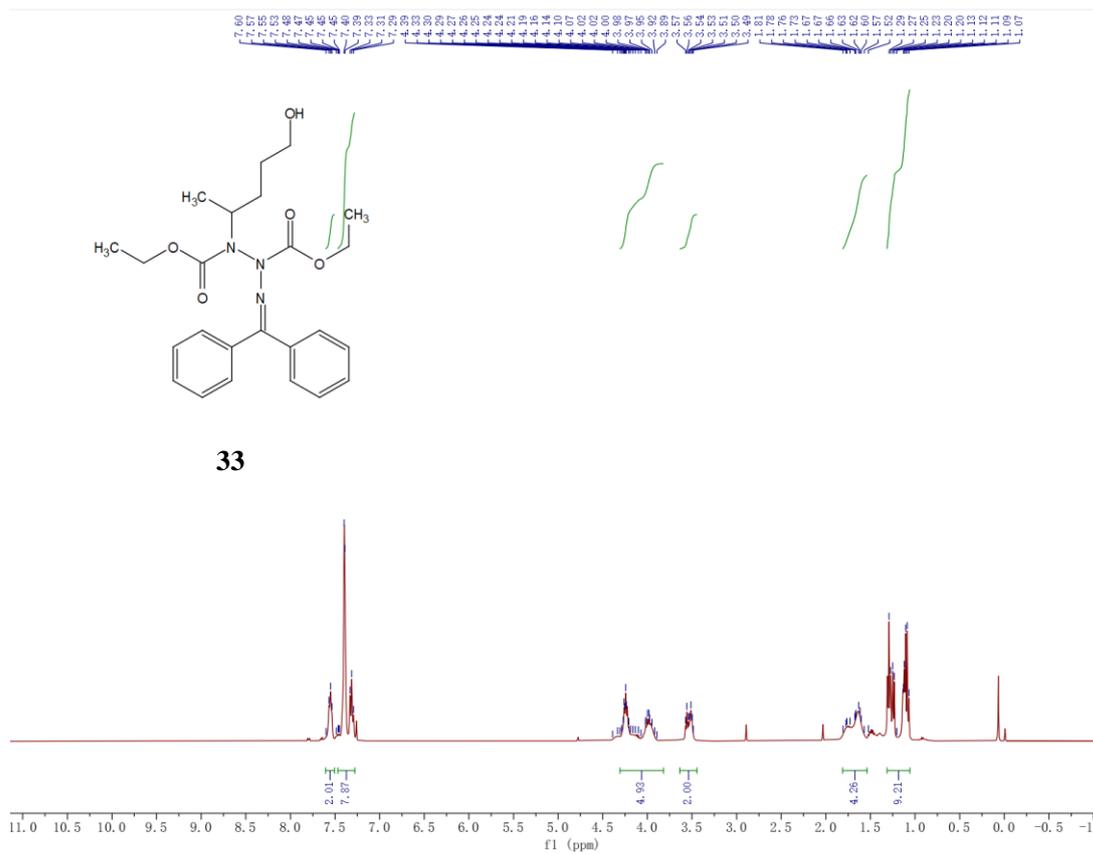
¹H NMR (400 MHz, CDCl₃)



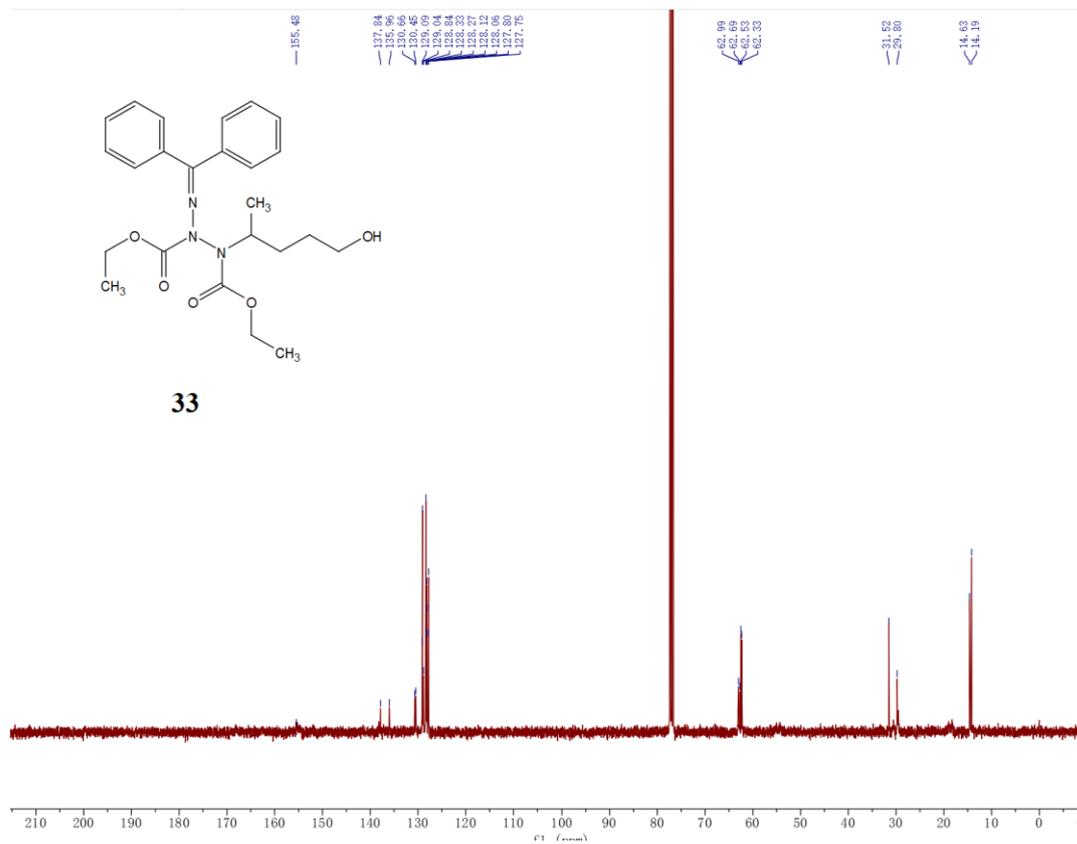
¹³C NMR (101 MHz, CDCl₃)



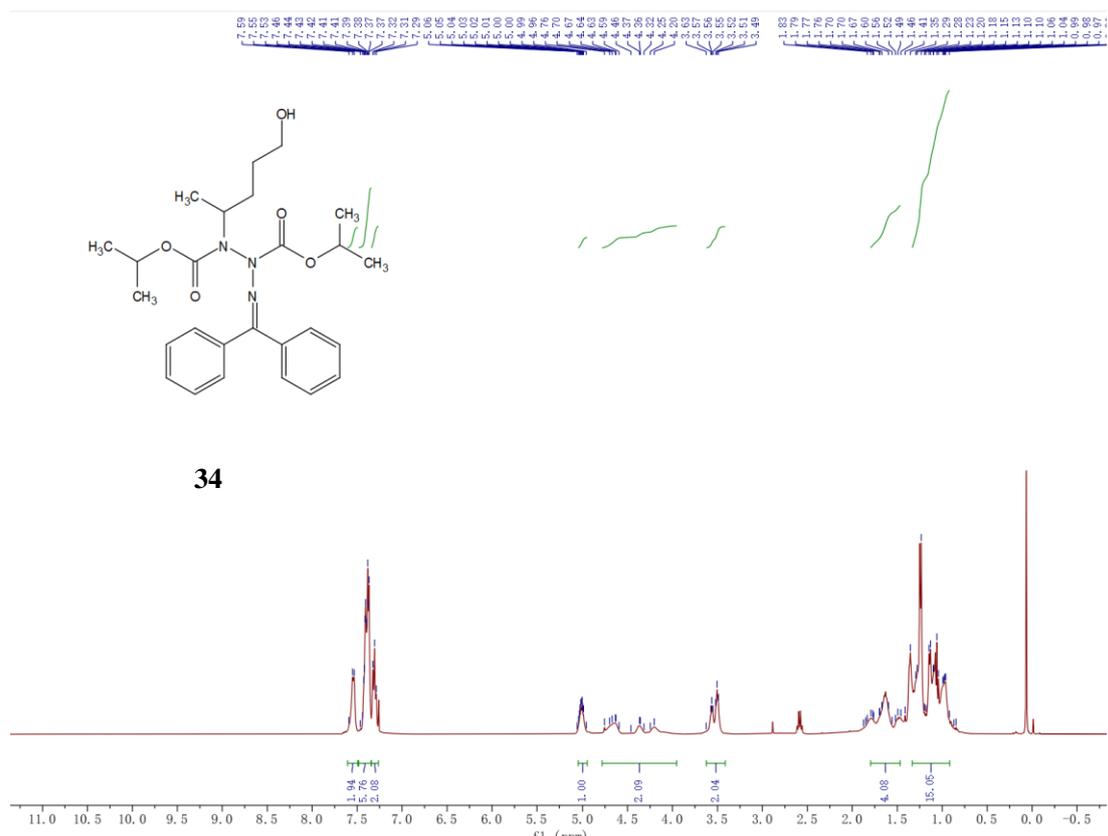
^1H NMR (400 MHz, CDCl_3)



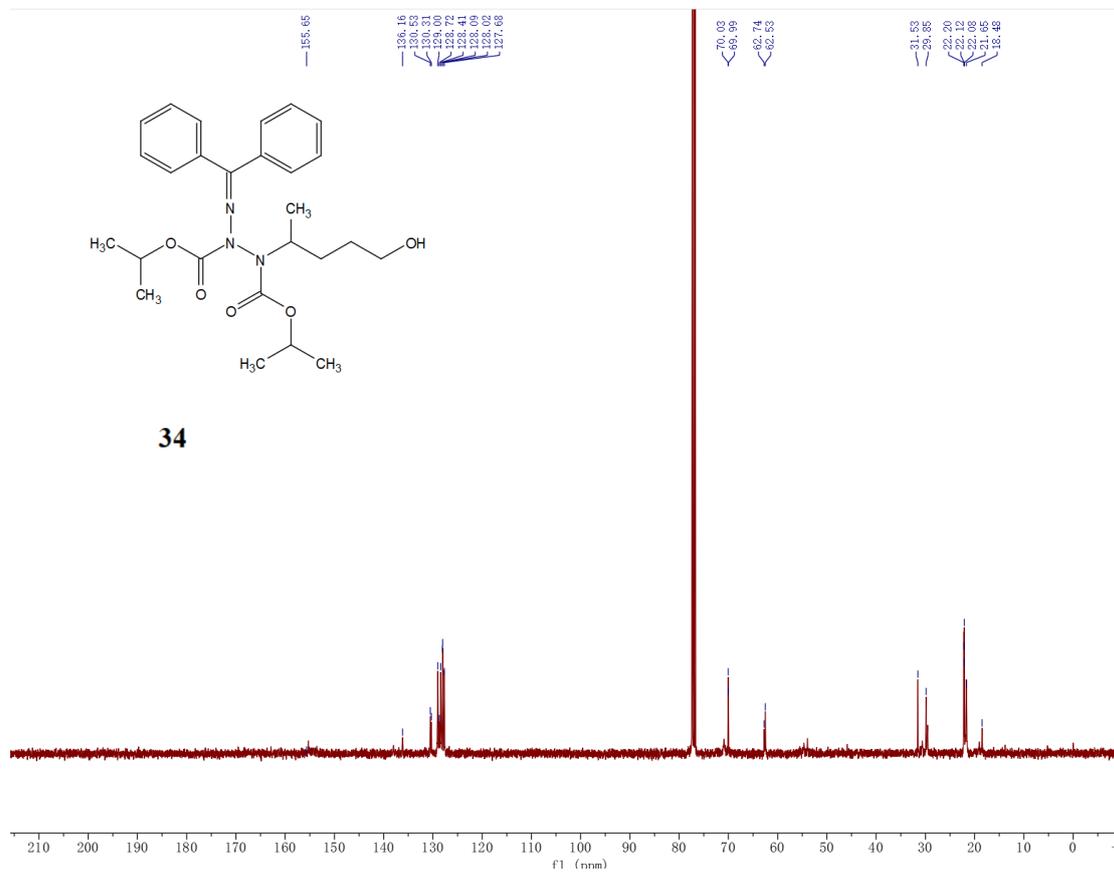
^{13}C NMR (101 MHz, CDCl_3)



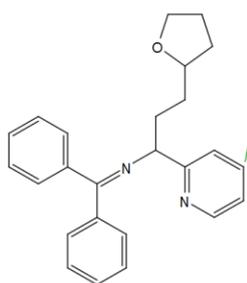
¹H NMR (400 MHz, CDCl₃)



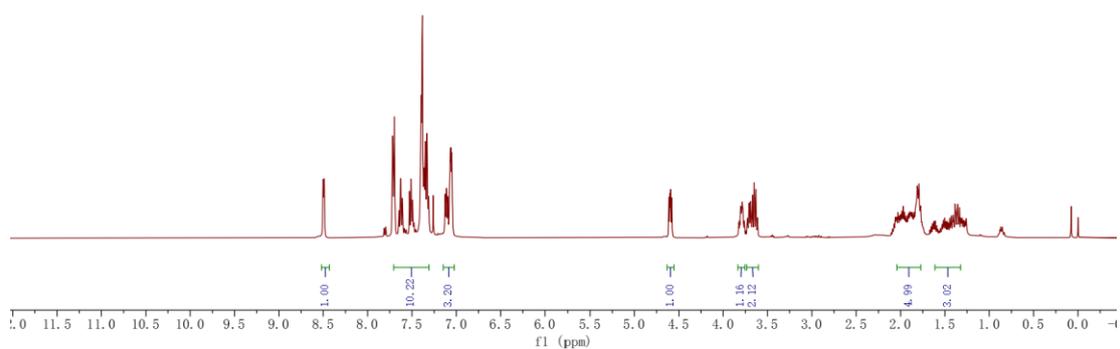
¹³C NMR (101 MHz, CDCl₃)



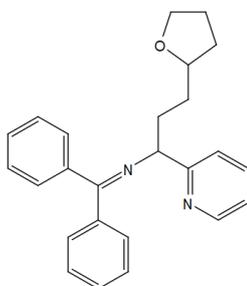
^1H NMR (400 MHz, CDCl_3)



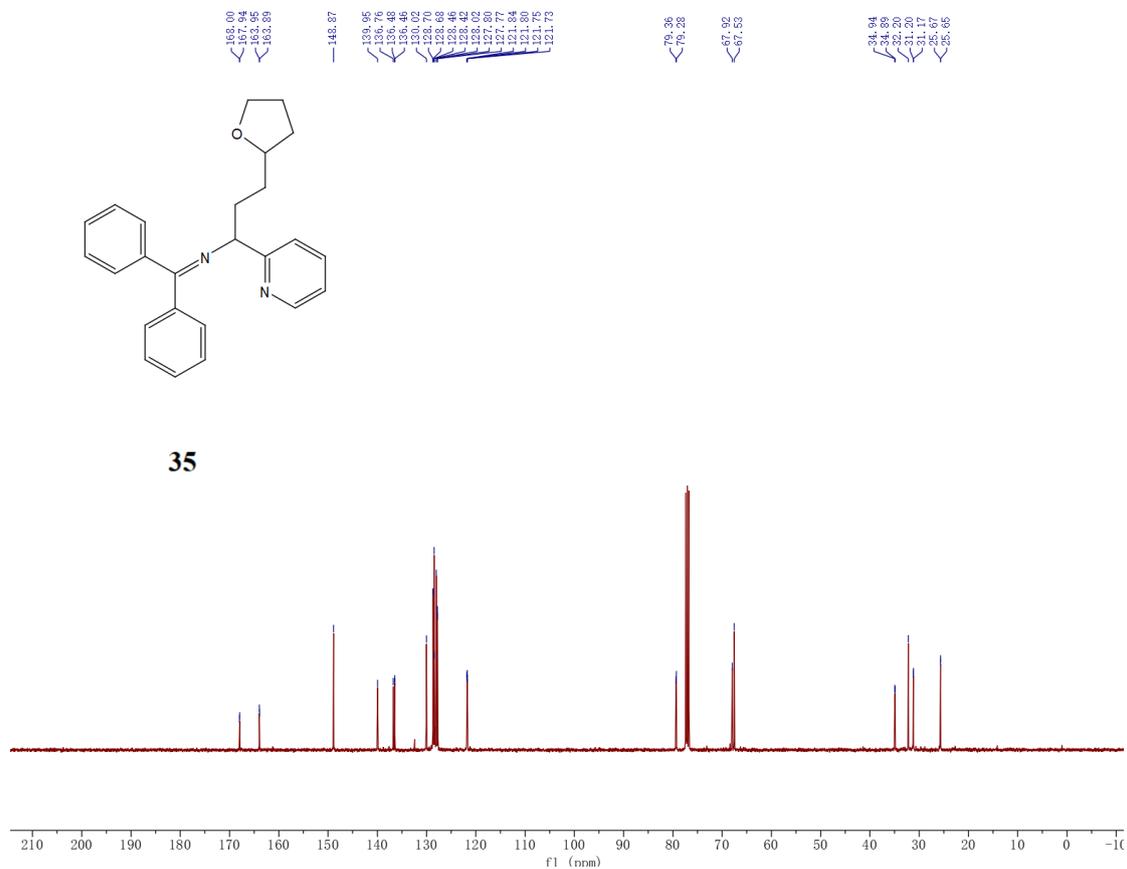
35



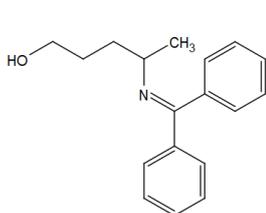
^{13}C NMR (101 MHz, CDCl_3)



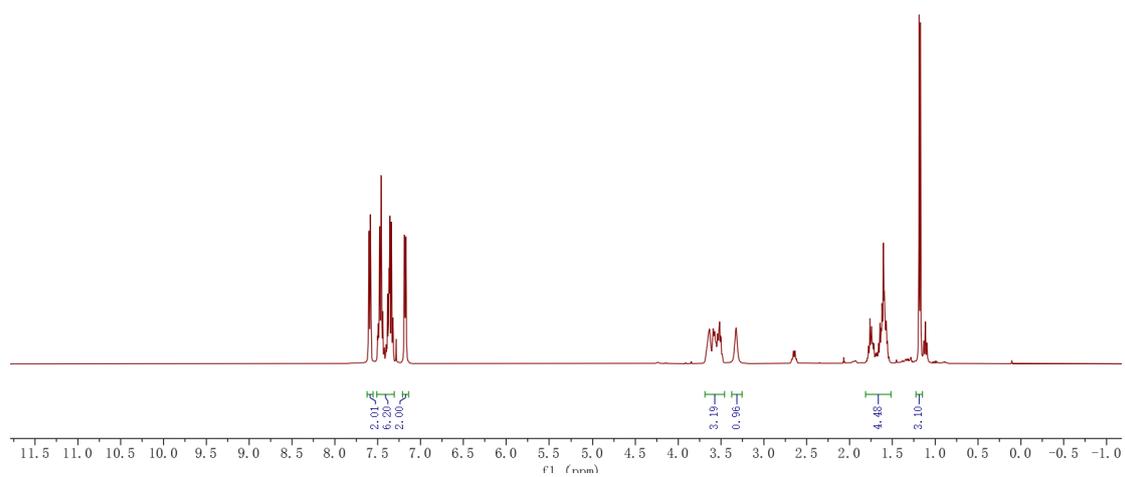
35



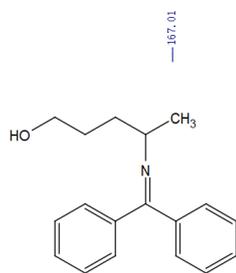
¹H NMR (400 MHz, CDCl₃)



36



¹³C NMR (101 MHz, CDCl₃)



36

